Electronic supporting information

"On water" Synthesis of Highly Functionalized 4*H*-chromenes via Carbon-Carbon Bond Formation under Microwave Irradiation and Their Antibacterial Properties.

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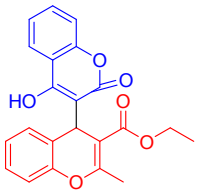
General information: All the chemicals were purchased from Sigma Aldrich and Alpha Aesar company and used without further purification as received. All ^{1}H and ^{13}C NMR spectra were recorded in CDCl3 on Avance 300 or Avance 500 spectrometers. Chemical shifts (δ) are reported in parts per million (ppm) relative to residual CHCl3 (1H: δ 7.26 ppm, 13C: δ 77.00 ppm) as an internal reference. Coupling constants (J) are reported in Hertz (Hz). Peak multiplicity is indicated as follows: s—singlet, d—doublet, t—triplet, q—quartet, m—multiplet and dd—doublet of doublet. Melting points were measured on a BUCHI melting point machine. IR spectra were recorded on Thermo Nicolet FT/IR-5700 spectrometer. Mass spectra were recorded using Waters mass spectrometer. High resolution mass spectrums (HRMS) were recorded using Applied Bio-Sciences HRMS spectrometer at national center for mass spectroscopy-IICT. S3

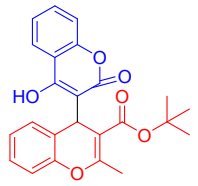
General procedure: In a microwave reaction vial 2*H*-chromene(1a)(1mmol) and 4-hydroxy coumarin(2a)(1mmol) in 2ml of water kept under microwave irradiation for 20 minutes at 105^{o}C as mentioned in Table 1. After completion of the reaction (indicated by TLC), the free flowing solid was filtered and washed with water (20 mL) to afford the desired products as pale yellow solids. The product thus obtained was recrystallized from ethanol to get pure compounds as white or pale yellow crystals. The isolated compounds were well characterized by IR, 1H NMR, 13C NMR and HRMS.

Ethyl 4-hydroxy-2'-methyl-2-oxo-2H,4'H-[3,4'-bichromene]-3'-carboxylate (3a):



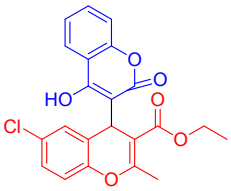
White solid; Mp 158-160° C; IR: ν_{max} 3238, 2877, 1682, 1612, 1502, 1485, 1376, 1231, 1151, 1046, 880, 751 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 8.00 (d, *J* = 7.55 Hz, 1H), 7.63 (s, 1H), 7.42-7.53 (m, 1H), 7.06-7.31 (m, 4H), 6.88-7.00 (2H, m), 5.54 (s, 1H), 4.00-4.19(m, 2H), 2.45(s, 3H), 1.20 (t, *J* = 7.18 Hz, 3H), ^{13}C NMR (75 MHz, $CDCl_3$): δ 167.2, 161.3, 151.8, 149.7, 130.7, 127.4, 126.8, 123.2, 122.9, 122.6, 121.7, 115.9, 115.4, 114.6, 108.9, 100.7, 59.5, 30.4, 19.2, 13.4; m/z (ESI); 379 $[M+H]^+$, 401 $[M+Na]^+$.

Tert-butyl 4-hydroxy-2'-methyl-2-oxo-2H,4'H-[3,4'-bichromene]-3'-carboxylate (3b):



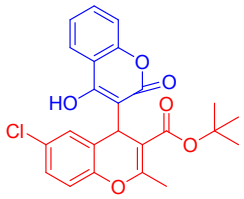
White solid; Mp 170-172° C; IR: ν_{max} 3219, 2917, 1667, 1608, 1512, 1477, 1363, 1262, 1135, 1051, 895, 761 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 10.74 (s, 1H), 7.98 (d, *J* = 8.31 Hz, 1H), 7.44-7.52 (m, 1H), 7.13-7.32 (m, 3H), 6.94-7.06 (3H, m), 5.12 (s, 1H), 2.46(s, 3H), 1.60 (s, 9H), ^{13}C NMR (75 MHz, $CDCl_3$): δ 170.6, 164.0, 161.2, 160.2, 153.0, 151.3, 131.5, 127.9, 124.2, 124.1, 123.5, 121.8, 116.1, 115.4, 114.6, 109.8, 102.0, 83.3, 30.1, 28.3, 21.3; m/z (ESI); 407 $[M+H]^+$, 429 $[M+Na]^+$. HRMS calcd for $C_{24}H_{22}O_6Na$: 429.13150, found: 429.13086.

Ethyl 6'-chloro-4-hydroxy-2'-methyl-2-oxo-2H,4'H-[3,4'-bichromene]-3'-carboxylate (3c):



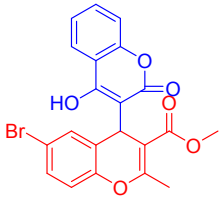
White solid; Mp 180-182° C; IR: ν_{max} 3162, 2875, 1709, 1675, 1607, 1465, 1231, 1202, 1085, 974, 751 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 8.00 (d, *J* = 7.93 Hz, 1H), 7.44-7.53 (m, 1H), 7.38 (s, 1H), 7.19-7.39 (m, 2H), 7.03-7.13 (m, 2H), 6.92 (d, *J* = 8.50 Hz, 1H), 5.39 (s, 1H), 4.07-4.26 (m, 2H), 2.46 (s, 3H), 1.25 (t, *J* = 7.93 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.6, 161.2, 160.9, 159.6, 151.8, 148.4, 130.8, 127.4, 127.1, 126.7, 123.0, 122.8, 116.1, 115.8, 115.4, 108.3, 100.6, 59.4, 30.2, 18.3, 13.3; m/z (ESI); 413 $[M+H]^+$, 435 $[M+Na]^+$. HRMS calcd for $C_{22}H_{17}O_6ClNa$: 435.06039, found: 435.06059.

Tert-butyl 6'-chloro-4-hydroxy-2'-methyl-2-oxo-2H,4'H-[3,4'-bichromene]-3'-carboxylate (3d):



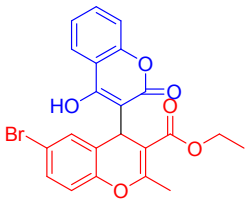
White solid; Mp 218-220° C; IR: ν_{max} 3182, 2916, 1707, 1673, 1618, 1445, 1376, 1258, 1236, 1036, 927, 749 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 8.02 (d, *J* = 7.36 Hz, 1H), 7.56 (s, 1H), 7.47-7.55 (m, 1H), 7.21-7.32 (m, 2H), 7.05-7.11 (m, 2H), 6.90 (d, *J* = 9.25 Hz, 1H), 5.42 (s, 1H), 2.40 (s, 3H), 1.41 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 164.6, 157.9, 150.8, 147.4, 130.1, 126.2, 126.1, 125.8, 123.4, 122.1, 115.4, 114.8, 114.6, 78.6, 29.4, 26.5, 17.7; m/z (ESI); 441 $[M+H]^+$, 463 $[M+Na]^+$.

Methyl 6'-bromo-4-hydroxy-2'-methyl-2-oxo-2H,4'H-[3,4'-bichromene]-3'-carboxylate (3e):



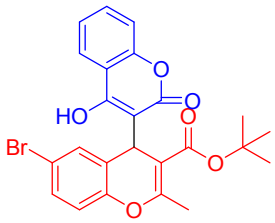
White solid; Mp 190-192° C; IR: ν_{max} 3218, 2929, 1717, 1675, 1621, 1572, 1485, 1376, 1212, 1046, 915, 751 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 8.02 (d, *J* = 8.12 Hz, 1H), 7.62 (s, 1H), 7.46-7.55 (m, 1H), 7.22-7.32 (m, 4H), 6.86 (d, *J* = 8.69 Hz, 1H), 5.53 (s, 1H), 3.66 (s, 3H), 2.44 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 167.6, 161.5, 159.1, 149.2, 131.1, 130.2, 129.9, 124.4, 123.0, 116.7, 116.0, 115.7, 115.4, 108.5, 100.6, 51.0, 30.5, 19.4 m/z (ESI); 443 $[M+H]^+$, 465 $[M+Na]^+$.

Ethyl 6-bromo-4-(4-hydroxy-2-oxo-2H-chromen-3-yl)-2-methylchroman-3-carboxylate (3f):



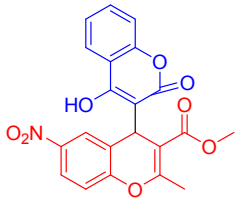
White solid; Mp 184-186° C; IR: ν_{max} 3214, 2925, 1715, 1673, 1624, 1567, 1481, 1397, 1206, 1064, 988, 754 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 10.36 (br-s, 1H), 7.93 (d, *J* = 7.93 Hz, 1H), 7.47-7.52 (m, 1H), 7.25-7.31 (m, 3H), 7.22 (d, *J* = 8.24 Hz, 1H), 7.11 (d, *J* = 2.14 Hz, 1H), 6.91 (d, *J* = 8.70 Hz, 1H), 5.10 (s, 1H), 4.23-4.34 (m, 2H), 2.48 (s, 3H), 1.34 (t, *J* = 7.17 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 171.0, 164.5, 161.1, 160.4, 152.9, 150.4, 131.8, 130.9, 130.5, 124.2, 123.8, 123.7, 117.2, 116.6, 116.5, 116.2, 109.2, 100.6, 62.2, 31.9, 21.0, 14.2 m/z (ESI); 457 $[M+H]^+$, 479 $[M+Na]^+$. HRMS calcd for $C_{24}H_{16}O_6BrNa$: 479.01056, found: 479.01248.

Tert-butyl 6'-bromo-4-hydroxy-2'-methyl-2-oxo-2H,4'H-[3,4'-bichromene]-3'-carboxylate (3g):



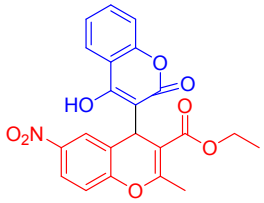
White solid; Mp 218-220° C; IR: ν_{max} 3209, 2917, 1707, 1636, 1566, 1465, 1376, 1213, 1058, 955, 756 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 8.07 (s, 1H), 8.03 (d, J = 7.74 Hz, 1H), 7.51-7.59 (m, 1H), 7.22-7.34 (m, 3H), 7.20 (d, J = 2.27 Hz, 1H), 6.88 (d, J = 8.50 Hz, 1H), 5.48 (s, 1H), 2.34 (s, 3H), 1.33 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.9, 163.9, 157.1, 150.4, 147.4, 135.6, 129.9, 128.6, 128.4, 123.6, 121.8, 115.6, 114.4, 113.3, 101.2, 78.0, 28.8, 26.0, 17.2; m/z (ESI); 485 $[M+H]^+$, 507 $[M+Na]^+$. HRMS calcd for $C_{24}H_{21}O_6BrNa$: 507.04295, found: 507.04135.

Methyl 4-hydroxy-2'-methyl-6'-nitro-2-oxo-2H,4'H-[3,4'-bichromene]-3'-carboxylate (3h):



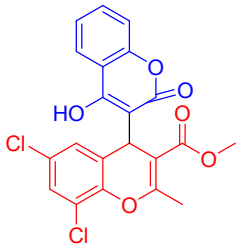
White solid; Mp 178-180° C; IR: ν_{max} 3208, 2926, 1703, 1672, 1623, 1524, 1339, 1242, 1206, 1065, 988, 759 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.99-8.09 (m, 3H), 7.65 (br-s, 1H), 7.48-7.56 (m, 1H), 7.26-7.33 (m, 1H), 7.22 (d, J = 8.12 Hz, 1H), 7.10(d, J = 9.44 Hz, 1H), 5.66 (s, 1H), 3.68 (s, 3H), 2.47 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.8, 159.5, 153.9, 151.4, 143.3, 130.6, 123.1, 122.9, 122.5, 122.3, 115.2, 115.1, 115.0, 107.2, 100.8, 50.2, 29.6, 18.2; m/z (ESI); 432 $[M+Na]^+$.

Ethyl 4-hydroxy-2'-methyl-6'-nitro-2-oxo-2H,4'H-[3,4'-bichromene]-3'-carboxylate (3i):



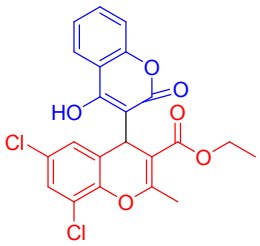
White solid; Mp 182-184° C; IR: ν_{max} 3212, 2927, 1702, 1675, 1624, 1522, 1346, 1245, 1208, 1056, 975, 756 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 10.23-10.52 (br-s, 1H), 8.08 (dd, J_1 = 9.06 Hz, J_2 = 2.64 Hz, 1H), 8.01 (dd, J_1 = 7.93 Hz, J_2 = 1.32 Hz , 1H), 7.93 (d, J = 2.45 Hz, 1H), 7.48-7.56 (m, 1H), 7.28-7.35 (m, 1H), 7.21 (d, J = 8.31 Hz, 1H), 7.14 (d, J = 8.88 Hz, 1H), 5.17 (s, 1H), 4.25-4.39 (m, 2H), 2.53 (s, 2H), 1.36 (t, J = 7.17 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 170.6, 163.7, 161.3, 161.0, 155.7, 152.9, 143.9, 132.2, 124.4, 124.0, 122.9, 116.4, 116.2, 143.9, 132.2, 124.0, 122.9, 116.4, 116.2, 108.8, 101.5, 62.5, 31.9, 20.8, 14.1; m/z (ESI); 424 $[M+H]^+$, 446 $[M+Na]^+$. HRMS calcd for $C_{22}H_{18}O_8N$: 424.10319, found: 424.10269.

Methyl 6',8'-dichloro-4-hydroxy-2'-methyl-2-oxo-2H,4'H-[3,4'-bichromene]-3'-carboxylate (3j):



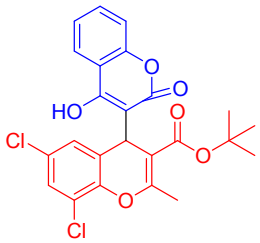
White solid; Mp 230-232° C; IR: ν_{max} 3153, 1718, 1683, 1607, 1559, 1461, 1237, 1201, 1058, 975, 756 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 8.03 (d, *J* = 7.93 Hz, 1H), 7.82 (br-s, 1H), 7.50-7.56 (m, 1H), 7.20-7.31 (m, 3H), 7.01 (d, *J* = 2.54 Hz, 1H), 5.52 (s, 1H), 4.05-4.21 (m, 2H), 2.50 (s, 3H), 1.22 (t, *J* = 6.99 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.7, 159.5, 151.3, 144.0, 130.6, 126.6, 126.5, 125.2, 124.9, 122.6, 122.4, 119.8, 115.2, 115.0, 107.3, 100.7, 50.1, 29.9, 18.1; m/z (ESI); 433 $[M+H]^+$, 455 $[M+Na]^+$. HRMS calcd for $C_{21}H_{14}O_6Cl_2Na$: 455.00601, found: 429.00596.

Ethyl 6',8'-dichloro-4-hydroxy-2'-methyl-2-oxo-2H,4'H-[3,4'-bichromene]-3'-carboxylate (3k):



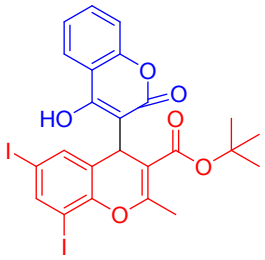
White solid; Mp 220-222° C; IR: ν_{max} 3150, 1717, 1681, 1568, 1475, 1243, 1207, 1076, 965, 753 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 8.01 (d, *J* = 7.93 Hz, 1H), 7.47-7.55 (m, 1H), 7.46 (br-s, 1H), 7.22-7.31 (m, 2H), 7.19 (d, *J* = 2.45 Hz, 1H), 7.03 (d, *J* = 2.45 Hz, 1H), 5.57 (s, 1H), 3.64 (s, 3H), 2.47 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.6, 160.6, 152.1, 144.8, 131.2, 127.5, 127.3, 125.9, 125.5, 123.1, 120.6, 115.8, 108.4, 101.6, 59.9, 30.8, 18.9, 13.5; m/z (ESI); 447 $[M+H]^+$, 469 $[M+Na]^+$. HRMS calcd for $C_{22}H_{16}O_6Cl_2Na$: 469.02214, found: 469.02161.

Tert-butyl 6',8'-dichloro-4-hydroxy-2'-methyl-2-oxo-2H,4'H-[3,4'-bichromene]-3'-carboxylate (3l):



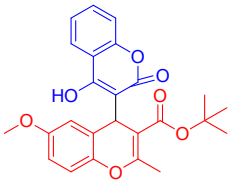
White solid; Mp 216-218° C; IR: ν_{max} 3148, 1715, 1676, 1542, 1455, 1239, 1198, 1081, 974, 757 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 8.04 (d, *J* = 6.98 Hz, 1H), 8.01 (s, 1H), 7.51-7.58 (m, 1H), 7.23-7.33 (m, 3H), 7.03 (d, *J* = 2.26 Hz, 1H), 5.51 (s, 1H), 2.39 (s, 3H), 1.35 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 164.1, 157.6, 151.0, 143.8, 130.4, 126.2, 124.9, 122.3, 119.6, 114.9, 114.8, 102.3, 79.1, 30.0, 26.6, 17.7; m/z (ESI); 475 $[M+H]^+$, 497 $[M+Na]^+$.

Tert-butyl 4-hydroxy-6',8'-diiodo-2'-methyl-2-oxo-2H,4'H-[3,4'-bichromene]-3'-carboxylate (3m):

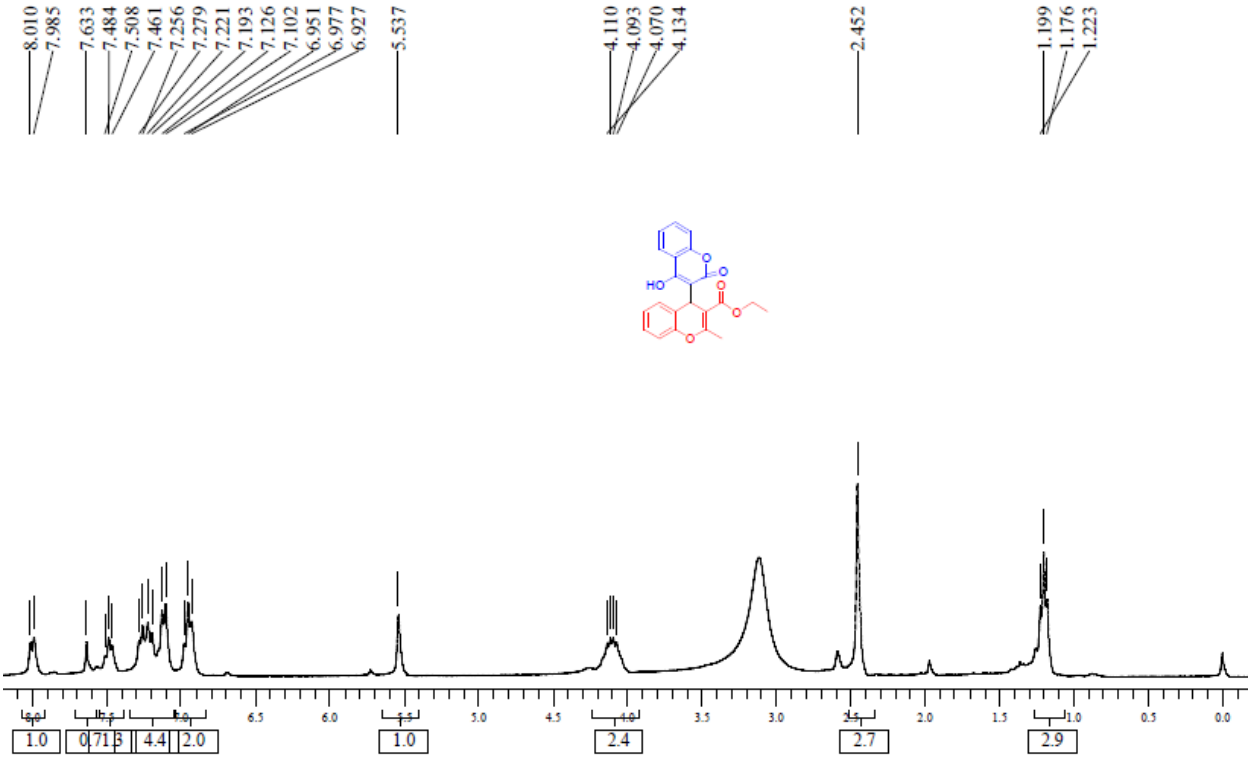


White solid; Mp 214-216° C; IR: ν_{max} 3292, 2966, 2922, 1691, 1668, 1623, 1446, 1396, 1254, 1201, 1065, 900, 755 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 8.04 (d, J = 7.74 Hz, 1H), 7.89-7.96 (br-s, 1H), 7.84 (d, J = 1.70 Hz, 1H), 7.50-7.58 (m, 1H), 7.37 (s, 1H), 7.23-7.33 (m, 2H), 5.50 (s, 1H), 2.40 (s, 3H), 1.36 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.3, 160.0, 151.6, 148.9, 143.4, 136.0, 130.8, 125.4, 122.8, 122.7, 115.5, 115.4, 108.2, 85.9, 84.1, 79.9, 30.6, 27.2, 18.5; m/z (ESI); 659 $[M+H]^+$, 681 $[M+Na]^+$. HRMS calcd for $C_{24}H_{21}O_6I_2$: 658.94300, found: 680.92480.

Tert-butyl 4-hydroxy-6'-methoxy-2'-methyl-2-oxo-2H,4'H-[3,4'-bichromene]-3'-carboxylate (3n):



White solid; Mp 178-180° C; IR: ν_{max} 3203, 2973, 1675, 1619, 1561, 1498, 1226, 1163, 1065, 992, 763 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.98 (dd, J_1 = 7.93 Hz, J_2 = 1.53 Hz , 1H), 7.46-7.50 (m, 1H), 7.27-7.30 (m, 1H), 7.21 (d, J = 8.39 Hz, 1H), 6.96 (d, J = 9.00 Hz, 1H), 6.92 (d, J = 8.50 Hz, 1H), 6.72 (dd, J_1 = 9.00 Hz, J_2 = 2.90 Hz, 1H), 6.48 (d, J = 2.90 Hz, 1H), 5.10 (s, 1H), 3.70 (s, 3H), 2.44 (s, 3H), 1.52 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 170.7, 164.3, 161.2, 160.3, 156.1, 152.9, 145.6, 131.5, 124.1, 123.5, 122.6, 116.9, 116.2, 113.6, 112. 3, 109.7, 101.2, 83.2, 55.6, 32.5, 28.3, 21.4; m/z (ESI); 459 $[M+Na]^+$. HRMS calcd for $C_{25}H_{24}O_7Na$: 459.14241, found: 459.14142.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7.193
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6.927
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4.110
4.093
4.070
4.134
2.452
1.199
1.176
1.223
HO
O
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O
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4.4
2.0
1.0
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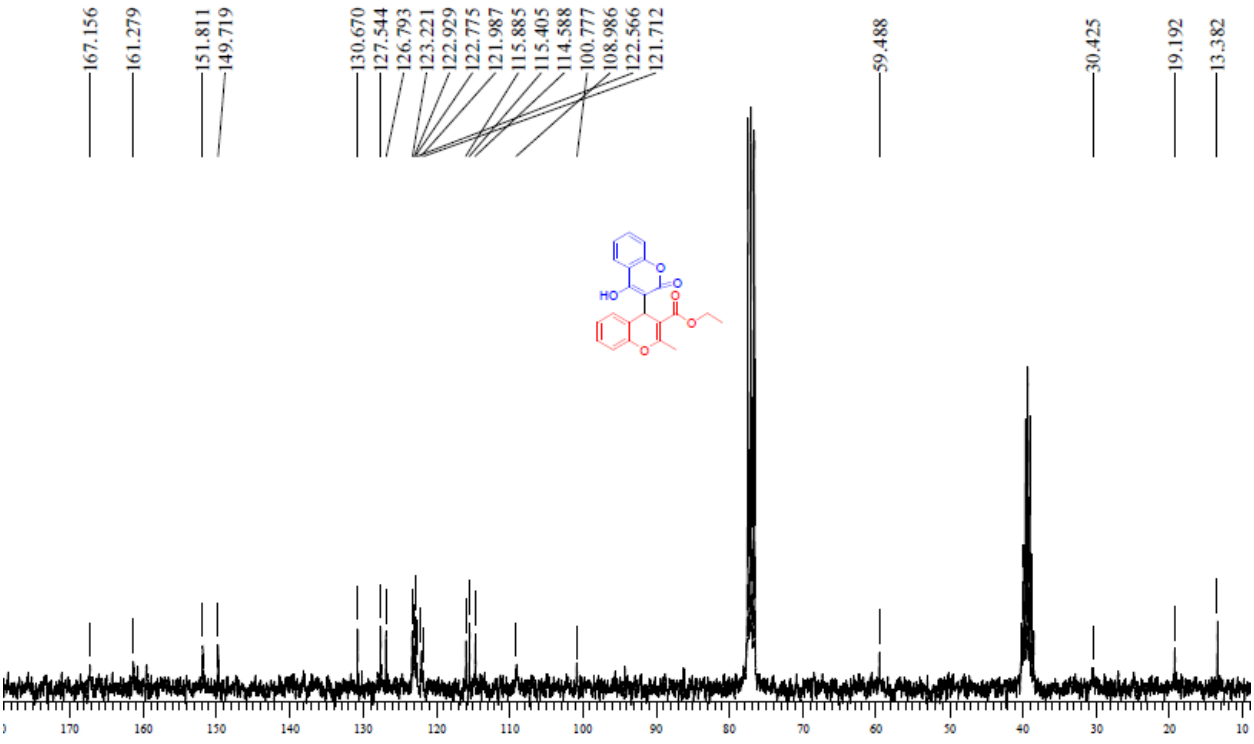
2.7
2.9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114.588
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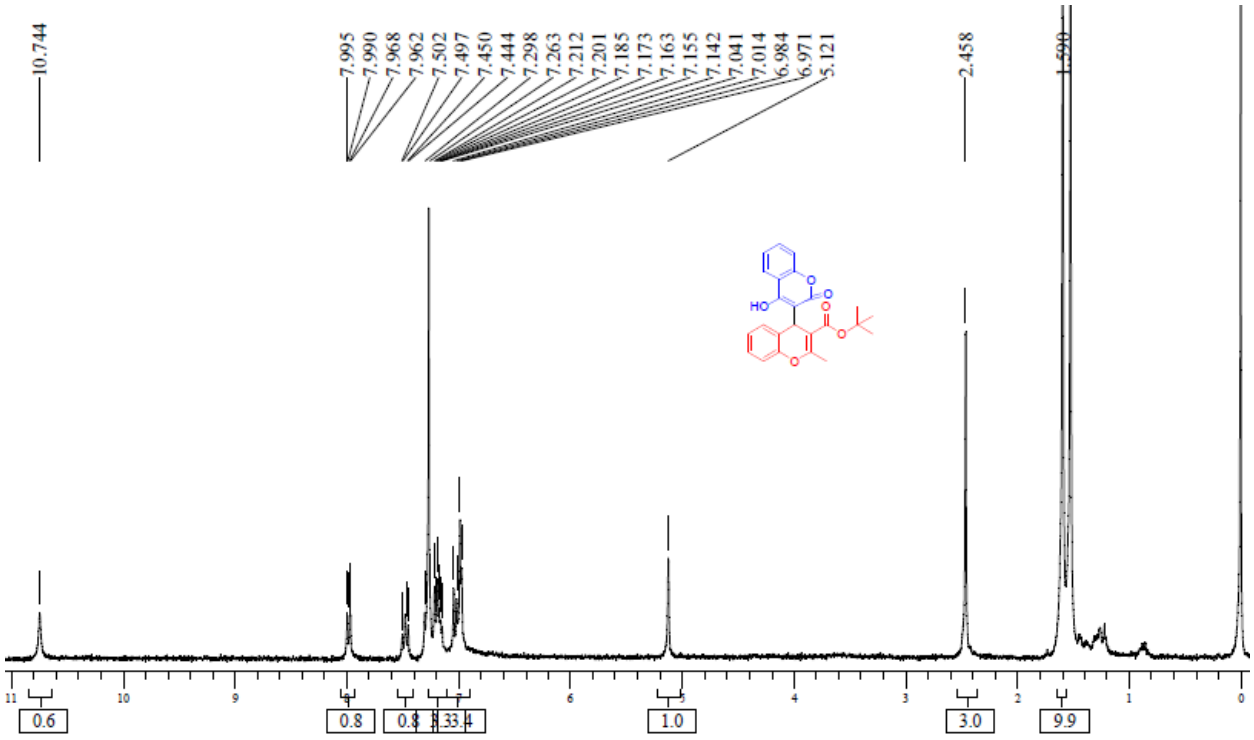
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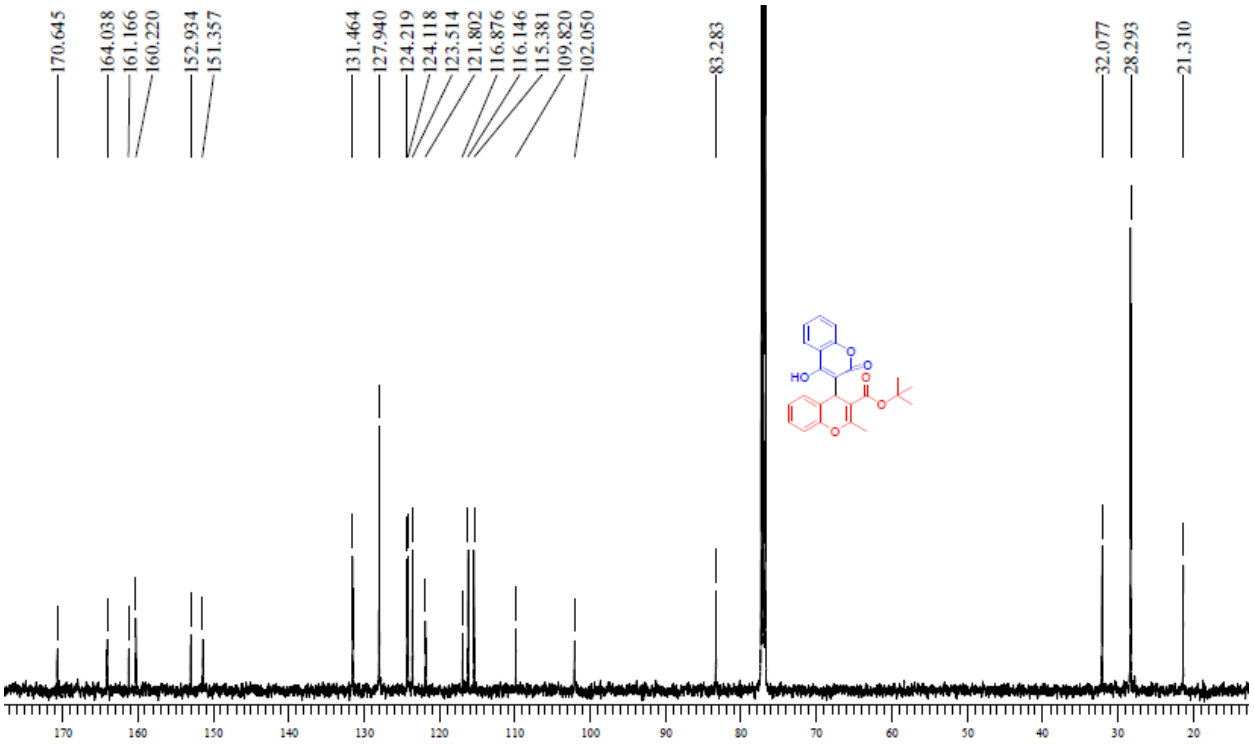
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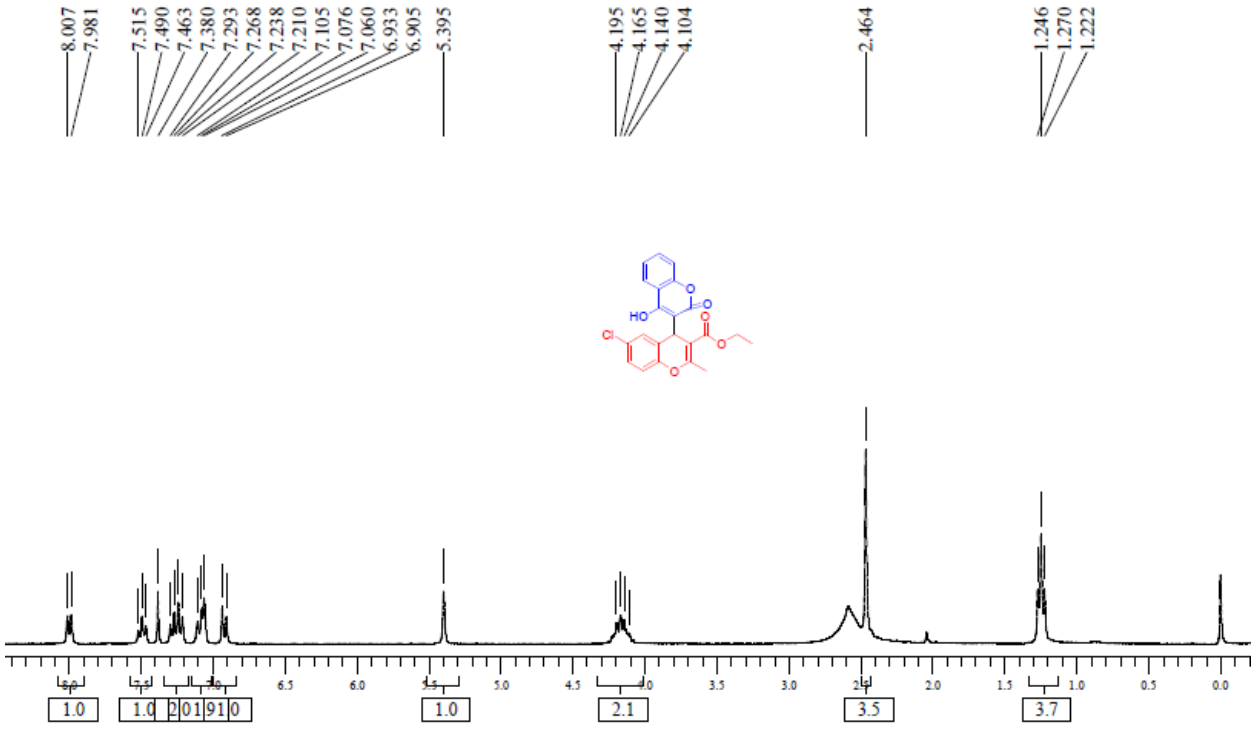
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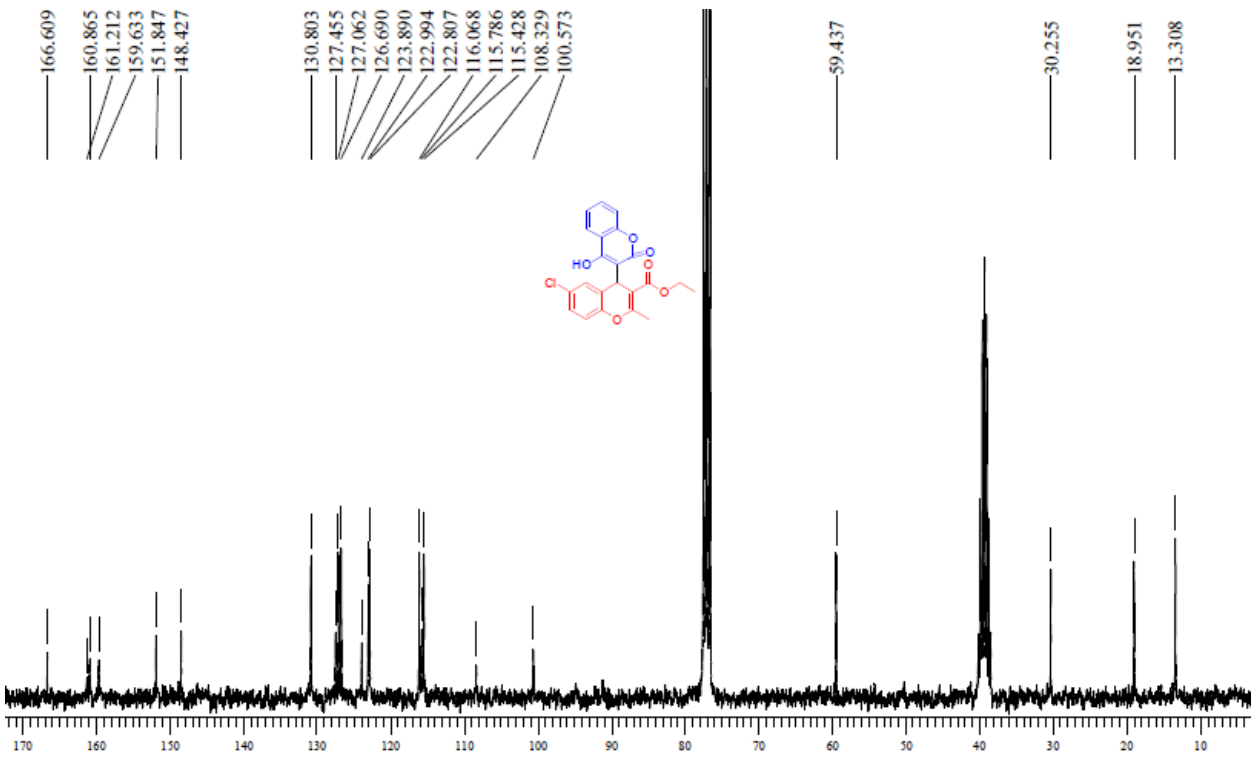
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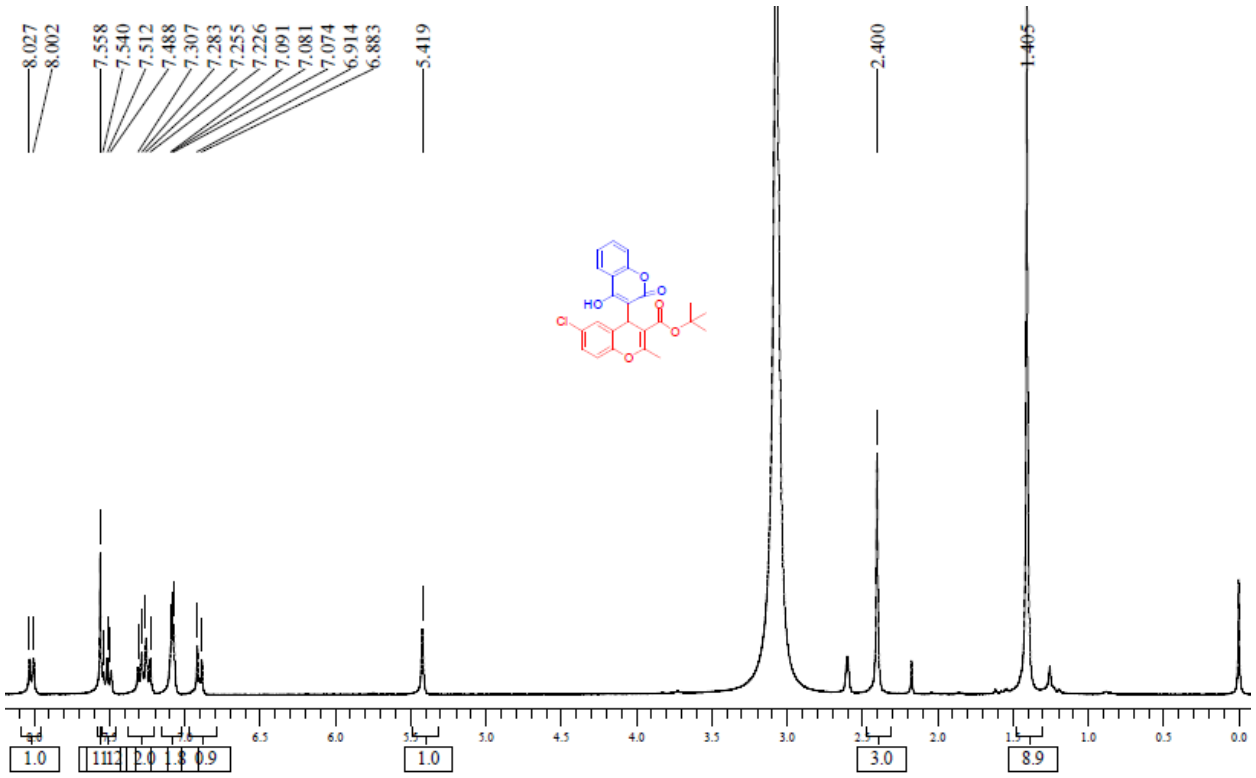
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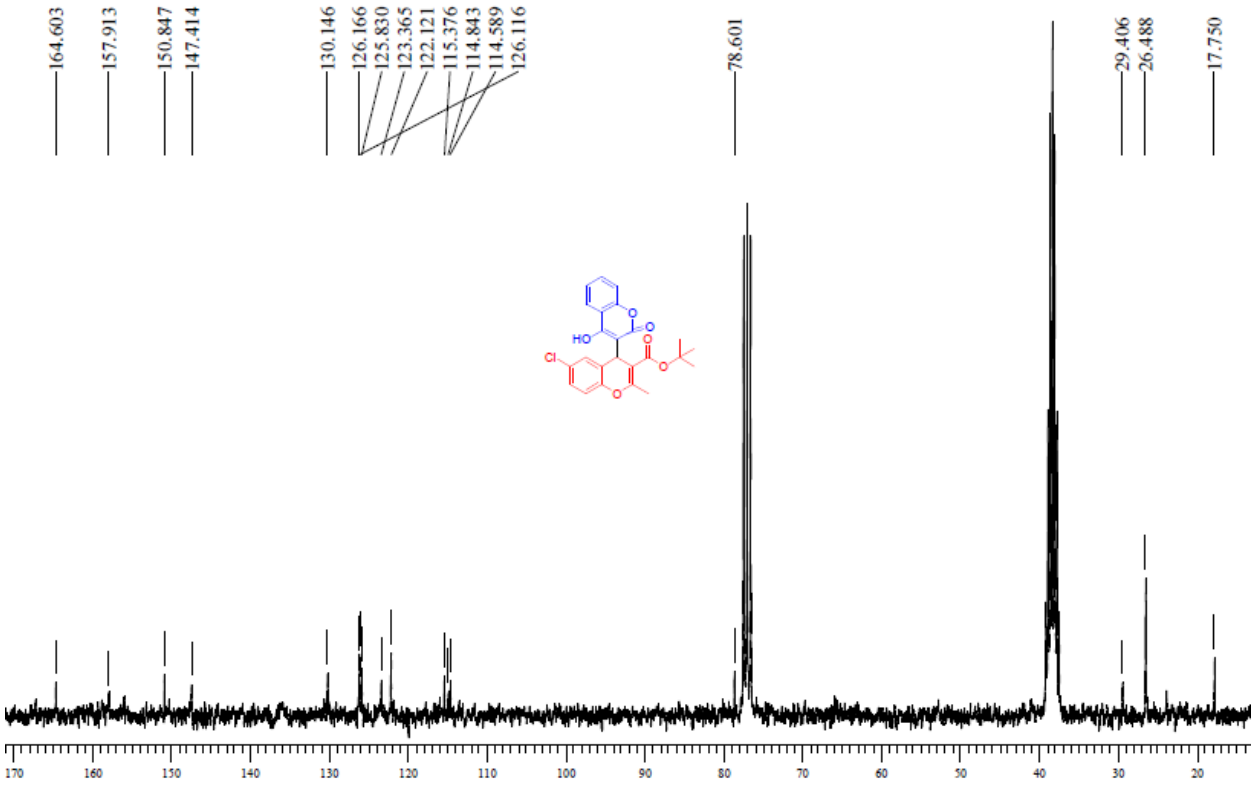
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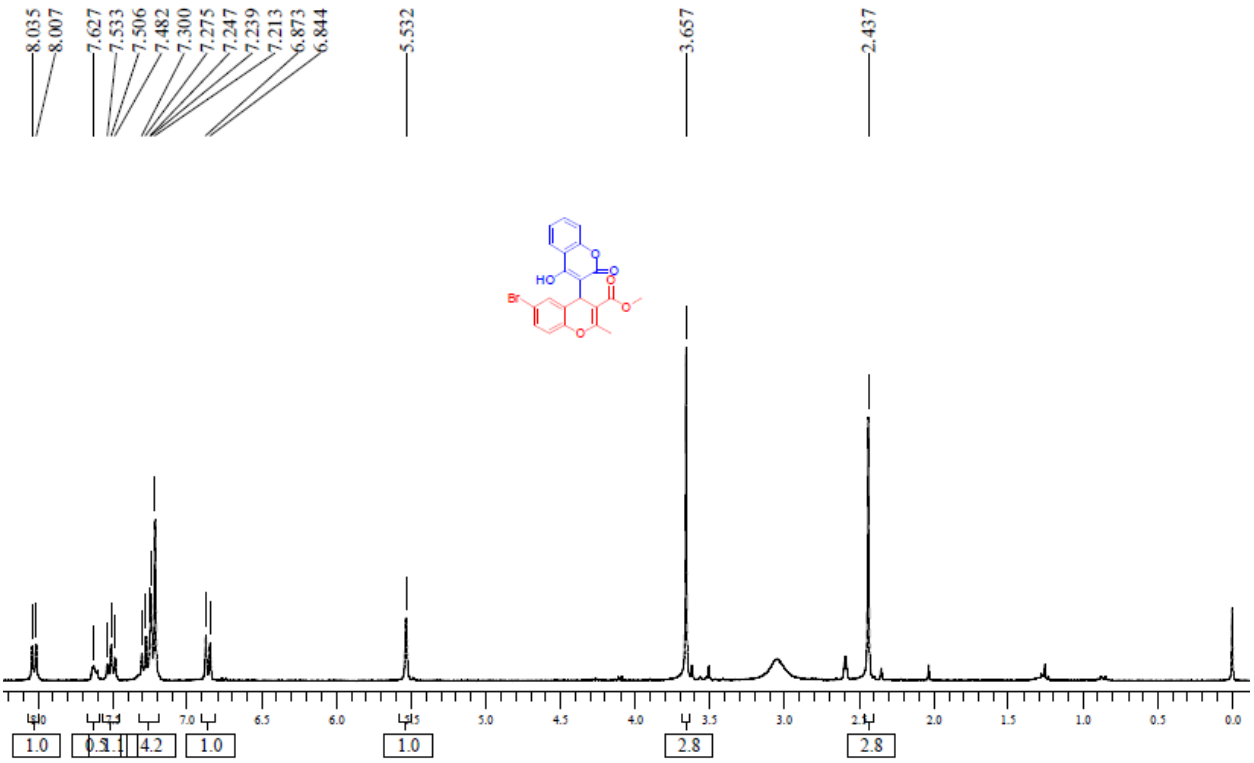
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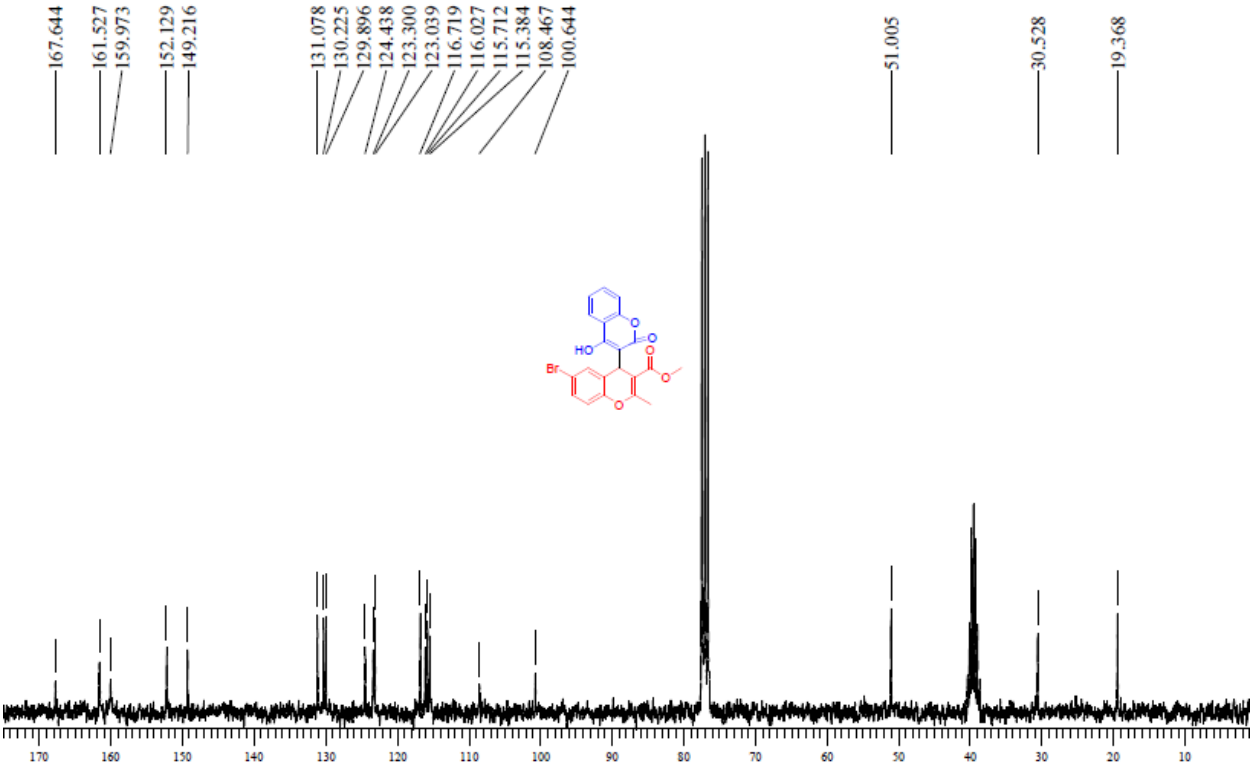
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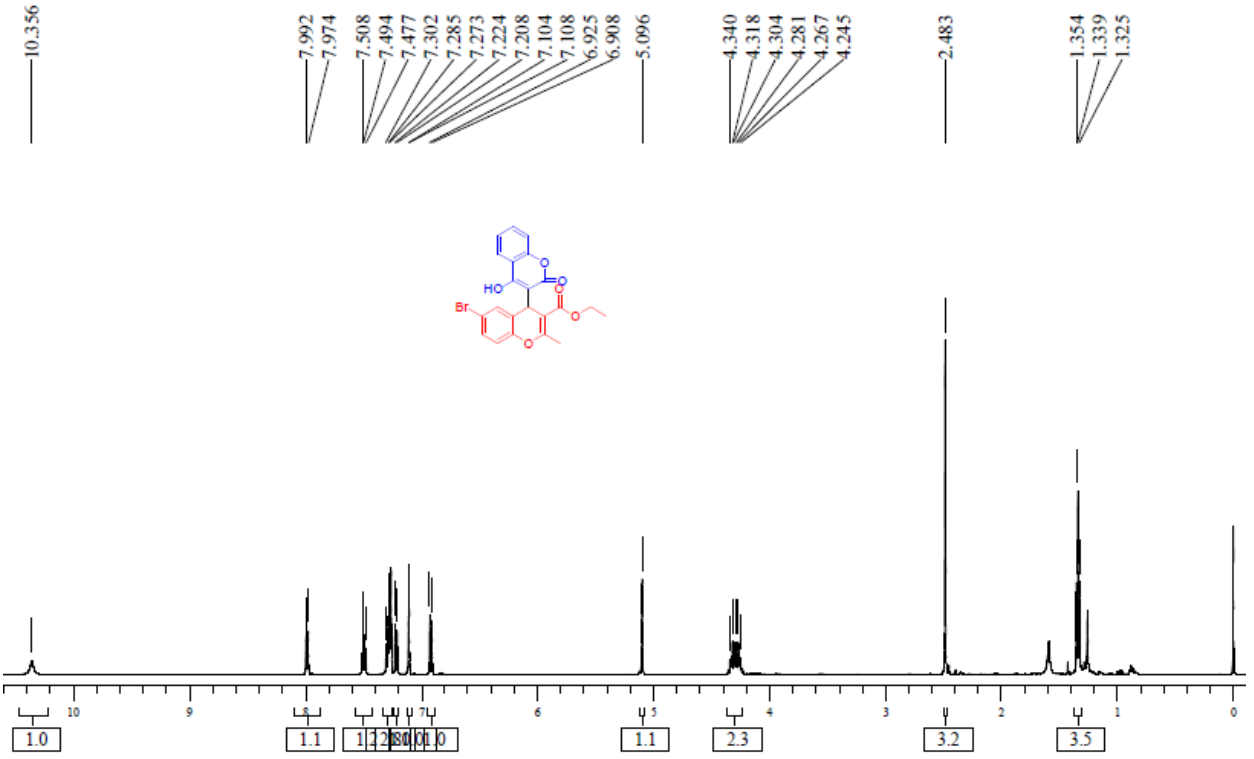
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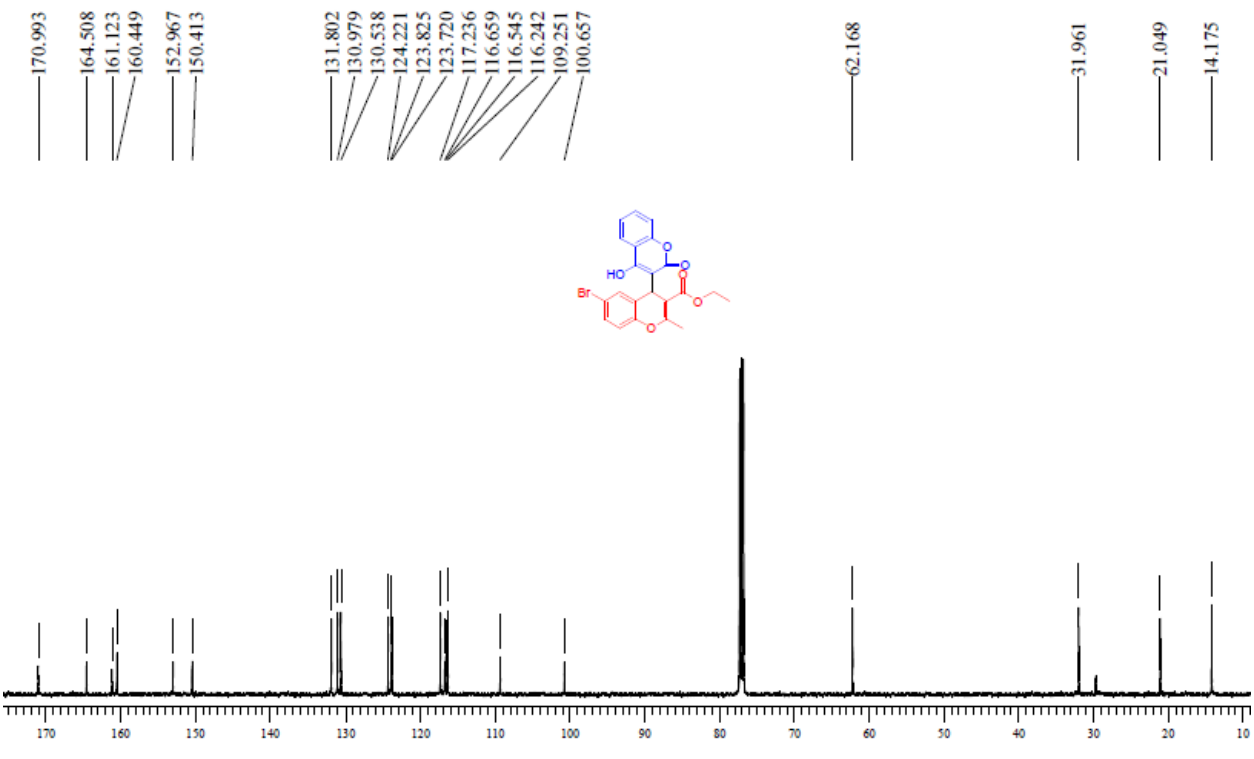
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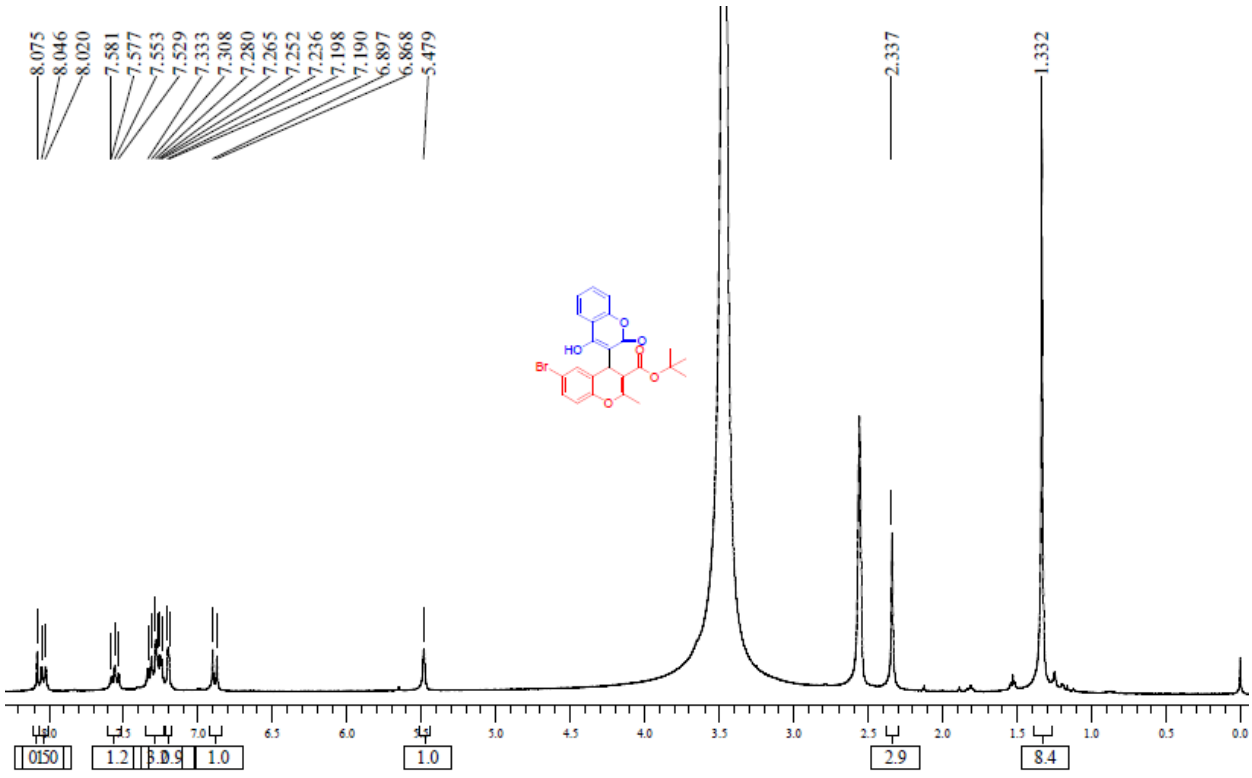
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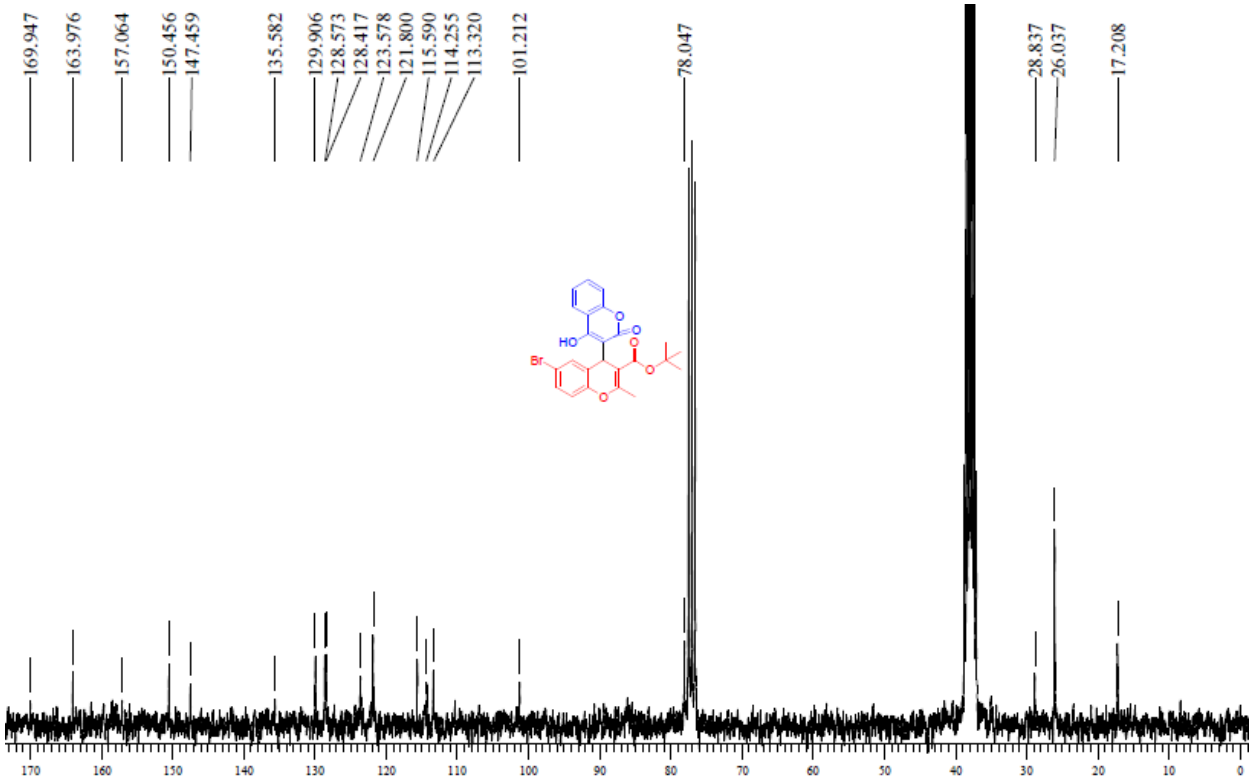
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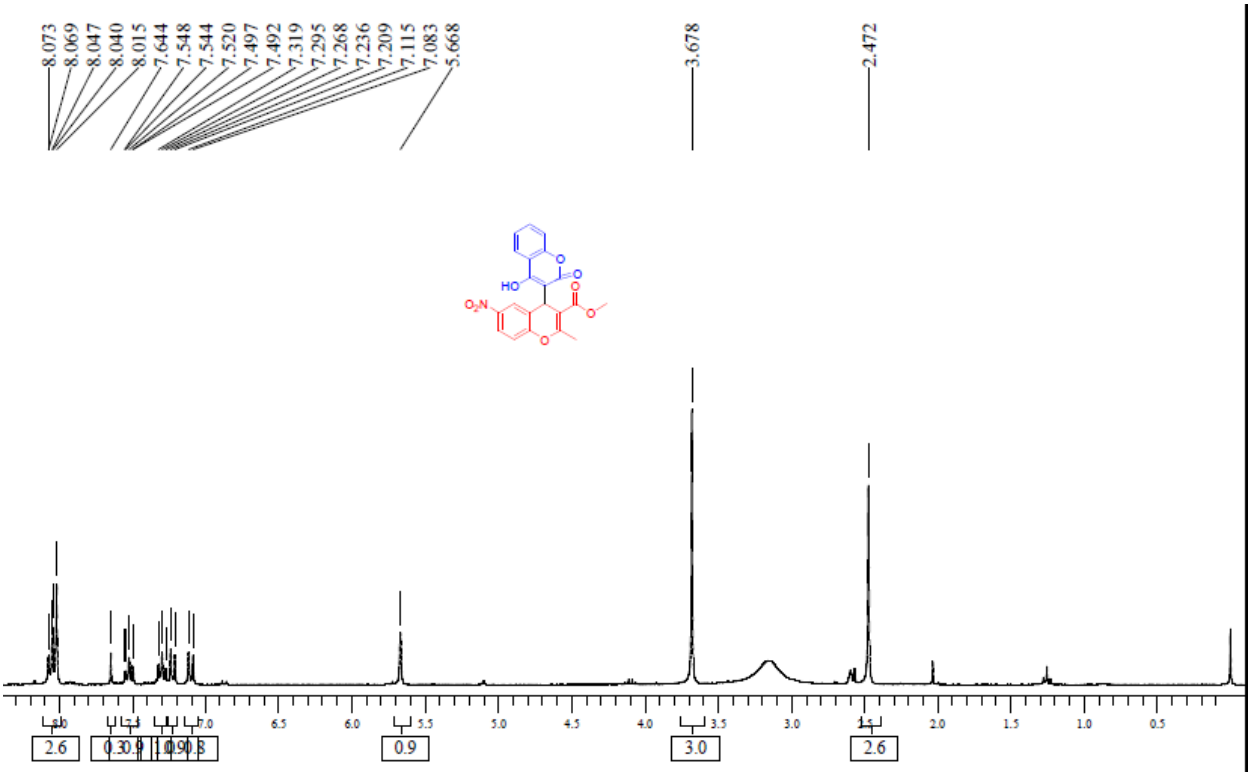
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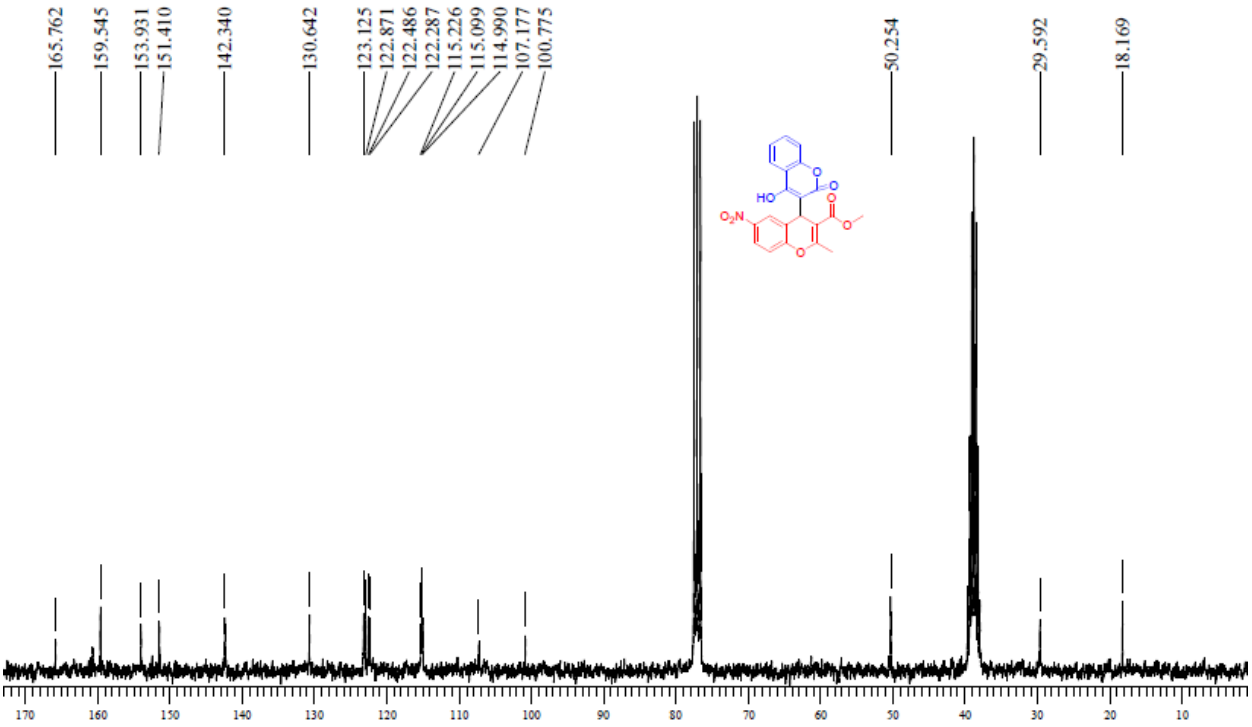
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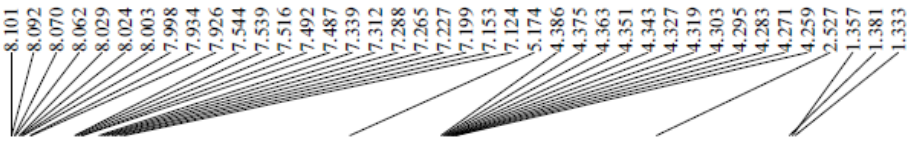
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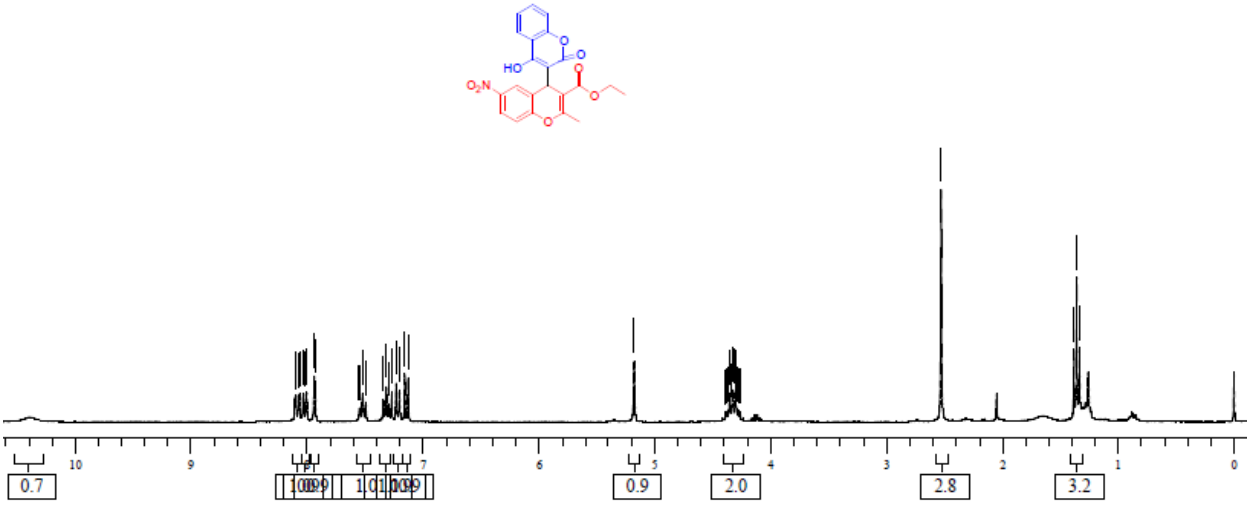
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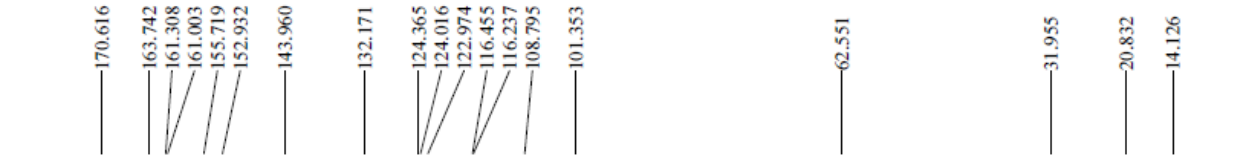
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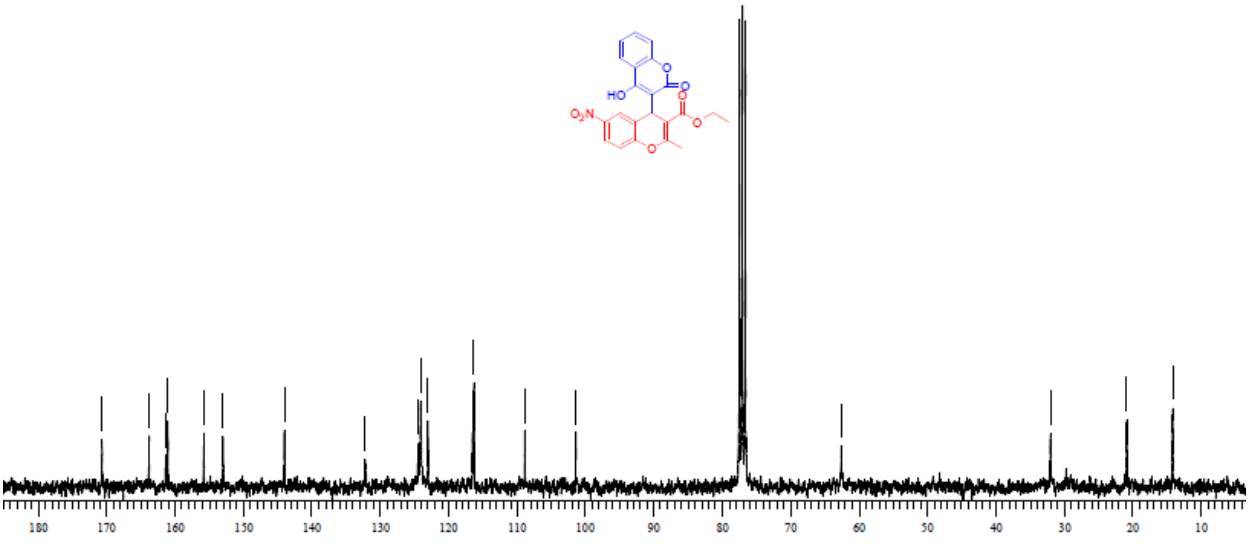
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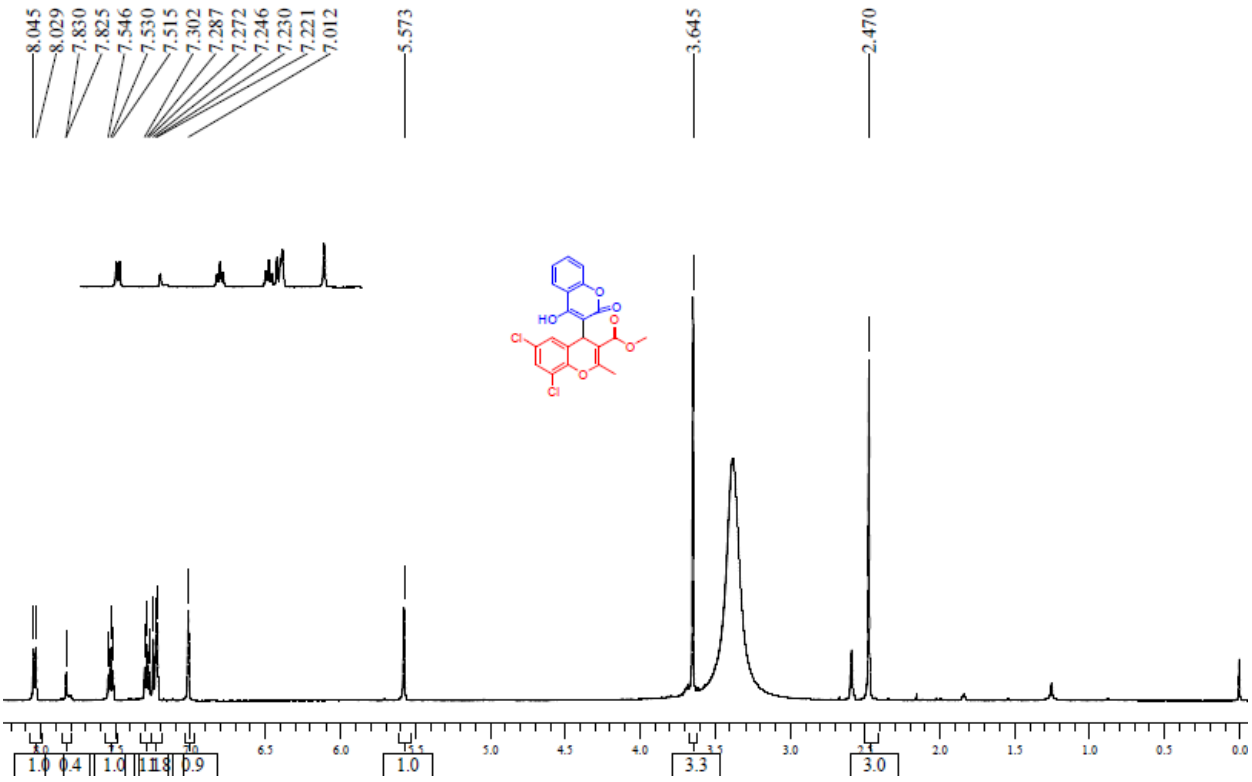
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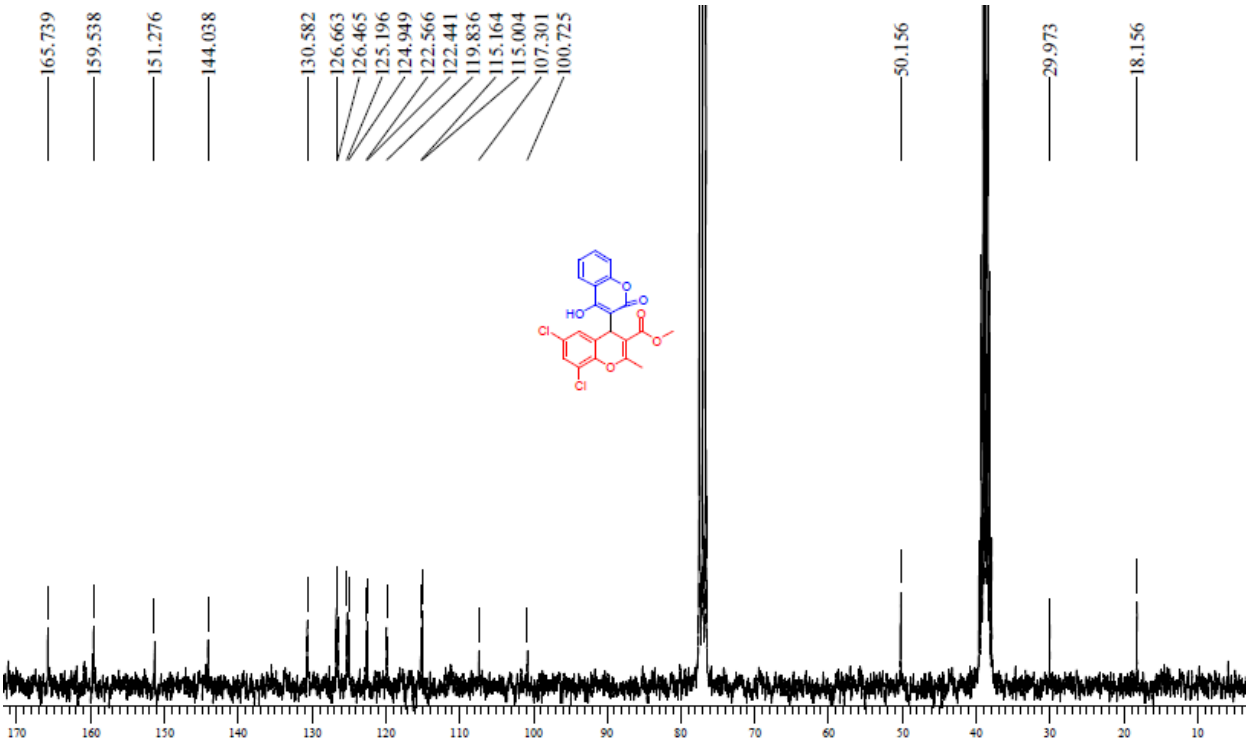
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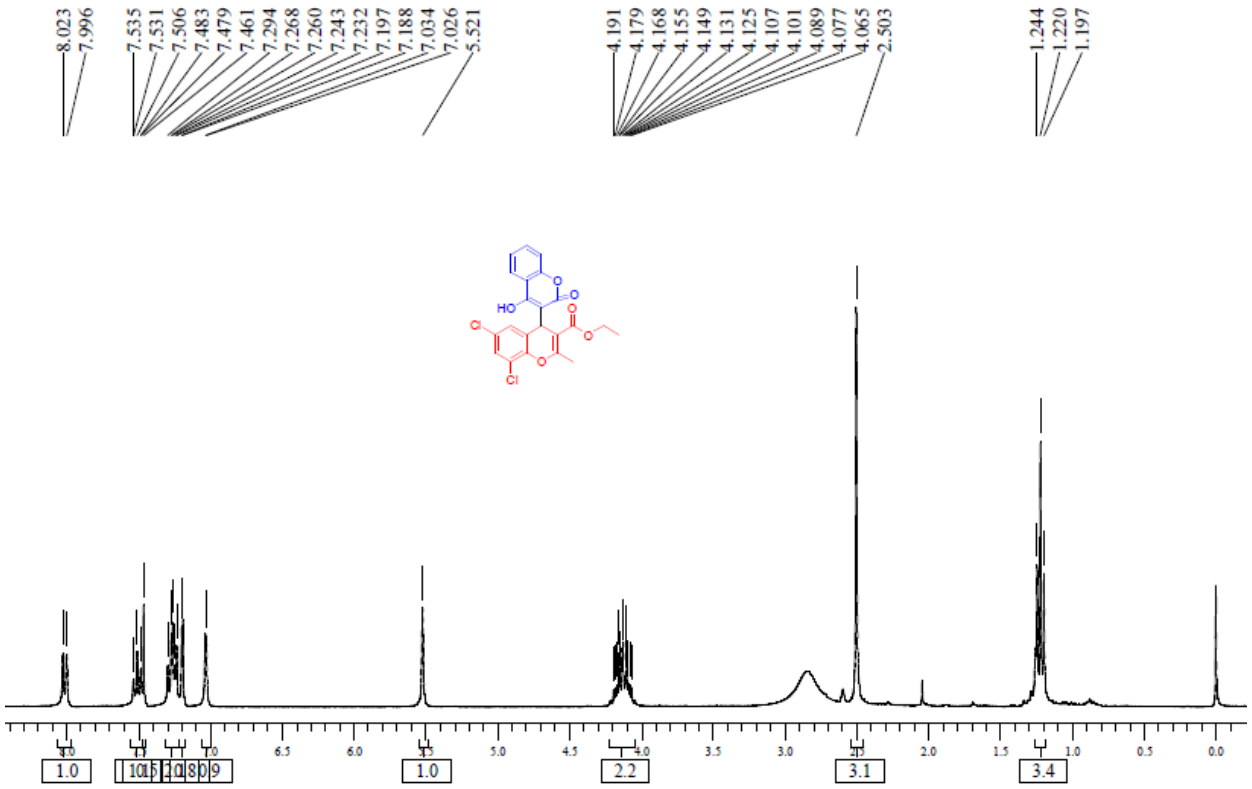
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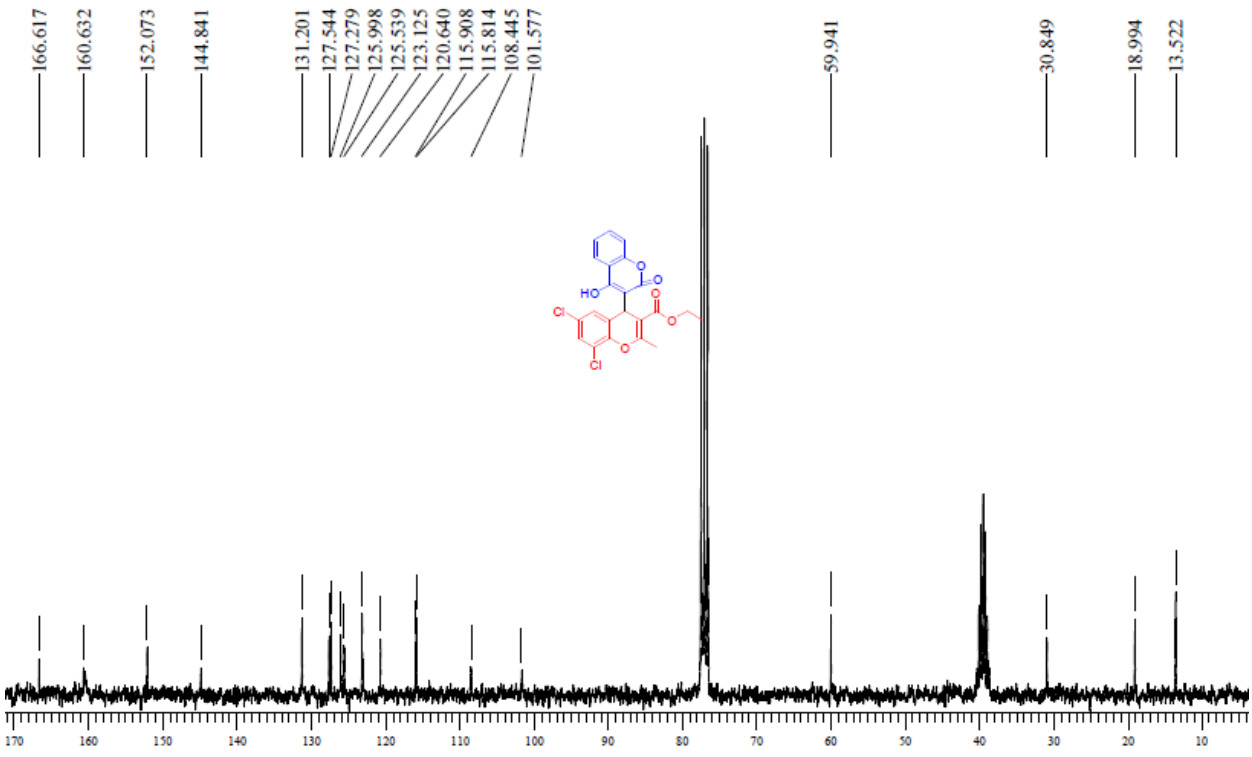
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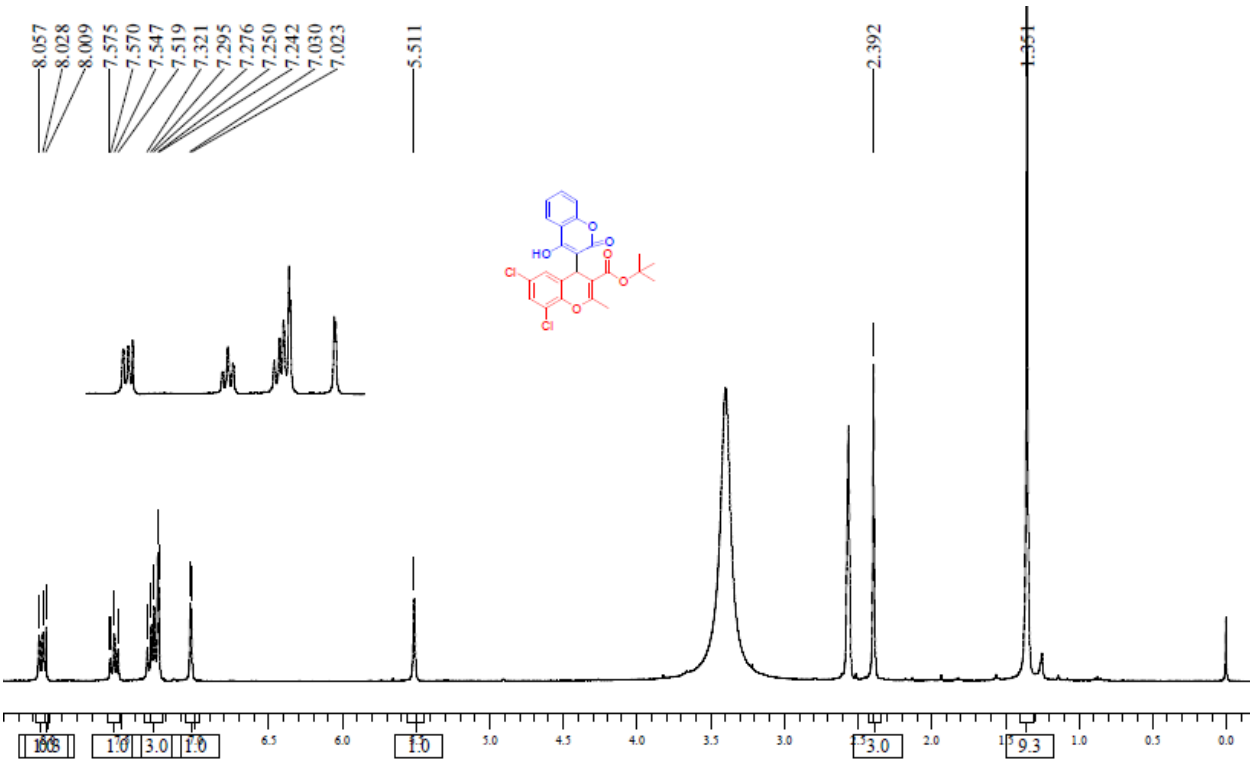
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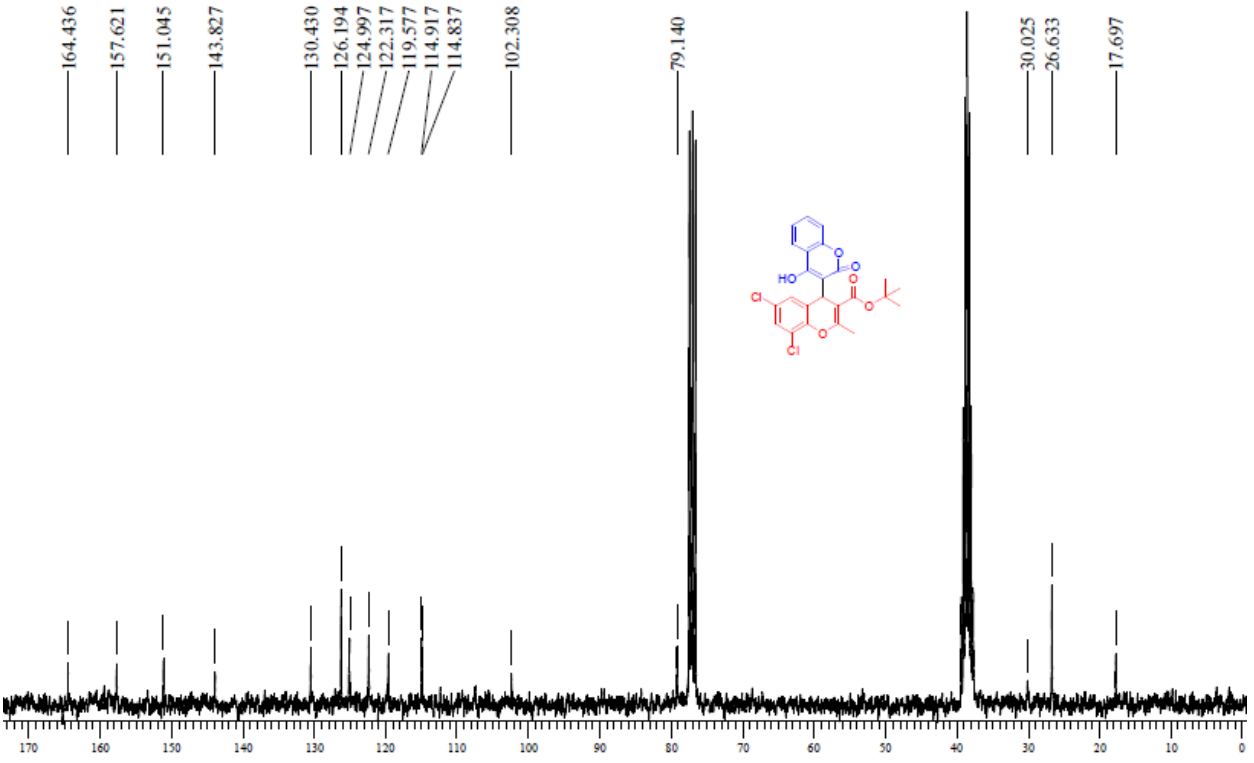
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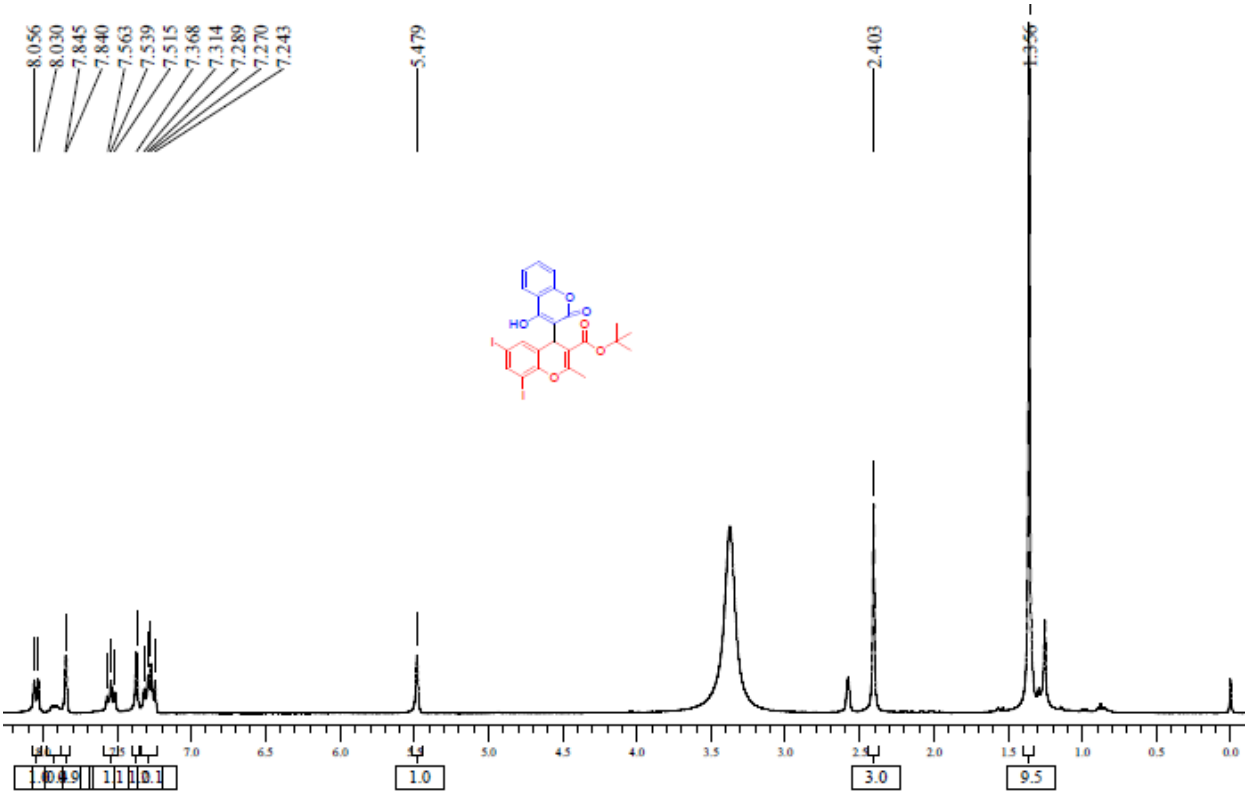
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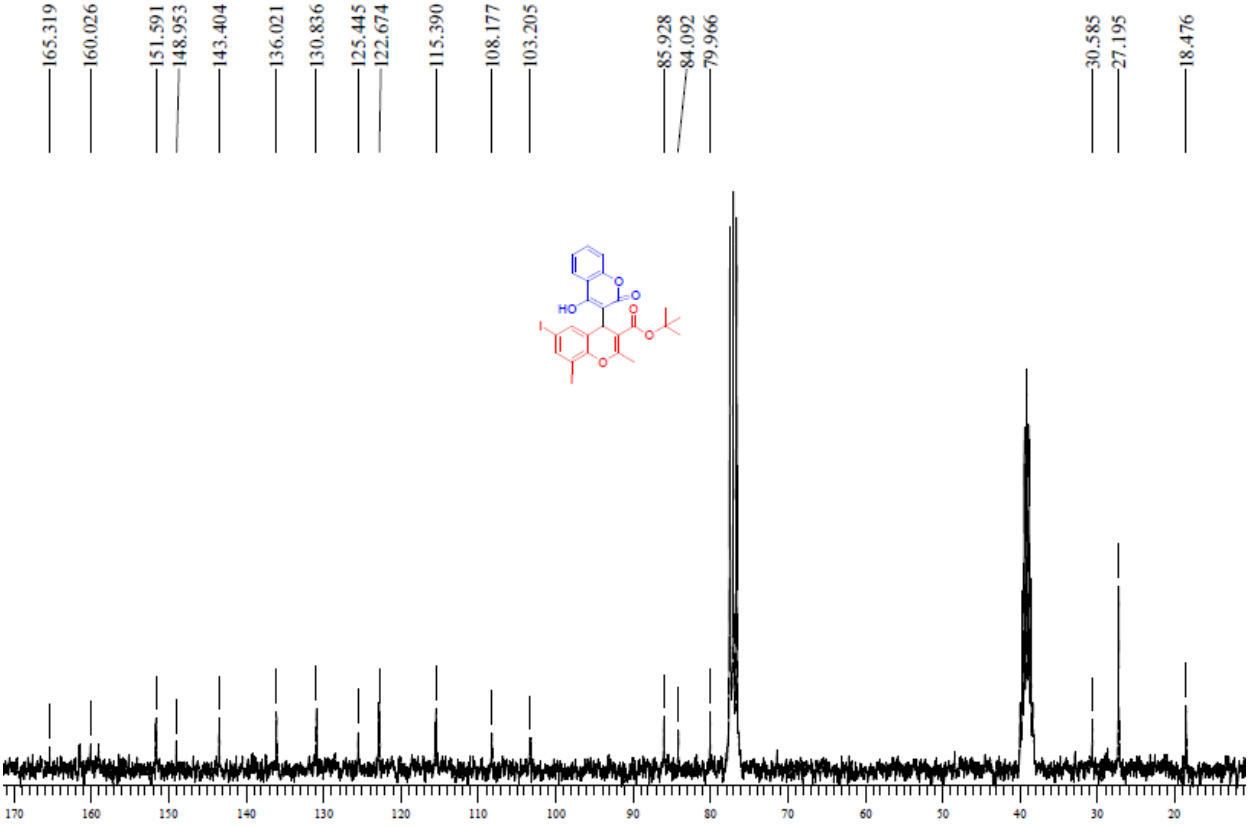
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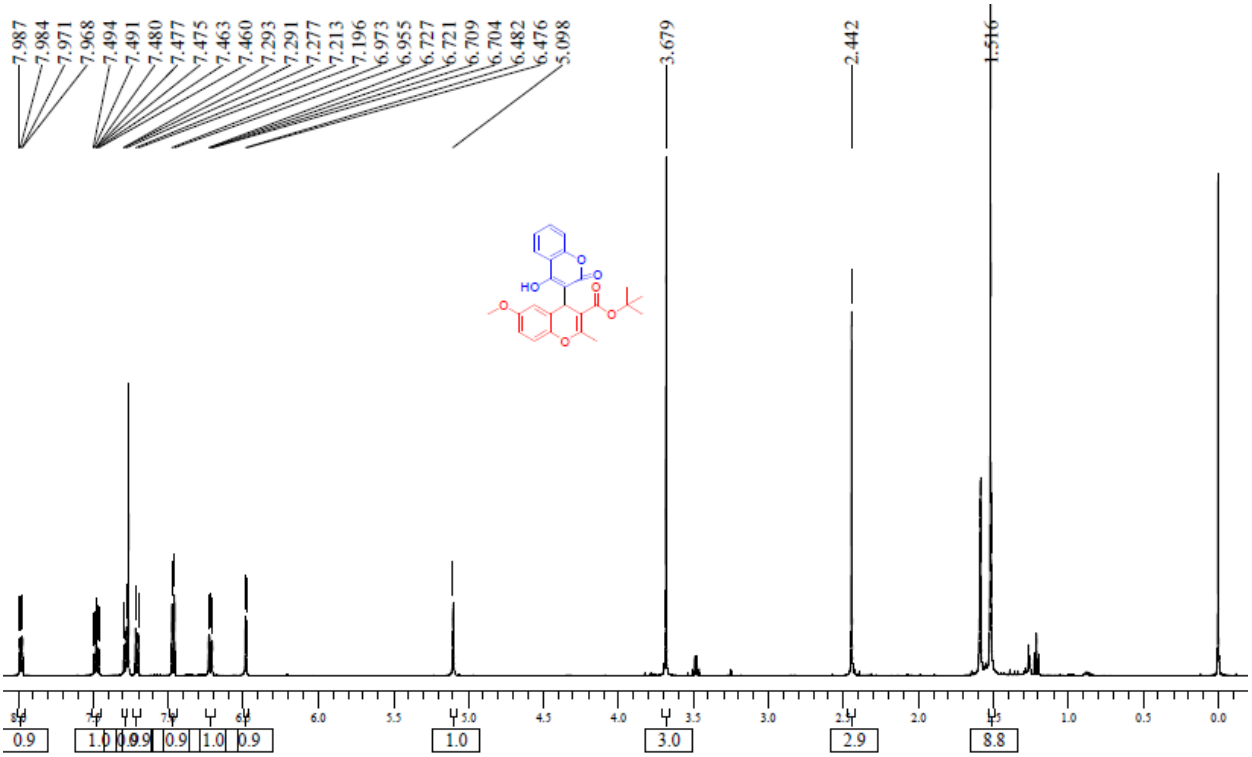
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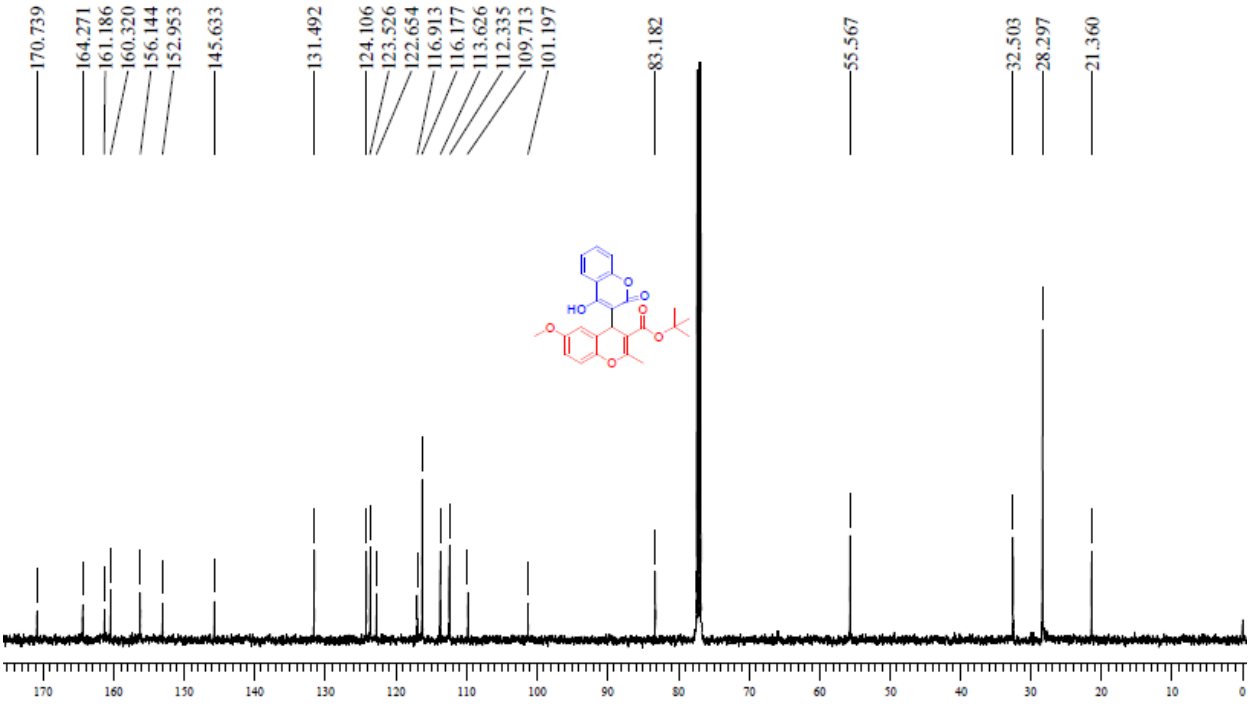
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