Facile construction of the functionalized 4*H*-chromene via tandem benzylation and cyclization

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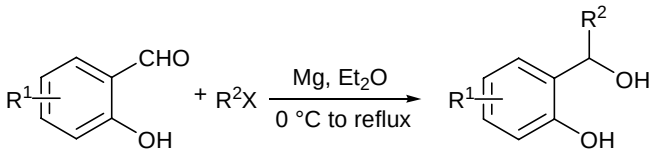
Contents:

General Remarks: ^{1}H NMR and ^{13}C NMR were recorded on a Bruker AC-300 FT (^{1}H: 300 MHz, ^{13}C: 75 MHz) using TMS as internal reference. The chemical shifts (δ) and coupling constants (J) were expressed in ppm and Hz respectively. Infrared samples were recorded on a Perkin-Elmer 2000 FTIR spectrometer. Dioxane was distilled from sodium/benzophenone. CH_3CN and CH_2Cl_2 were distilled from CaH_2 and stored over 4 Å molsieves in screw-cap flask. CH_3NO_2 was predried over 4 Å molsieves and stored in screw-cap flask. 4 Å molsieves was predried in oven at 250 ˚C for 48 h. All commercially available reagents were used as received.

1. Synthesis of substrates 1a-1h

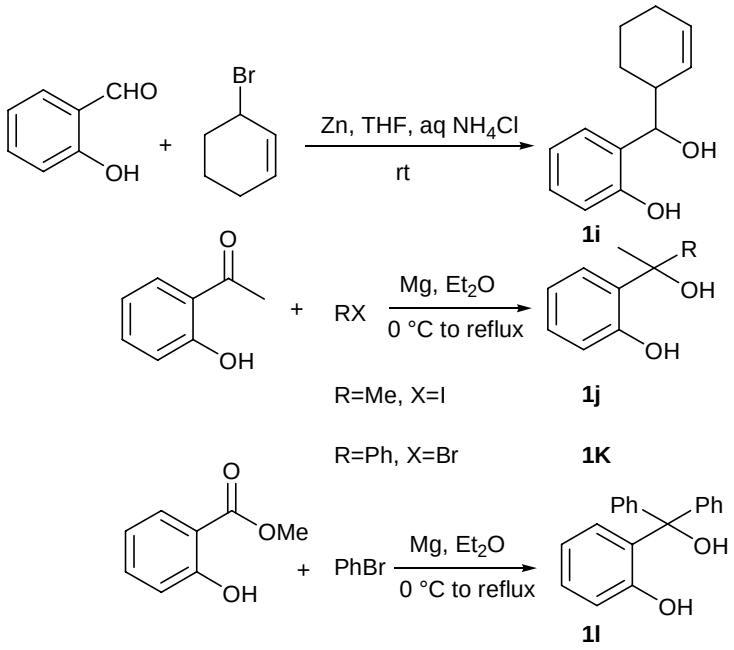
1.1 Synthesis of substrates 1a-1h (Scheme SI-1).



R^1=H, R^2=Ph, X=Br	**1a**
R^1=H, R^2=Me, X=I	**1b**
R^1=5-Cl, R^2=PH, X=Br	**1c**
R^1=5-Br, R^2=Me, X=I	**1d**
R^1=H, R^2=Ph-*p*-Me, X=Br	**1e**
R^1=5-OMe, R^2=Me, X=I	**1f**
R^1=5-OMe, R^2=Ph, X=Br	**1g**
R^1=3,5-di-*tert*-butyl, R^2=Me, X=I	**1h**

Scheme SI-1. synthesis of **1a-1h**.

1.2 Synthesis of substrates 1i-1l (Scheme SI-2)



Scheme SI-2. synthesis of **1i-1l**

General procedure for the Grignard additions:

To a solution of magnesium (80.0 mmol) and a granule of I_2 in anhydrous Et_2O (30 ml) was added dropwise a solution of bromobenzene (80.0 mmol) in anhydrous Et_2O (20 ml), controlling the speed to maintain ether boiling. After adding, the system was refluxed for 30 min. Then cooled to 0 ˚C, a solution of salicylaldehyde (20.0 mmol) in THF (20 ml) was added dropwise to the mixture in 15 min, and then the system was refluxed for a subsequent 30 min. After fefluxing, saturated NH_4Cl was added dropwise to the system at 0 ˚C, then the resulting solution was extracted with Et_2O (50 ml× 3).The combined organic extracts were dried with anhydrous sodium sulphate and concentrated in vacuo. The residue was chromatographed on silica gel eluting with petroleum ether/EtOAc (6:1) to give **1a** 2-(hydroxy(phenyl)methyl)phenol as a white solid (3.64 g, 91%).

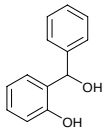
1i was synthesized according to the literature[1]

1. Einhorn,C.; Luche, J.-L. *J. Organoment. Chem.* **1987**, *322*, 177-183.

2. Characterization data of all substances

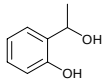
2.1. Characterization data for substrates 1a-1l

2-(hydroxy(phenyl)methyl)phenol (1a)



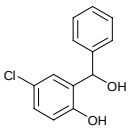
The title compound was a white solid, mp: 84-86 ˚C. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = (s, 1 H), 7.32-7.29 (m, 5 H), 7.14-7.12 (m, 1 H), 6.85-6.76 (m, 3 H), 5.91 (s, 1 H), 3.35 (d, J = 2.7 Hz, 1 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 154.9, 142.0, 129.1, 128.6, 128.3, 127.9, 127.3, 126.8, 120.1, 116.9, 76.1; IR (KBr, cm^{-1}): ν = 3339, 1588, 1491, 1456, 1388, 1238, 1008, 754, 698; HRMS calc. $C_{13}H_{12}O_2$ (M^+): 200.0837. Found: 200.0833.

2-(1-hydroxyethyl)phenol (1b)



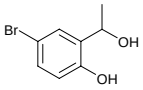
The title compound was a colorless oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.03 (s, 1 H), 7.16-7.13 (m, 1 H), 6.99-6.97 (m, 1 H), 6.87-6.81 (m, 2 H), 5.05 (q, J = 6.6 Hz, 1 H), 2.92 (s, 1 H), 1.57 (d, J = 6.6 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 155.5, 129.0, 128.6, 126.6, 120.0, 117.2, 71.6, 23.5; IR (liquid film, cm^{-1}): ν = 3344, 1586, 1489, 1455, 1232, 1066, 752; HRMS calc. $C_8H_{10}O_2$ (M^+): 138.0681. Found: 138.0675.

4-chloro-2-(hydroxy(phenyl)methyl)phenol (1c)



The title compound was a colorless oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.00 (br, 1 H), 7.40-7.30 (m, 5 H), 7.11-7.08 (m, 1 H), 6.81-6.75 (m, 2 H), 5.88 (s, 1 H), 3.25 (br, 1 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 153.2, 141.3, 128.9, 128.3, 127.7, 127.4, 127.0, 126.3, 124.3, 117.7, 74.7; IR (liquid film, cm^{-1}): ν = 3319, 1602, 1485, 1420, 1265, 1116, 738, 700; HRMS calc. $C_{13}H_{11}ClO_2$ (M^+): 234.0448. Found: 234.0444.

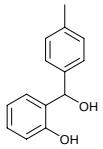
4-bromo-2-(1-hydroxyethyl)phenol (1d)



The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.07 (br, 1 H), 7.17-7.13 (m, 1 H), 7.01 (d, J = 2.4 Hz, 1 H), 6.64 (d, J = 8.7 Hz, 1 H), 4.91 (q, J = 6.6 Hz, 1 H), 3.20 (br, 1 H), 1.46 (d, J = 6.6 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 154.6, 131.6, 130.5, 129.2, 119.0, 111.8, 71.0, 23.4; IR (liquid film, cm^{-1}): ν = 3338, 1579, 1483, 1420, 1241,

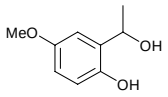
1071, 815, 624; HRMS calc. $C_8H_9BrO_2$ (M^+): 215.9786. Found: 215.9781.

2-(hydroxy(p-tolyl)methyl)phenol (1e)



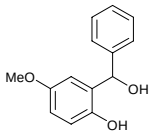
The title compound was a light yellow solid, mp: 94-96 ˚C. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.01 (s, 1 H), 7.24 (m, 5 H), 6.86-6.80 (m, 3 H), 5.90 (s, 1 H), 3.32 (br, 1 H), 2.32 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 155.2, 139.3, 137.7, 129.3, 128.3, 127.4, 126.8, 125.5, 120.0, 117.0, 76.3, 21.1; IR (KBr, cm^{-1}): ν = 3341, 1588, 1488, 1456, 1240, 1009, 755; HRMS calc. $C_{14}H_{14}O_2$ (M^+): 214.0994. Found: 214.0997.

2-(1-hydroxyethyl)-4-methoxyphenol (1f)



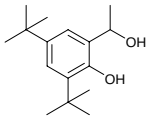
The title compound was a yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.86 (s, 1 H), 6.74-6.64 (m, 2 H), 6.53 (d, J = 2.7 Hz, 1 H), 4.93 (q, J = 6.6 Hz, 1 H), 3.84 (br, 1 H), 3.70 (s, 3 H), 1.50 (d, J = 6.6 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 153.0, 148.7, 130.2, 117.2, 114.1, 112.4, 70.2, 55.9, 23.3; IR (liquid film, cm^{-1}): ν = 3367, 1618, 1503, 1433, 1204, 1038, 811; HRMS calc. $C_9H_{12}O_3$ (M^+): 168.0786. Found: 168.0789.

2-(hydroxy(phenyl)methyl)-4-methoxyphenol (1g)



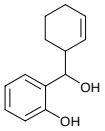
The title compound was a brown yellow solid, mp: 100-102 ˚C. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.49 (s, 1 H), 7.36-7.32 (m, 5 H), 6.80-6.73 (m, 2 H), 6.43 (s, 1 H), 5.90 (s, 1 H), 3.66 (s, 3 H), 3.27 (s, 1 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 153.0, 149.1, 141.9, 128.7, 128.1, 126.8, 117.7, 114.1, 76.2, 55.8; IR (KBr, cm^{-1}): ν = 3368, 1600, 1498, 1451, 1239, 1034, 756, 699; HRMS calc. $C_{14}H_{14}O_3$ (M^+): 230.0943. Found: 230.0938.

2,4-di-tert-butyl-6-(1-hydroxyethyl)phenol (1h)



The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.14 (s, 1 H), 6.85 (d, J = 1.8 Hz, 1 H), 5.05 (q, J = 6.6 Hz, 1 H), 2.33 (s, 1 H), 1.63 (d, J = 6.6 Hz, 3 H), 1.43 (s, 9 H), 1.29 (s, 9 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 152.5, 141.4, 136.9, 127.6, 123.5, 121.2, 73.0, 35.1, 34.3, 31.7, 29.8, 23.1; IR (liquid film, cm^{-1}): ν = 3368, 2959, 1603, 1481, 1362, 1227, 1070, 734; HRMS calc. $C_{16}H_{26}O_2$ (M^+): 250.1933. Found: 250.1939.

2-(cyclohex-2-enyl(hydroxy)methyl)phenol (1i)

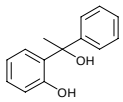


The title compound was a white solid, mp: 56-58 ˚C. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.17 (s, 1 H), 7.19-7.14 (m, 1 H), 6.93 (d, J = 7.5 Hz, 1 H), 6.87-6.80 (m, 2 H), 5.93-5.89 (m, 1 H), 5.41-5.38 (m, 1 H), 4.79 (d, J = 6.0 Hz, 1 H), 2.63-2.60 (m, 1 H), 2.57 (s, 1 H), 2.10-1.92 (m, 2 H), 1.81-1.77 (m, 2 H), 1.67-1.45 (m, 2 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 156.0, 131.5, 128.7, 128.0, 127.5, 125.2, 119.5, 117.2, 79.4, 42.4, 25.2, 23.6, 21.1; IR (KBr, cm^{-1}): ν = 3367, 1587, 1490, 1456, 1241, 1008, 908, 733; HRMS calc. $C_{13}H_{16}O_2$ (M^+): 204.1150. Found: 204.1157.

2-(2-hydroxypropan-2-yl)phenol (1j)

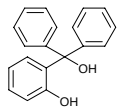
The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 9.12 (s, 1 H), 7.20-7.11 (m, 1 H), 7.08 (d, J = 7.5 Hz, 1 H), 6.90-6.78 (m, 2 H), 3.09 (s, 1 H), 1.65 (s, 6 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 155.2, 131.5, 128.9, 125.6, 120.0, 117.4, 75.8, 30.3; IR (liquid film, cm^{-1}): ν = 3309, 1583, 1491, 1237, 1040, 865, 753; HRMS calc. $C_9H_{12}O_2$ (M^+): 152.0837. Found: 152.0843.

2-(1-hydroxy-1-phenylethyl)phenol (1k)



The title compound was a light yellow solid, mp: 108-110 ˚C. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.44 (s. 1 H), 7.36-7.13 (m, 6 H), 6.98 (d, J = 7.2 Hz, 1 H), 6.82 (d, J = 7.5 Hz, 2 H), 3.18 (s, 1 H), 1.94 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 155.5, 129.6, 129.2, 128.6, 128.3, 127.5, 127.3, 125.6, 119.6, 117.6, 79.0, 30.7; IR (KBr, cm^{-1}): ν = 3421, 1605, 1458, 1377, 1098, 753, 700; HRMS calc. $C_{14}H_{14}O_2$ (M^+): 214.0994. Found: 214.0990.

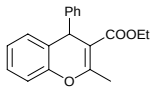
2-(hydroxydiphenylmethyl)phenol (1l)



The title compound was a white solid, 122-124 ˚C. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 8.10 (s, 1 H), 7.37-7.32 (m, 8 H), 7.23-7.18 (m, 6 H), 3.70 (s, 1 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 155.8, 145.1, 130.6, 130.1, 129.6, 128.6, 128.2, 125.6, 119.4, 117.6, 115.5, 84.4; IR (KBr, cm^{-1}): ν = 3187, 1585, 1445, 1232, 998, 753, 698, 637; HRMS calc. $C_{19}H_{16}O_2$ (M^+): 276.1150. Found: 276.1144.

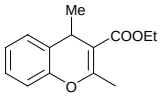
2.2. Characterization data for all 4*H*-Chromene products 3a-3s

Ethyl 2-methyl-4-phenyl-4H-chromene-3-carboxylate (3a)



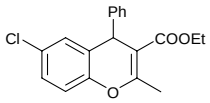
The title compound was a colorless oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.13-7.11 (m, 4 H), 7.04-6.99 (m, 2 H), 6.94-6.85 (m, 3 H), 4.93 (s, 1 H), 4.00-3.95 (m, 2 H), 2.39 (s, 3 H), 1.06 (t, J = 7.2 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 167.2, 160.1, 149.6, 146.8, 129.4, 128.5, 128.0, 127.6, 126.6, 125.0, 124.6, 116.3, 106.3, 60.2, 41.7, 19.6, 14.2; IR (liquid film, cm^{-1}): ν = 2979, 1711, 1643, 1585, 1488, 1456, 1380, 1218, 1106, 1064, 985, 754, 698, 621; HRMS calc. $C_{19}H_{18}O_3$ (M^+): 294.1256. Found: 294.1263.

Ethyl 2,4-dimethyl-4H-chromene-3-carboxylate (3b)



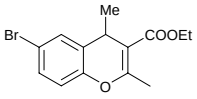
The title compound was a colorless oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.18-7.13 (m, 2 H), 7.09-7.06 (m, 1 H), 6.96-6.93 (m, 1 H), 4.29-4.20 (m, 2 H), 3.88 (q, J = 6.9 Hz, 1 H), 2.40 (s, 3 H), 1.36-1.27 (m, 6 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 167.7, 160.6, 150.2, 128.2, 127.3, 124.4, 116.0, 107.5, 60.2, 30.5, 26.0, 19.7, 14.5; IR (liquid film, cm^{-1}): ν = 2964, 1712, 1642, 1585, 1489, 1459, 1382, 1348, 1287, 1112, 1068, 986, 943, 754, 688, 625; HRMS calc. $C_{14}H_{16}O_3$ (M^+): 232.1099. Found: 232.1089.

Ethyl 6-chloro-2-methyl-4-phenyl-4H-chromene-3-carboxylate (3c)



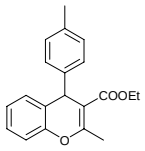
The title compound was a colorless oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.24-7.15 (m, 5 H), 7.06 (d, J = 2.1 Hz, 1 H), 7.01 (d, J = 2.1 Hz, 1 H), 6.96-6.93 (m, 1 H), 4.96 (s, 1 H), 4.10-4.05 (m, 2 H), 2.48 (s, 3 H), 1.15 (t, J = 6.9 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 166.9, 160.0, 148.1, 146.1, 129.3, 129.0, 128.7, 127.9, 127.8, 126.9, 126.6, 117.8, 106.0, 60.4, 41.6, 19.5, 14.2; IR (liquid film, cm^{-1}): ν =2980, 1713, 1644, 1581, 1482, 1412, 1380, 1323, 1277, 1115, 1066, 986, 750, 698, 636; HRMS calc. $C_{19}H_{17}ClO_3$ (M^+): 328.0866. Found: 328.0879.

Ethyl 6-bromo-2,4-dimethyl-4H-chromene-3-carboxylate (3d)



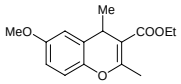
The title compound was a colorless oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.26-7.22 (m, 2 H), 6.82 (d, J = 8.4 Hz, 1 H), 4.26-4.22 (m, 2 H), 3.84 (q, J = 6.6 Hz, 1 H), 2.38 (s, 3 H), 1.35-1.26 (m, 6 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 167.2, 160.3, 149.2, 130.8, 130.2, 129.4, 117.8, 116.5, 107.3, 60.3, 30.4, 25.9, 19.5, 14.4; IR (liquid film, cm^{-1}): ν = 2977, 1713, 1641, 1578, 1479, 1409, 1380, 1338, 1277, 1122, 1072, 987, 778, 699, 636; HRMS calc. $C_{14}H_{15}BrO_3$ (M^+): 310.0205. Found: 310.0201.

Ethyl 2-methyl-4-p-tolyl-4H-chromene-3-carboxylate (3e)



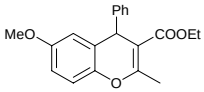
The title compound was a colorless oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.13-7.00 (m, 8 H), 4.99 (s, 1 H), 4.10 (q, J = 7.2 Hz. 2 H), 2.49 (s, 3 H), 2.27 (s, 3 H), 1.20 (t, J = 7.2 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 167.3, 160.0, 149.6, 144.0, 136.0, 129.2, 127.8, 127.5, 125.2, 124.6, 116.3, 106.5, 60.2, 41.3, 21.1, 19.6, 14.3; IR (liquid film, cm^{-1}): v = 2979, 1712, 1643, 1585, 1487, 1457, 1380, 1288, 1105, 1065, 985, 754, 692, 641; HRMS calc. $C_{20}H_{20}O_3$ (M^+): 308.1412. Found: 308.1419.

Ethyl 6-methoxy-2,4-dimethyl-4H-chromene-3-carboxylate (3f)



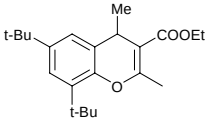
The title compound was a colorless oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 6.87 (d, J = 8.7 Hz, 1 H), 6.71-6.65 (m, 2 H), 4.26-4.28 (m, 2 H), 3.76 (s, 3 H), 2.37 (s, 3 H), 1.34-1.25 (m, 6 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 167.7, 160.8, 156.4, 144.3, 128.1, 116.7, 113.0, 112.5, 106.5, 60.1, 55.7, 30.8, 25.8, 19.6, 14.4; IR (liquid film, cm^{-1}): v = 2965, 1709,1638, 1498, 1456, 1381, 1278, 1159, 1071, 1044, 989, 779, 664, 624; HRMS calc. $C_{15}H_{18}O_4$ (M^+): 262.1205. Found: 262.1211.

Ethyl 6-methoxy-2-methyl-4-phenyl-4H-chromene-3-carboxylate (3g)



The title compound was a colorless oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.22-7.20 (m, 4 H), 7.18-7.05 (m, 1 H), 6.96-6.90 (m, 1 H), 6.70-6.60 (m, 1 H), 6.53 (d, J = 2.7 Hz, 1 H), 4.98 (s, 1 H), 4.08-4.05 (m, 2 H), 3.64 (s, 3 H), 2.47 (s, 3 H), 1.14 (t, J = 7.2 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 167.4, 160.5, 156.5, 146.8, 143.9, 128.6, 128.0, 126.6, 125.8, 117.2, 113.8, 113.5, 105.5, 60.2, 55.7, 42.2, 19.7, 14.3; IR (liquid film, cm^{-1}): v = 2980, 1710, 1638, 1496, 1454, 1380, 1328, 1279, 1152, 1068, 988, 734, 702, 645; HRMS calc. $C_{20}H_{20}O_4$ (M^+): 324.1362. Found: 324.1353.

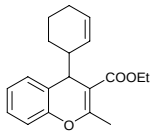
Ethyl 6,8-di-tert-butyl-2,4-dimethyl-4H-chromene-3-carboxylate (3h)



The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.19 (d, J = 2.4 Hz, 1 H), 7.02 (d, J = 2.1 Hz, 1 H), 4.26-4.22 (m, 2 H), 3.85 (q, J = 6.6 Hz, 1 H), 2.46 (s, 3 H), 1.43 (s, 9 H), 1.35-1.23 (m, 15 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 167.6, 160.7, 146.9, 146.4, 136.2, 127.2, 122.8, 121.8, 107.7, 60.0, 35.0, 34.7, 31.7, 31.4, 30.3, 25.8, 19.6, 14.5; IR (liquid film, cm^{-1}): v = 2963, 1713, 1644, 1597, 1478, 1380, 1345, 1216, 1174, 1075, 956, 909,

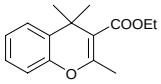
779, 734, 650,; HRMS calc. $C_{22}H_{32}O_3$ (M^+): 344.2351. Found: 344.2342.

Ethyl 4-(cyclohex-2-enyl)-2-methyl-4H-chromene-3-carboxylate (3i)



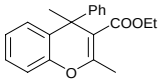
The title compound was a yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.20-7.03 (m, 3 H), 6.99-6.96 (m, 1 H), 5.68-5.60 (m, 1 H), 5.52-5.48 (m, 1 H), 4.22 (q, J = 6.9 Hz, 2 H), 3.93 (d, J = 4.2 Hz, 1 H), 2.44-2.33 (m, 4 H), 1.86-1.59 (m, 4 H), 1.43-1.26 (m, 5 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 168.0, 161.3, 151.9, 129.0, 128.6, 127.3, 124.4, 123.9, 115.8, 105.6, 60.2, 44.3, 40.3, 25.8, 25.3, 22.1, 19.5, 14.4; IR (liquid film, cm^{-1}): ν = 2930, 1710, 1640, 1584, 1487, 1458, 1381, 1289, 1216, 1106, 1061, 983, 756, 689, 630; HRMS calc. $C_{19}H_{22}O_3$ (M^+): 298.1569. Found: 298.1567.

Ethyl 2,4,4-trimethyl-4H-chromene-3-carboxylate (3j)



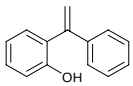
The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.36-7.29 (m, 1 H), 7.18-7.02 (m, 2 H), 6.90-6.87 (m, 1 H), 4.26 (q, J = 7.2 Hz, 2 H), 2.12 (s, 3 H), 1.55 (s, 6 H), 1.35 (t, J = 7.2 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 168.7, 152.9, 149.2, 130.6, 127.2, 126.1, 124.0, 116.0, 112.1, 60.4, 33.7, 30.8, 19.3, 14.3; IR (liquid film, cm^{-1}): ν = 2977, 1712, 1654, 1583, 1490, 1450, 1384, 1313, 1236, 1120, 1094, 1052, 979, 906, 831, 755, 646; HRMS calc. $C_{15}H_{18}O_3$ (M^+): 246.1256. Found: 246.1264.

Ethyl 2,4-dimethyl-4-phenyl-4H-chromene-3-carboxylate (3k)



The title compound was a colorless oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.37 (d, J = 7.5 Hz, 2 H), 7.28-7.23 (m, 2 H), 7.16-7.14 (m, 1 H), 7.06-7.03 (m, 1 H), 6.95-6.92 (m, 1 H), 6.88-6.83 (m, 1 H), 6.68-6.65 (m, 1 H), 3.88-3.80 (m, 2 H), 2.33 (s, 3 H), 1.94 (s, 3 H), 0.87 (t, J = 7.2 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 167.8, 156.4, 149.1, 148.6, 130.9, 129.1, 128.0, 127.2, 125.9, 124.2, 115.8, 111.9, 60.0, 42.0, 30.0, 19.7, 13.8; IR (liquid film, cm^{-1}): ν = 2980, 1704, 1639, 1488, 1453, 1288, 1220, 1116, 1068, 968, 909, 734, 700, 648; HRMS calc. $C_{20}H_{20}O_3$ (M^+): 308.1412. Found: 308.1418.

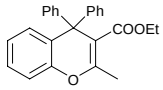
2-(1-phenylvinyl)phenol



The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.38-7.30 (m, 4 H), 7.24-7.21 (m, 2 H), 7.14-7.11 (m, 1 H), 6.95-6.89 (m, 2 H), 5.85 (d, J = 0.9 Hz, 1 H), 5.40 (d, J = 0.9 Hz, 1 H), 5.19 (s, 1 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 153.3, 145.5, 139.6, 130.6, 129.6, 128.8, 127.8, 127.2, 120.6, 116.8, 116.0; IR (liquid film, cm^{-1}): ν = 3518, 3058, 1708, 1580,

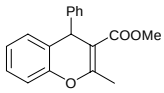
1485, 1447, 1196, 1028, 909, 855, 756, 702.

Ethyl 2-methyl-4,4-diphenyl-4H-chromene-3-carboxylate (3l)



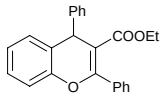
The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.28-7.13 (m, 11 H), 7.00-6.95 (m, 2 H), 6.88-6.85 (m, 1 H), 3.82 (q, J = 7.2 Hz, 2 H), 2.62 (s, 3 H), 0.87 (t, J = 7.2 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 167.7, 157.8, 150.8, 145.9, 130.7, 130.0, 129.8, 127.8, 127.6, 126.4, 123.9, 116.0, 113.0, 60.4, 52.7, 19.6, 13.8; IR (liquid film, cm^{-1}): ν = 2980, 1709, 1640, 1482, 1447, 1382, 1298, 1219, 1104, 1065, 989, 909, 731, 699, 624; HRMS calc. $C_{25}H_{22}O_3$ (M^+): 370.1569. Found: 370.1561.

Methyl 2-methyl-4-phenyl-4H-chromene-3-carboxylate (3m)



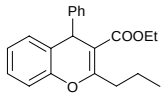
The title compound was a colorless oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.16-7.10 (m, 4 H), 7.04-6.99 (m, 2 H), 6.93-6.85 (m, 3 H), 4.91 (s, 1 H), 3.51 (s, 3 H), 2.38 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 167.7, 160.4, 149.5, 146.7, 129.3, 128.6, 127.8, 127.6, 126.6, 125.0, 124.7, 116.3, 106.2, 51.3, 41.6, 19.7; IR (liquid film, cm^{-1}): ν = 2949, 1714, 1643, 1586, 1487, 1456, 1380, 1290, 1219, 1131, 1106, 1066, 991, 946, 754, 698, 620; HRMS calc. $C_{18}H_{16}O_3$ (M^+): 280.1099. Found: 280.1107.

Ethyl 2,4-diphenyl-4H-chromene-3-carboxylate (3n)



The title compound was a colorless oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.49-7.42 (m, 2 H), 7.40-7.34 (m, 5 H), 7.28-7.23 (m, 2 H), 7.17-7.06 (m, 4 H), 7.01-6.99 (m, 1 H), 5.15 (s, 1 H), 3.86 (q, J = 7.2 Hz, 2 H), 0.82 (t, J = 7.2 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 167.2, 158.6, 150.2, 146.2, 135.6, 129.7, 129.5, 129.0, 128.8, 128.1, 128.0, 127.9, 126.9, 124.9, 124.8, 116.7, 107.9, 60.3, 42.6, 13.7; IR (liquid film, cm^{-1}): ν = 3028, 2981, 2935, 1695, 1645, 1599, 1584, 1487, 1455, 1390, 1369, 1342, 1297, 1233, 1190, 1112, 1068, 1031, 974, 910, 834, 791, 755, 697, 648, 604; HRMS calc. $C_{24}H_{20}O_3$ (M^+): 356.1412. Found: 356.1419.

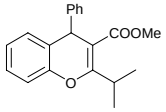
Ethyl 4-phenyl-2-propyl-4H-chromene-3-carboxylate (3o)



The title compound was a colorless oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.28-7.21 (m, 4 H), 7.20-6.94 (m, 5 H), 5.03 (s, 1 H), 4.10-4.03 (m, 2 H), 2.90-2.84 (m, 2 H), 1.76 (q, J = 7.5 Hz, 2 H), 1.15 (t, J = 6.9 Hz, 3 H), 1.02 (t, J = 7.5 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 167.2, 163.7, 149.7, 146.9, 129.4, 128.6, 128.0, 127.7, 126.6, 125.1, 124.6, 116.4, 106.4, 60.3, 41.9, 34.5,

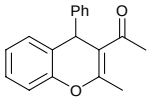
21.2, 14.3, 14.1; IR (liquid film, cm^{-1}): ν = 2963, 1711, 1638, 1538, 1488, 1456, 1367, 1284, 1216, 1106, 1045, 903, 754, 698, 632; HRMS calc. $C_{21}H_{22}O_3$ (M^+): 322.1569. Found: 322.1560.

Methyl 2-isopropyl-4-phenyl-4H-chromene-3-carboxylate (3p)



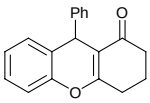
The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.23-7.20 (m, 4 H), 7.15-6.94 (m, 5 H), 5.01 (s, 1 H), 3.98 (sep, J = 6.9 Hz, 1 H), 3.61 (s, 3 H), 1.32 (d, J = 6.9 Hz, 3 H), 1.21 (d, J = 6.9 Hz, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 167.6, 167.2, 149.8, 146.7, 129.1, 128.6, 127.7, 126.6, 125.2, 124.5, 116.2, 104.8, 51.4, 42.0, 29.9, 20.1, 19.6; IR (liquid film, cm^{-1}): ν = 2967, 1713, 1635, 1585, 1488, 1457, 1342, 1286, 1218, 1063, 945, 827, 754, 699, 624; HRMS calc. $C_{20}H_{20}O_3$ (M^+): 308.1412. Found: 308.1419.

1-(2-methyl-4-phenyl-4H-chromen-3-yl)ethanone (3q)



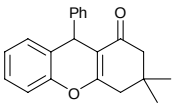
The title compound was a colorless oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.16-7.13 (m, 4 H), 7.06-6.99 (m, 3 H), 6.91-6.87 (m, 2 H), 4.92 (s, 1 H), 2.36 (s, 3 H), 2.06 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 198.9, 159.1, 149.2, 146.0, 129.1, 129.0, 127.8, 127.7, 127.0, 125.0, 124.6, 116.4, 114.4, 42.4, 30.2, 20.2; IR (liquid film, cm^{-1}): ν = 2925, 1683, 1619, 1577, 1488, 1457, 1379, 1357, 1219, 1131, 1030, 938, 882, 820, 755, 699, 655, 617; HRMS calc. $C_{18}H_{16}O_2$ (M^+): 264.1150. Found: 264.1143.

9-phenyl-3,4-dihydro-2H-xanthen-1(9H)-one (3r)



The title compound was a white solid, mp: 137-139 ˚C. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.21-6.96 (m, 9 H), 5.05 (s, 1 H), 2.67-2.58 (m, 2 H), 2.37-2.30 (m, 2 H), 2.01-1.94 (m, 2 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 196.9, 166.3, 149.7, 146.4, 130.2, 128.6, 128.1, 127.8, 126.5, 125.6, 125.2, 116.6, 114.9, , 38.0, 37.1, 28.0, 20.5; IR (KBr, cm^{-1}): ν = 2955, 1644, 1581, 1487, 1454, 1373, 1236, 1177, 1129, 995, 916, 756, 698, 647, 622; HRMS calc. $C_{19}H_{16}O_2$ (M^+): 276.1150. Found: 276.1152.

3,3-dimethyl-9-phenyl-3,4-dihydro-2H-xanthen-1(9H)-one (3s)



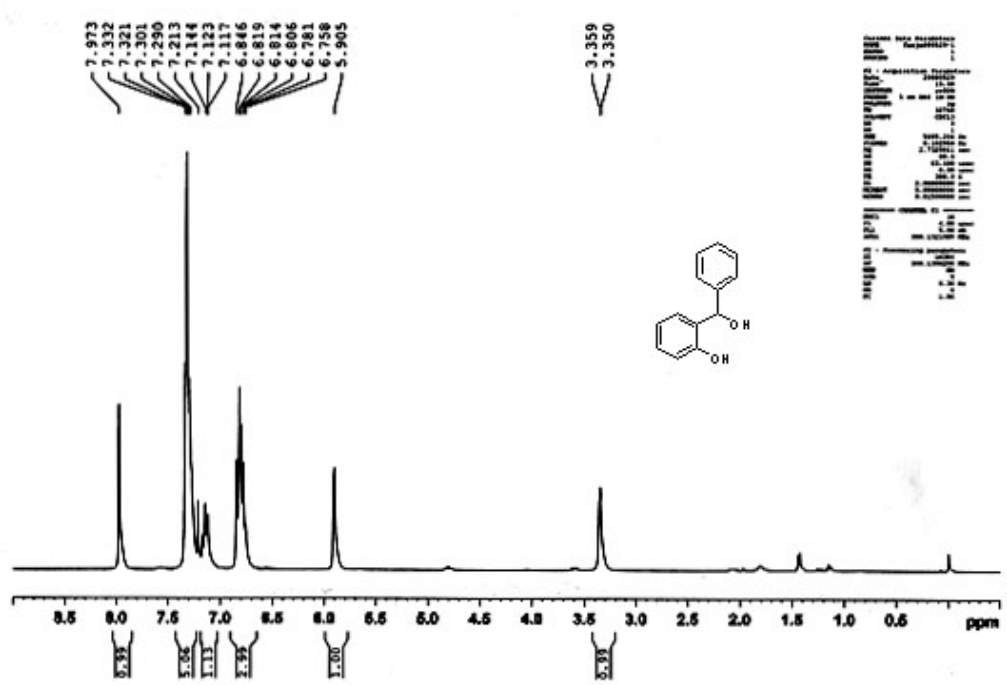
The title compound was a white solid, mp: 150-152 ˚C. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.29-7.01 (m, 9 H), 5.07 (s, 1 H), 2.58 (s, 2 H), 2.28-2.26 (m, 2 H), 1.14 (s, 3 H), 1.06 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 196.7, 164.6, 149.6, 146.3, 130.3, 128.6, 128.0, 127.8, 126.5, 125.6, 125.2, 116.7, 113.6, 51.0, 41.7, 38.1, 32.2, 29.4, 27.6; IR (KBr, cm^{-1}): ν = 2962,

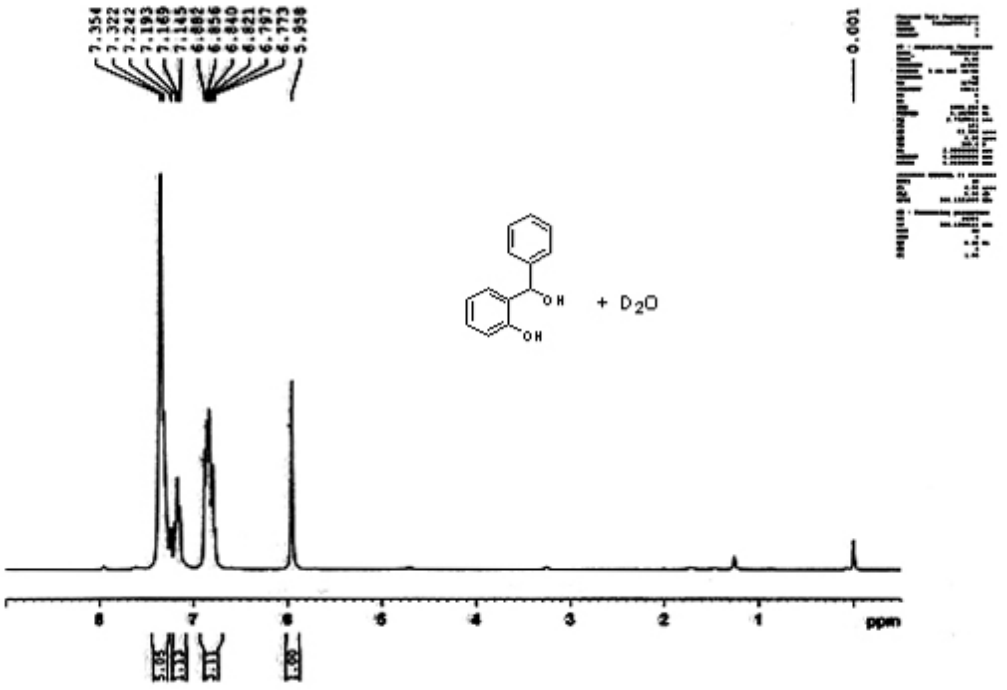
1644, 1581, 1487, 1454, 1376, 1234, 1123, 1027, 910, 733, 700, 659, 623; HRMS calc. $C_{21}H_{20}O_2$ (M^+): 304.1463. Found: 304.1459.

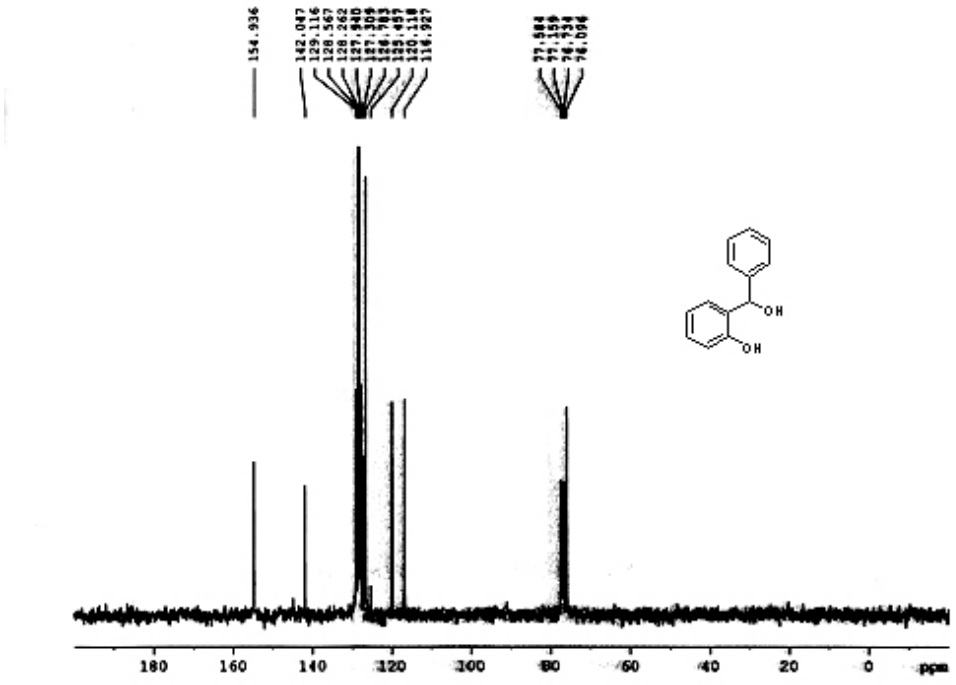
3 NMR Spectra of all compounds

3.1 ^{1}H NMR of the substrates 1a-1l

1a



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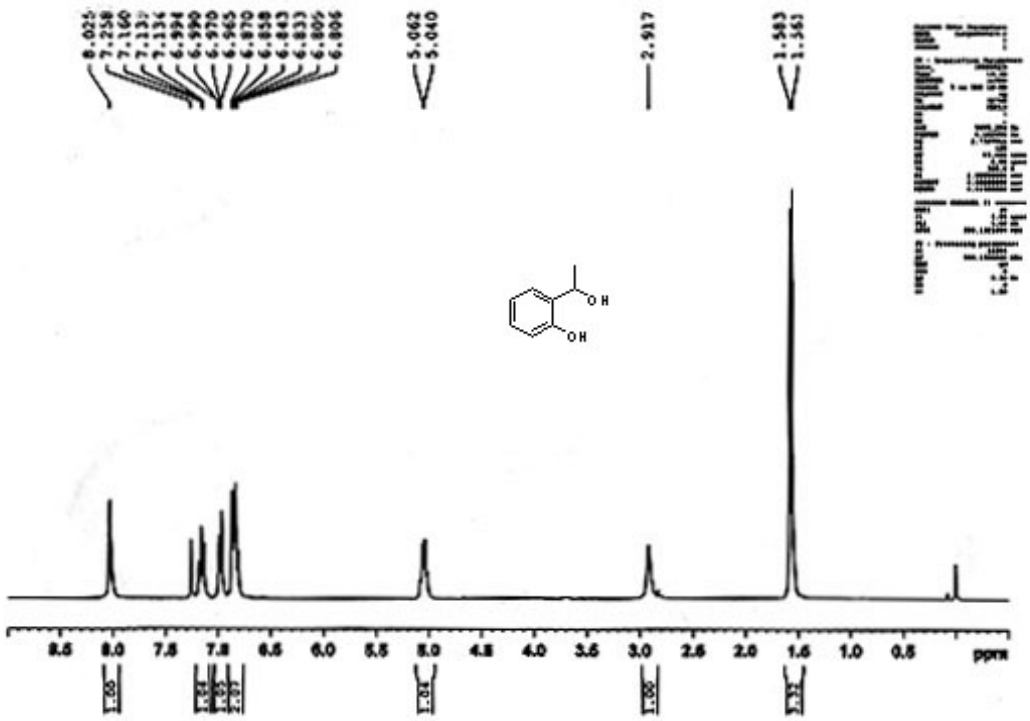
129.116
128.567
128.262
127.940
127.309
124.783
123.457
120.118
114.927
77.584
77.159
76.734
76.094
OH
OH
180
160
140
120
100
80
60
40
20
0
ppm

1b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7.160
7.133
7.134
6.994
6.990
6.970
6.965
6.870
6.858
6.843
6.833
6.806
6.806
5.062
5.040
2.917
1.583
1.561
OH
OH
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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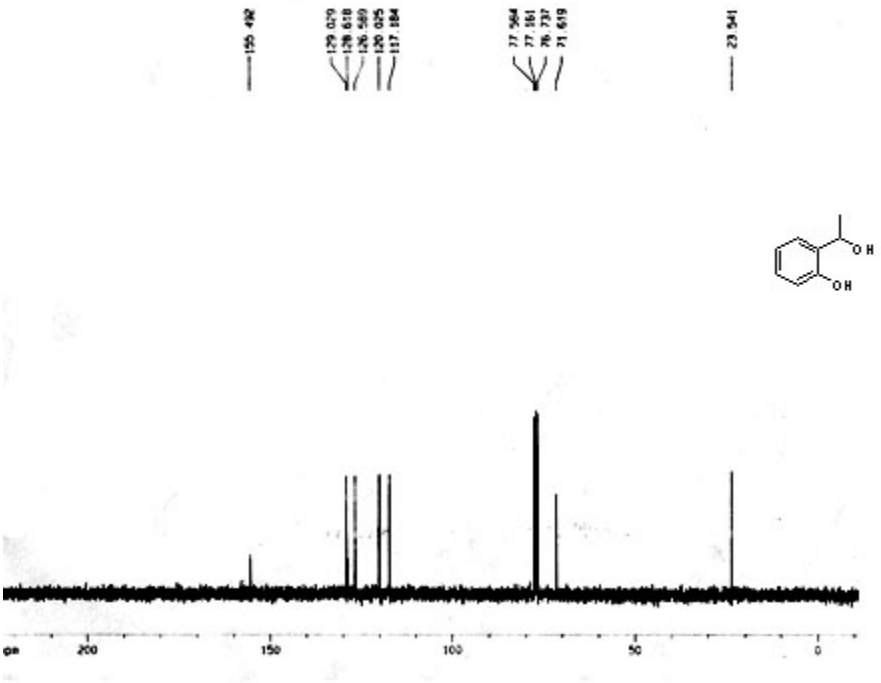
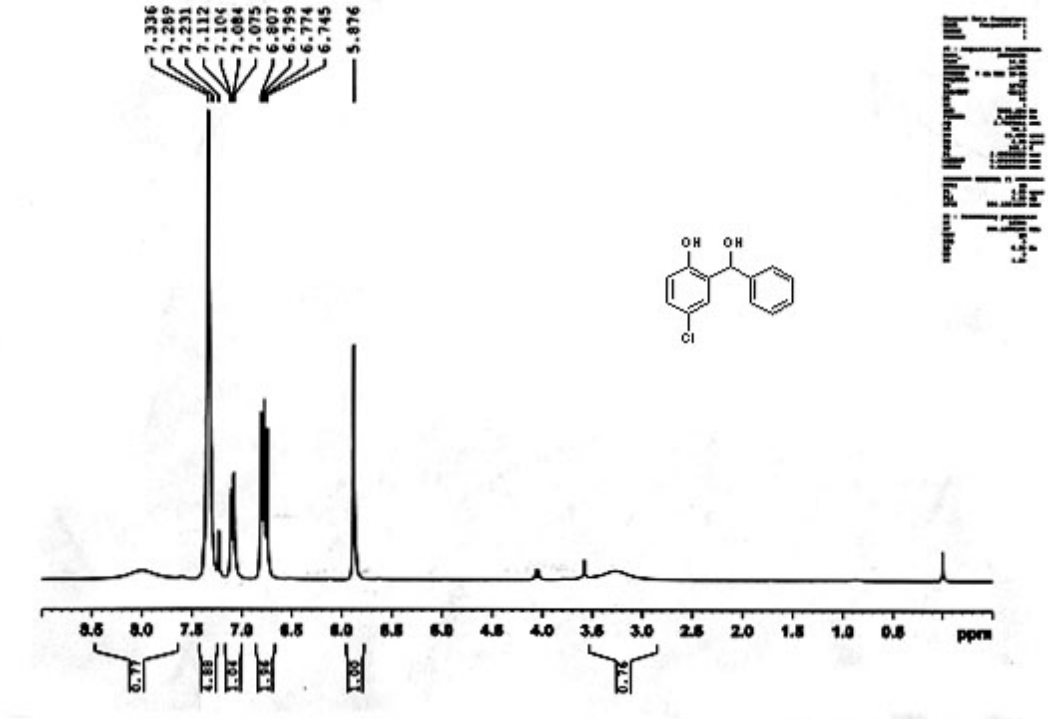
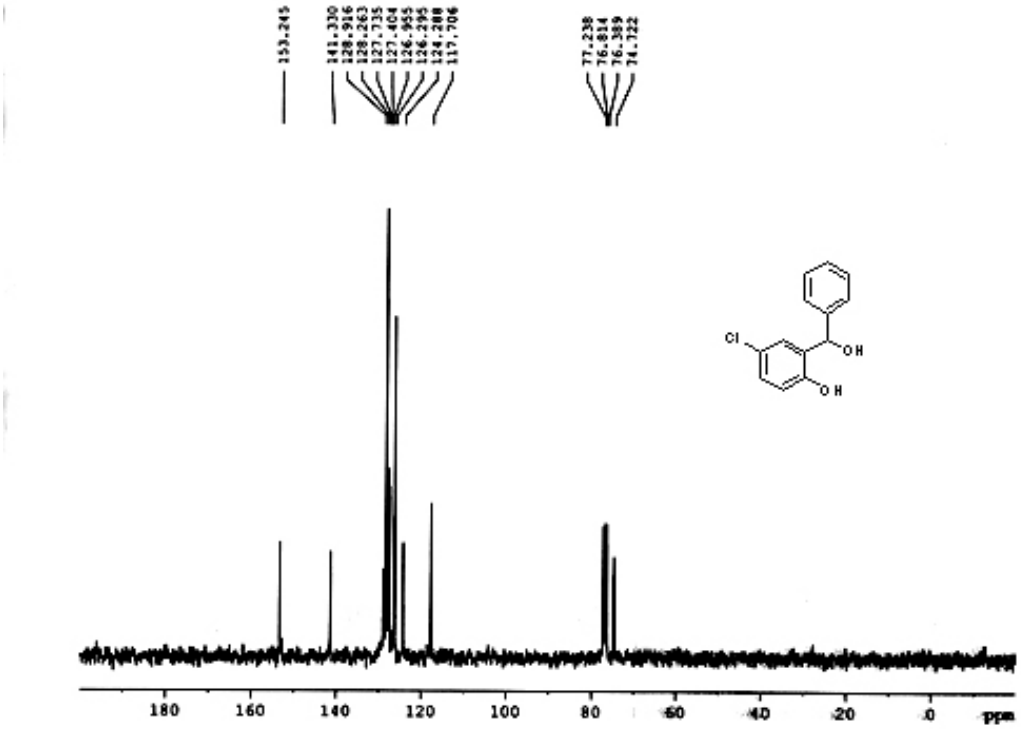
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

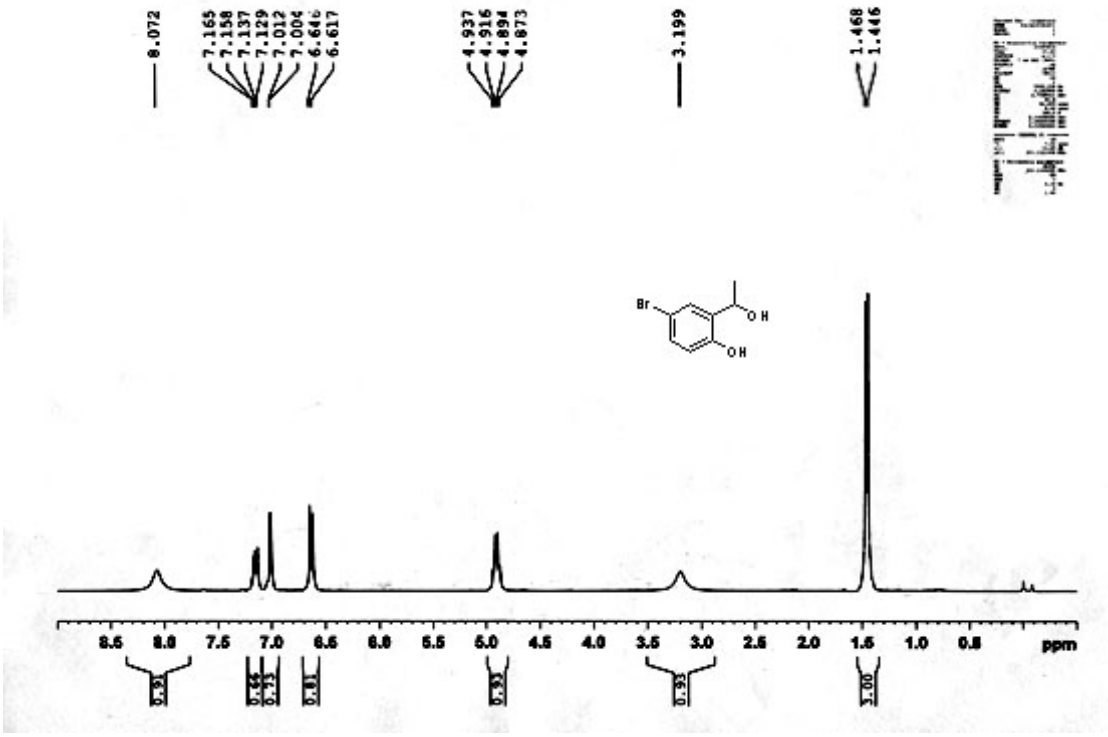

155.492
129.029
128.618
126.589
120.025
117.184
77.564
77.351
76.737
71.619
23.541
OH
OH
200
150
100
50
0

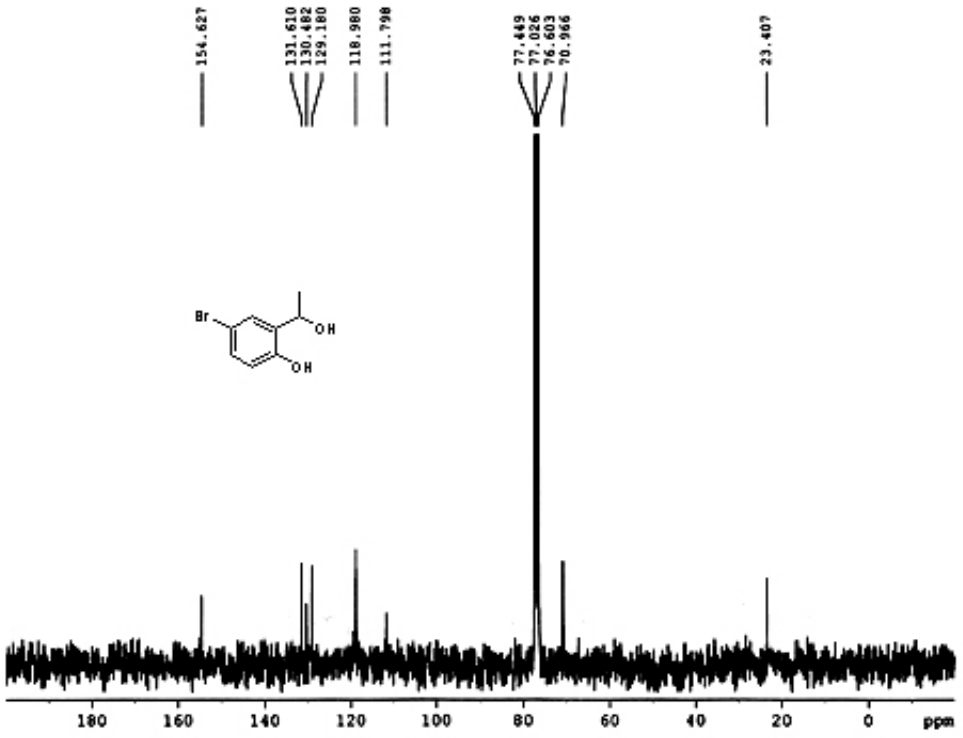
1c


7.336
7.289
7.231
7.112
7.104
7.084
7.075
6.807
6.799
6.774
6.745
5.876
OH
OH
Cl
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
ppm

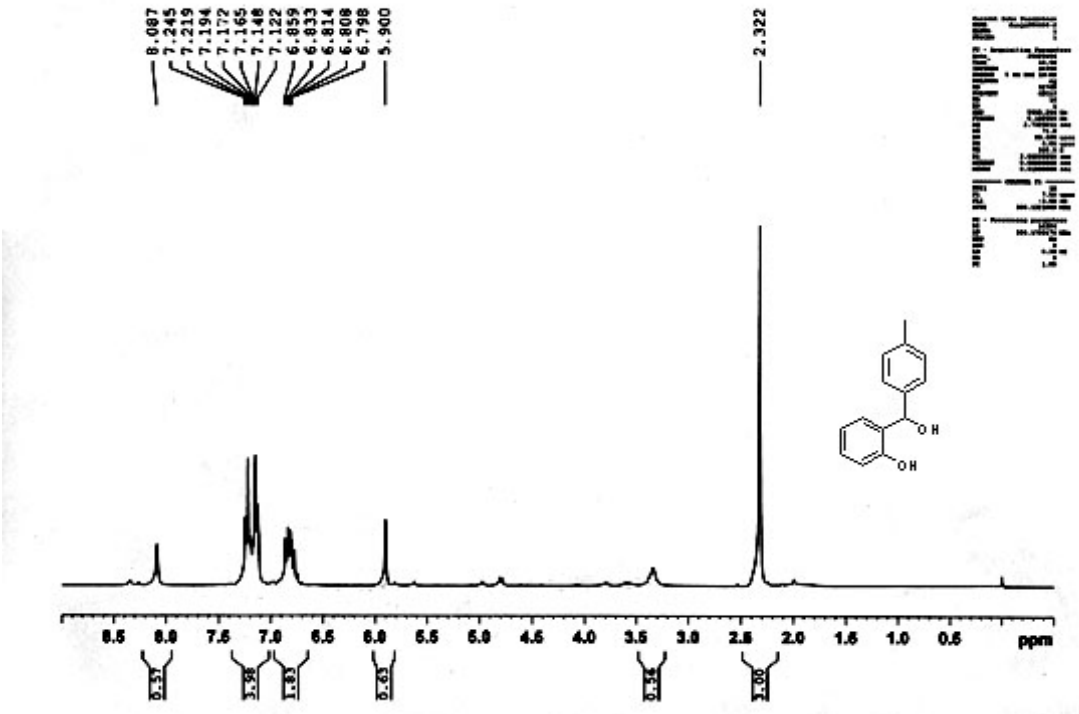

153.245
141.330
128.916
128.263
127.735
127.404
126.955
126.295
124.280
117.706
77.238
76.811
76.389
74.722
Cl
OH
OH
180
160
140
120
100
80
60
40
20
0
ppm

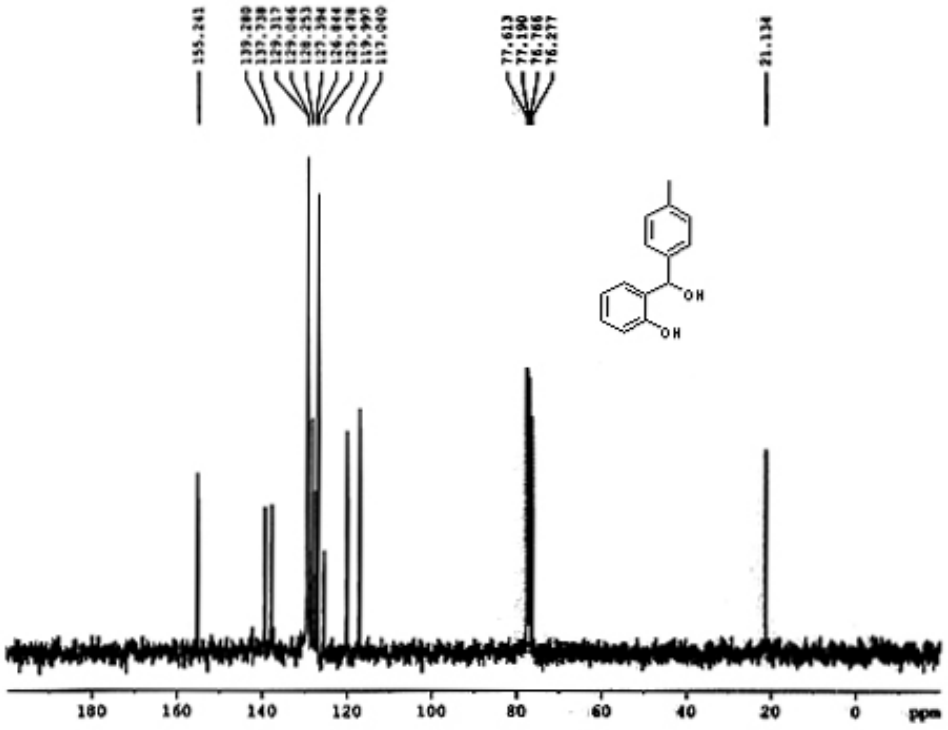
1d


8.072
7.165
7.158
7.137
7.129
7.012
7.004
6.646
6.617
4.937
4.916
4.894
4.873
3.199
1.468
1.446
Br
OH
OH
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
ppm
0.93
0.46
0.73
0.81
0.93
0.93
3.00

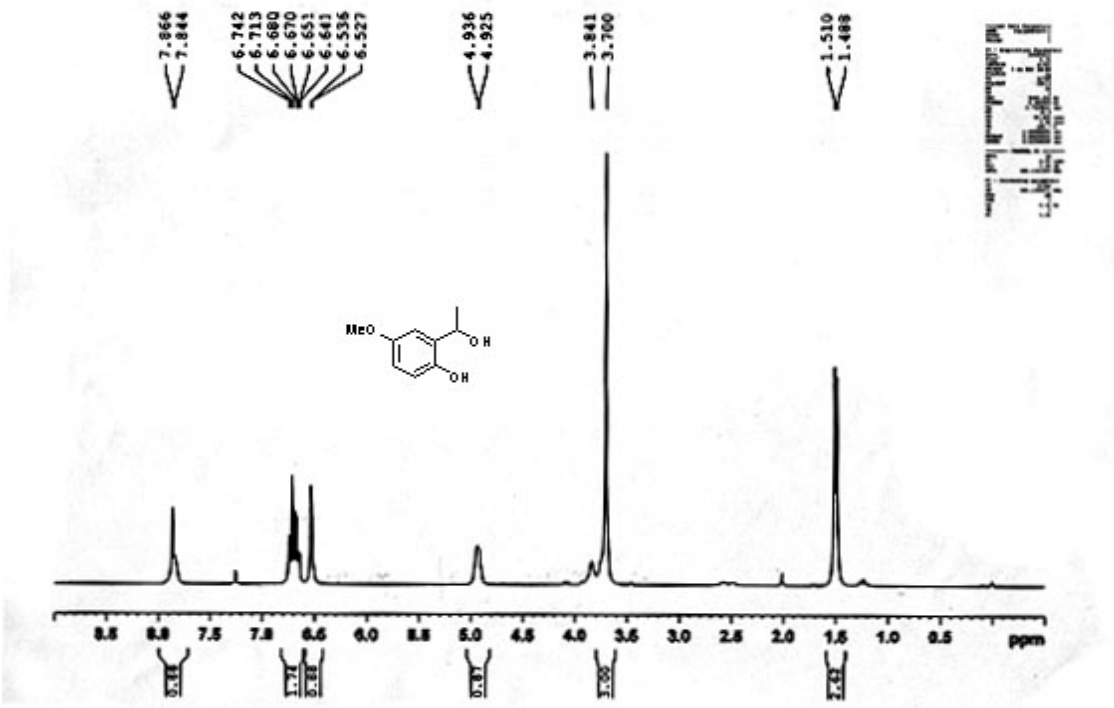

154.627
131.610
130.482
129.180
118.980
111.798
77.449
77.026
76.603
70.966
23.407
Br
OH
OH
180
160
140
120
100
80
60
40
20
0
ppm

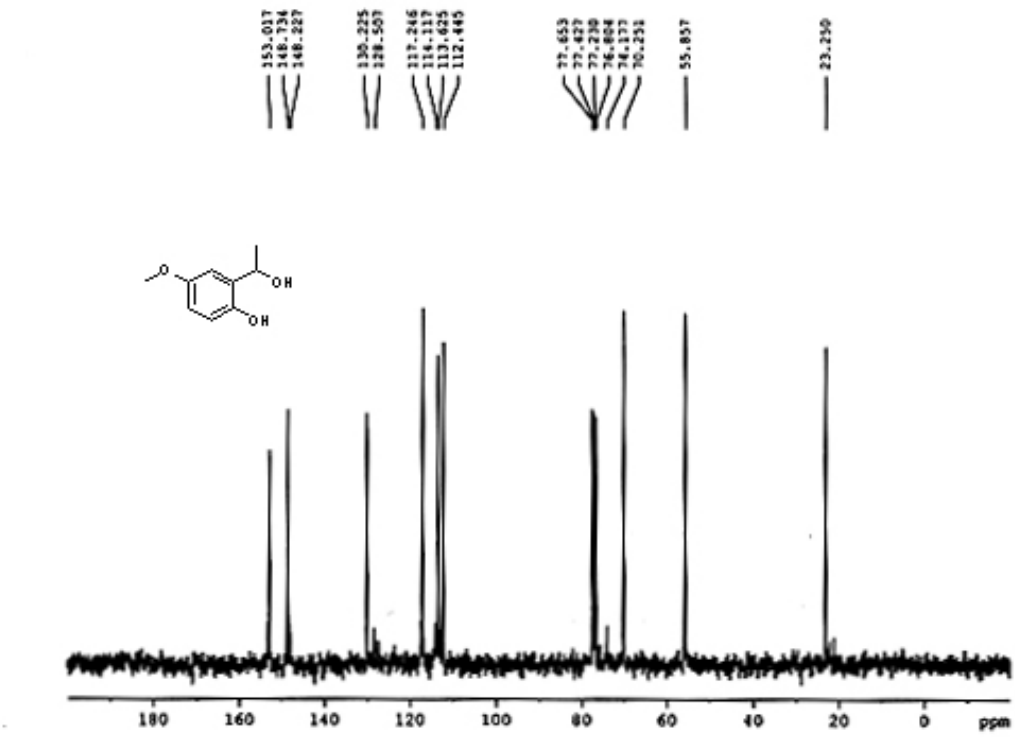
1e


8.087
7.245
7.219
7.194
7.172
7.165
7.148
7.122
6.859
6.833
6.814
6.808
6.798
5.900
2.322
OH
OH
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
ppm
0.97
3.98
1.83
0.65
0.96
3.00

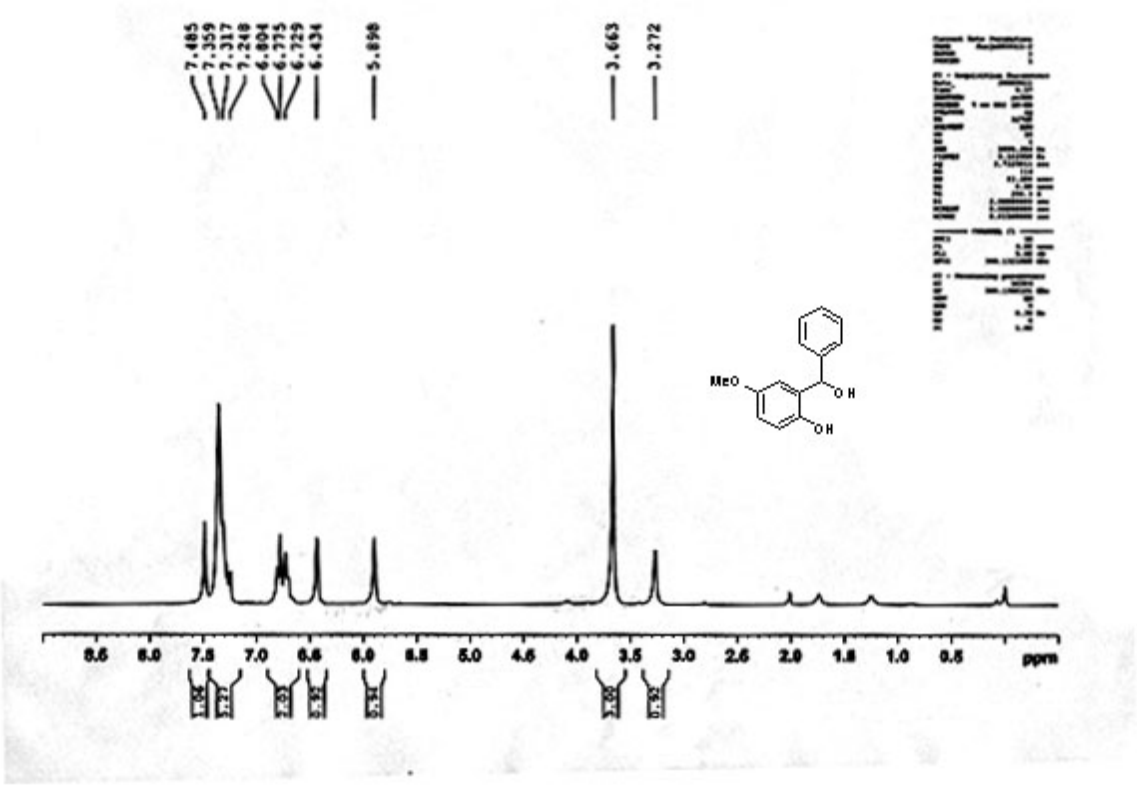

155.241
139.280
137.738
129.317
129.046
128.253
127.394
124.844
125.478
119.997
117.040
77.613
77.190
76.766
76.277
21.134
OH
OH
180
160
140
120
100
80
60
40
20
0
ppm

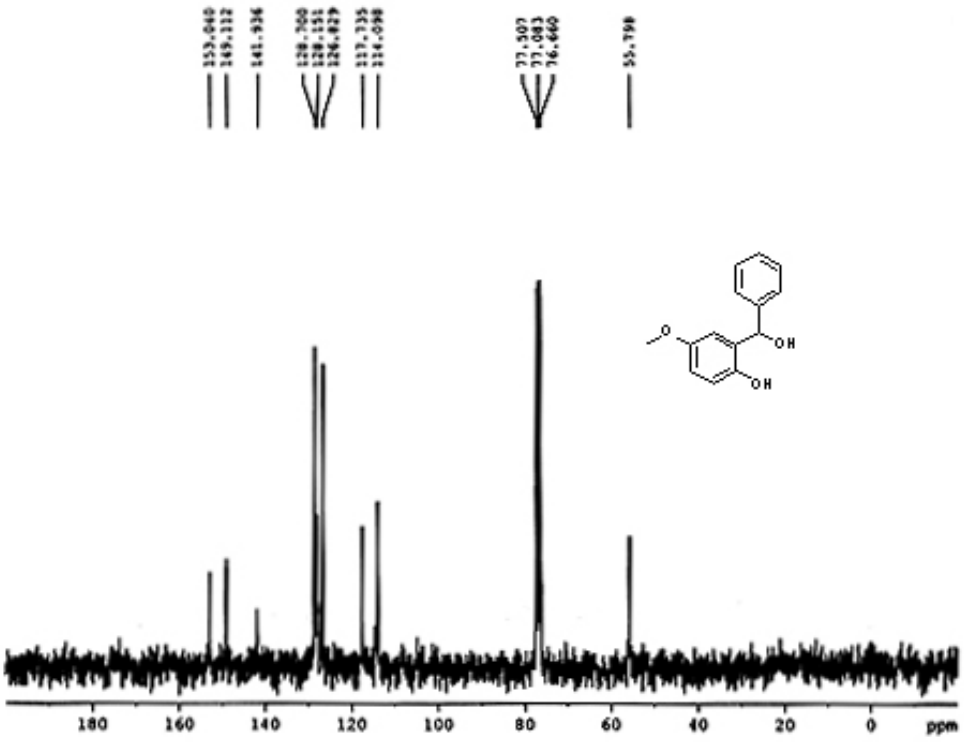
1f


7.866
7.844
6.742
6.713
6.680
6.670
6.651
6.641
6.536
6.527
4.936
4.925
3.841
3.700
1.510
1.488
MeO
OH
OH
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
ppm

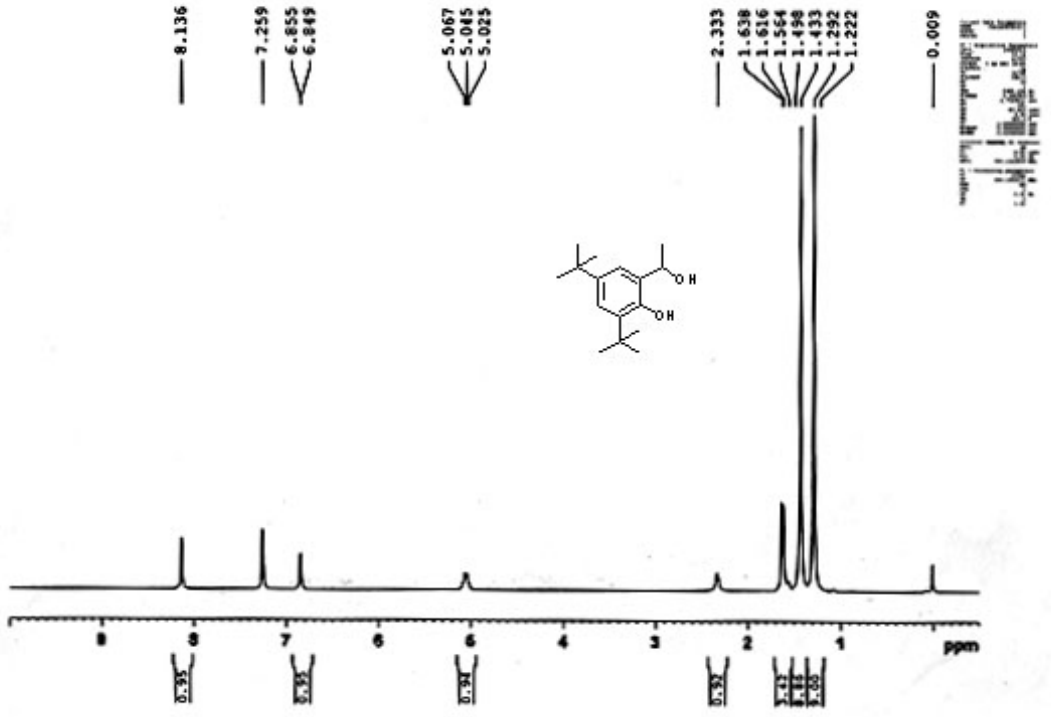

153.017
146.734
148.227
130.225
128.503
117.246
114.117
113.625
112.445
77.653
77.427
77.230
76.804
74.177
70.251
55.857
23.250
OH
OH
180
160
140
120
100
80
60
40
20
0
ppm

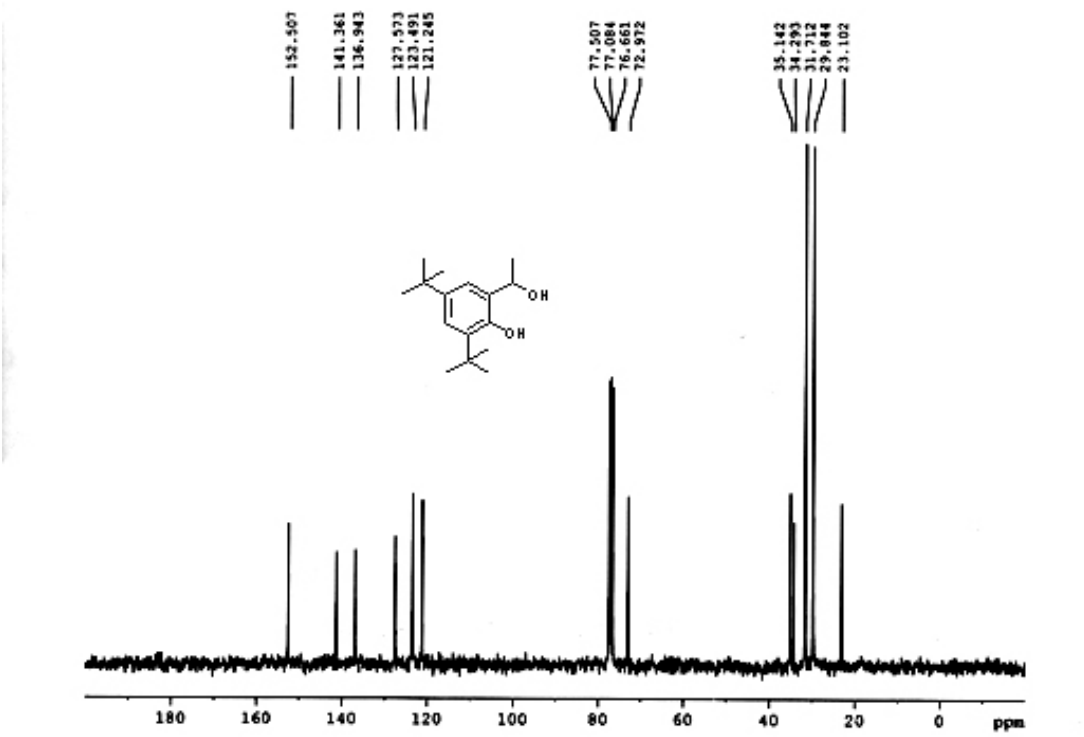
1g


7.485
7.359
7.317
7.248
6.804
6.775
6.729
6.434
5.898
3.663
3.272
MeO
OH
OH
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
ppm

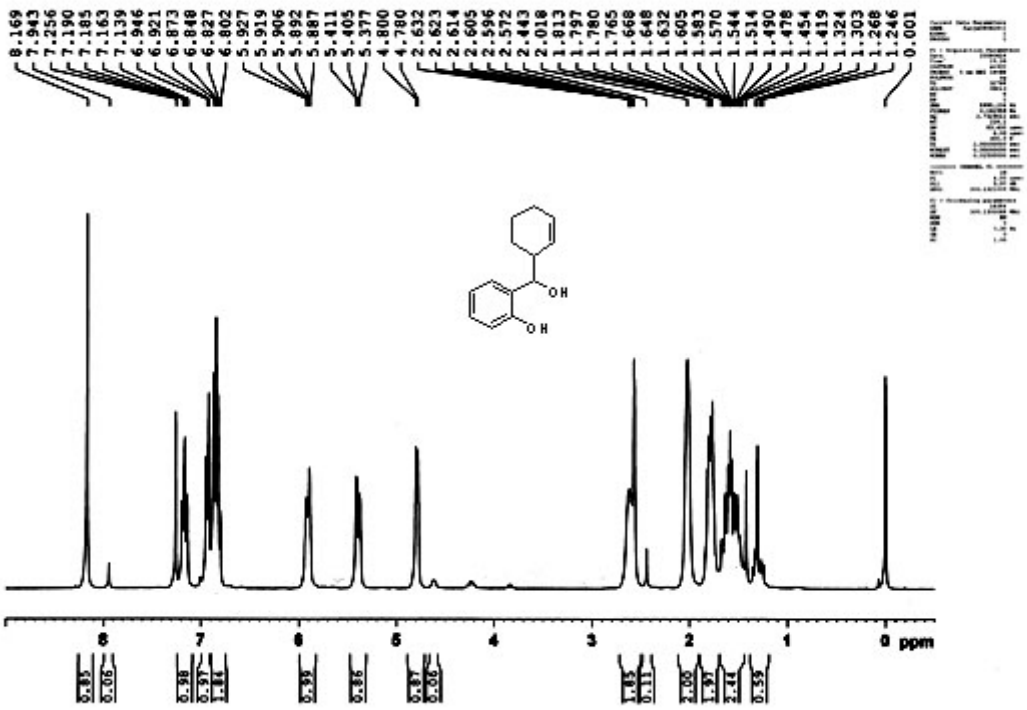

153.040
145.112
141.934
128.700
128.151
126.829
117.735
114.098
77.307
77.083
76.660
55.798
OH
OH
O
180
160
140
120
100
80
60
40
20
0
ppm

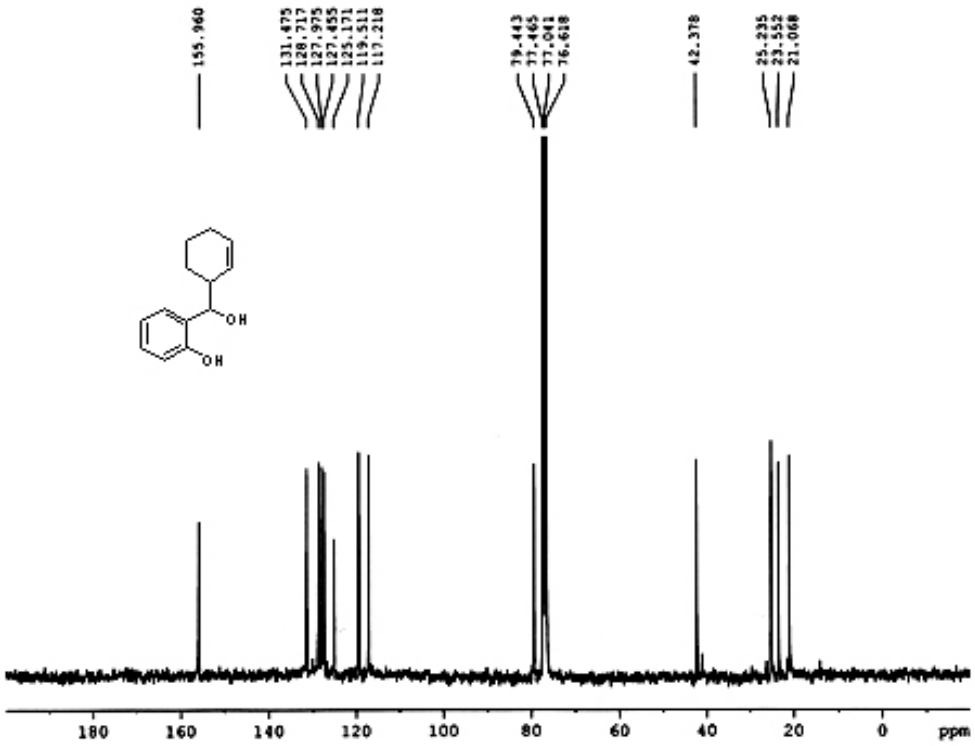
1h


8.136
7.259
6.855
6.849
5.067
5.045
5.025
2.333
1.638
1.616
1.564
1.498
1.433
1.292
1.222
0.009
OH
OH
8
7
6
5
4
3
2
1
ppm

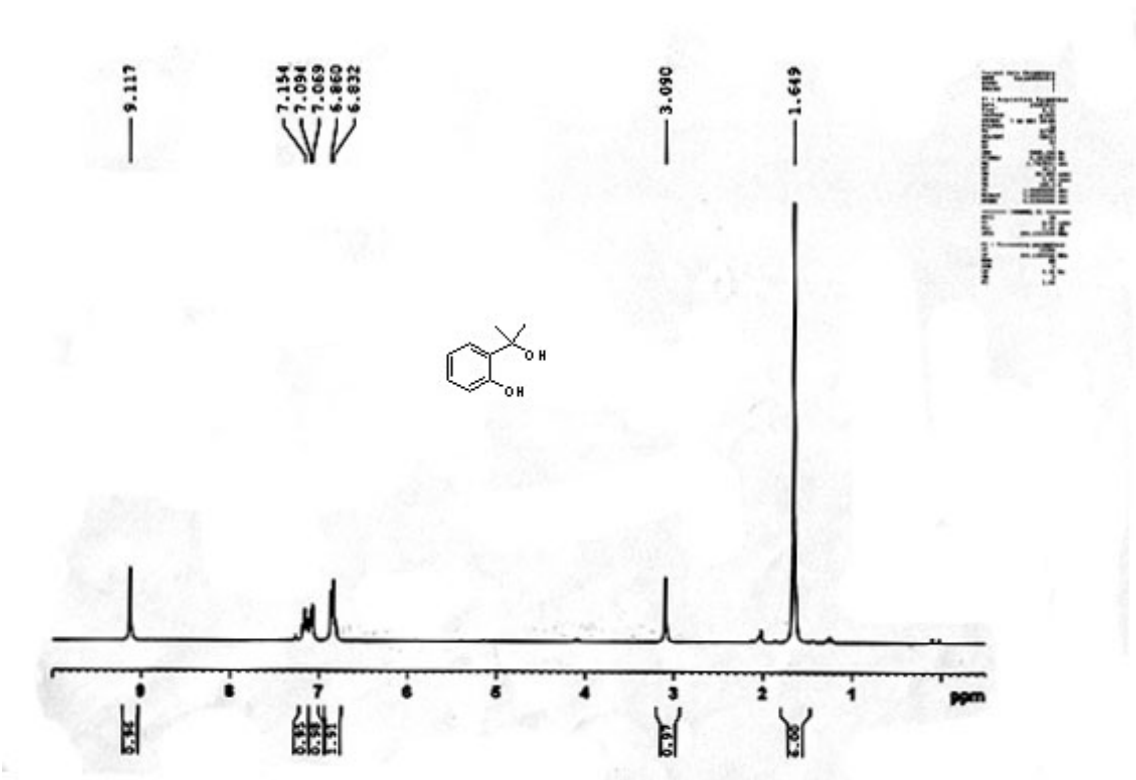

152.507
141.361
136.943
127.573
123.491
121.245
77.507
77.084
76.661
72.972
35.142
34.293
31.712
29.844
23.102
OH
OH
180
160
140
120
100
80
60
40
20
0
ppm

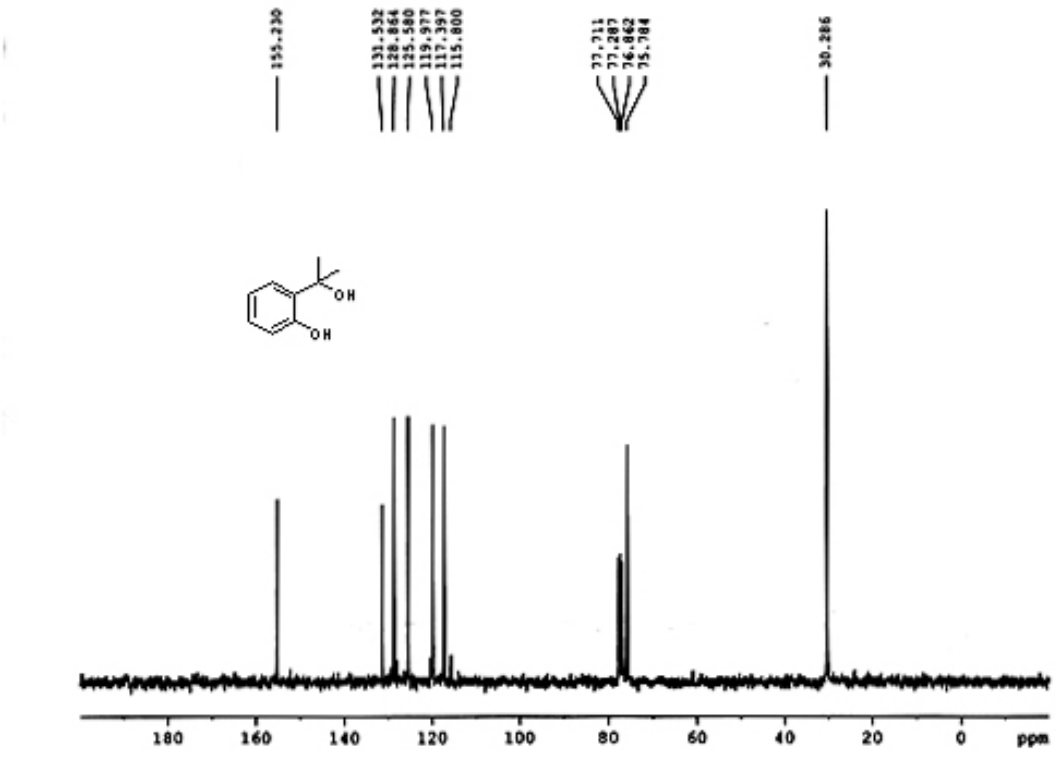
1i


8.169
7.943
7.256
7.190
7.185
7.163
7.139
6.946
6.921
6.873
6.848
6.827
6.802
5.927
5.919
5.906
5.892
5.887
5.411
5.405
5.377
4.800
4.780
2.632
2.623
2.614
2.605
2.596
2.572
2.443
2.018
1.813
1.797
1.780
1.765
1.668
1.648
1.632
1.605
1.583
1.570
1.544
1.514
1.490
1.478
1.454
1.419
1.324
1.303
1.268
1.246
0.001
OH
OH
8
7
6
5
4
3
2
1
0 ppm
0.85
0.06
0.98
0.97
1.84
0.98
0.86
0.87
0.68
0.85
0.11
2.00
1.9
2.14
0.55

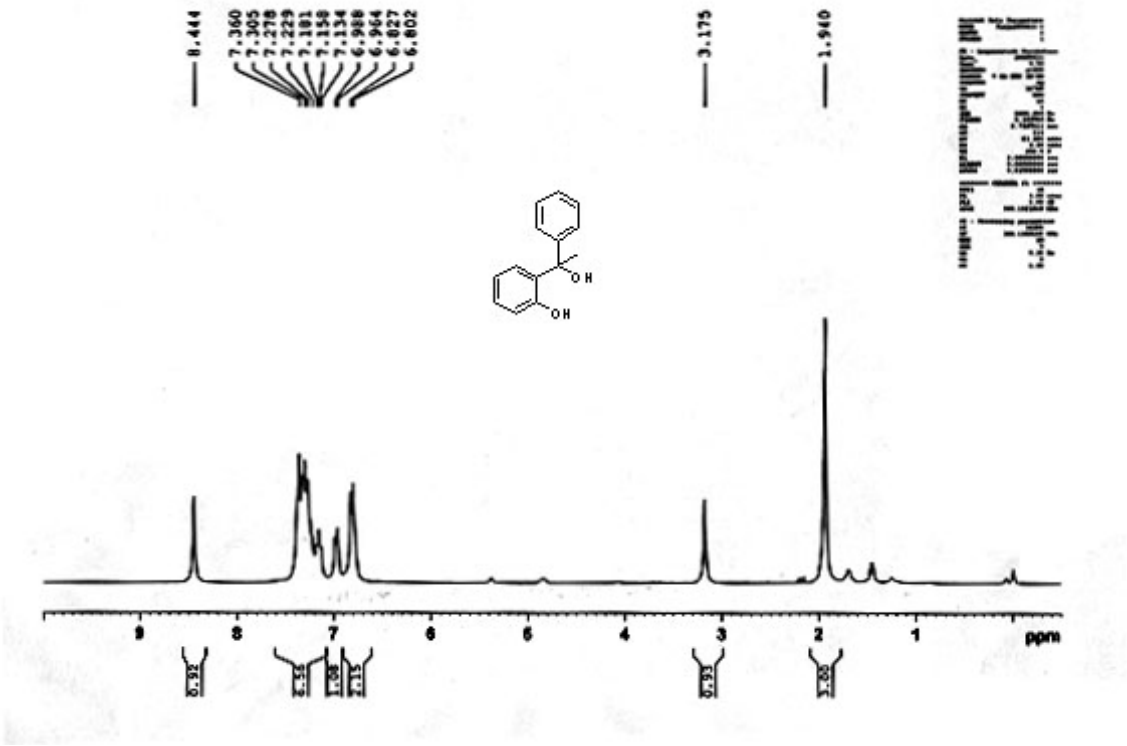

155.960
131.475
128.717
127.975
127.455
125.171
119.511
117.218
79.443
77.465
77.041
76.618
42.378
25.235
23.552
21.068
OH
OH
180
160
140
120
100
80
60
40
20
0
ppm

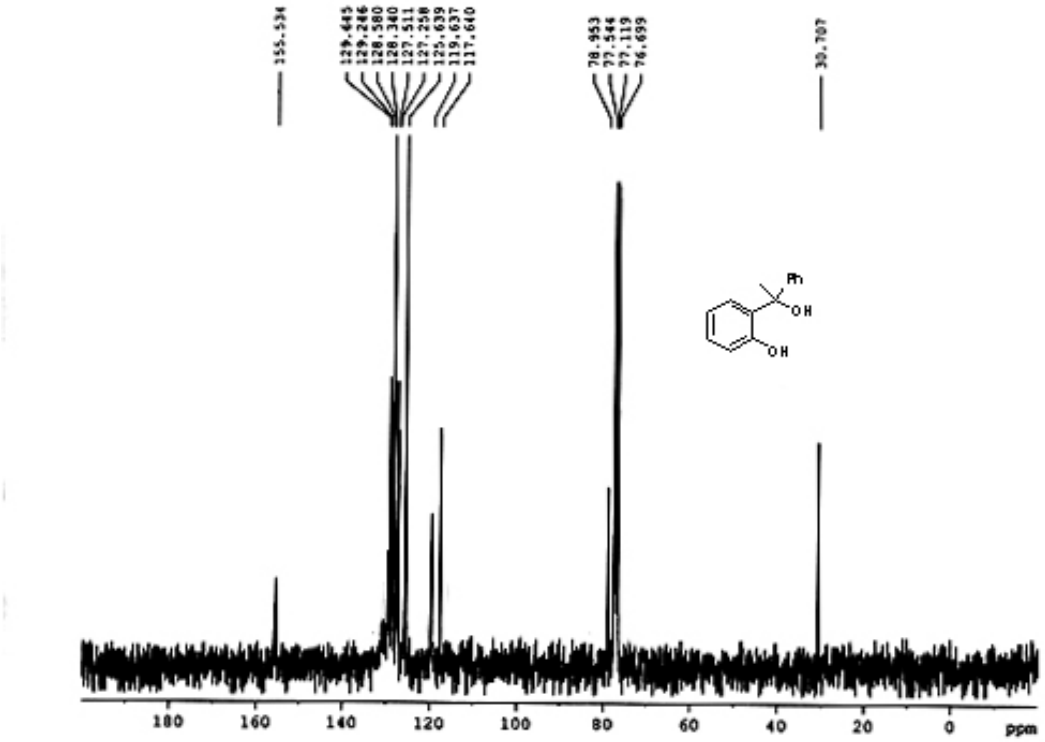
1j


9.117
7.154
7.094
7.069
6.860
6.832
3.090
1.649
OH
OH
9
8
7
6
5
4
3
2
1
ppm

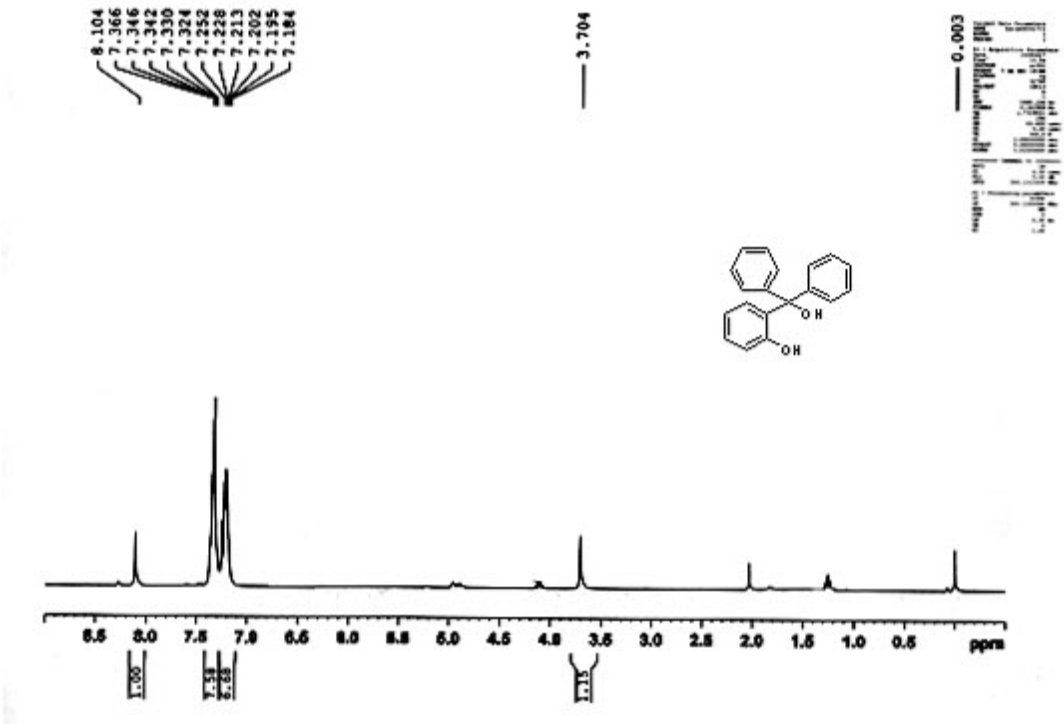

155.230
131.532
128.864
125.580
119.977
117.397
115.800
77.711
77.287
76.862
75.784
30.286
OH
OH
180
160
140
120
100
80
60
40
20
0
ppm

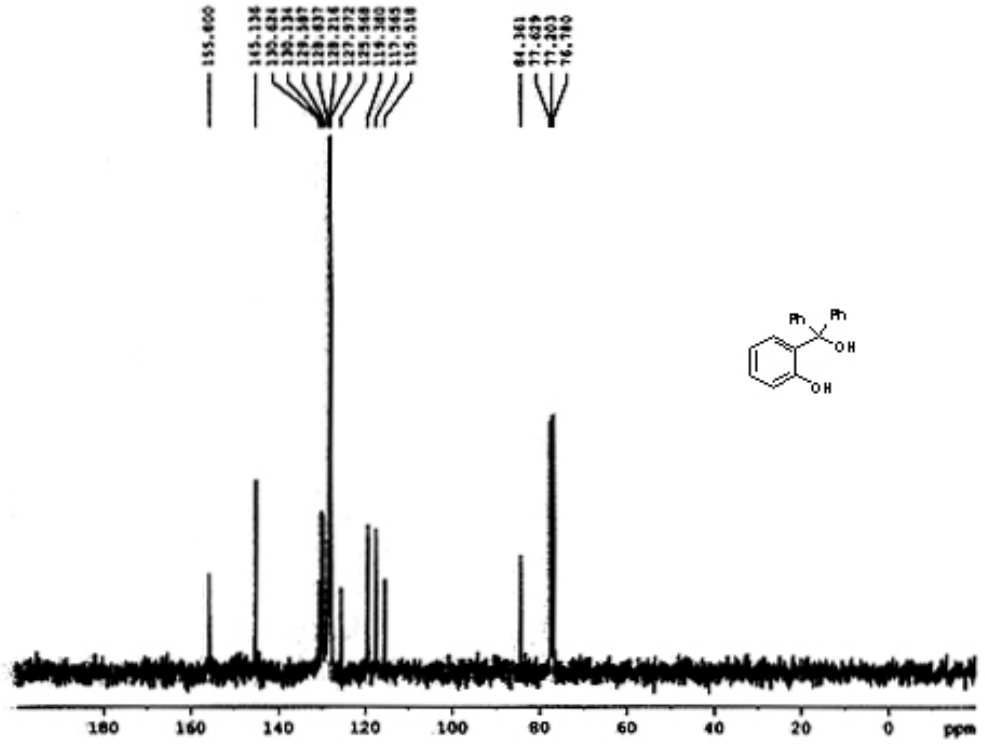
1k


8.444
7.360
7.305
7.278
7.229
7.181
7.158
7.134
6.988
6.964
6.827
6.802
3.175
1.940
OH
OH
9
8
7
6
5
4
3
2
1
ppm
0.92
2.55
1.00
2.15
0.93
3.00


155.534
129.445
129.246
128.560
128.340
127.511
127.258
125.639
119.637
117.640
78.953
77.544
77.119
76.699
30.707
Ph
OH
OH
180
160
140
120
100
80
60
40
20
0
ppm

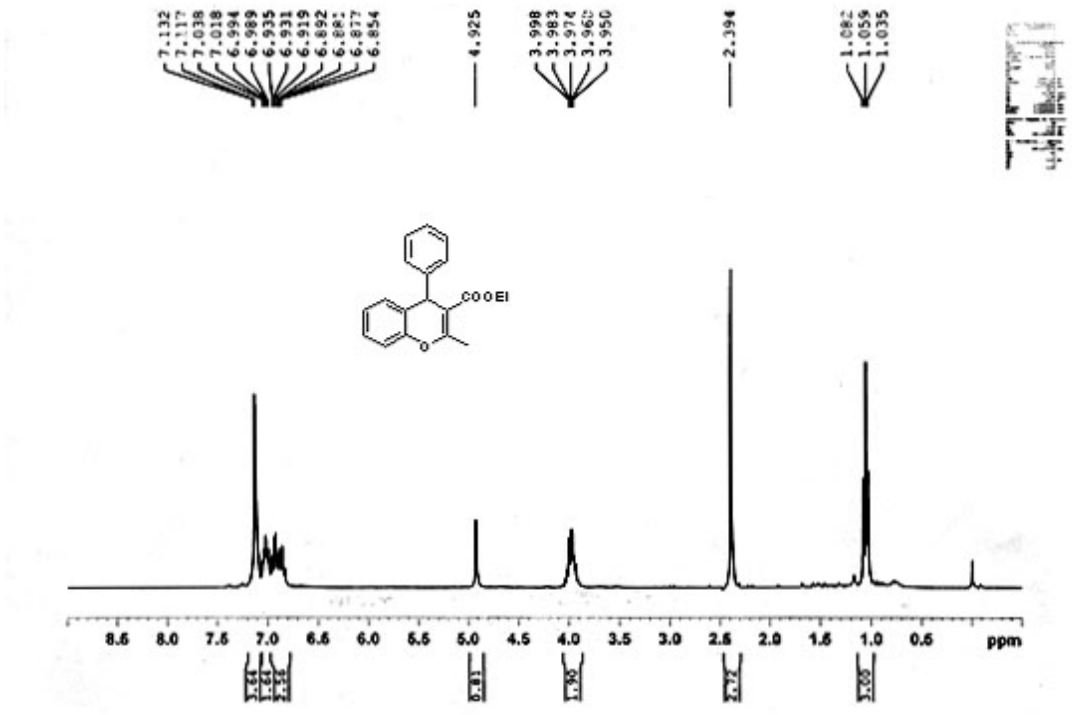
1l

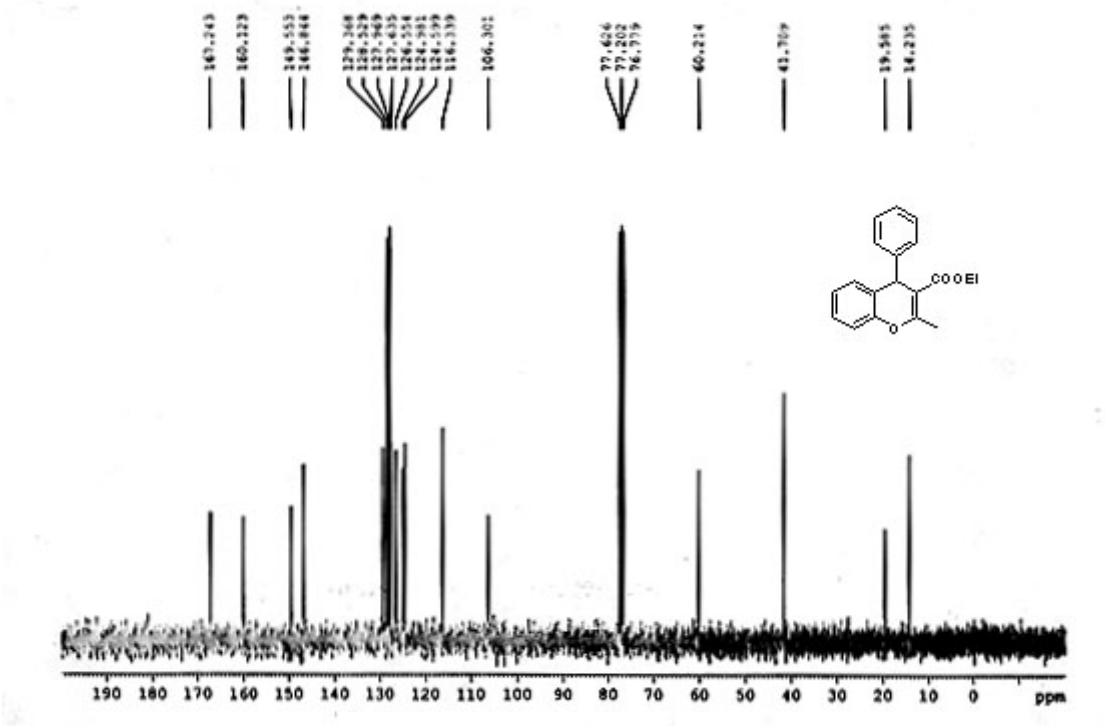

8.104
7.366
7.346
7.342
7.330
7.324
7.252
7.228
7.213
7.202
7.195
7.184
3.704
0.003
OH
OH
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
7.54
6.88
1.15



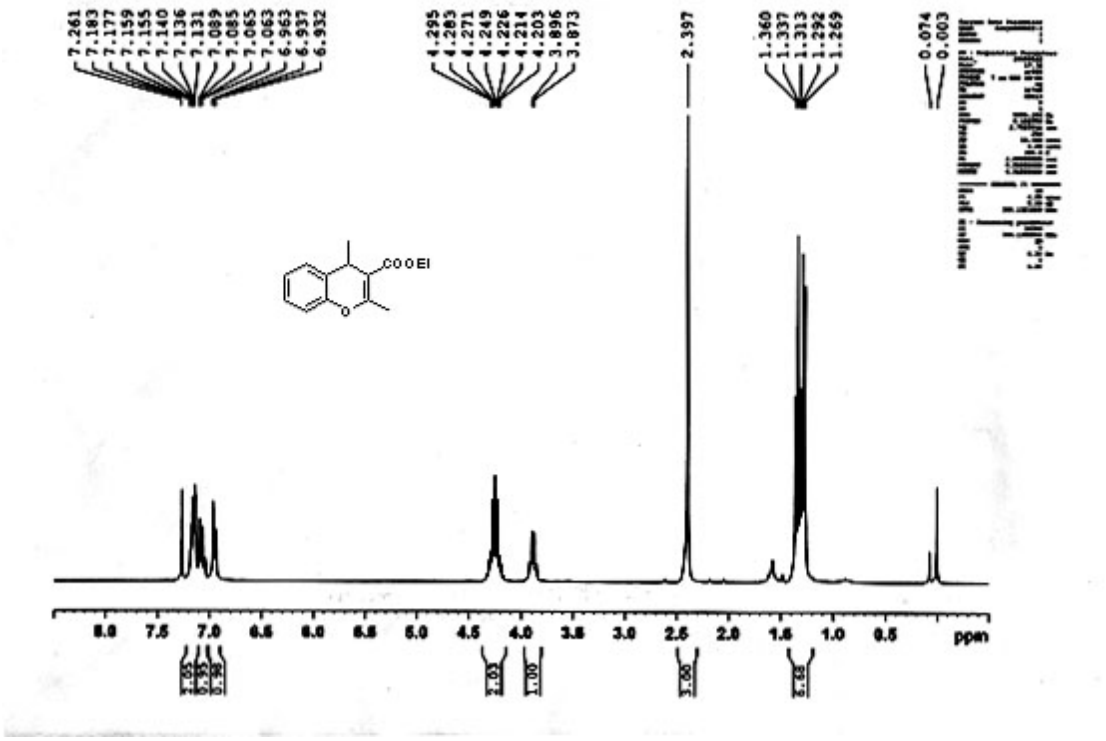
3.2 NMR of the 4H-Chromene products 3a-3s

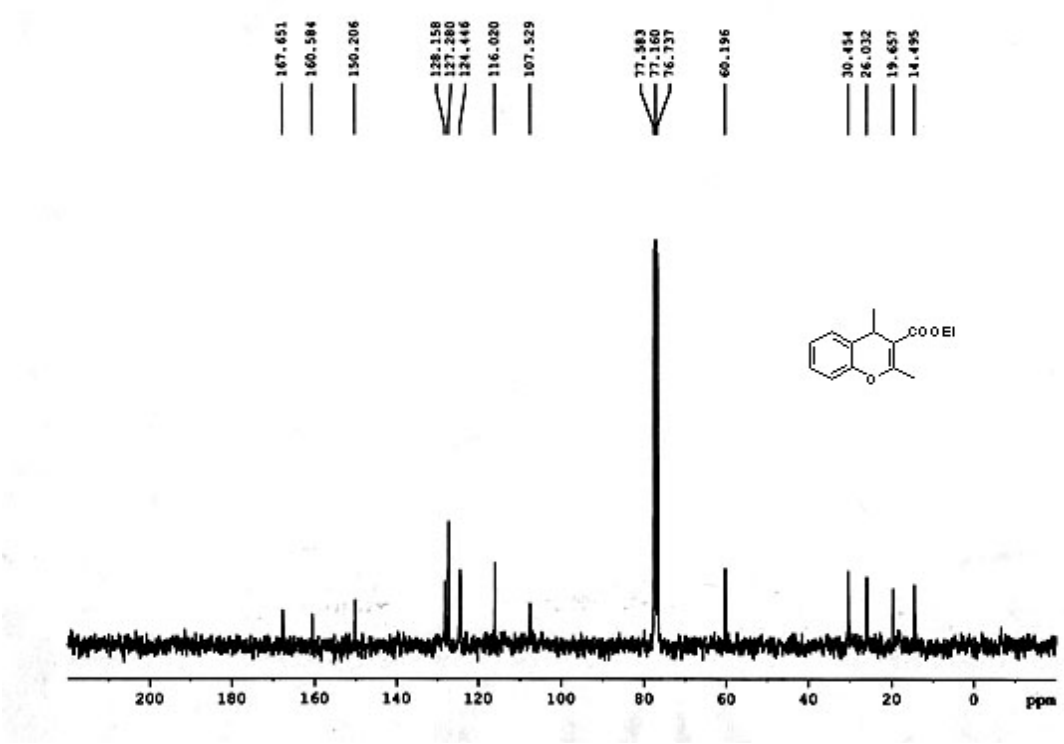
3a



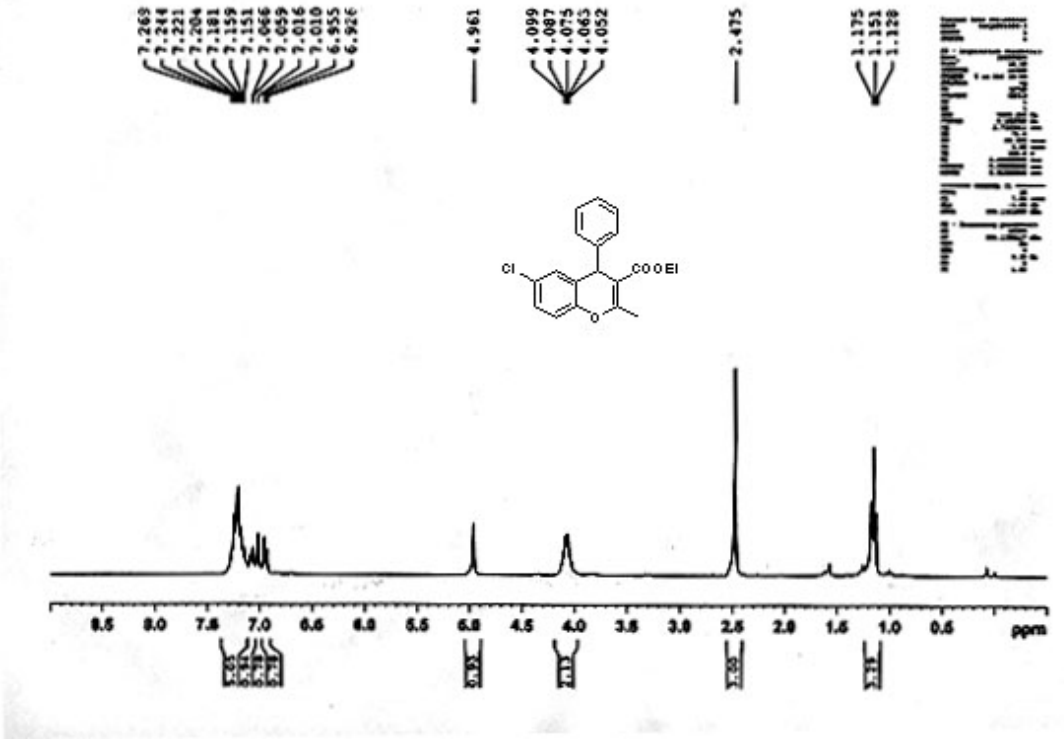

COOEt
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

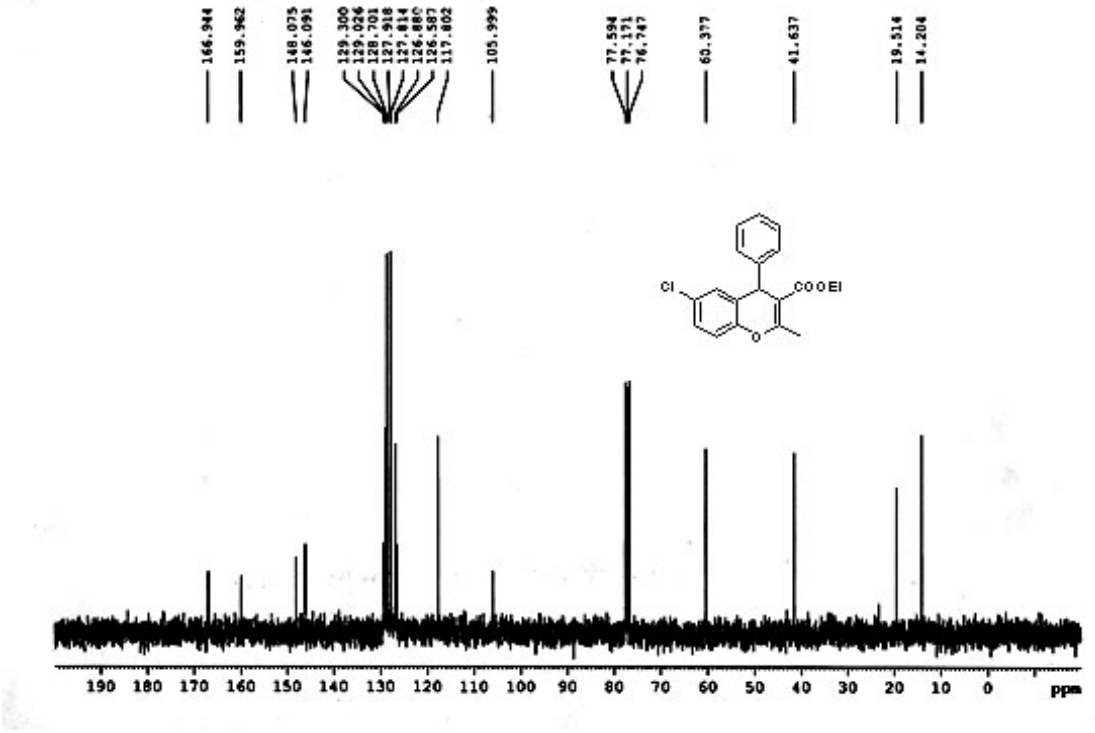
3b


7.261
7.183
7.177
7.159
7.155
7.140
7.136
7.131
7.089
7.085
7.065
7.063
6.963
6.937
6.932
4.295
4.283
4.271
4.249
4.226
4.214
4.203
3.896
3.873
2.397
1.360
1.337
1.313
1.292
1.269
0.074
0.003
COOEt
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 ppm

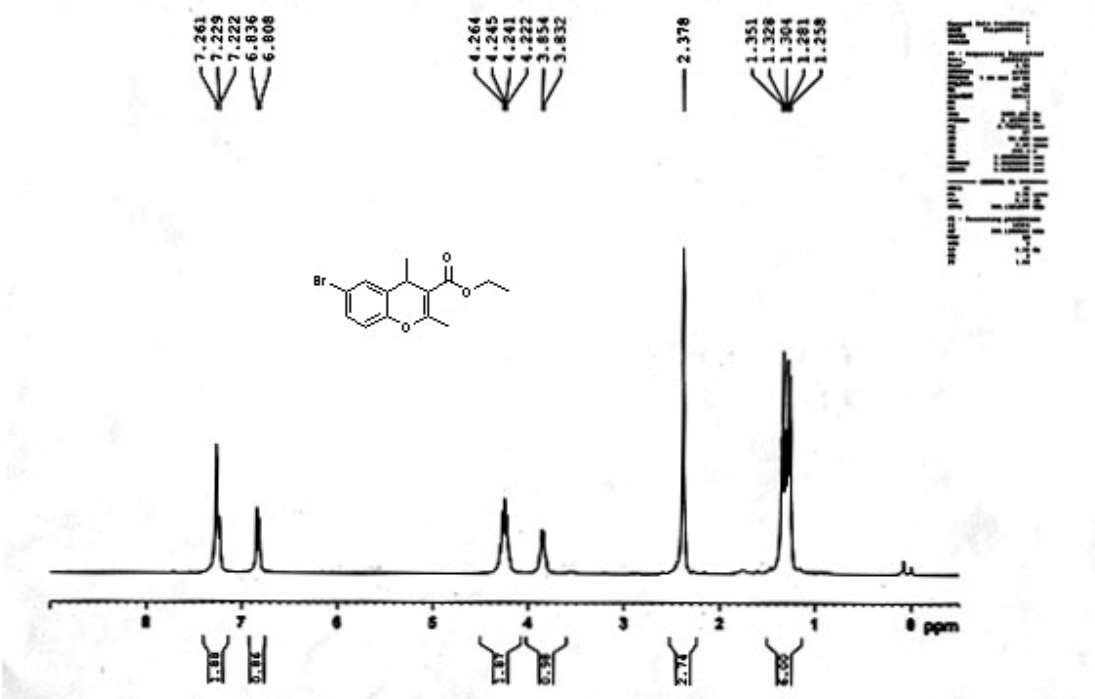

167.651
160.584
150.206
128.158
127.280
124.446
116.020
107.529
77.583
77.160
76.737
60.196
30.454
26.032
19.657
14.495
COOEt
200
180
160
140
120
100
80
60
40
20
0
ppm

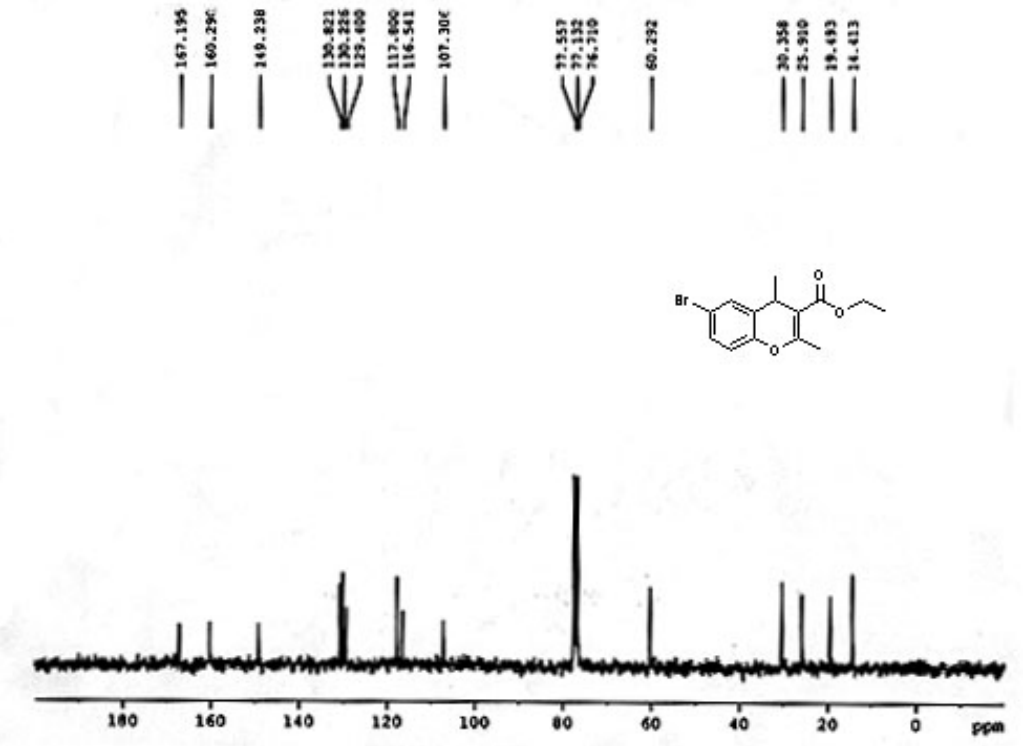
3c


7.269
7.244
7.221
7.204
7.181
7.159
7.151
7.066
7.059
7.016
7.010
6.955
4.961
4.099
4.087
4.075
4.065
4.052
2.475
1.175
1.151
1.128
Cl
COOEt
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
ppm

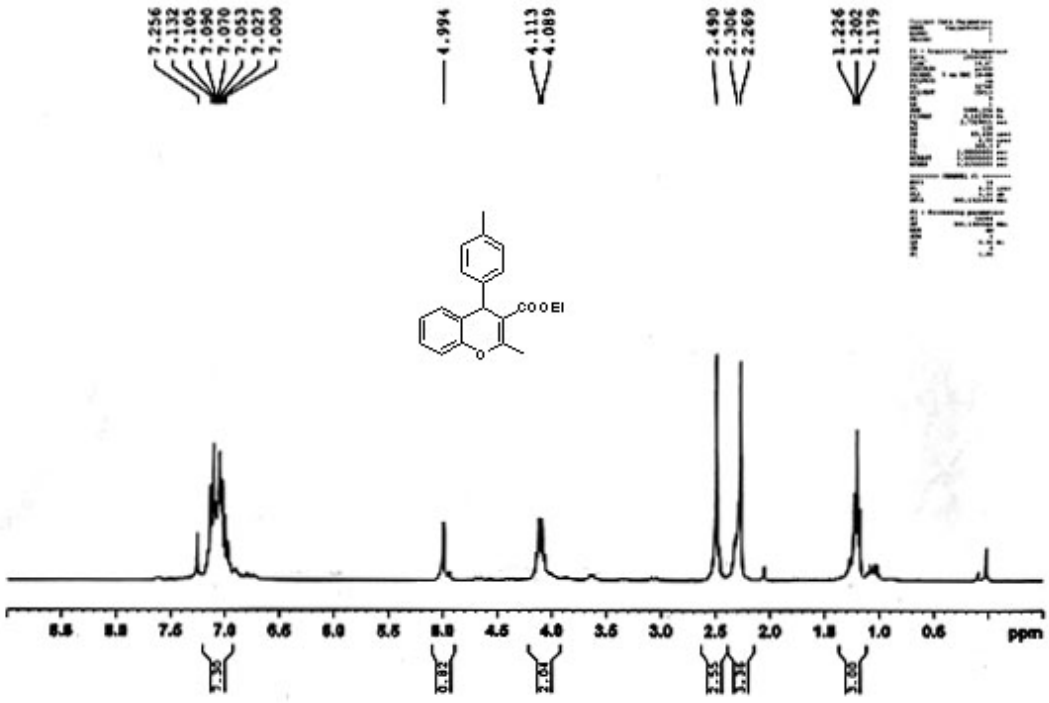

166.944
159.962
148.075
146.091
129.300
129.026
128.701
127.918
127.814
126.880
126.587
117.802
105.999
77.594
77.171
76.747
60.377
41.637
19.514
14.204
Cl
COOEt
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

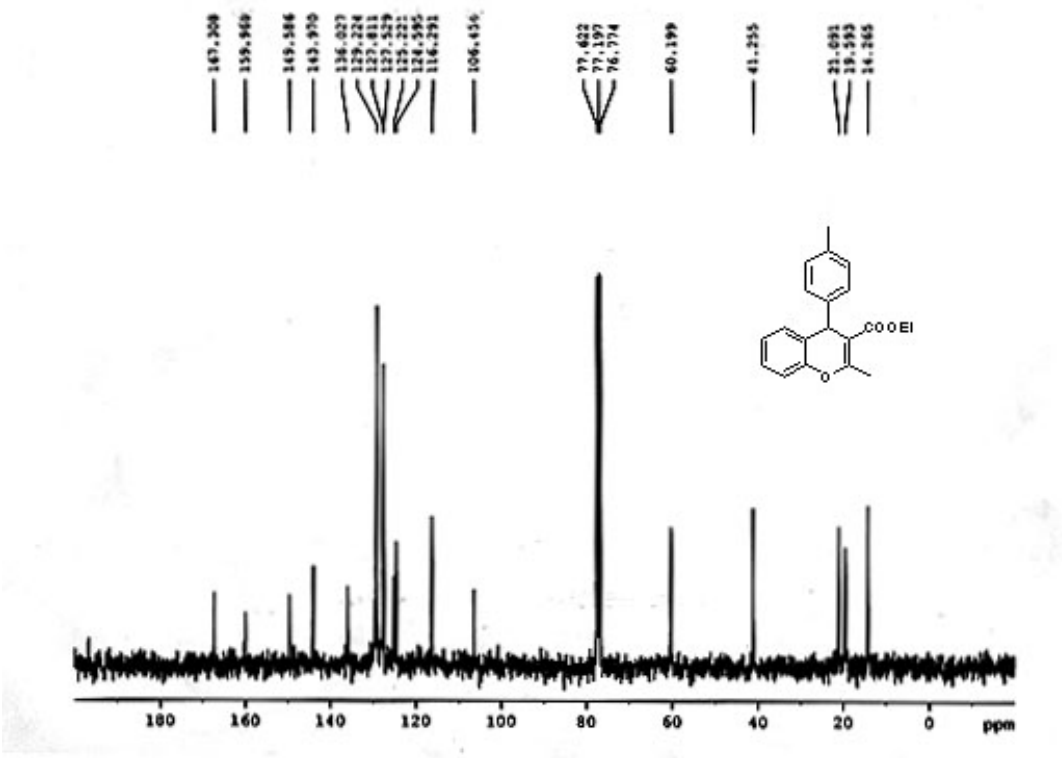
3d


7.261
7.229
7.222
6.836
6.808
4.264
4.245
4.241
4.222
3.854
3.832
2.378
1.351
1.328
1.304
1.281
1.258
Br
O
O
O
8 7 6 5 4 3 2 1 0 ppm


167.195
160.290
149.238
130.821
130.226
129.480
117.800
116.541
107.306
77.557
77.132
76.710
60.292
30.358
25.930
18.493
14.413
Br
O
O
O
180
160
140
120
100
80
60
40
20
0
ppm

3e


7.256
7.132
7.105
7.090
7.070
7.053
7.027
7.000
4.994
4.113
4.089
2.490
2.306
2.269
1.226
1.202
1.179
COOEt
O
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
ppm
7.36
0.82
2.04
2.35
3.31
2.00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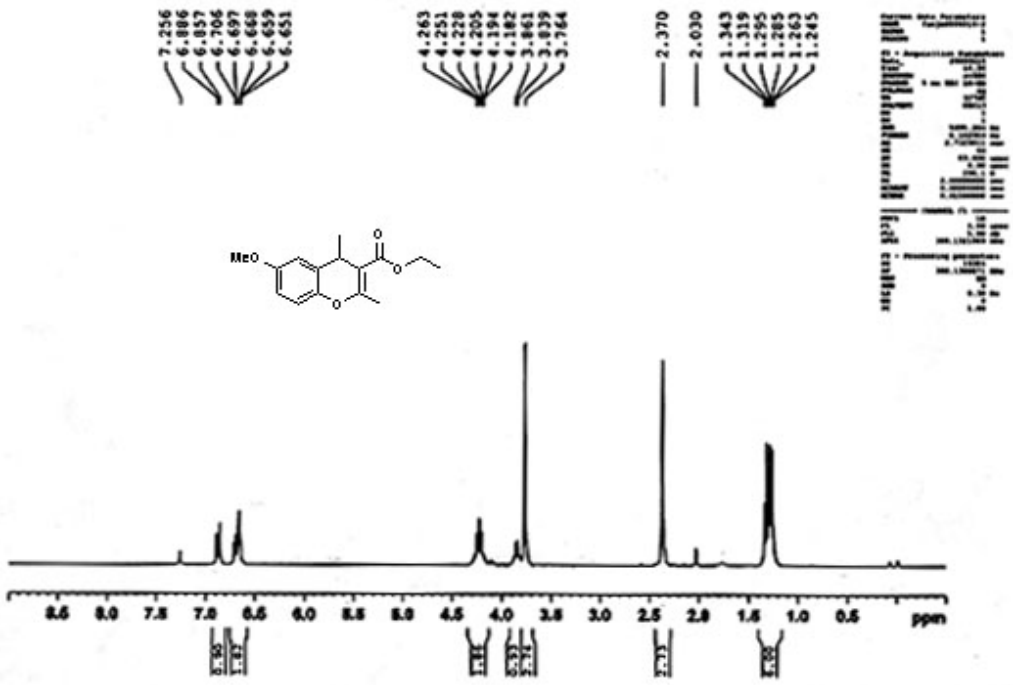
159.960
149.586
143.970
136.027
129.224
127.811
127.529
125.222
124.595
116.291
106.436
77.622
77.197
76.774
60.199
41.255
21.691
19.593
14.265
COOEt
O
180
160
140
120
100
80
60
40
20
0
ppm

3f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6.886
6.857
6.706
6.697
6.668
6.659
6.651
4.263
4.251
4.228
4.205
4.194
4.182
3.861
3.839
3.764
2.370
2.030
1.343
1.319
1.295
1.285
1.263
1.245
MeO
O
O
8.5
8.0
7.5
7.0
6.5
6.0
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4.5
4.0
3.5
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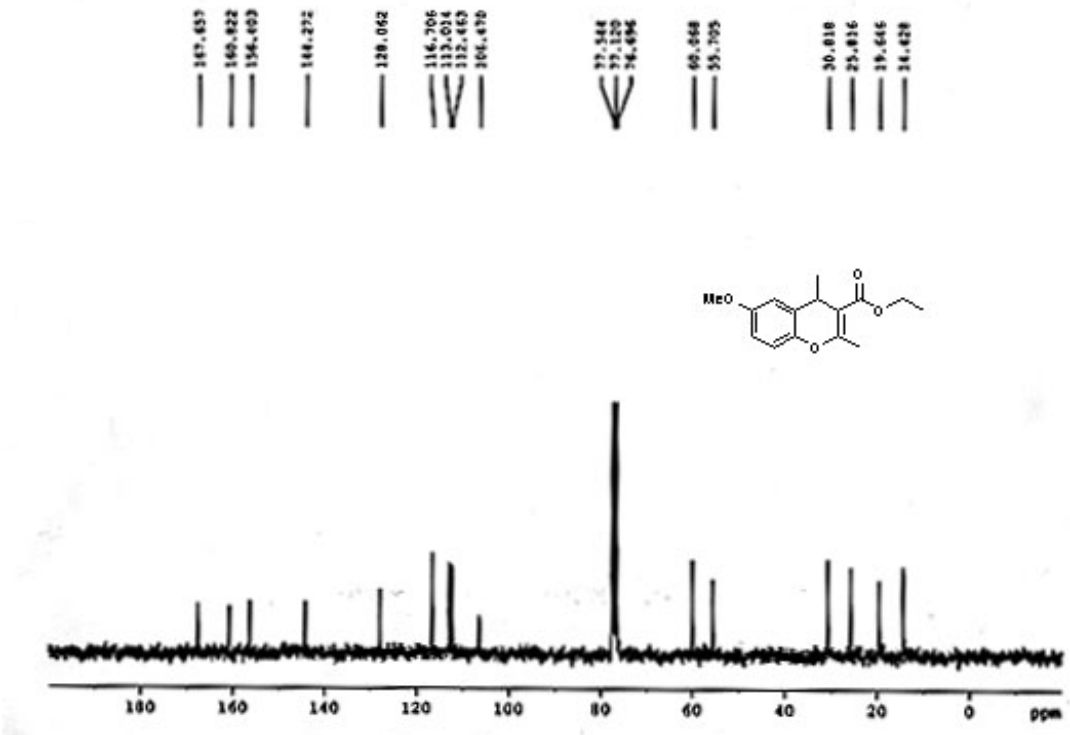
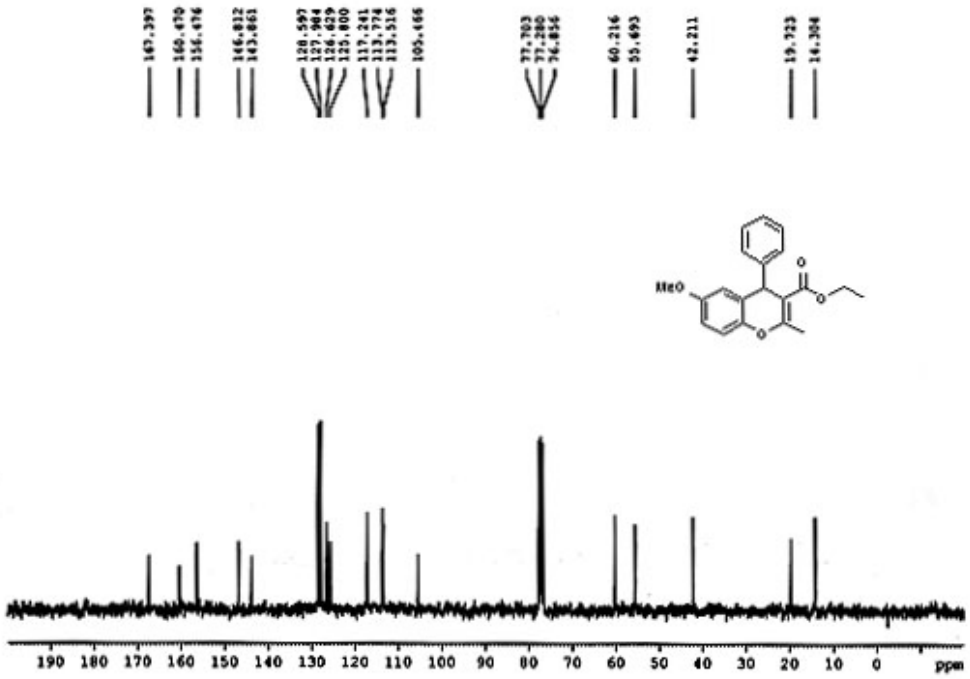
2.5
2.0
1.5
1.0
0.5
ppm

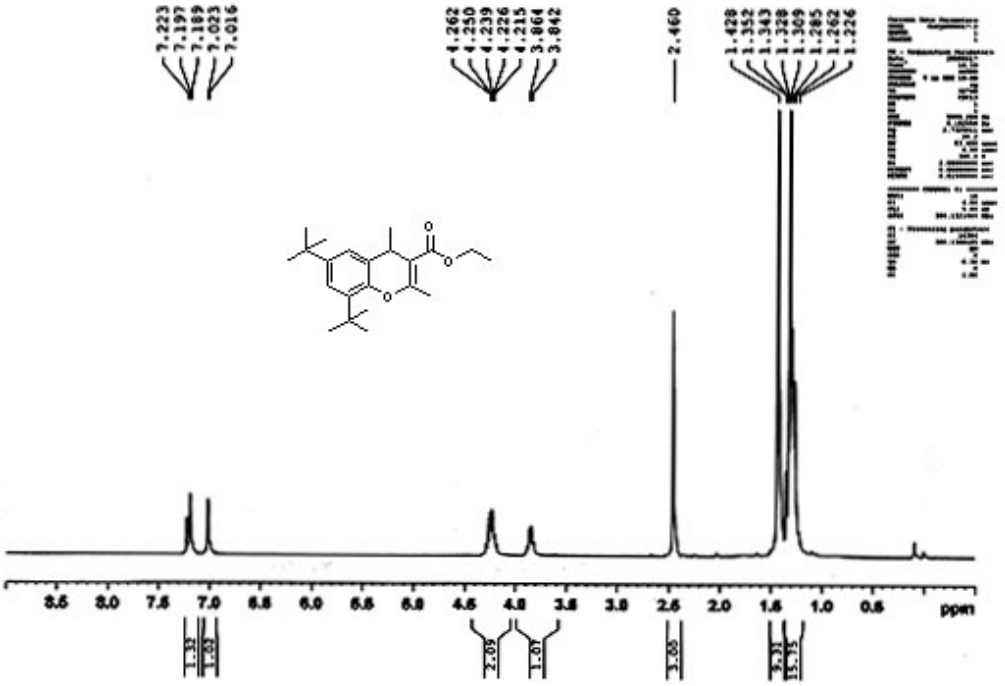

MeO
O
O
O
180
160
140
120
100
80
60
40
20
0
ppm

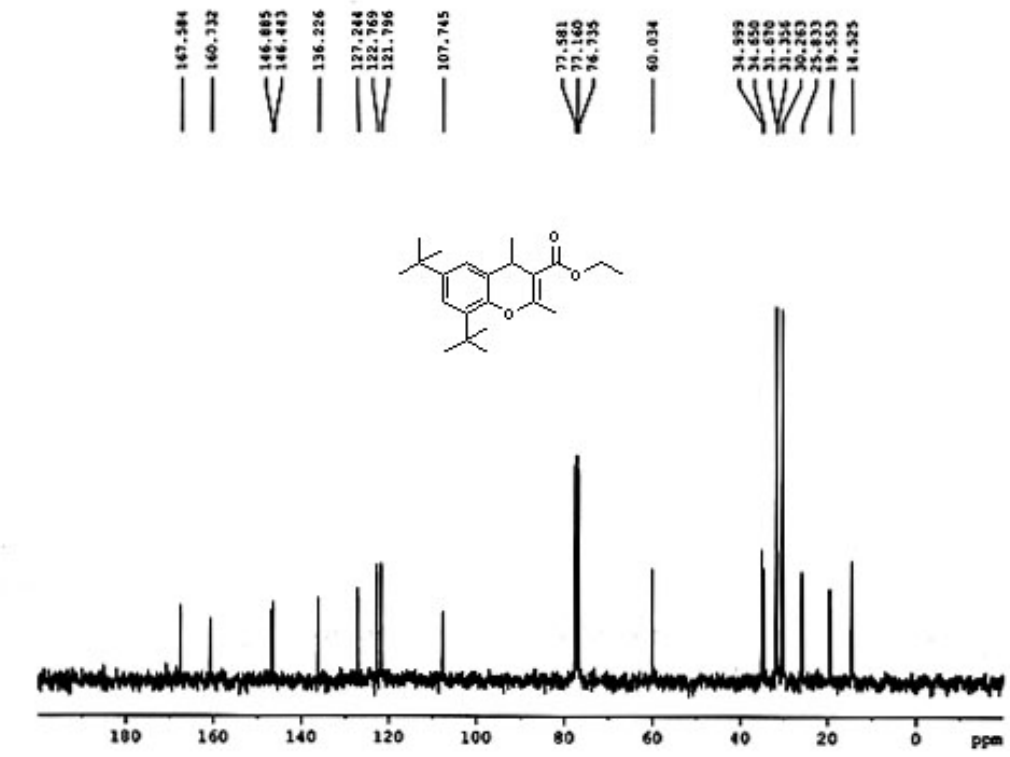
3g


7.217
7.203
6.920
6.539
6.530
4.978
4.084
4.069
4.056
4.041
3.635
2.466
1.167
1.144
1.120
MeO
O
O
O
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
ppm

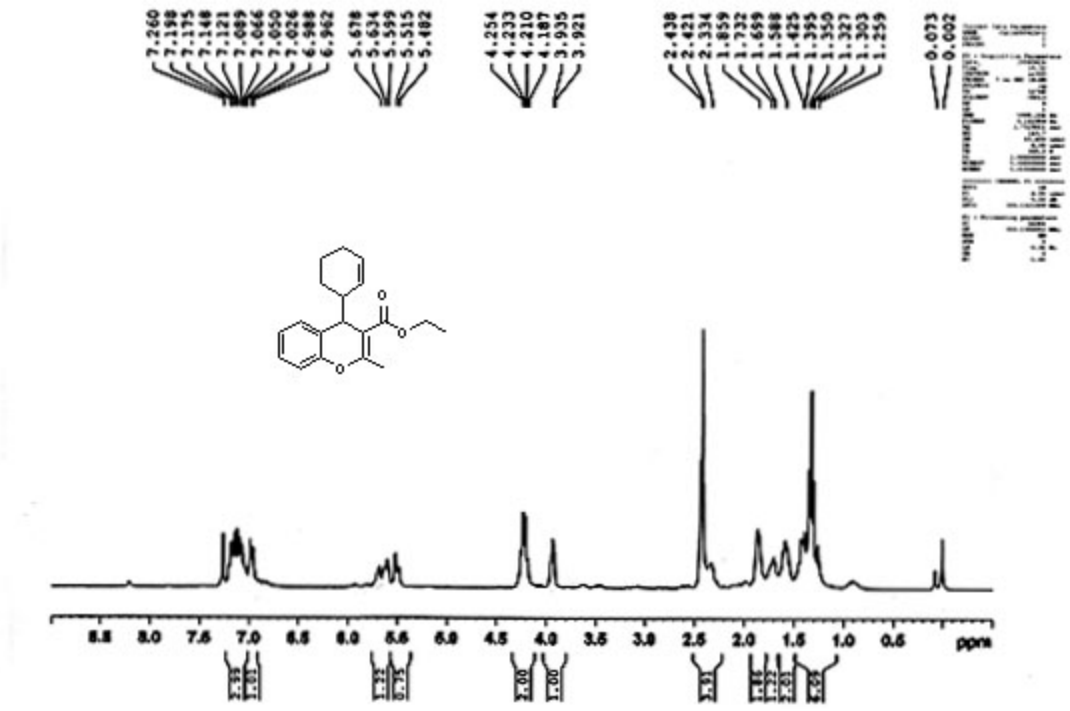

MeO
O
O
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

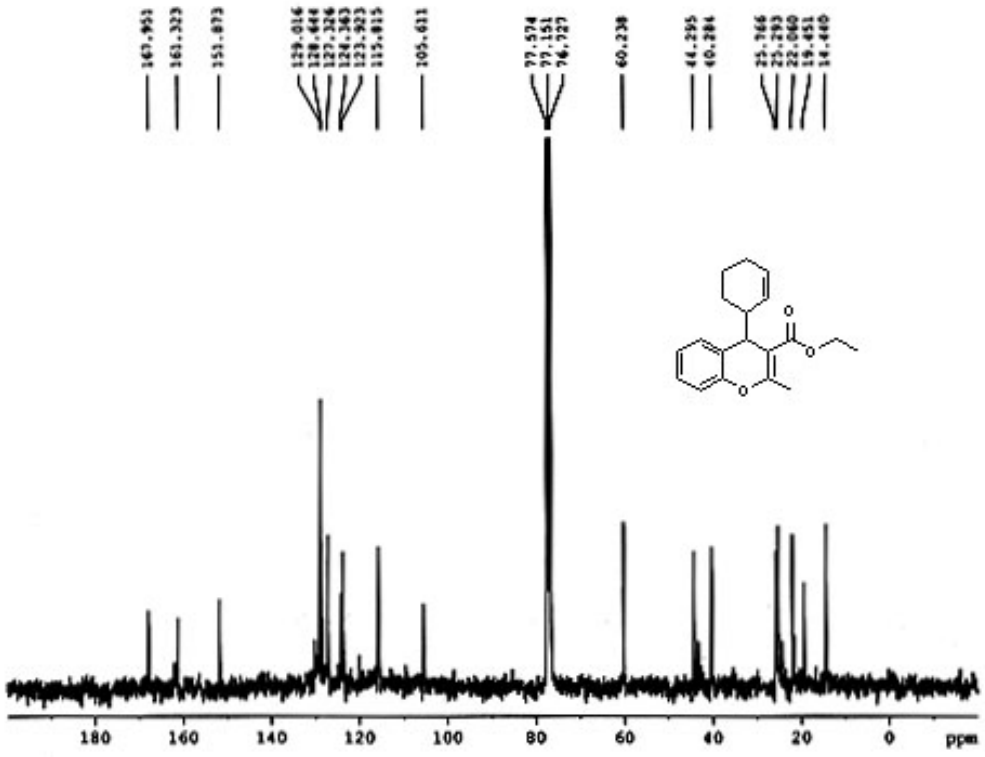
3h


O
O
O
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 ppm

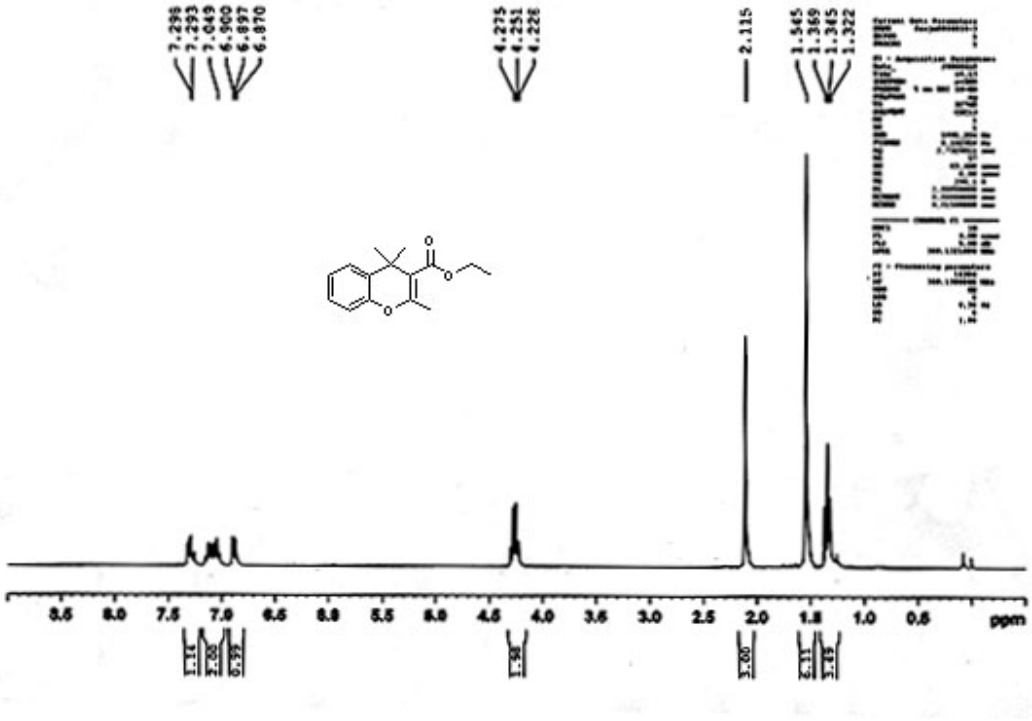

167.384
160.732
146.885
146.443
136.226
127.244
122.769
121.796
107.745
77.581
77.160
76.735
60.034
34.959
34.850
31.670
31.356
30.263
25.833
19.553
14.325
180
160
140
120
100
80
60
40
20
0
ppm

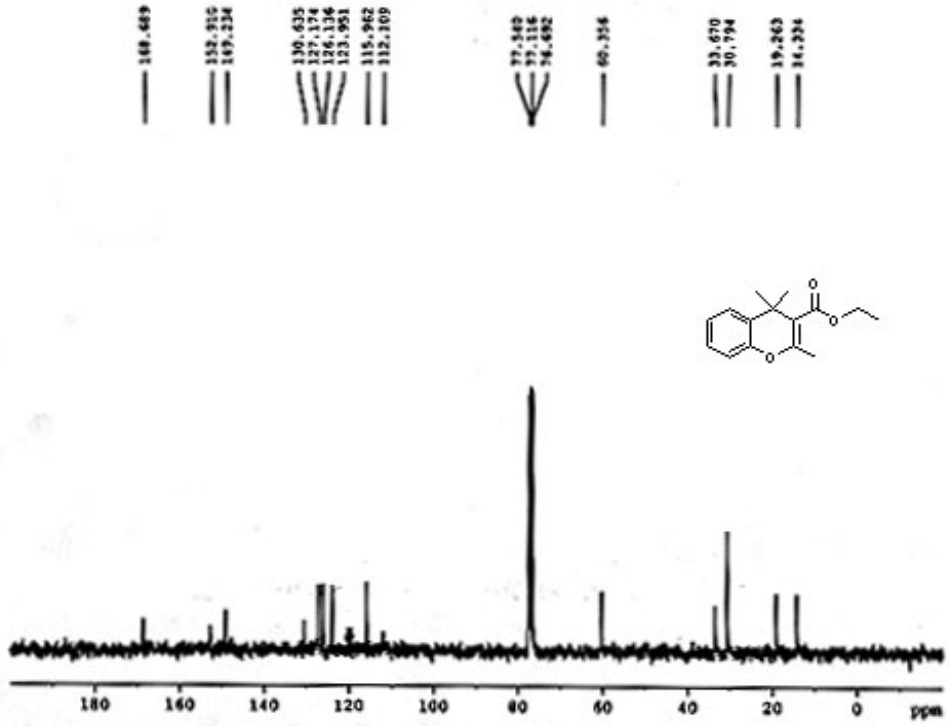
3i


7.260
7.198
7.175
7.148
7.121
7.089
7.066
7.050
7.026
6.988
6.962
5.678
5.634
5.599
5.515
5.482
4.254
4.233
4.210
4.187
3.935
3.921
2.438
2.421
2.334
1.859
1.732
1.659
1.588
1.425
1.395
1.350
1.327
1.303
1.259
0.073
0.002
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
ppm

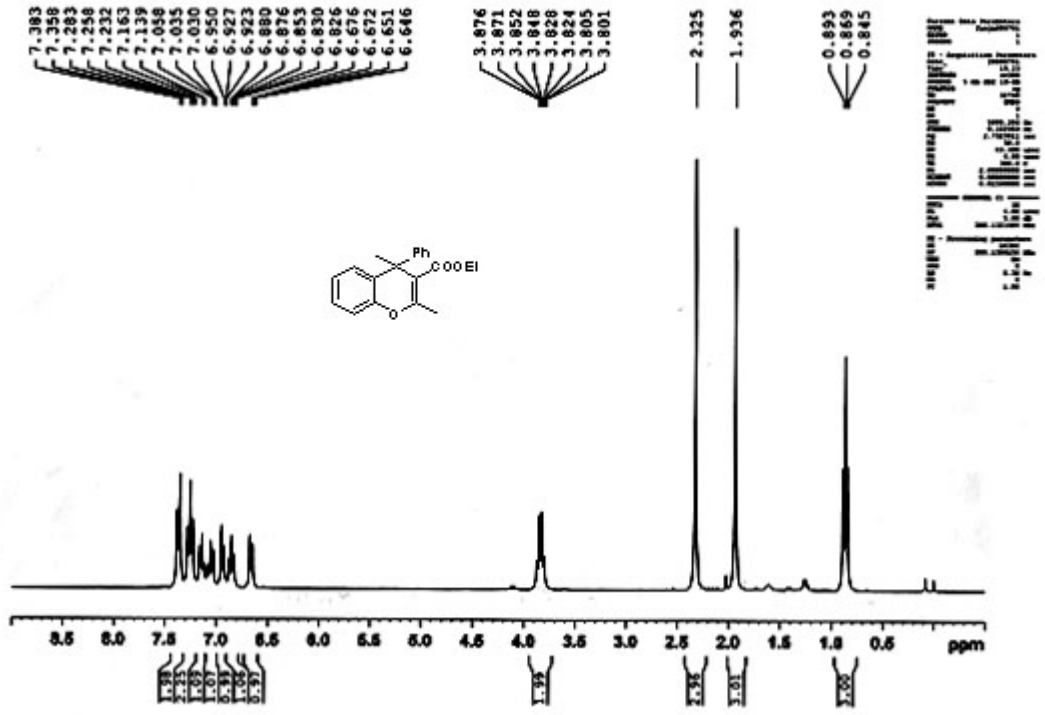

167.953
161.323
153.873
129.016
128.444
127.326
124.343
123.923
115.815
105.611
77.574
77.151
76.727
60.238
44.295
40.284
25.366
25.253
22.060
19.451
14.440
O
O
O
180
160
140
120
100
80
60
40
20
0
ppm

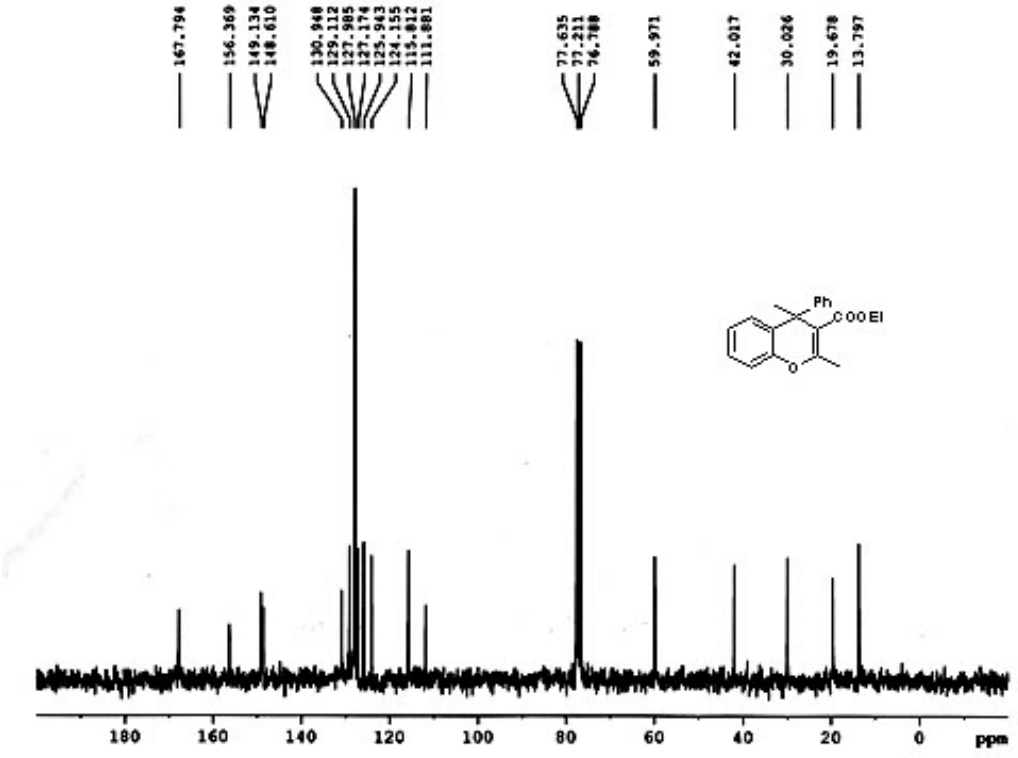
3j


7.296
7.293
7.049
6.900
6.897
6.870
4.275
4.251
4.228
2.115
1.545
1.369
1.345
1.322
O
O
O
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
ppm
1.14
2.00
0.97
1.98
3.00
2.11
3.19

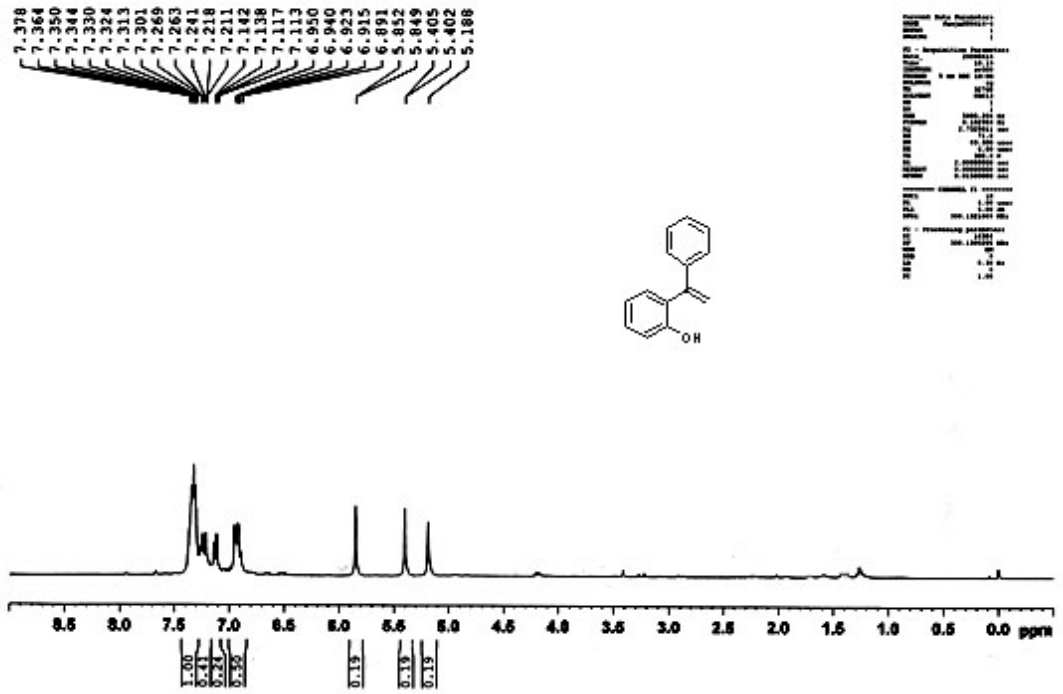

168.489
152.515
149.234
130.635
127.174
126.134
123.951
115.962
112.109
77.349
77.116
76.882
60.356
33.670
30.794
19.263
14.334
O
O
O
180
160
140
120
100
80
60
40
20
0
ppm

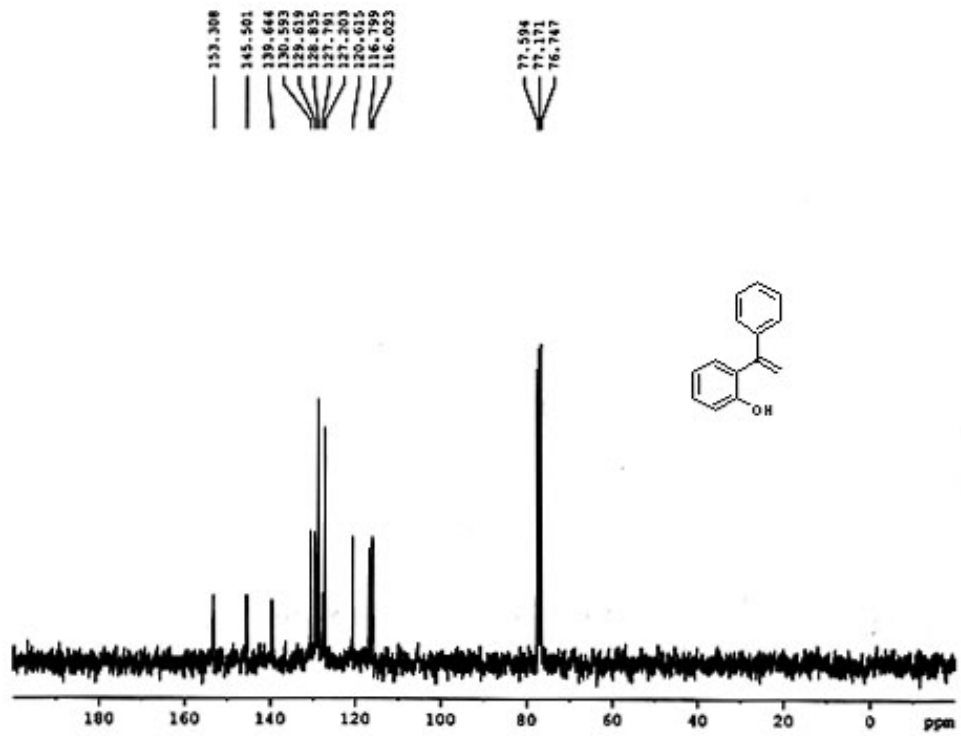
3k


7.383
7.358
7.283
7.258
7.232
7.163
7.139
7.058
7.035
7.030
6.950
6.927
6.923
6.880
6.876
6.853
6.830
6.826
6.676
6.672
6.651
6.646
3.876
3.871
3.852
3.848
3.828
3.824
3.805
3.801
2.325
1.936
0.893
0.869
0.845
Ph
COOEt
O
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
ppm



2-(1-phenylvinyl)phenol

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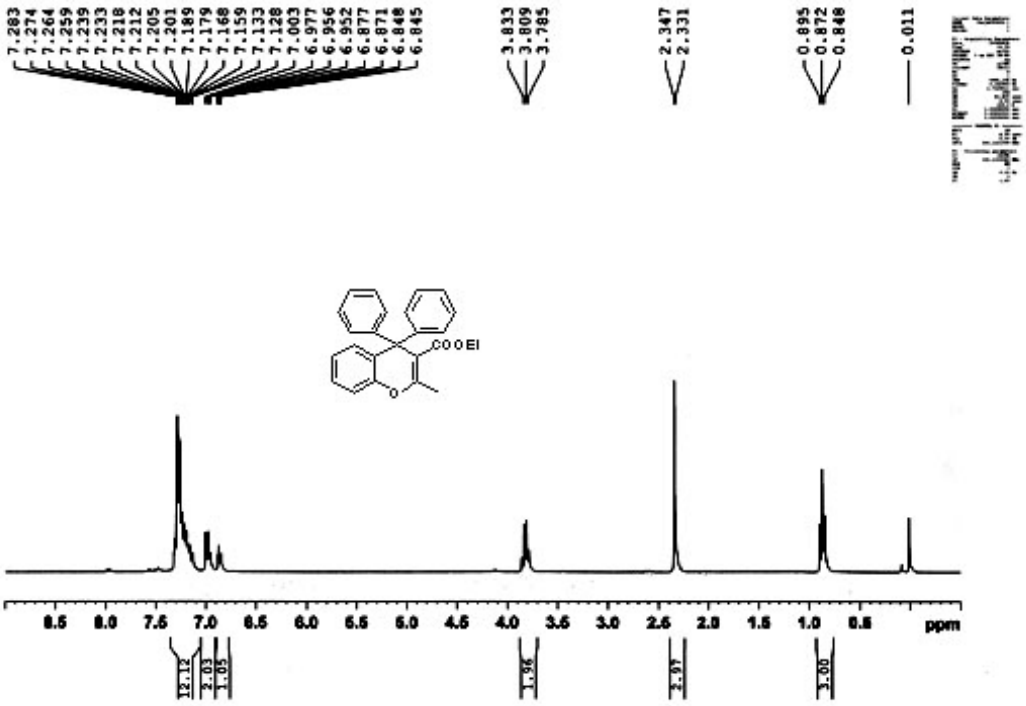
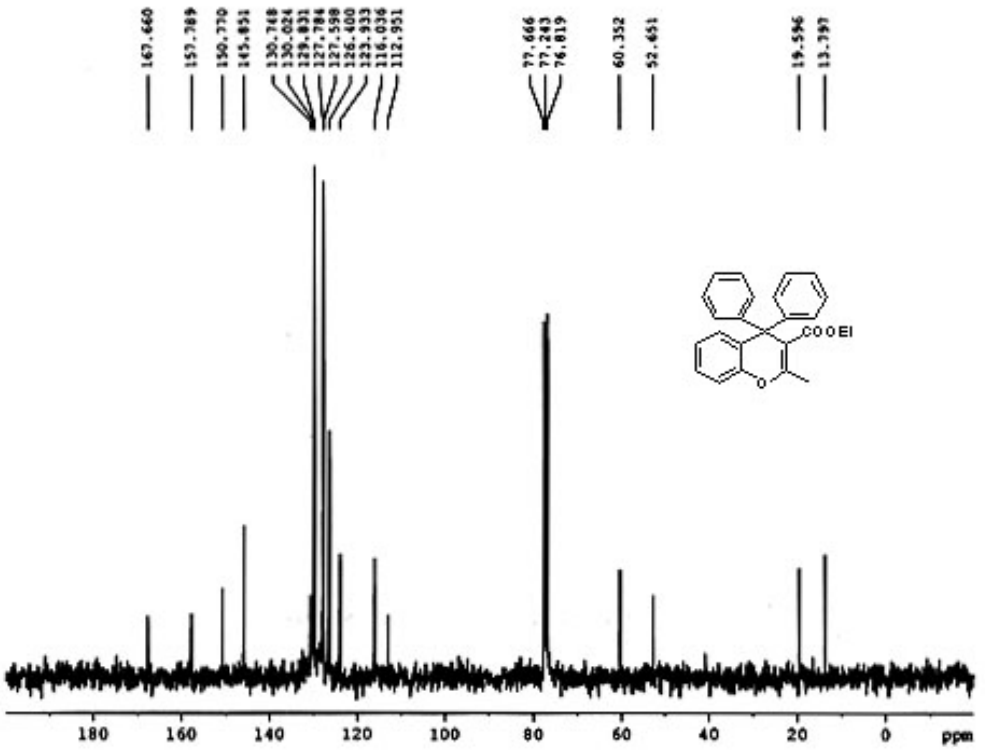
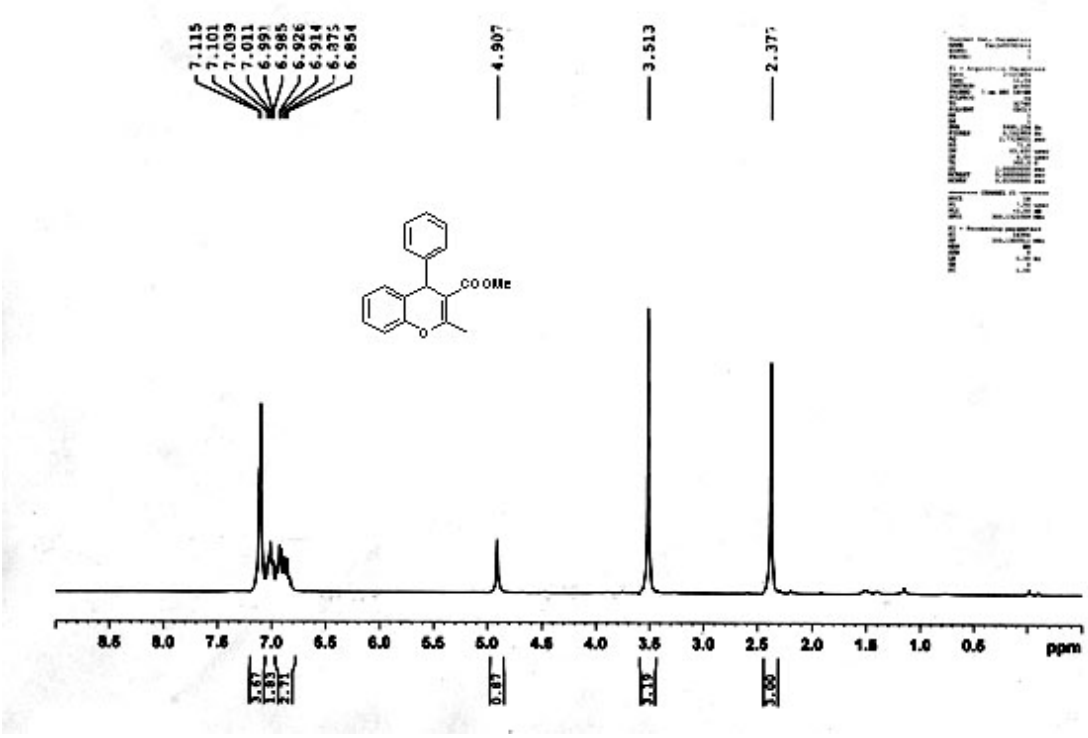
145.501
139.644
130.593
129.619
128.835
127.791
127.203
120.615
116.799
116.023
77.594
77.171
76.747
OH
180
160
140
120
100
80
60
40
20
0
ppm

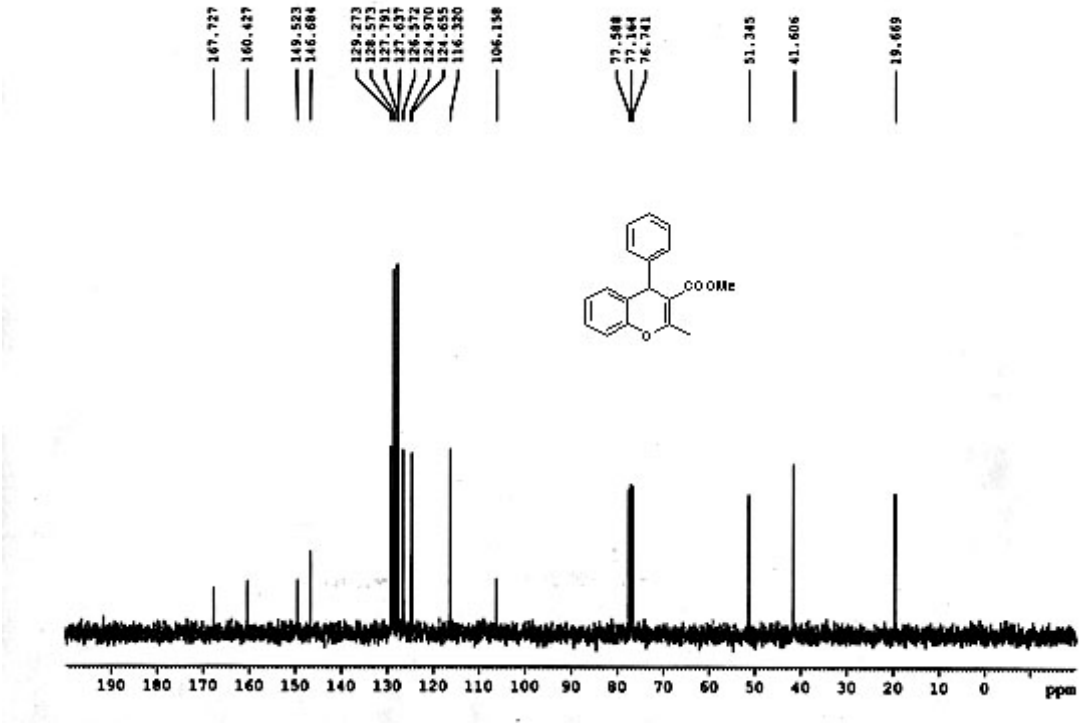
3l


7.283
7.274
7.264
7.259
7.239
7.233
7.218
7.212
7.205
7.201
7.189
7.179
7.168
7.159
7.133
7.128
7.003
6.977
6.956
6.952
6.877
6.871
6.848
6.845
3.833
3.809
3.785
2.347
2.331
0.895
0.872
0.848
0.011
COOEt
O
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
ppm
12.12
2.03
1.05
1.96
2.07
3.00

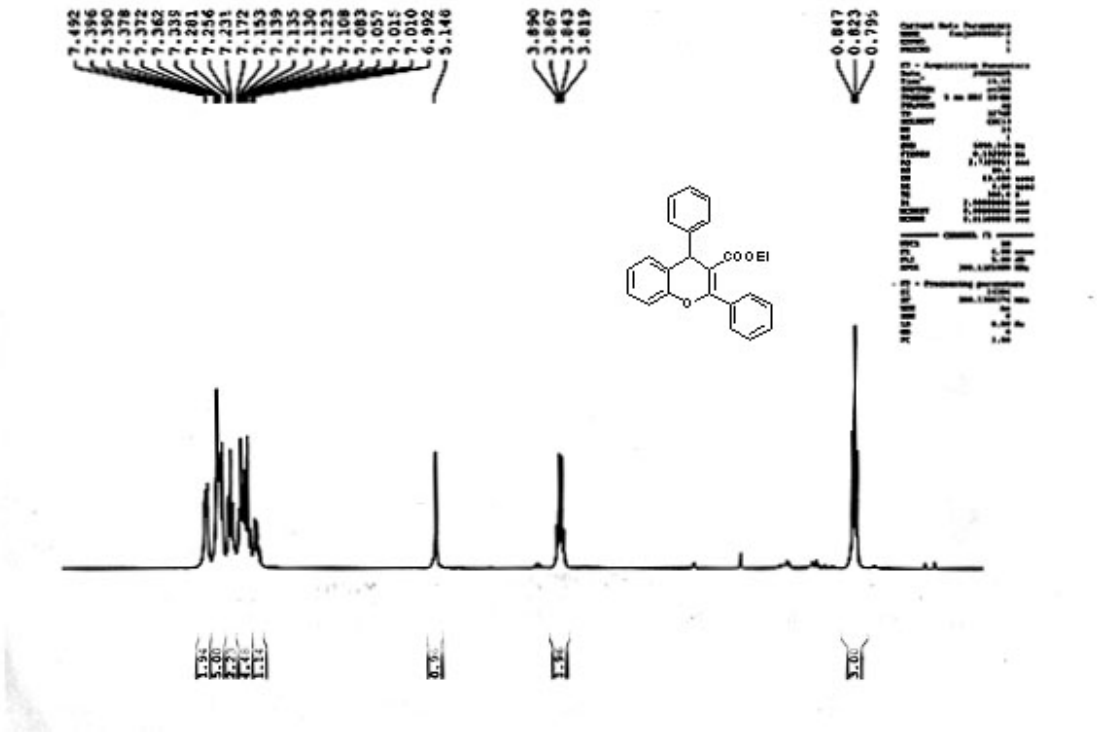

167.660
153.789
150.770
145.851
130.748
130.024
129.831
127.784
127.598
126.400
123.933
116.036
112.951
77.666
77.243
76.819
60.352
52.651
19.596
13.797
COOEt
O
180
160
140
120
100
80
60
40
20
0
ppm

3m


7.115
7.101
7.039
7.011
6.991
6.985
6.926
6.914
6.375
6.854
4.907
3.513
2.377
COOMe
O
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
ppm


167.727
160.427
149.523
146.684
129.273
128.573
127.791
127.637
126.572
124.970
124.655
116.320
106.158
77.588
77.164
76.741
51.345
41.606
19.669
COOMe
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

3n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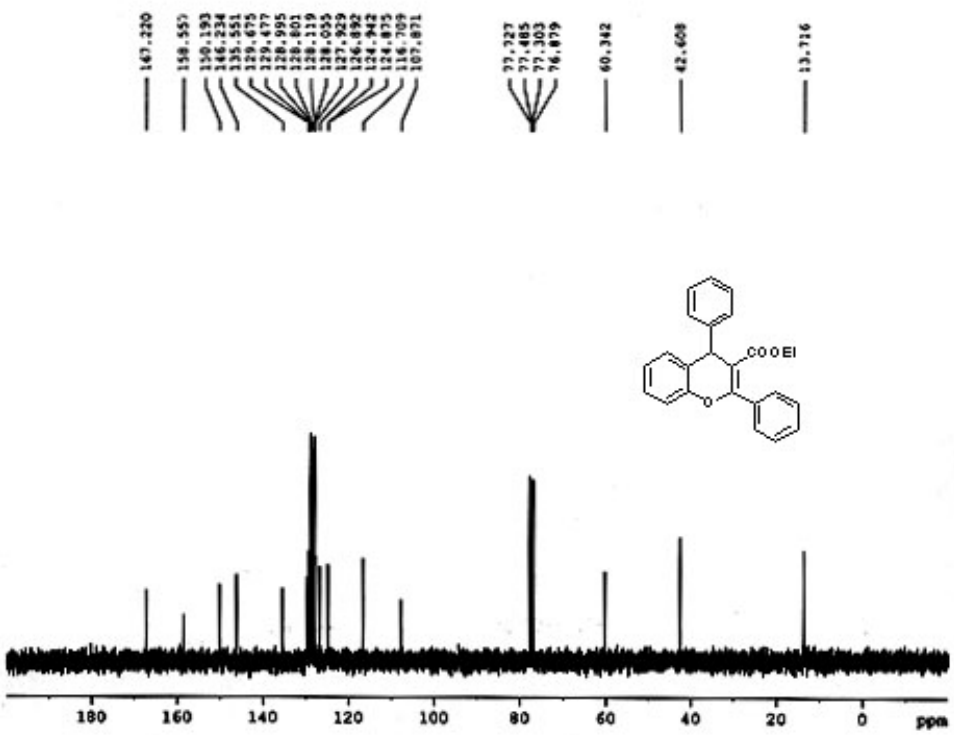
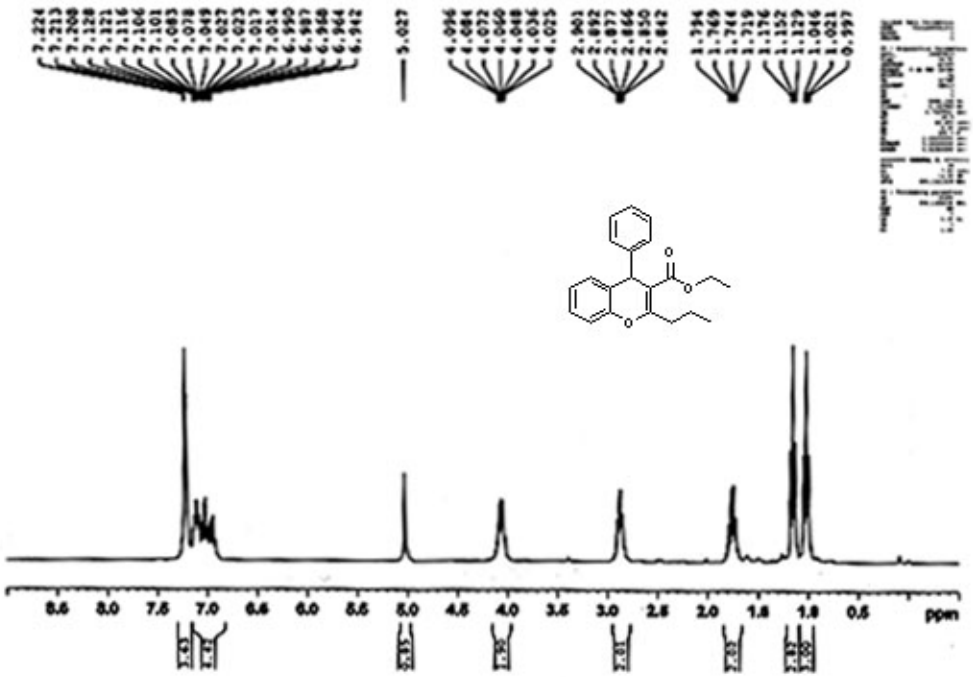
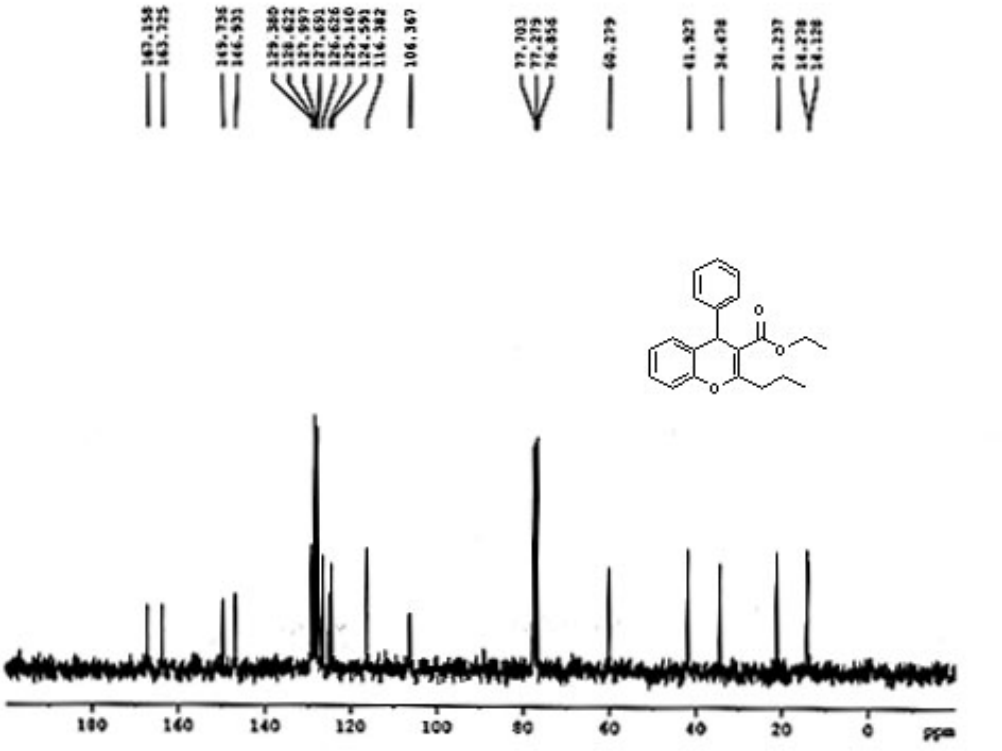
7.396
7.390
7.378
7.372
7.362
7.335
7.281
7.256
7.231
7.172
7.153
7.139
7.135
7.130
7.123
7.108
7.083
7.057
7.015
7.010
6.992
5.146
3.890
3.867
3.843
3.819
0.847
0.823
0.795
COOEt

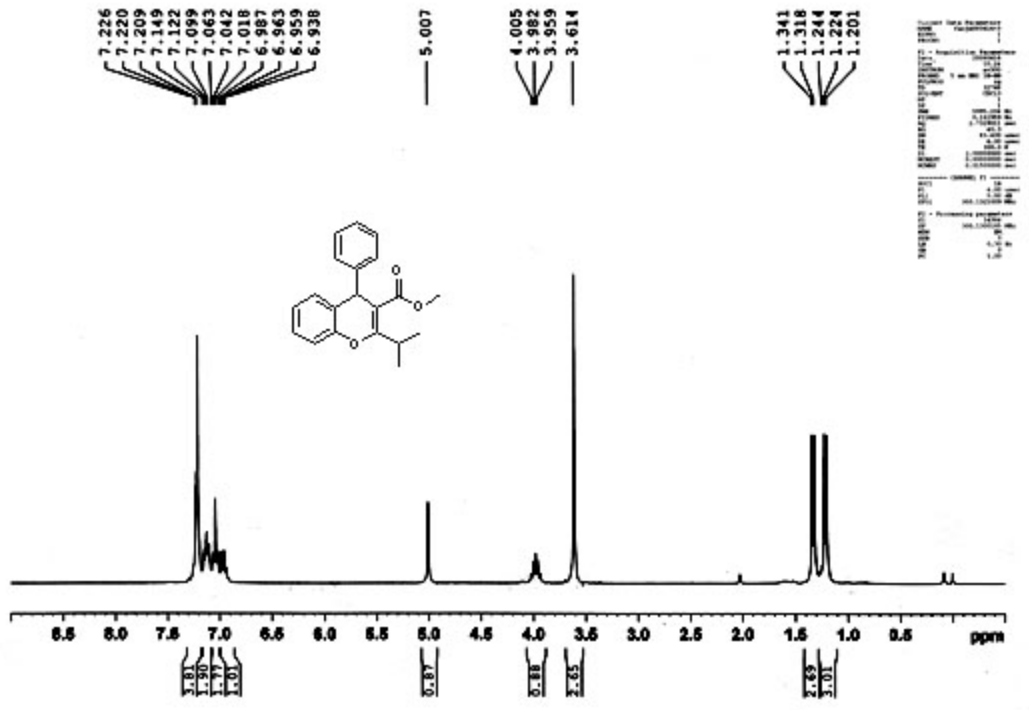


3o




167.158
163.725
145.736
146.933
129.380
128.622
127.997
127.651
126.626
125.140
124.593
116.382
104.367
77.703
77.279
76.856
68.279
41.927
34.478
21.237
14.298
14.128
180
160
140
120
100
80
60
40
20
0
ppm

3p


7.226
7.220
7.209
7.149
7.122
7.099
7.063
7.042
7.018
6.987
6.963
6.959
6.938
5.007
4.005
3.982
3.959
3.614
1.341
1.318
1.244
1.224
1.201
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
ppm
3.01
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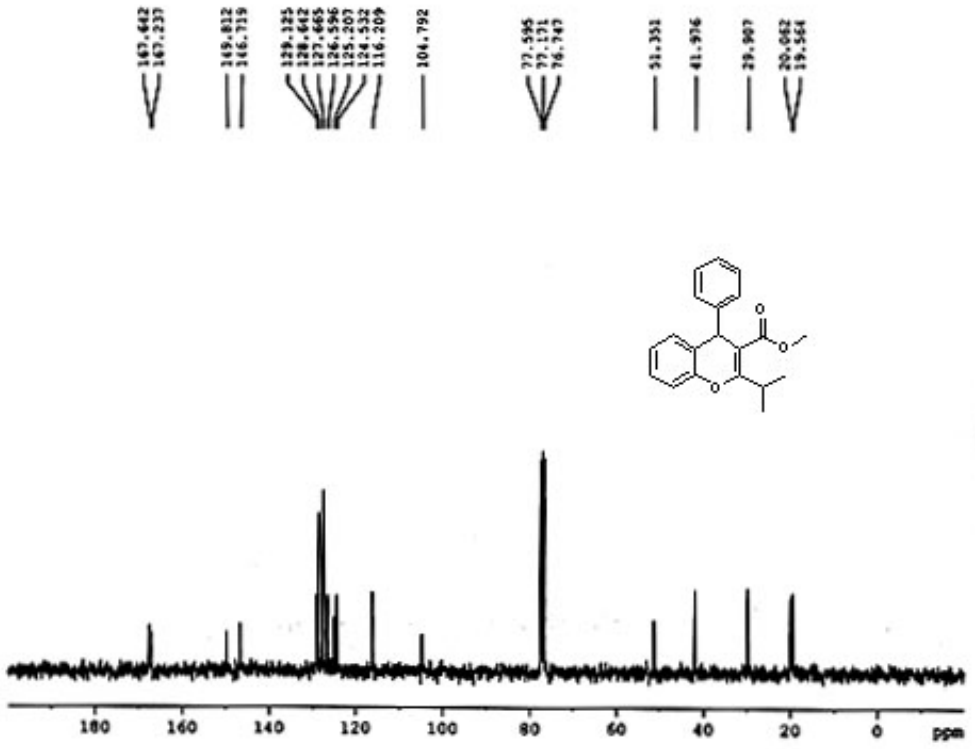
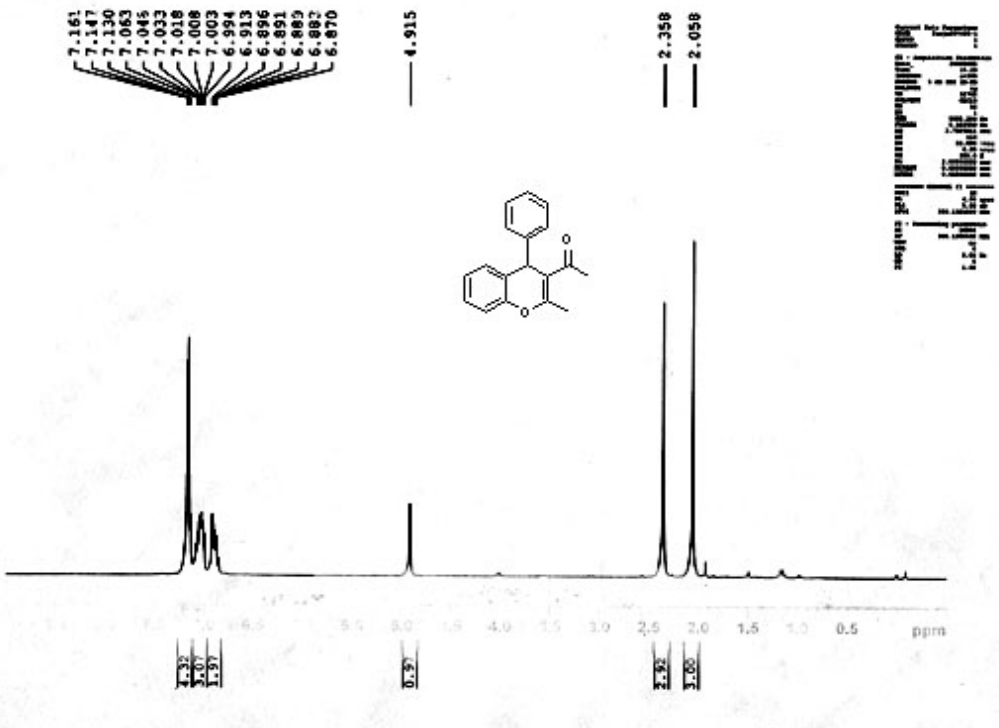
1.77
1.01
0.87
0.88
2.85
2.69
3.01


167.442
167.239
149.832
146.719
129.325
128.642
127.665
126.596
125.209
124.552
116.269
104.792
77.595
77.171
76.747
51.351
41.976
29.907
20.662
19.564
O
O
180
160
140
120
100
80
60
40
20
0
ppm

3q


7.161
7.147
7.130
7.063
7.045
7.033
7.018
7.008
7.003
6.994
6.913
6.896
6.891
6.889
6.882
6.870
4.915
2.358
2.058
O
O
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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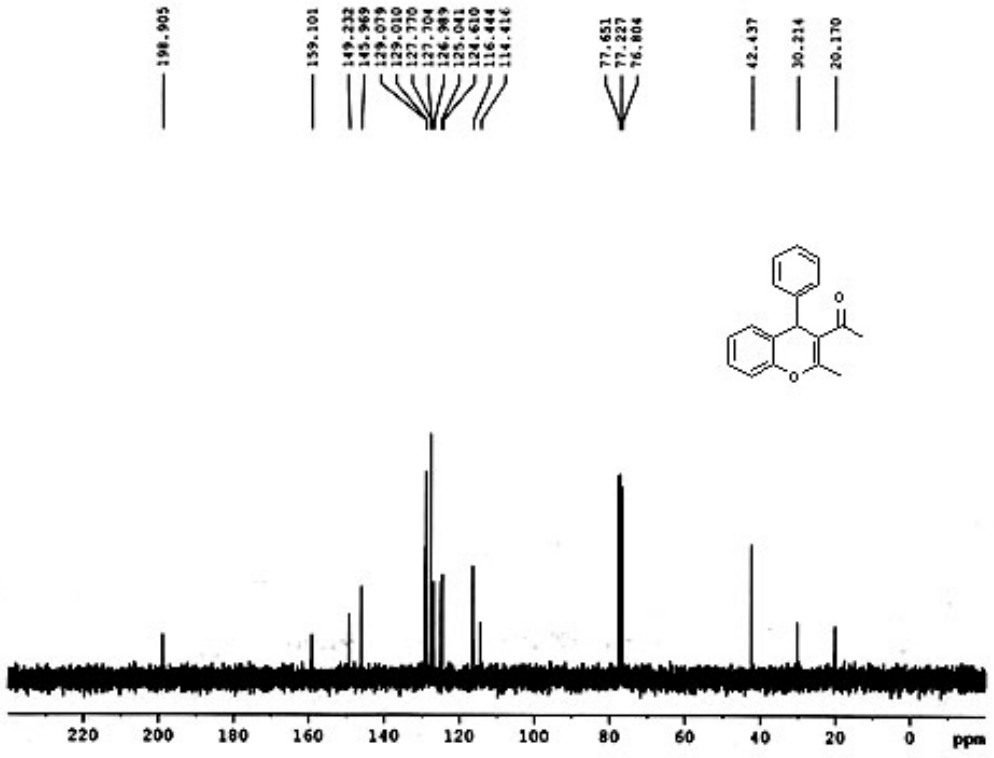
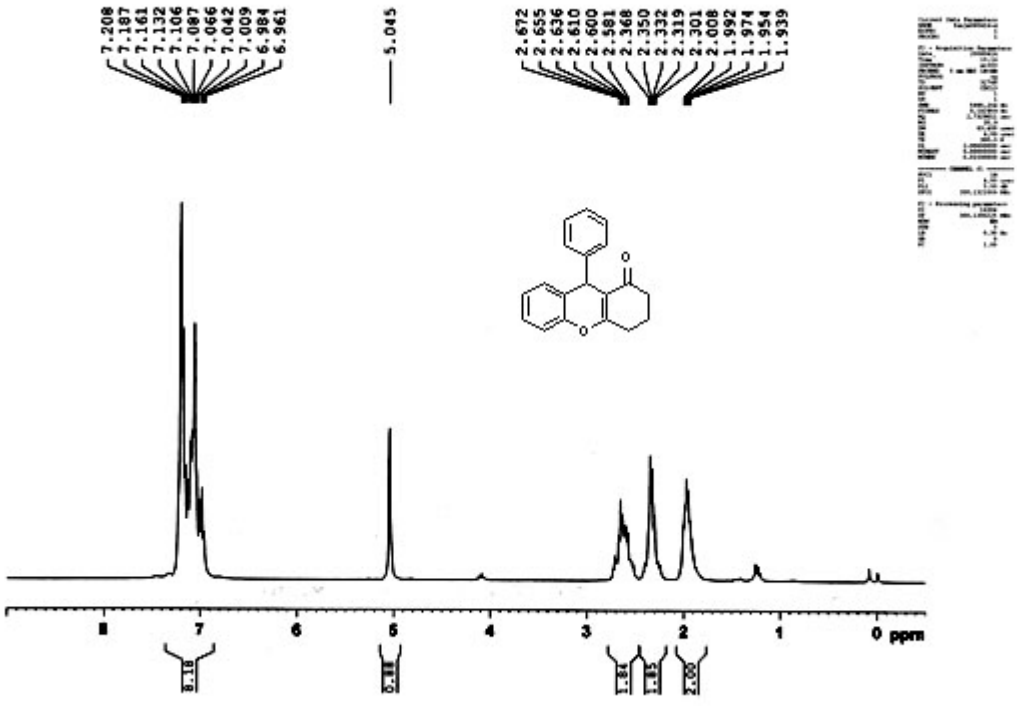
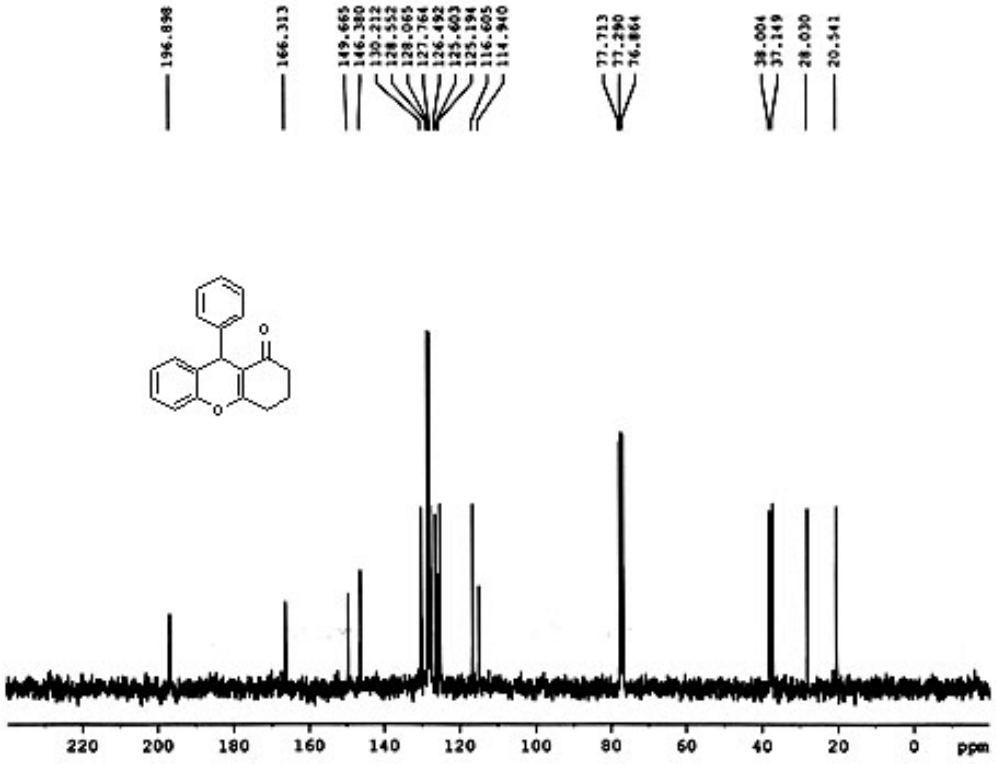
ppm
4.32
3.07
1.97
0.97
2.92
3.00

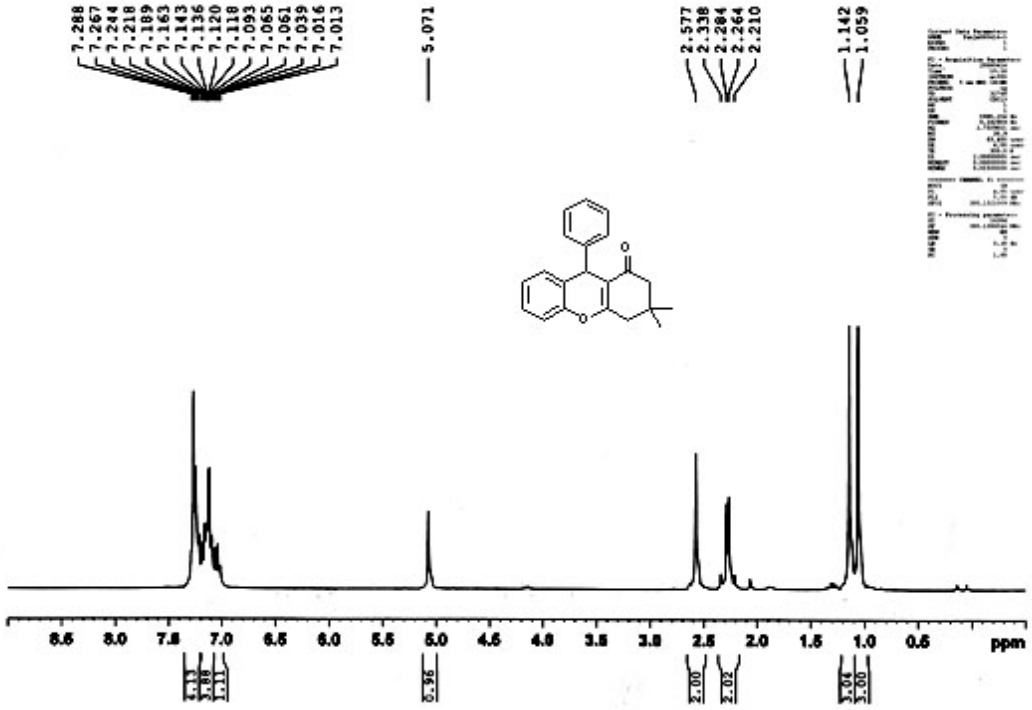

198.905
159.101
149.232
145.969
129.079
129.010
127.770
127.704
126.989
125.041
124.610
116.444
114.416
77.651
77.227
76.804
42.437
30.214
20.170
O
O
220
200
180
160
140
120
100
80
60
40
20
0
ppm

3r


7.208
7.187
7.161
7.132
7.106
7.087
7.066
7.042
7.009
6.984
6.961
5.045
2.672
2.655
2.636
2.610
2.600
2.581
2.368
2.350
2.332
2.319
2.301
2.008
1.992
1.974
1.954
1.939
O
O
8
7
6
5
4
3
2
1
0 ppm


196.898
166.313
149.665
146.380
130.212
128.552
128.065
127.764
126.492
125.603
125.194
116.605
114.940
77.713
77.290
76.864
38.004
37.149
28.030
20.541
O
O
220
200
180
160
140
120
100
80
60
40
20
0
ppm

3s


7.288
7.267
7.244
7.218
7.189
7.163
7.143
7.136
7.120
7.118
7.093
7.065
7.061
7.039
7.016
7.013
5.071
2.577
2.338
2.284
2.264
2.210
1.142
1.059
O
O
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
ppm
2.15
3.08
3.10
7.11
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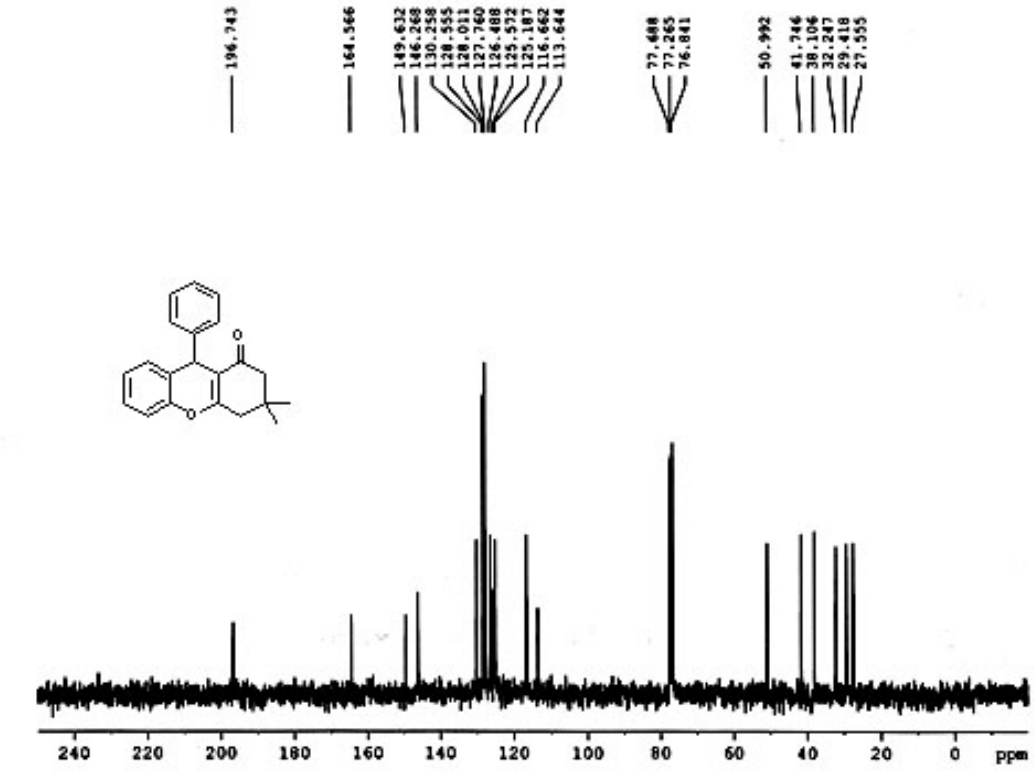
2.00
2.02
3.04
3.00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76.841
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41.746
38.104
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29.418
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O
O
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm