

Supporting Information

Studies toward the Total Synthesis of Parvifolals A/B: An Intramolecular o-Quinone Methide [4 + 2]-Cycloaddition to Construct the Central Tetracyclic Core

Dinesh J. Paymode^{†‡} and Chepuri V. Ramana^{*†}

[†]Division of Organic Chemistry, CSIR-National Chemical Laboratory, Dr. Homi Bhabha Road, Pune, Maharashtra, India- 411 008.

[‡]Academy of Scientific and Innovative Research (AcSIR), New Delhi, India- 110 020.

Email: vr.chepuri@ncl.res.in

Contents

¹ H/ ¹³ C/DEPT NMR and HRMS Spectra of Compound 6	S2 – S5
¹ H/ ¹³ C/DEPT NMR and HRMS Spectra of Compound 3	S6 – S9
¹ H/ ¹³ C/DEPT NMR and HRMS Spectra of Compound 13	S10 – S17
¹ H/ ¹³ C DEPT/NOESY/HMBC NMR and HRMS Spectra of Compound 15	S18 – S23
¹ H/ ¹³ C DEPT NMR and HRMS Spectra of Compound 17	S24 – S27
¹ H/ ¹³ C DEPT NMR and HRMS Spectra of Compound 18	S28 – S31
¹ H/ ¹³ C DEPT NMR and HRMS Spectra of Compound 19	S32 – S35
¹ H/ ¹³ C DEPT NMR and HRMS Spectra of Compound 20	S36 – S39

Figure S1: ¹H NMR of compound 6

Acquisition Time (sec)	1.6384			
Comment	Dinesh 1H			
Date	06 Jun 2016 10:46:32	Frequency (MHz)	400.13	Nucleus 1H
Number of Transients	11	Original Points Count	16384	Points Count 16384
Solvent	CHLOROFORM-d	Sweep Width (Hz)	9999.39	Temperature (grad C) 22.200

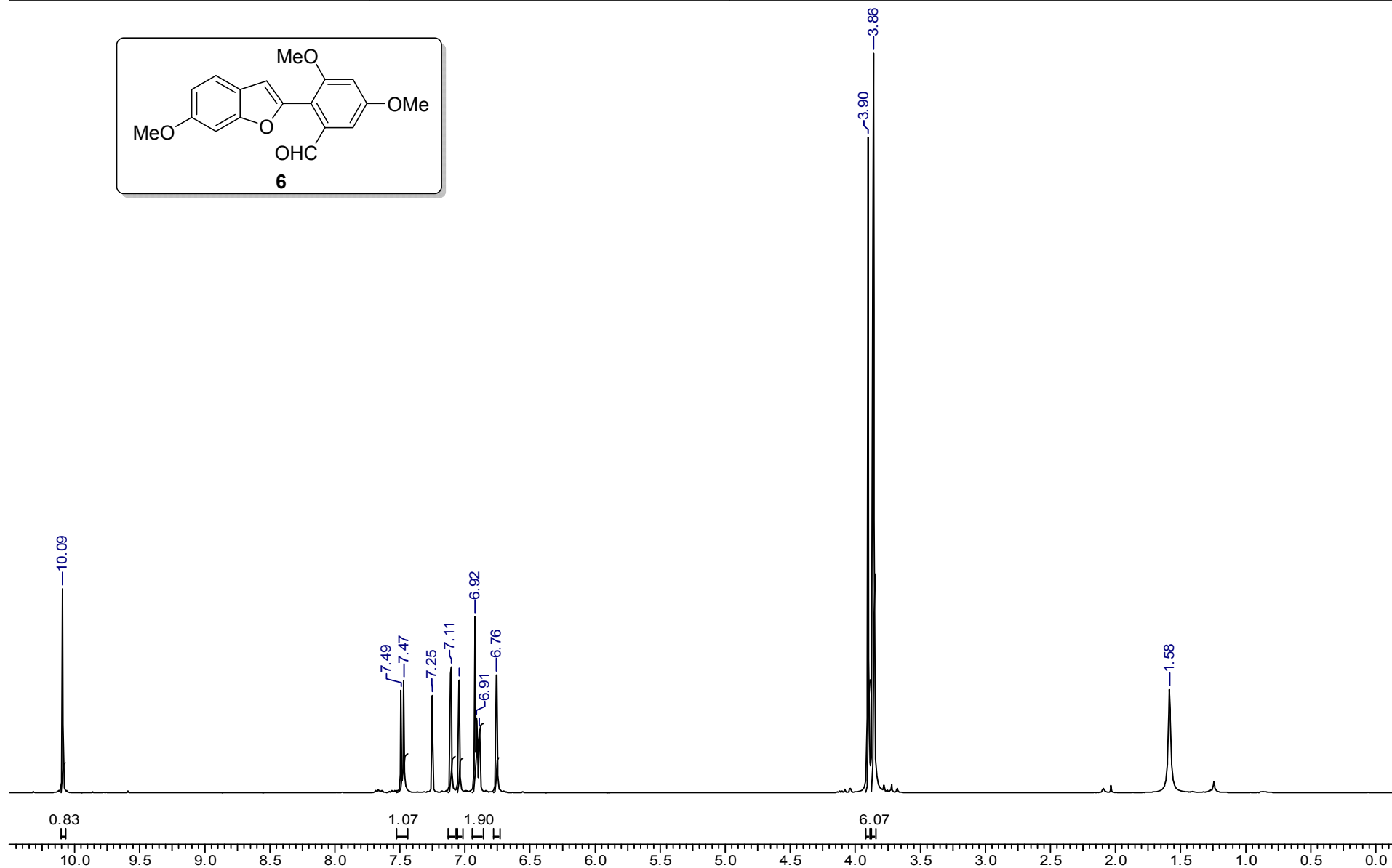
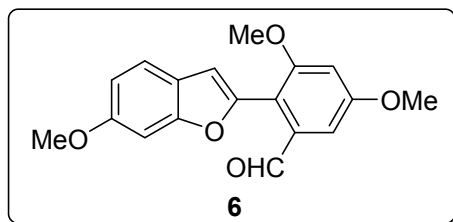


Figure S2: ^{13}C NMR of compound 6

Acquisition Time (sec)	0.6554	Comment	13C	Date	15 Jul 2017 13:56:48	Frequency (MHz)	125.76
Nucleus	^{13}C	Number of Transients	1000	Original Points Count	20480	Points Count	32768
Sweep Width (Hz)	31249.05			Temperature (grad C)	25.000	Solvent	CHLOROFORM-d

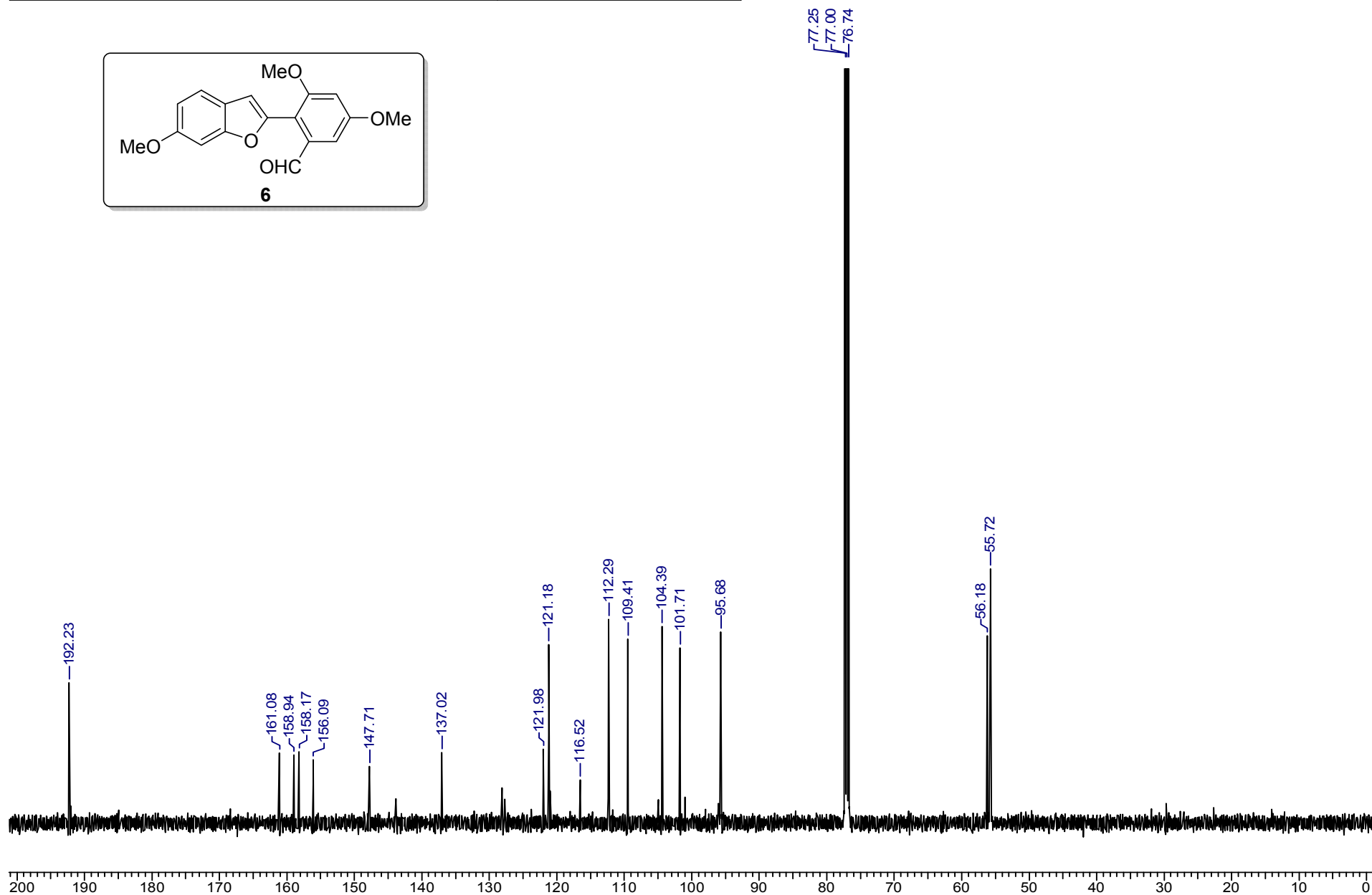
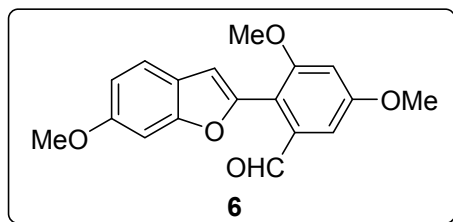


Figure S3: DEPT ^{13}C NMR of compound 6

Acquisition Time (sec)	0.6554	Comment	DEPT	Date	15 Jul 2017 13:39:44	Frequency (MHz)	125.76
Nucleus	^{13}C	Number of Transients	800	Original Points Count	20480	Points Count	32768
Sweep Width (Hz)	31249.05	Temperature (grad C)	25.000	Solvent	CHLOROFORM-d		

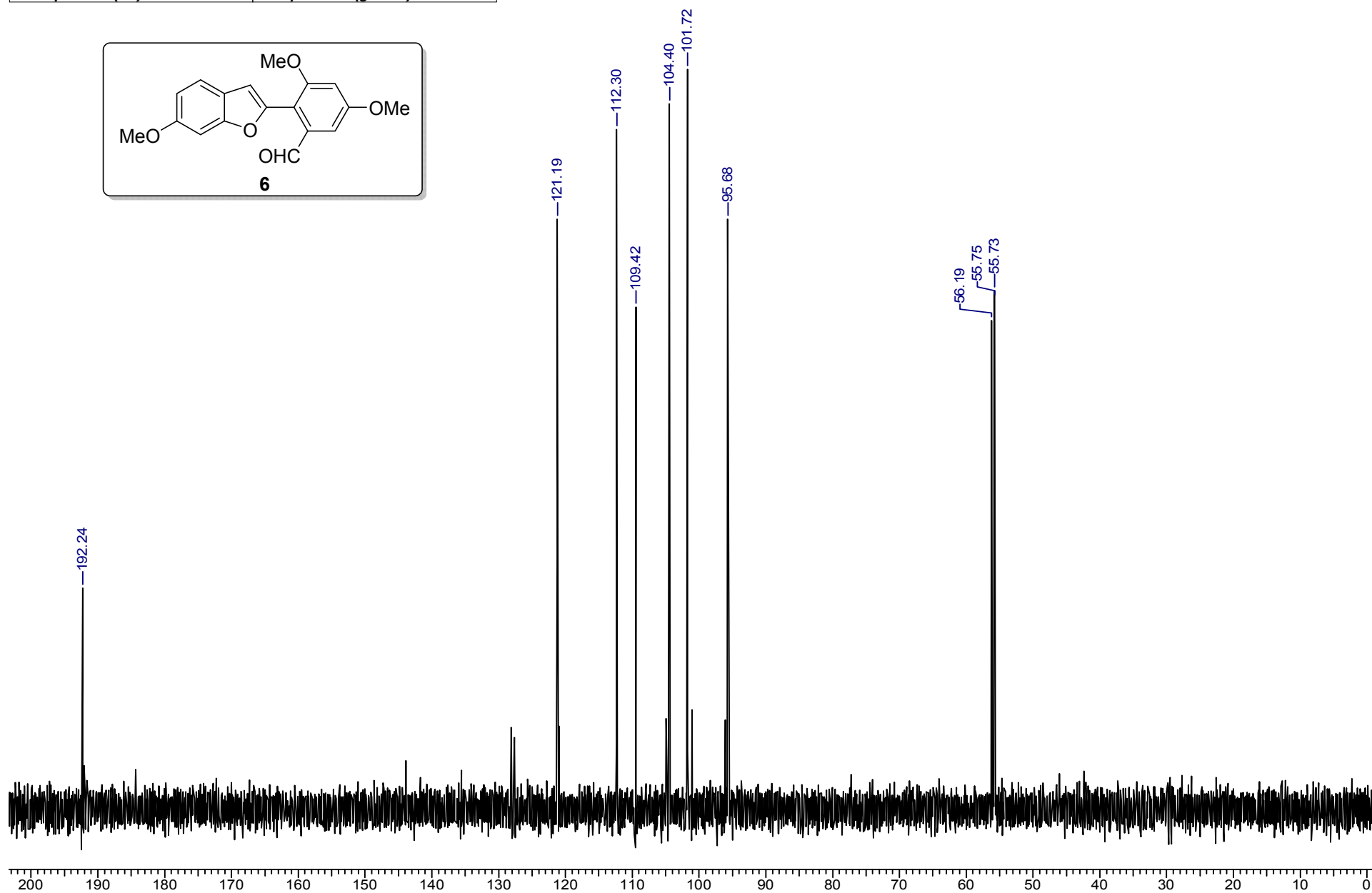
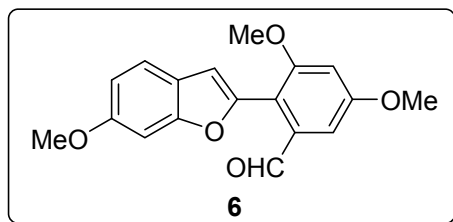


Figure S4: HRMS spectrum of compound 6

DP-837 #143 RT: 0.63 AV: 1 NL: 3.80E8

T: FTMS + p ESI Full ms [100.00-1500.00]

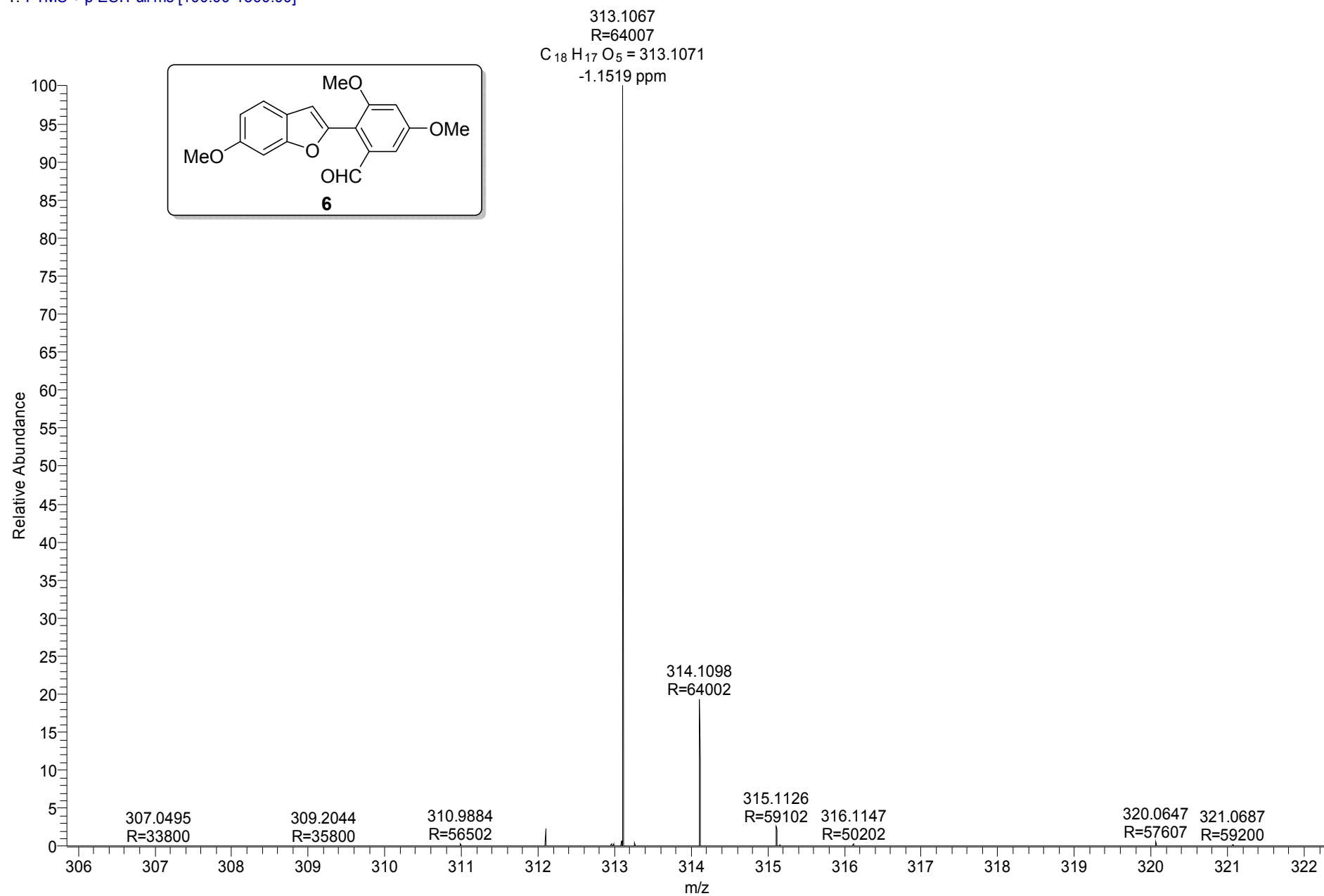


Figure S5: ¹H NMR of compound 3

Acquisition Time (sec)	1.2800	Comment	Dinesh 1H	Date	17 Jul 2017 13:01:20
Frequency (MHz)	500.13	Nucleus	1H	Number of Transients	32
Solvent	CHLOROFORM-d	Sweep Width (Hz)	12499.62	Original Points Count	16000
				Points Count	32768
				Temperature (grad C)	25.000

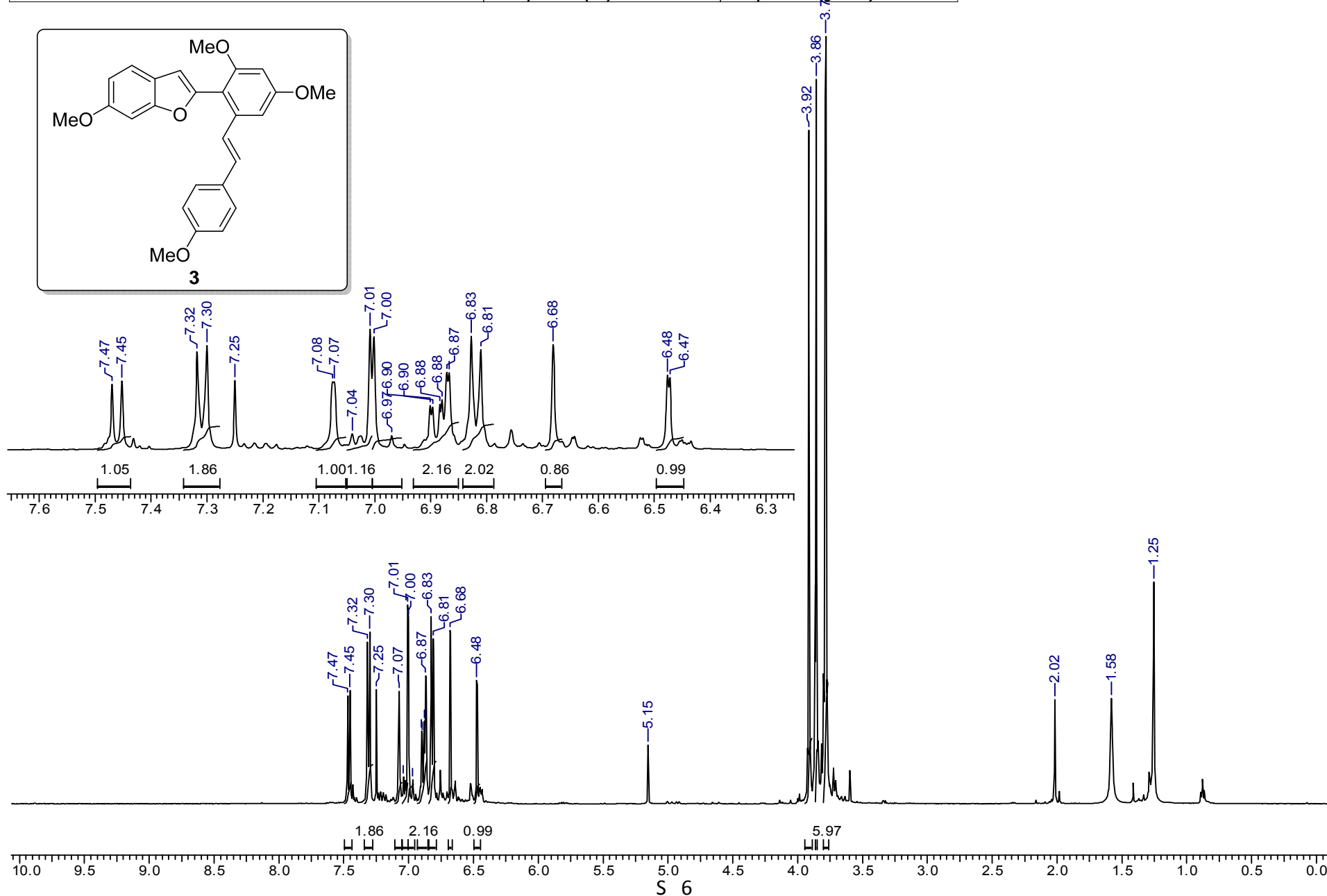


Figure S6: ^{13}C NMR of compound 3

Acquisition Time (sec)	0.6554	Comment	13C	Date	17 Jul 2017 13:46:08	Frequency (MHz)	125.76
Nucleus	13C	Number of Transients	2249	Original Points Count	20480	Points Count	32768
Sweep Width (Hz)	31249.05			Temperature (grad C)	25.000	Solvent	CHLOROFORM-d

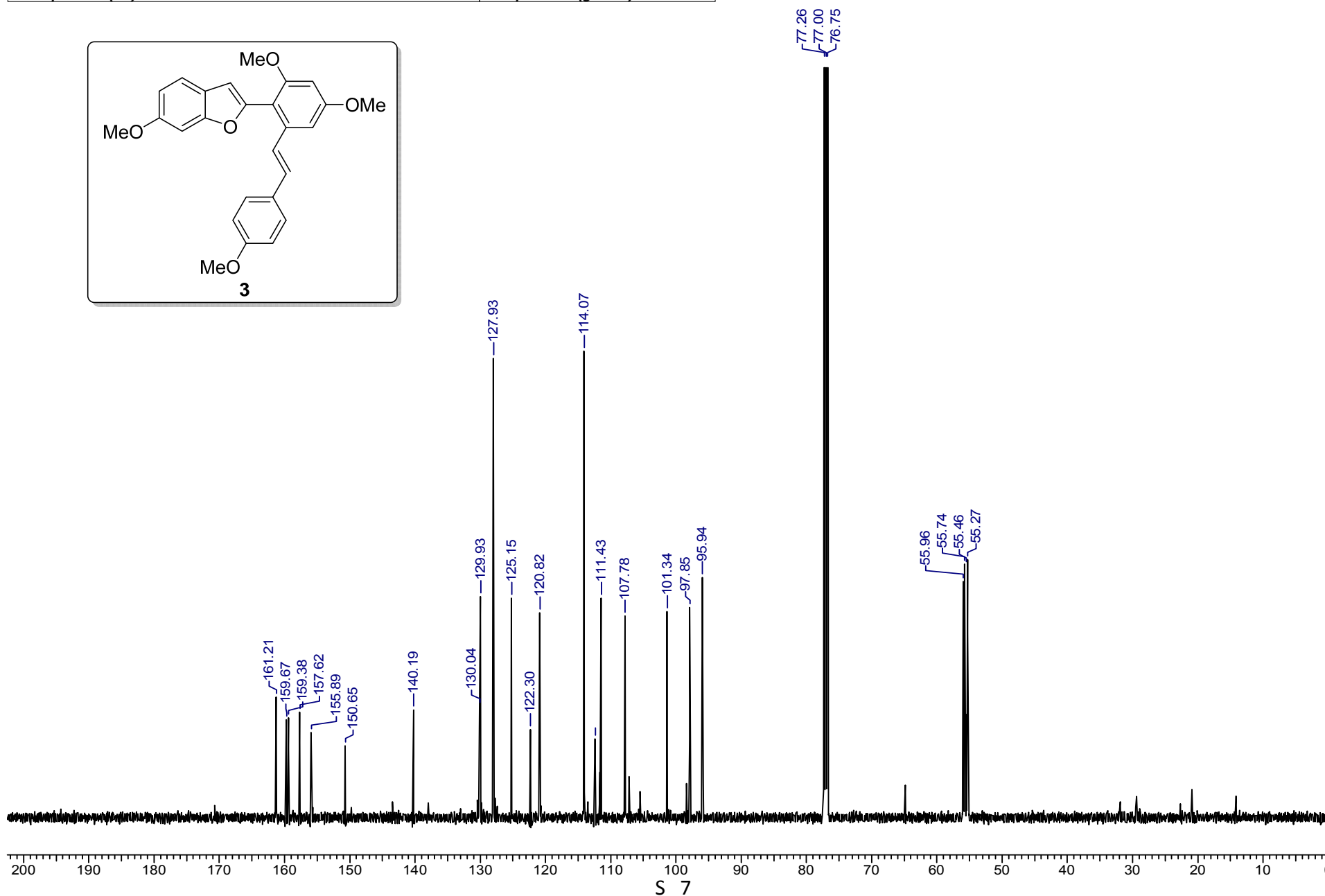
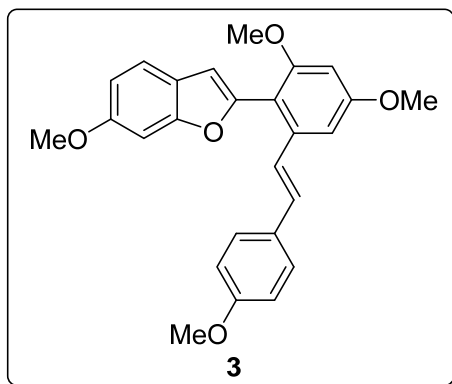


Figure S7: DEPT ^{13}C NMR of compound 3

Acquisition Time (sec)	0.6554	Comment	DEPT	Date	17 Jul 2017 13:24:48	Frequency (MHz)	125.76
Nucleus	^{13}C	Number of Transients	800	Original Points Count	20480	Points Count	32768
Sweep Width (Hz)	31249.05	Temperature (grad C)	25.000	Solvent	CHLOROFORM-d		

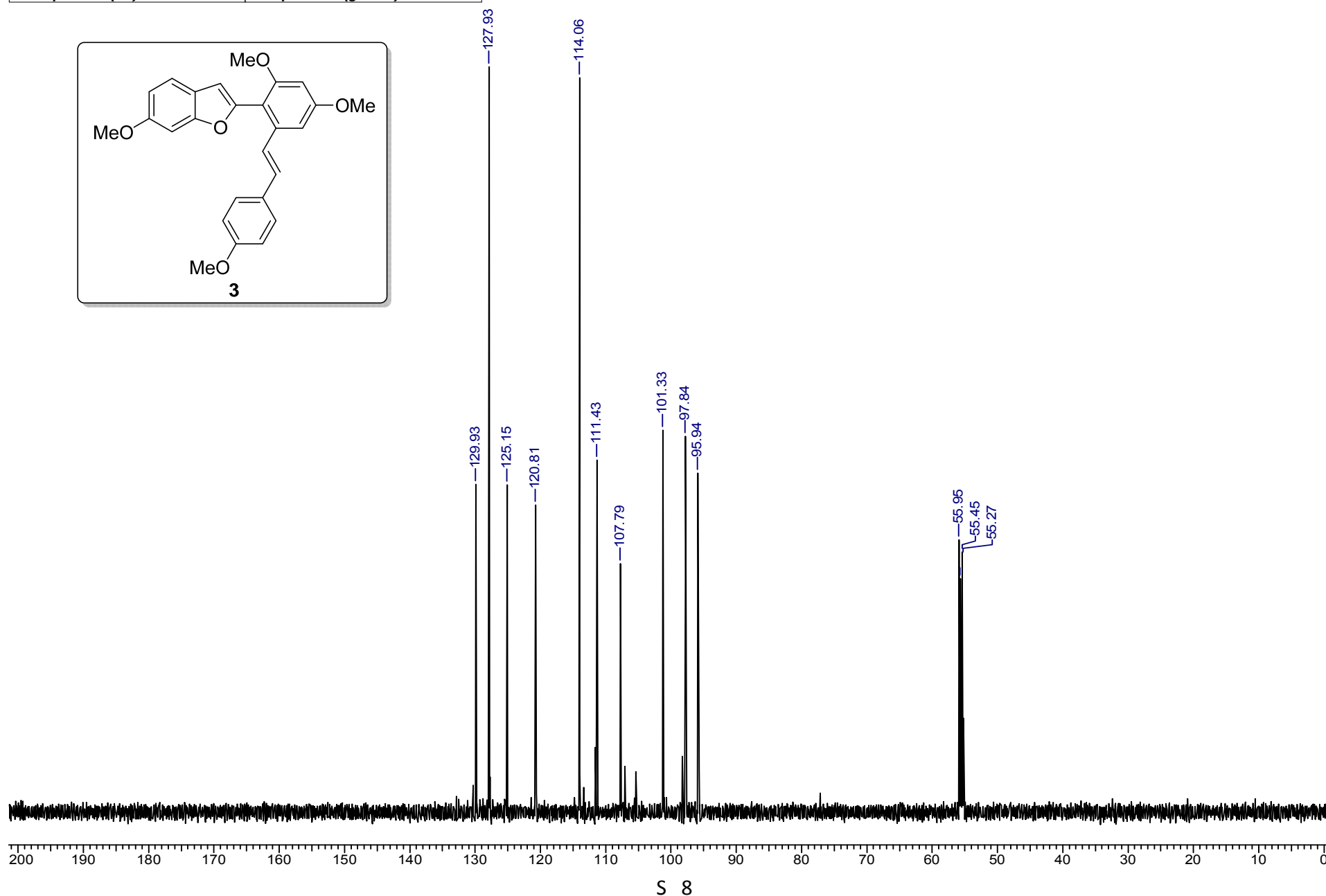
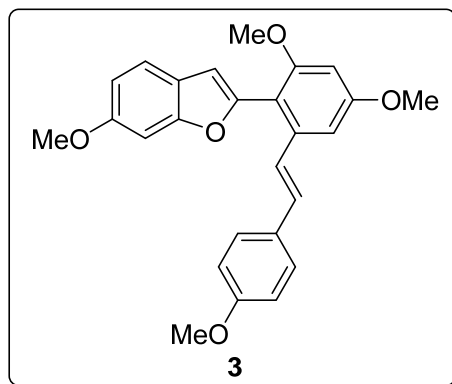


Figure S8: HRMS spectrum of compound 3

DP-843 #192 RT: 0.85 AV: 1 NL: 3.97E8

T: FTMS + p ESI Full ms [100.00-1500.00]

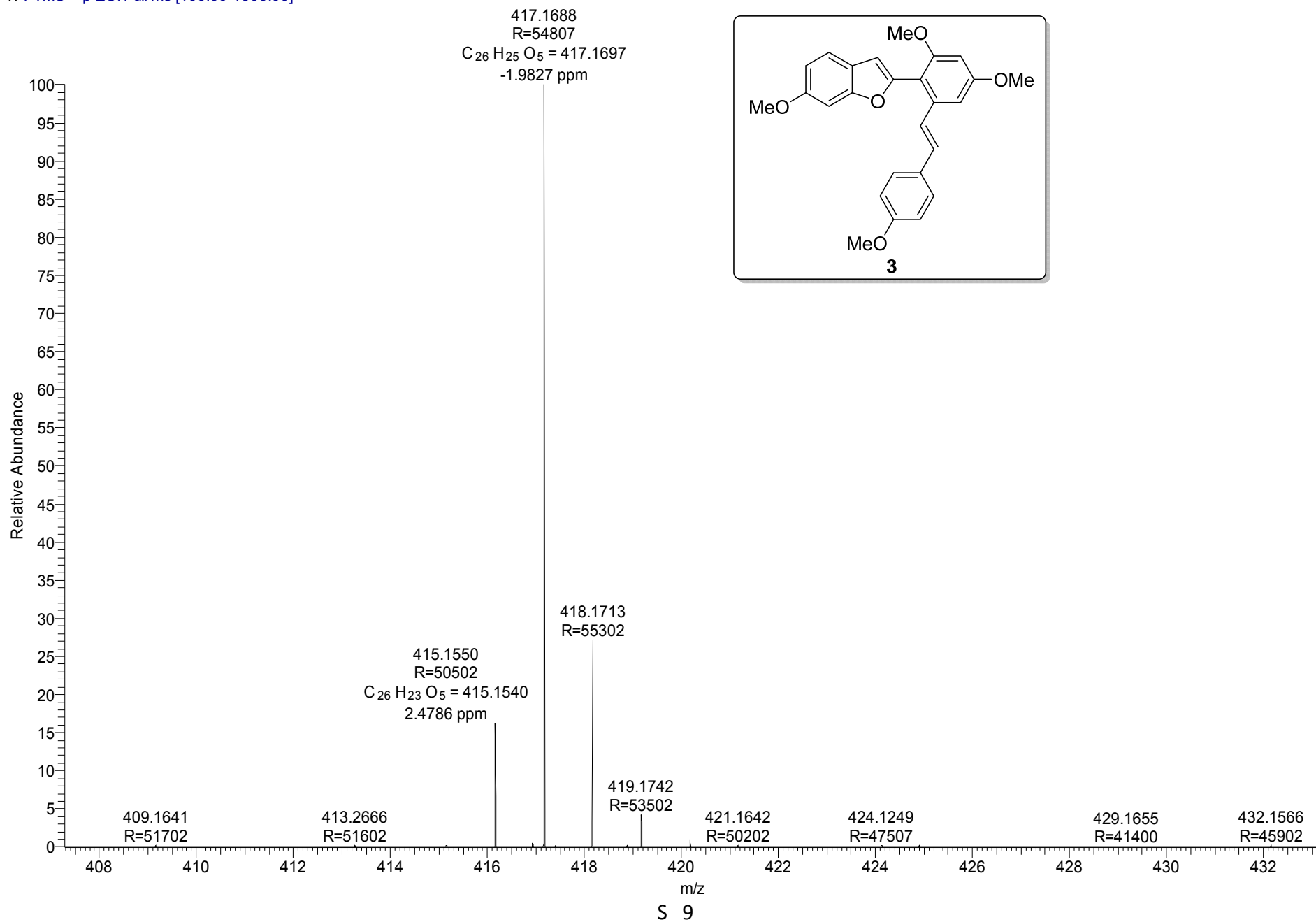


Figure S9: ¹H NMR of compound 9

Acquisition Time (sec)	3.9584	Comment	Dinesh	Date	02 Aug 2016 22:30:32	Frequency (MHz)	200.13
Nucleus	1H	Number of Transients	4	Original Points Count	16384	Points Count	32768
Sweep Width (Hz)	4138.95	Temperature (grad C)	27.000	Solvent	CHLOROFORM-d		

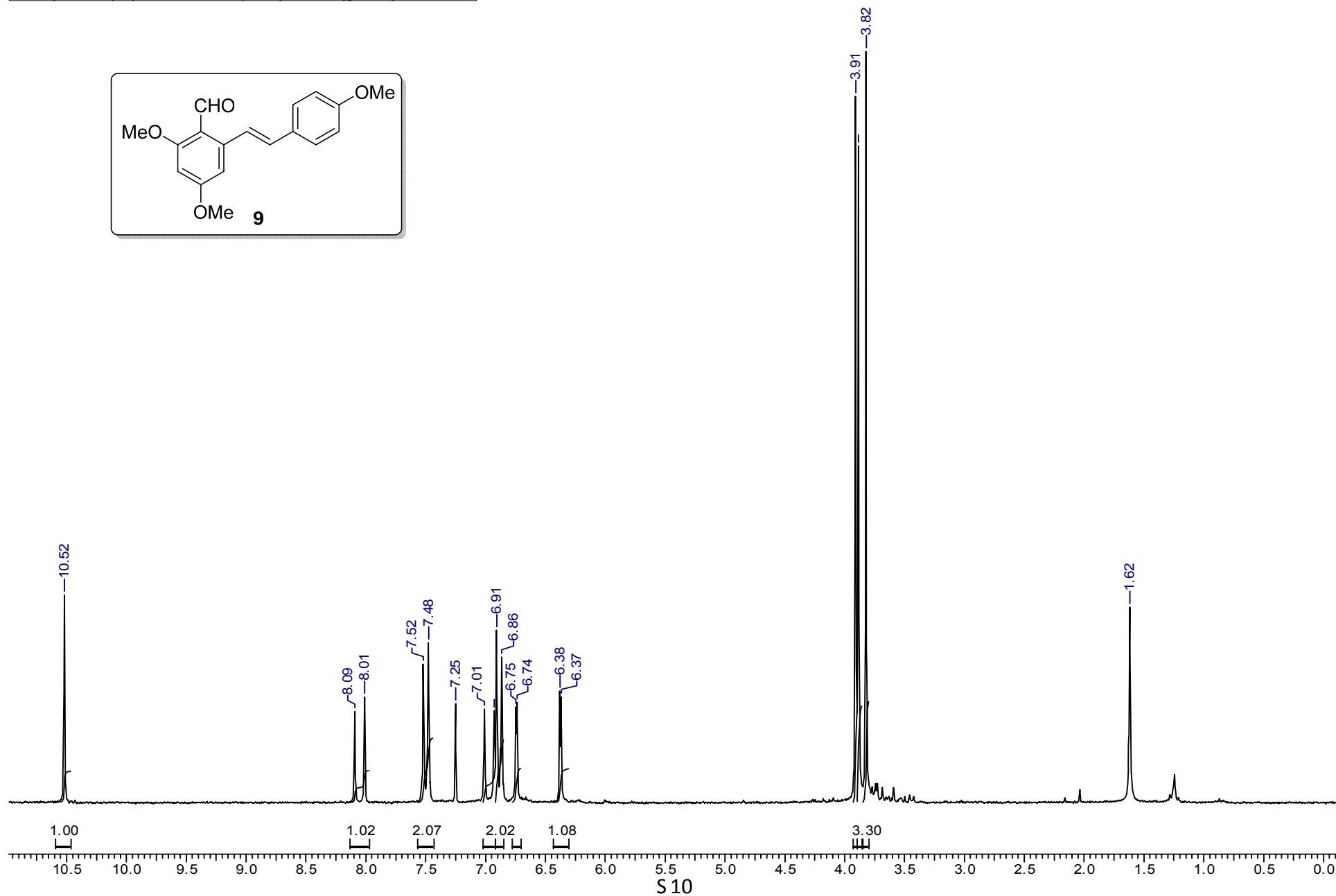
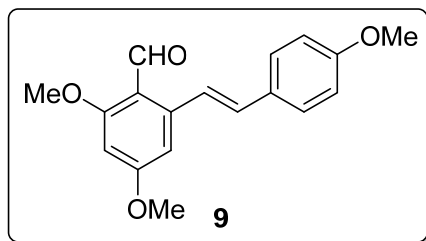


Figure S10: ^{13}C NMR of compound 9

Acquisition Time (sec)	0.6832	Comment	Dinesh	Date	06 Aug 2016 19:59:04	Frequency (MHz)	50.32
Nucleus	^{13}C	Number of Transients	600	Original Points Count	8192	Points Count	32768
Sweep Width (Hz)	11990.04	Temperature (grad C)	27.000	Solvent	CHLOROFORM-d		

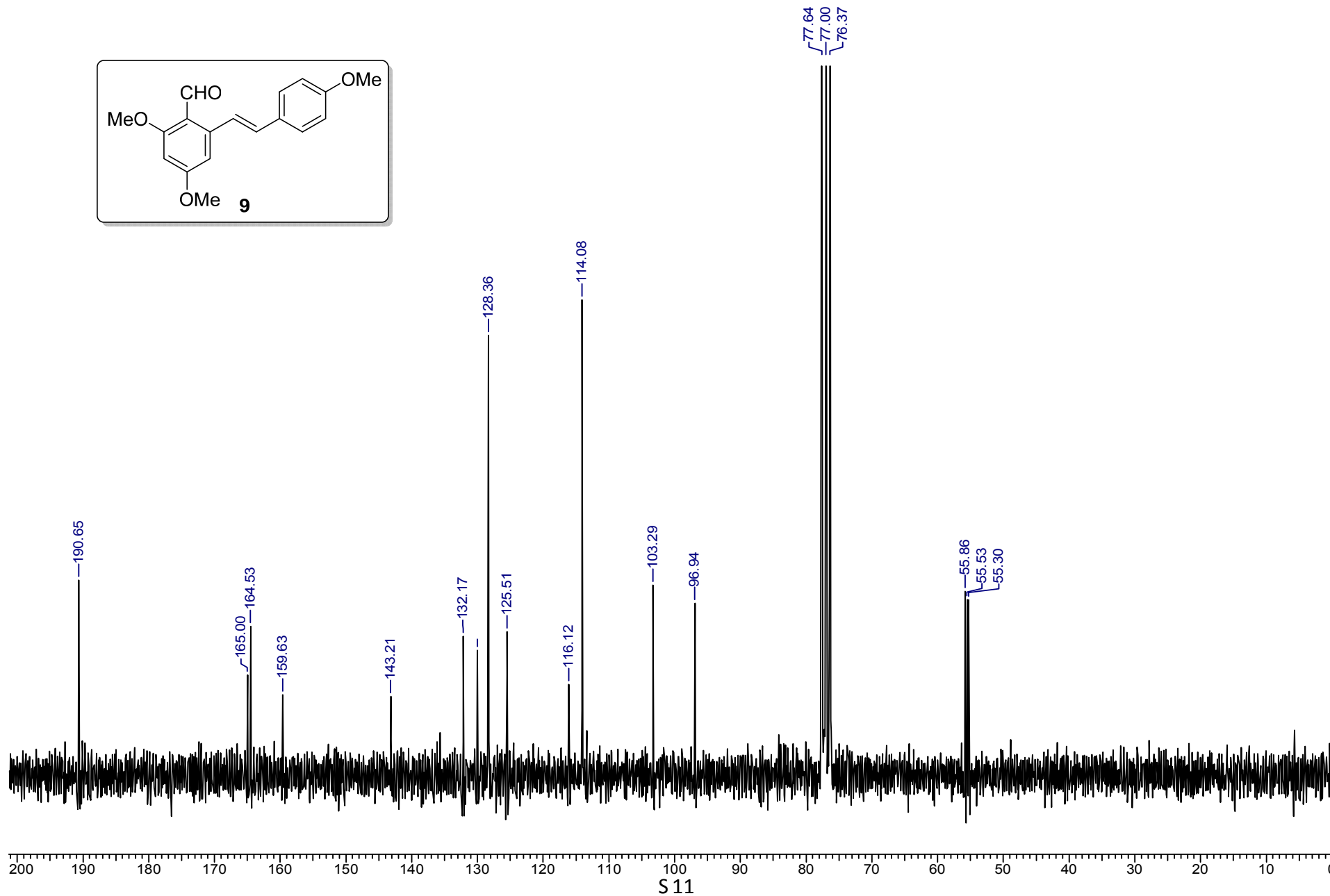
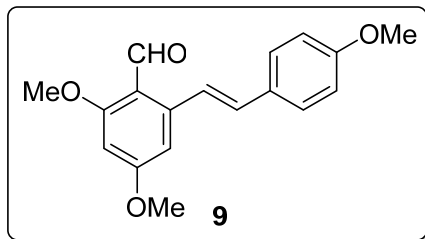


Figure S11: DEPT ^{13}C NMR of compound 9

Acquisition Time (sec)	0.6832	Comment	Dinesh	Date	06 Aug 2016 19:29:12	Frequency (MHz)	50.32
Nucleus	^{13}C	Number of Transients	400	Original Points Count	8192	Points Count	32768
Sweep Width (Hz)	11990.04			Temperature (grad C)	27.000	Solvent	CHLOROFORM-d

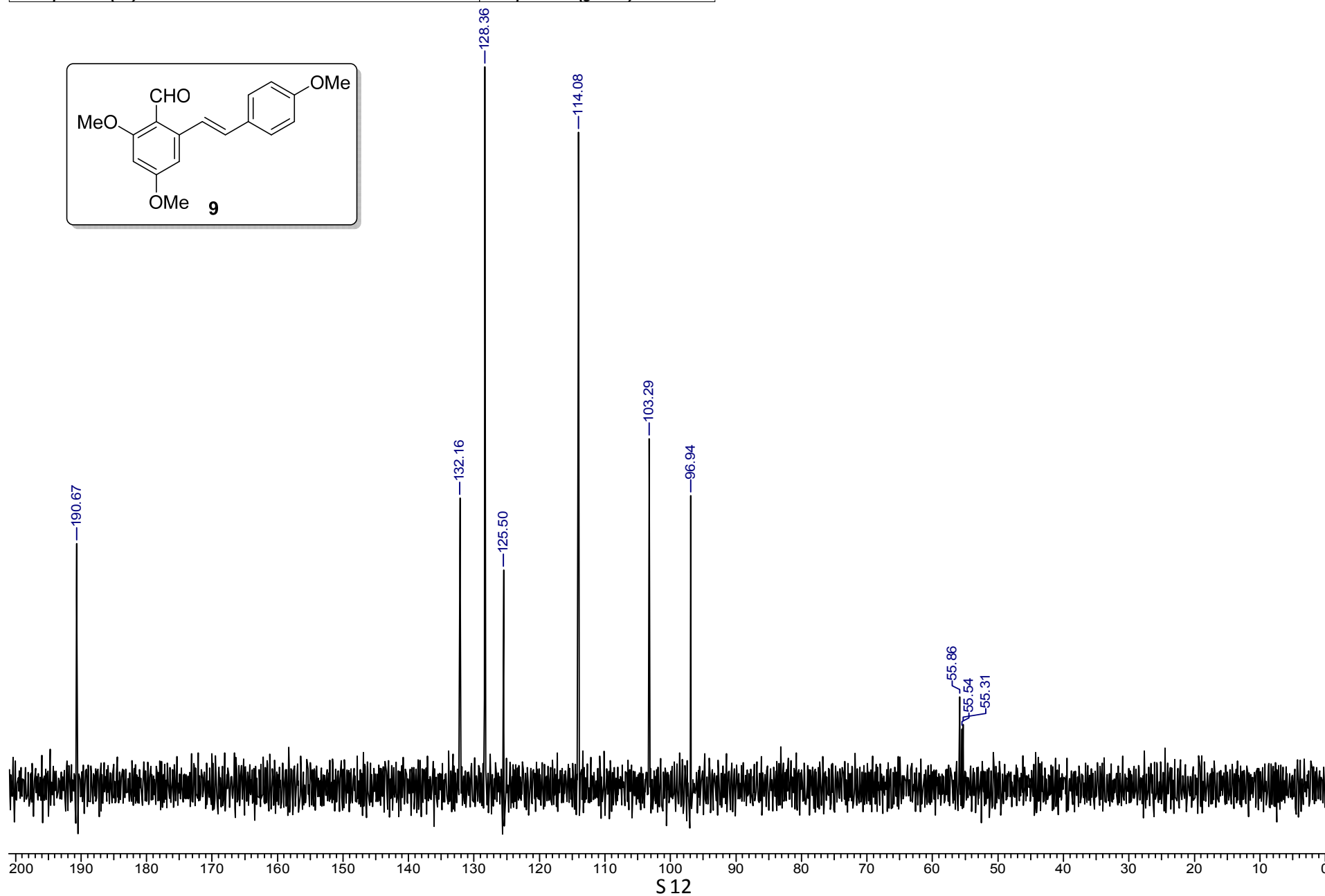
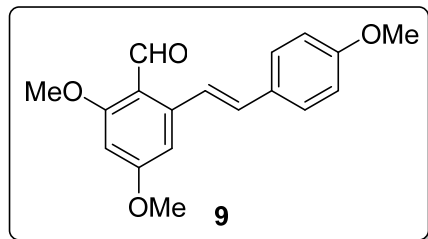


Figure S12: HRMS spectrum of compound 9

DP-873 #140 RT: 0.62 AV: 1 NL: 9.60E8
T: FTMS + p ESI Full ms [100.00-1500.00]

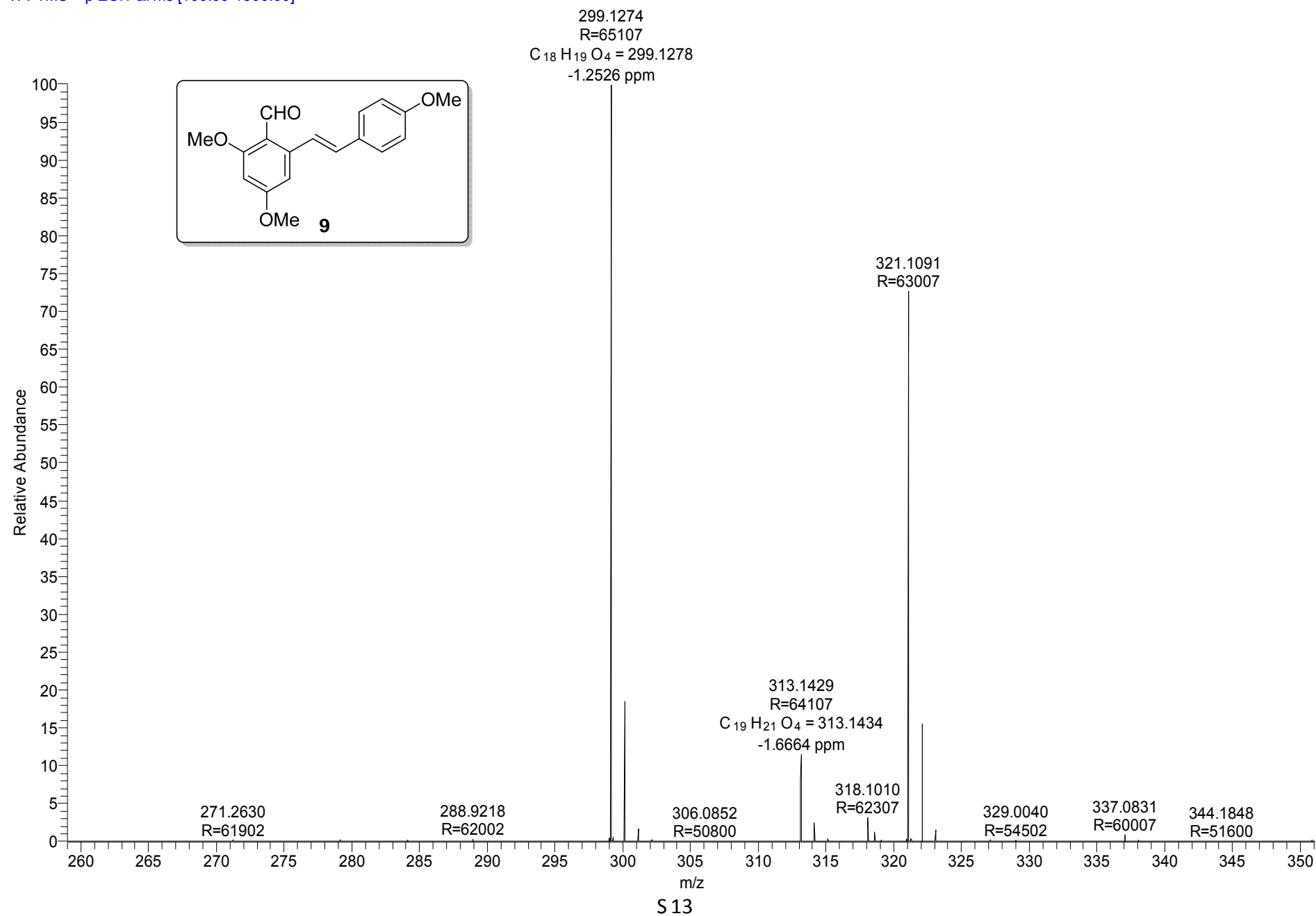


Figure S13: ¹H NMR of compound 13

Acquisition Time (sec)	3.9584	Comment	Dinesh	Date	05 Nov 2016 14:54:00	Frequency (MHz)	200.13
Nucleus	1H	Number of Transients	32	Original Points Count	16384	Points Count	32768
Sweep Width (Hz)	4138.95	Temperature (grad C)	27.000	Solvent	CHLOROFORM-d		

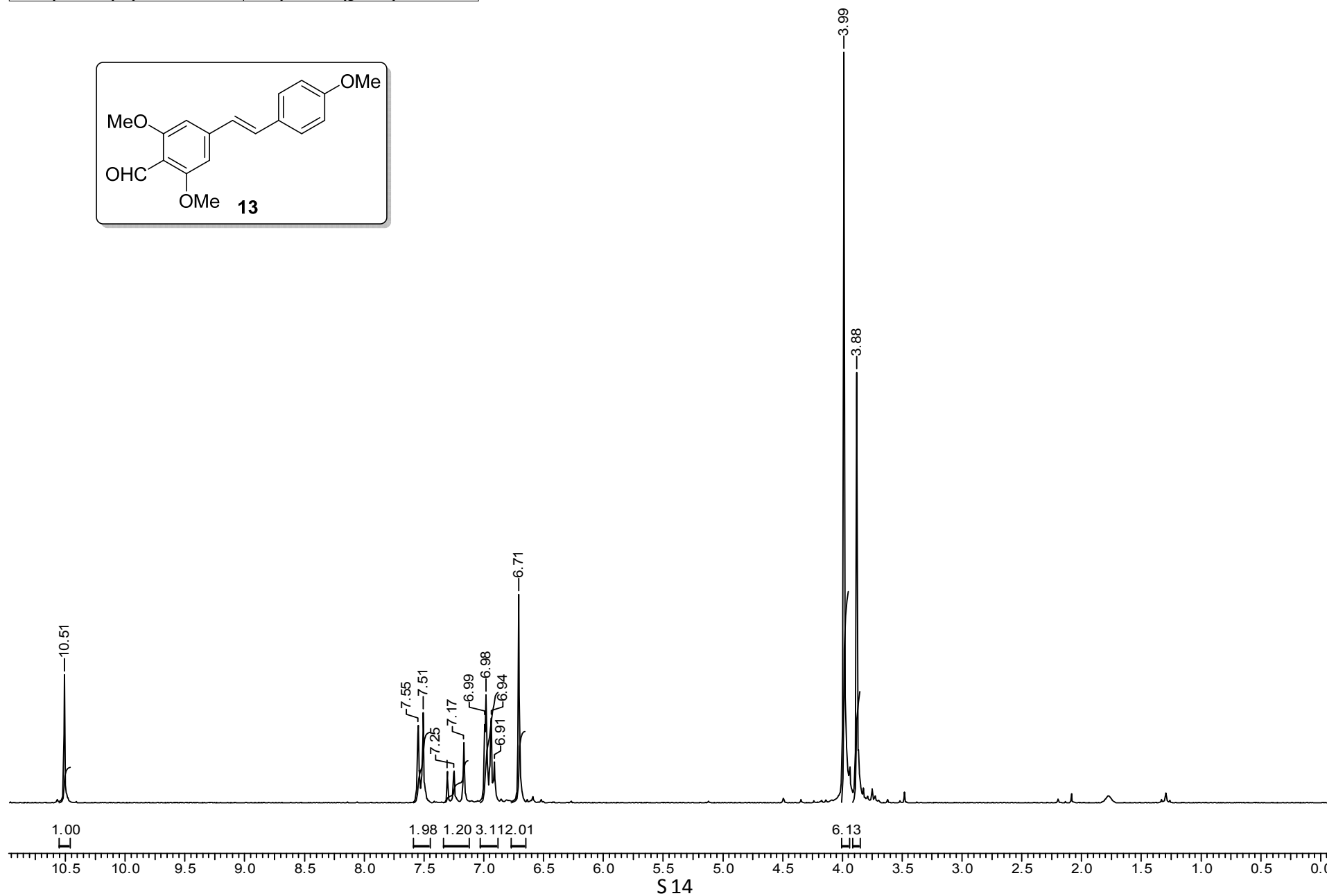
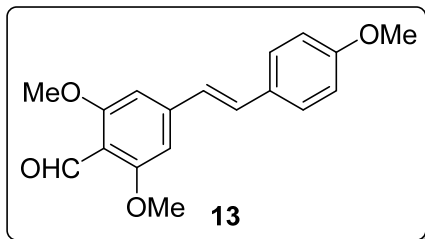


Figure S14: ^{13}C NMR of compound 13

Acquisition Time (sec)	0.6832	Comment	Dinesh	Date	09 Nov 2016 08:51:20	Frequency (MHz)	50.32
Nucleus	^{13}C	Number of Transients	3000	Original Points Count	8192	Points Count	32768
Sweep Width (Hz)	11990.04	Temperature (grad C)	27.000	Solvent	CHLOROFORM-d		

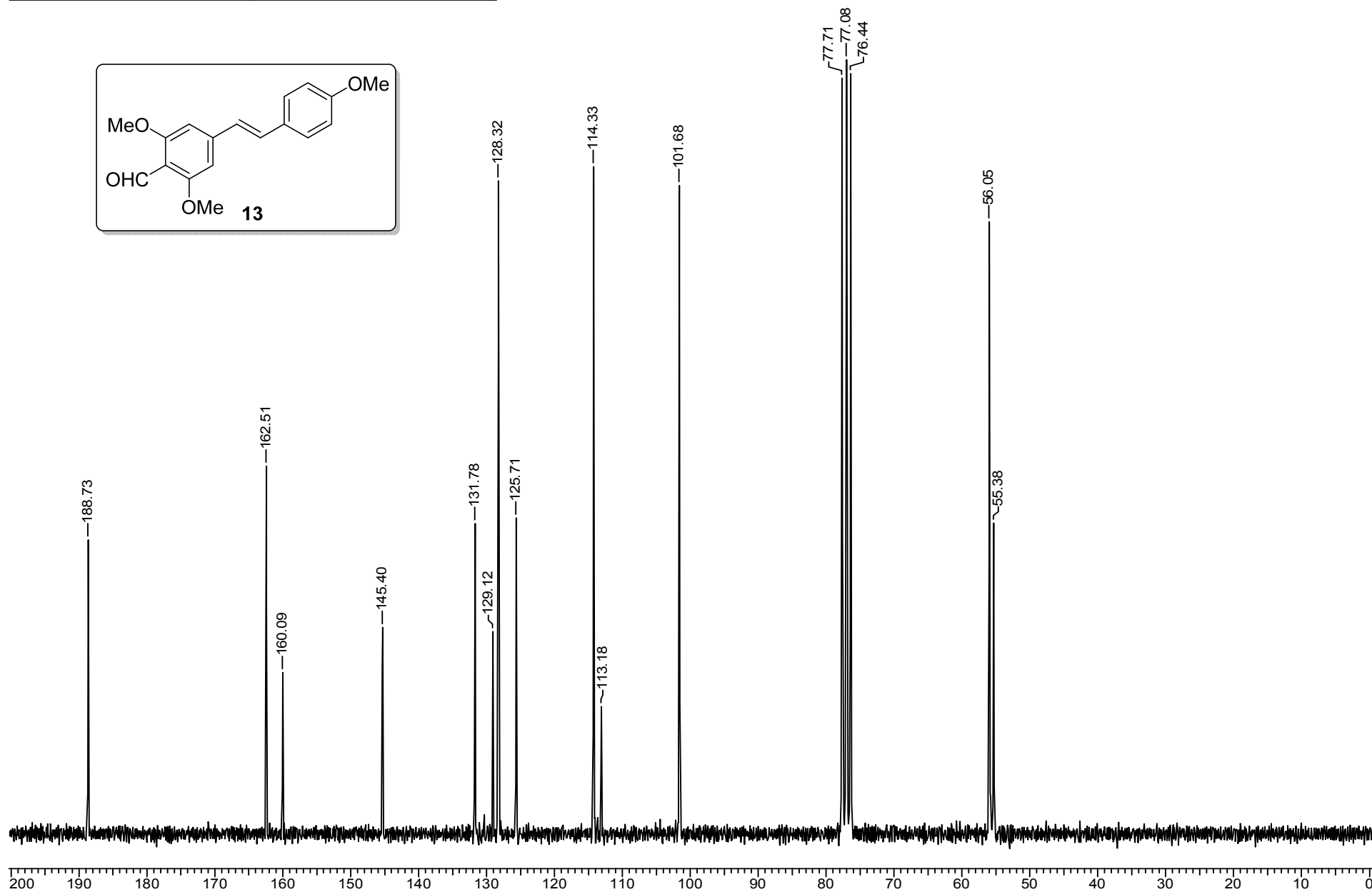
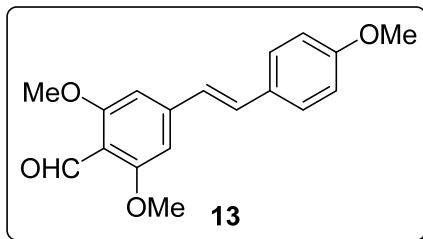


Figure S15: DEPT ^{13}C NMR of compound 13

Acquisition Time (sec)	0.6832	Comment	Dinesh	Date	09 Nov 2016 06:34:48	Frequency (MHz)	50.32
Nucleus	^{13}C	Number of Transients	800	Original Points Count	8192	Points Count	32768
Sweep Width (Hz)	11990.04	Temperature (grad C)	27.000	Solvent	CHLOROFORM-d		

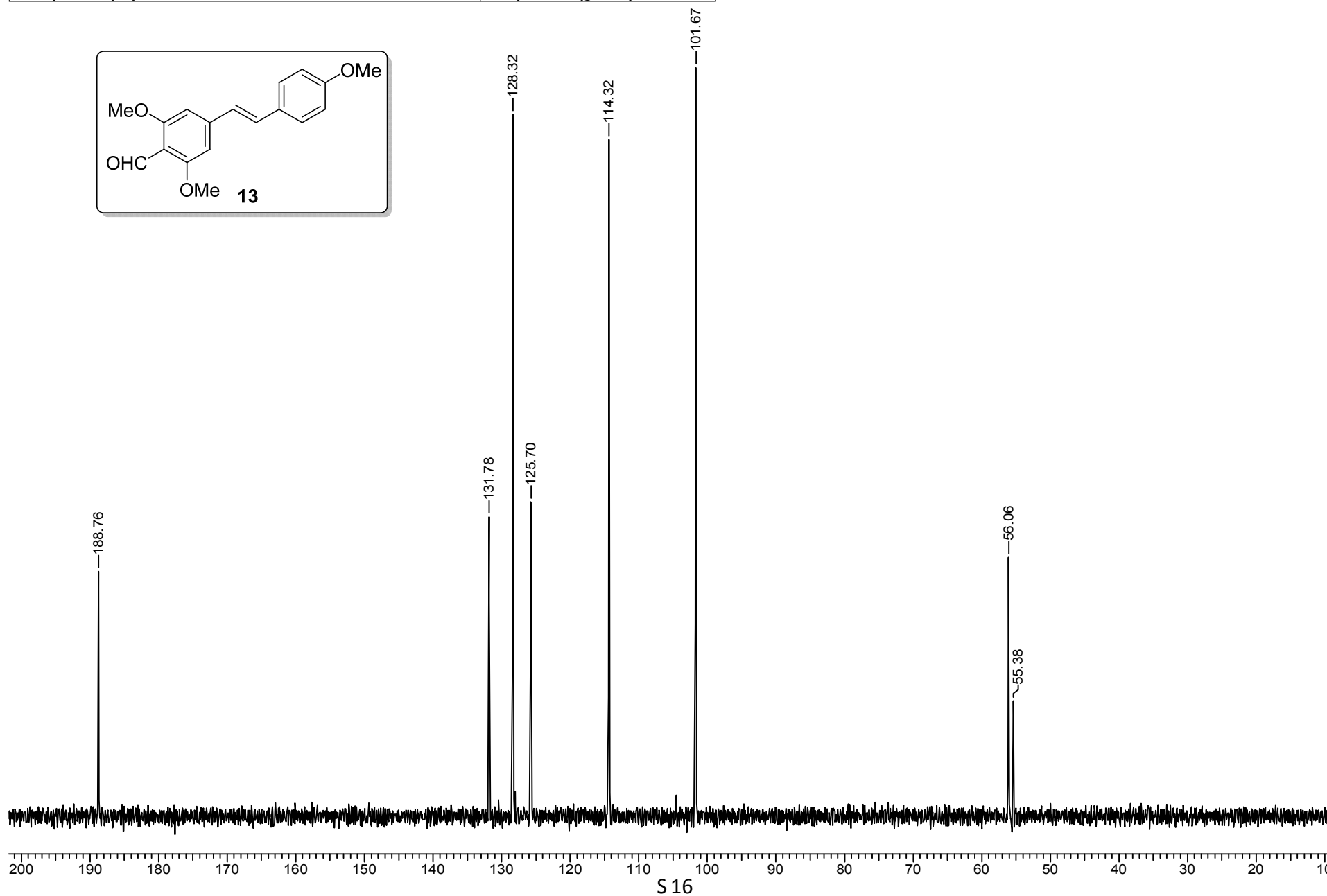
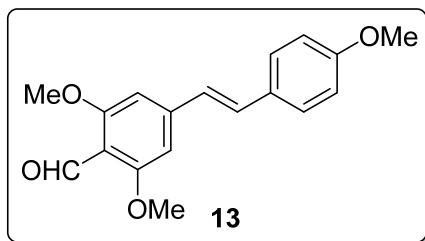


Figure S16: HRMS spectrum of compound 13

DP-873-2 #285 RT: 1.27 AV: 1 NL: 8.34E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

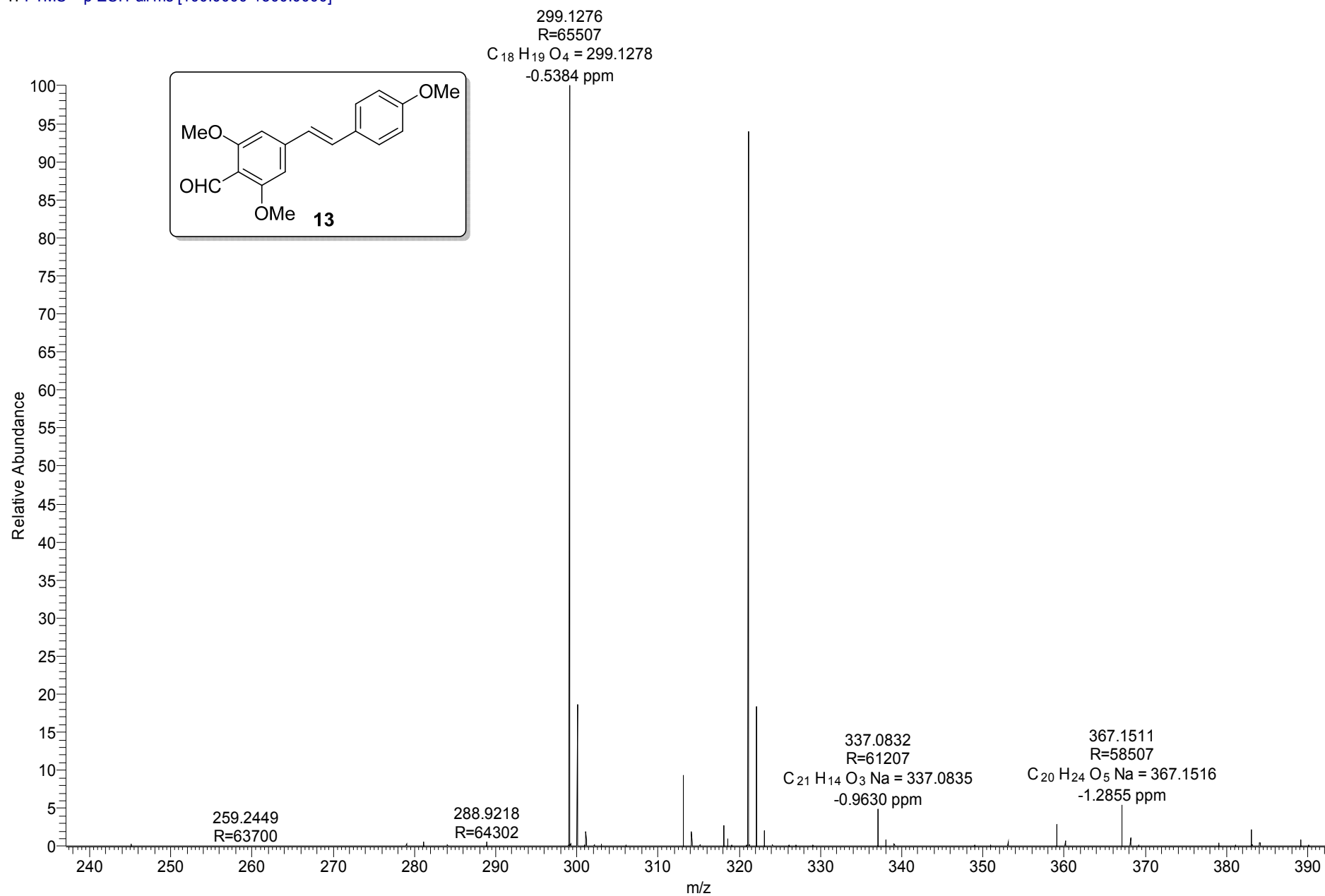


Figure S17: ^1H NMR of compound 15

Acquisition Time (sec)	3.9584	Comment	Dinesh	Date	26 Sep 2017 00:40:40	Frequency (MHz)	200.13
Nucleus	^1H	Number of Transients	4	Original Points Count	16384	Points Count	32768
Sweep Width (Hz)	4138.95	Temperature (grad C)	27.000	Solvent	Acetone		

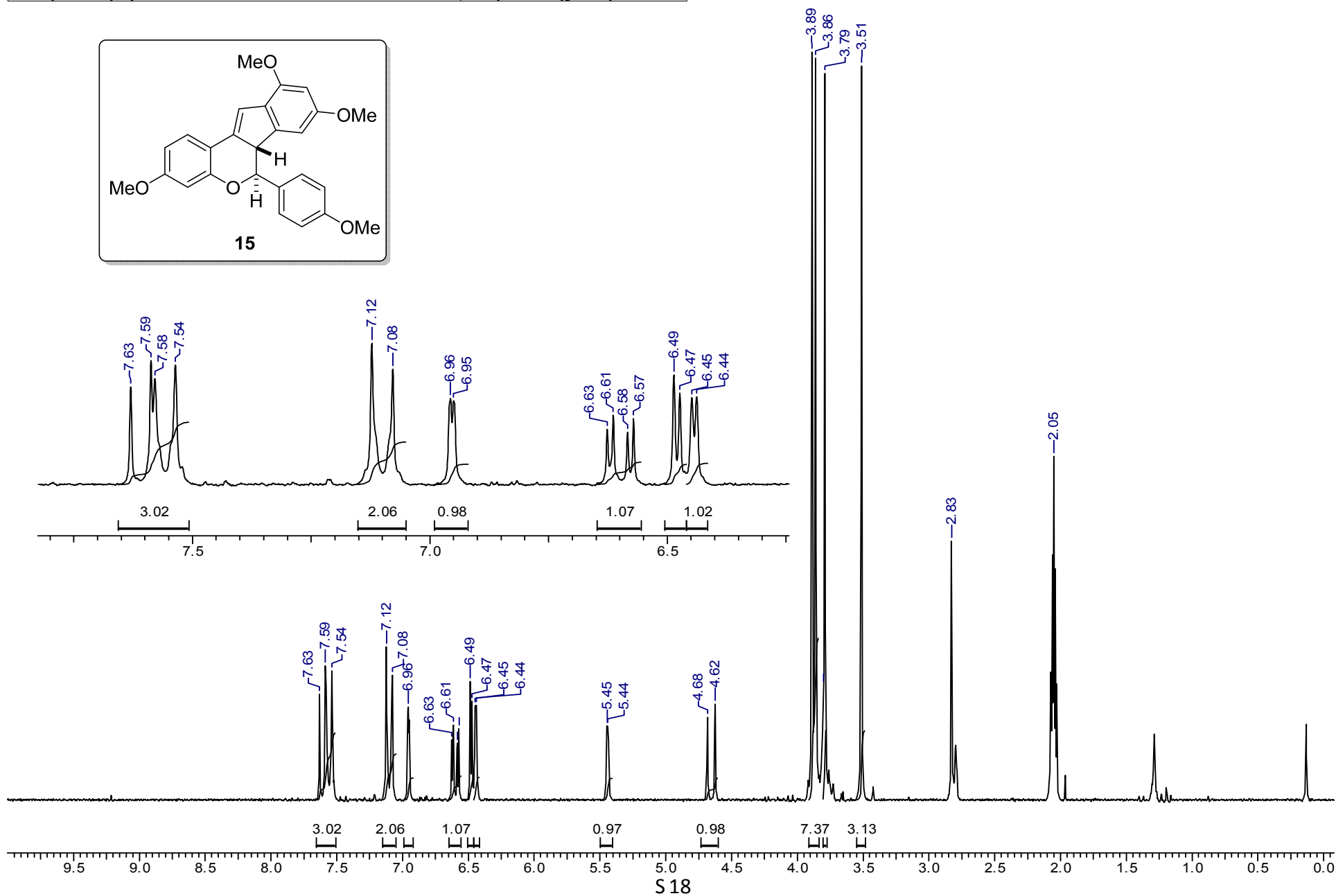
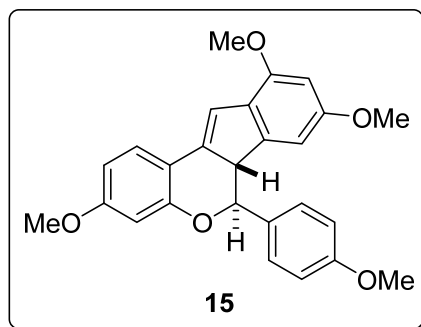


Figure S18: ^{13}C NMR of compound 15

Acquisition Time (sec)	0.6554	Comment	13C	Date	20 Sep 2016 13:58:56	Frequency (MHz)	125.76
Nucleus	^{13}C	Number of Transients	1234	Original Points Count	20480	Points Count	32768
Sweep Width (Hz)	31249.05	Temperature (grad C)	25.000	Solvent	Acetone		

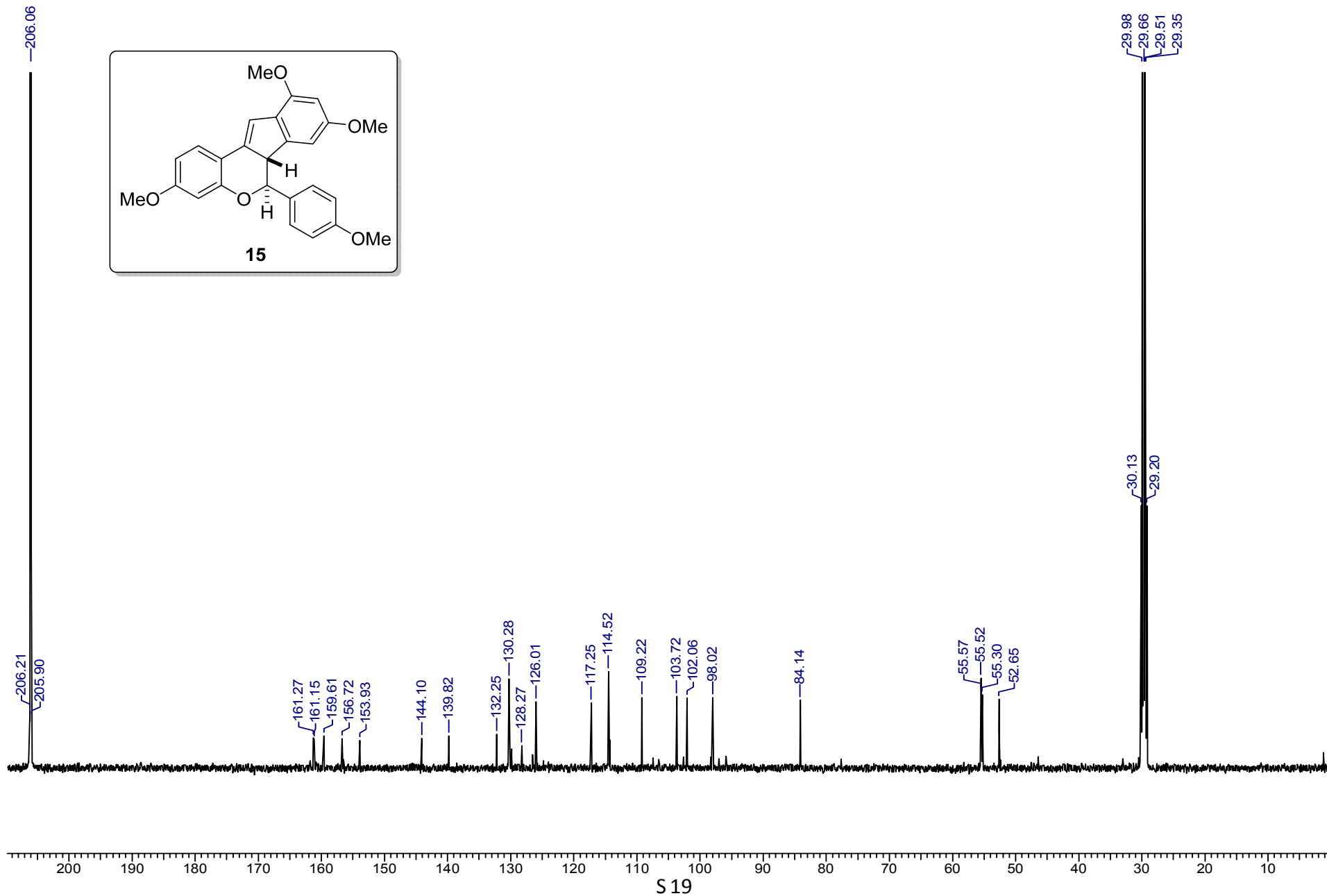


Figure S19: DEPT ^{13}C NMR of compound 15

Acquisition Time (sec)	0.6554	Comment	DEPT	Date	20 Sep 2016 13:24:48	Frequency (MHz)	125.76
Nucleus	^{13}C	Number of Transients	800	Original Points Count	20480	Points Count	32768
Sweep Width (Hz)	31249.05	Temperature (grad C)	25.000	Solvent	Acetone		

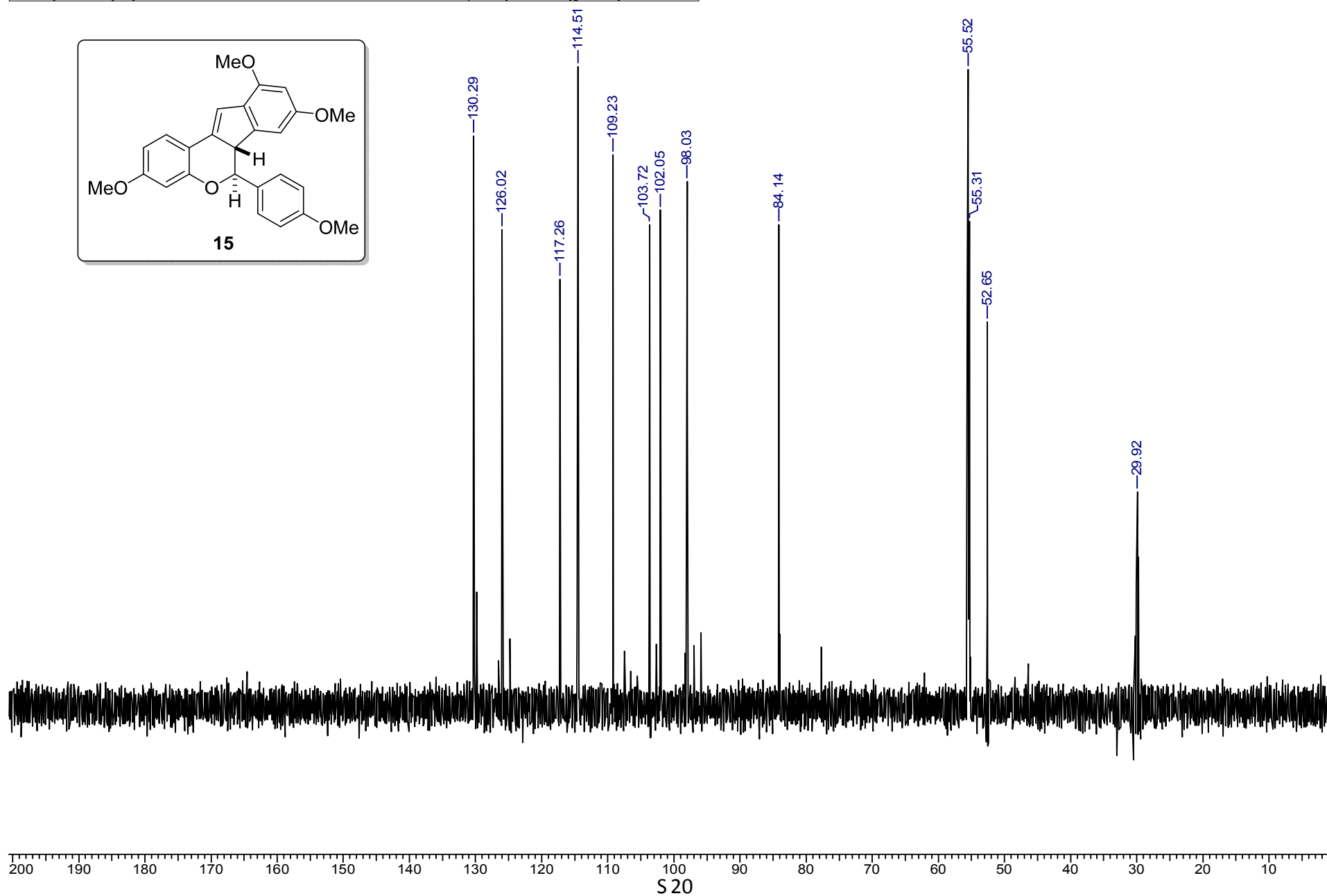
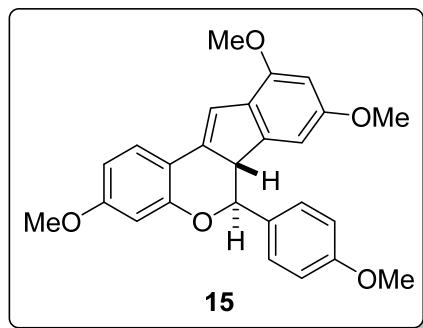


Figure S20: NOESY spectrum of compound 15

Tue4av500#020_11285.005.001.2rr.esp

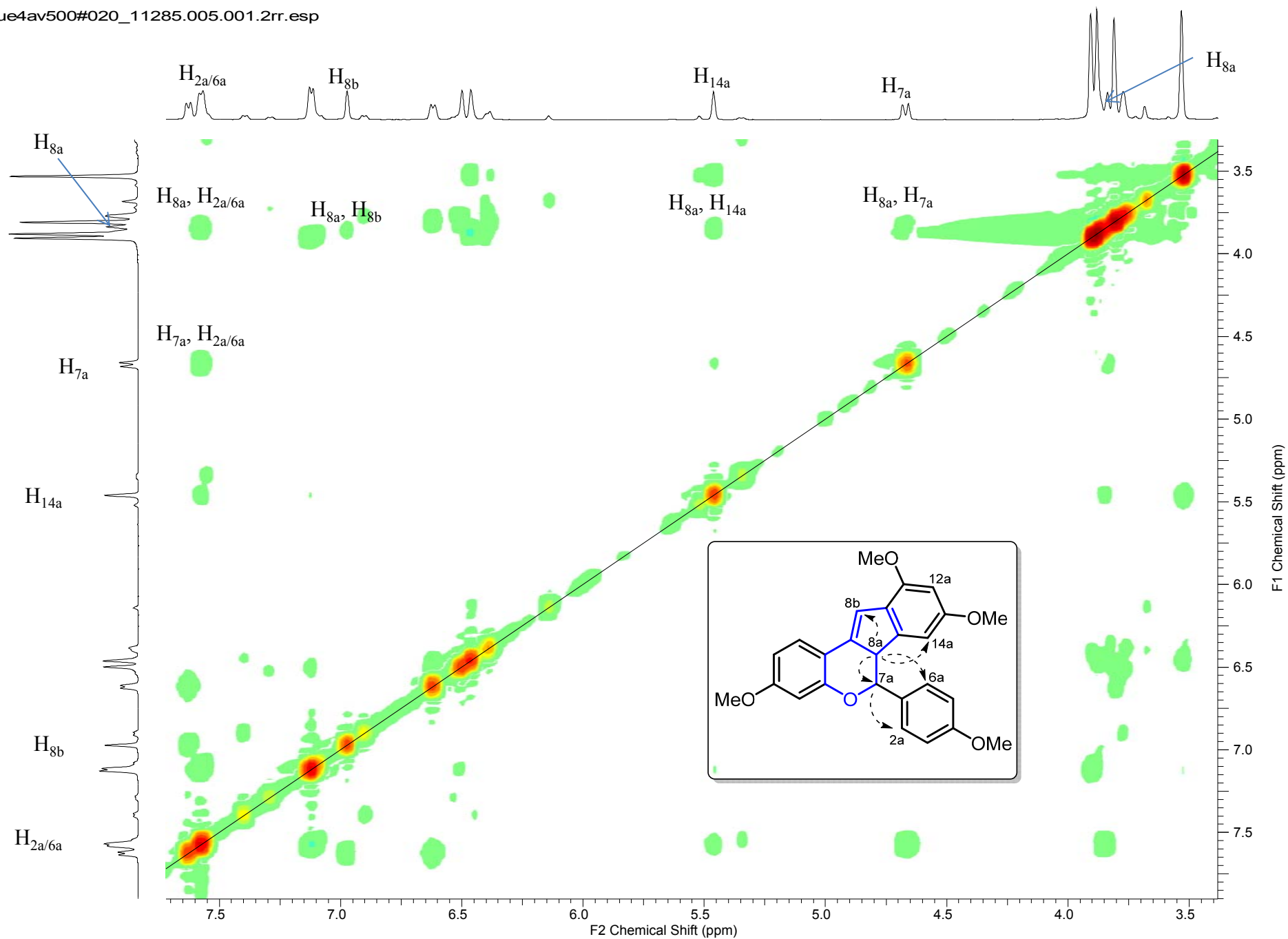


Figure S21: HMBC spectrum of compound 15

Tue4av500#020_11285.007.001.2rr.esp

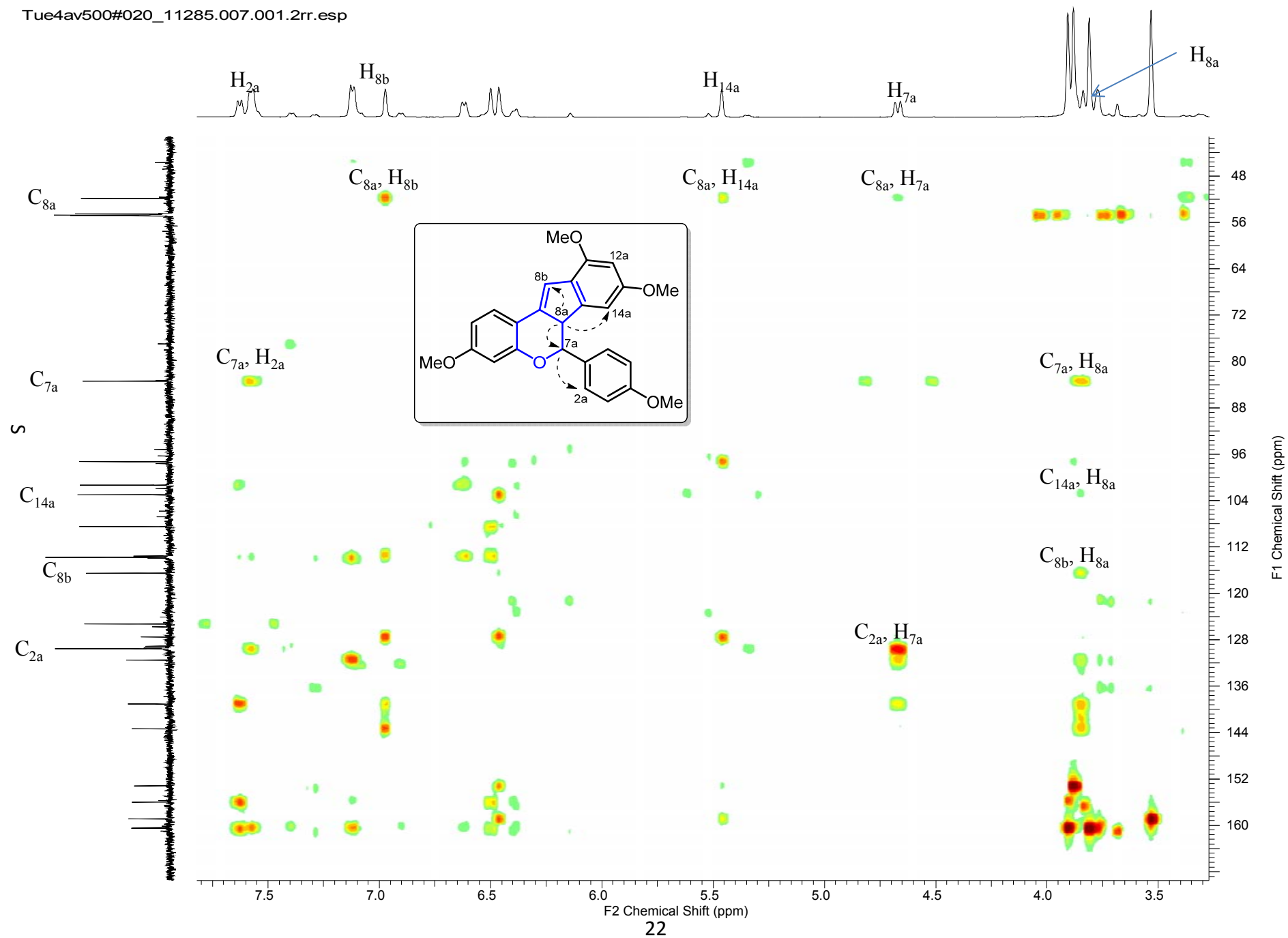


Figure S22: HRMS spectrum of compound 15

DP-843 #192 RT: 0.85 AV: 1 NL: 3.97E8
T: FTMS + p ESI Full ms [100.00-1500.00]

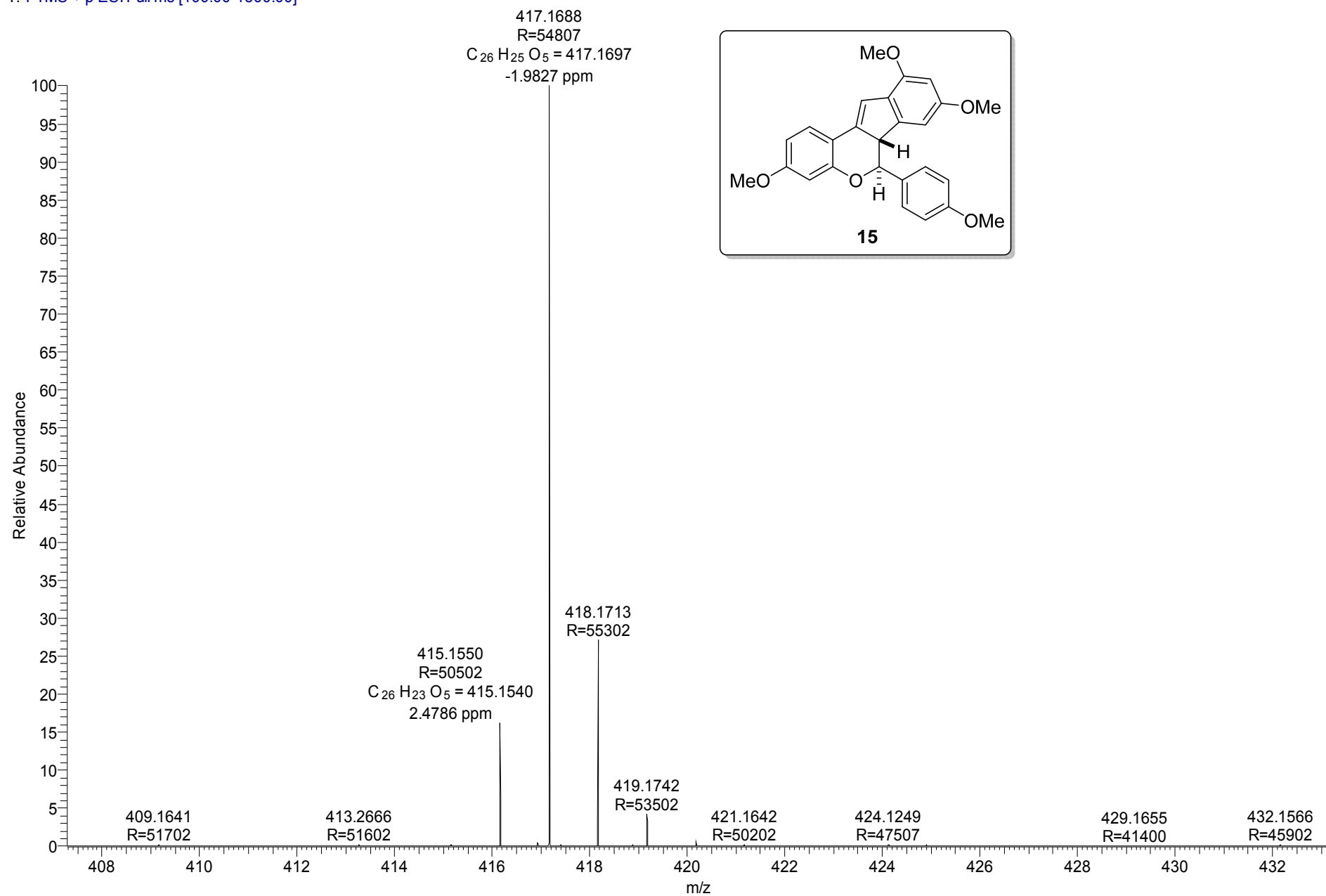


Figure S23: ¹H NMR of compound 17

Acquisition Time (sec)	1.2800	Comment	Dinesh 1H	Date	08 Mar 2017 12:46:08
Frequency (MHz)	500.13	Nucleus	1H	Number of Transients	64
Solvent	CHLOROFORM-d			Original Points Count	16000
				Points Count	32768
				Sweep Width (Hz)	12499.62
				Temperature (grad C)	22.400

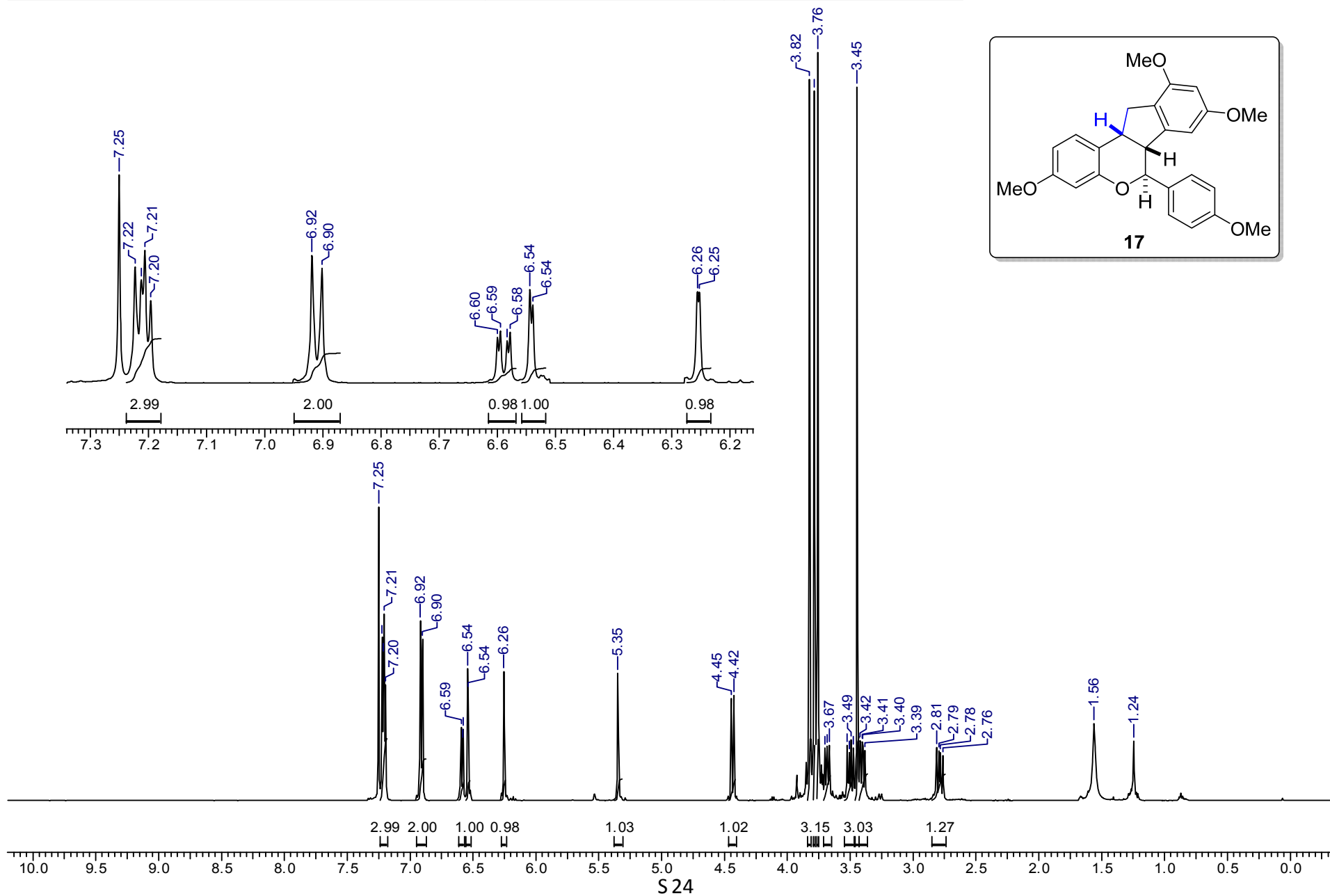


Figure S24: ^{13}C NMR of compound 17

Acquisition Time (sec)	0.6554	Comment	13C	Date	08 Mar 2017 14:05:04	Frequency (MHz)	125.76
Nucleus	^{13}C	Number of Transients	2041	Original Points Count	20480	Points Count	32768
Sweep Width (Hz)	31249.05	Temperature (grad C)	23.700	Solvent	CHLOROFORM-d		

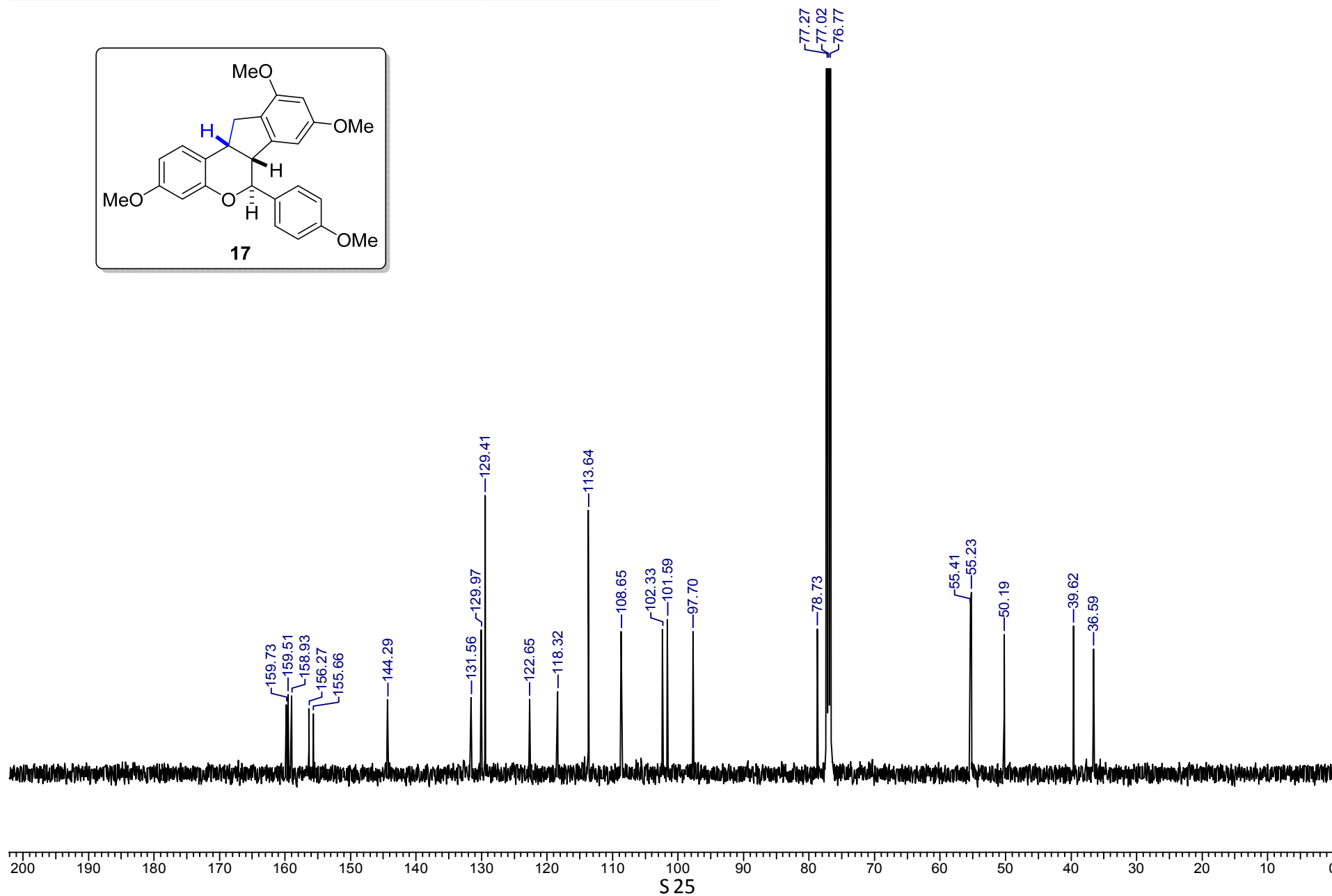
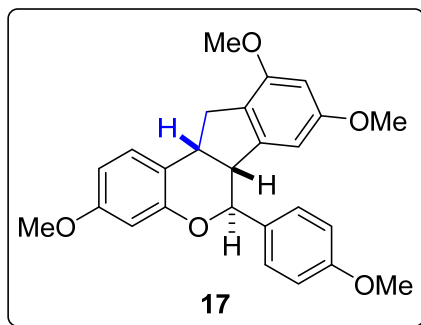


Figure S25: DEPT ^{13}C NMR of compound 17

Acquisition Time (sec)	0.6554	Comment	DEPT	Date	08 Mar 2017 13:07:28	Frequency (MHz)	125.76
Nucleus	^{13}C	Number of Transients	800	Original Points Count	20480	Points Count	32768
Sweep Width (Hz)	31249.05	Temperature (grad C)	22.900	Solvent	CHLOROFORM-d		

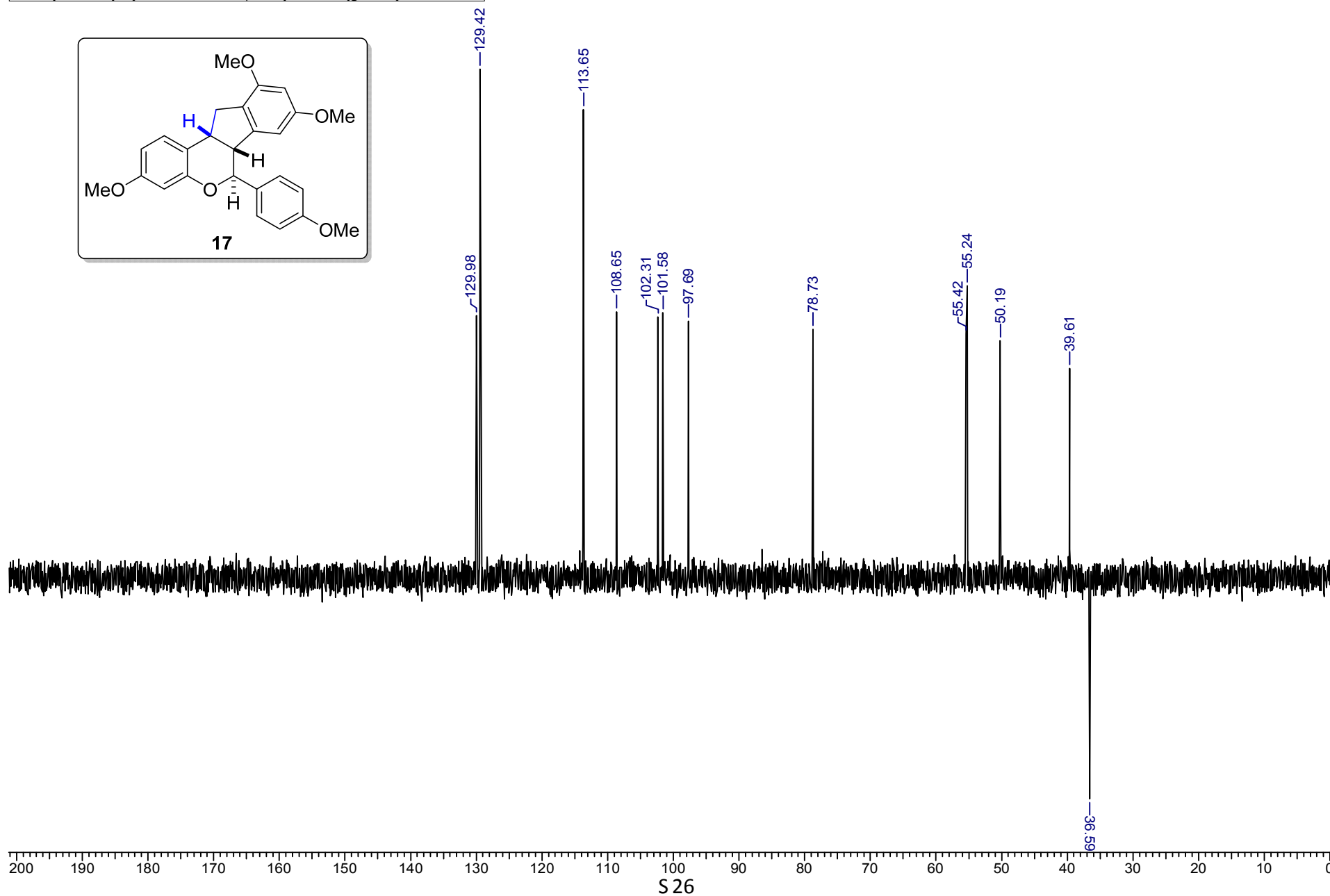
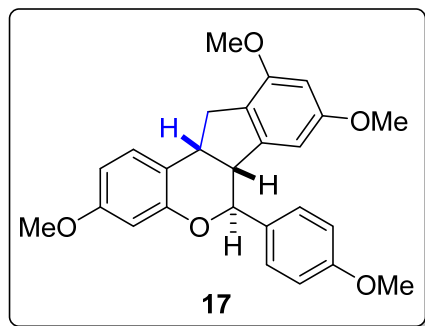


Figure S26: HRMS spectrum of compound 17

PJ 61

PJ 61 145 (2.668) AM2 (Ar,20000.0,556.28,0.00,LS 3); ABS; Sm (SG, 2x1.00); Cm (144:145)

IISER PUNE

419.1857

1: TOF MS ES+
6.61e4

CALCULATED MASS
(M+H)+419.1858

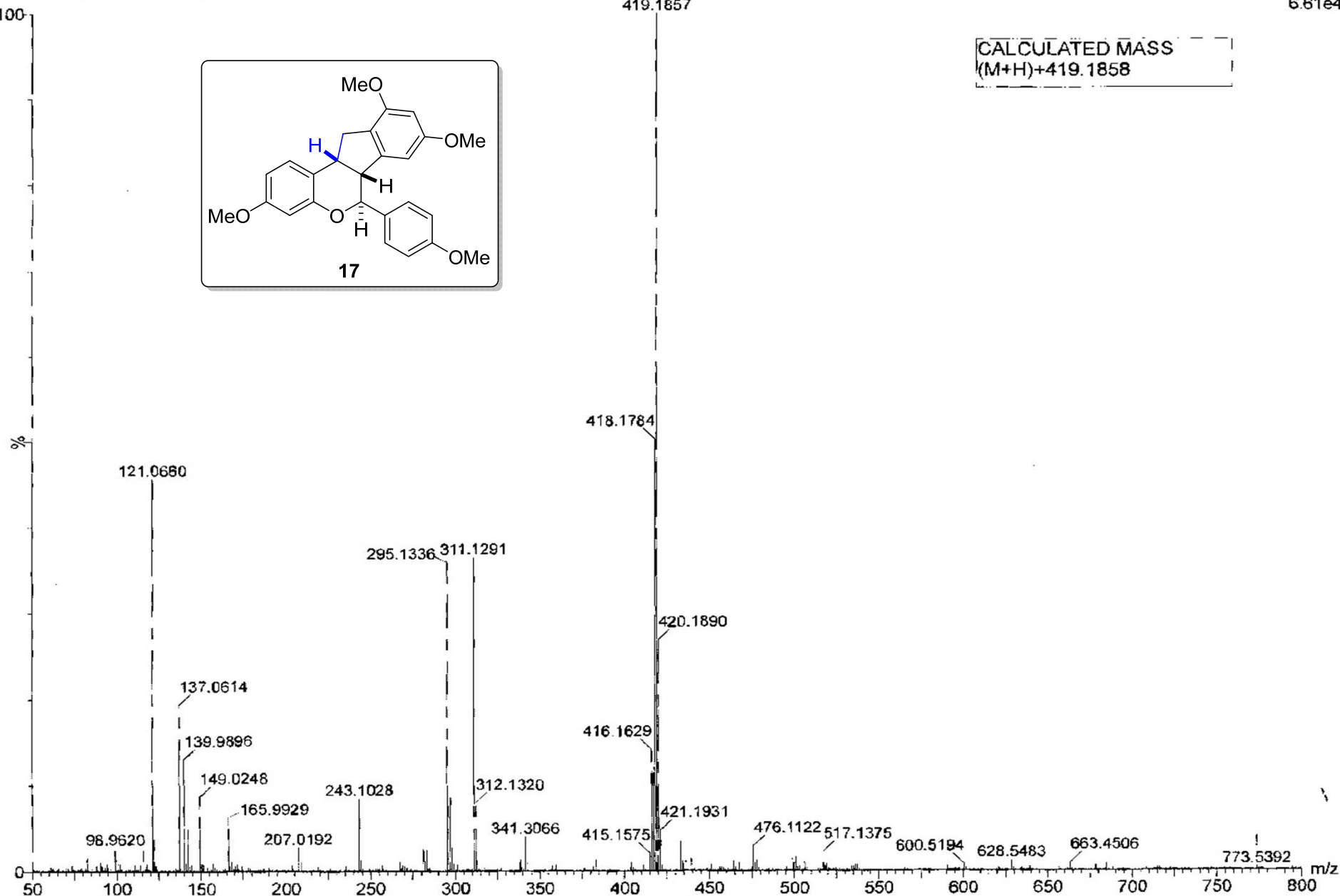
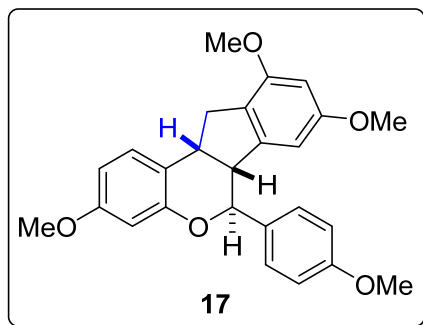


Figure S27: ^1H NMR of compound 18

Acquisition Time (sec)	1.2800	Comment	Dinesh 1H	Date	29 Nov 2016 16:08:48
Frequency (MHz)	500.13	Nucleus	^1H	Number of Transients	64
Solvent	CHLOROFORM-d			Original Points Count	16000
				Points Count	32768
				Sweep Width (Hz)	12499.62
				Temperature (grad C)	24.000

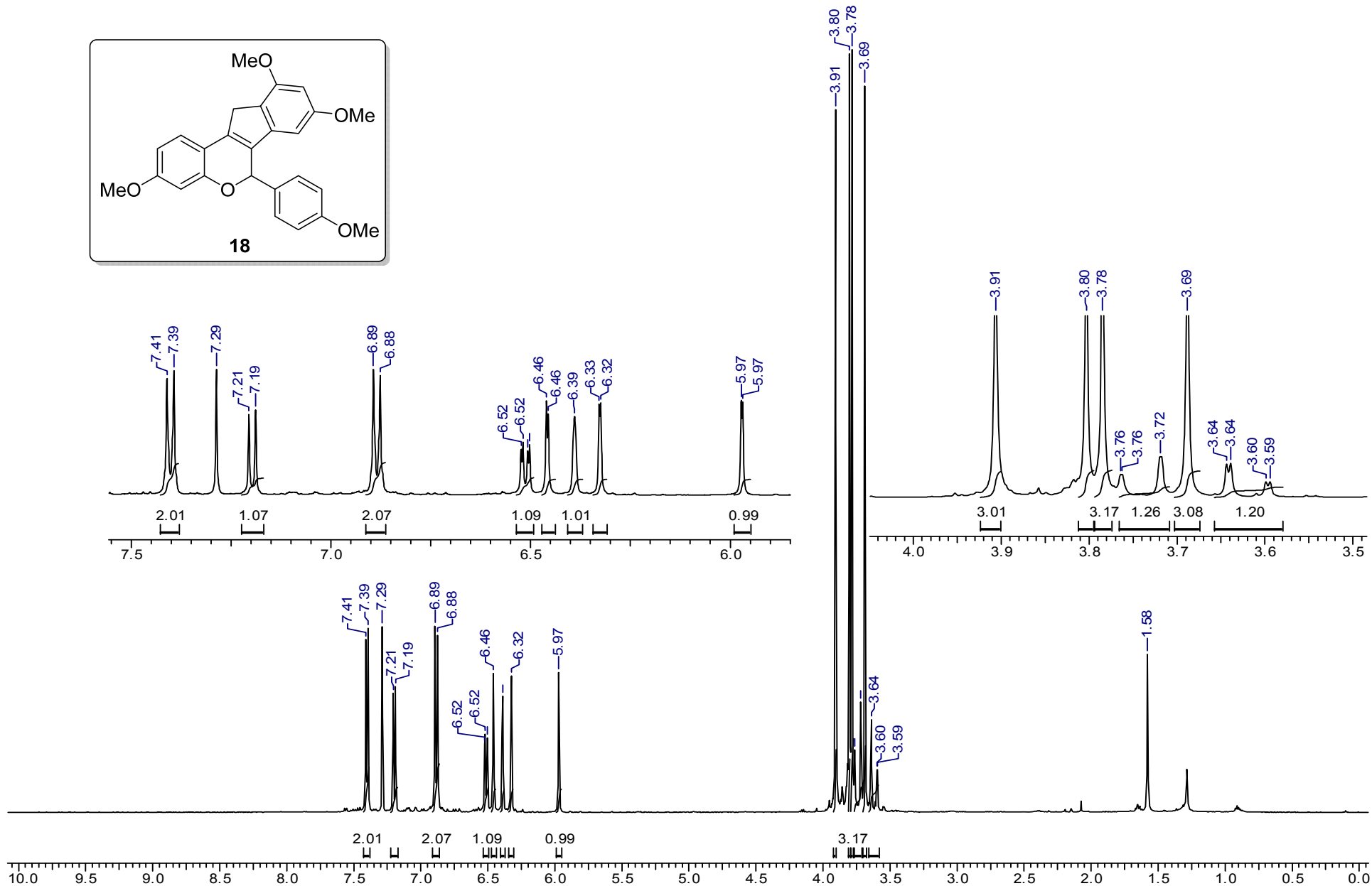
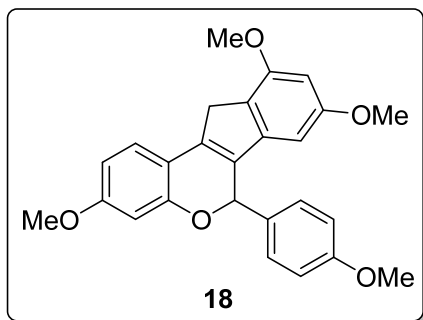


Figure S28: ^{13}C NMR of compound 18

Acquisition Time (sec)	0.6488			
Comment	13C			
Date	01 Dec 2016 13:43:36	Frequency (MHz)	100.61	Nucleus 13C
Number of Transients	1126	Original Points Count	16384	Points Count 32768
Solvent	CHLOROFORM-d	Sweep Width (Hz)	25251.75	Temperature (grad C) 23.400

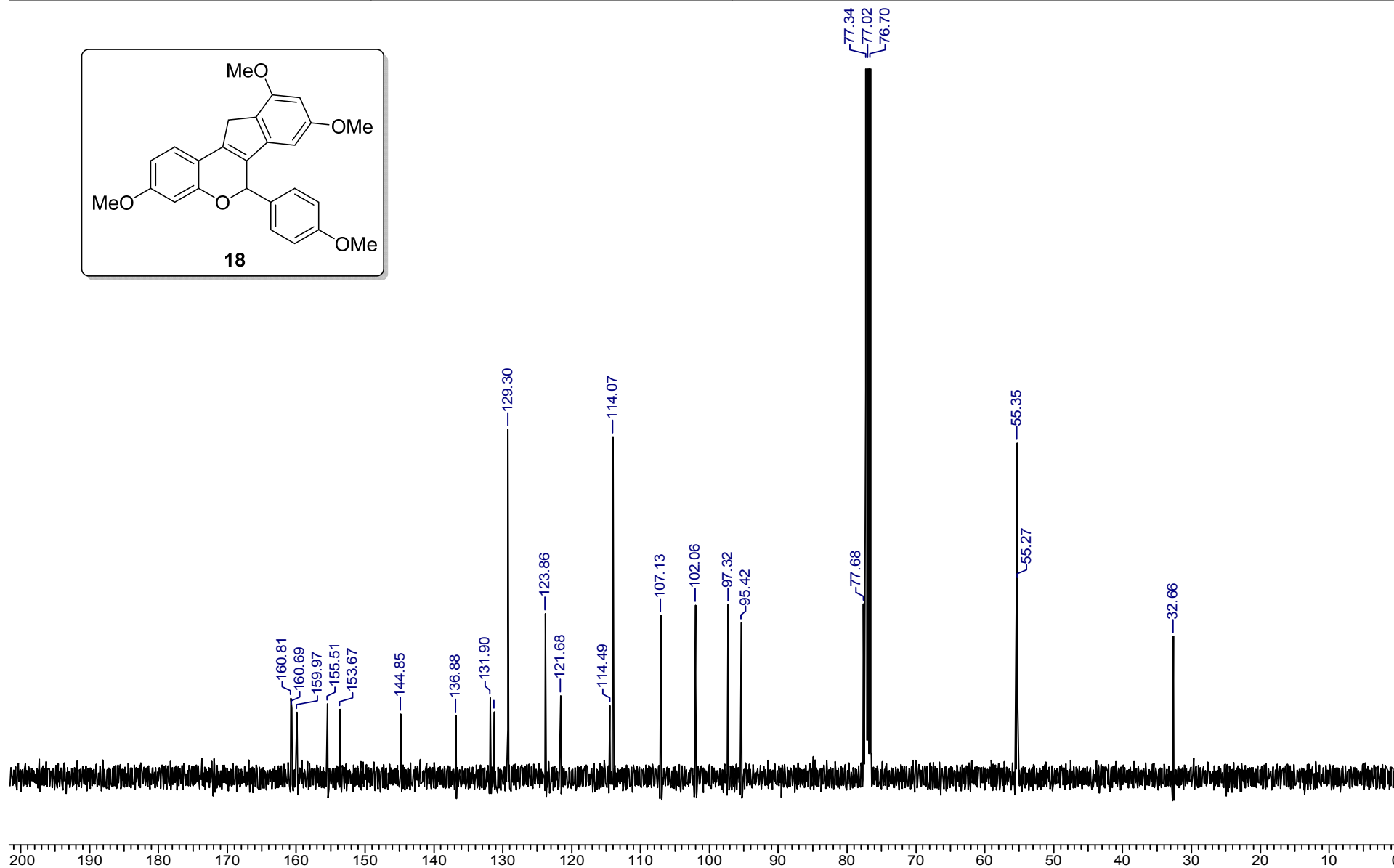
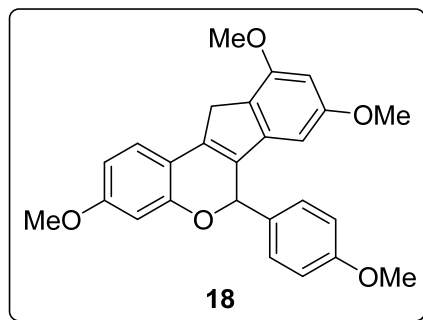


Figure S29: DEPT ^{13}C NMR of compound 18

Acquisition Time (sec)	0.6488				
Comment	DEPT				
Date	01 Dec 2016 13:13:44	Frequency (MHz)	100.61	Nucleus	13C
Number of Transients	800	Original Points Count	16384	Points Count	32768
Solvent	CHLOROFORM-d	Sweep Width (Hz)	25251.75	Temperature (grad C)	22.900

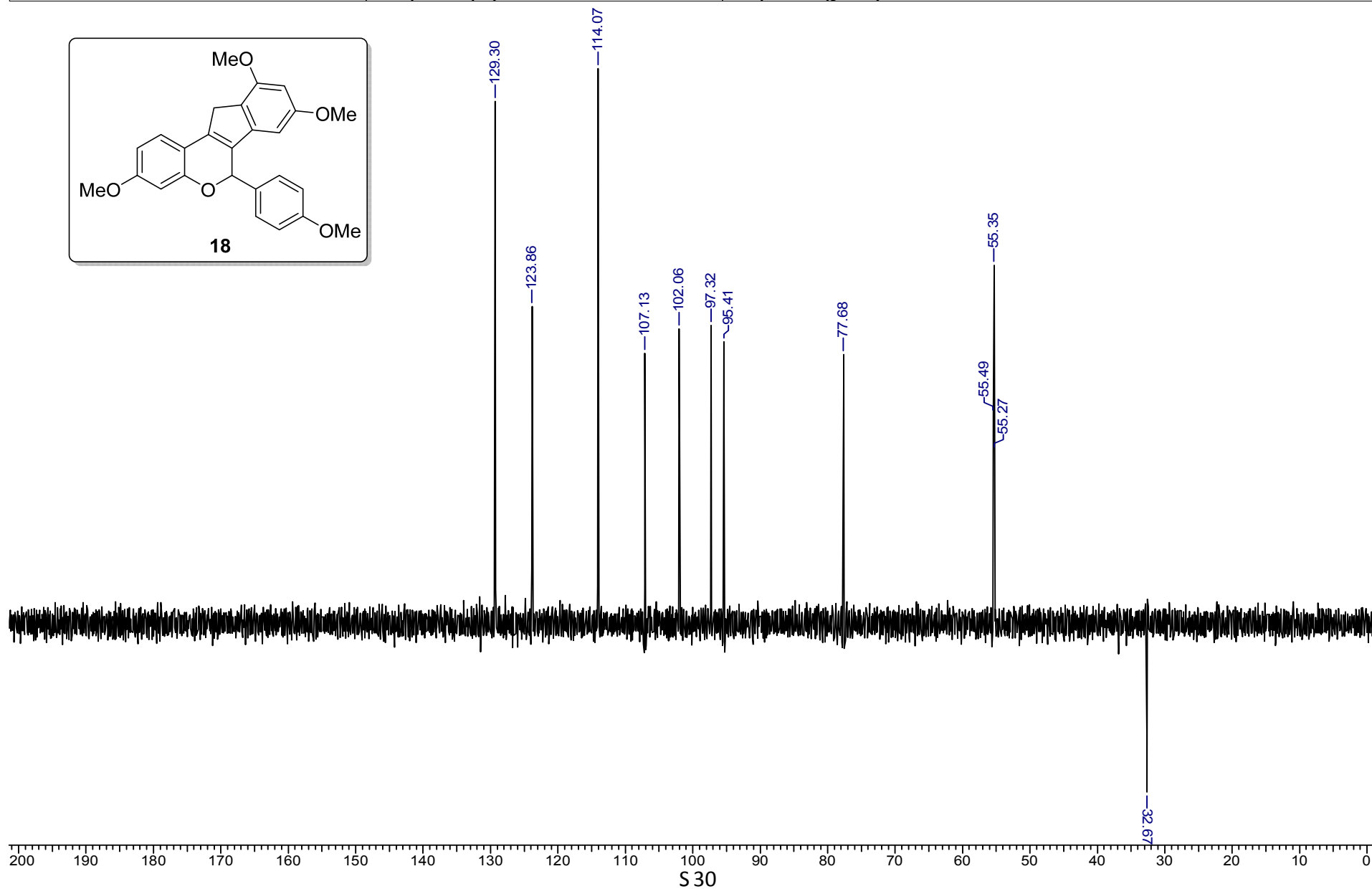
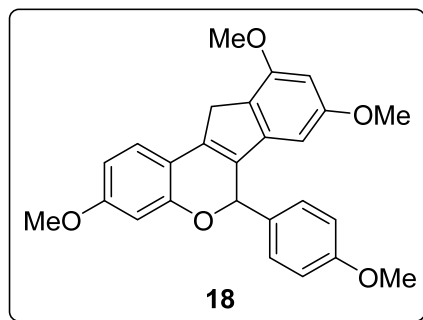


Figure S30: HRMS spectrum of compound 18

DP-887_171123181333 #288 RT: 1.28 AV: 1 NL: 2.07E5
T: FTMS + p ESI Full ms [100.0000-1500.0000]

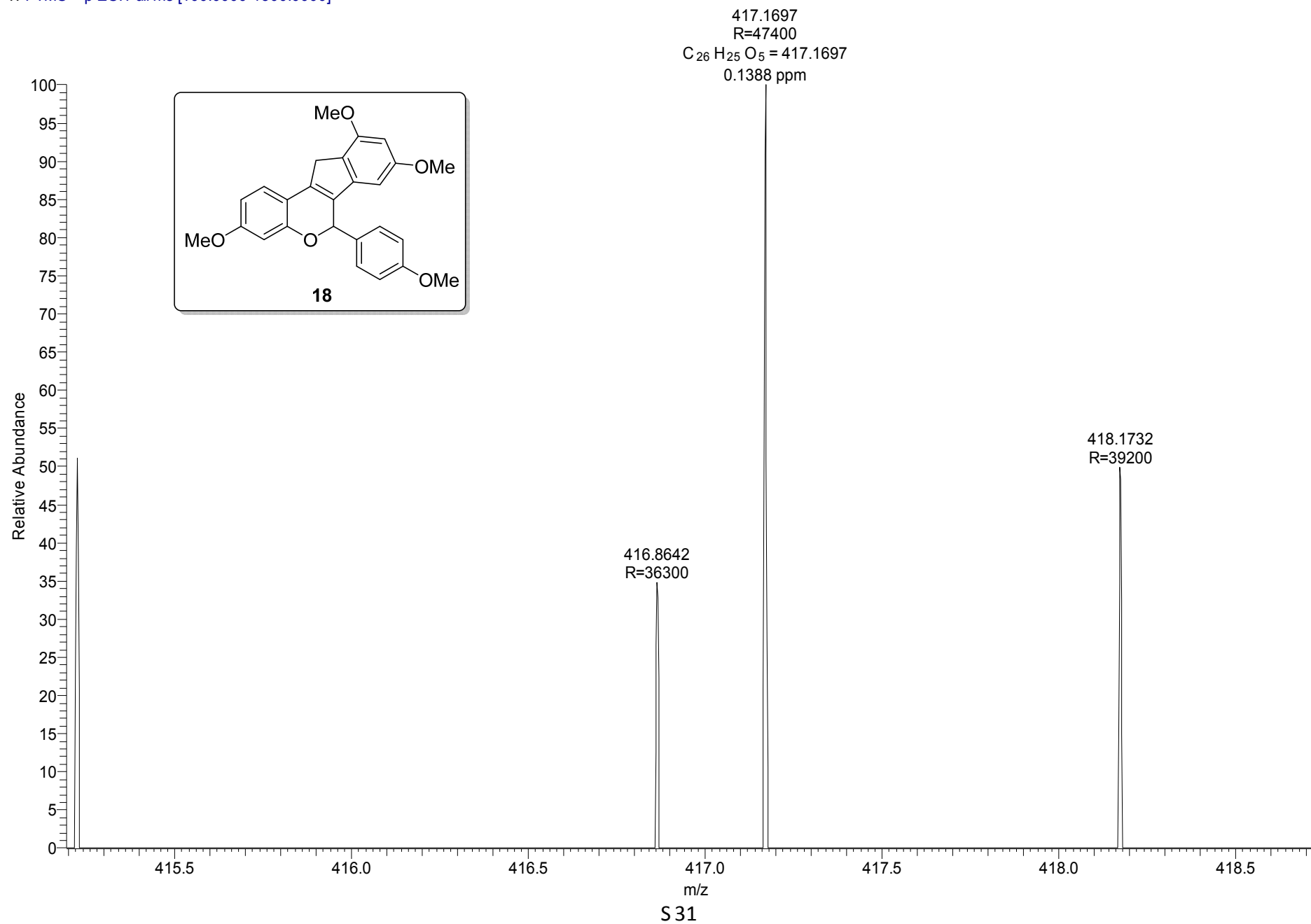


Figure S31: ¹H NMR of compound 19

Acquisition Time (sec)	1.2800	Comment	Dinesh 1H	Date	02 Apr 2017 13:09:52
Frequency (MHz)	500.13	Nucleus	1H	Number of Transients	64
Solvent	CHLOROFORM-d			Original Points Count	16000
				Points Count	32768
				Sweep Width (Hz)	12499.62
				Temperature (grad C)	25.000

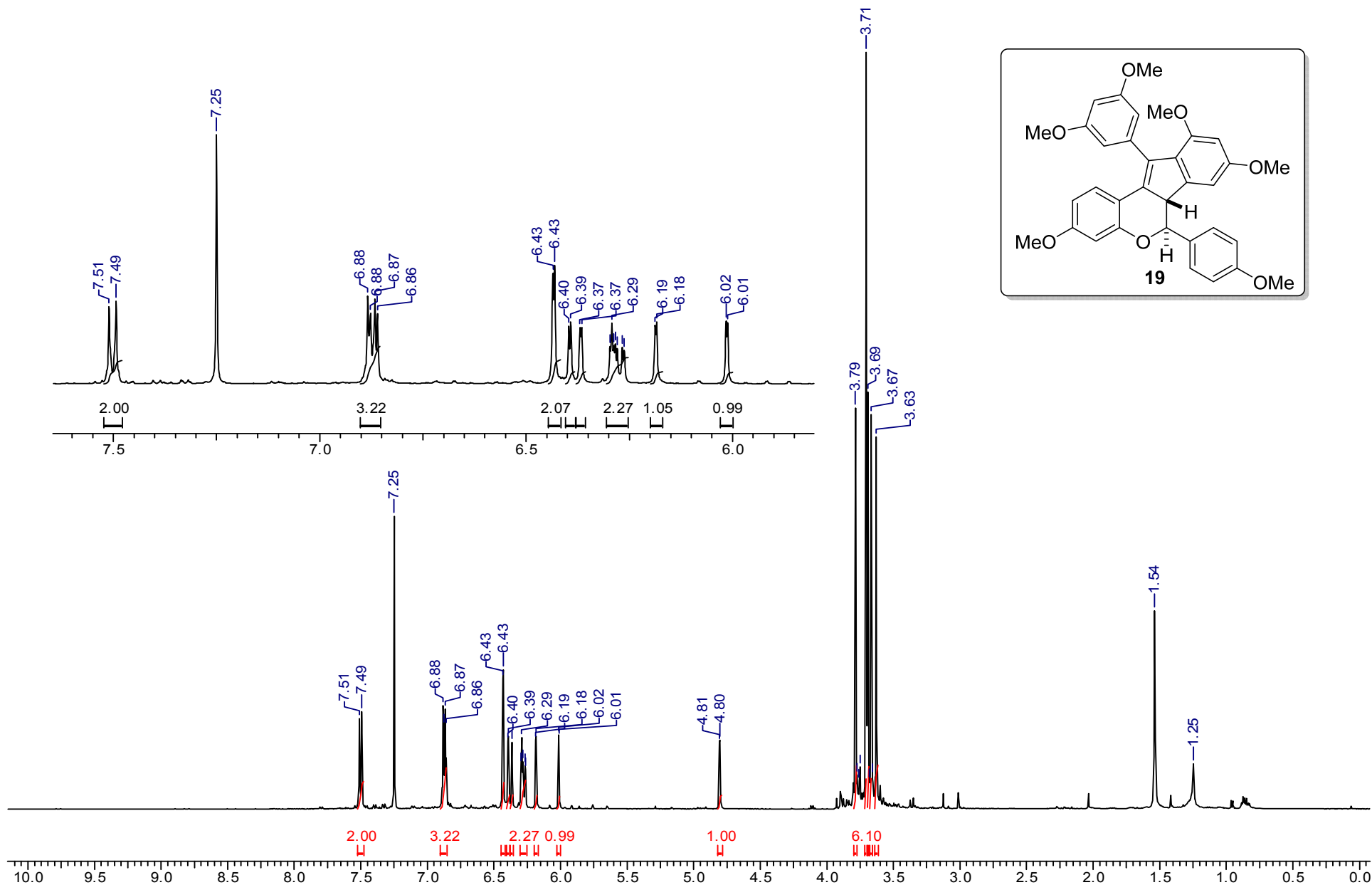
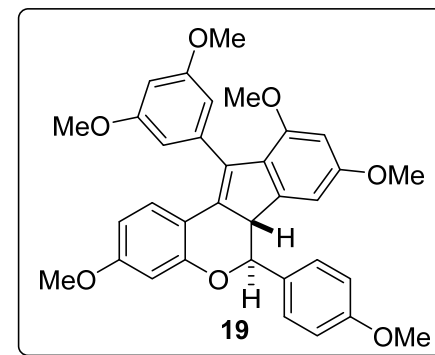


Figure S32: ^{13}C NMR of compound 19

Acquisition Time (sec)	0.6554	Comment	13C	Date	02 Apr 2017 14:01:04	Frequency (MHz)	125.76
Nucleus	^{13}C	Number of Transients	2254	Original Points Count	20480	Points Count	32768
Sweep Width (Hz)	31249.05			Temperature (grad C)	25.000	Solvent	CHLOROFORM-d

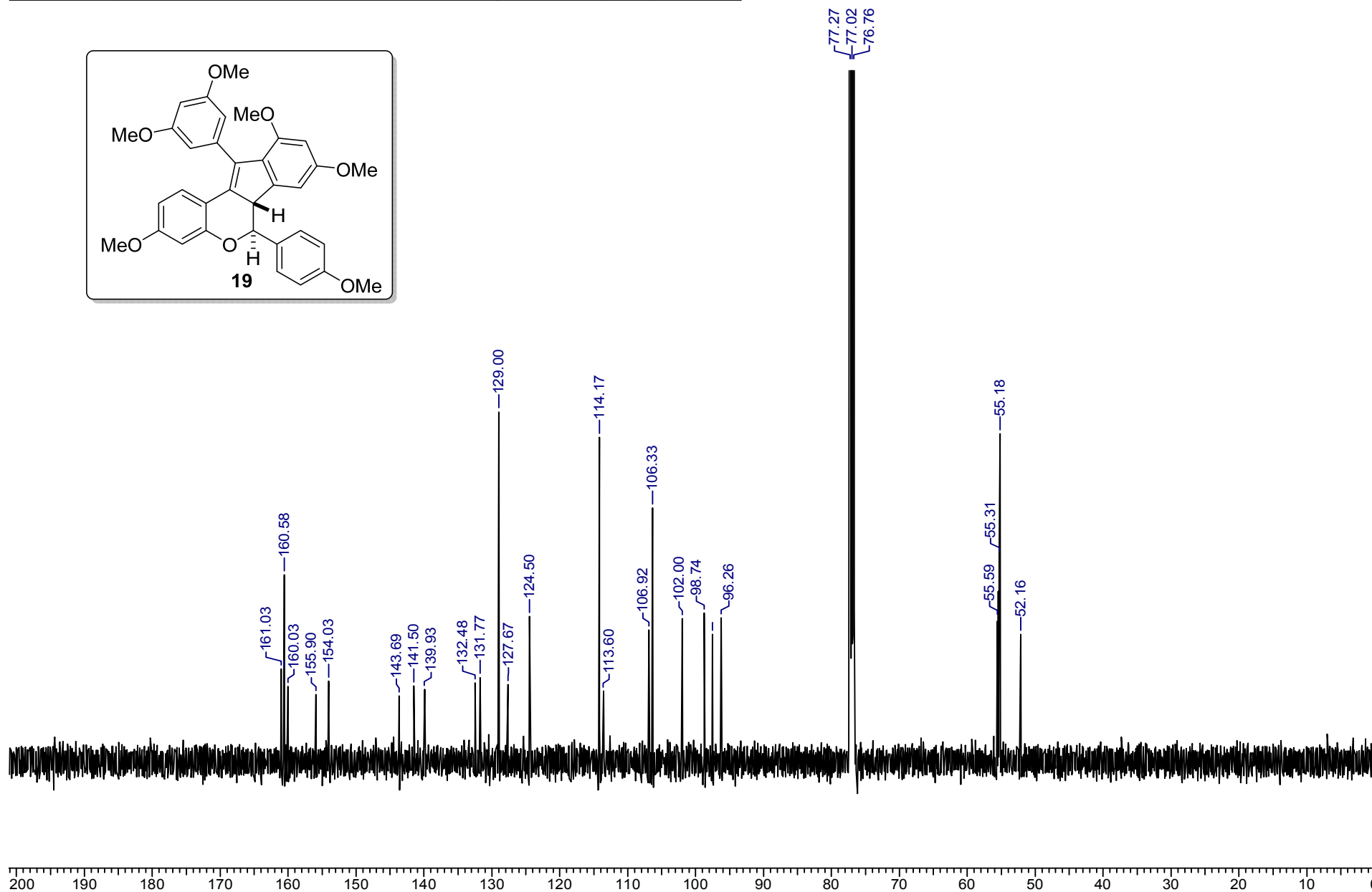
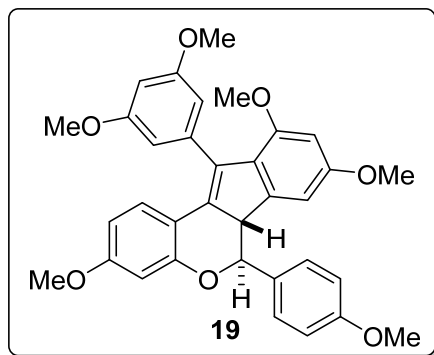


Figure S33: DEPT ^{13}C NMR of compound 19

Acquisition Time (sec)	0.6554	Comment	DEPT	Date	02 Apr 2017 13:31:12	Frequency (MHz)	125.76
Nucleus	^{13}C	Number of Transients	800	Original Points Count	20480	Points Count	32768
Sweep Width (Hz)	31249.05	Temperature (grad C)	25.000	Solvent	CHLOROFORM-d		

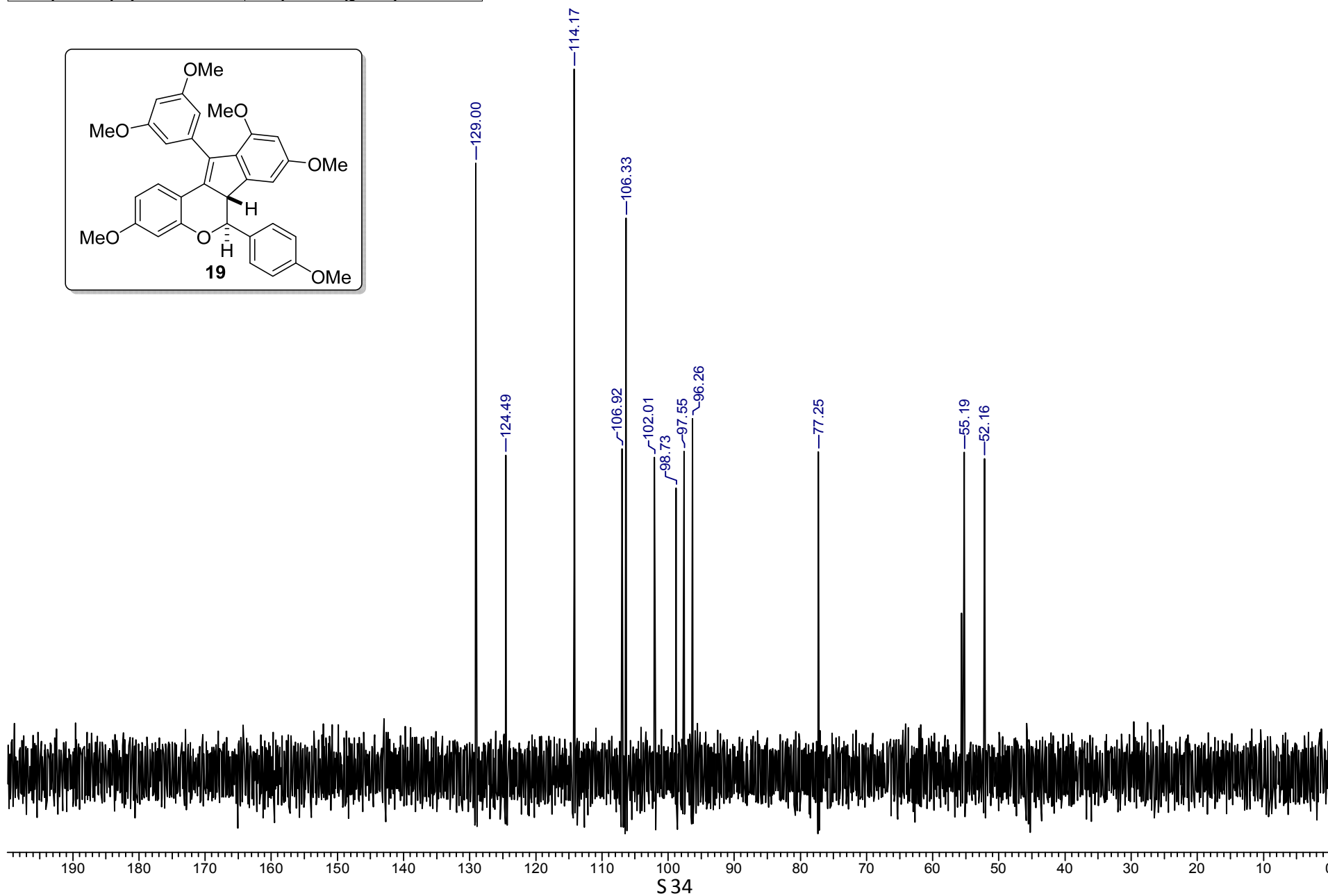
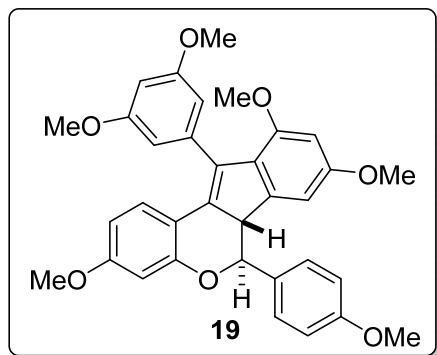


Figure S34: HRMS spectrum of compound 19

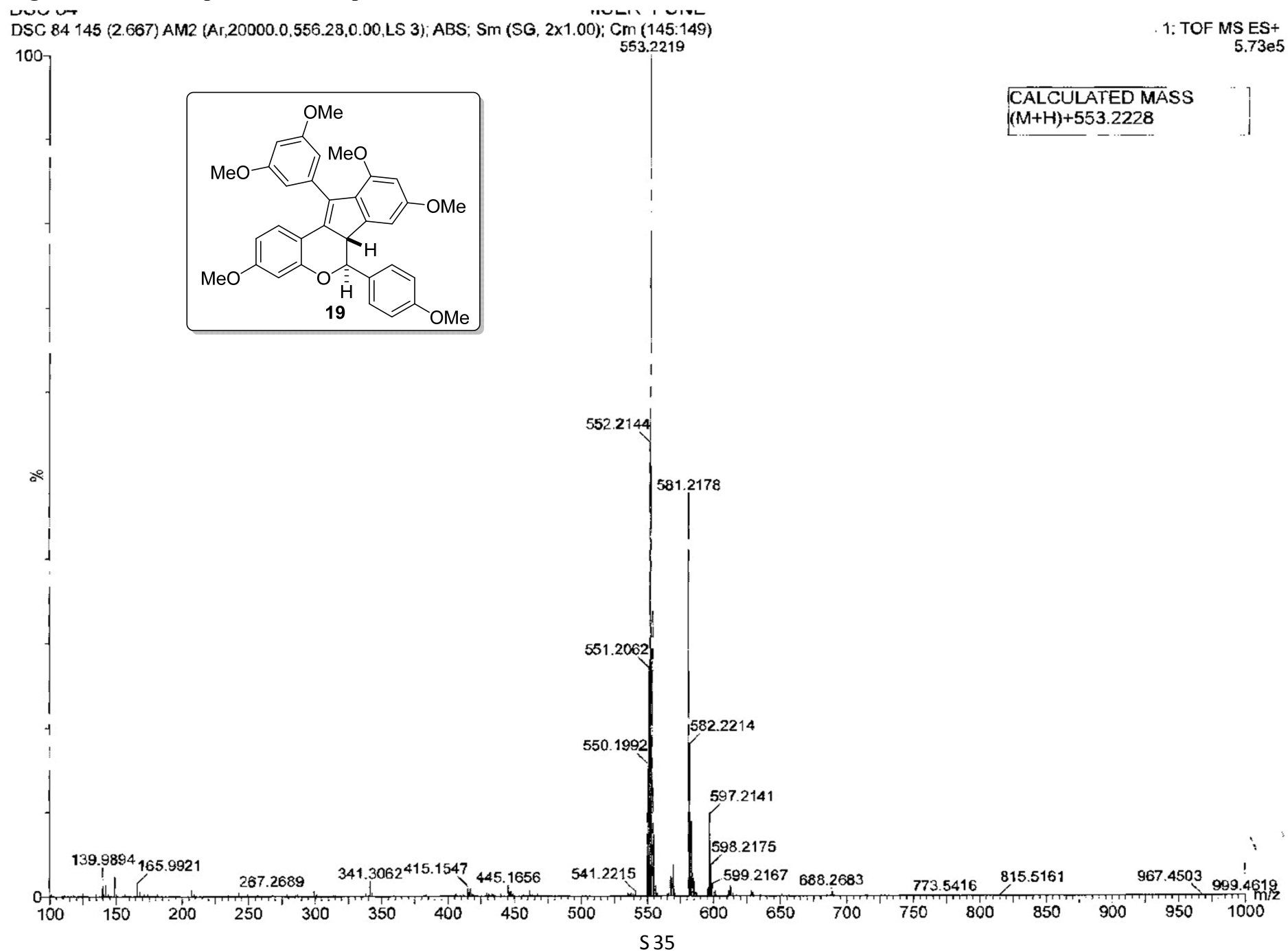


Figure F35: ^1H NMR of compound 20

Acquisition Time (sec)	1.2800	Comment	Ashwini 1H	Date	11 Aug 2017 17:55:44
Date Stamp	11 Aug 2017 17:55:44			File Name	\\172.16.2.4\nmr_data\AV500\2017_AV500\Aug_17_500\Fri2av500#009_44473\1\PDATA\1\1r
Frequency (MHz)	500.13	Nucleus	^1H	Number of Transients	32
Original Points Count	16000	Owner	nmr	Points Count	32768
Receiver Gain	362.00	SW(cyclical) (Hz)	12500.00	Pulse Sequence	zg30
				Solvent	CHLOROFORM-d

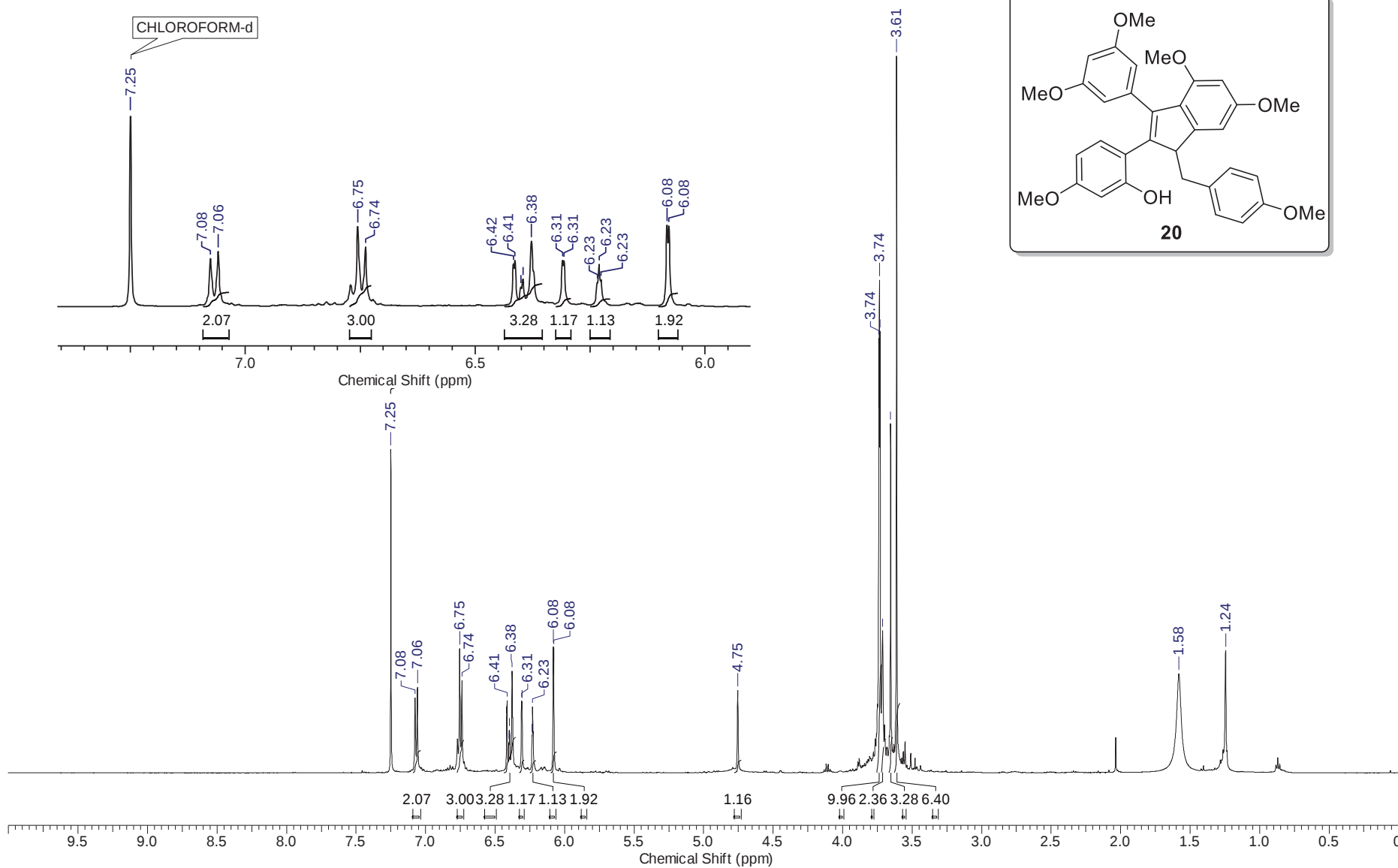


Figure S36: ^{13}C NMR of compound 20

Acquisition Time (sec)	1.1010	Comment	13C	Date	12 Aug 2017 09:42:56	Frequency (MHz)	125.76
Nucleus	^{13}C	Number of Transients	16775	Original Points Count	32768	Points Count	32768
Sweep Width (Hz)	29761.00	Temperature (grad C)	24.900	Solvent	CHLOROFORM-d		

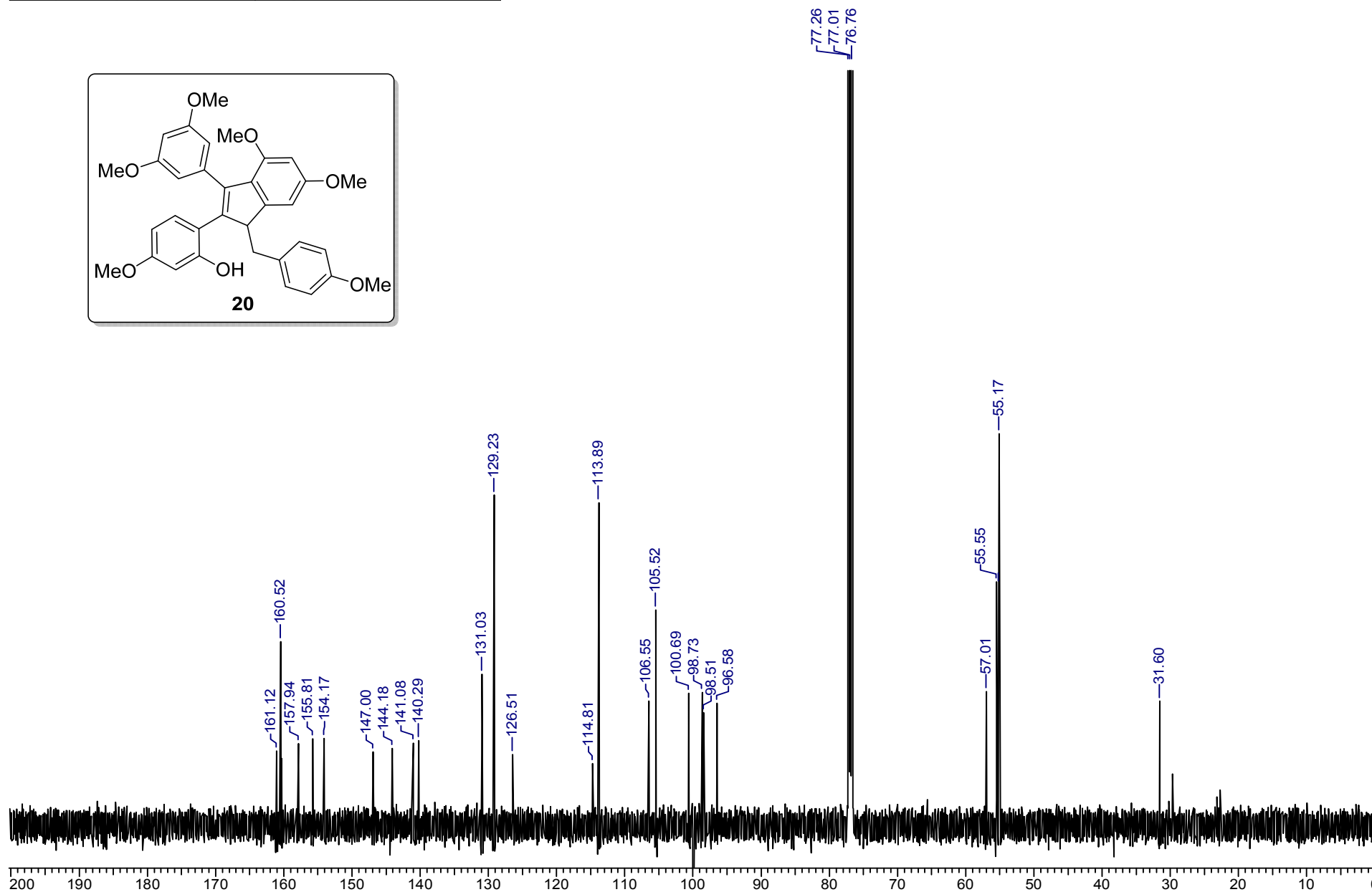
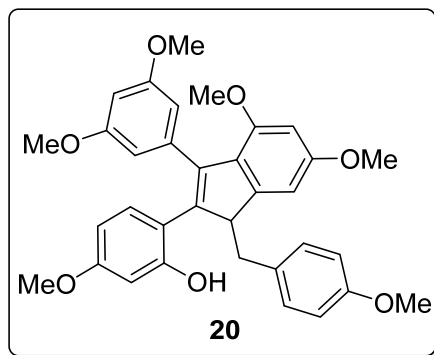


Figure S37: DEPT ^{13}C NMR of compound 20

Acquisition Time (sec)	0.6554	Comment	DEPT	Date	11 Aug 2017 23:39:12	Frequency (MHz)	125.76
Nucleus	^{13}C	Number of Transients	8000	Original Points Count	20480	Points Count	32768
Sweep Width (Hz)	31249.05	Temperature (grad C)	24.900	Solvent	CHLOROFORM-d		

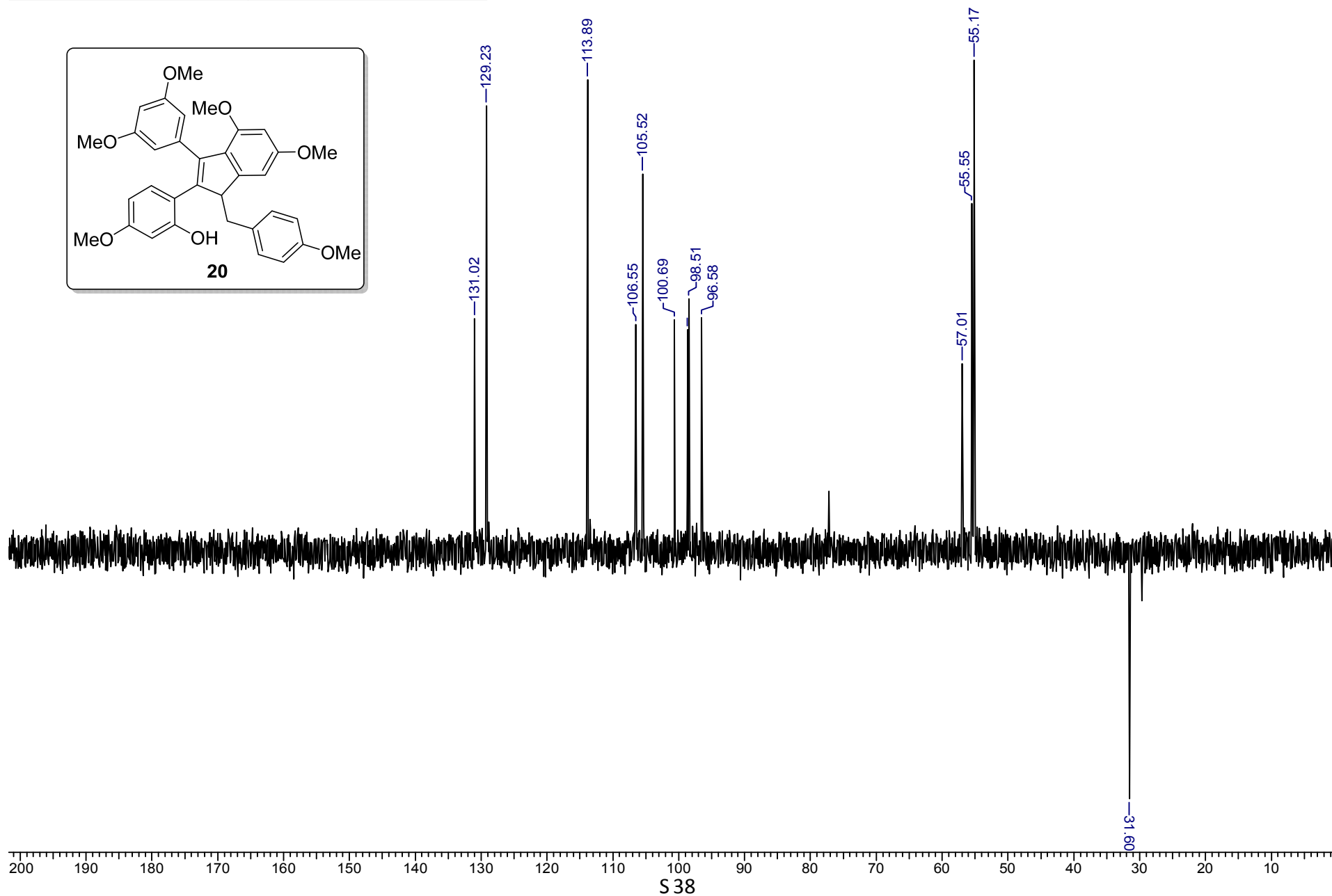
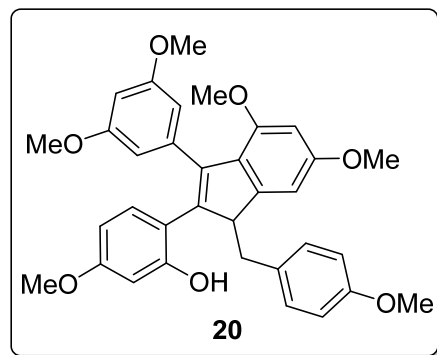


Figure S38: HRMS spectrum of compound 20

DSC 85

IISER PUNE

DSC 85 129 (2.375) AM2 (Ar.20000.0,556.28,0.00,LS 3); ABS; Sm (SG, 2x1.00)

1: TOF MS ES+
1.51e5

