The acid-promoted reactions of phenyliodonium ylides with substituted anilines and their applications to the synthesis of indoles

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Phenyliodonium ylides (**2**)[1] were prepared according to reported methods.

General procedure for the reactions of 1 with 2:

13 μL of $BF_3{\cdot}Et_2O$ was added to the mixture of 1 mmol **1** and 1 mmol **2** in 2 mL methanol, and the mixture was stirred at room temperature for 5 min. The solvent was then removed under reduced pressure, and the residual was treated with silica gel chromatography to give the pure product **3**.

Procedure for the one-pot synthesis of indoles under thermal conditions:

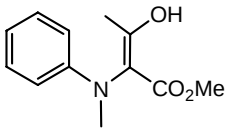
A mixure of 1 mmol **1**, 1 mmol **2,** and 13 μL of $BF_3{\cdot}Et_2O$ in 3 mL toluene was stirred in a 10 mL round bottom flask at room temperature for 30 min. Then 300 mg of Amberlyst^R 15 was added into the reaction mixture, followed by fitting the flask with a condenser. The reaction mixture was then stirred at reflux for 12 hours. The solvent was removed under reduced pressure, and the residual was subject to silica gel chromatography to give the indole product **4**.

General procedure for the synthesis of indoles from 1 and 2 under photochemical conditions:

To a Pyrex tube containing a solution of 1 mmol **1** in 10 mL of benzene and 10 mL of methanol was added 1 mmol **2.** The solution was bubbled with argon for 15 min. Then 0.6 mL TFAA was added in and the solution was irradiated with a 500W medium-pressure mercury lamp under argon atmosphere at room temperature for 12 hours. After irradiation, the solvent was removed under reduced pressure, and the

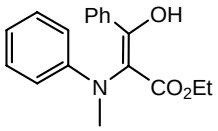
residual was treated with silica gel chromatography to give the product **4**.

Spectroscopic data of the products



3aa Ref. 1

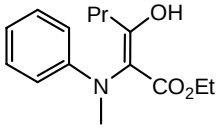
^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 1.95 (s, 3H), 3.03 (s, 3H), 3.66 (s, 3H), 6.60–6.62 (d, 2H, *J* = 8.0 Hz), 6.70–6.74 (m, 1H), 7.19–7.23 (m, 2H), 12.26 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 17.4, 38.7, 51.7, 110.6, 111.7, 116.9, 129.1, 148.8, 172.5, 175.9, EI-MS *m*/*z* (rel. int., %): 221 (M^+, 34), 118 (100), 104 (48), 77 (45), 43 (69).



3ab New compound

White solid, mp: 126-129°C.

^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 1.07-1.10 (t, 3H, *J* = 7.2 Hz), 2.92 (s, 3H), 4.11-4.22 (q, 2H, *J* = 7.2 Hz), 6.69-6.72 (m, 2H), 6.74-6.76 (m, 1H), 7.19-7.23 (m, 2H), 7.27-7.29 (m, 2H), 7.30-7.37 (m, 2H), 7.72-7.74 (d, 2H, *J* = 8.8 Hz), 12.88 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 14.0, 38.9, 60.9, 111.2, 112.5, 117.2, 127.9, 128.1, 129.0, 130.6, 133.2, 149.4, 170.3, 173.1; EI-MS *m*/*z* (rel. int., %): 297 (M^+, 34), 192 (51), 118 (67), 105 (100), 77 (96), 51 (43).

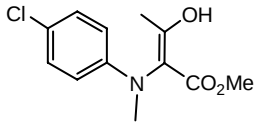


3ac New compound

Oil liquid.

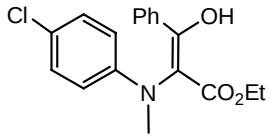
^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 0.89-0.92 (t, 3H, *J* = 7.2 Hz), 1.09-1.13 (t, 3H, *J*

= 7.2 Hz), 1.58-1.63 (m, 2H), 2.21-2.35 (m, 2H), 3.03 (s, 1H), 4.04-4.10 (m, 1H), 4.12-4.20 (m, 1H), 6.61-6.64 (d, 2H, *J* = 7.2 Hz), 6.70-6.74 (t, 1H, *J* = 7.2 Hz), 7.18-7.21 (t, 2H, *J* = 7.2 Hz), 12.40 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 14.0, 14.2, 19.4, 32.7, 39.4, 60.5, 110.8, 112.1, 116.9, 128.9, 149.4, 172.2, 178.7; EI-MS *m*/*z* (rel. int., %): 263 (M^+, 39), 164 (21), 147 (48), 40 (100); HRMS (ESI): calcd for $C_{15}H_{21}NO_3$ + Na = 286.1414, found: 286.1415.



3ba Ref. 1

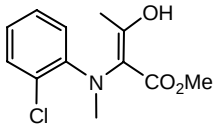
^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 1.95 (s, 3H), 3.01 (s, 3H), 3.68 (s, 3H), 6.52–6.54 (d, 2H, *J* = 8.8 Hz), 7.13–7.15 (d, 2H, *J* = 8.8 Hz), 12.24 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 17.4, 39.0, 51.9, 110.5, 113.0, 122.0, 128.9, 147.5, 172.2, 176.0; EI-MS *m*/*z* (rel. int., %): 255 (M^+, 14), 196 (43), 152 (59), 138 (20), 111 (23), 75 (18), 43 (100).



3bb New compound

Oil liquid.

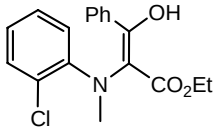
^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 1.10-1.13 (t, 3H, *J* = 7.2 Hz), 2.90 (s, 3H), 4.14-4.21 (q, 2H, *J* = 7.2 Hz), 6.60-6.62 (d, 2H, *J* = 8.8 Hz), 7.13-7.16 (d, 2H, *J* = 8.8 Hz), 7.29-7.32 (m, 2H), 7.36-7.39 (m, 1H), 7.68-7.70 (d, 2H, *J* = 8.8 Hz), 12.89 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 14.1, 39.2, 61.1, 110.8, 113.0, 122.2, 127.8, 128.2, 128.9, 130.8, 133.0, 148.0, 170.6, 172.8; EI-MS *m*/*z* (rel. int., %): 331 (M^+, 14), 152 (70), 105 (100), 77 (80), 51 (23); HRMS (ESI): calcd for $C_{18}H_{18}ClNO_3$ + H = 332.1048, found: 332.1054.



3ca New compound

White solid, mp: 107-110°C.

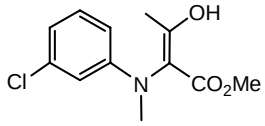
^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 1.95 (s, 3H), 3.06 (s, 3H), 3.74 (s, 3H), 6.77–6.81 (t, 1H, *J* = 8.2 Hz), 6.92-6.95 (d, 1H, *J* = 9.2 Hz), 7.13–7.17 (t, 1H, *J* = 8.2 Hz), 7.22–7.25 (d, 1H, *J* = 9.2 Hz), 12.16 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 18.0, 41.5, 51.6, 112.9, 119.4, 121.0, 124.0, 127.6, 131.4, 147.1, 172.4, 174.3; EI-MS *m*/*z* (rel. int., %): 255 (M^+, 14), 212 (21), 152 (42), 138 (24), 111 (15), 75 (22), 43 (100); HRMS (ESI): calcd for $C_{12}H_{14}ClNO_3$ + H = 256.0735, found: 256.0738.



3cb New compound

Oil liquid.

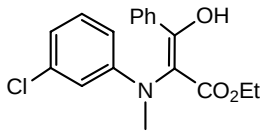
As the mixture of enol and ketone form.



3da New compound.

White solid, mp: 110-112°C

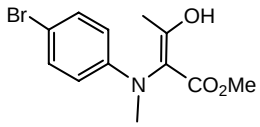
^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 1.96 (s, 3H), 3.02 (s, 3H), 3.69 (s, 3H), 6.47–6.49 (d, 1H, *J* = 8.2 Hz), 6.59–6.60 (s, 1H), 6.69–6.71 (d, 1H, *J* = 8.2 Hz), 7.09-7.13 (t, 1H, *J* = 8.2 Hz), 12.26 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 17.4, 38.9, 51.9, 110.2, 111.8, 117.0, 130.1, 135.1, 150.1, 172.1, 176.0; EI-MS *m*/*z* (rel. int., %): 255 (M^+, 10), 152 (44), 138 (24), 111 (27), 75 (29), 43 (100). HRMS (ESI): calcd for $C_{12}H_{14}ClNO_3$ + H = 256.0735, found: 256.0741.



3db New compound

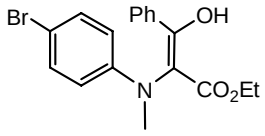
Oil liquid.

^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 1.11-1.14 (t, 3H, *J* = 7.2 Hz), 2.91 (s, 3H), 4.16-4.22 (q, 2H, *J* = 7.2 Hz), 6.55-6.58 (d, 1H, *J* = 10.4 Hz), 6.67 (s, 1H), 6.71-6.73 (d, 1H, 8.8 Hz), 7.09-7.13 (t, 1H, *J* = 8.0 Hz), 7.29-7.33 (m, 2H), 7.36-7.40 (m, 1H), 7.67-7.69 (d, 2H, *J* = 8.4 Hz), 12.90 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 14.1, 39.2, 61.1, 110.6, 110.8, 112.3, 117.3, 127.8, 128.3, 130.0, 130.8, 132.9, 135.0, 150.6, 170.7, 172.7; EI-MS *m*/*z* (rel. int., %): 331 (M^+, 26), 228 (14), 222 (17), 152 (53), 105 (100), 77 (76); HRMS (ESI): calcd for $C_{18}H_{18}ClNO_3$ + H = 332.1048, found: 332.1052.



3ea Ref. 2

^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 1.95 (s, 3H), 3.01 (s, 3H), 3.68 (s, 3H), 6.47–6.50 (d, 2H, *J* = 9.2 Hz), 7.26–7.28 (d, 2H, *J* = 9.2 Hz), 12.24 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 17.4, 39.0, 51.9, 109.1, 110.4, 113.5, 131.8, 148.0, 172.2, 176.0; EI-MS *m*/*z* (rel. int., %): 299 (17), 267 (7), 198 (40), 43 (100); HRMS (ESI): calcd for $C_{12}H_{14}BrNO_3$ + H = 300.0230, found: 300.0228 .

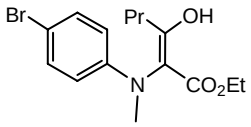


3eb New compound

Oil liquid.

^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 1.13-1.17 (t, 3H, *J* = 7.2 Hz), 2.92 (s, 3H), 4.14-4.27 (q, 2H, *J* = 7.2 Hz), 6.58-6.60 (d, 2H, *J* = 9.2 Hz), 7.27-7.35 (m, 4H),

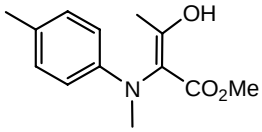
7.38-7.42 (m, 1H), 7.70-7.72 (d, 2H, *J* = 9.2 Hz), 12.91 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 14.1, 39.2, 61.1, 109.4, 110.7, 114.1, 127.8, 128.2, 130.8, 131.7, 133.0, 148.5, 170.6, 172.7; EI-MS *m*/*z* (rel. int., %): 377 (M^+, 17), 272 (17), 196 (50), 105 (100), 77 (93); HRMS (ESI): calcd for $C_{18}H_{18}BrNO_3$ + H = 376.0543, found: 378.0513.



3ec New compound

Oil liquid.

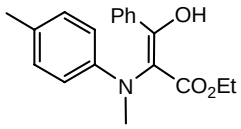
^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 0.88-0.92 (t, 3H, *J* = 7.2 Hz), 1.10-1.14 (t, 3H, *J* = 7.2 Hz), 1.57-1.62 (m, 2H), 2.19-2.29 (m, 2H), 3.01 (s, 1H), 4.05-4.11 (m, 1H), 4.12-4.21 (m, 1H), 6.48-6.50 (d, 2H, *J* = 8.8 Hz), 7.25-7.27 (d, 2H, *J* = 8.8 Hz), 12.38 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 14.0, 14.2, 19.4, 32.7, 39.5, 60.7, 109.0, 110.4, 113.7, 113.8, 131.6, 131.7, 148.5, 171.8, 178.8; EI-MS *m*/*z* (rel. int., %): 341 (M^+, 17), 211 (17), 43 (100); HRMS (ESI): calcd for $C_{15}H_{20}BrNO_3$ + H = 342.0699, found: 342.0695.



3fa New compound

White solid, mp：59–62°C.

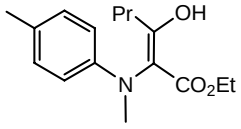
^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 1.99 (s, 3H), 2.28 (s, 3H), 3.05 (s, 3H), 3.70 (s, 3H), 6.55–6.57 (d, 2H, *J* = 8.0 Hz), 7.04-7.06 (d, 2H, *J* = 8.0 Hz), 12.29 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 17.4, 20.2, 38.8, 51.7, 110.7, 111.7, 125.9, 129.7 146.7, 172.6, 175.9; EI-MS *m*/*z* (rel. int., %): 235 (M^+, 24), 132 (100), 118 (40), 91 (34), 43 (35); HRMS (ESI): calcd for $C_{13}H_{17}NO_3$ + H = 236.1285, found: 236.1289 .



3fb New compound.

Oil liquid.

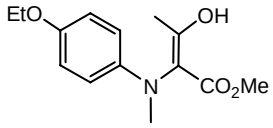
^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 1.08-1.12 (t, 3H, *J* = 7.2 Hz), 2.25 (s, 3H), 2.90 (s, 3H), 4.09-4.21 (q, 2H, *J* = 7.2 Hz), 6.59-6.61 (d, 2H, *J* = 8.4 Hz), 7.00-7.02 (d, 2H, *J* = 7.6 Hz), 7.26-7.34 (m, 3H), 7.74-7.76 (d, 2H, *J* = 8.4 Hz), 12.90 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 14.1, 20.3, 38.9, 60.9, 111.3, 112.4, 126.1, 127.9, 128.1, 129.6, 130.5, 133.3, 147.2, 170.2, 173.2; EI-MS *m*/*z* (rel. int., %): 311 (M^+, 13), 206 (9), 132 (100), 105 (52), 91 (34), 77 (94); HRMS (ESI): calcd for $C_{19}H_{21}NO_3$ + H = 312.1594 , found: 312.1597.



3fc New compound

Oil liquid.

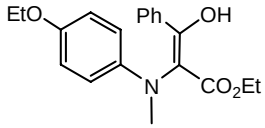
^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 0.88-0.92 (t, 3H, *J* = 7.2 Hz), 1.10-1.13 (t, 3H, *J* = 7.2 Hz), 1.57-1.63 (m, 2H), 2.24 (s, 3H), 2.25-2.35 (m, 2H), 3.01 (s, 3H), 4.04-4.10 (m, 1H), 4.14-4.21 (m, 1H), 6.52-6.54 (d, 2H, *J* = 8.4 Hz), 6.99-7.01 (d, 2H, *J* = 8.4 Hz), 12.39 (s, 1H); ^{3}C NMR ($CDCl_3$, 100 MHz, δ ppm): 14.0, 14.2, 19.4, 20.2, 32.6, 39.4, 60.4, 110.8, 112.0, 125.8, 129.6, 130.2, 137.5, 147.2, 172.3, 178.7; EI-MS *m*/*z* (rel. int., %): 277 (M^+, 30), 160 (64), 147 (62), 132 (72), 91 (99), 43 (100); HRMS (ESI): calcd for $C_{16}H_{23}NO_3$ + H = 278.1751 , found: 278.1754.



3ga New compound

Yellow solid, mp：46–48 °C.

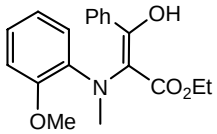
^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 1.36-1.39 (t, 3H, *J* = 7.2 Hz), 1.97 (s, 3H), 3.00 (s, 3H), 3.67 (s, 3H), 3.94-3.99 (q, 2H, *J* = 7.2 Hz), 6.52-6.55 (d, 2H, *J* = 9.2 Hz), 6.79-6.81 (d, 2H, *J*= 9.2 Hz), 12.23 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 15.1, 17.5, 39.0, 51.7, 64.0, 111.1, 112.6, 115.5, 143.3, 150.9, 172.7, 176.5; EI-MS *m*/*z* (rel. int., %): 265 (M^+, 23), 237 (34), 162 (100), 150 (47), 134 (29), 122 (75), 43 (84); HRMS (ESI): calcd for $C_{14}H_{19}NO_4$ + H = 266.1387, found: 266.1389.



3gb

Oil liquid.

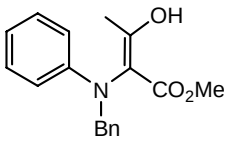
As the mixture of enol and ketone form.
EI-MS *m*/*z* (rel. int., %): 251 (31), 208 (48), 176 (64), 134 (76), 77 (36), 43 (100); HRMS (ESI): calcd for $C_{13}H_{17}NO_4$ + H = 252.1230, found: 252.1227.



3hb New compound.

Yellow solid, mp：81–83°C

^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 1.25-1.28 (t, 3H, *J* = 7.2 Hz), 2.92 (s, 3H), 3.87 (s, 3H), 4.24-4.28 (q, 2H, *J* = 7.2 Hz), 5.90 (s, 1H), 6.78-6.93 (m, 3H), 6.96-6.98 (m, 1H), 7.39-7.43 (t, 2H, 7.8 Hz), 7.54-7.58 (t, 1H, 7.4 Hz), 8.04-8.07 (d, 2H, *J* = 8.4 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 14.2, 36.4, 55.5, 60.9, 69.5, 111.6, 120.3, 121.2, 122.6, 127.8, 128.3, 128.5, 128.8, 129.0, 129.9, 133.6, 135.8, 139.4, 151.7, 169.8, 195.3; EI-MS *m*/*z* (rel. int., %): 327 (M^+, 11), 238 (18), 222 (100), 194 (20), 148 (22), 134 (48), 105 (62), 77 (96), 51 (33), 42 (21); HRMS (ESI): calcd for $C_{19}H_{21}NO_4$ + H = 328.1543, found: 328.1550.

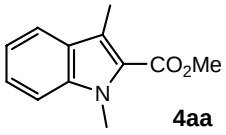


3ia Ref. 1

^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 1.83 (s, 3H), 3.68 (s, 3H), 4.38-4.42 (d, 1H, *J* = 15.2 Hz), 4.69-4.72 (d, 1H, *J* = 15.2 Hz), 6.70-6.78 (m, 3H), 7.14-7.37 (m, 7H), 12.38 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 18.1, 51.7, 56.2, 109.7, 113.0, 117.7, 127.0, 128.1, 128.3, 129.2, 138.2, 148.7, 172.6, 176.8; EI-MS *m*/*z* (rel. int., %): 297 (M^+, 12), 206 (26), 104 (86), 91 (80), 77 (60), 43 (100).

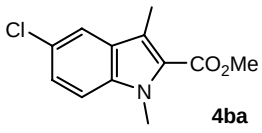
192 (51), 118 (67), 105 (100), 77 (96), 51 (43).

127.8, 128.2, 128.9, 130.8, 133.0, 148.0, 170.6, 172.8; EI-MS *m*/*z* (rel. int., %): 331 (M^+, 14), 152 (70), 105 (100), 77 (80), 51 (23); HRMS (ESI): calcd for $C_{18}H_{18}ClNO_3$ + H = 332.1048, found: 332.1054.



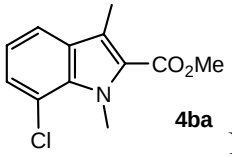
4aa Ref. 1

^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 2.58 (s, 3H), 3.93 (s, 3H), 3.99 (s, 3H), 7.11-7.15 (m, 1H), 7.33-7.37 (m, 2H), 7.65-7.67 (d, 1H, *J* = 8.0 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 10.8, 32.0, 51.3, 110.0, 119.6, 120.7, 124.8, 125.3, 127.1, 138.8, 163.5; EI-MS *m*/*z* (rel. int., %): 203 (M^+, 100), 188 (79), 144 (72), 77 (46).



4ba Ref. 1

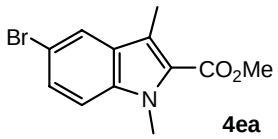
^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 2.52 (s, 3H), 3.94 (s, 3H), 3.97 (s, 3H), 7.23-7.29 (m, 2H), 7.61 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 10.7, 32.2, 51.5, 111.2, 119.9, 120.0, 125.4, 125.6, 125.9, 127.9, 137.0, 163.2; EI-MS *m*/*z* (rel. int., %): 237 (M^+, 100), 222 (89), 178 (53), 75 (35).



4ba New compound.

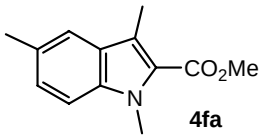
White solid, mp：41–44°C

^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 2.51 (s, 3H), 3.95 (s, 3H), 4.32 (s, 3H), 6.99–7.02 (t, 1H, *J* = 7.8 Hz), 7.26–7.28 (d, 1H, *J* = 8.0 Hz), 7.51–7.53 (d, 1H, *J* = 8.0 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 10.7, 34.5, 51.6, 117.4, 119.5, 120.1, 120.8, 127.1, 130.2, 134.5, 163.0; EI-MS *m*/*z* (rel. int., %): 237 (M^+, 100), 222 (61), 178 (31); HRMS (ESI): calcd for $C_{12}H_{12}ClNO_2$ + H = 238.0648 , found: 238.0654.



4ea Ref. 3.

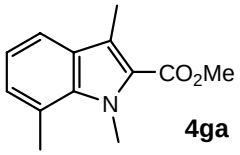
^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 2.51 (s, 3H), 3.94 (s, 3H), 3.96 (s, 3H), 7.18-7.21 (d, 1H, *J* = 8.8 Hz), 7.38-7.41 (s, 1H, *J* = 8.8 Hz), 7.77 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 10.7, 32.2, 51.5, 111.5, 112.8, 119.8, 123.2, 125.7, 128.0, 128.6, 137.2, 163.2; EI-MS *m*/*z* (rel. int., %): 283 (M^+, 100), 268 (67), 222 (34), 143 (26), 102 (20).



4fa New compound

Oil liquid.

^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 2.45 (s, 3H), 2.55 (s, 3H), 3.93 (s, 3H), 3.97 (s, 3H), 7.16-7.18 (d, 1H, *J* = 8.4 Hz), 7.21-7.24 (d, 1H, *J* = 8.4 Hz), 7.42-7.43 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 10.8, 21.4, 32.0, 51.2, 109.7, 120.0, 120.2, 124.8, 127.2, 128.9, 137.4, 163.6; EI-MS *m*/*z* (rel. int., %): 217 (M^+, 100), 202 (76), 186 (14), 158 (37), 115 (63), 39 (34).

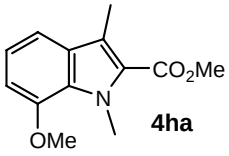


4ga New compound.

Yellow solid, mp：81–83°C

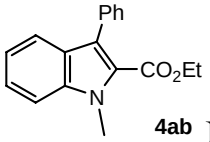
^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 2.53 (s, 3H), 2.78 (s, 3H), 3.94 (s, 3H), 4.21 (s, 3H), 6.98-7.05 (m, 2H), 7.48-7.50 (d, 1H, *J* = 7.6 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz,

δ ppm): 10.8, 20.8, 34.7, 51.4, 118.7, 119.8, 121.0, 121.9, 126.1, 128.0, 128.2, 138.6, 163.5; EI-MS *m*/*z* (rel. int., %): 217 (M^+, 93), 202 (42), 115 (100), 91 (53), 77 (48), 65 (38), 51 (42), 43 (39); HRMS (ESI): calcd for $C_{13}H_{15}NO_2$ + H = 218.1176, found: 218.1175.



4ha Ref. 1

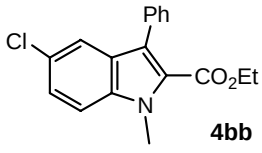
^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 2.53 (s, 3H), 3.92 (s, 3H), 3.93 (s, 3H), 4.28 (s, 3H), 6.69-6.72 (d, 1H, *J* = 7.6 Hz), 6.99-7.03 (t, 1H, *J* = 7.6 Hz), 7.22-7.26 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 11.0, 35.0, 51.3, 55.5, 105.4, 113.2, 120.0, 121.0, 125.7, 129.1, 148.0, 163.4; EI-MS *m*/*z* (rel. int., %): 233 (M+, 100), 218 (64), 45 (18).



4ab New compound.

Oil liquid.

^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 1.02-1.06 (t, 3H, *J* = 7.2 Hz), 4.06 (s, 3H), 4.16-4.19 (q, 2H, *J* = 7.2 Hz), 7.11-7.15 (m,1H), 7.33-7.44 (m, 7H), 7.55-7.57 (d, 1H, *J* = 8.4 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 13.6, 31.9, 60.5, 110.0, 120.6, 121.5, 124.4, 125.2, 126.6, 126.8, 127.7, 130.4, 134.8, 138.4, 162.7; EI-MS *m*/*z* (rel. int., %): 279 (M^+, 100), 251 (48), 207 (36), 190 (24), 165 (42); HRMS (ESI): calcd for $C_{18}H_{17}NO_2$ + H = 280.1332, found: 280.1334.

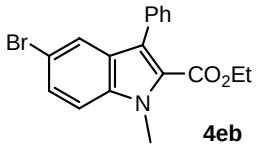


4bb New compound.

Oil liquid.

^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 1.03-1.06 (t, 3H, *J* = 7.2 Hz), 4.06 (s, 3H), 4.15-4.20 (q, 2H, *J* = 7.2 Hz), 7.32-7.34 (m, 2H), 7.36-7.45 (m, 5H), 7.51 (s, 1H); ^{13}C

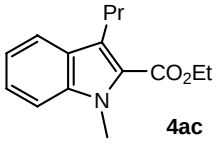
NMR ($CDCl_3$, 100 MHz, δ ppm): 13.6, 32.2, 60.7, 111.3, 113.4, 120.7, 123.8, 125.7, 126.0, 126.4, 127.1, 127.5, 127.9, 130.3, 134.1, 136.7, 162.4; EI-MS *m*/*z* (rel. int., %): 131 (M^+, 100), 285 (60), 204 (41), 190 (24), 163 (24); HRMS (ESI): calcd for $C_{18}H_{16}ClNO_2$ + H = 314.0942, found: 314.0936.



4eb New compound.

Yellow solid，mp：88–90°C

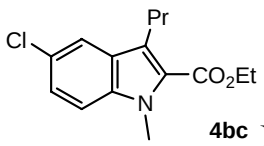
^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 1.02-1.06 (t, 3H, *J* = 7.2 Hz), 4.05 (s, 3H), 4.15-4.20 (q, 2H, *J* = 7.2 Hz), 7.28-7.30 (m, 1H), 7.37-7.45 (m, 6H), 7.66-7.67 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 13.6, 32.1, 60.7, 111.6, 113.9, 123.7, 123.9, 125.8, 127.1, 127.8, 128.1, 130.3, 134.0, 136.9, 162.3; EI-MS *m*/*z* (rel. int., %): 359 (M^+, 100), 329 (68), 204 (89), 190 (49), 163 (47); HRMS (ESI): calcd for $C_{18}H_{16}BrNO_2$ + H = 358.0437, found: 358.0431.



4ac New compound.

Oil liquid

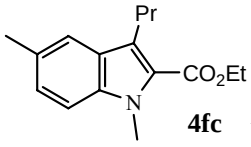
^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 0.99-1.02 (t, 3H, *J* = 7.2 Hz), 1.43-1.47 (t, 3H, *J* = 7.2 Hz), 1.65-1.75 (m, 2H), 3.05-3.09 (t, 2H, *J* = 7.6 Hz), 4.01 (s, 3H), 4.39-4.45 (q, 2H, *J* = 7.2 Hz), 7.12-7.16 (m, 1H), 7.35-7.36 (m, 2H), 7.69-7.71 (d, 1H, *J* = 8.0 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 14.3, 24.6, 27.4, 32.0, 60.3, 110.0, 119.6, 120.8, 125.0, 125.5, 126.8, 138.7, 163.0; EI-MS *m*/*z* (rel. int., %): 245 (M^+, 58), 216 (100), 188 (69); HRMS (ESI): calcd for $C_{15}H_{19}NO_2$ + H = 246.1489, found: 246.1485.



4bc New compound.

White solid, mp：70–72°C

^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 0.96-1.00 (t, 3H, *J* = 7.2 Hz), 1.41-1.45 (t, 3H, *J* = 7.2 Hz), 1.62-1.68 (m, 2H), 2.97-3.00 (t, 2H, *J* = 7.8 Hz), 3.97 (s, 1H), 4.38-4.43 (q, 2H, *J* = 7.2 Hz), 7.26-7.27 (m, 2H), 7.63 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 14.2, 14.3, 24.5, 27.3, 32.2, 60.6, 111.2, 120.0, 124.7, 125.4, 125.9, 127.6, 137.0, 162.7; EI-MS *m*/*z* (rel. int., %): 279 (M^+, 45), 250 (100), 222 (66), 43 (29); HRMS (ESI): calcd for $C_{15}H_{18}ClNO_2$ + H = 280.1099, found: 280.1097.



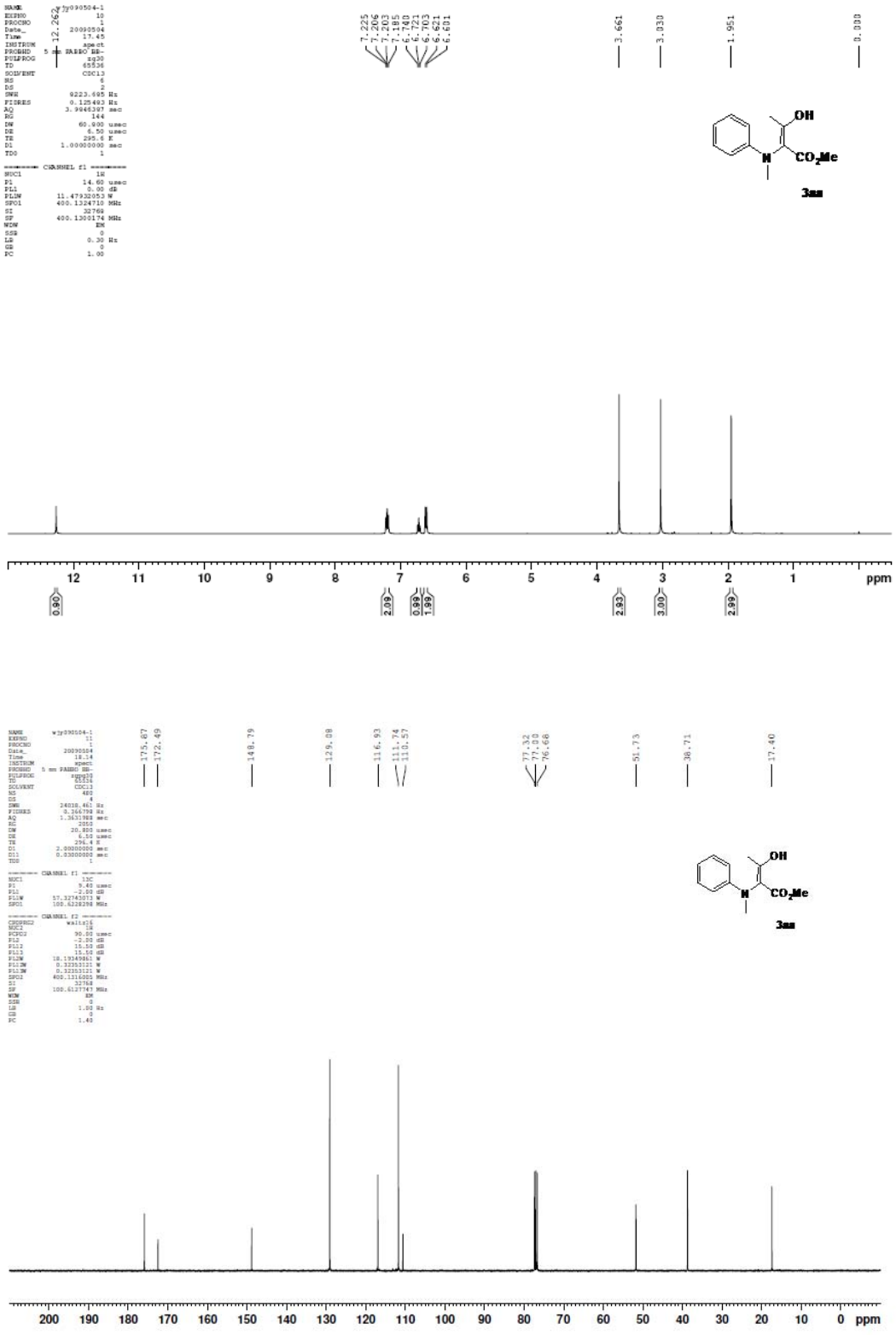
4fc New compound

Oil liquid

^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 0.97-1.01 (t, 3H, *J* = 7.2 Hz), 1.41-1.45 (t, 3H, -7.2 Hz), 1.66-1.71 (m, 2H), 2.46 (s, 3H), 3.00-3.04 (t, 2H, *J* = 7.6 Hz), 3.97 (s, 3H), 4.37-4.42 (q, 2H, *J* = 7.2 Hz), 7.16-7.18 (d, 1H, *J* = 8.4 Hz), 7.22-7.24 (d, 1H, *J* = 8.4 Hz), 7.44 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, δ ppm): 14.3, 21.4, 24.6, 27.4, 32.1, 60.3, 109.8, 120.1, 124.8, 125.0, 127.0, 128.9, 130.2, 137.4, 163.1; EI-MS *m*/*z* (rel. int., %): 259 (M^+, 41), 230 (100), 202 (72), 43 (33); HRMS (ESI): calcd for $C_{16}H_{21}NO_2$ + H = 260.1645, found: 260.1640.

References

1. Bashford, K. E.; Cooper, A. L.; Kane, P. D.; Moody, C. J.; Muthusamy, S.; Swann, E. *J. Chem. Soc. Perkin Trans. 1*, **2002**, 1672–1687.
2. Moody C. J.; Swann, E. *Synlett*, **1998**, 135.
3. Honey, M. A.; Blake, A.; J.; Campbell, I. B.; Judkins, B. D.; Moody. C. J. *Tetrahedron* **2009**, *65*, 8995.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3.00
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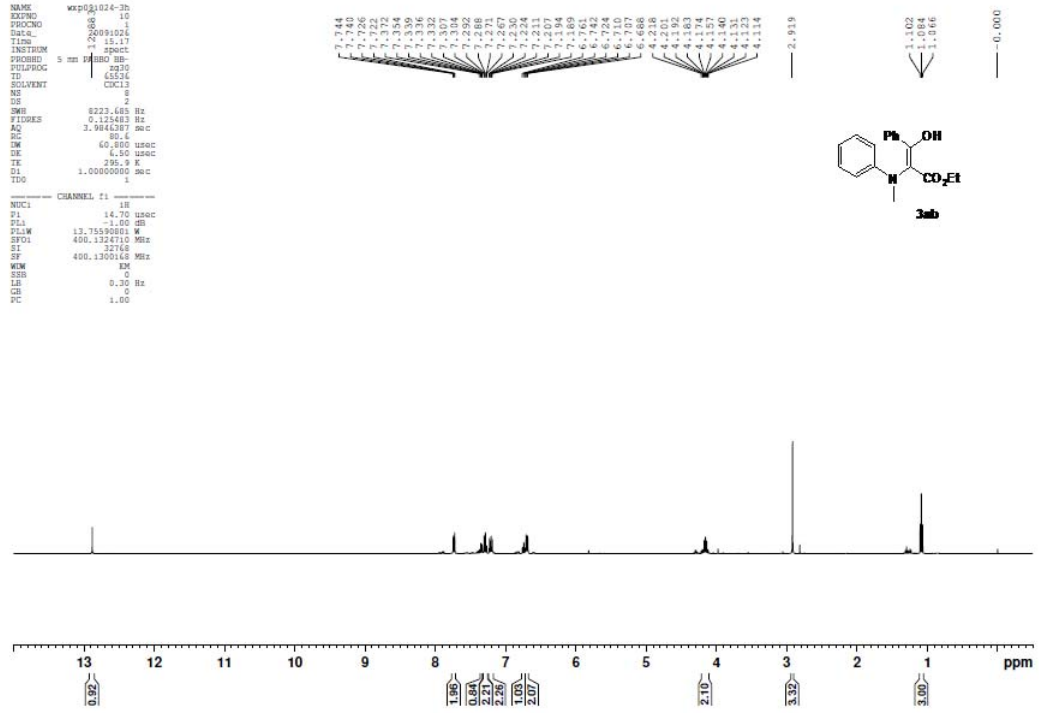
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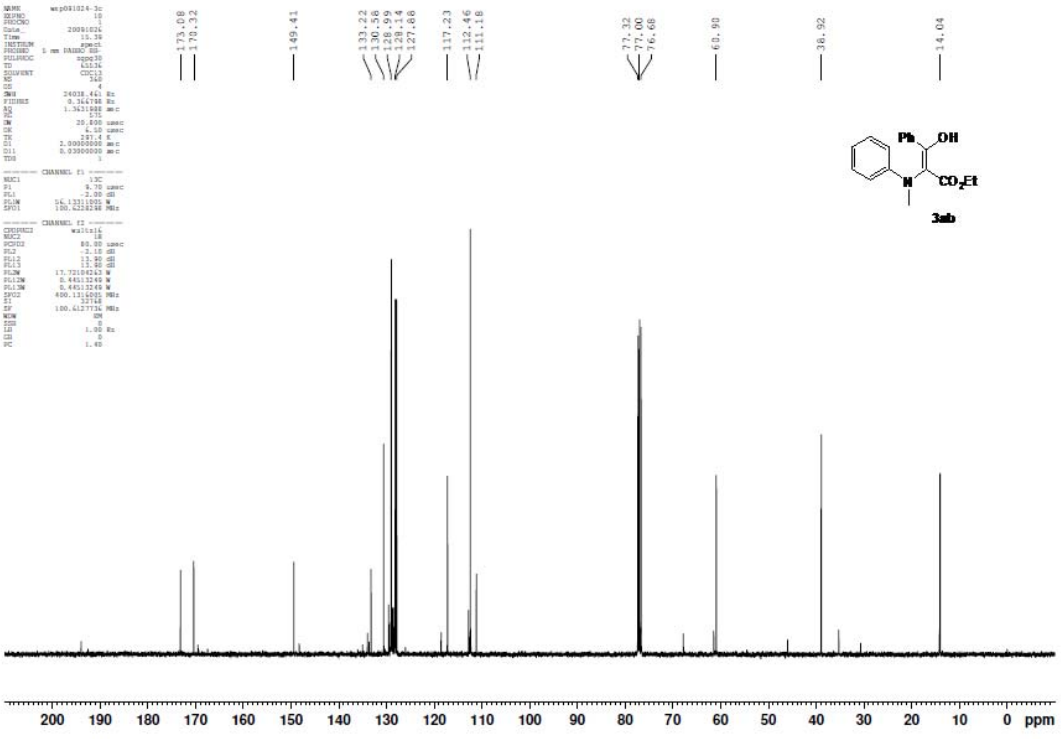
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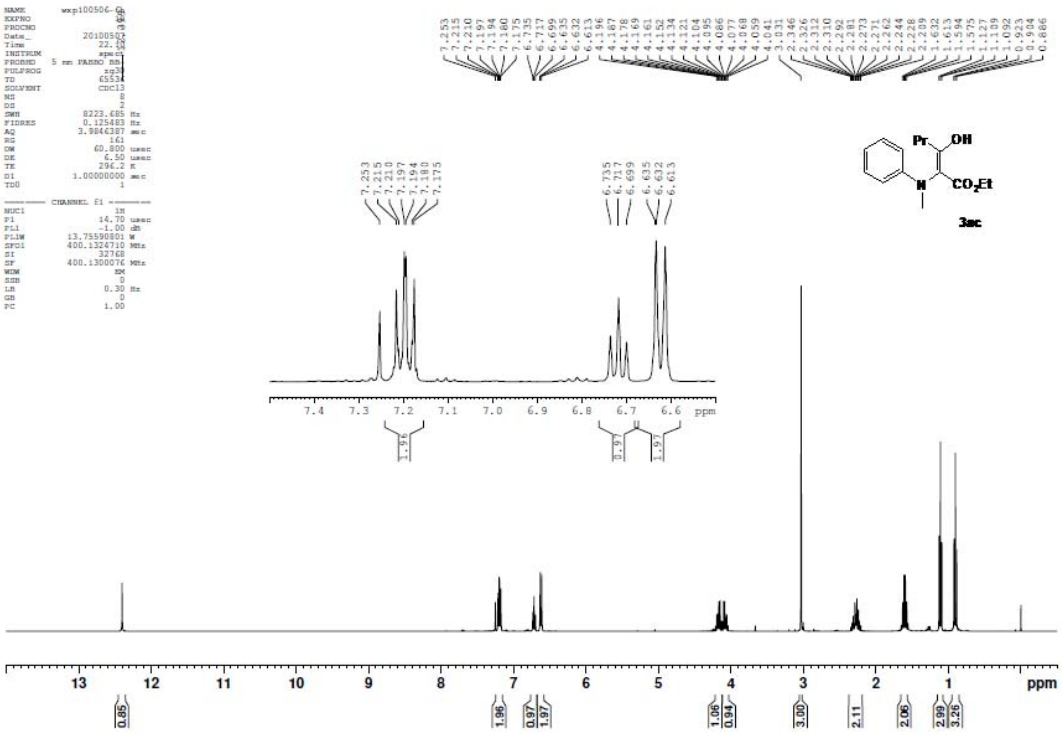
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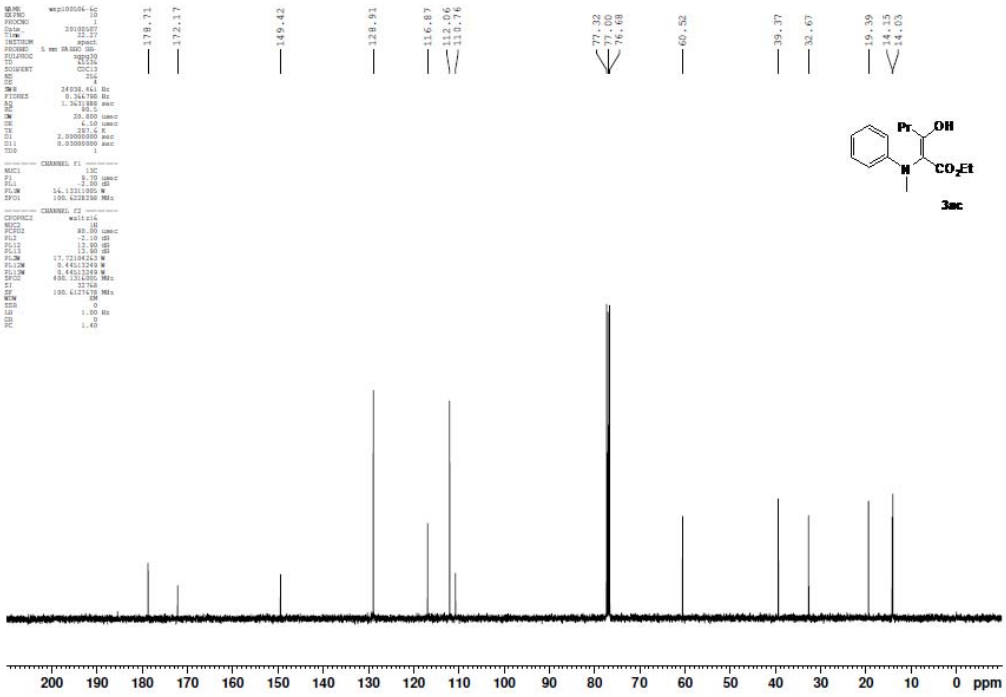
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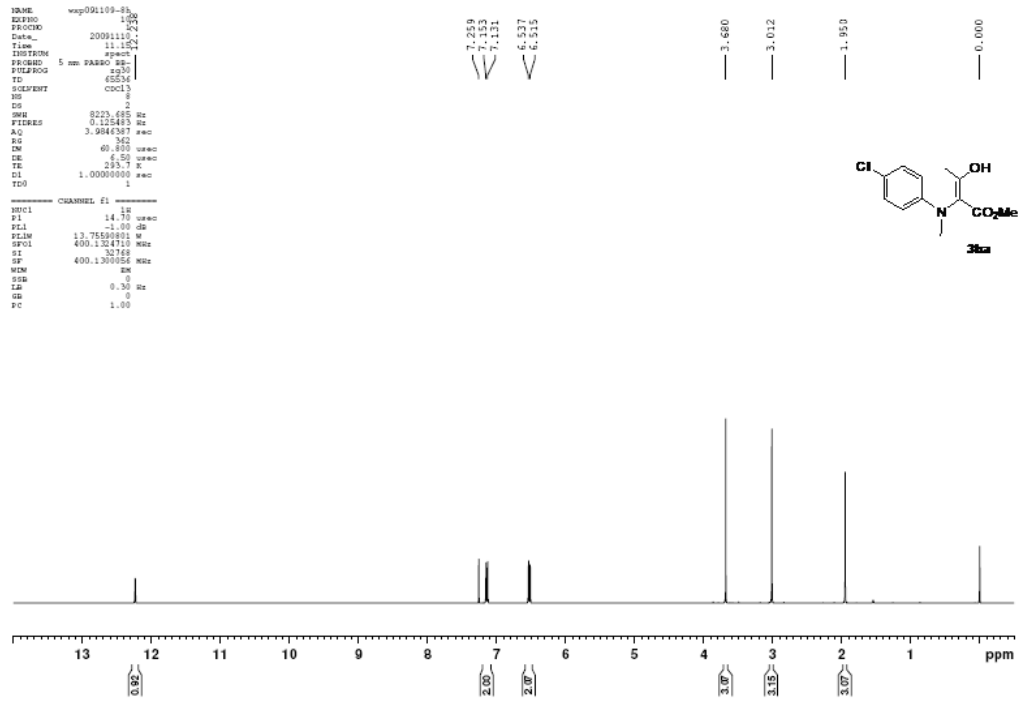
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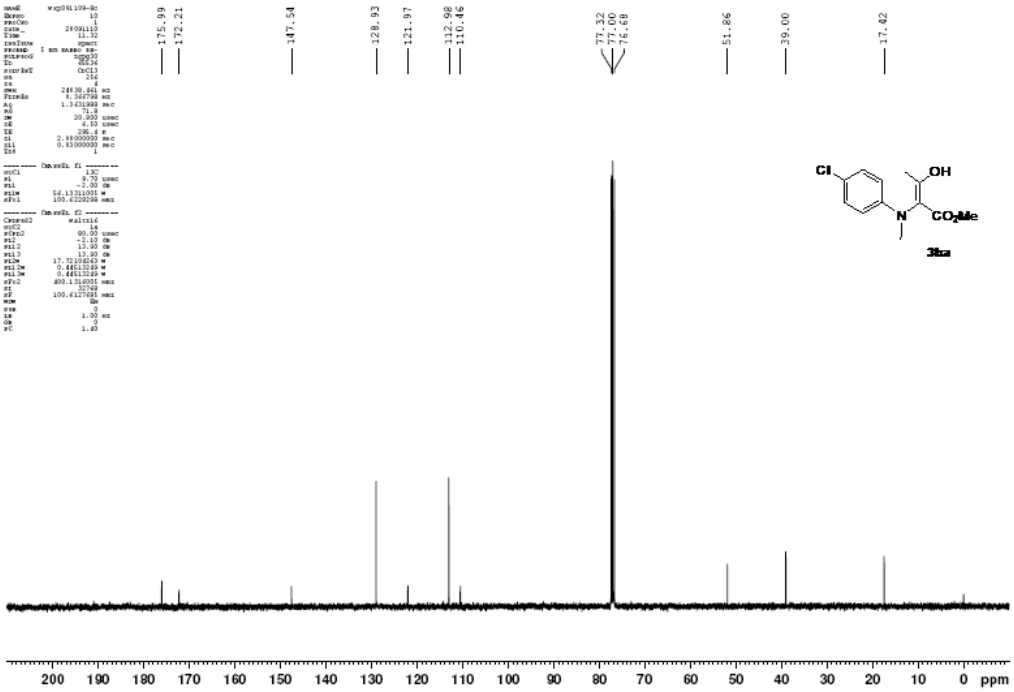
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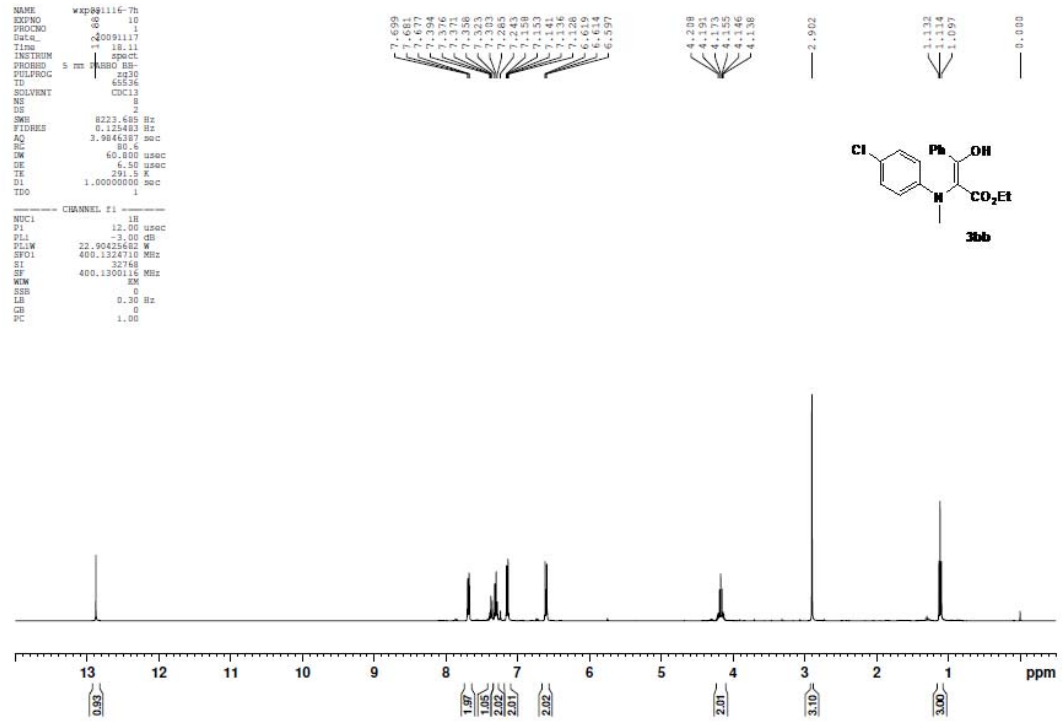
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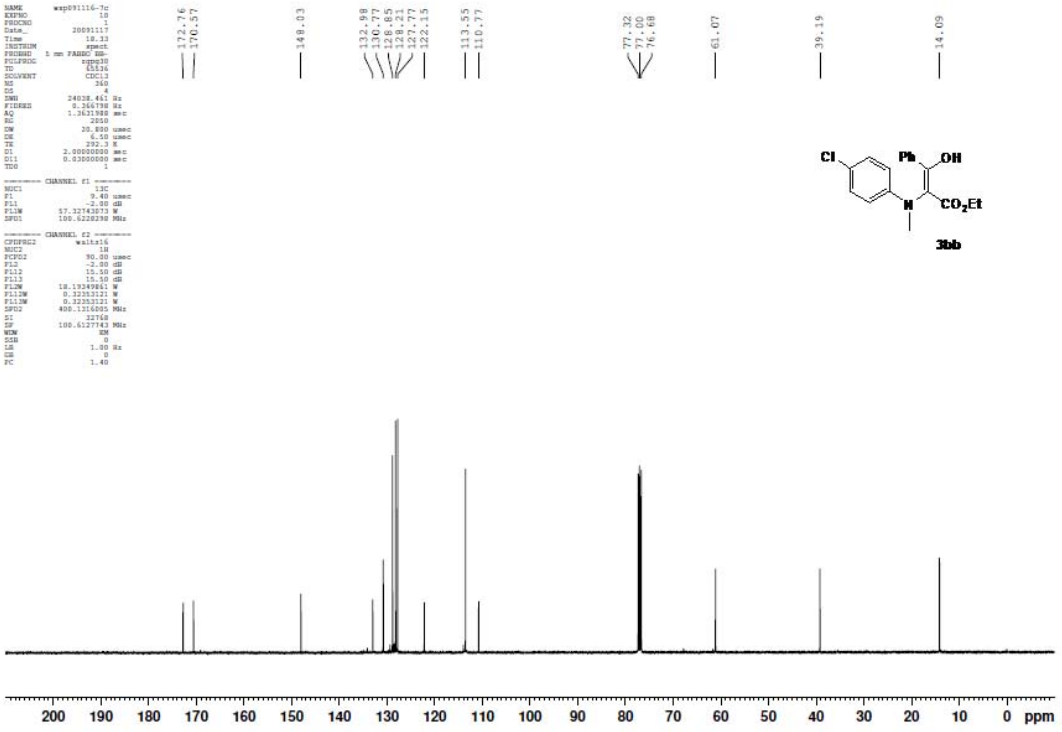
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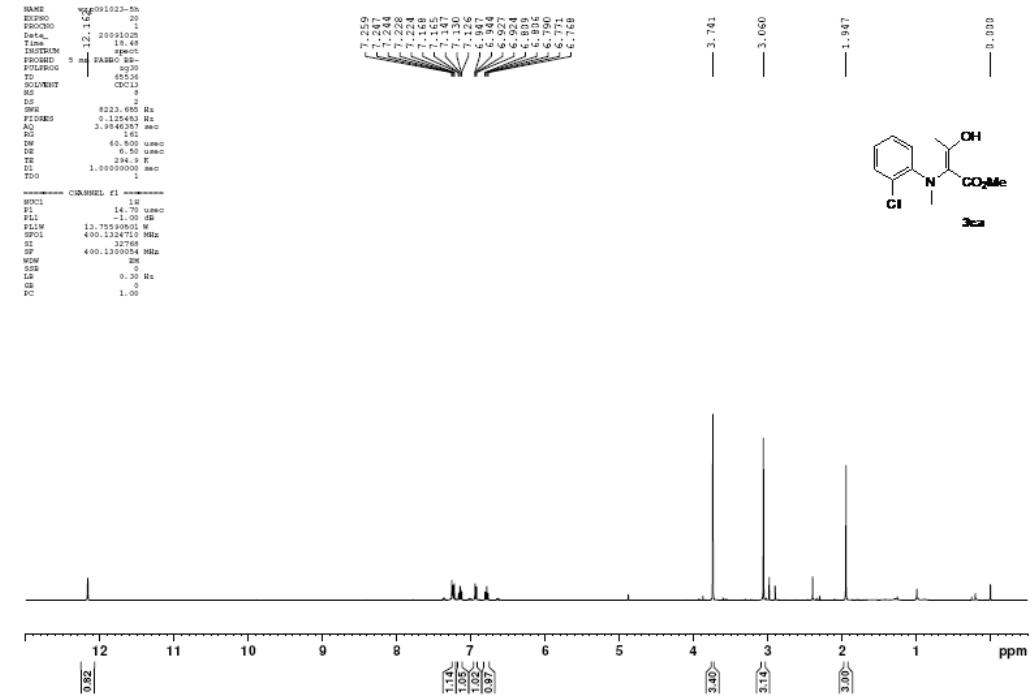
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7.147
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7.126
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6.927
6.924
6.805
6.802
6.790
6.771
6.768
3.741
3.060
1.947
0.000
OH
N
CO2Me
Cl
3ca
12
11
10
9
8
7
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1.02
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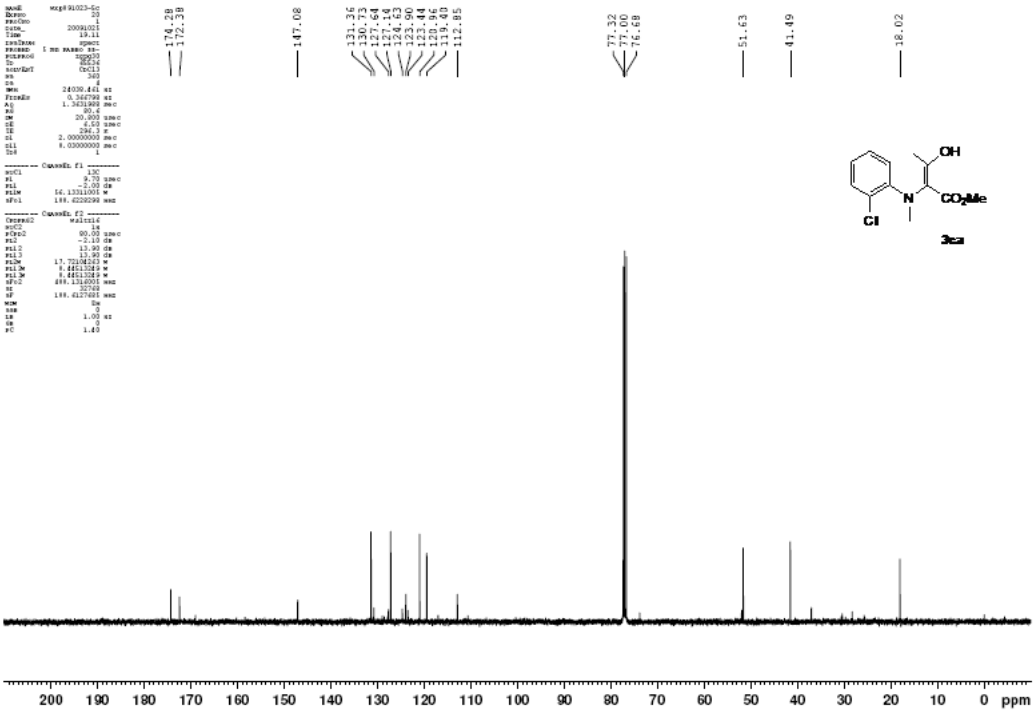
3.14
3.00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127.14
125.43
123.90
123.44
120.96
119.49
112.85
77.32
77.00
76.68
51.63
41.49
18.02
OH
N
CO2Me
Cl
3ca
200
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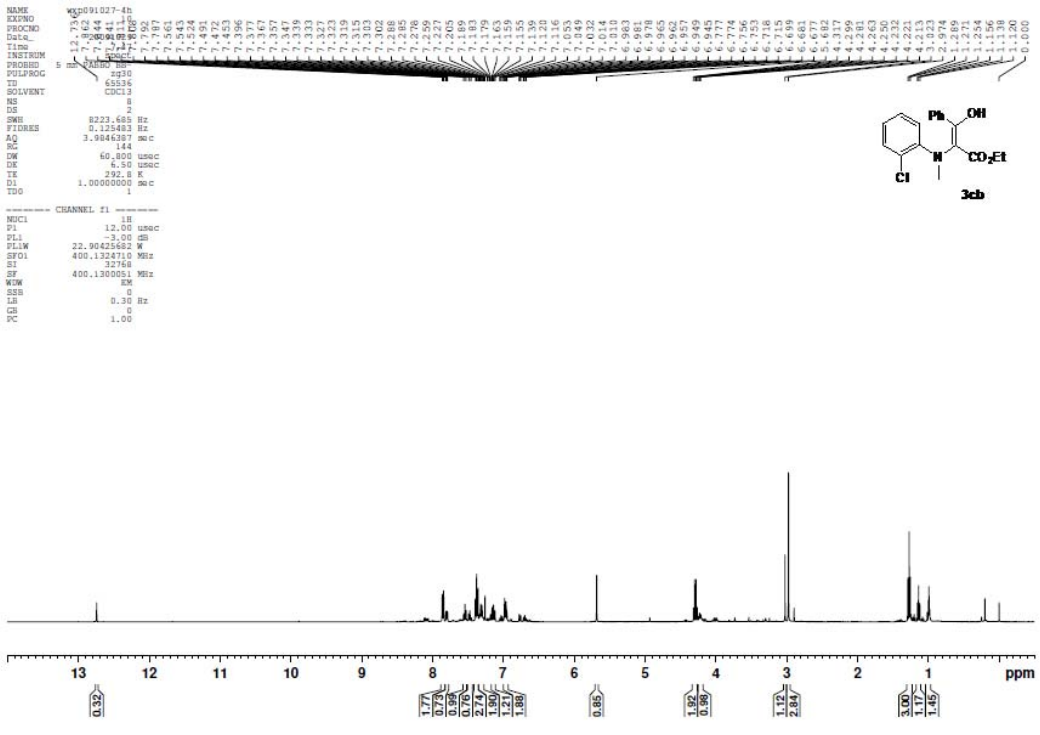
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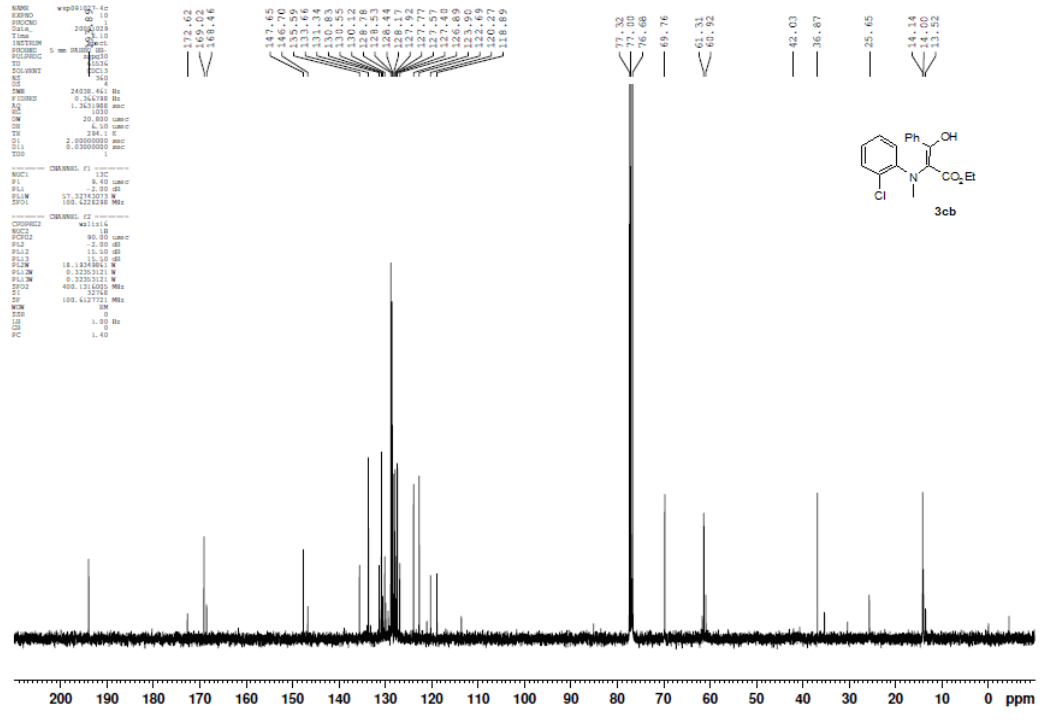
CO2Et
Cl
3cb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Cl
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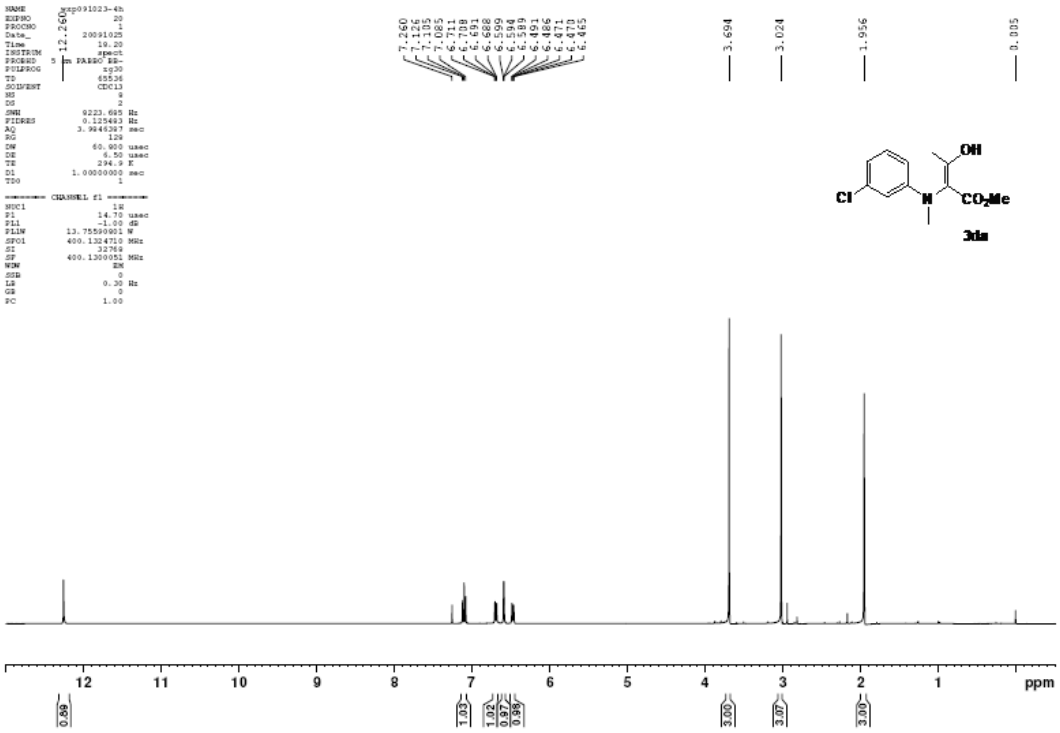
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6.688
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6.594
6.589
6.491
6.486
6.476
6.471
6.470
6.465
3.694
3.024
1.956
0.005
OH
Cl
N
CO2Me
3da
12
11
10
9
8
7
6
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ppm
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1.02
0.97
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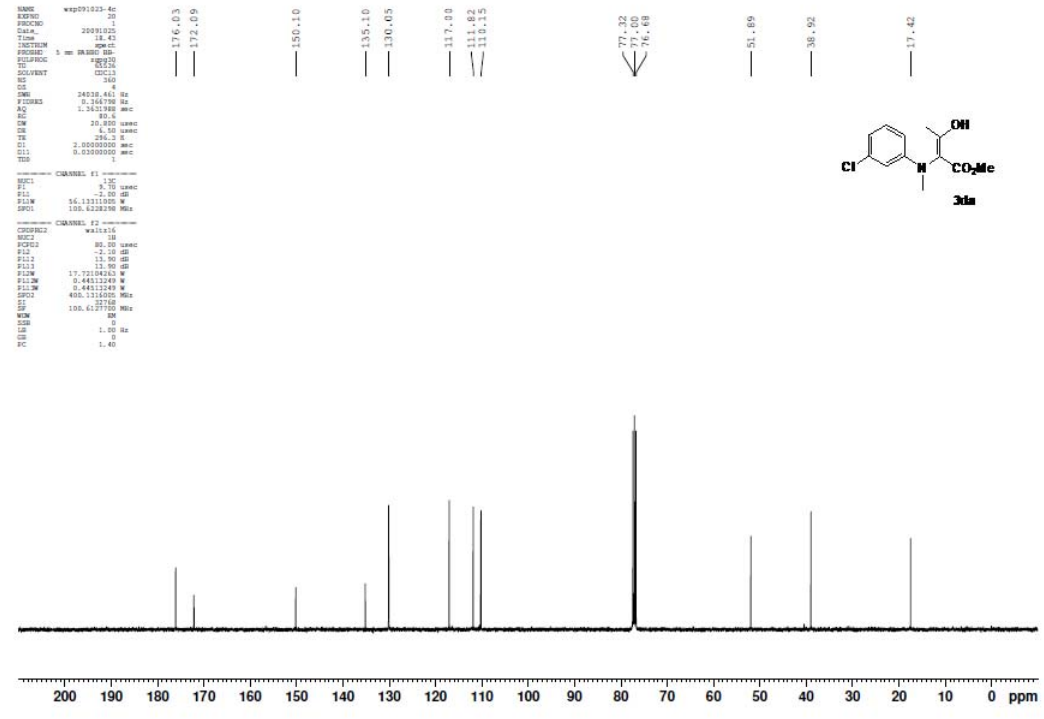
3.00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110.15
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77.00
76.68
51.89
38.92
17.42
OH
Cl
N
CO2Me
3da
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
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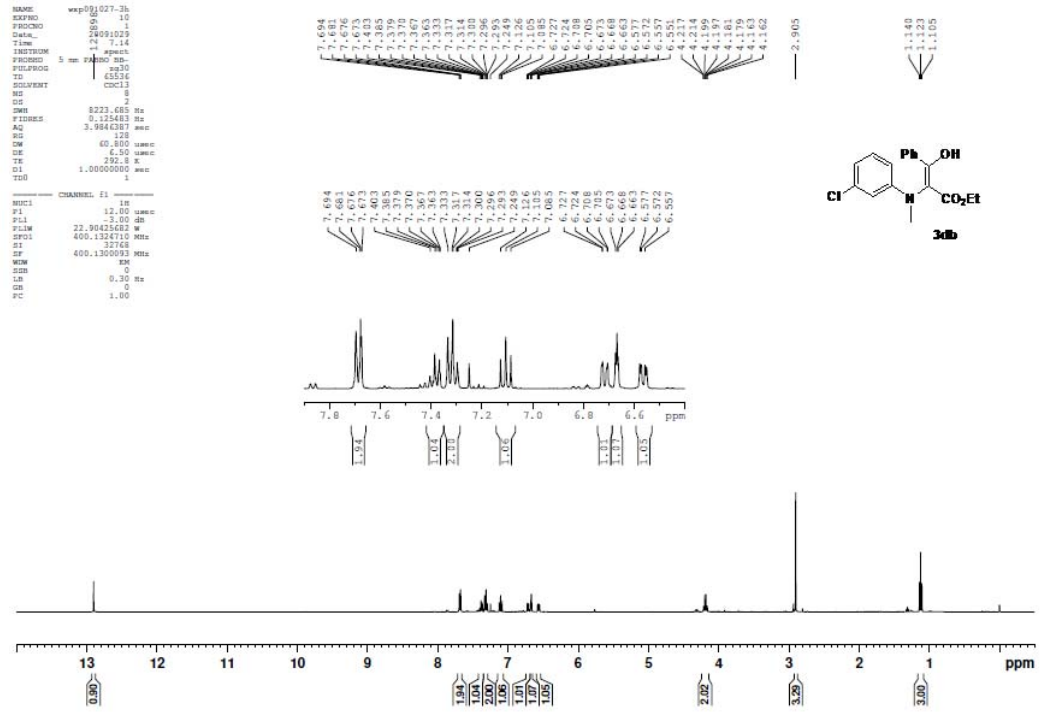
30
20
10
0
ppm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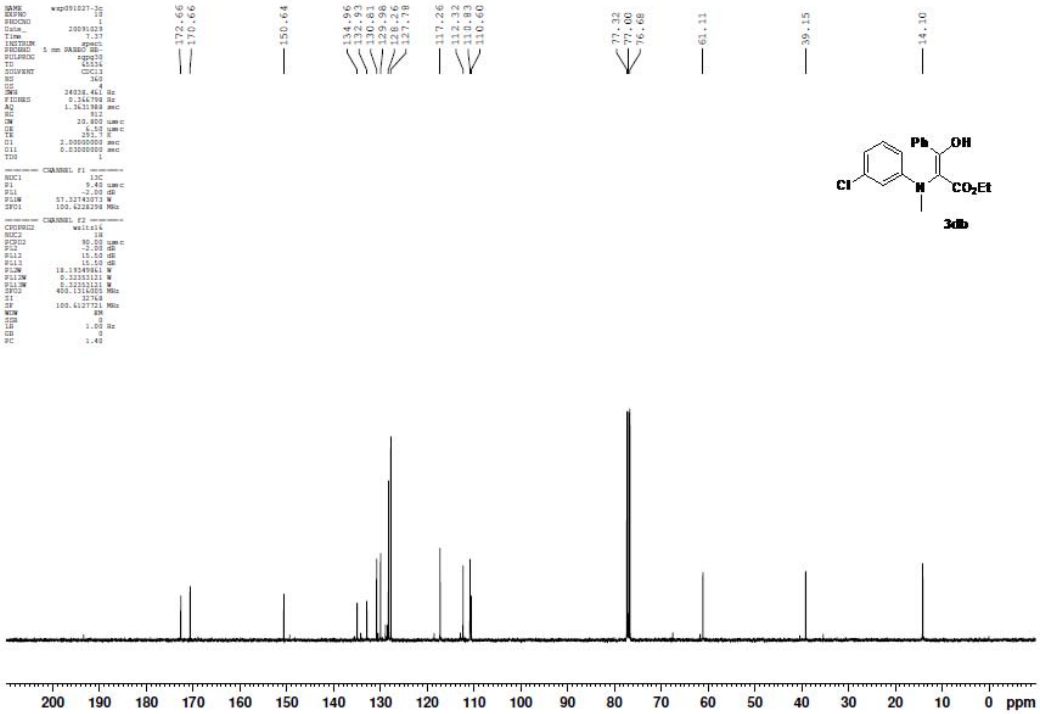
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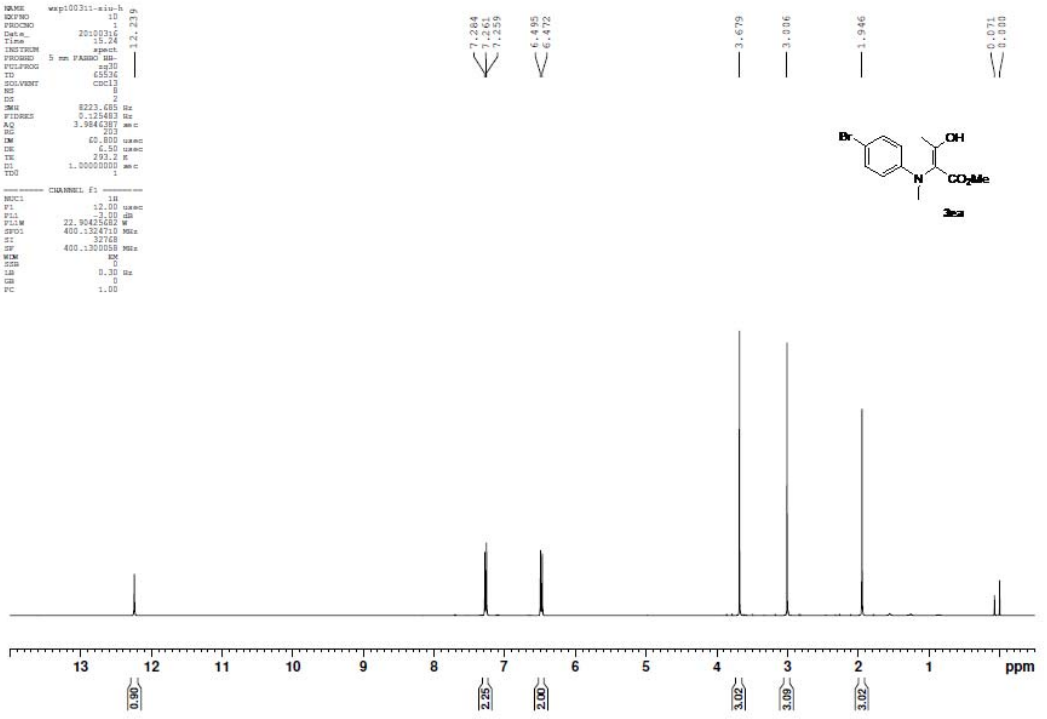
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Br
OH
N
CO2Me
2ea
13
12
11
10
9
8
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6
5
4
3
2
1
ppm
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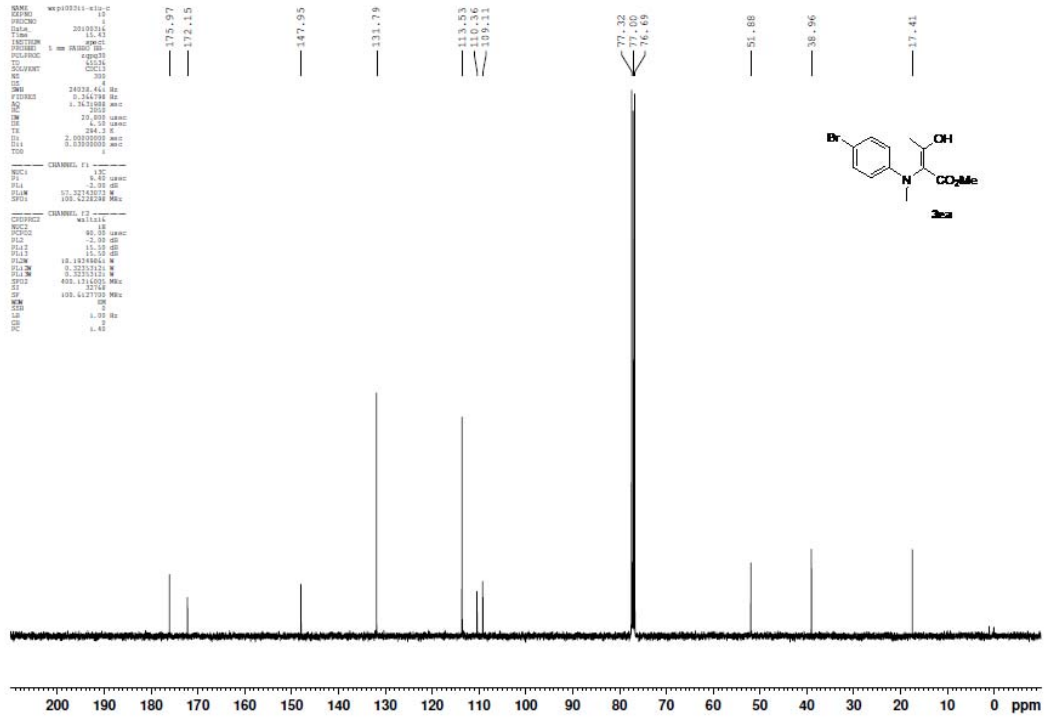
3.02
3.09
3.02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113.53
110.36
109.11
77.32
77.00
76.69
51.88
38.96
17.41
Br
OH
N
CO2Me
2ea
200
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180
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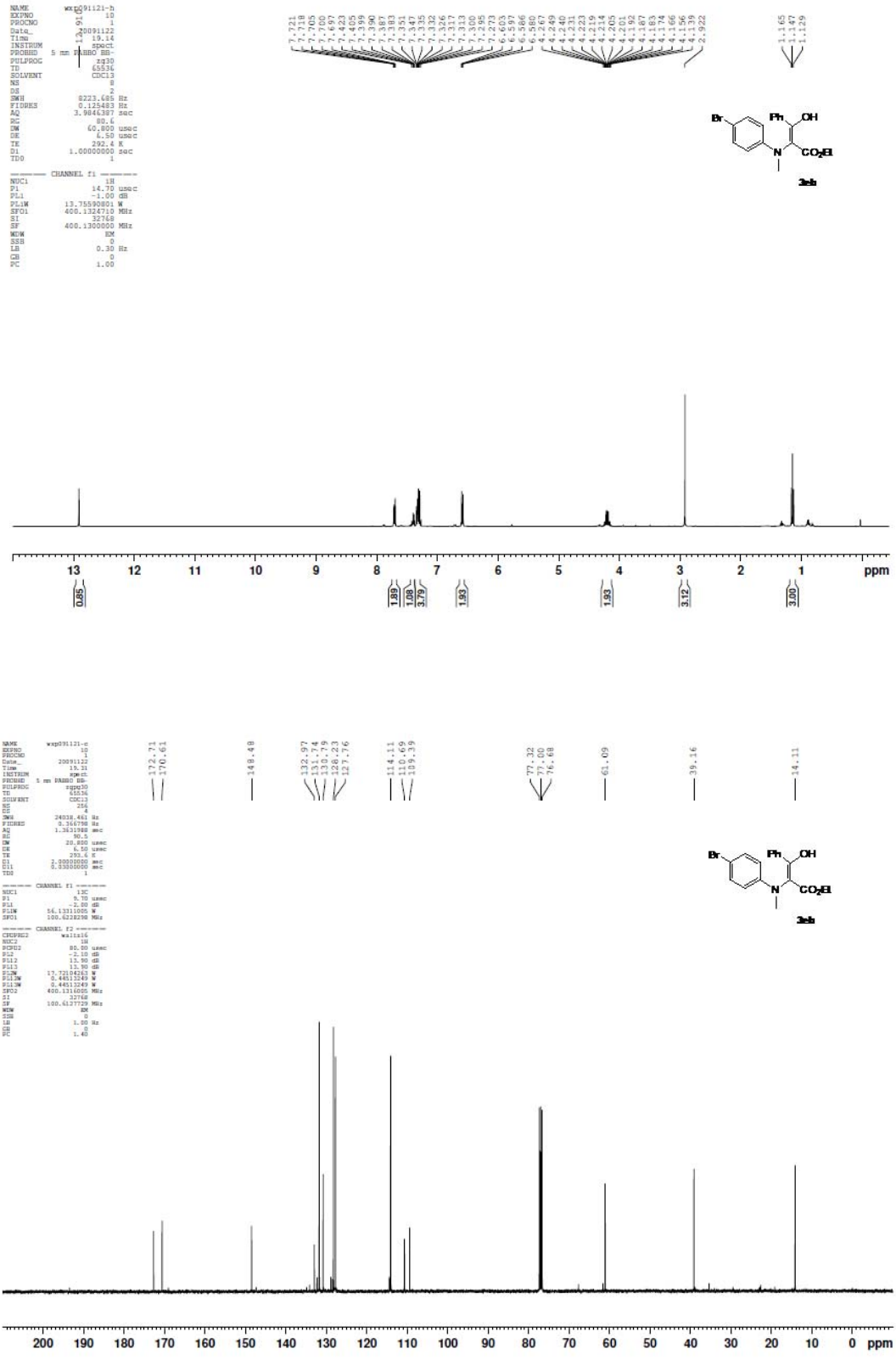
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.399
7.390
7.387
7.383
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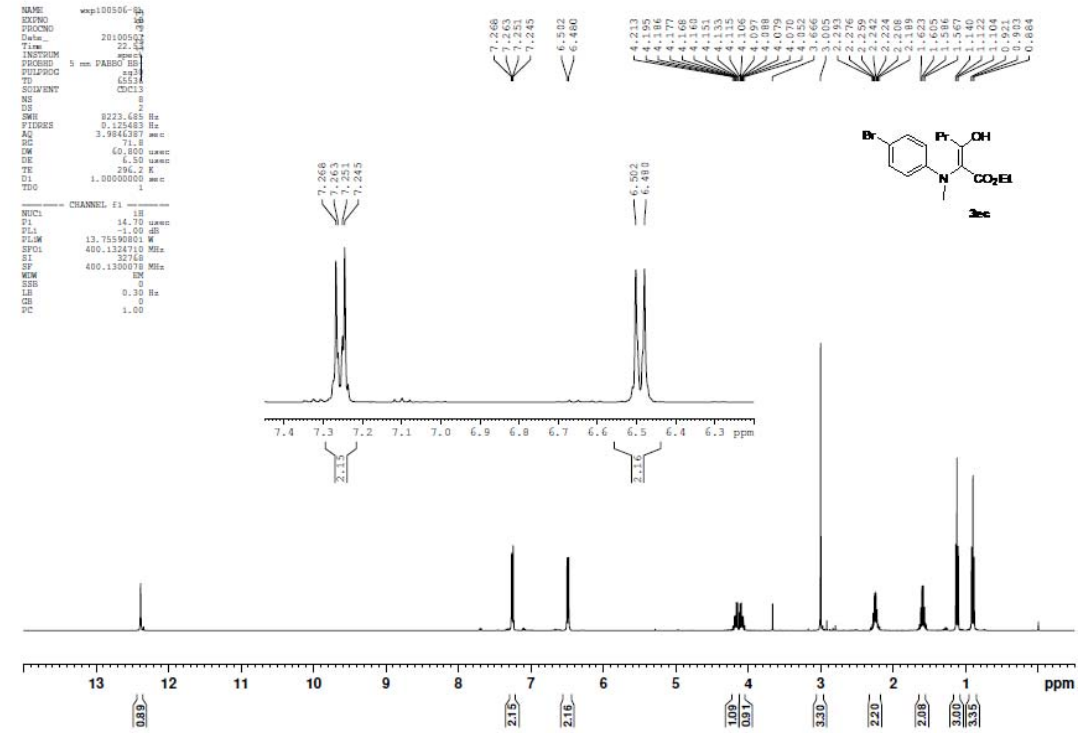
7.341
7.335
7.332
7.326
7.317
7.313
7.310
7.295
7.273
6.603
6.597
6.586
6.580
4.267
4.249
4.240
4.231
4.223
4.214
4.205
4.201
4.192
4.187
4.183
4.174
4.166
4.156
4.139
2.922
1.165
1.147
1.129
Br
Ph
OH
N
CO2Et
3eh
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.85
1.89
1.08
3.79
1.93
1.93
3.12
3.00
172.71
170.61
148.48
132.97
131.74
130.79
128.23
127.76
114.11
110.60
109.39
77.32
77.00
76.68
61.09
39.16
14.11
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm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7.263
7.251
7.245
6.502
6.480
4.213
4.195
4.186
4.177
4.168
4.160
4.151
4.133
4.115
4.106
4.097
4.088
4.079
4.070
4.052
3.666
3.005
2.276
2.259
2.242
2.224
2.208
2.189
1.623
1.605
1.586
1.567
1.140
1.122
1.104
1.021
0.903
0.884
Br
Pr
OH
N
CO2Et
3ec
7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 ppm
2.15
2.16
13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
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2.15
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1.09
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3.30
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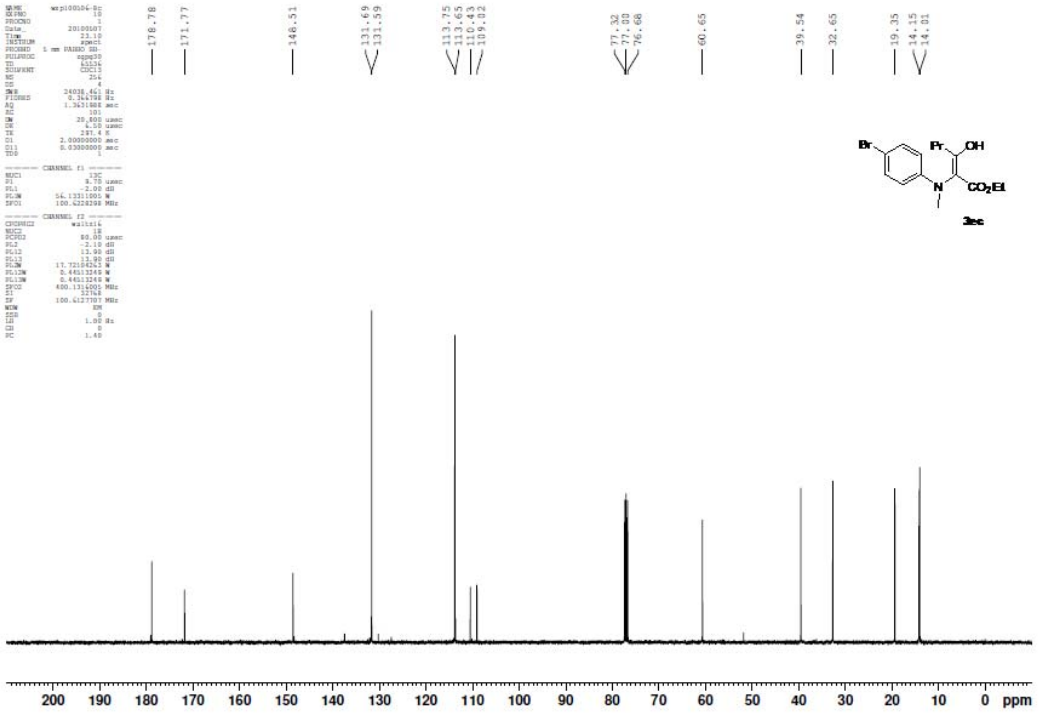
3.00
3.35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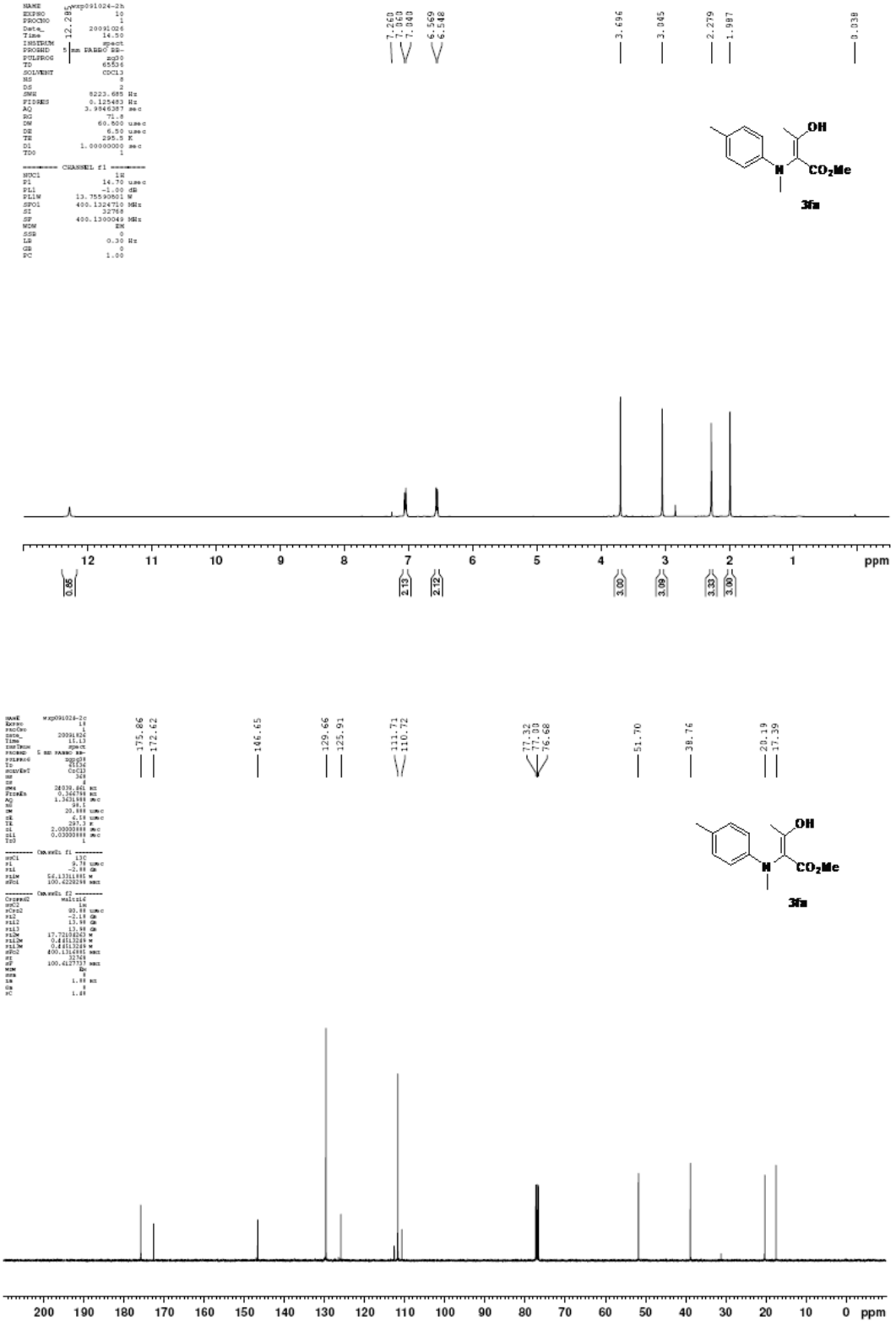
113.75
113.65
110.43
109.02
77.32
77.00
76.68
60.65
39.54
32.65
19.35
14.15
14.01
Br
Pr
OH
N
CO2Et
3ec
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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2.279
1.987
0.038
OH
H
CO2Me
3fa
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.85
2.13
2.12
3.00
3.09
3.33
3.00
175.86
172.62
146.65
129.66
125.91
111.71
110.72
77.32
77.03
76.68
51.70
38.76
20.19
17.39
OH
H
CO2Me
3fa
200
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180
170
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150
140
130
120
110
100
90
80
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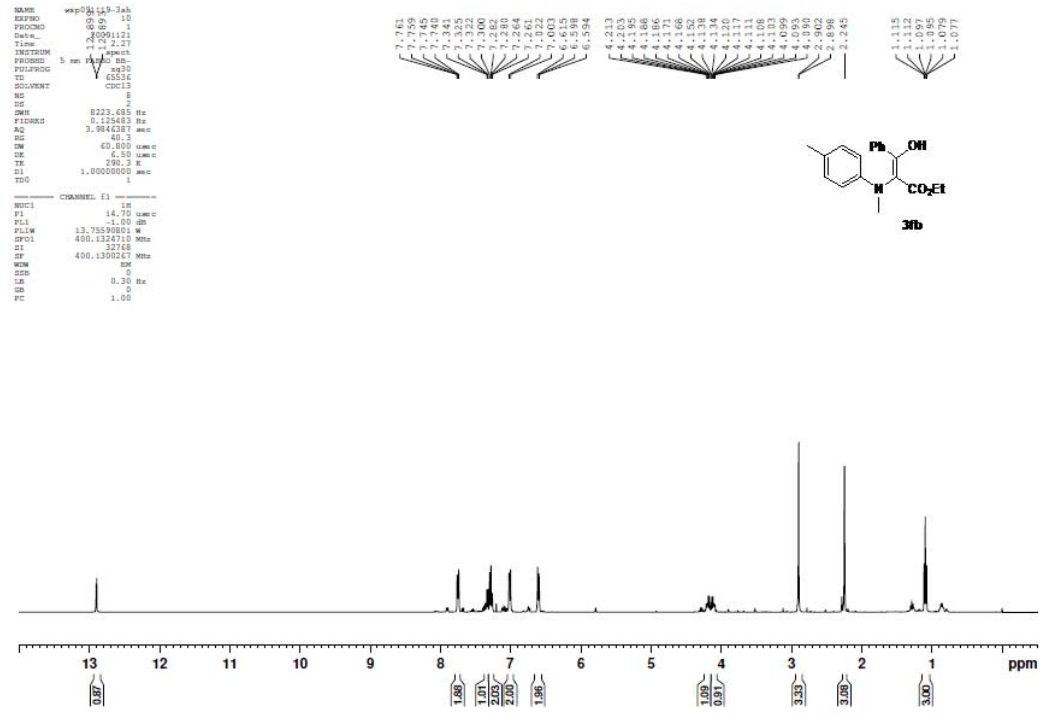
60
50
40
30
20
10
0
ppm


Ph
OH
N
CO2Et
3fb
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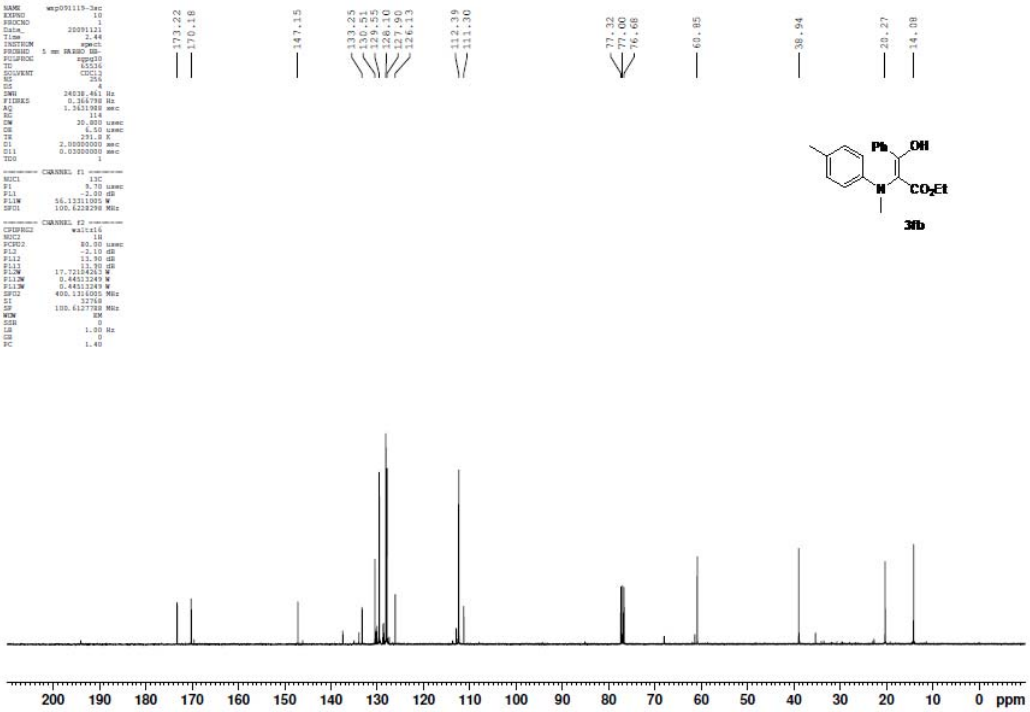
12
11
10
9
8
7
6
5
4
3
2
1
ppm


Ph
OH
N
CO2Et
3fb
200
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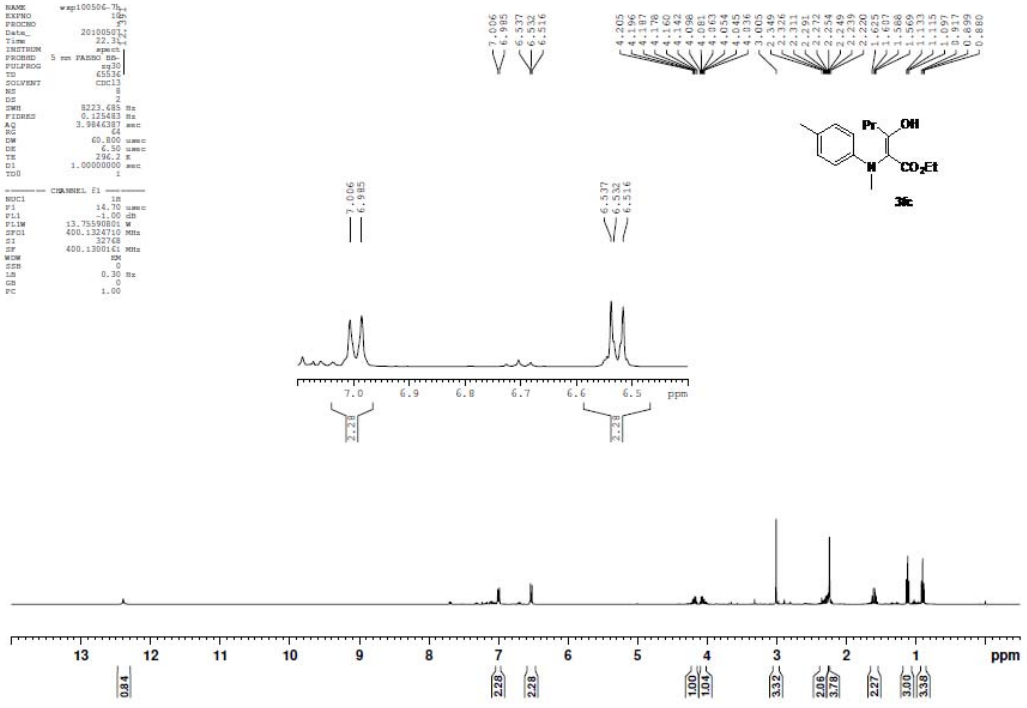
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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4.196
4.187
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4.160
4.142
4.096
4.081
4.063
4.054
4.045
4.036
3.005
2.349
2.336
2.311
2.291
2.272
2.254
2.349
2.239
2.220
1.625
1.607
1.568
1.569
1.533
1.115
1.097
0.917
0.899
0.880
Pr
OH
N
CO2Et
36c
7.0
6.9
6.8
6.7
6.6
6.5
ppm
2.28
2.28
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
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2.06
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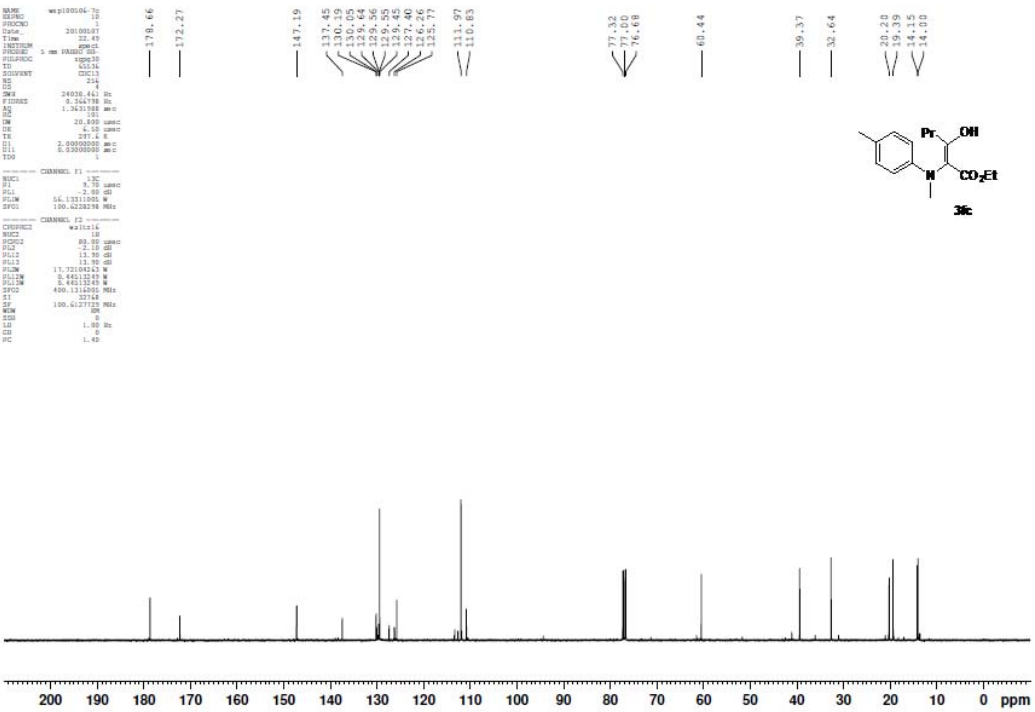
2.27
3.00
3.38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129.45
127.40
126.26
125.77
111.97
110.83
77.32
77.00
76.68
68.44
39.37
32.64
28.28
19.39
14.15
14.00
Pr
OH
N
CO2Et
36c
200
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130
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20
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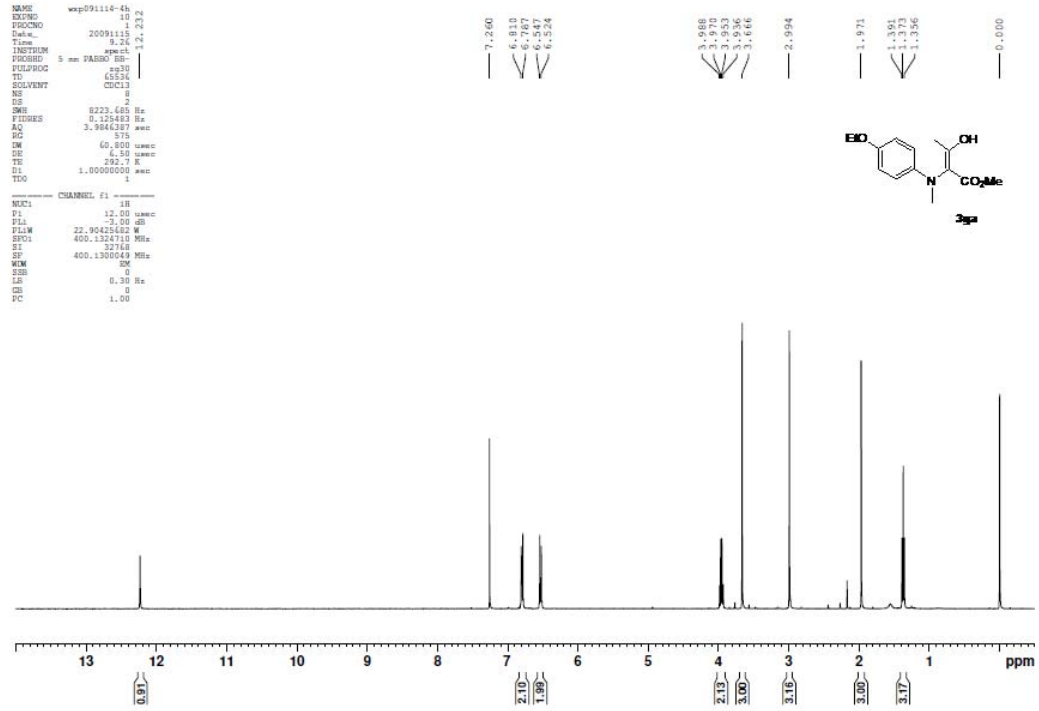
ppm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3.953
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1.971
1.391
1.373
1.356
0.000
EtO
OH
N
CO2Me
3ga
13
12
11
10
9
8
7
6
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4
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2
1
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2.13
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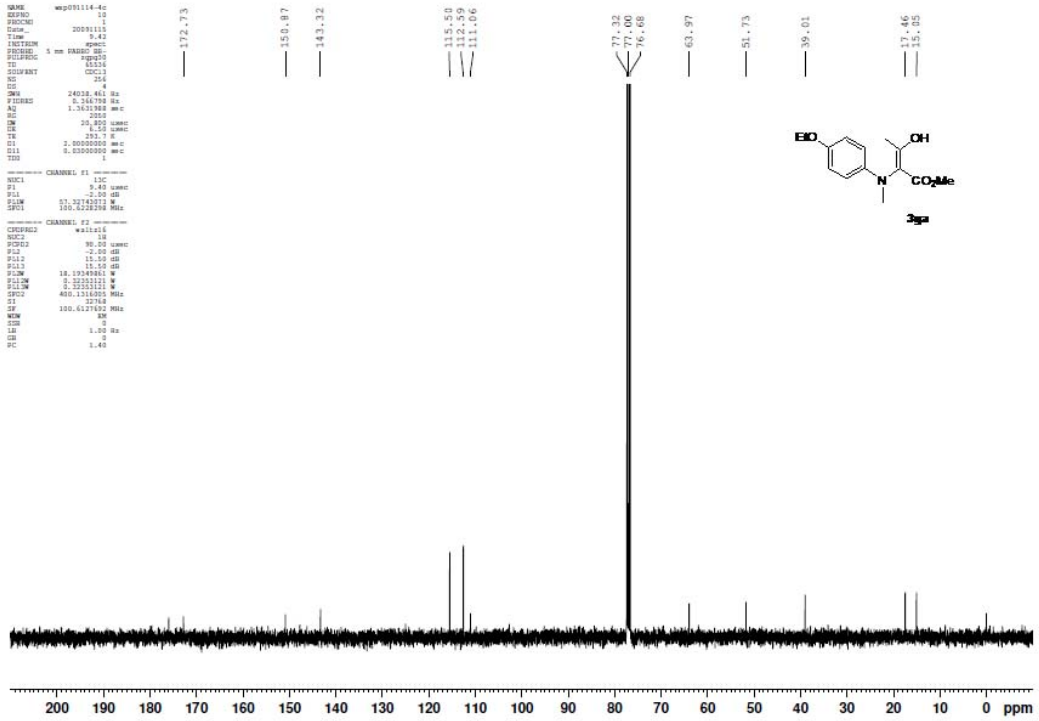
3.16
3.00
3.17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111.06
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77.00
76.68
63.97
51.73
39.01
17.46
15.05
EtO
OH
N
CO2Me
3ga
200
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140
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100
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60
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20
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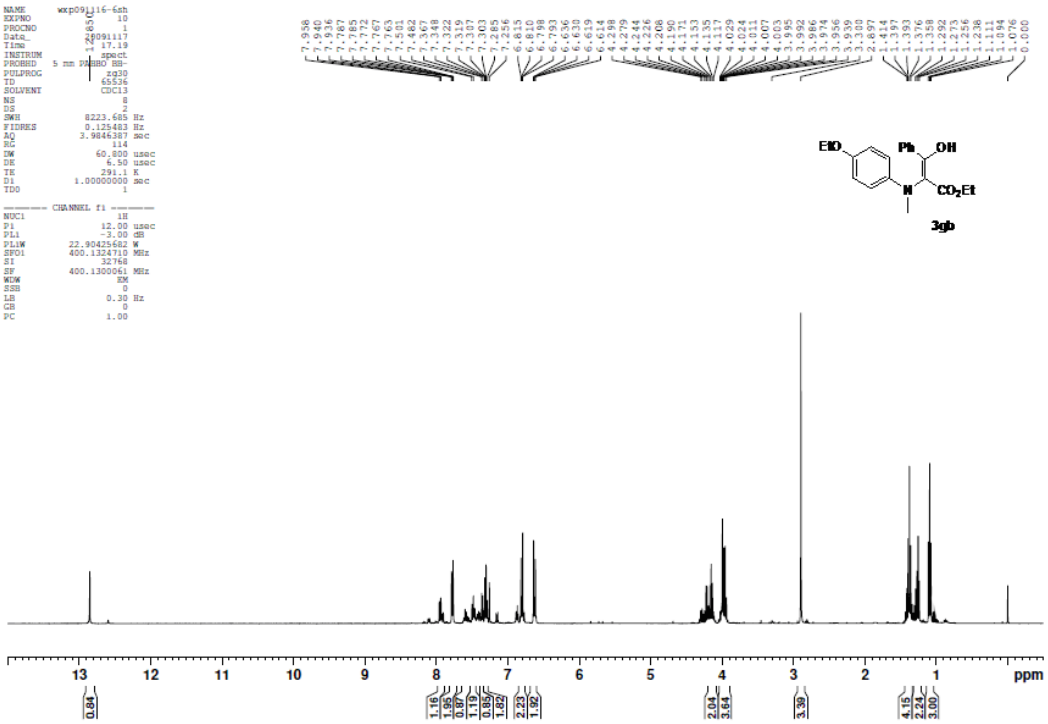
0
ppm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Ph
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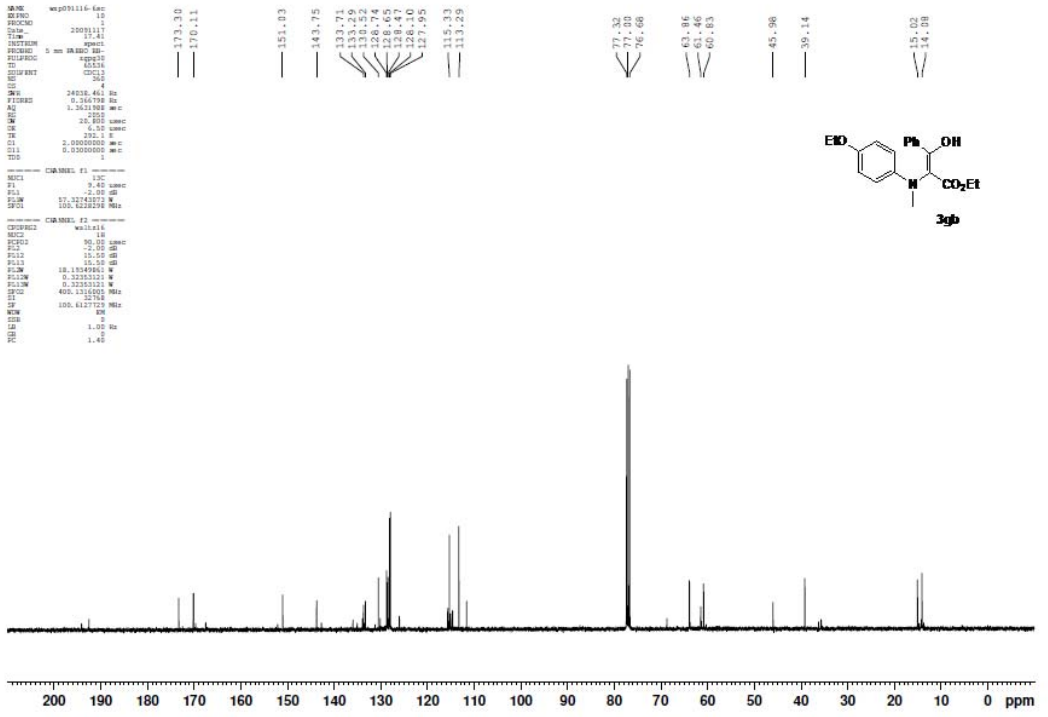
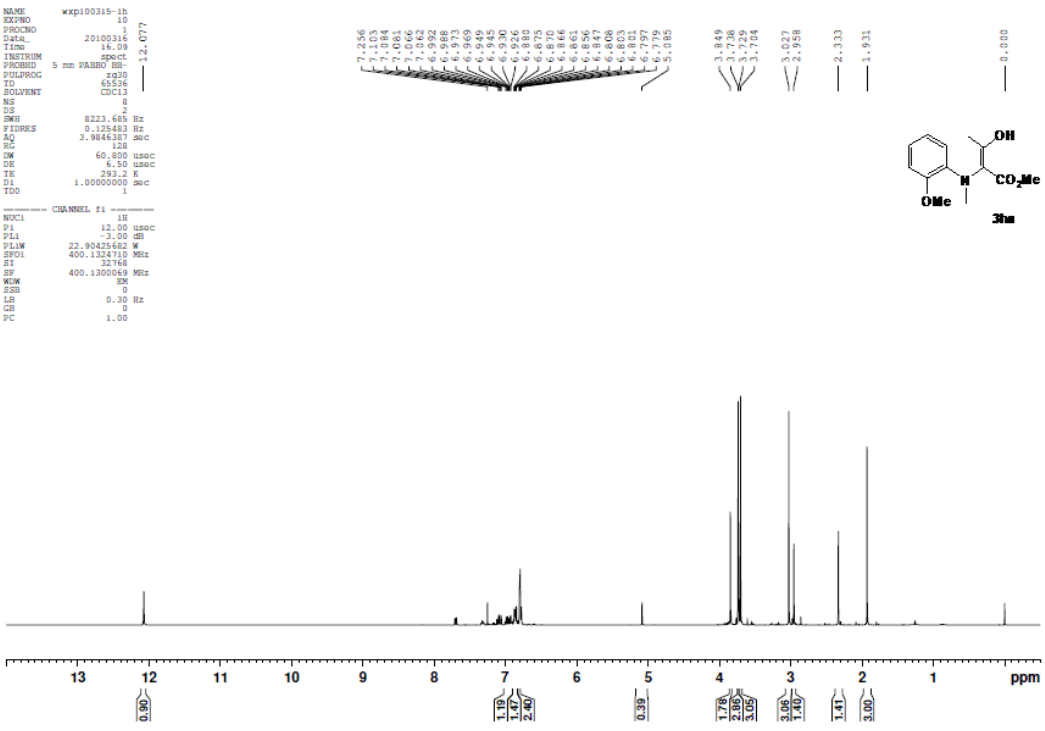
CO2Et
3gb
ppm


EtO
Ph
OH
N
CO2Et
3gb
ppm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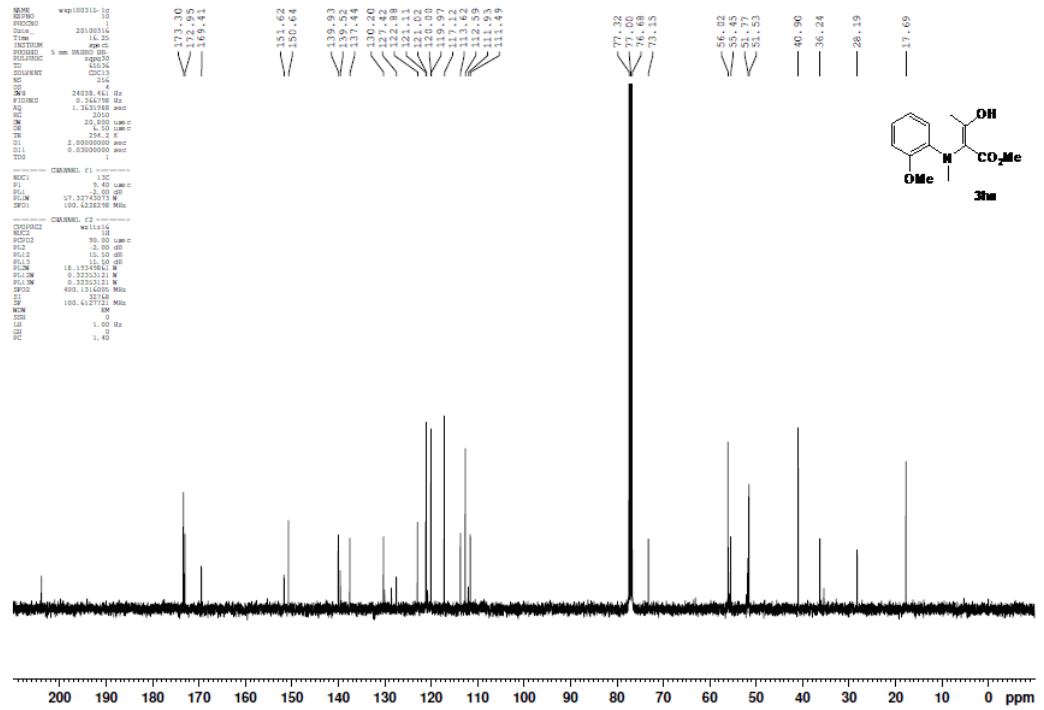
N
OMe
3hm
ppm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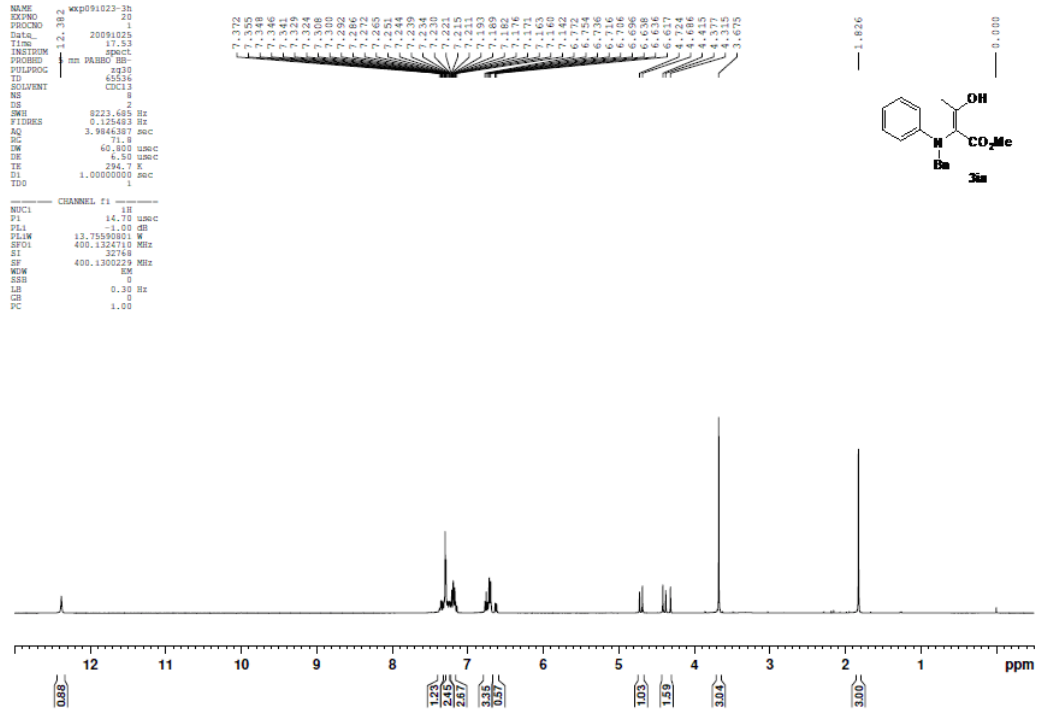
CO2Me
N
OMe
3hm
ppm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EXPNO 20
PROCNO 1
Date_ 20091025
Time 18.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 71.8
DW 60.800 usec
DE 6.50 usec
TE 294.7 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.70 usec
PL1 -1.00 dB
PL1W 13.75590801 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300229 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.372
7.355
7.348
7.346
7.341
7.329
7.324
7.308
7.300
7.292
7.286
7.272
7.265
7.251
7.244
7.239
7.234
7.230
7.221
7.215
7.211
7.193
7.169
7.162
7.176
7.171
7.163
7.160
7.142
6.772
6.754
6.736
6.716
6.706
6.696
6.638
6.636
6.617
4.724
4.686
4.415
4.377
4.345
3.675
1.826
0.000
OH
CO2Me
N
Bn
3kn
12
11
10
9
8
7
6
5
4
3
2
1
ppm
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2.87
3.35
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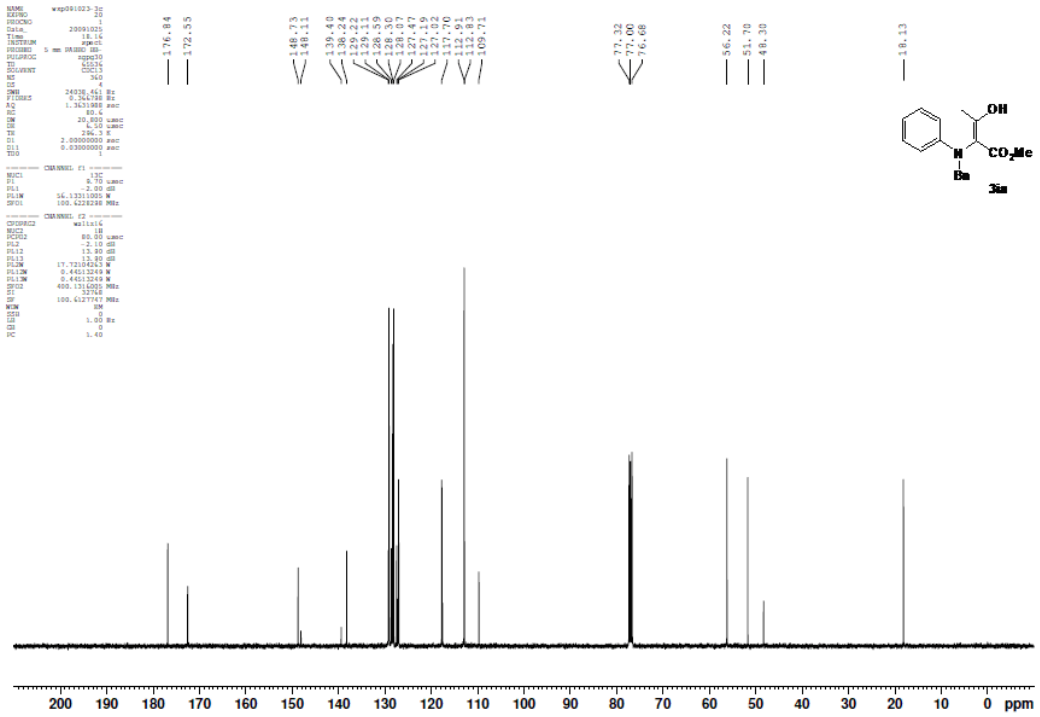
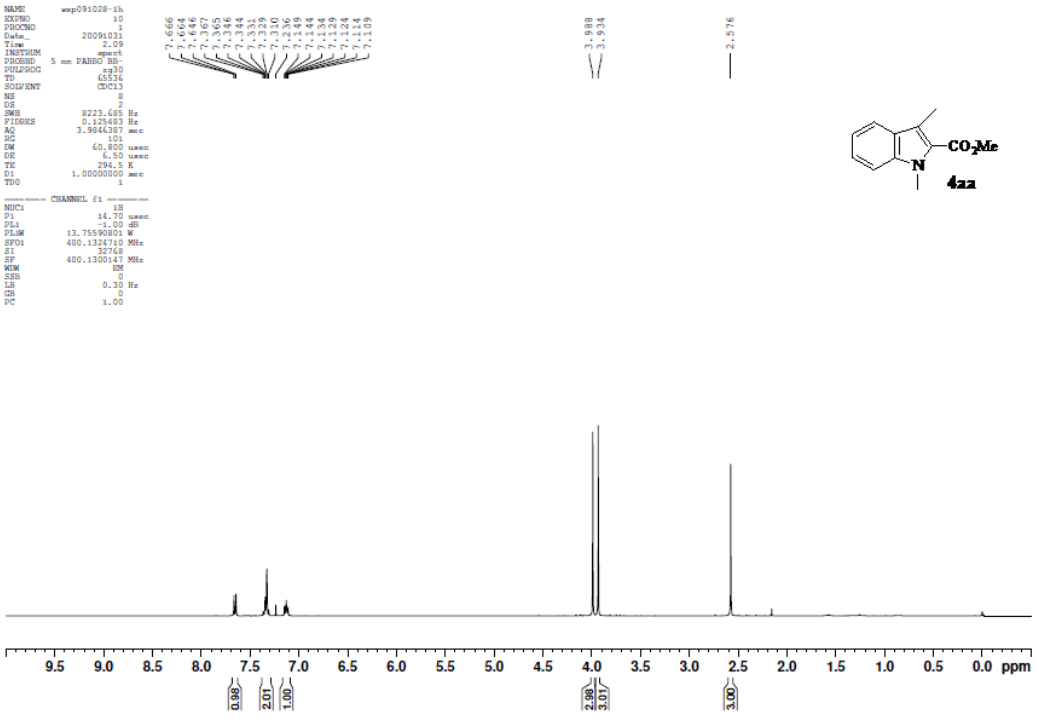
1.59
3.04
3.00


NAME wxp091023-3c
EXPNO 20
PROCNO 1
Date_ 20091025
Time 18.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 340
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631986 sec
RG 80.6
DW 20.800 usec
DE 6.50 usec
TE 296.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.70 usec
PL1 -2.00 dB
PL1W 56.13311005 W
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.10 dB
PL12 13.90 dB
PL13 20.00 dB
PL1W 17.72104332 W
PL12W 0.44513249 W
PL13W 0.44513249 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127747 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
176.84
172.55
148.73
148.11
139.40
138.24
129.22
129.11
128.59
128.50
128.07
127.47
127.19
127.02
117.00
112.91
112.83
109.71
77.32
77.00
76.68
56.22
51.70
48.30
18.13
OH
CO2Me
N
Bn
3kn
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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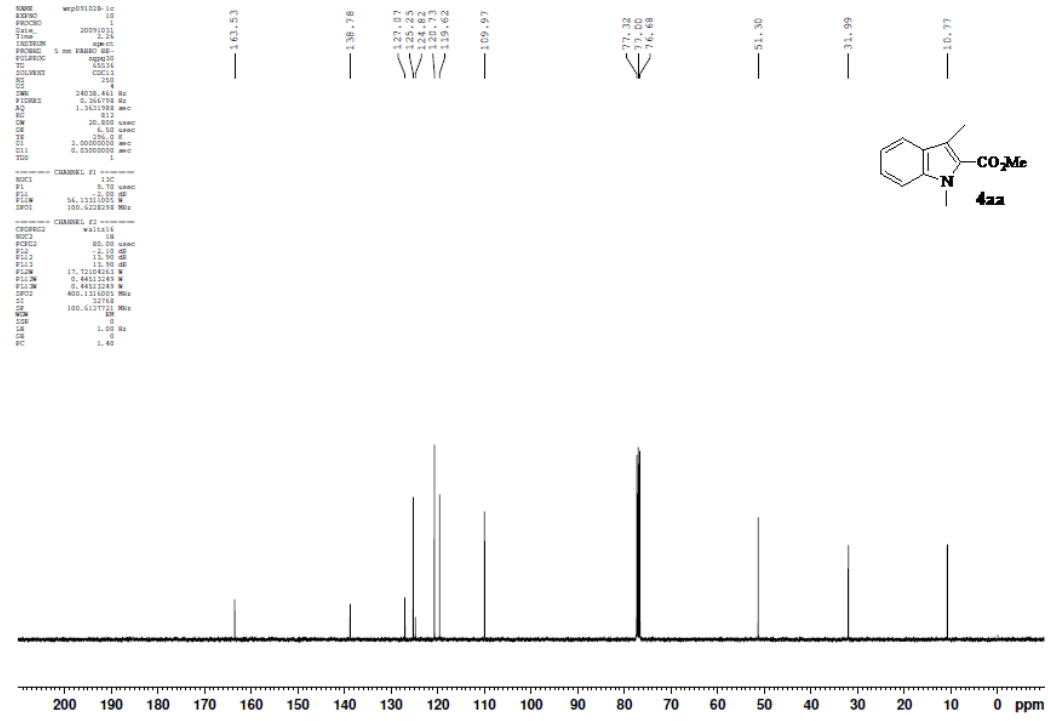
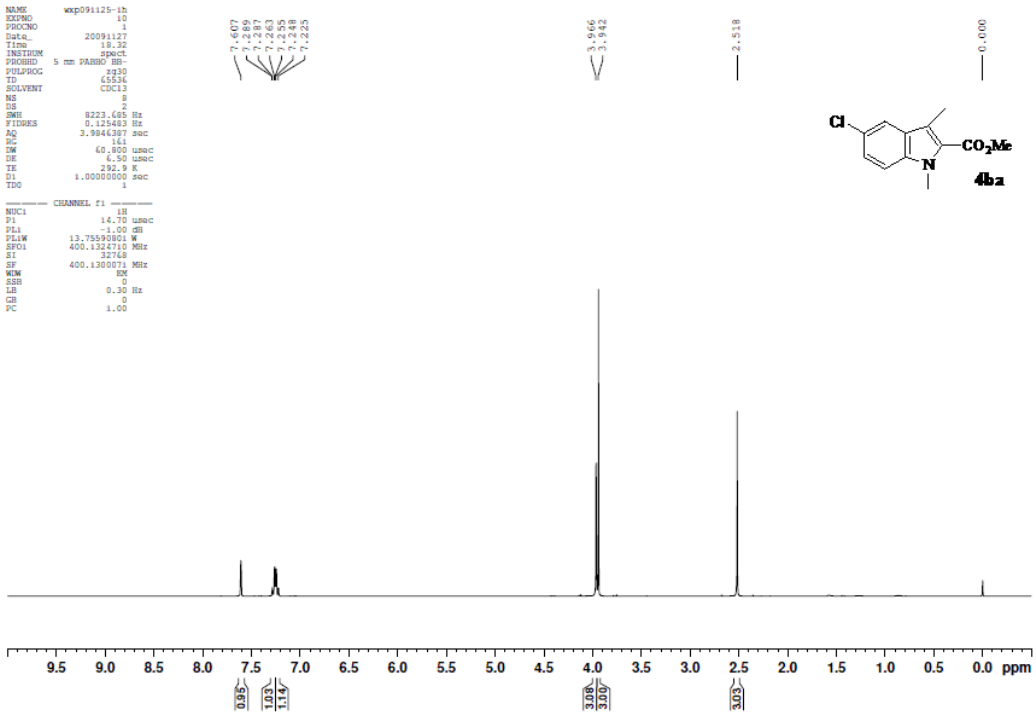
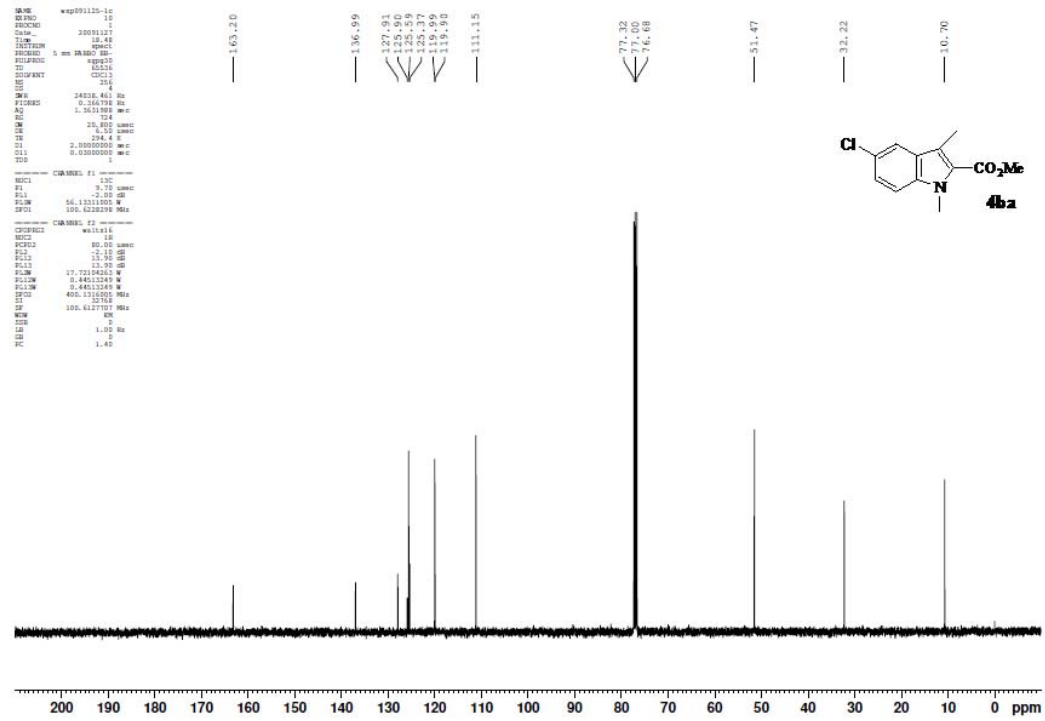
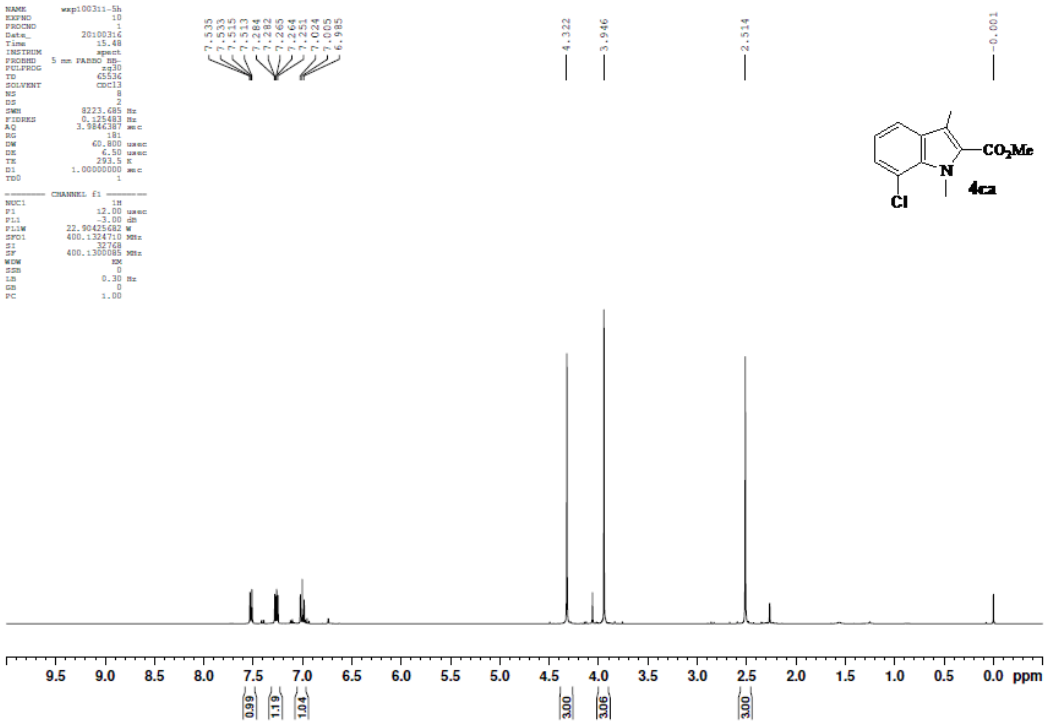
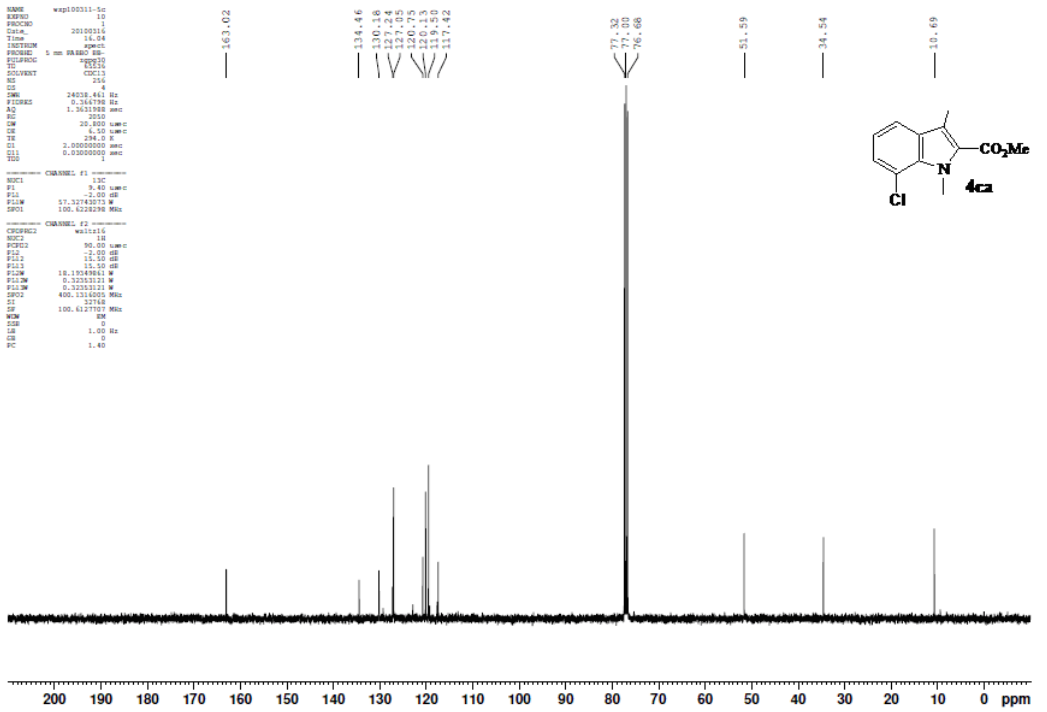
7.134
7.129
7.124
7.114
7.109
3.988
3.934
2.576
CO2Me
N
4aa
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.98
2.01
1.00
2.98
3.01
3.00

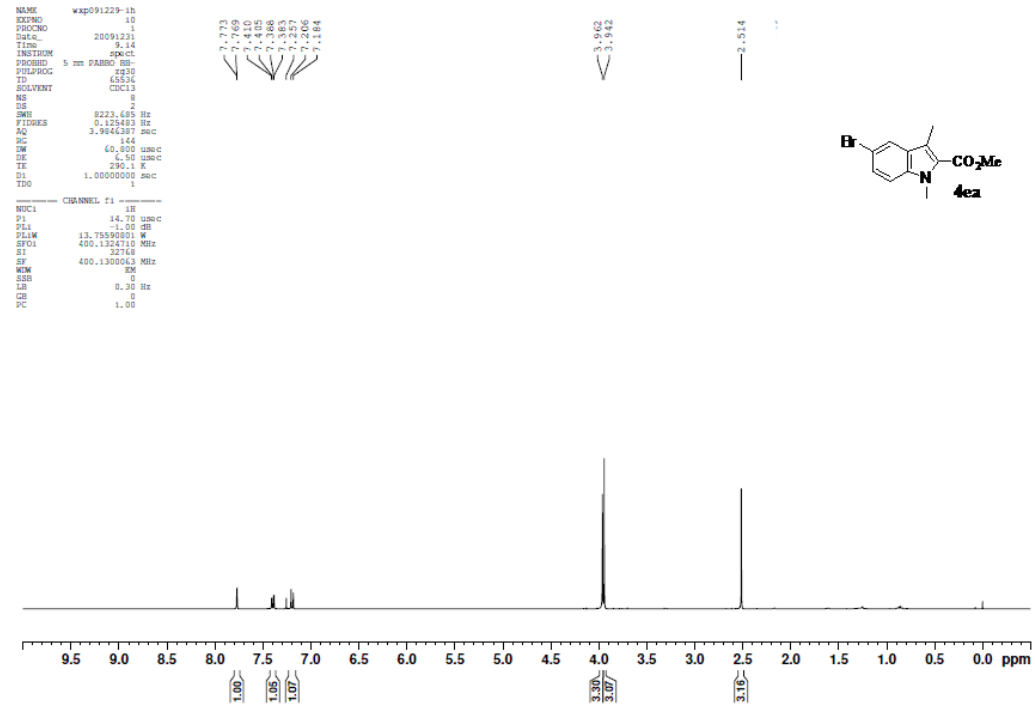

163.53
136.78
127.87
127.25
124.82
120.73
119.62
109.97
77.32
77.00
76.68
51.30
31.99
10.77
CO2Me
N
4aa
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

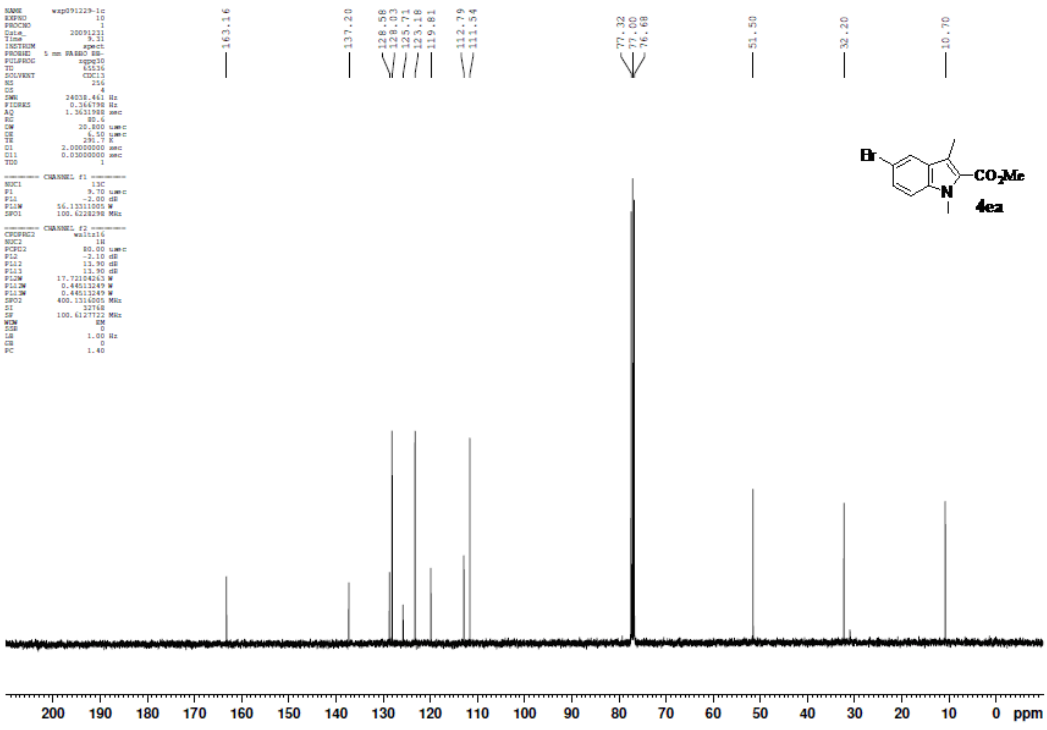

7.607
7.289
7.287
7.263
7.255
7.248
7.225
3.966
3.942
2.518
0.000
Cl
CO2Me
N
4ba
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.95
1.03
1.14
3.08
3.00
3.03

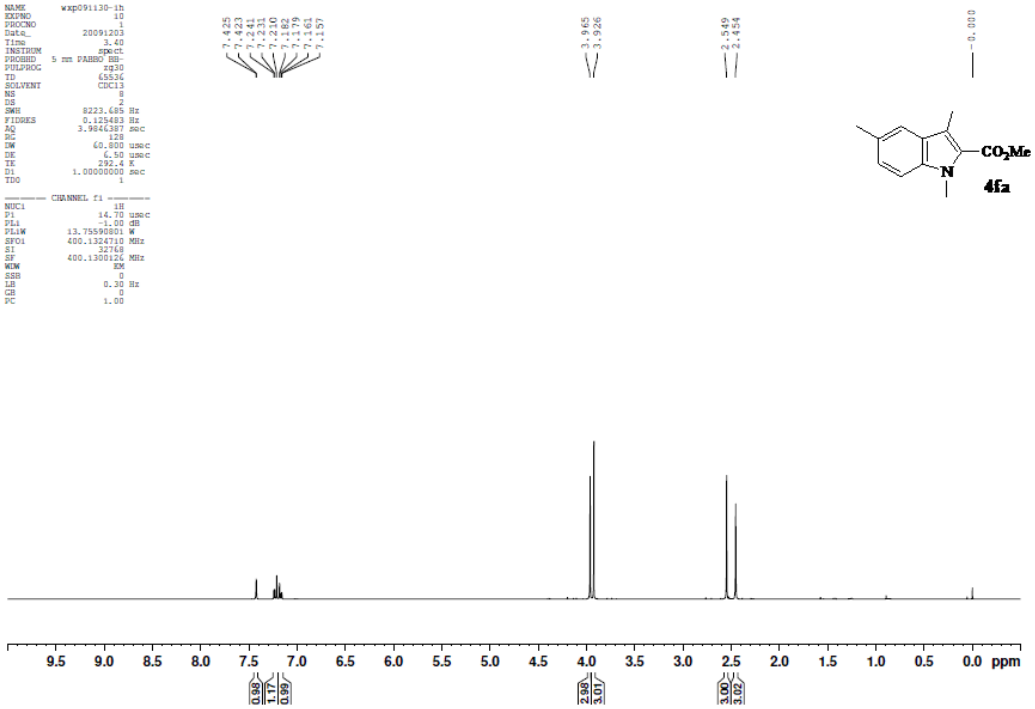

163.20
136.99
127.91
125.90
125.59
125.37
119.99
119.90
111.15
77.32
77.00
76.68
51.47
32.22
10.70
Cl
CO2Me
N
4ba
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

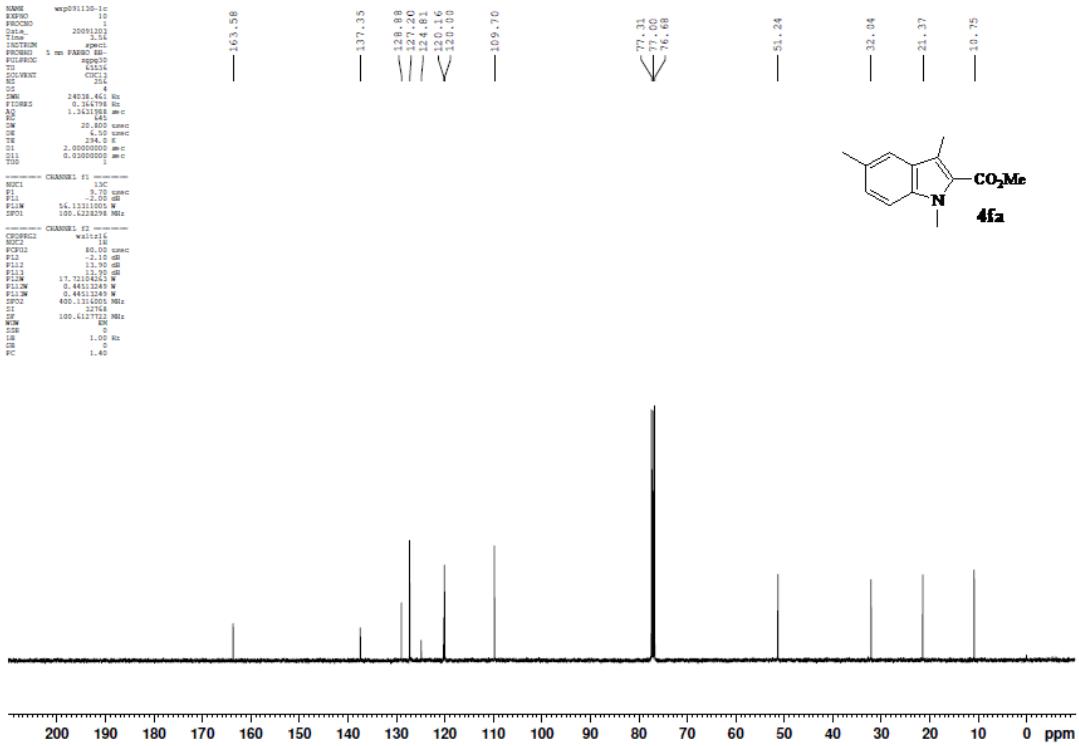

7.535
7.533
7.515
7.513
7.284
7.282
7.265
7.264
7.251
7.084
7.065
6.985
4.322
3.946
2.514
0.001
CO2Me
N
Cl
4ca
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.99
1.19
1.04
3.00
3.06
3.00

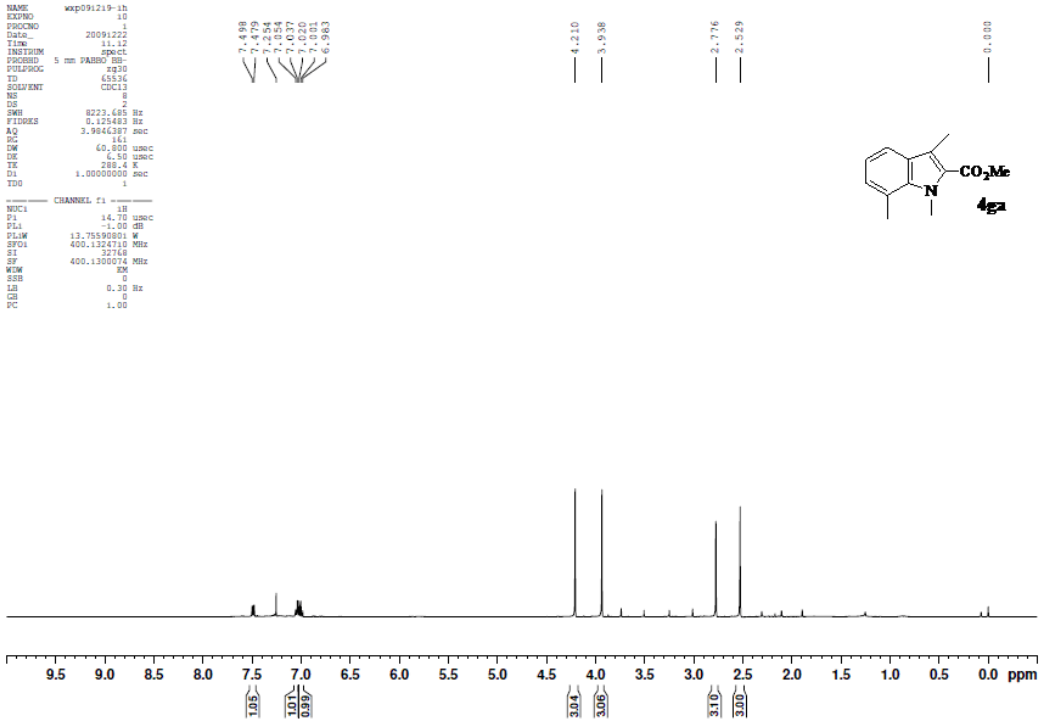

163.02
134.46
130.16
127.24
123.45
120.75
120.13
119.50
117.42
77.32
77.00
76.68
51.59
34.54
18.69
CO2Me
N
Cl
4ca
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

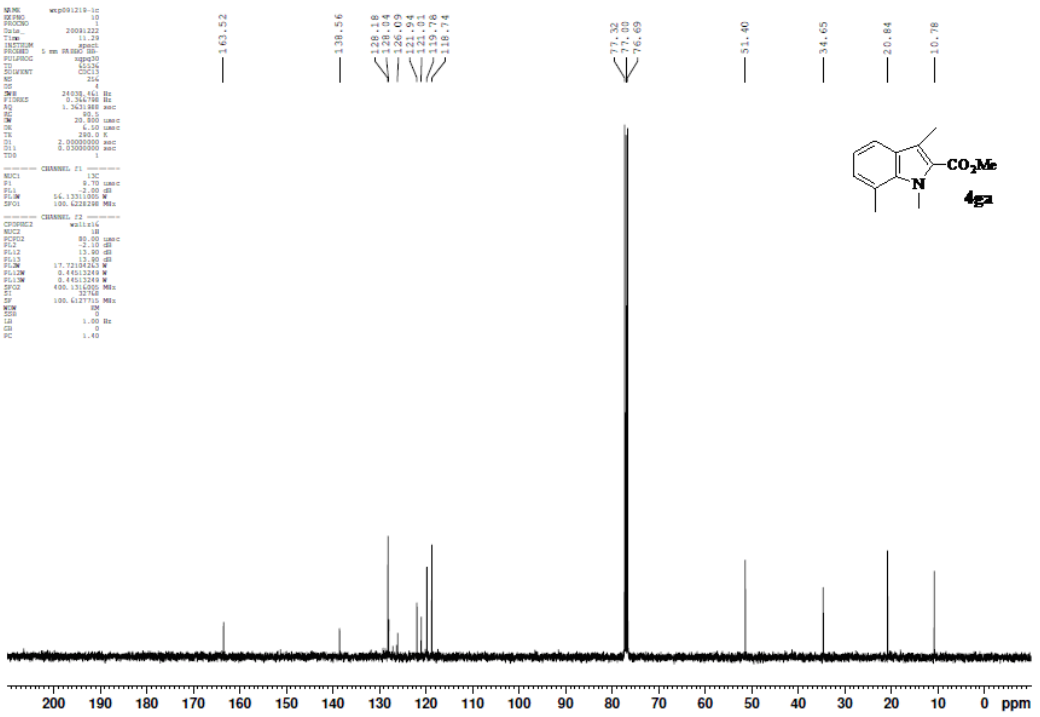

7.773
7.769
7.410
7.405
7.386
7.383
7.257
7.206
7.184
3.962
3.942
2.514
Br
CO2Me
N
4ea
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
1.05
1.07
3.30
3.07
3.16

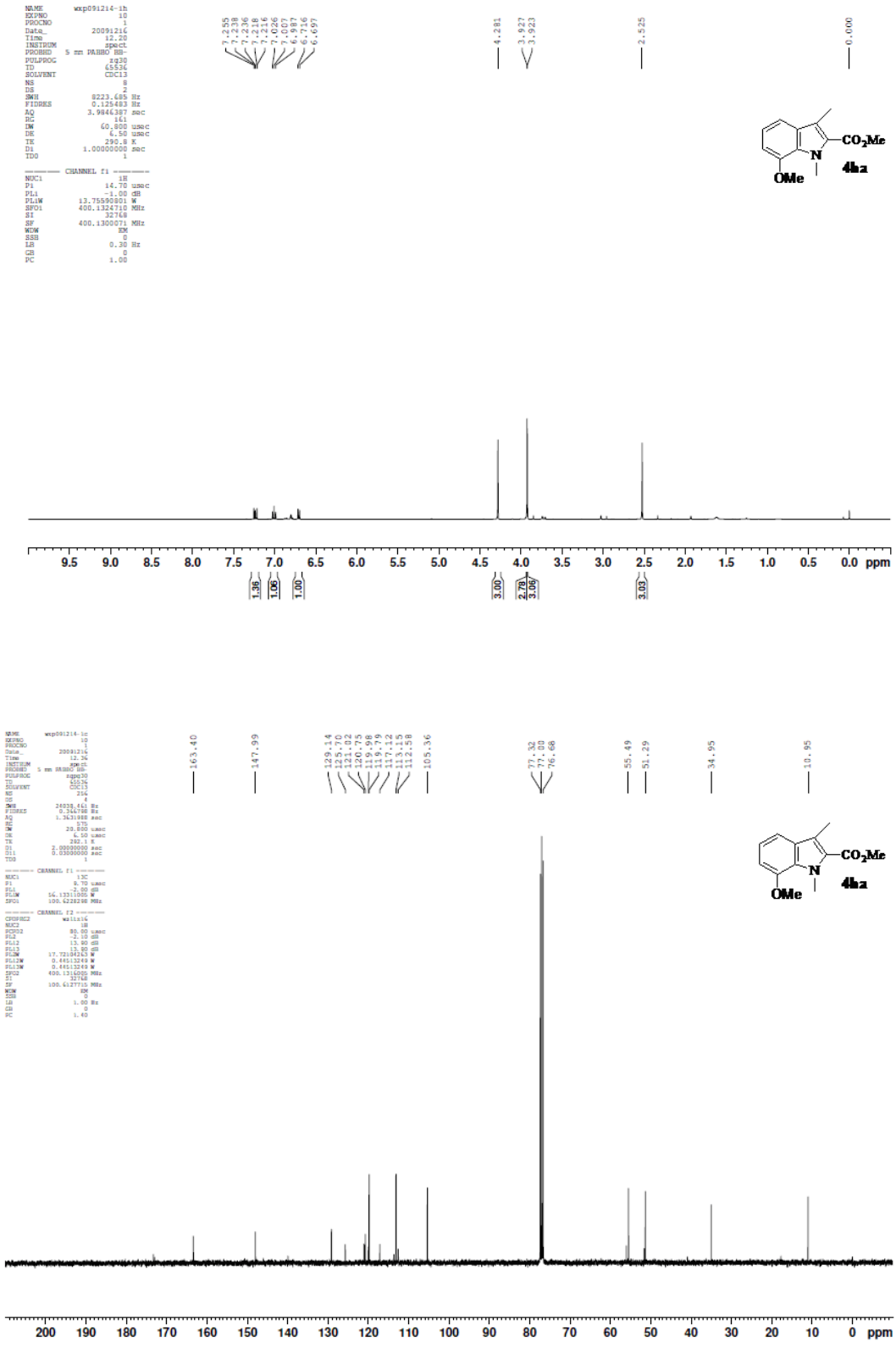

163.16
137.20
128.58
128.03
125.71
123.18
119.81
112.79
111.54
77.32
77.00
76.68
51.50
32.20
10.70
Br
CO2Me
N
4ea
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

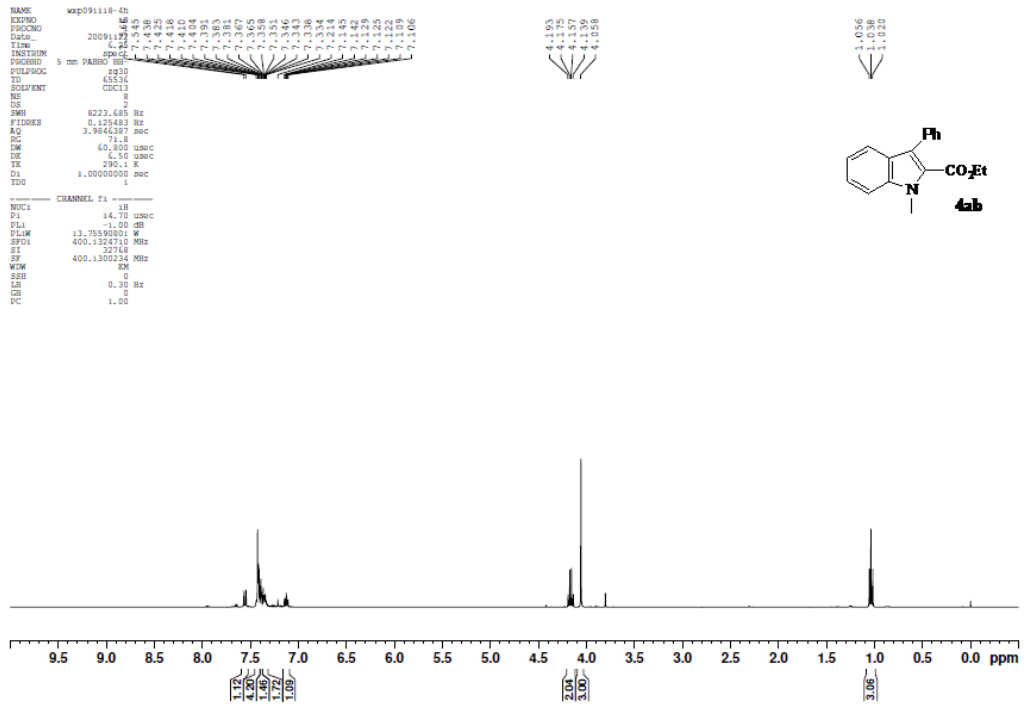

7.425
7.423
7.241
7.231
7.210
7.162
7.159
7.161
7.157
3.965
3.926
2.549
2.454
0.000
CO2Me
N
4fa
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.98
1.17
0.99
2.98
3.01
3.00
3.02

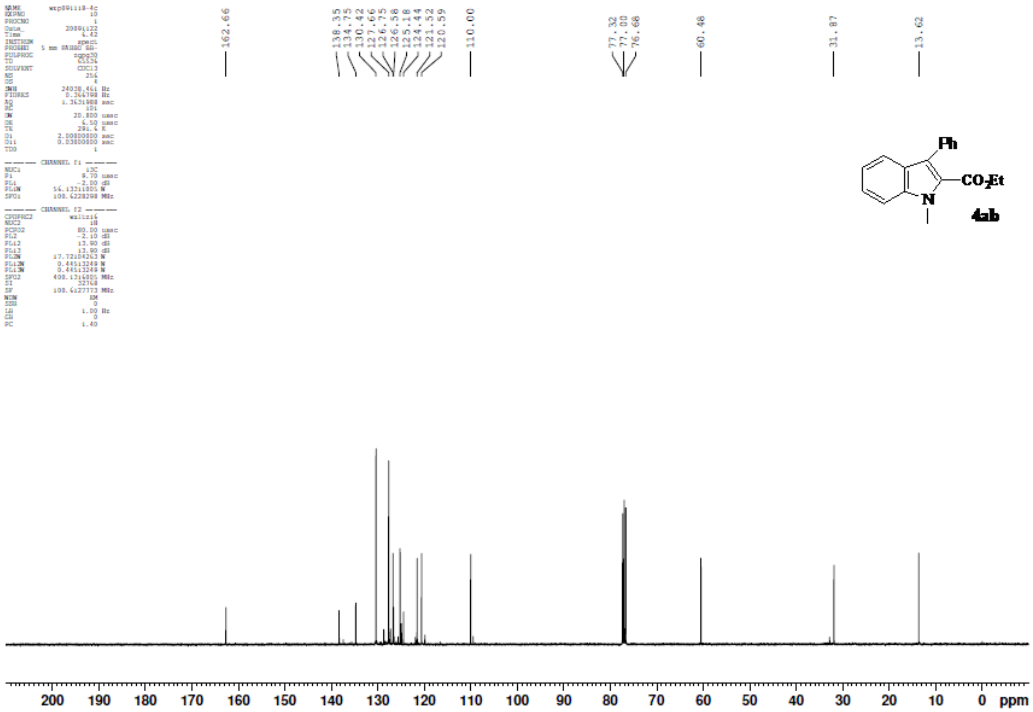

163.58
137.35
129.88
127.20
124.81
120.16
120.00
109.70
77.31
77.00
76.68
51.24
32.04
21.37
10.75
CO2Me
N
4fa
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

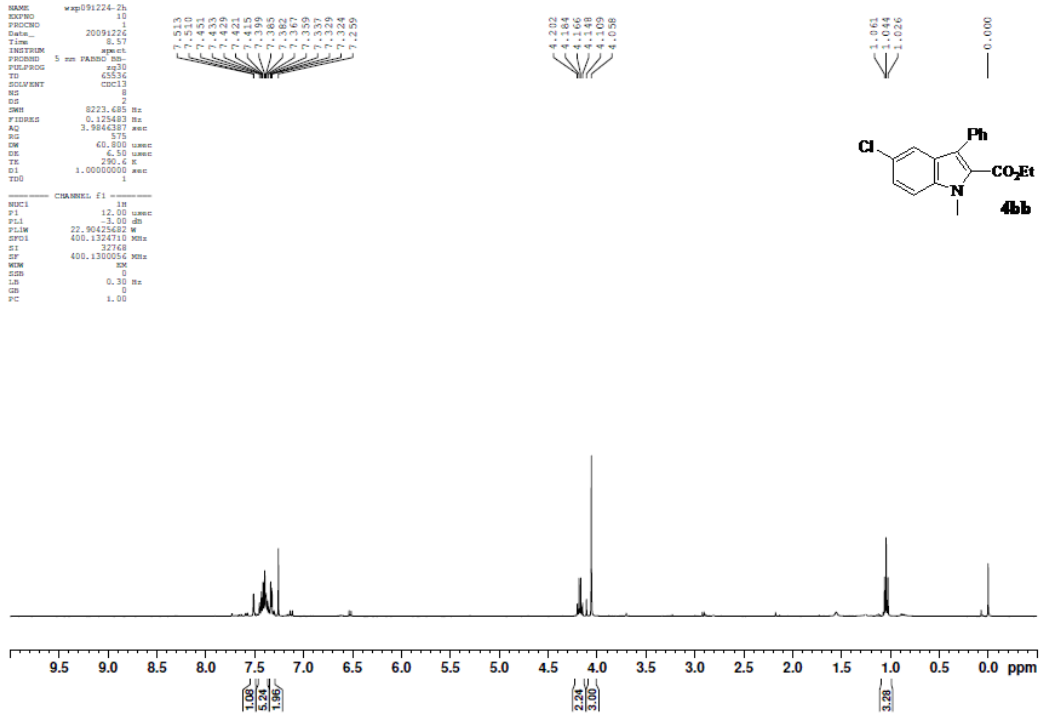

7.498
7.479
7.254
7.054
7.037
7.020
6.983
4.210
3.938
2.776
2.529
0.000
CO2Me
N
4ga
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.05
1.01
0.99
3.04
3.06
3.10
3.00

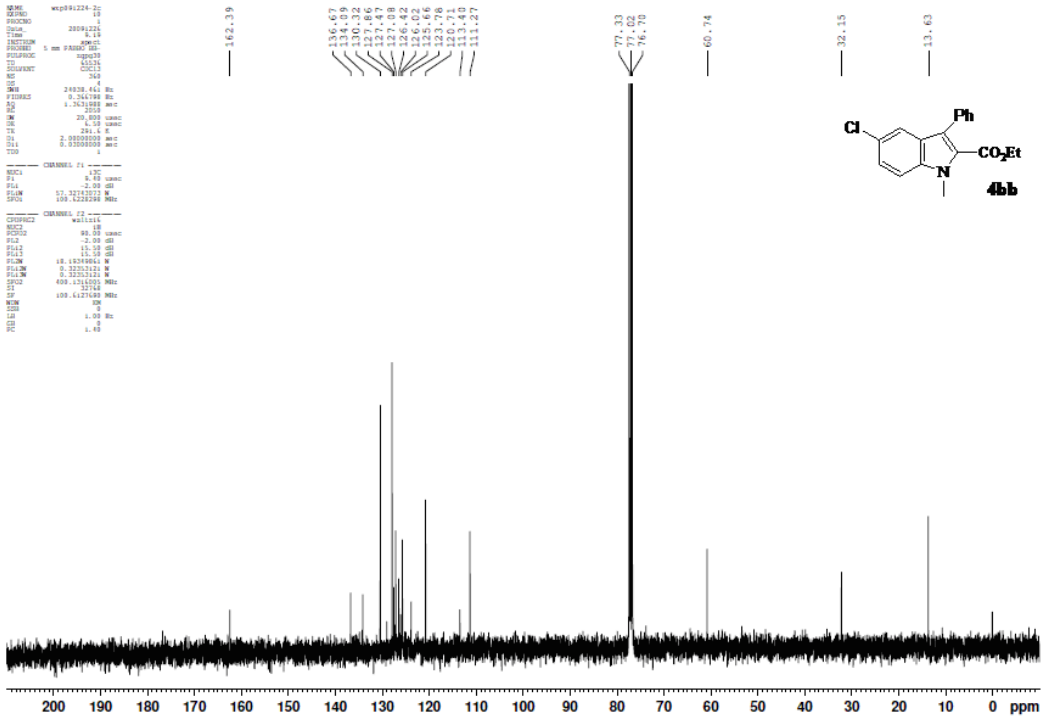

163.52
138.56
128.68
128.04
126.09
122.04
121.01
119.78
118.74
77.32
77.00
76.69
51.40
34.65
20.84
10.78
CO2Me
N
4ga
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

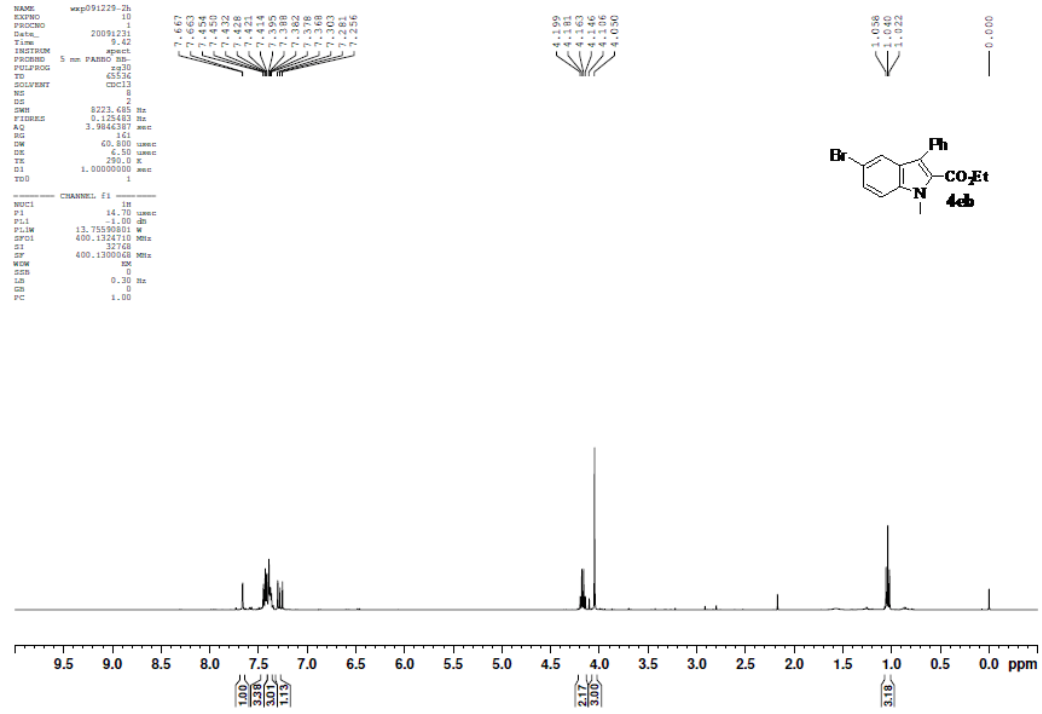

4ha
4ha

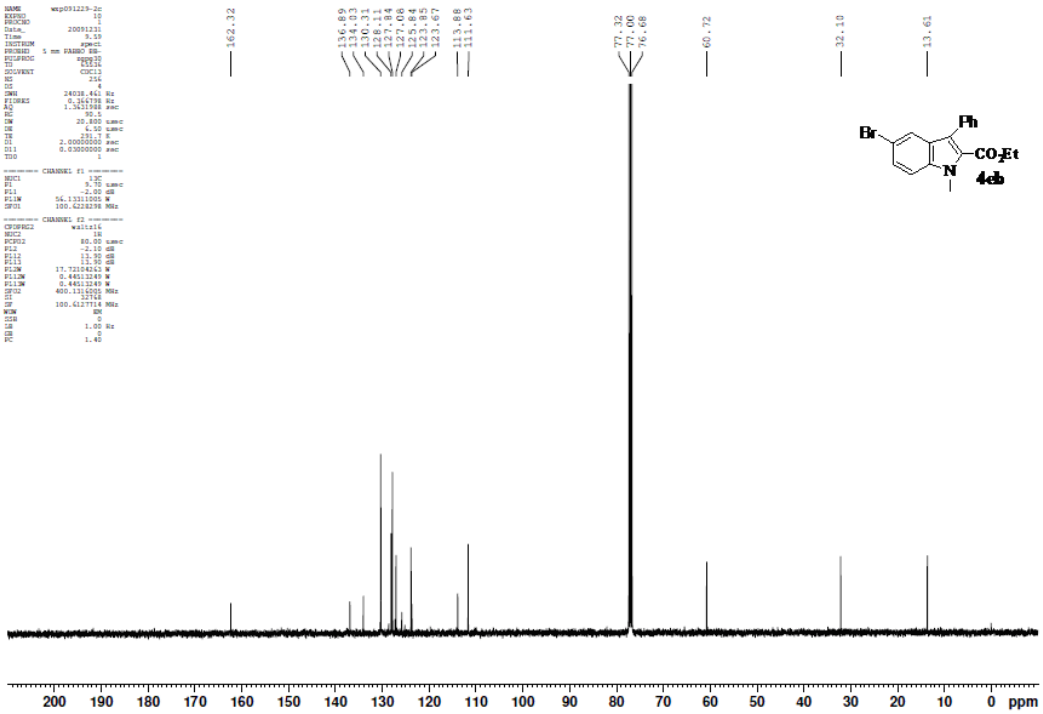

Ph
CO2Et
N
4ab
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.12
4.20
1.46
1.72
1.09
2.04
3.00
3.06

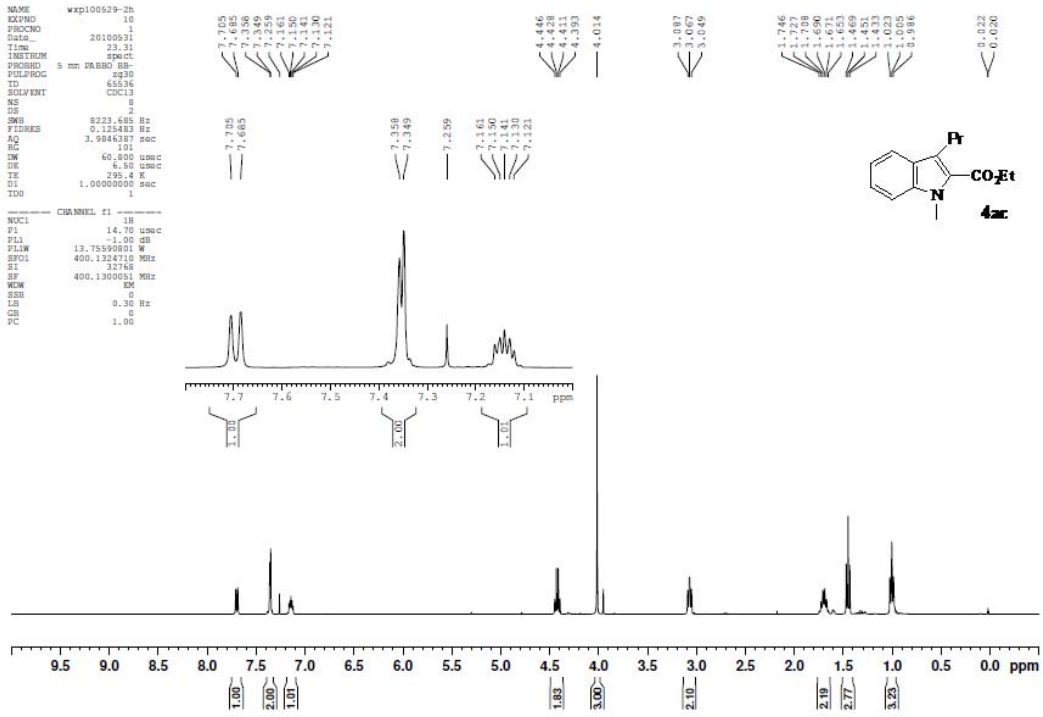

Ph
CO2Et
N
4ab
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

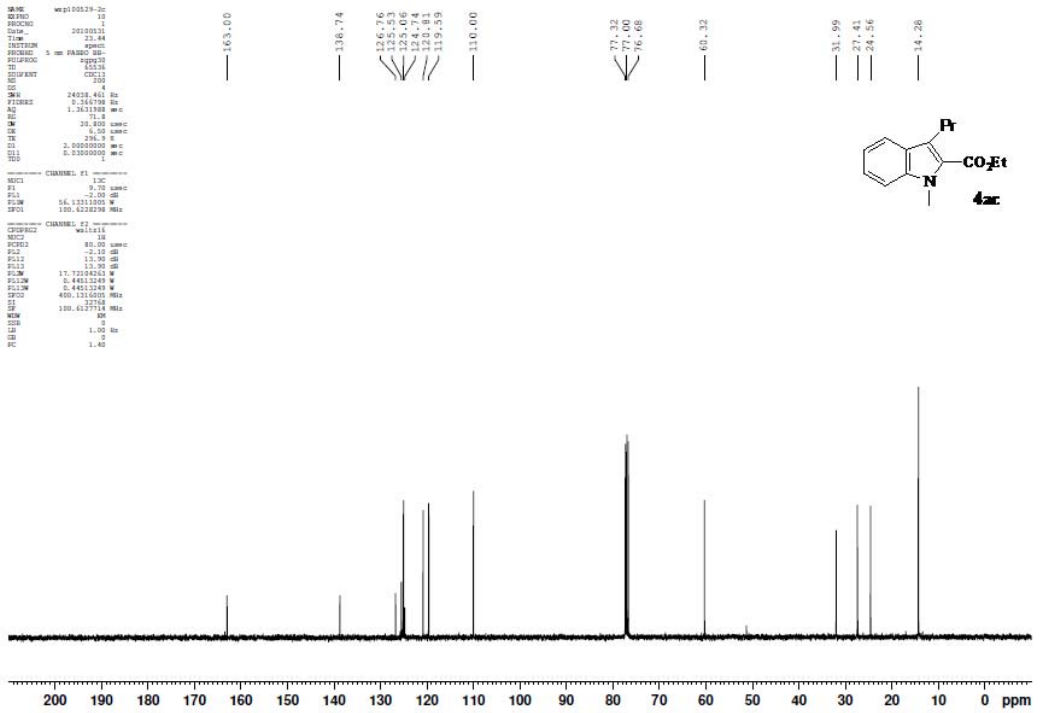

7.513
7.510
7.451
7.433
7.429
7.421
7.415
7.399
7.385
7.362
7.367
7.359
7.337
7.329
7.324
7.259
4.202
4.184
4.166
4.148
4.109
4.098
1.061
1.044
1.026
0.000
Cl
Ph
CO2Et
N
4bb
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
1.04
5.24
1.85
2.24
3.00
3.28

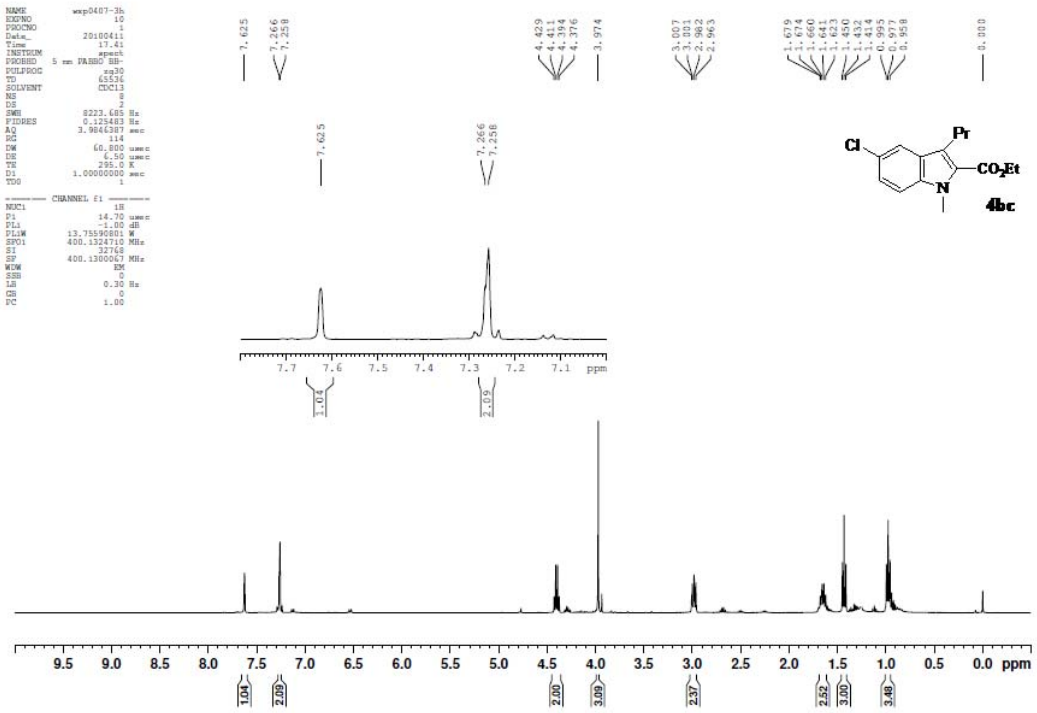

162.39
136.67
134.09
130.32
127.86
127.47
127.08
126.62
126.22
125.66
123.76
120.71
113.49
111.27
77.33
77.02
76.70
60.74
32.15
13.63
Cl
Ph
CO2Et
N
4bb
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

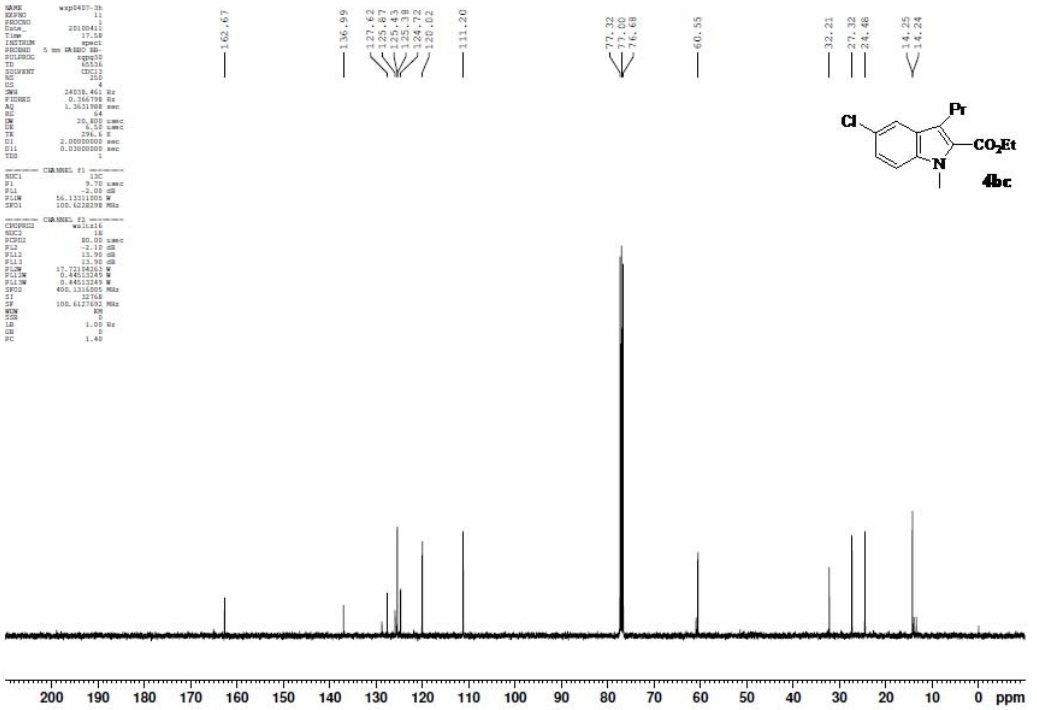

Br
Ph
CO2Et
N
4eb
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm

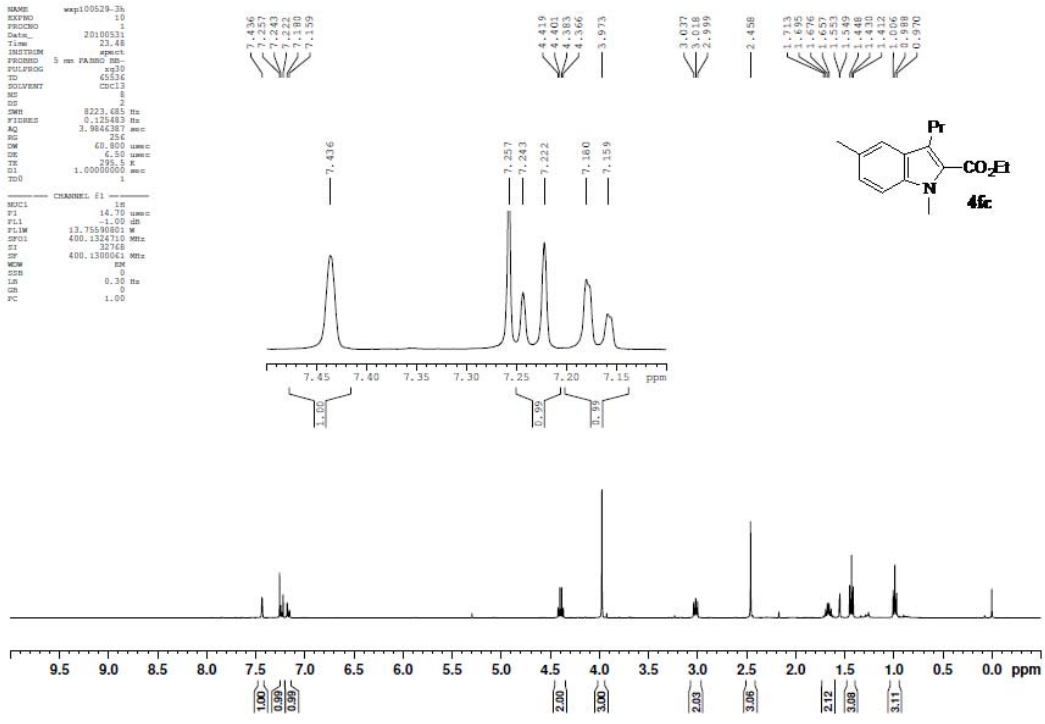

Br
Ph
CO2Et
N
4eb
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

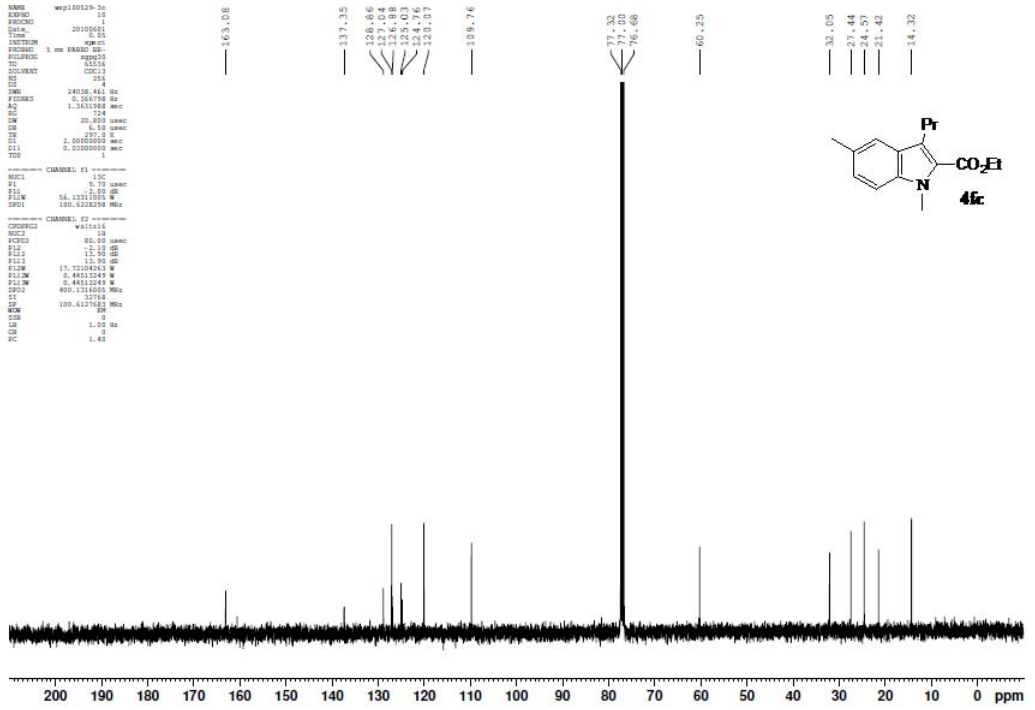

7.705
7.685
7.358
7.349
7.259
7.161
7.150
7.141
7.130
7.121
4.446
4.428
4.411
4.393
4.014
3.087
3.067
3.049
1.746
1.727
1.708
1.690
1.671
1.653
1.469
1.451
1.433
1.023
1.005
0.986
0.022
0.020
Pr
CO2Et
N
4ac
7.7
7.6
7.5
7.4
7.3
7.2
7.1
ppm
1.00
2.00
1.01
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0 ppm
1.00
2.00
1.01
1.83
3.00
2.10
2.19
2.77
3.25


163.60
136.74
126.76
125.63
125.06
122.74
120.81
119.59
110.00
77.32
77.00
76.68
60.32
31.99
27.41
24.56
14.26
Pr
CO2Et
N
4ac
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm


7.625
7.266
7.259
4.429
4.411
4.394
4.376
3.974
3.007
3.001
2.982
2.963
1.679
1.674
1.660
1.641
1.623
1.450
1.432
1.414
0.995
0.977
0.958
0.000
Cl
Pr
CO2Et
N
4bc
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.04
2.09
2.00
3.09
2.57
2.52
3.00
3.48


162.67
136.99
127.62
125.87
125.43
125.38
124.72
120.02
111.20
77.32
77.00
76.68
60.55
32.21
27.32
24.46
14.25
14.24
Cl
Pr
CO2Et
N
4bc
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


Pr
CO2Et
N
4fc
ppm


Pr
CO2Et
N
4fc
ppm