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Supporting Information

Lipophilic NHC Assisted One-pot Synthesis of Syncarpamide Analogues in Aqueous Medium

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Supporting information

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Table of Context

Materials and methods:

1. Materials:

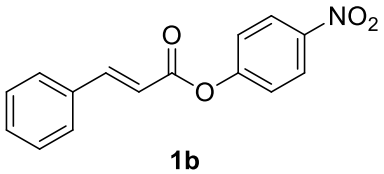
All reactions were carried out in oven-dried glassware under an argon atmosphere employing standard techniques in handling air-sensitive materials. Substituted ester was prepared from substituted acid, ethanolamine was purchased from SRL, chiral alcohols ((*S*)-(−)-2-Amino-3-phenyl-1-propanol (190438), (*S*)-2-Amino-1-phenylethanol (494577) and (*R*)-(−)-2-Phenylglycinol (A19030) were purchased from Aldrich and Alfa Aesar. Potassium carbonate was purchased from Merck. Flash chromatography was performed on silica gel 100-200 mesh (code 95178), 200-400 (code 96671) mesh size was purchased from SRL. Sodium chloride was purchased from SRL (Assay =99.5%). ^{1}H NMR was recorded Brucker model avance-II 300 MHz and ^{13}C 75 MHz spectrometer using TMS as an internal standard and $CDCl_3$, DMSO-d6 as a solvent. The JEOL GCMATE II GC-MS with Data system is a high resolution, Electron Spray Ionization (ESI) methods were used for analyzing mass of molecules. Melting points were recorded on an digital melting point apparatus and are uncorrected. Optical rotations were measured on a PerkinElmer Precisely/Model-341 polarimeter operating at the sodium D line with a 100 mm path cell. FT-IR spectrum was taken Perkin Elmer Spectrum RX I model.

Methods:

General Procedure

2. Synthesis of 4-nitrophenyl cinnamate

The synthesis of 4-nitrophenyl cinnamate procedure was performed by closely following the Literature procedure.[1a]



1b

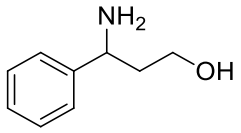
To the mixture of cinnamic acid (0.15 g, 1 mmol) and 4-nitrophenol (0.14 g, 1 mmol) in dichloromethane, DCC (0.248 g, 1.2 mmol) and DMAP (0.013 g, 0.1 mmol) was added. Then, the reaction was heated at 50 ^{o}C for 3 h. After complete consumption of starting material, the reaction mixture was cooled to room temperature and dissolved with DCM and filtered. The filtrate was concentrated under reduced pressure and obtained crude was purified by column chromatography on silica gel (100-200 mesh) in 10% Ethyl acetate in pet ether to afford 0.215 g of white solid, Yield- 80%. M.p: 141-142 ^{o}C, Lit (145-146 ^{o}C)[1b].

2a. Synthesis of 1,3-Didodecyl Benzimidazolium Bromide (NHC-D)

The synthesis of 1,3-Didodecyl Benzimidazolium Bromide was performed by following the Literature procedure.[2] ^{1}H NMR ($CDCl_3$): δ = 11.41 (s, 1H) , 7.74-7.65 (m, 4H), 4.64 (t, *J* =7.5 Hz, 1H), 2.10- 2.00 (m, 4H), 1.42- 1.24 (m, 36H), 0.87 (t, *J* = 6.9 Hz, 6H). Spectra data compared with Literature.[2]

3. Synthesis of 3-Amino-3-phenylpropan-1-ol

The preparation was carried out with slightly modified literature procedure.[3]



Step 1: NH_4OAc was added in a solution of malonic acid (2.0 mmol) and Benzaldehyde (2.0 mmol) in EtOH (10 ml). The mixture was reflux for overnight. The reaction mixture was cooled to room temperature. The resulting precipitate was collected by filtration and washed with ice-cold EtOH. The white solid was dried in vacuo, to give 3-amino-3-phenylpropionic acid. The afford product was taken to next step without further purification.

Step 2: The acid (2 mmol) was dissolved in THF (20 mL) and cooled to 0 °C. To this mixture was added protionwise $LiAlH_4$ (6 mmol) and the mixture was stirred for 15 mins at same temperature. The reaction mixture was refluxed for 2 h. After Complete consumption of starting material by TLC analysis. The reaction mixture was cooled to 0 ^oC and slowly quenched with water. The grey mixture was extracted with ethyl acetate (3 × 20 mL) and the combined organic layer was dried over Na_2SO_4 and concentrated under reduced pressure to afford crude product. The crude product was washed with n-hexane and triturate to afford white solid. Yield: 66%. Mp: 71-73 ^oC, (Lit: 76-77 ^oC)[3]. ^1H NMR ($CDCl_3$): δ = 7.39-7.23 (m, 5H), 4.15-4.11 (m, 1H), 3.86-3.78 (m, 2H), 2.62 (br s, 3H), 1.94-1.85 (m, 2H).

4. Synthesis of symmetrically substituted analogues of syncarpamide derivatives using NHC organocatalysis:

A 10 mL reaction vial was charged with amino alcohol derivatives (0.2 mmol), Substituted esters (0.2 mmol) and water (2 mL). To this mixture, potassium carbonate (0.042 g, 0.3 mmol) and NHC-D (0.09 g, 0.02 mmol) were added; the reaction vial was closed with rubber septa and purged with argon. After purging for 5 minutes, the reaction vial was tightly sealed with crimper cap and allowed to stir for 12 h at room temperature. (For **3h-n**, the reaction mixture needs to be heated to 60 ^{o}C for 5 h). After completion, the reaction mixture was extracted with ethyl acetate (3×10 mL). The combined organic layer was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The afforded crude mixture was purified by flash chromatography on silica gel (200-400 mesh) in 50-100% ethyl acetate in hexanes to afford the product.

2-[(2E)-3-phenylprop-2-enamido]ethyl (2E)-3-phenylprop-2-enoate (3a). White solid, Yield: 26.4 mg (82%), Mp: 93-95 °C (Lit 94–97 °C)[4], ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.74 (d, *J* = 15.9 Hz, 1H), 7.66 (d, *J* = 15.6 Hz, 1H), 7.56-7.50 (m, 4H), 7.42 – 7.35 (m, 6H), 6.47 (d, *J* = 14.4 Hz, 1H), 6.42 (d, *J* = 14.1 Hz, 1H), 6.06 (s, 1H), 4.40 (t, *J* = 5.1 Hz, 2H), 3.75 (q, *J* = 5.1 Hz, 2H). NMR details were compared with literature report[4].

2-[(2E)-3-(4-chlorophenyl)prop-2-enamido]ethyl(2E)-3-(4-chlorophenyl)prop-2-enoate (3b). White solid, Yield: 29.9 mg (77%), Mp: 236-238 °C, ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.64 (dd, *J* = 22.1, 16.2 Hz, 2H), 7.48-7.33 (m, 8H), 6.41 (t, *J* = 16.2 Hz, 2H), 6.02 (s, 1H), 4.39 (t, *J* = 5.1 Hz, 2H), 3.77- 3.74 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$, DMSO-d6) δ 166.2, 165.6, 143.3,

138.6, 135.8, 134.7, 133.2, 132.3, 128.9, 128.8, 128.6, 128.5, 121.4, 117.9, 62.9, 38.3. HRMS calcd for $C_{20}H_{17}Cl_2NO_3Na$ $[M+Na]^+$: 412.0483, found: 412.0483.

2-[(2E)-3-(4-fluorophenyl)prop-2-enamido]ethyl (2E)-3-(4-fluorophenyl)prop-2-enoate (3c).

White solid, Yield: 30 mg (84%), Mp: 157-161 °C, ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.69 (d, *J* = 16.2 Hz, 1H), 7.62 (d, *J* = 15.6 Hz, 1H), 7.55- 7.47 (m, 4H), 7.08 (q, *J* = 9 Hz, 4H), 6.36 (t, *J* = 16.5 Hz, 2H), 6.01 (s, 1H), 4.39 (t, *J* = 5.1 Hz, 2H), 3.75 (q, *J* = 5.4 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.1, 165.9, 165.7, 165.3,144.4, 140.3, 130.9, 130.4, 130.1, 130.0, 129.7, 129.6, 120.0, 117.1, 116.3, 116.1, 116.0, 115.8, 63.4, 39.3. HRMS calcd for $C_{20}H_{18}F_2NO_3$ $[M+H]^+$: 358.1255, found: 358.1254.

2-[(2E)-3-(3-nitrophenyl)prop-2-enamido]ethyl (2E)-3-(3-nitrophenyl)prop-2-enoate (3d). White solid, Yield: 35.8 mg (88%), Mp: 115-116 °C, ^{1}H NMR (300 MHz, DMSO-d6) δ 8.57 (s,1H), 8.41 (br s, 2H), 8.24 (q, *J* = 7.2 Hz, 3H), 8.04 (d, *J* = 7.5 Hz, 1H), 7.86 (d, *J* = 15.9 Hz, 1H), 7.75- 7.69 (m, 2H), 7.60 (d, *J* = 15.9 Hz, 1H), 6.91 (d, *J* = 10.2 Hz, 1H), 6.85 (d, *J* = 10.2 Hz, 1H), 4.28 (t, *J* = 5.1 Hz, 2H), 3.55 (d, *J* = 5.4 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$, DMSO-d6) δ 164.5, 164.1, 147.2, 141.0, 135.72, 135.67, 134.7, 132.7, 129.0, 128.8, 123.4, 123.3, 122.3, 121.2, 120.2, 119.7, 62.0, 37.1. HRMS calcd for $C_{20}H_{18}N_3O_7$ $[M+H]^+$: 412.1145, found: 412.1144.

2-[(2E)-3-(4-cyanophenyl)prop-2-enamido]ethyl (2E)-3-(4-cyanophenyl)prop-2-enoate (3e). White solid, Yield: 27.2 mg (73%), Mp: 216-218 °C , ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.74 – 7.57 (m, 10H), 6.51 (t, *J* = 15.9 Hz, 2H), 6.08 (s, 1H), 4.42 (d, *J* = 4.2 Hz, 2H), 3.77 (d, *J* = 4.8 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$, DMSO-d6) δ 164.9, 164.5, 141.8, 138.7, 137.6, 136.8, 131.7,

131.6, 127.6, 127.2, 124.1, 120.4, 117.6, 117.4, 112.2, 111.3, 62.3, 37.5. HRMS calcd for $C_{22}H_{18}N_3O_3$ $[M+H]^+$: 372.1348, found: 372.1349.

[Ethane 2-[(3-nitrophenyl)formamido]ethyl 3-nitrobenzoate (3f). White solid, Yield: 47.3 mg (65%), Mp: 144-145 °C, (Lit 152–153°C)[5] , ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.88 (t, *J* = 1.8 Hz, 1H), 8.62 (t, *J* = 1.8 Hz, 1H), 8.46 – 8.36 (m, 3H), 8.19-8.16 (m, 1H), 7.72-7.65 (m, 2H), 6.85 (s, 1H), 4.66 (t, *J* = 5.1 Hz, 2H), 3.95 (q, *J* = 5.1 Hz, 2H). NMR details were compared with literature report[5].

N-(2-hydroxyethyl)benzamide (3g). Colorless gum, Yield: 28.4 mg (85%), ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7.2 Hz, 2H), 7.54 – 7.42 (m, 3H), 6.64 (s, 1H), 3.86 (t, *J* = 4.8 Hz, 2H), 3.65 (q, *J* = 5.4 Hz, 2H). NMR details were compared with literature report[6].

(2R)-2-phenyl-2-[(2E)-3-phenylprop-2-enamido]ethyl(2E)-3-phenylprop-2-enoate(3h). White solid, Yield: 26.8 mg (69%), Mp: 131-132 °C , ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.70 (d, *J* = 16.2 Hz, 1H), 7.64 (d, *J* = 15.6 Hz, 1H), 7.53-7.48 (m, 4H), 7.40 – 7.29 (m, 11H), 6.45 (dd, *J* = 15.6, 8.4 Hz, 3H), 5.54-5.48 (m, 1H), 4.68 (dd, *J* = 11.6, 7.6 Hz, 1H), 4.45 (dd, *J* = 11.6, 4.5 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.4, 165.5, 146.0, 141.8, 138.4, 134.7, 134.1, 130.6, 129.8, 128.93, 128.88, 128.8, 128.2, 128.0, 127.9, 126.8, 120.3, 117.2, 66.2, 53.2. HRMS calcd for $C_{20}H_{18}F_2NO_3$ $[M+H]^+$: 398.1756, found: 398.1757.

(2R)-2-[(2E)-3-(4-methoxyphenyl)prop-2-enamido]-2-phenylethyl(2E)-3-(4-methoxyphenyl)prop-2-enoate (3i). White solid, Yield: 34.1 mg (74%), Mp: 194-197 °C, 1H NMR (300 MHz, CDCl3) δ 7.65 (d, *J* = 15.9 Hz, 1H), 7.58 (d, *J* = 15.3 Hz, 1H) 7.48 – 7.30 (m, 9H), 6.89 (dd, *J* = 8.7, 5.1 Hz, 4H), 6.38 (d, *J* = 7.8 Hz, 1H), 6.34 (d, *J* = 8.7 Hz, 1H), 6.29 (d, *J* = 9.0 Hz, 1H), 5.52 - 5.46 (m, 1H), 4.66 (dd, *J* = 11.7, 7.8 Hz, 1H), 4.42 (dd, *J* = 11.7, 4.5 Hz,

1H), 3.83 (d, *J* = 3.6 Hz, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.8, 165.8, 161.6, 161.0, 145.6, 141.4, 138.6, 130.0, 129.5, 128.8, 127.9, 127.4, 126.9, 126.8, 117.9, 114.7, 114.4, 114.2, 66.1, 55.4. 55.4, 53.2. HRMS calcd for $C_{28}H_{28}NO_5$ $[M+H]^+$: 458.1967, found: 458.1968.

(2R)-2-[(2E)-3-(4-fluorophenyl)prop-2-enamido]-2-phenylethyl(2E)-3-(4-fluorophenyl)prop-2-enoate (3j). White solid, Yield: 23.7 mg (55%), Mp: 138-142 °C, ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.81-7.59 (m, 4H), 7.54 – 7.27 (m, 11H), 7.06 (q, *J* = 7.8 Hz, 4H), 6.56 (d, *J* = 15.9 Hz, 1H), 6.35 (d, *J* = 16.2 Hz, 1H), 5.50 (q, *J* = 5.7 Hz, 1H), 4.58 – 4.43 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.3, 165.3, 144.6, 140.6, 138.3, 130.9, 130.4, 130.3, 130.2, 130.1, 129.7, 129.6, 128.9, 128.1, 126.8, 120.0, 117.0, 116.3, 116.1, 116.0, 115.8, 66.2, 53.2. HRMS calcd for $C_{26}H_{22}F_2NO_3$ $[M+H]^+$: 434.1568, found: 434.1569.

(2R)-2-[(2E)-3-(4-chlorophenyl)prop-2-enamido]-2-phenylethyl(2E)-3-(4-chlorophenyl)prop-2-enoate (3k). White solid, Yield: 36.7 mg (79%), Mp: 120-123 °C, ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.60 (t, *J* = 16.8, 2H), 7.45 – 7.31 (m, 13H), 6.40 (q, *J* = 9 Hz, 3H), 5.53-5.47 (m, 1H), 4.68 (dd, *J* = 11.7, 8.1 Hz, 1H), 4.44 (dd, *J* = 11.7, 4.5 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.1, 165.2, 144.5, 140.5, 138.2, 136.6, 135.7, 133.2, 132.6, 129.4, 129.2, 129.1, 129.0, 128.9, 128.1, 126.8, 120.7, 117.8, 66.3, 53.2. HRMS calcd for $C_{26}H_{21}Cl_2NO_3Na$ $[M+Na]^+$:488.0796, found: 488.0786. IR (KBr) ν 3342, 1712, 1630, 1533, 1490, 1311, 1160, 1086, 973, 822, 700, 468 cm^{-1}.

(2S)-3-phenyl-2-[(2E)-3-phenylprop-2-enamido]propyl (2E)-3-phenylprop-2-enoate (3l). White solid, Yield: 33.8 mg (82%), Mp 164-166 °C, ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.72 (d, *J* = 15.9 Hz, 1H), 7.63 (d, *J* = 15.6 Hz, 1H), 7.56 – 7.48 (m, 4H), 7.41-7.28 (m, 10H), 6.48 (d, *J* = 15.9 Hz, 1H), 6.38 (d, *J* = 15.6 Hz, 1H), 5.93 (d, *J* = 8.1 Hz, 1H), 4.70-4.59 (m, 1H), 4.33-4.22

(m, 2H), 3.07 (dd, *J* = 13.5, 5.7 Hz, 1H), 2.93 (dd, *J* = 13.8, 8.1 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.2, 165.5, 145.8, 141.5, 137.0, 134.7, 134.2, 130.6, 129.8, 129.4, 129.0, 128.8, 128.7, 128.2, 127.9, 126.9, 120.4, 117.3, 64.8, 50.1, 37.6. HRMS calcd for $C_{27}H_{26}NO_3$ $[M+H]^+$: 412.1913, found: 412.1913.

(2S)-2-[(2E)-3-(4-bromophenyl)prop-2-enamido]-3-phenylpropyl(2E)-3-(4 bromophenyl)prop-2-enoate (3m). White solid, Yield: 32.3 mg (57%), Mp 101-103 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.68 – 7.64 (m, 3H), 7.60 – 7.43 (m, 5H), 7.40 – 7.20 (m, 7H), 6.46 (d, *J* = 15.9 Hz, 1H), 6.36 (d, *J* = 15.6 Hz, 1H), 5.99 (d, *J* = 8.1 Hz, 1H), 4.65 (d, *J* = 4.5 Hz, 1H), 4.33 – 4.22 (m, 2H), 3.05 (dd, *J* = 13.5, 6.0 Hz, 1H), 2.92 (dd, *J* = 13.5, 8.1 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.7, 165.0, 144.1, 140.0, 136.8, 136.2, 133.4, 132.6, 130.8, 130.5, 130.4, 130.3, 129.3, 128.8, 126.93, 126.87, 126.8, 123.1, 123.0, 121.8, 118.8, 65.0, 50.1, 37.6. HRMS calcd for $C_{27}H_{24}Br_2NO_3$ $[M+H]^+$: 568.0123, found: 568.0011. IR (KBr) ν 3273, 2921, 2851, 1710, 1626, 1555, 1175, 972, 783, 667, 443 cm^{-1}.

(2S)-2-[(2E)-3-(4-fluorophenyl)prop-2-enamido]-3-phenylpropyl(2E)-3-(4-fluorophenyl)prop-2-enoate (3n). White solid, Yield: 38.7 mg (86%), Mp: 104-106 °C ,^{1}H NMR (300 MHz, $CDCl_3$) δ 7.67 (d, *J* = 15.9 Hz, 1H), 7.59 (d, *J* = 15.9 Hz, 1H), 7.55-7.45 (m, 4H), 7.35 – 7.25 (m, 5H), 7.12- 7.02 (m, 4H), 6.39 (d, *J* = 15.9 Hz, 1H), 6.29 (d, *J* = 15.6 Hz, 1H), 5.93 (d, *J* = 8.4 Hz, 1H), 4.64 (d, *J* = 4.5 Hz, 1H), 4.33-4.21 (m, 2H), 3.05 (dd, *J* = 13.8, 5.7 Hz, 1H), 2.92 (dd, *J* = 13.8, 8.1 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.1, 165.7, 165.4, 162.4, 161.9, 144.5, 140.3, 136.9, 130.94, 130.90, 130.42, 130.37, 130.2, 130.1, 129.7, 129.6, 129.3, 128.7, 126.9, 120.1, 117.0, 116.3, 116.1, 116.0, 115.8, 64.8, 50.1, 37.6. HRMS calcd for $C_{27}H_{24}F_2NO_3$ $[M+H]^+$: 448.1724, found: 448.1725. IR (KBr) ν 3272, 1710, 1628, 1509, 1318, 1279, 1232, 972, 827, 700, 452 cm^{-1}.

(1R)-1-phenyl-2-[(2E)-3-phenylprop-2-enamido]ethyl(2E)-3-phenylprop-2-enoate (3o). White solid, Yield: 31 mg (72%), Mp: 178-180 °C, (Lit 181-183)[7], $[\alpha]_D^{31}$ - 69.0 (c 0.07, $CHCl_3$), (Lit: $[\alpha]_D^{28}$ - 57.3 (c 0.5, $CHCl_3$))[7], ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.74 (d, *J* = 15.9 Hz, 1H), 7.63 (d, *J* = 15.6 Hz, 1H), 7.55 – 7.34 (m, 15H), 6.52 (d, *J* = 15.9 Hz, 1H), 6.38 (d, *J* = 15.6 Hz, 1H), 6.06 (dd, *J* = 8.4, 4.0 Hz, 1H), 5.96 (br s, 1H), 3.98 – 3.78 (m, 2H). NMR details were compared with literature report[7].

(1R)-1-phenyl-2-[(2E)-3-(thiophen-2-yl)prop-2-enamido]ethyl(2E)-3-(thiophen-2-yl)prop-2-enoate (3p). White solid, Yield: 33.6 mg (82%), Mp: 164-166 °C, ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.83 (d, *J* = 15.6 Hz, 1H), 7.75 (d, *J* = 15 Hz, 1H), 7.44-7.28 (m, 8H), 7.21 (d, *J* = 3.3 Hz, 1H), 7.08 - 7.01 (m, 2H), 6.31 (d, *J* = 15.6 Hz, 1H), 6.17 (d, *J* = 15 Hz, 1H), 6.02 (dd, *J* = 8.7 Hz, 4.2 Hz, 1H), 5.86 (br s, 1H), 3.96 – 3.88 (m, 1H), 3.82 – 3.73 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.3, 165.7, 139.9, 139.4, 138.3, 137.7, 134.3, 131.5, 130.5, 128.9, 128.8, 128.6, 128.2, 128.0, 127.5, 126.5, 119.1, 116.2, 74.8, 44.8. HRMS calcd for $C_{22}H_{20}NO_3S_2$ $[M+H]^+$: 410.0885, found:410.0903. IR (KBr) ν 3320, 2920, 1705, 1619, 1546, 1204, 1165, 1103, 1041, 833, 698, 458 cm^{-1}.

(2R)-2-phenyl-2-[(2E)-3-(thiophen-2-yl)prop-2-enamido]ethyl(2E)-3-(thiophen-2-yl)prop-2-enoate (3q). White solid, Yield: 29.1 mg (71%), Mp: 138-140 °C, ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.80 (d, *J* = 13.8 Hz, 1H), 7.74 (d, *J* = 13.5 Hz, 1H), 7.40-7.31 (m, 8H), 7.20 (d, *J* = 3.3 Hz, 1H), 7.07- 7.01 (m, 2H), 6.34 (d, *J* = 7.8 Hz, 1H), 6.27 (d, *J* = 11.7 Hz, 1H), 6.21 (d, *J* = 12.3 Hz, 1H), 5.51- 5.45 (m, 1H), 4.64 (dd, *J* = 11.4, 7.2 Hz, 1H), 4.42 (dd, *J* = 11.7, 4.5 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.2, 165.2, 139.9, 139.3, 138.4, 138.3, 134.5, 131.4, 130.5, 128.93,

128.86, 128.2, 128.0, 127.5, 126.8, 119.1, 115.9, 66.2, 53.1. HRMS calcd for $C_{22}H_{20}NO_3S_2$ $[M+H]^+$: 410.0885, found: 410.0902.

(2S)-2-[(2E)-3-(furan-2-yl)prop-2-enamido]-3-phenylpropyl(2E)-3-(furan-2-yl)prop-2enoate(3r). White solid, Yield: 22.8 mg (58%), Mp: 122-124 °C, ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 – 7.42 (m, 4H), 7.37 – 7.23 (m, 4H), 6.65 (d, *J* = 3.6 Hz, 1H), 6.55 (d, *J* = 3.3 Hz, 1H), 6.50 - 6.44 (m, 2H), 6.35 (d, *J* = 15.6 Hz, 1H), 6.27 (d, *J* = 15.3 Hz, 1H), 5.86 (d, *J* = 8.1 Hz, 1H), 4.66 – 4.55 (m, 1H), 4.28 – 4.17 (m, 2H), 3.03 (dd, *J* = 13.8, 5.7 Hz, 1H), 2.90 (dd, *J* = 13.5, 8.1 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.2, 165.4, 151.2, 150.8, 145.0, 144.1, 137.0, 131.9, 129.4, 128.7, 128.3, 126.8, 118.1, 115.4, 114.9, 114.0, 112.4, 112.2, 64.6, 50.0, 37.6. HRMS calcd for $C_{23}H_{22}NO_5$ $[M+H]^+$: 392.1498, found: 392.1516. IR (KBr) ν 3376, 2926, 1716, 1634, 1535, 1262, 1166, 1020, 749, 701 cm^{-1}.

2-[(2E)-3-(furan-2-yl)prop-2-enamido]ethyl(2E)-3-(furan-2-yl)prop-2-enoate(3s)[8]. off-White solid, Yield: 18.7 mg (62%), Mp: 94-97 °C, (Lit 100-101 °C)[8]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50- 7.39 (m, 4H), 6.65 (d, *J* = 3.6 Hz, 1H), 6.56 (d, *J* = 3.6 Hz, 1H), 6.49- 6.44 (m, 2H), 6.35 (d, *J* = 5.1 Hz, 1H), 6.30 (d, *J* = 4.8 Hz, 1H), 5.99 (br s, 1H), 4.35 (t, *J* = 5.1 Hz, 2H), 3.74-3.69 (m, 2H).

3-phenyl-3-[(2E)-3-phenylprop-2-enamido]propyl(2E)-3-phenylprop-2enoate(3v). White solid, Yield: 27.7 mg (66%), Mp: 176-179 °C, ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.67 (d, *J* = 4.8 Hz, 1H), 7.62 (d, *J* = 4.5 Hz, 1H), 7.52 - 7.45 (m, 4H), 7.39-7.28 (m, 11H), 6.42 (d, *J* = 16.2 Hz, 2H), 6.11 (d, *J* = 8.1 Hz, 1H), 5.34 (q, *J* = 7.2 Hz, 1H), 4.27 (t, *J* = 6.3 Hz, 2H), 2.42-2.22 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.9, 165.2, 145.1, 141.6, 141.2, 134.7, 134.3, 130.4, 129.8, 128.93, 128.91, 128.8, 128.2, 127.82, 127.8, 126.6, 120.5, 117.8, 61.7, 51.3, 34.9. HRMS calcd

for $C_{27}H_{26}NO_3$ $[M+H]^+$: 412.1913, found: 412.1929. IR (KBr) ν 3273, 3054, 2940, 1711, 1617, 1544, 1316, 1172, 982, 767, 707, 464 cm^{-1}.

3-[(2E)-3-(4-bromophenyl)prop-2-enamido]-3-phenylpropyl(2E)-3-(4-bromophenyl)prop-2-enoate(3w). White solid, Yield: 44 mg (74%), Mp: 168-172 °C, ^1H NMR (300 MHz, $CDCl_3$) δ 7.61-7.59 (m, 1H), 7.59 – 7.46 (m, 5H), 7.40 - 7.28 (m, 9H), 6.41 (d, *J* = 3.0 Hz, 1H), 6.36 (d, *J* = 2.4 Hz, 1H), 6.03 (d, *J* = 8.1 Hz, 1H), 5.36-5.29 (m, 1H), 4.27 (t, *J* = 5.4 Hz, 2H), 2.39-2.18 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.6, 164.8, 143.7, 141.0, 140.3, 133.6, 133.2, 132.2, 132.1, 129.5, 129.2, 129.0, 127.9, 126.6, 124.7, 124.0, 121.0, 118.5, 61.8, 51.2, 34.8. HRMS calcd for $C_{27}H_{24}Br_2NO_3$ $[M+H]^+$: 568.0123, found: 568.0141.

2-[methyl(2-{[2E)-3-phenylprop-2-enoyl]oxy}ethyl)amino]ethyl(2E)-3-phenylprop-2-enoate(3x). Colorless oil, Yield: 20.5 mg (53%), ^1H NMR (300 MHz, $CDCl_3$) δ 7.68 (d, *J* =15.9 Hz, 2H), 7.51 – 7.48 (m, 4H), 7.37 – 7.34 (m, 6H), 6.47 (d, *J* =15.9 Hz, 2H), 4.34 (t, *J* = 5.7 Hz, 4H), 2.84 (t, *J* = 5.7 Hz, 4H), 2.44 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.0, 145.1, 134.4, 130.3, 128.9, 128.1, 117.9, 62.2, 56.0, 43.1. HRMS calcd for $C_{23}H_{26}NO_4$ $[M+H]^+$: 380.1862, found: 380.1878.

(2E)-3-phenyl-N-[2-{2-{2-[(2E)-3-phenylprop-2-enamido]ethoxy)ethyl]prop-2-enamide(3y).

Off-white solid, Yield: 16.6 mg (40%), M.P: 98 °C, ^1H NMR (300 MHz, $CDCl_3$) δ 7.63 (d, *J* = 15.6 Hz, 2H), 7.50-7.47 (m, 4H), 7.37-7.32 (m, 6H), 6.47 (d, *J* = 15.6 Hz, 2H), 6.37 (s, 2H), 3.66-3.61 (m, 12H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.2, 141.1, 134.8, 129.7, 128.8, 127.8, 120.6, 70.3, 70.0, 39.5. HRMS calcd for $C_{24}H_{29}N_2O_4$ $[M+H]^+$: 409.2127, found: 409.2139.

5. Synthesis of Unsymmetrically substituted analogues of syncarpamide derivatives using NHC organocatalysis:

A 10 mL reaction vial was charged with amino alcohol derivatives (0.3 mmol), Methyl cinnamate (0.3 mmol) and water (2 mL). To this mixture, potassium carbonate (0.042 g, 0.3 mmol) were added; the reaction vial was closed with rubber septa and heated at 60 ^{o}C for 5 h. After 5 h, the substituted esters derivatives (0.2 mmol) and NHC-D (0.014 g, 0.03 mmol) were added. Then, the reaction vial was tightly sealed with crimper cap and allowed to stir for 12 h at room temperature. After completion of 12 h, the reaction mixture was extracted with ethyl acetate (3×10 mL). The combined organic layer was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The afforded crude mixture was purified by flash chromatography on silica gel (200-400 mesh) in 50-80% ethyl acetate in hexanes to afford the product.

2-[(2E)-3-phenylprop-2-enamido] ethyl benzoate (4a). White solid, Yield: 40.8 mg (45%), Mp: 105-109 °C (Lit 110–111°C)[9], ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.2 Hz, 2H), 7.74 (d, *J*=16.2 Hz, 1H), 7.54 – 7.39 (m, 8H), 6.68 (br s, 1H), 6.47 (d, *J* = 15.9 Hz, 1H), 4.46 (t, *J* = 5.1 Hz, 2H), 3.82 (q, *J* = 5.1 Hz, 2H). NMR details were compared with literature report[9].

2-[(2E)-3-(4-bromophenyl)prop-2-enamido]ethyl benzoate (4b). White solid, Yield: 53.3 mg (47%), Mp: 138-140 °C, ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.79 (d, *J* = 6.9 Hz, 2H), 7.68 (s, 1H), 7.62 (s, 1H), 7.54- 7.43 (m, 5H), 7.30 – 7.25 (m, 2H), 6.61 (s, 1H), 6.46 (d, *J* = 15.9 Hz, 1H), 4.45 (t, *J* = 15.9 Hz, 2H), 3.82 (q, *J* = 4.8 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.6, 167.1, 144.4, 134.2, 133.1, 132.2, 131.7, 129.6, 128.7, 127.0, 124.9, 118.0, 63.6, 39.8. HRMS calcd for $C_{18}H_{16}BrNO_3Na$ $[M+Na]^+$: 396.0211, found: 396.0214.

2-[(2E)-3-(3-nitrophenyl)prop-2-enamido]ethyl benzoate (4c). White solid, Yield: 53.4 mg (52%), Mp: 110-130 °C, ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.39 (s, 1H), 8.25 (d, *J* = 8.1 Hz, 1H), 7.84- 7.74 (m, 4H), 7.62- 7.43 (m, 4H), 6.46 (d, *J* = 15.9 Hz, 1H), 4.48 (t, *J* = 5.1 Hz, 2H), 3.83 (q, *J* = 5.1 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.7, 166.5, 148.7, 142.8, 135.9, 134.2, 133.8, 131.7, 130.1, 128.7, 127.0, 124.8, 122.5, 120.6, 63.8, 39.6. HRMS calcd for $C_{18}H_{17}N_2O_5$ $[M+H]^+$: 341.1137, found: 341.1138. IR (KBr) ν 3294, 3072, 2929, 1715, 1638, 1529, 1352, 1175, 994, 803, 697, 468 cm^{-1}.

(2R)-2-phenyl-2-[(2E)-3-phenylprop-2-enamido] ethyl benzoate (4d). Off-white solid, Yield: 54 mg (40%), Mp: 170-173 °C, ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.83 (d, *J* = 7.5 Hz, 1H), 7.67 (d, *J* = 15.6 Hz, 1H), 7.52 – 7.47 (m, 3H), 7.45 – 7.29 (m, 11H), 6.48 (d, *J* = 15.9 Hz, 1H), 6.35 (d, *J* = 6.3 Hz, 1H), 5.30 – 5.19 (m, 1H), 4.04 – 3.93 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.4, 142.1, 138.8, 134.6, 129.9, 129.0, 128.9, 128.1, 127.9, 127.1, 126.8, 120.0, 66.9, 56.4. HRMS calcd for $C_{24}H_{22}NO_3$ $[M+H]^+$: 372.1600, found: 372.1602.

6. NHC mediated transesterification reaction in aqueous medium:

A 10 mL reaction vial was charged with alcohol derivatives (0.2 mmol), substituted ester (0.2 mmol) and water (2 mL). To this mixture, potassium carbonate (0.042 g, 0.3 mmol) and NHC-D (0.013 g, 0.03 mmol) were added; the reaction vial was closed with rubber septa and purged with argon. After purging for 5 minutes, the reaction vial was tightly sealed with crimper cap and allowed to stir for 12 h at room temperature. After completion of 12 h, the reaction mixture was extracted with ethyl acetate (3×10 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The afforded crude

mixture was purified by flash chromatography on silica gel (200-400 mesh) in 10% ethyl acetate in hexanes to afford the product.

Methyl (2E)-3-phenylprop-2-enoate (5a). Colorless gum, Yield: 29 mg (88%), ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.70 (d, *J* = 15.9 Hz, 1H), 7.55 – 7.50 (m, 2H), 7.40 – 7.36 (m, 3H), 6.45 (d, *J* = 15.9 Hz, 1H), 3.81 (s, 3H). NMR details were compared with literature report[10].

(2-Bromophenyl)methyl (2E)-3-phenylprop-2-enoate (5b). Colorless gum, Yield: 56.3 mg (89%), ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.76 (d, *J* = 15.9 Hz, 1H), 7.60 (dd, *J* = 7.9, 1.0 Hz, 1H), 7.56– 7.51 (m, 2H), 7.47 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.40 – 7.31 (m, 4H), 7.24- 7.18 (m, 1H), 6.52 (d, *J* = 16.2 Hz, 1H), 5.34 (s, 2H). NMR details were compared with literature report[10].

(2-Iodophenyl)methyl (2E)-3-phenylprop-2-enoate (5c). Colorless gum, Yield: 61.8 mg (85%), ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 7.9, 0.9 Hz, 1H), 7.77 (d, *J* = 15.9 Hz, 1H), 7.56 – 7.53 (m, 2H), 7.46 – 7.35 (m, 5H), 7.08- 7.02 (m, 1H), 6.52 (d, *J* = 16.2 Hz, 1H), 5.28 (s, 2H). NMR details were compared with literature report[11].

Octadecyl (2E)-3-phenylprop-2-enoate (5d). Colorless gum, Yield: 57.3 mg (73%), ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.68 (d, *J* = 15.9 Hz, 1H), 7.55 – 7.52 (m, 2H), 7.39 – 7.36 (m, 3H), 6.45 (d, *J* = 15.9 Hz, 1H), 4.19 (t, *J* = 13.5 Hz, 6.9 Hz, 2H) , 1.74-1.63 (m, 2H), 1.25 (s, 30H), 0.87 (t, *J* = 13.2 Hz, 6.9 Hz, 3H). NMR details were compared with literature report[12].

Naphthalen-2-yl (2E)-3-phenylprop-2-enoate (5e). White solid, Yield: 38.3 mg (70%), Mp 106-109 °C (Lit 109-110 °C)[13], ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.90 – 7.81 (m, 4H), 7.65 – 7.60 (m, 3H), 7.53 – 7.43 (m, 5H), 7.32 (dd, *J* = 8.9, 2.2 Hz, 1H), 6.69 (d, *J* = 16.2 Hz, 1H). NMR details were compared with literature report[13].

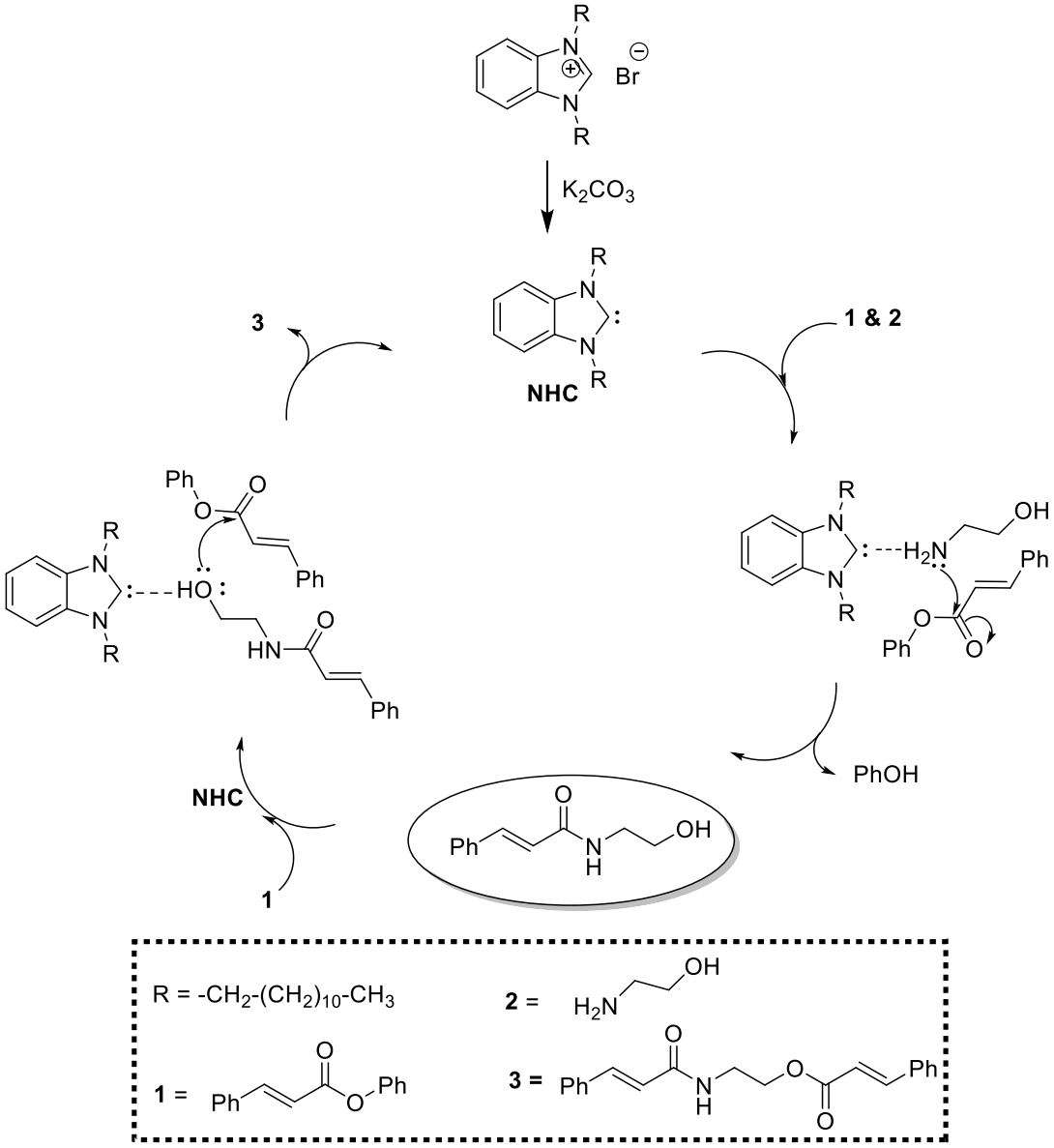
Phenyl (2E)-3-phenylprop-2-enoate (5f). White solid, Yield: 36.8 mg (82%), Mp: 70-71 °C, (Lit 73 °C)[13]; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.88 (d, *J* = 15.9 Hz, 1H), 7.61 – 7.56 (m, 2H), 7.45 – 7.38 (m, 5H), 7.28 – 7.23 (m, 1H), 7.19 – 7.16 (m, 2H), 6.64 (d, *J* = 15.9 Hz, 1H). NMR details were compared with literature report[13].

7. Synthesis of transesterification of benzoin derivatives using NHC organocatalysis:

A 10 mL reaction vial was charged with benzoin derivatives (0.2 mmol), Substituted esters (0.2 mmol) and water (2 mL). To this mixture, potassium carbonate (0.069 g, 0.5 mmol) and NHC-D (0.022 g, 0.05 mmol) were added; the reaction vial was closed with rubber septa and purged with nitrogen. After purging for 5 minutes, the reaction vial was tightly sealed with crimper cap and allowed to stir for 3 h at 60 °C. After completion of 3 h, the reaction mixture was extracted with ethyl acetate (3×10 mL). The combined organic layer was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The afforded crude mixture was purified by flash chromatography on silica gel (200-400 mesh) in 10% ethyl acetate in hexanes to afford the product.

1,2-Bis(phenyl)-2-oxoethyl (2E)-3-phenylprop-2-enoate (5g). White solid, Yield: 50.2 mg (74%), Mp: 100-102 °C, (Lit 104-106 °C)[14]; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.98 (d, *J* = 7.2 Hz, 2H), 7.80 (d, *J* = 15.9 Hz, 1H), 7.55 – 7.50 (m, 5H), 7.44 – 7.36 (m, 8H), 7.01 (s, 1H), 6.59 (d, *J* = 15.9 Hz, 1H). NMR details were compared with literature report[14].

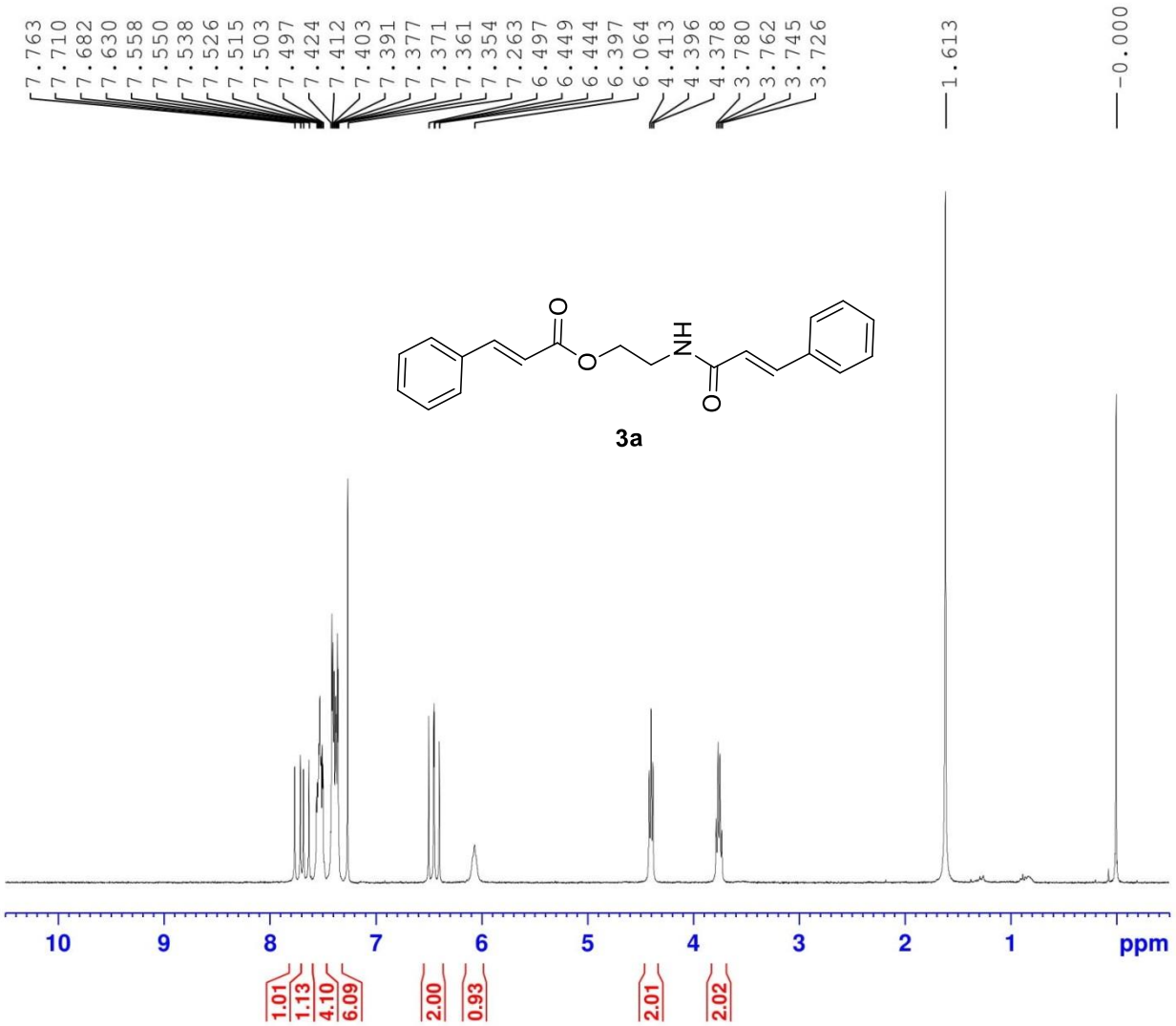
8. Mechanism



9. References

1. (a) B.M. Trost and K. Hirano, *Org. Lett.*, 2012, **14**, 2446–2449. (b) A Matviitsuk, M.D. Greenhalgh, D.J.B. Antfflnez, A. M. Z. Slawin, and A. D. Smith, *Angew. Chem*, 2017, **129**, 1-7.
2. L. Wang and E. Y.-X. Chen, *Green Chem.*, 2015, **17**, 5149-5153.
3. O. Torre, V. Gotor-Ferna´ndez and V. Gotor, *Tetrahedron:Asymmetry*, 2006, **17**, 860-866.
4. H. Sato, and S. Hosokawa, *Synthesis*, 2018, **50**, 1343-1349.
5. H. Brintzinger and H. Koddebusch, *Chem. Ber.*, 1949, **82,** 201-203.
6. M. Movassaghi, and M. A. Schmidt, *Org. Lett.*, 2005, **7**, 2453–2456.
7. E. K. Aratikatla , T. R. Valkute , S. K. Puri , K. Srivastava, and A. K. Bhattacharya, *Eur J. Med. Chem*., 2017, **138**, 1089-1105.
8. I. Castro, C. Diaz de Arce, J. Popelis, M.D. Gonzalez, E. Pastrana, E. Lukevics, *Latv. PSR Zinat. Akad. Vestis, Kim. Ser.*, 1989, **6**, 744-750.
9. W.R. Bowman, D.R. Coghlan, and H. Shah, *Comptes Rendus de l'Académie des Sciences - Series IIC – Chemistry*, 2001, 4, 625-640.
10. A. B. Lutjen, M. A. Quirk, A. M. Barbera and E. M. Kolonko, *Bioorg. Med. Chem.*, 2018, 26, 5291- 5298.
11. B. M. Jordan, Synthesis of benzoheterocycles using intramolecular cyclisation of aryl radicals, 1990, URL: https://dspace.lboro.ac.uk/2134/14445, Page 163.
12. C.T. Chen, J.H. Kuo, C.H. Ku, S.S. Weng, and C.Y. Liu, *J. Org. Chem.*, 2005, **70**, 1328-1339.

13. V. Palermo, D. M. Ruiz, J. C. Autino, P. G. Vázquez, and G. P. Romanelli, *Pure Appl. Chem.*, 2012, **84**, 529–540.
14. Q. Zhao, B. Han, B. Wang, H.J. Leng, C. Peng, and W. Huang, *RSC Adv.*, 2015, **5**, 26972-26976.

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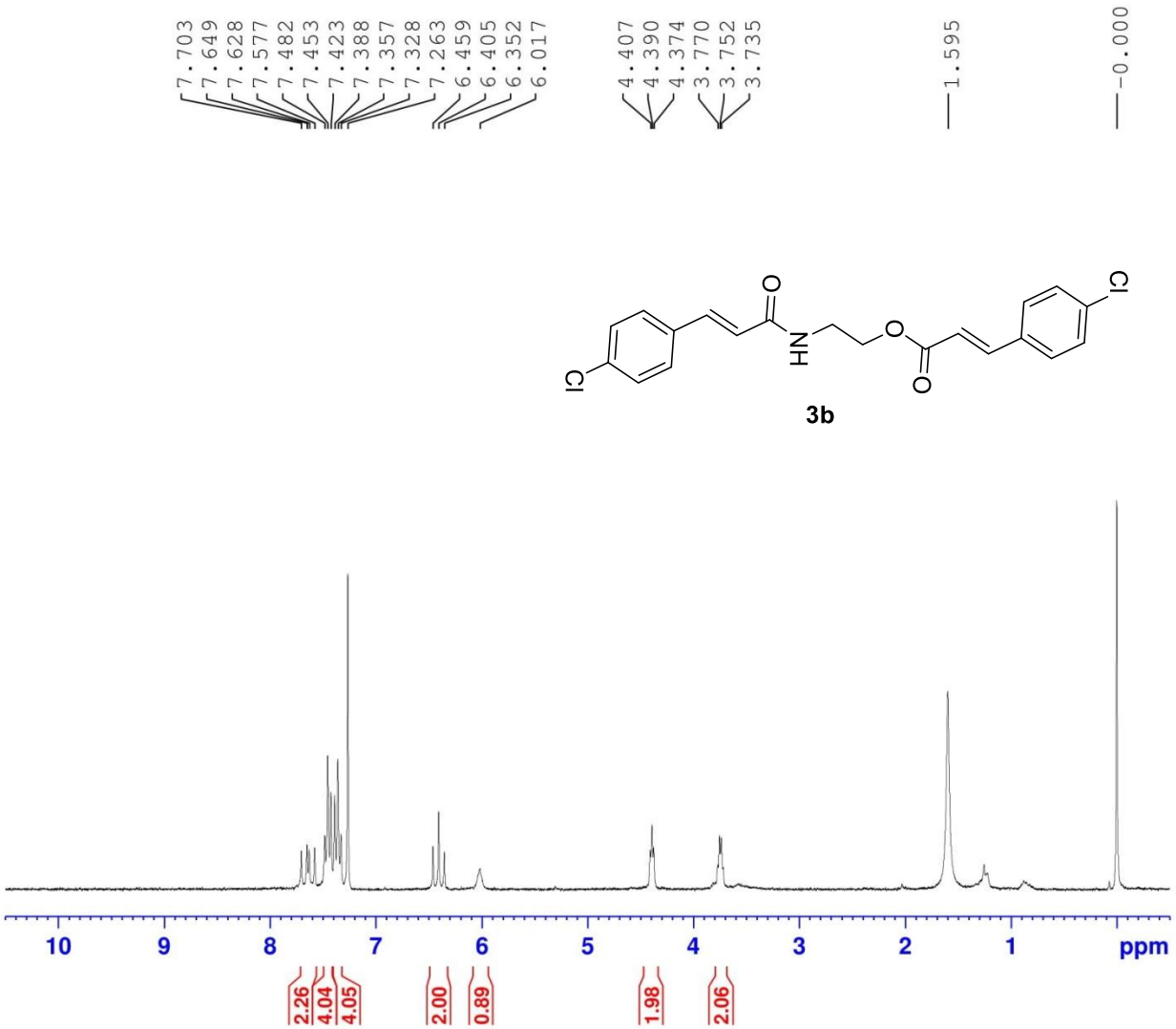


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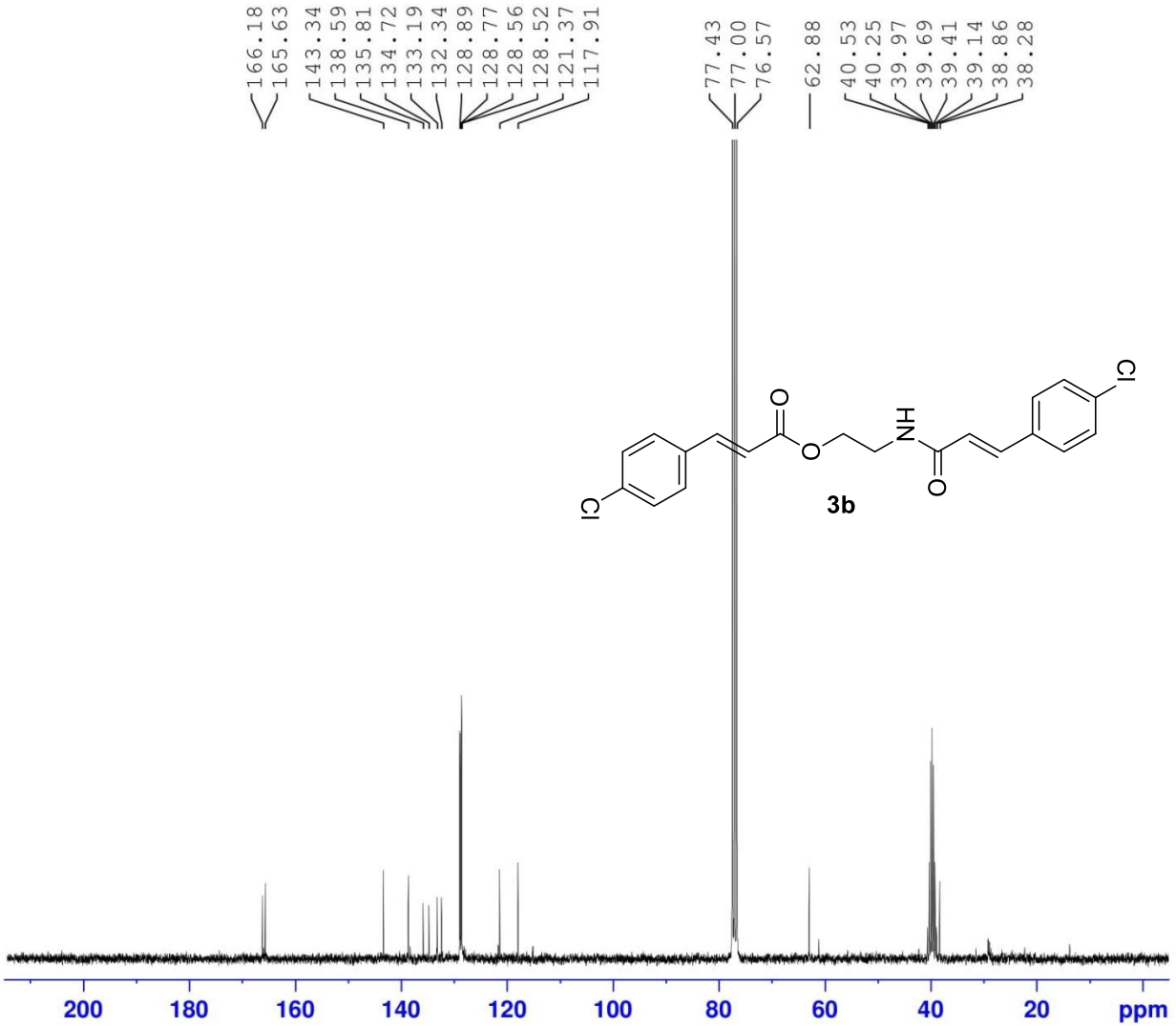


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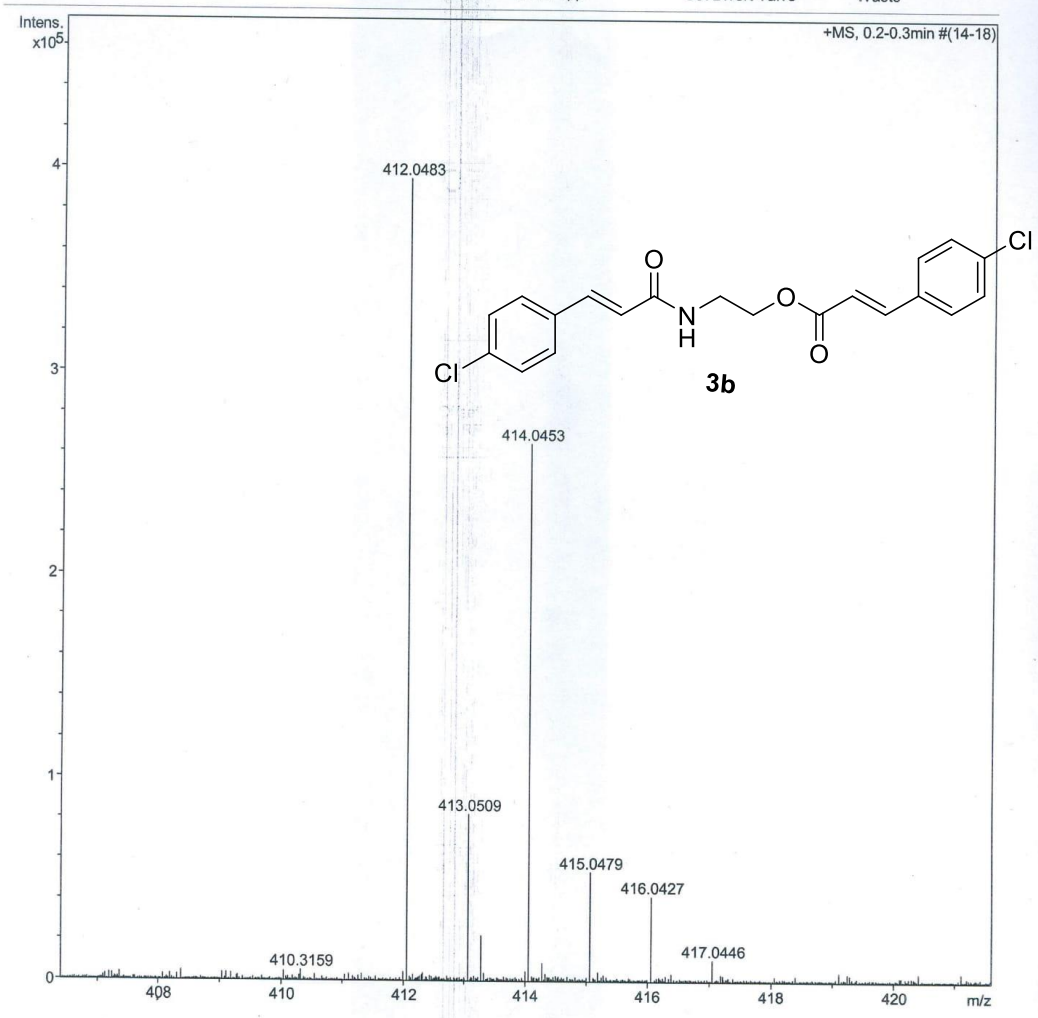

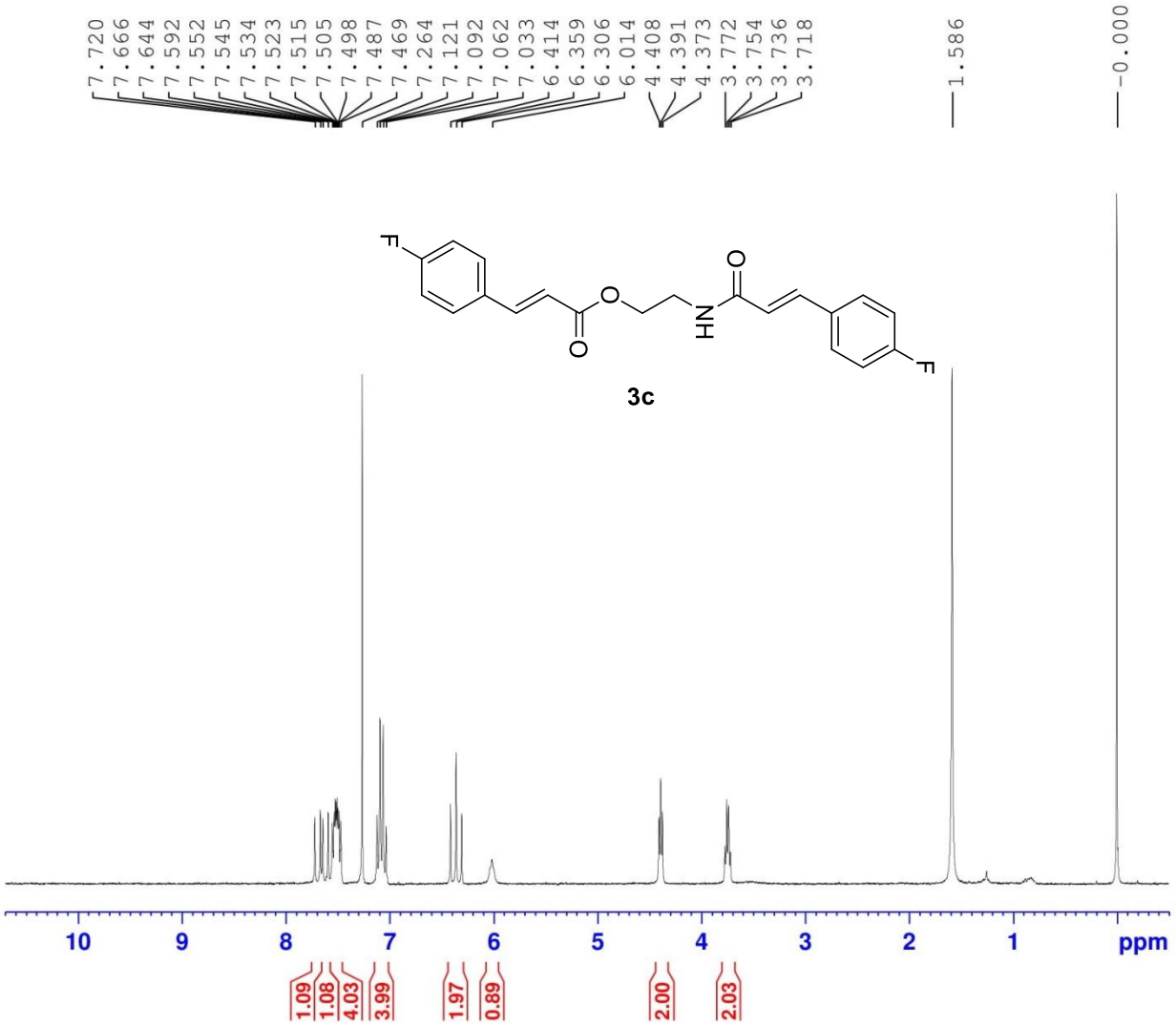


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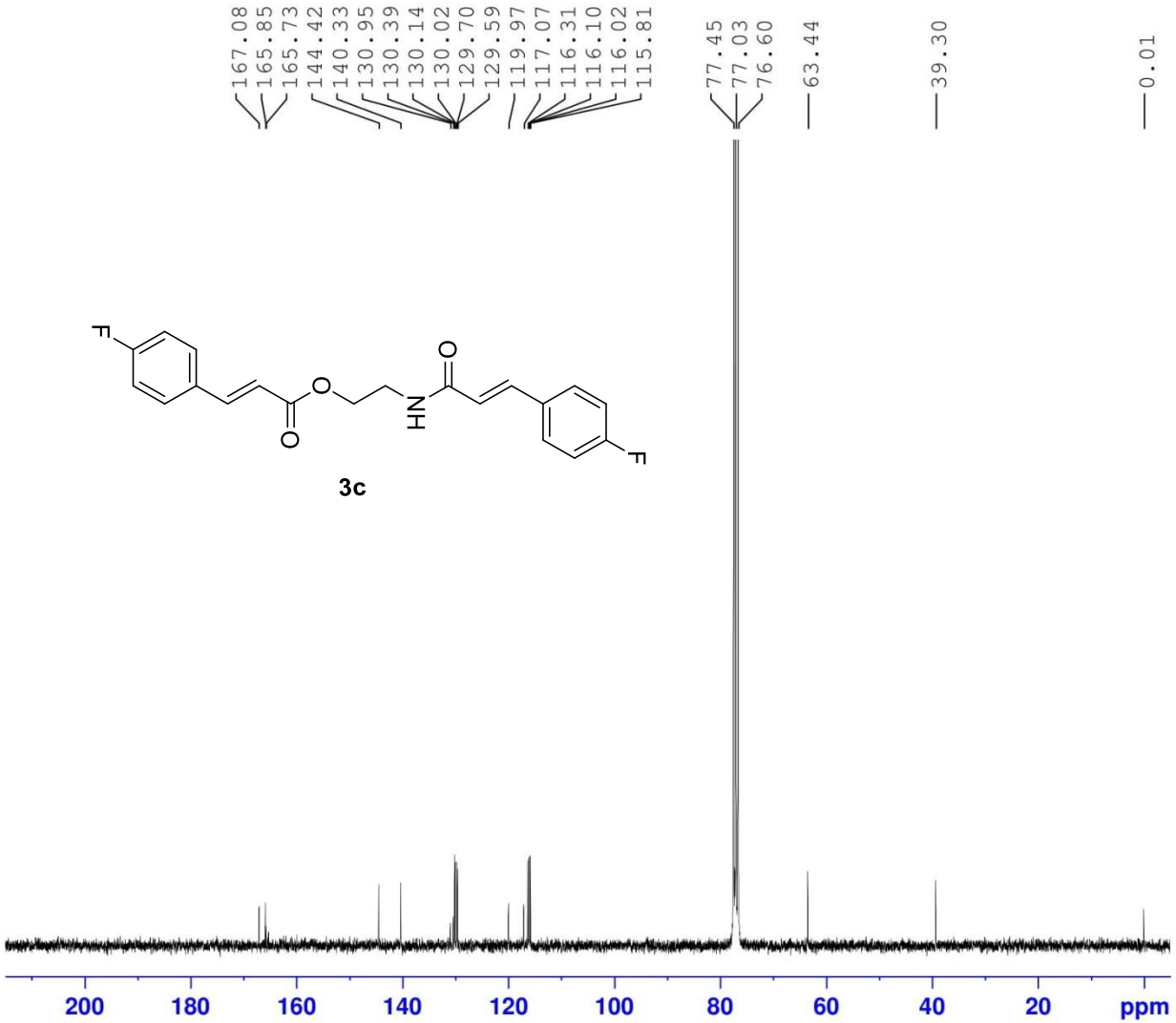
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190124
Time               9.36
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4525952 sec
RG                   456
DW                83.200 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                 1H
P1                13.00 usec
PLW1         10.69999981 W

F2 - Processing parameters
SI                 65536
SF           300.1300057 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```




```
Current Data Parameters
NAME                 C2552F7
EXPNO                      3
PROCNO                     1

F2 - Acquisition Parameters
Date_               20190124
Time                   23.11
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG              zgpg30
TD                     65536
SOLVENT                CDCl3
NS                      5120
DS                         4
SWH                18028.846 Hz
FIDRES             0.275098 Hz
AQ                 1.8175317 sec
RG                       575
DW                    27.733 usec
DE                     6.50 usec
TE                    300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1               75.4752949 MHz
NUC1                      13C
P1                       9.47 usec
PLW1              38.00000000 W

======== CHANNEL f2 ========
SFO2              300.1312005 MHz
NUC2                      1H
CPDPRG[2              waltz16
PCPD2                  90.00 usec
PLW2              10.69999981 W
PLW12              0.22325000 W
PLW13              0.11229000 W

F2 - Processing parameters
SI                       32768
SF                 75.4677485 MHz
WDW                         EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```

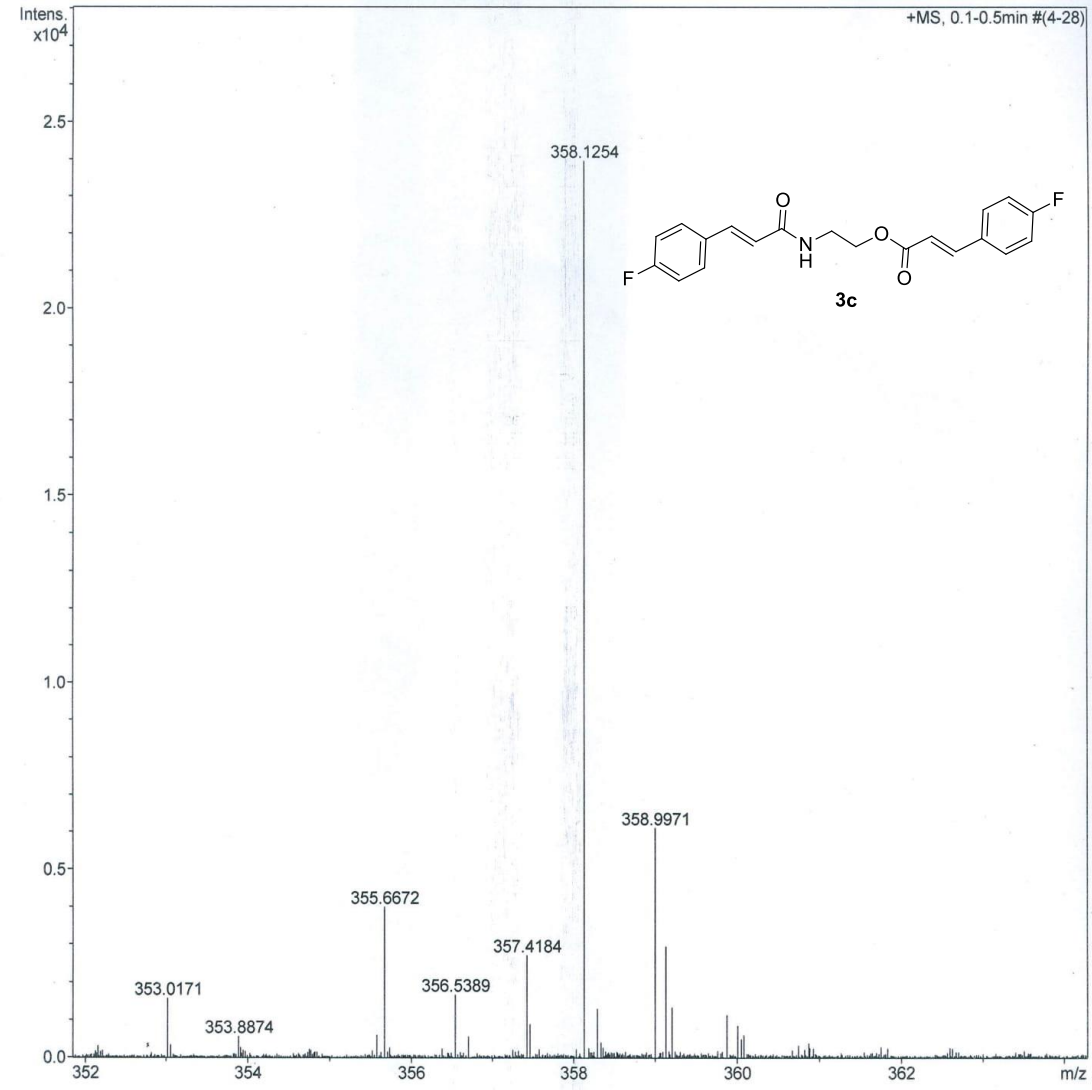
UOH -SCHOOL OF CHEMISTRY -HRMS

Analysis Info

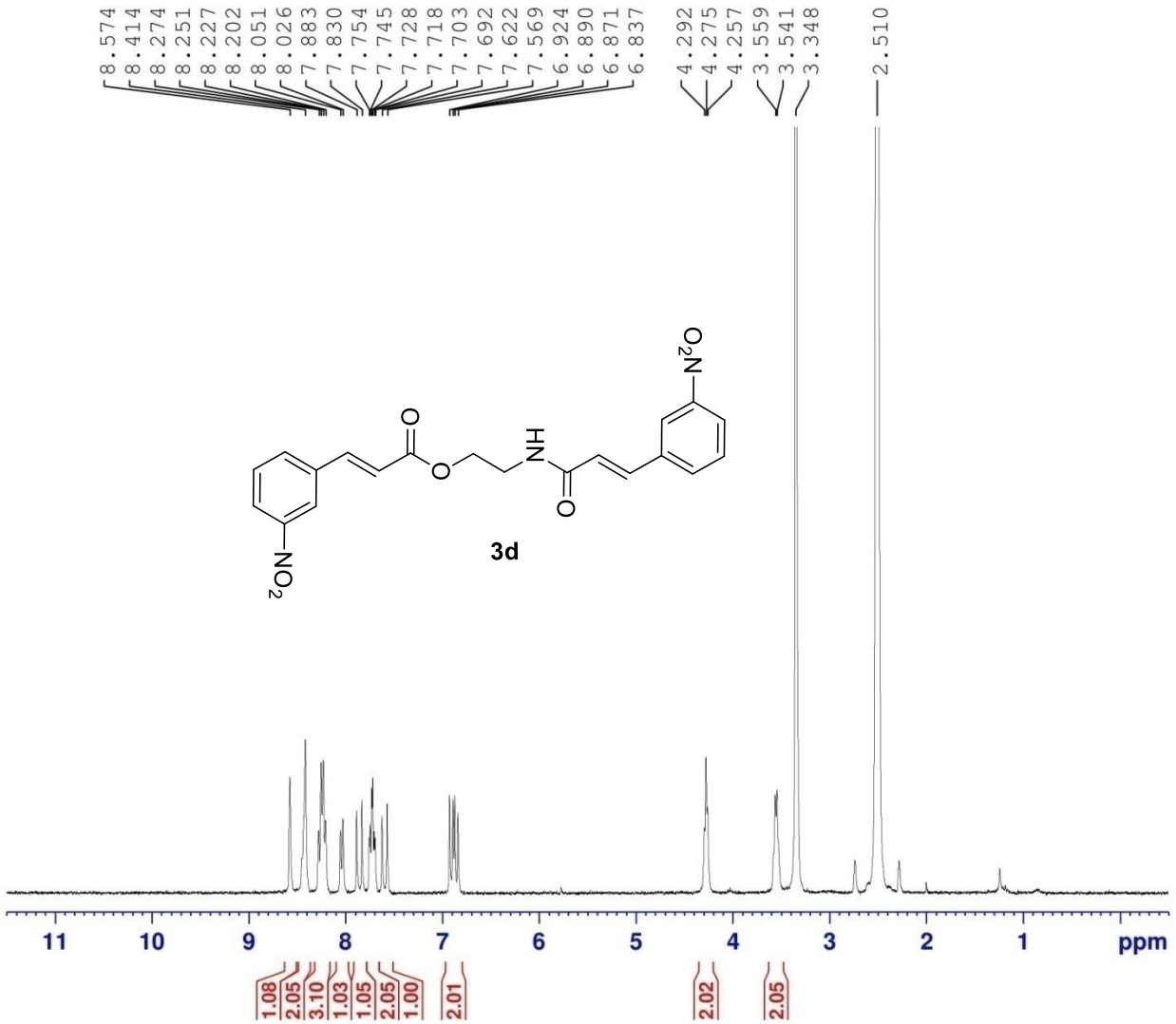
Analysis Name	D:\Data\2018\PROF MP\DEC\2.d	Acquisition Date	12/11/2018 4:14:49 PM
Method	tune_low.m	Operator	UOH-Chemistry
Sample Name	2	Instrument	maXis 10138
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3800 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1800 m/z	Set Collision Cell RF	350.0 Vpp	Set Divert Valve	Waste



Bruker Compass DataAnalysis 4.0 printed: 12/11/2018 5:10:49 PM Page 1 of 1

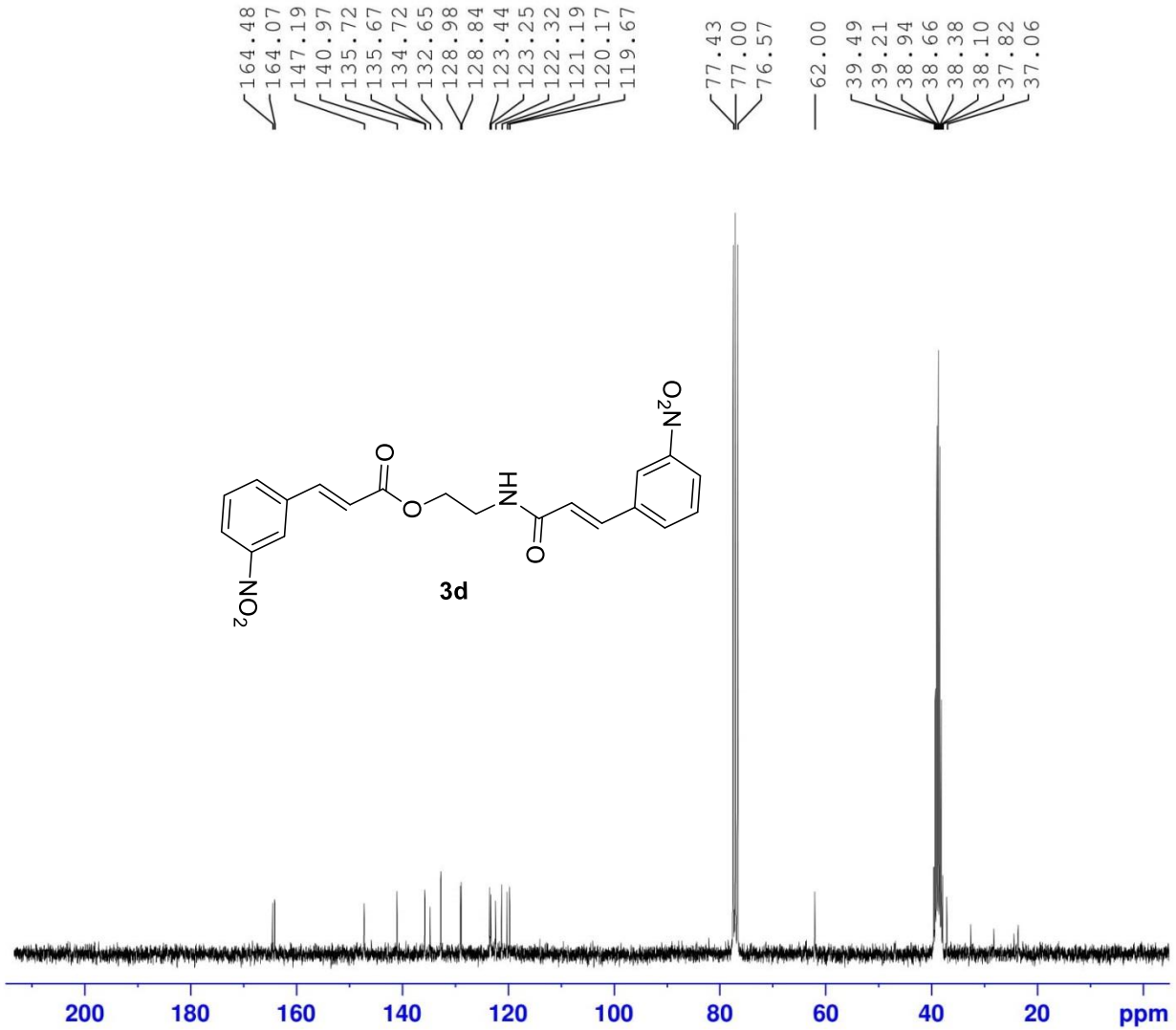



```
Current Data Parameters
NAME            H2552B10
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180725
Time               16.26
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    32
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4525952 sec
RG                   228
DW                83.200 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          300.1318534 MHz
NUC1                  1H
P1                 13.00 usec
PLW1          10.69999981 W

F2 - Processing parameters
SI                 65536
SF            300.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME                 C2552D9
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_               20180929
Time                   20.41
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG              zgpg30
TD                     65536
SOLVENT                CDCl3
NS                      1024
DS                         4
SWH                18028.846 Hz
FIDRES             0.275098 Hz
AQ                 1.8175317 sec
RG                       812
DW                    27.733 usec
DE                     6.50 usec
TE                    300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1               75.4752949 MHz
NUC1                      13C
P1                     9.47 usec
PLW1              38.00000000 W

======== CHANNEL f2 ========
SFO2              300.1312005 MHz
NUC2                      1H
CPDPRG[2              waltz16
PCPD2                 90.00 usec
PLW2              10.69999981 W
PLW12              0.22325000 W
PLW13              0.11229000 W

F2 - Processing parameters
SI                        32768
SF                 75.4682120 MHz
WDW                          EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```

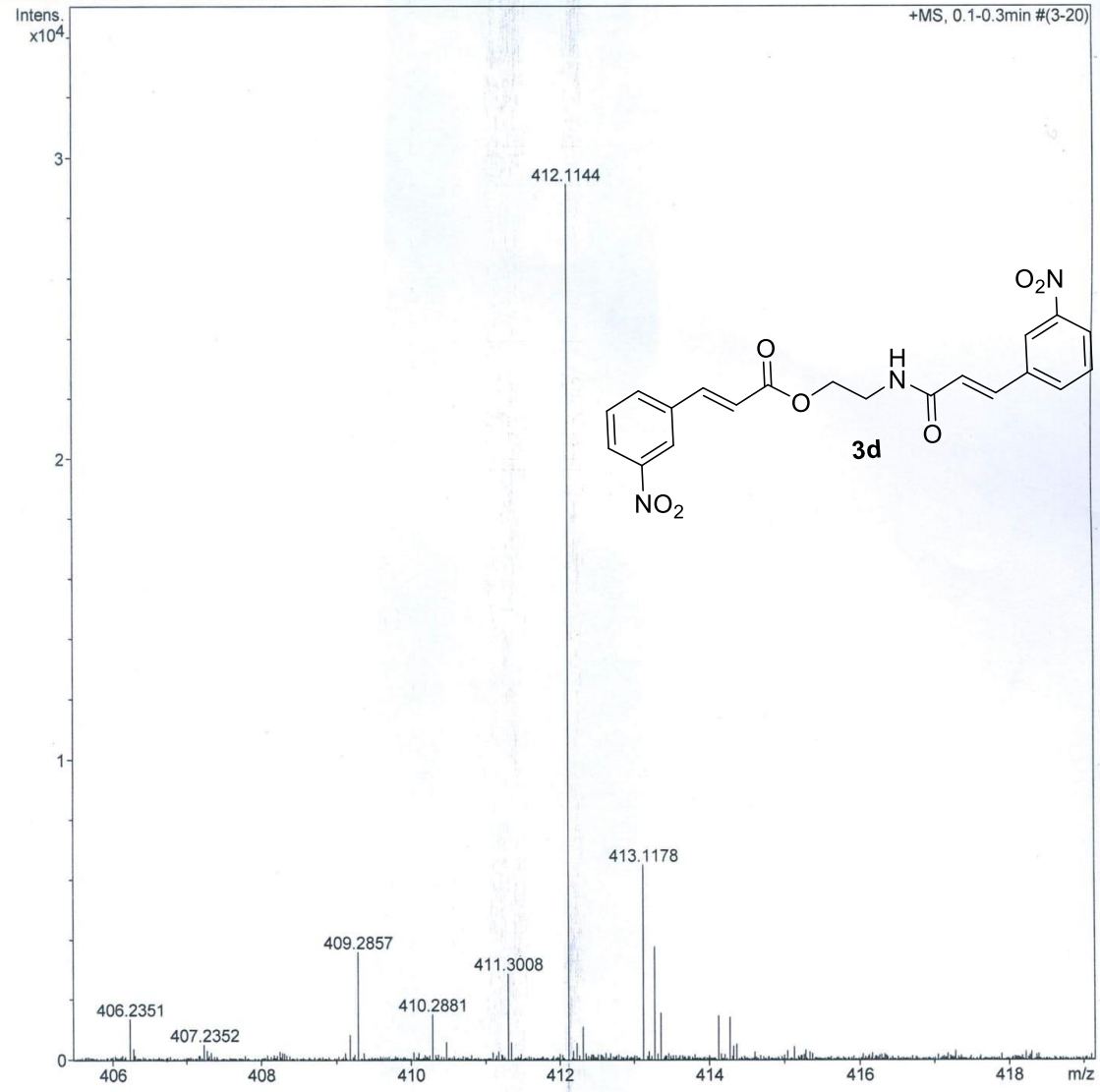
UOH -SCHOOL OF CHEMISTRY -HRMS

Analysis Info

Analysis Name	D:\Data\2018\PROF MP\DEC\1.d	Acquisition Date	12/11/2018 4:14:15 PM
Method	tune_low.m	Operator	UOH-Chemistry
Sample Name	1	Instrument	maXis 10138
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3800 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1800 m/z	Set Collision Cell RF	350.0 Vpp	Set Divert Valve	Waste



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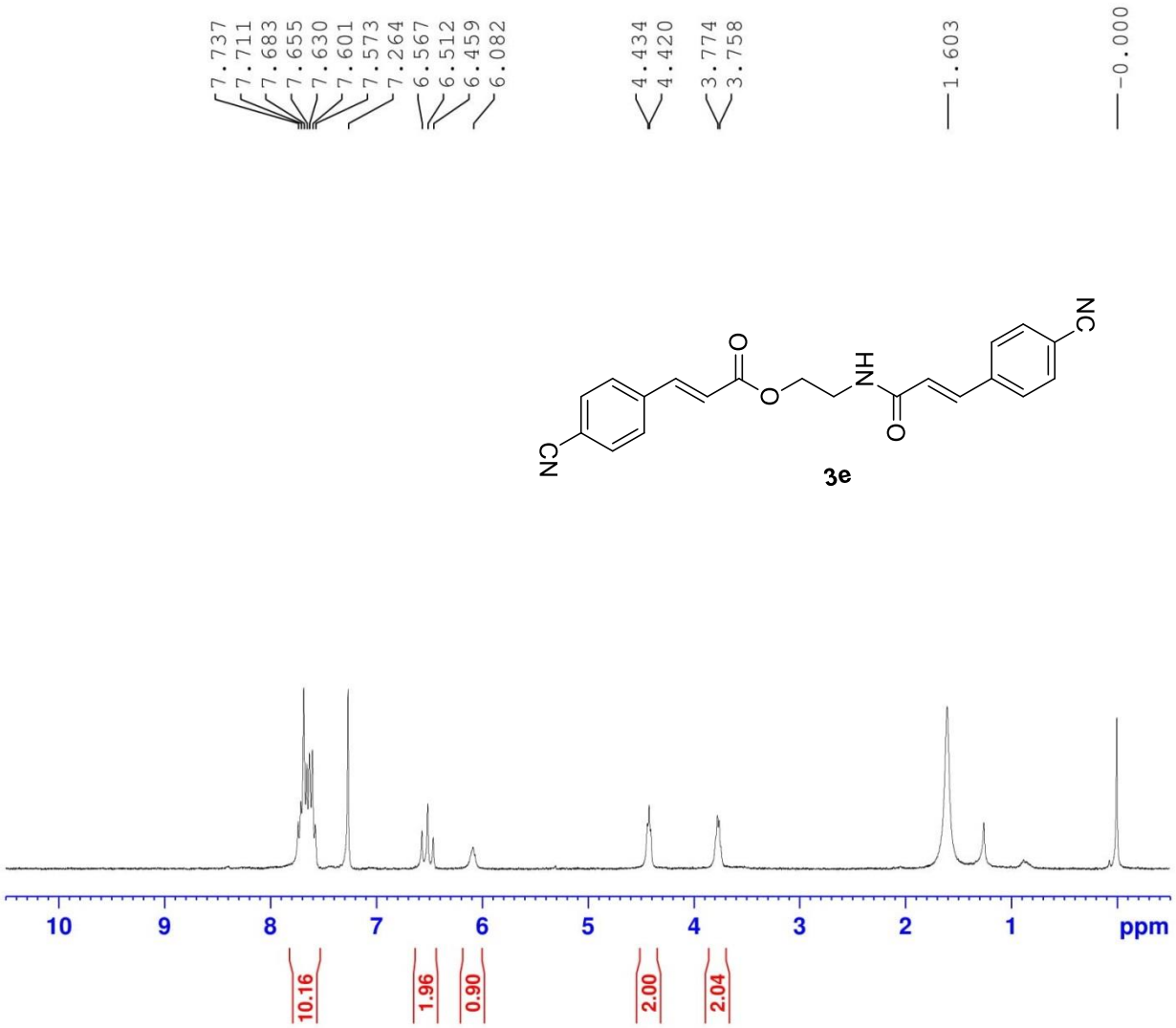
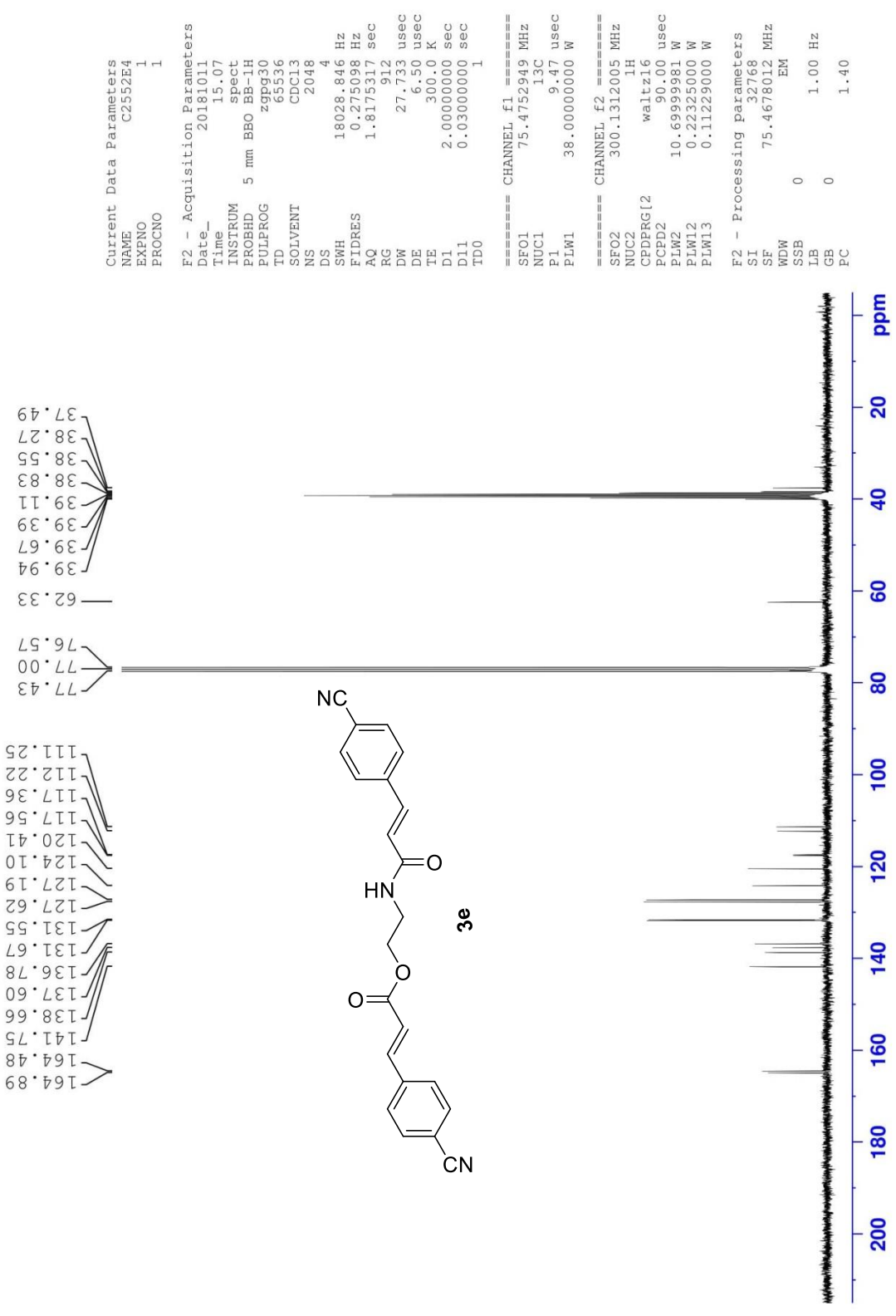



```
Current Data Parameters
NAME            H2552B16
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180731
Time               16.06
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4525952 sec
RG                   456
DW                83.200 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                   1H
P1                 13.00 usec
PLW1         10.69999981 W

F2 - Processing parameters
SI                 65536
SF           300.1300056 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```


Current Data Parameters
NAME C2552E4
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20181011
Time_ 15.07
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81175317 sec
RG 912
DW 27.733 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 75.4752949 MHz
NUC1 13C
P1 9.47 usec
PLW1 38.00000000 W
======== CHANNEL f2 ========
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 10.69999981 W
PLW12 0.22325000 W
PLW13 0.11229000 W
F2 - Processing parameters
SI 32768
SF 75.4678012 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
77.43
77.00
76.57
62.33
39.94
39.67
39.39
39.11
38.83
38.55
38.27
37.49
164.89
164.48
141.75
138.66
137.60
136.78
131.67
131.55
127.62
127.19
124.10
120.41
117.56
117.36
112.22
111.25
NC
O
HN
O
O
CN
3e
ppm
200
180
160
140
120
100
80
60
40
20

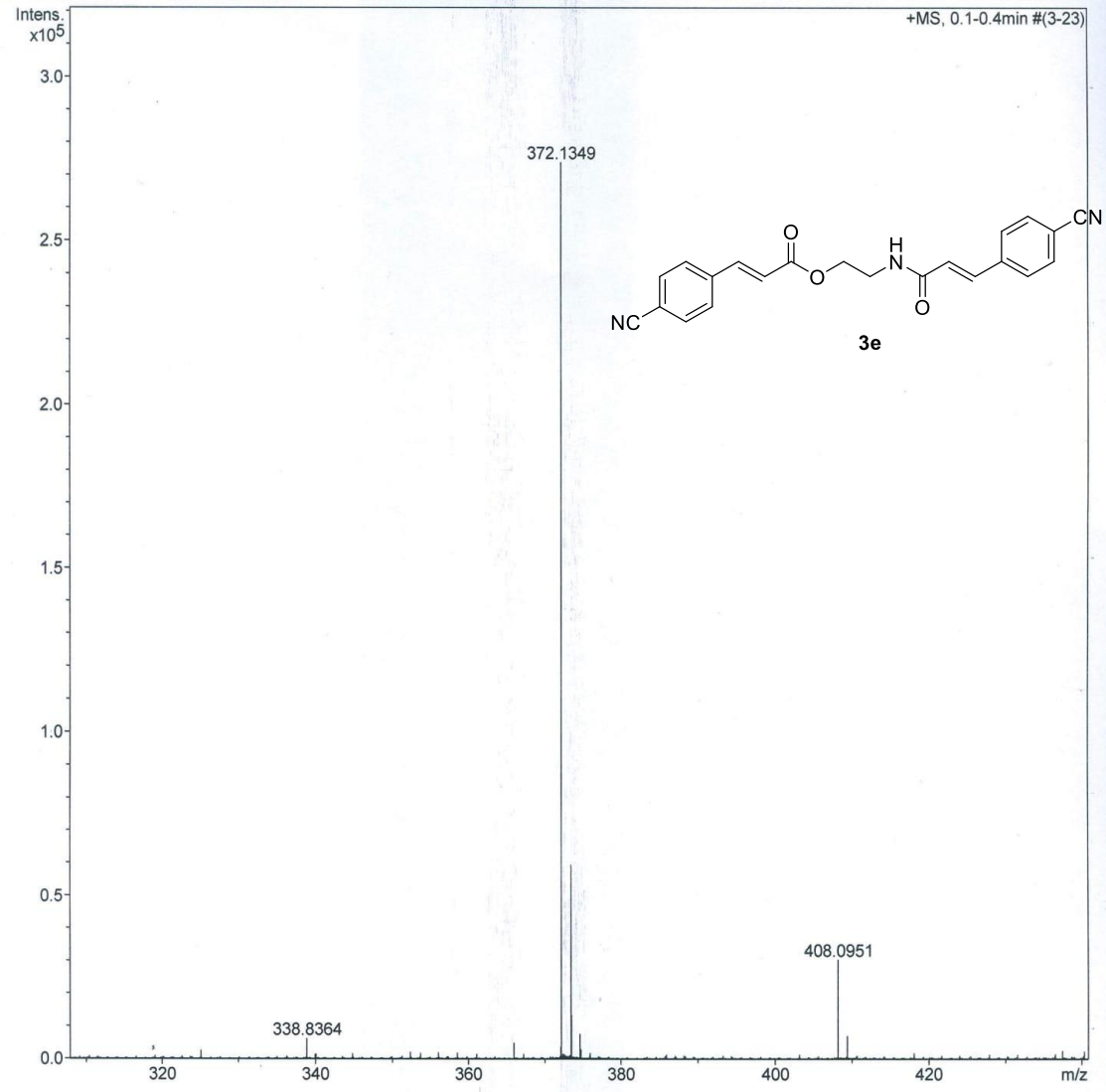
UOH -SCHOOL OF CHEMISTRY -HRMS

Analysis Info

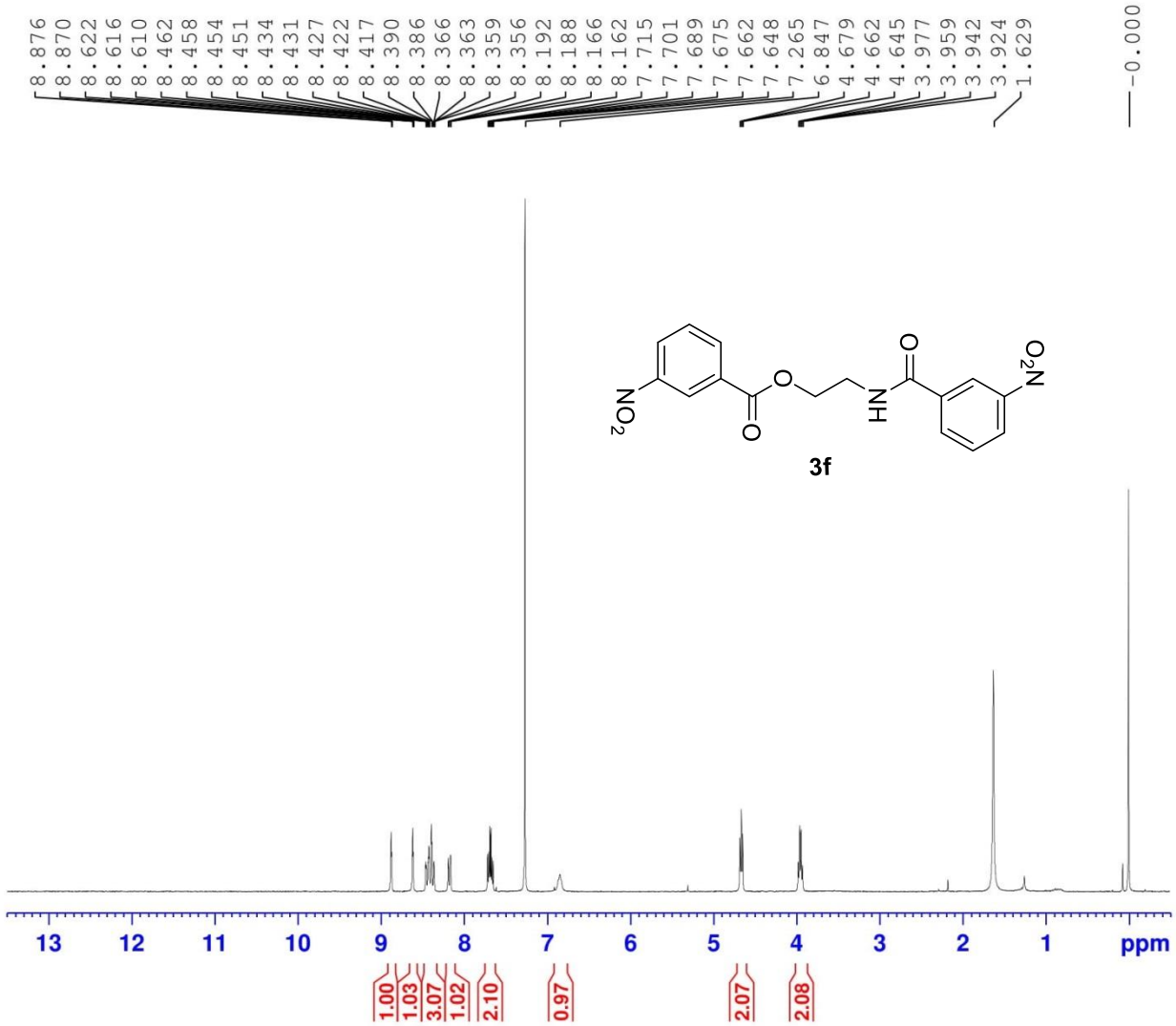
Analysis Name	D:\Data\2018\PROF MP\DEC\3.d	Acquisition Date	12/11/2018 4:18:23 PM
Method	tune_low.m	Operator	UOH-Chemistry
Sample Name	3	Instrument	maXis 10138
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3800 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1800 m/z	Set Collision Cell RF	350.0 Vpp	Set Divert Valve	Waste



3e

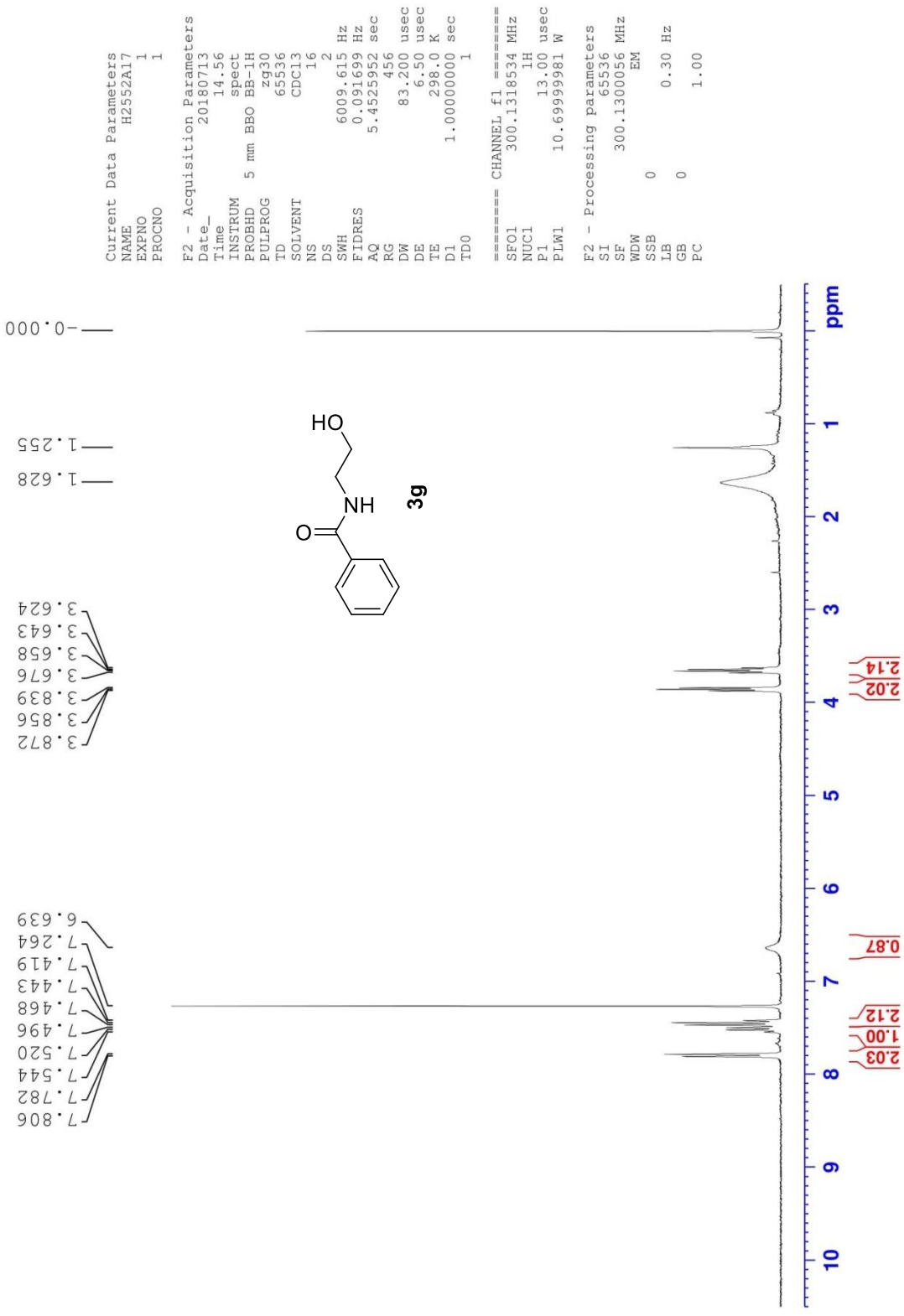


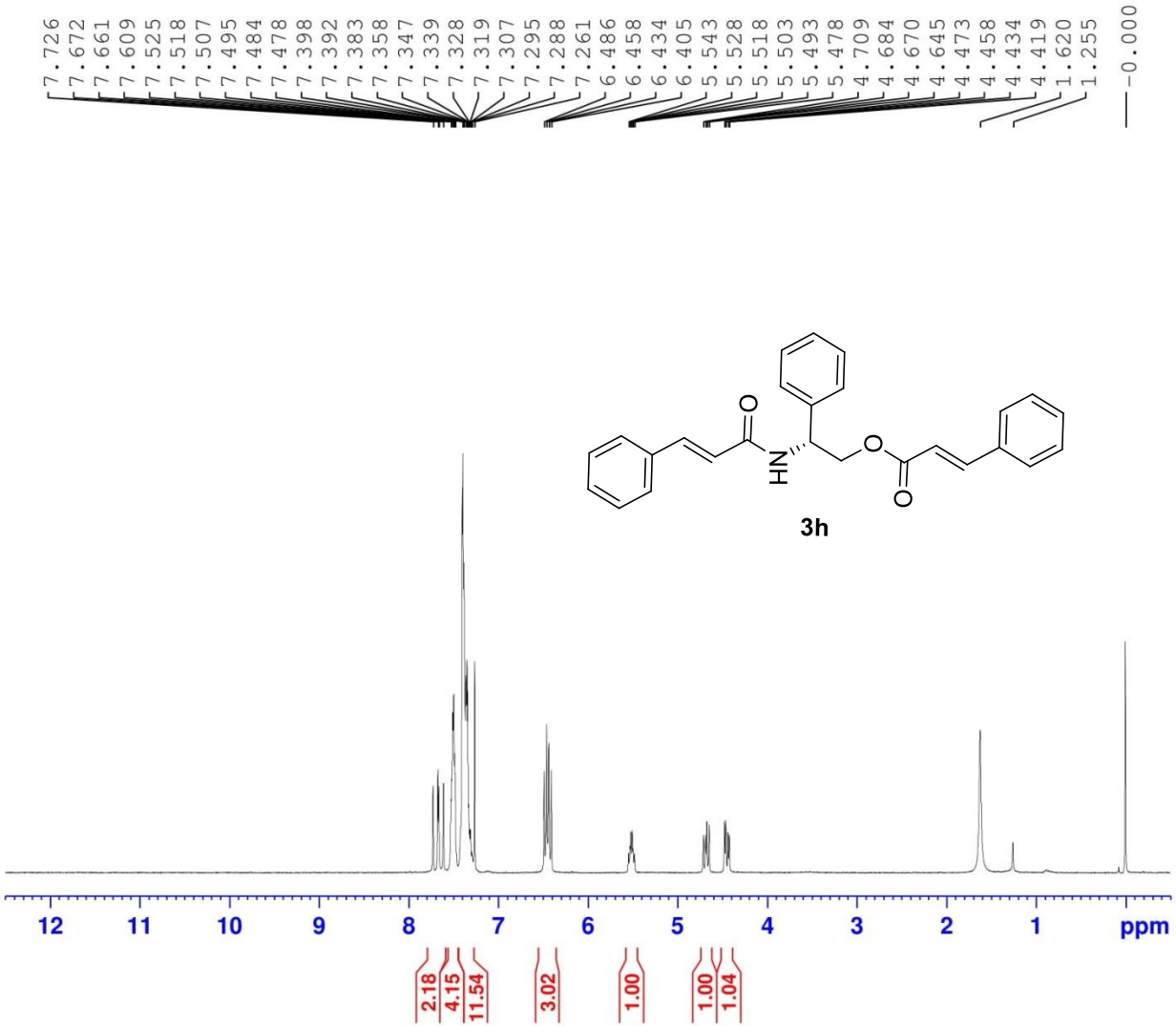

```
Current Data Parameters
NAME              H2552J8
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20190308
Time                16.52
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              6009.615 Hz
FIDRES           0.091699 Hz
AQ               5.4525952 sec
RG                    406
DW                 83.200 usec
DE                  6.50 usec
TE                 298.0 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                1H
P1                13.00 usec
PLW1         10.69999981 W

F2 - Processing parameters
SI                  65536
SF           300.1300051 MHz
WDW                    EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```


Current Data Parameters
NAME H2552A17
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180713
Time 14.56
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 456
DW 83.200 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 300.1318534 MHz
NUC1 1H
P1 13.00 usec
PLW1 10.69999981 W
F2 - Processing parameters
SI 65536
SF 300.1300056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
HO
NH
O
3g
-0.000
1.255
1.628
3.624
3.643
3.658
3.676
3.839
3.856
3.872
6.639
7.264
7.419
7.443
7.468
7.496
7.520
7.544
7.782
7.806
2.03
1.00
2.12
0.87
2.02
2.14
ppm

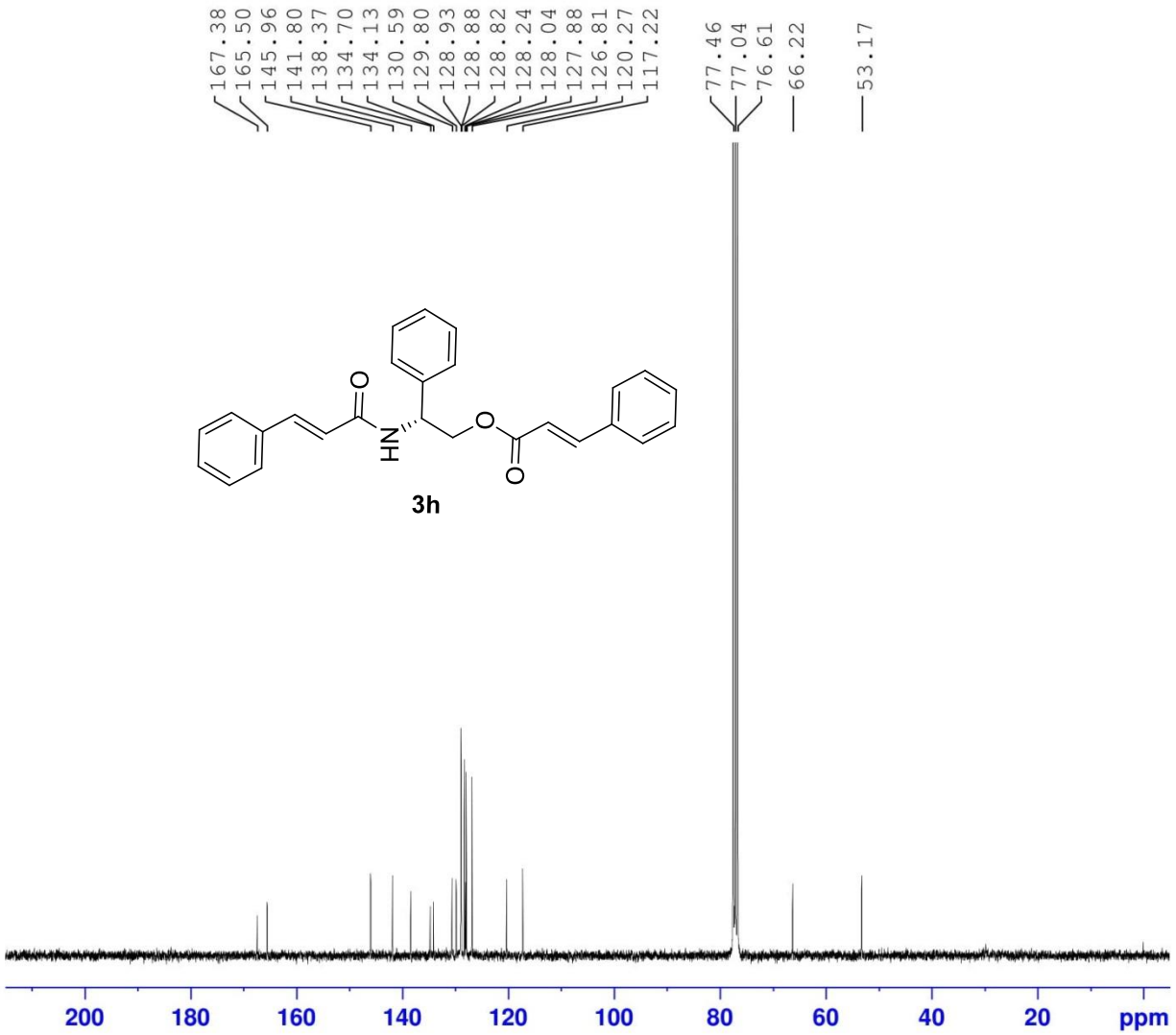



```
Current Data Parameters
NAME              H2552D2
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20180831
Time                16.58
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              6009.615 Hz
FIDRES           0.091699 Hz
AQ               5.4525952 sec
RG                    228
DW                 83.200 usec
DE                  6.50 usec
TE                 298.0 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          300.1318534 MHz
NUC1                1H
P1                 13.00 usec
PLW1          10.69999981 W

F2 - Processing parameters
SI                  65536
SF            300.1300063 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```




```
Current Data Parameters
NAME            C2552C10
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180904
Time               20.52
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3072
DS                     4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ            1.8175317 sec
RG                   812
DW                27.733 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         75.4752949 MHz
NUC1                13C
P1                 9.47 usec
PLW1        38.00000000 W

======== CHANNEL f2 ========
SFO2        300.1312005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.69999981 W
PLW12        0.22325000 W
PLW13        0.11229000 W

F2 - Processing parameters
SI                 32768
SF           75.4677485 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

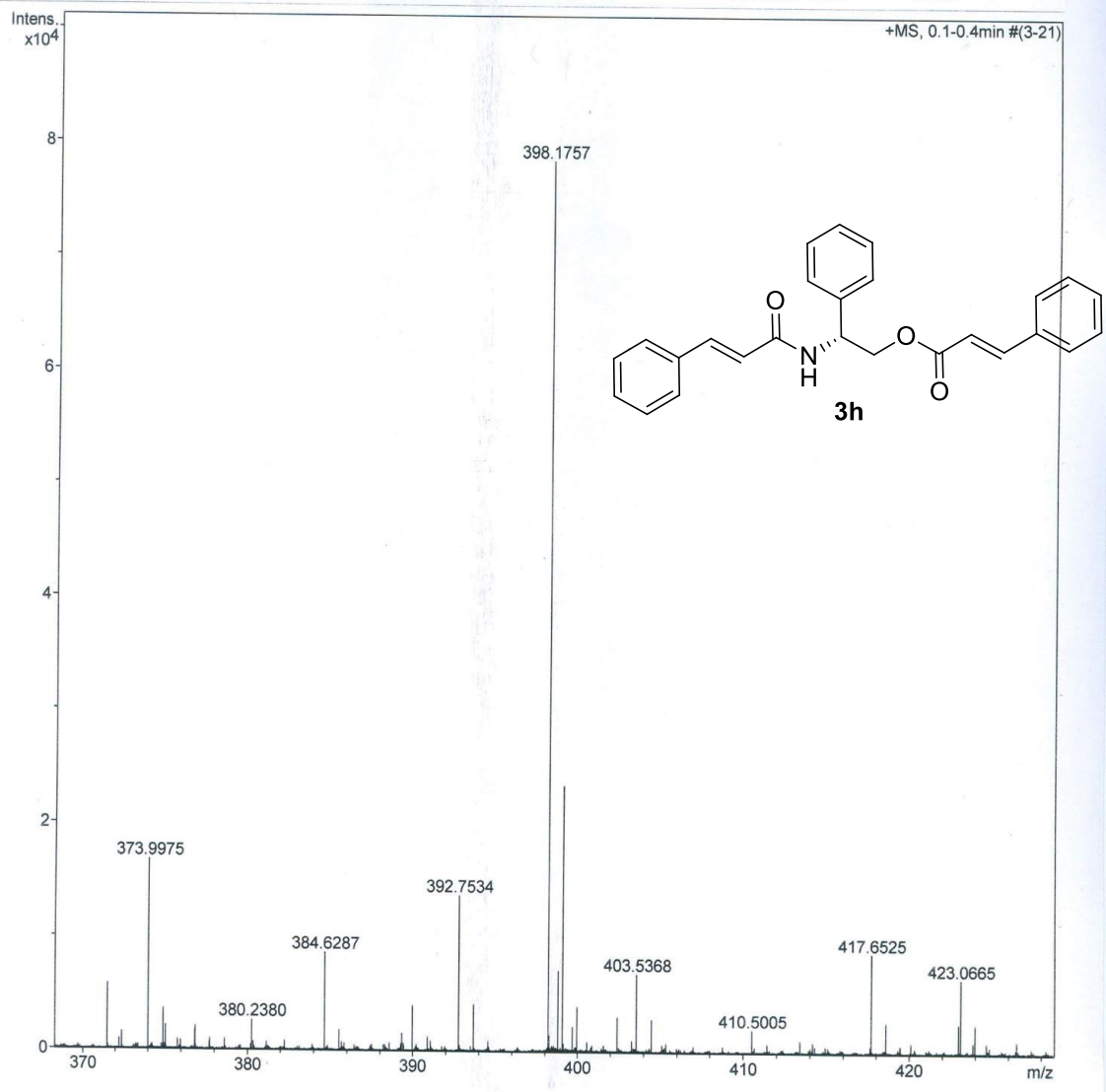
UOH -SCHOOL OF CHEMISTRY -HRMS

Analysis Info

Analysis Name	D:\Data\2018\PROF MP\DEC\13.d	Acquisition Date	12/11/2018 4:45:59 PM
Method	tune_low.m		
Sample Name	13	Operator	UOH-Chemistry
Comment		Instrument	maXis 10138

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3800 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1800 m/z	Set Collision Cell RF	350.0 Vpp	Set Divert Valve	Waste



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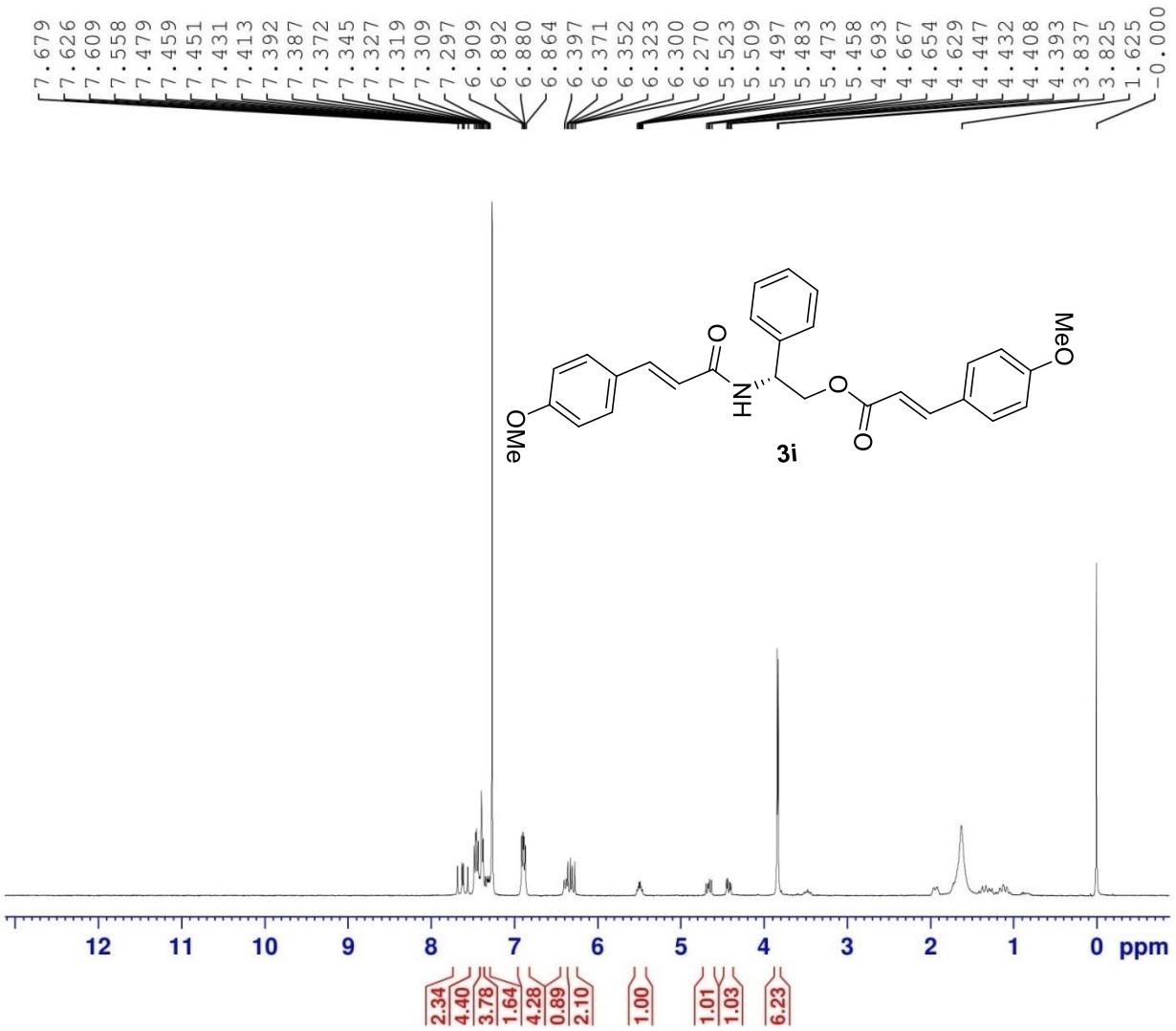
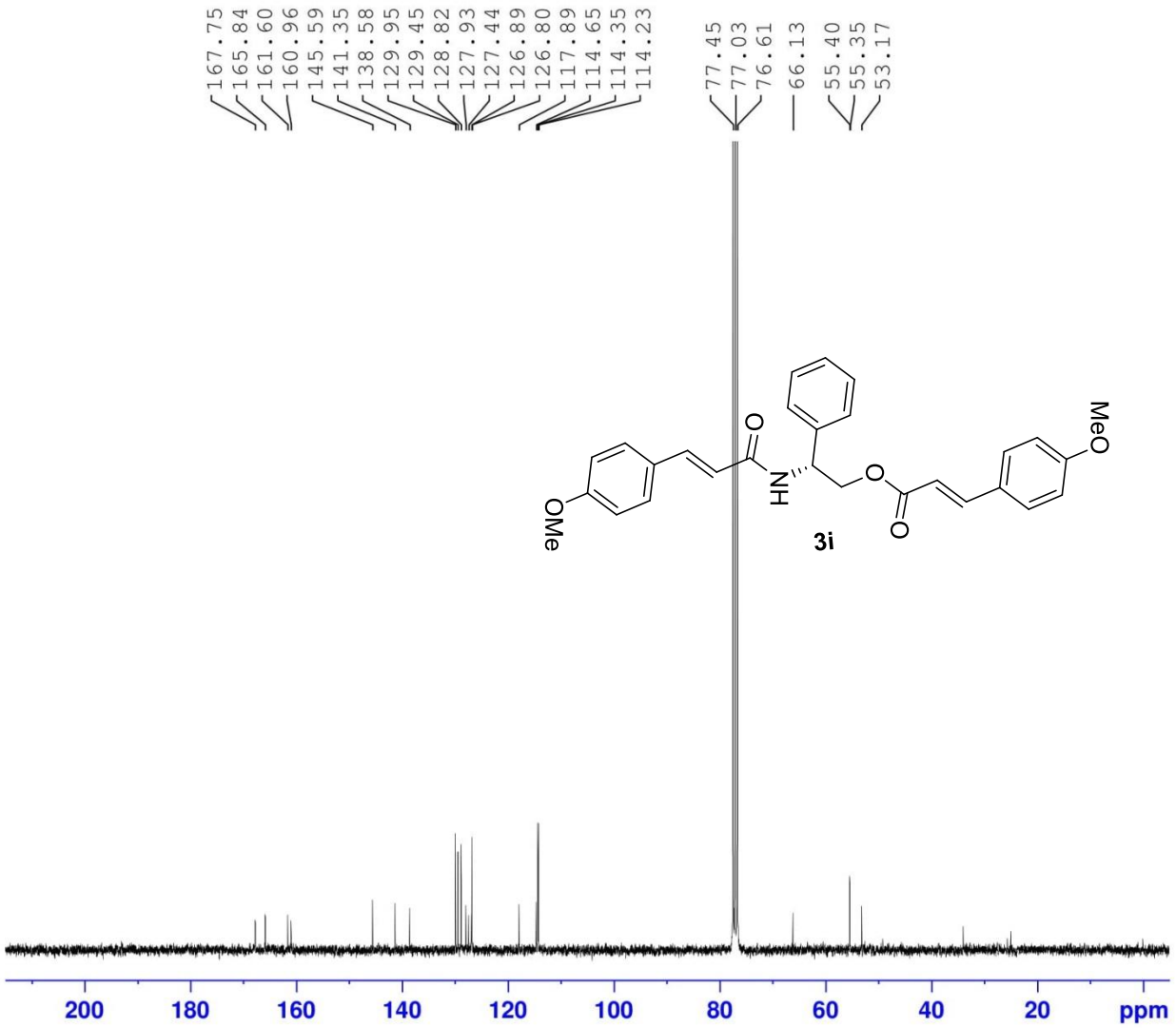



```
Current Data Parameters
NAME            H2552J17
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190314
Time               11.18
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4525952 sec
RG                   362
DW                83.200 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                1H
P1                13.00 usec
PLW1         10.69999981 W

F2 - Processing parameters
SI                 65536
SF           300.1300059 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME              C2552C9
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20180904
Time                13.24
INSTRUM             spect
PROBHD    5 mm BBO BB-1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  2048
DS                     4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ            1.8175317 sec
RG                  1030
DW                27.733 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                  13C
P1                   9.47 usec
PLW1        38.00000000 W

======== CHANNEL f2 ========
SFO2         300.1312005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2         10.69999981 W
PLW12         0.22325000 W
PLW13         0.11229000 W

F2 - Processing parameters
SI                 32768
SF            75.4677485 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

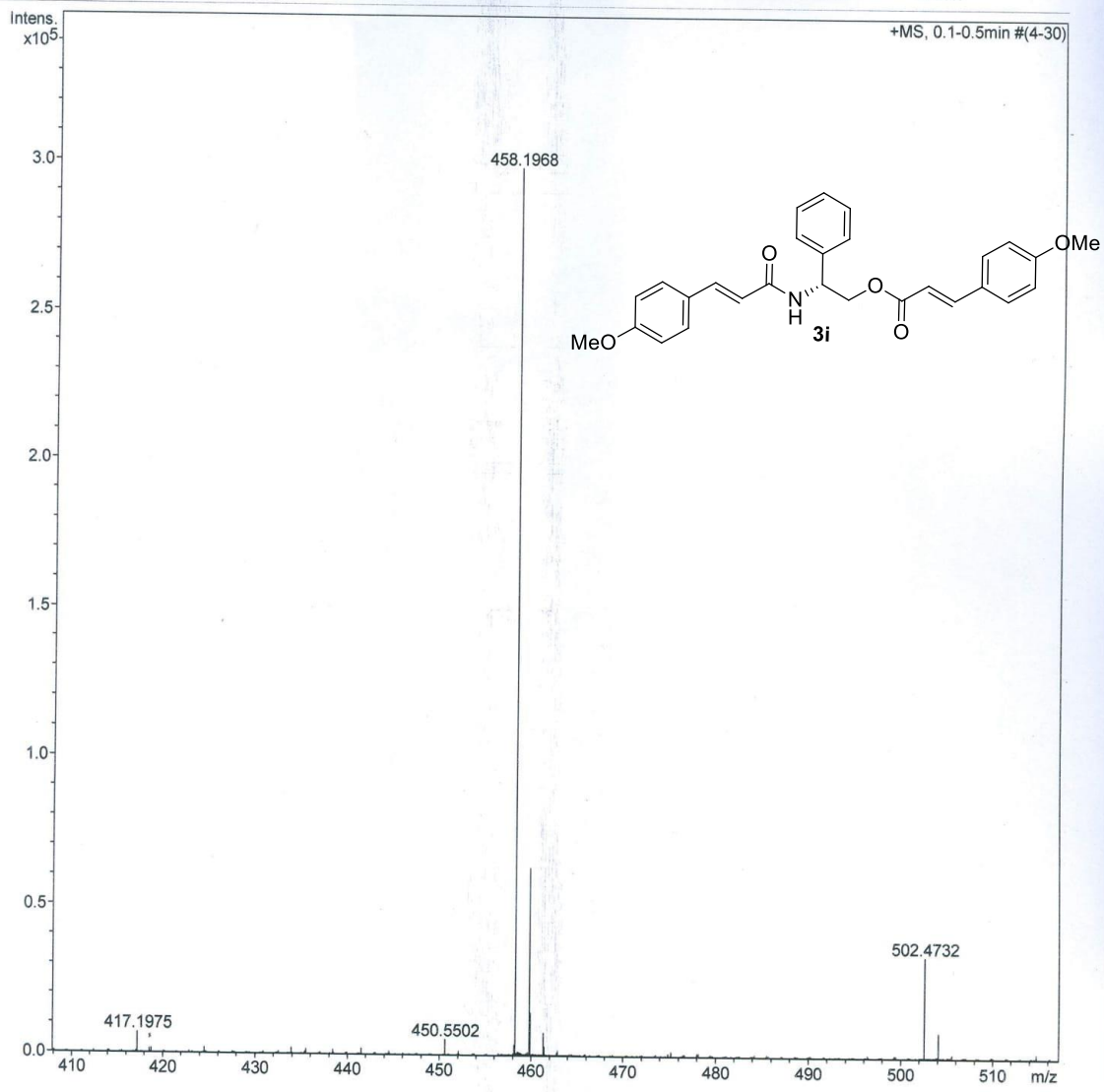
UOH -SCHOOL OF CHEMISTRY -HRMS

Analysis Info

Analysis Name	D:\Data\2018\PROF MP\DEC\7.d	Acquisition Date	12/11/2018 4:30:41 PM
Method	tune_low.m		
Sample Name	7	Operator	UOH-Chemistry
Comment		Instrument	maXis 10138

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3800 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1800 m/z	Set Collision Cell RF	350.0 Vpp	Set Divert Valve	Waste



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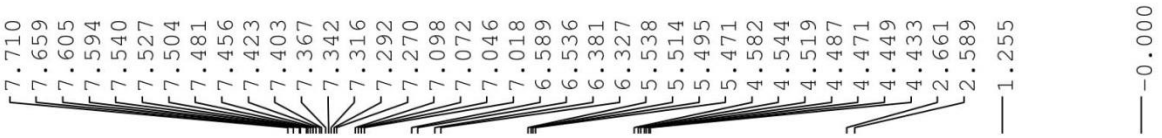
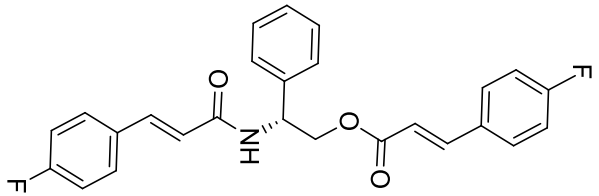
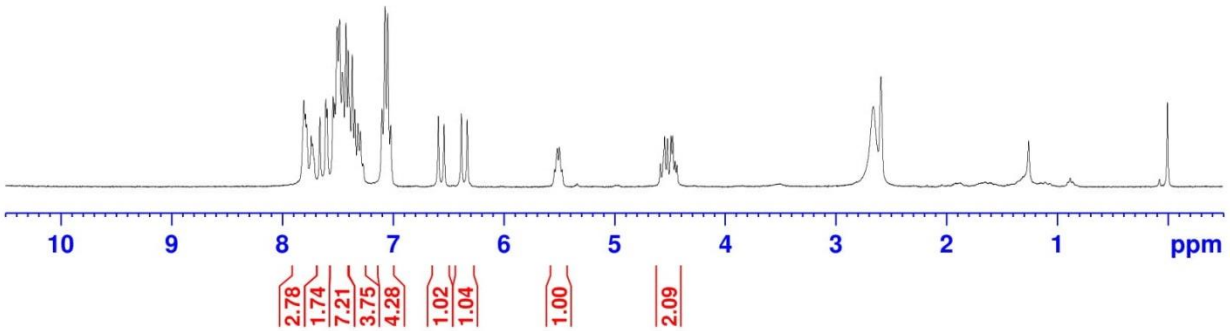




3j

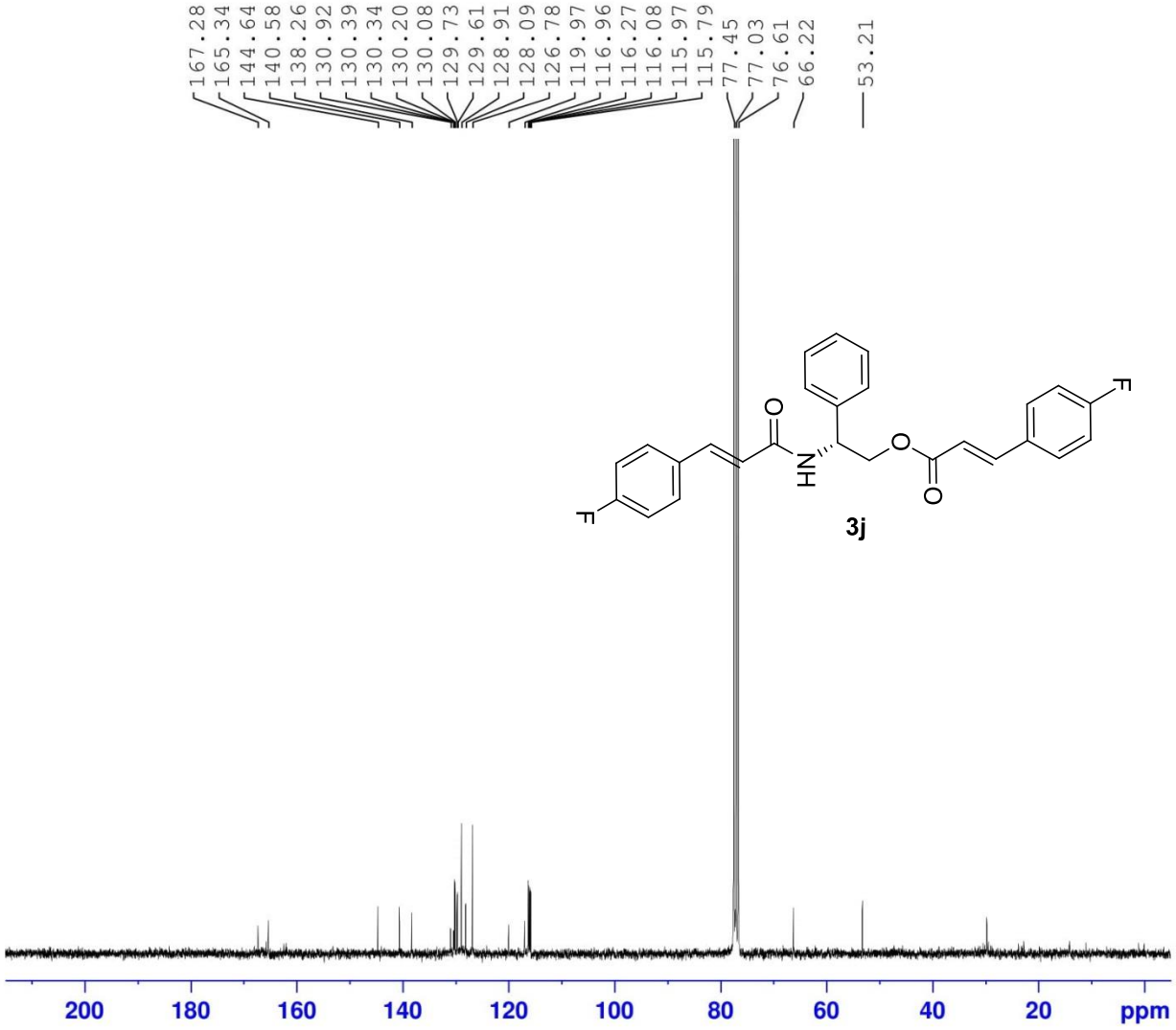



```
Current Data Parameters
NAME              H2552D7
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20180904
Time                10.50
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              6009.615 Hz
FIDRES           0.091699 Hz
AQ               5.4525952 sec
RG                    203
DW                 83.200 usec
DE                  6.50 usec
TE                 298.0 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          300.1325511 MHz
NUC1                1H
P1                 13.00 usec
PLW1          10.69999981 W

F2 - Processing parameters
SI                  65536
SF            300.1299641 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```




```
Current Data Parameters
NAME             C2552D8
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180920
Time               20.41
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3072
DS                     4
SWH            18028.846 Hz
FIDRES          0.275098 Hz
AQ             1.8175317 sec
RG                   812
DW                27.733 usec
DE                 6.50 usec
TE                300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                  13C
P1                   9.47 usec
PLW1         38.00000000 W

======== CHANNEL f2 ========
SFO2         300.1312005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2         10.69999981 W
PLW12         0.22325000 W
PLW13         0.11229000 W

F2 - Processing parameters
SI                 32768
SF            75.4677485 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

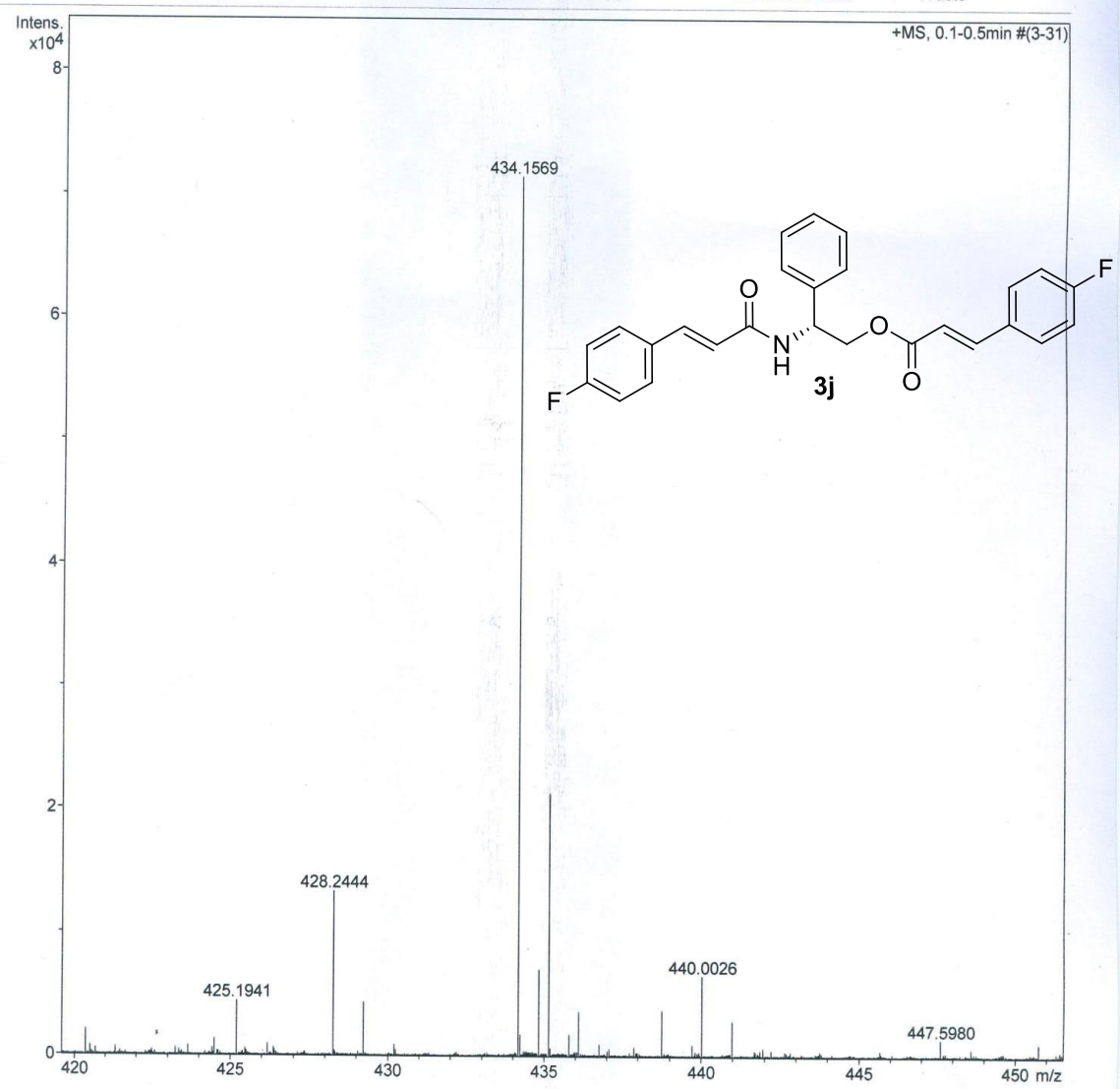
UOH -SCHOOL OF CHEMISTRY -HRMS

Analysis Info

Analysis Name	D:\Data\2018\PROF MP\DEC\6.d	Acquisition Date	12/11/2018 4:25:57 PM
Method	tune_low.m	Operator	UOH-Chemistry
Sample Name	6	Instrument	maXis 10138
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3800 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1800 m/z	Set Collision Cell RF	350.0 Vpp	Set Divert Valve	Waste



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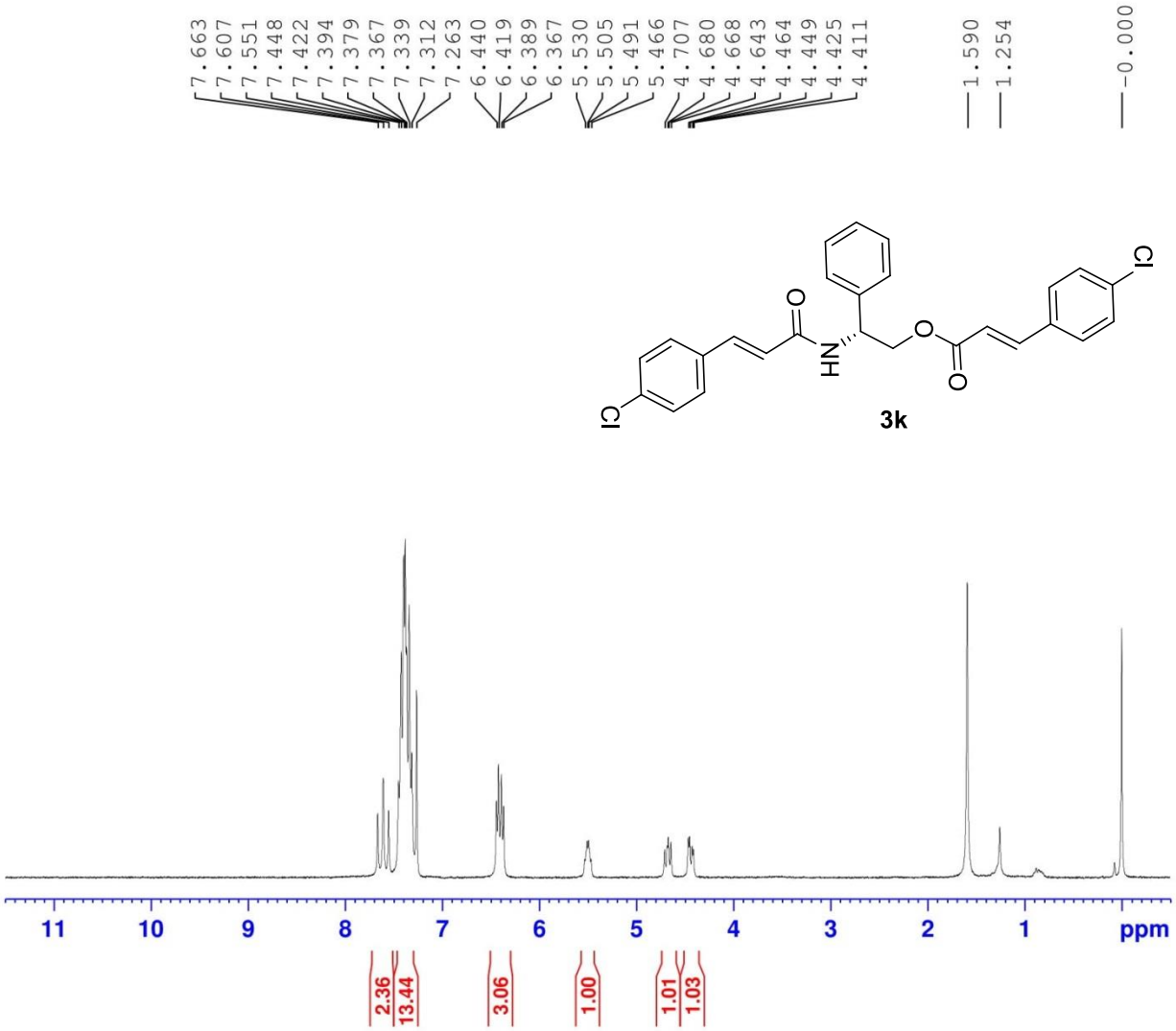



```
Current Data Parameters
NAME            H2552D12
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180910
Time               17.12
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4525952 sec
RG                   406
DW                83.200 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          300.1318534 MHz
NUC1                  1H
P1                 13.00 usec
PLW1          10.69999981 W

F2 - Processing parameters
SI                 65536
SF            300.1300065 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```


```
Current Data Parameters
NAME            C2552D6
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20180911
Time              15.05
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2048
DS                    4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ            1.8175317 sec
RG                  812
DW               27.733 usec
DE                6.50 usec
TE               300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         75.4752949 MHz
NUC1                13C
P1                 9.47 usec
PLW1        38.00000000 W

======== CHANNEL f2 ========
SFO2        300.1312005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.69999981 W
PLW12        0.22325000 W
PLW13        0.11229000 W

F2 - Processing parameters
SI                32768
SF           75.4677485 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

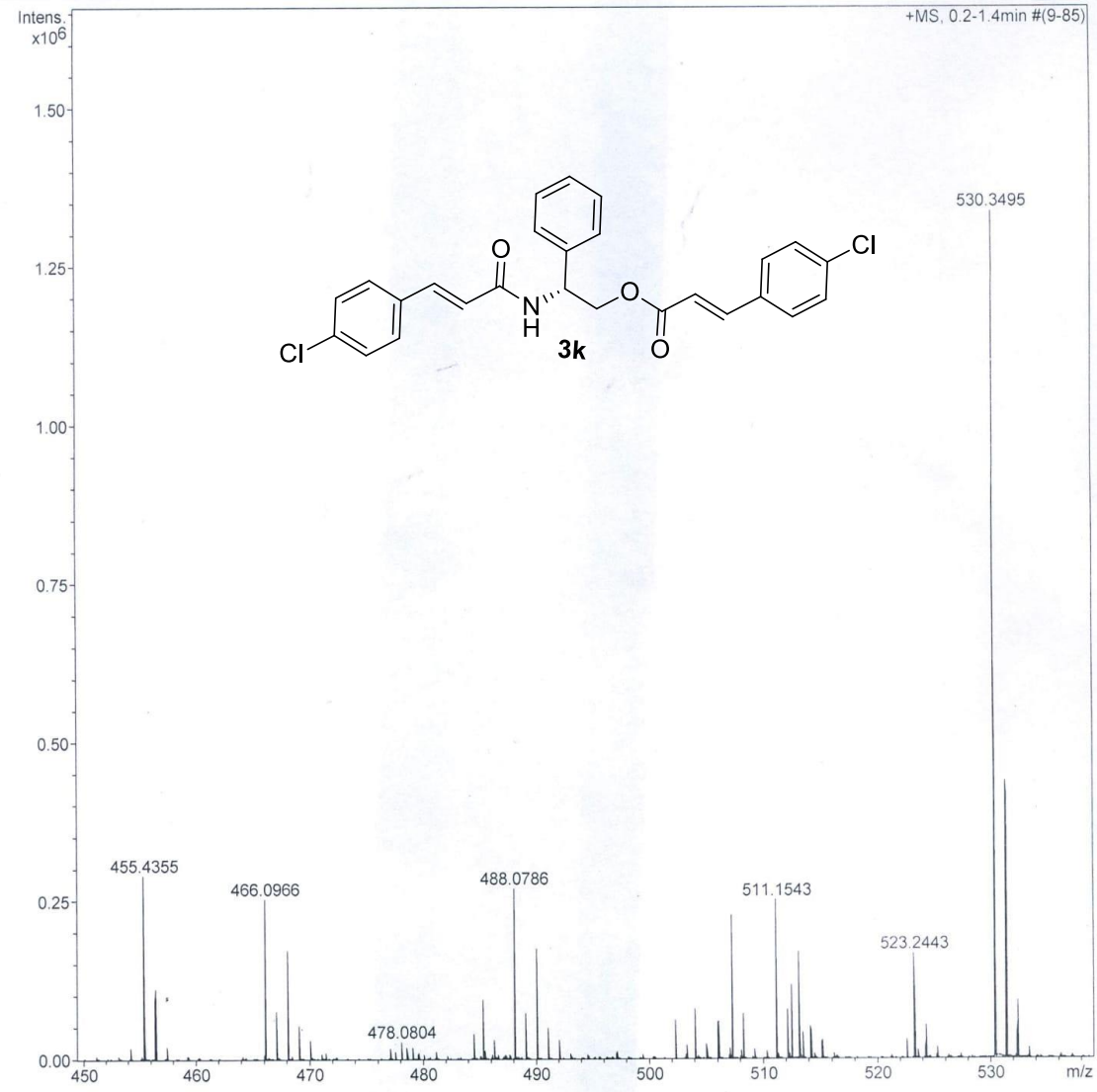
UOH -SCHOOL OF CHEMISTRY -HRMS

Analysis Info

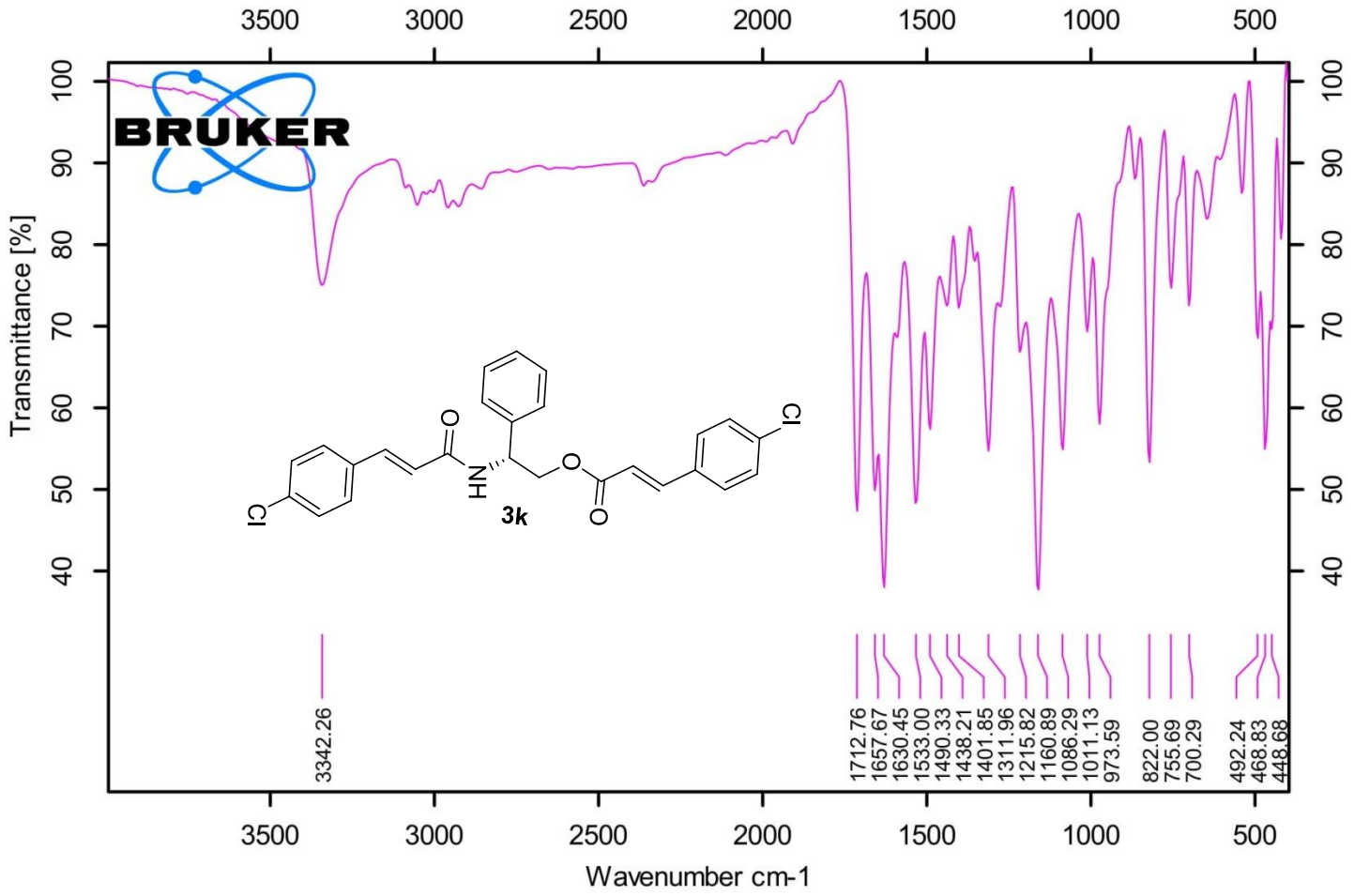
Analysis Name	D:\Data\2018\PROF MP\DEC\4.d	Acquisition Date	12/12/2018 2:07:41 PM
Method	tune_low_PosR.m	Operator	UOH-Chemistry
Sample Name	4--DCM-MEOH	Instrument	maXis 10138
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active	Set Capillary	3800 V	Set Dry Heater	250 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	5.0 l/min
Scan End	2500 m/z	Set Collision Cell RF	350.0 Vpp	Set Divert Valve	Waste

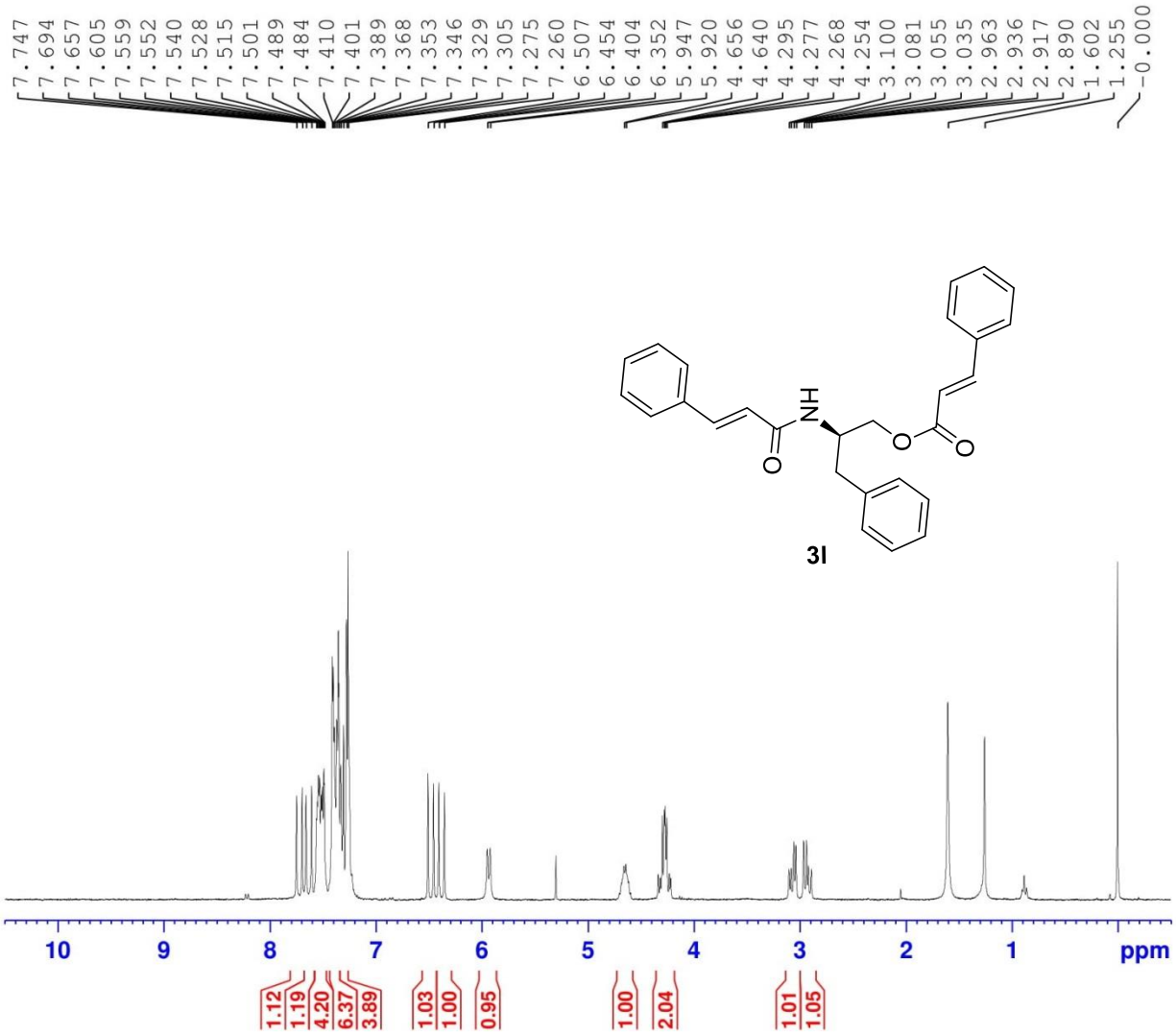


Bruker Compass DataAnalysis 4.0 printed: 1/8/2019 3:46:07 PM Page 1 of 1



SAMPLE SCANS : 48
DATE : 11-03-2019
TIME : 18:47:03

SAMPLE : PVS 2
TECHNIQUE : Thinfilm
USER : Default

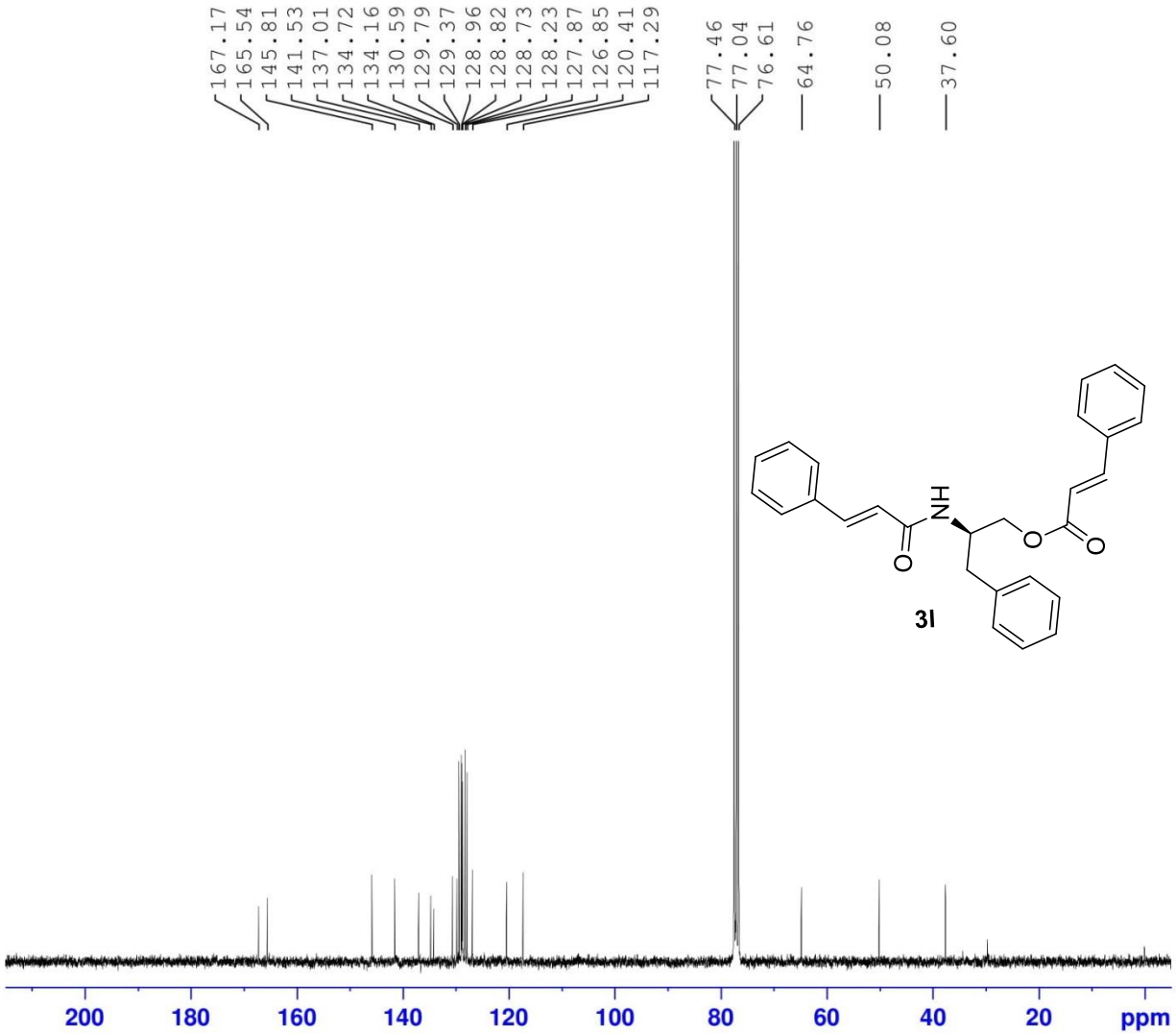



```
Current Data Parameters
NAME            H2552C17
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180821
Time               16.35
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4525952 sec
RG                   362
DW                83.200 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          300.1318534 MHz
NUC1                  1H
P1                 13.00 usec
PLW1          10.69999981 W

F2 - Processing parameters
SI                 65536
SF            300.1300067 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME            C2552C4
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20180822
Time              14.48
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2048
DS                    4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ            1.8175317 sec
RG                  912
DW               27.733 usec
DE                6.50 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         75.4752949 MHz
NUC1                13C
P1                 9.47 usec
PLW1        38.00000000 W

======== CHANNEL f2 ========
SFO2        300.1312005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.69999981 W
PLW12        0.22325000 W
PLW13        0.11229000 W

F2 - Processing parameters
SI                32768
SF           75.4677485 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

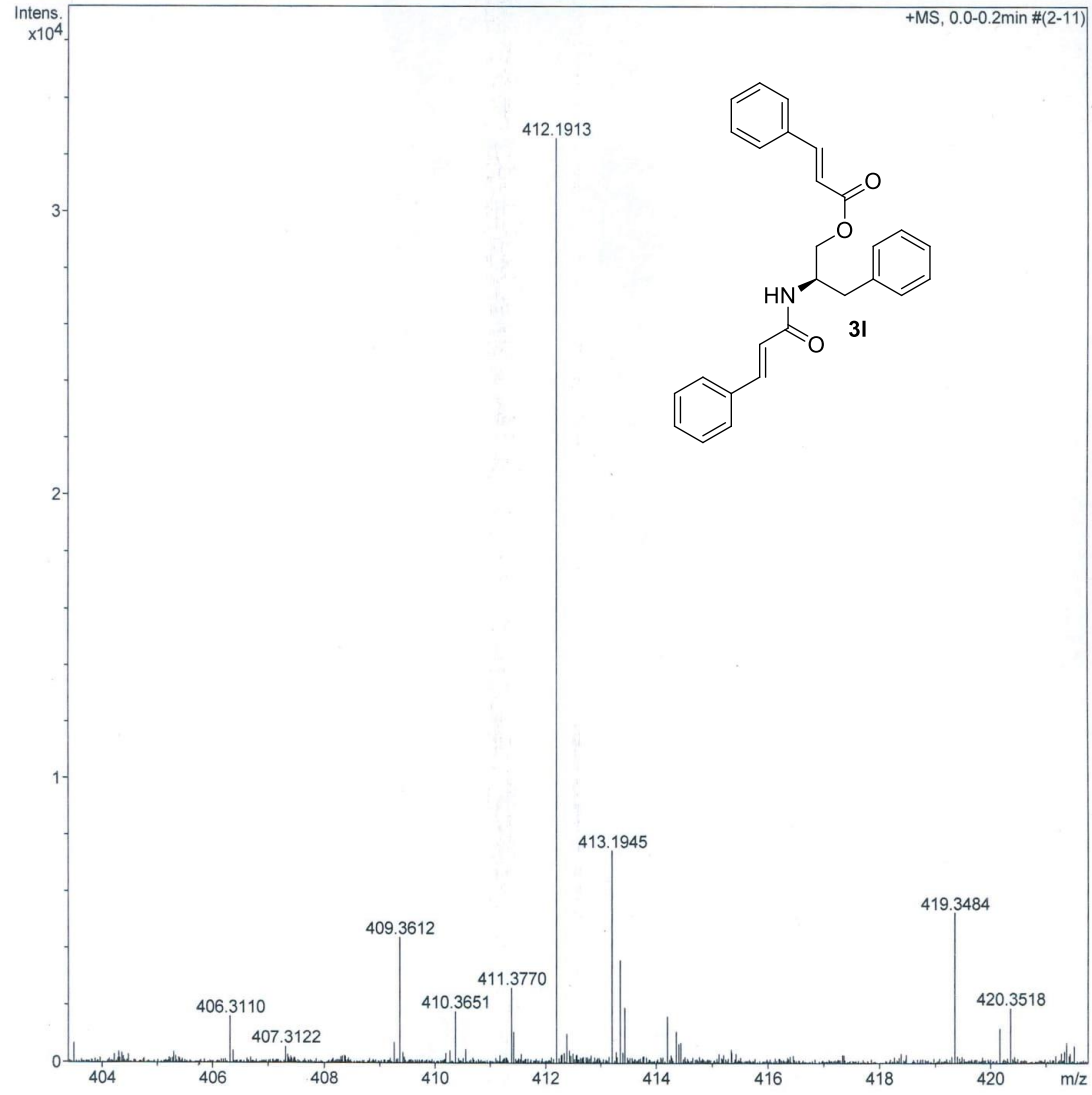
UOH -SCHOOL OF CHEMISTRY -HRMS

Analysis Info

Analysis Name	D:\Data\2018\PROF MP\DEC\14.d	Acquisition Date	12/11/2018 4:58:43 PM
Method	tune_low.m	Operator	UOH-Chemistry
Sample Name	14	Instrument	maXis 10138
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3800 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1800 m/z	Set Collision Cell RF	350.0 Vpp	Set Divert Valve	Waste



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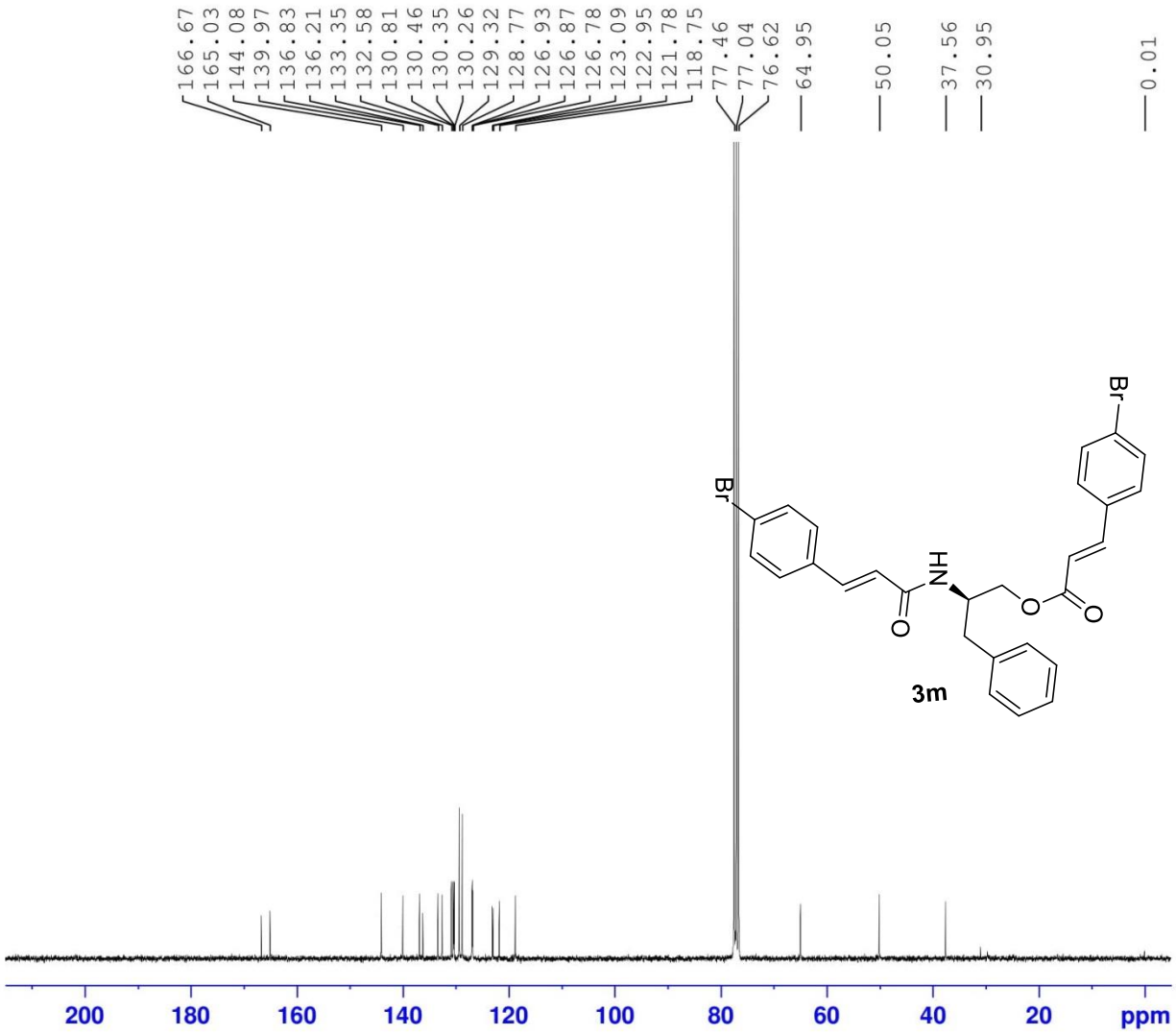
Current Data Parameters
NAME H2552E16
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20181008
Time 17.27
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 181
DW 83.200 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 300.1318534 MHz
NUC1 1H
P1 13.00 usec
PLW1 10.69999981 W
F2 - Processing parameters
SI 65536
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Br
O
O
HN
O
Br
3m
ppm
1.01
1.21
2.17
1.00
0.95
2.06
2.90
4.32
2.10
1.68
1.20
2.77
7.647
7.636
7.595
7.569
7.532
7.516
7.474
7.452
7.429
7.396
7.370
7.328
7.306
7.294
7.265
7.251
7.225
7.199
6.488
6.435
6.389
6.337
6.000
5.973
4.655
4.640
4.331
4.293
4.274
4.259
4.235
4.220
3.081
3.061
3.036
3.016
2.959
2.932
2.914
2.887
1.672
0.000




```
Current Data Parameters
NAME             C2552E2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181009
Time               20.43
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3072
DS                    4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ            1.8175317 sec
RG                  912
DW               27.733 usec
DE                6.50 usec
TE               300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                 13C
P1                 9.47 usec
PLW1        38.00000000 W

======== CHANNEL f2 ========
SFO2         300.1312005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         10.69999981 W
PLW12         0.22325000 W
PLW13         0.11229000 W

F2 - Processing parameters
SI                 32768
SF            75.4677485 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

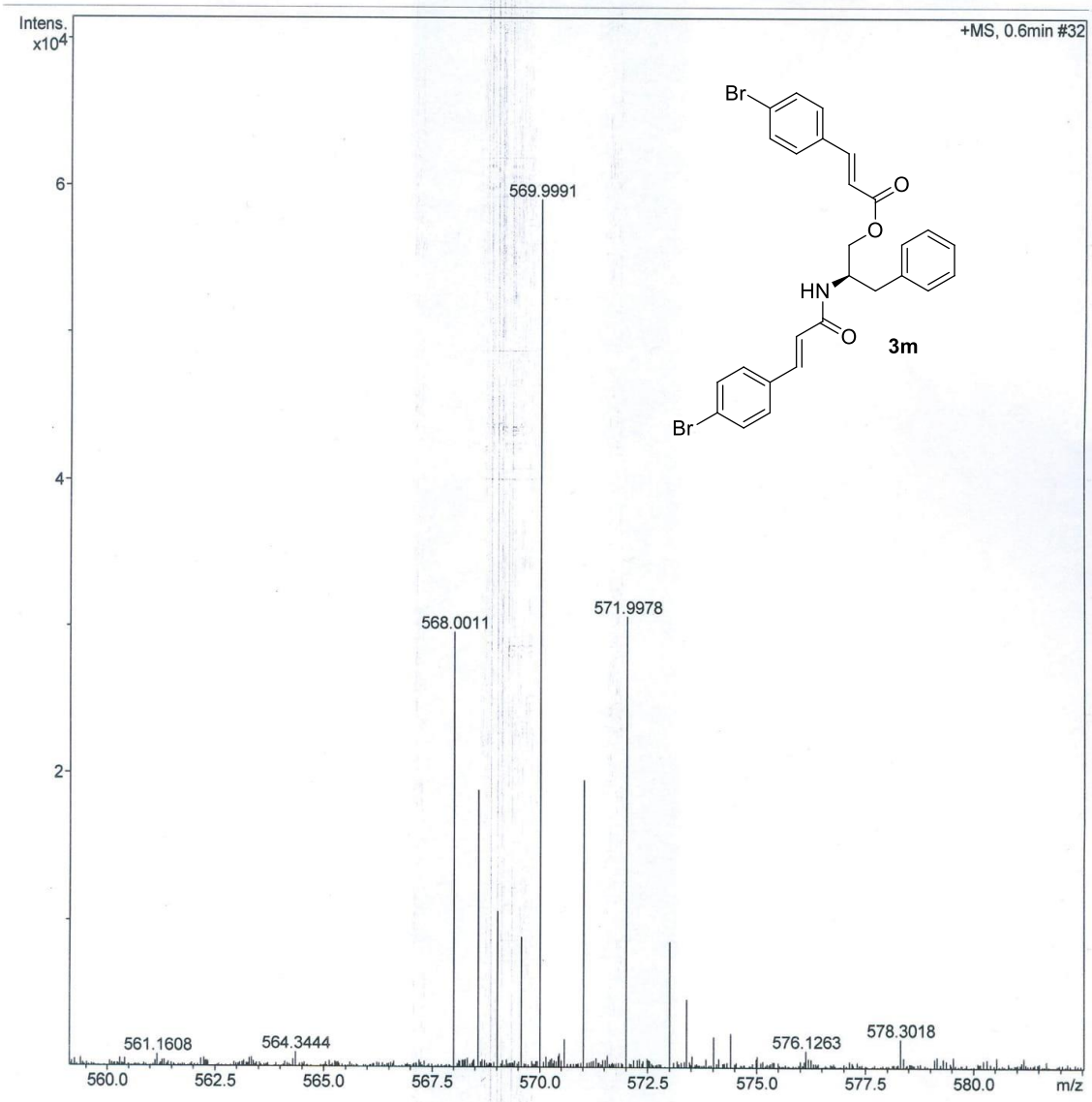
UOH -SCHOOL OF CHEMISTRY -HRMS

Analysis Info

Analysis Name	D:\Data\2018\PROF MP\DEC\5.d	Acquisition Date	12/11/2018 3:33:01 PM
Method	tune_low.m	Operator	UOH-Chemistry
Sample Name	5	Instrument	maXis 10138
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3800 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1800 m/z	Set Collision Cell RF	350.0 Vpp	Set Divert Valve	Waste



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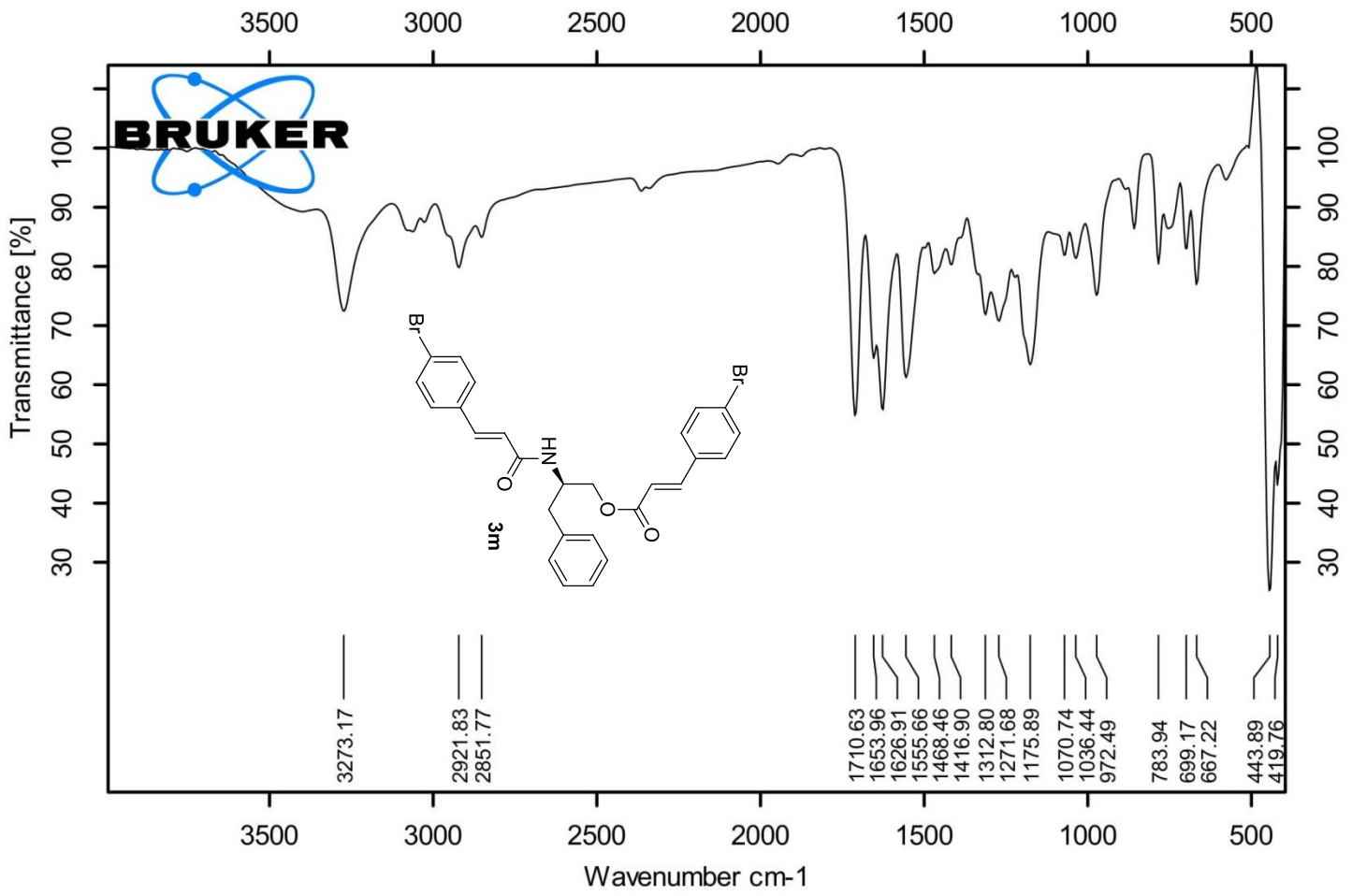
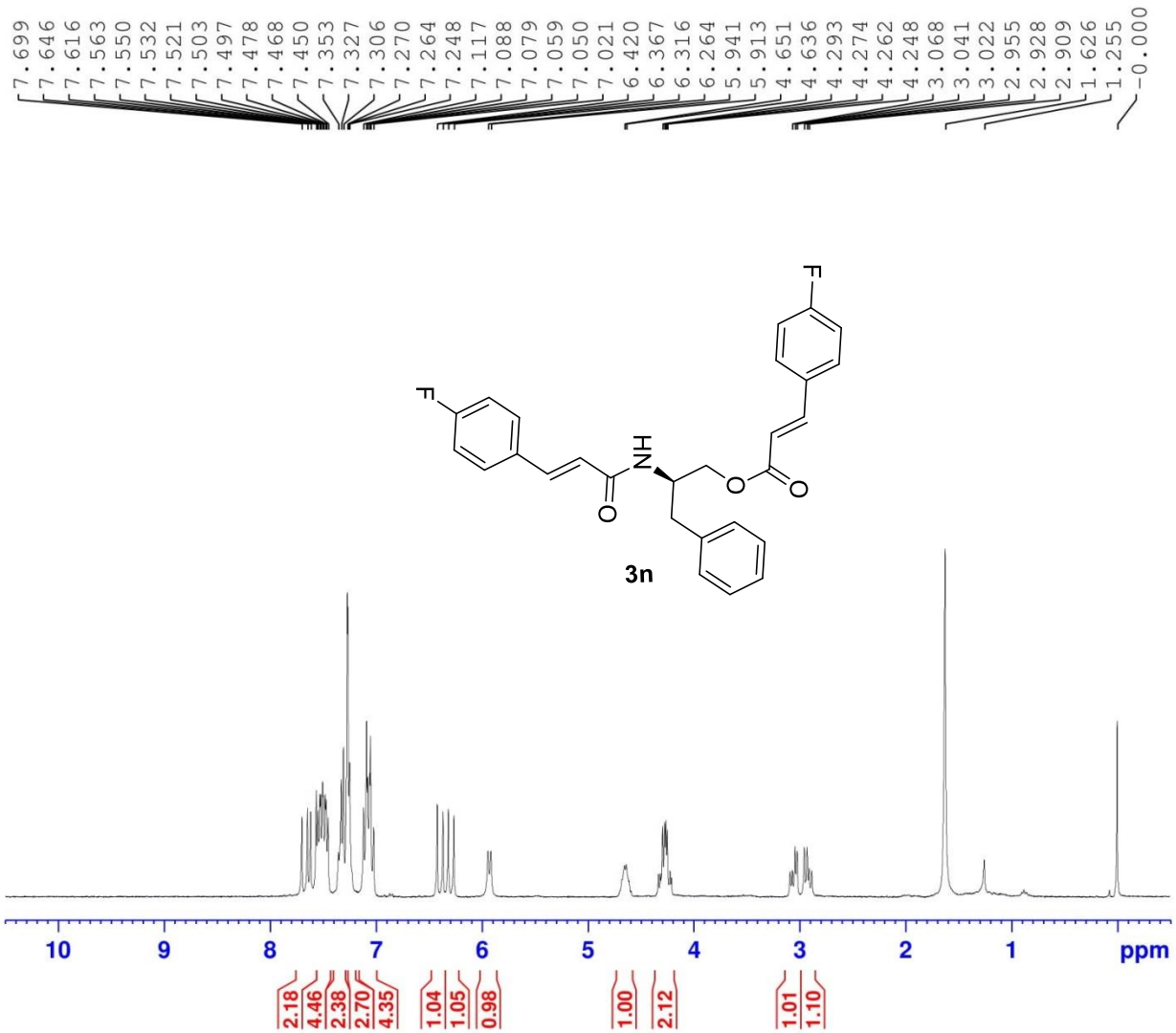


SAMPLE SCANS : 48
DATE : 11-03-2019
TIME : 15:35:54

SAMPLE : PVS 6
TECHNIQUE : Thinfilm
USER : Default

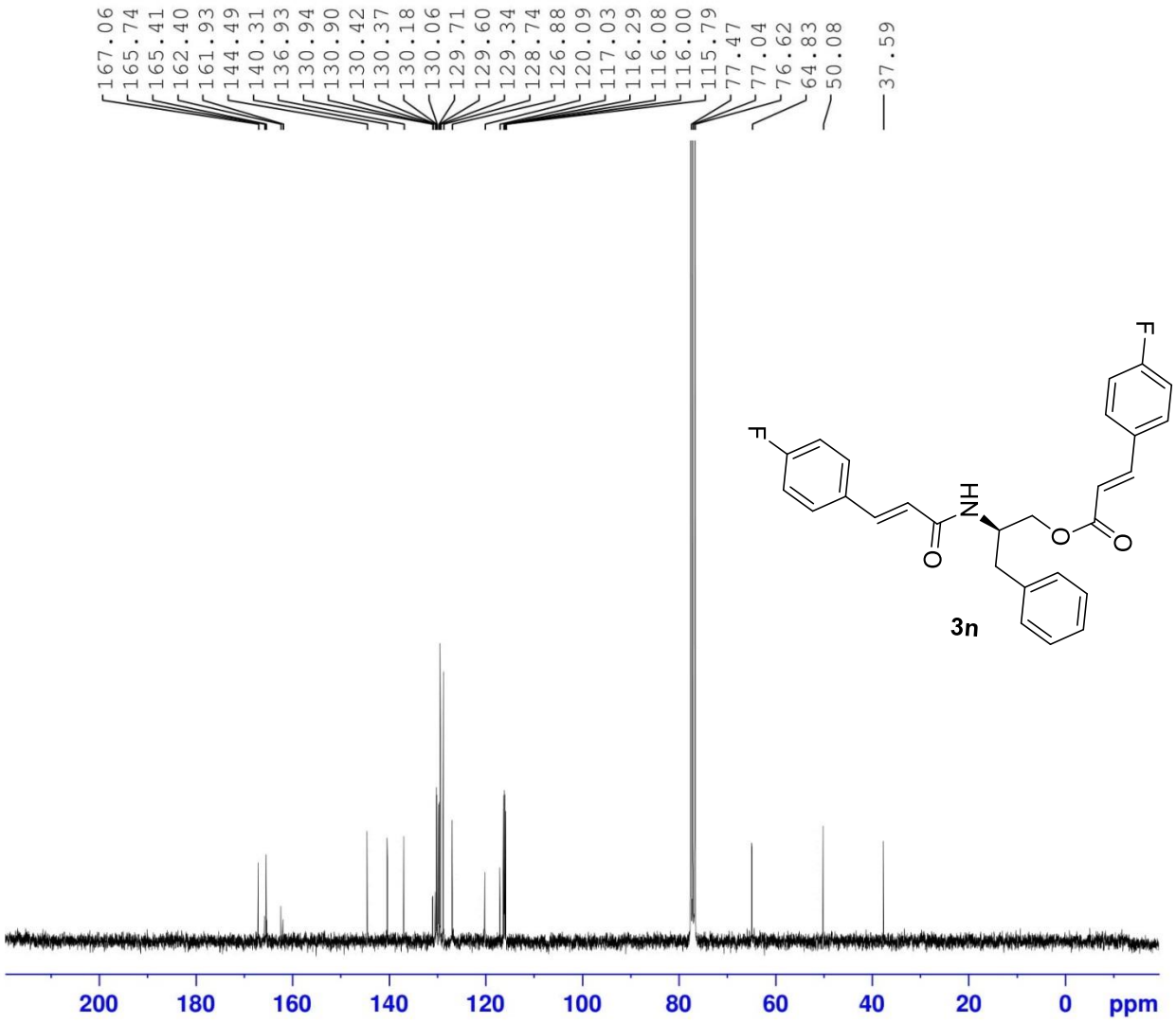



```
Current Data Parameters
NAME                 H2552D8
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_               20180904
Time                   15.46
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                6009.615 Hz
FIDRES            0.091699 Hz
AQ                5.4525952 sec
RG                      362
DW                   83.200 usec
DE                    6.50 usec
TE                   298.0 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1           300.1318534 MHz
NUC1                    1H
P1                   13.00 usec
PLW1           10.69999981 W

F2 - Processing parameters
SI                    65536
SF             300.1300056 MHz
WDW                     EM
SSB          0
LB                    0.30 Hz
GB           0
PC                    1.00
```




```
Current Data Parameters
NAME                 C2552D3
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_               20180905
Time                   16.58
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG              zgpg30
TD                     65536
SOLVENT                CDCl3
NS                      1040
DS                         4
SWH                18028.846 Hz
FIDRES             0.275098 Hz
AQ                 1.8175317 sec
RG                       912
DW                    27.733 usec
DE                     6.50 usec
TE                    300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1               75.4752949 MHz
NUC1                      13C
P1                       9.47 usec
PLW1              38.00000000 W

======== CHANNEL f2 ========
SFO2              300.1312005 MHz
NUC2                      1H
CPDPRG[2              waltz16
PCPD2                 90.00 usec
PLW2              10.69999981 W
PLW12              0.22325000 W
PLW13              0.11229000 W

F2 - Processing parameters
SI                        32768
SF                 75.4677485 MHz
WDW                          EM
SSB          0
LB                        1.00 Hz
GB           0
PC                        1.40
```

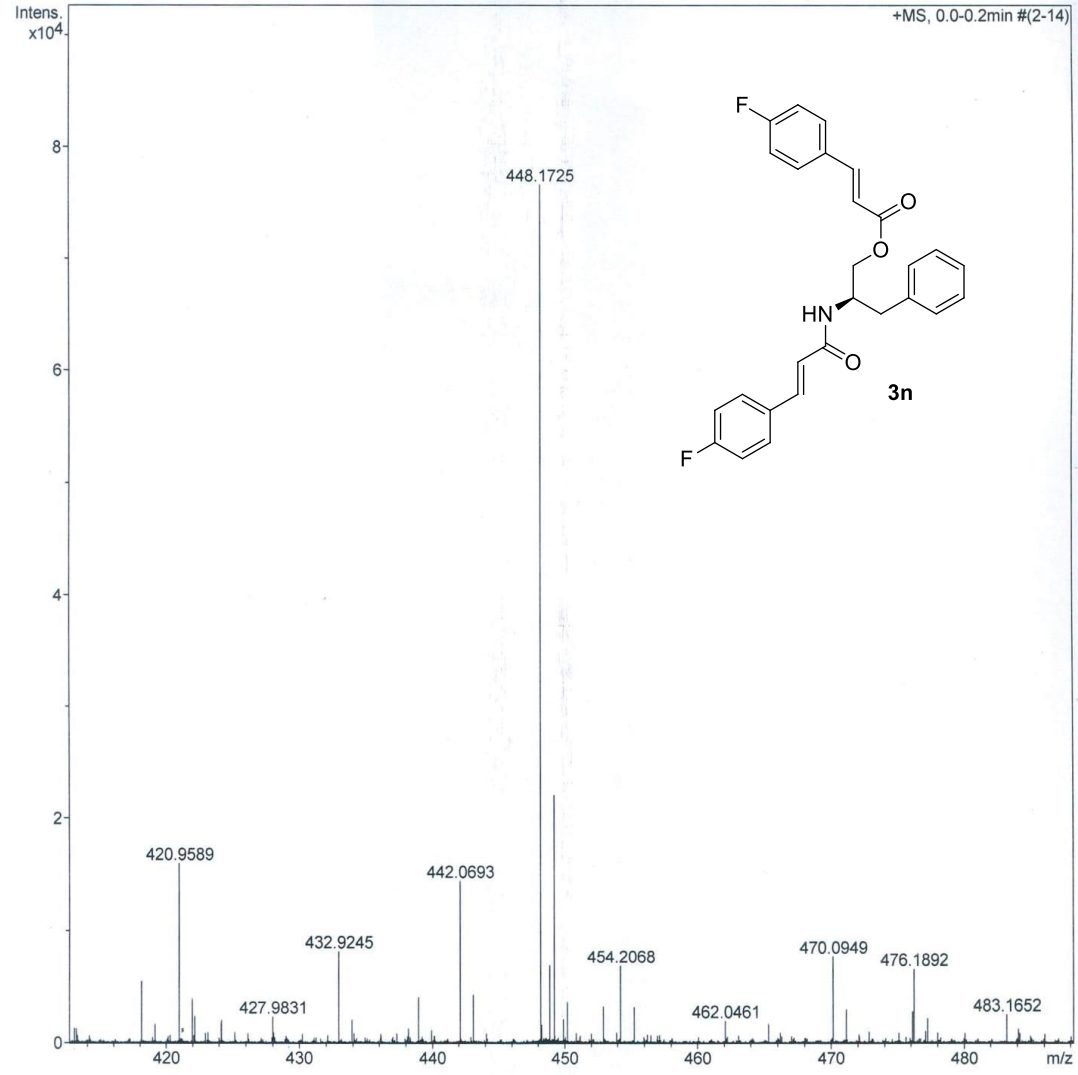
UOH -SCHOOL OF CHEMISTRY -HRMS

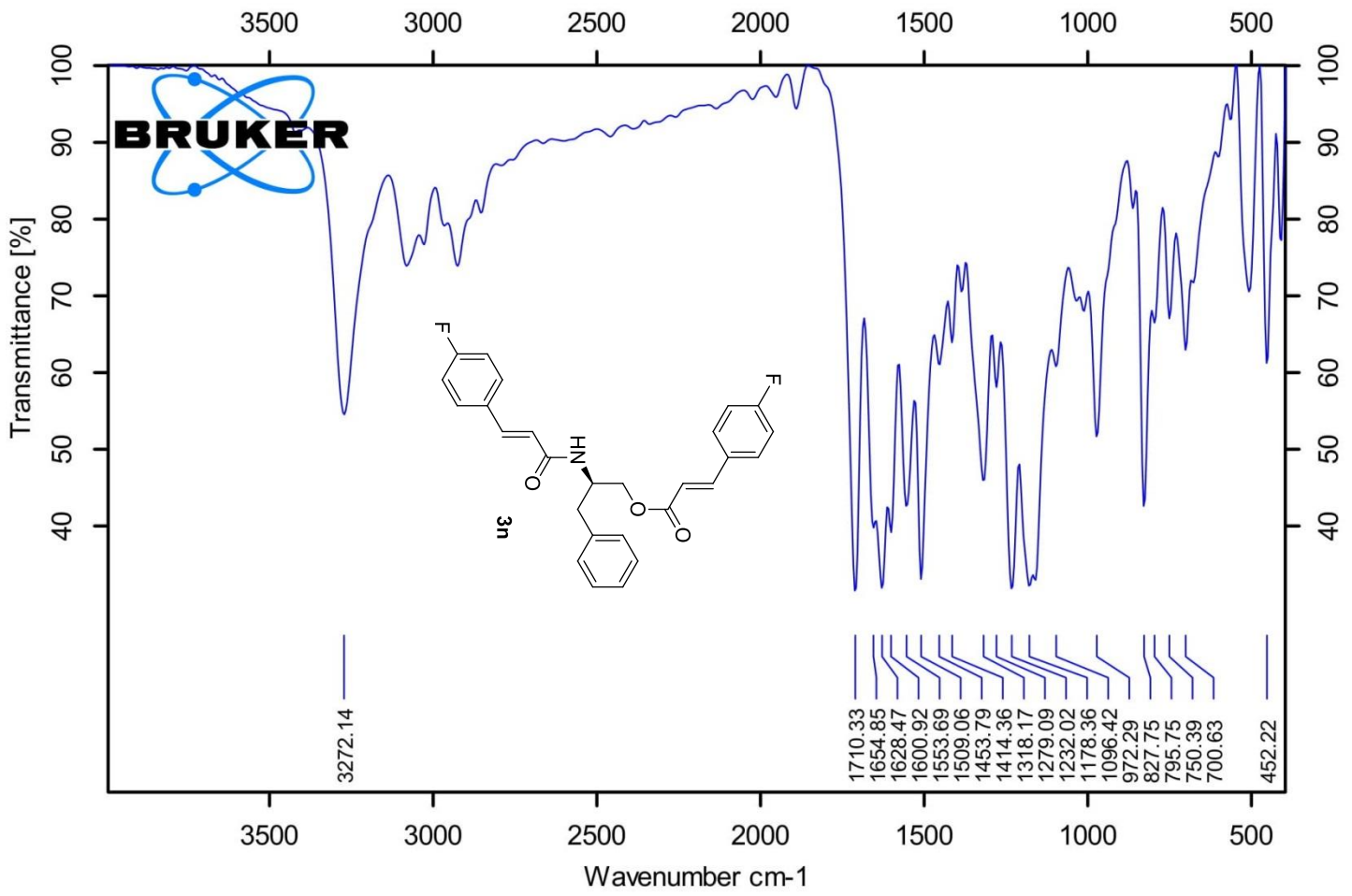
Analysis Info

Analysis Name	D:\Data\2018\PROF MP\DEC\8.d	Acquisition Date	12/11/2018 4:31:21 PM
Method	tune_low.m	Operator	UOH-Chemistry
Sample Name	8	Instrument	maXis 10138
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3800 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1800 m/z	Set Collision Cell RF	350.0 Vpp	Set Divert Valve	Waste

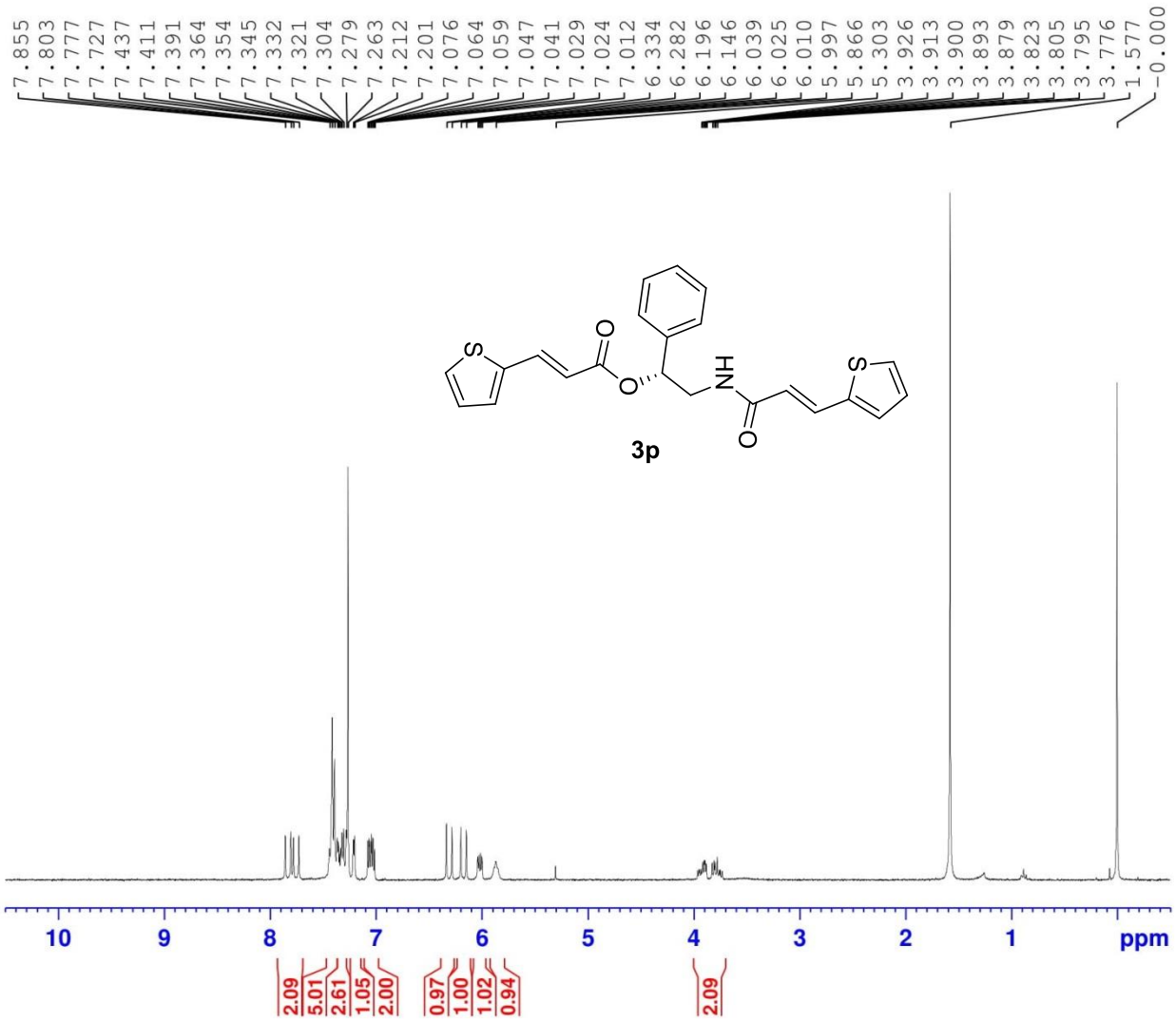




SAMPLE SCANS : 48
DATE : 11-03-2019
TIME : 17:15:44

SAMPLE : PVS 7
TECHNIQUE : Thinfilm
USER : Default

Current Data Parameters
NAME H2552F14
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20181113
Time 16.26
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 406
DW 83.200 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 300.1318534 MHz
NUC1 1H
P1 13.00 usec
PLW1 10.69999981 W
F2 - Processing parameters
SI 65536
SF 300.1300063 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
HN
O
O
O
3o
1.10
1.17
3.86
11.71
1.00
1.02
1.01
0.95
1.03
1.06
ppm
7.765
7.712
7.660
7.608
7.545
7.532
7.521
7.509
7.496
7.483
7.458
7.432
7.401
7.381
7.363
7.350
7.342
7.262
6.544
6.491
6.401
6.349
6.079
6.066
6.051
6.038
5.975
5.959
5.301
3.965
3.944
3.931
3.912
3.898
3.871
3.853
3.843
3.824
3.795
1.600
1.255
0.905
0.882
0.859
0.072
-0.000

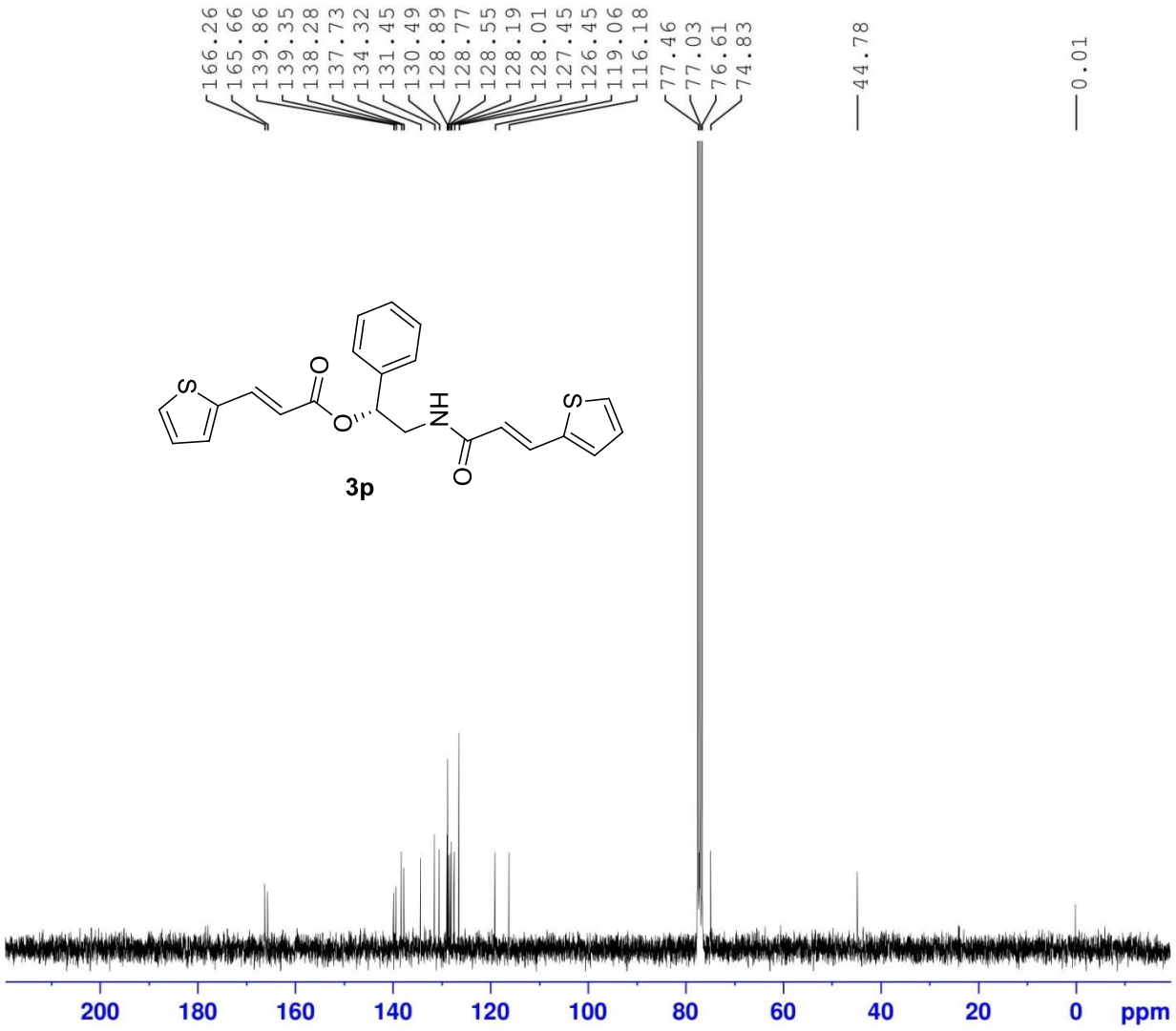



```
Current Data Parameters
NAME            H2552H19
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190212
Time               16.45
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4525952 sec
RG                   512
DW                83.200 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                1H
P1                13.00 usec
PLW1         10.69999981 W

F2 - Processing parameters
SI                 65536
SF           300.1300060 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



Current Data Parameters
NAME C2552F10
EXPNO 1
PROCNO 1

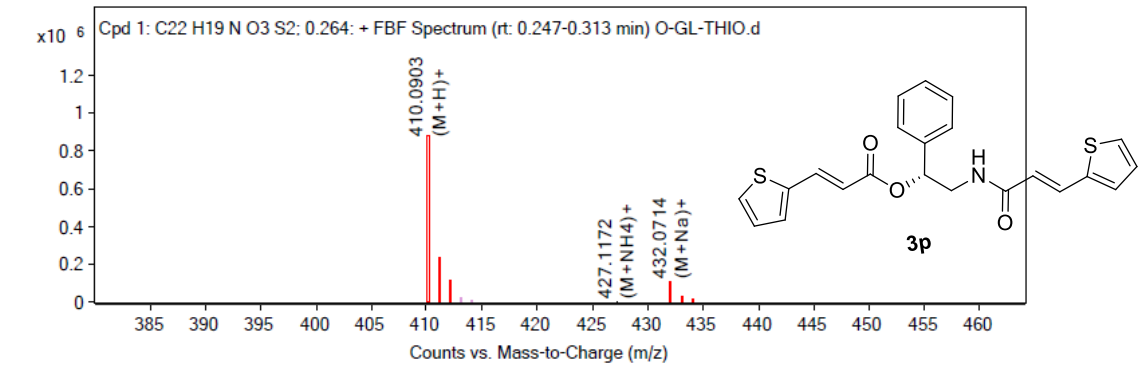
F2 - Acquisition Parameters
Date_ 20190218
Time 11.00
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 724
DW 27.733 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 75.4752949 MHz
NUC1 13C
P1 9.47 usec
PLW1 38.00000000 W

======== CHANNEL f2 ========
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 10.69999981 W
PLW12 0.22325000 W
PLW13 0.11229000 W

F2 - Processing parameters
SI 32768
SF 75.4677485 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

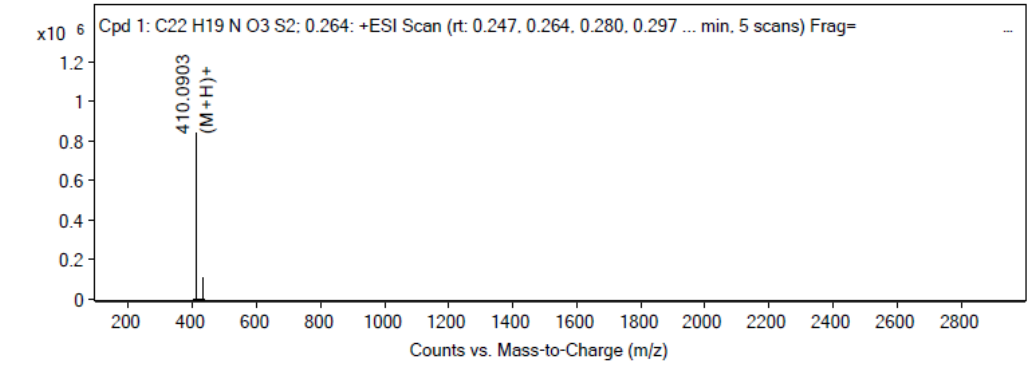
Qualitative Compound Identification Report



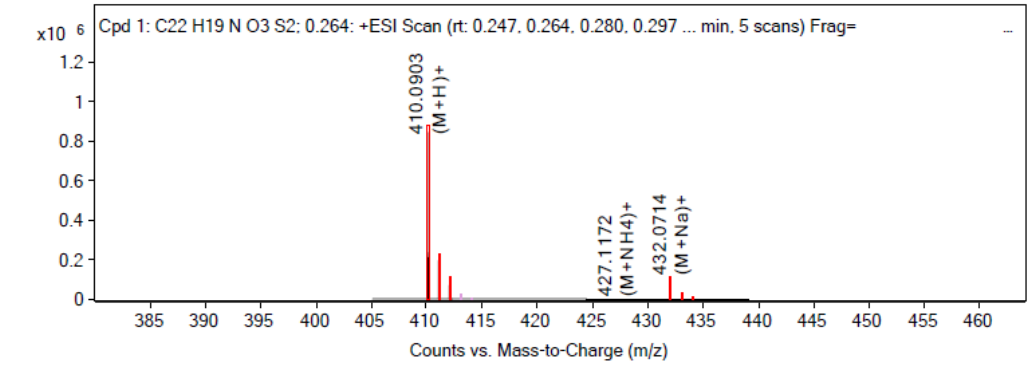
MS Spectrum Peak List

m/z	z	Abund	Ion
410.0903	1	878525.31	(M+H)+
411.0929	1	191678.69	(M+H)+
412.0882	1	68229.55	(M+H)+
427.1172	1	77.48	(M+NH4)+
432.0714	1	108341.65	(M+Na)+
433.0735	1	22135.11	(M+Na)+
434.0688	1	8466.07	(M+Na)+

MS Spectrum



MS Zoomed Spectrum

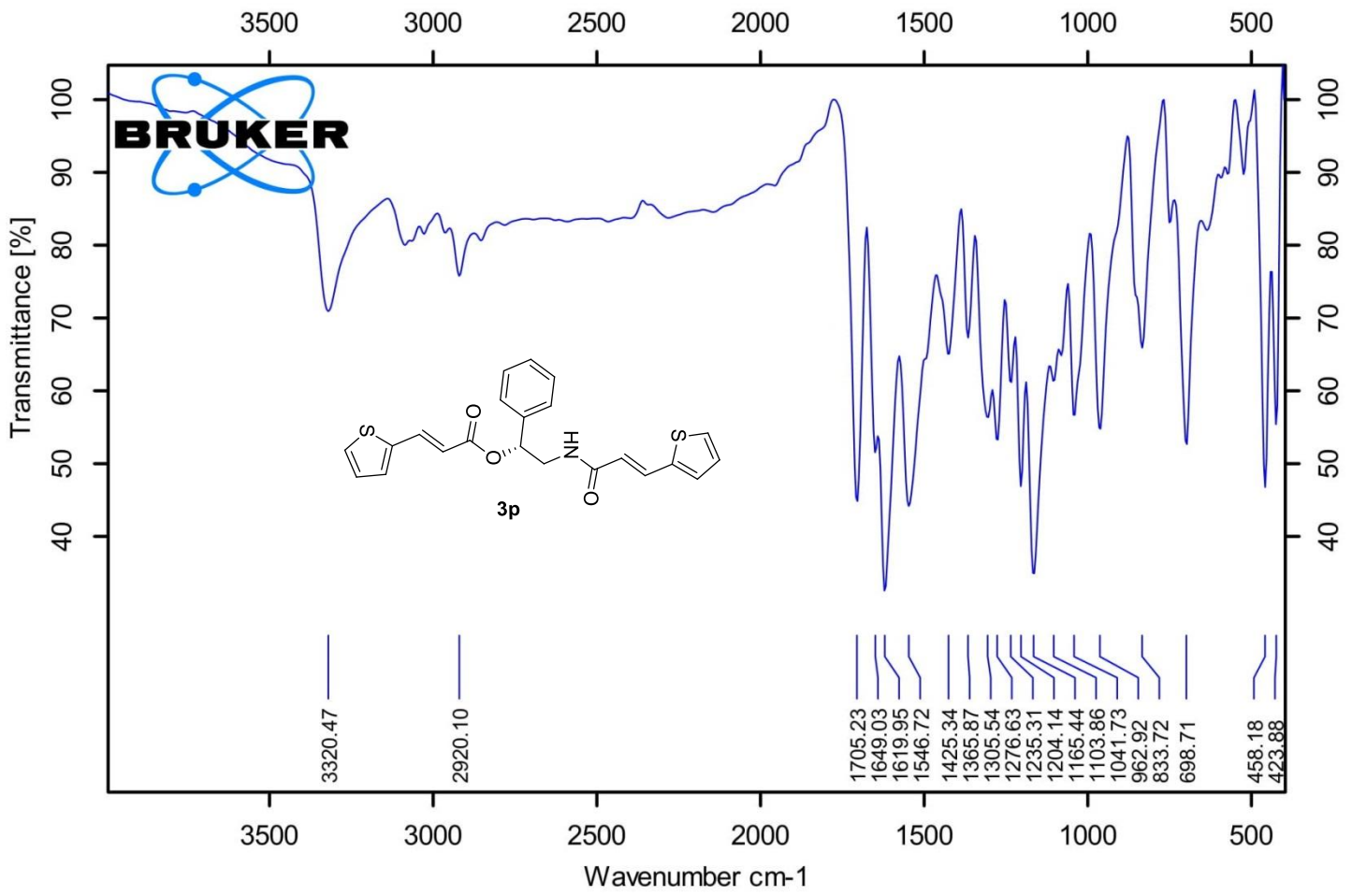


MS Spectrum Peak List

m/z	Calc m/z	Diff(ppm)	z	Abund	Ion
410.0902	410.0879	-5.66		878433.87	
410.0903	410.0879	-5.71	1	878525.31	(M+H)+

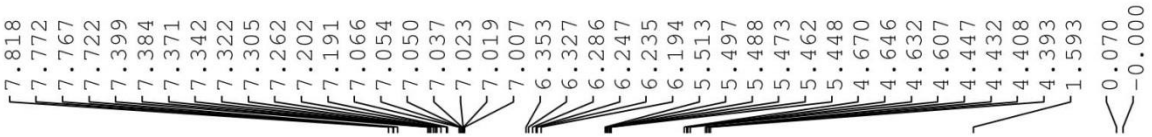
Printed at 2:24 PM on 7-Mar-2019

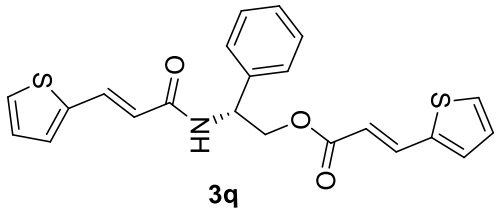
Agilent Technologies



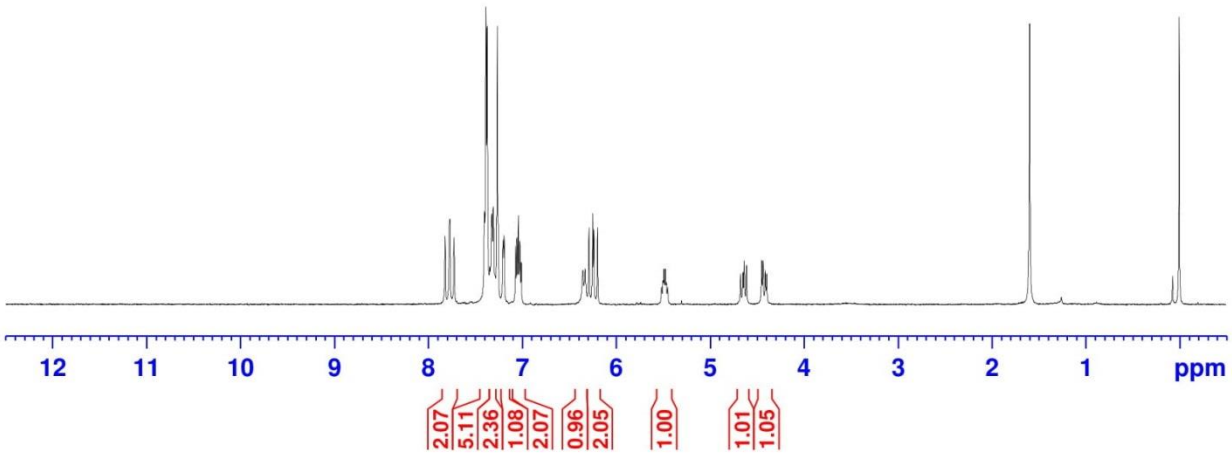
SAMPLE SCANS : 48
DATE : 11-03-2019
TIME : 17:53:58

SAMPLE : PVS 3
TECHNIQUE : Thinfilm
USER : Default





3q

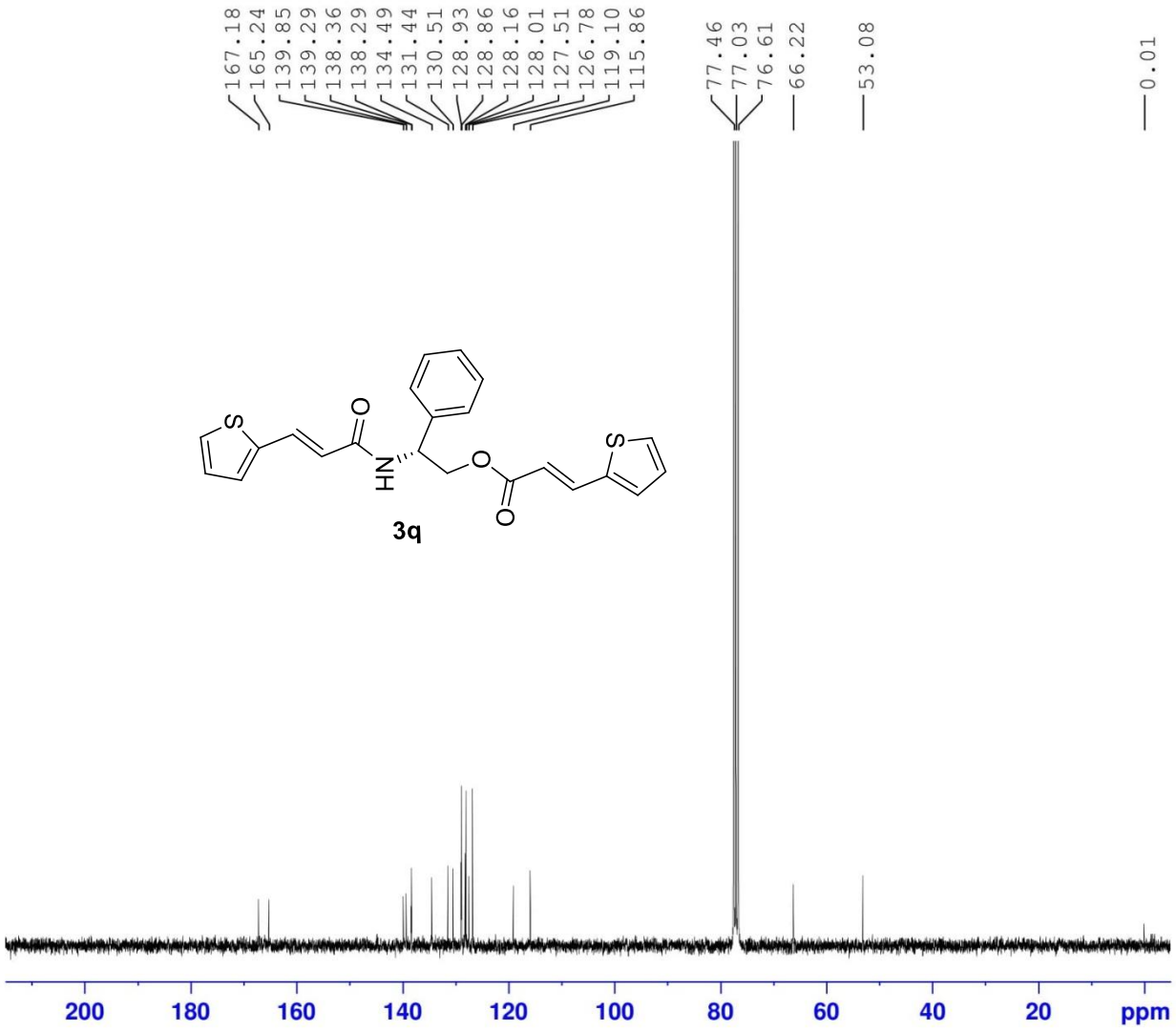



```
Current Data Parameters
NAME            H22552H18
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20190209
Time                14.50
INSTRUM             spect
PROBHD    5 mm BBO BB-1H
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              6009.615 Hz
FIDRES           0.091699 Hz
AQ               5.4525952 sec
RG                    406
DW                 83.200 usec
DE                  6.50 usec
TE                 298.0 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          300.1318534 MHz
NUC1                   1H
P1                  13.00 usec
PLW1          10.69999981 W

F2 - Processing parameters
SI                  65536
SF            300.1300062 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



Current Data Parameters
NAME C2552F9
EXPNO 1
PROCNO 1

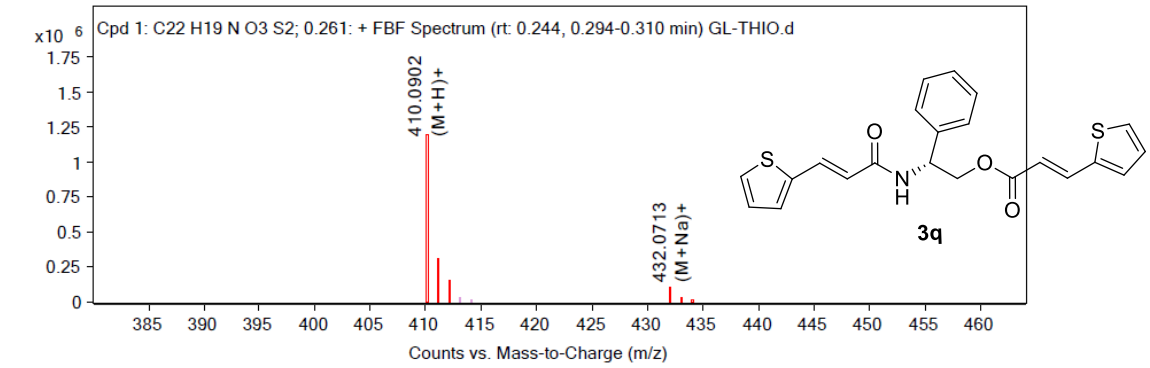
F2 - Acquisition Parameters
Date_ 20190214
Time 13.57
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 645
DW 27.733 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 75.4752949 MHz
NUC1 13C
P1 9.47 usec
PLW1 38.00000000 W

======== CHANNEL f2 ========
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 10.69999981 W
PLW12 0.22325000 W
PLW13 0.11229000 W

F2 - Processing parameters
SI 32768
SF 75.4677485 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

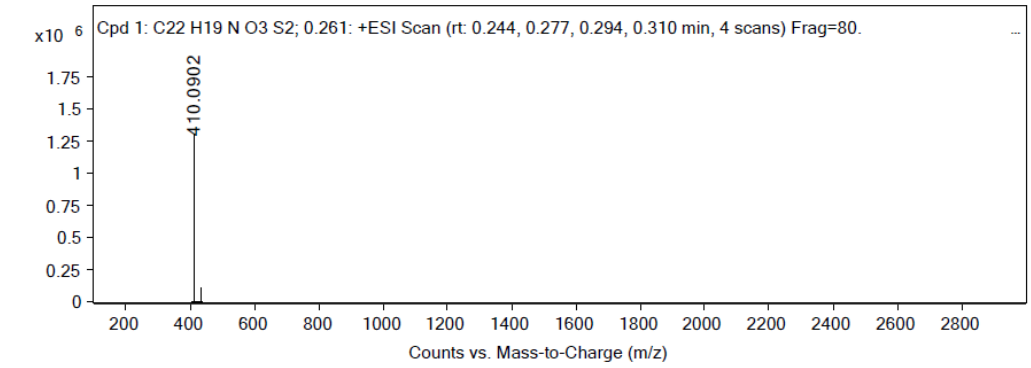
Qualitative Compound Identification Report



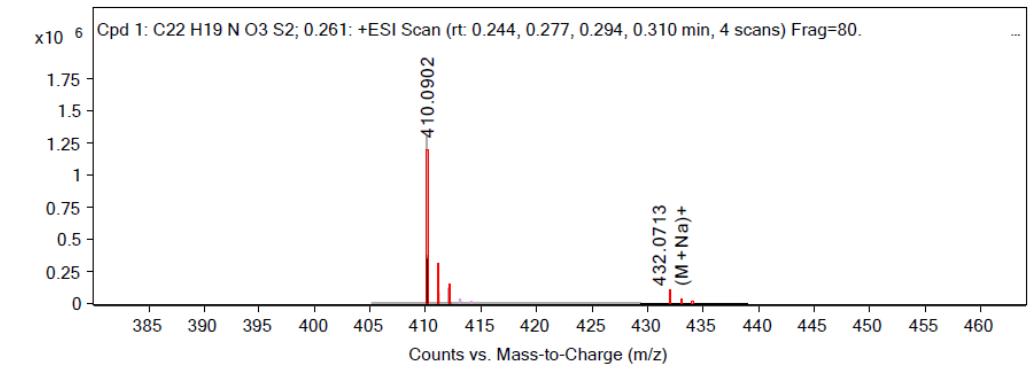
MS Spectrum Peak List

m/z	z	Abund	Ion
410.0902	1	1193832.63	(M+H)+
411.093	1	263636.94	(M+H)+
412.0885	1	97219.84	(M+H)+
432.0713	1	110280.63	(M+Na)+
433.0739	1	22857.13	(M+Na)+
434.069	1	9524.97	(M+Na)+

MS Spectrum



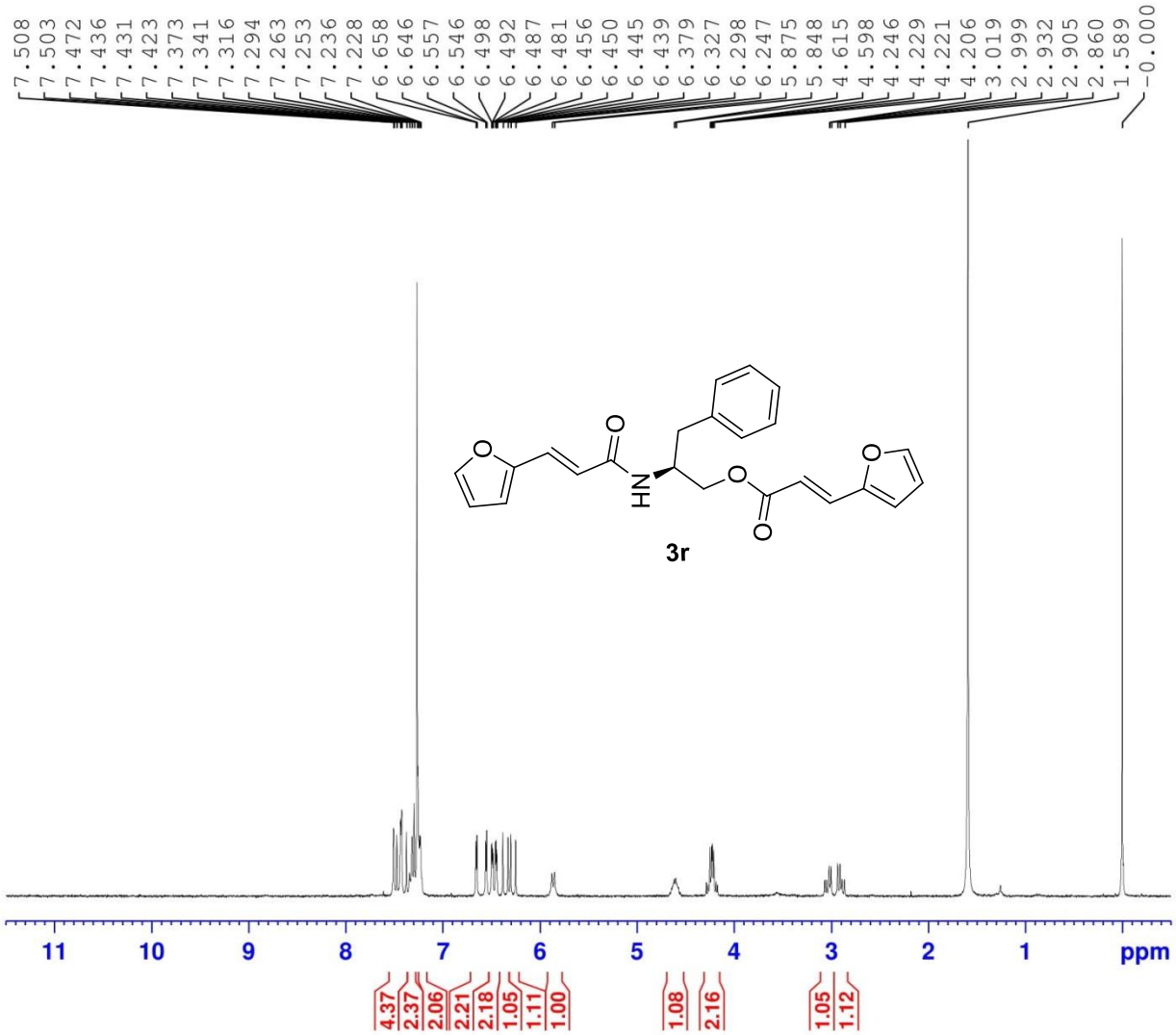
MS Zoomed Spectrum



MS Spectrum Peak List

m/z	Calc m/z	Diff(ppm)	z	Abund	Ion
410.0902	410.0879	-5.65	1	1193832.63	(M+H)+
410.0902				1362153.67	
411.093	411.091	-4.83	1	263636.94	(M+H)+

Agilent Technologies

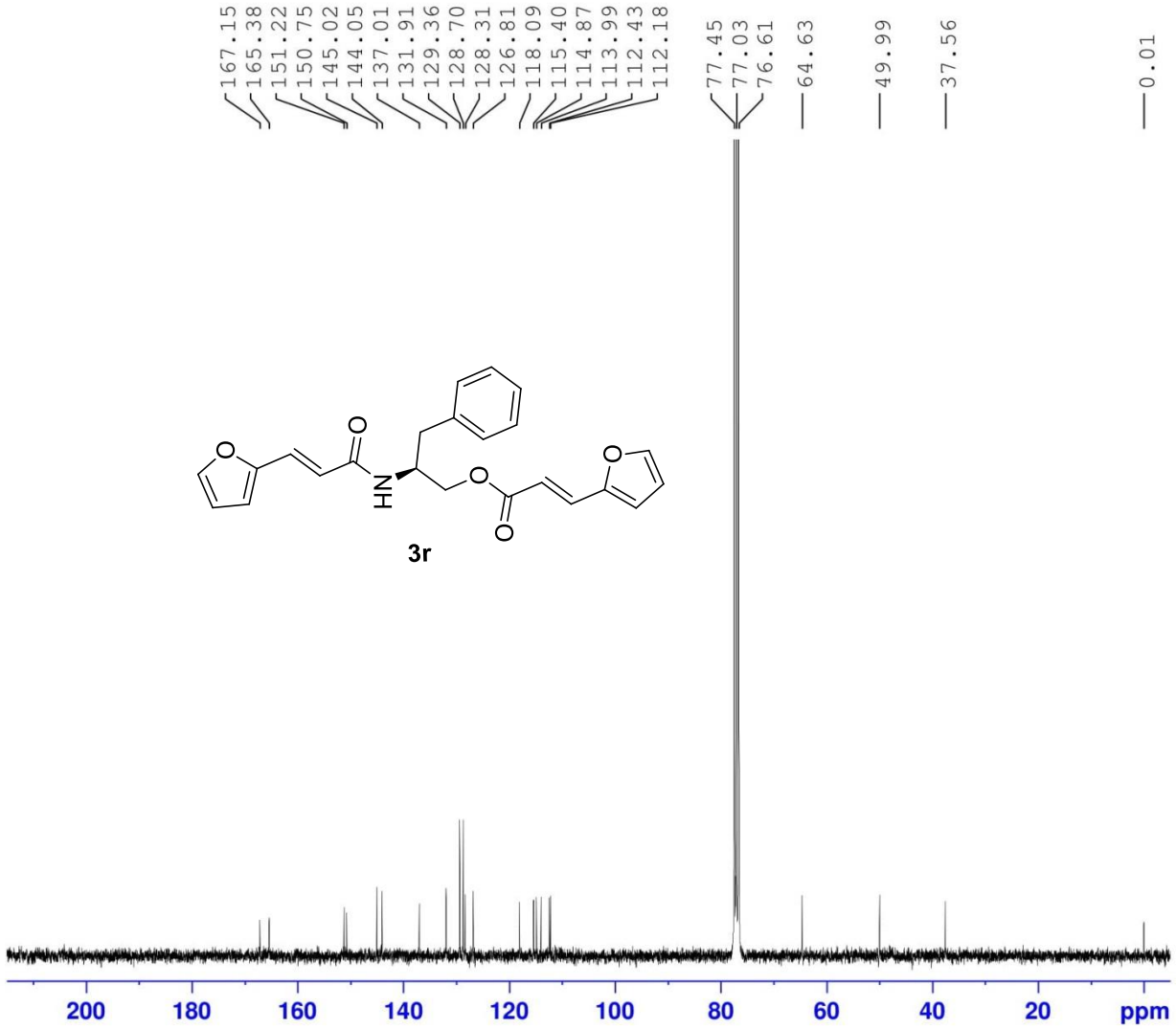



```
Current Data Parameters
NAME                 H2552I5
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_               20190218
Time                   15.55
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                6009.615 Hz
FIDRES            0.091699 Hz
AQ                5.4525952 sec
RG                      456
DW                   83.200 usec
DE                    6.50 usec
TE                   298.0 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1           300.1318534 MHz
NUC1                    1H
P1                   13.00 usec
PLW1           10.69999981 W

F2 - Processing parameters
SI                    65536
SF             300.1300057 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```




```
Current Data Parameters
NAME          C3339A1-R
EXPNO                 1
PROCNO                1

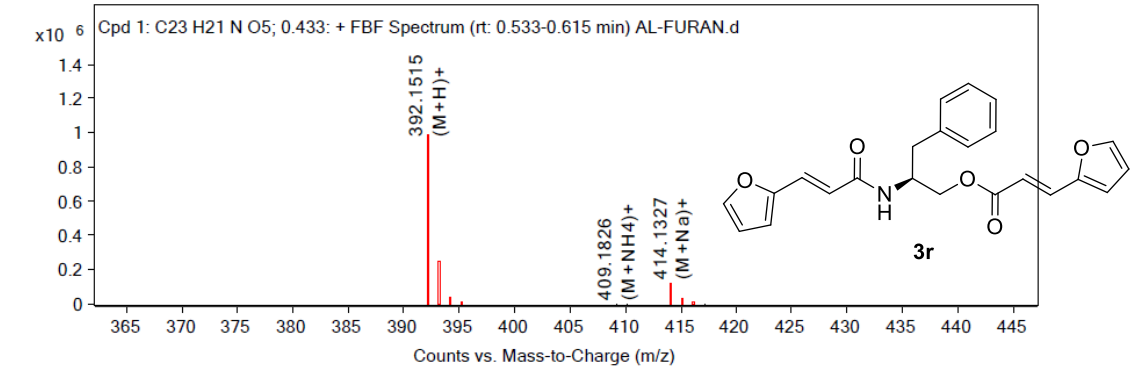
F2 - Acquisition Parameters
Date_          20190224
Time               2.03
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 5120
DS                    4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ            1.8175317 sec
RG                  812
DW               27.733 usec
DE                6.50 usec
TE               300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         75.4752949 MHz
NUC1                13C
P1                 9.47 usec
PLW1        38.00000000 W

======== CHANNEL f2 ========
SFO2        300.1312005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.69999981 W
PLW12        0.22325000 W
PLW13        0.11229000 W

F2 - Processing parameters
SI                32768
SF           75.4677485 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

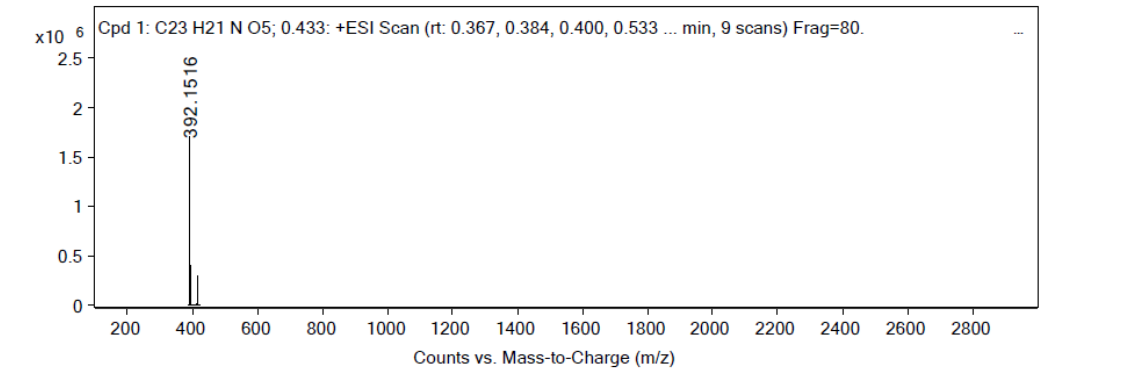
Qualitative Compound Identification Report



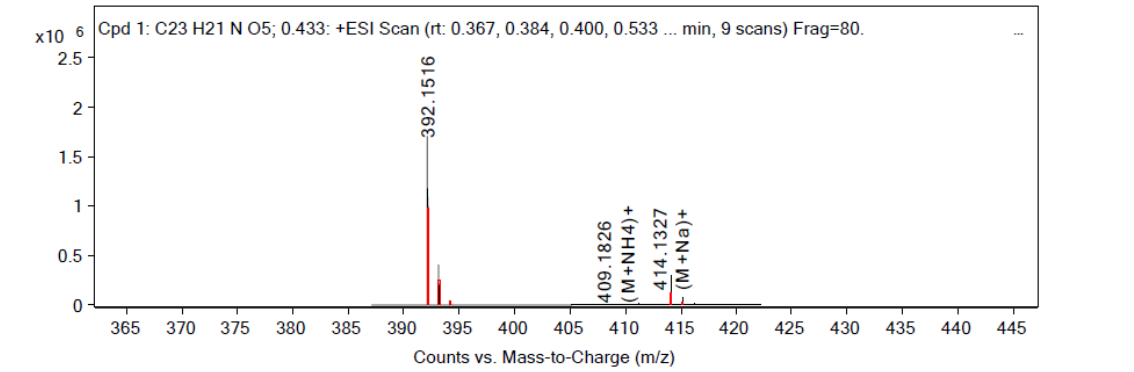
MS Spectrum Peak List

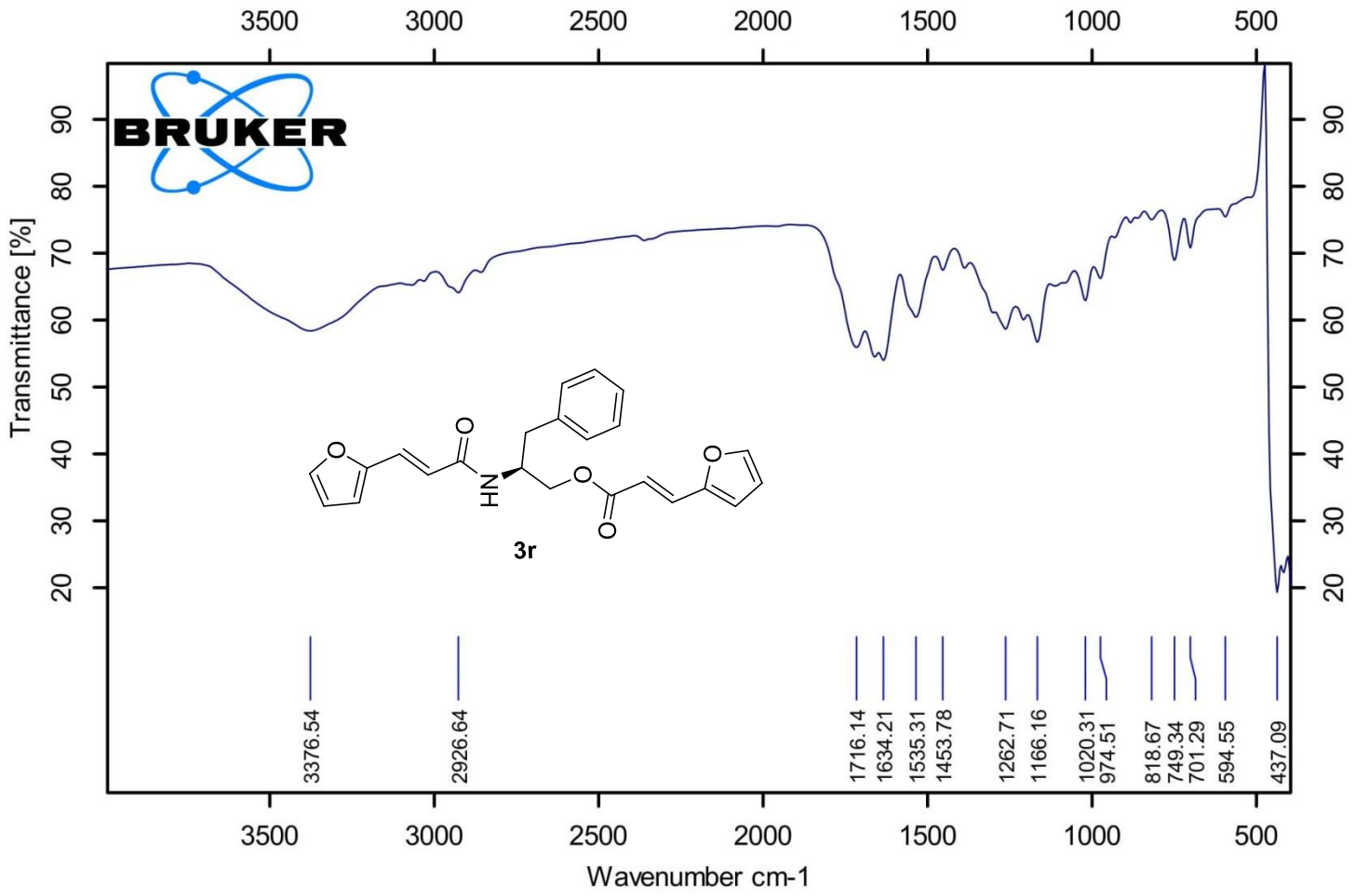
m/z	z	Abund	Ion
392.1515	1	982760.44	(M+H)+
393.1543	1	225074.88	(M+H)+
394.1559	1	26647.7	(M+H)+
395.1589	1	2851.44	(M+H)+
409.1826	1	344.36	(M+NH4)+
410.192	1	49.66	(M+NH4)+
414.1327	1	124636.46	(M+Na)+
415.1354	1	25822.48	(M+Na)+
416.1375	1	3815.51	(M+Na)+
417.14	1	488.55	(M+Na)+

MS Spectrum



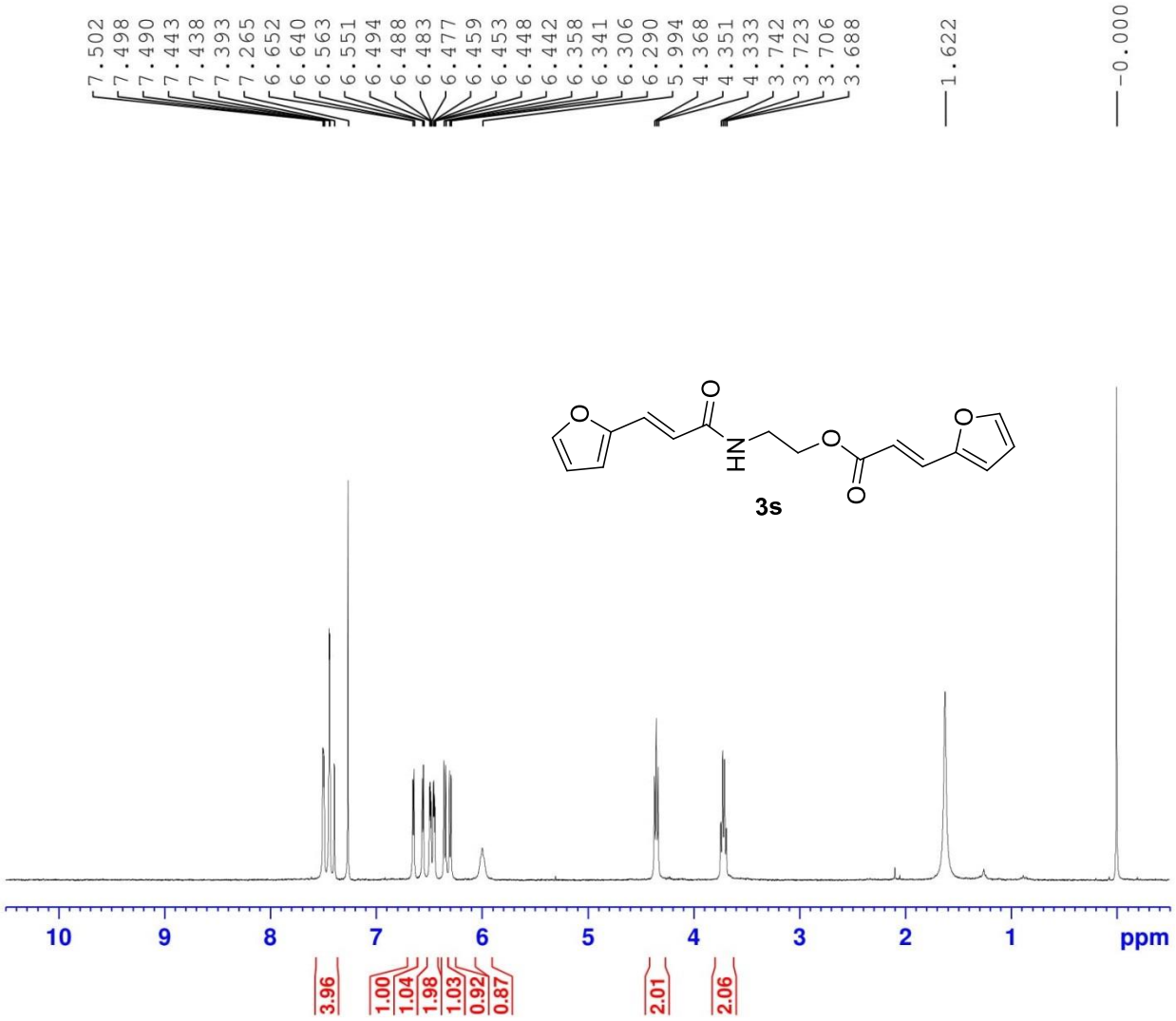
MS Zoomed Spectrum





SAMPLE SCANS : 48
DATE : 11-03-2019
TIME : 15:13:37

SAMPLE : PVS 4
TECHNIQUE : Thinfilm
USER : Default

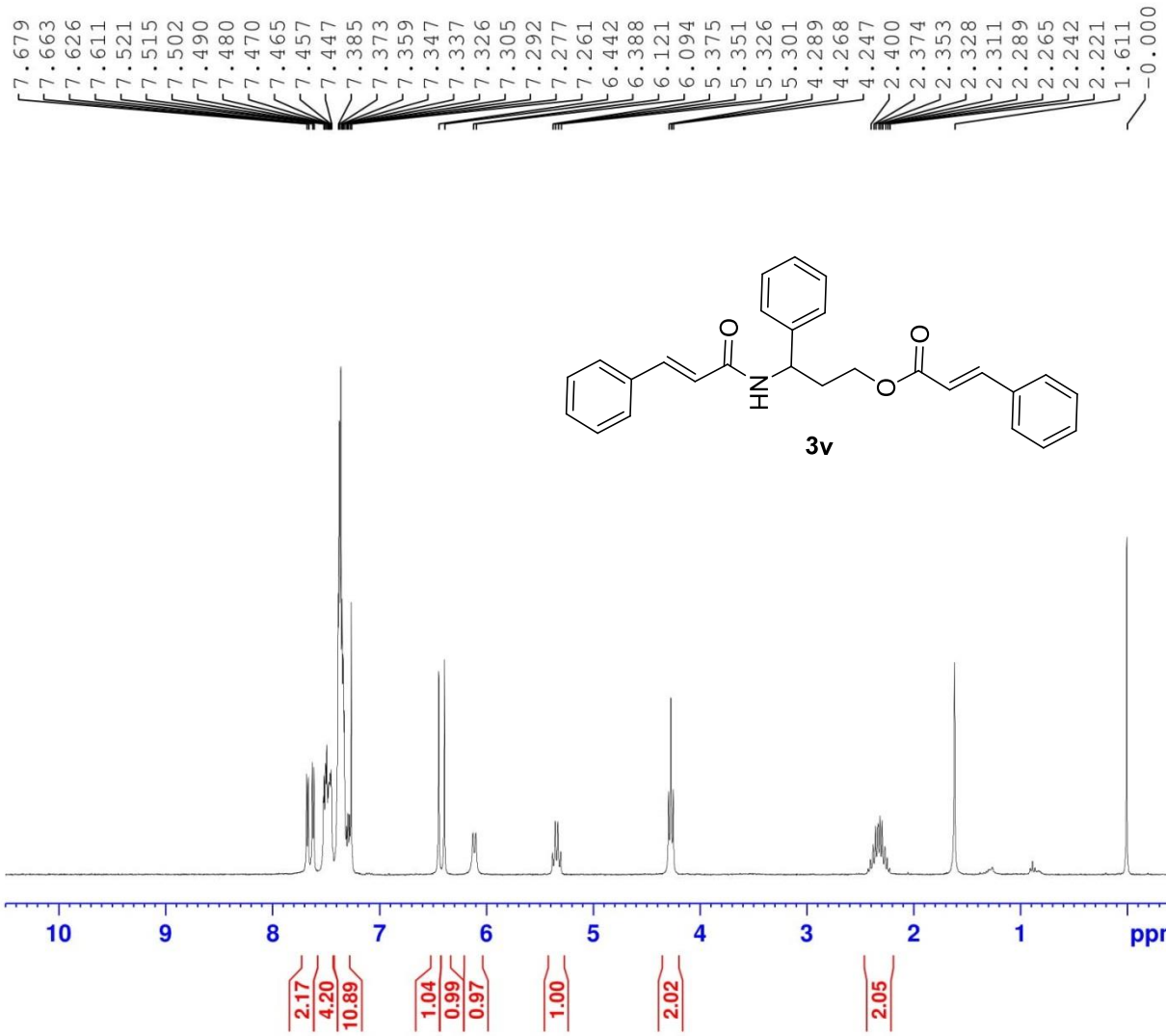



```
Current Data Parameters
NAME              H2552I2
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20190214
Time                15.52
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              6009.615 Hz
FIDRES           0.091699 Hz
AQ               5.4525952 sec
RG                    406
DW                 83.200 usec
DE                  6.50 usec
TE                 298.0 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                   1H
P1                 13.00 usec
PLW1         10.69999981 W

F2 - Processing parameters
SI                  65536
SF           300.1300053 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

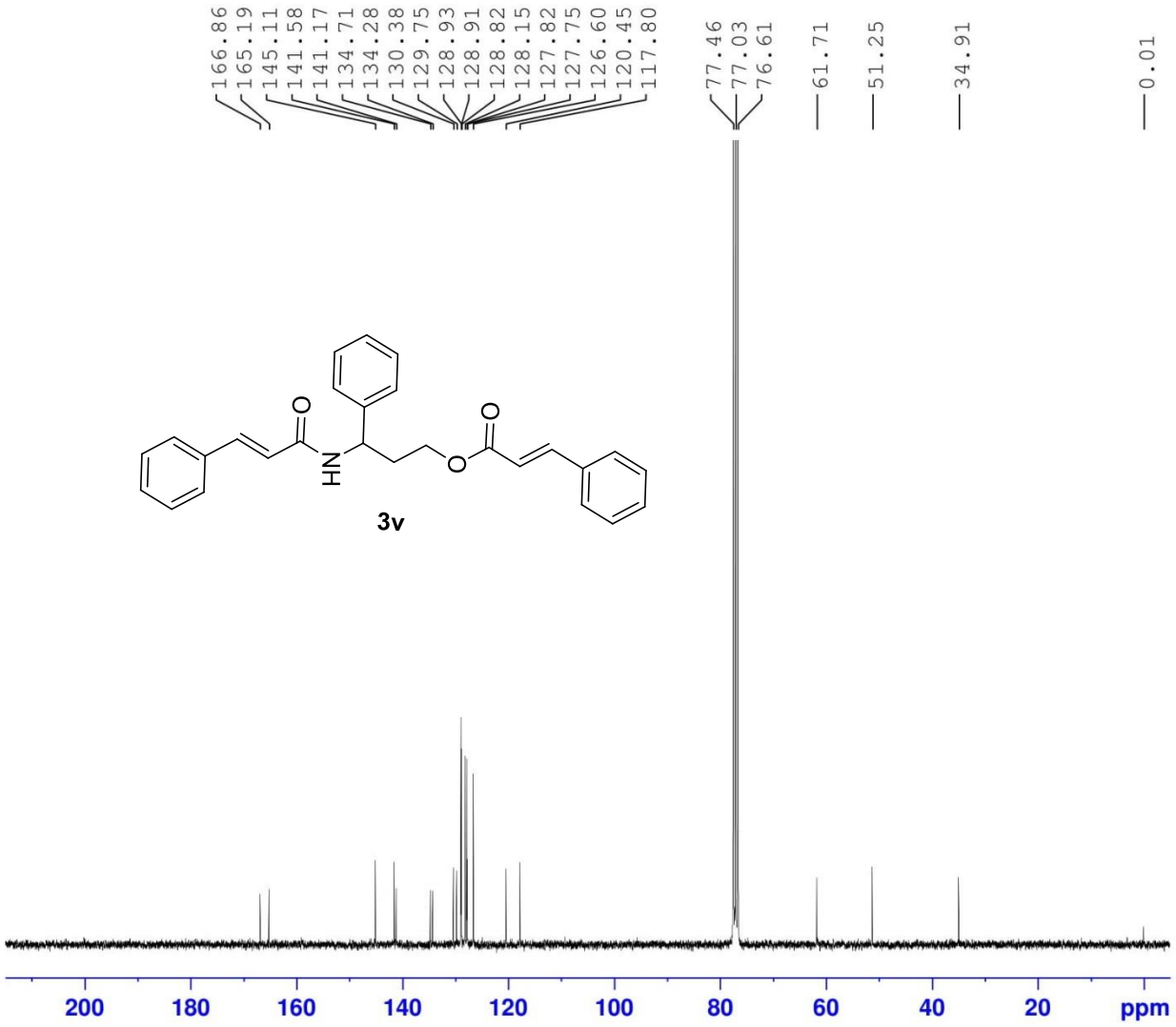



```
Current Data Parameters
NAME            H2552I16
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190226
Time               18.30
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4525952 sec
RG                   362
DW                83.200 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                   1H
P1                 13.00 usec
PLW1         10.69999981 W

F2 - Processing parameters
SI                 65536
SF           300.1300065 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME            C3339A3
EXPNO                 1
PROCNO                1

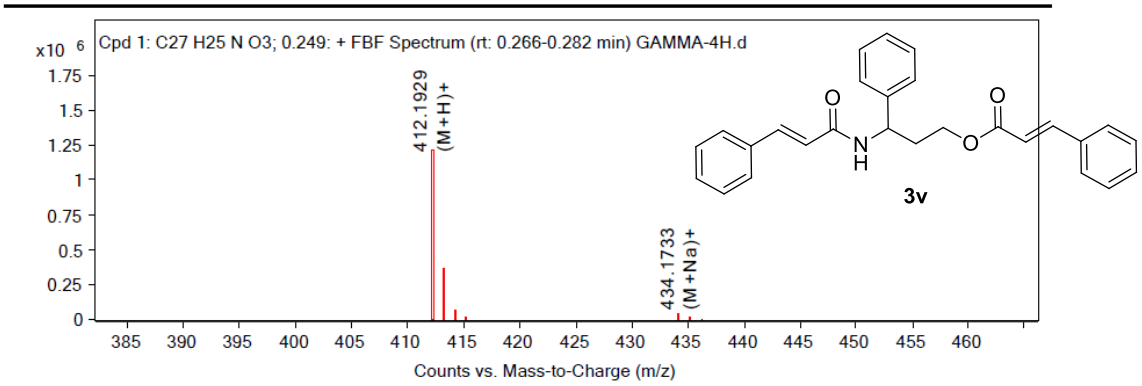
F2 - Acquisition Parameters
Date_          20190227
Time              22.29
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3072
DS                    4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ            1.8175317 sec
RG                  645
DW               27.733 usec
DE                6.50 usec
TE               300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         75.4752949 MHz
NUC1                13C
P1                 9.47 usec
PLW1        38.00000000 W

======== CHANNEL f2 ========
SFO2        300.1312005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.69999981 W
PLW12        0.22325000 W
PLW13        0.11229000 W

F2 - Processing parameters
SI                32768
SF           75.4677485 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

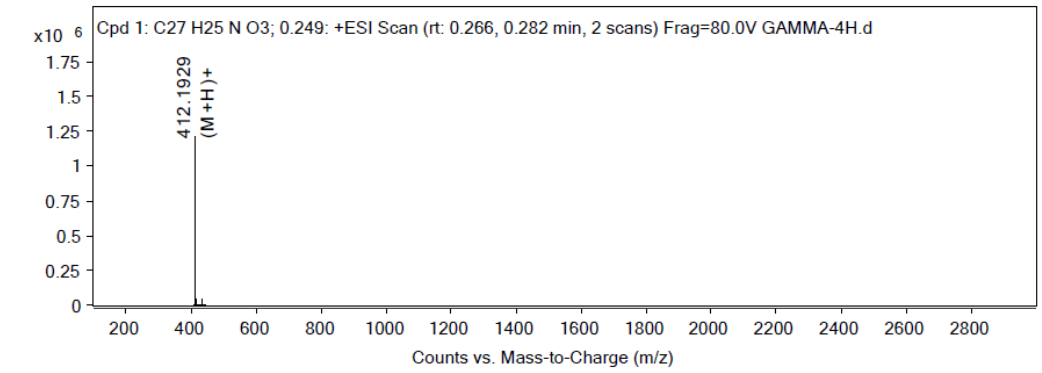
Qualitative Compound Identification Report



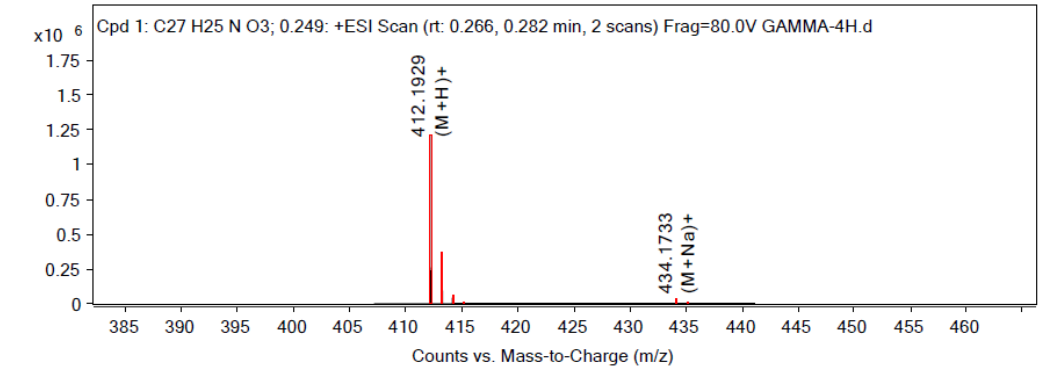
MS Spectrum Peak List

m/z	z	Abund	Ion
412.1929	1	1210681.25	(M+H)+
413.196	1	323498.75	(M+H)+
414.199	1	39831.24	(M+H)+
415.2021	1	4043.6	(M+H)+
434.1733	1	42720.44	(M+Na)+
435.1765	1	11606.66	(M+Na)+
436.1795	1	1932.75	(M+Na)+

MS Spectrum

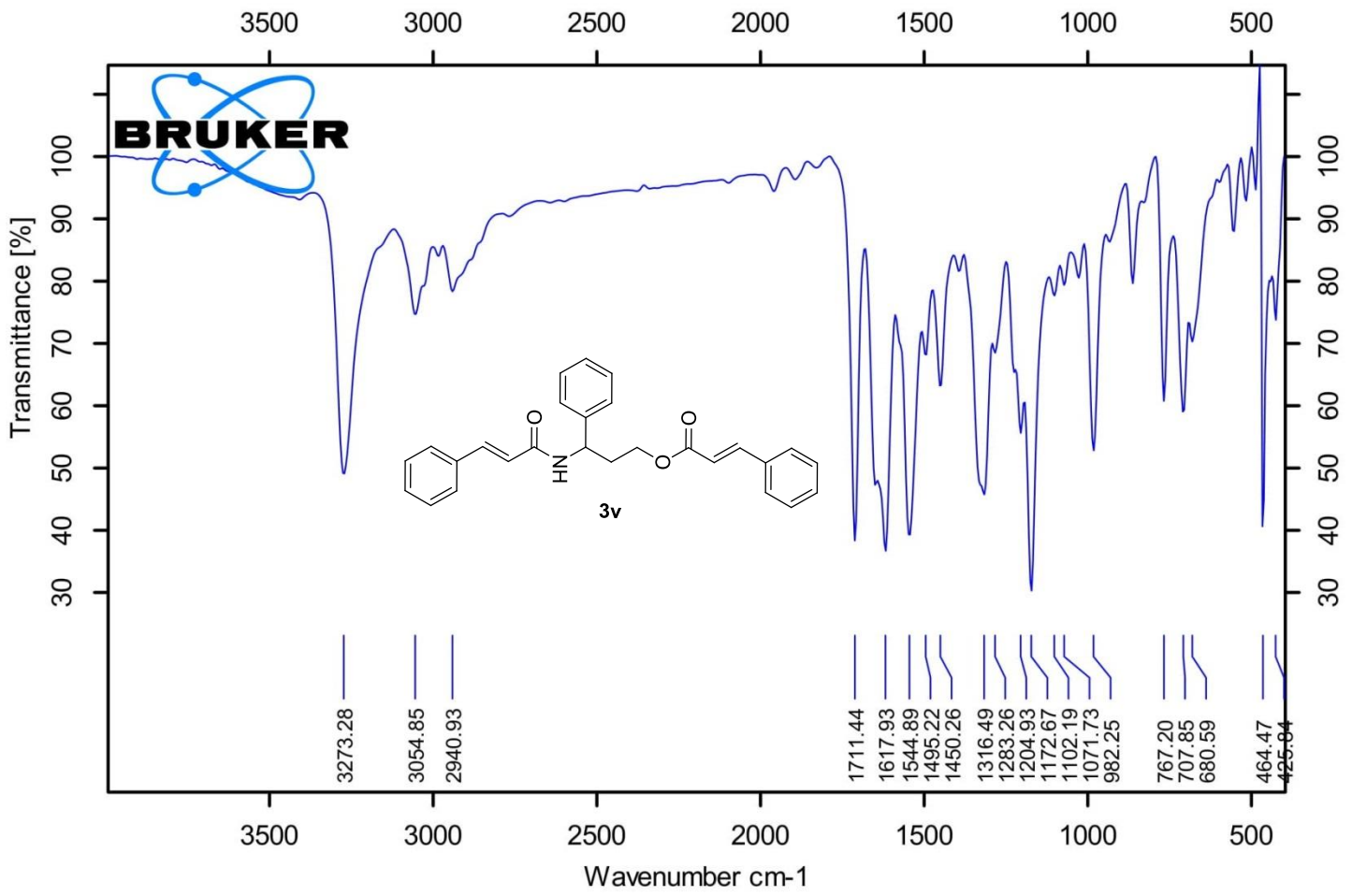


MS Zoomed Spectrum



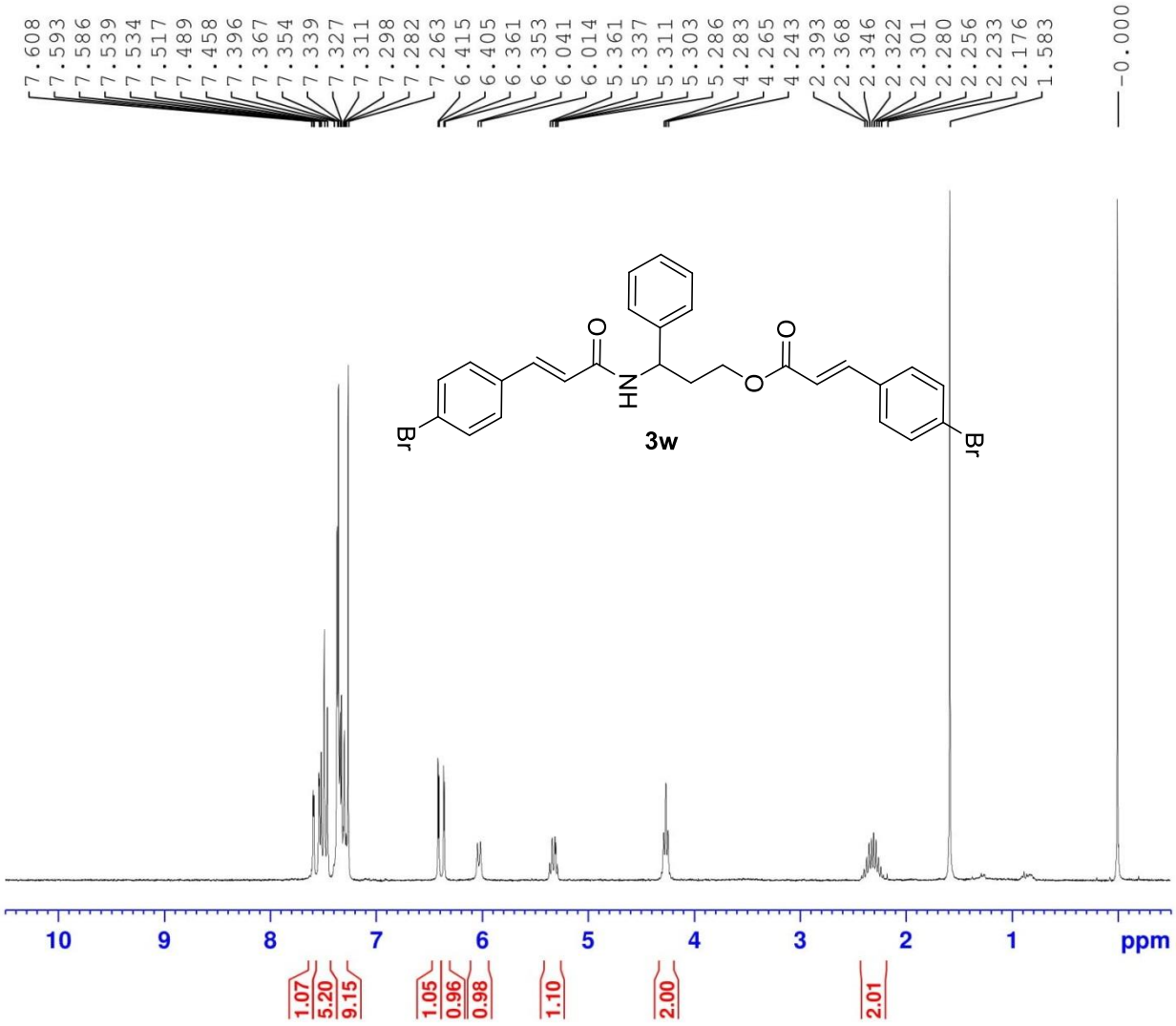
MS Spectrum Peak List

m/z	Calc m/z	Diff(ppm)	z	Abund	Ion
412.1929	412.1907	-5.36		1210878.71	
412.1929	412.1907	-5.4	1	1210681.25	(M+H)+



SAMPLE SCANS : 48
DATE : 11-03-2019
TIME : 18:07:14

SAMPLE : PVS 5
TECHNIQUE : Thinfilm
USER : Default

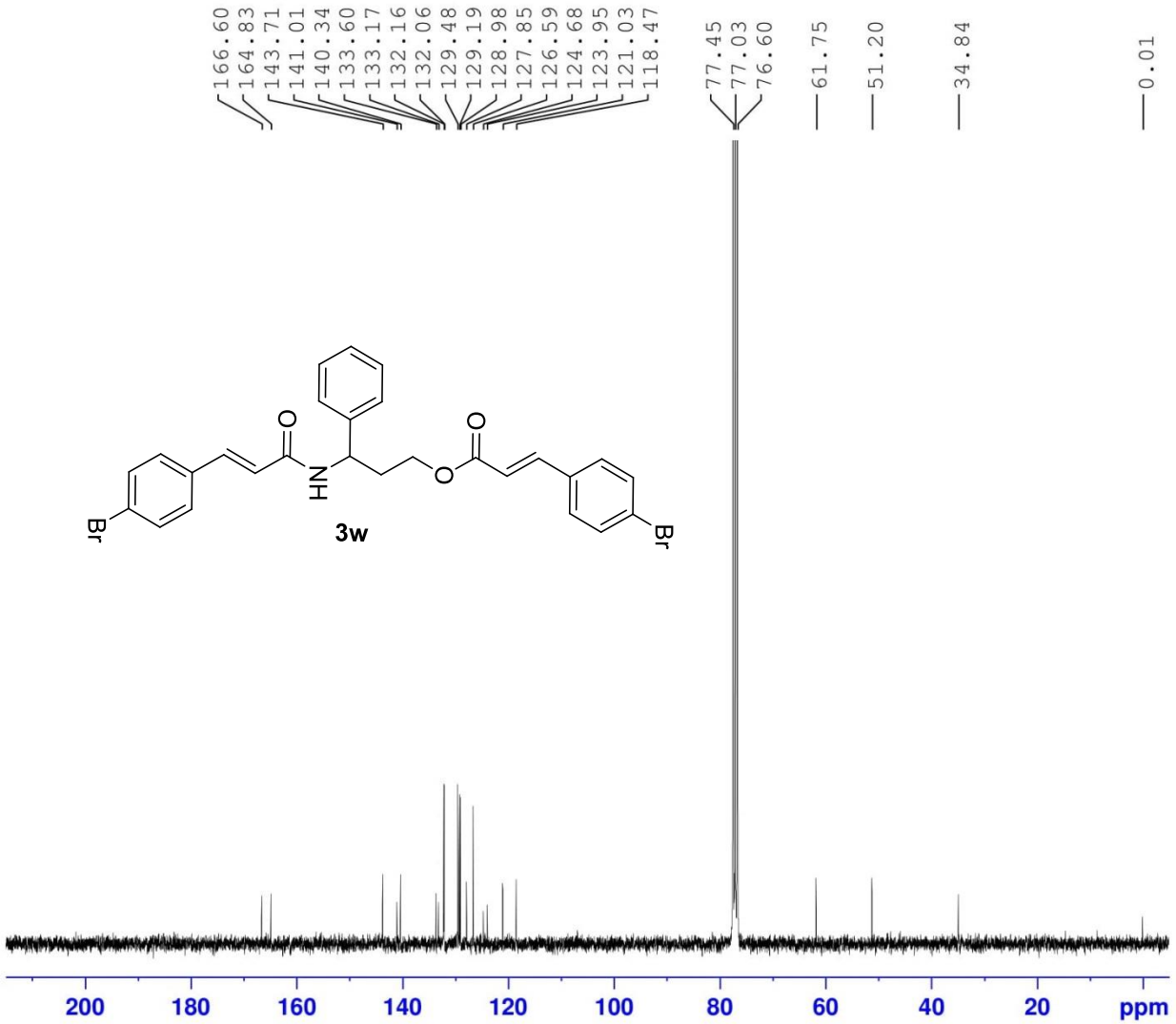



```
Current Data Parameters
NAME            H2552I17
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190226
Time               18.52
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4525952 sec
RG                   406
DW                83.200 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                1H
P1                13.00 usec
PLW1         10.69999981 W

F2 - Processing parameters
SI                 65536
SF           300.1300059 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME                 C333A4
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20190228
Time                  20.50
INSTRUM               spect
PROBHD    5 mm BBO BB-1H
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     3072
DS                        4
SWH              18028.846 Hz
FIDRES            0.275098 Hz
AQ                1.8175317 sec
RG                      724
DW                   27.733 usec
DE                    6.50 usec
TE                   300.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            75.4752949 MHz
NUC1                   13C
P1                    9.47 usec
PLW1           38.00000000 W

======== CHANNEL f2 ========
SFO2           300.1312005 MHz
NUC2                   1H
CPDPRG[2            waltz16
PCPD2                90.00 usec
PLW2           10.69999981 W
PLW12           0.22325000 W
PLW13           0.11229000 W

F2 - Processing parameters
SI                    32768
SF              75.4677485 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

Qualitative Compound Identification Report

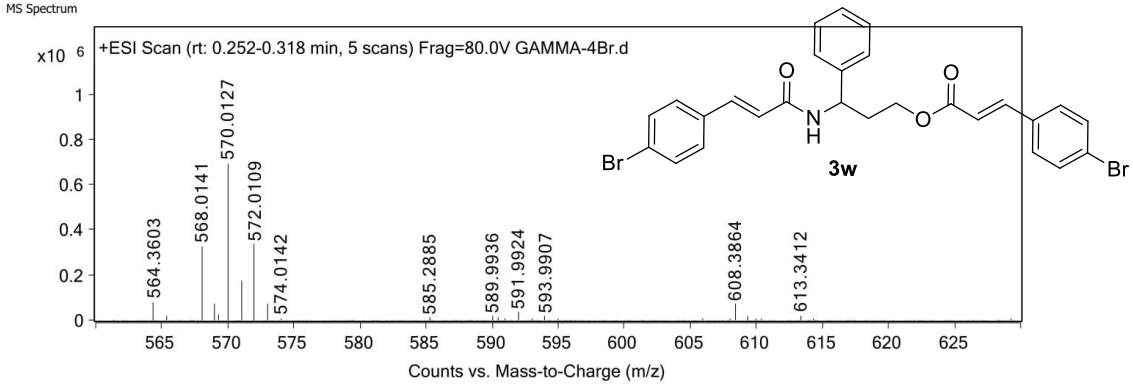
Data File	GAMMA-4Br.d	**Sample Name**	070319-35-EXT-GAMMA-4Br
Sample Type	Sample	**Position**	P1-D5
Instrument Name	Instrument 1	**User Name**	
Acq Method	Direct Infusion_HPLC.m	**Acquired Time**	07-03-2019 14:19:22 (UTC+05:30)
IRM Calibration Status	Success	**DA Method**	Default.m
Comment			
Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition Time (Local)**	07-03-2019 14:19:22 (UTC+05:30)
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.08.00 (B8058.0)	**QTOF Driver Version**	8.00.00
QTOF Firmware Version	20.698	**Tune Mass Range Max.**	3200

Compound Table

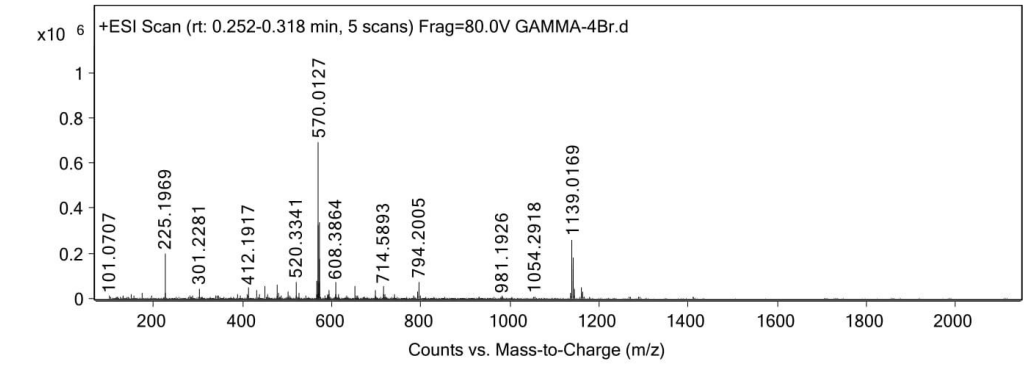
Compound Label	RT	Mass
Cpd 1: 0.269	0.269	
Cpd 2: 0.467	0.467	

Compound Label	RT	Algorithm
Cpd 1: 0.269	0.269	Spectrum Extraction

MS Spectrum

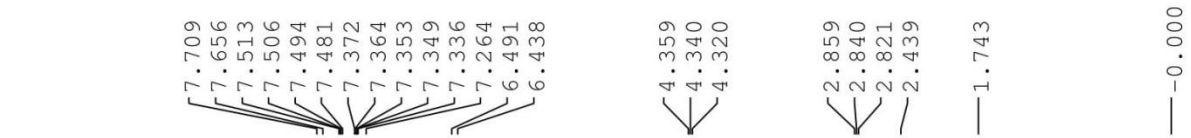


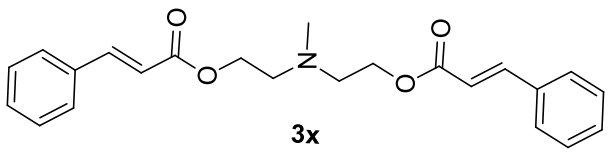
MS Zoomed Spectrum



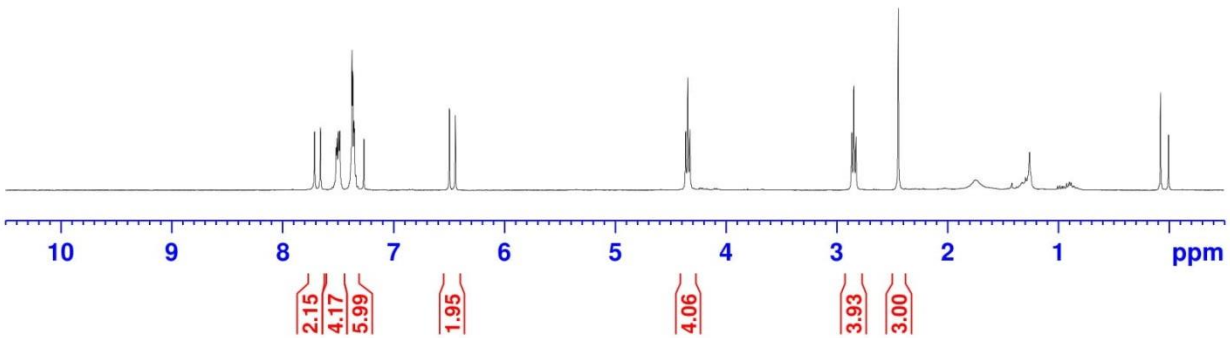
MS Spectrum Peak List

m/z	z	Abund
225.1969	1	200764.67
564.3603	1	81158.5
568.0141	1	327633.81
570.0127	1	692811.69
571.015	1	174667.17
572.0109	1	335541.81
1137.0173	1	150325.94





3x




```
Current Data Parameters
NAME            H25552I18
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20190301
Time                16.48
INSTRUM             spect
PROBHD    5 mm BBO BB-1H
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              6009.615 Hz
FIDRES           0.091699 Hz
AQ               5.4525952 sec
RG                    181
DW                 83.200 usec
DE                  6.50 usec
TE                 298.0 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          300.1318534 MHz
NUC1                   1H
P1                  13.00 usec
PLW1          10.69999981 W

F2 - Processing parameters
SI                  65536
SF            300.1300056 MHz
WDW                    EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```


```
Current Data Parameters
NAME            C3339B1
EXPNO                 1
PROCNO                1

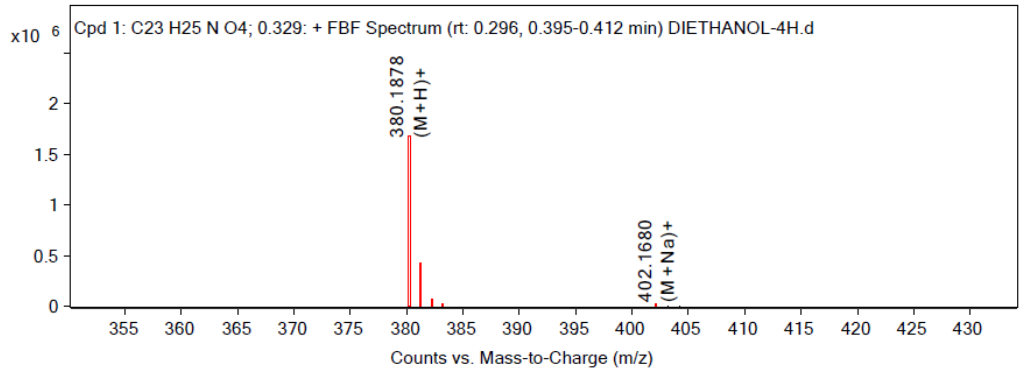
F2 - Acquisition Parameters
Date_          20190315
Time              21.02
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3072
DS                    4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ            1.8175317 sec
RG                  724
DW               27.733 usec
DE                6.50 usec
TE               300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         75.4752949 MHz
NUC1                13C
P1                 9.47 usec
PLW1        38.00000000 W

======== CHANNEL f2 ========
SFO2        300.1312005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.69999981 W
PLW12        0.22325000 W
PLW13        0.11229000 W

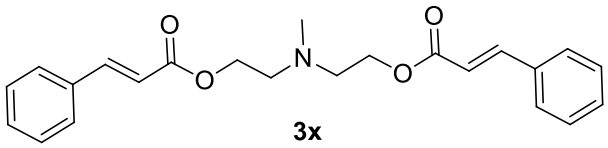
F2 - Processing parameters
SI                32768
SF           75.4677485 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Qualitative Compound Identification Report



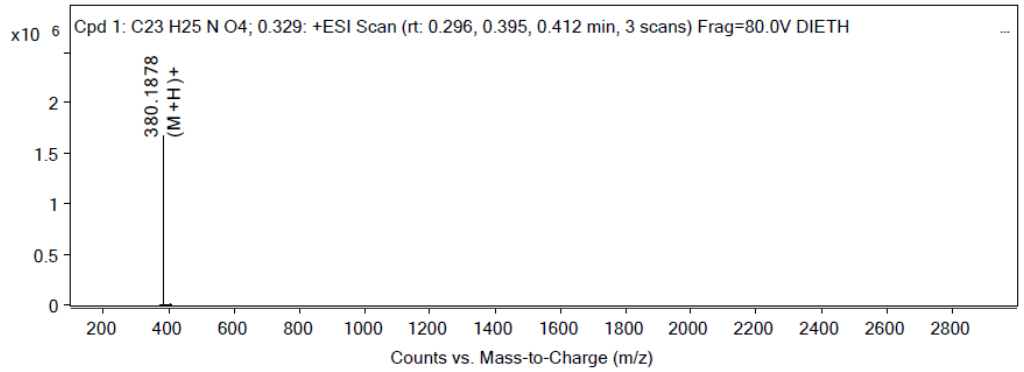
MS Spectrum Peak List

m/z	z	Abund	Ion
380.1878	1	1672459.63	(M+H)+
381.1909	1	383206.41	(M+H)+
382.193	1	41640.9	(M+H)+
383.1954	1	4396.21	(M+H)+
402.168	1	13357.99	(M+Na)+
403.1708	1	3175	(M+Na)+
404.1752	1	491.79	(M+Na)+

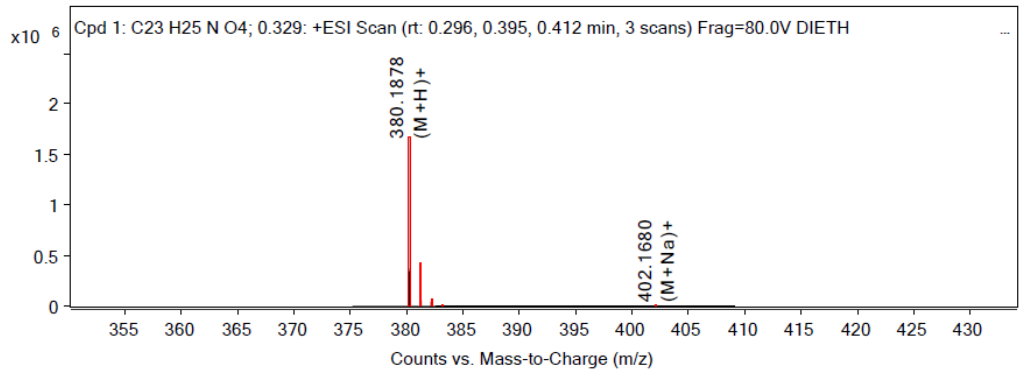


3x

MS Spectrum

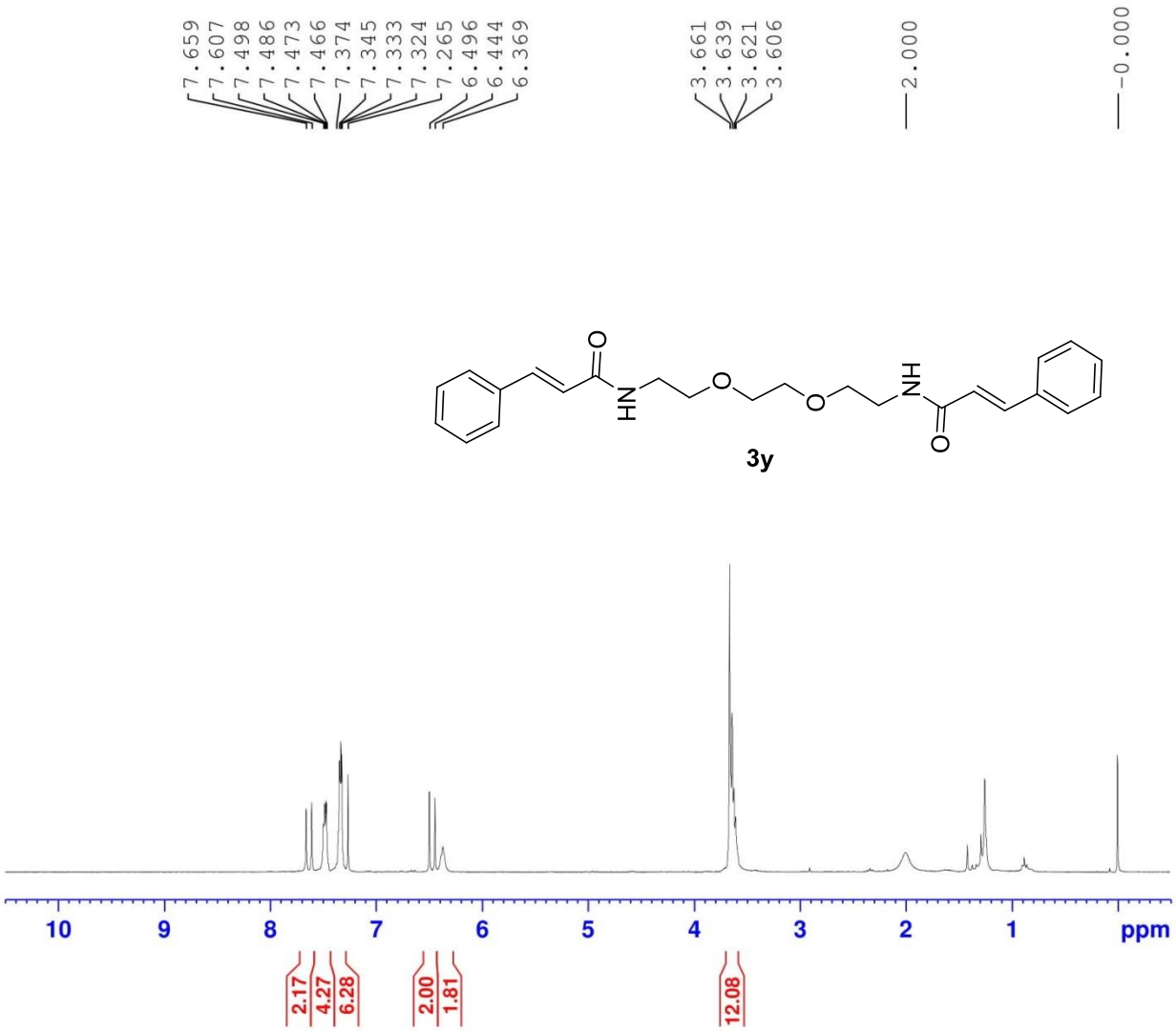


MS Zoomed Spectrum



MS Spectrum Peak List

m/z	Calc m/z	Diff(ppm)	z	Abund	Ion
380.1878	380.1856	-5.67	1	1672459.63	(M+H)+
380.1878				1672434.4	




```
Current Data Parameters
NAME            H2552I20
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190301
Time               17.16
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4525952 sec
RG                   287
DW                83.200 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                1H
P1                13.00 usec
PLW1         10.69999981 W

F2 - Processing parameters
SI                 65536
SF           300.1300056 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```


```
Current Data Parameters
NAME                 C3339A6
EXPNO                      1
PROCNO                     1

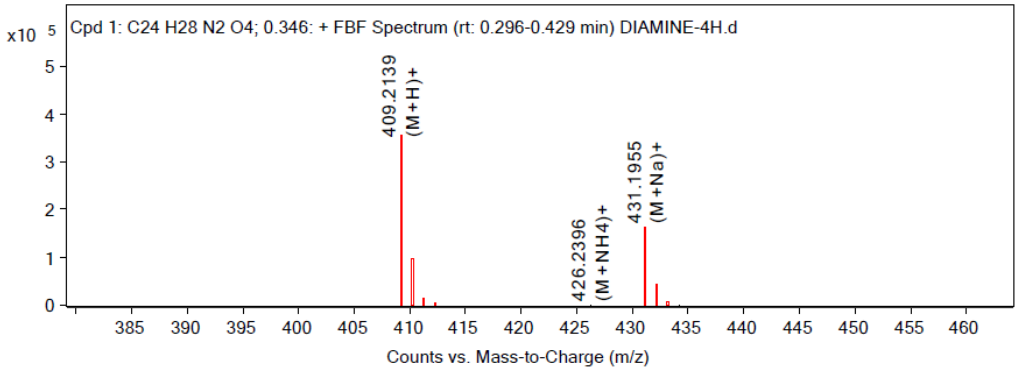
F2 - Acquisition Parameters
Date_               20190306
Time                   15.25
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG              zgpg30
TD                     65536
SOLVENT                CDCl3
NS                      2048
DS                         4
SWH                18028.846 Hz
FIDRES             0.275098 Hz
AQ                 1.8175317 sec
RG                       724
DW                    27.733 usec
DE                     6.50 usec
TE                    300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1               75.4752949 MHz
NUC1                      13C
P1                       9.47 usec
PLW1              38.00000000 W

======== CHANNEL f2 ========
SFO2              300.1312005 MHz
NUC2                      1H
CPDPRG[2              waltz16
PCPD2                  90.00 usec
PLW2              10.69999981 W
PLW12              0.22325000 W
PLW13              0.11229000 W

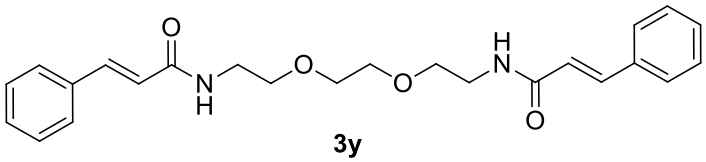
F2 - Processing parameters
SI                       32768
SF                 75.4677485 MHz
WDW                         EM
SSB       0
LB                       1.00 Hz
GB        0
PC                       1.40
```

Qualitative Compound Identification Report



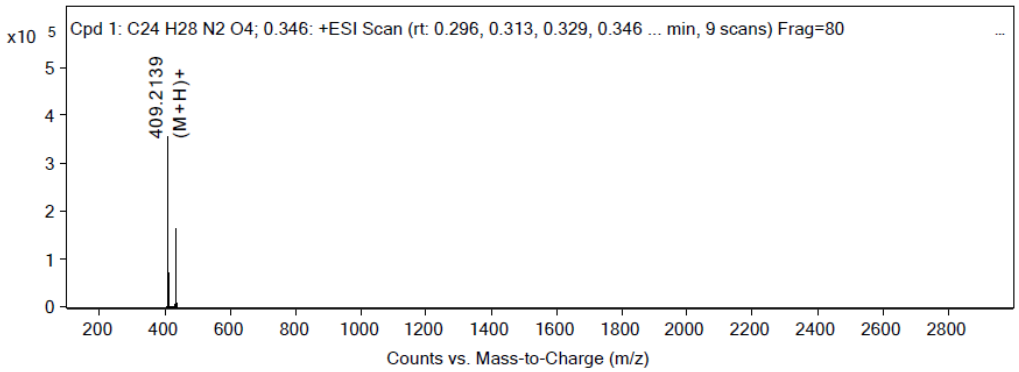
MS Spectrum Peak List

m/z	z	Abund	Ion
409.2139	1	354375.47	(M+H)+
410.2162	1	72250.06	(M+H)+
411.2196	1	10408.9	(M+H)+
412.2228	1	1292.17	(M+H)+
426.2396	1	312.73	(M+NH4)+
431.1955	1	163306.84	(M+Na)+
432.1981	1	34727.71	(M+Na)+
433.2018	1	5256.5	(M+Na)+
434.206	1	732.08	(M+Na)+

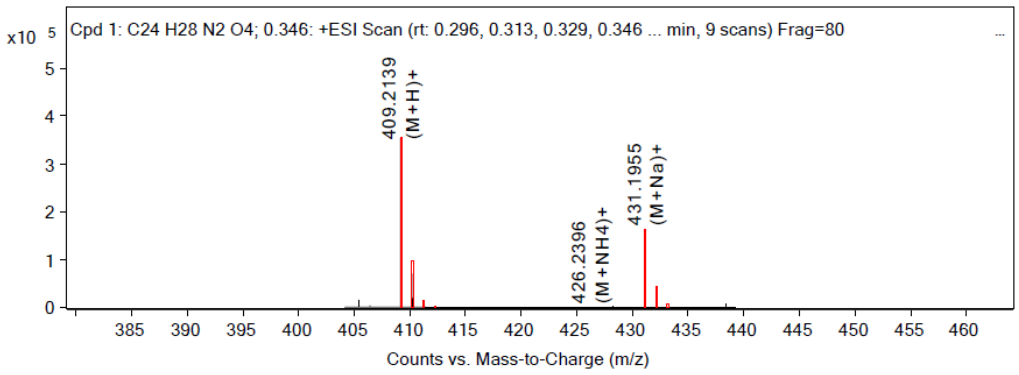


3y

MS Spectrum



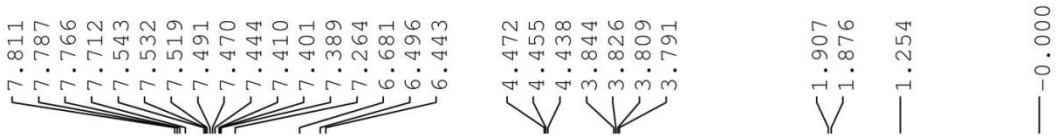
MS Zoomed Spectrum

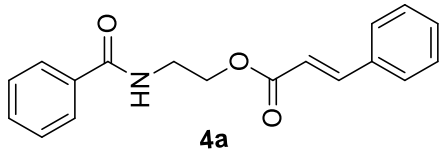


MS Spectrum Peak List

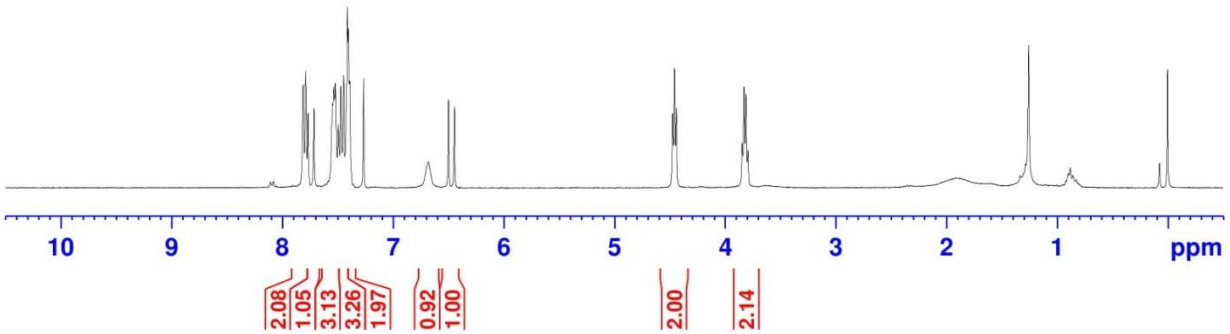
m/z	Calc m/z	Diff(ppm)	z	Abund	Ion

Agilent Technologies





4a

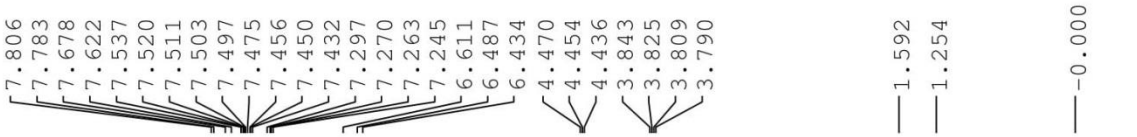


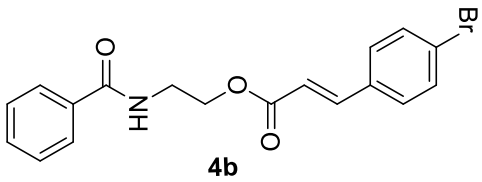

```
Current Data Parameters
NAME            H2552E19
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181010
Time               15.50
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4525952 sec
RG                   322
DW                83.200 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

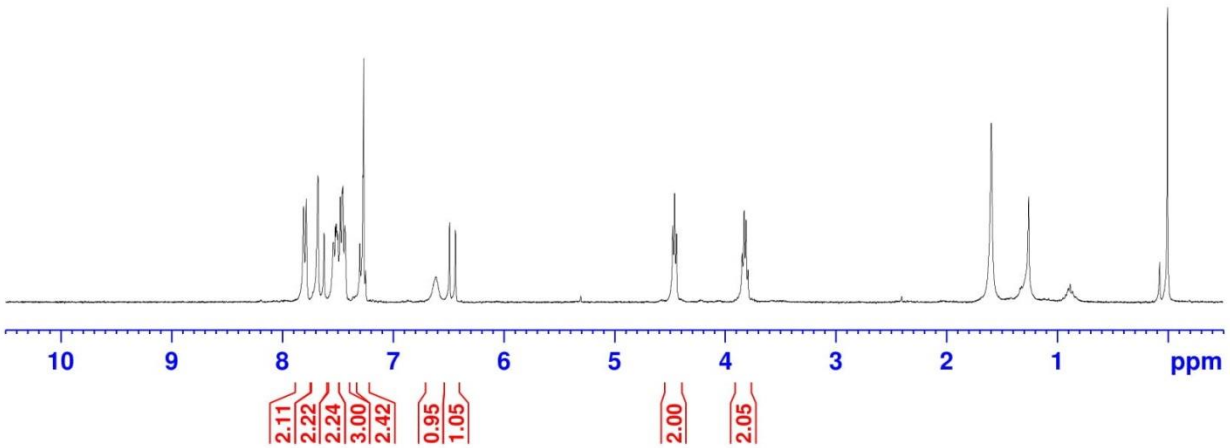
======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                1H
P1                13.00 usec
PLW1         10.69999981 W

F2 - Processing parameters
SI                 65536
SF           300.1300058 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```





4b

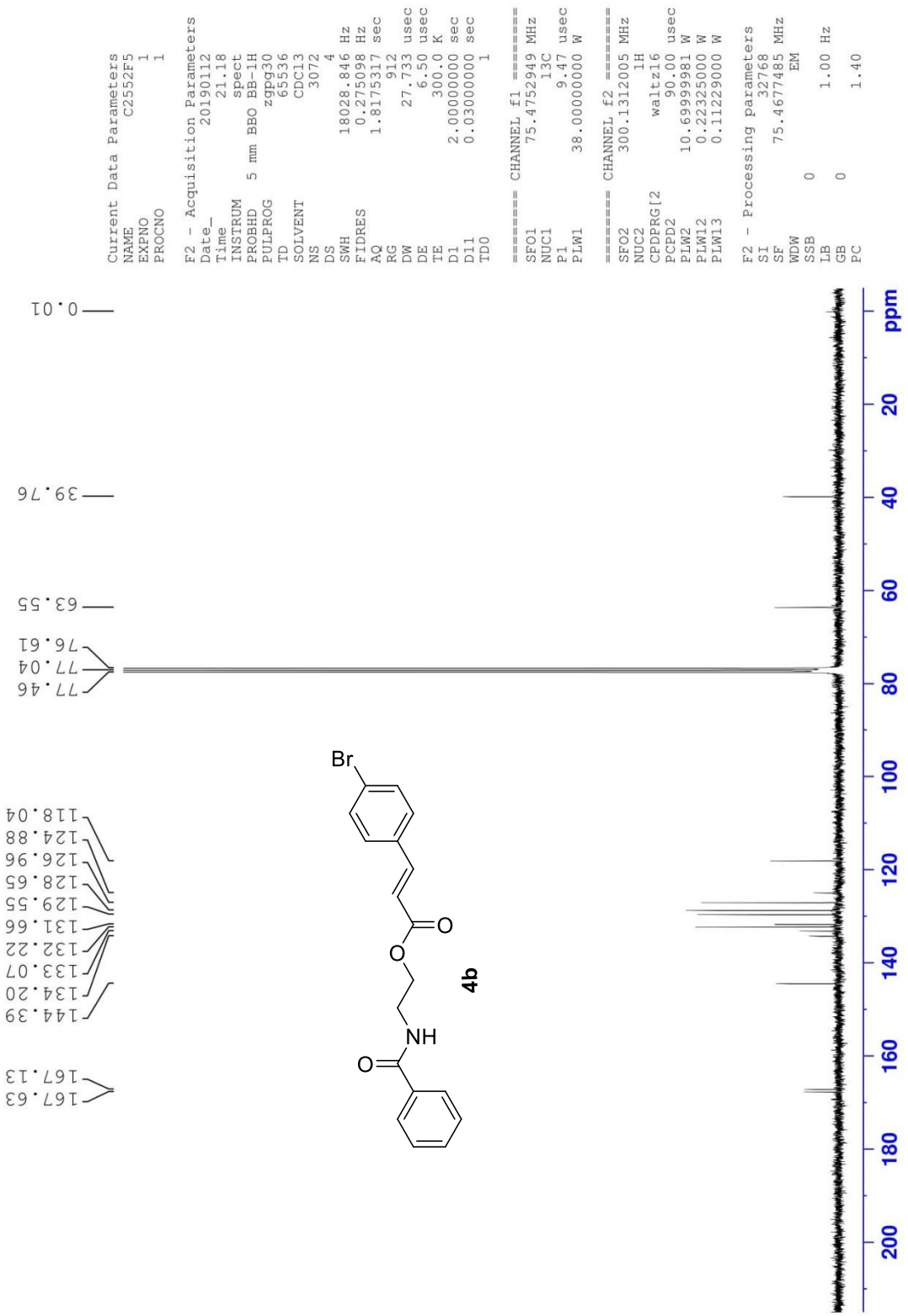



```
Current Data Parameters
NAME            H2552E17
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181009
Time               16.47
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4525952 sec
RG                   406
DW                83.200 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                1H
P1                13.00 usec
PLW1         10.69999981 W

F2 - Processing parameters
SI                 65536
SF           300.1300057 MHz
WDW                  EM
SSB       0
LB              0.30 Hz
GB        0
PC                 1.00
```


Current Data Parameters
NAME C2552F5
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190112
Time_ 21.18
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3072
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81175317 sec
RG 912
DW 27.733 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 75.4752949 MHz
NUC1 13C
P1 9.47 usec
PLW1 38.00000000 W
======== CHANNEL f2 ========
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 10.69999981 W
PLW12 0.22325000 W
PLW13 0.11229000 W
F2 - Processing parameters
SI 32768
SF 75.4677485 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
0.01
39.76
63.55
77.46
77.04
76.61
118.04
124.88
126.96
128.65
129.55
131.66
132.22
133.07
134.20
144.39
167.13
167.63
Br
O
O
NH
O
4b
ppm
200
180
160
140
120
100
80
60
40
20

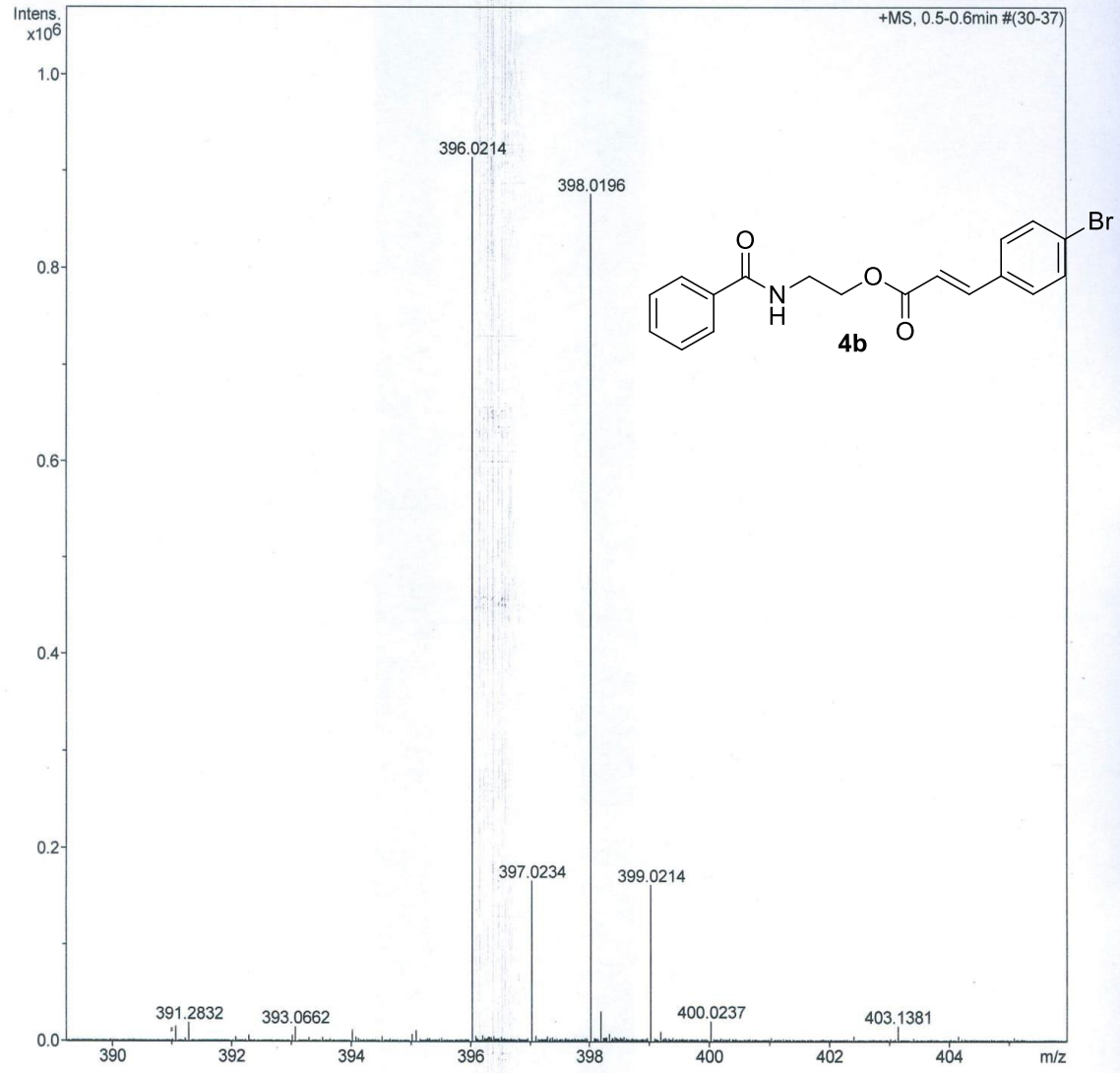
UOH -SCHOOL OF CHEMISTRY -HRMS

Analysis Info

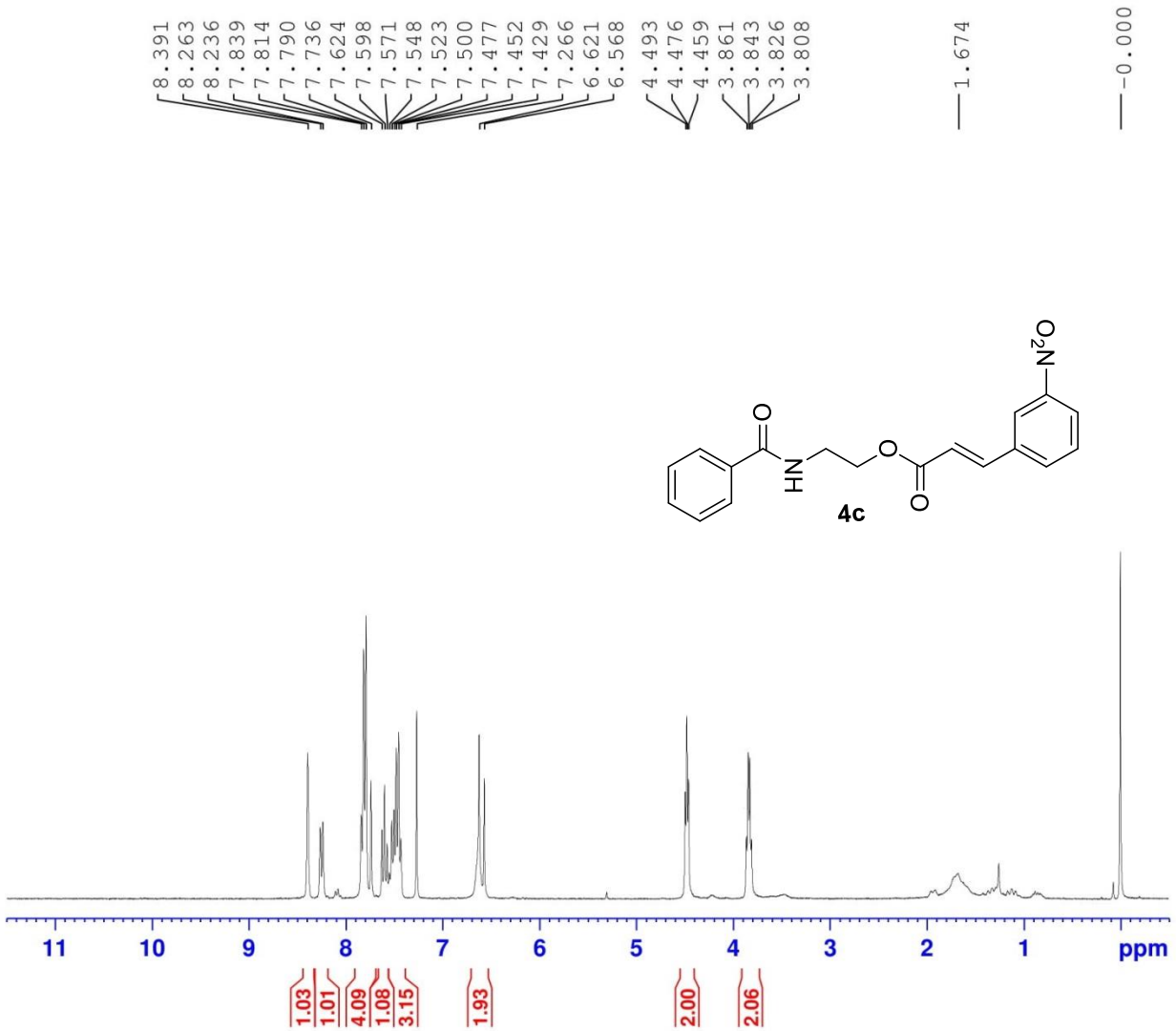
Analysis Name	D:\Data\2018\PROF MP\DEC\10r.d	Acquisition Date	12/12/2018 1:44:54 PM
Method	tune_low_PosR.m	Operator	UOH-Chemistry
Sample Name	-10-DCM-MEOH	Instrument	maXis 10138
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active	Set Capillary	3800 V	Set Dry Heater	250 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	5.0 l/min
Scan End	2500 m/z	Set Collision Cell RF	350.0 Vpp	Set Divert Valve	Waste



Bruker Compass DataAnalysis 4.0 printed: 12/12/2018 1:48:07 PM Page 1 of 1

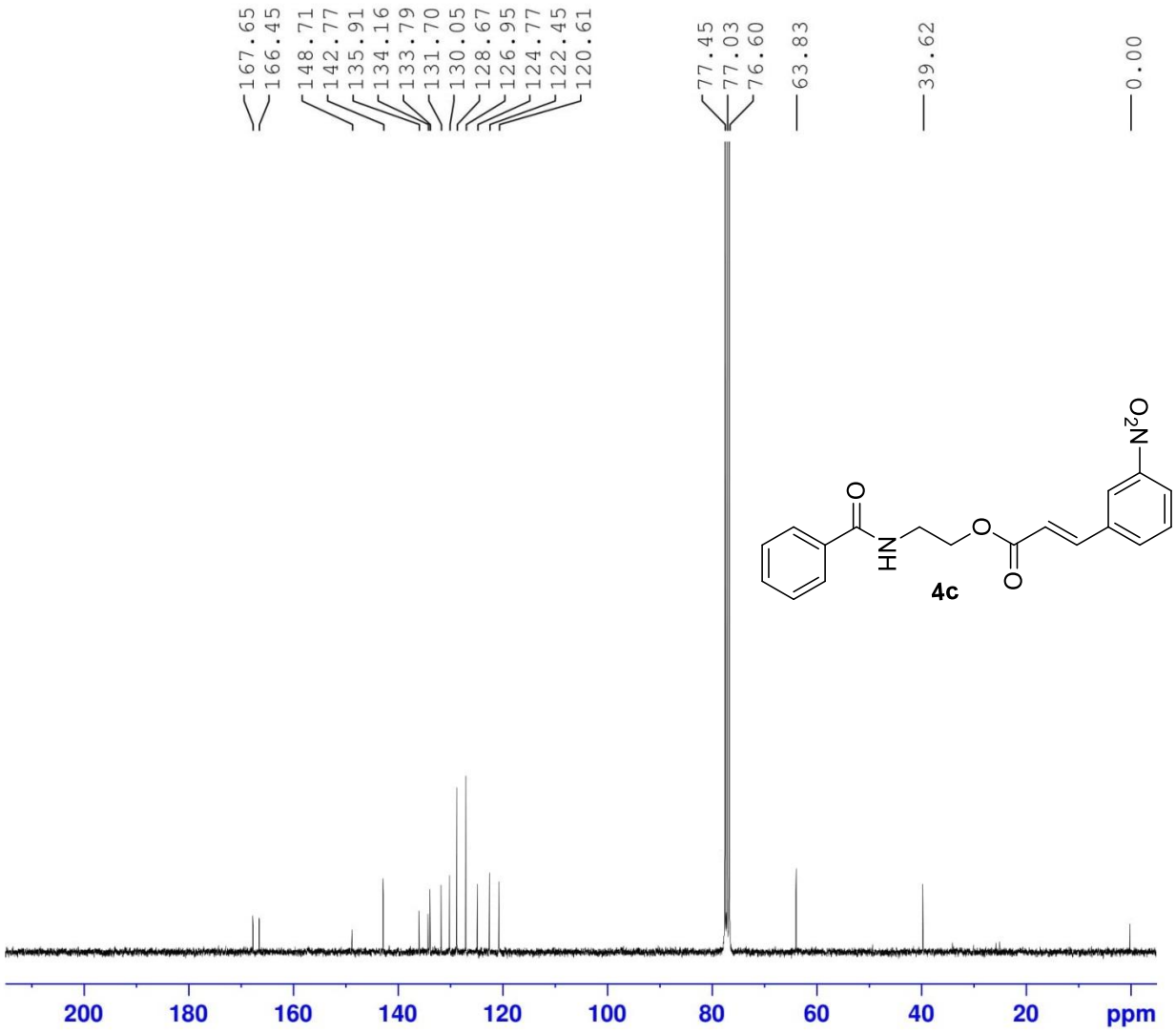



```
Current Data Parameters
NAME            H2552F10
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181102
Time               15.15
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4525952 sec
RG                   228
DW                83.200 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                1H
P1                13.00 usec
PLW1         10.69999981 W

F2 - Processing parameters
SI                 65536
SF           300.1300052 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```




```
Current Data Parameters
NAME            C2552E6-R
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20181101
Time                20.45
INSTRUM             spect
PROBHD    5 mm BBO BB-1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3072
DS                     4
SWH            18028.846 Hz
FIDRES          0.275098 Hz
AQ             1.8175317 sec
RG                   912
DW                27.733 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                  13C
P1                   9.47 usec
PLW1        38.00000000 W

======== CHANNEL f2 ========
SFO2         300.1312005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2         10.69999981 W
PLW12         0.22325000 W
PLW13         0.11229000 W

F2 - Processing parameters
SI                 32768
SF            75.4677485 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

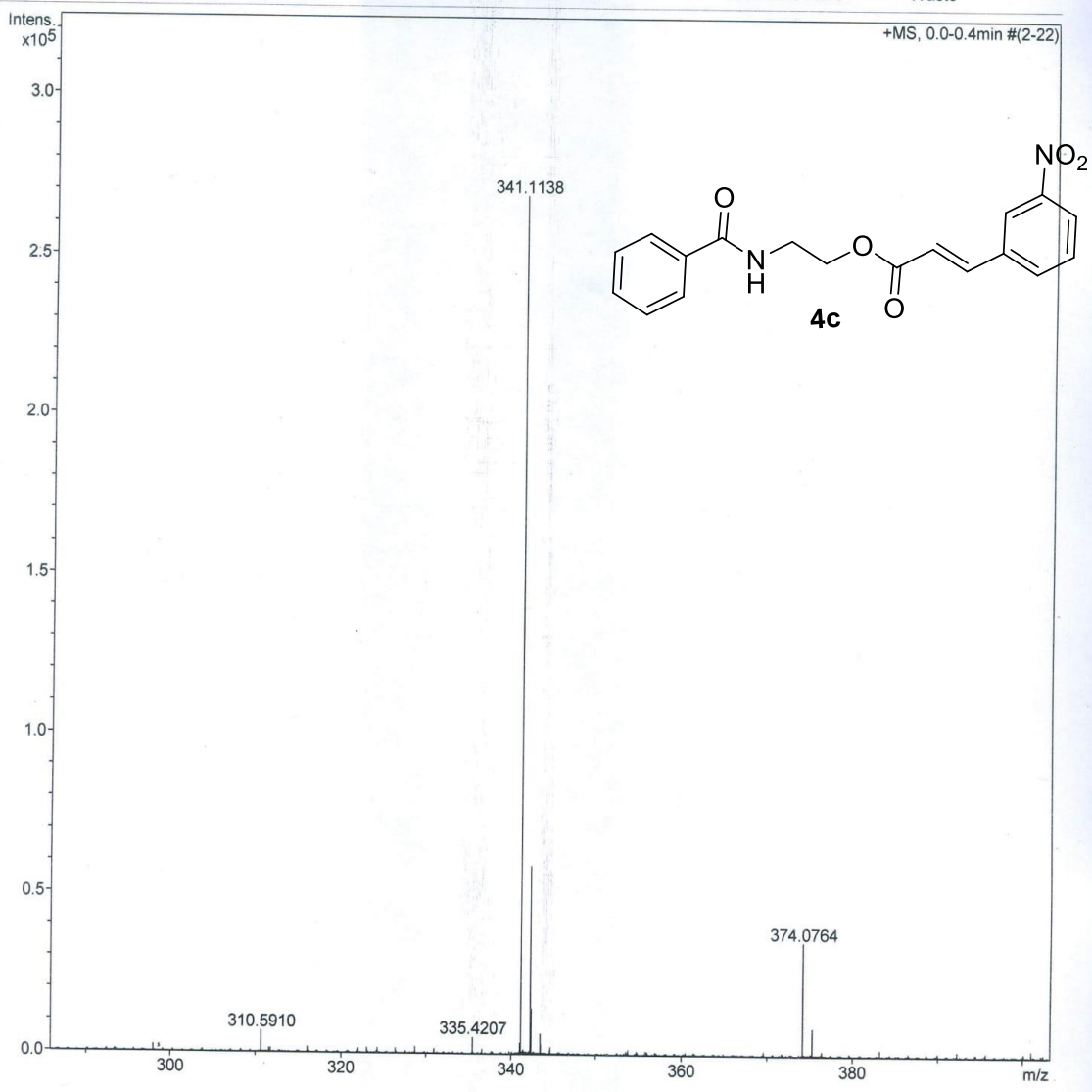
UOH -SCHOOL OF CHEMISTRY -HRMS

Analysis Info

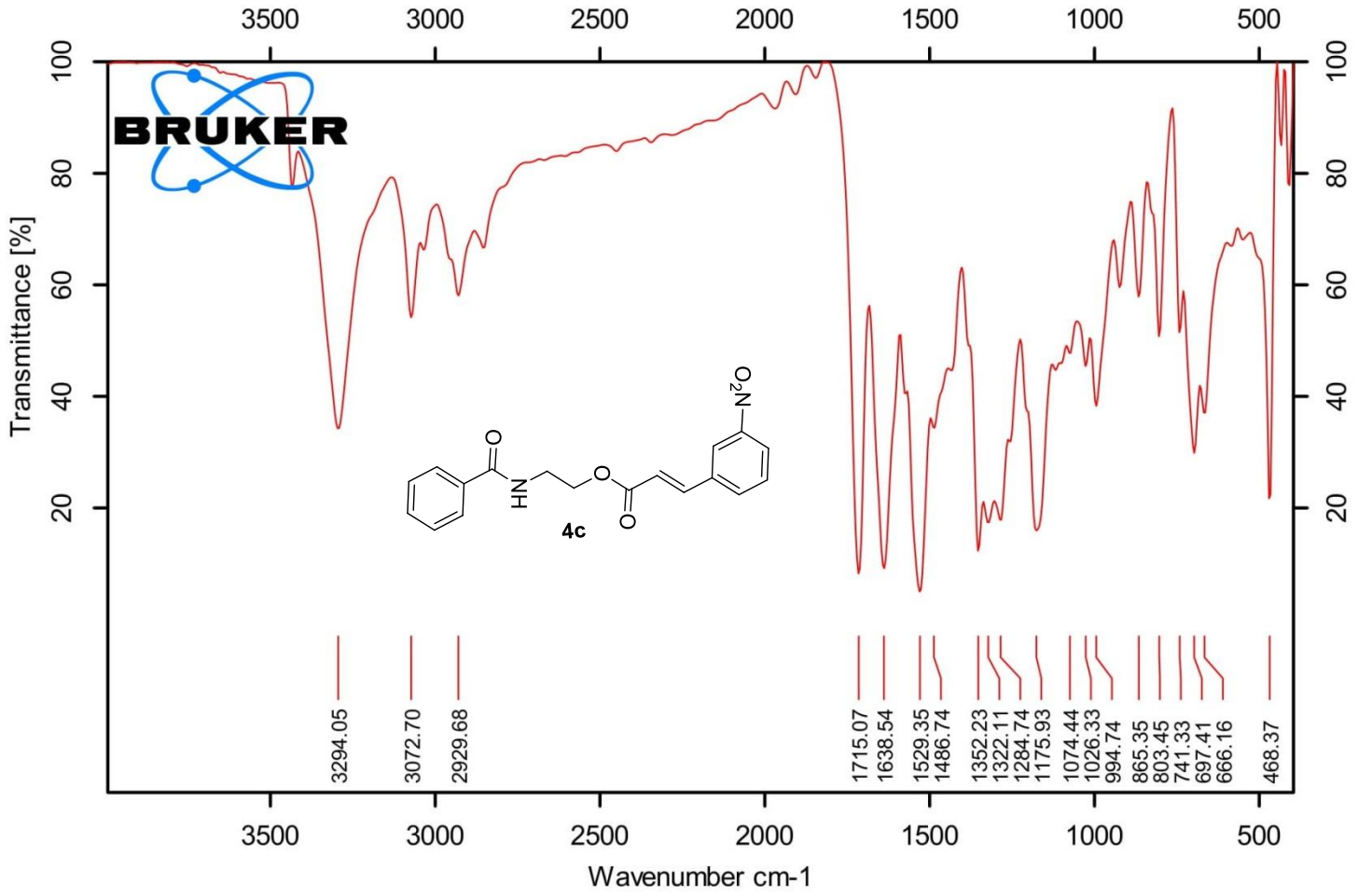
Analysis Name	D:\Data\2018\PROF MP\DEC\11.d	Acquisition Date	12/11/2018 4:38:54 PM
Method	tune_low.m		
Sample Name	11	Operator	UOH-Chemistry
Comment		Instrument	maXis 10138

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3800 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1800 m/z	Set Collision Cell RF	350.0 Vpp	Set Divert Valve	Waste

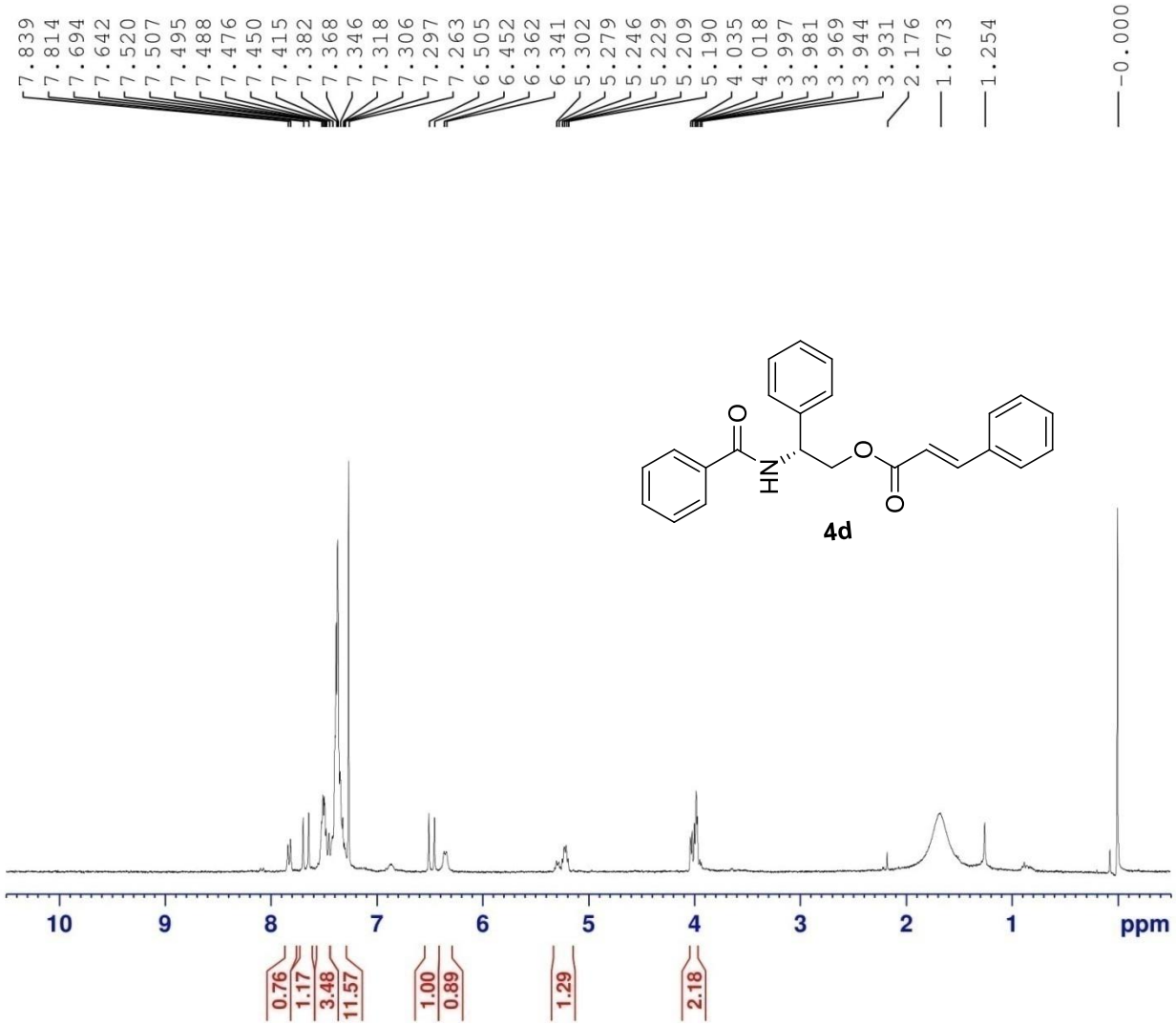


Bruker Compass DataAnalysis 4.0 printed: 12/11/2018 5:17:47 PM Page 1 of 1



SAMPLE SCANS : 48
DATE : 11-03-2019
TIME : 18:39:31

SAMPLE : PVS 1
TECHNIQUE : Thinfilm
USER : Default

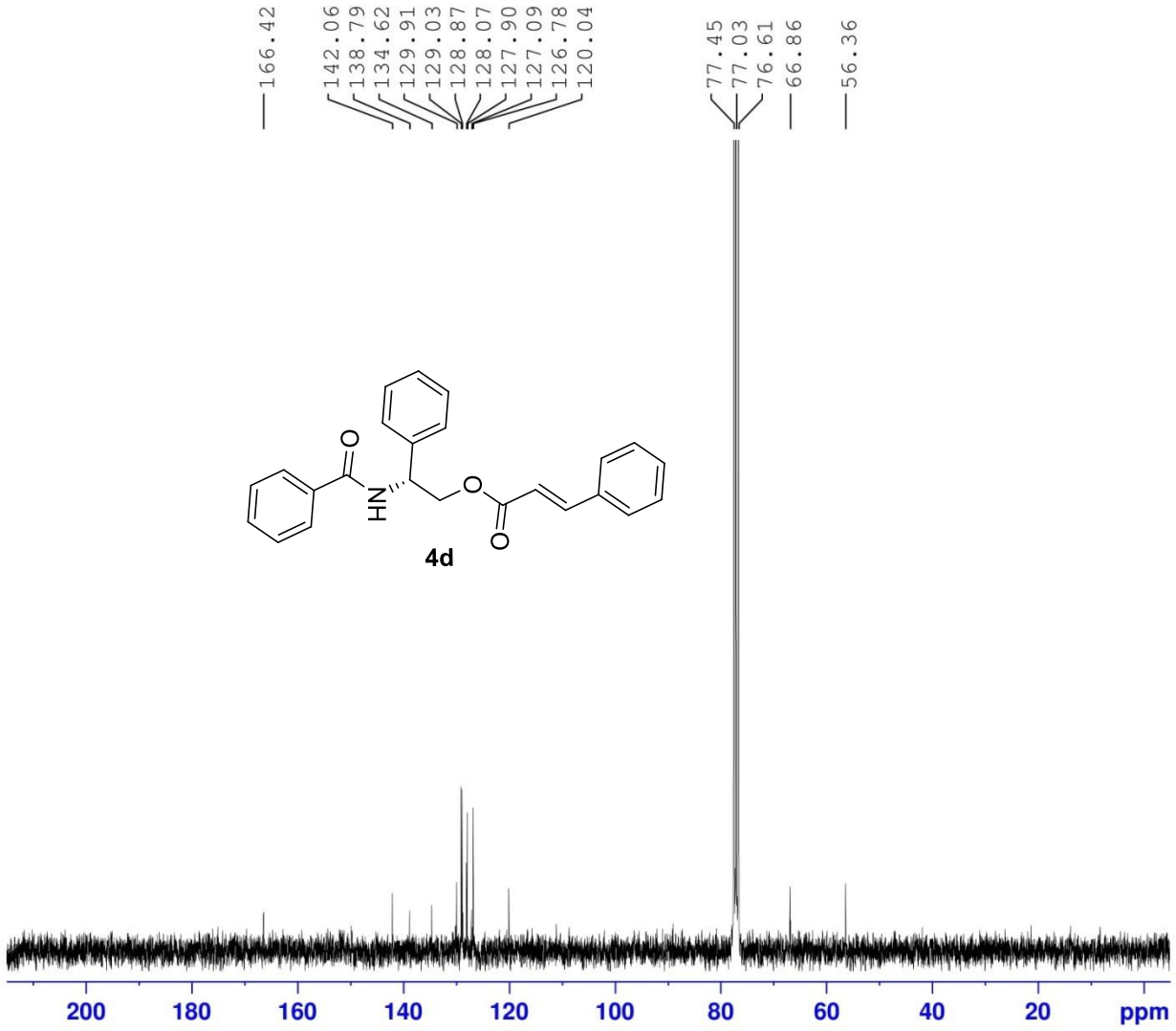



```
Current Data Parameters
NAME            H2552F3b
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181016
Time               11.58
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4525952 sec
RG                   406
DW                83.200 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                1H
P1                13.00 usec
PLW1         10.69999981 W

F2 - Processing parameters
SI                 65536
SF           300.1300056 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME            C2552E9
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20181029
Time              17.06
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1038
DS                    4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ            1.8175317 sec
RG                  912
DW               27.733 usec
DE                6.50 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         75.4752949 MHz
NUC1                13C
P1                 9.47 usec
PLW1        38.00000000 W

======== CHANNEL f2 ========
SFO2        300.1312005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.69999981 W
PLW12        0.22325000 W
PLW13        0.11229000 W

F2 - Processing parameters
SI                32768
SF           75.4677485 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

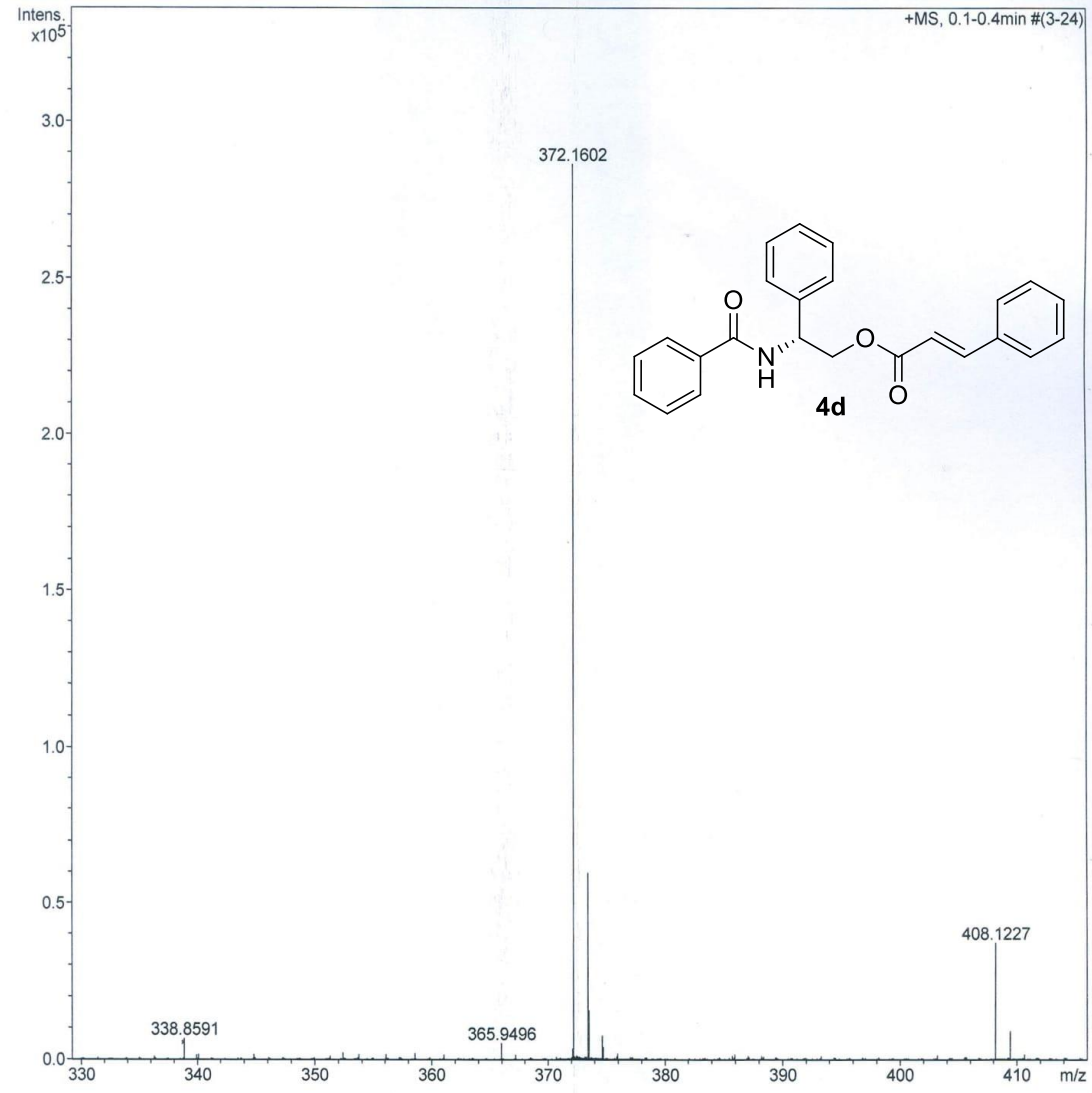
UOH -SCHOOL OF CHEMISTRY -HRMS

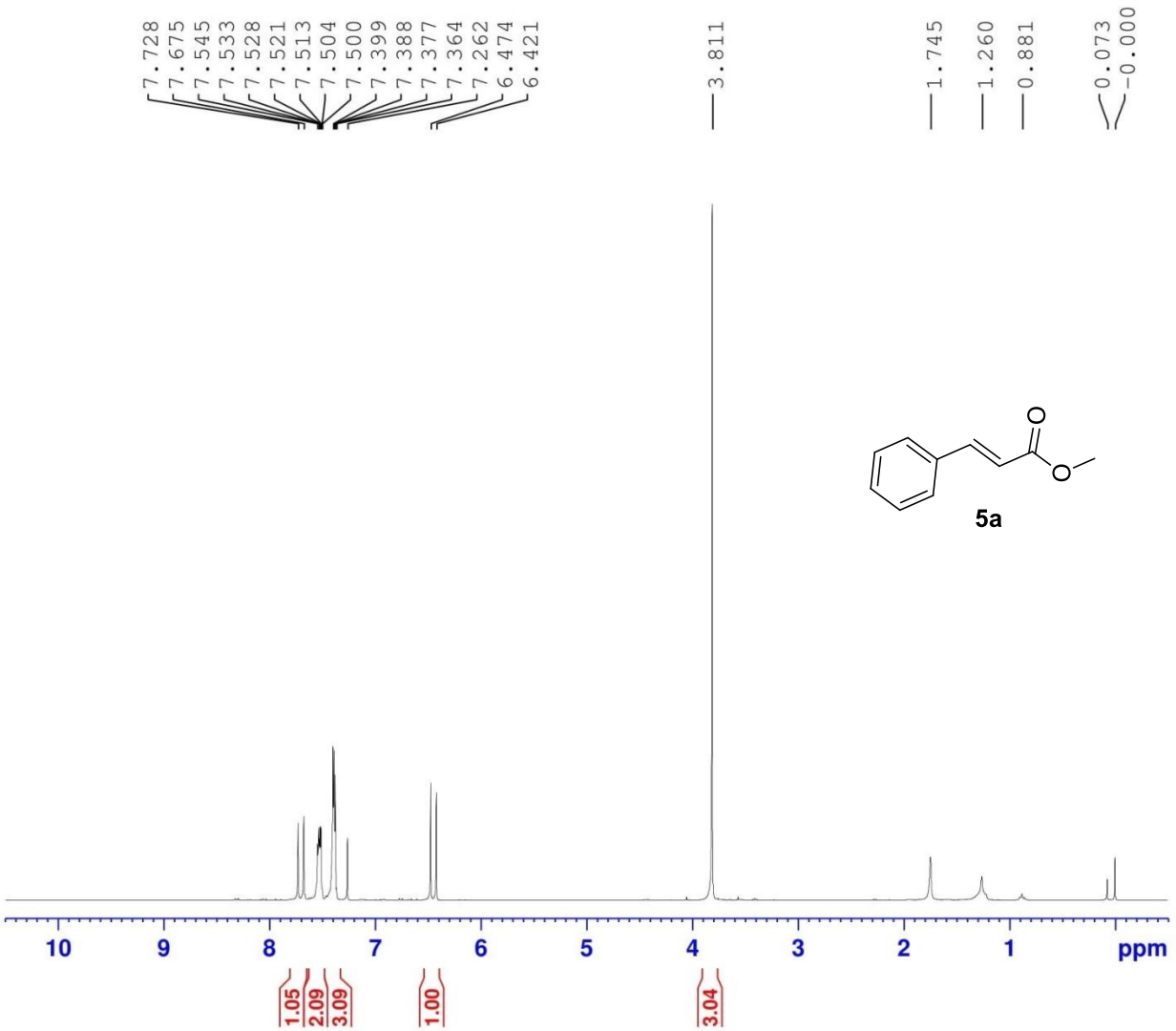
Analysis Info

Analysis Name	D:\Data\2018\PROF MP\DEC\12.d	Acquisition Date	12/11/2018 4:39:23 PM
Method	tune_low.m	Operator	UOH-Chemistry
Sample Name	12	Instrument	maXis 10138
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3800 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1800 m/z	Set Collision Cell RF	350.0 Vpp	Set Divert Valve	Waste





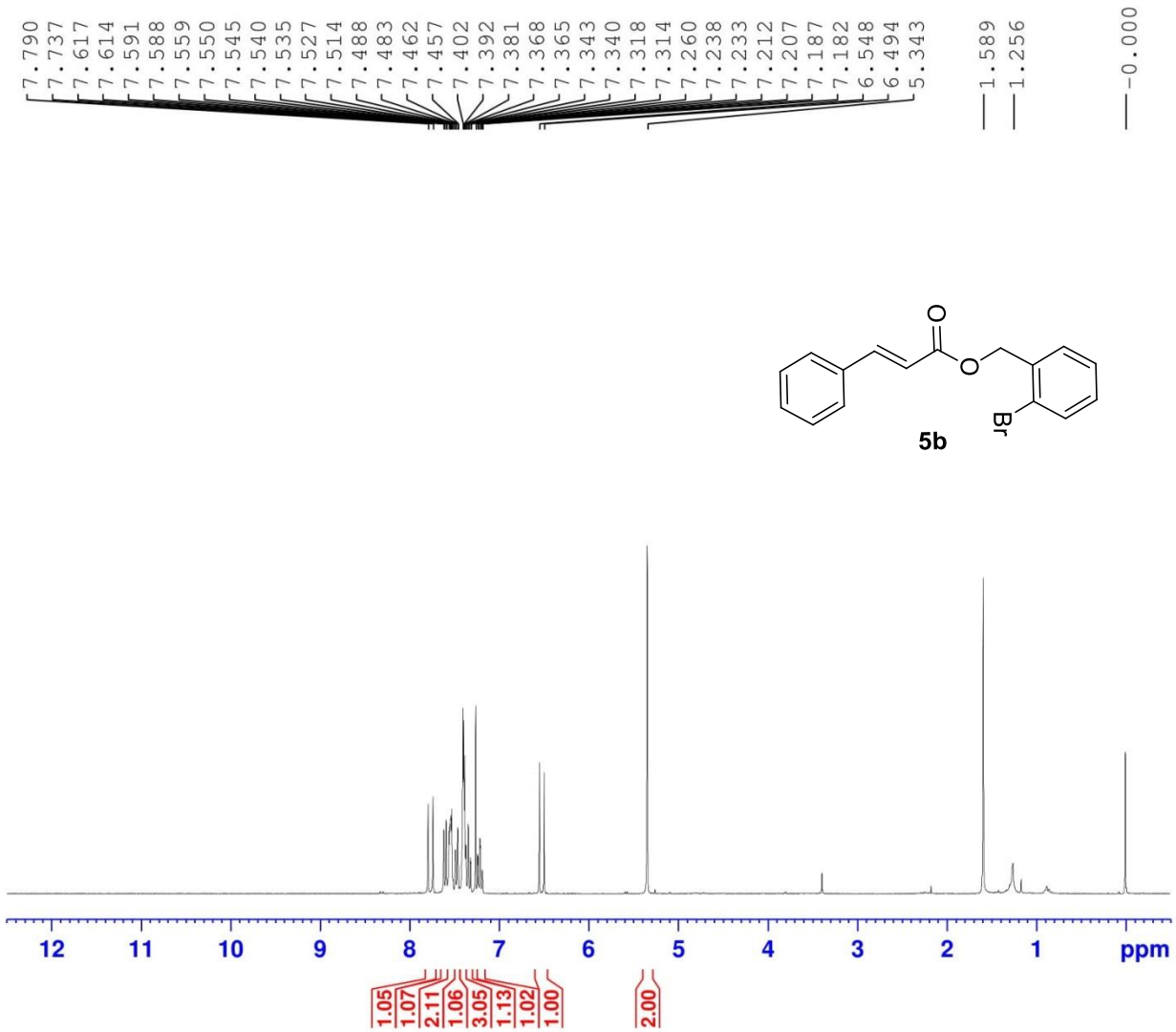
5a

```
Current Data Parameters
NAME                  3296J
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20180212
Time                  17.04
INSTRUM               spect
PROBHD    5 mm BBO BB-1H
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                6009.615 Hz
FIDRES            0.091699 Hz
AQ                5.4525952 sec
RG                      144
DW                   83.200 usec
DE                    6.50 usec
TE                   298.0 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           300.1318534 MHz
NUC1                    1H
P1                   13.00 usec
PLW1           10.69999981 W

F2 - Processing parameters
SI                    65536
SF             300.1300059 MHz
WDW                     EM
SSB          0
LB                    0.30 Hz
GB           0
PC                    1.00
```



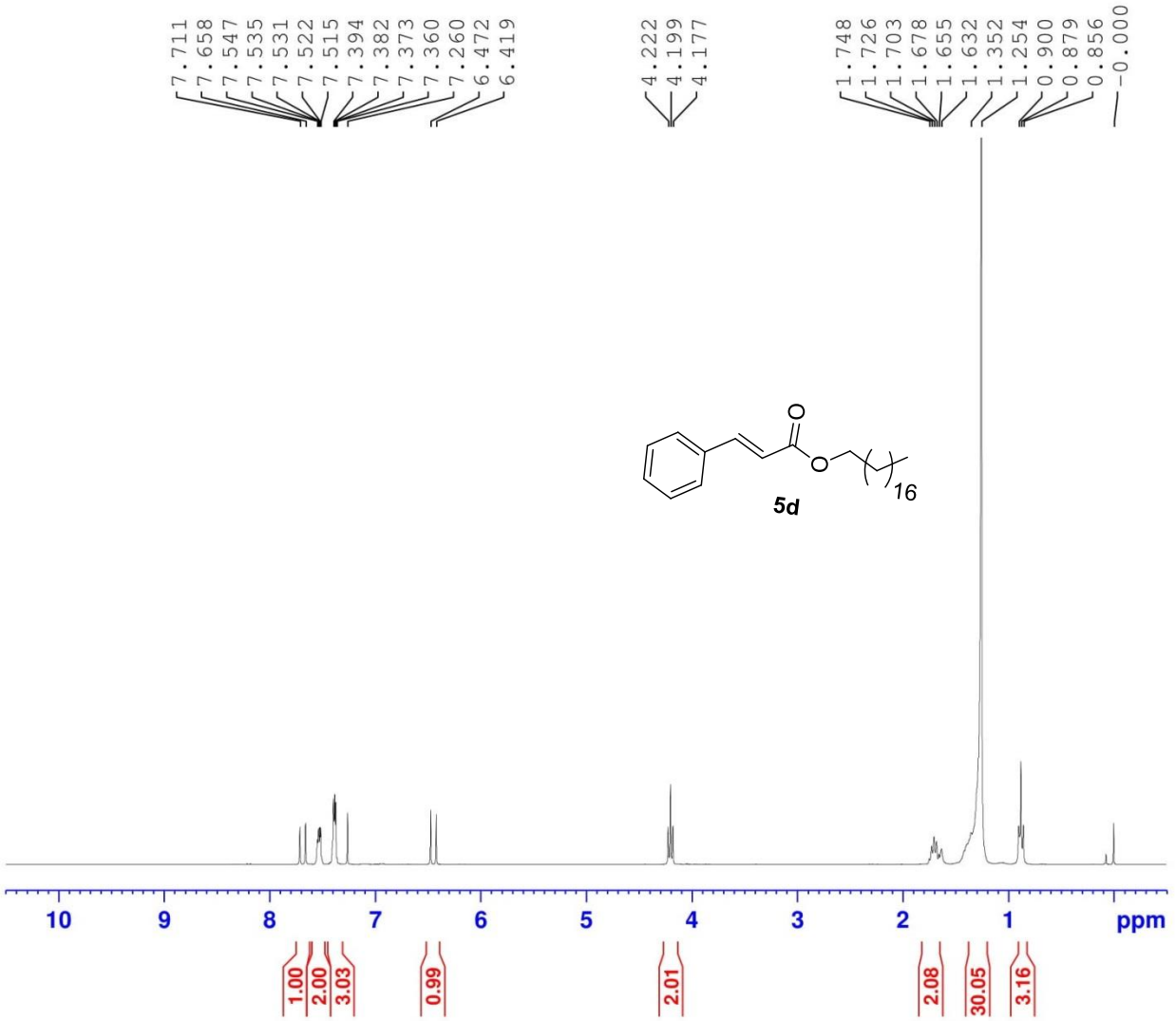

```
Current Data Parameters
NAME          3423A2_H1
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20180320
Time              19.57
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            6009.615 Hz
FIDRES        0.091699 Hz
AQ            5.4525952 sec
RG                  181
DW               83.200 usec
DE                6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        300.1318534 MHz
NUC1                1H
P1                13.00 usec
PLW1        10.69999981 W

F2 - Processing parameters
SI                65536
SF          300.1300068 MHz
WDW                  EM
SSB       0
LB              0.30 Hz
GB        0
PC                1.00
```

Current Data Parameters
NAME 3580F
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180410
Time 15.47
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 406
DW 83.200 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 300.1318534 MHz
NUC1 1H
P1 13.00 usec
PLW1 10.69999981 W
F2 - Processing parameters
SI 65536
SF 300.1300060 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
5c
ppm
1.577
0.000
7.901
7.898
7.875
7.872
7.797
7.744
7.564
7.550
7.540
7.532
7.462
7.457
7.437
7.431
7.406
7.400
7.396
7.385
7.380
7.354
7.350
7.263
7.075
7.069
7.049
7.044
7.024
7.018
6.550
6.496
5.275
2.02
0.99
1.00
5.09
2.07
1.02
0.99

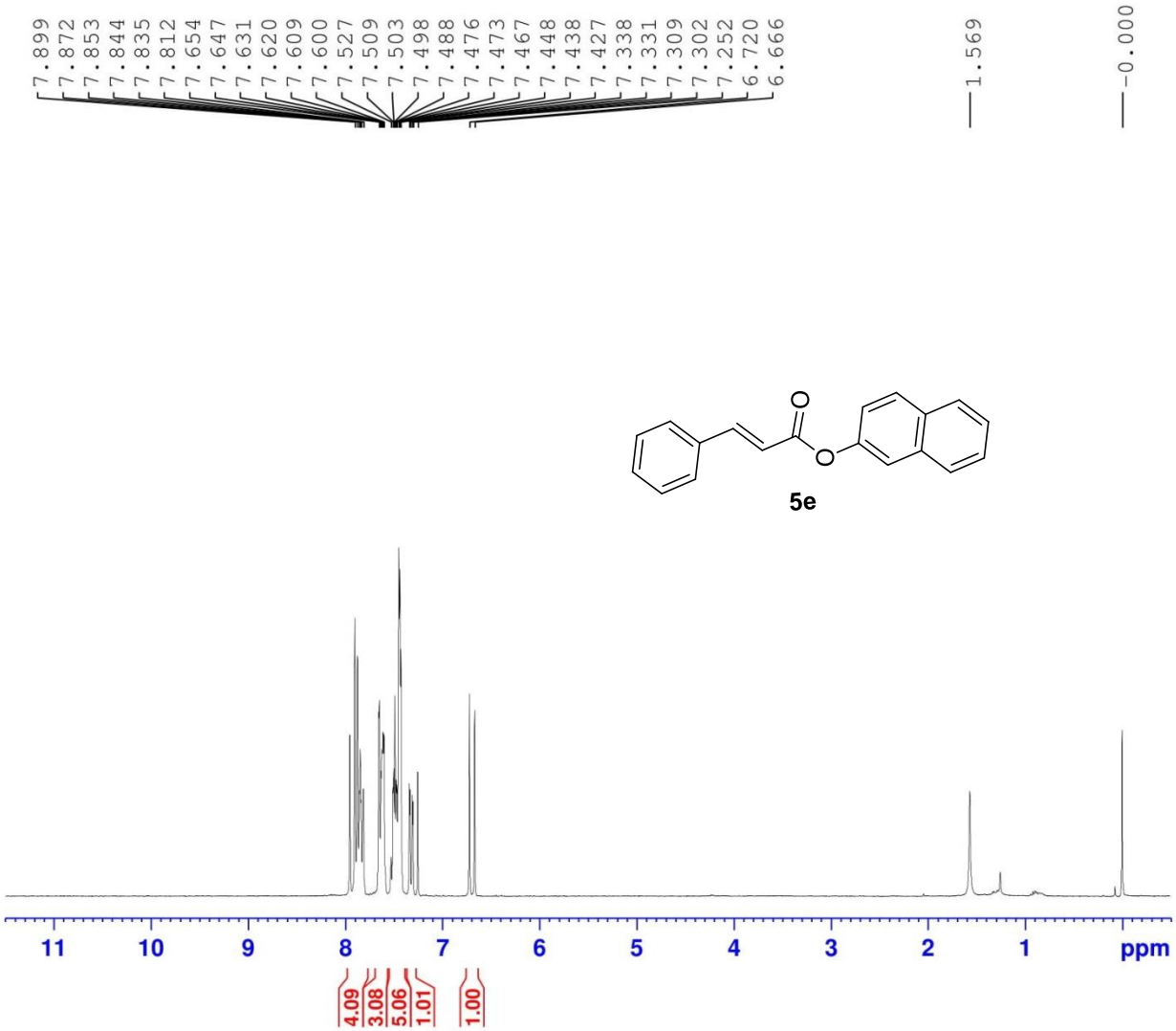



```
Current Data Parameters
NAME            3297D1_H1
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20180222
Time                17.01
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              6009.615 Hz
FIDRES           0.091699 Hz
AQ               5.4525952 sec
RG                    101
DW                 83.200 usec
DE                  6.50 usec
TE                 298.0 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          300.1318534 MHz
NUC1                   1H
P1                  13.00 usec
PLW1          10.69999981 W

F2 - Processing parameters
SI                  65536
SF            300.1300066 MHz
WDW                    EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```



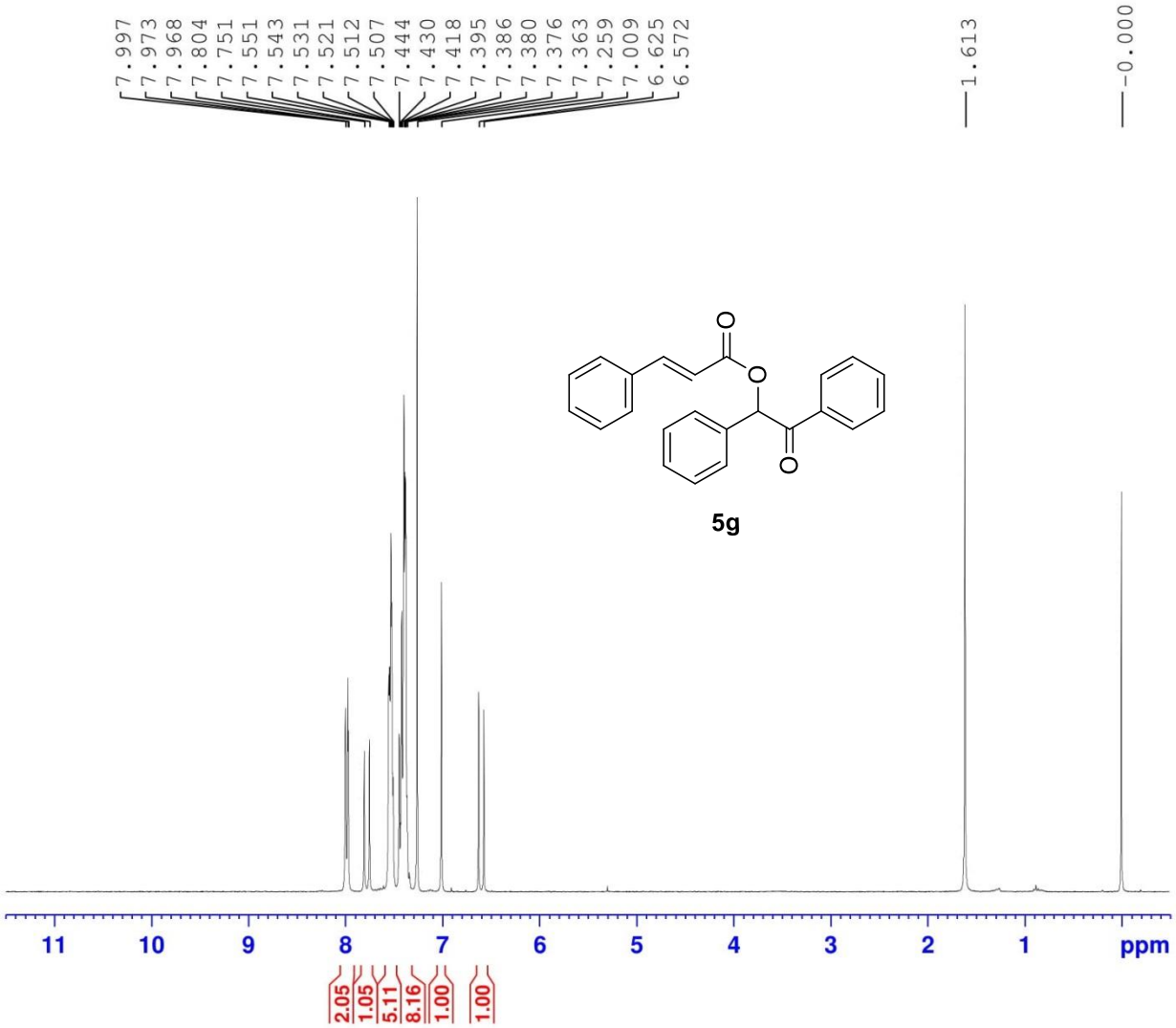

```
Current Data Parameters
NAME            H2552D13
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180910
Time               17.22
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4525952 sec
RG                   287
DW                83.200 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                1H
P1                13.00 usec
PLW1         10.69999981 W

F2 - Processing parameters
SI                 65536
SF           300.1300093 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Current Data Parameters
NAME H2552G9
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20181219
Time 17.23
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 181
DW 83.200 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 300.1318534 MHz
NUC1 1H
P1 13.00 usec
PLW1 10.69999981 W
F2 - Processing parameters
SI 65536
SF 300.1300083 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
5f
7.905
7.852
7.611
7.603
7.597
7.592
7.589
7.579
7.566
7.449
7.438
7.427
7.416
7.395
7.390
7.382
7.279
7.255
7.230
7.191
7.187
7.162
6.668
6.615
1.574
1.570
0.000
1.01
2.06
5.13
1.25
1.99
1.00
ppm



5g

```
Current Data Parameters
NAME            H2552J10
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190308
Time               17.10
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6009.615 Hz
FIDRES          0.091699 Hz
AQ              5.4525952 sec
RG                   322
DW                83.200 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                1H
P1                13.00 usec
PLW1         10.69999981 W

F2 - Processing parameters
SI                 65536
SF           300.1300070 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```