Supporting Information

Lewis Acid-Catalyzed Cascade Reactions of Arylmethylenecyclopropanes with 1,1,3-Triarylprop-2-yn-1-ols or Their Methyl Ethers

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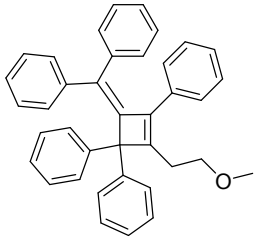
CONTENTS

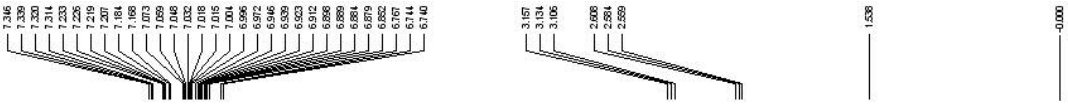
General Remarks. ^{1}H and ^{13}C NMR spectra were recorded at 300 and 75 MHz, respectively. Mass and HRMS spectra were recorded by EI method. Organic solvents used were dried by standard methods when necessary. Satisfactory CHN microanalyses for several solid compounds were obtained with an analyzer. Commercially obtained reagents were used without further purification. All these reactions were monitored by TLC with silica gel coated plates. Flash column chromatography was carried out using silica gel at increased pressure.

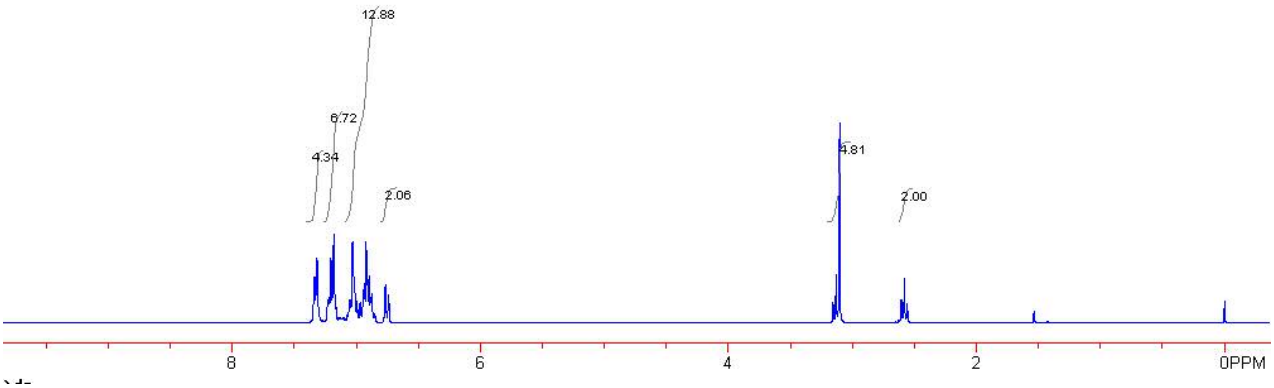
General Procedure for Lewis Acid-Catalyzed Reaction of Arylmethylenecyclopropanes and 1,1,3-Triarylprop-2-yn-1-ols or Their Methyl Ethers. Under an argon atmosphere, arylmethylenecyclopropanes **1** (0.2 mmol), 1,1,3-triarylprop-2-yn-1-ols or their methyl ethers **2** (0.4 mmol), $BF_3{\cdot}OEt_2$ (10 mol %) were added into a Schlenk tube. The reaction mixture was stirred at room temperature for 6 h, then the solvent was removed under reduced pressure and the residue was purified by a flash column chromatography.

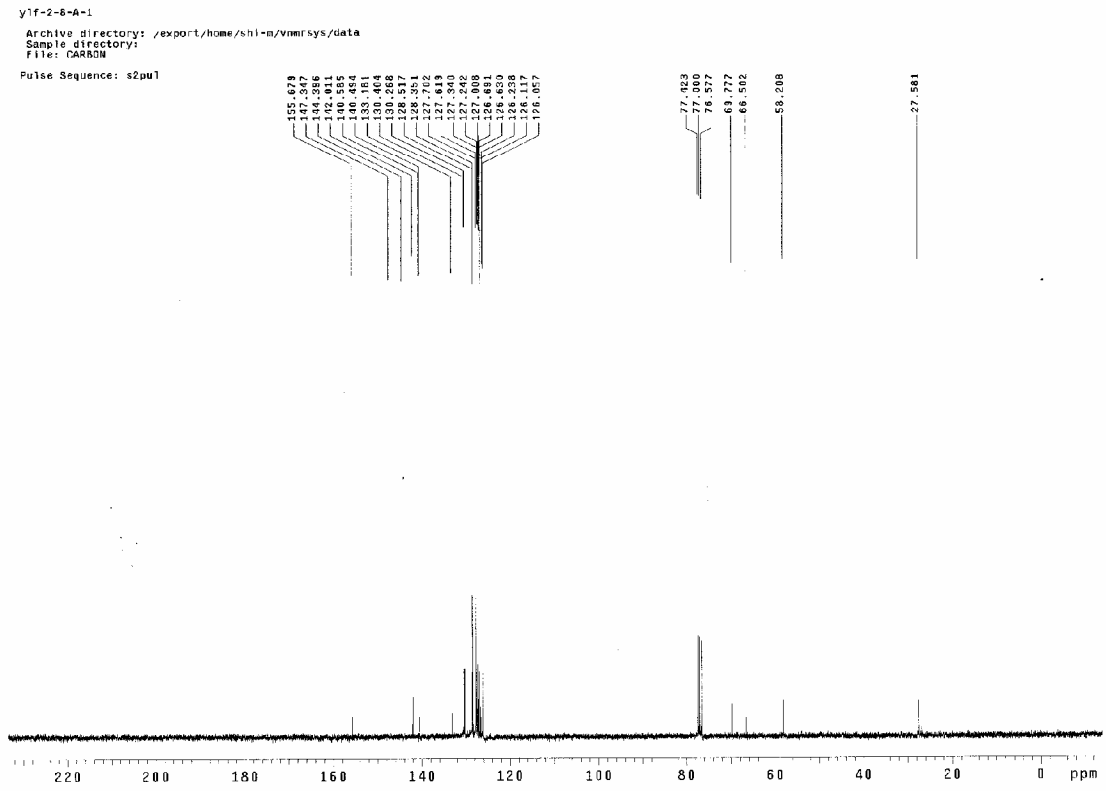


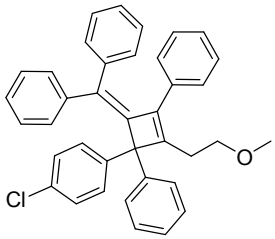
3a

A yellow solid, mp: 121-123 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.58 (t, J = 7.2 Hz, 2H, CH_2), 3.11 (s, 3H, OCH_3), 3.14 (t, J = 7.2 Hz, 2H, CH_2), 6.74-6.77 (m, 2H, Ar), 6.89-7.08 (m, 13H, Ar), 7.17-7.31 (m, 6H, Ar), 7.32-7.35 (m, 4H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 27.6, 58.2, 66.5, 69.8, 126.06, 126.12, 126.2, 126.6, 126.7, 127.0, 127.2, 127.3, 127.6, 127.7, 128.4, 128.5, 130.3, 130.4, 133.2, 140.5, 140.6, 142.0, 144.4, 147.3, 155.7. IR (CH_2Cl_2) ν 3079, 3055, 3026, 2980, 2926, 2891, 2826, 2807, 1598, 1492, 1442, 1184, 1113, 1076, 1028, 1000, 917, 762, 744, 731, 698, 669, 638 cm^{-1}. MS (MALDI) m/z 527 (M+Na^{+}). Anal. calcd. for $C_{38}H_{32}O$: C, 90.44%; H, 6.39%; Found: C, 90.46%; H, 6.63%.



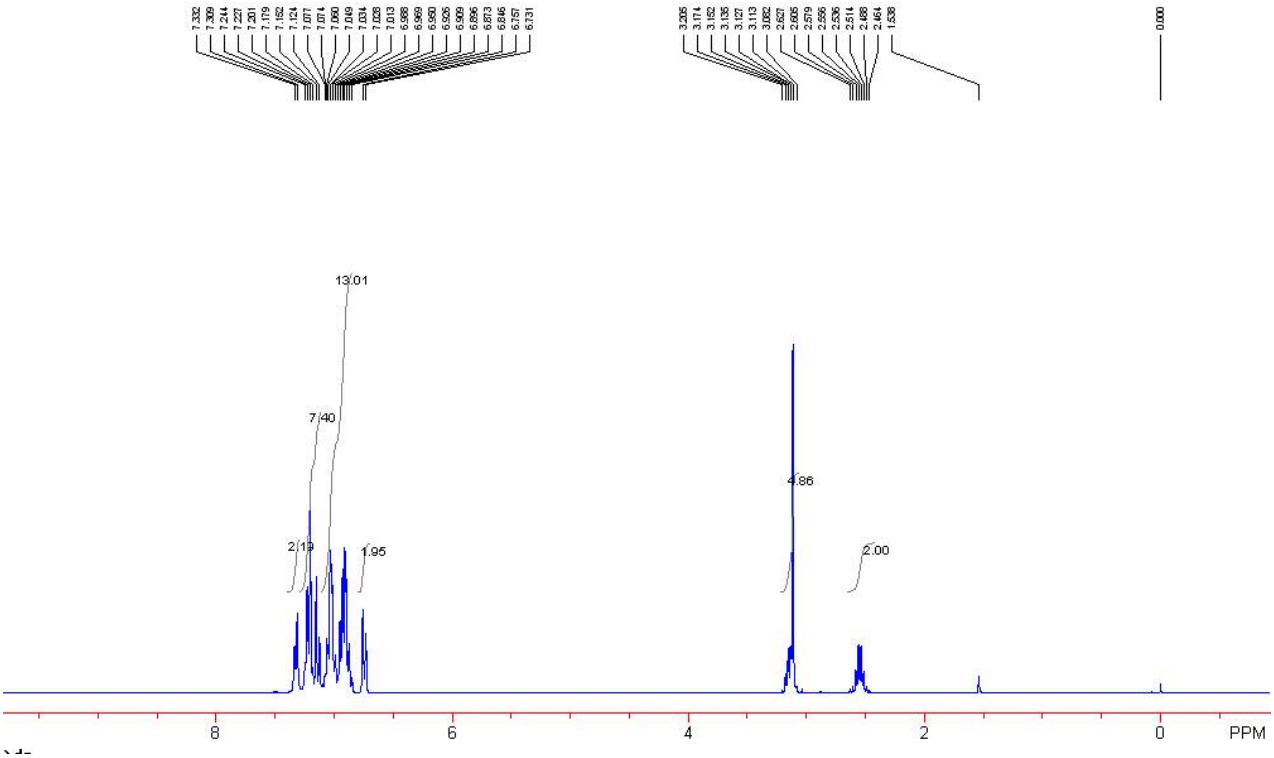






3b

A yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.47-2.63 (m, 2H, CH_2), 3.12 (s, 3H, OCH_3), 3.13-3.18 (m, 2H, CH_2), 6.75 (d, J = 7.8 Hz, 2H, Ar), 6.85-6.99 (m, 7H, Ar), 7.02-7.08 (m, 6H, Ar), 7.13-7.24 (m, 7H, Ar), 7.33 (d, J = 7.8 Hz, 2H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 27.5, 58.3, 66.0, 69.7, 126.25, 126.31, 126.4, 126.8, 127.0, 127.1, 127.3, 127.4, 127.6, 127.7, 127.9, 128.4, 129.9, 130.2, 130.3, 131.8, 132.9, 140.3, 140.6, 141.7, 144.0, 147.6, 155.3. IR (CH_2Cl_2) ν 3053, 3027, 2926, 1598, 1489, 1443, 1113, 1092, 1013, 920, 831, 770, 750, 698, 668 cm^{-1}. MS (MALDI) m/z 561 ($M+Na^+$). HRMS (MALDI) calcd. for $C_{38}H_{31}OClNa^+$: 561.1975, Found: 561.1956.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0
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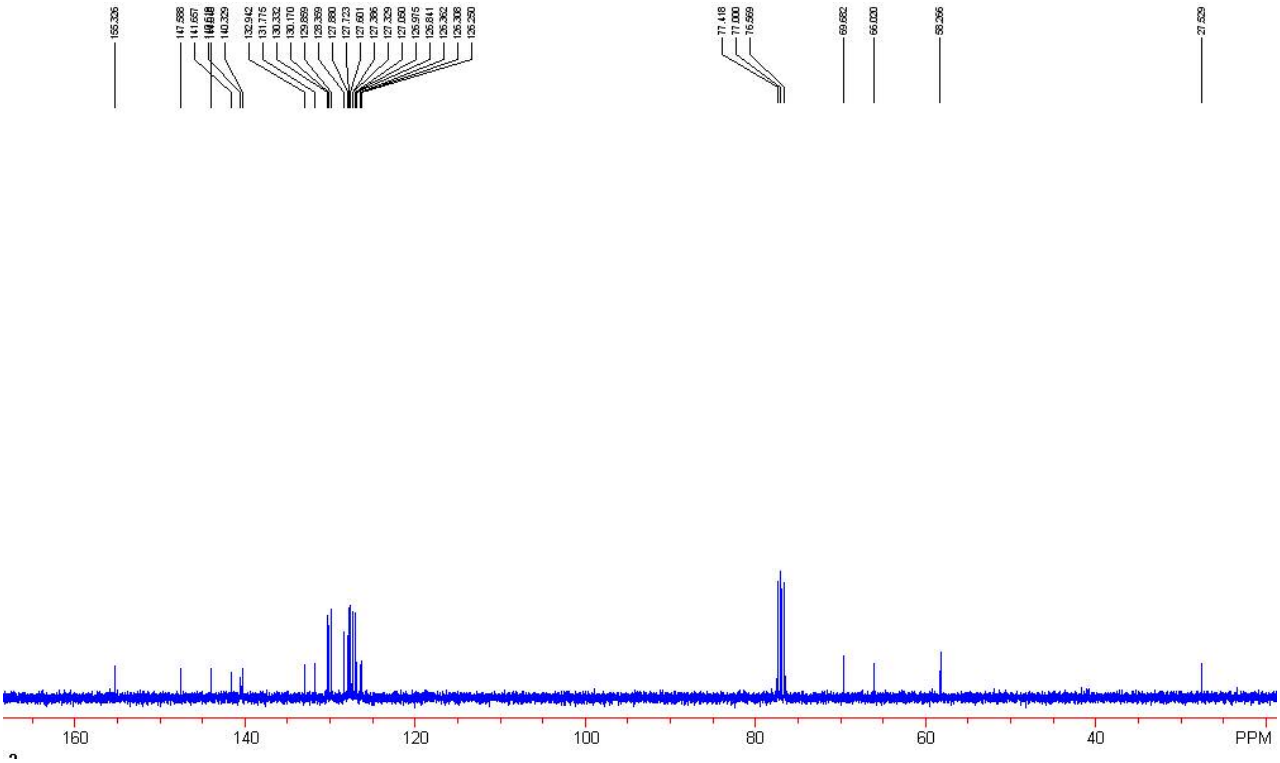
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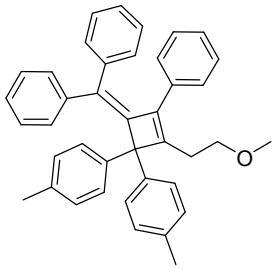
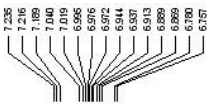
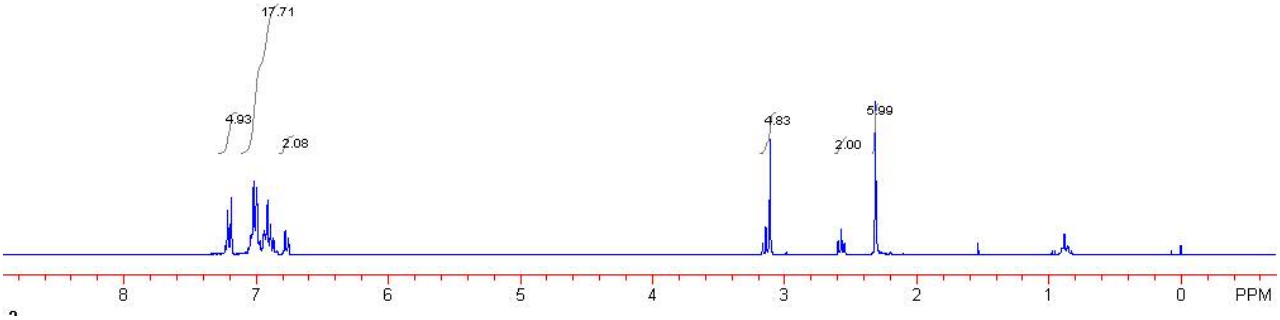
40
PPM

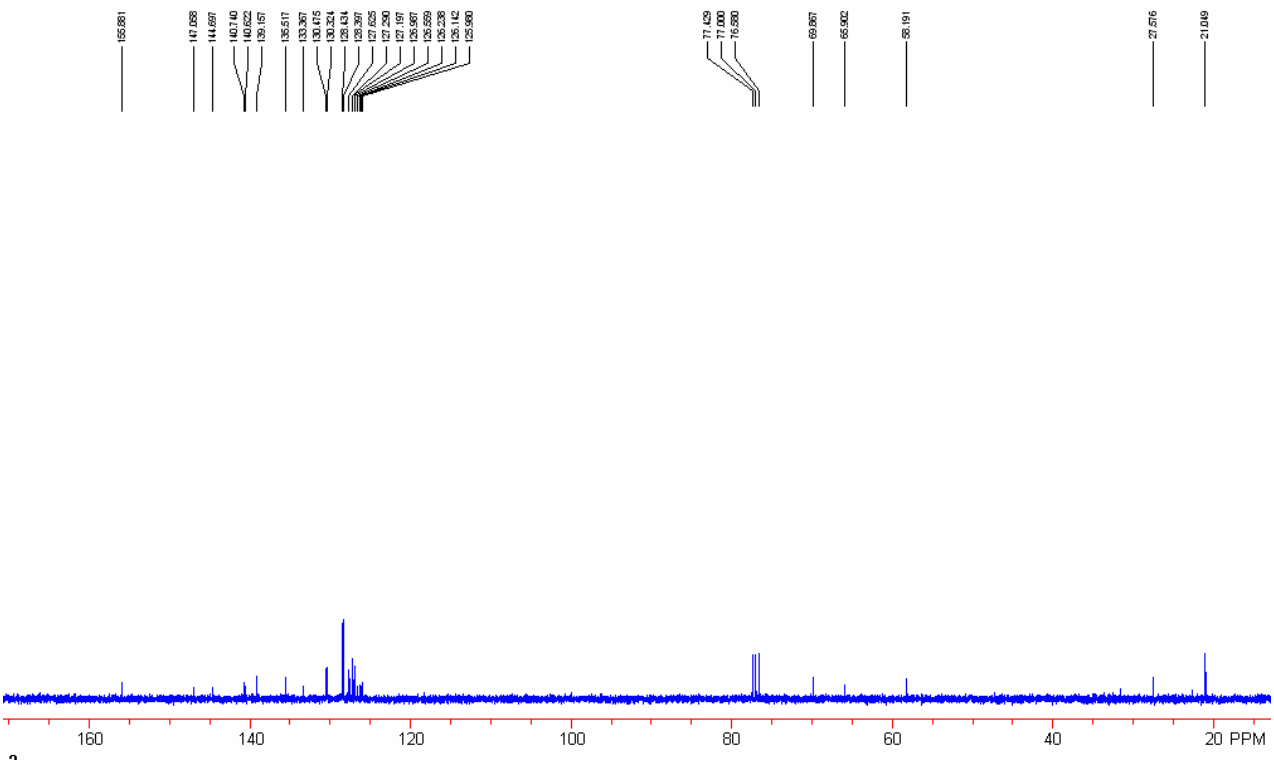


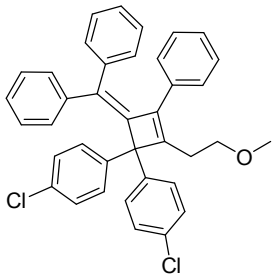
3c

A yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.31 (s, 6H, CH_3), 2.57 (t, J = 7.5 Hz, 2H, CH_2), 3.11 (s, 3H, OCH_3), 3.14 (t, J = 7.5 Hz, 2H, CH_2), 6.77 (d, J = 7.8 Hz, 2H, Ar), 6.87-7.02 (m, 17H, Ar), 7.20 (d, J = 7.8 Hz, 4H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 21.0, 27.5, 58.2, 65.9, 69.8, 126.0, 126.1, 126.2, 126.6, 127.0, 127.2, 127.3, 127.6, 128.38, 128.41, 130.3, 130.5, 133.3, 135.5, 139.1, 140.6, 140.7, 144.7, 147.0, 155.9. IR (CH_2Cl_2) ν 3078, 3053, 3021, 2922, 2874, 2826, 2807, 1731, 1599, 1575, 1510, 1492, 1442, 1382, 1266, 1186, 1114, 1071, 1022, 1000, 922, 821, 765, 744, 699, 635, 560, 542 cm^{-1}. MS (MALDI) m/z 555 ($M+Na^+$). HRMS (MALDI) calcd. for $C_{40}H_{36}ONa^+$: 555.2675, Found: 555.2658.









3d

A yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.52 (t, *J* = 7.2 Hz, 2H, CH_2), 3.13 (s, 3H, OCH_3), 3.16 (t, *J* = 7.2 Hz, 2H, CH_2), 6.71 (d, *J* = 8.1 Hz, 2H, Ar), 6.74-7.26 (m, 21H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 27.5, 58.3, 65.6, 69.6, 126.4, 126.5, 127.0, 127.1, 127.36, 127.44, 127.6, 127.9, 128.3, 129.7, 130.1, 130.3, 132.0, 132.7, 140.1, 140.2, 140.3, 143.7, 147.9, 155.0. IR (CH_2Cl_2) ν 3056, 3026, 2926, 1489, 1442, 1113, 1092, 1013, 920, 831, 772, 750, 731, 697, 669 cm^{-1}. MS (MALDI) m/z 595 ($M+Na^+$). HRMS (MALDI) calcd. for $C_{38}H_{30}OCl_2Na^+$: 595.1566, Found:595.1566.


7.246
7.230
7.224
7.201
7.182
7.161
7.154
7.098
7.083
7.075
7.069
7.046
7.023
6.996
6.984
6.959
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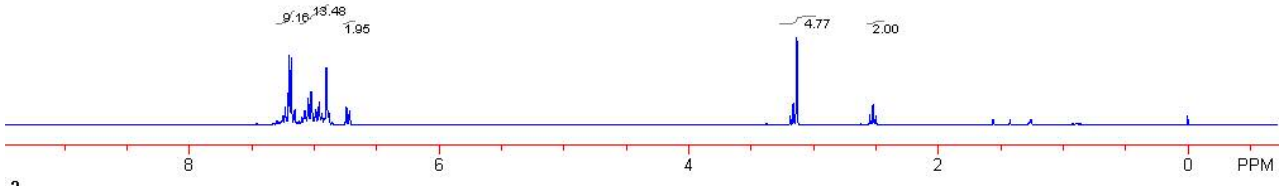
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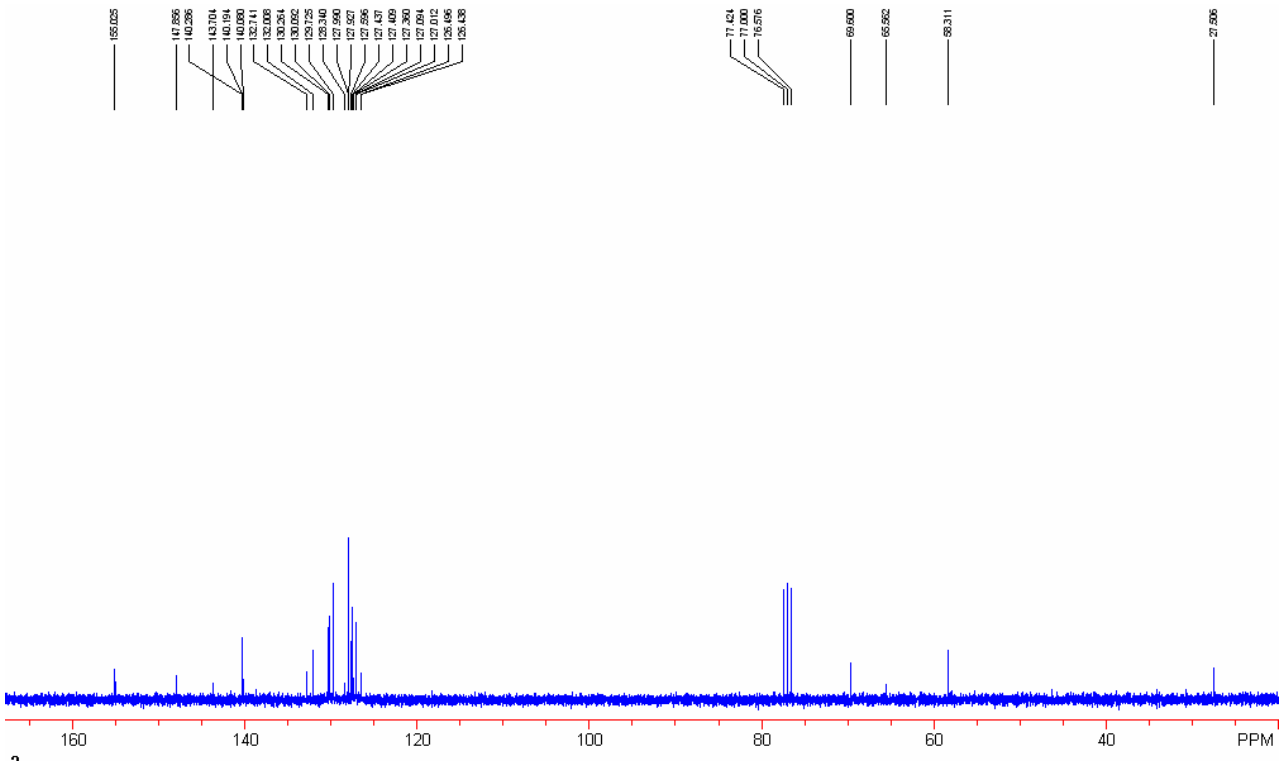
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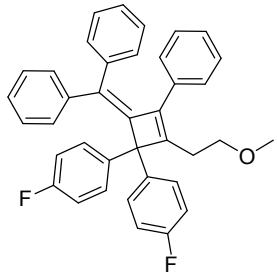
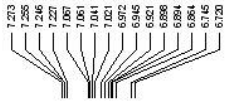
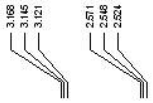
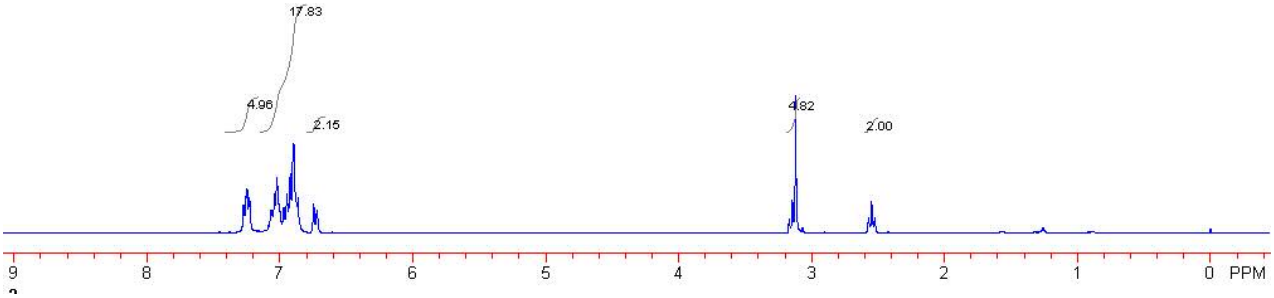


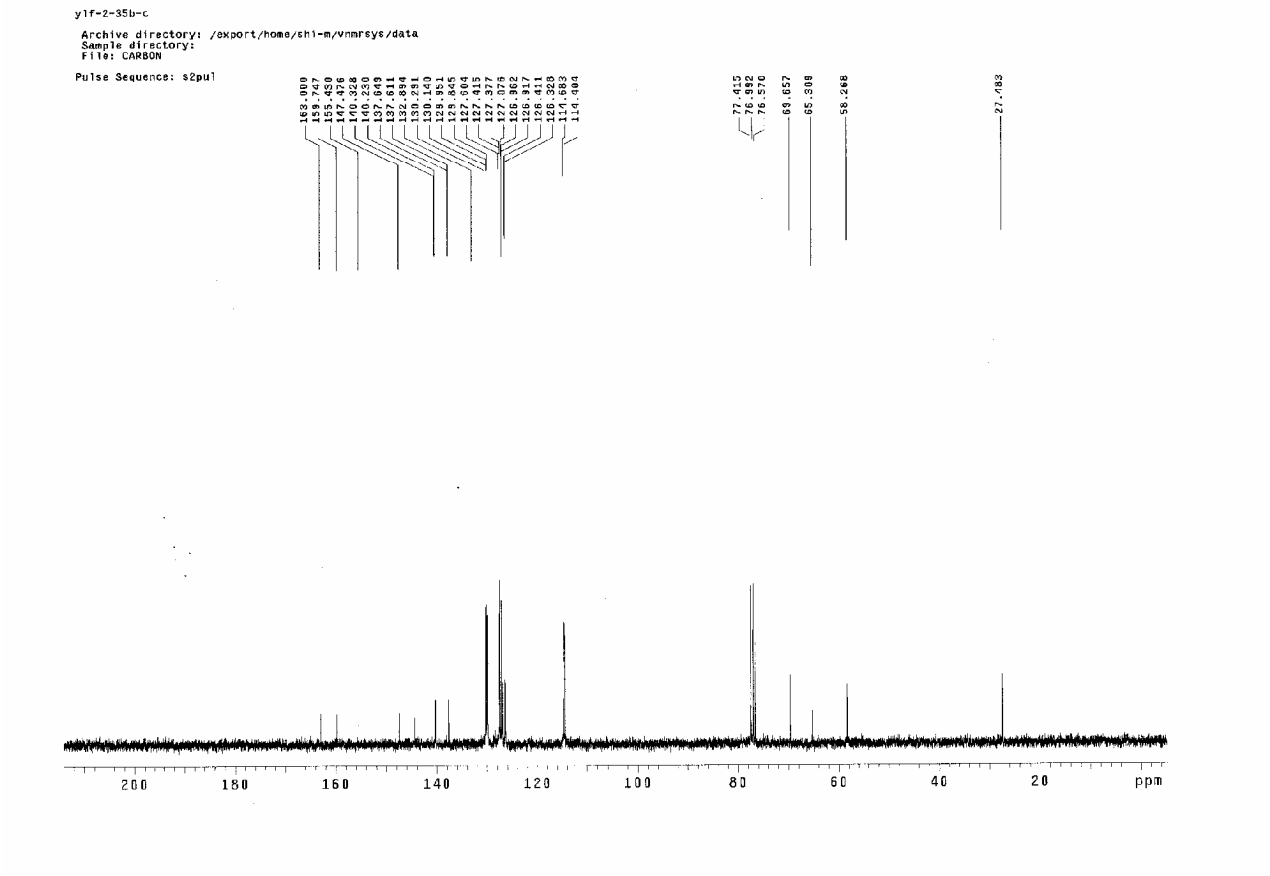
3e

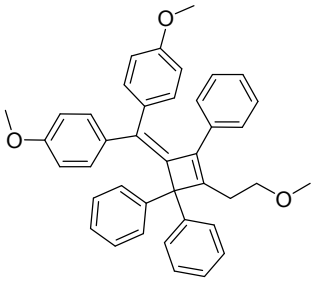
A yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.55 (t, *J* = 7.2 Hz, 2H, CH_2), 3.12 (s, 3H, OCH_3), 3.15 (t, *J* = 7.2 Hz, 2H, CH_2), 6.73 (d, *J* = 7.2 Hz, 2H, Ar), 6.86-7.09 (m, 17H, Ar), 7.23-7.27 (m, 4H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 27.5, 58.3, 65.3, 69.7, 114.6 (d, *J* = 20.9 Hz), 126.3, 126.4, 126.9, 127.0, 127.1, 127.38, 127.42, 127.6, 129.8, 130.0, 130.2 (d, *J* = 11.0 Hz), 132.9, 137.61, 137.65, 140.2, 140.3, 147.5, 155.4, 161.4 (d, *J* = 244.0 Hz). IR (CH_2Cl_2) ν 3055, 2926, 2892, 2827, 1601, 1506, 1443, 1229, 1159, 1113, 1015, 923, 837, 808, 766, 746, 700, 558, 545, 515 cm^{-1}. MS (MALDI) m/z 563 ($M+Na^+$). HRMS (MALDI) calcd. for $C_{38}H_{30}OF_2Na^+$: 563.2166, Found: 563.2157.







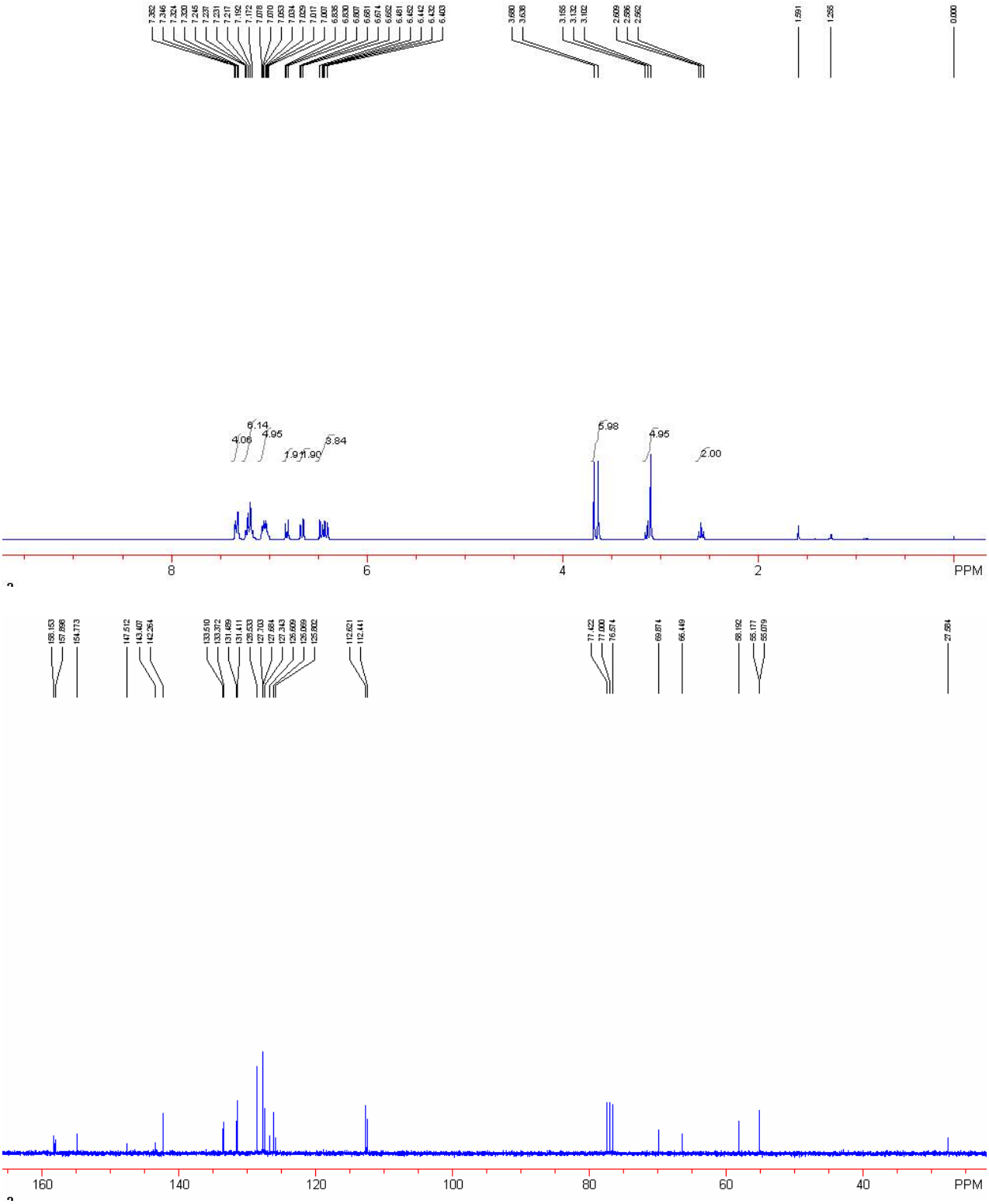


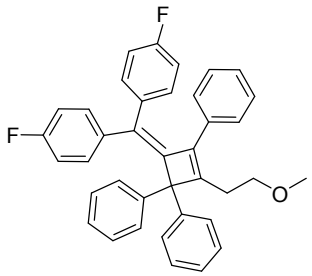


3f

A yellow solid, m.p. 126-128 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.59 (t, J = 7.2 Hz, 2H, CH_2), 3.10 (s, 3H, OCH_3), 3.13 (t, J = 7.2 Hz, 2H, CH_2), 3.64 (s, 3H, OCH_3), 3.68 (s, 3H, OCH_3), 6.42 (d, J = 8.7, Hz, 2H, Ar), 6.46 (d, J = 8.7, Hz, 2H, Ar), 6.67 (d, J = 8.7 Hz, 2H, Ar), 6.82 (d, J = 8.7 Hz, 2H, Ar), 7.01-7.08 (m, 5H, Ar), 7.17-7.24 (m, 6H, Ar), 7.32-7.35 (m, 4H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 27.6, 55.0, 55.2, 58.2, 66.4, 69.9, 112.4, 112.6, 125.8, 126.1, 126.6, 127.3, 127.7, 128.5, 131.4, 131.5, 133.4, 133.5, 142.3, 143.4, 147.5, 154.8, 157.9, 158.2. IR (CH_2Cl_2) ν 3056, 3029, 2996, 2931,2897, 2834, 1604, 1574, 1508, 1493, 1463, 1442, 1301, 1271, 1245, 1173, 1112, 1035, 832, 764, 733, 702, 598, 579 cm^{-1}. MS (MALDI) m/z 587 ($M+Na^+$).

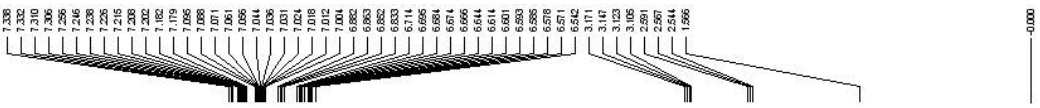
HRMS (MALDI) calcd. for $C_{40}H_{36}O_3Na^+$: 587.2576, Found:587.2557.

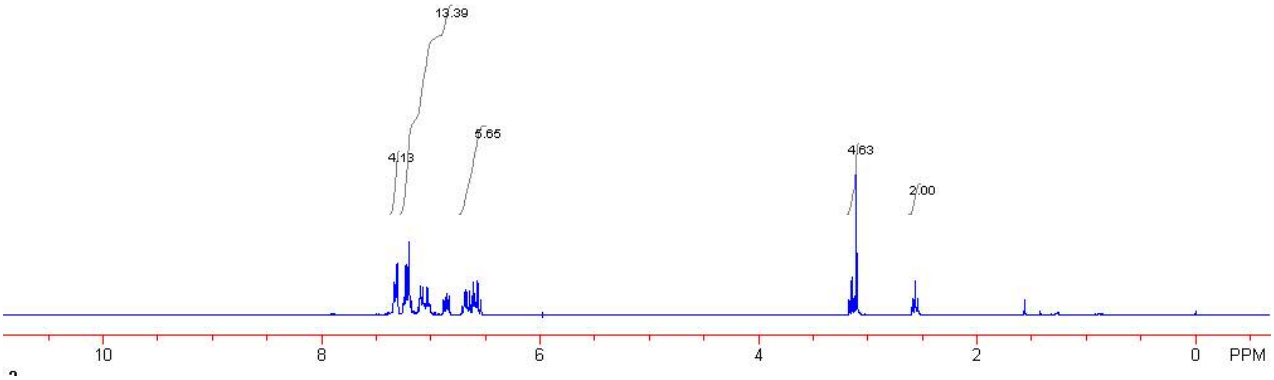


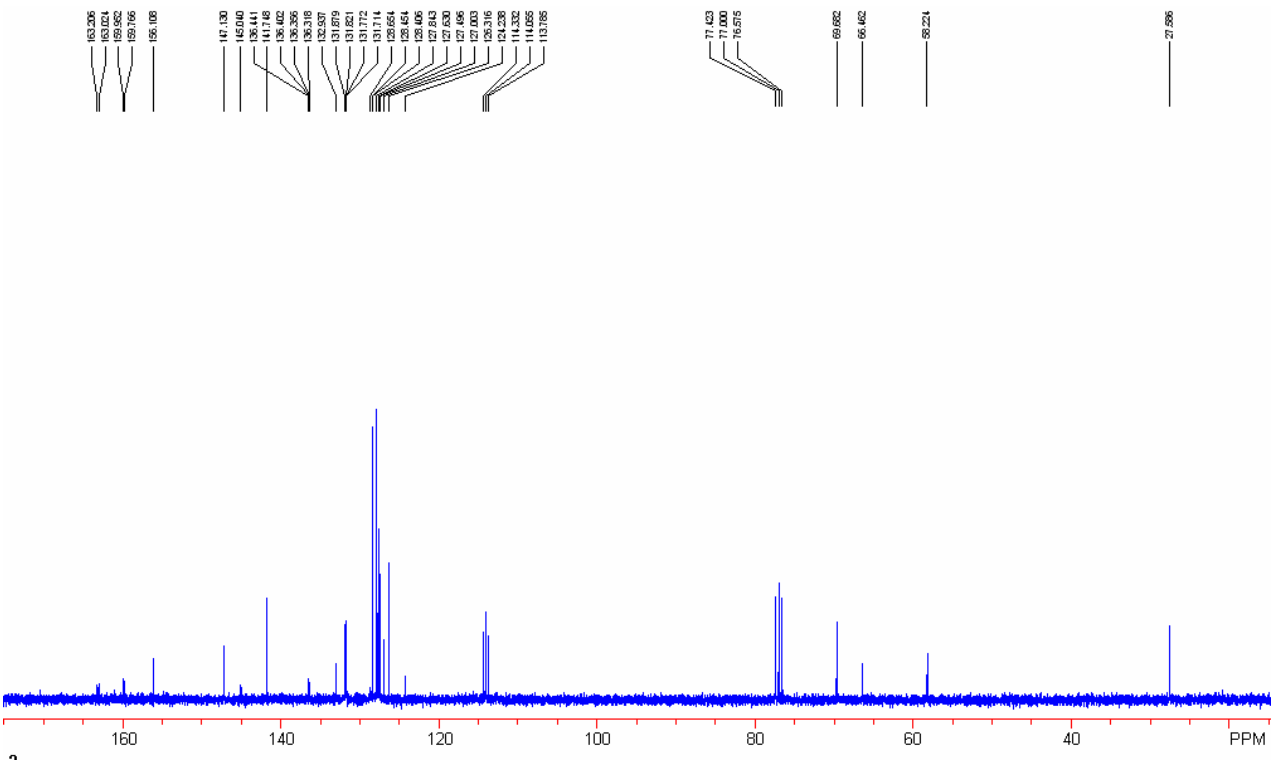


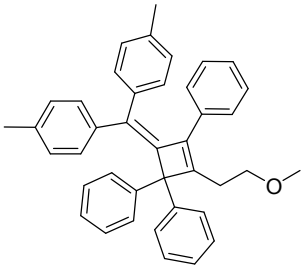
3g

A yellow solid, m.p. 138-140 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.57 (t, *J* = 7.2 Hz, 2H, CH_2), 3.11 (s, 3H, OCH_3), 3.15 (t, *J* = 7.2 Hz, 2H, CH_2), 6.54-6.71 (m, 6H, Ar), 6.83-6.88 (m, 2H, Ar), 7.00-7.11 (m, 5H, Ar), 7.18-7.34 (m, 10H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 27.6, 58.2, 66.5, 69.7, 113.8 (d, *J* = 20.8 Hz), 114.2 (d, *J* = 20.8 Hz), 124.2, 126.3, 127.0, 127.5, 127.6, 127.8, 128.4, 128.5, 131.8 (d, *J* = 8.0 Hz), 131.9 (d, *J* = 8.0 Hz), 132.9, 136.3 (d, *J* = 2.8 Hz), 136.4 (d, *J* = 2.9 Hz), 141.7, 145.0, 147.1, 156.1, 161.4 (d, *J* = 244.0 Hz), 161.7 (d, *J* = 244.0 Hz). IR (CH_2Cl_2) ν 3058, 2927, 2894, 2828, 1888, 1599, 1505, 1443, 1379, 1296, 1223, 1189, 1156, 1113, 1028, 1013, 1001, 951, 917, 836, 809, 765, 726, 704, 672, 653, 590, 573, 526, 509, 501 cm^{-1}. MS (MALDI) m/z 563 (M+Na$^+$). HRMS (MALDI) calcd. for $C_{38}H_{30}OF_2Na^+$: 563.2140, Found: 563.2157.



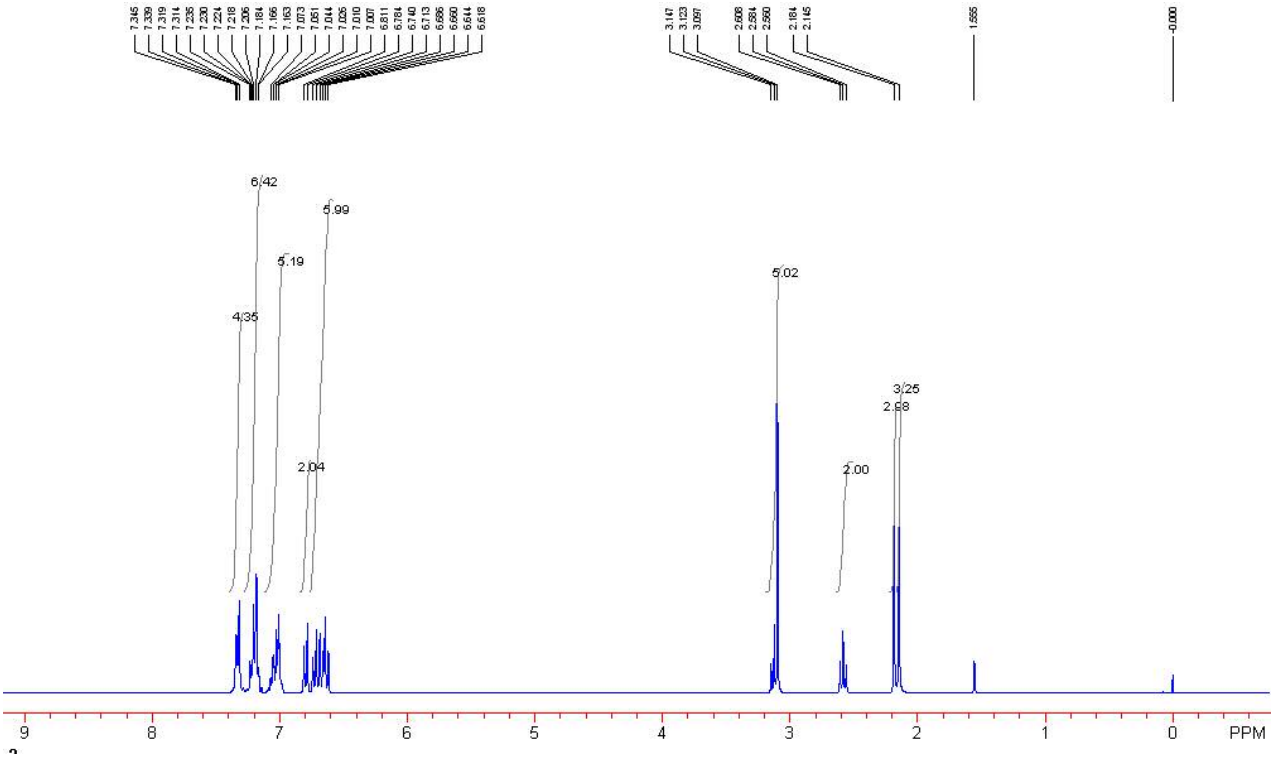






3h

A yellow solid, m.p. 144-146 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.14 (s, 3H, CH_3), 2.18 (s, 3H, CH_3), 2.58 (t, *J* = 7.2 Hz, 2H, CH_2), 3.10 (s, 3H, OCH_3), 3.12 (t, *J* = 7.2 Hz, 2H, CH_2), 6.62-6.81 (m, 8H, Ar), 7.01-7.05 (m, 5H, Ar), 7.18-7.23 (m, 6H, Ar), 7.31-7.34 (m, 4H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 21.0, 21.1, 27.6, 58.2, 66.4, 69.8, 126.0, 126.5, 126.6, 127.2, 127.6, 127.7, 127.9, 128.6, 130.1, 130.3, 133.3, 135.6, 135.8, 137.8, 137.9, 142.2, 143.7, 147.5, 155.0. IR (CH_2Cl_2) ν 3080, 3055, 3021, 2923, 1598, 1509, 1492, 1443, 1181, 1113, 1021, 820, 762, 744, 724 cm^{-1}. MS (MALDI) m/z 555 ($M+Na^+$). HRMS (MALDI) calcd. for $C_{40}H_{36}ONa^+$: 555.2662, Found: 555.2658.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4.35
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3.25
2.98
2.00
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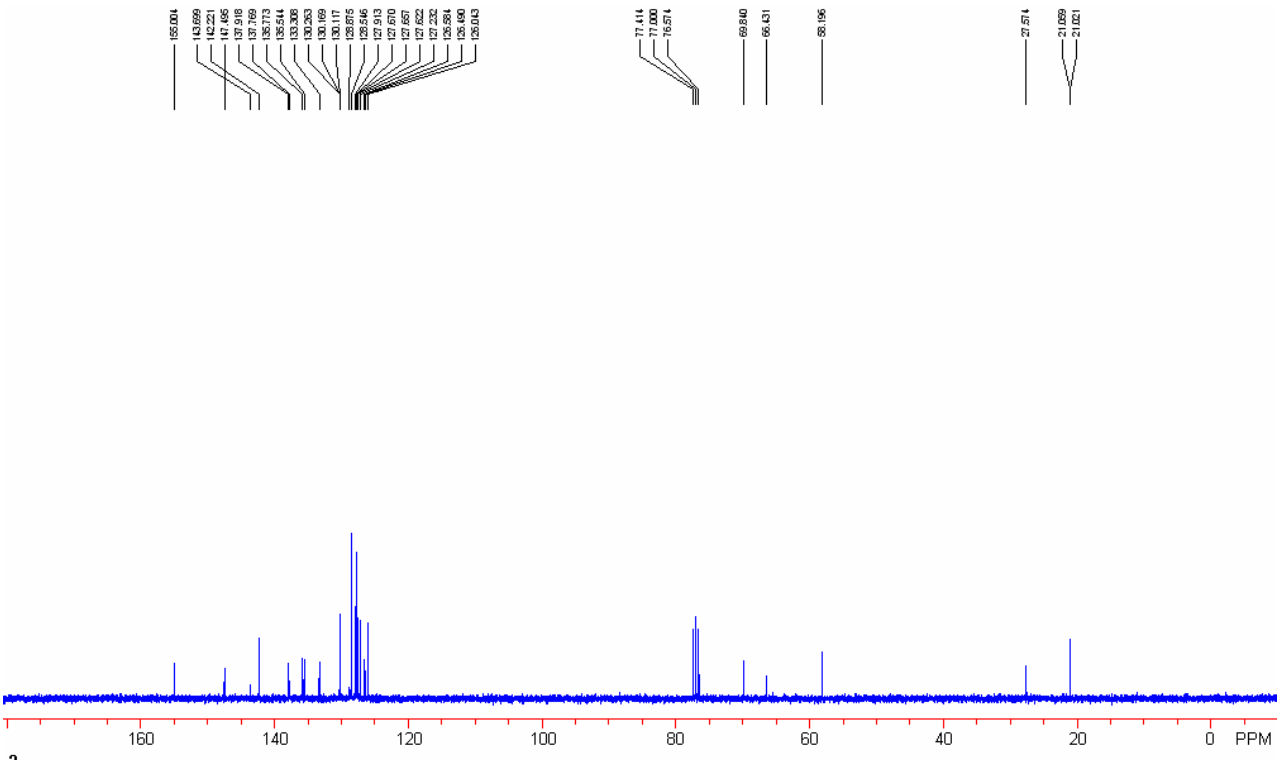
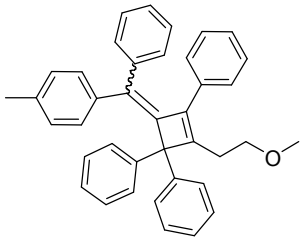
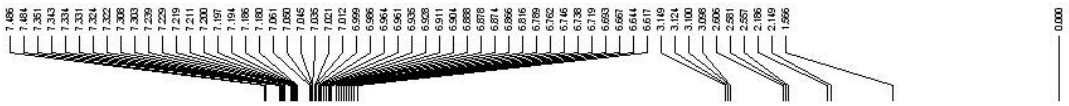
6
5
4
3
2
1
0
PPM

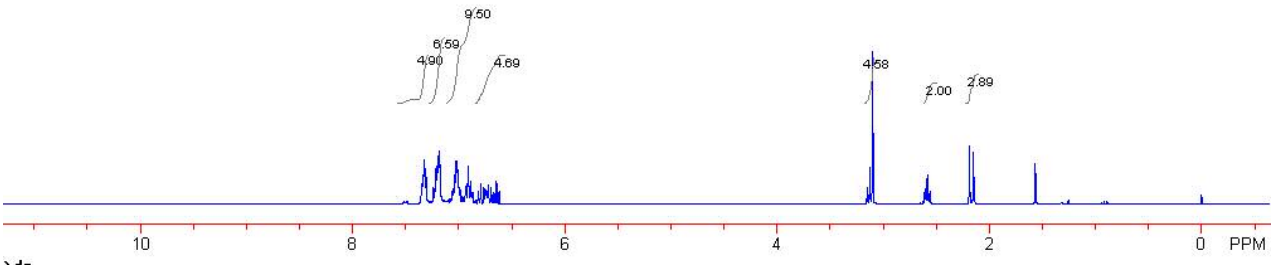

160
140
120
100
80
60
40
20
0
PPM

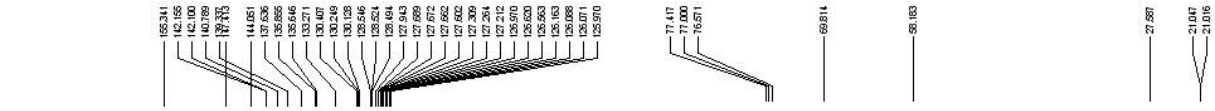


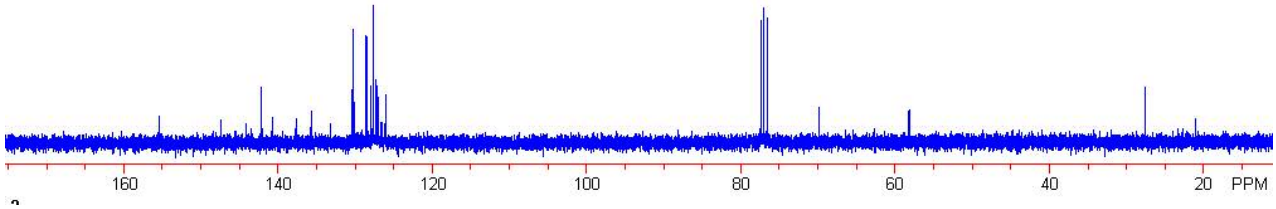
3i

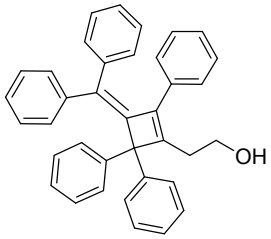
A yellow oil, (*E*-isomer and *Z*-isomer = 1:1). ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.15 (s, 3H, CH_3), 2.19 (s, 3H, CH_3), 2.58 (t, *J* = 7.2 Hz, 4H, CH_2), 3.10 (s, 6H, OCH_3), 3.13 (t, *J* = 7.2 Hz, 4H, CH_2), 6.62-6.87 (m, 10H, Ar), 6.88-6.94 (m, 8H, Ar), 6.95-7.06 (m, 10H, Ar), 7.18-7.31 (m, 12H, Ar), 7.32-7.35 (m, 8H, Ar). (*E*-isomer and *Z*-isomer = 1:1) ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 21.0, 21.1, 27.6, 58.2, 66.5, 69.8, 126.0, 126.1, 126.2, 126.6, 127.0, 127.2, 127.27, 127.31, 127.6, 127.7, 127.9, 128.5, 128.6, 130.1, 130.3, 130.4, 133.3, 135.6, 135.9, 137.7, 140.7, 140.8, 142.1, 142.2, 144.1, 147.4, 155.4. IR (CH_2Cl_2) ν 3079, 3054, 3019, 2983, 2922, 2890, 2825, 2809, 1731, 1631, 1598, 1575, 1541, 1510, 1492, 1456, 1442, 1111, 1098, 913, 825, 808, 791, 783, 775, 768, 759, 740, 733, 722, 697, 685, 672, 633, 623, 612, 603, 577, 569, 561, 549, 504 cm^{-1}. MS (MALDI) m/z 541 ($M+Na^+$). HRMS (MALDI) calcd. for $C_{39}H_{34}ONa^+$: 541.2505, Found: 541.2502.





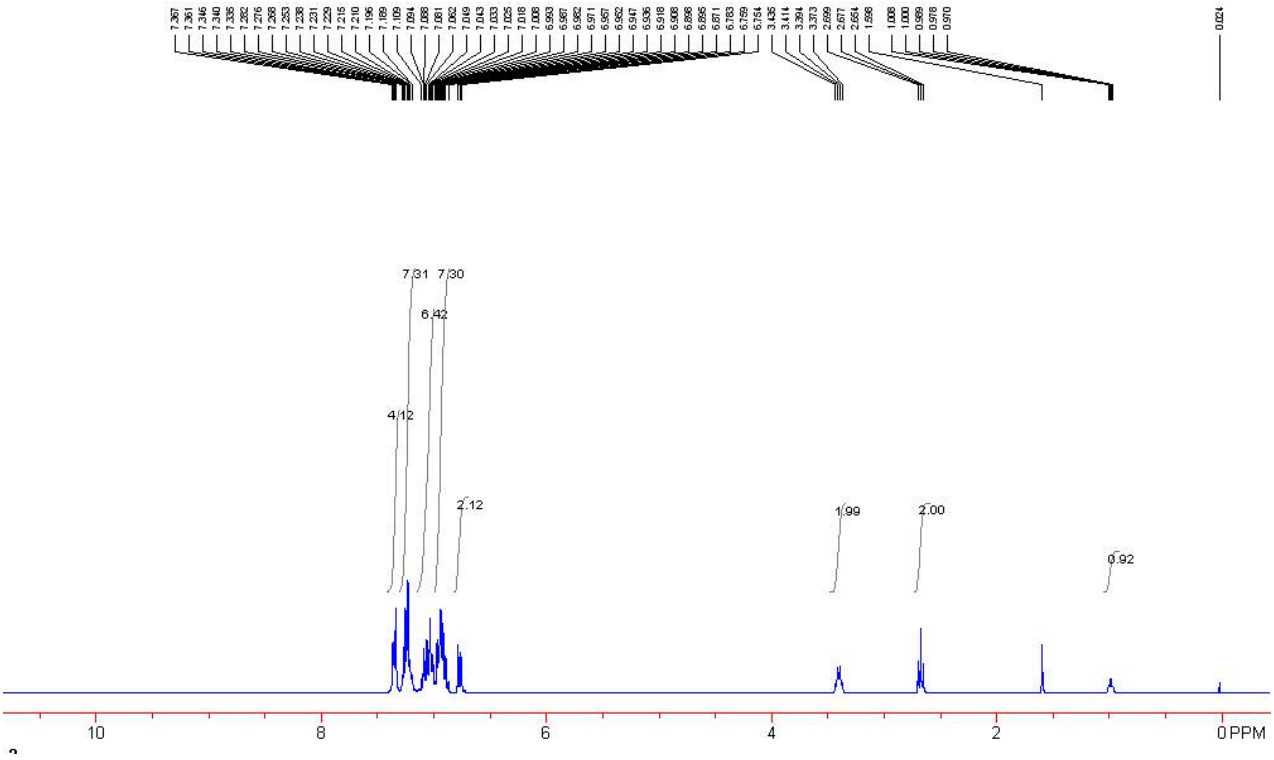


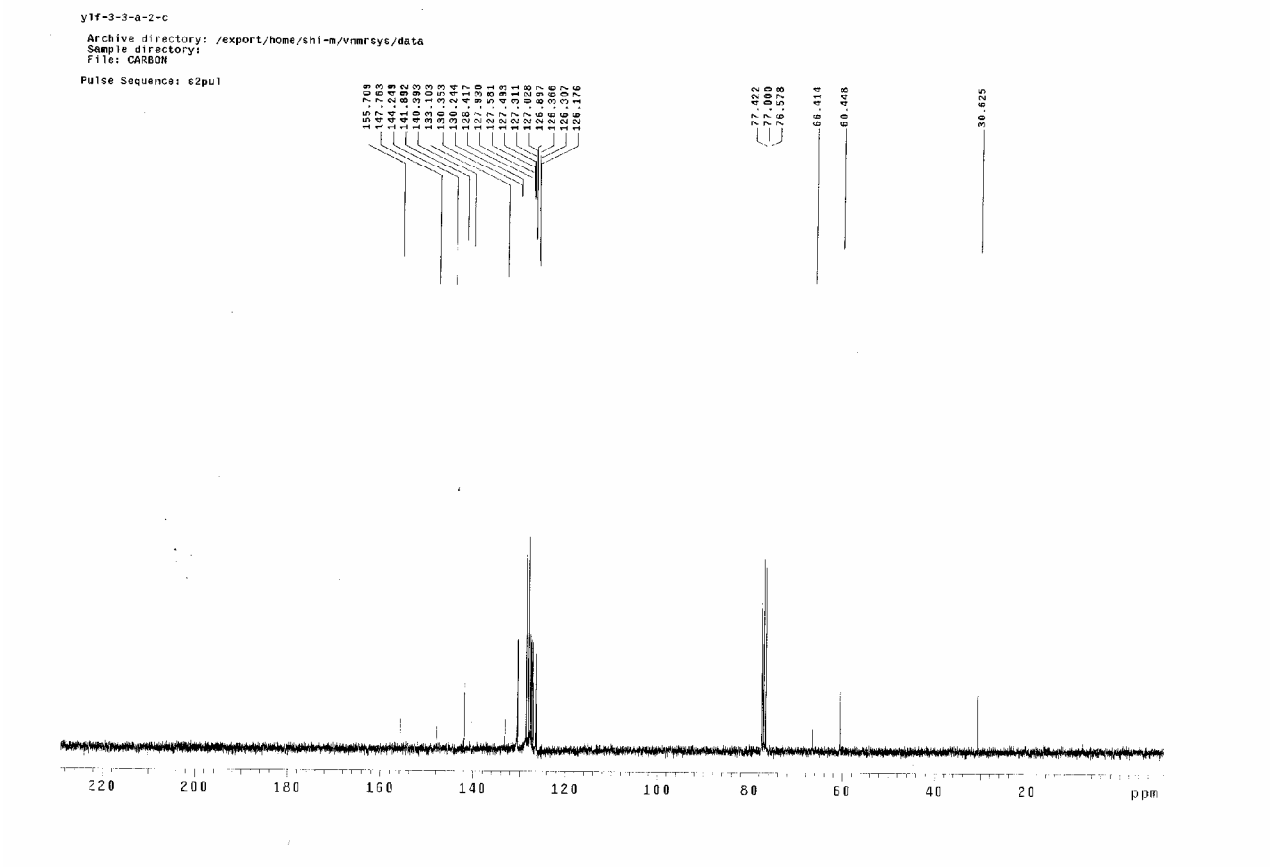


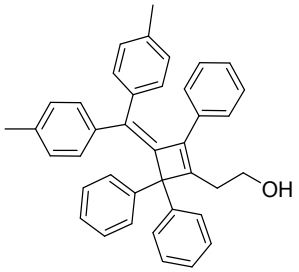


3j

A yellow solid, mp: 116-118 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 0.97 (t, J = 6.3 Hz, 1H, OH), 2.65 (t, J = 6.3 Hz, 2H, CH_2), 3.38 (q, J = 6.3 Hz, 2H, CH_2), 6.73 (d, J = 1.5 Hz, 2H, Ar), 6.76-7.08 (m, 13H, Ar), 7.17-7.26 (m, 6H, Ar), 7.31-7.34 (m, 4H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 30.6, 60.4, 66.4, 126.2, 126.3, 126.4, 126.9, 127.0, 127.3, 127.5, 127.6, 127.9, 128.4, 130.2, 130.4, 133.1, 140.4, 141.9, 144.2, 147.8, 155.7. IR (CH_2Cl_2) ν 3578, 3412, 3079, 3055, 3026, 2884, 1598, 1492, 1442, 1073, 1030, 916, 762, 747, 699, 636, 541 cm^{-1}. MS (MALDI) m/z 513 ($M+Na^+$). HRMS (MALDI) calcd. for $C_{37}H_{30}ONa^+$: 513.2198, Found: 513.2189.

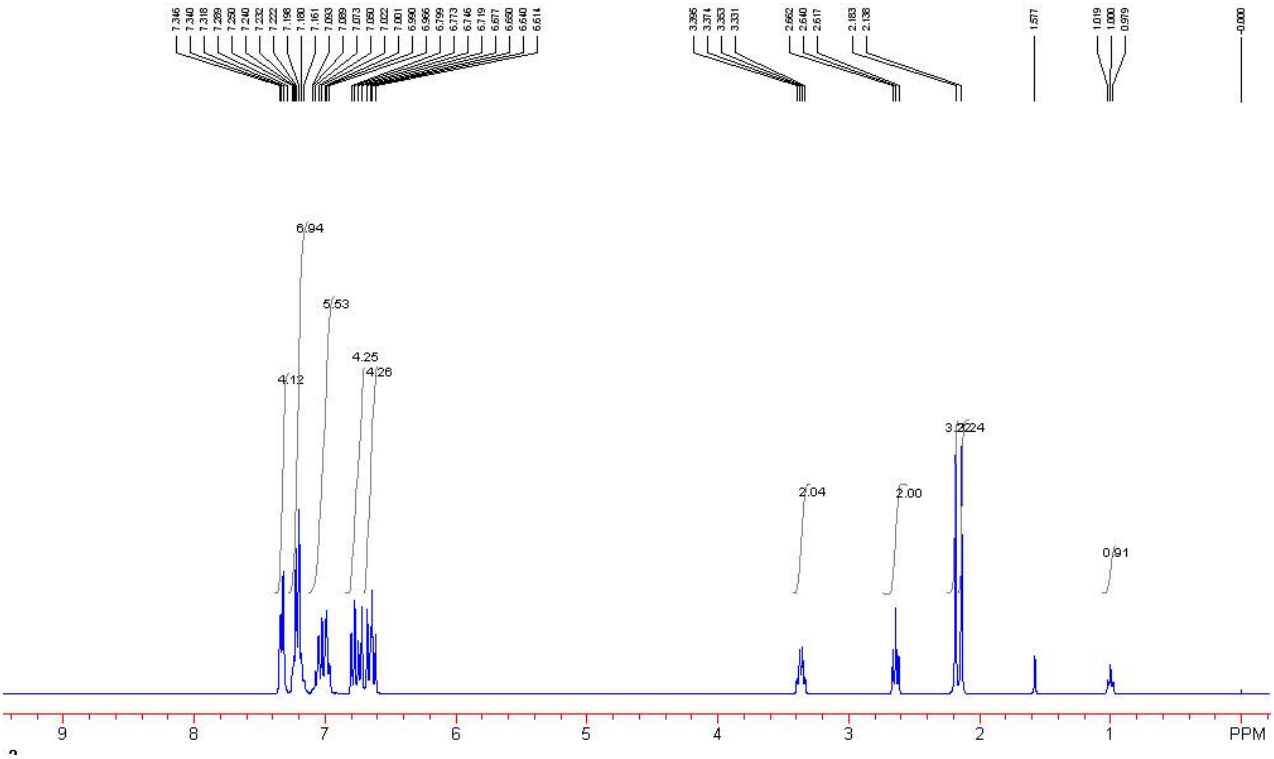

7.31
7.30
6.42
4.12
2.12
1.99
2.00
0.92
10
8
6
4
2
0PPM

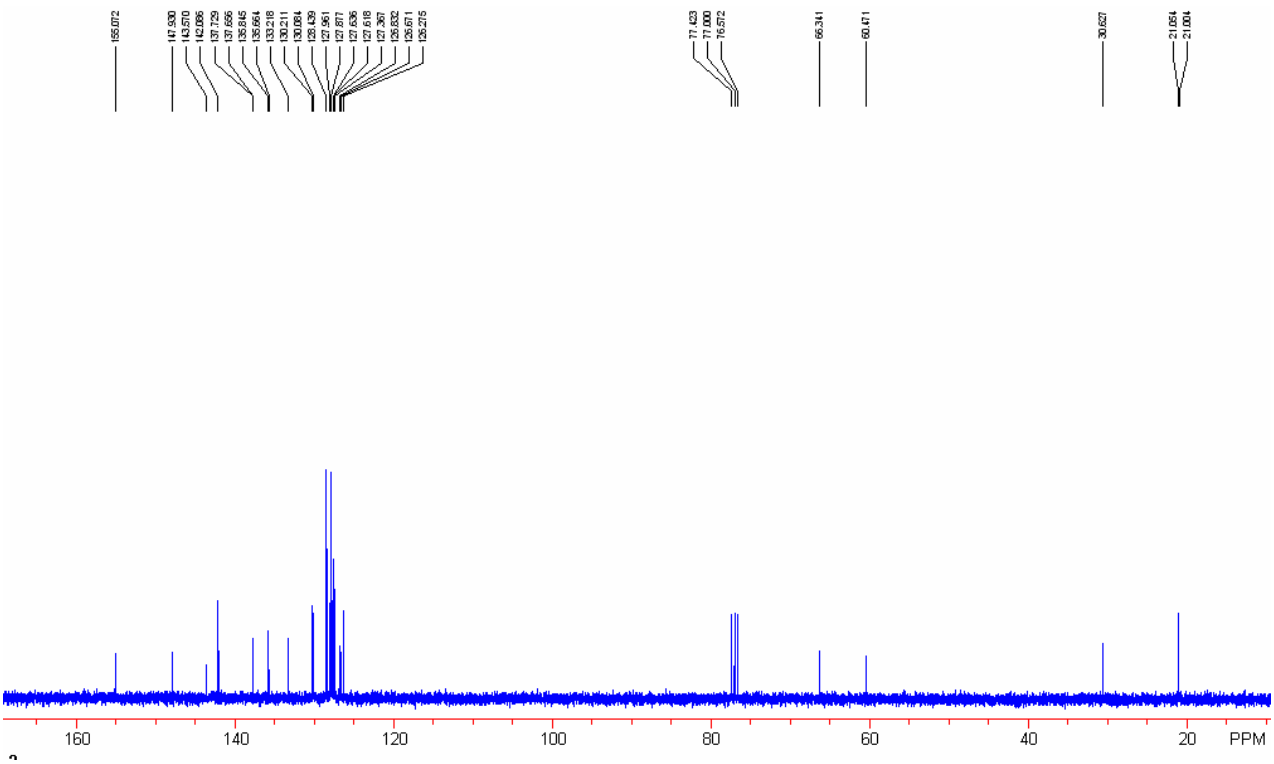

ylf-3-3-a-2-c
Archive directory: /export/home/shi-m/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
155.709
147.763
144.249
141.892
140.988
133.109
130.353
130.244
128.417
127.930
127.581
127.493
127.311
127.028
126.897
126.366
126.307
126.176
77.422
77.000
76.578
66.414
60.448
30.625
220
200
180
160
140
120
100
80
60
40
20
ppm

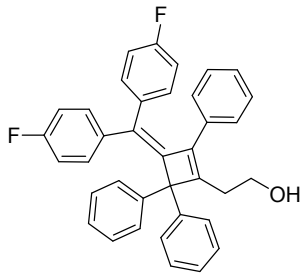


3k

A yellow solid, m.p. 161-163 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.00 (t, J = 6.6 Hz, 1H, OH), 2.14 (s, 3H, CH_3), 2.18 (s, 3H, CH_3), 2.64 (t, J = 6.6 Hz, 2H, CH_2), 3.36 (q, J = 6.6 Hz, 2H, CH_2), 6.61-6.80 (m, 8H, Ar), 6.97-7.07 (m, 5H, Ar), 7.16-7.25 (m, 6H, Ar), 7.32-7.35 (m, 4H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 20.99, 21.04, 30.6, 60.5, 66.3, 126.3, 126.7, 126.8, 127.4, 127.6, 127.9, 128.0, 128.4, 130.1, 130.2, 133.2, 135.7, 135.8, 137.66, 137.73, 142.1, 143.6, 147.9, 155.1. IR (CH_2Cl_2) ν 3578, 3385, 3080, 3055, 3023, 2920, 2885, 1940, 1886, 1805, 1598, 1509, 1492, 1443, 1406, 1266, 1181, 1110, 1034, 916, 820, 763, 738, 700, 672, 561, 538, 524, 514 cm^{-1}. ESI-MS (MALDI) m/z 518 (M^++1), 541(M^++Na). HRMS (MALDI) calcd. for $C_{39}H_{34}ONa^+$: 541.2486, Found: 541.2502.

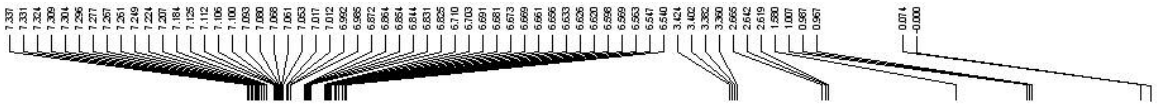


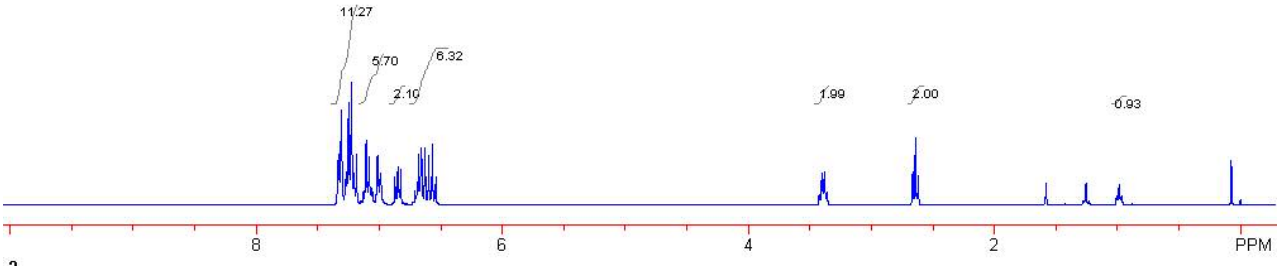


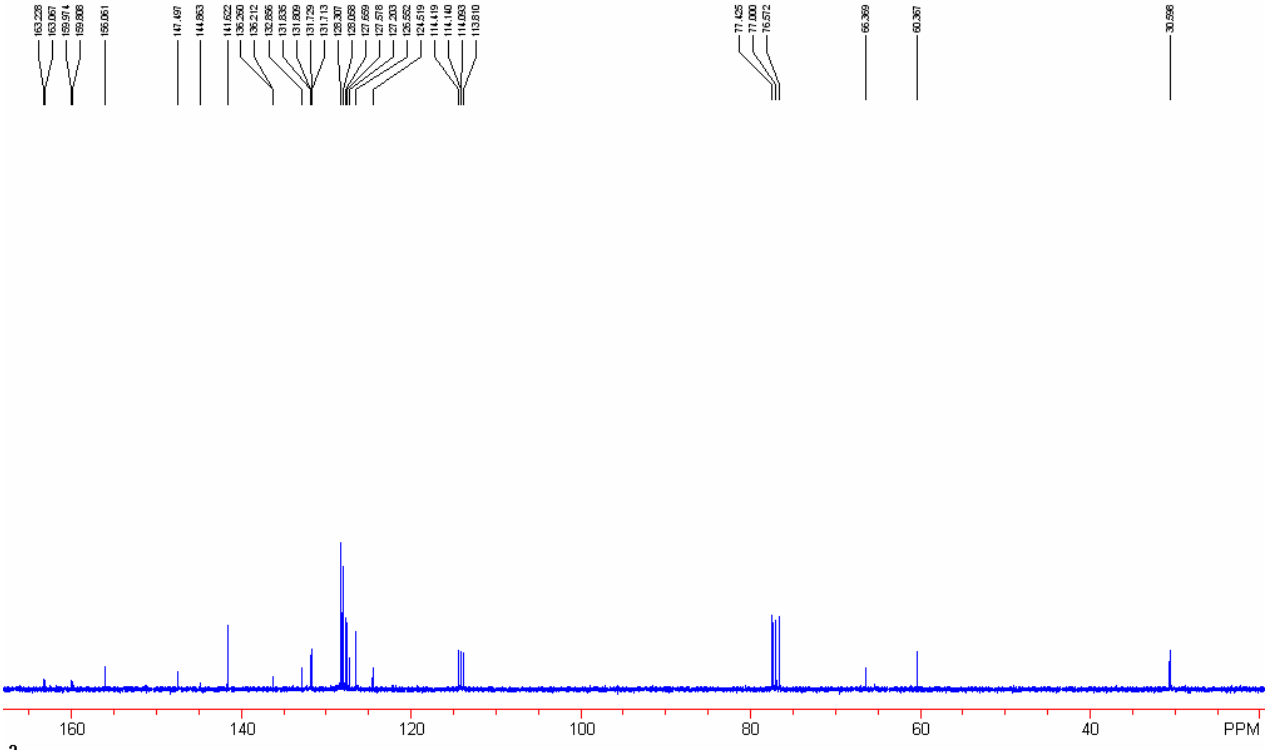


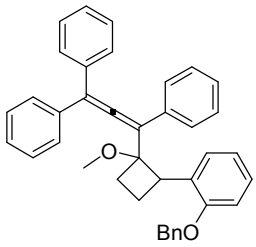
3l

A yellow solid, m.p. 154-156 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 0.99 (t, J = 6.6 Hz, 1H, OH), 2.64 (t, J = 6.6 Hz, 2H, CH_2), 3.39 (q, J = 6.6 Hz, 2H, CH_2), 6.54-6.71 (m, 6H, Ar), 6.82-6.87 (m, 2H, Ar), 6.98-7.13 (m, 5H, Ar), 7.18-7.34 (m, 10H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 30.6, 60.4, 66.4, 114.0 (d, J = 21.3 Hz), 114.3 (d, J = 21.3 Hz), 124.5, 126.6, 127.2, 127.6, 127.7, 127.9, 128.1, 128.2, 128.3, 131.8 (d, J = 8.2 Hz), 132.9, 136.2 (d, J = 3.3 Hz), 141.6, 144.9, 147.5, 156.1, 161.4 (d, J = 244.5 Hz), 161.6 (d, J = 244.5 Hz). IR (CH_2Cl_2) ν 3580, 3415, 3057, 3029, 2930, 2887, 1599, 1500, 1443, 1265, 1222, 1156, 1032, 1015, 836, 810, 765, 739, 701, 568, 526 cm^{-1}. MS (MALDI) m/z 549 ($M+Na^+$). HRMS (MALDI) calcd. for $C_{37}H_{28}OF_2Na^+$: 549.2014, Found: 549.2000.



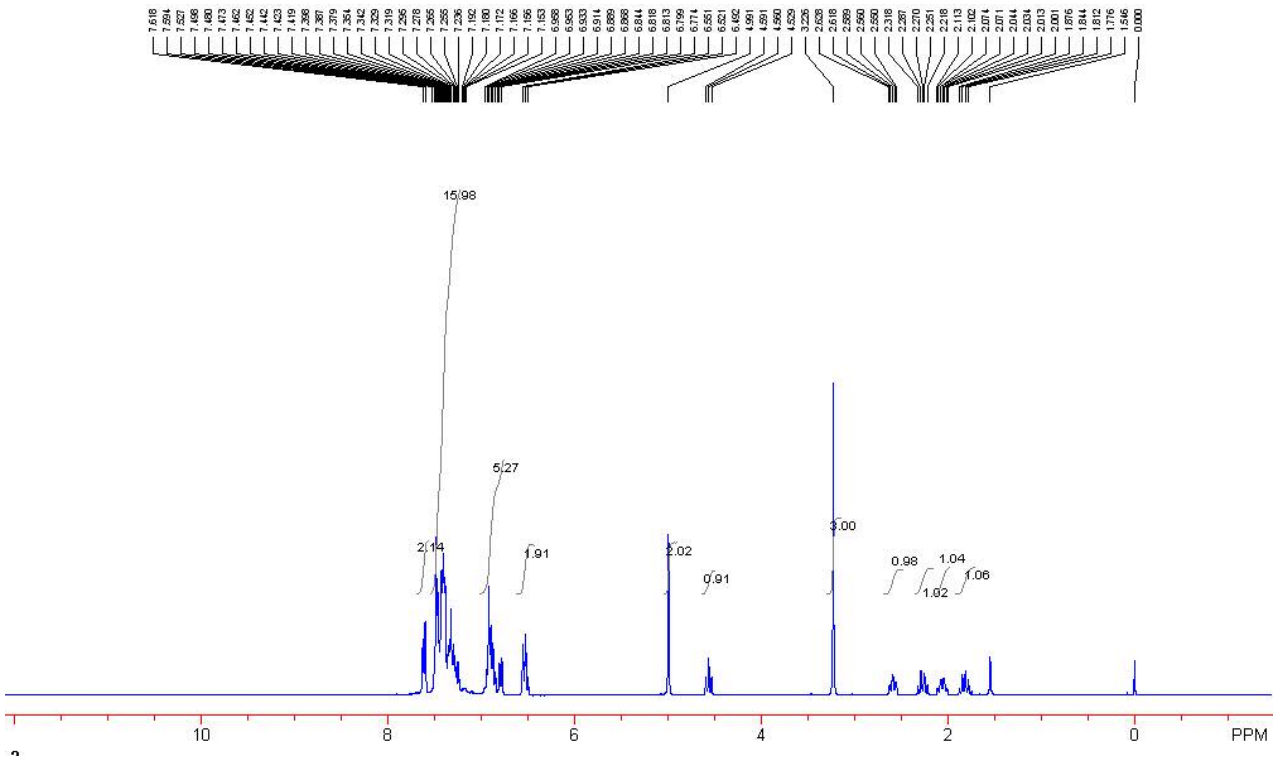

11.27
5.70
6.32
2.10
1.99
2.00
0.93
8
6
4
2
PPM

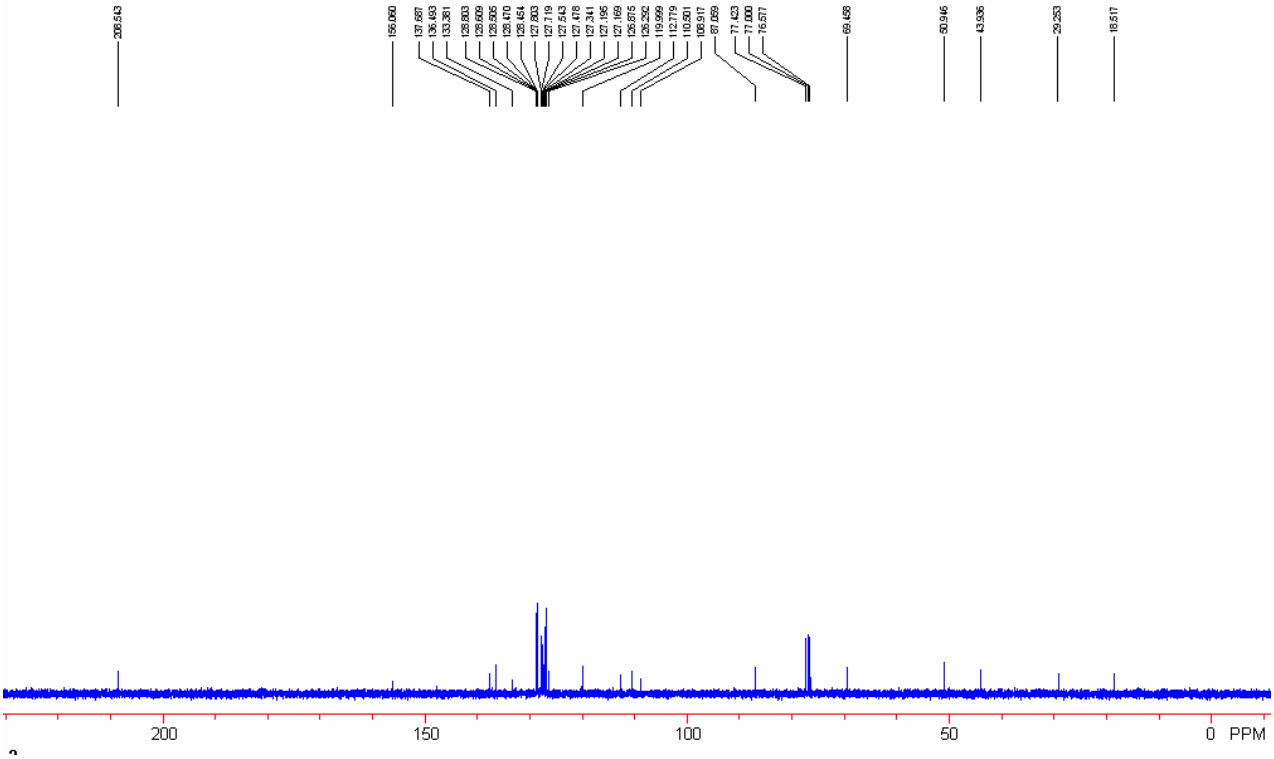

160
140
120
100
80
60
40
PPM

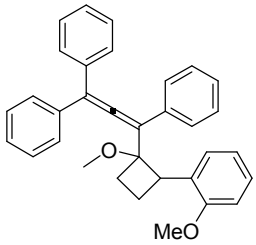

O
BnO

4a

A white solid, mp: 147-149 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.75-1.88 (m, 1H, CH_2), 2.01-2.11 (m, 1H, CH_2), 2.22-2.32 (m, 1H, CH_2), 2.55-2.63 (m, 1H, CH_2), 3.23 (s, 3H, OCH_3), 4.56 (t, J = 9.3 Hz, 1H, CH), 4.99 (s, 2H, CH_2), 6.49-6.55 (m, 2H, Ar), 6.78-6.95 (m, 5H, Ar), 7.22-7.48 (m, 15H, Ar), 7.60 (d, J = 7.5 Hz, 2H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 18.5, 29.3, 43.9, 50.9, 69.5, 87.1, 108.9, 110.5, 112.8, 120.0, 126.3, 126.9, 127.2, 127.3, 127.5, 127.6, 127.7, 127.8, 128.46, 128.51, 128.6, 128.8, 133.4, 136.5, 137.7, 156.1, 208.6. IR (CH_2Cl_2) ν 3057, 3028, 2985, 2936, 2820, 1598, 1491, 1450, 1243, 1186, 1112, 1051, 1028, 767, 749, 731, 694, 595 cm^{-1}. MS (MALDI) m/z 568 (M+Na^{+}). Anal. calcd. for $C_{39}H_{34}O_2$: C, 87.61%; H, 6.41%; Found: C, 87.96%; H, 6.57%.

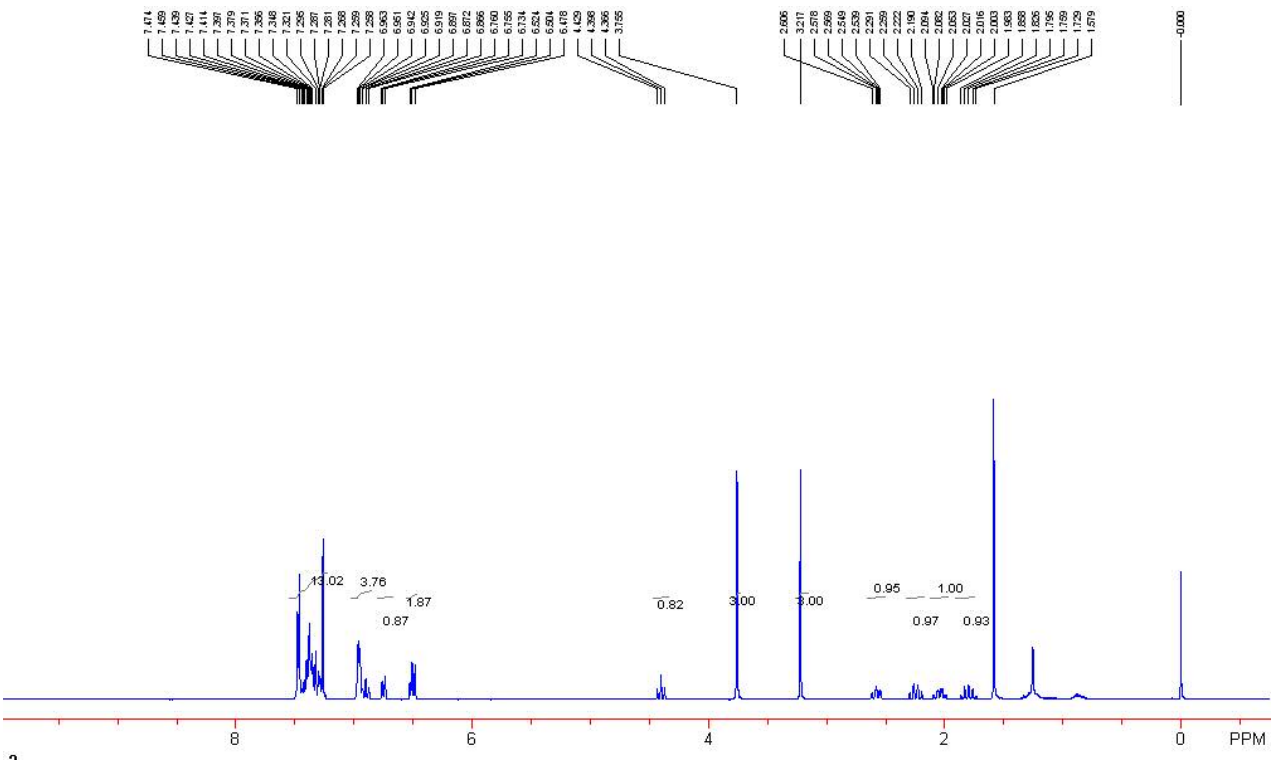


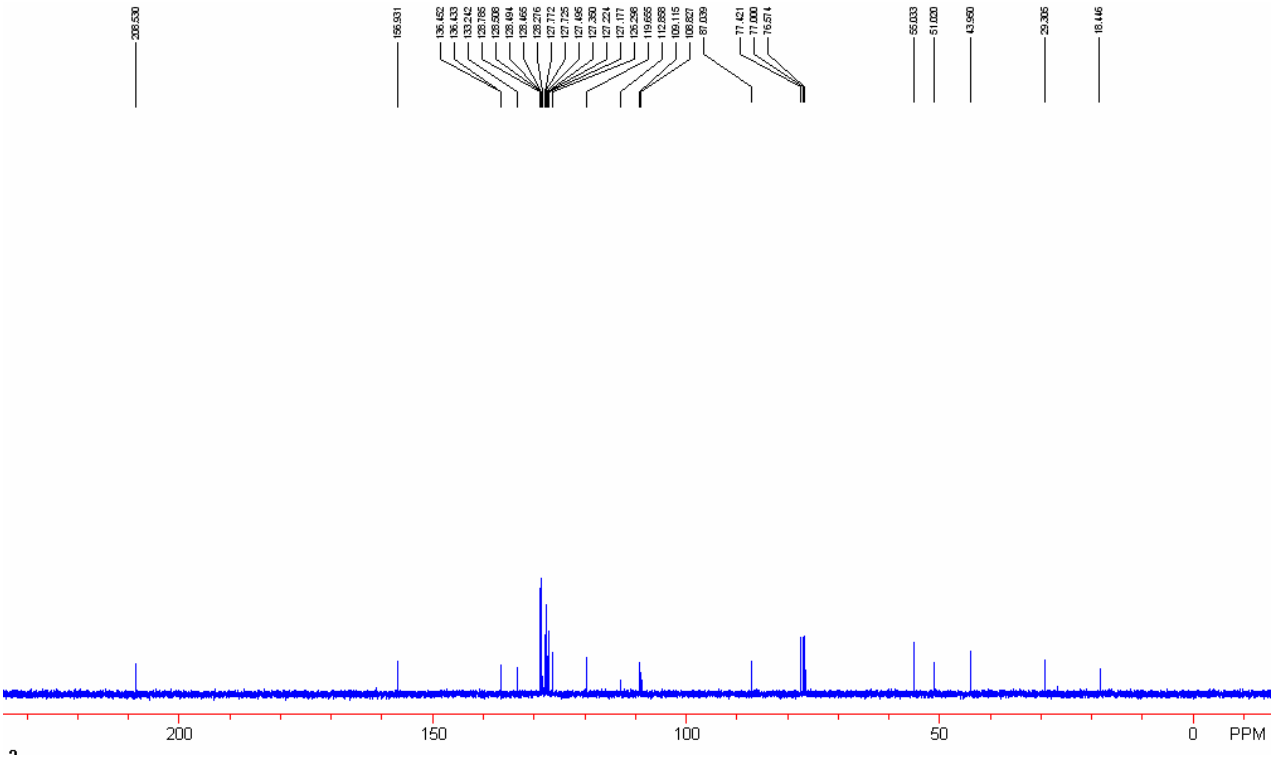


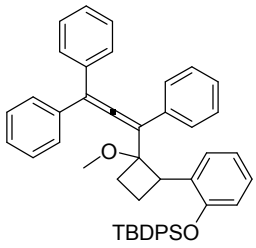


4b

A yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.76-1.86 (m, 1H, CH_2), 1.99-2.10 (m, 1H, CH_2), 2.19-2.30 (m, 1H, CH_2), 2.54-2.62 (m, 1H, CH_2), 3.22 (s, 3H, OCH_3), 3.76 (s, 3H, OCH_3), 4.40 (t, J = 9.3 Hz, 1H, CH), 6.48-6.53 (m, 2H, Ar), 6.74-6.77 (m, 1H, Ar), 6.87-6.97 (m, 4H, Ar), 7.26-7.48 (m, 12H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 18.4, 29.3, 43.9, 51.0, 55.0, 87.0, 108.8, 109.1, 112.9, 119.7, 126.3, 127.18, 127.22, 127.4, 127.5, 127.7, 127.8, 128.3, 128.47, 128.50, 128.8, 133.2, 136.4, 156.9, 208.5. IR (CH_2Cl_2) ν 3056, 3027, 2985, 2936, 2900, 2833, 2821, 1923, 1598, 1493, 1463, 1445, 1291, 1248, 1108, 1051, 1030, 921, 899, 814, 797, 767, 695, 671, 629, 609, 596, 509 cm^{-1}. MS (MALDI) m/z 481 ($M+Na^+$). HRMS (MALDI) calcd. for $C_{33}H_{30}O_2Na^+$: 481.2139, Found: 481.2138.

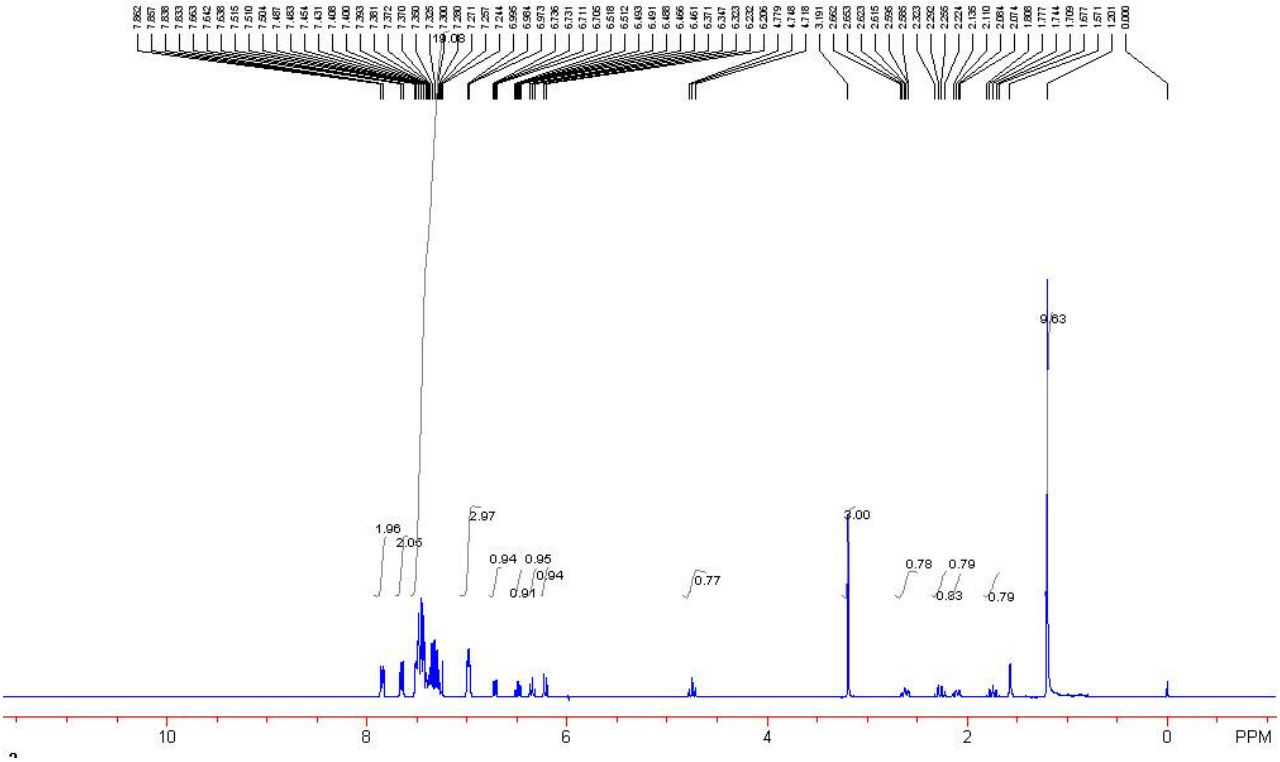

13.02
3.76
1.87
0.87
0.82
3.00
3.00
0.95
1.00
0.97
0.93
8
6
4
2
0
PPM

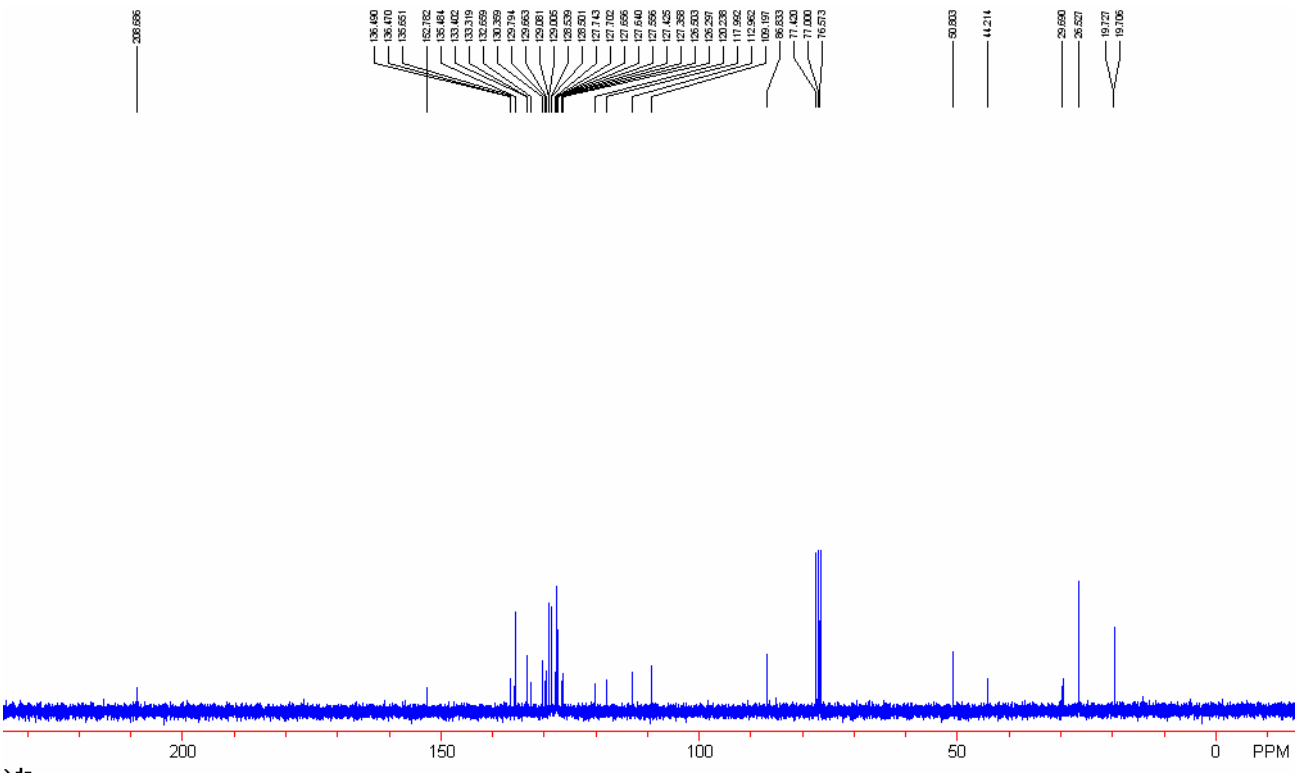

200
150
100
50
0
PPM

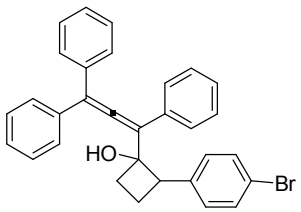


4c

A yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.20 (s, 9H, CH_3), 1.71-1.78 (m, 1H, CH_2), 2.07-2.22 (m, 1H, CH_2), 2.26-2.32 (m, 1H, CH_2), 2.59-2.66 (m, 1H, CH_2), 3.19 (s, 3H, OCH_3), 4.75 (t, *J* = 9.3 Hz, 1H, CH), 6.22 (d, *J* = 7.8 Hz, 1H, Ar), 6.35 (t, *J* = 7.5 Hz, 1H, Ar), 6.46-6.52 (m, 1H, Ar), 6.72 (dd, *J* = 1.5, 7.5 Hz, 1H, Ar), 6.98 (t, *J* = 3.3 Hz, 3H, Ar), 7.24-7.52 (m, 18H, Ar), 7.65 (d, *J* = 7.0 Hz, 2H, Ar), 7.85 (dd, *J* = 1.5, 7.5 Hz, 2H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 19.7, 26.5, 29.7, 44.2, 50.8, 86.8, 109.2, 113.0, 118.0, 120.2, 126.3, 126.5, 127.36, 127.43, 127.56, 127.64, 127.70, 127.75, 128.51, 128.54, 129.0, 129.1, 129.7, 129.8, 130.4, 132.7, 133.3, 133.4, 135.5, 135.7, 136.5, 152.8, 208.7. IR (CH_2Cl_2) ν 3054, 3025, 2942, 2895, 2859, 2821, 1923, 1653, 1597, 1578, 1490, 1473, 1451, 1429, 1256, 1184, 1144, 1111, 1052, 1045, 928, 921, 822, 782, 774, 761, 753, 741, 715, 704, 696, 676, 670, 650, 629, 623, 612, 598, 591, 581, 565, 559, 548, 534, 528, 504 cm^{-1}. MS (MALDI) m/z 705 ($M+Na^+$). HRMS (MALDI) calcd. for $C_{48}H_{46}O_2SiNa^+$: 705.3166, Found: 705.3159.

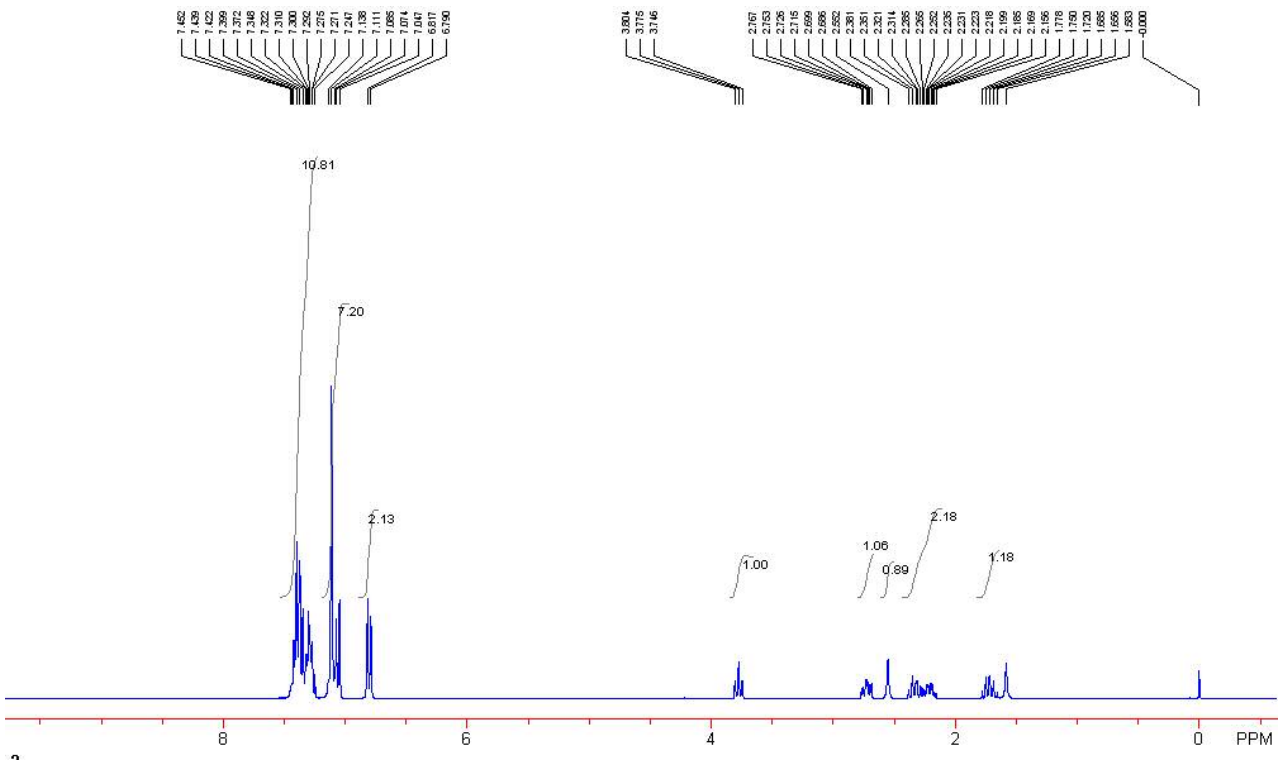


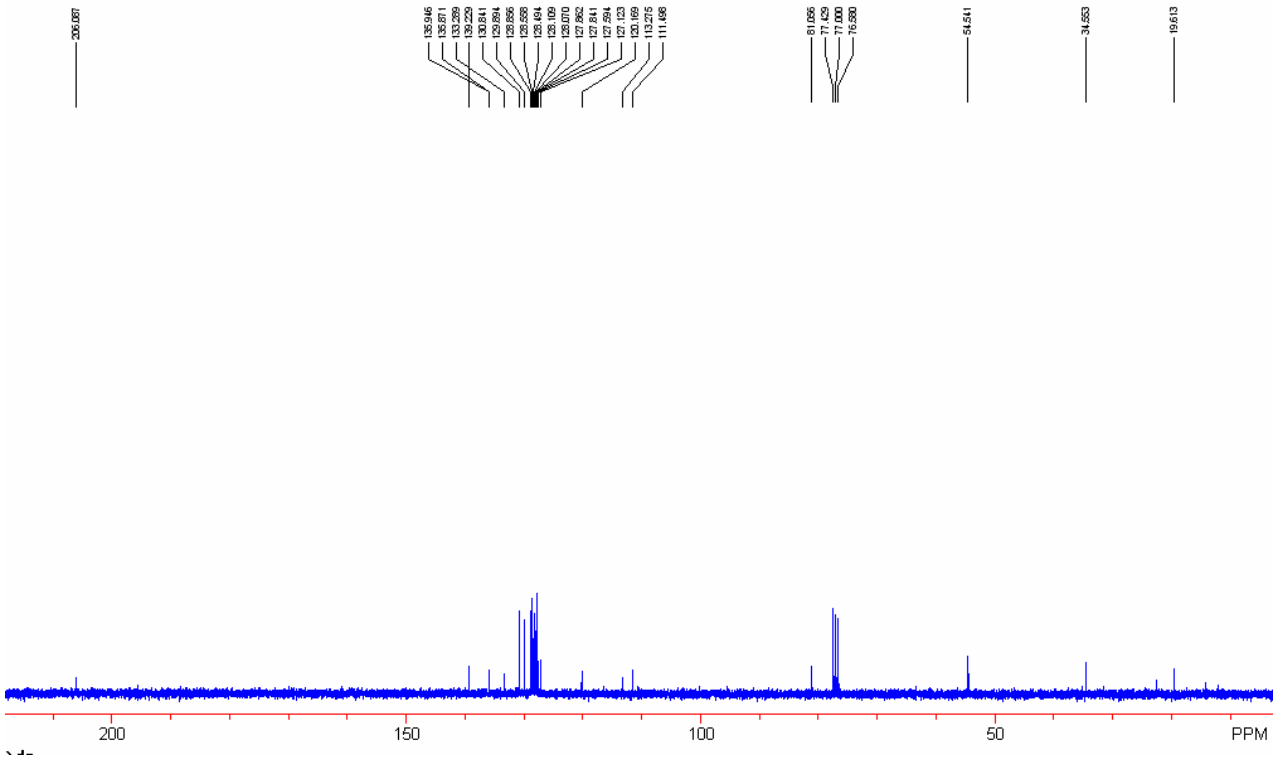


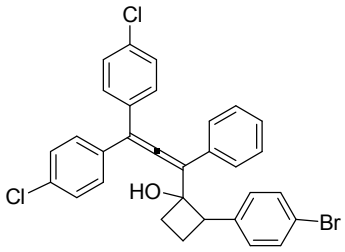


4d

A yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.67-1.78 (m, 1H, CH_2), 2.16-2.38 (m, 2H, CH_2), 2.55 (s, 1H, OH), 2.69-2.77 (m, 1H, CH_2), 3.78 (t, J = 8.7 Hz, 1H, CH), 6.80 (d, J = 7.5 Hz, 2H, Ar), 7.05-7.14 (m, 7H, Ar), 7.25-7.45 (m, 10H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 19.6, 34.5, 54.5, 81.1, 111.5, 113.3, 120.2, 127.1, 127.6, 127.8, 128.07, 128.11, 128.5, 128.6, 128.9, 129.9, 130.8, 133.3, 135.87, 135.95, 139.2, 206.1. IR (CH_2Cl_2) ν 3556, 3395, 3080, 3057, 3026, 2985, 2948, 1922, 1883, 1716, 1596, 1491, 1444, 1113, 1073, 1010, 921, 831, 768, 735, 695, 666, 634, 611, 599, 572, 535 cm^{-1}. MS (MALDI) m/z 515 ($M+Na^+$). HRMS (MALDI) calcd. for $C_{31}H_{25}OBrNa^+$: 515.0990, Found: 515.0981.

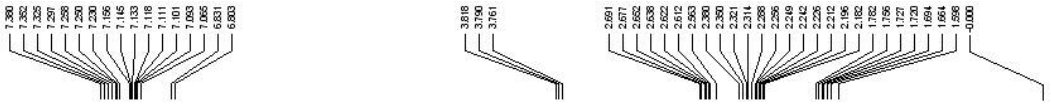

10.81
7.20
2.13
1.00
1.06
0.89
2.18
1.18
8
6
4
2
0
PPM

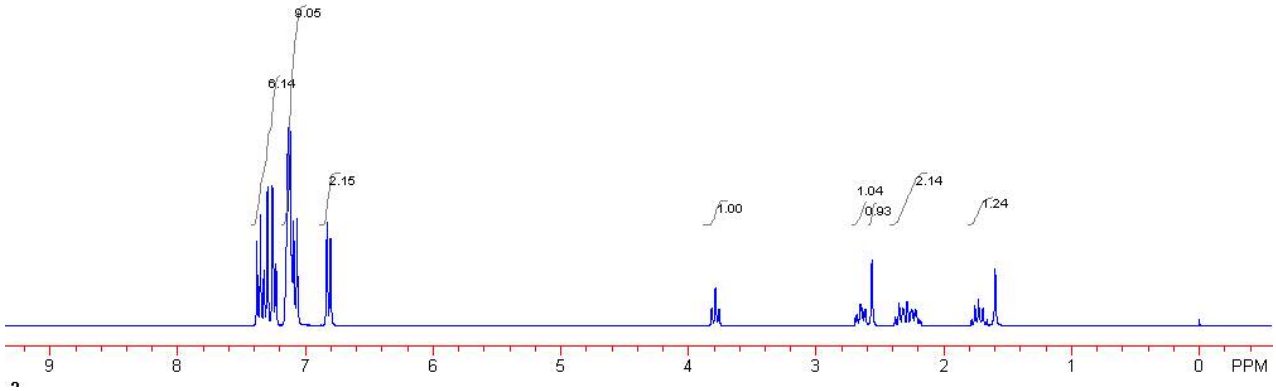

200
150
100
50
PPM

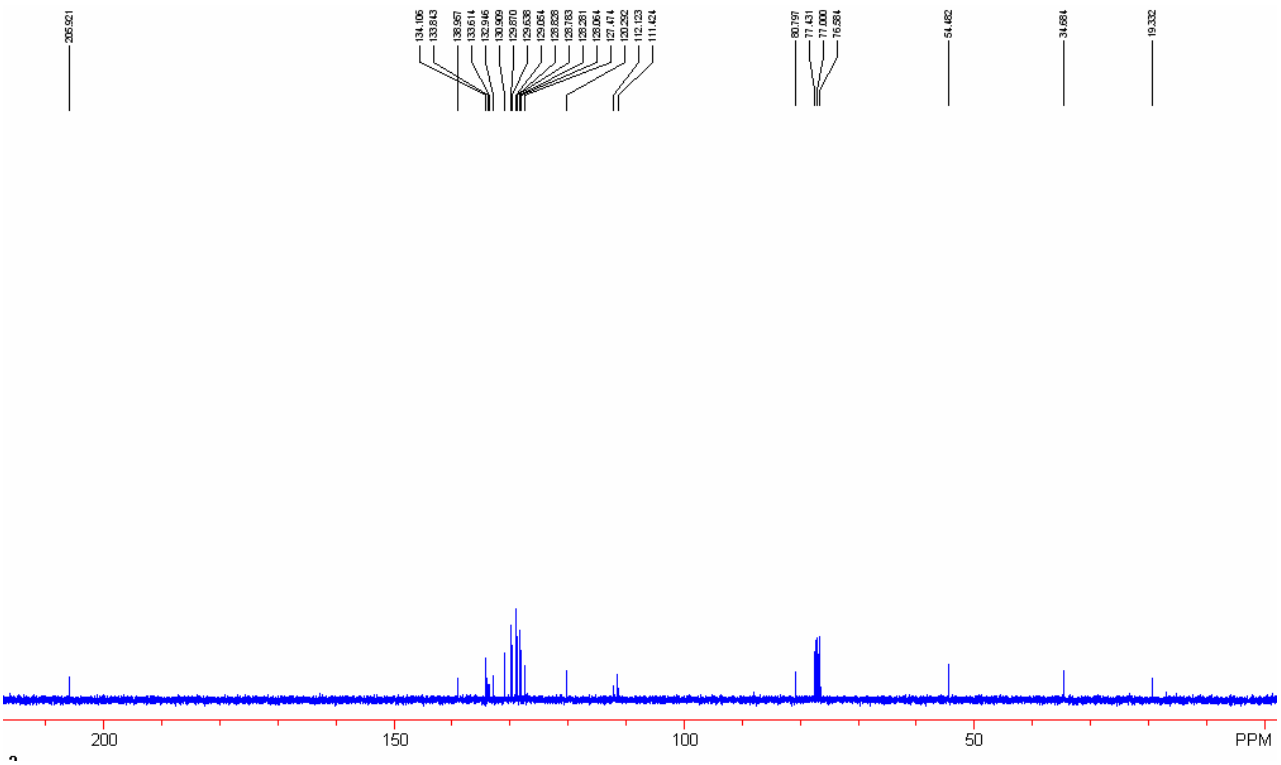


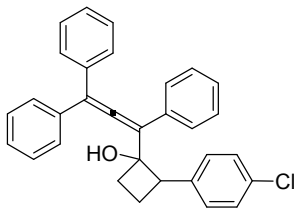
4e

A yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.66-1.78 (m, 1H, CH_2), 2.18-2.38 (m, 2H, CH_2), 2.56 (s, 1H, OH), 2.61-2.69 (m, 1H, CH_2), 3.79 (t, J = 8.4 Hz, 1H, CH), 6.82 (d, J = 8.4 Hz, 2H, Ar), 7.06-7.14 (m, 9H, Ar), 7.23-7.38 (m, 6H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 19.3, 34.7, 54.5, 80.8, 111.4, 112.1, 120.3, 127.5, 128.1, 128.3, 128.79, 128.83, 129.1, 129.6, 129.9, 130.9, 132.9, 133.6, 133.8, 134.1, 139.0, 205.9. IR (CH_2Cl_2) ν 3566, 3451, 3432, 3356, 3349, 3082, 3054, 3026, 2983, 2952, 2871, 1920, 1901, 1590, 1488, 1450, 1408, 1396, 1264, 1233, 1211, 1140, 1128, 1112, 1100, 1087, 1069, 1012, 994, 978, 965, 930, 907, 836, 830, 821, 814, 803, 766, 756, 739, 726, 714, 703, 695, 667, 634, 617, 605, 573, 566, 559, 548, 542, 534, 517, 510 cm^{-1}. MS (MALDI) m/z 561 ($M+H^+$). HRMS (MALDI) calcd. for $C_{31}H_{24}OCl_2Br^+$: 561.0365, Found: 561.0382.



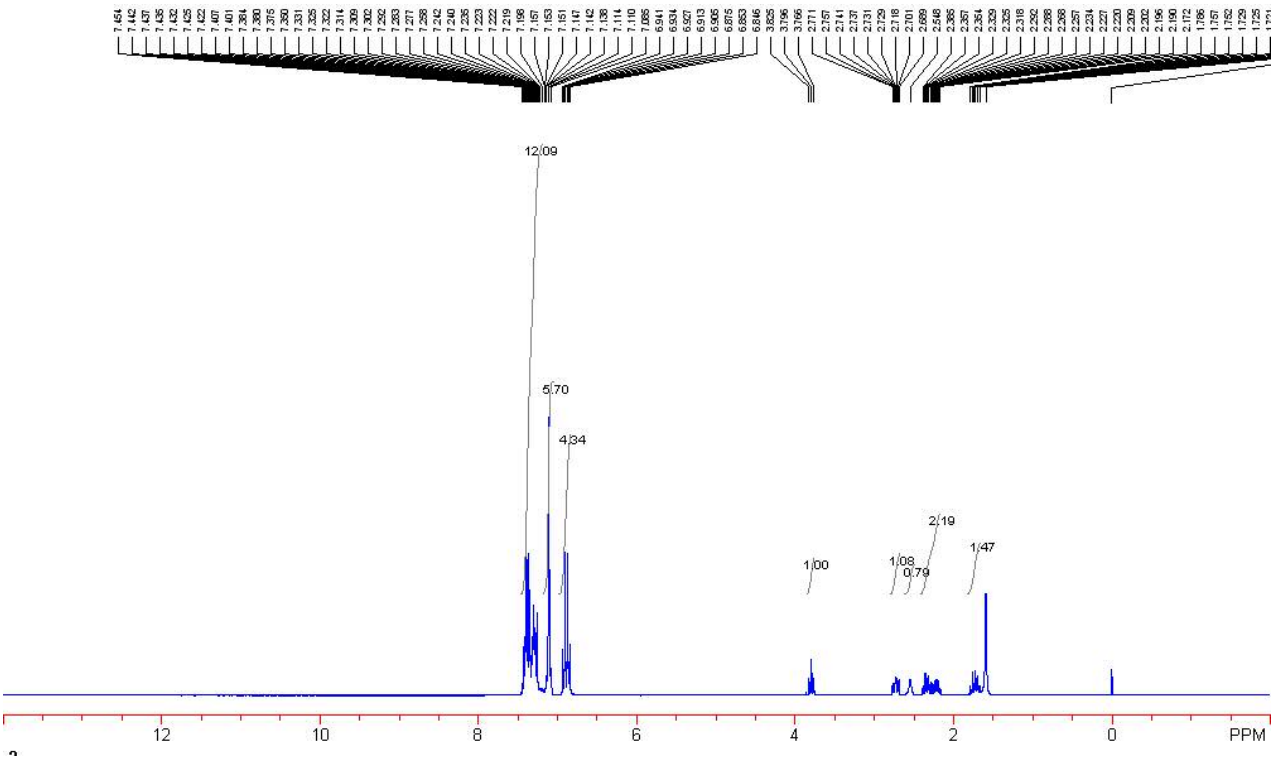






4f

A yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.66-1.78 (m, 1H, CH_2), 2.16-2.38 (m, 2H, CH_2), 2.54 (s, 1H, OH), 2.69-2.77 (m, 1H, CH_2), 3.80 (t, J = 9.0 Hz, 1H, CH), 6.85 (d, J = 6.6 Hz, 2H, Ar), 6.93 (d, J = 6.6 Hz, 2H, Ar), 7.01-7.11 (m, 6H, Ar), 7.28-7.45 (m, 9H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 19.7, 34.5, 54.5, 81.1, 111.5, 113.3, 127.1, 127.6, 127.8, 127.9, 128.1, 128.5, 128.6, 128.9, 129.5, 132.0, 133.3, 135.9, 138.7, 206.1. IR (CH_2Cl_2) ν 3552, 3439, 3081, 3057, 3026, 2984, 2870, 1923, 1890, 1728, 1680, 1660, 1596, 1578, 1492, 1445, 1265, 1239, 1117, 1092, 1031, 1014, 1001, 969, 918, 901, 833, 773, 735, 693, 664, 635, 627, 609, 596, 574, 563, 548, 543, 535, 518, 509, 503 cm^{-1}. MS (MALDI) m/z 471 ($M+Na^+$). HRMS (MALDI) calcd. for $C_{31}H_{25}OClNa^+$: 471.1499, Found: 471.1486.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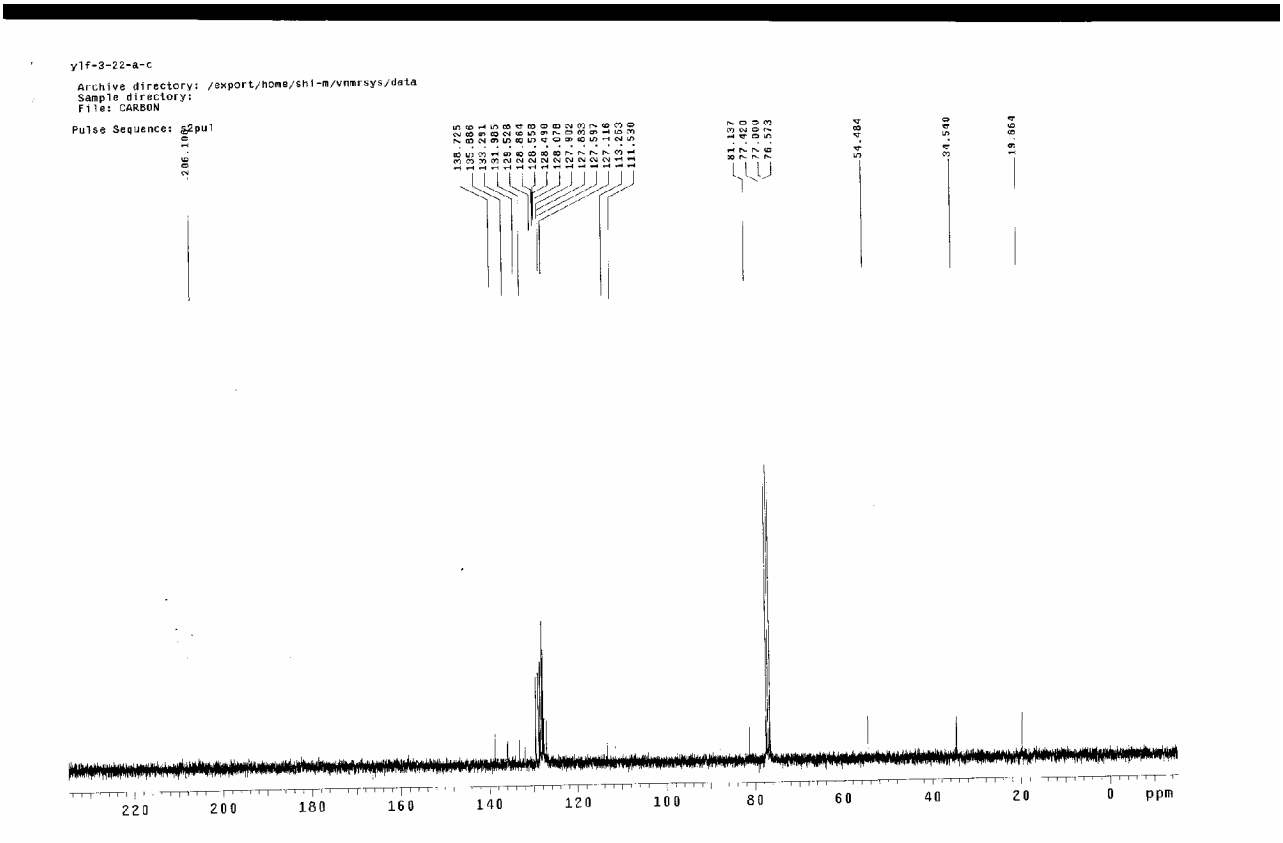
2.19
1.47
1.00
1.08
0.79
12
10
8
6
4
2
0
PPM


ylf-3-22-a-c
Archive directory: /export/home/shi-m/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
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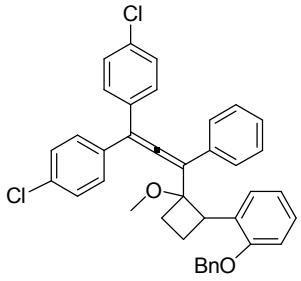
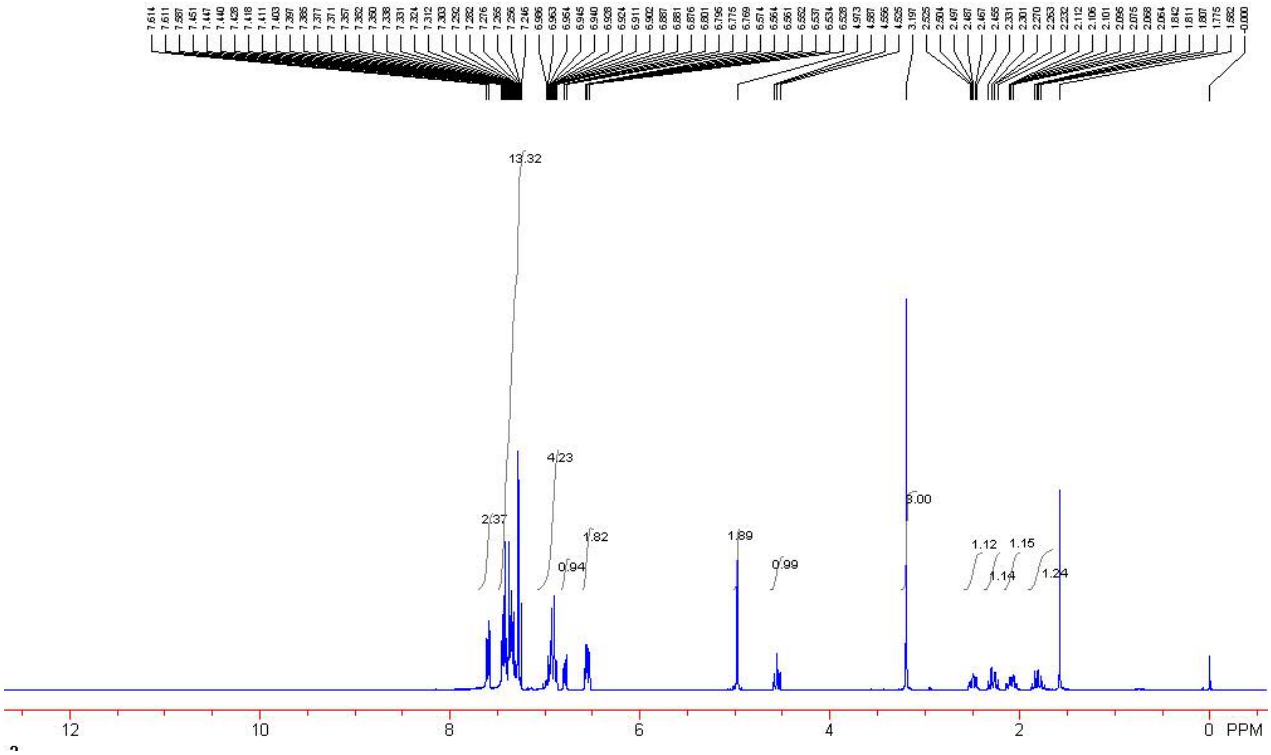
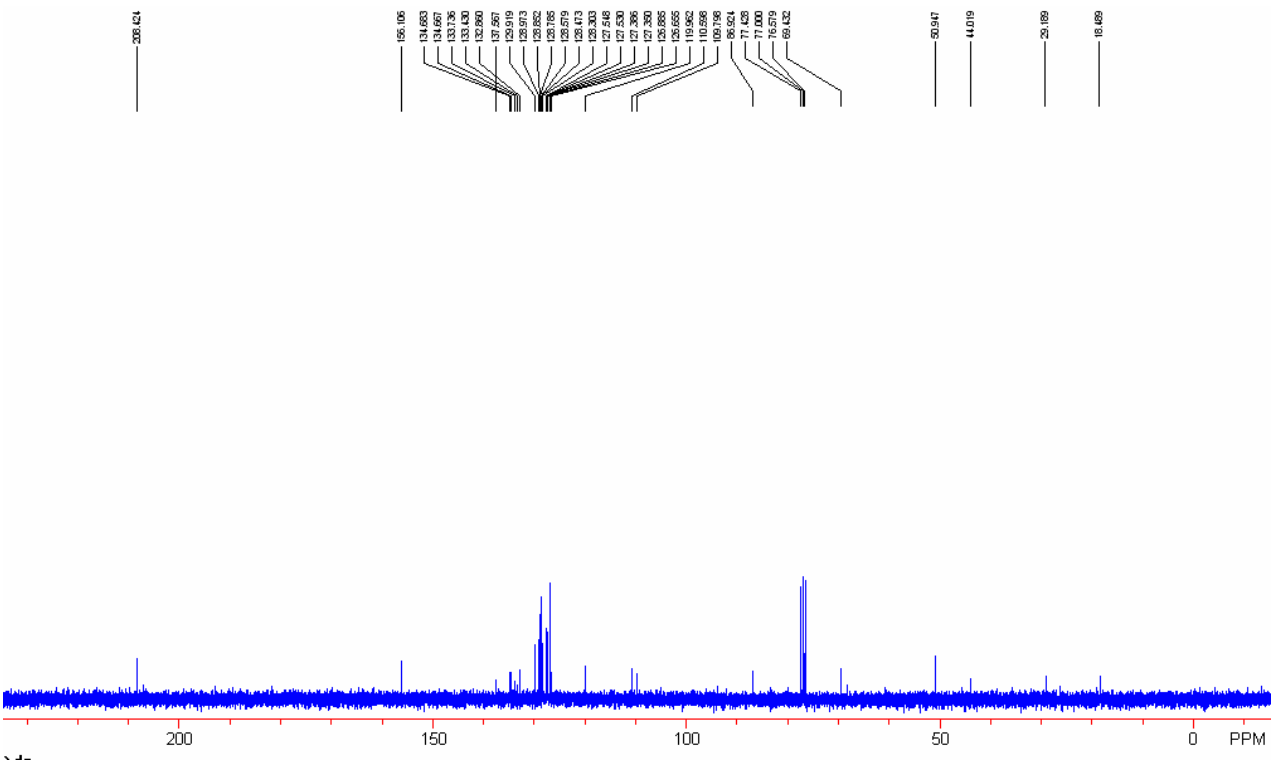
138.725
135.866
133.291
131.985
129.528
128.864
128.558
128.430
128.078
127.912
127.833
127.597
127.116
113.263
111.530
81.137
77.420
77.000
76.573
54.484
34.540
19.864
220
200
180
160
140
120
100
80
60
40
20
0
ppm

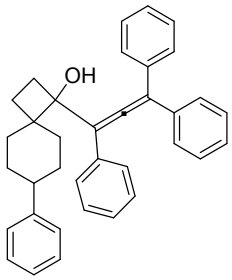


4g

A yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.77-1.87 (m, 1H, CH_2), 2.03-2.14 (m, 1H, CH_2), 2.23-2.33 (m, 1H, CH_2), 2.46-2.54 (m, 1H, CH_2), 3.20 (s, 3H, OCH_3), 4.56 (t, *J* = 9.3 Hz, 1H, CH), 4.97 (s, 2H, CH_2), 6.53-6.57 (m, 2H, Ar), 6.82 (dd, *J* = 1.8, 7.7 Hz, 1H, Ar), 6.88-6.98 (m, 4H, Ar), 7.25-7.45 (m, 13H, Ar), 7.60 (d, *J* = 7.7 Hz, 2H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 18.5, 29.2, 44.0, 50.9, 69.4, 86.9, 109.8, 110.6, 120.0, 126.7, 126.9, 127.35, 127.39, 127.6, 128.3, 128.5, 128.6, 128.8, 128.9, 129.0, 130.0, 132.9, 133.4, 133.7, 134.7, 137.6, 156.1, 208.4. IR (CH_2Cl_2) ν 3060, 2936, 2811, 1911, 1589, 1490, 1456, 1223, 839, 830, 820, 775, 770, 759, 749, 740, 728, 702, 688, 668, 643, 631, 618, 611, 599, 584, 573, 562, 549, 540, 531 cm^{-1}. MS (MALDI) m/z 625 ($M+Na^+$). HRMS (MALDI) calcd. for $C_{39}H_{32}O_2Cl_2Na^+$: 625.1670, Found: 625.1672.

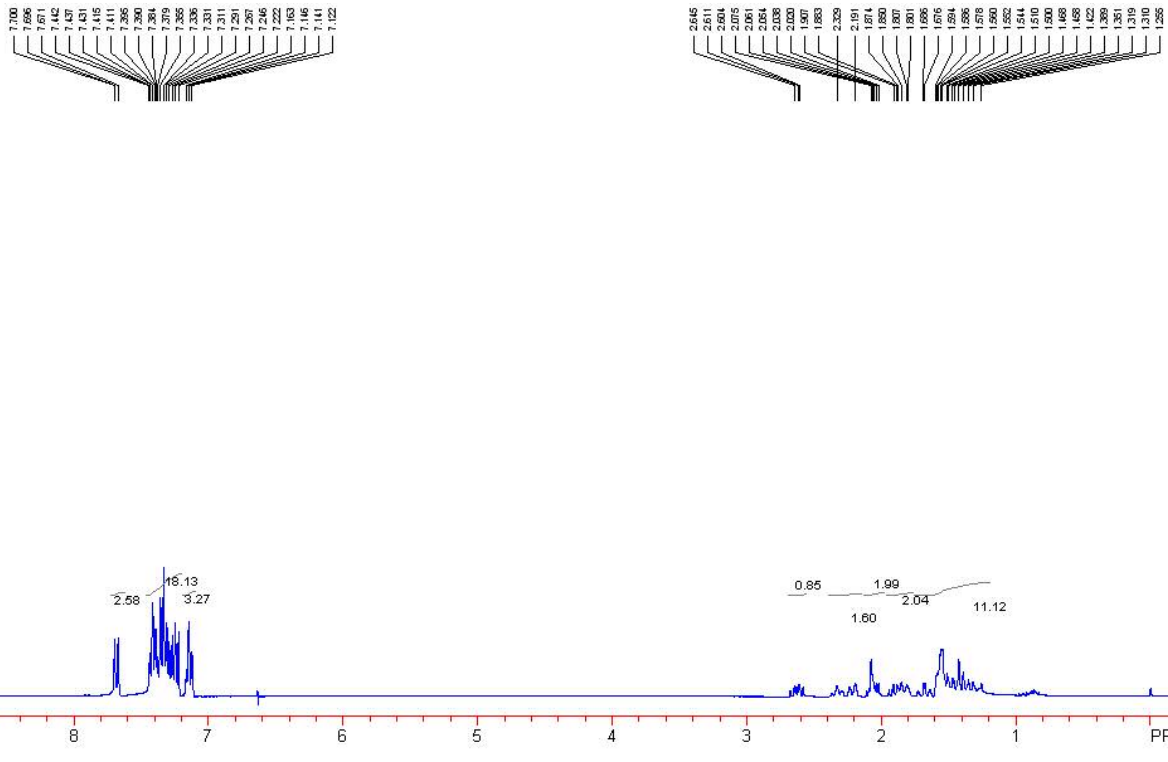


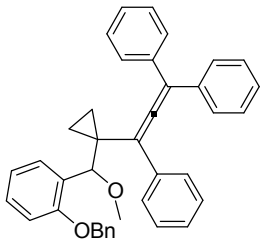




4h

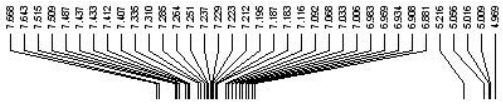
A yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.26-1.73 (m, 7H, CH_2), 1.81-1.94 (m, 2H, CH_2), 2.02-2.11 (m, 2H, CH_2), 2.20 (dd, *J* = 1.8, 12.9 Hz, 1H, CH_2), 2.23-2.37 (m, 1H, CH_2), 2.58-2.67 (m, 1H, CH), 7.12-7.16 (m, 3H, Ar), 7.22-7.44 (m, 15H, Ar), 7.68 (dd, *J* = 1.2, 8.4 Hz, 2H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 25.4, 29.8, 30.2, 32.1, 32.2, 34.4, 43.8, 48.3, 81.0, 113.0, 113.6, 125.8, 126.7, 127.2, 127.5, 128.23, 128.25, 128.28, 128.41, 128.45, 128.53, 134.8, 136.2, 136.3, 147.3, 207.2. IR (CH_2Cl_2) ν 3078, 3059, 3024, 2930, 2851, 1944, 1920, 1798, 1725, 1665, 1597, 1491, 1444, 1298, 1265, 1223, 1182, 1162, 1130, 1116, 1070, 1029, 999, 969, 937, 922, 900, 843, 769, 758, 739, 665, 647, 633, 611, 596, 545, 532 cm^{-1}. MS (MALDI) m/z 505 ($M+Na^+$). HRMS (MALDI) calcd. for $C_{36}H_{34}ONa^+$: 505.2504, Found: 505.2502.

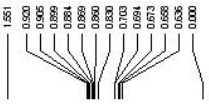

18.13
2.58
3.27
0.85
1.99
2.04
1.60
11.12
9
8
7
6
5
4
3
2
1
PPM
200
150
100
50
0
PPM

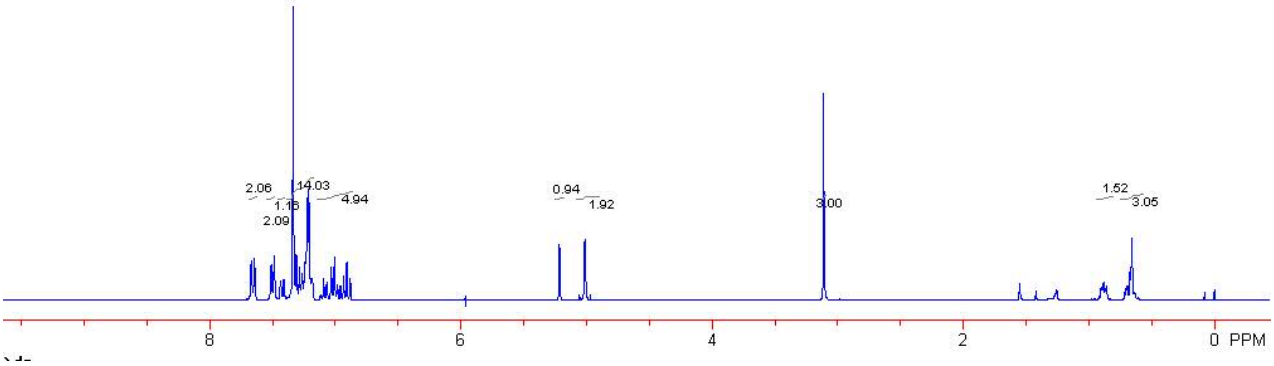


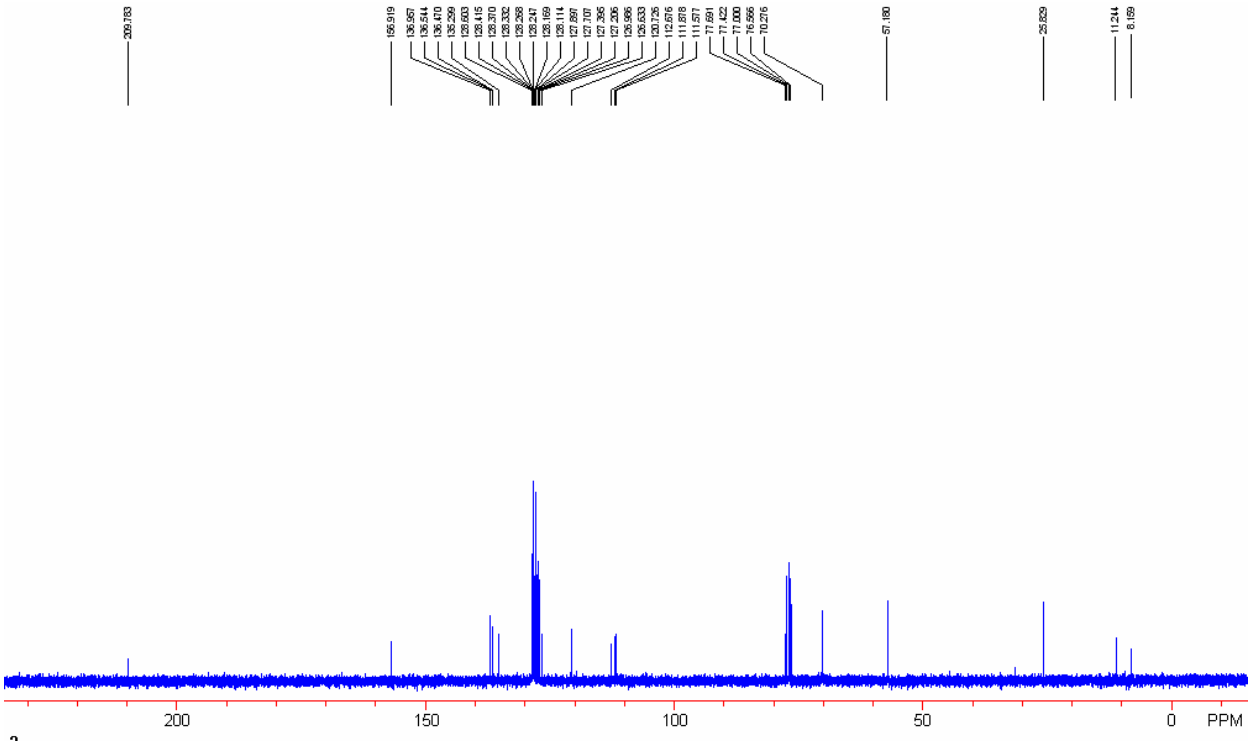
5a

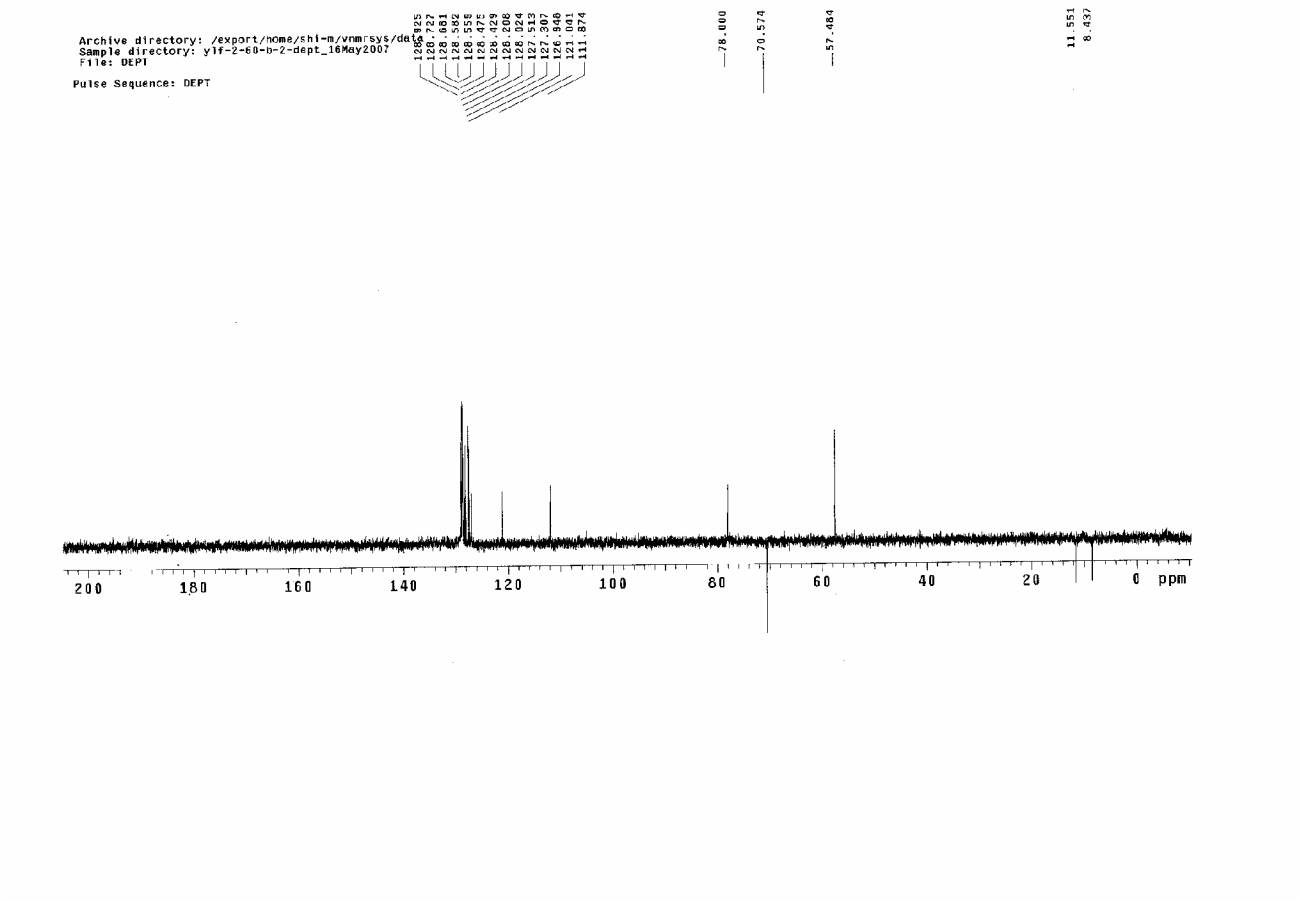
A yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 0.64-0.70 (m, 3H, CH_2), 0.86-0.91 (m, 1H, CH_2), 3.11 (s, 3H, OCH_3), 5.01 (d, *J* = 12.0 Hz, 1H, CH_2), 5.03 (d, *J* = 12.0 Hz, 1H, CH_2), 5.22 (s, 1H, CH), 6.88-7.09 (m, 4H, Ar), 7.18-7.34 (m, 15H, Ar), 7.42 (dd, *J* = 1.5, 7.5 Hz, 1H, Ar), 7.50 (d, *J* = 7.5 Hz, 2H, Ar), 7.65 (d, *J* = 7.5 Hz, 2H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 8.2, 11.2, 25.8, 57.2, 70.3, 77.7, 111.6, 111.9, 112.7, 120.7, 126.6, 127.0, 127.2, 127.4, 127.7, 127.9, 128.12, 128.18, 128.25, 128.28, 128.34, 128.37, 128.42, 128.6, 135.3, 136.5, 136.6, 137.0, 156.9, 209.8. IR (CH_2Cl_2) ν 3080, 3057, 3030, 2973, 2929, 2821, 1597, 1493, 1451, 1284, 1266, 1232, 1186, 1106, 1095, 1026, 1007, 999, 982, 753, 741, 727, 696, 686, 672, 657, 649, 640, 633, 618, 609, 597, 583, 575, 556, 541, 533, 513, 504 cm^{-1}. MS (MALDI) m/z 557 ($M+Na^+$). HRMS (MALDI) calcd. for $C_{39}H_{34}O_2Na^+$: 557.2457, Found: 557.2451.



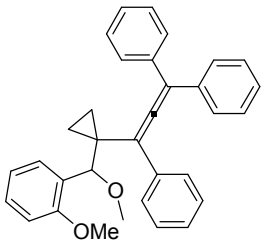






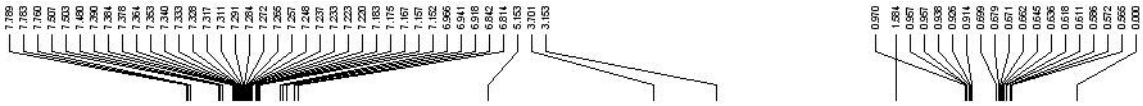


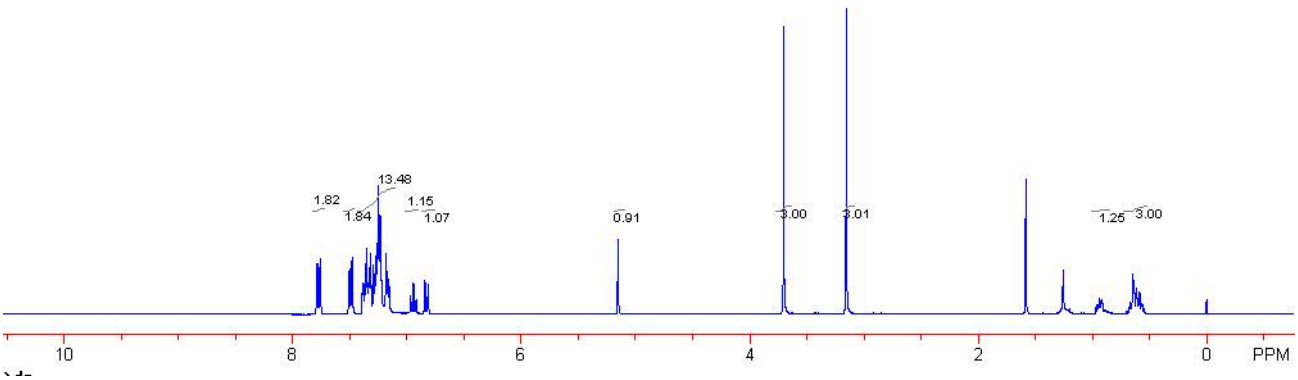
^{1}H NMR DEPT spectrum

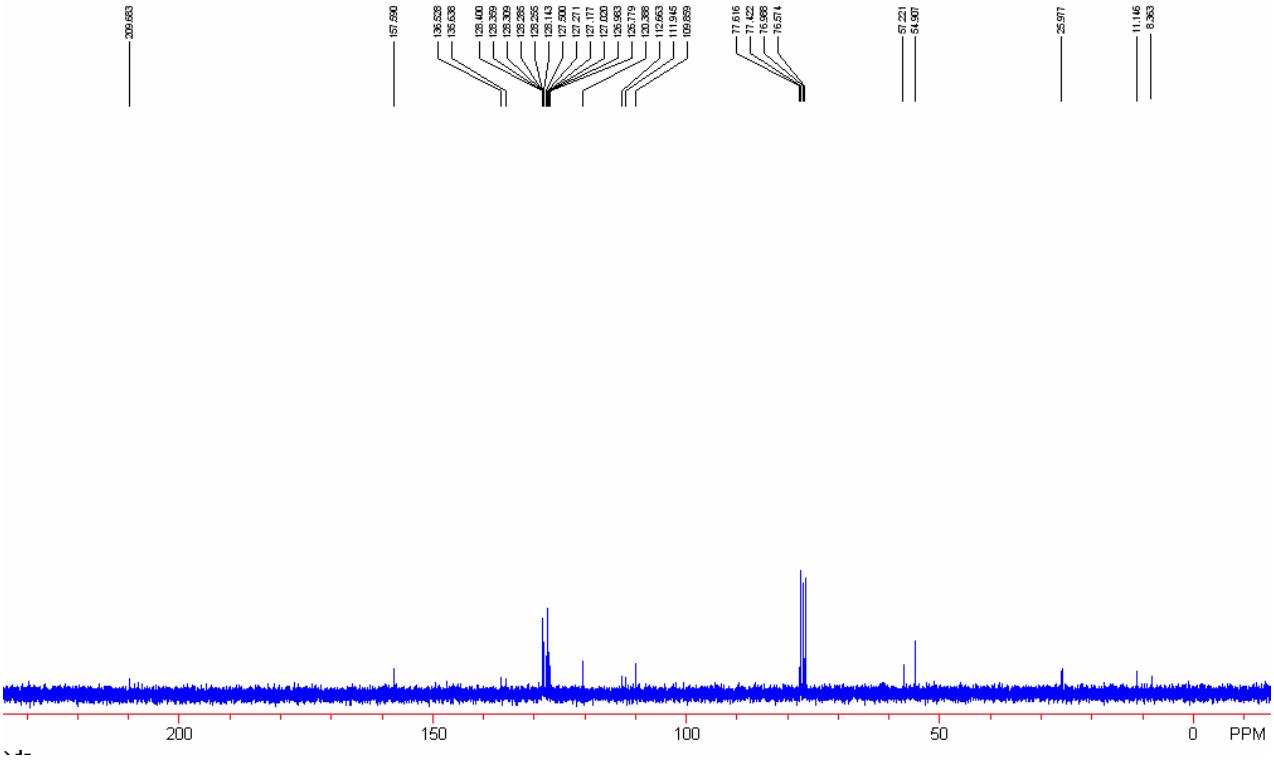


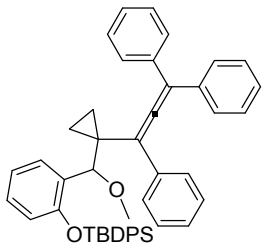
5b

A yellow solid, m.p. 127-129 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 0.56-0.70 (m, 3H, CH_2), 0.91-0.97 (m, 1H, CH_2), 3.15 (s, 3H, OCH_3), 3.70 (s, 3H, OCH_3), 5.15 (s, 1H, CH), 6.83 (d, J = 8.1 Hz, 1H, Ar), 6.94 (t, J = 7.2 Hz, 1H, Ar), 7.15-7.39 (m, 13H, Ar), 7.49 (dd, J = 1.5, 7.2 Hz, 2H, Ar), 7.77 (dd, J = 1.5, 8.1 Hz, 2H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 8.4, 11.1, 26.0, 54.9, 57.2, 77.6, 109.9, 112.0, 112.7, 120.4, 126.8, 127.0, 127.3, 127.5, 128.2, 128.27, 128.30, 128.38, 128.41, 135.7, 136.5, 157.6, 158.0, 209.7. IR (CH_2Cl_2) ν 3089, 3060, 2932, 1925, 1598, 1493, 1464, 1444, 1360, 1278, 1242, 1110, 1099, 1091, 1028, 934, 918, 912, 899, 886, 875, 851, 825, 815, 807, 782, 760, 752, 736, 720, 712, 695, 609, 589, 548, 535, 508 cm^{-1}. MS (MALDI) m/z 481 (M+Na^{+}). HRMS (MALDI) calcd. for $C_{33}H_{30}O_2Na^{+}$: 481.2145, Found: 481.2138.



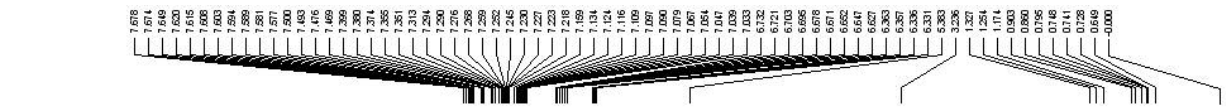


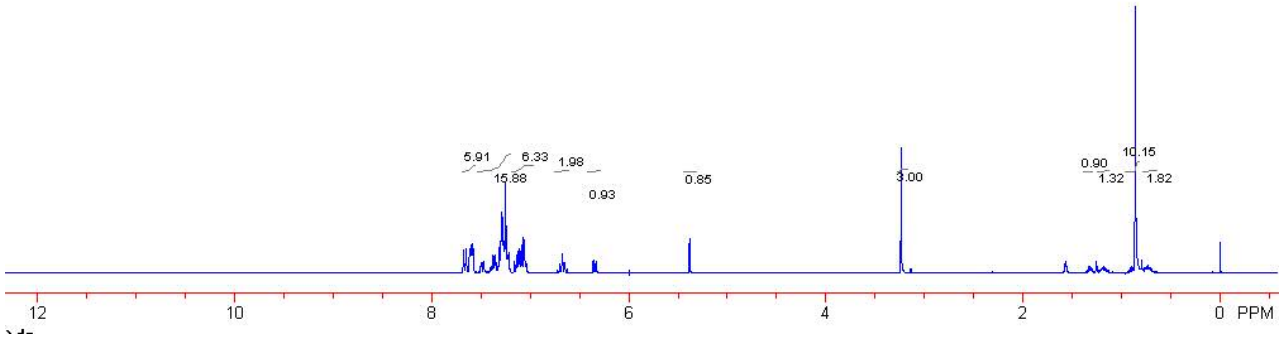




5c

A yellow solid, m.p. 149-151 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 0.65-0.80 (m, 1H, CH_2), 0.86 (s, 9H, CH_3), 0.89-0.95 (m, 1H, CH_2), 1.09-1.21 (m, 1H, CH_2), 1.21-1.36 (m, 1H, CH_2), 3.24 (s, 3H, CH_3), 5.39 (s, 1H, CH), 6.35 (dd, J = 1.5, 8.1 Hz, 1H, Ar), 6.63-6.73 (m, 2H, Ar), 7.04-7.16 (m, 5H, Ar), 7.22-7.32 (m, 11H, Ar), 7.35-7.50 (m, 4H, Ar), 7.58-7.68 (m, 6H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 10.2, 10.3, 19.1, 26.2, 26.3, 56.8, 111.8, 112.4, 119.0, 121.1, 126.8, 126.9, 127.1, 127.3, 127.7, 128.0, 128.2, 128.3, 128.5, 129.1, 129.4, 129.8, 132.2, 132.4, 135.2, 135.3, 135.8, 136.2, 136.5, 153.9, 210.0. IR (CH_2Cl_2) ν 3072, 3056, 3029, 2930, 2894, 2858, 2818, 1940, 1598, 1582, 1489, 1471, 1462, 1447, 1428, 1361, 1346, 1279, 1235, 1184, 1106, 1091, 1030, 916, 823, 761, 742, 696, 616, 598, 513 cm^{-1}. MS (MALDI) m/z 705 ($M+Na^+$). HRMS (MALDI) calcd. for $C_{48}H_{46}SiO_2Na^+$: 705.3143, Found: 705.3159.

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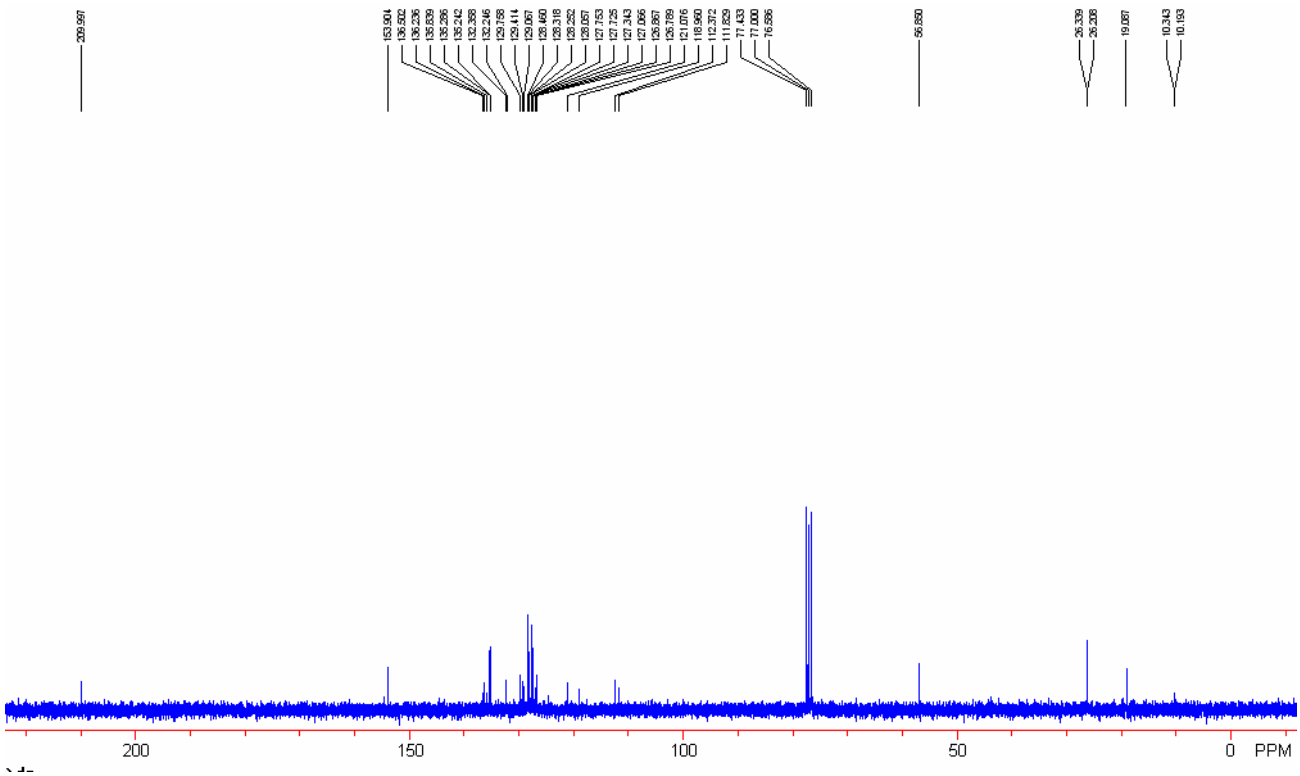
0 PPM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150
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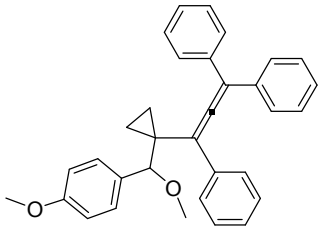
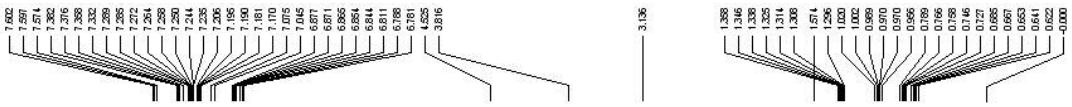
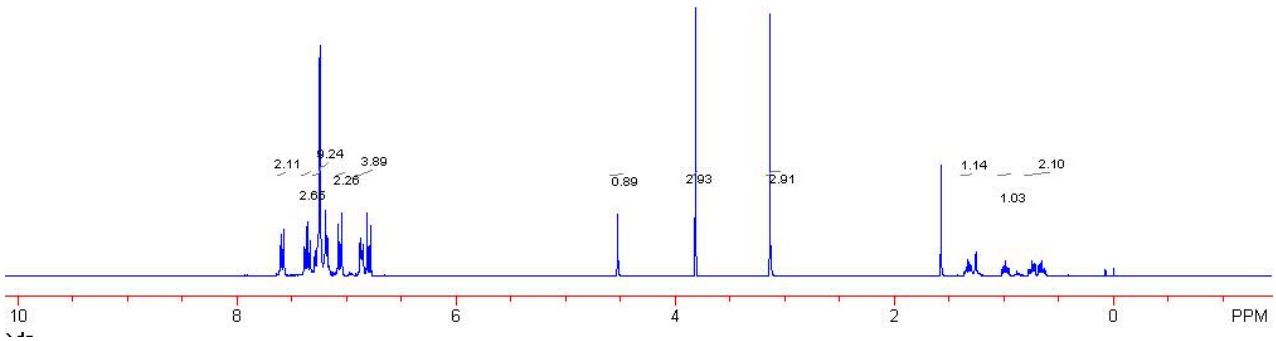
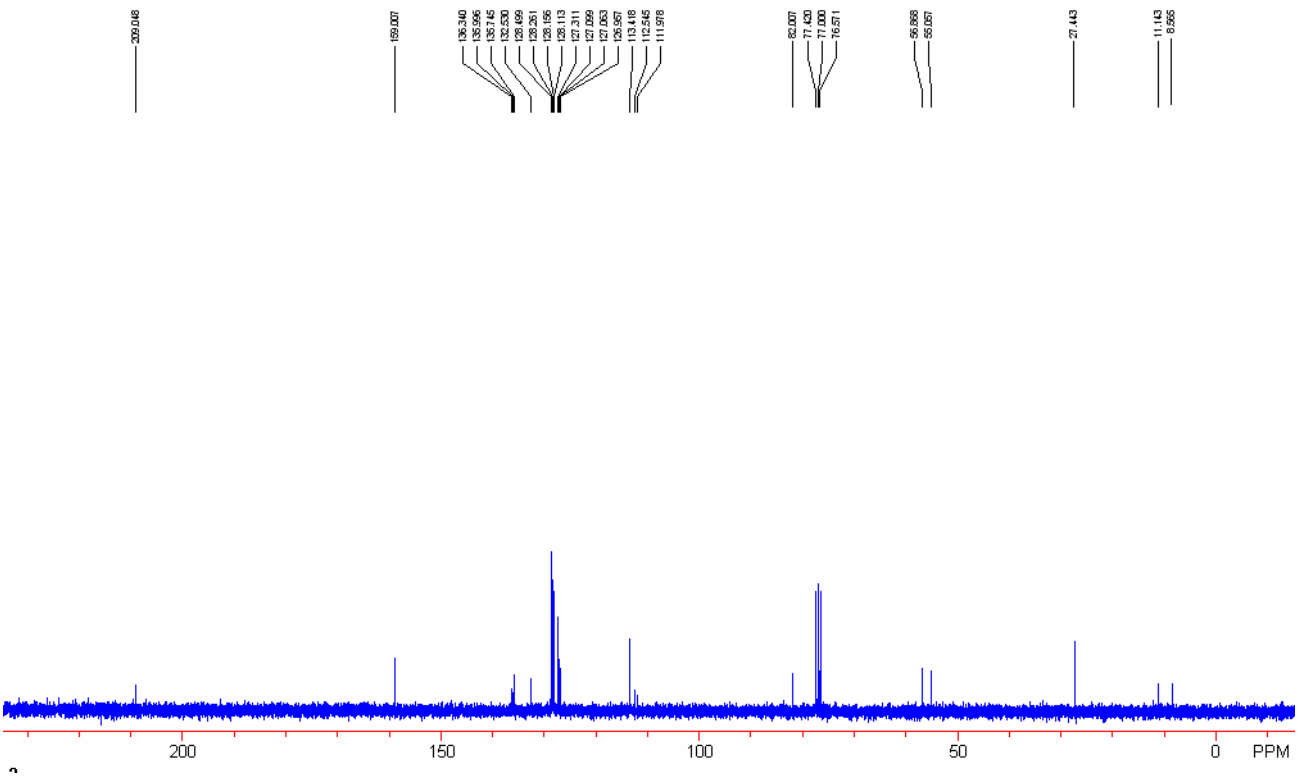


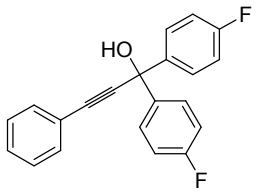
5d

A yellow solid, m.p. 165-167 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 0.64-0.68 (1H, m, CH_2), 0.72-0.75 (m, 1H, CH_2), 0.96-1.01 (m, 1H, CH_2), 1.30-1.34 (m, 1H, CH_2), 3.14 (s, 3H, OCH_3), 3.82 (s, 3H, OCH_3), 4.53 (s, 1H, CH), 6.79 (d, *J* = 8.7 Hz, 2H, Ar), 6.84-6.88 (m, 2H, Ar), 7.06 (d, *J* = 8.7 Hz, 2H, Ar), 7.15-7.29 (m, 9H, Ar), 7.36 (t, *J* = 7.5 Hz, 2H, Ar), 7.59 (dd, *J* = 1.5, 8.4 Hz, 2H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 8.6, 11.1, 27.4, 55.0, 56.9, 82.0, 112.0, 112.5, 113.4, 127.0, 127.07, 127.10, 127.3, 128.1, 128.2, 128.3, 128.5, 132.5, 135.7, 136.0, 136.3, 159.0, 209.1. IR (CH_2Cl_2) ν 3077, 3051, 3038, 3013, 2910, 1510, 1489, 1251, 1244, 1097, 1084, 1050, 1039, 917, 822, 813, 760, 748, 697, 689, 673, 661, 648, 638, 623, 609, 599, 588, 581, 572, 563, 552, 540, 531, 524 cm^{-1}. MS (MALDI) m/z 481 (M+Na^{+}). HRMS (MALDI) calcd. for $C_{33}H_{30}O_2Na^{+}$: 481.2148, Found: 481.2138.





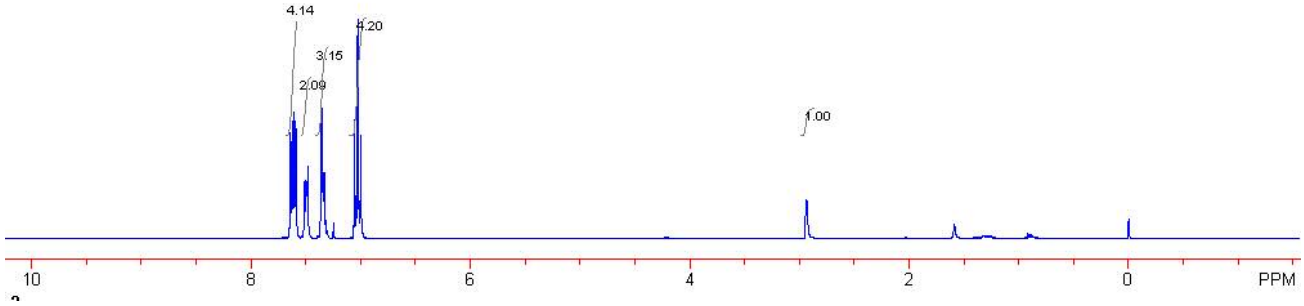


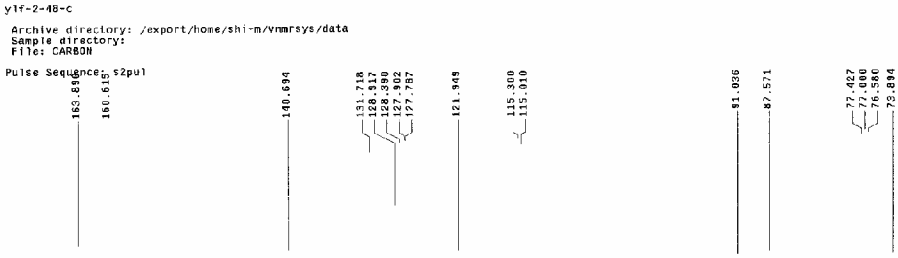


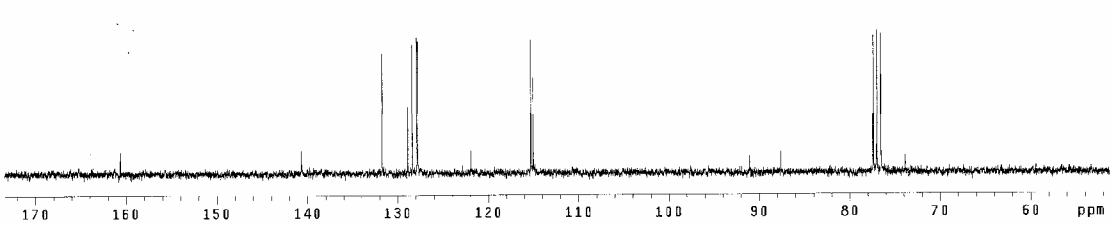
2c-OH

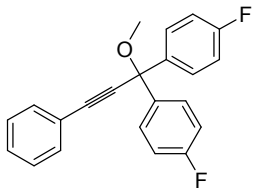
A white solid, m.p. 100-102 °C. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.94 (s, 1H, OH), 6.99-7.06 (m, 4H, Ar), 7.32-7.36 (m, 3H, Ar), 7.48-7.51 (m, 2H, Ar), 7.58-7.65 (m, 4H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 73.9, 87.6, 91.0, 115.2 (d, J = 21.8 Hz), 122.0, 127.8 (d, J = 8.6 Hz), 128.4, 128.9, 131.7, 140.7, 162.3 (d, J = 245.6 Hz). IR (CH_2Cl_2) ν 3566, 3430, 3037, 1602, 1500, 1443, 1412, 1330, 1230, 1158, 1054, 1015, 988, 940, 902, 836, 803, 777, 756, 691, 582, 556, 517 cm^{-1}. MS (%) m/z 320 (M^+, 32), 123 (100), 95 (52), 224 (47), 242 (32), 196 (32), 75 (25), 225 (23). HRMS (EI) calcd. for $C_{21}H_{14}OF_2$: 320.1013, Found: 320.1016.


7.636
7.630
7.619
7.613
7.608
7.596
7.590
7.581
7.512
7.506
7.494
7.489
7.481
7.362
7.354
7.347
7.335
7.329
7.316
7.245
7.064
7.048
7.025
7.002
6.996
6.986
2.934
1.588
0.000


4.14
4.20
3.15
2.09
1.00
10
8
6
4
2
0
PPM

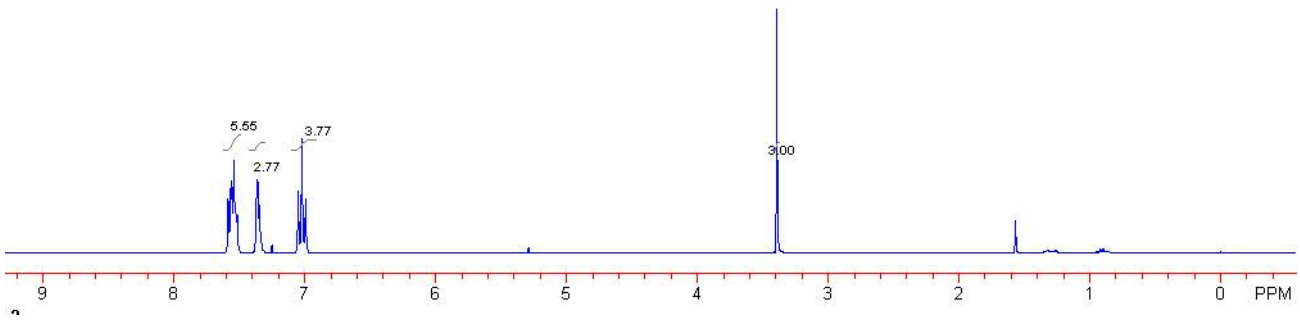

y1f-2-48-c
Archive directory: /export/home/shi-m/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
163.896
160.615
140.694
131.718
128.517
128.390
127.902
127.787
121.949
115.300
115.010
91.036
87.571
77.427
77.000
76.580
73.804

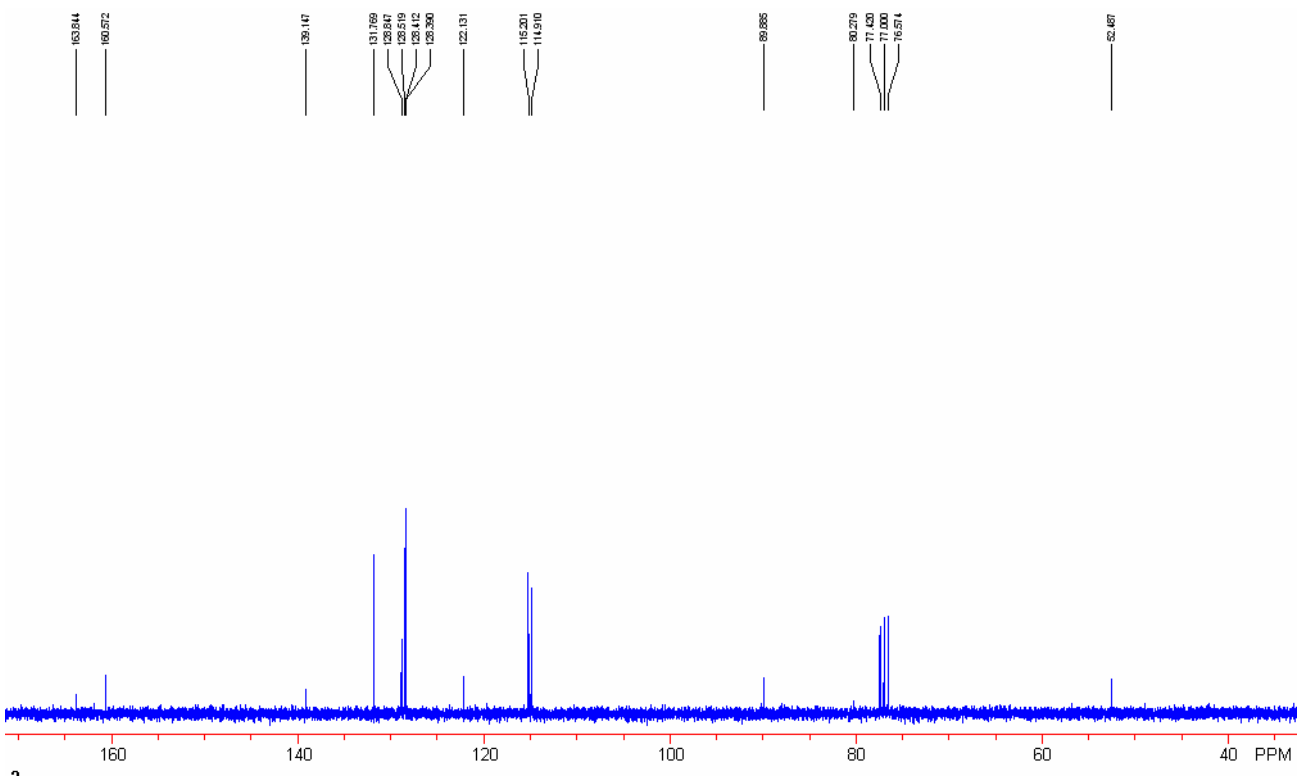

170
160
150
140
130
120
110
100
90
80
70
60
ppm

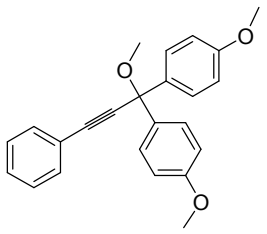


2c

A white solid, m.p. 68-70 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 3.39 (s, 3H, OCH_3), 7.02 (t, J = 8.7 Hz, 4H, Ar), 7.35-7.36 (m, 3H, Ar), 7.52-7.59 (m, 6H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 52.5, 80.3, 88.0, 89.9, 115.1 (d, J = 21.8 Hz), 122.1, 128.4, 128.5 (d, J = 8.0 Hz), 128.9, 131.8, 139.2, 162.2 (d, J = 245.6 Hz). IR (CH_2Cl_2) ν 3065, 2991, 2936, 2902, 2826, 2223, 1602, 1505, 1465, 1444, 1409, 1299, 1258, 1228, 1173, 1157, 1077, 1042, 1015, 957, 945, 835, 781, 756, 691, 594, 579, 553 cm^{-1}. MS (%) m/z 334 (M^+, 41), 303 (100), 319 (75), 291 (40), 239 (37), 123 (37), 301 (33), 304 (26). Anal. calcd. for $C_{22}H_{16}OF_2$: C, 79.03%; H, 4.82%; Found: C, 78.91%; H, 4.87%.

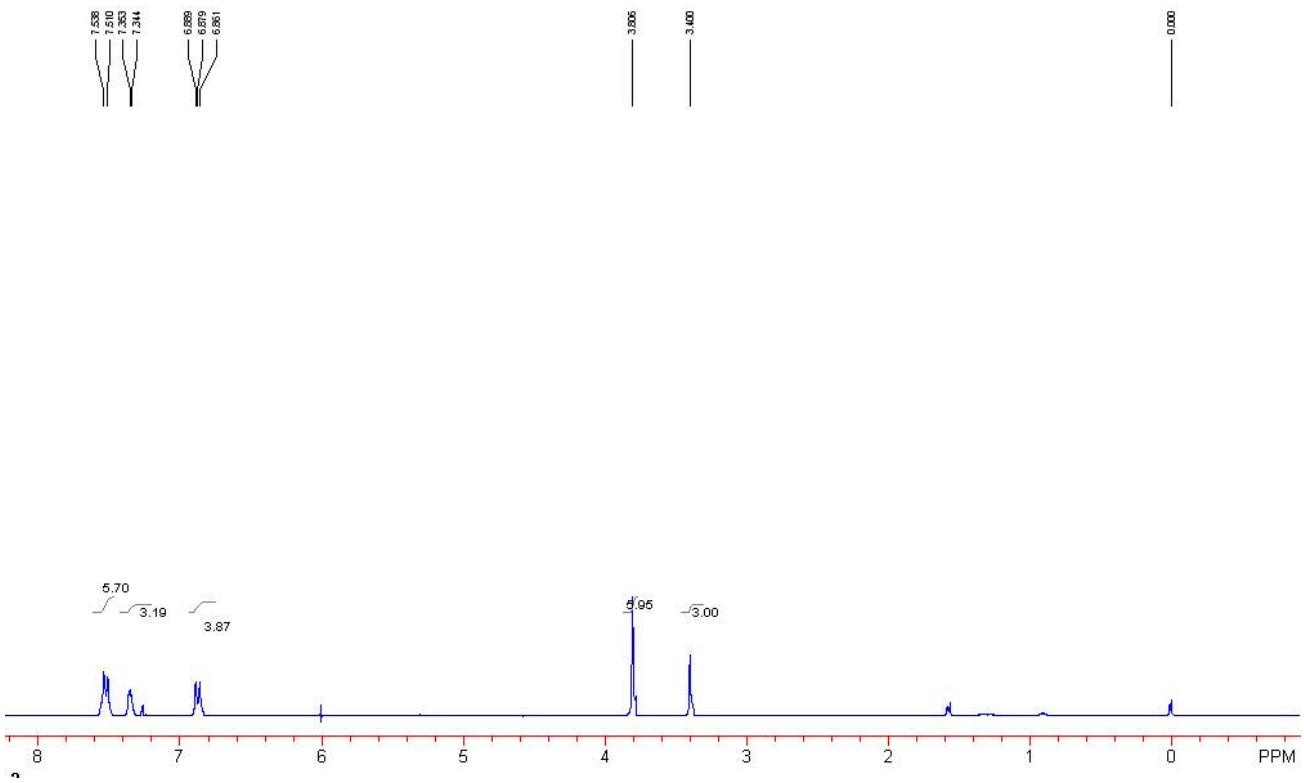






2b

A yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 3.40 (s, 3H, OCH_3), 3.81 (s, 6H, OCH_3), 6.86-6.89 (m, 4H, Ar), 7.34-7.35 (m, 3H, Ar), 7.51-7.54 (m, 6H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 52.4, 55.2, 80.5, 88.9, 89.1, 113.4, 122.6, 128.0, 128.3, 128.5, 131.7, 135.8, 158.9. IR (CH_2Cl_2) ν 3060, 3035, 2998, 2934, 2903, 2835, 2222, 1878, 1607, 1585, 1508, 1491, 1463, 1442, 1415, 1303, 1249, 1170, 1074, 1035, 942, 918, 831, 807, 777, 757, 691, 600, 586, 557, 526 cm^{-1}. MS (%) m/z 358 (M^+, 32), 327 (100), 343 (79), 328 (30), 251 (29), 344 (25), 315 (21), 135 (20). HRMS (EI) calcd. for $C_{24}H_{22}O_3$: 358.1569, Found: 358.1573.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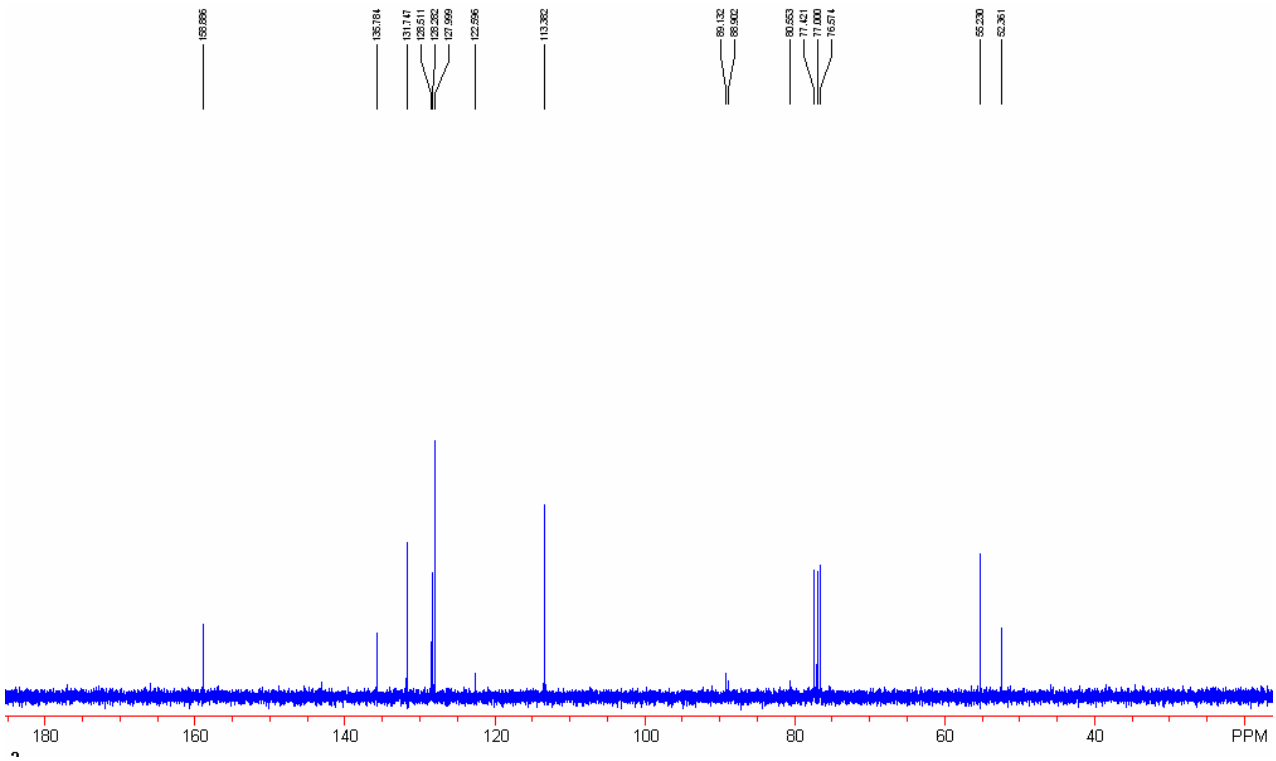
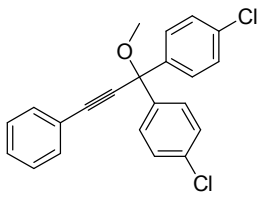
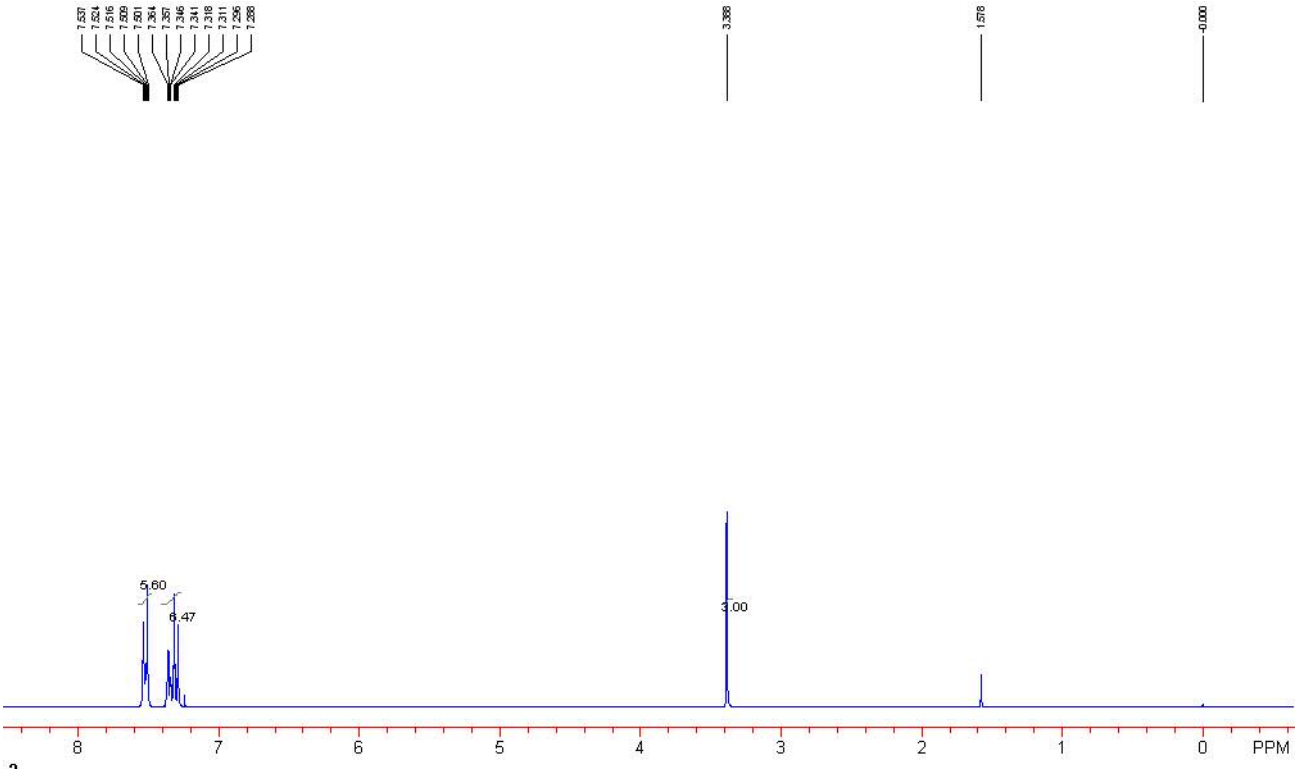
7.510
7.353
7.344
6.889
6.879
6.861
3.806
3.400
0.000
5.70
3.19
3.87
5.95
3.00
8
7
6
5
4
3
2
1
0
PPM

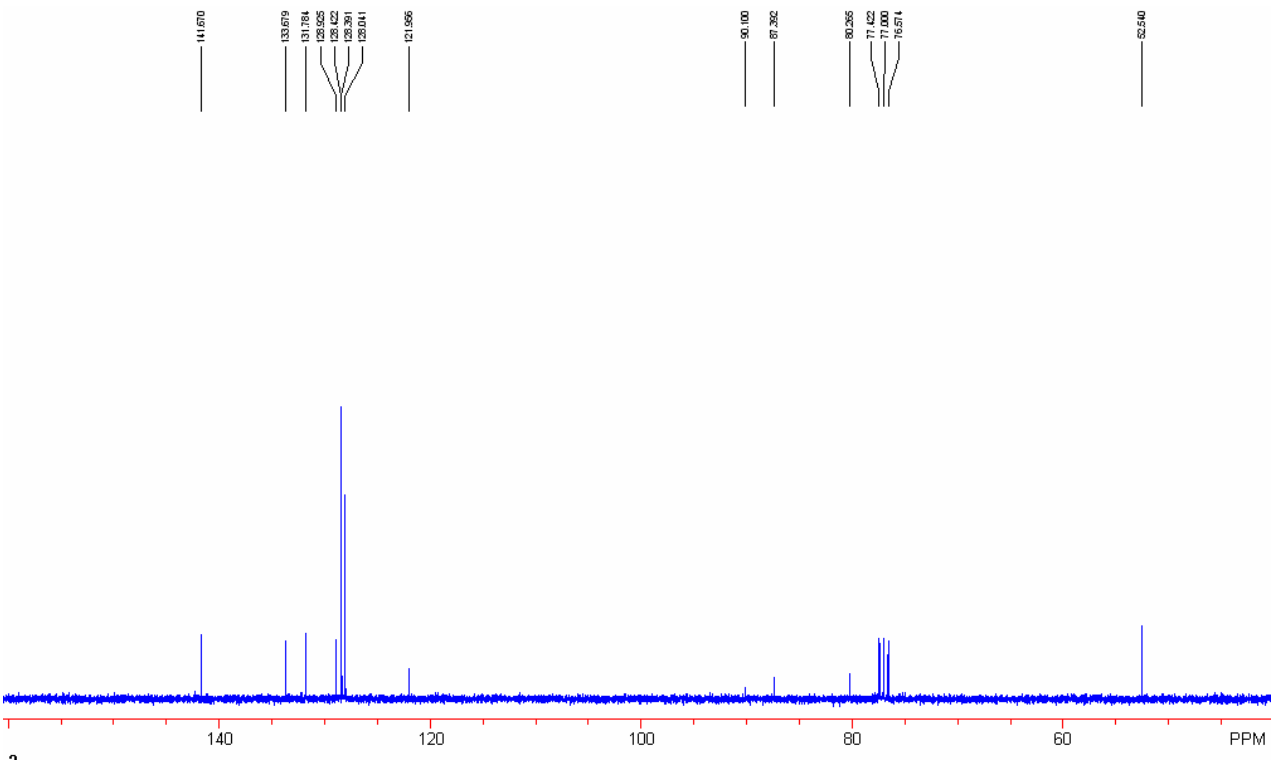

159.886
135.784
131.747
129.511
129.282
127.999
122.996
113.382
89.132
88.902
80.553
77.421
77.000
76.574
55.230
52.361
180
160
140
120
100
80
60
40
PPM

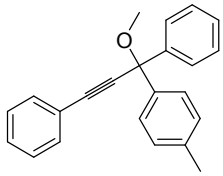

Cl
O
Cl

2f

A white solid, m.p. 91-93 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 3.39 (s, 3H, OCH_3), 7.29-7.36 (m, 7H, Ar), 7.51-7.54 (m, 6H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 52.5, 80.3, 87.4, 90.1, 122.0, 128.1, 128.41, 128.43, 128.9, 131.8, 133.7, 141.7. IR (CH_2Cl_2) ν 3057, 3033, 2989, 2934, 2901, 2825, 2223, 1592, 1578, 1487, 1443, 1401, 1262, 1216, 1172, 1092, 1078, 1040, 1015, 958, 946, 919, 835, 820, 787, 756, 690, 614, 555 cm^{-1}. MS (%) m/z 366 (M^+, 19), 331 (100), 335 (69), 255 (46), 337 (45), 333 (40), 265 (40), 132 (38), 351 (35). Anal. calcd. for $C_{22}H_{16}OCl_2$: C, 71.95%; H, 4.39%; Found: C, 72.04%; H, 4.44%.

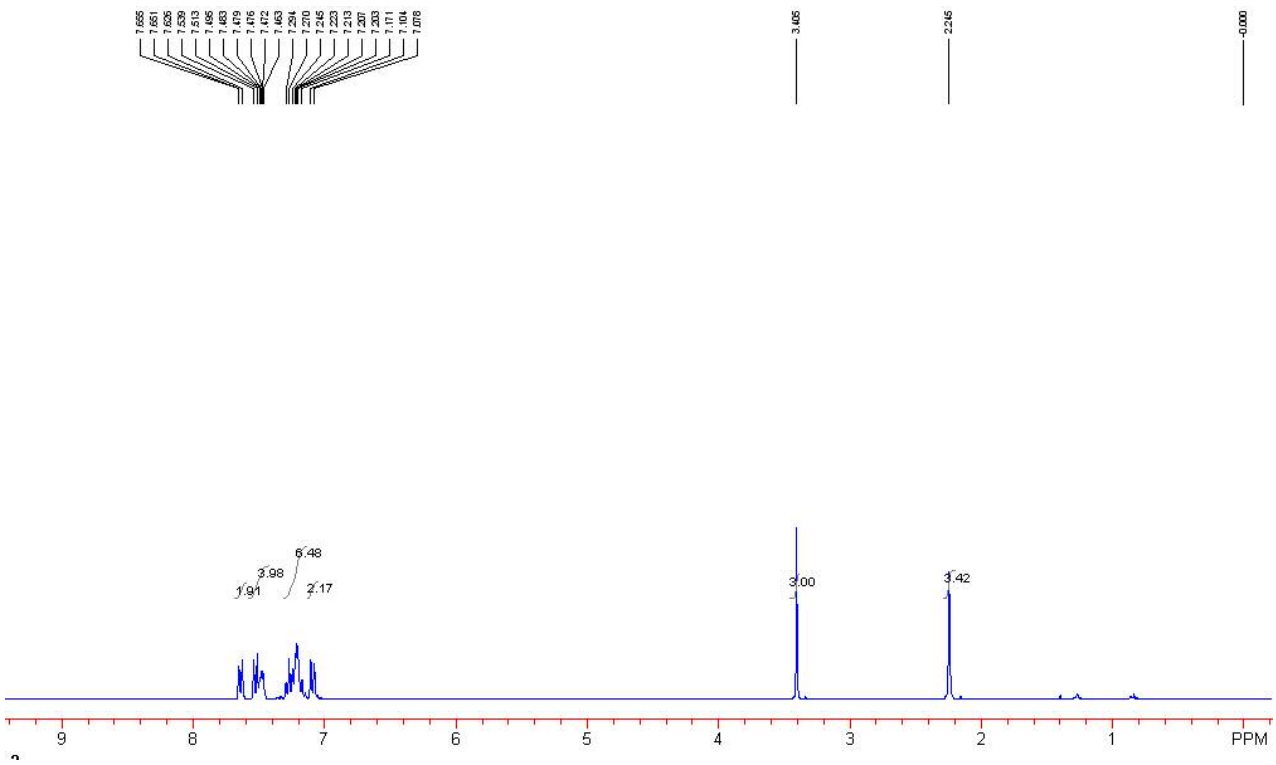


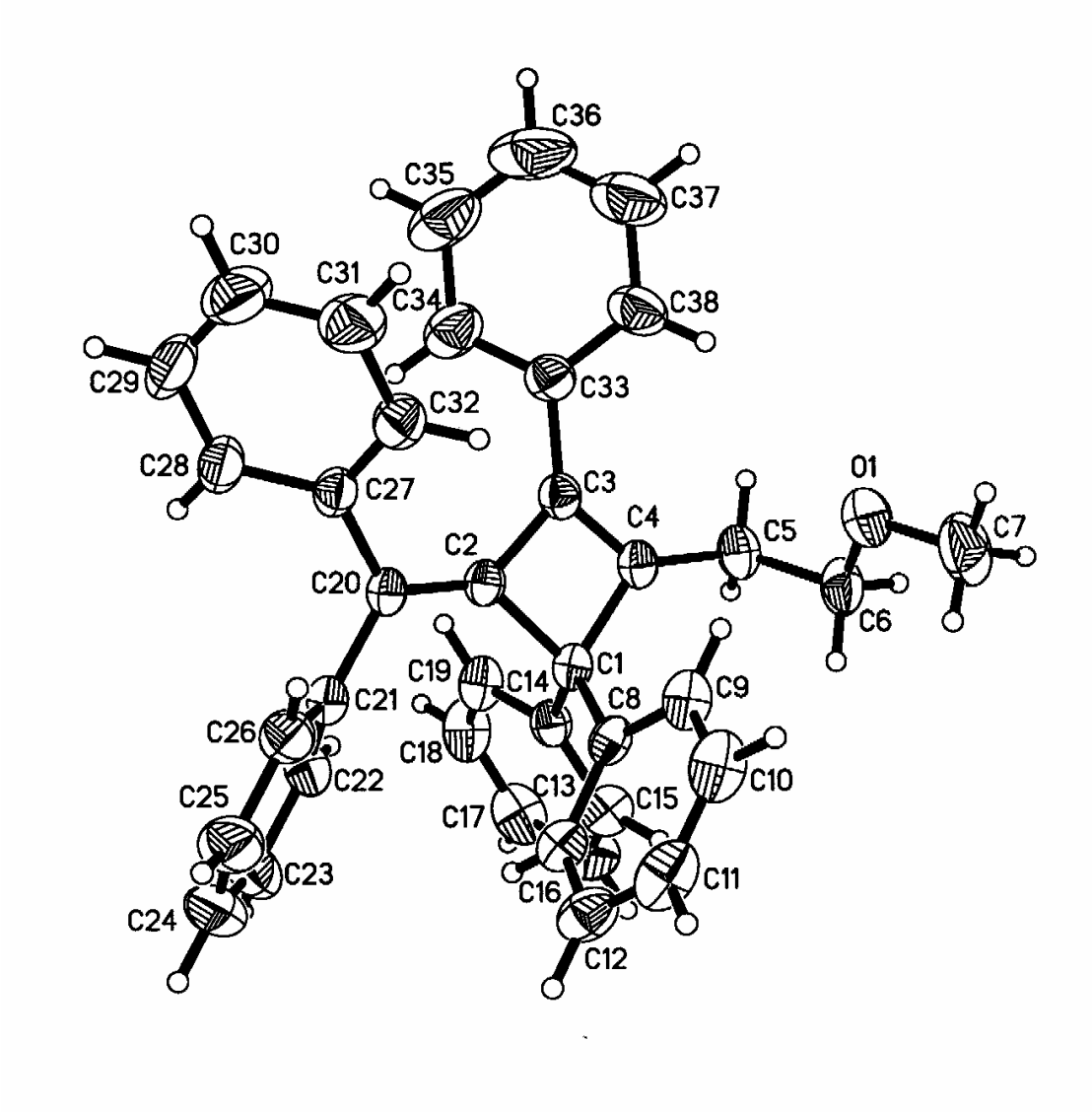




2e

A yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.25 (s, 3H, CH_3), 3.41 (s, 3H, CH_3), 7.09 (d, *J* = 7.8 Hz, 2H, Ar), 7.15-7.30 (m, 6H, Ar), 7.46-7.63 (m, 4H, Ar), 7.65 (dd, *J* = 0.9, 7.8 Hz, 2H, Ar). ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 20.9, 52.3, 81.0, 88.7, 89.3, 122.4, 126.52, 126.56, 127.4, 127.6, 128.0, 128.2, 128.4, 128.7, 131.6, 137.1, 140.4, 143.6. IR (CH_2Cl_2) ν 3058, 3026, 2986, 2934, 2901, 2823, 2212, 1598, 1509, 1489, 1447, 1260, 1172, 1074, 1043, 1030, 1022, 945, 818, 756, 691, 608, 592, 582, 553, 533, 523, 508 cm^{-1}. MS (%) m/z 312 (M^+, 10), 211 (100), 105 (25), 119 (19), 91 (18), 212 (17), 281 (16), 77 (15), 297 (15). HRMS (EI) calcd. for $C_{23}H_{20}O$: 312.1538, Found: 312.1514.


7.655
7.651
7.626
7.539
7.513
7.495
7.479
7.476
7.472
7.463
7.394
7.370
7.245
7.233
7.213
7.207
7.200
7.111
7.104
7.078
3.405
2.245
-0.000
6.48
3.98
1.91
2.17
3.00
3.42
9
8
7
6
5
4
3
2
1
PPM
143.594
140.436
137.067
129.146
128.424
131.619
128.774
129.013
127.647
127.391
126.785
126.677
126.555
126.516
122.394
89.254
88.698
80.998
77.425
77.000
76.573
52.284
20.909
160
140
120
100
80
60
40
20
PPM



The crystal data of **3a** have been deposited in CCDC with number 644870. Empirical Formula: $C_{38}H_{32}O$; Formula Weight: 504.64; Crystal Color, Habit: colorless, prismatic; Crystal System: Triclinic; Lattice Type: Primitive; Lattice Parameters: a = 10.9684(13)Å, b = 11.1701(14)Å, c = 12.9433(15)Å, $\alpha = 87.150(2)^{o}$, $\beta = 67.964(2)^{o}$, $\gamma = 81.135(2)^{o}$, V = 1452.3(3)Å^3; Space group: P-1; Z = 2; D_{calc} = 1.154 g/cm^3; F_{000} = 536; Diffractometer: Rigaku AFC7R; Residuals: R; Rw: 0.0524, 0.1271.

Table 1. Crystal data and structure refinement for cd27199.

Identification code	cd27199
Empirical formula	C38 H32 O
Formula weight	504.64
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 10.9684(13) A alpha = 87.150(2) deg. b = 11.1701(14) A beta = 67.964(2) deg. c = 12.9433(15) A gamma = 81.135(2) deg.
Volume	1452.3(3) A^3
Z, Calculated density	2, 1.154 Mg/m^3
Absorption coefficient	0.067 mm^-1
F(000)	536
Crystal size	0.489 x 0.367 x 0.201 mm
Theta range for data collection	1.85 to 26.00 deg.
Limiting indices	-13<=h<=13, -13<=k<=13, -15<=l<=13
Reflections collected / unique	8001 / 5603 [R(int) = 0.0943]
Completeness to theta = 26.00	97.9 %
Absorption correction	Empirical
Max. and min. transmission	1.0000 and 0.7736
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5603 / 0 / 353
Goodness-of-fit on F^2	0.915
Final R indices [I>2sigma(I)]	R1 = 0.0524, wR2 = 0.1271
R indices (all data)	R1 = 0.0856, wR2 = 0.1437
Largest diff. peak and hole	0.137 and -0.153 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for cd27199.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	-995(2)	1732(1)	-151(1)	83(1)
C(1)	-1226(2)	3279(1)	2704(1)	49(1)
C(2)	231(2)	3022(1)	2661(1)	49(1)
C(3)	704(2)	3119(2)	1443(1)	52(1)
C(4)	-529(2)	3310(2)	1430(1)	53(1)
C(5)	-1094(2)	3677(2)	553(2)	66(1)
C(6)	-1868(2)	2799(2)	320(2)	73(1)
C(7)	-1648(3)	813(2)	-324(2)	108(1)
C(8)	-1922(2)	2185(2)	3206(2)	50(1)
C(9)	-1661(2)	1146(2)	2574(2)	62(1)
C(10)	-2170(2)	106(2)	3053(2)	79(1)
C(11)	-2917(2)	80(2)	4158(2)	83(1)
C(12)	-3189(2)	1094(2)	4802(2)	74(1)
C(13)	-2690(2)	2143(2)	4323(2)	61(1)
C(14)	-2024(2)	4504(2)	3121(1)	52(1)
C(15)	-3387(2)	4714(2)	3422(2)	67(1)
C(16)	-4105(2)	5843(2)	3762(2)	82(1)
C(17)	-3468(3)	6786(2)	3807(2)	88(1)
C(18)	-2119(3)	6617(2)	3480(2)	82(1)
C(19)	-1401(2)	5490(2)	3133(2)	65(1)
C(20)	774(2)	2650(1)	3421(1)	49(1)
C(21)	-7(2)	2665(2)	4629(1)	53(1)
C(22)	-836(2)	3685(2)	5186(2)	68(1)
C(23)	-1573(2)	3672(3)	6311(2)	91(1)
C(24)	-1489(3)	2633(3)	6903(2)	99(1)
C(25)	-660(2)	1617(2)	6375(2)	90(1)
C(26)	85(2)	1627(2)	5254(2)	68(1)
C(27)	2189(2)	2097(2)	3060(2)	55(1)
C(28)	3033(2)	2456(2)	3521(2)	74(1)
C(29)	4350(2)	1899(3)	3174(2)	97(1)
C(30)	4810(2)	984(3)	2396(3)	111(1)
C(31)	3988(2)	621(2)	1952(2)	97(1)
C(32)	2701(2)	1169(2)	2278(2)	70(1)
C(33)	2028(2)	3080(2)	566(2)	62(1)
C(34)	3006(2)	3603(2)	733(2)	87(1)
C(35)	4260(3)	3518(3)	-89(3)	124(1)
C(36)	4529(3)	2939(4)	-1080(3)	143(1)
C(37)	3569(3)	2423(3)	-1238(2)	122(1)
C(38)	2334(2)	2498(2)	-432(2)	82(1)

Table 3. Bond lengths [A] and angles [deg] for cd27199.

O(1)-C(7)	1.409(2)
O(1)-C(6)	1.411(2)
C(1)-C(14)	1.510(2)
C(1)-C(8)	1.528(2)
C(1)-C(4)	1.536(2)
C(1)-C(2)	1.560(2)
C(2)-C(20)	1.349(2)
C(2)-C(3)	1.467(2)
C(3)-C(4)	1.342(2)
C(3)-C(33)	1.465(2)
C(4)-C(5)	1.498(2)
C(5)-C(6)	1.500(2)
C(5)-H(5A)	0.9700
C(5)-H(5B)	0.9700
C(6)-H(6A)	0.9700
C(6)-H(6B)	0.9700
C(7)-H(7A)	0.9600
C(7)-H(7B)	0.9600
C(7)-H(7C)	0.9600
C(8)-C(13)	1.376(2)
C(8)-C(9)	1.384(2)
C(9)-C(10)	1.384(3)
C(9)-H(9)	0.9300
C(10)-C(11)	1.357(3)
C(10)-H(10)	0.9300
C(11)-C(12)	1.368(3)
C(11)-H(11)	0.9300
C(12)-C(13)	1.388(3)
C(12)-H(12)	0.9300
C(13)-H(13)	0.9300
C(14)-C(15)	1.381(2)
C(14)-C(19)	1.387(2)
C(15)-C(16)	1.376(3)
C(15)-H(15)	0.9300
C(16)-C(17)	1.365(3)
C(16)-H(16)	0.9300
C(17)-C(18)	1.363(3)
C(17)-H(17)	0.9300
C(18)-C(19)	1.375(3)
C(18)-H(18)	0.9300
C(19)-H(19)	0.9300
C(20)-C(21)	1.474(2)
C(20)-C(27)	1.483(2)
C(21)-C(22)	1.383(2)
C(21)-C(26)	1.393(2)
C(22)-C(23)	1.376(3)
C(22)-H(22)	0.9300
C(23)-C(24)	1.368(3)
C(23)-H(23)	0.9300
C(24)-C(25)	1.370(3)
C(24)-H(24)	0.9300
C(25)-C(26)	1.374(3)
C(25)-H(25)	0.9300
C(26)-H(26)	0.9300
C(27)-C(32)	1.382(2)
C(27)-C(28)	1.387(2)
C(28)-C(29)	1.393(3)
C(28)-H(28)	0.9300
C(29)-C(30)	1.370(3)
C(29)-H(29)	0.9300
C(30)-C(31)	1.353(3)
C(30)-H(30)	0.9300
C(31)-C(32)	1.363(3)
C(31)-H(31)	0.9300
C(32)-H(32)	0.9300
C(33)-C(38)	1.377(3)
C(33)-C(34)	1.385(3)
C(34)-C(35)	1.380(3)

C(34)-H(34)	0.9300
C(35)-C(36)	1.376(4)
C(35)-H(35)	0.9300
C(36)-C(37)	1.357(4)
C(36)-H(36)	0.9300
C(37)-C(38)	1.357(3)
C(37)-H(37)	0.9300
C(38)-H(38)	0.9300

C(7)-O(1)-C(6)	113.16(18)
C(14)-C(1)-C(8)	115.87(14)
C(14)-C(1)-C(4)	109.42(13)
C(8)-C(1)-C(4)	117.55(13)
C(14)-C(1)-C(2)	118.29(13)
C(8)-C(1)-C(2)	108.88(13)
C(4)-C(1)-C(2)	82.83(11)
C(20)-C(2)-C(3)	136.40(16)
C(20)-C(2)-C(1)	133.68(15)
C(3)-C(2)-C(1)	88.91(12)
C(4)-C(3)-C(33)	133.08(16)
C(4)-C(3)-C(2)	93.51(14)
C(33)-C(3)-C(2)	133.35(15)
C(3)-C(4)-C(5)	134.35(17)
C(3)-C(4)-C(1)	94.70(13)
C(5)-C(4)-C(1)	129.94(16)
C(4)-C(5)-C(6)	116.04(16)
C(4)-C(5)-H(5A)	108.3
C(6)-C(5)-H(5A)	108.3
C(4)-C(5)-H(5B)	108.3
C(6)-C(5)-H(5B)	108.3
H(5A)-C(5)-H(5B)	107.4
O(1)-C(6)-C(5)	108.90(16)
O(1)-C(6)-H(6A)	109.9
C(5)-C(6)-H(6A)	109.9
O(1)-C(6)-H(6B)	109.9
C(5)-C(6)-H(6B)	109.9
H(6A)-C(6)-H(6B)	108.3
O(1)-C(7)-H(7A)	109.5
O(1)-C(7)-H(7B)	109.5
H(7A)-C(7)-H(7B)	109.5
O(1)-C(7)-H(7C)	109.5
H(7A)-C(7)-H(7C)	109.5
H(7B)-C(7)-H(7C)	109.5
C(13)-C(8)-C(9)	117.84(17)
C(13)-C(8)-C(1)	121.39(15)
C(9)-C(8)-C(1)	120.30(17)
C(10)-C(9)-C(8)	120.8(2)
C(10)-C(9)-H(9)	119.6
C(8)-C(9)-H(9)	119.6
C(11)-C(10)-C(9)	120.5(2)
C(11)-C(10)-H(10)	119.7
C(9)-C(10)-H(10)	119.7
C(10)-C(11)-C(12)	119.9(2)
C(10)-C(11)-H(11)	120.1
C(12)-C(11)-H(11)	120.1
C(11)-C(12)-C(13)	119.8(2)
C(11)-C(12)-H(12)	120.1
C(13)-C(12)-H(12)	120.1
C(8)-C(13)-C(12)	121.16(19)
C(8)-C(13)-H(13)	119.4
C(12)-C(13)-H(13)	119.4
C(15)-C(14)-C(19)	117.16(17)
C(15)-C(14)-C(1)	121.68(15)
C(19)-C(14)-C(1)	120.97(16)
C(16)-C(15)-C(14)	121.52(18)
C(16)-C(15)-H(15)	119.2
C(14)-C(15)-H(15)	119.2
C(17)-C(16)-C(15)	119.9(2)
C(17)-C(16)-H(16)	120.1
C(15)-C(16)-H(16)	120.1
C(18)-C(17)-C(16)	120.0(2)
C(18)-C(17)-H(17)	120.0

C(16)-C(17)-H(17)	120.0
C(17)-C(18)-C(19)	120.1(2)
C(17)-C(18)-H(18)	120.0
C(19)-C(18)-H(18)	120.0
C(18)-C(19)-C(14)	121.3(2)
C(18)-C(19)-H(19)	119.4
C(14)-C(19)-H(19)	119.4
C(2)-C(20)-C(21)	122.88(15)
C(2)-C(20)-C(27)	120.45(15)
C(21)-C(20)-C(27)	116.45(14)
C(22)-C(21)-C(26)	117.64(18)
C(22)-C(21)-C(20)	122.56(17)
C(26)-C(21)-C(20)	119.80(17)
C(23)-C(22)-C(21)	121.5(2)
C(23)-C(22)-H(22)	119.2
C(21)-C(22)-H(22)	119.2
C(24)-C(23)-C(22)	119.8(2)
C(24)-C(23)-H(23)	120.1
C(22)-C(23)-H(23)	120.1
C(23)-C(24)-C(25)	119.9(2)
C(23)-C(24)-H(24)	120.1
C(25)-C(24)-H(24)	120.1
C(24)-C(25)-C(26)	120.6(2)
C(24)-C(25)-H(25)	119.7
C(26)-C(25)-H(25)	119.7
C(25)-C(26)-C(21)	120.6(2)
C(25)-C(26)-H(26)	119.7
C(21)-C(26)-H(26)	119.7
C(32)-C(27)-C(28)	117.63(18)
C(32)-C(27)-C(20)	120.81(15)
C(28)-C(27)-C(20)	121.52(18)
C(27)-C(28)-C(29)	119.8(2)
C(27)-C(28)-H(28)	120.1
C(29)-C(28)-H(28)	120.1
C(30)-C(29)-C(28)	120.3(2)
C(30)-C(29)-H(29)	119.8
C(28)-C(29)-H(29)	119.8
C(31)-C(30)-C(29)	120.1(2)
C(31)-C(30)-H(30)	120.0
C(29)-C(30)-H(30)	120.0
C(30)-C(31)-C(32)	120.0(2)
C(30)-C(31)-H(31)	120.0
C(32)-C(31)-H(31)	120.0
C(31)-C(32)-C(27)	122.1(2)
C(31)-C(32)-H(32)	118.9
C(27)-C(32)-H(32)	118.9
C(38)-C(33)-C(34)	118.6(2)
C(38)-C(33)-C(3)	120.62(18)
C(34)-C(33)-C(3)	120.79(19)
C(35)-C(34)-C(33)	119.9(3)
C(35)-C(34)-H(34)	120.1
C(33)-C(34)-H(34)	120.1
C(36)-C(35)-C(34)	120.0(3)
C(36)-C(35)-H(35)	120.0
C(34)-C(35)-H(35)	120.0
C(37)-C(36)-C(35)	119.9(3)
C(37)-C(36)-H(36)	120.1
C(35)-C(36)-H(36)	120.1
C(38)-C(37)-C(36)	120.4(3)
C(38)-C(37)-H(37)	119.8
C(36)-C(37)-H(37)	119.8
C(37)-C(38)-C(33)	121.2(3)
C(37)-C(38)-H(38)	119.4
C(33)-C(38)-H(38)	119.4

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for cd27199.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	94(1)	83(1)	78(1)	-17(1)	-35(1)	-16(1)
C(1)	52(1)	46(1)	54(1)	-1(1)	-26(1)	-9(1)
C(2)	53(1)	45(1)	53(1)	-2(1)	-24(1)	-11(1)
C(3)	58(1)	51(1)	52(1)	0(1)	-23(1)	-12(1)
C(4)	63(1)	48(1)	55(1)	0(1)	-30(1)	-12(1)
C(5)	83(1)	64(1)	65(1)	4(1)	-43(1)	-9(1)
C(6)	80(1)	82(2)	71(1)	-4(1)	-47(1)	-7(1)
C(7)	152(2)	96(2)	94(2)	-15(1)	-53(2)	-46(2)
C(8)	48(1)	46(1)	65(1)	4(1)	-30(1)	-6(1)
C(9)	73(1)	45(1)	79(1)	1(1)	-42(1)	-5(1)
C(10)	97(2)	46(1)	109(2)	4(1)	-57(2)	-11(1)
C(11)	88(2)	54(1)	124(2)	27(1)	-56(2)	-22(1)
C(12)	62(1)	72(2)	89(2)	23(1)	-30(1)	-18(1)
C(13)	57(1)	56(1)	72(1)	4(1)	-27(1)	-10(1)
C(14)	61(1)	47(1)	51(1)	3(1)	-27(1)	-9(1)
C(15)	68(1)	53(1)	79(1)	2(1)	-28(1)	-6(1)
C(16)	77(1)	65(2)	90(2)	1(1)	-24(1)	10(1)
C(17)	116(2)	56(1)	90(2)	-10(1)	-45(2)	13(1)
C(18)	115(2)	47(1)	103(2)	-2(1)	-64(2)	-8(1)
C(19)	79(1)	50(1)	81(1)	4(1)	-45(1)	-11(1)
C(20)	53(1)	48(1)	53(1)	-1(1)	-26(1)	-12(1)
C(21)	54(1)	56(1)	55(1)	-1(1)	-27(1)	-11(1)
C(22)	78(1)	74(1)	59(1)	-2(1)	-32(1)	-9(1)
C(23)	91(2)	113(2)	63(2)	-17(1)	-27(1)	3(1)
C(24)	85(2)	147(3)	55(2)	11(2)	-15(1)	-15(2)
C(25)	83(2)	107(2)	77(2)	34(1)	-27(1)	-22(1)
C(26)	67(1)	71(1)	67(1)	12(1)	-26(1)	-14(1)
C(27)	55(1)	64(1)	55(1)	10(1)	-27(1)	-17(1)
C(28)	70(1)	96(2)	72(1)	14(1)	-40(1)	-29(1)
C(29)	68(2)	144(2)	102(2)	33(2)	-53(1)	-40(2)
C(30)	61(2)	157(3)	104(2)	20(2)	-28(2)	-2(2)
C(31)	74(2)	114(2)	90(2)	-4(1)	-27(1)	17(1)
C(32)	64(1)	76(1)	73(1)	-1(1)	-32(1)	-1(1)
C(33)	62(1)	68(1)	55(1)	13(1)	-20(1)	-12(1)
C(34)	73(2)	97(2)	95(2)	21(1)	-31(1)	-31(1)
C(35)	78(2)	155(3)	139(3)	52(2)	-35(2)	-49(2)
C(36)	87(2)	193(4)	104(3)	50(2)	9(2)	-18(2)
C(37)	104(2)	161(3)	69(2)	12(2)	-3(2)	-7(2)
C(38)	82(2)	100(2)	52(1)	9(1)	-15(1)	-6(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for cd27199.

	x	y	z	U(eq)
H(5A)	-368	3809	-135	79
H(5B)	-1671	4446	776	79
H(6A)	-2566	2611	1007	87
H(6B)	-2277	3155	-192	87
H(7A)	-2223	545	383	162
H(7B)	-1001	144	-712	162
H(7C)	-2166	1125	-759	162
H(9)	-1136	1146	1818	75
H(10)	-2000	-581	2614	94
H(11)	-3244	-627	4477	100
H(12)	-3706	1081	5558	88
H(13)	-2877	2830	4766	73
H(15)	-3830	4077	3394	80
H(16)	-5023	5964	3960	98
H(17)	-3955	7543	4061	105
H(18)	-1683	7264	3492	98
H(19)	-480	5388	2901	78
H(22)	-897	4395	4791	82
H(23)	-2126	4367	6669	109
H(24)	-1995	2618	7662	119
H(25)	-600	913	6780	108
H(26)	656	935	4908	81
H(28)	2720	3067	4059	89
H(29)	4920	2149	3473	116
H(30)	5689	611	2172	133
H(31)	4300	-1	1425	116
H(32)	2149	911	1965	84
H(34)	2817	4011	1396	104
H(35)	4924	3852	28	149
H(36)	5367	2900	-1641	172
H(37)	3758	2016	-1902	146
H(38)	1683	2150	-557	99

Table 6. Torsion angles [deg] for cd27199.

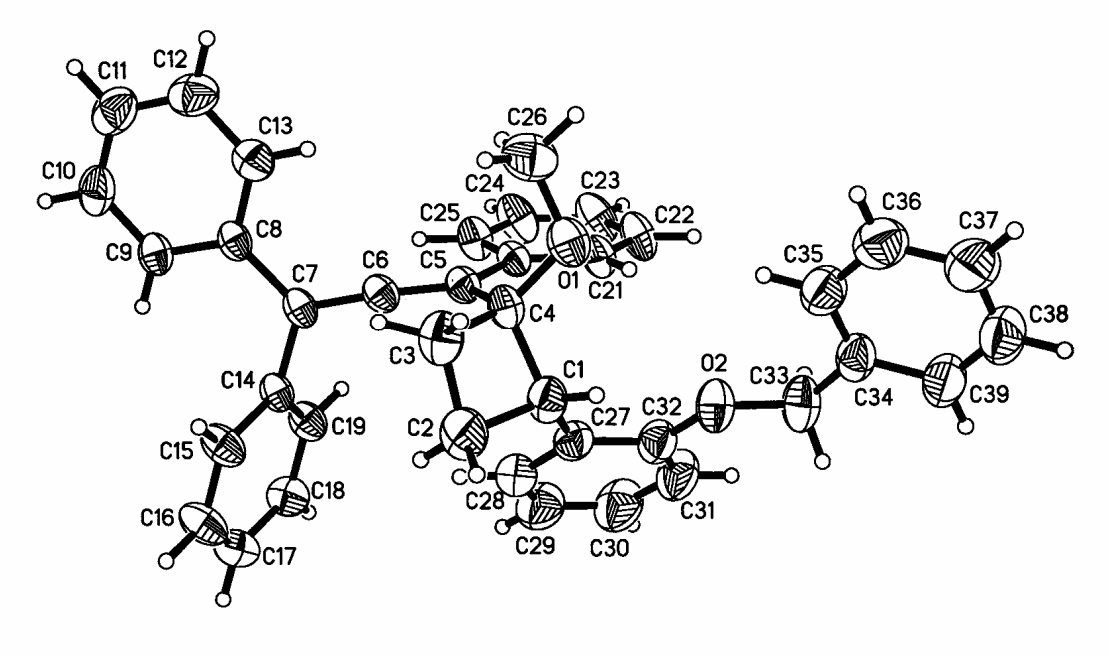
C(14)-C(1)-C(2)-C(20)	83.7(2)
C(8)-C(1)-C(2)-C(20)	-51.4(2)
C(4)-C(1)-C(2)-C(20)	-168.09(19)
C(14)-C(1)-C(2)-C(3)	-106.78(15)
C(8)-C(1)-C(2)-C(3)	118.17(14)
C(4)-C(1)-C(2)-C(3)	1.45(11)
C(20)-C(2)-C(3)-C(4)	167.4(2)
C(1)-C(2)-C(3)-C(4)	-1.64(13)
C(20)-C(2)-C(3)-C(33)	-15.1(3)
C(1)-C(2)-C(3)-C(33)	175.88(19)
C(33)-C(3)-C(4)-C(5)	-7.0(3)
C(2)-C(3)-C(4)-C(5)	170.51(19)
C(33)-C(3)-C(4)-C(1)	-175.86(19)
C(2)-C(3)-C(4)-C(1)	1.68(13)
C(14)-C(1)-C(4)-C(3)	115.94(14)
C(8)-C(1)-C(4)-C(3)	-109.18(15)
C(2)-C(1)-C(4)-C(3)	-1.58(12)
C(14)-C(1)-C(4)-C(5)	-53.7(2)
C(8)-C(1)-C(4)-C(5)	81.2(2)
C(2)-C(1)-C(4)-C(5)	-171.18(17)
C(3)-C(4)-C(5)-C(6)	120.6(2)
C(1)-C(4)-C(5)-C(6)	-74.0(2)
C(7)-O(1)-C(6)-C(5)	175.52(16)
C(4)-C(5)-C(6)-O(1)	-66.1(2)
C(14)-C(1)-C(8)-C(13)	-40.9(2)
C(4)-C(1)-C(8)-C(13)	-172.97(14)
C(2)-C(1)-C(8)-C(13)	95.32(18)
C(14)-C(1)-C(8)-C(9)	147.18(15)
C(4)-C(1)-C(8)-C(9)	15.1(2)
C(2)-C(1)-C(8)-C(9)	-76.56(18)
C(13)-C(8)-C(9)-C(10)	0.7(3)
C(1)-C(8)-C(9)-C(10)	172.88(16)
C(8)-C(9)-C(10)-C(11)	-1.2(3)
C(9)-C(10)-C(11)-C(12)	1.1(3)
C(10)-C(11)-C(12)-C(13)	-0.5(3)
C(9)-C(8)-C(13)-C(12)	-0.1(2)
C(1)-C(8)-C(13)-C(12)	-172.22(16)
C(11)-C(12)-C(13)-C(8)	0.0(3)
C(8)-C(1)-C(14)-C(15)	-33.1(2)
C(4)-C(1)-C(14)-C(15)	102.58(18)
C(2)-C(1)-C(14)-C(15)	-165.15(16)
C(8)-C(1)-C(14)-C(19)	152.00(16)
C(4)-C(1)-C(14)-C(19)	-72.29(18)
C(2)-C(1)-C(14)-C(19)	20.0(2)
C(19)-C(14)-C(15)-C(16)	-2.5(3)
C(1)-C(14)-C(15)-C(16)	-177.53(17)
C(14)-C(15)-C(16)-C(17)	0.0(3)
C(15)-C(16)-C(17)-C(18)	2.2(3)
C(16)-C(17)-C(18)-C(19)	-1.7(3)
C(17)-C(18)-C(19)-C(14)	-0.9(3)
C(15)-C(14)-C(19)-C(18)	2.9(3)
C(1)-C(14)-C(19)-C(18)	178.04(17)
C(3)-C(2)-C(20)-C(21)	-179.82(17)
C(1)-C(2)-C(20)-C(21)	-15.1(3)
C(3)-C(2)-C(20)-C(27)	-5.3(3)
C(1)-C(2)-C(20)-C(27)	159.42(16)
C(2)-C(20)-C(21)-C(22)	-51.5(2)
C(27)-C(20)-C(21)-C(22)	133.74(17)
C(2)-C(20)-C(21)-C(26)	129.03(18)
C(27)-C(20)-C(21)-C(26)	-45.7(2)
C(26)-C(21)-C(22)-C(23)	-1.7(3)
C(20)-C(21)-C(22)-C(23)	178.90(17)
C(21)-C(22)-C(23)-C(24)	0.2(3)
C(22)-C(23)-C(24)-C(25)	0.9(4)
C(23)-C(24)-C(25)-C(26)	-0.4(4)
C(24)-C(25)-C(26)-C(21)	-1.1(3)
C(22)-C(21)-C(26)-C(25)	2.1(3)
C(20)-C(21)-C(26)-C(25)	-178.44(16)

C(2)-C(20)-C(27)-C(32)	-49.6(2)
C(21)-C(20)-C(27)-C(32)	125.25(18)
C(2)-C(20)-C(27)-C(28)	132.91(18)
C(21)-C(20)-C(27)-C(28)	-52.2(2)
C(32)-C(27)-C(28)-C(29)	1.2(3)
C(20)-C(27)-C(28)-C(29)	178.78(18)
C(27)-C(28)-C(29)-C(30)	-1.2(4)
C(28)-C(29)-C(30)-C(31)	0.5(4)
C(29)-C(30)-C(31)-C(32)	0.2(4)
C(30)-C(31)-C(32)-C(27)	-0.1(4)
C(28)-C(27)-C(32)-C(31)	-0.6(3)
C(20)-C(27)-C(32)-C(31)	-178.2(2)
C(4)-C(3)-C(33)-C(38)	-42.4(3)
C(2)-C(3)-C(33)-C(38)	141.0(2)
C(4)-C(3)-C(33)-C(34)	139.0(2)
C(2)-C(3)-C(33)-C(34)	-37.6(3)
C(38)-C(33)-C(34)-C(35)	-0.9(3)
C(3)-C(33)-C(34)-C(35)	177.7(2)
C(33)-C(34)-C(35)-C(36)	1.5(4)
C(34)-C(35)-C(36)-C(37)	-1.7(5)
C(35)-C(36)-C(37)-C(38)	1.4(5)
C(36)-C(37)-C(38)-C(33)	-0.9(4)
C(34)-C(33)-C(38)-C(37)	0.6(3)
C(3)-C(33)-C(38)-C(37)	-178.0(2)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd27199 [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)



The crystal data of **4a** have been deposited in CCDC with number 650905. Empirical Formula: $C_{39}H_{34}O_2$; Formula Weight: 534.66; Crystal Color, Habit: colorless, prismatic; Crystal System: Monoclinic; Lattice Type: Primitive; Lattice Parameters: a = 11.9815(12)Å, b = 28.144(3)Å, c = 9.7933(10)Å, $\alpha = 90^o$, $\beta = 111.828(2)^o$, $\gamma = 90^o$, V = 3065.6(5)Å^3; Space group: P2(1)/c; Z = 4; D_{calc} = 1.158 g/cm^3; F_{000} = 1136; Diffractometer: Rigaku AFC7R; Residuals: R; Rw: 0.0520, 0.1163.

Table 1. Crystal data and structure refinement for cd27299.

Identification code	cd27299
Empirical formula	C39 H34 O2
Formula weight	534.66
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 11.9815(12) A alpha = 90 deg. b = 28.144(3) A beta = 111.828(2) deg. c = 9.7933(10) A gamma = 90 deg.
Volume	3065.6(5) A^3
Z, Calculated density	4, 1.158 Mg/m^3
Absorption coefficient	0.070 mm^-1
F(000)	1136
Crystal size	0.501 x 0.439 x 0.287 mm
Theta range for data collection	1.97 to 27.00 deg.
Limiting indices	-14<=h<=15, -33<=k<=35, -12<=l<=12
Reflections collected / unique	17934 / 6659 [R(int) = 0.0887]
Completeness to theta = 27.00	99.5 %
Absorption correction	Empirical
Max. and min. transmission	1.0000 and 0.7936
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6659 / 1 / 376
Goodness-of-fit on F^2	0.813
Final R indices [I>2sigma(I)]	R1 = 0.0520, wR2 = 0.1163
R indices (all data)	R1 = 0.1335, wR2 = 0.1447
Extinction coefficient	0.0031(6)
Largest diff. peak and hole	0.148 and -0.159 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for cd27299.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	455(2)	992(1)	6477(2)	81(1)
O(2)	747(2)	2168(1)	5151(2)	90(1)
C(1)	2217(2)	1522(1)	7075(3)	74(1)
C(2)	3186(3)	1368(1)	8548(3)	103(1)
C(3)	2442(2)	931(1)	8564(2)	84(1)
C(4)	1726(2)	999(1)	6892(2)	64(1)
C(5)	2140(2)	696(1)	5891(2)	57(1)
C(6)	3064(2)	413(1)	6471(2)	59(1)
C(7)	4045(2)	152(1)	7041(2)	55(1)
C(8)	4002(2)	-362(1)	7333(2)	55(1)
C(9)	5017(2)	-644(1)	7664(2)	66(1)
C(10)	4964(3)	-1130(1)	7824(3)	85(1)
C(11)	3902(3)	-1343(1)	7657(3)	96(1)
C(12)	2879(3)	-1069(1)	7344(3)	91(1)
C(13)	2934(2)	-584(1)	7190(2)	71(1)
C(14)	5222(2)	392(1)	7324(2)	54(1)
C(15)	6067(2)	441(1)	8736(2)	75(1)
C(16)	7138(2)	667(1)	8974(3)	86(1)
C(17)	7395(2)	846(1)	7820(3)	80(1)
C(18)	6558(2)	801(1)	6422(3)	76(1)
C(19)	5483(2)	576(1)	6174(2)	66(1)
C(20)	1550(2)	735(1)	4252(2)	55(1)
C(21)	657(2)	1062(1)	3572(2)	69(1)
C(22)	164(2)	1100(1)	2051(3)	87(1)
C(23)	541(2)	807(1)	1208(3)	93(1)
C(24)	1411(2)	478(1)	1851(3)	91(1)
C(25)	1915(2)	442(1)	3359(3)	72(1)
C(26)	-3(2)	532(1)	6573(3)	102(1)
C(27)	2572(2)	1759(1)	5937(3)	74(1)
C(28)	3651(3)	1663(1)	5776(3)	92(1)
C(29)	3951(3)	1870(1)	4678(4)	115(1)
C(30)	3182(4)	2182(1)	3725(4)	125(1)
C(31)	2107(3)	2295(1)	3864(4)	107(1)
C(32)	1801(3)	2084(1)	4951(3)	80(1)
C(33)	-87(3)	2488(1)	4190(3)	102(1)
C(34)	-1170(3)	2509(1)	4607(3)	82(1)
C(35)	-1555(3)	2126(1)	5190(3)	97(1)
C(36)	-2584(3)	2160(1)	5493(3)	110(1)
C(37)	-3241(3)	2567(1)	5221(4)	119(1)
C(38)	-2852(3)	2950(1)	4667(4)	121(1)
C(39)	-1829(3)	2925(1)	4368(3)	105(1)

Table 3. Bond lengths [A] and angles [deg] for cd27299.

O(1)-C(26)	1.423(3)
O(1)-C(4)	1.423(2)
O(2)-C(32)	1.369(3)
O(2)-C(33)	1.413(3)
C(1)-C(27)	1.490(3)
C(1)-C(2)	1.540(3)
C(1)-C(4)	1.570(3)
C(1)-H(1)	0.977(14)
C(2)-C(3)	1.522(3)
C(2)-H(2A)	0.9700
C(2)-H(2B)	0.9700
C(3)-C(4)	1.552(3)
C(3)-H(3A)	0.9700
C(3)-H(3B)	0.9700
C(4)-C(5)	1.514(3)
C(5)-C(6)	1.311(3)
C(5)-C(20)	1.498(3)
C(6)-C(7)	1.320(3)
C(7)-C(8)	1.481(3)
C(7)-C(14)	1.494(3)
C(8)-C(13)	1.384(3)
C(8)-C(9)	1.385(3)
C(9)-C(10)	1.380(3)
C(9)-H(9)	0.9300
C(10)-C(11)	1.360(3)
C(10)-H(10)	0.9300
C(11)-C(12)	1.383(4)
C(11)-H(11)	0.9300
C(12)-C(13)	1.377(3)
C(12)-H(12)	0.9300
C(13)-H(13)	0.9300
C(14)-C(19)	1.377(3)
C(14)-C(15)	1.384(3)
C(15)-C(16)	1.371(3)
C(15)-H(15)	0.9300
C(16)-C(17)	1.373(3)
C(16)-H(16)	0.9300
C(17)-C(18)	1.370(3)
C(17)-H(17)	0.9300
C(18)-C(19)	1.373(3)
C(18)-H(18)	0.9300
C(19)-H(19)	0.9300
C(20)-C(21)	1.380(3)
C(20)-C(25)	1.386(3)
C(21)-C(22)	1.387(3)
C(21)-H(21)	0.9300
C(22)-C(23)	1.357(3)
C(22)-H(22)	0.9300
C(23)-C(24)	1.361(3)
C(23)-H(23)	0.9300
C(24)-C(25)	1.376(3)
C(24)-H(24)	0.9300
C(25)-H(25)	0.9300
C(26)-H(26A)	0.9600
C(26)-H(26B)	0.9600
C(26)-H(26C)	0.9600
C(27)-C(28)	1.386(3)
C(27)-C(32)	1.399(3)
C(28)-C(29)	1.383(4)
C(28)-H(28)	0.9300
C(29)-C(30)	1.361(4)
C(29)-H(29)	0.9300
C(30)-C(31)	1.381(4)
C(30)-H(30)	0.9300
C(31)-C(32)	1.381(3)
C(31)-H(31)	0.9300
C(33)-C(34)	1.500(3)
C(33)-H(33A)	0.9700

C(33)-H(33B)	0.9700
C(34)-C(35)	1.377(3)
C(34)-C(39)	1.381(3)
C(35)-C(36)	1.374(4)
C(35)-H(35)	0.9300
C(36)-C(37)	1.361(4)
C(36)-H(36)	0.9300
C(37)-C(38)	1.364(4)
C(37)-H(37)	0.9300
C(38)-C(39)	1.365(4)
C(38)-H(38)	0.9300
C(39)-H(39)	0.9300

C(26)-O(1)-C(4)	113.08(18)
C(32)-O(2)-C(33)	118.5(2)
C(27)-C(1)-C(2)	120.1(2)
C(27)-C(1)-C(4)	122.30(19)
C(2)-C(1)-C(4)	87.86(18)
C(27)-C(1)-H(1)	112.5(12)
C(2)-C(1)-H(1)	107.9(12)
C(4)-C(1)-H(1)	102.7(11)
C(3)-C(2)-C(1)	89.8(2)
C(3)-C(2)-H(2A)	113.7
C(1)-C(2)-H(2A)	113.7
C(3)-C(2)-H(2B)	113.7
C(1)-C(2)-H(2B)	113.7
H(2A)-C(2)-H(2B)	110.9
C(2)-C(3)-C(4)	89.13(18)
C(2)-C(3)-H(3A)	113.8
C(4)-C(3)-H(3A)	113.8
C(2)-C(3)-H(3B)	113.8
C(4)-C(3)-H(3B)	113.8
H(3A)-C(3)-H(3B)	111.0
O(1)-C(4)-C(5)	112.36(18)
O(1)-C(4)-C(3)	114.36(18)
C(5)-C(4)-C(3)	115.36(18)
O(1)-C(4)-C(1)	111.19(19)
C(5)-C(4)-C(1)	113.66(18)
C(3)-C(4)-C(1)	87.65(18)
C(6)-C(5)-C(20)	119.66(19)
C(6)-C(5)-C(4)	119.2(2)
C(20)-C(5)-C(4)	121.03(19)
C(5)-C(6)-C(7)	175.8(2)
C(6)-C(7)-C(8)	122.4(2)
C(6)-C(7)-C(14)	117.29(19)
C(8)-C(7)-C(14)	120.25(18)
C(13)-C(8)-C(9)	117.7(2)
C(13)-C(8)-C(7)	121.2(2)
C(9)-C(8)-C(7)	121.0(2)
C(10)-C(9)-C(8)	121.3(2)
C(10)-C(9)-H(9)	119.3
C(8)-C(9)-H(9)	119.3
C(11)-C(10)-C(9)	120.2(3)
C(11)-C(10)-H(10)	119.9
C(9)-C(10)-H(10)	119.9
C(10)-C(11)-C(12)	119.6(3)
C(10)-C(11)-H(11)	120.2
C(12)-C(11)-H(11)	120.2
C(13)-C(12)-C(11)	120.1(3)
C(13)-C(12)-H(12)	119.9
C(11)-C(12)-H(12)	119.9
C(12)-C(13)-C(8)	121.1(2)
C(12)-C(13)-H(13)	119.5
C(8)-C(13)-H(13)	119.5
C(19)-C(14)-C(15)	118.4(2)
C(19)-C(14)-C(7)	120.29(19)
C(15)-C(14)-C(7)	121.35(19)
C(16)-C(15)-C(14)	120.4(2)
C(16)-C(15)-H(15)	119.8
C(14)-C(15)-H(15)	119.8
C(15)-C(16)-C(17)	120.8(2)
C(15)-C(16)-H(16)	119.6

C(17)-C(16)-H(16)	119.6
C(18)-C(17)-C(16)	118.9(2)
C(18)-C(17)-H(17)	120.5
C(16)-C(17)-H(17)	120.5
C(17)-C(18)-C(19)	120.6(2)
C(17)-C(18)-H(18)	119.7
C(19)-C(18)-H(18)	119.7
C(18)-C(19)-C(14)	120.8(2)
C(18)-C(19)-H(19)	119.6
C(14)-C(19)-H(19)	119.6
C(21)-C(20)-C(25)	117.5(2)
C(21)-C(20)-C(5)	122.5(2)
C(25)-C(20)-C(5)	119.91(19)
C(20)-C(21)-C(22)	120.9(2)
C(20)-C(21)-H(21)	119.6
C(22)-C(21)-H(21)	119.6
C(23)-C(22)-C(21)	120.1(2)
C(23)-C(22)-H(22)	120.0
C(21)-C(22)-H(22)	120.0
C(22)-C(23)-C(24)	120.2(3)
C(22)-C(23)-H(23)	119.9
C(24)-C(23)-H(23)	119.9
C(23)-C(24)-C(25)	120.1(2)
C(23)-C(24)-H(24)	119.9
C(25)-C(24)-H(24)	119.9
C(24)-C(25)-C(20)	121.2(2)
C(24)-C(25)-H(25)	119.4
C(20)-C(25)-H(25)	119.4
O(1)-C(26)-H(26A)	109.5
O(1)-C(26)-H(26B)	109.5
H(26A)-C(26)-H(26B)	109.5
O(1)-C(26)-H(26C)	109.5
H(26A)-C(26)-H(26C)	109.5
H(26B)-C(26)-H(26C)	109.5
C(28)-C(27)-C(32)	116.9(3)
C(28)-C(27)-C(1)	122.3(2)
C(32)-C(27)-C(1)	120.7(2)
C(29)-C(28)-C(27)	122.0(3)
C(29)-C(28)-H(28)	119.0
C(27)-C(28)-H(28)	119.0
C(30)-C(29)-C(28)	120.0(3)
C(30)-C(29)-H(29)	120.0
C(28)-C(29)-H(29)	120.0
C(29)-C(30)-C(31)	119.9(3)
C(29)-C(30)-H(30)	120.0
C(31)-C(30)-H(30)	120.0
C(30)-C(31)-C(32)	120.1(3)
C(30)-C(31)-H(31)	120.0
C(32)-C(31)-H(31)	120.0
O(2)-C(32)-C(31)	123.9(2)
O(2)-C(32)-C(27)	115.0(2)
C(31)-C(32)-C(27)	121.1(3)
O(2)-C(33)-C(34)	108.3(2)
O(2)-C(33)-H(33A)	110.0
C(34)-C(33)-H(33A)	110.0
O(2)-C(33)-H(33B)	110.0
C(34)-C(33)-H(33B)	110.0
H(33A)-C(33)-H(33B)	108.4
C(35)-C(34)-C(39)	118.4(3)
C(35)-C(34)-C(33)	122.7(3)
C(39)-C(34)-C(33)	119.0(3)
C(36)-C(35)-C(34)	120.0(3)
C(36)-C(35)-H(35)	120.0
C(34)-C(35)-H(35)	120.0
C(37)-C(36)-C(35)	121.1(3)
C(37)-C(36)-H(36)	119.4
C(35)-C(36)-H(36)	119.4
C(36)-C(37)-C(38)	119.0(3)
C(36)-C(37)-H(37)	120.5
C(38)-C(37)-H(37)	120.5
C(37)-C(38)-C(39)	120.8(3)
C(37)-C(38)-H(38)	119.6

C(39)-C(38)-H(38)	119.6
C(38)-C(39)-C(34)	120.7(3)
C(38)-C(39)-H(39)	119.7
C(34)-C(39)-H(39)	119.7

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for cd27299. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	70(1)	99(1)	78(1)	2(1)	34(1)	13(1)
O(2)	95(1)	90(1)	80(1)	25(1)	29(1)	28(1)
C(1)	85(2)	74(2)	52(2)	-3(1)	12(1)	14(2)
C(2)	120(2)	99(2)	57(2)	0(1)	-5(2)	6(2)
C(3)	105(2)	96(2)	50(2)	11(1)	28(2)	19(2)
C(4)	59(2)	77(2)	51(1)	5(1)	16(1)	12(1)
C(5)	53(1)	64(1)	50(1)	4(1)	15(1)	3(1)
C(6)	54(1)	73(2)	47(1)	2(1)	15(1)	1(1)
C(7)	50(1)	76(2)	34(1)	2(1)	12(1)	7(1)
C(8)	52(1)	73(2)	36(1)	7(1)	13(1)	6(1)
C(9)	66(2)	78(2)	47(1)	10(1)	12(1)	9(1)
C(10)	99(2)	85(2)	58(2)	16(1)	16(2)	22(2)
C(11)	126(3)	82(2)	75(2)	16(1)	34(2)	-3(2)
C(12)	102(2)	97(2)	81(2)	8(2)	40(2)	-22(2)
C(13)	69(2)	87(2)	59(2)	4(1)	23(1)	-3(1)
C(14)	49(1)	70(1)	41(1)	2(1)	15(1)	6(1)
C(15)	63(2)	111(2)	44(1)	-2(1)	11(1)	-9(2)
C(16)	69(2)	123(2)	54(2)	-18(1)	9(1)	-14(2)
C(17)	68(2)	88(2)	84(2)	-18(2)	29(2)	-13(1)
C(18)	79(2)	85(2)	69(2)	2(1)	34(2)	-8(2)
C(19)	61(2)	87(2)	48(1)	4(1)	17(1)	-2(1)
C(20)	45(1)	66(1)	50(1)	4(1)	12(1)	2(1)
C(21)	59(2)	85(2)	53(2)	3(1)	10(1)	13(1)
C(22)	66(2)	121(2)	58(2)	15(2)	4(1)	21(2)
C(23)	71(2)	154(3)	46(2)	7(2)	12(1)	14(2)
C(24)	75(2)	138(2)	55(2)	-8(2)	19(1)	18(2)
C(25)	65(2)	89(2)	58(2)	1(1)	19(1)	17(1)
C(26)	90(2)	119(2)	113(2)	-5(2)	56(2)	-14(2)
C(27)	80(2)	62(2)	71(2)	-5(1)	17(2)	0(1)
C(28)	81(2)	79(2)	106(2)	2(2)	24(2)	2(2)
C(29)	109(3)	97(2)	158(3)	15(2)	70(3)	-6(2)
C(30)	139(3)	103(3)	155(3)	36(2)	79(3)	3(2)
C(31)	120(3)	86(2)	124(3)	37(2)	57(2)	15(2)
C(32)	86(2)	69(2)	82(2)	14(1)	29(2)	8(2)
C(33)	107(2)	97(2)	92(2)	30(2)	25(2)	36(2)
C(34)	88(2)	76(2)	67(2)	1(1)	13(2)	17(2)
C(35)	106(2)	80(2)	97(2)	-4(2)	28(2)	8(2)
C(36)	115(3)	96(2)	117(3)	-12(2)	42(2)	-8(2)
C(37)	117(3)	103(3)	139(3)	-34(2)	50(2)	-3(2)
C(38)	120(3)	100(3)	140(3)	-11(2)	45(2)	29(2)
C(39)	109(3)	83(2)	111(3)	3(2)	28(2)	20(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for cd27299.

	x	y	z	U(eq)
H(2A)	3955	1293	8481	123
H(2B)	3285	1587	9349	123
H(3A)	1959	968	9162	101
H(3B)	2905	639	8806	101
H(9)	5749	-503	7781	80
H(10)	5656	-1312	8047	102
H(11)	3864	-1671	7752	115
H(12)	2152	-1212	7237	109
H(13)	2241	-403	6986	86
H(15)	5907	321	9529	90
H(16)	7697	698	9928	103
H(17)	8125	997	7986	96
H(18)	6719	924	5633	91
H(19)	4925	548	5218	80
H(21)	382	1260	4141	82
H(22)	-425	1326	1608	104
H(23)	203	831	188	112
H(24)	1664	277	1270	109
H(25)	2513	218	3787	86
H(26A)	186	319	5925	154
H(26B)	-860	549	6293	154
H(26C)	355	418	7565	154
H(28)	4188	1453	6425	110
H(29)	4678	1796	4591	139
H(30)	3379	2319	2981	151
H(31)	1588	2513	3226	128
H(33A)	-317	2381	3181	123
H(33B)	270	2801	4270	123
H(35)	-1119	1844	5379	116
H(36)	-2835	1900	5891	132
H(37)	-3946	2585	5410	142
H(38)	-3289	3232	4492	145
H(39)	-1572	3190	3998	126
H(1)	1601(15)	1698(6)	7290(20)	68(7)

Table 6. Torsion angles [deg] for cd27299.

C(27)-C(1)-C(2)-C(3)	-144.3(2)
C(4)-C(1)-C(2)-C(3)	-17.69(19)
C(1)-C(2)-C(3)-C(4)	17.89(19)
C(26)-O(1)-C(4)-C(5)	62.7(2)
C(26)-O(1)-C(4)-C(3)	-71.3(2)
C(26)-O(1)-C(4)-C(1)	-168.65(19)
C(2)-C(3)-C(4)-O(1)	-129.8(2)
C(2)-C(3)-C(4)-C(5)	97.6(2)
C(2)-C(3)-C(4)-C(1)	-17.55(19)
C(27)-C(1)-C(4)-O(1)	-102.6(3)
C(2)-C(1)-C(4)-O(1)	132.6(2)
C(27)-C(1)-C(4)-C(5)	25.4(3)
C(2)-C(1)-C(4)-C(5)	-99.4(2)
C(27)-C(1)-C(4)-C(3)	142.1(2)
C(2)-C(1)-C(4)-C(3)	17.35(19)
O(1)-C(4)-C(5)-C(6)	-135.1(2)
C(3)-C(4)-C(5)-C(6)	-1.6(3)
C(1)-C(4)-C(5)-C(6)	97.5(2)
O(1)-C(4)-C(5)-C(20)	48.1(3)
C(3)-C(4)-C(5)-C(20)	-178.41(19)
C(1)-C(4)-C(5)-C(20)	-79.3(2)
C(20)-C(5)-C(6)-C(7)	82(3)
C(4)-C(5)-C(6)-C(7)	-95(3)
C(5)-C(6)-C(7)-C(8)	-168(3)
C(5)-C(6)-C(7)-C(14)	9(3)
C(6)-C(7)-C(8)-C(13)	-8.0(3)
C(14)-C(7)-C(8)-C(13)	174.87(18)
C(6)-C(7)-C(8)-C(9)	167.64(19)
C(14)-C(7)-C(8)-C(9)	-9.5(3)
C(13)-C(8)-C(9)-C(10)	1.0(3)
C(7)-C(8)-C(9)-C(10)	-174.82(19)
C(8)-C(9)-C(10)-C(11)	0.0(3)
C(9)-C(10)-C(11)-C(12)	-0.8(4)
C(10)-C(11)-C(12)-C(13)	0.6(4)
C(11)-C(12)-C(13)-C(8)	0.4(4)
C(9)-C(8)-C(13)-C(12)	-1.2(3)
C(7)-C(8)-C(13)-C(12)	174.6(2)
C(6)-C(7)-C(14)-C(19)	-63.0(3)
C(8)-C(7)-C(14)-C(19)	114.3(2)
C(6)-C(7)-C(14)-C(15)	116.1(2)
C(8)-C(7)-C(14)-C(15)	-66.7(3)
C(19)-C(14)-C(15)-C(16)	-0.3(3)
C(7)-C(14)-C(15)-C(16)	-179.4(2)
C(14)-C(15)-C(16)-C(17)	-0.1(4)
C(15)-C(16)-C(17)-C(18)	0.5(4)
C(16)-C(17)-C(18)-C(19)	-0.5(4)
C(17)-C(18)-C(19)-C(14)	0.0(3)
C(15)-C(14)-C(19)-C(18)	0.4(3)
C(7)-C(14)-C(19)-C(18)	179.42(19)
C(6)-C(5)-C(20)-C(21)	-173.1(2)
C(4)-C(5)-C(20)-C(21)	3.7(3)
C(6)-C(5)-C(20)-C(25)	5.5(3)
C(4)-C(5)-C(20)-C(25)	-177.64(19)
C(25)-C(20)-C(21)-C(22)	-1.2(3)
C(5)-C(20)-C(21)-C(22)	177.5(2)
C(20)-C(21)-C(22)-C(23)	1.4(4)
C(21)-C(22)-C(23)-C(24)	-0.6(4)
C(22)-C(23)-C(24)-C(25)	-0.2(4)
C(23)-C(24)-C(25)-C(20)	0.4(4)
C(21)-C(20)-C(25)-C(24)	0.3(3)
C(5)-C(20)-C(25)-C(24)	-178.4(2)
C(2)-C(1)-C(27)-C(28)	31.0(4)
C(4)-C(1)-C(27)-C(28)	-77.4(3)
C(2)-C(1)-C(27)-C(32)	-150.7(2)
C(4)-C(1)-C(27)-C(32)	100.9(3)
C(32)-C(27)-C(28)-C(29)	-1.0(4)
C(1)-C(27)-C(28)-C(29)	177.3(3)
C(27)-C(28)-C(29)-C(30)	0.6(5)

C(28)-C(29)-C(30)-C(31)	0.6(5)
C(29)-C(30)-C(31)-C(32)	-1.3(5)
C(33)-O(2)-C(32)-C(31)	0.4(4)
C(33)-O(2)-C(32)-C(27)	-179.4(2)
C(30)-C(31)-C(32)-O(2)	-178.8(3)
C(30)-C(31)-C(32)-C(27)	0.9(4)
C(28)-C(27)-C(32)-O(2)	-180.0(2)
C(1)-C(27)-C(32)-O(2)	1.7(3)
C(28)-C(27)-C(32)-C(31)	0.3(4)
C(1)-C(27)-C(32)-C(31)	-178.0(2)
C(32)-O(2)-C(33)-C(34)	178.4(2)
O(2)-C(33)-C(34)-C(35)	-31.7(4)
O(2)-C(33)-C(34)-C(39)	149.9(2)
C(39)-C(34)-C(35)-C(36)	1.2(4)
C(33)-C(34)-C(35)-C(36)	-177.2(3)
C(34)-C(35)-C(36)-C(37)	0.3(5)
C(35)-C(36)-C(37)-C(38)	-1.4(5)
C(36)-C(37)-C(38)-C(39)	1.0(5)
C(37)-C(38)-C(39)-C(34)	0.6(5)
C(35)-C(34)-C(39)-C(38)	-1.6(4)
C(33)-C(34)-C(39)-C(38)	176.8(3)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd27299 [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)