

SUPPORTING INFORMATION (Part 2/2)

Facile and selective procedure for the synthesis of bridged 1,2,4,5-tetraoxanes. Strong acids as cosolvents and catalysts for the addition of hydrogen peroxide to β -diketones.

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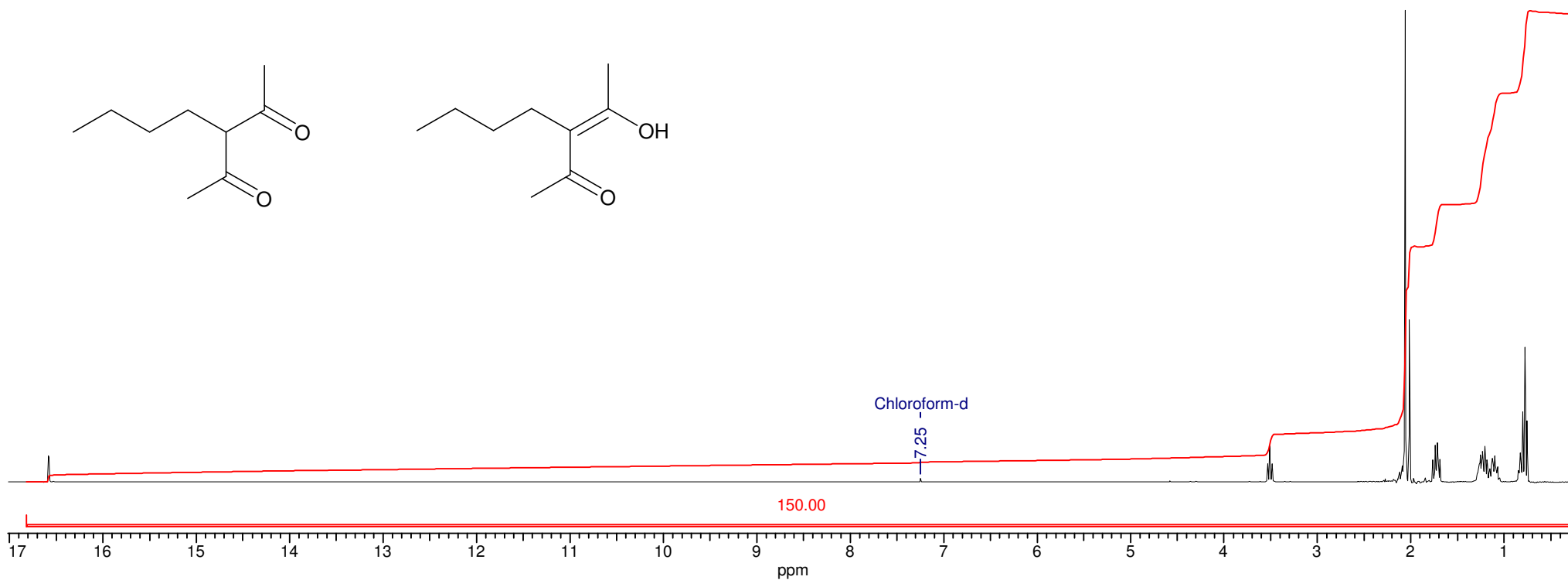
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3-Butylpentane-2,4-dione (1a)

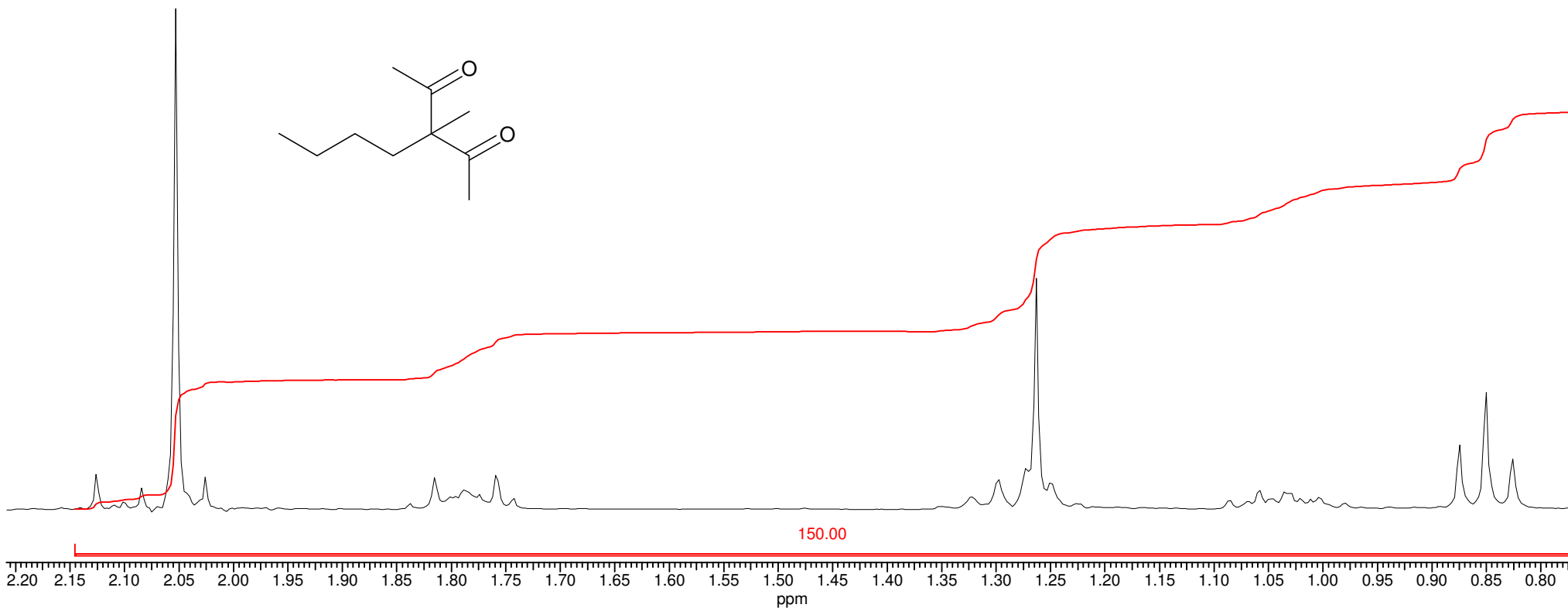
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3-Butyl-3-methylpentane-2,4-dione (1b)

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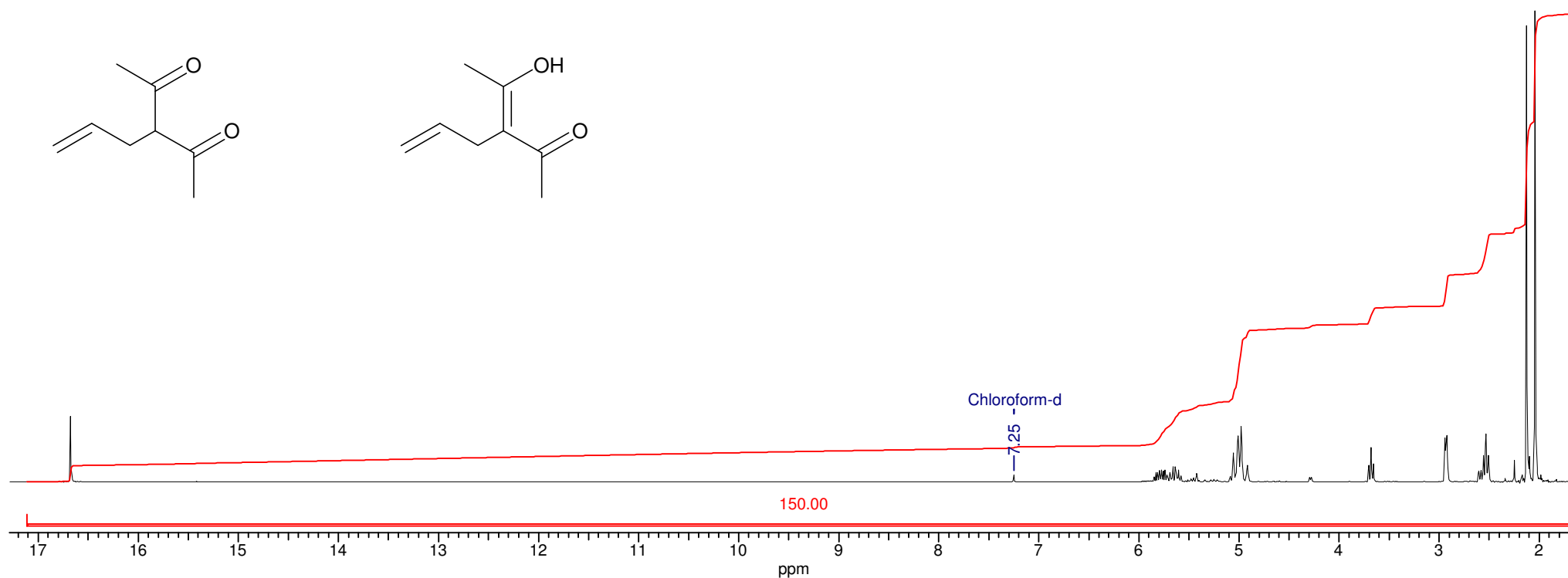
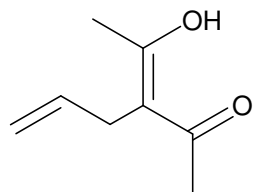
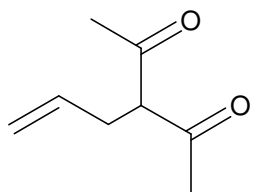
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No.	Annotation	(ppm)
1	Chloroform-d	7.25

No.	(ppm)	Value
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3-Allylpentane-2,4-dione (1c)

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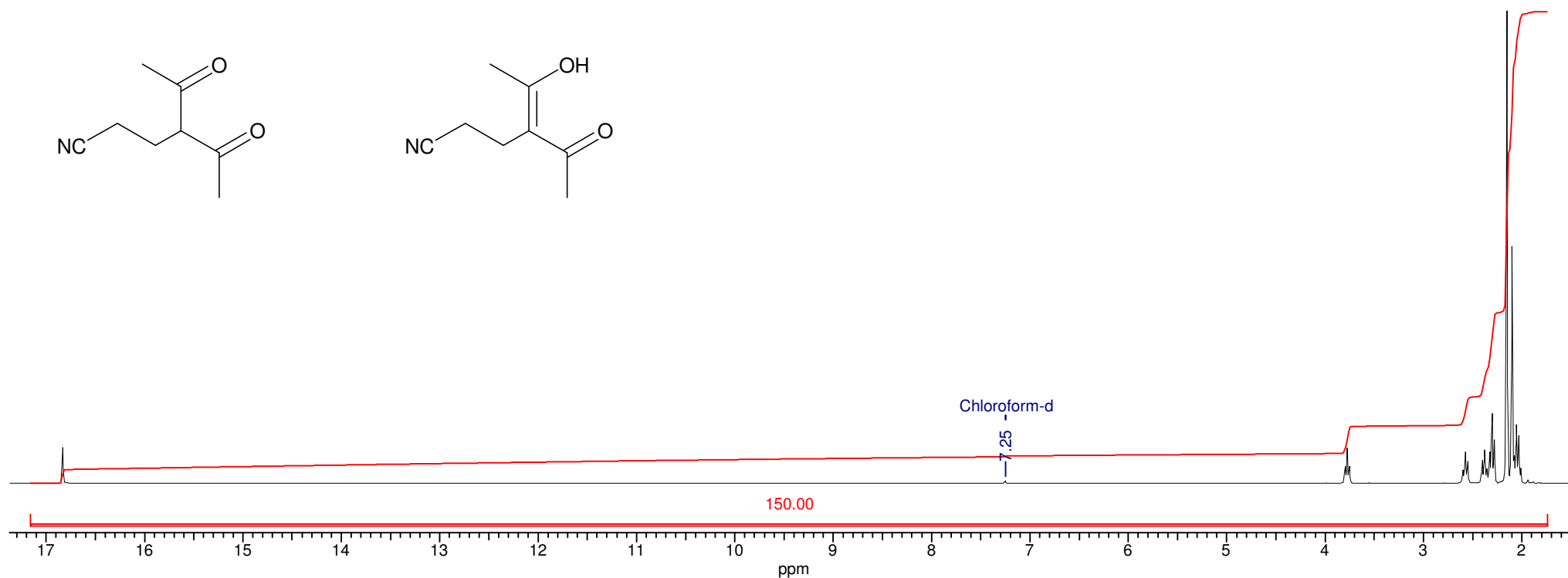
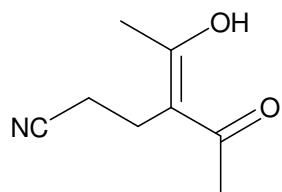
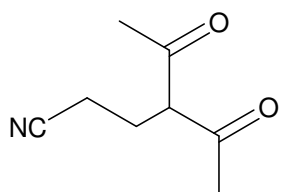
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No.	Annotation	(ppm)
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No.	(ppm)	Value
1	60 .. 17.1	150.000

4-Acetyl-5-oxohexanenitrile (1d)

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File Name					
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	1
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Temperature (degree C)	22.800			Sweep Width (Hz)	6009.62



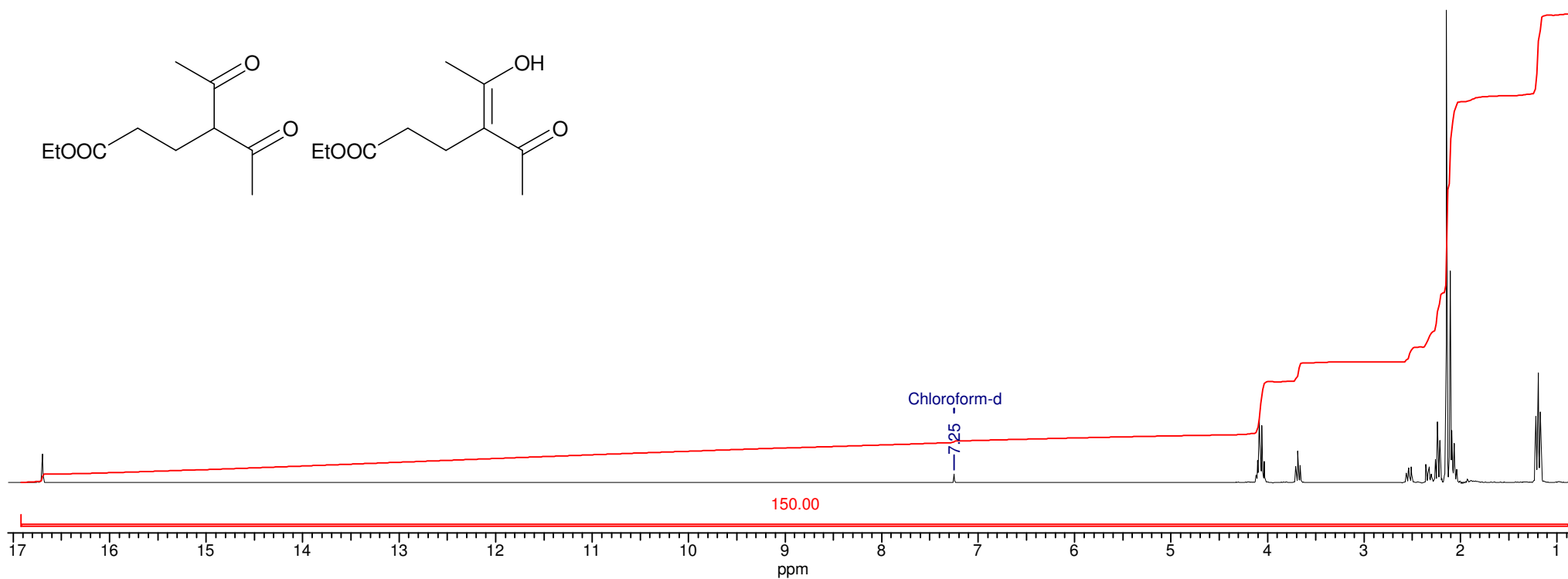
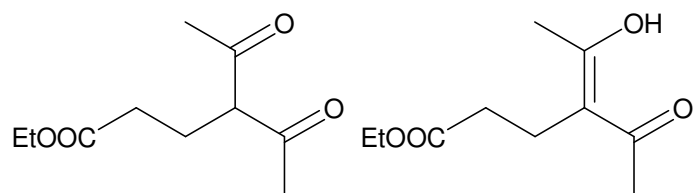
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No.	Annotation	(ppm)
1	Chloroform-d	7.25

No.	(ppm)	Value
1	74 .. 17.1	150.000

Ethyl 4-acetyl-5-oxohexanoate (1e)

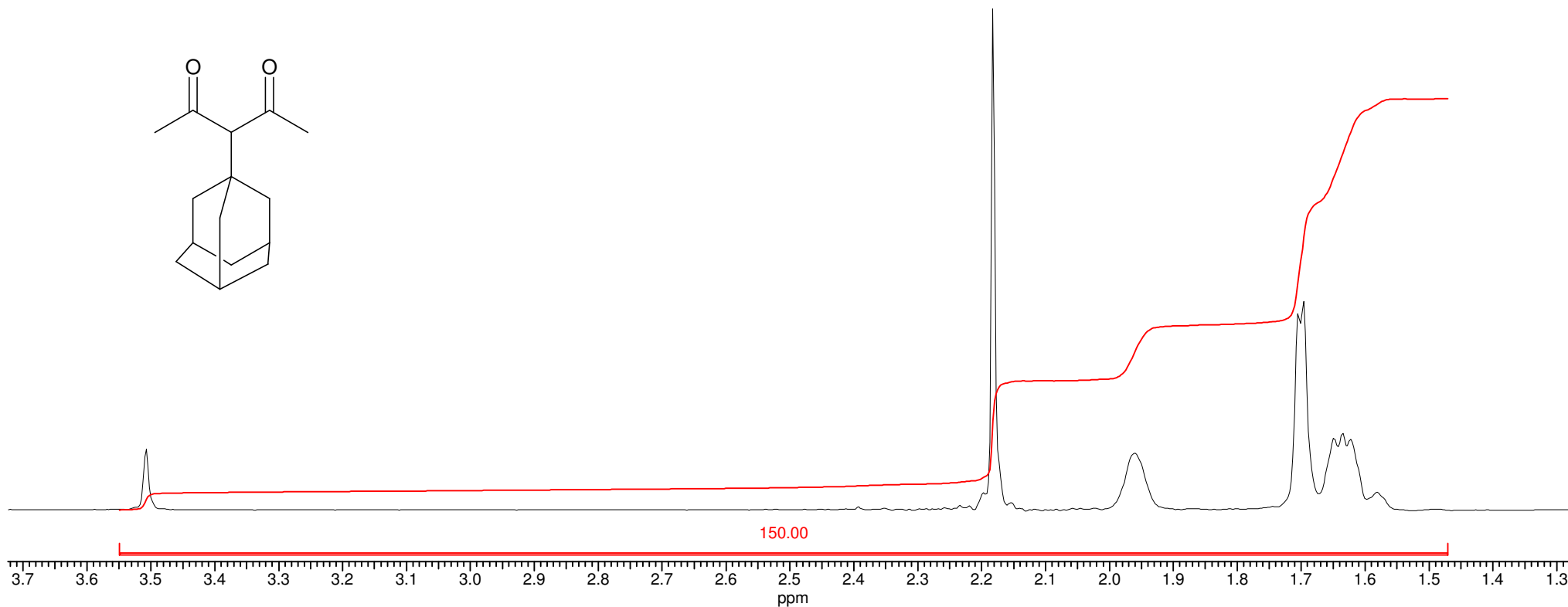
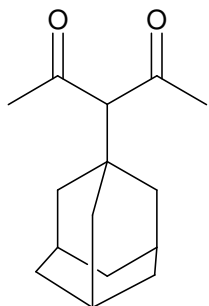
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Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	24.000			Sweep Width (Hz)	6009.62



No.	(ppm)	(Hz)	Height	No.	Annotation	(ppm)	No.	(ppm)	Value
1	7.25	2175.9	0.0178	1	Chloroform-d	7.25	1	88 .. 16.9	150.000

3-(1-Adamantyl)pentane-2,4-dione (1f)

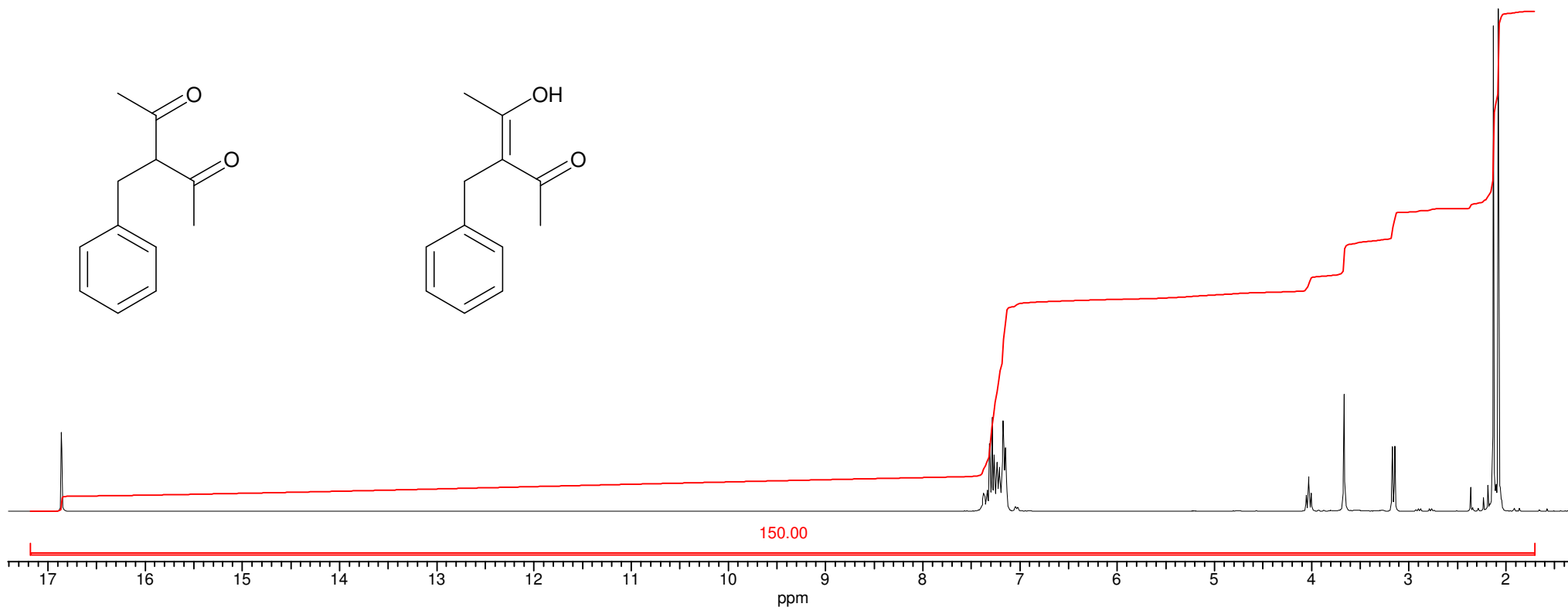
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Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	24.000			Sweep Width (Hz)	6009.62



No.	(ppm)	(Hz)	Height	No.	Annotation	(ppm)	No.	(ppm)	Value
1	7.25	2175.9	0.0284	1	Chloroform-d	7.25	1	.47 .. 3.5	150.000

3-Benzylpentane-2,4-dione (1g)

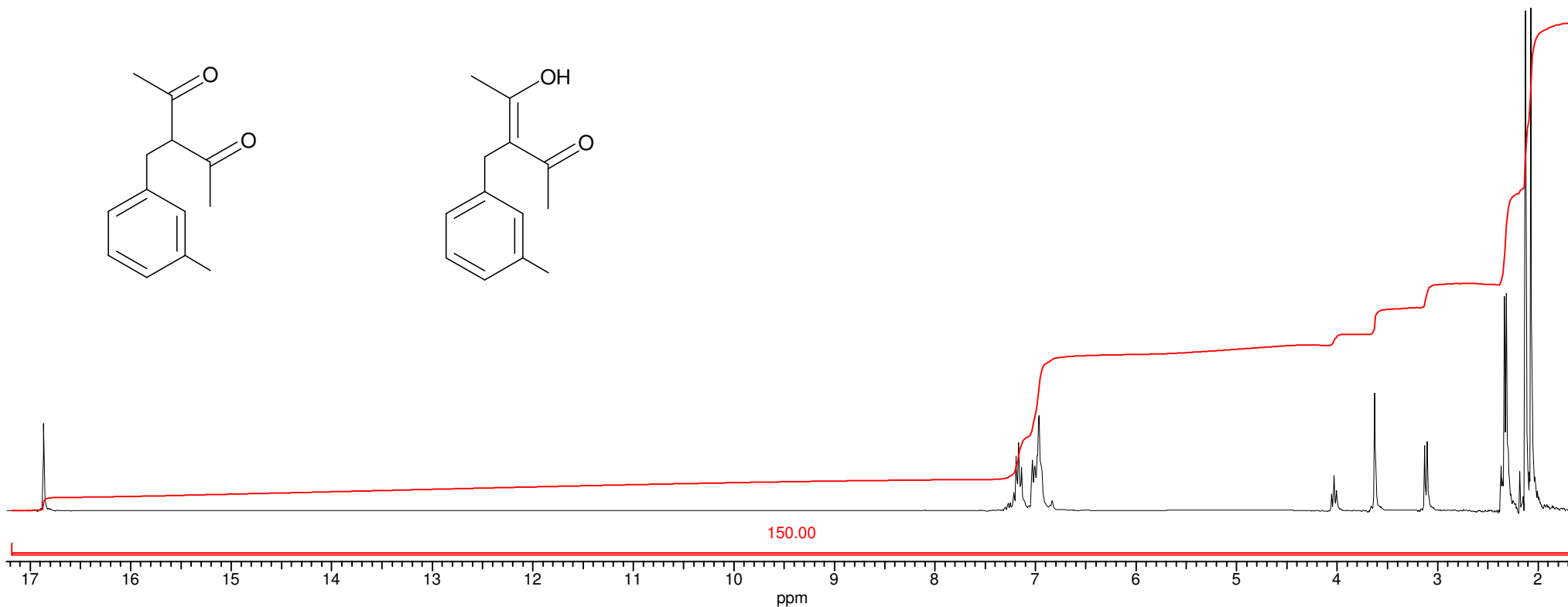
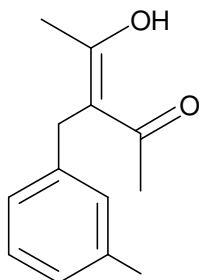
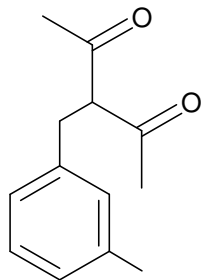
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Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	19.000			Sweep Width (Hz)	6009.62



No.	(ppm)	Value
1	70 .. 17.1	150.000

3-(3-Methylbenzyl)pentane-2,4-dione (1h)

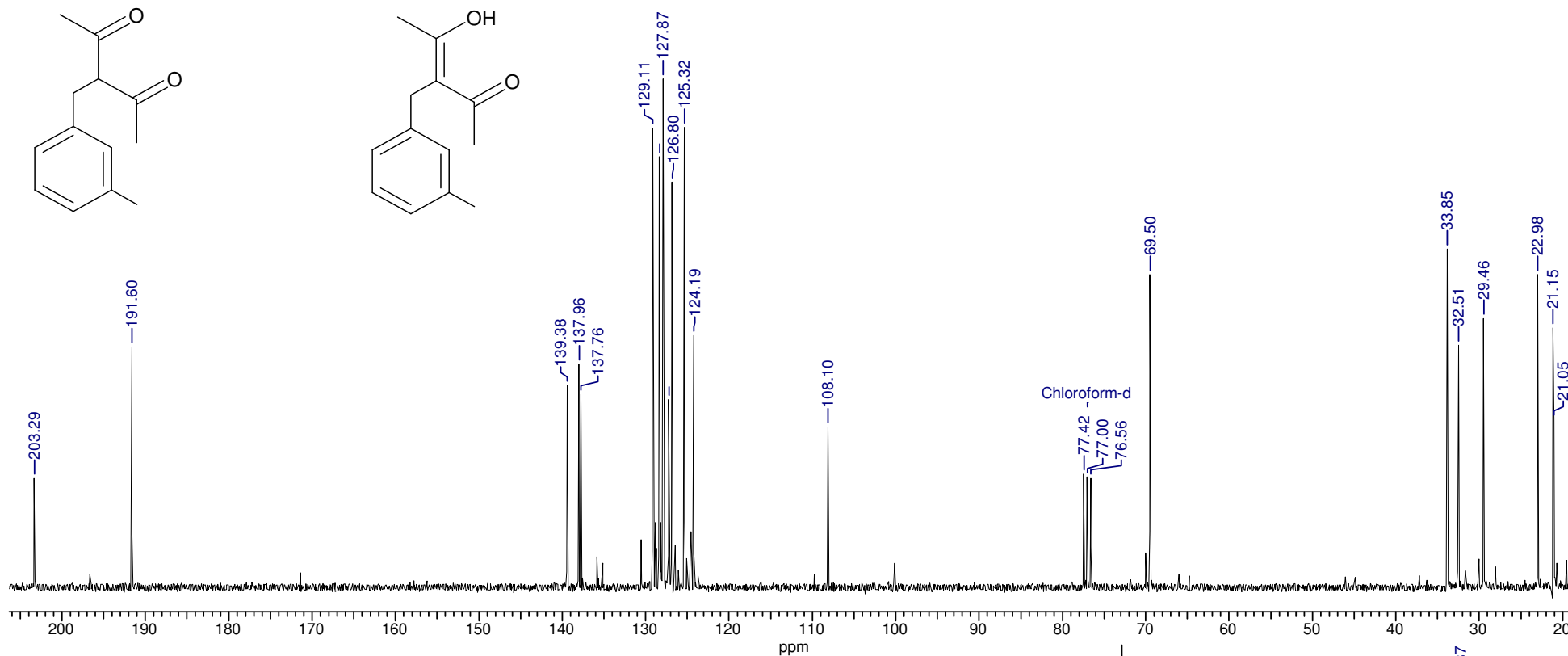
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Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	24.100			Sweep Width (Hz)	6009.62



No.	(ppm)	Value
1	68 .. 17.1	150.000

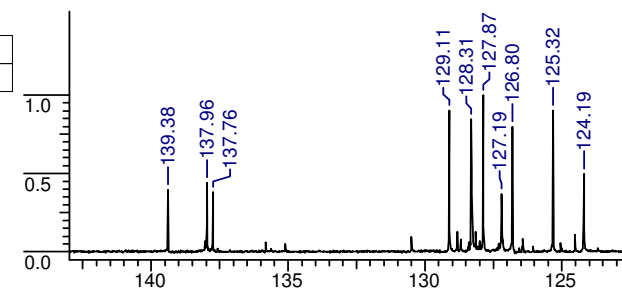
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Temperature (degree C)	24.200						



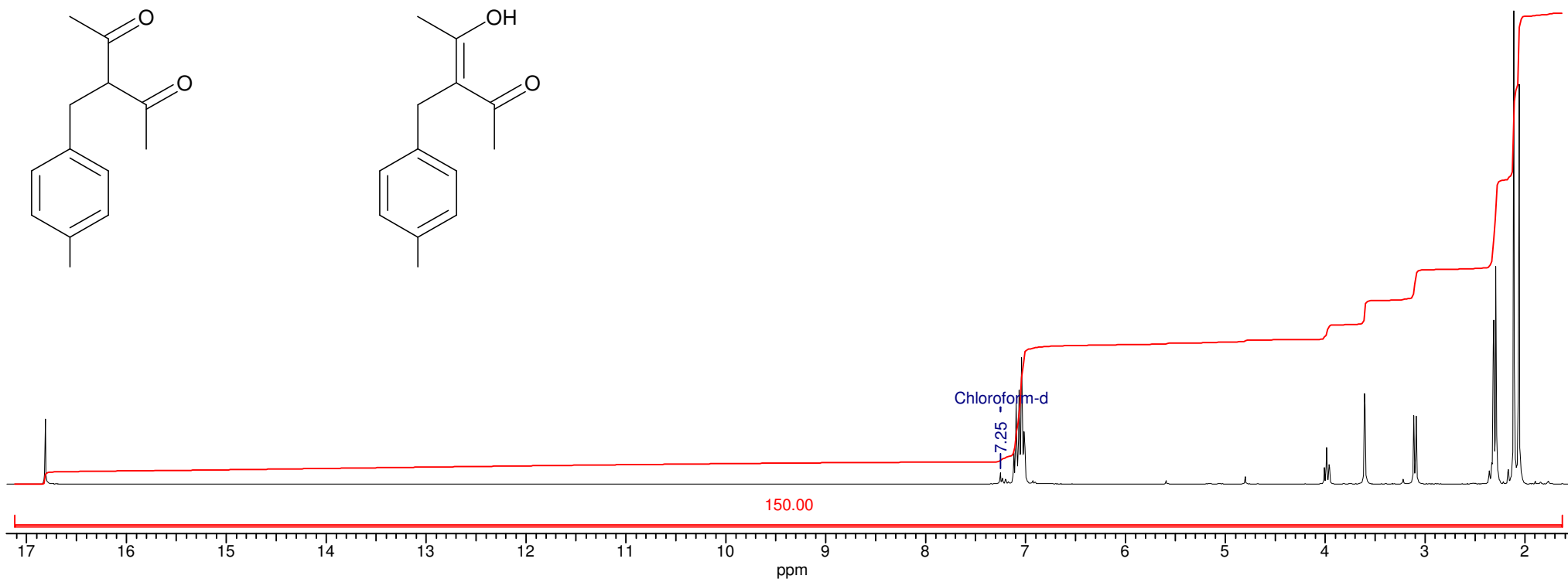
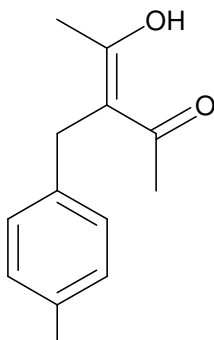
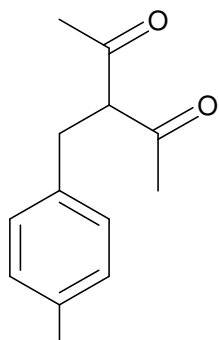
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2	21.15	1596.4	0.5105	10	77.42	5843.7	0.2228	18	129.11	9744.9	0.9034
3	22.98	1734.6	0.6151	11	108.10	8159.2	0.3158	19	137.76	10397.3	0.3798
4	29.46	2223.4	0.5286	12	124.19	9373.3	0.4958	20	137.96	10412.8	0.4391
5	32.51	2453.4	0.4764	13	125.32	9458.5	0.9043	21	139.38	10520.0	0.3974
6	33.85	2555.1	0.6651	14	126.80	9570.2	0.7964	22	191.60	14461.0	0.4734
7	69.50	5245.5	0.6151	15	127.19	9600.0	0.3697	23	203.29	15343.4	0.2144
8	76.56	5778.4	0.2151	16	127.87	9650.9	1.0000				

No.	Annotation	(ppm)
1	Chloroform-d	77.00



3-(4-Methylbenzyl)pentane-2,4-dione (1i)

Acquisition Time (sec)	0.6759	Comment	Avance-300, CDCl ₃	Date	07 Feb 2008 11:12:00
File Name					
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Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	20.400			Sweep Width (Hz)	6009.62



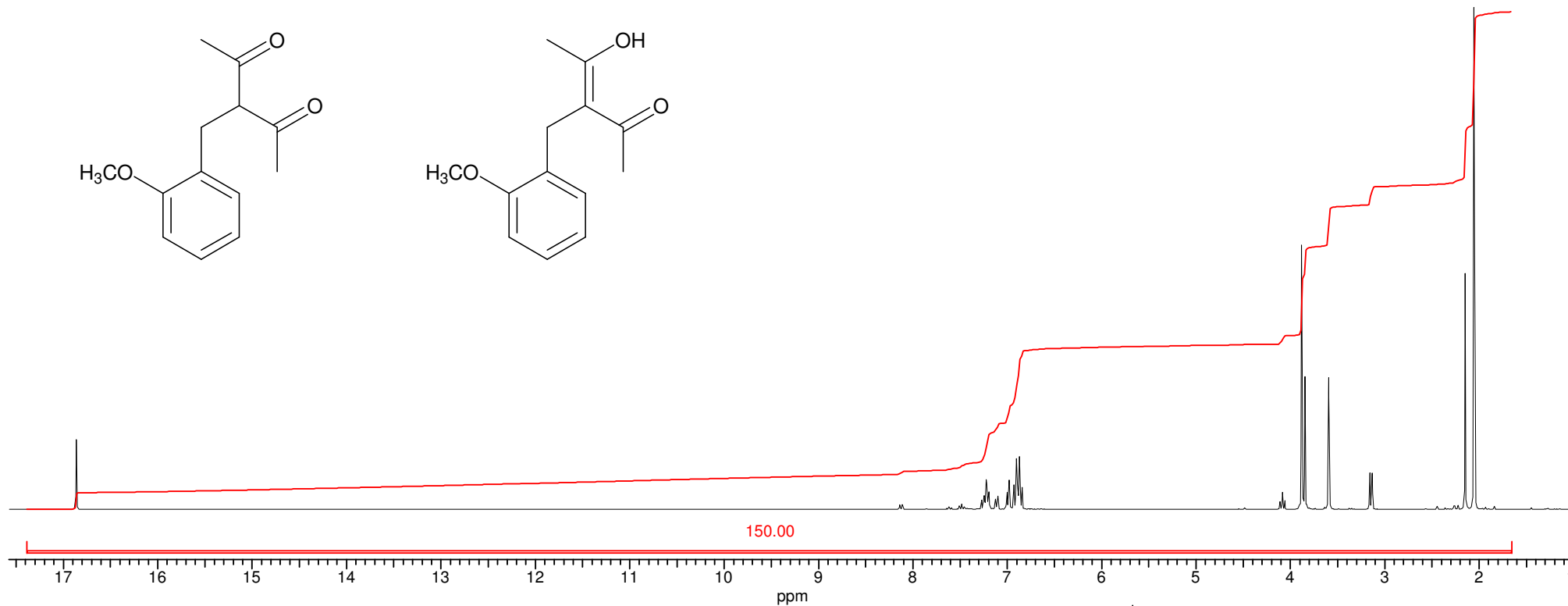
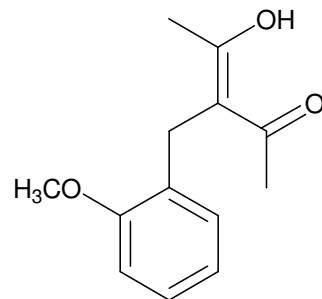
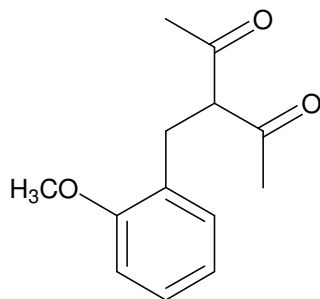
No.	(ppm)	(Hz)	Height
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No.	Annotation	(ppm)
1	Chloroform-d	7.25

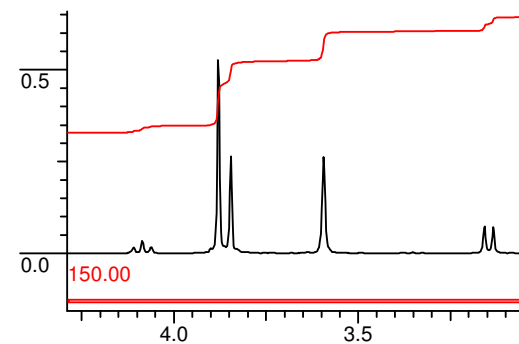
No.	(ppm)	Value
1	63 .. 17.1	150.000

3-(2-Methoxybenzyl)pentane-2,4-dione (1j)

Acquisition Time (sec)	0.6759	Comment	Avance-300, CDCl3	Date	26 Dec 2007 12:20:16
File Name					
Frequency (MHz)	300.13	Nucleus	¹ H	Number of Transients	1
Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	23.600			Original Points Count	8124
				Sweep Width (Hz)	6009.62

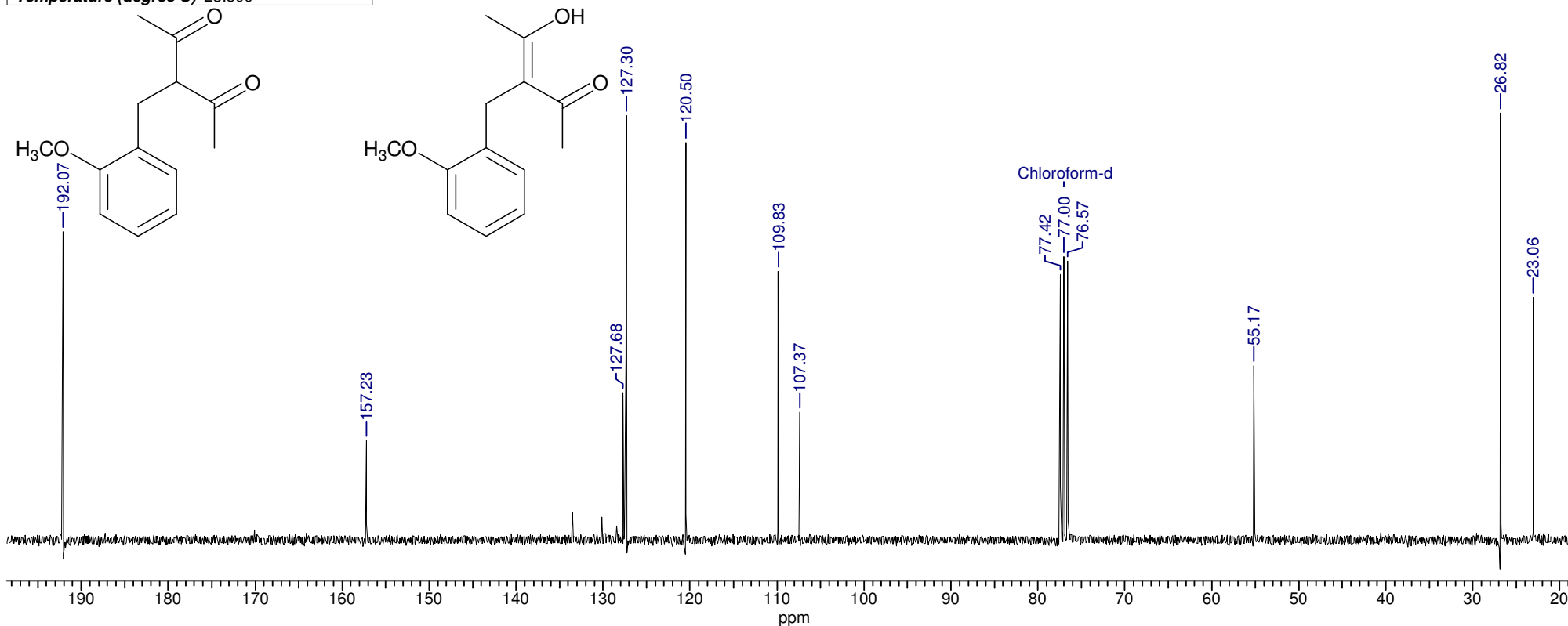


No.	(ppm)	Value
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3-(2-Methoxybenzyl)pentane-2,4-dione (1j)

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Temperature (degree C)	23.500						



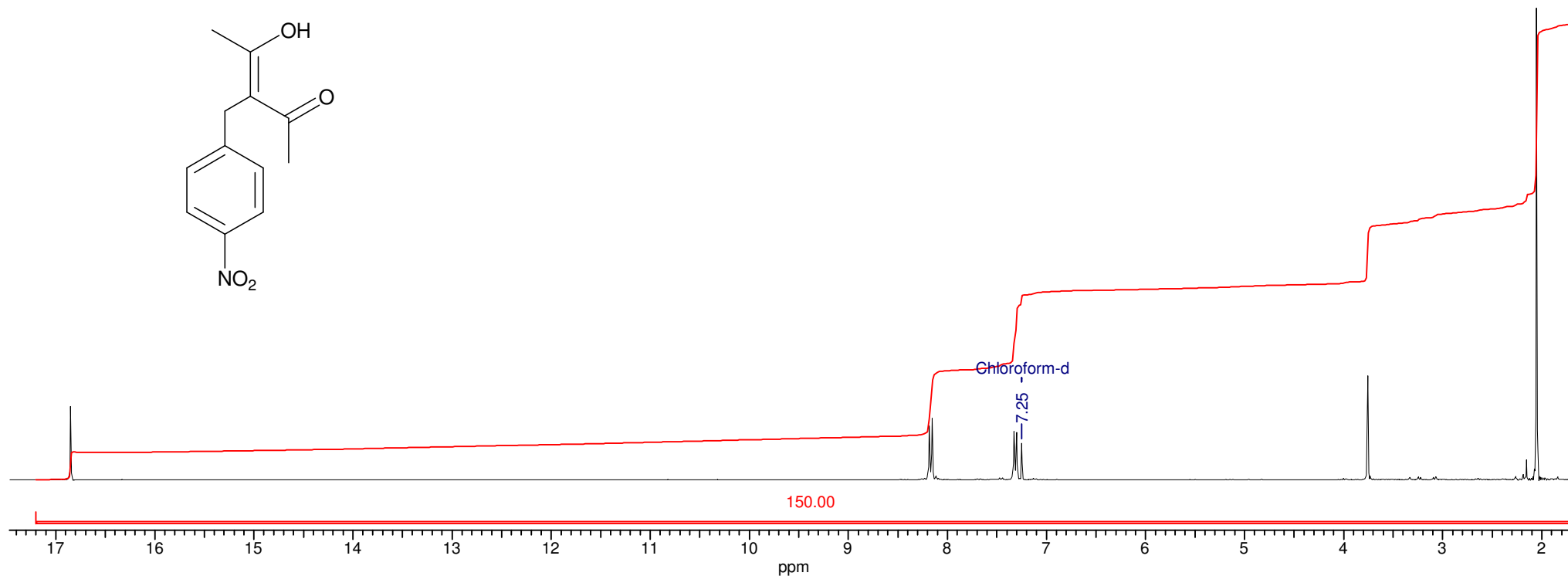
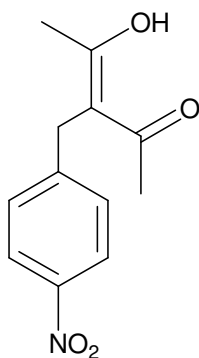
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3	55.17	4164.0	0.4091
4	76.57	5779.6	0.6530
5	77.00	5811.6	0.6640
6	77.42	5843.7	0.6223
7	107.37	8103.9	0.2994

No.	(ppm)	(Hz)	Height
8	109.83	8289.7	0.6298
9	120.50	9094.7	0.9318
10	127.30	9607.8	0.9947
11	127.68	9636.5	0.3455
12	157.23	11866.9	0.2333
13	192.07	14496.4	0.7225

No.	Annotation	(ppm)
1	Chloroform-d	77.00

3-(4-Nitrobenzyl)pentane-2,4-dione (1k)

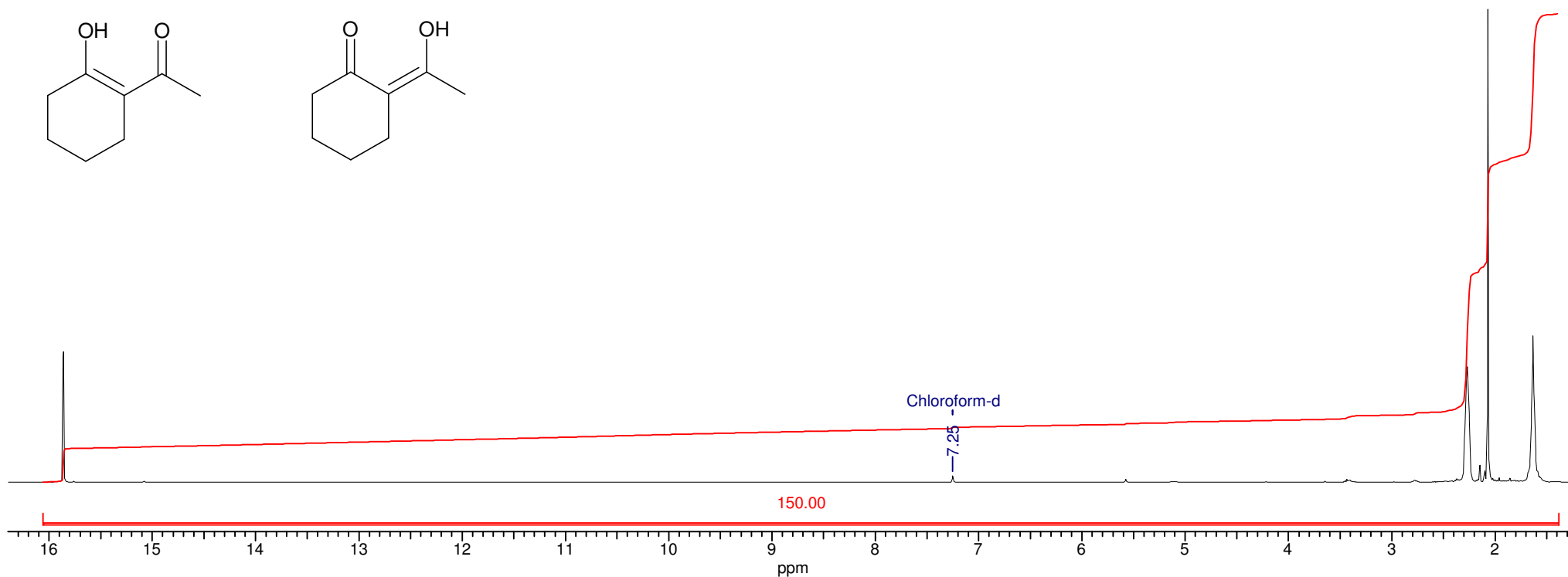
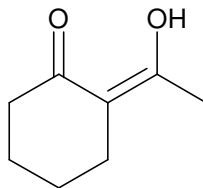
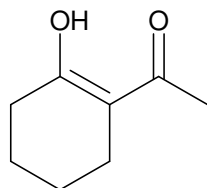
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Temperature (degree C)	21.600			Original Points Count	8124
				Sweep Width (Hz)	6009.62



No.	(ppm)	(Hz)	Height	No.	Annotation	(ppm)	No.	(ppm)	Value
1	7.25	2175.9	0.0774	1	Chloroform-d	7.25	1	57 .. 17.2	150.000

2-Acetylcyclohexanone (1l)

Acquisition Time (sec)	0.6759	Comment	Avance-300, CDCl3	Date	25 Dec 2007 11:50:24
File Name					
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	1
Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	22.700			Sweep Width (Hz)	6009.62



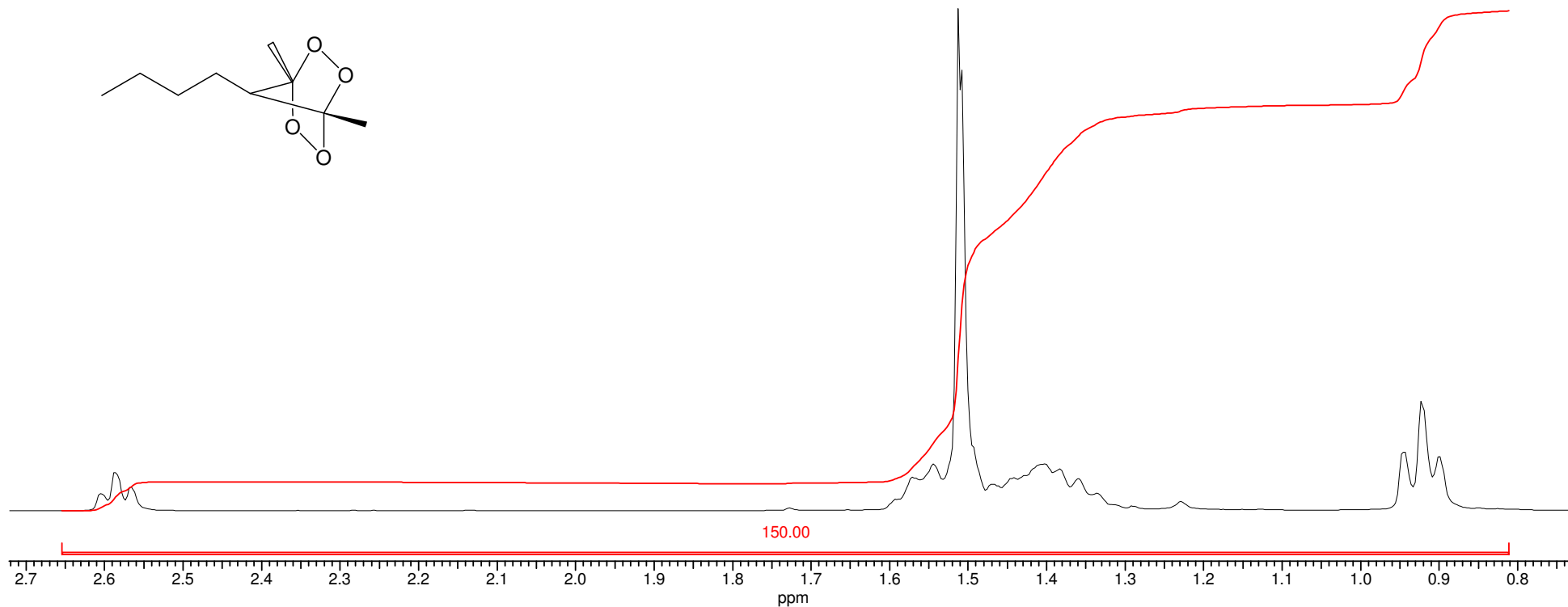
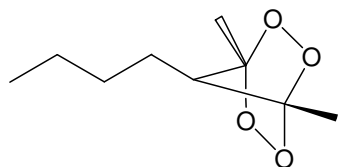
No.	(ppm)	(Hz)	Height
1	7.25	2175.9	0.0137

No.	Annotation	(ppm)
1	Chloroform-d	7.25

No.	(ppm)	Value
1	38 .. 16.0	150.000

7-Butyl-1,4-dimethyl-2,3,5,6-tetraoxa-bicyclo[2.2.1]heptanes (2a).

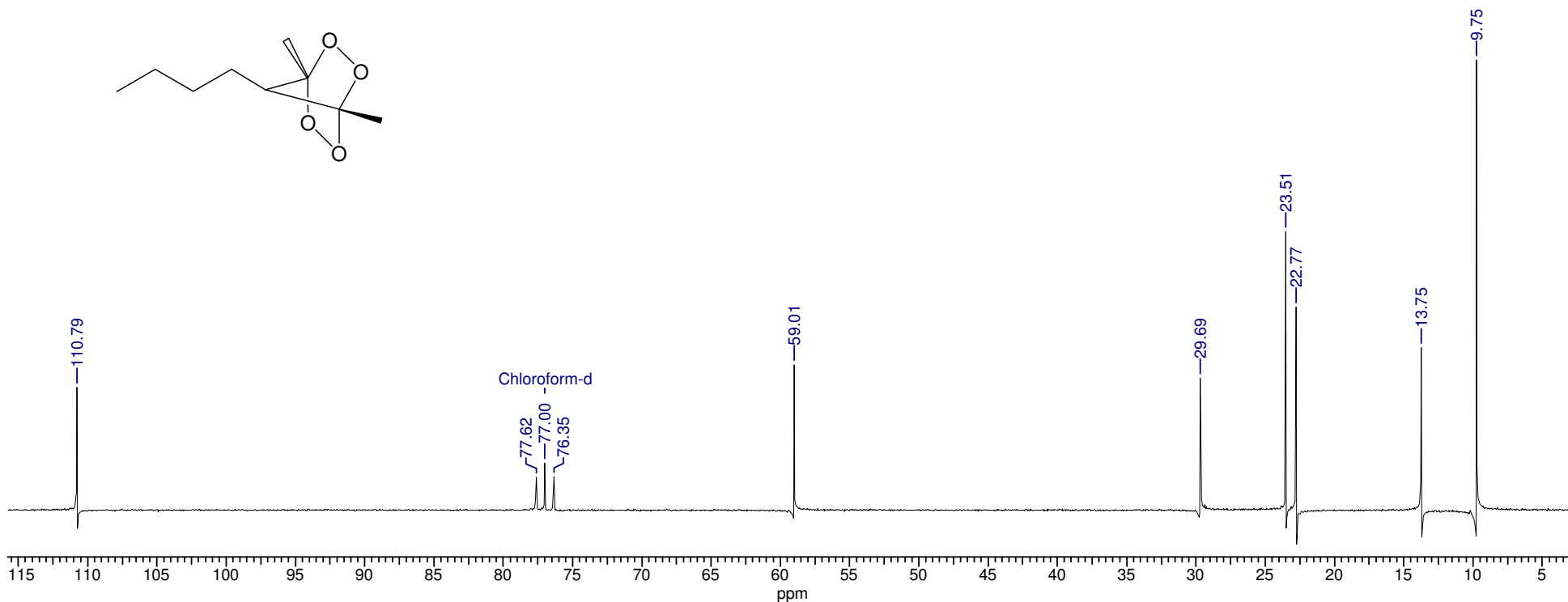
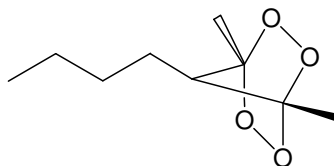
Acquisition Time (sec)	0.6759	Comment	Avance-300, CDCl3	Date	02 Nov 2007 14:19:44
File Name					
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	1
Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	37.000			Sweep Width (Hz)	6009.62



No.	Annotation	(ppm)	No.	(ppm)	Value
1	Chloroform-d	7.25	1	0.81 .. 2.6	150.000

7-Butyl-1,4-dimethyl-2,3,5,6-tetraoxa-bicyclo[2.2.1]heptanes (2a).

Acquisition Time (sec) 0.3359		Comment /TERN IODID524.C13□Opr:Struchkova M.I.;Solv:CDCl3;		Date	03/11/2007 16:50:24
File Name				Frequency (MHz)	50.32
Nucleus	13C	Original Points Count 8192	Points Count 8192	Solvent	CHLOROFORM-D
Sweep Width (Hz)	12195.12	Temperature (degree C) 24.000			

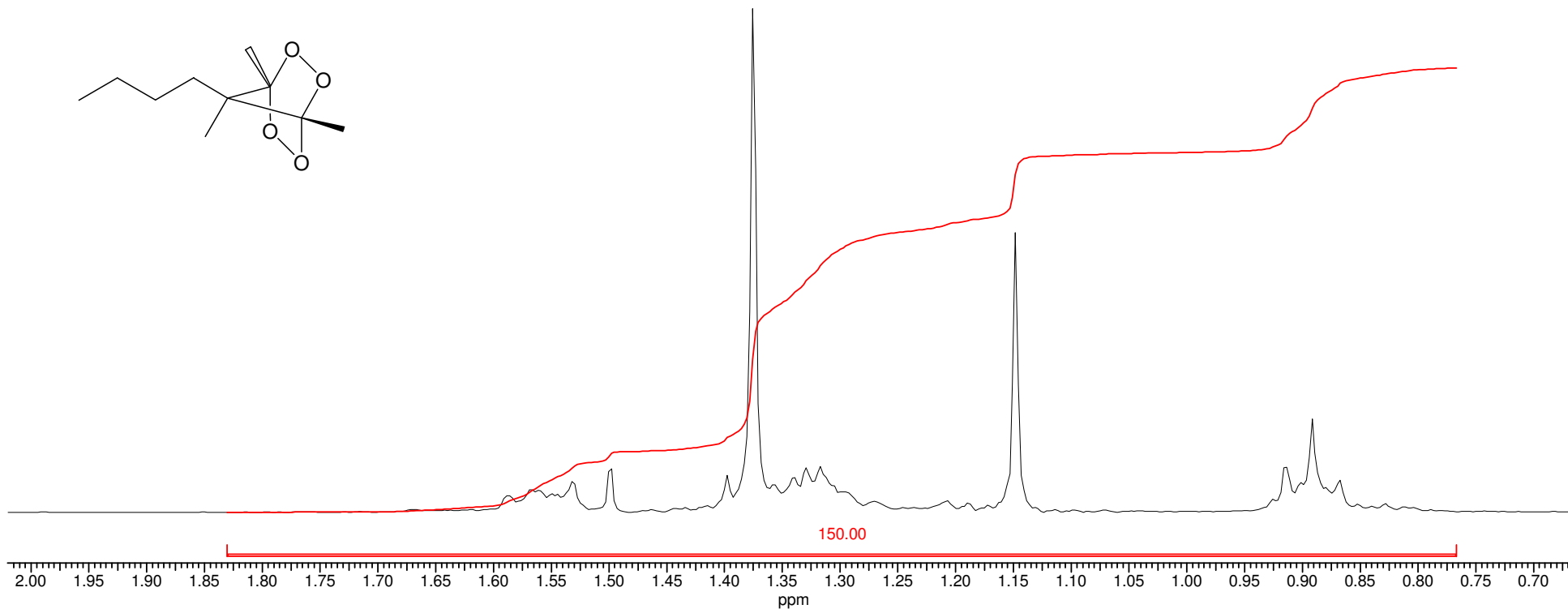
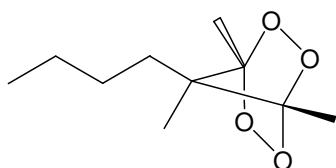


No.	(ppm)	(Hz)	Height
1	9.75	490.8	1.0000
2	13.75	691.8	0.3605
3	22.77	1145.9	0.4510
4	23.51	1183.1	0.6186
5	29.69	1494.3	0.2931
6	59.01	2969.7	0.3228
7	76.35	3842.2	0.0735
8	77.00	3874.9	0.1037
9	77.62	3906.2	0.0724
10	110.79	5575.2	0.2719

No.	Annotation	(ppm)
1	Chloroform-d	77.00

7-Butyl-1,4,7-trimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptanes (2b)

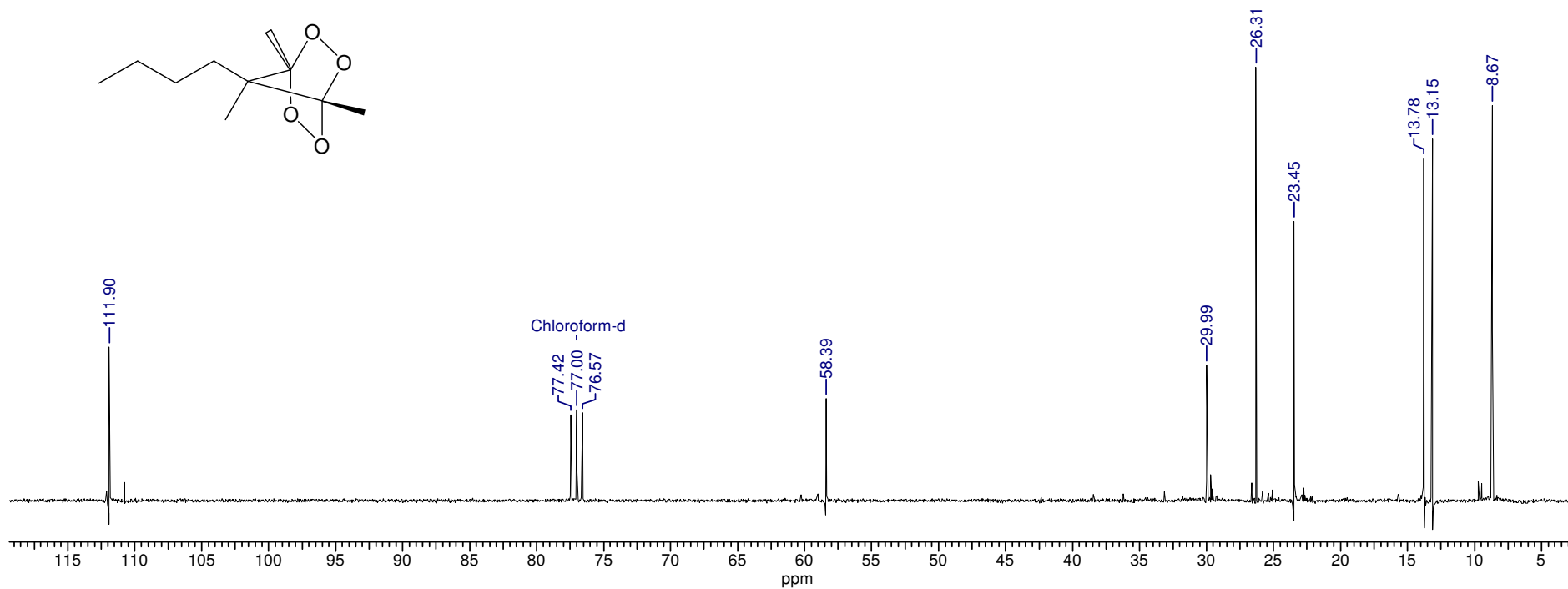
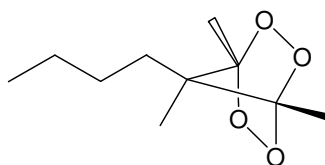
Acquisition Time (sec)	0.6759	Comment	Avance-300, CDCl ₃	Date	10 Jul 2008 10:20:48
File Name					
Frequency (MHz)	300.13	Nucleus	¹ H	Number of Transients	1
Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	24.100			Sweep Width (Hz)	6009.62



No.	(ppm)	(Hz)	Height	No.	Annotation	(ppm)	No.	(ppm)	Value
1	7.25	2175.9	0.0091	1	Chloroform-d	7.25	1	1.77 .. 1.8	150.000

7-Butyl-1,4,7-trimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2b)

Acquisition Time (sec)	0.4501	Comment	Avance-300, C-13, CDCl3			Date	10 Jul 2008 10:22:56
File Name							
Frequency (MHz)	75.48	Nucleus	13C	Number of Transients	916	Original Points Count	16308
Points Count	16384	Pulse Sequence	zgpg30base	Solvent	CHLOROFORM-D	Sweep Width (Hz)	18115.94
Temperature (degree C)	24.200						

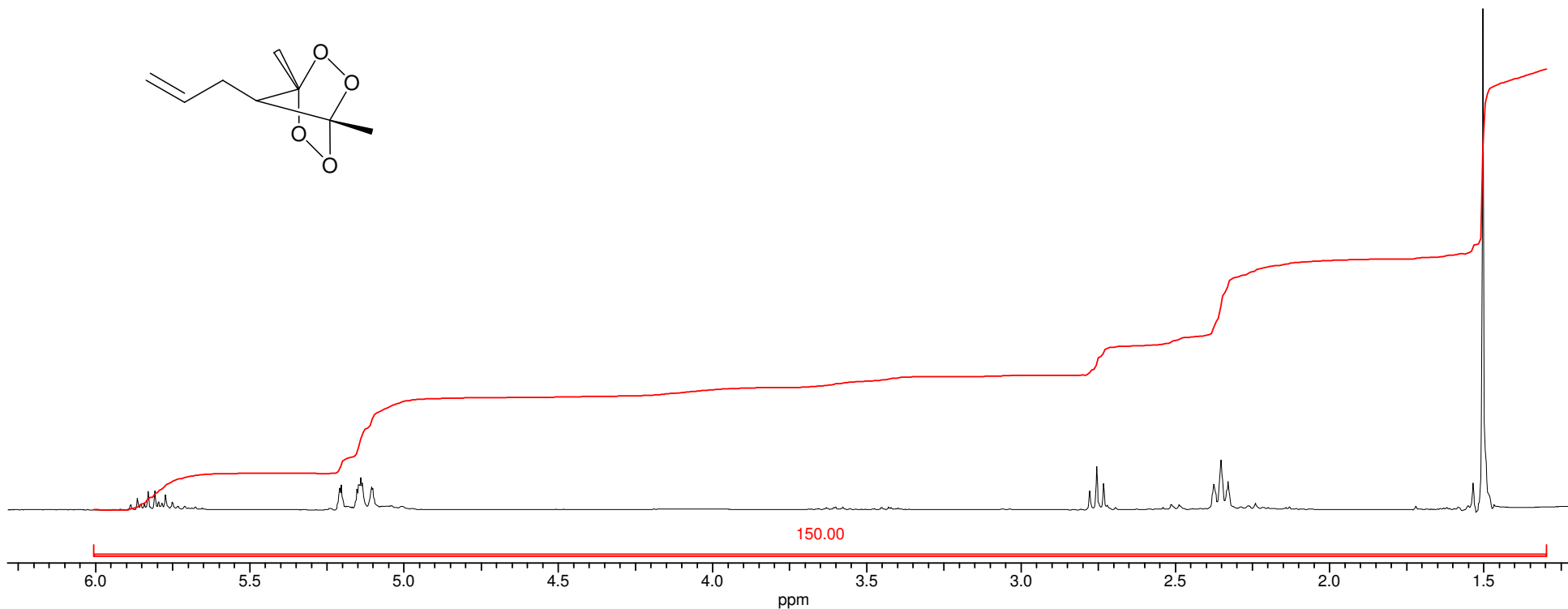
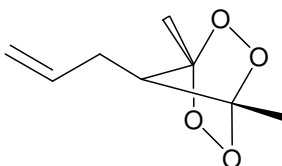


No.	(ppm)	(Hz)	Height
1	8.67	654.3	0.9115
2	13.15	992.7	0.8338
3	13.78	1040.2	0.7901
4	23.45	1770.0	0.6445
5	26.31	1985.6	1.0000
6	29.99	2263.2	0.3121
7	58.39	4407.3	0.2359
8	76.57	5779.6	0.2027
9	77.00	5811.6	0.2095
10	77.42	5843.7	0.1979
11	111.90	8445.6	0.3547

No.	Annotation	(ppm)
1	Chloroform-d	77.00

7-Allyl-1,4-dimethyl-2,3,5,6-tetraoxa-bicyclo[2.2.1]heptane (2c)

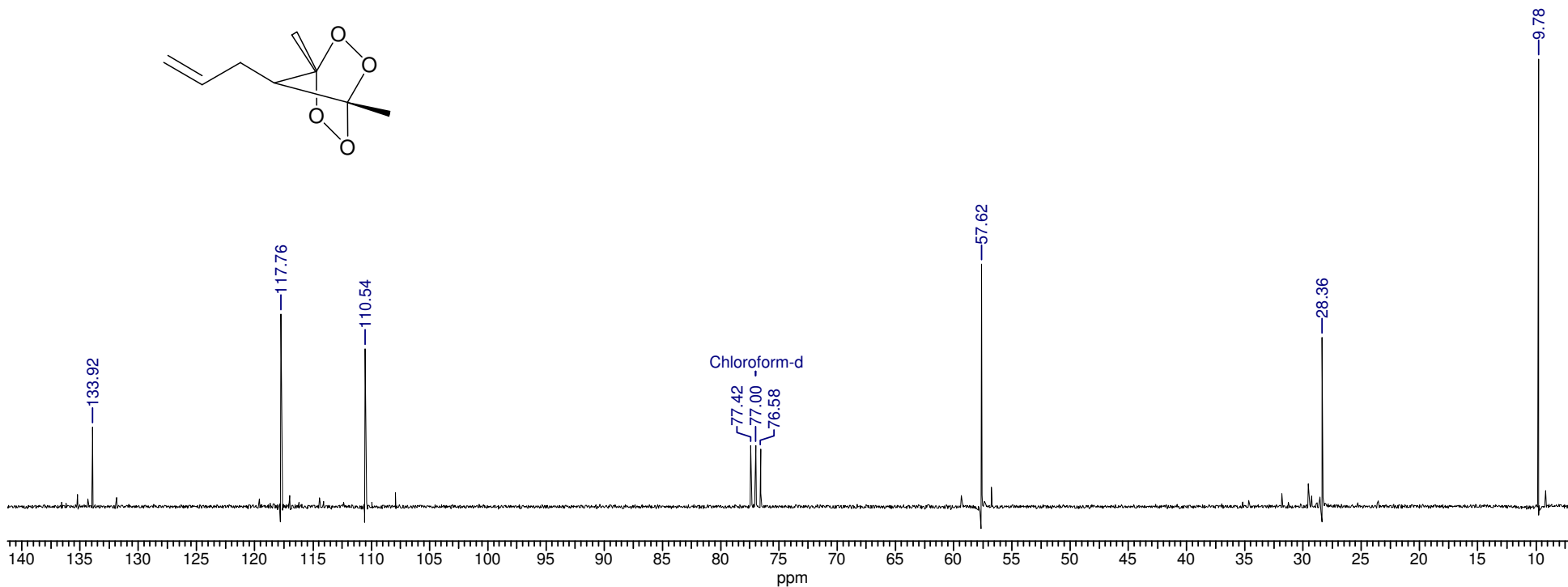
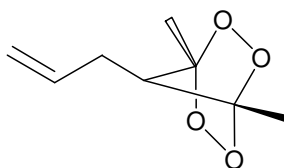
Acquisition Time (sec)	0.6759	Comment	Avance-300, CDCl3	Date	23 Apr 2008 11:16:16
File Name					
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	1
Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	21.500			Sweep Width (Hz)	6009.62



No.	(ppm)	(Hz)	Height	No.	Annotation	(ppm)	No.	(ppm)	Value
1	7.25	2175.9	0.0095	1	Chloroform-d	7.25	1	.30 .. 6.0	150.000

7-Allyl-1,4-dimethyl-2,3,5,6-tetraoxa-bicyclo[2.2.1]heptane (2c)

Acquisition Time (sec)	0.4501	Comment	Avance-300, C-13, CDCl3	Date	23 Apr 2008 11:16:16
File Name					
Frequency (MHz)	75.48	Nucleus	13C	Number of Transients	1054
Points Count	16384	Pulse Sequence	zgpg30base	Solvent	CHLOROFORM-D
Temperature (degree C)	21.700			Sweep Width (Hz)	18115.94

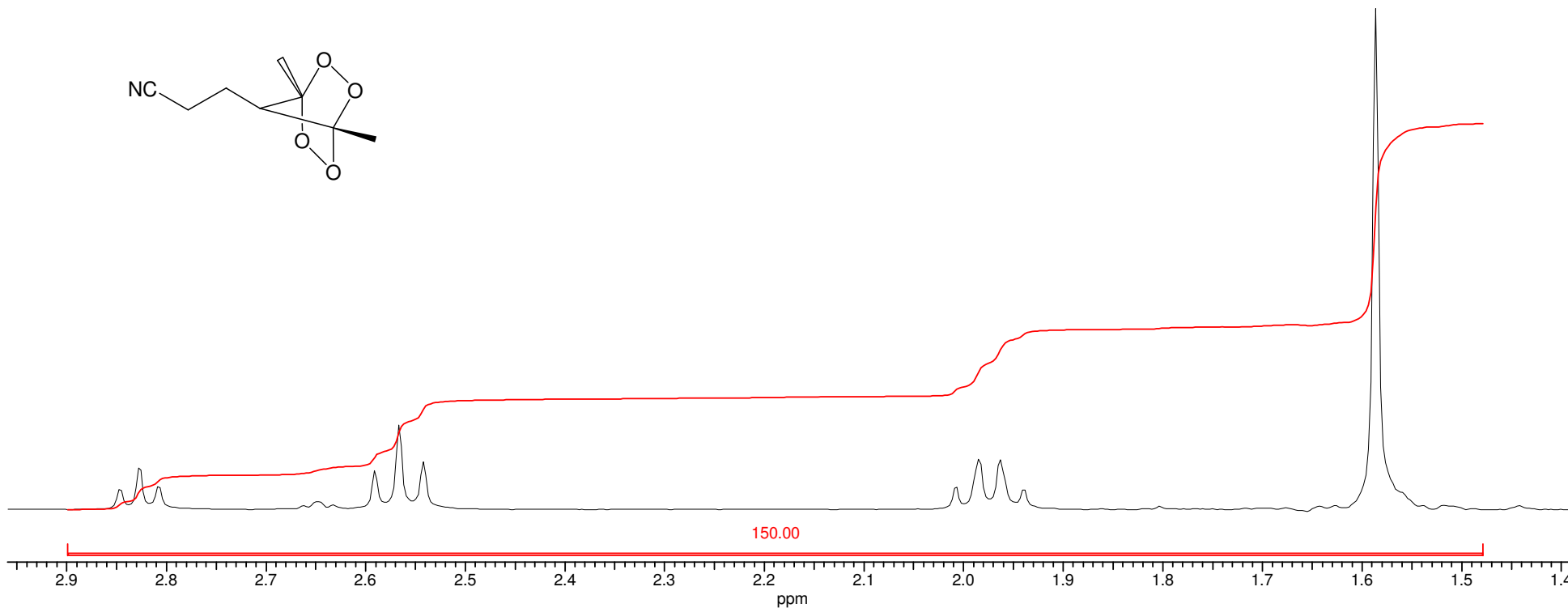
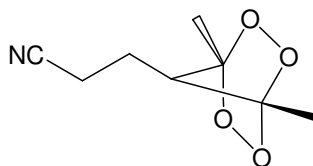


No.	(ppm)	(Hz)	Height
1	9.78	738.4	1.0000
2	28.36	2140.5	0.3785
3	57.62	4348.7	0.5423
4	76.58	5779.6	0.1291
5	77.00	5811.7	0.1368
6	77.42	5843.7	0.1368
7	110.54	8342.8	0.3536
8	117.76	8887.9	0.4308
9	133.92	10107.6	0.1783

No.	Annotation	(ppm)
1	Chloroform-d	77.00

3-(1,4-Dimethyl-2,3,5,6-tetraoxa-bicyclo[2.2.1]heptan-7-yl)propanenitrile (2d)

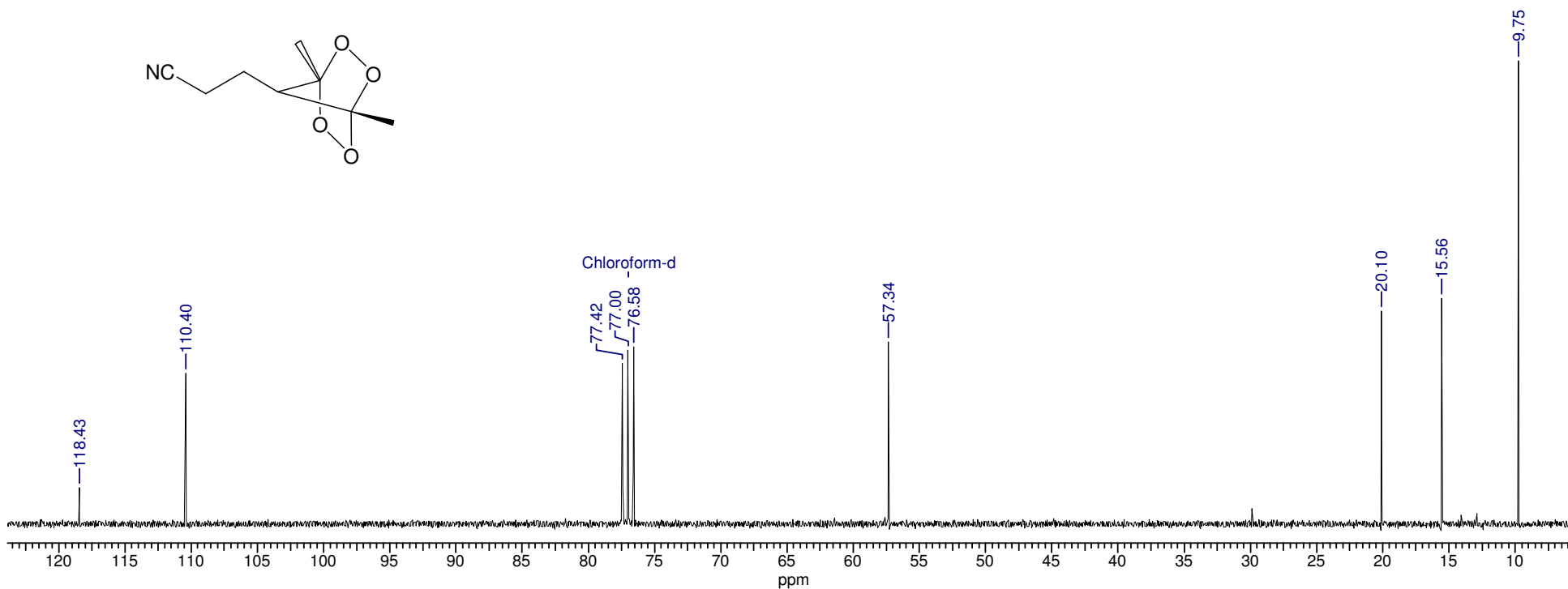
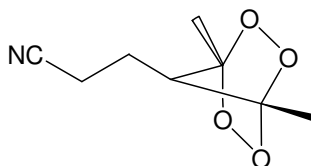
Acquisition Time (sec)	0.6759	Comment	Avance-300, CDCl3	Date	04 Mar 2008 11:50:24
File Name					
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	1
Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	27.000			Sweep Width (Hz)	6009.62



No.	(ppm)	(Hz)	Height	No.	Annotation	(ppm)	No.	(ppm)	Value
1	7.25	2176.1	0.0223	1	Chloroform-d	7.25	1	.48 .. 2.9	150.000

3-(1,4-Dimethyl-2,3,5,6-tetraoxa-bicyclo[2.2.1]heptan-7-yl)propanenitrile (2d)

Acquisition Time (sec)	0.4501	Comment	Avance-300, C-13, CDCl3	Date	04 Mar 2008 11:52:32
File Name					
Frequency (MHz)	75.48	Nucleus	¹³ C	Number of Transients	798
Points Count	16384	Pulse Sequence	zgpg30base	Solvent	CHLOROFORM-D
Temperature (degree C)	20.500			Sweep Width (Hz)	18115.94

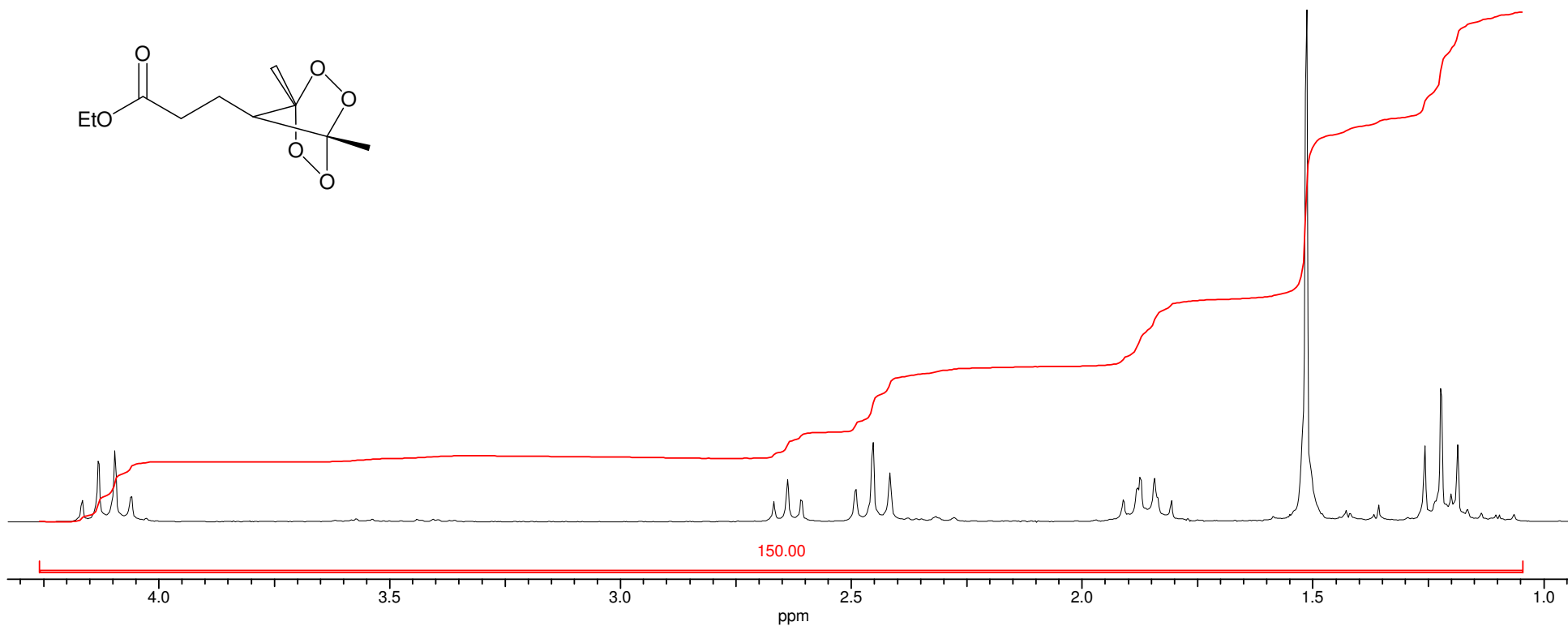
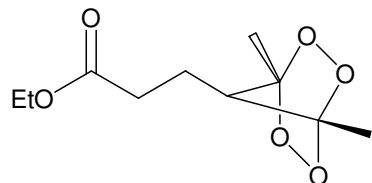


No.	(ppm)	(Hz)	Height
1	9.75	736.1	1.0000
2	15.56	1174.0	0.4876
3	20.10	1516.8	0.4595
4	57.34	4327.7	0.3931
5	76.58	5779.6	0.3827
6	77.00	5811.7	0.3757
7	77.42	5843.7	0.3471
8	110.40	8332.8	0.3259
9	118.43	8938.8	0.0789

No.	Annotation	(ppm)
1	Chloroform-d	77.00

Ethyl 3-(1,4-dimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]hept-7-yl)propanoate (2e)

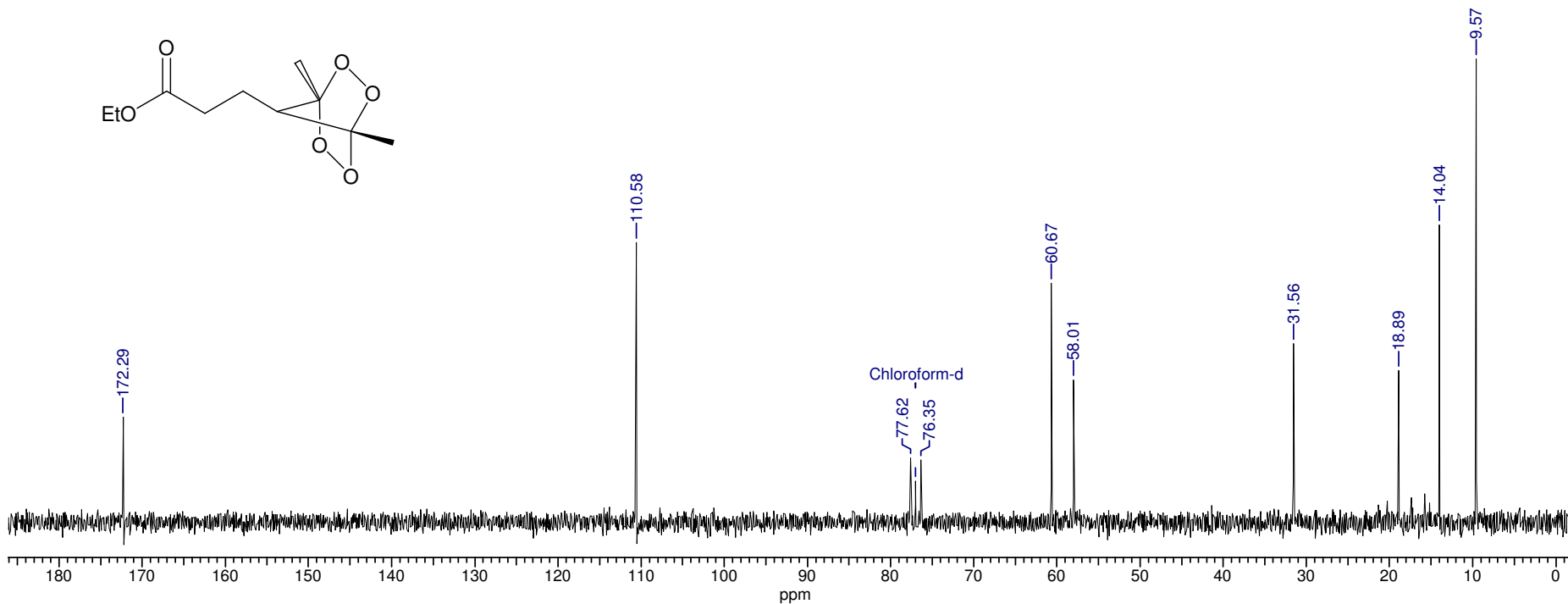
Acquisition Time (sec)	1.0240	Comment /TERN BORIS123 Opr:Struchkova M.I.;Solv:CDCl3;		Date	04/07/2008 18:57:45
File Name				Frequency (MHz)	200.13
Nucleus	¹ H	Number of Transients	16	Original Points Count	8192
Solvent	CHLOROFORM-D	Sweep Width (Hz)	4000.00	Points Count	8192
				Temperature (degree C)	24.000



No.	(ppm)	(Hz)	Height	No.	Annotation	(ppm)	No.	(ppm)	Value
1	7.25	1451.0	0.0077	1	Chloroform-d	7.25	1	.05 .. 4.2	150.000

Ethyl 3-(1,4-dimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]hept-7-yl)propanoate (2e)

Acquisition Time (sec)	0.3359	Comment	/TERN BORIS123.C13□Opr:Struchkova M.I.;Solv:CDCl3;		Date	04/07/2008 19:23:47	
File Name					Frequency (MHz)	50.32	
Nucleus	13C	Original Points Count	8192	Points Count	8192	Solvent	CHLOROFORM-D
Sweep Width (Hz)	12195.12	Temperature (degree C)	24.000				

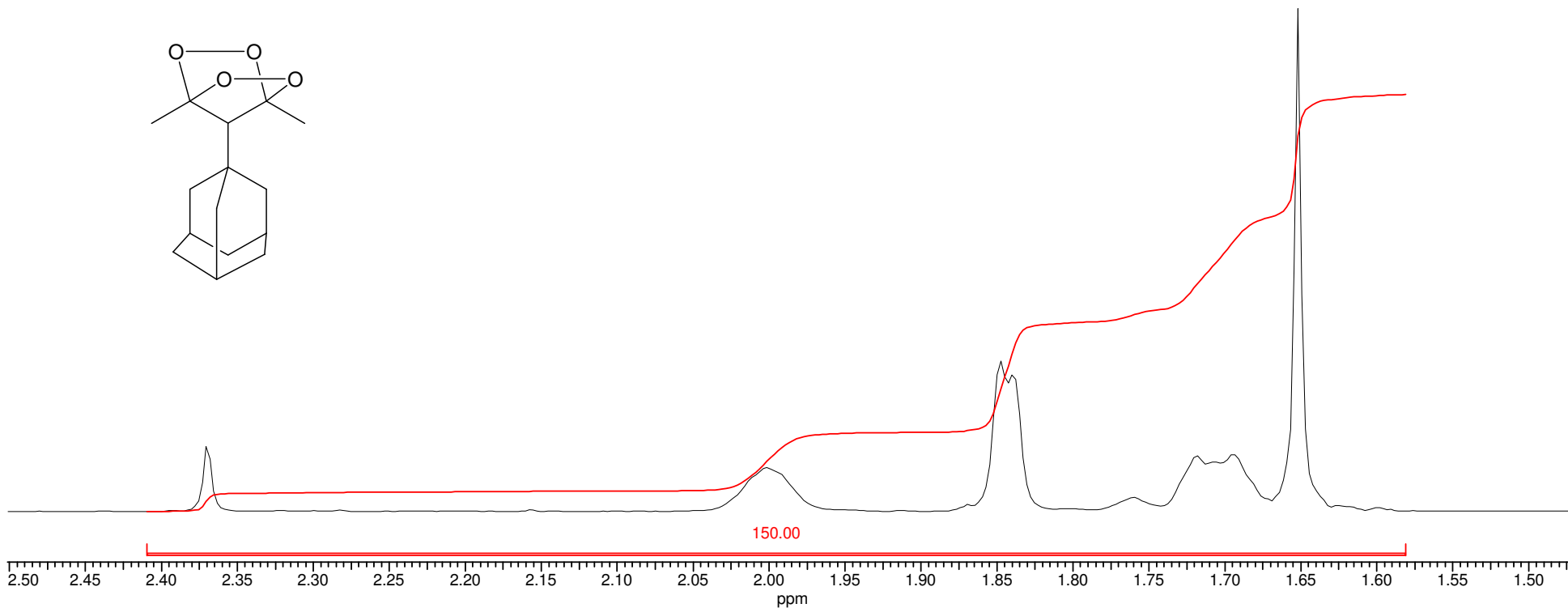
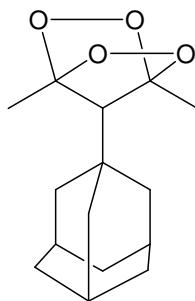


No.	(ppm)	(Hz)	Height
1	9.57	481.8	1.0000
2	14.04	706.7	0.6414
3	18.89	950.8	0.3271
4	31.56	1588.1	0.3851
5	58.01	2919.1	0.3072
6	60.67	3053.1	0.5151
7	76.35	3842.2	0.1336
8	77.00	3874.9	0.0880
9	77.62	3906.2	0.1388
10	110.58	5564.8	0.6038
11	172.29	8670.5	0.2266

No.	Annotation	(ppm)
1	Chloroform-d	77.00

7-(1-Adamantyl)-1,4-dimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2f)

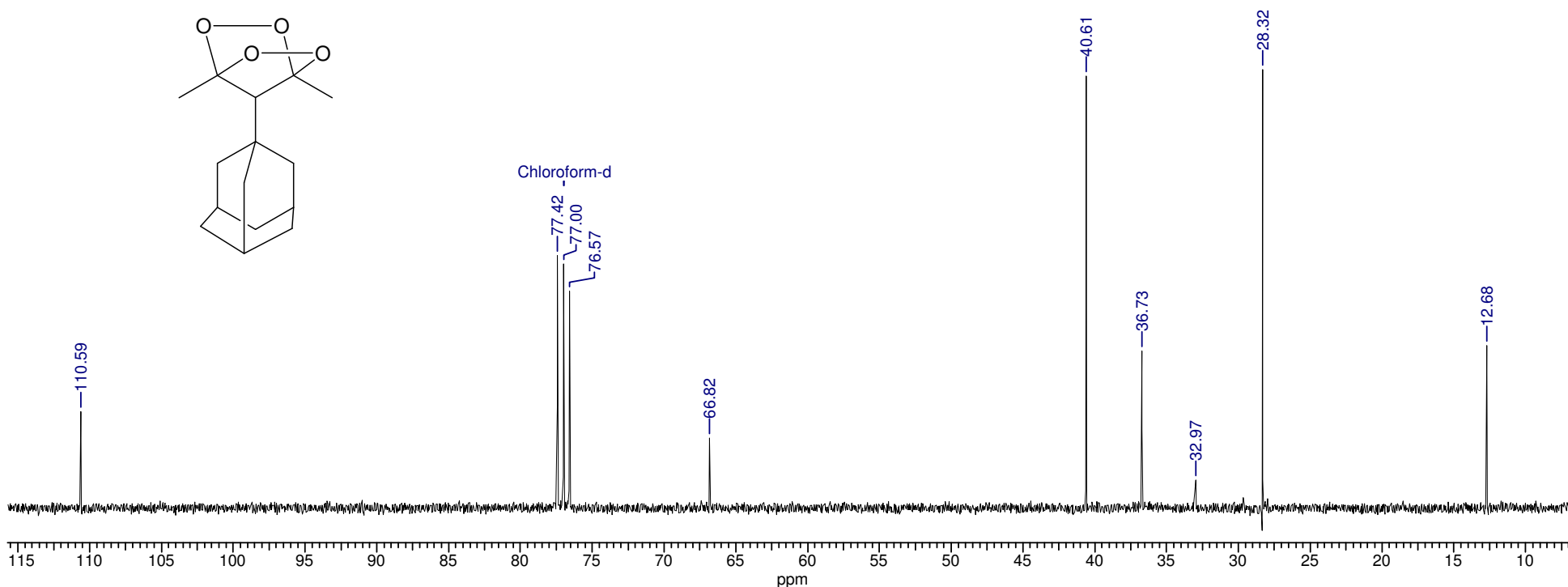
Acquisition Time (sec)	0.6759	Comment	Avance-300, CDCl3	Date	08 Jul 2008 12:33:04
File Name					
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	1
Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	24.100			Original Points Count	8124
				Sweep Width (Hz)	6009.62



No.	(ppm)	(Hz)	Height	No.	Annotation	(ppm)	No.	(ppm)	Value
1	7.25	2175.9	0.0581	1	Chloroform-d	7.25	1	.58 .. 2.4	150.000

7-(1-Adamantyl)-1,4-dimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2f)

Acquisition Time (sec)	0.4501	Comment	Avance-300, C-13, CDCl3	Date	08 Jul 2008 12:35:12
File Name					
Frequency (MHz)	75.48	Nucleus	13C	Number of Transients	738
Points Count	16384	Pulse Sequence	zgpg30base	Solvent	CHLOROFORM-D
Temperature (degree C)	24.200			Sweep Width (Hz)	18115.94

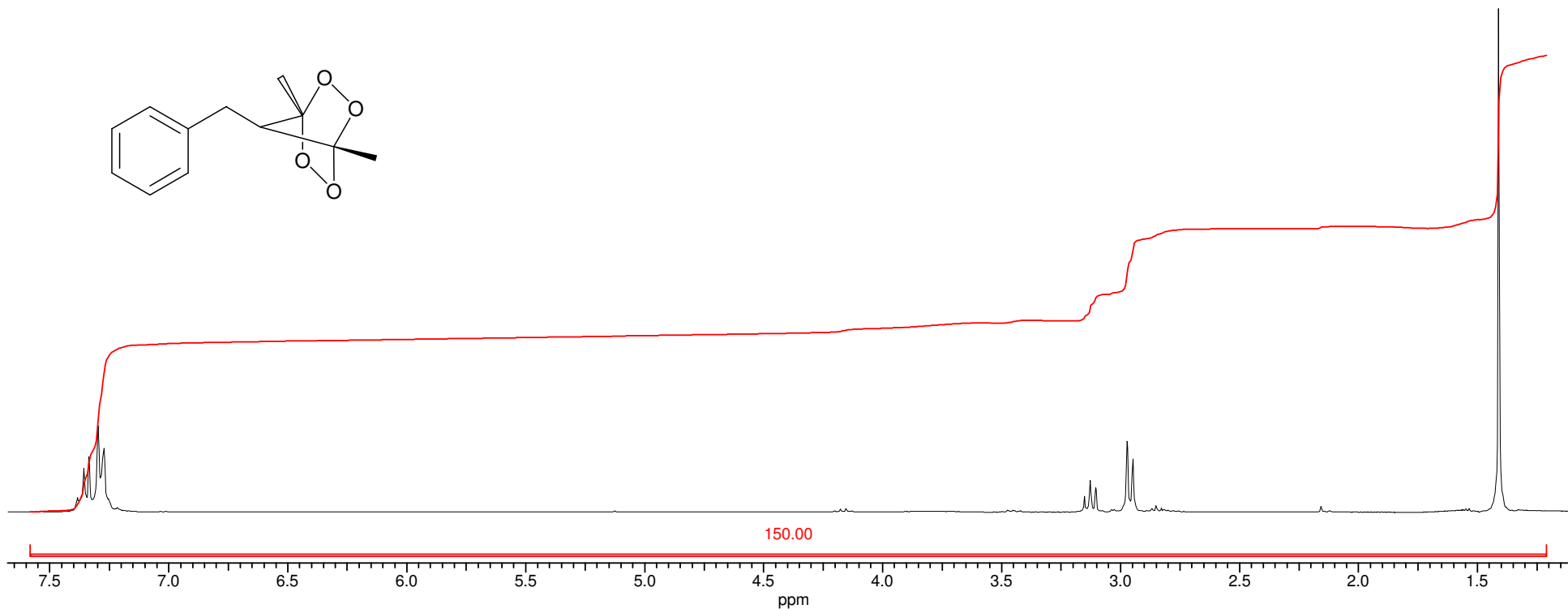
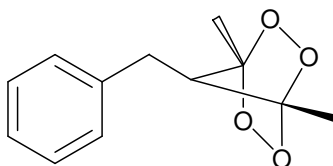


No.	(ppm)	(Hz)	Height
1	12.68	957.3	0.3708
2	28.32	2137.1	1.0000
3	32.97	2488.8	0.0638
4	36.73	2771.9	0.3581
5	40.61	3064.9	0.9851
6	66.82	5043.1	0.1598
7	76.57	5779.6	0.4949
8	77.00	5811.6	0.5566
9	77.42	5843.7	0.5759
10	110.59	8347.2	0.2199

No.	Annotation	(ppm)
1	Chloroform-d	77.00

7-Benzyl-1,4-dimethyl-2,3,5,6-tetraoxa-bicyclo[2.2.1]heptane (2g)

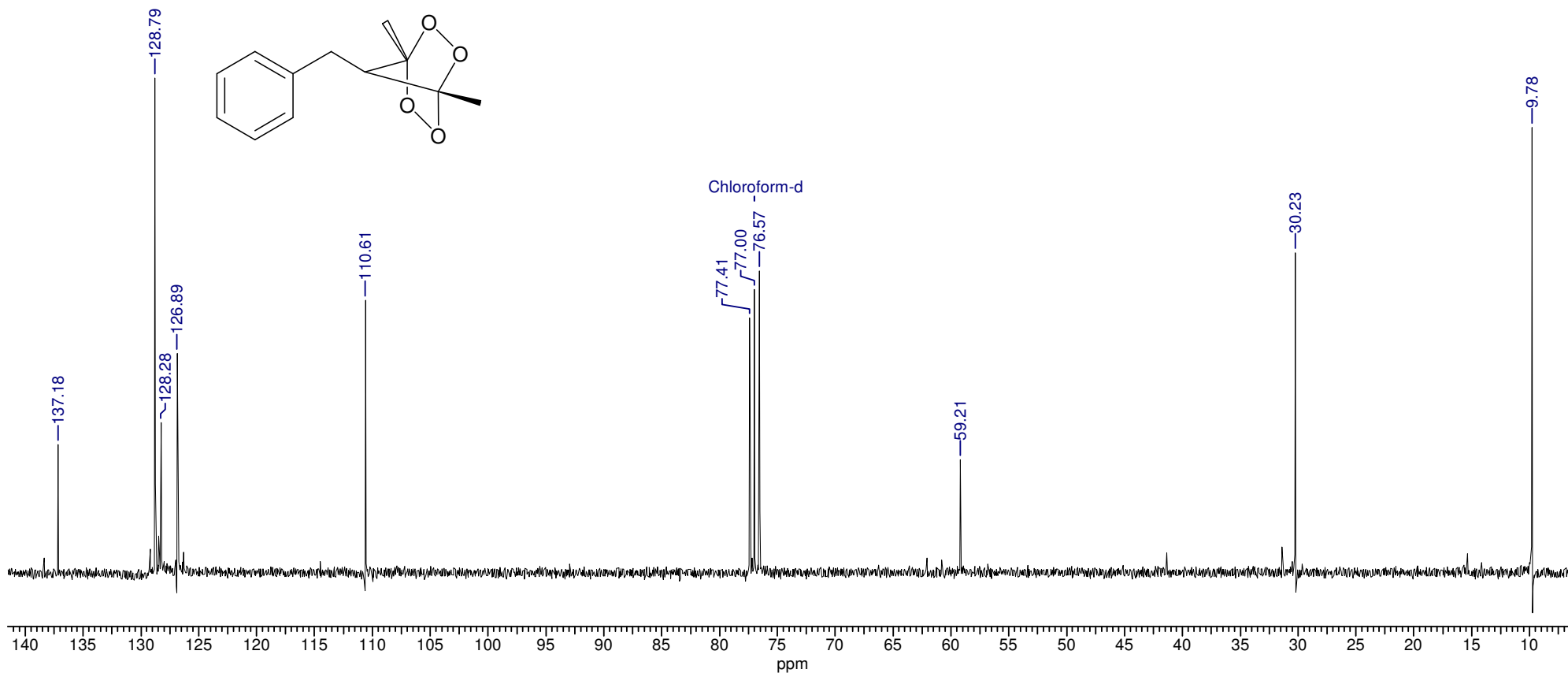
Acquisition Time (sec)	0.6759	Comment	Avance-300, CDCl3	Date	31 Jan 2008 12:26:40
File Name					
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	1
Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	23.500			Sweep Width (Hz)	6009.62



No.	(ppm)	Value
1	.21 .. 7.5	150.000

7-Benzyl-1,4-dimethyl-2,3,5,6-tetraoxa-bicyclo[2.2.1]heptane (2g)

Acquisition Time (sec)	0.4501	Comment	Avance-300, C-13, CDCl3			Date	31 Jan 2008 12:26:40
File Name							
Frequency (MHz)	75.48	Nucleus	13C	Number of Transients	1712	Original Points Count	16308
Points Count	16384	Pulse Sequence	zgpg30base	Solvent	CHLOROFORM-D	Sweep Width (Hz)	18115.94
Temperature (degree C)	23.600						



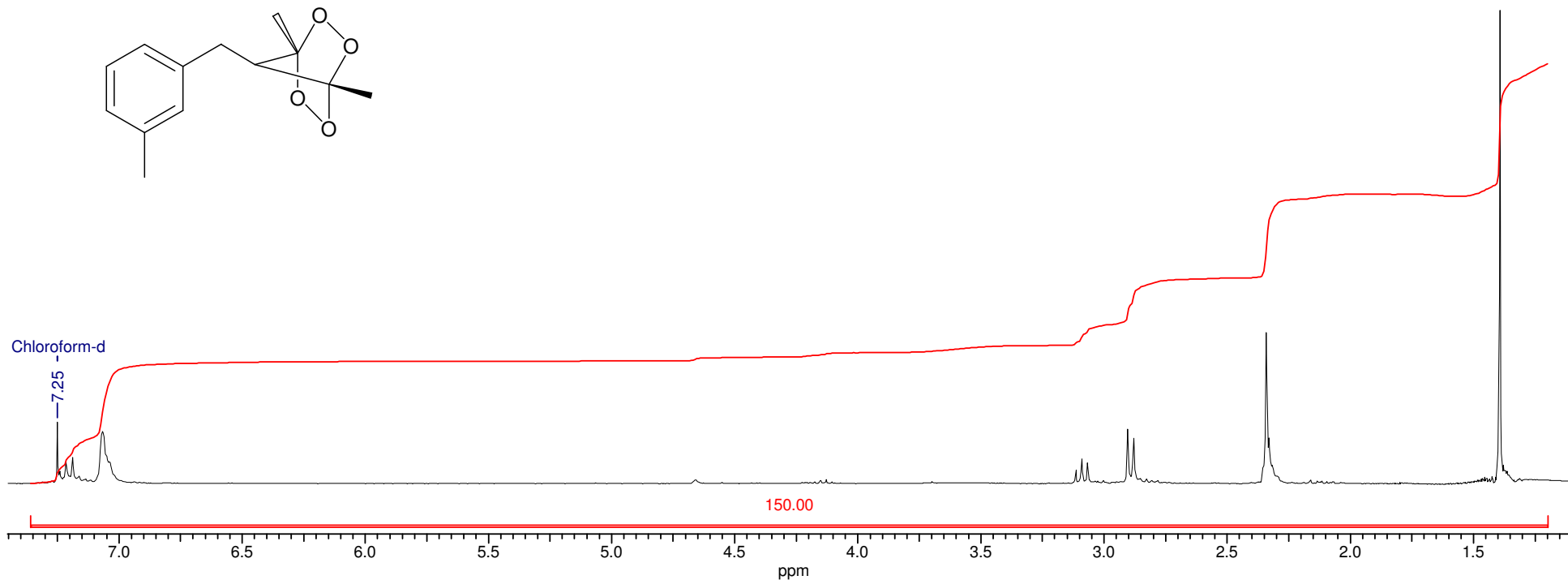
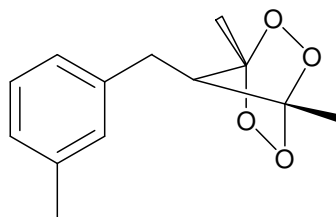
No.	(ppm)	(Hz)	Height
1	9.78	738.3	0.9001
2	30.23	2282.0	0.6468
3	59.21	4469.2	0.2281
4	76.57	5779.6	0.6102
5	77.00	5811.6	0.5733
6	77.41	5842.6	0.5152

No.	(ppm)	(Hz)	Height
7	110.61	8348.3	0.5511
8	126.89	9576.8	0.4429
9	128.28	9681.8	0.3037
10	128.79	9720.5	1.0000
11	137.18	10354.2	0.2590

No.	Annotation	(ppm)
1	Chloroform-d	77.00

1,4-Dimethyl-7-(3-methylbenzyl)-2,3,5,6-tetraoxa-bicyclo[2.2.1]heptane (2h)

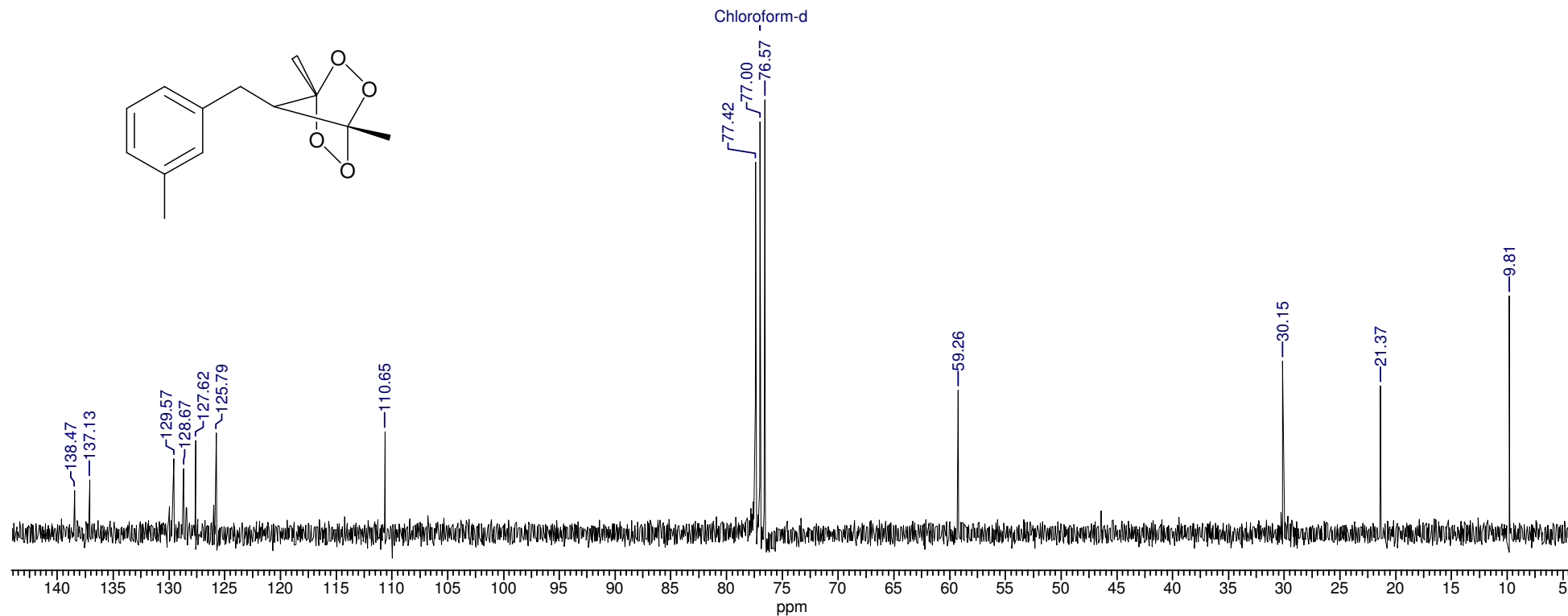
Acquisition Time (sec)	0.6759	Comment	Avance-300, CDCl3	Date	30 Apr 2008 09:33:52
File Name					
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	1
Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	22.900			Sweep Width (Hz)	6009.62



No.	(ppm)	(Hz)	Height	No.	Annotation	(ppm)	No.	(ppm)	Value
1	7.25	2175.9	0.1295	1	Chloroform-d	7.25	1	.20 .. 7.3	150.000

1,4-Dimethyl-7-(3-methylbenzyl)-2,3,5,6-tetraoxa-bicyclo[2.2.1]heptane (2h)

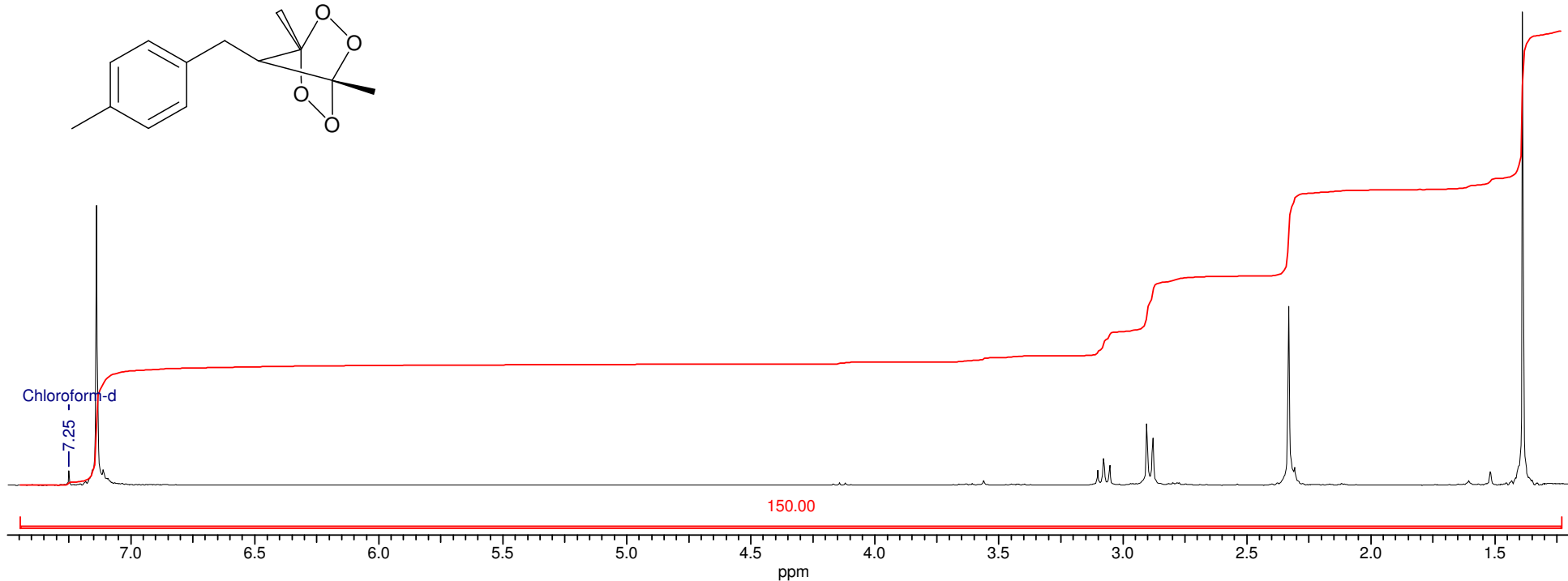
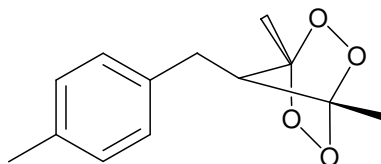
Acquisition Time (sec)	0.4501	Comment	Avance-300, C-13, CDCl3		Date	30 Apr 2008 09:36:00	
File Name							
Frequency (MHz)	75.48	Nucleus	13C	Number of Transients	864	Original Points Count	16308
Points Count	16384	Pulse Sequence	zgpg30base	Solvent	CHLOROFORM-D	Sweep Width (Hz)	18115.94
Temperature (degree C)	23.000						



No.	(ppm)	(Hz)	Height	No.	(ppm)	(Hz)	Height	No.	Annotation	(ppm)
1	9.81	740.5	0.5478	8	110.65	8351.6	0.2358	1	Chloroform-d	77.00
2	21.37	1613.0	0.3412	9	125.79	9493.9	0.2324			
3	30.15	2275.4	0.3980	10	127.62	9632.1	0.2155			
4	59.26	4472.5	0.3306	11	128.67	9711.7	0.1508			
5	76.57	5779.6	1.0000	12	129.57	9779.2	0.1733			
6	77.00	5811.6	0.9494	13	137.13	10349.7	0.1245			
7	77.42	5843.7	0.8571	14	138.47	10451.5	0.0997			

1,4-Dimethyl-7-(4-methylbenzyl)-2,3,5,6-tetraoxa-bicyclo[2.2.1]heptane (2i)

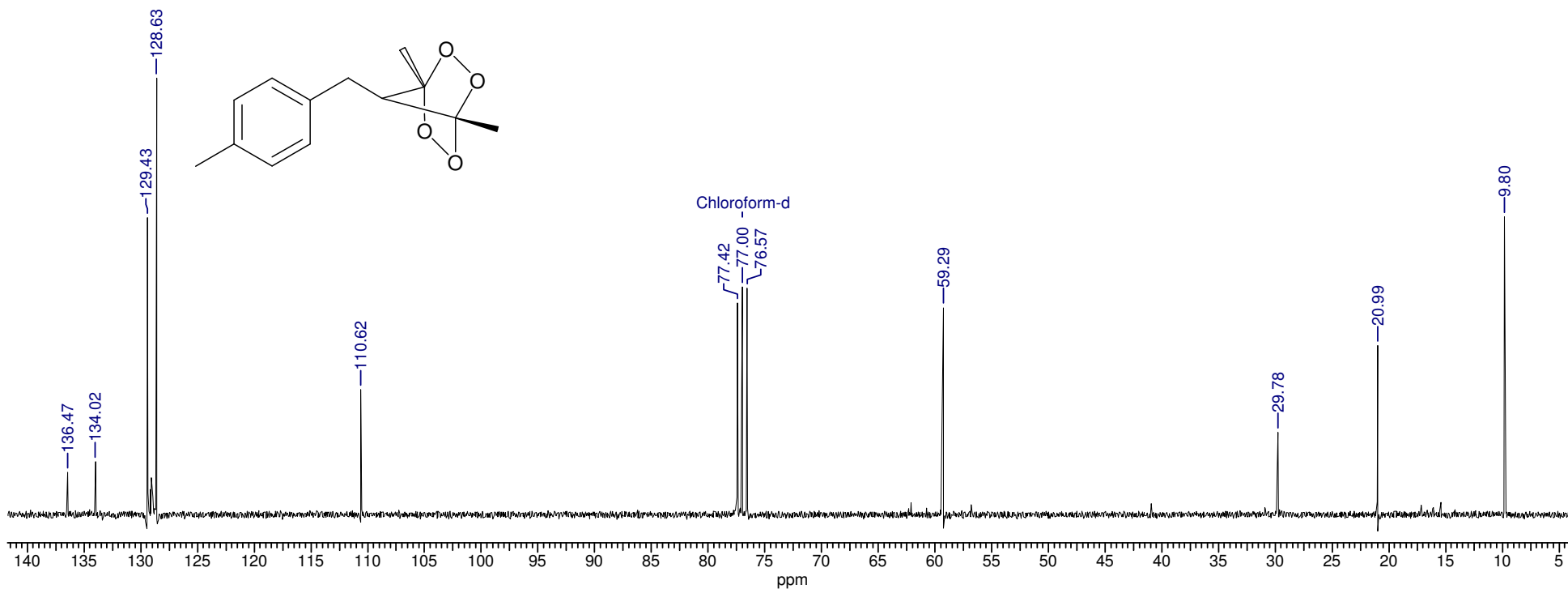
Acquisition Time (sec)	0.6759	Comment	Avance-300, CDCl3	Date	27 Feb 2008 13:07:12
File Name					
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	1
Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	20.400			Sweep Width (Hz)	6009.62



No.	(ppm)	(Hz)	Height	No.	Annotation	(ppm)	No.	(ppm)	Value
1	7.25	2176.1	0.0301	1	Chloroform-d	7.25	1	.23 .. 7.4	150.000

1,4-Dimethyl-7-(4-methylbenzyl)-2,3,5,6-tetraoxa-bicyclo[2.2.1]heptane (2i)

Acquisition Time (sec)	0.4501	Comment	Avance-300, C-13, CDCl3	Date	27 Feb 2008 12:35:12
File Name					
Frequency (MHz)	75.48	Nucleus	13C	Number of Transients	1354
Points Count	16384	Pulse Sequence	zgpg30base	Solvent	CHLOROFORM-D
Temperature (degree C)	20.400			Sweep Width (Hz)	18115.94



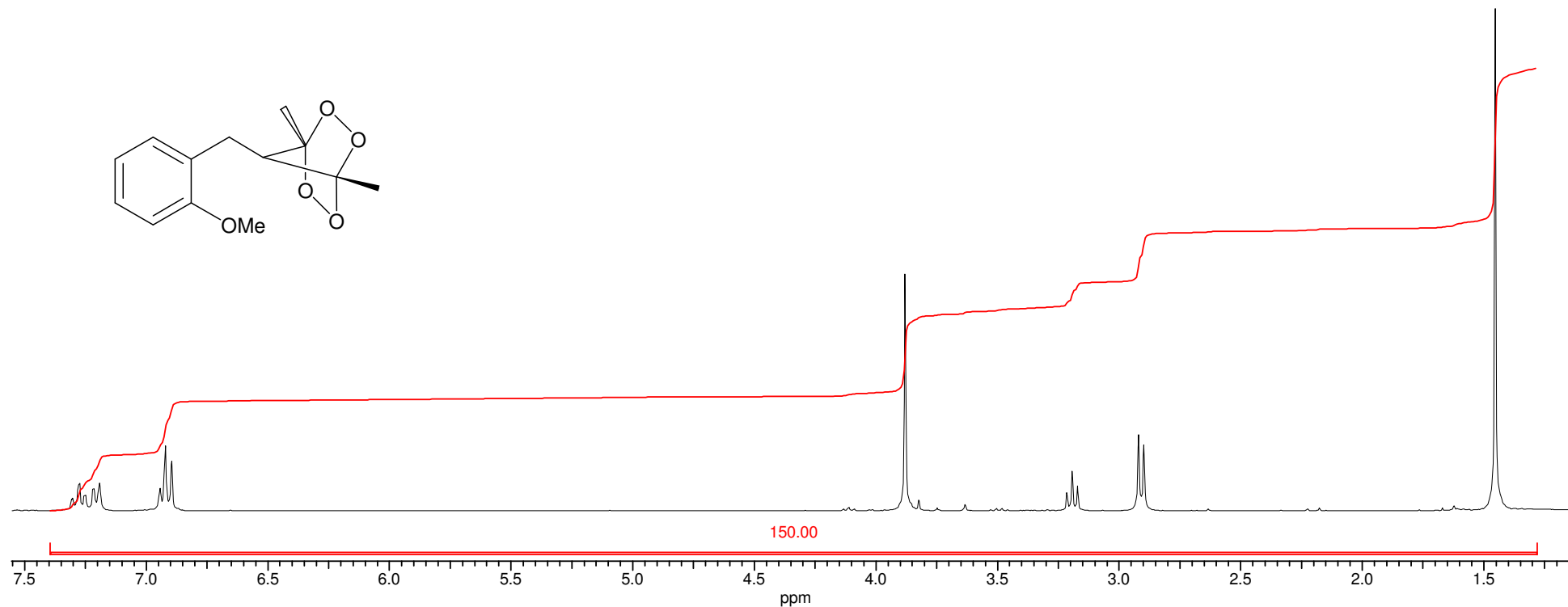
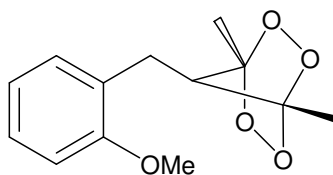
No.	(ppm)	(Hz)	Height
1	9.80	739.4	0.6833
2	20.99	1584.2	0.3882
3	29.78	2247.7	0.1889
4	59.29	4474.7	0.4747
5	76.57	5779.6	0.5188
6	77.00	5811.6	0.5220

No.	(ppm)	(Hz)	Height
7	77.42	5843.7	0.4856
8	110.62	8349.4	0.2871
9	128.63	9708.4	1.0000
10	129.43	9769.2	0.6802
11	134.02	10115.3	0.1221
12	136.47	10300.0	0.0972

No.	Annotation	(ppm)
1	Chloroform-d	77.00

7-(2-Methoxybenzyl)-1,4-dimethyl-2,3,5,6-tetraoxa-bicyclo[2.2.1]heptane (2j)

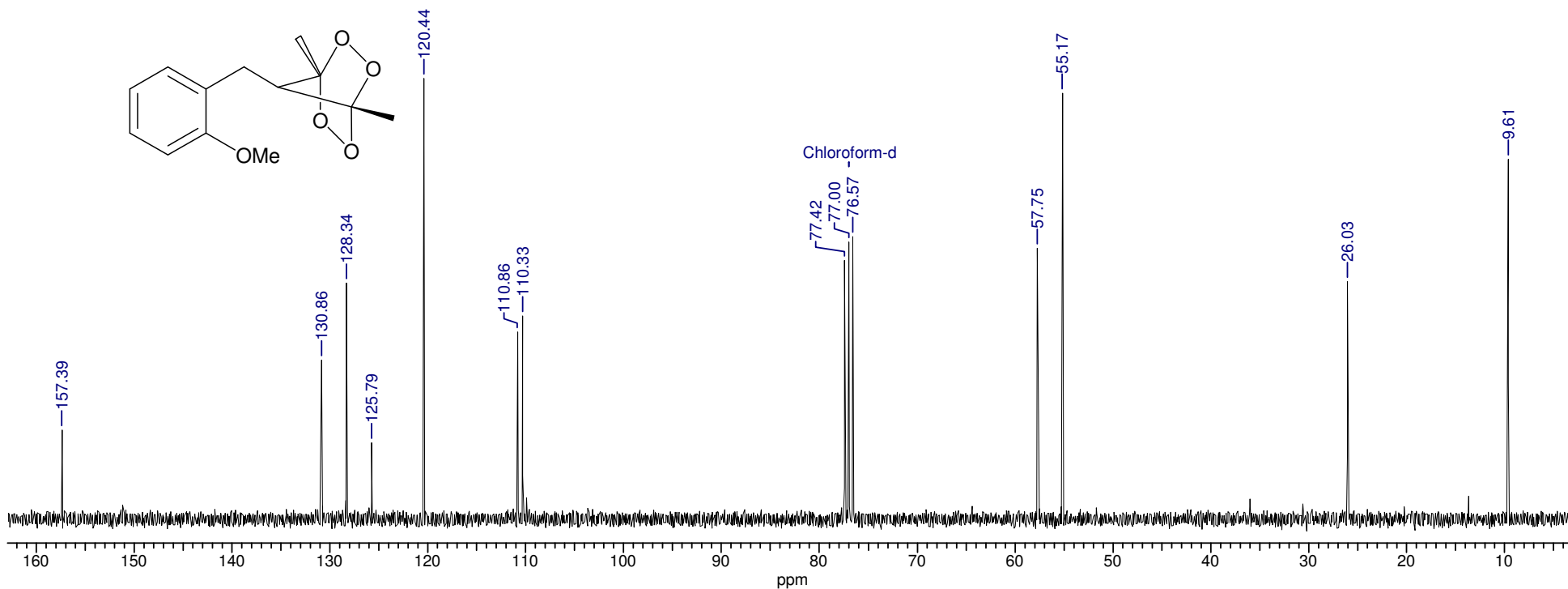
Acquisition Time (sec)	0.6759	Comment	Avance-300, CDCl3	Date	23 Jan 2008 09:59:28
File Name					
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	1
Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	21.400			Sweep Width (Hz)	6009.62



No.	(ppm)	Value
1	.28 .. 7.4	150.000

7-(2-Methoxybenzyl)-1,4-dimethyl-2,3,5,6-tetraoxa-bicyclo[2.2.1]heptane (2j)

Acquisition Time (sec)	0.4501	Comment	Avance-300, C-13, CDCl3	Date	23 Jan 2008 10:01:36
File Name					
Frequency (MHz)	75.48	Nucleus	13C	Number of Transients	474
Points Count	16384	Pulse Sequence	zgpg30base	Solvent	CHLOROFORM-D
Temperature (degree C)	21.500			Sweep Width (Hz)	18115.94

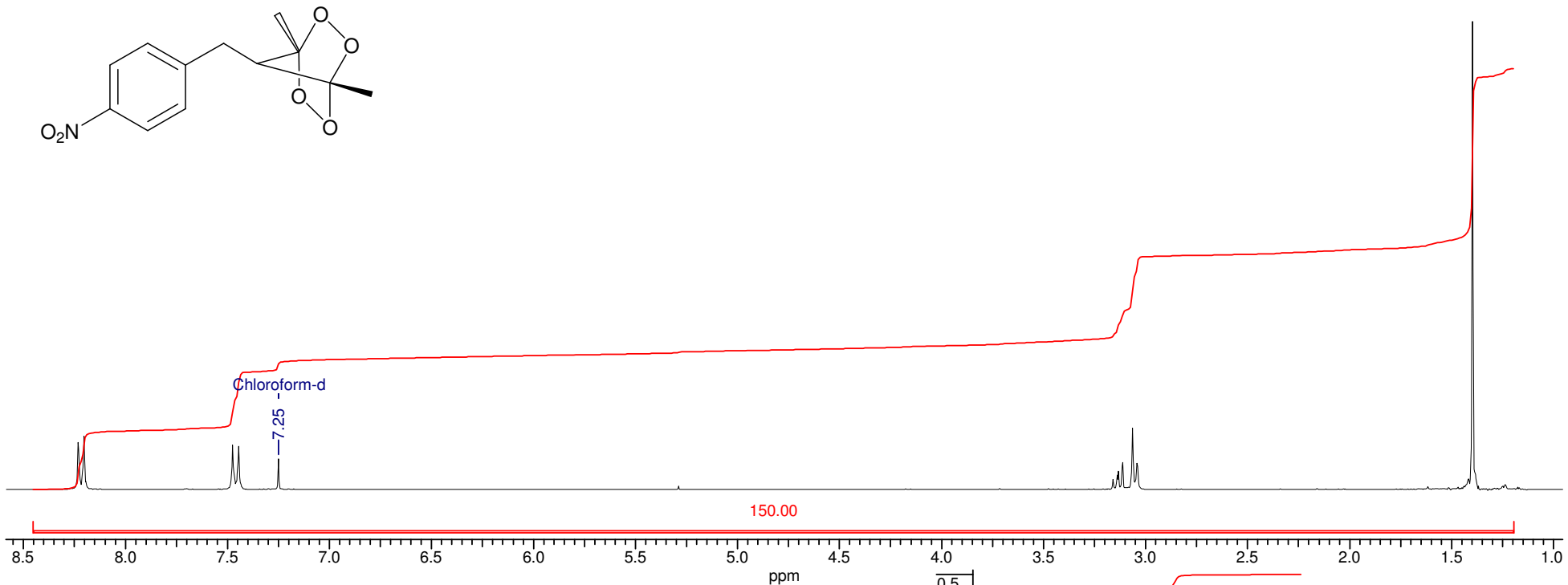
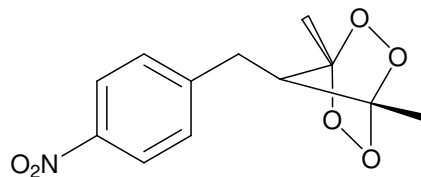


No.	(ppm)	(Hz)	Height	No.	(ppm)	(Hz)	Height
1	9.61	725.0	0.8165	8	110.33	8327.3	0.4608
2	26.03	1964.6	0.5390	9	110.86	8367.1	0.4240
3	55.17	4164.0	0.9665	10	120.44	9090.3	1.0000
4	57.75	4358.6	0.6149	11	125.79	9493.9	0.1734
5	76.57	5779.6	0.6416	12	128.34	9686.3	0.5360
6	77.00	5811.6	0.6285	13	130.86	9876.5	0.3606
7	77.42	5843.7	0.5866	14	157.39	11879.0	0.2027

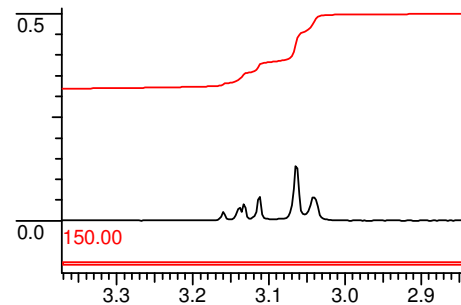
No.	Annotation	(ppm)
1	Chloroform-d	77.00

1,4-Dimethyl-7-(4-nitrobenzyl)-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2k)

Acquisition Time (sec)	0.6759	Comment	Avance-300, CDCI3	Date	24 Apr 2008 12:54:24
File Name					
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	1
Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	22.500			Sweep Width (Hz)	6009.62

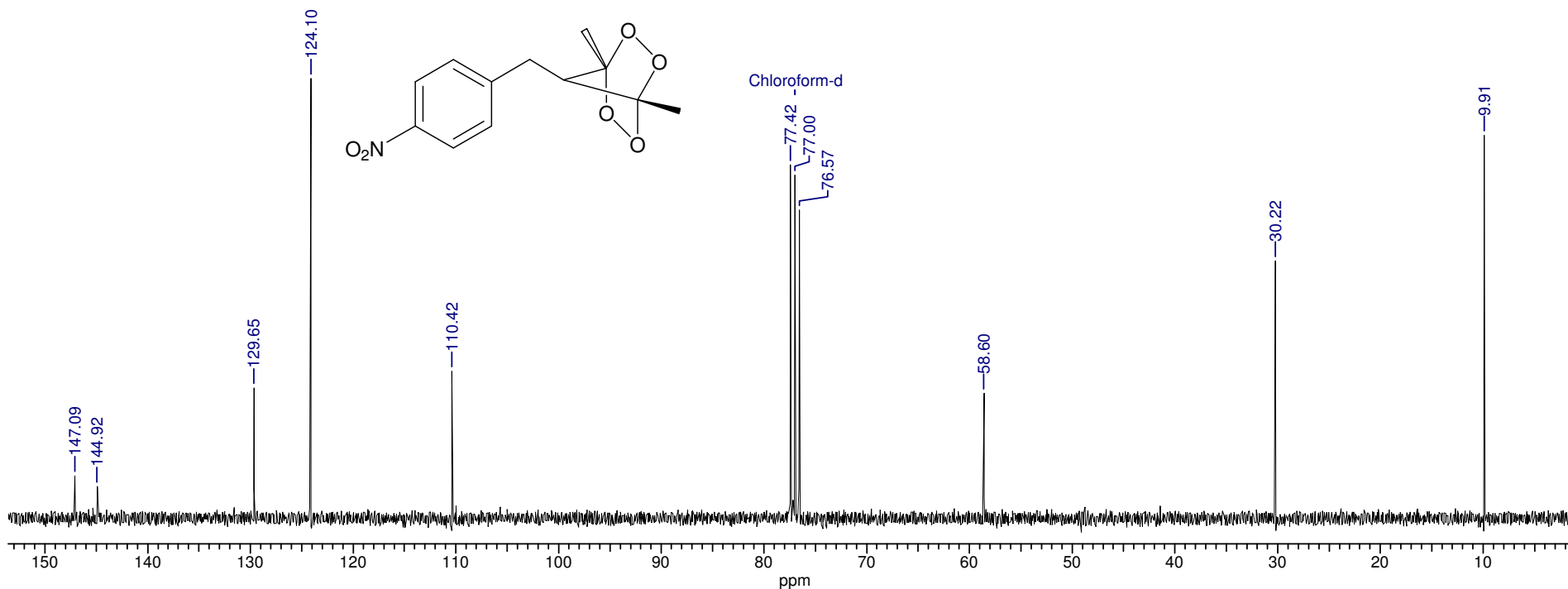


No.	(ppm)	(Hz)	Height	No.	Annotation	(ppm)	No.	(ppm)	Value
1	7.25	2175.9	0.0655	1	Chloroform-d	7.25	1	.19 .. 8.4	150.000



1,4-Dimethyl-7-(4-nitrobenzyl)-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2k)

Acquisition Time (sec)	0.4501	Comment	Avance-300, C-13, CDCl3	Date	24 Apr 2008 11:37:36
File Name					
Frequency (MHz)	75.48	Nucleus	13C	Number of Transients	1182
Points Count	16384	Pulse Sequence	zgpg30base	Solvent	CHLOROFORM-D
Temperature (degree C)	21.800			Sweep Width (Hz)	18115.94

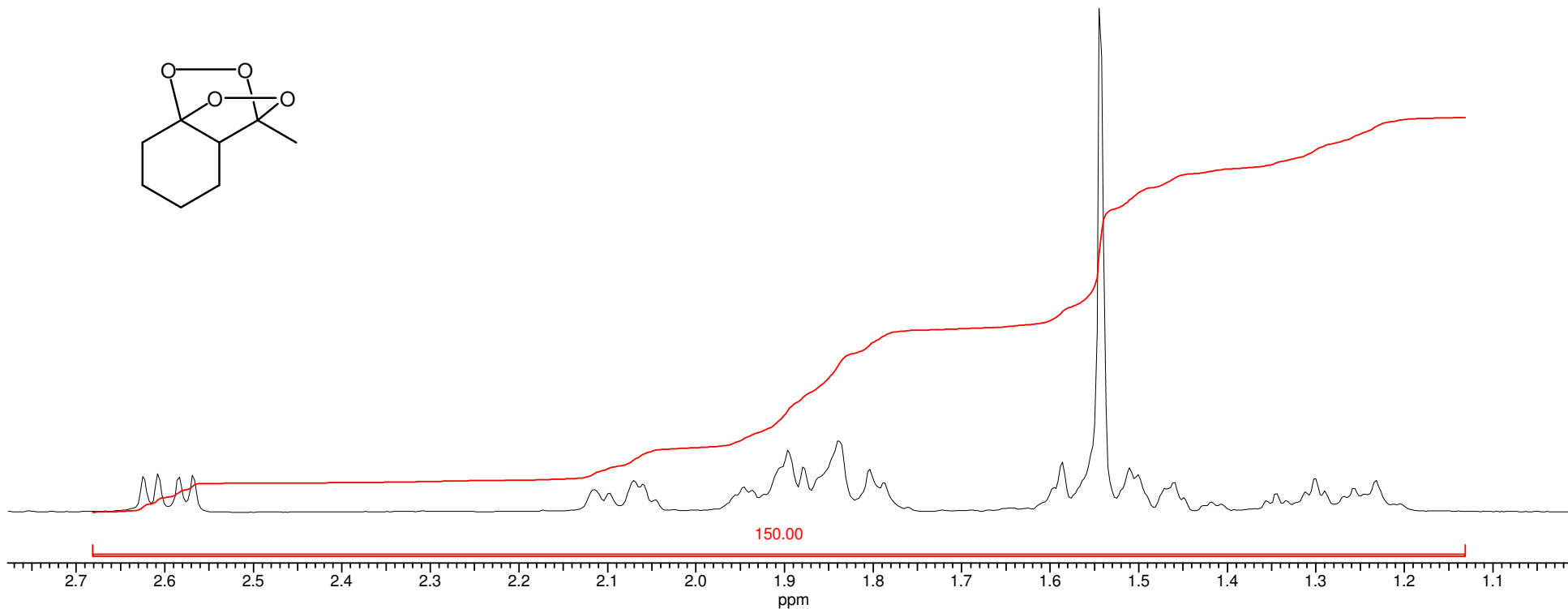
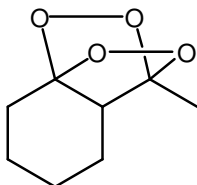


No.	(ppm)	(Hz)	Height
1	9.91	748.3	0.8709
2	30.22	2280.9	0.5851
3	58.60	4422.8	0.2838
4	76.57	5779.6	0.7011
5	77.00	5811.6	0.7807
6	77.42	5843.7	0.8040
7	110.42	8333.9	0.3349
8	124.10	9366.7	1.0000
9	129.65	9785.8	0.2964
10	144.92	10938.0	0.0722
11	147.09	11101.7	0.0969

No.	Annotation	(ppm)
1	Chloroform-d	77.00

3-Methylhexahydro-3,7a-epidioxy-1,2-benzodioxole (2l)

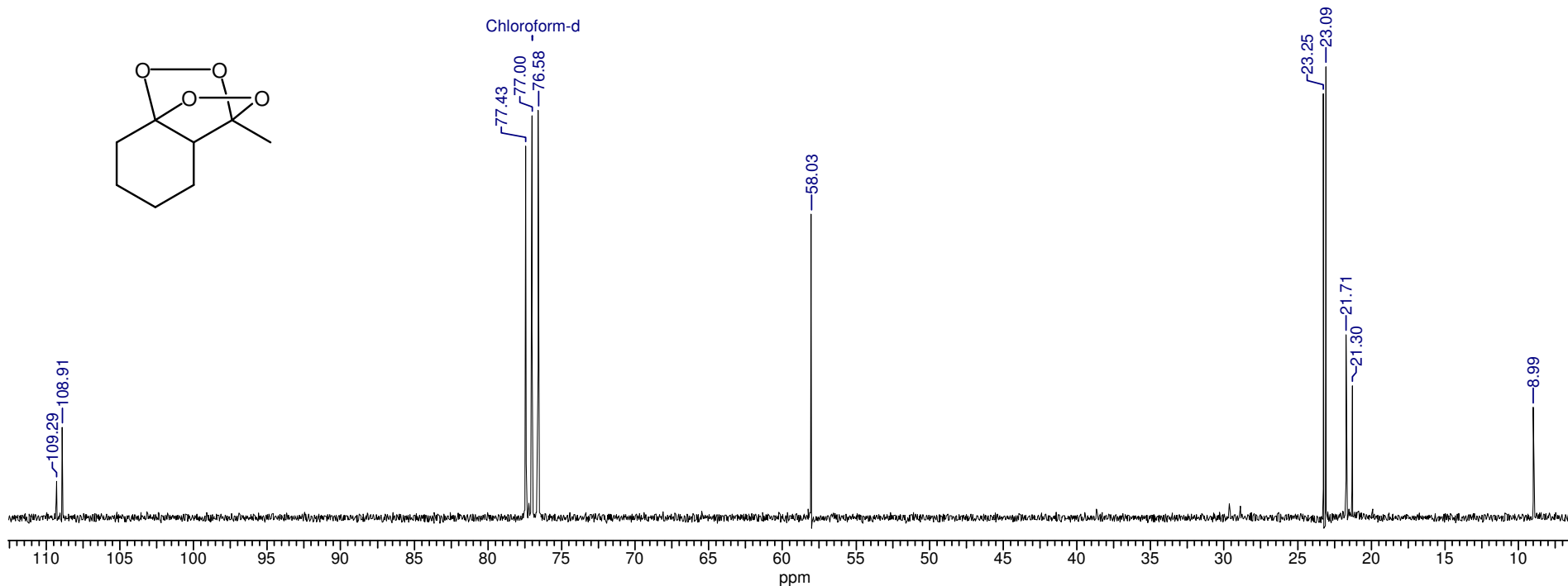
Acquisition Time (sec)	0.6759	Comment	Avance-300, CDCl ₃	Date	07 Oct 2008 11:16:16
File Name					
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	1
Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	20.400			Sweep Width (Hz)	6009.62



No.	(ppm)	(Hz)	Height	No.	Annotation	(ppm)	No.	(ppm)	Value
1	7.25	2176.0	0.0542	1	Chloroform-d	7.25	1	.13 .. 2.6	150.000

3-Methylhexahydro-3,7a-epidioxy-1,2-benzodioxole (2l)

Acquisition Time (sec)	0.4501	Comment	Avance-300, C-13, CDCl3		Date	07 Oct 2008 11:18:24	
File Name							
Frequency (MHz)	75.48	Nucleus	13C	Number of Transients	3731	Original Points Count	16308
Points Count	16384	Pulse Sequence	zgpg30base	Solvent	CHLOROFORM-D	Sweep Width (Hz)	18115.94
Temperature (degree C)	20.500						

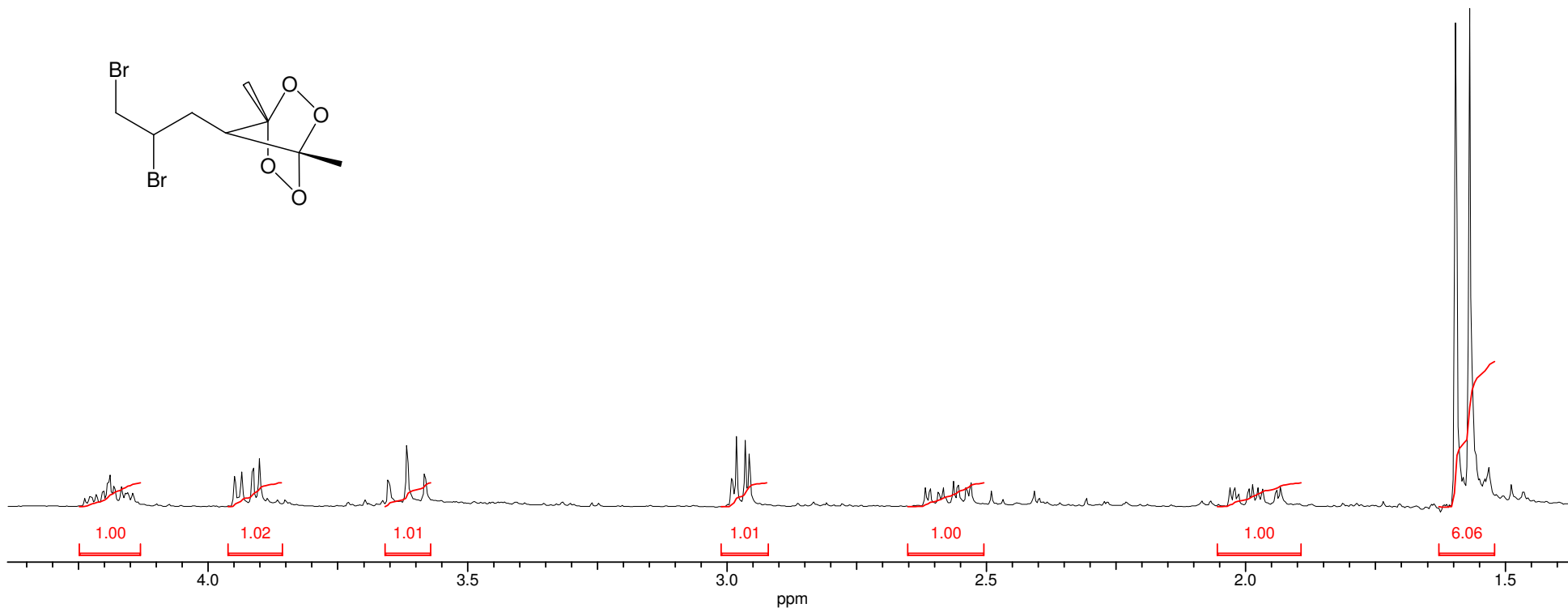
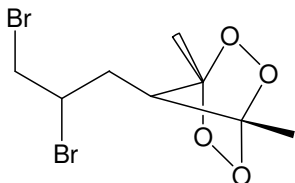


No.	(ppm)	(Hz)	Height
1	8.99	678.7	0.2454
2	21.30	1607.5	0.2931
3	21.71	1638.5	0.4053
4	23.09	1742.4	1.0000
5	23.25	1754.6	0.9397
6	58.03	4379.7	0.6723
7	76.58	5779.6	0.9030
8	77.00	5811.7	0.8905
9	77.43	5843.8	0.8232
10	108.91	8220.1	0.2004
11	109.29	8248.8	0.0806

No.	Annotation	(ppm)
1	Chloroform-d	77.00

7-(2,3-Dibromopropyl)-1,4-dimethyl-2,3,5,6-tetraoxa-bicyclo[2.2.1]heptane (5)

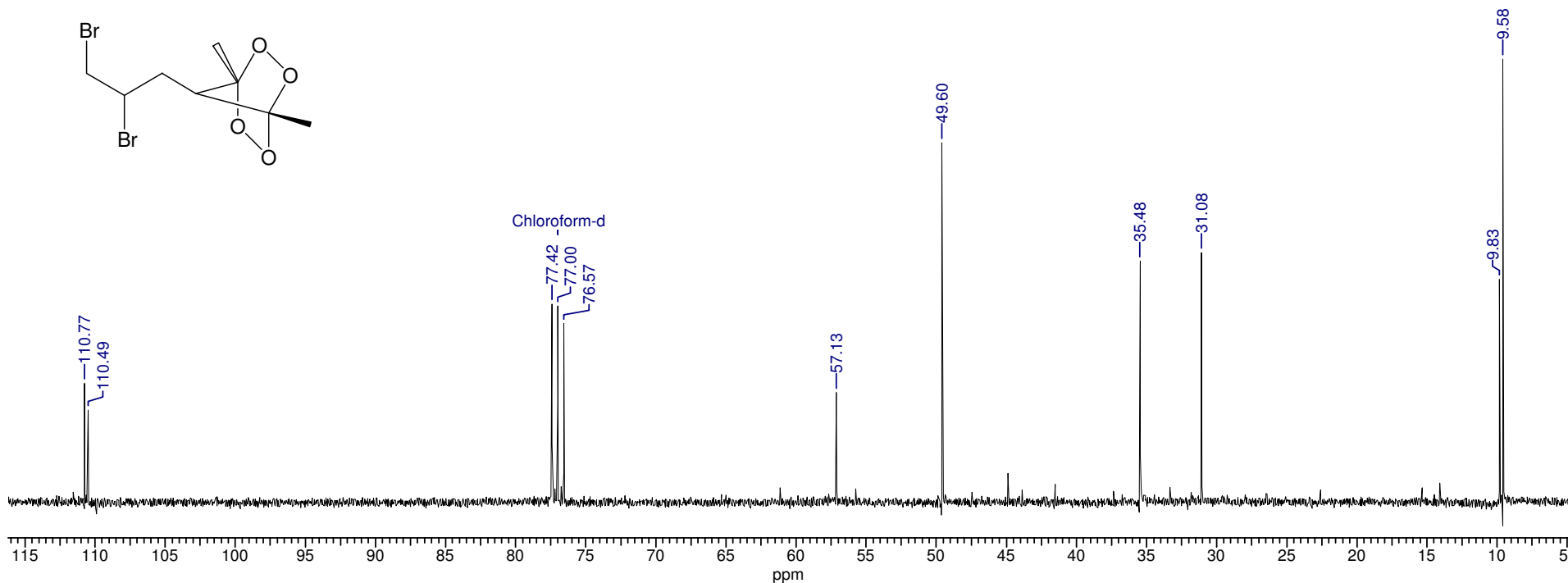
Acquisition Time (sec)	0.6759	Comment	Avance-300, CDCl3	Date	04 Jun 2008 10:01:36
File Name					
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	1
Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	22.800			Sweep Width (Hz)	6009.62



No.	(ppm)	(Hz)	Height	No.	Annotation	(ppm)	No.	(ppm)	Value
1	7.25	2175.9	0.0367	1	Chloroform-d	7.25	1	.52 .. 1.6	6.064
							2	.89 .. 2.0	0.998
							3	.51 .. 2.6	1.000
							4	.92 .. 3.0	1.009
							5	.57 .. 3.6	1.013
							6	.86 .. 3.9	1.019
							7	.13 .. 4.2	1.003

7-(2,3-Dibromopropyl)-1,4-dimethyl-2,3,5,6-tetraoxa-bicyclo[2.2.1]heptane (5)

Acquisition Time (sec)	0.4501	Comment	Avance-300, C-13, CDCl3			Date	04 Jun 2008 11:12:00
File Name							
Frequency (MHz)	75.48	Nucleus	13C	Number of Transients	962	Original Points Count	16308
Points Count	16384	Pulse Sequence	zgpg30base	Solvent	CHLOROFORM-D	Sweep Width (Hz)	18115.94
Temperature (degree C)	22.600						

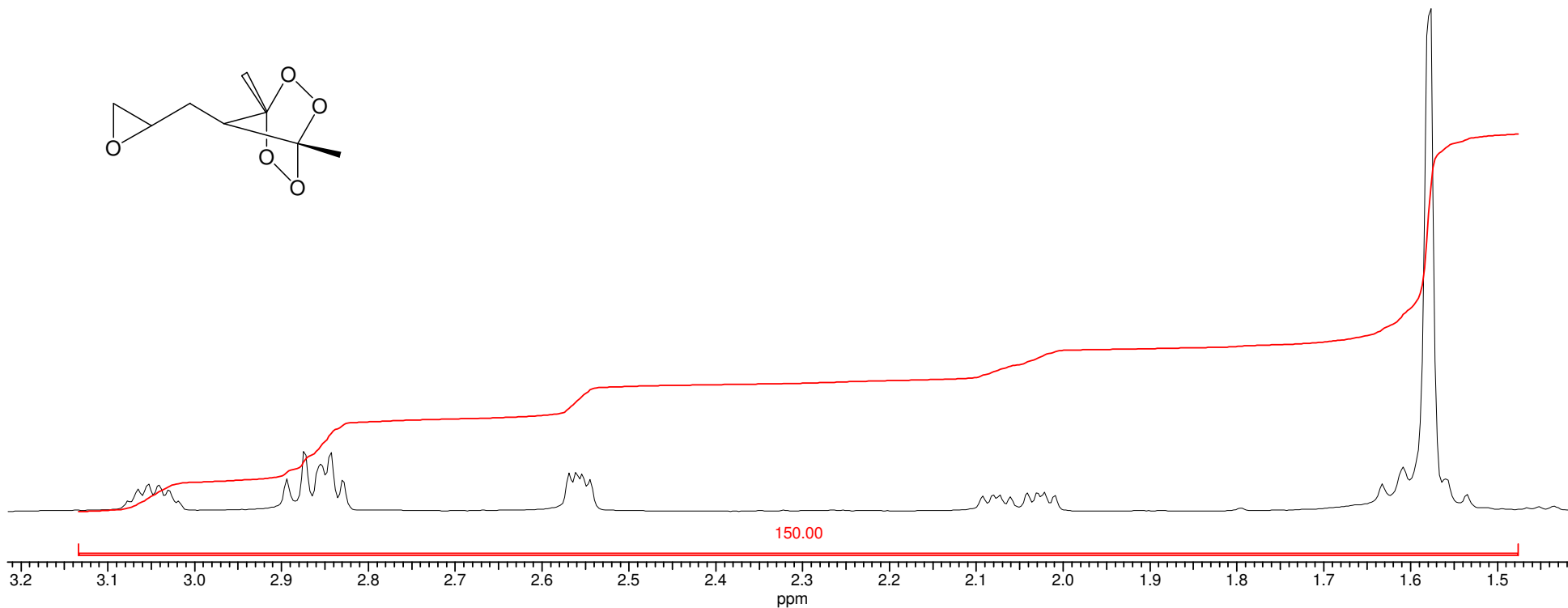
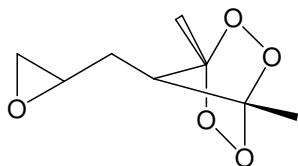


No.	(ppm)	(Hz)	Height
1	9.58	722.9	1.0000
2	9.83	741.6	0.5033
3	31.08	2346.1	0.5636
4	35.48	2677.9	0.5441
5	49.60	3743.8	0.8117
6	57.13	4312.2	0.2479
7	76.57	5779.6	0.4039
8	77.00	5811.6	0.4434
9	77.42	5843.7	0.4477
10	110.49	8339.4	0.2082
11	110.77	8360.5	0.2683

No.	Annotation	(ppm)
1	Chloroform-d	77.00

1,4-Dimethyl-7-(oxiran-2-ylmethyl)-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (6)

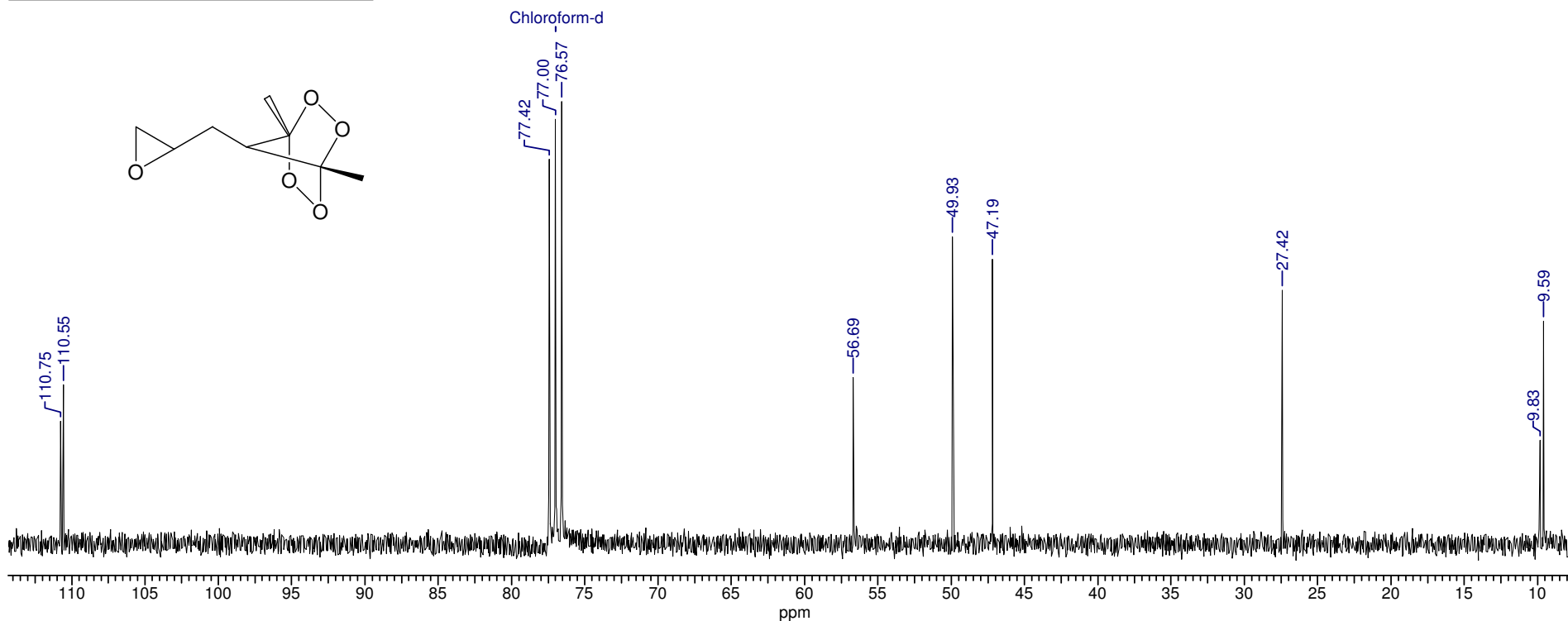
Acquisition Time (sec)	0.6759	Comment	Avance-300, CDCl ₃	Date	02 Sep 2008 10:31:28
File Name					
Frequency (MHz)	300.13	Nucleus	¹ H	Number of Transients	1
Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	21.900			Original Points Count	8124
				Sweep Width (Hz)	6009.62



No.	(ppm)	(Hz)	Height	No.	Annotation	(ppm)	No.	(ppm)	Value
1	7.25	2176.0	0.0306	1	Chloroform-d	7.25	1	.48 .. 3.1	150.000

1,4-Dimethyl-7-(oxiran-2-ylmethyl)-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (6)

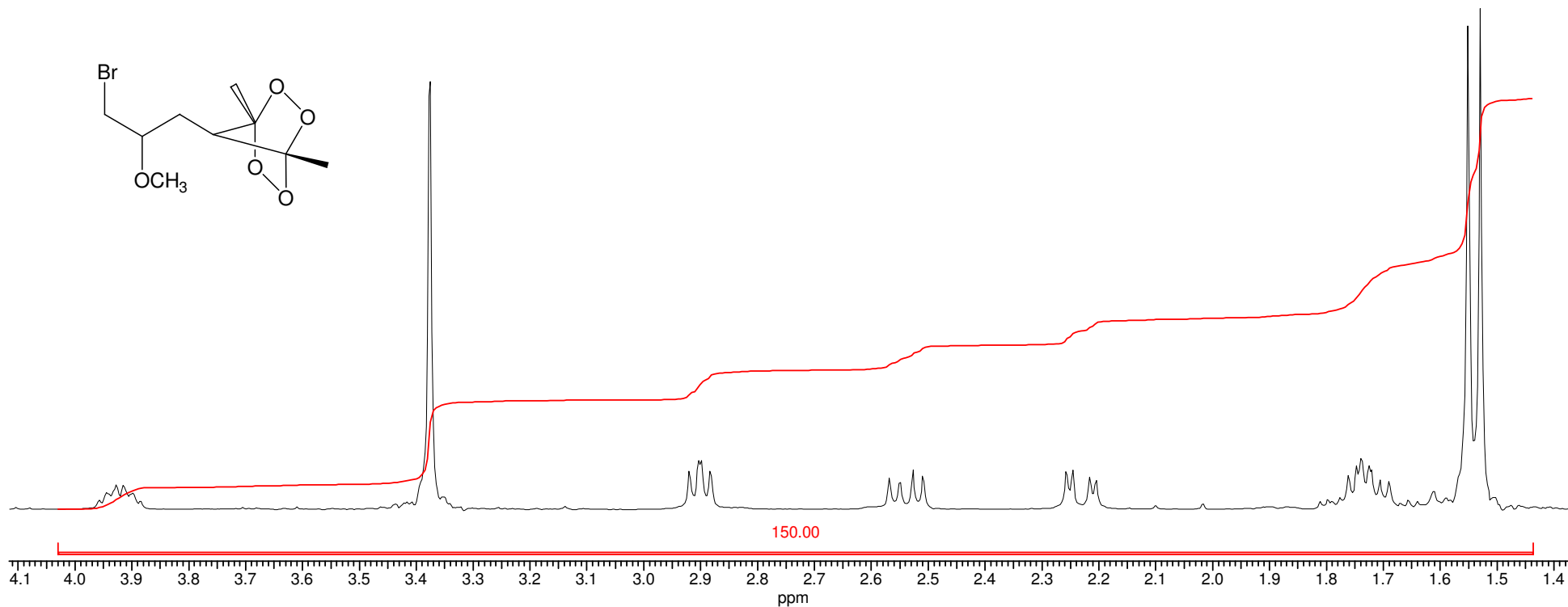
Acquisition Time (sec)	0.4501	Comment	Avance-300, C-13, CDCl3	Date	02 Sep 2008 10:31:28
File Name					
Frequency (MHz)	75.48	Nucleus	13C	Number of Transients	1546
Points Count	16384	Pulse Sequence	zgpg30base	Solvent	CHLOROFORM-D
Temperature (degree C)	22.000			Sweep Width (Hz)	18115.94



No.	Annotation	(ppm)
1	Chloroform-d	77.00

7-(3-Bromo-2-methoxypropyl)-1,4-dimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (7)

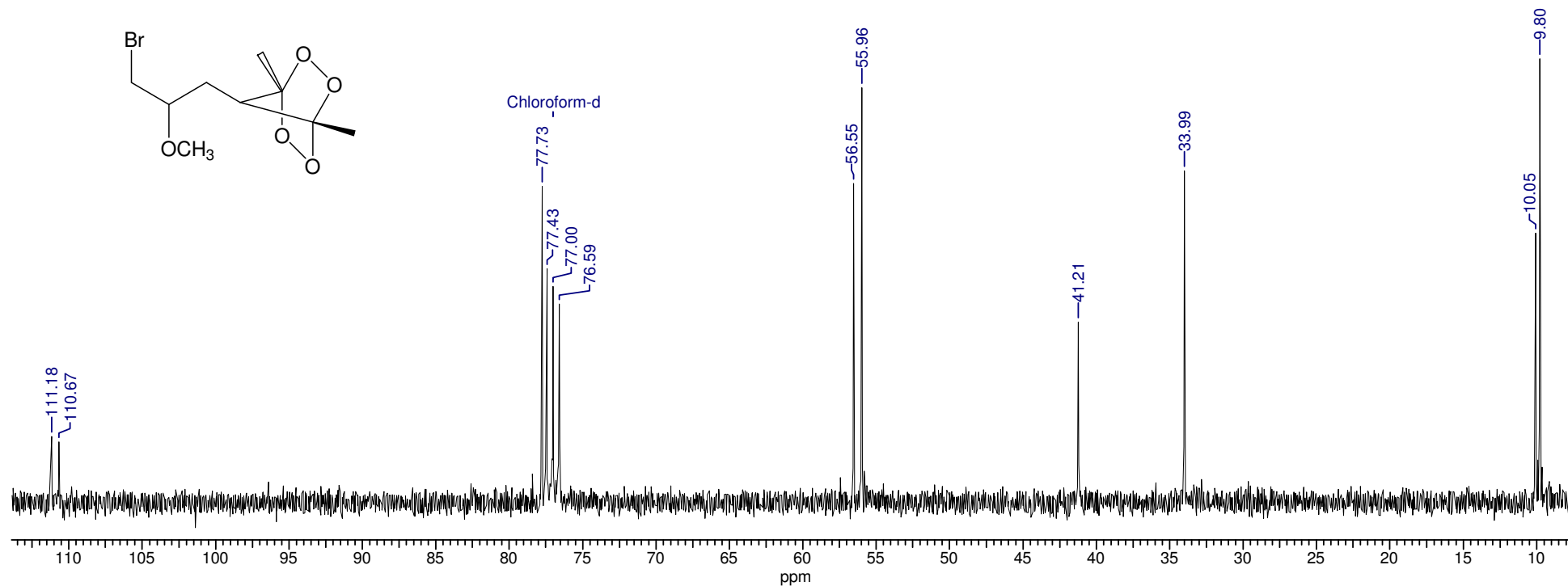
Acquisition Time (sec)	0.6759	Comment	Avance-300, CDCl ₃	Date	03 Dec 2008 11:09:52
File Name					
Frequency (MHz)	300.13	Nucleus	¹ H	Number of Transients	1
Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	23.700			Sweep Width (Hz)	6009.62



No.	(ppm)	(Hz)	Height	No.	Annotation	(ppm)	No.	(ppm)	Value
1	7.25	2175.9	0.0189	1	Chloroform-d	7.25	1	.44 .. 4.0	150.000

7-(3-Bromo-2-methoxypropyl)-1,4-dimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (7)

Acquisition Time (sec)	0.4503	Comment	Avance-300, 13C	Date	05 Sep 2008 10:52:48
File Name					
Frequency (MHz)	75.48	Nucleus	13C	Number of Transients	698
Points Count	16384	Pulse Sequence	zgpg30base	Solvent	CHLOROFORM-D
Temperature (degree C)	30.000			Sweep Width (Hz)	18115.94

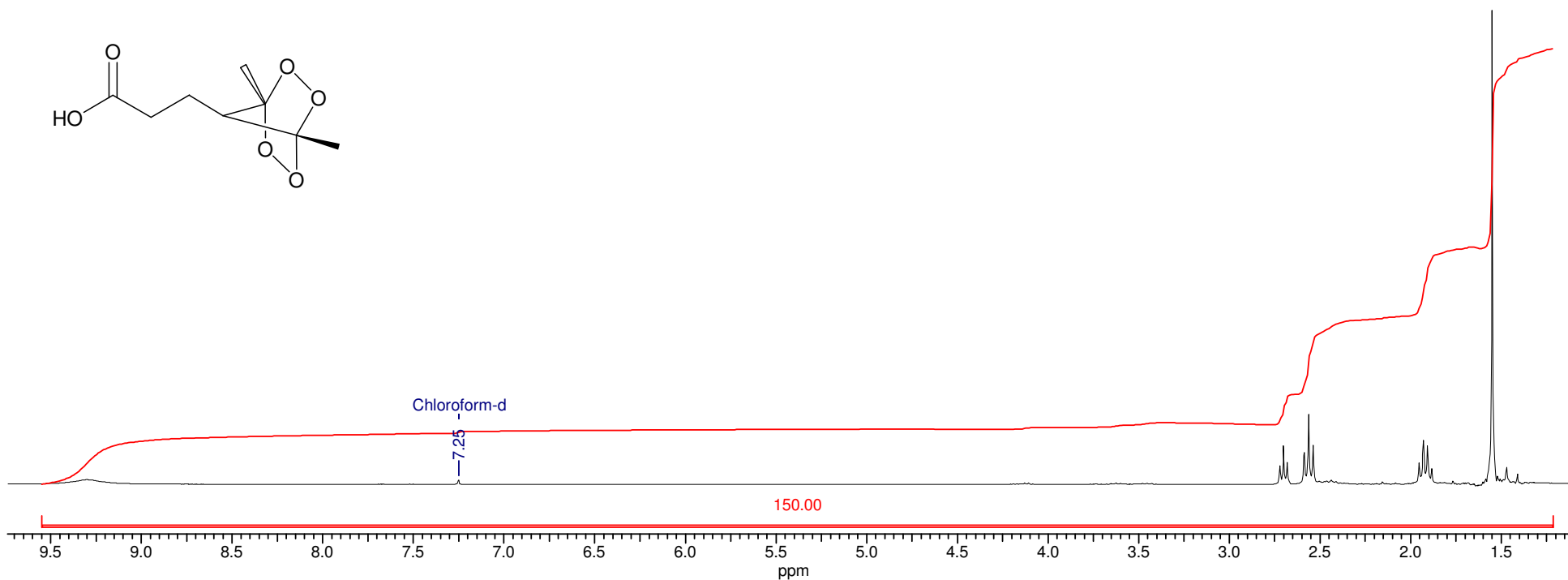
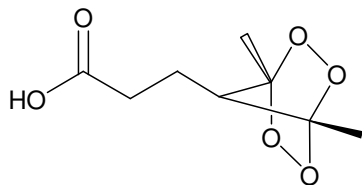


No.	(ppm)	(Hz)	Height	No.	(ppm)	(Hz)	Height
1	9.80	739.5	1.0000	7	76.59	5780.7	0.4477
2	10.05	758.3	0.6068	8	77.00	5811.7	0.4868
3	33.99	2565.1	0.7481	9	77.43	5843.7	0.5268
4	41.21	3110.3	0.4064	10	77.73	5867.0	0.7127
5	55.96	4223.8	0.9343	11	110.67	8352.7	0.1368
6	56.55	4268.0	0.7192	12	111.18	8391.4	0.1494

No.	Annotation	(ppm)
1	Chloroform-d	77.00

3-(1,4-Dimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]hept-7-yl)propanoic acid (8)

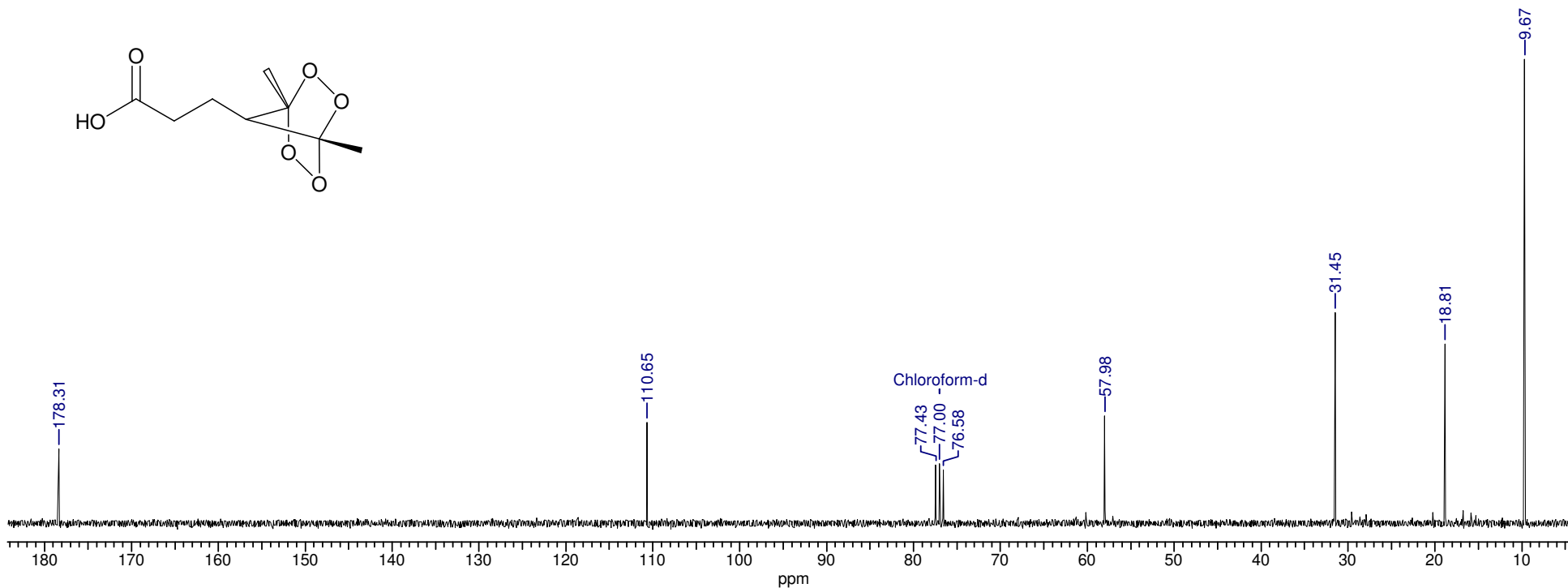
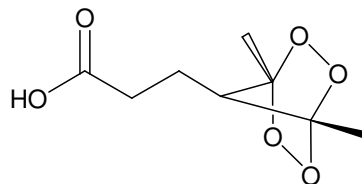
Acquisition Time (sec)	0.6759	Comment Avance-300, CDCl ₃		Date	11 Jul 2008 12:22:24
File Name				Frequency (MHz)	300.13
Nucleus	¹ H	Number of Transients	1	Original Points Count	8124
Pulse Sequence	zg	Solvent	CHLOROFORM-D	Points Count	8192
Temperature (degree C)	35.000			Sweep Width (Hz)	6009.62



No.	(ppm)	(Hz)	Height	No.	Annotation	(ppm)	No.	(ppm)	Value
1	7.25	2176.0	0.0092	1	Chloroform-d	7.25	1	.21 .. 9.5	150.000

3-(1,4-Dimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]hept-7-yl)propanoic acid (8)

Acquisition Time (sec)	0.4501	Comment	Avance-300, C-13, CDCl3		Date	11 Jul 2008 12:41:36	
File Name							
Frequency (MHz)	75.48	Nucleus	13C	Number of Transients	158	Original Points Count	16308
Points Count	16384	Pulse Sequence	zgpg30base	Solvent	CHLOROFORM-D	Sweep Width (Hz)	18115.94
Temperature (degree C)	35.000						

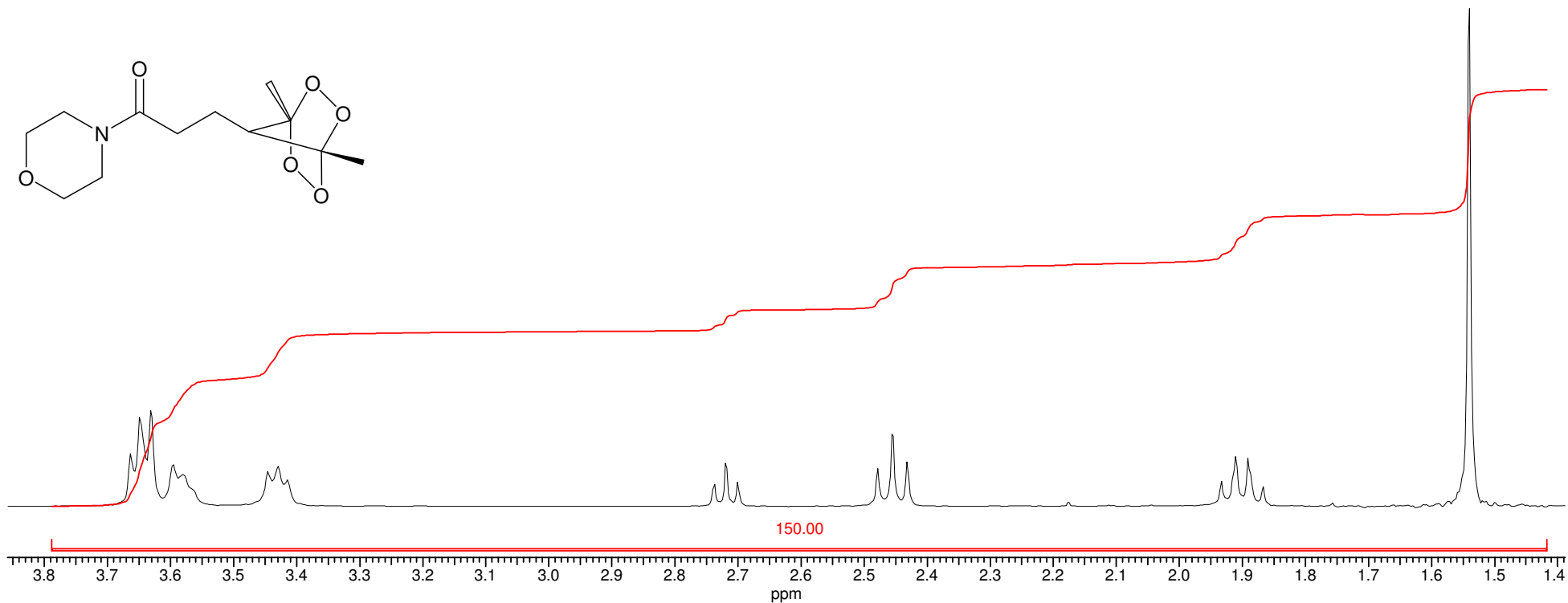
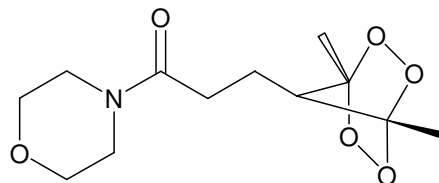


No.	Annotation	(ppm)
1	Chloroform-d	77.00

No.	(ppm)	(Hz)	Height
1	9.67	729.5	1.0000
2	18.81	1419.5	0.3869
3	31.45	2373.8	0.4547
4	57.98	4376.4	0.2311
5	76.58	5779.6	0.1159
6	77.00	5811.7	0.1292
7	77.43	5843.7	0.1258
8	110.65	8351.6	0.2178
9	178.31	13458.1	0.1614

4-[3-(1,4-Dimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]hept-7-yl)propanoyl]morpholine (9)

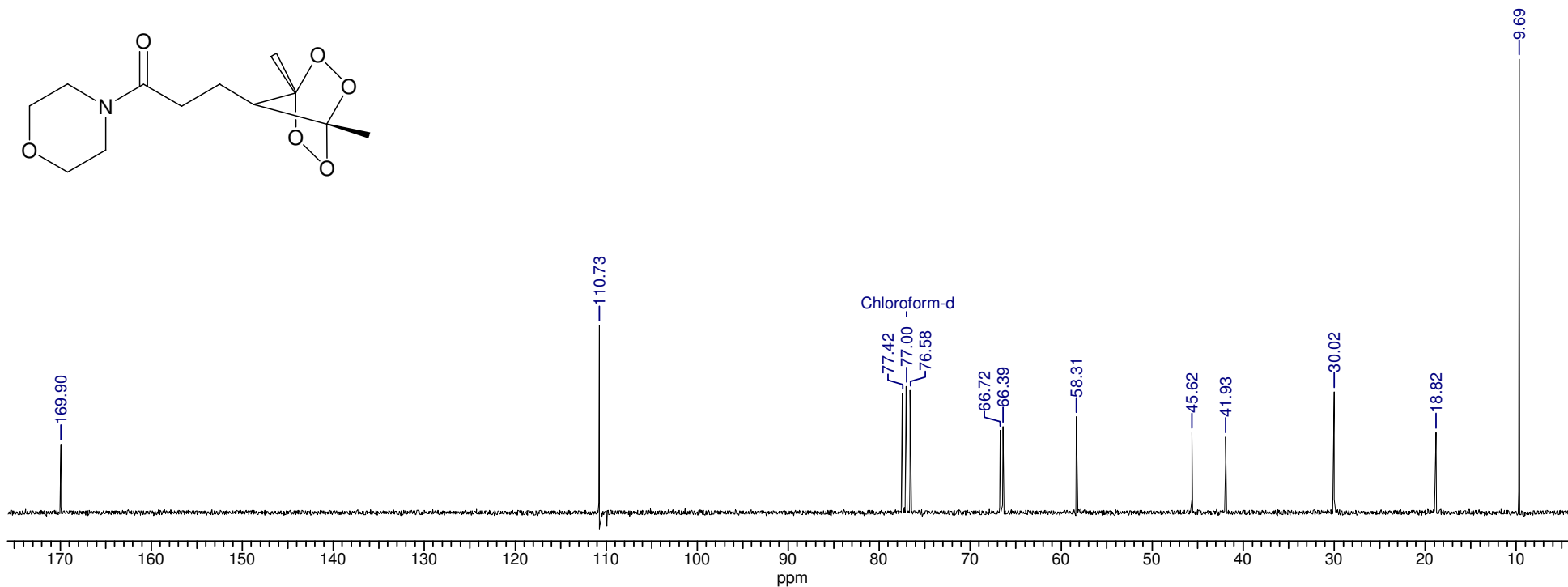
Acquisition Time (sec)	0.6759	Comment	Avance-300, CDCl ₃	Date	15 Jul 2008 11:39:44
File Name					
Frequency (MHz)	300.13	Nucleus	¹ H	Number of Transients	1
Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	25.200			Sweep Width (Hz)	6009.62



No.	(ppm)	(Hz)	Height	No.	Annotation	(ppm)	No.	(ppm)	Value
1	7.25	2175.9	0.0247	1	Chloroform-d	7.25	1	.42 .. 3.7	150.000

4-[3-(1,4-Dimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]hept-7-yl)propanoyl]morpholine (9)

Acquisition Time (sec)	0.4501	Comment	Avance-300, C-13, CDCl3			Date	15 Jul 2008 11:41:52
File Name							
Frequency (MHz)	75.48	Nucleus	13C	Number of Transients	1021	Original Points Count	16308
Points Count	16384	Pulse Sequence	zgpg30base	Solvent	CHLOROFORM-D	Sweep Width (Hz)	18115.94
Temperature (degree C)	25.200						



No.	(ppm)	(Hz)	Height	No.	(ppm)	(Hz)	Height	No.	Annotation	(ppm)
1	9.69	731.7	1.0000	8	66.72	5035.4	0.1802	1	Chloroform-d	77.00
2	18.82	1420.6	0.1770	9	76.58	5779.6	0.2695			
3	30.02	2265.4	0.2664	10	77.00	5811.6	0.2781			
4	41.93	3164.4	0.1662	11	77.42	5843.7	0.2629			
5	45.62	3443.1	0.1768	12	110.73	8357.1	0.4125			
6	58.31	4400.7	0.2114	13	169.90	12823.4	0.1508			
7	66.39	5011.1	0.1890							