Supporting Information

Copper-Catalyzed Tandem Process: An Efficient Approach to 2-Substituted-1, 4-benzodioxanes

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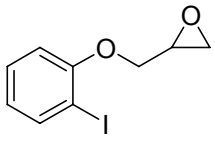
A. Experimental Procedure

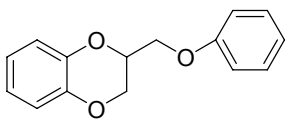
General Information. All reactions were carried out in Schlenk tube and run under a N_2 atmosphere. All the copper sources, ligands and bases were commercially available. Organic solvents, also were commercially available, were dried over 4Å molecular sieves before used. 2-((2-iodophenoxy)methyl)oxirane were prepared according to the known literature.[1] Thin-layer chromatography (TLC) was carried out with 0.2 mm thick silica gel plates (GF 254) and visualized by UV light. All melting points are uncorrected. All products were confirmed by 1H NMR, 13C NMR. Unknown compounds were additionally confirmed by MS and HRMS. MS and HRMS were obtained using EI ionization. The NMR spectra were recorded in CDCl3 on a 400 MHz instrument with TMS as internal standard. IR spectrums were taken in ATR apparatus.

General Procedure for the Synthesis of 2-Substituted-1, 4-benzodioxanes. A Schlenk tube was charged with CuBr (22 mg, 15% mol), 1,10-phenanthroline (57 mg, 30% mol), Cs_2CO_3 (650 mg, 2 mmol), phenol (113 mg, 1.2 mmol), evacuated and backfilled with nitrogen. Then 2-((2-iodophenoxy)methyl)oxirane (276 mg, 1.0 mmol), DMA (2 mL) were successively added. The reaction tube was quickly sealed and the contents were stirred at 120 ºC for 48 h. Then the cooled reaction mixture was dissolved in H_2O and extracted with Et_2O. The combined organic layer was dried ($MgSO_4$). The product was further purified by column chromatography (silica gel, PE-EtOAC).

B. Spectra Data

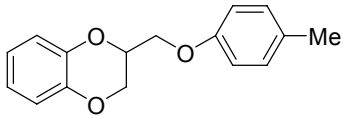


2-((2-iodophenoxy)methyl)oxirane (**1a)**: Oil, ^{1}H NMR (400MHz, $CDCl_3$): δ 7.76-7.74 (m, 1H), 7.29-7.24 (m, 1H), 6.83-6.81 (m, 1H), 6.73-6.69 (m, 1H), 4.28-4.25 (m, 1H), 4.04-4.00 (m, 1H), 3.37-3.36 (m, 1H), 2.90-2.87 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.98, 139.52, 129.55, 123.12, 112.56, 86.62, 69.27, 50.13, 44.74.

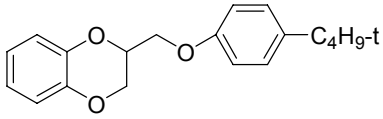


2-phenoxymethyl-2,3-dihydrobenzo[1,4]dioxin (**3a)**: White solid, m.p. 35-36°C[2]; ^{1}H NMR (400MHz, $CDCl_3$): δ 7.33 (t, *J* = 7.6 Hz, 2H), 7.02 (t, *J* = 7.6 Hz, 1H), 6.96-6.90 (m, 6H), 4.59-4.56 (m, 1H), 4.43 (dd, *J* = 1.4, 11.4Hz, 1H),4.30-4.22 (m, 2H), 4.19-4.15

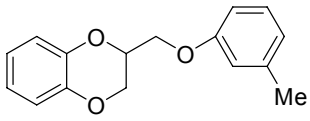
(m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.26, 143.18, 142.91, 129.55, 121.74, 121.53, 121.40, 117.40, 117.25, 114.56, 71.34, 66.24, 65.34; IR: υ = 3019, 2930, 2882, 1578, 1493, 1269, 1237, 1076, 745, 689 cm^{-1}.



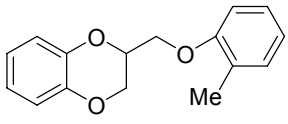
2-(p-tolyloxymethyl)-2,3-dihydrobenzo[1,4]dioxine (**3b**): White solid, m.p. 56-57°C; ^{1}H NMR (400MHz, $CDCl_3$): δ 7.11 (d, *J* = 8.4Hz, 2H), 6.96-6.92 (m, 2H), 6.89-6.84 (m, 4H), 4.58-4.54 (m, 1H), 4.43-4.40 (m, 1H), 4.26-4.19 (m, 2H), 4.15-4.10 (m, 1H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.22, 143.22, 142.96, 130.67, 130.00, 121.73, 121.52, 117.42, 117.26, 114.45, 71.41, 66.47, 65.39, 20.47; IR: υ = 3034, 2947, 2922, 2882, 1591, 1492, 235, 1041, 842, 739 cm^{-1}; EI-MS: m/z = 256 (M^+); HRMS (EI) calcd for $C_{16}H_{16}O_3$ 256.1099, found 256.1100.



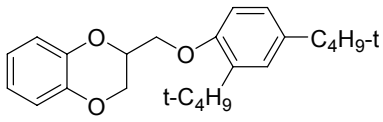
2-((4-tert-butylphenoxy)methyl)-2,3-dihydrobenzo[1,4]dioxine (**3c**): White solid, m.p. 59-60°C; ^{1}H NMR (400MHz, $CDCl_3$): δ 7.34 (d, *J* = 7.6 Hz, 2H), 6.95-6.87 (m, 6H), 4.59-4.55 (m, 1H), 4.43-4.40 (m, 1H), 4.28-4.20 (m, 2H), 4.17-4.12 (m, 1H), 1.33 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.02, 144.14, 143.19, 142.92, 126.33, 121.71, 121.49, 117.38, 117.24, 114.03, 71.35, 66.32, 65.40, 34.09, 31.49; IR: υ = 3053, 2958, 2932, 2901, 2875, 1592, 1493, 1267, 1030, 841, 744 cm^{-1}; EI-MS: *m/z* = 298 (M^+); HRMS (EI) calcd for $C_{19}H_{22}O_3$ 258.1569, found 298.1566.



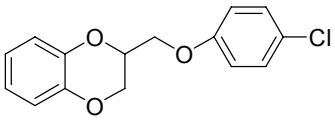
2-(m-tolyloxymethyl)-2,3-dihydrobenzo[1,4]dioxine (**3d**): Oil; ^{1}H NMR (400MHz, $CDCl_3$): δ 7.15 (t, *J* = 8.0Hz, 1H), 6.92-6-88 (m, 2H), 6.85-6.82 (m, 2H), 6.78 (d, *J* = 7.8Hz, 1H), 6.74-6.70 (m, 2H), 4.54-4.50 (m, 1H), 4.38-4.35 (m, 1H), 4.23-4.15 (m, 2H), 4.11-4.07 (m, 1H), 2.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.31, 143.22, 142.95, 139.66, 129.31, 122.23, 121.74, 121.53, 117.42, 117.26, 115.44, 111.44, 71.37, 66.20, 65.37, 21.49; IR: υ = 3048, 2977, 2921, 1590, 1490, 1248, 1043, 848, 780, 745 cm^{-1}; EI-MS: *m/z* = 256 (M^+). HRMS (EI) calcd for $C_{16}H_{16}O_3$ 256.1099, found 256.1101.



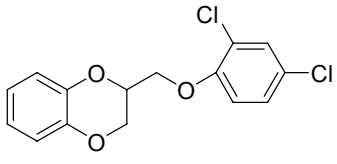
2-(o-tolyloxymethyl)-2,3-dihydrobenzo[1,4]dioxine (**3e**)[3]: Oil; ^{1}H NMR (400MHz, $CDCl_3$): δ 720-7.16 (m, 2H), 6.96-6.92 (m, 3H), 6.90-6.84 (m, 3H), 4.62-4.58 (m, 1H), 4.46 (dd, *J* = 1.6, 11.4 Hz, 1H), 4.31-4.24 (m, 2H), 4.20-4.16 (m, 1H), 2.26 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.35, 143.21, 143.03, 130.84, 126.91, 126.86, 121.72, 121.50, 121.10, 117.35, 117.24, 111.04, 71.42, 66.35, 65.51, 16.15; IR: υ = 3053, 2923, 2882, 1592, 1491, 1239, 1044, 743 cm^{-1}.



2-((2,4-di-tert-butylphenoxy)methyl)-2,3-dihydrobenzo[1,4]dioxine (**3f**): Oil; ^{1}H NMR (400MHz, $CDCl_3$): δ 7.34 (s, 1H), 7.18 (d, *J* = 8.4 Hz, 1H), 6.90-6.86 (m, 4H), 6.80 (d, *J* = 8.4 Hz, 1H), 4.62-4.60 (m, 1H), 4.48-4.45 (m, 1H), 4.27-4.19 (m, 3H), 1.40 (s, 9H), 1.30 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.64, 143.30, 143.15, 142.95, 137.30, 124.10, 123.39, 121.73, 121.45, 117.39, 117.16, 111.22, 71.50, 66.28, 65.79, 35.01, 34.27, 31.53, 29.90; IR: υ = 3057, 2961, 2857, 2878, 1591, 1493, 1235, 1061, 811, 749 cm^{-1}; EI-MS: *m/z* = 354 (M^+); HRMS (EI) calcd for $C_{23}H_{30}O_3$ 354.2195, found 354.2204.

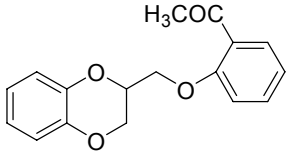


2-((4-chlorophenoxy)methyl)-2,3-dihydrobenzo[1,4]dioxine (**3g**): White solid, m.p. 81-82°C; ^{1}H NMR (400MHz, $CDCl_3$): δ 7.23 (d, *J* = 8.4 Hz, 2H), 6.89 (t, *J* = 4.8 Hz, 2H), 6.86-6.83 (m, 4H), 4.56-4.51 (m, 1H), 4.39-4.36 (m, 1H), 4.22-4.17 (m, 2H), 4.13-4.09 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.04, 143.20, 142.61, 129.40, 126.37, 121.79, 121.57, 117.35, 117.24, 115.84, 71.22, 66.61, 65.15; IR: υ = 3076, 3042, 2924, 2886, 2852, 1588, 1492, 1240, 1043, 853, 753 cm^{-1}; EI-MS: *m/z* = 276(M^+); HRMS (EI) calcd for $C_{15}H_{13}ClO_3$ 276.0553, found 276.0559.

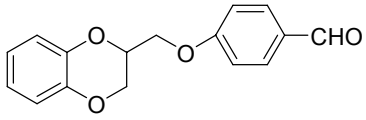


2-((2,4-dichlorophenoxy)methyl)-2,3-dihydrobenzo[1,4]dioxine (**3h**): White solid, m.p. 70-71°C; ^{1}H NMR (400MHz, $CDCl_3$): δ 7.37 (s, 1H), 7.18 (d, *J* = 8.4Hz, 1H), 6.92-6.85 (m, 5H), 4.61-4.59 (m, 1H), 4.45-4.42(m, 1H), 4.31-4.26 (m, 2H), 4.20-4.16 (m,

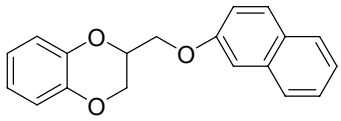
1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 152.72, 143.09, 142.70, 130.12, 127.62, 126.67, 124.11, 121.81, 121.63, 117.34, 117.30, 114.56, 71.05, 67.59, 65.05; IR: υ = 3087, 2962, 2935, 2891, 1593, 1490, 1243, 1038, 834, 748 cm^{-1} ; EI-MS: *m/z* = 310(M^+); HRMS (EI) calcd for $C_{15}H_{12}Cl_2O_3$ 310.1063, found 310.0170.



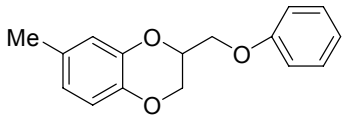
2-((2-acetylphenoxy)methyl)-2,3-dihydrobenzo[1,4]dioxine (**3i**): Oil; ^{1}H NMR (400MHz, $CDCl_3$): δ 7.72 (d, *J* = 7.6 Hz, 1H),7.44 (t, *J* = 7.6Hz, 1H), 7.02 (t, *J* = 7.6Hz, 1H), 6.94 (d, *J* = 8.4Hz, 1H), 6.90-6.84 (m, 4H), 4.63-4.59 (m, 1H), 4.41-4.38 (m, 1H), 4.33-4.20 (m, 3H), 2.63 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.42, 157.35, 142.96, 142.61, 133.60, 130.53, 128.63, 121.92, 121.67, 121.39, 117.36 117.28, 112.44, 71.19, 66.95, 65.16, 31.88; IR: υ = 3072, 2985, 2925, 2886, 1663, 1597, 1493, 1234, 1045, 831, 752 cm^{-1}; EI-MS: *m/z* = 284(M^+); HRMS (EI) calcd for $C_{17}H_{16}O_4$ 284.1049, found 284.1050.



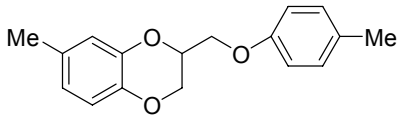
2-((4-formylphenoxy)methyl)-2,3-dihydrobenzo[1,4]dioxine (**3j**): White solid, m.p. 60-61°C[2]; ^{1}H NMR (400MHz, $CDCl_3$): δ 9.89 (s 1H), 7.85 (d, *J* = 8.0Hz, 2H), 7.04 (d, *J* = 8.0Hz, 2H), 6.92-6.87 (m, 4H), 4.62-4.58 (m, 1H), 4.41-4.38 (m, 1H), 4.34-4.30 (m, 1H), 4.26-4.22 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.71, 163.11, 143.00, 142.63, 131.97, 130.46, 121.89, 121.68, 117.38, 117.30, 114.81, 71.06, 66.46, 64.97; IR: υ = 3038, 2907, 2883, 2742, 1685, 1598, 1490, 1243, 1046, 830, 749 cm^{-1}.



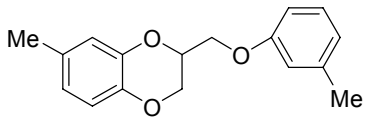
2-((naphthalen-2-yloxy)methyl)-2,3-dihydrobenzo[1,4]dioxine (**3k**): White solid, m.p. 80-81°C; ^{1}H NMR (400MHz, $CDCl_3$): δ 7.81-7.74 (m, 3H), 7.48 (t, *J* = 7.6Hz, 1H), 7.39 (t, *J* = 7.6Hz, 1H), 7.26-7.19 (m, 2H), 7.00-6.95 (m, 2H), 6.93-6.90 (m, 2H), 4.67-4.63 (m, 1H), 4.48-4.45 (m, 1H), 4.39 (dd, *J* = 4.8, 10.0 Hz, 1H), 4.31-4.26 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.23, 143.18, 142.92, 134.39, 129.59, 129.23, 127.67, 126.82, 126.53, 123.96, 121.81, 121.56, 118.63, 117.46, 117.30, 106.91, 71.36, 66.33, 65.34; IR: υ = 3051, 2977, 2919, 1593, 1492, 1250, 1055, 848, 747 cm^{-1}; EI-MS: *m/z* = 292(M^+); HRMS (EI) calcd for $C_{19}H_{16}O_3$ 292.1099, found 292.1105.



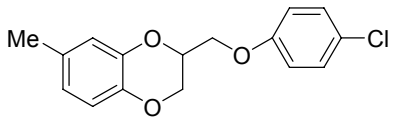
7-methyl-2-(phenoxymethyl)-2,3-dihydrobenz[1,4]dioxine (**3l**): White solid, m.p. 55-56°C[3]; ^{1}H NMR (400MHz, $CDCl_3$): δ 7.33 (t, *J* = 8.0Hz, 2H), 7.02 (t, *J* = 7.2 Hz, 1H), 6.96 (d, *J* = 8.0 Hz, 2H), 6.82 (d, *J* = 8.4 Hz, 1H), 6.78 (s, 1H), 6.69 (d, J = 8.0 HZ, 1H), 4.59-4.55 (m, 1H), 4.40 (dd, *J* = 2.0, 11.2 Hz, 1H), 4.29-4.15 (m, 3H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.29, 142.44, 140.86, 131.42, 129.54, 122.06, 121.35, 117.70, 116.85, 114.55, 71.37, 66.22, 65.29, 20.66; IR: υ = 3103, 2973, 2921, 2875, 1588, 1499, 1239, 1053, 800, 750 cm^{-1}.



7-methyl-2-(p-tolyloxymethyl)-2,3-dihydrobenzo[1,4]dioxine (**3m**): White solid, m.p. 80-81°C; ^{1}H NMR (400MHz, $CDCl_3$): δ 7.12 (d, *J* = 8.4 Hz, 2H), 6.86 (d, *J* = 8.4Hz, 2H), 6.81 (d, *J* = 7.2 Hz, 1H), 6.77 (s, 1H), 6.68 (d, *J* = 8.0 Hz, 1H), 4.57-4.53 (m, 1H), 4.40-4.38 (m, 1H), 4.26-4.18 (m, 2H), 4.15-4.10 (m, 1H), 2.32 (s, 3H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.23, 142.48, 140.87, 131.39, 130.62, 129.96, 122.02, 117.71, 116.83, 114.43, 71.42, 66.45, 65.34, 20.66, 20.45; IR: υ = 3034, 2992, 2926, 2901, 2875, 1589, 1507, 1240, 1058, 842, 747 cm^{-1}; EI-MS: *m/z* = 270(M^+); HRMS (EI) calcd for $C_{17}H_{18}O_3$ 270.1256, found 270.1258.

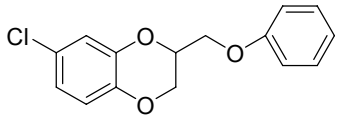


7-methyl-2-(m-tolyloxymethyl)-2,3-dihydrobenzo[1,4]dioxine (**3n**): White solid, m.p. 50-51°C; ^{1}H NMR (400MHz, $CDCl_3$): δ 7.21 (t, *J* = 7.6Hz, 1H), 6.83-6.81 (m, 2H), 6.78-6.76 (m, 3H), 6.69 (d, *J* = 8.0Hz, 1H), 4.58-4.56 (m, 1H), 4.41-4.38 (m, 1H), 4.28-4.19 (m, 2H), 4.17-4.13 (m, 1H), 2.37 (s, 3H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.29, 142.44, 140.85, 139.62, 131.41, 129.26, 122.16, 122.02, 117.70, 116.83, 115.37, 111.40, 71.36, 66.14, 65.30, 21.47, 20.65; IR: υ = 3034, 2951, 2921, 2879, 1583, 1506, 1255, 1060, 802, 767, 747 cm^{-1}; EI-MS: *m/z* = 270(M^+); HRMS (EI) calcd for $C_{17}H_{18}O_3$ 270.1256, found 270.1253.



2-((4-chlorophenoxy)methyl)-7-methyl-2,3-dihydrobenzo[1,4]

dioxine (**3o**): White solid, m.p. 84-85°C; ^{1}H NMR (400MHz, $CDCl_3$): δ 7.25 (d, *J* = 8.8 Hz, 2H), 6.86 (d, *J* = 8.8Hz, 2H), 6.79 (d, *J* = 8.4Hz, 1H), 6.74 (s, 1H), 6.67 (d, *J* = 8.0Hz, 1H), 4. 56-4.52 (m, 1H), 4.36 (m, 1H), 4.23-4.17 (m, 2H), 4.12 (dd, *J* = 6.4, 9.8Hz, 1H), 2.26 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.90, 142.29, 140.82, 131.51, 129.39, 126.29, 122.11, 117.67, 116.85, 115.84, 71.25, 66.61, 65.10, 20.63; IR: υ = 3053, 2958, 2928, 2867, 1590, 1492, 1242, 1012, 822, 748 cm^{-1}; EI-MS: *m/z* = 290(M^+); HRMS (EI) calcd for $C_{16}H_{15}ClO_3$ 290.0710, found 290.0710.



7-chloro-2-(phenoxymethyl)-2,3-dihydrobenzo[1,4]dioxine (**3p**)[3]: Oil; ^{1}H NMR (400MHz, $CDCl_3$): δ 7.31 (t, *J* =7.6Hz, 2H), 7.00, (t, *J* = 7.2 Hz, 1H), 6.94-6.92 (m, 3H), 6.83 (br, 2H), 4.58-4.53 (m, 1H), 4.41-4.39 (m, 1H), 4.27-4.19 (m, 2H), 4.16-4.12 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.12, 143.40, 141.91, 129.59, 126.13 121.50, 121.48, 118.03, 117.49, 114.49, 71.44, 65.97, 65.23; IR: υ = 3072, 2981, 2926, 2882, 1585, 1487, 1237, 1031, 802, 752 cm^{-1}.

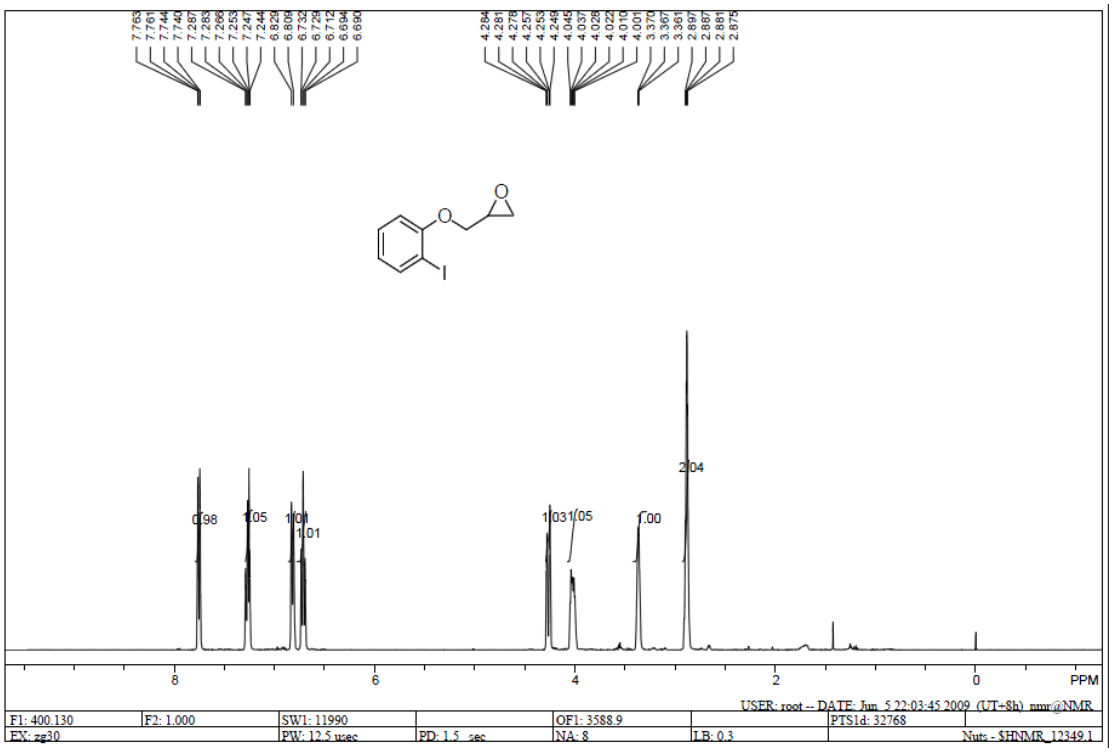
C. References

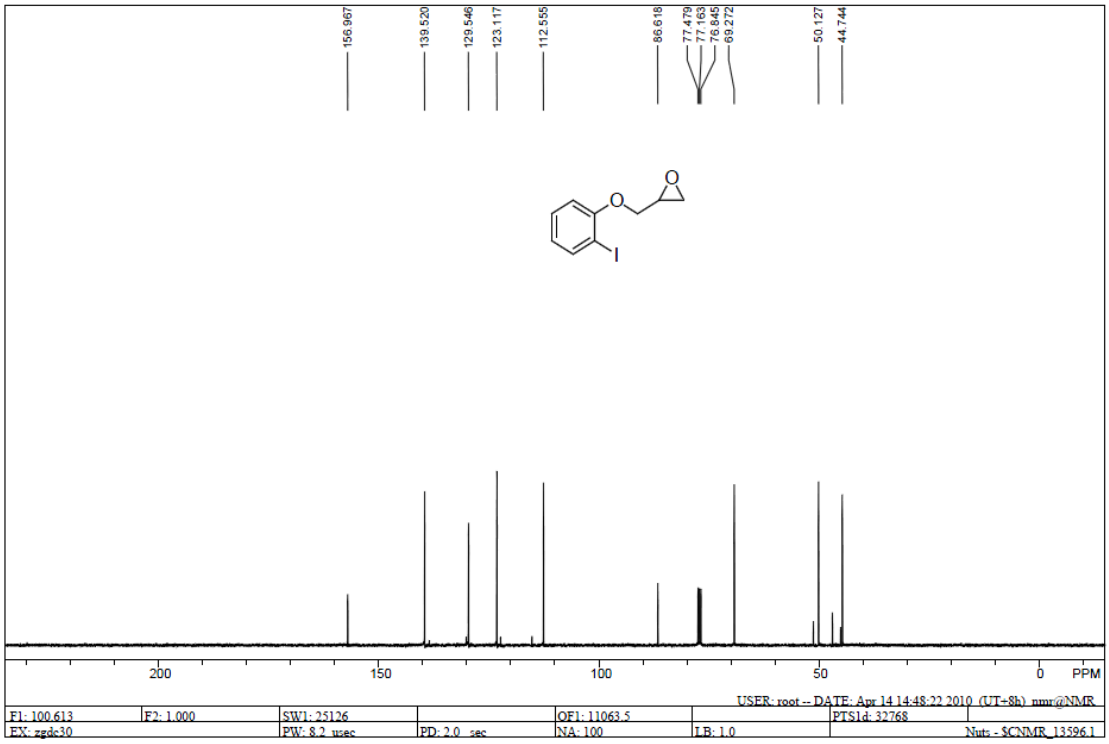
1. S. Wagner, K. Kopka, M. P. Law, B. Riemann, V. W. Pike, O. Schober, M. Schäfers, *Bioorg. Med. Chem.* **2004,** *12,* 4117.

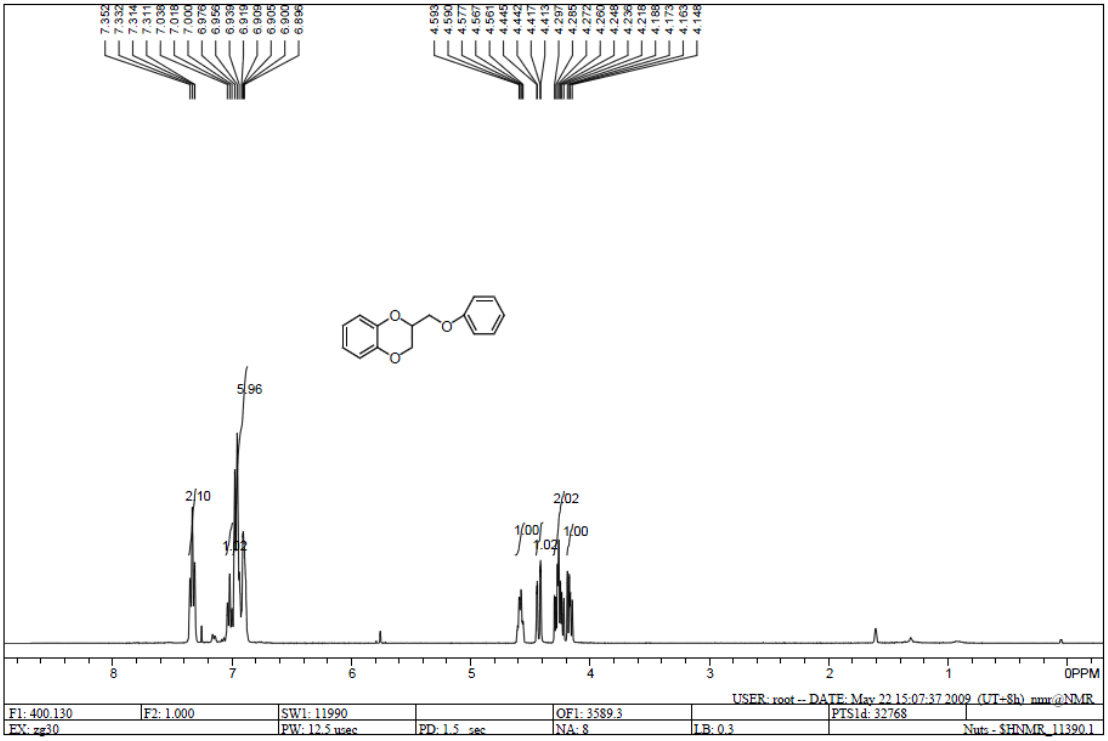
2. L. Juha′sz, T. Docsa, A. Brunya′szki, P. Gergely and S. Antus, *Bioorg. Med. Chem.* **2007**, *15*, 4048.

3. W. L. Bao, Y.Y. Liu, X. Lv and W. X. Qian, *Org. Lett.* **2008**, *10*, 3899.

D. Copies of ^{1}H NMR and ^{13}C NMR spectra



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6.909
6.905
6.900
6.896
4.593
4.590
4.577
4.567
4.561
4.445
4.442
4.417
4.413
4.287
4.285
4.272
4.269
4.249
4.245
4.218
4.188
4.173
4.163
4.148
5.96
2.10
1.92
2.02
1.00
1.02
1.00
8
7
6
5
4
3
2
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PD: 1.5 sec
NA: 8
LB: 0.3
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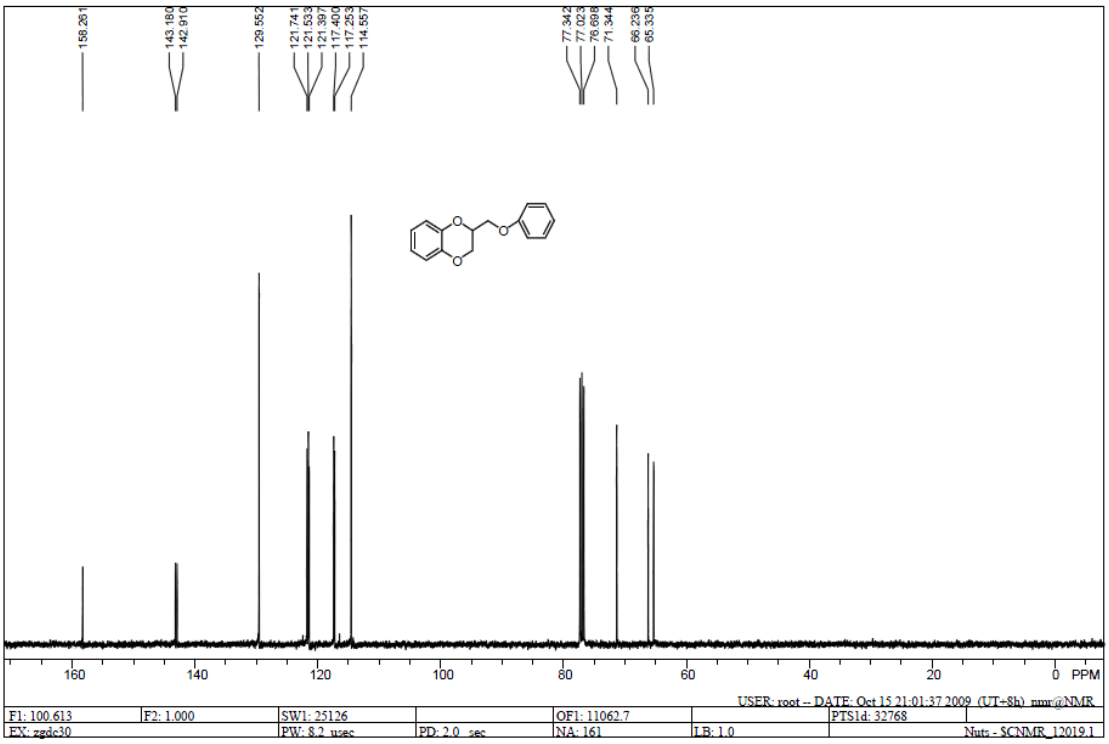
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76.698
71.344
66.236
65.335
160
140
120
100
80
60
40
20
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OF1: 11062.7
PTS1d: 32768
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PW: 8.2 usec
PD: 2.0 sec
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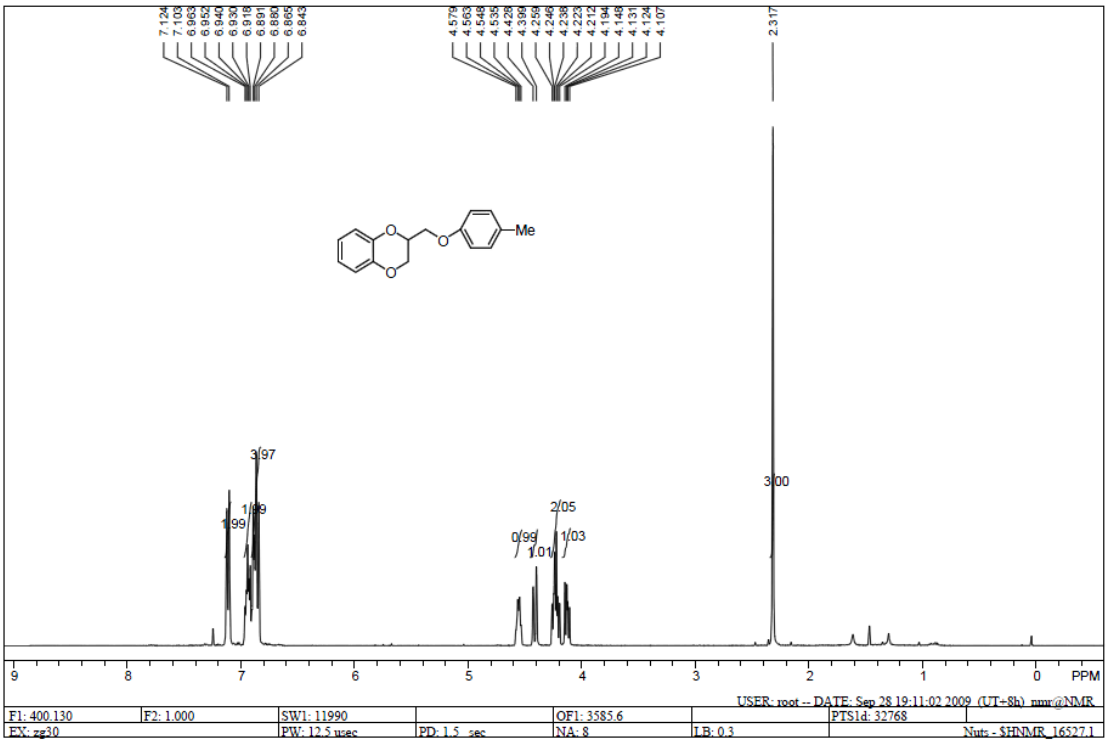
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6.918
6.891
6.880
6.865
6.843
4.579
4.563
4.548
4.535
4.428
4.399
4.259
4.246
4.238
4.223
4.212
4.194
4.148
4.131
4.124
4.107
2.317
Me
3.97
1.89
1.99
2.05
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1.01
1.03
3.00
9
8
7
6
5
4
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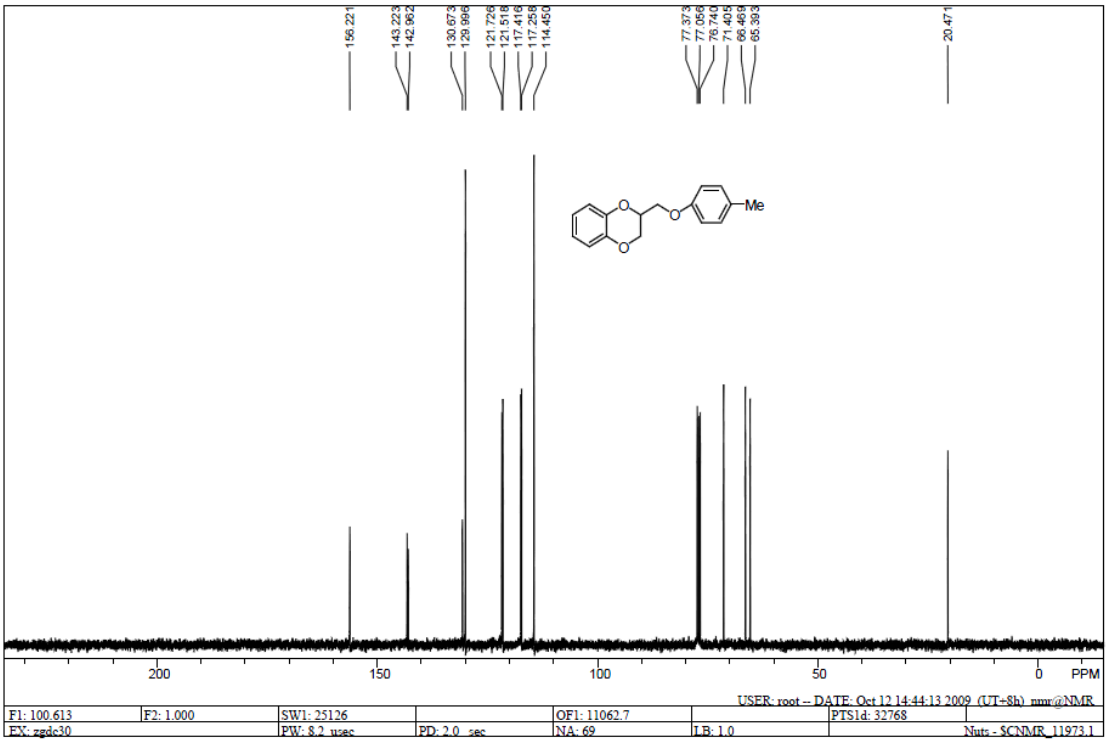
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76.740
71.405
68.469
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20.471
Me
200
150
100
50
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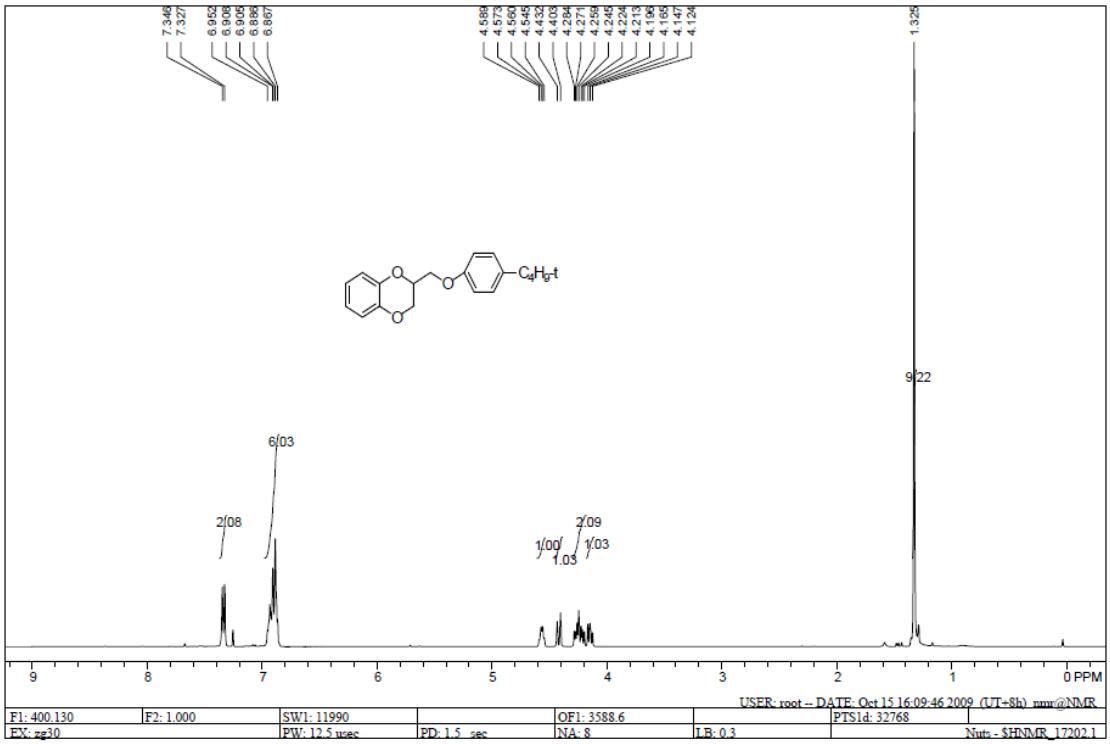
PD: 2.0 sec
NA: 69
LB: 1.0
Nuts - $CNMR_11973.1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4.560
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4.432
4.403
4.284
4.271
4.259
4.245
4.224
4.213
4.196
4.165
4.147
4.124
1.325
C4H9-t
9.22
6.03
2.08
2.09
1.00
1.03
1.05
9
8
7
6
5
4
3
2
1
0 PPM
USER: root -- DATE: Oct 15 16:09:46 2009 (UT+8h) nmr@NMR
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F2: 1.000
SW1: 11990
OF1: 3588.6
PTS1d: 32768
EX: zg30
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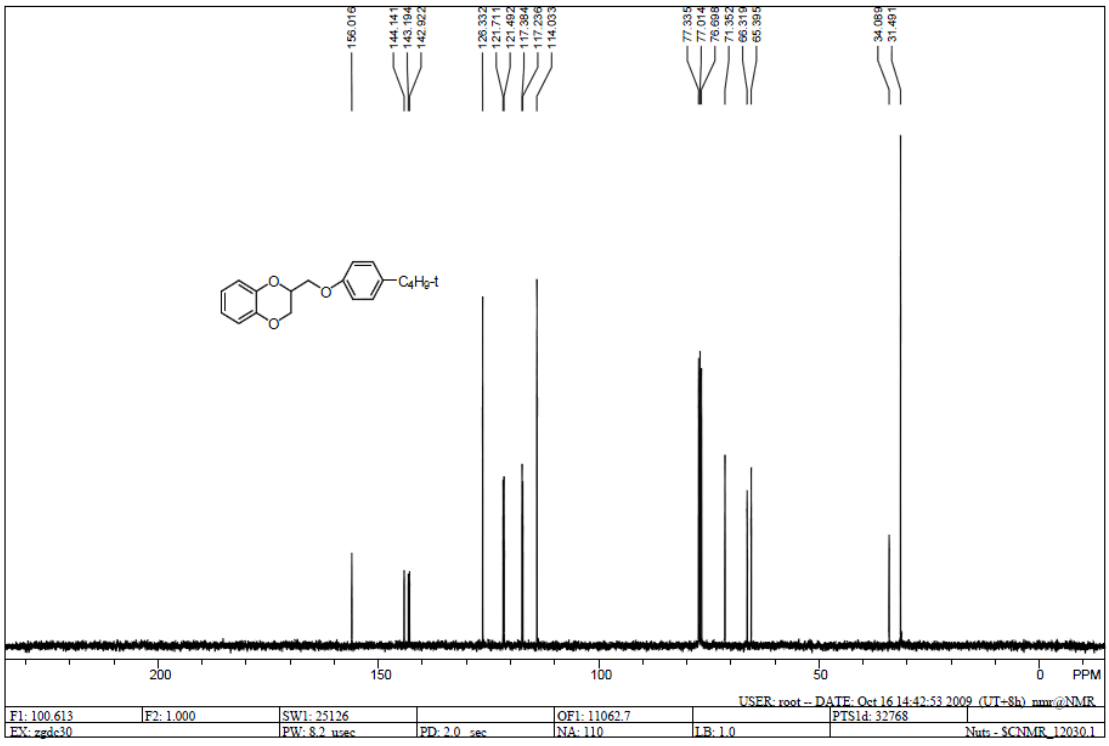
PD: 1.5 sec
NA: 8
LB: 0.3
Nuts - $HNMR_17202.1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117.236
114.033
77.335
77.014
76.698
71.352
68.319
65.395
34.089
31.491
C4H9-t
200
150
100
50
0 PPM
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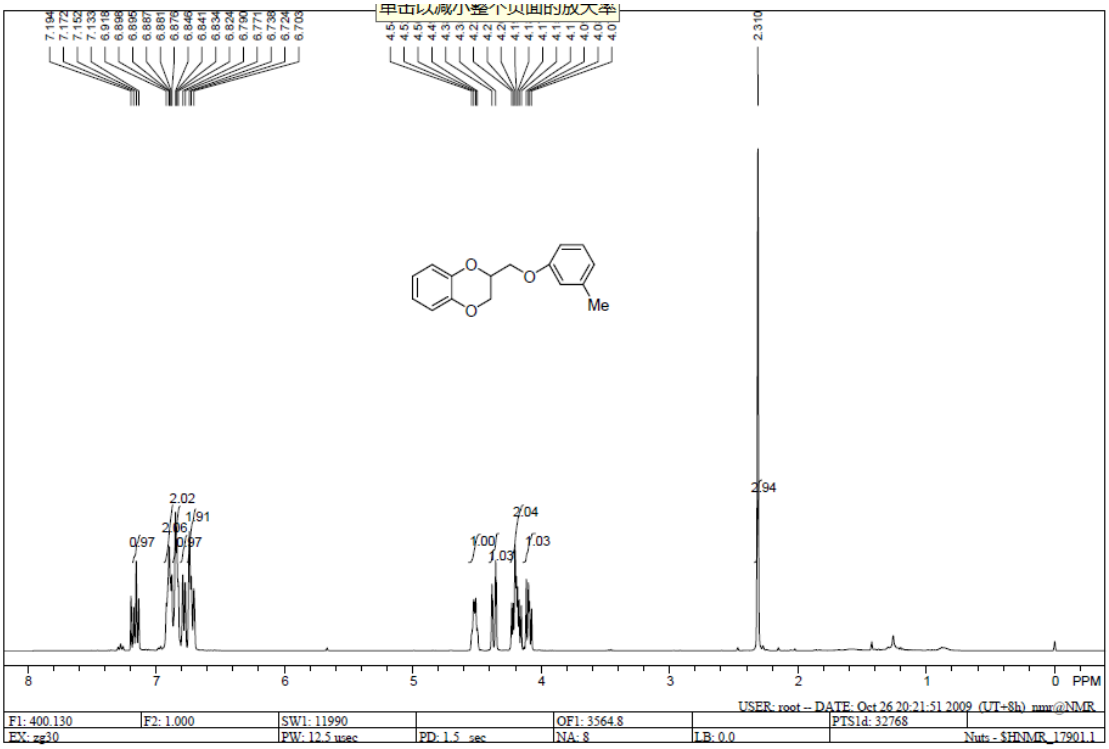
F2: 1.000
SW1: 25126
OF1: 11062.7
PTS1d: 32768
EX: zgdc30
PW: 8.2 usec
PD: 2.0 sec
NA: 110
LB: 1.0
Nuts - $CNMR_12030.1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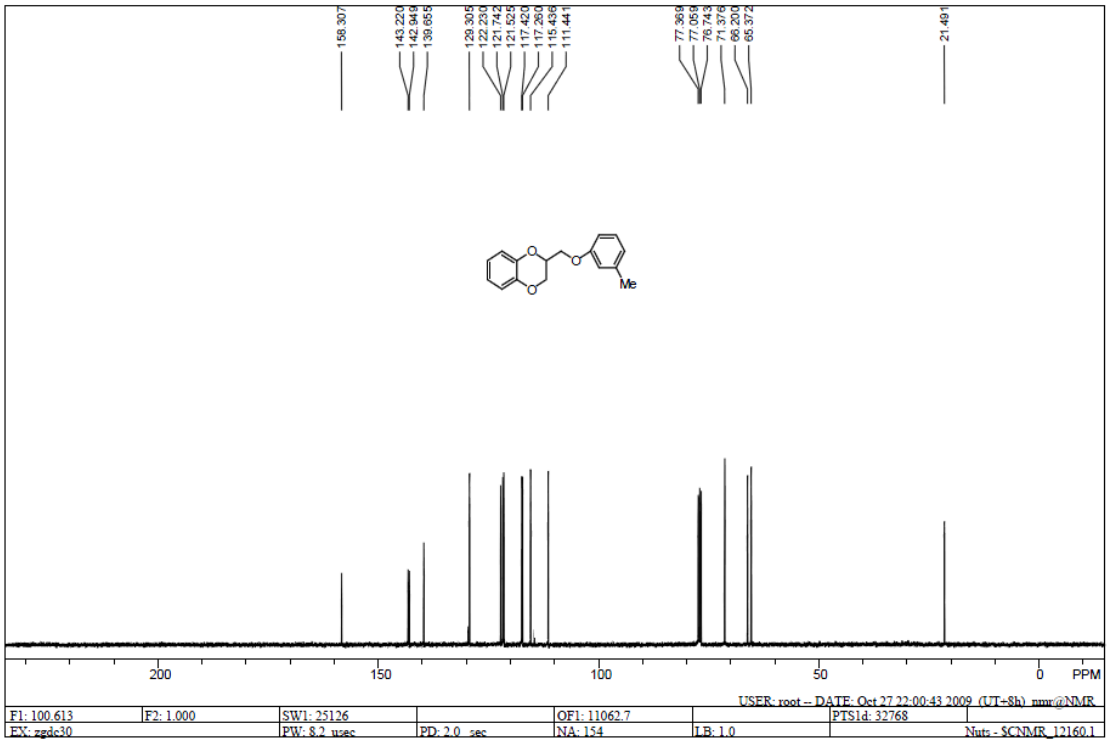
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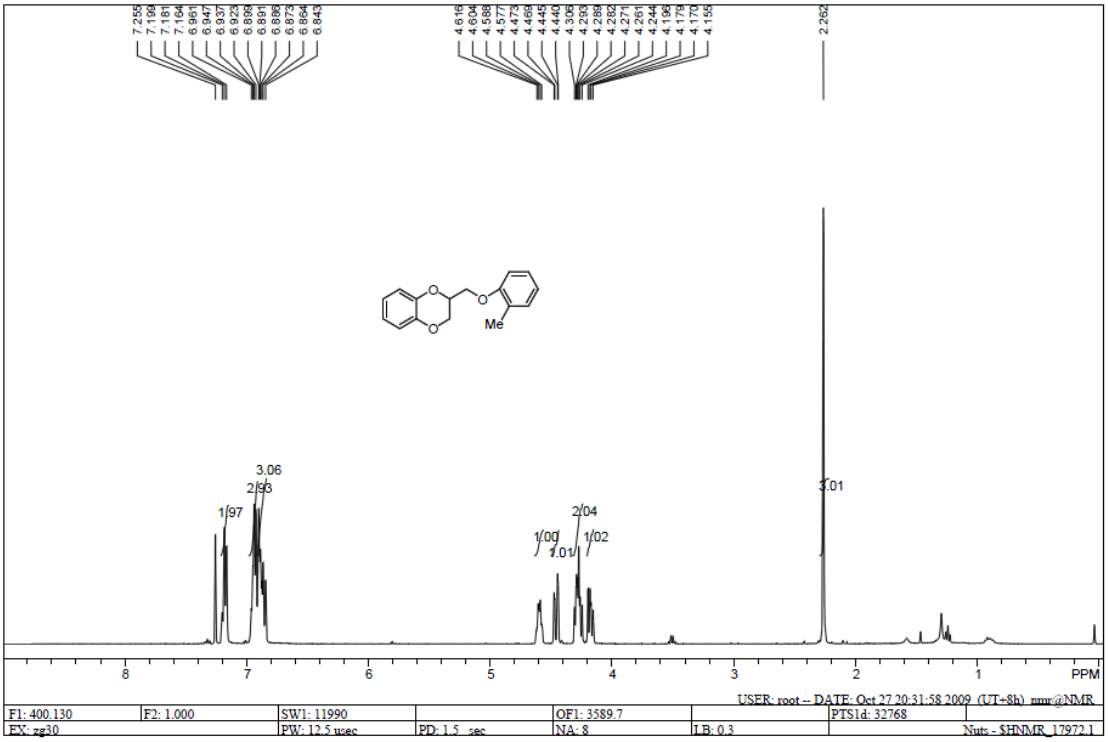
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6.843
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4.282
4.271
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4.244
4.196
4.179
4.170
4.155
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2.93
1.97
2.04
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1.01
1.02
3.01
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6
5
4
3
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LB: 0.3
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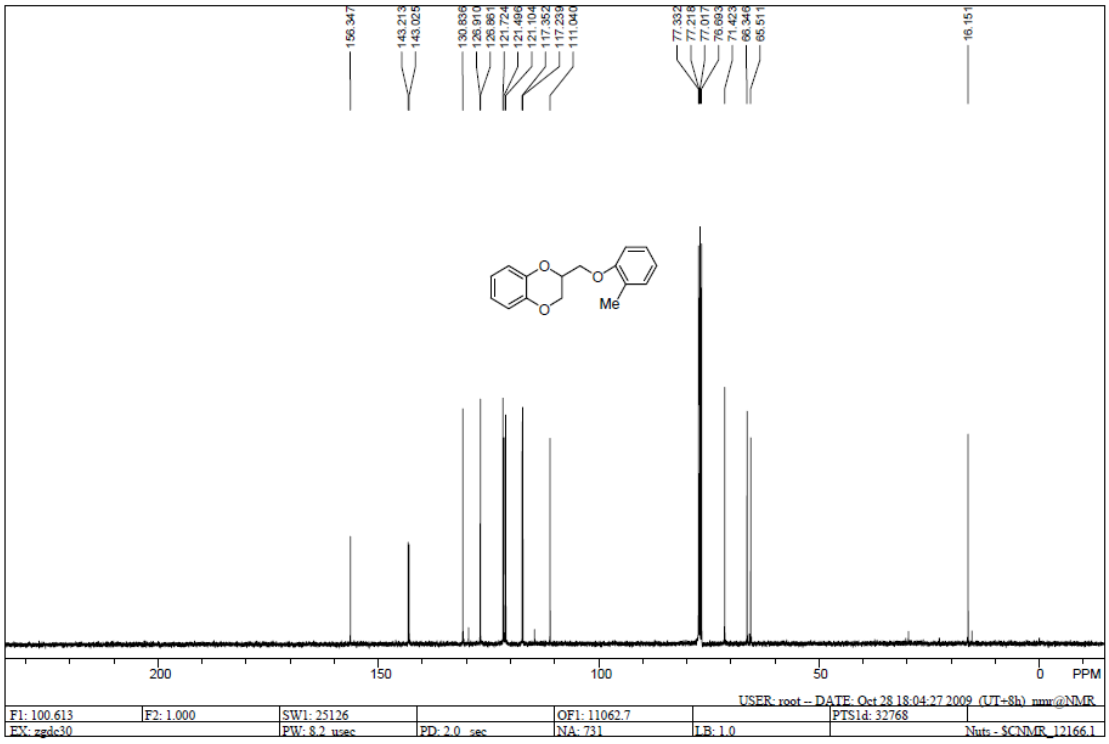
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77.218
77.017
76.693
71.423
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65.511
16.151
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SW1: 25126
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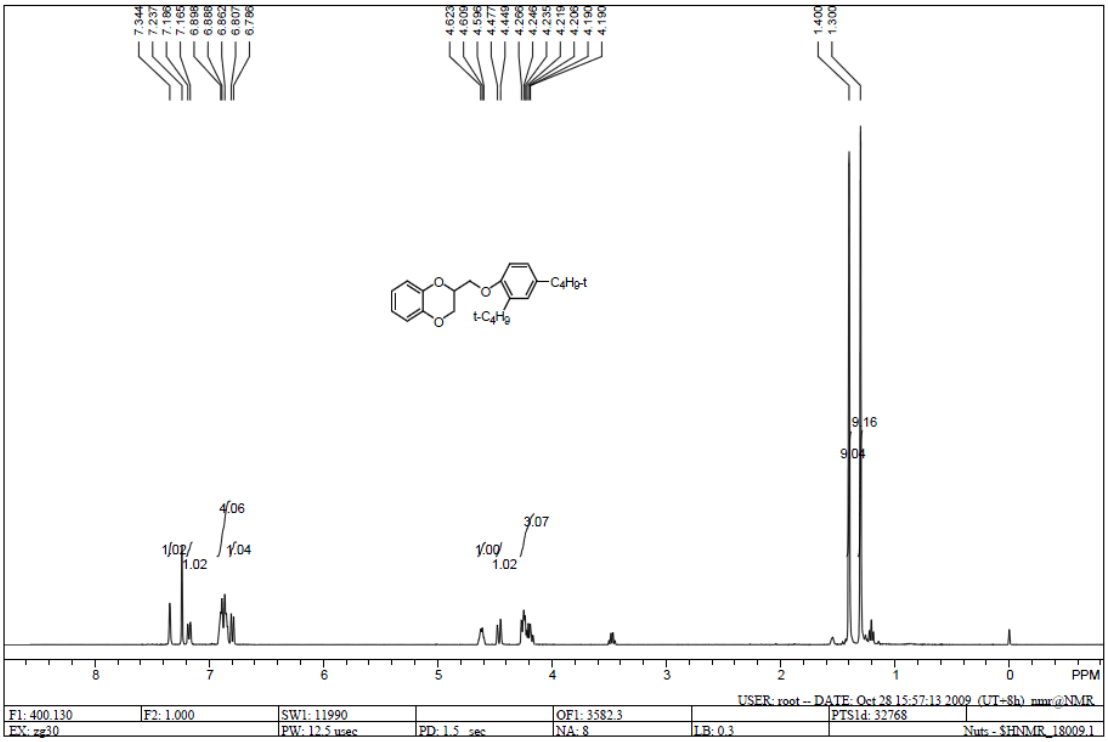
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1.300
C4H9-t
t-C4H9
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9.04
4.06
3.07
1.02
1.04
1.00
1.02
8
7
6
5
4
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SW1: 11990
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PW: 12.5 usec
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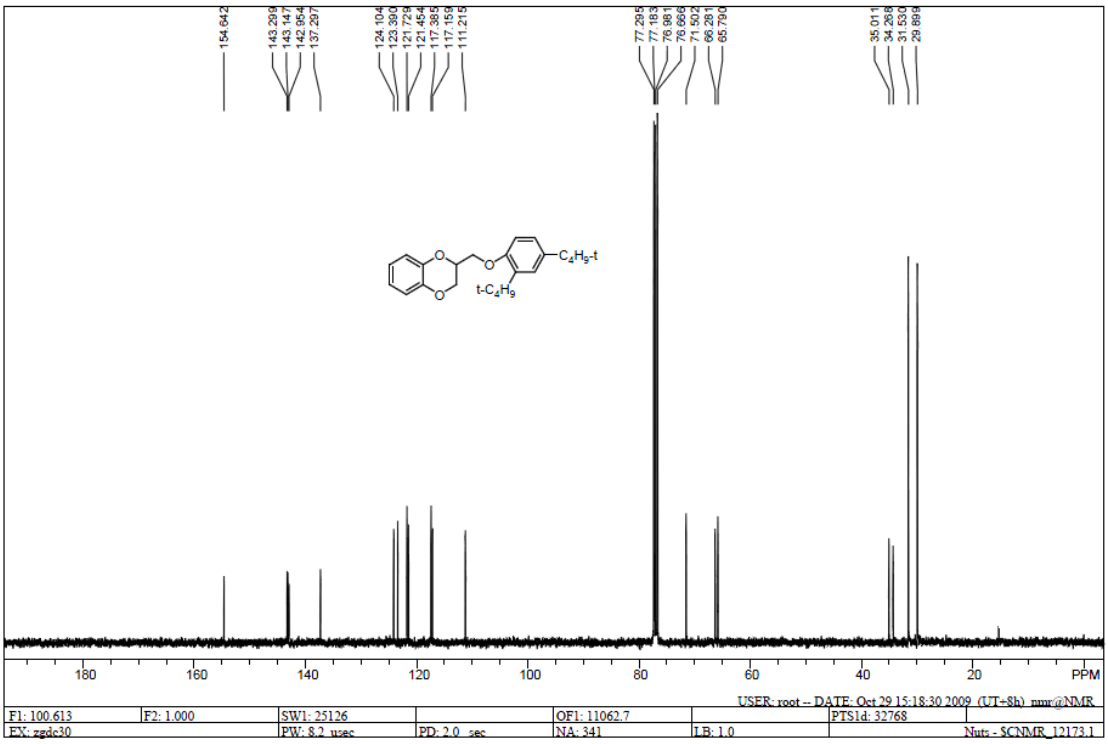
NA: 8
LB: 0.3
Nuts - $HNMR_18009.1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77.295
77.183
76.881
76.666
71.502
66.281
65.790
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34.268
31.530
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t-C4H9
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160
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120
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80
60
40
20
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LB: 1.0
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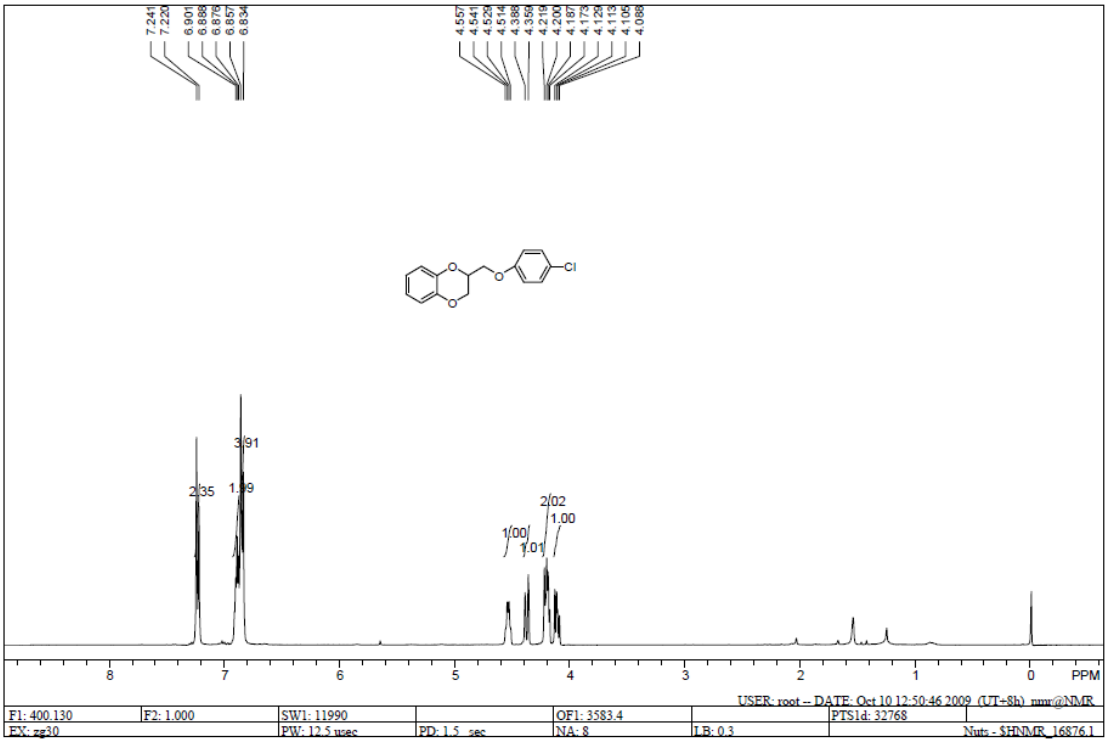
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4.187
4.173
4.129
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4.105
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O
O
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2.02
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1.00
1.01
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4
3
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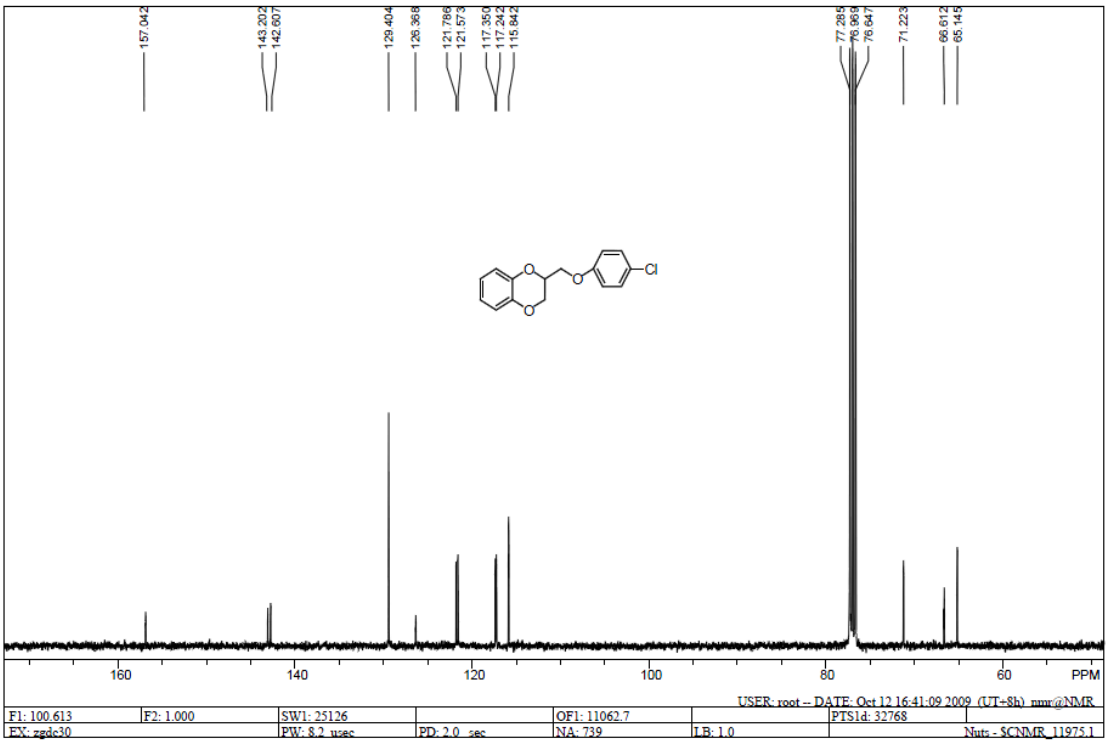
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76.647
71.223
66.612
65.145
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140
120
100
80
60
PPM
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OF1: 11062.7
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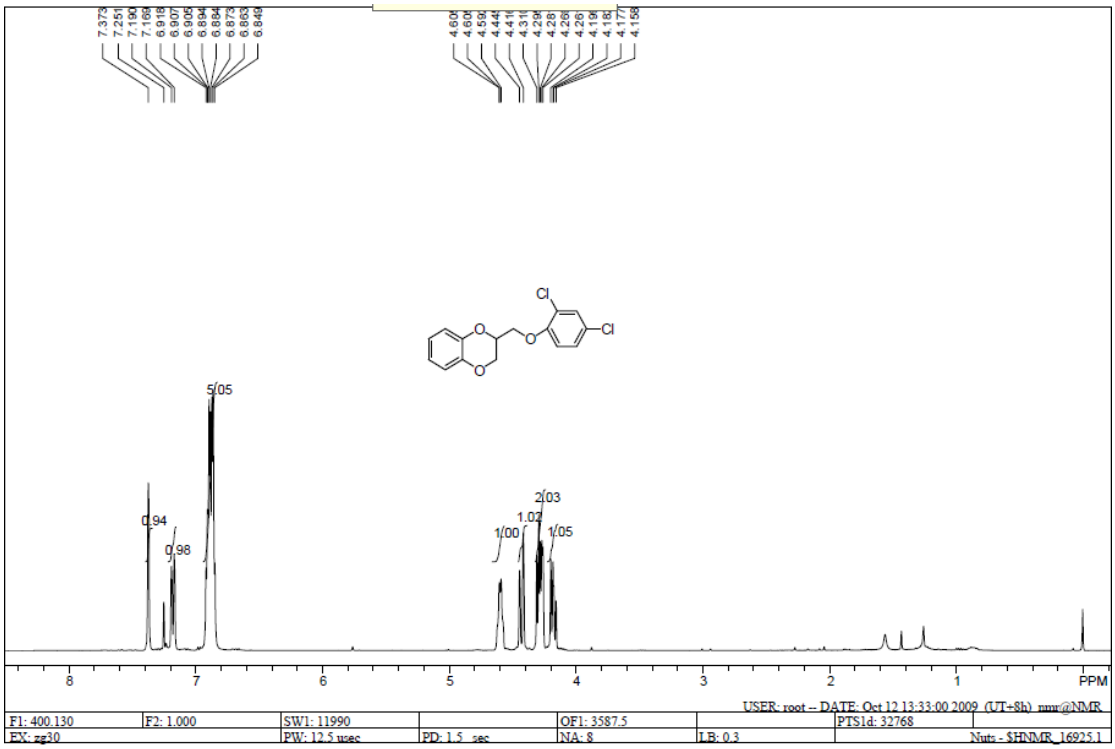
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6.894
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4.601
4.592
4.444
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4.311
4.294
4.281
4.261
4.251
4.194
4.182
4.177
4.158
Cl
Cl
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O
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0.94
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1.02
1.00
1.05
8
7
6
5
4
3
2
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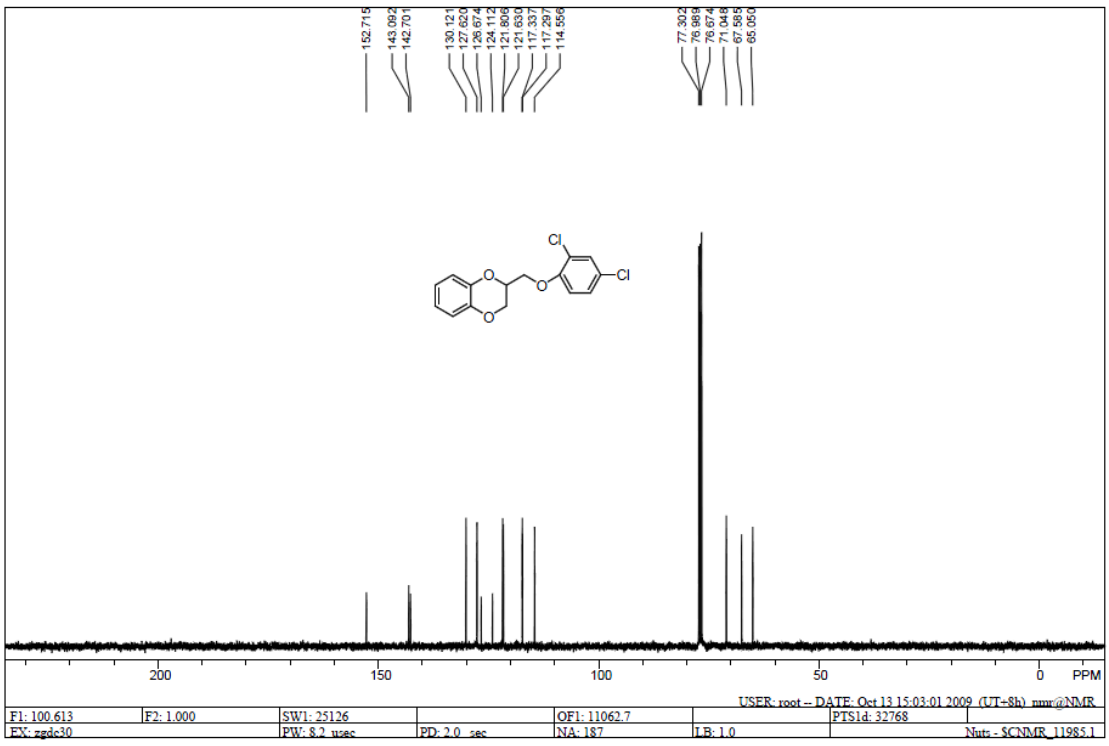
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117.337
117.297
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77.302
76.989
76.674
71.048
67.585
65.050
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Cl
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O
O
200
150
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50
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PPM
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F2: 1.000
SW1: 25126
OF1: 11062.7
PTS1d: 32768
EX: zgdc30
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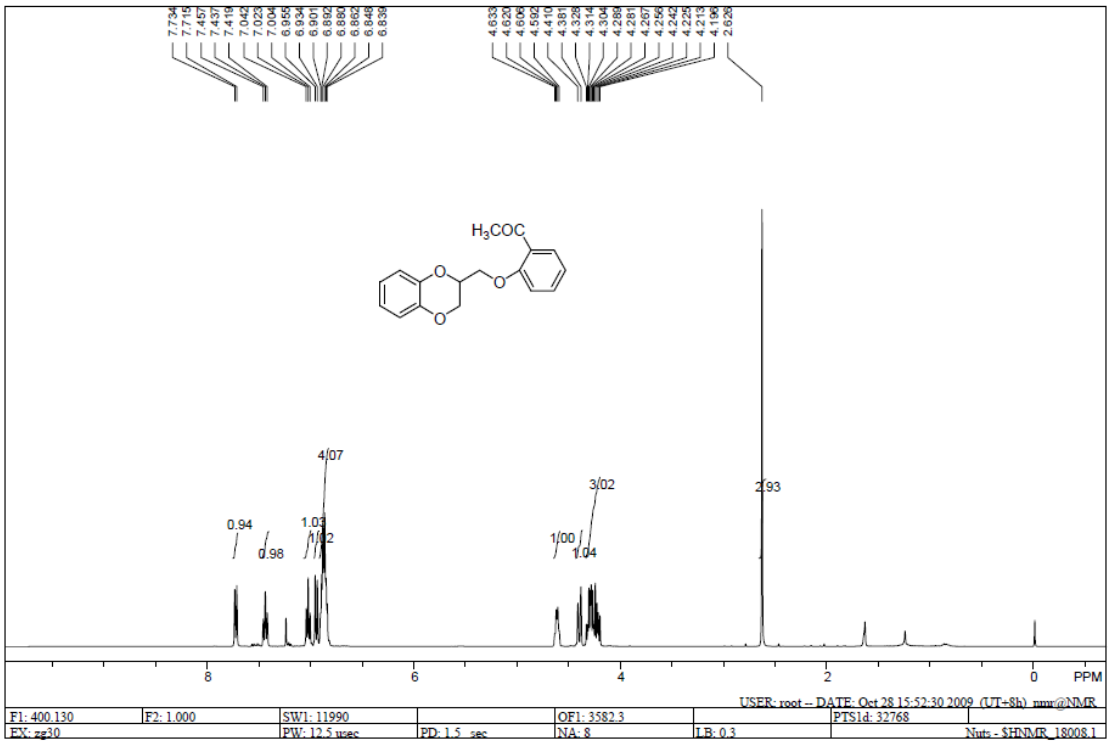
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4.242
4.225
4.213
4.199
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H3COC
4.07
3.02
2.93
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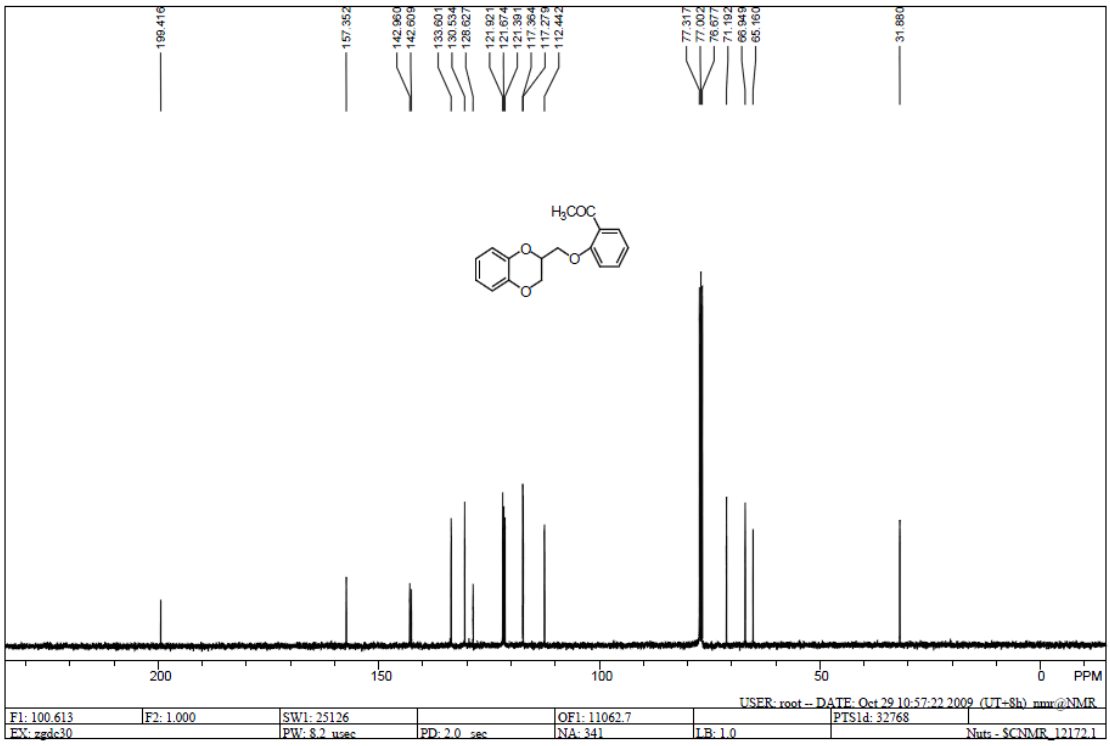
0.98
1.03
1.02
1.00
1.04
8
6
4
2
0
PPM
USER: root -- DATE: Oct 28 15:52:30 2009 (UT+8h) nmr@NMR
F1: 400.130
F2: 1.000
SW1: 11990
OF1: 3582.3
PTS1d: 32768
EX: zg30
PW: 12.5 usec
PD: 1.5 sec
NA: 8
LB: 0.3
Nuts - $HNMR_18008.1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130.534
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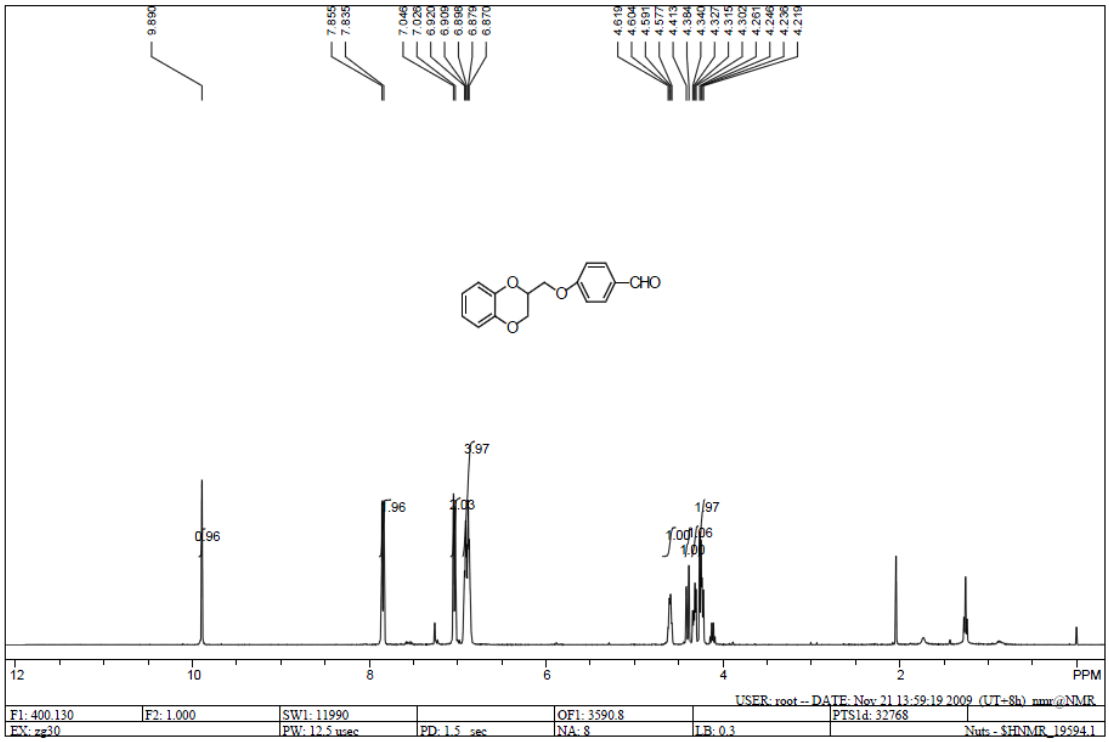
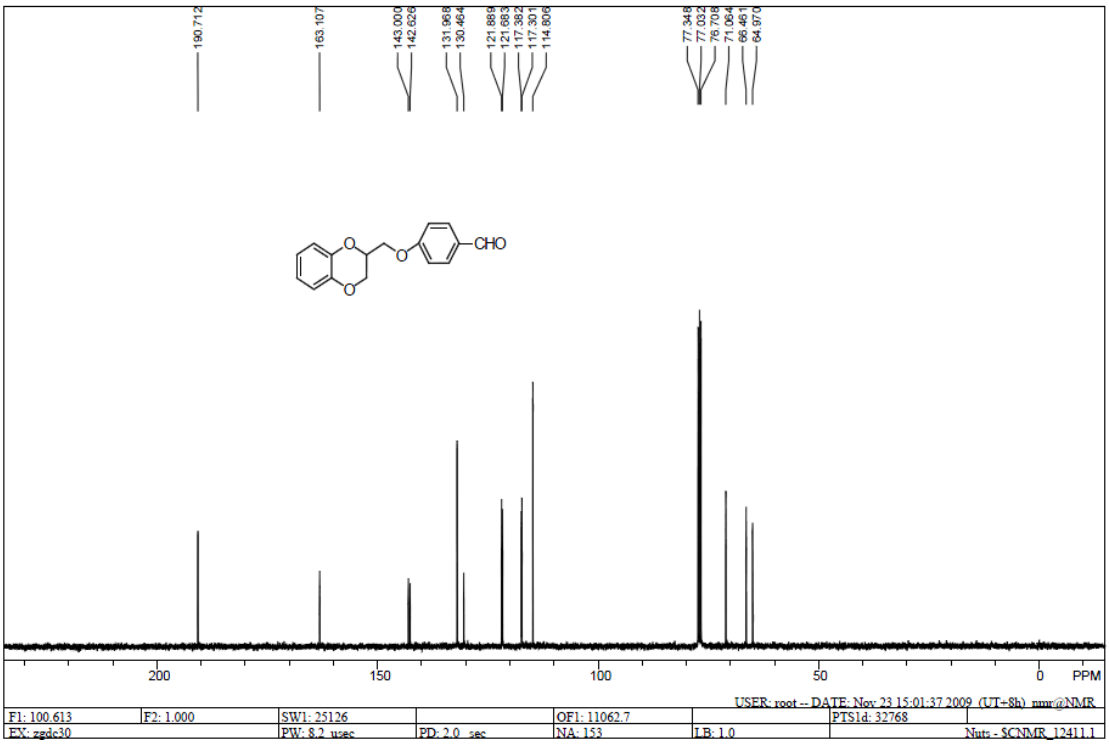
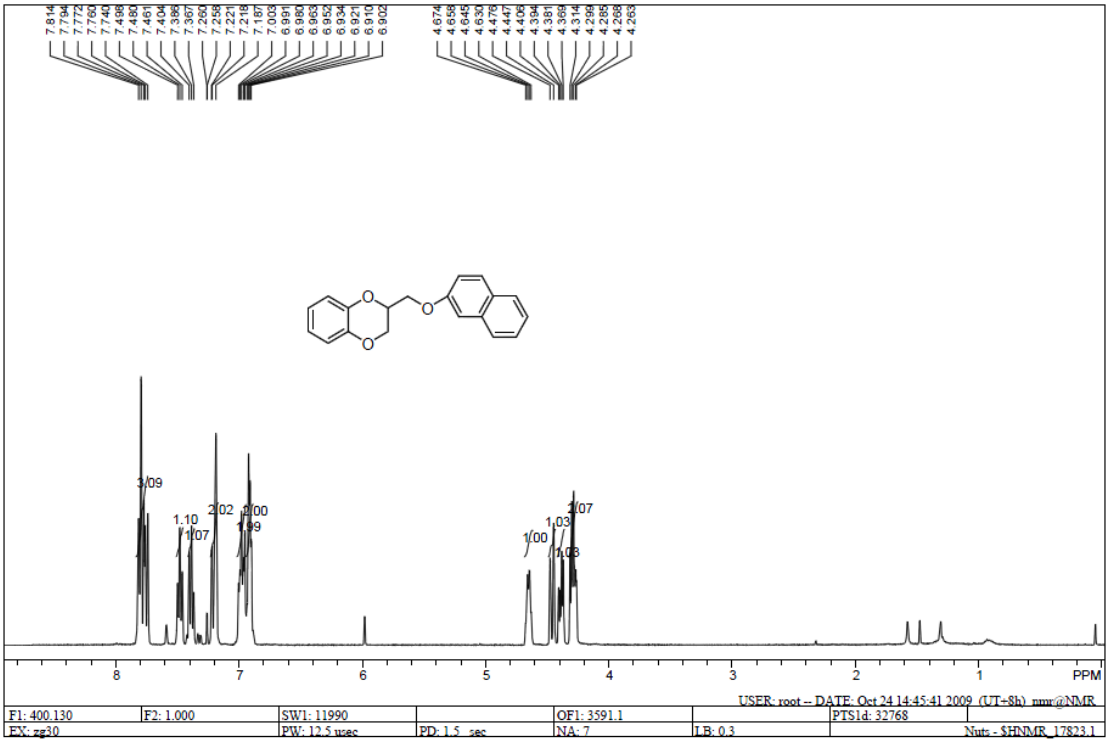
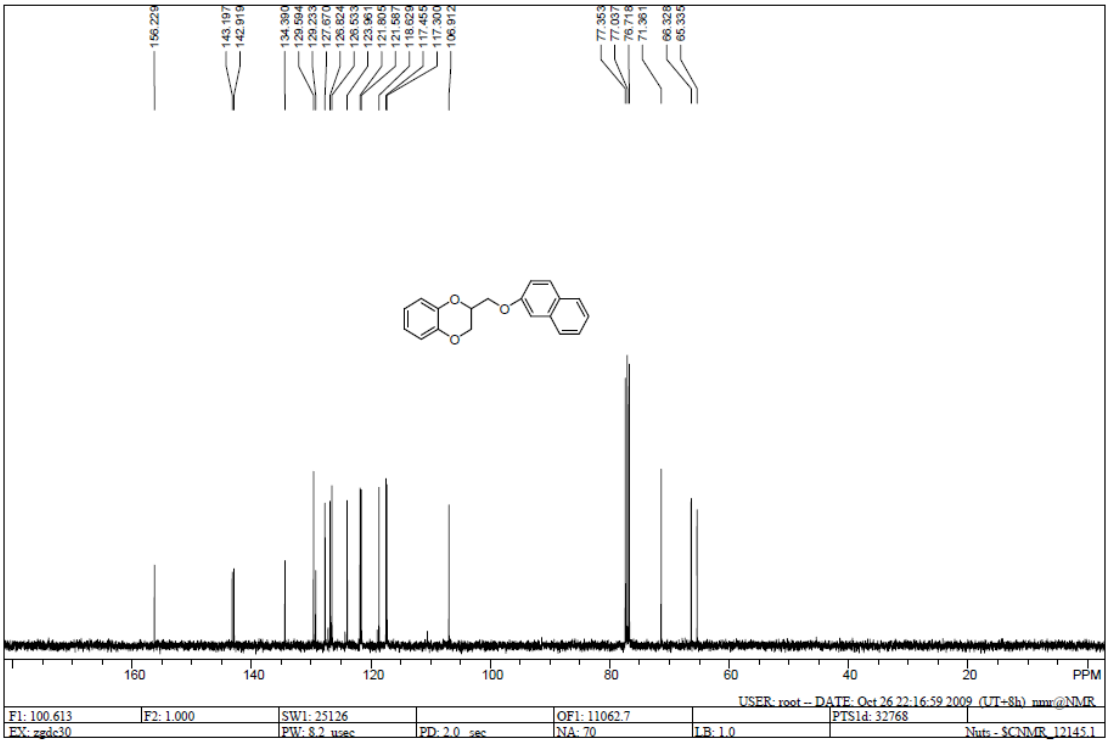
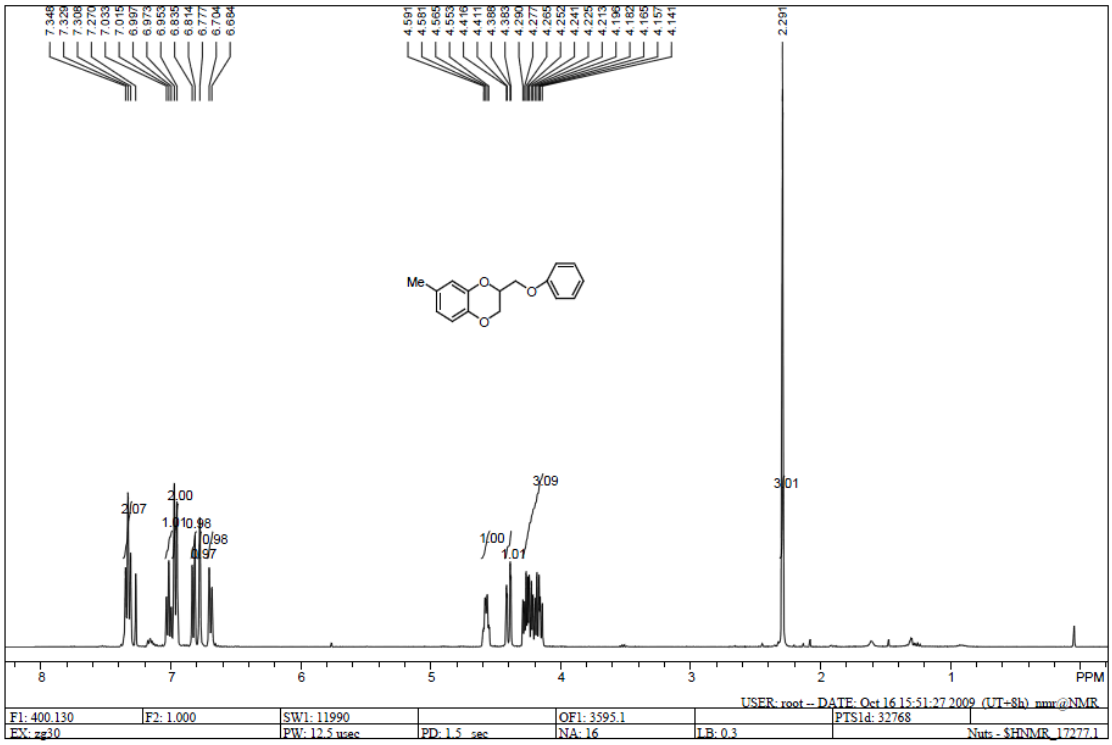
121.921
121.674
121.384
117.384
117.279
112.442
77.317
77.002
76.677
71.192
68.949
65.160
31.880
H3COC
200
150
100
50
0
PPM
USER: root -- DATE: Oct 29 10:57:22 2009 (UT+8h) nmr@NMR
F1: 100.613
F2: 1.000
SW1: 25126
OF1: 11062.7
PTS1d: 32768
EX: zgdc30
PW: 8.2 usec
PD: 2.0 sec
NA: 341
LB: 1.0
Nuts - $CNMR_12172.1

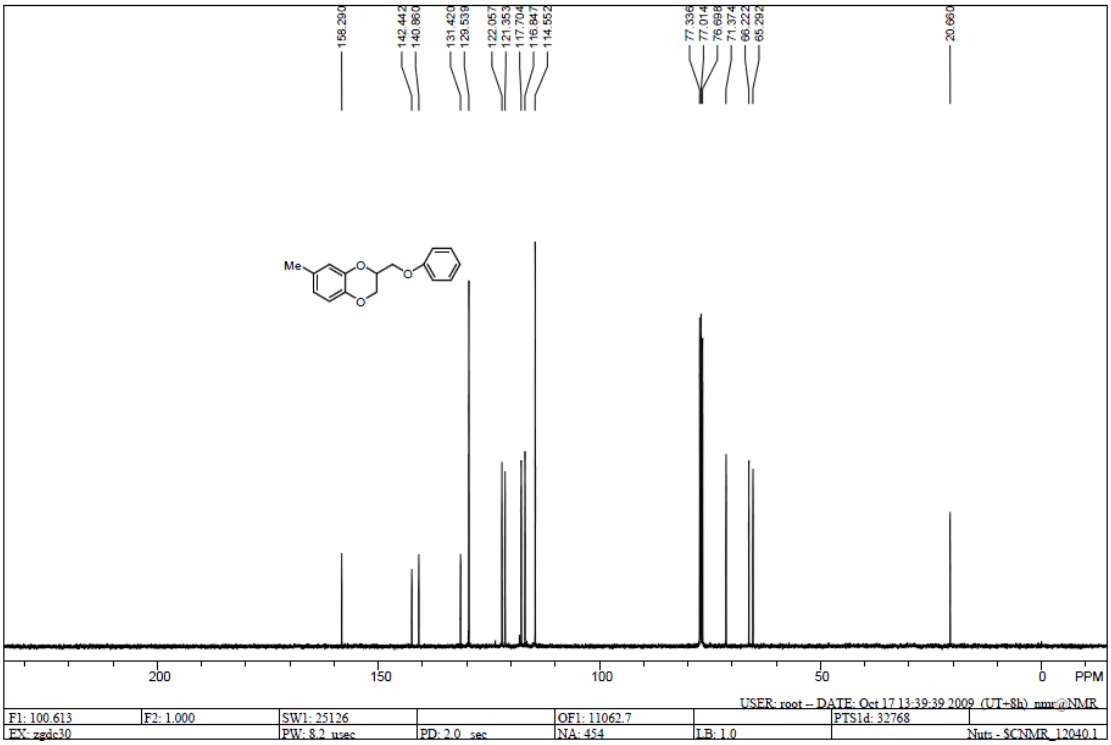

9.890
7.855
7.835
7.046
7.028
6.920
6.909
6.898
6.879
6.870
4.619
4.604
4.591
4.577
4.413
4.384
4.340
4.327
4.315
4.302
4.261
4.246
4.236
4.219
CHO
3.97
0.96
1.96
2.03
1.97
1.00
1.06
1.00
12
10
8
6
4
2
PPM
USER: root -- DATE: Nov 21 13:59:19 2009 (UT+8h) nmr@NMR
F1: 400.130
F2: 1.000
SW1: 11990
OF1: 3590.8
PTS1d: 32768
EX: zg30
PW: 12.5 usec
PD: 1.5 sec
NA: 8
LB: 0.3
Nuts - $HNMR_19594.1

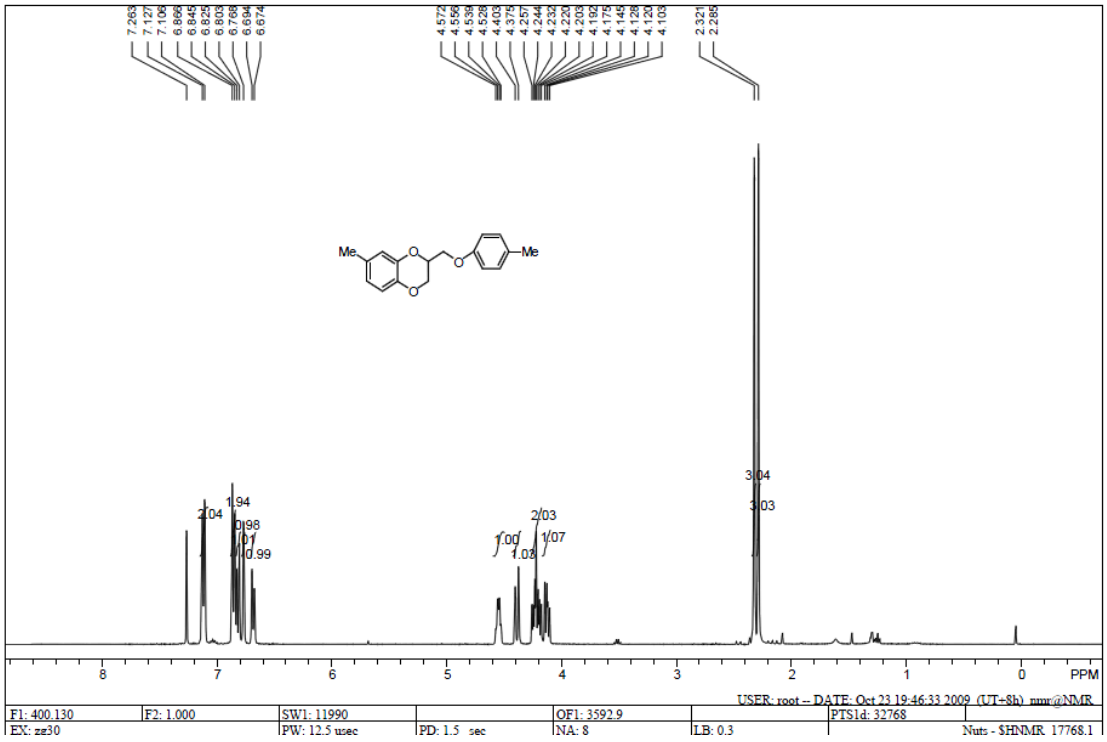

190.712
163.107
143.000
142.626
131.968
130.464
121.889
121.683
117.382
117.301
114.806
77.348
77.032
76.708
71.084
69.461
64.970
CHO
200
150
100
50
0
PPM
USER: root -- DATE: Nov 23 15:01:37 2009 (UT+8h) nmr@NMR
F1: 100.613
F2: 1.000
SW1: 25126
OF1: 11062.7
PTS1d: 32768
EX: zgdc30
PW: 8.2 usec
PD: 2.0 sec
NA: 153
LB: 1.0
Nuts - $CNMR_12411.1

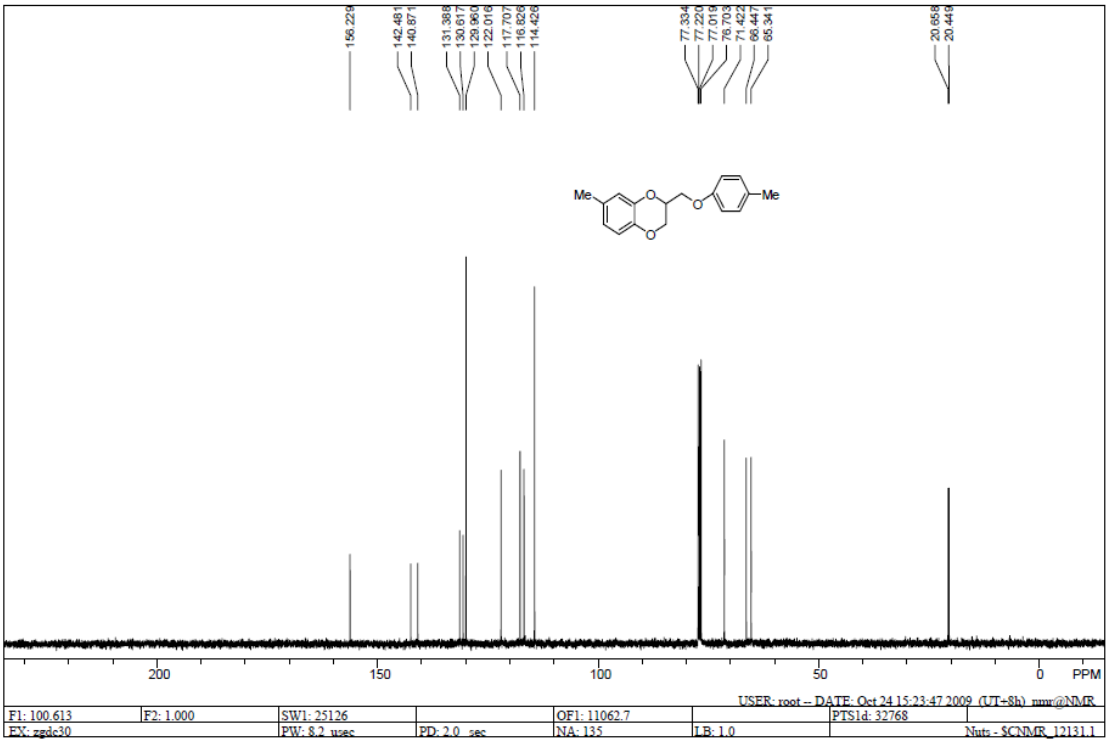

7.814
7.794
7.772
7.760
7.740
7.498
7.480
7.461
7.404
7.386
7.367
7.260
7.258
7.221
7.218
7.187
7.003
6.991
6.980
6.963
6.952
6.934
6.921
6.910
6.902
4.674
4.658
4.645
4.633
4.476
4.447
4.406
4.394
4.381
4.369
4.314
4.299
4.285
4.268
4.263
3.09
1.10
1.07
2.02
2.00
1.99
1.00
1.03
1.08
2.07
8
7
6
5
4
3
2
1
PPM
USER: root -- DATE: Oct 24 14:45:41 2009 (UT+8h) nmr@NMR
F1: 400.130
F2: 1.000
SW1: 11990
OF1: 3591.1
PTS1d: 32768
EX: zg30
PW: 12.5 usec
PD: 1.5 sec
NA: 7
LB: 0.3
Nuts - $HNMR_17823.1

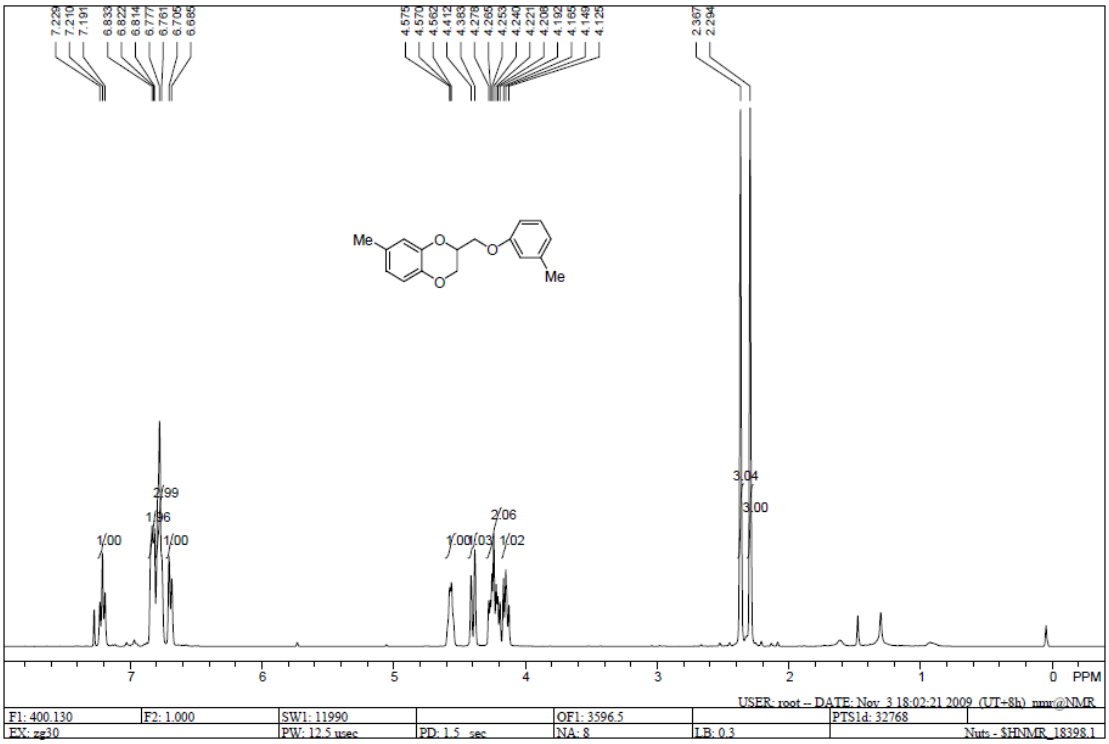

156.229
143.197
142.919
134.390
129.594
129.233
127.670
126.824
126.533
123.961
121.805
121.587
118.629
117.455
117.300
106.912
77.353
77.037
76.718
71.361
66.328
65.335
160
140
120
100
80
60
40
20
PPM
USER: root -- DATE: Oct 26 22:16:59 2009 (UT+8h) nmr@NMR
F1: 100.613
F2: 1.000
SW1: 25126
OF1: 11062.7
PTS1d: 32768
EX: zgdc30
PW: 8.2 usec
PD: 2.0 sec
NA: 70
LB: 1.0
Nuts - $CNMR_12145.1

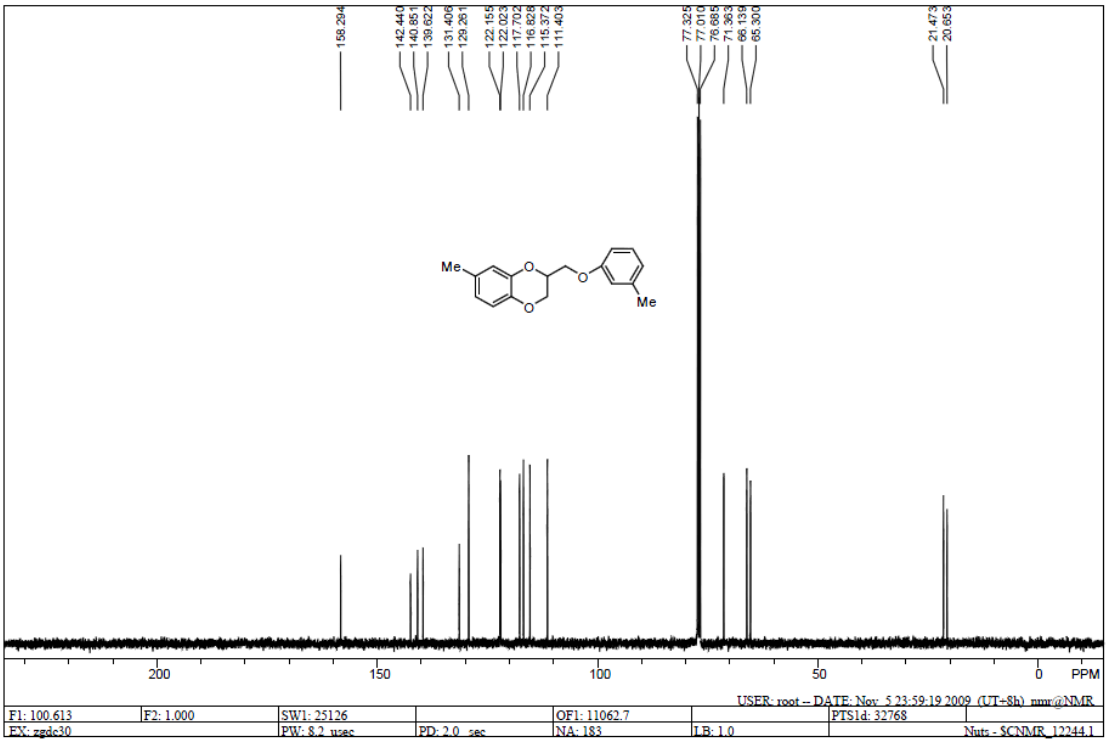

7.348
7.329
7.308
7.270
7.033
7.015
6.997
6.973
6.953
6.835
6.814
6.777
6.704
6.684
4.591
4.581
4.565
4.553
4.416
4.411
4.388
4.383
4.284
4.277
4.265
4.252
4.241
4.225
4.213
4.186
4.182
4.165
4.157
4.141
2.291
Me
O
O
O
2.07
2.00
1.91
0.98
0.98
0.97
1.00
3.09
1.01
3.01
8
7
6
5
4
3
2
1
PPM
USER: root -- DATE: Oct 16 15:51:27 2009 (UT+8h) nmr@NMR
F1: 400.130
F2: 1.000
SW1: 11990
OF1: 3595.1
PTS1d: 32768
EX: zg30
PW: 12.5 usec
PD: 1.5 sec
NA: 16
LB: 0.3
Nuts - $HNMR_17277.1

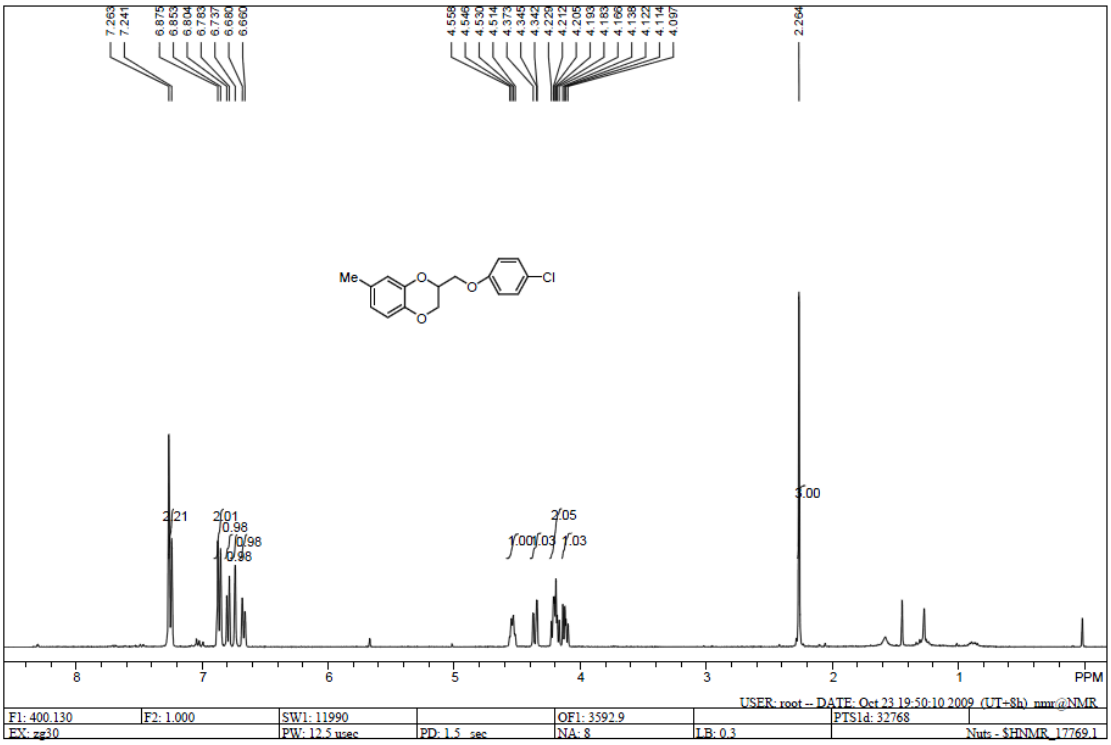

158.290
142.442
140.860
131.420
129.539
122.057
121.353
117.704
116.947
114.552
77.336
77.014
76.698
71.374
66.222
65.292
20.660
Me
O
O
O
200
150
100
50
0
PPM
USER: root -- DATE: Oct 17 13:39:39 2009 (UT+8h) nmr@NMR
F1: 100.613
F2: 1.000
SW1: 25126
OF1: 11062.7
PTS1d: 32768
EX: zgdc30
PW: 8.2 usec
PD: 2.0 sec
NA: 454
LB: 1.0
Nuts - $CNMR_12040.1

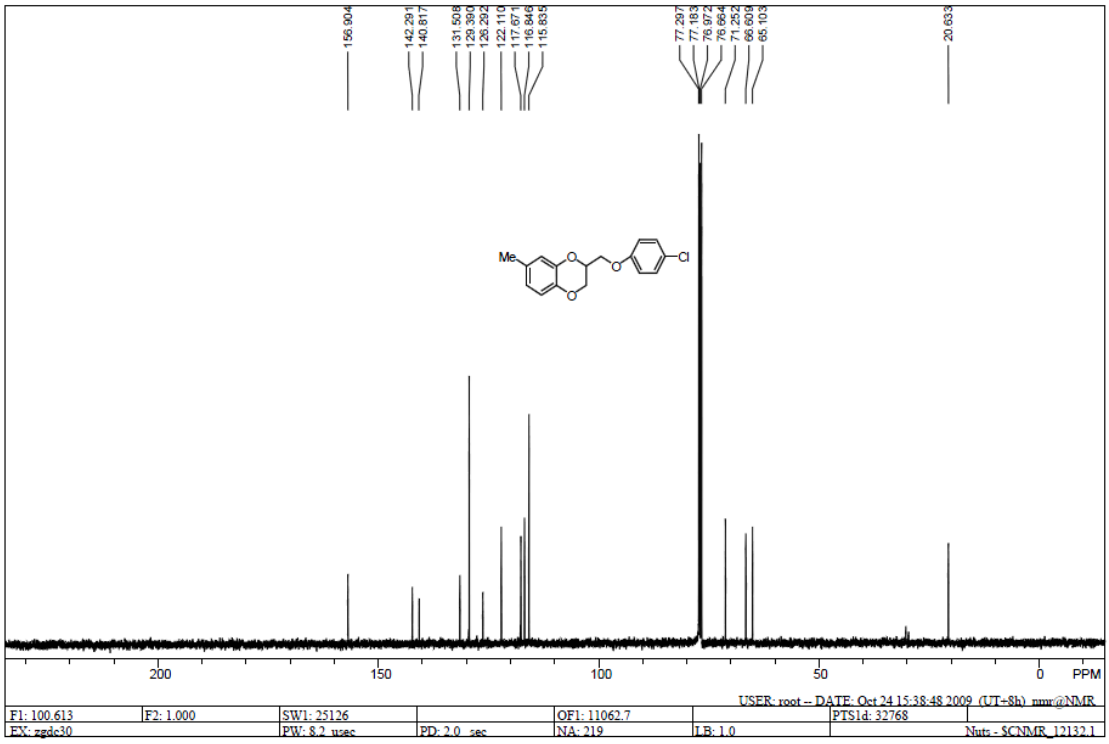

7.263
7.127
7.106
6.866
6.845
6.825
6.803
6.768
6.694
6.674
4.572
4.556
4.539
4.528
4.403
4.375
4.257
4.244
4.232
4.220
4.203
4.192
4.175
4.145
4.128
4.120
4.103
2.321
2.285
Me
Me
2.04
1.94
0.98
1.01
0.99
1.00
1.03
2.03
1.07
3.04
3.03
8
7
6
5
4
3
2
1
0
PPM
USER: root -- DATE: Oct 23 19:46:33 2009 (UT+8h) nmr@NMR
F1: 400.130
F2: 1.000
SW1: 11990
OF1: 3592.9
PTS1d: 32768
EX: ze30
PW: 12.5 usec
PD: 1.5 sec
NA: 8
LB: 0.3
Nuts - $HNMR_17768.1

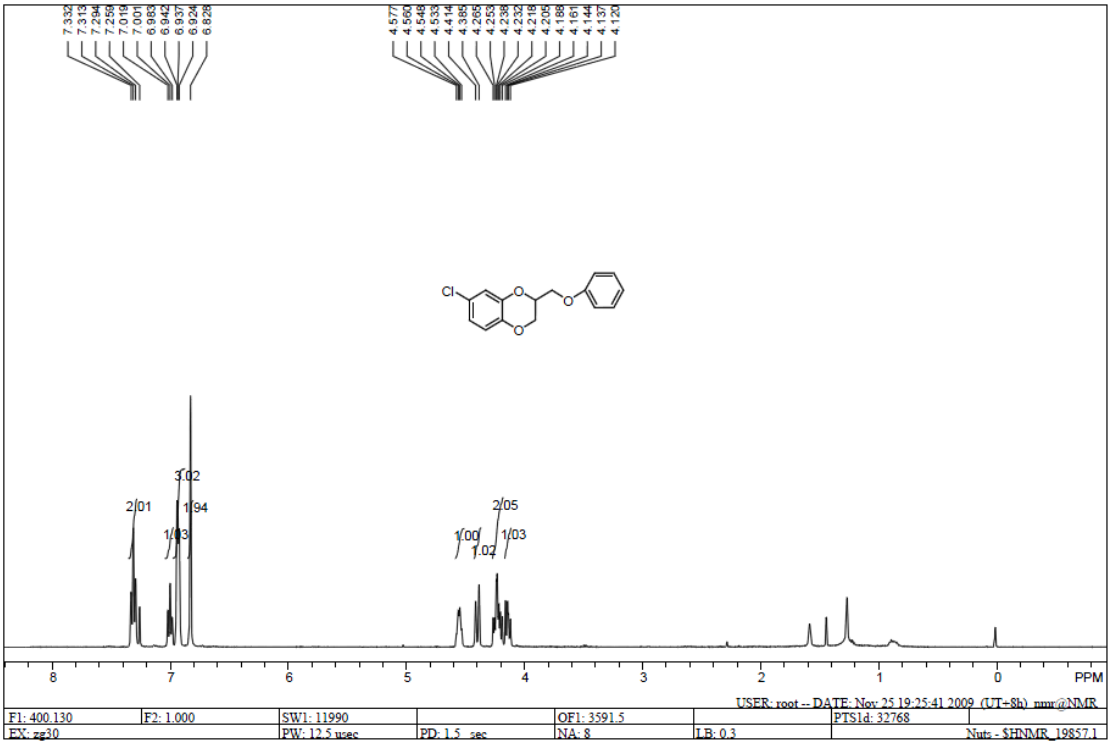

156.229
142.481
140.871
131.388
130.617
129.960
122.016
117.707
116.826
114.426
77.334
77.220
77.019
76.703
71.422
68.447
65.341
20.658
20.449
Me
Me
200
150
100
50
0
PPM
USER: root -- DATE: Oct 24 15:23:47 2009 (UT+8h) nmr@NMR
F1: 100.613
F2: 1.000
SW1: 25126
OF1: 11062.7
PTS1d: 32768
EX: zgdc30
PW: 8.2 usec
PD: 2.0 sec
NA: 135
LB: 1.0
Nuts - $CNMR_12131.1

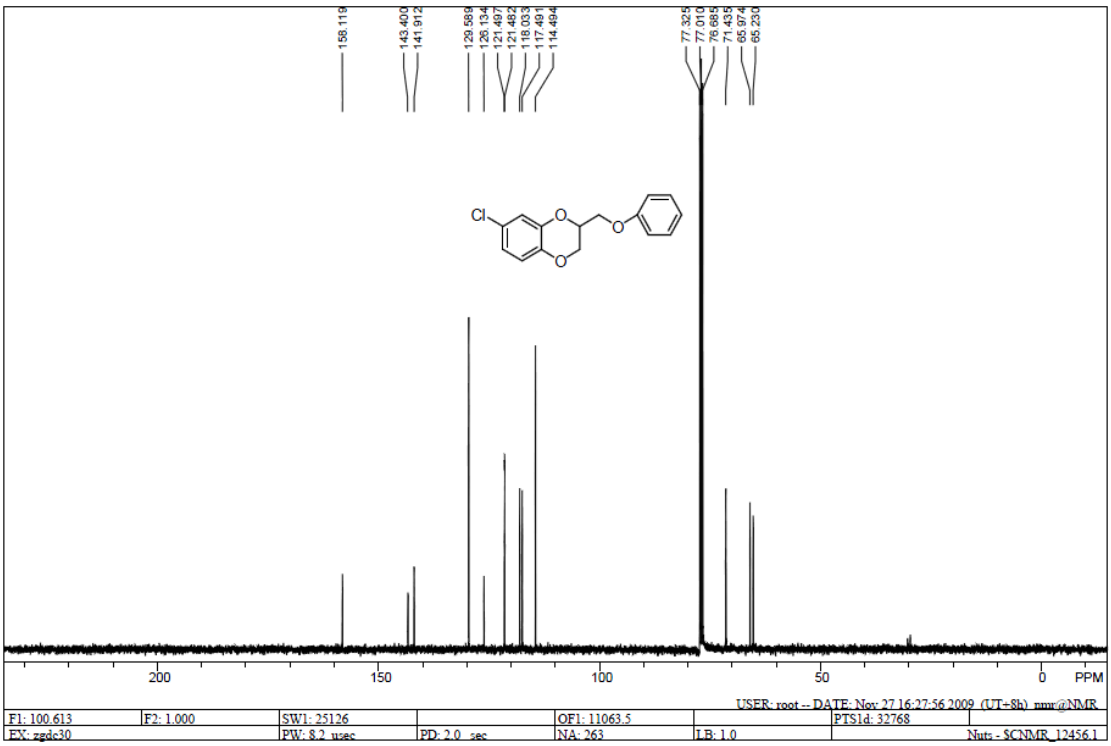

7.229
7.210
7.191
6.833
6.822
6.814
6.777
6.761
6.705
6.685
4.575
4.570
4.562
4.412
4.353
4.278
4.255
4.244
4.221
4.208
4.193
4.186
4.149
4.125
2.367
2.294
Me
O
O
O
Me
1.00
2.99
1.96
1.00
1.00
1.03
2.06
1.02
3.04
3.00
7
6
5
4
3
2
1
0
PPM
USER: root -- DATE: Nov 3 18:02:21 2009 (UT+8h) nmr@NMR
F1: 400.130
F2: 1.000
SW1: 11990
OF1: 3596.5
PTS1d: 32768
EX: zg30
PW: 12.5 usec
PD: 1.5 sec
NA: 8
LB: 0.3
Nuts - $HNMR_18398.1


158.294
142.440
140.851
139.622
131.406
129.261
122.155
122.023
117.702
116.829
115.372
111.403
77.325
77.010
76.685
71.383
66.139
65.300
21.473
20.653
Me
O
O
O
Me
200
150
100
50
0
PPM
USER: root -- DATE: Nov 5 23:59:19 2009 (UT+8h) nmr@NMR
F1: 100.613
F2: 1.000
SW1: 25126
OF1: 11062.7
PTS1d: 32768
EX: zgdc30
PW: 8.2 usec
PD: 2.0 sec
NA: 183
LB: 1.0
Nuts - $CNMR_12244.1


7.263
7.241
6.875
6.853
6.804
6.783
6.737
6.680
6.660
4.558
4.546
4.530
4.514
4.373
4.345
4.342
4.229
4.212
4.205
4.193
4.183
4.166
4.138
4.122
4.114
4.097
2.264
Me
O
O
O
Cl
2.21
2.01
0.98
0.98
0.98
2.05
1.00
1.03
1.03
3.00
8
7
6
5
4
3
2
1
PPM
USER: root -- DATE: Oct 23 19:50:10 2009 (UT+8h) nmr@NMR
F1: 400.130
F2: 1.000
SW1: 11990
OF1: 3592.9
PTS1d: 32768
EX: zg30
PW: 12.5 usec
PD: 1.5 sec
NA: 8
LB: 0.3
Nuts - $HNMR_17769.1


156.904
142.291
140.817
131.508
129.390
126.292
122.110
117.671
116.846
115.835
77.297
77.183
76.972
76.664
71.252
66.609
65.103
20.633
Me
O
O
O
Cl
200
150
100
50
0
PPM
USER: root -- DATE: Oct 24 15:38:48 2009 (UT+8h) nmr@NMR
F1: 100.613
F2: 1.000
SW1: 25126
OF1: 11062.7
PTS1d: 32768
EX: zgdc30
PW: 8.2 usec
PD: 2.0 sec
NA: 219
LB: 1.0
Nuts - $CNMR_12132.1


7.332
7.313
7.294
7.259
7.019
7.001
6.983
6.942
6.937
6.924
6.828
4.577
4.560
4.548
4.533
4.414
4.385
4.265
4.253
4.238
4.232
4.218
4.205
4.188
4.161
4.144
4.137
4.120
Cl
O
O
O
3.02
2.01
1.94
1.03
2.05
1.00
1.02
1.03
8
7
6
5
4
3
2
1
0
PPM
USER: root -- DATE: Nov 25 19:25:41 2009 (UT+8h) nmr@NMR
F1: 400.130
F2: 1.000
SW1: 11990
OF1: 3591.5
PTS1d: 32768
EX: zg30
PW: 12.5 usec
PD: 1.5 sec
NA: 8
LB: 0.3
Nuts - $HNMR_19857.1


158.119
143.400
141.912
129.589
126.134
121.497
121.482
118.033
117.491
114.494
77.325
77.010
76.695
71.435
65.974
65.230
Cl
O
O
O
200
150
100
50
0
PPM
USER: root -- DATE: Nov 27 16:27:56 2009 (UT+8h) nmr@NMR
F1: 100.613
F2: 1.000
SW1: 25126
OF1: 11063.5
PTS1d: 32768
EX: zgdc30
PW: 8.2 usec
PD: 2.0 sec
NA: 263
LB: 1.0
Nuts - $CNMR_12456.1