

Chemoenzymatic collective synthesis of optically active hydroxyl(methyl)tetrahydronaphthalene-based bioactive terpenoids

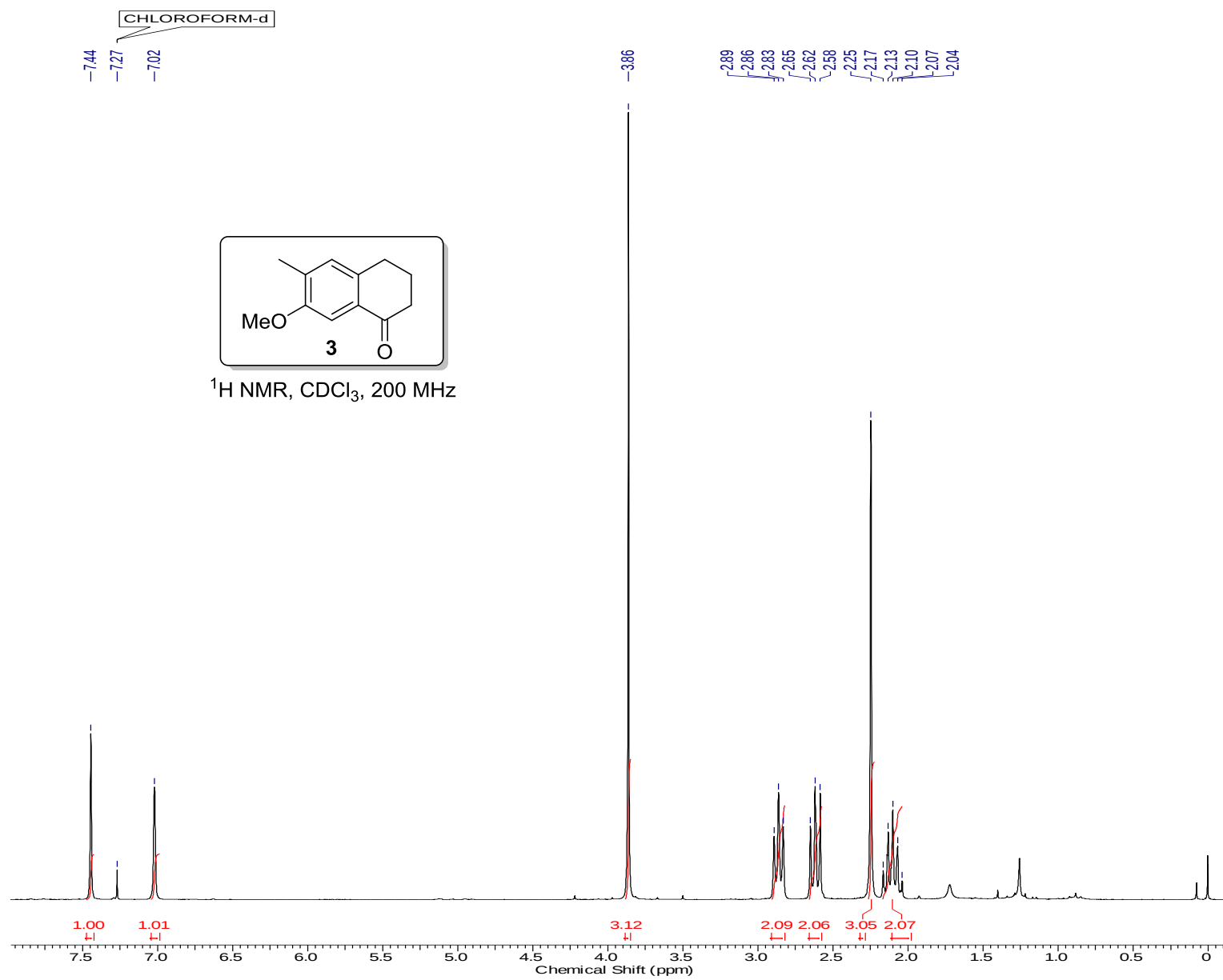
Ramesh U. Batwal and Narshinha P. Argade*

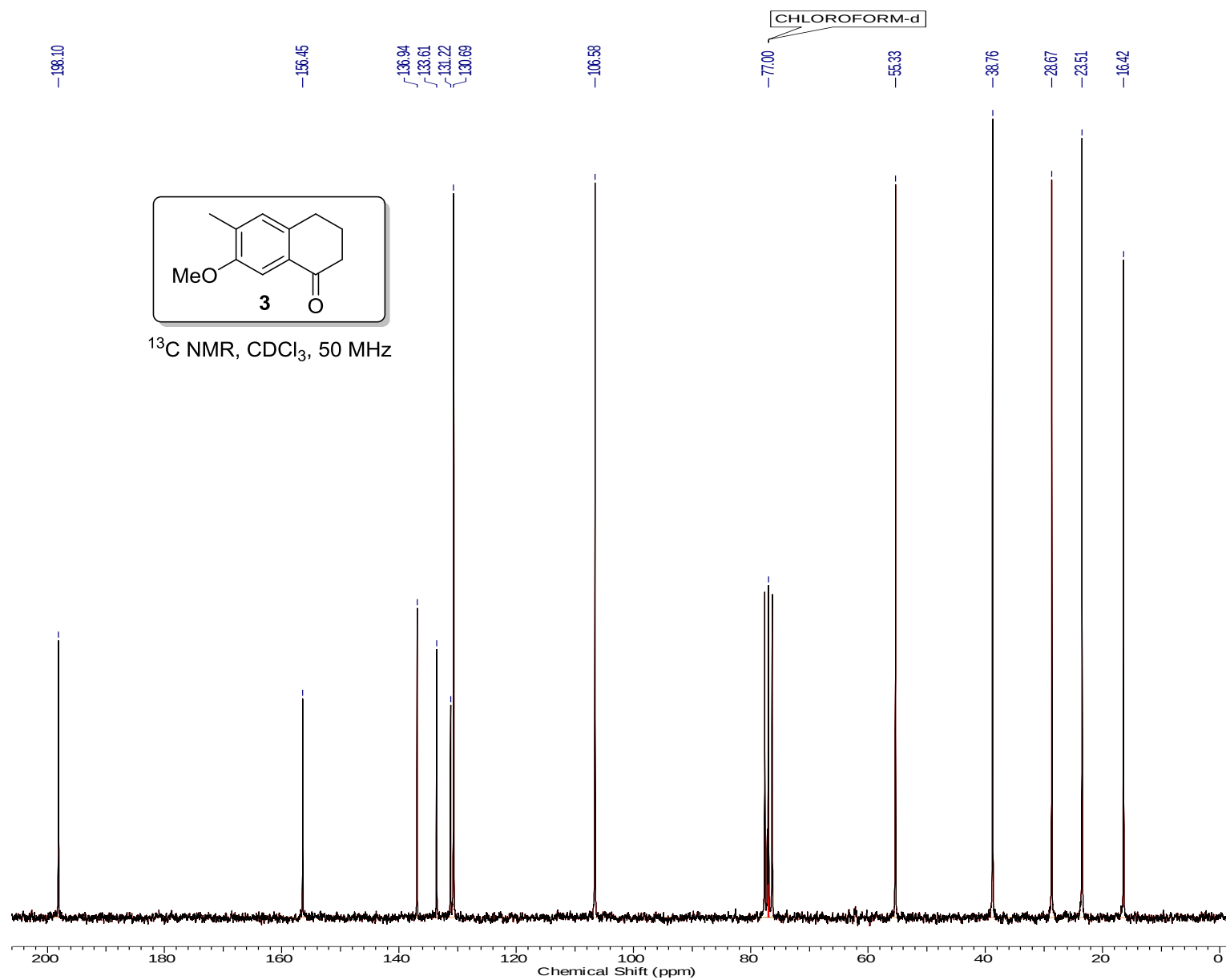
Division of Organic Chemistry, National Chemical Laboratory (CSIR), Pune 411 008, India

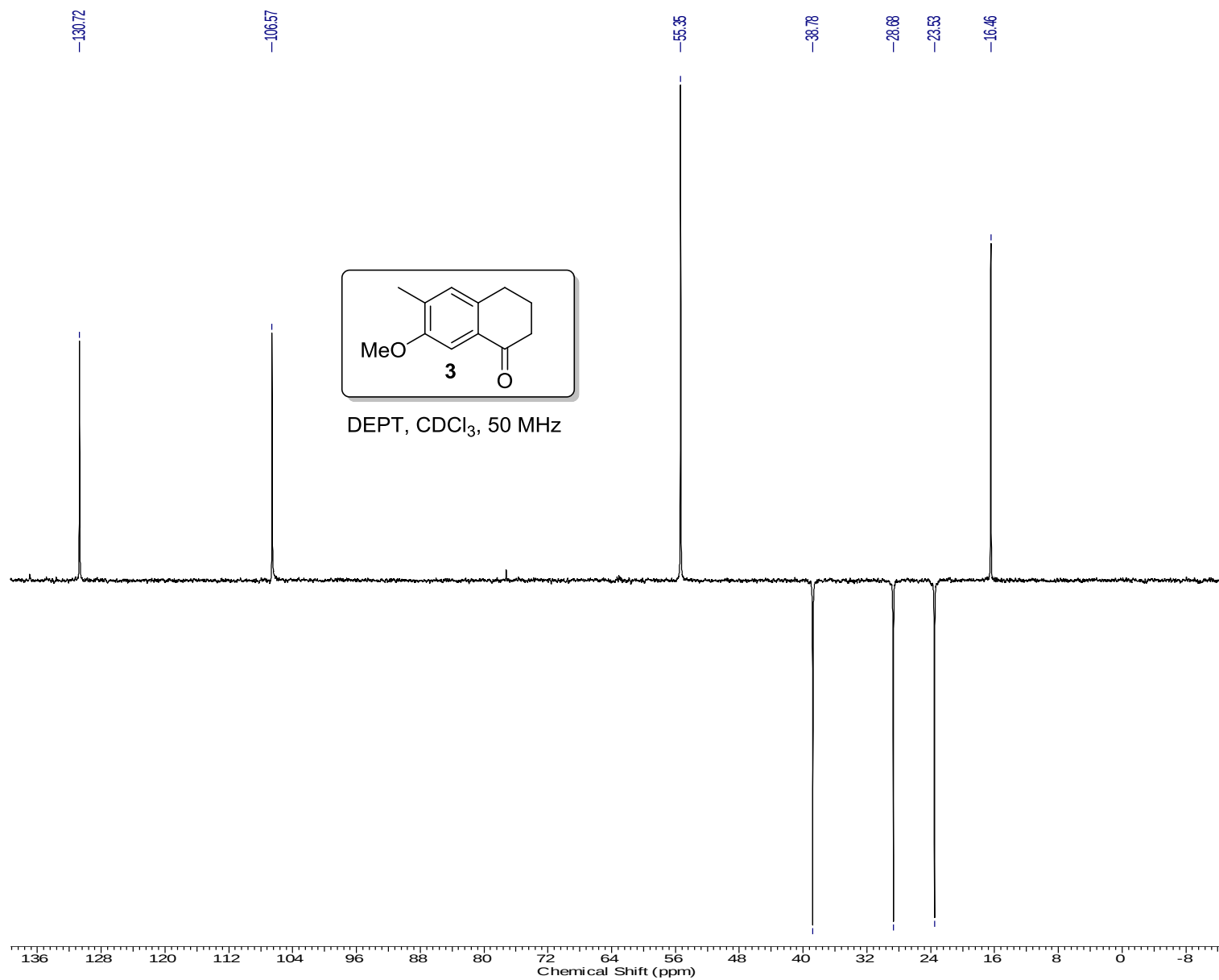
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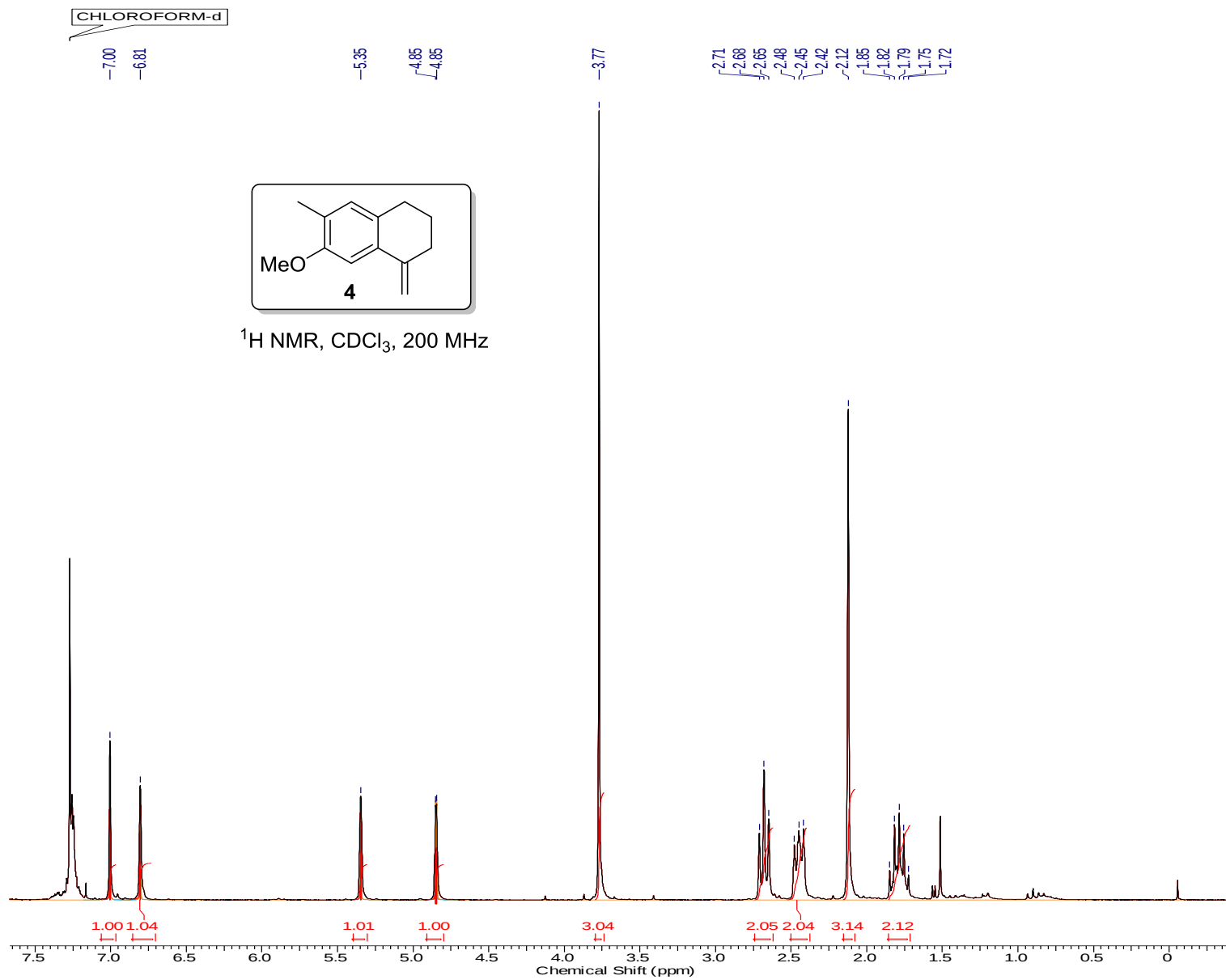
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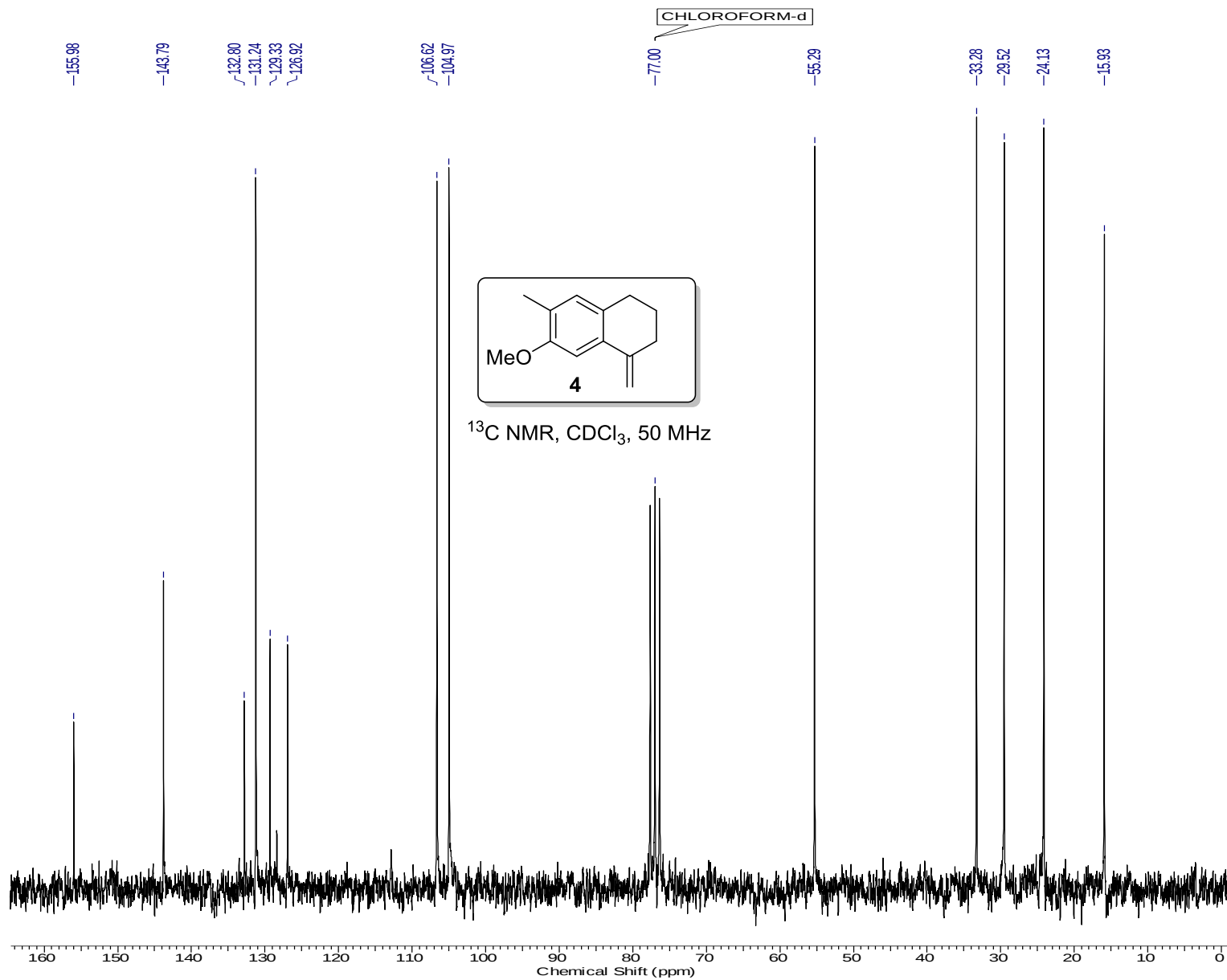
Copies of ^1H , ^{13}C and DEPT NMR Spectra.....	02 to 57
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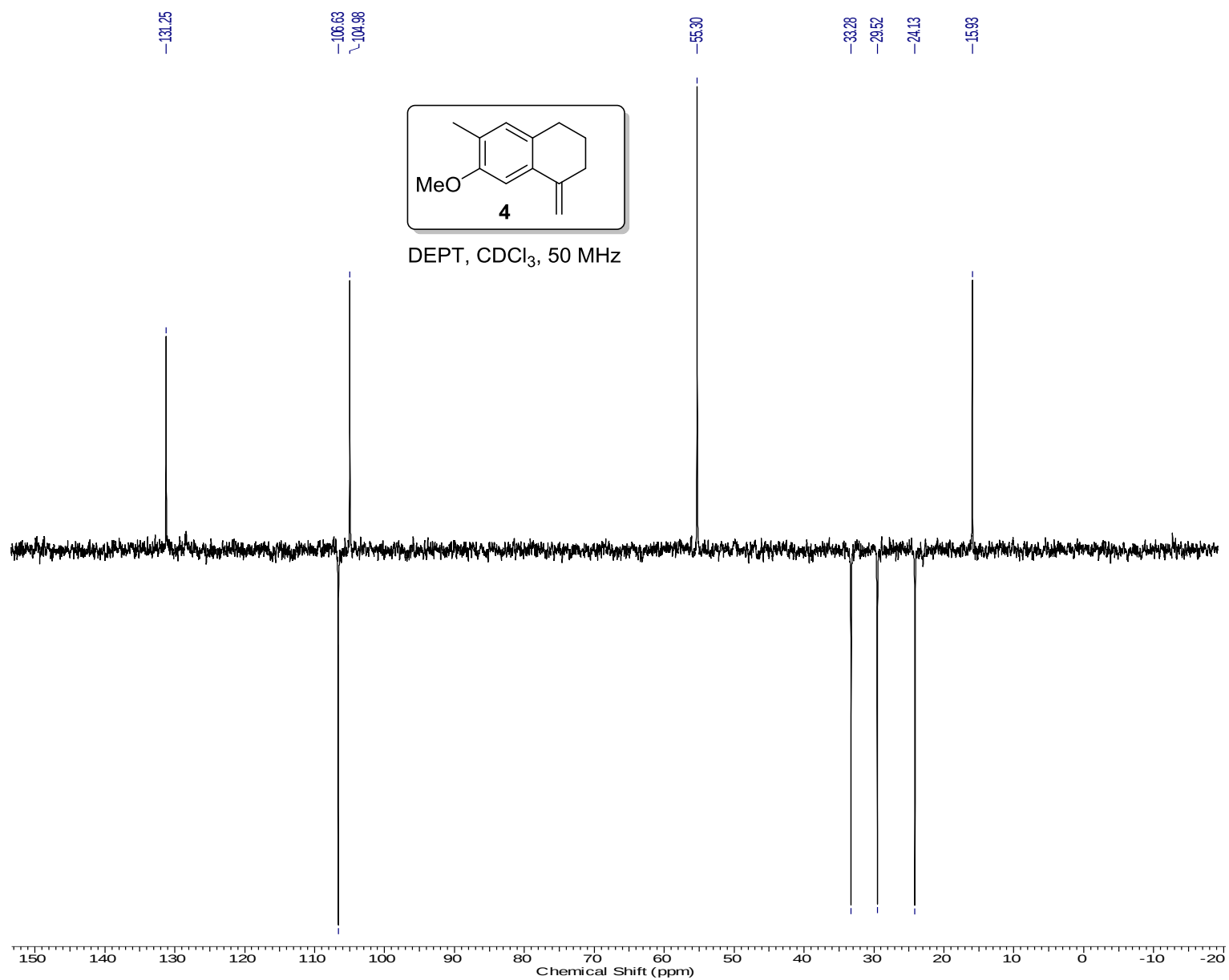


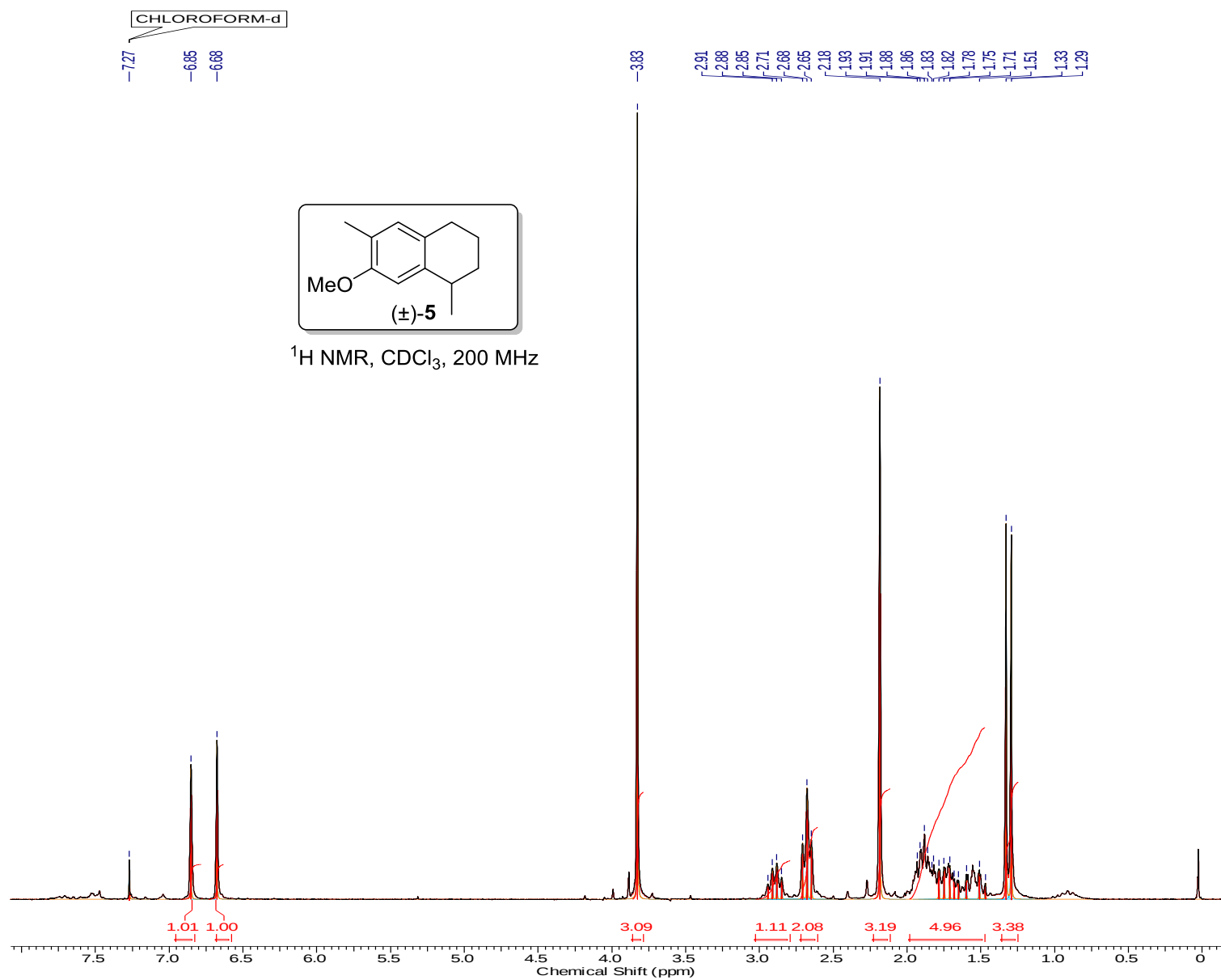


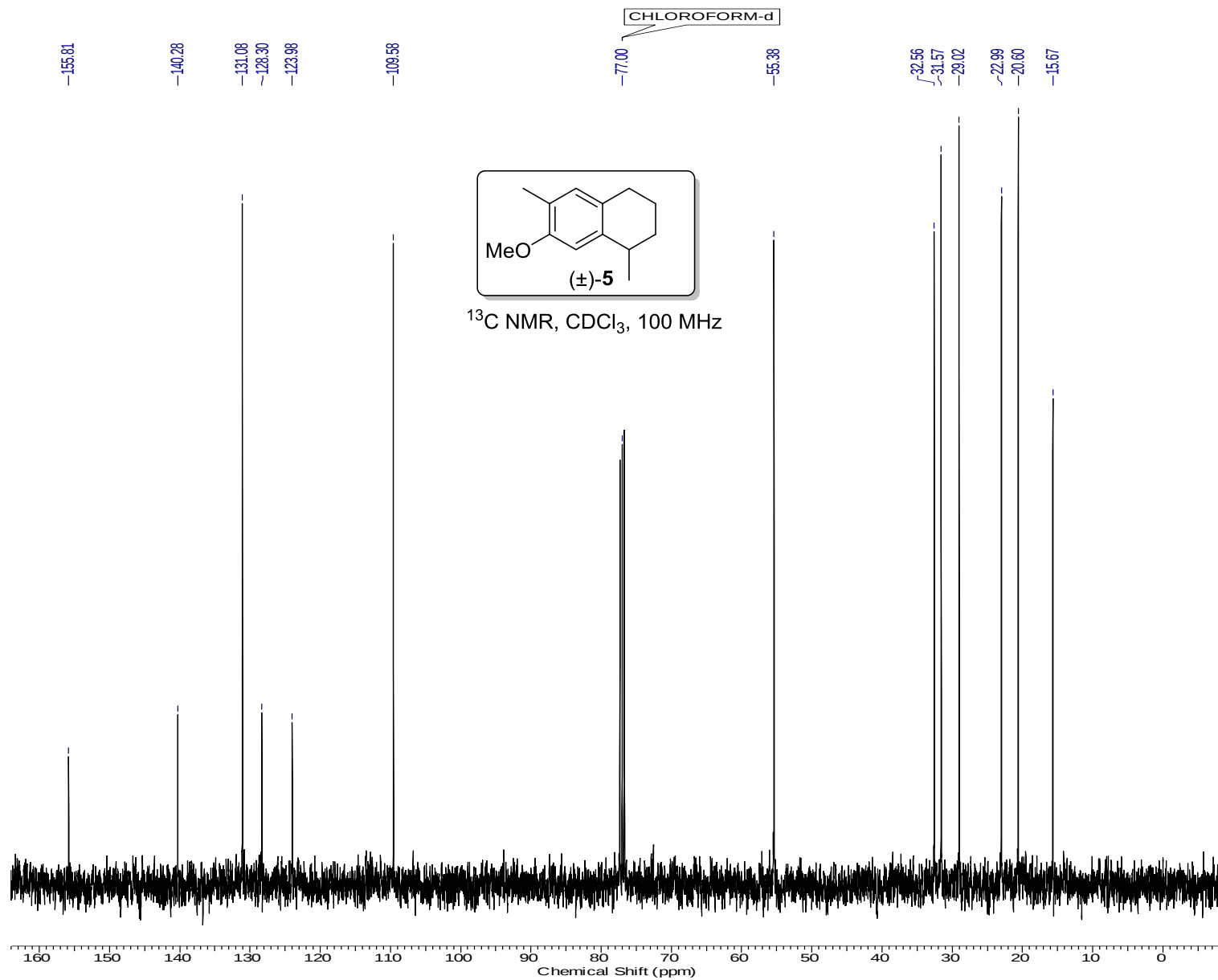


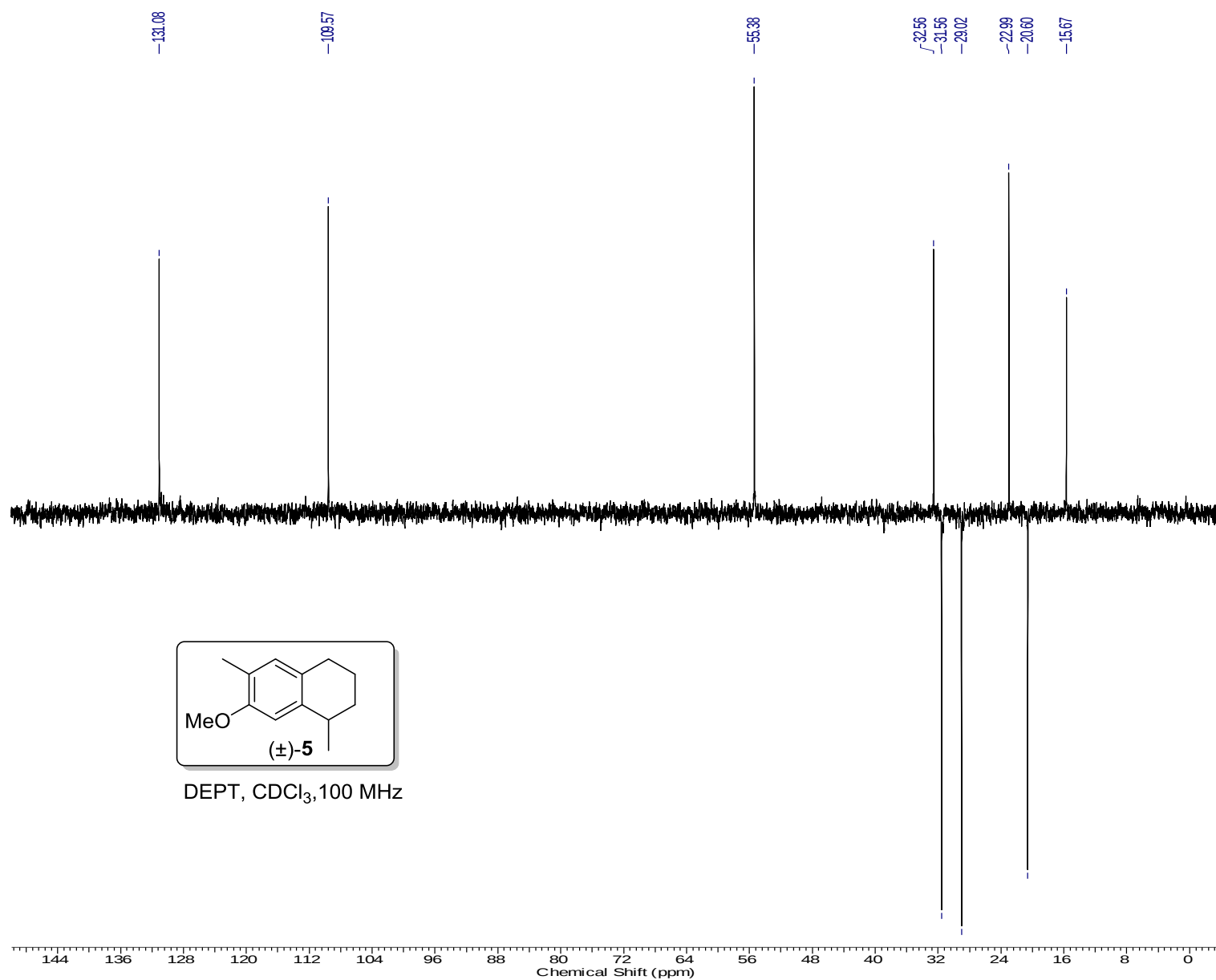


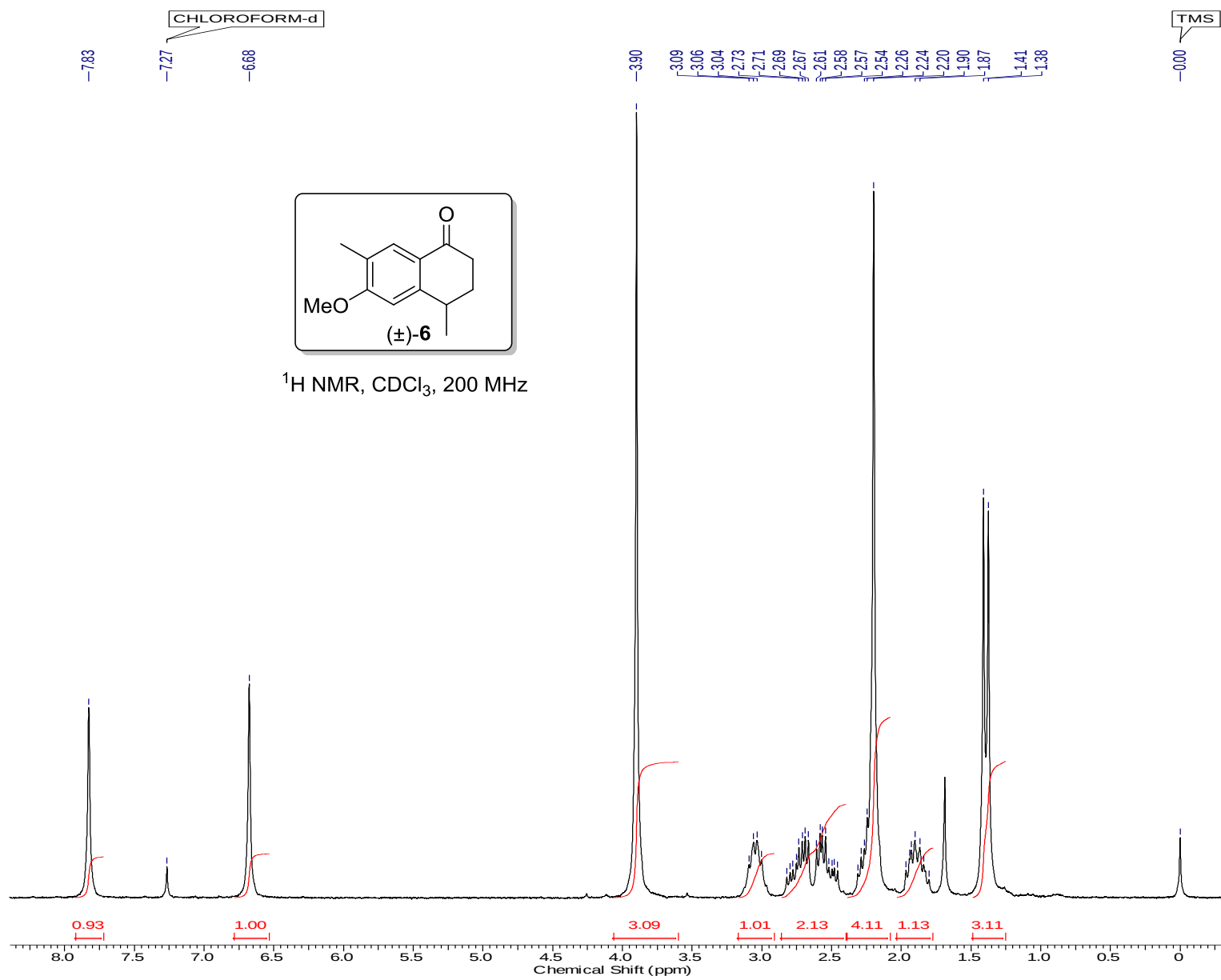


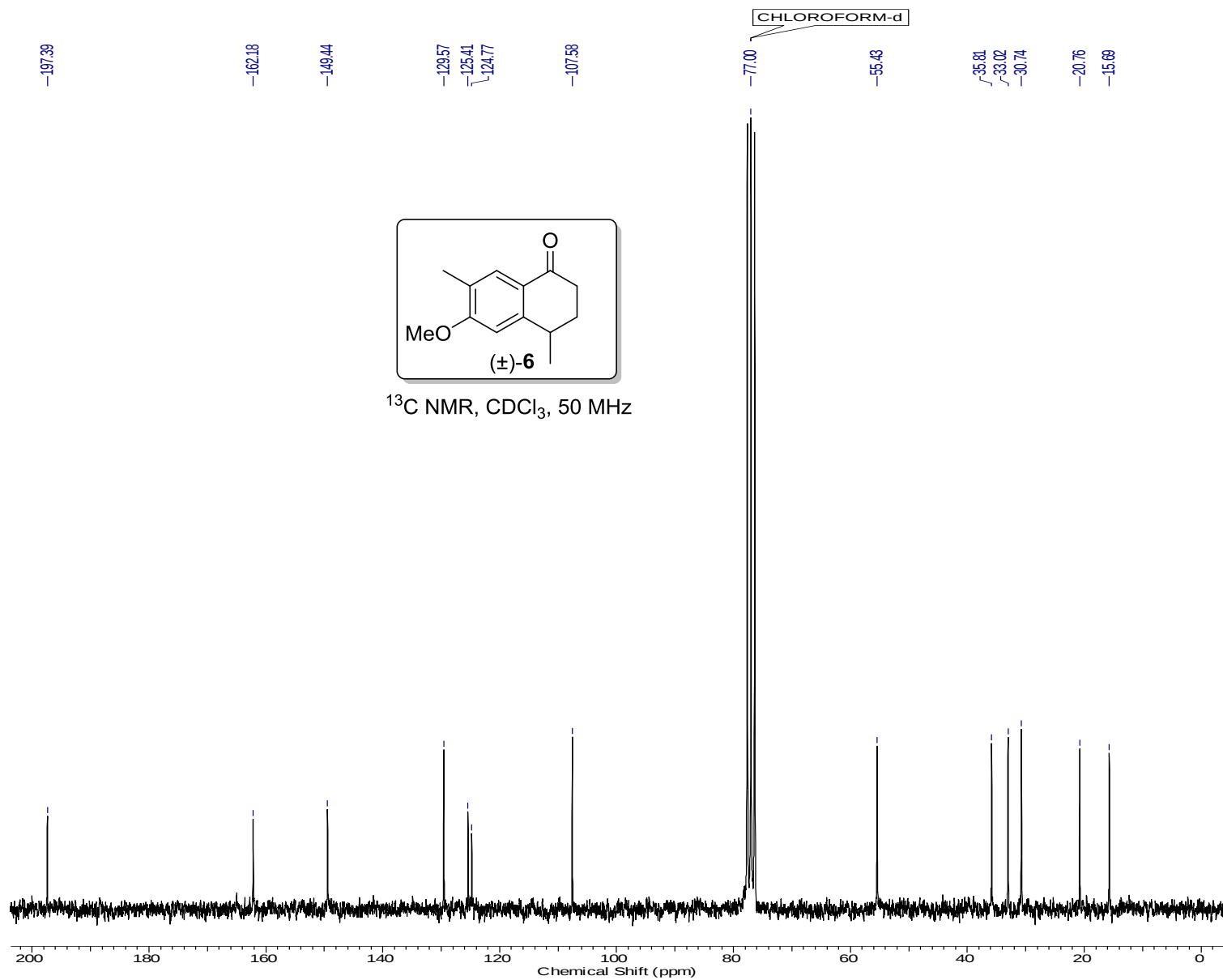


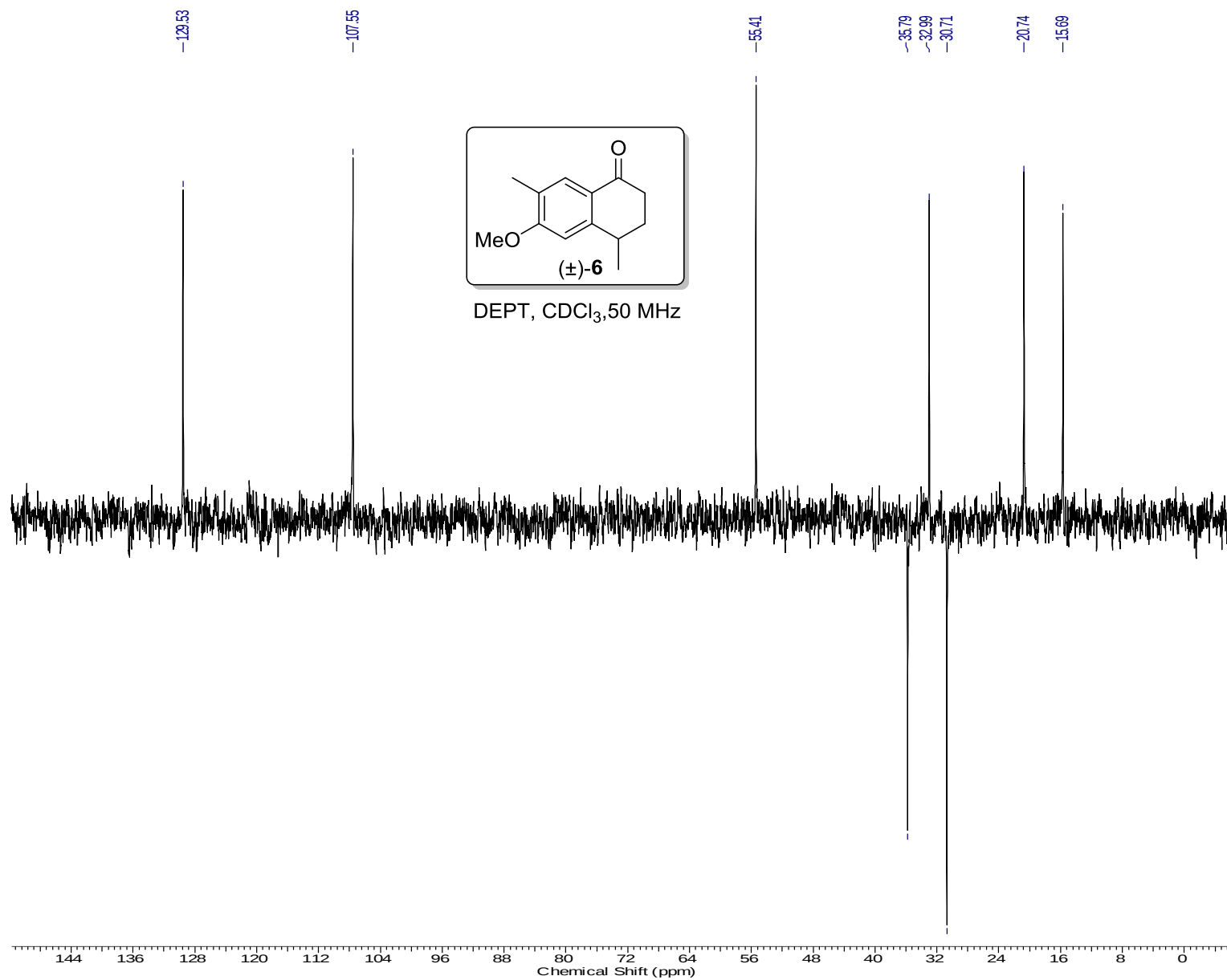


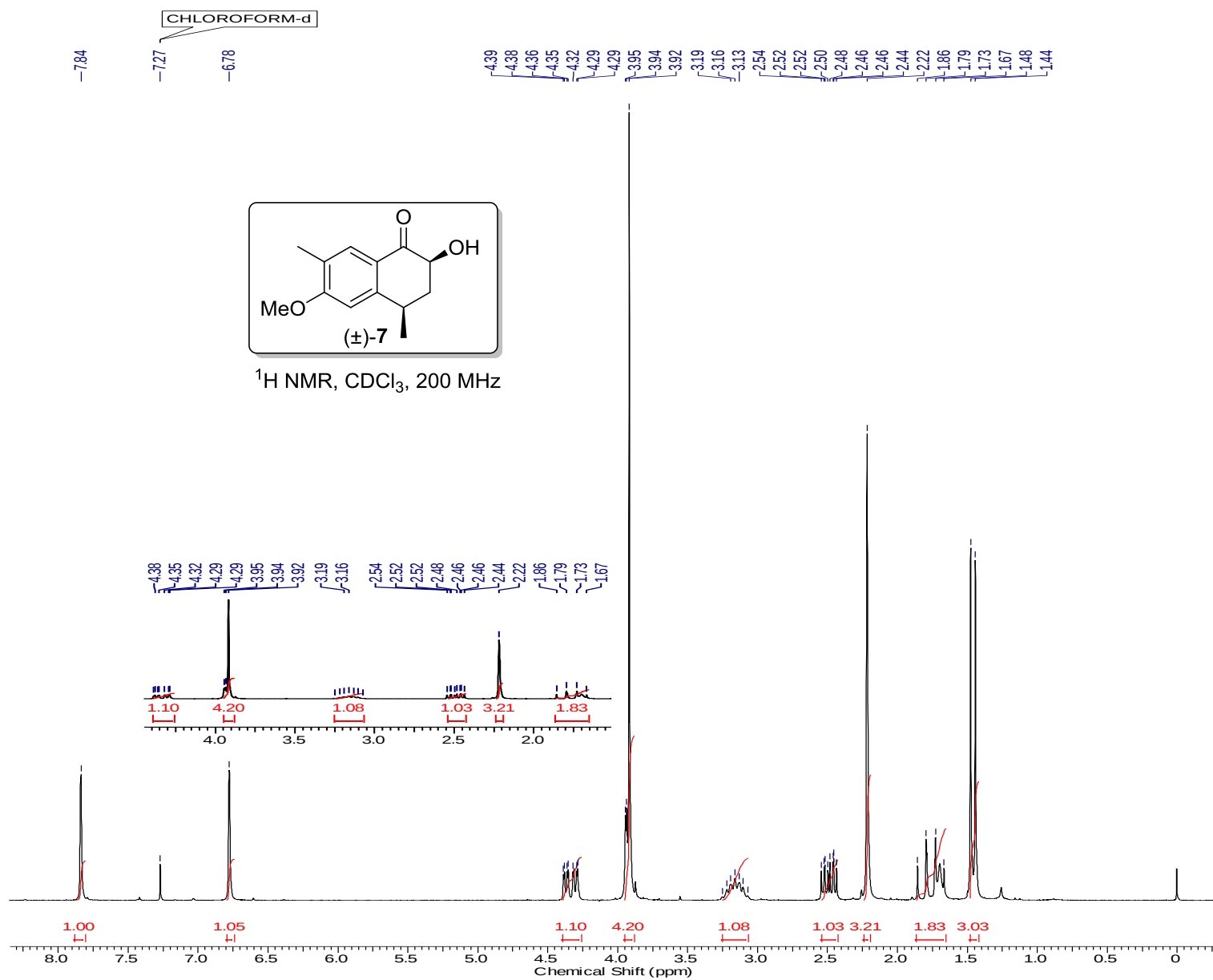


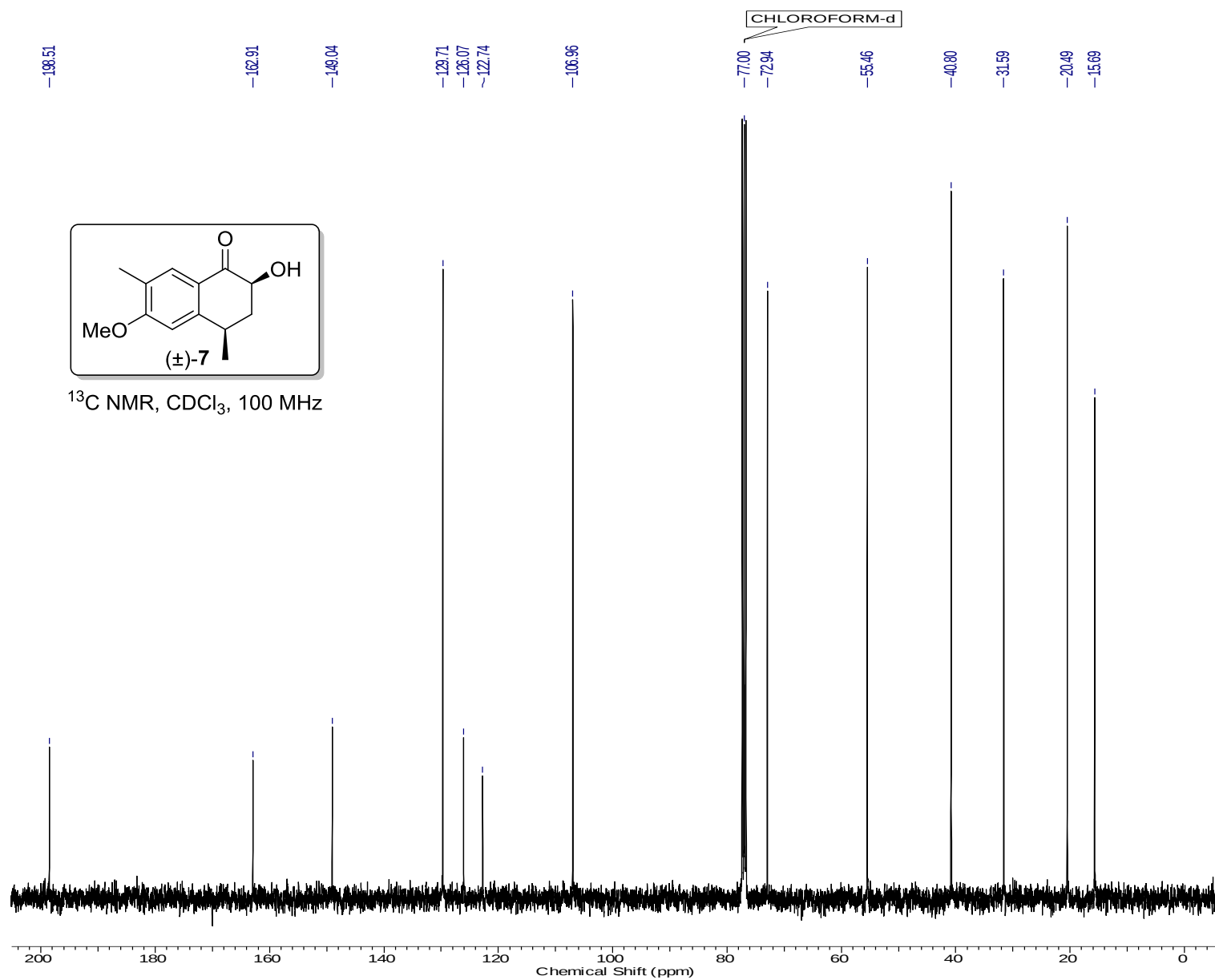


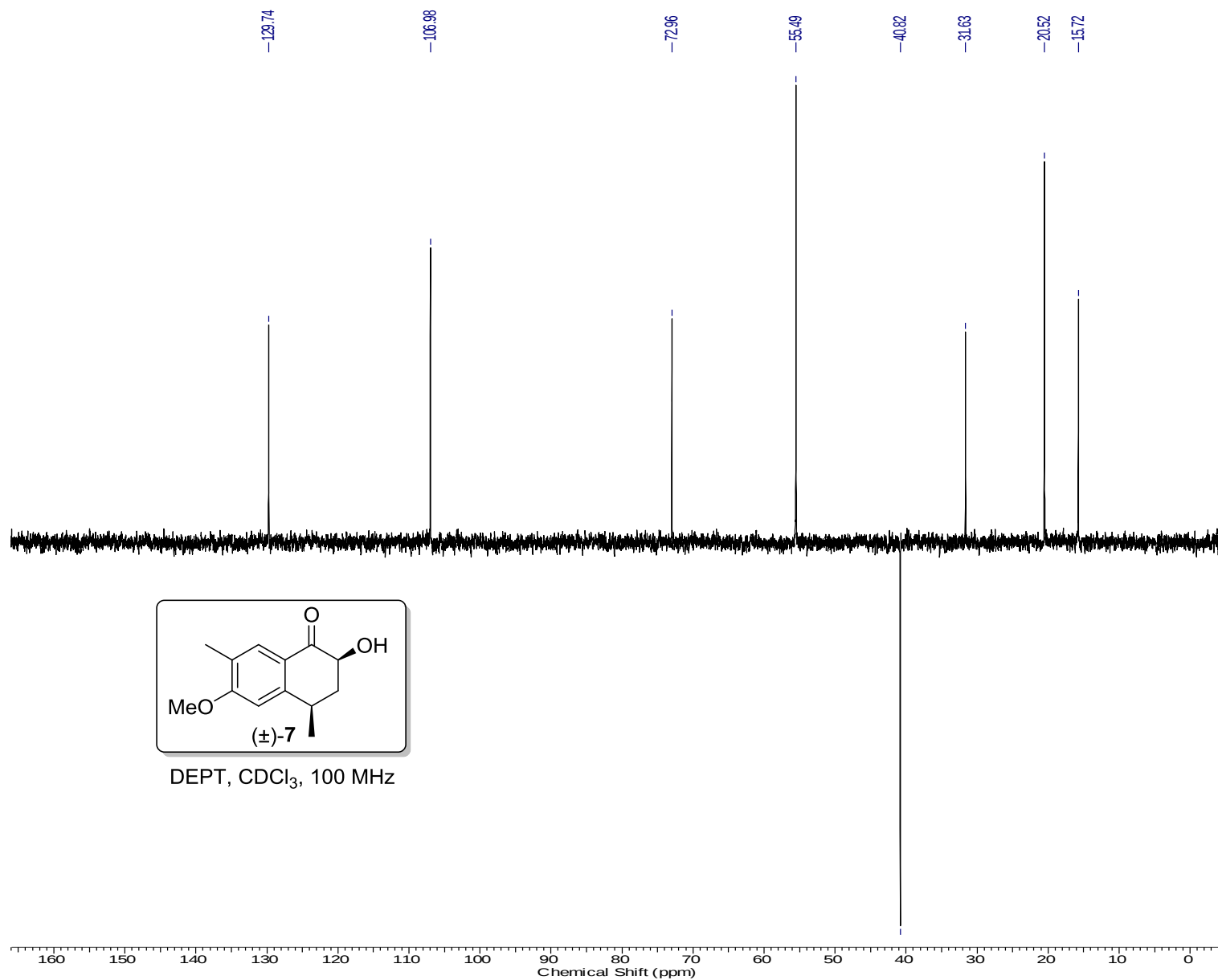


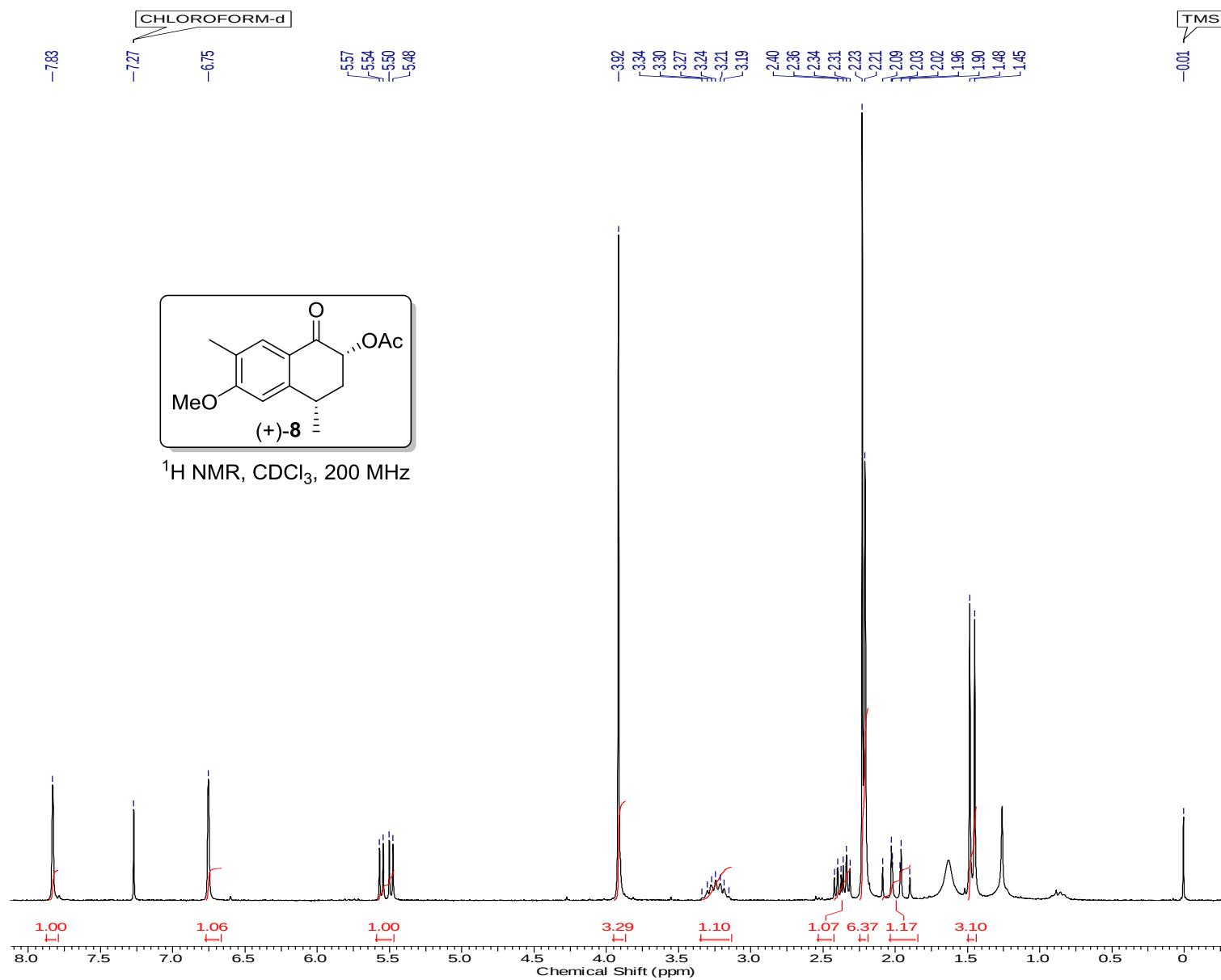


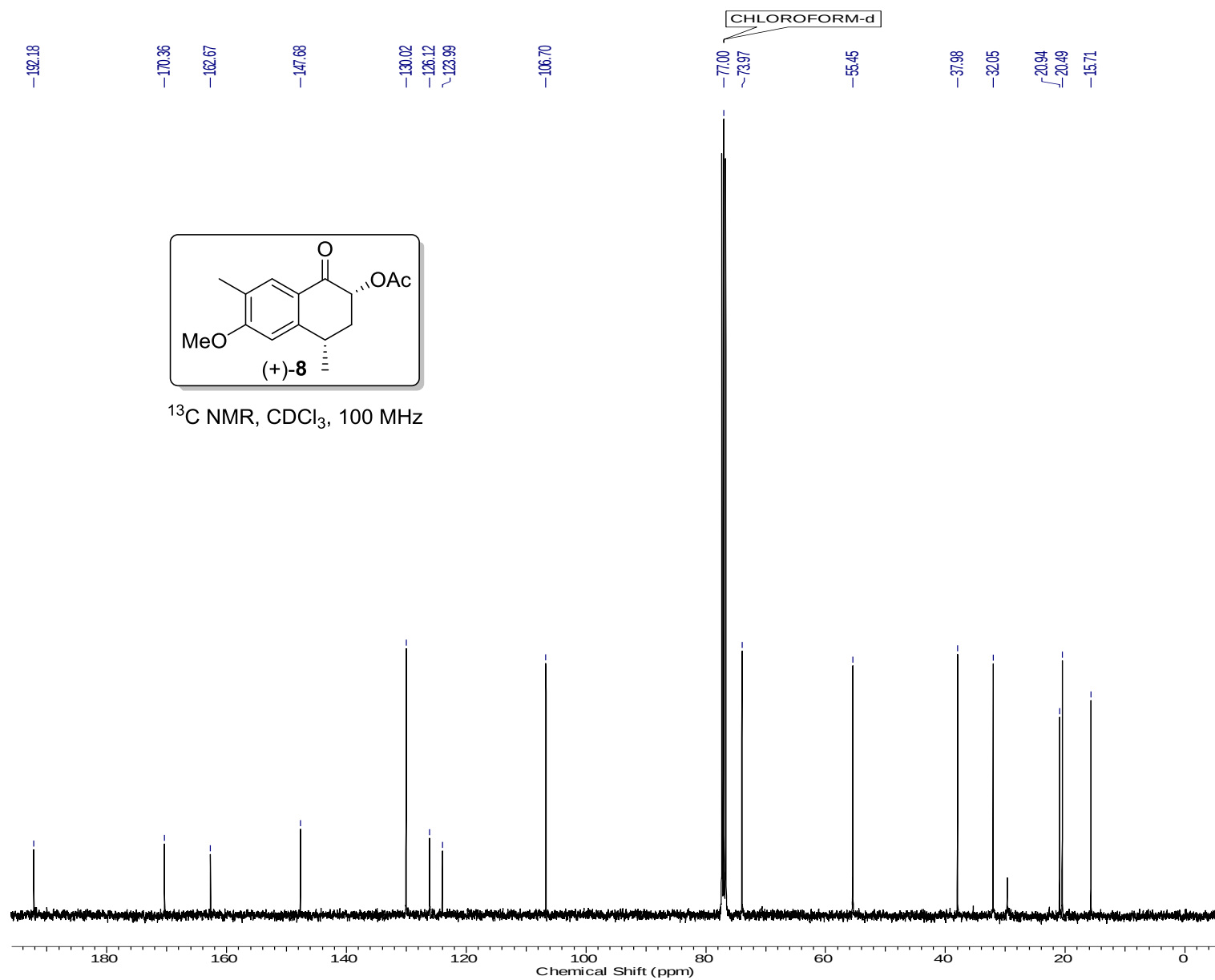


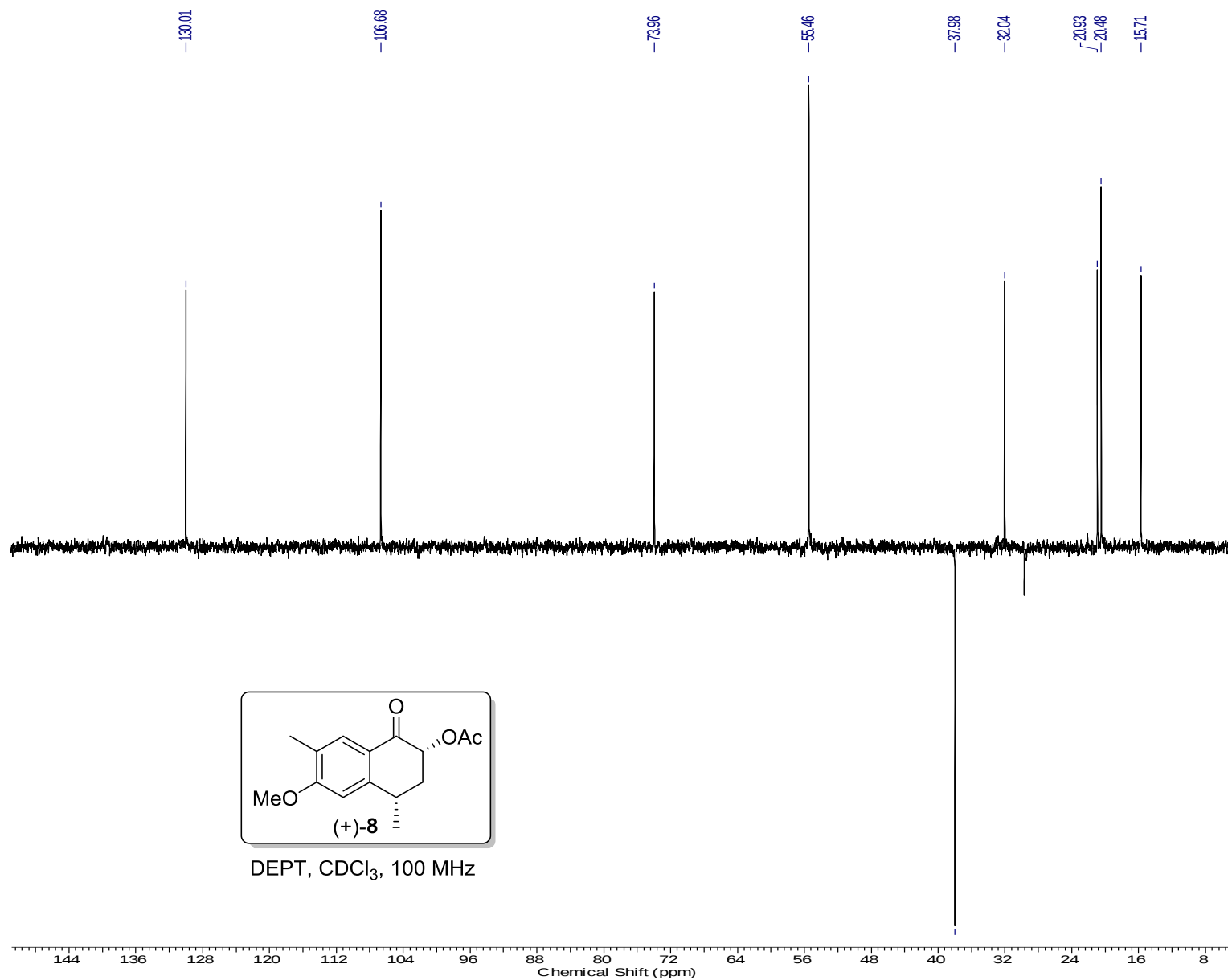


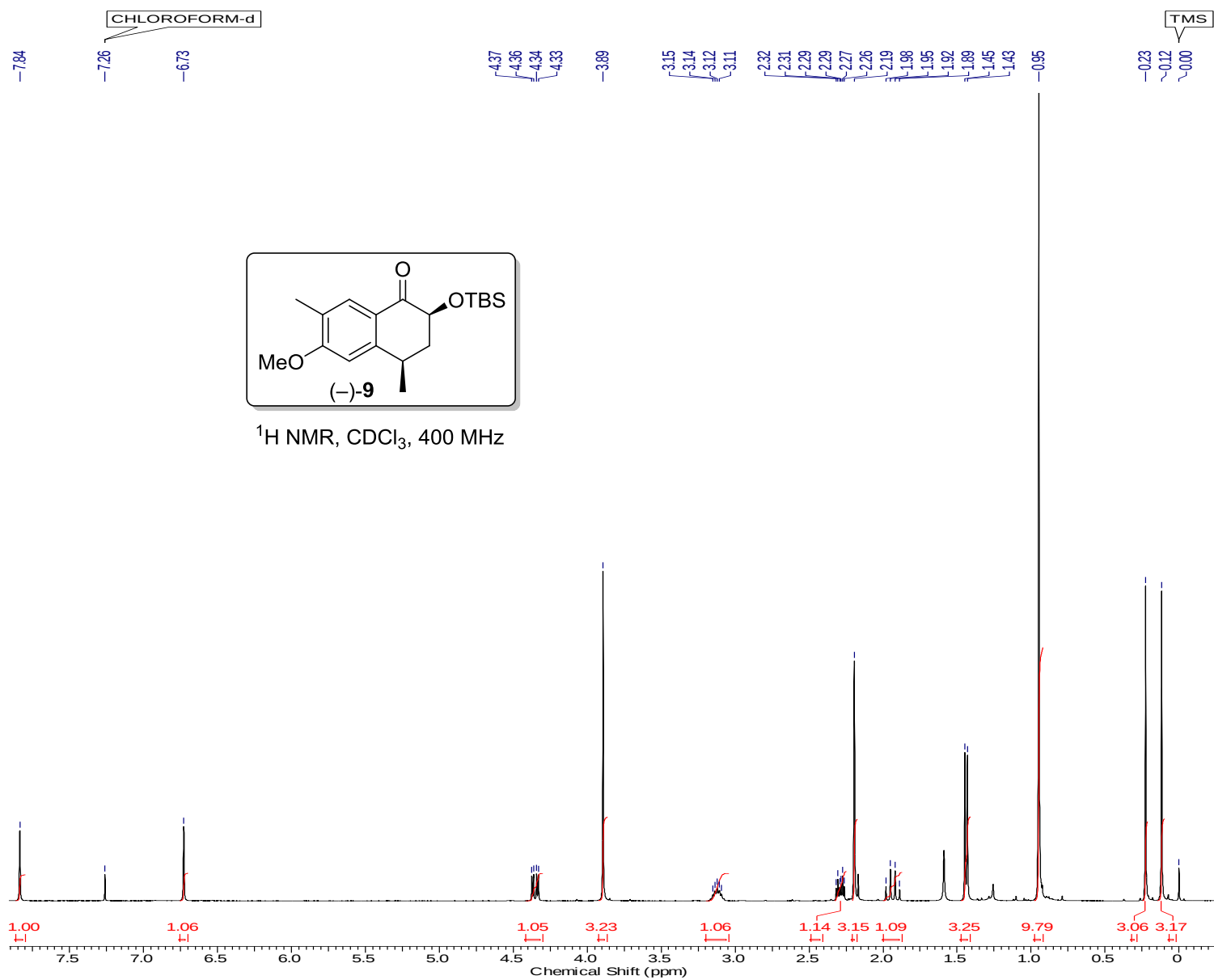


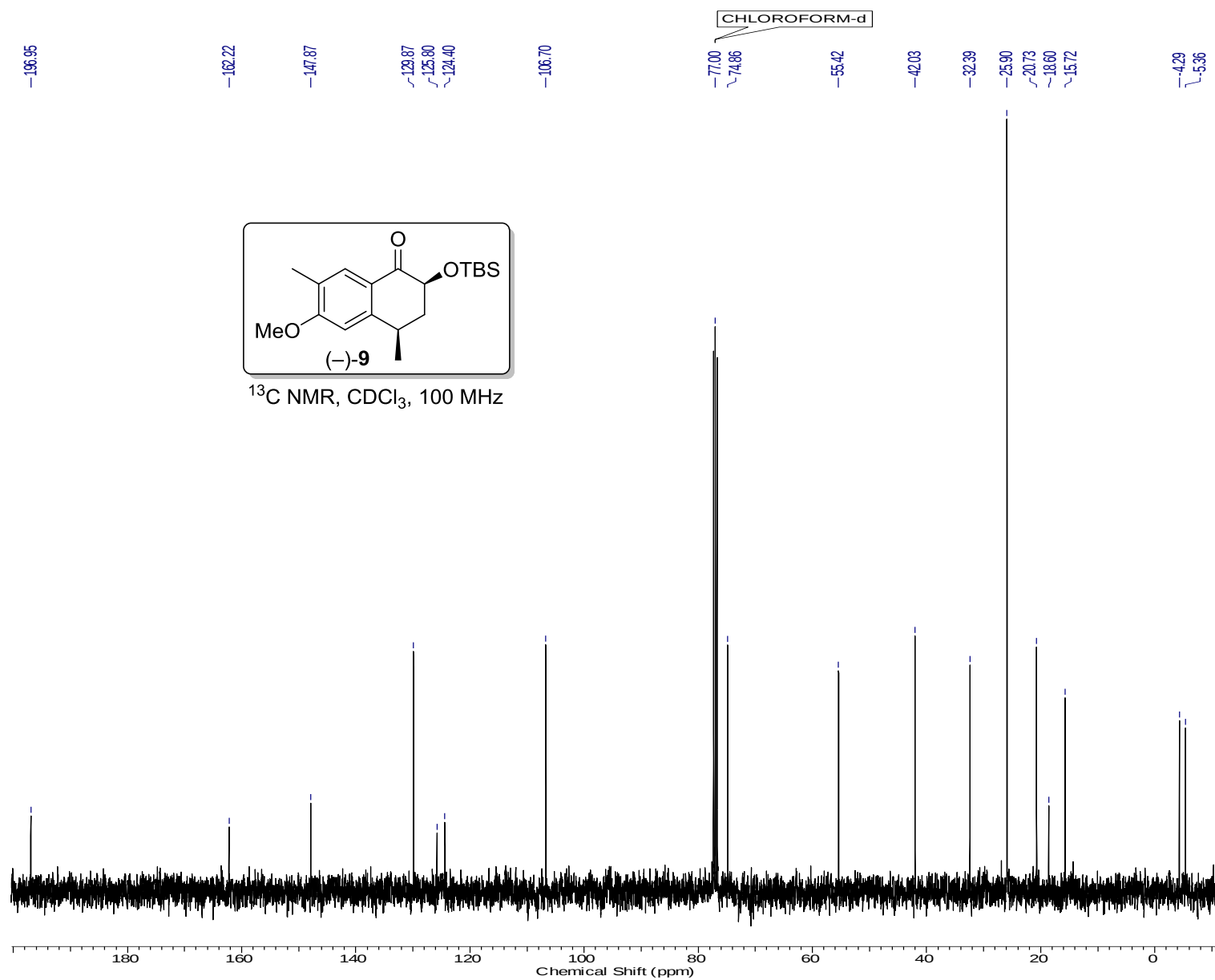


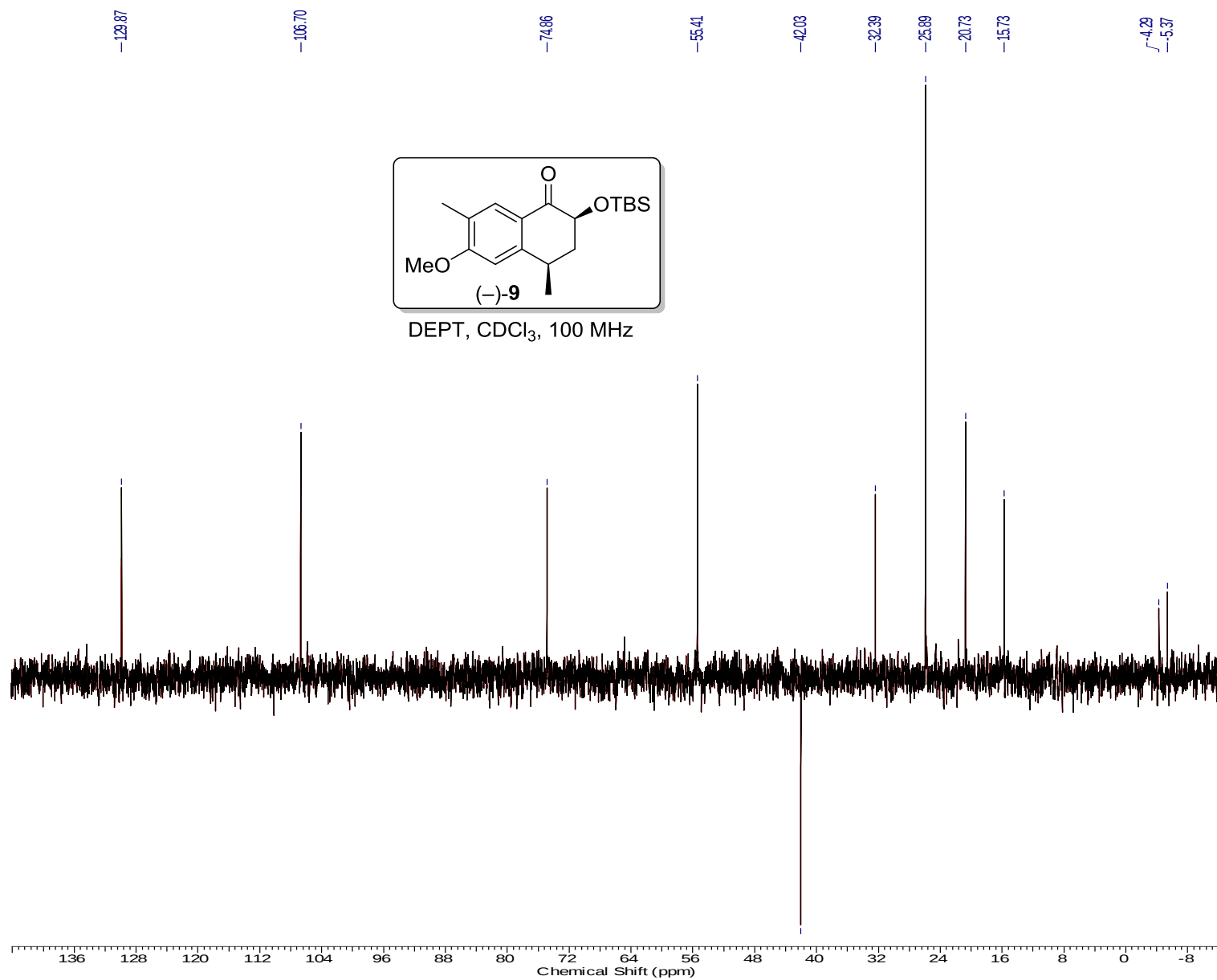


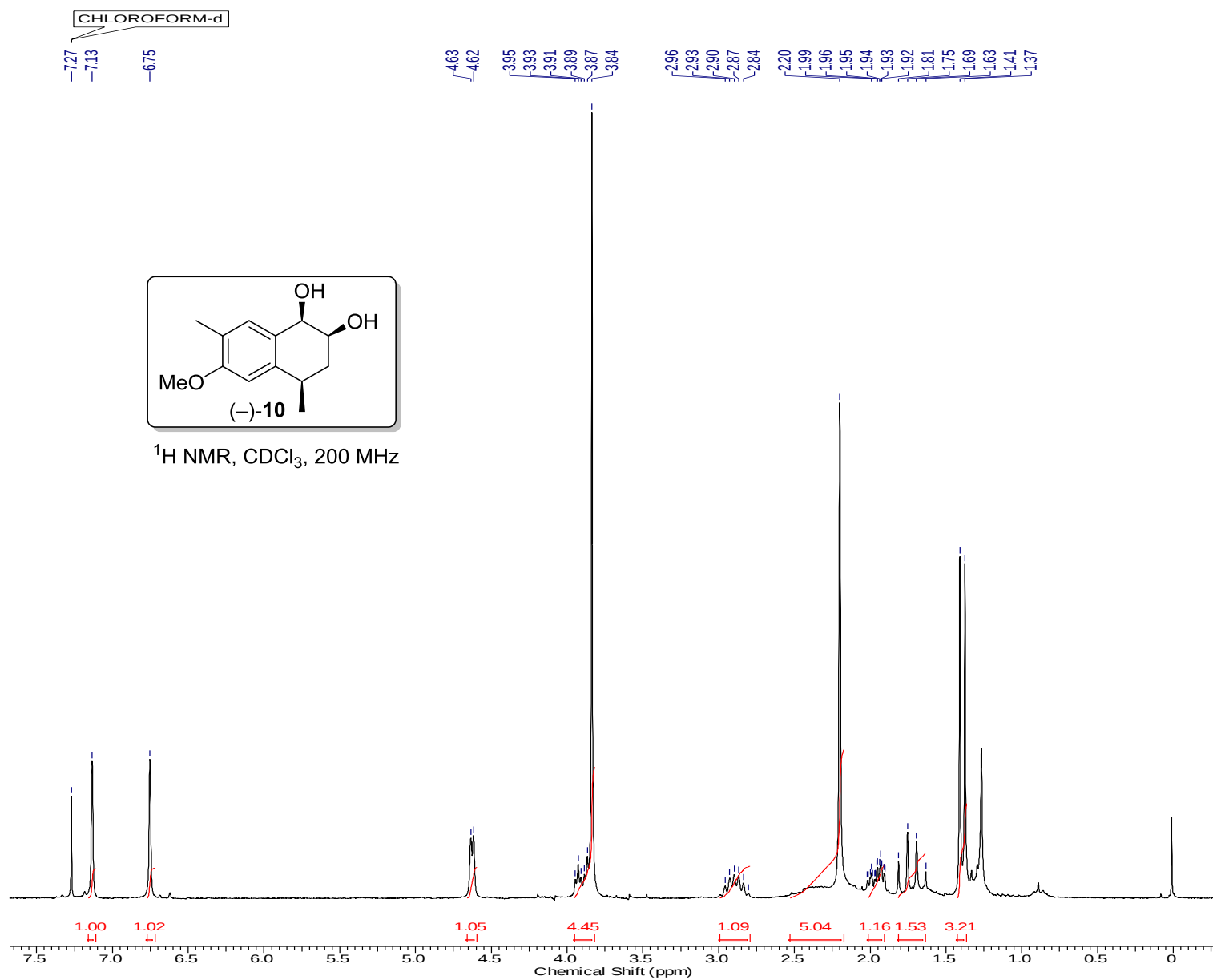


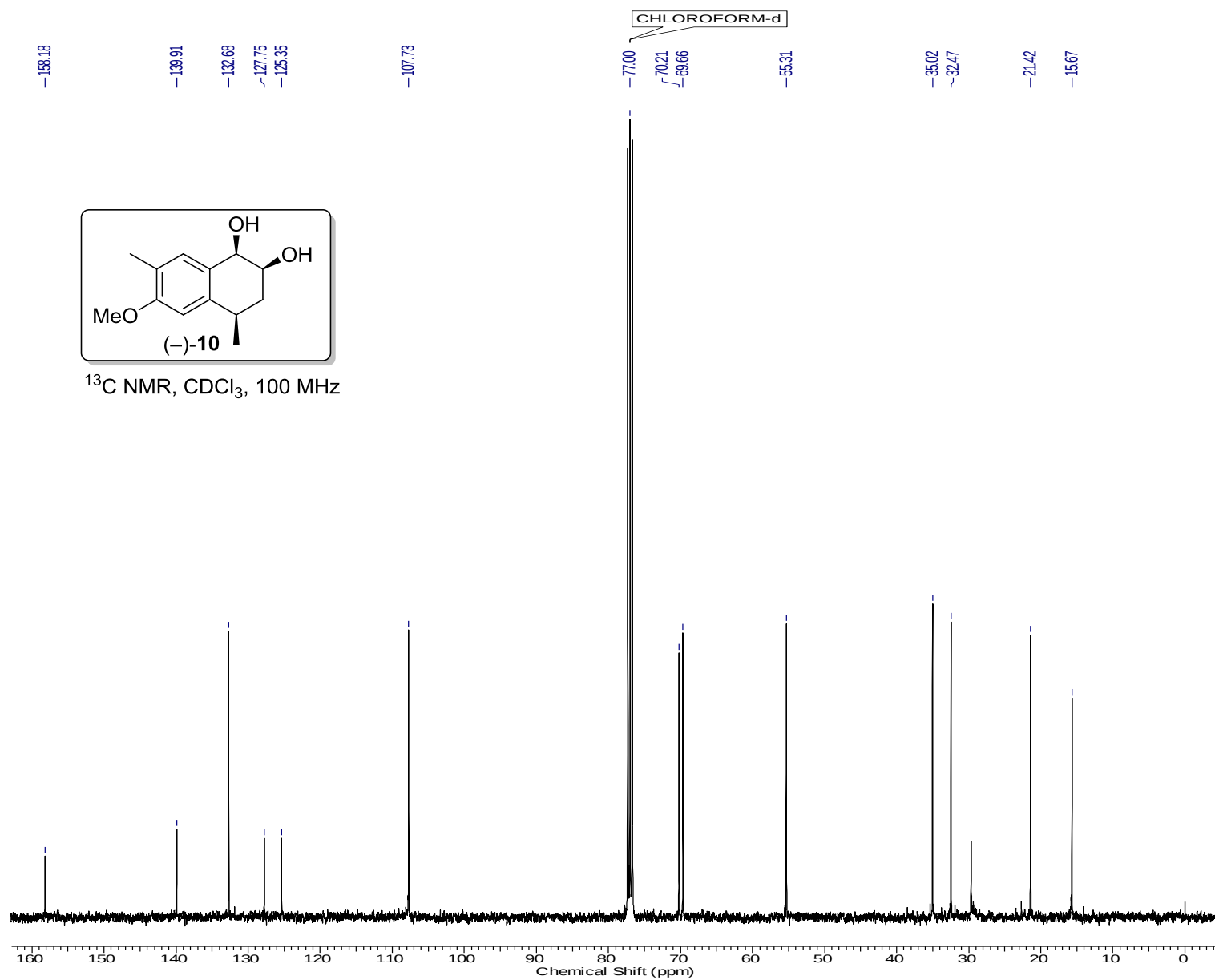


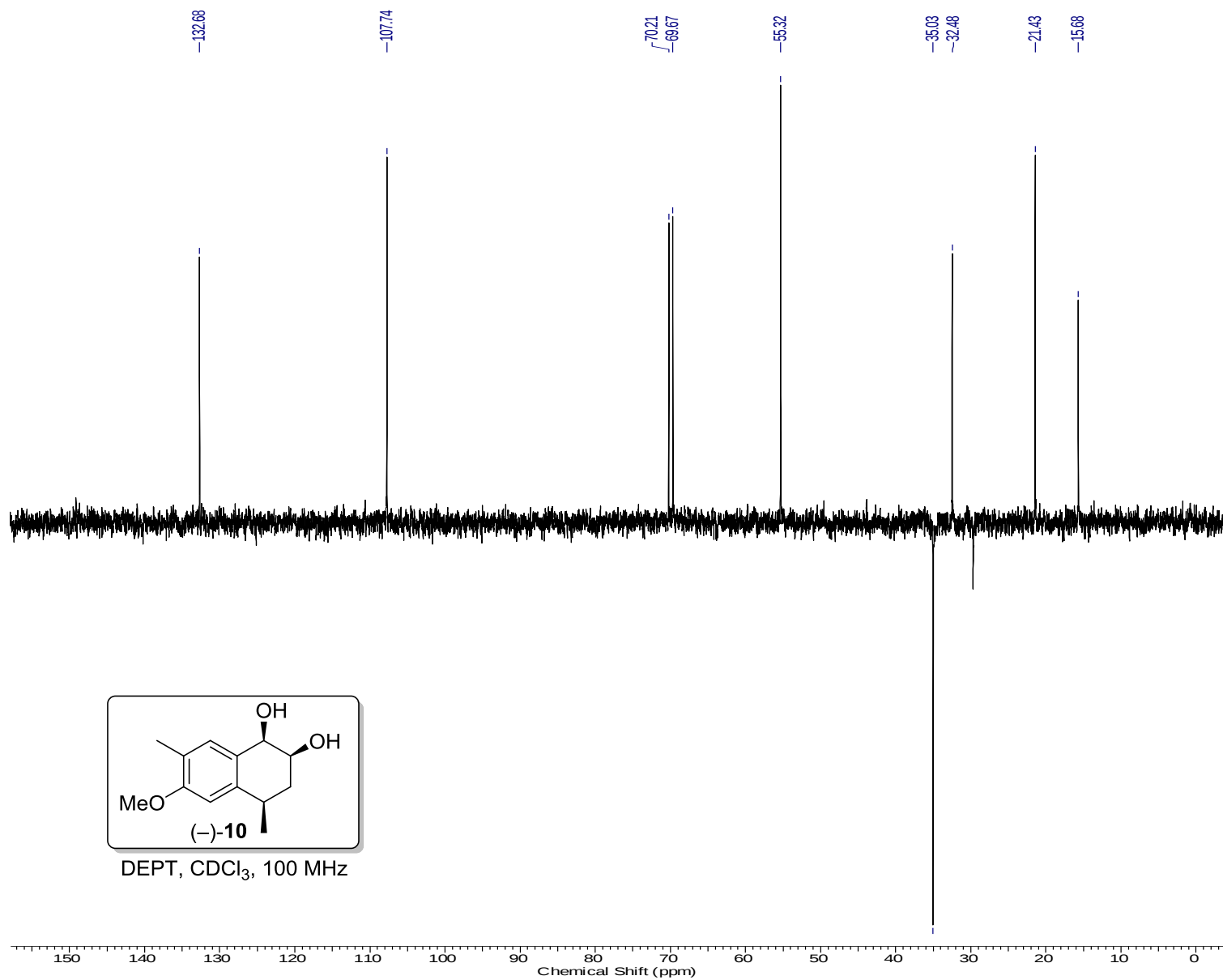


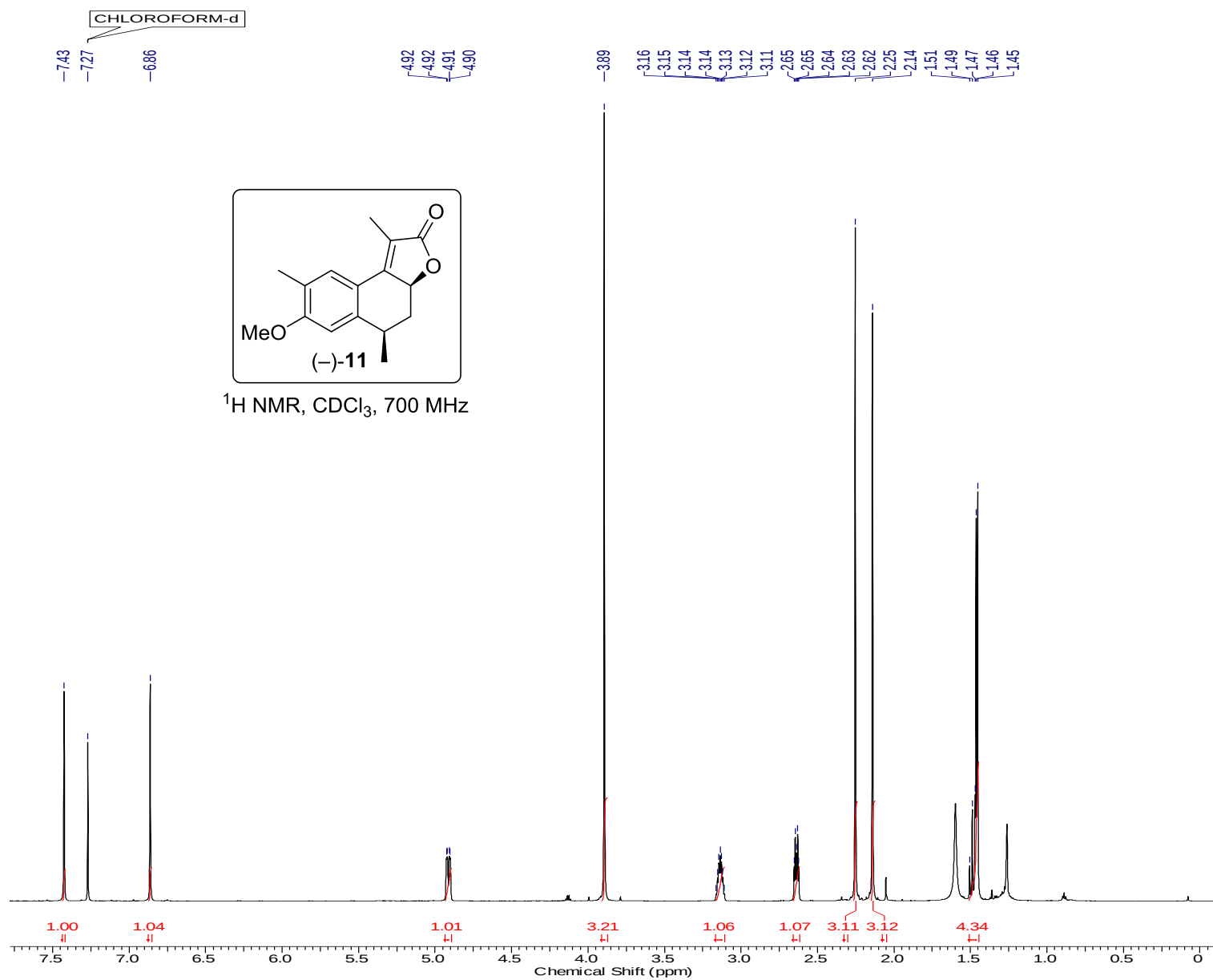


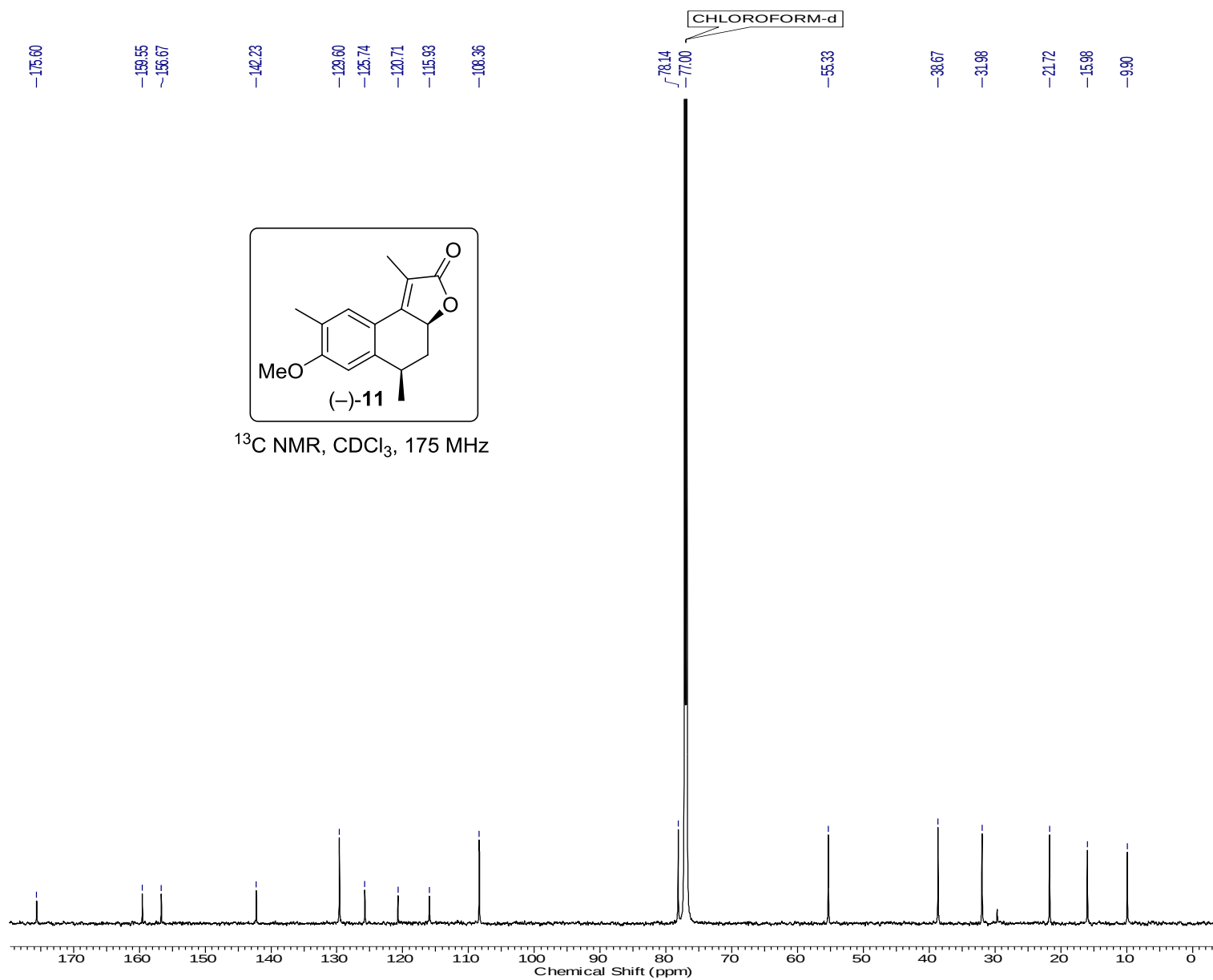


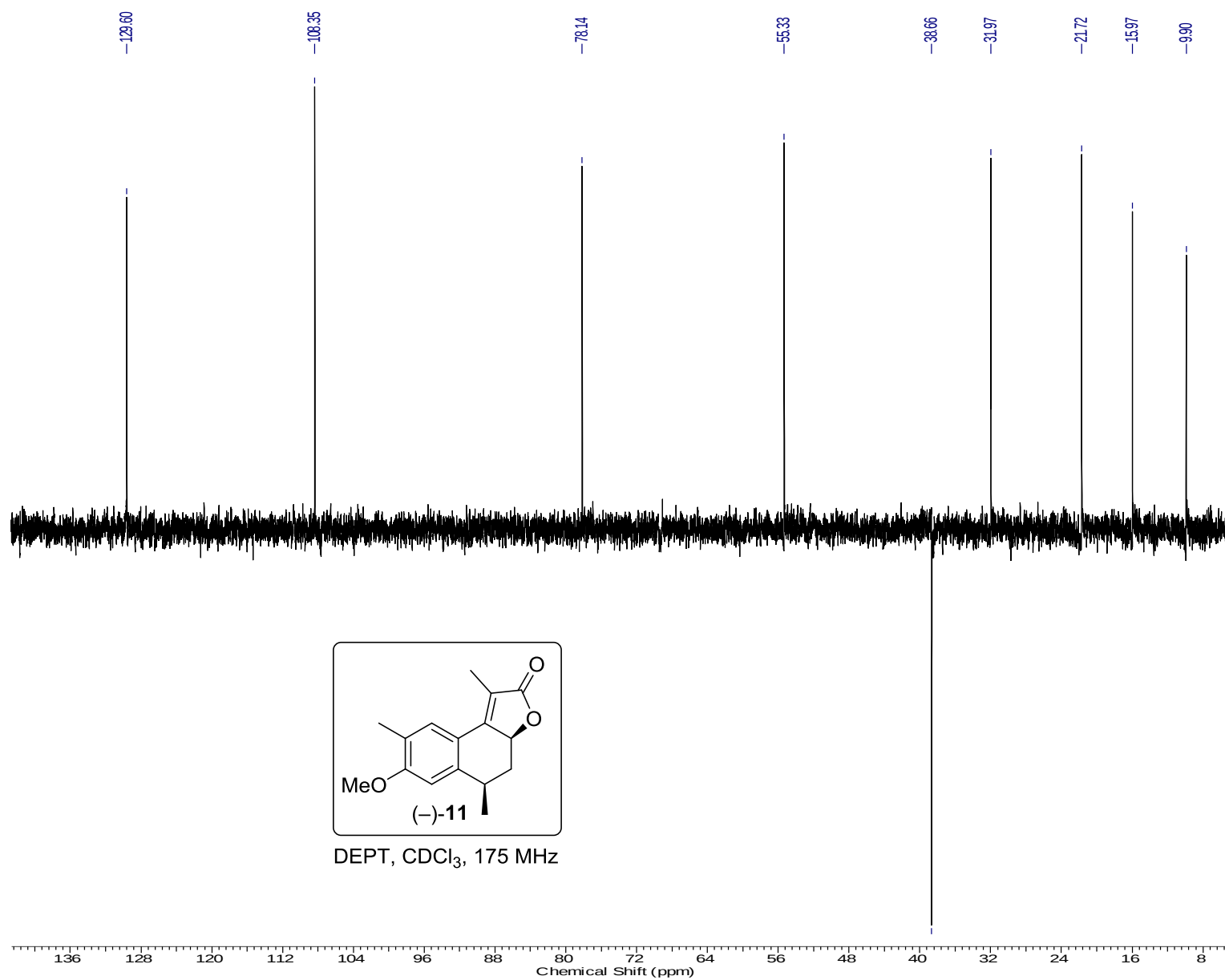


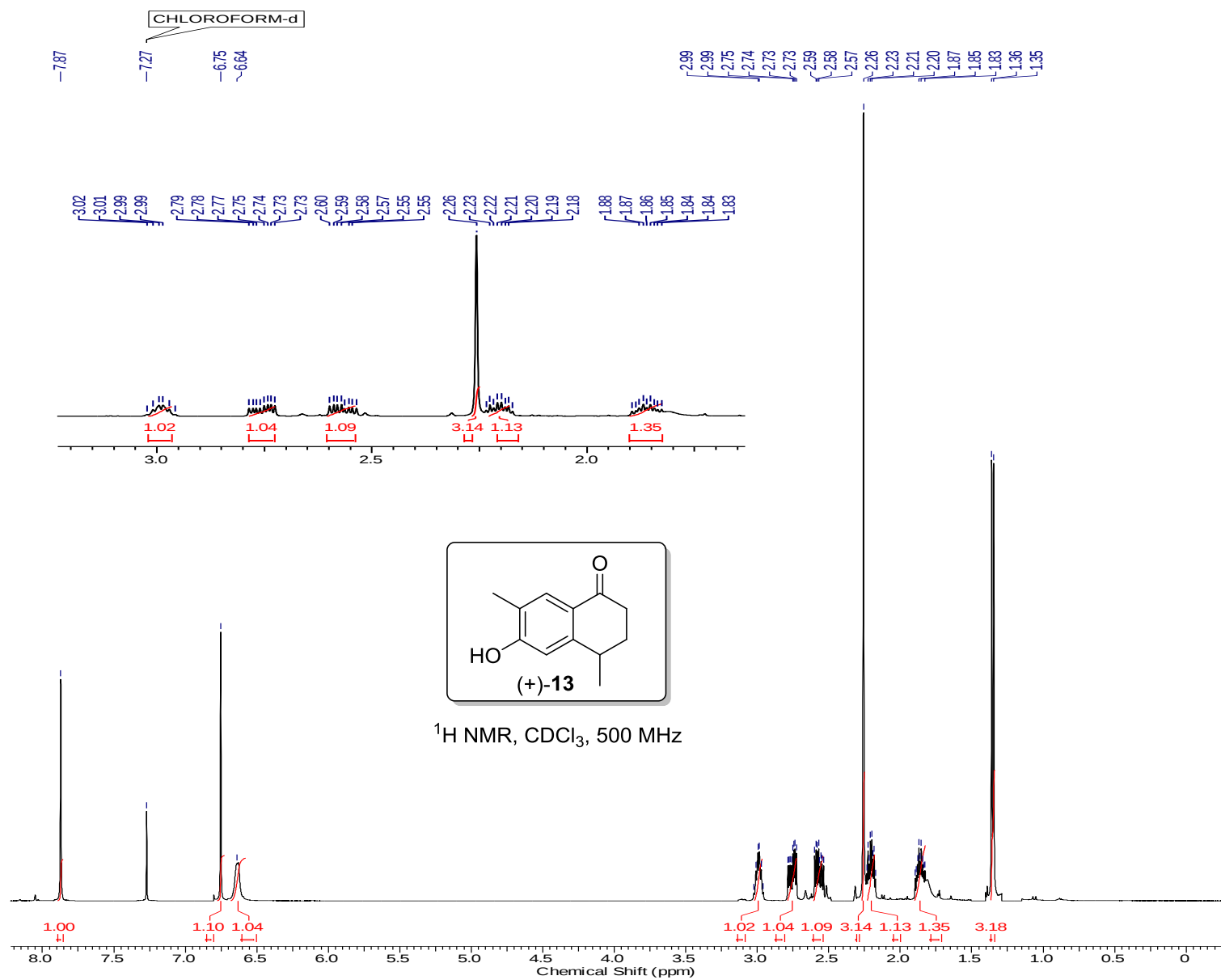


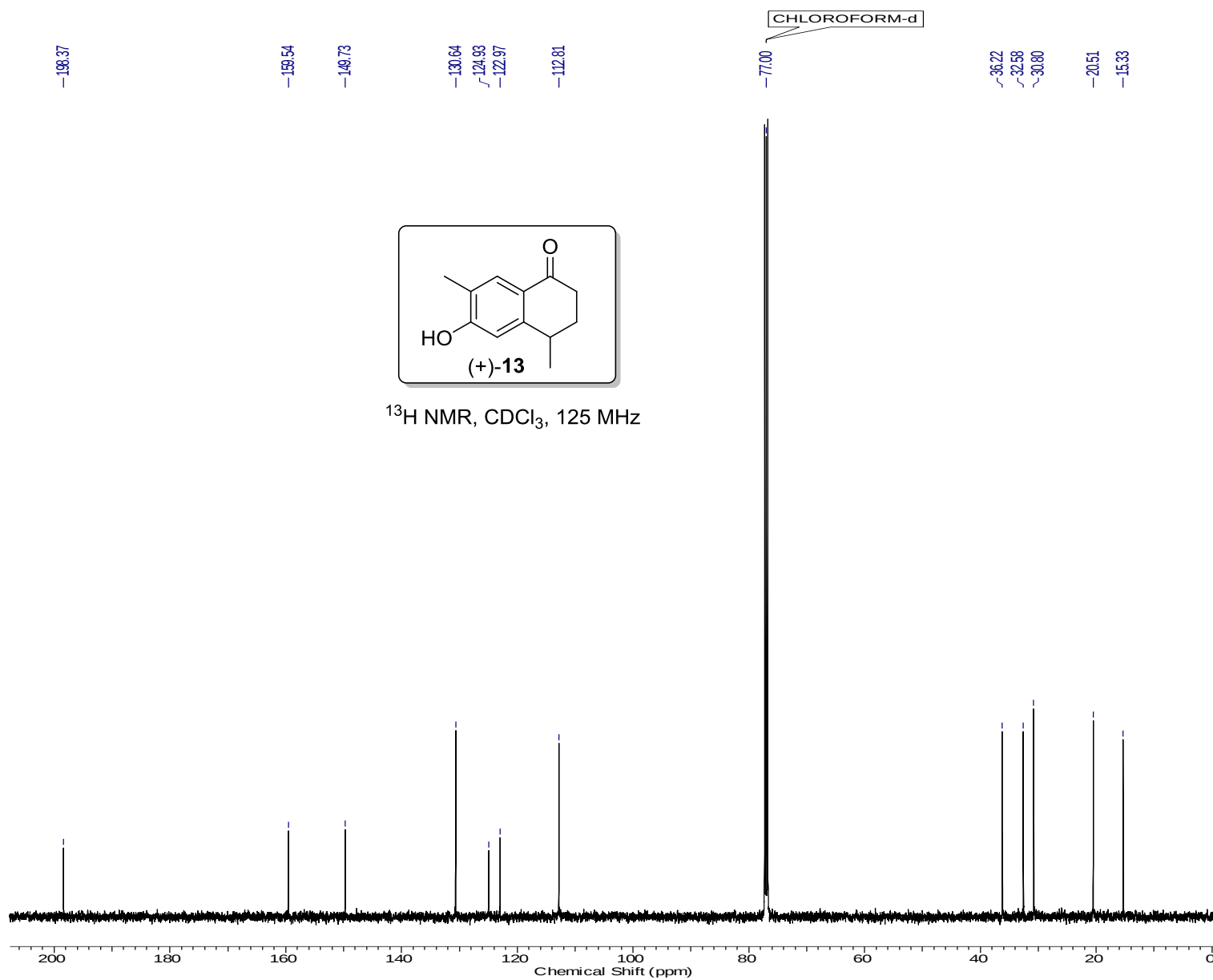


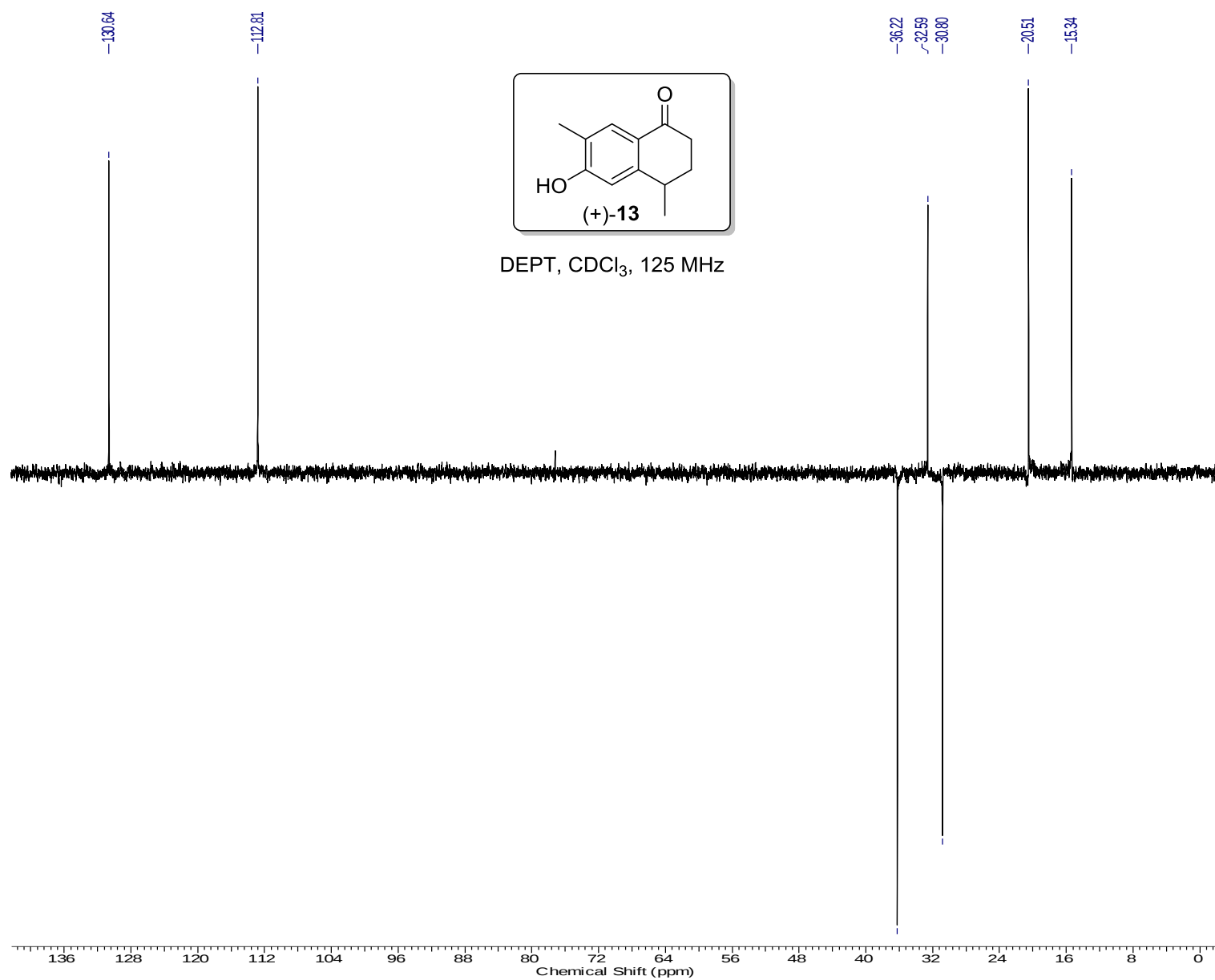


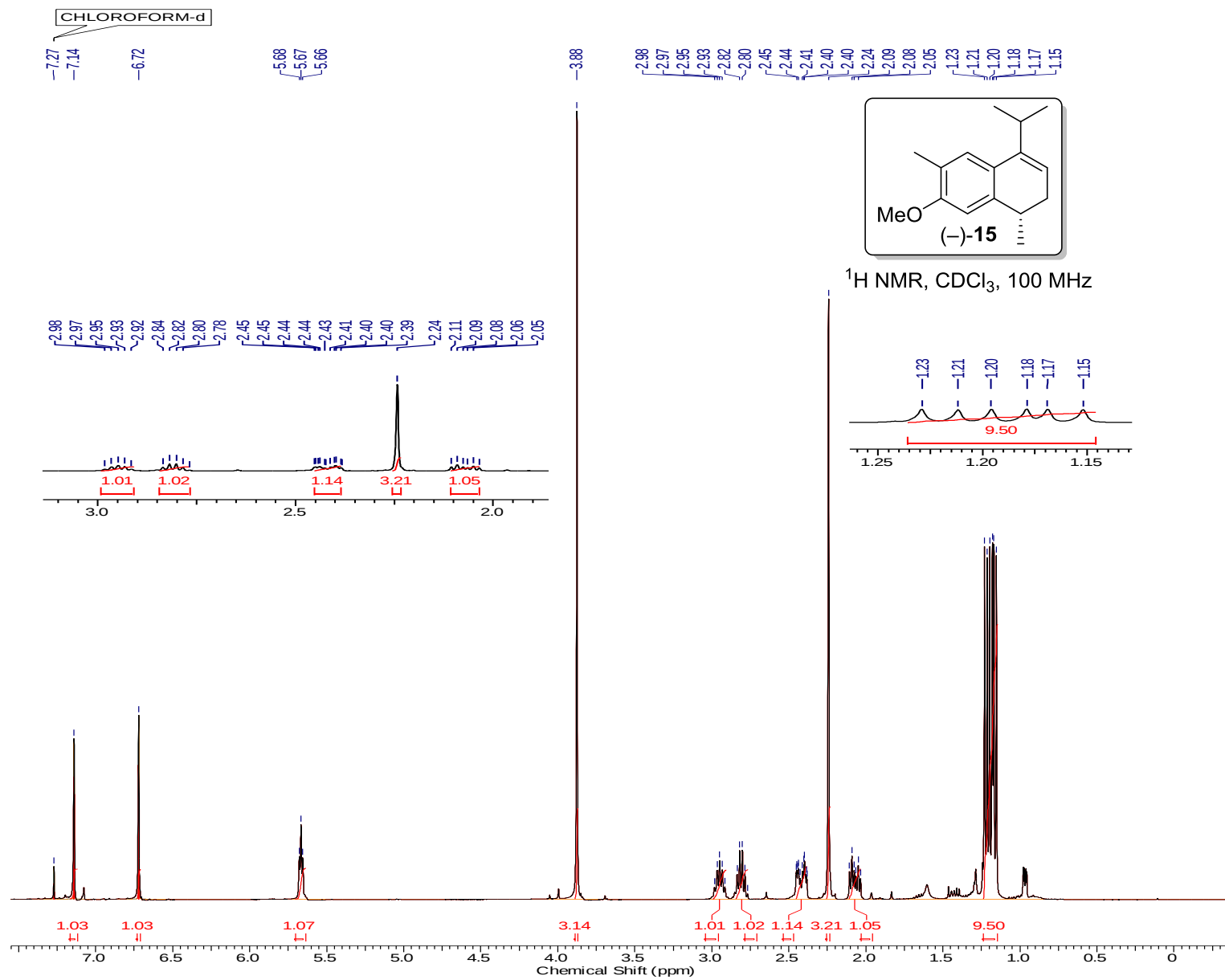


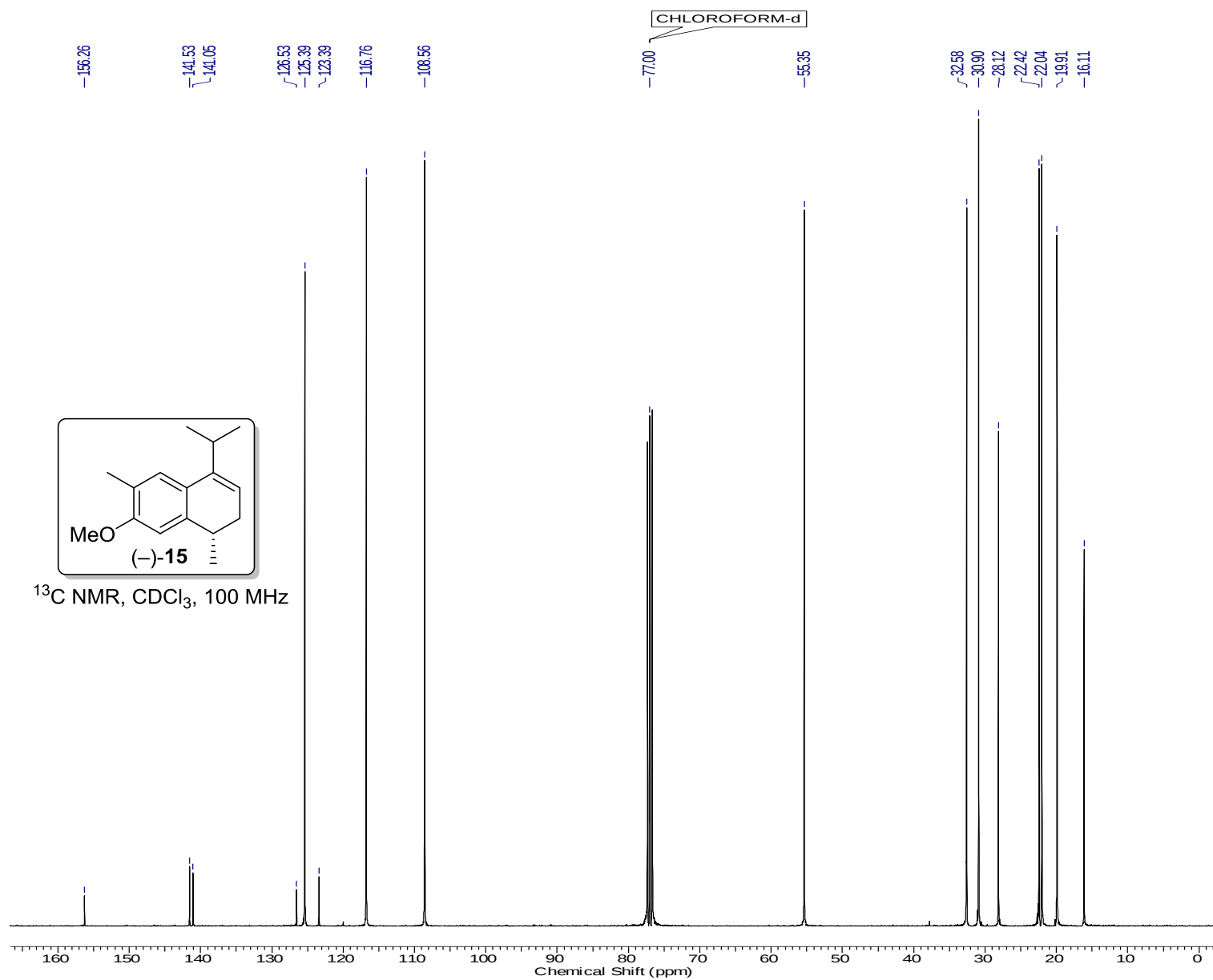


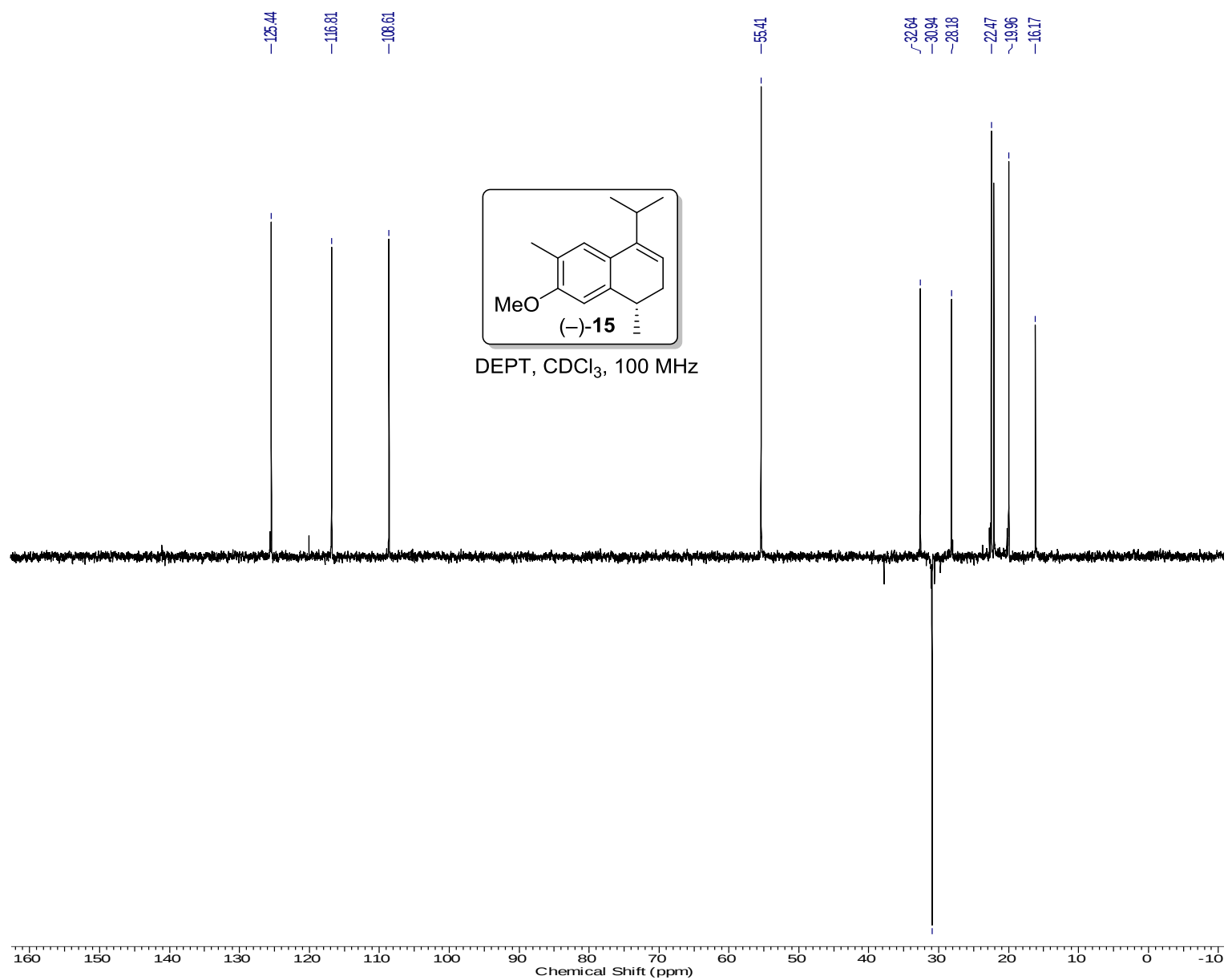


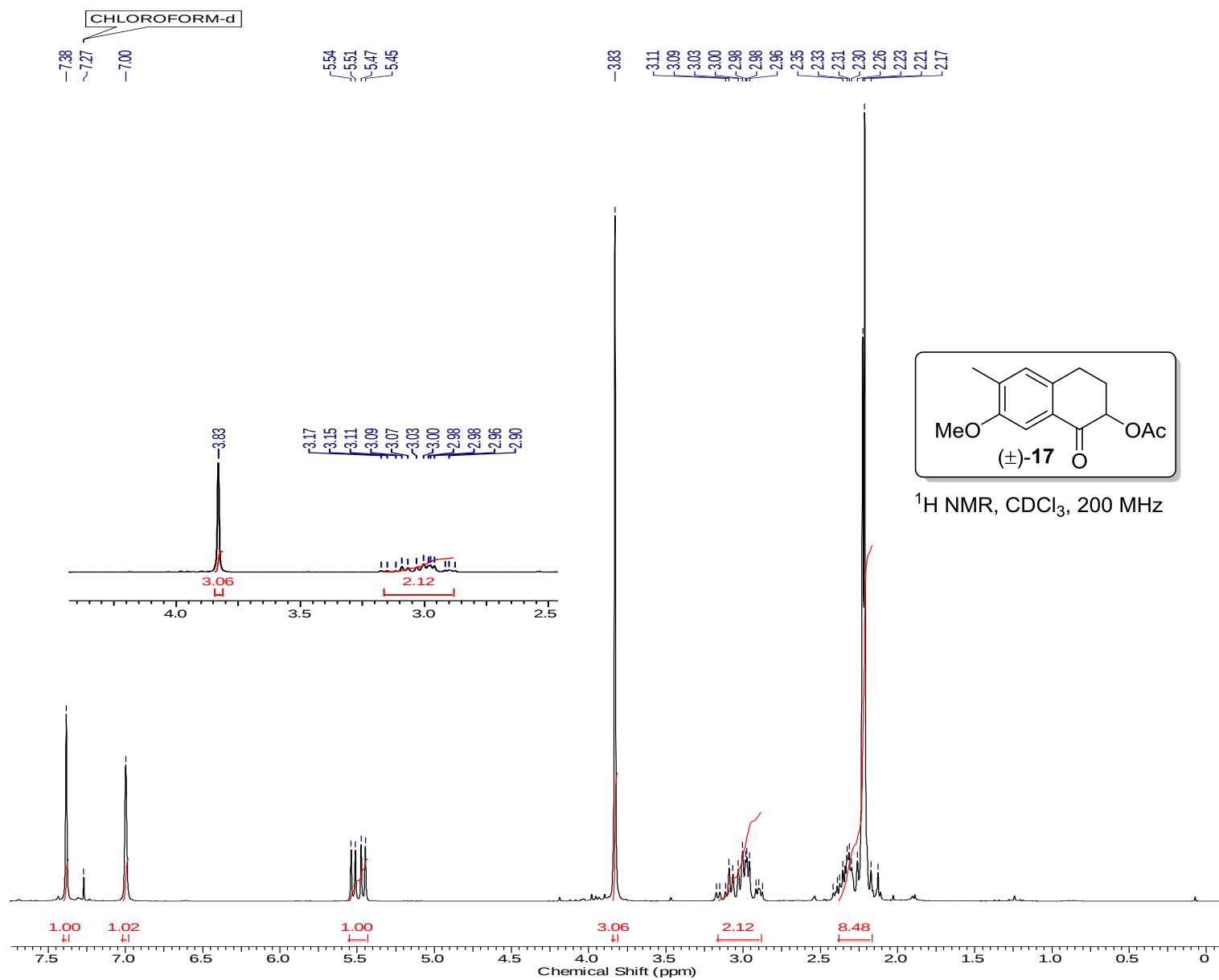


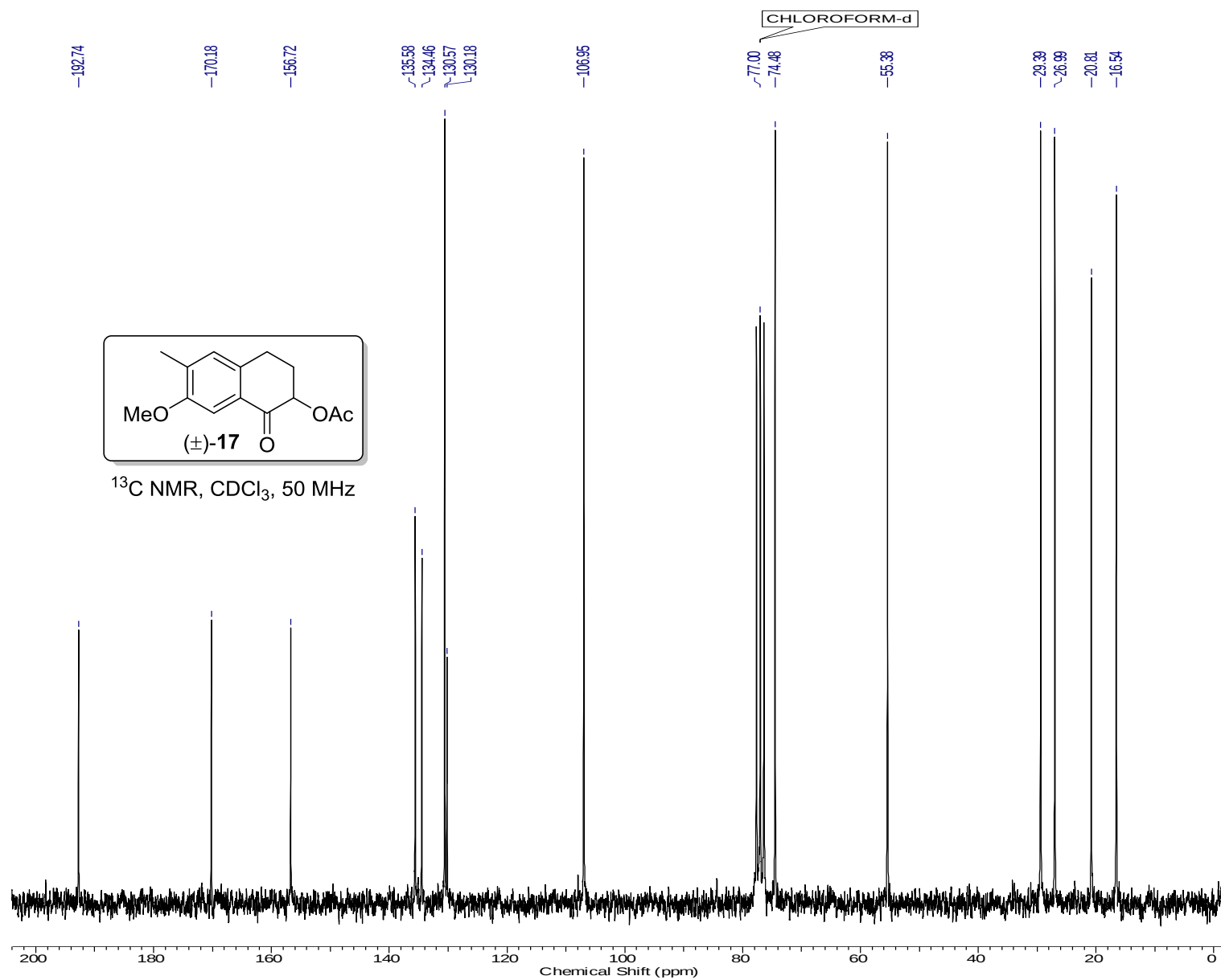


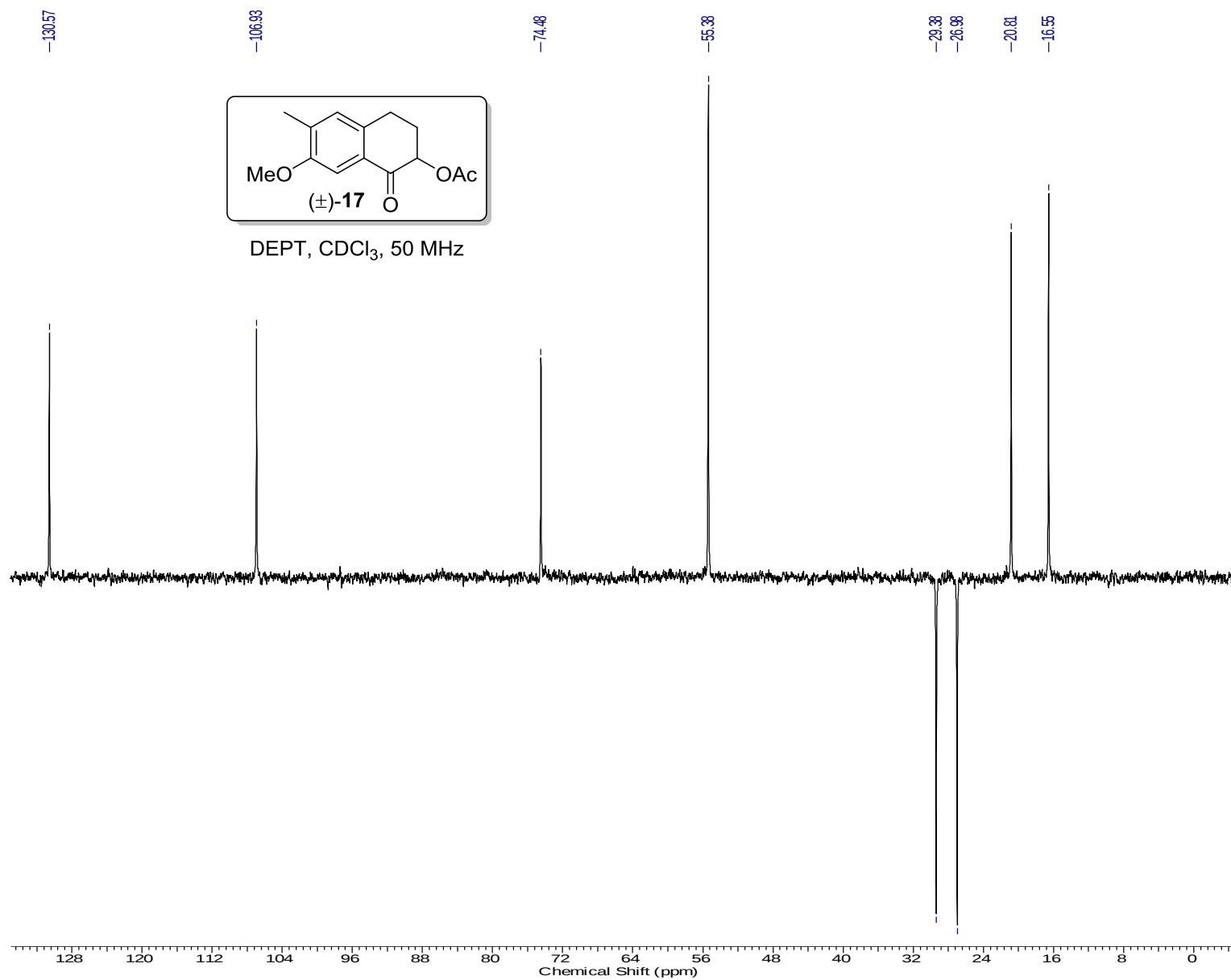


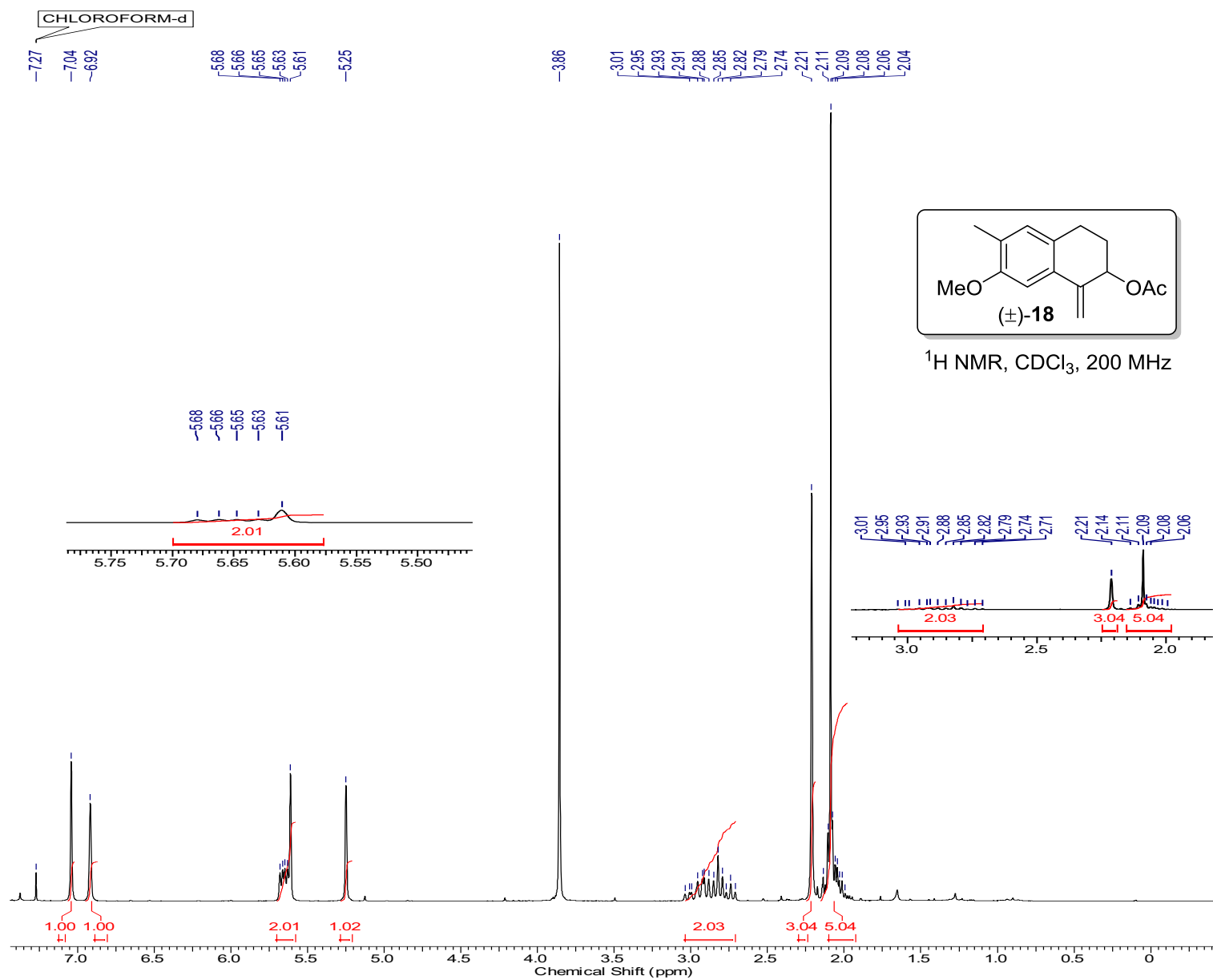


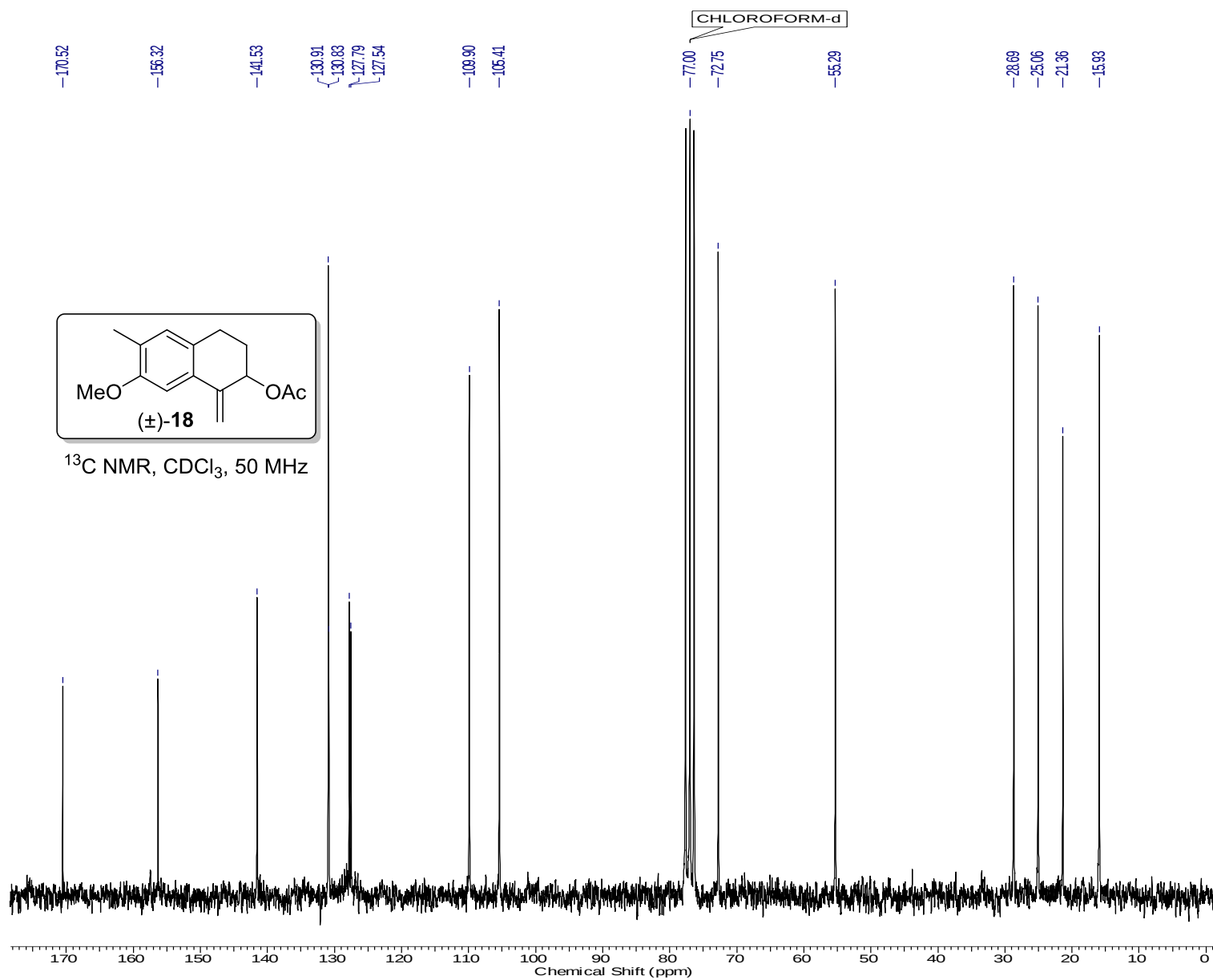


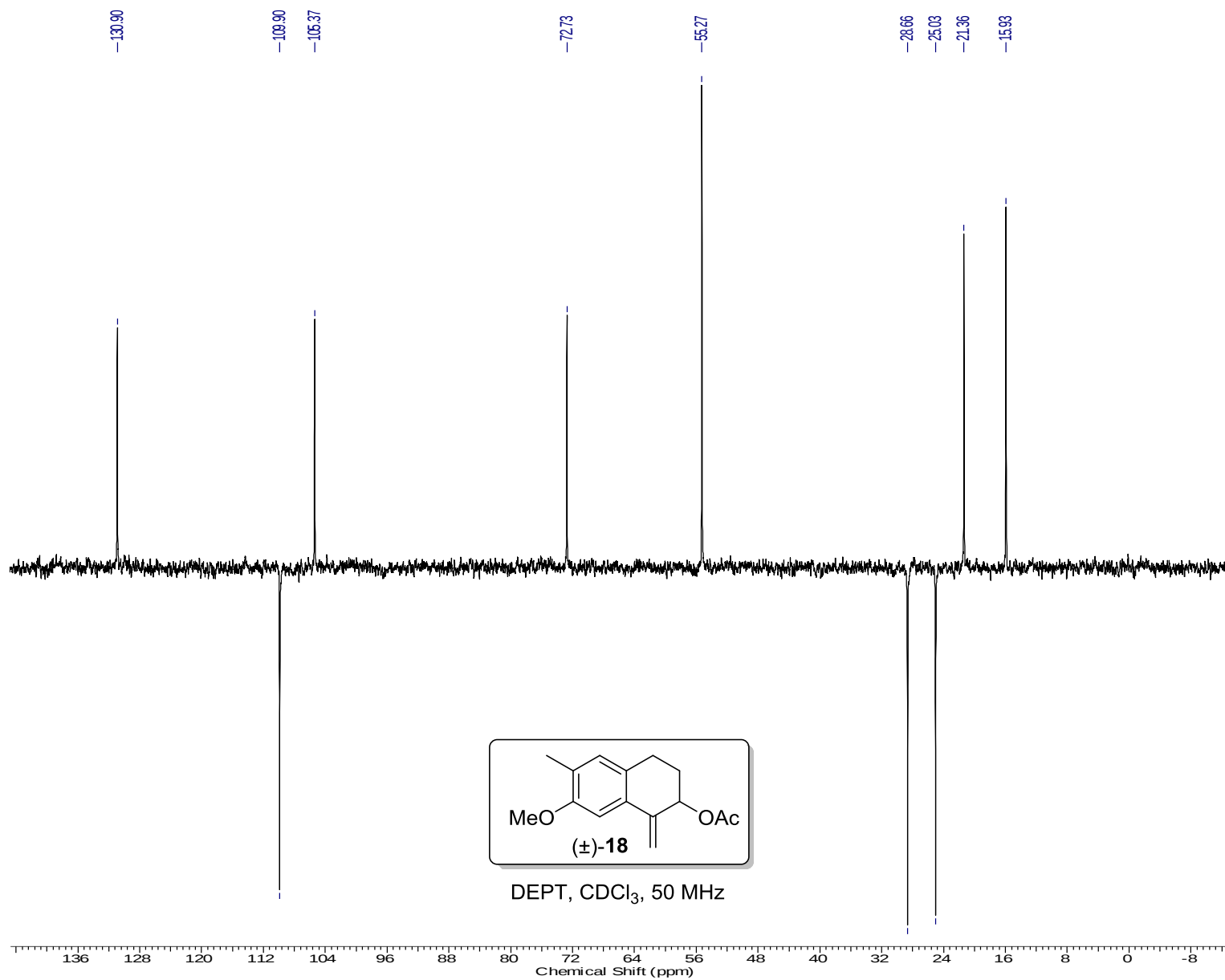


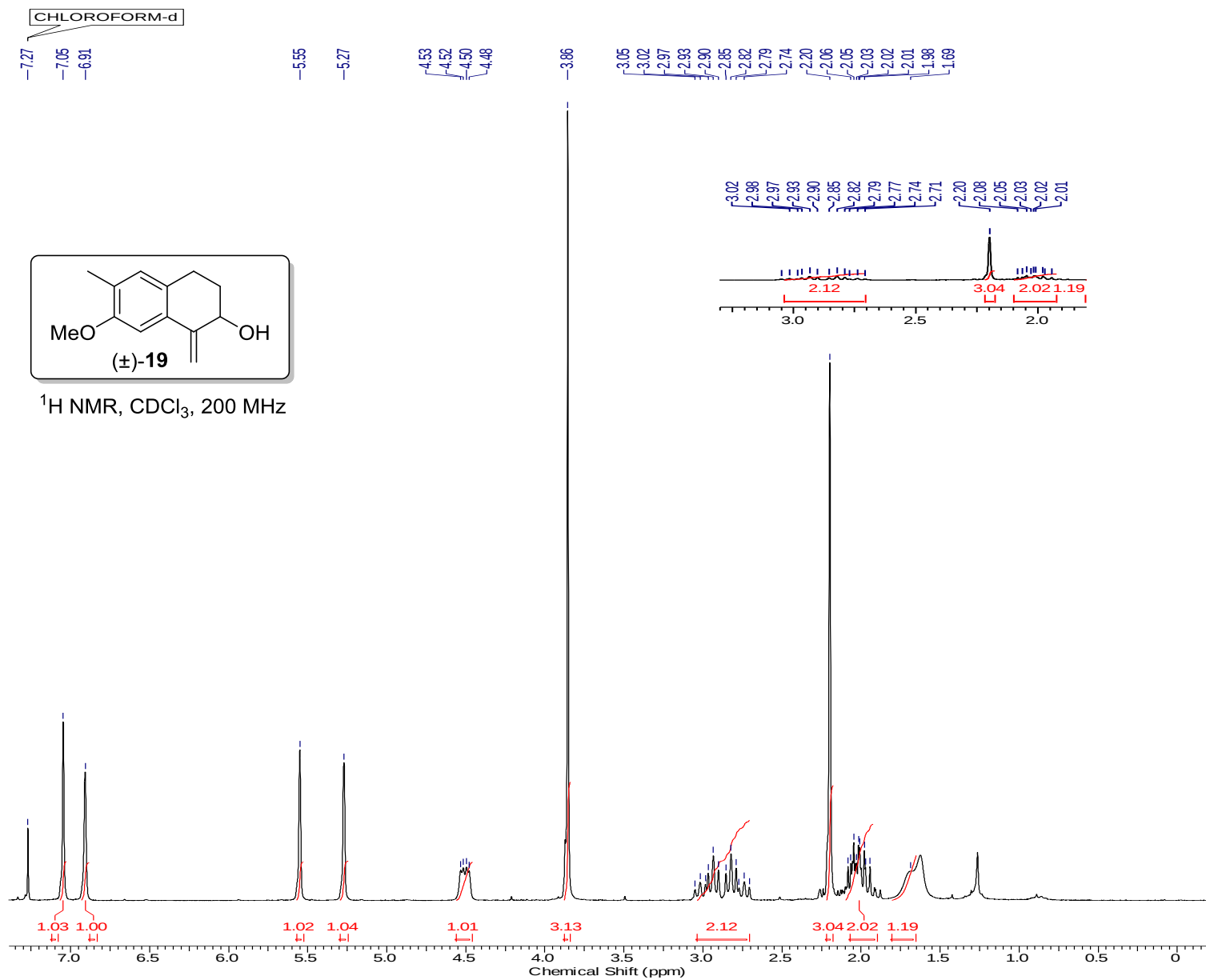


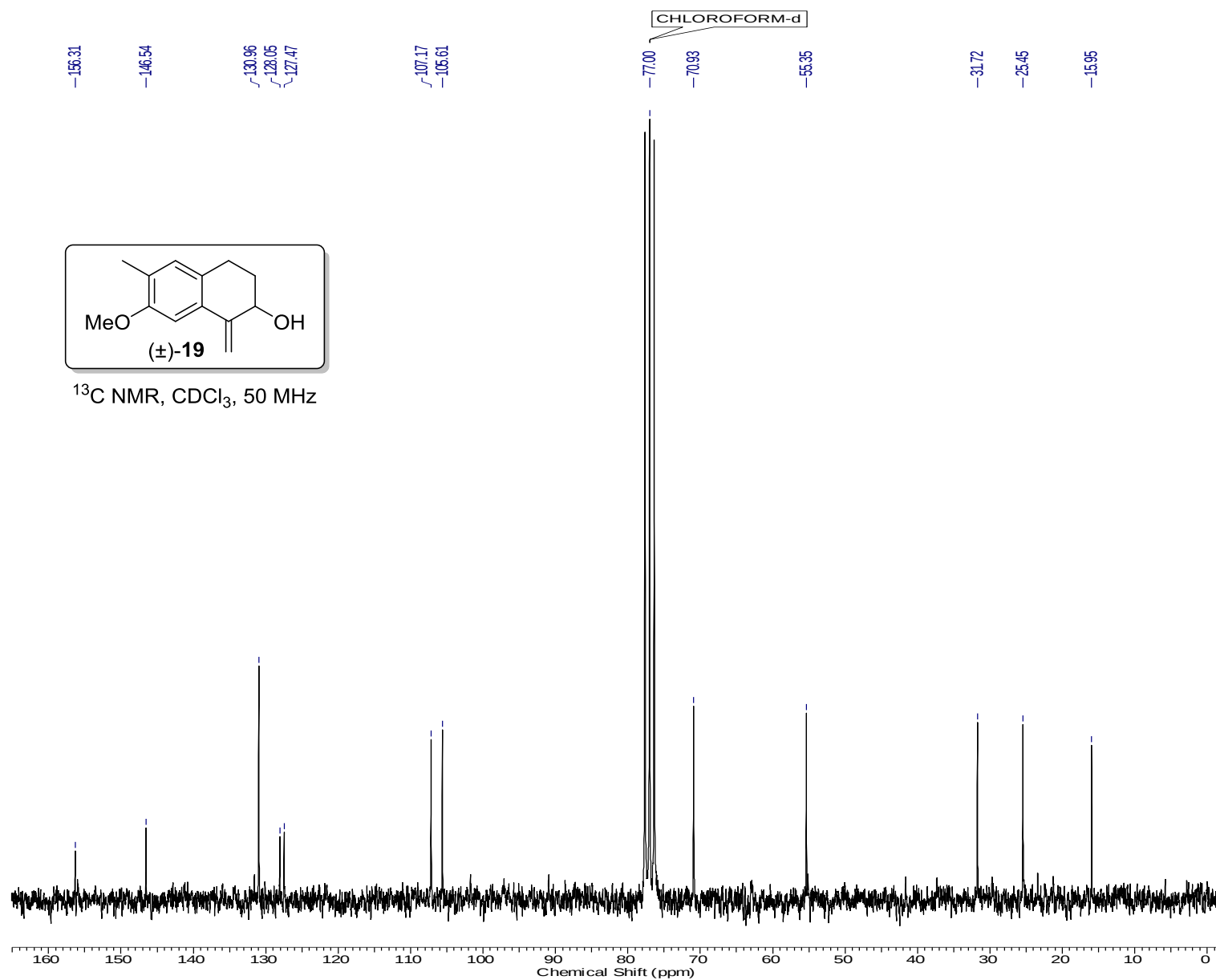


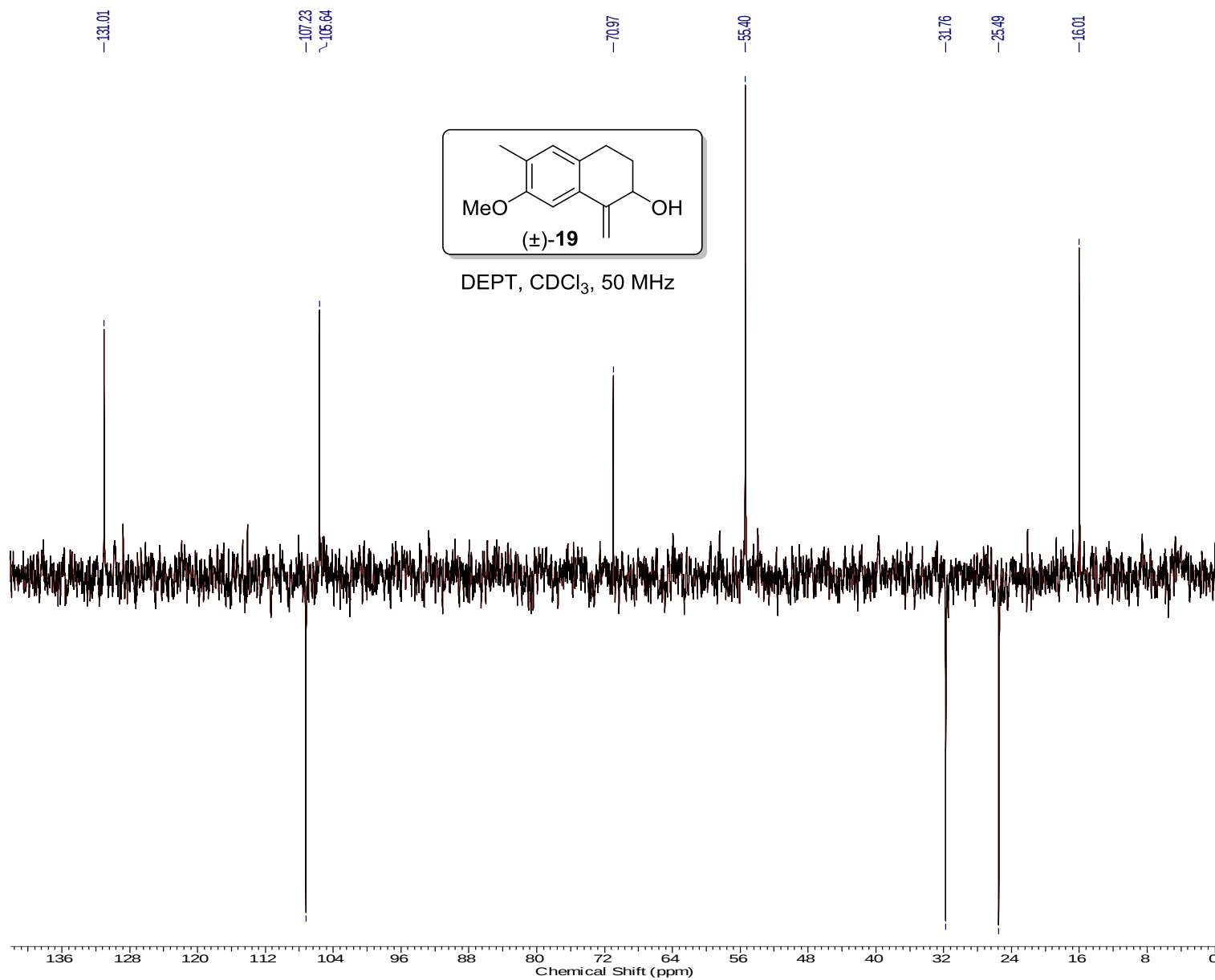


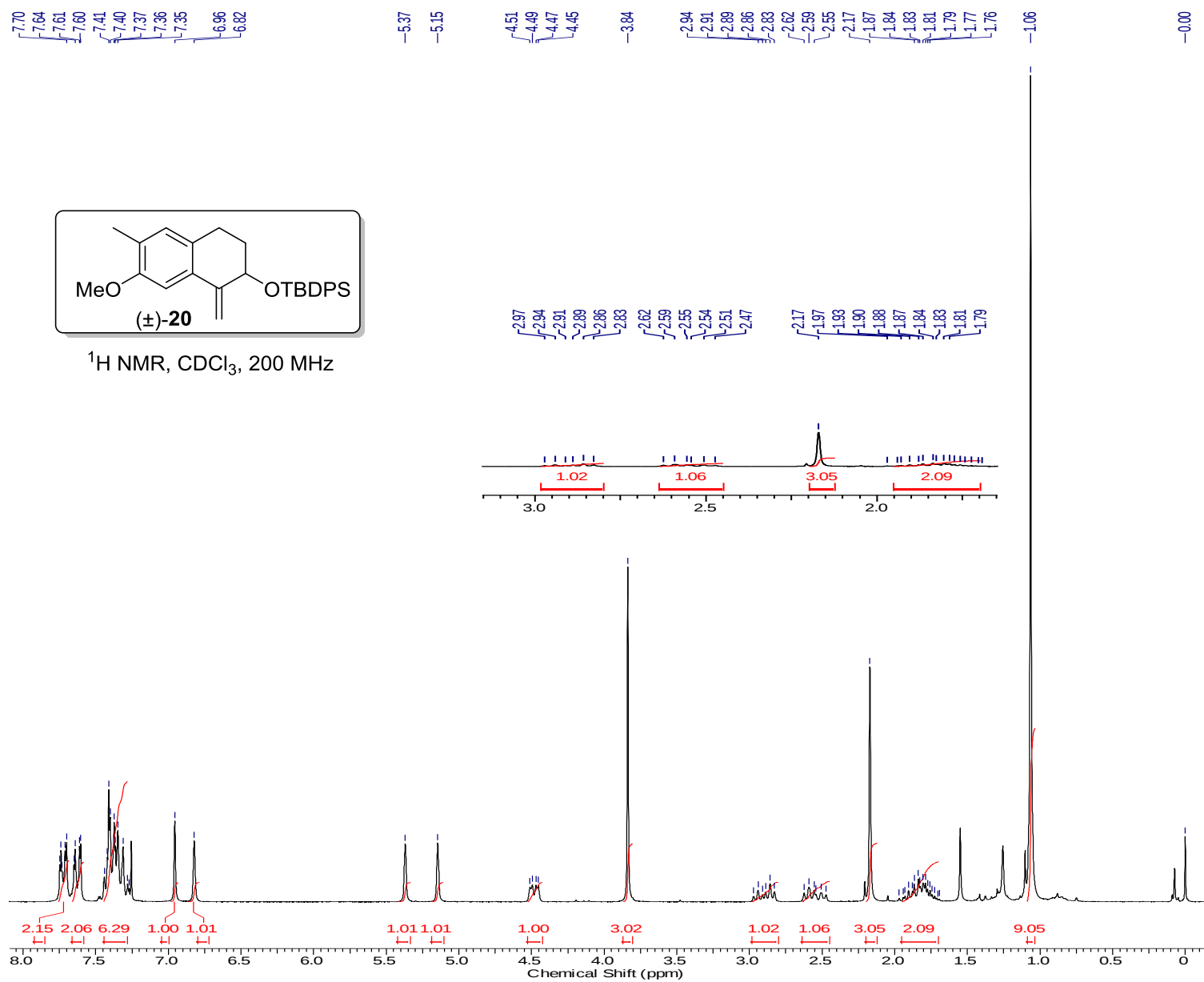


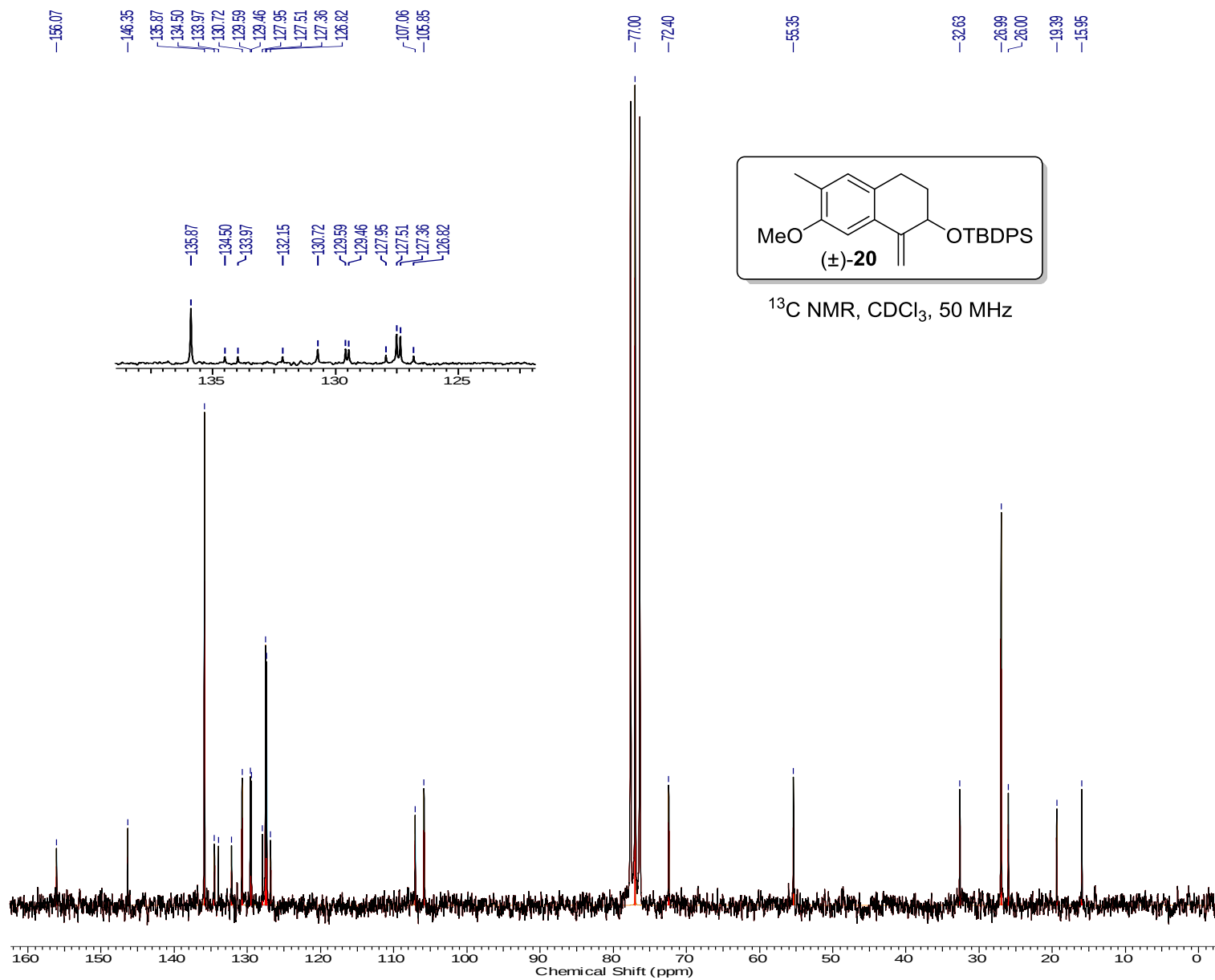


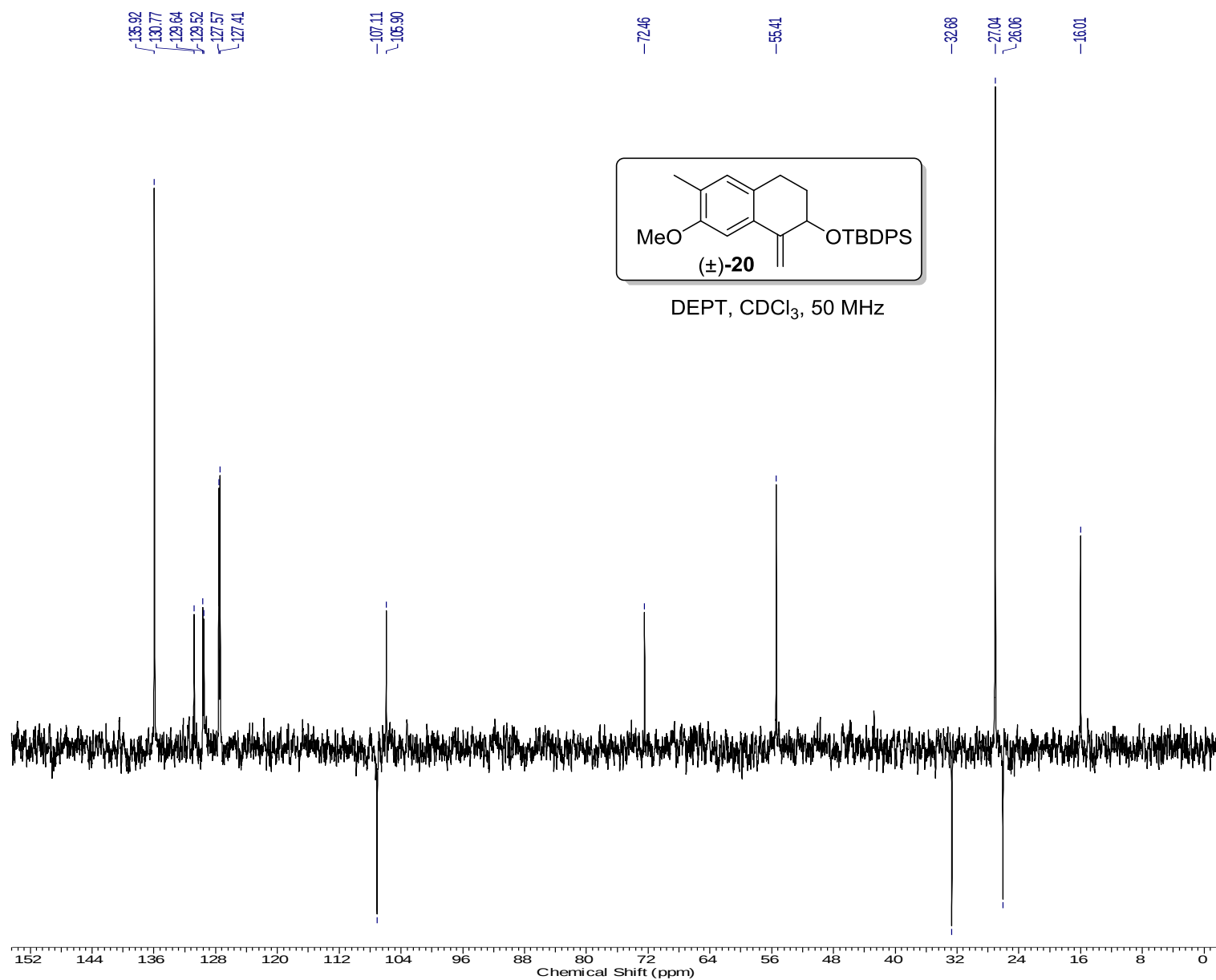


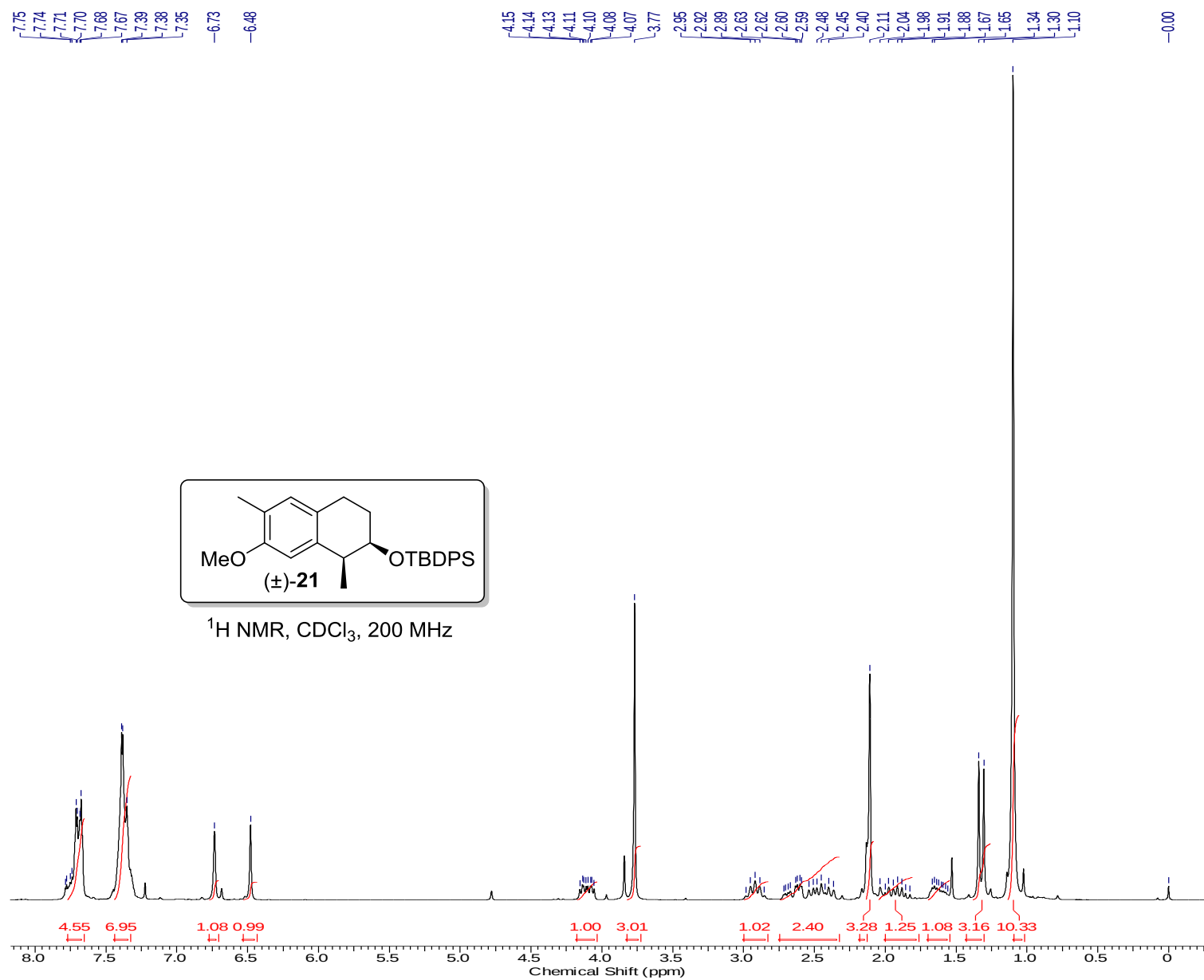


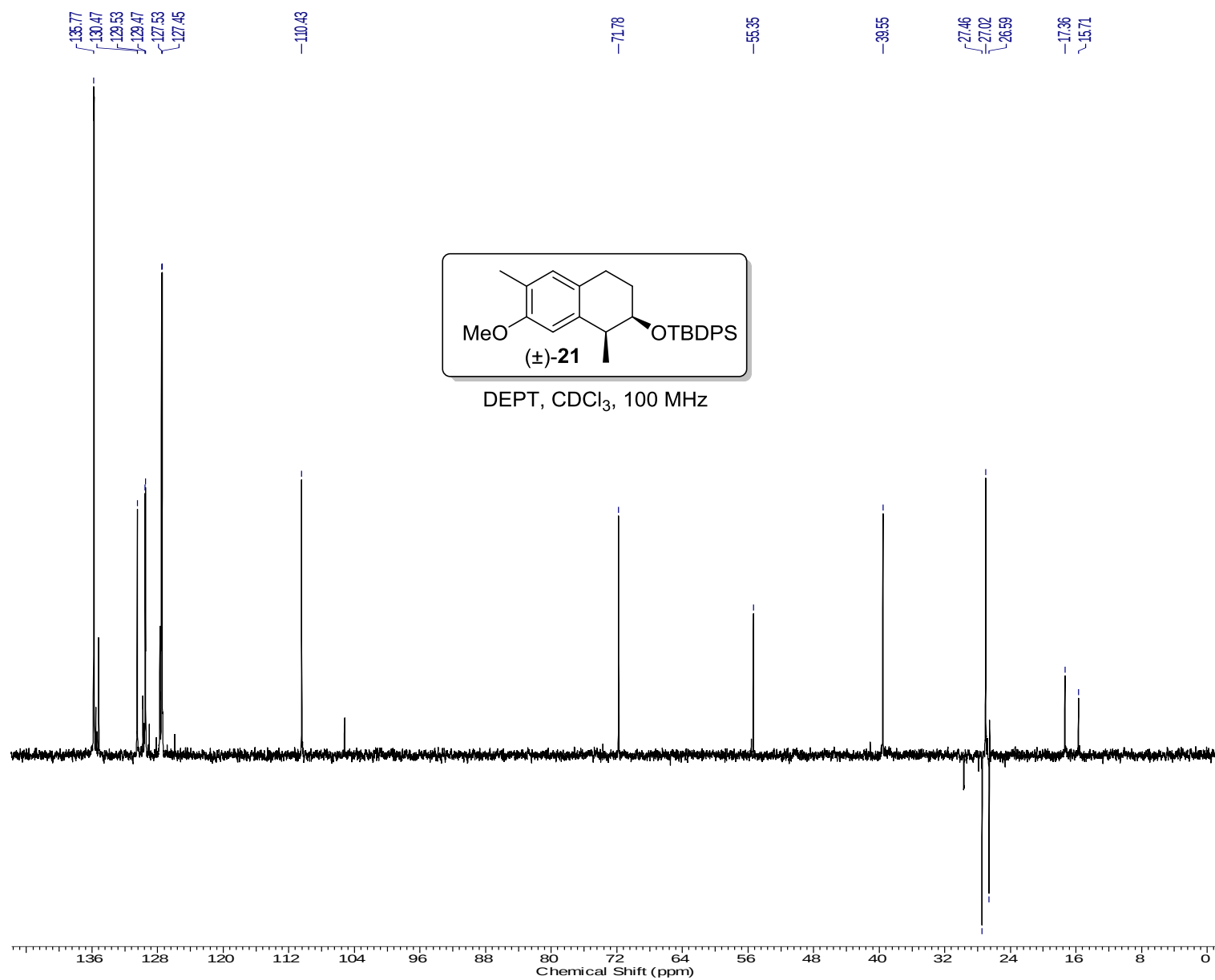


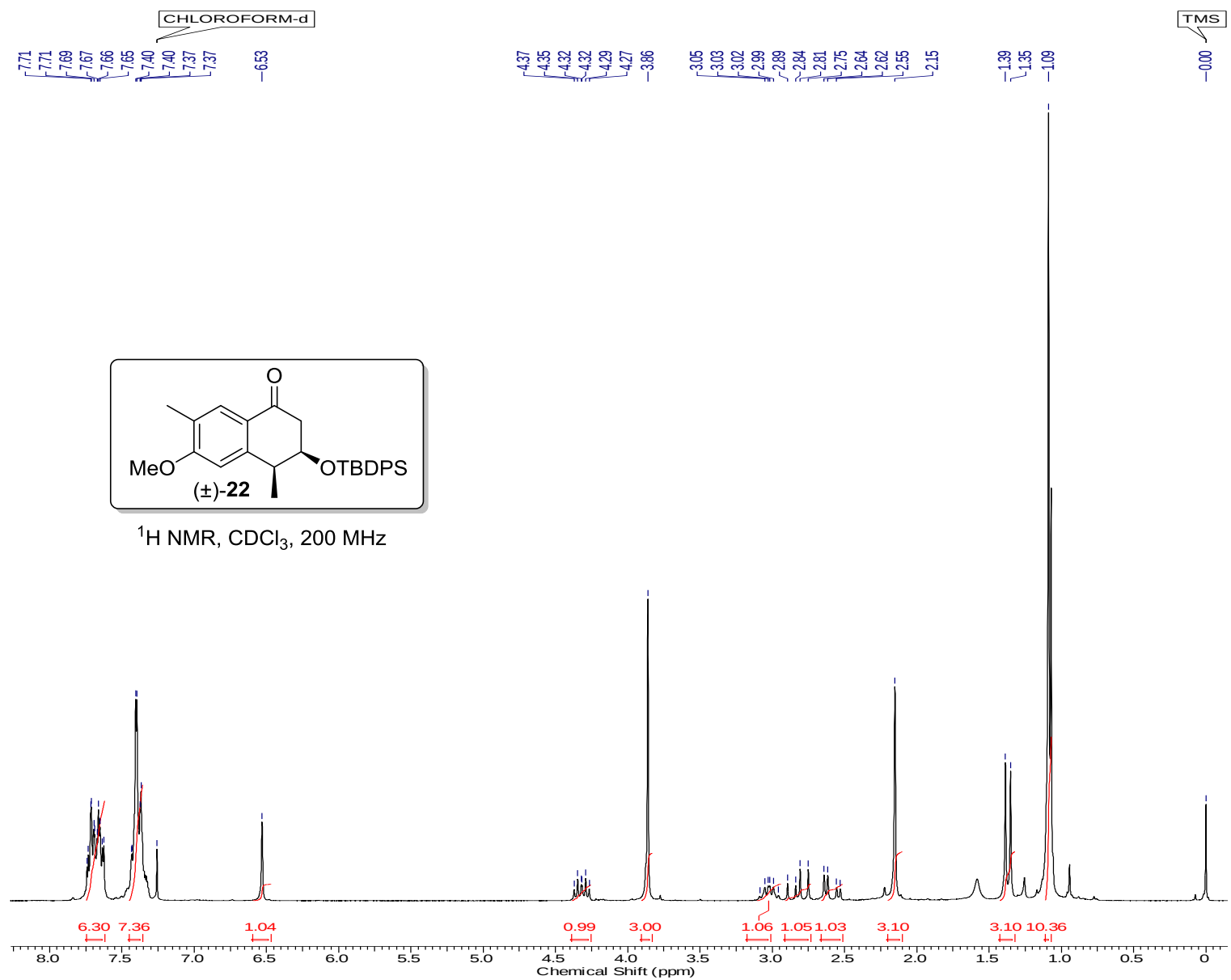


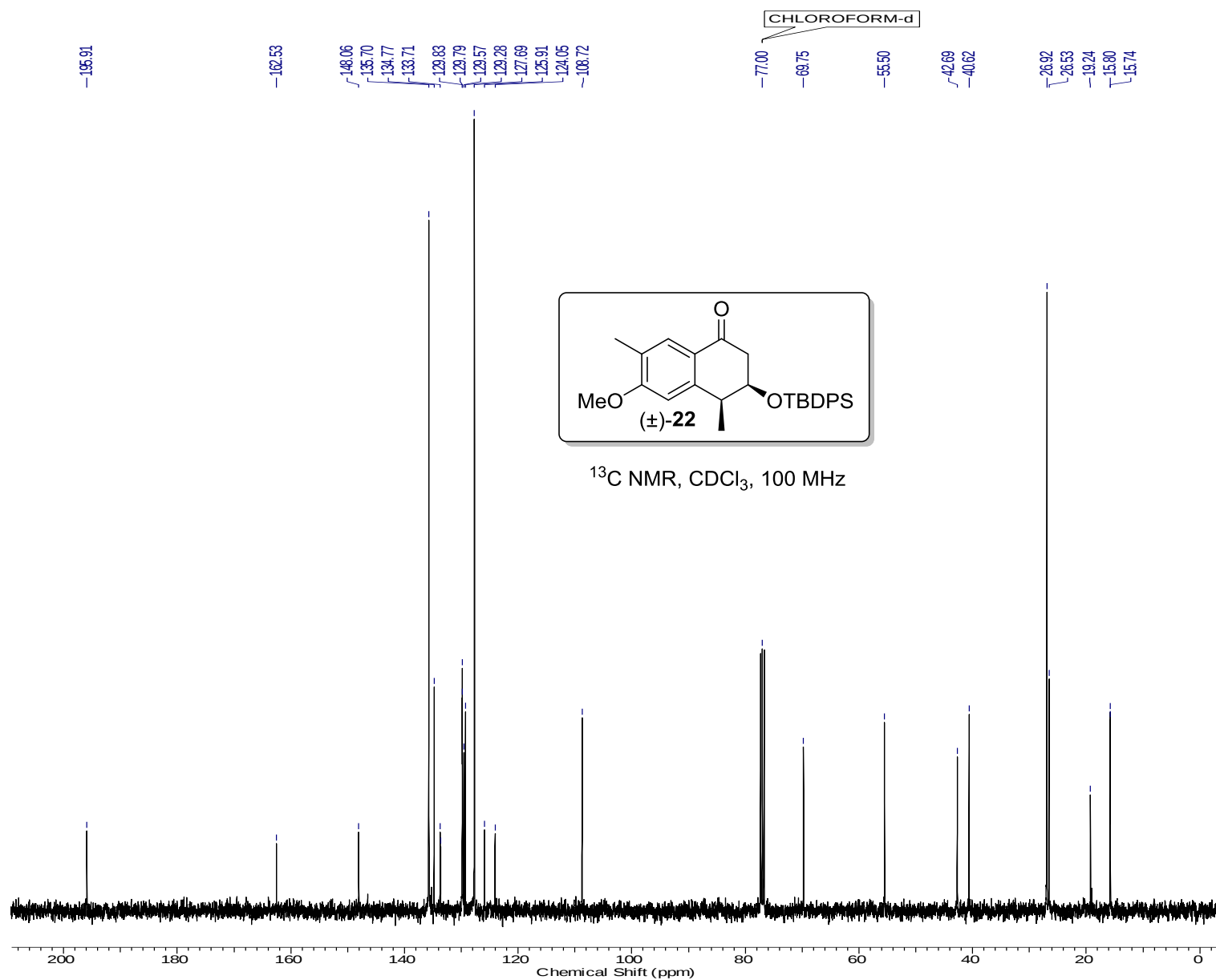


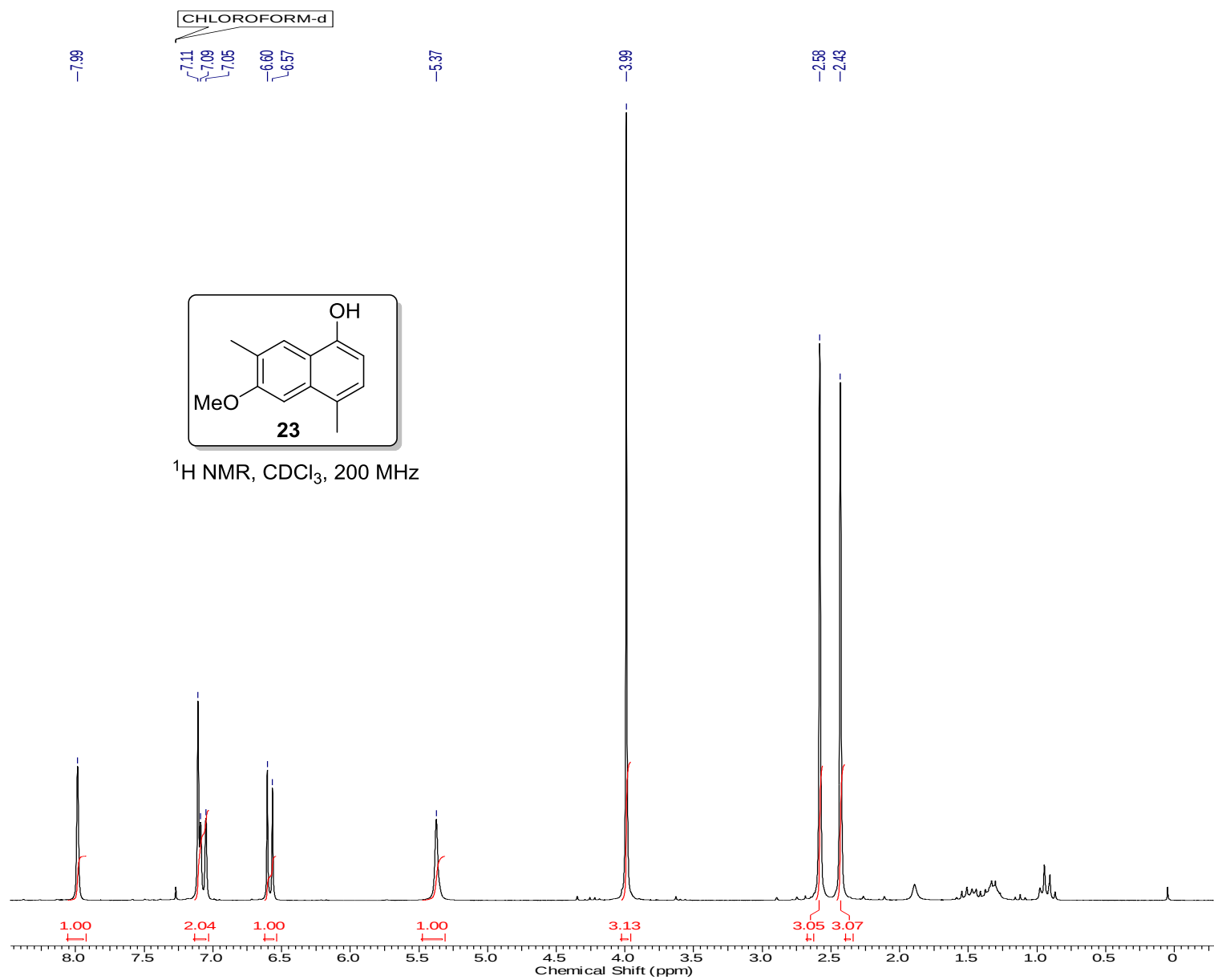


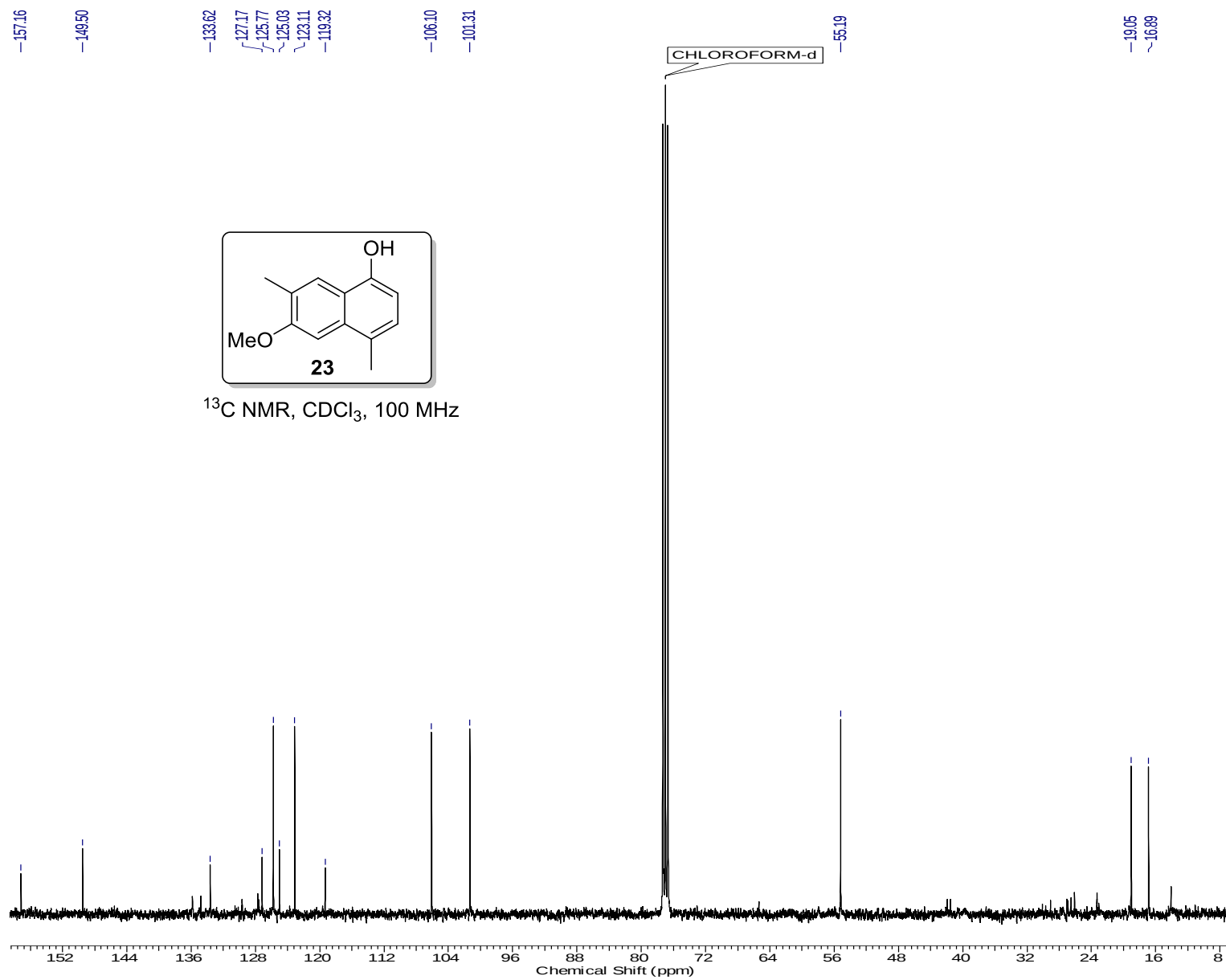


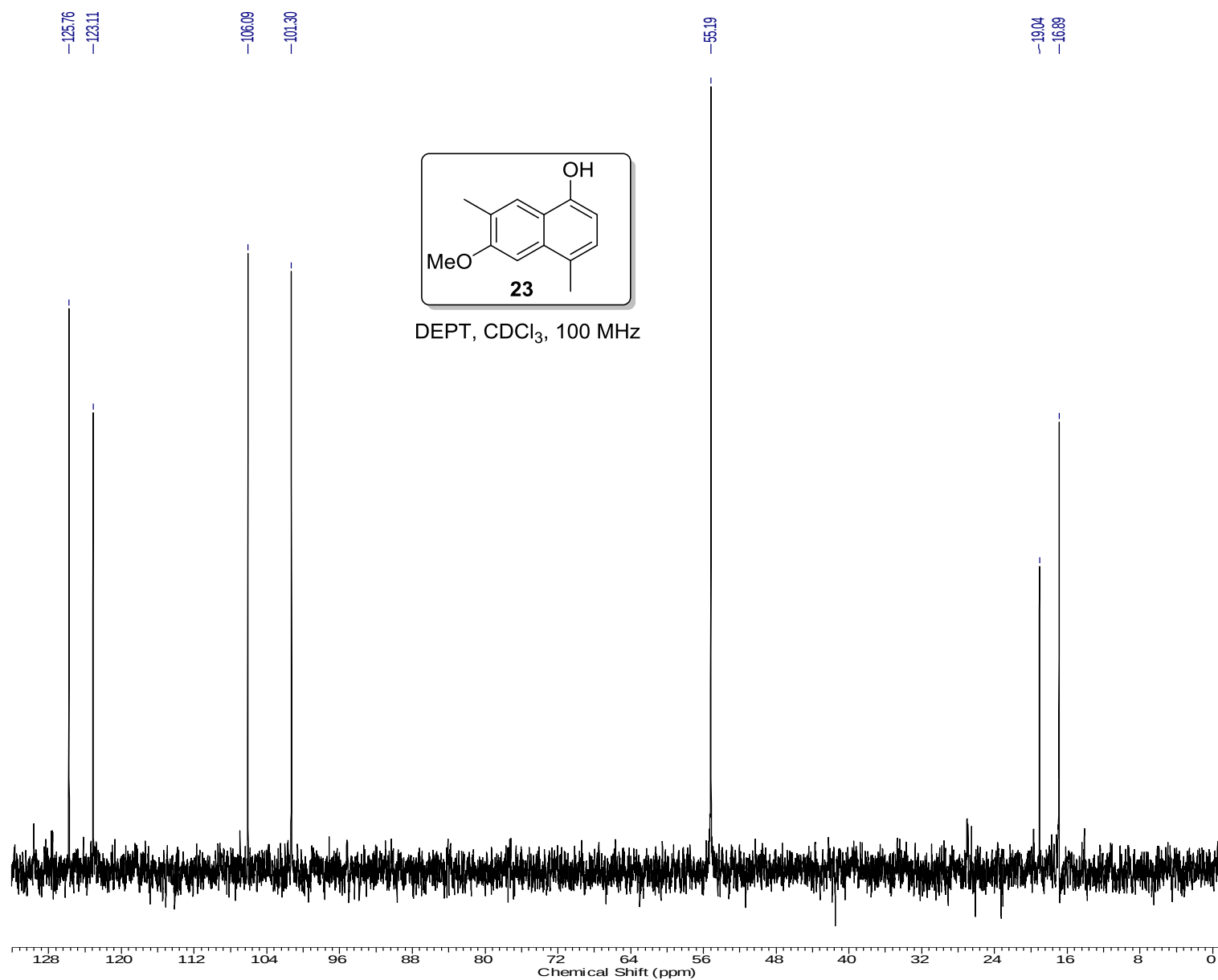


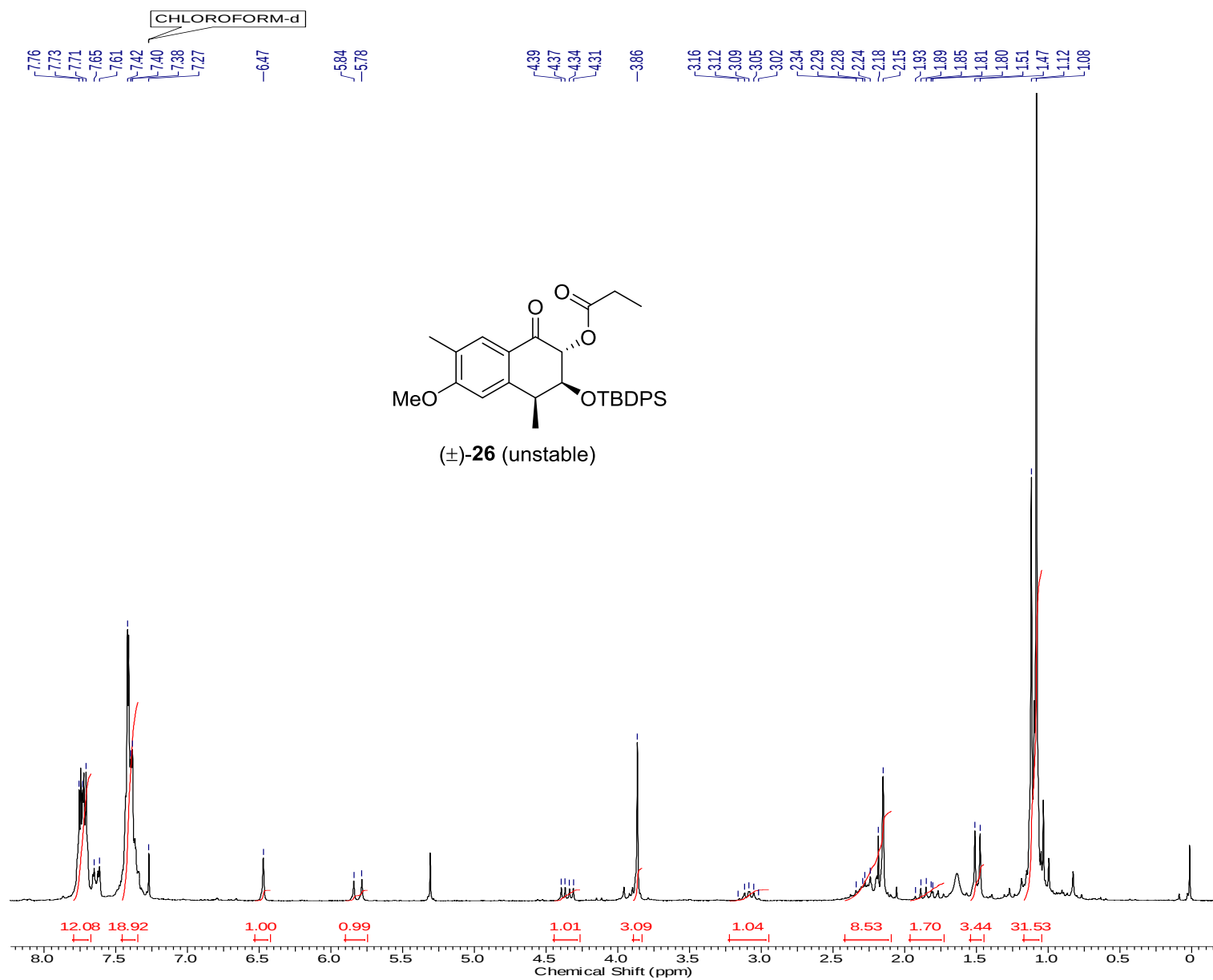












2/19/2015 2:27:21 PM

531.2561
R=48907
C₃₂ H₃₉ O₅ Si = 531.2561
-0.0447 ppm

Relative Abundance

100
95
90
85
80
75
70
60
55
50
45
40
35
30
25
20
15
10
5
0

498.9001
R=41300

531.2561
R=48907

550.7312
R=46702

553.2379
R=48207

562.2979
R=47607

569.2125
R=44107

m/z

495 500 505 510 515 520 525 530 535 540 545 550 555 560 565 570

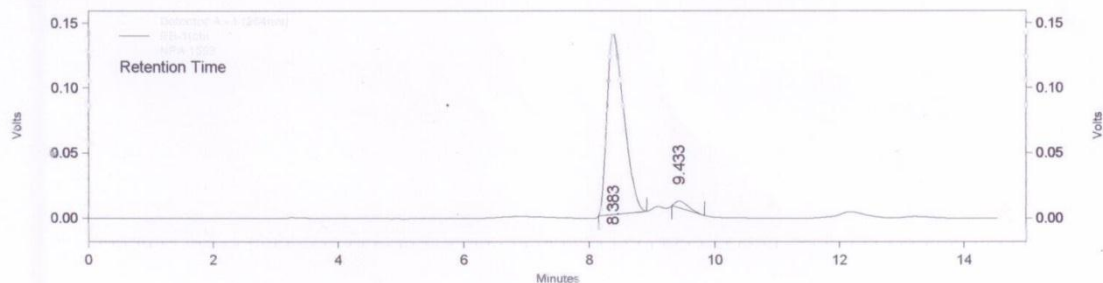
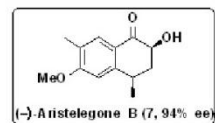
(±)-26

CCOC1=CC=C2C(=C1)C(=O)C(C)(C)[C@H](OC(=O)CC)[C@@H](C)C2

Area % Report

Page 1 of 1

RB-I(chi)
 Method Name: C:\CLASS-VP\Data\Dr. Patil N. TVSS
 Data Name: C:\CLASS-VP\Data\Dr. Argade\NPA 1593
 User: System
 Acquired: 2/21/15 9:12:10 AM
 Printed: 2/21/15 10:06:24 AM



Detector A - 1 (254nm)

Pk #	Retention Time	Area	Area %
1	8.383	2610674	97.114
2	9.433	77584	2.886
Totals		2688258	100.000

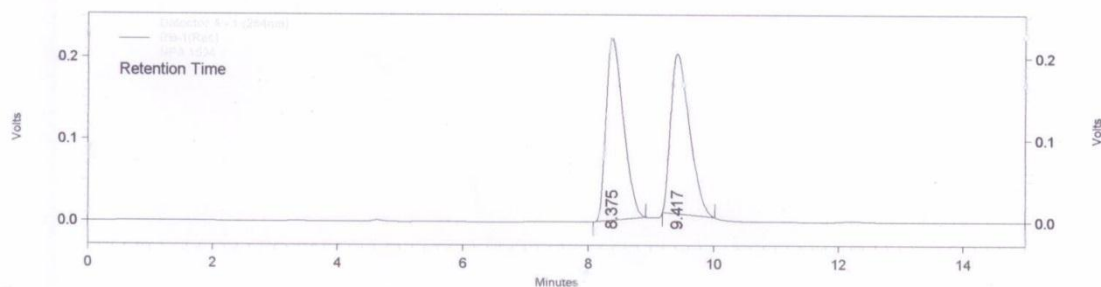
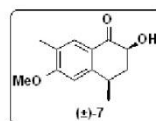
Project Leader :Dr.N. P. Argade
 Column Chiralcel OJ-H (250mm x 4.6mm)
 Mobile Phase :IPA :Pet Ether (10:90)
 Wavelength : 254 nm
 Flow Rate :1 ml/min
 Conc. : 1mg/1ml
 Inj vol- : 5ul

Area % Report

Page 1 of 1

RB-1(Rac)

Method Name: C:\CLASS-VP\Data\Dr. Patil N. T\VS
Data Name: C:\CLASS-VP\Data\Dr. Argade\NPA 1594
User: System
Acquired: 2/21/15 9:28:30 AM
Printed: 2/21/15 10:07:55 AM



Detector A - 1 (254nm)

PK #	Retention Time	Area	Area %
1	8.375	4284704	51.180
2	9.417	4087200	48.820
Totals		8371904	100.000

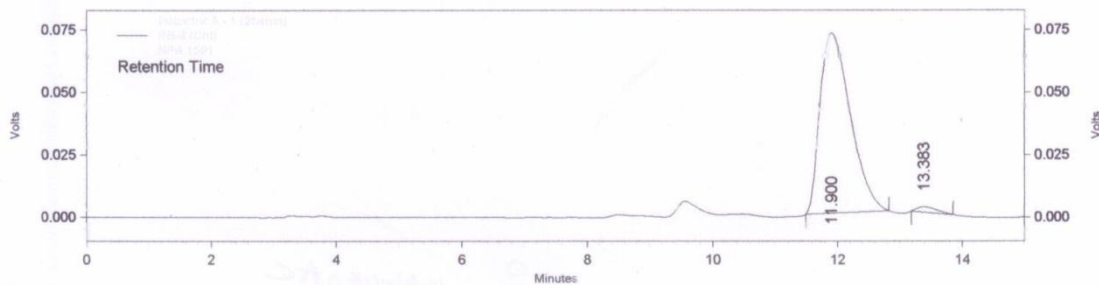
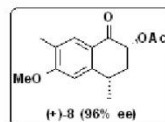
Project Leader :Dr.N. P. Argade
Column Chiralcel OJ-H (250mm x 4.6mm)
Mobile Phase :IPA :Pet Ether (10:90)
Wavelength : 254 nm
Flow Rate :1 ml/min
Conc. : 1mg/1ml
Inj vol- : 5ul

Area % Report

Page 1 of 1

RB-3 (Chi)

Method Name: C:\CLASS-VP\Data\Dr. Patil N. TVSS
Data Name: C:\CLASS-VP\Data\Dr. Argade\NPA 1591
User: System
Acquired: 2/21/15 8:34:32 AM
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Detector A - 1 (254nm)

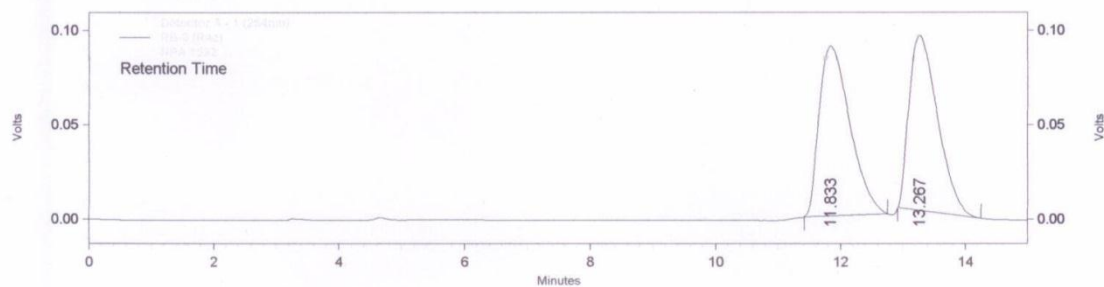
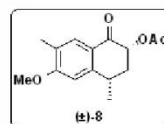
PK #	Retention Time	Area	Area %
1	11.900	2448817	98.144
2	13.383	46312	1.856
Totals		2495129	100.000

Project Leader :Dr.N. P. Argade
Column Chiralcel OJ-H (250mm x 4.6mm)
Mobile Phase :IPA :Pet Ether (10:90)
Wavelength : 254 nm
Flow Rate : 1 ml/min
Conc. : 1mg/ml
Inj vol- : 5ul

Area % Report

Page 1 of 1

RB-3 (Rac)
 Method Name: C:\CLASS-VP\Data\Dr. Patil N. TVSS
 Data Name: C:\CLASS-VP\Data\Dr. Argade\NPA 1592
 User: System
 Acquired: 2/21/15 8:53:50 AM
 Printed: 5/7/15 5:22:45 PM



Detector A - 1 (254nm)

Pk #	Retention Time	Area	Area %
1	11.833	3068477	50.997
2	13.267	2948449	49.003
Totals		6016926	100.000

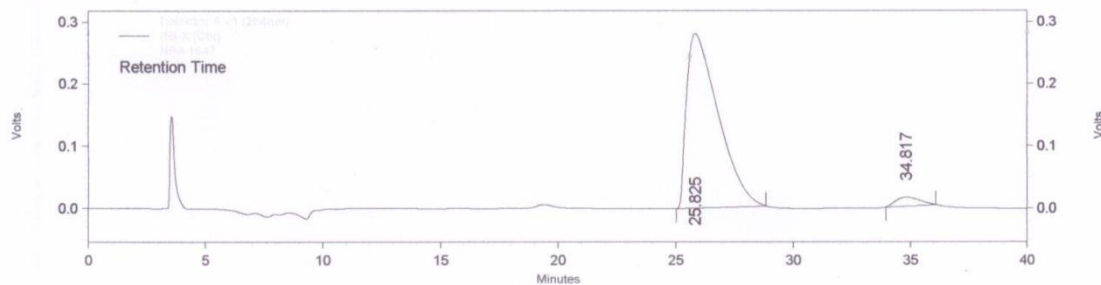
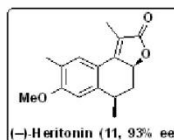
Project Leader :Dr.N. P. Argade
 Column :Chiralcel Oj-H (250mm x 4.6mm)
 Mobile Phase :IPA:PE (10 : 90)
 Wavelength : 254 nm
 Flow Rate :1mL/min (620psi)
 Conc. : 1mg/1ml
 Inj vol- : 2uL

Area % Report

Page 1 of 1

RB-X (Chi)

Method Name: C:\CLASS-VP\Data\Dr. Patil N. TVSS
Data Name: C:\CLASS-VP\Data\Dr. Argade\NPA 1647
User: System
Acquired: 4/27/15 3:59:06 PM
Printed: 4/27/15 5:31:51 PM



Detector A - 1 (254nm)

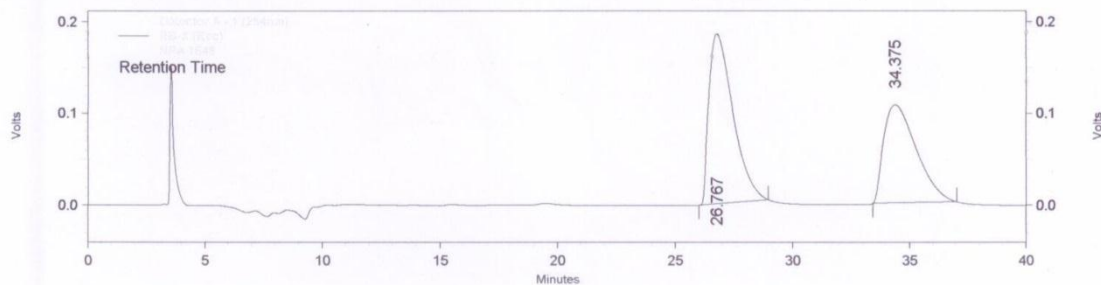
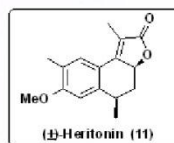
Pk #	Retention Time	Area	Area %
1	25.825	26230038	96.349
2	34.817	994035	3.651
Totals		27224073	100.000

Project Leader :Dr.N. P. Argade
Column :Chiralcel OJ-H (250mm x 4.6mm)
Mobile Phase :IPA:Pet Ether (2 : 98)
Wavelength : 254nm
Flow Rate :1mL/min (620psi)
Conc. : 1mg/0.5ml
Inj vol- : 10uL

Area % Report

Page 1 of 1

RB-X (Rac)
 Method Name: C:\CLASS-VP\Data\Dr. Patil N. TVSS
 Data Name: C:\CLASS-VP\Data\Dr. Argade\NPA 1648
 User: System
 Acquired: 4/27/15 4:40:09 PM
 Printed: 4/27/15 5:28:21 PM



Detector A - 1 (254nm)			
Pk #	Retention Time	Area	Area %
1	26.767	13255397	55.995
2	34.375	10416875	44.005
Totals		23672272	100.000

Project Leader :Dr.N. P. Argade
 Column :Chiralcel OJ-H (250mm x 4.6mm)
 Mobile Phase :IPA:Pet Ether (2 : 98)
 Wavelength : 254nm
 Flow Rate :1mL/min (620psi)
 Conc. : 1mg/0.5ml
 Inj vol- : 10uL

Shimadzu CLASS-VP V6.12 SP5

Method Name: C:\CLASS-VP\Method ch 2.met

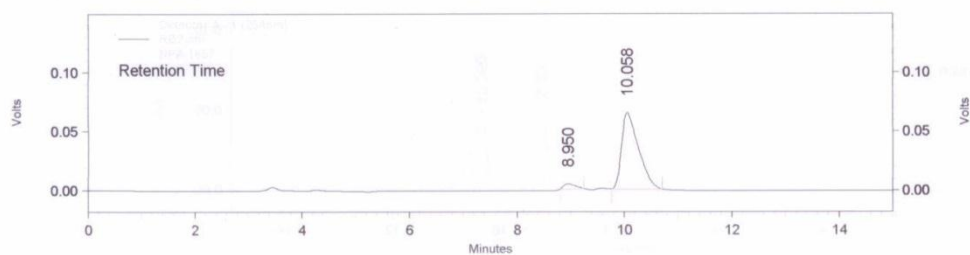
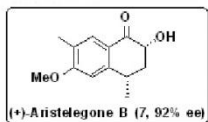
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User: System

Acquired: 7/7/15 12:37:33 PM

Printed: 7/7/15 12:57:02 PM

Sample Name RB2-chi



Detector A - 1 (254nm)			
Retention Time		C Area	Area %
8.950		59376	4.043
10.058		1409276	95.957
Totals		1468652	100.000

Project Leader :Dr.Argade N.P.

Column : Chiralcel OJ-H (0.46cm X 25cm)

Mobile Phase :IPA:Pet ether (10:90)

Flow Rate : 1.0 ml/min 620psi

Wavelength : 254nm

Con. : 1mg /1.0ml

Inject vol. :5uL

Shimadzu CLASS-VP V6.12 SP5

Method Name: C:\CLASS-VP\Method ch 2.met

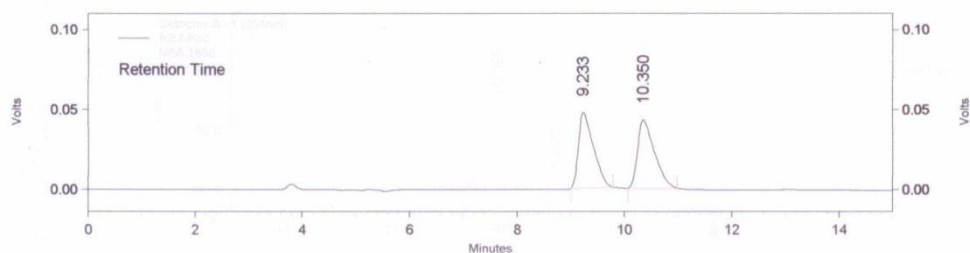
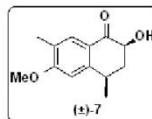
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User: System

Acquired: 7/7/15 12:57:58 PM

Printed: 7/7/15 1:16:29 PM

Sample Name RB2-Rac



Detector A - 1 (254nm)			
Retention Time		C Area	Area %
9.233		913819	49.503
10.350		932150	50.497
Totals		1845969	100.000

Project Leader :Dr.Argade N.P.

Column : Chiralcel OJ-H (0.46cm X 25cm)

Mobile Phase :IPA:Pet ether (10:90)

Flow Rate : 1.0 ml/min 620psi

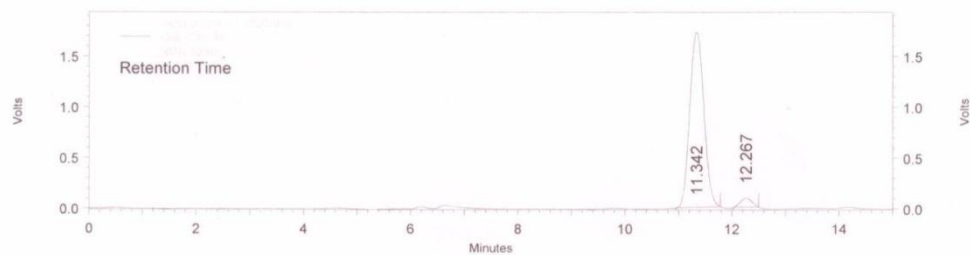
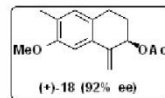
Wavelength : 254nm

Con. : 1mg /1.0ml

Inject vol. :5uL

Shimadzu CLASS-VP V6.12 SP5

Method Name: C:\CLASS-VP\Method ch 2.met
 Data Name: C:\CLASS-VP\Data\Dr. Argade\NPA 1250
 User: System
 Acquired: 6/18/10 4:01:05 PM
 Printed: 6/18/10 4:20:16 PM
 Sample Name Oac-Chi-Ar



Detector A - 1 (220nm)

Pk #	Retention Time	Area	Area %
1	11.342	31662915	96.009
2	12.267	1316335	3.991
Totals		32979250	100.000

Grp Leader : Dr.Argade NP
 Column : Kromasil 5-AmyCoat (250x4.6mm)
 Mobile Phase : IPA:PE (02:98)
 Wavelength : 220nm
 Flow Rate : 0.5ml/min 25kgf
 Sample Con : 1mg/3 ml Inj vol-5ul

Shimadzu CLASS-VP V6.12 SP5

Method Name: C:\CLASS-VP\Method ch 2.met

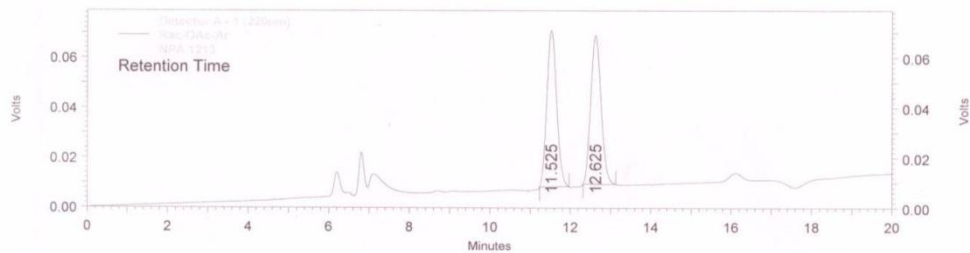
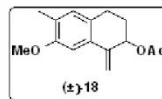
Data Name: C:\CLASS-VP\Data\Dr. Argade\NPA 1213

User: System

Acquired: 5/17/10 12:34:00 PM

Printed: 5/17/10 4:23:46 PM

Sample Name Rac-OAc-Ar



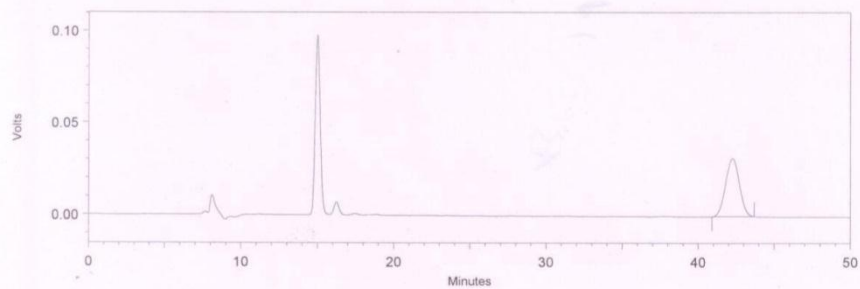
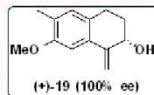
Detector A - 1 (220nm)

PK #	Retention Time	Area	Area %
1	11.525	1067485	49.973
2	12.625	1068618	50.027
Totals		2136103	100.000

Grp Leader : Dr.N.P.Argade
Column : Kromasil 5-AmyCoat
Mobile Phase : IPA:PE(2:98)
Wavelength : 220nm
Flow Rate : 0.5ml/min 23kgf
Sample Con : 1mg/1 ml Inj vol-5ul

Area % Report

Sample Name: RB-I Chiral 1 d
 Method Name: C:\CLASS-VP\method ch 1.met
 Data Name: C:\CLASS-VP\Data\Dr. Argade\NPA 1310
 User: System
 Acquired: 3/21/11 12:02:43 PM
 Printed: 3/21/11 1:00:39 PM

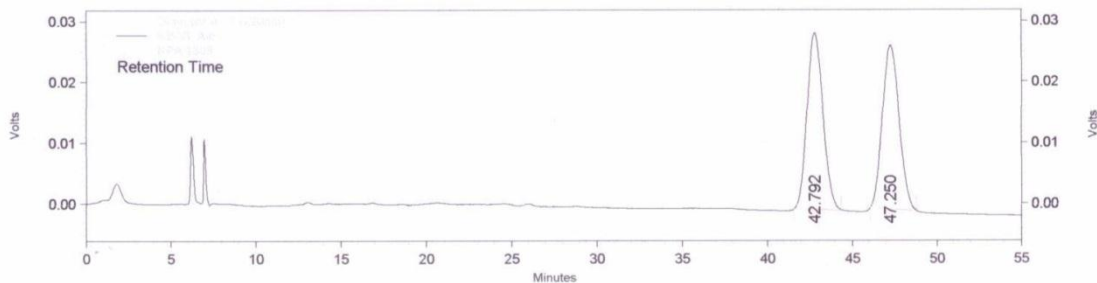
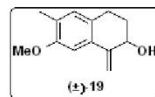


Detector A - 1 (220nm)

Pk #	Retention Time	Area	Area %
1	42.267	2003945	100.000
Totals		2003945	100.000

Project Leader :- Dr. N. P. Argade
 Column :- Kromasil 5-AmyCoat(250x4.6 mm)
 M.Phase :- IPA:PE (02:98)
 Wavelength :- 220 nm
 Flow :- 0.5ml/min (360psi)
 conc. :- 1 mg/ 1 ml IPA
 Injection vol :- 05 ul

Method Name: C:\CLASS-VP\Methods\Date6june.met
Data Name: C:\CLASS-VP\Data\Dr. Argade\NPA 1309
User: System
Acquired: 3/2/11 2:39:12 PM
Printed: 7/28/15 4:28:12 PM



Detector A - 1 (220nm)

Pk #	Retention Time	Area	Area %
1	42.792	1881105	50.655
2	47.250	1832428	49.345
Totals		3713533	100.000

Project Leader :Dr. N. P. Argade

Column : Kromasil 5-AmyCoat (0.46cm X 25cm)

Mobile Phase :EtOH:PE:DEA (5:95:0.1)

Flow Rate : 0.5 ml/min 360psi

Wavelength : 220nm

Con. : 1mg /1.0ml

Inject vol. :5uL