Chemical Communications

Electronic Supporting Information

Access to unusual polycyclic spiro enones from 2,2´-bis(allyloxy)-1,1´-binaphthyls using Grubbs´ catalysts: An unprecedented *one-pot* RCM/Claisen sequence

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General methods: All reagents were obtained from commercial suppliers and used without further purification with the exception of compounds 2,2´-bis(allyloxy)-1,1´-binaphthyl (**1a**),[1] 6,6´-dibromo-2,2´-dihydroxy-1,1´-binaphthyl,[2] 7,7´-dibromo-2,2´-dihydroxy-1,1´-binaphthyl,[3] and 7,7´-dimethoxy-2,2´-dihydroxy-1,1´-binaphthyl,[4] which were prepared by following the methods reported in the literature. Flash chromatography was performed using Merck silica gel 60 (230-400 mesh). Infrared spectra were recorded on a Perkin-Elmer 1720-XFT spectrometer. NMR spectra were recorded on a Bruker DPX-300 instrument at 300 MHz (^{1}H) or 75.4 MHz (^{13}C). The chemical shift values (δ) are given in parts per million and are referred to the residual peak of the deuterated solvent used ($CDCl_3$). ESI-TOF high-resolution mass spectra were provided by the mass spectrometry service of the University of Seville (Spain).

Preparation of 2,2´-bis(allyloxy)-6,6´-dibromo-1,1´-binaphthyl (1b): A solution of 6,6´-dibromo-2,2´-dihydroxy-1,1´-binaphthyl (4.44 g, 10 mmol) in 60 mL of dry acetone was treated, under nitrogen atmosphere, with KOH (1.68 g, 30 mmol) at 60 °C for 1 hour. Allyl bromide (2.2 mL, 25 mmol) was then added and the resulting solution heated at 60 °C for additional 24 hours. The mixture was cooled to room temperature, filtered, and the filtrate concentrated under reduced pressure to give a pale yellow solid. The crude product was purified by flash chromatography (silica gel; eluent AcOEt/hexane 1:20) to afford **1b** as a white solid (0.40 g, 84%). IR (Nujol) ν 1580 (C=C) cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.55 (m, 4H), 5.05 (m, 4H), 5.76 (m, 2H), 7.01 (d, J = 9.0 Hz, 2H), 7.30 (dd, J = 9.0 and 2.0 Hz, 2H), 7.42 (d, J = 9.0 Hz, 2H), 7.87 (d, J = 9.0 Hz, 2H), 8.04 (d, J = 2.0 Hz, 2H) ppm; ^{13}C{^{1}H} NMR ($CDCl_3$, 75.4 MHz) δ 70.2, 116.8, 117.1, 117.9, 120.2, 127.5, 128.9,

130.0, 130.3, 130.7, 132.9, 133.7, 154.7 ppm; HRMS (ESI-TOF) *m/e* (M^+) 523.9803 ($C_{26}H_{20}Br_2O_2$ requires 523.9810).

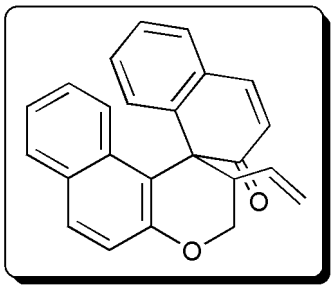
Preparation of 2,2´-bis(allyloxy)-7,7´-dibromo-1,1´-binaphthyl (1c): Compound **1c**, isolated as a white solid in 81% yield (4.25 g), was prepared as described for **1b** starting from 7,7´-dibromo-2,2´-dihydroxy-1,1´-binaphthyl (4.44 g, 10 mmol) and allyl bromide (2.2 mL, 25 mmol). IR (Nujol) ν 1615 (C=C) cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 4.58 (br, 4H), 5.06 (m, 4H), 5.77 (m, 2H), 7.37-7.46 (m, 6H), 7.75 (d, J = 8.7 Hz, 2H), 8.93 (d, J = 9.0 Hz, 2H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$, 75.4 MHz) δ 69.7, 115.6, 116.9, 118.7, 121.1, 127.1, 127.2, 127.7, 129.6, 129.7, 133.3, 135.3, 154.7 ppm; HRMS (ESI-TOF) *m/e* (M^+) 523.9797 ($C_{26}H_{20}Br_2O_2$ requires 523.9810).

Preparation of 2,2´-bis(allyloxy)-7,7´-dimethoxy-1,1´-binaphthyl (1d): Compound **1d**, isolated as a white solid in 74% yield (3.15 g), was prepared as described for **1b** starting from 7,7´-dimethoxy-2,2´-dihydroxy-1,1´-binaphthyl (4.26 g, 10 mmol) and allyl bromide (2.2 mL, 25 mmol). IR (Nujol) ν 1614 (C=C) cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 3.60 (s, 6H), 4.64 (br, 4H), 5.15 (m, 4H), 5.89 (m, 2H), 6.74 (s, 2H), 7.16 (d, J = 8.8 Hz, 2H), 7.36 (d, J = 8.9 Hz, 2H), 7.86 (d, J = 8.8 Hz, 2H), 7.95 (d, J = 8.9 Hz, 2H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$, 75.4 MHz) δ 55.0, 69.8, 104.2, 113.0, 116.3, 116.5, 119.6, 125.1, 129.1, 129.7, 134.0, 135.6, 154.8, 158.3 ppm; HRMS (ESI-TOF) *m/e* (M^+) 426.1833 ($C_{28}H_{26}O_4$ requires 426.1831).

General procedure for the catalytic reactions: Under nitrogen atmosphere, the corresponding 2,2´-bis(allyloxy)-1,1´-binaphthyl derivative **1a-d** (0.5 mmol), the ruthenium catalyst [$RuCl_2$(=CHPh)(PCy_3)$_2$] (0.012 g, 0.015 mmol; 3 mol%

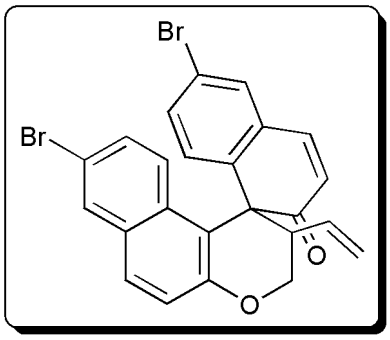
of Ru) and dichloromethane (10 mL) were introduced into a crimp-sealed thick-walled glass tube equipped with a pressure sensor and a magnetic stirrer. The tube was then placed inside the cavity of a CEM Discover® S-Class microwave synthesizer and exposed to MW-irradiation at a constant temperature of 120 °C (temperature monitored by a built-in infrared sensor) for 3 hours (MW power 300 W; P_{max} 70-100 psi). After removal of volatiles under vacuum, the solid residue was purified by column chromatography over silica gel. Thus, initial elution with EtOAc/hexanes (1:10) gave a colourless band from which spriro enones **2a-d** were obtained by solvent removal. Further elution with EtOAc/hexanes (1:5) gave a second band from which macrocycles **3a-b** could be isolated in pure form. Characterization data for all these new compounds are as follows:

2-vinyl-2,3-dihydro-2'*H*-spiro[benzo[*f*]chromene-1,1'-naphthalen]-2'-one (2a): White solid; Yield: 0.133 g (79%);



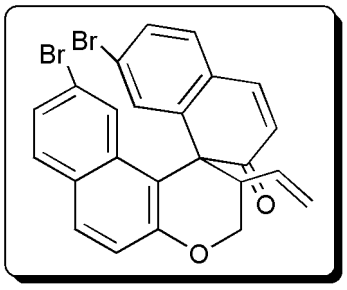
IR (Nujol) ν 1617 (C=C), 1660 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 2.99 (m, 1H), 4.12 (dd, J = 11.1 and 3.4 Hz, 1H), 4.56 (t, J = 10.8 Hz, 1H), 4.66 (d, J = 17.0 Hz, 1H), 4.93 (d, J = 10.5 Hz, 1H), 5.50 (m, 1H), 6.38 (d, J = 9.5 Hz, 1H), 6.65 (d, J = 8.5 Hz, 1H), 6.80 (d, J = 8.5 Hz, 1H), 7.01-7.27 (m, 5H), 7.42 (d, J = 8.5 Hz, 1H), 7.67 (m, 3H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$, 75.4 MHz) δ 54.4, 56.9, 63.0, 115.7, 118.5, 119.0, 123.0, 123.8, 126.3, 126.9, 127.5, 128.5, 129.1, 129.9, 13021, 130.3, 130.7, 131.1, 131.6, 145.5, 147.6, 154.9, 201.5 ppm; HRMS (ESI-TOF) *m/e* (M^+ + H) 339.1386 ($C_{24}H_{19}O_2$ requires 339.1385).

7´,8-dibromo-2-vinyl-2,3-dihydro-2'*H*-spiro[benzo[*f*]chromene-1,1'-naphthalen]-2'-one (2b): White



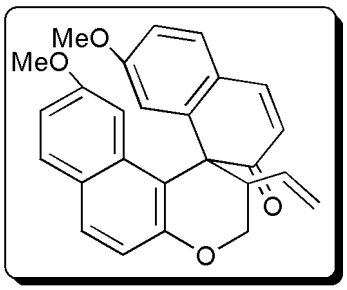
solid; Yield: 0.181 g (73%); IR (Nujol) ν 1616 (C=C), 1655 (C=O) cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 2.94 (m, 1H), 4.12 (dd, J = 10.5 and 2.6 Hz, 1H), 4.54 (t, J = 10.6 Hz, 1H), 4.72 (d, J = 17.2 Hz, 1H), 4.99 (d, J = 10.3 Hz, 1H), 5.49 (m, 1H), 6.40 (d, J = 9.9 Hz, 1H), 6.48 (d, J = 9.2 Hz, 1H), 6.64 (d, J = 8.3 Hz, 1H), 7.12-7.28 (m, 3H), 7.54-7.65 (m, 3H), 7.86 (br, 1H) ppm; $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 75.4 MHz) δ 56.4, 58.7, 64.9, 117.4, 118.8, 121.2, 122.3, 122.8, 127.3, 129.4, 131.2, 131.7, 132.1, 132.5, 132.6, 133.5, 133.6, 134.2, 135.5, 145.9, 148.0, 157.3, 202.5 ppm; HRMS (ESI-TOF) *m/e* (M^+ + H) 496.9590 ($C_{24}H_{17}Br_2O_2$ requires 496.9575).

6´,9-dibromo-2-vinyl-2,3-dihydro-2'*H*-spiro[benzo[*f*]chromene-1,1'-naphthalen]-2'-one (2c): White

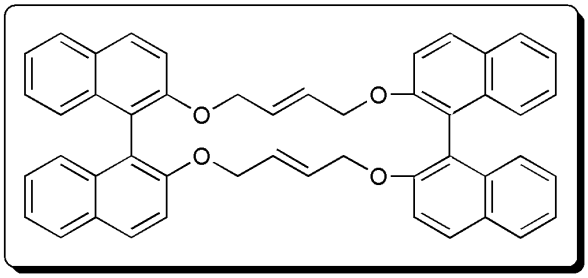


solid; Yield: 0.166 g (67%); IR (Nujol) ν 1615 (C=C), 1651 (C=O) cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.00 (m, 1H), 4.14 (dd, J = 11.2 and 3.7 Hz, 1H), 4.57 (t, J = 10.9 Hz, 1H), 4.74 (d, J = 17.0 Hz, 1H), 4.98 (d, J = 10.4 Hz, 1H), 5.47 (m, 1H), 6.44 (d, J = 9.9 Hz, 1H), 6.84 (d, J = 1.3 Hz, 1H), 6.94 (d, J = 1.8 Hz, 1H), 7.21-7.72 (m, 7H) ppm; $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 75.4 MHz) δ 54.5, 56.4, 63.0, 114.5, 119.3, 119.7, 121.1, 125.7, 125.9, 126.6, 126.7, 128.7, 129.4, 130.1, 130.3, 130.4, 130.5, 130.8, 132.8, 144.4, 149.0, 155.7, 200.3 ppm; HRMS (ESI-TOF) *m/e* (M^+ + H) 496.9592 ($C_{24}H_{17}Br_2O_2$ requires 496.9575).

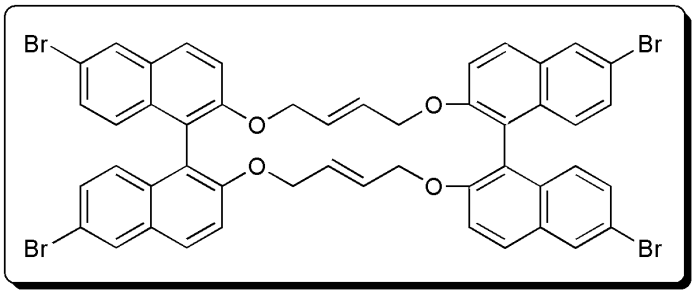
6´,9-dimethoxy-2-vinyl-2,3-dihydro-2'*H*-spiro[benzo[*f*]chromene-1,1'-naphthalen]-2'-one (2d): White solid; Yield: 0.139 g (70%); IR (Nujol) ν 1621 (C=C), 1656 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 3.06 (m, 1H), 3.34 (s, 3H), 3.61 (s, 3H), 4.12 (dd, J = 11.0 and 3.7 Hz, 1H), 4.62 (t, J = 11.2 Hz, 1H), 4.75 (d, J = 17.0 Hz, 1H), 4.98 (d, J = 10.4 Hz, 1H), 5.55 (m, 1H), 6.02 (d, J = 2.4 Hz, 1H), 6.32 (d, J = 9.9 Hz, 1H), 6.39 (d, J = 2.6 Hz, 1H), 6.78 (d, J = 2.4 Hz, 1H), 6.81 (d, J = 2.6 Hz, 1H), 7.07 (d, J = 9.0 Hz, 1H), 7.36 (d, J = 8.4 Hz, 1H), 7.55-7.65 (m, 3H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$, 75.4 MHz) δ 54.6, 54.7, 55.2, 56.9, 63.1, 103.1, 112.3, 113.9, 115.3, 115.9, 116.4, 118.6, 124.2, 124.5, 125.4, 129.5, 129.9, 130.3, 130.9, 132.9, 144.8, 150.2, 155.5, 157.9, 162.0, 201.3 ppm; HRMS (ESI-TOF) *m/e* (M^+ + H) 399.1592 ($C_{26}H_{23}O_4$ requires 399.1596).



(13*E*,31*E*)-12,15,30,33-tetrahydrotetranaphtho[2,1-*b*:1',2'-*d*:2'',1''-*l*:1''',2'''][1,6,11,16]tetraoxacycloicosine (3a): White solid; Yield: 0.014 g (8%); IR (Nujol) ν 1591 (C=C) cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 4.44 (m, 8H), 5.59 (br, 4H), 7.10-7.40 (m 16H), 7.82 (d, J = 8.9 Hz, 4H), 7.94 (d, J = 8.3 Hz, 4H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$, 75.4 MHz) δ 70.4, 116.8, 121.8, 125.4, 127.4, 128.2, 129.9, 131.1, 136.1, 155.7 ppm; HRMS (ESI-TOF) *m/e* (M^+) 676.2611 ($C_{48}H_{36}O_4$ requires 676.2614).



(13*E*,31*E*)-2,7,20,25-tetrabromo-12,15,30,33-tetrahydrotetranaphtho[2,1-*b*:1',2'-*d*:2'',1''-*l*:1''',2'''-*n*][1,6,11,16]tetraoxacycloicosine (3b): White solid; Yield: 0.022 g (9%); IR (Nujol) ν 1585 (C=C) cm^{-1}; ^1H NMR ($CDCl_3$,



300 MHz) δ 4.43 (m, 8H), 5.67 (br, 4H), 6.90 (d, J = 9.0 Hz, 4H), 7.13 (d, J = 9.0 Hz, 4H), 7.30 (m, 4H), 7.68 (d, J = 9.0 Hz, 4H), 8.08 (s, J = 1.7 Hz, 4H) ppm; $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 75.4 MHz) δ 70.0, 117.3, 119.3, 121.1, 129.0, 129.6, 130.3, 131.6, 131.8, 132.0, 134.4, 155.7 ppm; HRMS (ESI-TOF) *m/e* (M^+) 991.9042 ($C_{48}H_{32}Br_4O_4$ requires 991.9034).

Theoretical Calculations: The theoretical calculations were performed using the program package Gaussian03,[5] at density functional theory (DFT) level by means of the hybrid B3LYP functional.[6] In all geometry optimizations, Pople's 6-31G(d) split valence basis set was used for C, H and O elements. Frequency calculations were performed to determine whether the optimized geometries were minima on the potential energy surface. Optimized geometries of (*SS*)-**3a** and (*RS*)-**3a** are shown in Figures S1 and S2, respectively.

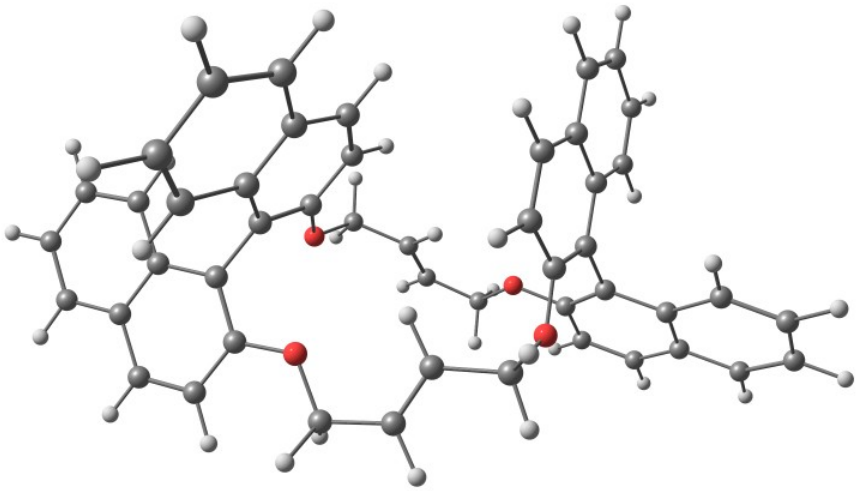


Figure S1. Optimized structure of (*SS*)-**3a**.

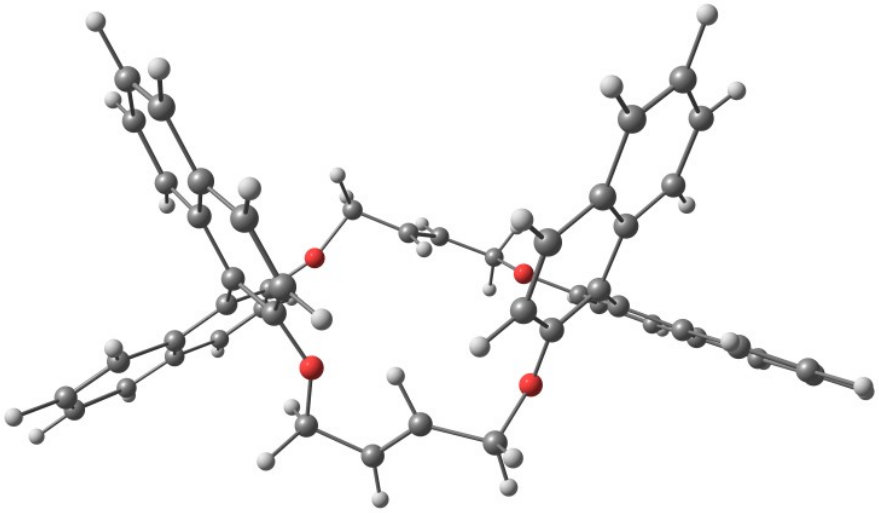


Figure S2. Optimized structure of (*RS*)-**3a**.

Cartesian coordinates and total electronic energies (Hartree) for the computed species (*SS*)-3a and (*RS*)-3a:

(*SS*)-**3a:** E(gas) : -2151.660805

C	7.368488	0.075914	1.887285
C	-4.017659	4.116627	2.215994
C	8.142743	-1.087474	1.652089
C	-4.814909	2.972042	2.465472
C	1.401880	-0.720719	2.836043
C	-0.629680	-2.153420	2.709704
C	0.013441	-1.039729	2.357931
C	-2.017476	-2.536608	2.289067
C	6.127336	0.223895	1.311730
C	-4.641238	1.819530	1.733543
C	-3.056172	4.076688	1.233399
C	7.649707	-2.082760	0.841618
C	1.664254	1.692317	1.288442
C	-4.603220	-2.850475	1.183292
C	2.471037	0.547581	1.056656
C	-3.657384	1.742211	0.705407

C	-2.849170	2.902547	0.459442
C	5.584610	-0.785319	0.464385
C	-3.824761	-1.688034	0.950717
C	-5.844354	-2.977295	0.602773
C	6.370863	-1.963386	0.232094
C	1.858807	2.836286	0.549669
C	-4.299365	-0.656319	0.145577
C	3.459576	0.561375	0.074063
C	-6.370728	-1.963220	-0.232475
C	-3.459601	0.561699	-0.074068
C	-1.858631	2.836408	-0.549987
C	4.299254	-0.656732	-0.145403
C	-7.649192	-2.082943	-0.842702
C	-5.584354	-0.785257	-0.464923
C	2.849510	2.902280	-0.459609
C	5.843970	-2.977961	-0.602202
C	3.657621	1.741839	-0.705435
C	-8.141773	-1.088074	-1.653983
C	3.056734	4.076378	-1.233569
C	-6.126601	0.223503	-1.313075
C	-2.471263	0.547784	-1.056868
C	-1.664374	1.692413	-1.288796
C	3.824147	-1.688934	-0.949653
C	-7.367423	0.075203	-1.889292
C	4.641567	1.819003	-1.733482
C	4.602446	-2.851516	-1.181983
C	4.018330	4.116174	-2.216069
C	4.815444	2.971478	-2.465435
C	-0.013885	-1.040119	-2.357380
C	2.016480	-2.537719	-2.287386
C	-1.402104	-0.720795	-2.835986
C	0.628961	-2.154145	-2.708582
H	7.760507	0.859425	2.529862
H	-4.168188	5.019298	2.800939

H	9.121020	-1.188767	2.112930
H	-5.571974	3.005901	3.244088
H	1.784077	-1.533728	3.459767
H	1.418906	0.195027	3.442608
H	-0.142866	-2.883991	3.356714
H	-2.635856	-2.726918	3.182003
H	5.542924	1.118232	1.498190
H	-5.257029	0.949262	1.932768
H	-2.436433	4.947229	1.031367
H	0.883194	1.673373	2.038404
H	8.232165	-2.981592	0.653731
H	-4.228977	-3.643455	1.819096
H	-1.979086	-3.481341	1.722525
H	-0.477117	-0.324295	1.704140
H	-6.436901	-3.870105	0.785515
H	1.237259	3.707842	0.737076
H	-8.231758	-2.981678	-0.654693
H	-1.236998	3.707878	-0.737522
H	6.436386	-3.870888	-0.784763
H	2.437072	4.946994	-1.031622
H	-9.119778	-1.189637	-2.115344
H	-5.542135	1.117781	-1.499623
H	5.257264	0.948666	-1.932663
H	-7.759067	0.858403	-2.532482
H	-0.883448	1.673357	-2.038904
H	4.169025	5.018801	-2.801039
H	0.476699	-0.324610	-1.703695
H	4.227756	-3.644959	-1.816954
H	5.572581	3.005196	-3.243987
H	1.977553	-3.482393	-1.720785
H	-1.418664	0.194911	-3.442660
H	-1.784281	-1.533754	-3.459793
H	2.635153	-2.728303	-3.180058
H	0.142085	-2.884748	-3.355499

O	2.349171	-0.613341	1.765054
O	-2.584869	-1.506519	1.500363
O	-2.349729	-0.613098	-1.765378
O	2.583877	-1.507758	-1.498531

(*RS*)-**3a**: E (gas) = -2151.574701

C	7.311635	-1.289481	-1.771841
C	-4.631431	4.426671	0.827904
C	7.740919	-2.525906	-1.234294
C	-5.382845	3.323643	1.300239
C	1.527228	-0.072105	2.733825
C	-0.585101	-1.237863	3.047652
C	0.192515	-0.422689	2.291280
C	-1.974173	-1.716619	2.779638
C	6.164538	-0.679555	-1.307793
C	-5.075325	2.039133	0.906934
C	-3.601424	4.213845	-0.059717
C	7.020472	-3.120833	-0.216093
C	2.345442	1.979931	-1.755298
C	-4.589278	-2.491533	1.847922
C	2.748638	0.718898	-1.244119
C	-4.003295	1.777003	0.003358
C	-3.265002	2.903088	-0.492027
C	5.386979	-1.279018	-0.276244
C	-3.825338	-1.462834	1.221565
C	-5.873842	-2.766605	1.434825
C	5.827439	-2.533466	0.276562
C	2.896927	3.138993	-1.267179
C	-4.382869	-0.711953	0.187297
C	3.732533	0.650039	-0.247903
C	-6.477174	-2.035431	0.389085
C	-3.645586	0.444908	-0.407725
C	-2.209979	2.669745	-1.404618

C	4.209192	-0.666721	0.255521
C	-7.815572	-2.282638	-0.025950
C	-5.717192	-1.000889	-0.247424
C	3.859972	3.123355	-0.228666
C	5.062509	-3.147502	1.309197
C	4.277327	1.855503	0.295710
C	-8.391688	-1.542446	-1.031933
C	4.403482	4.317358	0.313275
C	-6.341801	-0.268409	-1.300264
C	-2.598786	0.270028	-1.306735
C	-1.883257	1.396324	-1.803837
C	3.493349	-1.288273	1.305722
C	-7.639283	-0.529767	-1.679130
C	5.206314	1.850019	1.376074
C	3.934074	-2.558256	1.817579
C	5.310282	4.277472	1.349527
C	5.695641	3.028652	1.895996
C	-0.047325	-1.249501	-2.440219
C	2.288946	-1.652536	-2.207617
C	-1.488869	-1.272415	-2.841497
C	0.931889	-2.135800	-2.616698
H	7.894459	-0.809688	-2.562181
H	-4.868066	5.437561	1.167038
H	8.639392	-3.009997	-1.623481
H	-6.217528	3.492502	1.984792
H	1.764096	-0.471900	3.724138
H	1.797080	0.986011	2.710505
H	-0.152120	-1.630375	3.975005
H	-2.622572	-1.471397	3.632456
H	5.845506	0.273434	-1.731225
H	-5.661087	1.205709	1.293213
H	-3.026965	5.060770	-0.444927
H	1.589494	2.020606	-2.537700
H	7.367290	-4.059131	0.225235

H	-4.168146	-3.078719	2.658235
H	-1.923334	-2.810575	2.685198
H	-0.167009	-0.052875	1.328277
H	-6.437726	-3.565458	1.923088
H	2.580817	4.098838	-1.683717
H	-8.385092	-3.074121	0.468455
H	-1.645949	3.519974	-1.796477
H	5.380370	-4.120224	1.696734
H	4.091433	5.279182	-0.103253
H	-9.424482	-1.732255	-1.332758
H	-5.775938	0.510667	-1.811698
H	5.531528	0.895710	1.792350
H	-8.093631	0.048555	-2.486866
H	-1.067977	1.252158	-2.505678
H	5.731213	5.202604	1.749125
H	0.431619	-0.411877	-1.889181
H	3.351005	-3.043012	2.601020
H	6.388569	3.000423	2.740466
H	2.754941	-2.207846	-1.388284
H	-1.688470	-0.516926	-3.609269
H	-1.794670	-2.240788	-3.250077
H	2.991806	-1.614410	-3.048743
H	0.802636	-3.133449	-3.055543
O	2.091864	-0.356439	-1.726020
O	-2.532132	-1.131933	1.604308
O	-2.291181	-1.017518	-1.665369
O	2.413532	-0.745496	1.786398

References:

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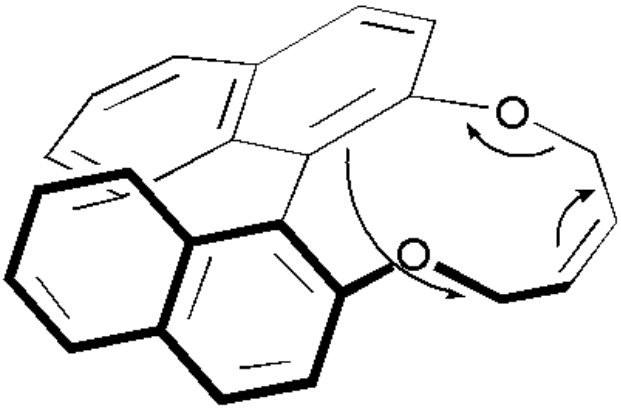
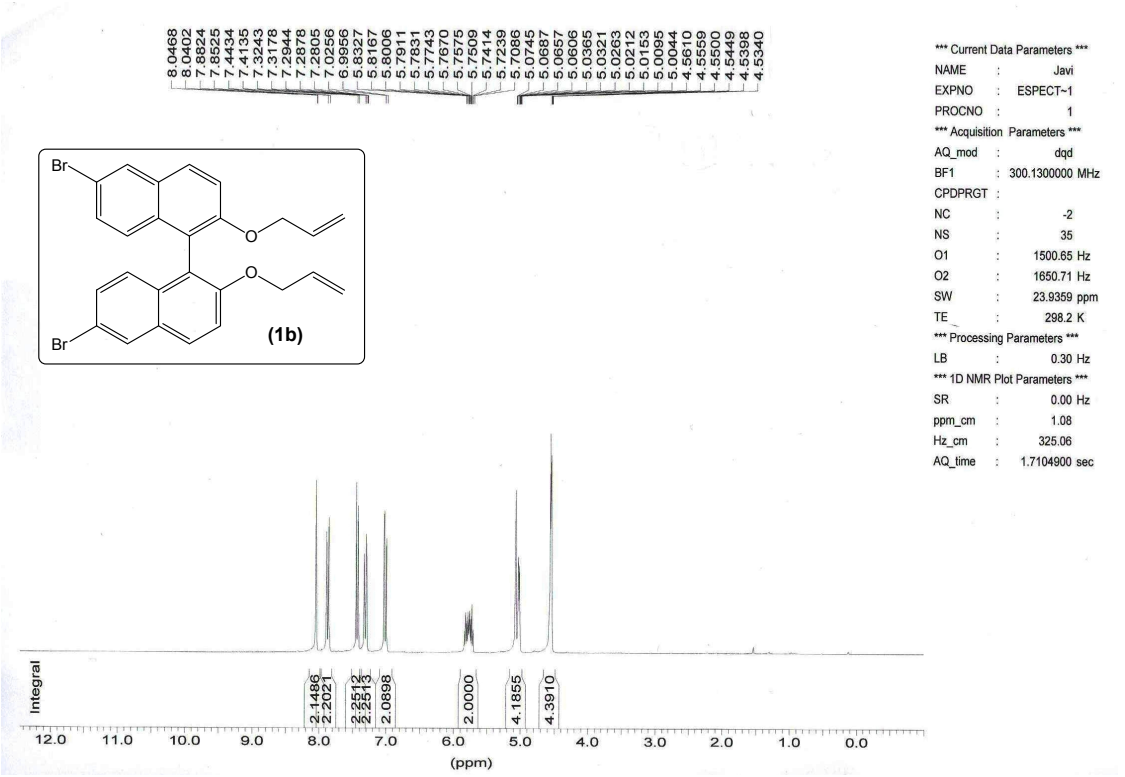
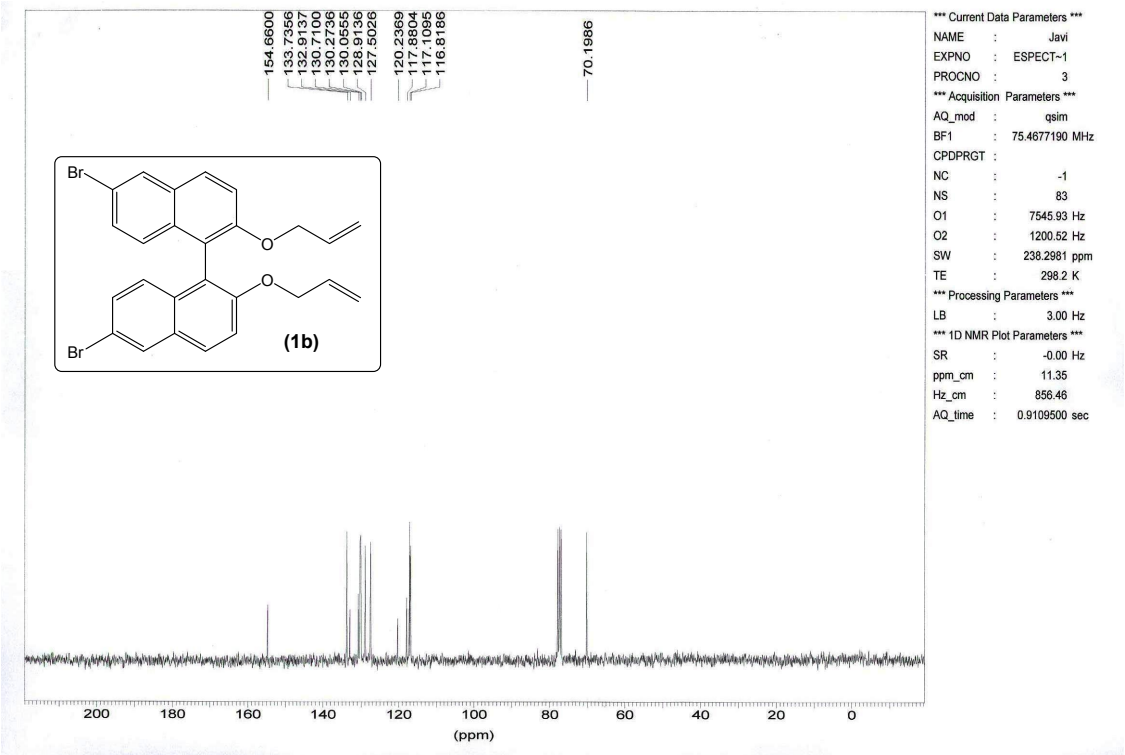
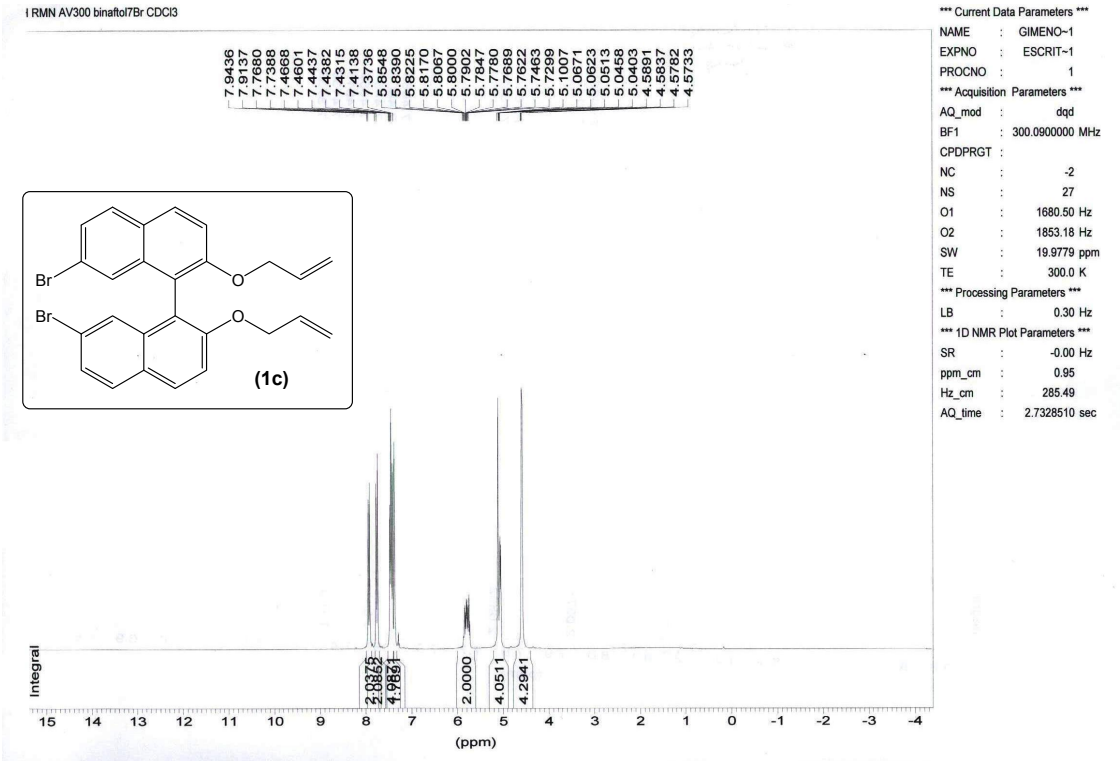


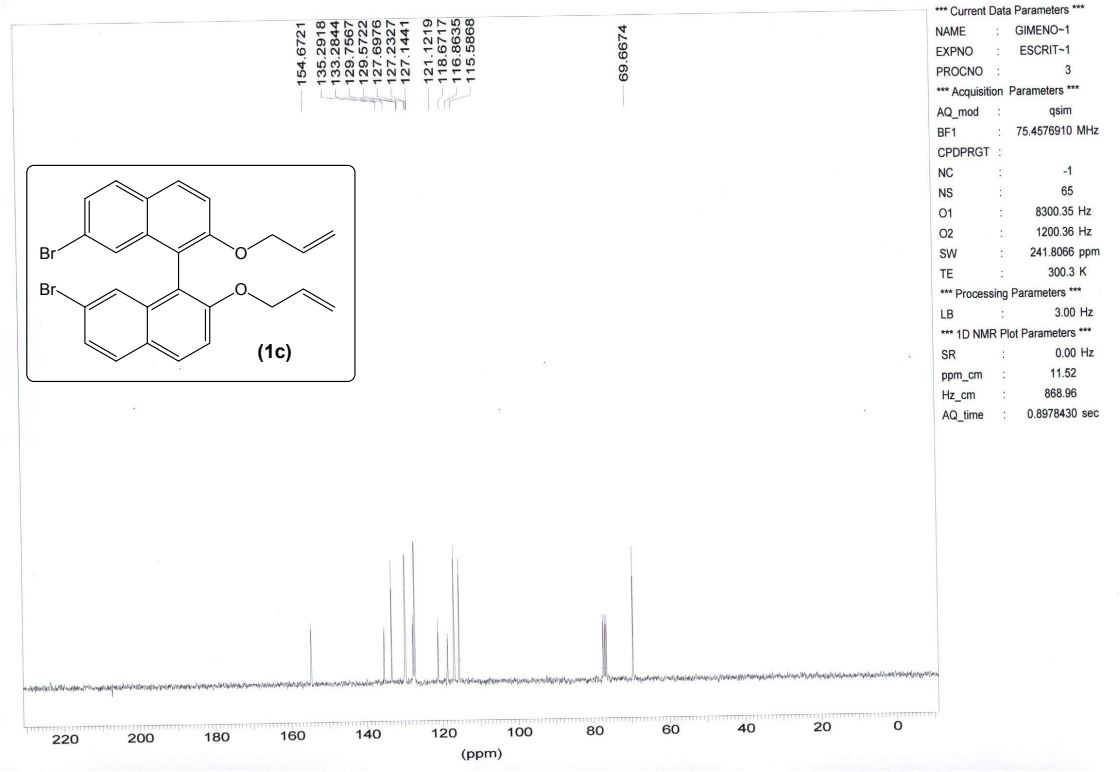
Figure S3. The twisted conformation adopted by the 1,1′-binaphthyl-2,2′-diyl unit in intermediate **A**.

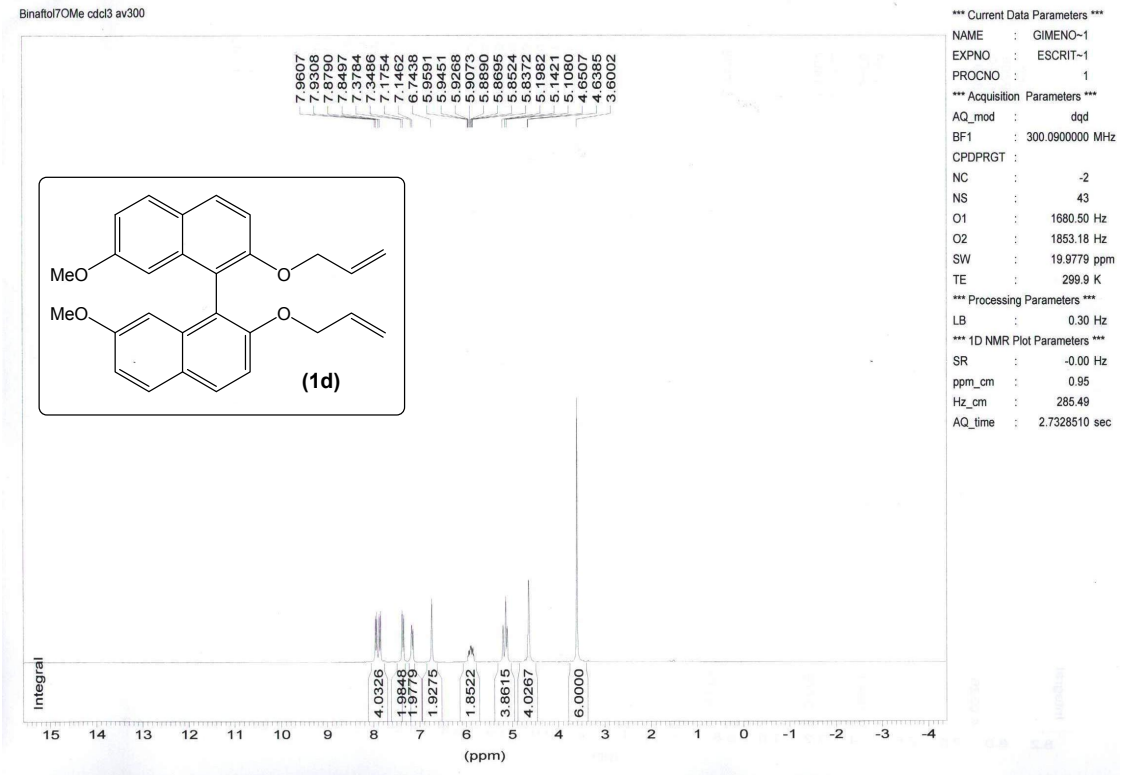
Copies of the ^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of all new compounds:

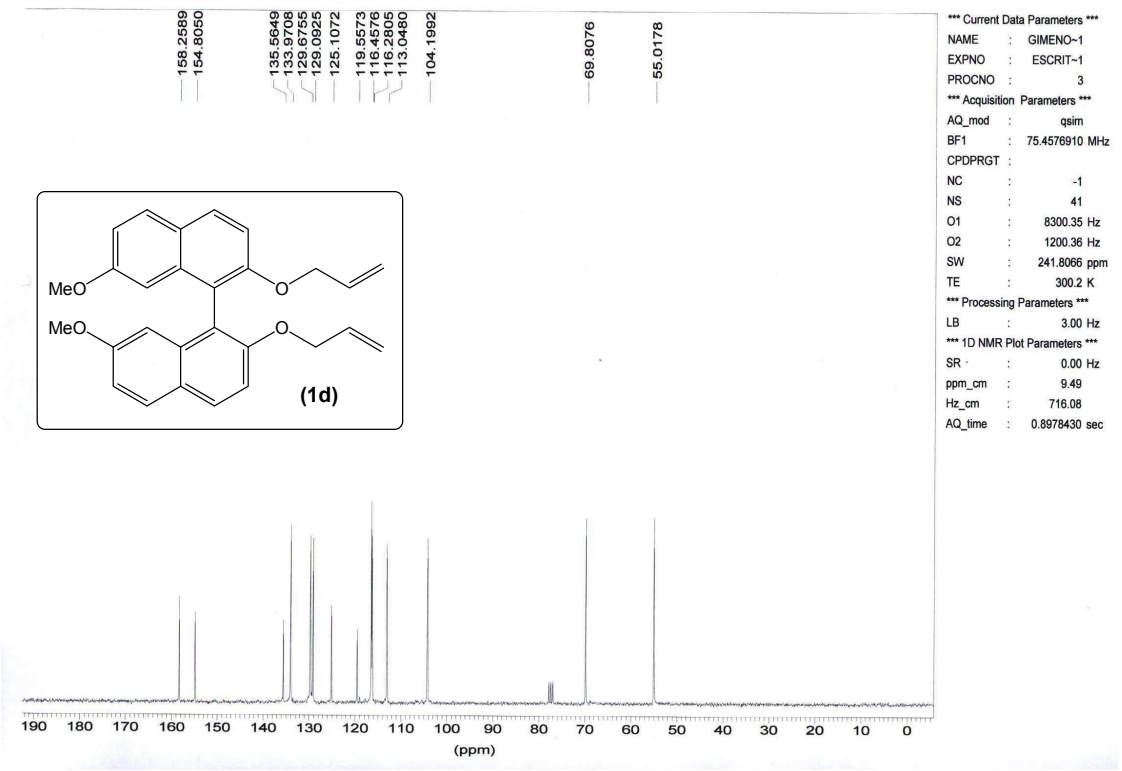


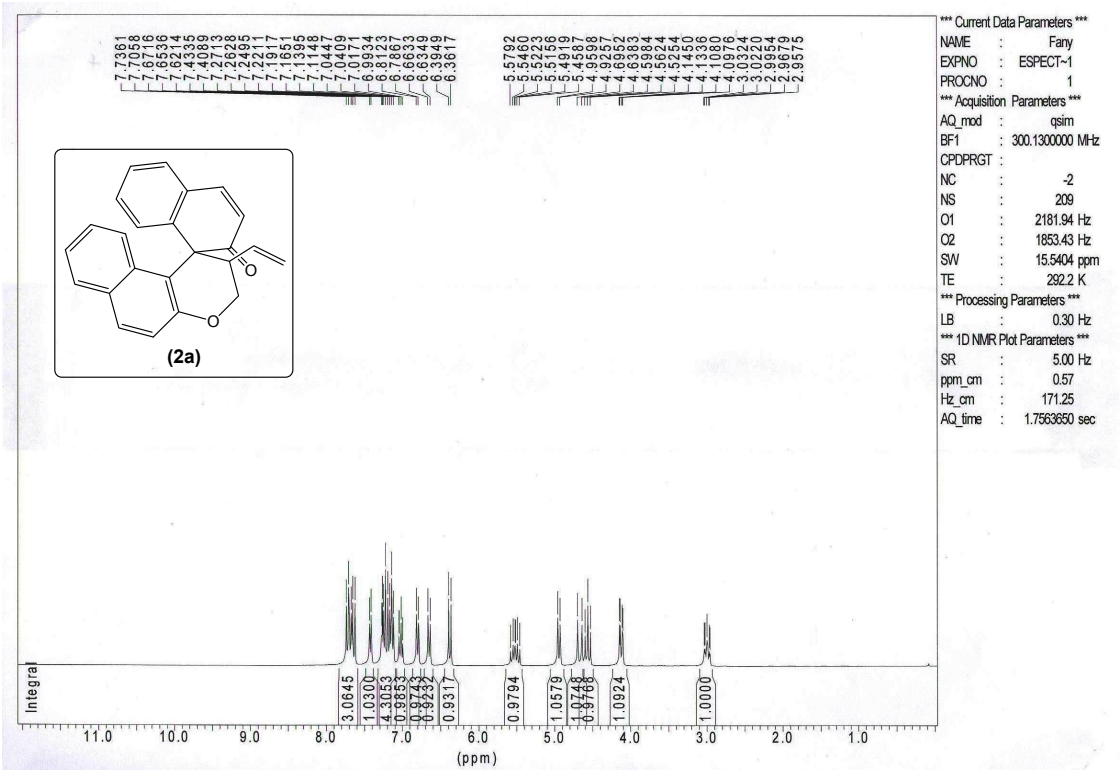


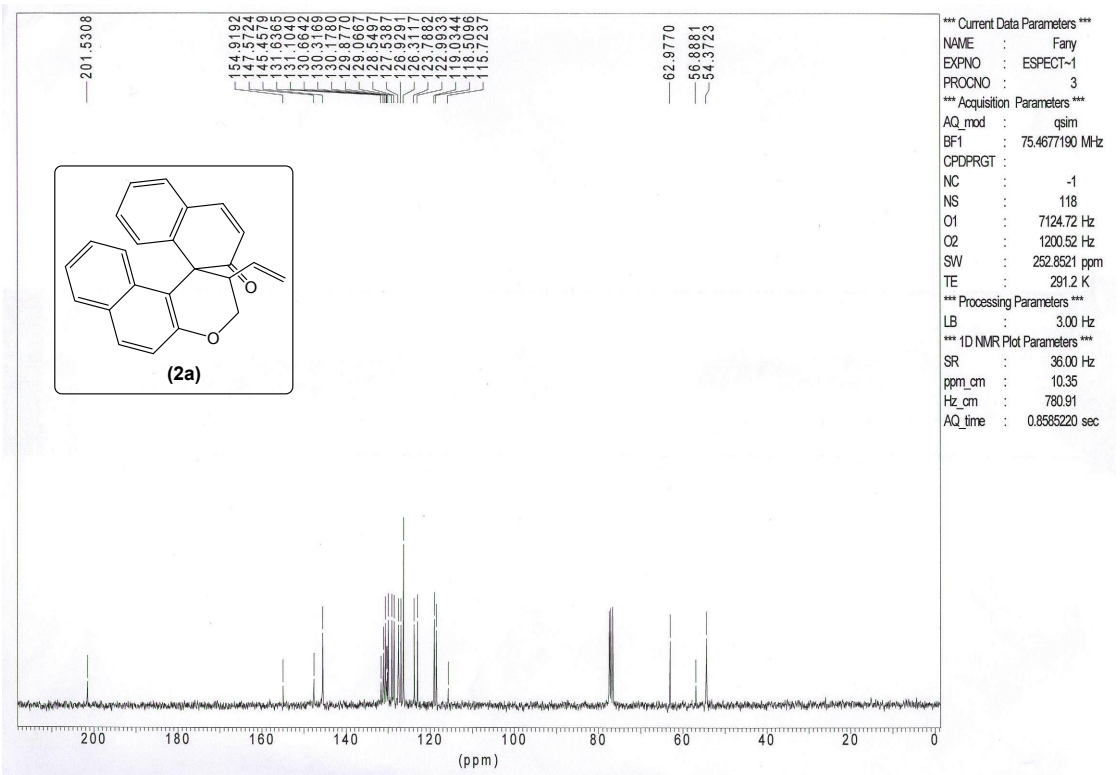

I RMN AV300 binaftol7Br CDCl3
7.9436
7.9137
7.7680
7.7388
7.4668
7.4601
7.4437
7.4382
7.4315
7.4138
7.3736
5.8548
5.8390
5.8225
5.8170
5.8067
5.8000
5.7902
5.7847
5.7780
5.7689
5.7622
5.7463
5.7299
5.1007
5.0671
5.0623
5.0513
5.0458
5.0403
4.5891
4.5837
4.5782
4.5733
Br
Br
O
O
(1c)
Integral
2.0000
4.0511
4.2941
(ppm)
*** Current Data Parameters ***
NAME : GIMENO~1
EXPNO : ESCRIT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 300.0900000 MHz
CPDPRGT :
NC : -2
NS : 27
O1 : 1680.50 Hz
O2 : 1853.18 Hz
SW : 19.9779 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : -0.00 Hz
ppm_cm : 0.95
Hz_cm : 285.49
AQ_time : 2.7328510 sec


154.6721
135.2918
133.2644
129.7567
129.5722
127.6976
127.2327
127.1441
121.1219
118.6717
116.8635
115.5868
69.6674
Br
Br
O
O
(1c)
(ppm)
*** Current Data Parameters ***
NAME : GIMENO~1
EXPNO : ESCRIT~1
PROCNO : 3
*** Acquisition Parameters ***
AQ_mod : qsim
BF1 : 75.4576910 MHz
CPDPRGT :
NC : -1
NS : 65
O1 : 8300.35 Hz
O2 : 1200.36 Hz
SW : 241.8066 ppm
TE : 300.3 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
ppm_cm : 11.52
Hz_cm : 868.96
AQ_time : 0.8978430 sec


Binaftol7OMe cdcl3 av300
7.9607
7.9308
7.8790
7.8497
7.3784
7.3486
7.1754
7.1462
6.7438
5.9591
5.9451
5.9268
5.9073
5.8890
5.8695
5.8524
5.8372
5.1982
5.1421
5.1080
4.6507
4.6385
3.6002
MeO
O
MeO
O
(1d)
Integral
4.0326
1.9848
1.9779
1.9275
1.8522
3.8615
4.0267
6.0000
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
(ppm)
*** Current Data Parameters ***
NAME : GIMENO~1
EXPNO : ESCRIT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 300.0900000 MHz
CPDPRGT :
NC : -2
NS : 43
O1 : 1680.50 Hz
O2 : 1853.18 Hz
SW : 19.9779 ppm
TE : 299.9 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : -0.00 Hz
ppm_cm : 0.95
Hz_cm : 285.49
AQ_time : 2.7328510 sec


158.2589
154.8050
135.5649
133.9708
129.6755
129.0925
125.1072
119.5573
116.4576
116.2805
113.0480
104.1992
69.8076
55.0178
MeO
O
MeO
O
(1d)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
(ppm)
*** Current Data Parameters ***
NAME : GIMENO~1
EXPNO : ESCRIT~1
PROCNO : 3
*** Acquisition Parameters ***
AQ_mod : qsim
BF1 : 75.4576910 MHz
CPDPRGT :
NC : -1
NS : 41
O1 : 8300.35 Hz
O2 : 1200.36 Hz
SW : 241.8066 ppm
TE : 300.2 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
ppm_cm : 9.49
Hz_cm : 716.08
AQ_time : 0.8978430 sec


(2a)
*** Current Data Parameters ***
NAME : Fany
EXPNO : ESPECT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : qsim
BF1 : 300.1300000 MHz
CPDPRGT :
NC : -2
NS : 209
O1 : 2181.94 Hz
O2 : 1853.43 Hz
SW : 15.5404 ppm
TE : 292.2 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : 5.00 Hz
ppm_cm : 0.57
Hz_cm : 171.25
AQ_time : 1.7563650 sec
Integral
(ppm)


(2a)
*** Current Data Parameters ***
NAME : Fany
EXPNO : ESPECT~1
PROCNO : 3
*** Acquisition Parameters ***
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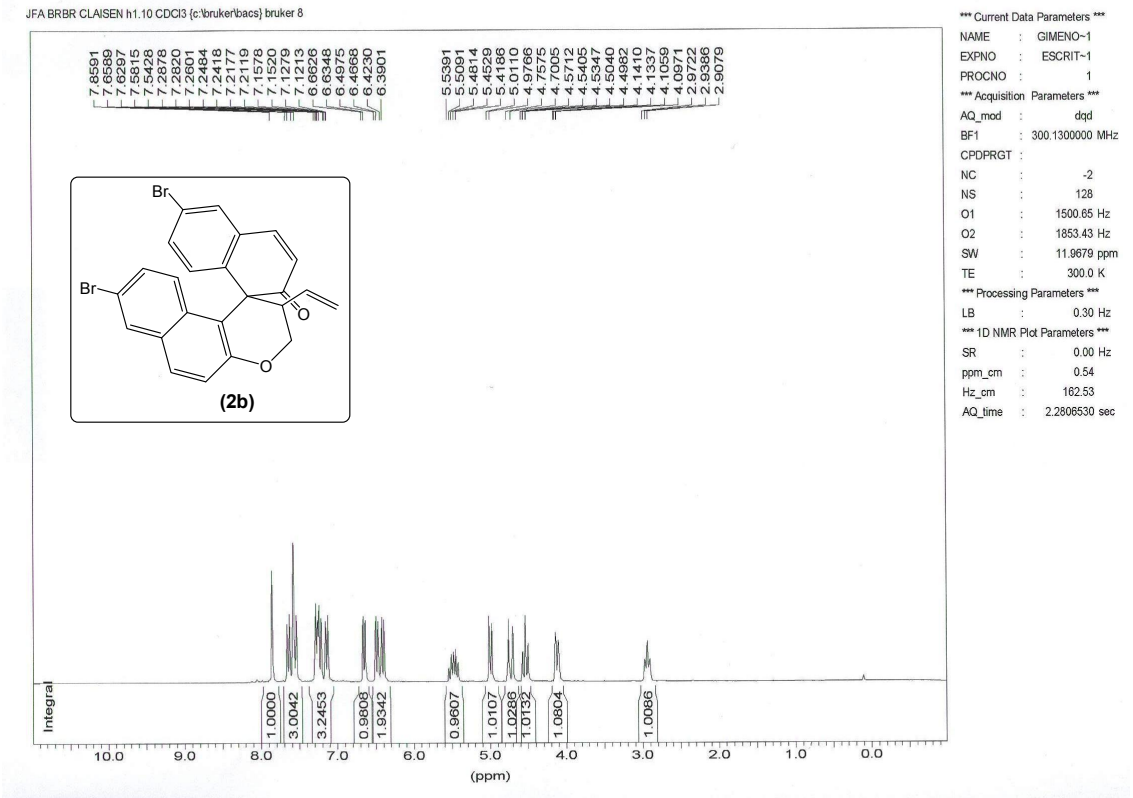
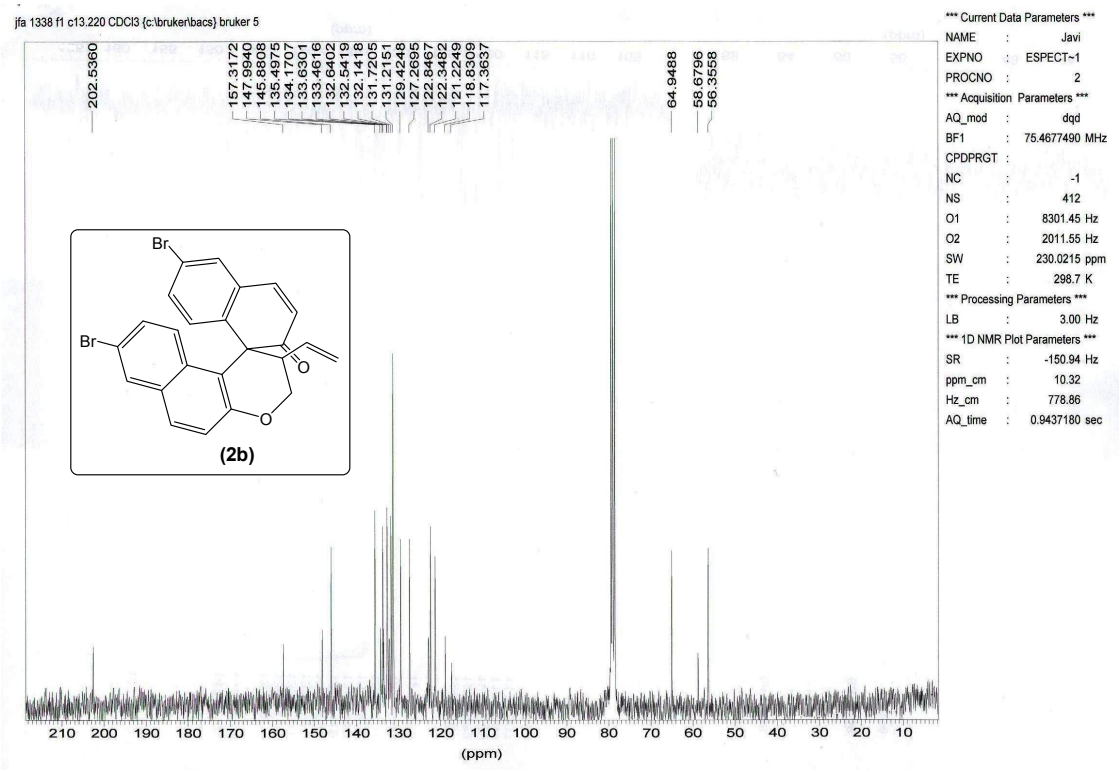
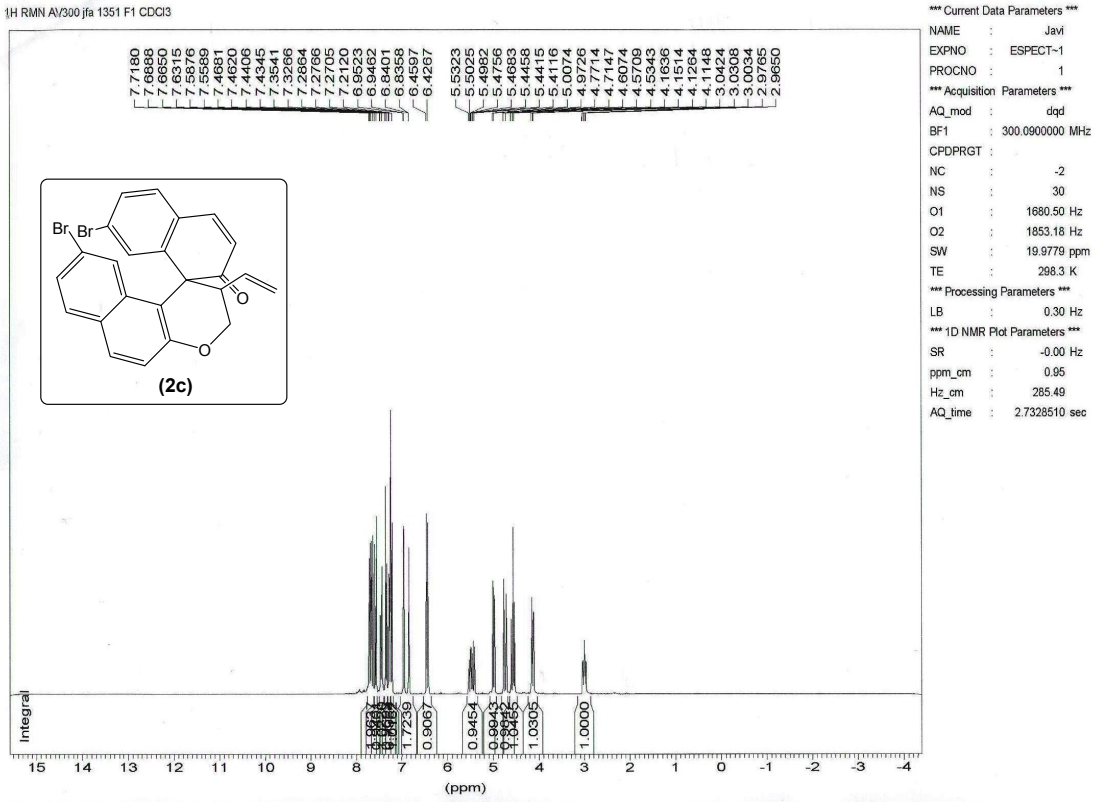
BF1 : 75.4677190 MHz
CPDPRGT :
NC : -1
NS : 118
O1 : 7124.72 Hz
O2 : 1200.52 Hz
SW : 252.8521 ppm
TE : 291.2 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : 36.00 Hz
ppm_cm : 10.35
Hz_cm : 780.91
AQ_time : 0.8585220 sec
(ppm)


JFA BRBR CLAISEN h1.10 CDCl3 {c:\bruker\bacs} bruker 8
7.8591 7.6589 7.6297 7.5815 7.5428 7.2878 7.2820 7.2601 7.2484 7.2418 7.2177 7.2119 7.1578 7.1520 7.1279 7.1213 6.6626 6.6348 6.4975 6.4668 6.4230 6.3901
5.5381 5.5091 5.4814 5.4529 5.4186 5.0110 4.9766 4.7575 4.7005 4.5712 4.5405 4.5347 4.5040 4.4982 4.1410 4.1337 4.1059 4.0971 2.9722 2.9386 2.9079
Br
Br
O
O
(2b)
Integral
1.0000 3.0042 3.2453 0.9808 1.9342 0.9607 1.0107 1.0286 1.0132 1.0804 1.0086
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
(ppm)
*** Current Data Parameters ***
NAME : GIMENO~1
EXPNO : ESCRIT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 300.1300000 MHz
CPDPRGT :
NC : -2
NS : 128
O1 : 1500.65 Hz
O2 : 1853.43 Hz
SW : 11.9679 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
ppm_cm : 0.54
Hz_cm : 162.53
AQ_time : 2.2806530 sec


jfa 1338 f1 c13.220 CDCl3 {c:\bruker\bacs} bruker 5
202.5360
157.3172 147.9940 145.8808 135.4975 134.1707 133.6301 133.4616 132.6402 132.5419 132.1418 131.7205 131.2151 129.4248 127.2695 122.8467 122.3482 121.2249 118.8309 117.3637
64.9488 58.6796 56.3558
Br
Br
O
O
(2b)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
(ppm)
*** Current Data Parameters ***
NAME : Javi
EXPNO : ESPECT~1
PROCNO : 2
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 75.4677490 MHz
CPDPRGT :
NC : -1
NS : 412
O1 : 8301.45 Hz
O2 : 2011.55 Hz
SW : 230.0215 ppm
TE : 298.7 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : -150.94 Hz
ppm_cm : 10.32
Hz_cm : 778.86
AQ_time : 0.9437180 sec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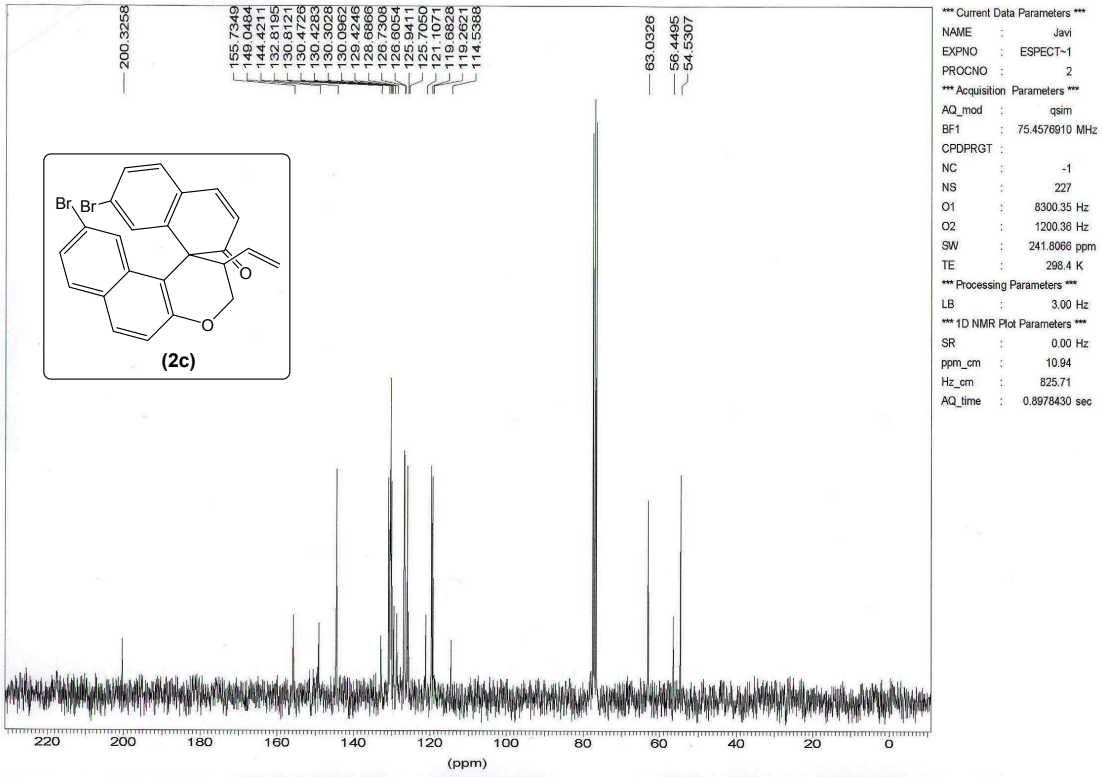
7.6888
7.6650
7.6315
7.5876
7.5589
7.4681
7.4620
7.4406
7.4345
7.3541
7.3266
7.2864
7.2766
7.2705
7.2120
6.9523
6.9462
6.8401
6.8358
6.4597
6.4267
5.5323
5.5025
5.4982
5.4756
5.4683
5.4458
5.4415
5.4116
5.0074
4.9726
4.7714
4.7147
4.6074
4.5709
4.5343
4.1636
4.1514
4.1264
4.1148
3.0424
3.0308
3.0034
2.9785
2.9650
Br
Br
O
O
(2c)
Integral
1.7239
0.9067
0.9454
0.9043
1.0305
1.0000
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
(ppm)
*** Current Data Parameters ***
NAME : Javi
EXPNO : ESPECT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 300.0900000 MHz
CPDPRGT :
NC : -2
NS : 30
O1 : 1680.50 Hz
O2 : 1853.18 Hz
SW : 19.9779 ppm
TE : 298.3 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : -0.00 Hz
ppm_cm : 0.95
Hz_cm : 285.49
AQ_time : 2.7328510 sec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155.7349
149.0484
144.4211
132.8195
130.8121
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130.4283
130.3028
130.0962
129.4246
128.8866
126.7308
126.6054
125.9411
125.7050
121.1077
119.8828
119.2621
114.5368
63.0326
56.4495
54.5307
Br
Br
O
O
(2c)
220 200 180 160 140 120 100 80 60 40 20 0
(ppm)
*** Current Data Parameters ***
NAME : Javi
EXPNO : ESPECT~1
PROCNO : 2
*** Acquisition Parameters ***
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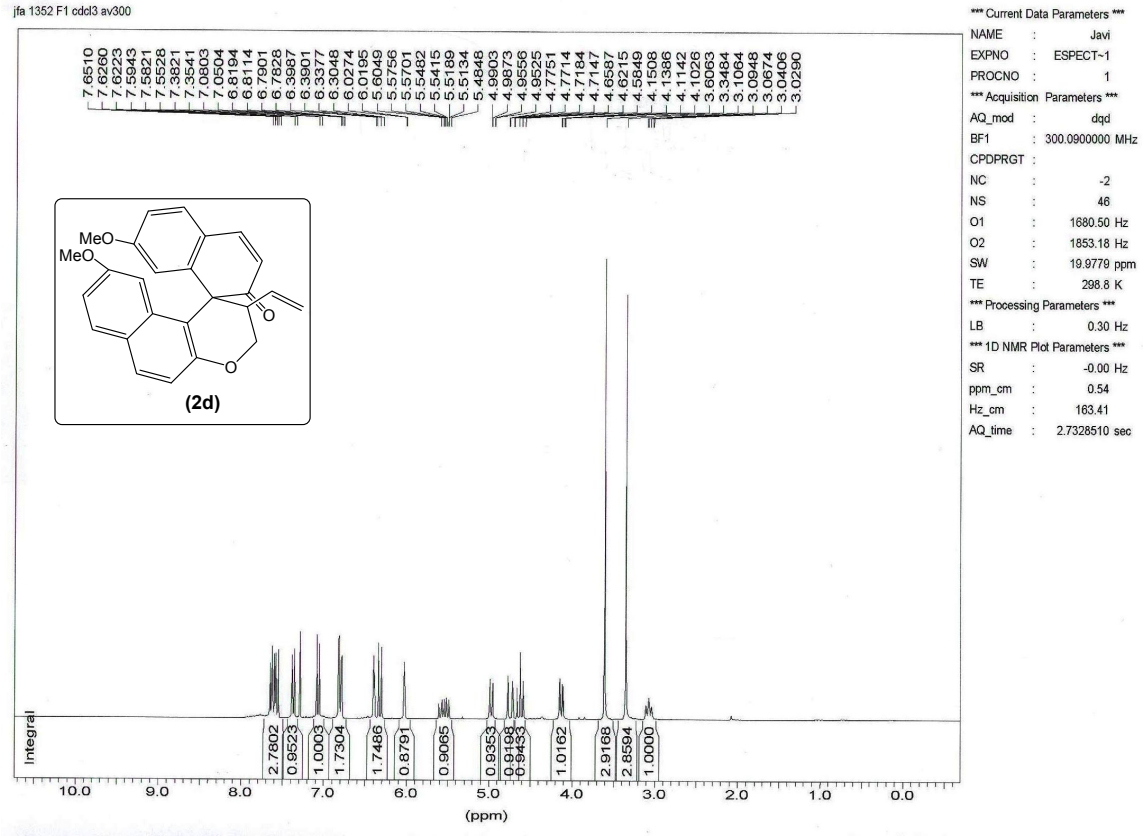
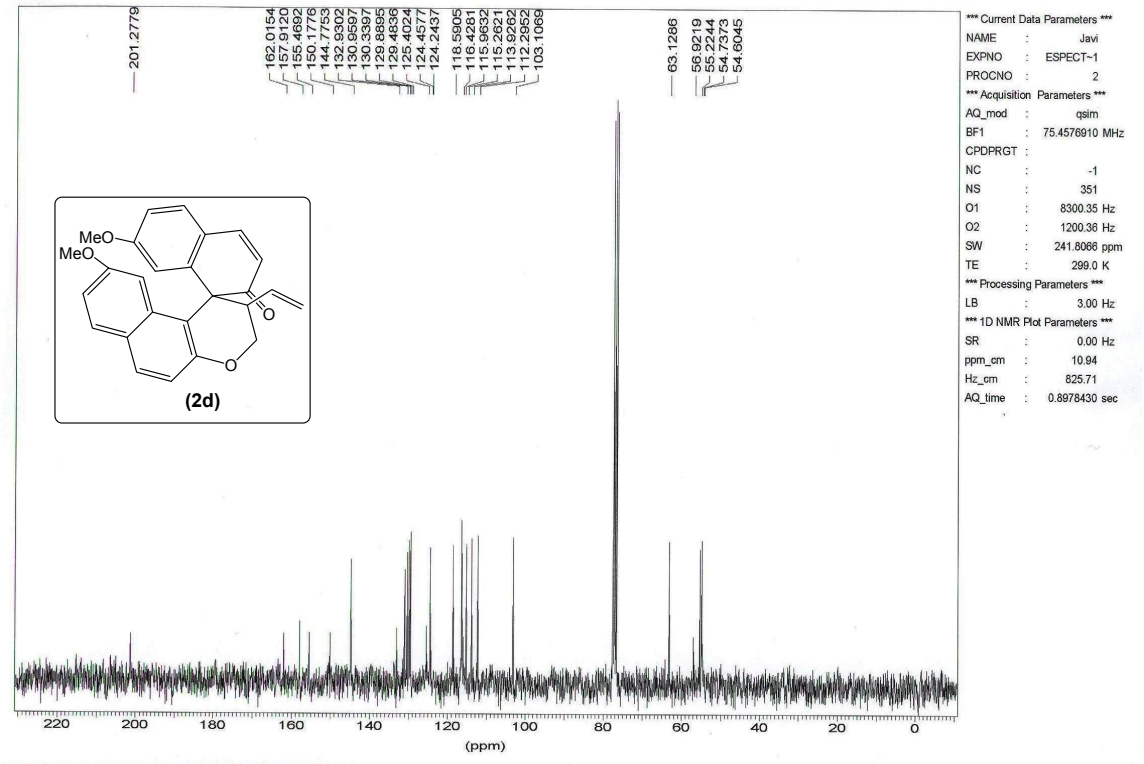
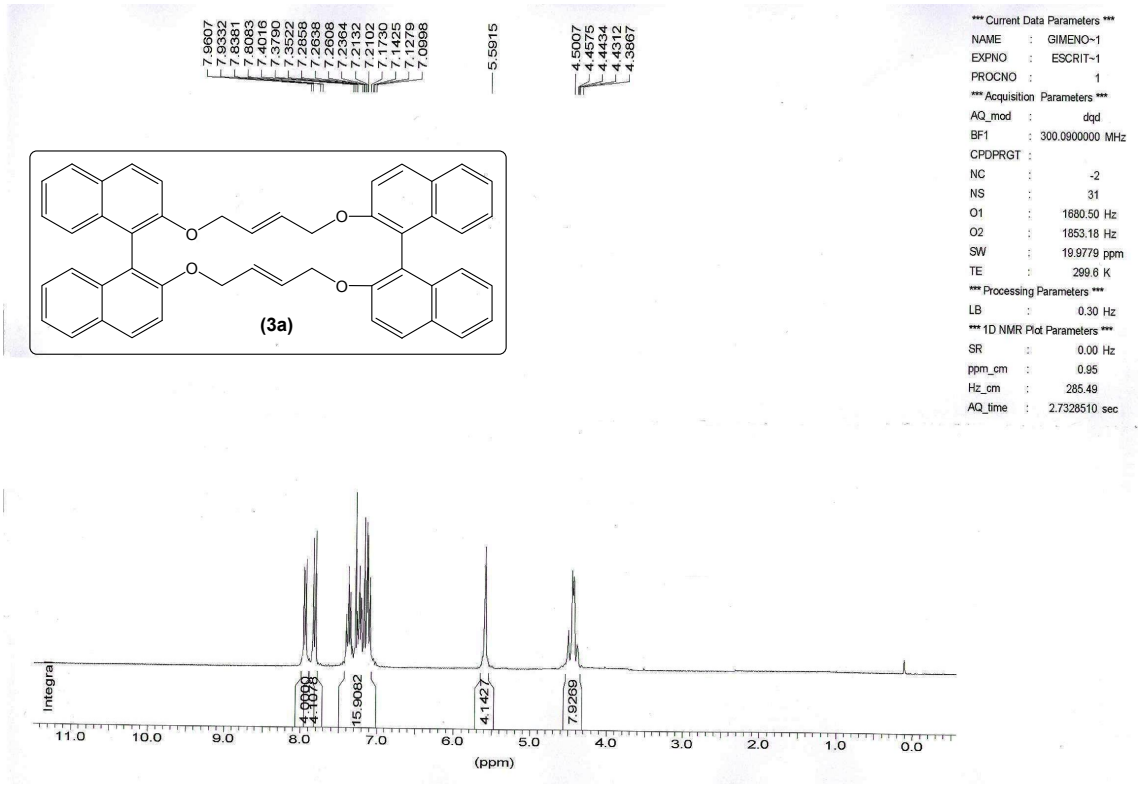
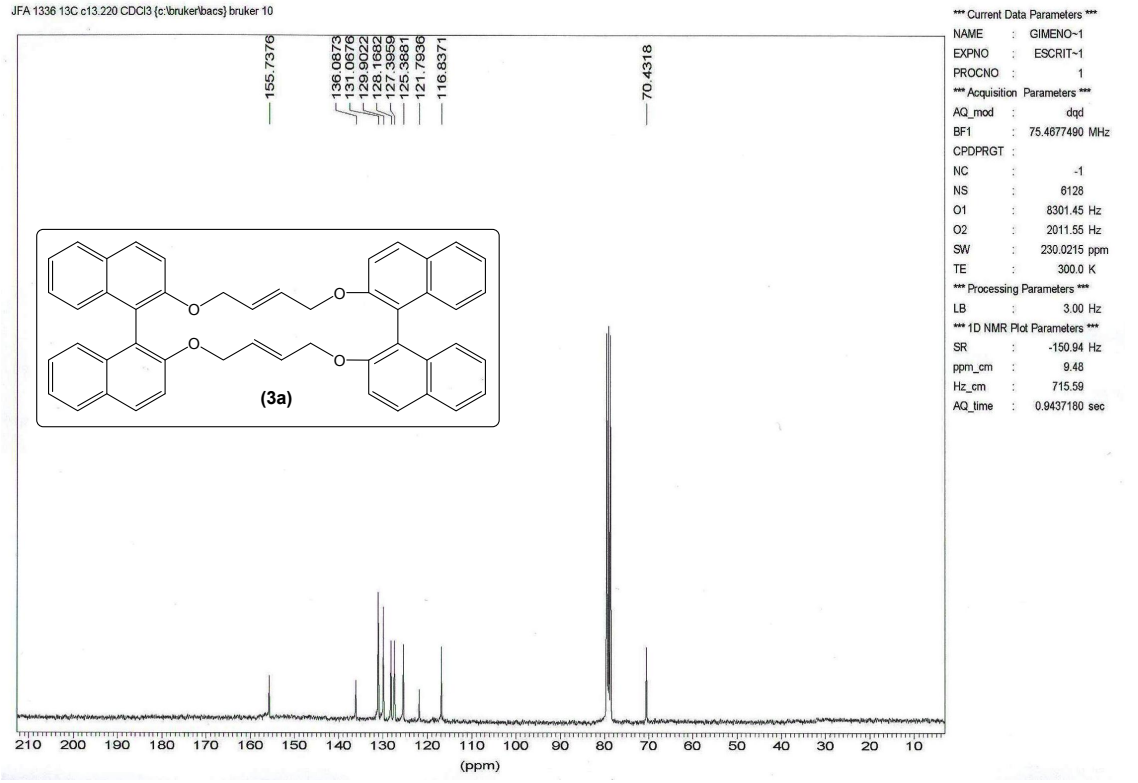
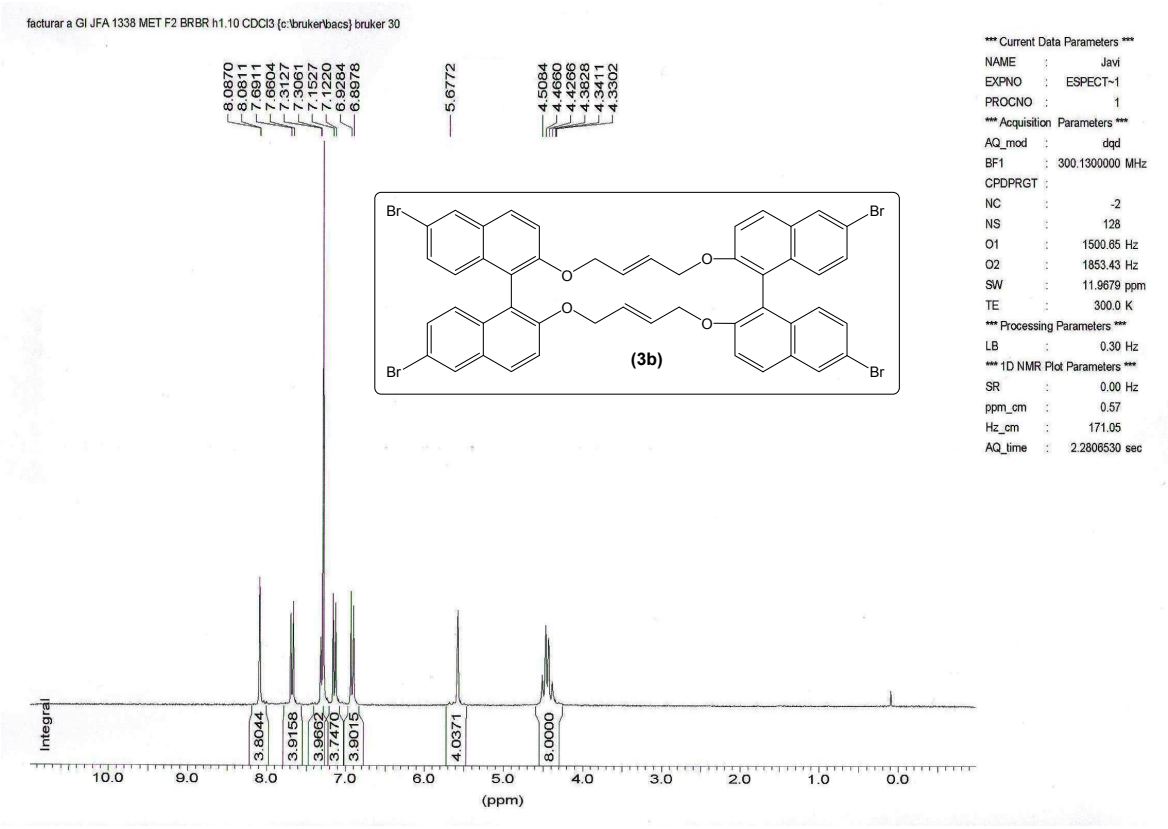
BF1 : 75.4576910 MHz
CPDPRGT :
NC : -1
NS : 227
O1 : 8300.35 Hz
O2 : 1200.38 Hz
SW : 241.8066 ppm
TE : 298.4 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
ppm_cm : 10.94
Hz_cm : 825.71
AQ_time : 0.8978430 sec

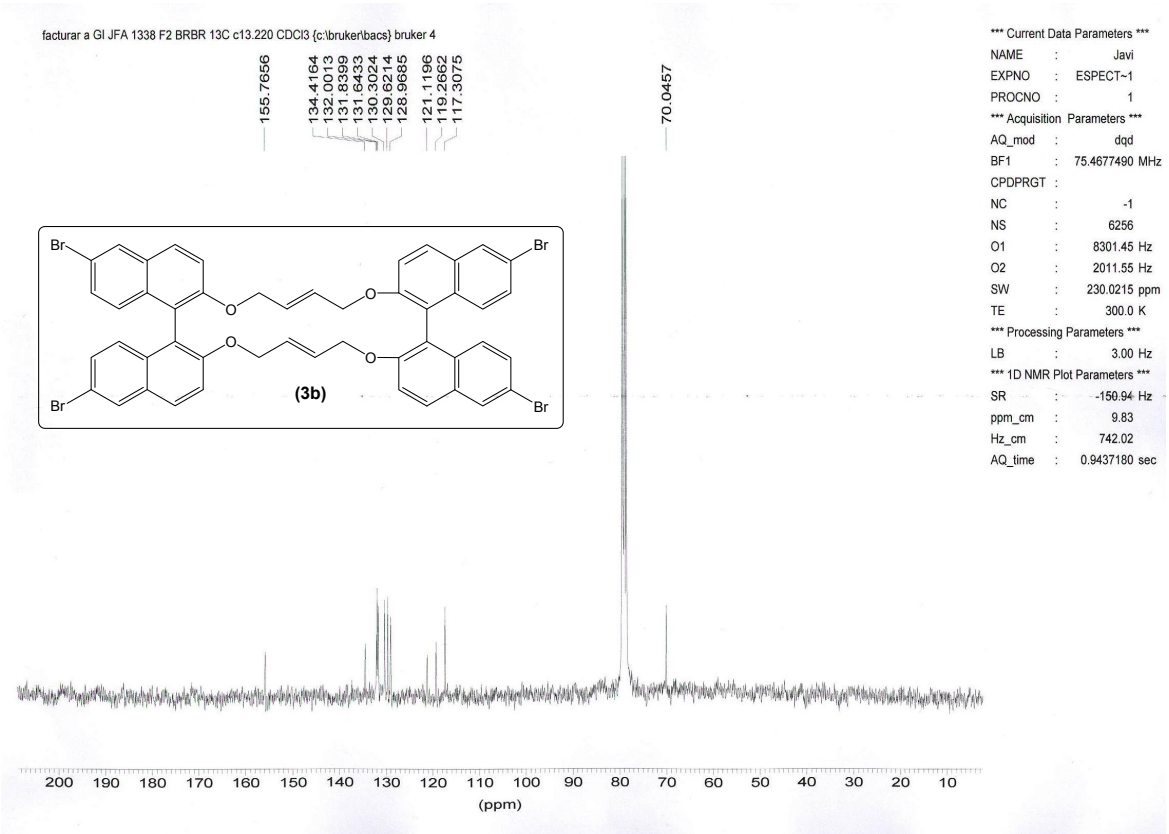

jfa 1352 F1 cdcl3 av300
7.6510
7.6260
7.6223
7.5943
7.5821
7.5528
7.3821
7.3541
7.0803
7.0504
6.8194
6.8114
6.7901
6.7828
6.3987
6.3901
6.3377
6.3048
6.0274
6.0195
5.6049
5.5756
5.5701
5.5482
5.5415
5.5189
5.5134
5.4848
4.9903
4.9873
4.9556
4.9525
4.7751
4.7714
4.7184
4.7147
4.6587
4.6215
4.5849
4.1508
4.1386
4.1142
4.1026
3.6063
3.3484
3.1064
3.0948
3.0674
3.0406
3.0290
MeO
MeO
O
O
(2d)
Integral
2.7802
0.9523
1.0003
1.7304
1.7486
0.8791
0.9065
0.9353
0.9108
0.9433
1.0162
2.9168
2.8594
1.0000
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)
*** Current Data Parameters ***
NAME : Javi
EXPNO : ESPECT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 300.0900000 MHz
CPDPRGT :
NC : -2
NS : 46
O1 : 1680.50 Hz
O2 : 1853.18 Hz
SW : 19.9779 ppm
TE : 298.8 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : -0.00 Hz
ppm_cm : 0.54
Hz_cm : 163.41
AQ_time : 2.7328510 sec


201.2779
162.0154
157.9120
155.4692
150.1776
144.7753
132.9302
130.9597
130.3397
129.8895
129.4836
125.4024
124.4577
124.2437
118.5905
116.4281
115.9632
115.2621
113.9262
112.2952
103.1069
63.1286
56.9219
55.2244
54.7373
54.6045
MeO
MeO
O
O
(2d)
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)
*** Current Data Parameters ***
NAME : Javi
EXPNO : ESPECT~1
PROCNO : 2
*** Acquisition Parameters ***
AQ_mod : qsim
BF1 : 75.4576910 MHz
CPDPRGT :
NC : -1
NS : 351
O1 : 8300.35 Hz
O2 : 1200.36 Hz
SW : 241.8066 ppm
TE : 299.0 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
ppm_cm : 10.94
Hz_cm : 825.71
AQ_time : 0.8978430 sec


7.9607
7.9332
7.8381
7.8083
7.4016
7.3790
7.3522
7.2858
7.2638
7.2608
7.2364
7.2132
7.2102
7.1730
7.1425
7.1279
7.0998
5.5915
4.5007
4.4575
4.4414
4.4312
4.3867
(3a)
*** Current Data Parameters ***
NAME : GIMENO~1
EXPNO : ESCRIT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 300.0900000 MHz
CPDPRGT :
NC : -2
NS : 31
O1 : 1680.50 Hz
O2 : 1853.18 Hz
SW : 19.9779 ppm
TE : 299.6 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
ppm_cm : 0.95
Hz_cm : 285.49
AQ_time : 2.7328510 sec
Integra
4.0000
4.1078
15.9062
4.1427
7.9269
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)


JFA 1336 13C c13.220 CDCl3 {c:\bruker\bacs} bruker 10
155.7376
136.0873
131.0676
129.9022
128.1692
127.3959
125.3881
121.7936
116.8371
70.4318
(3a)
*** Current Data Parameters ***
NAME : GIMENO~1
EXPNO : ESCRIT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 75.4677490 MHz
CPDPRGT :
NC : -1
NS : 6128
O1 : 8301.45 Hz
O2 : 2011.55 Hz
SW : 230.0215 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : -150.94 Hz
ppm_cm : 9.48
Hz_cm : 715.59
AQ_time : 0.9437180 sec
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)


facturar a GI JFA 1338 MET F2 BRBR h1.10 CDCl3 {c:\bruker\bacs} bruker 30
8.0870
8.0811
7.6911
7.6604
7.3127
7.3061
7.1527
7.1220
6.9284
6.8978
5.6772
4.5084
4.4660
4.4266
4.3828
4.3411
4.3302
Br
Br
O
O
O
O
Br
Br
(3b)
*** Current Data Parameters ***
NAME : Javi
EXPNO : ESPECT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 300.1300000 MHz
CPDPRGT :
NC : -2
NS : 128
O1 : 1500.65 Hz
O2 : 1853.43 Hz
SW : 11.9679 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
ppm_cm : 0.57
Hz_cm : 171.05
AQ_time : 2.2806530 sec
Integral
3.8044
3.9158
3.9662
3.7470
3.9015
4.0371
8.0000
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)


facturar a GI JFA 1338 F2 BRBR 13C c13.220 CDCl3 {c:\bruker\bacs} bruker 4
155.7656
134.4164
132.0013
131.8399
131.6433
130.3024
129.6214
128.9685
121.1196
119.2662
117.3075
70.0457
Br
Br
O
O
O
O
Br
Br
(3b)
*** Current Data Parameters ***
NAME : Javi
EXPNO : ESPECT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 75.4677490 MHz
CPDPRGT :
NC : -1
NS : 6256
O1 : 8301.45 Hz
O2 : 2011.55 Hz
SW : 230.0215 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : -150.94 Hz
ppm_cm : 9.83
Hz_cm : 742.02
AQ_time : 0.9437180 sec
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)