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A Distal Vinyl Shift through Quadruple Domino Reaction: Synthesis of *N*-vinyl Benzoheterocyclic Scaffolds

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Supplementary Information

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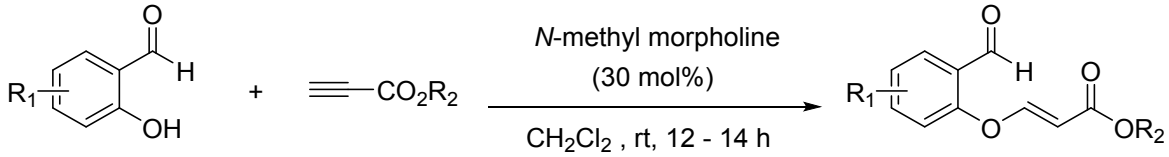
General Consideration:

Commercial reagents were used without further purification. IR spectra were recorded on a Perkin Elmer-FTIR spectrometer using solid samples as KBr plates. For compounds ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra were recorded in deuterochloroform with one drop of DMSO-d_6 (some compounds were recorded in pure DMSO-d_6)on a Bruker 400 MHz spectrometer using tetramethylsilane (TMS, $\delta = 0$) as an internal standard at room temperature. Mass spectra were recorded on Agilent 1200 LC/MS-6110 mass spectrometer. Aldehydes, propiolates and aminophenols were purchased from Sigma Aldrich. Compounds spectral data and copy of ^{1}H, ^{13}C NMR and ESI-HRMS spectra of all compounds **3a-s, 5a-e** and **7a-b** are listed below (pages 17-95).

Preparation of vinylogous carbonates (1)[1]



To a solution of salicyladehyde (1 equiv) in dichloromethane was added *N*-methyl morpholine (30 mol %) and the mixture stirred at room tempertaure for 10 minutes. Alkyl propiolates (1.1 equiv) were added and the reaction kept for stirring for 12-14h. The progress of the reaction was monitored by TLC. DCM was removed using rotary evaporator and the mixture was quenched by addition of water and 5% dilute HCl. The aqueous layer was extracted with ethyl acetate. The organic layer was washed with brine, dried over Na_2SO_4, filtrated, and concentrated under reduced pressure to give the crude product. The crude product was purified by silica gel column chromatography (60-120 mesh) [ethyl acetate / hexanes (0.5:9.5)] to give the final compound.

References

1. (a) V. Srinivas, M. Koketsu. *J. Org. Chem*. 2013, **78**, 11612-11617.

(b) L.-Q. Lu, F. Li, J. An, J.-J. Zhang, X.-L. An, Q.-L. Hua, W.-J. Xiao, *Angew. Chem., Int. Ed.* 2009, **48**, 9542.

Typical experimental procedure for the synthesis of compounds (3a-s) and (5a-e)

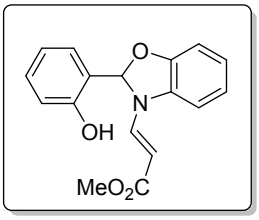
A mixture of methyl (*E*)-3-(2-formylphenoxy)acrylate (**1**, 1mmol), and 2-aminophenol or 2-aminothiophenol or 2-aminobenzylalcohol (**2** or **4,** 1 mmol) in acetonitrile (10 mL) was placed in a round bottom flask and stirred at room temperature for 2-4 h. The reaction was monitored by the TLC. After the completion of the reaction (based on the dissapearance of the starting material), the reaction was stopped and the precipitate was filtered and washed with ethylacetate : hexanes (0.5 : 9.5) to afford **3** or **5** as the final compound.

Typical experimental procedure for the synthesis of compounds (7a-b)

A mixture of dimethyl 2-((*N*-(2-formylphenyl)-4-methylphenyl)sulfonamido)fumarate (**6**, 1mmol), and 2-aminophenol (**2**, 1 mmol) in acetonitrile (10 mL) was placed in a round bottom flask and stirred at room temperature for 8h. The reaction was monitored by the TLC. After the completion of the reaction (based on the dissapearance of the starting material), the reaction was stopped and the precipitate was filtered and washed with ethylacetate : hexanes (0.5 : 9.5) to afford the final compound.

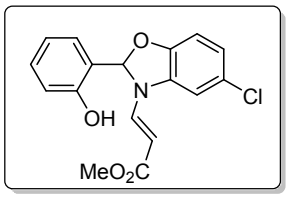
Analytical Data of the Products

Methyl (*E*)-3-(2-(2-hydroxyphenyl)benzo[d]oxazol-3(2H)-yl)acrylate (3a)



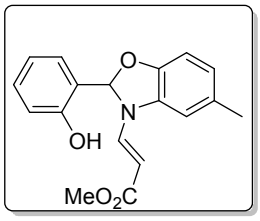
Yield : 80%; white solid; M.P. = 181-182 ˚C; Reaction time: 3 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 3.72 (s, 3H), 4.92 (d, J = 13.6 Hz, 1H), 6.86 – 7.55 (m, 8H), 7.97 (d, J = 13.6 Hz, 1H), 9.73 (s, 1H) ^{13}C NMR (100 MHz, $CDCl_3$) : δ 50.51, 90.67, 92.09, 107.38, 108.70, 115.94, 119.40, 120.88, 120.98, 122.67, 126.67, 130.91, 131.89, 137.78, 149.78, 155.17, 168.12; IR (KBr) : 1635, 1660, 3202, 3482; HRMS calculated for $C_{17}H_{15}NO_4$ $[M+H]^+$ 298.1077, found 298.1068.

Methyl (*E*)-3-(5-chloro-2-(2-hydroxyphenyl)benzo[d]oxazol-3(2H)-yl)acrylate (3b)



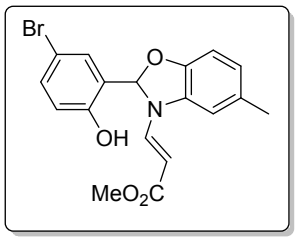
Yield : 78%; white solid; M.P. = 202-203 ˚C; Reaction time: 2 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 3.55 (s, 3H), 4.66 (d, J = 13.6 Hz, 1H), 6.80 – 7.48 (m, 8H), 7.92 (d, J = 13.6Hz, 1H), 10.29 (s,1H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 50.86, 92.33, 93.25, 108.96, 109.50, 116.25, 119.72, 120.93, 122.22, 125.42, 127.76, 131.81, 133.85, 138.27, 148.97, 155.78, 167.34 ; IR (KBr) : 1655, 1680, 3226, 3418 cm^{-1}; HRMS calculated for $C_{17}H_{14}ClNO_4$ $[M+H]^+$ 332.0687, found 332.0683.

Methyl(E)-3-(2-(2-hydroxyphenyl)-5-methylbenzo[d]oxazol-3(2H)-yl)acrylate (3c)



Yield : 85%; white solid; M.P. = 202-203 ˚C; Reaction time: 4 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 2.33 (s, 3H), 3.63 (s, 3H), 4.79 (d, J = 16 Hz, 1H), 6.63-7.20 (m, 8H), 7.83 (d, J = 12Hz, 1H), 9.72 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) : δ 20.57, 50.18, 90.33, 91.57, 95.41, 107.91, 107.98, 115.60, 119.06, 120.73, 122.48, 126.30, 130.34, 130.55, 131.49, 137.39, 147.44, 154.87, 167.69; IR (KBr) : 1632, 1667, 3231, 3455 cm^{-1}; HRMS calculated for $C_{18}H_{17}NO_4$ $[M+H]^+$ 312.1234, found 312.1235

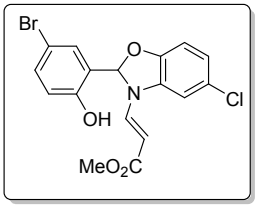
Methyl(E)-3-(2-(5-bromo-2-hydroxyphenyl)-5-methylbenzo[d]oxazol-3(2H)-yl)acrylate (3d)



Yield : 88%; white solid; M.P. = 202-203 ˚C; Reaction time: 3 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 2.27 (s, 3H), 3.54 (s, 3H), 4.67 (d, J = 13.2 Hz, 1H), 6.66 (s, 2H), 6.87 (d, J = 8.4Hz, 1H), 7.02 (s, 2H), 7.12 (s, 1H), 7.31 (d, J = 7.6Hz, 1H), 7.81 (d, J = 13.6Hz, 1H), 10.46 (s, 1H) ; ^{13}C NMR

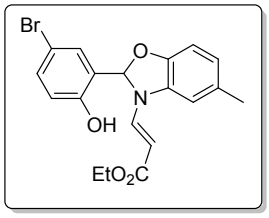
(100 MHz, $CDCl_3$) : δ 20.70, 50.35, 78.30, 78.96, 89.71, 91.87, 108.12, 108.95, 110.38, 118.13, 122.89, 123.44, 129.03, 130.76, 131.49, 133.62, 137.76, 147.36, 154.68, 167.12; IR (KBr) : 1628, 1661, 3227, 3443 cm^{-1}; HRMS calculated for $C_{18}H_{16}BrNO_4$ $[M+H]^+$ 390.0329, found 390.0327.

Methyl (E)-3-(2-(5-bromo-2-hydroxyphenyl)-5-chlorobenzo[d]oxazol-3(2H)-yl)acrylate (3e)



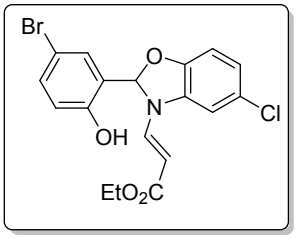
Yield : 83%; white solid; M. P : 201-202 °C; Reaction time : 2.5 h; ^1H NMR (400 MHz, $CDCl_3$) : δ 3.55 (s, 3H), 4.73 (d, J = 13.6Hz, 1H), 6.64 – 7.74 (m, 7H), 7.78 (d, J = 13.6Hz, 1H), 10.28 (s, 1H) ; ^{13}C NMR (100 MHz, $CDCl_3$) : δ 50.46, 90.77, 93.56, 108.16, 109.06, 110.61, 117.95, 121.88, 122.69, 125.71, 129.10, 133.02, 133.68, 137.22, 148.27, 154.56, 167.05; IR (KBr) : 1631, 1670, 3181, 3467 cm^{-1}; HRMS calculated for $C_{17}H_{13}BrClNO_4$ $[M+H]^+$ 409.9792, found 409.9789

Ethyl (*E*)-3-(2-(5-bromo-2-hydroxyphenyl)-5-methylbenzo[d]oxazol-3(2H)-yl)acrylate (3f)



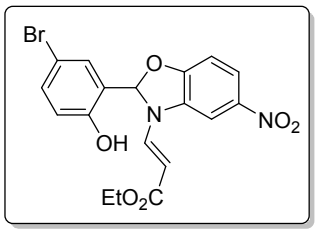
Yield : 90%; white solid; M. P : 190-191°C; Reaction time : 3 h; ^1H NMR (400 MHz, $CDCl_3$) : δ 1.14 (d, J = 6.8Hz, 3H), 2.26 (s, 3H), 4.00 (q, J = 4Hz, 2H), 4.64 (d, J = 13.6Hz, 1H), 6.71 – 7.43 (m, 7H), 7.86 (d, J = 13.6Hz, 1H), 10.62 (s, 1H) ; ^{13}C NMR (100 MHz, $CDCl_3$) : δ 14.44, 20.79, 59.08, 90.11, 92.14, 108.34, 109.48, 110.36, 118.53, 123.14, 123.71, 129.35, 131.00, 131.71, 134.07, 138.12, 147.50, 155.05, 166.90; IR (KBr) : 1587, 1642, 2991, 3484cm^{-1}; HRMS calculated for $C_{19}H_{18}BrNO_4$ $[M+H]^+$ 404.0495, found 404.0493

Ethyl (*E*)-3-(2-(5-bromo-2-hydroxyphenyl)-5-chlorobenzo[d]oxazol-3(2H)-yl)acrylate (3g)



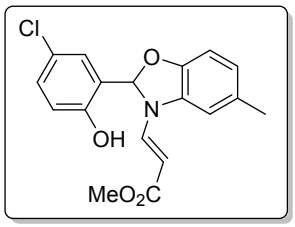
Yield : 86%; white solid; M. P : 189-190°C; Reaction time : 2 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 1.16 (t, *J* = 6.8 Hz, 3H), 4.00 (q, *J* = 1.8Hz, 2H), 4.68 (d, *J*=13.6 Hz, 1H), 6.71-7.33 (m, 7H), 7.83 (d, J = 13.6Hz, 1H), 10.47 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) : δ 14.19, 58.99, 90.93, 93.79, 108.58, 109.12, 110.42, 118.16, 121.88, 122.93, 125.57, 129.28, 133.27, 133.83, 137.58, 148.34, 154.79, 166.50; IR (KBr) : 1603, 1634, 2987, 3411cm^{-1}; HRMS calculated for $C_{18}H_{15}BrClNO_4$ $[M+H]^+$ 423.9949, found 423.9946.

Ethyl (*E*)-3-(2-(5-bromo-2-hydroxyphenyl)-5-nitrobenzo[d]oxazol-3(2H)-yl)acrylate (3h)



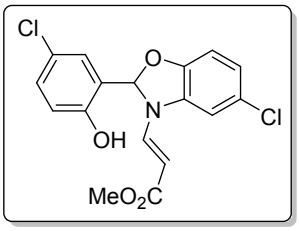
Yield : 84%; Pale yellow solid; M. P : 155-156°C; Reaction time : 4 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 1.29 (t, *J*=7.2 Hz, 3H), 4.22 (q, J = 7.2Hz, 2H), 5.70 (d, *J*=12.4 Hz, 1H), 7.06-8.32 (m, 8H), 9.01 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 14.41, 60.71, 105.14, 112.53, 115.60, 118.75, 120.12, 125.49, 127.30, 130.94, 135.12, 136.87, 141.41, 153.15, 155.13, 157.54, 158.09, 166.50; IR (KBr) : 1596, 1698, 2831, 3437cm^{-1}; HRMS calculated for $C_{18}H_{15}BrN_2O_6$ $[M+H]^+$ 435.0189, found 435.0179.

Methyl (*E*)-3-(2-(5-chloro-2-hydroxyphenyl)-5 methylbenzo[d]oxazol-3(2H)-yl)acrylate (3i)



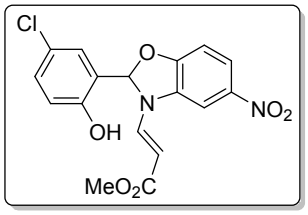
Yield : 80%; white solid; M. P : 204-205°C; Reaction time : 3 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 2.27 (s, 3H), 3.55 (s, 3H), 4.65 (d, J = 12Hz, 1H) 6.69-7.32 (m, 7H), 7.87 (d, J = 12Hz, 1H), 10.59 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 20.76, 50.65, 90.16, 91.80, 108.32, 109.51, 118.02, 122.89, 123.13, 126.53, 130.98, 131.18, 131.66, 138.15, 147.52, 154.60, 167.25; IR (KBr) : 1611, 1663, 3228, 3443cm^{-1}; HRMS calculated for $C_{18}H_{16}ClNO_4$ $[M+H]^+$ 346.0844, found 346.0843.

Methyl (E)-3-(5-chloro-2-(5-chloro-2-hydroxyphenyl)benzo[d]oxazol-3(2H)-yl)acrylate (3j)



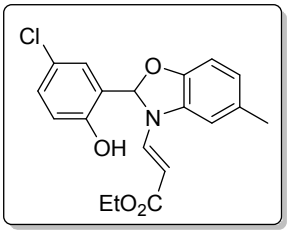
Yield : 75%; white solid; M. P : 201-202°C; Reaction time : 2 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 3.55 (s, 3H), 4.67 (d, J = 13.6Hz, 1H), 6.83 – 7.53 (m, 7H), 7.95 (d, J = 13.6Hz, 1H), 10.58 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 49.16, 89.54, 92.27, 106.86, 107.77, 116.19, 120.59, 120.86, 122.16, 124.42, 124.90, 129.51, 131.73, 135.92, 146.98, 152.77, 165.78; IR (KBr) : 1601, 1665, 2831, 3427cm^{-1}; **HRMS** calculated for $C_{17}H_{13}Cl_2NO_4$ $[M+H]^+$ 366.0298, found 366.0299.

Methyl (*E*)-3-(2-(5-chloro-2-hydroxyphenyl)-5-nitrobenzo[d]oxazol-3(2H)-yl)acrylate (3k)



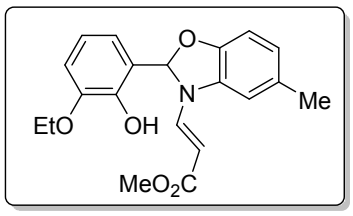
Yield : 73%; Pale yellow solid; M. P : 155-156°C; Reaction time : 3.5 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 3.59 (s, 3H), 4.83 (d, J = 13.6Hz, 1H), 6.77 – 7.82 (m, 7H), 10.08 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 50.54, 92.42, 95.07, 102.52, 107.58, 117.38, 119.97, 121.01, 123.66, 126.16, 131.07, 133.03, 136.46, 142.07, 144.04, 153.97, 154.66, 166.86; IR (KBr) : 1651, 1697, 3073, 3425 cm^{-1}; HRMS calculated for $C_{17}H_{13}ClN_2O_6$ $[M+H]^+$ 377.0538, found 377.0525.

Ethyl (*E*)-3-(2-(5-chloro-2-hydroxyphenyl)-5-methylbenzo[d]oxazol-3(2H)-yl)acrylate (3l)



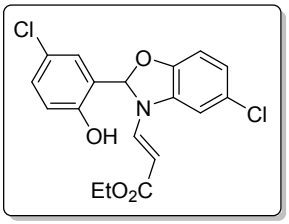
Yield : 89%; white solid; M. P : 186-187°C; Reaction time : 4 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 1.14 (t, *J* = 8Hz, 3H), 2.27 (s, 3H), 4.01 (q, J = 7.2Hz, 2H), 4.63 (d, *J* = 12Hz, 1H), 6.71 – 7.32 (m, 7H), 7.86 (d, J = 12Hz, 1H), 10.59 (s, 1H) ; ^{13}C NMR (100 MHz, $CDCl_3$) : δ 14.45, 20.79, 59.08, 90.17, 92.11, 108.35, 109.50, 118.05, 122.91, 123.17, 126.52, 131.00, 131.21, 131.71, 138.12, 147.50, 154.62, 166.90; IR (KBr) : 1620, 1670, 3181, 3448cm^{-1}; HRMS calculated for $C_{19}H_{18}ClNO_4$ $[M+H]^+$ 360.1000, found 360.0997.

Methyl (*E*)-3-(2-(3-ethoxy-2-hydroxyphenyl)-5-methylbenzo[d]oxazol-3(2H)-yl)acrylate (3m)



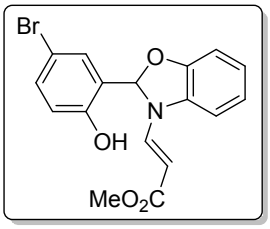
Yield : 84%; M. P : 159-160°C; Reaction time : 4 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 1.45 (t, *J*=7.2Hz, 3H), 2.33 (s, 3H), 3.66 (s, 3H), 4.11 (q, J= 6.8Hz, 2H), 4.80 (d, *J*=13.6 Hz, 1H), 6.09 (s, 1H), 6.66 – 6.88 (m, 6H), 7.12 (s, 1H), 7.86 (d, *J* = 13.6, 1H) ; ^{13}C NMR (75 MHz, $CDCl_3$) : δ 14.98, 21.36, 51.13, 64.91, 91.13, 92.58, 108.84, 112.94, 118.80, 120.39, 120.88, 123.31, 131.35, 132.25, 138.27, 144.22, 146.13, 148.26, 168.75; IR (KBr) : 1620, 3031, 3243, 3434cm^{-1}; HRMS calculated for $C_{25}H_{22}N_3O_2$ $[M+H]^+$ 356.1496, found 356.1496.

Methyl (*E*)-3-(5-chloro-2-(5-chloro-2-hydroxyphenyl)benzo[d]oxazol-3(2H)-yl)acrylate (3n)



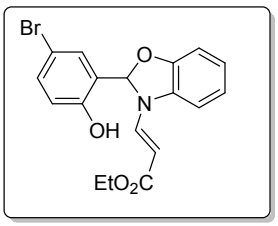
Yield : 80%; white solid; M. P : 171-172°C; Reaction time : 2 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 1.14 (t, J = 7.2Hz, 3H), 4.02 (q, J = 7.2Hz, 2H), 4.65 (d, J = 13.6Hz, 1H), 6.82 – 7.52 (m, 7H), 7.93 (d, J = 13.6Hz, 1H), 10.60 (s, 1H) ; ^{13}C NMR (100 MHz, $CDCl_3$) : δ14.41, 59.19, 91.48, 93.62, 109.15, 109.43, 118.07, 122.12, 122.67, 122.93, 125.42, 127.00, 131.43, 133.60, 138.19, 148.62, 154.76, 166.63 ; IR (KBr) : 1631, 1676, 2990, 3456cm^{-1} ; HRMS calculated for $C_{18}H_{15}Cl_2NO_4$ $[M+H]^+$ 380.0454, found 380.0455.

Methyl (*E*)-3-(2-(5-bromo-2-hydroxyphenyl)benzo[d]oxazol-3(2H)-yl)acrylate (3o)



Yield : 86%; white solid; M. P : 188-189°C; Reaction time: 3 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 3.64 (s, 3H), 4.79 (d, J = 13.6 Hz, 1H), 6.78 – 7.72 (m, 8H), 7.86 (d, J = 13.6Hz, 1H), 10.21 (s, 1H) ; ^{13}C NMR (100 MHz, $CDCl_3$) : δ 50.75, 89.96, 92.66, 107.71, 109.02, 111.27, 118.03, 121.36, 122.99, 123.14, 129.28, 131.79, 133.75, 137.90, 149.64, 154.58, 168.12 ; IR (KBr) : 1600, 1656, 2946, 3437 cm^{-1}; HRMS calculated for $C_{17}H_{14}BrNO_4$ $[M+H]^+$ 376.0182, found 376.0171.

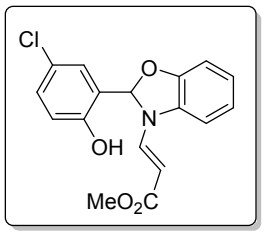
(Ethyl (*E*)-3-(2-(5-bromo-2-hydroxyphenyl)benzo[d]oxazol-3(2H)-yl)acrylate (3p)



Yield : 88%; white solid; M. P : 170-171 °C; Reaction time : 4 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 1.23 (t, J = 7.2Hz, 3H), 4.09 (q, J = 6.8Hz, 2H), 4.79 (d, J = 13.6Hz, 1H), 6.78 – 7.54 (m, 6H), 7.86 (d, J = 13.6Hz, 1H), 10.05 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) : δ 13.93, 58.99, 89.59, 92.68, 107.40, 108.59, 110.76, 117.73, 121.03, 122.59, 122.89, 128.84, 131.47, 133.36, 137.43,

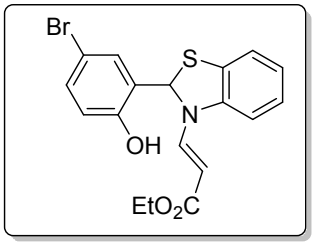
149.21, 154.32, 167.24; IR (KBr) : 1621, 1667, 3179, 3433cm^{-1}, HRMS calculated for $C_{18}H_{16}BrNO_4$ $[M+H]^+$ 390.0339, found 390.0338.

Methyl (*E*)-3-(2-(5-chloro-2-hydroxyphenyl)benzo[d]oxazol-3(2H)-yl)acrylate (3q)



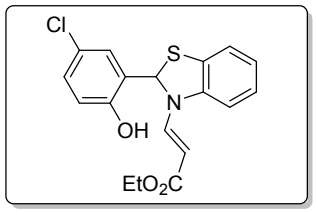
Yield : 79%; white solid; M. P : 191-192°C; Reaction time : 3 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 3.55 (s, 3H), 4.66 (d, J = 13.6Hz, 1H), 6.85-7.36 (m, 8H), 7.91 (d, J = 13.6Hz, 1H), 10.59 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 50.38, 89.62, 92.25, 107.49, 108.62, 117.33, 121.14, 122.43, 122.71, 123.62, 126.02, 130.54, 131.45, 137.52, 149.31, 153.90, 167.60; IR (KBr) : 1600, 1665, 3224, 3452 cm^{-1}; HRMS calculated for $C_{17}H_{14}ClNO_4$ $[M+H]^+$ 332.0687, found 332.0684.

Ethyl *(E)*-3-(2-(5-bromo-2-hydroxyphenyl)benzo*[d]*thiazol-3*(2H)*-yl)acrylate (3r)



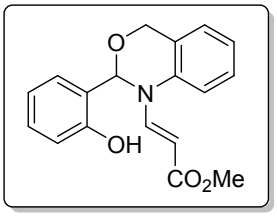
Yield : 65 %; M. P : 161-162°C; Reaction time : 4 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 1.29 (t, J = 6.8Hz, 3H), 4.20 (q, J = 7.2 Hz, 2H), 5.80 (d, J = 12.4 Hz, 1H); 7.08 – 8.12 (m, 7H), 8.69 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 14.41, 60.60, 104.73, 118.82, 120.31, 121.56, 123.53, 125.76, 126.64, 132.69, 134.65, 136.23, 152.22, 152.41, 157.48, 160.02, 166.59; IR (KBr) : 1650, 1693, 2832, 3425 cm^{-1}; HRMS calculated for $C_{18}H_{16}BrNO_3S$ $[M-H]^+$ 403.9958, found 403.9954.

Ethyl *(E)*-3-(2-(5-bromo-2-hydroxyphenyl)benzo*[d]*thiazol-3*(2H)*-yl)acrylate (3s)



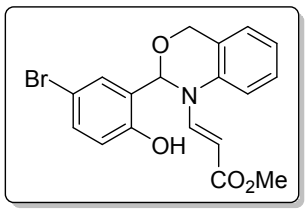
Yield : 68 %; M. P : 163-164°C; Reaction time : 4 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 1.29 (t, J =8 Hz, 3H), 4.19 (q, J = 4Hz, 2H), 5.80 (d, J = 12Hz, 1H), 7.15-8.55 (m,7H), 8.56 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) : δ 14.42, 60.60, 104.62, 120.09, 121.58, 123.53, 125.77, 126.03, 126.65, 129.77, 131.39, 131.73, 136.23, 151.89, 152.23, 157.66, 160.16, 166.63 ; IR (KBr) : 1615, 1680, 2945, 3437 cm^{-1}; HRMS calculated for $C_{18}H_{16}NO_3S$ $[M-H]^+$ 360.0463, found 360.0459.

Ethyl (*E*)-3-(2-(2-hydroxyphenyl)-2H-benzo*[d]*[1,3]oxazin-1*(4H)*-yl)acrylate (5a)



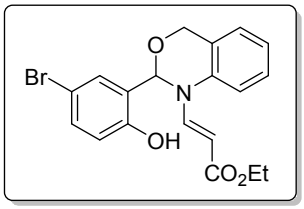
Yield : 92%; M. P : 202-203°C; Reaction time : 6 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 3.53 (s, 3H), 4.58 (d, J = 14Hz, 1H), 4.68 (dd, J = 14Hz, 8.8Hz, 2H), 6.63 (s, 1H), 6.71-7.40 (m, 8H), 7.93 (d, J = 14Hz, 1H), 10.04 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 50.50, 62.31, 81.49, 92.18, 114.25, 116.07, 118.82, 120.84, 122.02, 124.27, 124.84, 127.00, 128.21, 130.13, 137.53, 141.62, 155.32, 168.52; IR (KBr) : 1605, 1663, 3179, 3433cm^{-1}; HRMS calculated for $C_{18}H_{17}NO_4$ $[M+H]^+$ 312.1234, found 312.1214.

Methyl (*E*)-3-(2-(5-bromo-2-hydroxyphenyl)-2H-benzo*[d]*[1,3]oxazin-1*(4H)*-yl)acrylate (5b)



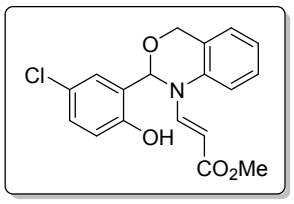
Yield : 85%; M. P : 204-205°C; Reaction time : 6 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 3.65 (s, 3H), 4.65 (s, 2H), 4.83 (d, J = 13.6Hz, 1H), 6.49 (s, 1H), 6.88 – 7.56 (m, 7H), 8.06 (d, J = 13.6Hz, 1H), 9.80 (s, 1H) ; ^{13}C NMR (100 MHz, $CDCl_3$) : δ 50.34, 62.70, 80.92, 92.18, 110.33, 114.47, 117.77, 122.12, 123.43, 124.55, 124.75, 128.29, 129.24, 132.61, 137.32, 141.50, 154.49, 168.10 ; IR (KBr) : 1610, 1673, 3231, 3444 cm^{-1}; HRMS calculated for $C_{18}H_{16}BrNO_4$ $[M+H]^+$ 390.0339, found 390.0320.

Ethyl (*E*)-3-(2-(5-bromo-2-hydroxyphenyl)-2H-benzo*[d]*[1,3]oxazin-1*(4H)*-yl)acrylate (5c)



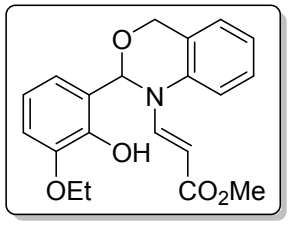
Yield : 90%; M. P : 200-201°C; Reaction time : 5 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 1.27 (t, J = 7.2 Hz, 3H), 4.18 (q, J =7.2 Hz, 2H), 4.70 (dd, 14.8Hz, 14.4Hz, 2H), 5.03 (d, J = 13.6 Hz, 1H), 6.47 (s, 1H), 6.83-7.35 (m, 7H), 8.15 (d, J = 13.6Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 13.99, 58.97, 62.64, 80.98, 92.66, 110.41, 114.50, 117.84, 122.11, 123.44, 124.44, 124.77, 128.29, 129.33, 132.65, 137.35, 141.45, 154.55, 167.79 ; IR (KBr) : 1606, 1668, 3240, 3432cm^{-1}; HRMS calculated for $C_{19}H_{18}BrNO_4$ $[M+H]^+$ 404.0492, found 404.0496.

Methyl (*E*)-3-(2-(5-chloro-2-hydroxyphenyl)-2H-benzo*[d]*[1,3]oxazin-1*(4H)*-yl)acrylate (5d)



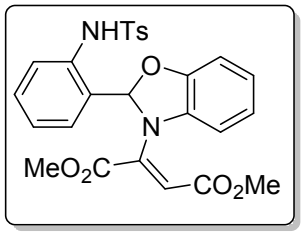
Yield : 84%; M. P : 201-202°C; Reaction time : 6 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 3.55 (s, 3H), 4.57 (s, 2H), 4.72 (d, J = 12 Hz, 1H), 6.39 (s, 1H), 6.71 – 7.69 (m, 7H), 7.95 (d, J = 12Hz, 1H), 9.98 (s, 1H) ; ^{13}C NMR (100 MHz, $CDCl_3$) : δ 49.77, 62.16, 80.35, 91.48, 113.99, 116.77, 121.66, 122.33, 122.54, 124.23, 124.28, 125.76, 127.83, 129.14, 136.86, 140.95, 153.53, 167.39; IR (KBr) : 1611, 1673, 3237, 3439cm^{-1}; HRMS calculated for $C_{18}H_{16}ClNO_4$ $[M+H]^+$ 346.0833, found 346.0838.

Methyl (*E*)-3-(2-(3-ethoxy-2-hydroxyphenyl)-2H-benzo*[d]*[1,3]oxazin-1*(4H)*-yl)acrylate (5e)



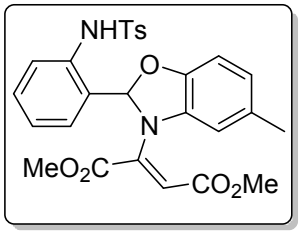
Yield : 87%; M. P : 203-204°C; Reaction time : 5 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 1.46 (t, *J*=7.2 Hz, 3H), 3.66 (s, 3H), 4.10 (q, *J*=6.8 Hz, 2H), 4.66 (s, 2H), 4.89 (d, *J* = 13.6Hz, 1H), 6.16 (s, 1H), 6.53 – 7.03 (m, 8H), 8.14 (d, *J* = 13.6 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) : δ 14.99, 31.04, 51.11, 63.03, 64.89, 81.85, 92.97, 112.64, 114.91, 119.39, 119.71, 121.14, 122.53, 124.73, 125.30, 128.75, 138.01, 142.19, 144.33, 146.34, 169.15; IR (KBr) : 1631, 3045, 3240, 3429cm^{-1}; HRMS calculated for $C_{25}H_{21}ClN_3O_2$ $[M+Na]^+$ 378.1317, found 378.1324

Dimethyl 2-(2-(2-((4-methylphenyl)sulfonamido)phenyl)benzo*[d]*oxazol-3*(2H)*-yl)fumarate (7a)



Yield : 72%; yellow solid, M. P : 159-160°C; Reaction time : 8 h; ^{1}H NMR (400 MHz, $CDCl_3$) : *δ* 2.19 (s, 3H), 3.62 (s, 3H), 4.01 (s, 3H), 5.02 (s, 1H), 6.77 – 7.58 (m, 12H), 8.19 (d, *J* = 1.6 Hz, 1H), 8.20 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) : δ 21.57, 51.94, 53.68, 105.71, 115.11, 116.62, 120.29, 128.49, 128.60, 129.86, 130.76, 132.23, 132.45, 133.75, 134.51, 134.59, 136.60, 145.98, 147.95, 150.90, 152.79, 164.45, 166.45 ; IR (KBr) : 1765, 1790, 3251, 3445cm^{-1}; HRMS calculated for $C_{26}H_{24}N_2O_7S$ $[M+H]^+$ 509.1380, found 509.1361.

Methyl *(E)*-3-(2-(3-ethoxy-2-hydroxyphenyl)-2H-benzo*[d]*[1,3]oxazin-1*(4H)*-yl)acrylate (7b)



Yield : 78%; Pink solid, M. P : 171-172°C; Reaction time : 8 h; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 2.22 (s, 3H), 2.34 (s, 3H), 3.62 (s, 3H), 4.01 (s, 3H), 5.02 (s, 1H), 6.85 – 8.50 (m, 10 H), 8.79 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) : δ 21.01, 21.59, 51.93, 53.09, 53.62, 105.67, 114.81, 116.89, 128.53, 128.63, 129.38, 129.83, 130.50, 130.73, 132.11, 132.26, 133.92, 134.15, 134.58, 136.54, 145.93, 147.96, 150.60, 165.49; IR (KBr) : 1735, 1785, 3267, 3466 cm^{-1}; HRMS calculated for $C_{27}H_{26}N_2O_7S$ $[M+H]^+$ 523.1537, found 523.1539.

X-ray Analysis

Ethyl (E)-3-(2-(5-bromo-2-hydroxyphenyl)-5-methylbenzo[d]oxazol-3(2H)-yl)acrylate (3d)

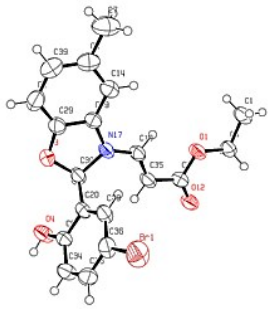


Table 1. Crystal data and structure refinement for (3d).

Identification code	EXP-134
Empirical formula	$C_{19}H_{18}BrNO_4$
Formula weight	404.25
Temperature/K	298
Crystal system	monoclinic
Space group	$P2_1/a$
a/Å	12.8540(7)
b/Å	9.8269(6)
c/Å	14.2696(11)
α/°	90
β/°	94.215(5)
γ/°	90
Volume/Å^3	1797.6(2)
Z	4

$\rho_{calc}g/cm^3$	1.494
μ/mm^{-1}	2.310
F(000)	824.0
Crystal size/mm^3	0.6 × 0.2 × 0.08
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	8.278 to 58.558
Index ranges	-15 ≤ h ≤ 17, -12 ≤ k ≤ 13, -17 ≤ l ≤ 18
Reflections collected	10756
Independent reflections	4183 [R_{int} = 0.0402, R_{sigma} = 0.0560]
Data/restraints/parameters	4183/0/232
Goodness-of-fit on F^2	1.017
Final R indexes [I>=2σ (I)]	R_1 = 0.0483, wR_2 = 0.1102
Final R indexes [all data]	R_1 = 0.1008, wR_2 = 0.1371
Largest diff. peak/hole / e $Å^{-3}$	0.41/-0.47

Methyl (E) -3-(2-(2-hydroxyphenyl)-2H-benzo[d][1,3]oxazin-1(4H)-yl)acrylate (5a)

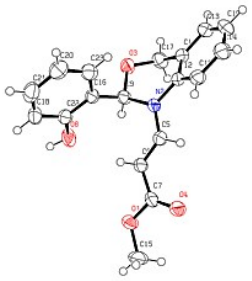
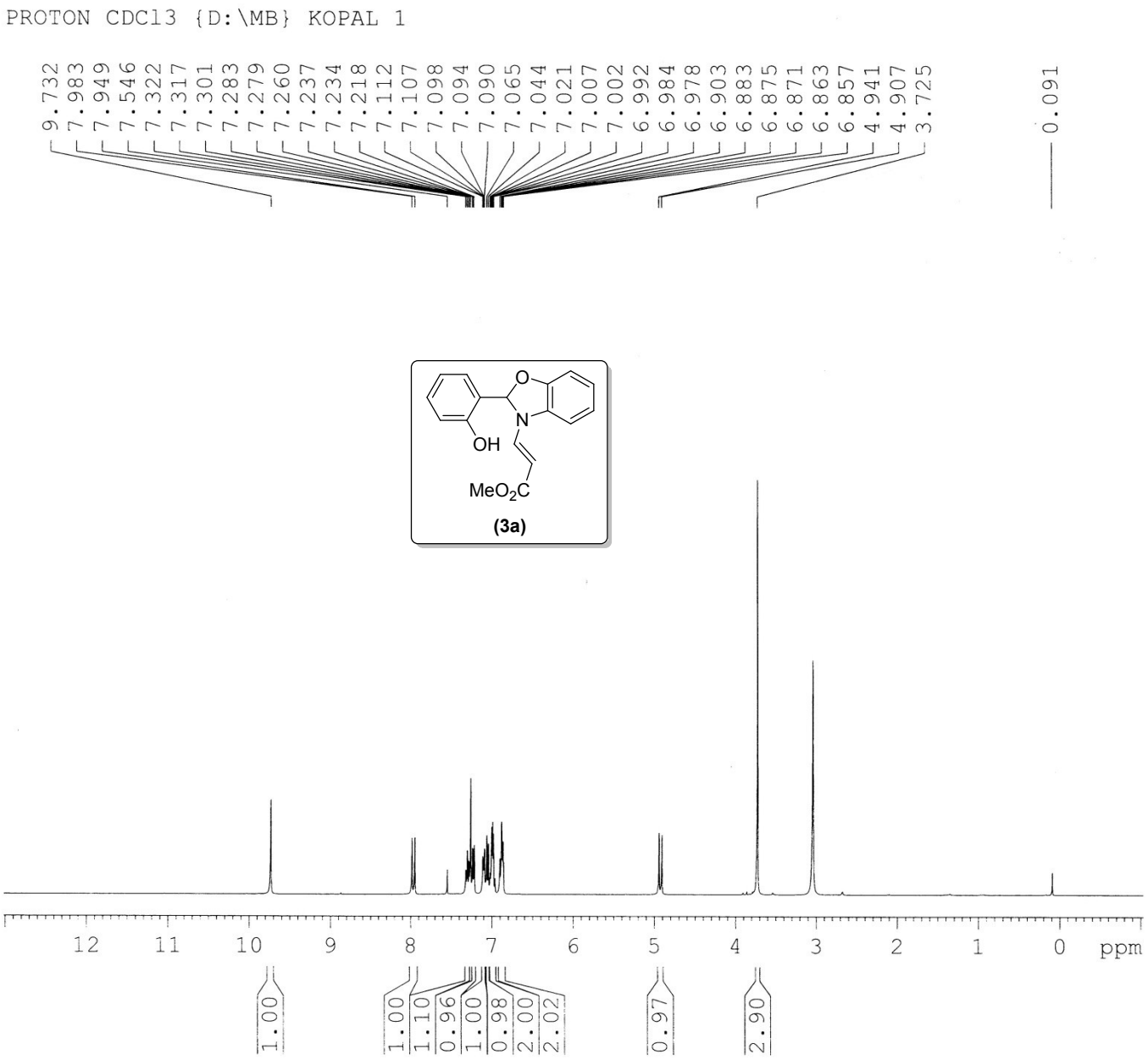


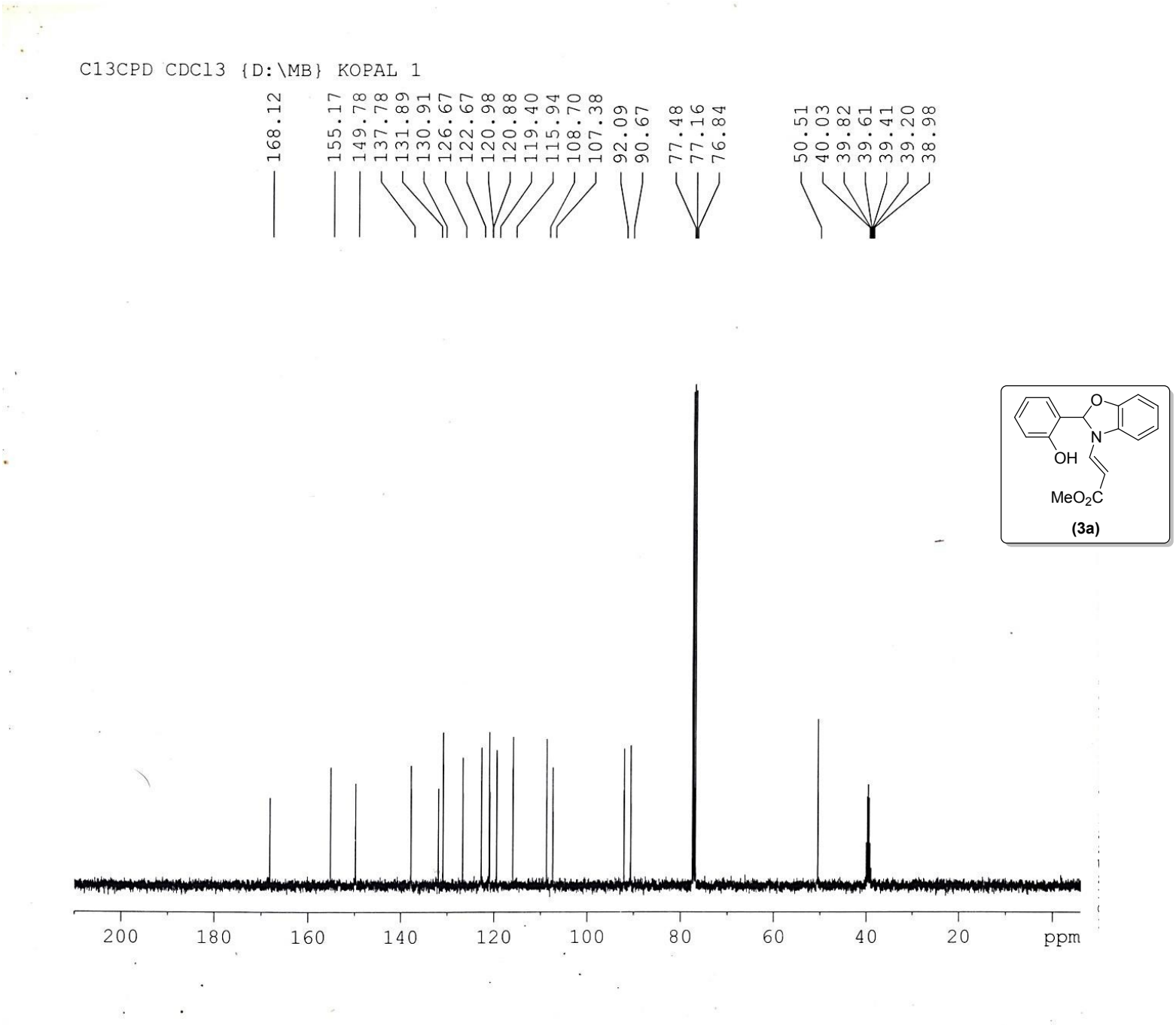
Table 2. Crystal data and structure refinement for (5a).

Identification code	MB-MM-39
Empirical formula	$C_{19}H_{18}NO_3$
Formula weight	308.34
Temperature/K	298(2)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	7.5340(5)
b/Å	13.2552(10)

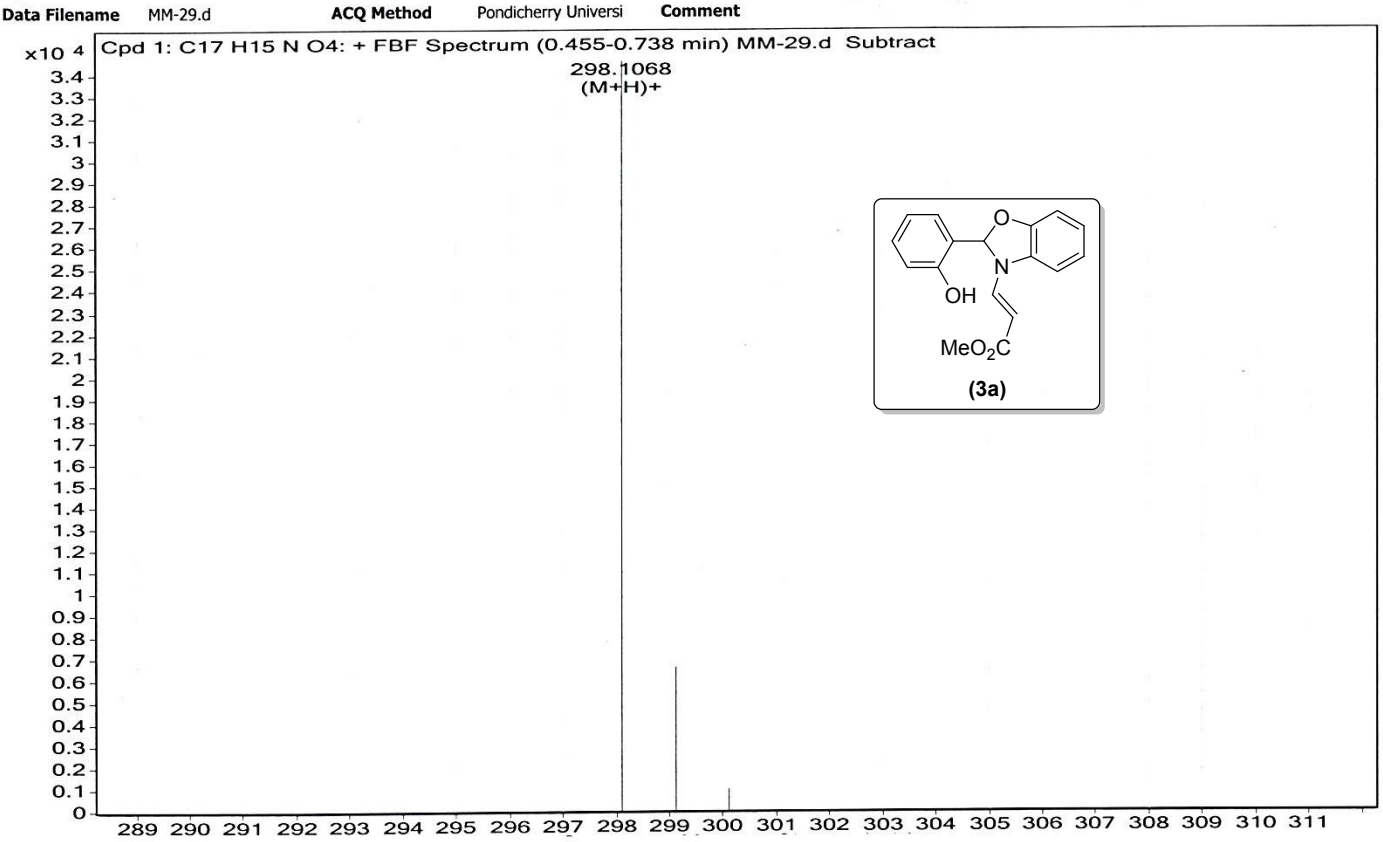
c/Å	15.2014(12)
α/°	90.00
β/°	90.00
γ/°	90.00
Volume/Å^3	1518.09(19)
Z	4
ρ_{calc}g/cm^3	1.349
μ/mm^{-1}	0.091
F(000)	652.0
Crystal size/mm^3	0.5 × 0.04 × 0.03
Radiation	Mo Kα (λ = 0.7107)
2Θ range for data collection/°	8.62 to 58.2
Index ranges	$-10 \leq h \leq 9$, $-16 \leq k \leq 17$, $-20 \leq l \leq 20$
Reflections collected	5373
Independent reflections	3203 [R_{int} = 0.0374, R_{sigma} = 0.0614]
Data/restraints/parameters	3203/0/213
Goodness-of-fit on F^2	1.011
Final R indexes [I>=2σ (I)]	R_1 = 0.0458, wR_2 = 0.0843
Final R indexes [all data]	R_1 = 0.0758, wR_2 = 0.0976
Largest diff. peak/hole /e Å^{-3}	0.14/-0.18

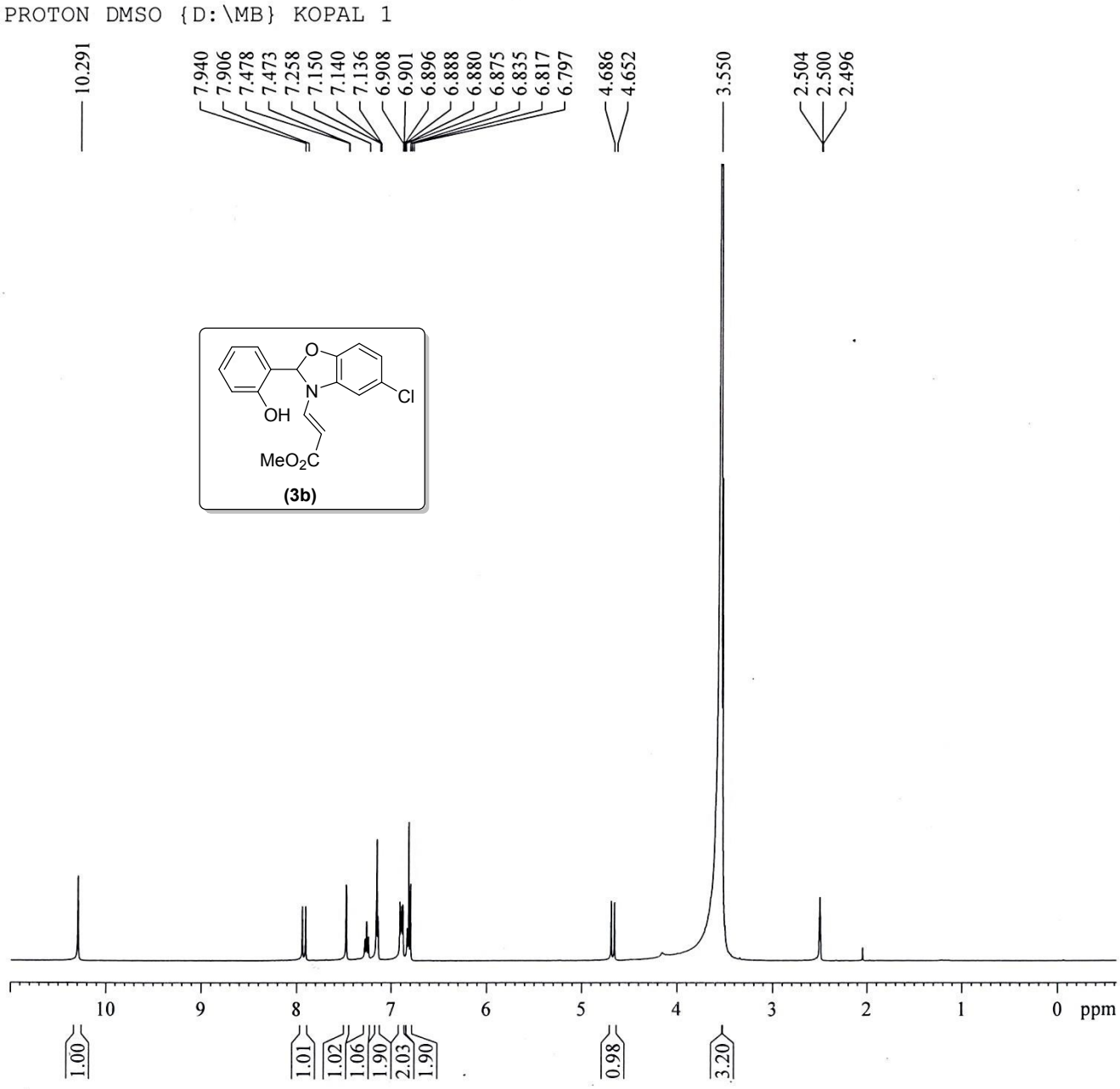
^{1}H, ^{13}C NMR & ESI-HRMS Spectra for the Compounds 3a-s, 5a-e & 7a-b

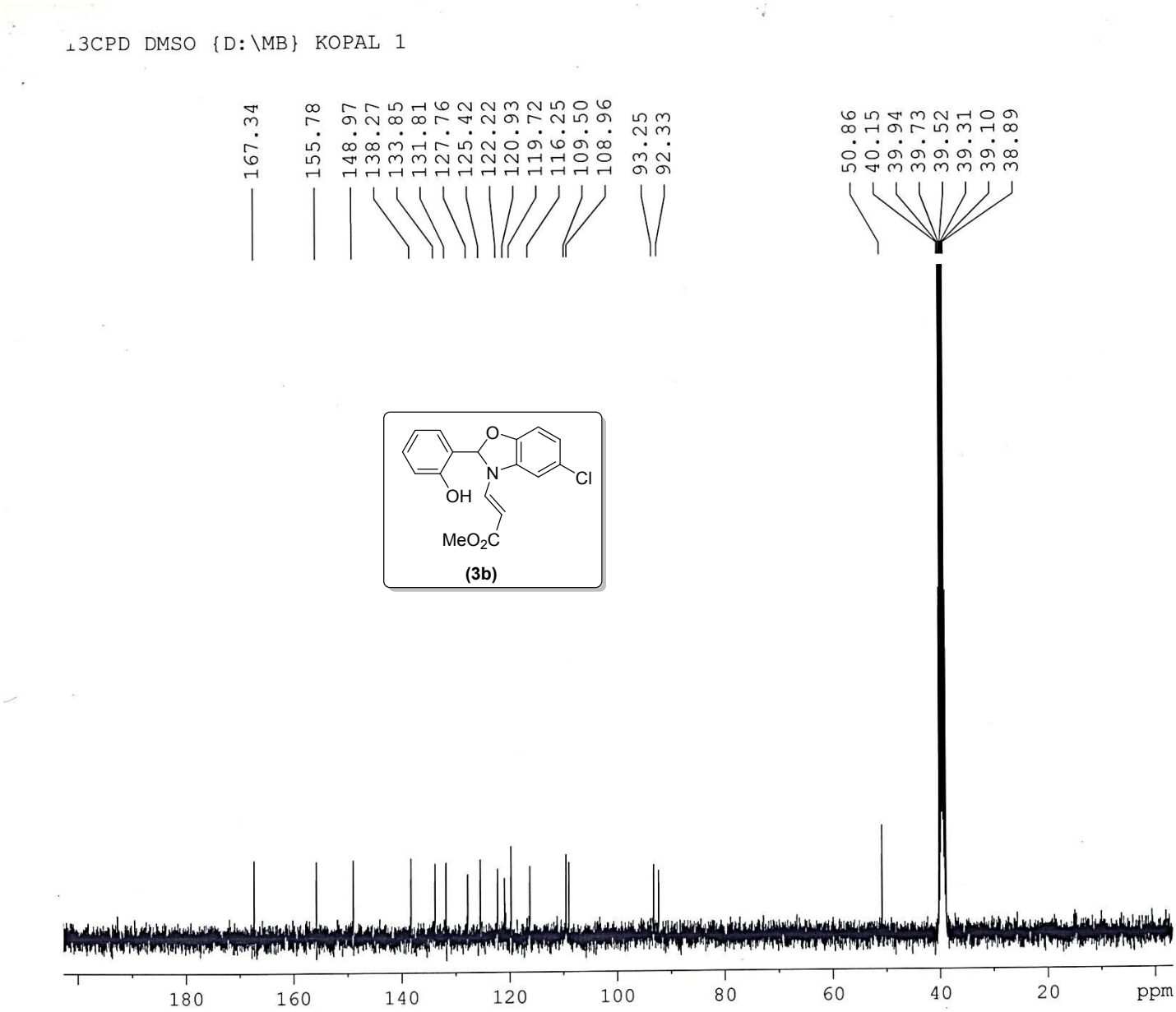



C13CPD CDCl3 {D:\MB} KOPAL 1
168.12
155.17
149.78
137.78
131.89
130.91
126.67
122.67
120.98
120.88
119.40
115.94
108.70
107.38
92.09
90.67
77.48
77.16
76.84
50.51
40.03
39.82
39.61
39.41
39.20
38.98
OH
MeO2C
(3a)
200
180
160
140
120
100
80
60
40
20
ppm

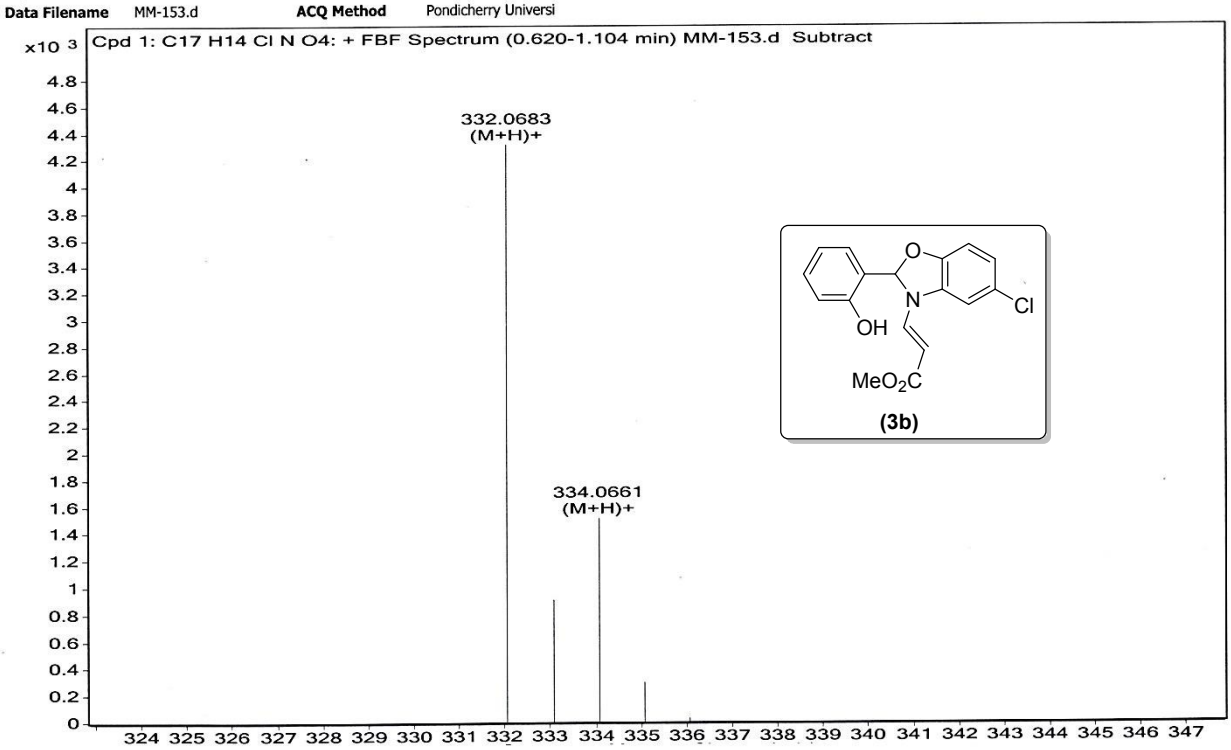
Sample Name	MM-29	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-29.d	**ACQ Method**	Pondicherry Universi	**Comment**			

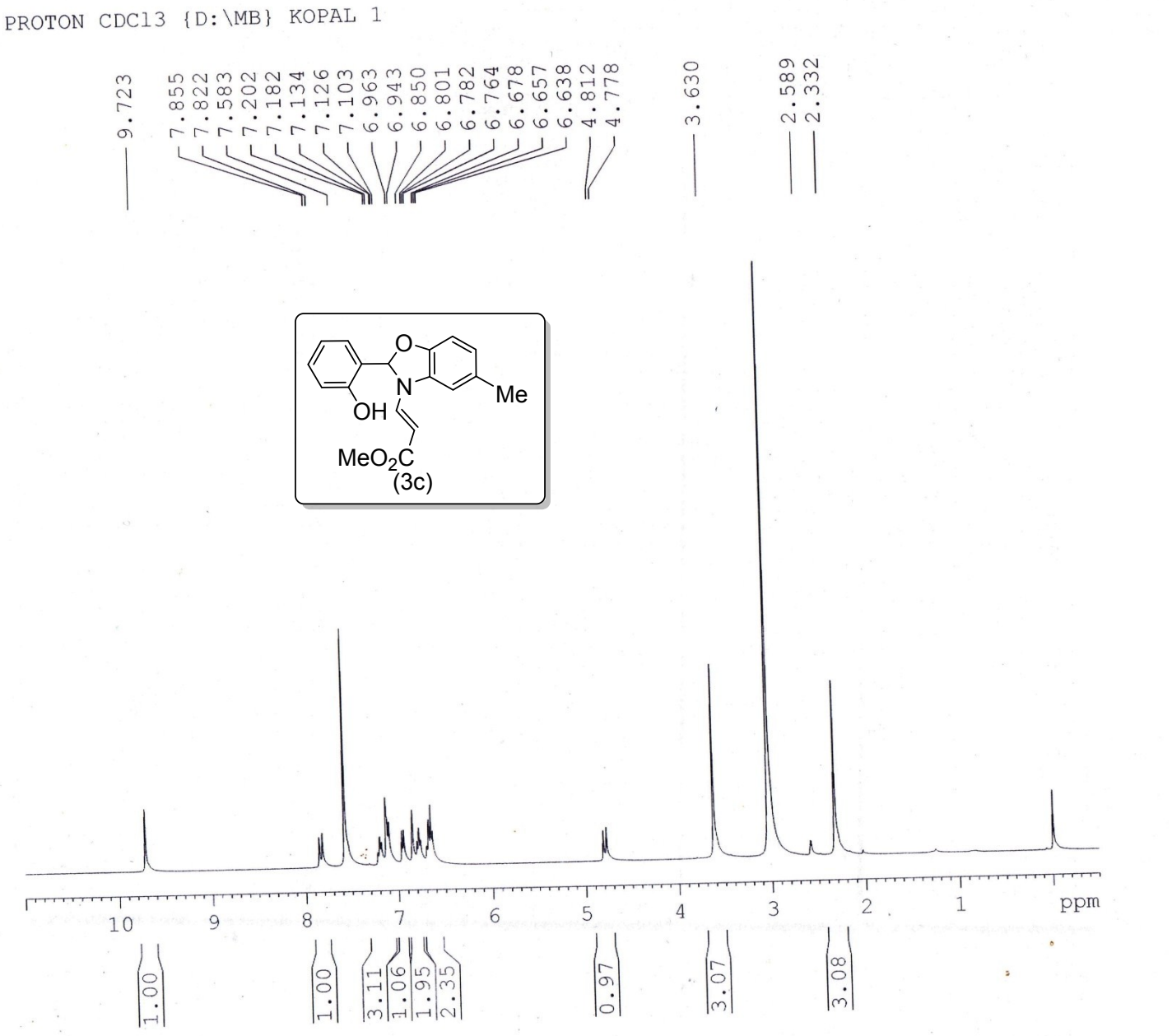


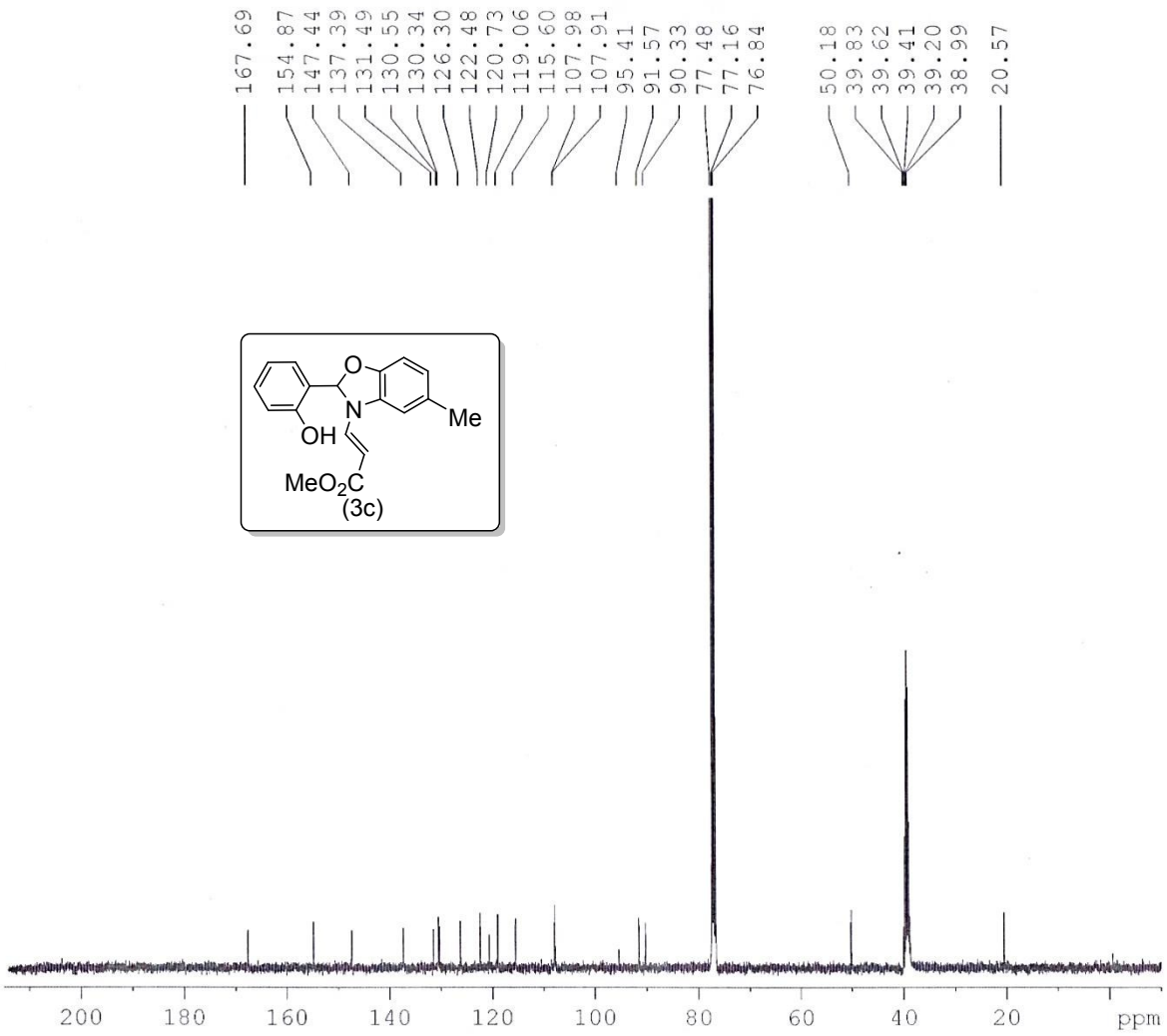

PROTON DMSO {D:\MB} KOPAL 1
10.291
7.940
7.906
7.478
7.473
7.258
7.150
7.140
7.136
6.908
6.901
6.896
6.888
6.880
6.875
6.835
6.817
6.797
4.686
4.652
3.550
2.504
2.500
2.496
O
N
Cl
OH
MeO2C
(3b)
10
9
8
7
6
5
4
3
2
1
0 ppm
1.00
1.01
1.02
1.06
1.90
2.03
1.90
0.98
3.20


13CPD DMSO {D:\MB} KOPAL 1
167.34
155.78
148.97
138.27
133.85
131.81
127.76
125.42
122.22
120.93
119.72
116.25
109.50
108.96
93.25
92.33
50.86
40.15
39.94
39.73
39.52
39.31
39.10
38.89
O
N
Cl
OH
MeO2C
(3b)
180
160
140
120
100
80
60
40
20
ppm

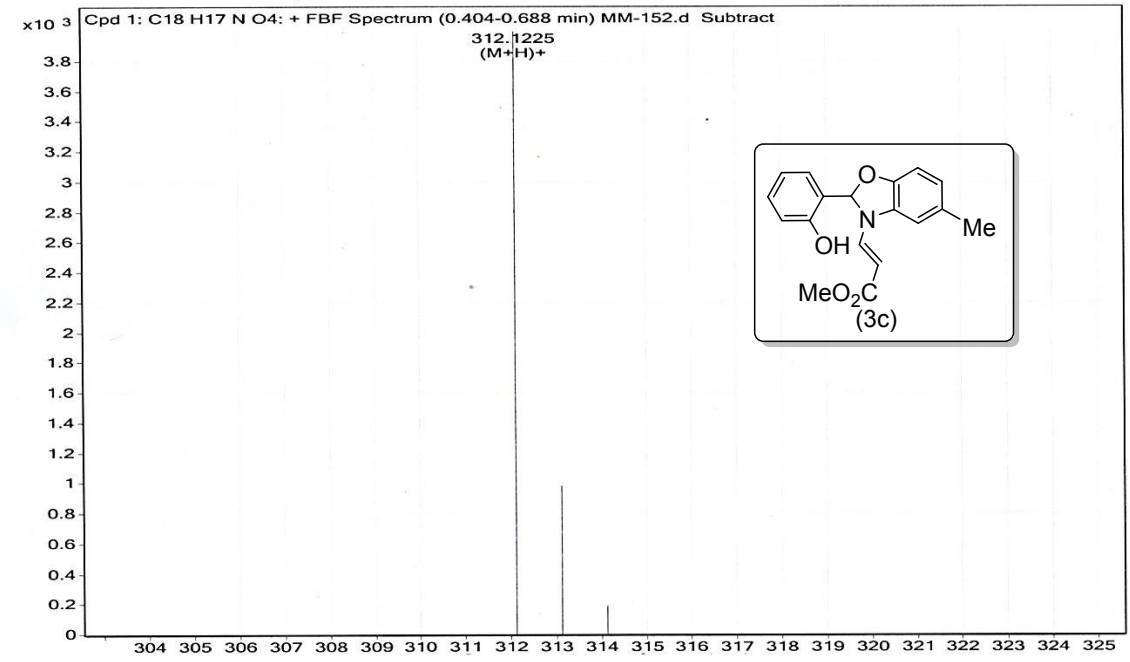
Sample Name	MM-153	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-153.d	**ACQ Method**	Pondicherry Universi				

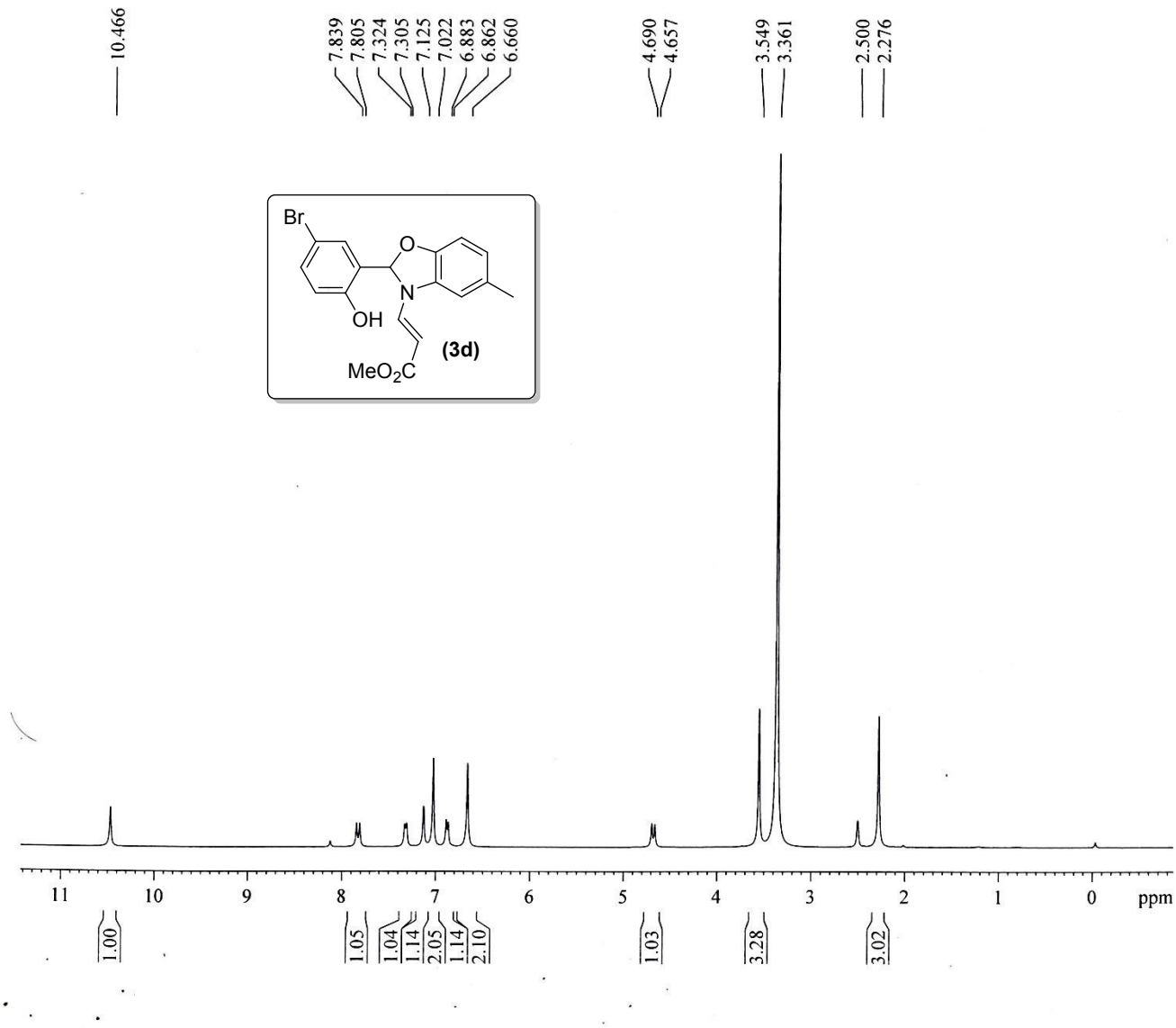


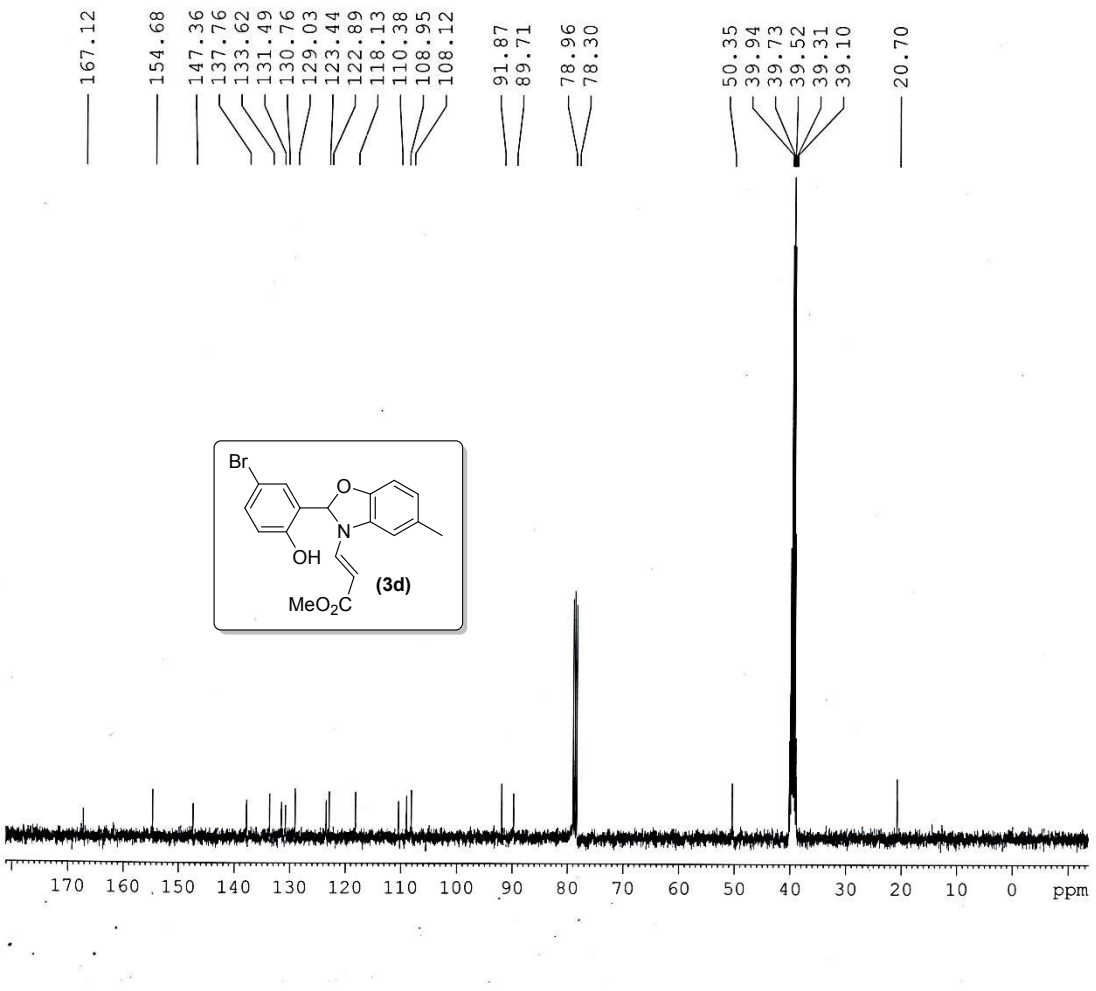

PROTON CDCl3 {D:\MB} KOPAL 1
9.723
7.855
7.822
7.583
7.202
7.182
7.134
7.126
7.103
6.963
6.943
6.850
6.801
6.782
6.764
6.678
6.657
6.638
4.812
4.778
3.630
2.589
2.332
O
N
Me
OH
MeO2C
(3c)
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.00
3.11
1.06
1.95
2.35
0.97
3.07
3.08


167.69
154.87
147.44
137.39
131.49
130.55
130.34
126.30
122.48
120.73
119.06
115.60
107.98
107.91
95.41
91.57
90.33
77.48
77.16
76.84
50.18
39.83
39.62
39.41
39.20
38.99
20.57
O
N
Me
OH
MeO2C
(3c)
200
180
160
140
120
100
80
60
40
20
ppm

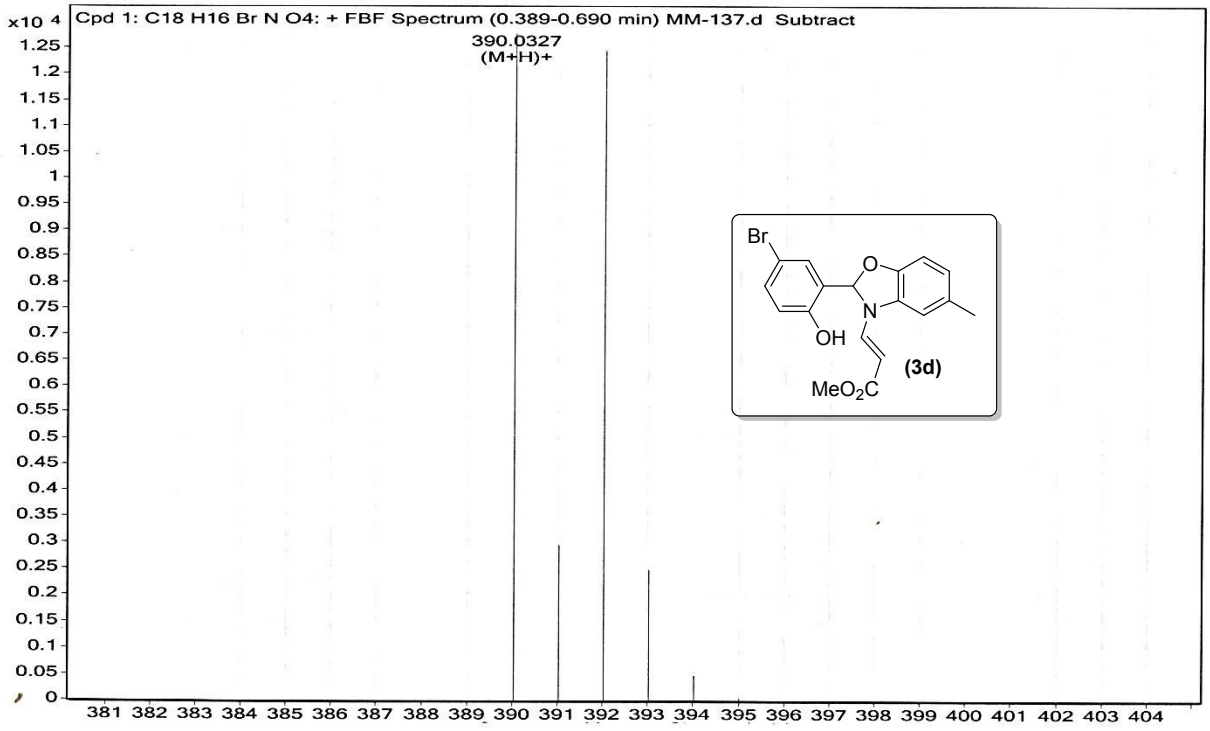
Sample Name	MM-152	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-152.d	**ACQ Method**	Pondicherry Universi				

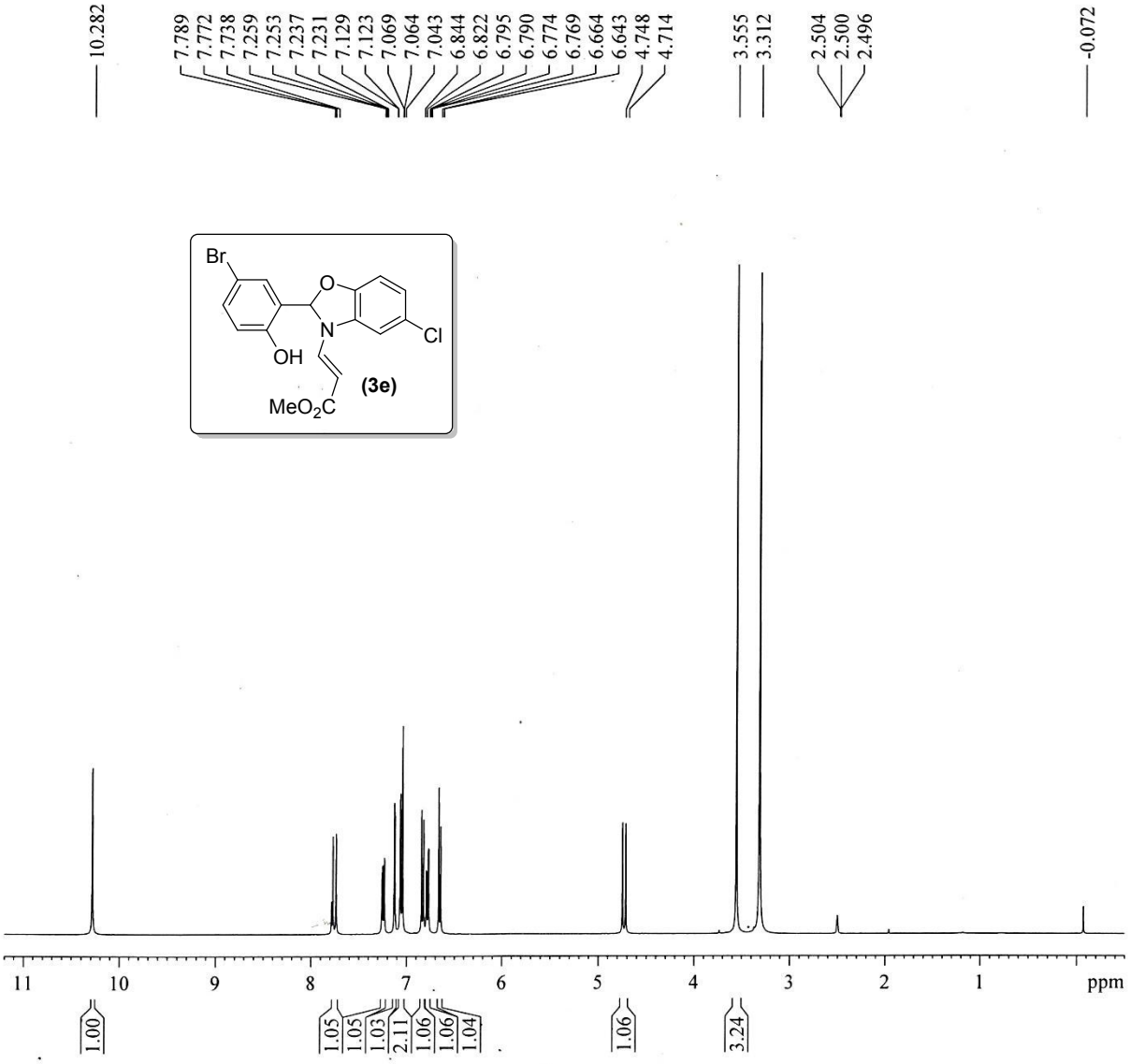


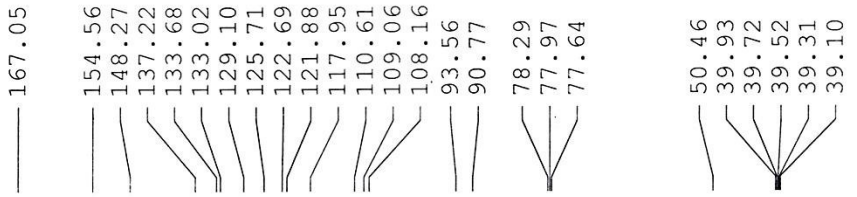

10.466
7.839
7.805
7.324
7.305
7.125
7.022
6.883
6.862
6.660
4.690
4.657
3.549
3.361
2.500
2.276
Br
O
N
OH
(3d)
MeO2C
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.05
1.04
1.14
2.05
1.14
2.10
1.03
3.28
3.02

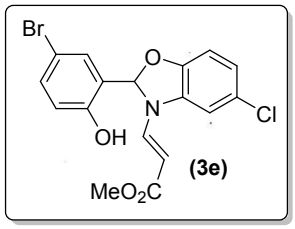

167.12
154.68
147.36
137.76
133.62
131.49
130.76
129.03
123.44
122.89
118.13
110.38
108.95
108.12
91.87
89.71
78.96
78.30
50.35
39.94
39.73
39.52
39.31
39.10
20.70
Br
O
N
OH
(3d)
MeO2C
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

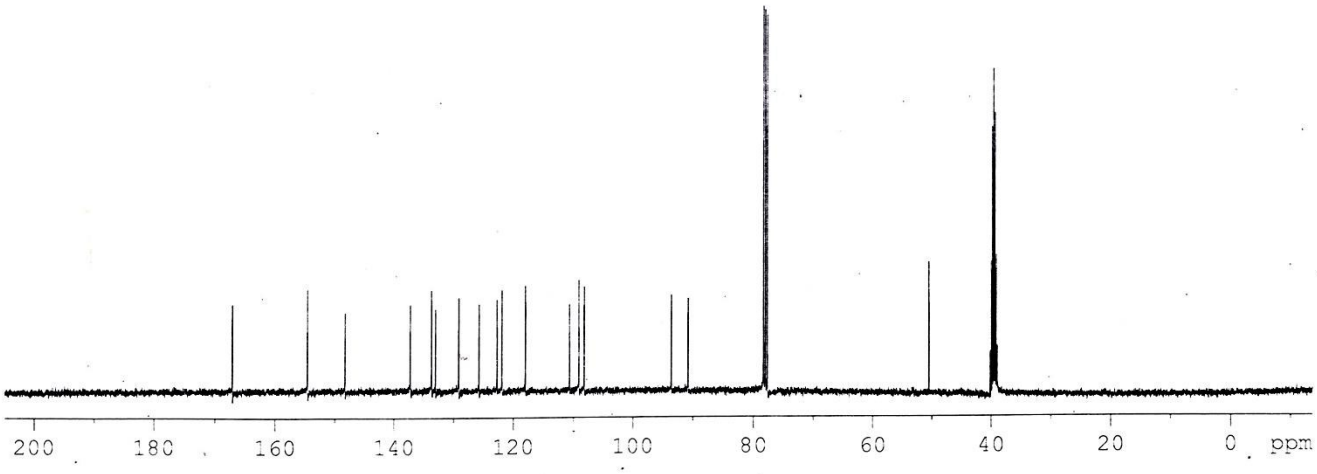
ample Name	MM-137	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-137.d	**ACQ Method**	Pondicherry Universi				

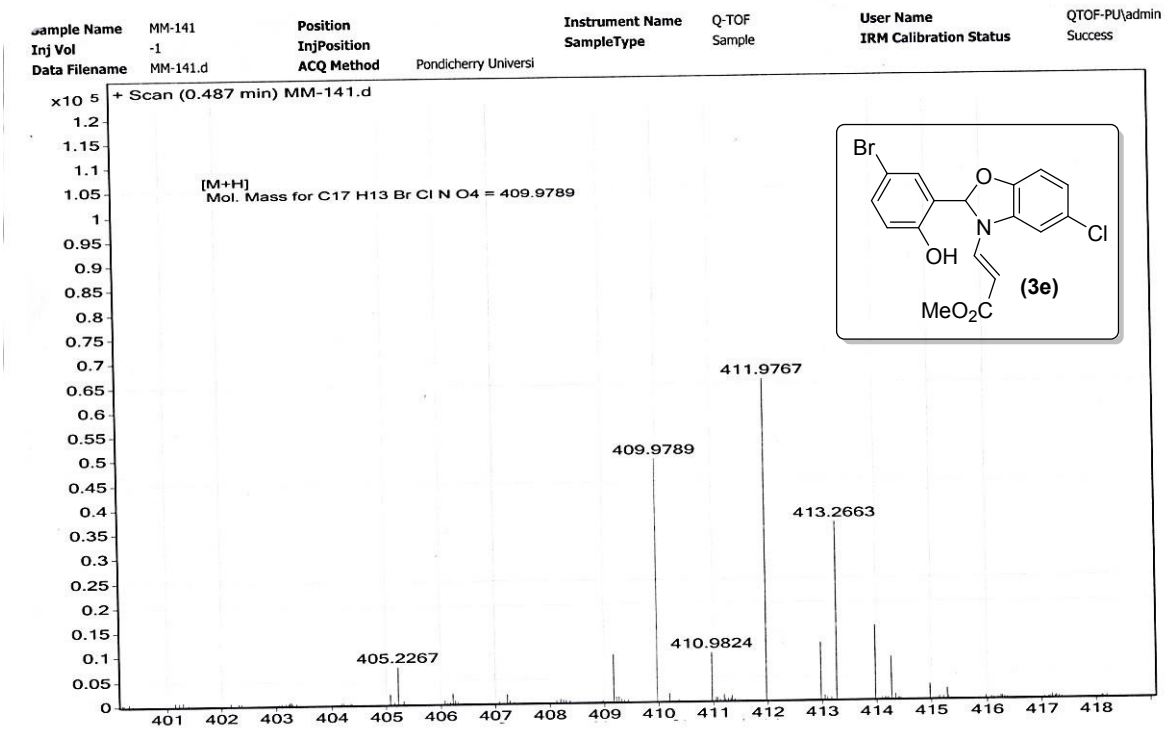


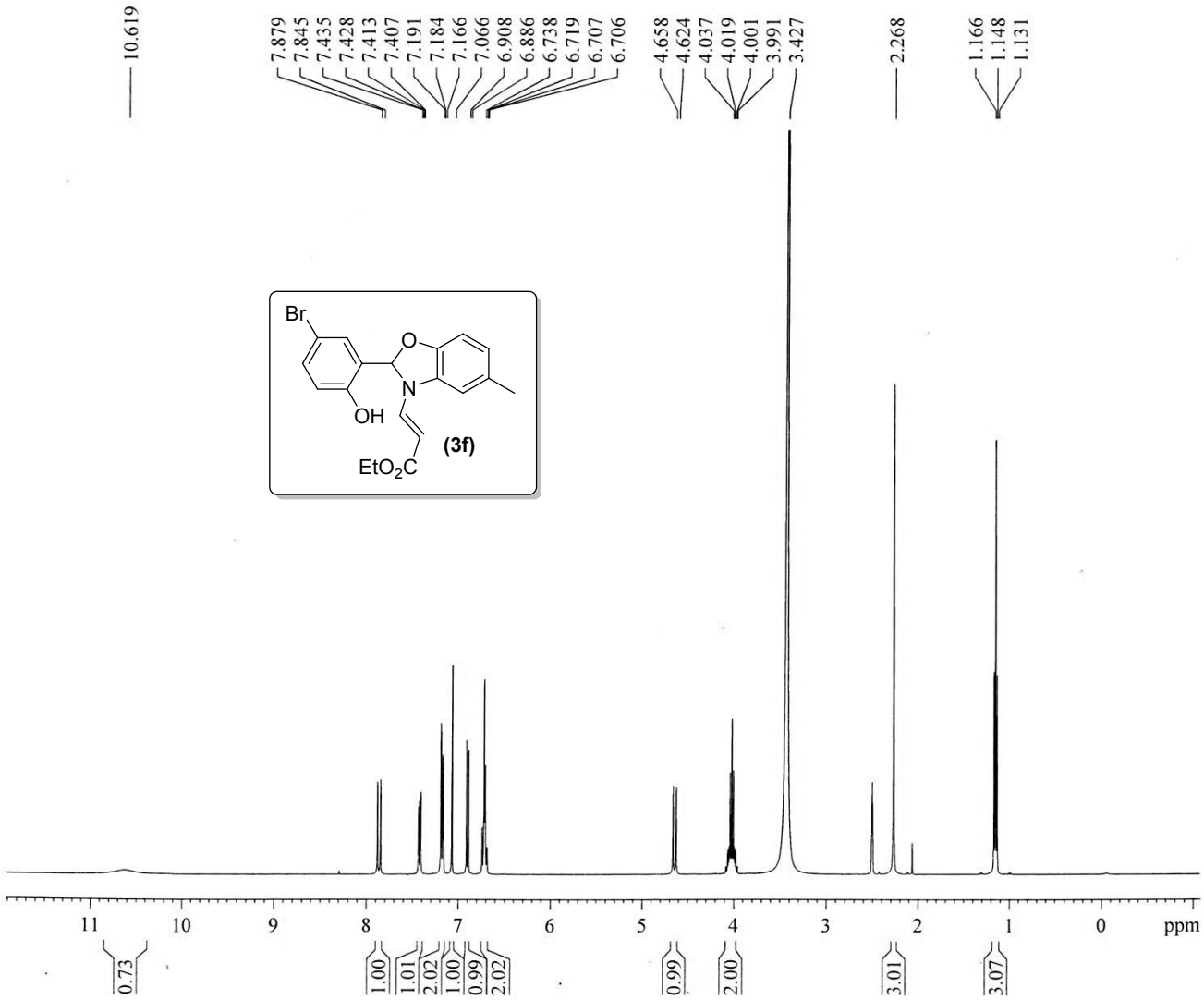

10.282
7.789
7.772
7.738
7.259
7.253
7.237
7.231
7.129
7.123
7.069
7.064
7.043
6.844
6.822
6.795
6.790
6.774
6.769
6.664
6.643
4.748
4.714
3.555
3.312
2.504
2.500
2.496
-0.072
Br
O
N
Cl
OH
MeO2C
(3e)
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.05
1.05
1.03
2.11
1.06
1.06
1.04
1.06
3.24

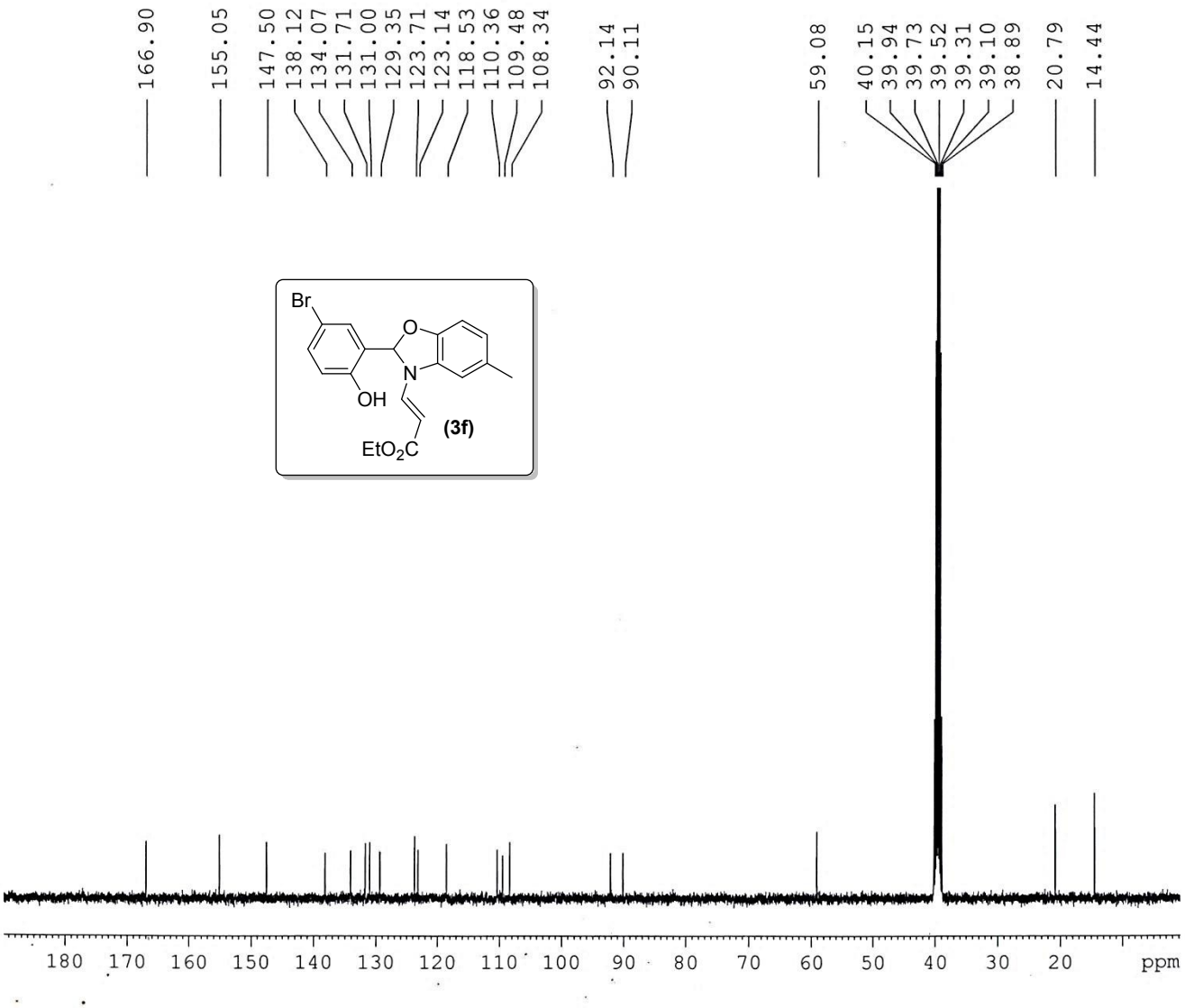

167.05
154.56
148.27
137.22
133.68
133.02
129.10
125.71
122.69
121.88
117.95
110.61
109.06
108.16
93.56
90.77
78.29
77.97
77.64
50.46
39.93
39.72
39.52
39.31
39.10


Br
O
N
Cl
OH
(3e)
MeO2C

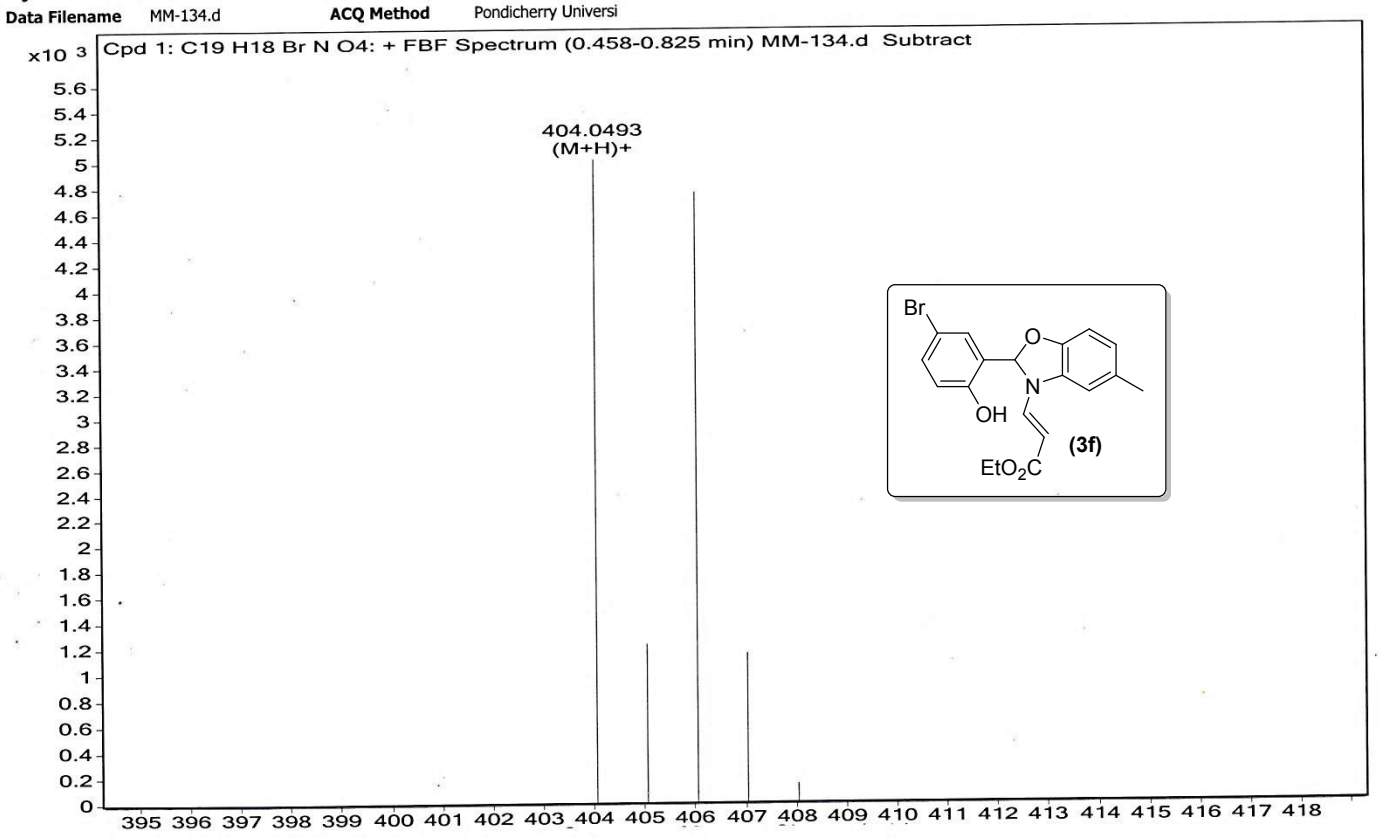

200
180
160
140
120
100
80
60
40
20
0
ppm

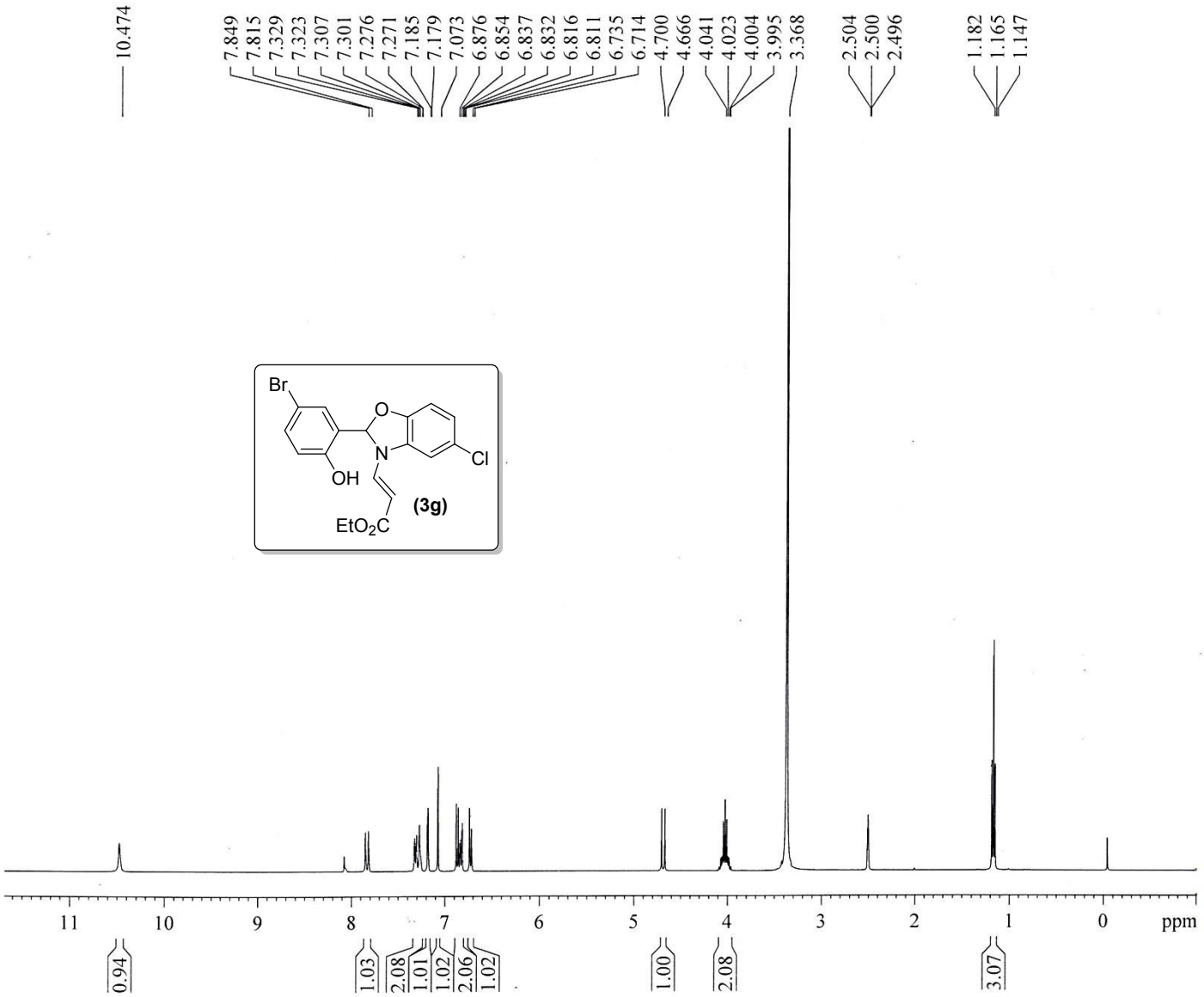

Sample Name MM-141
Inj Vol -1
Data Filename MM-141.d
Position
InjPosition
ACQ Method Pondicherry Universi
Instrument Name Q-TOF
SampleType Sample
User Name QTOF-PU\admin
IRM Calibration Status Success
+ Scan (0.487 min) MM-141.d
x10 5
[M+H]
Mol. Mass for C17 H13 Br Cl N O4 = 409.9789
411.9767
409.9789
413.2663
410.9824
405.2267
Br
O
N
Cl
OH
MeO2C
(3e)

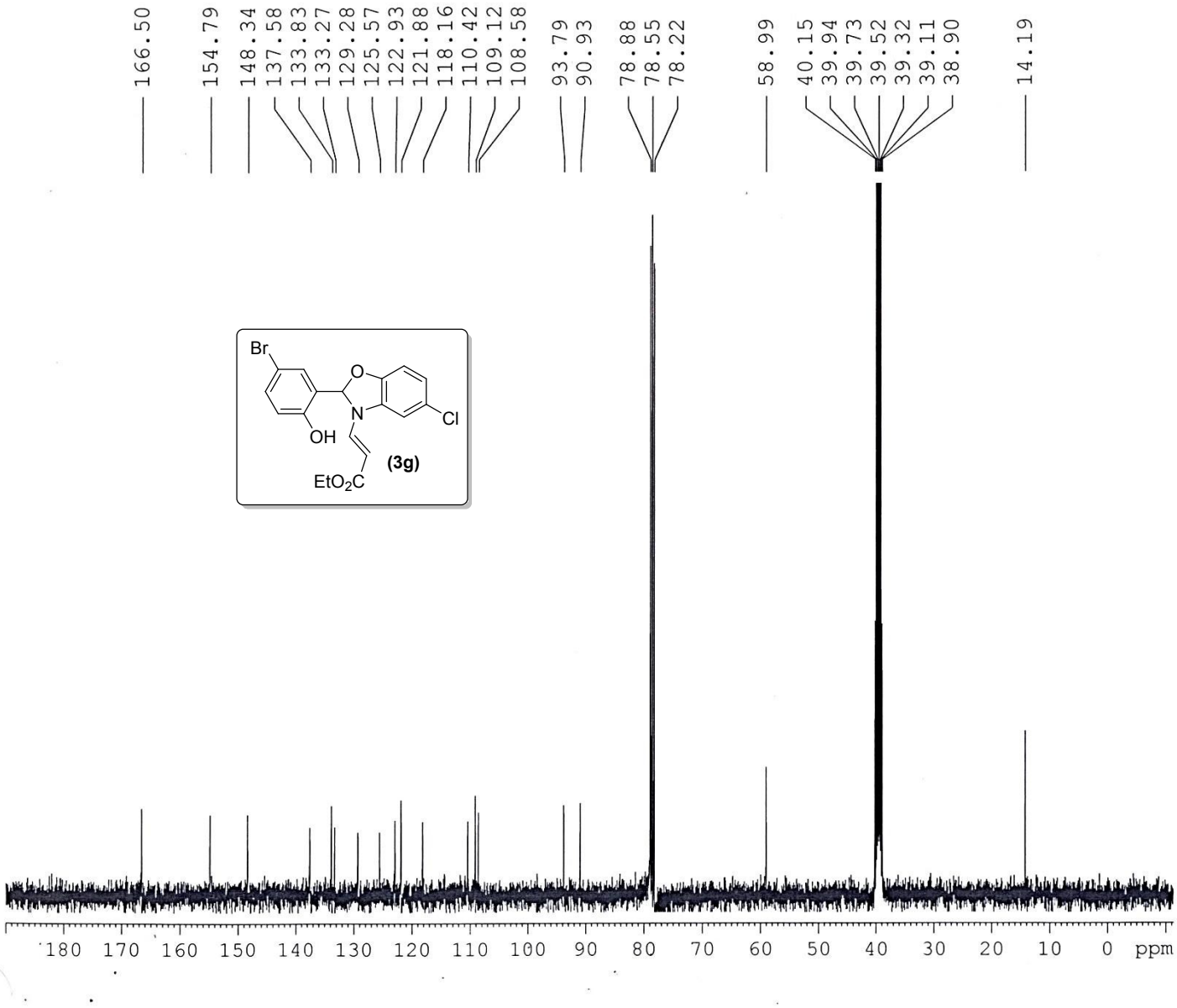

10.619
7.879
7.845
7.435
7.428
7.413
7.407
7.191
7.184
7.166
7.066
6.908
6.886
6.738
6.719
6.707
6.706
4.658
4.624
4.037
4.019
4.001
3.991
3.427
2.268
1.166
1.148
1.131
Br
O
N
OH
EtO2C
(3f)
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.73
1.00
1.01
2.02
1.00
0.99
2.02
0.99
2.00
3.01
3.07


166.90
155.05
147.50
138.12
134.07
131.71
131.00
129.35
123.71
123.14
118.53
110.36
109.48
108.34
92.14
90.11
59.08
40.15
39.94
39.73
39.52
39.31
39.10
38.89
20.79
14.44
Br
O
N
OH
EtO2C
(3f)
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

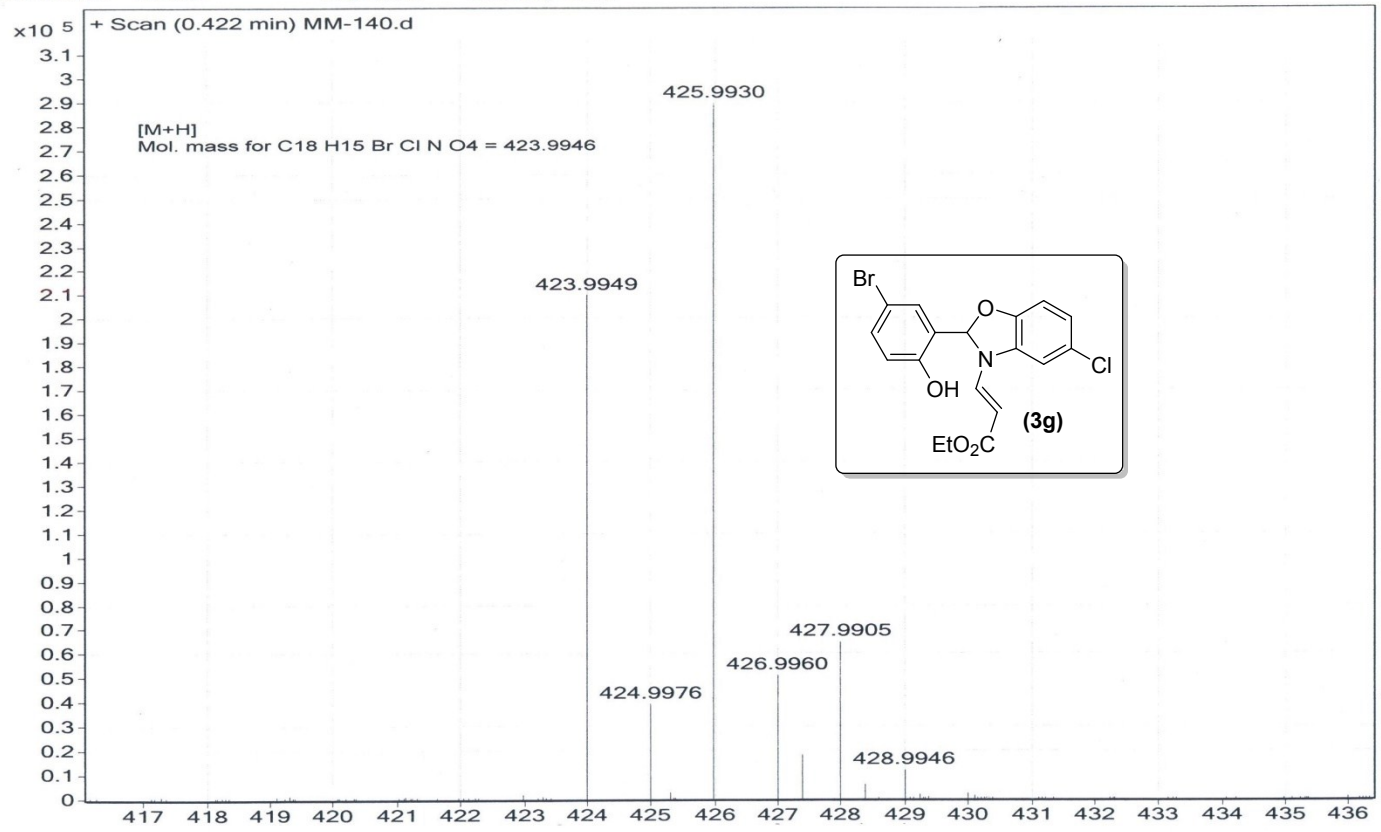
Sample Name	MM-134	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-134.d	**ACQ Method**	Pondicherry Universi				

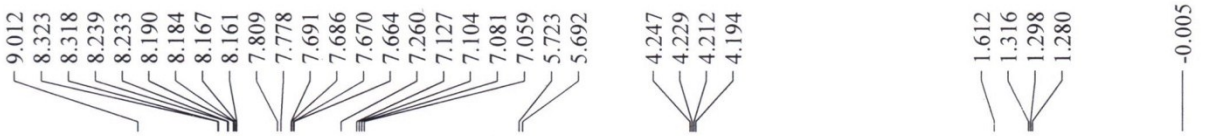


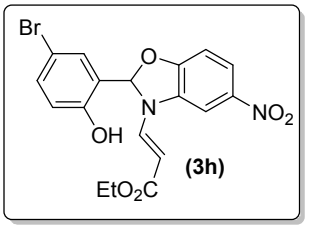

10.474
7.849
7.815
7.329
7.323
7.307
7.301
7.276
7.271
7.185
7.179
7.073
6.876
6.854
6.837
6.832
6.816
6.811
6.735
6.714
4.700
4.666
4.041
4.023
4.004
3.995
3.368
2.504
2.500
2.496
1.182
1.165
1.147
Br
O
N
Cl
OH
EtO2C
(3g)
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.94
1.03
2.08
1.01
1.02
2.06
1.02
1.00
2.08
3.07

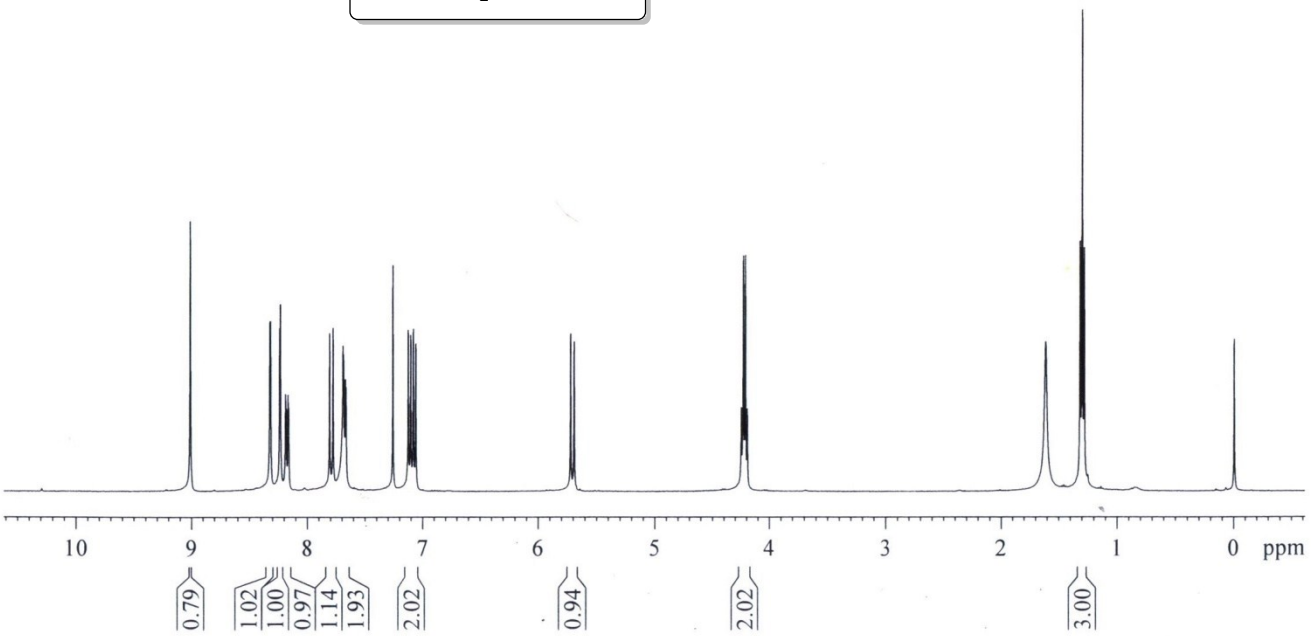

166.50
154.79
148.34
137.58
133.83
133.27
129.28
125.57
122.93
121.88
118.16
110.42
109.12
108.58
93.79
90.93
78.88
78.55
78.22
58.99
40.15
39.94
39.73
39.52
39.32
39.11
38.90
14.19
Br
O
N
Cl
OH
EtO2C
(3g)
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

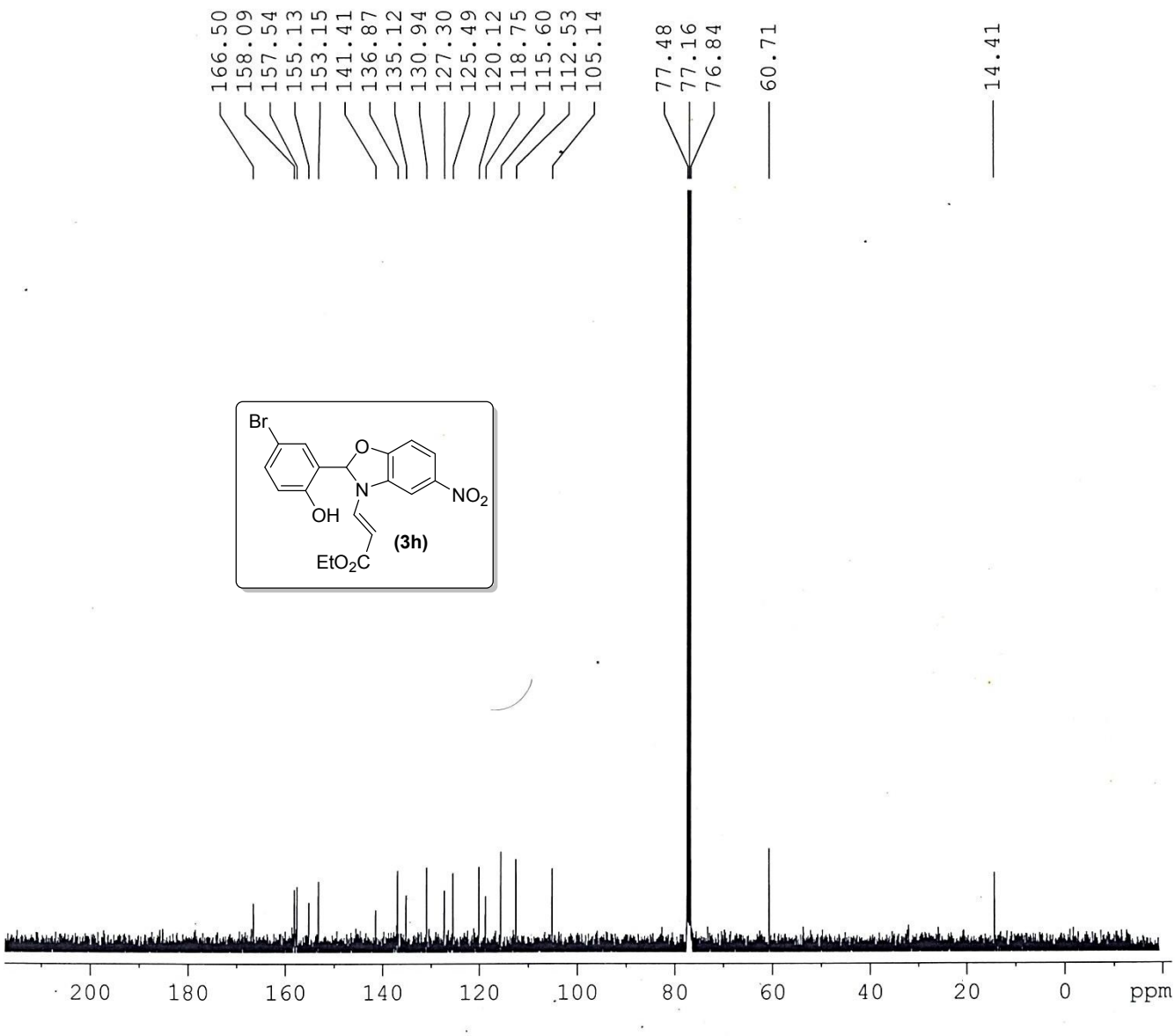
Sample Name	MM-140	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-140.d	**ACQ Method**	Pondicherry Universi				



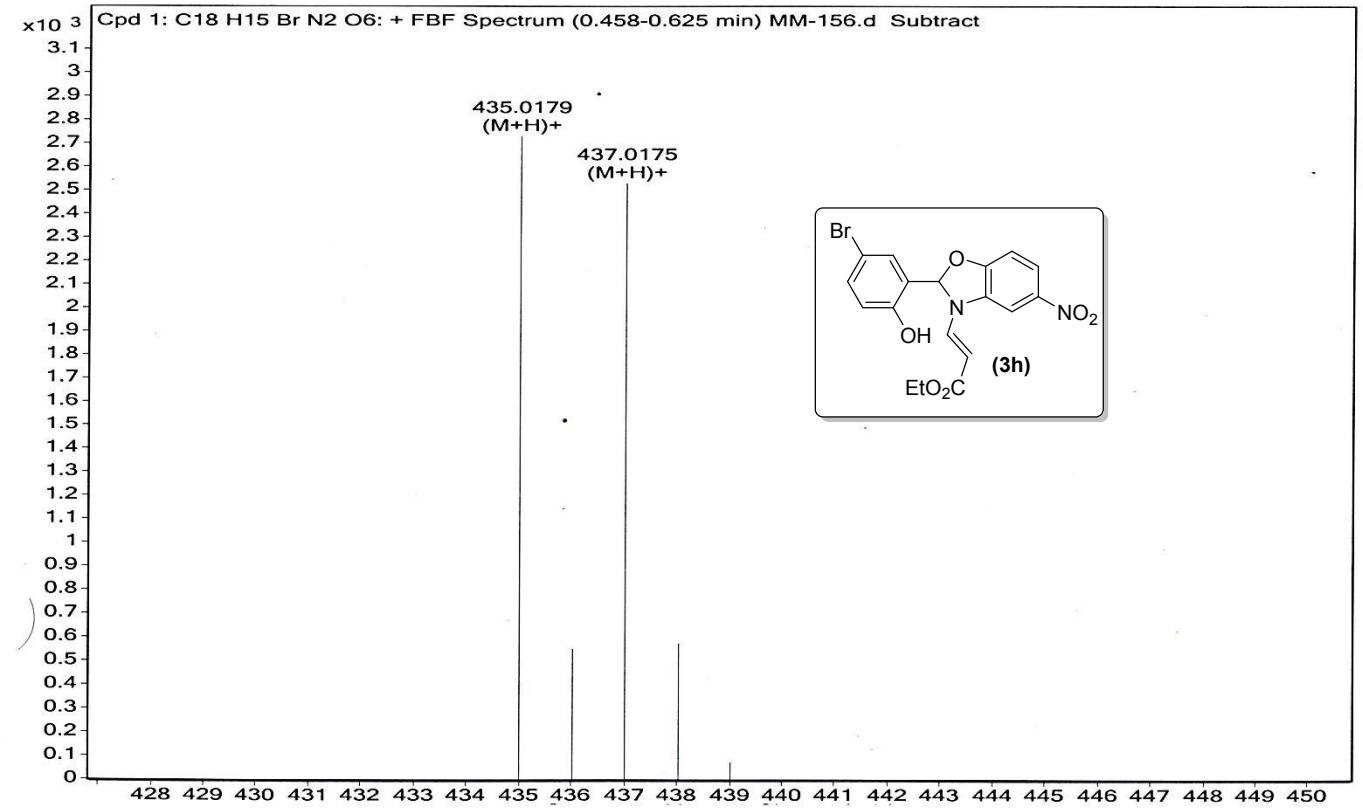

9.012
8.323
8.318
8.239
8.233
8.190
8.184
8.167
8.161
7.809
7.778
7.691
7.686
7.670
7.664
7.260
7.127
7.104
7.081
7.059
5.723
5.692
4.247
4.229
4.212
4.194
1.612
1.316
1.298
1.280
-0.005

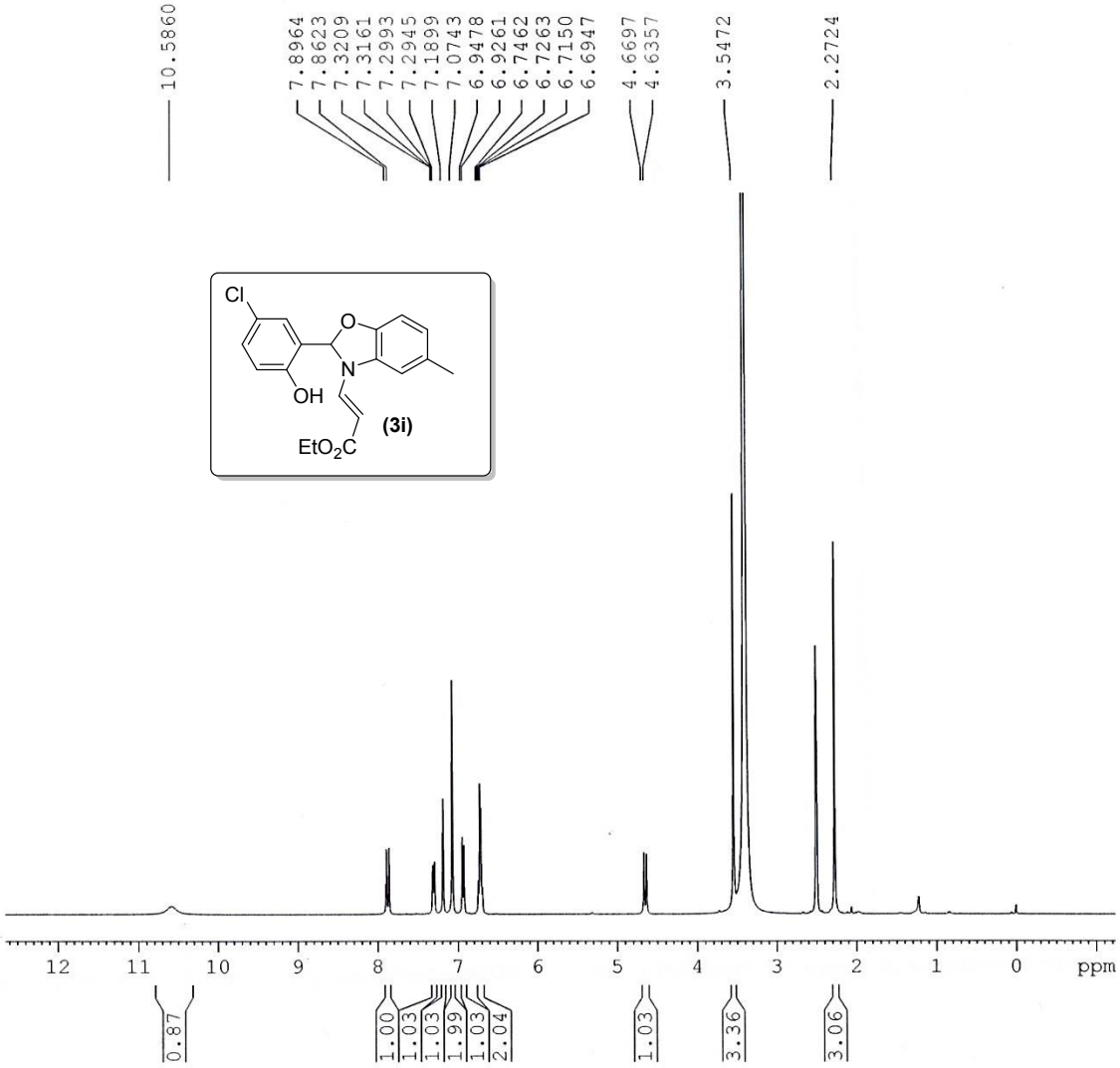

Br
O
N
NO2
OH
EtO2C
(3h)

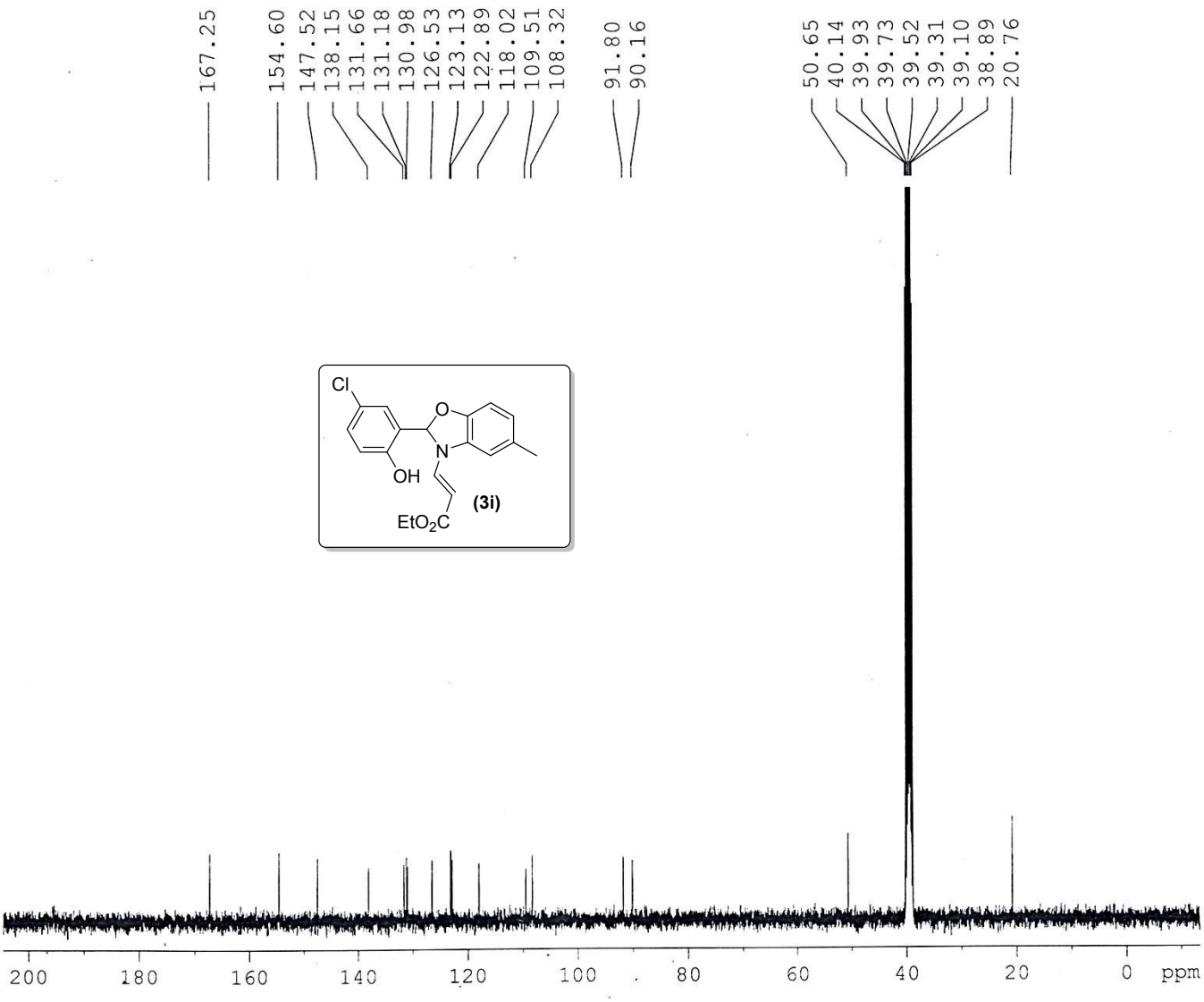

10
9
8
7
6
5
4
3
2
1
0
ppm
0.79
1.02
1.00
0.97
1.14
1.93
2.02
0.94
2.02
3.00


166.50
158.09
157.54
155.13
153.15
141.41
136.87
135.12
130.94
127.30
125.49
120.12
118.75
115.60
112.53
105.14
77.48
77.16
76.84
60.71
14.41
Br
O
N
NO2
OH
EtO2C
(3h)
200
180
160
140
120
100
80
60
40
20
0
ppm

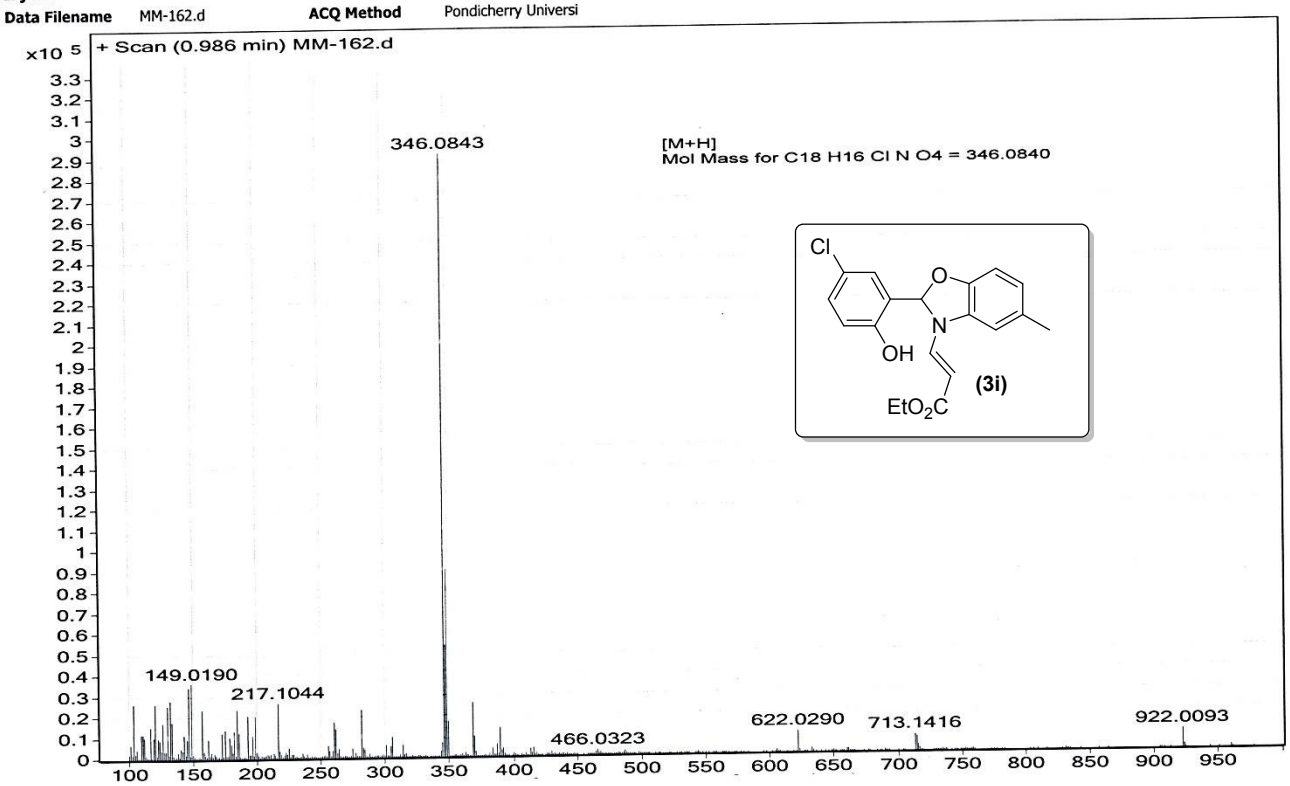
Sample Name	MM-156	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-156.d	**ACQ Method**	Pondicherry Universi				

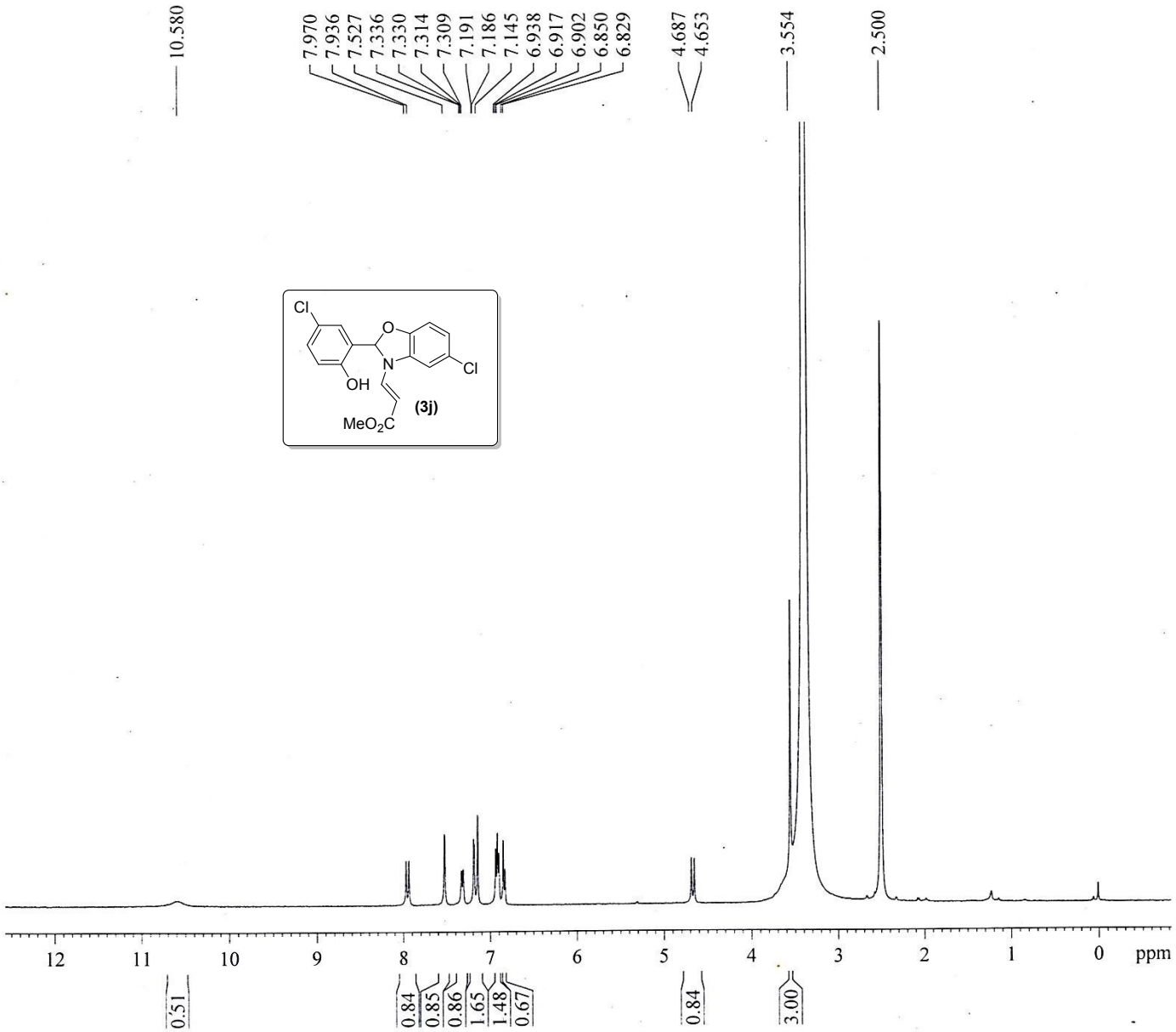


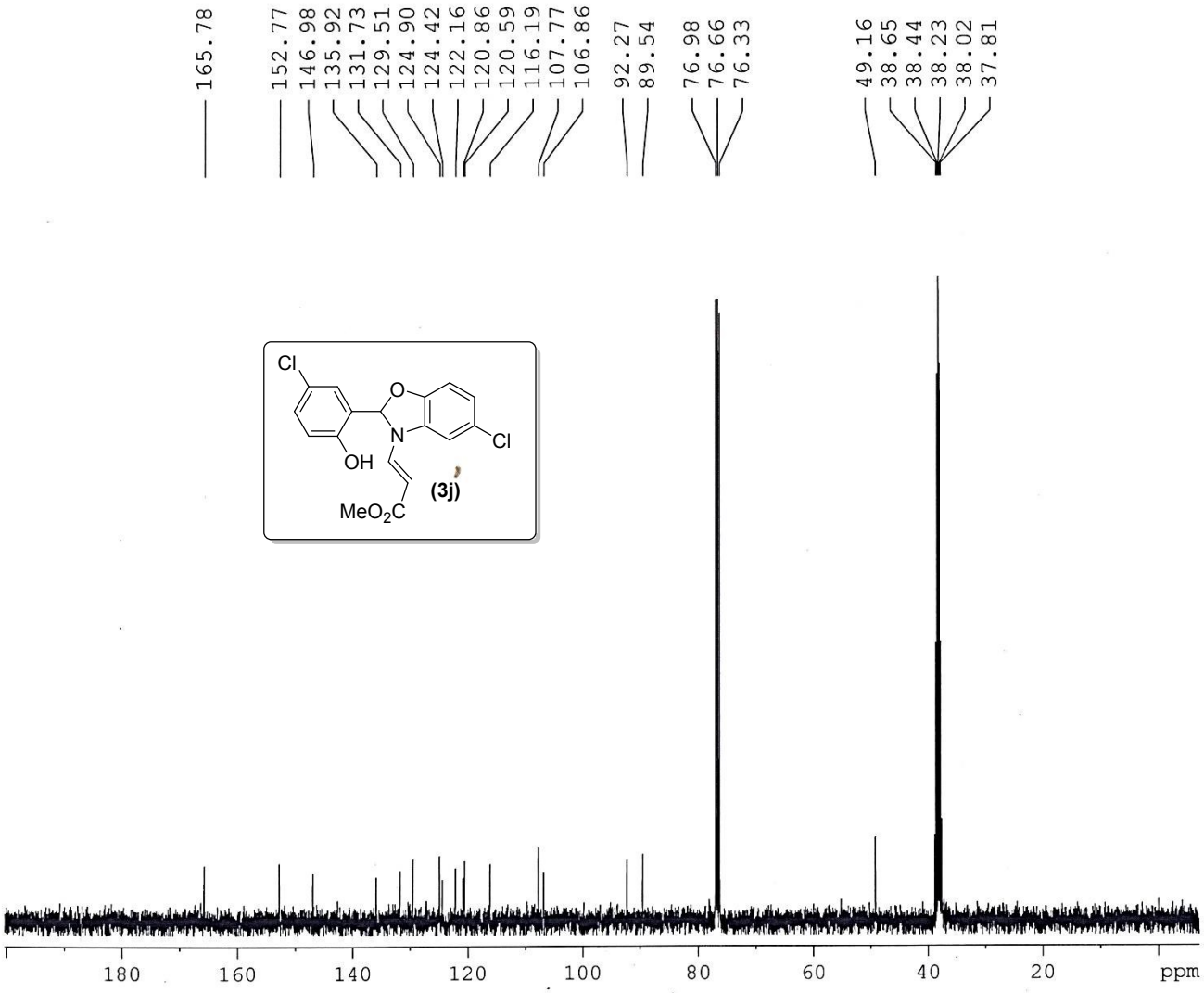

10.5860
7.8964
7.8623
7.3209
7.3161
7.2993
7.2945
7.1899
7.0743
6.9478
6.9261
6.7462
6.7263
6.7150
6.6947
4.6697
4.6357
3.5472
2.2724
Cl
O
N
OH
EtO2C
(3i)
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.87
1.00
1.03
1.03
1.99
1.03
2.04
1.03
3.36
3.06


167.25
154.60
147.52
138.15
131.66
131.18
130.98
126.53
123.13
122.89
118.02
109.51
108.32
91.80
90.16
50.65
40.14
39.93
39.73
39.52
39.31
39.10
38.89
20.76
Cl
O
N
OH
(3i)
EtO2C
200
180
160
140
120
100
80
60
40
20
0
ppm

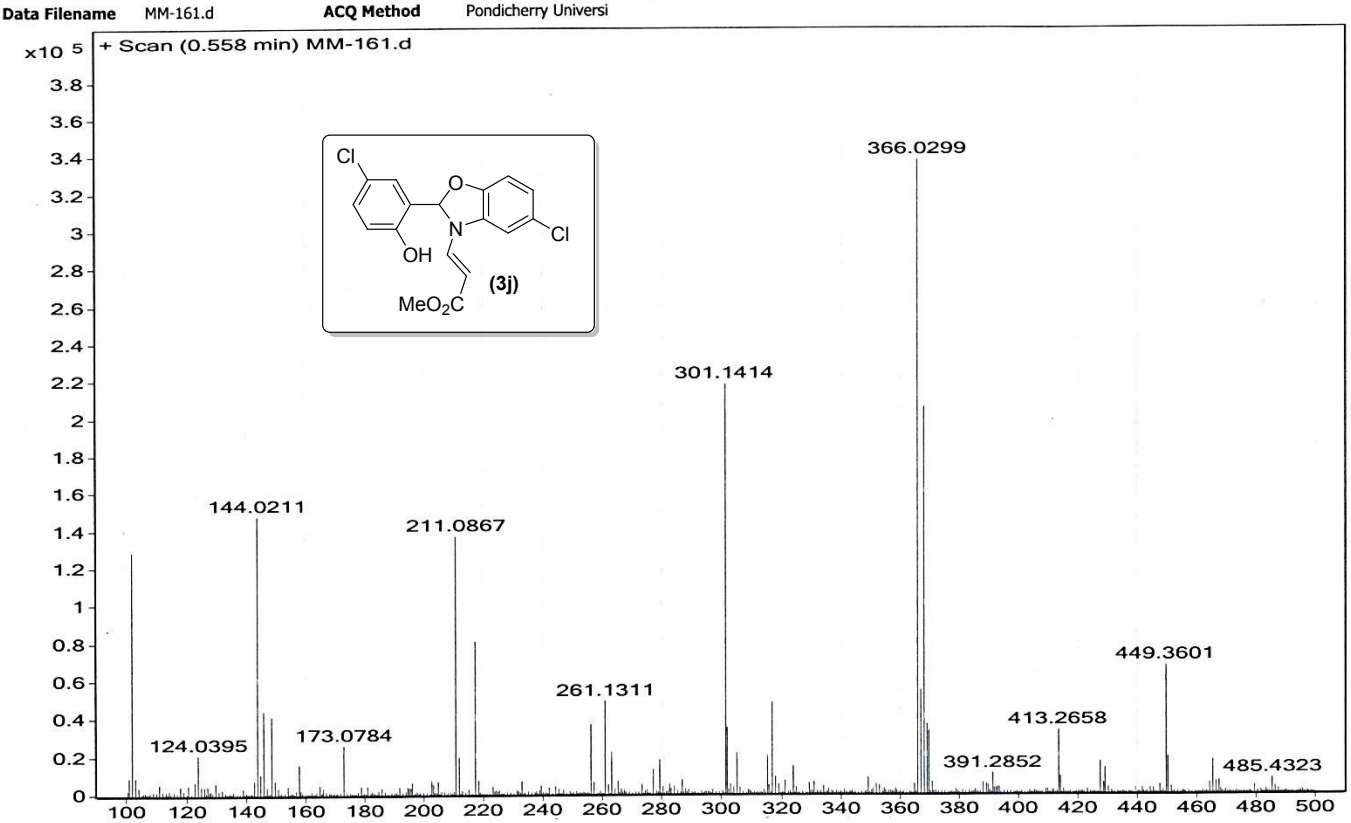
Sample Name	MM-162	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-162.d	**ACQ Method**	Pondicherry Universi				

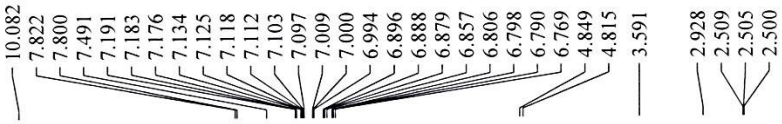


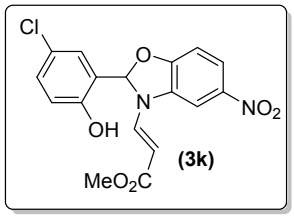

10.580
7.970
7.936
7.527
7.336
7.330
7.314
7.309
7.191
7.186
7.145
6.938
6.917
6.902
6.850
6.829
4.687
4.653
3.554
2.500
Cl
O
N
Cl
OH
(3j)
MeO2C
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.51
0.84
0.85
0.86
1.65
1.48
0.67
0.84
3.00

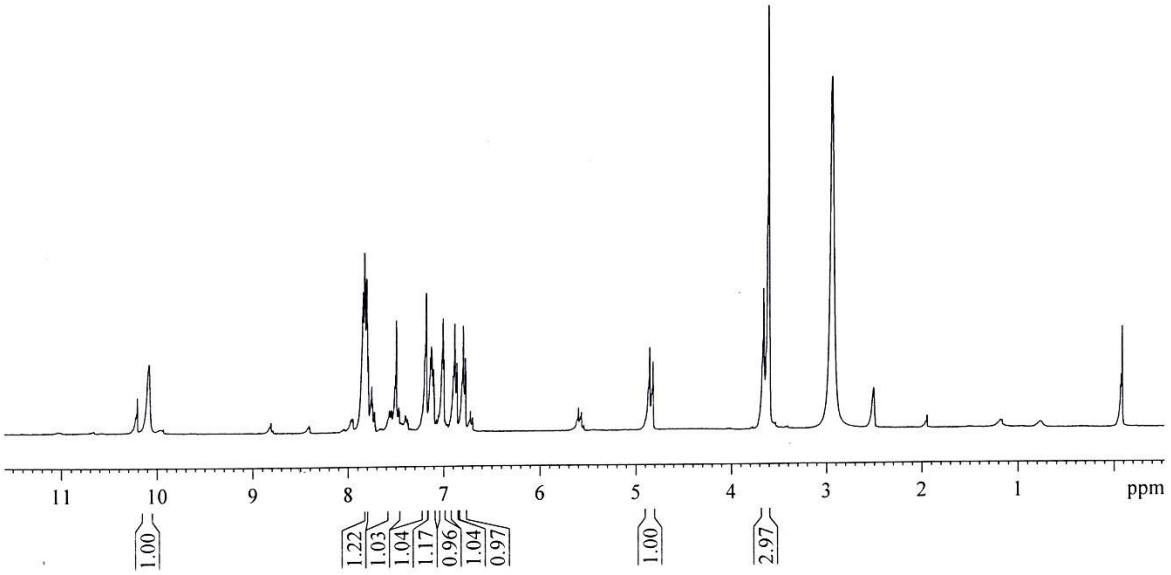

165.78
152.77
146.98
135.92
131.73
129.51
124.90
124.42
122.16
120.86
120.59
116.19
107.77
106.86
92.27
89.54
76.98
76.66
76.33
49.16
38.65
38.44
38.23
38.02
37.81
Cl
O
N
Cl
OH
(3j)
MeO2C
180
160
140
120
100
80
60
40
20
ppm

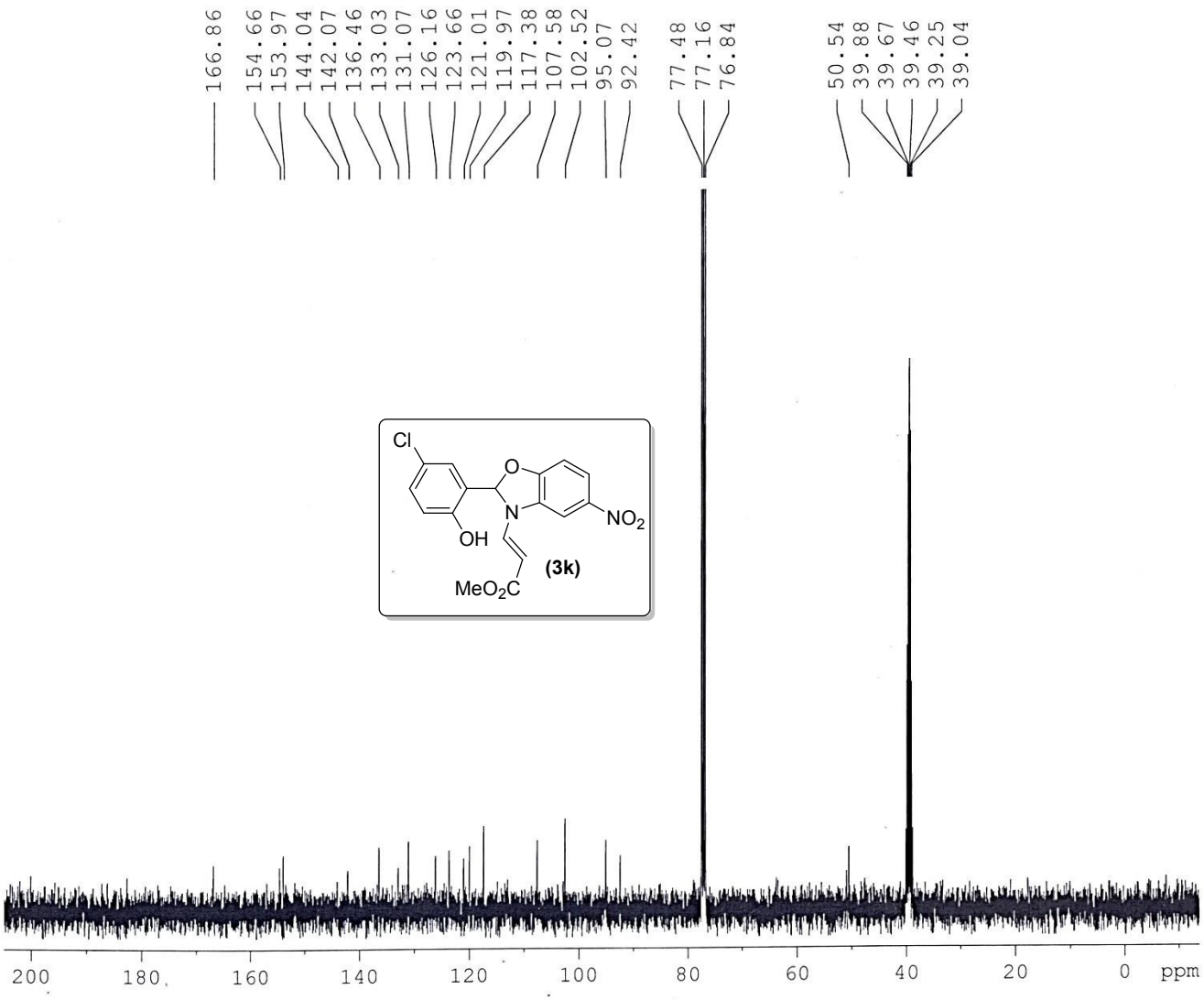
Sample Name	MM-161	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-161.d	**ACQ Method**	Pondicherry Universi				



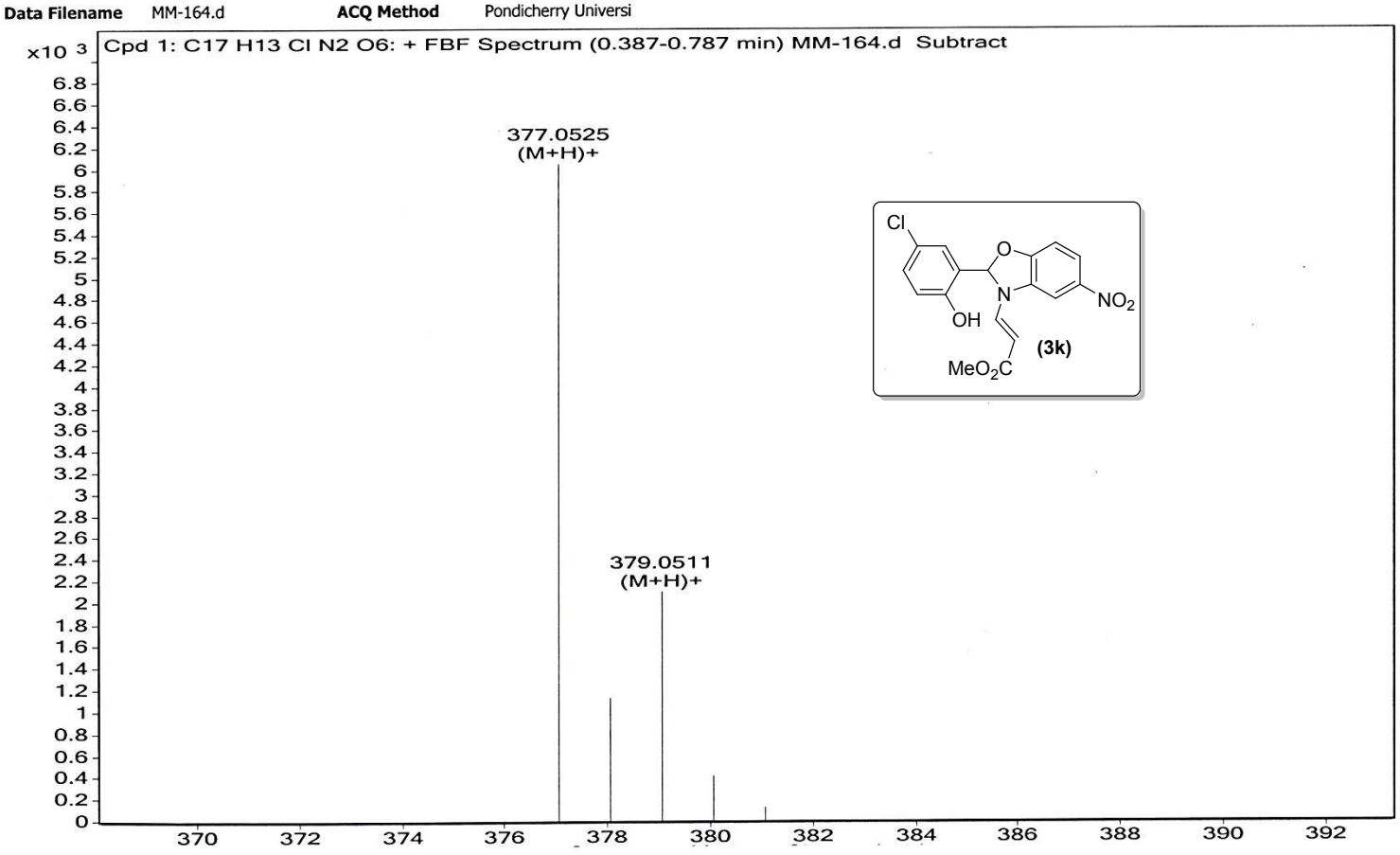

10.082
7.822
7.800
7.491
7.191
7.183
7.176
7.134
7.125
7.118
7.112
7.103
7.097
7.009
7.000
6.994
6.896
6.888
6.879
6.857
6.806
6.798
6.790
6.769
4.849
4.815
3.591
2.928
2.509
2.505
2.500


Cl
O
N
NO2
OH
(3k)
MeO2C

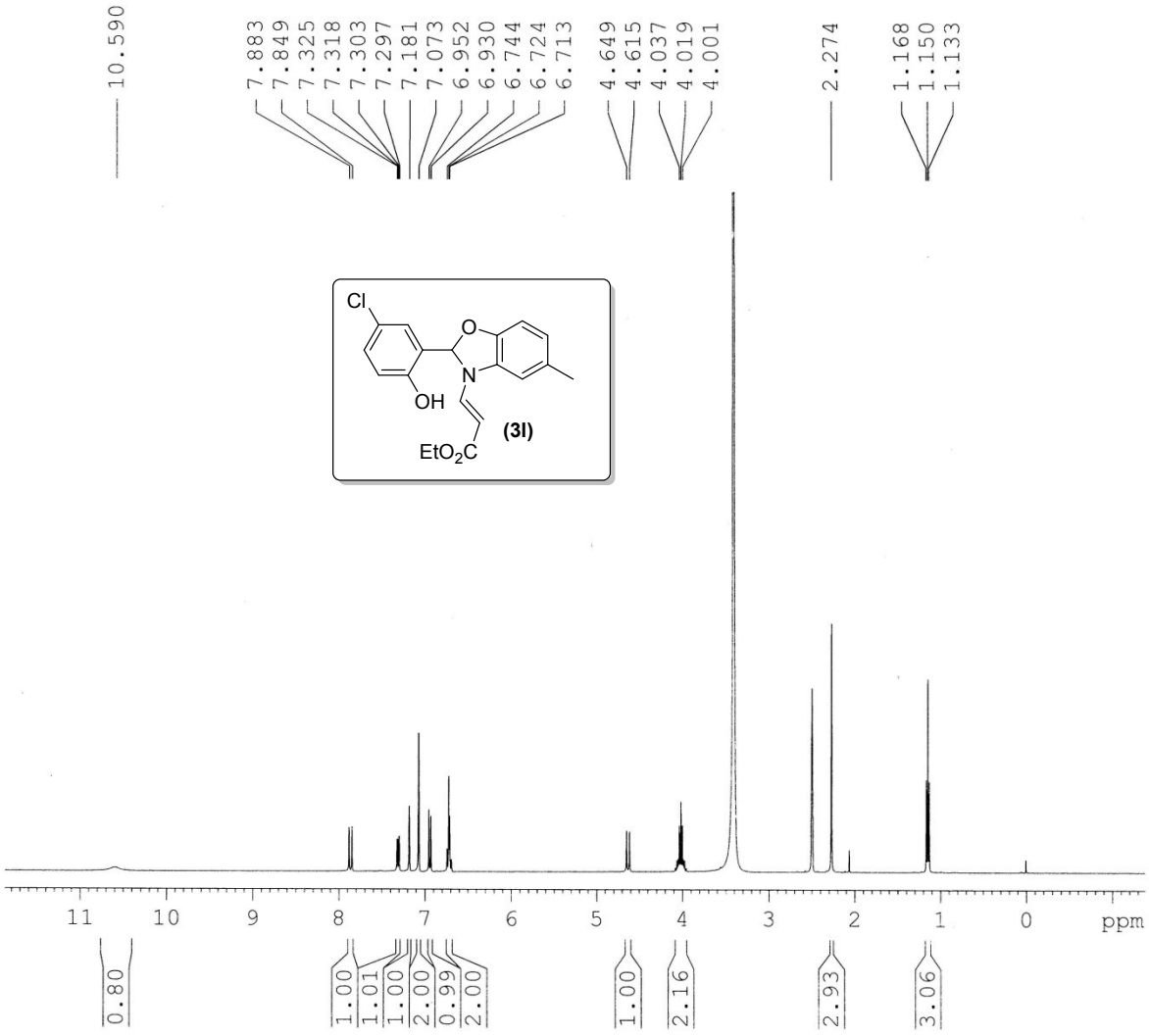

11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.22
1.03
1.04
1.17
0.96
1.04
0.97
1.00
2.97

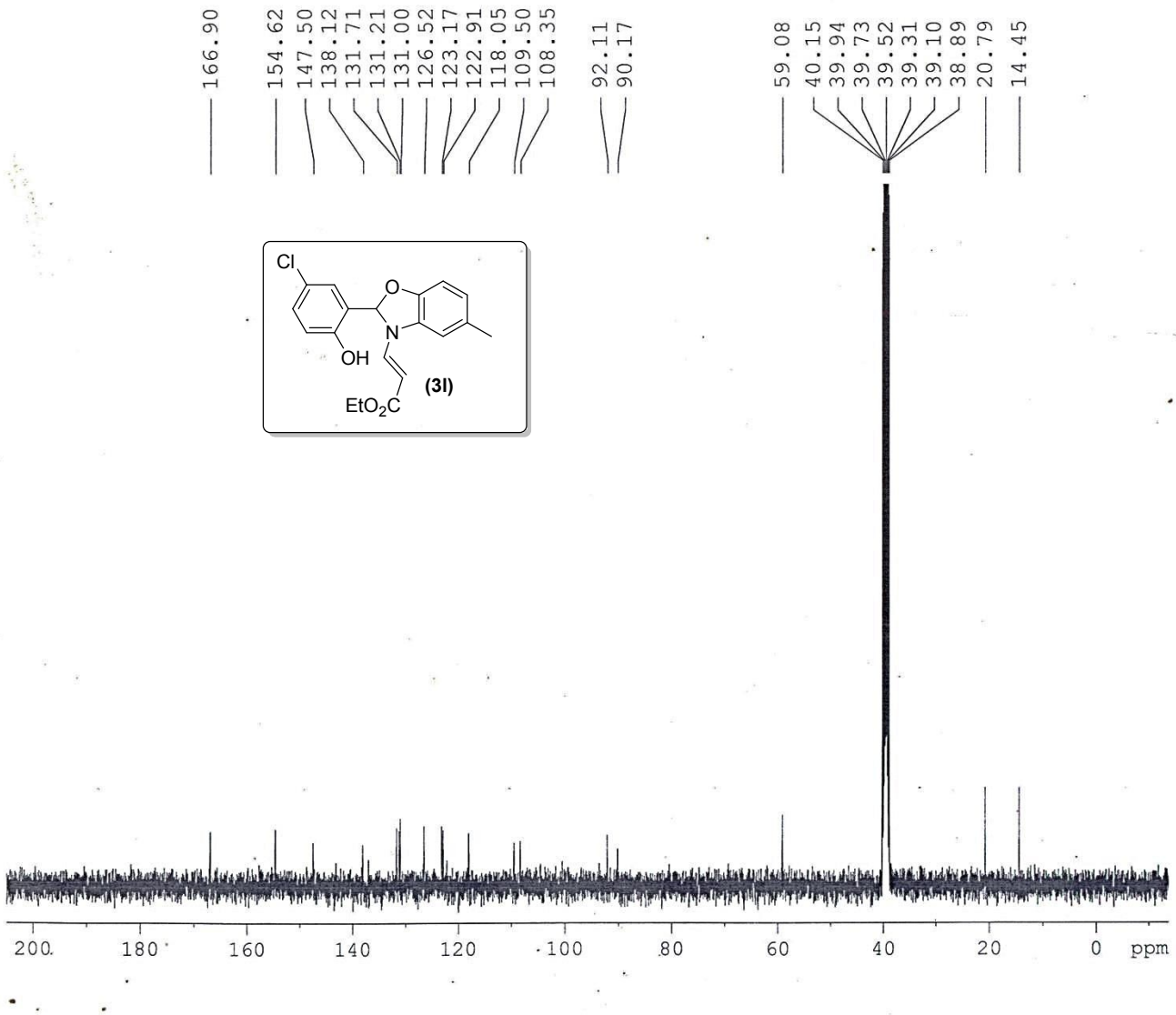

166.86
154.66
153.97
144.04
142.07
136.46
133.03
131.07
126.16
123.66
121.01
119.97
117.38
107.58
102.52
95.07
92.42
77.48
77.16
76.84
50.54
39.88
39.67
39.46
39.25
39.04
Cl
O
N
NO2
OH
(3k)
MeO2C
200
180
160
140
120
100
80
60
40
20
0
ppm

Sample Name	MM-164	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-164.d	**ACQ Method**	Pondicherry Universi				

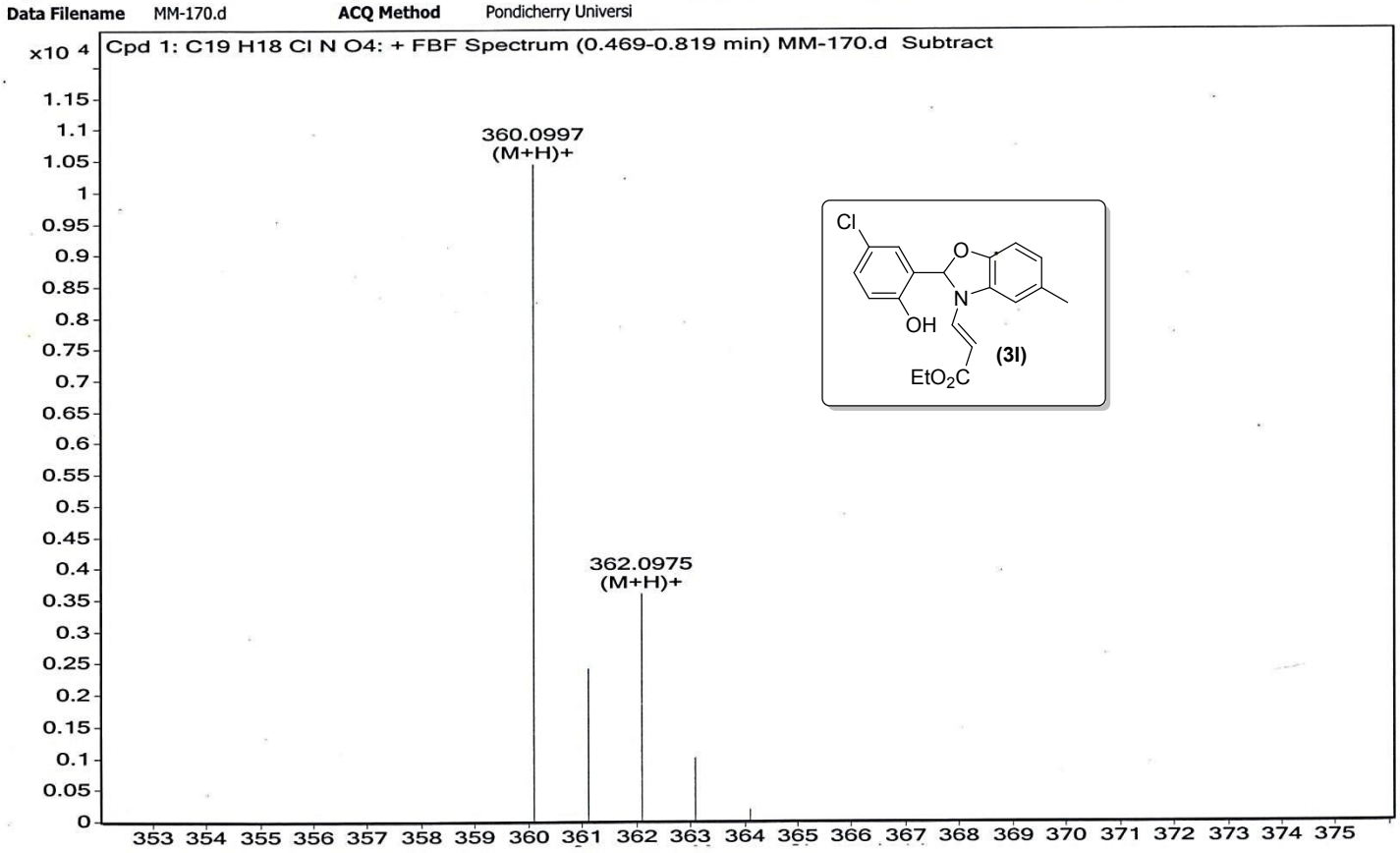


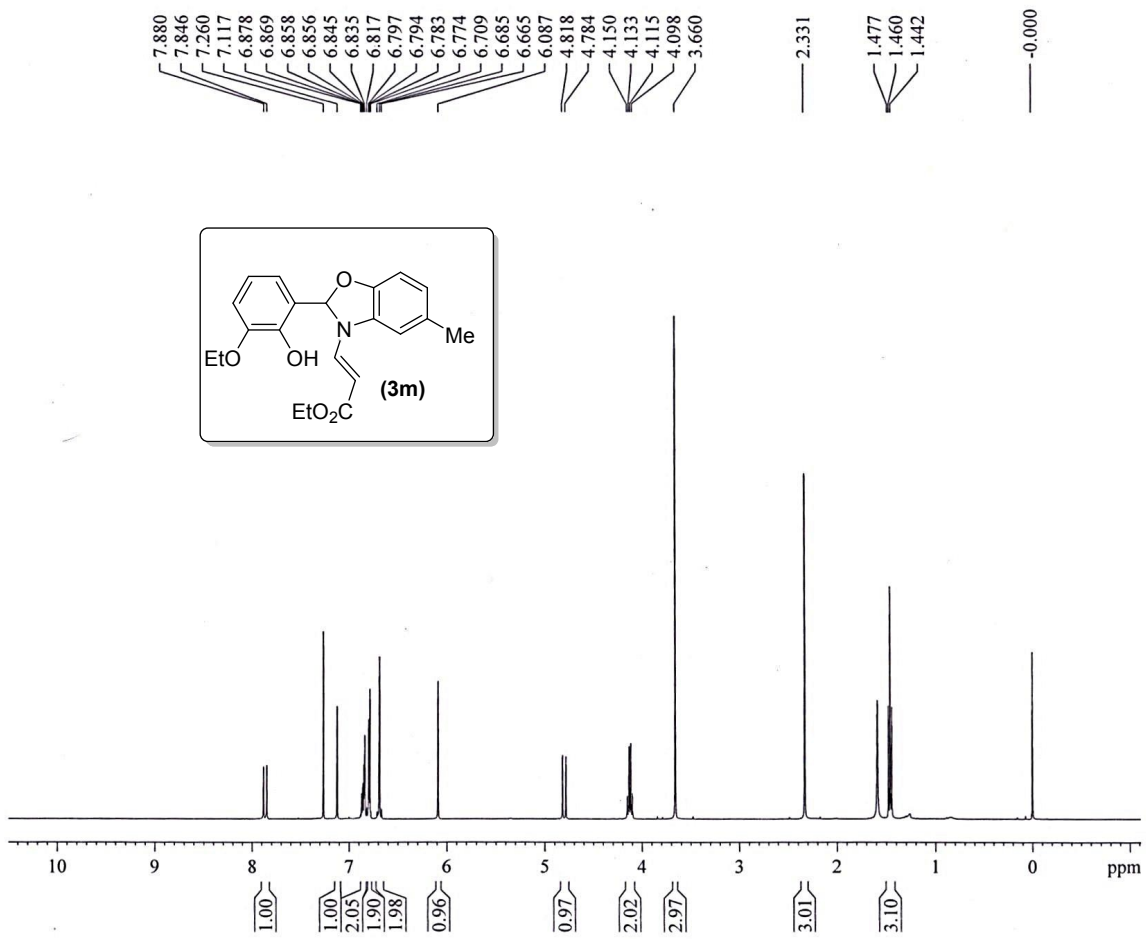
PROTON DMSO {D:\MB} KOPAL 1

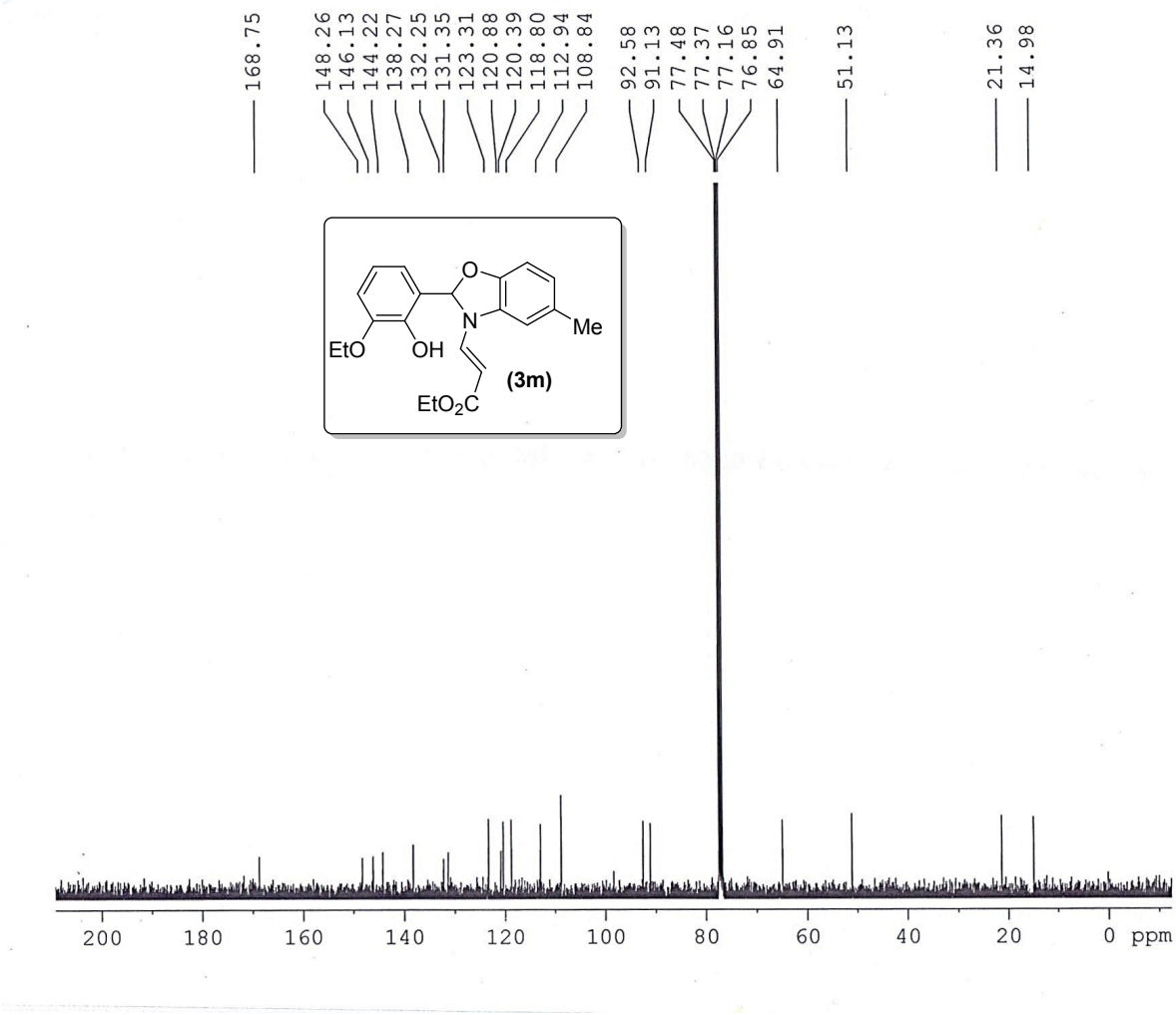



166.90
154.62
147.50
138.12
131.71
131.21
131.00
126.52
123.17
122.91
118.05
109.50
108.35
92.11
90.17
59.08
40.15
39.94
39.73
39.52
39.31
39.10
38.89
20.79
14.45
Cl
O
N
OH
(3l)
EtO2C
200
180
160
140
120
100
80
60
40
20
0
ppm

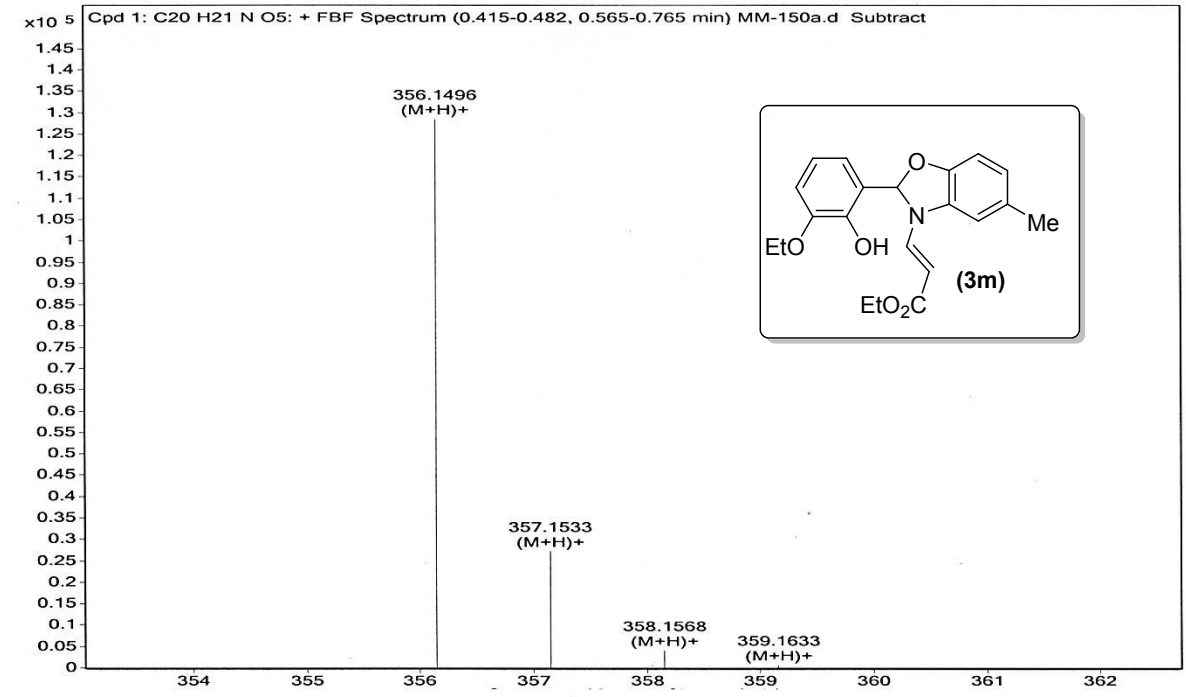
Sample Name	MM-170	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-170.d	**ACQ Method**	Pondicherry Universi				

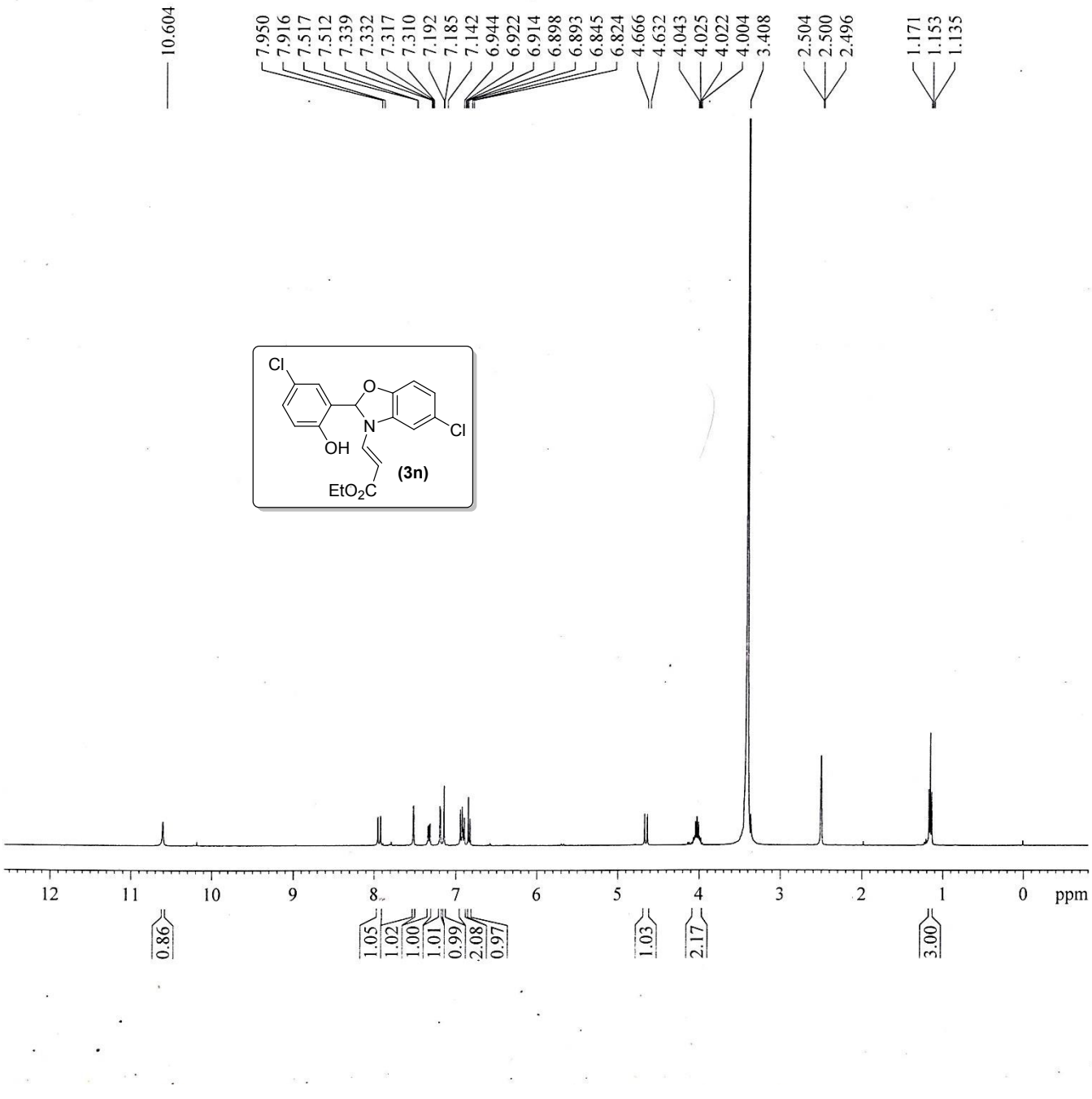


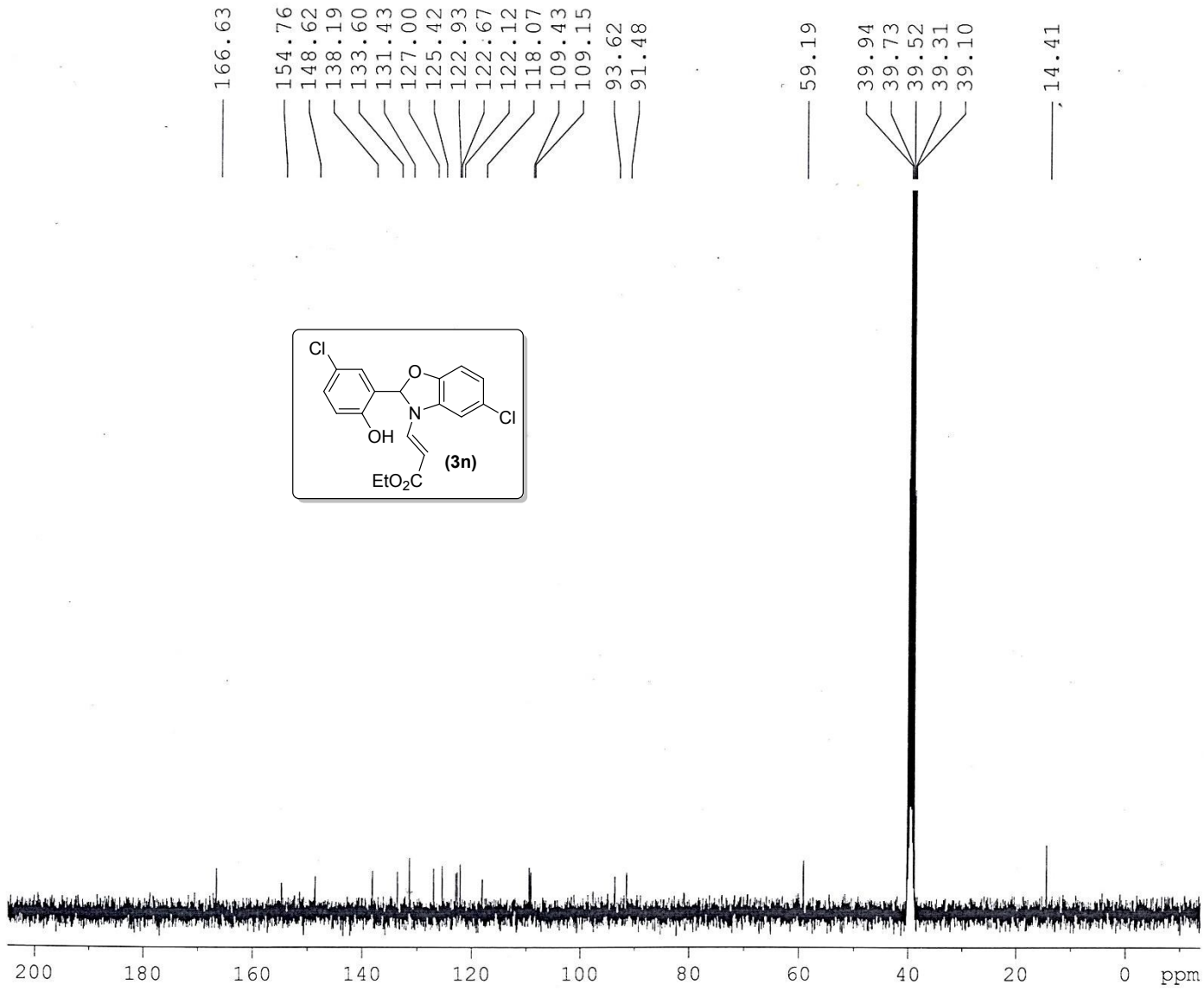

7.880
7.846
7.260
7.117
6.878
6.869
6.858
6.856
6.845
6.835
6.817
6.797
6.794
6.783
6.774
6.709
6.685
6.665
6.087
4.818
4.784
4.150
4.133
4.115
4.098
3.660
2.331
1.477
1.460
1.442
-0.000
O
N
Me
EtO
OH
(3m)
EtO2C
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.00
2.05
1.90
1.98
0.96
0.97
2.02
2.97
3.01
3.10


168.75
148.26
146.13
144.22
138.27
132.25
131.35
123.31
120.88
120.39
118.80
112.94
108.84
92.58
91.13
77.48
77.37
77.16
76.85
64.91
51.13
21.36
14.98
O
N
Me
EtO
OH
(3m)
EtO2C
200
180
160
140
120
100
80
60
40
20
0 ppm

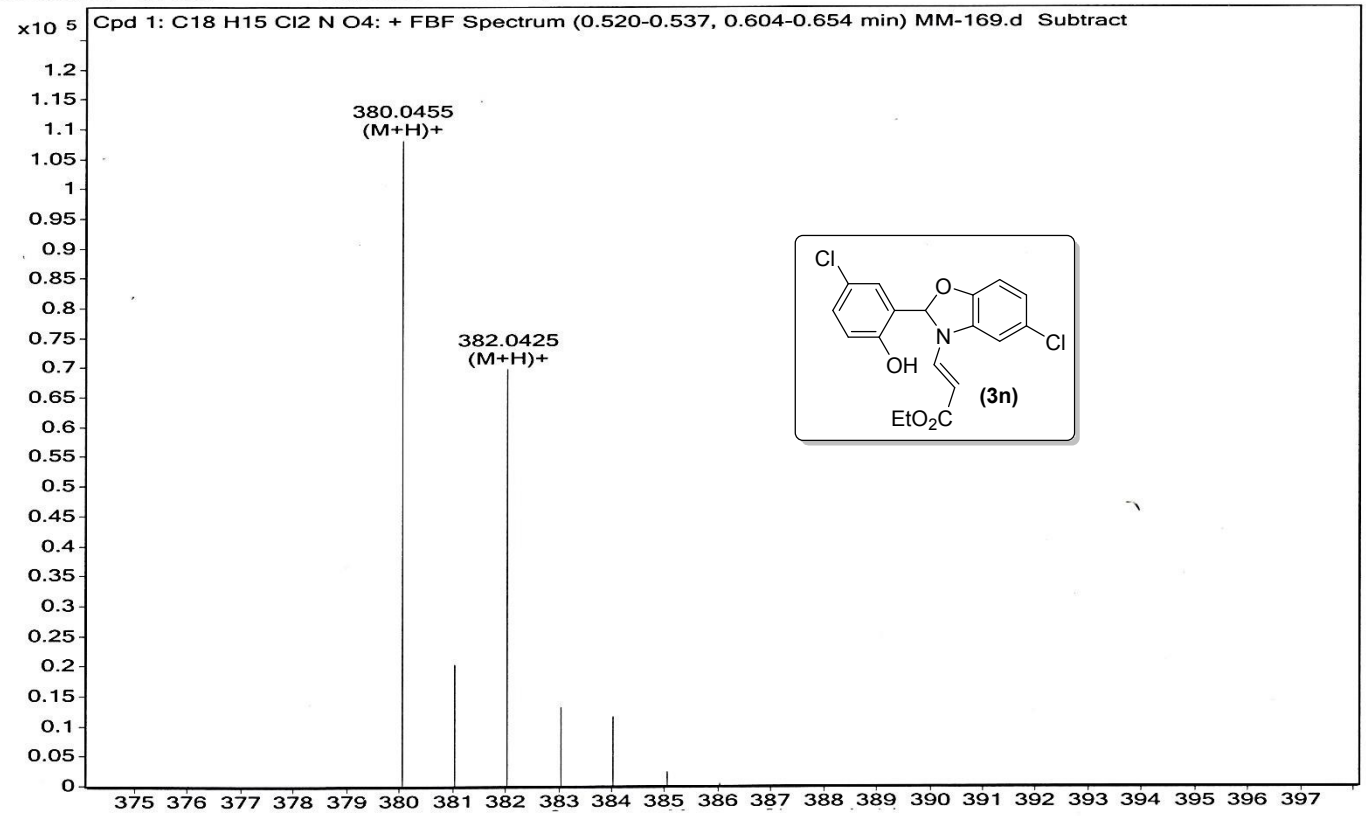
Sample Name	MM-150	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-150a.d	**ACQ Method**	Pondicherry Universi				

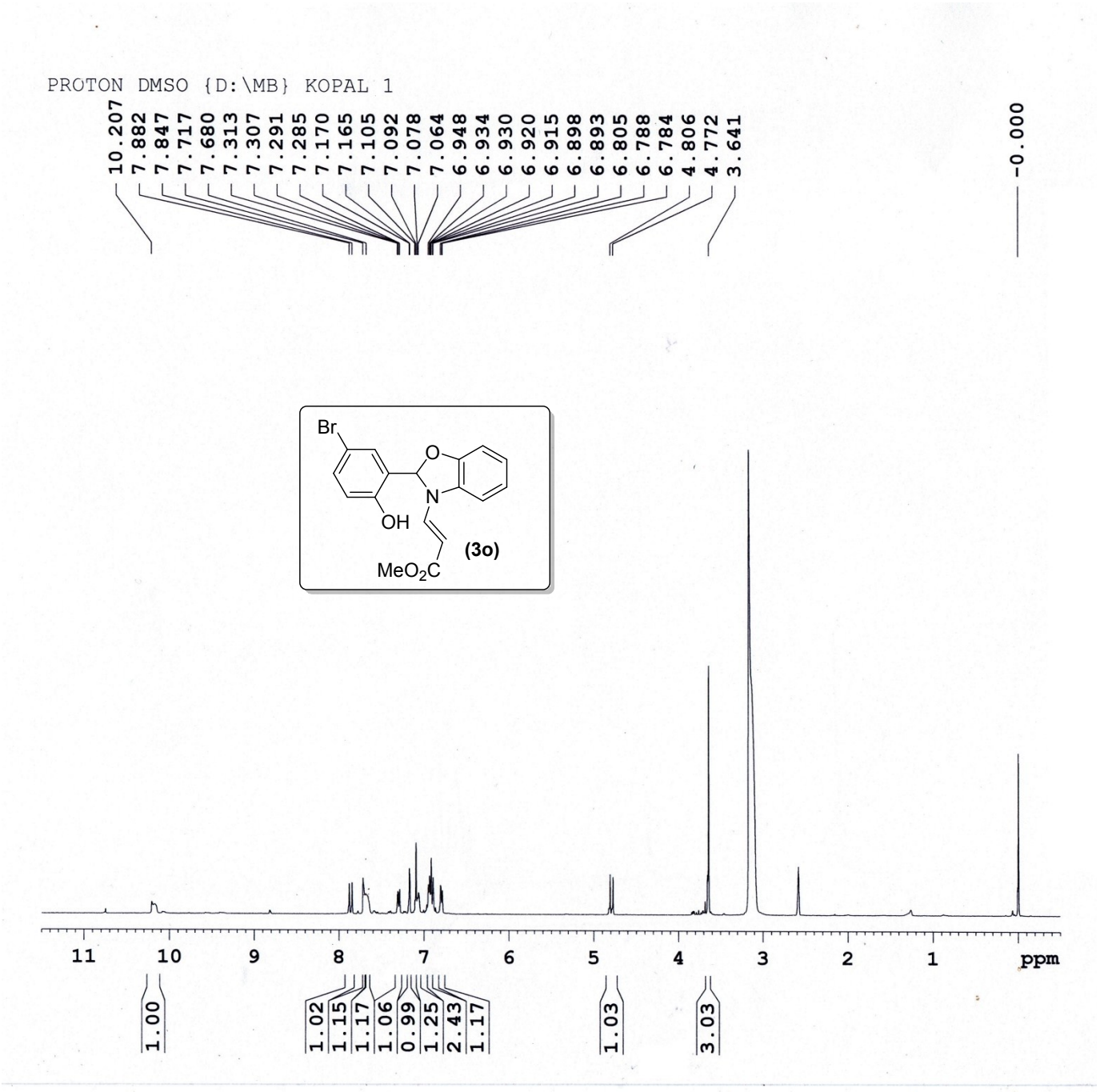


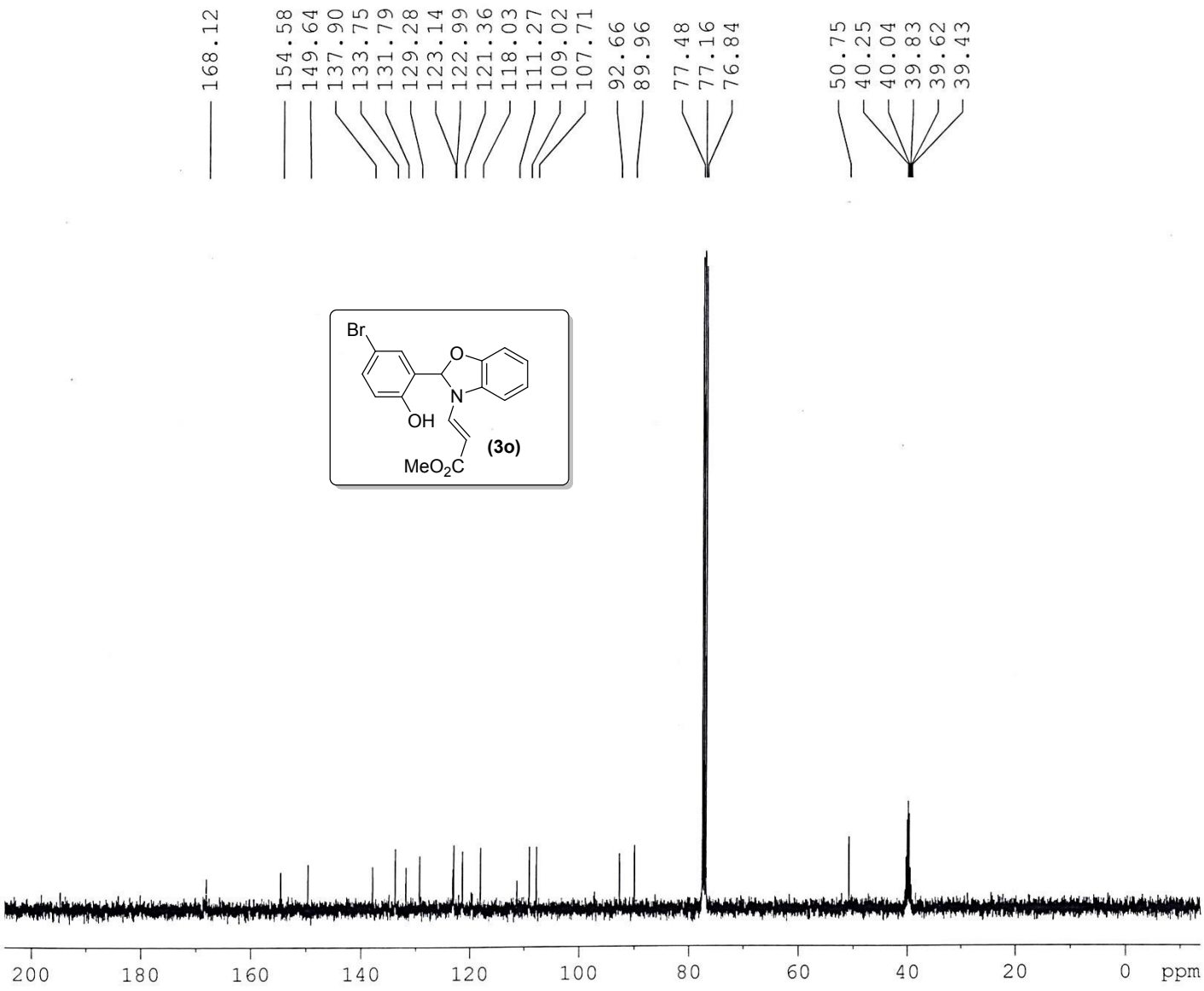

10.604
7.950
7.916
7.517
7.512
7.339
7.332
7.317
7.310
7.192
7.185
7.142
6.944
6.922
6.914
6.898
6.893
6.845
6.824
4.666
4.632
4.043
4.025
4.022
4.004
3.408
2.504
2.500
2.496
1.171
1.153
1.135
Cl
O
N
Cl
OH
(3n)
EtO2C
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.86
1.05
1.02
1.00
1.01
0.99
2.08
0.97
1.03
2.17
3.00


166.63
154.76
148.62
138.19
133.60
131.43
127.00
125.42
122.93
122.67
122.12
118.07
109.43
109.15
93.62
91.48
59.19
39.94
39.73
39.52
39.31
39.10
14.41
Cl
O
N
Cl
OH
(3n)
EtO2C
200
180
160
140
120
100
80
60
40
20
0
ppm

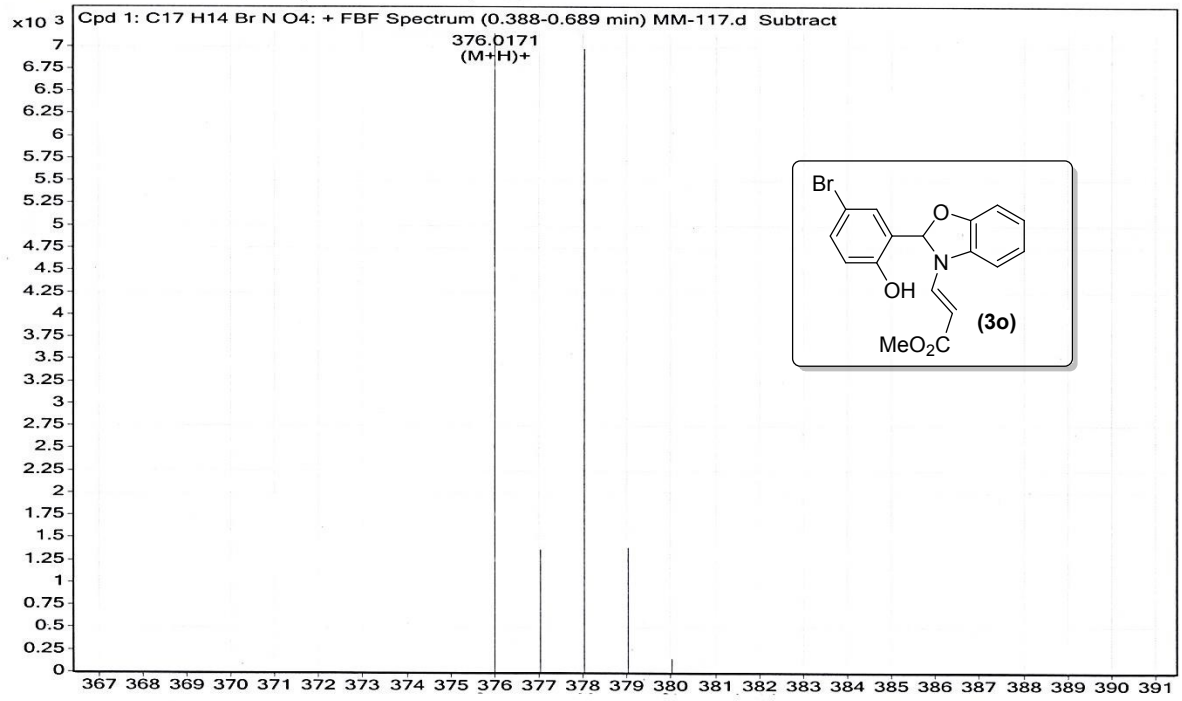
Sample Name	MM-169	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-169.d	**ACQ Method**	Pondicherry Universi				

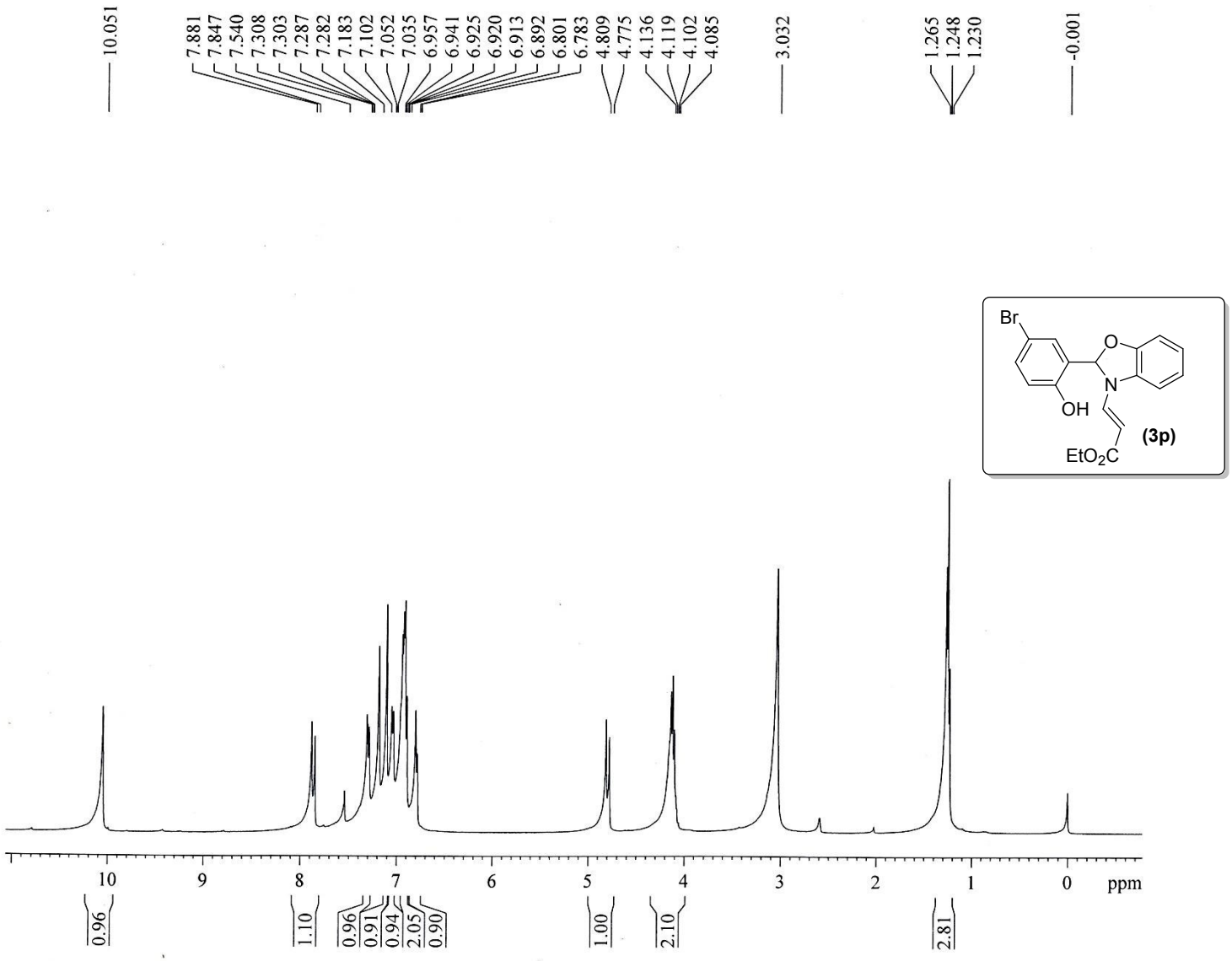


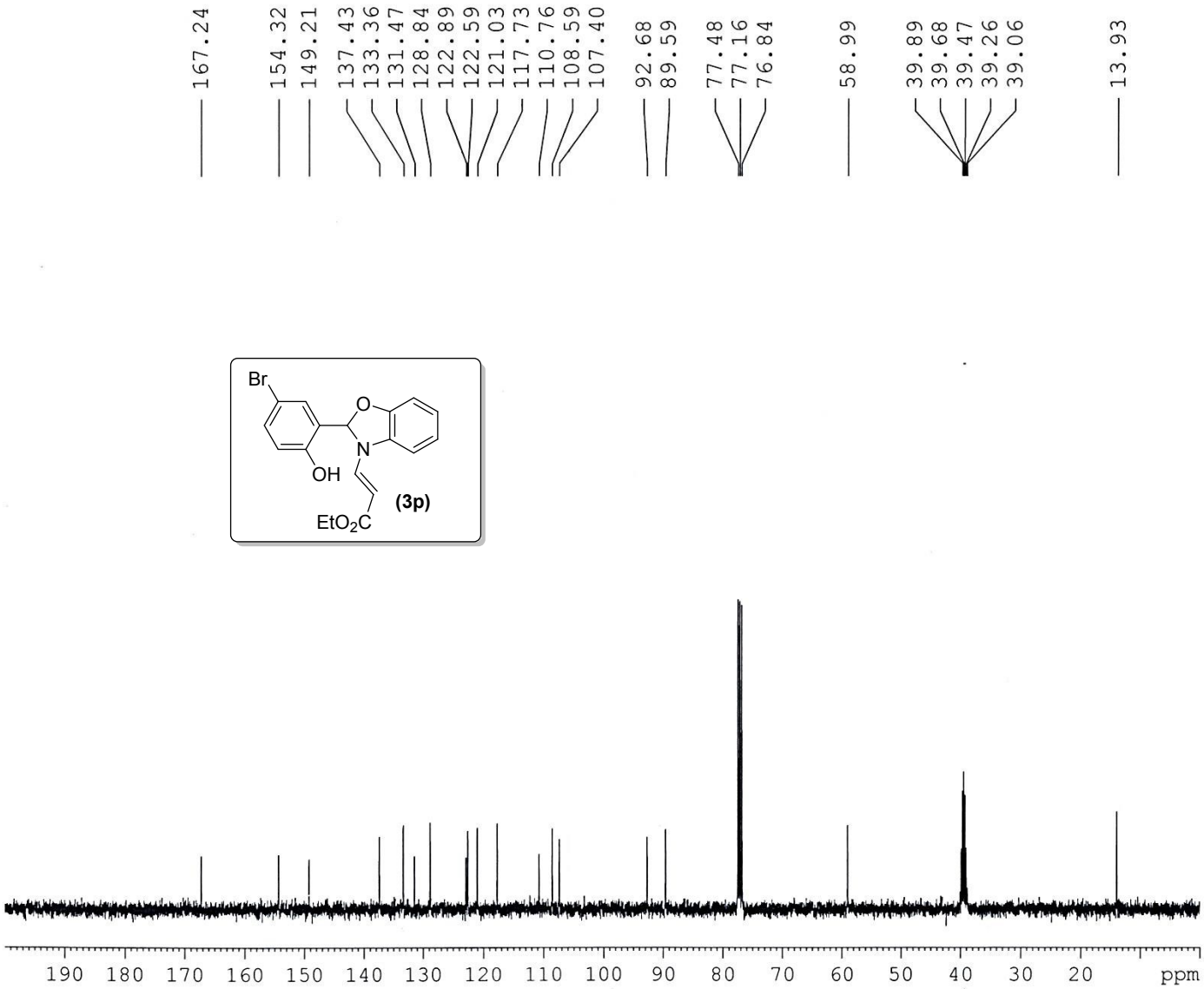

PROTON DMSO {D:\MB} KOPAL 1
10.207
7.882
7.847
7.717
7.680
7.313
7.307
7.291
7.285
7.170
7.165
7.105
7.092
7.078
7.064
6.948
6.934
6.930
6.920
6.915
6.898
6.893
6.805
6.788
6.784
4.806
4.772
3.641
-0.000
Br
O
N
OH
(3o)
MeO2C
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.02
1.15
1.17
1.06
0.99
1.25
2.43
1.17
1.03
3.03


168.12
154.58
149.64
137.90
133.75
131.79
129.28
123.14
122.99
121.36
118.03
111.27
109.02
107.71
92.66
89.96
77.48
77.16
76.84
50.75
40.25
40.04
39.83
39.62
39.43
Br
O
N
OH
(3o)
MeO2C
200
180
160
140
120
100
80
60
40
20
0
ppm

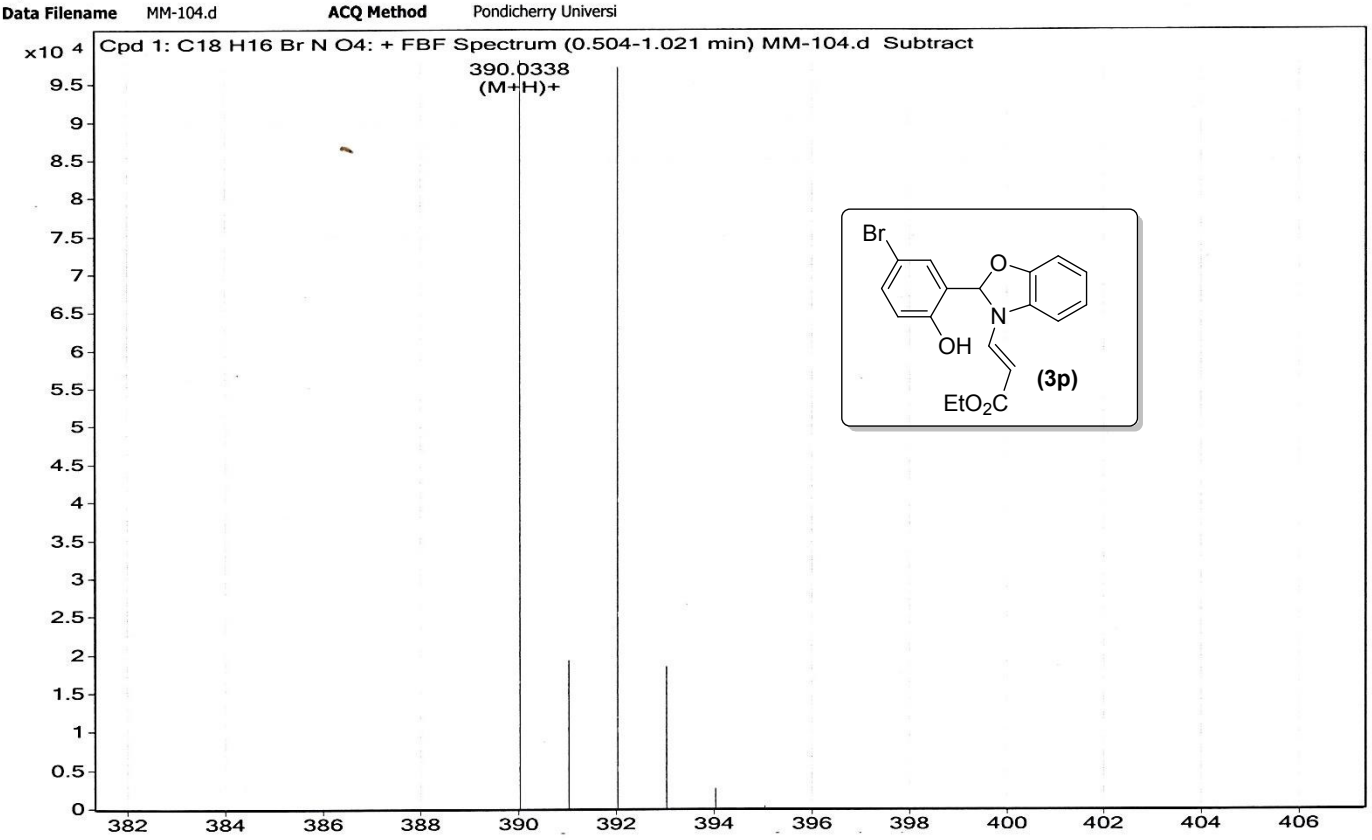
Sample Name	MM-117	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-117.d	**ACQ Method**	Pondicherry Universi				

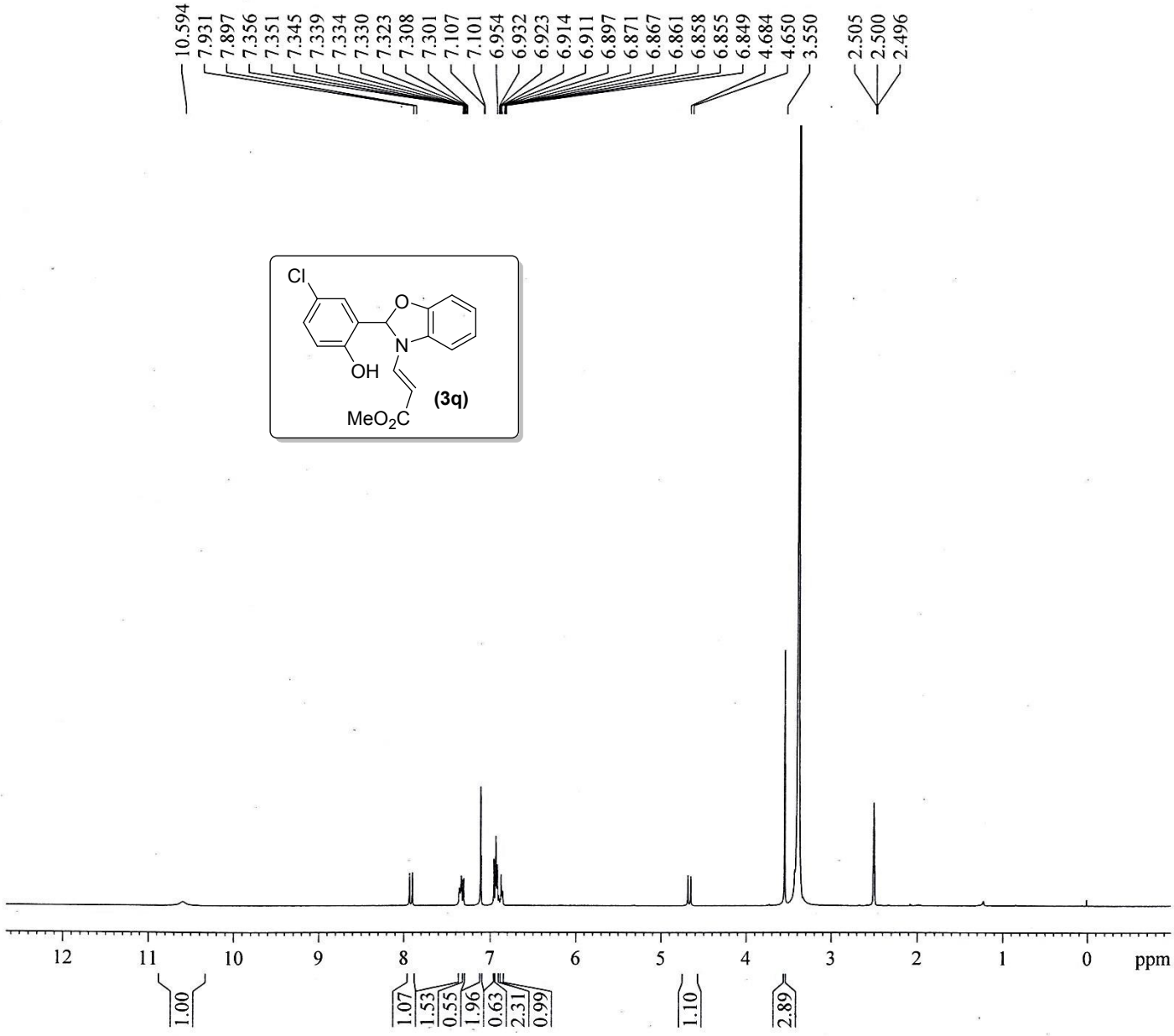


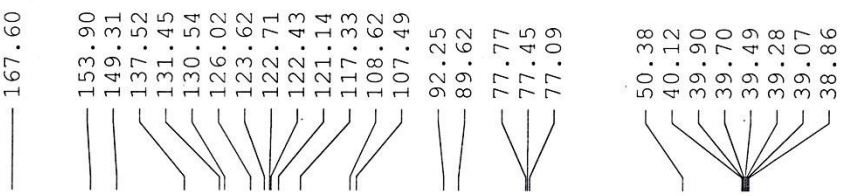

10.051
7.881
7.847
7.540
7.308
7.303
7.287
7.282
7.183
7.102
7.052
7.035
6.957
6.941
6.925
6.920
6.913
6.892
6.801
6.783
4.809
4.775
4.136
4.119
4.102
4.085
3.032
1.265
1.248
1.230
-0.001
Br
O
N
OH
(3p)
EtO2C
10
9
8
7
6
5
4
3
2
1
0
ppm
0.96
1.10
0.96
0.91
0.94
2.05
0.90
1.00
2.10
2.81

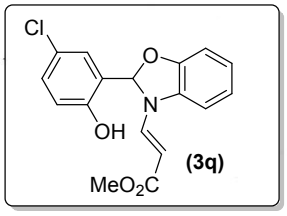

167.24
154.32
149.21
137.43
133.36
131.47
128.84
122.89
122.59
121.03
117.73
110.76
108.59
107.40
92.68
89.59
77.48
77.16
76.84
58.99
39.89
39.68
39.47
39.26
39.06
13.93
Br
O
N
OH
EtO2C
(3p)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

Sample Name	MM-104	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-104.d	**ACQ Method**	Pondicherry Universi				

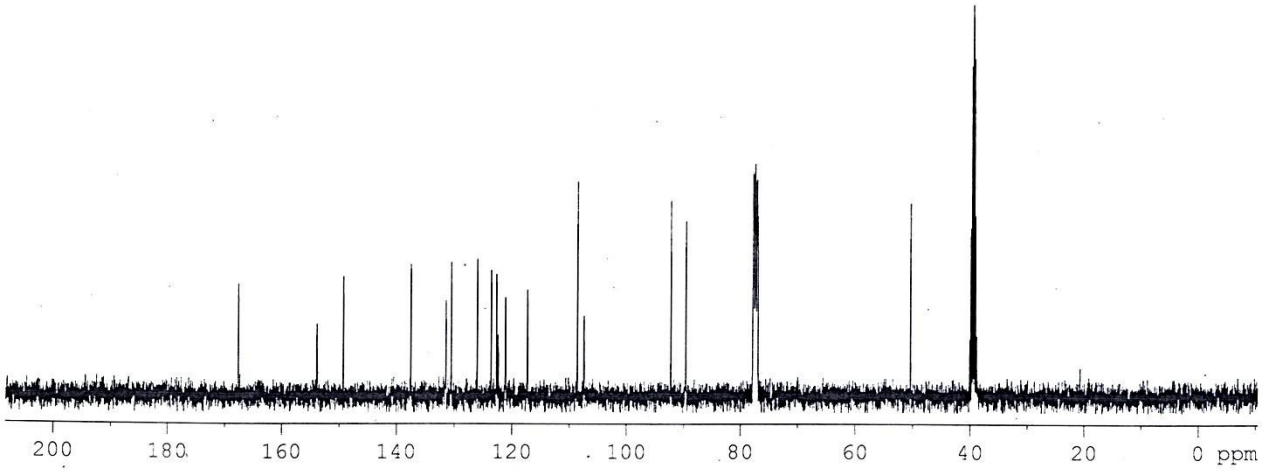



10.594
7.931
7.897
7.356
7.351
7.345
7.339
7.334
7.330
7.323
7.308
7.301
7.107
7.101
6.954
6.932
6.923
6.914
6.911
6.897
6.871
6.867
6.861
6.858
6.855
6.849
4.684
4.650
3.550
2.505
2.500
2.496
Cl
O
N
OH
(3q)
MeO2C
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.07
1.53
0.55
1.96
0.63
2.31
0.99
1.10
2.89

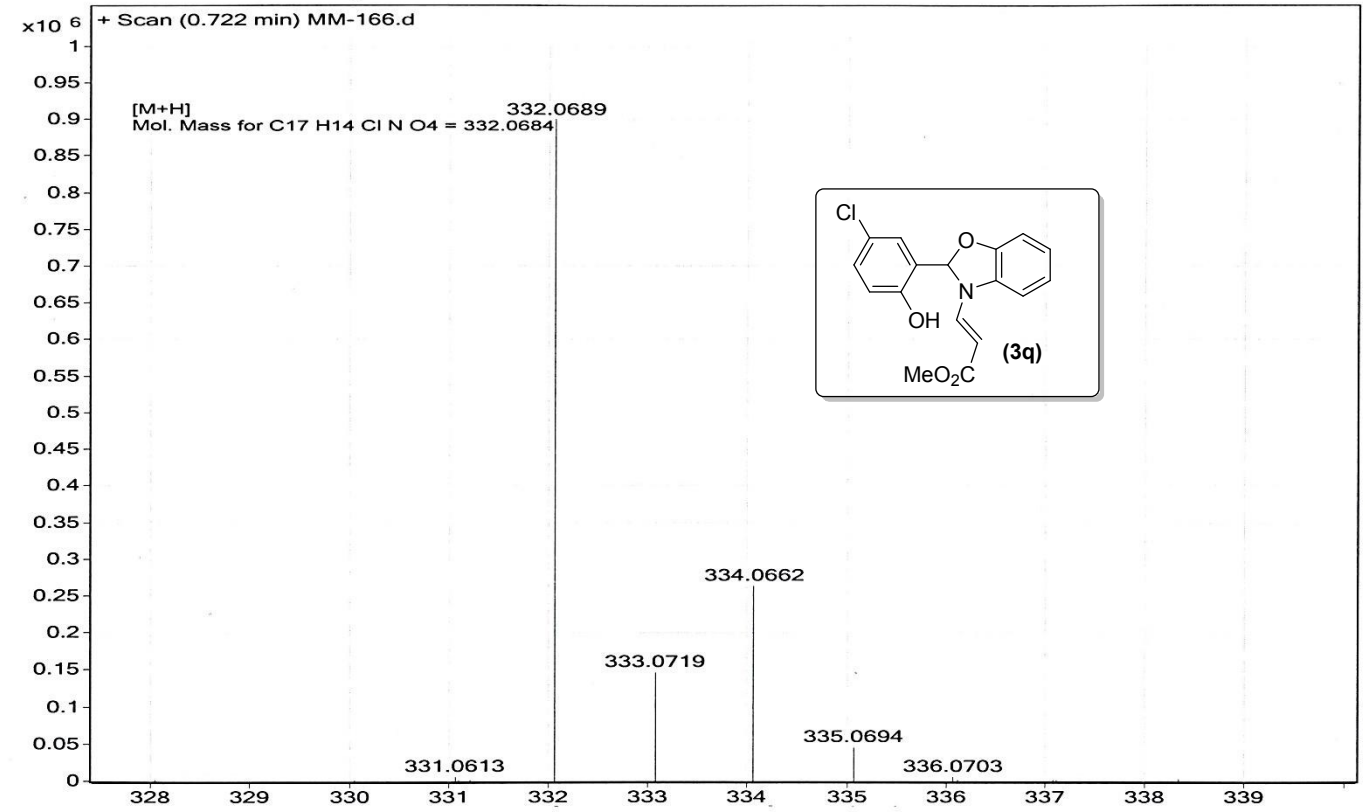

167.60
153.90
149.31
137.52
131.45
130.54
126.02
123.62
122.71
122.43
121.14
117.33
108.62
107.49
92.25
89.62
77.77
77.45
77.09
50.38
40.12
39.90
39.70
39.49
39.28
39.07
38.86


Cl
O
N
OH
MeO2C

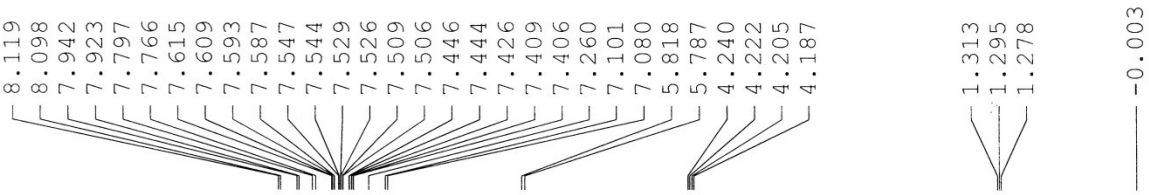
(3q)

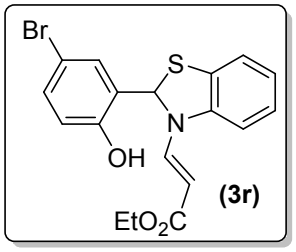

200
180
160
140
120
100
80
60
40
20
0 ppm

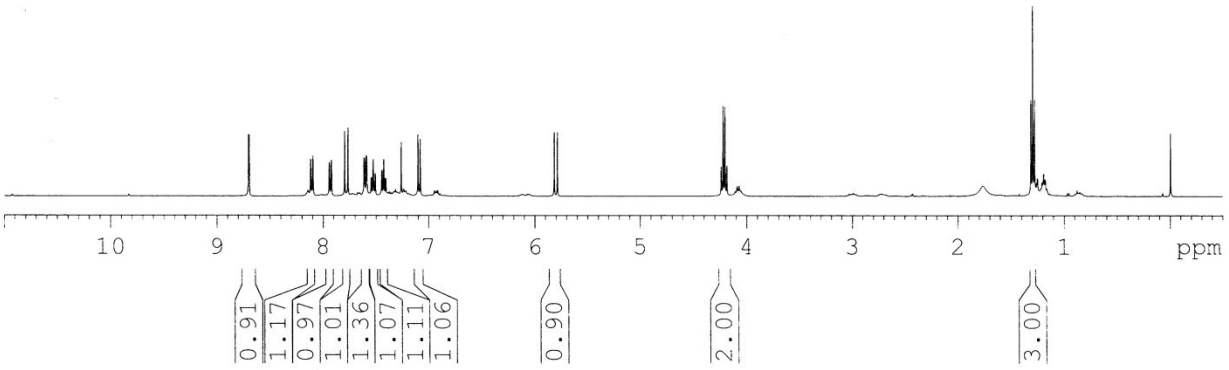
Sample Name	MM-166	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-166.d	**ACQ Method**	Pondicherry Universi				

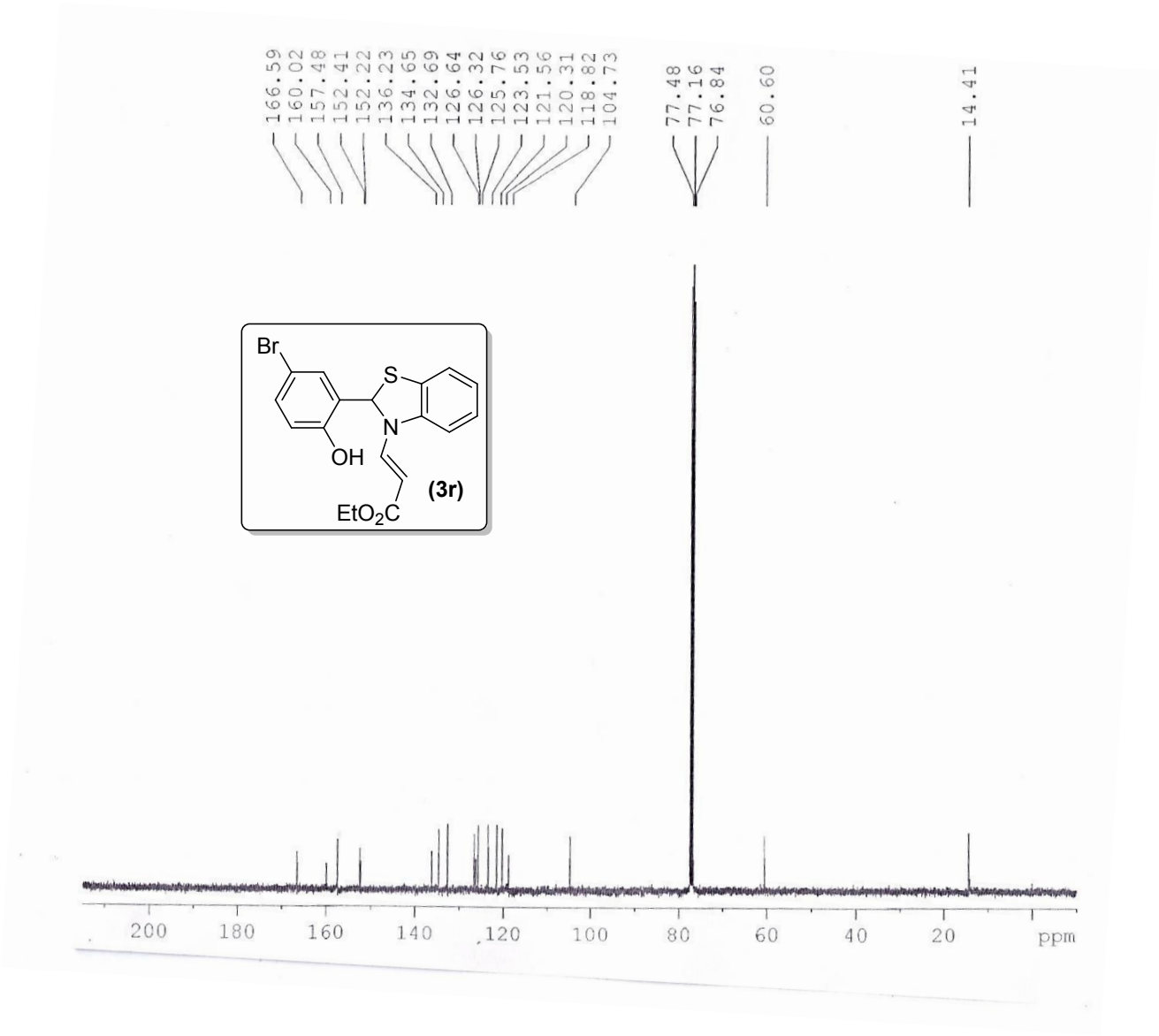


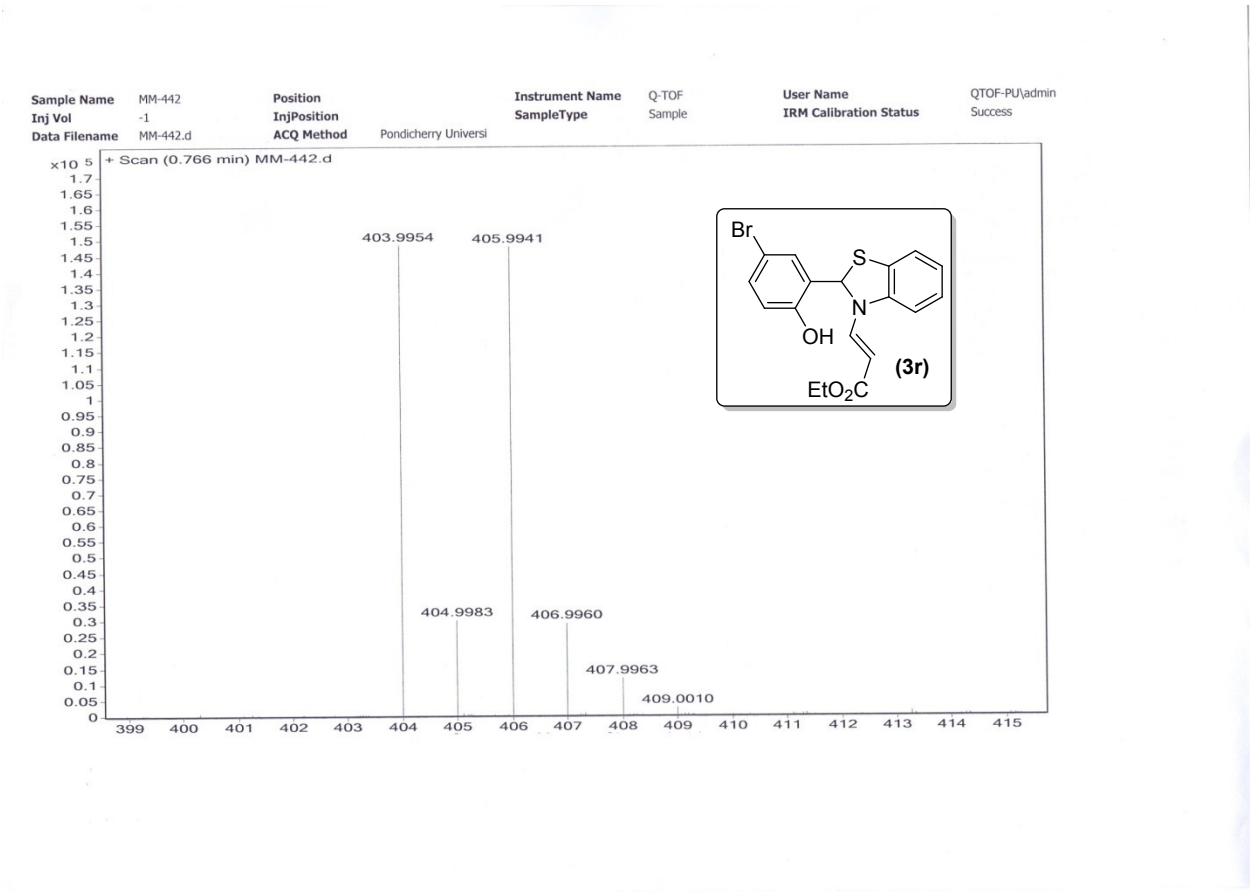
PROTON CDCl3 {D:\MB} KOPAL 1



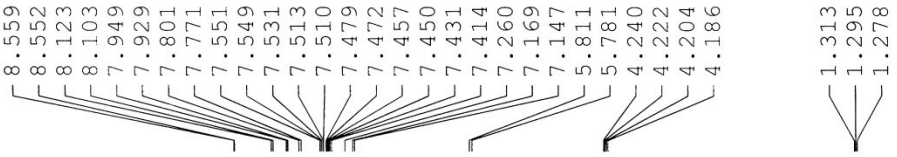


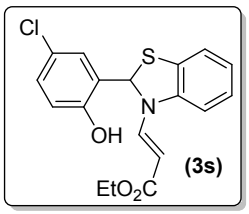


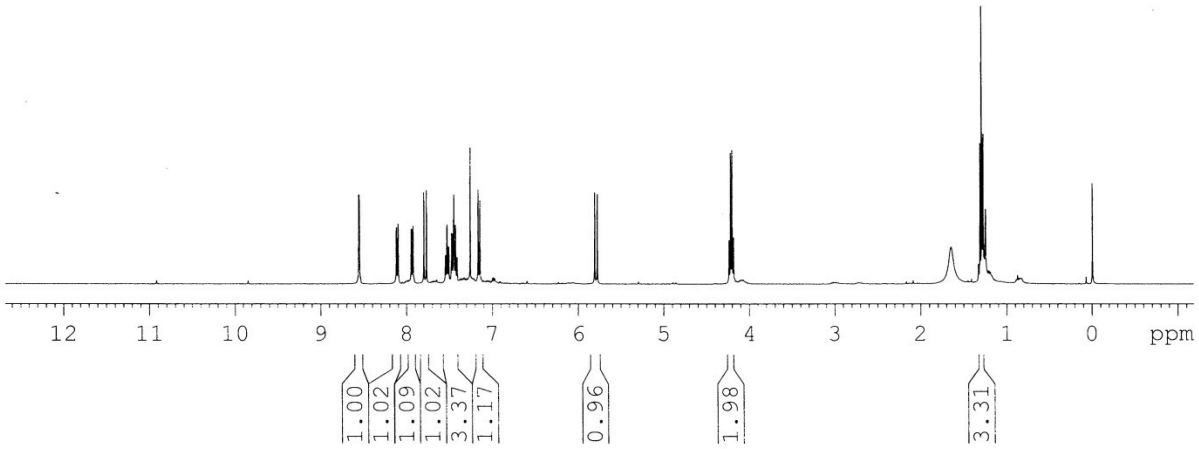

166.59
160.02
157.48
152.41
152.22
136.23
134.65
132.69
126.64
126.32
125.76
123.53
121.56
120.31
118.82
104.73
77.48
77.16
76.84
60.60
14.41
Br
S
N
OH
(3r)
EtO2C
200
180
160
140
120
100
80
60
40
20
ppm

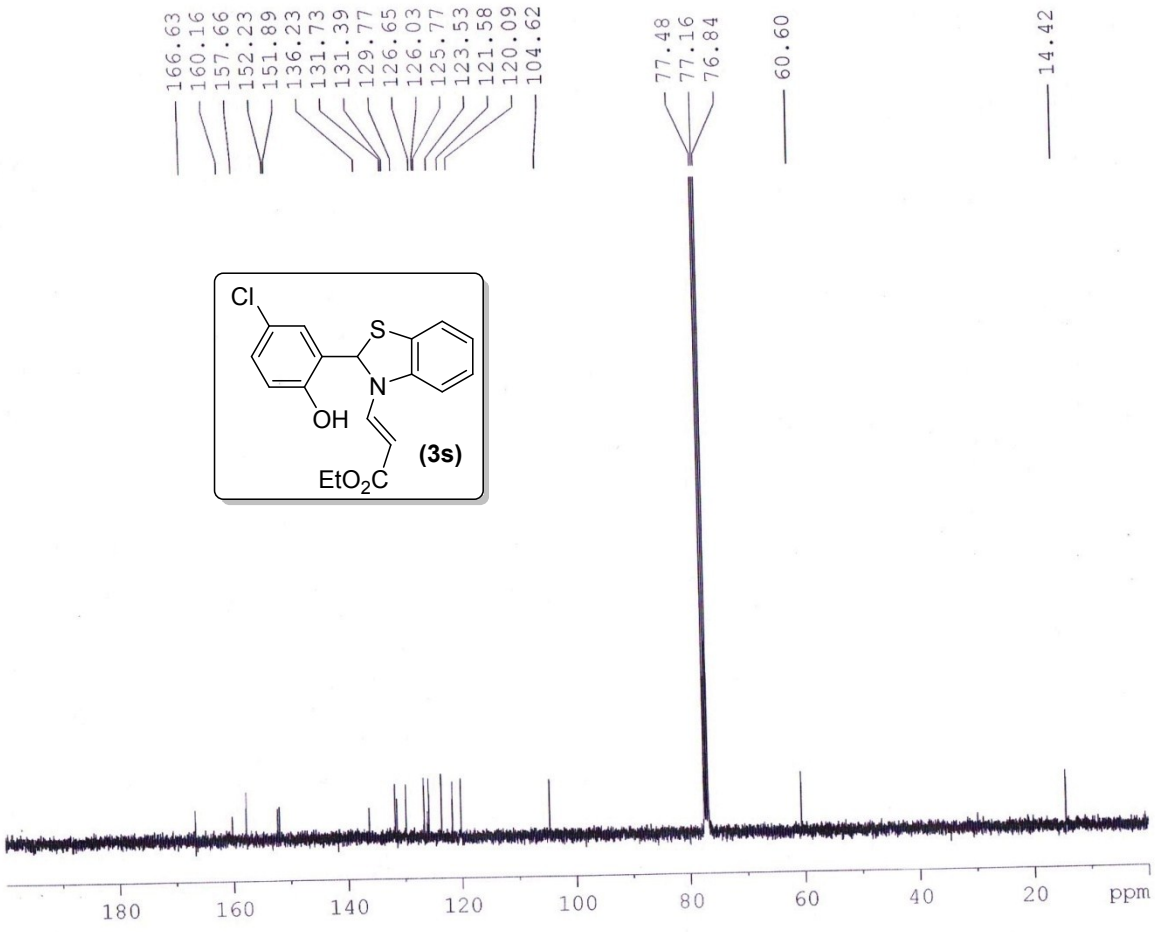

Sample Name MM-442
Inj Vol -1
Data Filename MM-442.d
Position
InjPosition
ACQ Method Pondicherry Universi
Instrument Name Q-TOF
SampleType Sample
User Name QTOF-PU\admin
IRM Calibration Status Success
+ Scan (0.766 min) MM-442.d
x10 5
403.9954
405.9941
404.9983
406.9960
407.9963
409.0010
Br
S
N
OH
EtO2C
(3r)

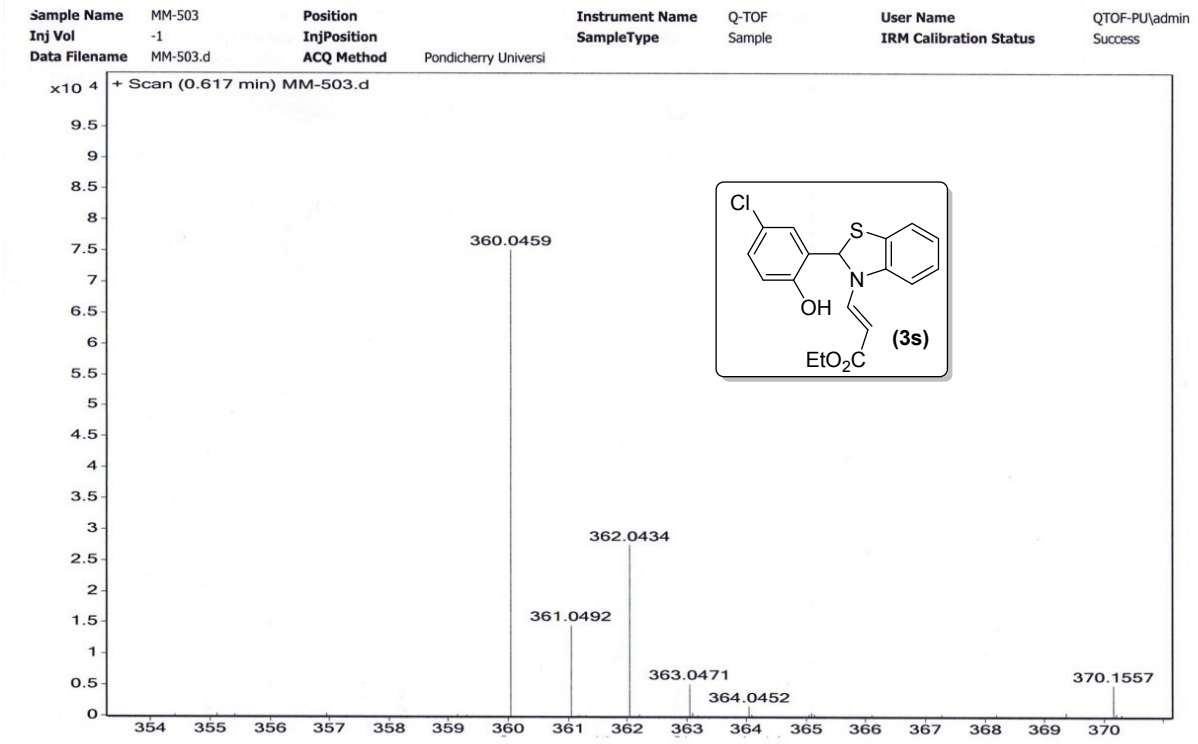
PROTON CDCl3 {D:\MB} KOPAL 1

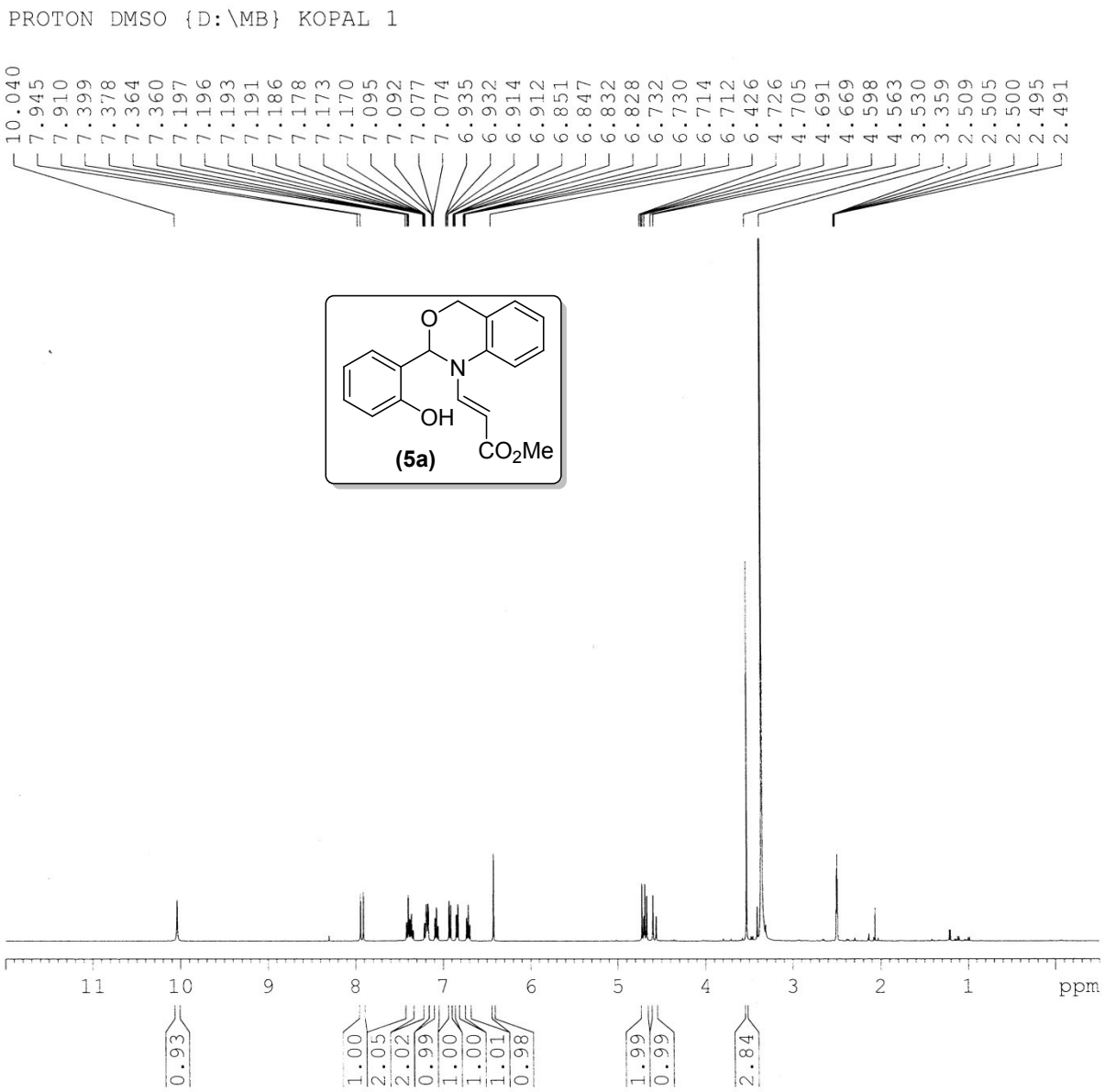


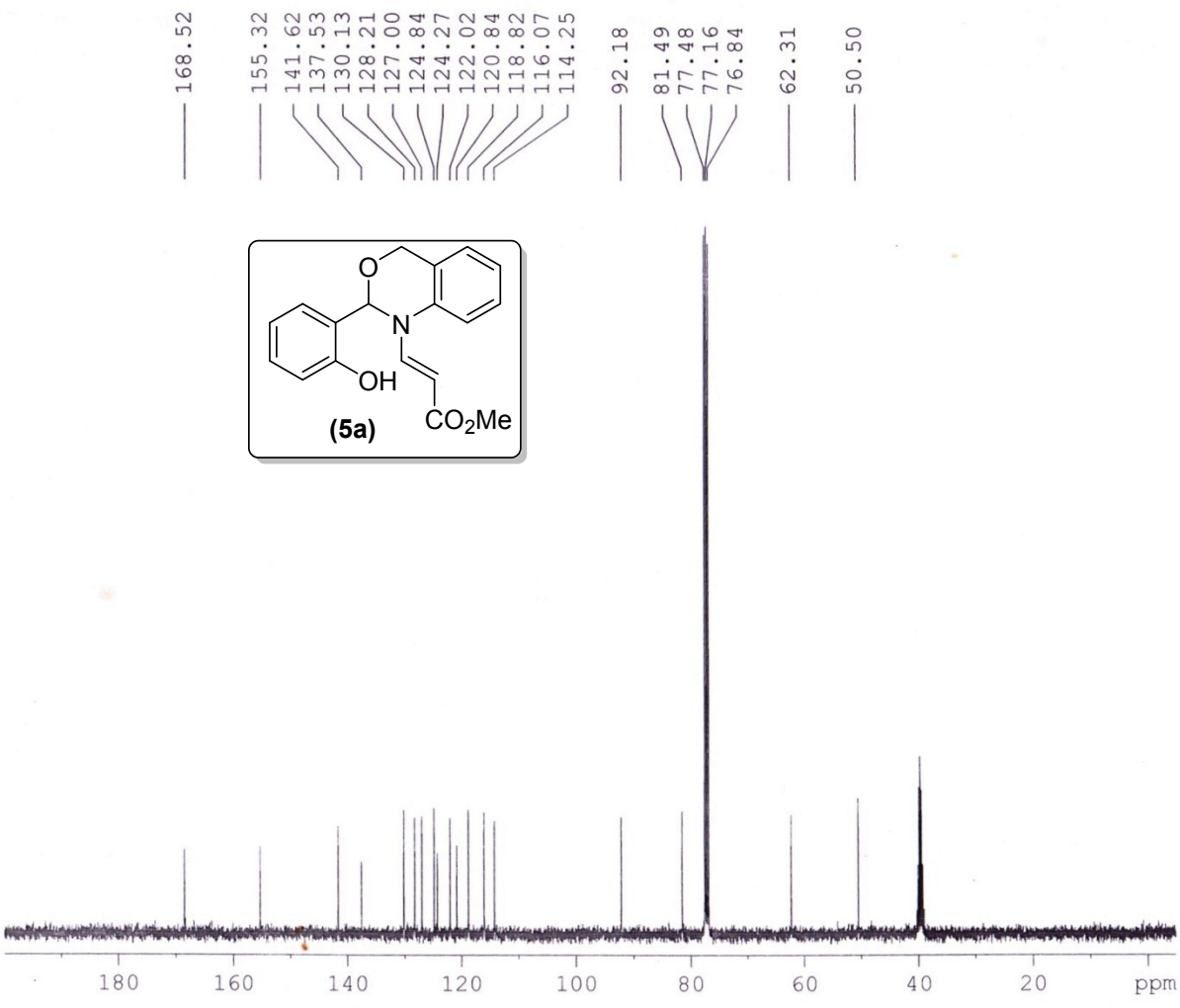




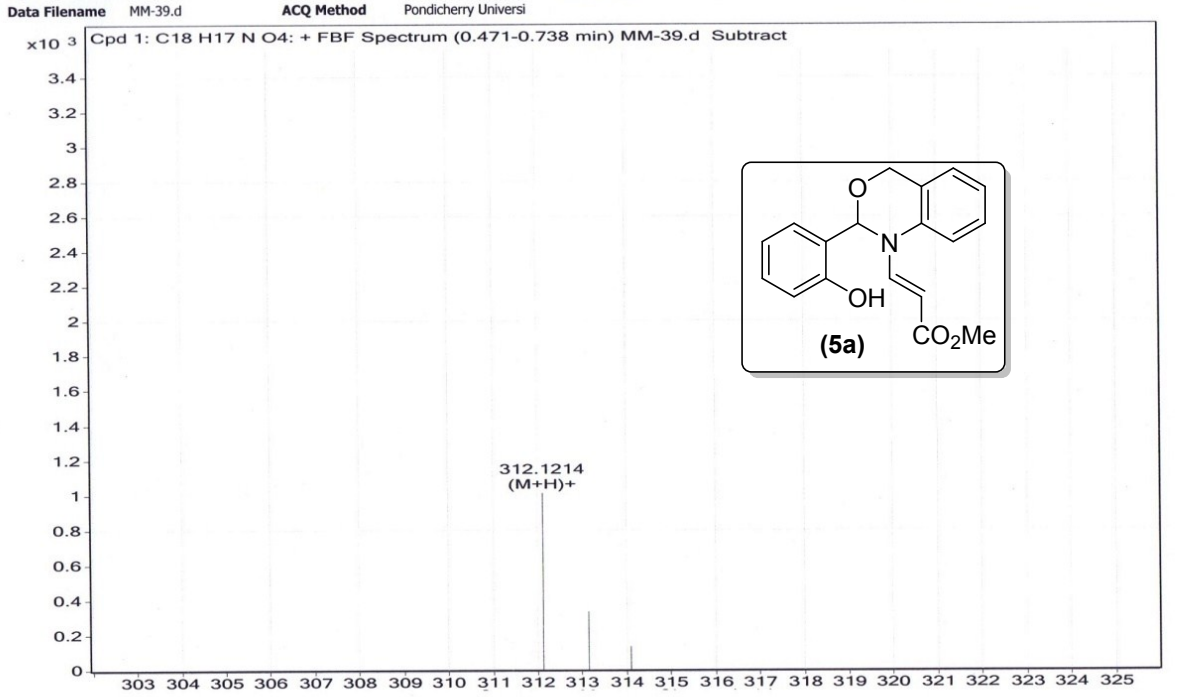

166.63
160.16
157.66
152.23
151.89
136.23
131.73
131.39
129.77
126.65
126.03
125.77
123.53
121.58
120.09
104.62
77.48
77.16
76.84
60.60
14.42
Cl
S
N
OH
EtO2C
(3s)
180
160
140
120
100
80
60
40
20
ppm


Sample Name MM-503
Position
Instrument Name Q-TOF
User Name QTOF-PU\admin
Inj Vol -1
InjPosition
SampleType Sample
IRM Calibration Status Success
Data Filename MM-503.d
ACQ Method Pondicherry Universi
+ Scan (0.617 min) MM-503.d
x10 4
360.0459
362.0434
361.0492
363.0471
364.0452
370.1557
Cl
S
N
OH
EtO2C
(3s)

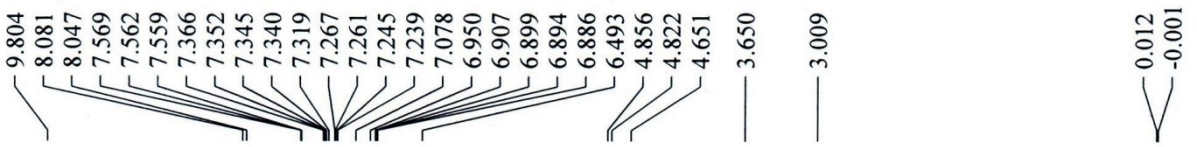

PROTON DMSO {D:\MB} KOPAL 1
10.040
7.945
7.910
7.399
7.378
7.364
7.360
7.197
7.196
7.193
7.191
7.186
7.178
7.173
7.170
7.095
7.092
7.077
7.074
6.935
6.932
6.914
6.912
6.851
6.847
6.832
6.828
6.732
6.730
6.714
6.712
6.426
4.726
4.705
4.691
4.669
4.598
4.563
3.530
3.359
2.509
2.505
2.500
2.495
2.491
O
N
OH
CO2Me
(5a)
11
10
9
8
7
6
5
4
3
2
1
ppm
0.93
1.00
2.05
2.02
0.99
1.00
1.00
1.01
0.98
1.99
0.99
2.84

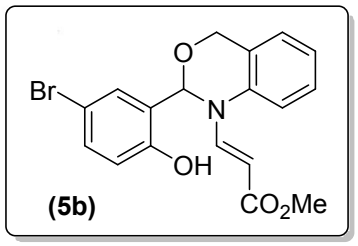

168.52
155.32
141.62
137.53
130.13
128.21
127.00
124.84
124.27
122.02
120.84
118.82
116.07
114.25
92.18
81.49
77.48
77.16
76.84
62.31
50.50
O
N
OH
CO2Me
(5a)
180
160
140
120
100
80
60
40
20
ppm

Sample Name	MM-39	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-39.d	**ACQ Method**	Pondicherry Universi				

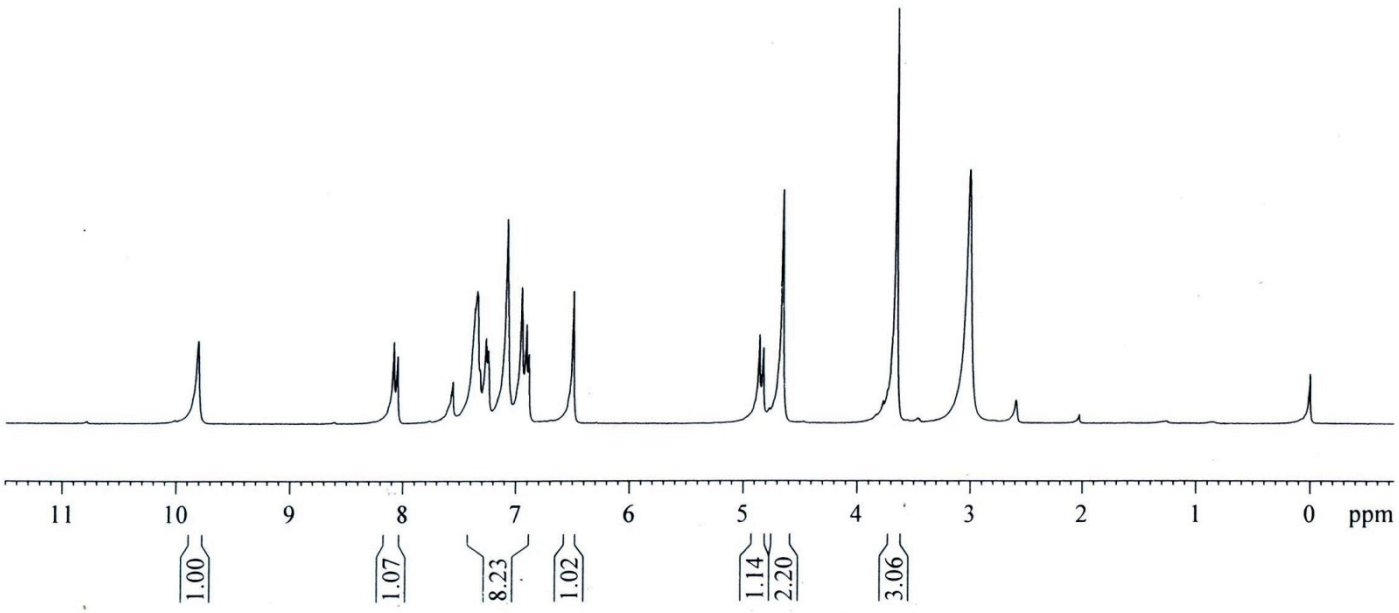


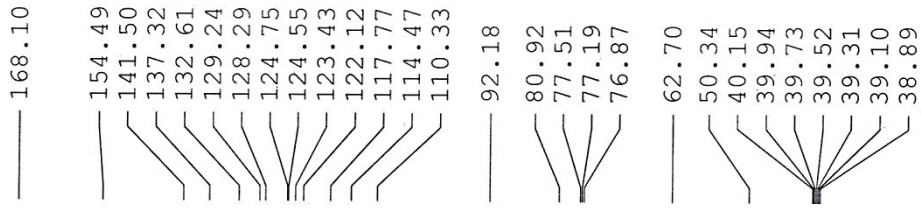
PROTON DMSO {D:\MB} KOPAL 1

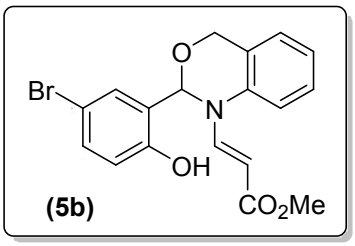


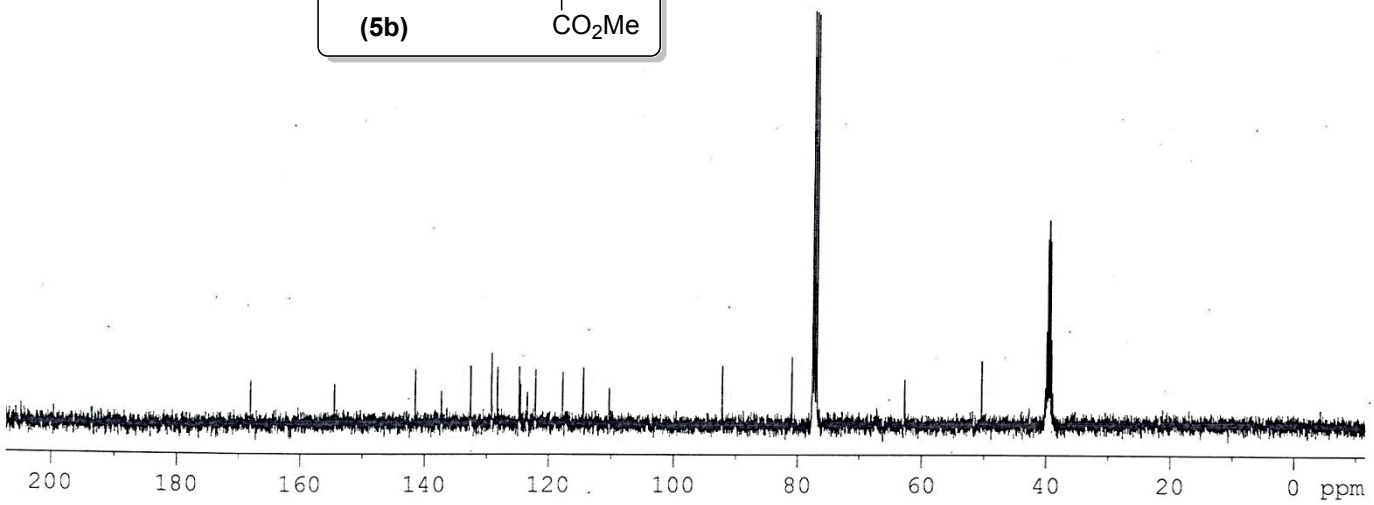


(5b)

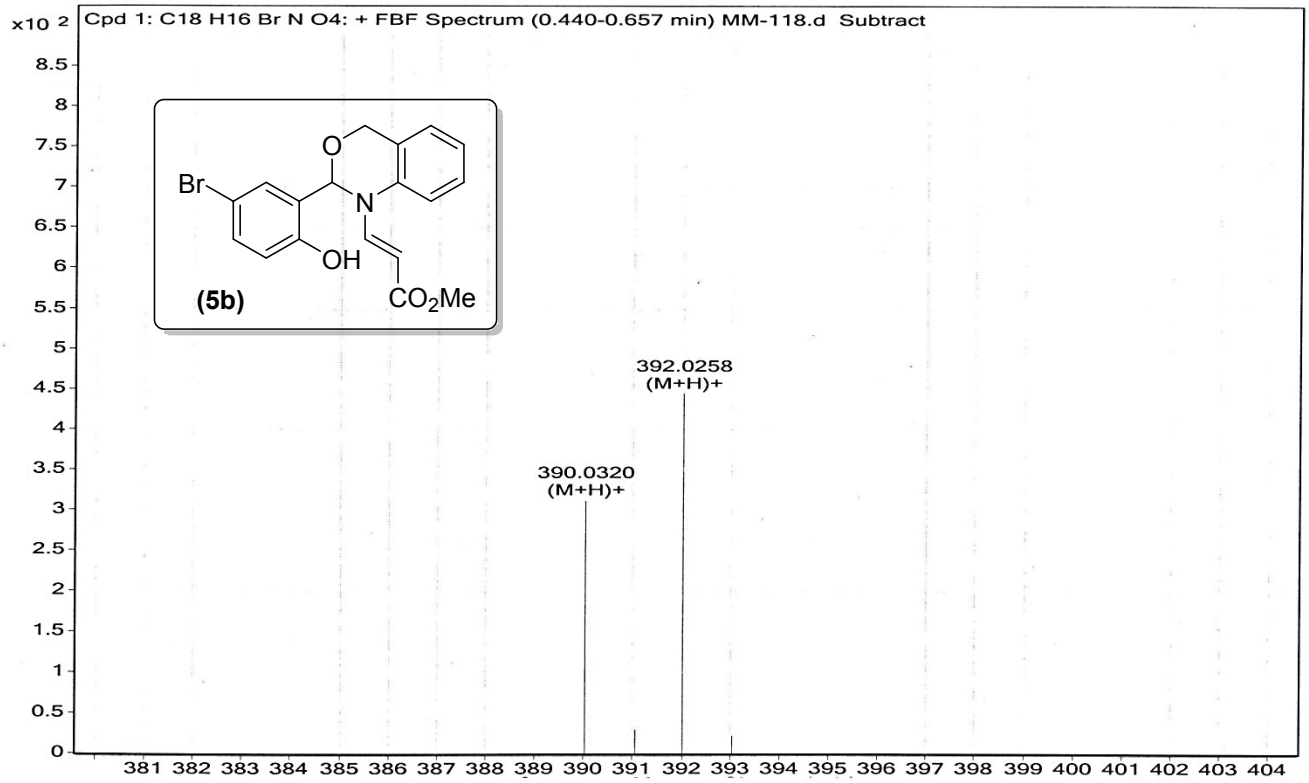



168.10
154.49
141.50
137.32
132.61
129.24
128.29
124.75
124.55
123.43
122.12
117.77
114.47
110.33
92.18
80.92
77.51
77.19
76.87
62.70
50.34
40.15
39.94
39.73
39.52
39.31
39.10
38.89

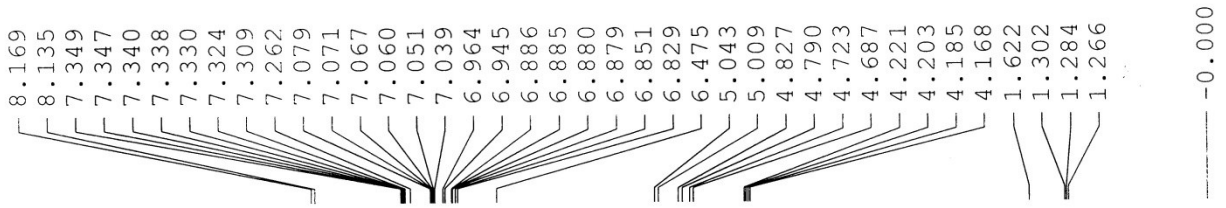

Br
O
N
OH
CO2Me
(5b)

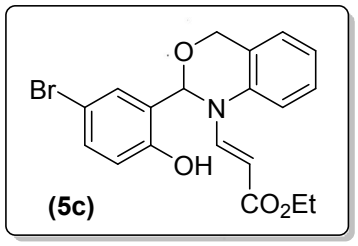

200
180
160
140
120
100
80
60
40
20
0 ppm

Sample Name	MM-118	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-118.d	**ACQ Method**	Pondicherry Universi				

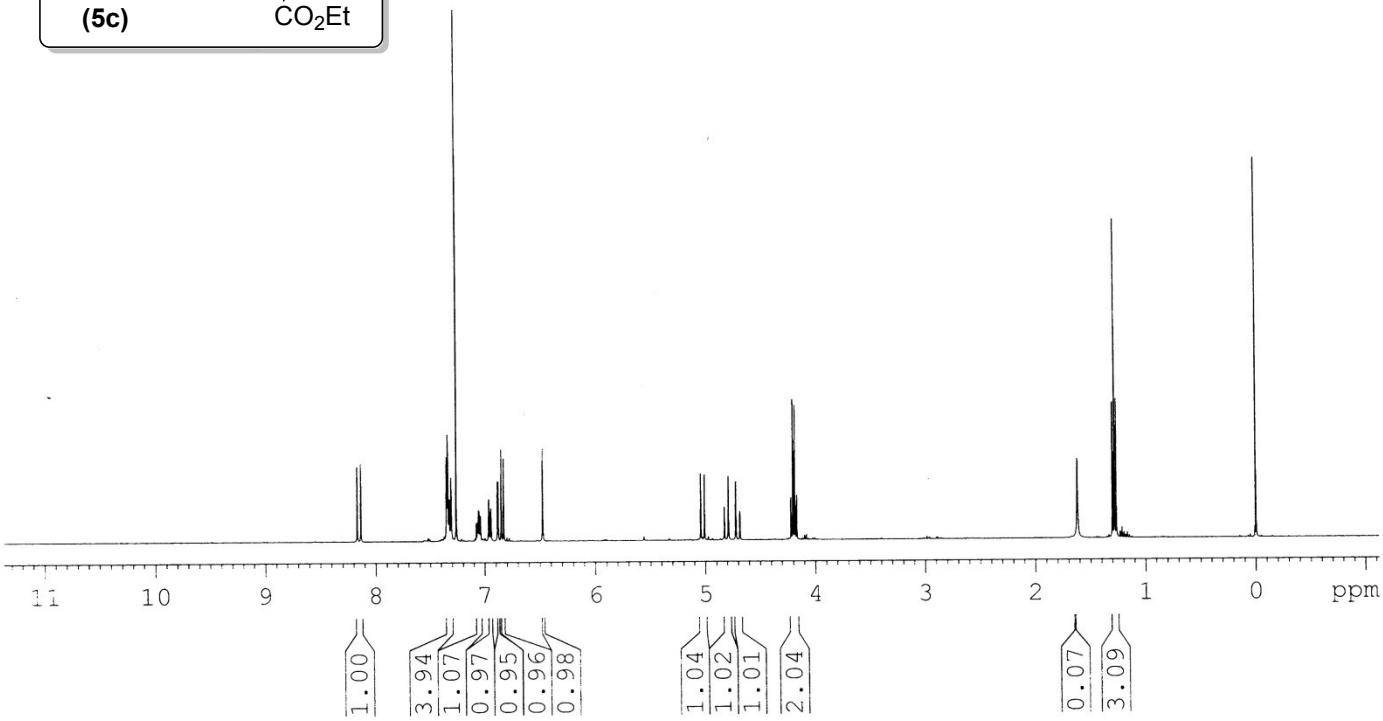


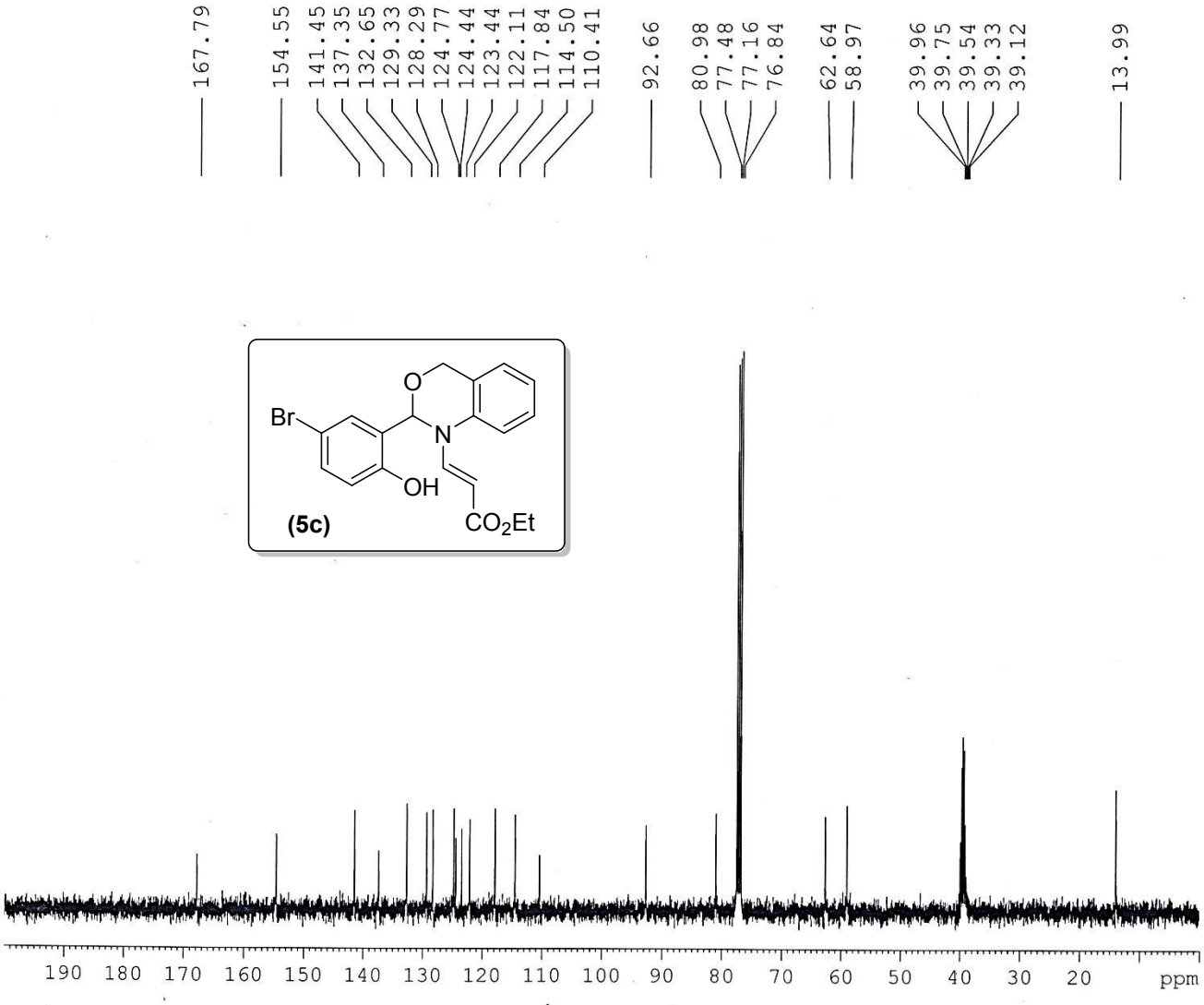
PROTON CDCl3 {D:\MB} KOPAL 1



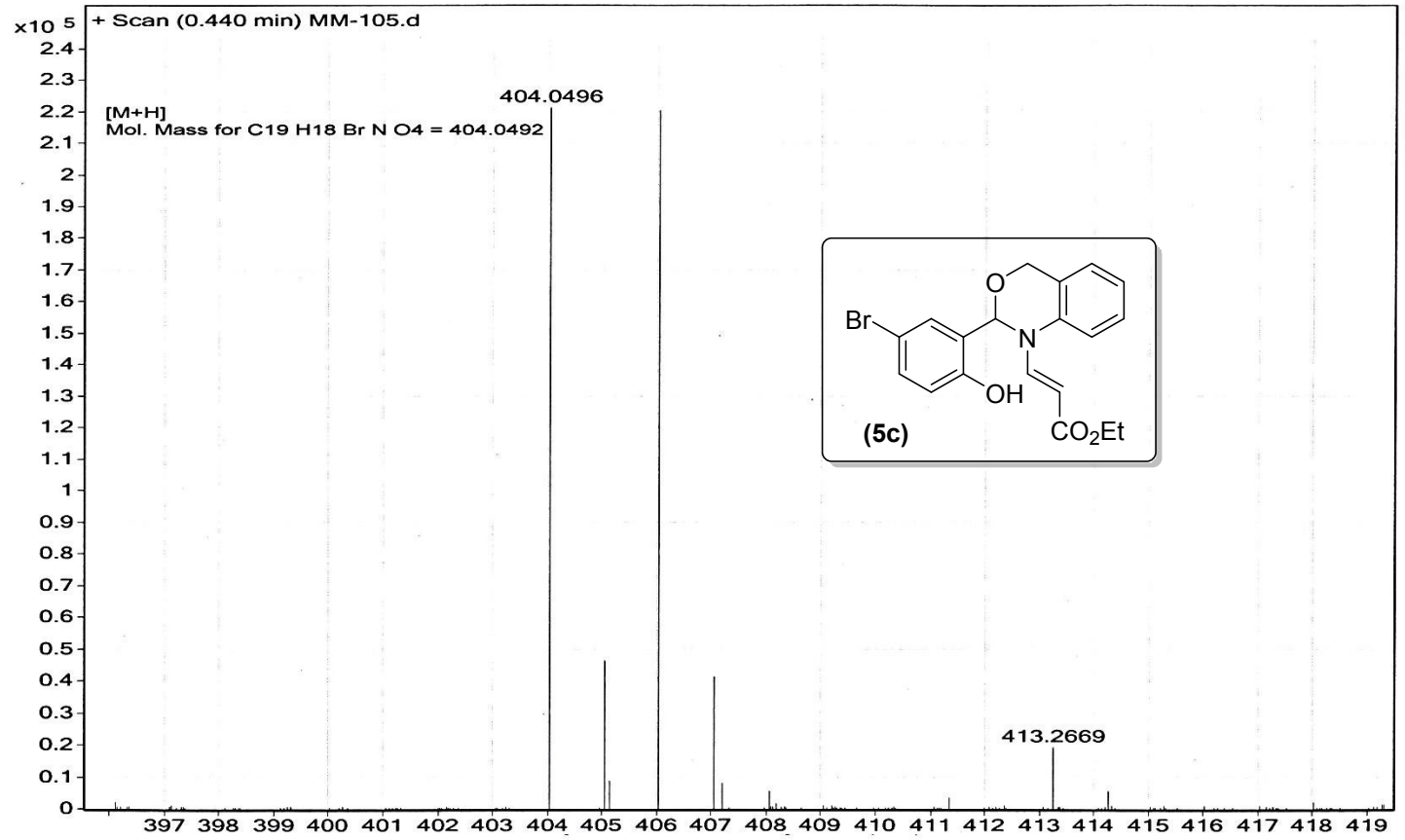


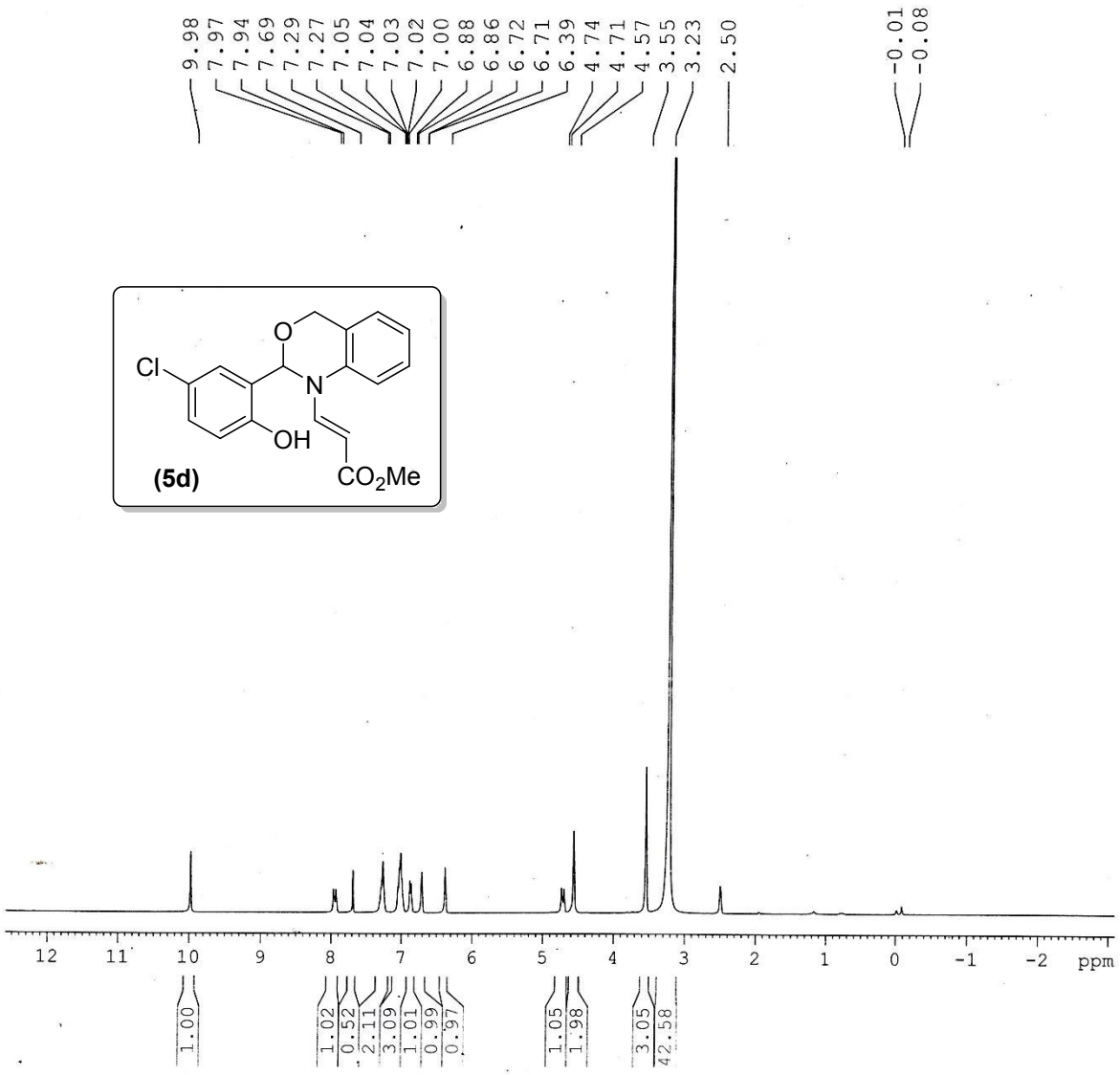
(5c)

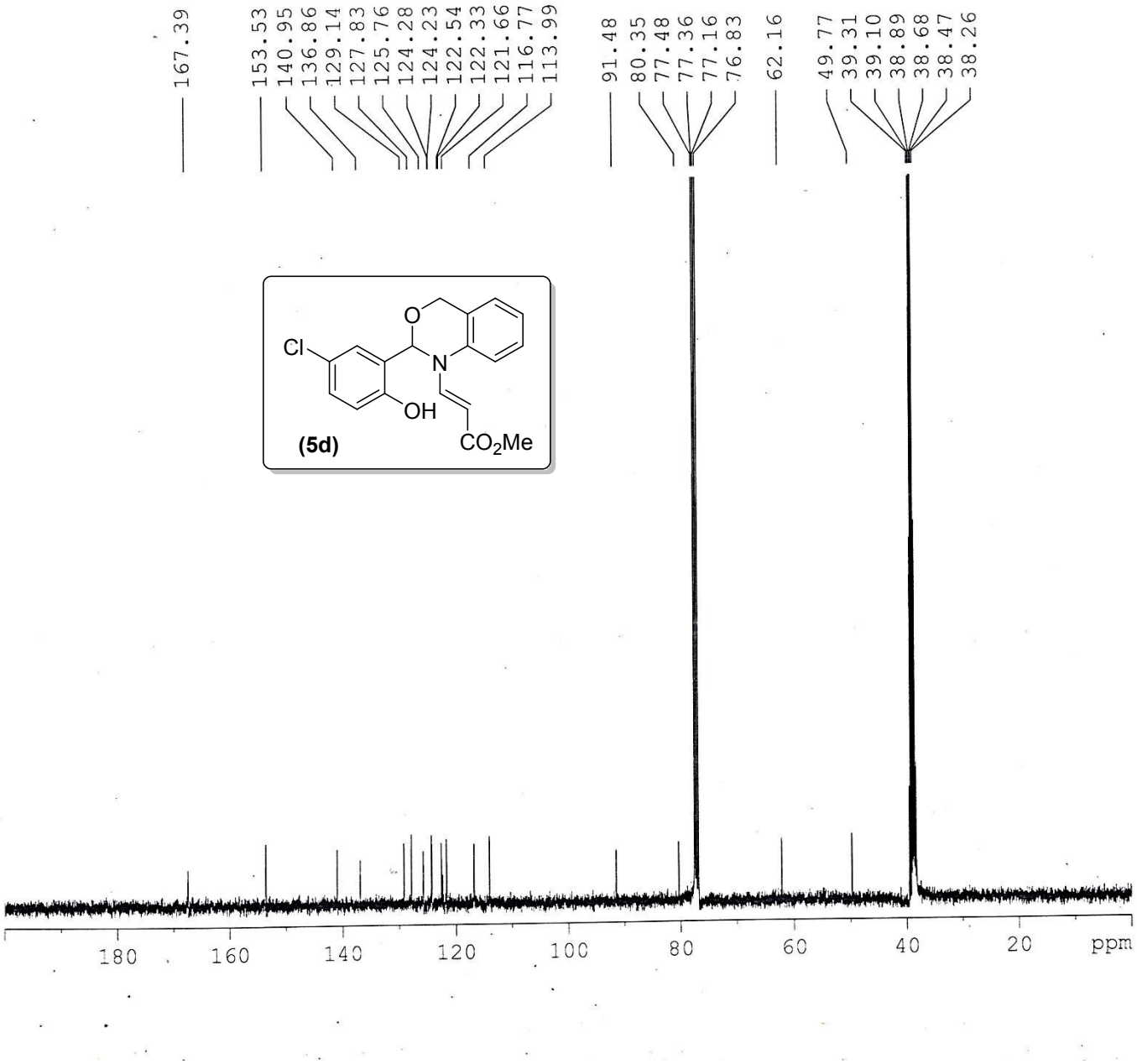



167.79
154.55
141.45
137.35
132.65
129.33
128.29
124.77
124.44
123.44
122.11
117.84
114.50
110.41
92.66
80.98
77.48
77.16
76.84
62.64
58.97
39.96
39.75
39.54
39.33
39.12
13.99
Br
O
N
OH
CO2Et
(5c)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

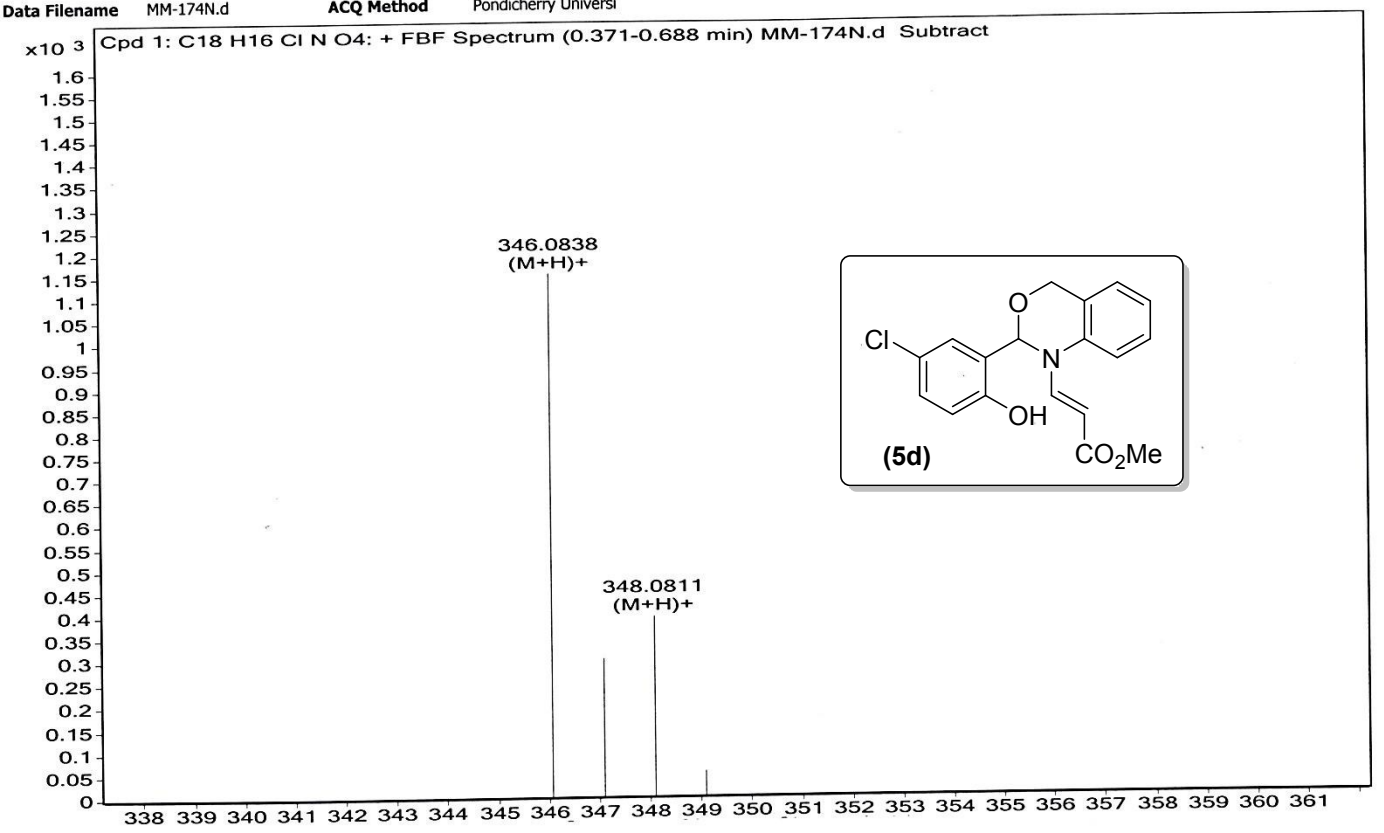
Sample Name	MM-105	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-105.d	**ACQ Method**	Pondicherry Universi			**Acquired Time**	

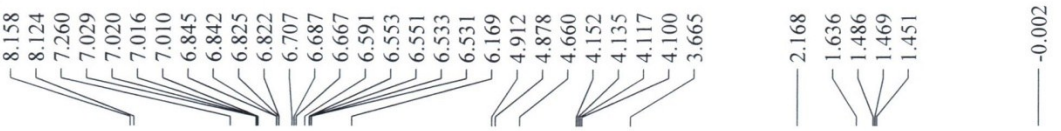


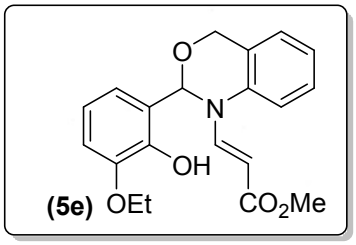

9.98
7.97
7.94
7.69
7.29
7.27
7.05
7.04
7.03
7.02
7.00
6.88
6.86
6.72
6.71
6.39
4.74
4.71
4.57
3.55
3.23
2.50
-0.01
-0.08
Cl
O
N
OH
CO2Me
(5d)
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
ppm
1.00
1.02
0.52
2.11
3.09
1.01
0.99
0.97
1.05
1.98
3.05
42.58

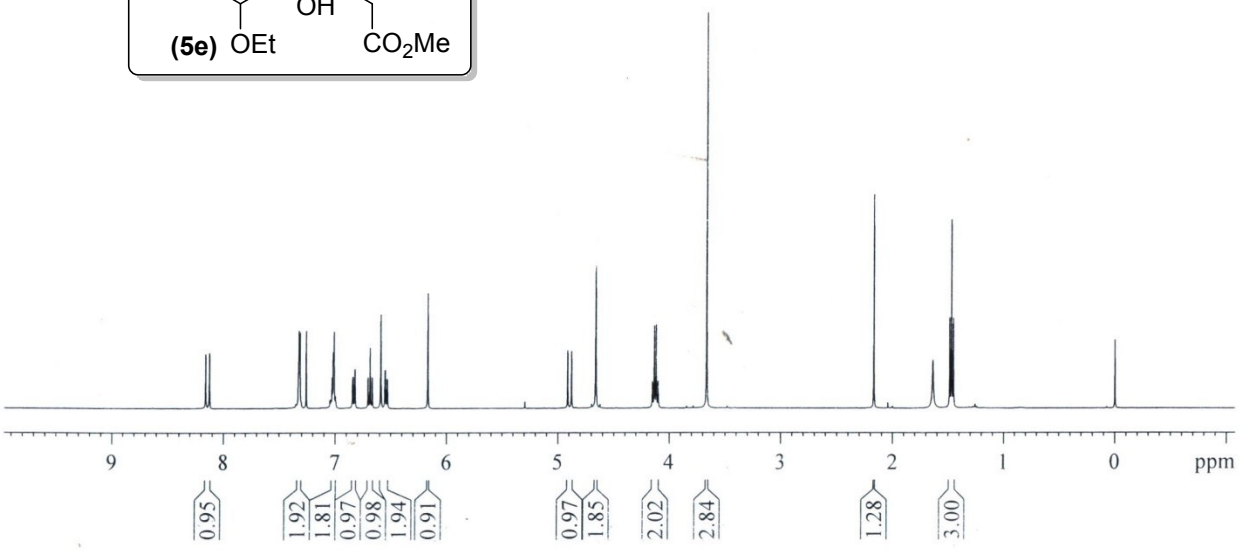

167.39
153.53
140.95
136.86
129.14
127.83
125.76
124.28
124.23
122.54
122.33
121.66
116.77
113.99
91.48
80.35
77.48
77.36
77.16
76.83
62.16
49.77
39.31
39.10
38.89
38.68
38.47
38.26
Cl
O
N
OH
CO2Me
(5d)
180
160
140
120
100
80
60
40
20
ppm

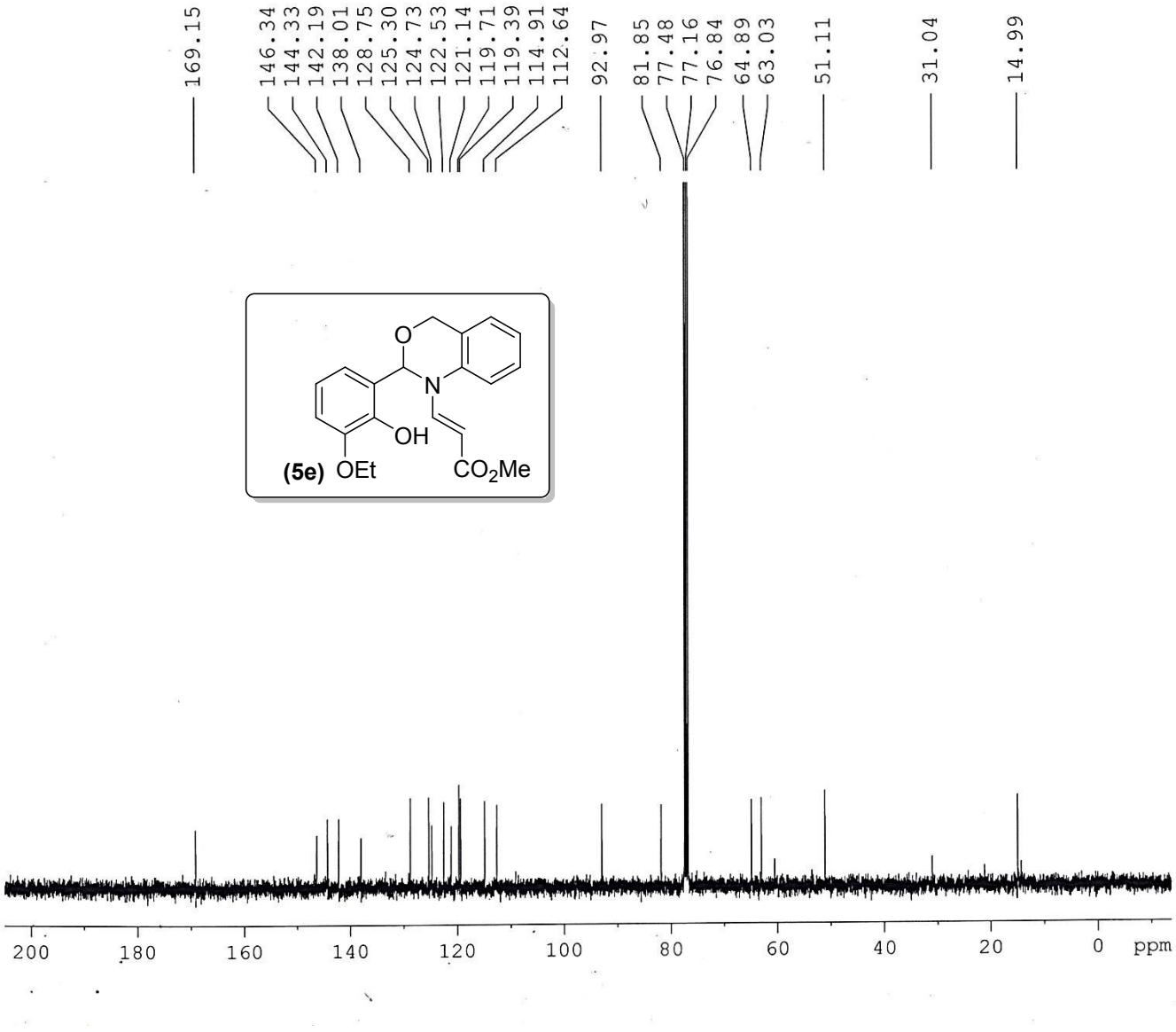
Sample Name	MM-174	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-174N.d	**ACQ Method**	Pondicherry Universi				



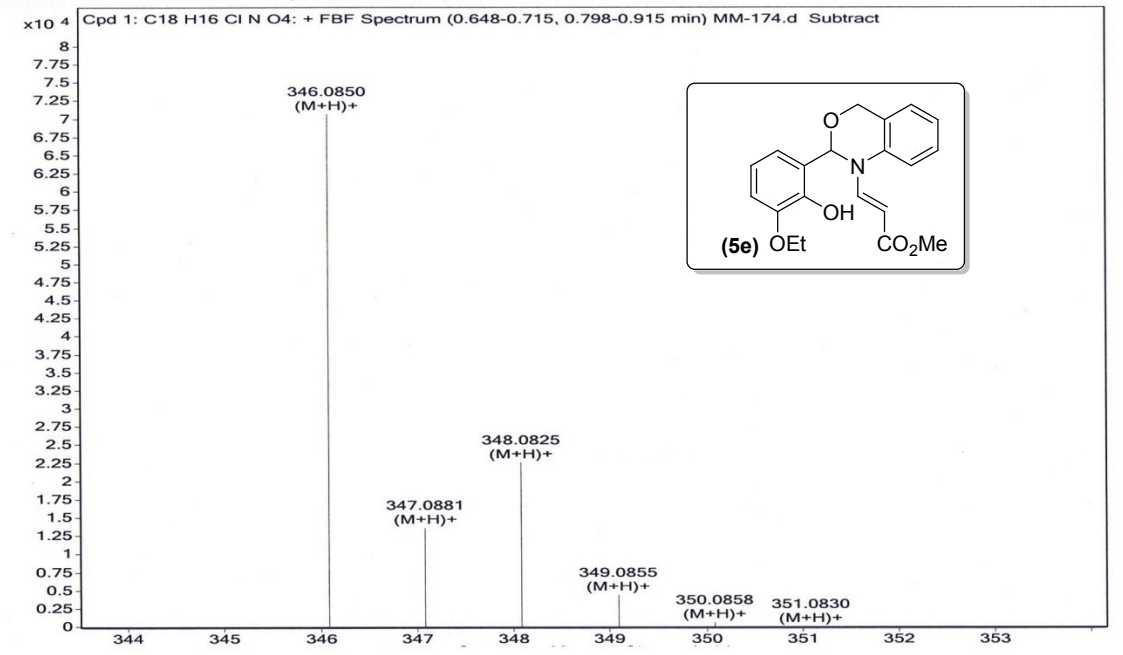

8.158
8.124
7.260
7.029
7.020
7.016
7.010
6.845
6.842
6.825
6.822
6.707
6.687
6.667
6.591
6.553
6.551
6.533
6.531
6.169
4.912
4.878
4.660
4.152
4.135
4.117
4.100
3.665
2.168
1.636
1.486
1.469
1.451
-0.002

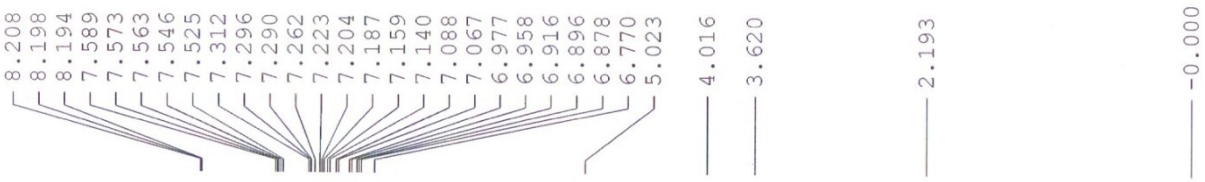

O
N
OH
(5e) OEt
CO2Me

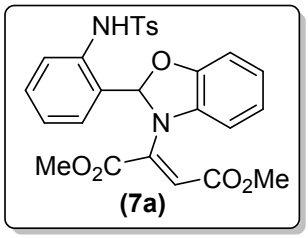

9
8
7
6
5
4
3
2
1
0
ppm
0.95
1.92
1.81
0.97
0.98
1.94
0.91
0.97
1.85
2.02
2.84
1.28
3.00


169.15
146.34
144.33
142.19
138.01
128.75
125.30
124.73
122.53
121.14
119.71
119.39
114.91
112.64
92.97
81.85
77.48
77.16
76.84
64.89
63.03
51.11
31.04
14.99
O
N
OH
(5e) OEt
CO2Me
200
180
160
140
120
100
80
60
40
20
0
ppm

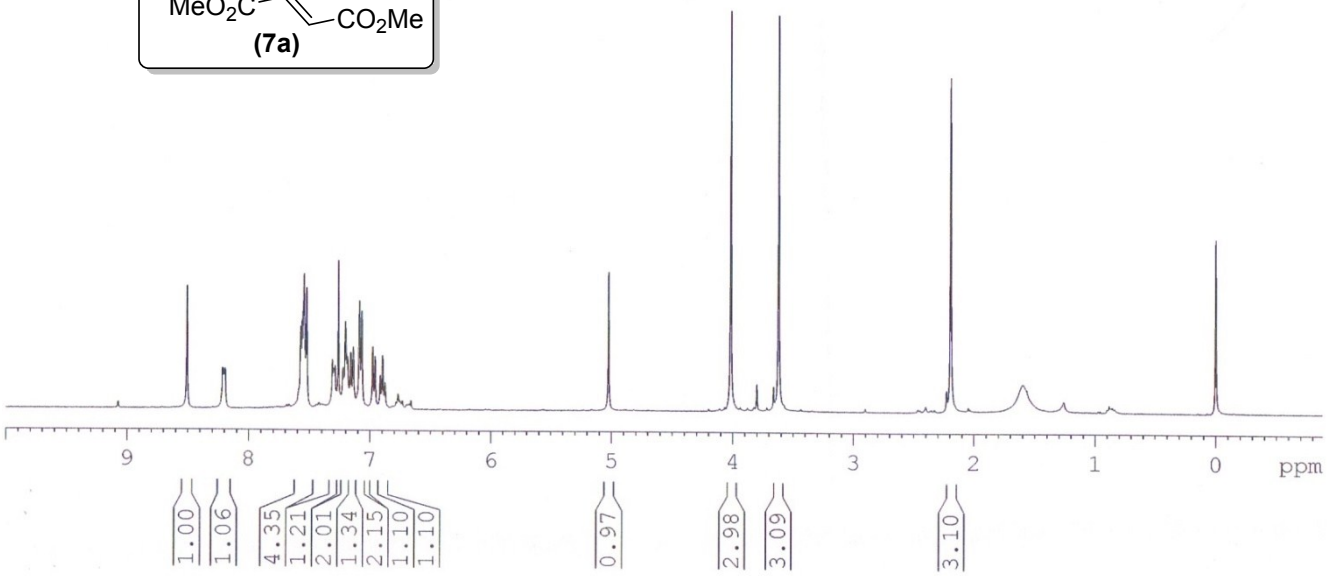
Sample Name	MM-174	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-174.d	**ACQ Method**	Pondicherry Universi				

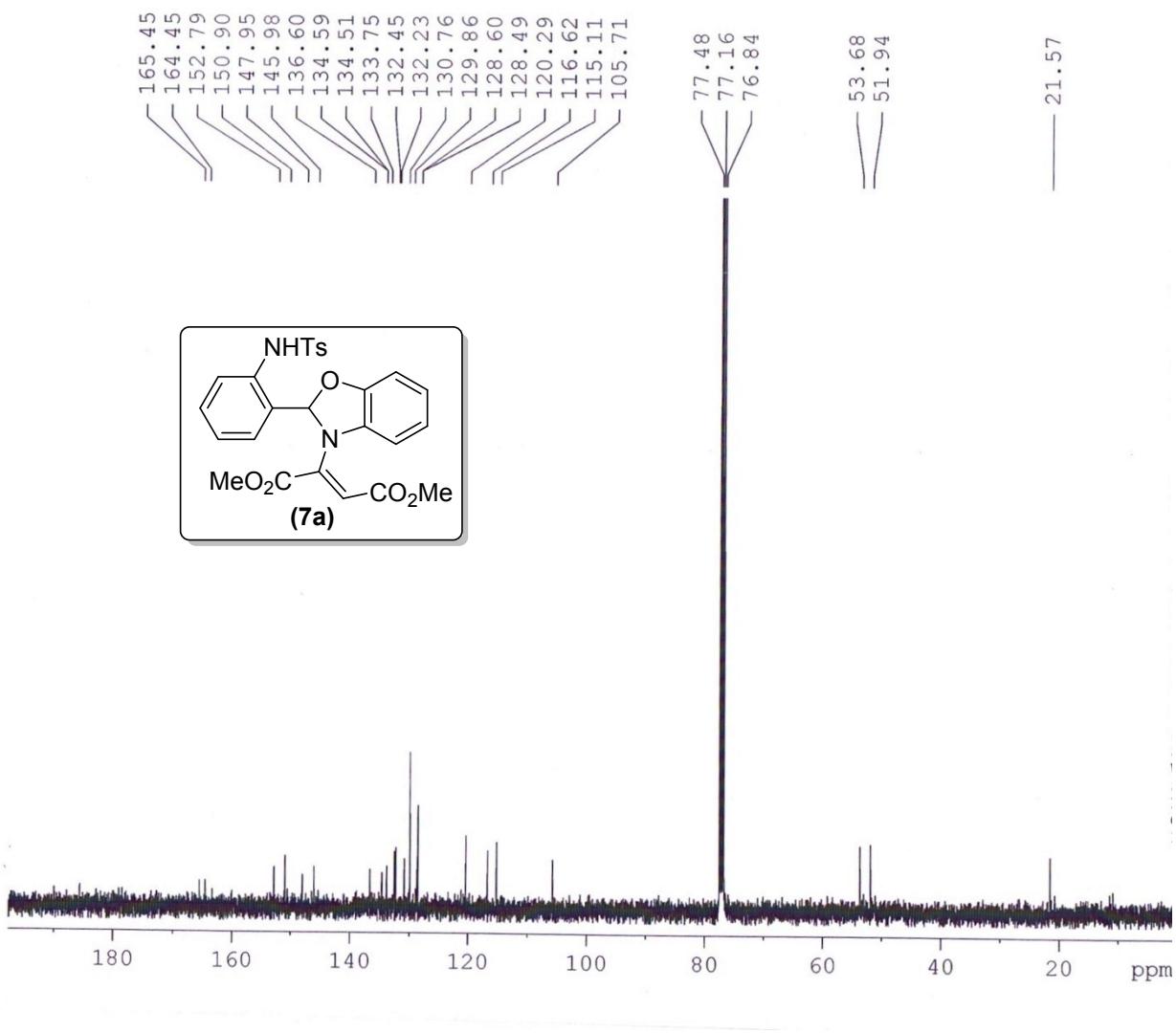



8.208
8.198
8.194
7.589
7.573
7.563
7.546
7.525
7.312
7.296
7.290
7.262
7.223
7.204
7.187
7.159
7.140
7.088
7.067
6.977
6.958
6.916
6.896
6.878
6.770
5.023
4.016
3.620
2.193
-0.000

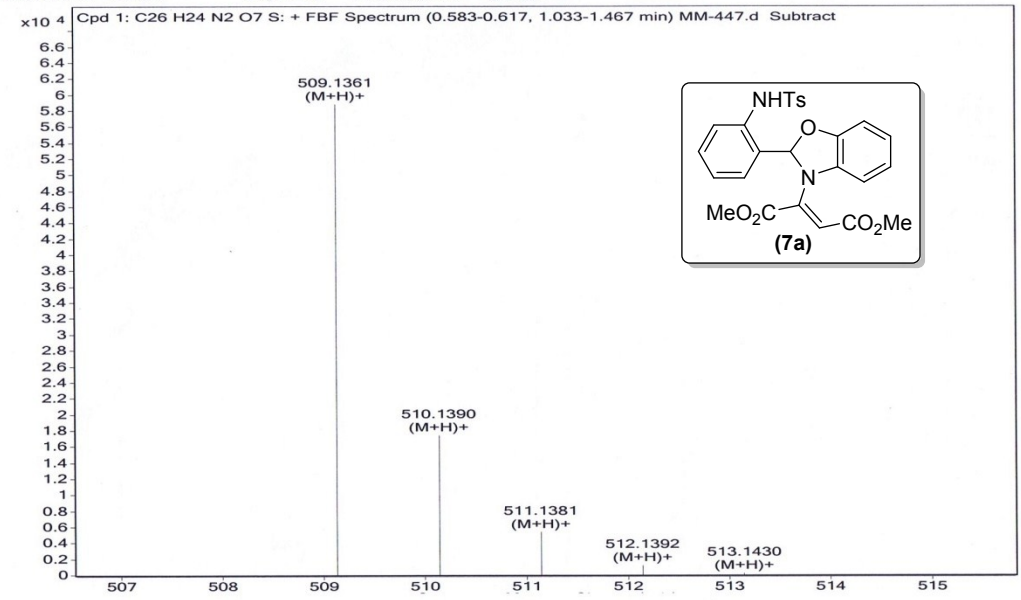

NHTs
O
N
MeO2C
CO2Me

(7a)

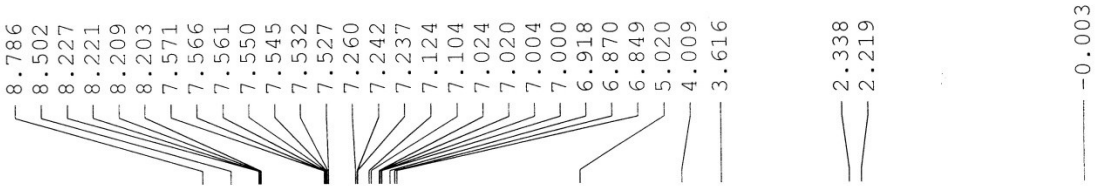

9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.06
4.35
1.21
2.01
1.34
2.15
1.10
1.10
0.97
2.98
3.09
3.10

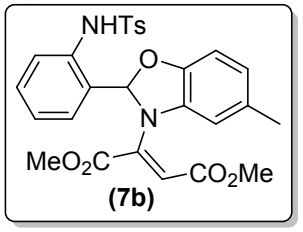

165.45
164.45
152.79
150.90
147.95
145.98
136.60
134.59
134.51
133.75
132.45
132.23
130.76
129.86
128.60
128.49
120.29
116.62
115.11
105.71
77.48
77.16
76.84
53.68
51.94
21.57
NHTs
O
N
MeO2C
CO2Me
(7a)
180
160
140
120
100
80
60
40
20
ppm

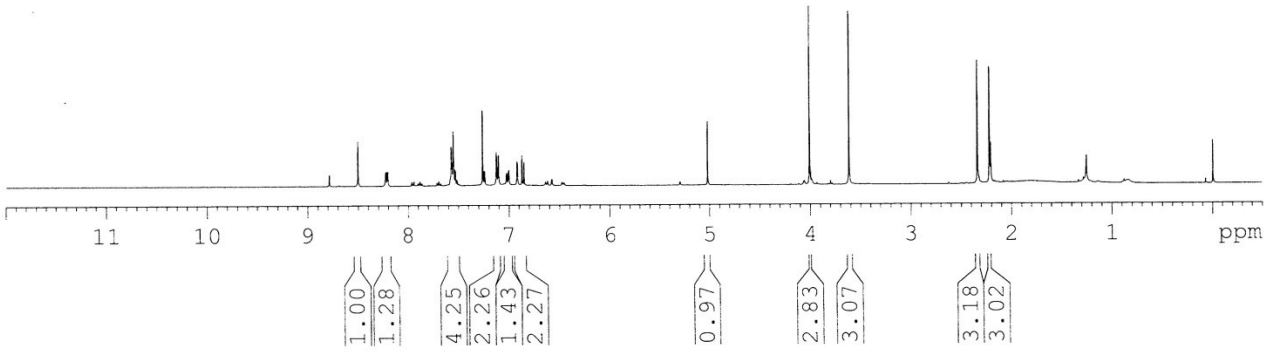
Sample Name	MM-447	**Position**		**Instrument Name**	Q-TOF	**User Name**	QTOF-PU\admin
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	MM-447.d	**ACQ Method**	Pondicherry Universi	**Comment**			

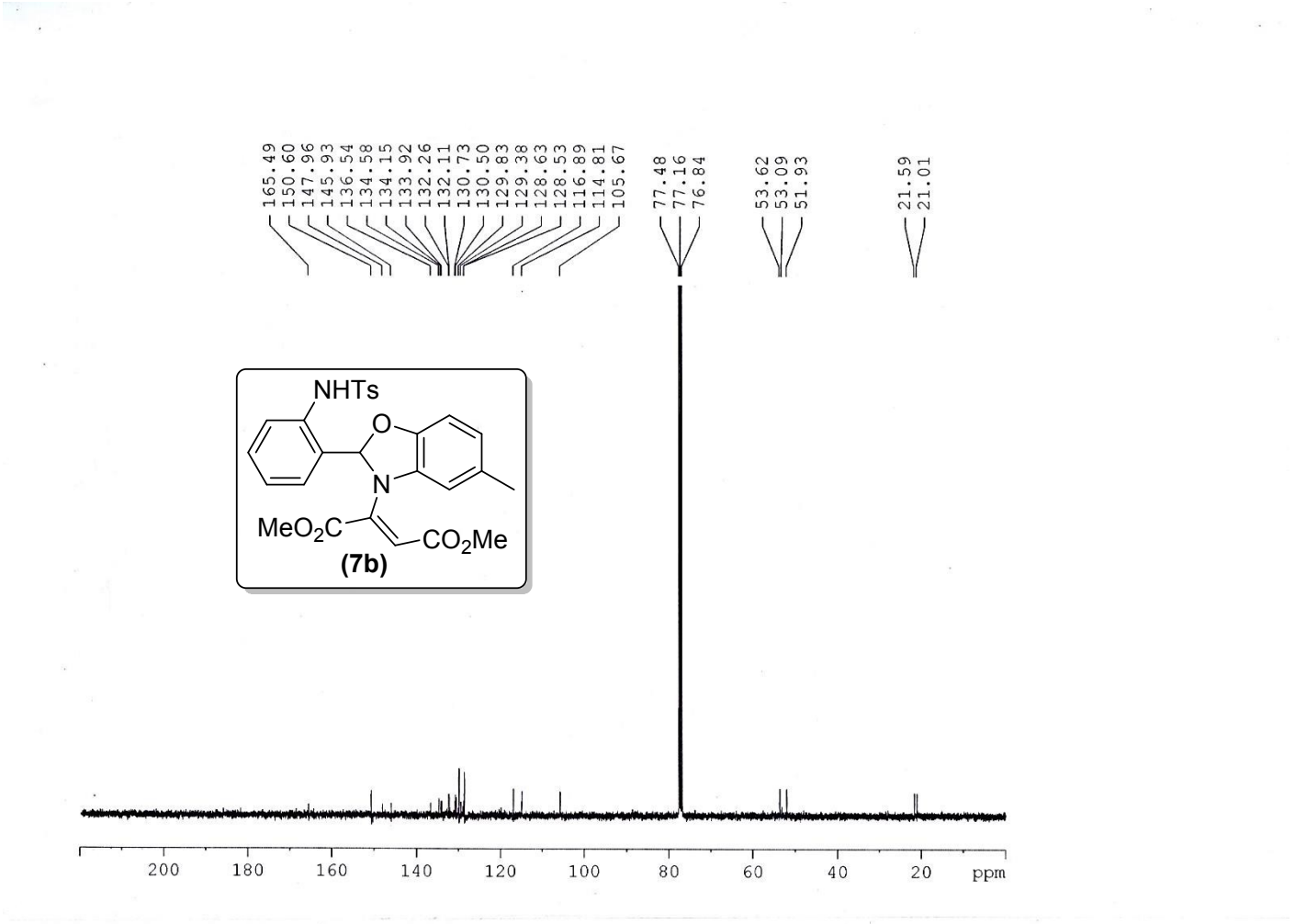


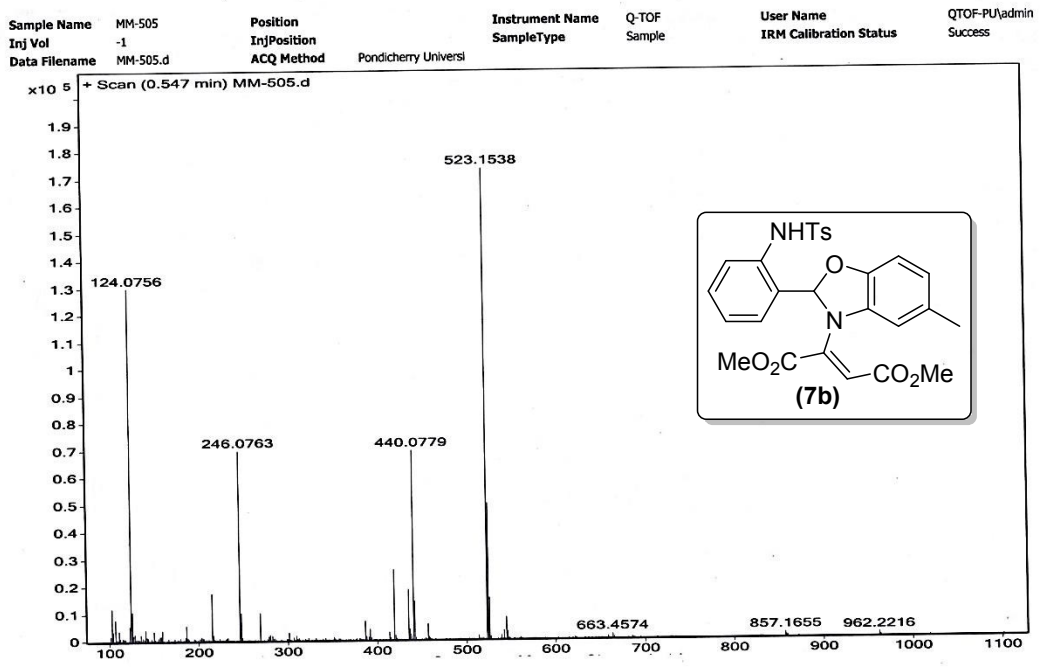
PROTON CDCl3 {D:\MB} KOPAL 1








165.49
150.60
147.96
145.93
136.54
134.58
134.15
133.92
132.26
132.11
130.73
130.50
129.83
129.38
128.63
128.53
116.89
114.81
105.67
77.48
77.16
76.84
53.62
53.09
51.93
21.59
21.01
NHTs
O
N
MeO2C
CO2Me
(7b)
200
180
160
140
120
100
80
60
40
20
ppm


Sample Name MM-505
Inj Vol -1
Data Filename MM-505.d
Position
InjPosition
ACQ Method Pondicherry Universi
Instrument Name Q-TOF
SampleType Sample
User Name QTOF-PU\admin
IRM Calibration Status Success
+ Scan (0.547 min) MM-505.d
x10 5
523.1538
124.0756
246.0763
440.0779
663.4574
857.1655
962.2216
NHTs
O
N
MeO2C
CO2Me
(7b)