

TiO₂ nano crystallites catalyzed water mediated microwave assisted regioselective three component domino Hydrolysis/Aldol/condensation/ Michael addition reaction-3-(1,5-dioxo-1,5-diphenylpentan-3-yl)quinolin-2(1H)-one

Eethamukkala Ubba^a, Fazlur- Rahman Nawaz Khan^{a,b*}, Euh Duck Jeong^b * Euh Hyuk Chung^b

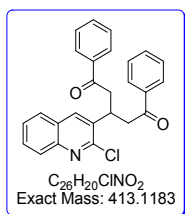
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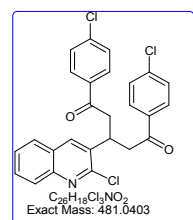
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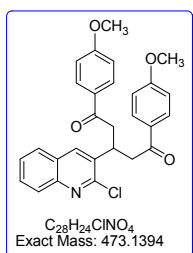
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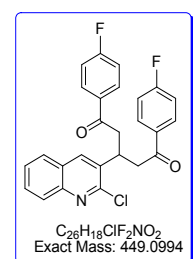
(3a): White Solid, mp: 183–185 °C, FT-IR (KBr, cm⁻¹): 3448, 2926, 2854, 1678, 1666, 1629, 1595, 1450, 1402, 1290, 1222, 1082, 777, 748, 690. ¹H NMR (CDCl₃, 400 MHz): δ = 3.64-3.53 (m, 4H), 4.58-4.52 (qt, 1H), 7.39-7.38 (d, *J* = 5.81 Hz, 4H), 7.50-7.46 (t, *J* = 7.44 Hz, 3H), 7.64-7.60 (t, *J* = 7.1 Hz, 1H), 7.72-7.68 (t, *J* = 6.53 Hz, 1H), 7.90 – 7.89 (d, *J* = 5.89 Hz, 5H), 8.09 (s, 1H). ¹³C NMR (CDCl₃, 100 MHz): δ = 33.94, 42.55, 125.27, 126.85, 127.32, 128.15, 128.70, 130.11, 133.35, 134.62, 136.35, 136.65, 137.29, 145.72, 149.70, 198.04. LCMS [M+1]: 414.0, HRMS [EI⁺] calcd for C₂₆H₂₀ClNO₂ m/z 413.1183 [M⁺], found 413.1180 [M⁺].



(3b): White solid, mp: 214–216 °C, FT-IR (KBr, cm⁻¹): 3057, 2922, 1683, 1587, 1487, 1400, 1338, 1282, 1211, 1091, 991, 873, 812, 783, 758, 597. ¹H NMR (CDCl₃, 400 MHz): δ = 3.68-3.53 (m, 4H), 4.61-4.55 (qt, 1H), 7.44-7.42 (d, *J* = 8.4 Hz, 4H), 7.55-7.52 (t, *J* = 7.2 Hz, 1H), 7.71-7.67 (t, *J* = 7.2 Hz, 1H), 7.79-7.77 (d, *J* = 8.0 Hz, 1H), 7.92-7.90 (d, *J* = 8.4 Hz, 4H), 7.99-7.97 (d, *J* = 8.8 Hz, 1H), 8.15 (s, 1H). ¹³C NMR (CDCl₃, 100Hz): δ = 34.07, 42.48, 124.50, 124.56, 127.27, 127.74, 128.15, 128.70, 130.30, 133.36, 133.64, 135.42, 136.66, 144.92, 149.56, 198.07. LCMS [M+1]: 482.0, HRMS [EI⁺] calcd for C₂₆H₁₈Cl₃NO₂ m/z 481.0403 [M⁺], found 481.0358 [M⁺].

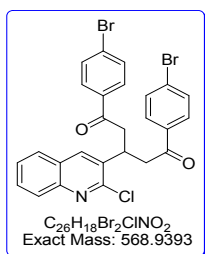


(3c): White solid, mp: 144–146 °C, FT-IR (KBr, cm⁻¹): 3007, 2920, 2839, 1666, 1600, 1487, 1357, 1261, 1026, 983, 831, 752, 698, 594, 547. ¹H NMR (CDCl₃, 400 MHz): δ = 3.64-3.50 (m, 4H), 3.85 (s, 6H), 4.62-4.55 (qt, 1H), 6.92-6.90 (d, *J* = 8.73 Hz, 4H), 7.52-7.49 (t, *J* = 7.76 Hz, 1H), 7.68-7.64 (t, *J* = 7.76 Hz, 1H), 7.77-7.75 (d, *J* = 8.0 Hz, 1H), 7.97-7.95 (d, *J* = 8.73 Hz, 5H), 8.16 (s, 1H). ¹³C NMR (CDCl₃, 100 MHz): δ = 29.72, 42.29, 55.51, 113.83, 127.03, 127.30, 127.40, 128.12, 129.74, 130.03, 130.48, 135.24, 137.04, 146.39, 150.86, 163.68, 196.57. LCMS [M+1]: 474.2, HRMS [EI⁺] calcd for C₂₈H₂₄ClNO₄ m/z 473.1394 [M⁺], found 473.1387 [M⁺].

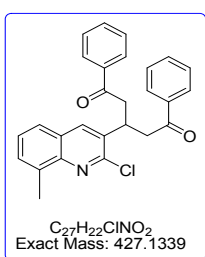


(3d): White solid, mp: 318–320 °C, FT-IR (KBr, cm⁻¹): 3064, 2916, 2895, 1683, 1597, 1409, 1336, 1234, 1211, 1060, 1033, 993, 848, 758, 624, 607, 569. ¹H NMR (CDCl₃, 400 MHz): δ = 3.69-3.54 (m, 4H), 4.62-4.56 (qt, 1H), 7.14-7.10 (t, *J* = 8.87 Hz, 4H), 7.54-7.51 (t, *J* = 7.50 Hz, 1H), 7.70-7.66 (t, *J* = 6.82 Hz, 1H), 7.78-7.76 (d, *J* = 8.18 Hz, 1H), 8.02-7.96 (m,

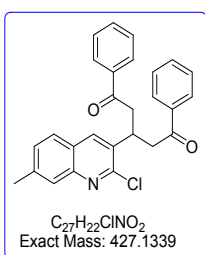
5H), 8.16 (s, 1H). ¹³C NMR (CDCl₃, 100 MHz): δ = 33.99, 42.39, 115.97, 127.23, 127.36, 128.17, 130.26, 130.78, 130.87, 132.98, 133.00, 134.70, 146.47, 150.60, 164.66, 196.37. LCMS [M+1]: 450.2. HRMS [EI⁺] calcd for C₂₆H₁₈ClF₂NO₂ m/z 449.0994 [M⁺], found 449.0987 [M⁺].



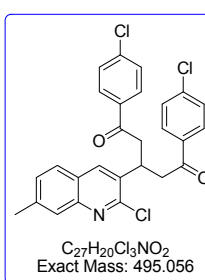
568.9387 [M⁺].



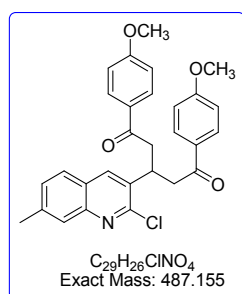
(3f): White solid, mp: 146-148 °C, FT-IR (KBr, cm⁻¹): 3448, 2926, 2854, 1680, 1678, 1668, 1666, 1579, 1450, 1402, 1292, 1222, 1082, 1033, 979, 767, 731, 690. ¹H NMR (CDCl₃, 400 MHz): δ = 2.73 (s, 3H), 3.70-3.58 (m, 4H), 4.65-4.58 (qt, 1H), 7.60-7.39 (m, 9H), 7.98-7.96 (d, *J* = 8.0 Hz, 4H), 8.11 (s, 1H). ¹³C NMR (CDCl₃, 100 MHz): δ = 17.76, 29.72, 42.55, 125.27, 126.85, 127.32, 128.15, 128.70, 130.11, 133.35, 134.62, 136.35, 136.65, 137.29, 145.72, 149.70, 198.03. LCMS [M+1]: 428.4



145.72, 149.70, 198.04. LCMS [M+1]: 428.2

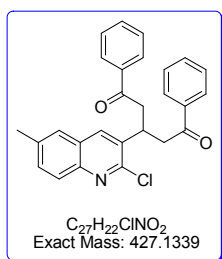


(3h): Yellow solid, mp: 152-154 °C, FT-IR (KBr, cm⁻¹): 3043, 2922, 2893, 2854, 1681, 1587, 1400, 1338, 1276, 1209, 1138, 1093, 1033, 929, 813, 619, 530. ¹H NMR (CDCl₃, 400 MHz): δ = 2.59 (s, 3H), 3.66 (m, 4H), 4.58-4.53 (qt, 1H), 7.37-7.35 (d, *J* = 8.4 Hz, 1H), 7.43-7.41 (d, *J* = 8.4 Hz, 4H), 7.67-7.65 (d, *J* = 8.4 Hz, 1H), 7.74 (s, 1H), 7.92-7.90 (d, *J* = 8.4 Hz, 4H), 8.09 (s, 1H). ¹³C NMR (CDCl₃, 100 MHz): δ = 26.57, 33.95, 42.43, 126.21, 127.25, 127.81, 129.04, 129.55, 132.54, 134.38, 134.88, 136.54, 137.28, 139.92, 145.08, 149.58, 196.78. MS: 495.0. LCMS [M+1]: 496.0



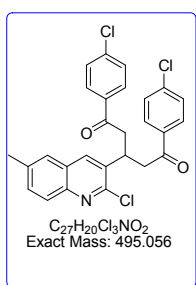
(3i): Yellow solid, mp: 150-152 °C, FT-IR (KBr, cm⁻¹): 3057, 2916, 2837, 1678, 1600, 1419, 1259, 1168, 1028, 987, 825, 617, 585. ¹H NMR (CDCl₃, 400 MHz): δ = 2.51 (s, 3H), 3.62-3.48 (m, 4H), 3.84 (s, 6H), 4.60-4.53 (qt, 1H), 6.91-6.89 (d, *J* = 8.87 Hz, 4H), 7.34-7.32 (d, *J* = 8.18 Hz, 1H), 7.65-7.62 (d, *J* = 8.18 Hz, 1H), 7.73 (s, 1H), 7.97-7.94 (d, *J* = 8.18 Hz, 4H), 8.10 (s, 1H). ¹³C NMR (CDCl₃, 100 MHz): δ = 21.90, 34.30, 42.35, 55.49, 113.81, 125.36, 127.01, 127.10, 129.27, 129.77, 130.47, 134.16, 136.75, 140.52, 146.66, 150.74, 163.65, 196.66. LCMS

[M+1]: 488.2, HRMS [EI⁺] calcd for C₂₉H₂₆ClNO₄ m/z 487.155 [M⁺], found 487.1540 [M⁺].

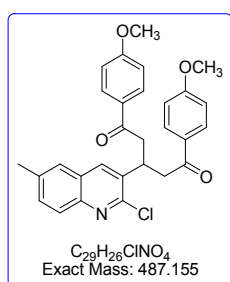


(3j): White solid, mp: 158-160 °C, FT-IR (KBr, cm⁻¹): 3441, 3057, 2922, 2856, 1683, 1672, 1587, 1568, 1487, 1402, 1338, 1282, 1220, 1091, 1060, 985, 821, 756, 690, 580. ¹H NMR (CDCl₃, 400 MHz): δ = 2.49 (s, 3H), 3.70-3.57 (m, 4H), 4.63-4.56 (qt, 1H), 7.46-7.43 (t, *J* = 8.0 Hz, 4H), 7.57-7.48 (m, 4H), 7.87-7.84 (d, *J* = 7.6 Hz, 1H), 7.98-7.96 (d, *J* = 8.0 Hz, 4H), 8.07 (s, 1H). ¹³C NMR (CDCl₃, 100 MHz): δ = 21.58, 33.99, 42.50, 126.25, 127.31, 127.79, 128.14, 128.70, 132.37, 133.36, 134.80, 136.50, 136.64, 137.10, 145.04, 149.80, 198.02. LCMS [M+1]: 428.0,

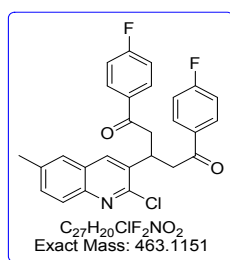
HRMS [EI⁺] calcd for C₂₇H₂₂ClNO₂ m/z 427.1339 [M⁺], found 427.1329 [M⁺].



(3k): White solid, mp: 170-172 °C, FT-IR (KBr, cm⁻¹): 2968, 2922, 2854, 1685, 1587, 1400, 1334, 1182, 1091, 873, 821, 584. ¹H NMR (CDCl₃, 400 MHz): δ = 2.50 (s, 3H), 3.66-3.52 (m, 4H), 4.58-4.53 (m, 1H), 7.43-7.41 (d, *J* = 8.4 Hz, 4H), 7.53-7.50 (d, *J* = 8.0 Hz, 2H), 7.87-7.85 (d, *J* = 8.4 Hz, 1H), 7.92-7.90 (d, *J* = 8.4 Hz, 4H), 8.05 (s, 1H). ¹³C NMR (CDCl₃, 100 MHz): δ = 21.59, 33.95, 42.43, 126.21, 127.25, 127.81, 128.90, 129.04, 129.55, 132.54, 134.38, 134.88, 137.28, 139.92, 145.08, 149.58, 196.78. LCMS [M+1]: 496.0, HRMS [EI⁺] calcd for C₂₇H₂₀Cl₃NO₂ m/z 495.056 [M⁺], found 495.0510 [M⁺].

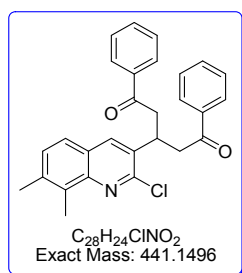


(3l): White solid, mp: 202-204 °C, FT-IR (KBr, cm⁻¹): 3028, 2966, 2841, 1666, 1604, 1419, 1348, 1263, 1176, 1024, 871, 842, 615, 578. ¹H NMR (CDCl₃, 400 MHz): δ = 2.49 (s, 3H), 3.62-3.49 (m, 4H), 3.85 (s, 6H), 4.60-4.53 (qt, 1H), 6.92-6.90 (d, *J* = 9.2 Hz, 4H), 7.50-7.48 (d, *J* = 8.51 Hz, 1H), 7.52 (s, 1H), 7.86-7.84 (d, *J* = 8.51 Hz, 1H), 7.97-7.95 (d, *J* = 8.51 Hz, 4H), 8.06 (s, 1H). ¹³C NMR (CDCl₃, 100 MHz): δ = 21.57, 34.37, 42.33, 55.49, 113.82, 126.27, 127.34, 127.76, 129.78, 130.47, 132.27, 135.03, 136.42, 137.01, 144.99, 149.89, 163.66, 196.61. LCMS [M+1]: 488.2



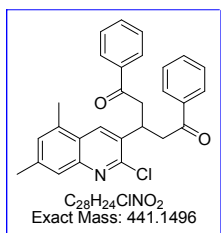
(3m): White solid, mp: 140-142 °C, FT-IR (KBr, cm⁻¹): 3111, 2918, 2850, 1681, 1598, 1508, 1409, 1232, 1157, 1035, 831, 700, 586. ¹H NMR (CDCl₃, 400 MHz): δ = 2.50 (s, 3H), 3.67-3.53 (m, 4H), 4.58-4.53 (qt, 1H), 7.14-7.10 (t, *J* = 8.8 Hz, 4H), 7.53-7.50 (dd, *J* = 4.8 Hz, 2H), 7.87-7.85 (d, *J* = 8.4 Hz, 1H), 8.02-7.99 (m, 4H), 8.07 (s, 1H). ¹³C NMR (CDCl₃, 100 MHz): δ = 21.60, 34.03, 42.42, 115.74, 115.96, 126.22, 127.27, 127.81, 130.87, 132.51, 134.48, 137.25, 145.07, 149.63, 164.65, 167.19, 196.42. LCMS [M+1]: 464.0, HRMS [EI⁺]

calcd for C₂₇H₂₀ClF₂NO₂ m/z 463.1151 [M⁺], found 463.1146 [M⁺].

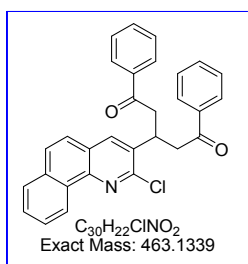


(3n): White solid, mp: 176-178 °C, FT-IR (KBr, cm⁻¹): 3062, 2968, 2848, 1680, 1678, 1670, 1595, 1446, 1402, 1327, 1288, 1220, 1097, 1026, 758, 690. ¹H NMR (CDCl₃, 400 MHz): δ = 2.46 (s, 3H), 2.67 (s, 3H), 3.69-3.56 (m, 4H), 4.63-4.56 (qt, 1H), 7.33-7.31 (d, *J* = 8.36 Hz, 1H), 7.46-7.42 (t, *J* = 7.89 Hz, 4H), 7.51-7.49 (d, *J* = 8.36 Hz, 1H), 7.57-7.53 (t, *J* = 7.43 Hz, 2H), 7.98-7.96 (d, *J* = 7.43 Hz, 4H), 8.06 (s, 1H). ¹³C NMR (CDCl₃, 100 MHz): δ = 21.82, 29.71, 34.53, 42.67, 124.73, 125.34, 128.14, 128.70, 129.95, 133.13, 133.34, 133.87,

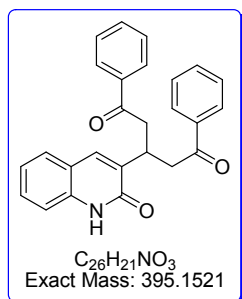
134.01, 136.72, 140.35, 147.19, 150.18, 198.26. LCMS [M+1]: 445.0



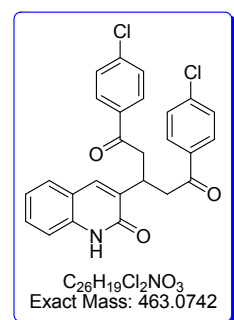
(3o): Light brown, mp: 138-140 °C, FT-IR (KBr, cm^{-1}): 3452, 2953, 2920, 2850, 2742, 1674, 1620, 1595, 1446, 1402, 1350, 1274, 1220, 1099, 1001, 974, 860, 758, 690, 538. 1H NMR ($CDCl_3$, 400 MHz): δ = 2.47 (s, 3H), 2.57 (s, 3H), 3.69-3.58 (m, 4H), 4.65-4.58 (qt, 1H), 7.17 (s, 1H), 7.46-7.42 (t, J = 7.6 Hz, 4H), 7.57-7.53 (t, J = 7.2 Hz, 2H), 7.58 (s, 1H), 7.98-7.96 (d, J = 7.6 Hz, 4H), 8.23 (s, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 18.59, 21.83, 34.54, 42.67, 124.73, 125.34, 128.14, 128.71, 129.95, 133.13, 133.35, 133.87, 134.01, 136.72, 140.36, 147.19, 150.19, 198.26. LCMS [M+1]: 442.2, HRMS [EI⁺] calcd for $C_{28}H_{24}ClNO_2$ m/z 441.1496 [M⁺], found 441.1489 [M⁺].



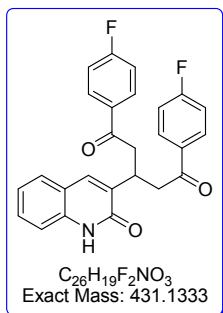
(3p): White solid, mp: 168-170 °C, FT-IR (KBr, cm^{-1}): 3448, 3028, 2960, 2922, 2854, 1681, 1641, 1595, 1448, 1402, 1363, 1292, 1215, 1111, 1092, 1056, 989, 754, 684, 594, 563. 1H NMR ($CDCl_3$, 400 MHz): δ = 3.75-3.62 (m, 4H), 4.70-4.63 (qt, 1H), 7.47-7.43 (t, J = 7.6 Hz, 4H), 7.57-7.54 (t, J = 7.6 Hz, 2H), 7.65-7.63 (d, J = 8.8 Hz, 1H), 7.73-7.66 (qt, 2H), 7.80-7.78 (d, J = 8.8 Hz, 1H), 7.89-7.87 (d, J = 8.0 Hz, 1H), 7.99-7.97 (d, J = 7.6 Hz, 4H), 8.19 (s, 1H), 9.17-9.15 (d, J = 7.6 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 32.87, 41.37, 126.17, 126.20, 126.31, 127.14, 127.87, 128.02, 128.51, 128.70, 129.24, 133.55, 133.81, 136.14, 138.92, 145.45, 149.50, 195.68. LCMS [M+1]: 464.0, HRMS [EI⁺] calcd for $C_{30}H_{22}ClNO_2$ m/z 463.1339 [M⁺], found 463.1335 [M⁺].



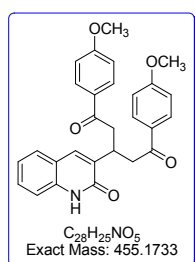
(4a): White solid, mp: 228-230 °C, FT-IR (KBr, cm^{-1}): 3523, 3462, 3010, 2918, 2850, 1678, 1658, 1595, 1400, 1363, 1226, 1155, 993, 881, 748, 688. 1H NMR ($CDCl_3$, 400 MHz): δ = 3.62-3.56 (dd, J = 6.60 Hz, J = 17.23 Hz, 2H), 3.83-3.77 (dd, J = 7.64 Hz, J = 17.11 Hz, 2H), 4.28-4.21 (qt, 1H), 7.23-7.16 (m, 2H), 7.44-7.40 (t, J = 7.29 Hz, 5H), 7.55-7.51 (m, 3H), 7.85 (s, 1H), 8.01-8.00 (d, J = 7.64 Hz, 4H), 10.79 (s, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 34.58, 41.46, 115.02, 117.92, 124.33, 127.73, 130.87, 130.96, 132.31, 133.43, 133.46, 137.65, 138.73, 140.80, 163.54, 197.56. LCMS [M+1]: 394.2 HRMS [EI⁺] calcd for $C_{26}H_{21}NO_3$ m/z 395.1521 [M⁺], found 395.1512 [M⁺].



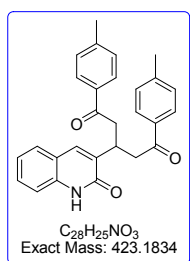
(4b): Greenish yellow solid, mp: 197-200 °C, FT-IR (KBr, cm^{-1}): 3545, 3446, 3064, 2918, 2850, 1680, 1649, 1431, 1263, 1215, 1153, 1093, 987, 831, 750, 628. 1H NMR ($CDCl_3$, 400 MHz): δ = 3.58-3.52 (dd, J = 6.80 Hz, J = 16.80 Hz, 2H), 3.77-3.71 (dd, J = 6.80 Hz, J = 17.60 Hz, 2H), 4.24-4.17 (qt, 1H), 7.23-7.21 (dd, J = 2.4 Hz, 2H), 7.41-7.39 (d, J = 8.4 Hz, 5H), 7.57-7.55 (d, J = 7.61 Hz, 1H), 7.83 (s, 1H), 7.96-7.94 (d, J = 8.82 Hz, 4H), 10.87 (s, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 29.72, 41.40, 115.19, 120.11, 122.50, 127.84, 128.35, 129.26, 129.78, 133.95, 134.56, 137.51, 138.71, 143.83, 163.59, 198.76. LCMS [M+1]: 462.0, HRMS [EI⁺] calcd for $C_{26}H_{19}Cl_2NO_3$ m/z 463.0742 [M⁺], found 463.0714 [M⁺].



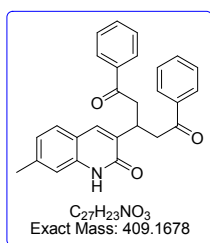
(4c): White solid, mp: 175-178 °C, FT-IR (KBr, cm^{-1}): 3433, 3165, 3012, 2902, 1680, 1649, 1597, 1409, 1276, 1228, 1157, 997, 873, 827, 599. ^1H NMR (CDCl_3 , 400 MHz): δ = 3.40-3.34 (dd, J = 8.00 Hz, J = 17.60 Hz, 2H), 3.93-3.88 (dd, J = 3.65 Hz, J = 16.80 Hz, 2H), 4.25-4.23 (qt, 1H), 6.91-6.87 (t, J = 7.77 Hz, 2H), 7.00-6.98 (d, J = 8.36 Hz, 2H), 7.52-7.46 (q, J = 7.77 Hz, 4H), 7.65-7.63 (d, J = 7.76 Hz, 2H), 7.85-7.83 (d, J = 8.27 Hz, 2H), 8.03 (s, 1H), 10.90 (s, 1H). ^{13}C NMR (CDCl_3 , 100 MHz): δ = 29.71, 41.37, 115.58, 115.80, 119.67, 122.97, 126.39, 126.63, 130.37, 130.91, 133.64, 140.20, 143.30, 163.07, 167.06, 197.39. LCMS [$\text{M}+1$]: 430.2 HRMS [EI^+] calcd for $\text{C}_{26}\text{H}_{19}\text{F}_2\text{NO}_3$ m/z 431.1333 [M^+], found 431.1320 [M^+].



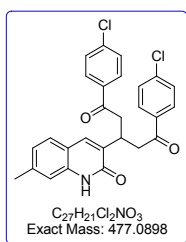
(4d): White solid, mp: 187-190 °C, FT-IR (KBr, cm^{-1}): 3481, 3007, 2839, 1681, 1649, 1573, 1402, 1259, 1170, 1029, 991, 829, 752, 613. ^1H NMR (CDCl_3 , 400 MHz): δ = 3.54-3.48 (dd, J = 6.40 Hz, J = 16.40 Hz, 2H), 3.76-3.70 (dd, J = 7.20 Hz, J = 16.40 Hz, 2H), 3.82 (s, 6H), 4.28-4.22 (qt, 1H), 6.89-6.87 (d, J = 8.4 Hz, 4H), 7.18-7.15 (t, J = 7.6 Hz, 1H), 7.33-7.31 (d, J = 8.0 Hz, 1H), 7.43-7.39 (t, J = 3.2 Hz, 1H), 7.53-7.51 (d, J = 7.6 Hz, 1H), 7.81 (s, 1H), 8.02-8.00 (d, J = 8.8 Hz, 4H), 11.93 (s, 1H). ^{13}C NMR (CDCl_3 , 100 MHz): δ = 34.91, 41.27, 55.45, 113.71, 115.24, 120.12, 122.49, 127.82, 129.76, 130.14, 130.55, 134.00, 137.54, 138.53, 163.46, 163.71, 197.73. LCMS [$\text{M}+1$]: 454.2 HRMS [EI^+] calcd for $\text{C}_{28}\text{H}_{25}\text{NO}_5$ m/z 455.1733 [M^+], found 455.1721 [M^+].



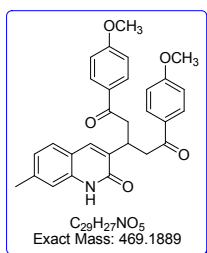
(4e): Greenish yellow solid, mp: 235-238 °C, FT-IR (KBr, cm^{-1}): 3446, 3010, 2958, 2866, 1672, 1604, 1431, 1402, 1365, 1284, 1211, 1058, 999, 873, 750. ^1H NMR (CDCl_3 , 400 MHz): δ = 2.37 (s, 6H), 3.58-3.52 (dd, J = 6.31 Hz, J = 17.04 Hz, 2H), 3.79-3.73 (dd, J = 7.36 Hz, J = 17.04 Hz, 2H), 4.28-4.22 (qt, 1H), 7.17-7.15 (d, J = 7.42 Hz, 1H), 7.22-7.20 (d, J = 7.94 Hz, 4H), 7.33-7.29 (t, J = 7.12 Hz, 1H), 7.43-7.39 (t, J = 7.12 Hz, 1H), 7.54-7.52 (d, J = 7.60 Hz, 1H), 7.83 (s, 1H), 7.92-7.90 (d, J = 8.07 Hz, 4H), 11.74 (s, 1H). ^{13}C NMR (CDCl_3 , 100 MHz): δ = 21.65, 34.68, 41.40, 53.45, 115.19, 120.11, 122.50, 127.84, 128.35, 129.26, 129.78, 133.95, 134.56, 137.51, 138.71, 143.83, 163.59, 198.76. LCMS [$\text{M}+1$]: 424.2, HRMS [EI^+] calcd for $\text{C}_{28}\text{H}_{25}\text{NO}_3$ m/z 423.1834 [M^+], found 423.1840 [M^+].



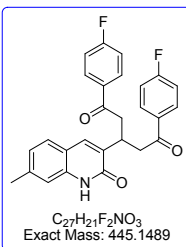
(4f): Greenish yellow solid, mp: 188-190 °C, FT-IR (KBr, cm^{-1}): 3545, 3419, 3062, 2854, 1680, 1649, 1446, 1400, 1271, 1213, 1093, 995, 858, 636. ^1H NMR (CDCl_3 , 400 MHz): δ = 2.40 (s, 3H), 3.62-3.56 (dd, J = 6.34 Hz, J = 17.20 Hz, 2H), 3.83-3.77 (dd, J = 7.25 Hz, J = 17.15 Hz, 2H), 4.27-4.21 (qt, 1H), 7.00-6.98 (d, J = 7.89 Hz, 1H), 7.06 (s, 1H), 7.43-7.39 (t, J = 7.29 Hz, 5H), 7.53-7.50 (t, J = 7.29 Hz, 2H), 7.80 (s, 1H), 8.02-8.00 (d, J = 8.4 Hz, 4H), 11.13 (s, 1H). ^{13}C NMR (CDCl_3 , 100 MHz): δ = 21.81, 34.61, 41.45, 114.94, 117.93, 124.15, 127.68, 128.20, 128.57, 132.49, 133.06, 137.03, 137.60, 138.80, 140.59, 163.43, 199.18. LCMS [$\text{M}+1$]: 410.2, HRMS [EI^+] calcd for $\text{C}_{27}\text{H}_{23}\text{NO}_3$ m/z 409.1678 [M^+], found 409.1654 [M^+].



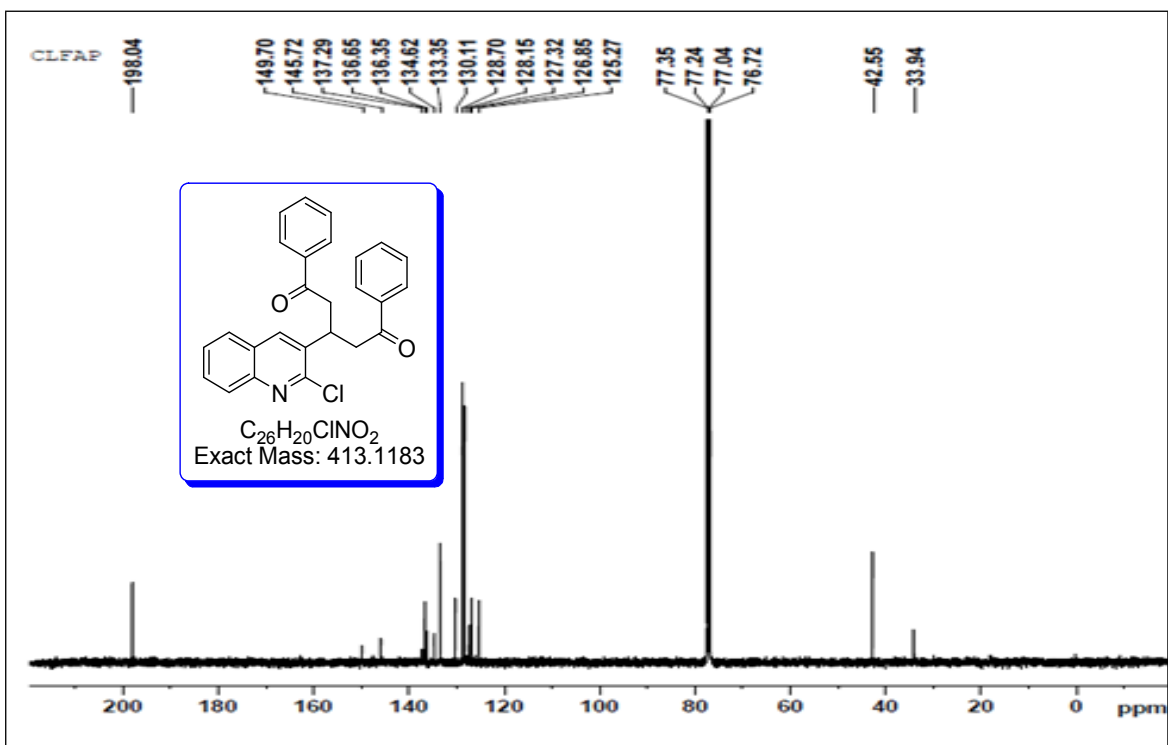
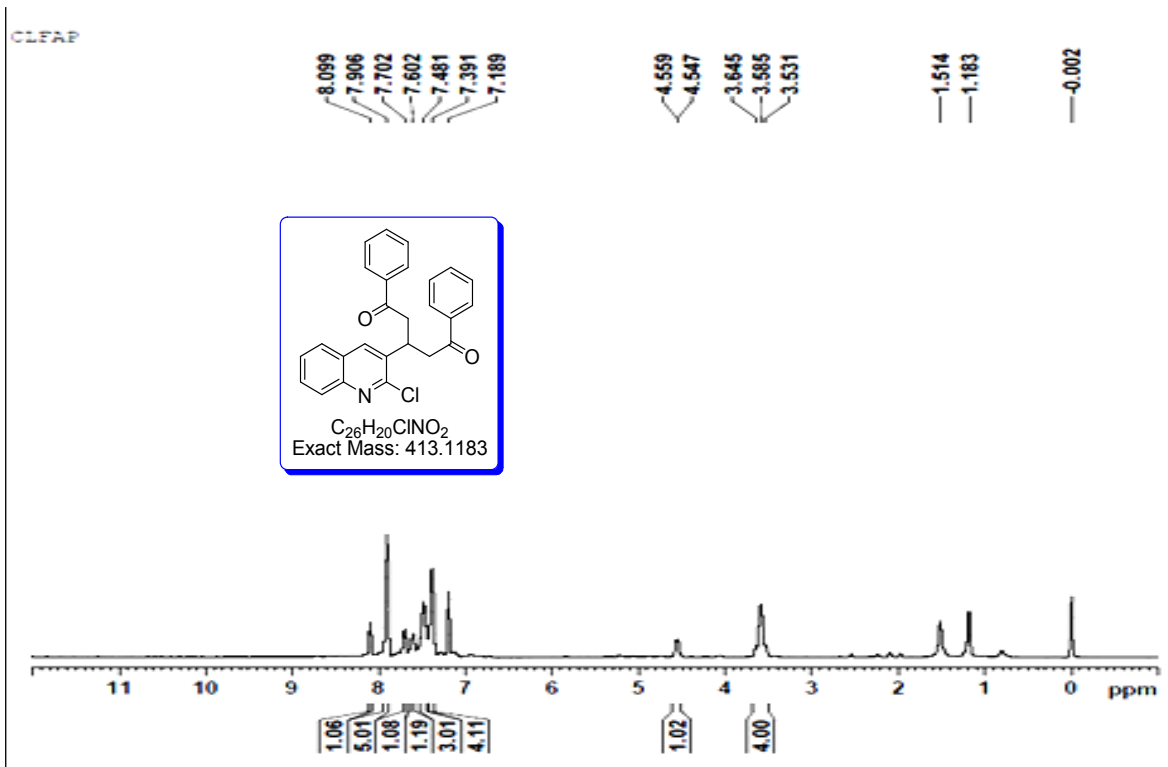
(4g): Greenish yellow solid, mp: 172-175 °C, FT-IR (KBr, cm^{-1}): 3672, 3404, 3194, 2918, 1678, 1651, 1598, 1508, 1409, 1226, 1159, 995, 839, 752, 599. 1H NMR ($CDCl_3$, 400 MHz): δ = 2.40 (s, 3H), 3.56-3.51 (dd, J = 6.31 Hz, J = 17.25 Hz, 2H), 3.76-3.70 (dd, J = 7.15 Hz, J = 17.25 Hz, 2H), 4.22-4.16 (qt, 1H), 7.03-7.00 (d, J = 11.48 Hz, 2H), 7.40-7.38 (d, J = 8.38 Hz, 5H), 7.77 (s, 1H), 7.96-7.94 (d, J = 8.69 Hz, 4H), 10.96 (s, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 21.81, 34.55, 41.41, 114.87, 117.84, 124.31, 127.74, 128.91, 129.65, 132.18, 135.25, 137.55, 138.78, 139.59, 140.83, 163.24, 197.88. LCMS [M+1]: 476.0, HRMS [EI⁺] calcd for $C_{27}H_{21}Cl_2NO_3$ m/z 477.0898 [M⁺], found 477.0871 [M⁺].

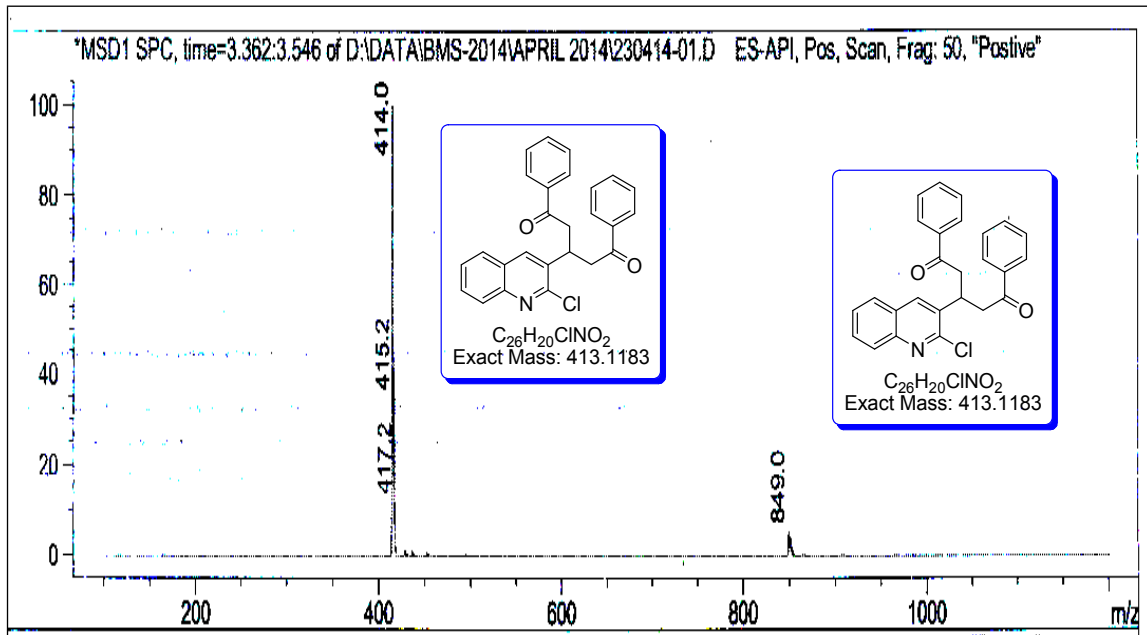
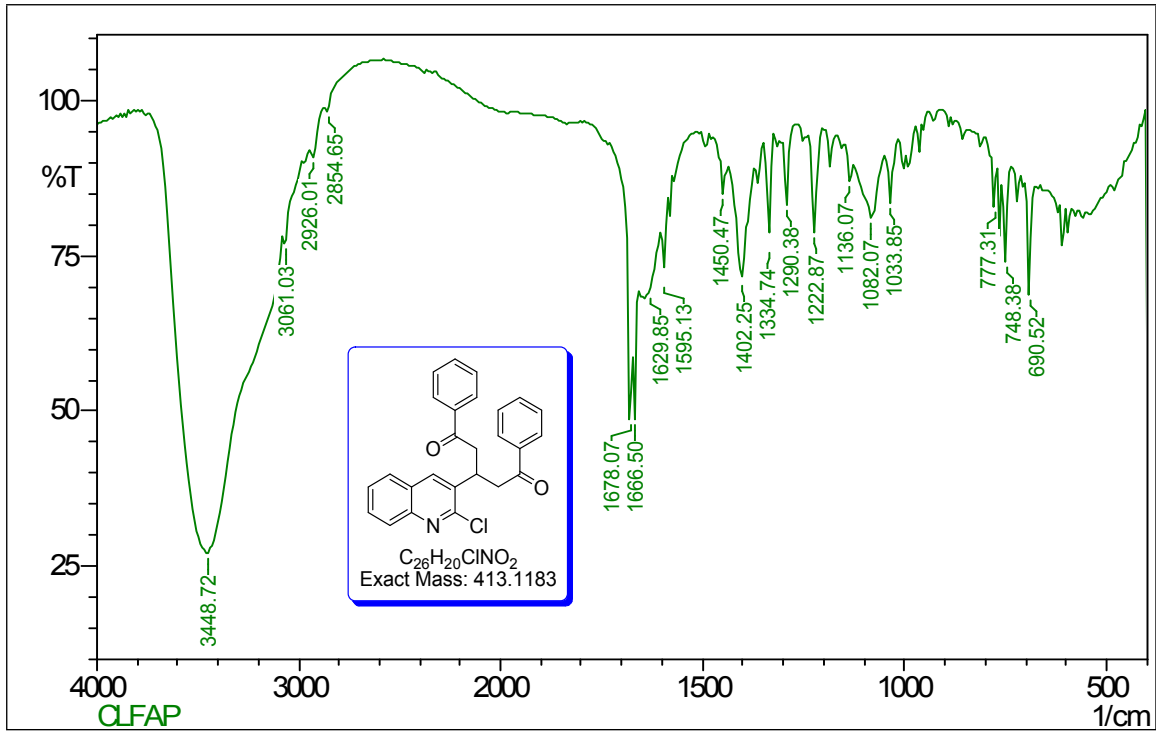


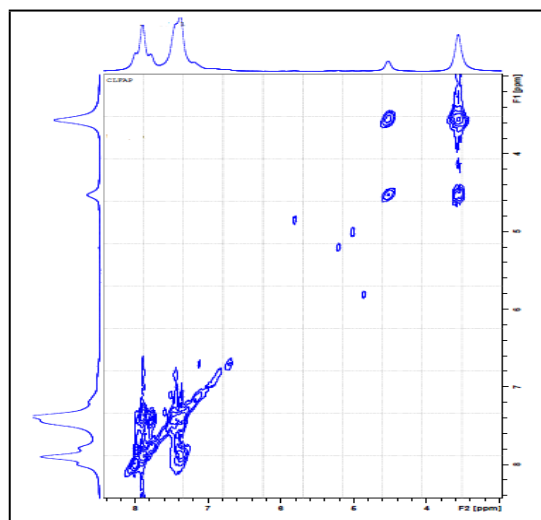
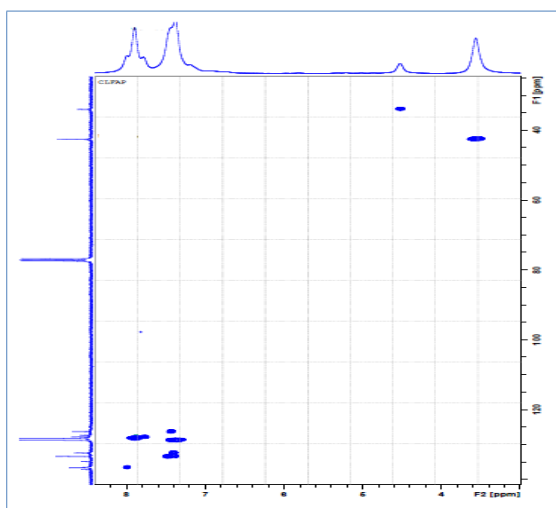
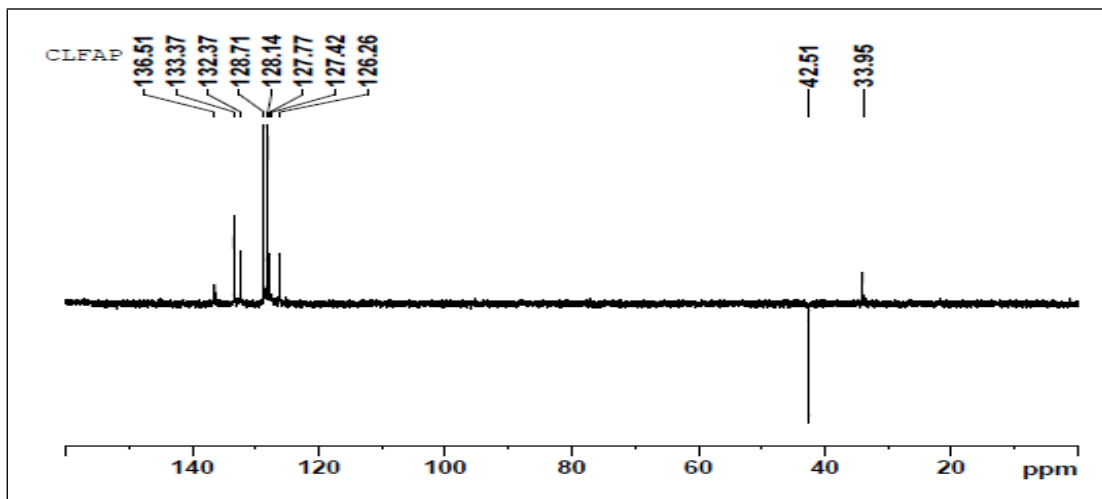
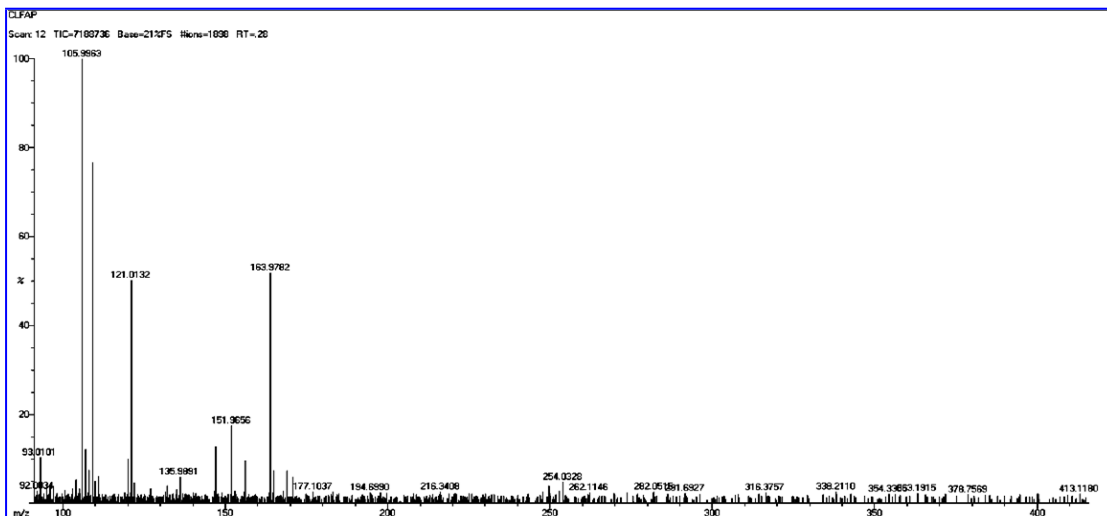
(4h): Greenish yellow solid, mp: 162-165 °C, FT-IR (KBr, cm^{-1}): 3444, 3008, 2954, 2848, 1660, 1600, 1462, 1402, 1334, 1257, 1112, 1029, 983, 871, 806, 630. 1H NMR ($CDCl_3$, 400 MHz): δ = 2.39 (s, 3H), 3.53-3.47 (dd, J = 6.52 Hz, J = 16.83 Hz, 2H), 3.75-3.69 (dd, J = 7.15 Hz, J = 16.83 Hz, 2H), 3.82 (s, 6H), 4.25-4.18 (qt, 1H), 6.88-6.86 (d, J = 8.78 Hz, 4H), 7.07 (s, 1H), 7.41-7.39 (d, J = 7.99 Hz, 1H), 7.76 (s, 1H), 8.02-8.00 (d, J = 8.78 Hz, 5H), 11.22 (s, 1H). ^{13}C NMR ($CDCl_3$, 400 MHz): δ = 21.80, 34.93, 41.22, 55.44, 113.68, 114.96, 117.96, 124.08, 127.65, 130.19, 130.53, 132.75, 137.58, 138.45, 140.48, 143.00, 163.43, 197.81. LCMS [M+1]: 470.2, HRMS [EI⁺] calcd for $C_{29}H_{27}NO_5$ m/z 469.1889 [M⁺], found 469.1870 [M⁺].

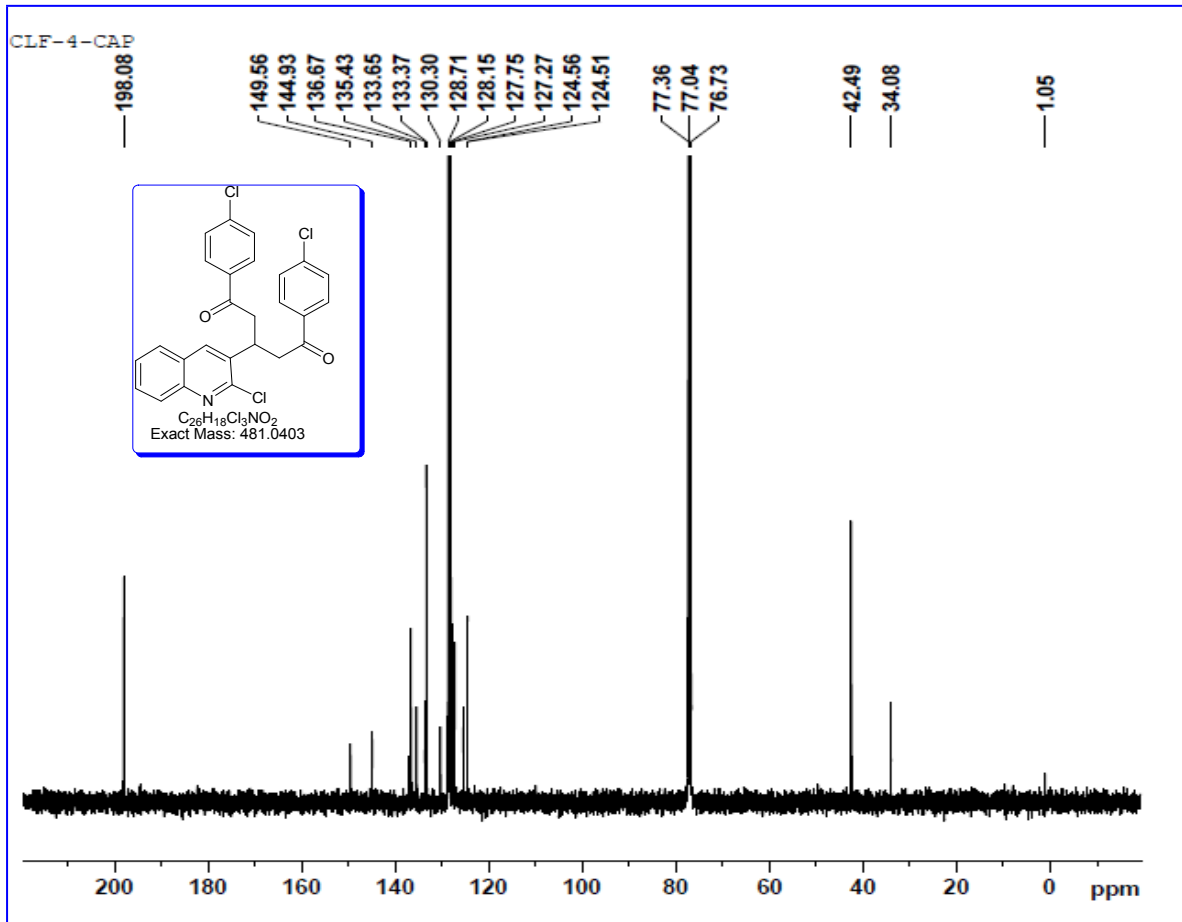
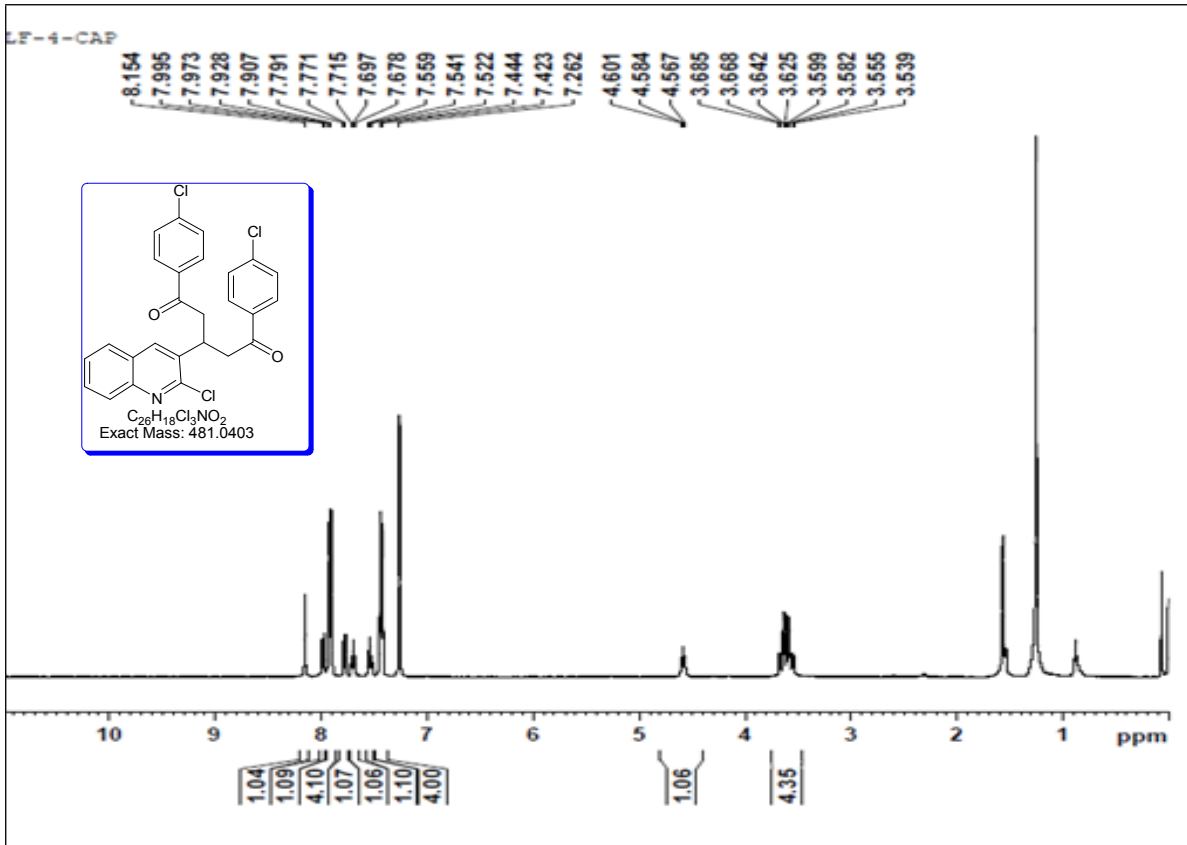


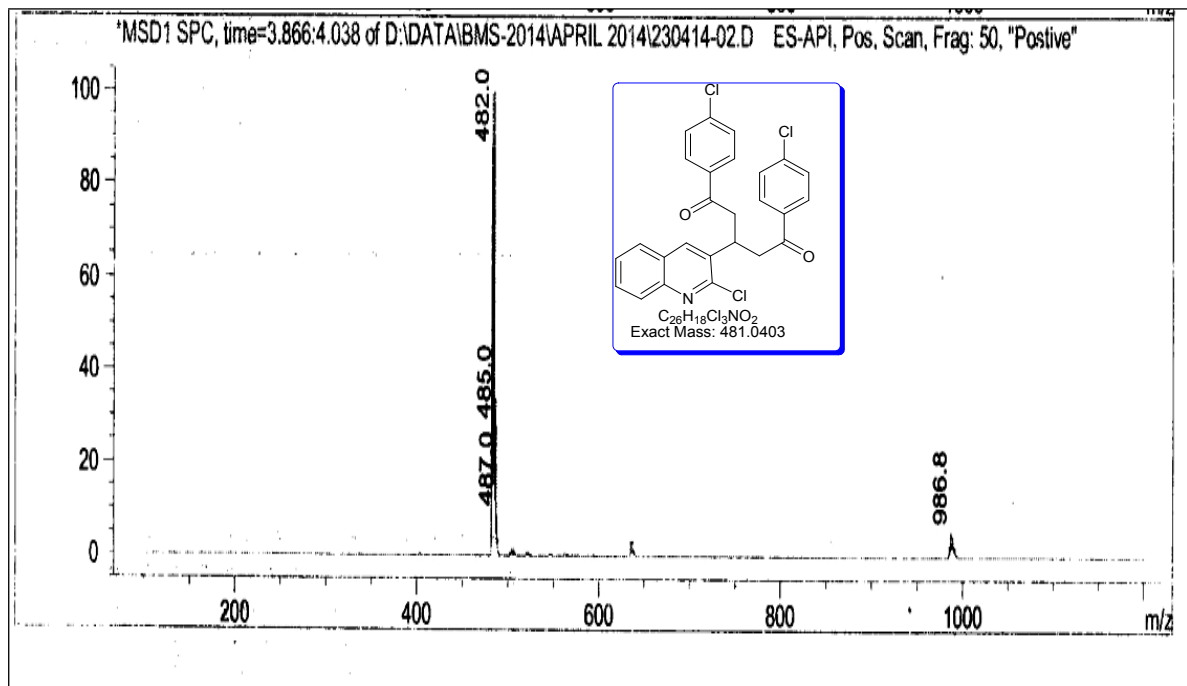
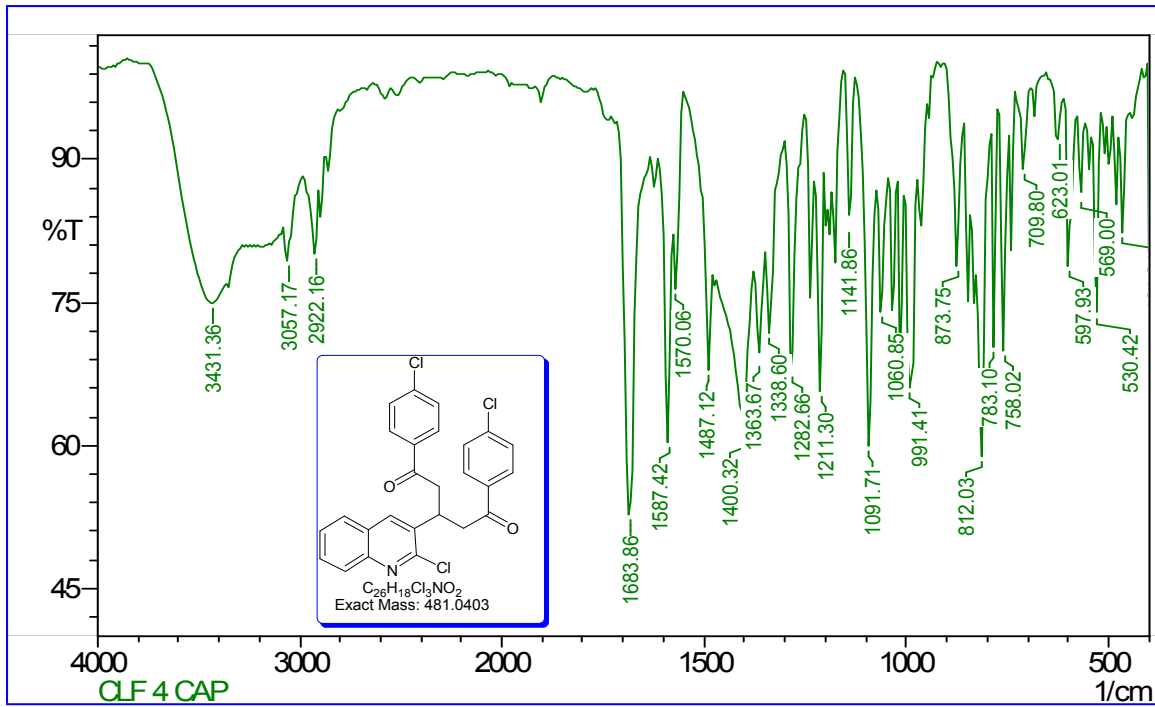
(4i): Pale greenish yellow solid, mp: 150-153 °C, FT-IR (KBr, cm^{-1}): 3437, 3147, 2926, 1670, 1654, 1597, 1504, 1408, 1261, 1230, 1157, 1099, 829, 808, 609. 1H NMR ($CDCl_3$, 400 MHz) δ = 2.41 (s, 3H), 3.57-3.51 (dd, J = 6.31 Hz, J = 17.04 Hz, 2H), 3.77-3.71 (dd, J = 7.15 Hz, J = 17.04 Hz, 2H), 4.21-4.18 (qt, 1H), 7.02-7.00 (d, J = 7.60 Hz, 2H), 7.11-7.07 (t, J = 8.44 Hz, 4H), 7.44-7.42 (d, J = 8.02 Hz, 1H), 7.78 (s, 1H), 8.06-8.03 (t, J = 7.60 Hz, 4H), 10.70 (s, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 21.79, 34.64, 41.35, 114.81, 115.57, 115.78, 124.28, 127.75, 130.82, 130.91, 132.30, 133.41, 137.49, 138.79, 140.78, 164.51, 197.51 ppm. LCMS [M+1]: 444.0, HRMS [EI⁺] calcd for $C_{27}H_{21}F_2NO_3$ m/z 445.1489 [M⁺], found 445.1480 [M⁺].

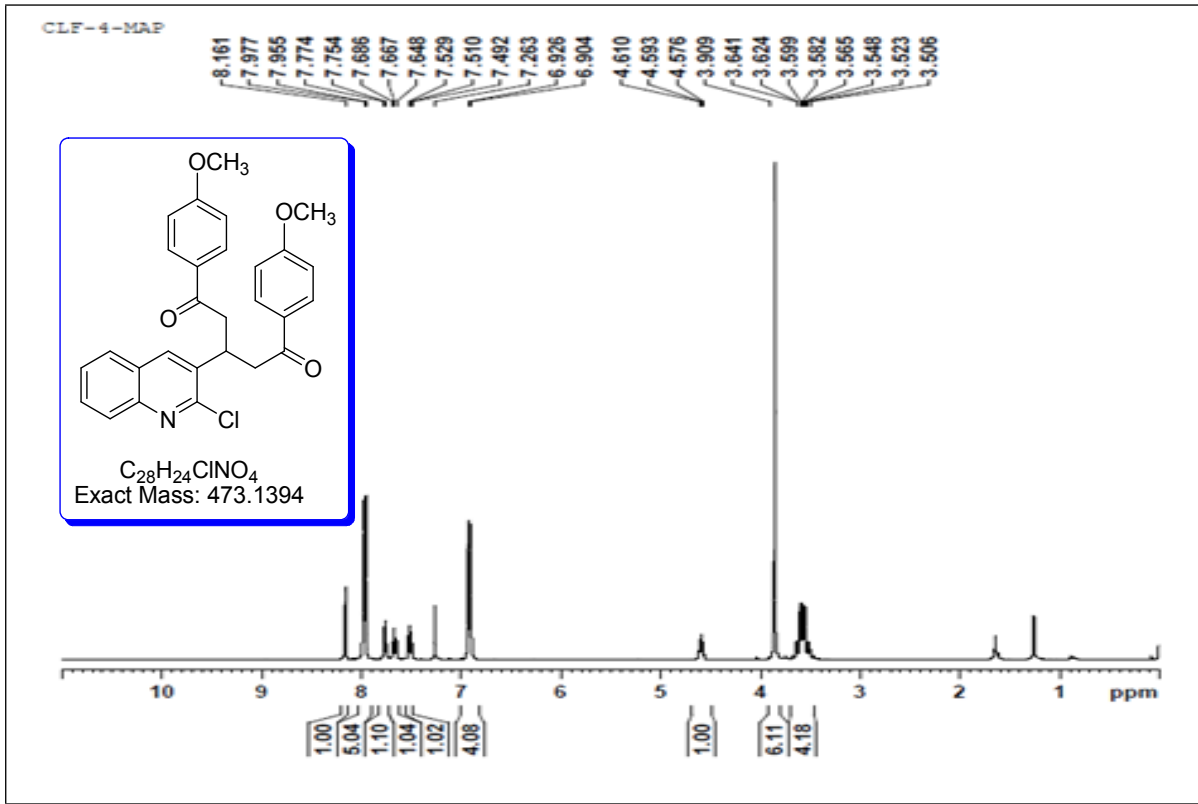
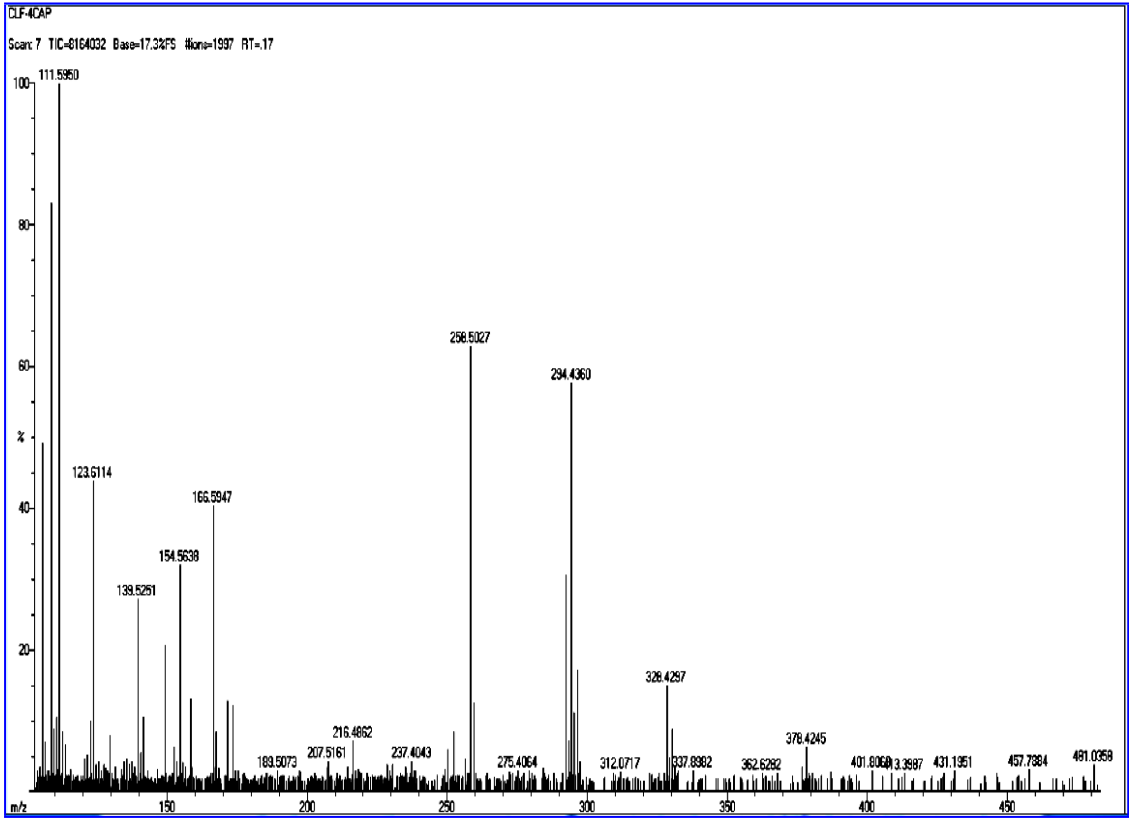


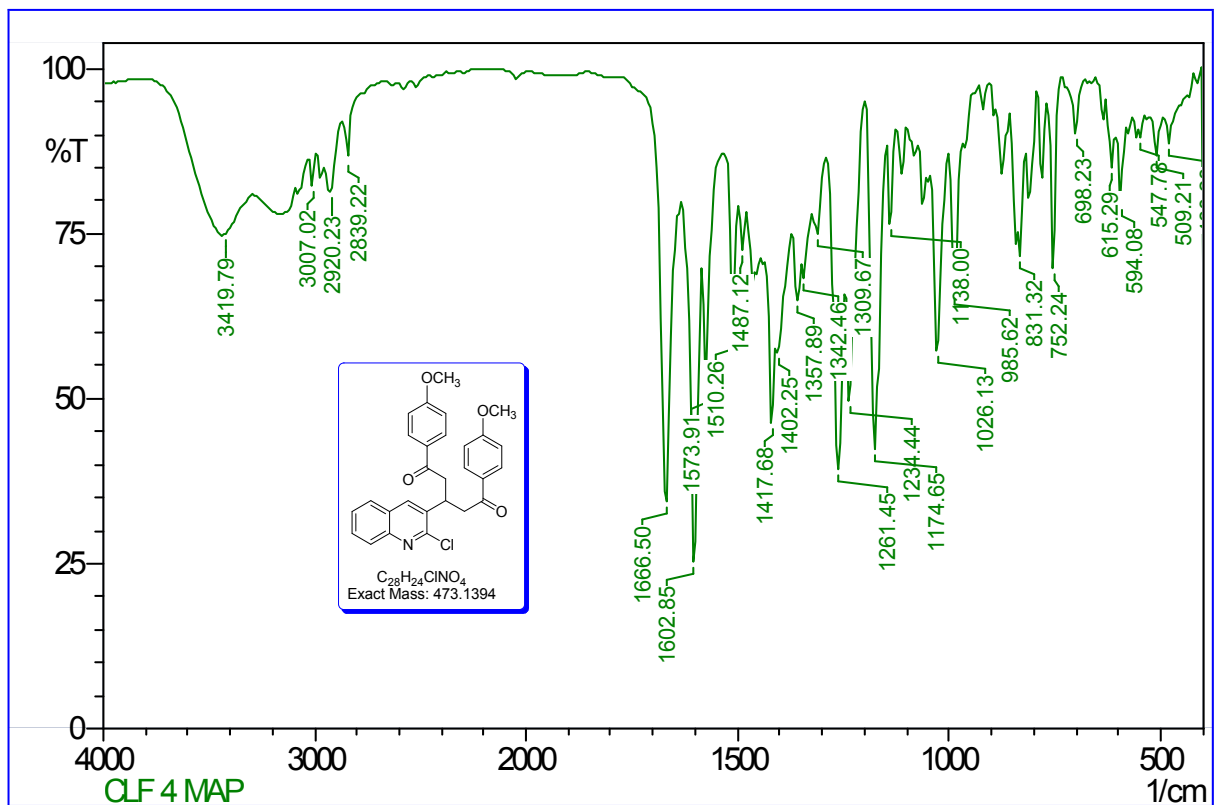
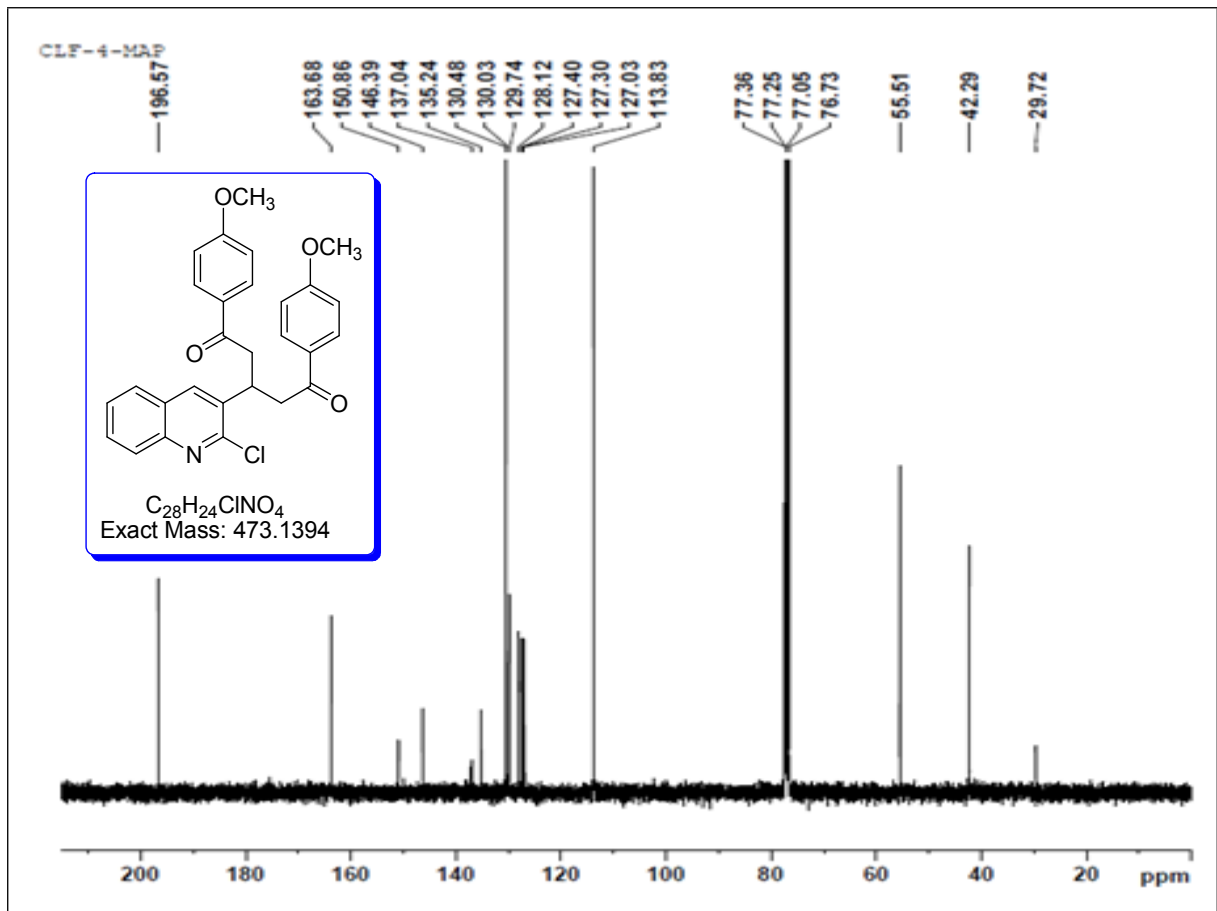


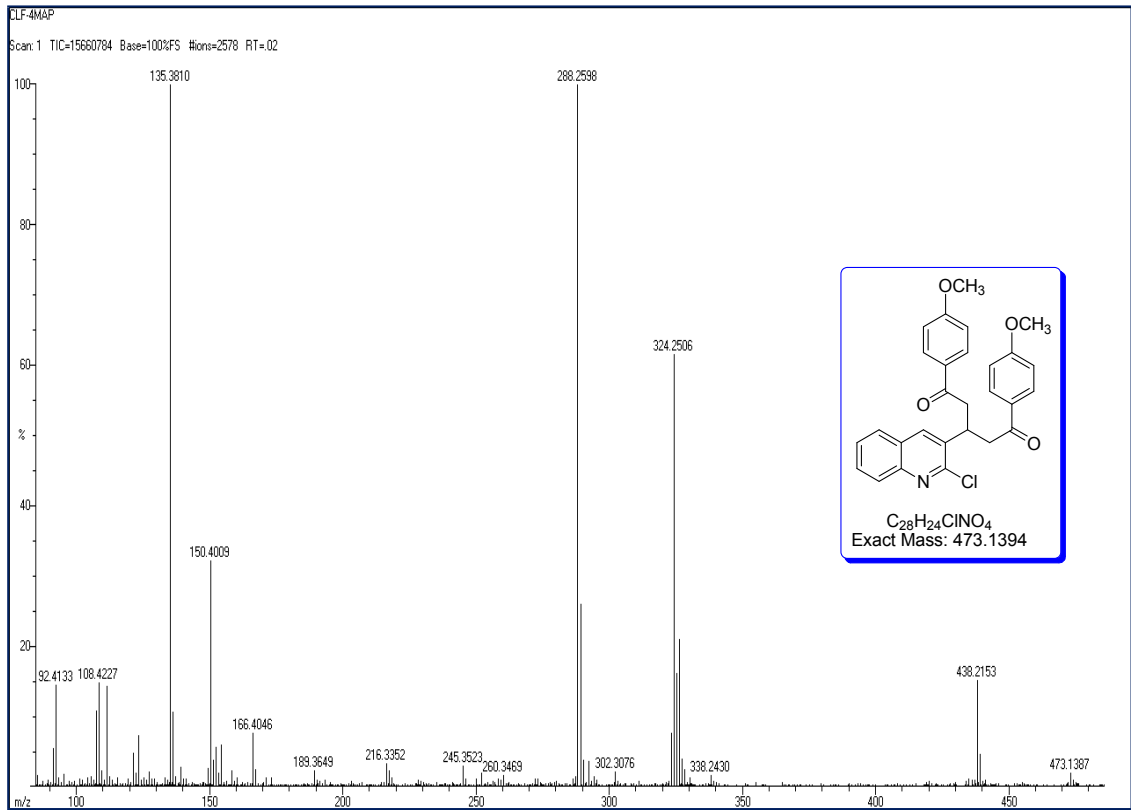
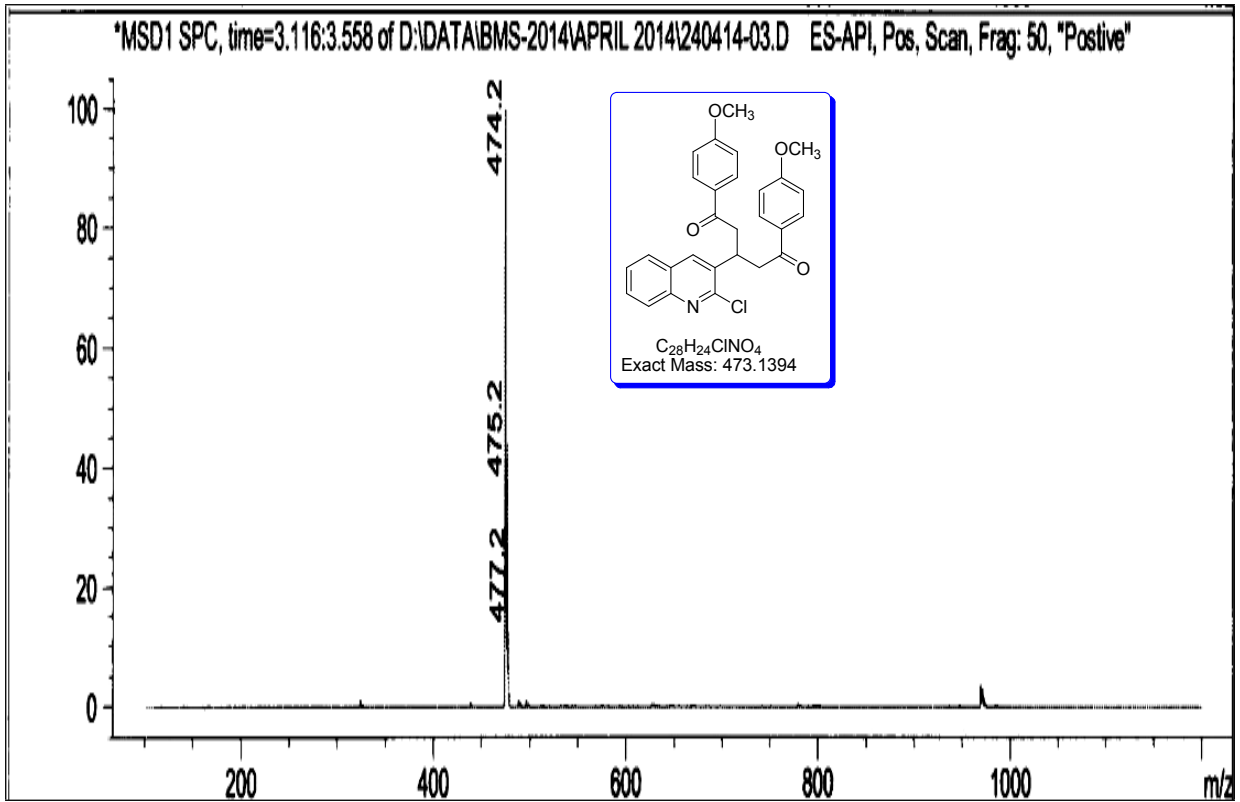


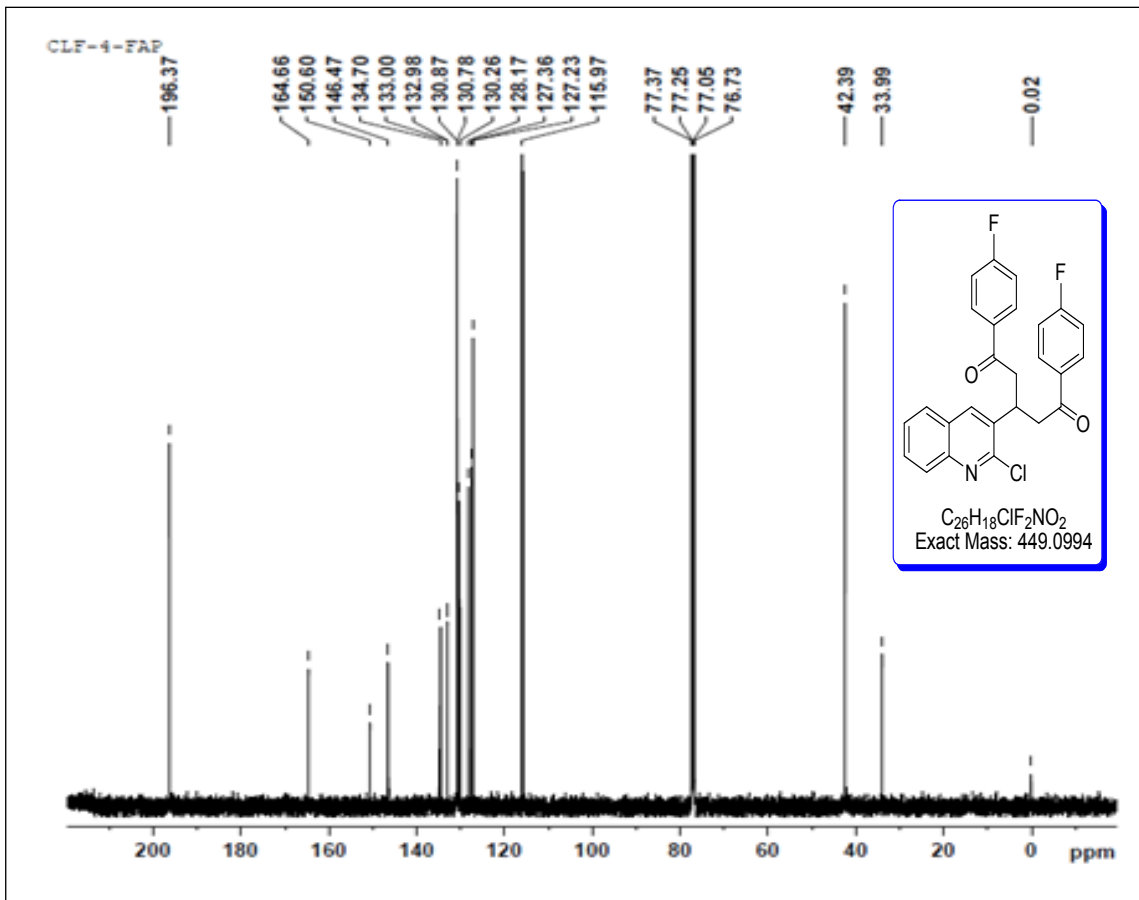
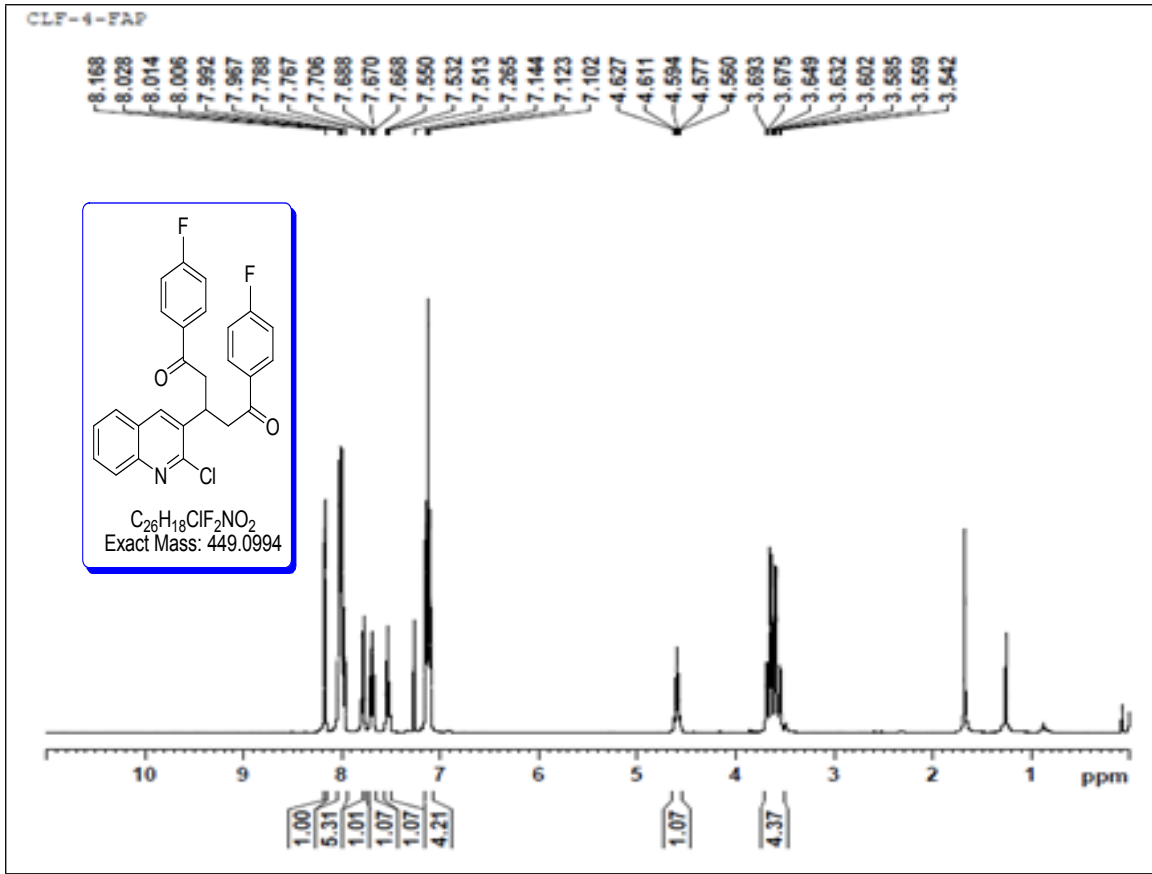


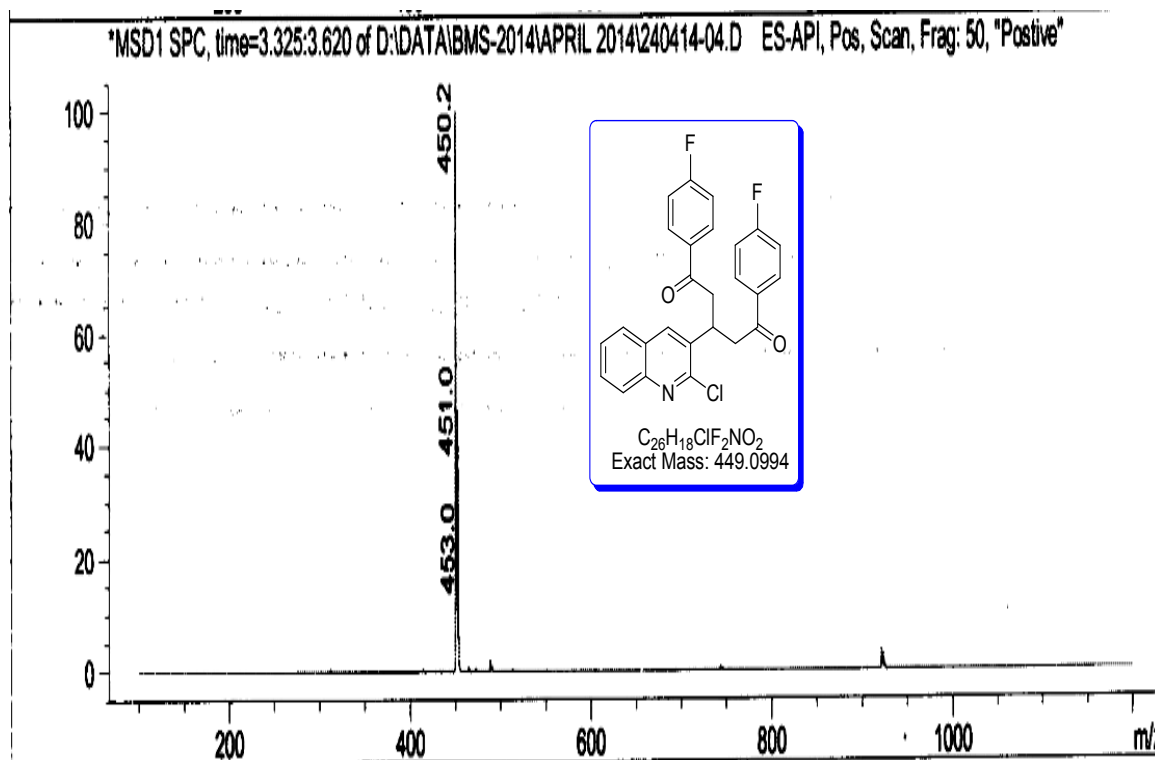
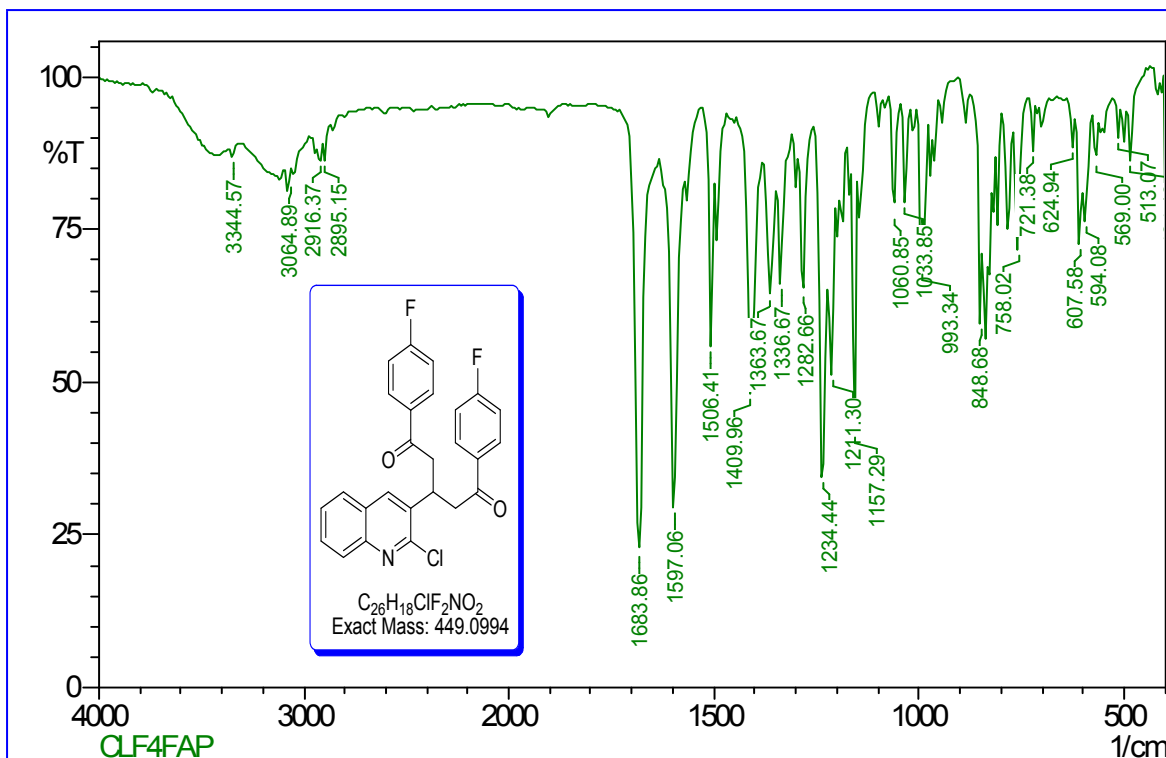


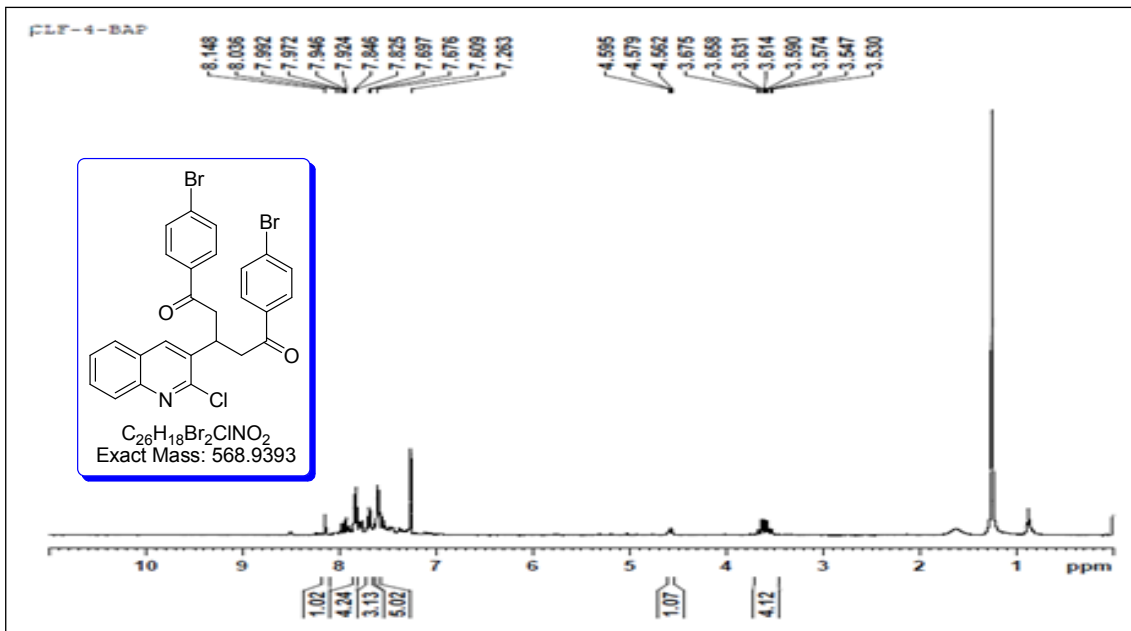
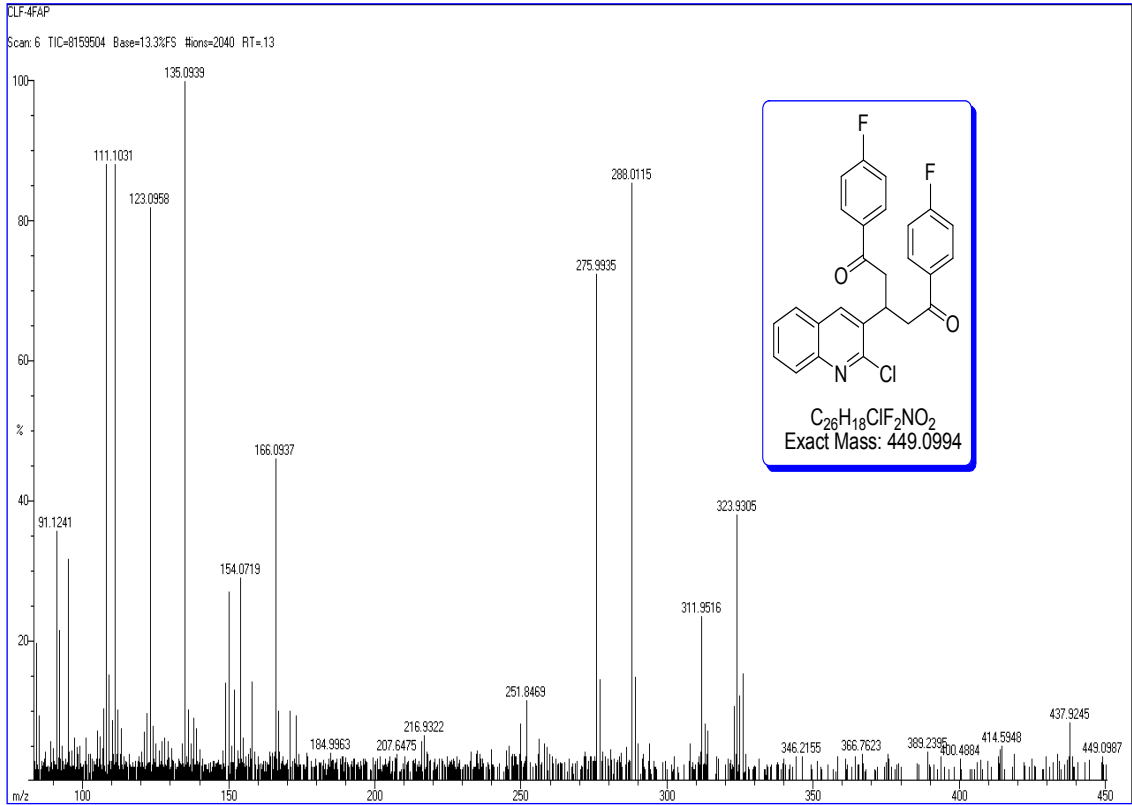


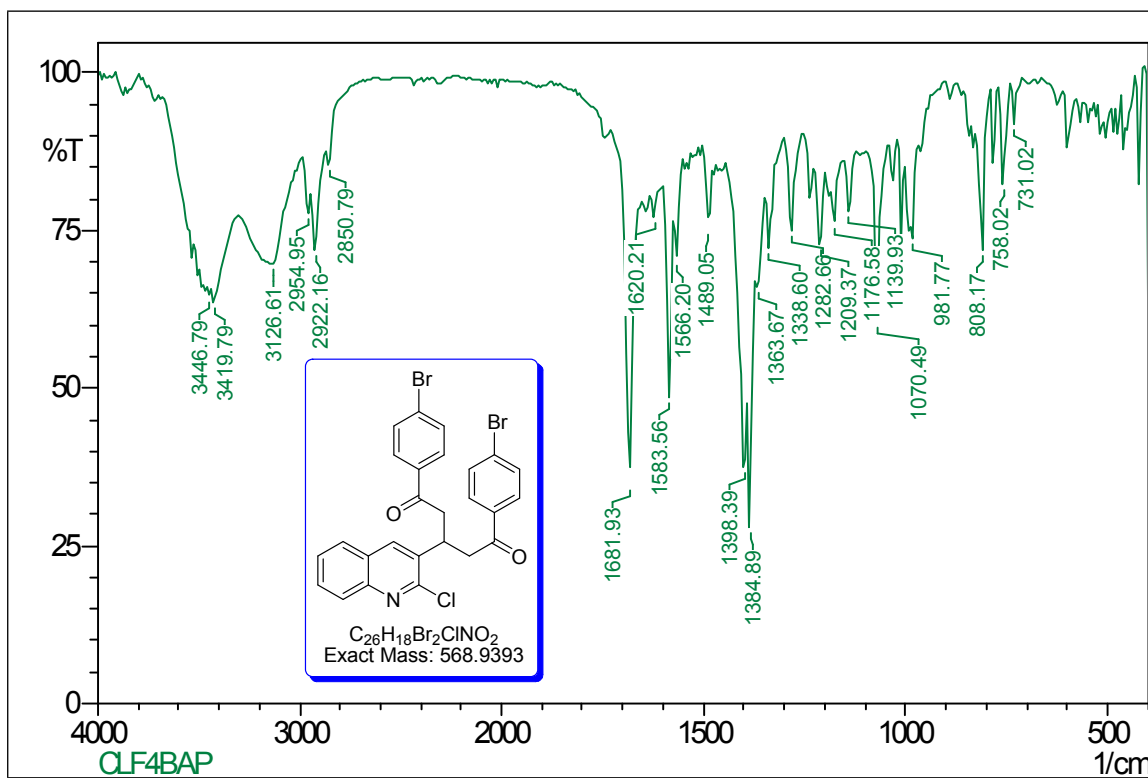
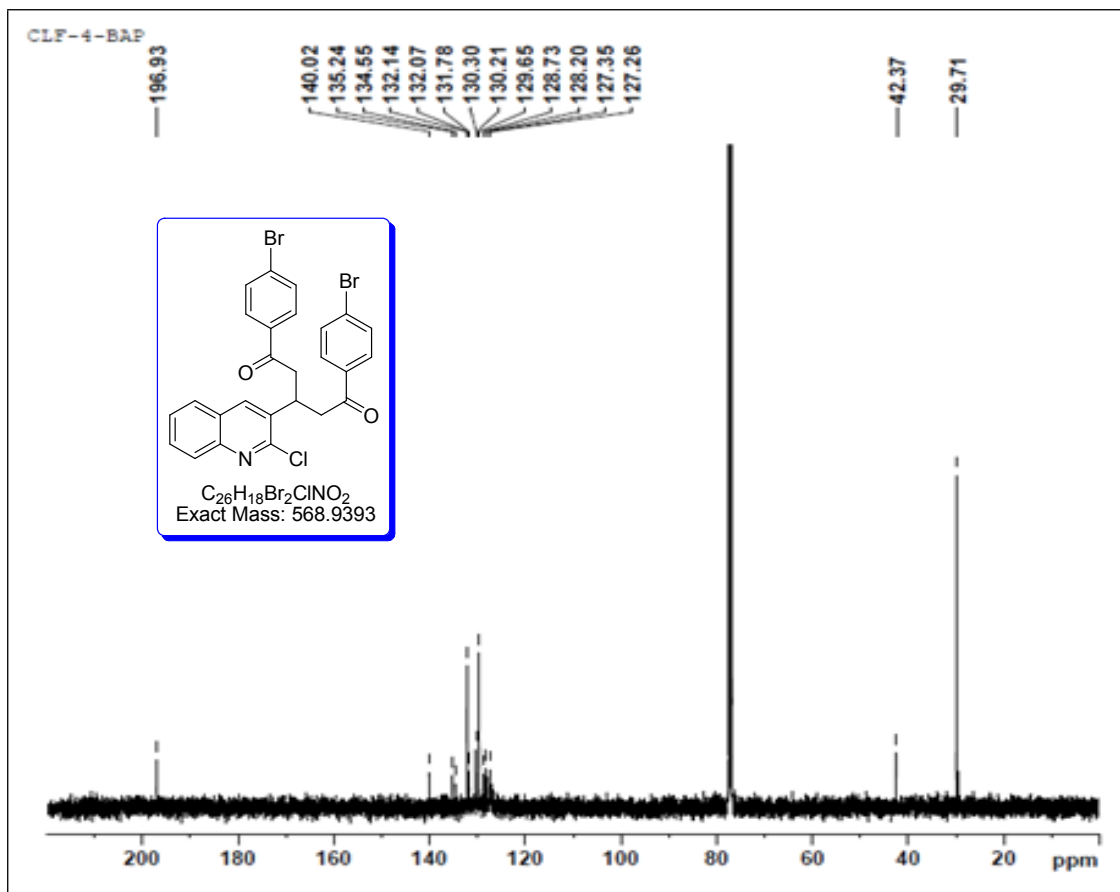


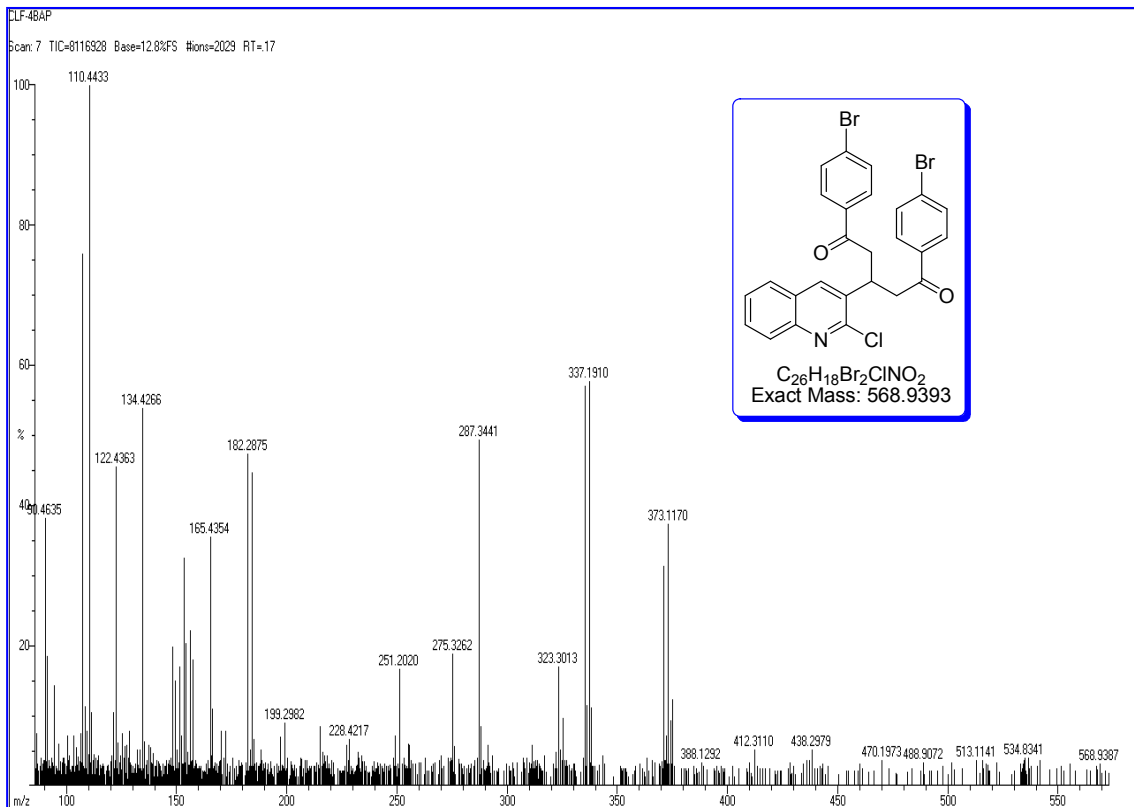
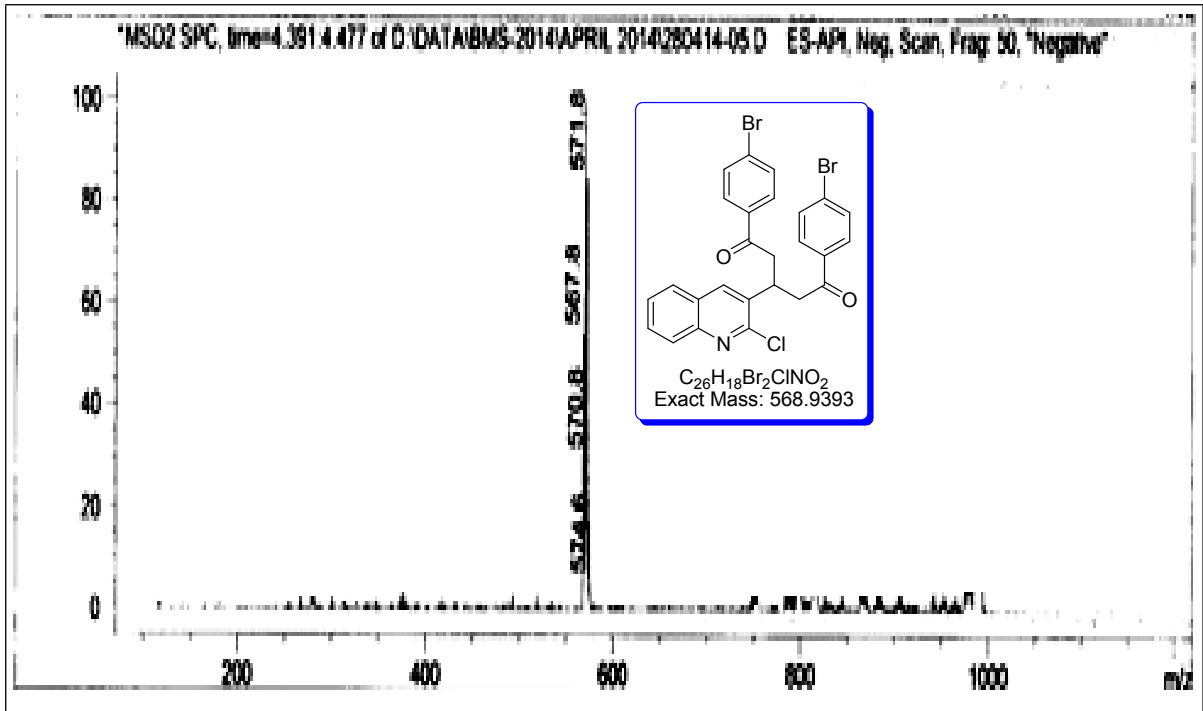


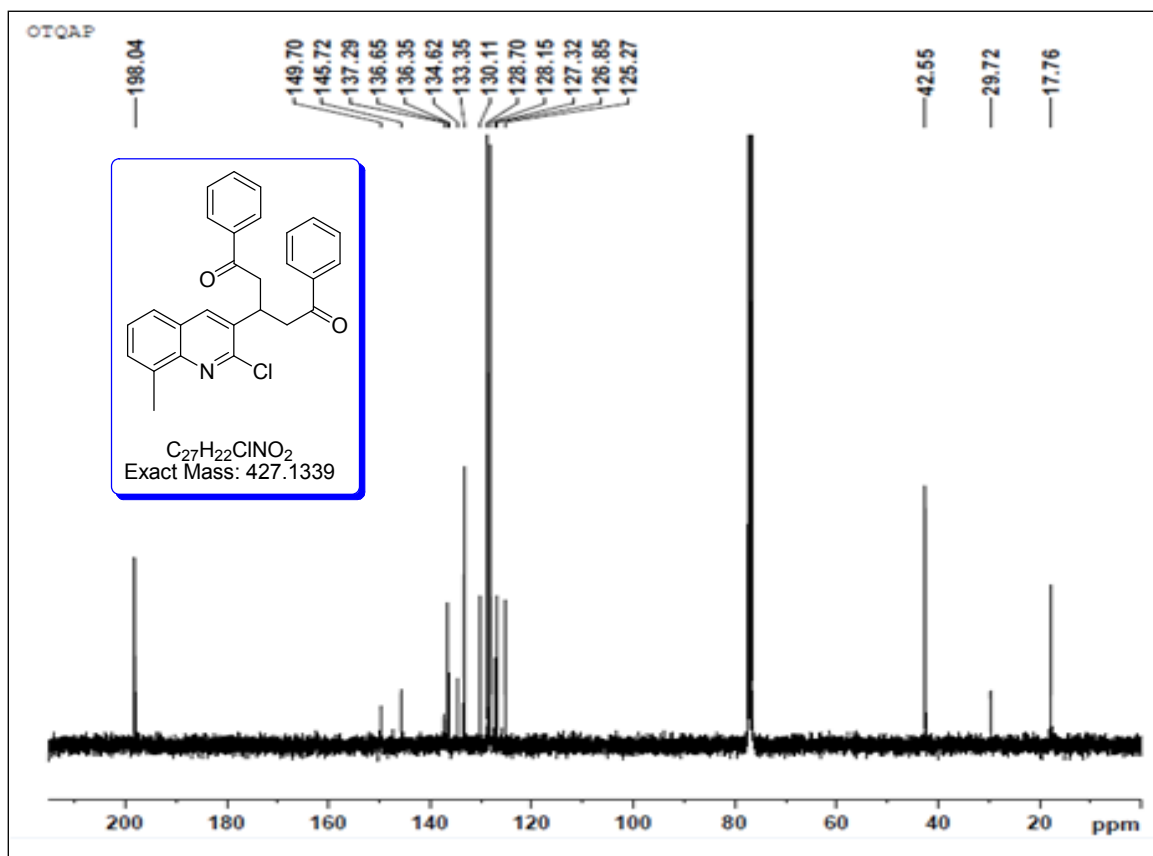
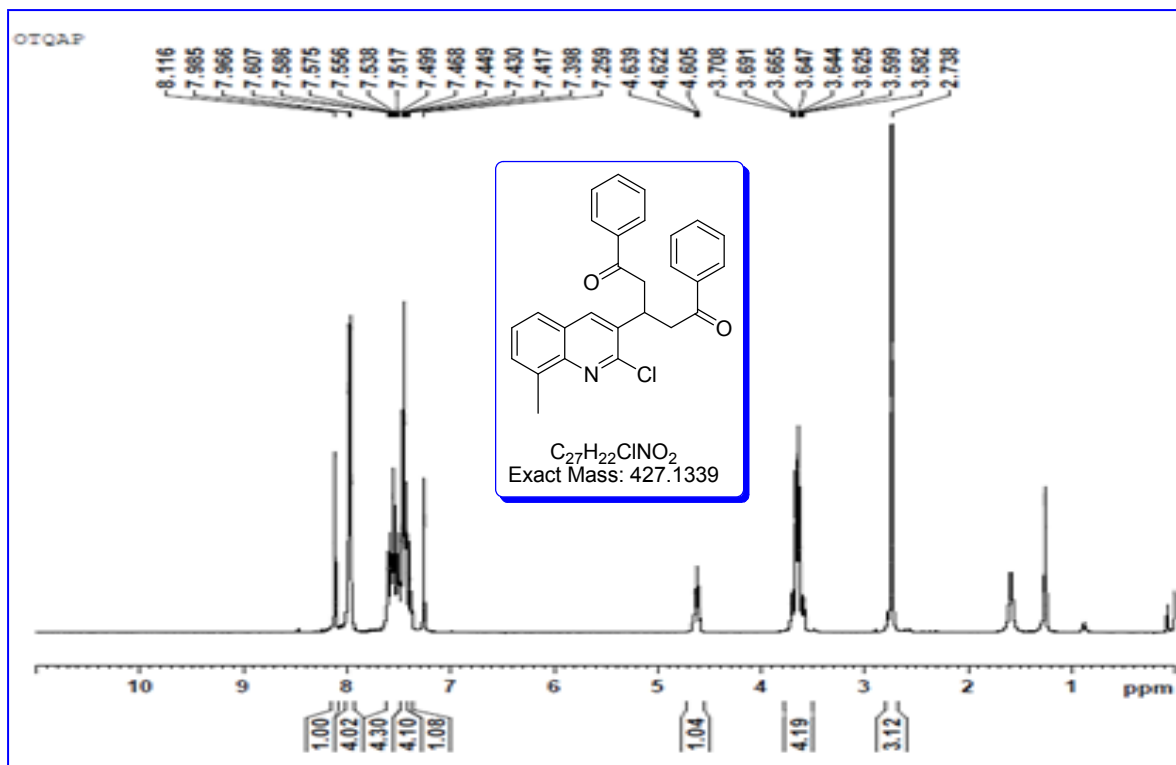


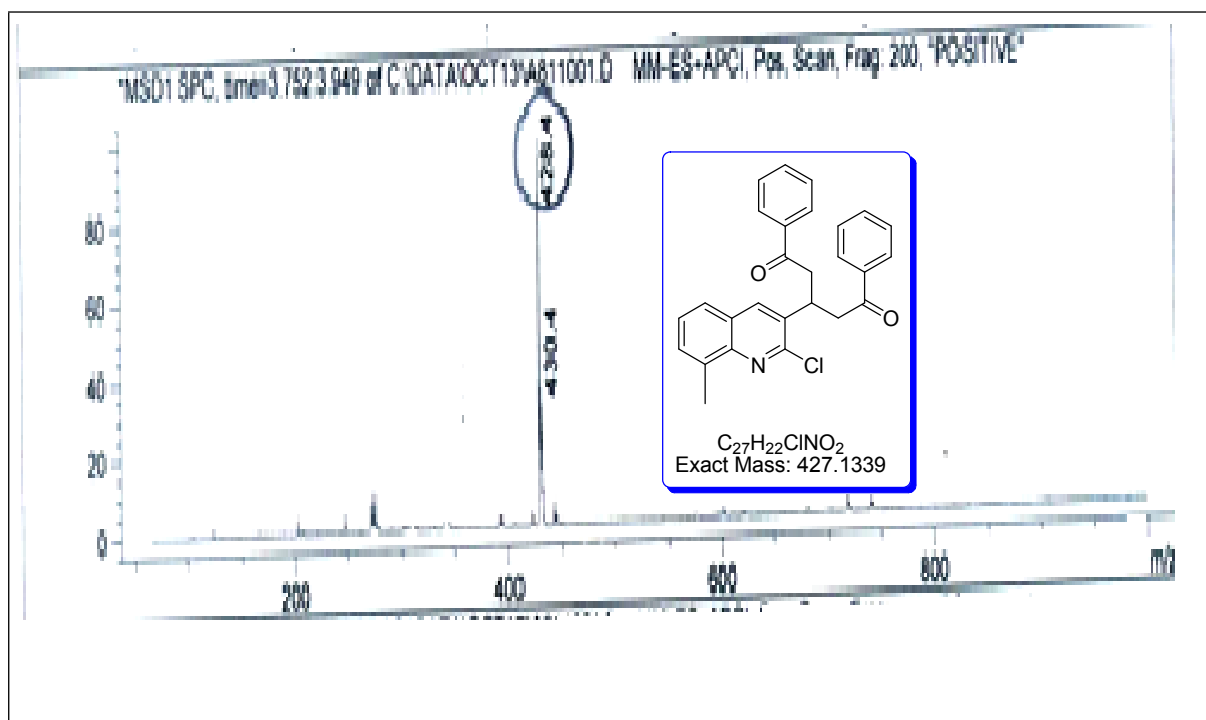
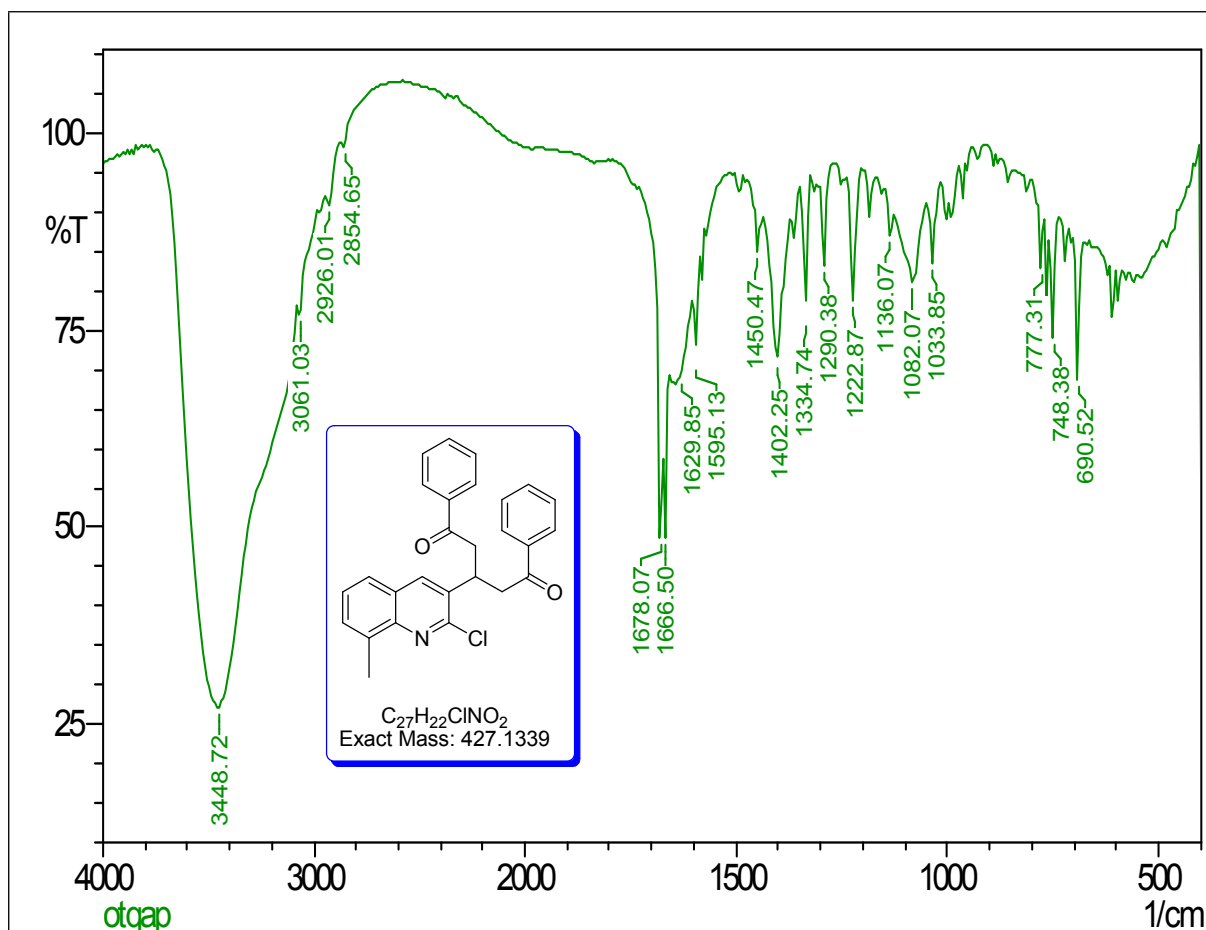


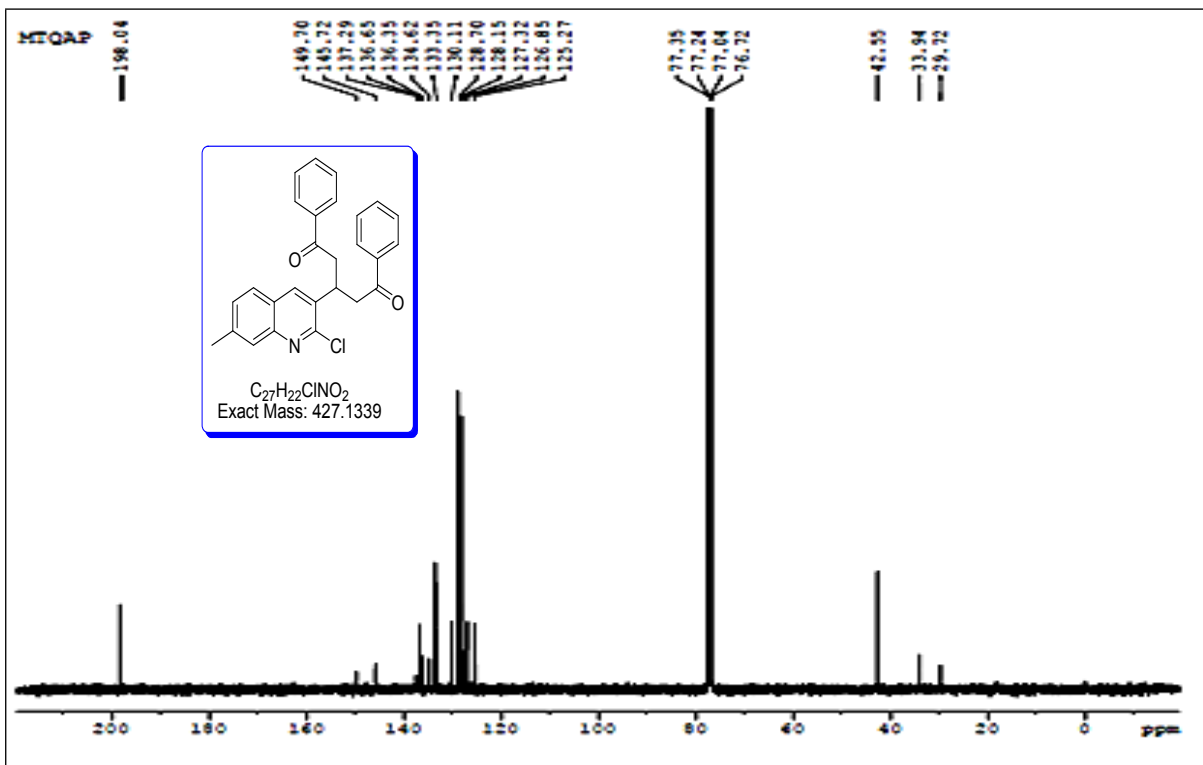
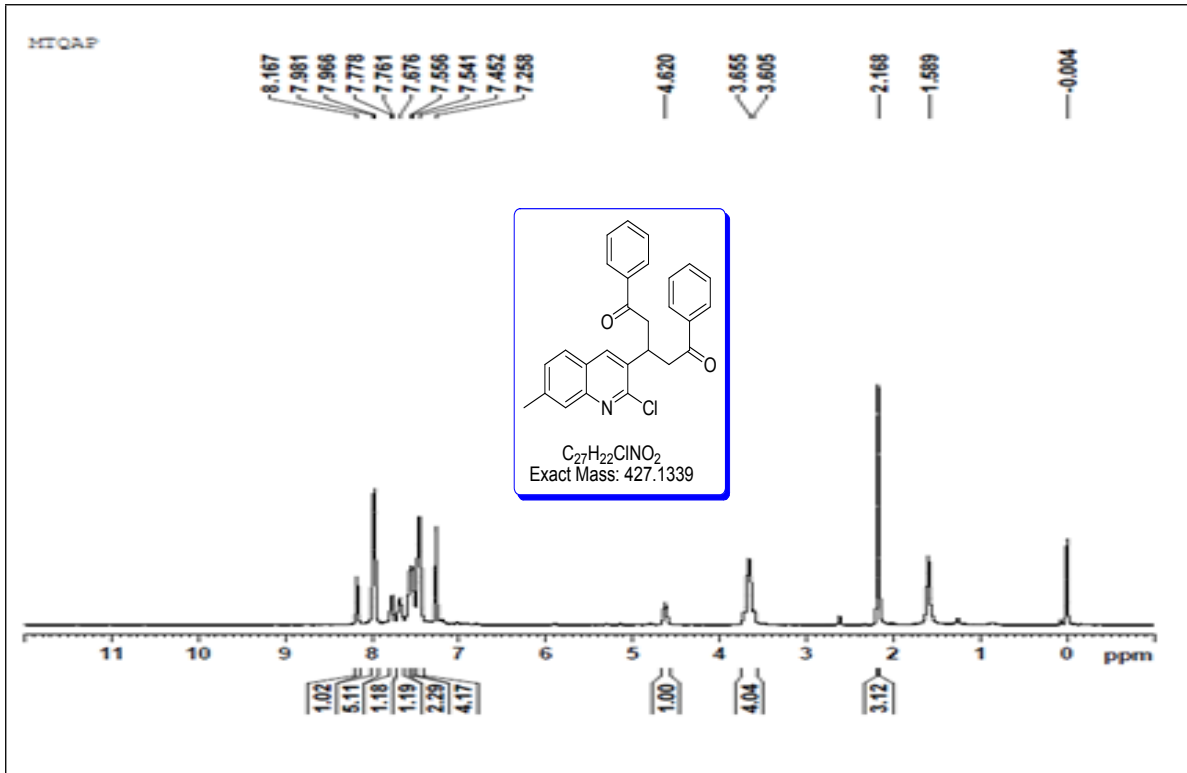


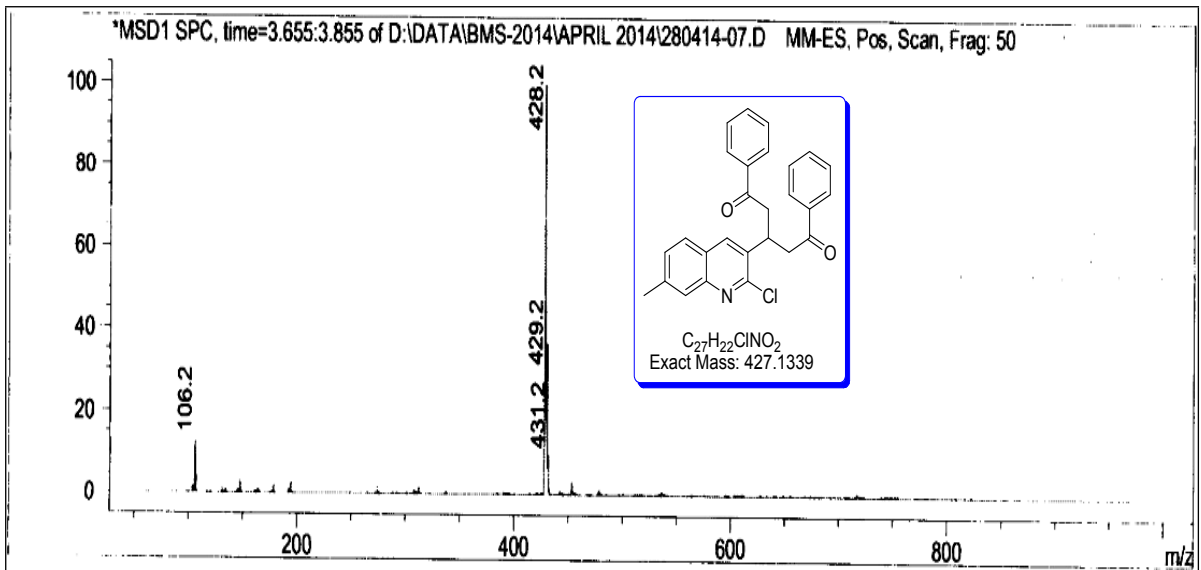
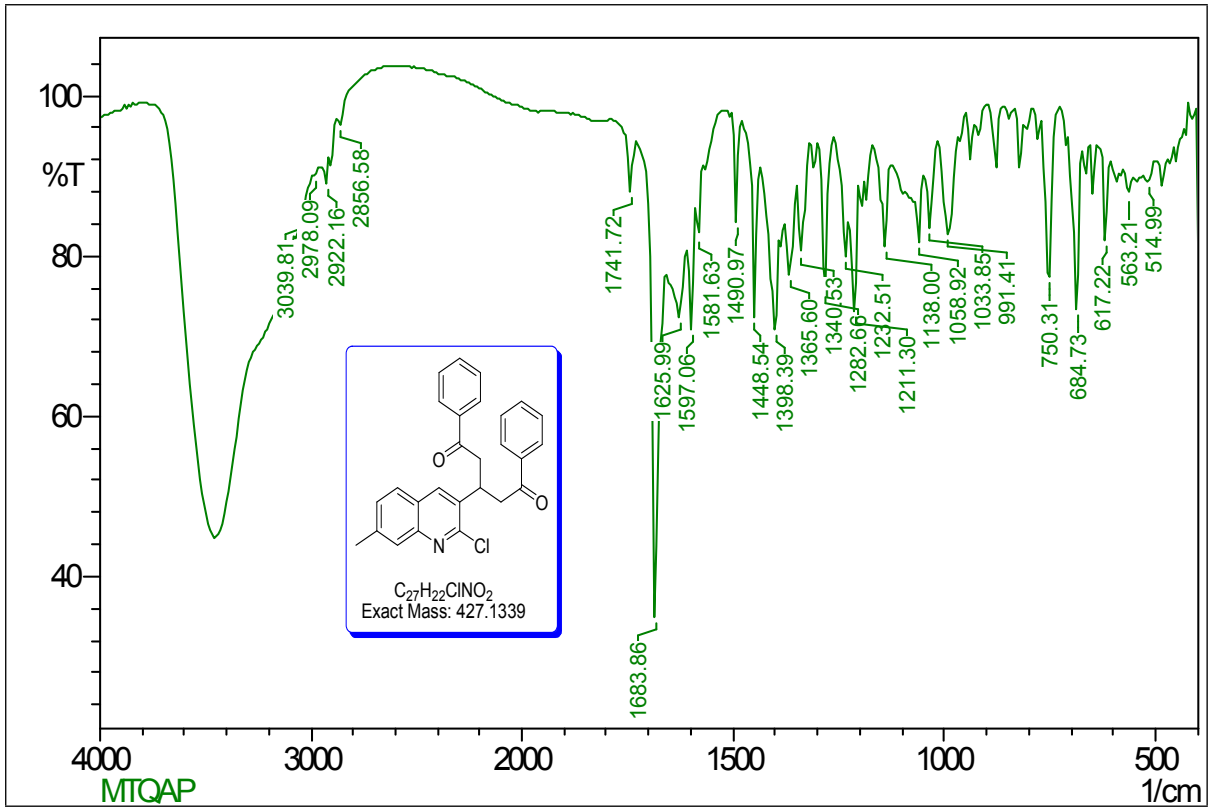


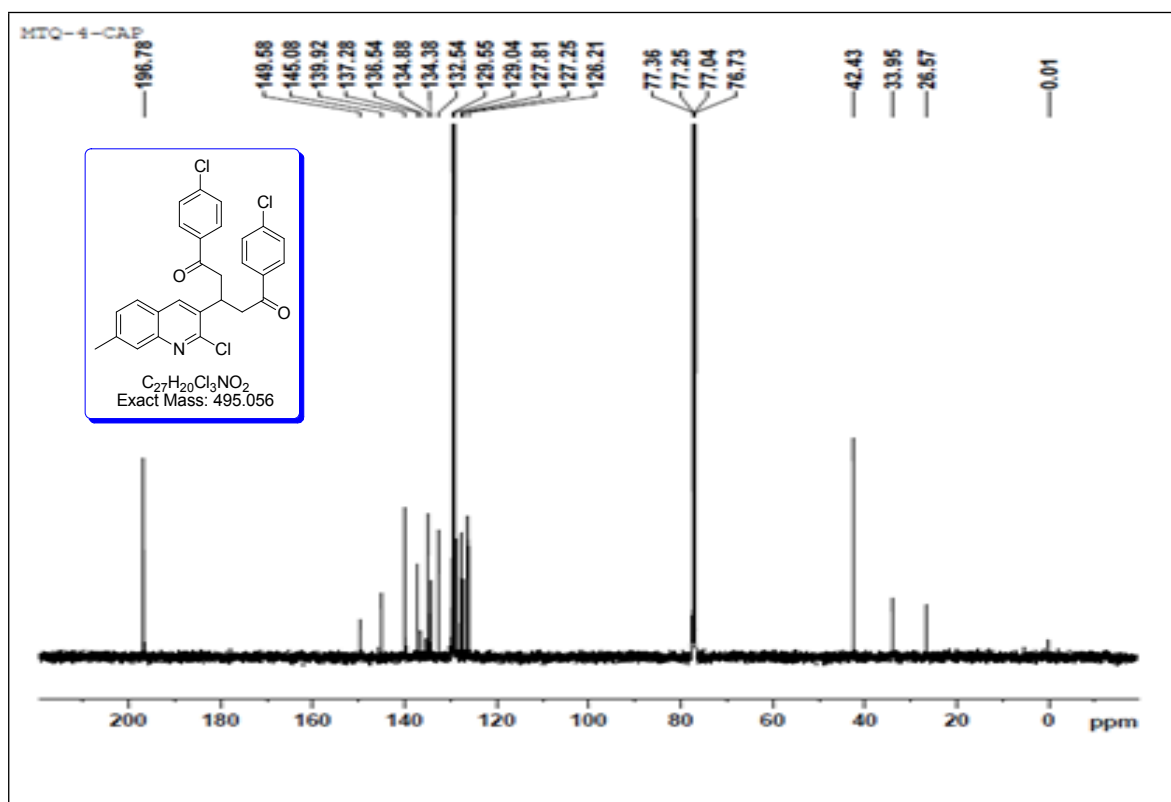
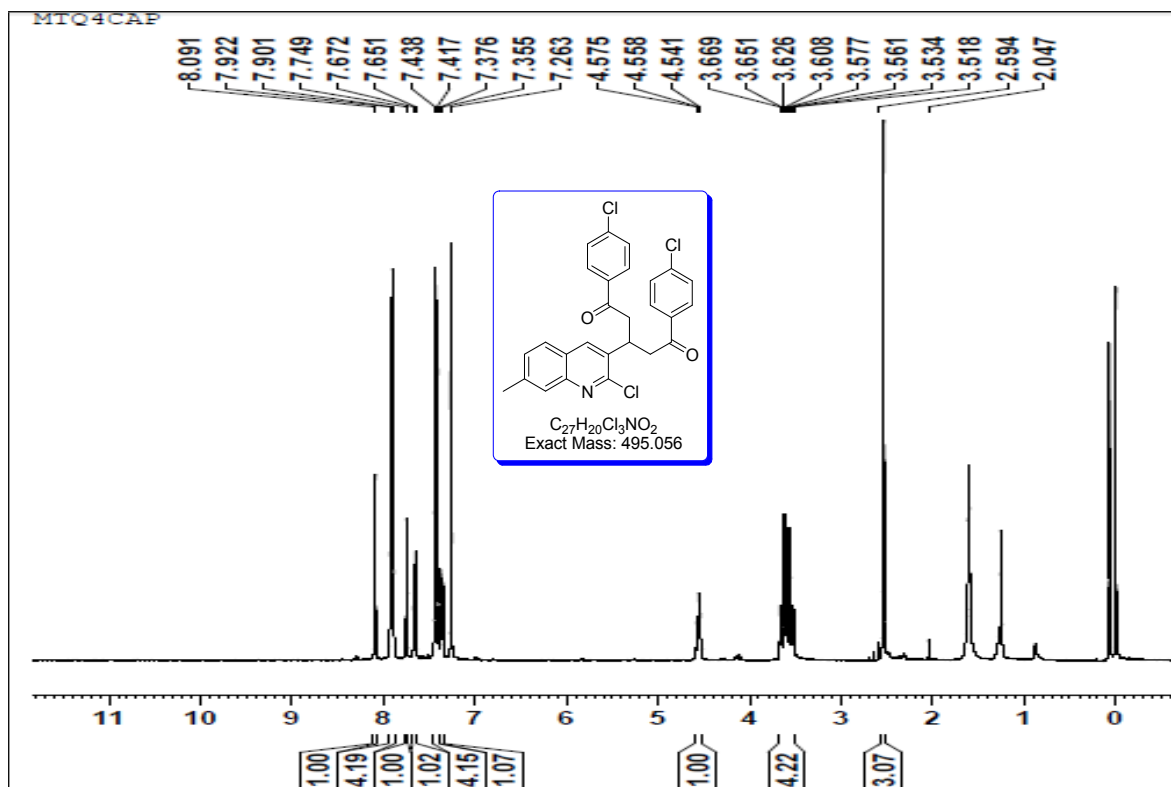


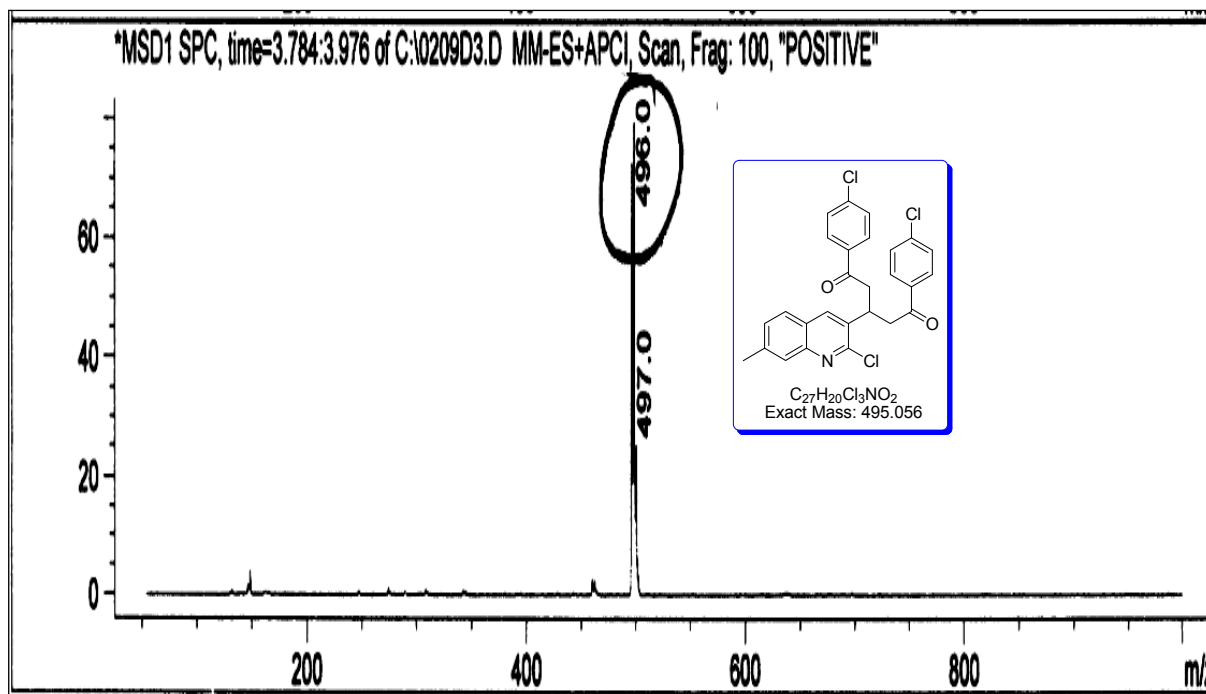
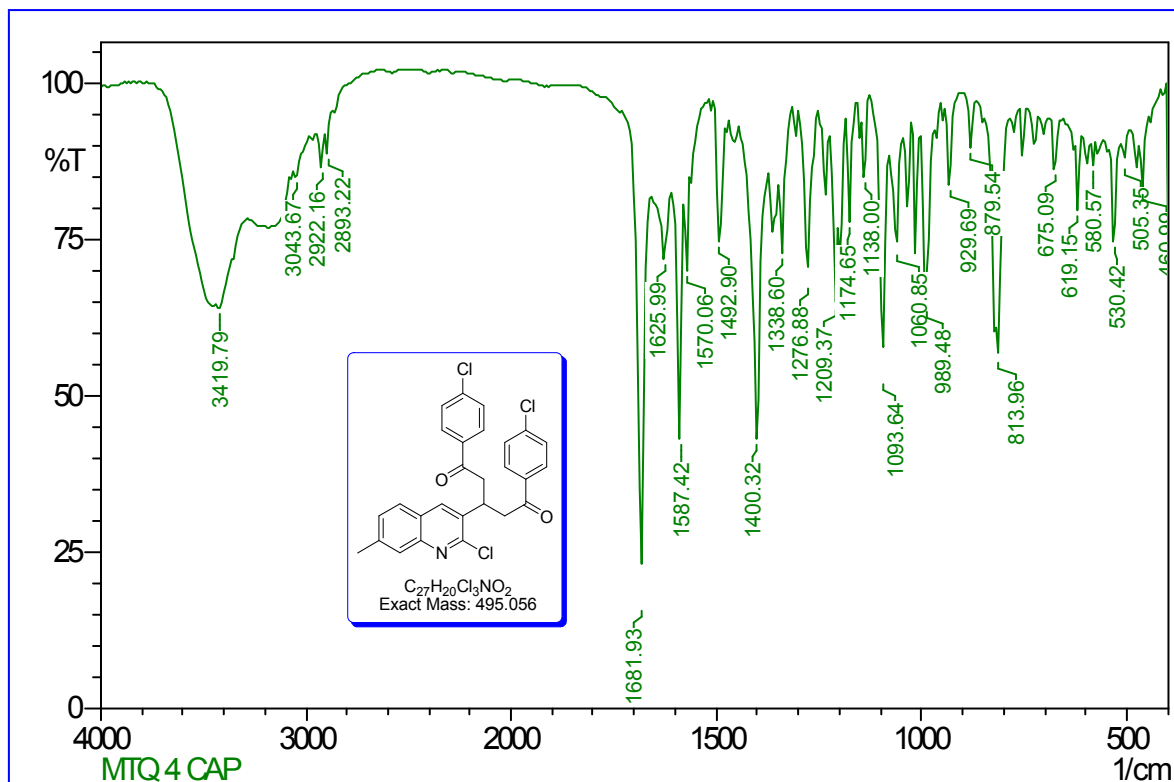


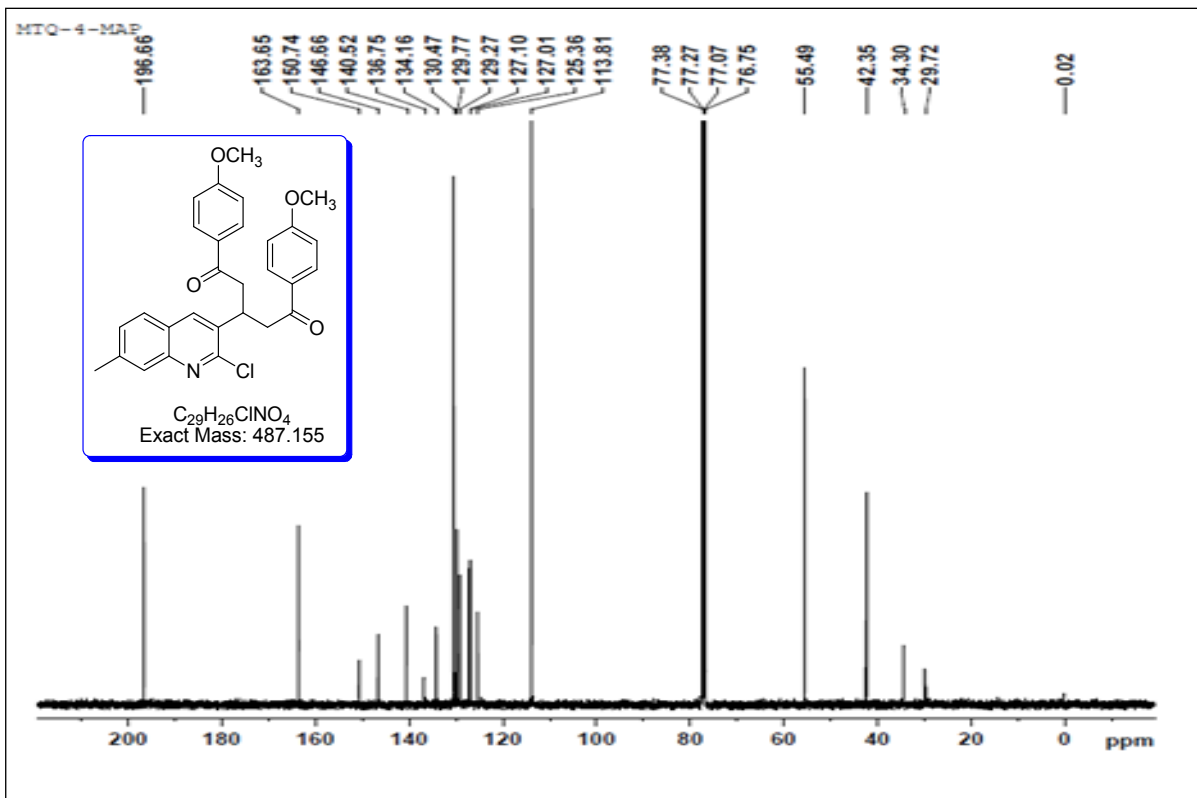
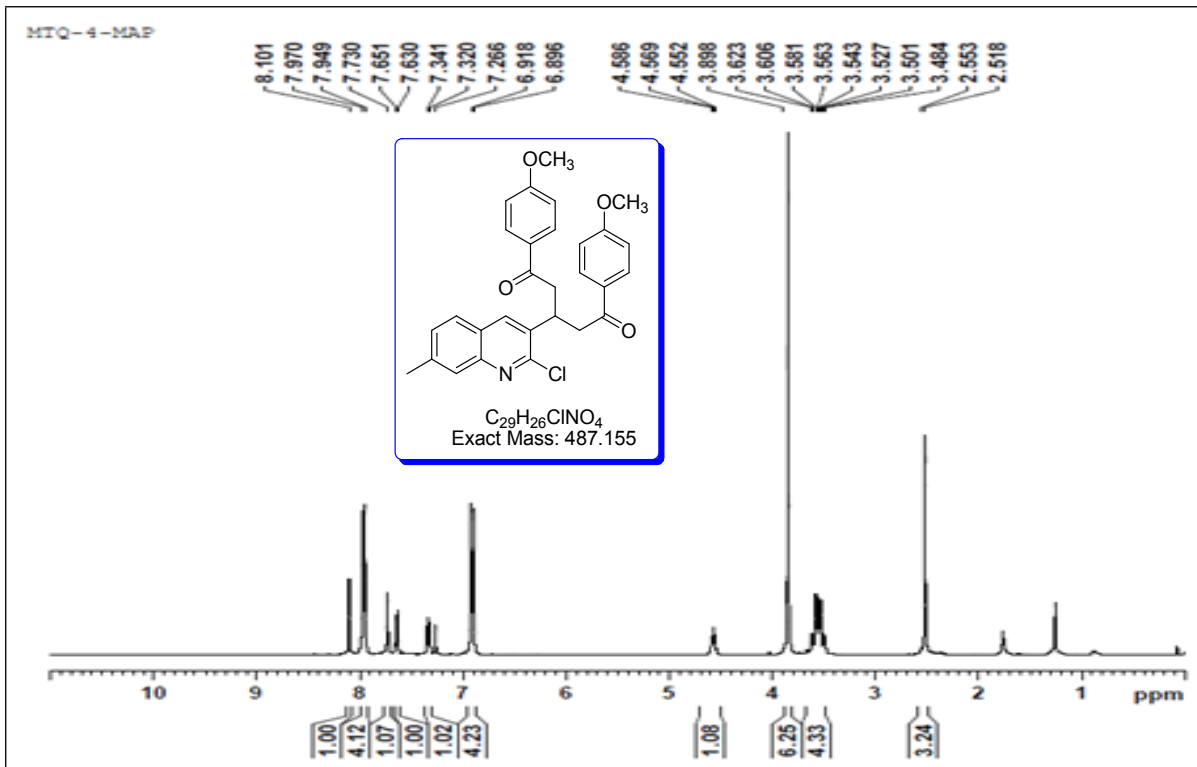


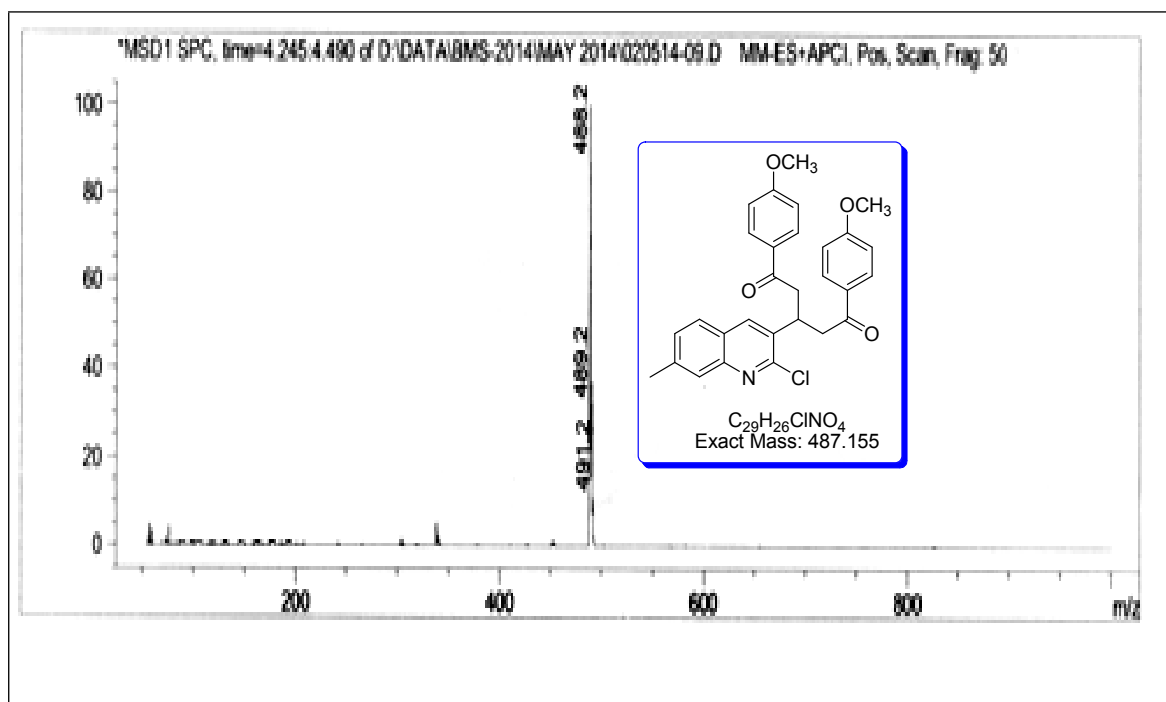
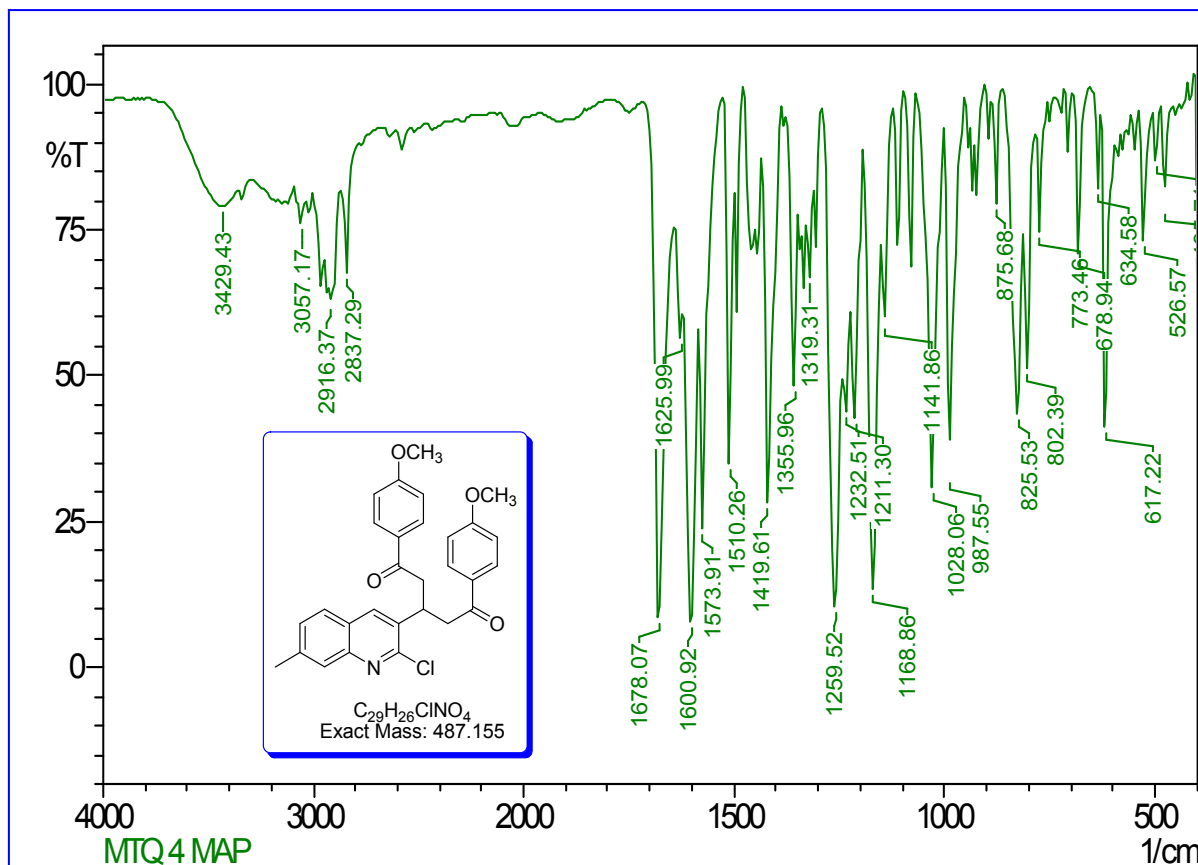


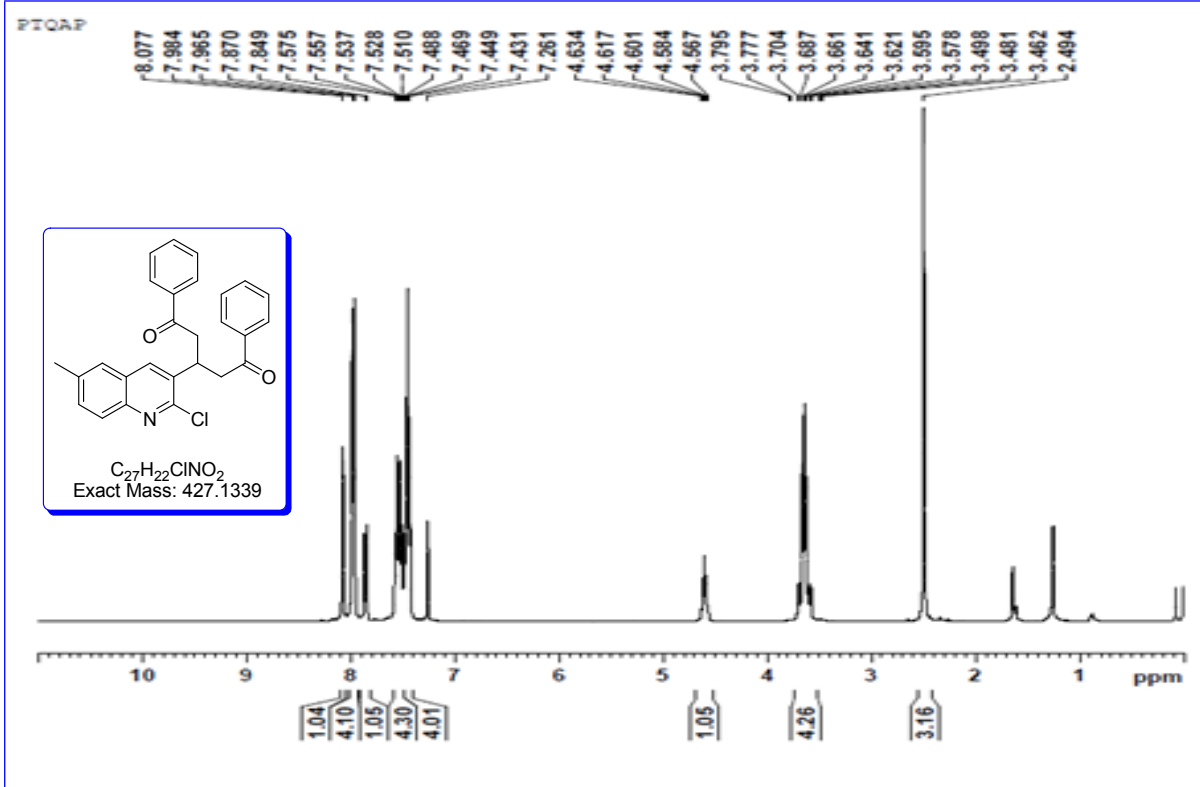
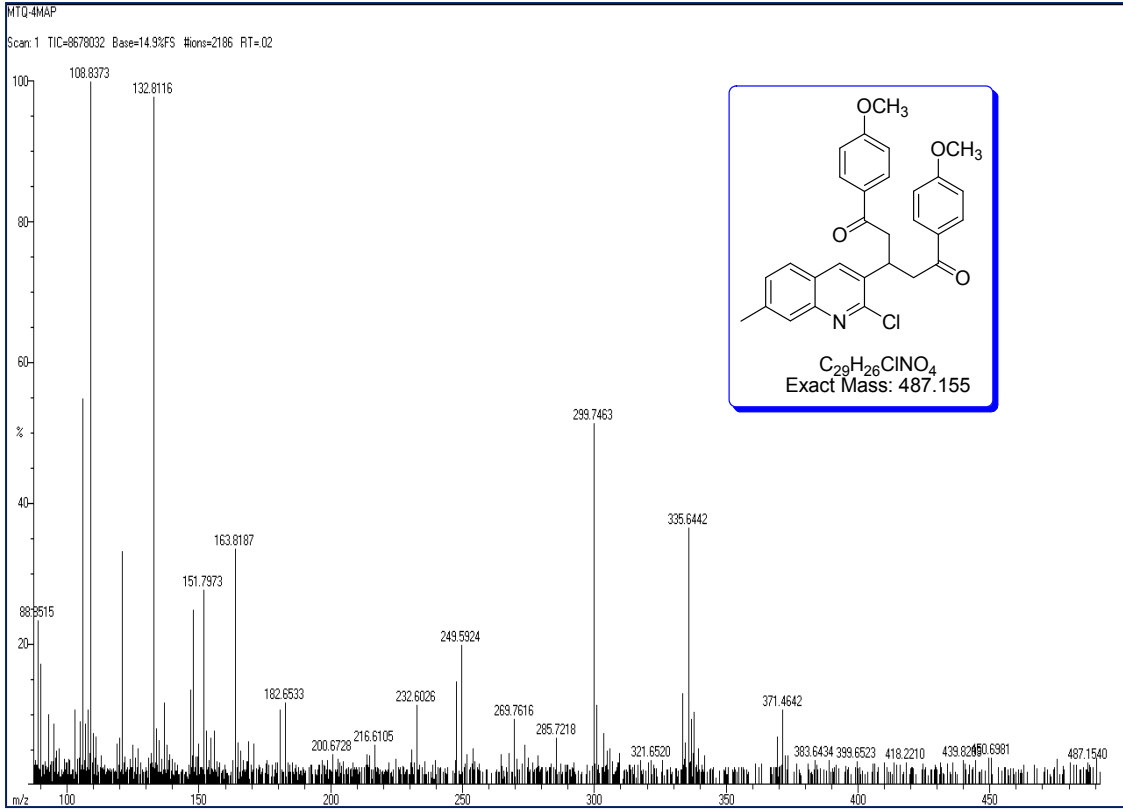


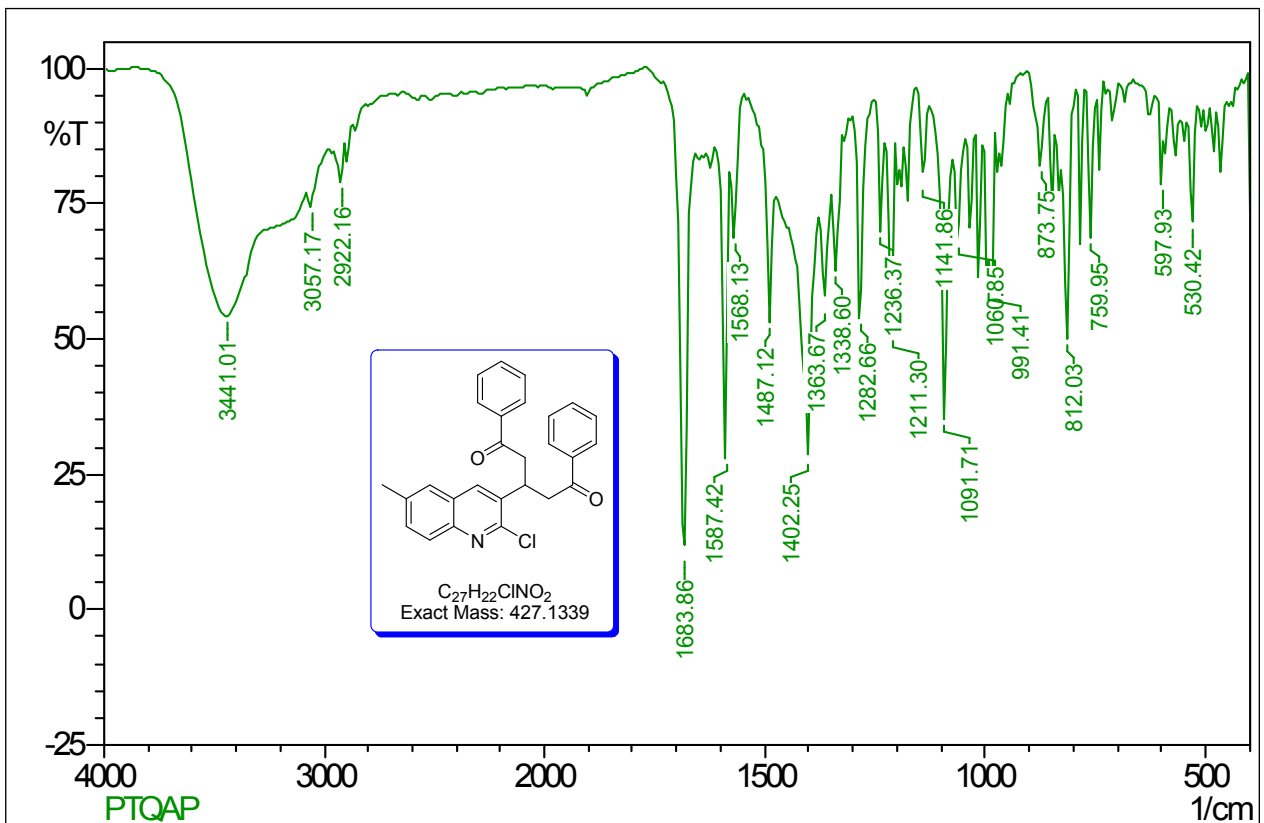
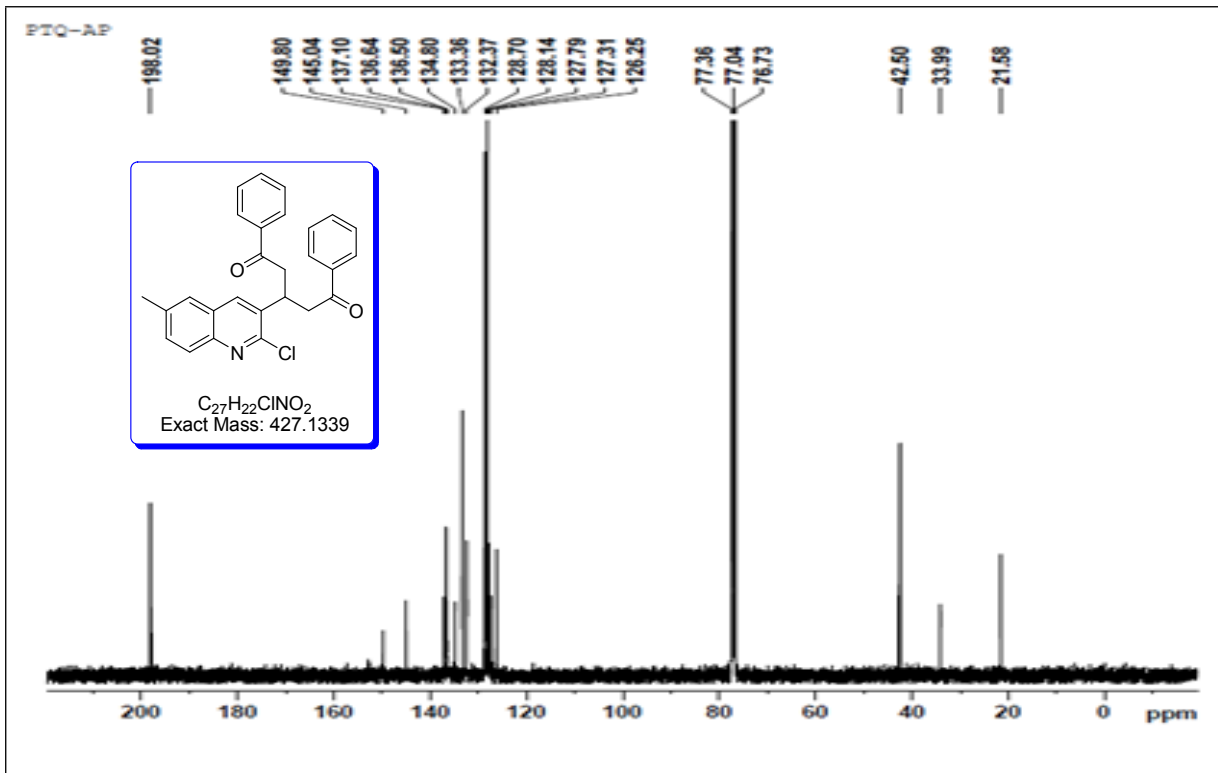


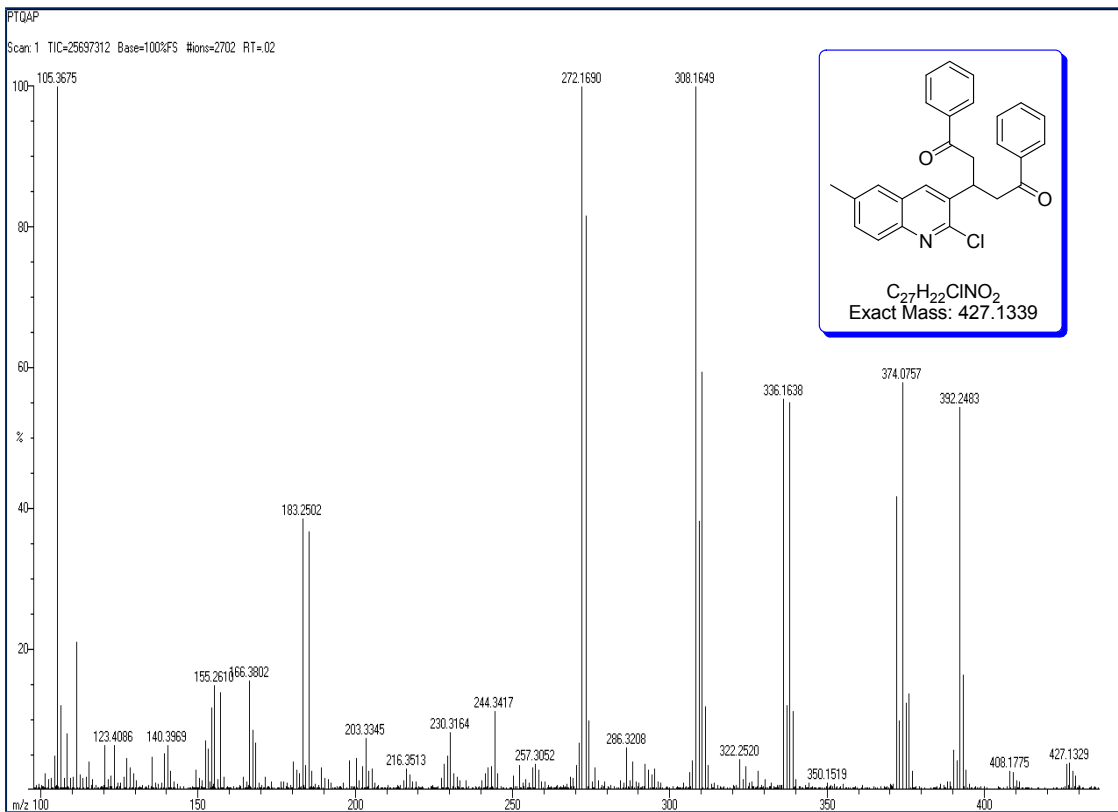
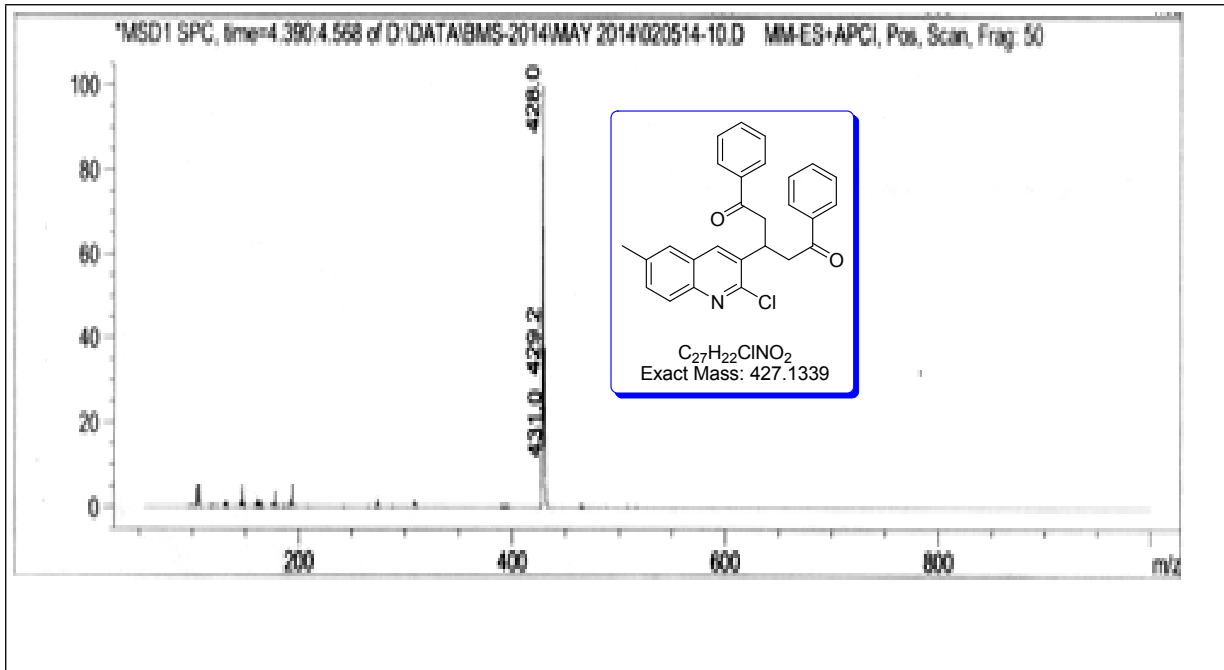


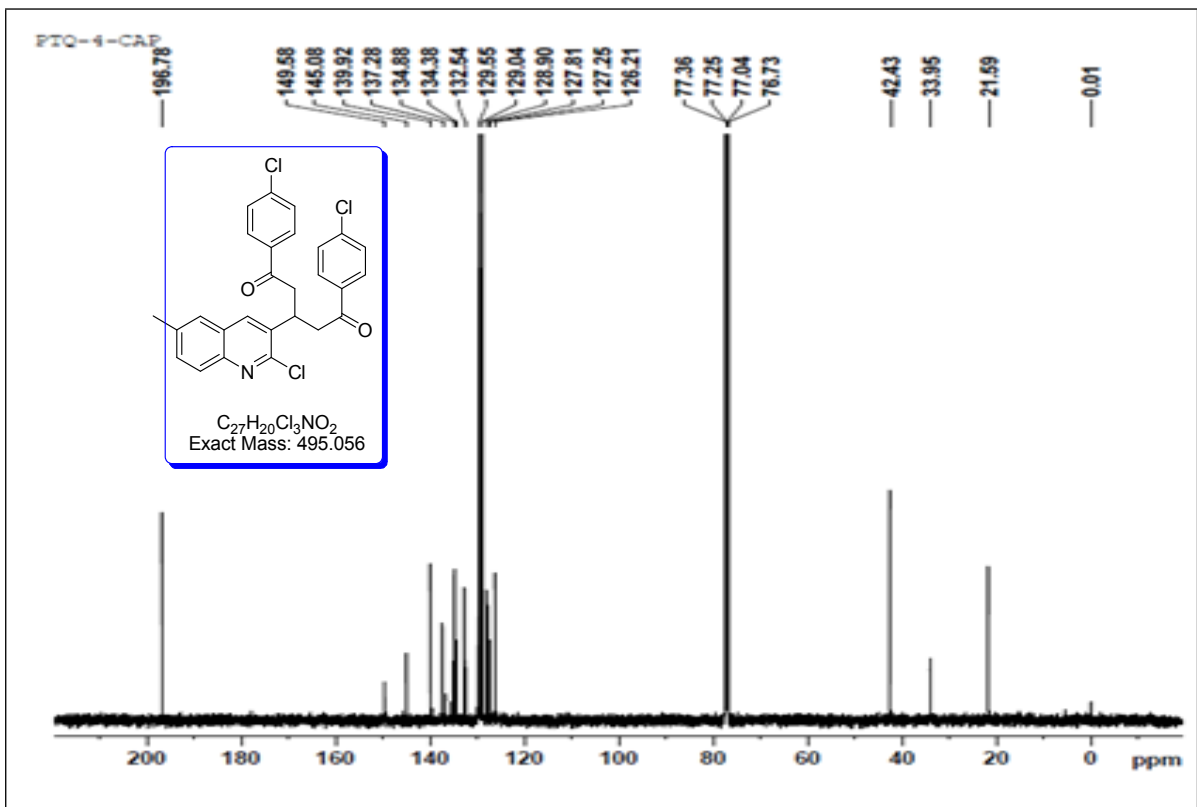
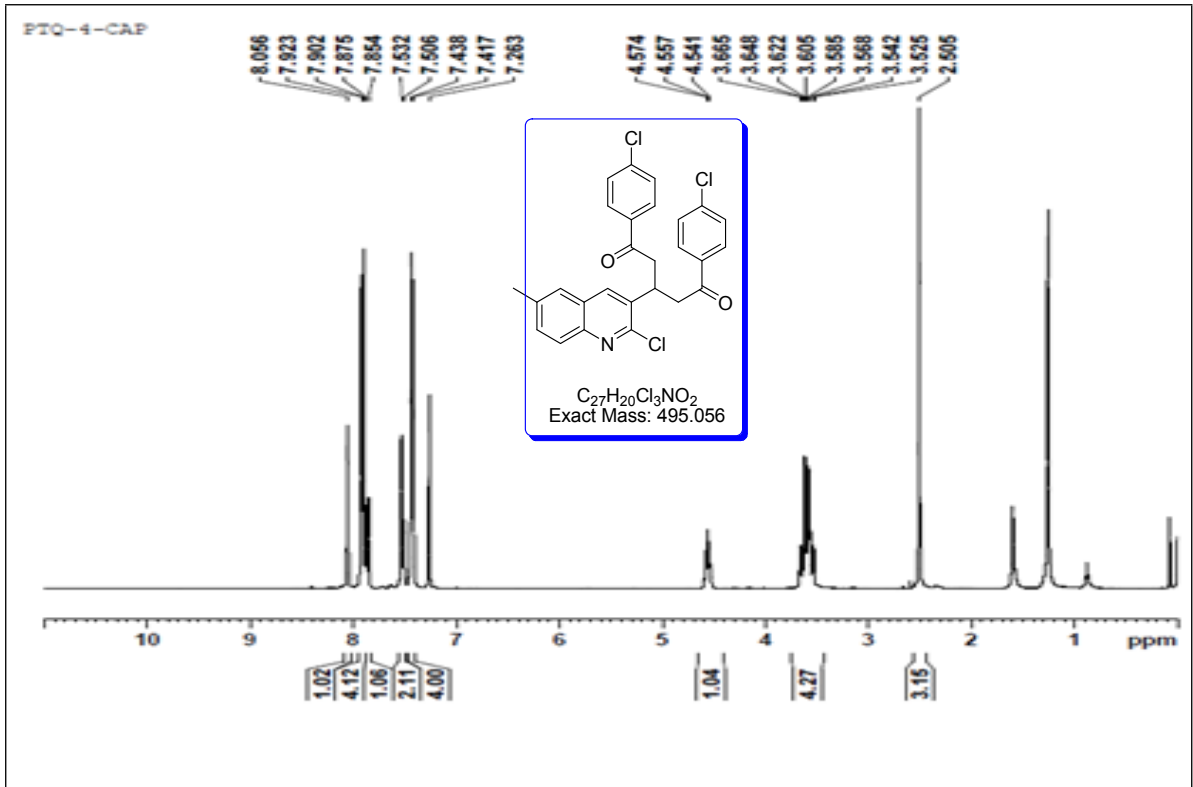


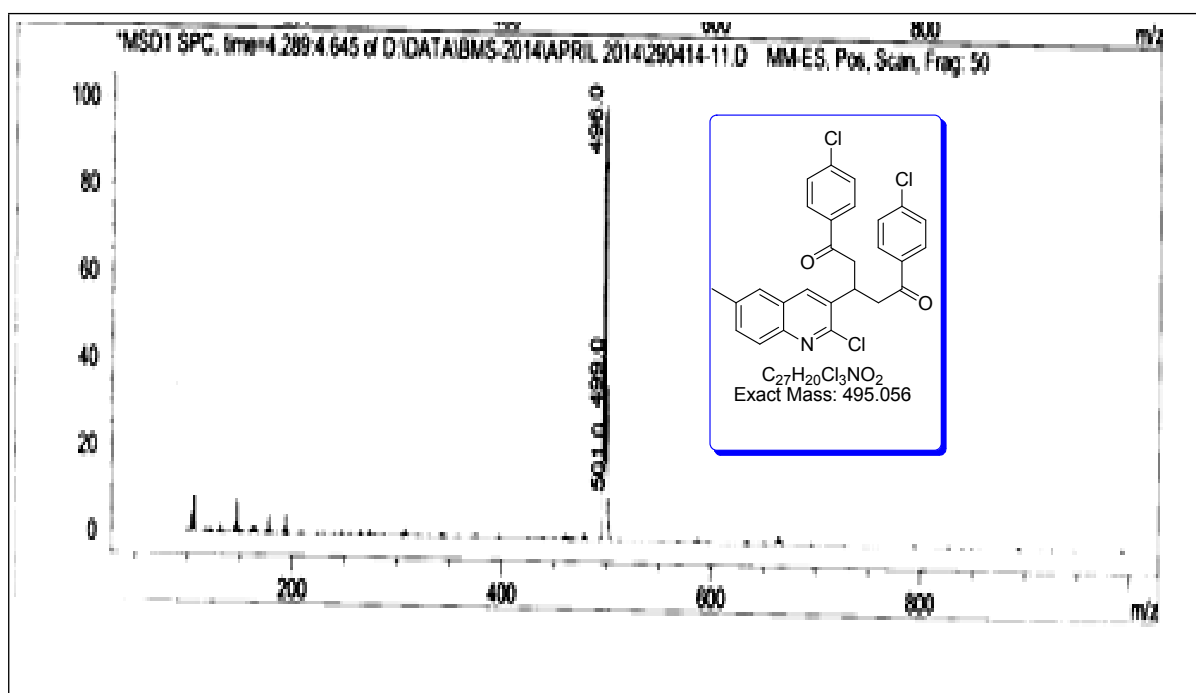
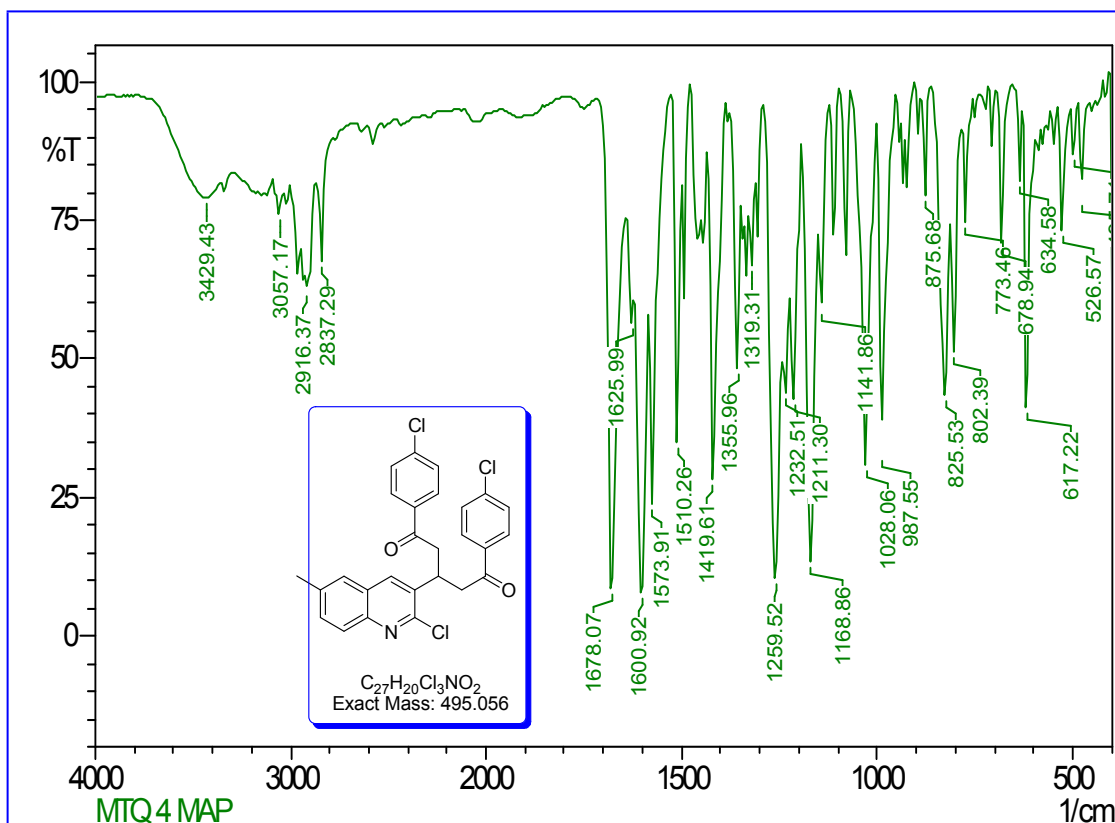






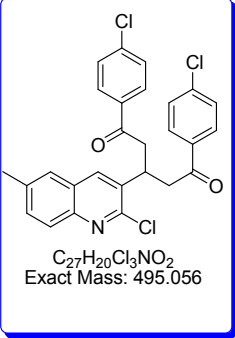
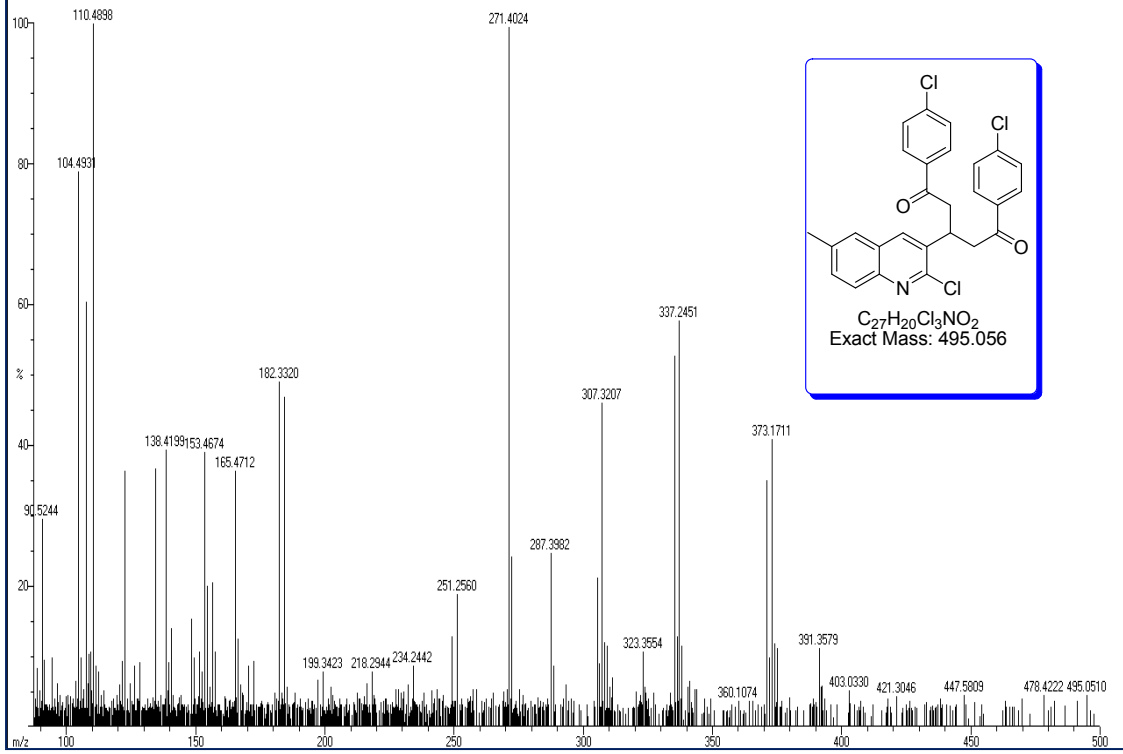


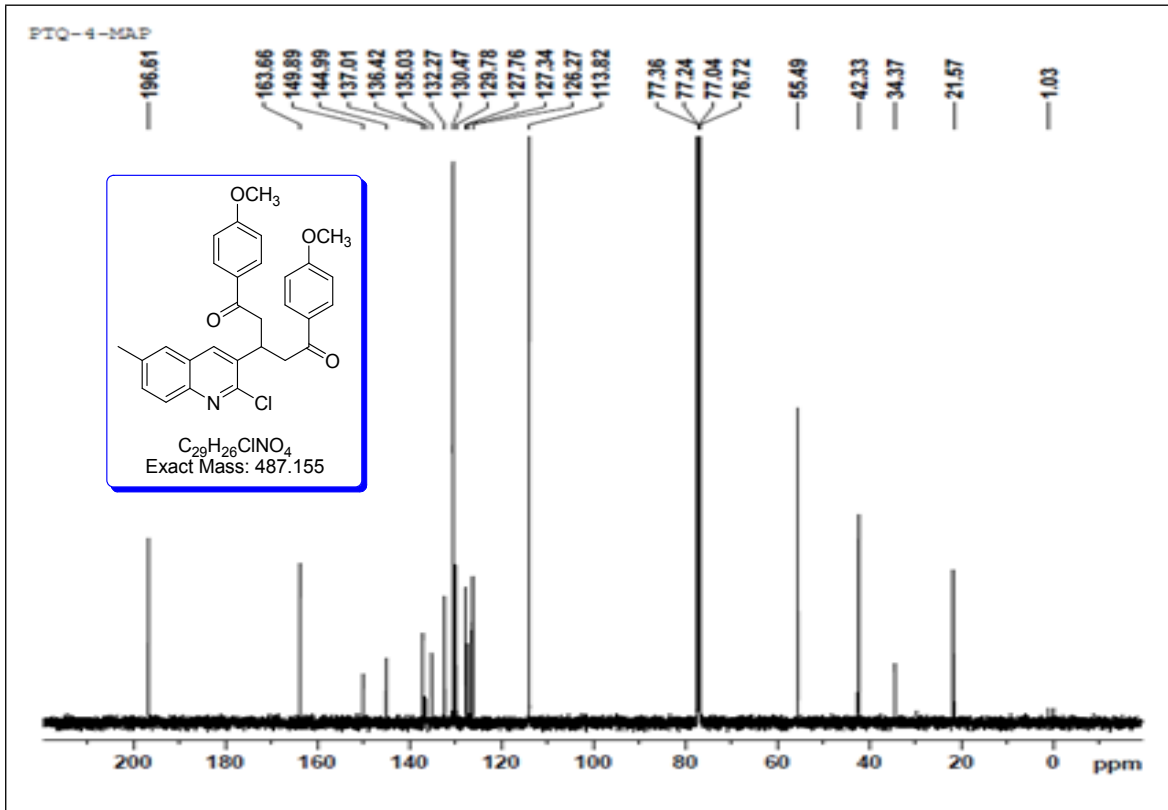
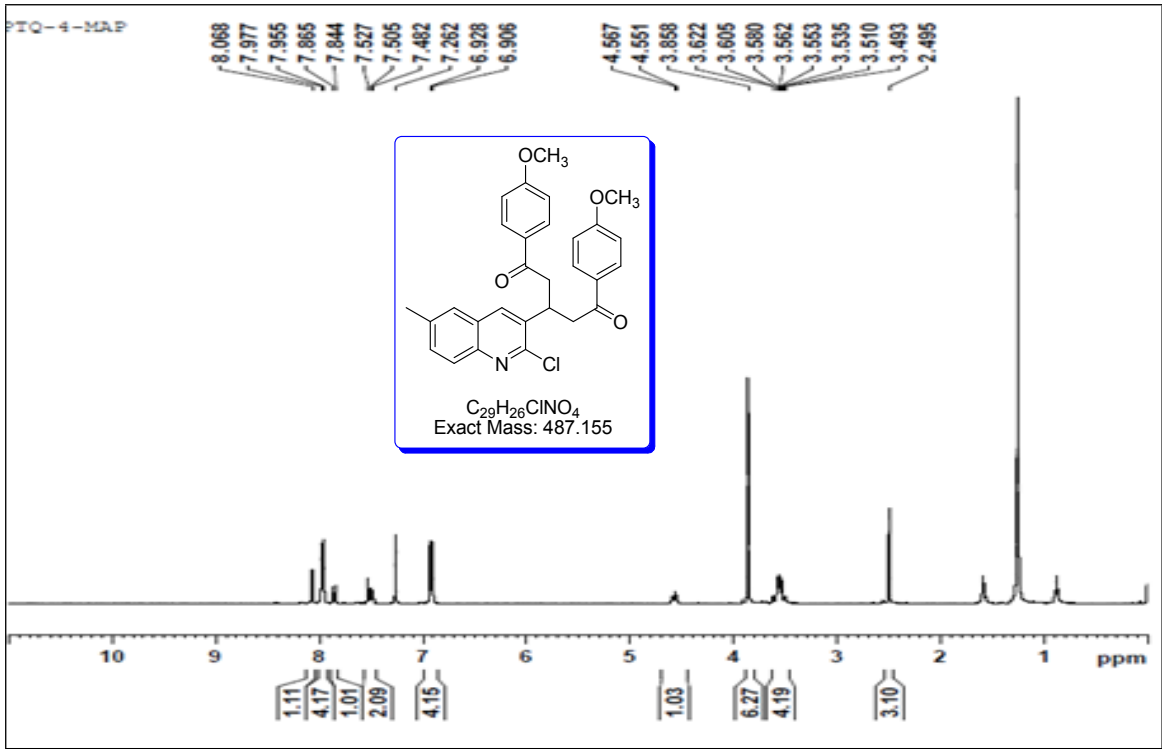


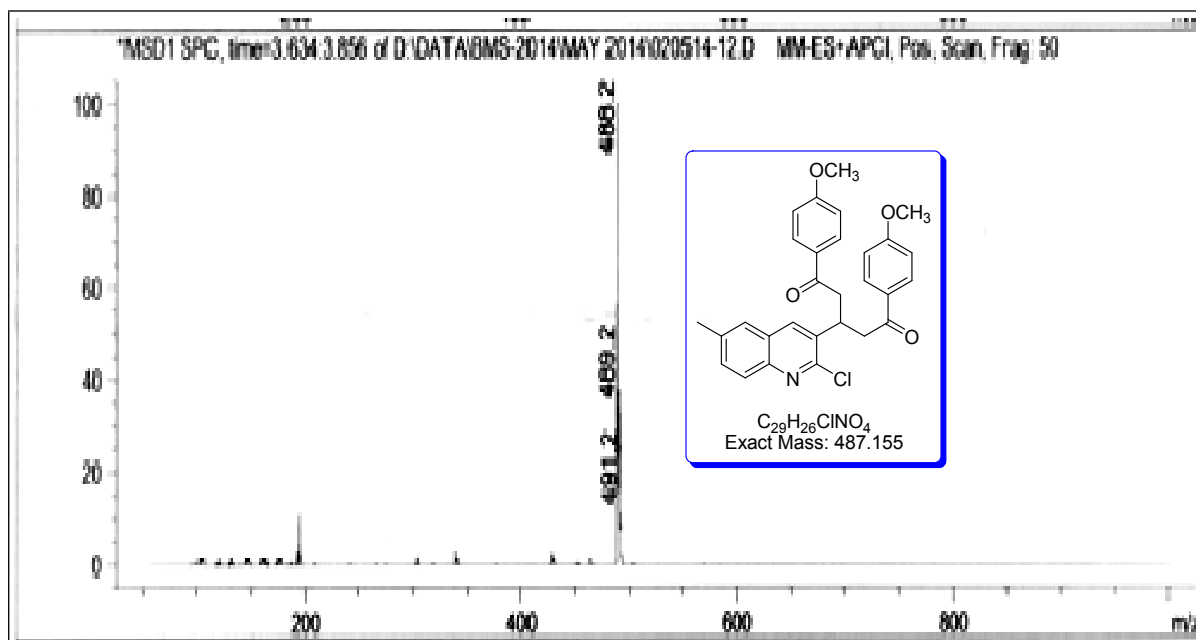
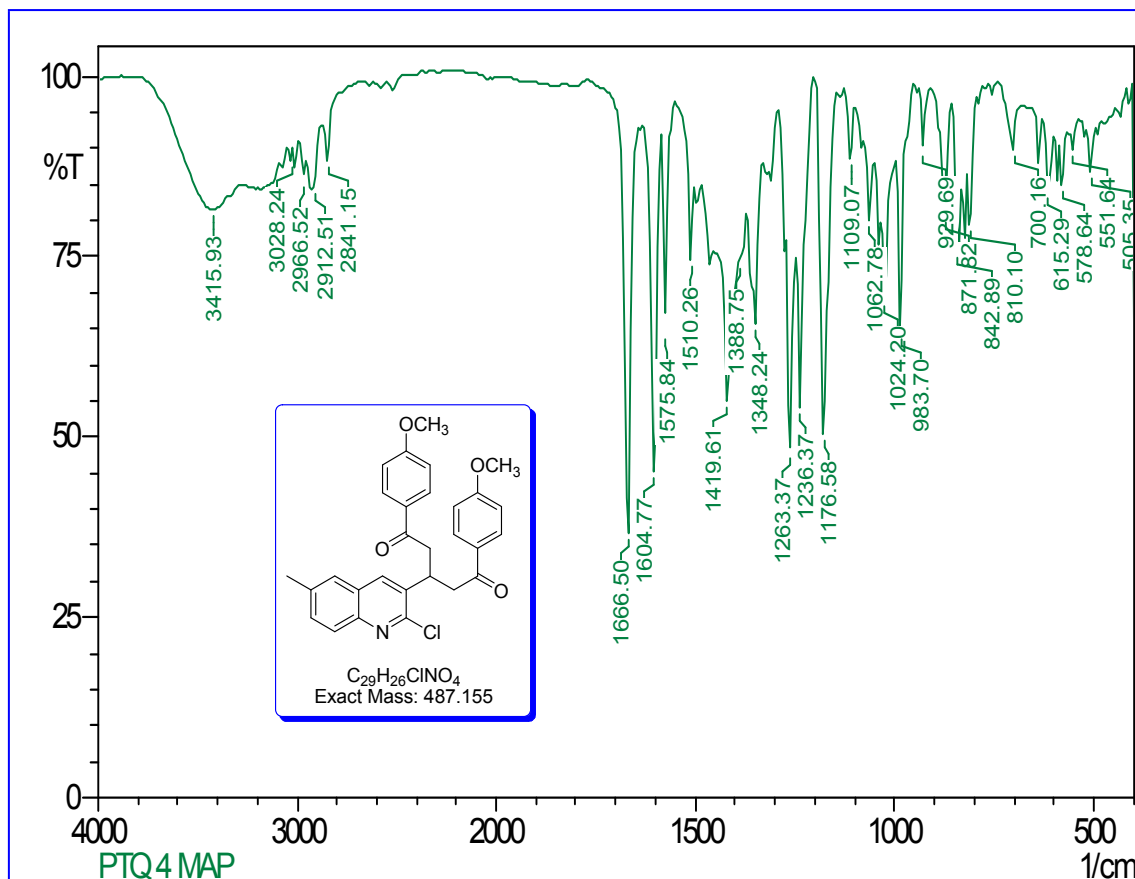


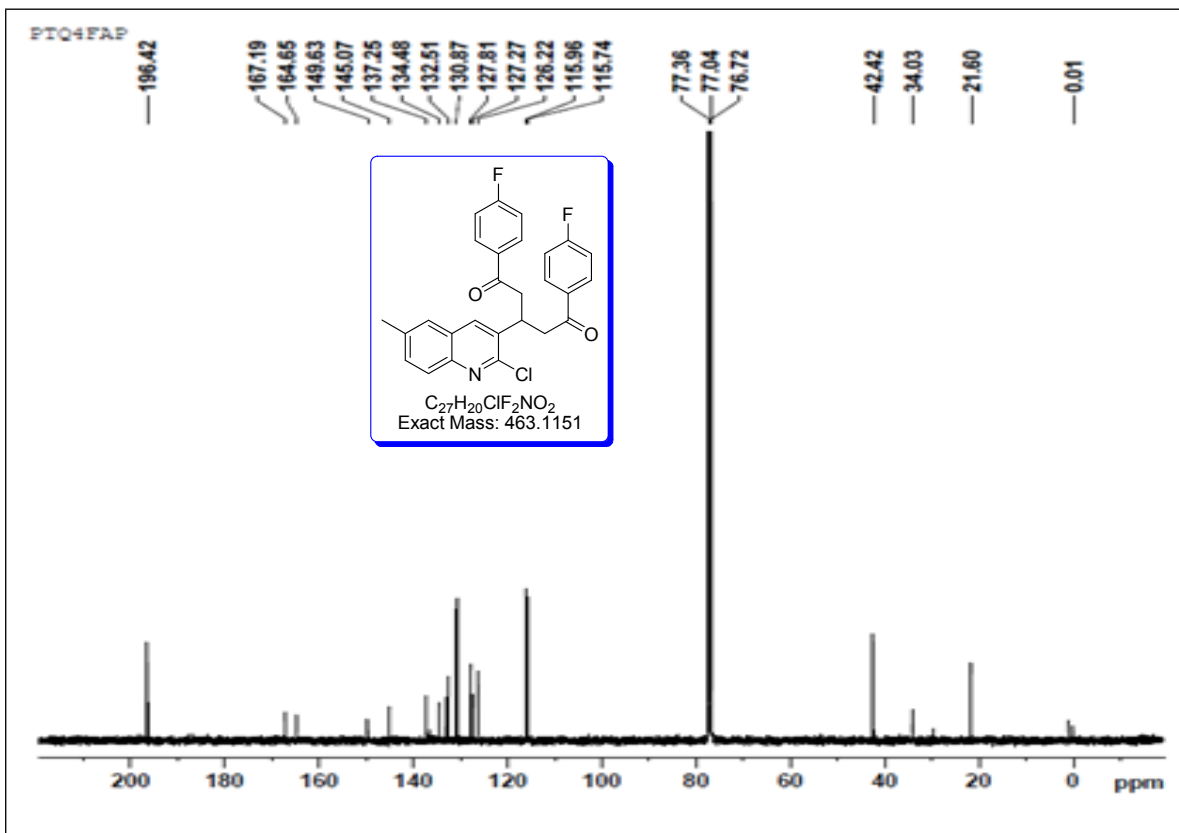
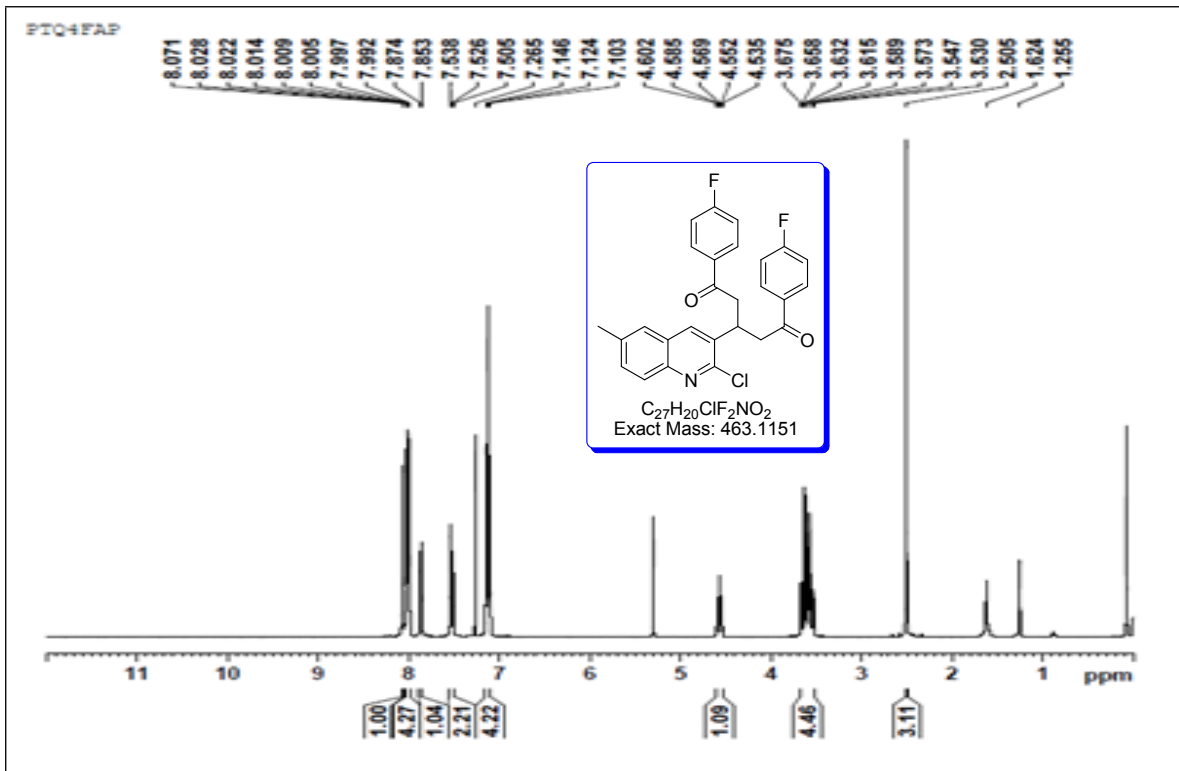
PTQ-4CAP

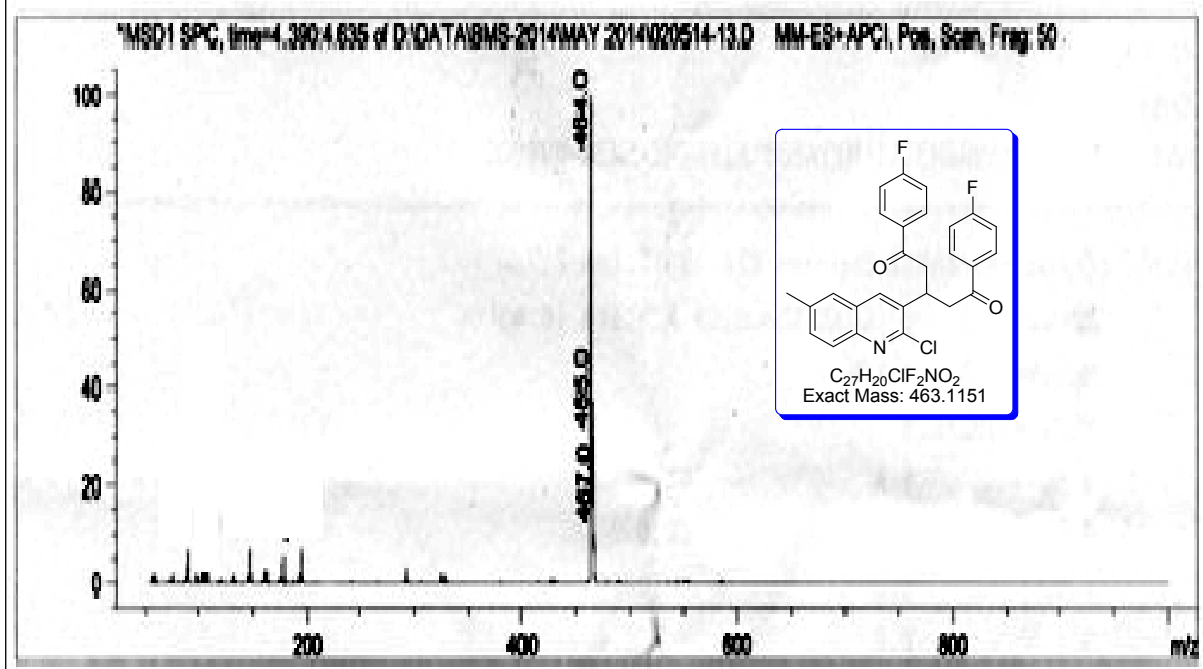
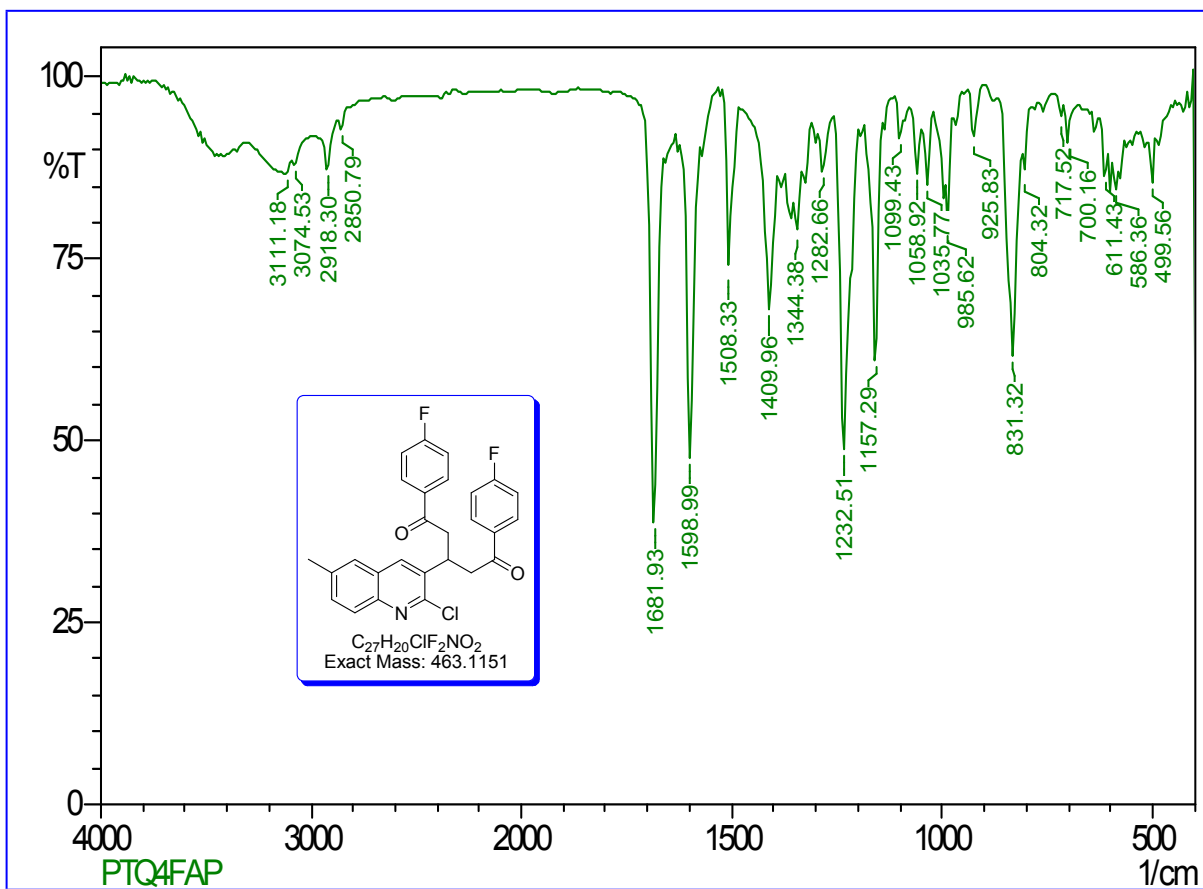
Scan: 8 TIC=8533600 Base=11.6%FS #Ions=2123 RT=18





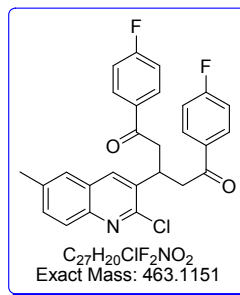
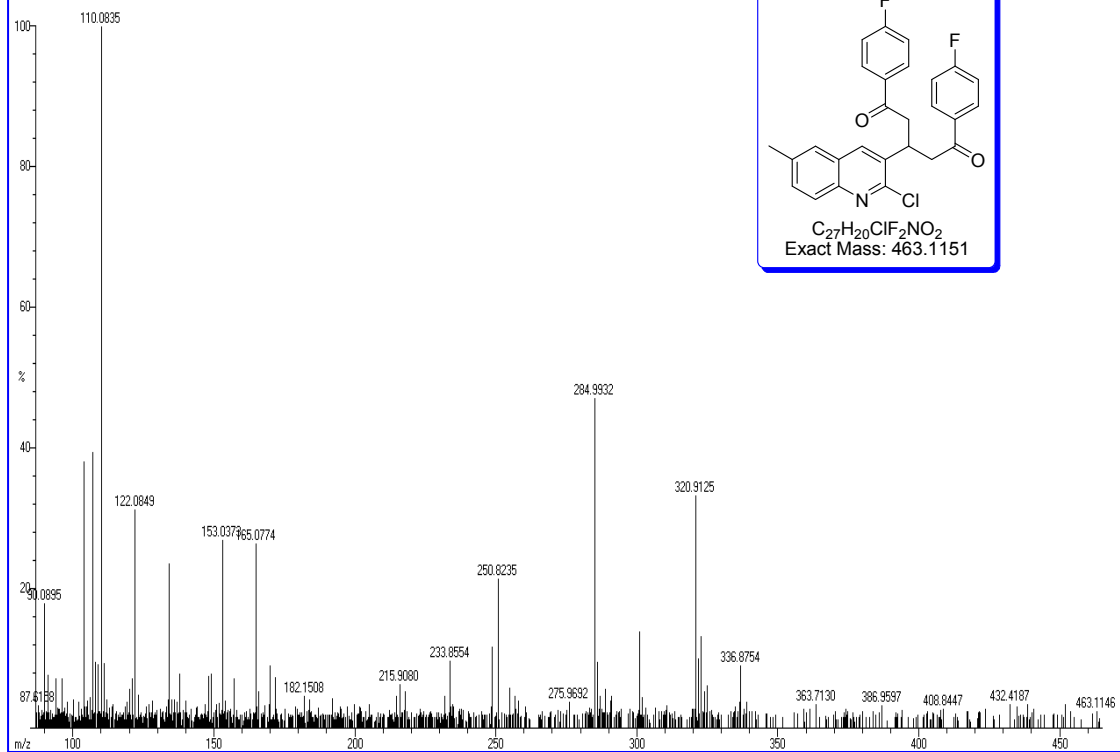


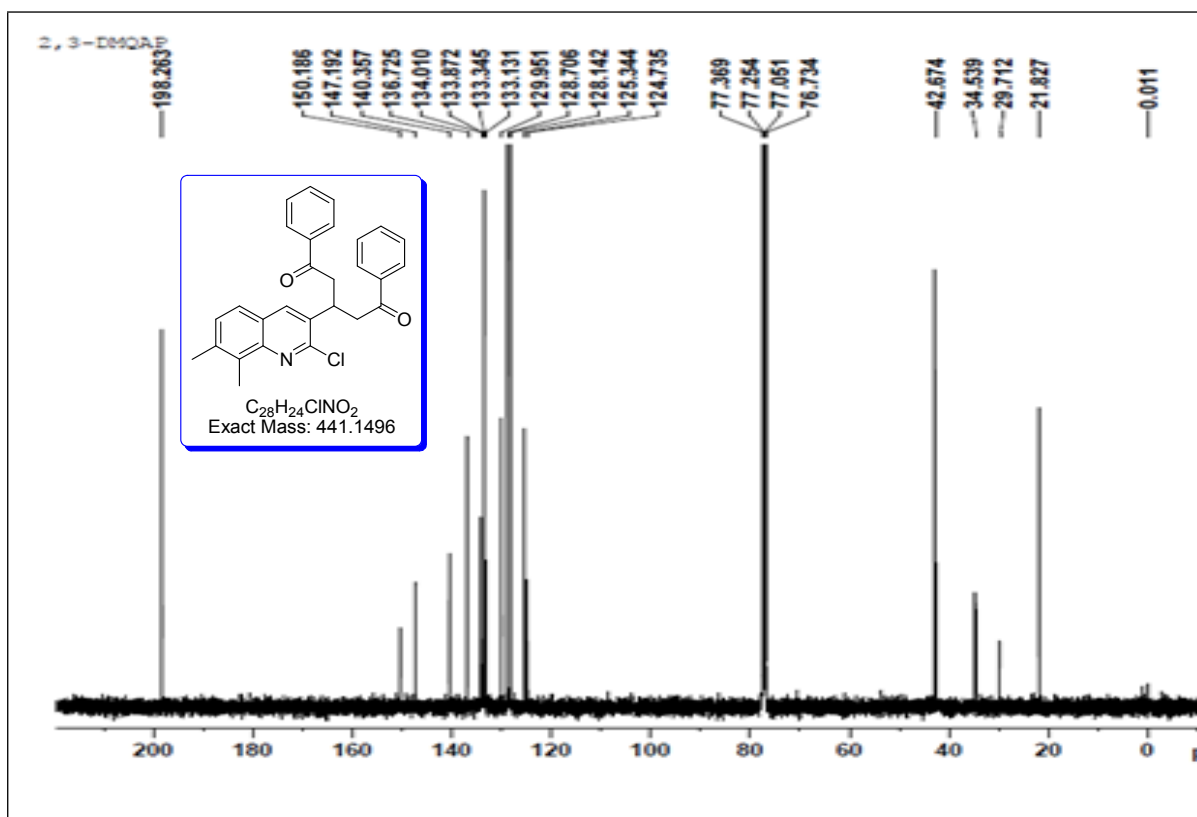
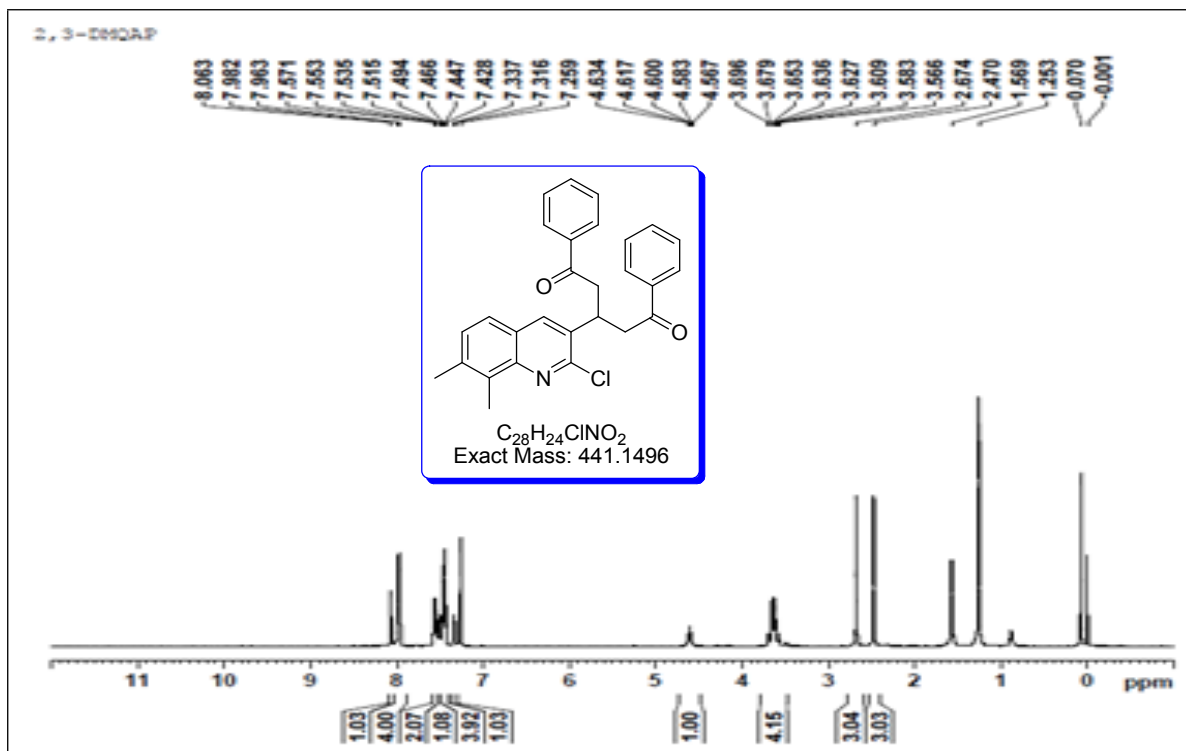


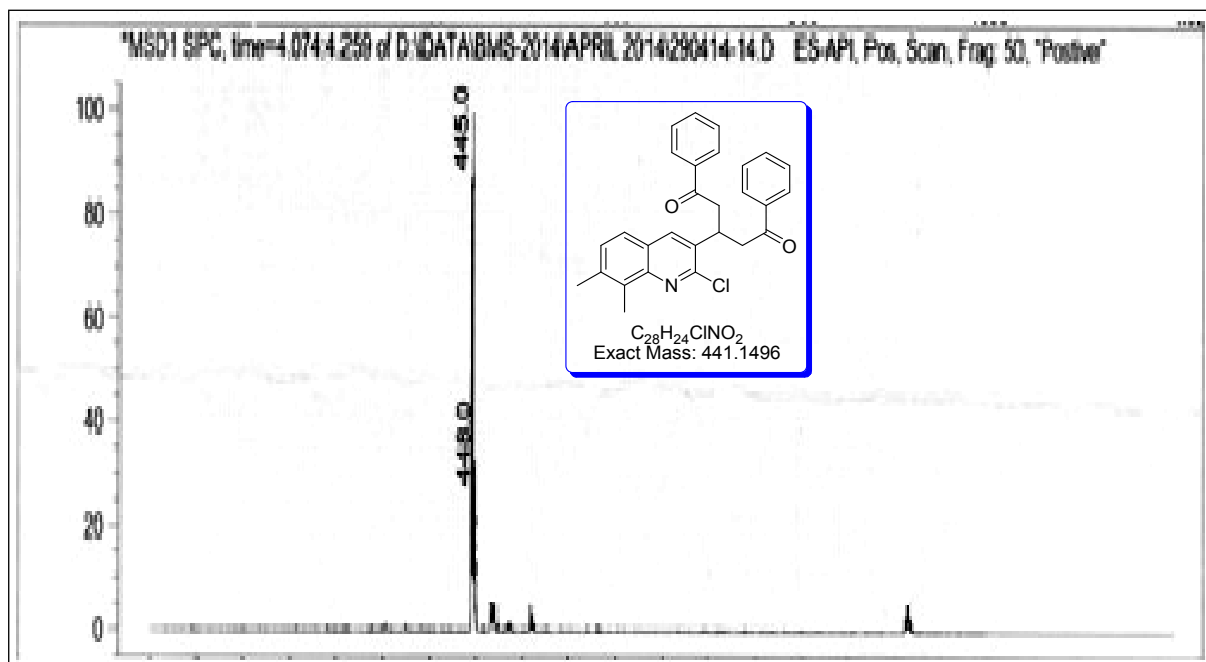
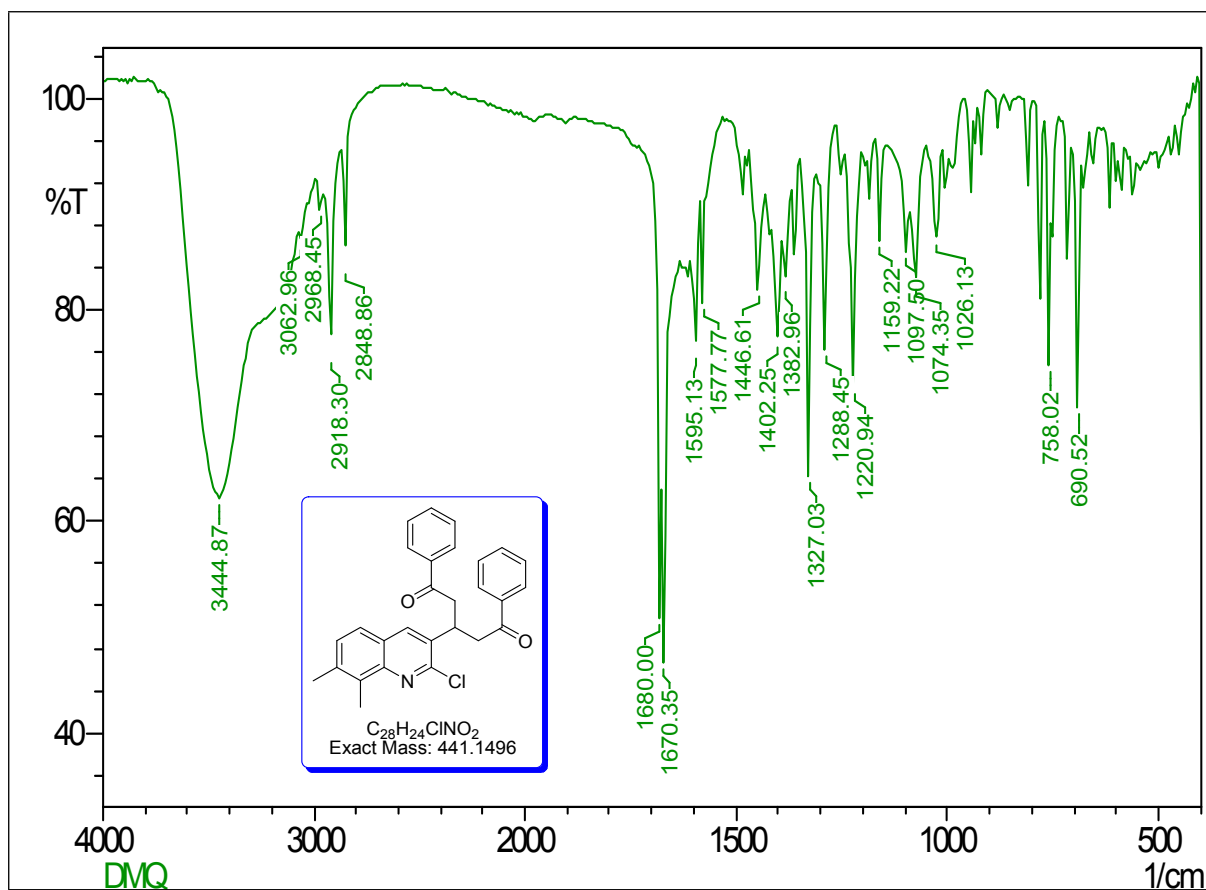


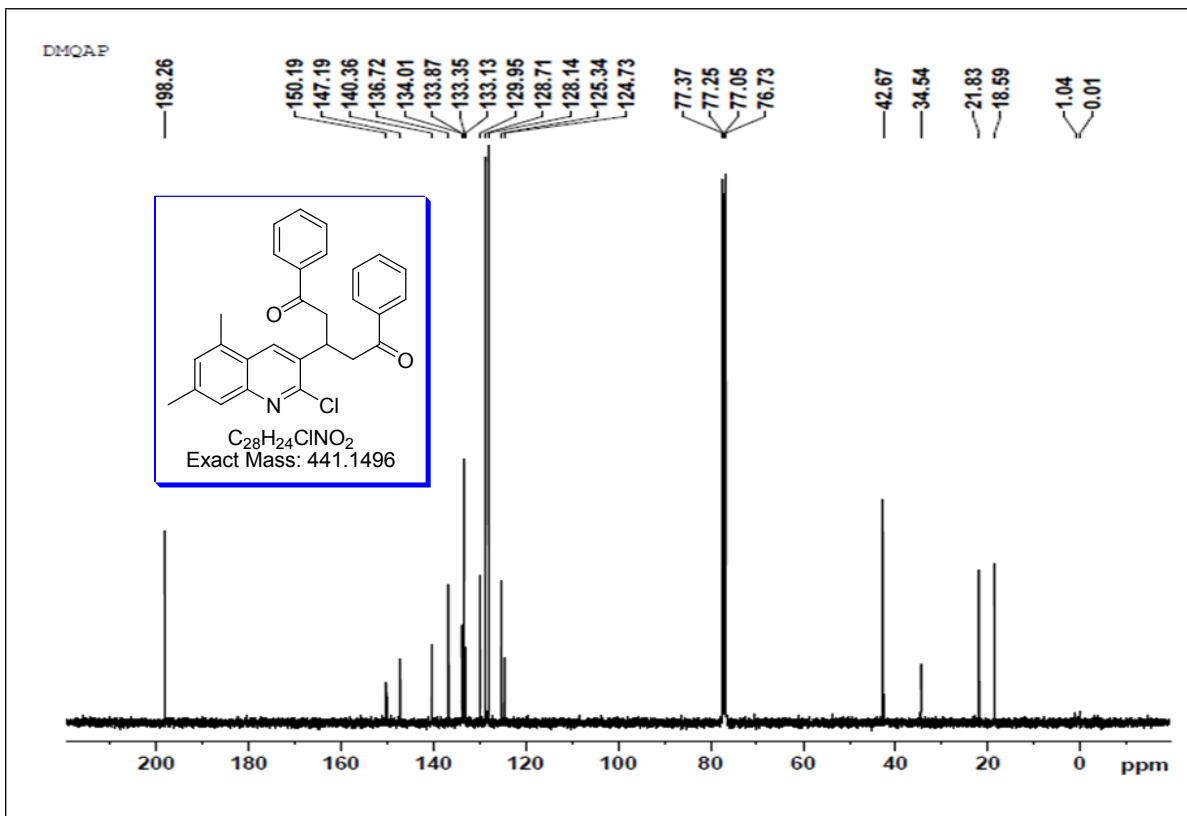
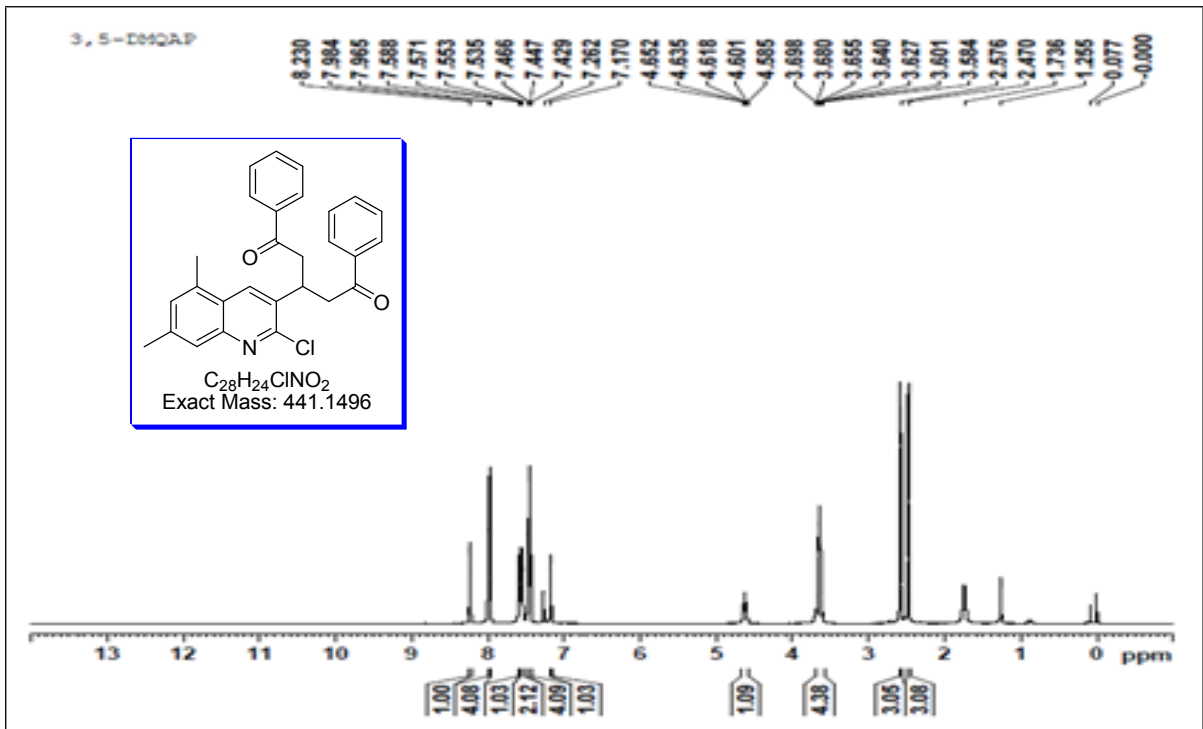
PTQ-4FAP

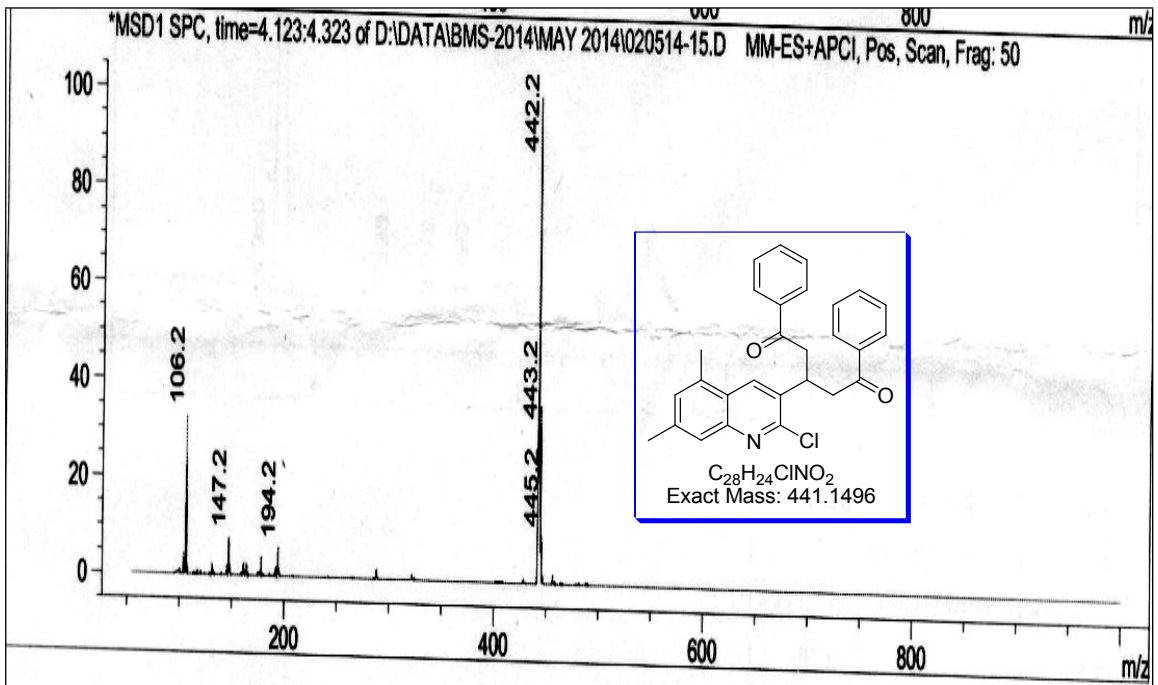
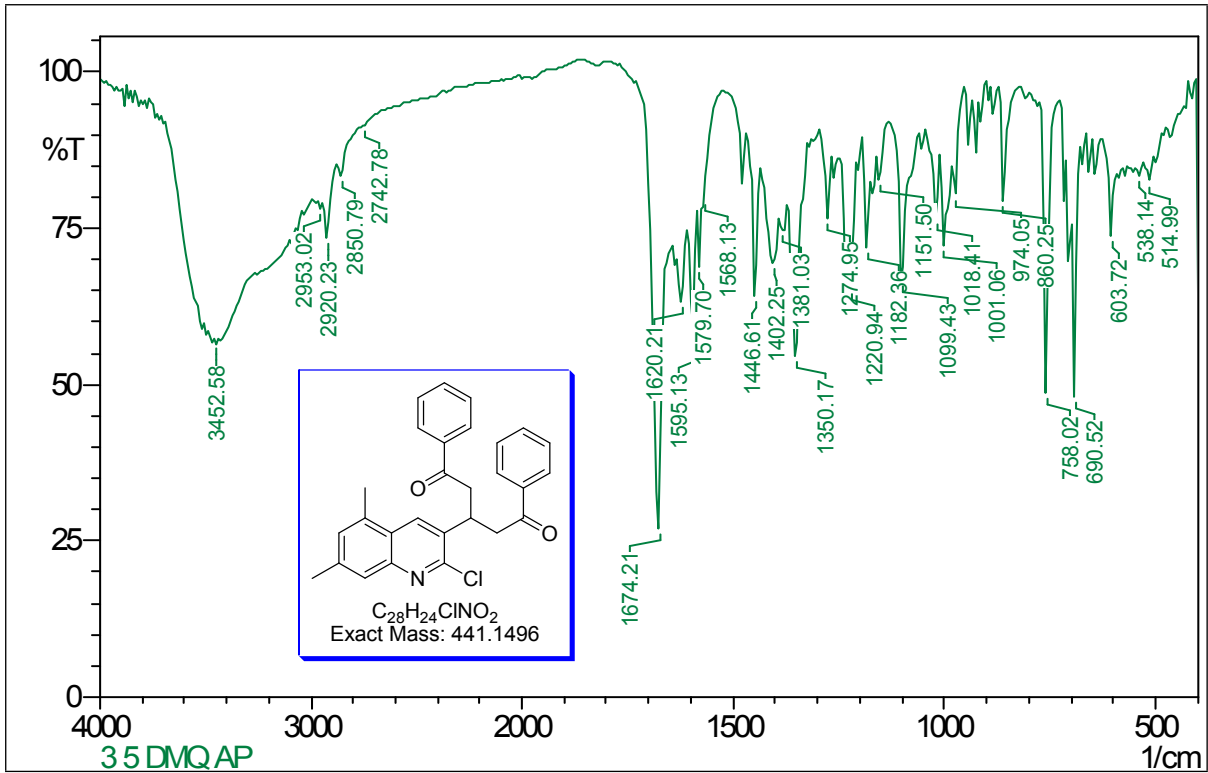
Scan: 7 TIC=7756832 Base=16.1%FS #Ions=2086 RT=17

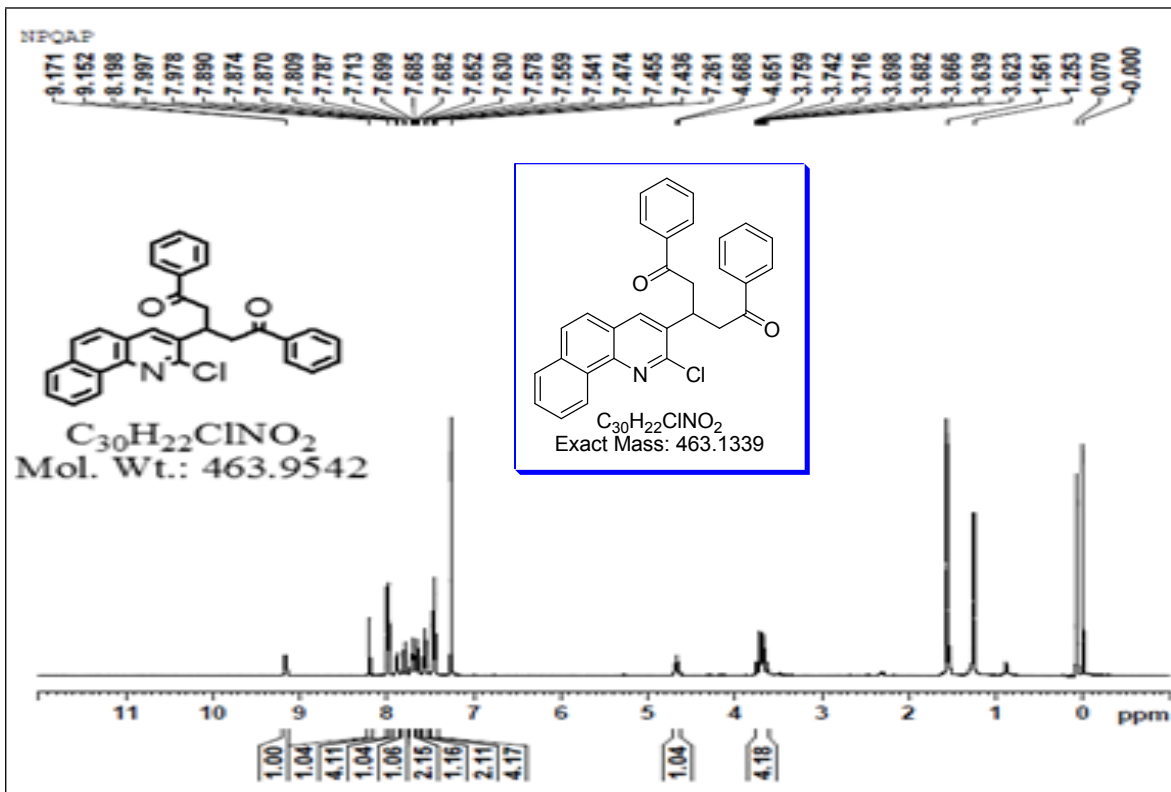
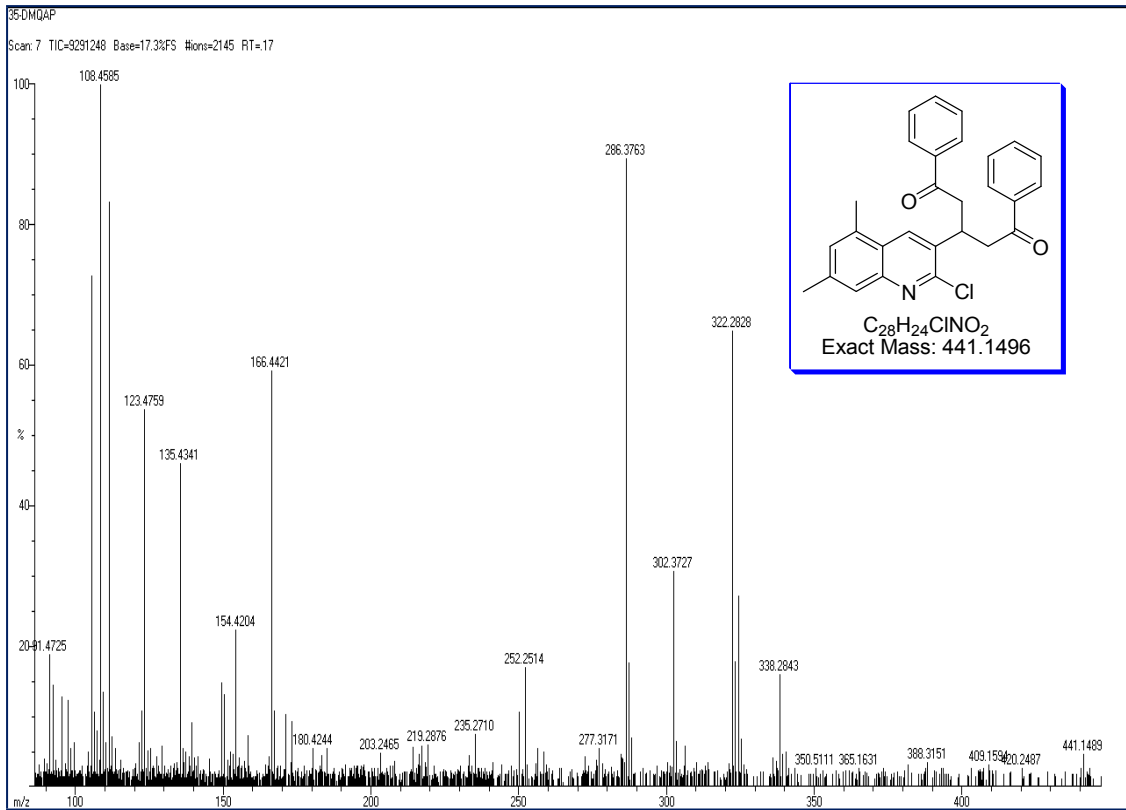


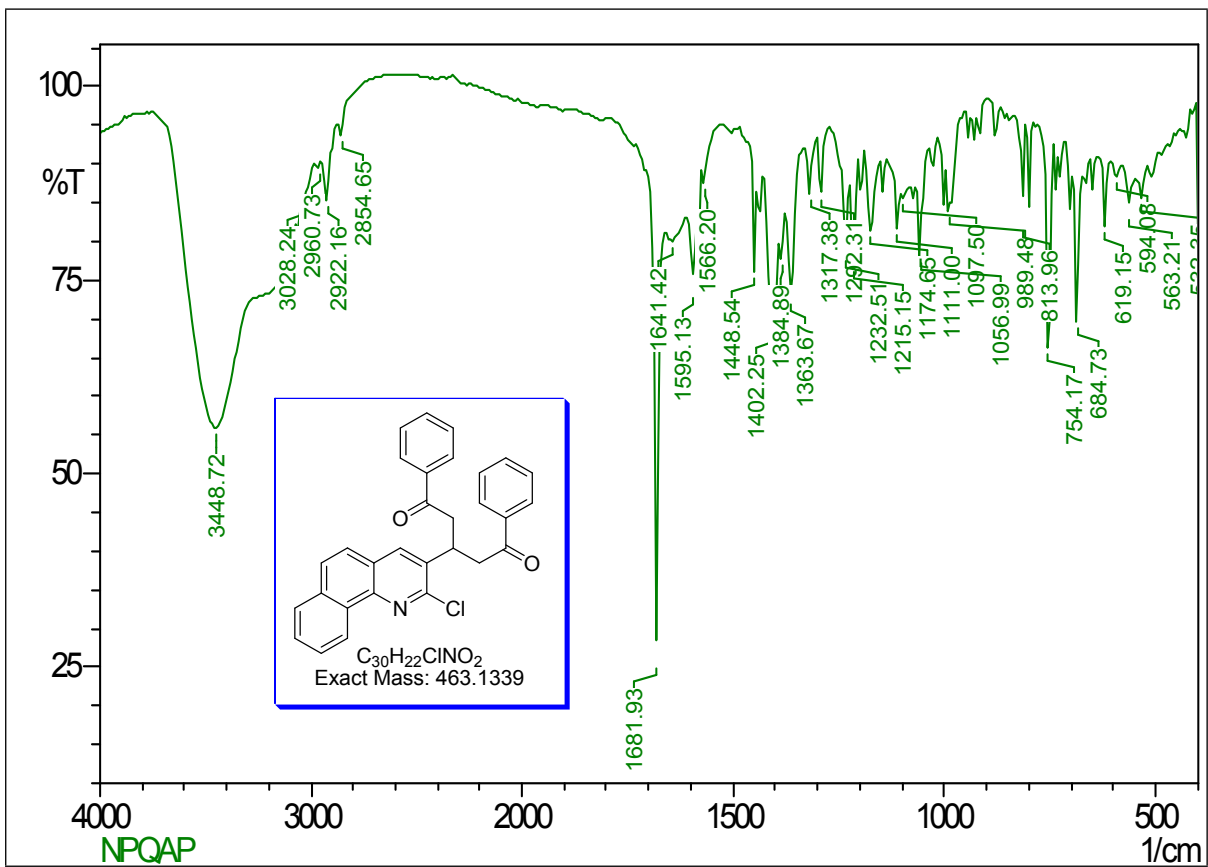
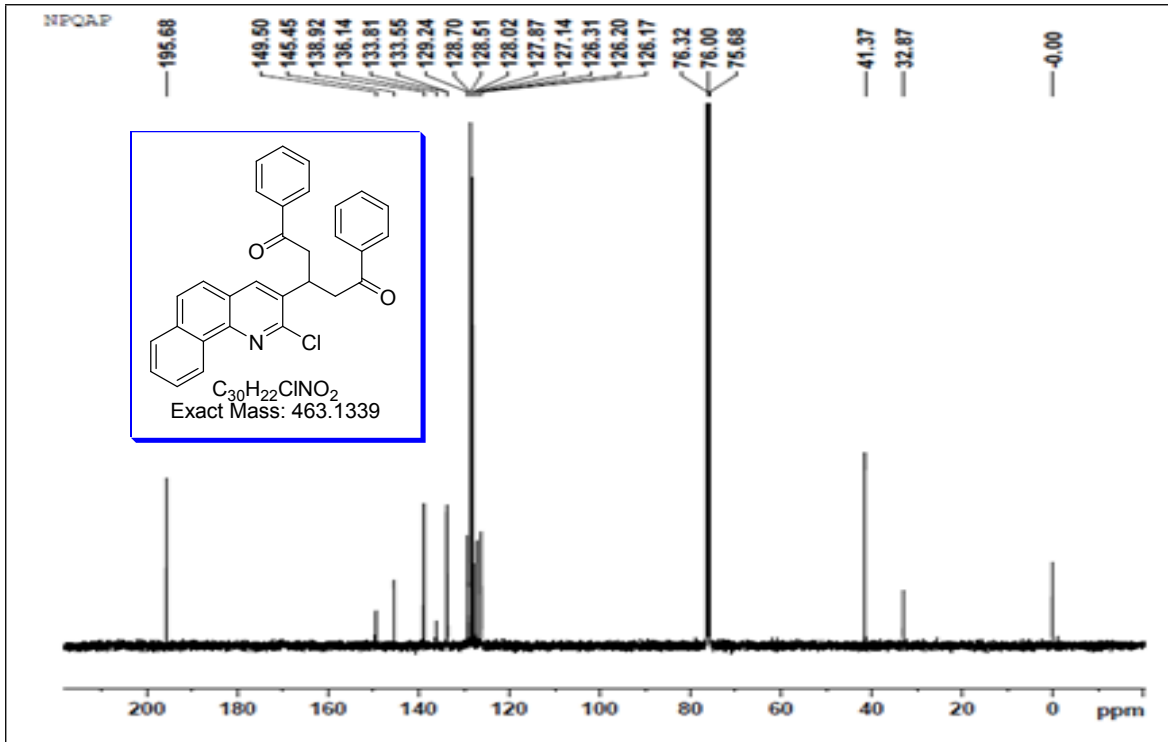


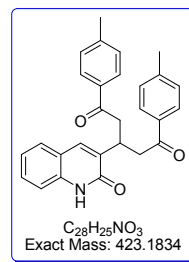
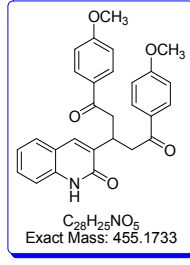
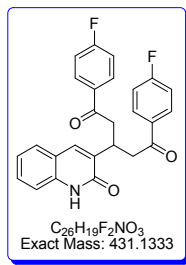
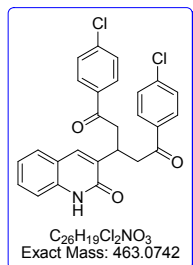
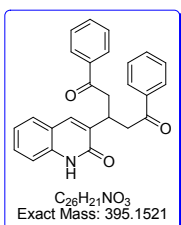
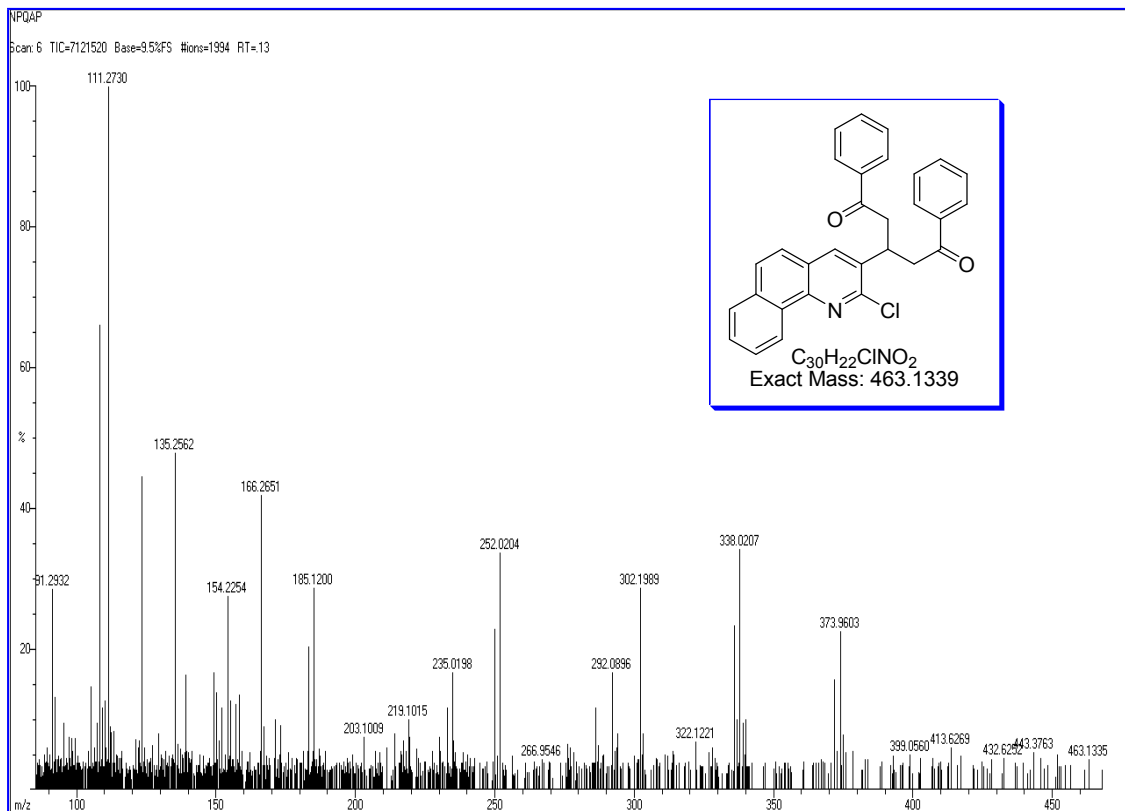
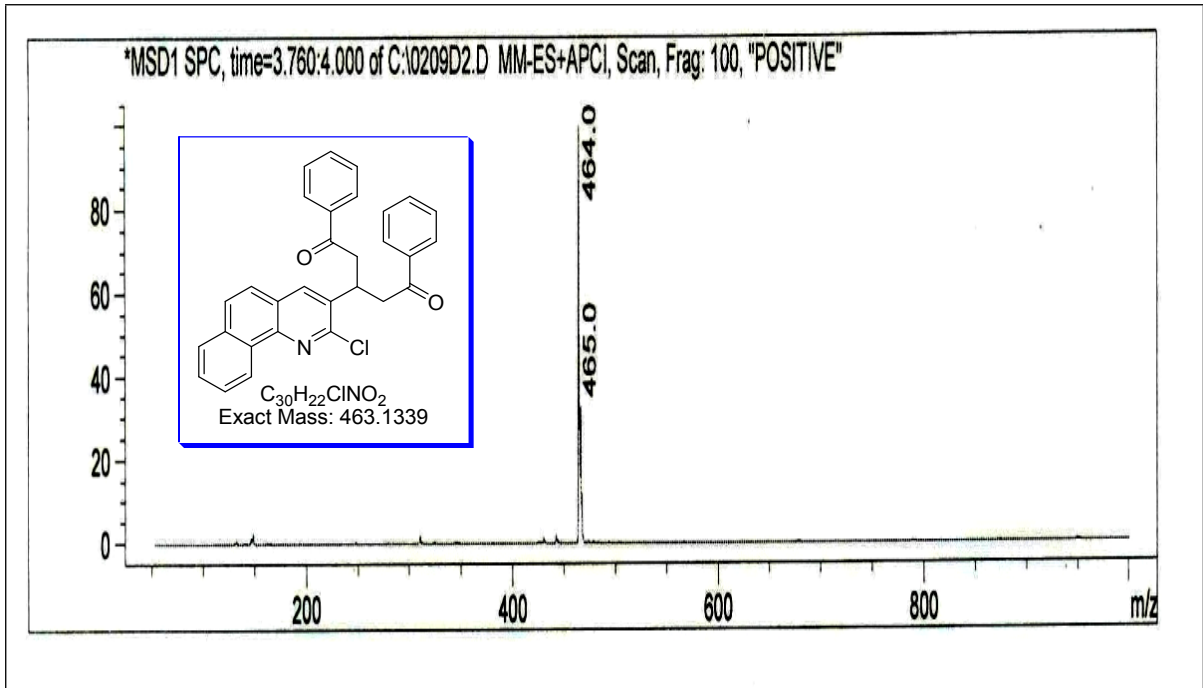


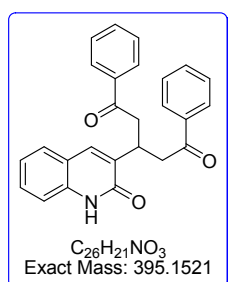
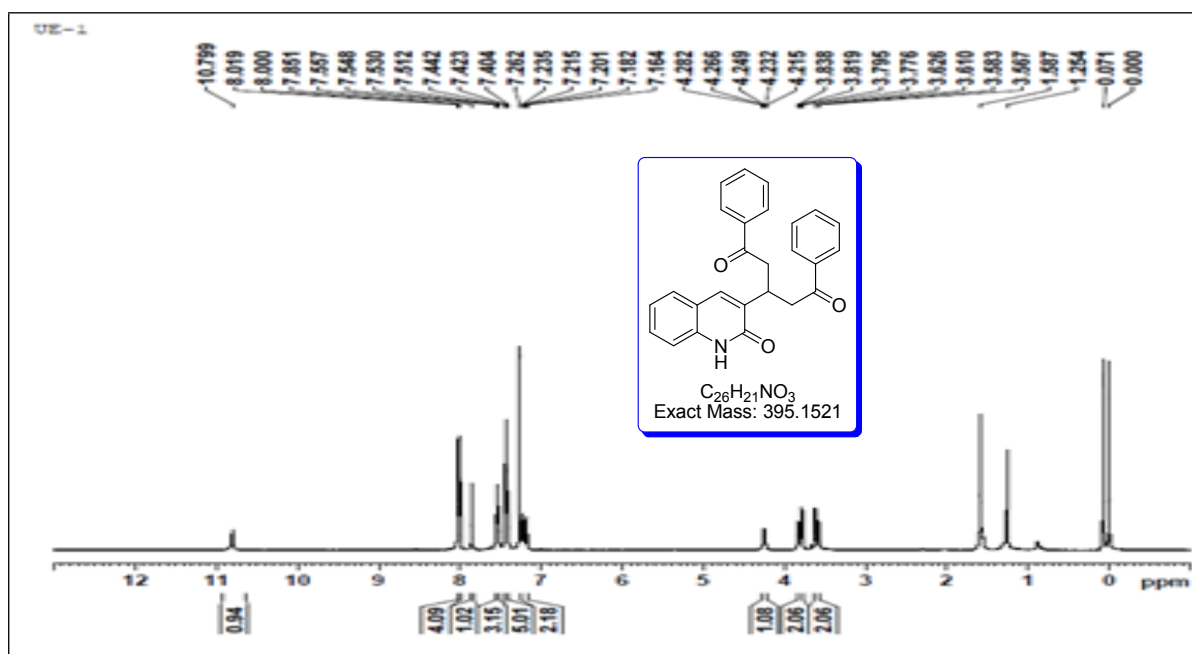
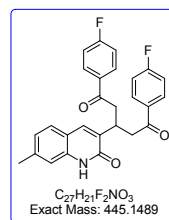
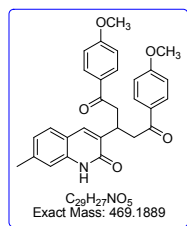
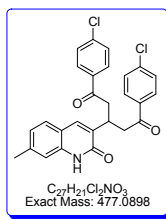
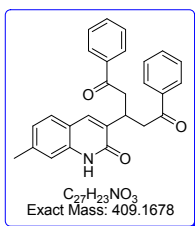


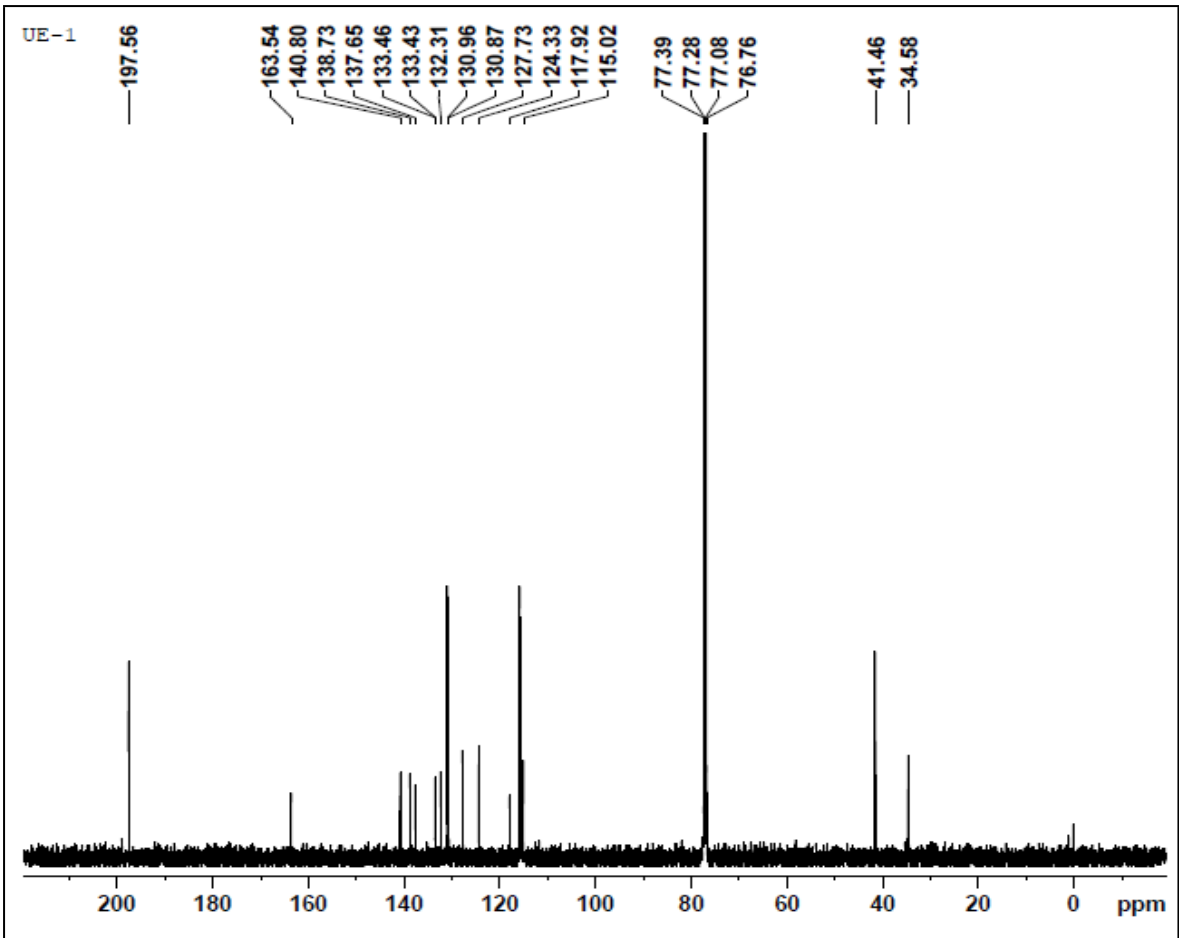


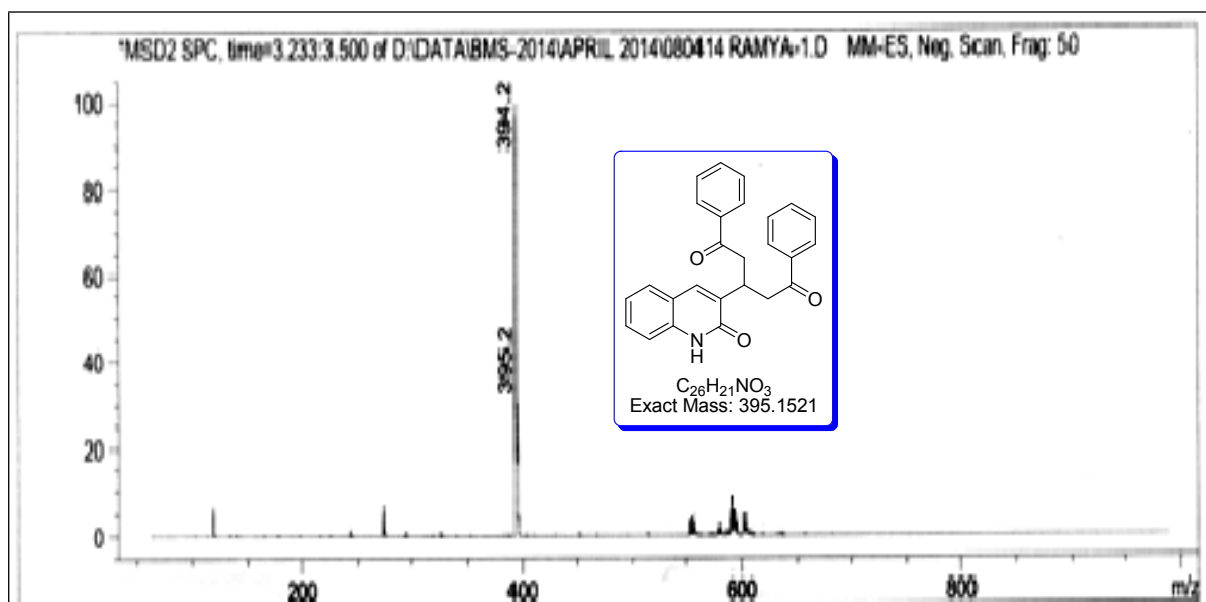
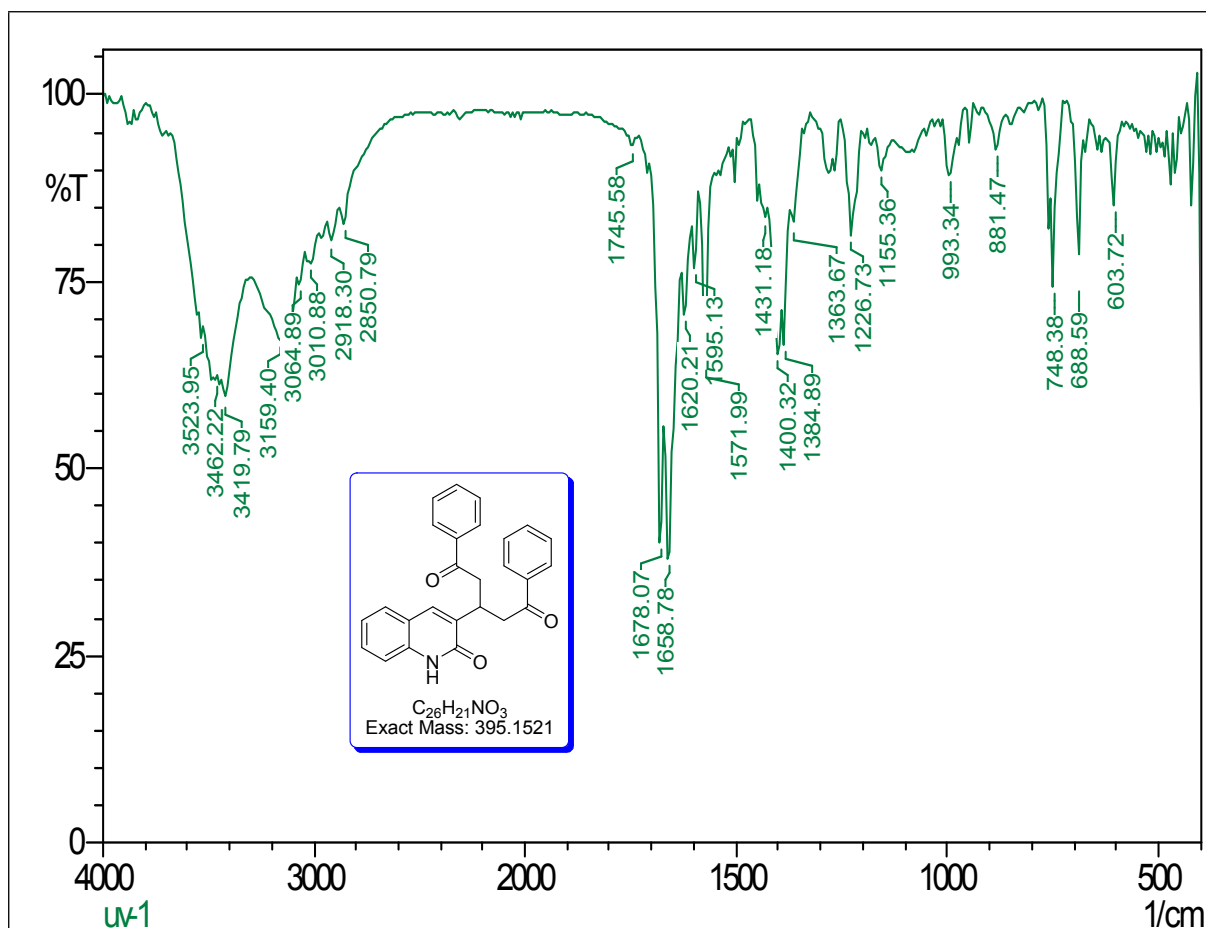


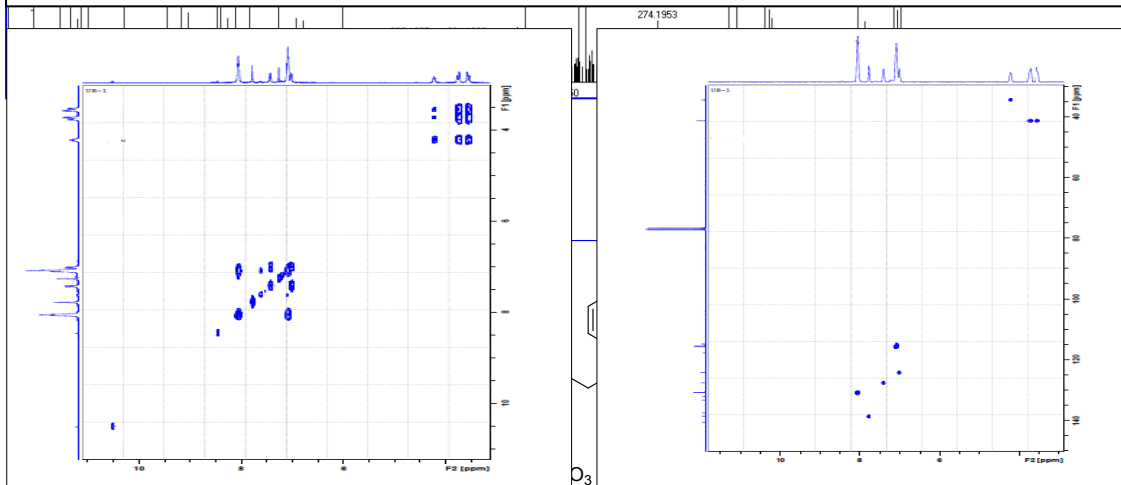
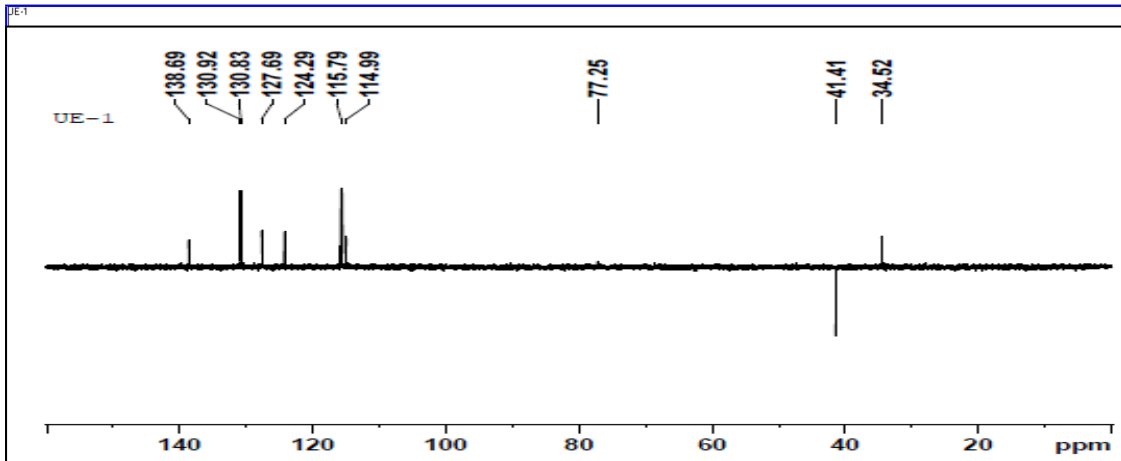




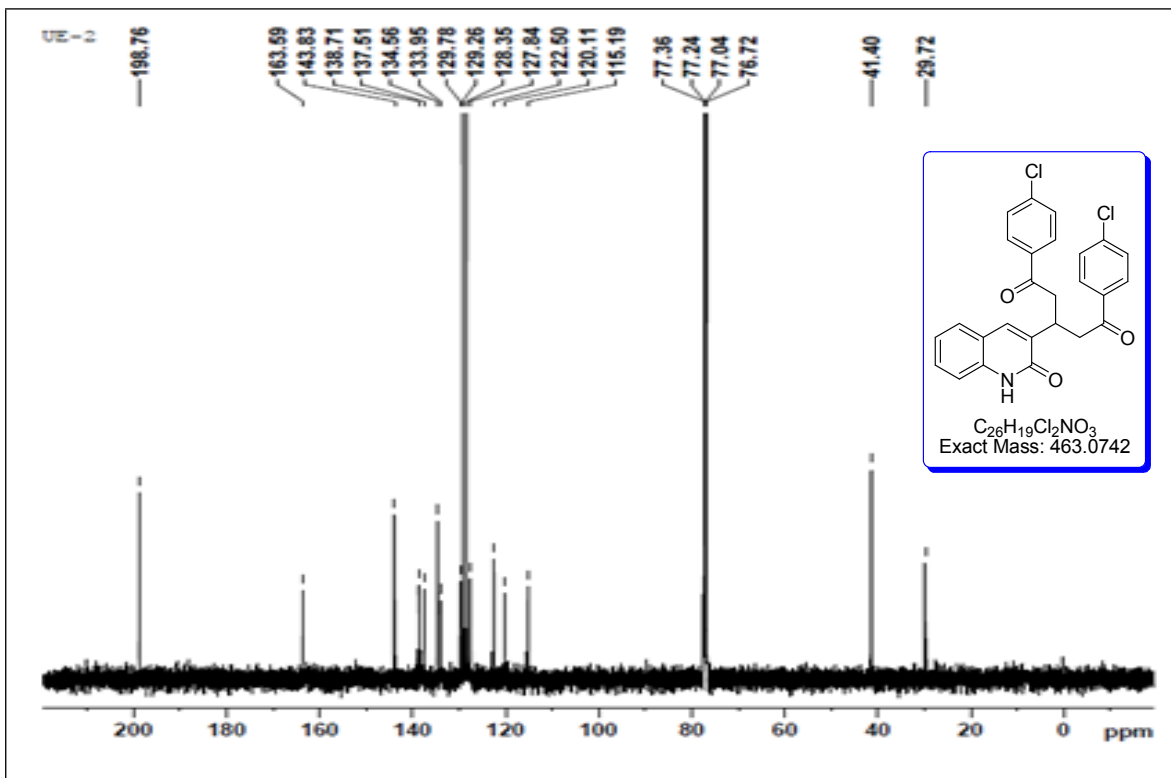
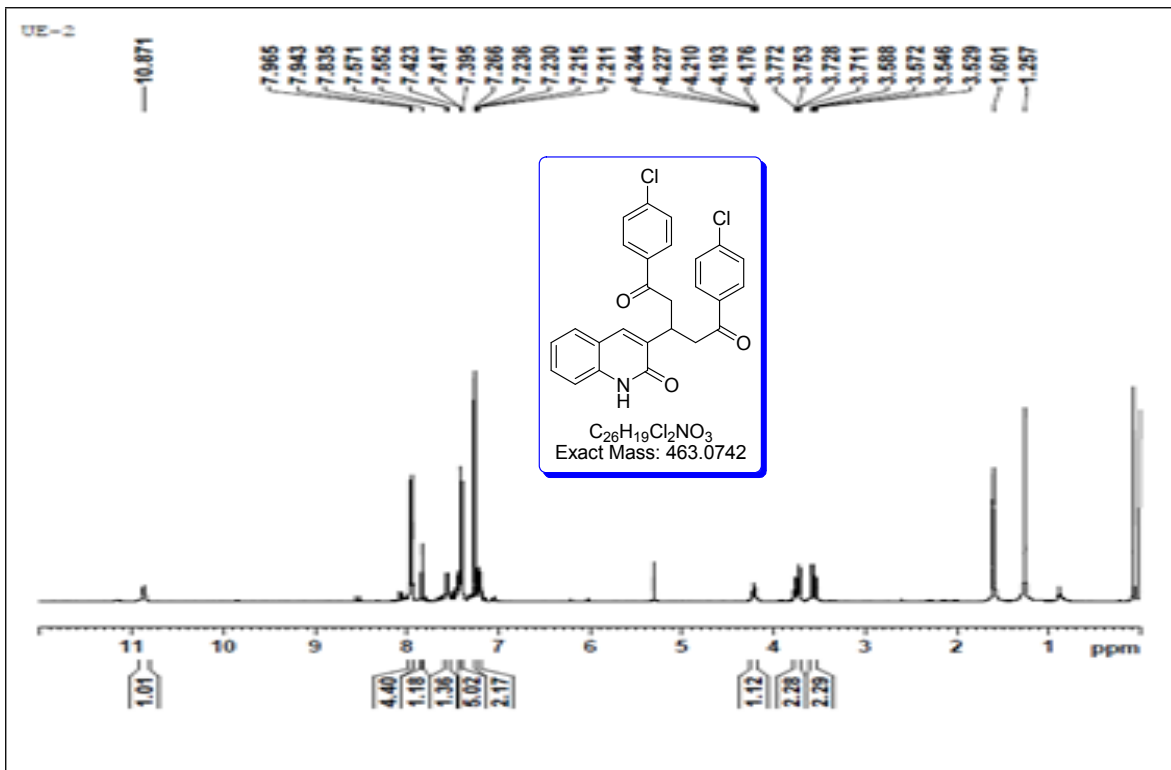


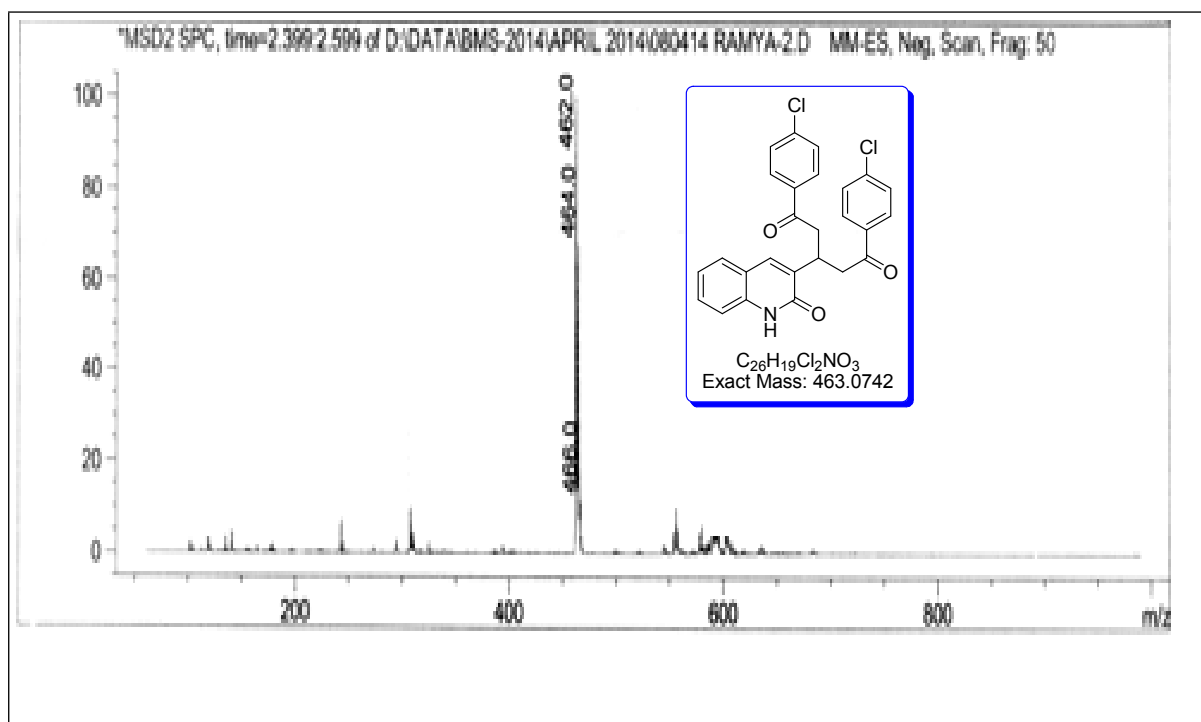
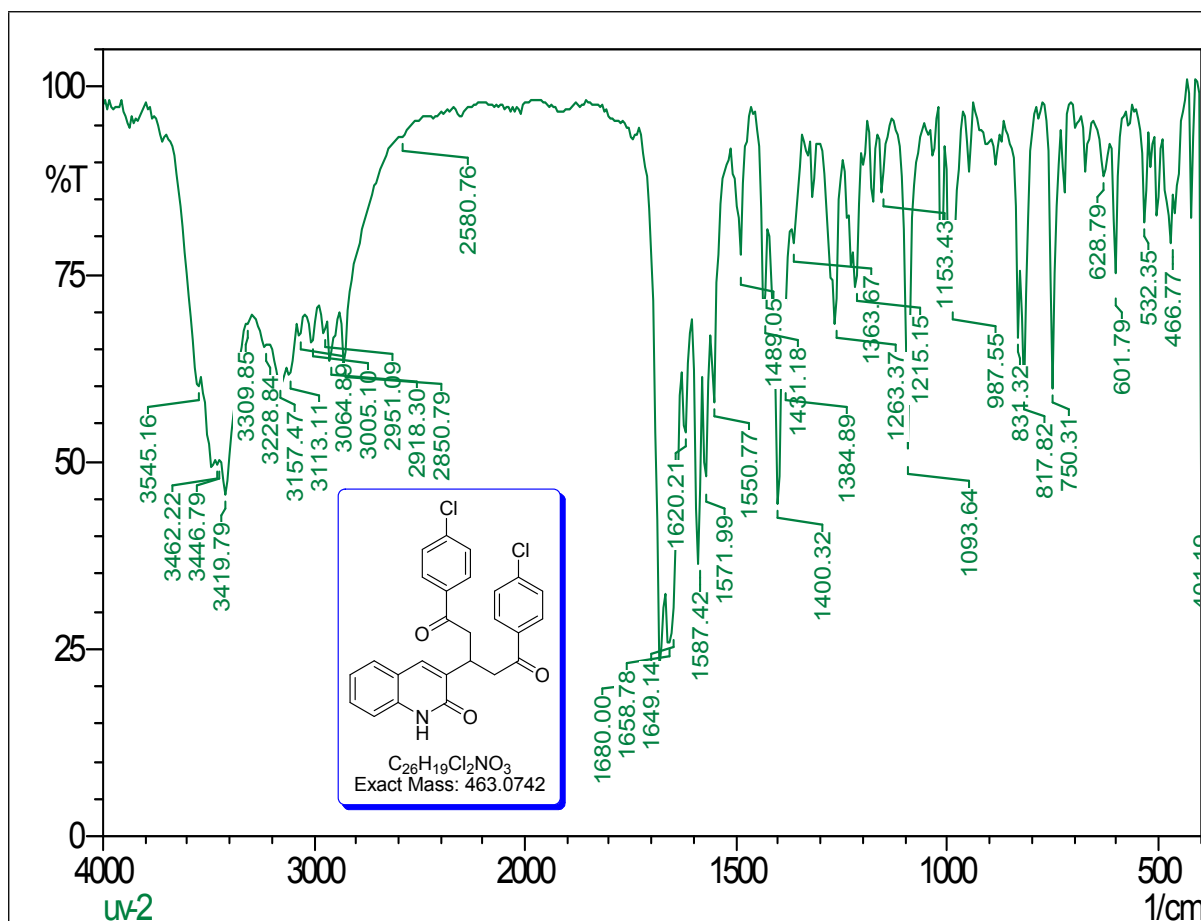


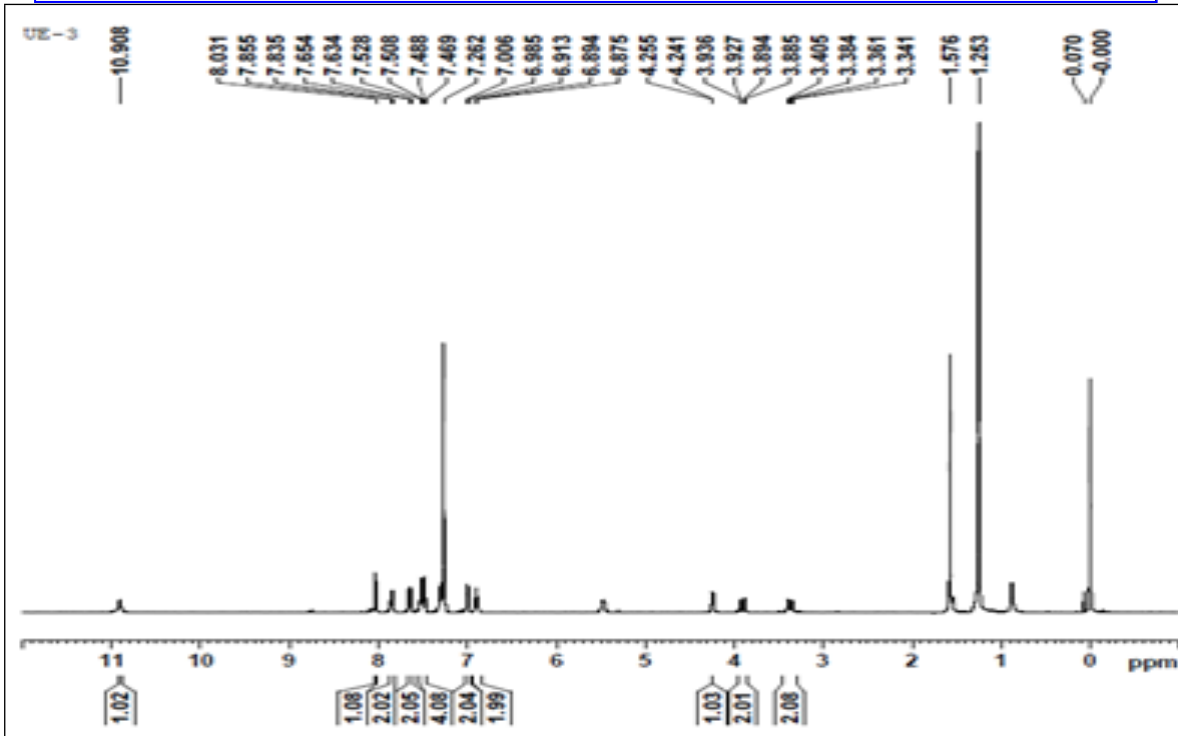
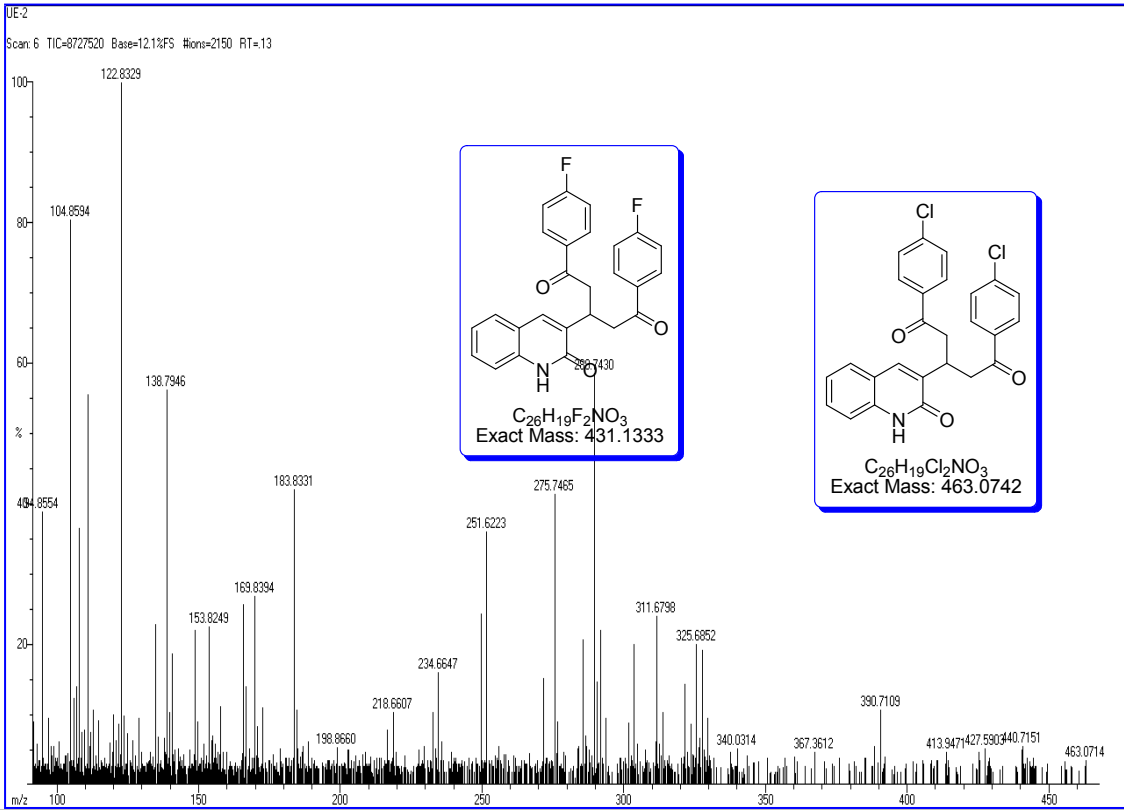


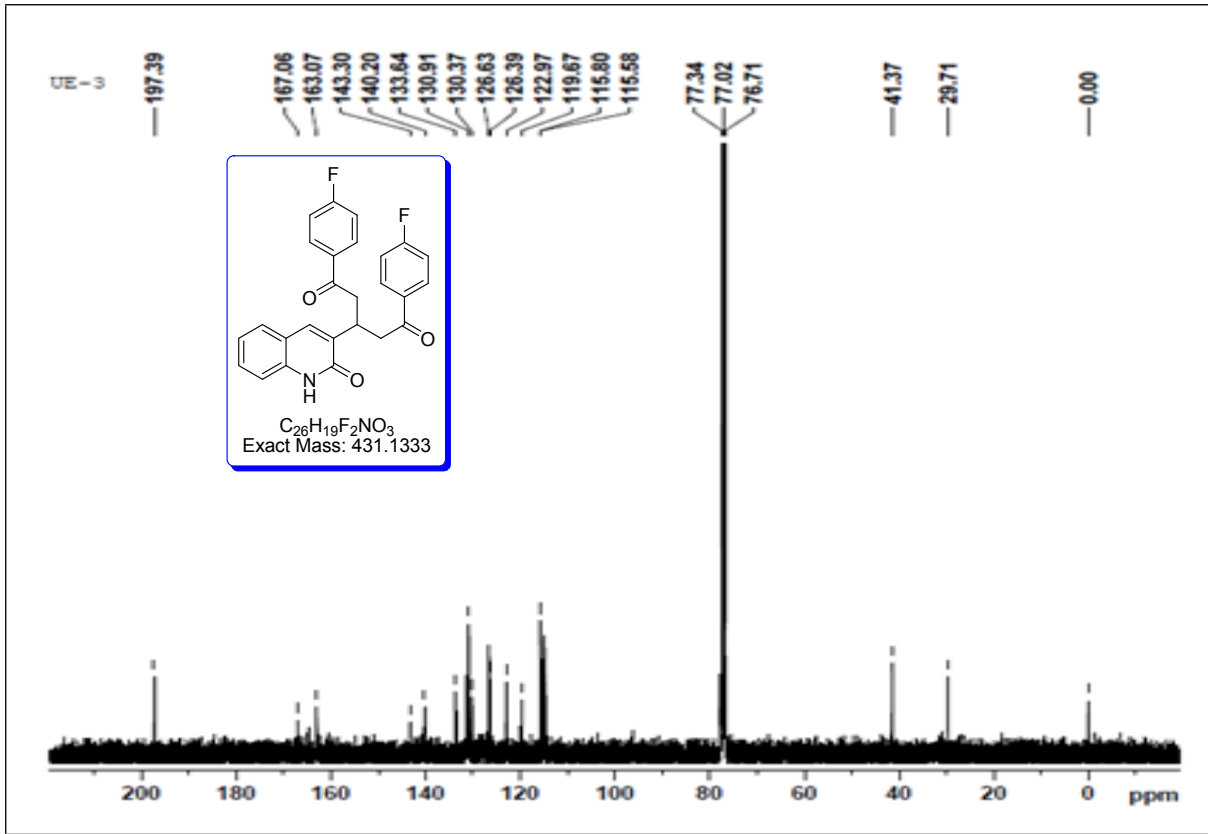


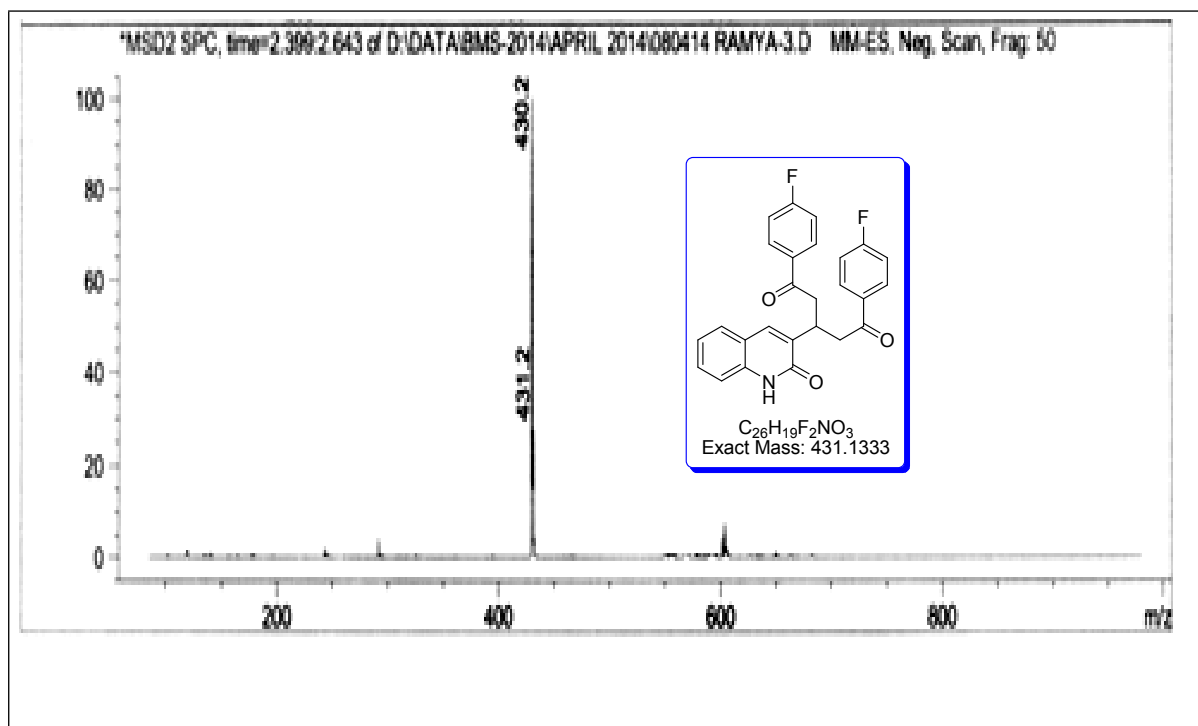
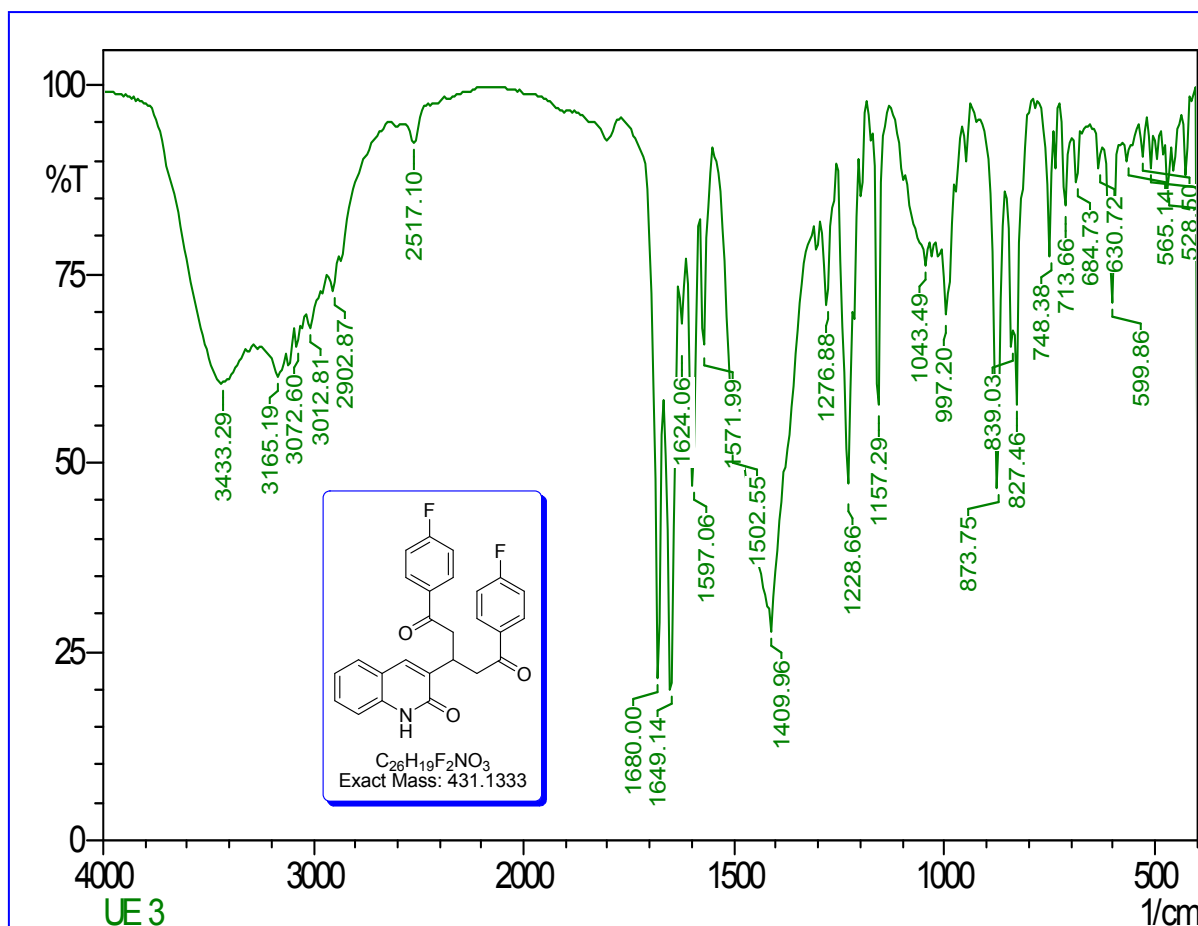
Exact Mass: 395.1521





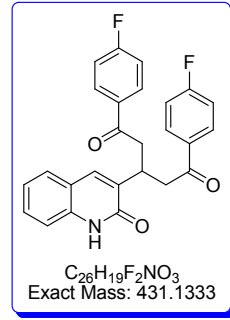
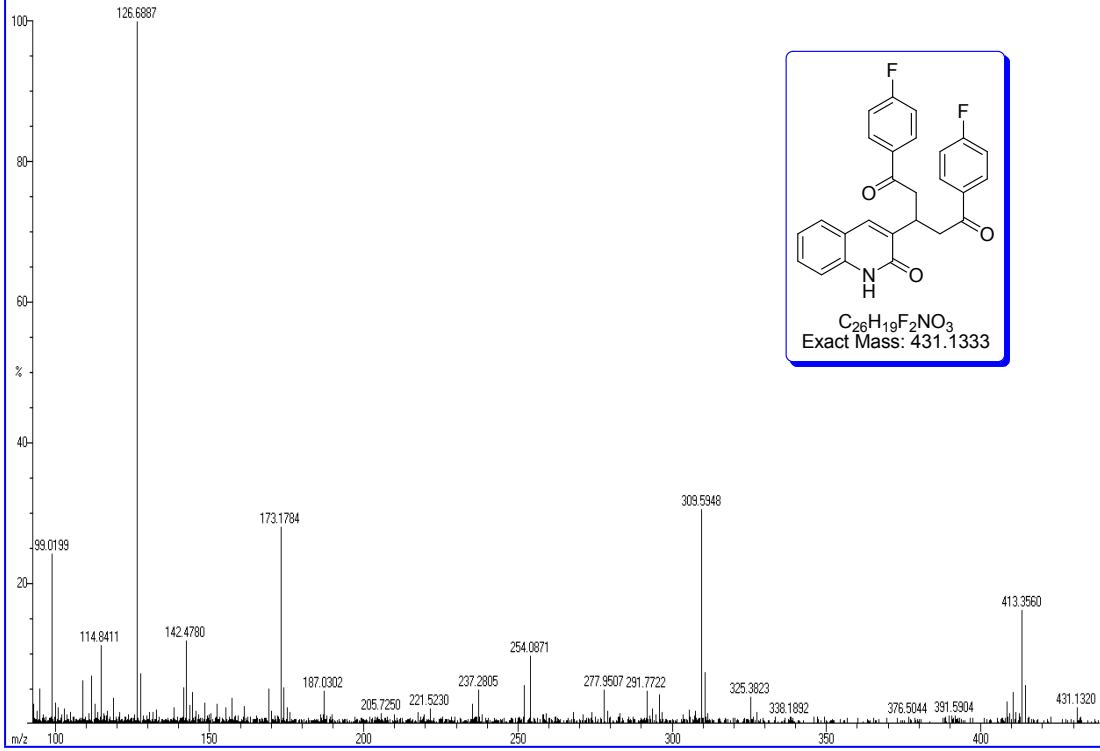


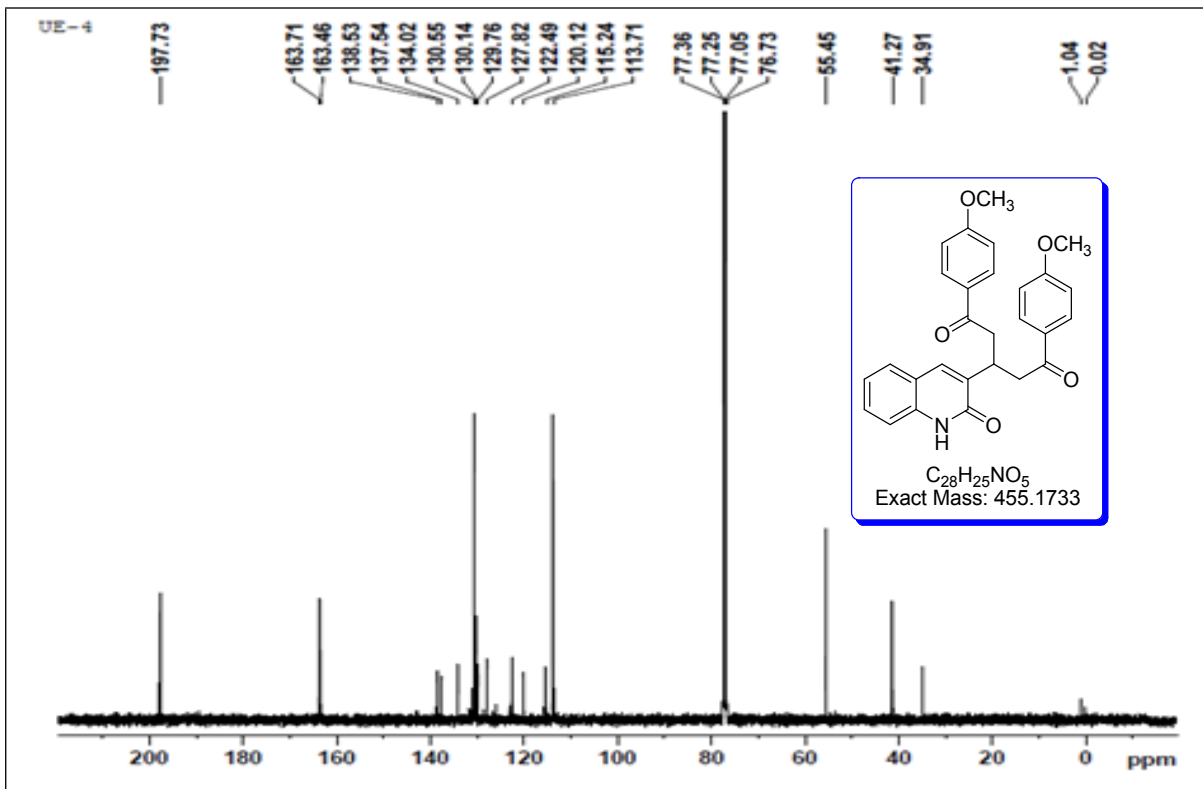
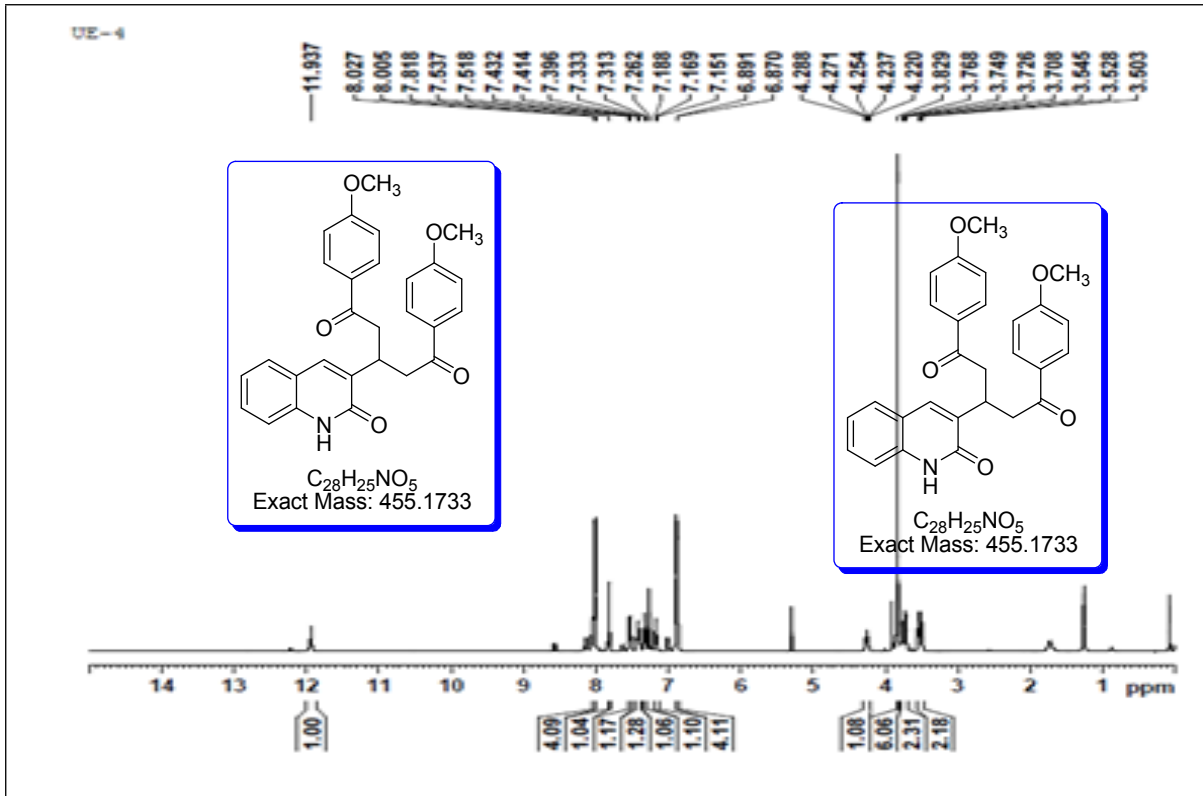


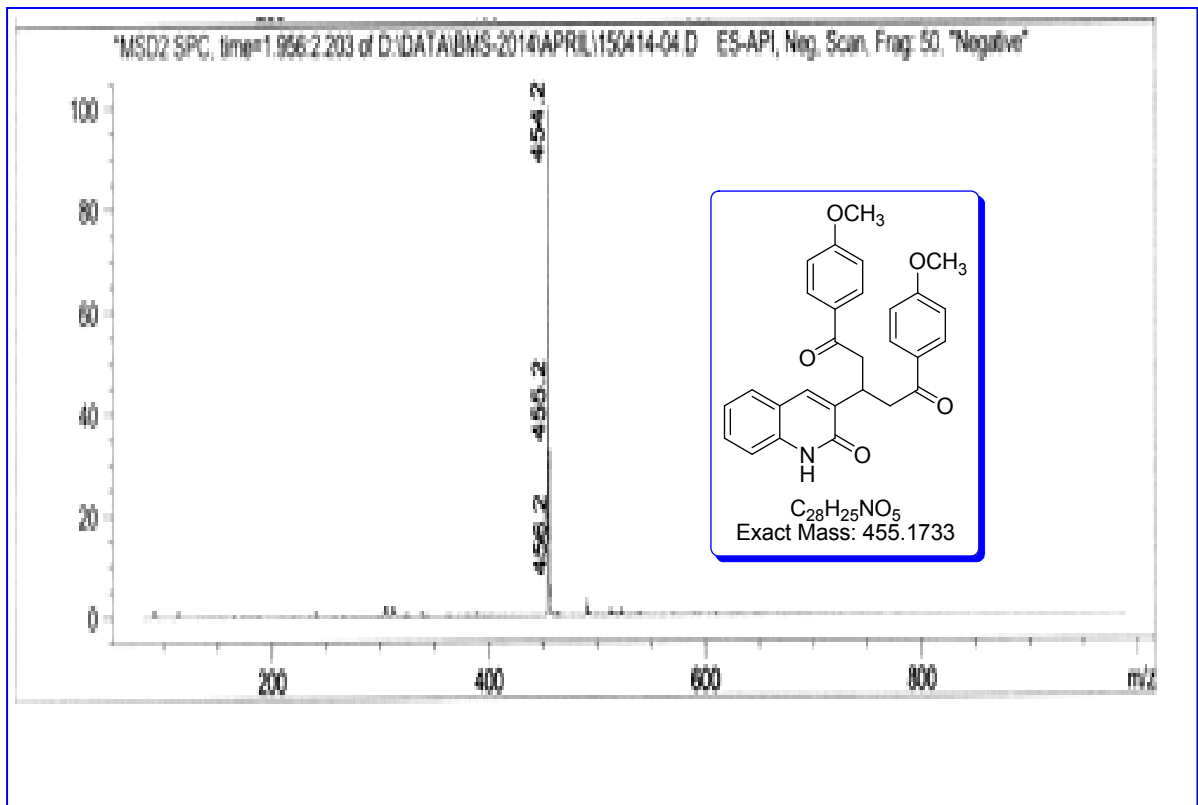
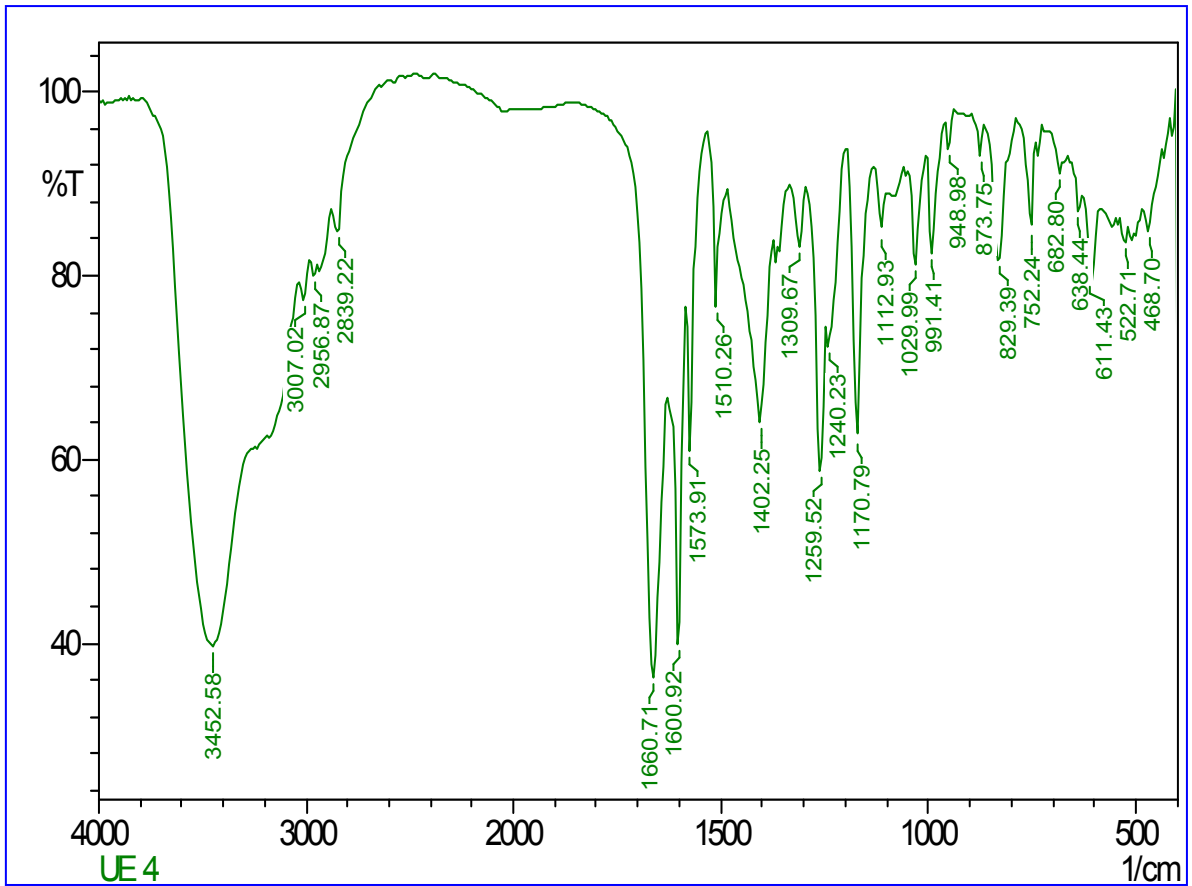


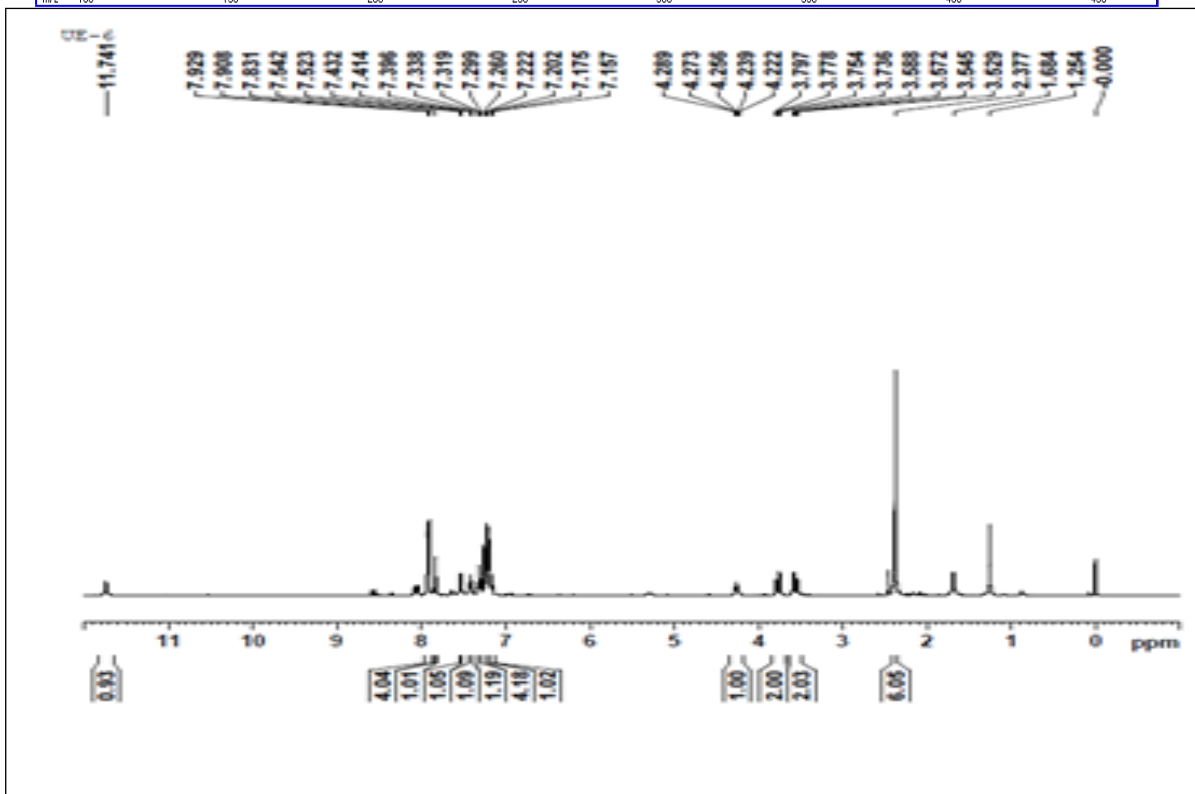
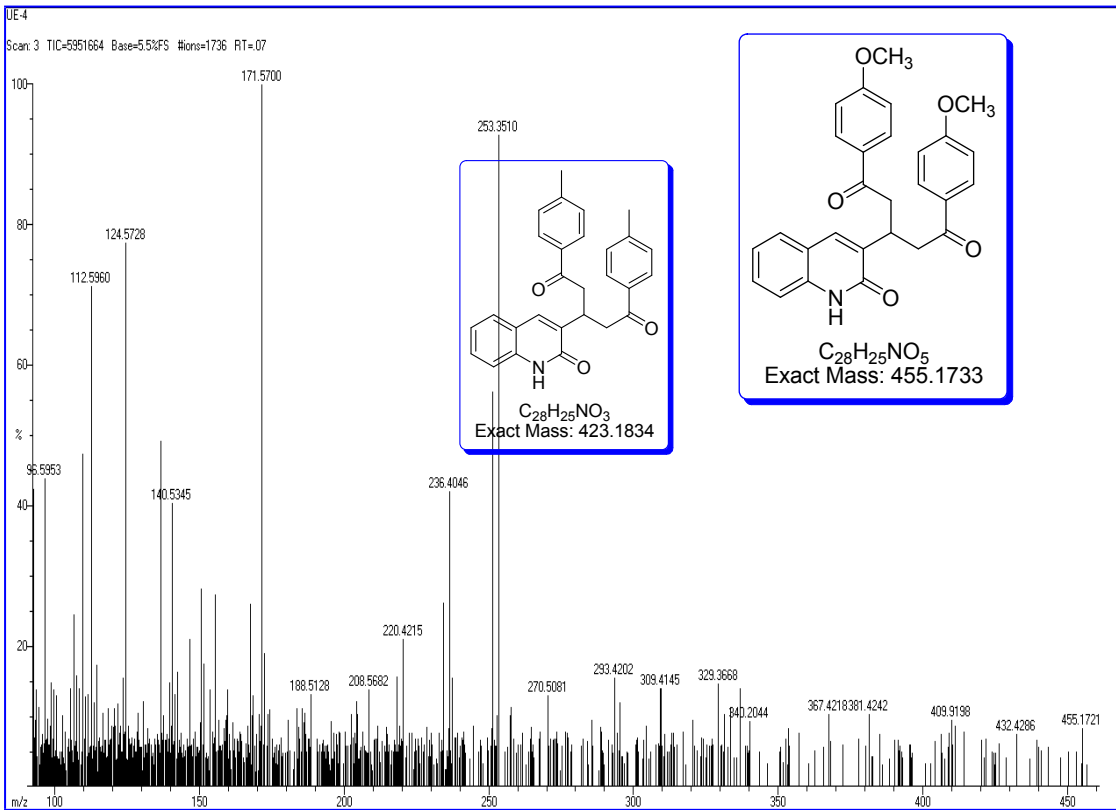
UE-3

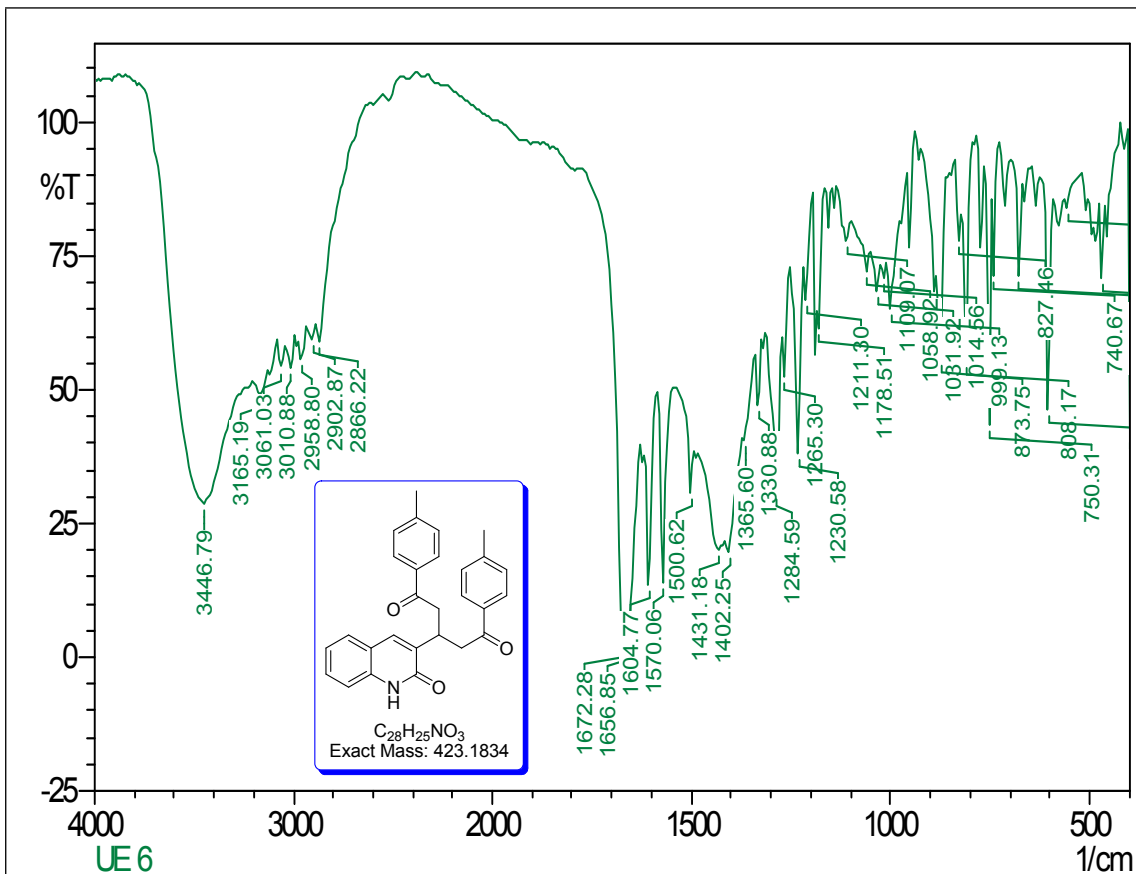
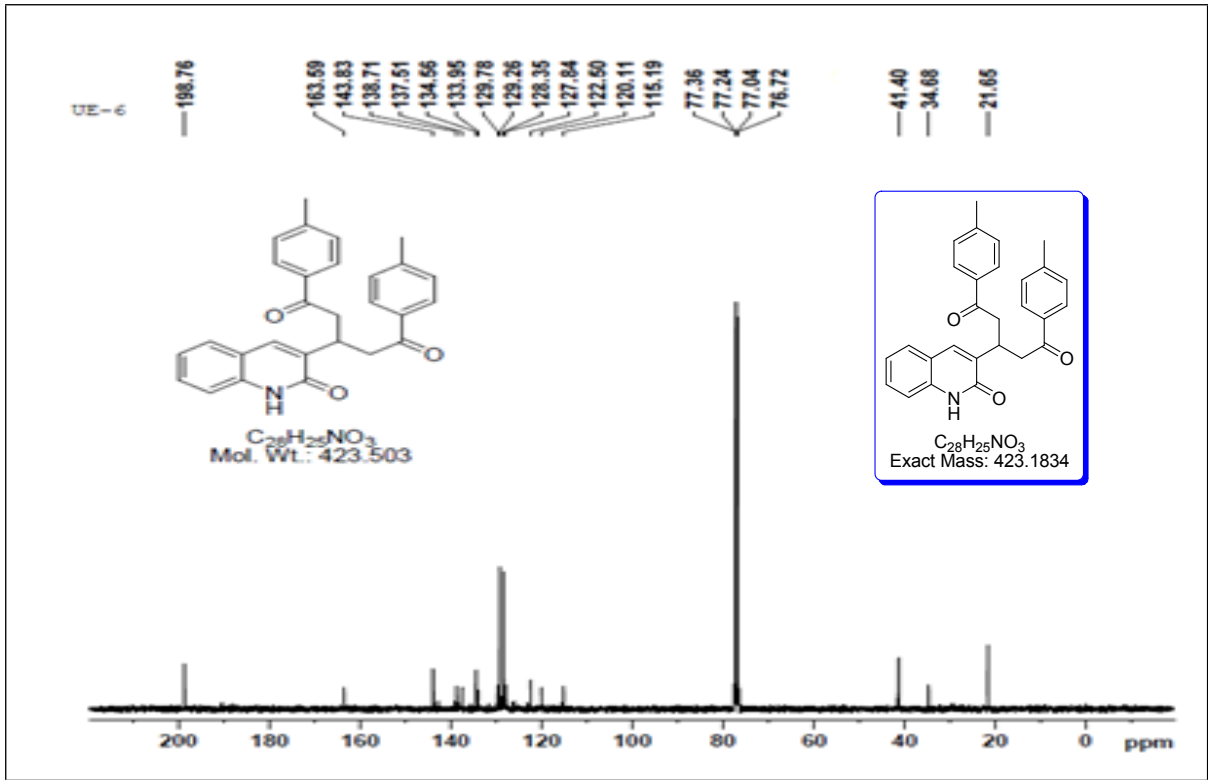
Scan: 9 TIC=9821584 Base=50.3%FS #Ions=2324 RT=22

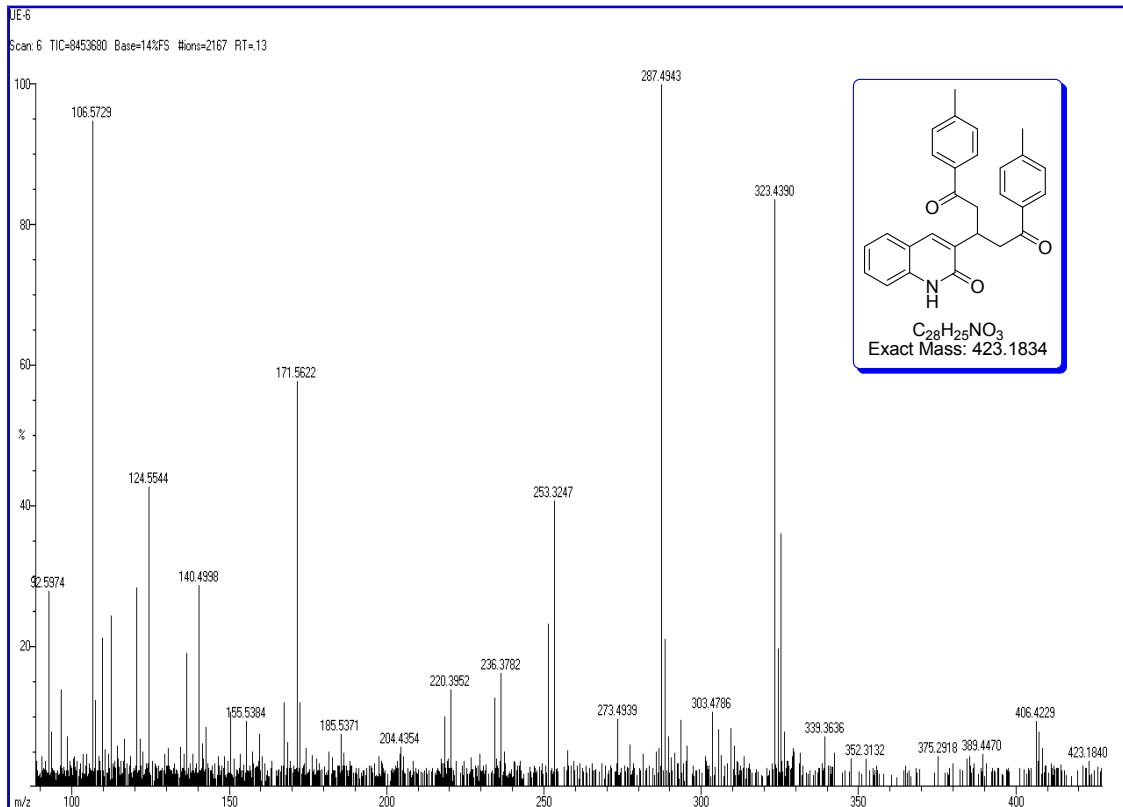
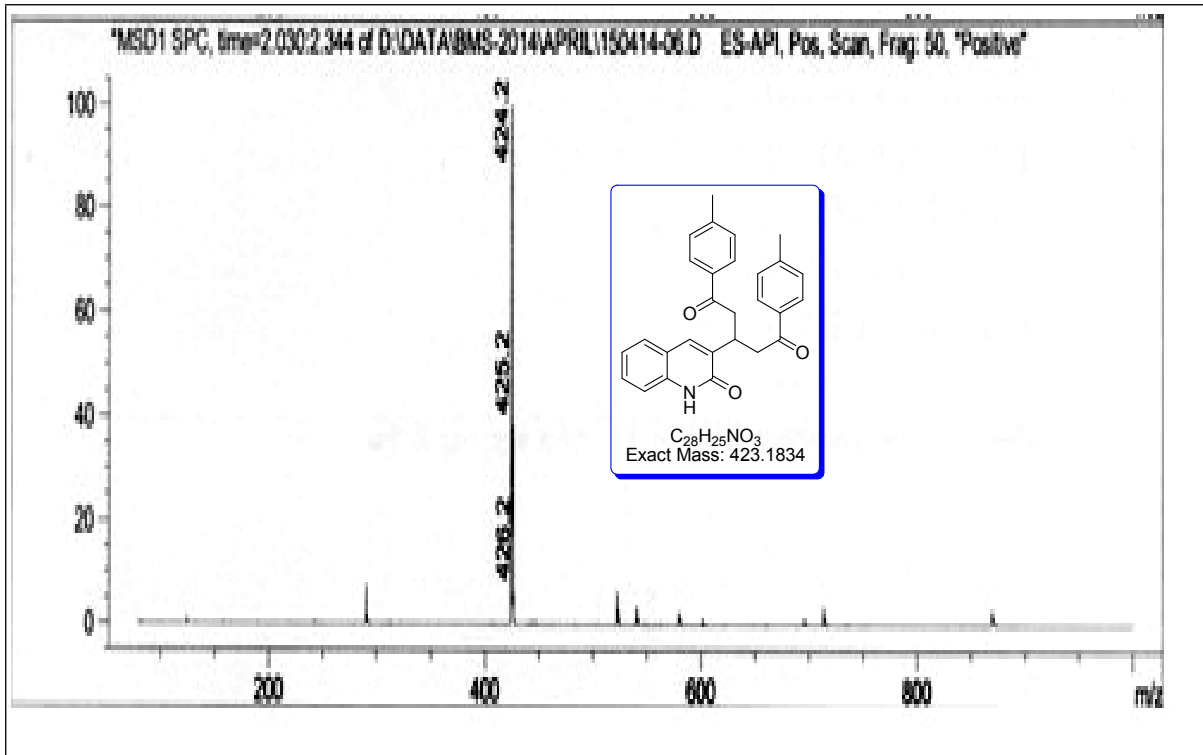


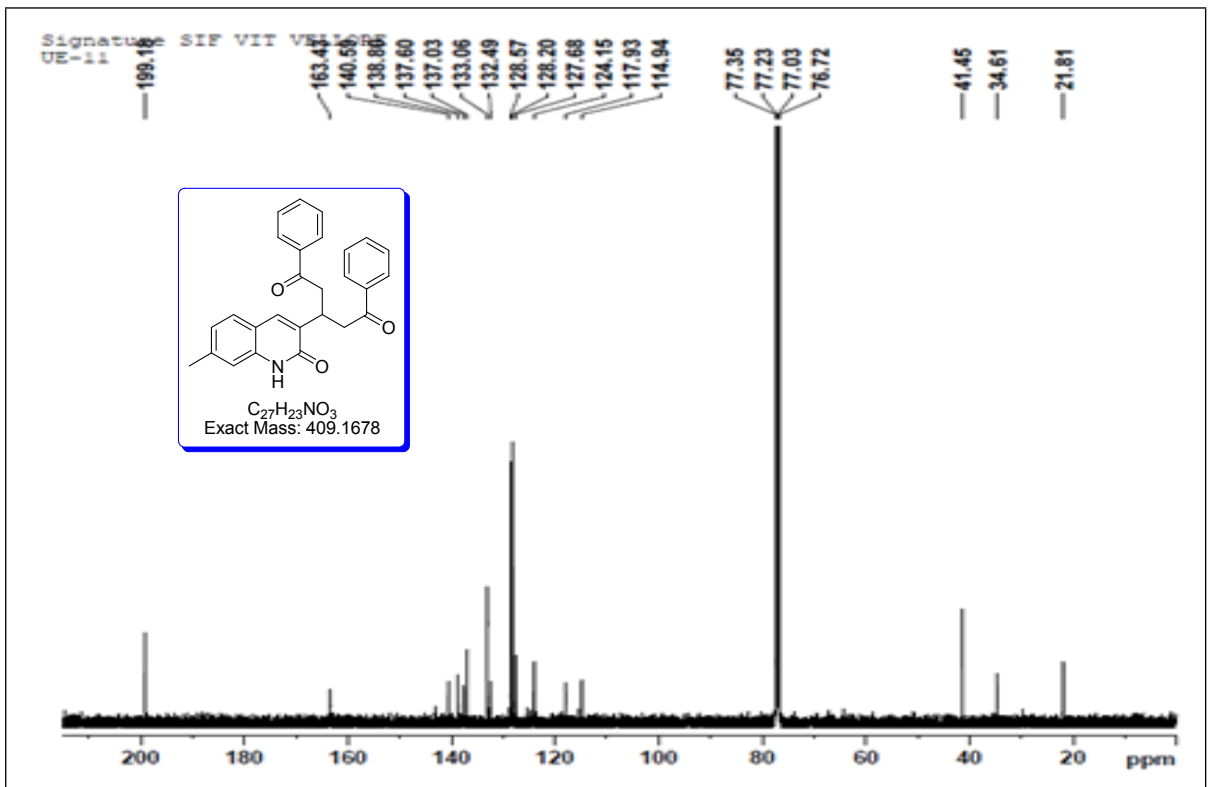
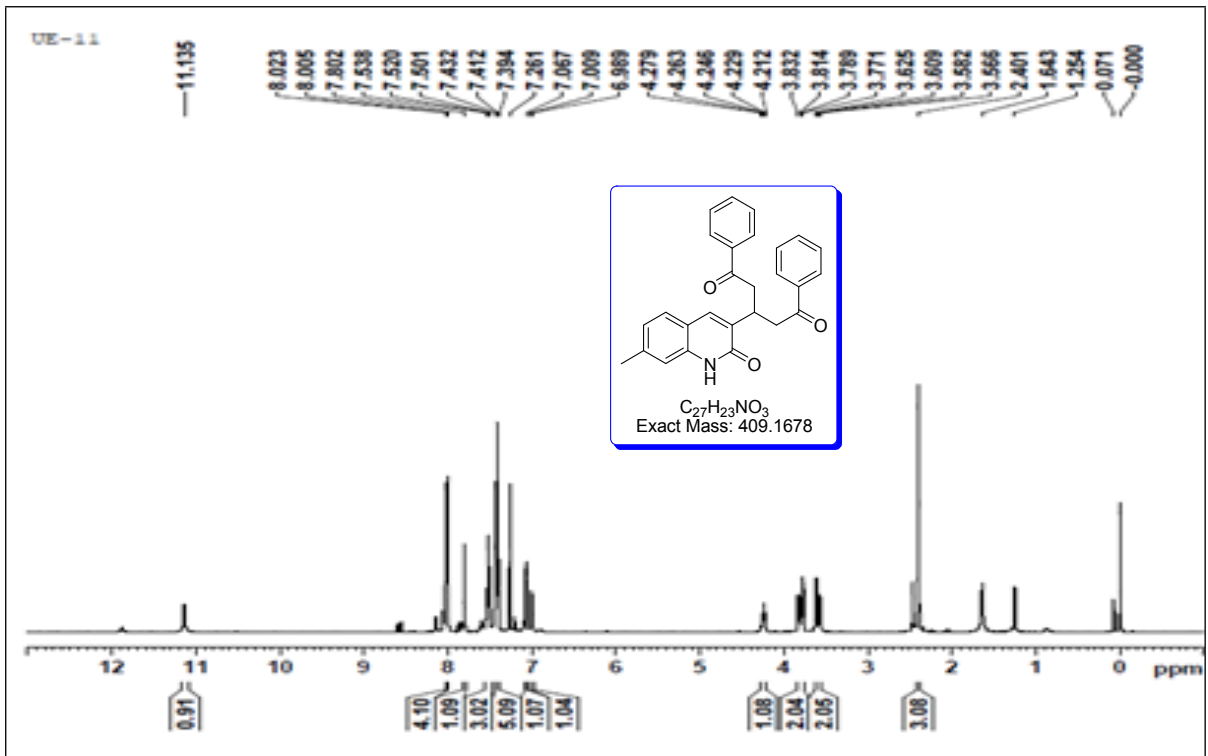


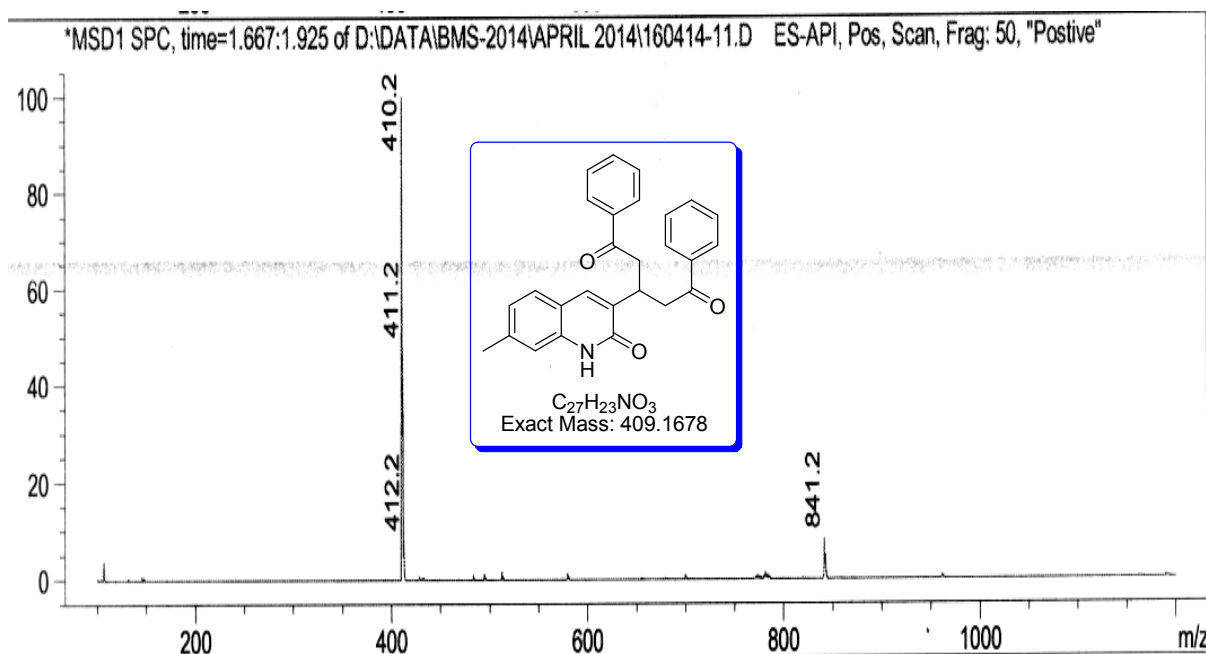
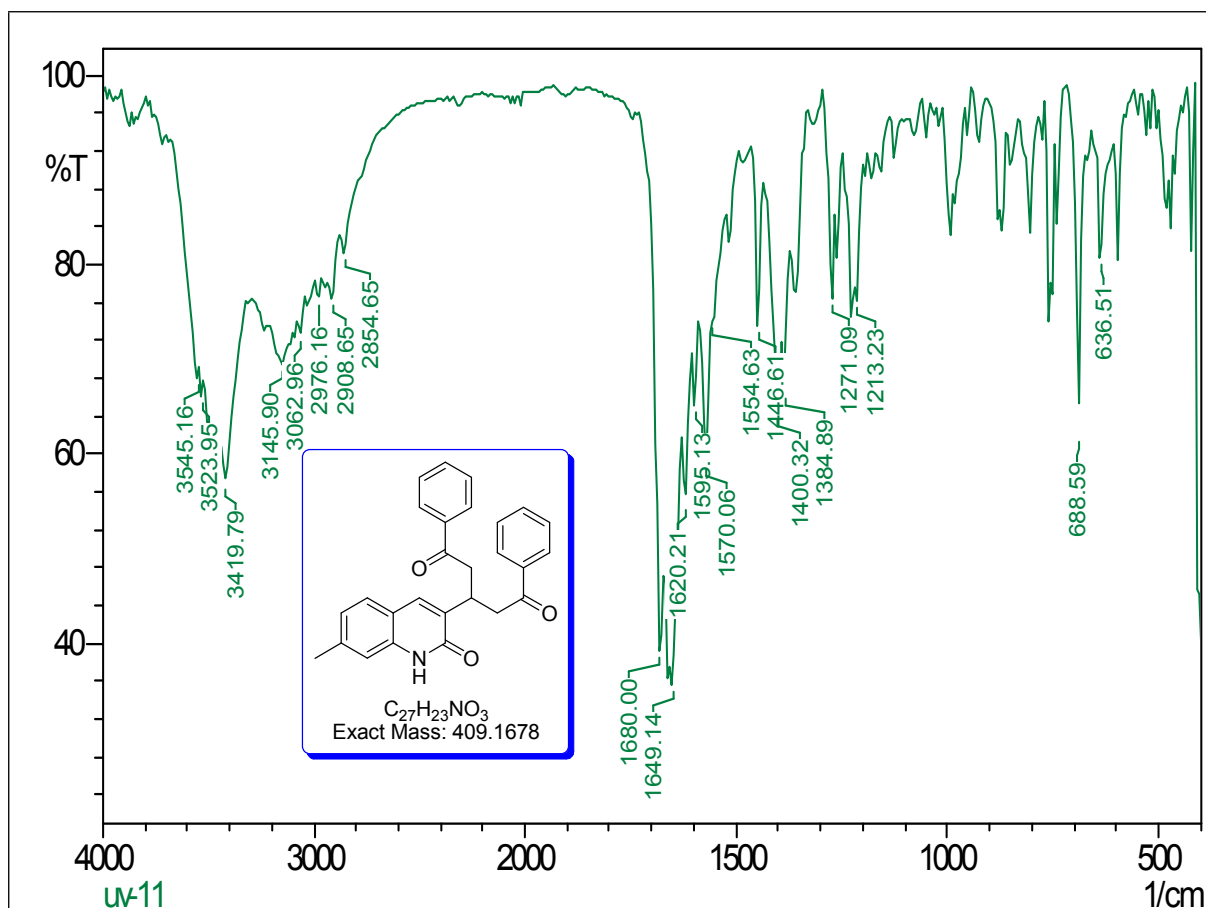






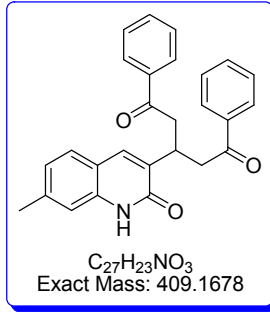
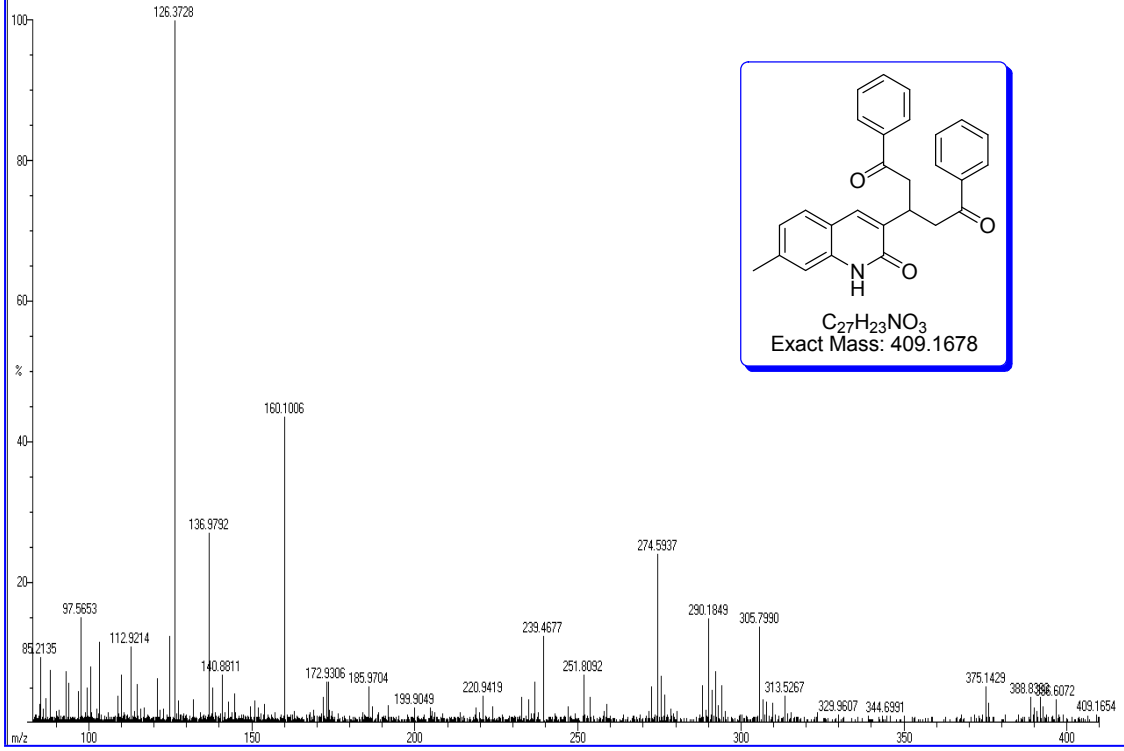


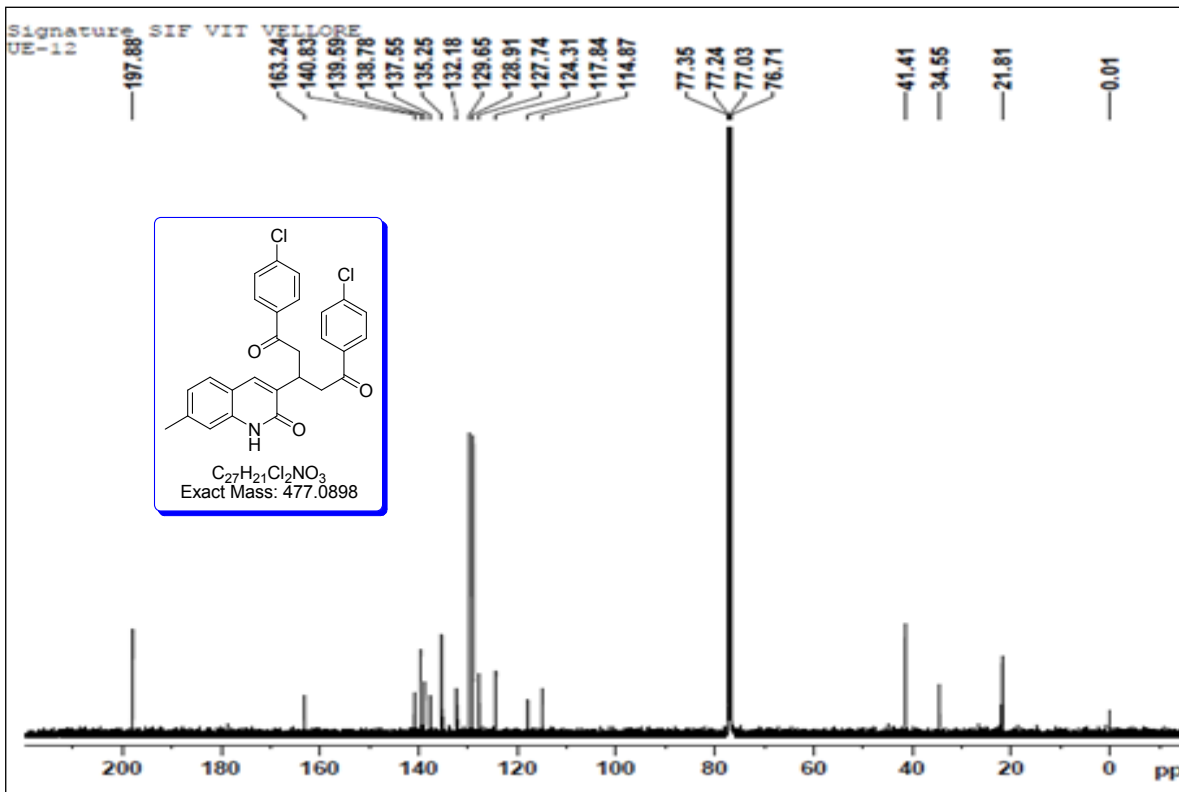
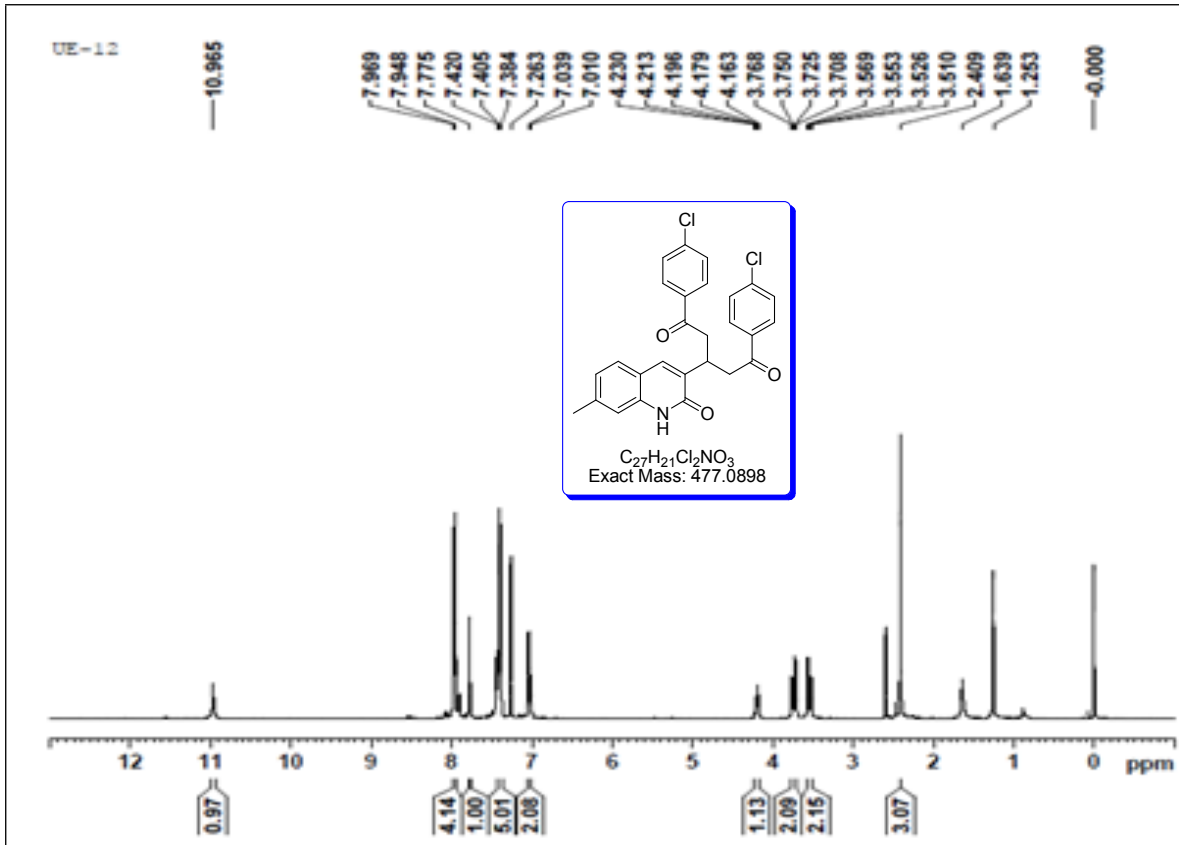


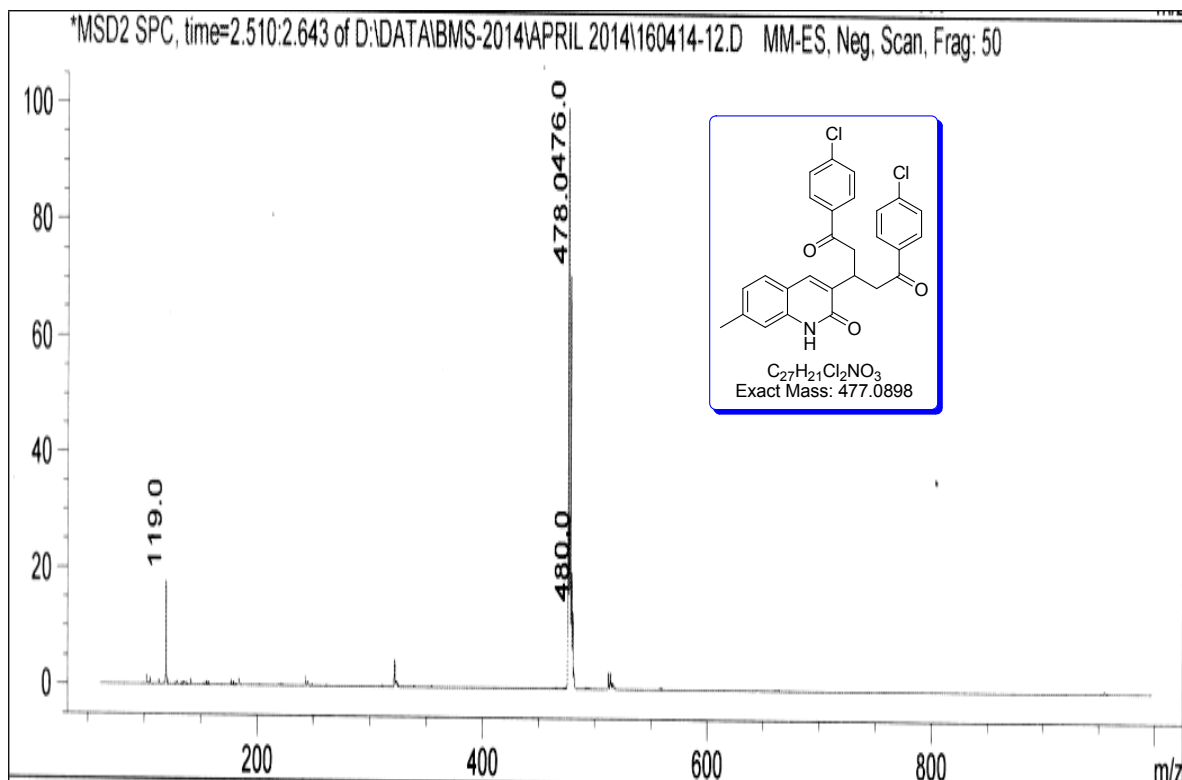
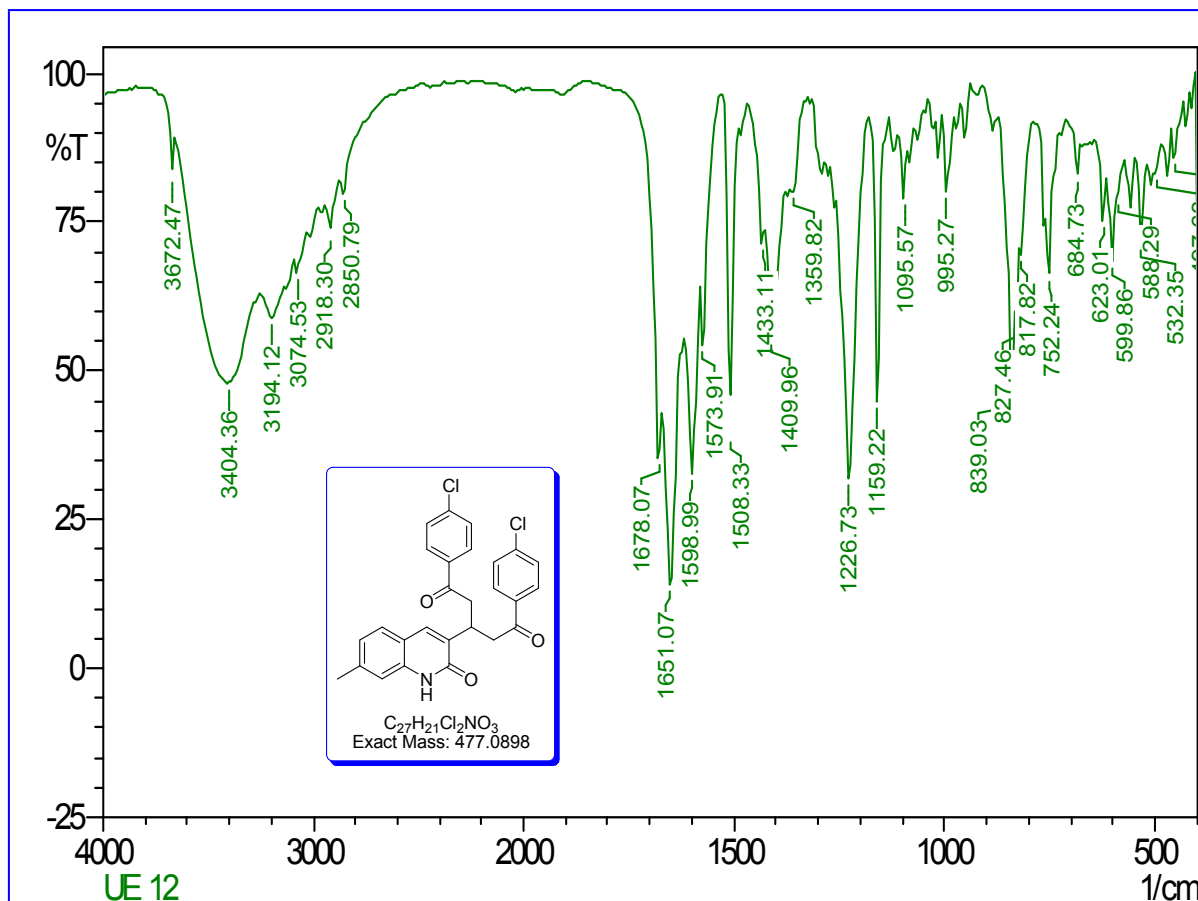


IE-11

Scan: 11 TIC=10660432 Base=44.8%FS #Ions=2359 RT=27







JE-12

Scan: 7 TIC=7738016 Base=12.4%FS #Ions=2131 RT=1.17

