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Difluoromethyl Phenyl Sulfone as a Selective Difluoromethylene Dianion Equivalent: One-Pot Stereoselective Synthesis of *anti*-2,2-Difluoropropan-1,3-diols

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General:

Unless otherwise mentioned, all other chemicals were purchased from commercial sources. Potassium *tert*-butoxide (95 %, Aldrich) was used as received. DMF was distilled over calcium hydride, and stored over activated molecular sieve. Difluoromethyl phenyl sulphone (**2**) was prepared using known procedures.[1] Silica gel column chromatography was used to isolate the products using 60-200 mesh silica gel (from J. T. Baker). The structures and ratios of *anti*- and *syn*- isomers of diols (**3**) were determined by ^{19}F NMR (with the splitting patterns and the coupling constants) of the reaction products before isolation. It was found that the *anti*- and *syn*- isomers of diols **3** were difficult to separate efficiently with the silica-gel chromatography.[2]

^{1}H, ^{13}C and ^{19}F NMR spectra were recorded on Bruker AMX 500 and AM 360 NMR spectrometers. ^{1}H NMR chemical shifts were determined relative to internal $(CH_3)_4Si$ (TMS) at δ 0.0 or to the signal of a residual protonated solvent: $CDCl_3$ δ 7.26. ^{13}C NMR chemical shifts were determined relative to internal TMS at δ 0.0 or to the ^{13}C signal of solvent: $CDCl_3$ δ 77.0. ^{19}F NMR chemical shifts were determined relative to internal $CFCl_3$ at δ 0.0. The ^{19}F NMR yields were determined by the integration of the corresponding product peaks with respect to $PhOCF_3$ internal standard. HRMS data were recorded on a VG 7070 high-resolution mass spectrometer.

Typical procedures for ^{t}BuOK induced difluoromethylenation:

The reaction was commonly carried out in a Schlenk flask under an argon atmosphere. Into 5 ml DMF solution of difluoromethyl phenyl sulfone (**2**, 480 mg, 2.5 mmol) and benzaldehyde (800 mg, 7.5 mmol) at – 50 ^{o}C, was added 5 ml DMF solution of ^{t}BuOK (1.12 g, 10 mmol). The reaction flask was then sealed and the reaction mixture was then stirred from – 50 ^{o}C for 1h, followed by stirring at – 50 ^{o}C to room temperature overnight. The reaction mixture was quenched with 20 ml of ice water, and extracted with ether (20 ml x 3). The combined ether phase was washed with saturated NH_4Cl aqueous solution, followed by washing with water. After drying over $MgSO_4$, the ether solvent was removed under vacuum. The crude product was further purified by silica gel column (first hexanes/ethyl acetate (v/v = 9:1); then hexanes/ethyl acetate (v/v = 1:1)) to

give 541 mg *2,2-Difluoro-1,3-diphenyl-1,3-propanediol* (**3a**) as white crystalline solid,[2] yield 82 %, anti-/syn- ratio = 97/3 determined by ^{19}F NMR. For anti- isomer: ^{1}H NMR (actone-d_6): δ 5.27 (m, 4H); 7.28-7.50 (m, 10 H). ^{13}C NMR (acetone-d_6): δ 72.3 (t, J = 28.8 Hz); 121.6 (t, J = 252.6 Hz); 128.5; 128.7; 129.0; 138.9. ^{19}F NMR (acetone-d_6): δ -120.9 (pseudo t, J = 11 Hz, 2F). For syn- isomer: ^{19}F (acetone-d_6): δ -120.1 (dm, J= 249.7 Hz); -125.0 (dt, J= 249.8 Hz, 15.5 Hz). HRMS (DCI/NH_3) *m/z* calced for $C_{15}H_{18}F_2NO_2$ ($M + NH_4^+$) 282.1305, found 282.1304.

2,2-Difluoro-1,3-bis(4'-chloro-phenyl)-1,3-propanediol (**3b**): pale yellow solid, 78 % yield. For anti- isomer: ^{1}H NMR ($CDCl_3$): δ 3.08 (d, J = 4.9 Hz, 2H); 5.06 (td, J = 11.3 Hz, 4.9 Hz, 2H); 7.37 (m, 8H). ^{13}C NMR ($CDCl_3$): δ 73.00 (t, J = 29.2 Hz); 118.75 (t, J = 251.6 Hz); 128.59; 129.06; 134.29; 134.87. ^{19}F NMR ($CDCl_3$): δ -119.222 (pseudo t, J = 10.7 Hz). For syn- isomer: ^{19}F NMR ($CDCl_3$): δ -119.35 (dt, J = 256.5 Hz, 9.1 Hz); -127.67 (dt, J = 256.5 Hz, 15.2 Hz). HRMS (EI): *m/z* calcd for $C_{15}H_{12}Cl_2F_2O_2$ (M^+) 332.0182, found 332.0176.

2,2-Difluoro-1,3-bis(4'-bromo-phenyl)-1,3-propanediol (**3c**): pale yellow solid, 70 % yield. For anti- isomer: ^{1}H NMR ($CDCl_3$): δ 3.20 (b, 2H); 5.02 (t, J = 11.2 Hz, 2H); 7.30 (d, J = 8.0 Hz, 4H); 7.51 (d, J = 8.0 Hz, 4 H). ^{13}C NMR ($CDCl_3$): δ 73.05 (t, J = 28.8 Hz); 118.67 (t, J = 252.6 Hz); 123.09; 129.36; 131.53; 134.84. ^{19}F NMR ($CDCl_3$): δ -119.14 (pseudo t, J = 10.7 Hz). For syn- isomer: ^{19}F NMR ($CDCl_3$): δ -118.96 (dt, J = 254.8 Hz, 9.2 Hz); -127.48 (dt, J = 254.8 Hz, 15.3 Hz). For anti- isomer: ^{19}F NMR ($CDCl_3$): δ -119.1 (dt, J = 254.8 Hz, 9.2 Hz); -127.5 (dt, J = 254.8 Hz, 15.2 Hz). HRMS (EI): *m/z* calcd for $C_{15}H_{12}Br_2F_2O_2$ (M^+) 421.9152, found 421.9156.

2,2-Difluoro-1,3-bis(4'-methoxy-phenyl)-1,3-propanediol (**3d**): pale yellow sticky liquid, 52 % yield. For anti- isomer: ^{1}H NMR ($CDCl_3$): δ 3.78 (s, 6H); 3.81 (b, 2H); 4.94 (t, J = 11.8 Hz, 2H); 6.86 (d, J = 8.5 Hz, 4 H); 7.30 (d, J = 8.4 Hz, 4H). ^{19}F NMR ($CDCl_3$): δ – 119.6 (pseudo t, J = 13.1 Hz). ^{13}C NMR ($CDCl_3$): δ 55.2; 73.1 (t, J = 28.8 Hz); 113.62; 119.3 (t, J = 251.3 Hz); 128.3; 129.0; 159.7. For Syn- isomer: ^{19}F NMR ($CDCl_3$): δ -119.4 (dt, J = 253.5 Hz, 9.2 Hz); -128.1 (dt, J = 253.3 Hz, 15.1 Hz). HRMS (EI): *m/z* calcd for $C_{15}H_{12}Br_2F_2O_2$ (M^+) 421.9152, found 421.9156.

2,2-Difluoro-1,3-bis(2'-naphthyl)-1,3-propanediol (**3e**): pale yellow solid, 69 % yield. For anti- isomer: ^{1}H NMR ($CDCl_3$): δ 3.33 (b, 2H); 5.25 (t, J = 11.3 Hz, 2H); 7.50~7.91 (m, 10 H). ^{13}C NMR ($CDCl_3$): δ 74.09 (t, J = 28.9 Hz); 119.46 (t, J = 251.7 Hz); 125.08; 126.30; 126.48; 127.34; 127.68; 128.10; 128.17; 132.97; 133.51. ^{19}F NMR ($CDCl_3$): δ -118.35 (pseudo t, J = 11.3 Hz). For syn- isomer: ^{19}F NMR ($CDCl_3$): δ -117.7 (dm, J = 250 Hz); -127.o (dt, J= 251 Hz, 15 Hz). HRMS (EI): *m/z* calcd for $C_{23}H_{18}F_2O_2$ (M^+) 364.1275, found 364.1277.

2,2-Difluoro-1,3-bis(4'-biphenl)-1,3-propanediol (**3f**): pale yellow solid, 75 % yield. For anti- isomer: ^{1}H NMR: δ 3.48 (b, 2H); 5.17 (t, J = 11.2 Hz, 2 H); 7.36~7.60 (m, 18 H). ^{13}C NMR ($CDCl_3$): δ 73.54 (t, J = 28.9 Hz); 119.32 (t, J = 252.1 Hz); 127.06; 127.23; 127.67; 128.19; 130.01; 135.04; 140.53; 141.71. ^{19}F NMR ($CDCl_3$): δ -119.04 (pseudo t, J = 11.2 Hz). For syn- isomer: ^{19}F NMR ($CDCl_3$): δ -119.3 (dm, J = 251 Hz); -127.1 (dt, J= 251 Hz, 15.1 Hz). HRMS (EI): *m/z* calcd for $C_{27}H_{22}F_2O_2$ (M^+) 416.1588, found 416.1570.

2,2-Difluoro-1,3-bis(2'-furanyl)-1,3-propanediol (**3g**): viscous liquid, 63 % yield. For anti- isomer: ^{1}H NMR ($CDCl_3$): δ 3.50 (b, 2H); 5.17 (t, J = 11.0 Hz, 2H); 6.39 (m, 2H); 6.45 (mb, 2H); 7.43 (m, 2H). ^{13}C NMR ($CDCl_3$): δ 67.38 (t, J = 29.1 Hz); 109.80; 110.60; 18.84 (t, J = 253.4 Hz); 143.13; 149.02. ^{19}F NMR ($CDCl_3$): δ -120.95 (pseudo t, J = 11.0 Hz). For syn- isomer: ^{19}F NMR ($CDCl_3$): δ -120.5 (dm, J= 253 Hz); -125.3 (dt, J = 253 Hz, 14 Hz). HRMS (EI): *m/z* calcd for $C_{11}H_{10}F_2O_4$ (M^+) 244.0547, found 244.0550.

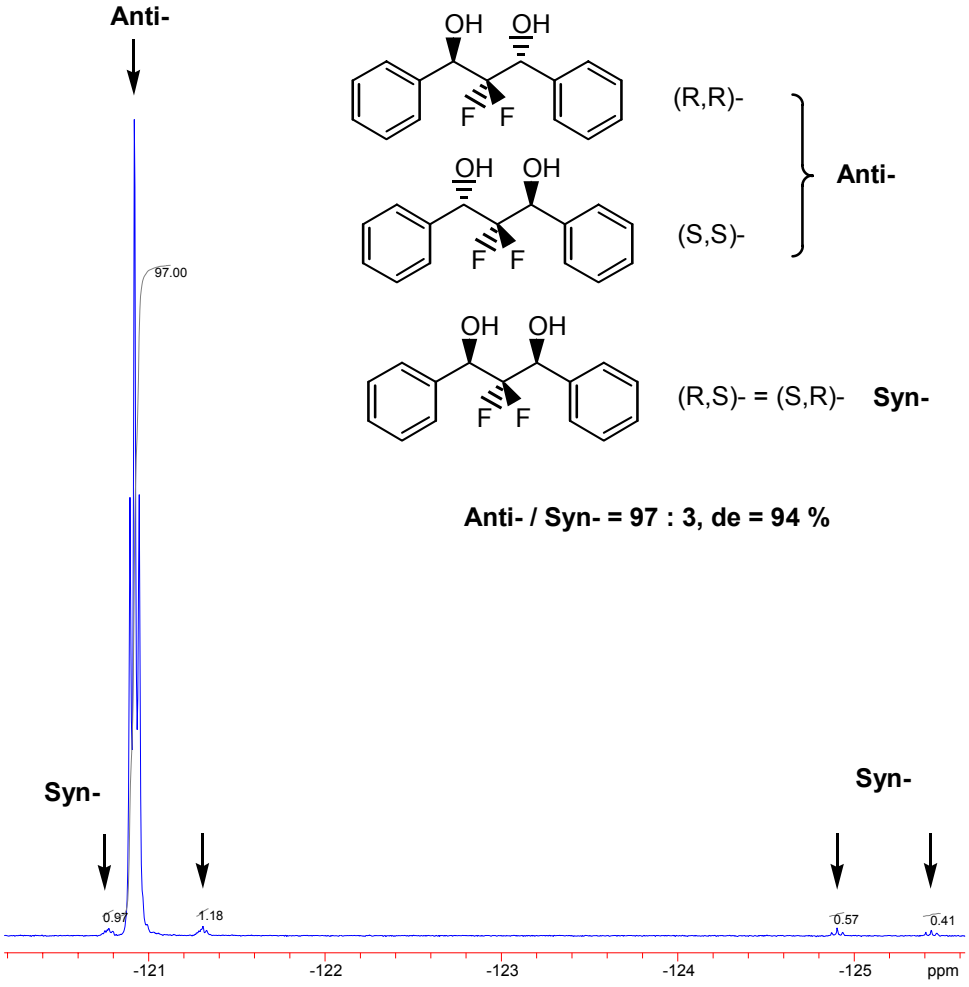
2,2-Difluoro-1-pehneyl-3-(4'-chlorophenyl)-1,3-propanediol (**20**): white solid, 76 % yield. For anti- isomer: ^{1}H NMR ($CDCl_3$): δ 3.22 (m, 2H); 5.04 (td, J = 11.0 Hz, 2H); 7.36 (m, 9H). ^{13}C NMR ($CDCl_3$): δ 72.9 (t, J = 29 Hz); 73.8 (t, J = 29 Hz); 118.7 (t, J = 259 Hz); 127.6; 128.6; 129.0; 134.3; 134.8. ^{19}F NMR ($CDCl_3$): δ -118.2~-120.1 (m).

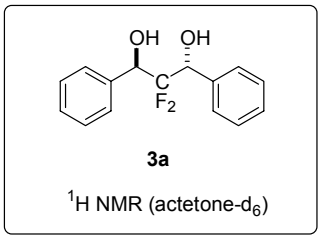
Refernces:

1. (a) J. Hine, J. Porter, *J. Am. Chem. Soc.* **1960**, *82*, 6178. (b) J. Hine, J. Porter, *J. Am. Chem. Soc.* **1957**, *79*, 5493. (c) P. Stahly, *J. Fluorine Chem.* **1989**, *43*, 53-66.

2. M. Kuroboshi, T. Ishihara, *Bull. Chem. Soc. Jpn.* **1990**, *63*, 1185-1190.

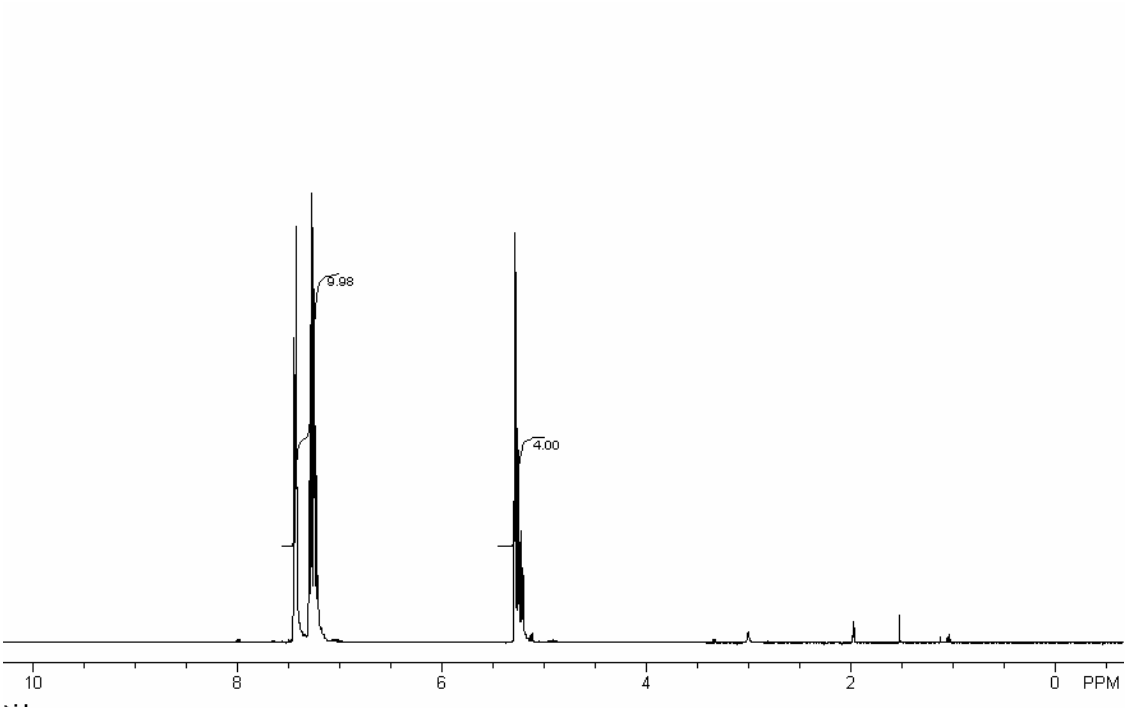
Example of determination of *anti*/*syn* ratio of 2,2-difluoropropan-1,3-diols (reaction products mixture) by ^{19}F NMR:

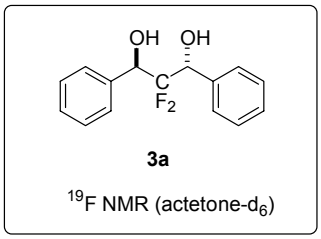




3a

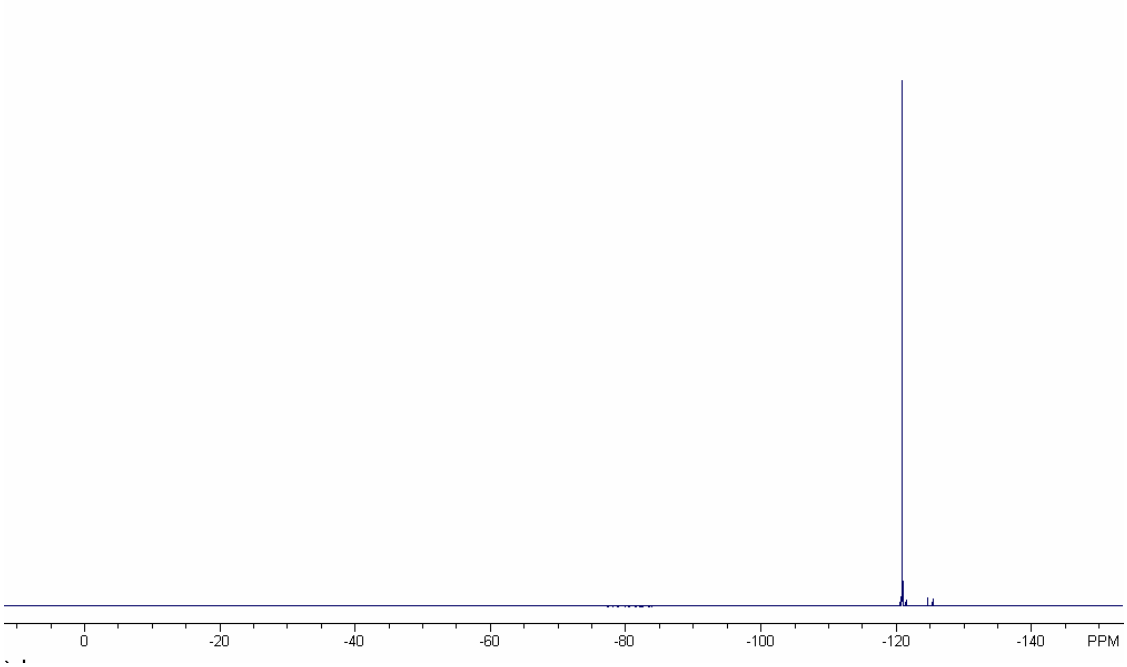
^{1}H NMR (actetone-d_6)





3a

^{19}F NMR (actetone-d_6)



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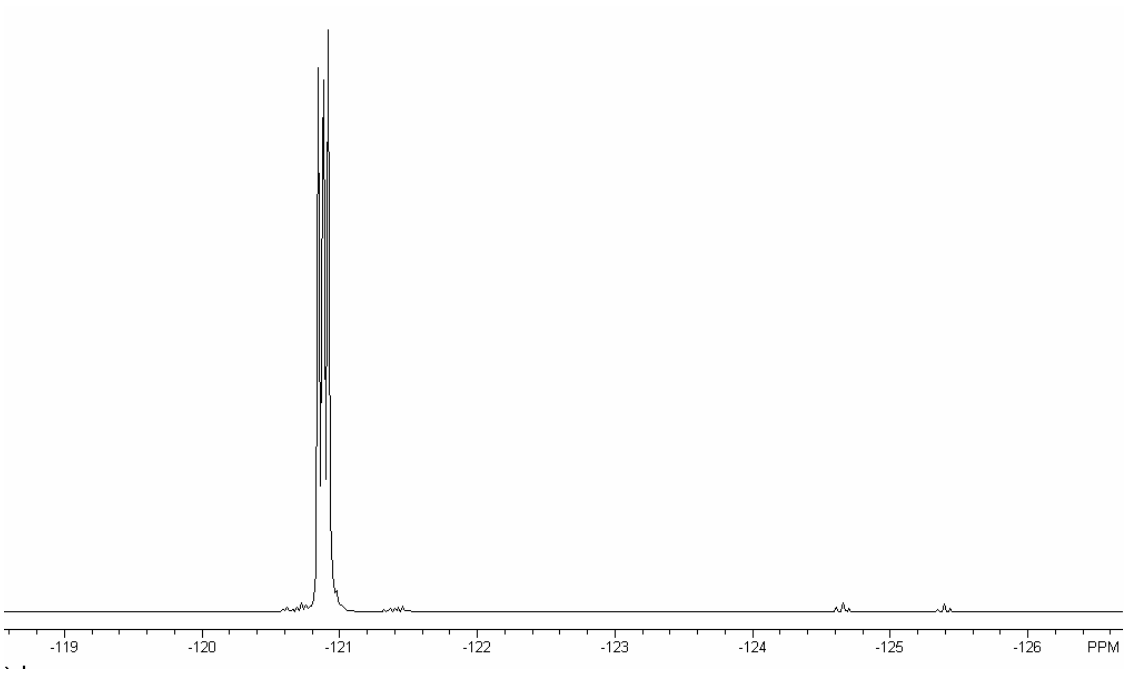
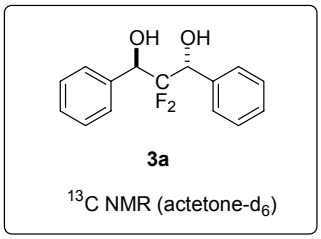
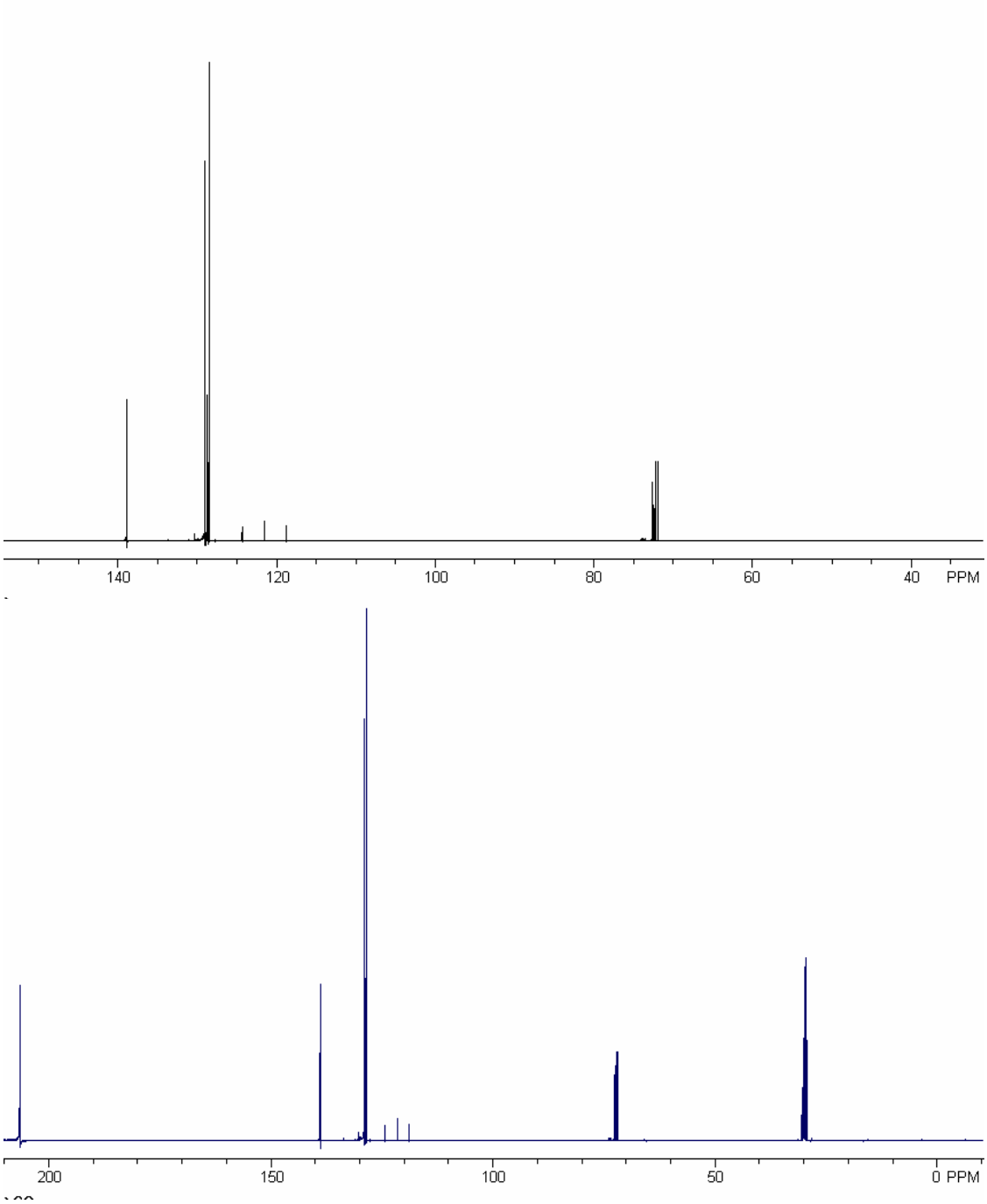
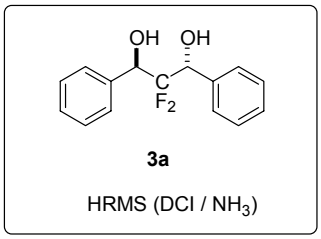




3a

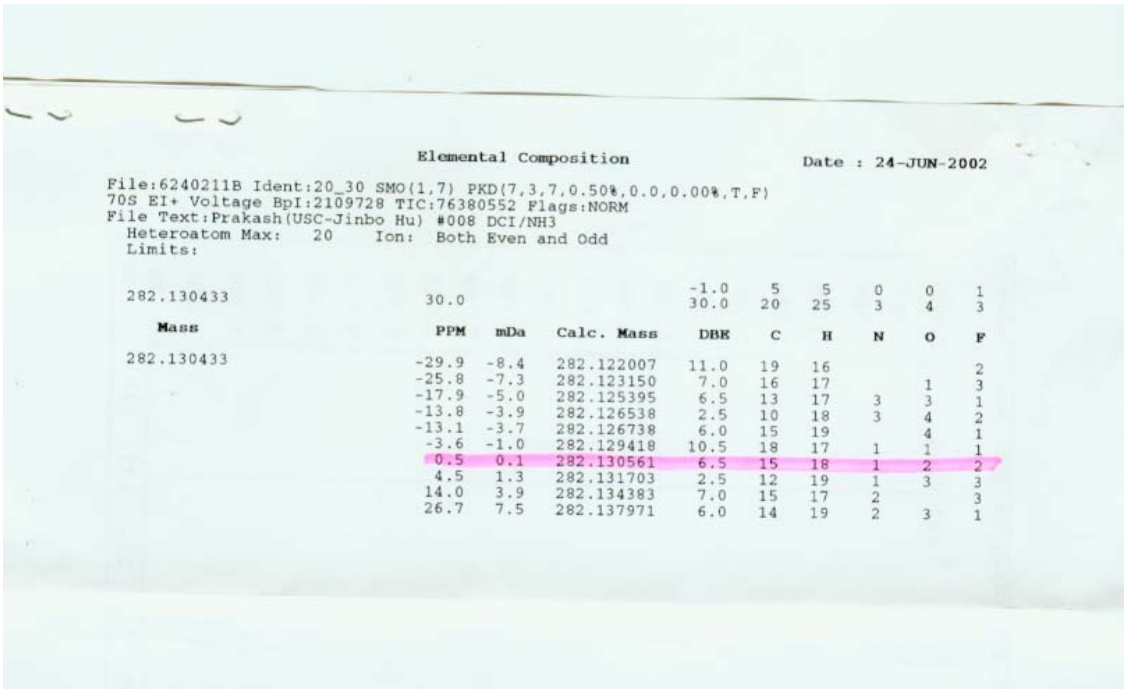
^{13}C NMR (actetone-d_6)





3a

HRMS (DCI / NH_3)



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Heteroatom Max: 20 Ion: Both Even and Odd
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	-13.1	-3.7	282.126738	6.0	15	19		4	1
	-3.6	-1.0	282.129418	10.5	18	17	1	1	1
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	4.5	1.3	282.131703	2.5	12	19	1	3	3
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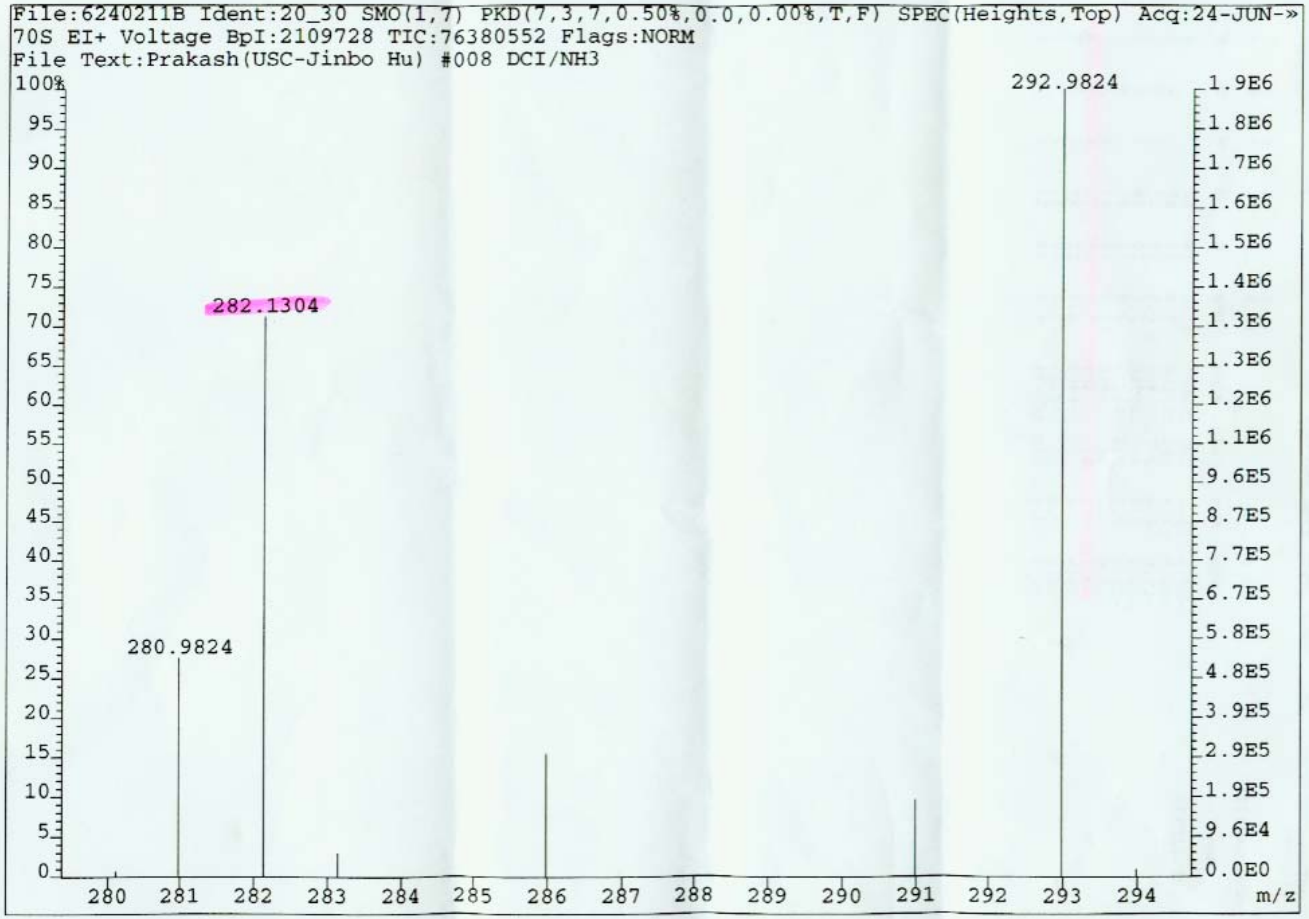
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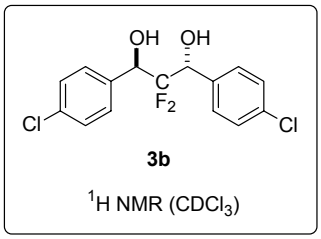
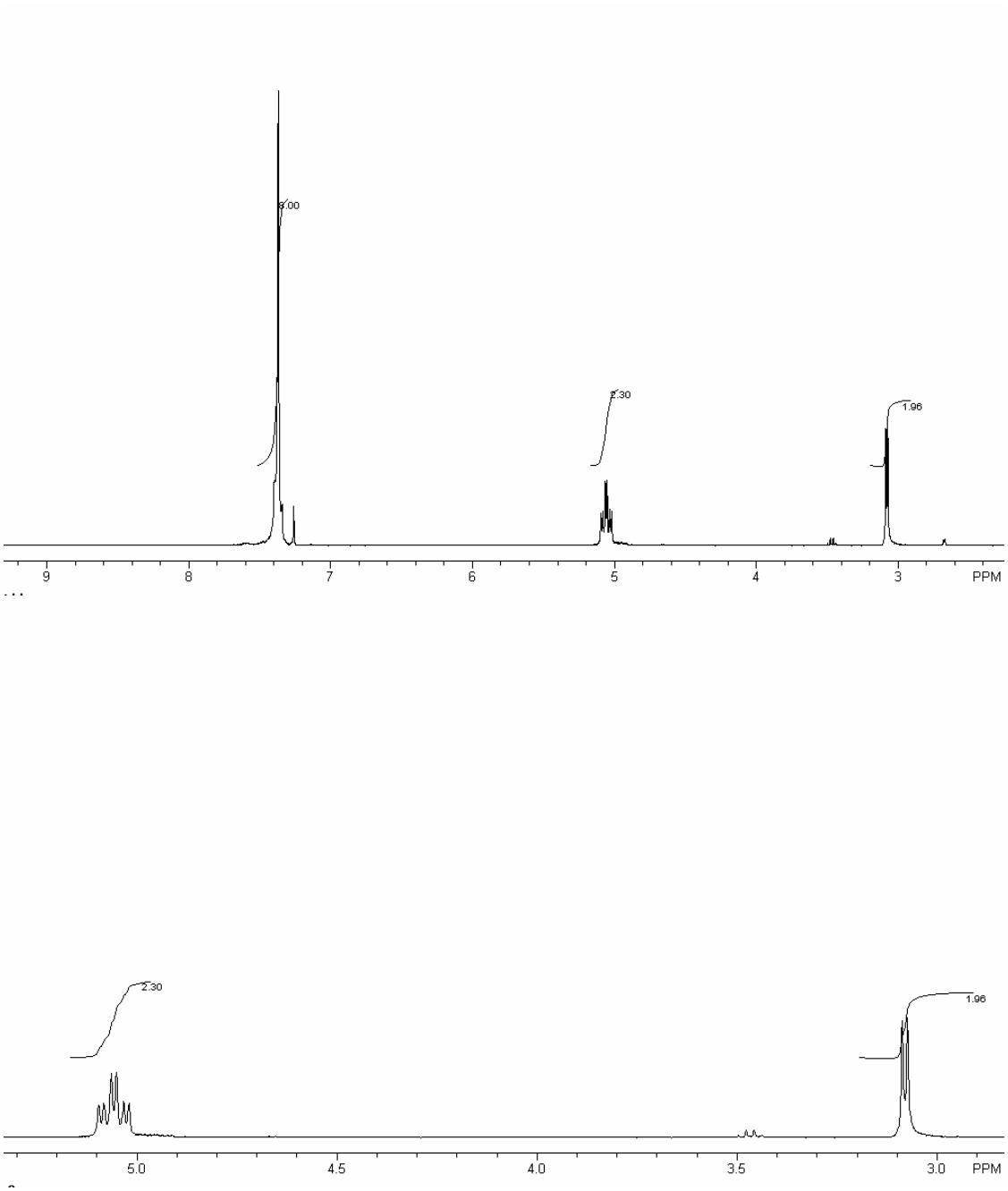
m/z

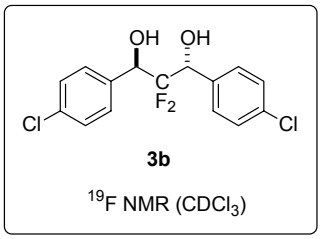
S11



3b

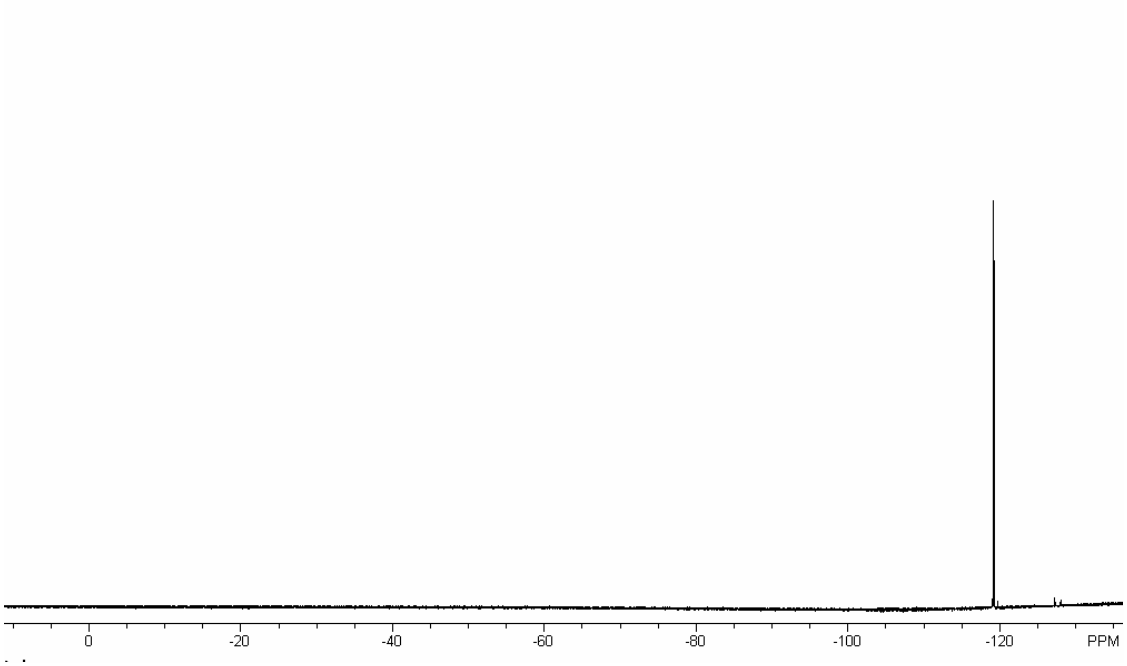
^{1}H NMR ($CDCl_3$)





3b

^{19}F NMR ($CDCl_3$)



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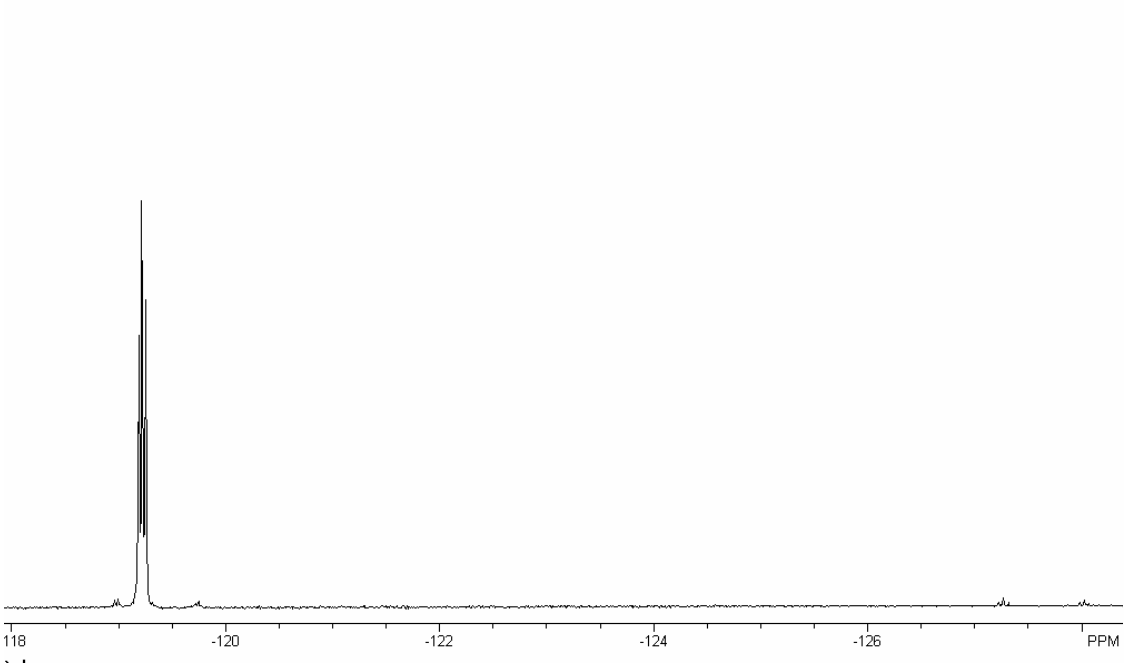
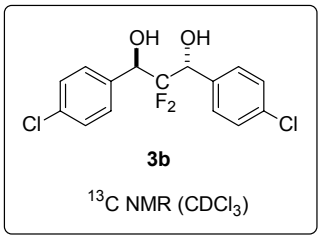
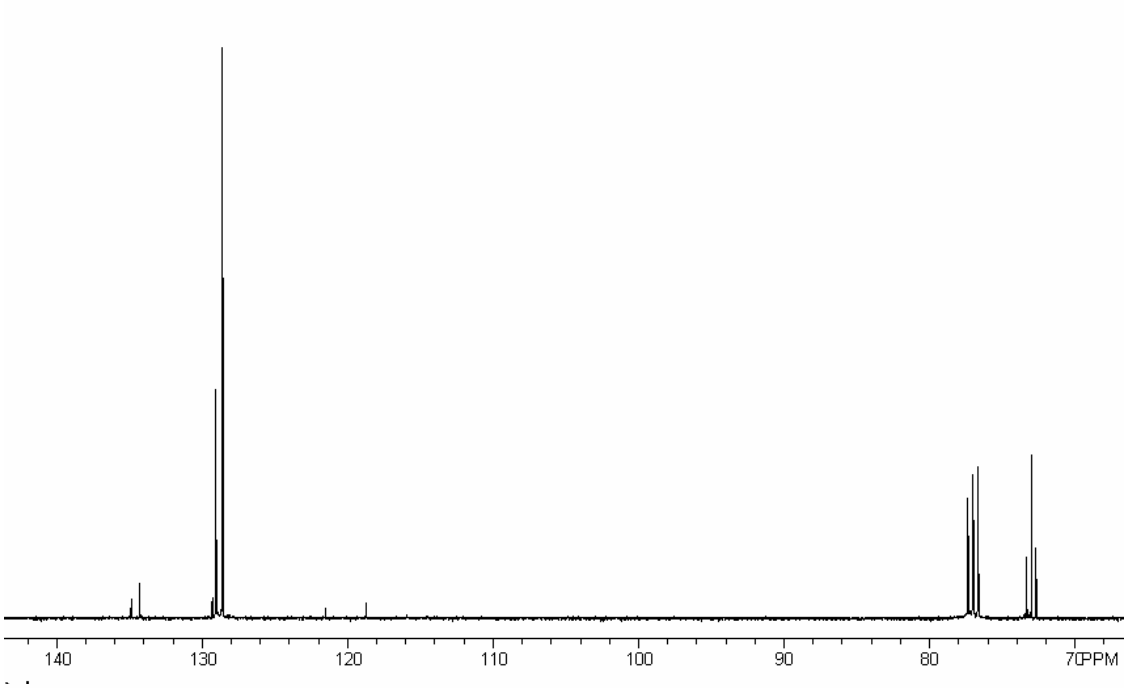
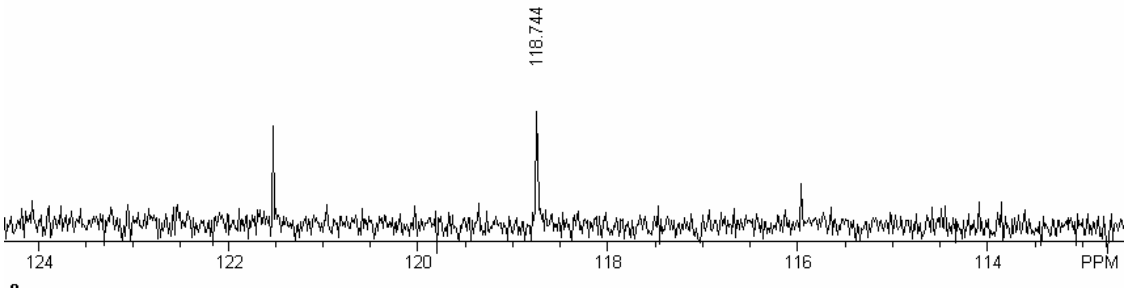


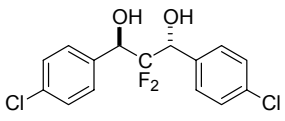


3b

^{13}C NMR ($CDCl_3$)







3b

HRMS (EI)

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336.489	100.0		15.0			20.0	15	1	12	2	2	2	2

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333.020276	0.5	20	0.0	4.0	1.3	333.021597	8.0	14	1	12	2	2	2	
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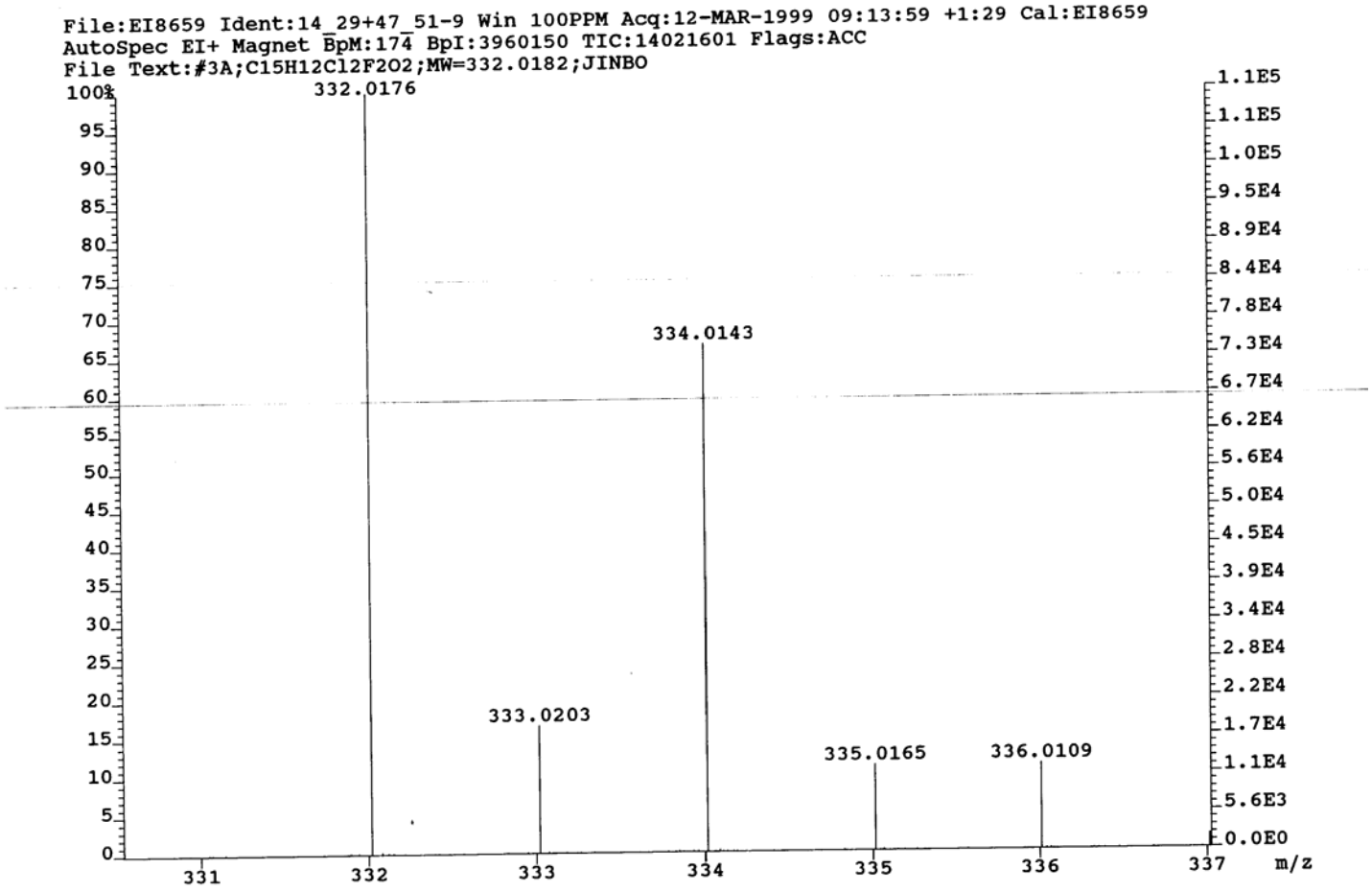
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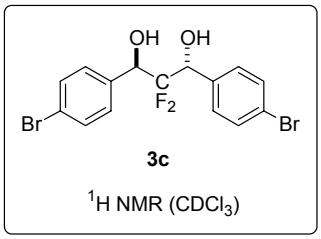
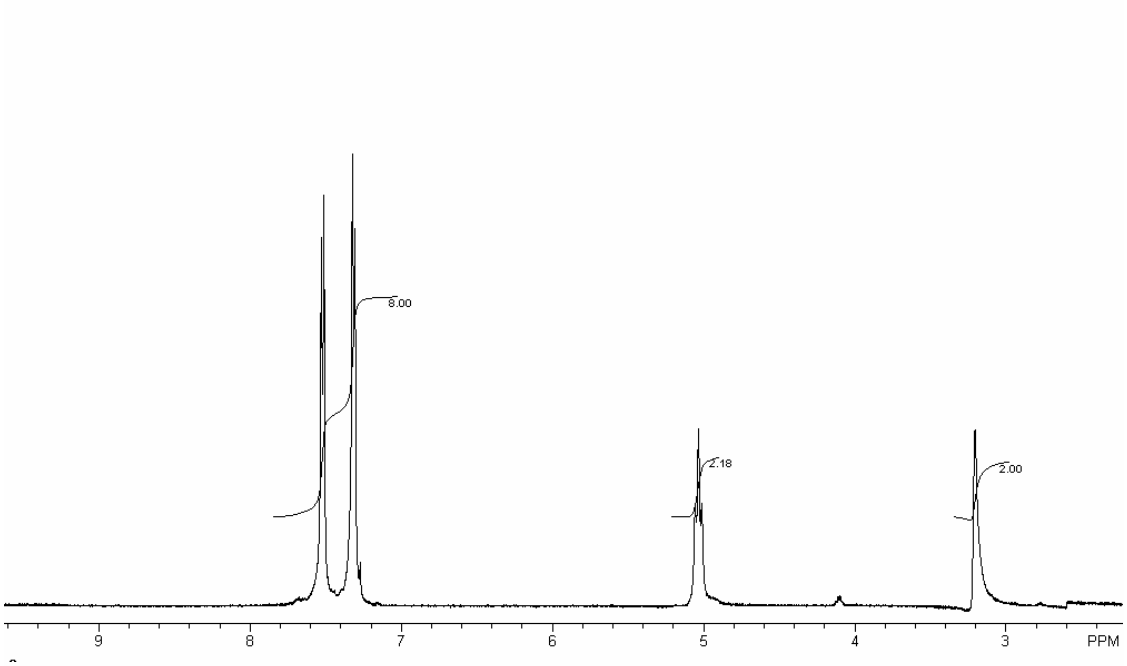
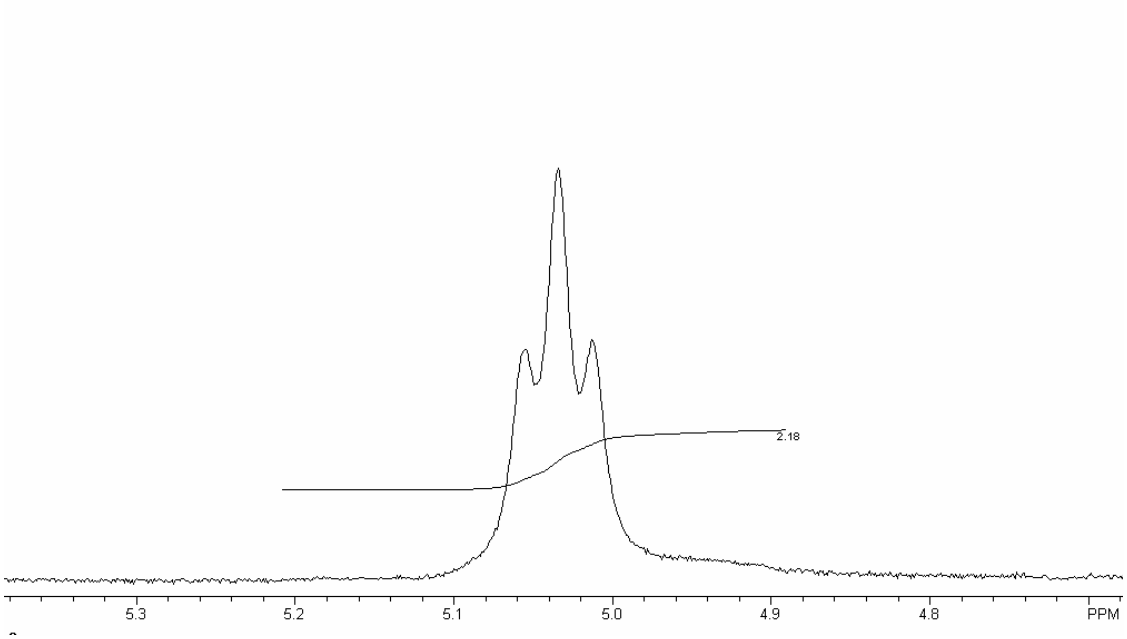
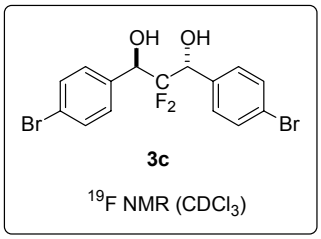


3c

^{1}H NMR ($CDCl_3$)

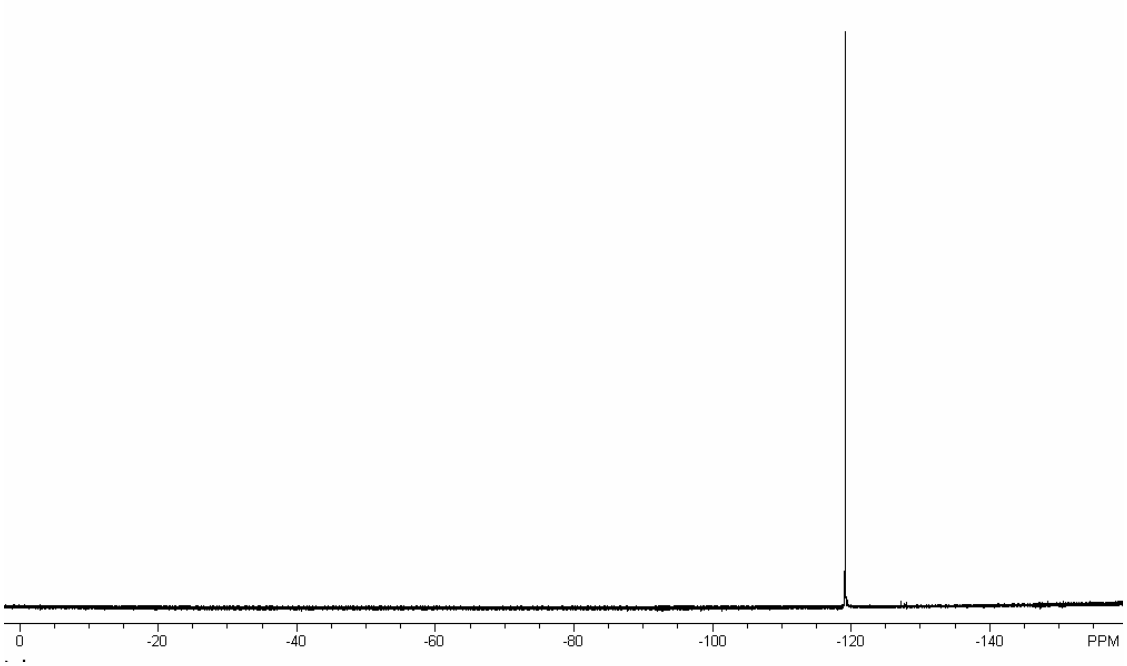


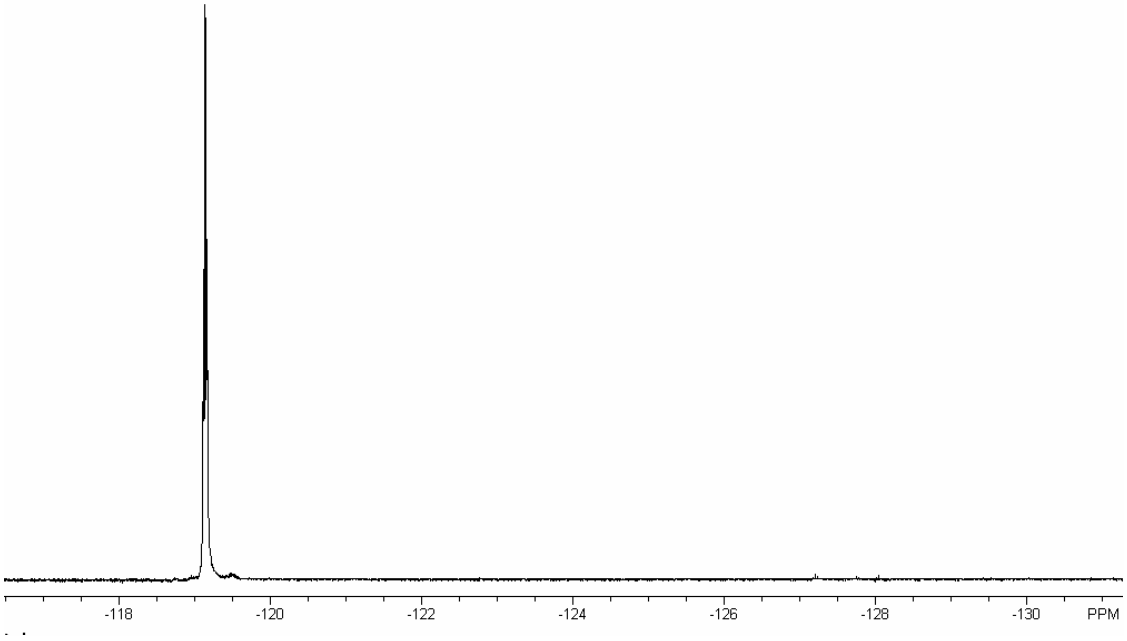


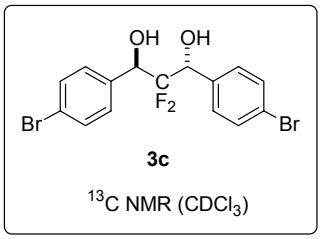


3c

^{19}F NMR ($CDCl_3$)

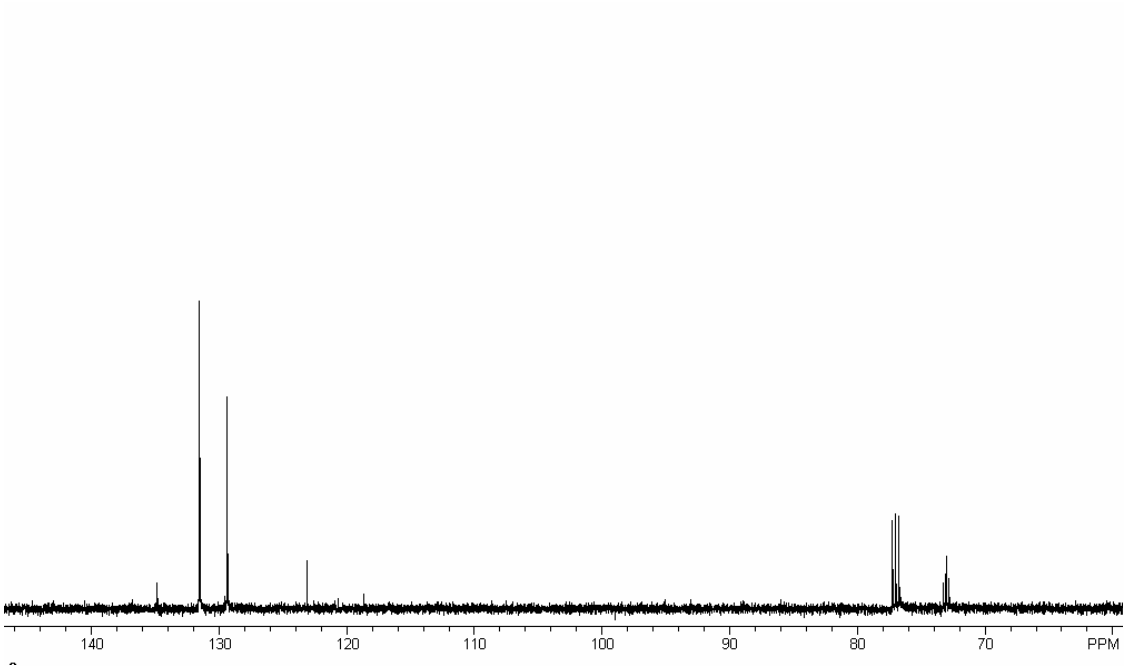


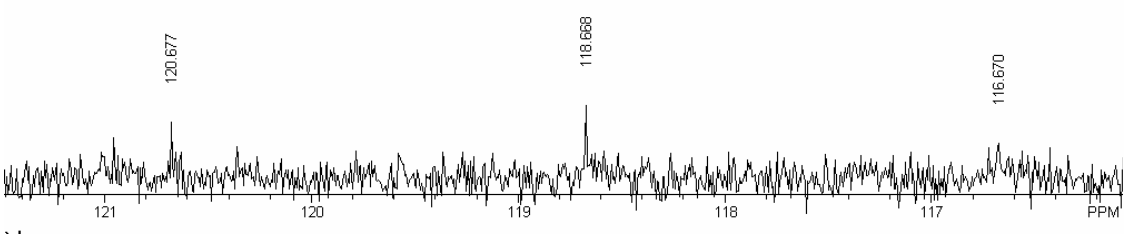


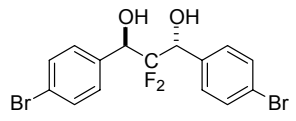


3c

^{13}C NMR ($CDCl_3$)







3c

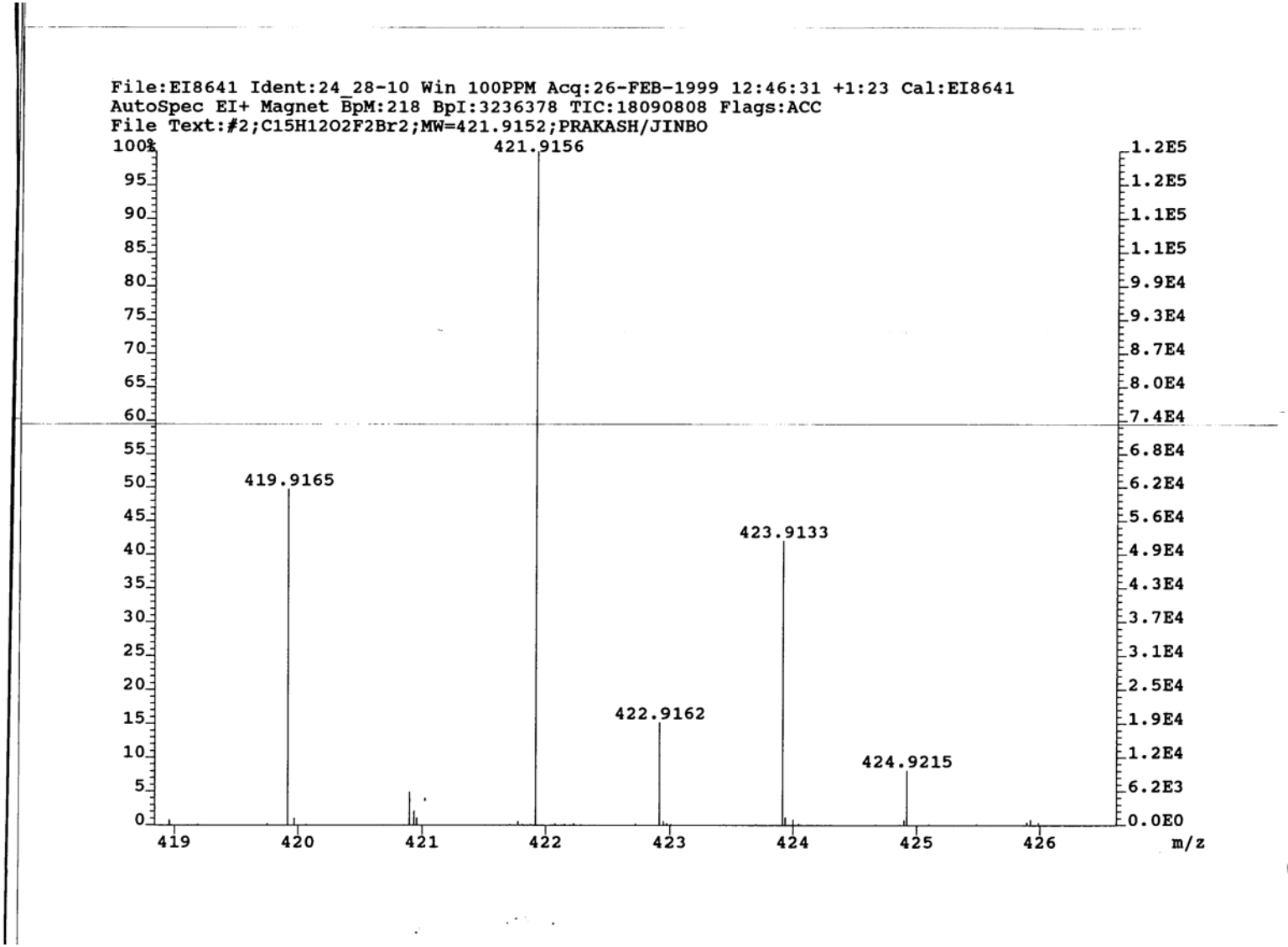
HRMS (EI)

Elemental Composition Date : 26-FEB-1999

File:EI8641 Ident:24_28-10 Win 100PPM Acq:26-FEB-1999 12:46:31 +1:23 Cal:EI8641
AutoSpec EI+ Magnet BpM:218 BpI:3236378 TIC:18090808 Flags:ACC
File Text:#2;C15H12O2F2Br2;MW=421.9152;PRAKASH/JINBO
Heteroatom Max: 20 Ion: Both Even and Odd
Limits:

418.845	0.0					-0.5	14	0	12	2	2	0	0
426.620	100.0		20.0			20.0	15	1	12	2	2	2	2

Mass	%RA	Pks	Std	PPM	mDa	Calc. Mass	DBE	C	13C	H	O	F	Br	81Br
424.921450	0.3	5	12.8	-11.7	-5.0	424.916471	8.0	14	1	12	2	2		2
423.913311	1.6	4	15.1	-0.5	-0.2	423.913116	8.0	15		12	2	2		2
422.916167	0.6	5	16.6	5.6	2.4	422.918517	8.0	14	1	12	2	2	1	1
421.915599	3.8	5	22.2	-1.0	-0.4	421.915162	8.0	15		12	2	2	1	1
419.916539	1.9	5	0.0	1.6	0.7	419.917208	8.0	15		12	2	2	2	


File:EI8641 Ident:24_28-10 Win 100PPM Acq:26-FEB-1999 12:46:31 +1:23 Cal:EI8641
AutoSpec EI+ Magnet BpM:218 BpI:3236378 TIC:18090808 Flags:ACC
File Text:#2;C15H12O2F2Br2;MW=421.9152;PRAKASH/JINBO
421.9156
419.9165
423.9133
422.9162
424.9215
100%
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
1.2E5
1.2E5
1.1E5
1.1E5
9.9E4
9.3E4
8.7E4
8.0E4
7.4E4
6.8E4
6.2E4
5.6E4
4.9E4
4.3E4
3.7E4
3.1E4
2.5E4
1.9E4
1.2E4
6.2E3
0.0E0
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421
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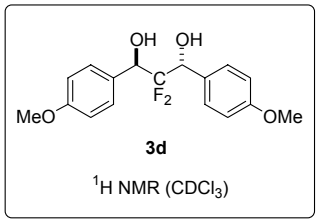
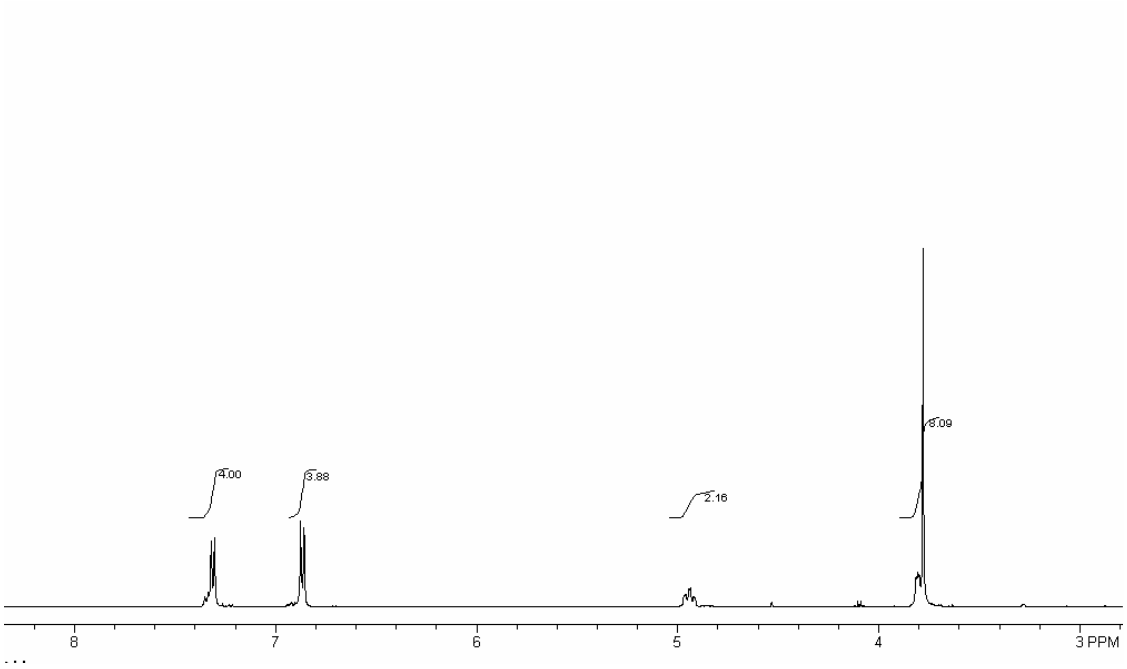
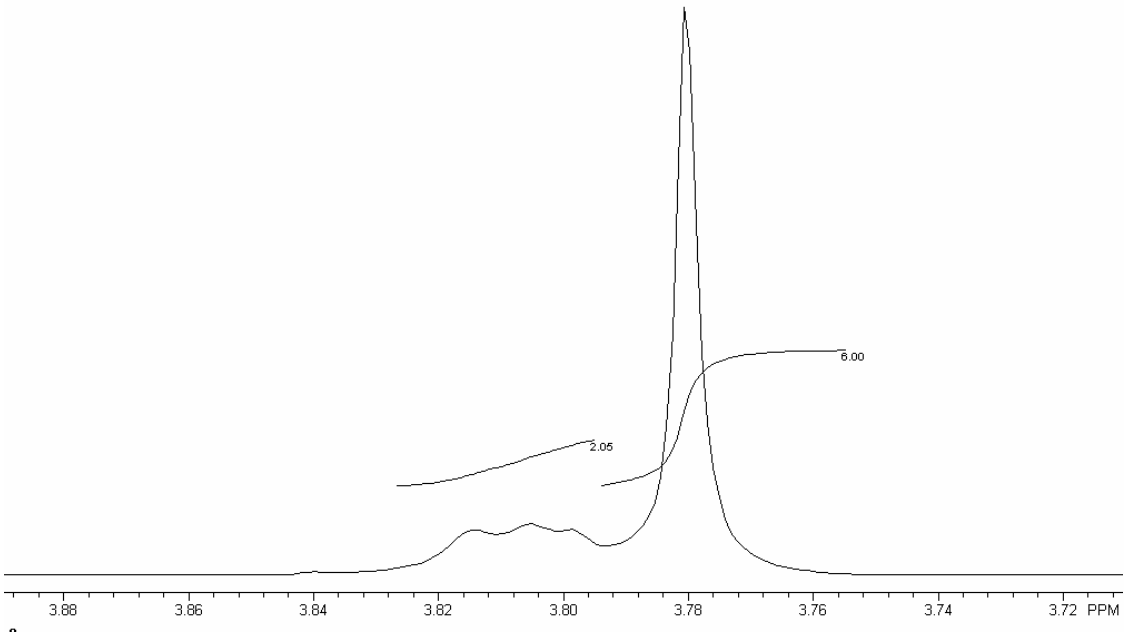
424
425
426
m/z

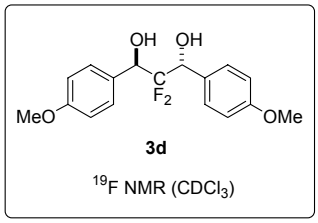


3d

^{1}H NMR ($CDCl_3$)

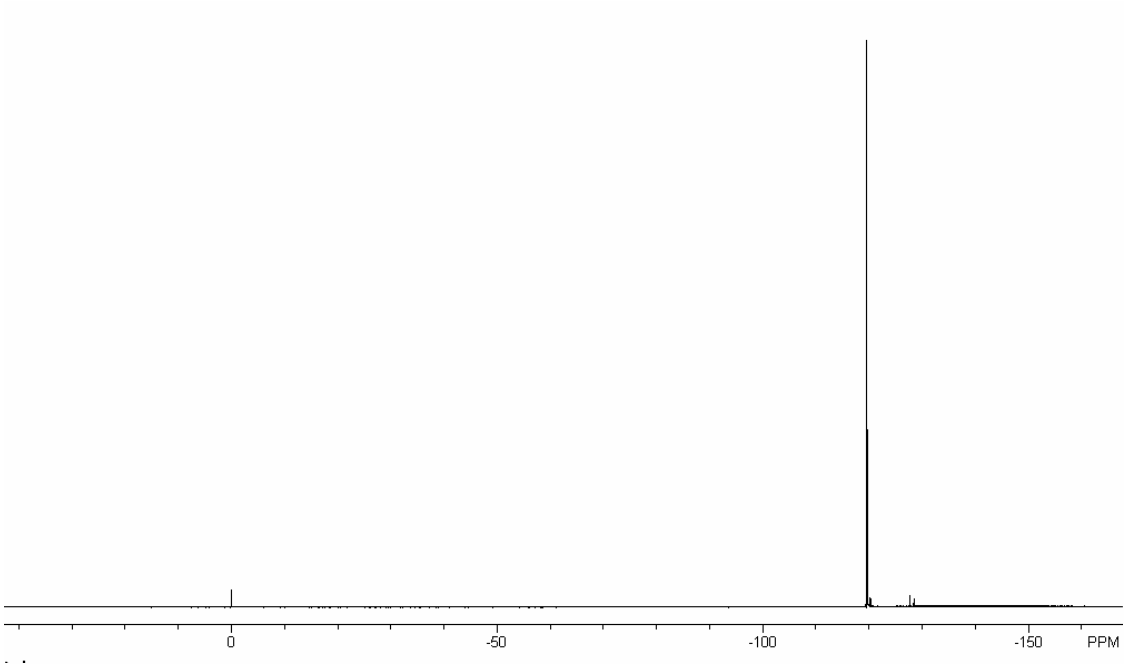


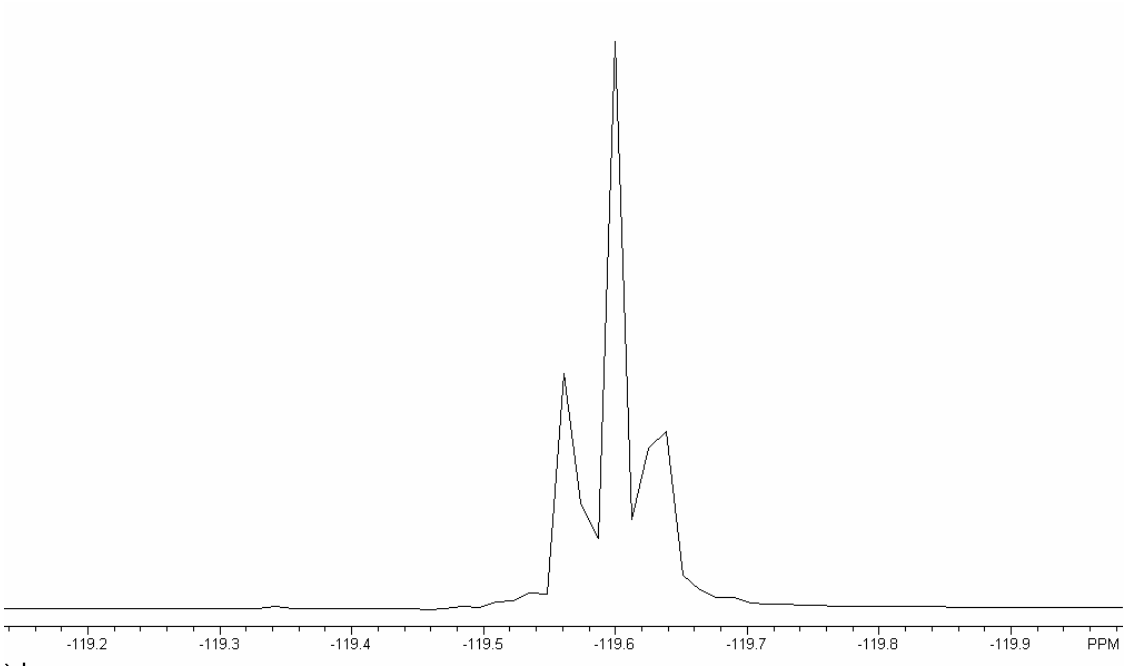


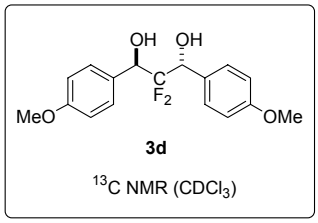


3d

^{19}F NMR ($CDCl_3$)

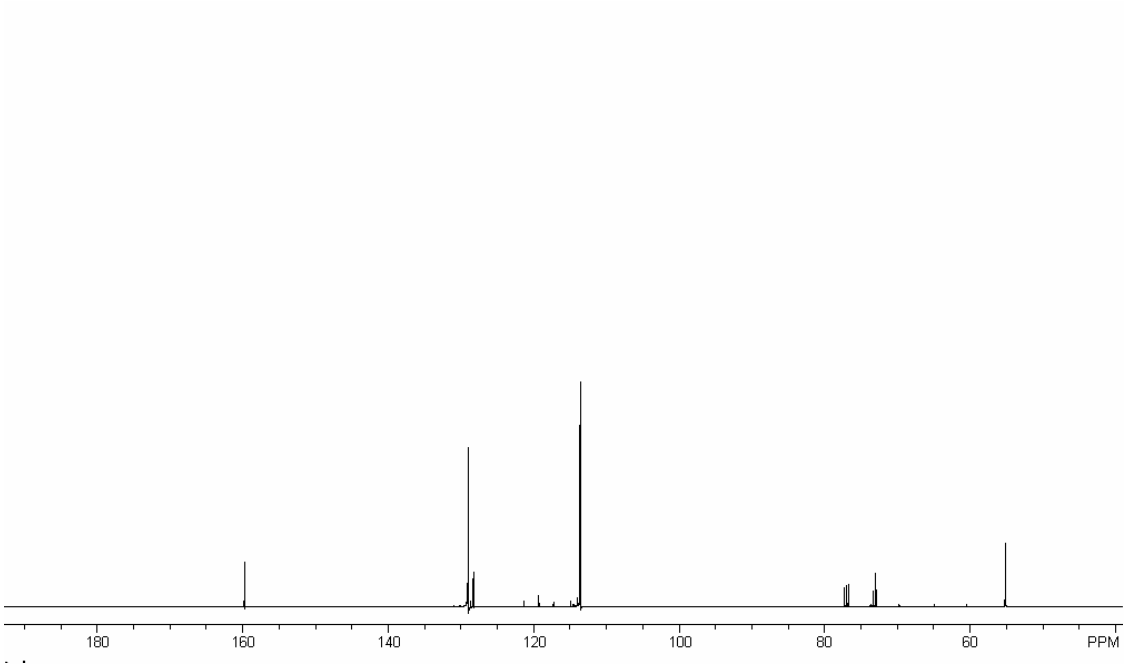


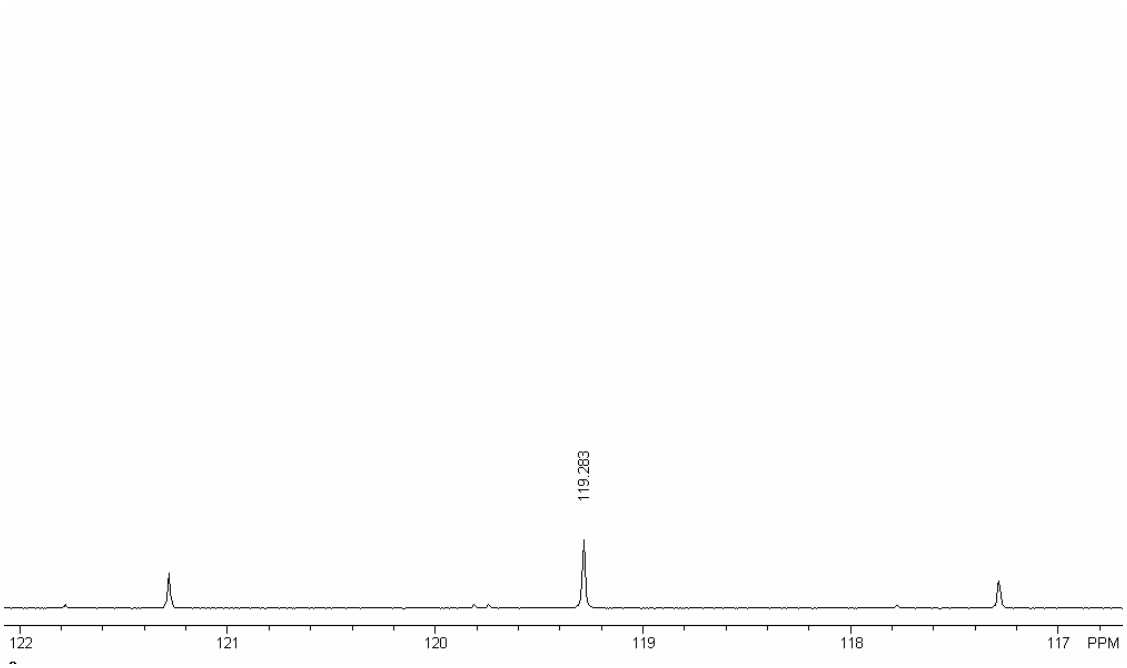


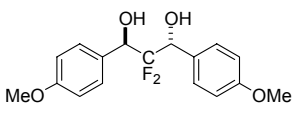


3d

^{13}C NMR ($CDCl_3$)







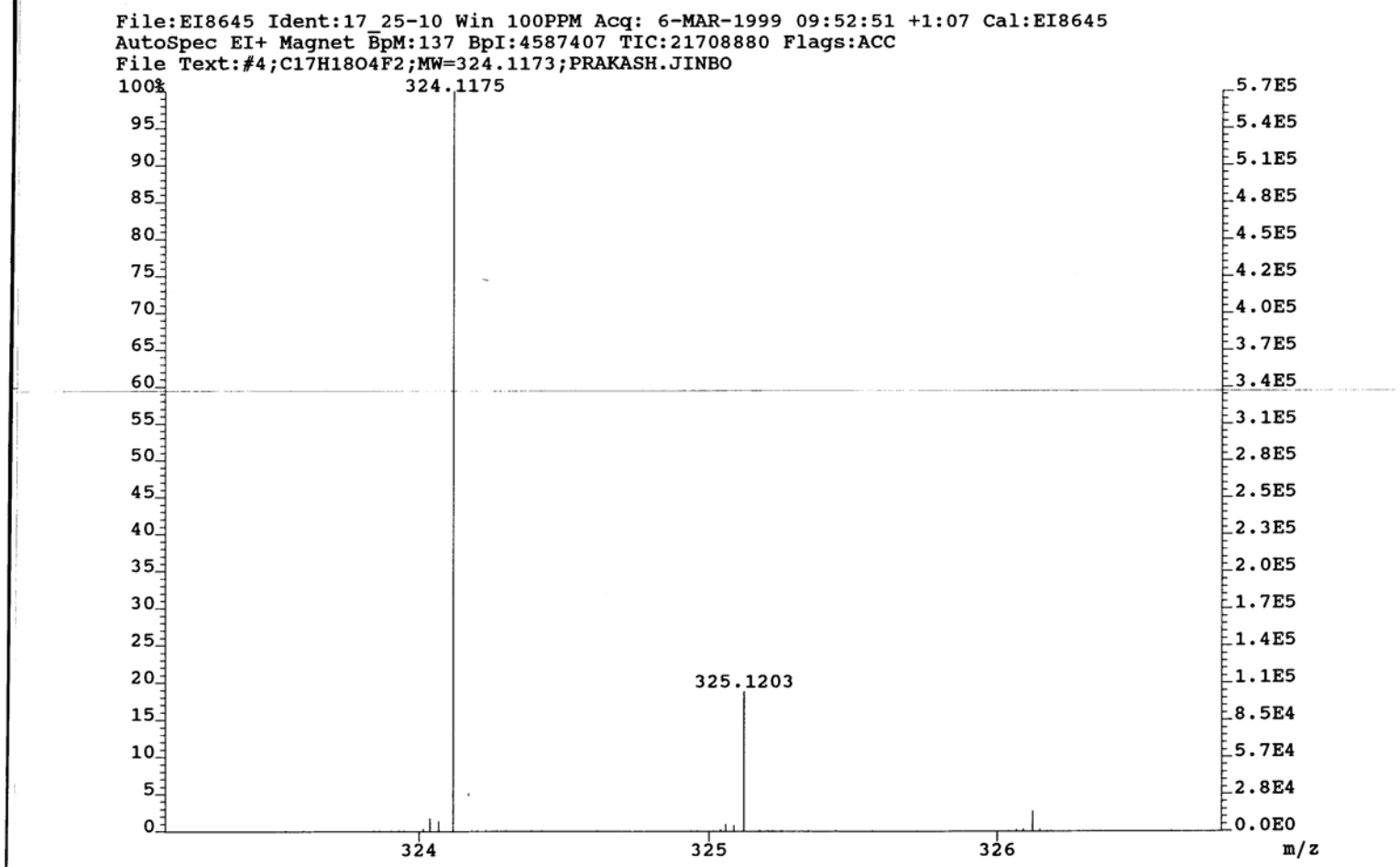
3d

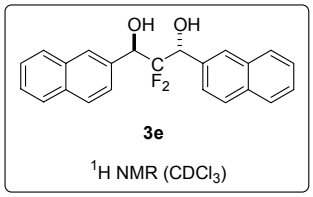
HRMS (EI)

Elemental Composition Date : 6-MAR-1999

File:EI8645 Ident:17_25-10 Win 100PPM Acq: 6-MAR-1999 09:52:51 +1:07 Cal:EI8645
AutoSpec EI+ Magnet BpM:137 BpI:4587407 TIC:21708880 Flags:ACC
File Text:#4;C17H18O4F2;MW=324.1173;PRAKASH.JINBO
Heteroatom Max: 20 Ion: Both Even and Odd
Limits:

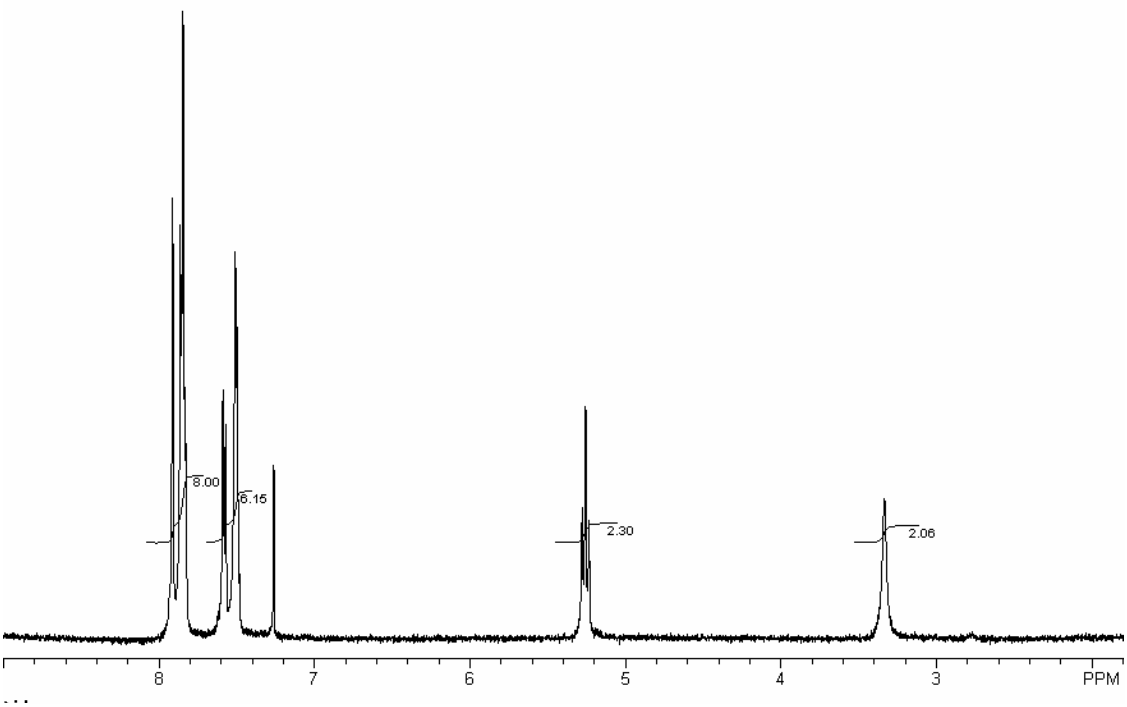
323.123	0.0					-0.5	16	0	18	4	2	
326.776	100.0		20.0			20.0	17	1	18	4	2	
Mass	%RA	Pks	Std	PPM	mDa	Calc. Mass	DBE	C	13C	H	O	F
325.120292	2.3	9	9.0	1.2	0.4	325.120671	8.0	16	1	18	4	2
324.117470	12.3	9	8.5	-0.5	-0.2	324.117316	8.0	17		18	4	2

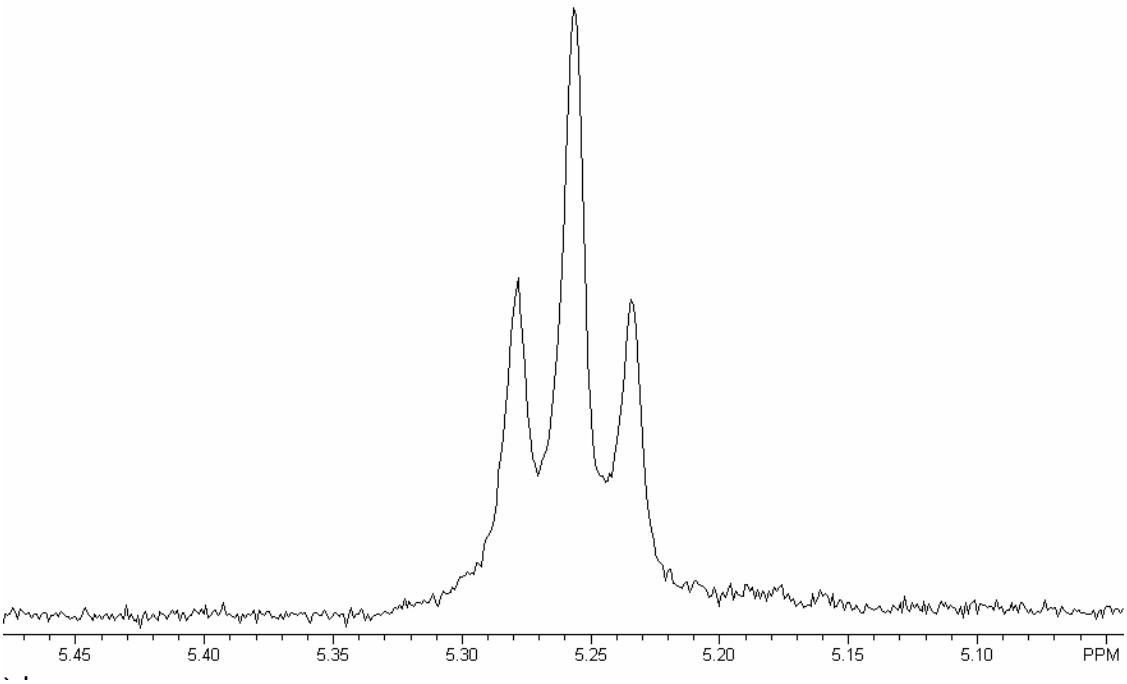

File:EI8645 Ident:17_25-10 Win 100PPM Acq: 6-MAR-1999 09:52:51 +1:07 Cal:EI8645
AutoSpec EI+ Magnet BpM:137 BpI:4587407 TIC:21708880 Flags:ACC
File Text:#4;C17H18O4F2;MW=324.1173;PRAKASH.JINBO
100%
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
324.1175
325.1203
5.7E5
5.4E5
5.1E5
4.8E5
4.5E5
4.2E5
4.0E5
3.7E5
3.4E5
3.1E5
2.8E5
2.5E5
2.3E5
2.0E5
1.7E5
1.4E5
1.1E5
8.5E4
5.7E4
2.8E4
0.0E0
324
325
326
m/z

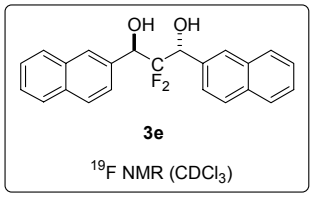


3e

^{1}H NMR ($CDCl_3$)

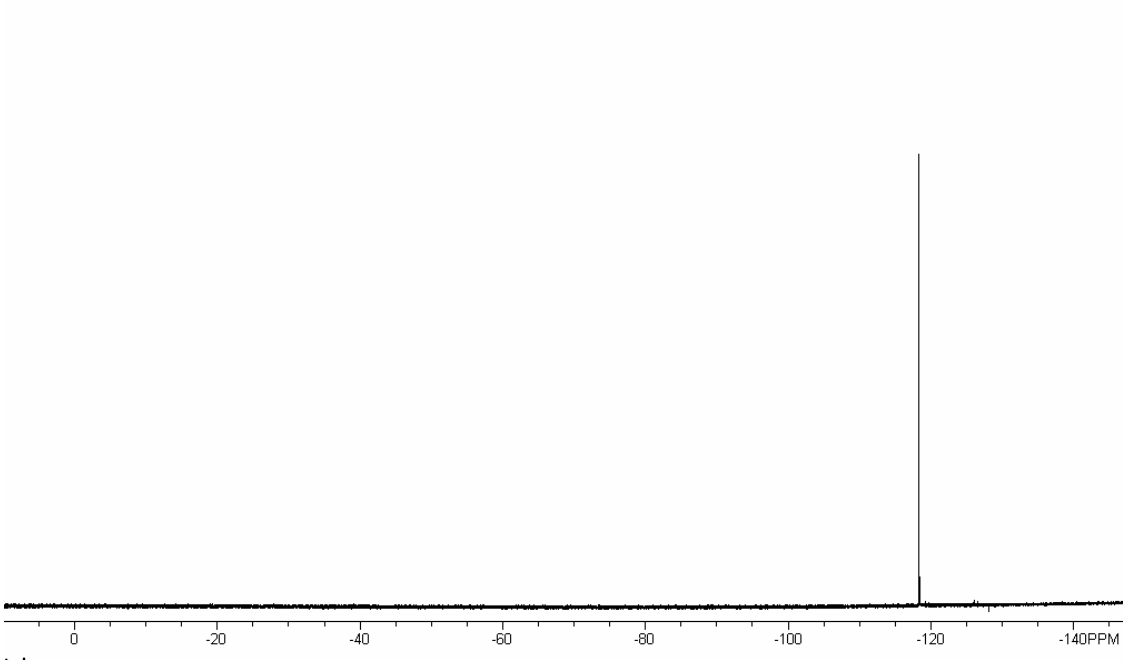


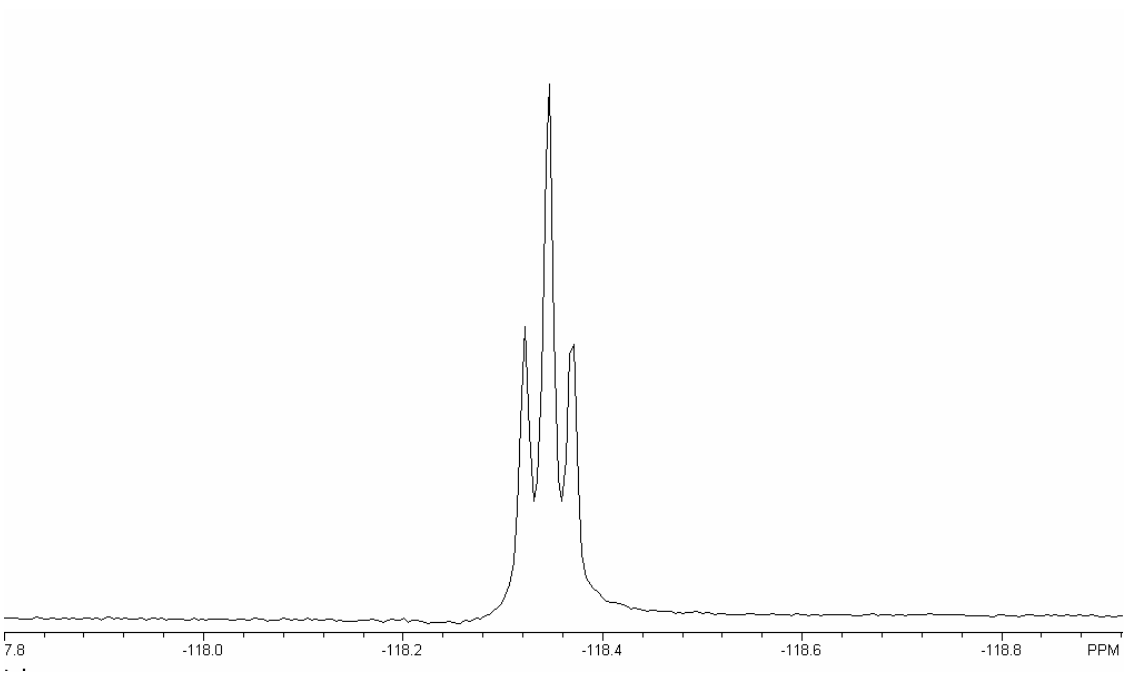


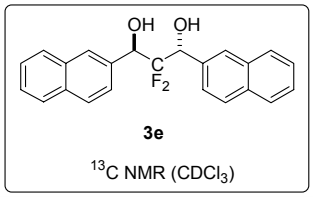


3e

^{19}F NMR ($CDCl_3$)

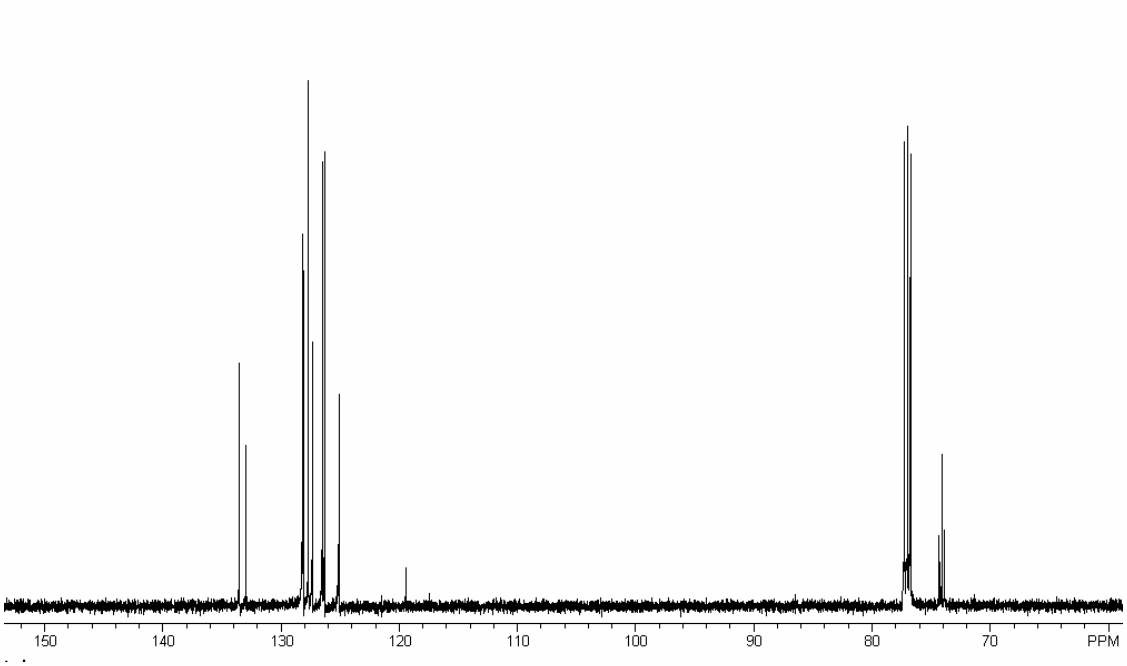


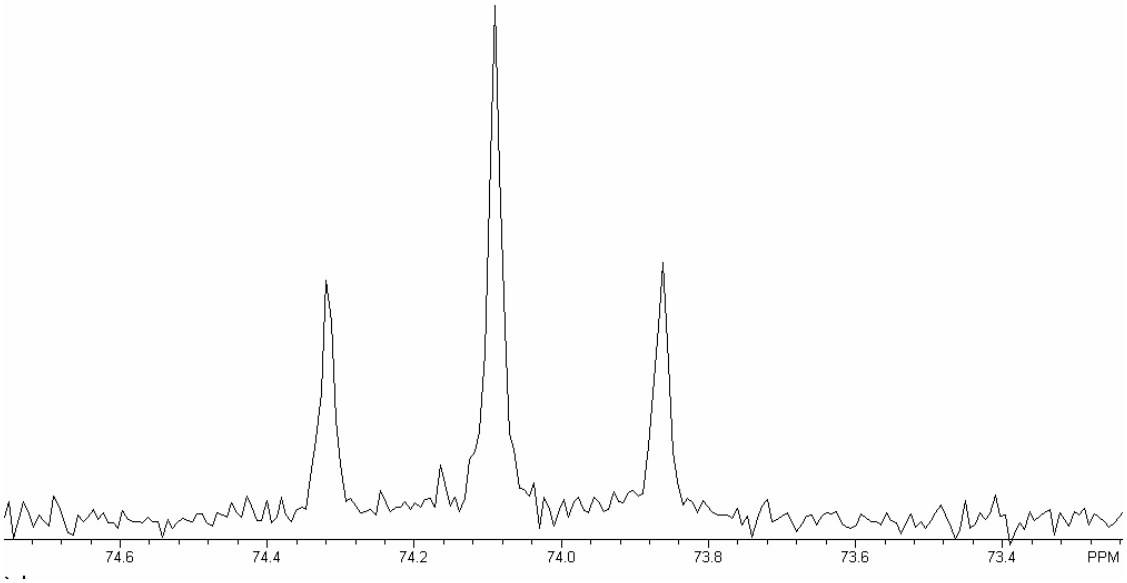


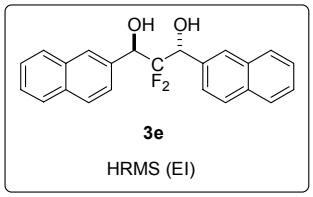


3e

^{13}C NMR ($CDCl_3$)







3e

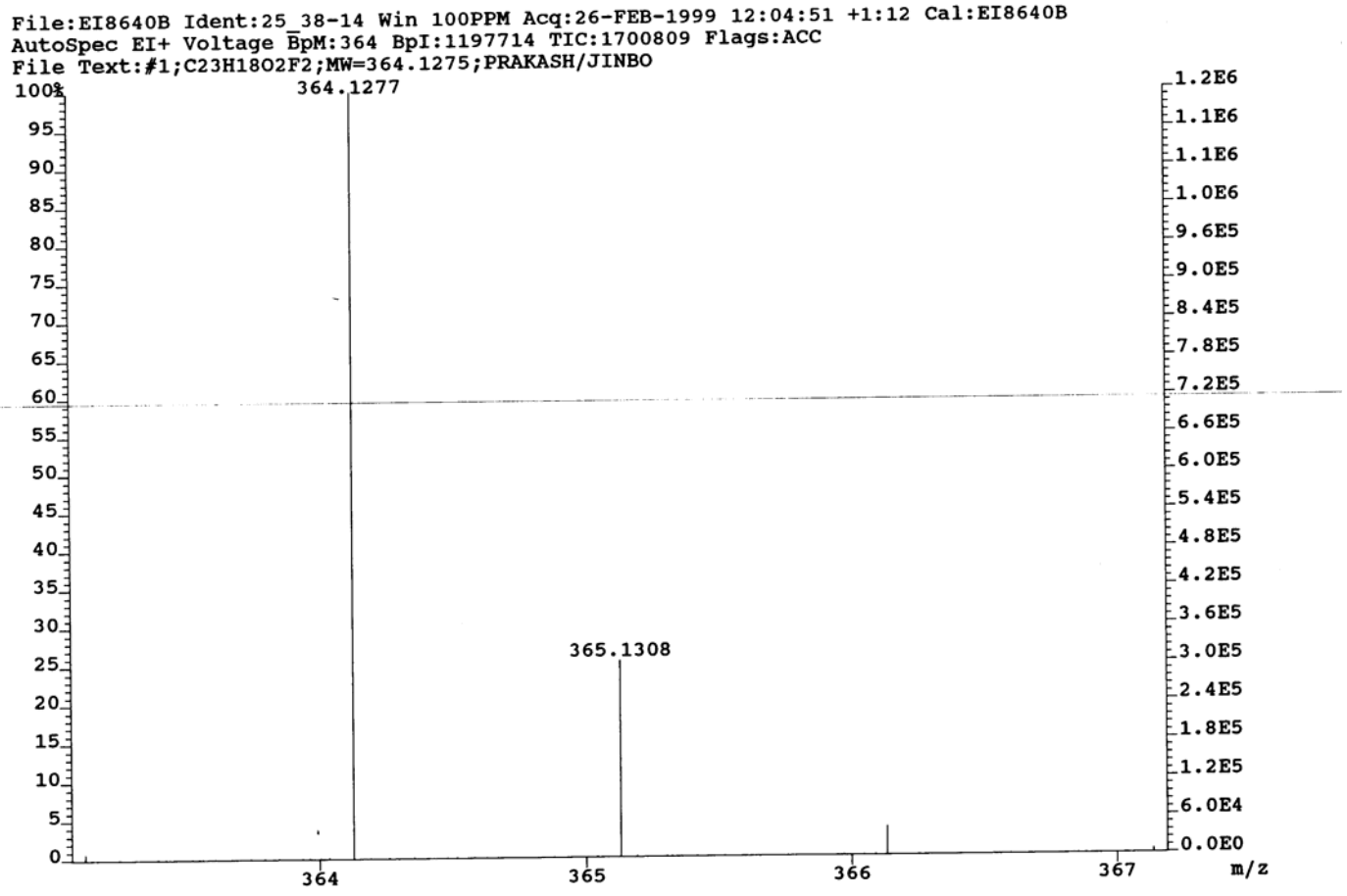
HRMS (EI)

Elemental Composition Date : 26-FEB-1999

File:EI8640B Ident:25_38-14 Win 100PPM Acq:26-FEB-1999 12:04:51 +1:12
AutoSpec EI+ Voltage BpM:364 BpI:1197714 TIC:1700809 Flags:ACC
File Text:#1;C23H18O2F2;MW=364.1275;PRAKASH/JINBO
Heteroatom Max: 20 Ion: Both Even and Odd
Limits:

363.069	0.0					-0.5	22	0	18	2	2
367.188	100.0		20.0			20.0	23	1	18	2	2

Mass	%RA	Pks	Std	PPM	mDa	Calc. Mass	DBE	C	13C	H	O	F
365.130833	25.5	14	4.7	0.0	0.0	365.130841	14.0	22	1	18	2	2
364.127730	100.0	14	4.7	-0.7	-0.2	364.127487	14.0	23		18	2	2


File:EI8640B Ident:25_38-14 Win 100PPM Acq:26-FEB-1999 12:04:51 +1:12 Cal:EI8640B
AutoSpec EI+ Voltage BpM:364 BpI:1197714 TIC:1700809 Flags:ACC
File Text:#1;C23H18O2F2;MW=364.1275;PRAKASH/JINBO
364.1277
365.1308
100%
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
1.2E6
1.1E6
1.1E6
1.0E6
9.6E5
9.0E5
8.4E5
7.8E5
7.2E5
6.6E5
6.0E5
5.4E5
4.8E5
4.2E5
3.6E5
3.0E5
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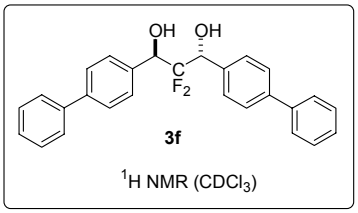
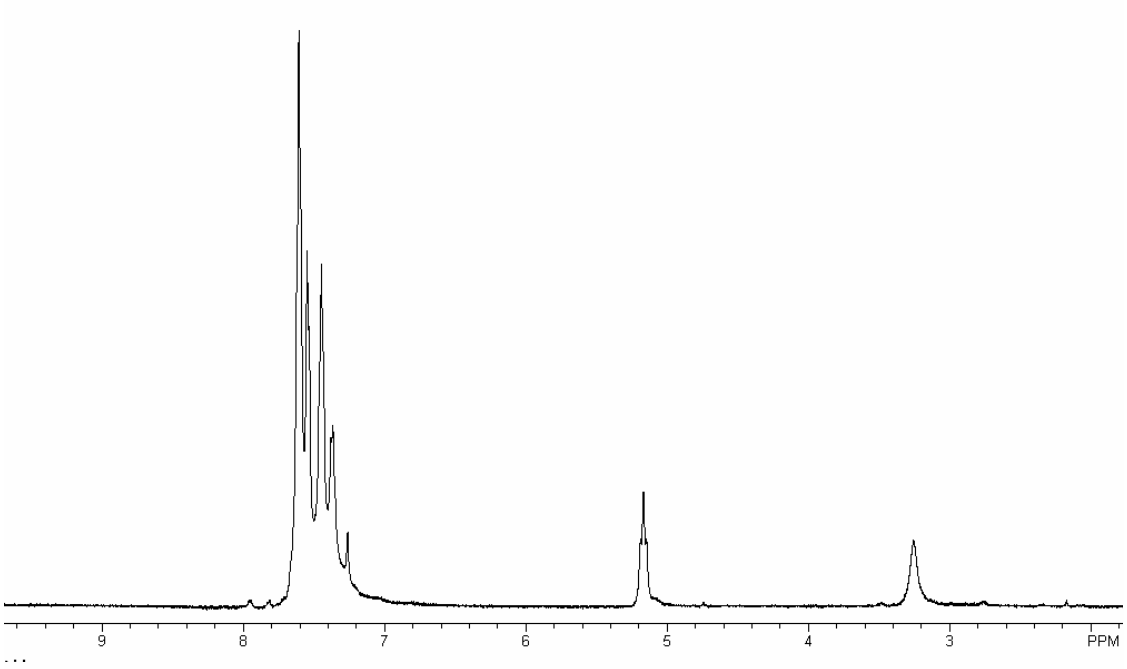
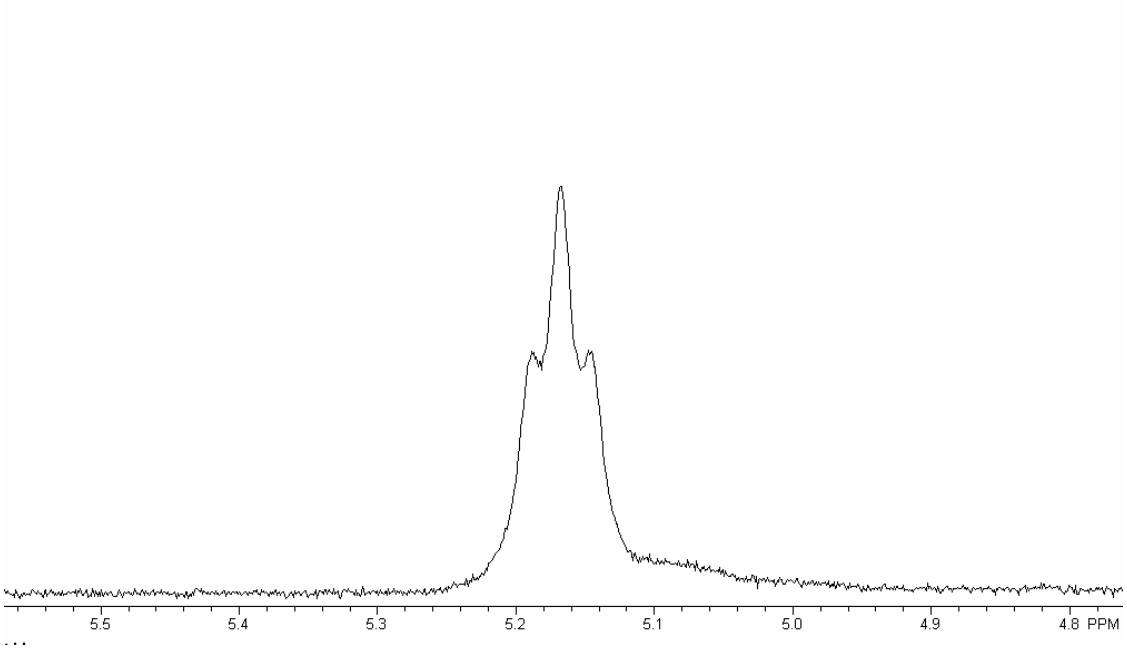
1.8E5
1.2E5
6.0E4
0.0E0
364
365
366
367
m/z

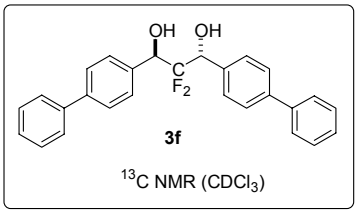


3f

^{1}H NMR ($CDCl_3$)

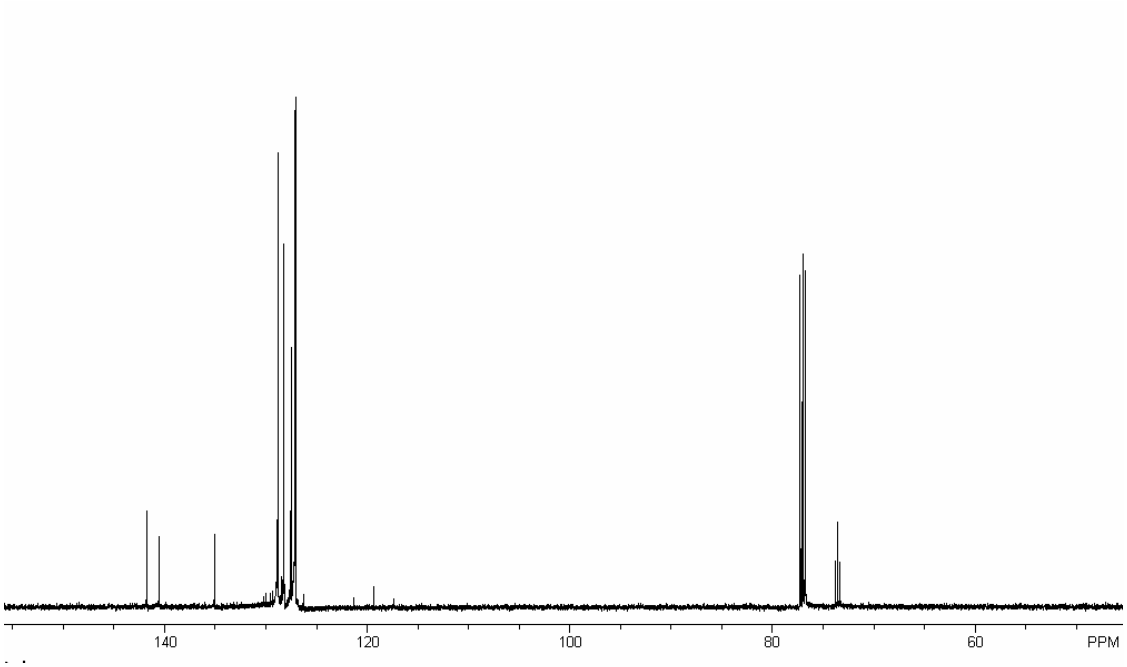


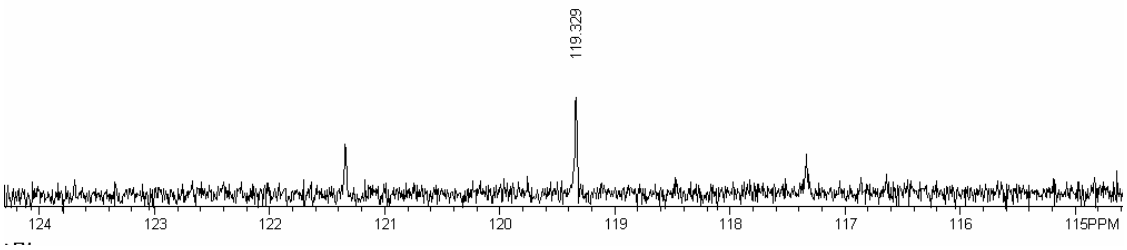


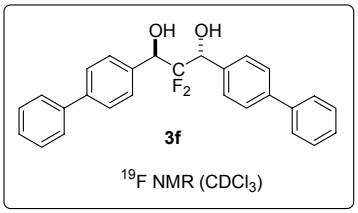


3f

^{13}C NMR ($CDCl_3$)

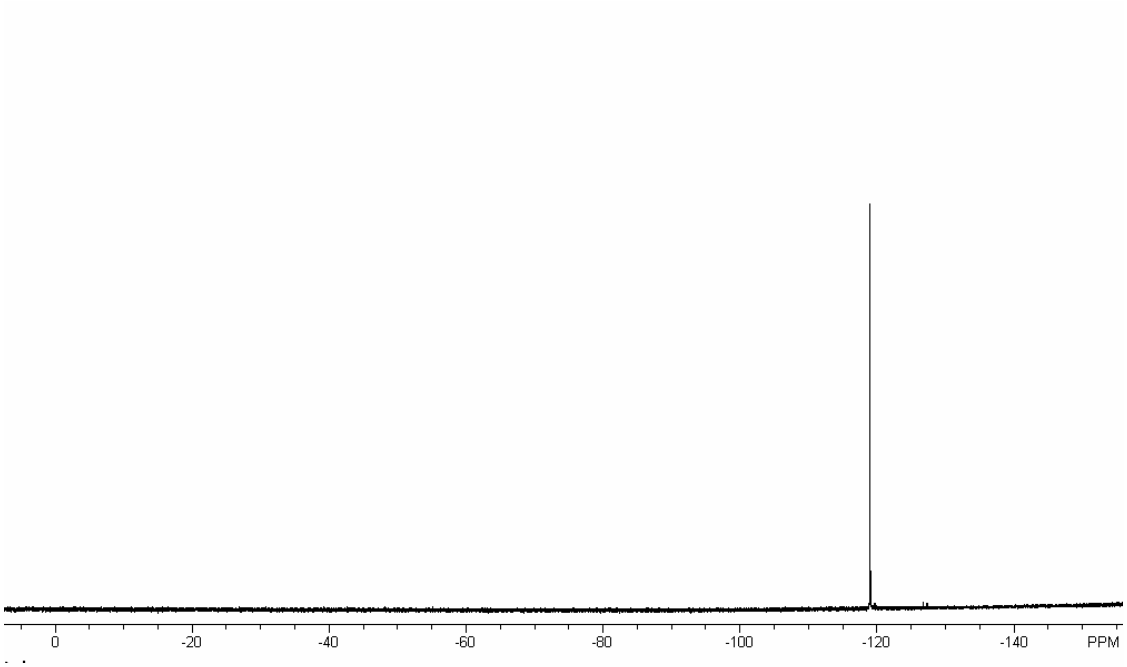


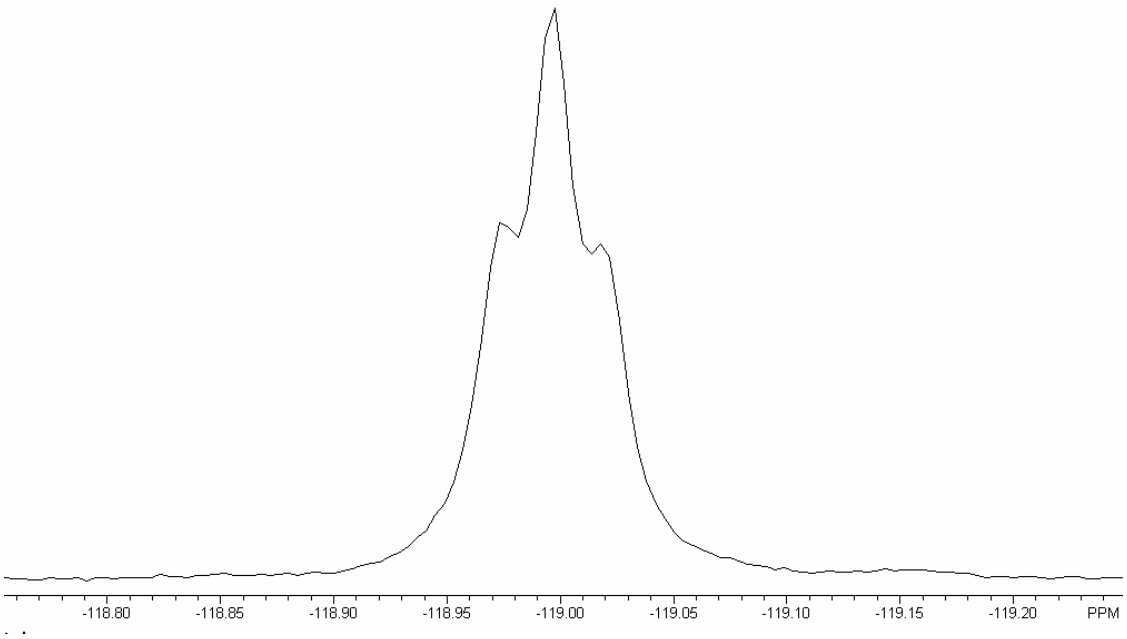


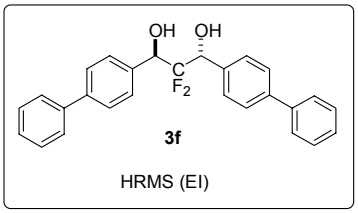


3f

^{19}F NMR ($CDCl_3$)







3f

HRMS (EI)

Elemental Composition Date : 6-MAR-1999

File:EI8644A Ident:19_34-12_13 Win 100PPM Acq:27-FEB-1999 11:47:57 +1:01
AutoSpec EI+ Voltage BpM:416 BpI:7036519 TIC:10510495 Flags:ACC
File Text:#5;C27H2402F2;MW=418.1744;PRAKASH.JINBO
Heteroatom Max: 20 Ion: Both Even and Odd
Limits:

414.908	0.0					-0.5	26	0	22	2	2	
419.063	100.0		20.0			20.0	27	1	22	2	2	

Mass	%RA	Pks	Std	PPM	mDa	Calc. Mass	DBE	C	13C	H	O	F
417.160084	30.5	16	13.5	4.9	2.1	417.162141	16.0	26	1	22	2	2
416.156959	100.0	15	12.0	4.4	1.8	416.158787	16.0	27		22	2	2

File:EI8644A Ident:19_34-12_13 Win 100PPM Acq:27-FEB-1999 11:47:57 +1:01 Cal:EI8644A
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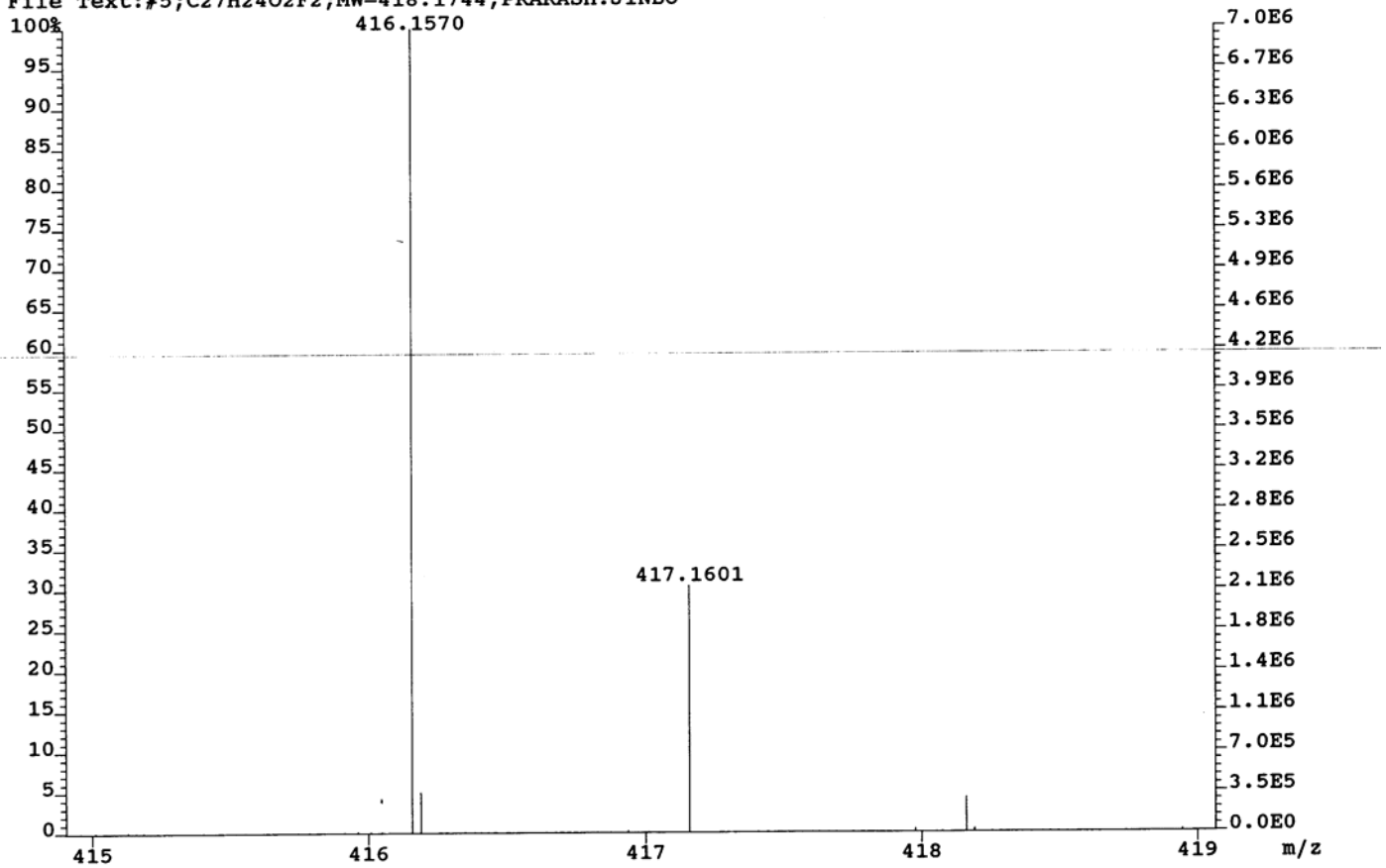
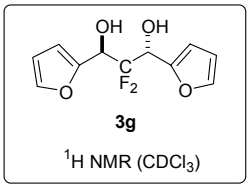
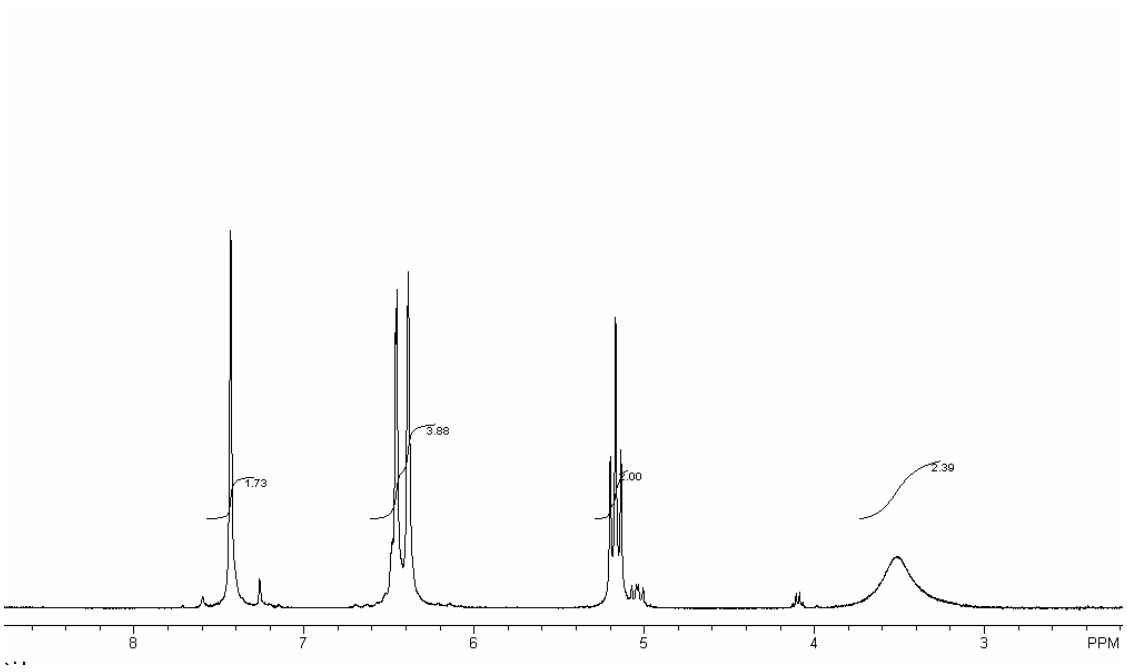
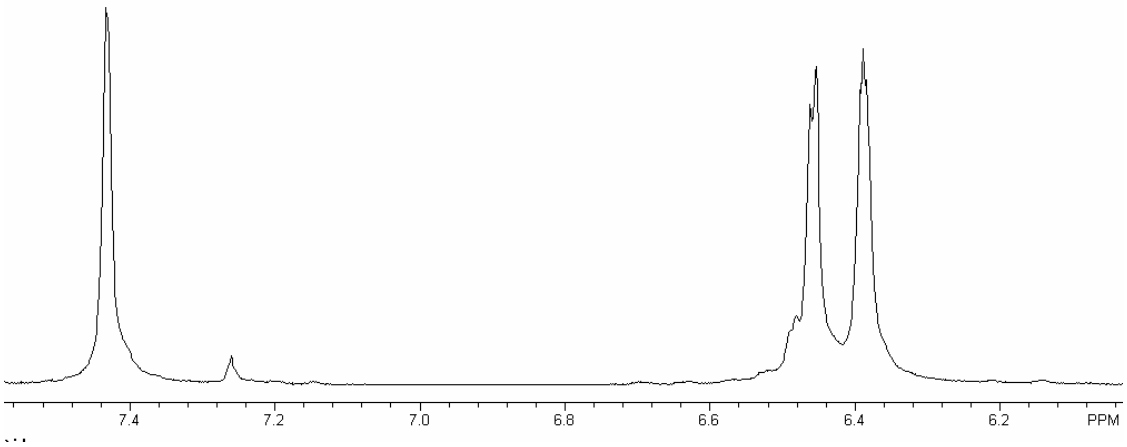


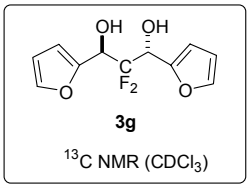


3g

^{1}H NMR ($CDCl_3$)

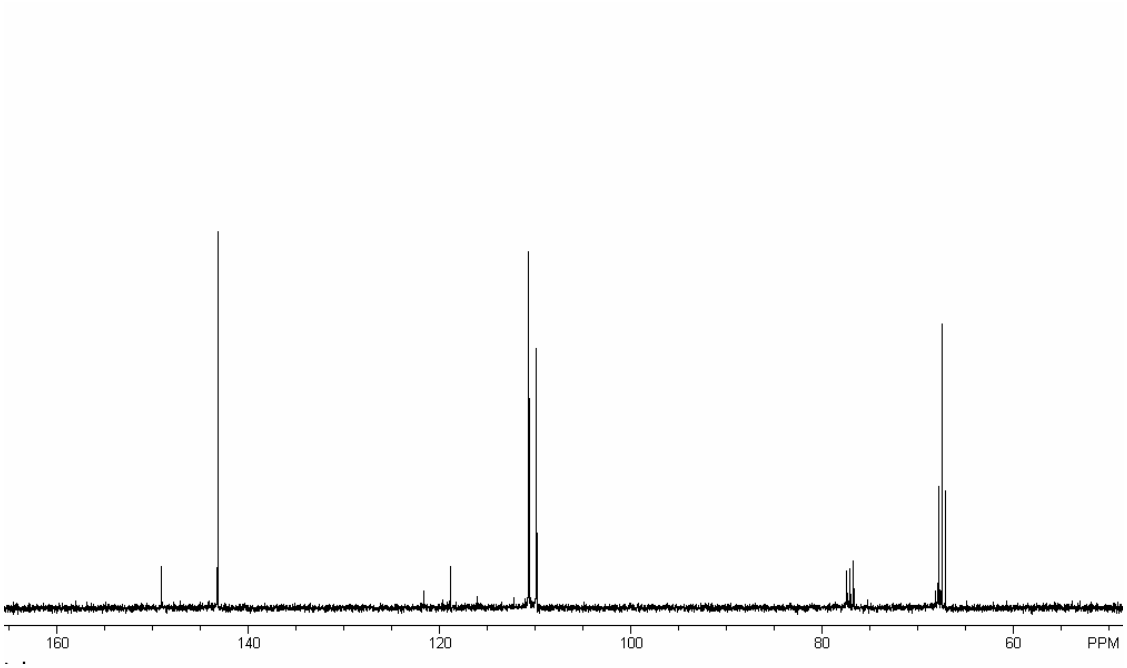


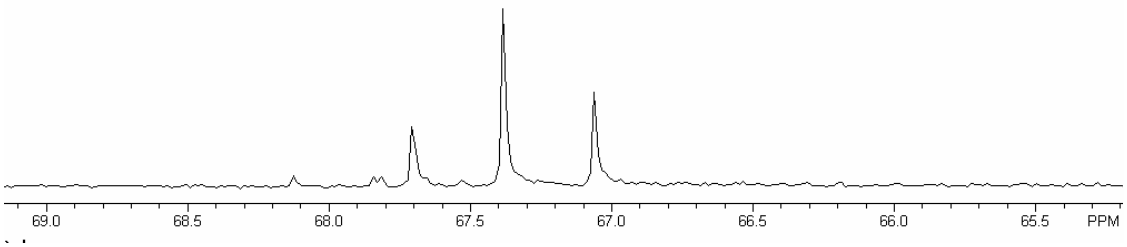


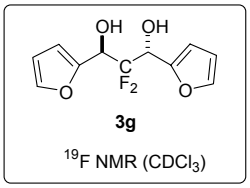


3g

^{13}C NMR ($CDCl_3$)

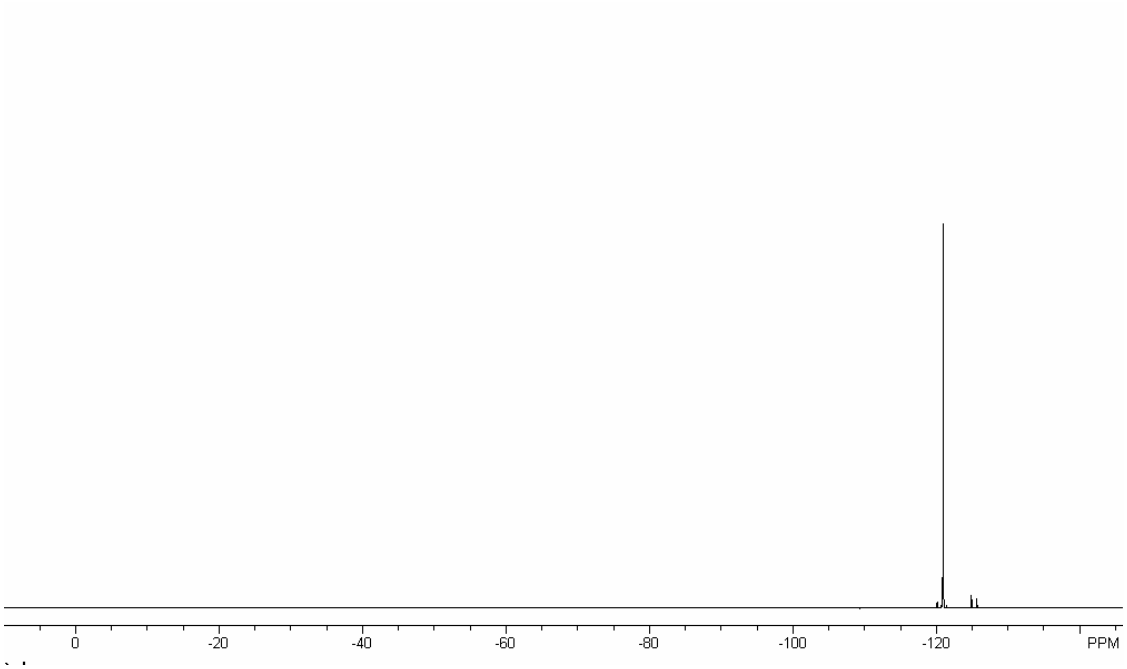


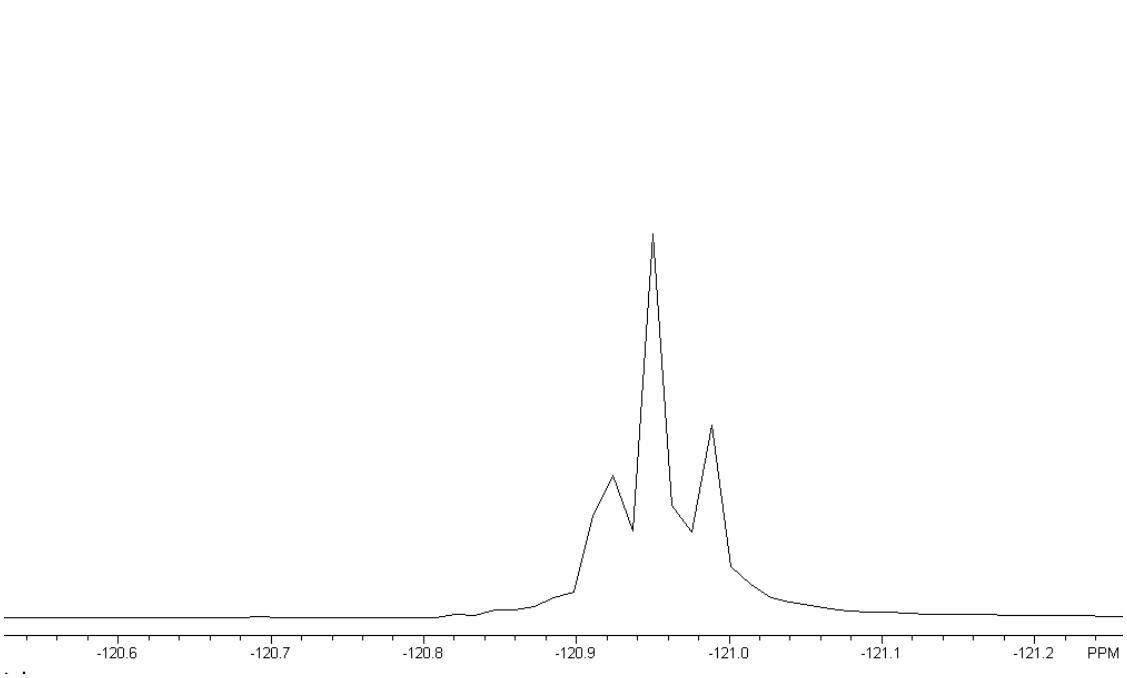


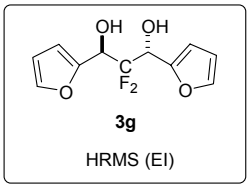


3g

^{19}F NMR ($CDCl_3$)







3g

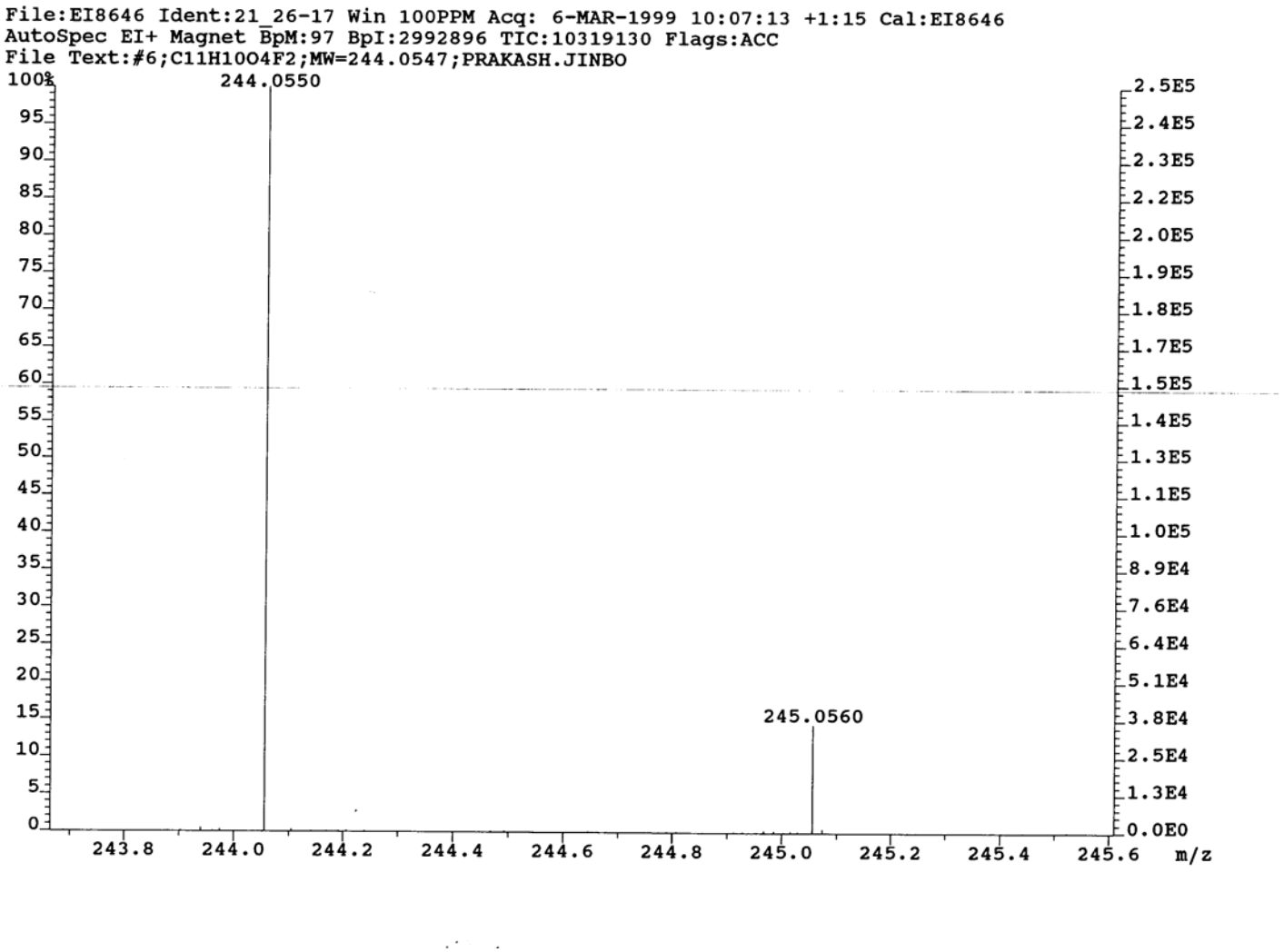
HRMS (EI)

Elemental Composition Date : 6-MAR-1999

File:EI8646 Ident:21_26-17 Win 100PPM Acq: 6-MAR-1999 10:07:13 +1:15 Cal:EI8646
AutoSpec EI+ Magnet BpM:97 BpI:2992896 TIC:10319130 Flags:ACC
File Text:#6;C11H1004F2;MW=244.0547;PRAKASH.JINBO
Heteroatom Max: 20 Ion: Both Even and Odd
Limits:

243.665	0.0					-0.5	10	0	10	4	2	
245.608	100.0			20.0		20.0	11	1	10	4	2	

Mass	%RA	Pks	Std	PPM	mDa	Calc. Mass	DBE	C	13C	H	O	F
245.055997	1.2	6	0.0	8.5	2.1	245.058070	6.0	10	1	10	4	2
244.054992	8.5	6	9.4	-1.1	-0.3	244.054715	6.0	11		10	4	2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File Text:#6;C11H10O4F2;MW=244.0547;PRAKASH.JINBO
244.0550
245.0560
100%
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
2.5E5
2.4E5
2.3E5
2.2E5
2.0E5
1.9E5
1.8E5
1.7E5
1.5E5
1.4E5
1.3E5
1.1E5
1.0E5
8.9E4
7.6E4
6.4E4
5.1E4
3.8E4
2.5E4
1.3E4
0.0E0
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244.0
244.2
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244.6
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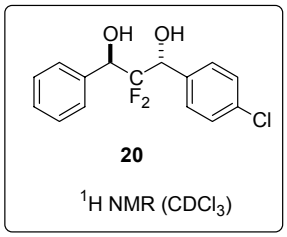
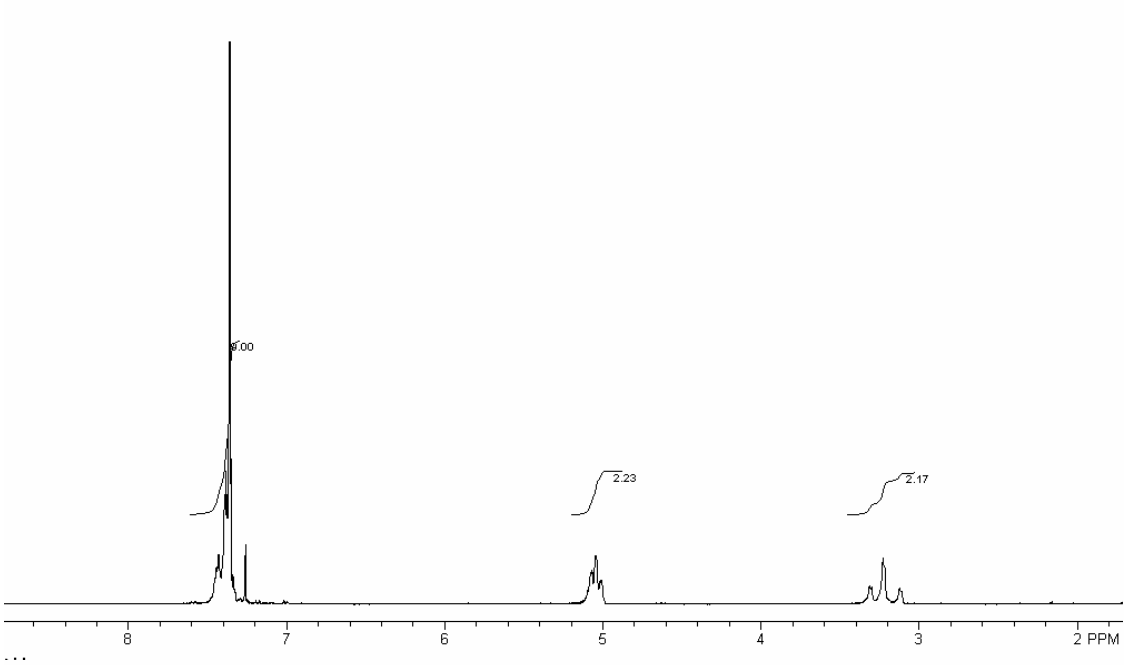
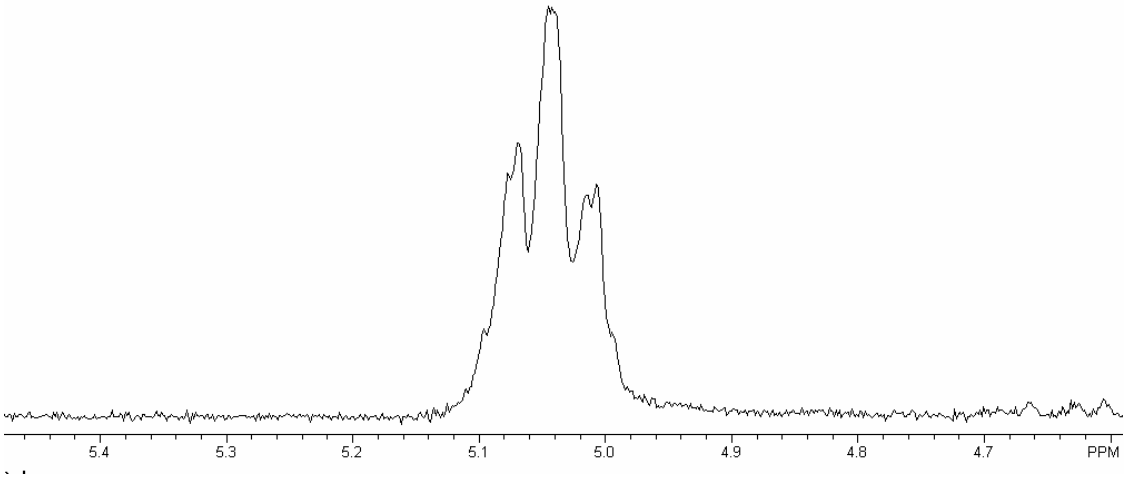
245.0
245.2
245.4
245.6
m/z

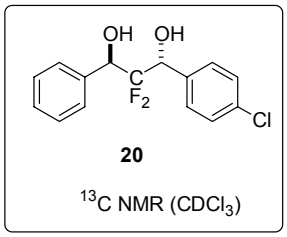


20

^{1}H NMR ($CDCl_3$)

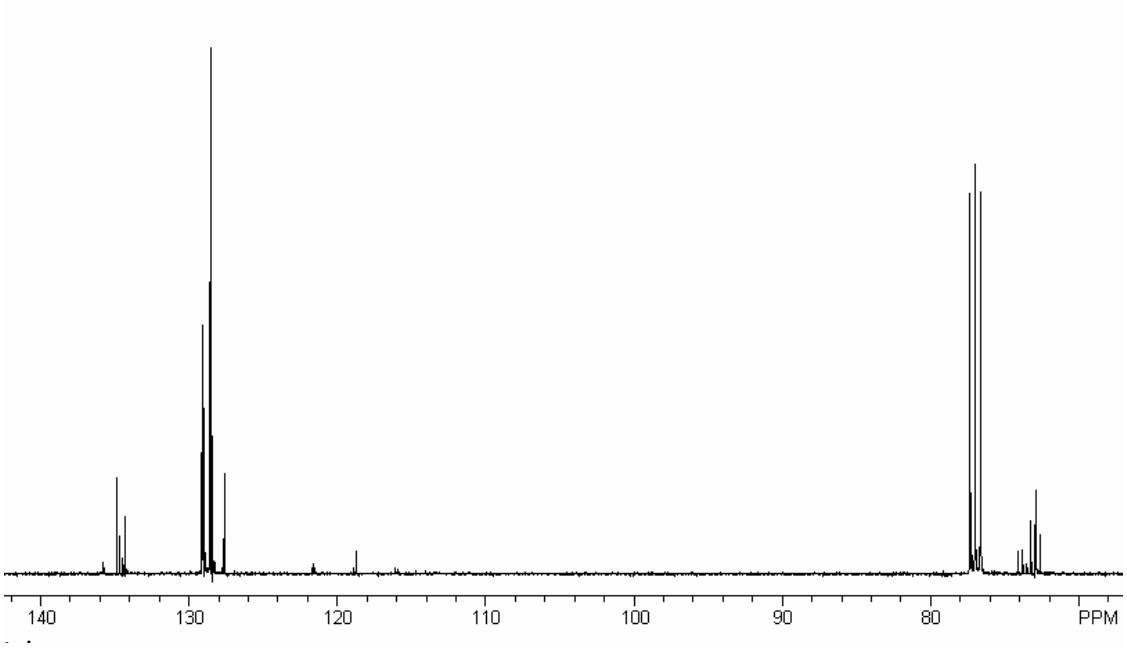


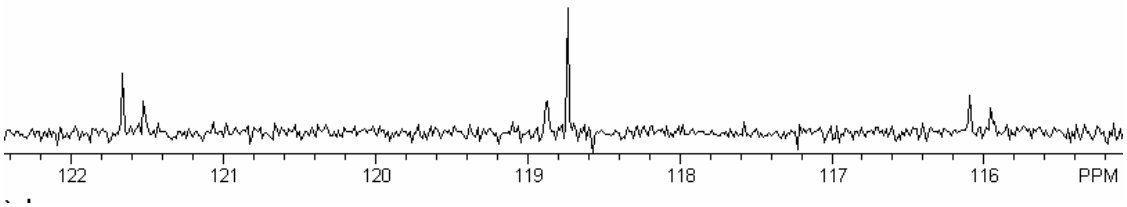


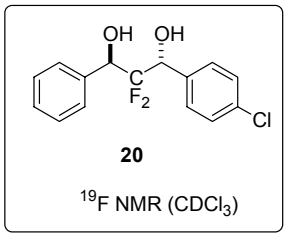


20

^{13}C NMR ($CDCl_3$)







20

^{19}F NMR ($CDCl_3$)