



Citation for published version:

Judd, KE & Caggiano, L 2011, 'Bi(OTf)₃-catalysed prenylation of electron-rich aryl ethers and phenols with isoprene: a direct route to prenylated derivatives', *Organic and Biomolecular Chemistry*, vol. 9, no. 14, pp. 5201-5210. <https://doi.org/10.1039/c1ob05365e>

DOI:

[10.1039/c1ob05365e](https://doi.org/10.1039/c1ob05365e)

Publication date:

2011

[Link to publication](#)

University of Bath

General rights

Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

Take down policy

If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.

Electronic Supporting Information

Bi(OTf)₃-catalysed prenylation of electron-rich aryl ethers and phenols with isoprene: a direct route to prenylated derivatives

Katie E. Judd and Lorenzo Caggiano*

General Experimental	S2
Synthetic Procedures	S3
Experimental References	S17
NMR Spectra of Compounds	S18

Experimental

General

Chemicals, solvents and reagents used are commercially available and were used without further purification. PE refers to petroleum ether, bp 40-60 °C. Anhydrous solvents were used where indicated. Glassware for dry reactions was dried either by heating in an oven at 120 °C for at least 1 h, or heating with a hot air gun for 5 min. The glassware was then allowed to cool under a stream of N₂.

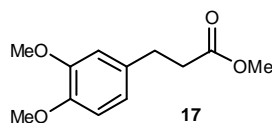
TLCs were carried out on Merck Aluminium backed TLC plates Silica Gel 60 F254 and viewed using UV light of wavelength 254 nm and then stained with potassium permanganate. Merck Silica Gel (0.040-0.063 mm) was used for column chromatography. Compounds were loaded as an oil, CH₂Cl₂ solution or dry loaded by adsorption onto silica.

Melting points were obtained using a Reichert-Jung heated-stage microscope. Infrared spectra were recorded on a Perkin-Elmer Spectrum RXI FT-IR system and all values are recorded in cm⁻¹.

NMR spectra were obtained on Varian Mercury VX (400 MHz) or Bruker Avance III (400 MHz) spectrometers. The chemical shifts are recorded in parts per million (ppm) with reference to tetramethylsilane. The coupling constants *J* are quoted to the nearest 0.5 Hz and are not corrected. The multiplicities are assigned as a singlet (s), doublet (d), triplet (t), quartet (q), doublet of doublets (dd), doublet of doublet of doublets (ddd), doublet of triplets (dt), triplet of doublets (td) and multiplet (m). Mass spectra and high resolution mass spectra were obtained on a micrOTOFTM from Bruker Daltonics (Bremen, Germany) coupled with an electrospray source (ESI-TOF) using an autosampler in an Agilent 1100 LC system. Data was processed using external calibration with the Bruker Daltonics software, DataAnalysisTM as part of the overall hardware control software, Compass 1.1TM.

Methyl 3,4,5-trimethoxybenzoate **9**, methyl *E*-3-(3,4,5-trimethoxyphenyl)propenoate **11**, methyl 3-(3,4,5-trimethoxyphenyl)propanoate **13**, methyl *E*-3-(3,4-dimethoxyphenyl)propenoate **15** and methyl 4-hydroxybenzoate **23** were all synthesised from their corresponding carboxylic acids following the procedure reported by Parrain¹ and gave samples that were consistent with the spectroscopic data reported for **9**,² **11**,¹ **13**,³ **15**⁴ and **23**.⁵

Methyl 3-(3,4-dimethoxyphenyl)propanoate (17)



10% Palladium on carbon (50 mg, 0.05 mmol) was added to a vigorously stirred solution of methyl 3,4-dimethoxycinnamate (819 mg, 3.69 mmol) in EtOH (20 mL). After 3 cycles of purging the flask with N₂ then a vacuum, the flask was put under an atmosphere of H₂. After 2 h, the mixture was filtered through Celite[®], washing thoroughly with EtOH, then the solvent removed under reduced pressure to afford the methyl propionate **17** (821 mg, 99%) as a colourless oil without need for further purification.

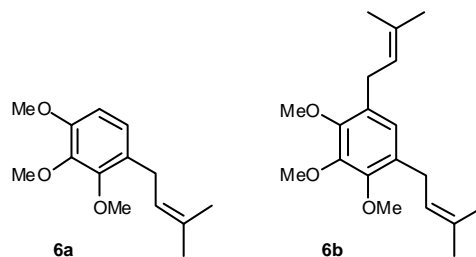
¹H NMR δ_H (400 MHz, CDCl₃) 6.84 (1H, d, *J* 8.5 Hz, C5-ArH), 6.77 (1H, dd, *J* 8.5 and 2.0 Hz, C6-ArH), 6.76 (1H, d, *J* 2.0 Hz, C2-ArH), 3.90 (3H, s, ArOMe), 3.88 (3H, s, ArOMe), 3.70 (3H, s, CO₂Me), 2.93 (2H, t, *J* 7.5 Hz, ArCH₂) and 2.64 (2H, t, *J* 7.5 Hz, CH₂CO₂Me); ¹³C NMR δ_C (100 MHz, CDCl₃) 173.4, 149.0, 147.6, 133.2, 120.1, 111.8, 111.4, 56.0, 55.9, 51.6, 36.0 and 30.6.

Consistent with the spectroscopic data previously reported.⁶

General procedure: Formation of propylated and chroman compounds

Bi(OTf)₃ (136 mg, 0.2 mmol) was added to a vigorously stirred solution of the arene (2 mmol) and isoprene (400 μl, 4 mmol) in anhydrous toluene (10 mL) in a thick-walled pressure tube at room temperature under Ar. The tube was sealed and the reaction heated at 40 °C for between 75 min and 24 h and the crude reaction mixture (often dark purple/black in colour) was applied directly to a silica gel chromatography column to afford the purified product(s). Representative reactions have also been performed in a conventional round-bottomed flask with a tightly fitted stopper to afford similar results.

1,2,3-Trimethoxy-4-(3-methylbut-2-en-1-yl)benzene (6a) and 2,3,4-trimethoxy-1,5-bis(3-methylbut-2-en-1-yl)benzene (6b)



Following the general procedure, 1,2,3-trimethoxybenzene **5** (336 mg, 2 mmol) gave, after 75 min and subsequent column chromatography [silica, PE-Et₂O gradient from 100:0 to 90:10], the mono-product **6a** (292 mg, 62%) and the bis-product **6b** (122 mg, 20%) as colourless oils.

Mono-product (6a)

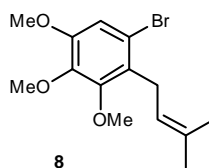
IR ν_{\max} (thin film) 2936, 1599, 1495, 1464, 1416, 1294, 1256, 1096 and 1017; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 6.86 (1H, d, *J* 8.5 Hz, ArH), 6.64 (1H, d, *J* 8.5 Hz, ArH), 5.28 (1H, triplet of septets, *J* 7.5 and 1.5 Hz, CH=CMe₂), 3.91 (3H, s, OMe), 3.90 (3H, s, OMe), 3.87 (3H, s, OMe), 3.31 (2H, d, *J* 7.5 Hz, ArCH₂), 1.77 (3H, br s, CH=CMe_AMe_B) and 1.77 (3H, br s, CH=CMe_AMe_B); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 152.0, 151.8, 142.4, 132.0, 127.9, 123.5, 123.3, 107.4, 60.7, 60.7, 56.0, 28.2, 25.7 and 17.7.

Consistent with the spectroscopic data previously reported.⁷

Bis-product (6b)

IR ν_{\max} (thin film) 2965, 2931, 1479, 1460, 1411, 1325, 1235, 1092, 1065 and 1015; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 6.67 (1H, s, ArH), 5.25 (2H, triplet of septets, *J* 7.0 and 1.5 Hz, CH=CMe₂), 3.91 (3H, s, OMe), 3.83 (6H, s, OMe), 3.27 (4H, d, *J* 7.0 Hz, ArCH₂) and 1.74 (12H, d, *J* 1.5 Hz, CH=CMe₂); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 149.8, 146.3, 132.0, 130.3, 124.2, 123.3, 60.8, 60.6, 28.4, 25.7 and 17.8; **MS** (+ESI) *m/z* 305 (MH⁺, 9%); **HRMS** (+ESI) Found MH⁺, 305.2103; C₁₉H₂₉O₃ requires MH⁺ 305.2117.

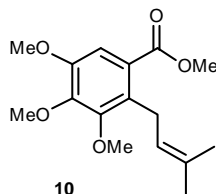
1-Bromo-3,4,5-trimethoxy-2-(3-methylbut-2-en-1-yl)benzene (8)



Following the general procedure, the aryl bromide **7** (494 mg, 2 mmol) gave, after 7 h and subsequent column chromatography [silica, PE-Et₂O gradient from 100:0 to 95:5], the product **8** (275 mg, 44%) as a colourless oil in addition to recovered starting material **7** (246 mg, 50%).

R_f [PE-Et₂O 70:30] 0.63; **IR** ν_{\max} (thin film) 2935, 1590, 1482, 1452, 1430, 1396, 1313, 1270, 1237, 1195, 1156, 1113, 1045 and 1019; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 6.87 (1H, s, ArH), 5.12 (1H, triplet of septets, *J* 7.0 and 1.5 Hz, CH=CMe₂), 3.84, (6H, s, OMe), 3.82 (3H, s, OMe), 3.42, (2H, d, *J* 7.0 Hz, ArCH₂), 1.79 (3H, br s, CH=CMe_AMe_B) and 1.68 (3H, d, *J* 1.0 Hz, CH=CMe_AMe_B); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 152.6, 152.1, 142.0, 131.8, 128.1, 122.1, 117.9, 112.0, 61.1, 60.7, 56.2, 29.3, 25.7 and 18.1; **MS** (+ESI) *m/z* 315 (MH⁺, 97%), 317 (MH⁺, 100) and 337 (MNa⁺, 23); **HRMS** (+ESI) Found MNa⁺, 337.0400; C₁₄H₁₉⁷⁹BrNaO₃ requires MNa⁺ 337.0415.

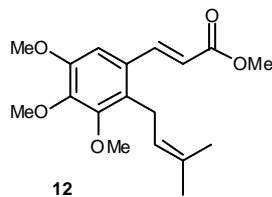
Methyl 3,4,5-trimethoxy-2-(3-methylbut-2-en-1-yl)benzoate (10)



Following the general procedure, ester **9** (452 mg, 2 mmol) gave, after 5 h and subsequent column chromatography [silica, PE-Et₂O gradient from 100:0 to 95:5], the product **10** (87 mg, 15%) as a colourless oil in addition to ester **9** (384 mg, 85%).

IR ν_{\max} (thin film) 2939, 1723 (C=O), 1594, 1491, 1455, 1431, 1401, 1337, 1222, 1154, 1115 and 1055; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 7.17 (1H, s, ArH), 5.12 (1H, triplet of septets, *J* 6.5 and 1.5 Hz, CH=CMe₂), 3.91 (3H, s, OMe), 3.86 (3H, s, OMe), 3.86 (3H, s, OMe), 3.82 (3H, s, OMe), 3.62 (2H, d, *J* 6.5 Hz, ArCH₂), 1.75 (3H, d, *J* 0.5 Hz, CH=CMe_AMe_B) and 1.66 (3H, d, *J* 1.0 Hz, CH=CMe_AMe_B); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 168.0, 152.3, 151.0, 145.7, 131.1, 130.7, 125.2, 123.8, 109.7, 61.0, 60.7, 56.1, 52.0, 25.9, 25.7 and 17.9; **MS** (+ESI) *m/z* 295 (MH⁺, 29%) and 317 (MNa⁺, 12); **HRMS** (+ESI) Found MH⁺, 295.1551; C₁₆H₂₃O₅ requires MH⁺ 295.1546.

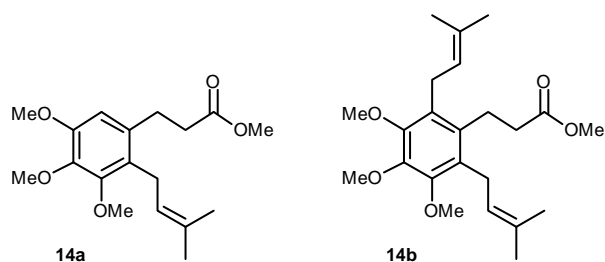
Methyl *E*-3-(3,4,5-trimethoxy-2-(3-methylbut-2-en-1-yl)phenyl)propenoate (12)



Following the general procedure, ester **11** (504 mg, 2 mmol) gave, after 4 h and subsequent column chromatography [silica, PE-Et₂O gradient from 100:0 to 90:10], the mono-product **12** (302 mg, 47%) as a colourless oil in addition to ester **11** (90 mg, 18%).

IR ν_{\max} (thin film) 2937, 1719 (C=O), 1631, 1592, 1566, 1487, 1409, 1347, 1289, 1254, 1168, 1124; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 7.92 (1H, d, *J* 15.5 Hz, CH=CHCO₂Me), 6.87 (1H, s, ArH), 6.26 (1H, d, *J* 15.5 Hz, CH=CHCO₂Me), 5.02 (1H, t, *J* 6.5 Hz, CH=CMe₂), 3.89 (3H, s, OMe), 3.86 (3H, s, OMe), 3.83 (3H, s, OMe), 3.79 (3H, s, OMe), 3.42 (2H, d, *J* 6.5 Hz, ArCH₂), 1.81 (3H, s, CH=CMe_AMe_B) and 1.67 (3H, s, CH=CMe_AMe_B); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 167.4, 151.9, 151.7, 144.2, 142.6, 131.6, 129.0, 128.6, 123.1, 118.0, 105.3, 61.0, 60.8, 55.9, 51.6, 25.7, 25.0 and 17.9; **MS** (+ESI) *m/z* 343 (MNa⁺, 11%); **HRMS** (+ESI) Found MNa⁺, 343.1508; C₁₈H₂₄NaO₅ requires MNa⁺ 343.1521.

Methyl 3-(3,4,5-trimethoxy-2-(3-methylbut-2-en-1-yl)phenyl)propanoate (14a) and methyl 3-(3,4,5-trimethoxy-2,6-bis(3-methylbut-2-en-1-yl)phenyl)propanoate (14b)



Following the general procedure, ester **13** (508 mg, 2 mmol) gave, after 90 min and subsequent column chromatography [silica, PE-Et₂O gradient from 100:0 to 80:20], the mono-product **14a** (376 mg, 58%) and the bis-product **14b** (233 mg, 30%) as colourless oils.

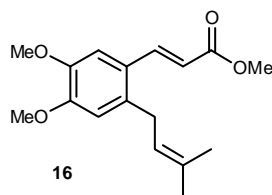
Mono-product (14a)

R_f [PE-Et₂O 80:20] 0.27; **IR** ν_{\max} (thin film) 2935, 1739 (C=O), 1599, 1578, 1494, 1453, 1406, 1338, 1283, 1239, 1196, 1121, 1073 and 1042; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 6.53 (1H, s, ArH), 5.08-5.04 (1H, m, CH=CMe₂), 3.87 (3H, s, OMe), 3.86 (3H, s, OMe), 3.84 (3H, s, OMe), 3.71 (3H, s, OMe), 3.33 (2H, d, *J* 6.5 Hz, ArCH₂CH=CMe₂), 2.93-2.89 (2H, m, ArCH₂CH₂CO₂Me) 2.60-2.56 (2H, m, CH₂CO₂Me), 1.79 (3H, br s, CH=CMe_AMe_B) and 1.71 (3H, d, *J* 1.0 Hz, CH=CMe_AMe_B); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 173.4, 152.1, 151.5, 140.9, 134.2, 131.1, 126.2, 123.8, 108.4, 60.9, 60.7, 56.0, 51.6, 35.5, 28.3, 25.6, 25.2 and 17.8; **MS** (+ESI) *m/z* 345 (MNa⁺, 24%); **HRMS** (+ESI) Found MNa⁺, 345.1661; C₁₈H₂₆NaO₅ requires MNa⁺ 345.1678.

Bis-product (14b)

R_f [PE-Et₂O 80:20] 0.51; **IR** ν_{\max} (thin film) 2948, 2933, 1740 (C=O), 1463, 1416, 1334, 1195, 1170, 1096, 1048 and 982; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 5.10-5.06 (2H, m, CH=CMe_AMe_B), 3.91 (3H, s, OMe), 3.85 (6H, s, OMe), 3.72 (3H, s, OMe), 3.36 (4H, d, *J* 6.5 Hz, ArCH₂CH=CMe₂), 2.93-2.89 (2H, m, ArCH₂CH₂CO₂Me), 2.50-2.46 (2H, m, CH₂CO₂Me), 1.79 (6H, d, *J* 1.0 Hz, CH=CMe_AMe_B) and 1.71 (6H, d, *J* 1.0 Hz, CH=CMe_AMe_B); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 173.4, 150.3, 144.8, 133.0, 131.1, 129.5, 124.0, 60.8, 60.4, 51.5, 34.9, 25.7, 25.6, 24.8 and 17.8; **MS** (+ESI) *m/z* 391 (MH⁺, 20%); **HRMS** (+ESI) Found MH⁺, 391.2652; C₂₃H₃₅O₅ requires MH⁺ 391.2485.

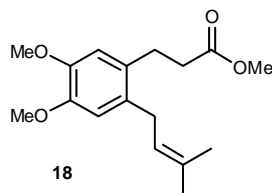
Methyl *E*-3-(4,5-dimethoxy-2-(3-methylbut-2-en-1-yl)phenyl)propenoate (16)



Following the general procedure, ester **15** (446 mg, 2 mmol) gave, after 6 h and subsequent column chromatography [silica, PE-Et₂O gradient from 100:0 to 90:10], the mono-product **16** (100 mg, 17%) as a colourless oil.

IR ν_{\max} (thin film) 2934, 1715 (C=O), 1602, 1514, 1458, 1268, 1167 and 1102; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 7.95 (1H, d, *J* 16.0 Hz, CH=CHCO₂Me), 7.05 (1H, s, ArH), 6.68 (1H, s, ArH), 6.24 (1H, d, *J* 16.0 Hz, CH=CHCO₂Me), 5.16 (1H, triplet of septets, *J* 7.0 and 1.5 Hz, CH=CMe₂), 3.87 (3H, s, OMe), 3.87 (3H, s, OMe), 3.78 (3H, s, OMe), 3.40 (2H, d, *J* 7.0 Hz, ArCH₂), 1.76 (3H, br s, CH=CMe_AMe_B) and 1.71 (3H, d, *J* 1.0 Hz, CH=CMe_AMe_B); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 167.7, 151.0, 147.6, 142.1, 135.5, 132.6, 125.0, 122.9, 116.3, 112.6, 109.0, 56.0, 55.9, 51.5, 31.8, 25.7 and 17.9; **MS** (+ESI) *m/z* 313 (MNa⁺, 9%); **HRMS** (+ESI) Found MNa⁺, 313.1424; C₁₇H₂₂NaO₄ requires MNa⁺ 313.1416.

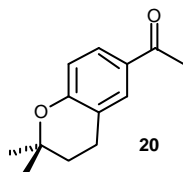
Methyl 3-(4,5-dimethoxy-2-(3-methylbut-2-en-1-yl)phenyl)propanoate (18)



Following the general procedure, ester **17** (448 mg, 2 mmol) gave, after 6 h and subsequent column chromatography [silica, PE-Et₂O gradient from 100:0 to 90:10], the mono-product **18** (376 mg, 64%) as a colourless oil.

R_f [PE-Et₂O 50:50] 0.32; **IR** ν_{\max} (thin film) 2934, 2851, 1737 (C=O), 1516, 1458, 1361, 1271, 1209 and 1093; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 6.69 (1H, s, ArH), 6.69 (1H, s, ArH), 5.21 (1H, triplet of septets, *J* 7.0 and 1.5 Hz, CH=CMe₂), 3.85 (3H, s, OMe), 3.85 (3H, s, OMe), 3.69 (3H, s, OMe), 3.29 (2H, d, *J* 7.0 Hz, ArCH₂CH=CMe₂), 2.92-2.88 (2H, m, ArCH₂CH₂CO₂Me), 2.59-2.55 (2H, m, CH₂CO₂Me), 1.75 (3H, br s, CH=CMe_AMe_B) and 1.74 (3H, br s, CH=CMe_AMe_B); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 173.4, 147.5, 147.2, 132.1, 131.7, 130.4, 123.4, 113.0, 112.7, 56.0, 55.9, 51.5, 35.5, 31.3, 27.8, 25.7 and 17.9; **MS** (+ESI) *m/z* 293 (MH⁺, 15%) and 315 (MNa⁺, 27); **HRMS** (+ESI) Found MH⁺, 293.1723; C₁₇H₂₅O₄ requires MH⁺ 293.1753.

1-(2,2-Dimethylchroman-6-yl)ethanone (20)

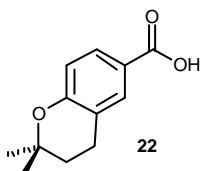


Following the general procedure, phenol **19** (272 mg, 2 mmol) gave, after 18 h and subsequent column chromatography [silica, PE-Et₂O-EtOAc gradient from 100:0:0 to 85:15:0 then 50:0:50], the chroman **20** (235 mg, 58%) as a white solid in addition to phenol **19** (62 mg, 23%).

R_f [PE-Et₂O 70:30] 0.63; **Mp** 89-93 °C (from CH₂Cl₂); **IR** ν_{\max} (thin film) 2976, 1670 (C=O), 1537, 1498, 1419, 1358, 1289, 1265, 1156 and 1117; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 7.75 (1H, d, *J* 2.5 Hz, C5-ArH), 7.73 (1H, dd, *J* 8.5 and 2.5 Hz, C7-ArH), 6.81 (1H, d, *J* 8.5 Hz, C8-ArH), 2.83 (2H, t, *J* 6.5 Hz, C4-CH₂), 2.54 (3H, s, COMe), 1.84 (2H, t, *J* 6.5 Hz, C3-CH₂) and 1.37 (6H, s, CMe₂); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 196.9, 158.6, 130.5, 129.4, 128.3, 120.7, 117.2, 75.5, 32.5, 26.9, 26.2 and 22.4.

Consistent with the spectroscopic data previously reported.⁸

2,2-Dimethylchroman-6-carboxylic acid (22)

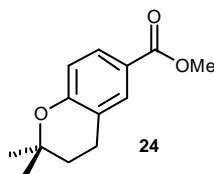


Following the general procedure, phenol **21** (276 mg, 2 mmol) gave, after 24 h and subsequent column chromatography [silica, PE-EtOAc gradient from 100:0 to 40:60], the chroman **22** (14 mg, 3%) as a white solid in addition to phenol **21** (254 mg, 92%).

IR ν_{\max} (thin film) 2975, 1681 (C=O), 1608, 1578, 1443, 1411, 1324, 1296, 1265, 1156 and 1120; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 7.86 (1H, d, *J* 2.0 Hz, C5-ArH), 7.84 (1H, dd, *J* 8.5 and 2.0 Hz, C7-ArH), 6.82 (1H, d, *J* 8.5 Hz, C8-ArH), 2.84 (2H, t, *J* 7.0 Hz, C4-CH₂), 1.84 (2H, t, *J* 7.0 Hz, C3-CH₂) and 1.36 (6H, s, CMe₂); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 172.1, 159.1, 132.4, 129.9, 120.8, 120.7, 117.4, 75.6, 32.5, 26.9 and 22.3; **MS** (+ESI) *m/z* 207 (MH⁺, 100%) and 229 (MNa⁺, 45); **HRMS** (+ESI) Found MH⁺, 207.1033; C₁₂H₁₅O₃ requires MH⁺ 207.1021.

Consistent with the spectroscopic data previously reported.⁹

Methyl 2,2-dimethylchroman-6-carboxylate (24)

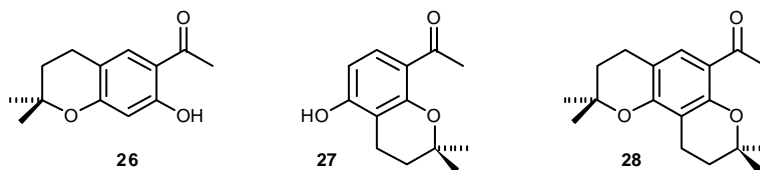


Following the general procedure, phenol **23** (304 mg, 2 mmol) gave, after 5 h and subsequent column chromatography [silica, PE-Et₂O gradient from 100:0 to 85:15] the chroman **24** (285 mg, 65%) as a white solid in addition to phenol **23** (32 mg, 11%).

R_f [PE-Et₂O 70:30] 0.61; **IR** ν_{\max} (thin film) 2975, 2948, 1716 (C=O), 1613, 1581, 1493, 1437, 1290, 1263, 1155 and 1118; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 7.78 (1H, d, *J* 2.0 Hz, C5-ArH), 7.76 (1H, dd, *J* 8.5 and 2.0 Hz, C7-ArH), 6.77 (1H, d, *J* 8.5 Hz, C8-ArH), 3.86 (3H, s, OMe), 2.80 (2H, t, *J* 7.0 Hz, C4-CH₂), 1.82 (2H, t, *J* 7.0 Hz, C3-CH₂) and 1.34 (6H, s, CMe₂); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 167.1, 158.3, 131.6, 129.1, 121.5, 120.6, 117.2, 75.3, 51.7, 32.6, 26.9 and 22.3.

Consistent with the spectroscopic data previously reported.¹⁰

1-(7-Hydroxy-2,2-dimethylchroman-6-yl)ethanone (26), **1-(5-hydroxy-2,2-dimethylchroman-8-yl)ethanone (27)** and **1-(2,2,8,8-tetramethyl-2,3,4,8,9,10-hexahydropyrano[2,3-f]chromen-6-yl)ethanone (28)**



Following general procedure, phenol **25** (304 mg, 2 mmol) gave, after 8 h and subsequent column chromatography [silica, PE-EtOAc gradient from 100:0 to 70:30], the chroman products **26** (105 mg, 24%) as a white solid, **27** (46 mg, 10%) as a white solid and **28** (77 mg, 13%) as a colourless oil.

Chroman (**26**)

Mp 115-118 °C (from EtOAc); lit.¹² 118-119 °C; **IR** ν_{\max} (thin film) 2937, 2957, 1867, 1647 (C=O), 1612, 1495, 1369, 1288, 1280, 1161, 1118, 1058 1020 and 885; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 12.30 (1H, s, OH), 7.41 (1H, s, C5-ArH), 6.28 (1H, s, C8-ArH), 2.71 (2H, t, *J* 7.0 Hz, C4-CH₂), 2.70 (3H, s, COMe), 1.80 (2H, t, *J* 7.0 Hz, C3-CH₂) and 1.33 (6H, s, CMe₂); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 202.3, 162.8, 161.4, 132.2, 113.9, 112.7, 104.6, 75.9, 32.7, 26.4, 26.1 and 21.7.

Consistent with the spectroscopic data previously reported.^{11,12}

Chroman (**27**)

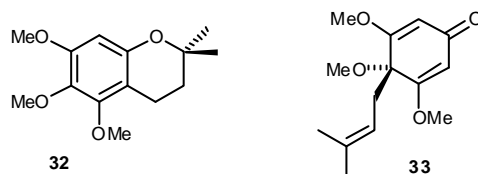
Mp 165-170 °C (from EtOAc); **IR** ν_{\max} (thin film) 3172, 2974, 2931, 1638 (C=O), 1583, 1433, 1362, 1277, 1217, 1157, 1119 and 1051; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 7.68 (1H, d, *J* 8.5 Hz, C7-ArH), 7.20 (1H, broad s, OH), 6.47 (1H, d, *J* 8.5 Hz, C6-ArH), 2.74 (2H, t, *J* 7.0 Hz, C4-CH₂), 2.64 (3H, s, COMe), 1.87 (2H, t, *J* 7.0 Hz, C3-CH₂) and 1.43 (6H, s, CMe₂); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 199.7, 159.2, 156.4, 129.9, 120.3, 108.5, 107.0, 75.3, 32.2, 31.6, 26.9 and 17.0.

Chroman (**28**)

IR ν_{\max} (thin film) 2974, 2933, 1662 (C=O), 1603, 1579, 1457, 1357, 1298, 1258, 1178, 1154, 1120 and 1096; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 7.48 (1H, s, C5-ArH), 2.70 (2H, t, *J* 7.0 Hz, C4-CH₂), 2.60 (2H, t, *J* 7.0 Hz, C10-CH₂), 2.56 (3H, s, COMe), 1.76 (2H, t, *J* 7.0 Hz, C9-CH₂), 1.76 (2H, t, *J* 7.0 Hz, C3-CH₂), 1.35 (6H, s, C(8)-CMe₂) and 1.32 (6H, s, C2-CMe₂); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 198.6, 156.2, 153.8, 129.2, 120.0, 111.9, 109.4, 75.3, 74.7, 32.8, 32.3, 31.8, 27.1, 26.8, 21.7 and 17.2.

Consistent with the spectroscopic data previously reported.¹²

5,6,7-Trimethoxy-2,2-dimethylchroman (32) and 3,4,5-trimethoxy-4-(3-methylbut-2-en-1-yl)-cyclohexa-2,5-dienone (33)



Following the general procedure, phenol **30** (368 mg, 2 mmol) gave, after 18 h and subsequent column chromatography [silica, PE-Et₂O gradient from 100:0 to 85:15 then PE-EtOAc gradient from 50:50 to 30:70], the chroman product **32** (279 mg, 55%) as a colourless oil and the dienone **33** (132 mg, 26%) as a white solid.

Chroman (32)

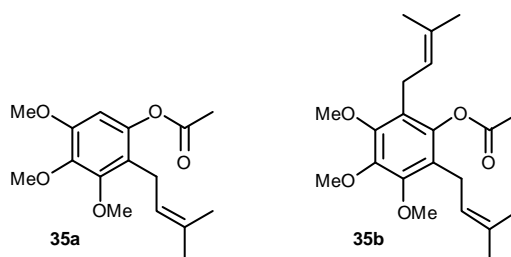
R_f [PE-Et₂O 80:20] 0.52; **IR** ν_{\max} (thin film) 2973, 2937, 1611, 1489, 1460, 1413, 1324, 1203, 1158, 1131, 1098, 1045 and 1013; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 6.15 (1H, s, C8-ArH), 3.87 (3H, s, C6-OMe), 3.79 (3H, s, C5-OMe), 3.78 (3H, s, C7-OMe), 2.63 (2H, t, *J* 7.0 Hz, C4-CH₂), 1.73 (2H, t, *J* 7.0 Hz, C3-CH₂), 1.30 (3H, s, C2-CMe_AMe_B) and 1.28 (3H, s, C2-CMe_AMe_B); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 152.4, 151.4, 150.0, 135.4, 106.7, 96.6, 74.0, 61.0, 60.5, 55.8, 32.4, 26.7, 26.7 and 17.0.

Consistent with the spectroscopic data previously reported.¹³

Dienone (33)

R_f [PE-EtOAc 40:60] 0.50; **Mp** 106-109 °C (from CH₂Cl₂); **IR** ν_{\max} (thin film) 2934, 2852, 1659 (C=O), 1625, 1597, 1459, 1374, 1240, 1215, 1163, 1078 and 888; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 5.56 (2H, s, C2/C6-CH), 4.66 (1H, triplet of septets, *J* 7.5 and 1.5 Hz, CH=CMe₂), 3.73 (6H, s, C3/C5-OMe), 3.08 (3H, s, C4-OMe), 2.67 (2H, d, *J* 7.5 Hz, CH₂CH=CMe₂), 1.56 (3H, br s, CH=CMe_AMe_B) and 1.52 (3H, br s, CH=CMe_AMe_B); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 187.3, 169.4, 136.3, 115.7, 104.3, 79.4, 56.0, 52.5, 35.6, 25.7 and 17.6; **MS** (+ESI) *m/z* 253 (MH⁺); **HRMS** (+ESI) Found MH⁺, 253.1427; C₁₄H₂₁O₄ requires MH⁺ 253.1440.

3,4,5-Trimethoxy-2-(3-methylbut-2-en-1-yl)-phenyl acetate (35a) and 3,4,5-trimethoxy-2,6-bis(3-methylbut-2-en-1-yl)phenyl acetate (35b)



Incorporating the procedure reported by Mohammadpoor-Baltork,¹⁴ Ac₂O (283 μ l, 3 mmol) was added to a rapidly stirred suspension of Bi(OTf)₃ (136 mg, 0.2 mmol) and phenol **30** (368 mg, 2 mmol) in anhydrous toluene (10 mL) in a thick-walled pressure tube at room temperature under Ar. After 5 min, isoprene (400 μ l, 4 mmol) was added to the solution and the tube was sealed and the reaction heated at 40 °C for 1 h. Column chromatography [silica, PE-Et₂O gradient from 100:0 to 90:10] gave the mono-prenylated product **35a** (414 mg, 70%) and the bis-prenylated product **35b** (108 mg, 15%) as colourless oils.

Mono-product (35a)

R_f [PE-Et₂O 80:20] 0.37; **IR** ν_{\max} (thin film) 2937, 1767 (C=O), 1608, 1490, 1456, 1408, 1368, 1339, 1206, 1122, 1075 and 1042; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 6.38 (1H, s, C6-ArH), 5.06 (1H, triplet of septets, *J* 7.0 and 1.5 Hz, CH=CMe₂), 3.85 (3H, s, OMe), 3.84 (3H, s, OMe), 3.80 (3H, s, OMe), 3.17 (2H, d, *J* 7.0 Hz, ArCH₂), 2.27 (3H, s, Ac), 1.73 (3H, br s, CH=CMe_AMe_B) and 1.66 (3H, d, *J* 1.0 Hz, CH=CMe_AMe_B); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 169.5, 152.3, 151.7, 144.5, 140.5, 131.3, 122.7, 120.1, 102.4, 61.0, 60.8, 56.0, 25.6, 23.5, 20.8 and 17.7; **MS** (+ESI) *m/z* 295 (MH⁺, 24%), 317 (MNa⁺, 20); **HRMS** (+ESI) Found MH⁺, 295.1533; C₁₆H₂₃O₅ requires MH⁺ 295.1546.

Bis-product (35b)

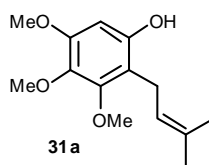
R_f [PE-Et₂O 80:20] 0.61; **IR** ν_{\max} (thin film) 2935, 1765 (C=O), 1600, 1463, 1416, 1367, 1346, 1204, 1097, 1048 and 982; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 5.08 (2H, triplet of septets, *J* 7.0 and 1.5 Hz, CH=CMe₂), 3.87 (3H, s, C4-OMe), 3.82 (6H, s, C3/5-OMe), 3.16 (4H, broad s, ArCH₂), 2.52 (3H, s, Ac), 1.73 (6H, d, *J* 1.0 Hz, CH=CMe_AMe_B) and 1.73 (6H, d, *J* 1.0 Hz, CH=CMe_AMe_B); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 169.3, 150.3, 144.8, 143.2, 131.3, 123.7, 122.7, 61.0, 60.6, 25.6, 24.1, 20.6 and 17.8; **MS** (+ESI) *m/z* 363 (MH⁺, 5%) and 385 (MNa⁺, 14); **HRMS** (+ESI) Found MNa⁺, 385.1975 C₂₁H₃₀NaO₅ requires MNa⁺ 385.1991.

General Procedure: Deacetylation of aromatic acetates

Method A: Following a procedure reported by Bates *et al.* but at a different concentration,¹⁵ K₂CO₃ (2 equiv.) was added to a solution of the acetate (1 equiv.) in MeOH (5 mL/mmol) at room temperature and the reaction was stirred for 2 h. The suspension was quenched with saturated aqueous NH₄Cl (30 mL) and extracted with EtOAc (3 × 15 mL). The combined organic fractions were washed with saturated brine (15 mL), dried over anhydrous Na₂SO₄, filtered and the solvent removed under reduced pressure. Column chromatography [silica, PE-EtOAc gradient from 100:1 to 75:25] afforded the phenol product.

Method B: Following a procedure reported by Narender *et al.*,¹⁶ NaOAc (10 equiv.) was added to a solution of the acetate (1 equiv.) in EtOH/H₂O (10:1, 5.5 mL/mmol) and the reaction heated at reflux for 5 h. After cooling, the reaction was diluted with H₂O (15 mL) and extracted with EtOAc (3 × 15 mL). The organic fractions were combined, washed with saturated brine (15 mL), dried over anhydrous Na₂SO₄, filtered and the solvent removed under reduced pressure. Column chromatography [silica, PE-EtOAc gradient from 100:1 to 75:25] afforded the phenol product.

3,4,5-Trimethoxy-2-(3-methylbut-2-en-1-yl)phenol (31a)

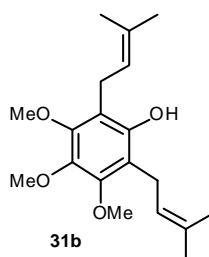


Following Method A, acetate **35a** (411 mg, 1.4 mmol) gave the phenol **31a** (173 mg, 49%) as a yellow amorphous solid. Following Method B, acetate **35a** (132 mg, 0.45 mmol) gave the phenol **31a** (41 mg, 36%) as a yellow amorphous solid, in addition to acetate **35a** (53 mg, 40%).

R_f [PE-EtOAc 75:25] 0.25; **IR** ν_{\max} (thin film) 3392, 2963, 1935, 1607, 1505, 1463, 1415, 1357, 1237, 1197, 1164, 1126, 1082, 1040 and 993; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 6.20 (1H, s, C6-ArH), 5.70 (1H, s, OH), 5.19 (1H, triplet of septets, *J* 7.0 and 1.5 Hz, CH=CMe₂), 3.83 (3H, s, OMe), 3.79 (3H, s, OMe), 3.72 (3H, s, OMe), 3.31 (2H, d, *J* 7.0 Hz, ArCH₂), 1.78 (3H, d, *J* 1.0 Hz, CH=CMe_AMe_B) and 1.70 (3H, d, *J* 1.0 Hz, CH=CMe_AMe_B); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 151.9, 151.9, 150.9, 136.1, 133.6, 122.6, 113.0, 96.6, 61.2, 61.0, 55.9, 25.7, 22.8 and 17.8; **MS** (+ESI) *m/z* 253 (MH⁺ 100%) and 275 (MNa⁺, 92); **HRMS** (+ESI) Found MNa⁺, 275.1272 C₁₄H₂₀NaO₄ requires MNa⁺ 275.1259.

Consistent with the spectroscopic data previously reported.¹⁷

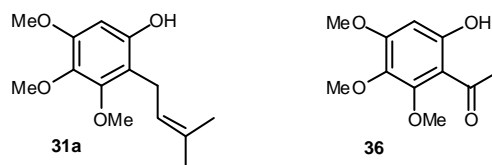
3,4,5-Trimethoxy-2,6-bis(3-methylbut-2-en-1-yl)phenol (31b)



Following Method A, acetate **35b** (100 mg, 0.27 mmol) gave the phenol **31b** (28 mg, 32%) as a colourless oil.

IR ν_{\max} (thin film) 3461, 2964, 2933, 1605, 1462, 1418, 1357, 1256, 1171, 1097, 1051 and 987; **¹H NMR** δ_{H} (400 MHz, CDCl₃) 5.59 (1H, s, OH), 5.21 (2H, triplet of septets, *J* 7.0 and 1.5 Hz, CH=CMe₂), 3.85 (3H, s, C4-OMe), 3.84 (6H, s, C3/5-OMe), 3.34 (4H, d, *J* 7.0 Hz, ArCH₂), 1.80 (6H, d, *J* 1.0 Hz, CH=CMe_AMe_B) and 1.72 (6H, d, *J* 1.0 Hz, CH=CMe_AMe_B); **¹³C NMR** δ_{C} (100 MHz, CDCl₃) 150.1, 149.2, 140.3, 133.4, 122.6, 116.8, 61.1, 60.9, 25.8, 23.1 and 17.8; **MS** (+ESI) *m/z* 321 (MH⁺, 50%); **HRMS** (+ESI) Found MH⁺, 321.2066; C₁₉H₂₉O₄ requires MH⁺ 321.2066.

3,4,5-Trimethoxy-2-(3-methylbut-2-en-1-yl)phenol (31a) and 1-(6-hydroxy-2,3,4-trimethoxyphenyl)ethanone (36)



Incorporating the procedure reported by Mohammadpoor-Baltork,¹⁴ Ac_2O (283 μl , 3 mmol) was added to a rapidly stirred suspension of $\text{Bi}(\text{OTf})_3$ (136 mg, 0.2 mmol) and phenol **30** (368 mg, 2 mmol) in anhydrous toluene (10 mL) in a thick-walled pressure tube at room temperature under Ar. After 5 min, isoprene (400 μl , 4 mmol) was added to the solution and the tube was sealed and the reaction heated at 40 °C for 4 h. The solvent was removed and following the procedure reported by Bates *et al.*,¹⁵ the residue was dissolved in MeOH (10 mL) and K_2CO_3 (552 mg, 4 mmol) was added. The reaction was stirred for 50 min at room temperature and then quenched with saturated aqueous NH_4Cl (30 mL) and extracted with CH_2Cl_2 (3 \times 30 mL). The combined organic fractions were washed with saturated brine (30 mL), dried over anhydrous Na_2SO_4 , filtered and the solvent removed under reduced pressure. Column chromatography [silica, PE-EtOAc gradient from 100:0 to 70:30] gave the mono-prenylated product **31a** (223 mg, 44%), consistent with the spectroscopic data reported, in addition to the acetophenone product **36** (39 mg, 9%) as colourless oils.

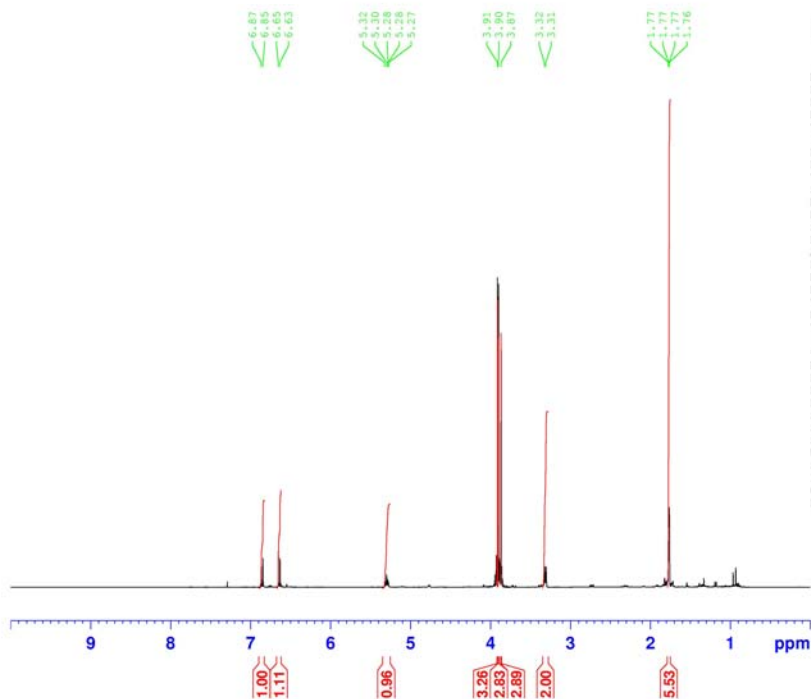
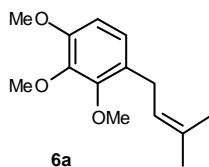
Acetophenone product (36)

$^1\text{H NMR}$ δ_{H} (400 MHz, CDCl_3) 13.39 (1H, s, OH), 6.22 (1H, s, C5-ArH), 3.97 (3H, s, OMe), 3.87 (3H, s, OMe), 3.76 (3H, s, OMe) and 2.63 (3H, s, COMe); $^{13}\text{C NMR}$ δ_{C} (100 MHz, CDCl_3) 203.3, 161.9, 160.1, 155.2, 134.8, 108.5, 96.1, 61.0, 60.9, 56.0 and 31.8.

Consistent with the spectroscopic data previously reported.¹⁸

References

- (1) J. Bourdrion, L. Commeiras, P. Barbier, V. Bourgarel-Rey, E. Pasquier, N. Vanthuynne, J.-C. Hubaud, V. Peyrot and J.-L. Parrain, *Bioorg. Med. Chem.*, 2006, **14**, 5540.
- (2) P. W. Elsinghorst, C. M. González Tanarro and M. Gütschow, *J. Med. Chem.*, 2006, **49**, 7540.
- (3) V. Kumar, A. Sharma and A. K. Sinha, *Helv. Chim. Acta*, 2006, **89**, 483.
- (4) A. El-Batta, C. Jiang, W. Zhao, R. Anness, A. L. Cooksy and M. Bergdahl, *J. Org. Chem.*, 2007, **72**, 5244.
- (5) R. Gopinath and B. K. Patel, *Org. Lett.*, 2000, **2**, 577.
- (6) D. d. L. Moreira, E. F. Guimarães and M. A. C. Kaplan, *Phytochemistry*, 2000, **55**, 783.
- (7) M. A. Tarselli, A. Liu and M. R. Gagné, *Tetrahedron*, 2009, **65**, 1785.
- (8) T. Narender, K. P. Reddy and Shweta, *Synth. Commun.*, 2009, **39**, 384.
- (9) M. O. Fatope and D. J. Abraham, *J. Med. Chem.*, 1987, **30**, 1973.
- (10) V. K. Ahluwalia, R. S. Jolly and A. K. Tehim, *Tetrahedron*, 1982, **38**, 3673.
- (11) Q. Wang, X. She, X. Ren, J. Ma and X. Pan, *Tetrahedron: Asymmetry*, 2004, **15**, 29.
- (12) V. K. Ahluwalia and K. K. Arora, *Tetrahedron*, 1981, **37**, 1437.
- (13) S. W. Youn, *Synlett*, 2007, 3050.
- (14) I. Mohammadpoor-Baltork, H. Aliyan and A. Reza Khosropour, *Tetrahedron*, 2001, **57**, 5851.
- (15) R. W. Bates, C. J. Gabel, J. Ji and T. Rama-Devi, *Tetrahedron*, 1995, **51**, 8199.
- (16) T. Narender, K. P. Reddy and G. Madhur, *Synth. Commun.*, 2009, **39**, 1949.
- (17) V. S. Parmar, S. Gupta, K. S. Bisht, S. Mukherjee, P. M. Boll and W. Errington, *Acta Chem. Scand.*, 1996, **50**, 558.
- (18) S. Combes, J.-P. Finet and D. Siri, *J. Chem. Soc., Perkin Trans. 1*, 2002, 38.



```

NAME      Apr20-2010-KEJ0748
EXPNO     10
PROCNO    1
Date_     20100420
Time      17.41
INSTRUM   AVIII400
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         32
DW         60.800 usec
DE         17.24 usec
TE         298.0 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      1H
P1         13.00 usec
PL1        0.00 dB
PL1W       9.74611950 W
SFO1      400.0424704 MHz
SI         65536
SF         400.0400000 MHz
WDW        EM
SSB        0
LB         0.20 Hz
GB         0
PC         1.00
    
```

MONOISOPRENYLTRIMETHOXYBENZENE

151.979
151.787
142.444
131.973
127.886
123.501
123.297

107.393

60.749
60.714
58.043

28.182
25.748

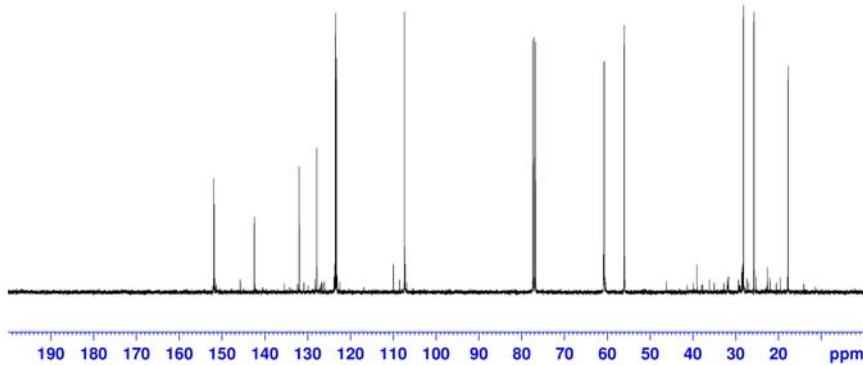
17.738

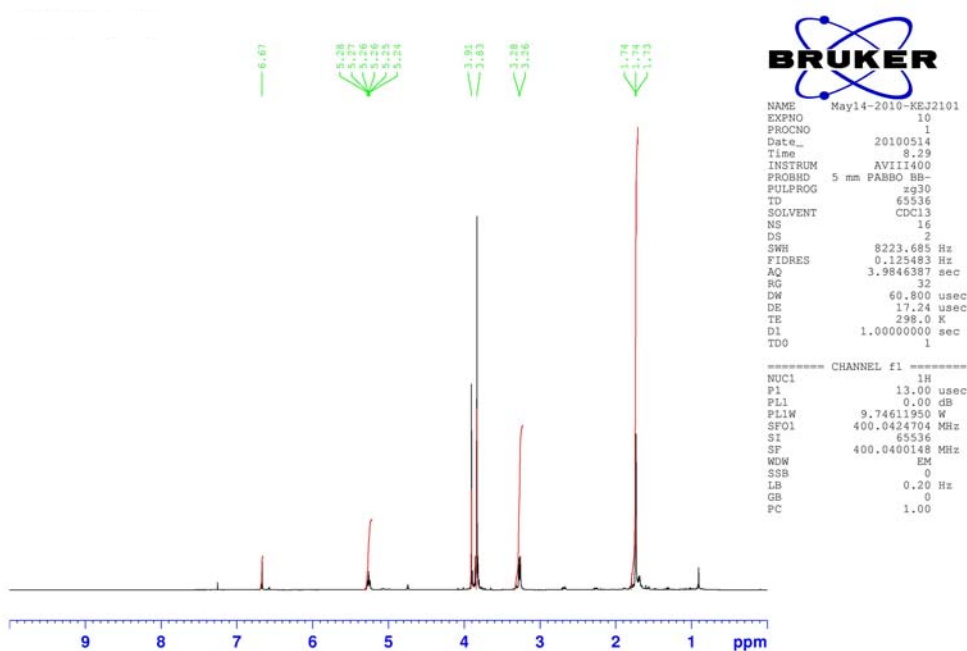
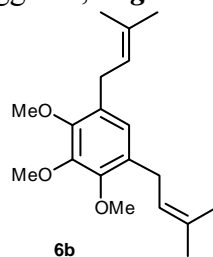
```

NAME      Apr20-2010-KEJ0748
EXPNO     12
PROCNO    1
Date_     20100420
Time      18.19
INSTRUM   AVIII400
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         4
DS         4
SWH        24038.461 Hz
FIDRES     0.368798 Hz
AQ         1.3631988 sec
RG         2050
DW         20.800 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.02000000 sec
TD0        1

===== CHANNEL F1 =====
NUC1      13C
P1         8.70 usec
PL1        -2.00 dB
PL1W       38.91986084 W
SFO1      100.6201970 MHz

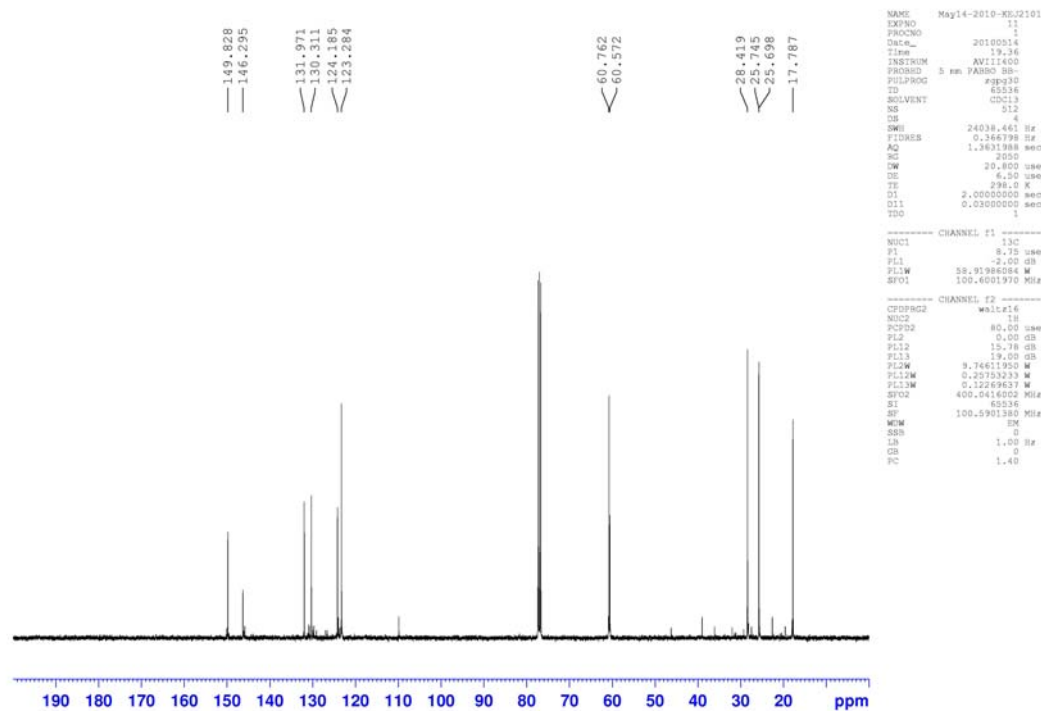
===== CHANNEL F2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2        0.00 dB
PL2W      15.78 dB
PL3        19.00 dB
PL3W      9.74611950 W
PL4W      0.2575323 W
PL5W      0.12269637 W
SFO2      400.0416000 MHz
SI         65536
SF         100.5901380 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```





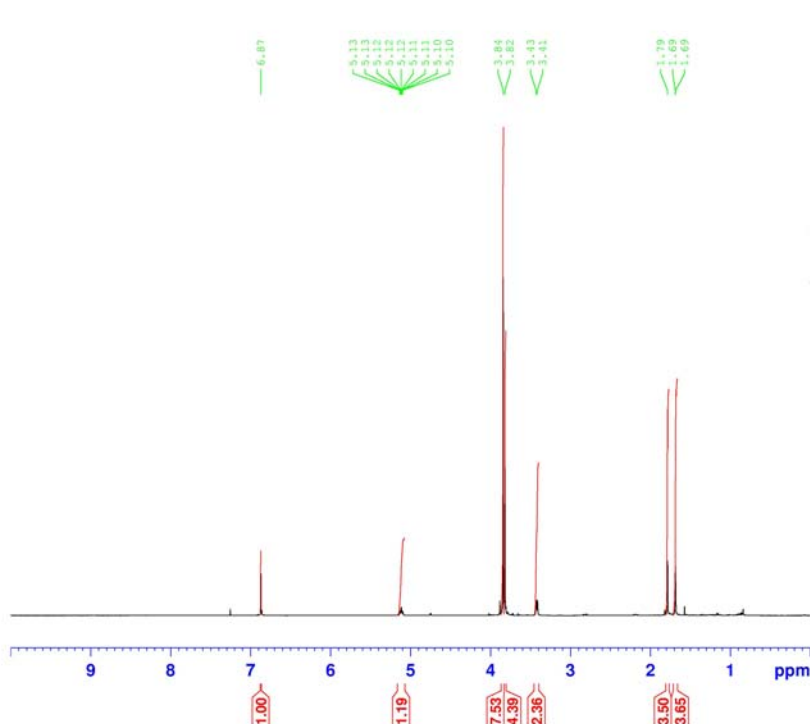
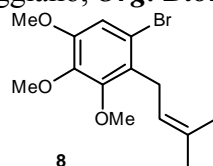
```

NAME May14-2010-KEJ2101
EXPNO 10
PROCNO 1
Date_ 20100514
Time 8.29
INSTRUM AVII1400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 32
DW 60.800 usec
DE 17.24 usec
TE 298.0 K
D1 1.0000000 sec
TDO 1
===== CHANNEL f1 =====
NUC1 1H
P1 13.00 usec
PL1 0.00 dB
PL1W 9.74611950 W
SFO1 400.0424704 MHz
SI 65536
SF 400.0400148 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
    
```



```

NAME May14-2010-KEJ2101
EXPNO 11
PROCNO 1
Date_ 20100514
Time 19.36
INSTRUM AVII1400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 312
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 4000
DW 20.800 usec
DE 4.250 usec
TE 298.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TDO 1
===== CHANNEL f1 =====
NUC1 13C
P1 8.750 usec
PL1 -2.00 dB
PL1W 58.9198606 W
SFO1 100.6001970 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL2W 15.78 GB
PL3 19.00 GB
PL3W 8.74611950 W
PL12W 0.29753233 W
PL13W 0.12269633 W
SFO2 400.0416002 MHz
SI 65536
SF 100.5901380 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
    
```



```

NAME Jun02-2010-KEJ2542
EXPNO 10
PROCNO 1
Date_ 20100602
Time 17.59
INSTRUM AVII1400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 71.8
DW 60.800 usec
DE 17.24 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
    
```

```

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 usec
PL1 0.00 dB
PL1W 9.74611950 W
SFO1 400.0424704 MHz
SI 65536
SF 400.0400148 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
    
```

152.575
152.139
142.051
131.803
128.127
122.052
117.919
112.008

61.078
60.726
56.184
29.314
25.724
18.076

```

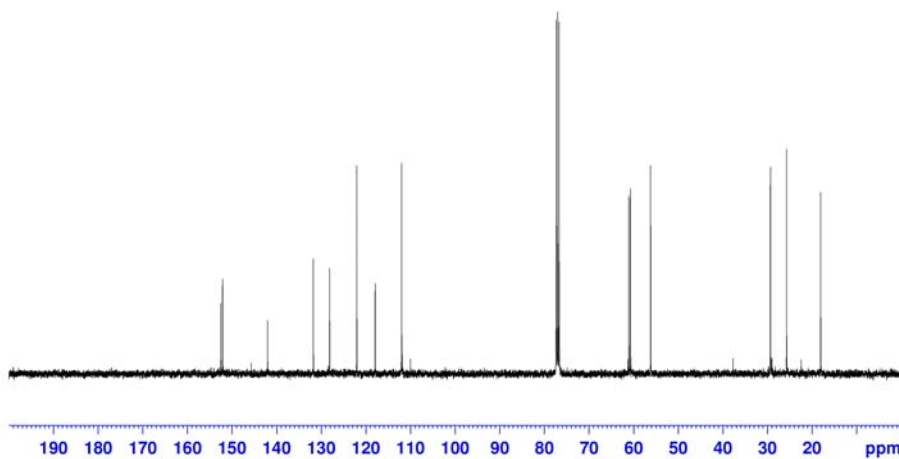
NAME Jun02-2010-KEJ2542
EXPNO 11
PROCNO 1
Date_ 20100603
Time 3.03
INSTRUM AVII1400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.346798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
    
```

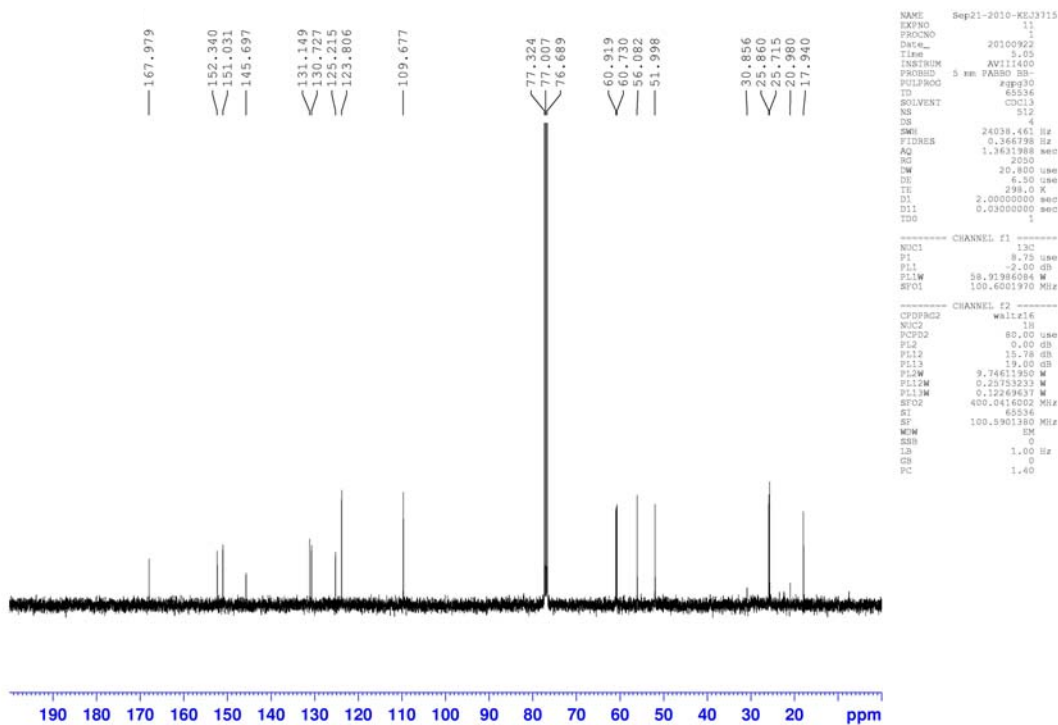
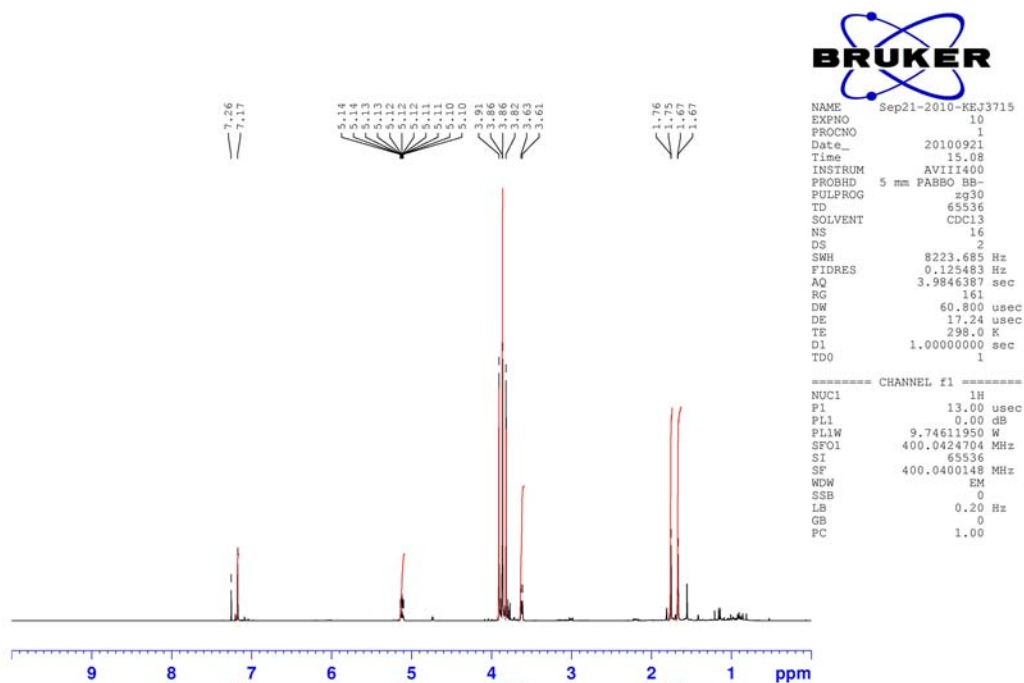
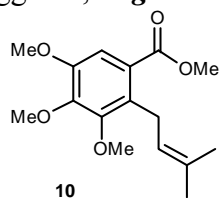
```

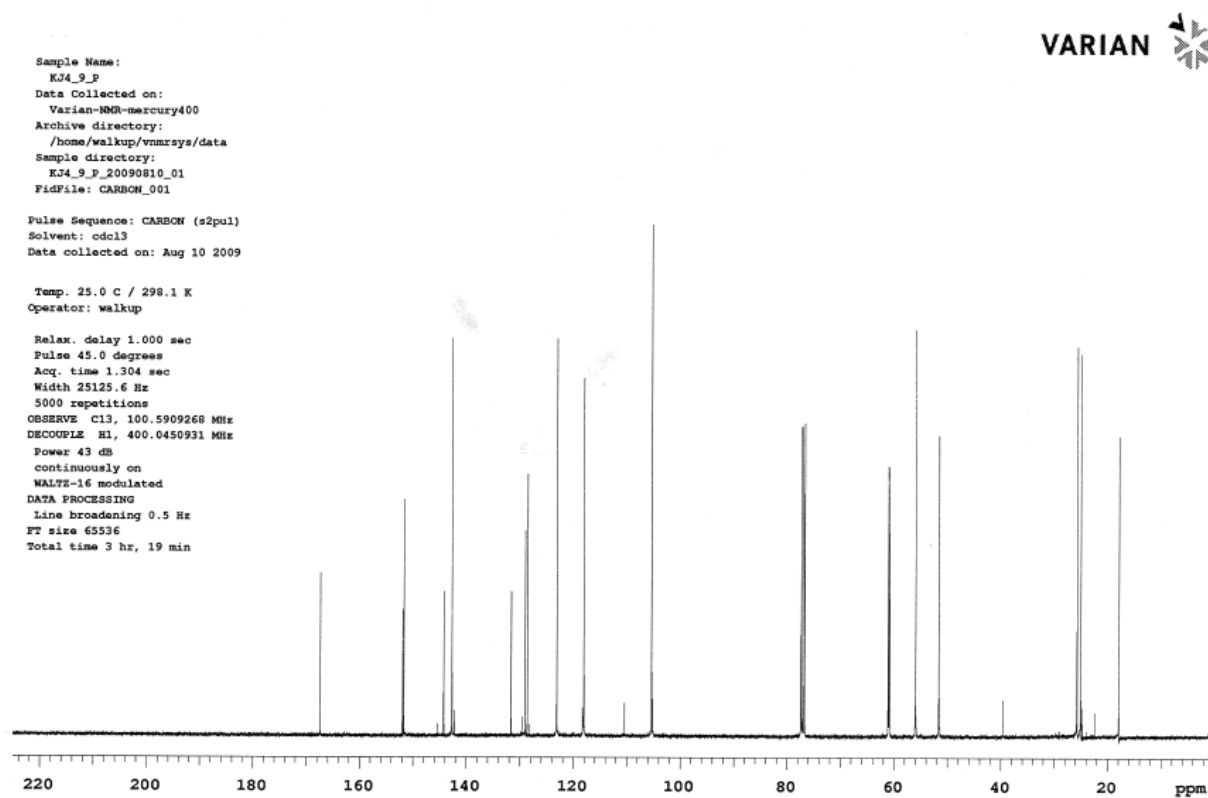
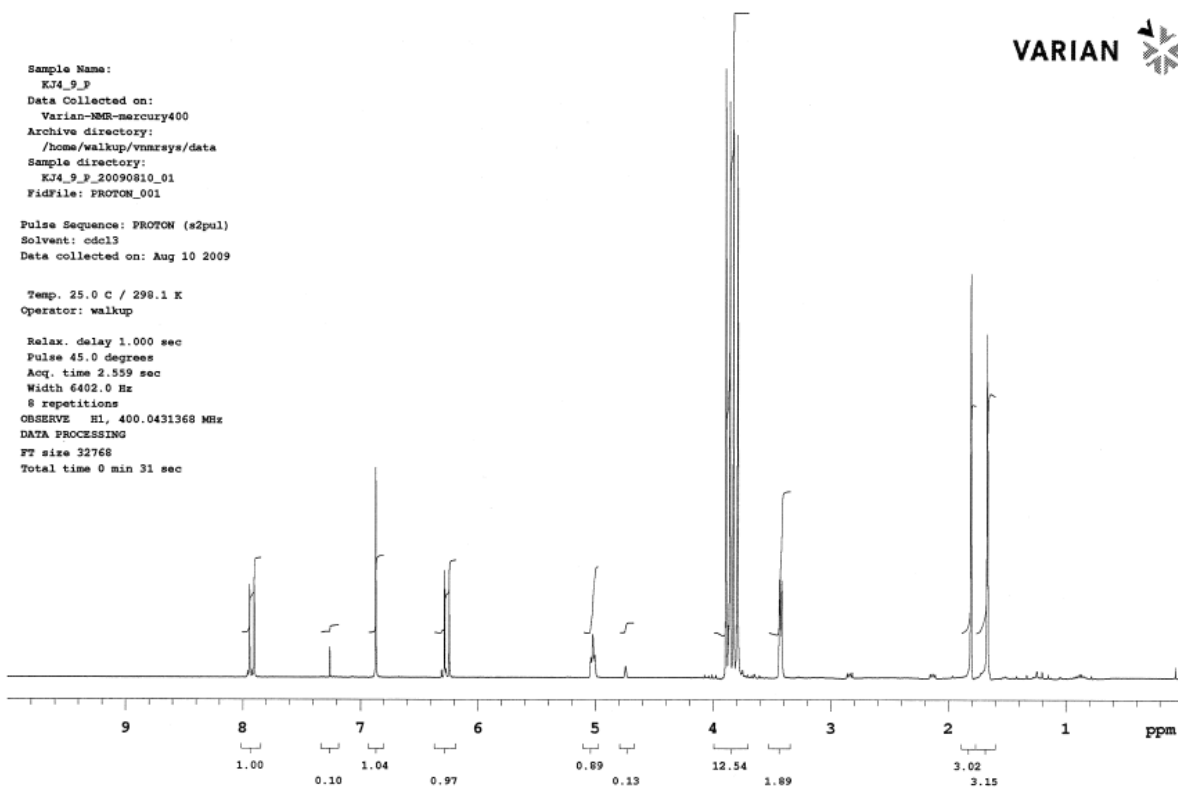
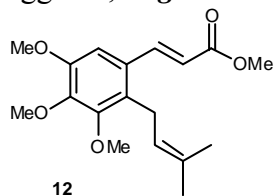
===== CHANNEL f1 =====
NUC1 13C
P1 8.75 usec
PL1 -2.00 dB
PL1W 58.91986084 W
SFO1 100.6001970 MHz
    
```

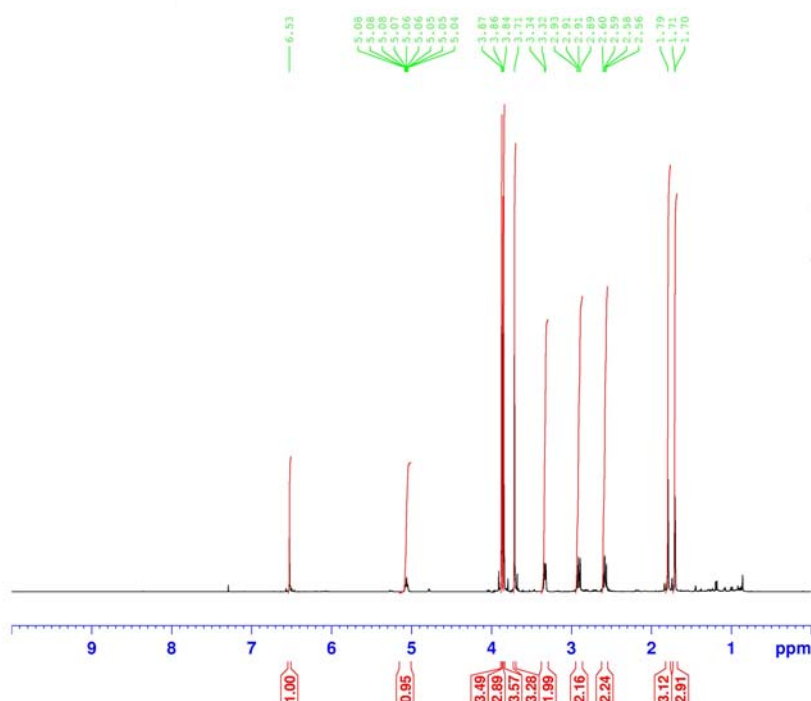
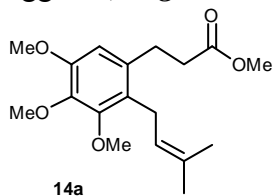
```

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL2 13.78 dB
PL13 19.00 dB
PL1W 9.74611950 W
PL12W 0.25753233 W
PL13W 0.12249637 W
SFO2 400.0436002 MHz
SI 65536
SF 100.5901360 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
    
```









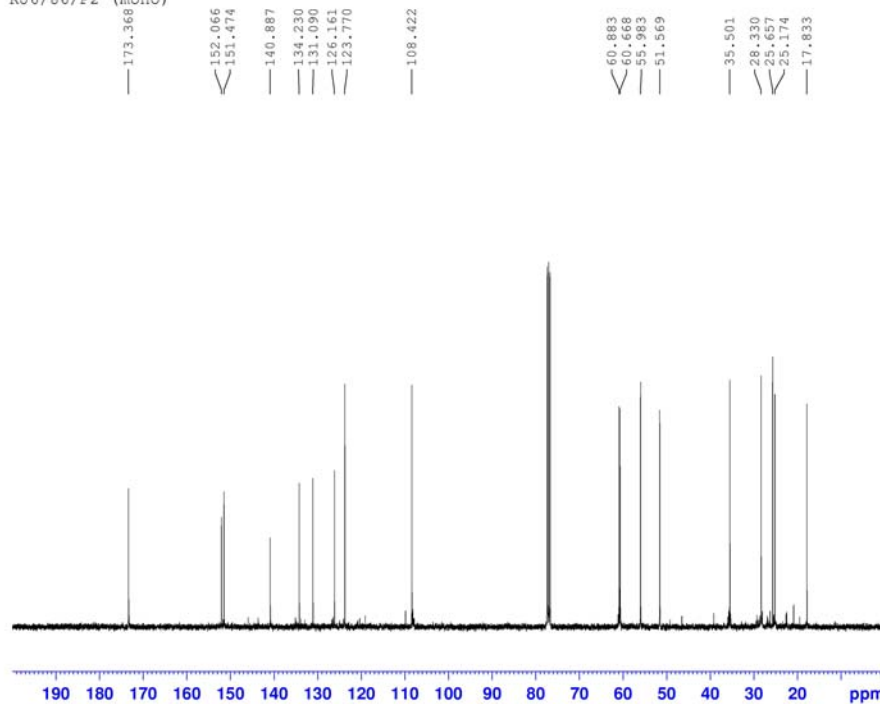
```

NAME      Apr26-2010-KEJ0887
EXPNO     10
PROCNO    1
Date_     20100427
Time      2.52
INSTRUM   AVII400
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         32
DW         60.800 usec
DE         17.24 usec
TE         298.0 K
D1         1.00000000 sec
TD0        1
    
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        13.00 usec
PL1       0.00 dB
PL1W      9.74611950 W
SFO1      400.0424704 MHz
SI        65536
SF        400.0400000 MHz
WDW       EM
SSB       0
LB        0.20 Hz
GB        0
PC        1.00
    
```

KJ6/86/P2 (mono)



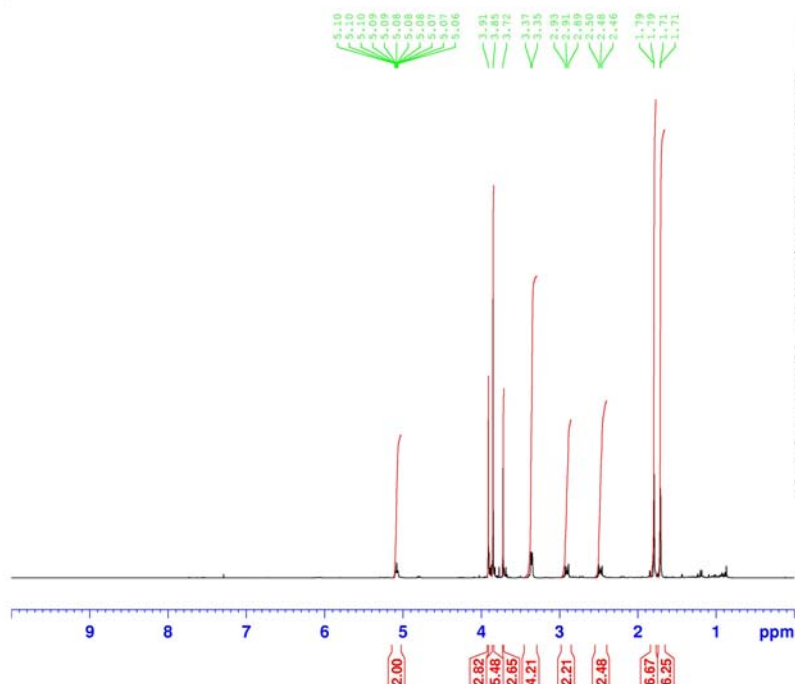
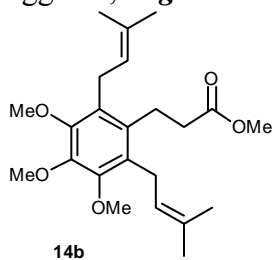
```

NAME      Apr26-2010-KEJ0887
EXPNO     11
PROCNO    1
Date_     20100427
Time      3.24
INSTRUM   AVII400
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         512
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.1631988 sec
RG         2000
DW         30.800 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
    
```

```

===== CHANNEL f1 =====
NUC1      13C
P1        8.75 usec
PL1       -2.00 dB
PL1W      58.91986084 W
SFO1      100.601970 MHz

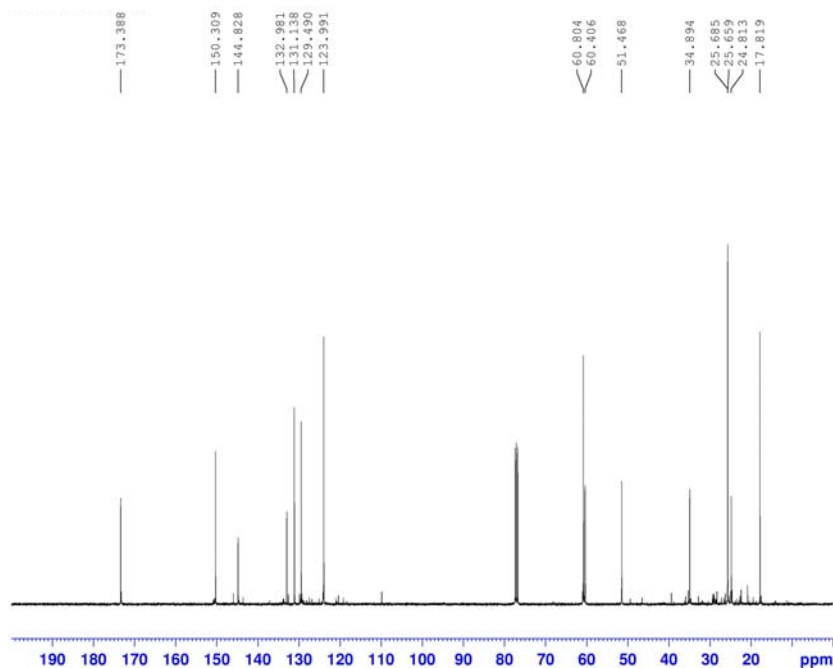
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       0.00 dB
PL12      15.78 dB
PL13      19.00 dB
PL1W      9.74611950 W
SFO1W     0.2373233 M
SFO13W    0.12269637 M
SFO2      400.0416002 MHz
SI        65536
SF        100.5901380 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```

```

NAME      Apr26-2010-KEJ0886
EXPNO     10
PROCNO    1
Date_     20100426
Time      23.32
INSTRUM   AVIII400
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         20.2
DW         60.800 usec
DE         17.24 usec
TE         298.0 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      1H
P1         13.00 usec
PL1        0.00 dB
PL1W       9.74611950 W
SFO1      400.0424704 MHz
SI         65536
SF         400.0400000 MHz
WDW        EM
SSB        0
LB         0.20 Hz
GB         0
PC         1.00
    
```

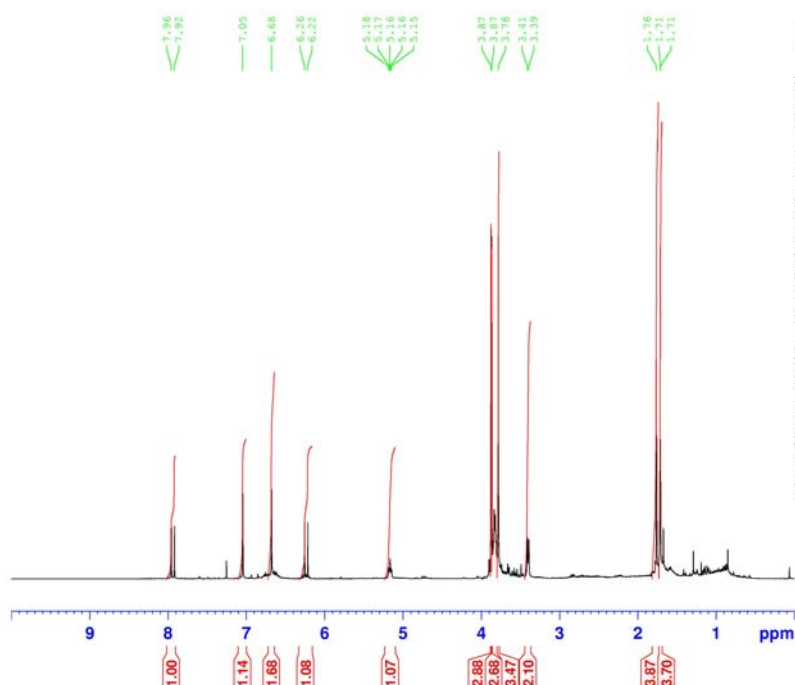
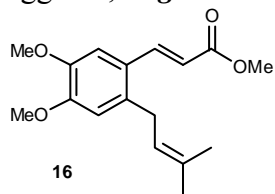


```

NAME      Apr26-2010-KEJ0886
EXPNO     11
PROCNO    1
Date_     20100426
Time      0.04
INSTRUM   AVIII400
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         4
DS         2
SWH        24038.461 Hz
FIDRES     0.164798 Hz
AQ         1.3631988 sec
RG         2000
DW         20.800 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      13C
P1         8.75 usec
PL1        -2.00 dB
PL1W       98.91786584 W
SFO1      100.6001970 MHz

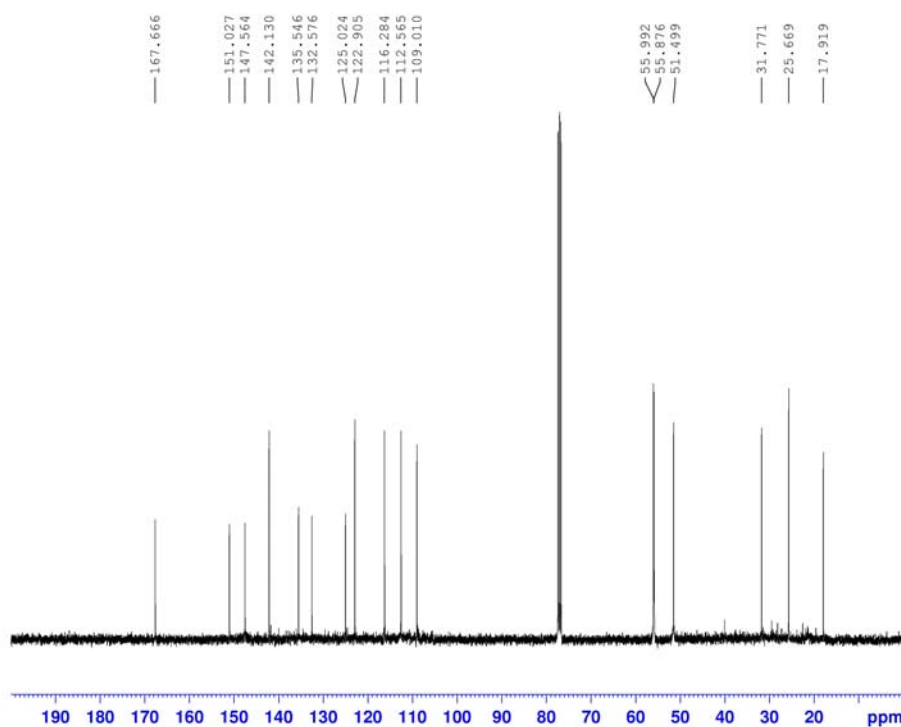
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2        0.00 dB
PL2W       19.78 dB
PL3        19.00 dB
PL3W       9.74611950 W
PL12W     0.25753233 W
PL13W     0.12549631 W
SFO2      400.0414002 MHz
SI         65536
SF         100.5901380 MHz
WDW        DM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```



```

NAME      May25-2010-KEJ2354
EXPNO    10
PROCNO   1
Date_    20100525
Time     18.41
INSTRUM  AVIII400
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        2
SWH       8223.685 Hz
FIDRES    0.125483 Hz
AQ        3.9846387 sec
RG        32
DW        60.800 usec
DE        17.24 usec
TE        298.0 K
D1        1.00000000 sec
TD0       1

===== CHANNEL f1 =====
NUC1      1H
P1        13.00 usec
PL1       0.00 dB
PL1W      9.74611950 W
SF01      400.0424704 MHz
SI        65536
SF        400.0400148 MHz
WDW       EM
SSB       0
LB        0.20 Hz
GB        0
PC        1.00
    
```

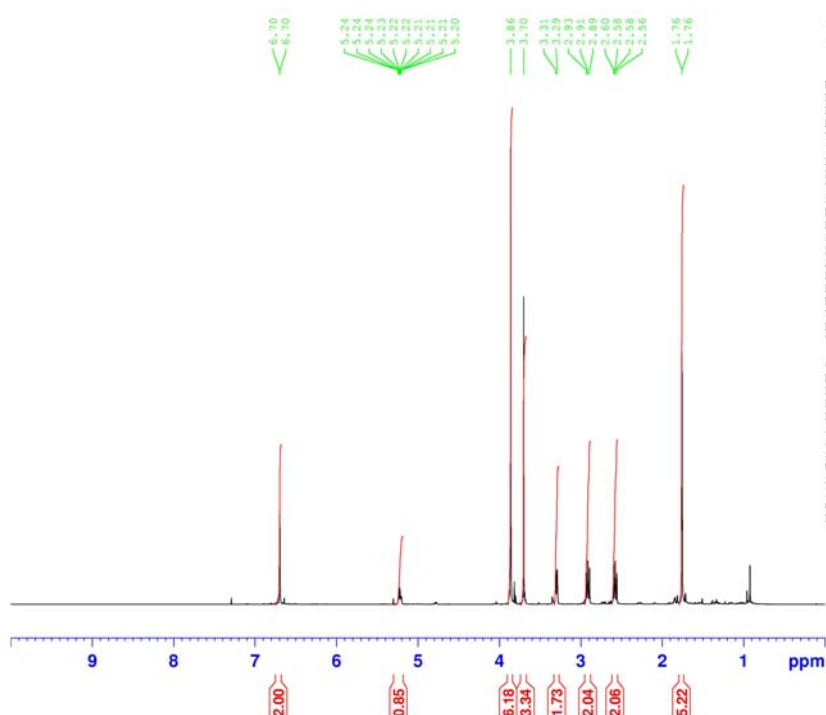
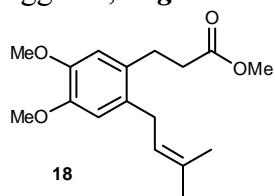


```

NAME      May25-2010-KEJ2354
EXPNO    11
PROCNO   1
Date_    20100526
Time     3.08
INSTRUM  AVIII400
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        12
DS        4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631988 sec
RG        2050
DW        20.800 usec
DE        6.50 usec
TE        298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1

===== CHANNEL f1 =====
NUC1      13C
P1        8.75 usec
PL1       -2.00 dB
PL1W      58.91986084 W
SF01      100.6001970 MHz

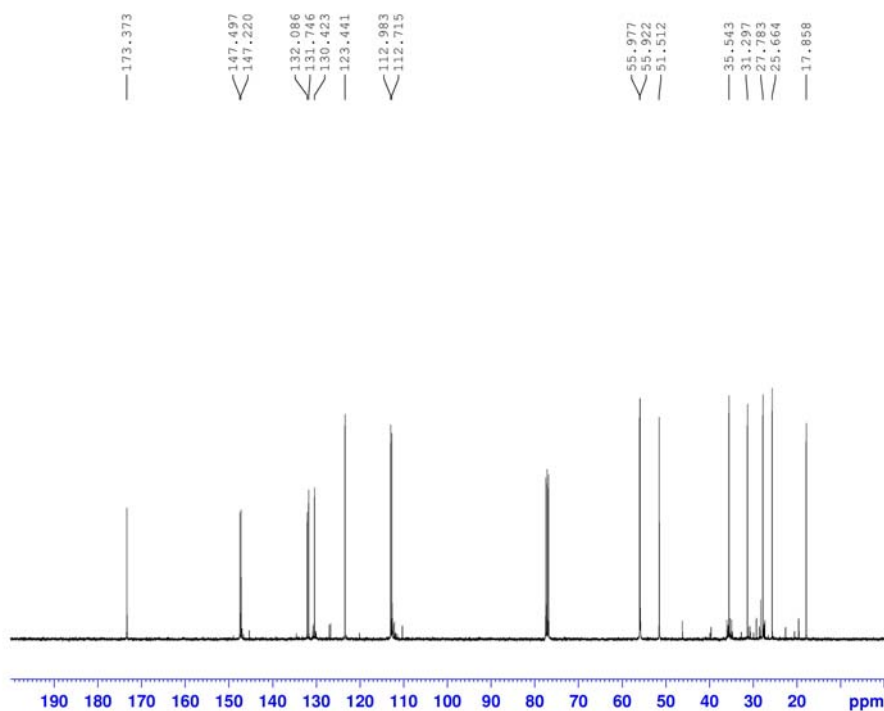
===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2      1H
PCPD2    80.00 usec
PL2       0.00 dB
PL12     15.78 dB
PL13     19.00 dB
PL1W     9.74611950 W
PL12W    0.25752333 W
PL13W    0.3269637 W
SF02     400.0416002 MHz
SI        65536
SF        100.5901380 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```



```

NAME      Apr28-2010-KEJ0964
EXPNO    10
PROCNO   1
Date_    20100429
Time     7.56
INSTRUM  AVI1400
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SWH      8223.685 Hz
FIDRES   0.125483 Hz
AQ       3.9846387 sec
RG       32
DM       60.800 usec
DE       17.24 usec
TE       298.0 K
D1       1.00000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1     1H
P1       13.00 usec
PL1      0.00 dB
PL1W     9.74611950 M
SF01     400.0424704 MHz
SI       65536
SF       400.0400000 MHz
WDW      EM
SSB      0
LB       0.20 Hz
GB       0
PC       1.00
    
```

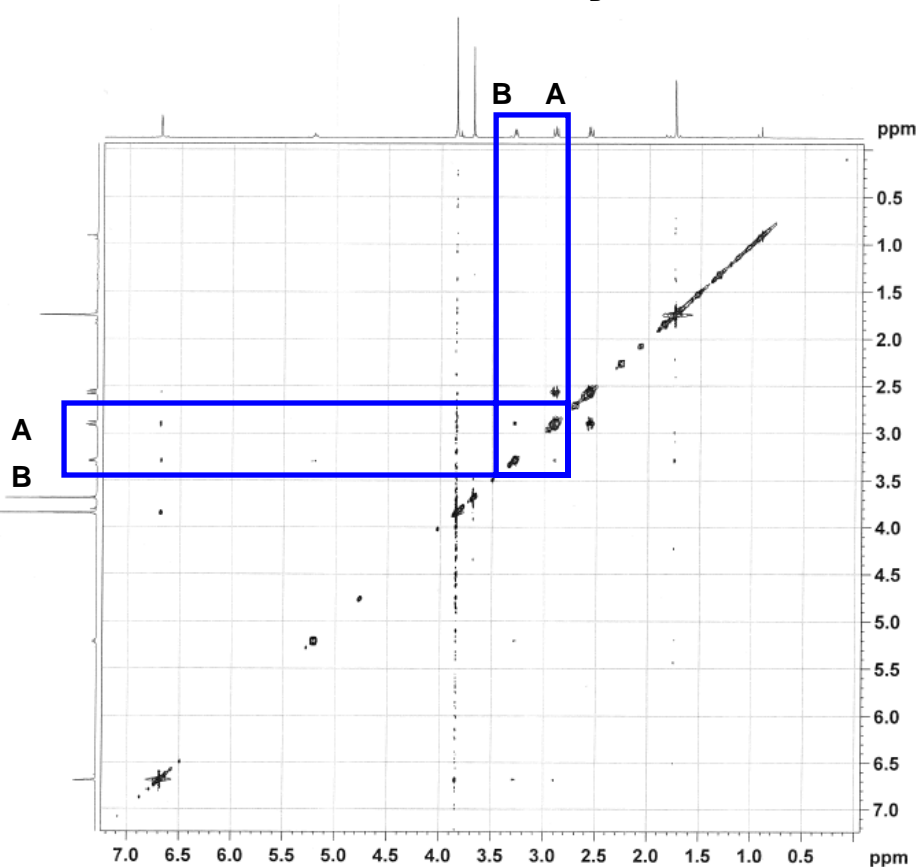
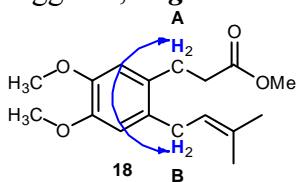


```

NAME      May11-2010-KEJ2003
EXPNO    11
PROCNO   1
Date_    20100511
Time     22.21
INSTRUM  AVI1400
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       512
DS       4
SWH      24038.461 Hz
FIDRES   0.366738 Hz
AQ       1.363388 sec
RG       2050
DM       20.800 usec
DE       6.50 usec
TE       298.0 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0      1

===== CHANNEL F1 =====
NUC1     13C
P1       8.75 usec
PL1      -2.00 dB
PL1W     58.91986284 M
SF01     100.601970 MHz

===== CHANNEL F2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    80.00 usec
PL2      0.00 dB
PL12     15.78 dB
PL13     19.00 dB
PL1W     9.74611950 M
PL12W    0.20703233 M
PL13W    0.32269631 M
SF02     400.0416002 MHz
SI       65536
SF       100.5903380 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```

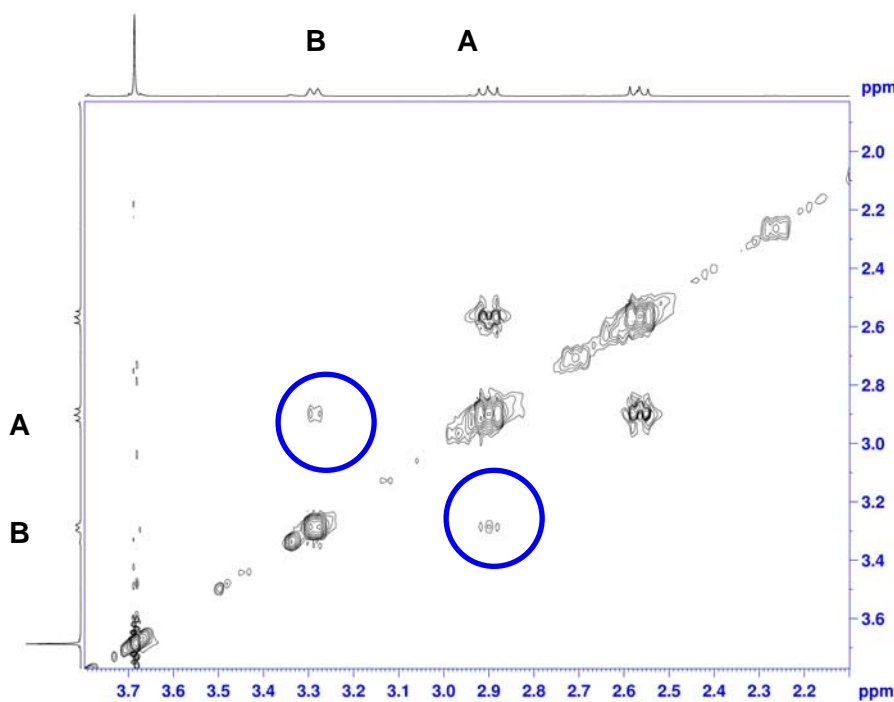


```

NAME May11-2010-KE2003
EXPNO 14
PROCNO 1
Date_ 20100511
Time_ 22.45
INSTRUM AVII400
PROBHD 5 mm PABBO BB-
PULPROG noesypph
TD 2048
SOLVENT CDCl3
NS 8
DS 8
SWH 2923.977 Hz
FIDRES 1.427723 Hz
AQ 0.3502580 sec
RG 11.3
DW 171.000 usec
DE 25.07 usec
TE 298.0 K
D0 0.0001545 sec
D1 1.86333001 sec
D8 0.30000001 sec
D16 0.00010000 sec
IN0 0.00034200 sec

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 usec
P2 26.00 usec
PL1 0.00 dB
PL1W 9.74611950 W
SFO1 400.0414368 MHz

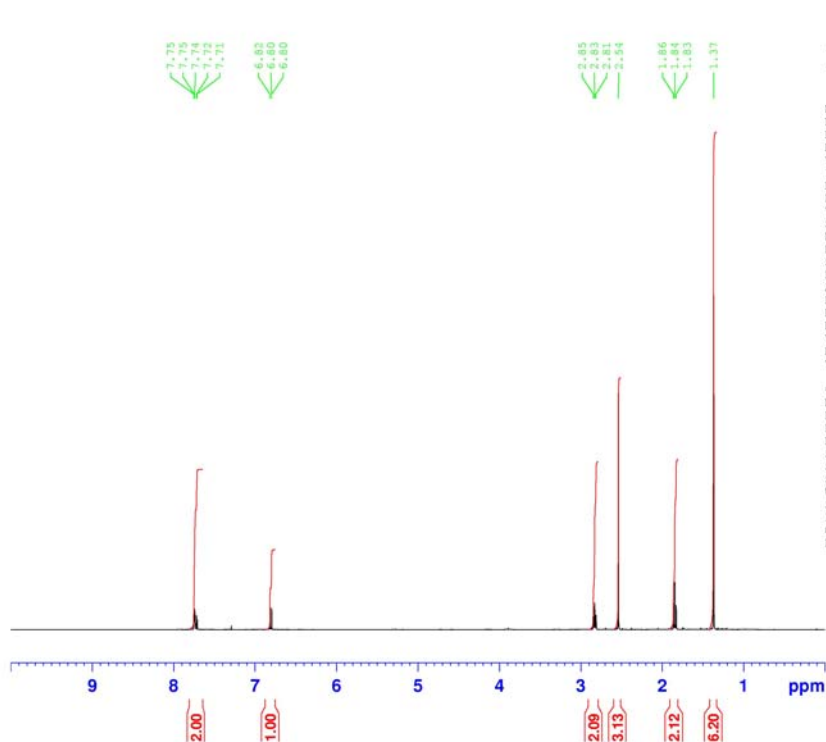
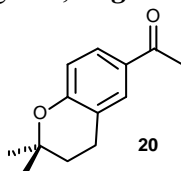
===== GRADIENT CHANNEL =====
GPMAM1 SMSQ10.100
GPMAM2 SMSQ10.100
GPE1 40.00 %
GPE2 -40.00 %
P1G 1000.00 usec
ND0 1
TD 256
SFO1 400.0414 MHz
FIDRES 11.421783 Hz
SW 7.309 ppm
F0MODE States-TPEI
SI 1024
SF 400.0400000 MHz
WDW QSI
SSB 2
LB 0.00 Hz
GB 0
PC 1.00
SI 1024
MC2 States-TPEI
SF 400.0400000 MHz
WDW QSI
SSB 2
LB 0.00 Hz
GB 0
    
```



```

NAME May11-2010-KE2200
EXPNO 10
PROCNO 1
Date_ 20100511
Time_ 14.38
INSTRUM AVII400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 20.2
DW 60.800 usec
DE 17.24 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 usec
PL1 0.00 dB
PL1W 9.74611950 W
SFO1 400.0424704 MHz
SI 65536
SF 400.0400000 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
    
```



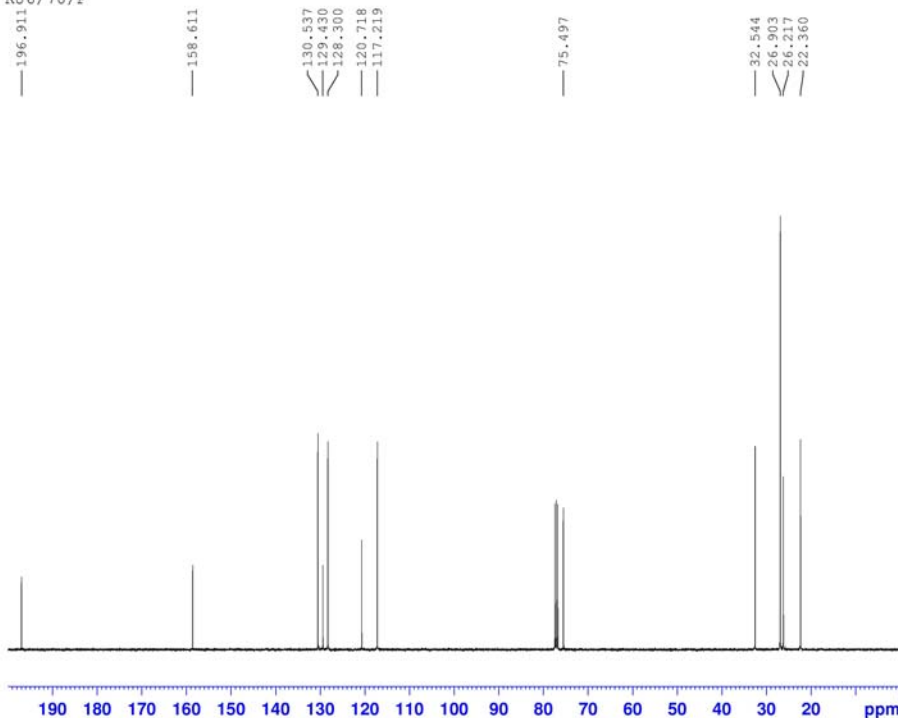
```

NAME      Apr26-2010-KEJ0885
EXPNO    10
PROCNO   1
Date_    20100426
Time     21.41
INSTRUM  AVII400
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        2
SWH       8223.685 Hz
FIDRES   0.125483 Hz
AQ        3.9846387 sec
RG        32
DW        60.800 usec
DE        17.24 usec
TE        298.0 K
D1        1.00000000 sec
TD0       1
    
```

```

===== CHANNEL f1 =====
NUC1      1H
P1         13.00 usec
PL1        0.00 dB
PL1W       9.74611950 W
SF01      400.0424704 MHz
SI         65536
SF         400.0400000 MHz
WDW        EM
SSB        0
LB         0.20 Hz
GB         0
PC         1.00
    
```

KJ6/70/P



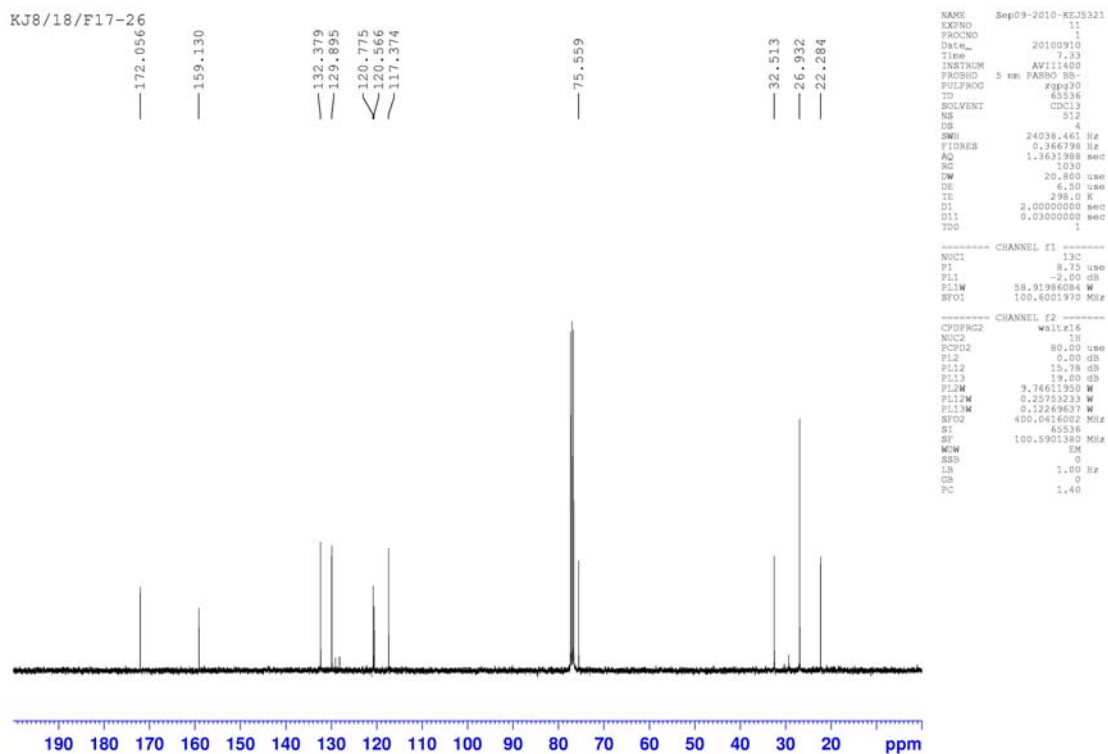
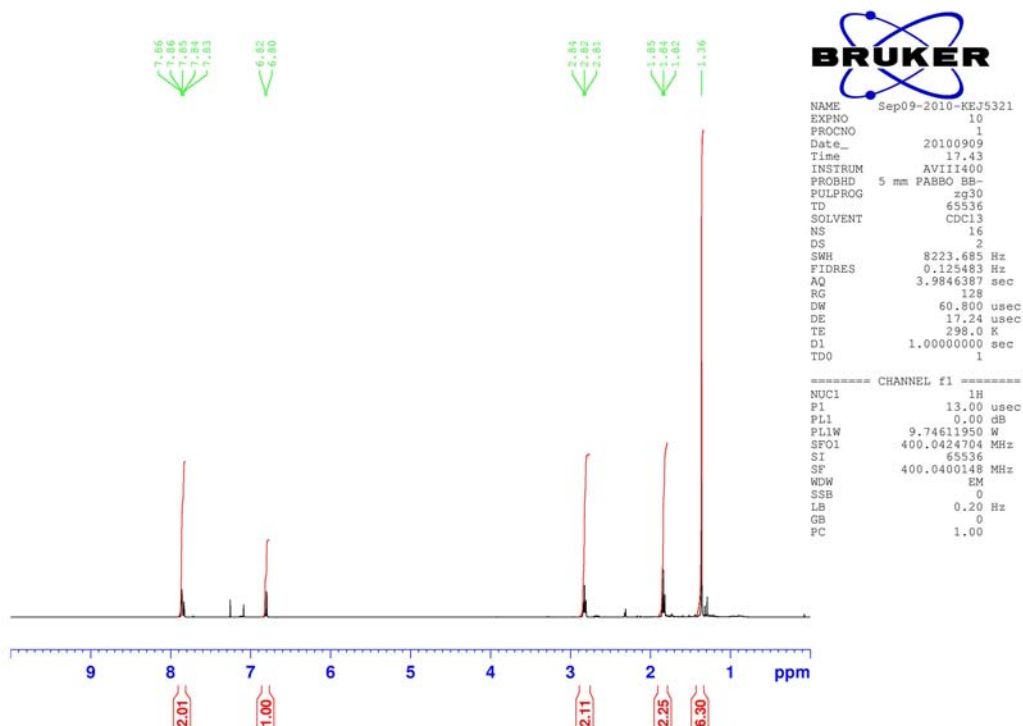
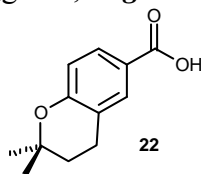
```

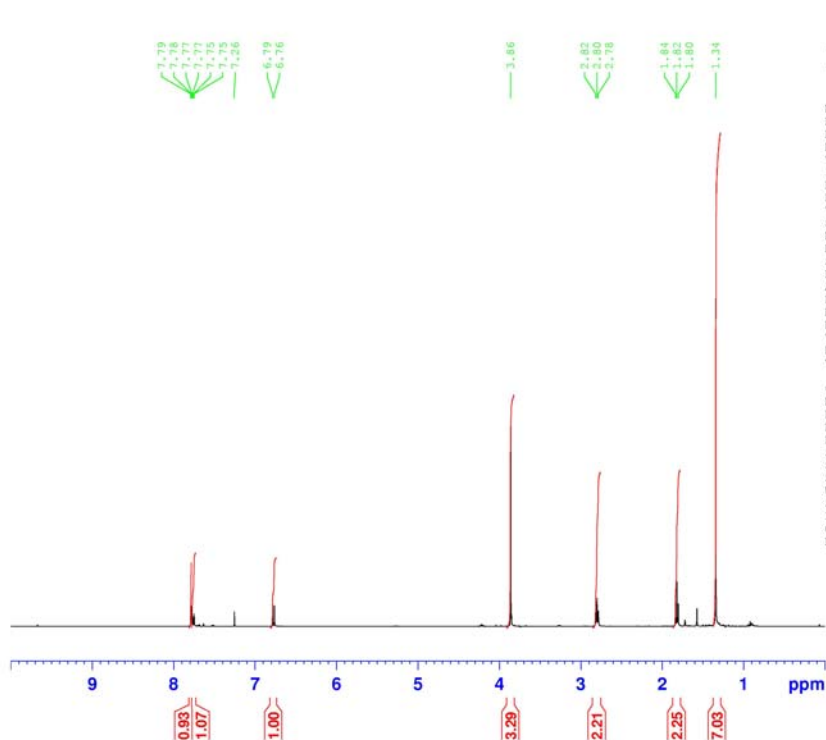
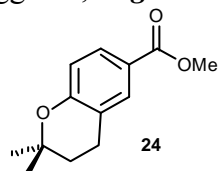
NAME      Apr26-2010-KEJ0885
EXPNO    11
PROCNO   1
Date_    20100426
Time     22.12
INSTRUM  AVII400
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        512
DS        4
SWH       24038.461 Hz
FIDRES   0.366798 Hz
AQ        1.3621988 sec
RG        2050
DW        20.800 usec
DE        6.50 usec
TE        298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1
    
```

```

===== CHANNEL f1 =====
NUC1      13C
P1         8.78 usec
PL1        -2.00 dB
PL1W       58.91986084 W
SF01      100.6001970 MHz

===== CHANNEL f2 =====
CFPPRG2  waltz16
NUC2      1H
PCPD2     80.00 usec
PL2        0.00 dB
PL2W       15.78 dB
PL13      19.00 dB
PL1W       9.74611950 W
PL13W     0.29753213 W
PL13W     0.12269637 W
SF02      400.0416002 MHz
SI         65536
MVM       100.5901380 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```



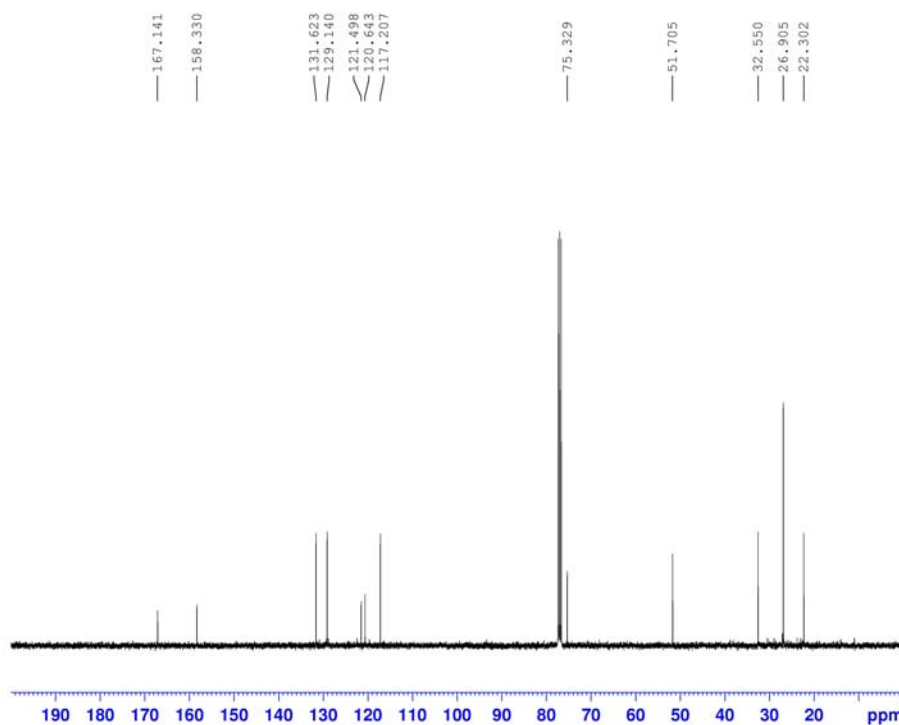


```

NAME      May27-2010-KEJ2417
EXPNO    10
PROCNO   1
Date_    20100527
Time     12.08
INSTRUM  AVII400
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD        65536
SOLVENT  CDC13
NS        16
DS        2
SWH       8223.685 Hz
FIDRES    0.125483 Hz
AQ        3.9846387 sec
RG        144
DW        60.800 usec
DE        17.24 usec
TE        298.0 K
D1        1.00000000 sec
TD0       1
    
```

```

===== CHANNEL f1 =====
NUC1     1H
P1       13.00 usec
PL1     0.00 dB
PL1W    9.74611950 W
SF01    400.0424704 MHz
SI       65536
SF      400.0400148 MHz
WDW      EM
SSB      0
LB       0.20 Hz
GB       0
PC       1.00
    
```



```

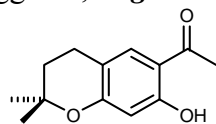
NAME      May27-2010-KEJ2417
EXPNO    11
PROCNO   1
Date_    20100527
Time     19.34
INSTRUM  AVII400
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD        65536
SOLVENT  CDC13
NS        512
DS        4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3637988 sec
RG        2050
DW        20.800 usec
DE        6.50 usec
TE        298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1
    
```

```

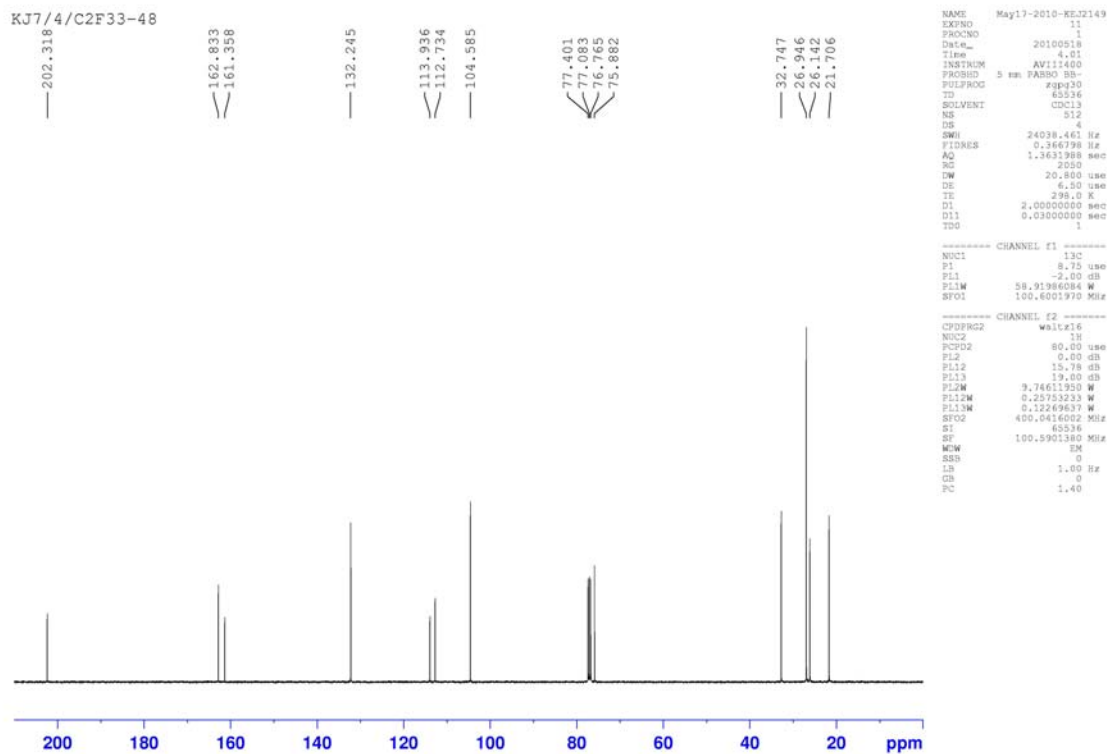
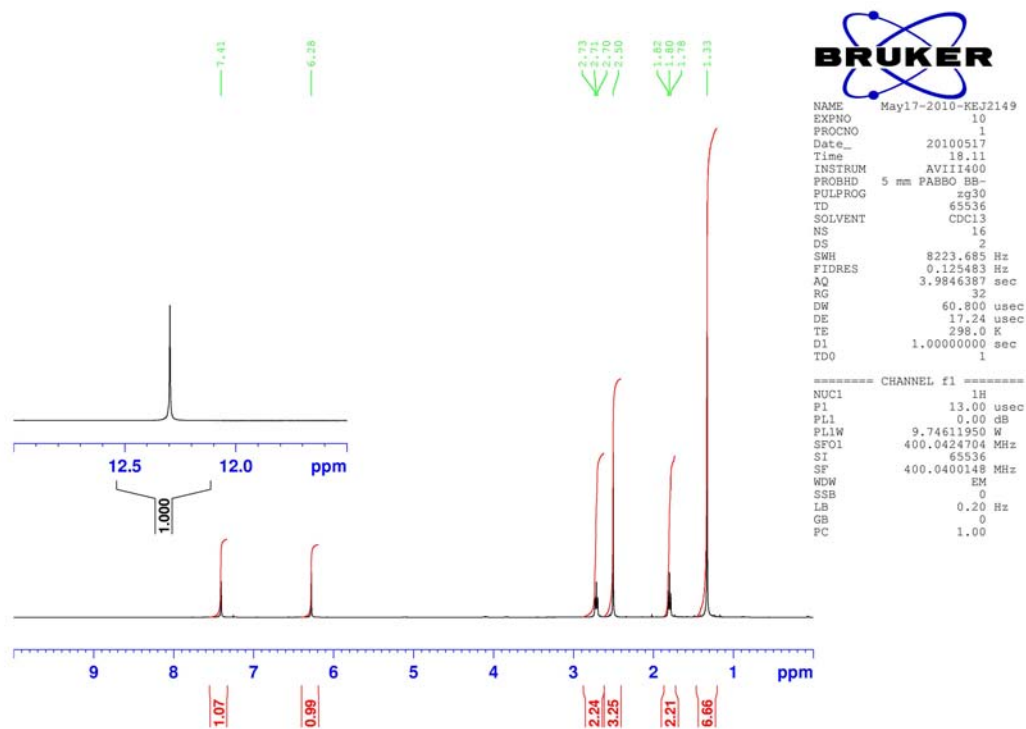
===== CHANNEL f1 =====
NUC1     13C
P1       8.75 usec
PL1     -2.00 dB
PL1W    58.91986084 W
SF01    100.6001970 MHz
    
```

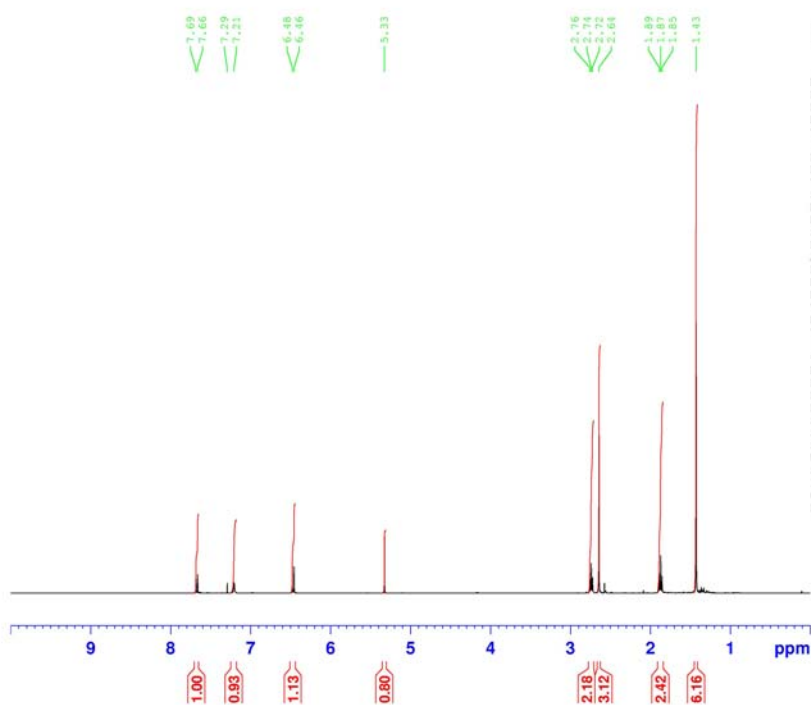
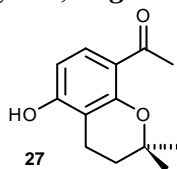
```

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    80.00 usec
PL2      0.00 dB
PL12     15.78 dB
PL13     19.00 dB
PL1W    9.74611950 W
PL1W    8.23753233 W
PL13W   0.12249637 W
SF02    400.0416002 MHz
SI       65536
SF      100.5901380 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```



26

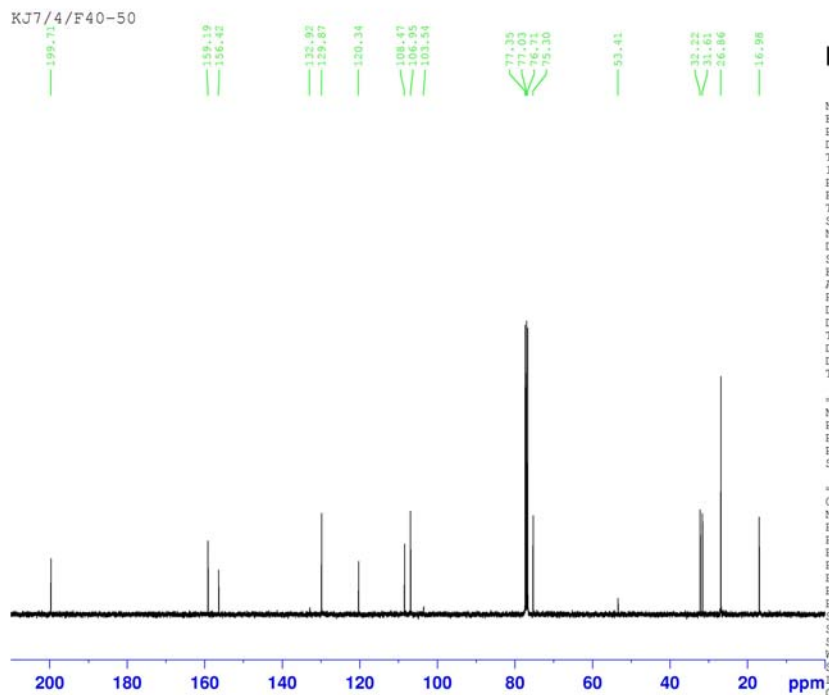




```

NAME      Apr29-2010-KEJ0970
EXPNO    10
PROCNO   1
Date_    20100429
Time     11.27
INSTRUM  AVIII400
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        2
SWH       8223.685 Hz
FIDRES   0.125483 Hz
AQ        3.9846387 sec
RG        114
DW        60.800 usec
DE        17.24 usec
TE        298.0 K
D1        1.00000000 sec
TD0       1

===== CHANNEL f1 =====
NUC1      1H
P1        13.00 usec
PL1       0.00 dB
PL1W      9.74611950 W
SFO1      400.0424704 MHz
SI        65536
SF        400.0400000 MHz
WDW       EM
SSB       0
LB        0.20 Hz
GB        0
PC        1.00
    
```

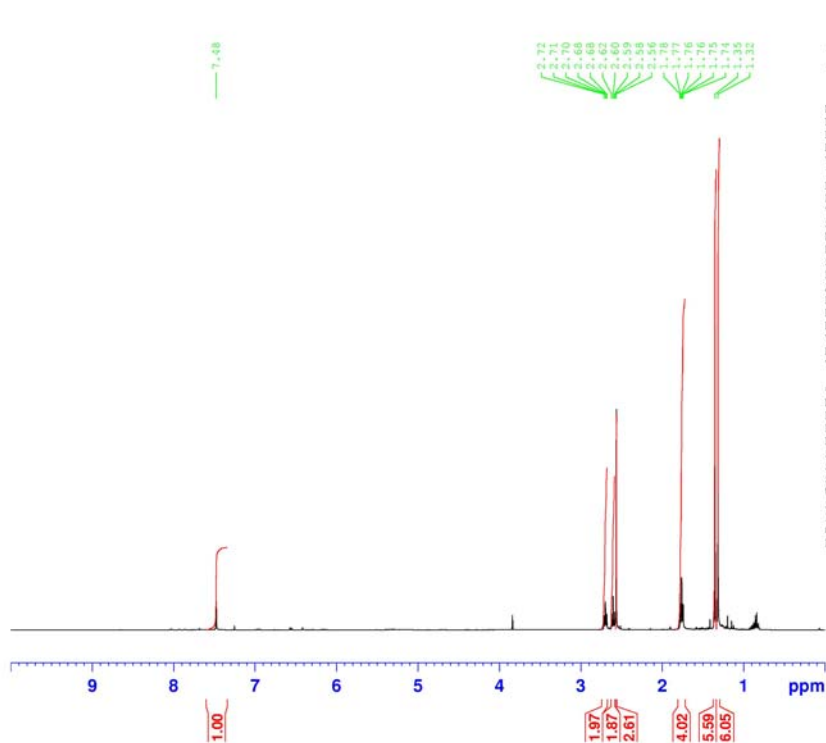
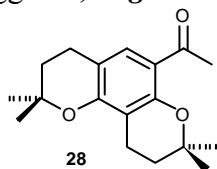


```

NAME      Apr29-2010-KEJ0970
EXPNO    11
PROCNO   1
Date_    20100429
Time     23.49
INSTRUM  AVIII400
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        512
DS        4
SWH       24038.461 Hz
FIDRES   0.366798 Hz
AQ        1.3631988 sec
RG        2050
DW        20.800 usec
DE        6.50 usec
TE        298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1

===== CHANNEL f1 =====
NUC1      13C
P1        8.75 usec
PL1       -2.00 dB
PL1W      58.91986084 W
SFO1      100.6001970 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2      1H
PCPD2    80.00 usec
PL2       0.00 dB
PL12     15.78 dB
PL13     19.00 dB
PL2W      9.74611950 W
PL12W    0.25753233 W
PL13W    0.12269637 W
SFO2     400.0416002 MHz
SI        65536
SF        100.5901380 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```



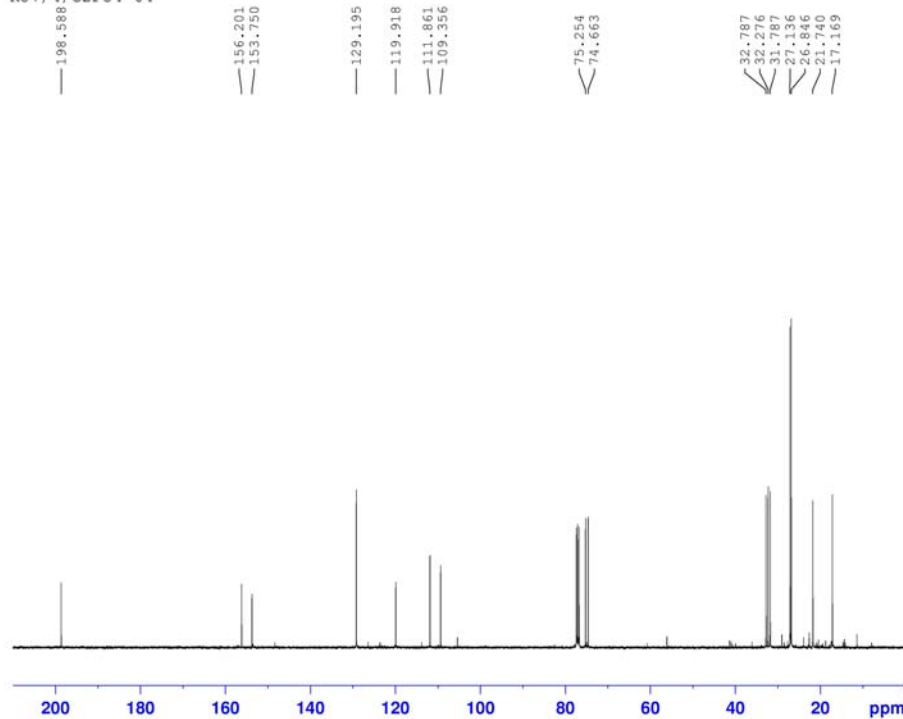
```

NAME      May17-2010-KEJ2150
EXPNO    10
PROCNO   1
Date_    20100517
Time     18.17
INSTRUM  AVII400
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SWH      8223.685 Hz
FIDRES   0.125483 Hz
AQ       3.9846387 sec
RG       32
DW       60.800 usec
DE       17.24 usec
TE       298.0 K
D1       1.0000000 sec
TD0      1
    
```

```

===== CHANNEL f1 =====
NUC1     1H
P1       13.00 usec
PL1      0.00 dB
PL1W     9.74611950 W
SF01     400.0424704 MHz
SI       65536
SF       400.0400148 MHz
WDW      EM
SSB      0
LB       0.20 Hz
GB       0
PC       1.00
    
```

KJ7/4/C2F54-64



```

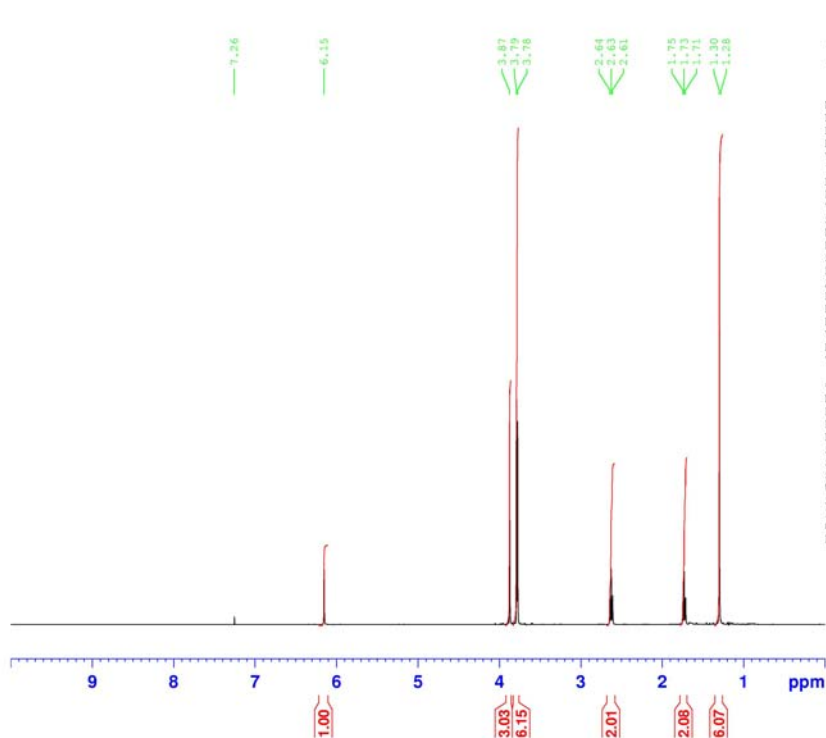
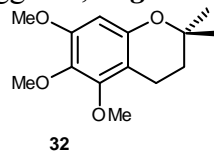
NAME      May17-2010-KEJ2150
EXPNO    12
PROCNO   1
Date_    20100518
Time     5.51
INSTRUM  AVII400
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       512
DS       4
SWH      24038.461 Hz
FIDRES   0.346798 Hz
AQ       1.3631988 sec
RG       2050
DW       20.800 usec
DE       6.50 usec
TE       298.0 K
D1       2.0000000 sec
D11      0.0300000 sec
TD0      1
    
```

```

===== CHANNEL f1 =====
NUC1     13C
P1       8.75 usec
PL1      -2.00 dB
PL1W     58.91986084 W
SF01     100.6001970 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2   80.00 usec
PL2      0.00 dB
PL2W    15.78 dB
PL13    19.00 dB
PL1W    9.74611950 W
PL13W   0.23783233 W
PL13W   0.12249637 W
SF02    400.0416002 MHz
SI       65536
SF       100.5901380 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```

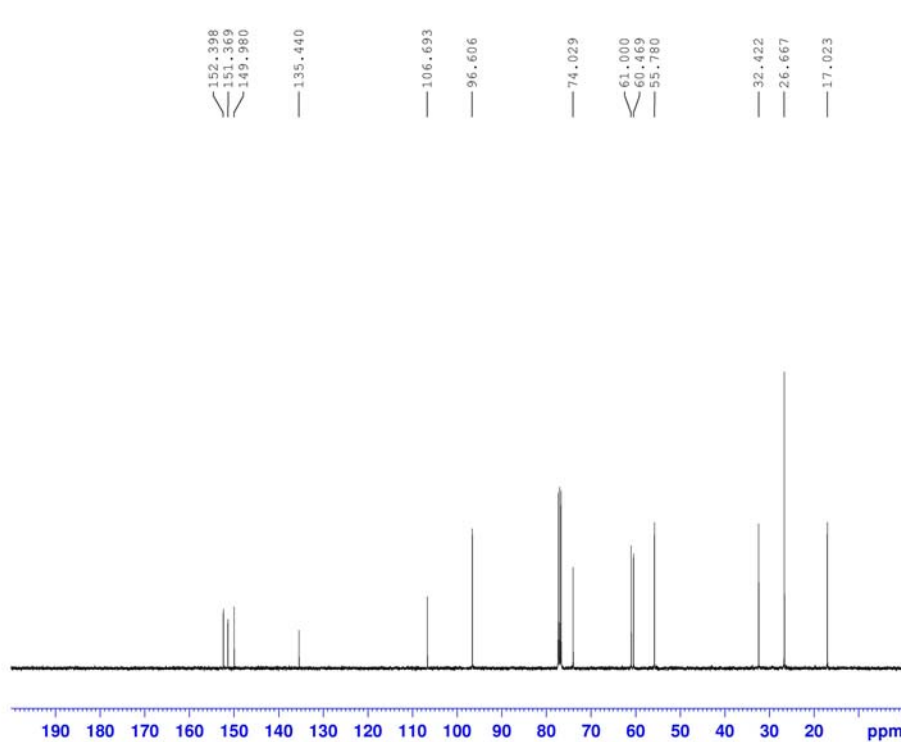


```

NAME      May20-2010-KEJ2230
EXPNO    10
PROCNO    1
Date_    20100520
Time     12.12
INSTRUM  AVIII400
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        2
SWH       8223.685 Hz
FIDRES   0.125483 Hz
AQ        3.9846387 sec
RG        57
DW        60.800 usec
DE        17.24 usec
TE        298.0 K
D1        1.00000000 sec
TD0       1
    
```

```

----- CHANNEL f1 -----
NUC1      1H
P1        13.00 usec
PL1       0.00 dB
PL1W      9.74611950 W
SFO1      400.0424704 MHz
SI        65536
SF        400.0400148 MHz
WDW       EM
SSB       0
LB        0.20 Hz
GB        0
PC        1.00
    
```



```

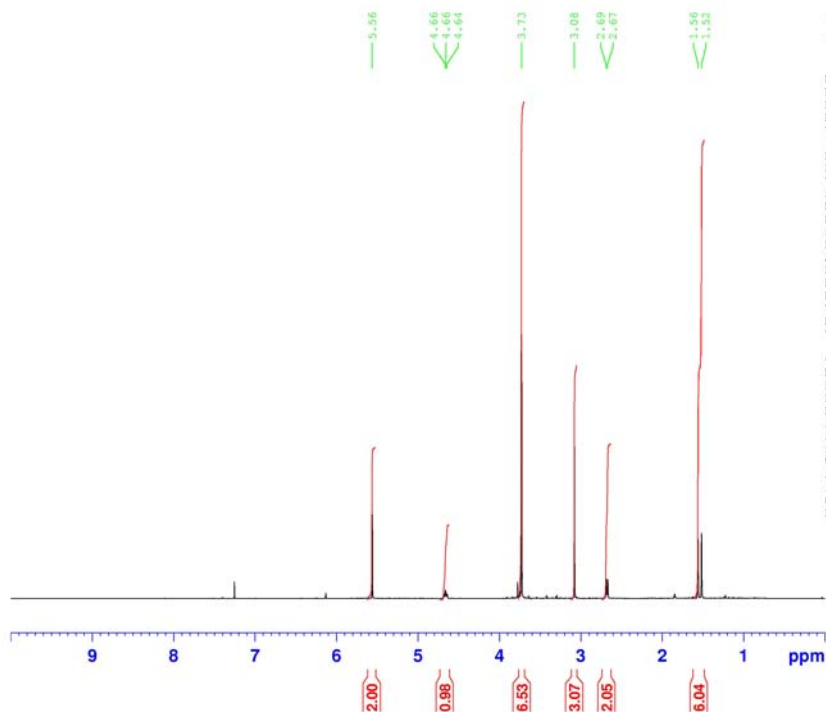
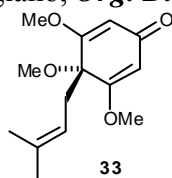
NAME      May20-2010-KEJ2230
EXPNO    11
PROCNO    1
Date_    20100521
Time     2.09
INSTRUM  AVIII400
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        512
DS        4
SWH       24038.461 Hz
FIDRES   0.346798 Hz
AQ        1.3637988 sec
RG        2050
DW        20.800 usec
DE        6.50 usec
TE        298.0 K
D1        2.00000000 sec
D11      0.03000000 sec
TD0       1
    
```

```

----- CHANNEL f1 -----
NUC1      13C
P1        8.75 usec
PL1       -2.00 dB
PL1W      58.91986084 W
SFO1      100.6001970 MHz
    
```

```

----- CHANNEL f2 -----
CPDPRG2  waltz16
NUC2      1H
PCPD2    80.00 usec
PL2       0.00 dB
PL12     15.78 dB
PL13     19.00 dB
PL1W     9.74611950 W
PL12W    0.23753233 W
PL13W    0.12249637 W
SFO2     400.0416002 MHz
SI        65536
SF        100.5901380 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```



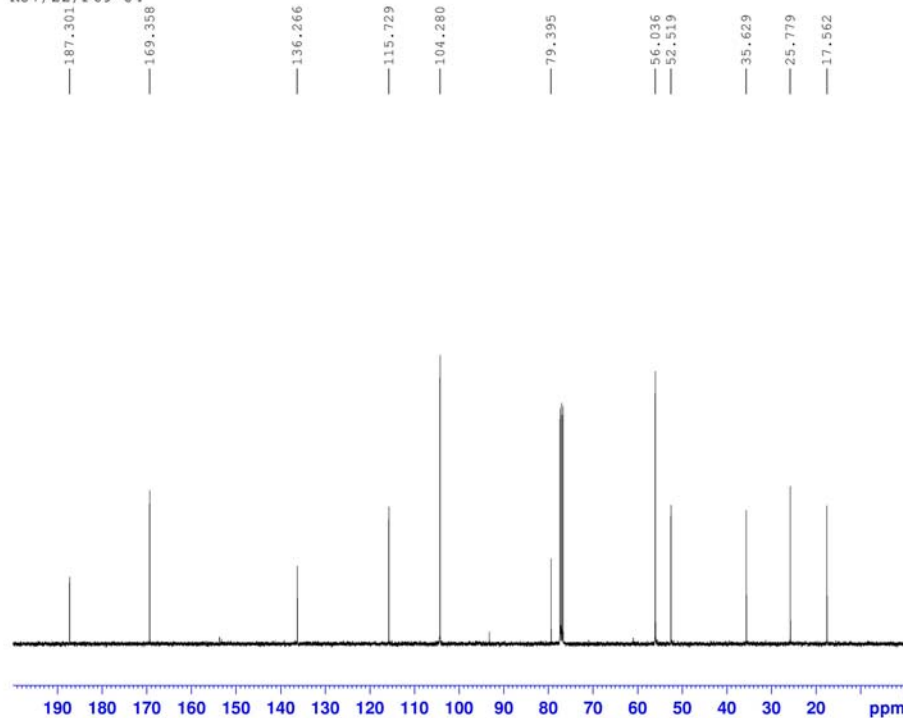
```

NAME      May20-2010-KEJ2244
EXPNO     16
PROCNO    1
Date_     20100521
Time      5.09
INSTRUM   AVII1400
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         64
DW         60.800 usec
DE         17.24 usec
TE         298.0 K
D1         1.00000000 sec
TD0        1
    
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        13.00 usec
PL1       0.00 dB
PL1W      9.74611950 W
SFO1      400.0424704 MHz
SI        65536
SF        400.0400148 MHz
WDW       EM
SSB       0
LB        0.20 Hz
GB        0
PC        1.00
    
```

KJ7/22/F69-84



```

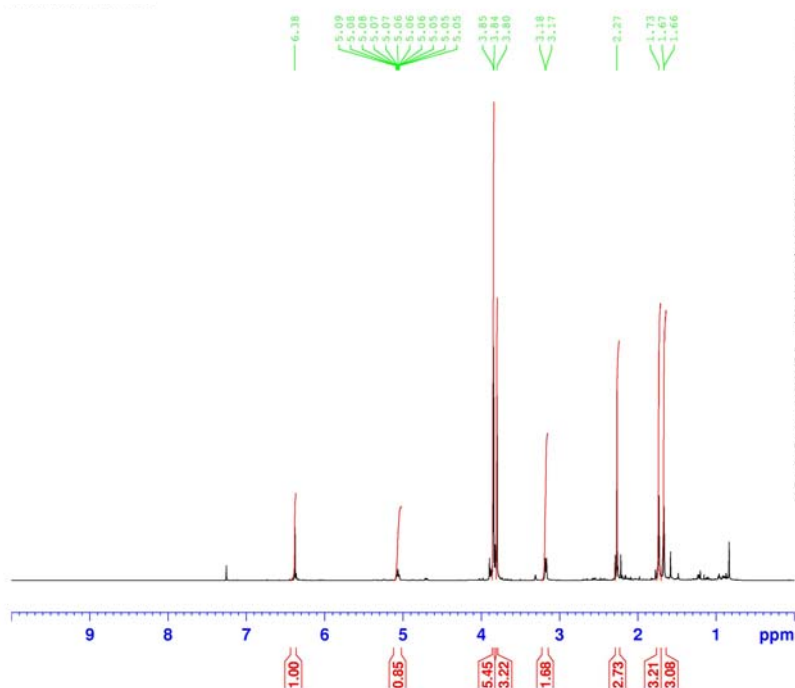
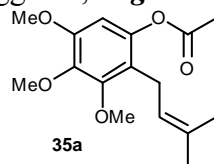
NAME      May20-2010-KEJ2244
EXPNO     11
PROCNO    1
Date_     20100521
Time      3.52
INSTRUM   AVII1400
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         512
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3637988 sec
RG         2050
DW         20.800 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
    
```

```

===== CHANNEL f1 =====
NUC1      13C
P1        8.75 usec
PL1       -2.00 dB
PL1W      58.91986084 W
SFO1      100.6201970 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       0.00 dB
PL12      15.78 dB
PL13      19.00 dB
PL1W      9.74611950 W
PL1W      0.23753233 W
PL13W     0.12249637 W
SFO2      400.0416002 MHz
SI        65536
SF        100.5901380 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```

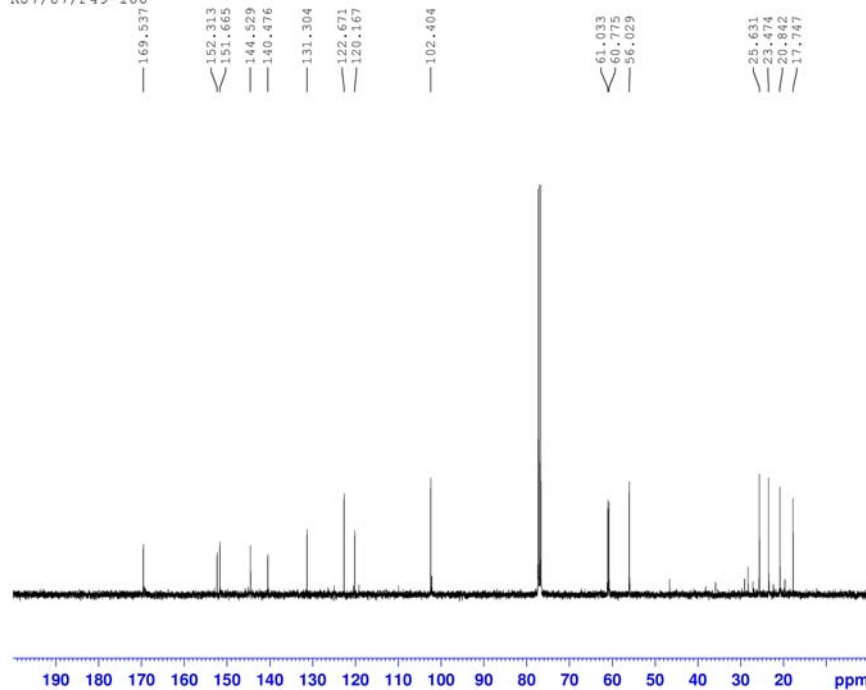


```

NAME      Jul26-2010-KJ4136
EXPNO     10
PROCNO    1
Date_     20100726
Time      13:24
INSTRUM   AVI1400
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         90.5
DW         60.800 usec
DE         17.24 usec
TE         298.0 K
D1         1.0000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      1H
P1        13.00 usec
PL1       0.00 dB
PL1W      9.74611950 W
SFO1      400.0424704 MHz
SI        65536
SF        400.0400148 MHz
WDW       EM
SSB       0
LB        0.20 Hz
GB        0
PC        1.00
    
```

KJ7/87/F49-100

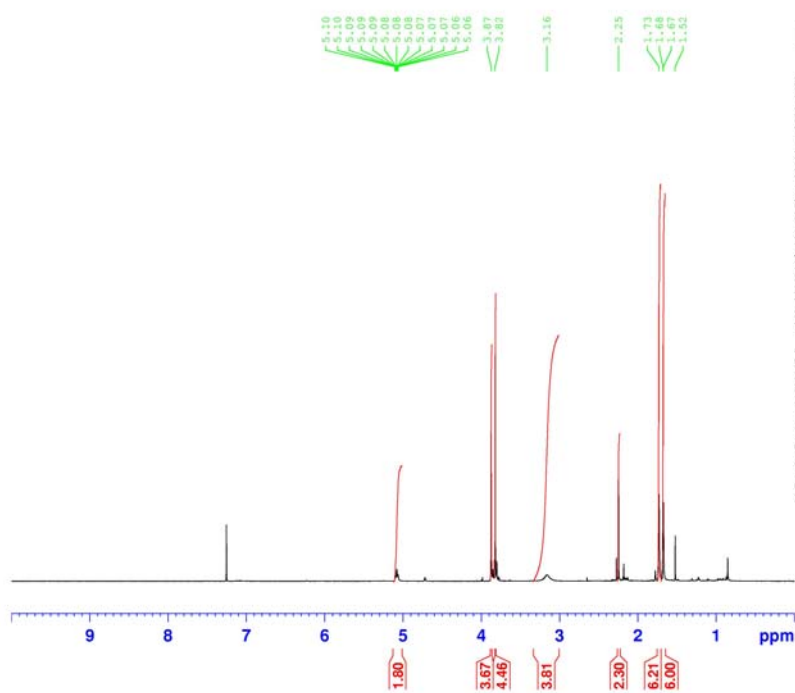
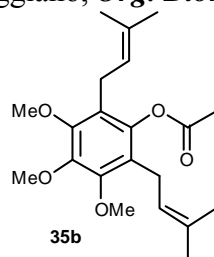


```

NAME      Jul26-2010-KJ24136
EXPNO     11
PROCNO    1
Date_     20100726
Time      21:06
INSTRUM   AVI1400
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         4
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3637988 sec
RG         2050
DW         20.800 usec
DE         4.50 usec
TE         298.0 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      13C
P1        8.75 usec
PL1       -2.00 dB
PL1W      58.91986084 W
SFO1      100.6001970 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       0.00 dB
PL12      15.78 dB
PL13      15.00 dB
PL1W      9.74611950 W
PL2W      0.25753233 W
PL3W      0.12269637 W
SFO2      400.0416002 MHz
SI        65536
SF        100.5901380 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```

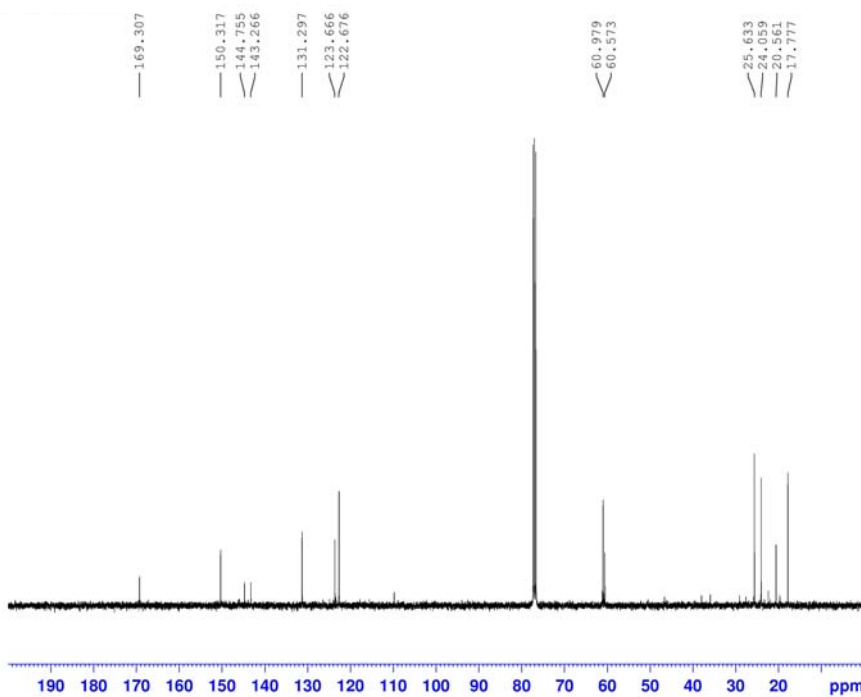


```

NAME Jul24-2010-KEJ4114
EXPNO 10
PROCNO 1
Date_ 20100724
Time 21.12
INSTRUM AVIII400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 256
DW 60.800 usec
DE 17.24 usec
TE 298.0 K
D1 1.0000000 sec
TD0 1
    
```

```

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 usec
PL1 0.00 dB
PL1W 9.74611950 W
SFO1 400.0424704 MHz
SI 65536
SF 400.0400148 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
    
```



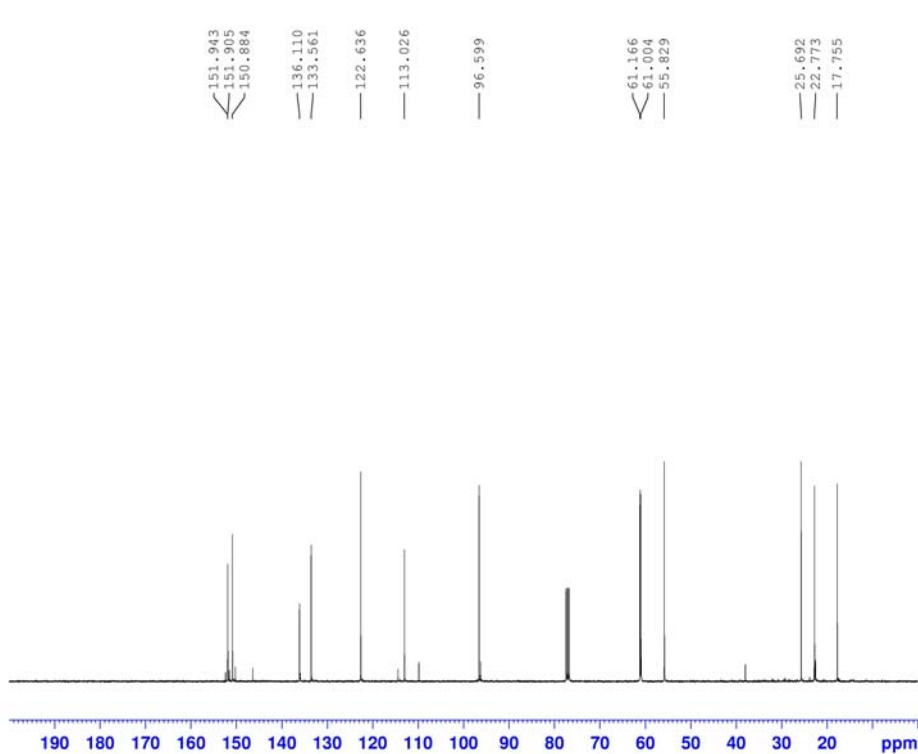
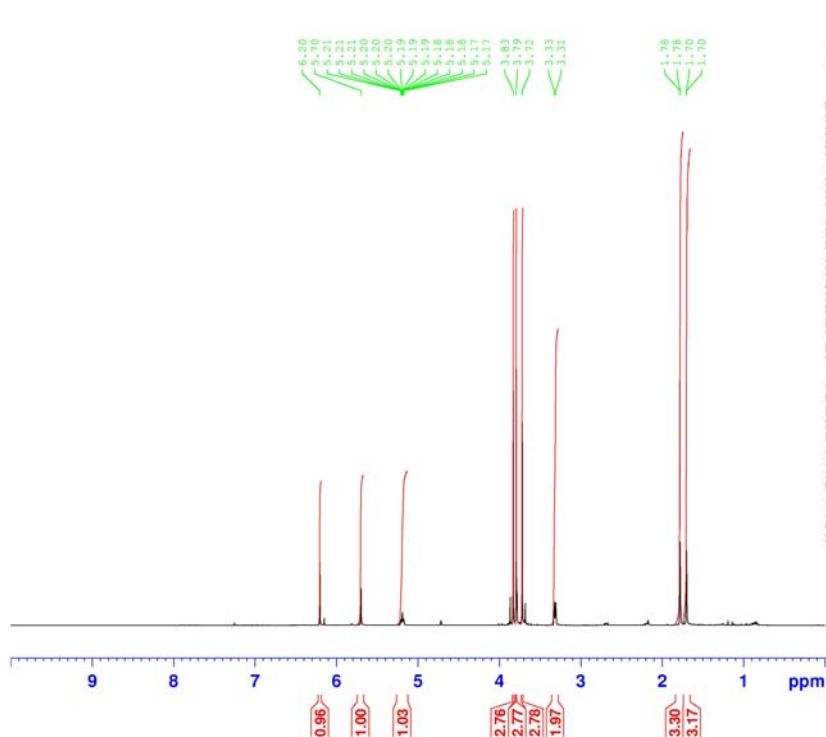
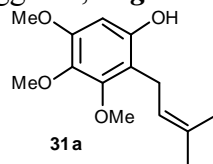
```

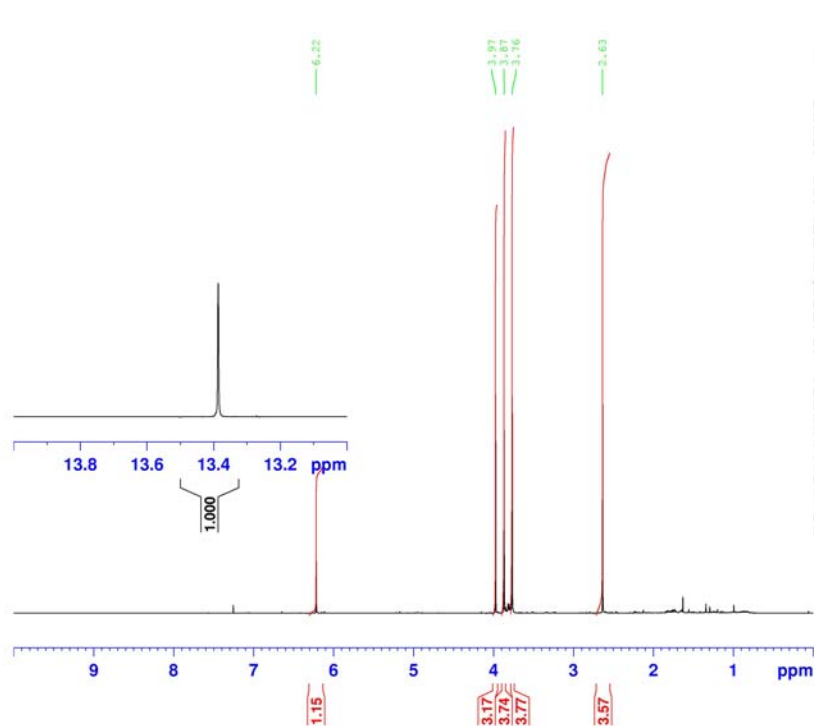
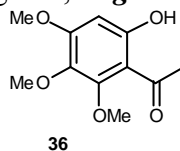
NAME Jul27-2010-KEJ4193
EXPNO 11
PROCNO 1
Date_ 20100728
Time 4.20
INSTRUM AVIII400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 12
DS 4
SWH 24038.445 Hz
FIDRES 0.266798 Hz
AQ 1.3631988 sec
RG 2000
DW 20.800 usec
DE 4.30 usec
TE 298.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1
    
```

```

===== CHANNEL f1 =====
NUC1 13C
P1 8.75 usec
PL1 -2.00 dB
PL1W 58.9198026 W
SFO1 100.6001970 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL2 15.78 dB
PL13 19.00 dB
PL1W 9.74611950 W
PL12W 0.20753233 W
PL13W 0.12269633 W
SFO2 400.0416002 MHz
SI 65536
SF 100.590380 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
    
```

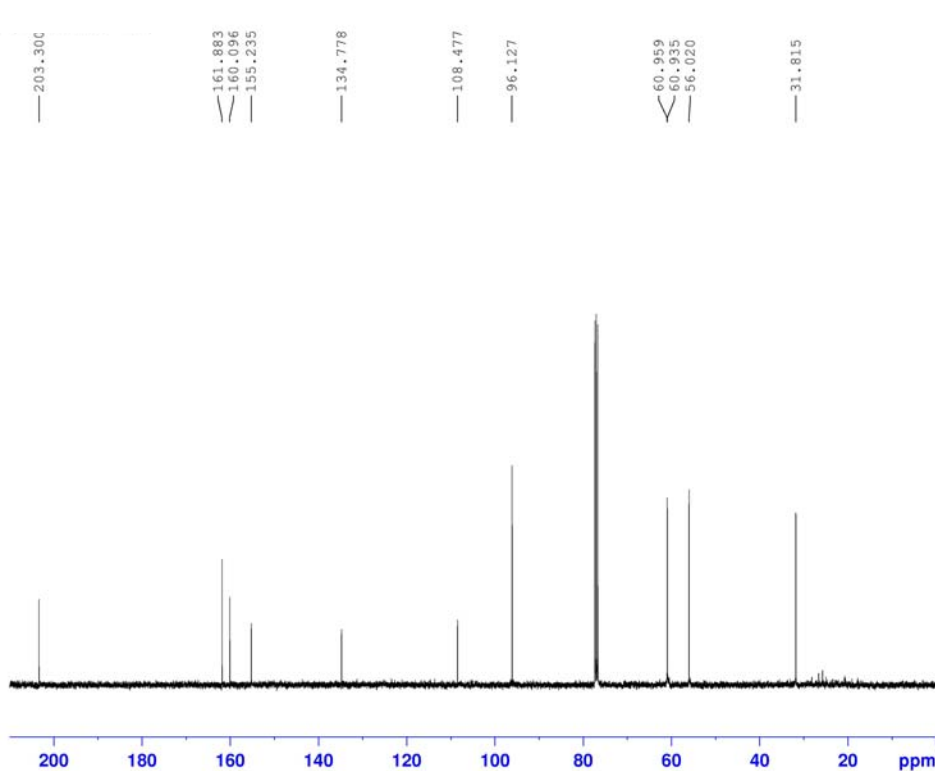




```

NAME      May29-2010-KEJ2462
EXPNO    10
PROCNO   1
Date_    20100529
Time     17.01
INSTRUM  AVIII400
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        2
SMH       8223.685 Hz
FIDRES    0.125483 Hz
AQ        3.9846387 sec
RG        71.8
DW        60.800 usec
DE        17.24 usec
TE        298.0 K
D1        1.00000000 sec
TD0       1

===== CHANNEL f1 =====
NUC1     1H
P1       13.00 usec
PL1      0.00 dB
PL1W     9.74611950 W
SFO1     400.0424704 MHz
SI       65536
SF       400.0400148 MHz
WDW      EM
SSB      0
LB       0.20 Hz
GB       0
PC       1.00
    
```



```

NAME      May29-2010-KEJ2462
EXPNO    11
PROCNO   1
Date_    20100530
Time     12.52
INSTRUM  AVIII400
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD        65536
SOLVENT  CDCl3
NS        12
DS        4
SMH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631988 sec
RG        2050
DW        20.800 usec
DE        6.50 usec
TE        298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1

===== CHANNEL f1 =====
NUC1     13C
P1       8.75 usec
PL1      -2.00 dB
PL1W     58.91986084 W
SFO1     100.6001970 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    80.00 usec
PL2      0.00 dB
PL12     15.78 dB
PL13     19.00 dB
PL1W     9.74611950 W
PL12W    0.25763214 W
PL13W    0.12269637 W
SFO2     400.0416002 MHz
SI       65536
SF       100.5901380 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```