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Synthesis of *Ortho*-Arylated and Alkenylated Benzamides by Palladium-Catalyzed Denitrogenative Cross-Coupling Reactions of 1,2,3-Benzotriazin-4(3*H*)-Ones with Organoboronic Acids

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Supporting Information

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Experimental Section

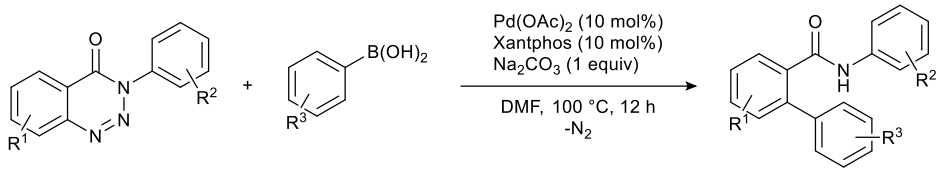
General Information

All experiments were carried out in an oven-dried glassware, in an atmosphere of argon, unless specified otherwise, by standard Schenk techniques. All the solvents used for extraction, filtration and flash chromatography were purchased from Fisher scientific and used without further purification. Flash column chromatography was performed on 100-200 mesh silica gel. ^{1}H-, ^{13}C-, NMR spectroscopy was performed on Bruker BBFO (500 MHz) spectrometer. Chemical shifts were determined relative to the residual solvent peaks ($CHCl_3$, $\delta = 7.26$ for ^{1}H NMR, $\delta = 77.0$ for ^{13}C NMR). The mass spectra were recorded on an Agilent 6200 Series TOF LC/MS spectrometer and Q-TOF Micro mass spectrometer. $Pd(OAc)_2$ were purchased from Sigma Aldrich and used. The starting materials, 1,2,3-Benzotriazin-4(3*H*)-one (**1a-1m)** were prepared according to literature procedure[1]. Organo boronic acid **2** were purchased from Spectrochem, Alfa Aesar Sigma Aldrich and used. Ligands such as Xantphos, triphenyl phosphine (PPh_3), tri(2-furyl) phosphine (TFP), diphenyl phosphino ethane (dppe), diphenyl phosphino propane (dppp), 2,2'-bipyridine, 1,10-Phenanthroline were purchased from Sigma Aldrich, Alfa Aesar and used.

Computational Methodology

All geometry optimizations of reactants, intermediates, transition states, and products are performed using M06L functional in conjunction with 6-311G* basis set for C, P, N, O, and H and SDD effective core potentials for Pd and Ni. Vibrational analysis has been carried out on optimized geometries and no imaginary frequencies were observed in the cases of reactants, intermediates, and products. However, in the cases of transition state one imaginary frequency related to bond formation is observed. Further, intrinsic reaction coordinate analysis has been carried to confirm reactants and products are connected through transition state. The solvents effects are studied using SMD model and 1,4-dioxane is considered as solvent. All calculations are performed using Gaussian 16 suite of package.[4]

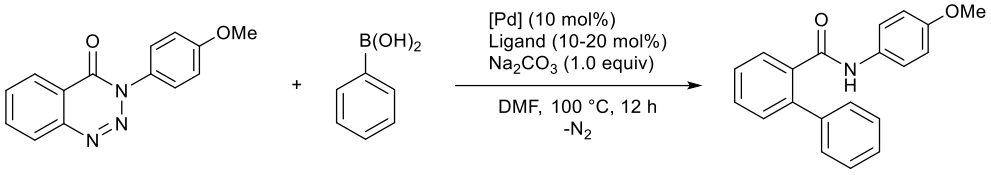
General procedure for palladium-catalyzed *ortho*-arylation and alkenylation of 1,2,3-Benzotriazin-4(3*H*)-ones (1) with organoboronic acids (2).



1 2 3

An oven-dried pressure tube containing 1,2,3-Benzotriazin-4(3*H*)-one **1** (0.40 mmol), organoboronic acid **2** (0.60 mmol), $Pd(OAc)_2$ (0.04 mmol), Xantphos (0.04 mmol), and Na_2CO_3 (0.4 mmol) was evacuated and purged with argon gas three times at room temperature. To that, dry DMF (2.0 mL) was added and stirred for 2 minutes at room temperature. The sealed tube was then placed in a preheated oil bath at 100 °C for 12 h. After completion of the reaction, the mixture was cooled and diluted with ethyl acetate (30 mL). The mixture was filtered through a Celite and silica gel pad and was washed with ethyl acetate (3 x 15 mL). The reaction mass was poured into 50 mL of water and shaken well. The organic layer was separated, dried over anhydrous sodium sulfate, and concentrated under vacuum. The residue was purified by silica gel column chromatography using suitable eluent to afford the desired product **3**.

Table-1: Effect of other palladium catalysts[a]



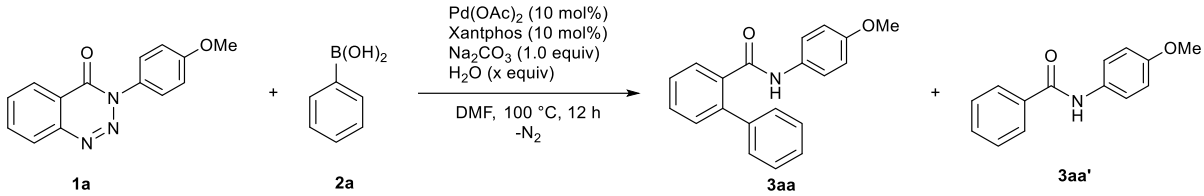
1a 2a 3aa

Entry	Catalyst	Ligand	Yield (%)[b]
1	$Pd(Tfa)_2$	Xantphos	90
2	$PdCl_2(PPh_3)_2$	-	81
3	$Pd(dba)_2$	Xantphos	47
4	$Pd(dba)_2$	PPh_3 (20 mol%)	73

5	$Pd(PPh_3)_4$	-	33

[a]All the reactions were carried out using 1,2,3-benzotriazin-4-(3*H*)-one **(1a)** (0.4 mmol), phenyl boronic acid **(2a)** (0.6 mmol), [Pd] (0.04 mmol), ligand (0-0.08 mmol), sodium carbonate (0.4 mmol) and dry DMF (2.0) mL at 100 °C for 12 h under argon atmosphere. [b]GC yields.

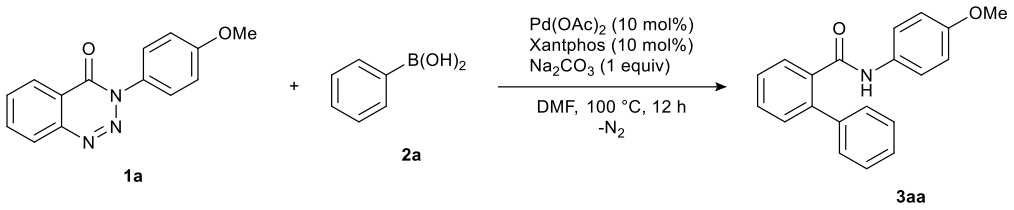
Table-2: Effect of water[a]



Entry	Water content	Yield (%) [b]	
		3aa	3aa'
1	-	95	3
2	1.0 equiv	93	5
3	2.0 equiv	92	7
4	3.0 equiv	90	9
5	5.0 equiv	88	10
6	10.0 equiv	85	15
7	100.0 equiv	51	43
[c]8	-	75	8

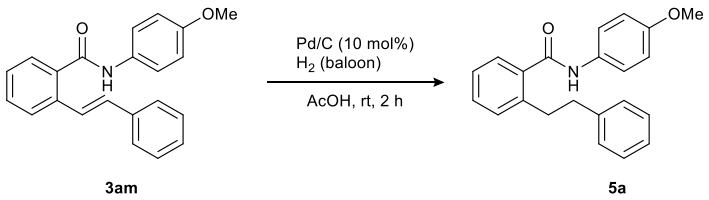
[a]All the reactions were carried out using 1,2,3-benzotriazin-4-(3*H*)-one **(1a)** (0.4 mmol), phenyl boronic acid **(2a)** (0.6 mmol), $Pd(OAc)_2$ (0.04 mmol), Xantphos (0.04 mmol), sodium carbonate (0.4 mmol) and H_2O (X.0 equiv) and dry DMF (2.0) mL at 100 °C for 12 h under argon atmosphere. [b]GC yields, [c]Normal DMF was used as solvent.

Procedure for the synthesis of *N*-(4-methoxyphenyl)-[1,1'-biphenyl]-2-carboxamide (3aa) in a gram scale.



A sealed tube containing 1,2,3-benzotriazin-4(3*H*)-ones **1a** (1g, 4 mmol), phenylboronic acid **2a** (0.731g, 6 mmol), $Pd(OAc)_2$ (0.045g, 0.2 mmol), Xantphos (0.116g, 0.2 mmol) and Na_2CO_3 (0.424g, 4 mmol) was evacuated and purged with argon gas three times. To this mixture, dry DMF (8.0 mL) was added via syringe under argon atmosphere at room temperature and stirred for 2 min. The sealed tube was then placed in a preheated oil bath at 100 °C for 12 h. After completion of reaction, the mixture was cooled and diluted with ethyl acetate (50 mL). The mixture was filtered through a celite bed and was washed with ethyl acetate (3 × 20 mL). The reaction mass was poured into 80 mL of water and shaken well. The organic layer was separated, dried on anhydrous sodium sulphate bed and concentrated under reduced pressure. The residue was purified by silica gel column chromatography using hexane:ethyl acetate (93:7) as eluent to afford the desired pure product **3aa** (1.08g, 89%).

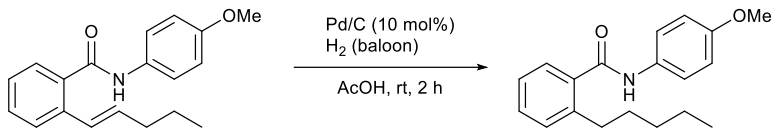
Procedure for the preparation of *N*-(4-methoxyphenyl)-2-phenethylbenzamide (5a).



N-(4-methoxyphenyl)-2-phenethylbenzamide (**5a**) has been prepared according to the literature procedure[2]. **3am** (132 mg, 0.40 mmol), Pd/C (0.04 mmol) and acetic acid (2 mL) were taken in a sealed tube closed with rubber septum. The tube was evacuated and filled with hydrogen gas purged from a balloon for three times. The reaction mixture was allowed to stir at rt for 2 h. After the reaction completion, the reaction mixture was diluted with ethyl acetate (20 mL), filtered through a celite bed, and washed the bed with ethyl acetate (3 × 10 mL). Then the reaction mixture was washed with water (30 mL) and brine (20 mL). The extract was dried with anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the crude

residue was purified through a silica gel column using hexane:ethyl acetate (9:1) as eluent to afford the desired pure product **5** (128 mg, 97% yield)**.**

Procedure for the preparation of *N*-(4-methoxyphenyl)-2-pentylbenzamide (5b).

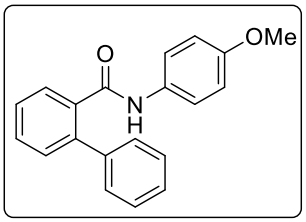


3ap **5b**

N-(4-methoxyphenyl)-2-pentylbenzamide (**5b**) has been prepared according to the literature procedure[2]. **3ap** (118 mg, 0.4 mmol), Pd/C (0.04 mmol) and acetic acid (2 mL) were taken in a sealed tube closed with rubber septum. The tube was then evacuated and filled with hydrogen gas purged from a balloon for three times. The reaction mixture was allowed to stir at rt for 2 h. After the reaction completion, the reaction mixture was filtered through a celite bed and washed the bed with ethyl acetate 5 mL as three times. Then, the reaction mixture was washed with water (30 mL) and brine (20 mL). The extract was dried with anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the crude residue was purified through a silica gel column using hexane:ethyl acetate (9:1) as eluent to afford the desired pure product **5b** (116 mg, 98% yield)**.**

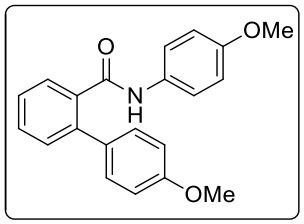
^{1}H NMR, ^{13}C NMR, HRMS data

***N*-(4-Methoxyphenyl)-[1,1'-biphenyl]-2-carboxamide (3aa)**



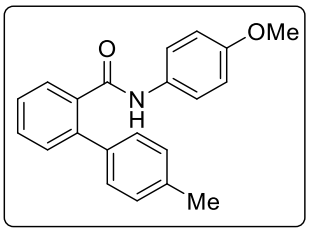
Colorless solid (113 mg, 93% yield); mp: 152-153 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.1 Hz, 1H), 7.57 – 7.50 (m, 1H), 7.44 (ddd, *J* = 13.8, 10.1, 5.2 Hz, 7H), 7.04 – 6.98 (m, 2H), 6.81 (s, 1H), 6.76 (d, *J* = 8.9 Hz, 2H), 3.75 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.0, 156.5, 140.0, 139.4, 135.3, 130.5, 130.2, 129.5, 128.9, 128.8, 128.0, 127.8, 121.8, 113.9, 55.4; HRMS calculated for $C_{20}H_{17}NO_2$ $[M+H]^+$: 304.1338, found 304.1330; Purification: Flash chromatography; Eluent: hexane/EtOAc (93/7); R_f = 0.35.

4'-Methoxy-*N*-(4-methoxyphenyl)-[1,1'-biphenyl]-2-carboxamide (3ab)



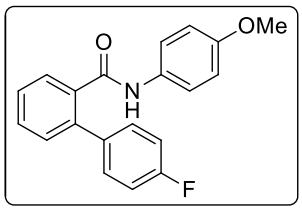
Colorless solid (104 mg, 78% yield); mp: 133-134 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 (d, *J* = 7.6 Hz, 1H), 7.51 (td, *J* = 7.5, 1.3 Hz, 1H), 7.44 (dd, *J* = 7.6, 1.0 Hz, 1H), 7.40 (t, *J* = 7.3 Hz, 3H), 7.10 – 7.05 (m, 2H), 6.97 (d, *J* = 8.7 Hz, 2H), 6.87 (s, 1H), 6.78 (d, *J* = 9.0 Hz, 2H), 3.83 (s, 3H), 3.76 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.32, 159.5, 156.5, 139.0, 135.2, 132.2, 130.7, 130.5, 130.3, 130.0, 129.4, 127.4, 121.8, 114.3, 114.0, 55.4, 55.4; HRMS calculated for $C_{21}H_{19}NO_3$ $[M+H]^+$: 334.1443, found 334.1441; Purification: Flash chromatography; Eluent: hexane/EtOAc (9/1); R_f = 0.32.

***N*-(4-Methoxyphenyl)-4'-methyl-[1,1'-biphenyl]-2-carboxamide (3ac)**



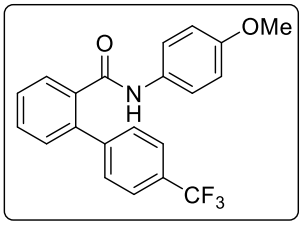
Colorless solid (103 mg, 81%); mp: 147-148 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.7 Hz, 1H), 7.52 (td, *J* = 7.4, 1.2 Hz, 1H), 7.48 – 7.43 (m, 1H), 7.40 (d, *J* = 7.5 Hz, 1H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.26 (s, 1H), 7.24 (s, 1H), 7.07 – 7.00 (m, 2H), 6.85 (s, 1H), 6.81 – 6.74 (m, 2H), 3.76 (s, 3H), 2.40 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.2, 156.4, 139.4, 137.8, 137.0, 135.2, 130.7, 130.5, 130.3, 129.6, 129.4, 128.7, 127.6, 121.8, 113.9, 55.4, 21.2; HRMS calculated for $C_{21}H_{19}NO_2$ $[M+H]^+$: 318.1494, found 318.1482; Purification: Flash chromatography; Eluent: hexane/EtOAc (95/5); R_f = 0.37.

4'-Fluoro-*N*-(4-methoxyphenyl)-[1,1'-biphenyl]-2-carboxamide (3ad)



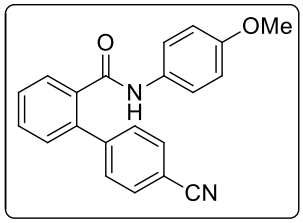
Colorless solid (122 mg, 95% yield); mp: 167-168 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 – 7.76 (m, 1H), 7.52 (td, *J* = 7.5, 1.3 Hz, 1H), 7.48 – 7.42 (m, 3H), 7.39 (dd, *J* = 7.6, 0.7 Hz, 1H), 7.15 – 7.07 (m, 4H), 6.88 (s, 1H), 6.82 – 6.77 (m, 2H), 3.76 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.2, δ 162.6 (d, J_{C-F} = 247.9 Hz), 138.3, 135.9 (d, J_{C-F} = 3.4 Hz), 135.6, 130.5 (d, J_{C-F} = 6.8 Hz), 130.4, 130.3 (d, J_{C-F} = 15.9 Hz), 129.1, 127.9, 121.8, 115.8 (d, J_{C-F} = 21.6 Hz), 114.1, 55.4; HRMS calculated for $C_{20}H_{16}NO_2F$ $[M+H]^+$: 322.1243, found 322.1236; Purification: Flash chromatography; Eluent: hexane/EtOAc (95/5); R_f = 0.31.

***N*-(4-Methoxyphenyl)-4'-(trifluoromethyl)-[1,1'-biphenyl]-2-carboxamide (3ae)**



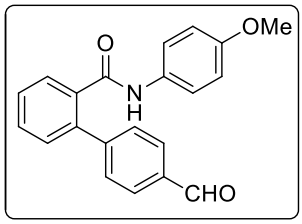
Colorless solid (138 mg, 93% yield); mp: 185-186 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.79 (d, *J* = 6.7 Hz, 1H), 7.68 (d, *J* = 7.7 Hz, 2H), 7.58 (dd, *J* = 18.9, 7.6 Hz, 3H), 7.51 (t, *J* = 7.0 Hz, 1H), 7.43 (d, *J* = 7.5 Hz, 1H), 7.07 (d, *J* = 8.3 Hz, 2H), 6.86 (s, 1H), 6.79 (d, *J* = 8.0 Hz, 2H), 3.77 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.0, 156.7, 143.7, 138.2, 135.9, 130.7, 130.3, 130.2, 130.1, 129.8, 129.1, 129.0, 128.5, 125.7 (m, J_{C-F} = 7.4, 3.7 Hz), 121.8, 114.1, 55.4; HRMS calculated for $C_{21}H_{16}NO_2F_3$ $[M+H]^+$: 372.1211, found 372.1204; Purification: Flash chromatography; Eluent: hexane/EtOAc (92/8); R_f = 0.35.

4'-Cyano-*N*-(4-methoxyphenyl)-[1,1'-biphenyl]-2-carboxamide (3af)



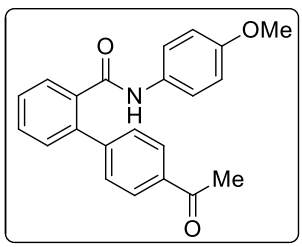
Colorless solid (119 mg, 91% yield); mp: 162-163 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.74 – 7.69 (m, 1H), 7.67 (d, *J* = 8.2 Hz, 2H), 7.59 – 7.53 (m, 3H), 7.49 (t, *J* = 7.3 Hz, 1H), 7.41 (d, *J* = 7.6 Hz, 1H), 7.15 (t, *J* = 6.0 Hz, 2H), 7.12 (s, 1H), 6.81 (d, *J* = 8.9 Hz, 2H), 3.77 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.9, 156.8, 144.7, 137.8, 136.0, 132.3, 130.7, 130.3, 130.1, 129.3, 128.7, 128.7, 121.7, 118.5, 114.2, 111.5, 55.4; HRMS calculated for $C_{21}H_{16}N_2O_2$ $[M+H]^+$: 328.1290, found 328.1284; Purification: Flash chromatography; Eluent: hexane/EtOAc (85/15); R_f = 0.37.

4'-Formyl-*N*-(4-methoxyphenyl)-[1,1'-biphenyl]-2-carboxamide (3ag)



Colorless solid (116 mg, 88% yield); mp: 172-173 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.04 (s, 1H), 7.96 – 7.91 (m, 2H), 7.80 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.66 (d, *J* = 8.1 Hz, 2H), 7.58 (td, *J* = 7.5, 1.4 Hz, 1H), 7.52 (td, *J* = 7.5, 1.3 Hz, 1H), 7.46 (dd, *J* = 7.6, 1.1 Hz, 1H), 7.13 – 7.09 (m, 2H), 6.90 (s, 1H), 6.81 – 6.77 (m, 2H), 3.76 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 191.8, 167.0, 156.7, 146.2, 138.3, 136.0, 135.5, 130.7, 130.4, 130.2, 130.1, 129.4, 129.0, 128.6, 121.7, 114.1, 55.4; HRMS calculated for $C_{21}H_{17}NO_3$ $[M+H]^+$: 332.1287, found 332.1296; Purification: Flash chromatography; Eluent: hexane/EtOAc (9/1); R_f = 0.4.

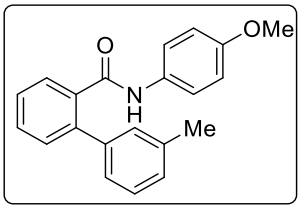
4'-Acetyl-*N*-(4-methoxyphenyl)-[1,1'-biphenyl]-2-carboxamide (3ah)



Colorless solid (127 mg, 92% yield); mp: 148-149 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.2 Hz, 2H), 7.76 (d, *J* = 7.5 Hz, 1H), 7.55 (dd, *J* = 11.8, 8.0 Hz, 3H), 7.48 (t, *J* = 7.4 Hz,

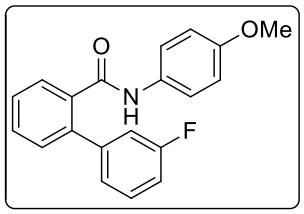
1H), 7.43 (d, *J* = 7.6 Hz, 1H), 7.11 (d, *J* = 8.9 Hz, 2H), 7.04 (s, 1H), 6.77 (d, *J* = 8.9 Hz, 2H), 3.75 (s, 3H), 2.60 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 197.6, 167.1, 156.6, 144.8, 138.4, 136.2, 135.9, 130.6, 130.5, 130.2, 128.9, 128.9, 128.7, 128.4, 121.7, 114.1, 55.4, 26.7; HRMS calculated for $C_{22}H_{19}NO_3$ $[M+H]^+$: 346.1443, found 346.1443; Purification: Flash chromatography; Eluent: hexane/EtOAc (9/1); R_f = 0.41.

***N*-(4-Methoxyphenyl)-3'-methyl-[1,1'-biphenyl]-2-carboxamide (3ai)**



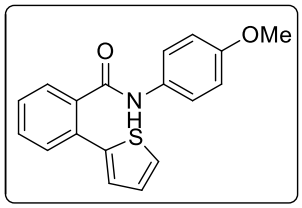
Colorless solid (93 mg, 73% yield); mp: 118-119 °C; ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.11, 156.49, 139.94, 139.59, 138.67, 135.17, 130.56, 130.51, 130.21, 129.54, 128.82, 128.76, 127.75, 125.91, 121.94, 113.94, 55.40, 21.38; ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.1, 156.5, 139.9, 139.6, 138.7, 135.7, 130.6, 130.5, 130.2, 129.5, 128.8, 128.8, 127.7, 125.9, 121.9, 113.9, 55.4, 21.4; HRMS calculated for $C_{21}H_{19}NO_2$ $[M+H]^+$: 318.1494, found 318.1488; Purification: Flash chromatography; Eluent: hexane/EtOAc (93/7); R_f = 0.35.

3'-Fluoro-N-(4-methoxyphenyl)-[1,1'-biphenyl]-2-carboxamide (3aj)



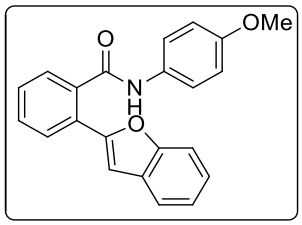
Colorless solid (104 mg, 81% yield); mp: 162-163 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7.5 Hz, 1H), 7.54 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.4 Hz, 1H), 7.44 – 7.35 (m, 2H), 7.24 (s, 1H), 7.20 (d, *J* = 9.5 Hz, 1H), 7.08 (t, *J* = 9.2 Hz, 3H), 6.90 (s, 1H), 6.79 (d, *J* = 8.5 Hz, 2H), 3.76 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.0, 162.8 (d, $J_{C\text{-}F}$= 247.6 Hz), 156.6, 142.2 (d, $J_{C\text{-}F}$= 7.5 Hz), 138.2, 135.7, 130.6, 130.4 (d, $J_{C\text{-}F}$ = 4.3 Hz), 130.3, 130.1, 129.1, 128.2, 124.6 (d, $J_{C\text{-}F}$ = 2.8 Hz), 121.9, 115.7 (d, $J_{C\text{-}F}$ = 22.0 Hz), 114.8 (d, $J_{C\text{-}F}$ = 20.9 Hz), 114.1, 55.4; HRMS calculated for $C_{20}H_{16}NO_2F$ $[M+H]^+$: 322.1243, found 322.1239; Purification: Flash chromatography; Eluent: hexane/EtOAc (93/7); R_f = 0.33.

***N*-(4-Methoxyphenyl)-2-(thiophen-2-yl)benzamide (3ak)**



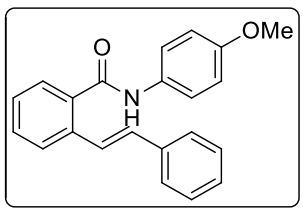
Colorless solid (104mg, 84% yield); mp: 124-125 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.77 (dd, *J* = 7.5, 1.1 Hz, 1H), 7.48 (dqd, *J* = 20.9, 7.4, 1.7 Hz, 3H), 7.39 (dd, *J* = 5.1, 1.1 Hz, 1H), 7.22 – 7.18 (m, 3H), 7.12 (s, 1H), 7.08 (dd, *J* = 5.1, 3.6 Hz, 1H), 6.84 – 6.79 (m, 2H), 3.77 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.0, 156.6, 140.9, 135.9, 131.7, 130.7, 130.3, 129.1, 128.3, 128.0, 127.2, 126.7, 121.9, 114.1, 55.4; HRMS calculated for $C_{18}H_{15}NO_2S$ $[M+H]^+$: 310.0902, found 310.0886; Purification: Flash chromatography; Eluent: hexane/EtOAc (93/7); R_f = 0.41.

2-(Benzofuran-2-yl)-*N*-(4-methoxyphenyl)benzamide (3al)



Pale brown solid (100 mg, 73%); mp: 173-174 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.91 (d, *J* = 7.7 Hz, 1H), 7.63 (d, *J* = 7.5 Hz, 1H), 7.55 (d, *J* = 7.2 Hz, 2H), 7.44 (d, *J* = 6.2 Hz, 2H), 7.42 – 7.35 (m, 3H), 7.28 (d, *J* = 7.6 Hz, 1H), 7.21 (t, *J* = 7.3 Hz, 1H), 7.07 (s, 1H), 6.86 (d, *J* = 7.7 Hz, 2H), 3.80 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.8, 156.7, 154.9, 153.2, 135.2, 130.7, 130.1, 128.9, 128.7, 128.2, 128.0, 127.6, 124.8, 123.0, 122.1, 121.3, 114.2, 111.2, 105.3, 55.4; HRMS calculated for $C_{22}H_{17}NO_3$ $[M+H]^+$: 344.1287, found 344.1278; Purification: Flash chromatography; Eluent: hexane/EtOAc (9/1); R_f = 0.49.

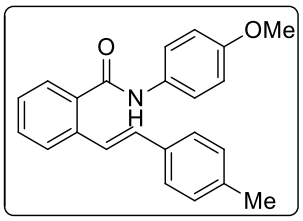
(*E*)-*N*-(4-Methoxyphenyl)-2-styrylbenzamide (3am)



Pale yellow solid (93 mg, 71% yield); mp: 182-183 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.73 (d, *J* = 7.8 Hz, 1H), 7.62 (dd, *J* = 7.5, 2.1 Hz, 1H), 7.57 – 7.44 (m, 7H), 7.35 (dd, *J* = 14.8, 7.4 Hz, 3H), 7.27 (d, *J* = 7.5 Hz, 1H), 7.10 (d, *J* = 16.2 Hz, 1H), 6.90 (d, *J* = 8.5 Hz, 2H), 3.81 (s, 3H);

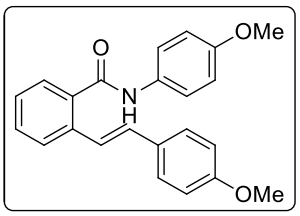
^{13}C NMR (126 MHz, $CDCl_3$) δ 167.3, 156.6, 136.8, 135.7, 135.5, 131.9, 130.9, 130.6, 128.7, 128.1, 127.8, 127.6, 126.8, 126.5, 125.6, 121.8, 114.3, 55.5; HRMS calculated for $C_{22}H_{19}NO_2$ $[M+H]^+$: 330.1494, found 330.1482; Purification: Flash chromatography; Eluent: hexane/EtOAc (9/1); R_f = 0.36.

(*E*)-*N*-(4-Methoxyphenyl)-2-(4-methylstyryl)benzamide (3an)



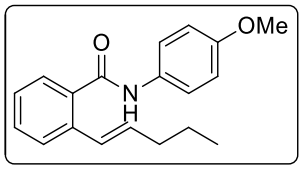
Pale yellow solid (96 mg, 70% yield); mp: 189-190 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.71 (d, *J* = 7.7 Hz, 1H), 7.61 (d, *J* = 7.4 Hz, 1H), 7.53 – 7.44 (m, 5H), 7.39 (d, *J* = 7.4 Hz, 2H), 7.33 (t, *J* = 7.3 Hz, 1H), 7.14 (d, *J* = 7.4 Hz, 2H), 7.07 (d, *J* = 16.2 Hz, 1H), 6.89 (d, *J* = 7.9 Hz, 2H), 3.81 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.3, 156.6, 138.1, 135.8, 135.4, 134.1, 131.9, 130.9, 130.6, 129.4, 127.9, 127.4, 126.7, 126.4, 124.6, 121.8, 114.2, 55.5, 21.3; HRMS calculated for $C_{23}H_{21}NO_2$ $[M+H]^+$: 344.1651, found 344.1645; Purification: Flash chromatography; Eluent: hexane/EtOAc (9/1); R_f = 0.42.

(*E*)-*N*-(4-Methoxyphenyl)-2-(4-methoxystyryl)benzamide (3ao)



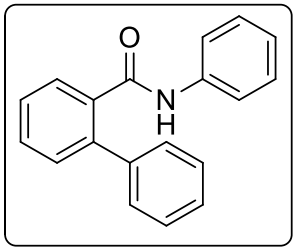
Pale yellow solid (87.6 mg, 61% yield); mp: 192-193 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.70 (d, *J* = 7.8 Hz, 1H), 7.61 (d, *J* = 7.5 Hz, 1H), 7.45 (ddd, *J* = 33.9, 22.1, 12.4 Hz, 7H), 7.33 (d, *J* = 7.4 Hz, 1H), 7.05 (d, *J* = 16.2 Hz, 1H), 6.88 (dd, *J* = 12.1, 8.7 Hz, 4H), 3.81 (d, *J* = 3.6 Hz, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.4, 159.6, 156.6, 136.0, 135.2, 131.5, 131.0, 130.5, 129.6, 128.1, 127.8, 127.2, 126.3, 123.4, 121.7, 114.2, 114.1, 55.5, 55.3; HRMS calculated for $C_{23}H_{21}NO_3$ $[M+H]^+$: 360.1600 , found 360.1597; Purification: Flash chromatography; Eluent: hexane/EtOAc (9/1); R_f = 0.34.

(*E*)-*N*-(4-Methoxyphenyl)-2-(pent-1-en-1-yl)benzamide (3ap)



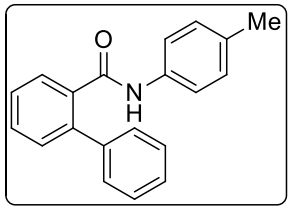
Pale brown solid (97 mg, 82% yield); mp: 100-101 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 (d, *J* = 7.6 Hz, 1H), 7.55 – 7.50 (m, 3H), 7.41 (dd, *J* = 14.7, 6.9 Hz, 2H), 7.29 (t, *J* = 7.5 Hz, 1H), 6.91 (d, *J* = 8.7 Hz, 2H), 6.76 (d, *J* = 15.7 Hz, 1H), 6.28 – 6.20 (m, 1H), 3.82 (s, 3H), 2.21 (q, *J* = 7.1 Hz, 2H), 1.55 – 1.42 (m, 2H), 0.94 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.4, 156.5, 136.1, 134.9, 134.8, 131.1, 130.4, 127.8, 127.3, 127.0, 126.7, 121.5, 114.2, 55.5, 35.2, 22.5, 13.7; HRMS calculated for $C_{19}H_{21}NO_2$ $[M+H]^+$: 296.1651, found 296.1644; Purification: Flash chromatography; Eluent: hexane/EtOAc (93/7); R_f = 0.35.

4'-Methoxy-*N*-phenyl-[1,1'-biphenyl]-2-carboxamide (3ba)



Colorless solid (97 mg, 89% yield); mp: 105-106 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.90 (d, *J* = 7.6 Hz, 1H), 7.55 (td, *J* = 7.5, 1.3 Hz, 1H), 7.51 – 7.40 (m, 7H), 7.23 (t, *J* = 7.9 Hz, 2H), 7.10 (d, *J* = 7.6 Hz, 2H), 7.05 (t, *J* = 7.4 Hz, 1H), 6.88 (s, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.1, 139.9, 139.5, 137.5, 135.2, 130.7, 130.3, 129.6, 129.0, 128.8, 128.1, 127.9, 124.4, 119.9; HRMS calculated for $C_{19}H_{15}NO$ $[M+H]^+$: 274.1232, found 274.1221; Purification: Flash chromatography; Eluent: hexane/EtOAc (93/7); R_f = 0.5.

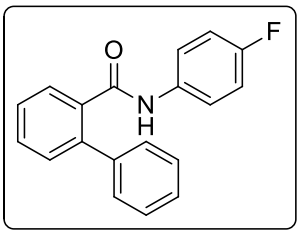
4'-Methoxy-*N*-(*p*-tolyl)-[1,1'-biphenyl]-2-carboxamide (3ca)



Colorless solid (95 mg, yield 83%); mp: 129-130 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.89 (d, *J* = 7.6 Hz, 1H), 7.54 (dt, *J* = 7.5, 3.8 Hz, 1H), 7.50 – 7.38 (m, 7H), 7.03 (d, *J* = 8.3 Hz, 2H), 6.98 (d, *J* = 8.4 Hz, 2H), 6.82 (s, 1H), 2.27 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.0,

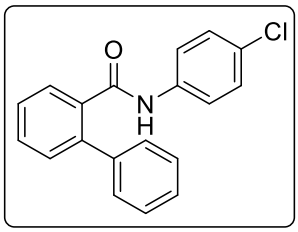
140.0, 139.5, 135.3, 134.9, 134.1, 130.6, 130.3, 129.6, 129.3, 129.0, 128.8, 128.1, 127.9, 120.0, 20.8; HRMS calculated for $C_{20}H_{17}NO$ $[M+H]^+$: 288.1388, found 288.1383; Purification: Flash chromatography; Eluent: hexane/EtOAc (93/7); R_f = 0.48.

***N*-(4-Fluorophenyl)-[1,1'-biphenyl]-2-carboxamide (3da)**



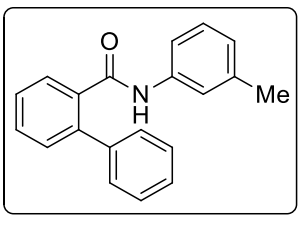
Colorless solid (107 mg, yield 92%); mp: 150-151 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.5 Hz, 1H), 7.54 (d, *J* = 7.4 Hz, 1H), 7.51 – 7.38 (m, 7H), 7.04 (dd, *J* = 7.6, 5.1 Hz, 2H), 6.90 (t, *J* = 8.5 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.1, 159.4 (d, J_{C-F} = 243.7 Hz), 139.9, 139.5, 135.0, 133.5, 130.7, 130.3, 129.5, 129.0, 128.8, 128.1, 127.9, 121.7 (d, J_{C-F} = 7.9 Hz), 115.4 (d, J_{C-F} = 22.5 Hz); ^{1}H NMR and ^{13}C NMR results matched with the reference **3**; Purification: Flash chromatography; Eluent: hexane/EtOAc (93/7); R_f = 0.46.

***N*-(4-Chlorophenyl)-4'-methoxy-[1,1'-biphenyl]-2-carboxamide (3ea)**



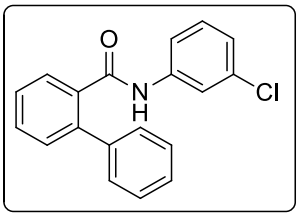
Colorless solid (104 mg, 85% yield); mp: 146-147 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.91 (d, *J* = 6.7 Hz, 1H), 7.65 – 7.38 (m, 8H), 7.20 (d, *J* = 8.3 Hz, 2H), 7.06 (d, *J* = 8.5 Hz, 2H), 6.89 (s, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.0, 139.9, 139.5, 136.1, 134.8, 130.9, 130.3, 129.6, 129.3, 129.0, 128.8, 128.8, 128.2, 128.0, 121.0; HRMS calculated for $C_{19}H_{14}NOCl$ $[M+H]^+$: 308.0842, found 308.0838; Purification: Flash chromatography; Eluent: hexane/EtOAc (93/7); R_f = 0.37.

***N*-(*m*-Tolyl)-[1,1'-biphenyl]-2-carboxamide (3fa)**



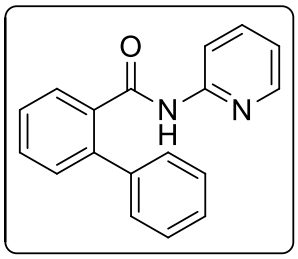
Colorless solid (85 mg, 74% yield); mp: 146-147 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.91 – 7.87 (m, 1H), 7.54 (dd, *J* = 7.5, 1.3 Hz, 1H), 7.50 – 7.41 (m, 7H), 7.10 (t, *J* = 7.8 Hz, 1H), 7.04 (s, 1H), 6.89 – 6.82 (m, 2H), 6.79 (d, *J* = 7.9 Hz, 1H), 2.28 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.1, 139.9, 139.5, 138.7, 137.4, 135.3, 130.6, 130.3, 129.5, 128.9, 128.8, 128.6, 128.0, 127.8, 125.2, 120.6, 116.9, 21.4; ^{1}H NMR and ^{13}C NMR results matched with the reference **3**; Purification: Flash chromatography; Eluent: hexane/EtOAc (93/7); R_f = 0.47.

***N*-(3-Chlorophenyl)-[1,1'-biphenyl]-2-carboxamide (3ga)**



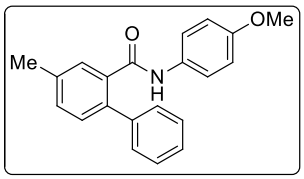
Colorless solid (98 mg, 80% yield); mp: 133-134 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.89 (d, *J* = 7.6 Hz, 1H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.52 – 7.39 (m, 7H), 7.28 (s, 1H), 7.12 (t, *J* = 8.0 Hz, 1H), 7.02 (d, *J* = 7.9 Hz, 1H), 6.91 – 6.81 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.1, 139.8, 139.6, 138.6, 134.8, 134.5, 131.0, 130.4, 129.8, 129.7, 129.1, 128.8, 128.3, 128.0, 124.5, 120.0, 117.8; ^{1}H NMR and ^{13}C NMR results matched with the reference **3**; Purification: Flash chromatography; Eluent: hexane/EtOAc (93/7); R_f = 0.35.

***N*-(Pyridin-2-yl)-[1,1'-biphenyl]-2-carboxamide (3ha)**



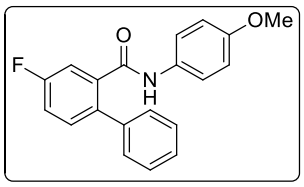
Pale brown solid (71 mg, 65% yield); mp: 168-169 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.01 (s, *J* = 42.3 Hz, 1H), 8.25 (d, *J* = 8.2 Hz, 1H), 7.69 – 7.57 (m, 3H), 7.52 (t, *J* = 7.5 Hz, 1H), 7.43 – 7.31 (m, 7H), 6.89 – 6.81 (m, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.4, 151.5, 147.3, 140.0, 139.7, 138.3, 135.7, 130.5, 130.5, 128.6, 128.6, 127.8, 127.4, 119.5, 114.0; HRMS calculated for $C_{18}H_{14}N_2O$ $[M+H]^+$: 275.1184 found 275.1180; Purification: Flash chromatography; Eluent: hexane/EtOAc (9/1); R_f = 0.4.

***N*-(4-Methoxyphenyl)-4-methyl-[1,1'-biphenyl]-2-carboxamide (3ia)**



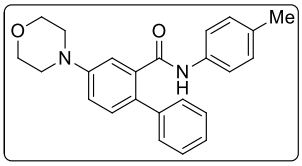
Colorless solid (90 mg, 71%); mp: 147-148 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.70 (s, 1H), 7.43 (dt, *J* = 15.4, 6.9 Hz, 5H), 7.33 (q, *J* = 7.7 Hz, 2H), 6.98 (d, *J* = 7.8 Hz, 2H), 6.76 (d, *J* = 8.2 Hz, 3H), 3.75 (s, 3H), 2.45 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.3, 156.5, 140.0, 137.8, 136.6, 135.1, 131.3, 130.6, 130.2, 130.0, 128.9, 129.0, 127.8, 121.8, 113.9, 55.4, 21.0; HRMS calculated for $C_{21}H_{19}NO_2$ $[M+Na]^+$: 340.1313, found 340.1314; Purification: Flash chromatography; Eluent: hexane/EtOAc (93/7); R_f = 0.36.

4-Fluoro-*N*-(4-methoxyphenyl)-[1,1'-biphenyl]-2-carboxamide (3ja)



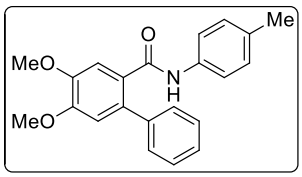
Colorless solid (95 mg, 74%); mp: 162-163 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63 – 7.57 (m, 1H), 7.47 – 7.38 (m, 6H), 7.25 – 7.21 (m, 1H), 6.97 (d, *J* = 8.9 Hz, 2H), 6.76 (d, *J* = 8.8 Hz, 3H), 3.75 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.6, 162.0 (d, *J* = 248.6 Hz), 156.6, 139.0, 136.9 (d, *J* = 7.0 Hz), 135.5 (d, *J* = 3.4 Hz), 132.1 (d, *J* = 7.7 Hz), 130.1, 129.0, 128.8, 128.1, 121.9, 117.5 (d, *J* = 21.3 Hz), 116.3 (d, *J* = 23.1 Hz), 113.9, 55.3; HRMS calculated for $C_{20}H_{16}NO_2F$ $[M+Na]^+$: 344.1063, found 344.1065; Purification: Flash chromatography; Eluent: hexane/EtOAc (93/7); R_f = 0.38.

4-Morpholino-*N*-(*p*-tolyl)-[1,1'-biphenyl]-2-carboxamide (3ka)



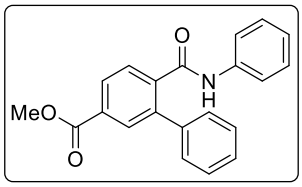
Colorless solid (125 mg, 81% yield); mp: 184-185 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 – 7.38 (m, 5H), 7.34 (dd, *J* = 12.4, 7.7 Hz, 2H), 7.06 (dd, *J* = 8.5, 2.4 Hz, 1H), 7.02 (d, *J* = 8.2 Hz, 2H), 6.96 (d, *J* = 8.2 Hz, 2H), 6.86 (s, 1H), 3.91 – 3.85 (m, 4H), 3.29 – 3.22 (m, 4H), 2.26 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.3, 150.6, 139.7, 135.9, 134.9, 134.0, 131.2, 130.4, 129.3, 128.9, 128.9, 127.5, 120.0, 117.4, 115.9, 66.7, 48.7, 20.8; HRMS calculated for $C_{24}H_{24}N_2O_2$ $[M+Na]^+$: 395.1735, found 395.1737; Purification: Flash chromatography; Eluent: hexane/EtOAc (88/12); R_f = 0.35.

4,5-Dimethoxy-*N*-(*p*-tolyl)-[1,1'-biphenyl]-2-carboxamide (3la)



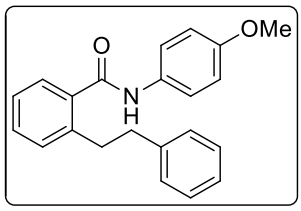
Colorless solid (86 mg, 62% yield); mp: 173-174 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.51 (s, 1H), 7.45 (t, *J* = 7.0 Hz, 4H), 7.01 (d, *J* = 8.3 Hz, 2H), 6.92 (d, *J* = 8.4 Hz, 2H), 6.84 (s, 2H), 3.98 (s, 3H), 3.94 (s, 3H), 2.26 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.2, 150.4, 148.4, 140.0, 135.0, 133.8, 132.9, 129.2, 129.1, 129.0, 128.0, 127.0, 119.7, 112.7, 112.4, 56.1, 56.1, 20.8, HRMS calculated for $C_{22}H_{21}NO_3$ $[M+Na]^+$: 370.1419, found 370.1416; Purification: Flash chromatography; Eluent: hexane/EtOAc (88/12); R_f = 0.39.

Methyl 6-(phenylcarbamoyl)-[1,1'-biphenyl]-3-carboxylate (3ma)



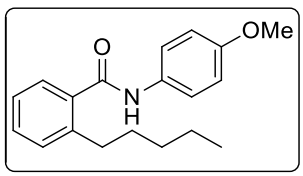
Colorless solid (94 mg, 71% yield); mp: 154-155 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.13 (dd, *J* = 5.9, 1.7 Hz, 2H), 7.98 – 7.93 (m, 1H), 7.54 – 7.40 (m, 5H), 7.24 (t, *J* = 7.9 Hz, 2H), 7.13 – 7.05 (m, 3H), 6.93 (s, 1H), 3.96 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.2, 166.2, 139.7, 139.0, 138.9, 137.2, 132.0, 131.5, 129.8, 129.1, 128.9, 128.8, 128.5, 124.7, 120.0, 52.5; ^{1}H NMR and ^{13}C NMR results matched with the reference **3**; Purification: Flash chromatography; Eluent: hexane/EtOAc (9/1); R_f = 0.45.

***N*-(4-Methoxyphenyl)-2-phenethylbenzamide (5a)**



Colorless solid (128 mg, 97% yield); mp:147-148 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48 – 7.36 (m, 4H), 7.28 (dd, *J* = 11.9, 8.4 Hz, 2H), 7.20 (t, *J* = 7.0 Hz, 2H), 7.17 – 7.13 (m, 1H), 7.12 – 7.04 (m, 3H), 6.89 (d, *J* = 8.4 Hz, 2H), 3.81 (s, 3H), 3.14 (t, *J* = 7.5 Hz, 2H), 2.97 (t, *J* = 7.5 Hz, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.9, 156.5, 141.5, 139.9, 136.8, 130.9, 130.5, 130.1, 128.7, 128.3, 126.6, 126.1, 125.9, 121.8, 114.1, 55.5, 38.0, 35.1; HRMS calculated for $C_{22}H_{21}NO_2$ $[M+H]^+$: 332.1651, found 332.1646; Purification: Flash chromatography; Eluent: hexane/EtOAc (9/1); R_f = 0.41.

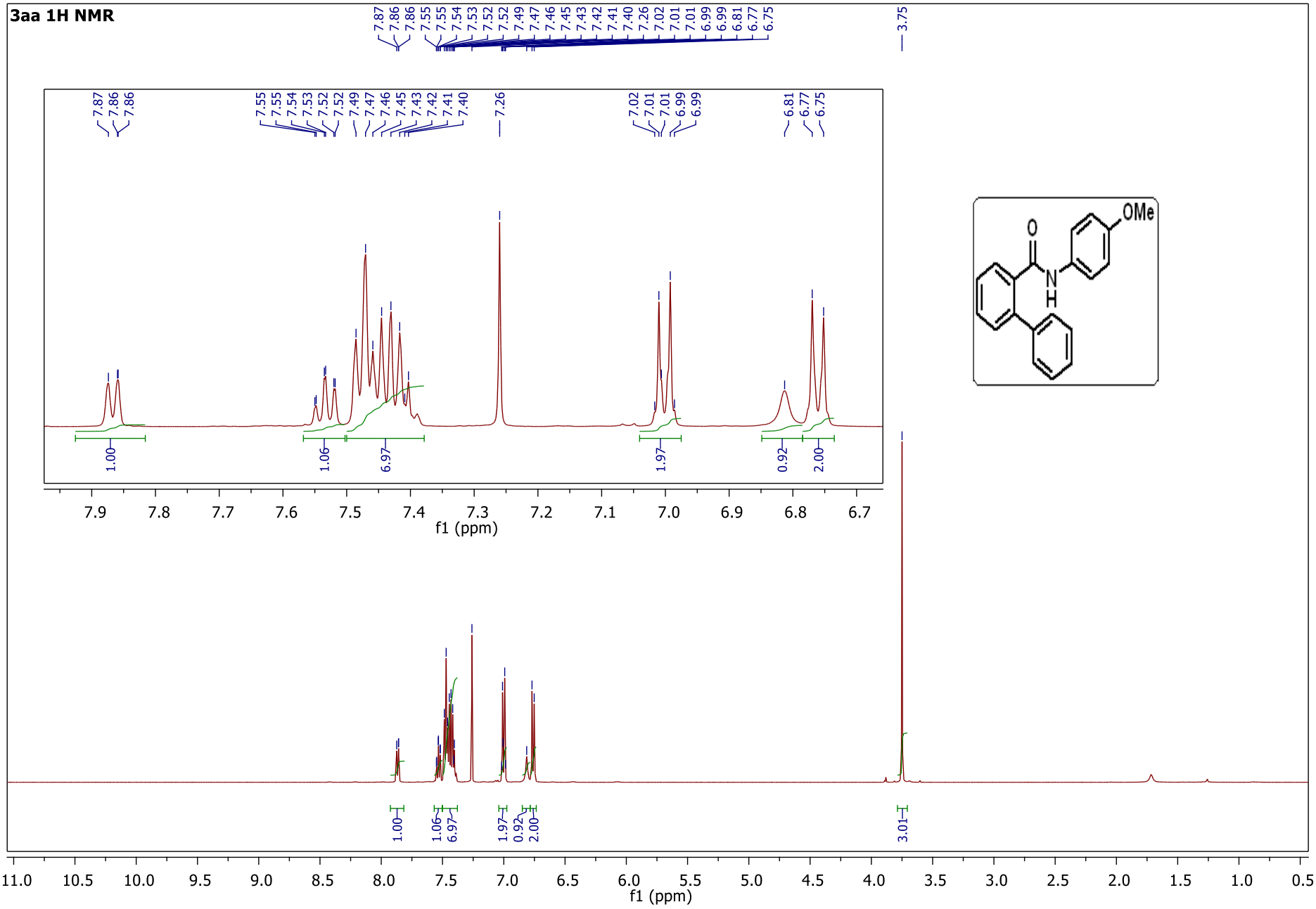
***N*-(4-Methoxyphenyl)-2-phenethylbenzamide (5b)**



Colorless solid (116 mg, 98% yield); mp: 116-117 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53 – 7.48 (m, 2H), 7.39 (dd, *J* = 34.2, 7.4 Hz, 3H), 7.28 (d, *J* = 7.6 Hz, 1H), 7.23 (s, 1H), 6.90 (d, *J* = 8.8 Hz, 2H), 3.81 (s, 3H), 2.85 – 2.77 (m, 2H), 1.65 – 1.60 (m, 2H), 1.31 (dd, *J* = 8.5, 5.3 Hz, 4H), 0.86 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.1, 156.6, 141.2, 136.5, 131.0, 130.2, 130.0, 126.6, 125.8, 121.8, 114.2, 55.49, 33.2, 31.7, 31.3, 22.5, 14.0; HRMS calculated for $C_{19}H_{23}NO_2$ $[M+Na]^+$: 320.1626, found 320.1622; Purification: Flash chromatography; Eluent: hexane/EtOAc (9/1); R_f = 0.52.

References

1. T. Miura, M. Morimoto, M. Yamauchi and M. Murakami, *J. Org. Chem.*, 2010, **75**, 5359.
2. S. Couty, B. Liegault, C. Meyer, J. Cossy, *Tetrahedron*, 2006, **62**, 3882.
3. V. H. Thorat, N. S. Upadhyay, C.-H. Cheng, *Adv. Synth. Catal.*, 2018, **360**, 4784.
4. M. J. Frisch; G. W. Trucks; H. B. Schlegel; G. E. Scuseria; M. A. Robb; J. R. Cheeseman; G. Scalmani; V. Barone; G. A. Petersson; H. Nakatsuji; X. Li; M. Caricato; A. V. Marenich; J. Bloino; B. G. Janesko; R. Gomperts; B. Mennucci; H. P. Hratchian; J. V. Ortiz; A. F. Izmaylov; J. L. Sonnenberg; Williams; F. Ding; F. Lipparini; F. Egidi; J. Goings; B. Peng; A. Petrone; T. Henderson; D. Ranasinghe; V. G. Zakrzewski; J. Gao; N. Rega; G. Zheng; W. Liang; M. Hada; M. Ehara; K. Toyota; R. Fukuda; J. Hasegawa; M. Ishida; T. Nakajima; Y. Honda; O. Kitao; H. Nakai; T. Vreven; K. Throssell; J. A. Montgomery Jr; J. E. Peralta; F. Ogliaro; M. J. Bearpark; J. J. Heyd; E. N. Brothers; K. N. Kudin; V. N. Staroverov; T. A. Keith; R. Kobayashi; J. Normand; K. Raghavachari; A. P. Rendell; J. C. Burant; S. S. Iyengar; J. Tomasi; M. Cossi; J. M. Millam; M. Klene; C. Adamo; R. Cammi; J. W. Ochterski; R. L. Martin; K. Morokuma; O. Farkas; J. B. Foresman; D. J. Fox, *Gaussian 16 Rev. C.01*; Wallingford, CT, 2016.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7.87
7.86
7.86
7.55
7.55
7.54
7.53
7.52
7.52
7.49
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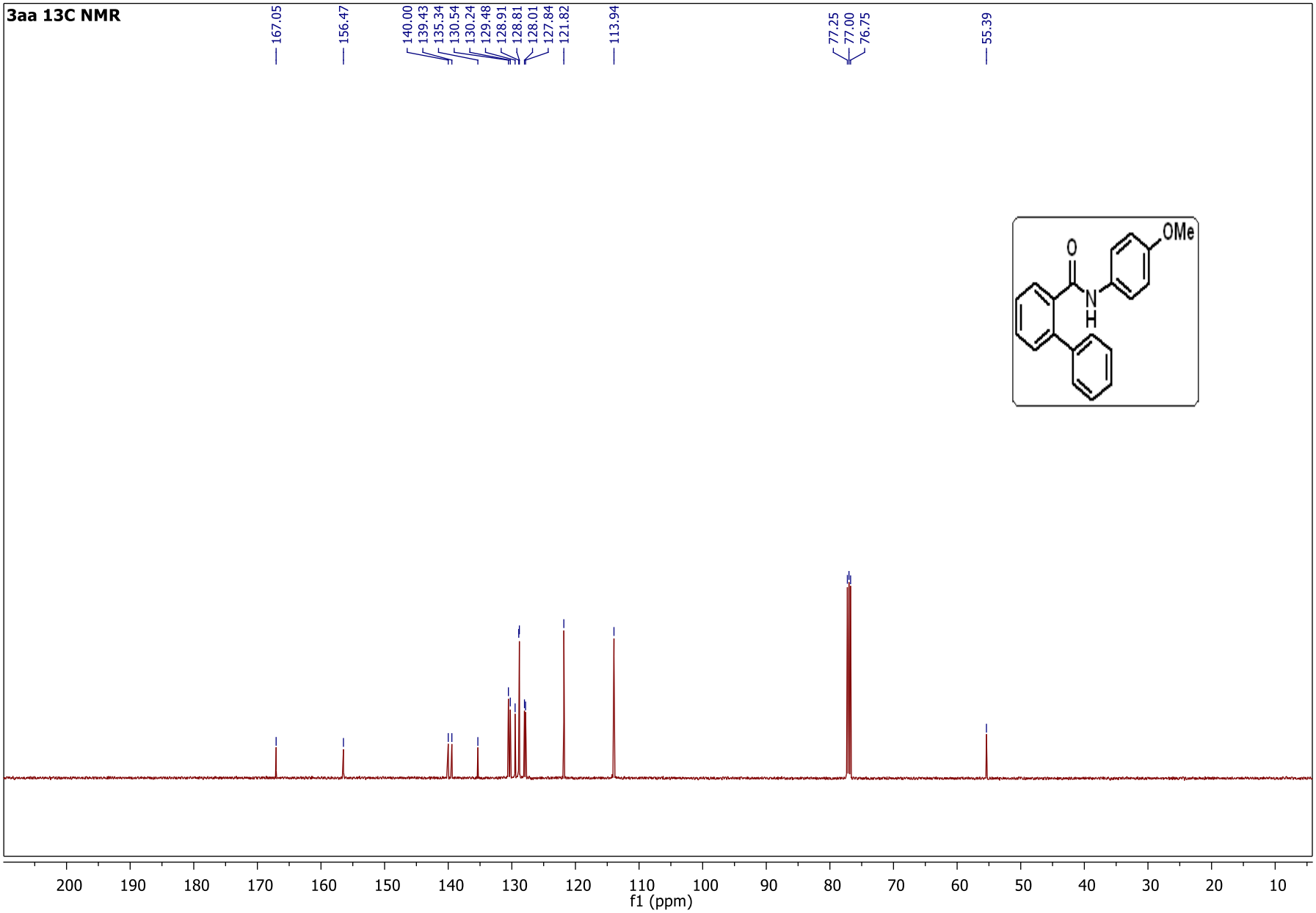
7.45
7.43
7.42
7.41
7.40
7.26
7.02
7.01
7.01
6.99
6.99
6.81
6.77
6.75
3.75
1.00
1.06
6.97
1.97
0.92
2.00
3.01
OMe
O
N
H
f1 (ppm)
f1 (ppm)

3aa 13C NMR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156.47
140.00
139.43
135.34
130.54
130.24
129.48
128.91
128.81
128.01
127.84
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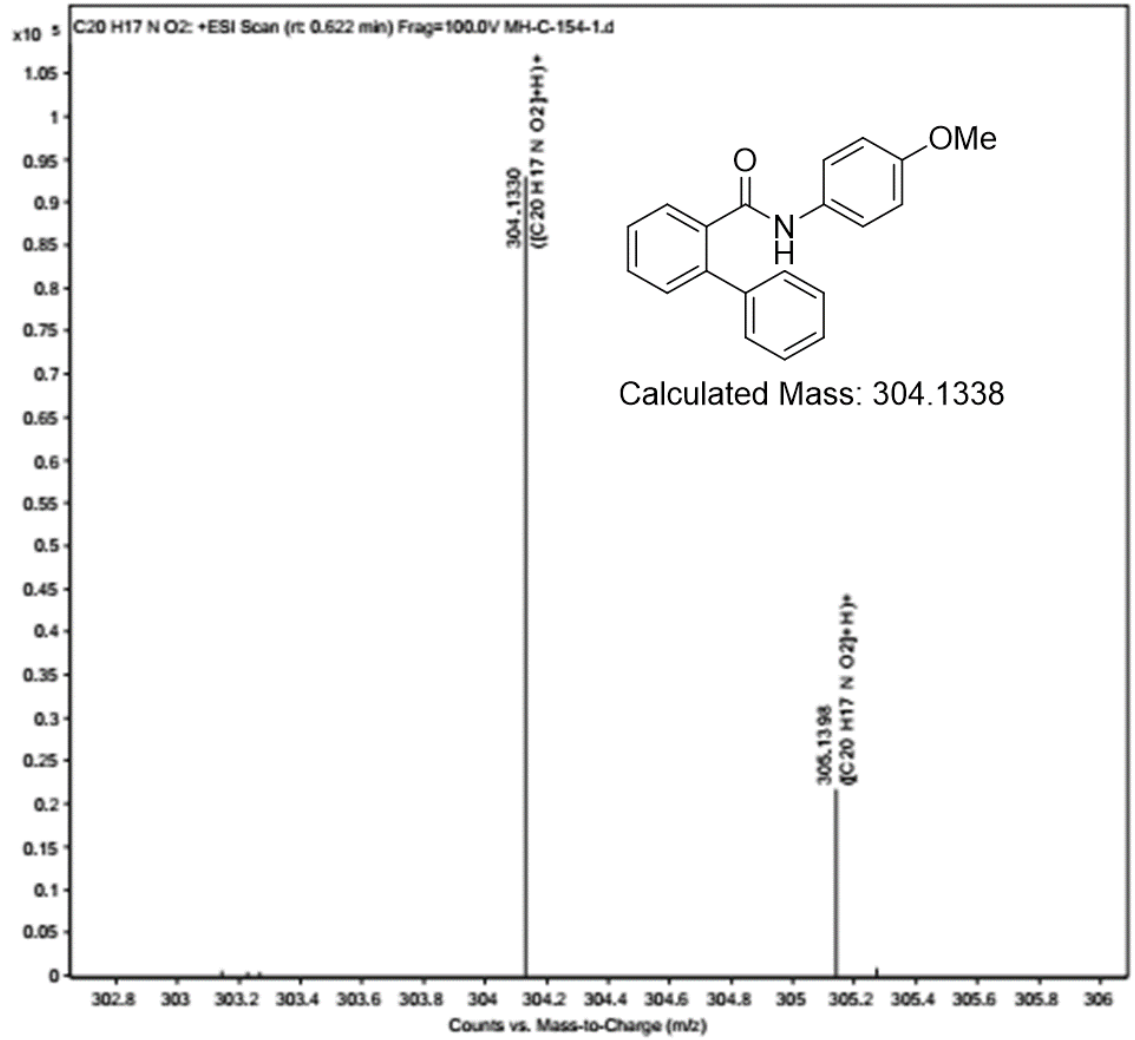
113.94
77.25
77.00
76.75
55.39
OMe
O
N
H
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

Sample Name	MH/C-154-1	**Position**		**Instrument Name**	Instrument 1
User Name		**Inj Vol**	Unknown / Injection Program	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	MH-C-154-1.d
ACQ Method	Demo.m	**Comment**		**Acquired Time**	10-04-2018 02:52:06 (UTC+05:30)



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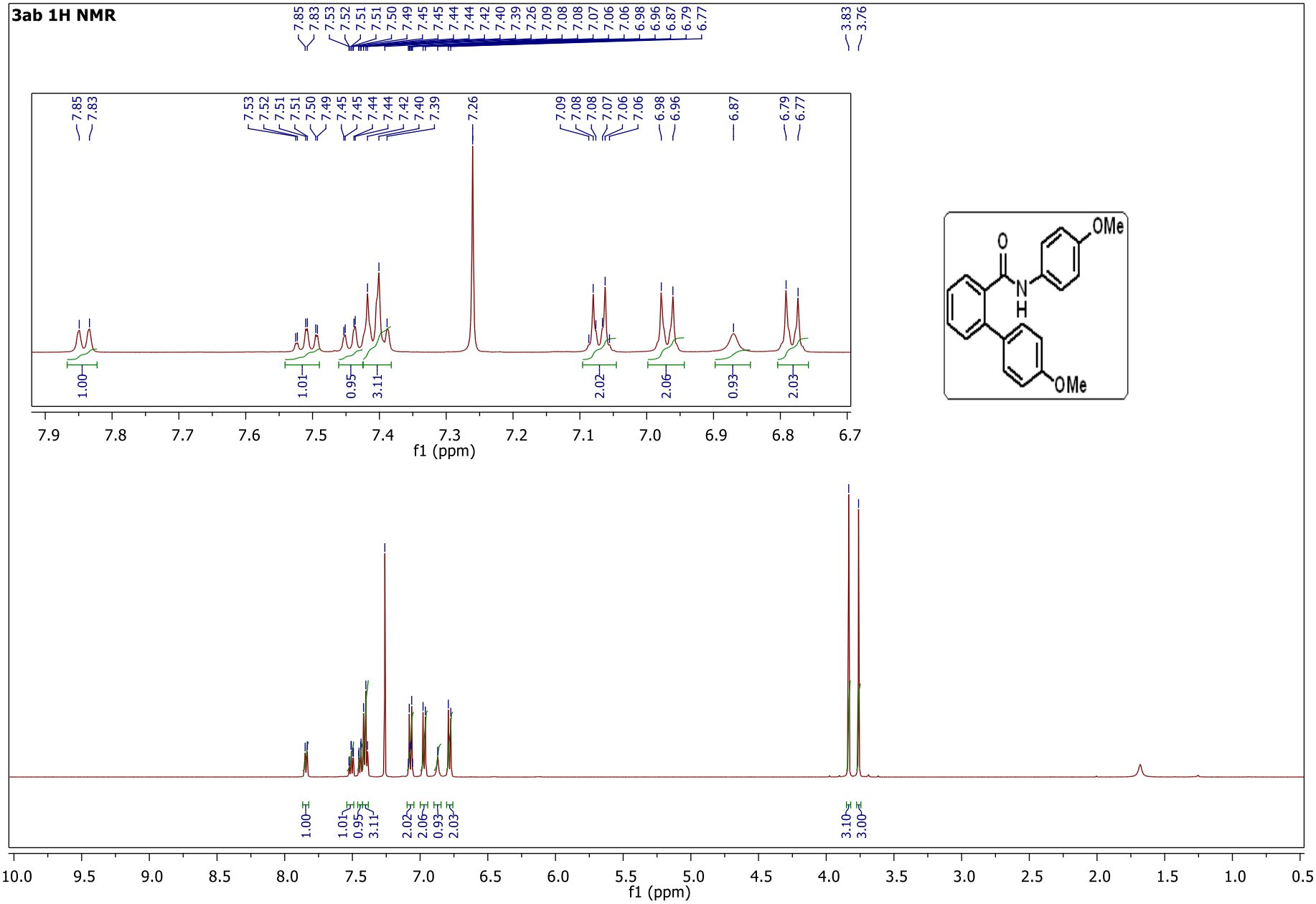
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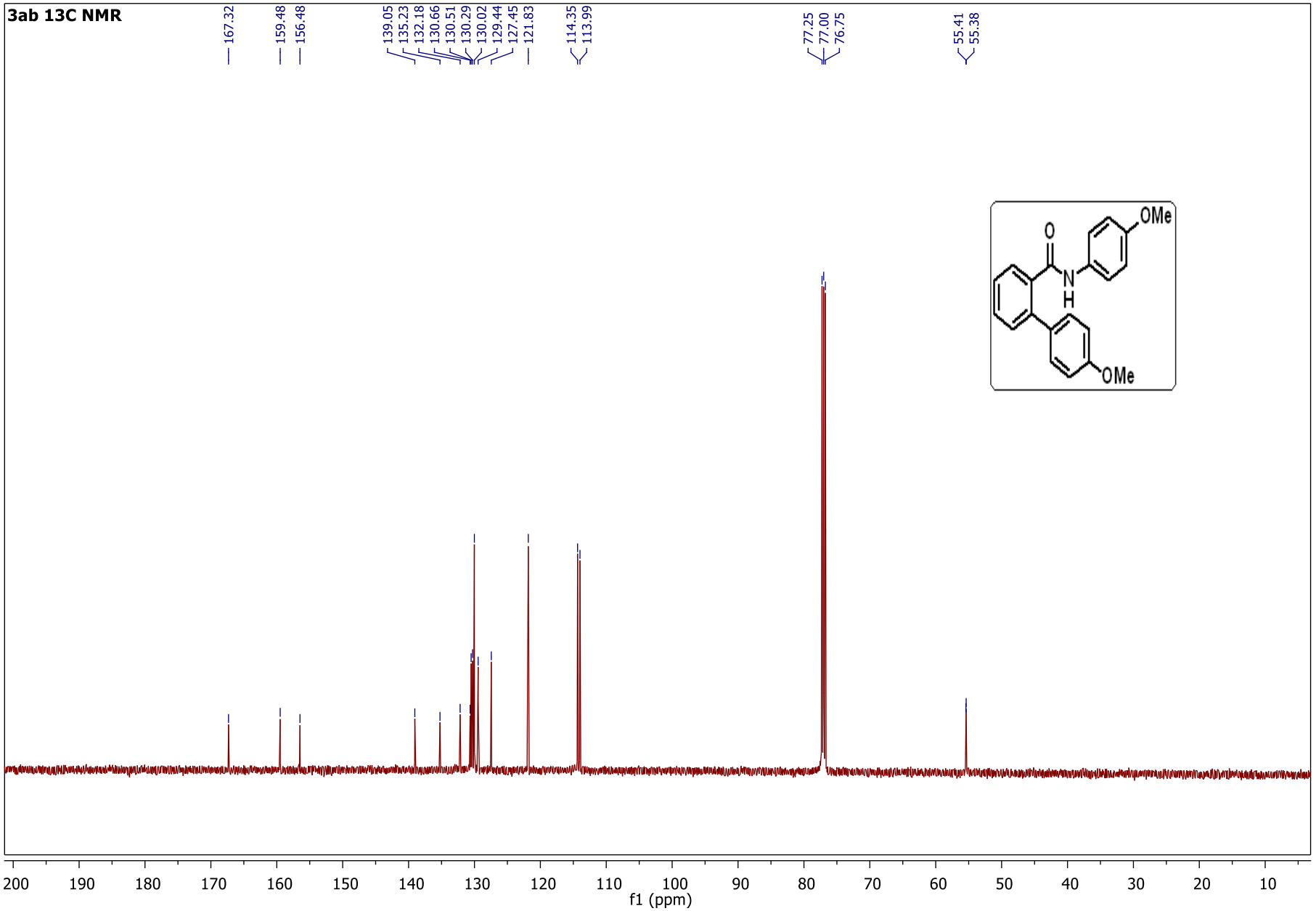
7.83
7.53
7.52
7.51
7.51
7.50
7.49
7.45
7.45
7.44
7.44
7.42
7.40
7.39
7.26
7.09
7.08
7.08
7.07
7.06
7.06
6.98
6.96
6.87
6.79
6.77
3.83
3.76
1.00
1.01
0.95
3.11
2.02
2.06
0.93
2.03
3.10
3.00
f1 (ppm)
OMe
O
N
H
OMe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159.48
156.48
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130.66
130.51
130.29
130.02
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121.83
114.35
113.99
77.25
77.00
76.75
55.41
55.38
O
N
H
OMe
OMe
200
190
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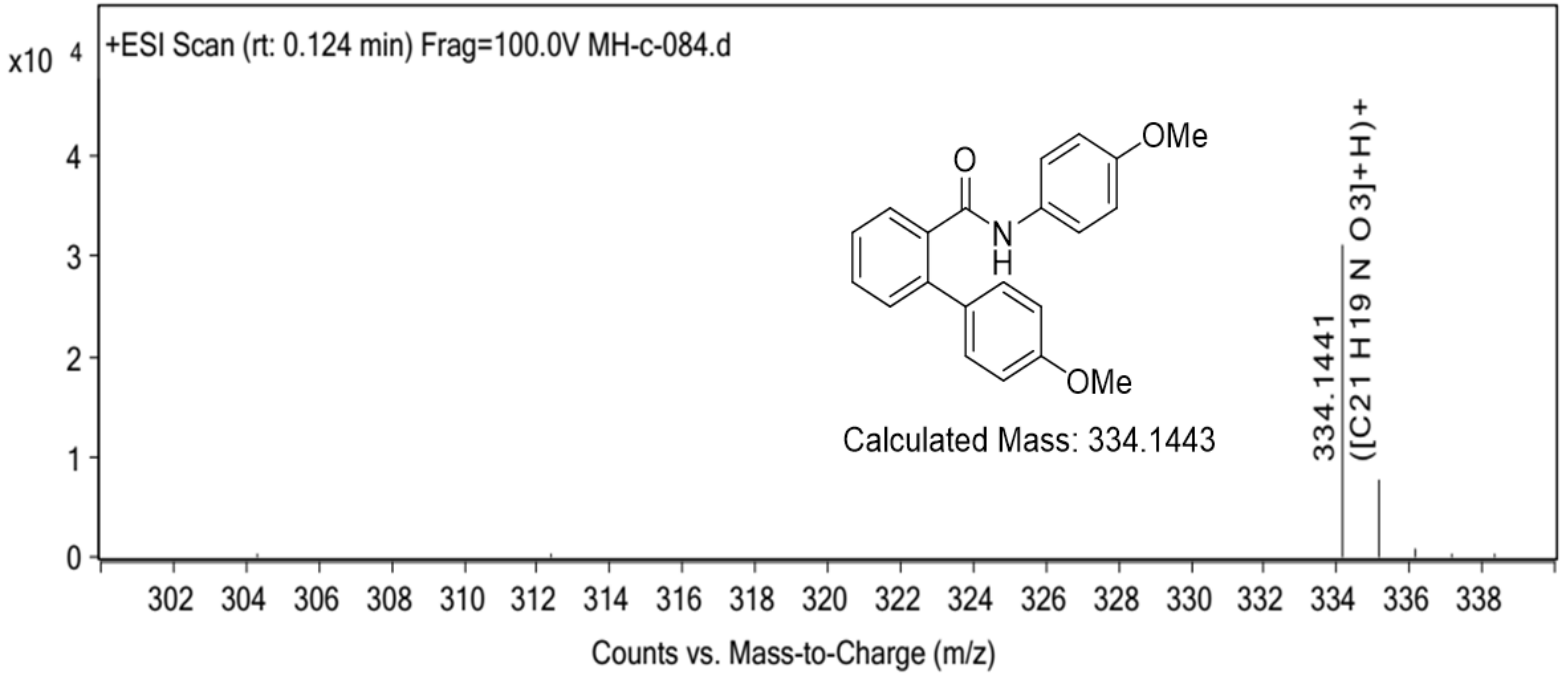
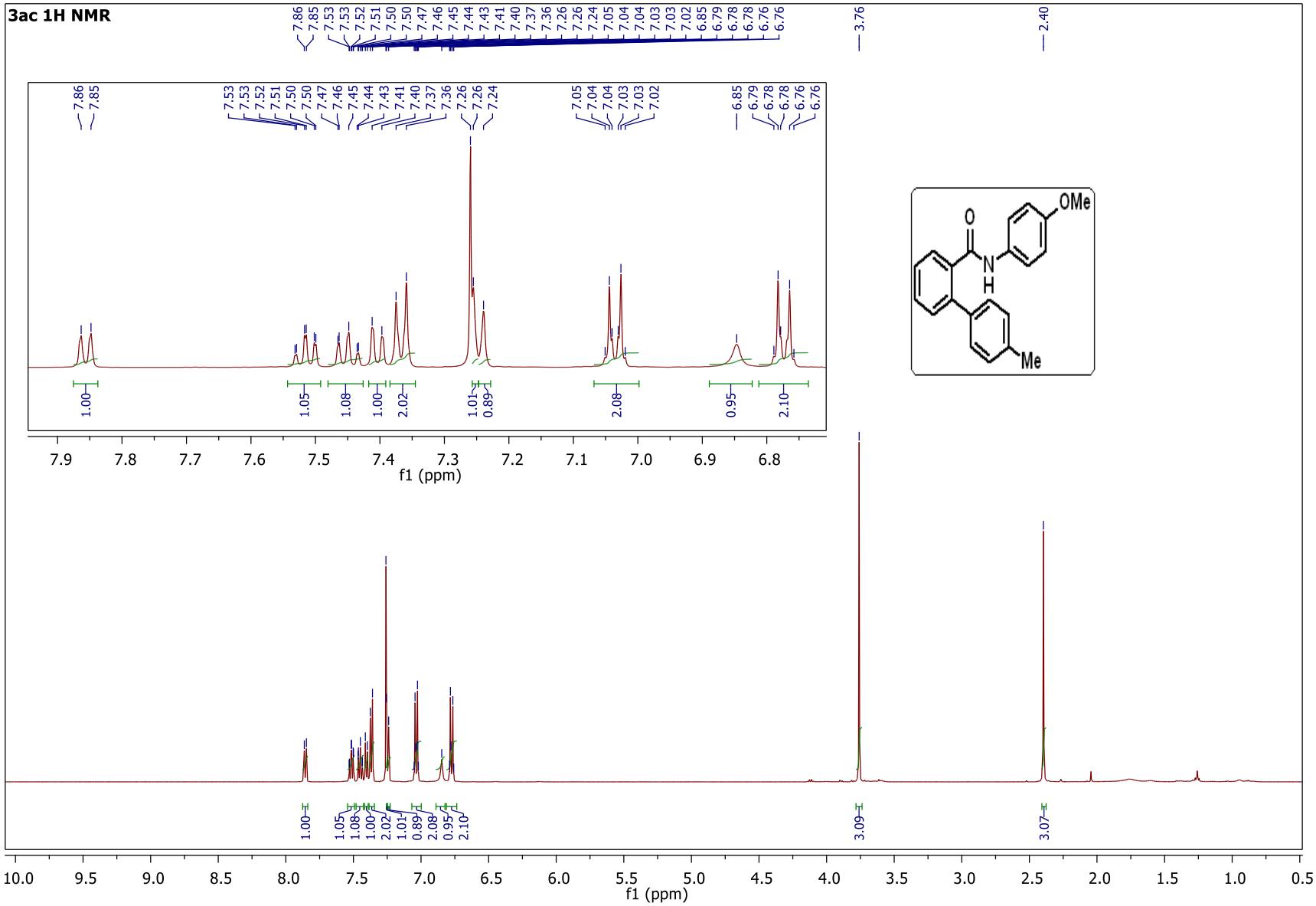
80
70
60
50
40
30
20
10
f1 (ppm)

Data File	MH-c-084.d	**Sample Name**	MH/c-083
Sample Type	Sample	**Position**	
Instrument Name	Instrument 1	**User Name**	
Acq Method	Demo.m	**Acquired Time**	14-03-2018 03:04:16 (UTC+05:30)
IRM Calibration Status	Success	**DA Method**	Default.m
Comment			
Sample Group		**Info.**	
Stream Name		**Acquisition Time (Local)**	14-03-2018 03:04:16 (UTC+05:30)
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.08.00 (B8058.0)	**TOF Driver Version**	8.00.00
TOF Firmware Version	17.698	**Tune Mass Range Max.**	3200

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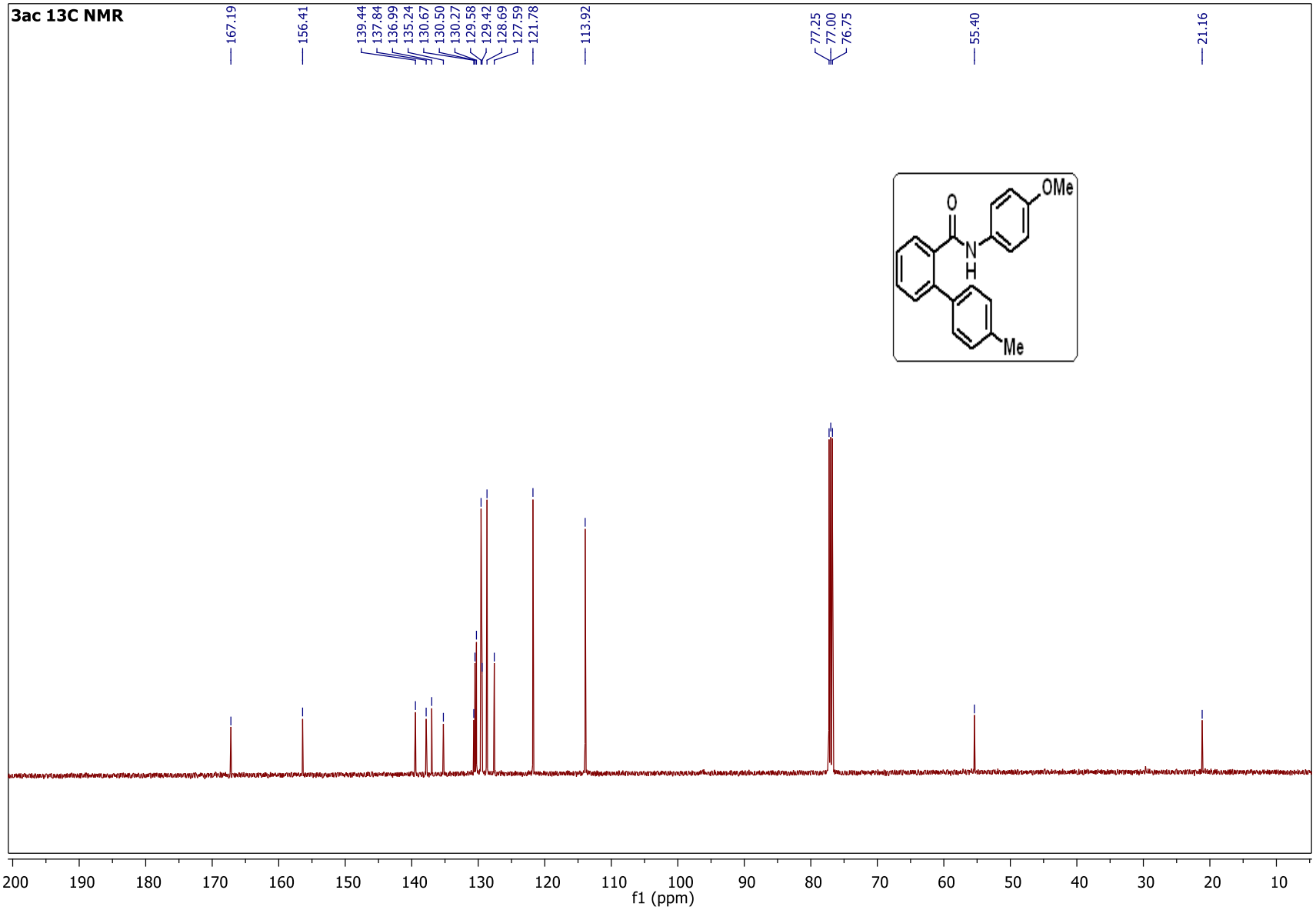
3.76
2.40
1.00 1.05 1.08 1.00 2.02 1.01 0.89 2.08 0.95 2.10
3.09
3.07
f1 (ppm)
OMe
O
N
H
Me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139.44
137.84
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130.67
130.50
130.27
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128.69
127.59
121.78
113.92
77.25
77.00
76.75
55.40
21.16
OMe
O
N
H
Me
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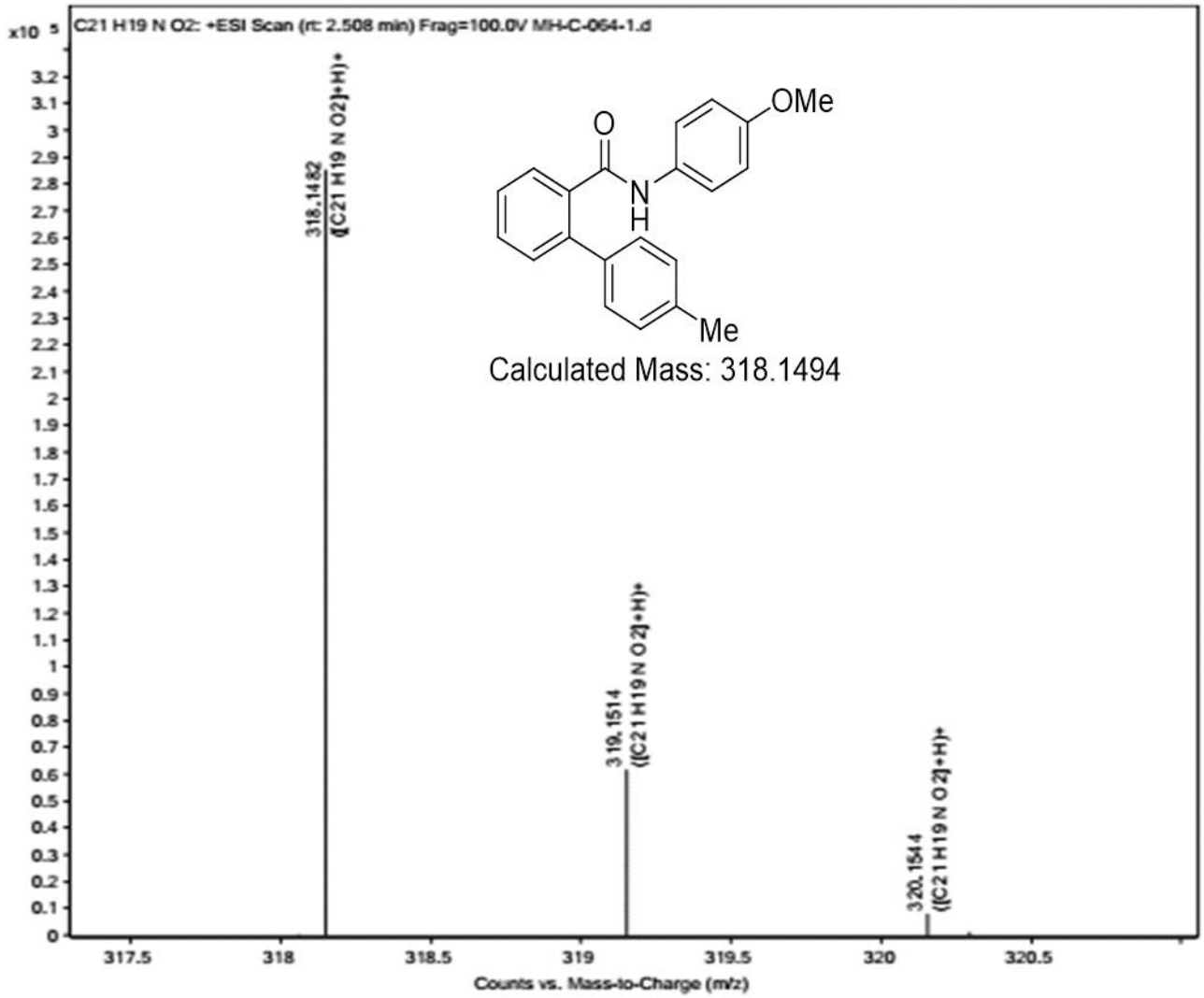
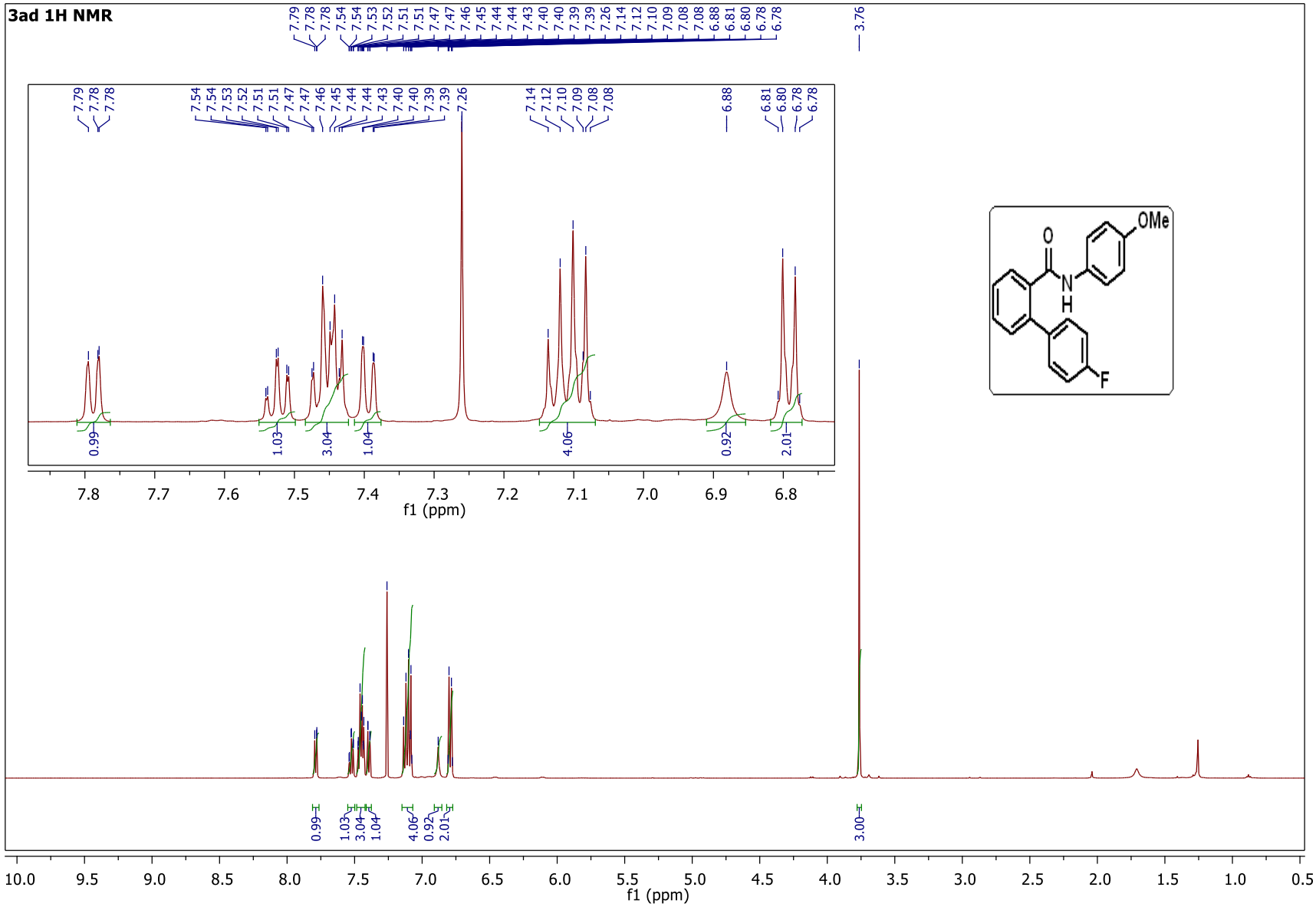
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

Sample Name	MH/C-064	**Position**		**Instrument Name**	Instrument 1
User Name		**Inj Vol**	Unknown / Injection Program	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	MH-C-064-1.d
ACQ Method	Demo.m	**Comment**		**Acquired Time**	10-04-2018 03:28:48 (UTC+05:30)

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3.76
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f1 (ppm)
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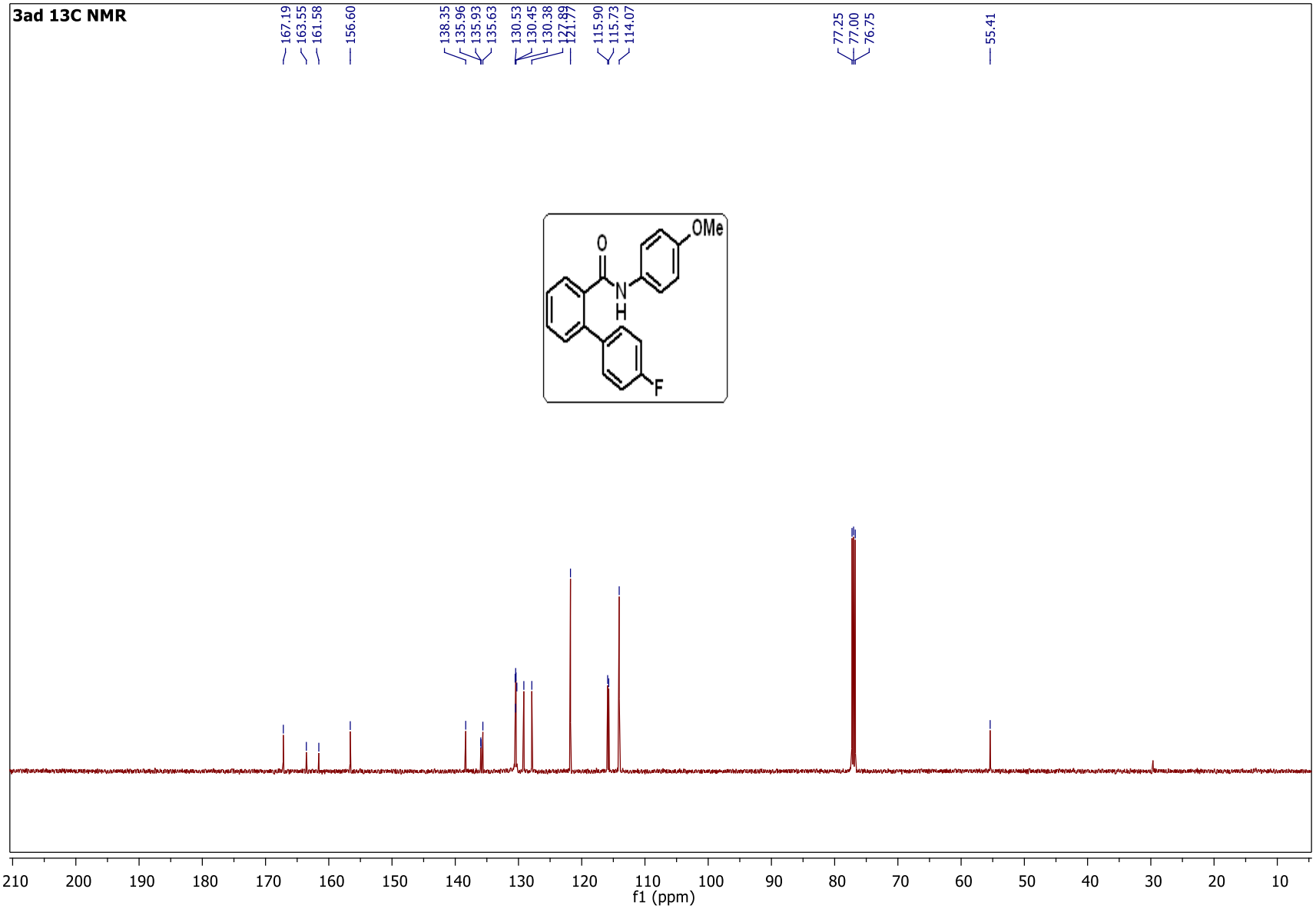
OMe
O
N
H
F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161.58
156.60
138.35
135.96
135.93
135.63
130.53
130.45
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121.77
115.90
115.73
114.07
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77.00
76.75
55.41
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O
N
H
F
210
200
190
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130
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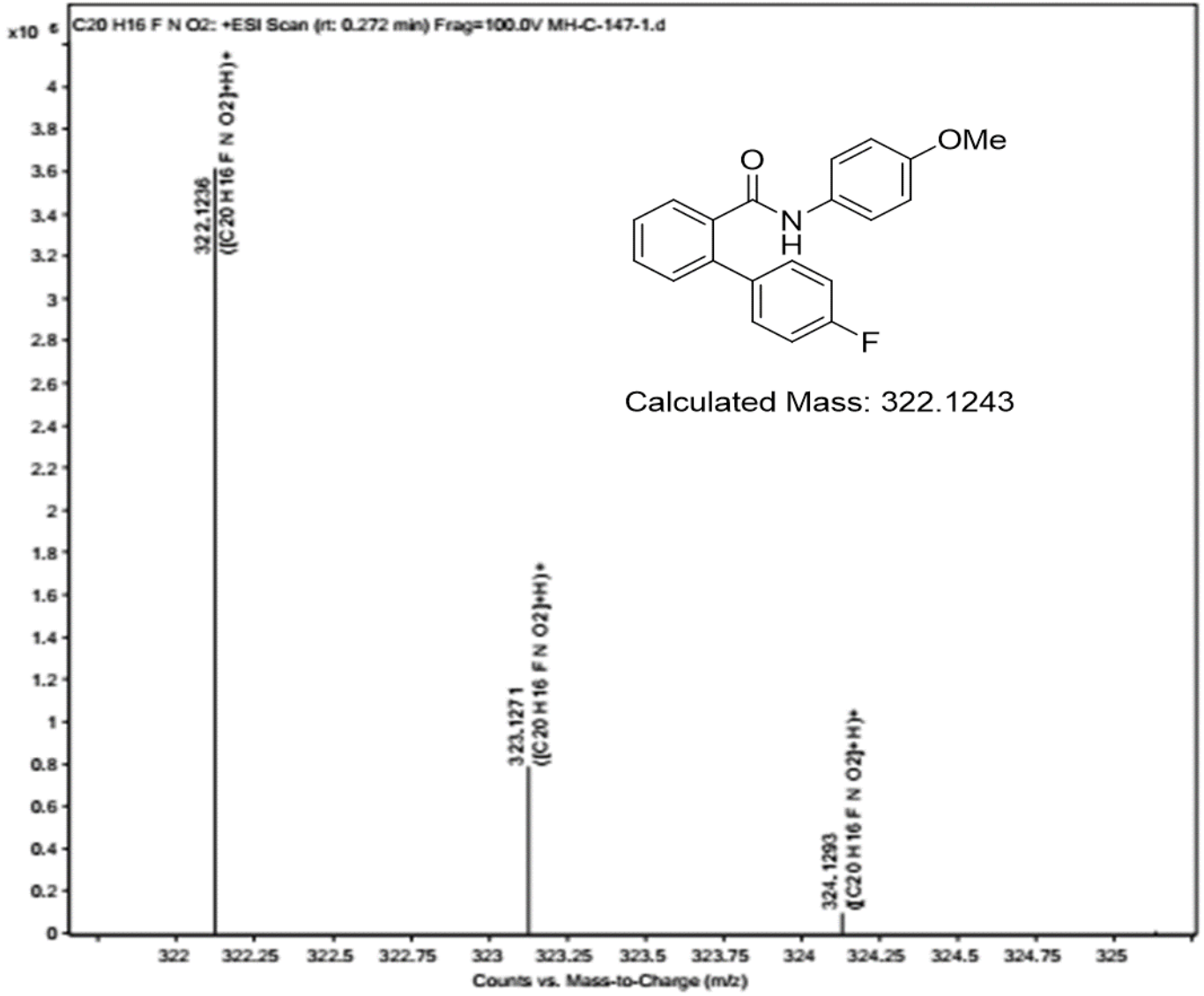
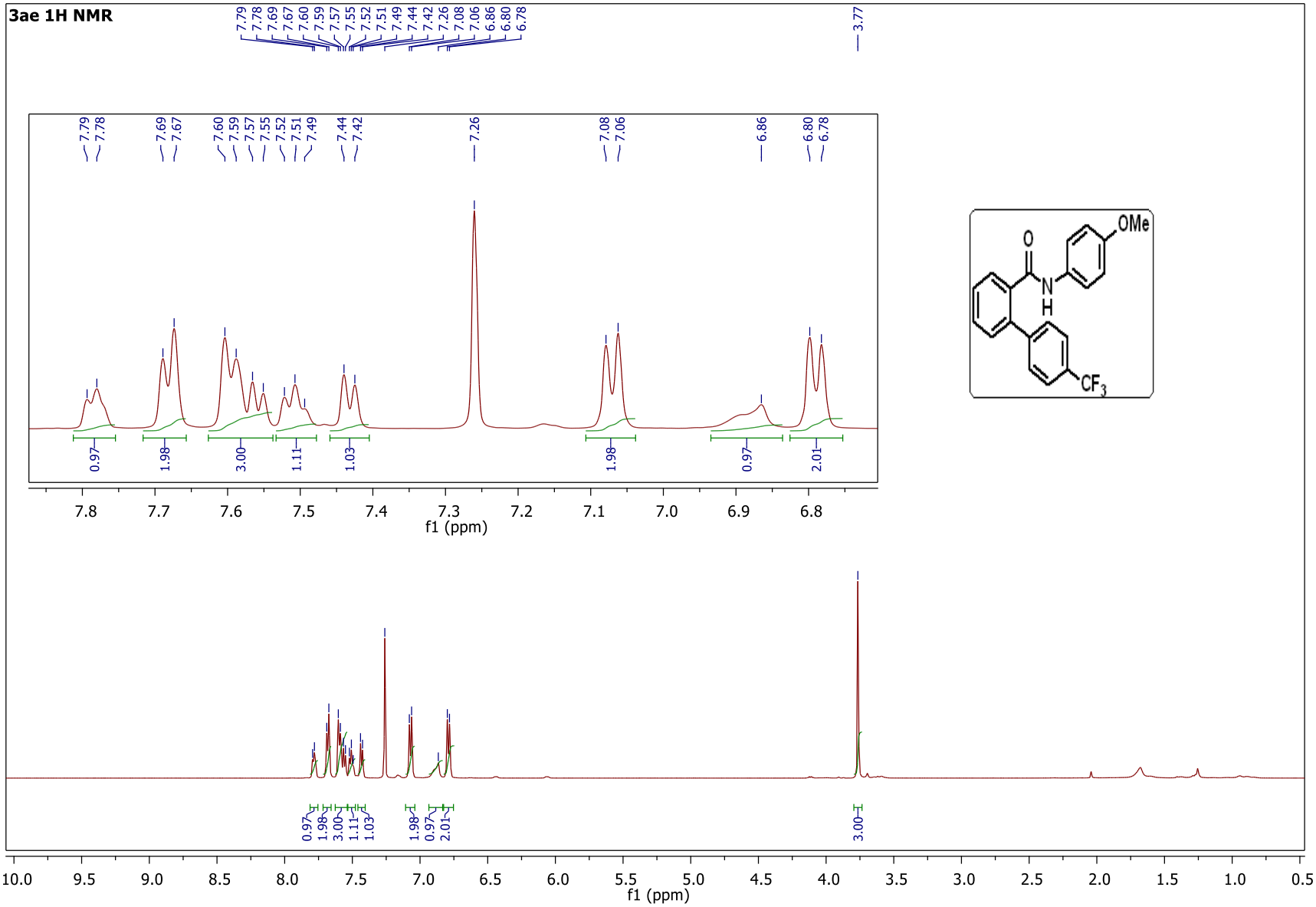
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

Sample Name	MH/C-182-bse	**Position**		**Instrument Name**	Instrument 1
User Name		**Inj Vol**	Unknown / Injection Program	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	MH-C-147-1.d
ACQ Method	Demo.m	**Comment**		**Acquired Time**	10-04-2018 10:07:49 (UTC+05:30)

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7.78
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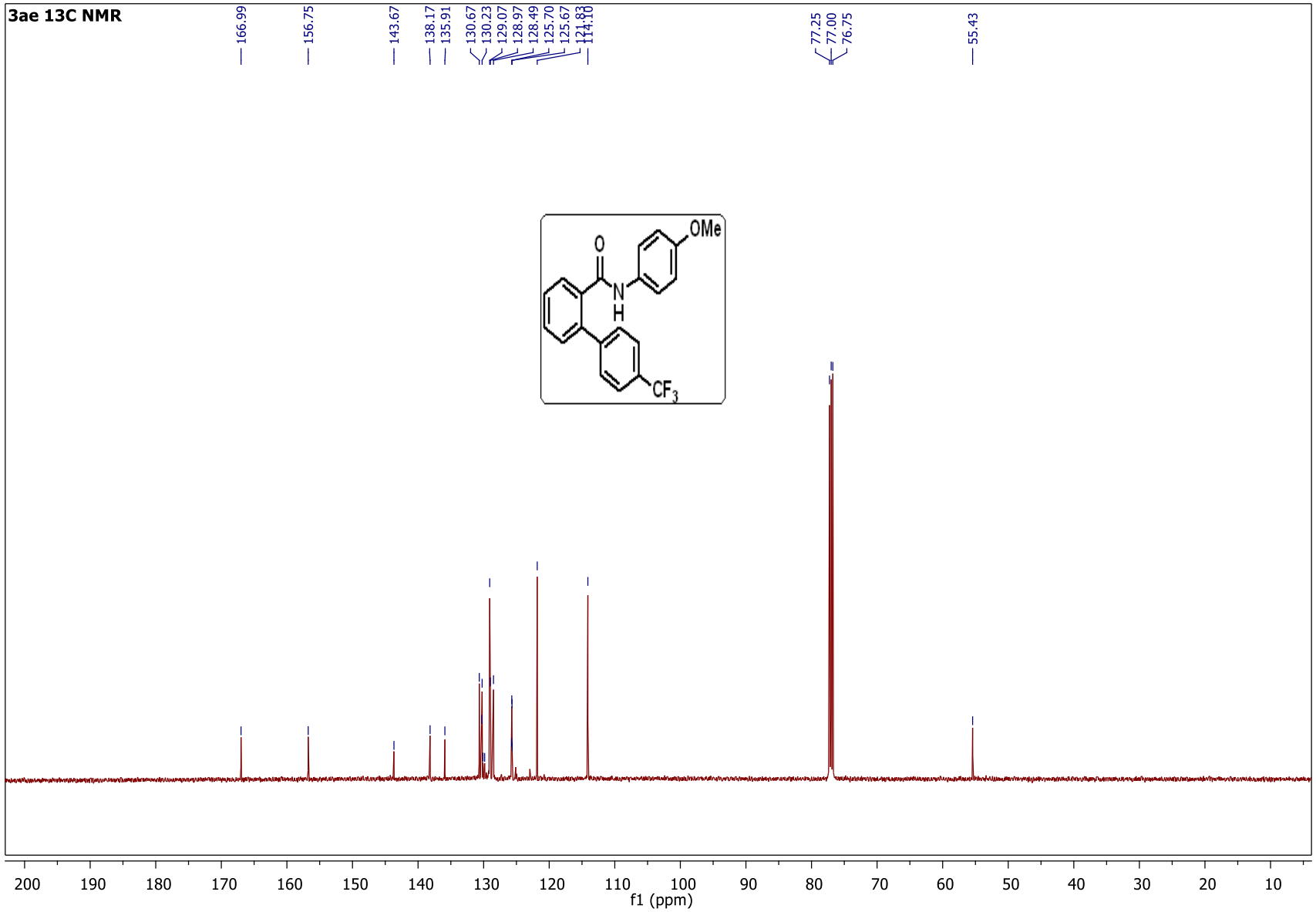
7.67
7.60
7.59
7.57
7.55
7.52
7.51
7.49
7.44
7.42
7.26
7.08
7.06
6.86
6.80
6.78
3.77
0.97
1.98
3.00
1.11
1.03
1.98
0.97
2.01
f1 (ppm)
OMe
O
N
H
CF3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128.49
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125.67
121.83
114.10
77.25
77.00
76.75
55.43
OMe
O
N
H
CF3
200
190
180
170
160
150
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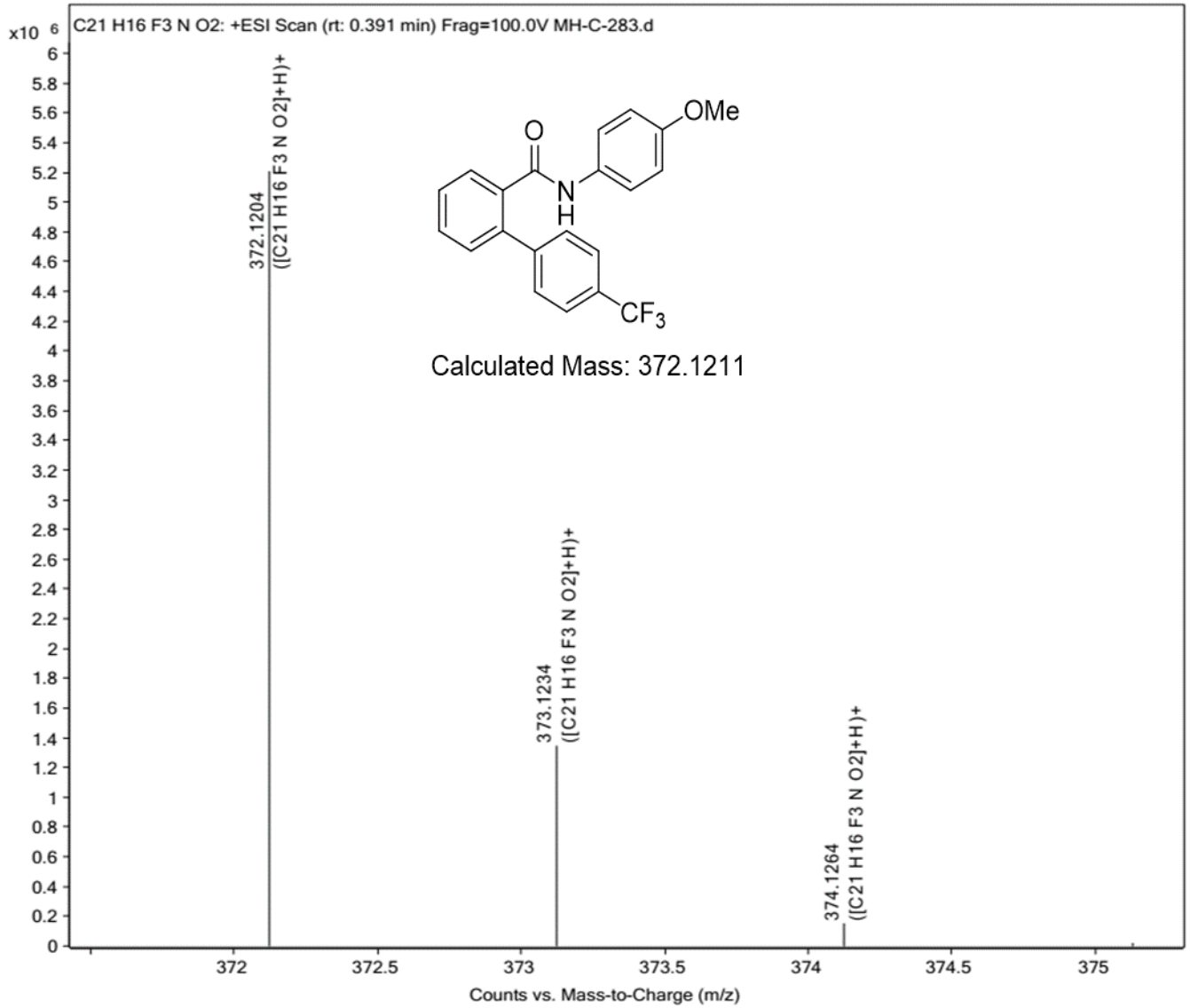
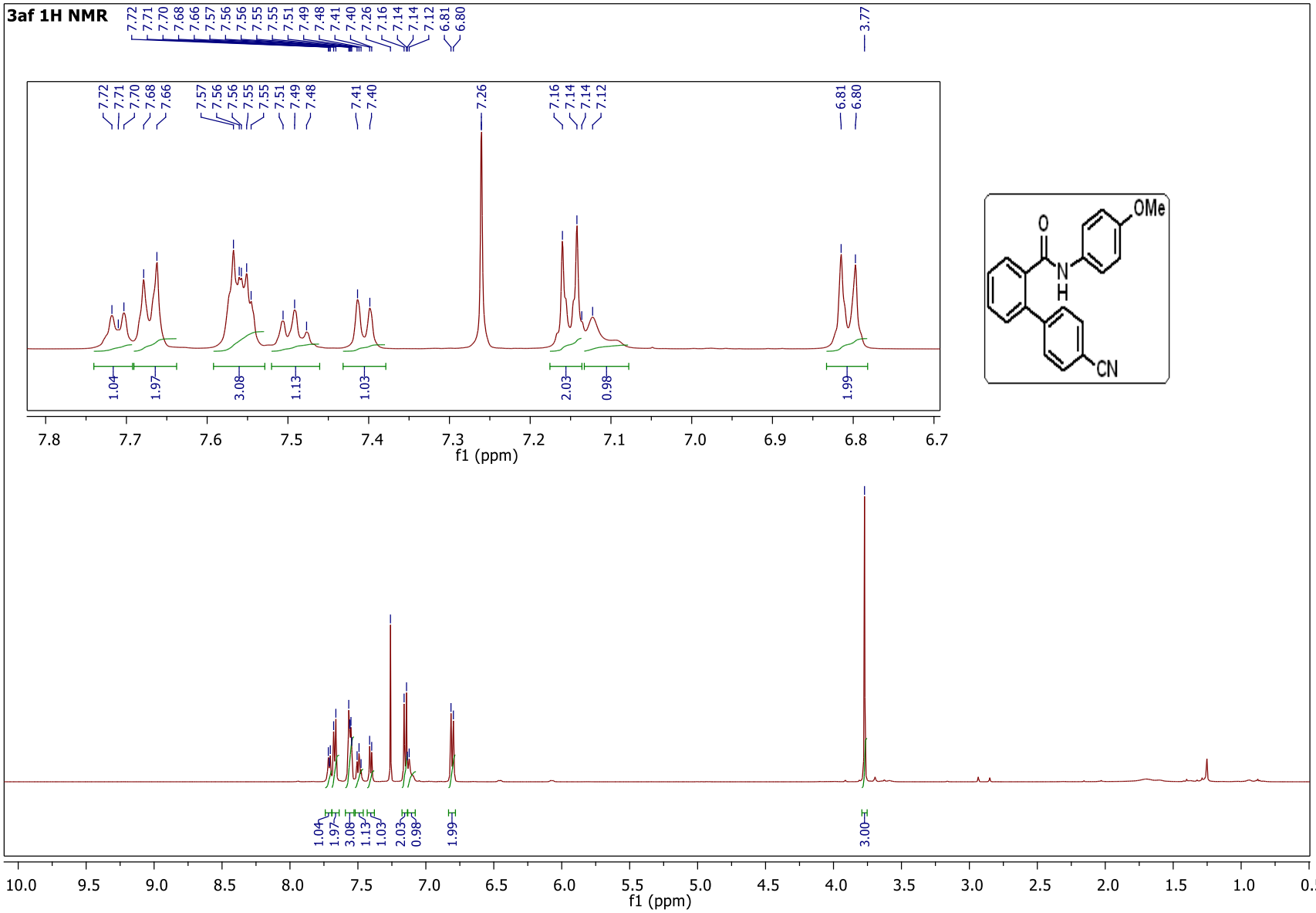
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

Sample Name	MH/C-182-bse	**Position**		**Instrument Name**	Instrument 1
User Name		**Inj Vol**	Unknown / Injection Program	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	MH-C-283.d
ACQ Method	Demo.m	**Comment**		**Acquired Time**	10-04-2018 10:00:44 (UTC+05:30)

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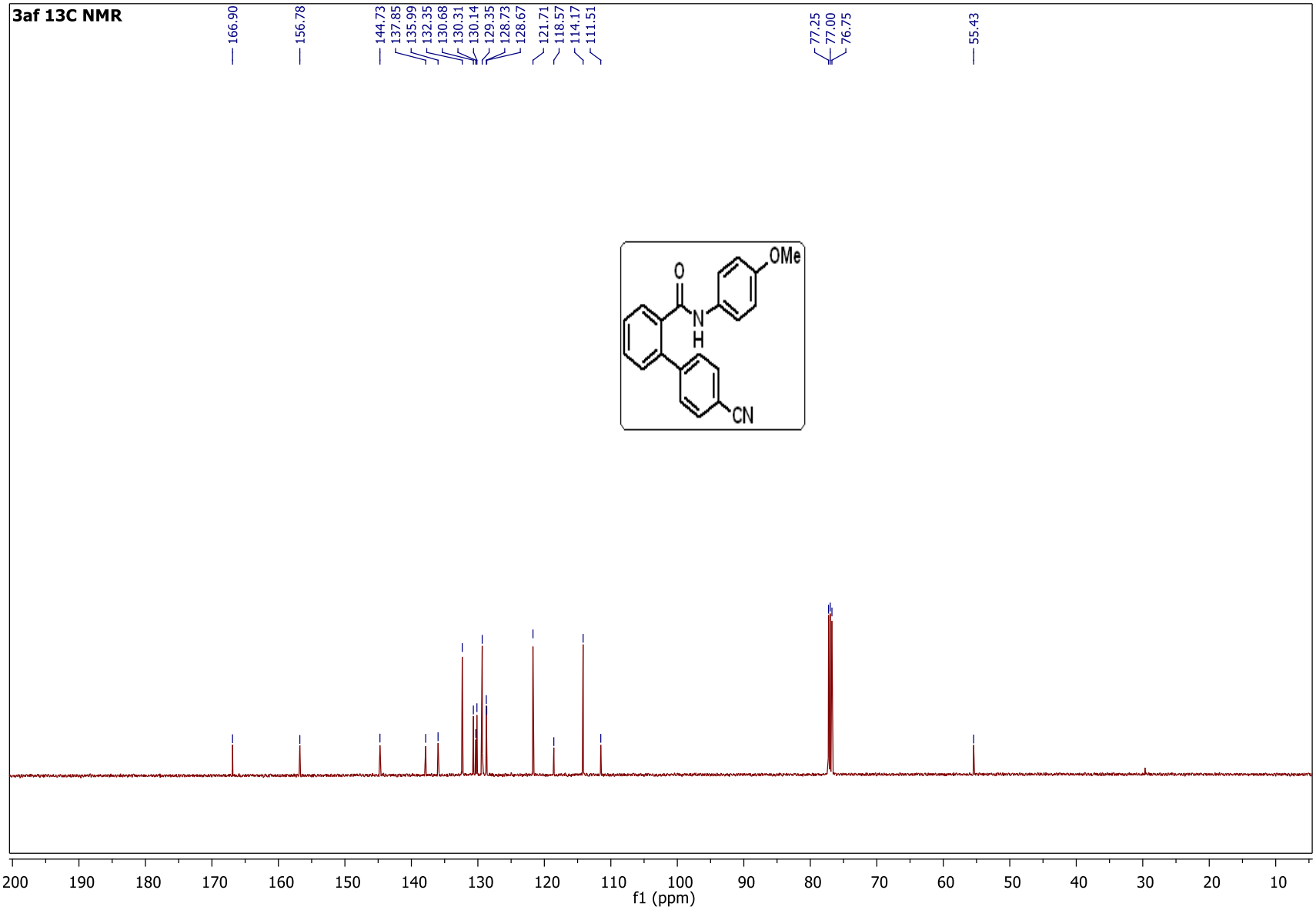
f1 (ppm)
OMe
O
N
H
CN


3af 13C NMR
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130.31
130.14
129.35
128.73
128.67
121.71
118.57
114.17
111.51
77.25
77.00
76.75
55.43
O
N
H
OMe
CN
200
190
180
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70
60
50
40
30
20
10
f1 (ppm)

Data File	MH-388-OME-CYANO.d	**Sample Name**	HARI
Sample Type	Sample	**Position**	
Instrument Name	Instrument 1	**User Name**	
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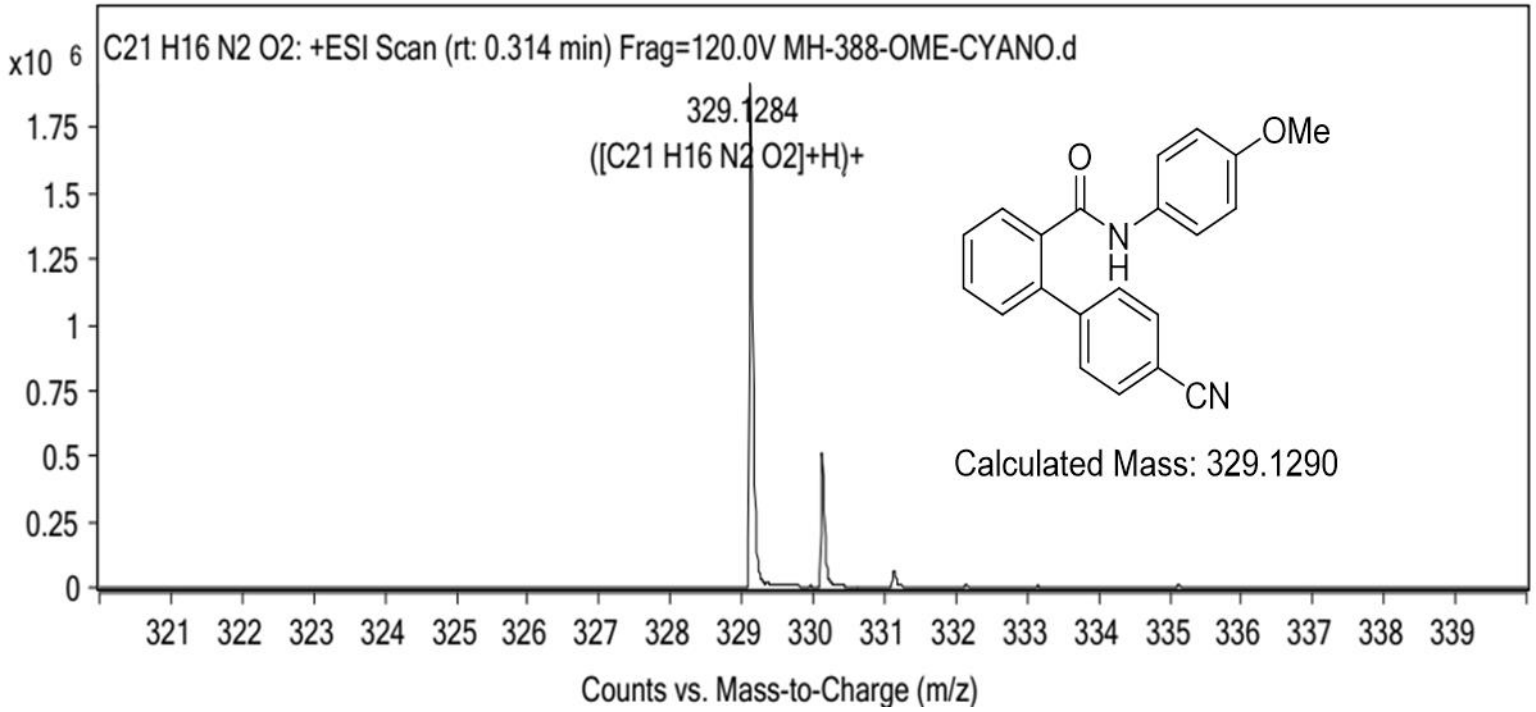
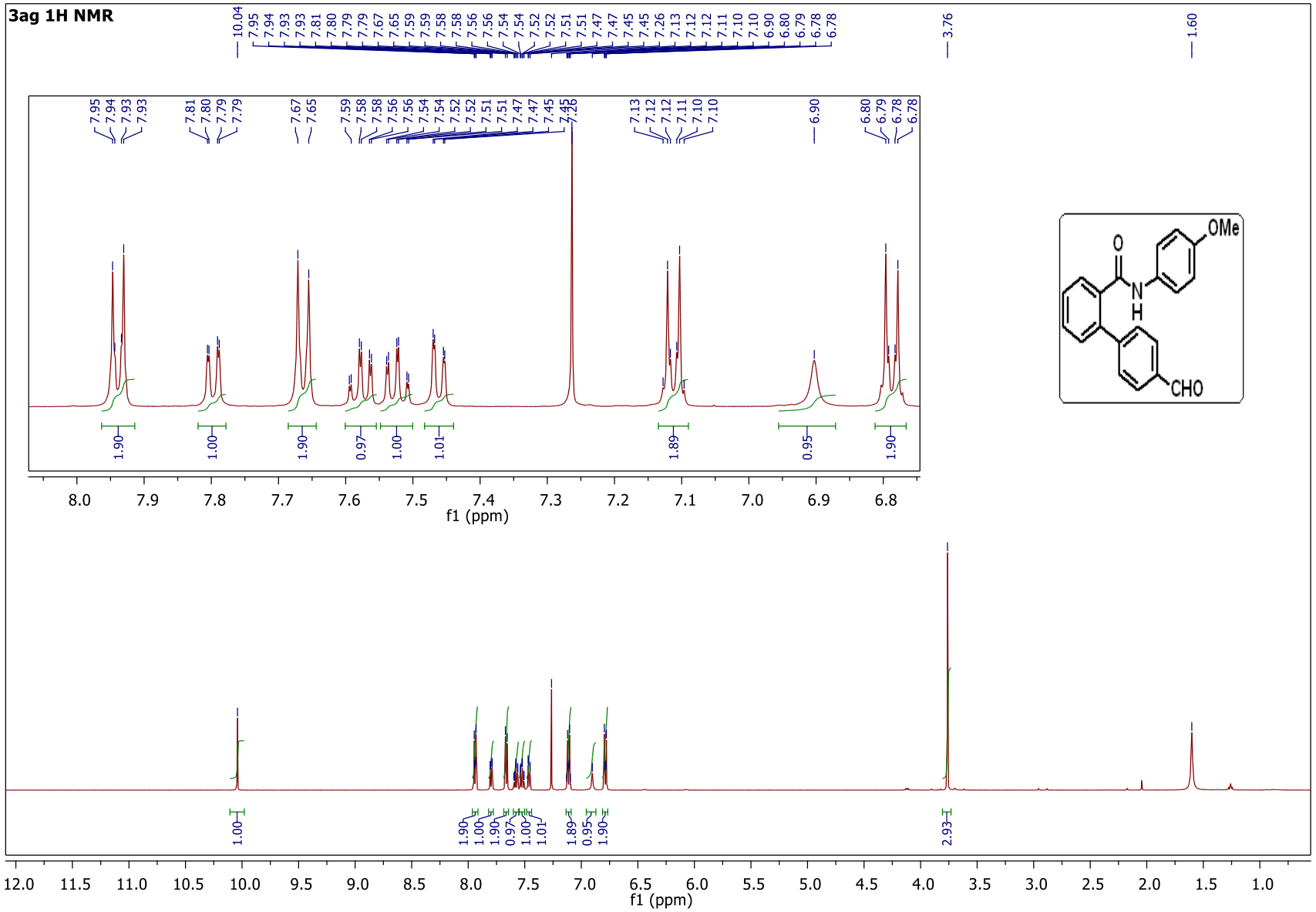
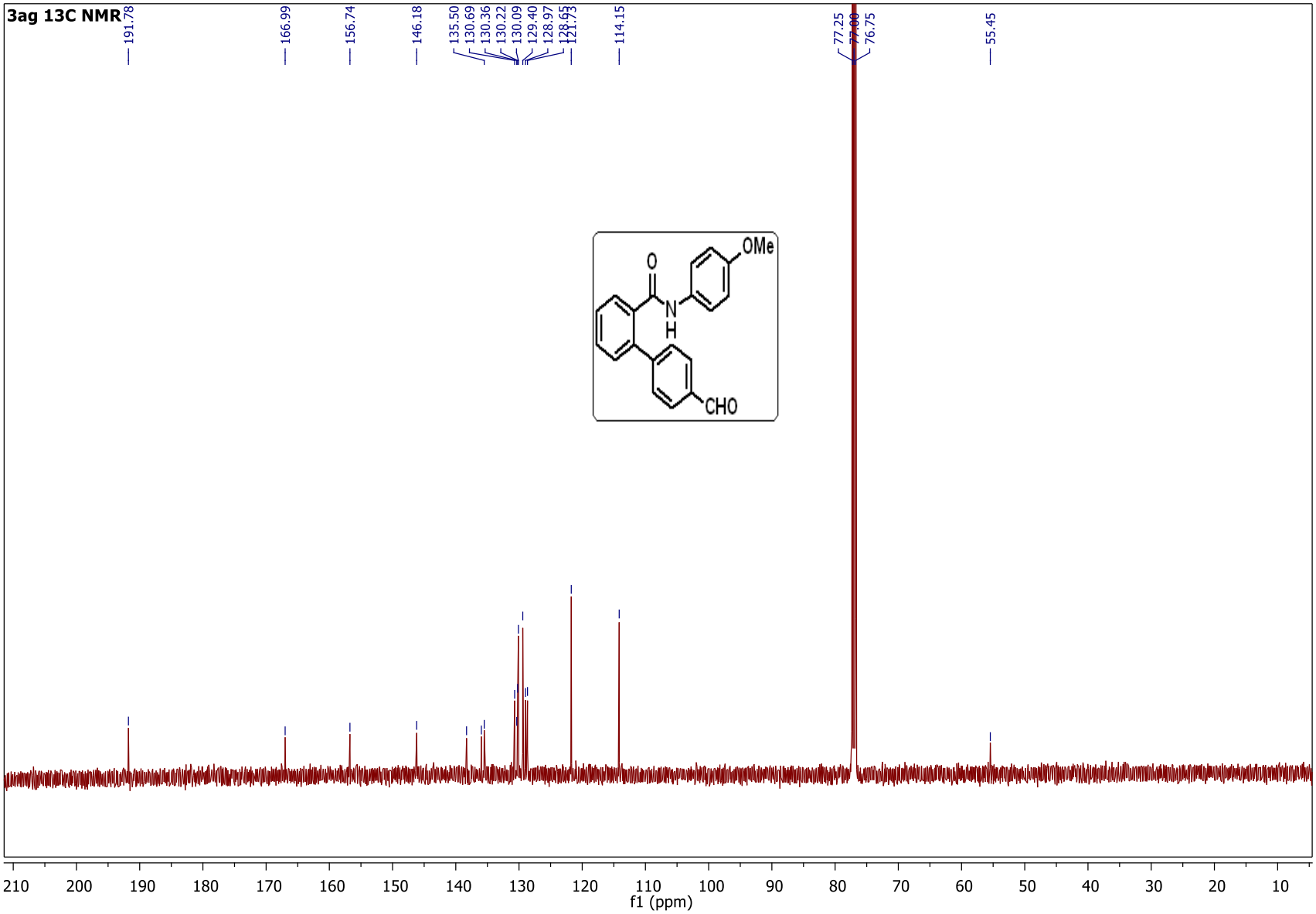
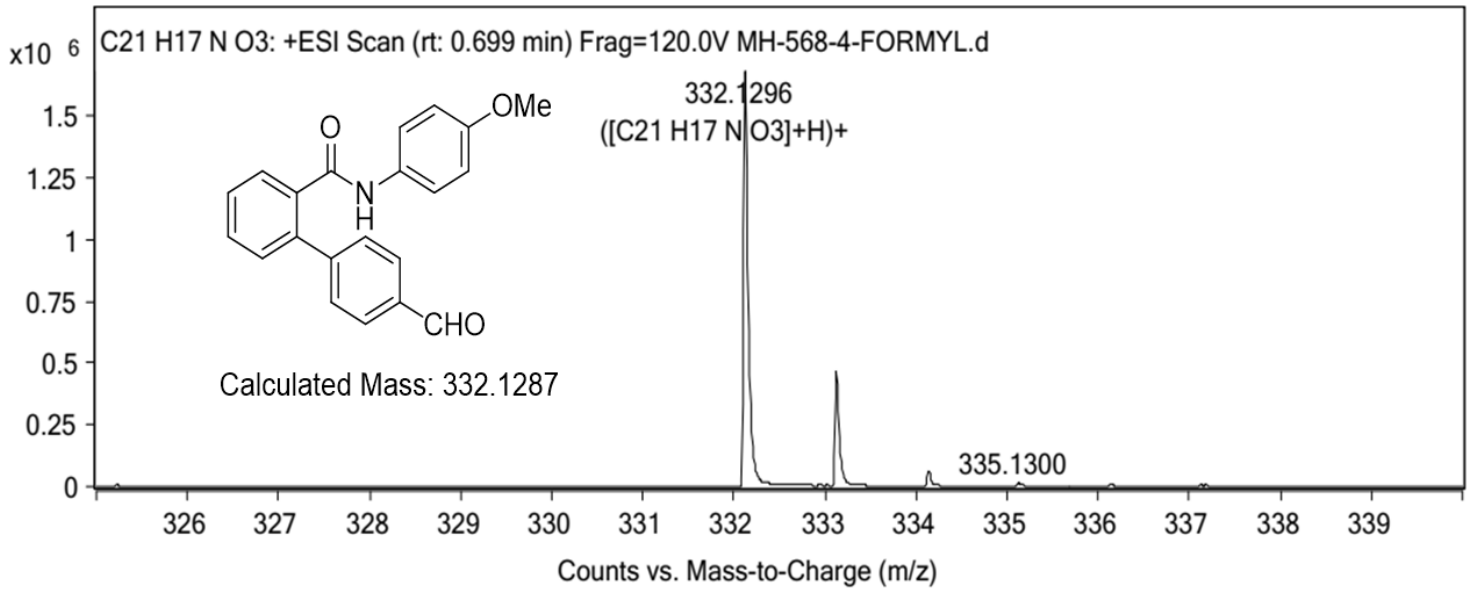
IRM Calibration Status	Success	**DA Method**	HARI.m
Comment			
Sample Group		**Info.**	
Stream Name		**Acquisition Time (Local)**	06-12-2019 16:27:01 (UTC+05:30)
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.08.00 (B8058.0)	**TOF Driver Version**	8.00.00
TOF Firmware Version	17.698	**Tune Mass Range Max.**	3200

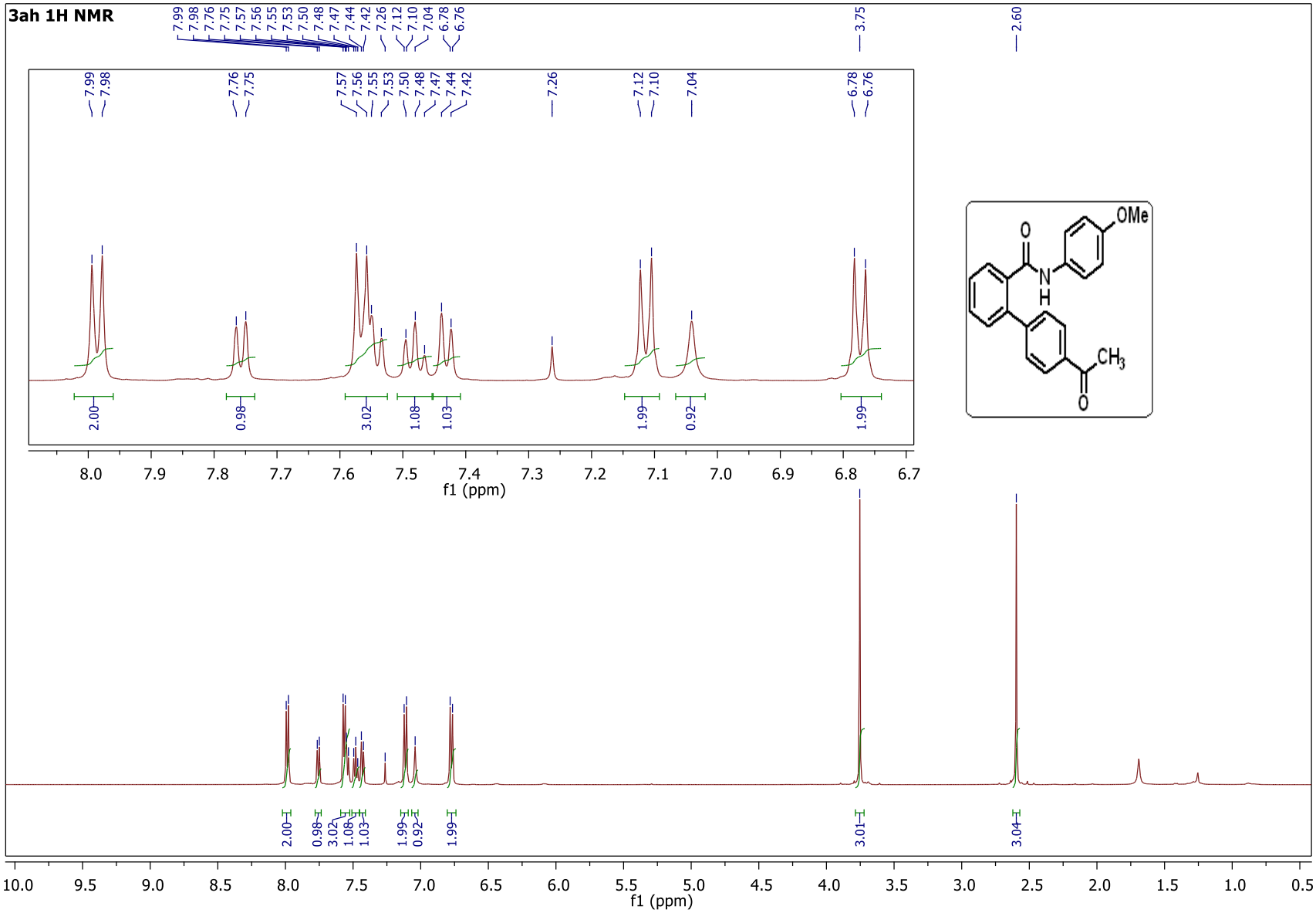


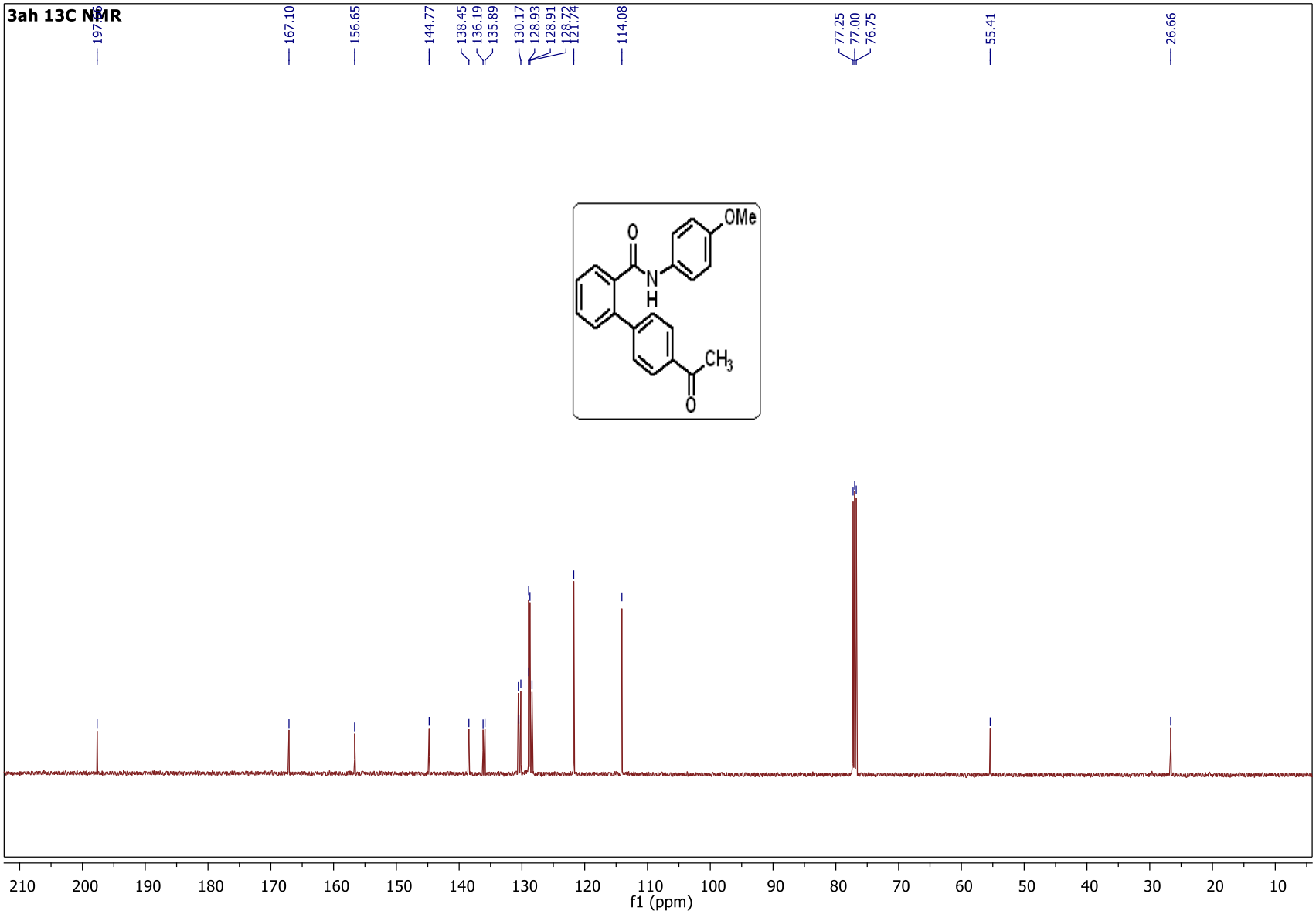

3ag 1H NMR
10.04
7.95
7.94
7.93
7.93
7.81
7.80
7.79
7.79
7.67
7.65
7.59
7.58
7.58
7.56
7.56
7.54
7.54
7.52
7.52
7.51
7.51
7.47
7.45
7.45
7.26
7.13
7.12
7.12
7.11
7.10
7.10
6.90
6.80
6.79
6.78
6.78
3.76
1.60
1.90
1.00
1.90
0.97
1.00
1.01
1.89
0.95
1.90
f1 (ppm)
1.00
1.90
1.00
1.90
0.97
1.00
1.01
1.89
0.95
1.90
2.93
f1 (ppm)
OMe
O
N
H
CHO


3ag 13C NMR
191.78
166.99
156.74
146.18
135.50
130.69
130.36
130.22
130.09
129.40
128.97
128.65
121.73
114.15
77.25
77.00
76.75
55.45
OMe
O
N
H
CHO
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

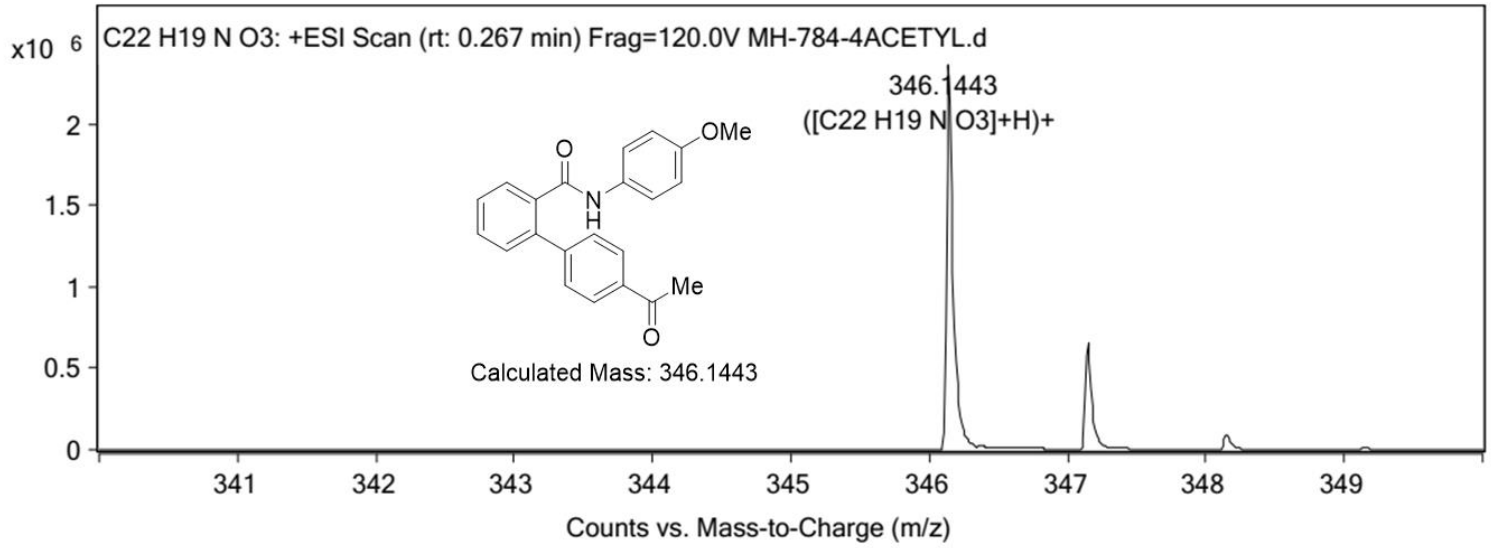
Data File	MH-568-4-FORMYL.d	**Sample Name**	HARI
Sample Type	Sample	**Position**	
Instrument Name	Instrument 1	**User Name**	
Acq Method	Sample_pos_01.m	**Acquired Time**	06-12-2019 15:25:19 (UTC+05:30)
IRM Calibration Status	Success	**DA Method**	HARI.m
Comment			
Sample Group		**Info.**	
Stream Name		**Acquisition Time (Local)**	06-12-2019 15:25:19 (UTC+05:30)
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.08.00 (B8058.0)	**TOF Driver Version**	8.00.00
TOF Firmware Version	17.698	**Tune Mass Range Max.**	3200

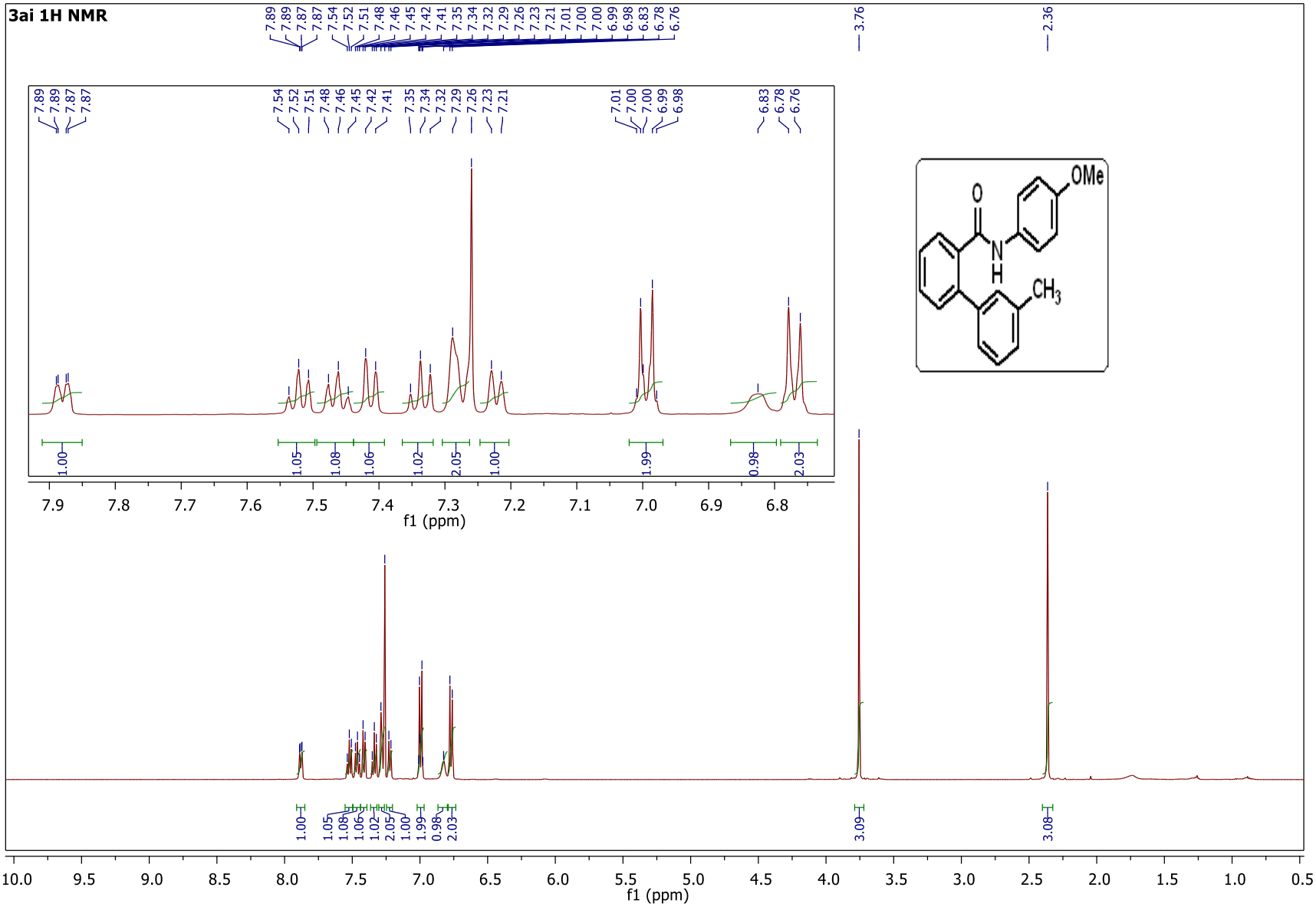



3ah 1H NMR
7.99
7.98
7.76
7.75
7.57
7.56
7.55
7.53
7.50
7.48
7.47
7.44
7.42
7.26
7.12
7.10
7.04
6.78
6.76
3.75
2.60
2.00
0.98
3.02
1.08
1.03
1.99
0.92
1.99
3.01
3.04
f1 (ppm)
OMe
O
N
H
CH3
O

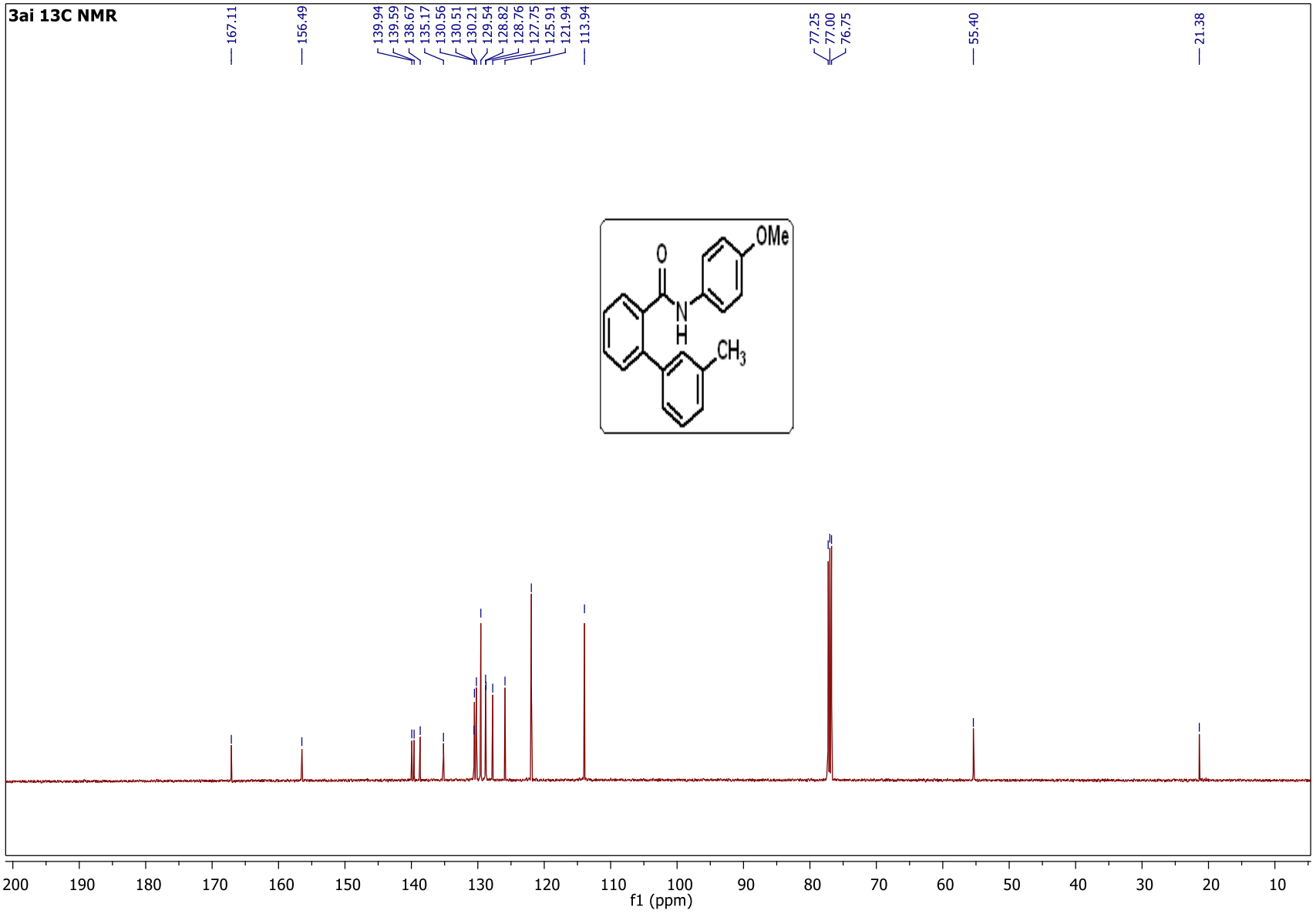

3ah 13C NMR
197.76
167.10
156.65
144.77
138.45
136.19
135.89
130.17
128.93
128.91
128.72
121.74
114.08
77.25
77.00
76.75
55.41
26.66
OMe
O
N
H
CH3
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

Data File	MH-784-4ACETYL.d	**Sample Name**	HARI
Sample Type	Sample	**Position**	
Instrument Name	Instrument 1	**User Name**	
Acq Method	Sample_pos_01.m	**Acquired Time**	06-12-2019 15:11:55 (UTC+05:30)
IRM Calibration Status	Success	**DA Method**	HARI.m
Comment			
Sample Group		**Info.**	
Stream Name		**Acquisition Time (Local)**	06-12-2019 15:11:55 (UTC+05:30)
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.08.00 (B8058.0)	**TOF Driver Version**	8.00.00
TOF Firmware Version	17.698	**Tune Mass Range Max.**	3200

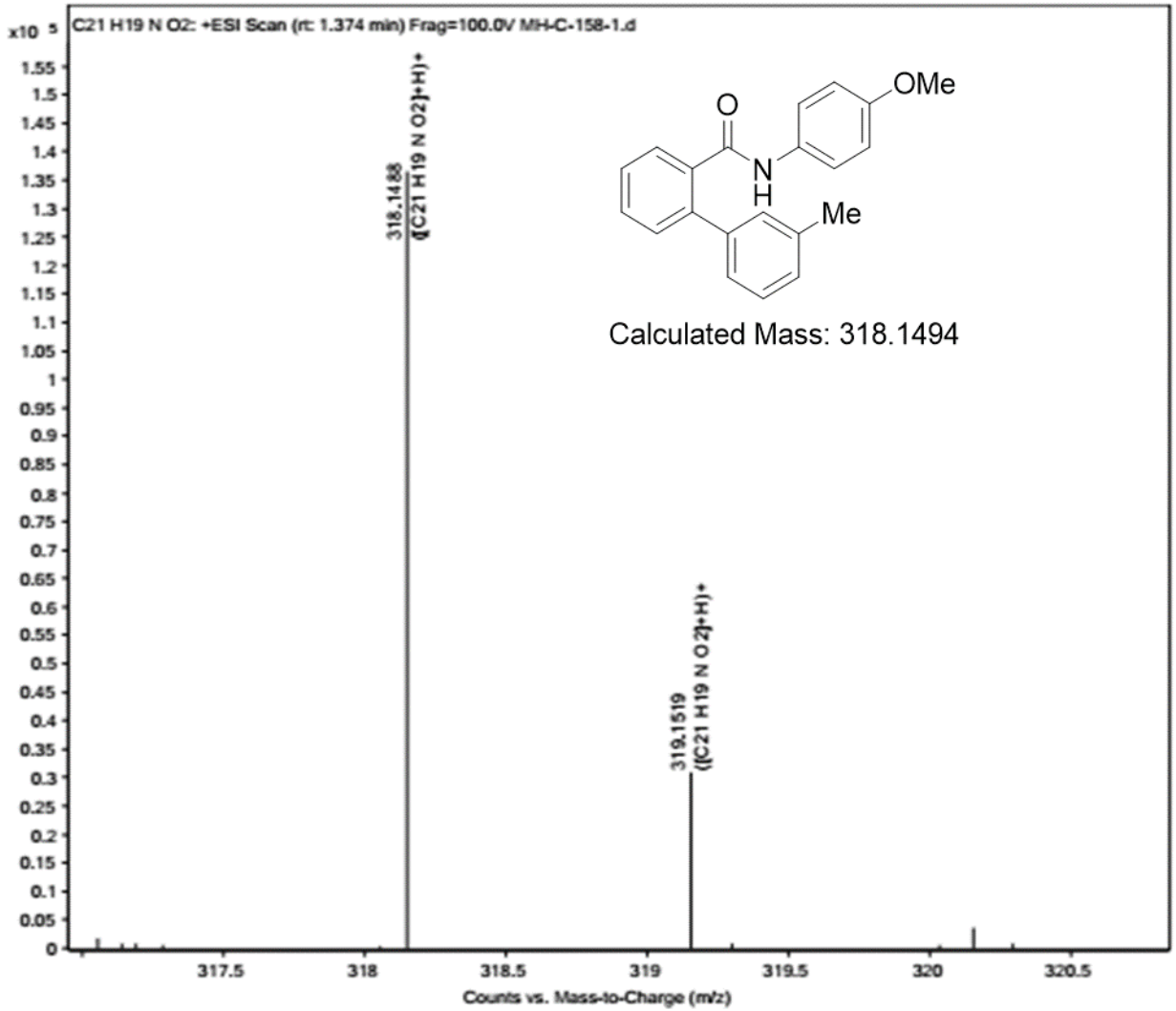


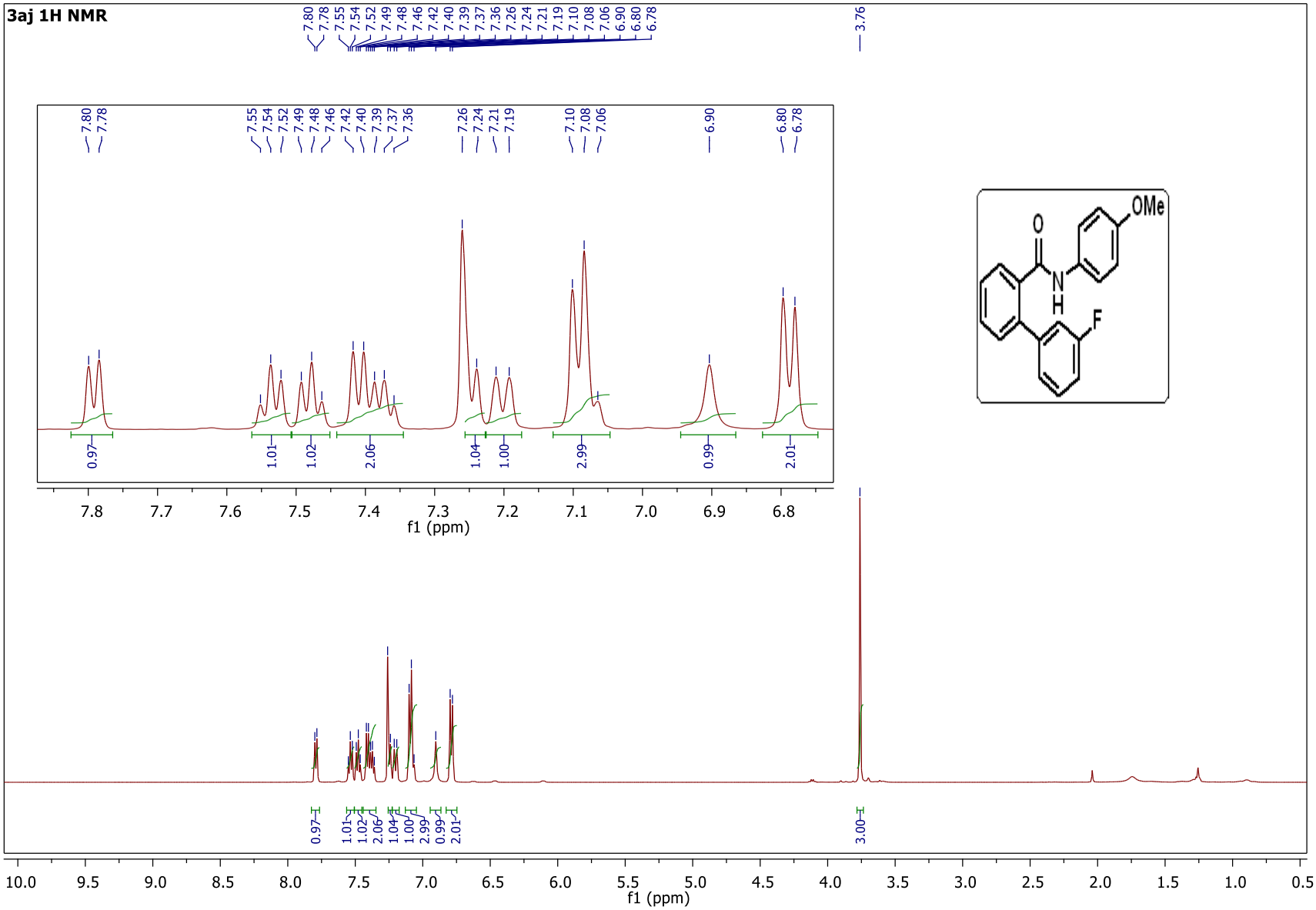

3ai 1H NMR
7.89 7.89 7.87 7.87 7.54 7.52 7.51 7.48 7.46 7.45 7.42 7.41 7.35 7.34 7.32 7.29 7.26 7.23 7.21 7.01 7.00 7.00 6.99 6.98 6.83 6.78 6.76 3.76 2.36
1.00 1.05 1.08 1.06 1.02 2.05 1.00 1.99 0.98 2.03
f1 (ppm)
3.09 3.08
f1 (ppm)
O
N
H
OMe
CH3

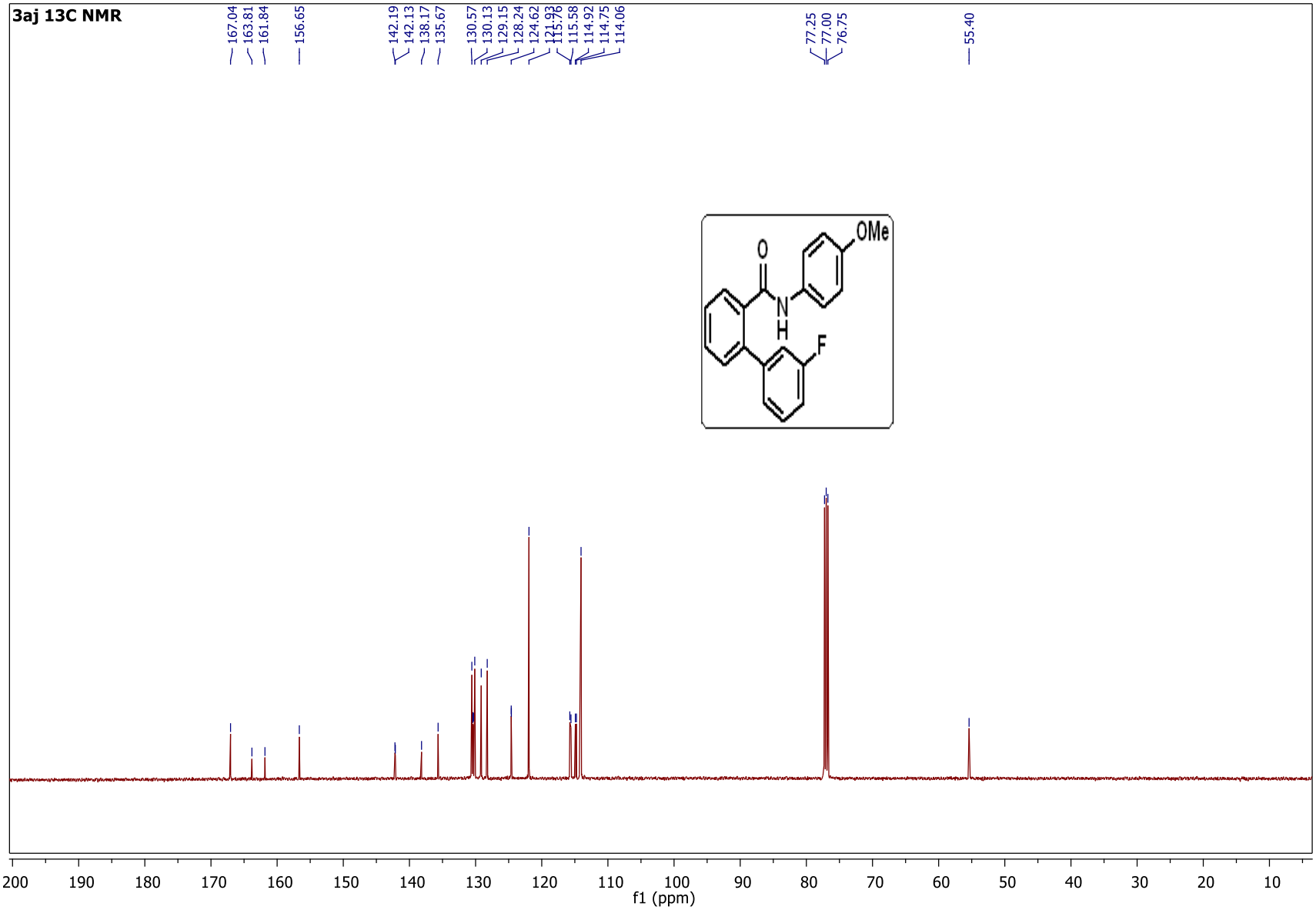
3ai 13C NMR


167.11
156.49
139.94
139.59
138.67
135.17
130.56
130.51
130.21
129.54
128.82
128.76
127.75
125.91
121.94
113.94
77.25
77.00
76.75
55.40
21.38
O
N
H
OMe
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

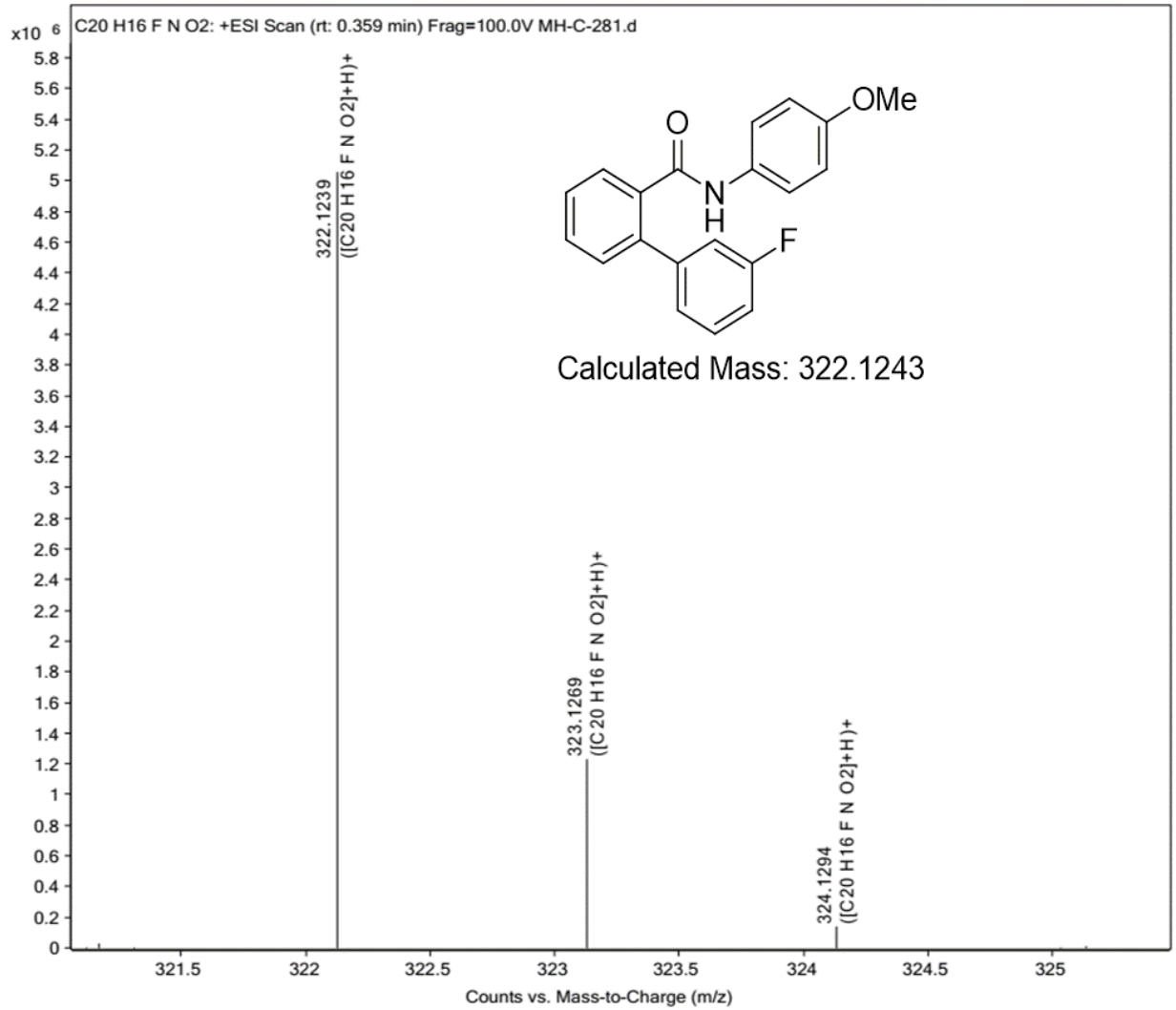
Sample Name	MH/C-158-1	**Position**		**Instrument Name**	Instrument 1
User Name		**Inj Vol**	Unknown / Injection Program	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	MH-C-158-1.d
ACQ Method	Demo.m	**Comment**		**Acquired Time**	10-04-2018 04:07:20 (UTC+05:30)

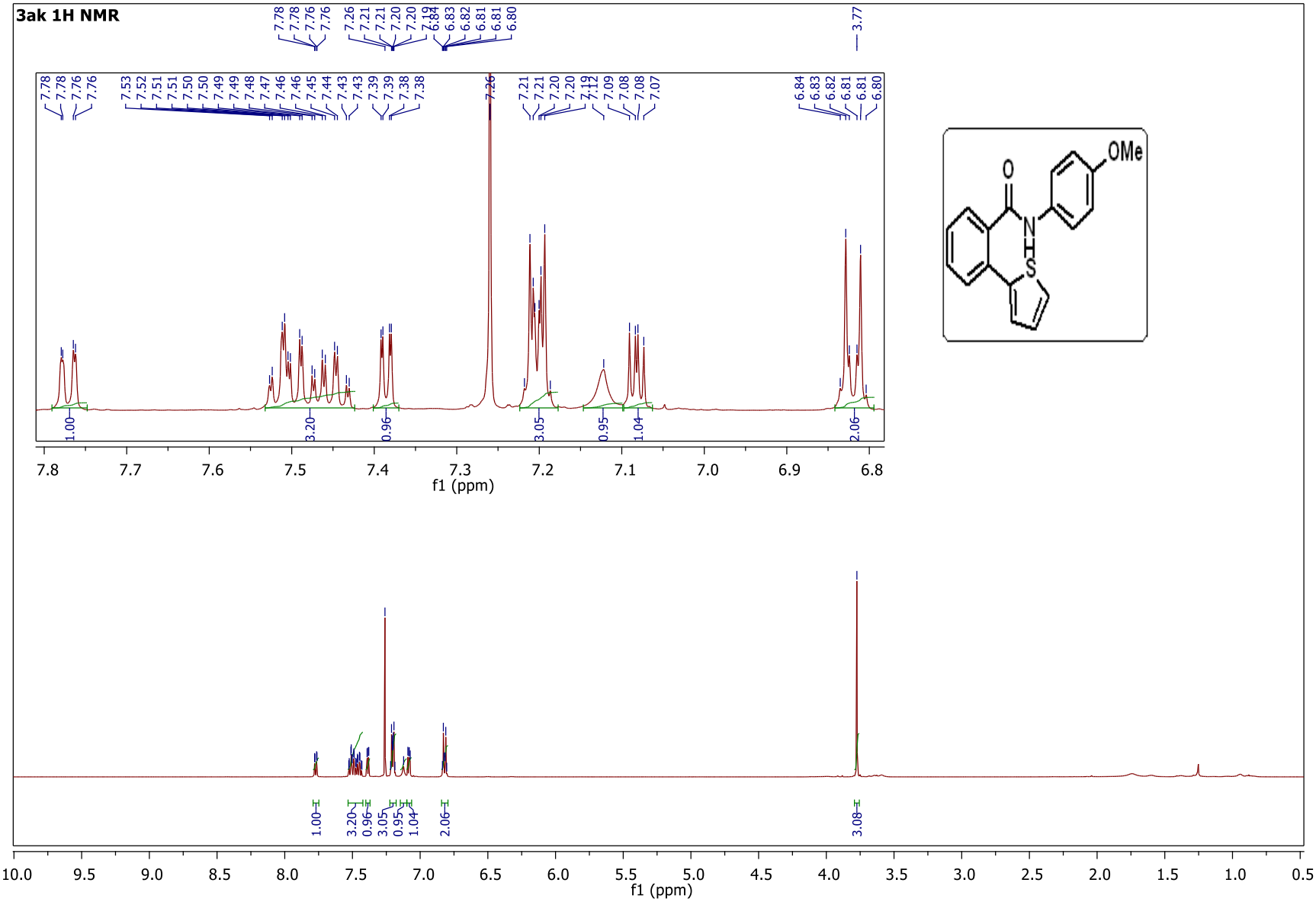


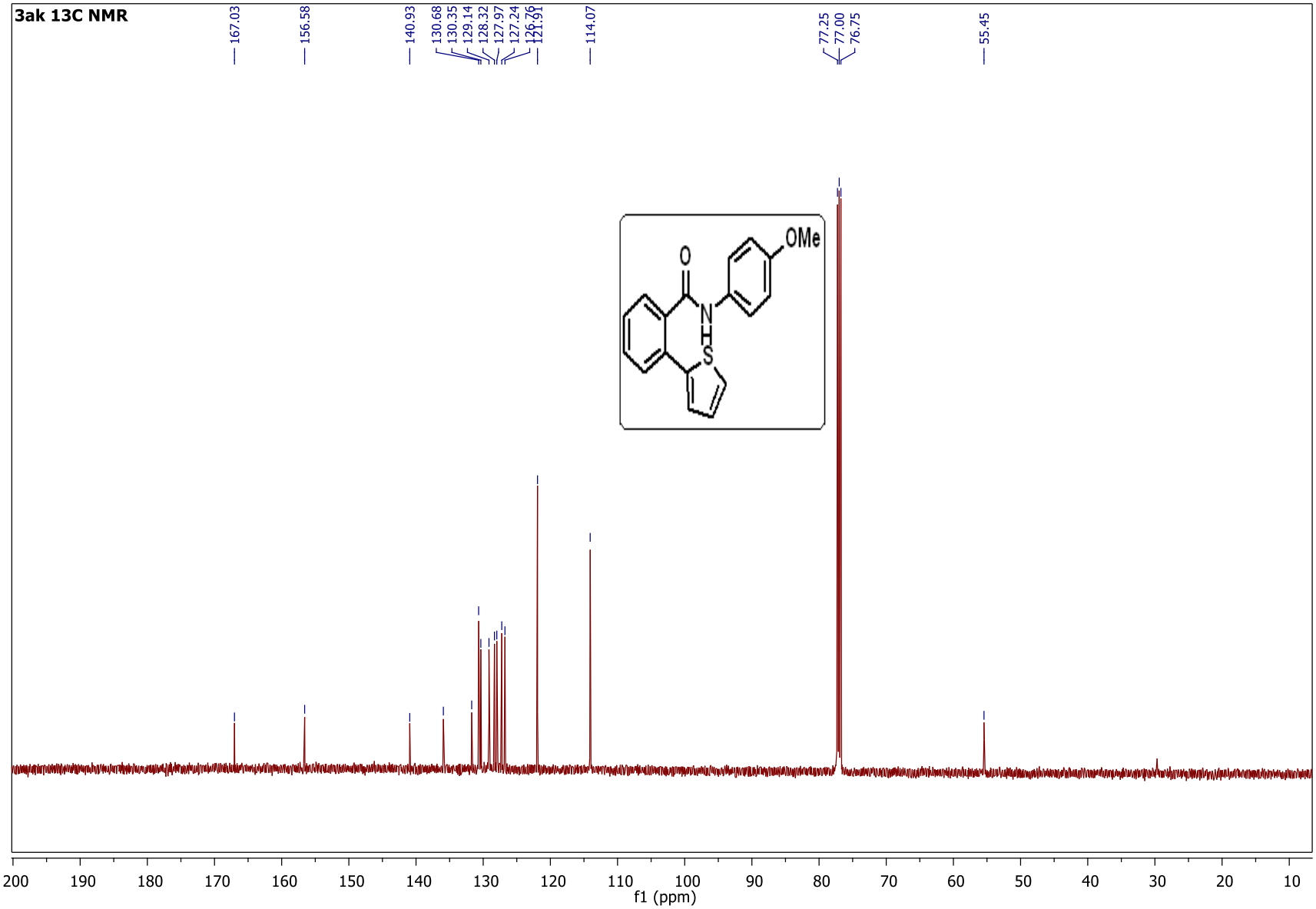

3aj 1H NMR
7.80
7.78
7.55
7.54
7.52
7.49
7.48
7.46
7.42
7.40
7.39
7.37
7.36
7.26
7.24
7.21
7.19
7.10
7.08
7.06
6.90
6.80
6.78
3.76
0.97
1.01
1.02
2.06
1.04
1.00
2.99
0.99
2.01
3.00
OMe
O
N
H
F
f1 (ppm)


3aj 13C NMR
167.04
163.81
161.84
156.65
142.19
142.13
138.17
135.67
130.57
130.13
129.15
128.24
124.62
121.93
115.76
115.58
114.92
114.75
114.06
77.25
77.00
76.75
55.40
O
N
H
F
OMe
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

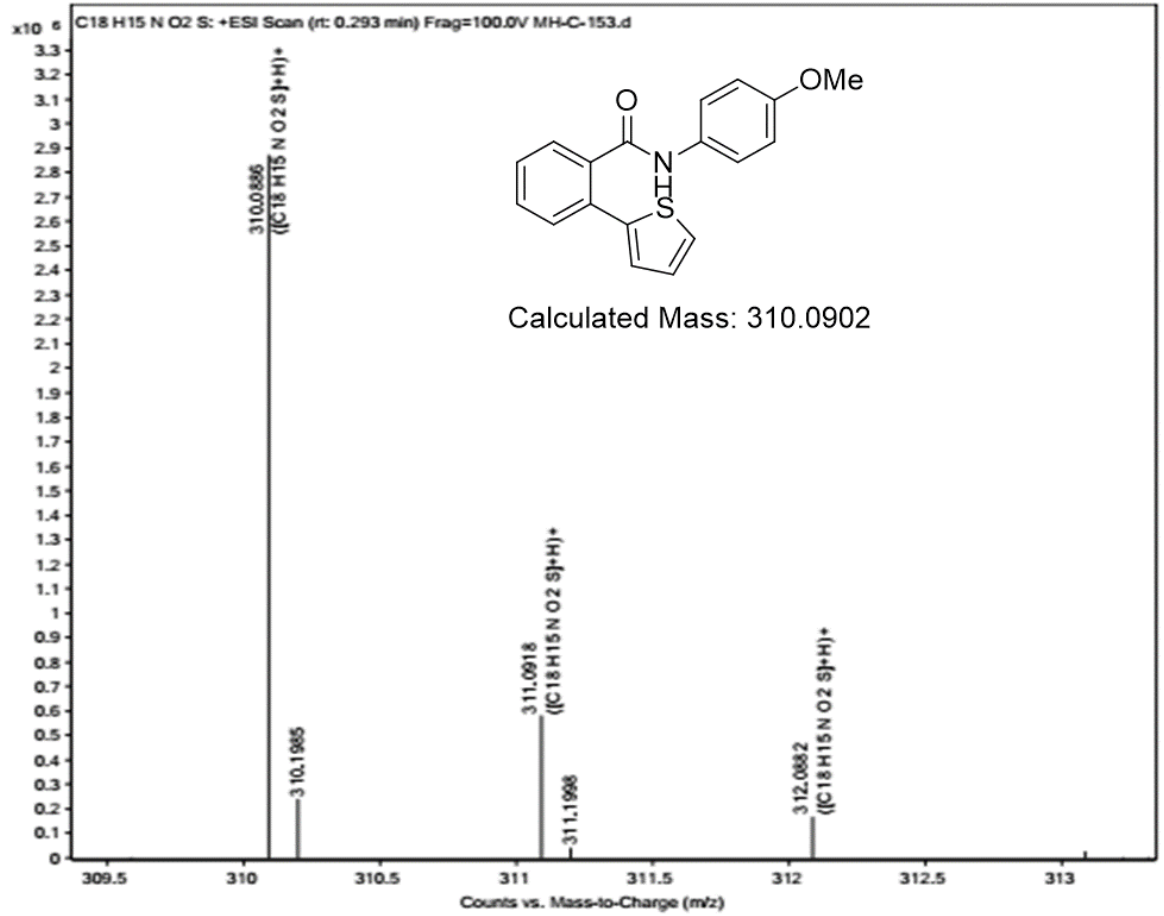
Sample Name	MH/C-182-bse	**Position**		**Instrument Name**	Instrument 1
User Name		**Inj Vol**	Unknown / Injection Program	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	MH-C-281.d
ACQ Method	Demo.m	**Comment**		**Acquired Time**	10-04-2018 10:12:55 (UTC+05:30)



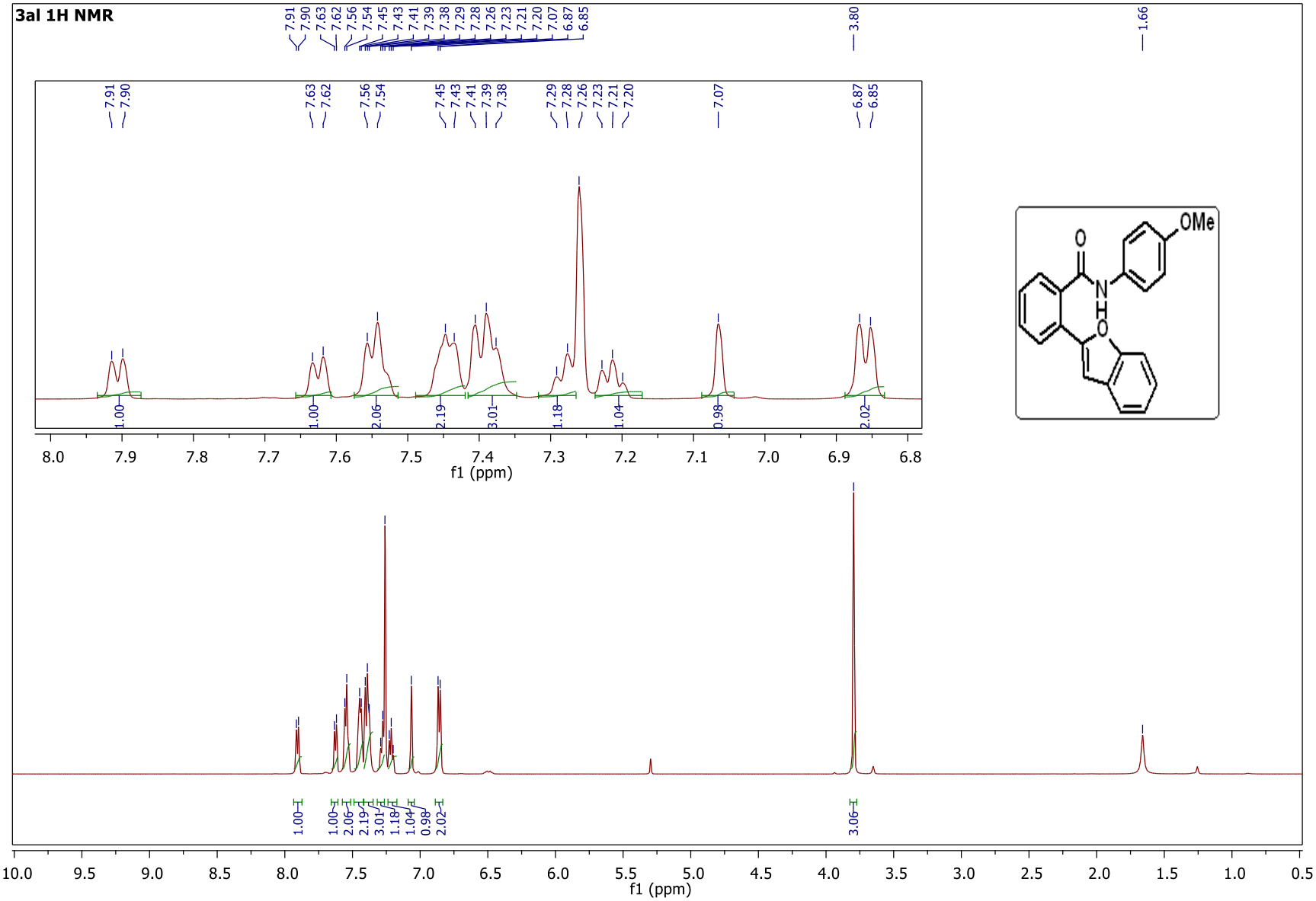

3ak 1H NMR
7.78 7.78 7.76 7.76 7.26 7.21 7.21 7.20 7.20 7.19 6.84 6.83 6.82 6.81 6.81 6.80 3.77
7.53 7.52 7.51 7.51 7.50 7.50 7.49 7.49 7.48 7.47 7.46 7.46 7.45 7.44 7.43 7.43 7.39 7.39 7.38 7.38
7.21 7.21 7.20 7.20 7.12 7.09 7.08 7.08 7.07
6.84 6.83 6.82 6.81 6.81 6.80
1.00 3.20 0.96 3.05 0.95 1.04 2.06
7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8
f1 (ppm)
OMe
O
N
H
S
1.00 3.20 0.96 3.05 0.95 1.04 2.06 3.08
10.0 9.5 9.0 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 0.5
f1 (ppm)

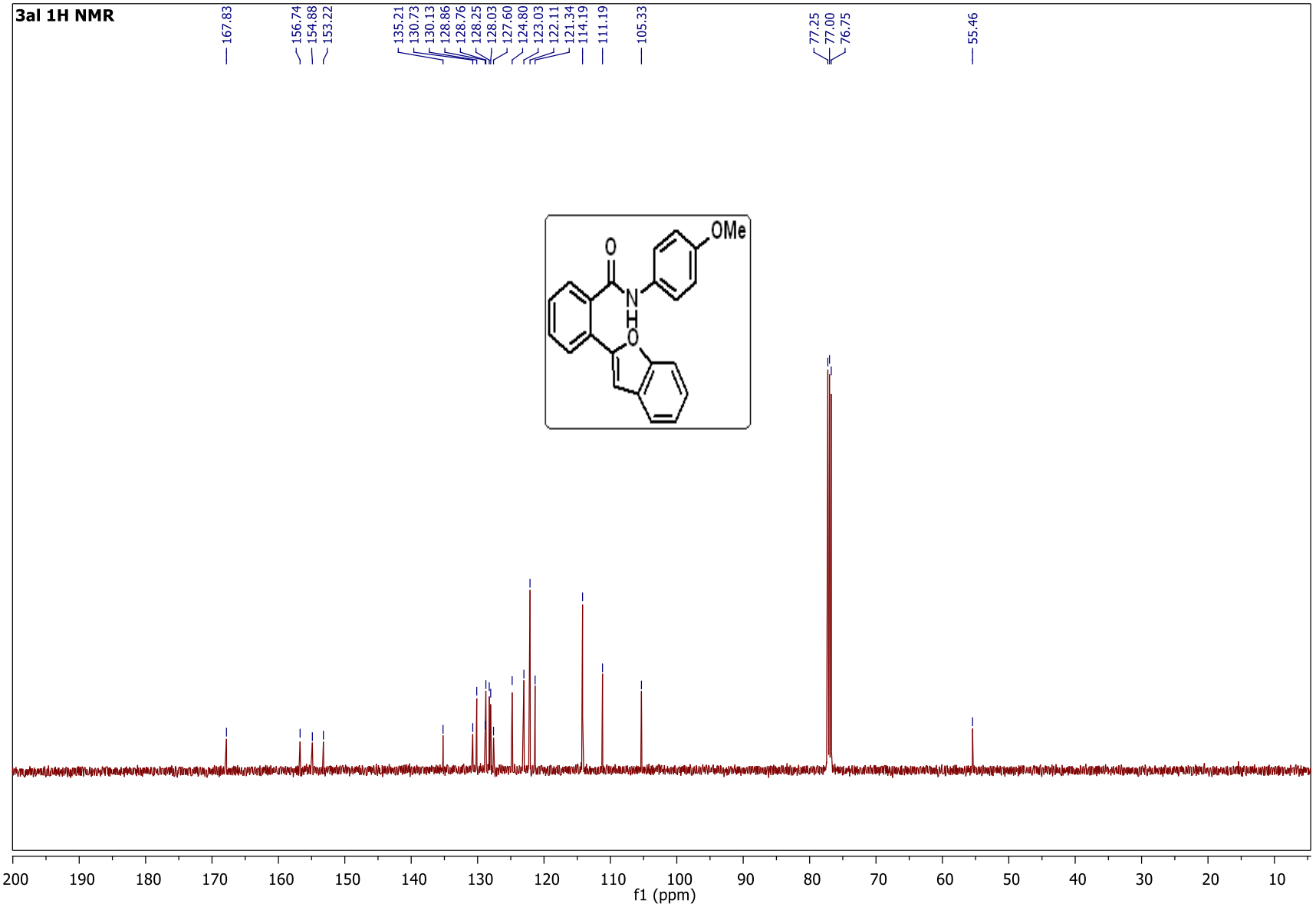

3ak 13C NMR
167.03
156.58
140.93
130.68
130.35
129.14
128.32
127.97
127.24
126.76
121.91
114.07
77.25
77.00
76.75
55.45
OMe
O
N
H
S
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

Sample Name	MH/C-153	**Position**		**Instrument Name**	Instrument 1
User Name		**Inj Vol**	Unknown / Injection Program	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	MH-C-153.d
ACQ Method	Demo.m	**Comment**		**Acquired Time**	10-04-2018 03:20:03 (UTC+05:30)

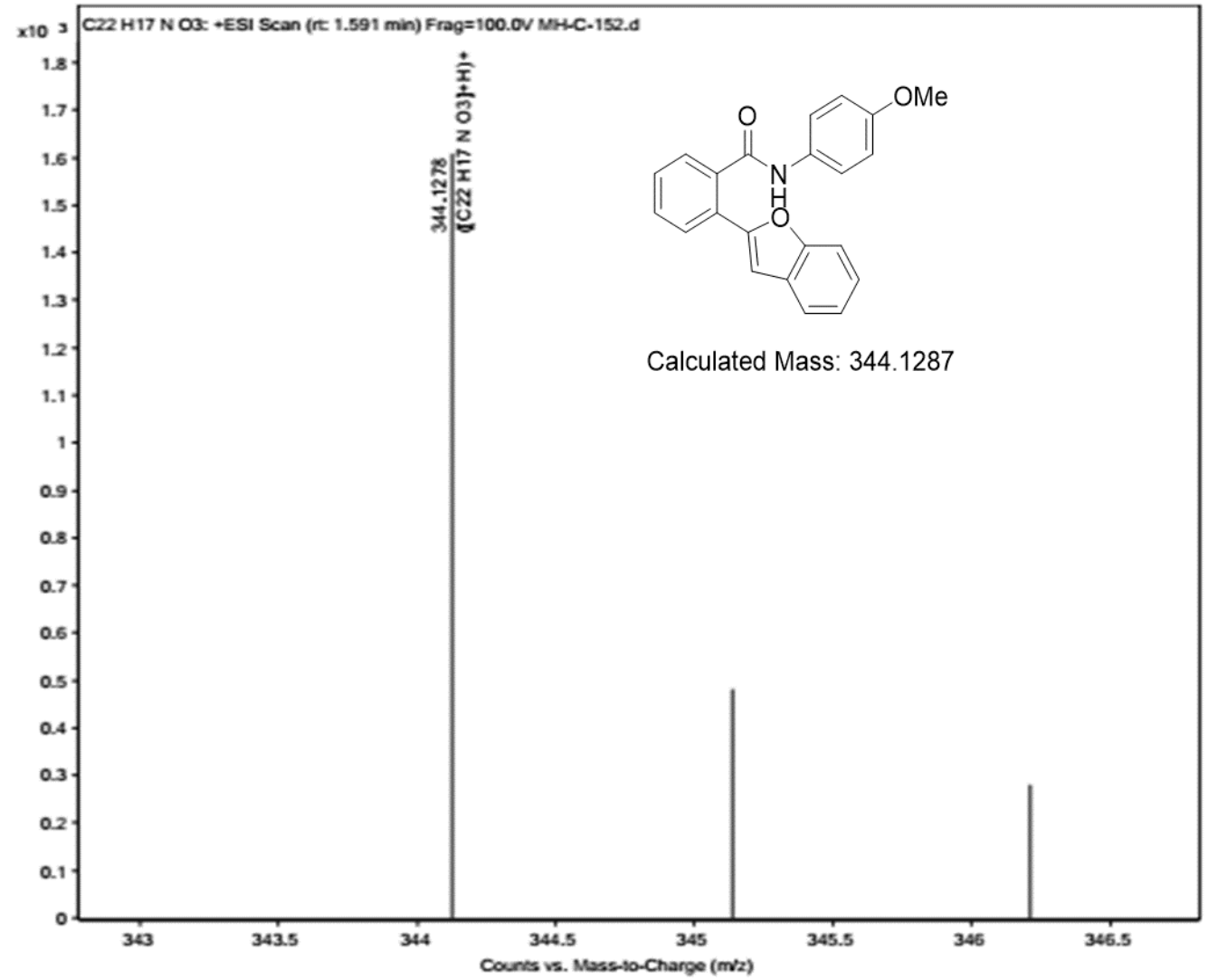


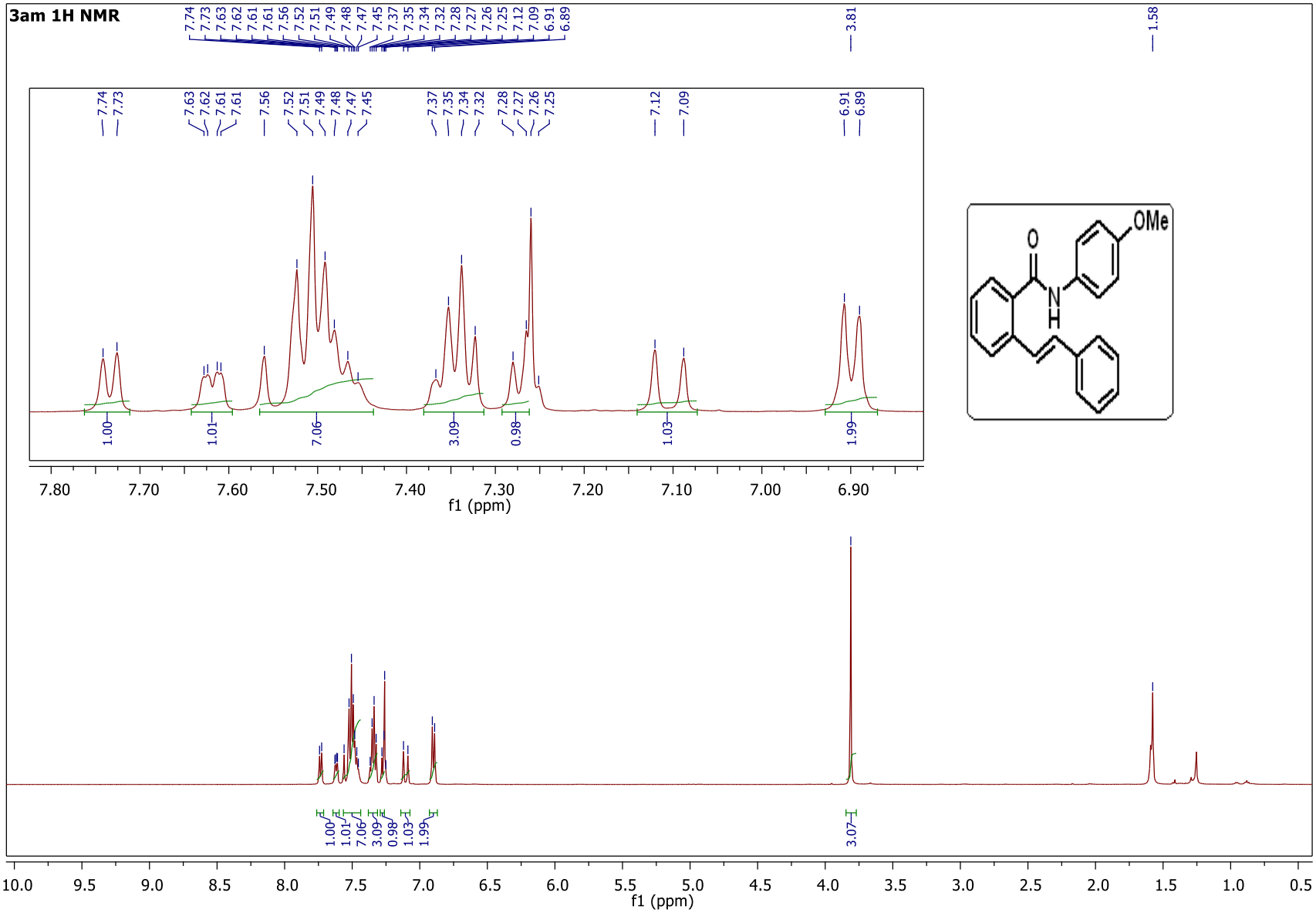
Calculated Mass: 310.0902

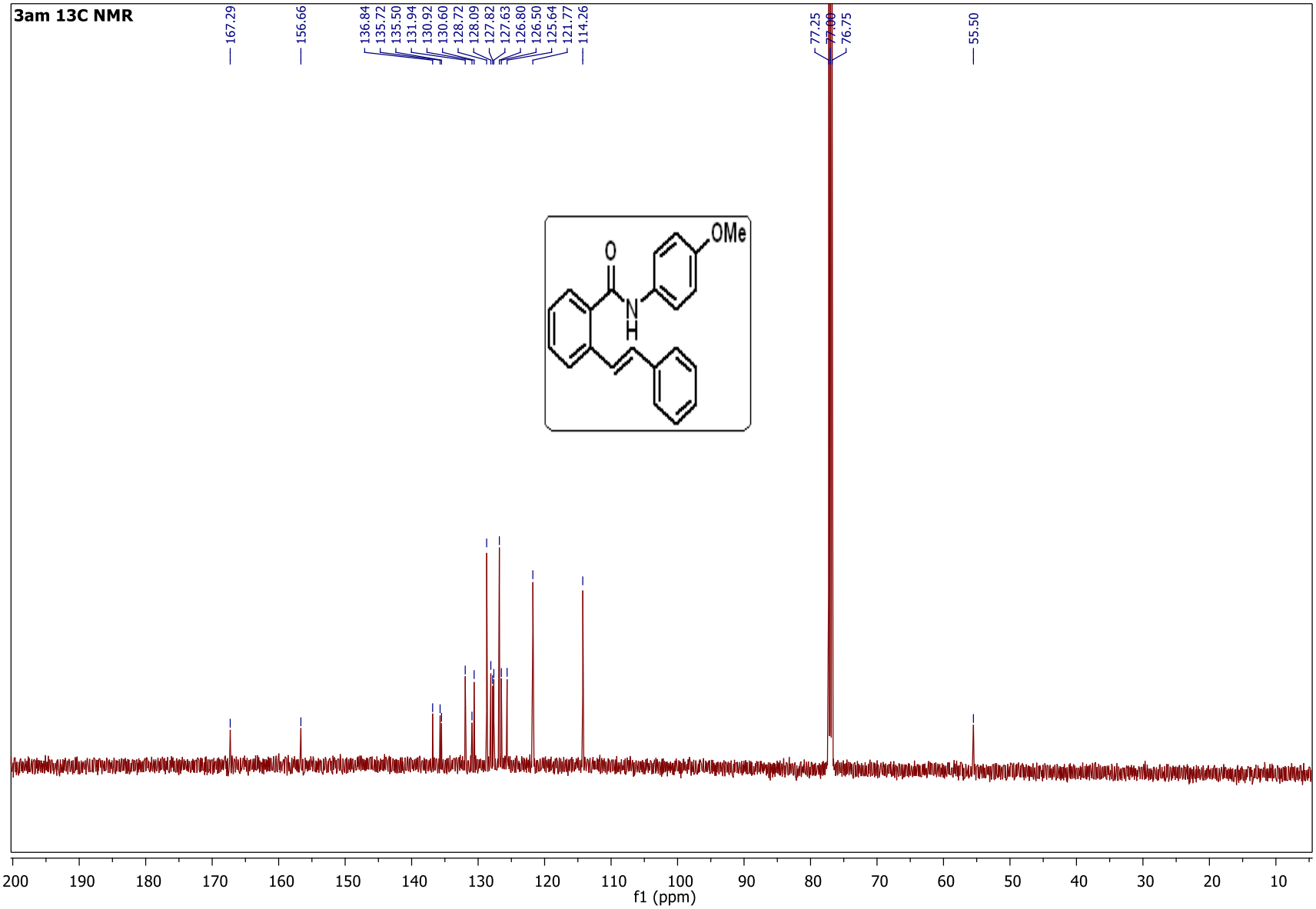

3al 1H NMR
7.91
7.90
7.63
7.62
7.56
7.54
7.45
7.43
7.41
7.39
7.38
7.29
7.28
7.26
7.23
7.21
7.20
7.07
6.87
6.85
3.80
1.66
1.00
1.00
2.06
2.19
3.01
1.18
1.04
0.98
2.02
3.06
f1 (ppm)
OMe
O
N
H
O


3al 1H NMR
167.83
156.74
154.88
153.22
135.21
130.73
130.13
128.86
128.76
128.25
128.03
127.60
124.80
123.03
122.11
121.34
114.19
111.19
105.33
77.25
77.00
76.75
55.46
OMe
O
N
H
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

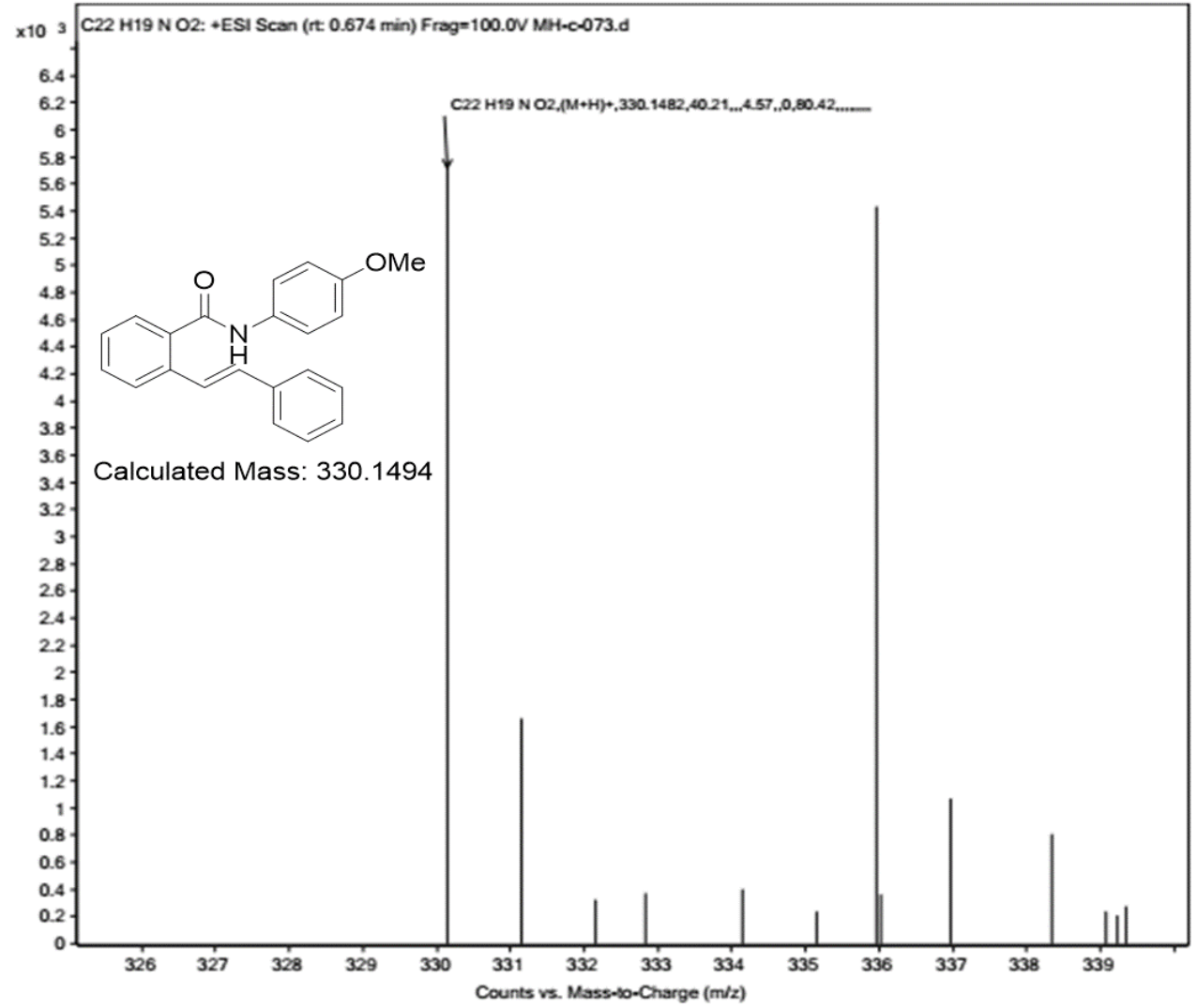
Sample Name	MHjC-152	**Position**		**Instrument Name**	Instrument 1
User Name		**Inj Vol**	Unknown / Injection Program	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	MH-C-152.d
ACQ Method	Demo.m	**Comment**		**Acquired Time**	09-04-2018 11:19:34 (UTC+05:30)

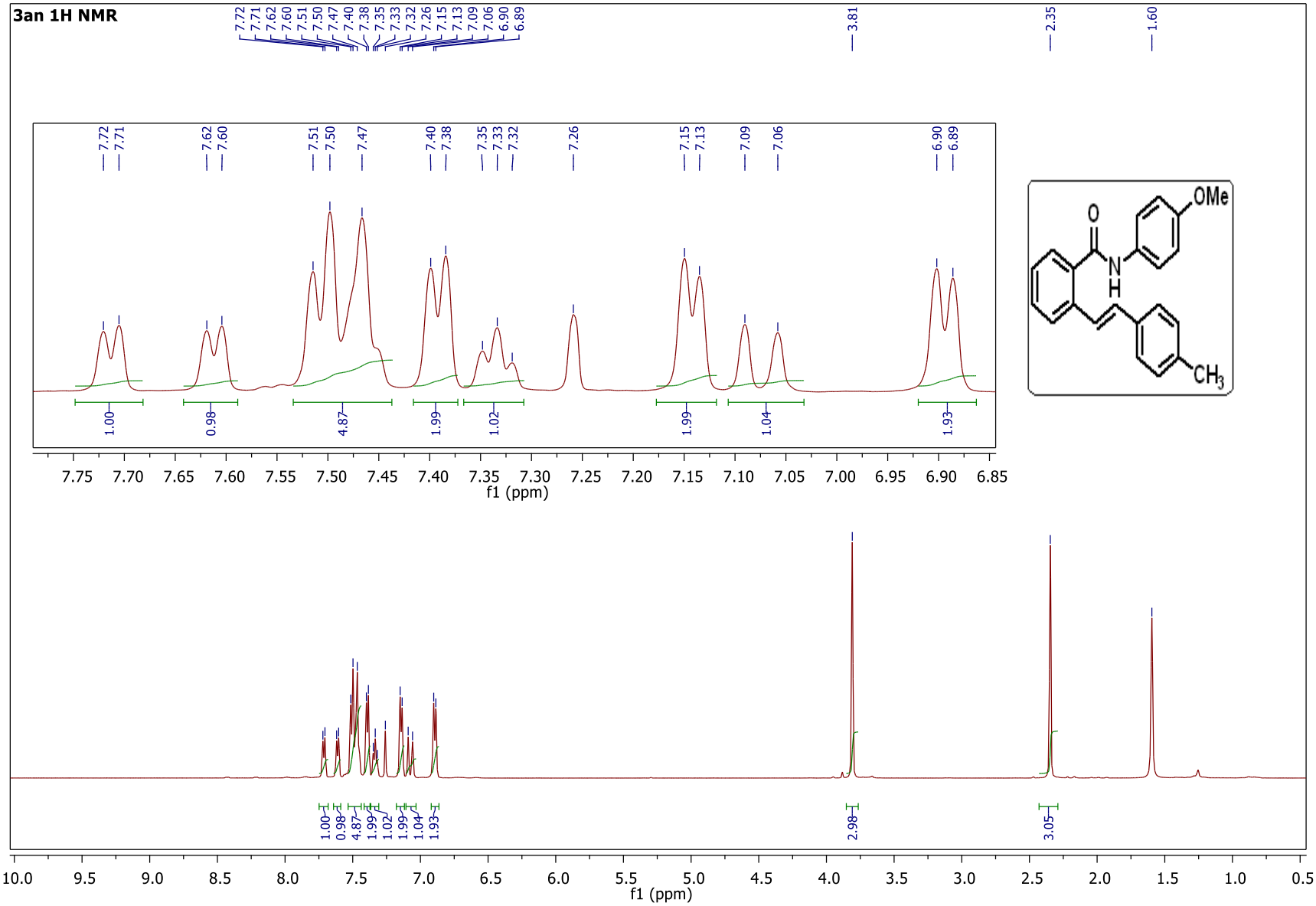


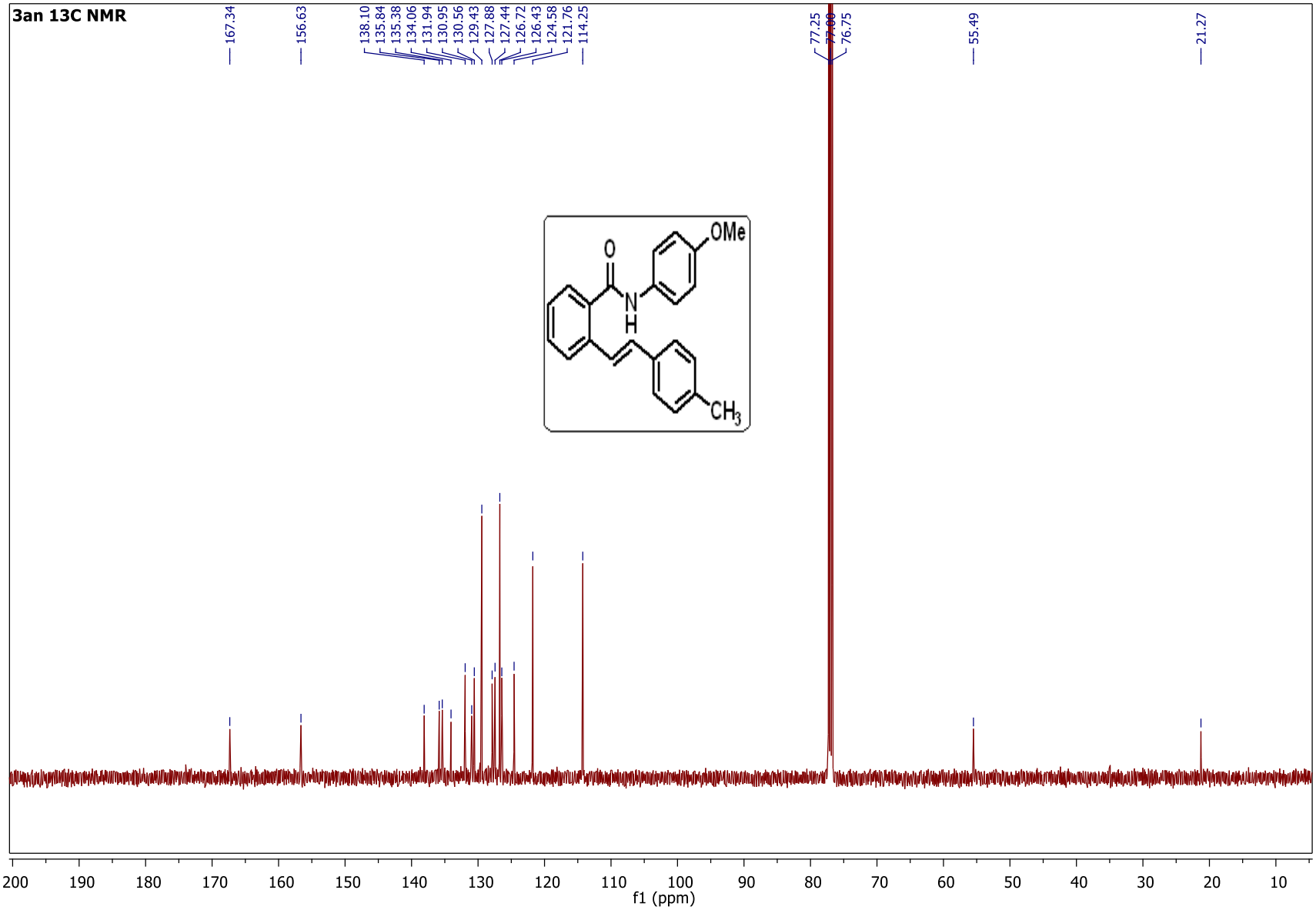

3am 1H NMR
7.74
7.73
7.63
7.62
7.61
7.61
7.56
7.52
7.51
7.49
7.48
7.47
7.45
7.37
7.35
7.34
7.32
7.28
7.27
7.26
7.25
7.12
7.09
6.91
6.89
3.81
1.58
1.00
1.01
7.06
3.09
0.98
1.03
1.99
3.07
OMe
O
N
H
f1 (ppm)
f1 (ppm)


3am 13C NMR
167.29
156.66
136.84
135.72
135.50
131.94
130.92
130.60
128.72
128.09
127.82
127.63
126.80
126.50
125.64
121.77
114.26
77.25
77.00
76.75
55.50
OMe
O
N
H
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

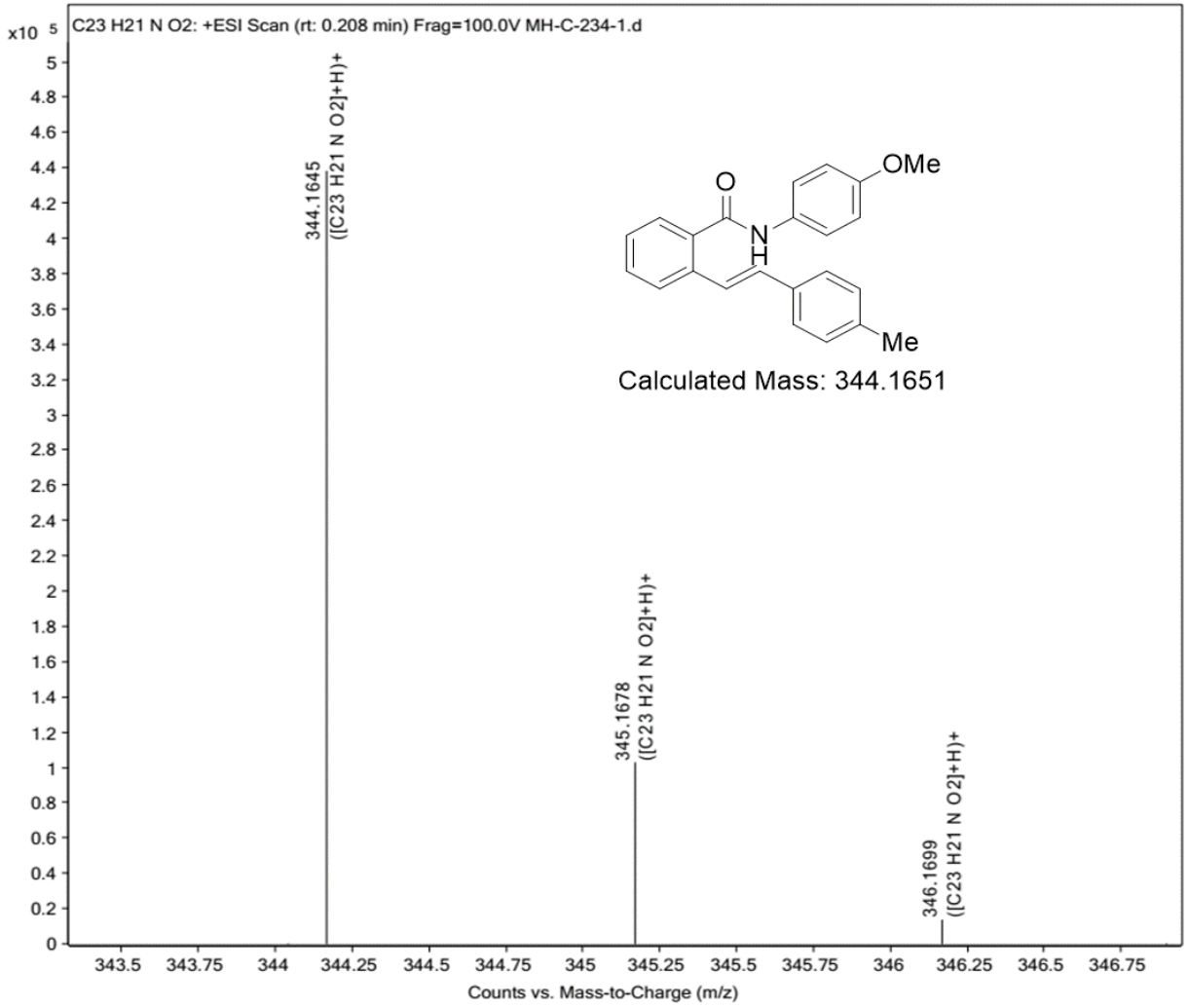
Sample Name	MH-c-073	**Position**		**Instrument Name**	Instrument 1
User Name		**Inj Vol**	Unknown / Injection Program	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	MH-c-073.d
ACQ Method	Demo.m	**Comment**		**Acquired Time**	14-03-2018 06:03:36 (UTC+05:30)

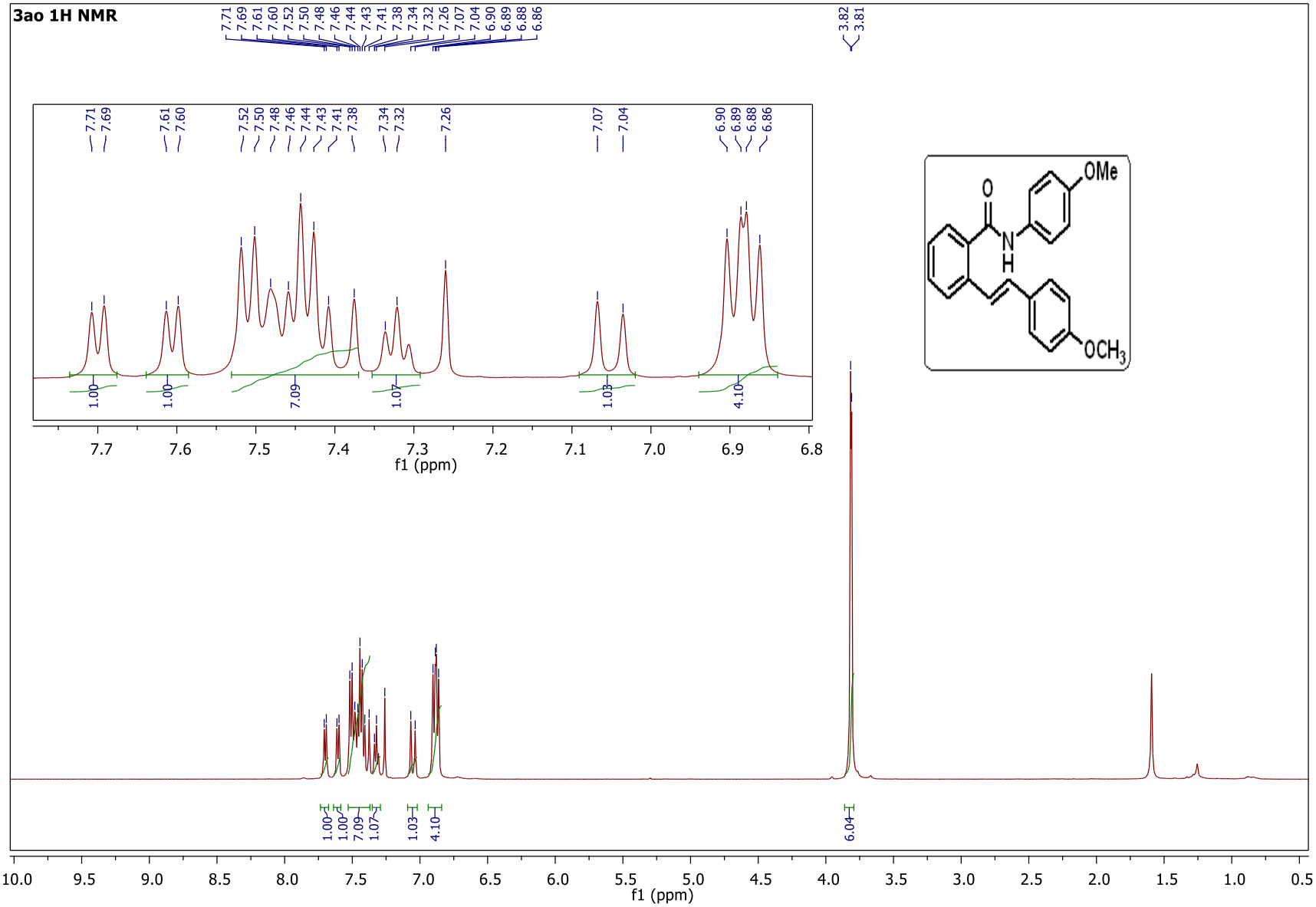


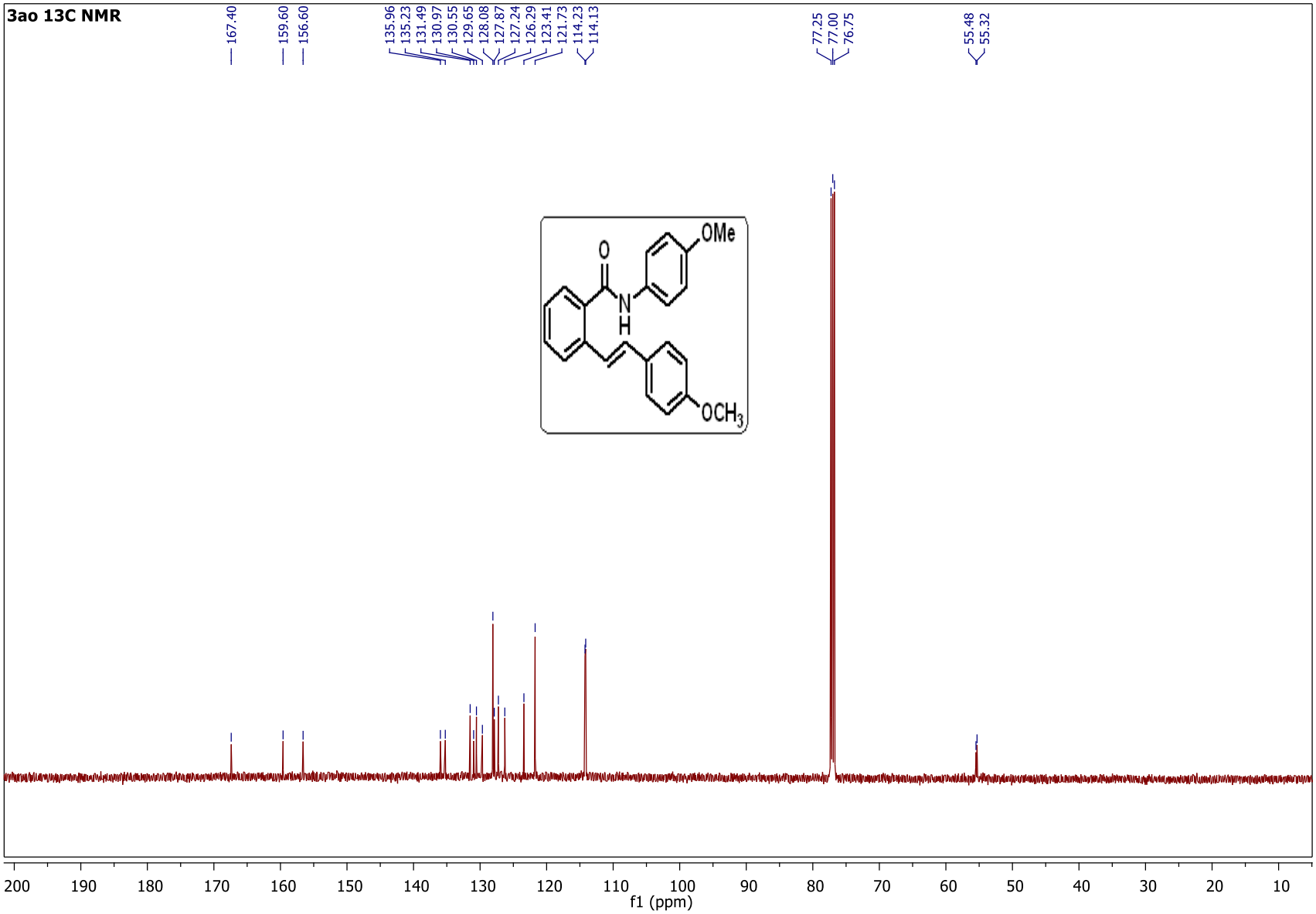

3an 1H NMR
7.72
7.71
7.62
7.60
7.51
7.50
7.47
7.40
7.38
7.35
7.33
7.32
7.26
7.15
7.13
7.09
7.06
6.90
6.89
3.81
2.35
1.60
1.00
0.98
4.87
1.99
1.02
1.99
1.04
1.93
2.98
3.05
f1 (ppm)
OMe
O
N
H
CH3


3an 13C NMR
167.34
156.63
138.10
135.84
135.38
134.06
131.94
130.95
130.56
129.43
127.88
127.44
126.72
126.43
124.58
121.76
114.25
77.25
77.00
76.75
55.49
21.27
OMe
O
N
H
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

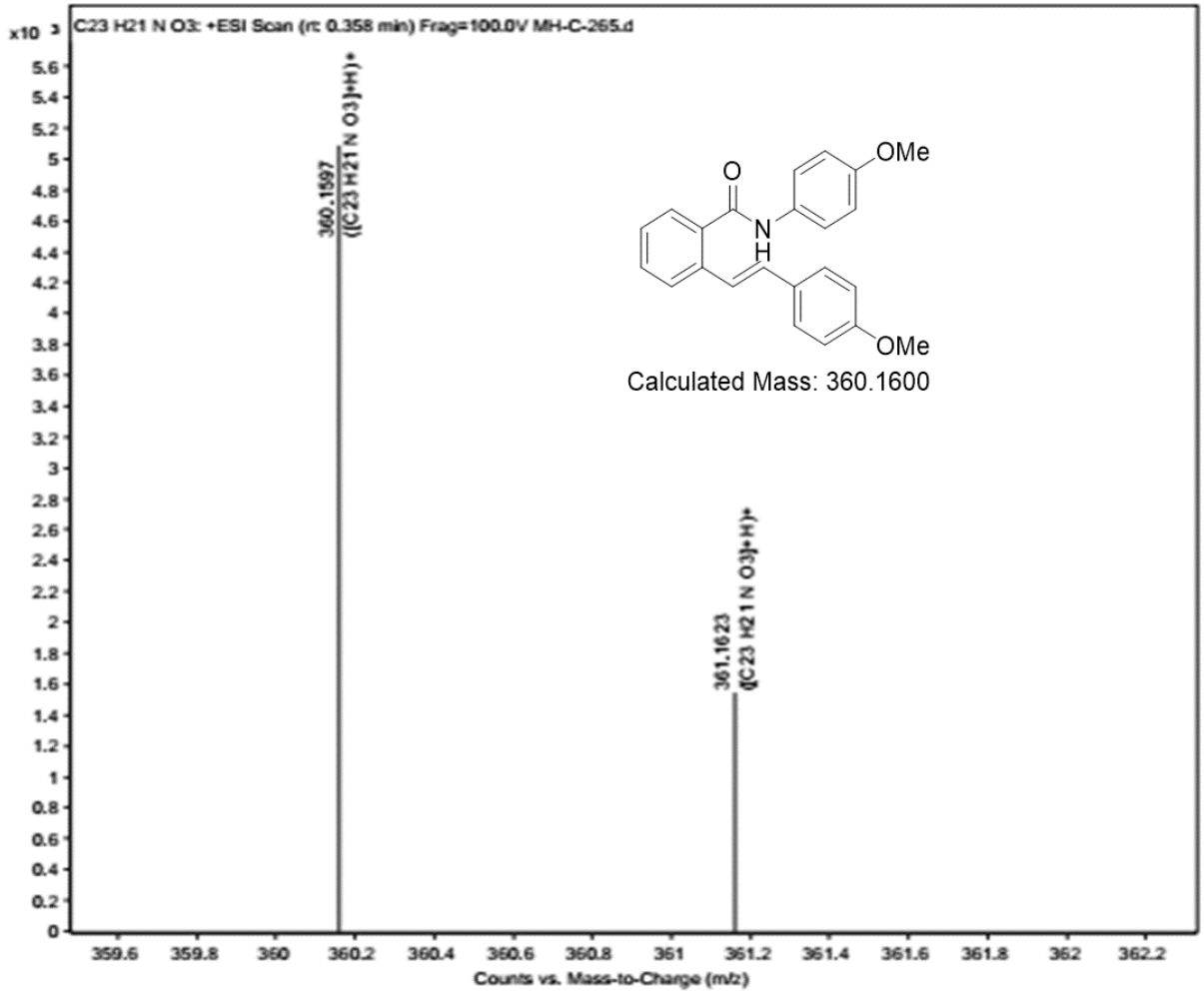
Sample Name	MH/C-234	**Position**		**Instrument Name**	Instrument 1
User Name		**Inj Vol**	Unknown / Injection Program	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	MH-C-234-1.d
ACQ Method	Demo.m	**Comment**		**Acquired Time**	10-04-2018 03:56:36 (UTC+05:30)



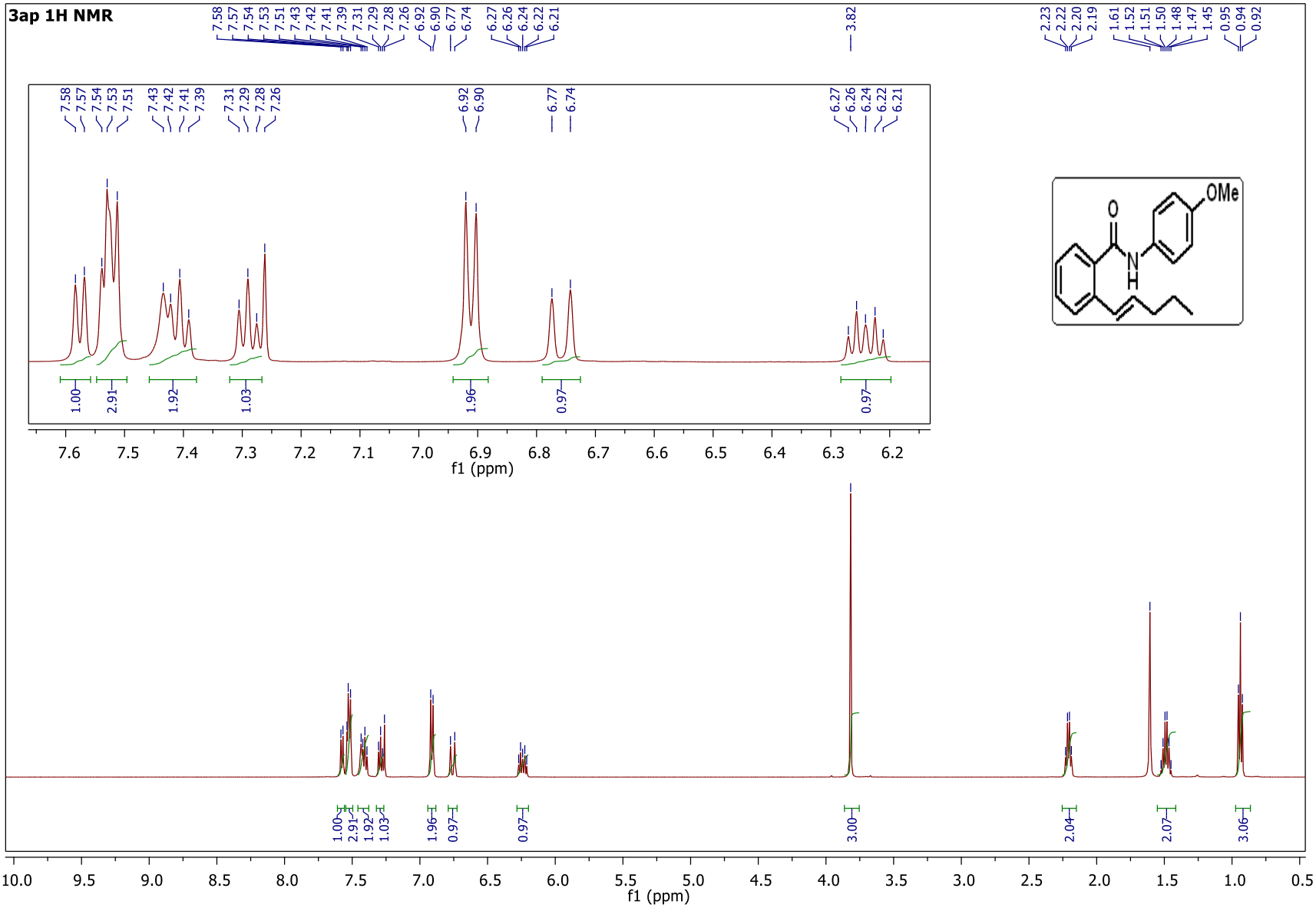

3ao 1H NMR
7.71
7.69
7.61
7.60
7.52
7.50
7.48
7.46
7.44
7.43
7.41
7.38
7.34
7.32
7.26
7.07
7.04
6.90
6.89
6.88
6.86
3.82
3.81
1.00
1.00
7.09
1.07
1.03
4.10
6.04
f1 (ppm)
OMe
O
N
H
OCH3

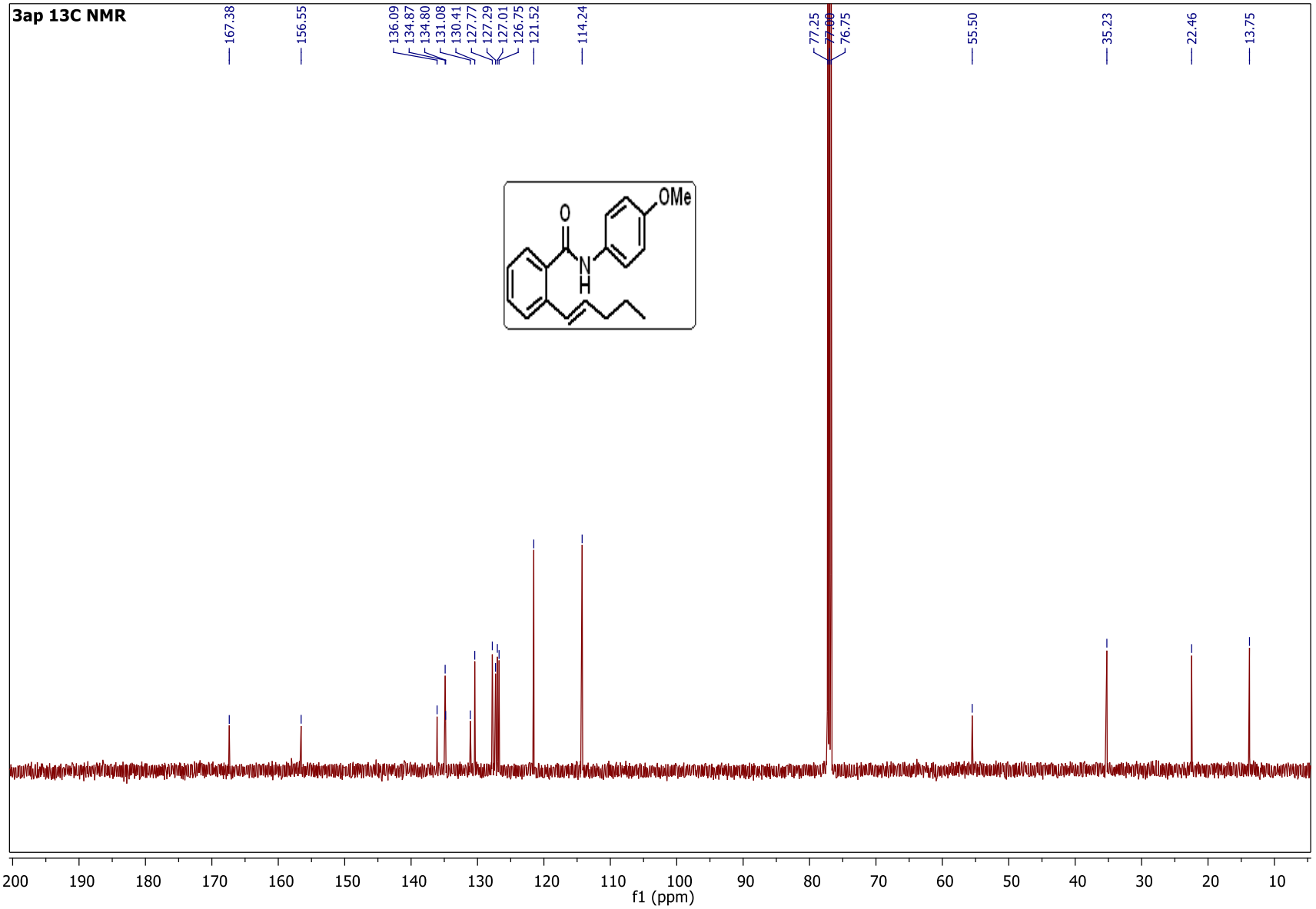

3ao 13C NMR
167.40
159.60
156.60
135.96
135.23
131.49
130.97
130.55
129.65
128.08
127.87
127.24
126.29
123.41
121.73
114.23
114.13
77.25
77.00
76.75
55.48
55.32
OMe
O
N
H
OCH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

Sample Name	MH-C-265	**Position**		**Instrument Name**	Instrument 1
User Name		**Inj Vol**	Unknown / Injection Program	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	MH-C-265.d
ACQ Method	Demo.m	**Comment**		**Acquired Time**	09-04-2018 13:01:53 (UTC+05:30)

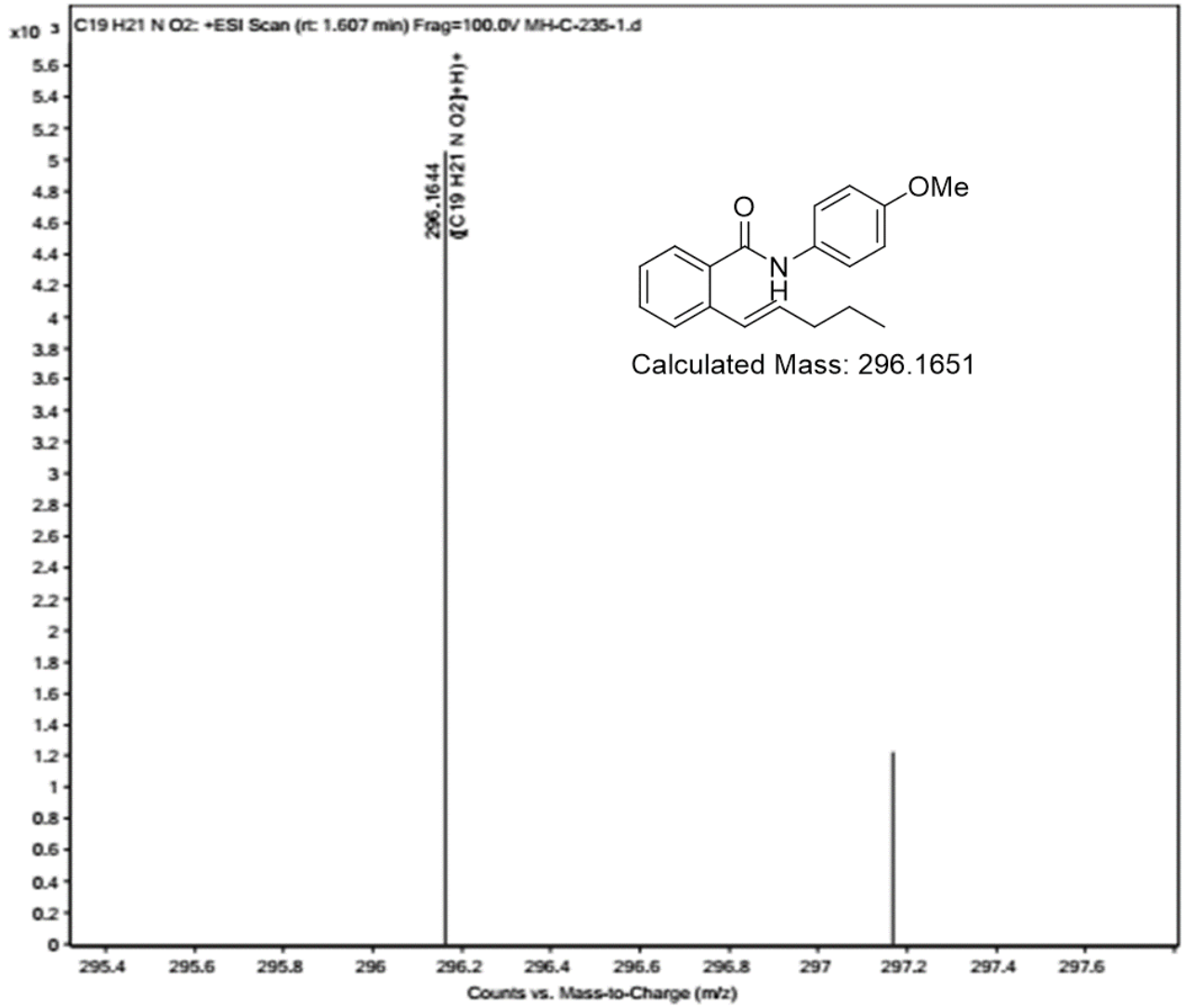


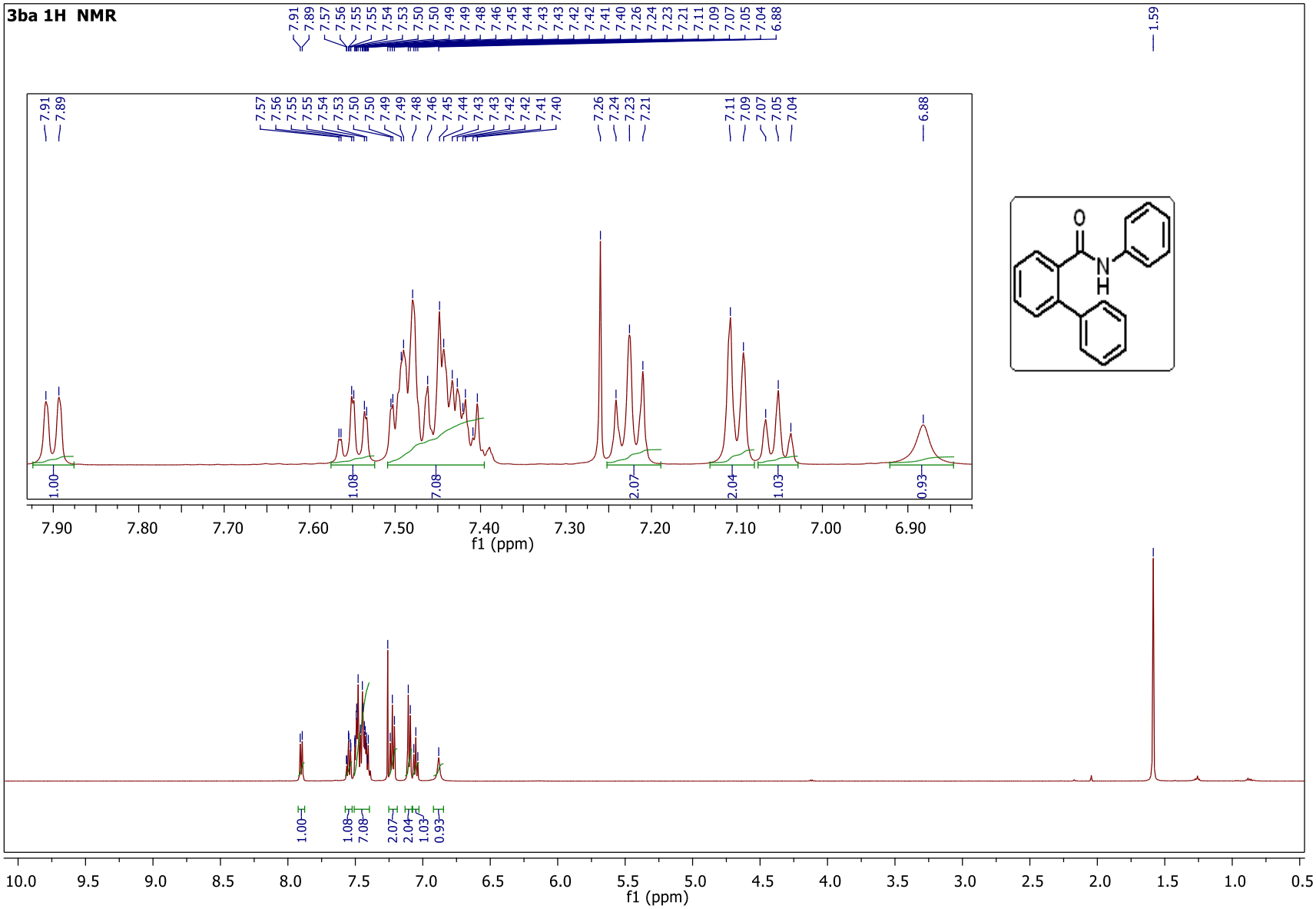
Calculated Mass: 360.1600

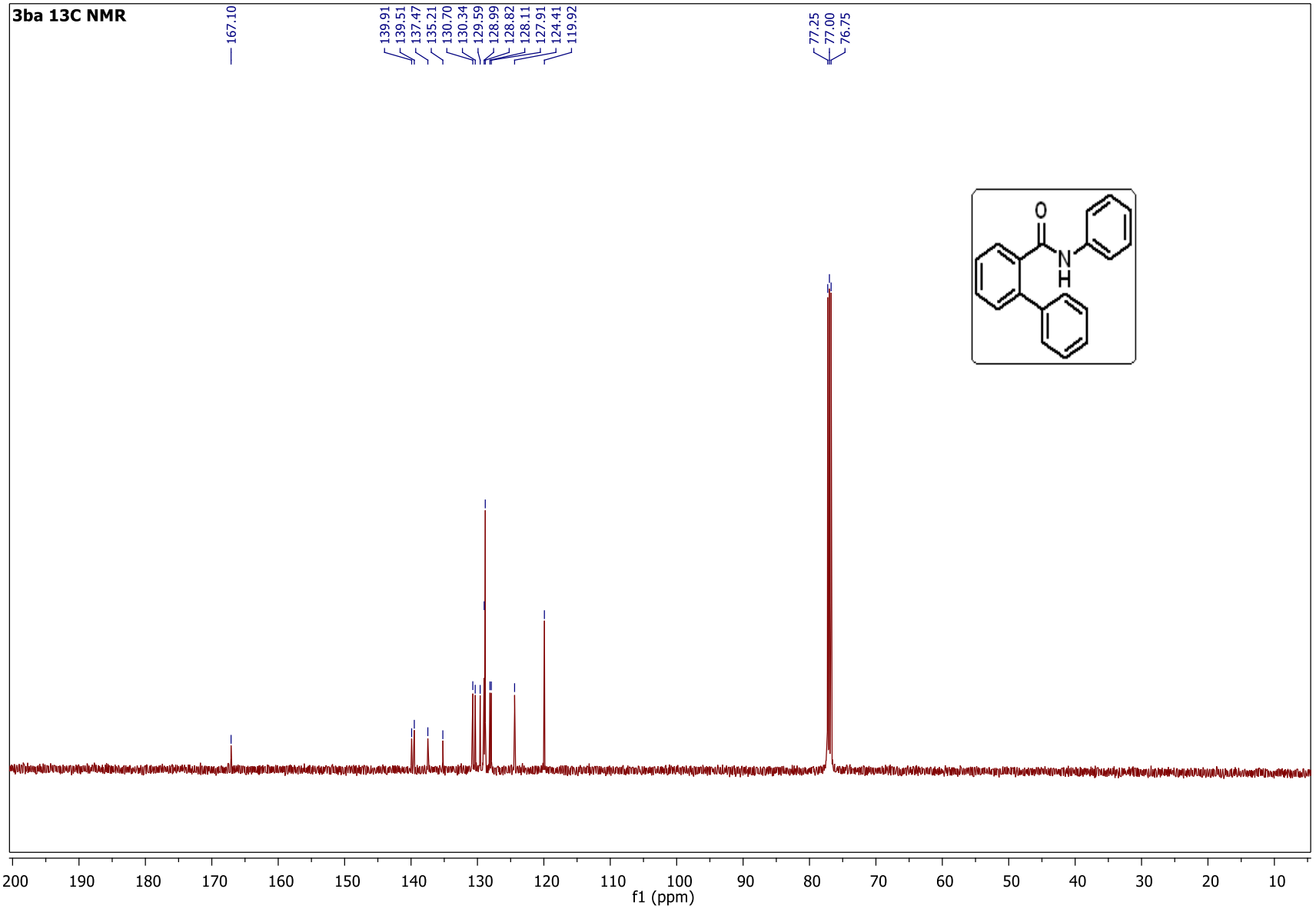

3ap 1H NMR
7.58 7.57 7.54 7.53 7.51 7.43 7.42 7.41 7.39 7.31 7.29 7.28 7.26 6.92 6.90 6.77 6.74 6.27 6.26 6.24 6.22 6.21 3.82 2.23 2.22 2.20 2.19 1.61 1.52 1.51 1.50 1.48 1.47 1.45 0.95 0.94 0.92
1.00 2.91 1.92 1.03 1.96 0.97 0.97
7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2
f1 (ppm)
OMe
O
N
H
1.00 2.91 1.92 1.03 1.96 0.97 0.97 3.00 2.04 2.07 3.06
10.0 9.5 9.0 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 0.5
f1 (ppm)


3ap 13C NMR
167.38
156.55
136.09
134.87
134.80
131.08
130.41
127.77
127.29
127.01
126.75
121.52
114.24
77.25
77.00
76.75
55.50
35.23
22.46
13.75
O
N
H
OMe
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

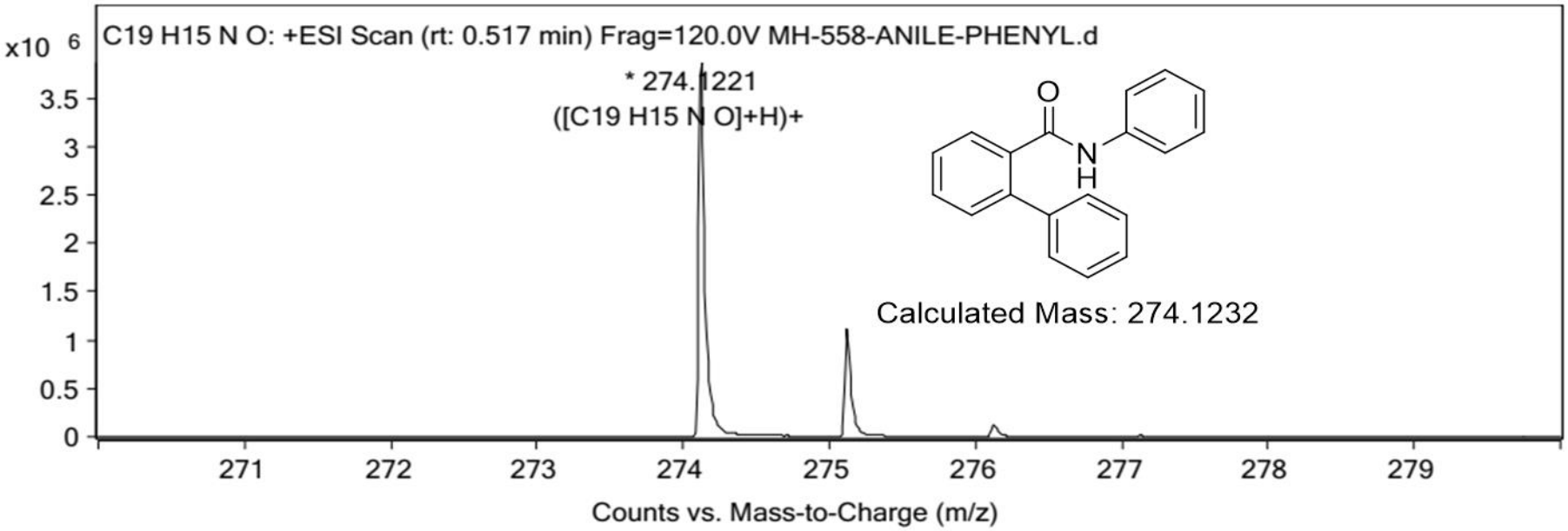
Sample Name	MH/C-235-1	**Position**		**Instrument Name**	Instrument 1
User Name		**Inj Vol**	Unknown / Injection Program	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	MH-C-235-1.d
ACQ Method	Demo.m	**Comment**		**Acquired Time**	09-04-2018 11:37:27 (UTC+05:30)

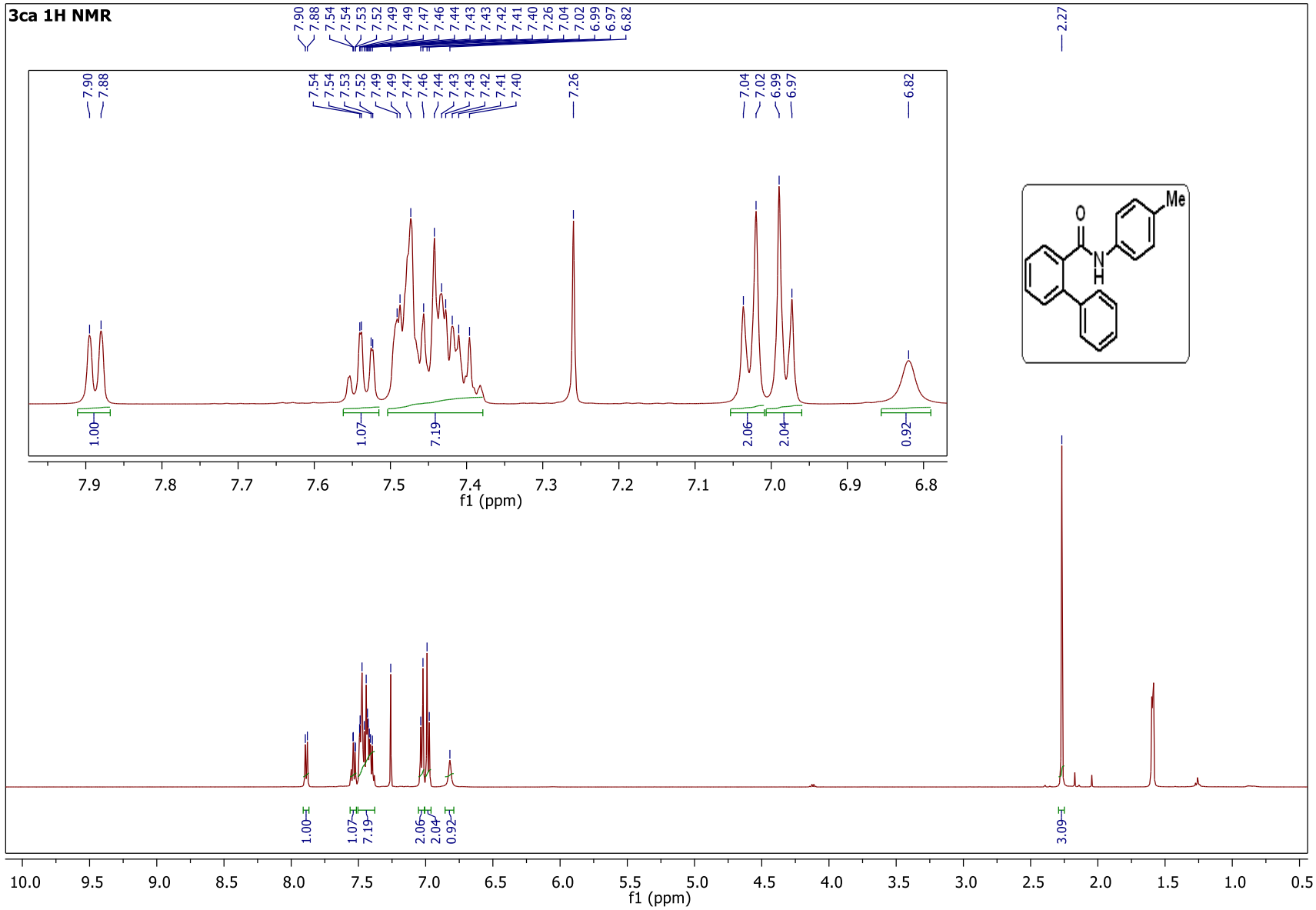


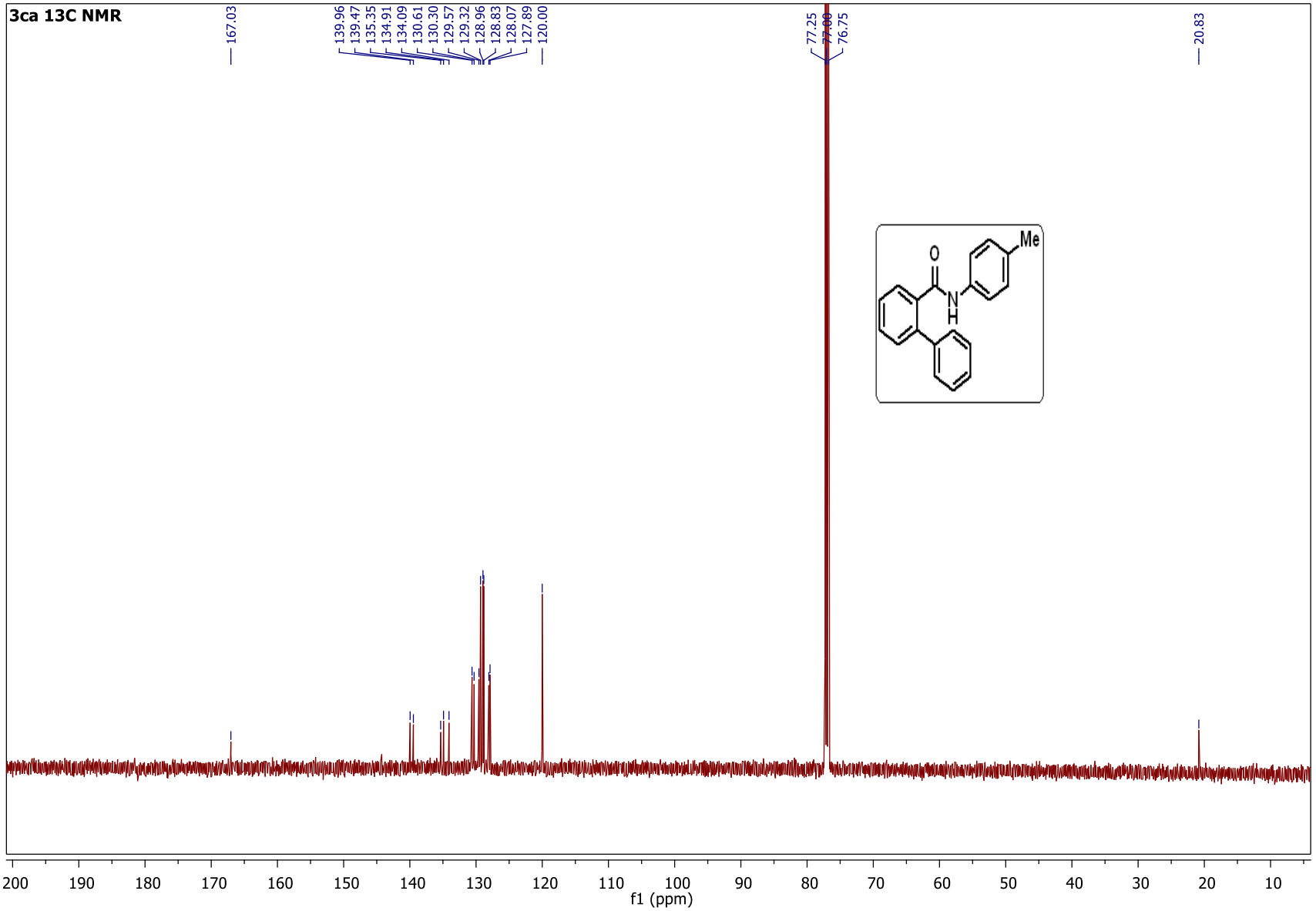

3ba 1H NMR
7.91
7.89
7.57
7.56
7.55
7.55
7.54
7.53
7.50
7.50
7.49
7.49
7.48
7.46
7.45
7.44
7.43
7.43
7.42
7.42
7.41
7.40
7.26
7.24
7.23
7.21
7.11
7.09
7.07
7.05
7.04
6.88
1.59
1.00
1.08
7.08
2.07
2.04
1.03
0.93
7.90
7.80
7.70
7.60
7.50
7.40
7.30
7.20
7.10
7.00
6.90
f1 (ppm)
O
N
H
10.0
9.5
9.0
8.5
8.0
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
0.5
f1 (ppm)


3ba 13C NMR
167.10
139.91
139.51
137.47
135.21
130.70
130.34
129.59
128.99
128.82
128.11
127.91
124.41
119.92
77.25
77.00
76.75
O
N
H
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

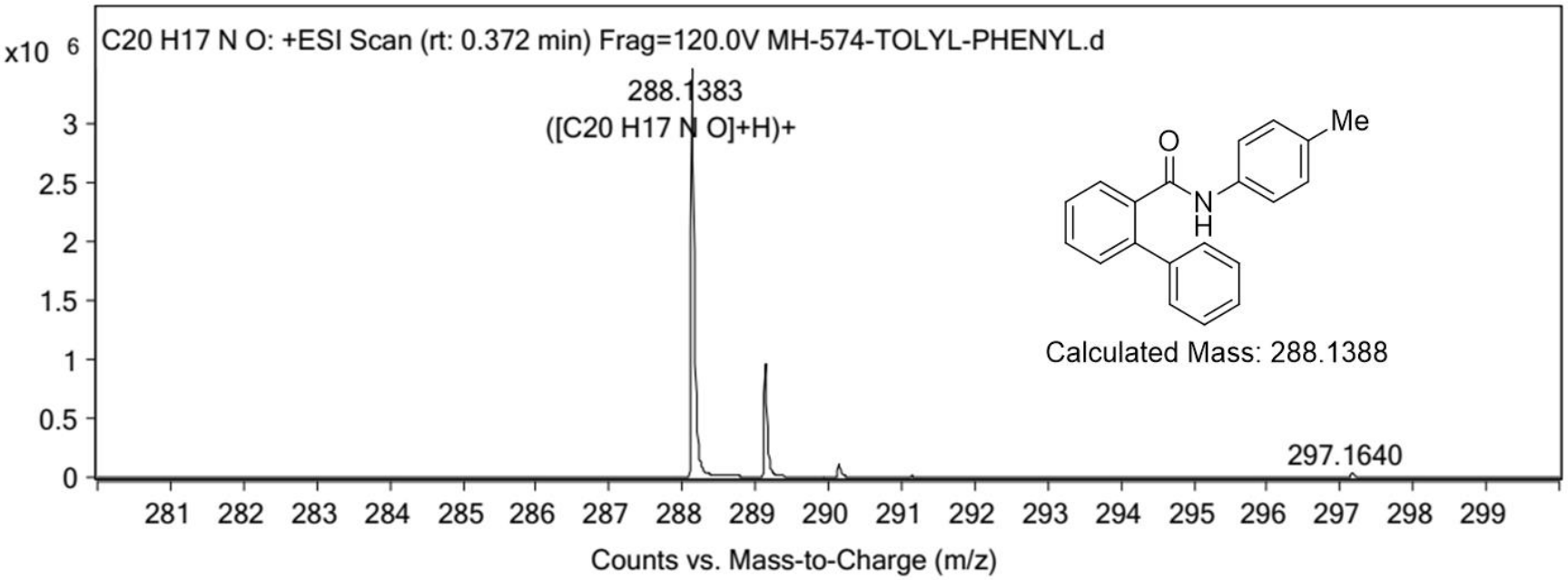
Data File	MH-558-ANILE-PHENYL.d	**Sample Name**	HARI
Sample Type	Sample	**Position**	
Instrument Name	Instrument 1	**User Name**	
Acq Method	Sample_pos_01.m	**Acquired Time**	06-12-2019 16:20:33 (UTC+05:30)
IRM Calibration Status	Success	**DA Method**	HARI.m
Comment			
Sample Group		**Info.**	
Stream Name		**Acquisition Time (Local)**	06-12-2019 16:20:33 (UTC+05:30)
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.08.00 (B8058.0)	**TOF Driver Version**	8.00.00
TOF Firmware Version	17.698	**Tune Mass Range Max.**	3200



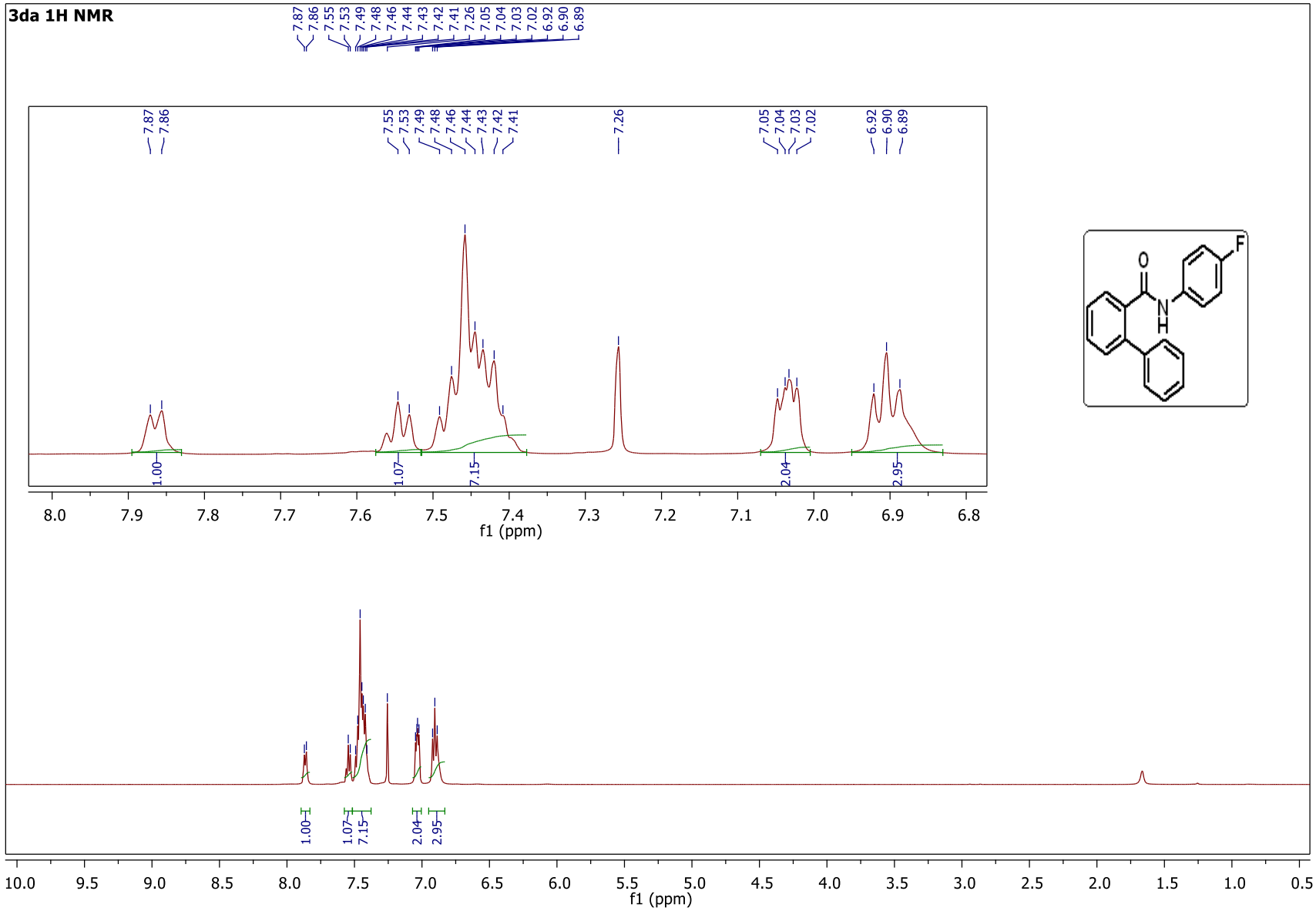

3ca 1H NMR
7.90
7.88
7.54
7.54
7.53
7.52
7.49
7.49
7.47
7.46
7.44
7.43
7.43
7.42
7.41
7.40
7.26
7.04
7.02
6.99
6.97
6.82
2.27
1.00
1.07
7.19
2.06
2.04
0.92
3.09
f1 (ppm)
O
N
H
Me

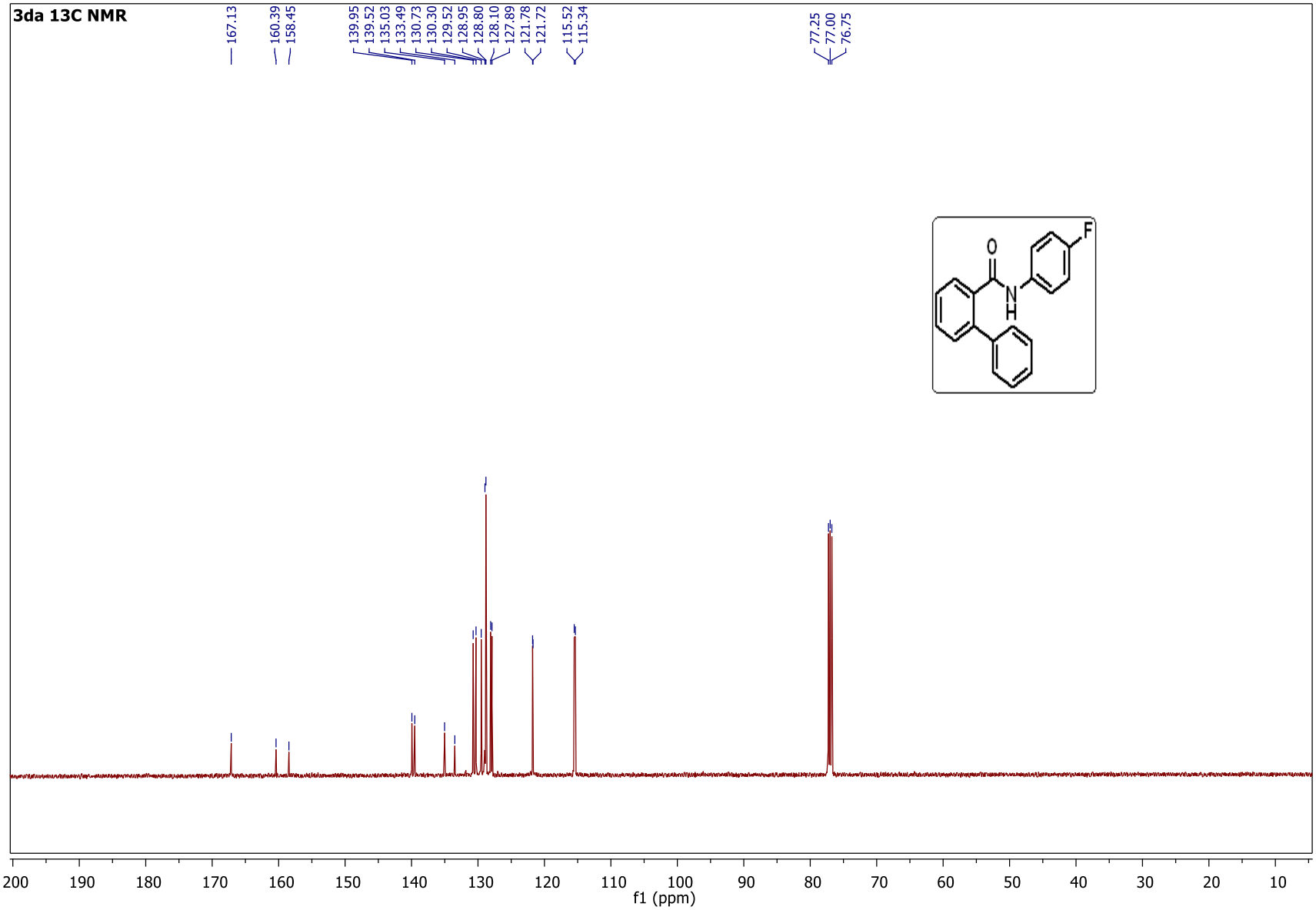

3ca 13C NMR
167.03
139.96
139.47
135.35
134.91
134.09
130.61
130.30
129.57
129.32
128.96
128.83
128.07
127.89
120.00
77.25
77.00
76.75
20.83
O
N
H
Me
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

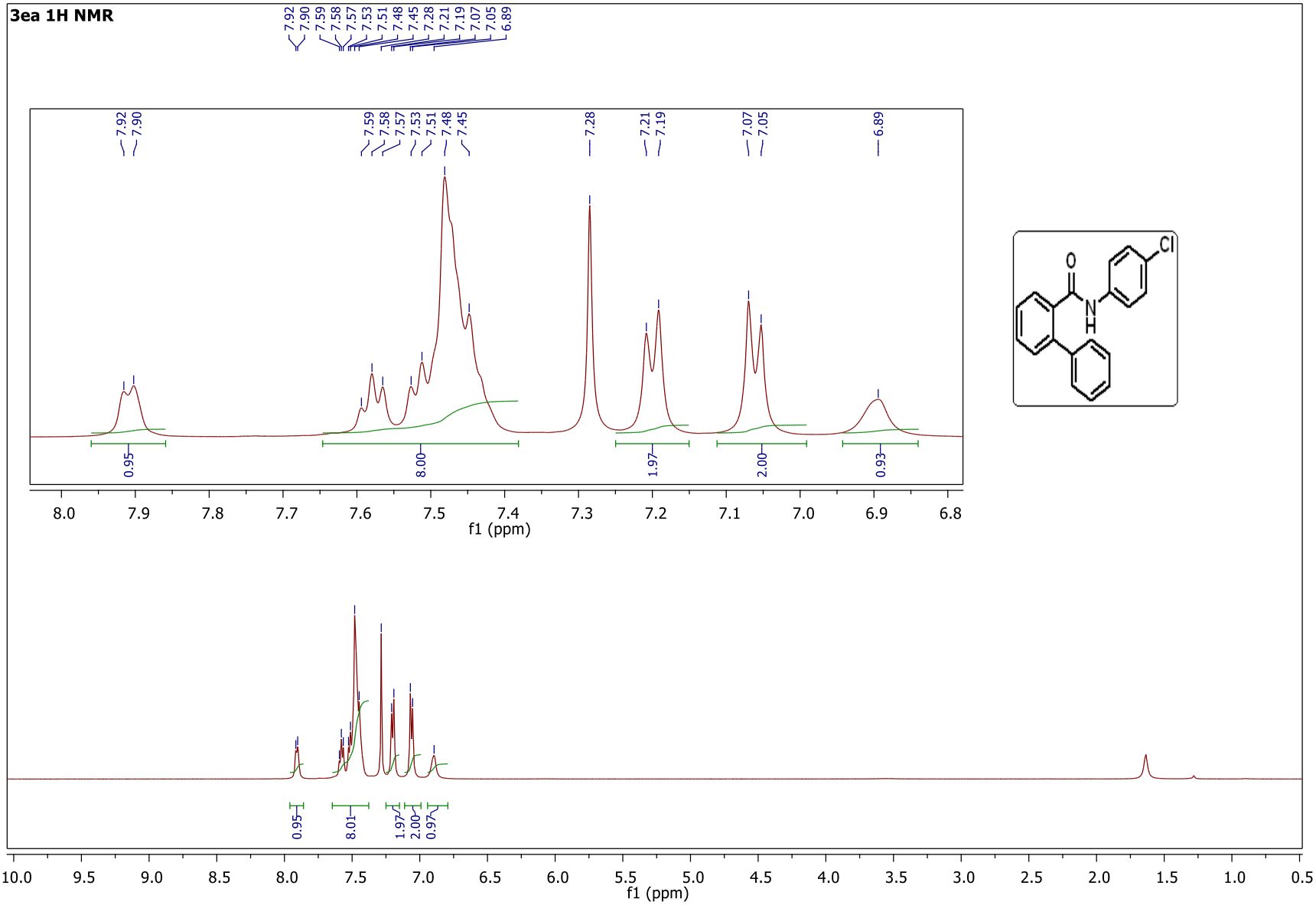
Data File	MH-574-TOLYL-PHENYL.d	**Sample Name**	HARI
Sample Type	Sample	**Position**	
Instrument Name	Instrument 1	**User Name**	
Acq Method	Sample_pos_01.m	**Acquired Time**	06-12-2019 17:05:12 (UTC+05:30)
IRM Calibration Status	Success	**DA Method**	HARI.m
Comment			
Sample Group		**Info.**	
Stream Name		**Acquisition Time (Local)**	06-12-2019 17:05:12 (UTC+05:30)
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.08.00 (B8058.0)	**TOF Driver Version**	8.00.00
TOF Firmware Version	17.698	**Tune Mass Range Max.**	3200

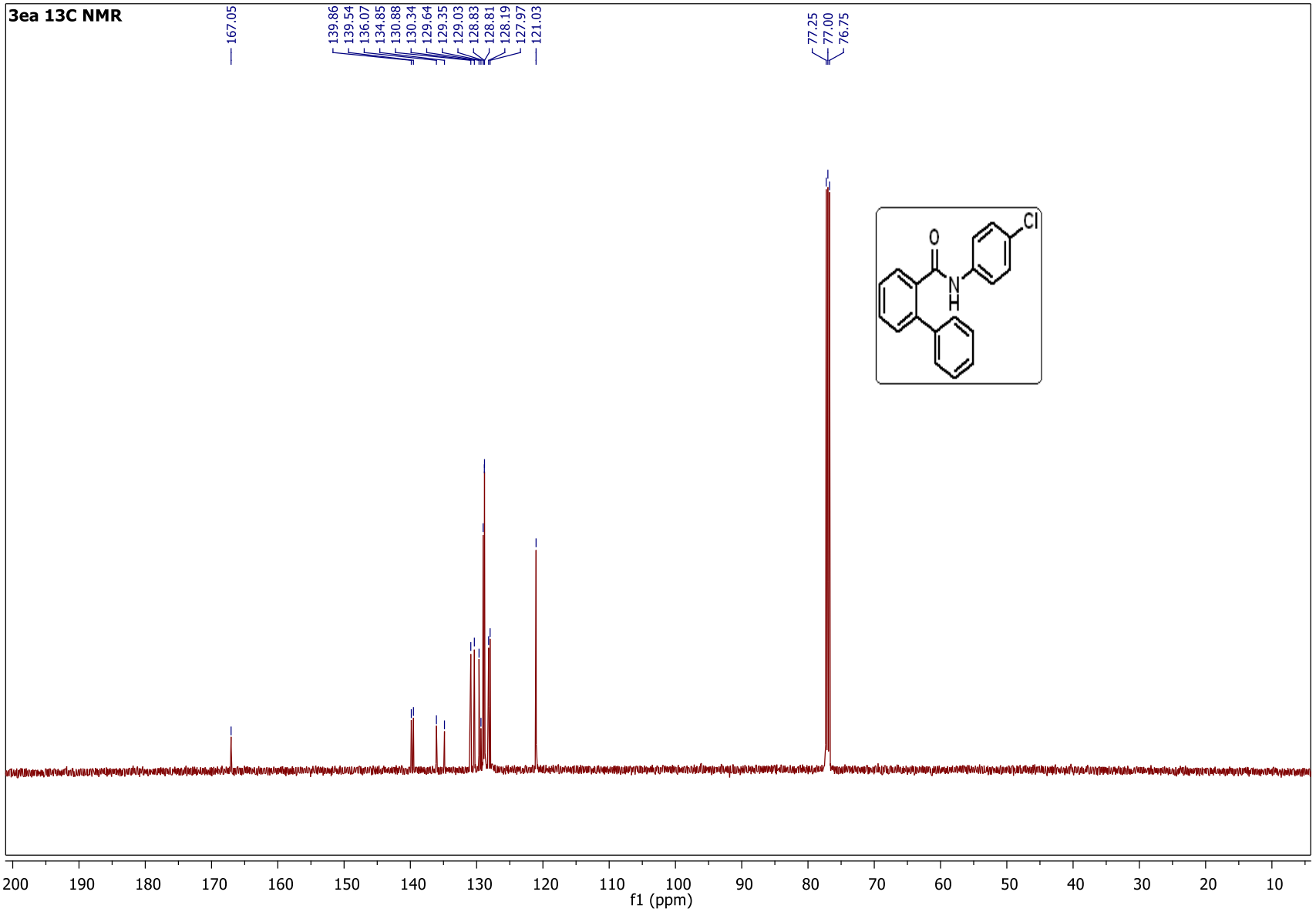


3da 1H NMR

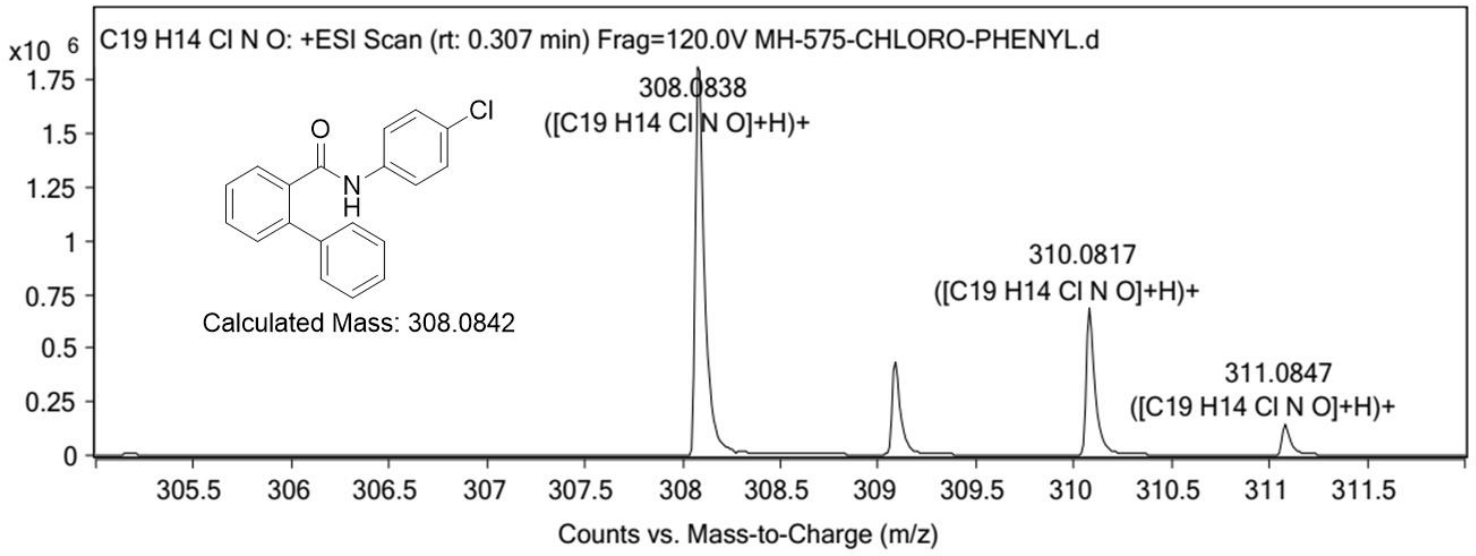

7.87
7.86
7.55
7.53
7.49
7.48
7.46
7.44
7.43
7.42
7.41
7.26
7.05
7.04
7.03
7.02
6.92
6.90
6.89
1.00
1.07
7.15
2.04
2.95
f1 (ppm)
F
O
N
H

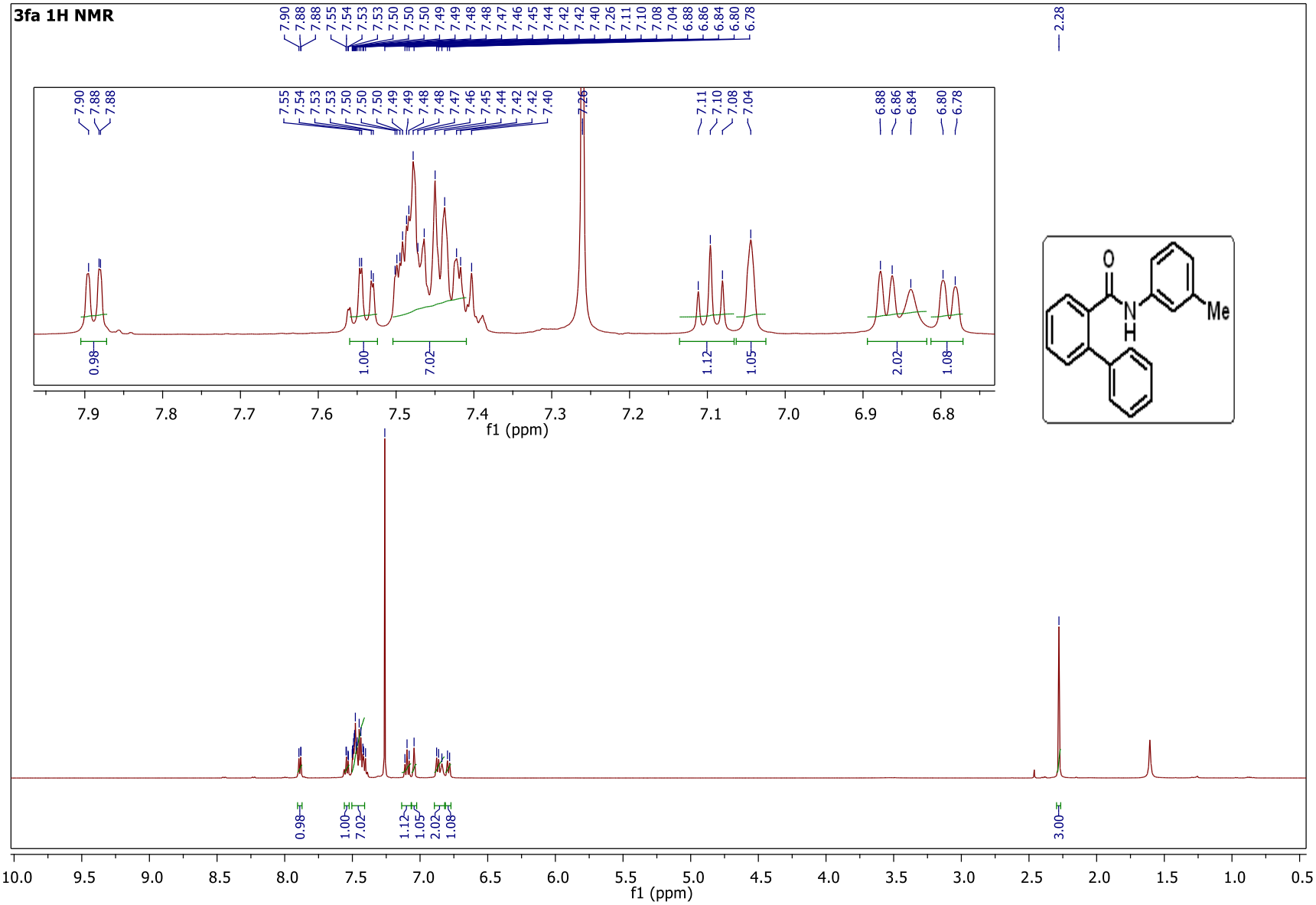

3da 13C NMR
167.13
160.39
158.45
139.95
139.52
135.03
133.49
130.73
130.30
129.52
128.95
128.80
128.10
127.89
121.78
121.72
115.52
115.34
77.25
77.00
76.75
O
N
H
F
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

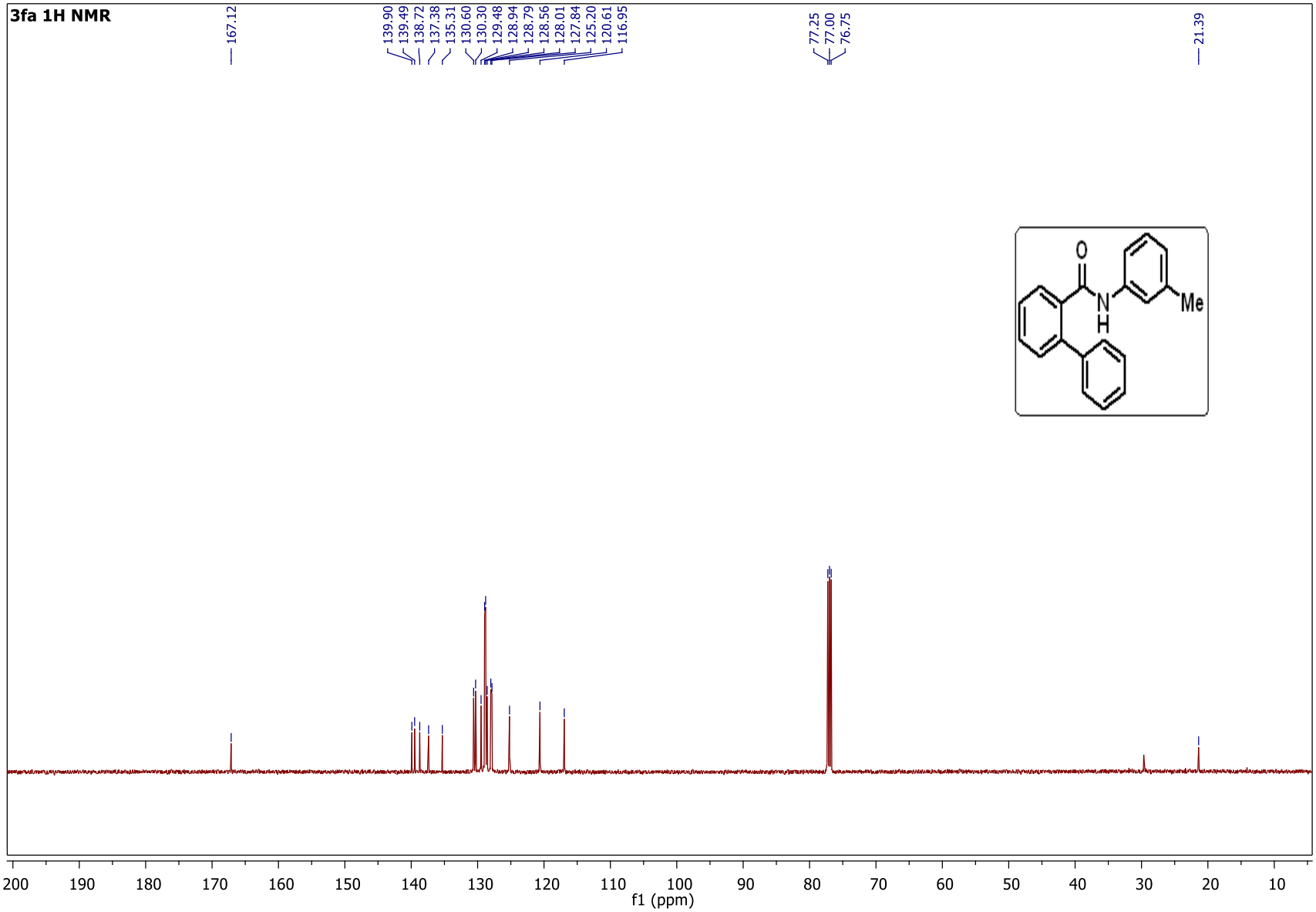

3ea 1H NMR
7.92
7.90
7.59
7.58
7.57
7.53
7.51
7.48
7.45
7.28
7.21
7.19
7.07
7.05
6.89
0.95
8.00
1.97
2.00
0.93
f1 (ppm)
O
N
H
Cl
0.95
8.01
1.97
2.00
0.97
f1 (ppm)


3ea 13C NMR
167.05
139.86
139.54
136.07
134.85
130.88
130.34
129.64
129.35
129.03
128.83
128.81
128.19
127.97
121.03
77.25
77.00
76.75
O
N
H
Cl
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

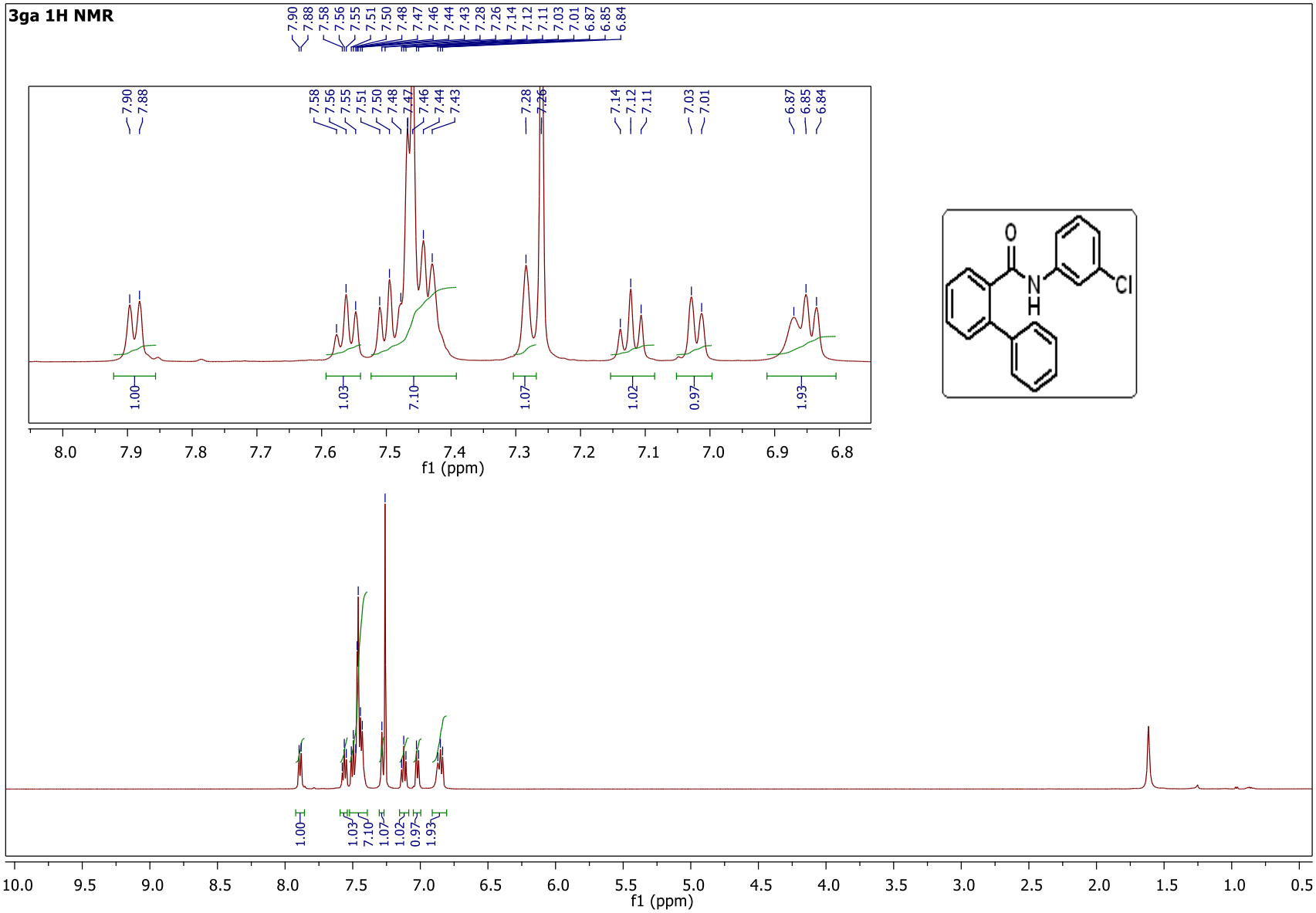
Data File	MH-575-CHLORO-PHENYL.d	**Sample Name**	HARI
Sample Type	Sample	**Position**	
Instrument Name	Instrument 1	**User Name**	
Acq Method	Sample_pos_01.m	**Acquired Time**	06-12-2019 16:58:53 (UTC+05:30)
IRM Calibration Status	Success	**DA Method**	HARI.m
Comment			
Sample Group		**Info.**	
Stream Name		**Acquisition Time (Local)**	06-12-2019 16:58:53 (UTC+05:30)
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.08.00 (B8058.0)	**TOF Driver Version**	8.00.00
TOF Firmware Version	17.698	**Tune Mass Range Max.**	3200

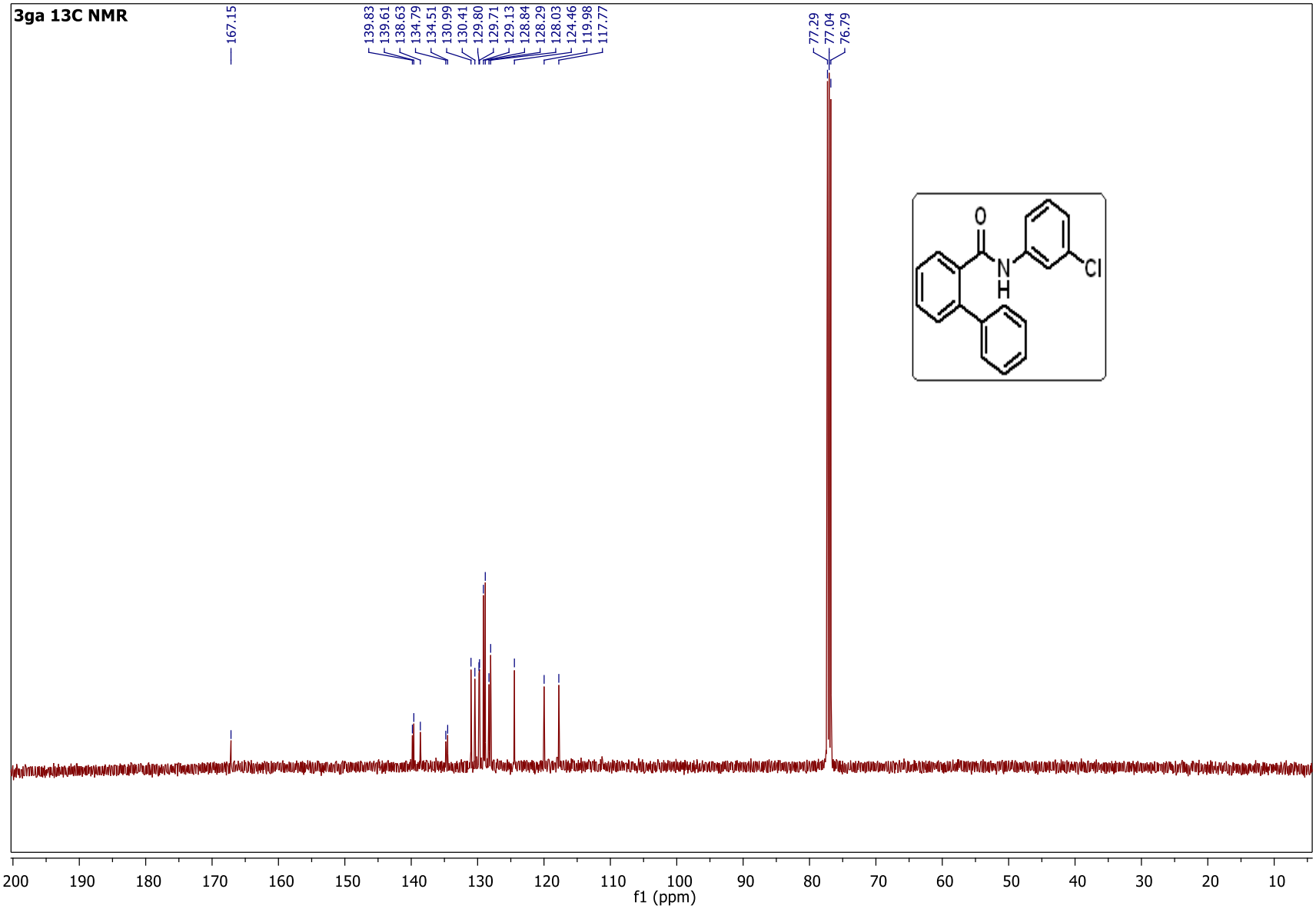


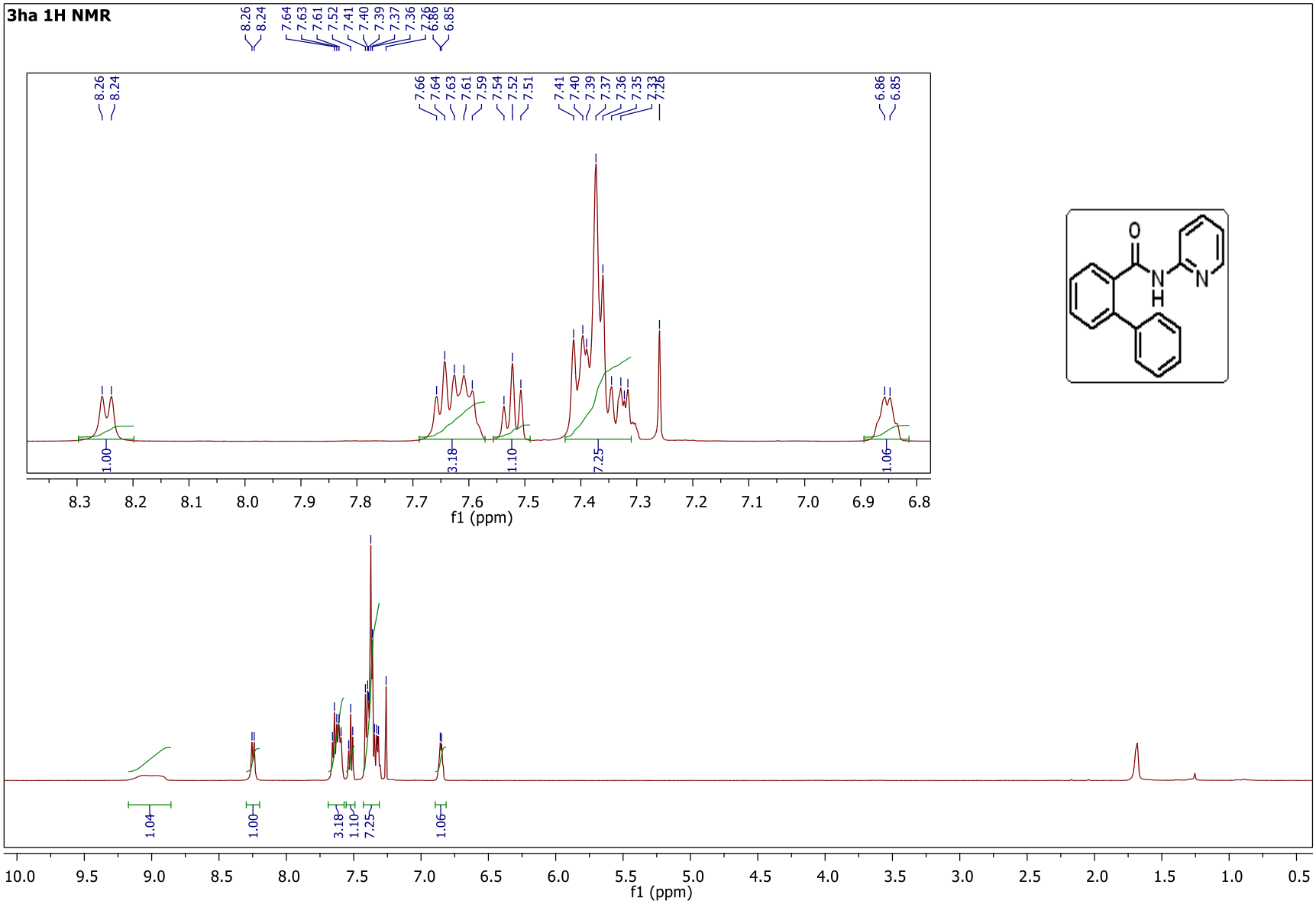

3fa 1H NMR
7.90 7.88 7.88 7.55 7.54 7.53 7.53 7.50 7.50 7.50 7.49 7.49 7.48 7.47 7.46 7.45 7.44 7.42 7.42 7.40 7.26 7.11 7.10 7.08 7.04 6.88 6.86 6.84 6.80 6.78
2.28
0.98 1.00 7.02 1.12 1.05 2.02 1.08
f1 (ppm)
O
N
H
Me
0.98 1.00 7.02 1.12 1.05 2.02 1.08 3.00
f1 (ppm)

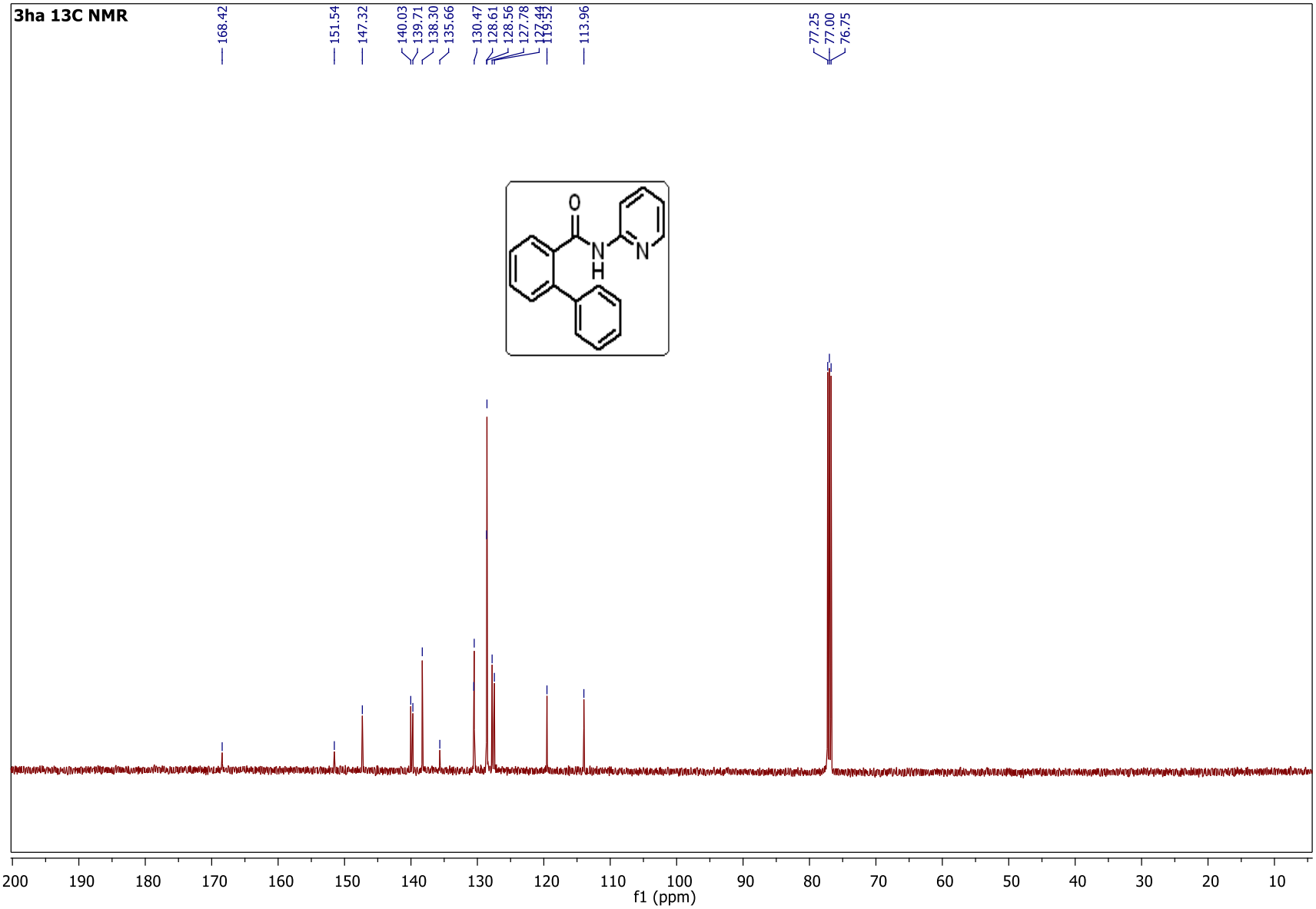

3fa 1H NMR
167.12
139.90
139.49
138.72
137.38
135.31
130.60
130.30
129.48
128.94
128.79
128.56
128.01
127.84
125.20
120.61
116.95
77.25
77.00
76.75
21.39
O
N
H
Me
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

3ga 1H NMR

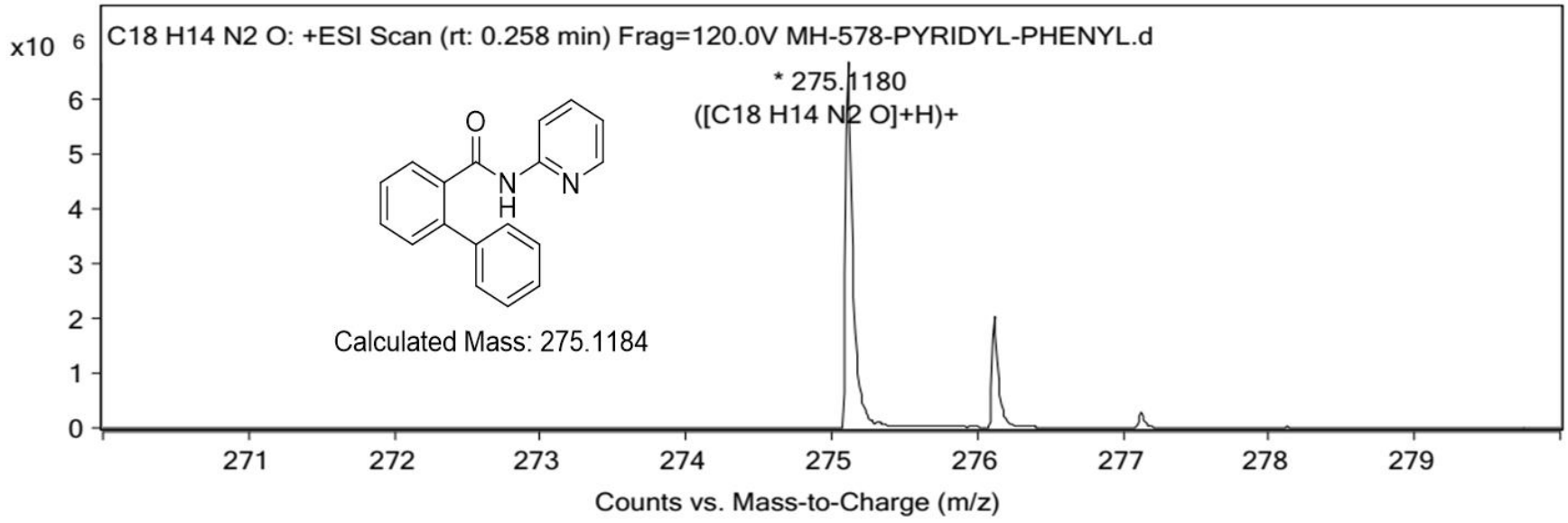

7.90
7.88
7.58
7.56
7.55
7.51
7.50
7.48
7.47
7.46
7.44
7.43
7.28
7.26
7.14
7.12
7.11
7.03
7.01
6.87
6.85
6.84
1.00
1.03
7.10
1.07
1.02
0.97
1.93
f1 (ppm)
O
N
H
Cl

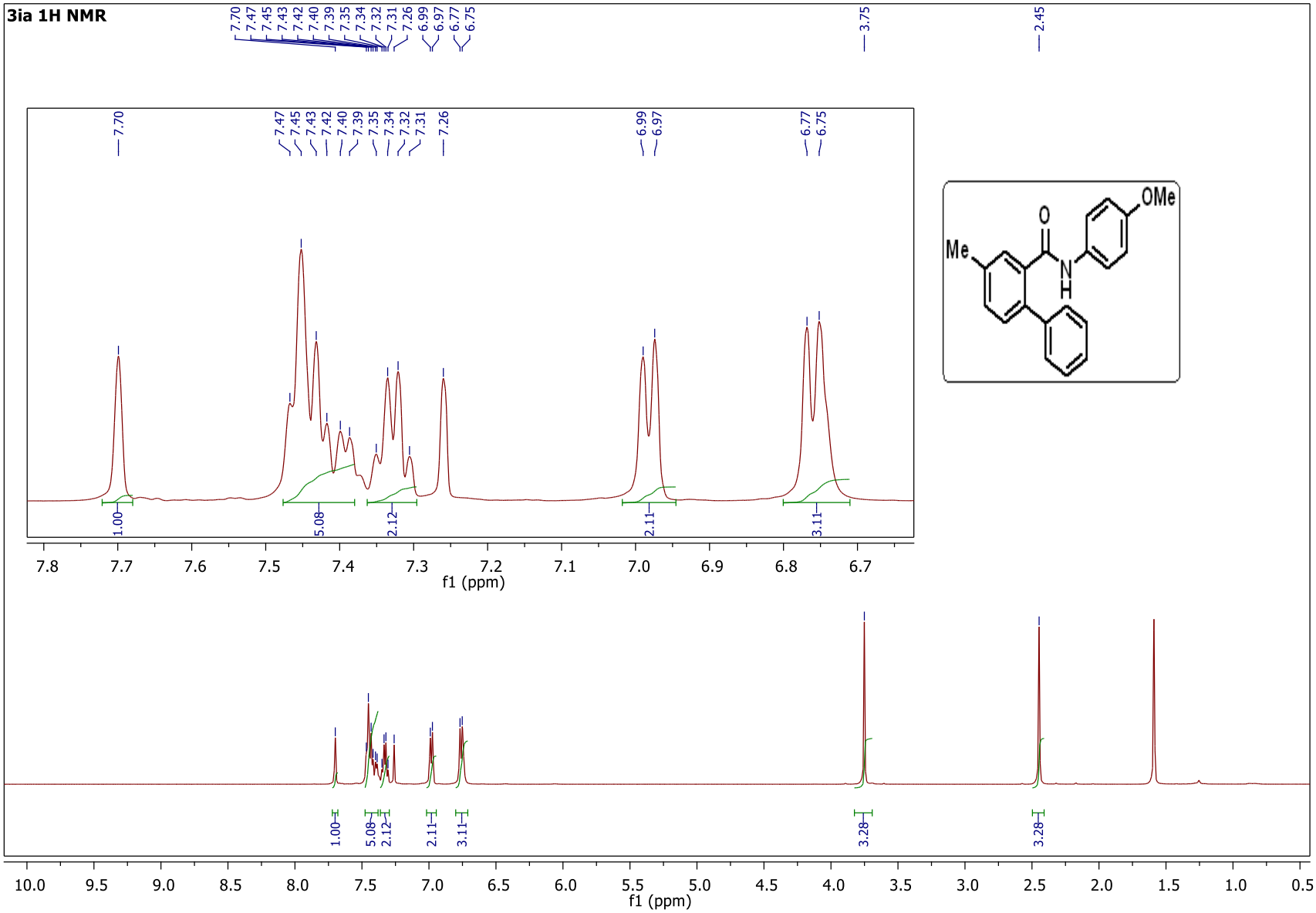

3ga 13C NMR
167.15
139.83
139.61
138.63
134.79
134.51
130.99
130.41
129.80
129.71
129.13
128.84
128.29
128.03
124.46
119.98
117.77
77.29
77.04
76.79
O
N
H
Cl
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

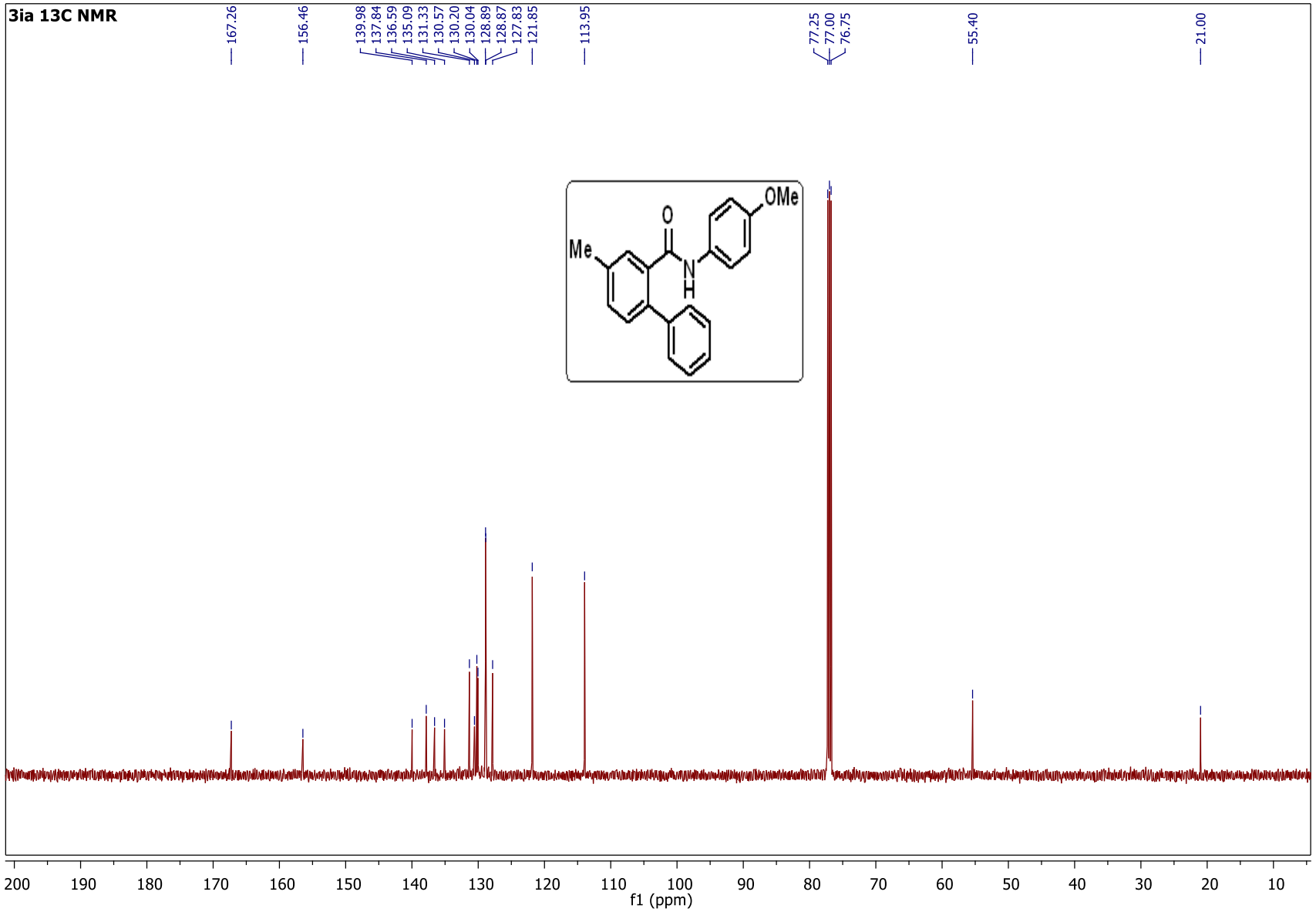

3ha 1H NMR
8.26
8.24
7.64
7.63
7.61
7.52
7.41
7.40
7.39
7.37
7.36
6.86
6.85
1.00
3.18
1.10
7.25
1.06
f1 (ppm)
1.04
1.00
3.18
1.10
7.25
1.06
f1 (ppm)


3ha 13C NMR
168.42
151.54
147.32
140.03
139.71
138.30
135.66
130.47
128.61
128.56
127.78
127.44
119.52
113.96
77.25
77.00
76.75
O
N
H
N
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

Data File	MH-578-PYRIDYL-PHENYL.d	**Sample Name**	HARI
Sample Type	Sample	**Position**	
Instrument Name	Instrument 1	**User Name**	
Acq Method	Sample_pos_01.m	**Acquired Time**	06-12-2019 16:12:42 (UTC+05:30)
IRM Calibration Status	Success	**DA Method**	HARI.m
Comment			
Sample Group		**Info.**	
Stream Name		**Acquisition Time (Local)**	06-12-2019 16:12:42 (UTC+05:30)
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.08.00 (B8058.0)	**TOF Driver Version**	8.00.00
TOF Firmware Version	17.698	**Tune Mass Range Max.**	3200

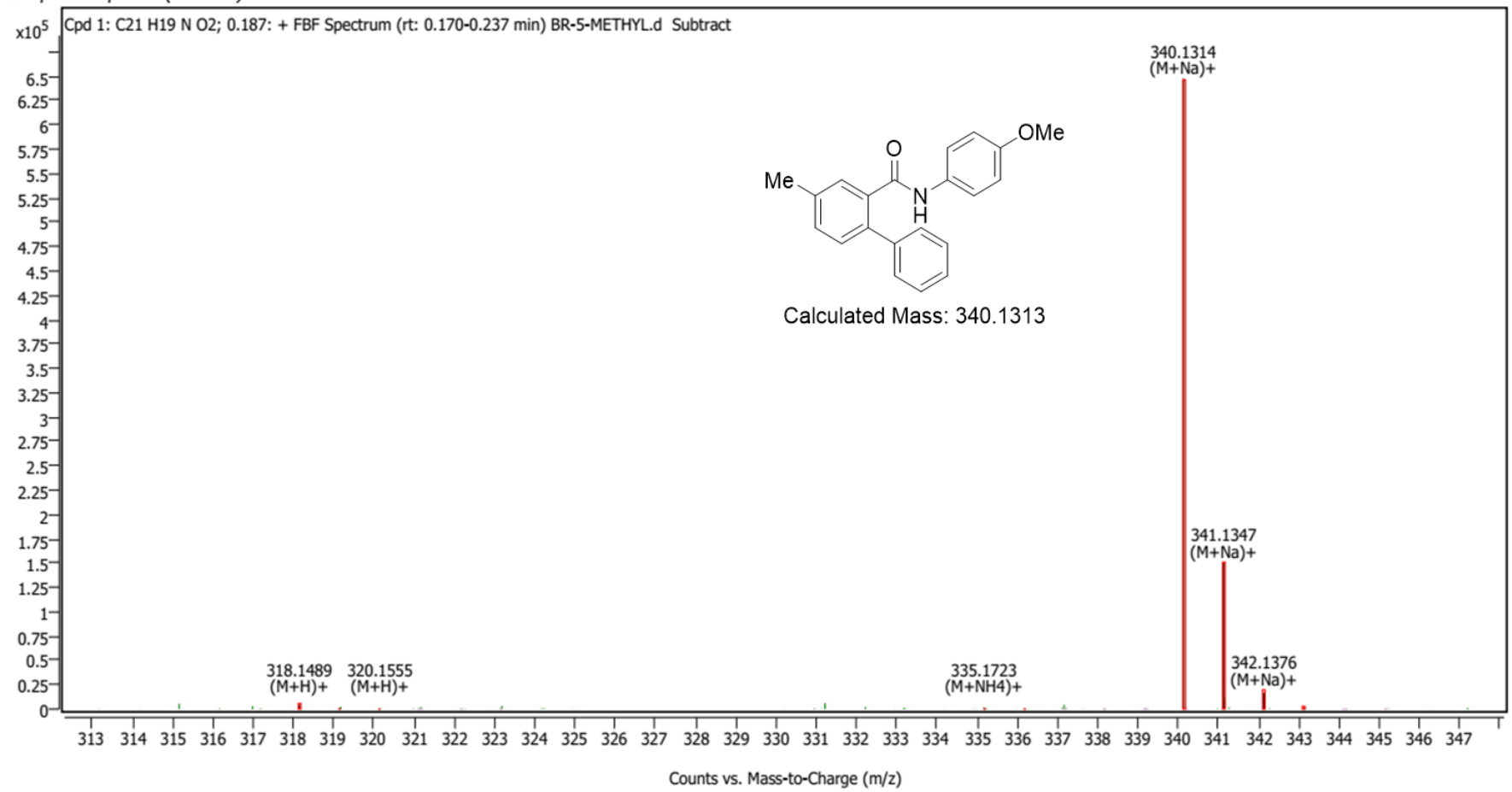


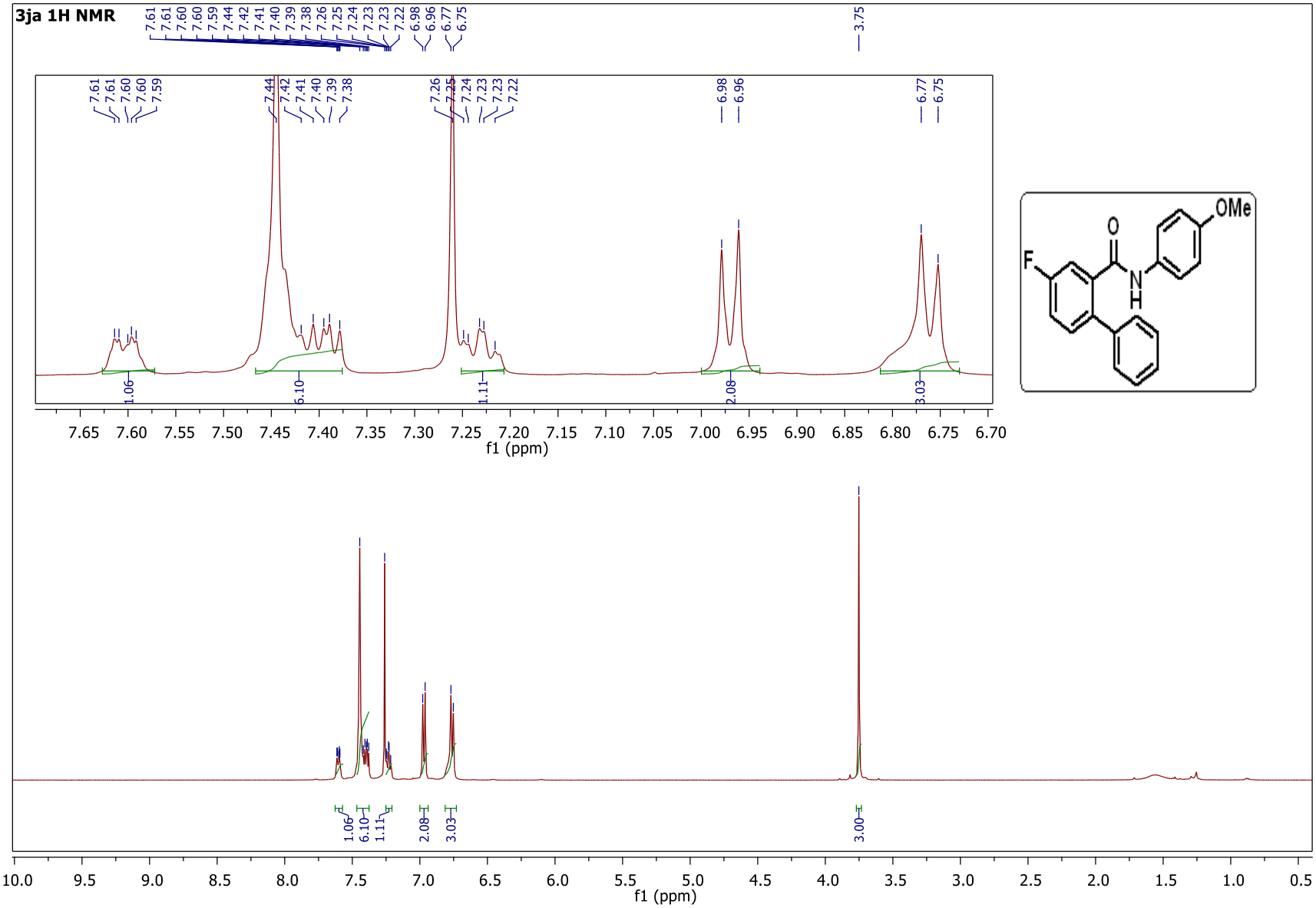

3ia 1H NMR
7.70
7.47
7.45
7.43
7.42
7.40
7.39
7.35
7.34
7.32
7.31
7.26
6.99
6.97
6.77
6.75
3.75
2.45
1.00
5.08
2.12
2.11
3.11
3.28
3.28
f1 (ppm)
f1 (ppm)
OMe
Me
O
N
H


3ia 13C NMR
167.26
156.46
139.98
137.84
136.59
135.09
131.33
130.57
130.20
130.04
128.89
128.87
127.83
121.85
113.95
77.25
77.00
76.75
55.40
21.00
OMe
O
Me
N
H
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

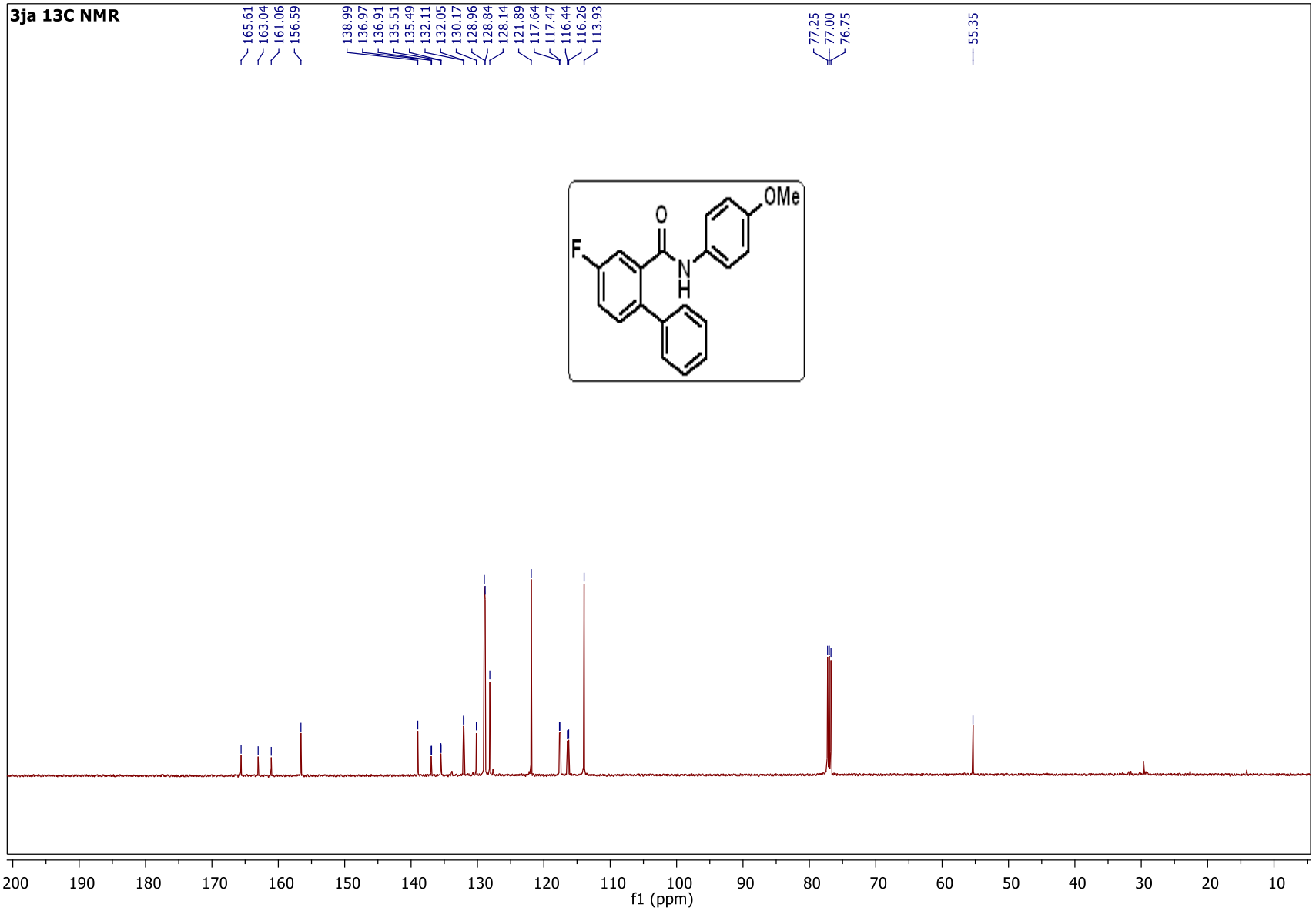
Cpd. 1: C21 H19 N O2

Compound Spectra (overlaid)



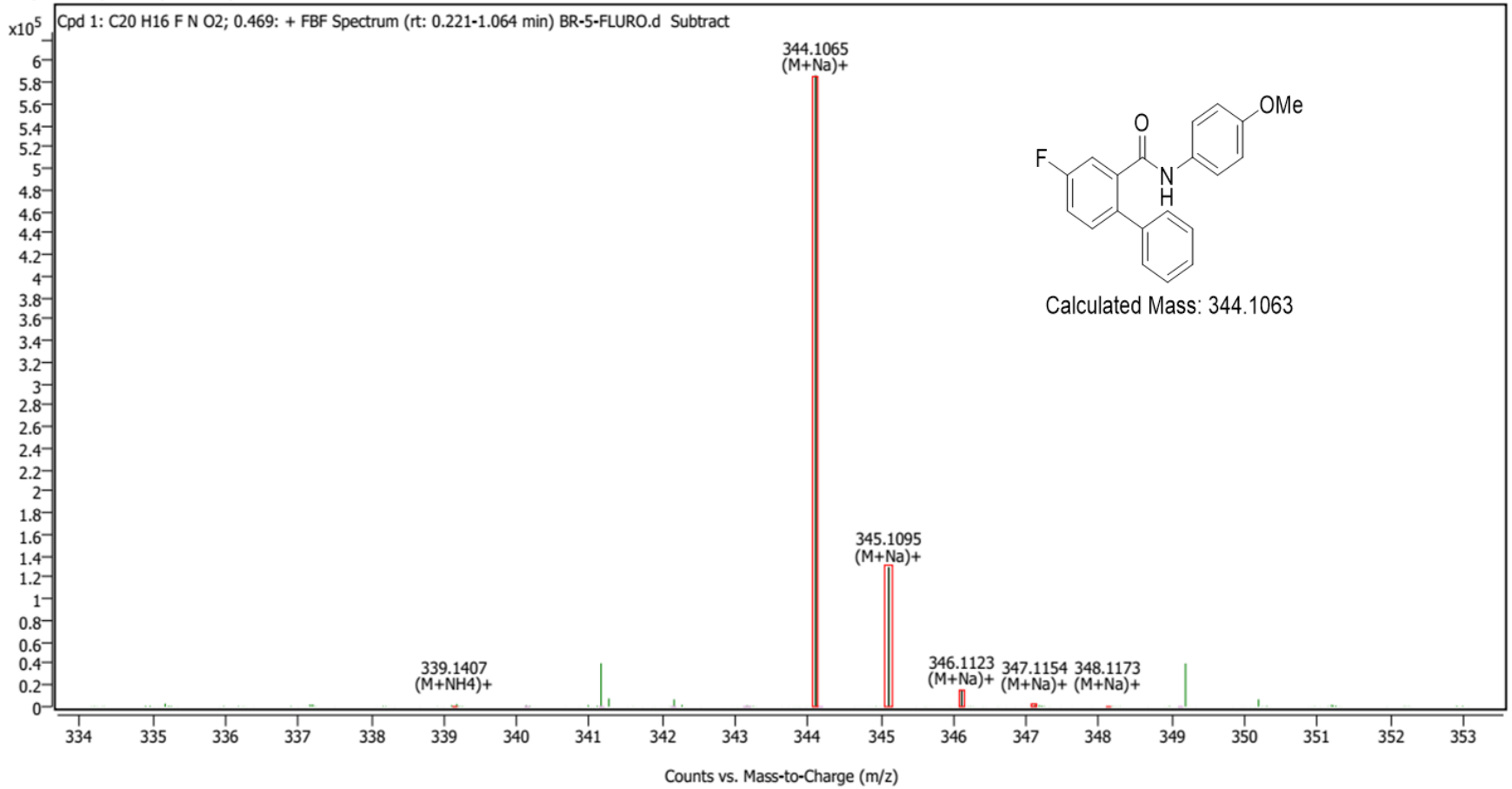

3ja 1H NMR
7.61 7.61 7.60 7.60 7.59 7.44 7.42 7.41 7.40 7.39 7.38 7.26 7.25 7.24 7.23 7.22 6.98 6.96 6.77 6.75 3.75
1.06 6.10 1.11 2.08 3.03
7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80 6.75 6.70
f1 (ppm)
1.06 6.10 1.11 2.08 3.03 3.00
10.0 9.5 9.0 8.5 8.0 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 0.5
f1 (ppm)
F
O
N
H
OMe

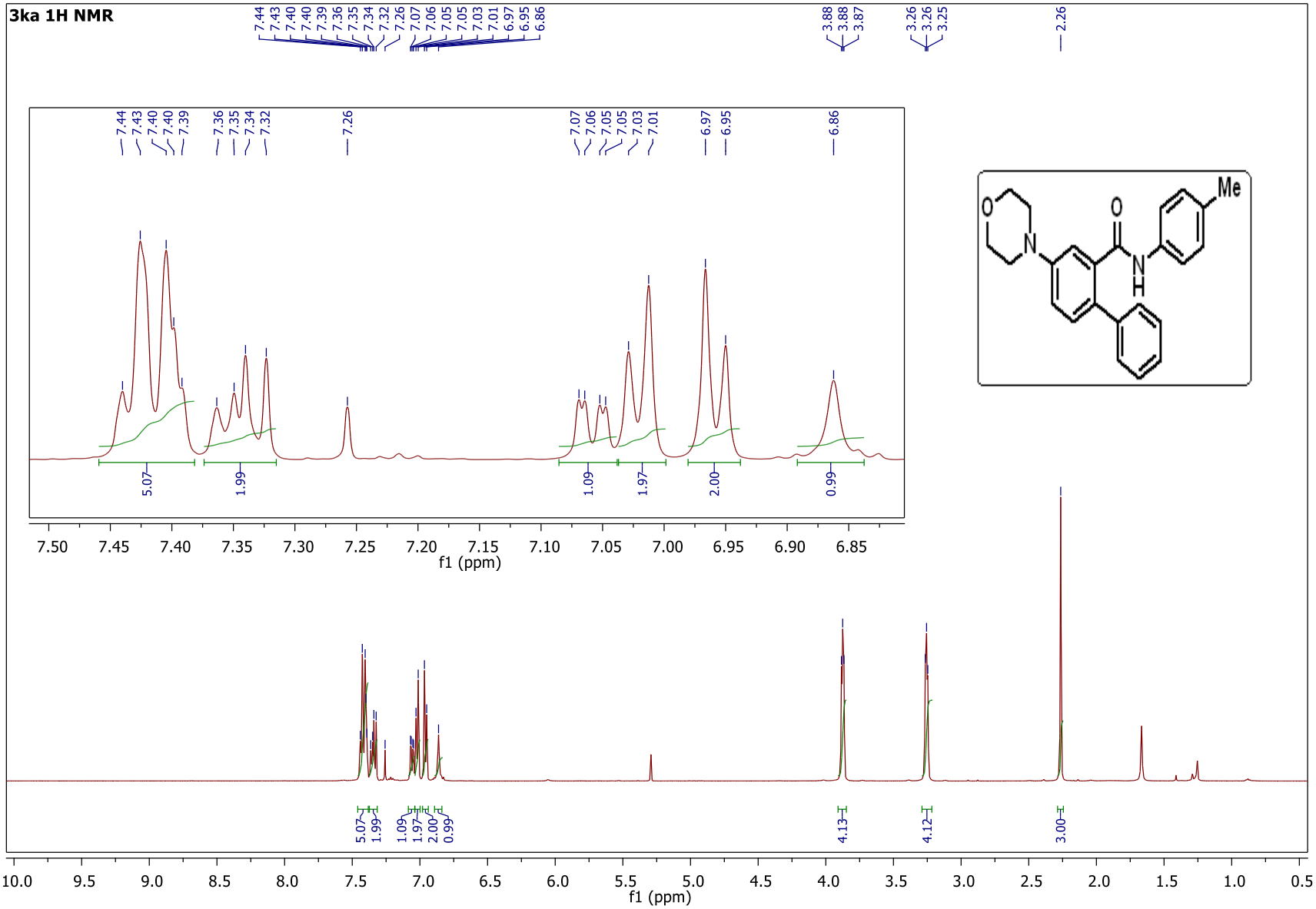
3ja 13C NMR

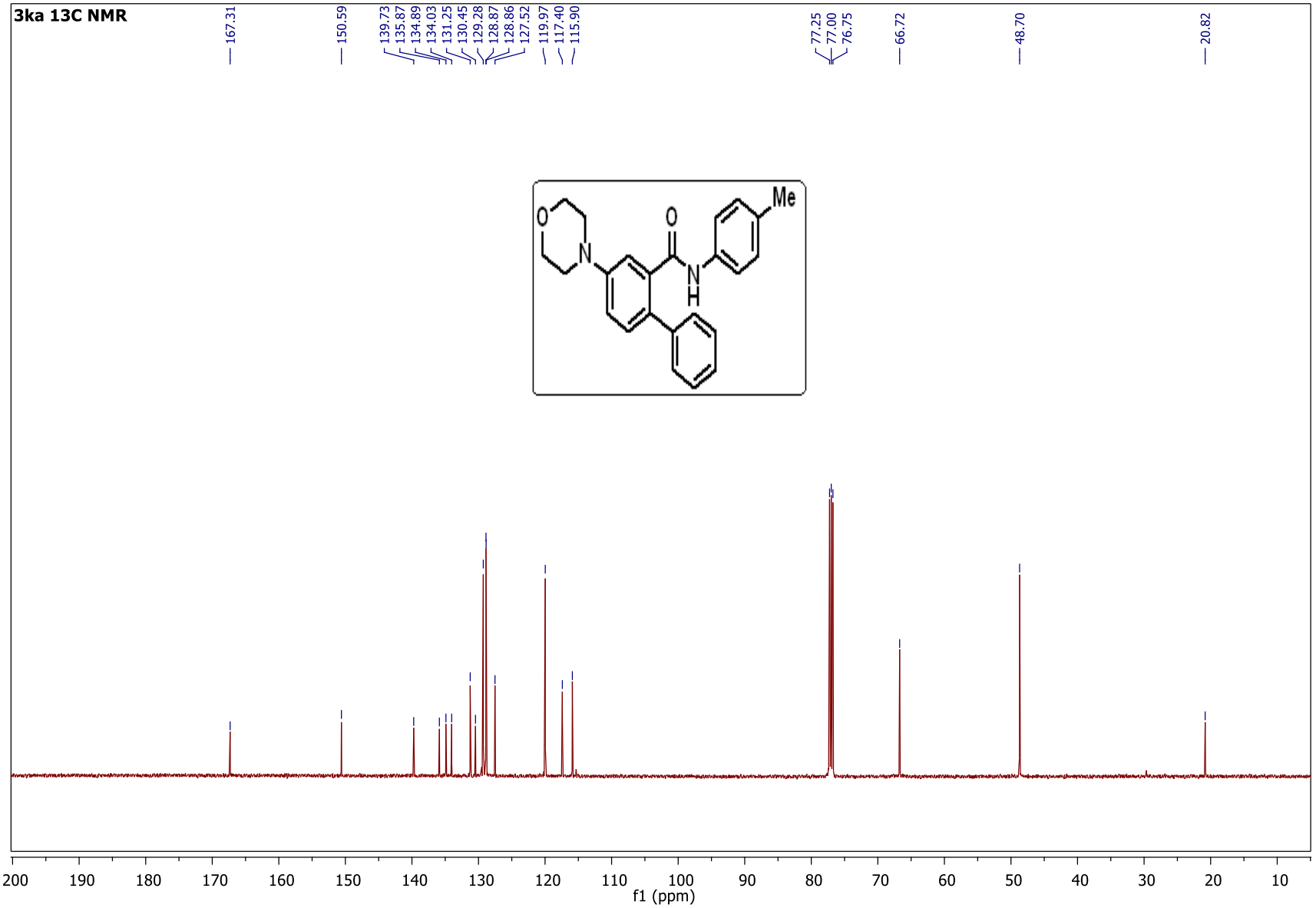

165.61
163.04
161.06
156.59
138.99
136.97
136.91
135.51
135.49
132.11
132.05
130.17
128.96
128.84
128.14
121.89
117.64
117.47
116.44
116.26
113.93
77.25
77.00
76.75
55.35
OMe
O
F
N
H
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

Cpd. 1: C20 H16 F N O2

Compound Spectra (overlaid)

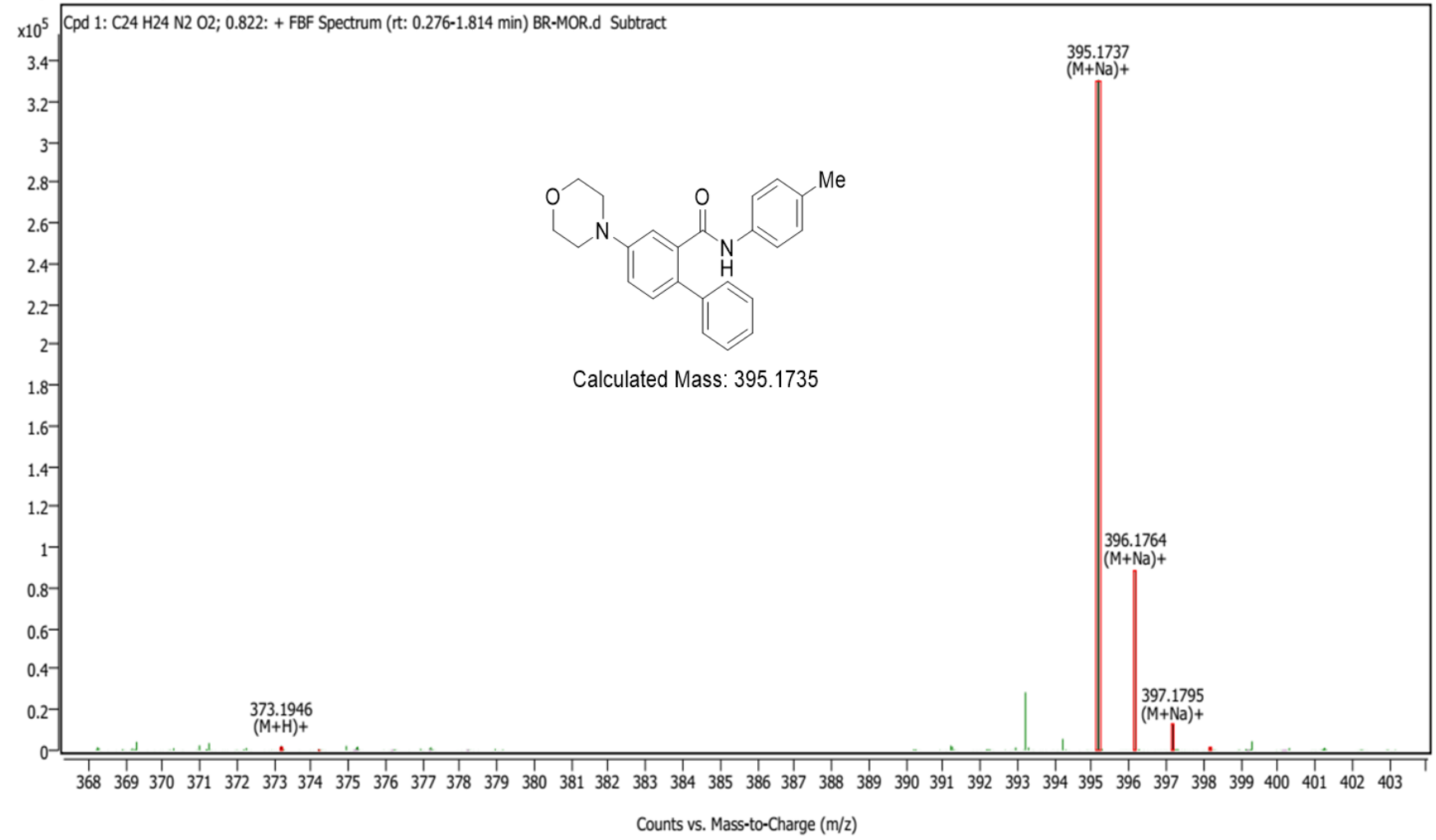


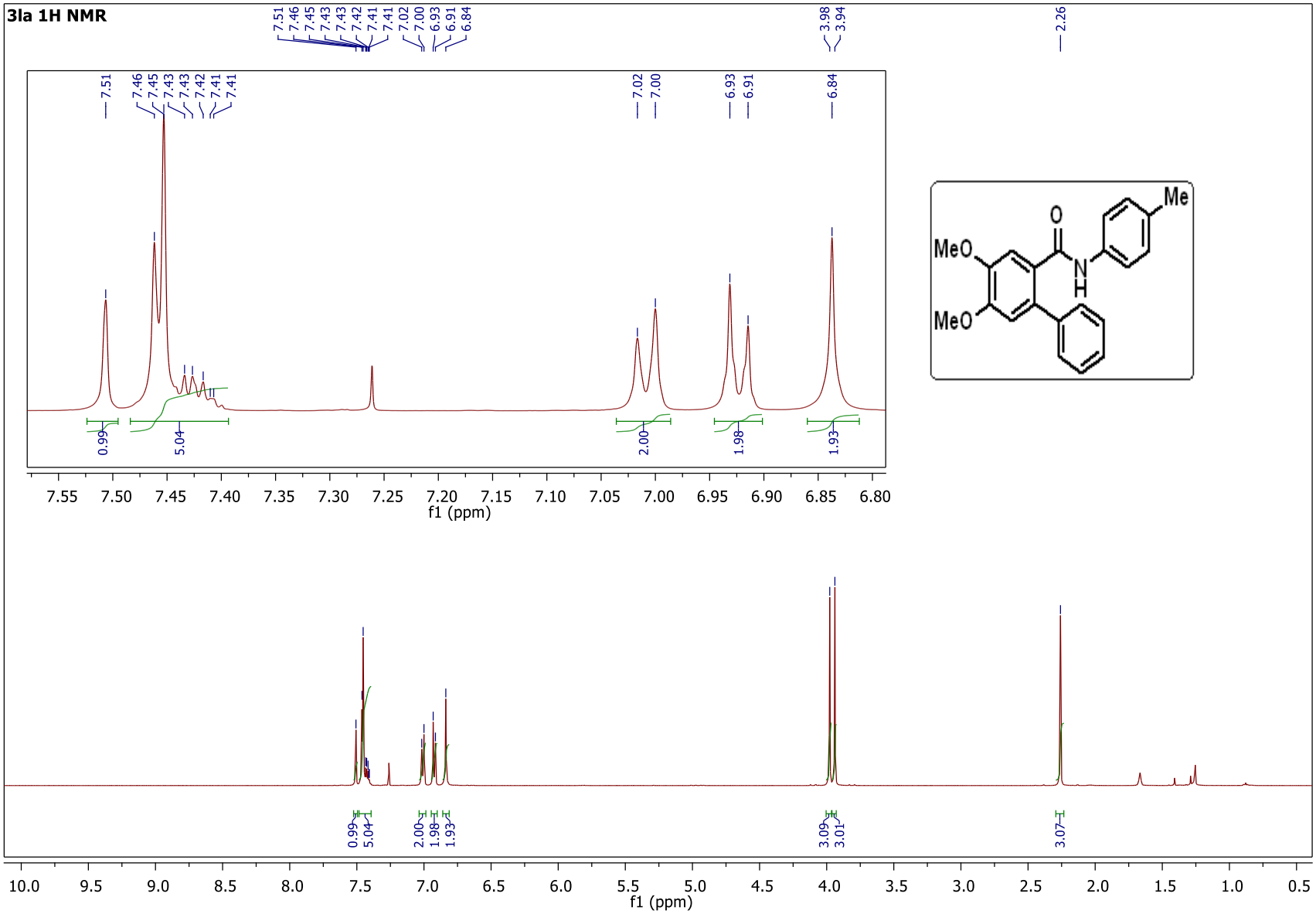

3ka 1H NMR
7.44 7.43 7.40 7.40 7.39 7.36 7.35 7.34 7.32 7.26 7.07 7.06 7.05 7.05 7.03 7.01 6.97 6.95 6.86
3.88 3.88 3.87 3.26 3.26 3.25 2.26
5.07 1.99 1.09 1.97 2.00 0.99
f1 (ppm)
4.13 4.12 3.00
Me
O
N
O
N
H


3ka 13C NMR
167.31
150.59
139.73
135.87
134.89
134.03
131.25
130.45
129.28
128.87
128.86
127.52
119.97
117.40
115.90
77.25
77.00
76.75
66.72
48.70
20.82
O
N
O
N
H
Me
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

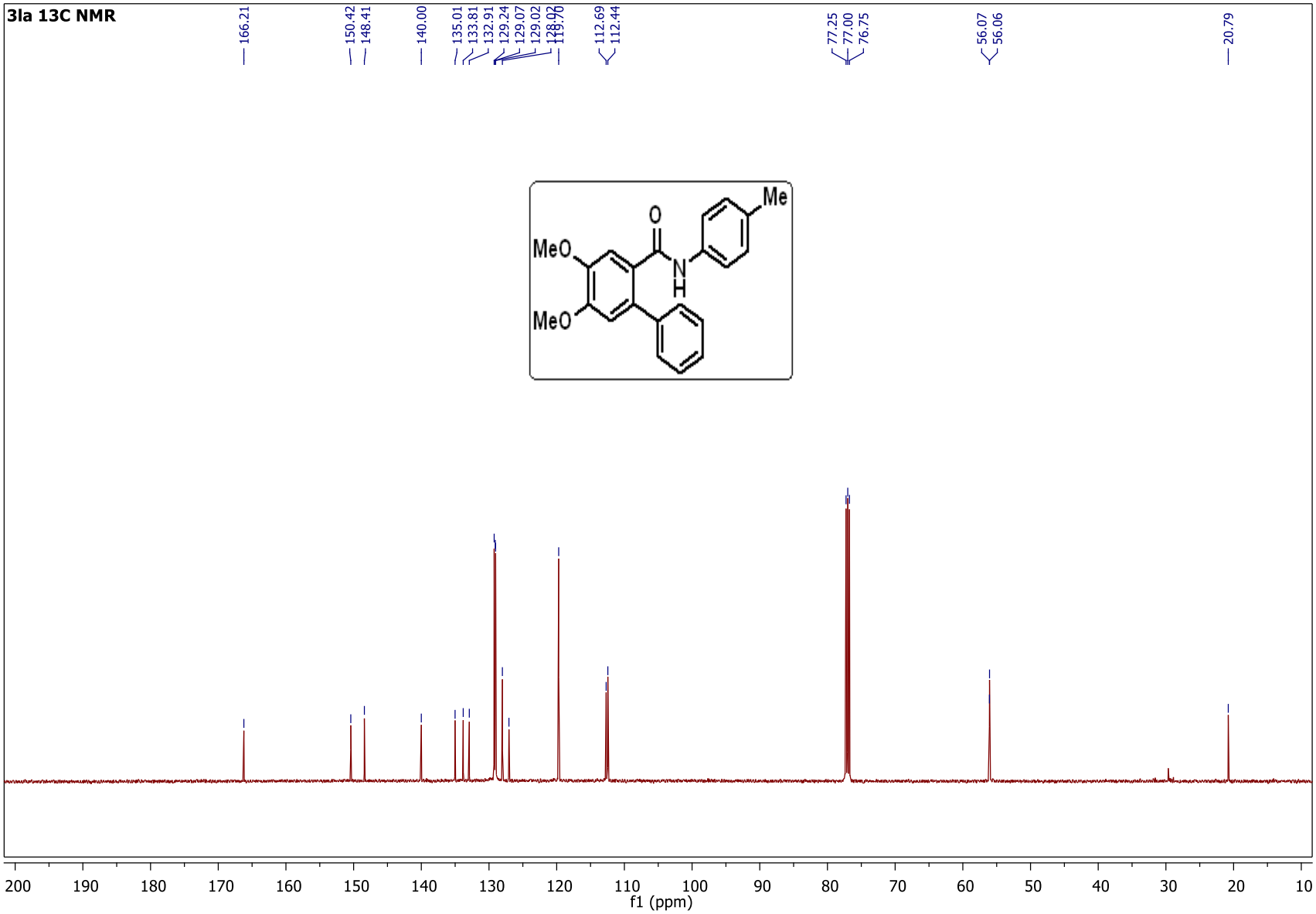
Cpd. 1: C24 H24 N2 O2

Compound Spectra (overlaid)



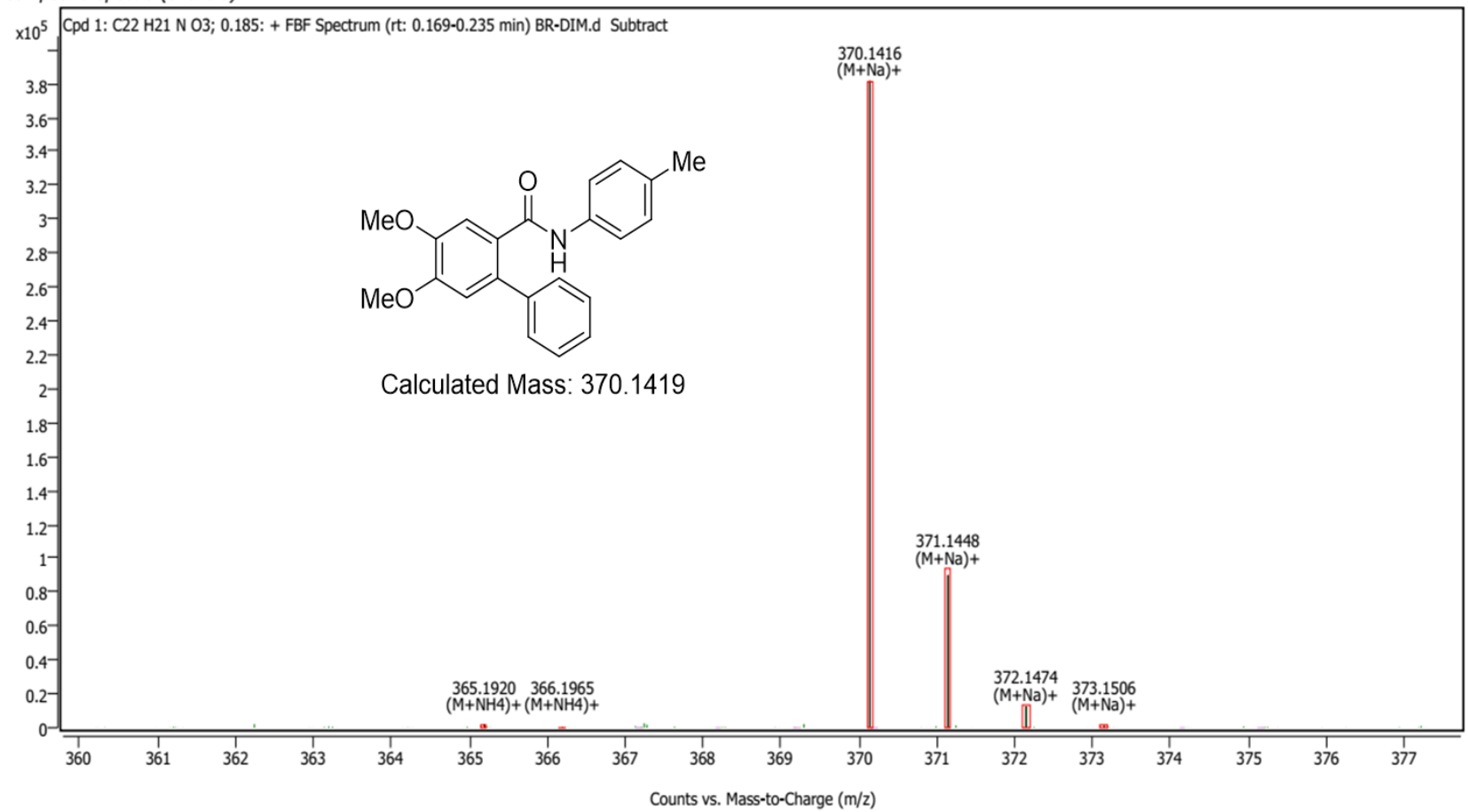

3la 1H NMR
7.51
7.46
7.45
7.43
7.43
7.42
7.41
7.41
7.02
7.00
6.93
6.91
6.84
3.98
3.94
2.26
0.99
5.04
2.00
1.98
1.93
f1 (ppm)
MeO
MeO
O
N
H
Me
3.09
3.01
3.07

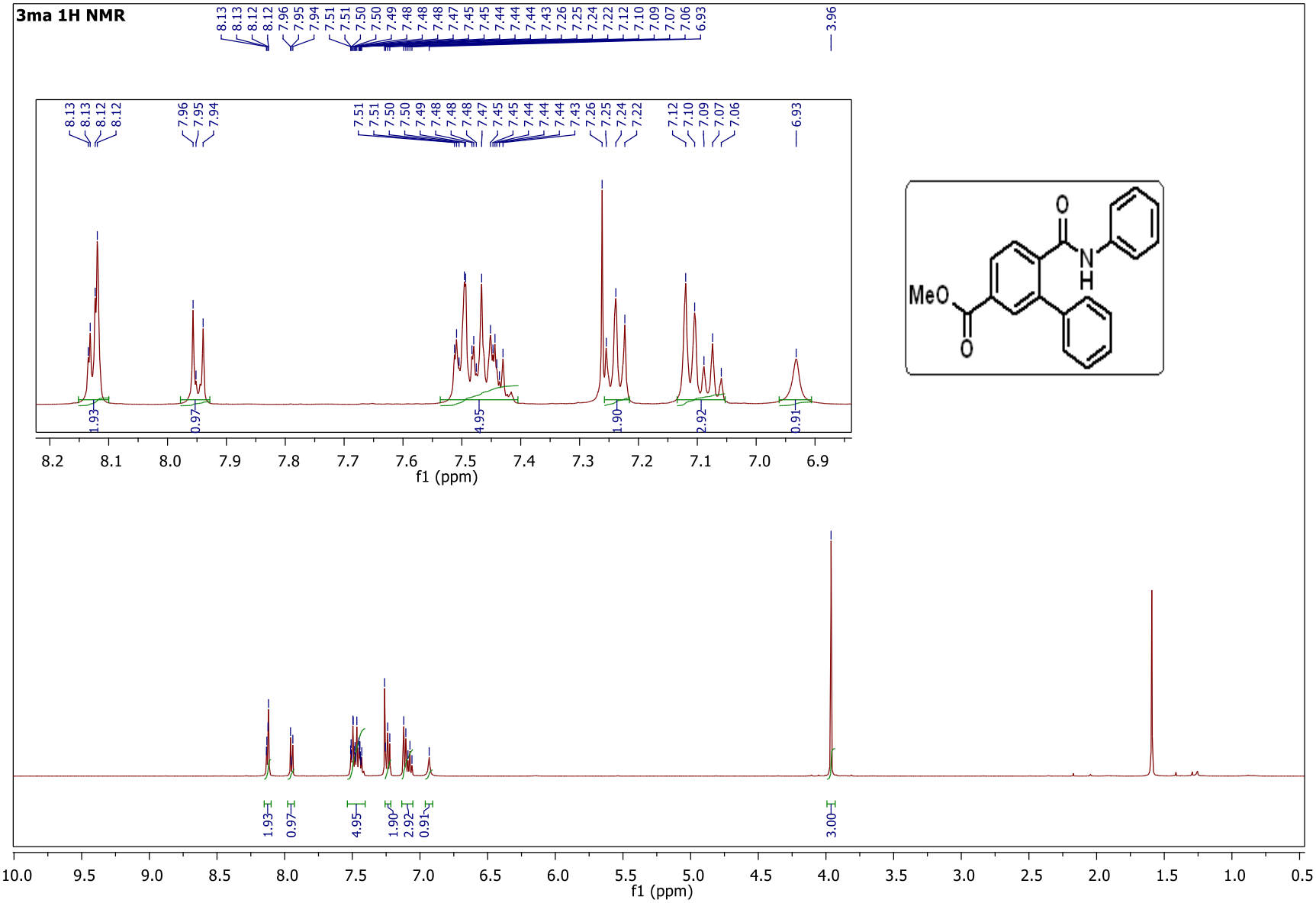
3la 13C NMR

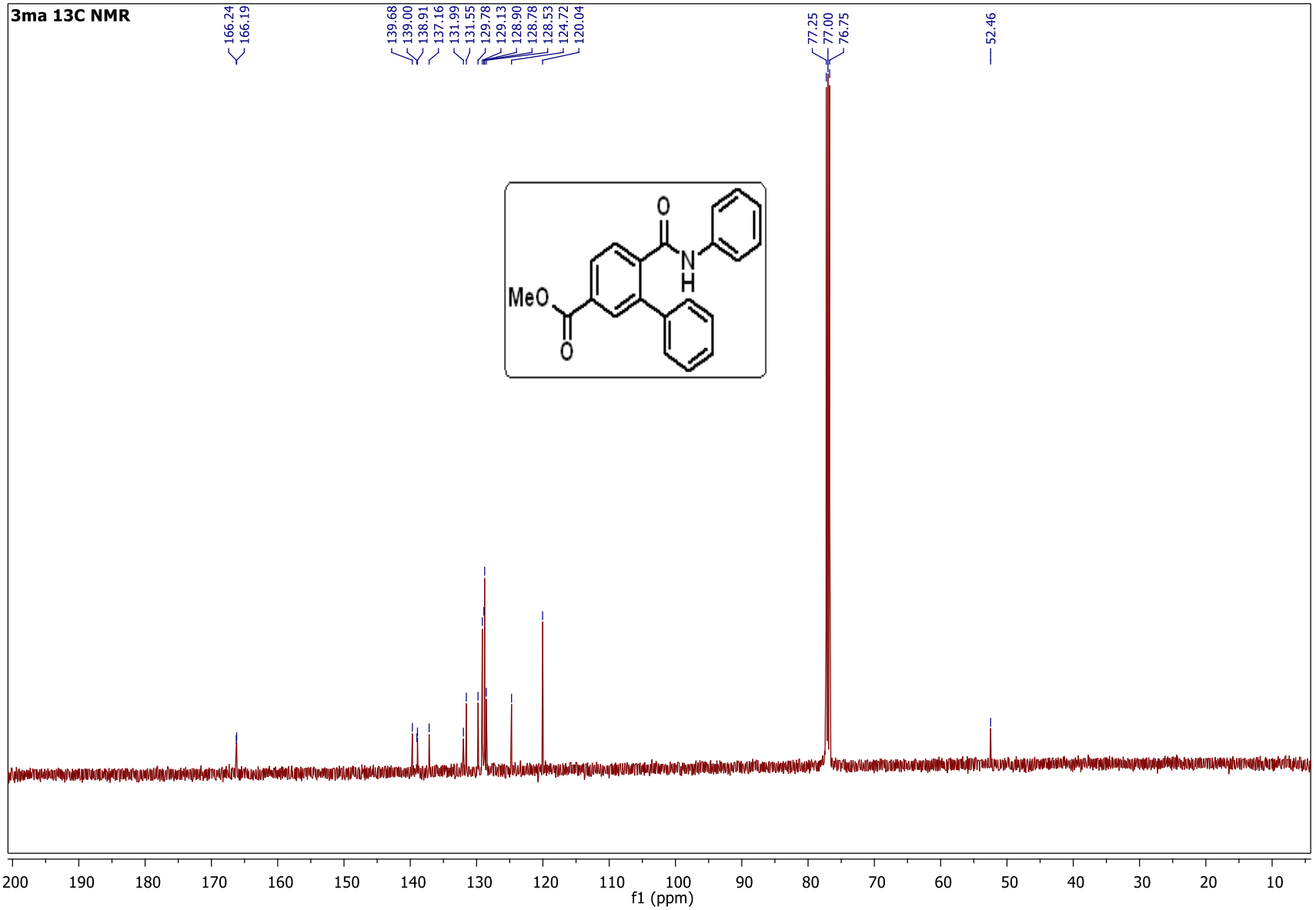

166.21
150.42
148.41
140.00
135.01
133.81
132.91
129.24
129.07
129.02
119.70
112.69
112.44
77.25
77.00
76.75
56.07
56.06
20.79
MeO
MeO
O
N
H
Me
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

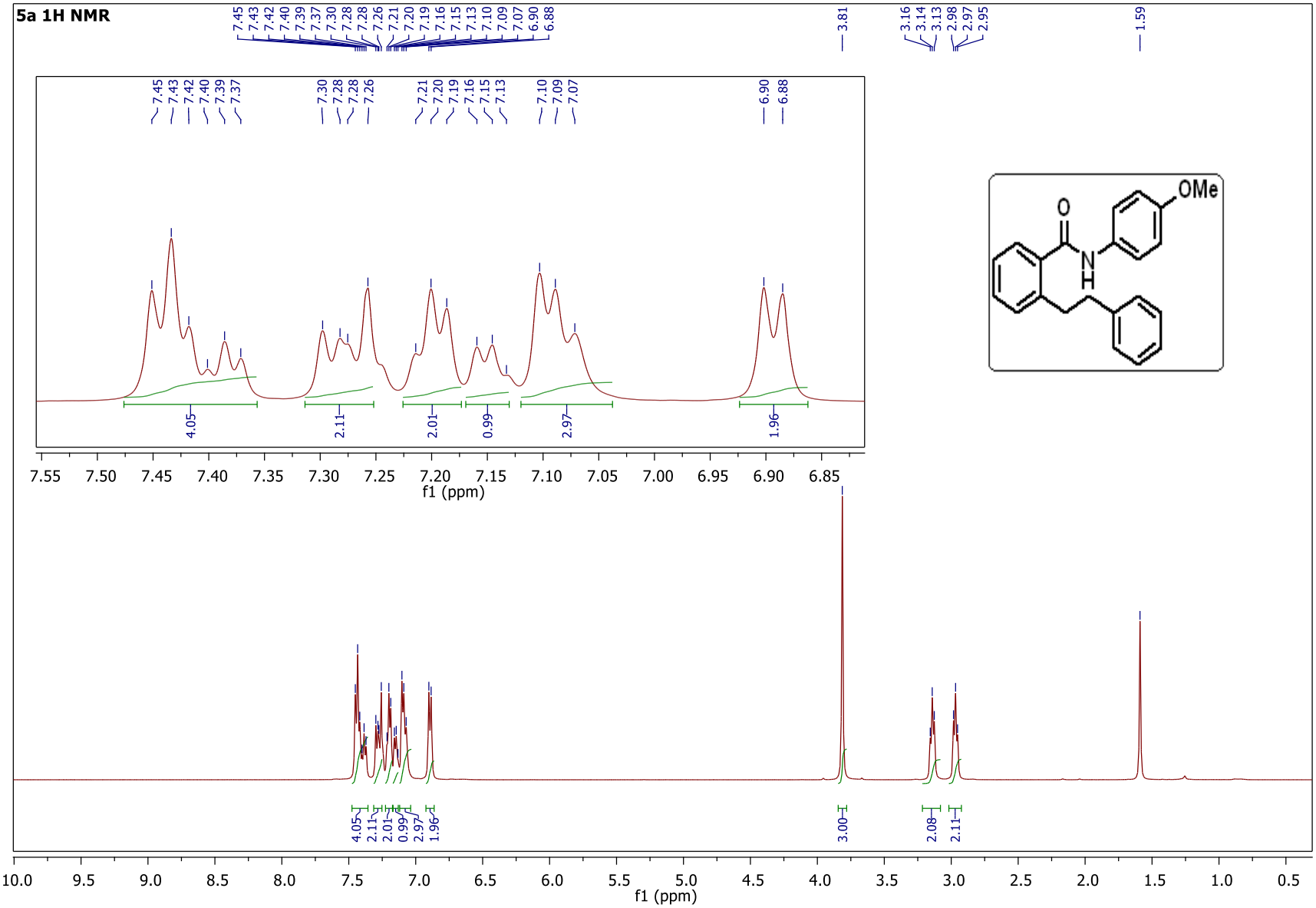
Cpd. 1: C22 H21 N O3

Compound Spectra (overlaid)

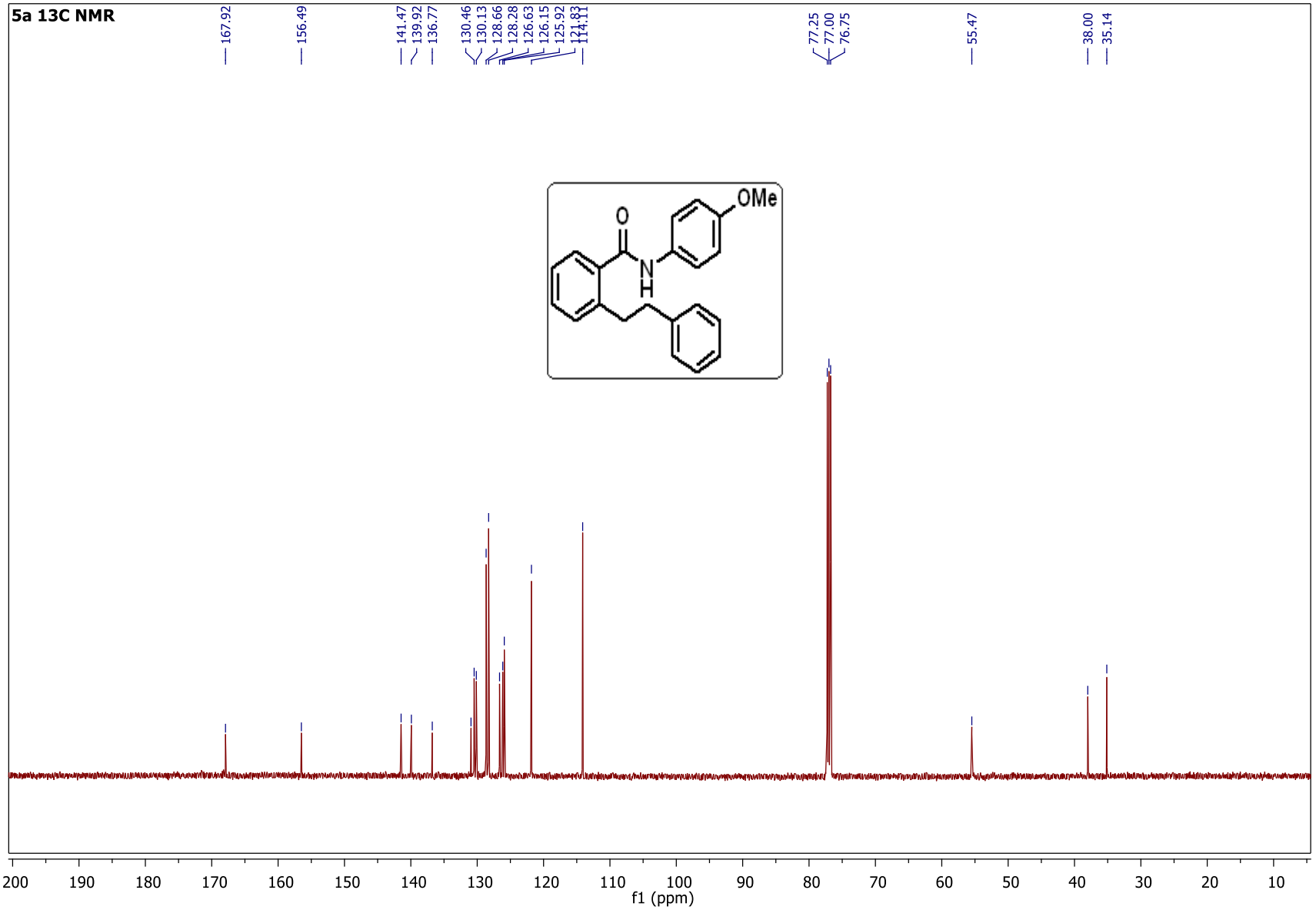



3ma 1H NMR
8.13 8.13 8.12 8.12 7.96 7.95 7.94 7.51 7.51 7.50 7.50 7.49 7.48 7.48 7.48 7.47 7.45 7.44 7.44 7.44 7.43 7.26 7.25 7.24 7.22 7.12 7.10 7.09 7.07 7.06 6.93 3.96
1.93 0.97 4.95 1.90 2.92 0.91
8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9
f1 (ppm)
MeO
O
N
H
O
1.93 0.97 4.95 1.90 2.92 0.91 3.00
10.0 9.5 9.0 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 0.5
f1 (ppm)

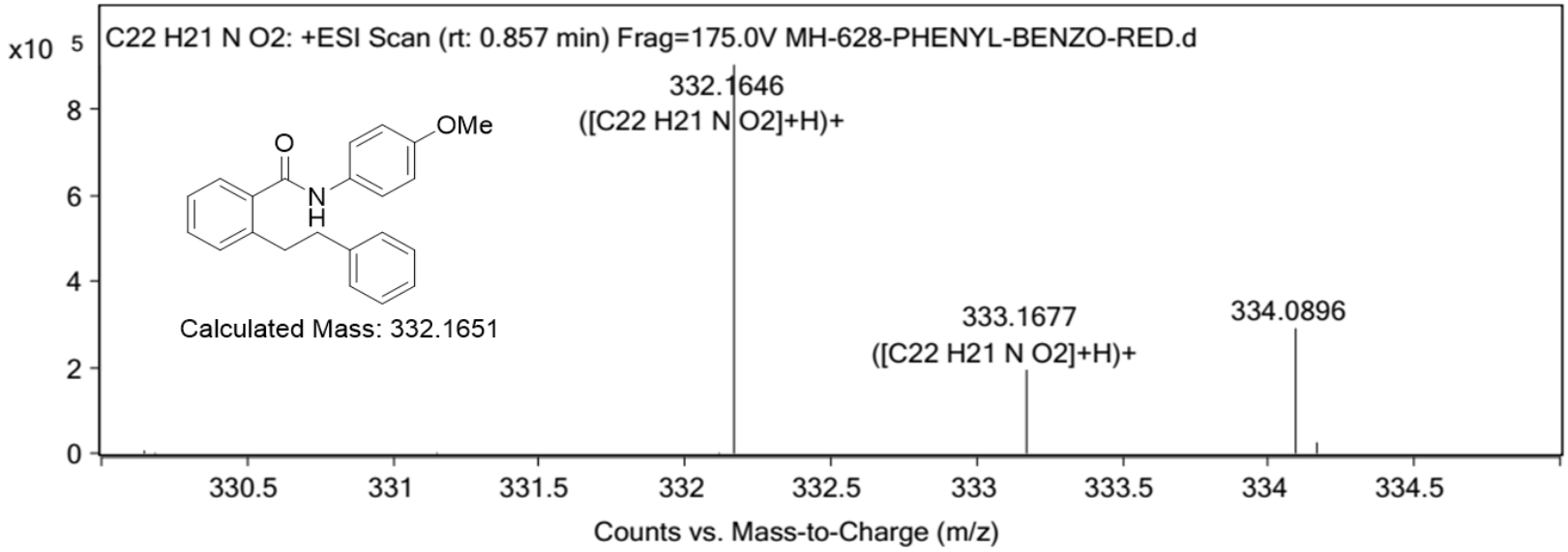

3ma 13C NMR
166.24
166.19
139.68
139.00
138.91
137.16
131.99
131.55
129.78
129.13
128.90
128.78
128.53
124.72
120.04
77.25
77.00
76.75
52.46
O
N
H
MeO
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

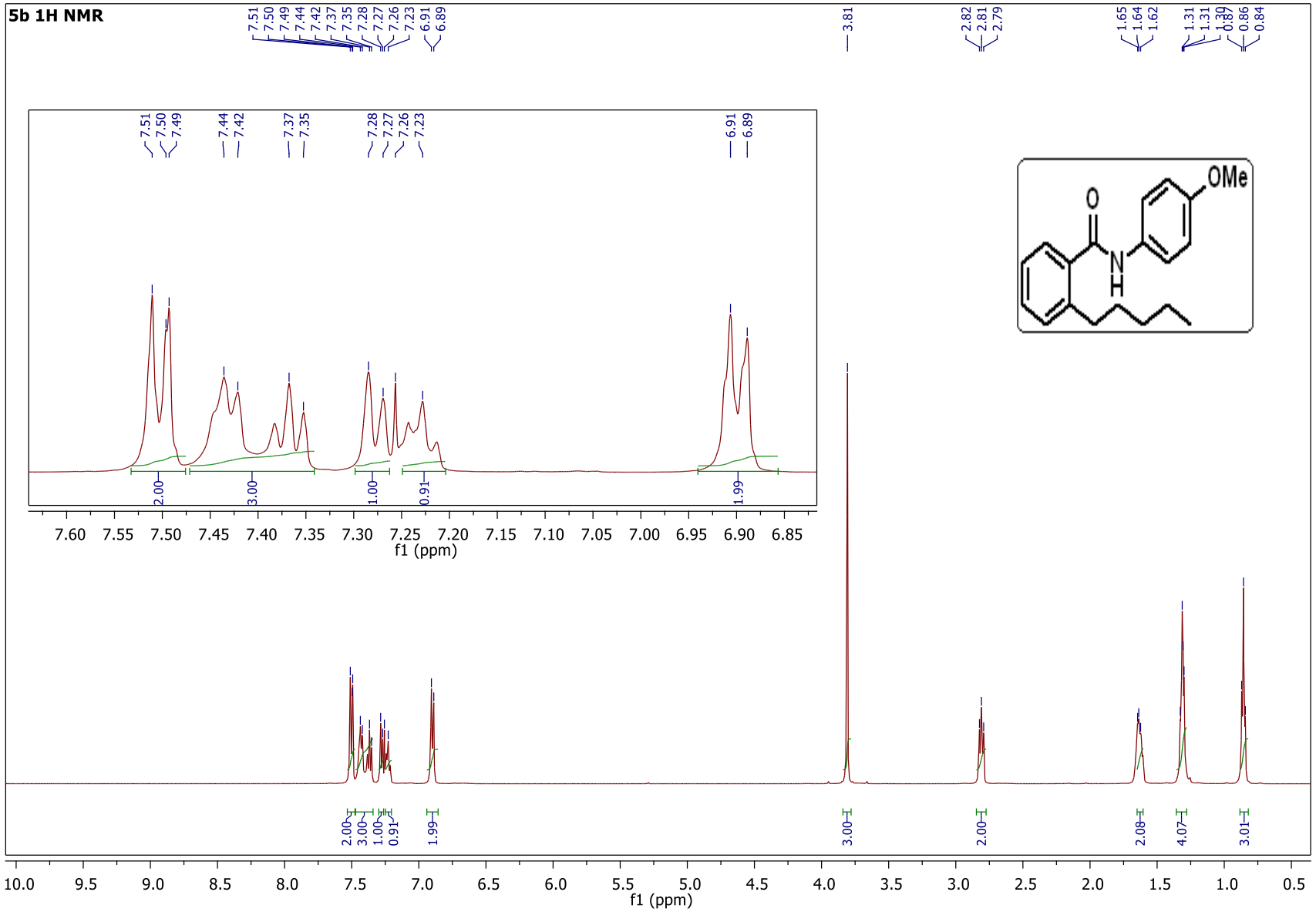

5a 1H NMR
7.45 7.43 7.42 7.40 7.39 7.37 7.30 7.28 7.28 7.26 7.21 7.20 7.19 7.16 7.15 7.13 7.10 7.09 7.07 6.90 6.88
3.81
3.16 3.14 3.13 2.98 2.97 2.95
1.59
4.05 2.11 2.01 0.99 2.97 1.96
7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85
f1 (ppm)
3.00 2.08 2.11
10.0 9.5 9.0 8.5 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 0.5
f1 (ppm)
O
N
H
OMe

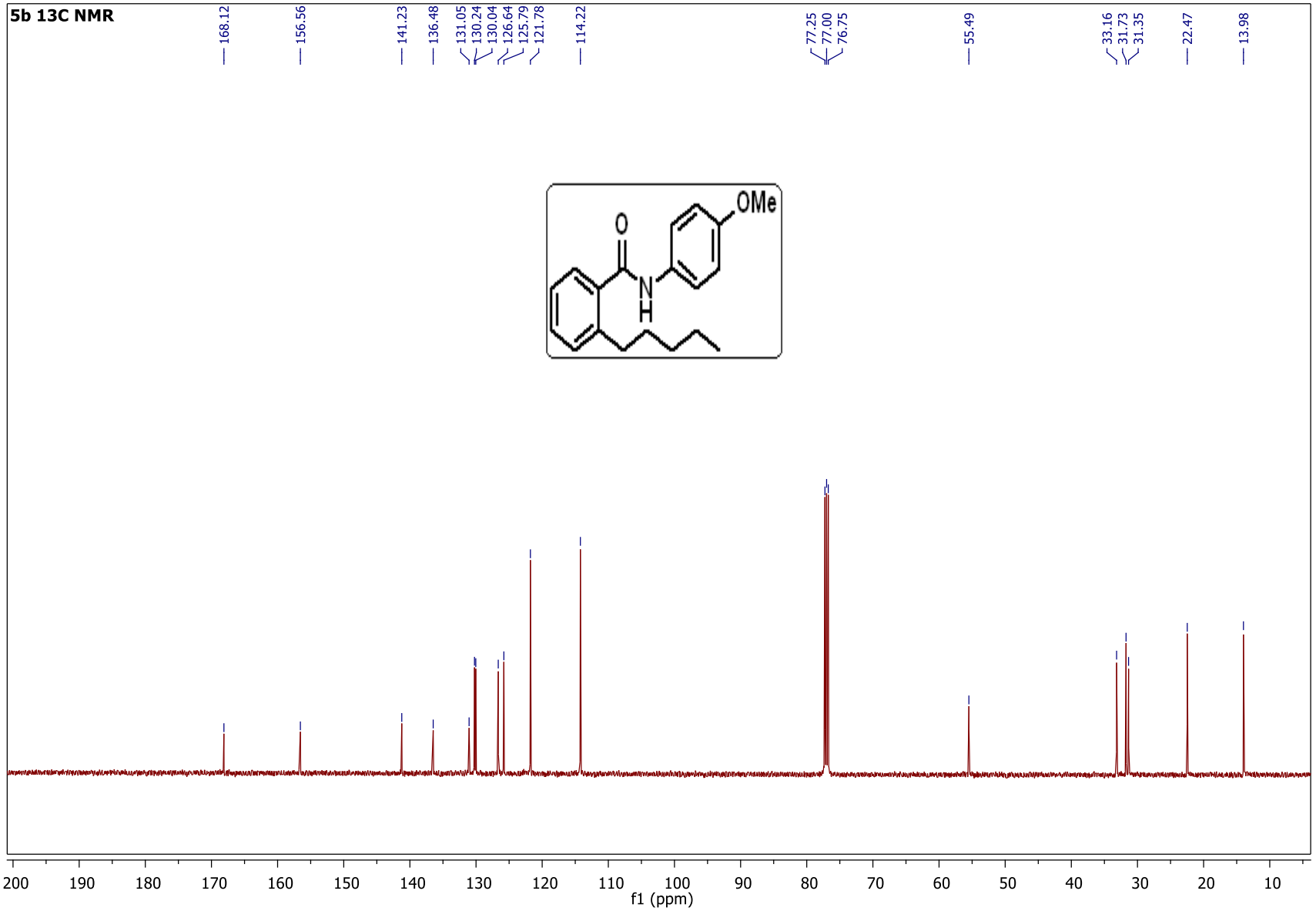
5a 13C NMR


167.92
156.49
141.47
139.92
136.77
130.46
130.13
128.66
128.28
126.63
126.15
125.92
121.83
114.11
77.25
77.00
76.75
55.47
38.00
35.14
O
N
H
OMe
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

Data File	MH-628-PHENYL-BENZO-RED.d	**Sample Name**	hari
Sample Type	Sample	**Position**	
Instrument Name	Instrument 1	**User Name**	
Acq Method	default.m	**Acquired Time**	31-05-2019 14:31:26 (UTC+05:30)
IRM Calibration Status	Success	**DA Method**	HARI.m
Comment			
Sample Group		**Info.**	
Stream Name		**Acquisition Time (Local)**	31-05-2019 14:31:26 (UTC+05:30)
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.08.00 (B8058.0)	**TOF Driver Version**	8.00.00
TOF Firmware Version	17.698	**Tune Mass Range Max.**	3200




5b 1H NMR
7.51
7.50
7.49
7.44
7.42
7.37
7.35
7.28
7.27
7.26
7.23
6.91
6.89
3.81
2.82
2.81
2.79
1.65
1.64
1.62
1.31
1.31
0.87
0.86
0.84
OMe
O
N
H
2.00
3.00
1.00
0.91
1.99
3.00
2.00
2.08
4.07
3.01
f1 (ppm)


5b 13C NMR
168.12
156.56
141.23
136.48
131.05
130.24
130.04
126.64
125.79
121.78
114.22
77.25
77.00
76.75
55.49
33.16
31.73
31.35
22.47
13.98
O
N
H
OMe
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

Cpd. 1: C19 H23 N O2

Compound Spectra (overlaid)