Electronic Supplementary Material (ESI) for Green Chemistry.
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A catalyst-free, waste-less ethanol-based solvothermal synthesis of amides

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1. *Materials and methods*

Commercially available reagents were used as received unless otherwise specified. Acids **1a-j** and amines (**2a-q**) were purchased from Sigma Aldrich.

^{1}H-NMR spectra were recorded on a 400 and 500 MHz Varian spectrometers at 27°C using $CDCl_3$, DMSO-d_6 or CD_3OD as solvent. ^{13}C NMR were recorded at 100 and 125 MHz at 27°C using $CDCl_3$, DMSO-d_6 or CD_3OD as solvent Chemical shifts (δ) are given in ppm. Coupling constants (*J*) are reported in Hz.

Infrared spectra were recorded on a FT-IR Bruker Equinox-55 spectrophotometer.

Low Mass spectra analyses were recorded on an Agilent-HP GC-MS (E.I. 70eV). High resolution mass spectra (HRMS) were obtained using a Bruker High Resolution Mass Spectrometer in fast atom bombardment (FAB+) ionization mode or acquired using an Bruker micrOTOF-Q II 10027.
Analytical thin layer chromatography was performed using 0.25 mm Aldrich silica gel 60-F plates. Flash chromatography was performed using Merk 70-200 mesh silica gel. Yields refer to chromatography or crystallized and spectroscopically pure product unless otherwise stated.
Melting points were determined with a Büchi M560.
HPLC analysis were obtained from Perkin Elmer Flexar Pump Bin Det UV.

2. *Solvothermal synthesis of N-oleyloleamide 4aa: reaction optimization*

Oleic acid **1a** (0.0041 mol to 0.0198 mol) and oleylamine **2a** (molar ratio **1a**/**2a** from 0.83 to 1.00) were poured into a teflon liner with 85 mL capacity and absolute ethanol was added to reach the final volume of 15 mL (full to empty ratio 0.21). The teflon liner was then inserted into a stainless steel autoclave (FigureS1 on the left) and sealed with screws. The autoclave was transferred into the oven at room temperature and the temperature was increased up to the desired final value (from 60 to 190°C) with a rate of 5°C · min^{-1}. Permanence time at each final temperature was varied from 1 to 18 hours. The autoclave was opened after cooling down at room temperature and the crude product was analyzed for ^{1}H-NMR for conversion calculated as follow:

$$^1H - NMR\ Conversion = \frac{Area\ of\ \alpha\ CH_2\ Amide}{Area\ of\ \alpha\ CH_2\ Amide + Area\ of\ \alpha\ CH_2\ Carboxylic\ acid}$$

The mixture was concentrated *in vacuo* and purified by flash chromatography on silica gel (Hexane-EtOAc, 10:1-5:1) to afford the corresponding *N*-oleyloleamide **4aa** and ethyl oleate **3a**.
One experiment was performed by using an autoclave with a capacity four times larger (teflon liner capacity 330 mL, Figure S1 on the right), in order to explore the possibility of scaling up the process (Table S1 entry 19), maintaining all the other parameters unchanged: temperature (160°C), time (6hrs), acid concentration (1.05 M), [carboxylic acid]/[amine] ratio (0.83) and full to empty ratio (0.21).
One experiment was performed to check possible influence of the presence of water in the azeotropic mixture (96%) on the conversion (Table S1 entry 20).

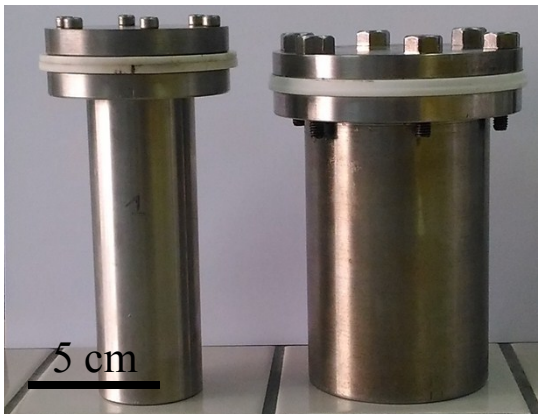
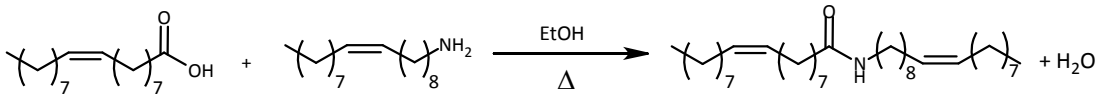


Figure S1 Stainless steel autoclaves with the inner teflon liner: capacity = 85mL (on the left), capacity = 330 mL (on the right).

Table S1 - *N-oleyloleamide optimization of reaction conditions*[a]



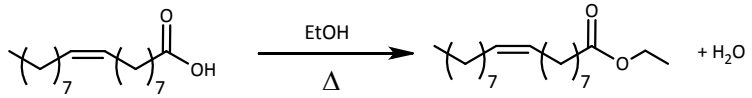
1a **2a** **4aa**

Entry	***T (°C)***	***t (hrs)***	***[Acid] (M)***	***1a/2a molar ratio***	***Solvent***	***Conversion[b] (%)***
1	60	18	0.52	0.83	Absolute Ethanol	n.a.
2	90	18	0.52	0.83	Absolute Ethanol	n.a.
3	130	18	0.52	0.83	Absolute Ethanol	26
4	150	18	0.52	0.83	Absolute Ethanol	57
5	180	18	0.52	0.83	Absolute Ethanol	79
6	160	18	0.52	0.83	Absolute Ethanol	77
7	160	18	0.42	0.83	Absolute Ethanol	80
8	160	18	0.27	0.83	Absolute Ethanol	67
9	160	18	1.32	0.83	Absolute Ethanol	89
10	160	18	0.81	0.83	Absolute Ethanol	90
11	160	18	0.74	0.83	Absolute Ethanol	85
12	160	18	1.05	0.83	Absolute Ethanol	92
13	160	12	1.05	0.83	Absolute Ethanol	96
14	160	6	1.05	0.83	Absolute Ethanol	97
15	160	4	1.05	0.83	Absolute Ethanol	71
16	160	2	1.05	0.83	Absolute Ethanol	47
17	160	1	1.05	0.83	Absolute Ethanol	6
18	160	6	1.05	1.00	Absolute Ethanol	86
19	160	18	1.05	0.83	Absolute Ethanol	91[c]
20	190	18	0.52	0.83	Ethanol 96.5 % v/v	80[d]
21	160	6	1.05	0.83	Methanol 99.8 %	99
22	160	6	1.05	0.83	2-Propanol 99.9 %	91
23	160	6	1.05	0.83	1-Pentanol 99.0 %	94

Table S1 [a]Reaction conditions: 85mL stainless steel autoclave, **1a** (from 0.27 to 1.32 M), **2a** (molar ratio **1a**/**2a** from 0.83 to 1), absolute ethanol to lead the final volume of 15mL, temperature (from 60 to 190°C). [b]Determined by ^{1}H-NMR. [c]330 mL autoclave capacity.

2.1 Study of the reaction mechanism.

Table S2 *Experiments performed to study the reaction mechanism*[a]

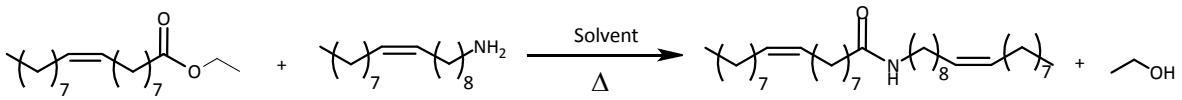


1a **3a**

Entry	***T (°C)***	***Conversion*** [b] ***(%)***
1	60	2
2	90	8
3	130	39
4	150	63
5	160	86
6	180	83

Table S2 *[a] Synthesis of Ethyl oleate **3a** using **1a** (0.0078 mol, 0.5 M), in absolute ethanol and t = 18 hrs. [b]Determined by ^{1}H-NMR.*

Table S3-*Experiments performed to study the mechanism starting from Ethyl oleate **3a**[a].*



3a **2a** **4aa**

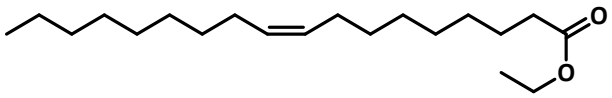
Entry	***Ethyl Oleate [mol]***	***H_2O***[b] ***[mol]***	***3a/H_2O molar ratio***	***CH_3COOH***[c] ***[mol]***	***3a/CH_3COOH molar ratio***	***Conversion***[d] ***(%)***
1	0.0158	0	-	0	-	14[e]
2	0.0158	0	-	0	-	20
3	0.0158	0.0032	0.2	0	-	53
4	0.0158	0.0158	1.0	0	-	58
5	0.0158	0.0236	1.5	0	-	57
6	0.0158	0	0	0.0032	0.2	69
7	0.0158	0.0032	0.2	0.0032	0.2	85

Table S3 *[a]Synthesis of N-oleyloleamide **4aa** starting from Ethyl Oleate **3a**, oleylamine **2a** (0.0191 mol; 1.27 M) in absolute ethanol at 160°C for 6 hours. [b]Bidistilled Water. [c]Glacial acetic acid (pKa 4.75) chosen to avoid any overproduction of oleic ester and because it shows a pKa similar to oleic acid (oleic acid pKa 4.99). [d]Determined by ^{1}H-NMR. [e]Experiment performed for a time reaction of 1 hour.*

3. *General Procedure*

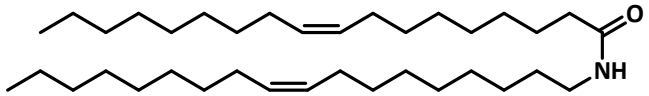
In a typical synthesis, 0.025mol of carboxylic acid (**1a-j**) and 0.030 mol of amine (**2a-q**) were poured into a teflon liner with 85 mL capacity and absolute ethanol was added to reach the final volume of 15 mL (full to empty ratio 0.21). The teflon liner was then inserted into a stainless steel autoclave and sealed with screws. The autoclave was transferred into the oven at room temperature and the temperature was increased up to 160 °C with a rate of 5°C · min^{-1}. After 6 hours the autoclave was opened after cooling down at room temperature and the crude product was analyzed by ^{1}H-NMR for conversion determination. The resulting mixture was concentrated *in vacuo* and purified by flash chromatography on silica gel or crystallization to afford the corresponding amide **4aa-4hq**.

4. *Characterization of ester 3a and amides 4aa-4hq*



(9Z)-ethyl-9-octadecenoate (3a)

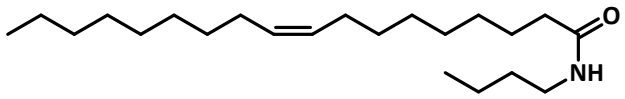
Flash chromatography (Hexane-EtOAc, 10:1-5:1); colourless oil; IR (film): *v* = 2928, 2855, 1733, 1461, 1373, 1279, 1260, 1184, 1097 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 5.34 (m, 2H, CH_2-C**H**=C**H**-CH_2), 4.12 (q, *J* =7.1 Hz, 2H, CH_2), 2.28 (t, *J* = 7.6 Hz, 2H, CH_2), 2.00 (m, 4H, C**H**$_2$-CH=CH-C**H**$_2$), 1.62 (m, 2H, CH_2), 1.38-1.19 (m, 18 H, CH_2 + 3H, CH_3), 0.88 (t, *J* = 6.9 Hz, 3H, CH_3); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 174.0, 130.1, 129.9, 60.3, 34.5, 32.1, 29.9, 29.8, 29.7, 29.5, 29.5, 29.3, 29.3, 29.3, 27.4, 27.3, 25.1, 22.9, 14.4, 14.2; HRMS (ESI): calcd for $C_{20}H_{38}O_2Na$ ($M+Na^+$) 333.5028, found 333.2773. Spectroscopic data are comparable to those previously reported in the literature[1,2].



(9Z)-N-[(9Z)-9-octadecen-1-yl]-9-octadecenamide (4aa)

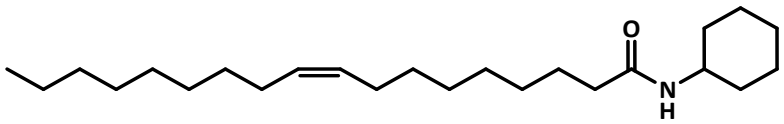
Flash chromatography (Hexane-EtOAc, 10:1-5:1) 88 % yield; white solid (wax), M.p. = 45-47 °C; IR (film): *v* = 3320, 3006, 2852, 1636, 1540, 1469 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ =5.38 (bs, 1H, NH), 5.34 (m, 4H, CH_2-C**H**=C**H**-CH_2), 3.23 (dt, *J* ≈ 6.1 Hz, 7.1 Hz, 2H, CH_2), 2.14 (t, *J* ≈ 7.6 Hz, 2H, CH_2), 2.07-1.93 (m, 8H, C**H**$_2$-CH=CH-C**H**$_2$), 1.61 (m, 2H, CH_2), 1.48 (m, 2H, CH_2), 1.40-1.20 (m, 43H, CH_2),

0.88 (t, *J* = 6.9 Hz, 6H, CH_3), ^{13}C NMR (126 MHz, $CDCl_3$): δ =173.1, 130.1, 130.1, 129.9, 129.9, 129.9, 39.7, 37.1, 32.1, 29.9, 29.9, 29.9, 29.9, 29.8, 29.8, 29.7, 29.6, 29.5, 29.5, 29.5, 29.5, 29.4, 29.4, 29.3, 27.4, 27.4, 27.3, 27.1, 26.0, 22.8, 14.2 ; HRMS (ESI) calcd for $C_{36}H_{70}NO$ ($M+H^+$) 532.9443, found 532.5461.



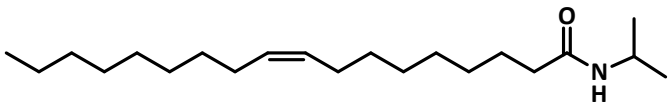
(9Z)-N-butyl-9-octadecenamide (4ab)

Flash chromatography (Hexane-EtOAc, 10:1-5:1) 86 % yield; colourless oil; IR (film): *v* = 3316, 3053, 2930, 2855, 1650, 1544, 1465, 1264 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 5.59 (s, 1H, NH), 5.32 (m, 2H, CH_2-C**H**=C**H**-CH_2), 3.22 (dt, *J* ≈ 5.9 Hz, 7.1 Hz, 2H, CH_2), 2.13 (t, *J* ≈ 7.7 Hz, 2H, CH_2), 1.99 (m, 4H, C**H**$_2$-CH=CH-C**H**$_2$), 1.60 (m, 2H, CH_2), 1.46 (m, *J* ≈ 7.4 Hz, 2H, CH_2), 1.36-1.21 (m, 22H, CH_2), 0.90 (t, *J* = 7.3 Hz, 3H, CH_3), 0.86 (t, *J* = 6.9 Hz, 3H, CH_3); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 173.3, 130.1, 129.8, 39.3, 37.0, 32.0, 31.9, 29.9, 29.8, 29.8, 29.6, 29.4, 29.4, 29.4, 29.2, 27.3, 27.3, 25.9, 22.8, 20.2, 14.2, 13.8; HRMS (ESI) calcd for $C_{22}H_{44}NO$ ($M+H^+$) 338.5891, found 338.3431. Spectroscopic data are comparable to those previously reported in the literature[3]**.**



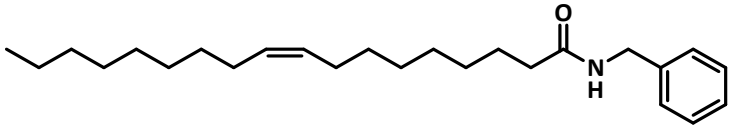
(9Z)-N-cyclohexyl-9-octadecenamide (4ac)

Flash chromatography (Hexane-EtOAc, 10:1-5:1) 76 % yield; white solid (wax), Mp = 36-37 °C; IR (film): *v* = 3286, 3002, 2927, 2855, 1643, 1547, 1458, 1254 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 5.94 (m, 1H, NH), 5.24 (m, 2H, CH_2-C**H**=C**H**-CH_2), 3.67 (m, 1H, CH), 2.06 (t, *J* = 7.6 Hz, 2H, CH_2), 1.92 (m, 4H, C**H**$_2$-CH=CH-C**H**$_2$), 1.84-0.99 (m, 32H, CH_2), 0.79 (t, *J* = 6.8 Hz, 3H, CH_3); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 172.3, 129.8, 129.6, 48.0, 36.8, 33.1, 31.8, 29.7, 29.7, 29.6, 29.6, 29.6, 29.4, 29.4, 29.3, 29.2, 29.1, 27.1, 27.1, 25.9, 25.5, 24.9, 22.6, 14.0; HRMS (ESI) calcd for $C_{24}H_{46}NO$ ($M+H^+$) 364.6263, found 364.3576. Spectroscopic data are comparable to those previously reported in the literature[4]**.**



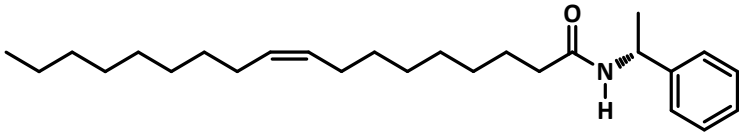
(9Z)-N-isopropyl-9-octadecenamide (4ad)

Flash chromatography (Hexane-EtOAc, 10:1-5:1) 80 % yield; yellow oil; IR (film): *v* = 3285, 3076, 3002, 2927, 2855, 1639, 1602, 1554, 1464, 1370, 1175 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 5.34 (m, 2H, CH_2-C**H**=C**H**-CH_2), 5.27 (bs, 1H, NH), 4.08 (m, 1H, CH), 2.11 (t, *J* = 7.6 Hz, 2H, CH_2), 2.00 (m, 4H, C**H**$_2$-CH=CH-C**H**$_2$), 1.61 (m, 2H, CH_2), 1.40-1.17 (m, 20H, CH_2), 1.14 (d, *J* = 6.6 Hz, 6H, CH_3), 0.87 (t, *J* = 6.7 Hz, 3H, CH_3); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 172.4, 130.1, 129.9, 41.4, 37.2, 32.0, 29.9, 29.9, 29.7, 29.5, 29.5, 29.4, 29.3, 27.4, 27.3, 26.0, 23.0, 23.0, 23.0, 22.8, 14.2; HRMS (ESI) calcd for $C_{21}H_{42}NO$ ($M+H^+$) 324.5626, found 324.3276.



(9Z)-N-benzyl-9-octadecenamide (4ae)

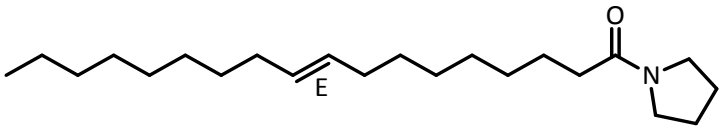
Flash chromatography (Hexane-EtOAc, 10:1-5:1) 87 % yield; white solid, Mp = 52-55 °C; IR (film): *v* = 3299, 3070, 3033, 3006, 2927, 2852, 1643, 1554, 1458, 1257, 1229, 1083, 1028 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7-35-7.24 (m, 5H, CH), 5.82 (bs, 1H, NH), 5.34 (m, 2H, CH_2-C**H**=C**H**-CH_2), 4.43 (d, *J* = 5.7 Hz, 2H, CH_2), 2.20 (t, *J* ≈ 7.6 Hz, 2H, CH_2), 2.01 (m, 4H, C**H**$_2$-CH=CH-C**H**$_2$), 1.65 (m, 2H, CH_2), 1.40-1.20 (m, 20H, CH_2), 0.88 (t, *J* = 6.9 Hz, 3H, CH_3); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 173.1, 138.6, 130.1, 129.9, 128.8, 128.8, 127.9, 127.9, 127.6, 43.7, 36.9, 32.0, 29.9, 29.8, 29.6, 29.4, 29.4, 29.4, 29.4, 29.3, 27.4, 27.3, 25.9, 22.8, 14.2; HRMS (ESI) calcd for $C_{25}H_{42}NO$ ($M+H^+$) 372.6054, found 372.3275. Spectroscopic data are comparable to those previously reported in the literature[5]**.**



(9Z)-(R)-N-(1-phenylethyl)-9-octadecenamide (4af)

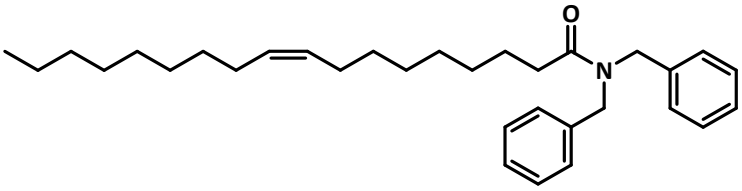
Flash chromatography (Petroleum Ether-Et_2O, 10:1-1:1) 82 % yield; colourless oil; IR (film): *v* = 3283, 2924, 2854, 1639, 1545, 1450, 1376, 700 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.37-7.22 (m, 5H, CH), 7.71 (bs, 1H, NH), 5.34 (m, 2H, CH_2-C**H**=C**H**-CH_2), 5.14 (m, 1H, CH), 2.15 (t, *J* = 7.6 Hz, 2H,

CH_2), 2.00 (m, 4H, C**H**$_2$-CH=CH-C**H**$_2$), 1.62 (m, 2H, CH_2), 1.48 (d, *J* = 6.8 Hz, 3H, CH_3), 1.37-1.21 (m, 20H, CH_2), 0.88 (t, *J* = 6.6 Hz, 3H, CH_3); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 172.2, 143.5, 130.1, 129.9, 128.8, 128.8, 127.4, 126.3, 126.3, 48.7, 37.0, 32.0, 29.9, 29.8, 29.6, 29.5, 29.4, 29.4, 29.4, 29.3, 27.4, 27.3, 25.9, 22.8, 21.8, 14.2; HRMS (ESI) calcd for $C_{26}H_{43}NONa$ ($M+Na^+$) 408.6138, found 408.3238. Chiral HPLC analysis (OJ-H column), *i*-PrOH/hexane 2:98, 1.0 mL min^{-1}. λ = 210 nm, R_t (major) = 19.9 min, R_t (minor) = 21.8 min, ee 98%. Spectroscopic data are comparable to those previously reported in the literature[5].



(9E)-1-(pyrrolidin-1-yl)octadec-9-en-1-one (4ag)

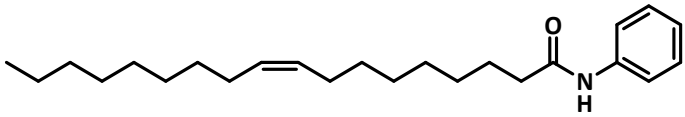
Flash chromatography (Hexane-EtOAc, 10:1-5:1) 92 % yield; yellow oil; IR (film): *v* = 3241, 3053, 2930, 2959, 1633, 1455, 1346, 1267 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 5.32 (m, 2H, CH_2-C**H**=CH_2-C**H**), 3.44 (t, *J* = 6.9 Hz, 2H, CH_2), 3.39 (t, *J* = 6.8 Hz, 2H, CH_2), 2.23 (t, *J* ≈ 7.6 Hz, 2H, CH_2), 1.99 (m, 4H, trans C**H**$_2$-CH=C**H**$_2$-CH), 1.93 (quint, *J* ≈ 6.6 Hz, 2H, CH_2), 1.83 (quint, *J* ≈ 6.7 Hz, 2H, CH_2), 1.62 (m, 2H, CH_2), 1.37-1.20 (m, 20H, CH_2), 0.86 (t, *J* = 7.0 Hz, 3H, CH_3); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 172.1, 130.0, 129.9, 46.8, 45.7, 35.0, 32.0, 29.9, 29.8, 29.6, 29.6, 29.5, 29.4, 29.4, 29.3, 27.3, 27.3, 26.2, 25.1, 24.5, 22.8, 14.2; HRMS (ESI) calcd for $C_{22}H_{42}NO$ ($M+H^+$) 336.5733, found 336.3270.



(9Z)-N,N-dibenzyl-9-octadecenamide (4ah)

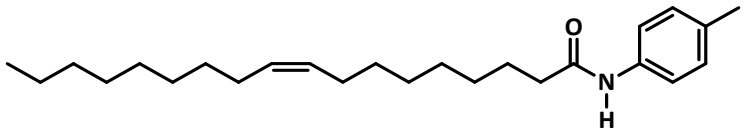
Flash chromatography (Petroleum Ether-Et_2O, 10:1-1:1) 40 % yield; colourless oil; IR (film): *v* = 2924, 2850, 1648, 1454, 1417, 1207, 1083, 729, 692 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.38-7.10 (m, 10H, CH), 5.34 (m, 2H, CH_2-C**H**=C**H**-CH_2), 4.60 (s, 2H, CH_2), 4.43 (s, 2H, CH_2), 2.41 (t, *J* = 7.7 Hz, 2H, CH_2), 2.01 (m, 4H, C**H**$_2$-CH=CH-C**H**$_2$), 1.71 (m, 2H, CH_2), 1.39-1.21 (m, 20H, CH_2), 0.87 (t, *J* = 6.9 Hz, 3H, CH_3); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 173.7, 137.6, 136.7, 130.0, 129.8, 128.9, 128.9, 128.6, 128.6, 128.3, 128.3, 127.6, 127.4, 126.4, 126.4, 50.0, 48.1, 33.3, 32.0, 29.8, 29.8, 29.6, 29.5, 29.4,

29.4, 29.4, 29.2, 27.3, 27.2, 25.5, 22.7, 14.2; HRMS (ESI) calcd for $C_{32}H_{47}NONa$ (M+Na^+) 484.7096, found 484.3533.



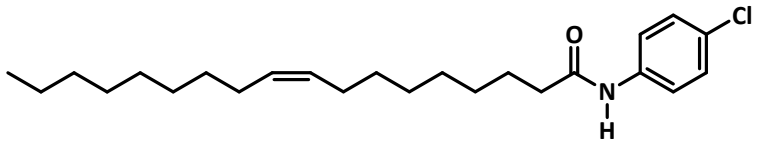
(9Z)-N-phenyl-9-Octadecenamide (4ai)

Flash chromatography (Petroleum Ether: Et_2O, 10:1--1:1) 50 % yield; orange oil; IR (film): *v* = 3303, 3064, 3006, 2927, 2855, 1667, 1602, 1547, 1503, 1446 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7-35-7.24 (m, 5H, CH), 5.82 (bs, 1H, NH), 5.34 (m, 2H, CH_2-C**H**=C**H**-CH_2), 4.43 (d, *J* = 5.7 Hz, 2H, CH_2), 2.20 (t, *J* ≈ 7.6 Hz, 2H, CH_2), 2.01 (m, 4H, C**H**$_2$-CH=CH-C**H**$_2$), 1.65 (m, 2H, CH_2), 1.40-1.20 (m, 20H, CH_2), 0.88 (t, *J* = 6.9 Hz, 3H, CH_3); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 171.4, 138.1, 130.2, 129.9, 129.1, 129.1, 124.3, 119.9, 119.9, 38.0, 32.1, 29.9, 29.9, 29.7, 29.5, 29.5, 29.4, 29.4, 29.3, 27.4, 27.3, 25.8, 22.8, 14.3; HRMS (ESI) calcd for $C_{24}H_{40}NO$ (M+H^+) 358.5789, found 358.3109. Spectroscopic data are comparable to those previously reported in the literature[6].



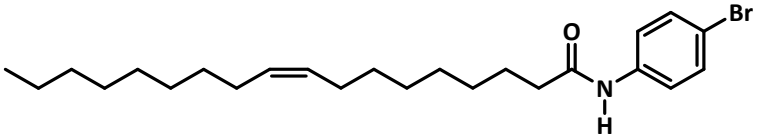
(9Z)-N-(4-methylphenyl)-9-octadecenamide (4aj)

Flash chromatography (Hexane-EtOAc, 10:1-5:1) 33 % yield; white/pale yellow solid; Mp = 40°C; IR (film): *v* = 3291, 2920, 2846, 1662, 1512, 1363, 1215, 1030 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.46 (bs, 1H, NH), 7.39 (d, *J* = 8.2 Hz, 2H, CH), 7.09 (d, *J* = 8.1 Hz, 2H, CH), 5.35 (m, 2H, CH_2-C**H**=C**H**-CH_2), 2.37-2.25 (m, 5H, CH_2 + CH_3), 2.01 (m, 4H, C**H**$_2$-CH=CH-C**H**$_2$), 1.70 (m, 2H, CH_2), 1.40-1.22 (m, 20H, CH_2), 0.89 (t, *J* = 6.9 Hz, 3H, CH_3); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 171.6, 135.6, 133.8, 130.1, 129.8, 129.5, 120.1, 37.8, 32.0, 29.9, 29.9, 29.8, 29.8, 29.6, 29.4, 29.4, 29.4, 29.4, 29.3, 27.3, 27.3, 25.8, 22.8, 20.9, 14.2; HRMS (ESI) calcd for $C_{24}H_{38}ClNONa$ (M+Na^+) 394.5873, found 394.3079.



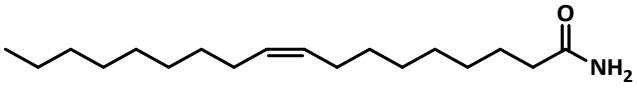
(9Z)-N-(4-chlorophenyl)-9-octadecenamide (4ak)

Flash chromatography (Petroleum Ether-Et_2O, 10:1-1:1) 21% yield; white solid; Mp = 134-136°C; IR (film): *v* = 3295, 2924, 2854, 1660, 1596, 1536, 1491, 1400, 1087, 828 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ = 7.46 (d, *J* = 8.4 Hz, 2H, CH), 7.27 (d, *J* = 8.2 Hz, 2H, CH), 7.08 (s, 1H, NH), 5.34 (m, 2H, CH_2-C**H**=C**H**-CH_2), 2.34 (t, *J* = 7.5 Hz, 2H, CH_2), 2.01 (m, 4H, C**H**$_2$-CH=CH-C**H**$_2$), 1.72 (m, 2H, CH_2), 1.40-1.23 (m, 20H, CH_2), 0.88 (t, *J* = 6.9 Hz, 3H, CH_3); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 171.6, 136.7, 130.2, 129.8, 129.3, 129.1, 129.1, 121.2, 121.2, 37.9, 32.0, 29.9, 29.8, 29.7, 29.5, 29.5, 29.4, 29.4, 29.3, 27.4, 27.3, 25.7, 22.8, 14.2; HRMS (ESI) calcd for $C_{24}H_{38}ClNONa$ (M+Na^+) 414.2540, found 414.2543.



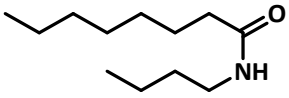
(9Z)-N-(4-bromophenyl)-9-octadecenamide (4al)

Flash chromatography (Petroleum Ether-Et_2O, 10:1-1:1) 22% yield; orange wax; IR (film): *v* = 3670, 3295, 2990, 2970, 2916, 1660, 1528, 1392, 1244, 1063, 890, 820 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ = 7.41 (s, 4H, CH), 7.18 (bs, 1H, NH), 5.34 (m, 2H, CH_2-C**H**=C**H**-CH_2), 2.33 (t, *J* = 7.6 Hz, 2H, CH_2), 2.01 (m, 4H, C**H**$_2$-CH=CH-C**H**$_2$), 1.71 (m, 2H, CH_2), 1.40-1.18 (m, 20H, CH_2), 0.88 (t, *J* = 6.9 Hz, 3H, CH_3); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 171.5, 137.2, 132.1, 130.2, 129.8, 121.4, 116.8, 37.9, 32.1, 29.9, 29.8, 29.7, 29.5, 29.5, 29.4, 29.4, 29.4, 29.3, 29.3, 27.4, 27.3, 25.7, 22.8, 14.3; HRMS (ESI) calcd for $C_{24}H_{38}BrNONa$ (M+Na^+) 459.4569, found 458.2041.



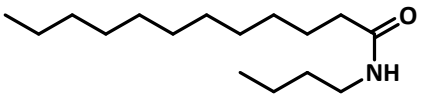
(9Z)-9-Octadecenamide (4an)

Flash chromatography (Hexane-EtOAc, 10:1-1:1) 35 % yield; wax, Mp = 73-76 °C; IR (film): *v* = 2930, 2853, 2668, 1705, 1570, 1462, 1414, 1209, 1093, 966, 937, 724 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ = 6.03 (s, 1H, NH_2), 5.60 (s, 1H, NH_2)[7], 5.32 (m, 2H, CH_2-C**H**=C**H**-CH_2), 2.18 (t, *J* ≈ 7.6 Hz, 2H, CH_2), 1.99 (m, 4H, C**H**$_2$-CH=CH-C**H**$_2$), 1.36-1.18 (m, 18H, CH_2), 0.86 (t, *J* = 6.9 Hz, 3H, CH_3); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 176.1, 130.1, 129.8, 36.1, 32.0, 29.9, 29.8, 29.6, 29.4, 29.4, 29.3, 29.3, 29.2, 27.3, 27.3, 25.6, 22.8, 14.2; HRMS (ESI) calcd for $C_{18}H_{36}NO$ (M+H^+) 282.4831 found 282.2800. Spectroscopic data are comparable to those previously reported in the literature[8].



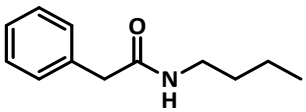
N-butyloctanamide (4bb)

Flash chromatography (Hexane-EtOAc, 10:1-5:1) 78 % yield; colourless oil; IR (film): *v* = 3292, 3084, 2958, 2930, 2862, 1646, 1557, 1465, 1380, 1270, 1229 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 5.84 (s, 1H, NH); 3.19 (dt, *J* = 6.80, 6.41 Hz, 2H, CH_2); 2.11 (t, *J* = 7.6 Hz, 2H, CH_2); 1.57 (m, 2H, CH_2); 1.43 (m, 2H, CH_2); 1.34-1.20 (m, 10 H, CH_2); 0.87 (t, *J* = 7.3 Hz, 3H, CH_3); 0.83 (t, *J* = 6.8 Hz, 3H, CH_3); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 173.3, 39.2, 36.9, 31.8, 31.8, 29.3, 29.1, 25.9, 22.6, 20.1, 14.1, 13.8; HRMS (ESI) calcd for $C_{12}H_{26}NO$ ($M+H^+$) 200.3399, found 200.2159. Spectroscopic data are comparable to those previously reported in the literature[9].



N-butyldodecanamide (4cb)

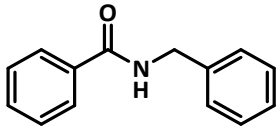
Flash chromatography (Hexane-EtOAc, 10:1-5:1) >98 % yield; white solid, M.p. = 55-58 °C; IR (film): *v* = 3306, 3050, 2965, 2951, 2930, 2859, 1643, 1554, 1469, 1376, 1267 cm^{-1}; ^{1}H NMR (500 MHz, CDCl3): δ = 5.56 (s, 1H, NH); 3.23 (dt, *J* ≈ 6.0, 7.0 Hz, 2H, CH_2); 2.14 (t, *J* ≈ 7.6 Hz, 2H, CH_2); 1.61 (m, 2H, CH_2); 1.47 (m, , *J* ≈ 7.5, 2H, CH_2); 1.38-1.18 (m, 18 H, CH_2); 0.91 (t, *J* = 7.3 Hz, 3H, CH_3); 0.86 (t, *J* = 7.0 Hz, 3H, CH_3); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 173.2, 39.3, 37.0, 32.0, 31.9, 29.7, 29.7, 29.6, 29.5, 29.5, 29.5, 26.0, 22.8, 20.2, 14.2, 13.9; HRMS (ESI) calcd for $C_{16}H_{34}NO$ ($M+H^+$) 256.4459, found 256.2662. Spectroscopic data are comparable to those previously reported in the literature[10].



N-butyl-2-phenylacetamide (4db)

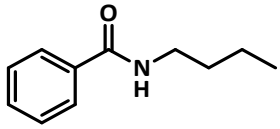
Flash chromatography (Hexane-EtOAc, 10:1-5:1) 90 % yield; wax (white solid), M.p. = 37-39 °C; IR (film): *v* = 3436, 3320, 3057, 2965, 2934, 2869, 1660, 1530, 1264 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.26-7.19 (m, 5H, CH); 5.53 (bs, 1H, NH); 3.53 (s, 2H, CH_2); 3.18 (dt, *J* ≈ 7.0, 6.0 Hz, 2H, CH_2); 1.38 (m, , *J* ≈ 7.4 Hz, 2H, CH_2); 1.23 (m, , *J* ≈ 7.4 Hz, 2H, CH_2); 0.85 (t, *J* = 7.4 Hz, 3H, CH_3); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 171.1, 135.3, 129.6, 129.6, 129.1, 129.1, 127.4, 44.0, 39.6, 31.7, 20.1, 13.8; HRMS

(ESI) calcd for $C_{12}H_{18}NO$ ($M+H^+$) 192.2688, found 192.1380. Spectroscopic data are comparable to those previously reported in the literature[11].



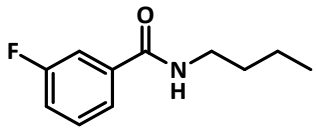
N-benzylbenzamide (4ee)

Crystallization (IPA, 0 °C) 59 % yield; white solid, M.p. = 97-100 °C; IR (film): *v* = 3316, 2961, 2927, 2859, 1643, 1544, 1267 cm^{-1}; ^1H NMR (500 MHz, DMSO): δ = 7.92 (m, 2H, CH), 7.49 (m, 2H, CH), 7.35 (m, 6H, CH), 4.01 (s, 2H, CH_2); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 167.6, 138.4, 134.4, 131.5, 128.7, 128.7, 128.5, 128.5, 127.8, 127.8, 127.5, 127.4, 127.1, 44.0; HRMS (ESI) calcd for $C_{14}H_{14}NO$ ($M+H^+$) 212.2665, found 212.1075. Spectroscopic data are comparable to those previously reported in the literature[12].



N-butylbenzamide (4eb)

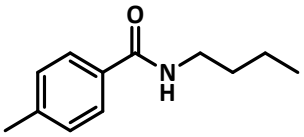
Flash chromatography (Hexane-EtOAc, 10:1-5:1) 41 % yield; colourless oil; IR (film): *v* = 3316, 3067, 2958, 2934, 2872, 1643, 1544, 1492, 1311, 697 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ = 7.77-7.73 (m, 2H, CH); 7.42-7.37 (m, 1H, CH); 7.33-7.28 (m, 2H, CH); 6.95 (bs, 1H, NH); 3.35 (dt, *J* ≈ 7.1, 6.1 Hz, 2H, CH_2); 1.52 (m, , *J* ≈ 7.4, 2H, CH_2); 1.32 (m, *J* ≈ 7.4, 2H, CH_2); 0.87 (t, *J* = 7.4 Hz, 3H, CH_3); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 167.7, 134.8, 131.1, 128.3, 128.3, 126.9, 126.9, 39.8, 31.6, 20.1, 13.7; HRMS (ESI) calcd for $C_{11}H_{16}NO$ ($M+H^+$) 178.2502, found 178.1228. Spectroscopic data are comparable to those previously reported in the literature[13].



N-butyl-3-fluorobenzamide (4fb)

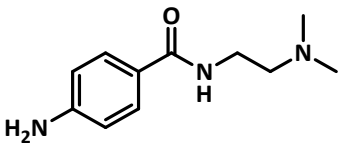
Flash chromatography (Hexane-EtOAc, 10:1-5:1) 50 % yield; colourless oil; IR (film): *v* = 3316, 3077, 2961, 2937, 2876, 1639, 1588, 1551, 1489, 1445, 1304 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ = 7.53-7.46 (m, 2H, CH); 7.41-7.35 (m, 1H, CH), 7.20-7.14 (m, 1H, CH), 6.27 (bs, 1H, NH); 3.45 (dt, *J* ≈ 7.0,

6.3 Hz, 2H, CH_2); 1.60 (m, 2H, CH_2); 1.41 (m, 2H, CH_2); 0.96 (t, *J* = 7.4 Hz, 3H, CH_3); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 166.4 (d, J_F = 2.4 Hz), 137.3 (d, *J* = 7.3 Hz), 130.3 (d, *J* = 8.3 Hz), 122.4 (d, *J* = 3.4 Hz), 118.4 (d, *J* = 21.4 Hz), 114.4 (d, *J* = 23.0 Hz), 107.5, 40.1, 31.8, 20.3, 13.9; HRMS (ESI) calcd for $C_{11}H_{15}FNO$ ($M+H^+$) 196.2407, found 196.1155.



N-butyl-4-methylbenzamide (4gb)

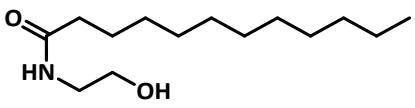
Flash chromatography (Hexane-EtOAc, 10:1-5:1) 22 % yield; white solid, M.p. = 52-53° C; IR (film): *v* = 3320, 3064, 2961, 2934, 2866, 1639, 1547, 1506, 1462, 1311 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.65 (d, *J* = 8.2 Hz, 2H, CH); 7.20 (d, *J* = 7.9 Hz, 2H, CH); 6.23 (bs, 1H, NH); 3.43 (dt, *J* ≈ 7.1, 5.9 Hz, 2H, CH_2); 2.38 (s, 3H, CH_3); 1.58 (m, 2H, CH_2); 1.40 (m, 2H, CH_2); 0.94 (t, *J* = 7.4 Hz, 3H, CH_3); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 167.6, 141.7, 132.1, 129.3, 129.3, 127.0, 127.0, 39.9, 31.9, 21.5, 20.3, 13.9; HRMS (ESI) calcd for $C_{12}H_{18}NO$ ($M+H^+$) 192.2767, found 192.1382. Spectroscopic data are comparable to those previously reported in the literature[14].



4-amino-N-[2-(dimethylamino)ethyl]benzamide (4ho)

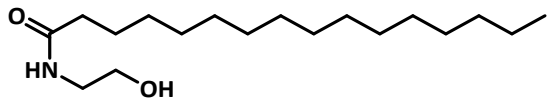
Crystallization (IPA/Hexane, 5:1) 78 % yield; hygroscopic white solid, Mp = 105 °C; IR (film): *v* = 3349, 3231, 1637, 1608, 1540, 1375, 1297, 1170, 1132, 1025, 850, 792, 704, 616 cm^{-1}; ^{1}H NMR (500 MHz, DMSO): δ = 7.58 (d, *J* = 8.7 Hz, 2H, CH); 6.49 (d, *J* = 8.7 Hz, 2H, CH); 5.49 (bs, 1H, NH); 2.70 (t, *J* = 6.4 Hz, 2H, CH_2); 2.32 (t, *J* = 6.4 Hz, 2H, CH_2), 2.13 (s, 6H, CH_3); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 169.2, 151.6, 130.9, 121.8, 112.5, 79.2, 59.5, 45.2, 37.9; HRMS (ESI) calcd for $C_{11}H_{18}NO$ ($M+H^+$) 208.2794, found 208.1461

4.1 Biologically active compounds



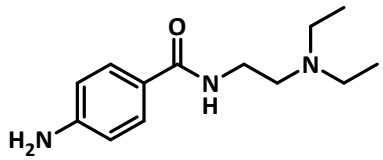
N-(2-hydroxyethyl)dodecanamide (4cp LEA)

^{1}H NMR (500 MHz, $CDCl_3$): δ = 6.31 (s, NH); 3.68 (t, *J* ≈ 5.0 Hz, CH_2); 3.49 (bs, OH); 3.38 (dt, *J* ≈ 5.3, 5.1 Hz, CH_2); 2.18 (t, *J* ≈ 7.6 Hz, CH_2); 1.60 (m, CH_2); 1.31-1.17 (m, CH_2); 0.86 (t, *J* = 6.9 Hz, CH_3). Spectroscopic data are comparable to those previously reported in the literature[15].



N-(2-hydroxyethyl)hexadecanamide (4jp PEA)

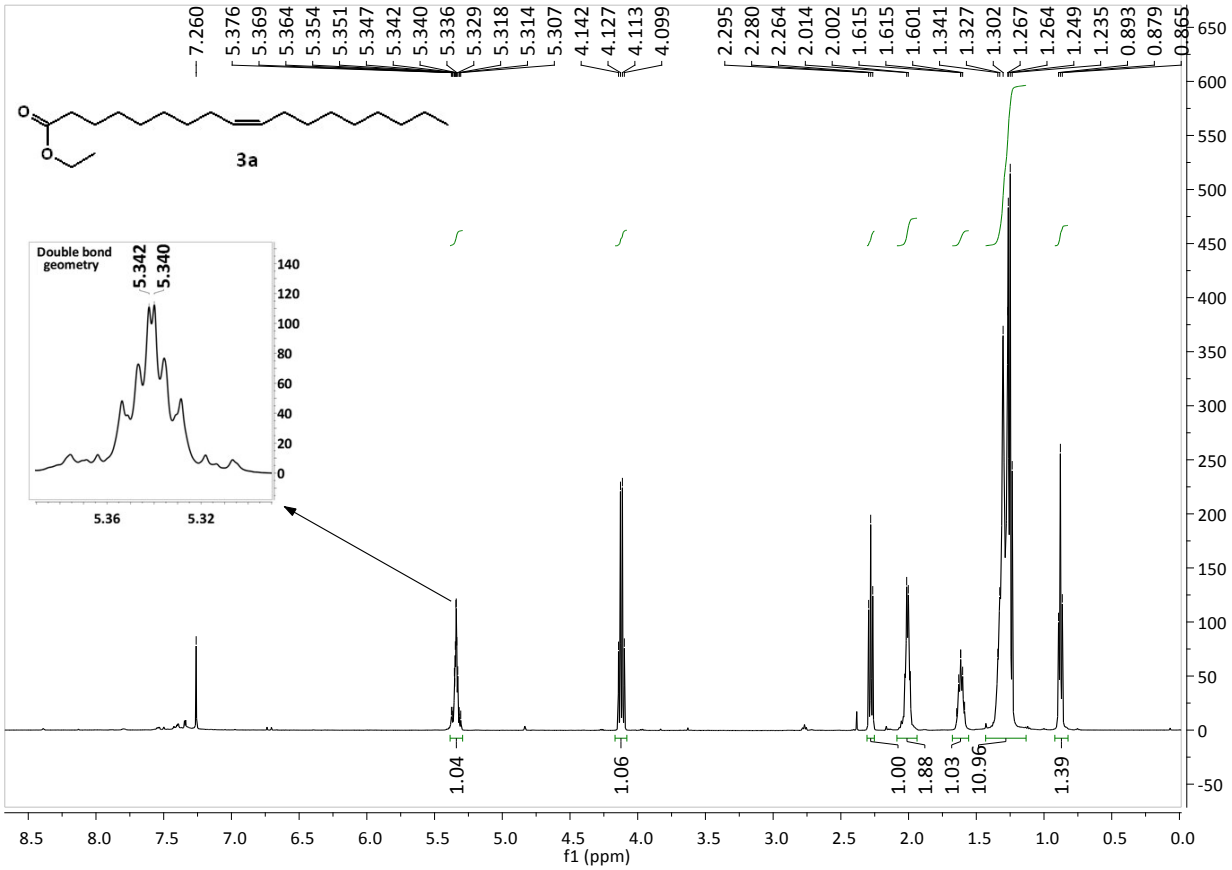
^{1}H NMR (500 MHz, $CDCl_3$): δ = 5.93 (bs, OH); 5.75 (bt, NH); 3.72 (t, *J* ≈ 5.4 Hz, CH_2); 3.42 (dt, *J* ≈ 5.4, 4.6 Hz, CH_2); 1.63 (m, CH_2); 1.40-1.20 (m, CH_2); 0.86 (t, *J* = 6.9 Hz, CH_3); Spectroscopic data are comparable to those previously reported in the literature[16].

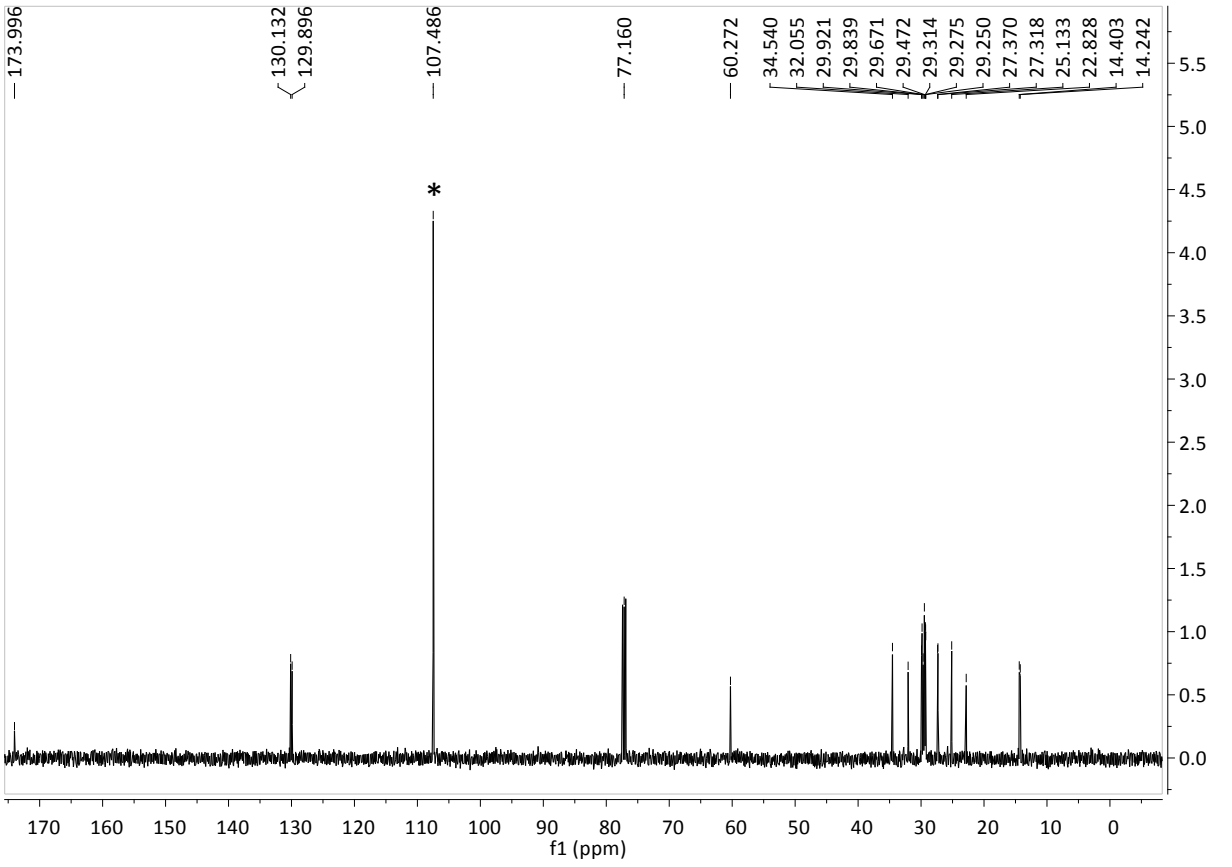


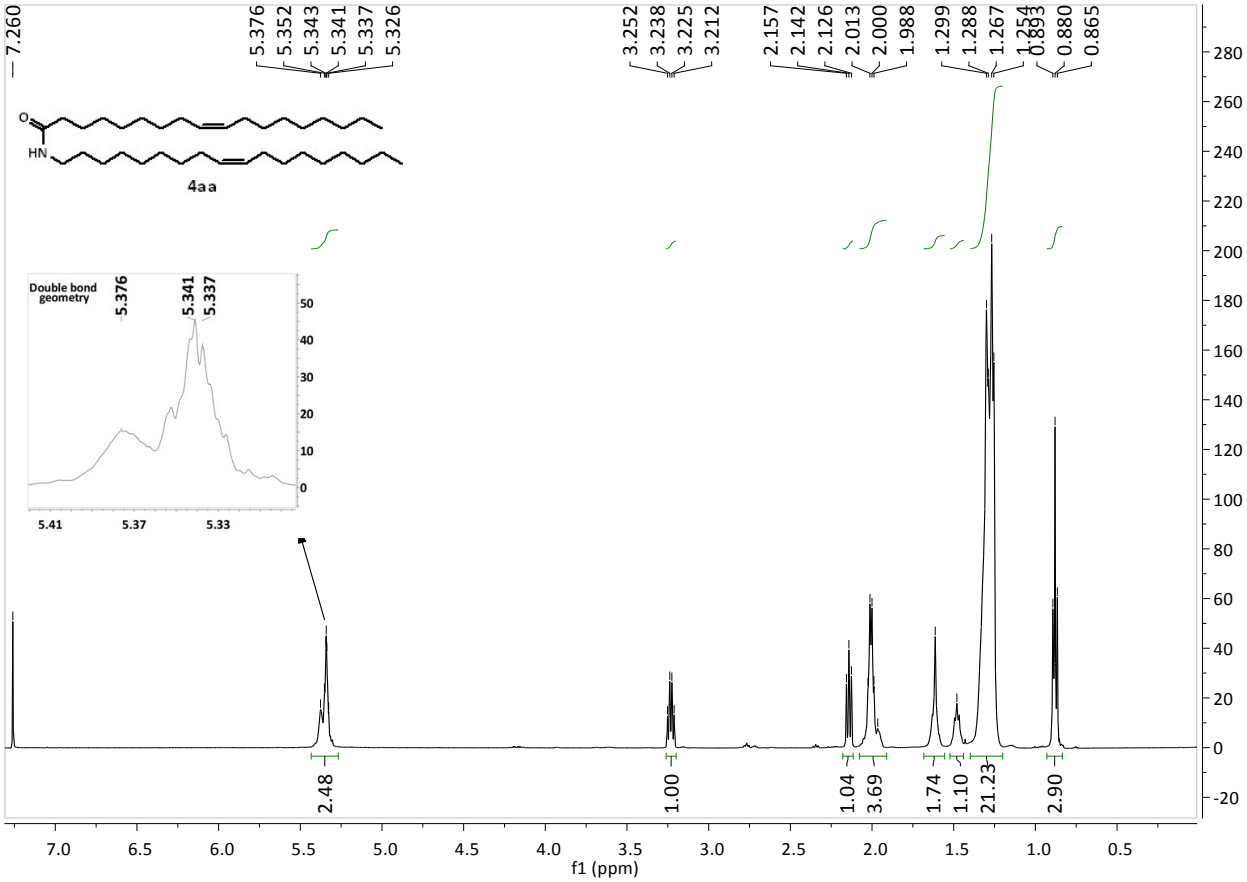
4-amino-N-[2-(diethylamino)ethyl]benzamide (4hq Procainamide)

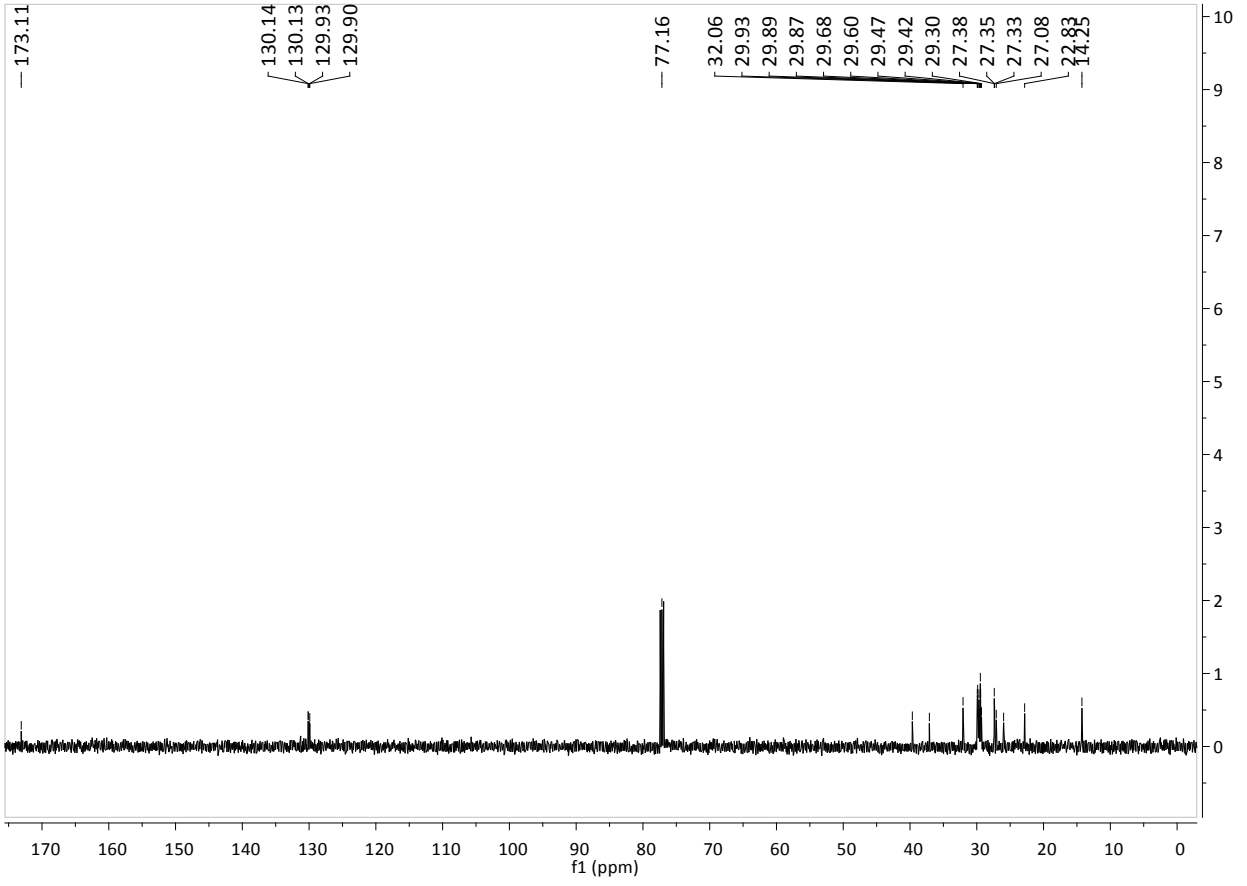
^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.88 (d, *J* = 8.6 Hz, CH); 6.64 (d, *J* = 8.6 Hz, CH); 3.59 (dt, *J* = 5.8, 5.3 Hz, CH_2); 2.83 (t, *J* = 5.7 Hz, CH_2); 2.77 (q, *J* = 7.3 Hz, CH_2); 1.12 (t, *J* = 7.3 Hz, CH_3);

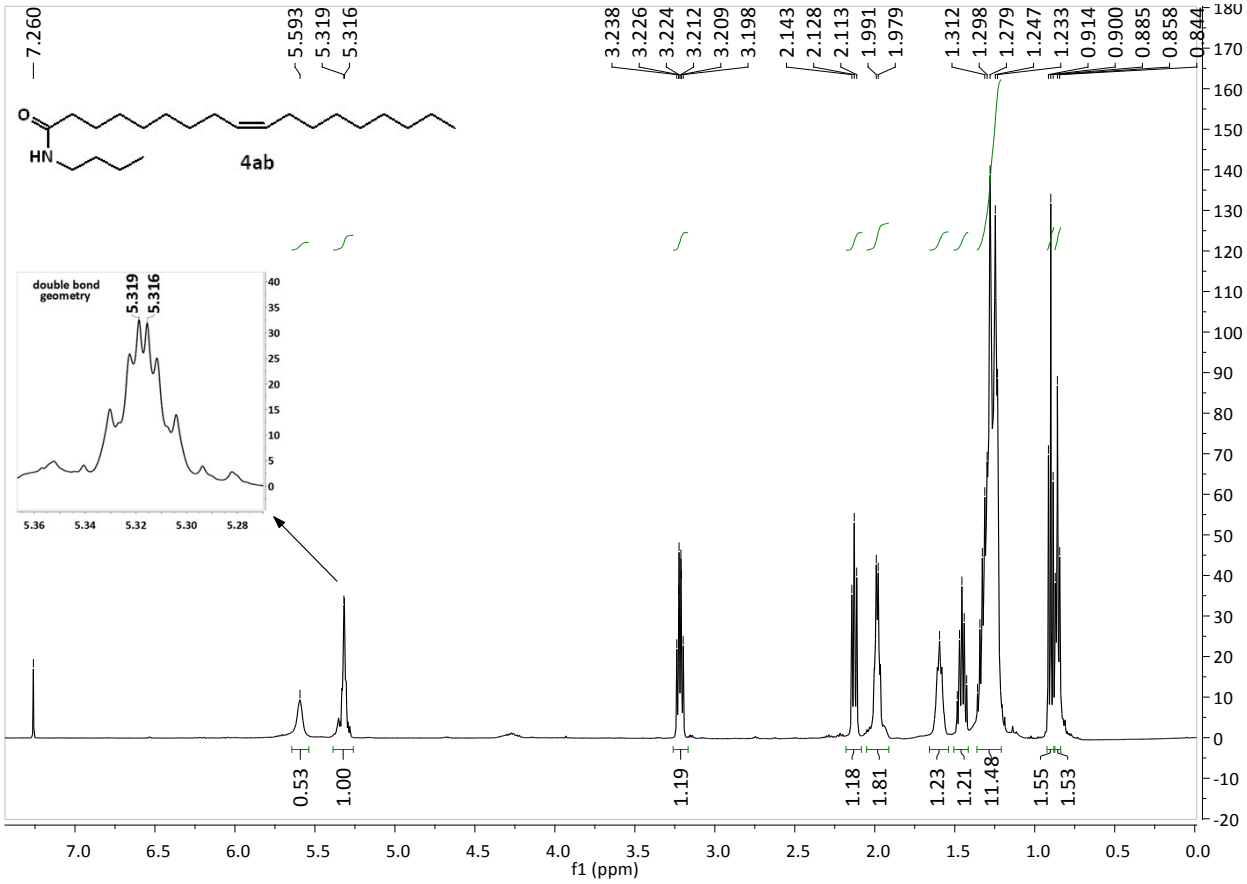
4.2 ^{1}H and ^{13}C NMR Spectra of Titled Compounds.

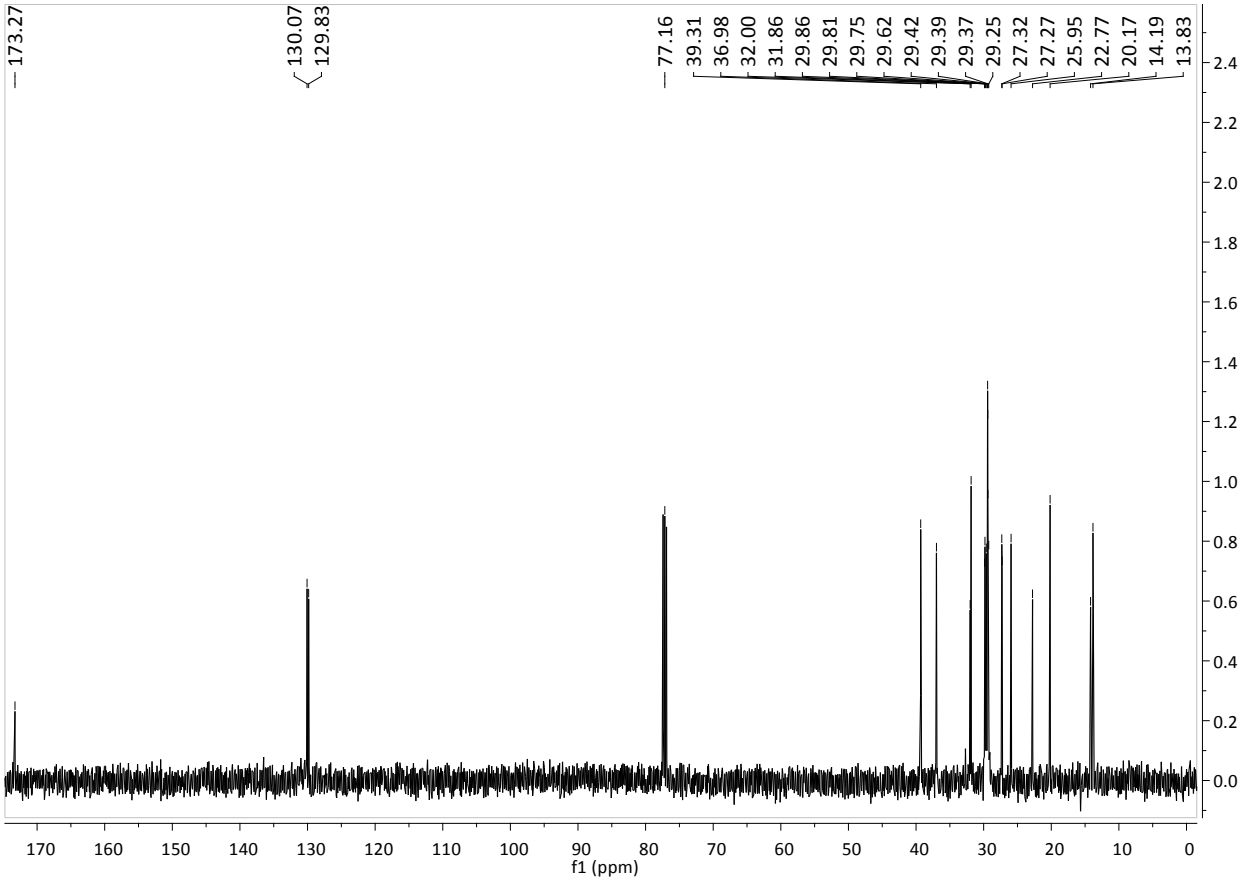


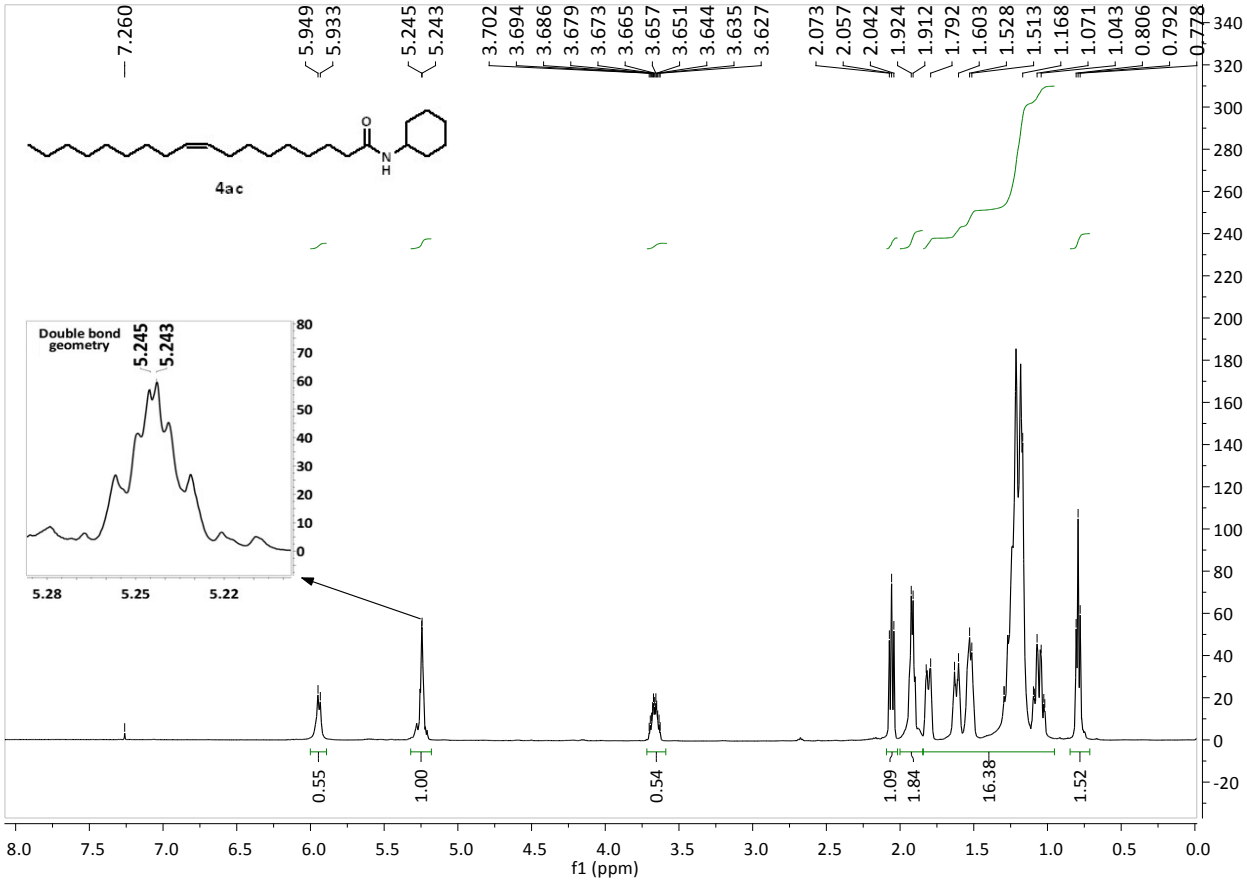


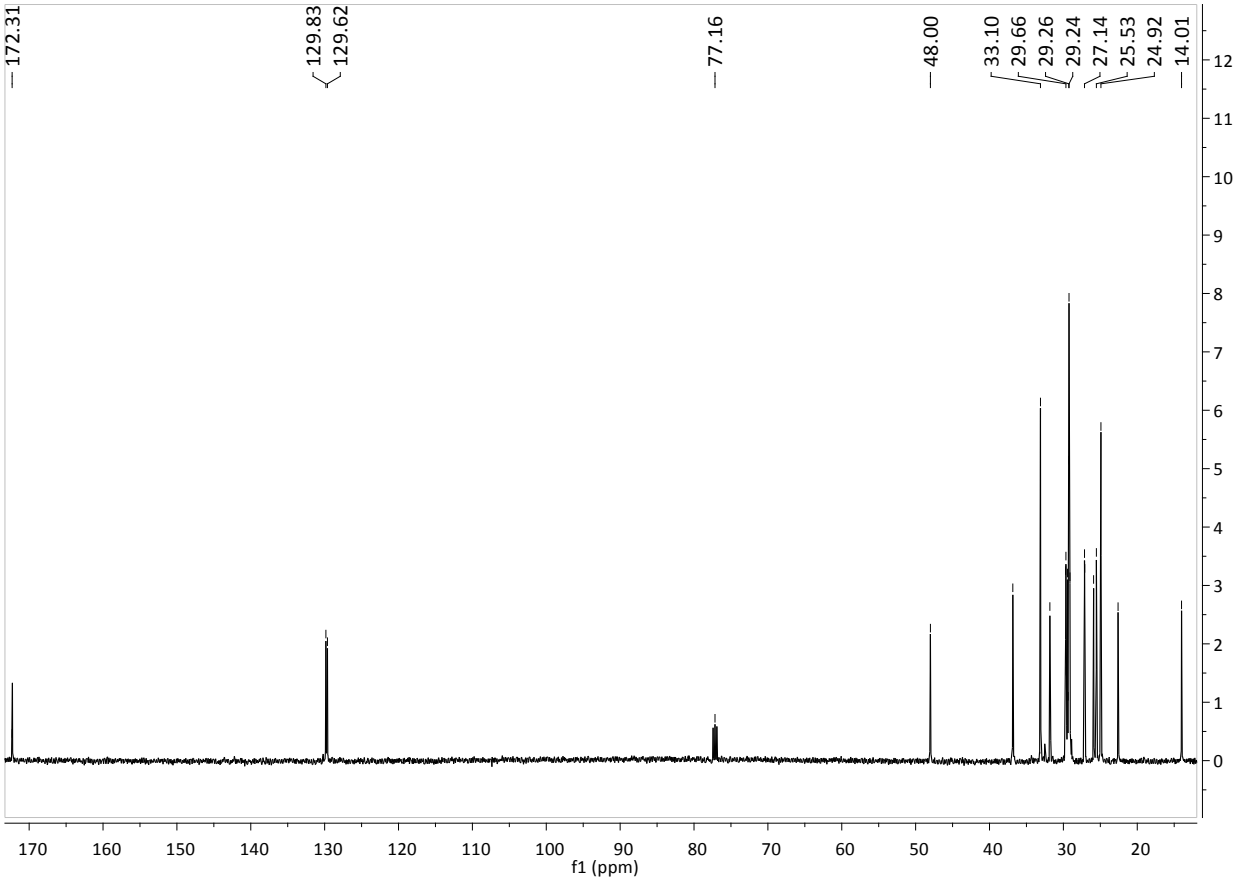

7.260
5.376
5.352
5.343
5.341
5.337
5.326
3.252
3.238
3.225
3.212
2.157
2.142
2.126
2.013
2.000
1.988
1.299
1.288
1.267
1.254
0.893
0.880
0.865
O
HN
4aa
Double bond geometry
5.376
5.341
5.337
2.48
1.00
1.04
3.69
1.74
1.10
21.23
2.90
f1 (ppm)

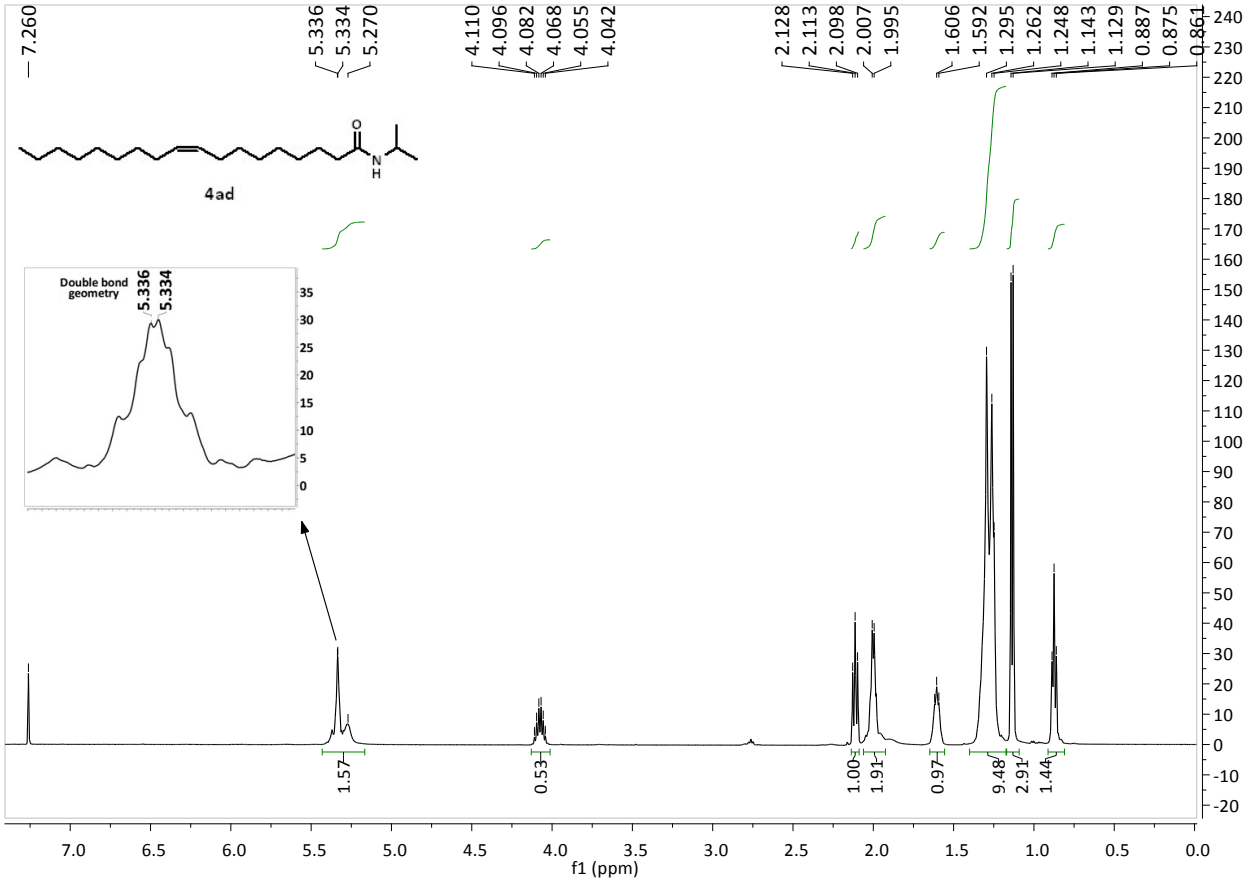

173.11
130.14
130.13
129.93
129.90
77.16
32.06
29.93
29.89
29.87
29.68
29.60
29.47
29.42
29.30
27.38
27.35
27.33
27.08
22.83
14.25
f1 (ppm)

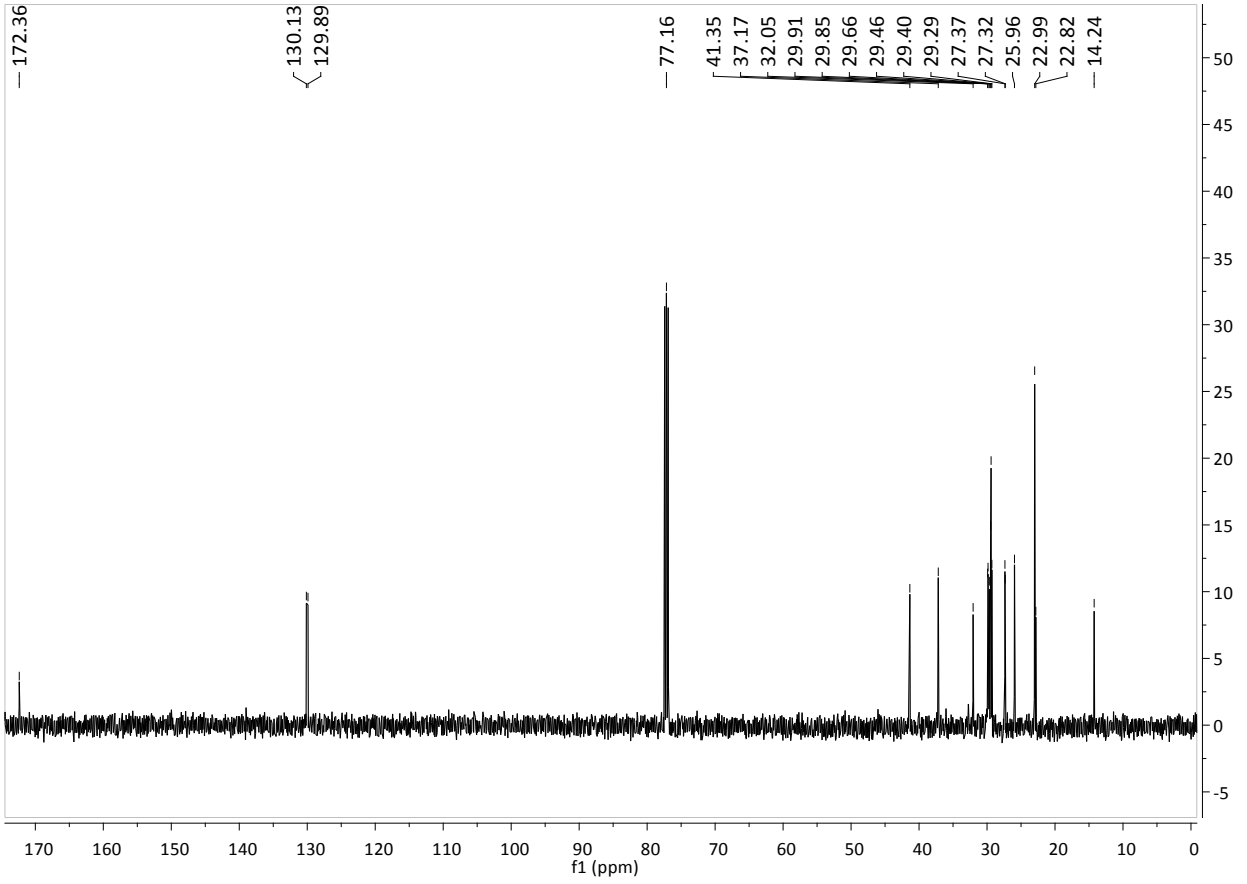

7.260
5.593
5.319
5.316
3.238
3.226
3.224
3.212
3.209
3.198
2.143
2.128
2.113
1.991
1.979
1.312
1.298
1.279
1.247
1.233
0.914
0.900
0.885
0.858
0.844
4ab
double bond geometry
5.319
5.316
0.53
1.00
1.19
1.18
1.81
1.23
1.21
11.48
1.55
1.53
f1 (ppm)

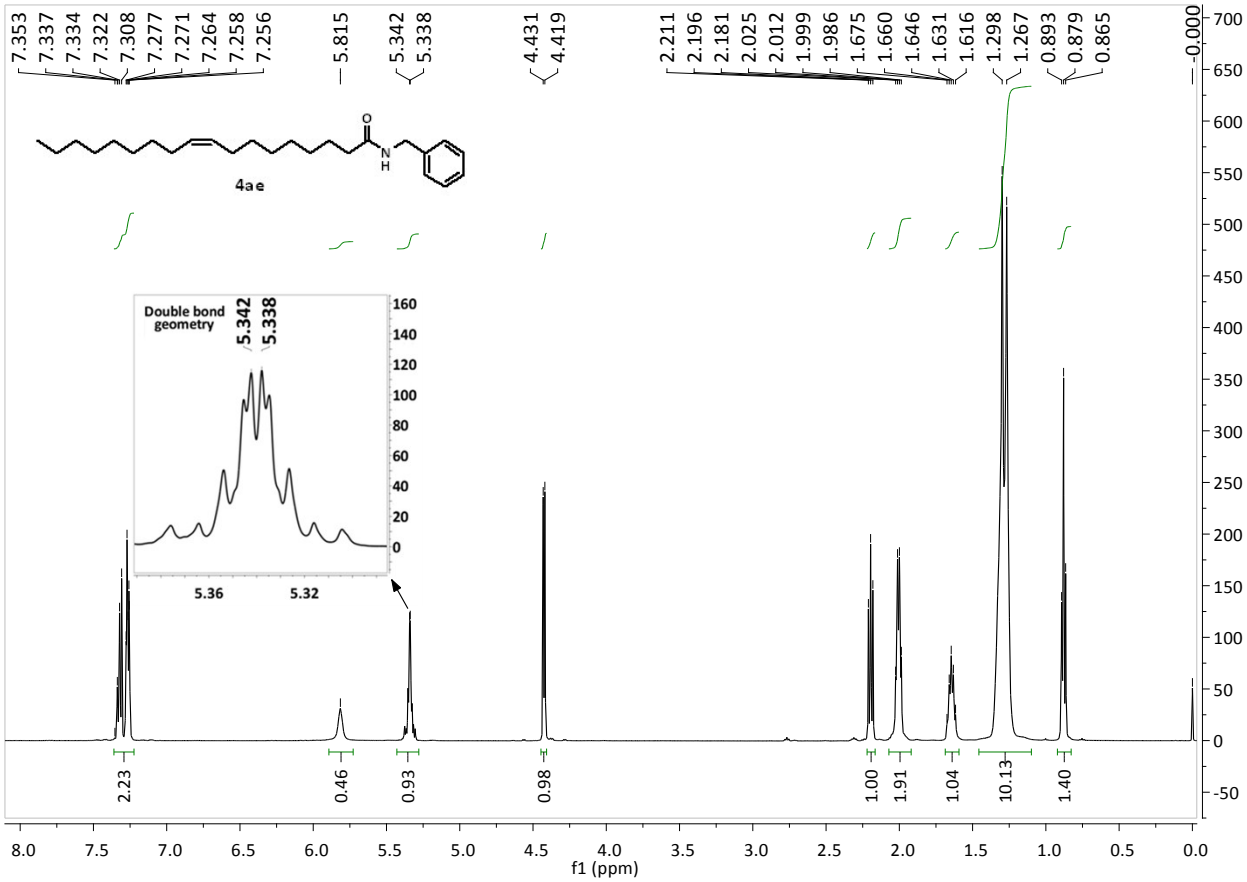

173.27
130.07
129.83
77.16
39.31
36.98
32.00
31.86
29.86
29.81
29.75
29.62
29.42
29.39
29.37
29.25
27.32
27.27
25.95
22.77
20.17
14.19
13.83
f1 (ppm)


7.260
5.949
5.933
5.245
5.243
3.702
3.694
3.686
3.679
3.673
3.665
3.657
3.651
3.644
3.635
3.627
2.073
2.057
2.042
1.924
1.912
1.792
1.603
1.528
1.513
1.168
1.071
1.043
0.806
0.792
0.778
4ac
Double bond geometry
5.245
5.243
0.55
1.00
0.54
1.09
1.84
16.38
1.52
f1 (ppm)

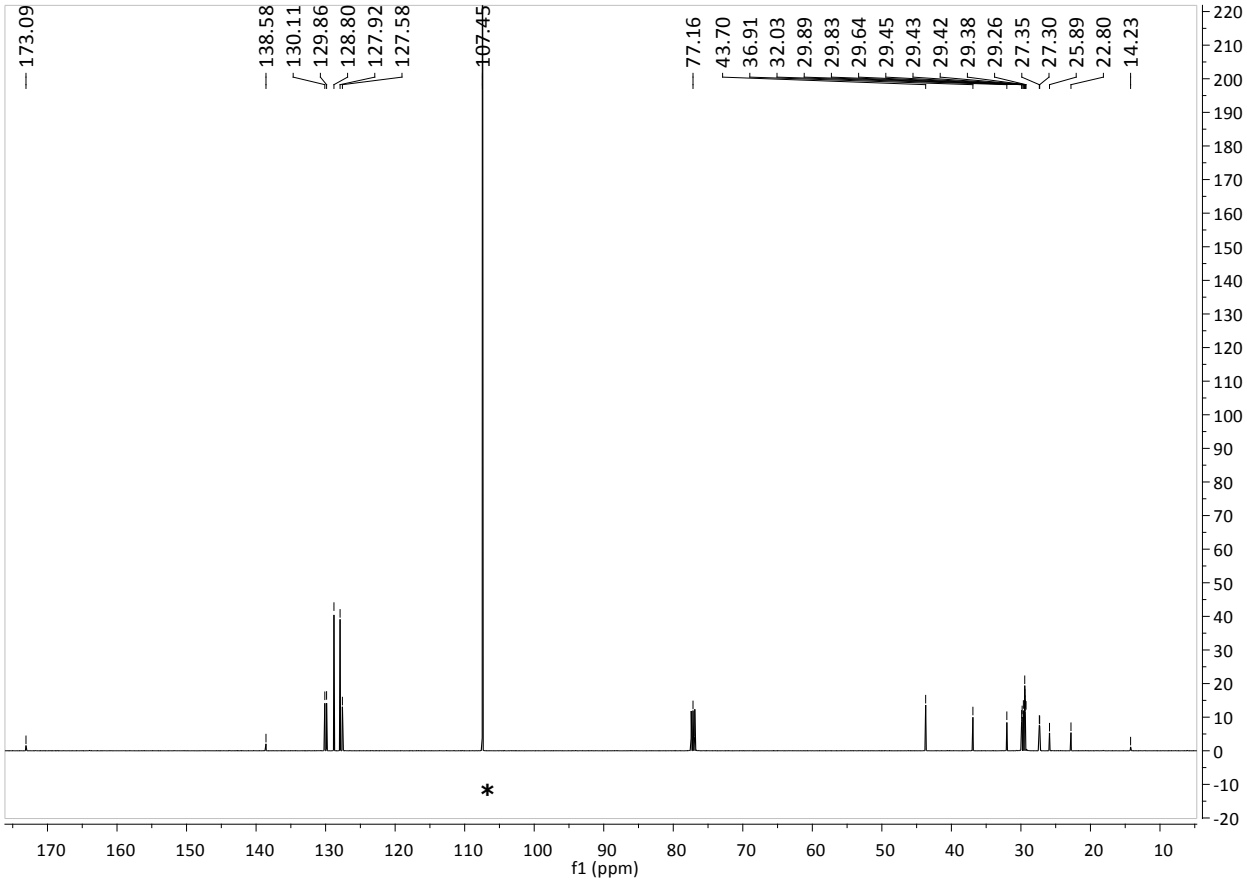

172.31
129.83
129.62
77.16
48.00
33.10
29.66
29.26
29.24
27.14
25.53
24.92
14.01
f1 (ppm)

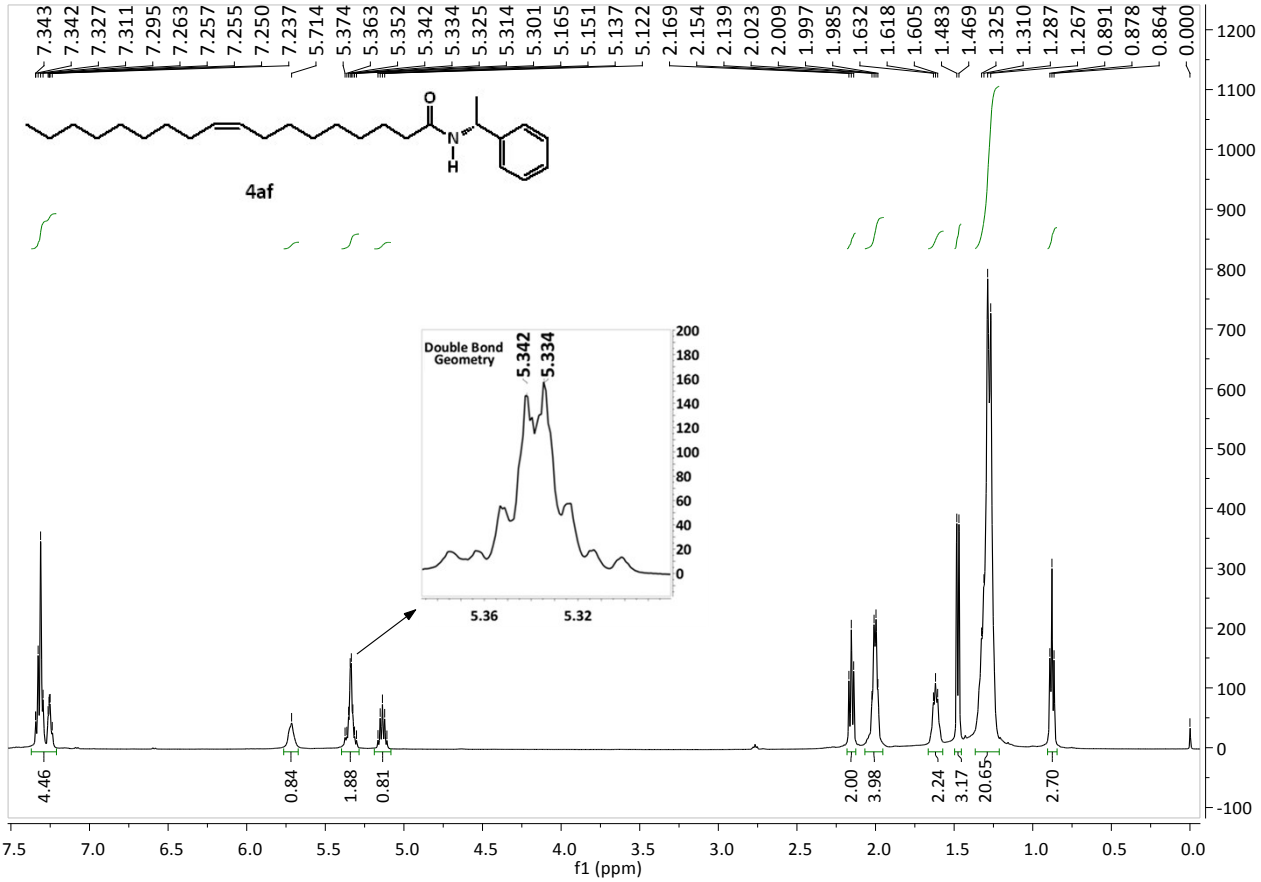

7.260
5.336
5.334
5.270
4.110
4.096
4.082
4.068
4.055
4.042
2.128
2.113
2.098
2.007
1.995
1.606
1.592
1.295
1.262
1.248
1.143
1.129
0.887
0.875
0.861
4ad
Double bond geometry
5.336
5.334
1.57
0.53
1.00
1.91
0.97
9.48
2.91
1.44
f1 (ppm)


172.36
130.13
129.89
77.16
41.35
37.17
32.05
29.91
29.85
29.66
29.46
29.40
29.29
27.37
27.32
25.96
22.99
22.82
14.24
f1 (ppm)



*signal due to the transmitter

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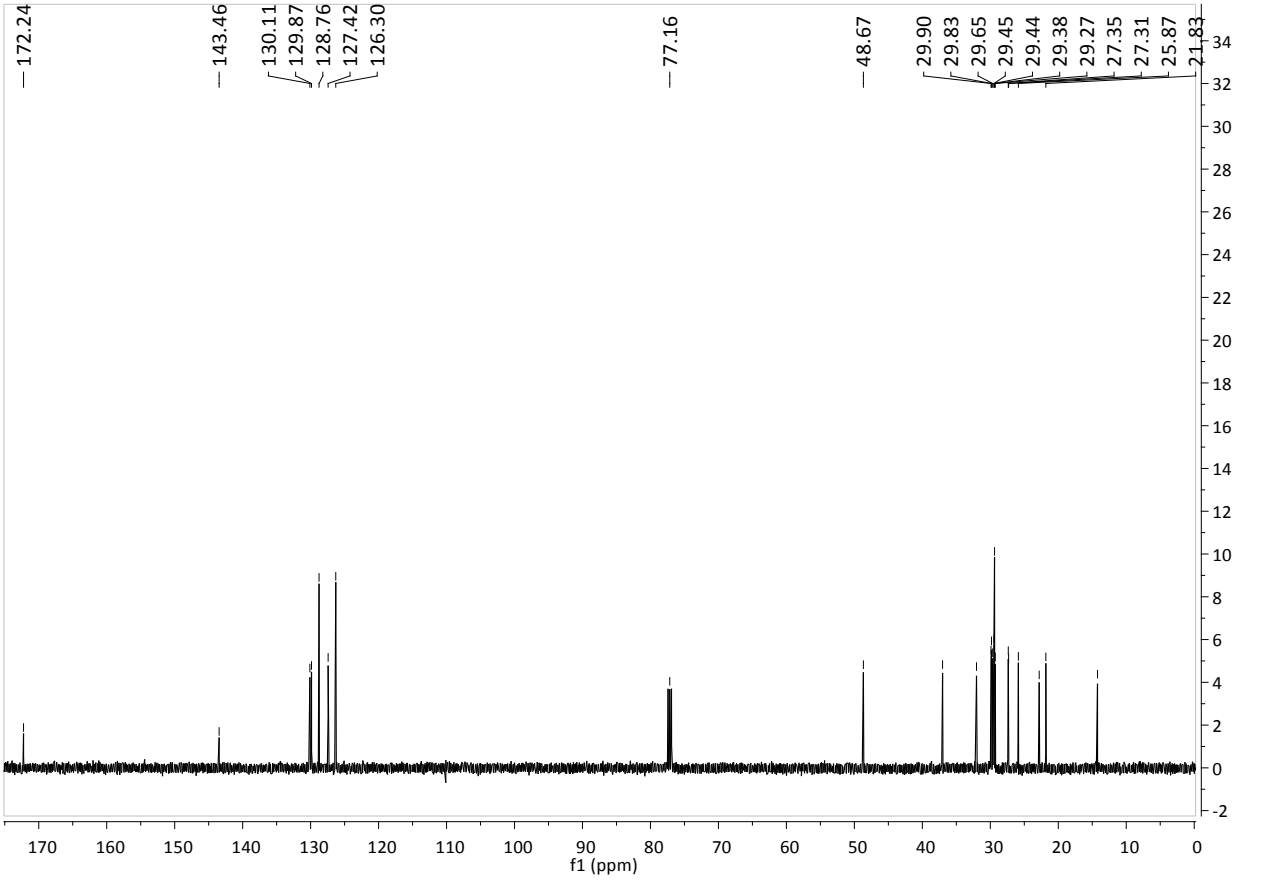
7.342
7.327
7.311
7.295
7.263
7.257
7.255
7.250
7.237
5.714
5.374
5.363
5.352
5.342
5.334
5.325
5.314
5.301
5.165
5.151
5.137
5.122
2.169
2.154
2.139
2.023
2.009
1.997
1.985
1.632
1.618
1.605
1.483
1.469
1.325
1.310
1.287
1.267
0.891
0.878
0.864
0.000
O
N
H
4af
Double Bond Geometry
5.342
5.334
5.36
5.32
4.46
0.84
1.88
0.81
2.00
3.98
2.24
3.17
20.65
2.70
f1 (ppm)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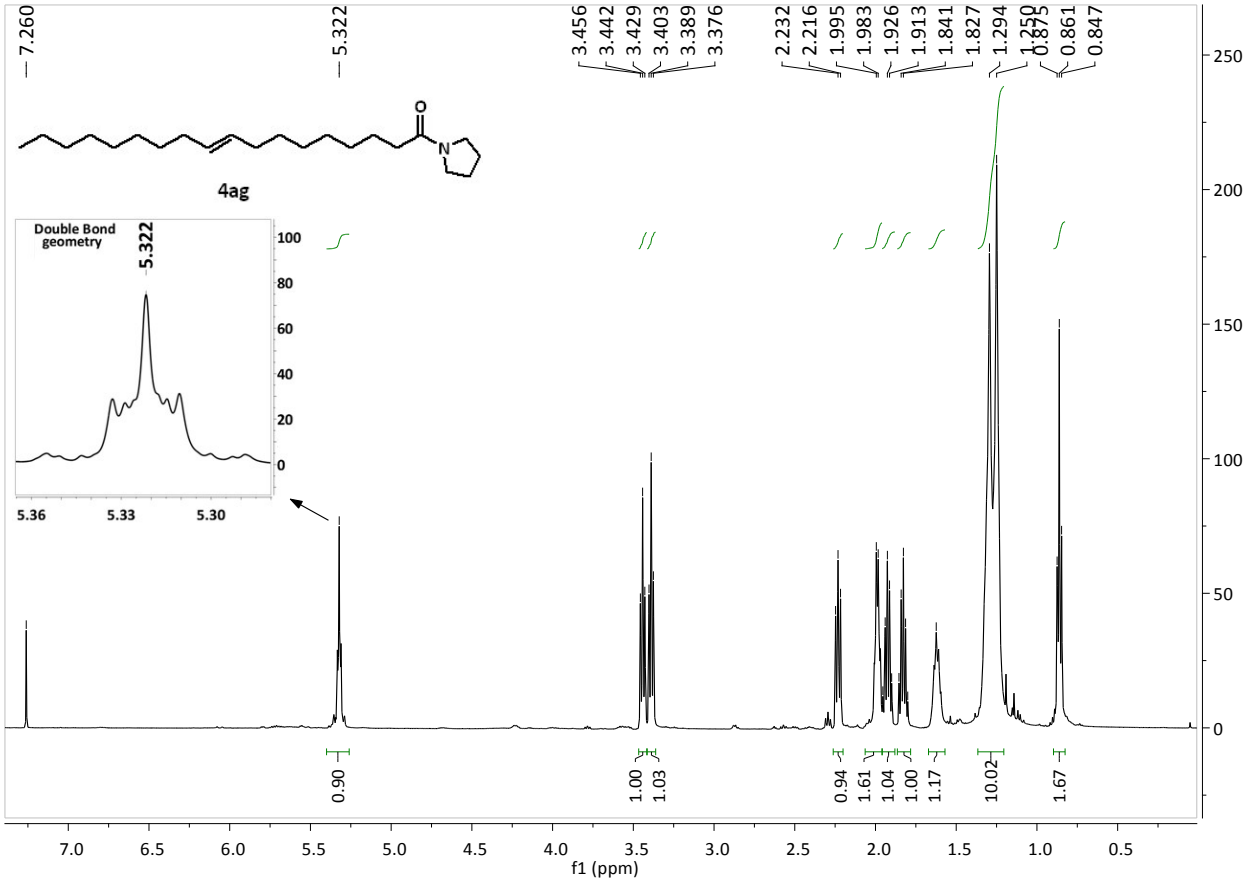
143.46
130.11
129.87
128.76
127.42
126.30
77.16
48.67
29.90
29.83
29.65
29.45
29.44
29.38
29.27
27.35
27.31
25.87
21.83
f1 (ppm)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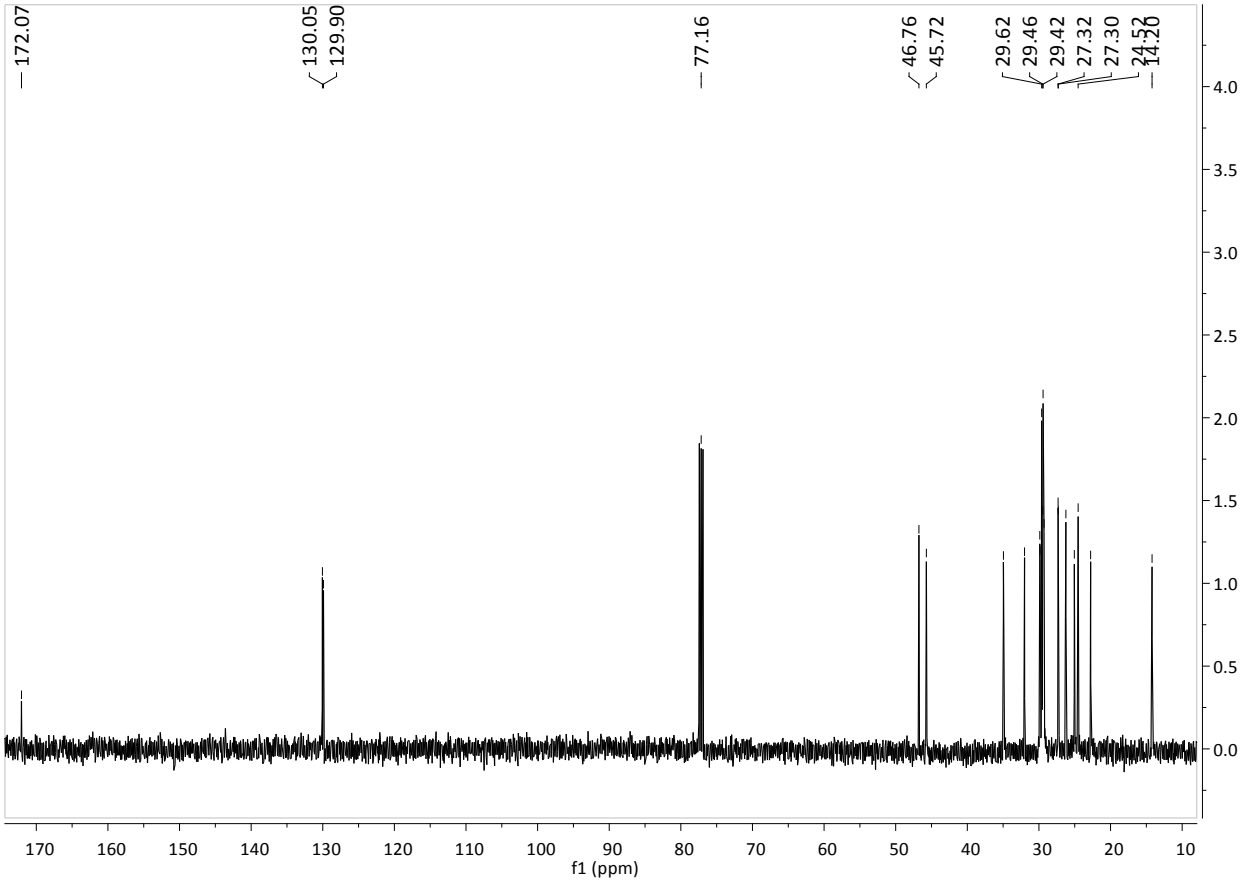
5.322
3.456
3.442
3.429
3.403
3.389
3.376
2.232
2.216
1.995
1.983
1.926
1.913
1.841
1.827
1.294
1.250
0.875
0.861
0.847
4ag
Double Bond geometry
5.322
0.90
1.00
1.03
0.94
1.61
1.04
1.00
1.17
10.02
1.67
f1 (ppm)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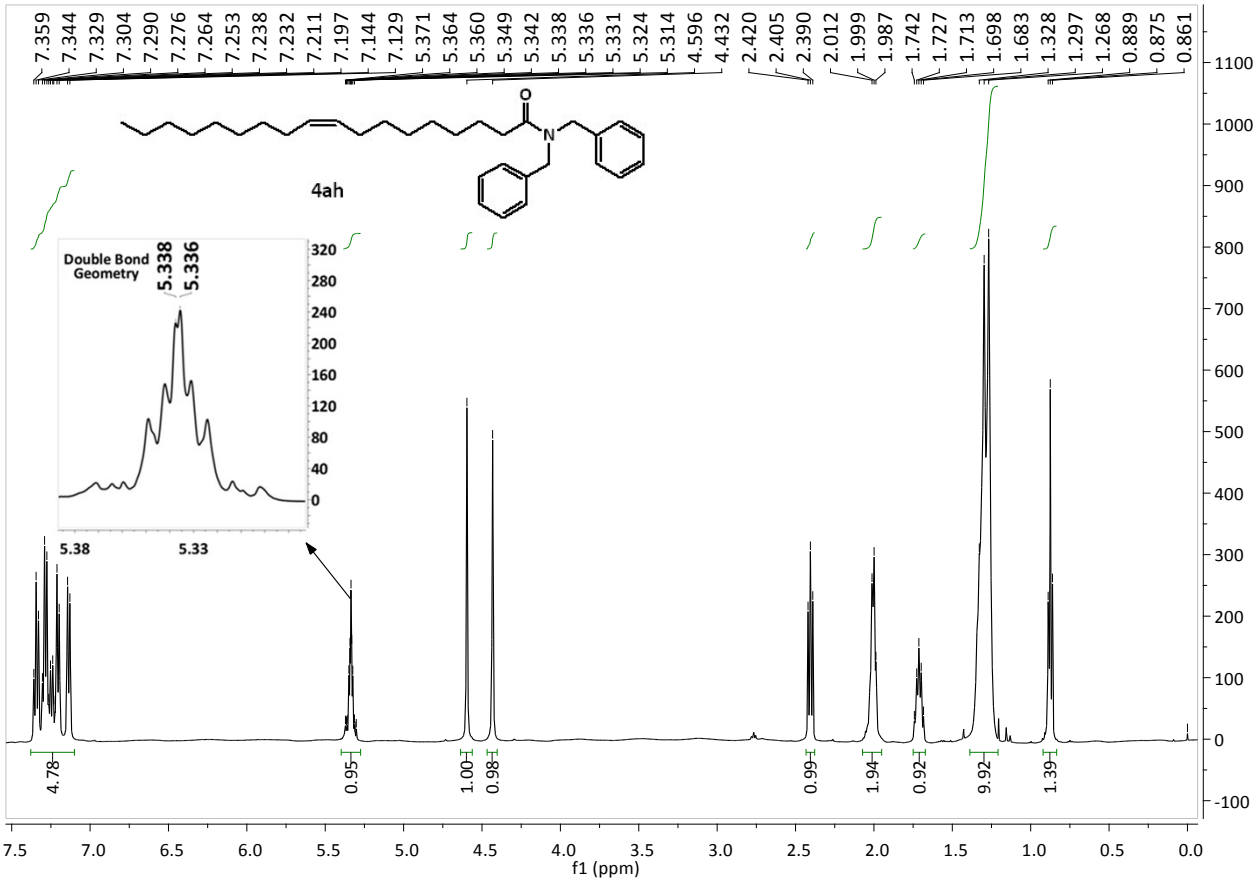
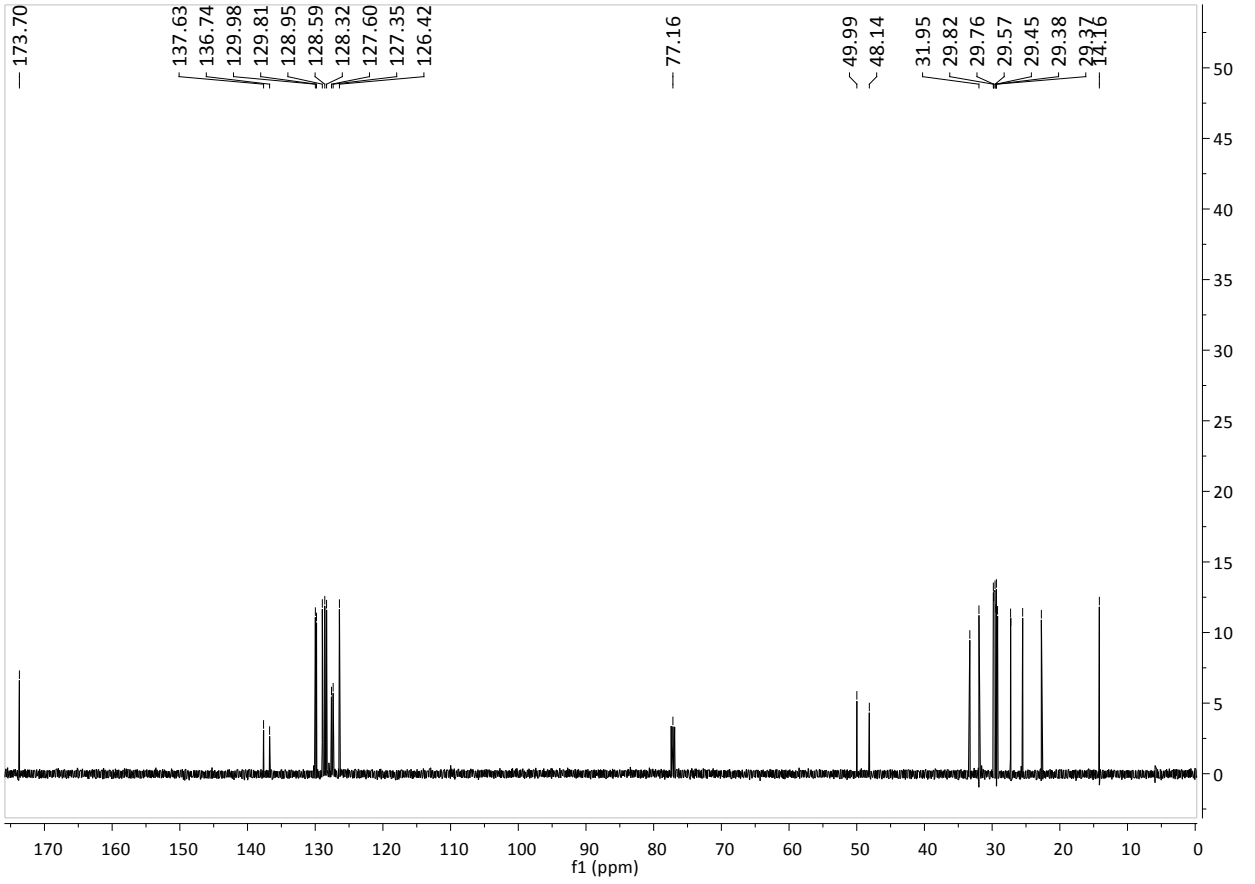
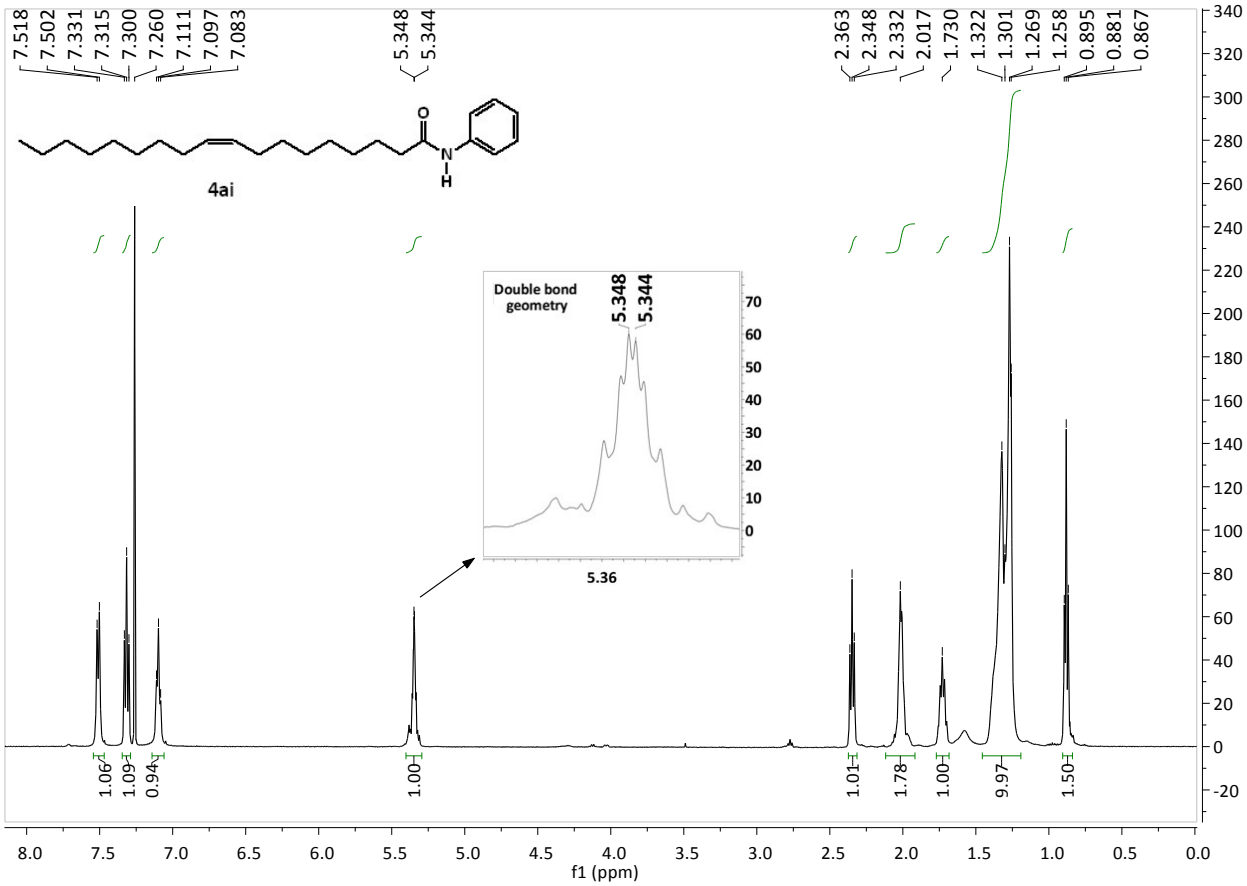
130.05
129.90
77.16
46.76
45.72
29.62
29.46
29.42
27.32
27.30
24.52
14.20
f1 (ppm)


7.359
7.344
7.329
7.304
7.290
7.276
7.264
7.253
7.238
7.232
7.211
7.197
7.144
7.129
5.371
5.364
5.360
5.349
5.342
5.338
5.336
5.331
5.324
5.314
4.596
4.432
2.420
2.405
2.390
2.012
1.999
1.987
1.742
1.727
1.713
1.698
1.683
1.328
1.297
1.268
0.889
0.875
0.861
4ah
Double Bond Geometry
5.338
5.336
5.38
5.33
4.78
0.95
1.00
0.98
0.99
1.94
0.92
9.92
1.39
f1 (ppm)


173.70
137.63
136.74
129.98
129.81
128.95
128.59
128.32
127.60
127.35
126.42
77.16
49.99
48.14
31.95
29.82
29.76
29.57
29.45
29.38
29.37
14.16
f1 (ppm)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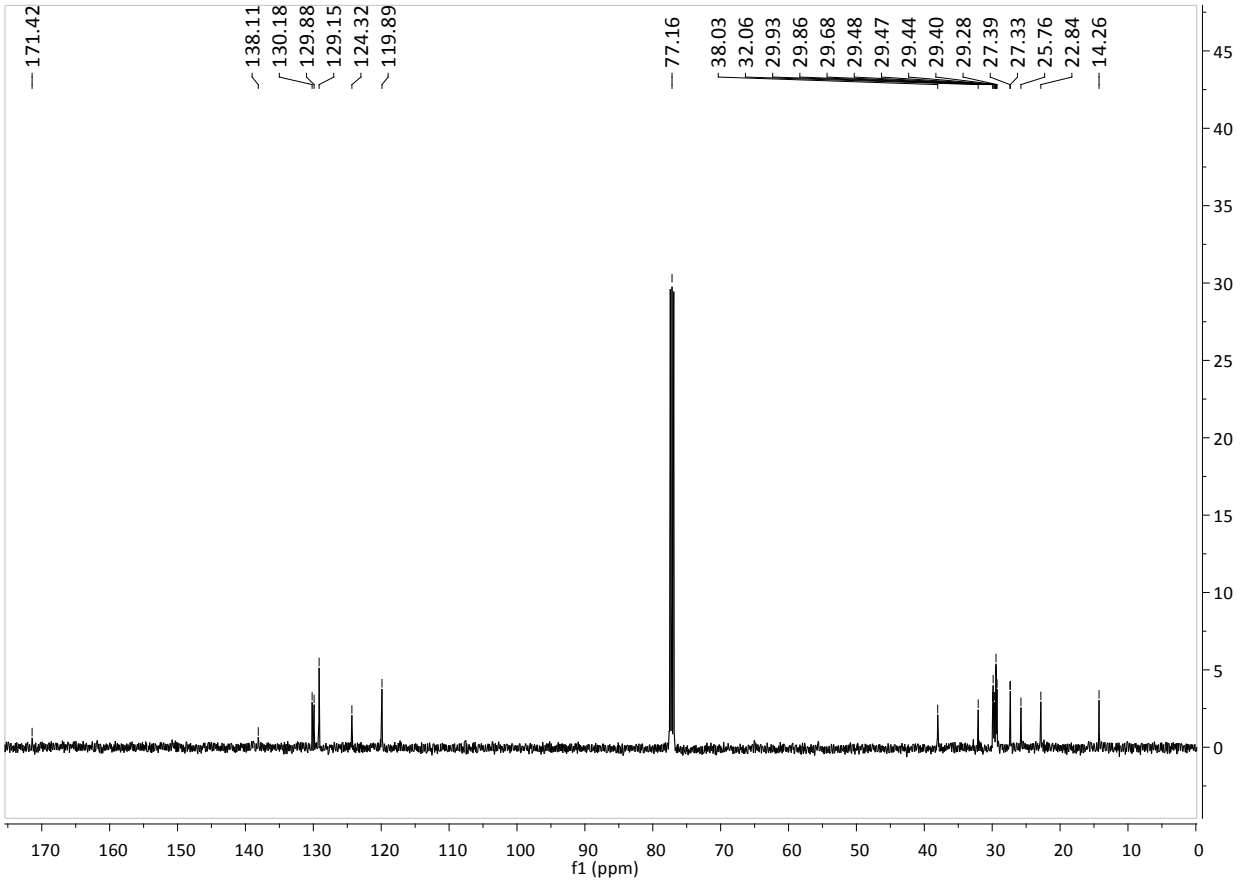
7.502
7.331
7.315
7.300
7.260
7.111
7.097
7.083
5.348
5.344
2.363
2.348
2.332
2.017
1.730
1.322
1.301
1.269
1.258
0.895
0.881
0.867
4ai
Double bond geometry
5.348
5.344
5.36
1.06
1.09
0.94
1.00
1.01
1.78
1.00
9.97
1.50
f1 (ppm)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138.11
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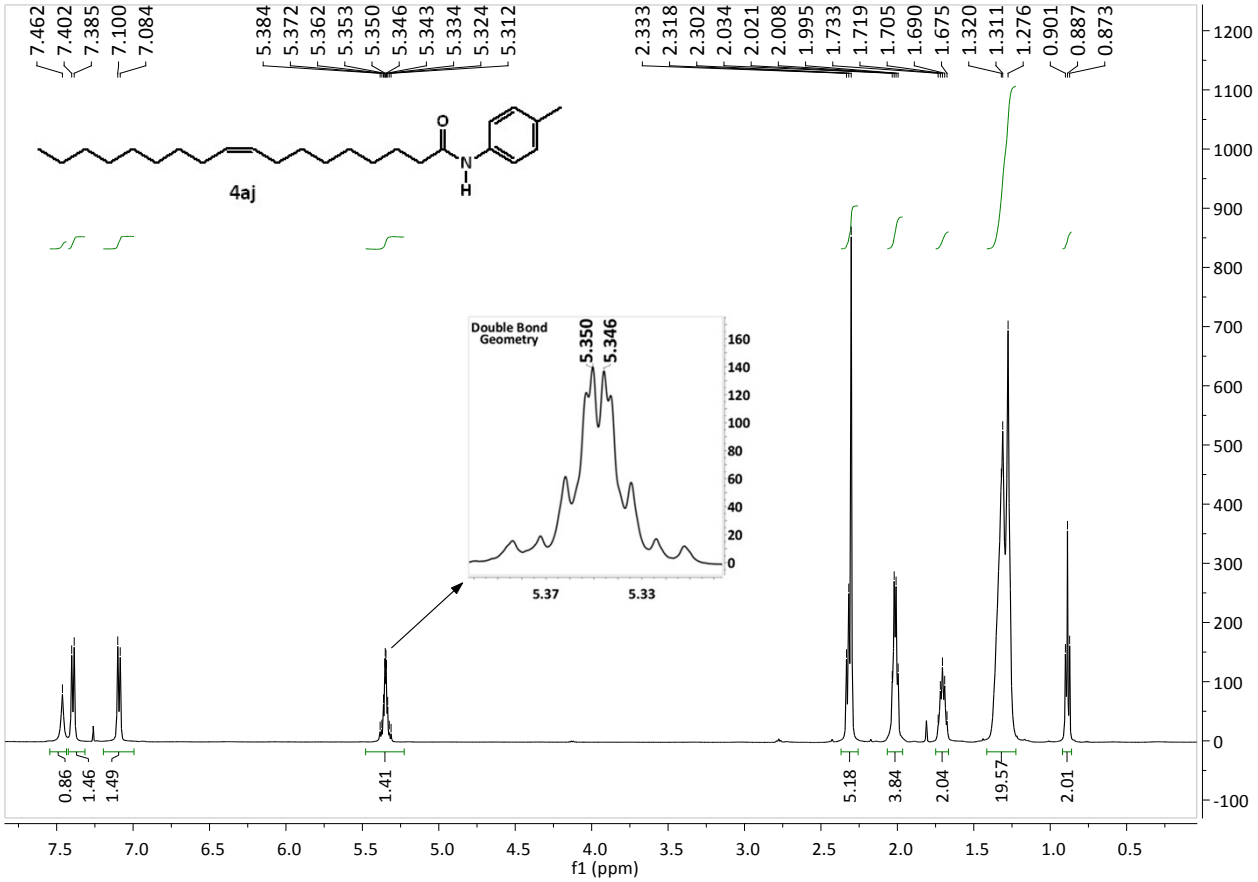
129.88
129.15
124.32
119.89
77.16
38.03
32.06
29.93
29.86
29.68
29.48
29.47
29.44
29.40
29.28
27.39
27.33
25.76
22.84
14.26
f1 (ppm)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7.402
7.385
7.100
7.084
5.384
5.372
5.362
5.353
5.350
5.346
5.343
5.334
5.324
5.312
2.333
2.318
2.302
2.034
2.021
2.008
1.995
1.733
1.719
1.705
1.690
1.675
1.320
1.311
1.276
0.901
0.887
0.873
4aj
Double Bond Geometry
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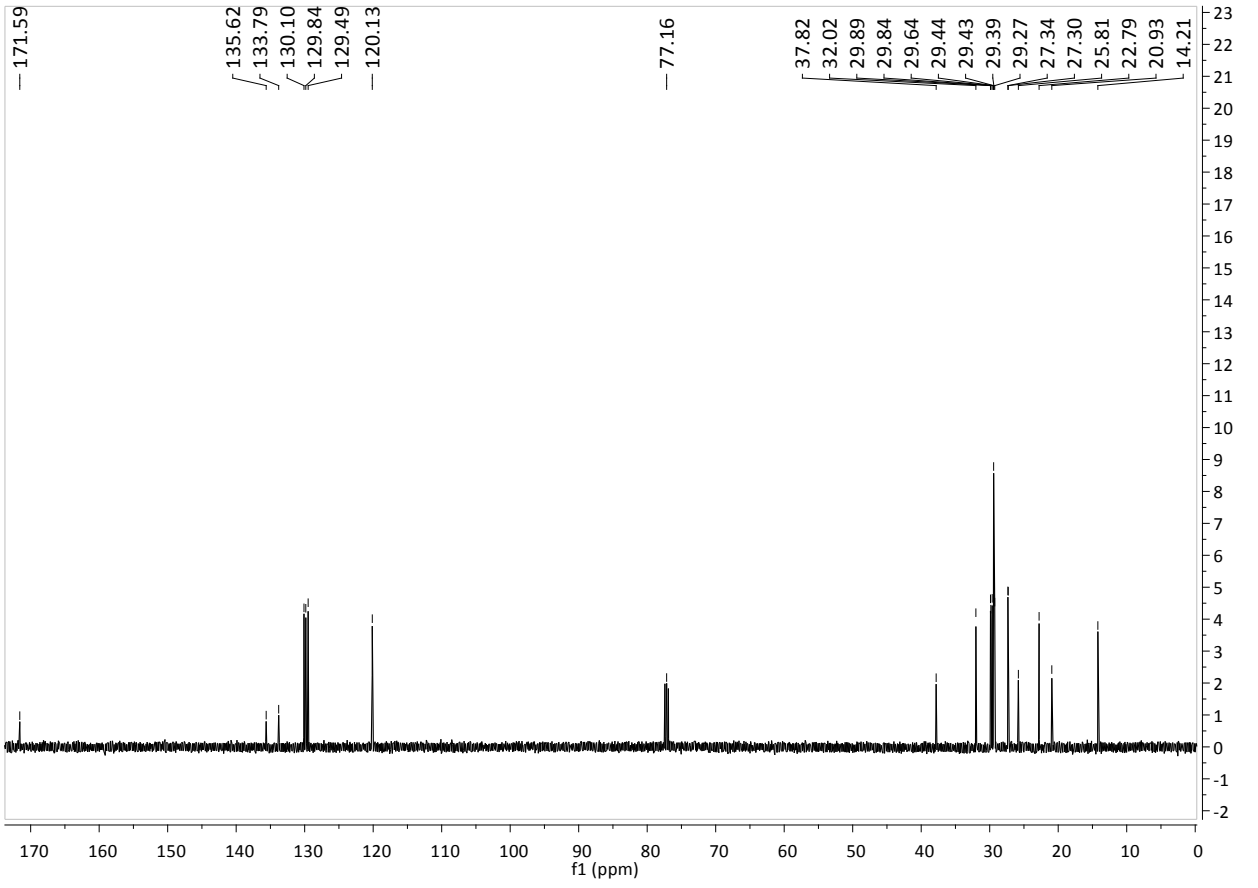
5.346
0.86
1.46
1.49
1.41
5.18
3.84
2.04
19.57
2.01
f1 (ppm)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130.10
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129.49
120.13
77.16
37.82
32.02
29.89
29.84
29.64
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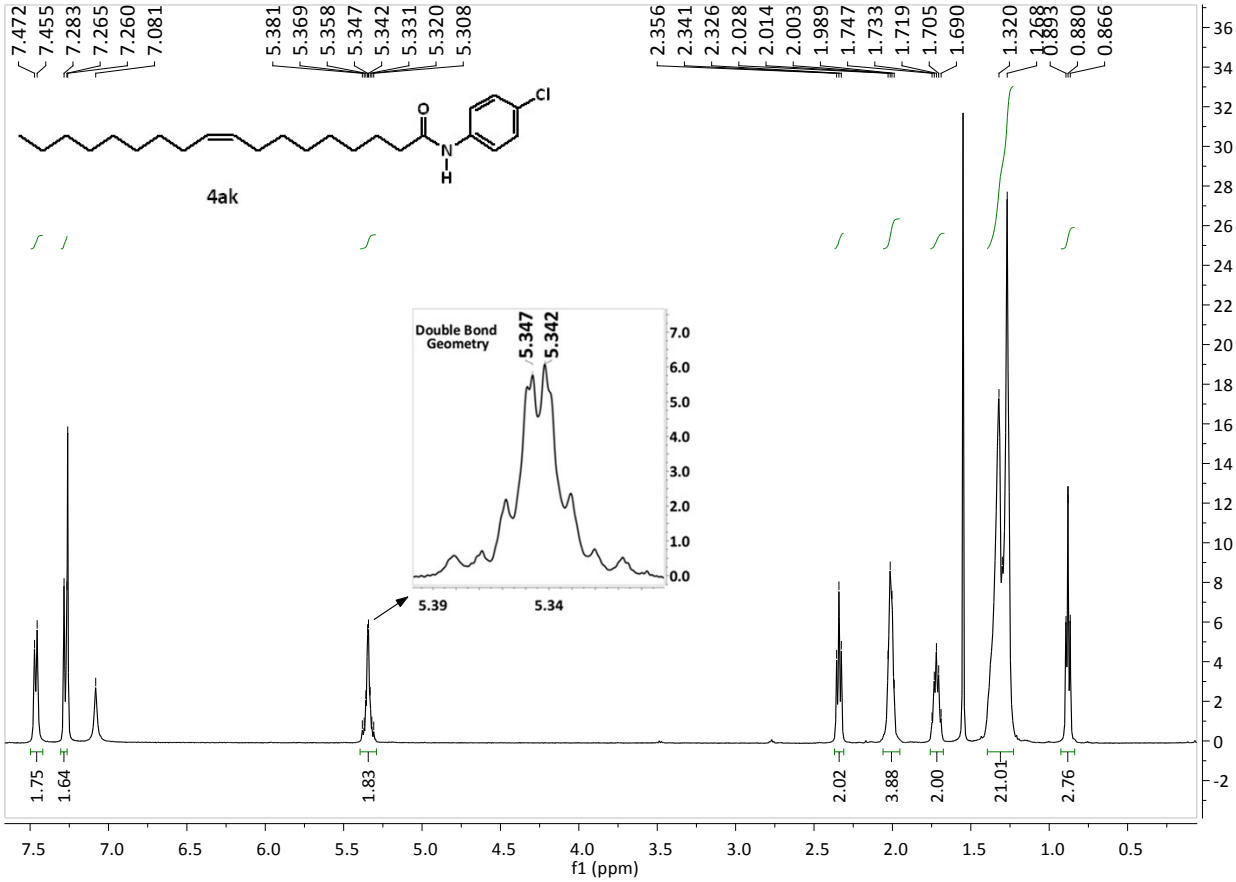
29.43
29.39
29.27
27.34
27.30
25.81
22.79
20.93
14.21
f1 (ppm)


7.472
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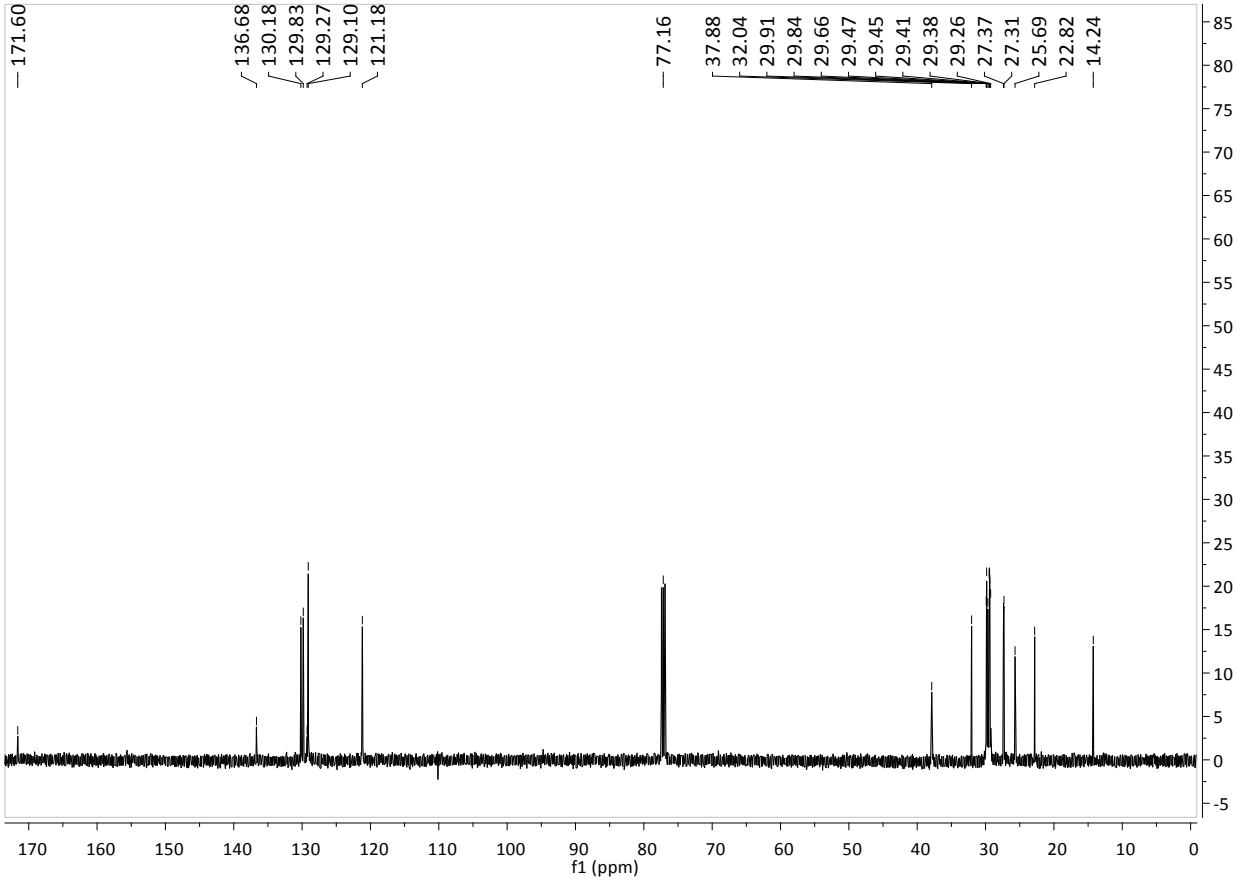
7.283
7.265
7.260
7.081
5.381
5.369
5.358
5.347
5.342
5.331
5.320
5.308
2.356
2.341
2.326
2.028
2.014
2.003
1.989
1.747
1.733
1.719
1.705
1.690
1.320
1.268
0.893
0.880
0.866
Cl
O
N
H
4ak
Double Bond Geometry
5.347
5.342
5.39
5.34
1.75
1.64
1.83
2.02
3.88
2.00
21.01
2.76
f1 (ppm)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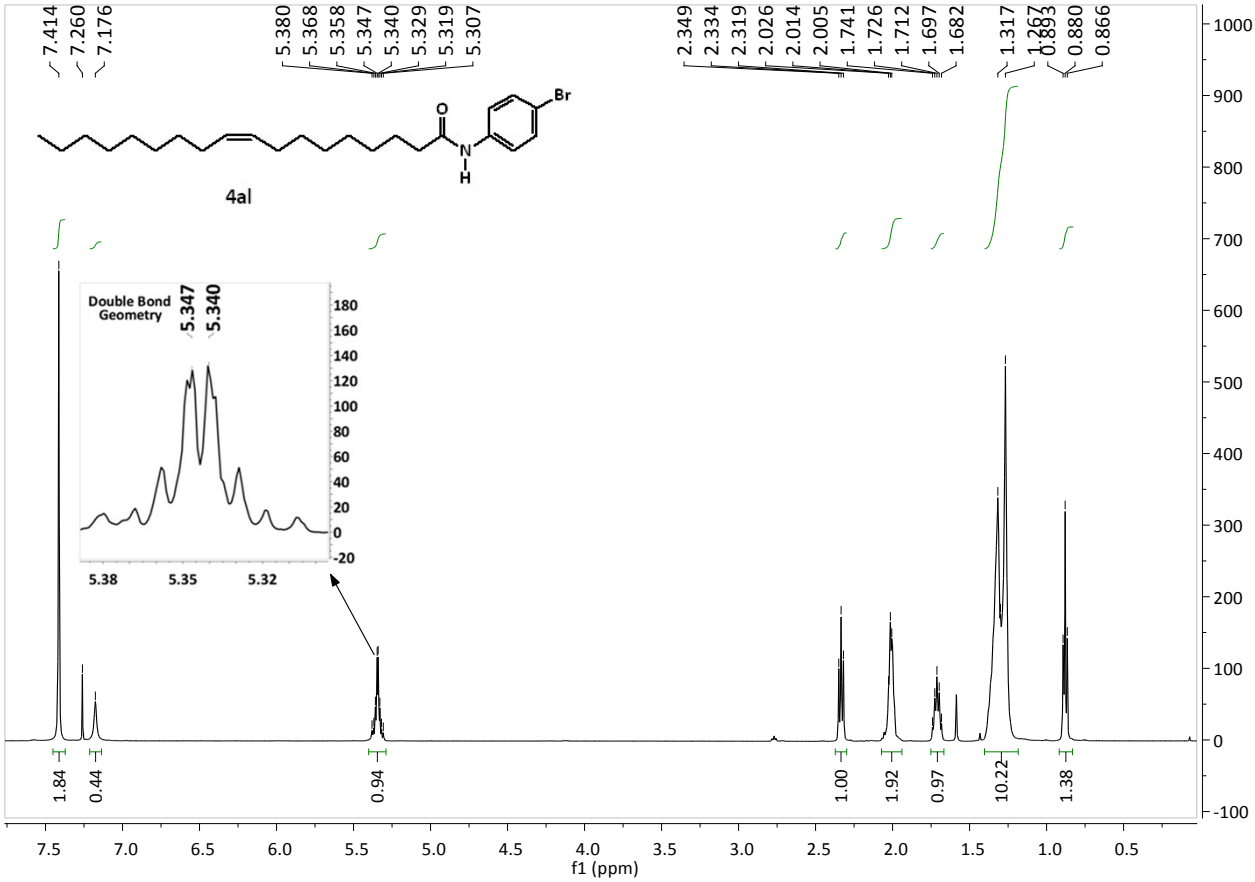
136.68
130.18
129.83
129.27
129.10
121.18
77.16
37.88
32.04
29.91
29.84
29.66
29.47
29.45
29.41
29.38
29.26
27.37
27.31
25.69
22.82
14.24
f1 (ppm)


7.414
7.260
7.176
5.380
5.368
5.358
5.347
5.340
5.329
5.319
5.307
2.349
2.334
2.319
2.026
2.014
2.005
1.741
1.726
1.712
1.697
1.682
1.317
1.267
0.893
0.880
0.866
Br
O
N
H
4al
Double Bond Geometry
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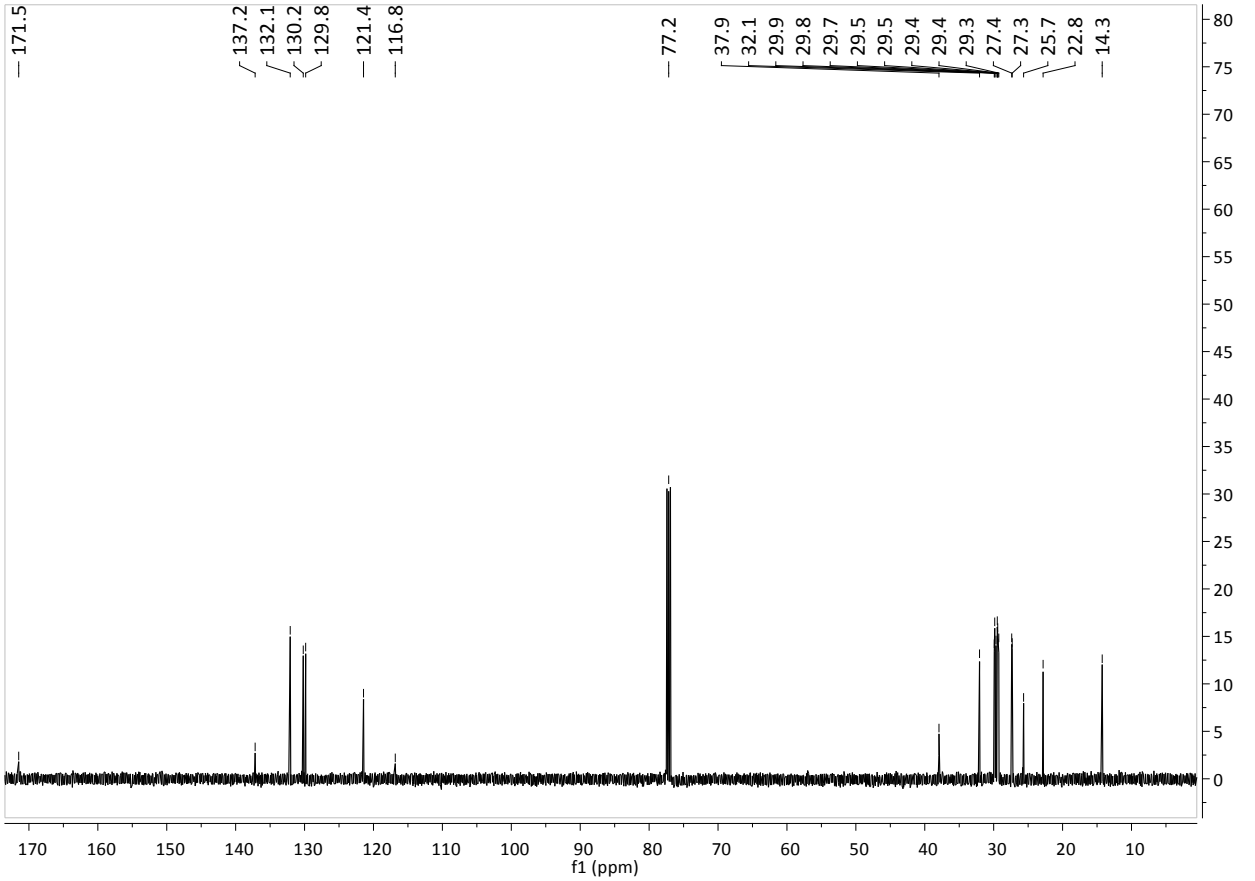
5.340
1.84
0.44
0.94
1.00
1.92
0.97
10.22
1.38
f1 (ppm)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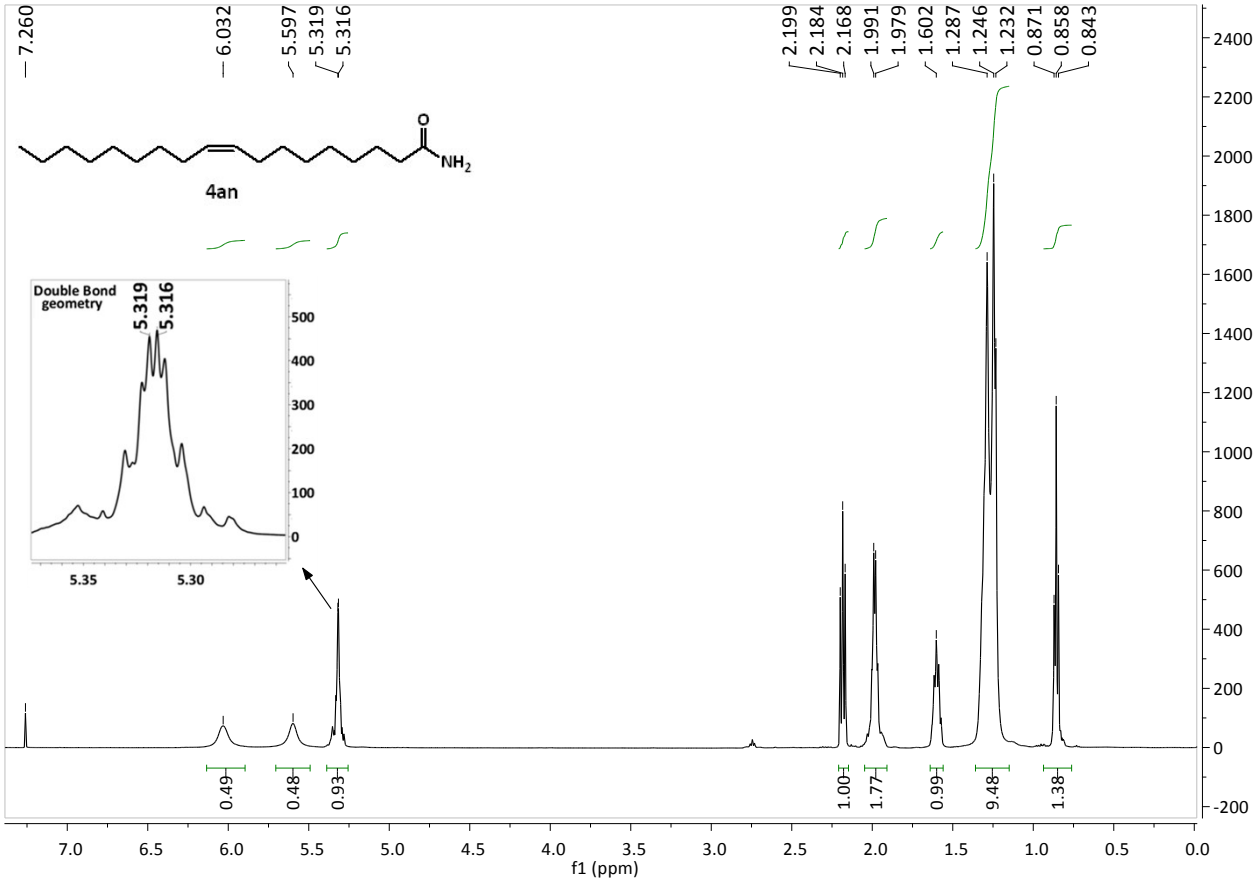
137.2
132.1
130.2
129.8
121.4
116.8
77.2
37.9
32.1
29.9
29.8
29.7
29.5
29.5
29.4
29.4
29.3
27.4
27.3
25.7
22.8
14.3
f1 (ppm)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5.319
5.316
2.199
2.184
2.168
1.991
1.979
1.602
1.287
1.246
1.232
0.871
0.858
0.843
O
NH2
4an
Double Bond geometry
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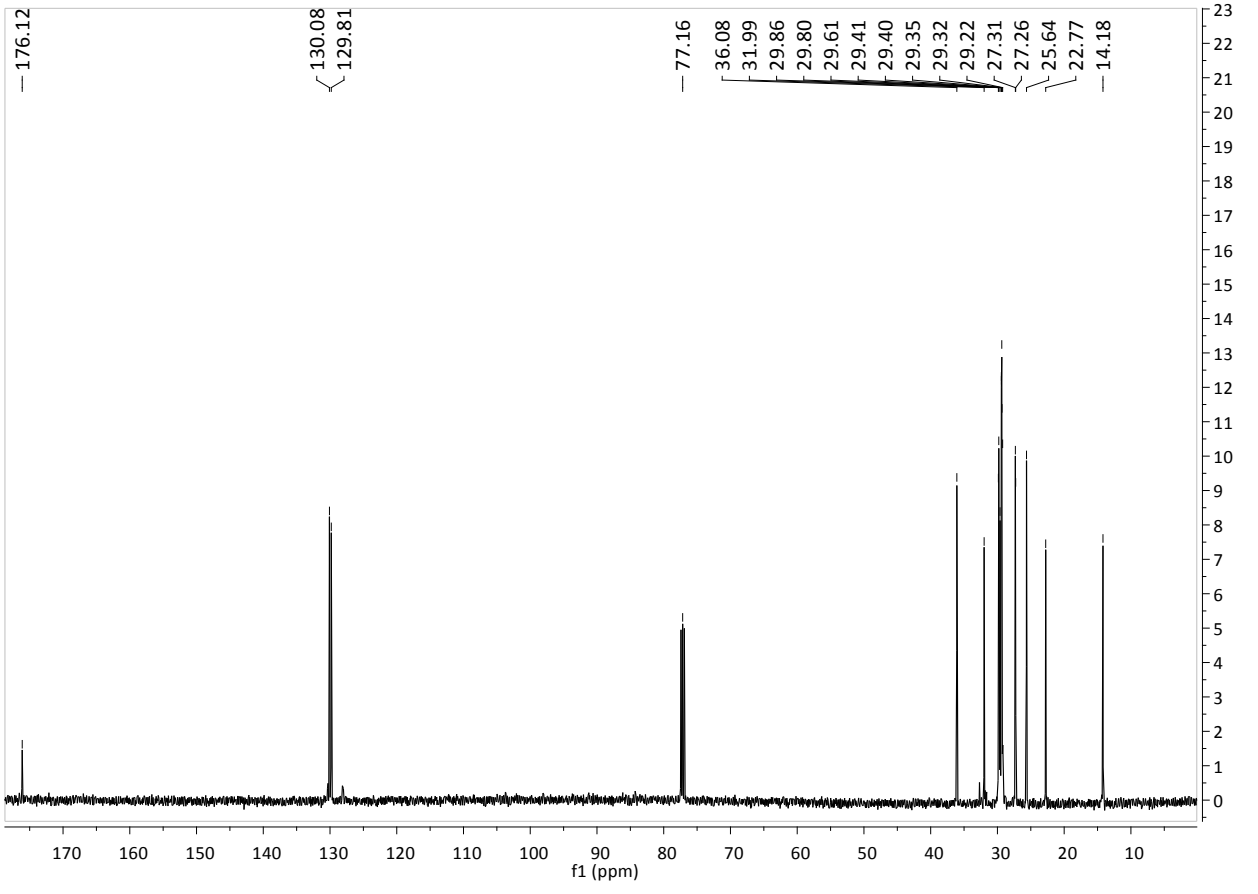
5.316
0.49
0.48
0.93
1.00
1.77
0.99
9.48
1.38
f1 (ppm)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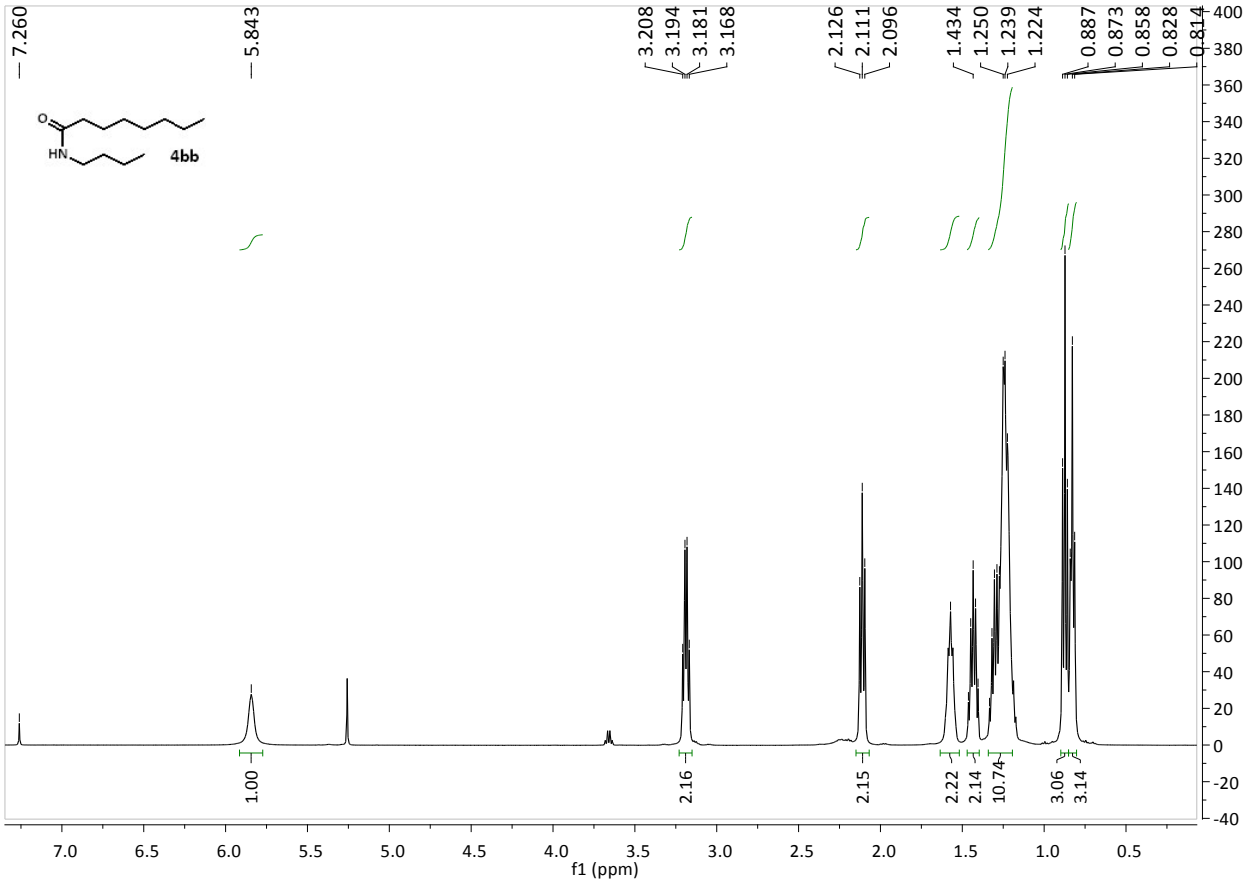
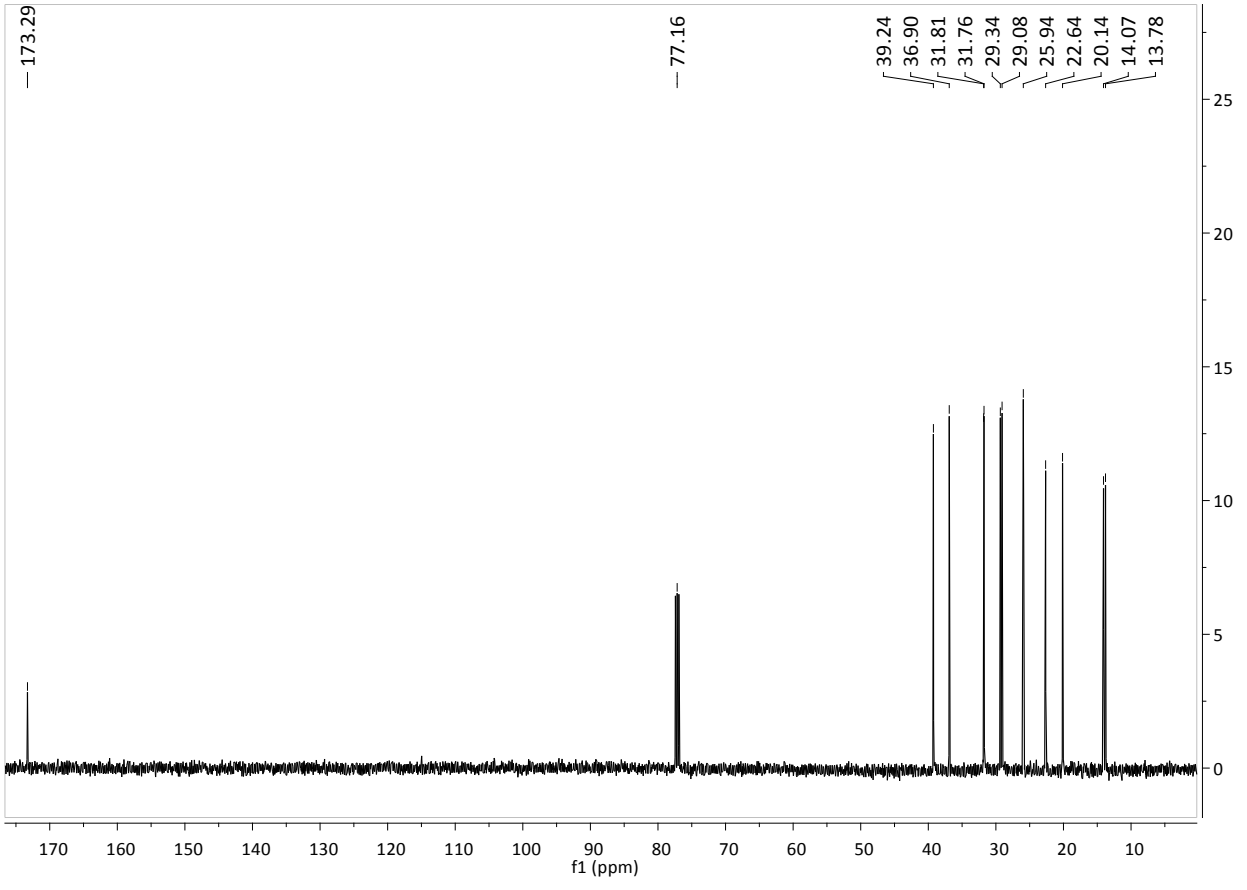
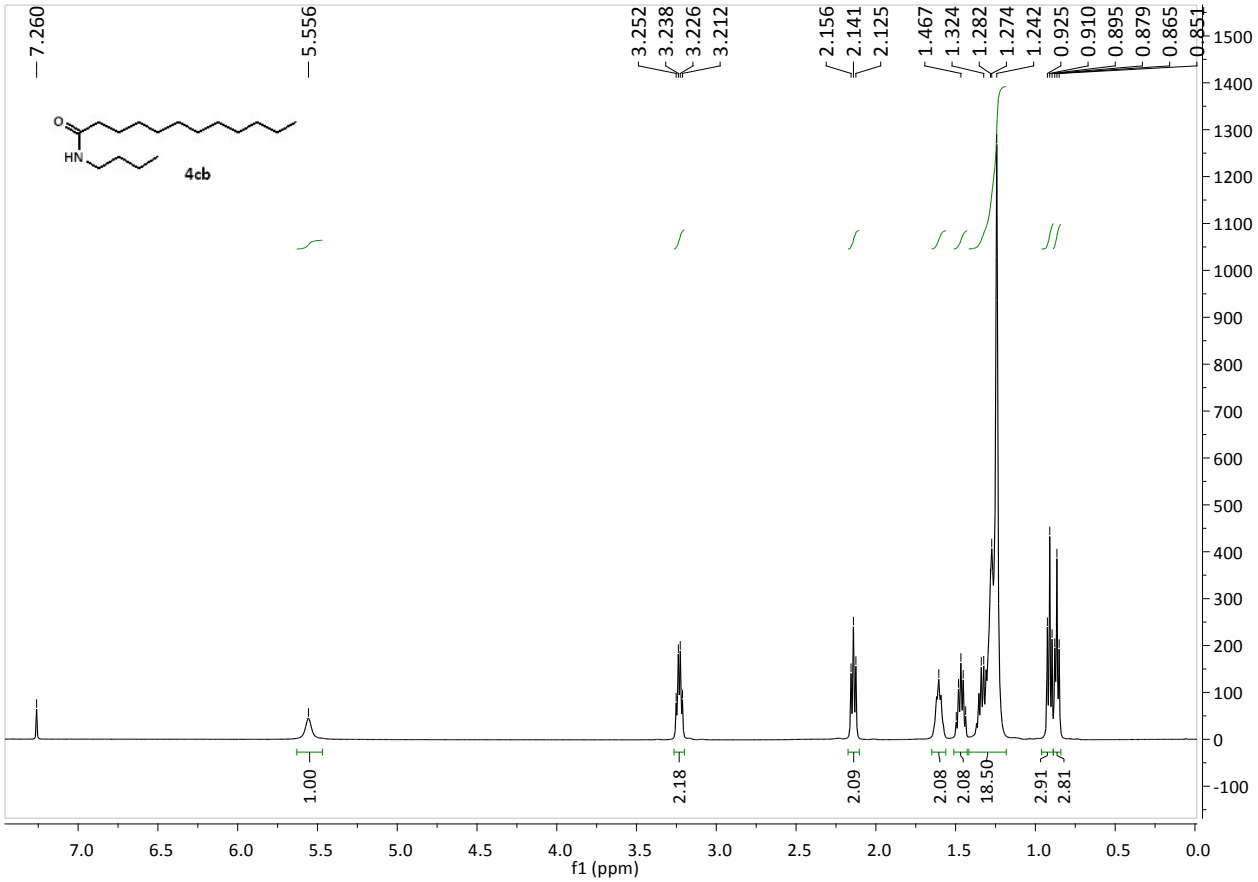
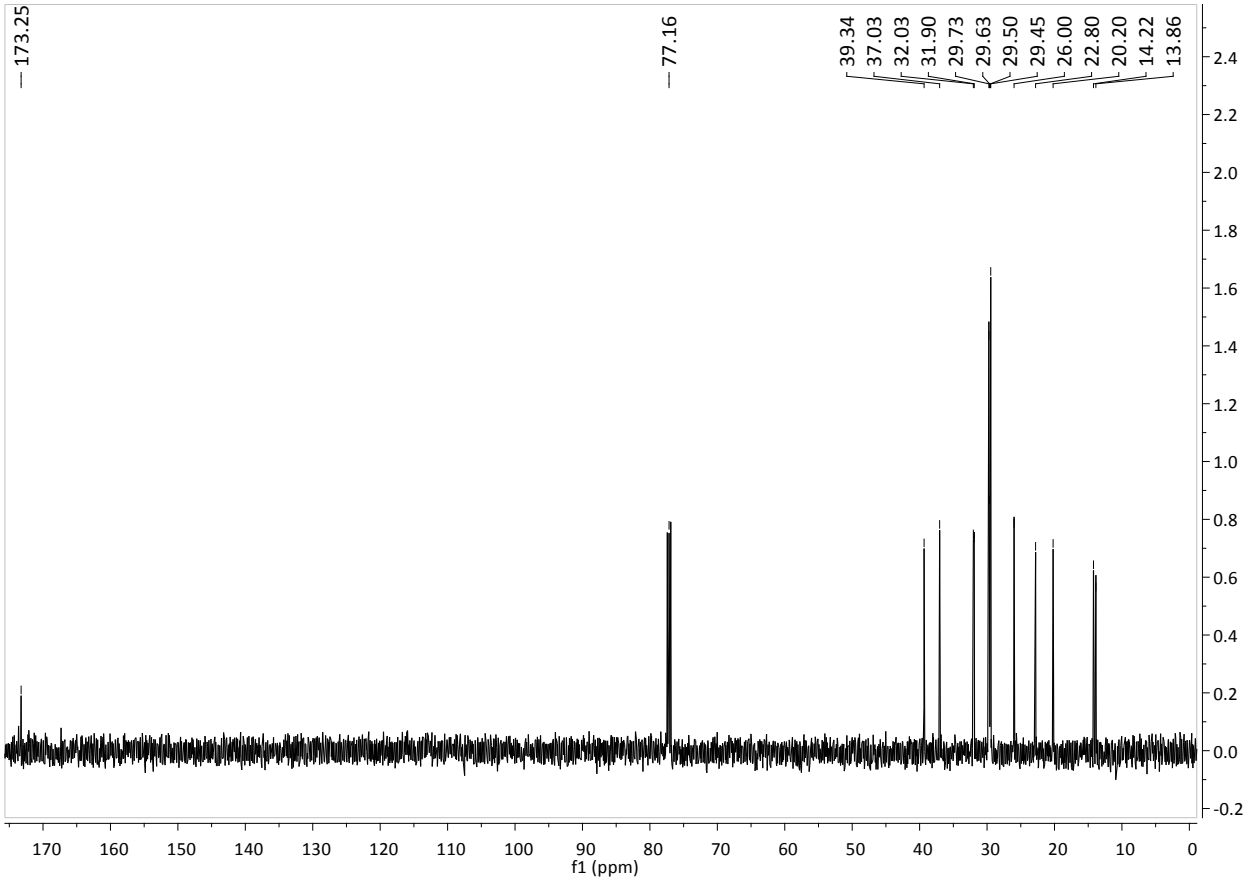
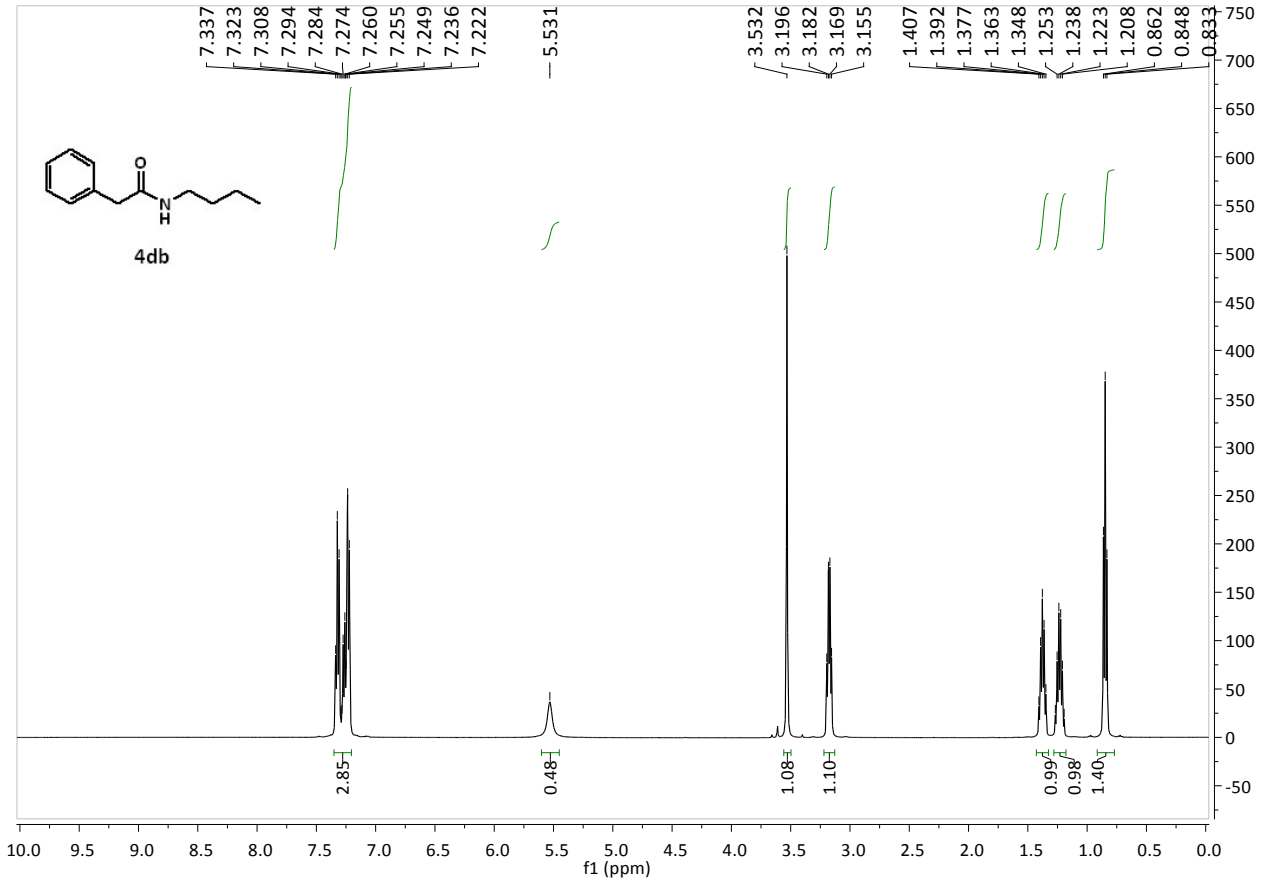
130.08
129.81
77.16
36.08
31.99
29.86
29.80
29.61
29.41
29.40
29.35
29.32
29.22
27.31
27.26
25.64
22.77
14.18
f1 (ppm)

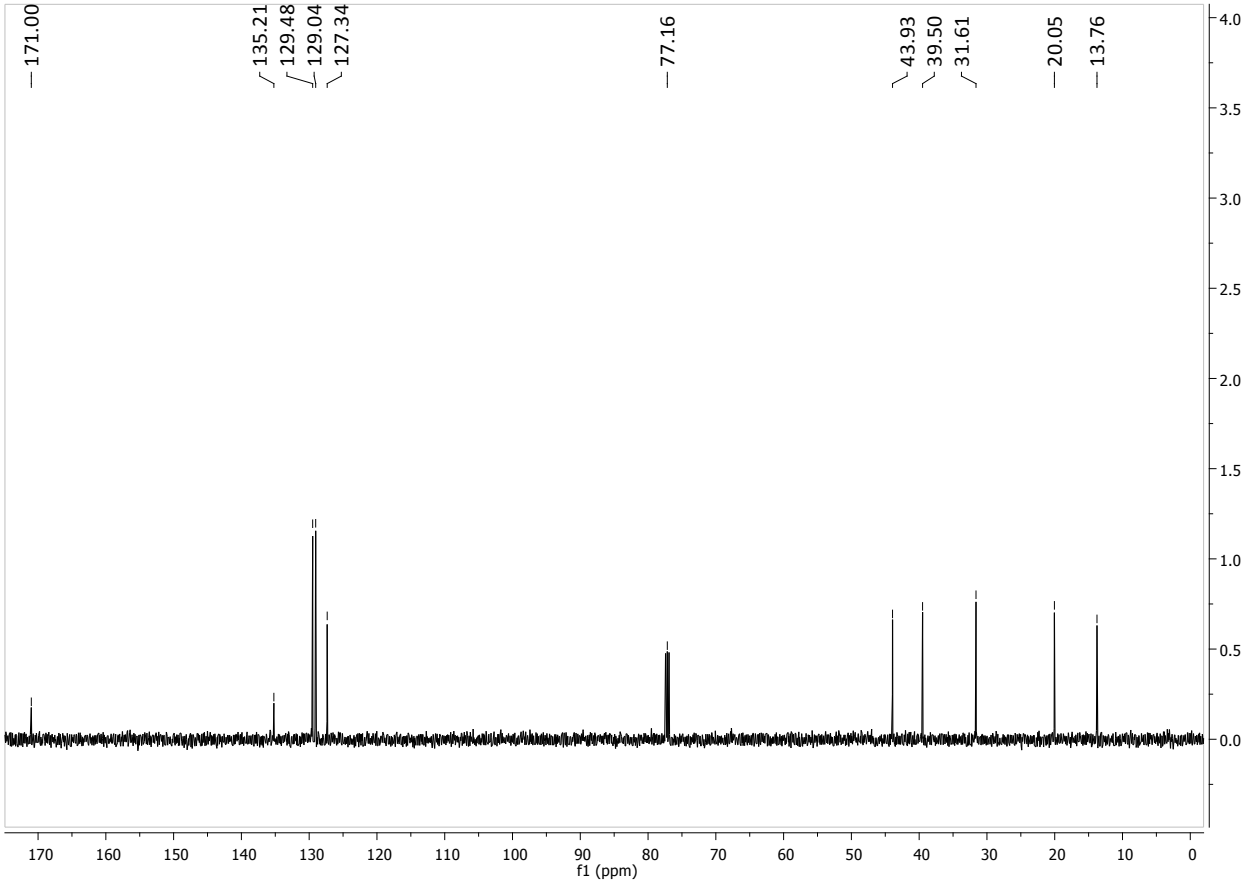

7.260
5.843
3.208
3.194
3.181
3.168
2.126
2.111
2.096
1.434
1.250
1.239
1.224
0.887
0.873
0.858
0.828
0.814
4bb
1.00
2.16
2.15
2.22
2.14
10.74
3.06
3.14
f1 (ppm)

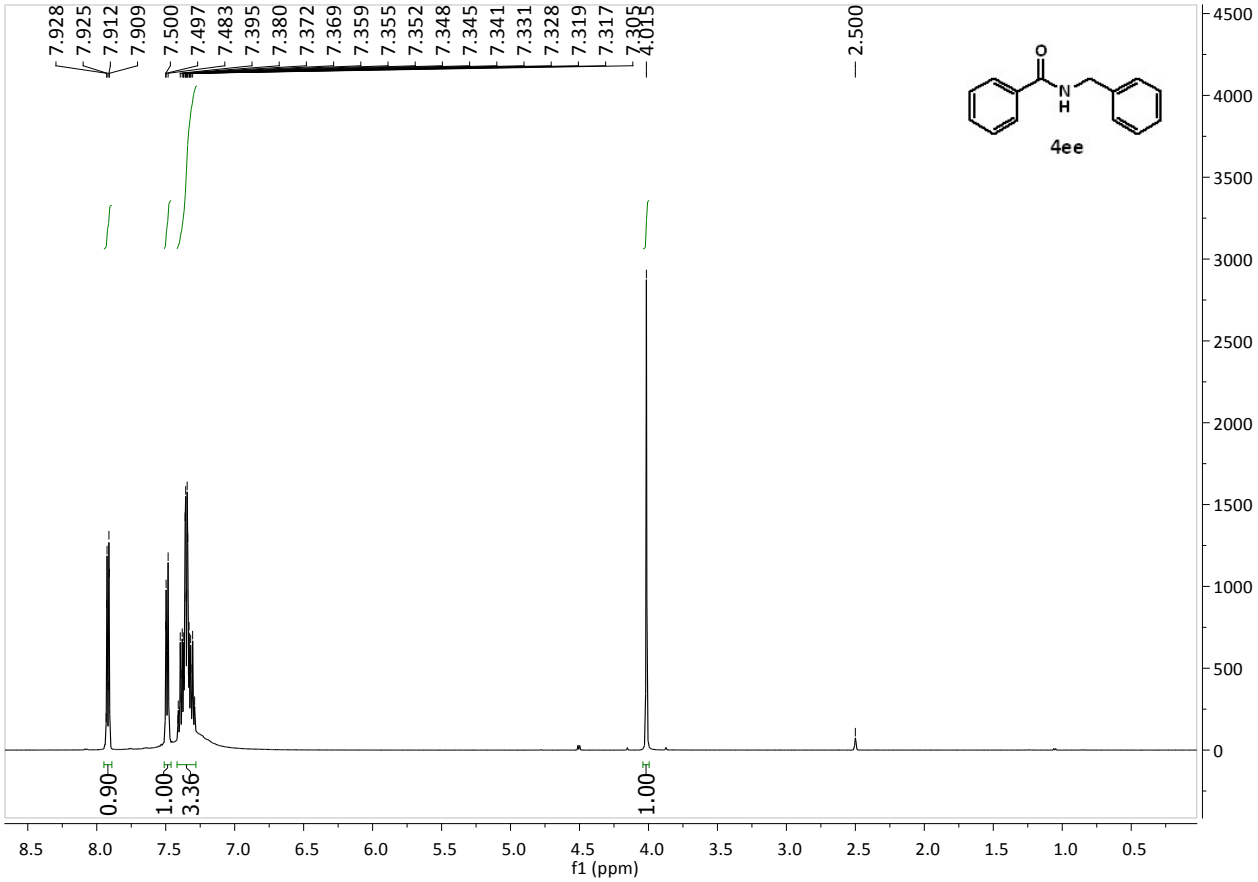

173.29
77.16
39.24
36.90
31.81
31.76
29.34
29.08
25.94
22.64
20.14
14.07
13.78
f1 (ppm)

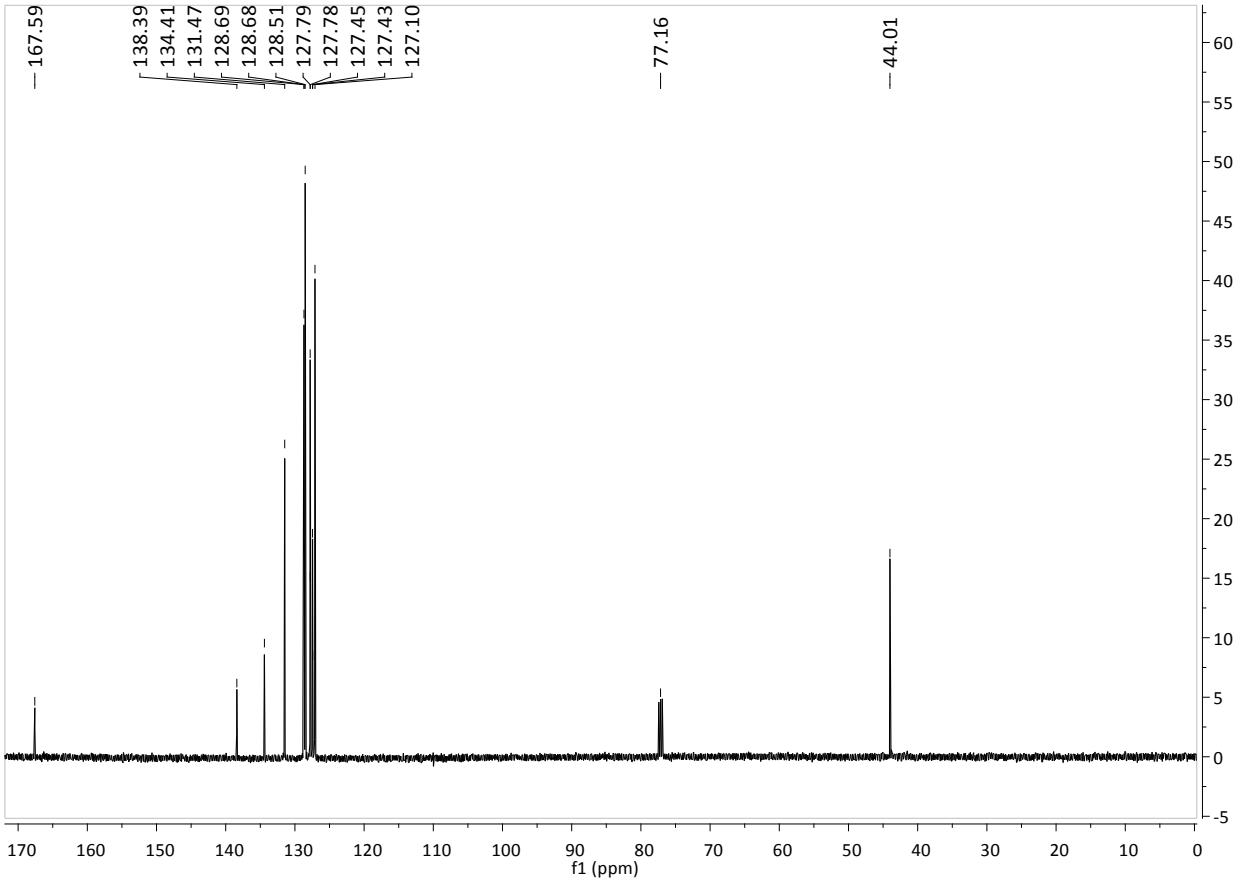

7.260
5.556
3.252
3.238
3.226
3.212
2.156
2.141
2.125
1.467
1.324
1.282
1.274
1.242
0.925
0.910
0.895
0.879
0.865
0.851
4cb
1.00
2.18
2.09
2.08
2.08
18.50
2.91
2.81
f1 (ppm)

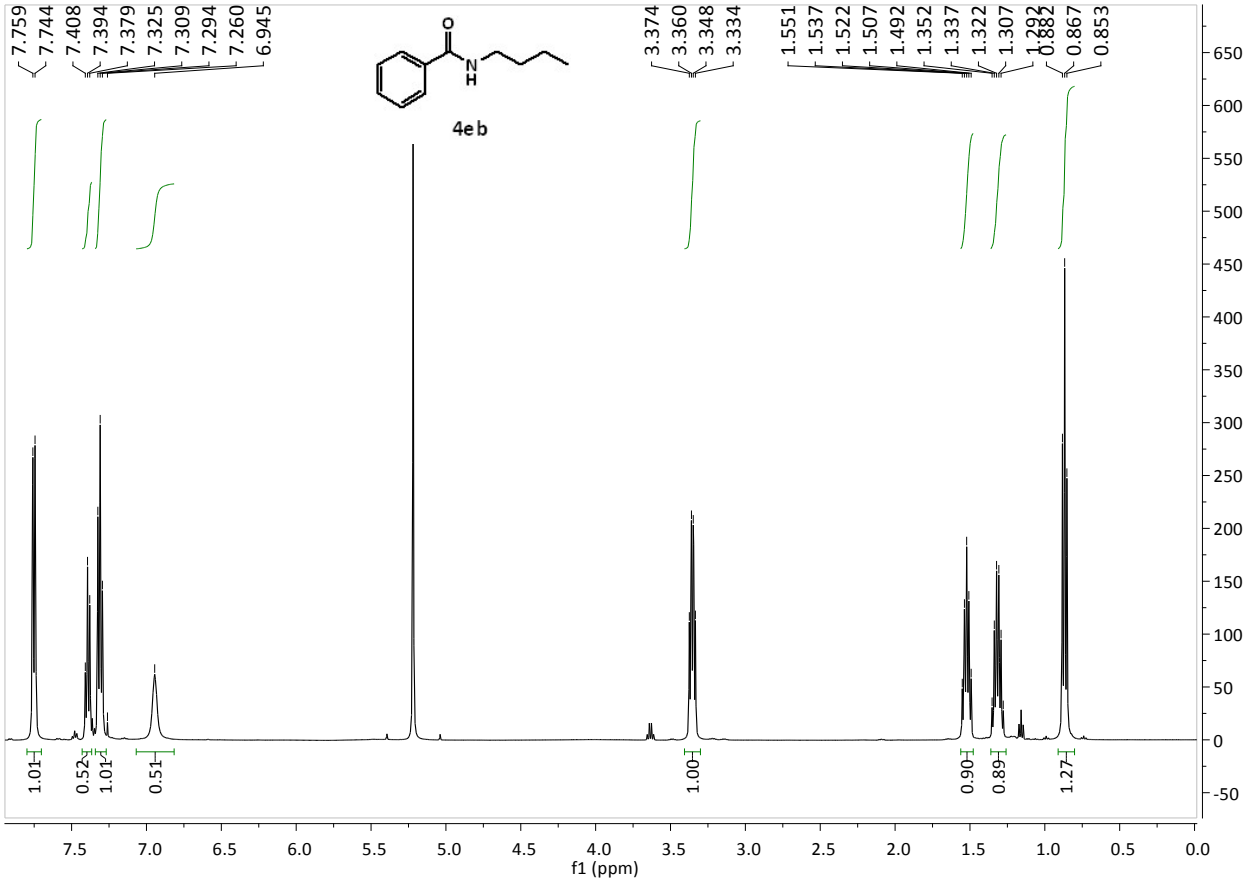

173.25
77.16
39.34
37.03
32.03
31.90
29.73
29.63
29.50
29.45
26.00
22.80
20.20
14.22
13.86
f1 (ppm)

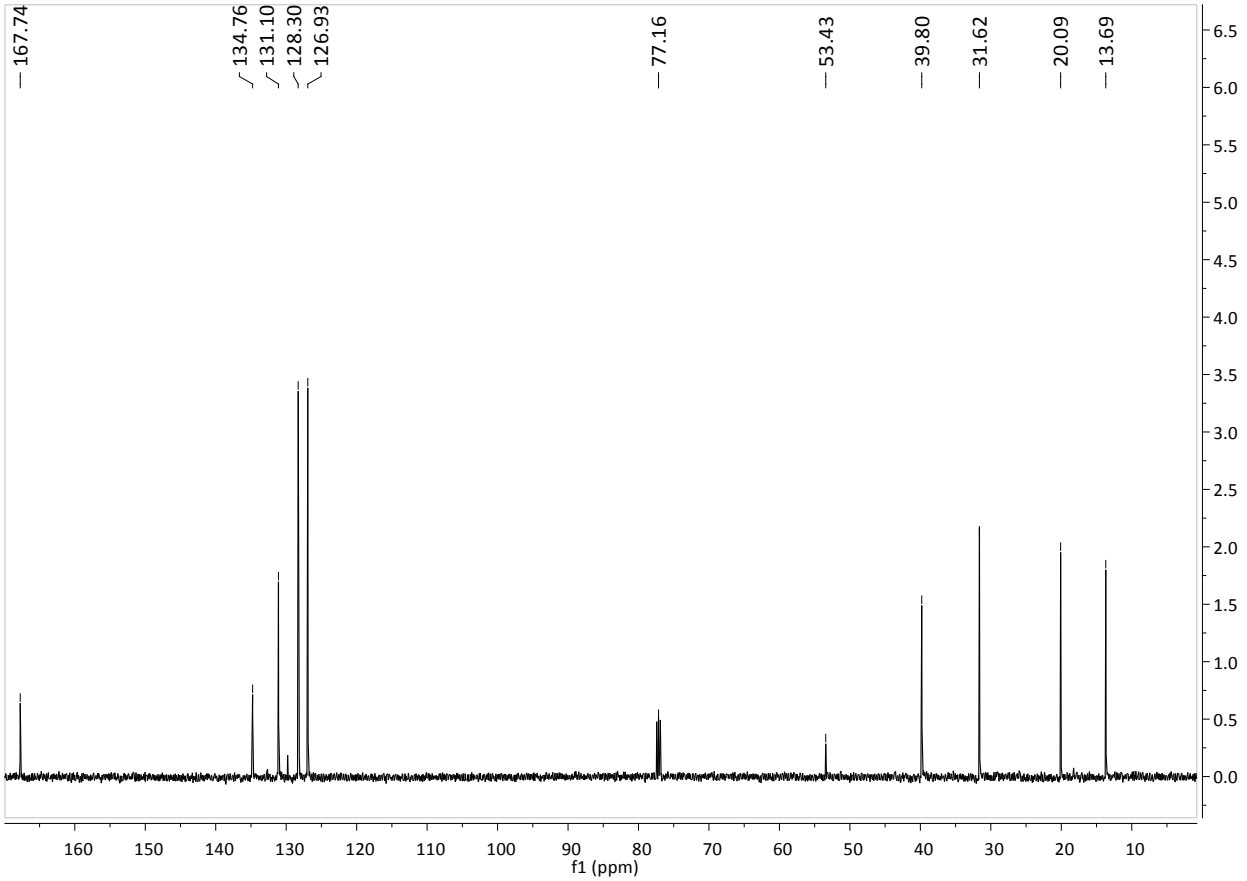

7.337
7.323
7.308
7.294
7.284
7.274
7.260
7.255
7.249
7.236
7.222
5.531
3.532
3.196
3.182
3.169
3.155
1.407
1.392
1.377
1.363
1.348
1.253
1.238
1.223
1.208
0.862
0.848
0.833
O
N
H
4db
2.85
0.48
1.08
1.10
0.99
0.98
1.40
f1 (ppm)

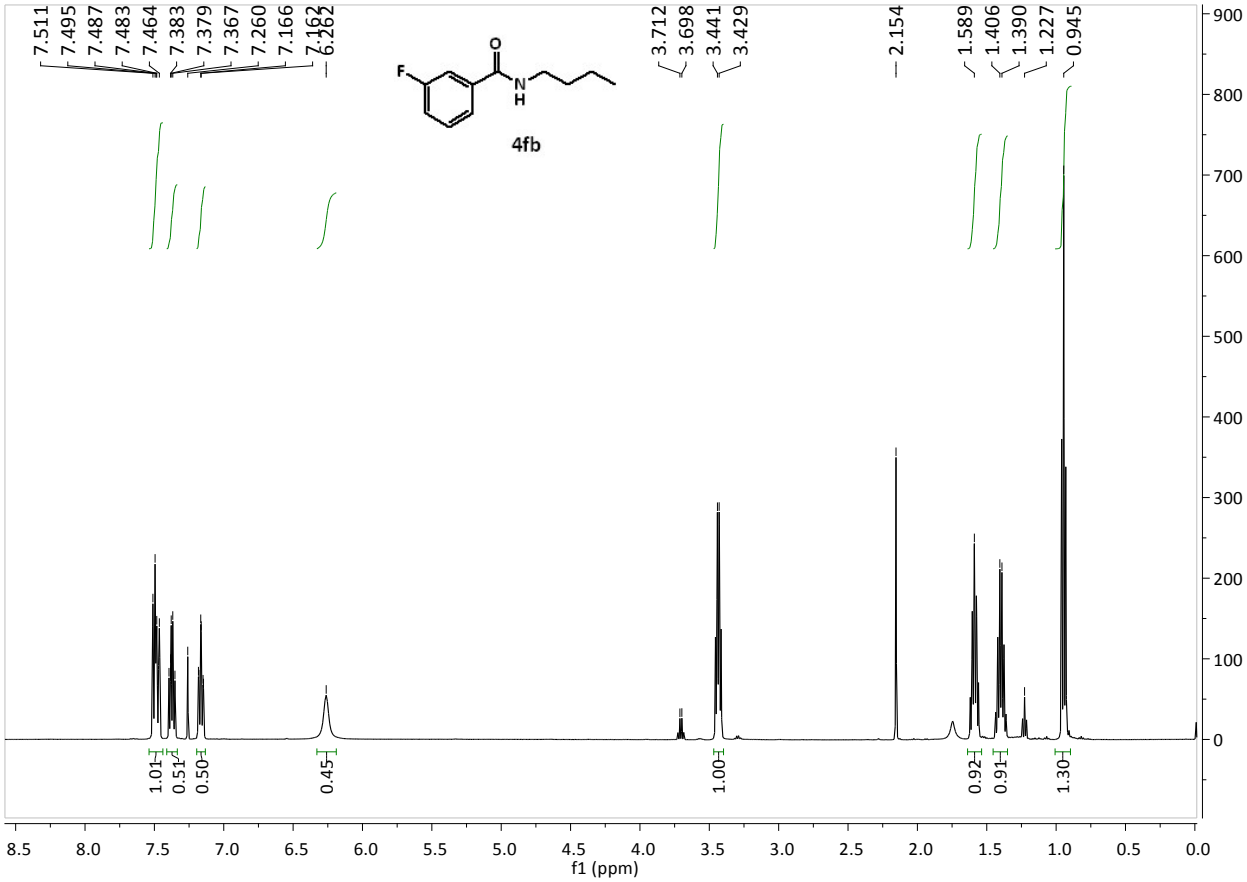

171.00
135.21
129.48
129.04
127.34
77.16
43.93
39.50
31.61
20.05
13.76
f1 (ppm)

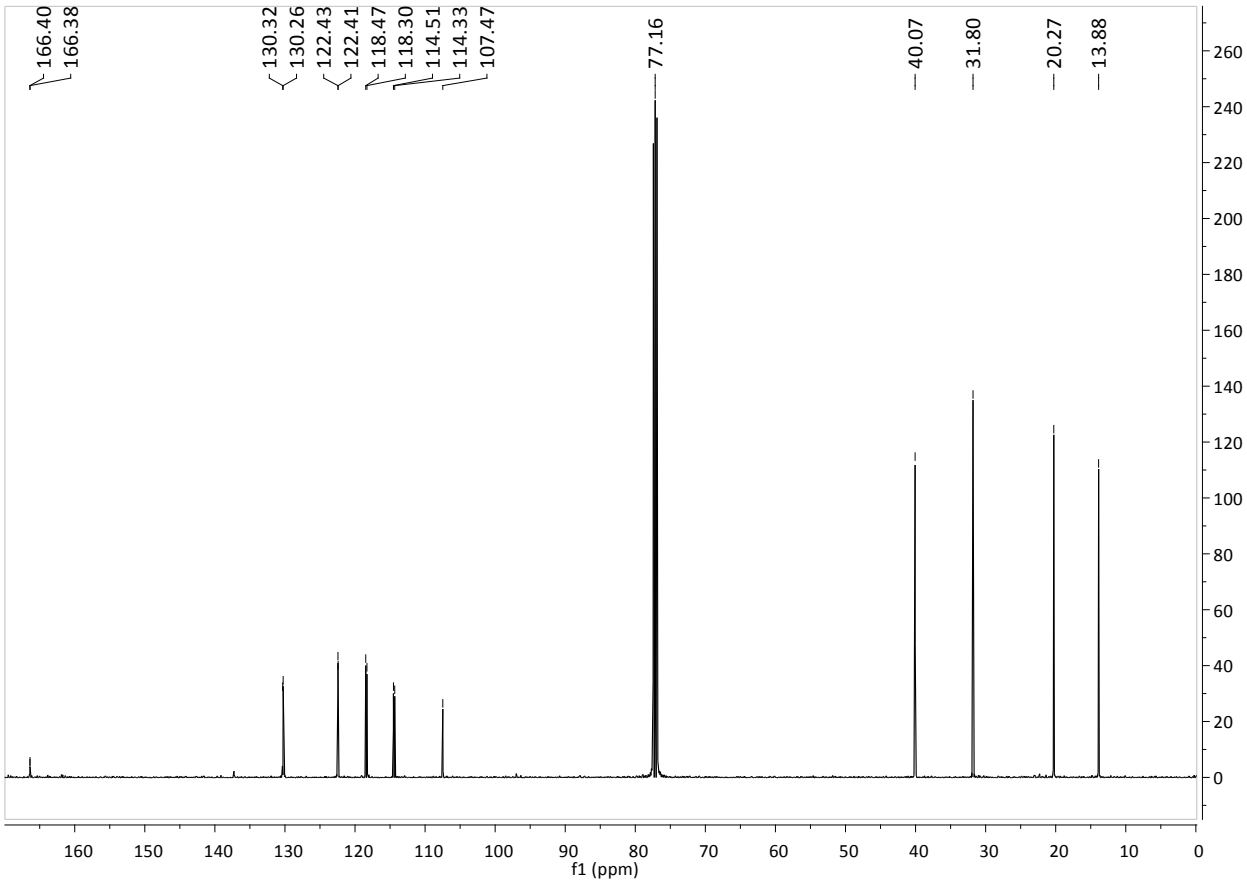

7.928
7.925
7.912
7.909
7.500
7.497
7.483
7.395
7.380
7.372
7.369
7.359
7.355
7.352
7.348
7.345
7.341
7.331
7.328
7.319
7.317
7.305
4.015
2.500
4ee
0.90
1.00
3.36
1.00
f1 (ppm)

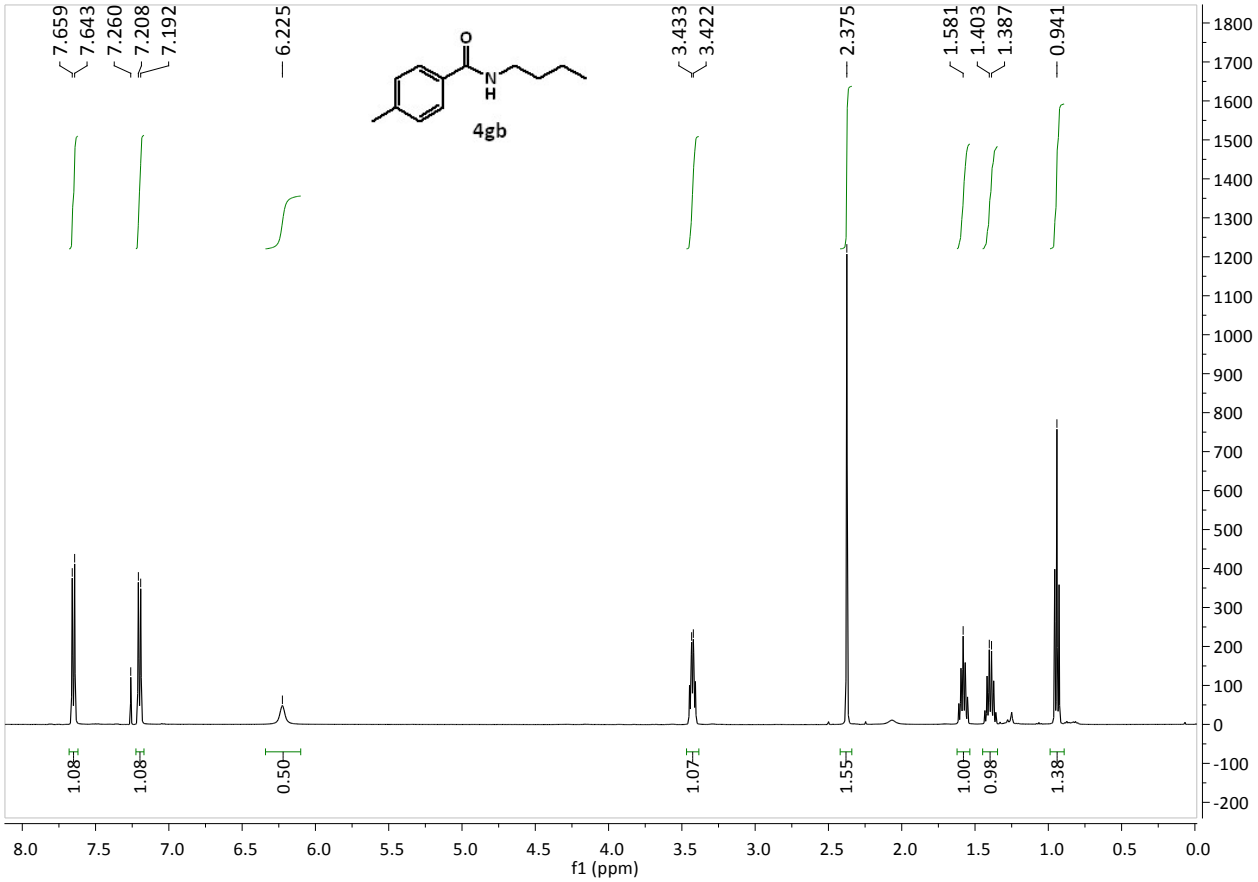

167.59
138.39
134.41
131.47
128.69
128.68
128.51
127.79
127.78
127.45
127.43
127.10
77.16
44.01
f1 (ppm)

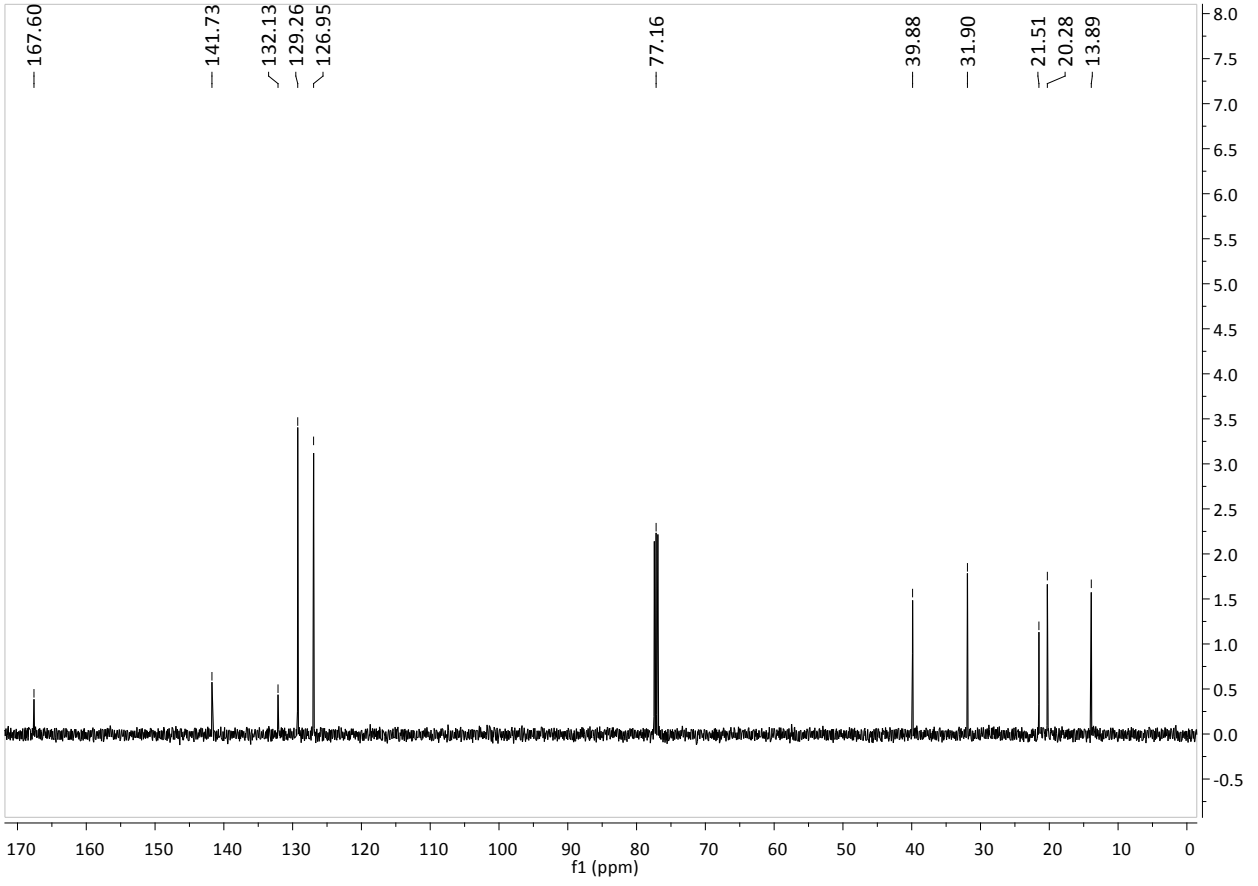

7.759
7.744
7.408
7.394
7.379
7.325
7.309
7.294
7.260
6.945
3.374
3.360
3.348
3.334
1.551
1.537
1.522
1.507
1.492
1.352
1.337
1.322
1.307
1.282
0.882
0.867
0.853
4eb
1.01
0.52
1.01
0.51
1.00
0.90
0.89
1.27
f1 (ppm)

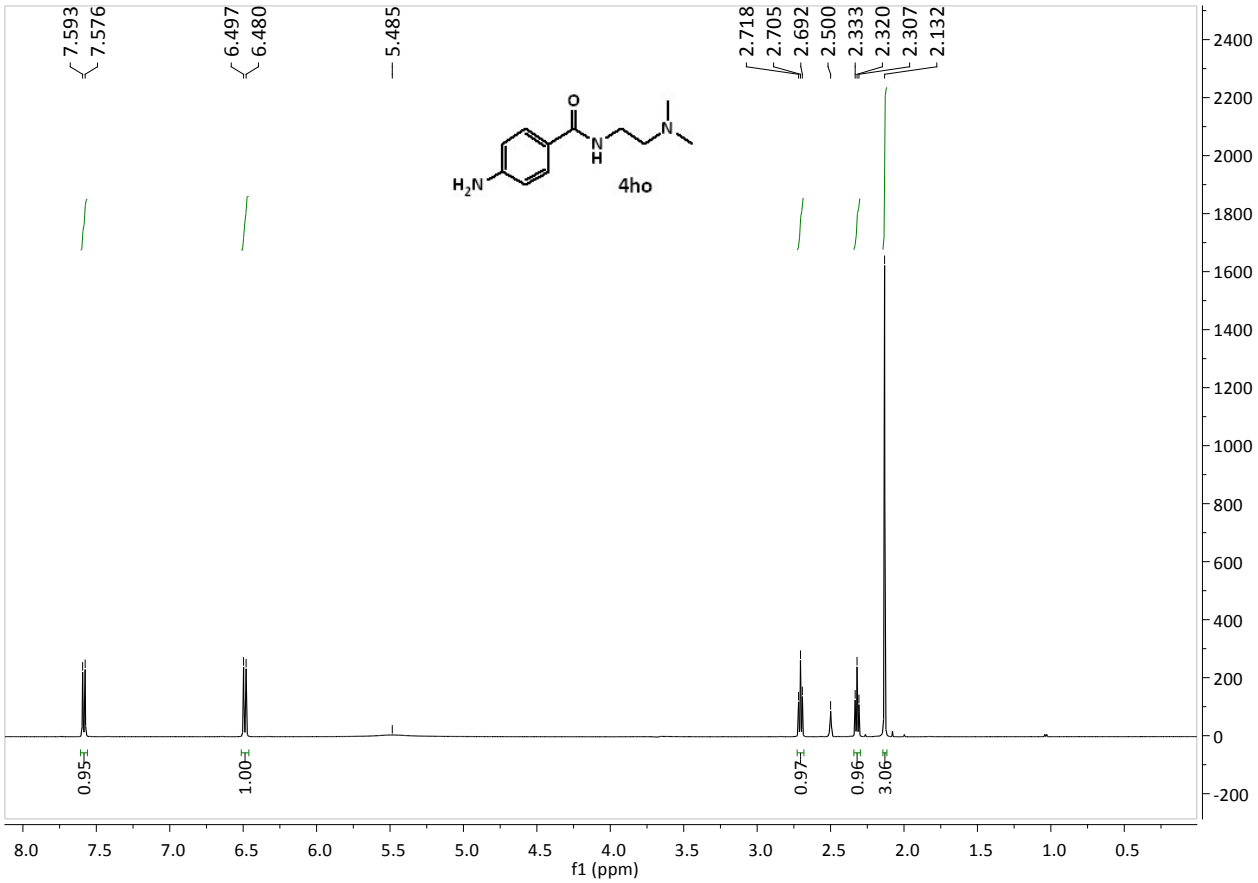

167.74
134.76
131.10
128.30
126.93
77.16
53.43
39.80
31.62
20.09
13.69
f1 (ppm)


7.511
7.495
7.487
7.483
7.464
7.383
7.379
7.367
7.260
7.166
7.162
6.262
3.712
3.698
3.441
3.429
2.154
1.589
1.406
1.390
1.227
0.945
F
O
N
H
4fb
1.01
0.51
0.50
0.45
1.00
0.92
0.91
1.30
f1 (ppm)

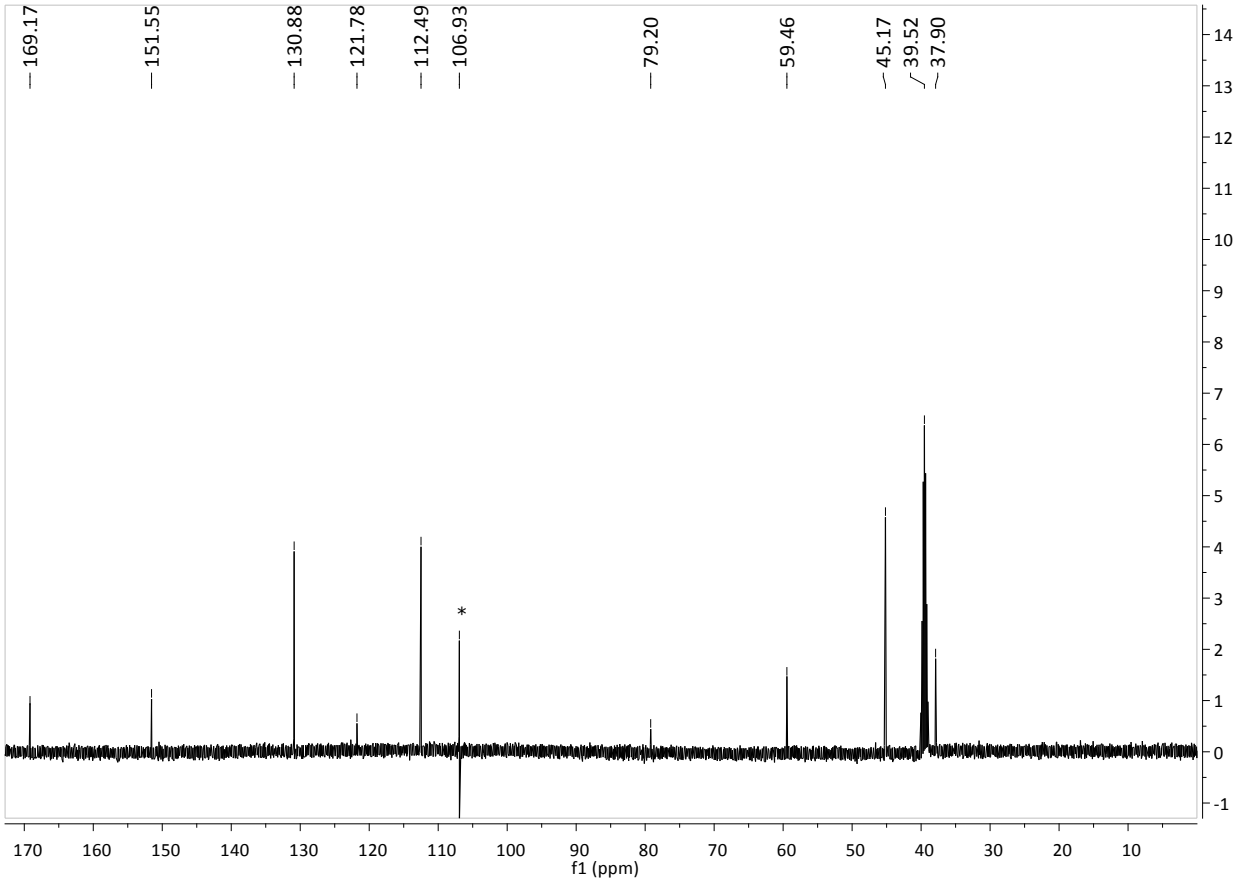

166.40
166.38
130.32
130.26
122.43
122.41
118.47
118.30
114.51
114.33
107.47
77.16
40.07
31.80
20.27
13.88
f1 (ppm)

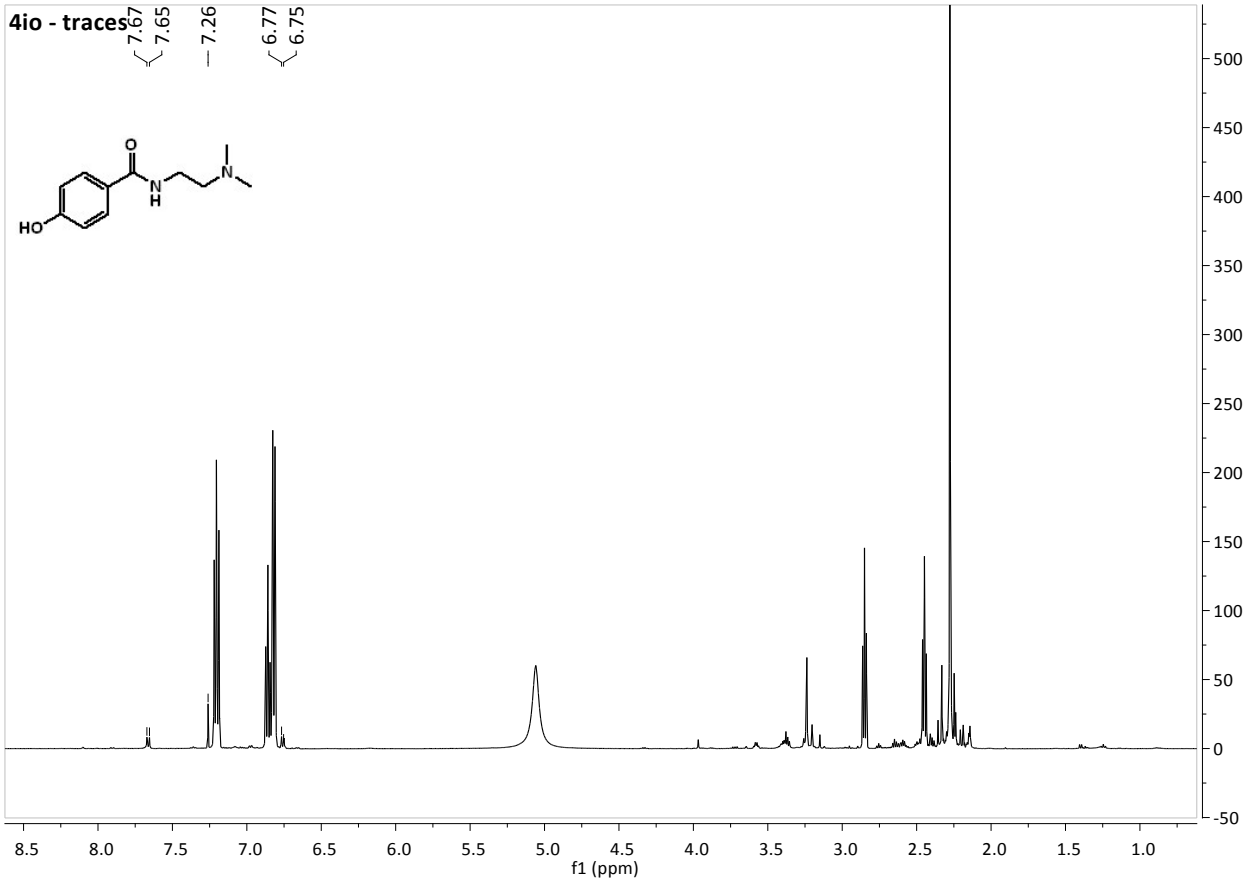

7.659
7.643
7.260
7.208
7.192
6.225
3.433
3.422
2.375
1.581
1.403
1.387
0.941
4gb
1.08
1.08
0.50
1.07
1.55
1.00
0.98
1.38
f1 (ppm)


167.60
141.73
132.13
129.26
126.95
77.16
39.88
31.90
21.51
20.28
13.89
f1 (ppm)



*signal due to the transmitter




4io - traces
7.67
7.65
7.26
6.77
6.75
O
N
H
N
HO
500
450
400
350
300
250
200
150
100
50
0
-50
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)

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