SUPPORTING INFORMATION

Phosphomolybdic and phosphotungstic acids as efficient catalysts for the synthesis of bridged 1,2,4,5-tetraoxanes from β-diketones and hydrogen peroxide

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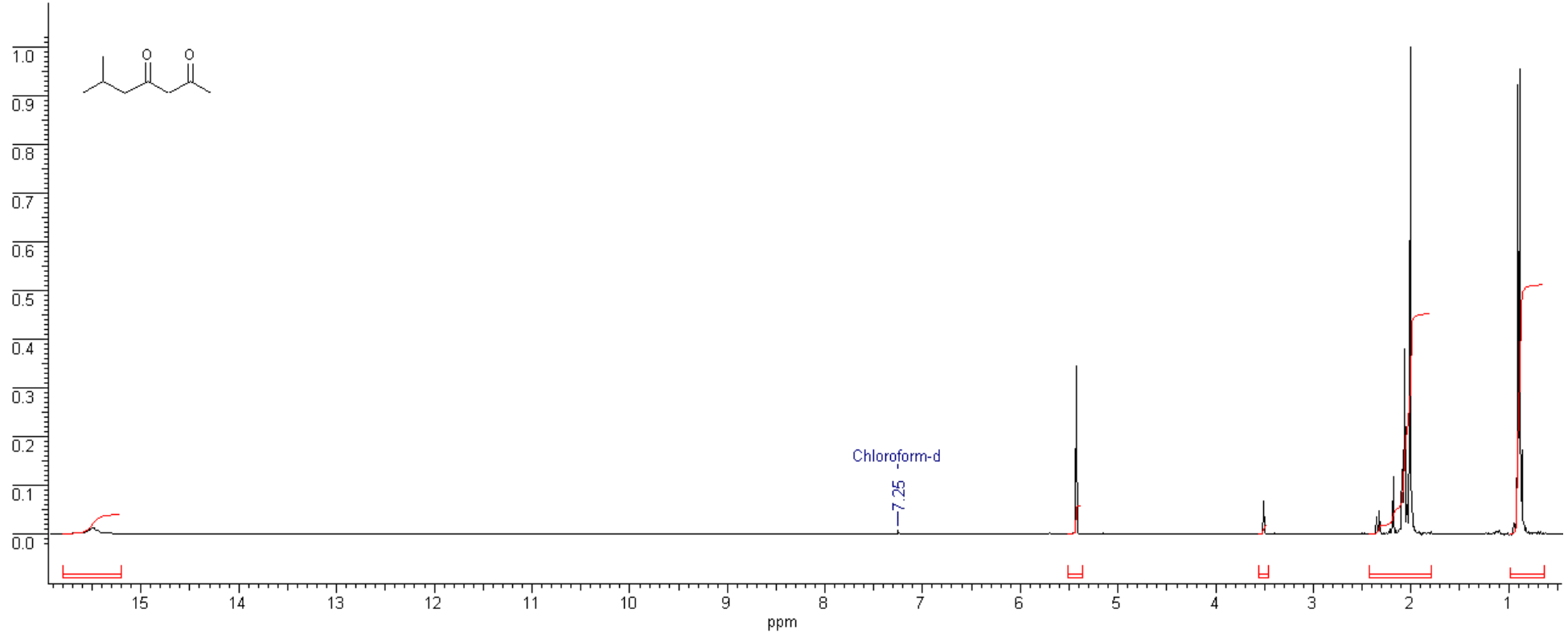
NMR spectra of β-diketones

6-Methylheptane-2,4-dione (1b)

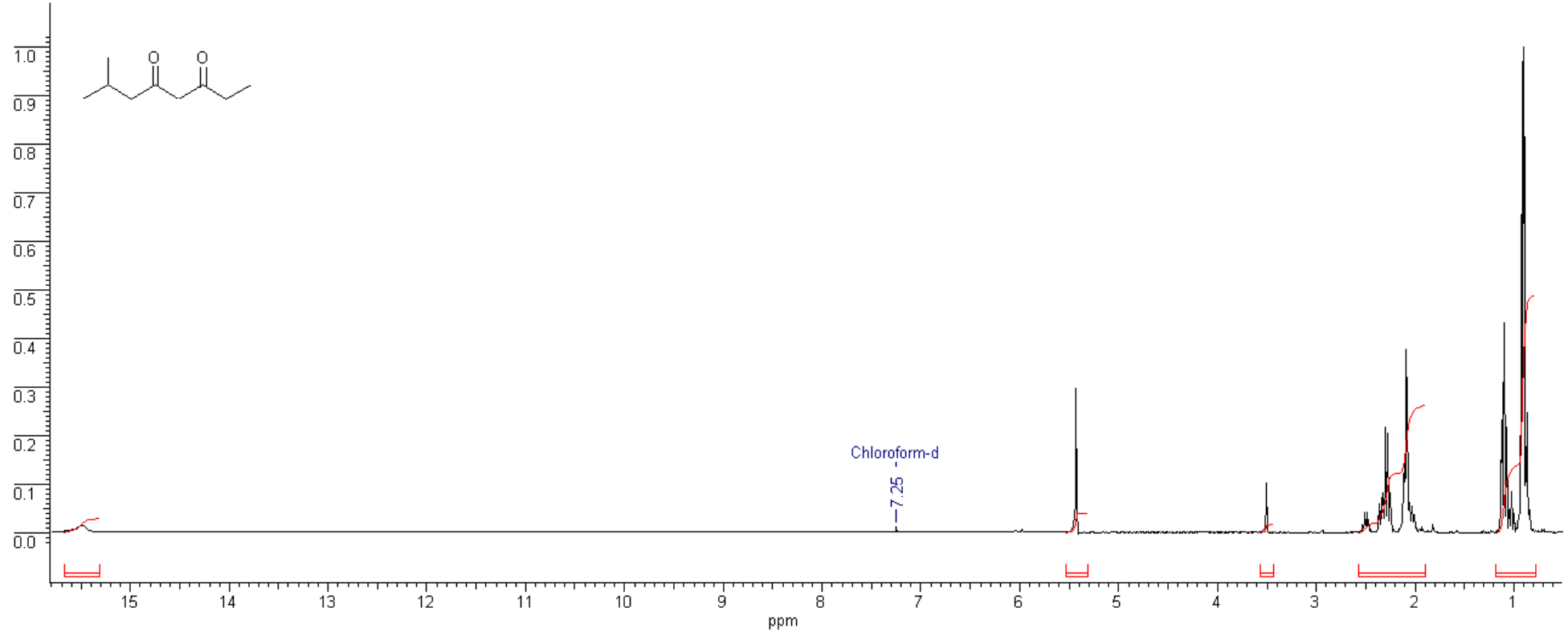
Acquisition Time (sec)	0.6759	***Comment***	NMR/50059953	***Date***	13 Mar 2012 12:41:36		
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	27.000						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

7-Methyloctane-3,5-dione (1c)

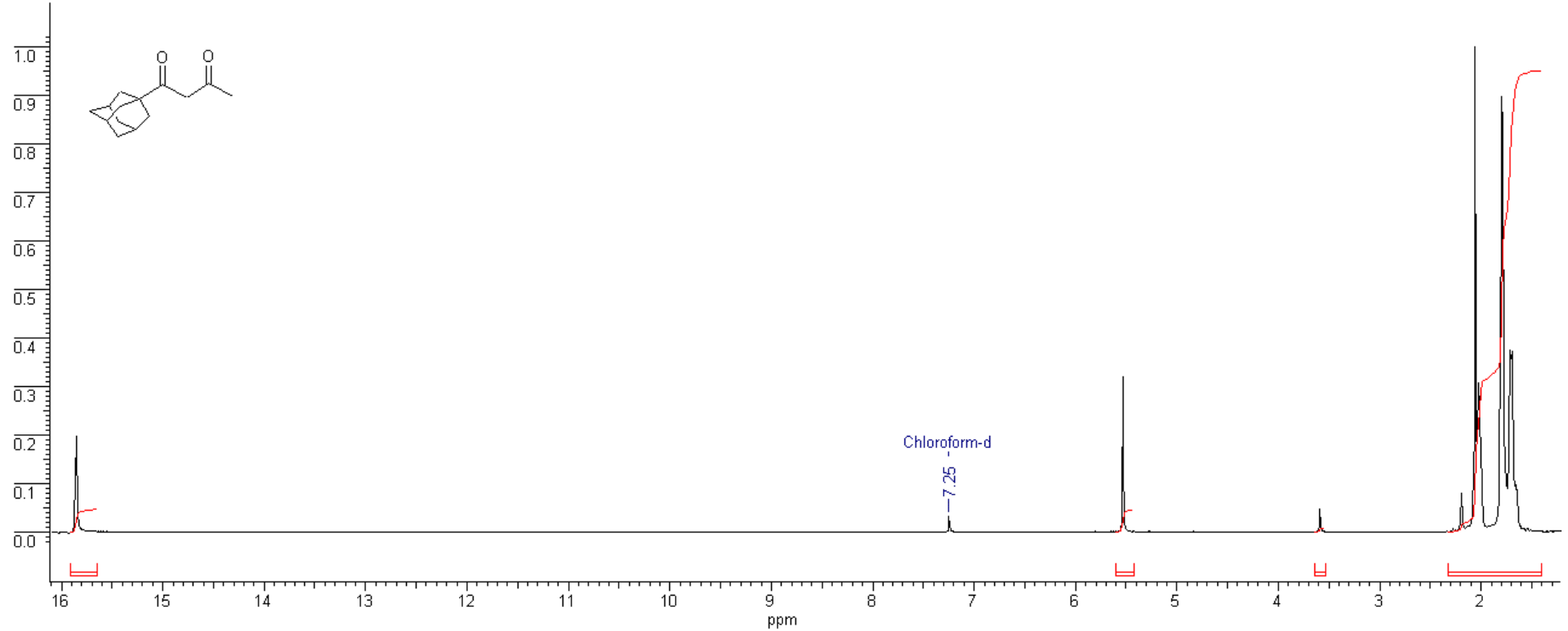
Acquisition Time (sec)	0.6759	***Comment***	NMR/50059953	***Date***	27 Mar 2012 12:16:00		
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	25.400						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

1-(1-Adamantyl)butane-1,3-dione (1e)

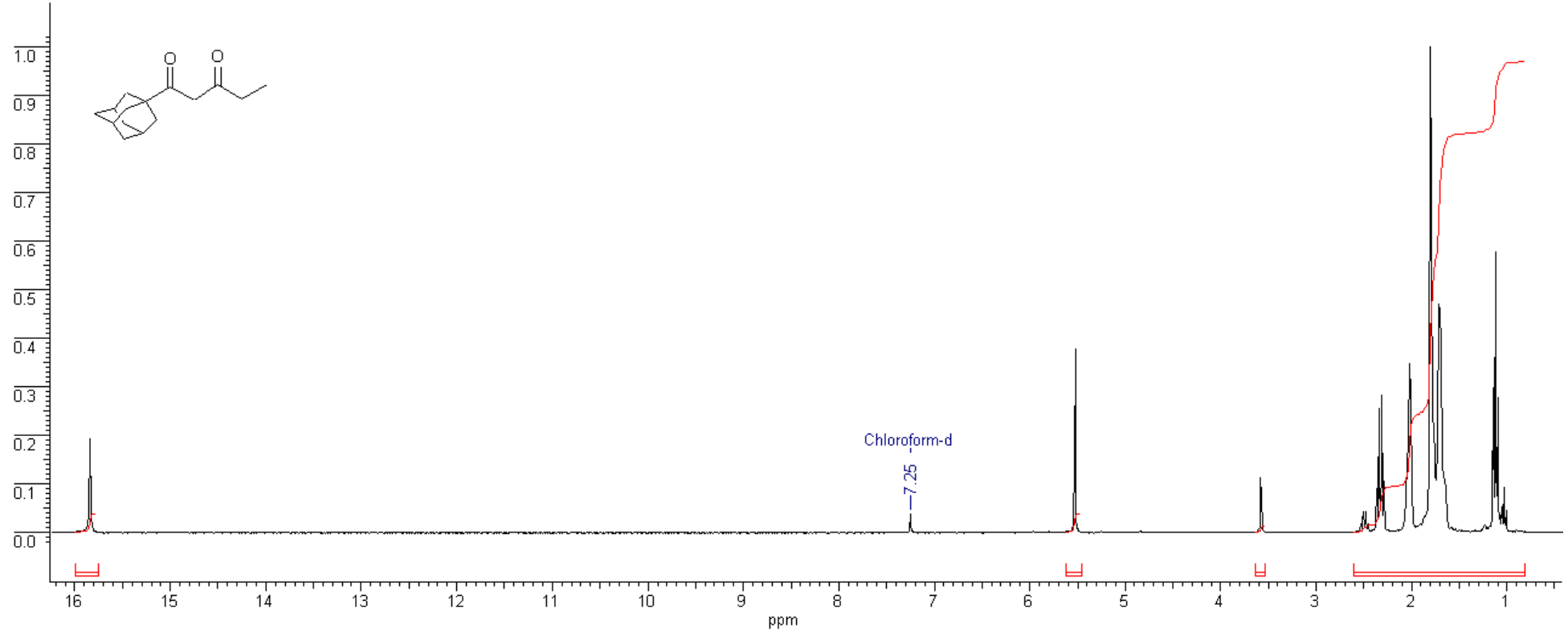
Acquisition Time (sec)	0.6759	***Comment***	/VMIG S520	***Date***	24 Nov 2011 14:21:52		
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	26.800						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

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

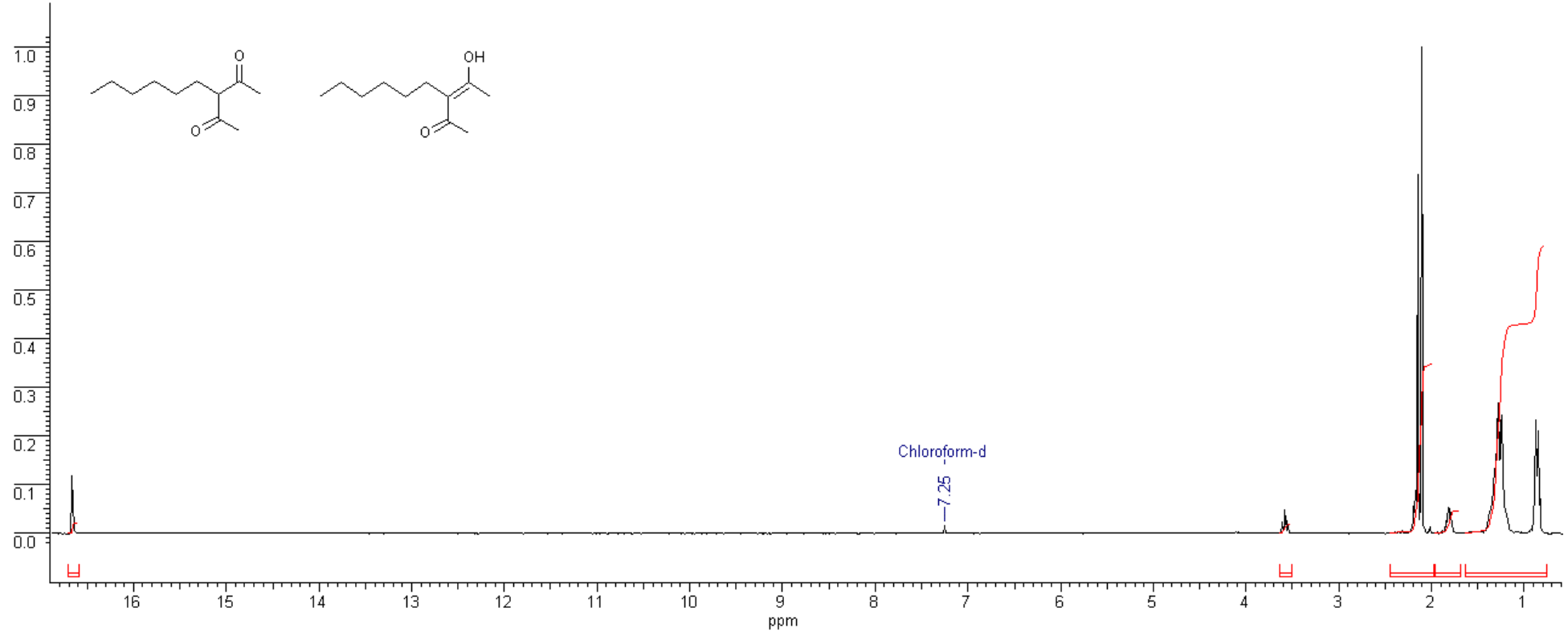
Acquisition Time (sec)	0.6759	*Comment*	/VMIG S520	*Date*	10 Jan 2012 14:36:48		
File Name						*Frequency (MHz)*	300.13
Nucleus	1H	*Number of Transients*	1	*Original Points Count*	8124	*Points Count*	8192
Pulse Sequence	zg	*Solvent*	CHLOROFORM-D			*Sweep Width (Hz)*	6009.62
Temperature (degree C)	27.000						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

3-Hexyl-2,4-pentanedione (1g)

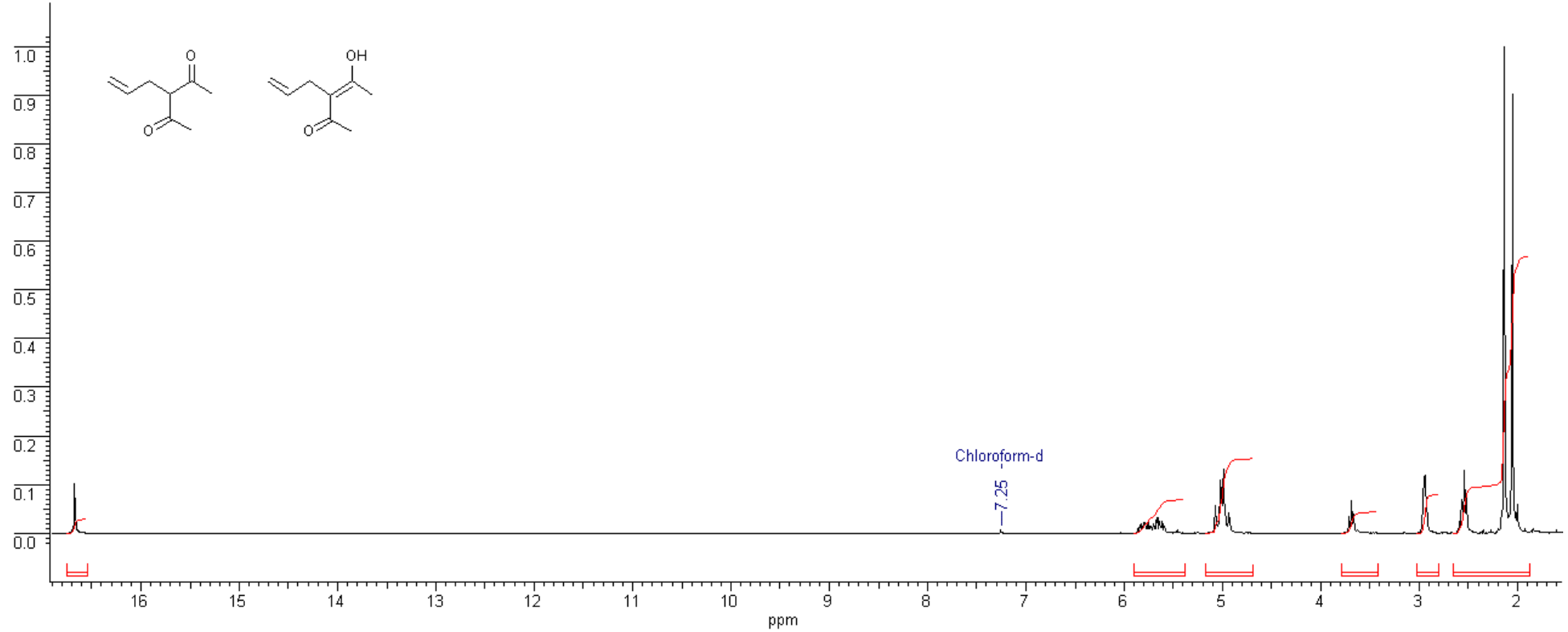
Acquisition Time (sec)	0.6759	***Comment***	NMR/50059953	***Date***	14 May 2012 09:12:32		
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	25.300						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

3-Allylpentane-2,4-dione (1h)

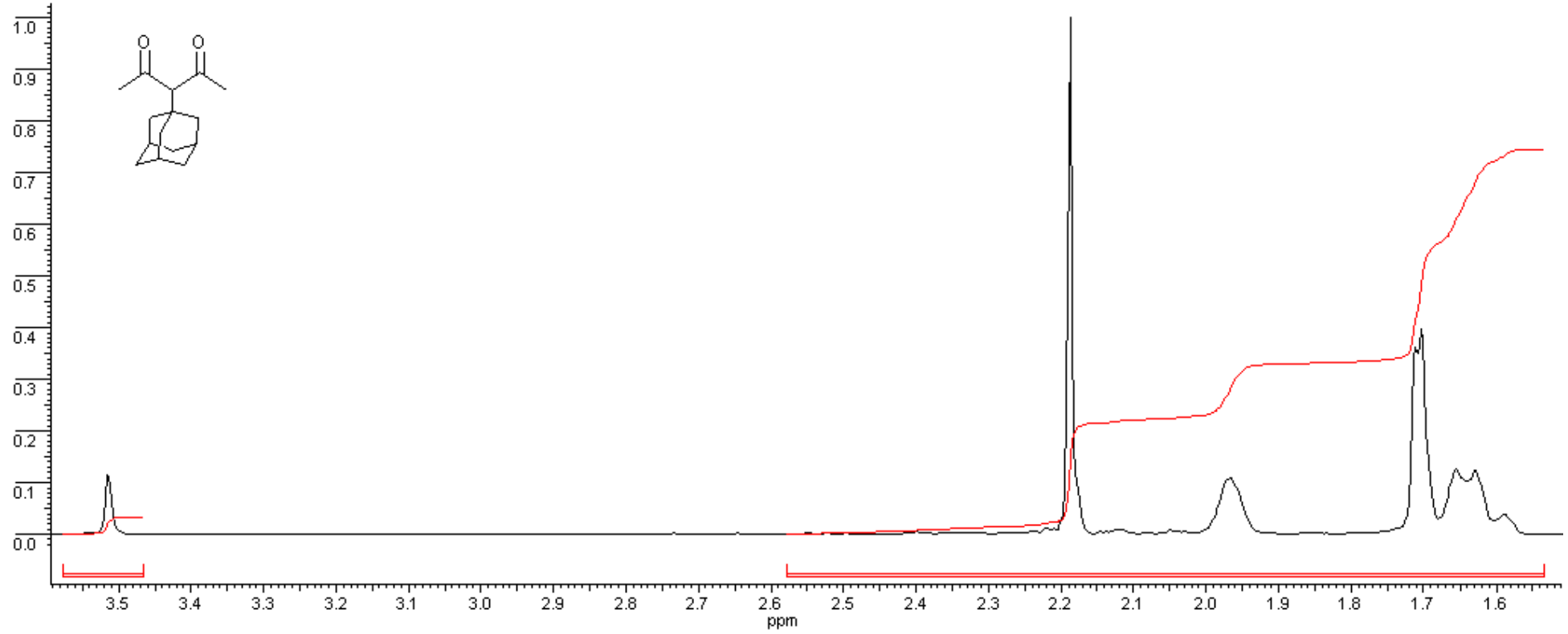
Acquisition Time (sec)	0.6759	***Comment***	Avance-300, CDCl3			***Date***	08 Jun 2011 09:33:52
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	27.000						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

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

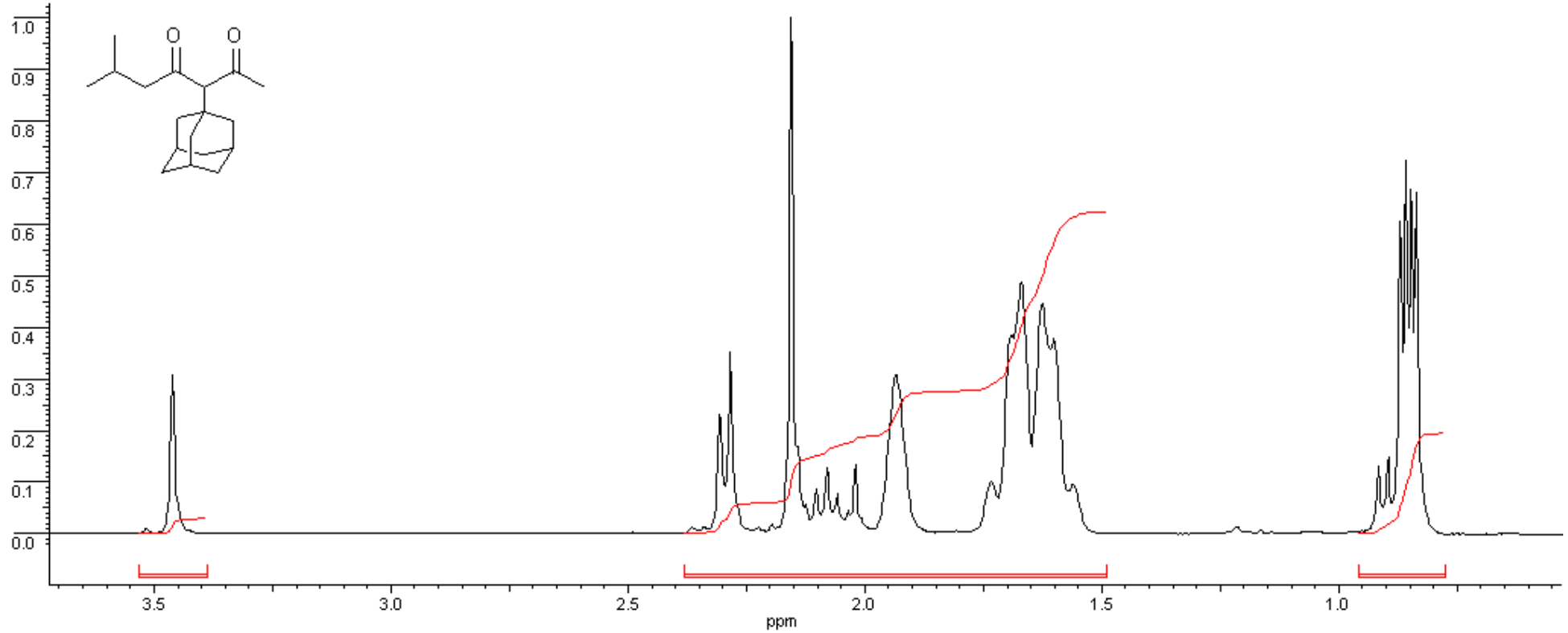
Acquisition Time (sec)	0.6759	***Comment***	NMR/50059953	***Date***	01 Feb 2012 10:50:40		
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	24.700						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

3-(1-Adamantyl)-6-methylheptane-2,4-dione (1j)

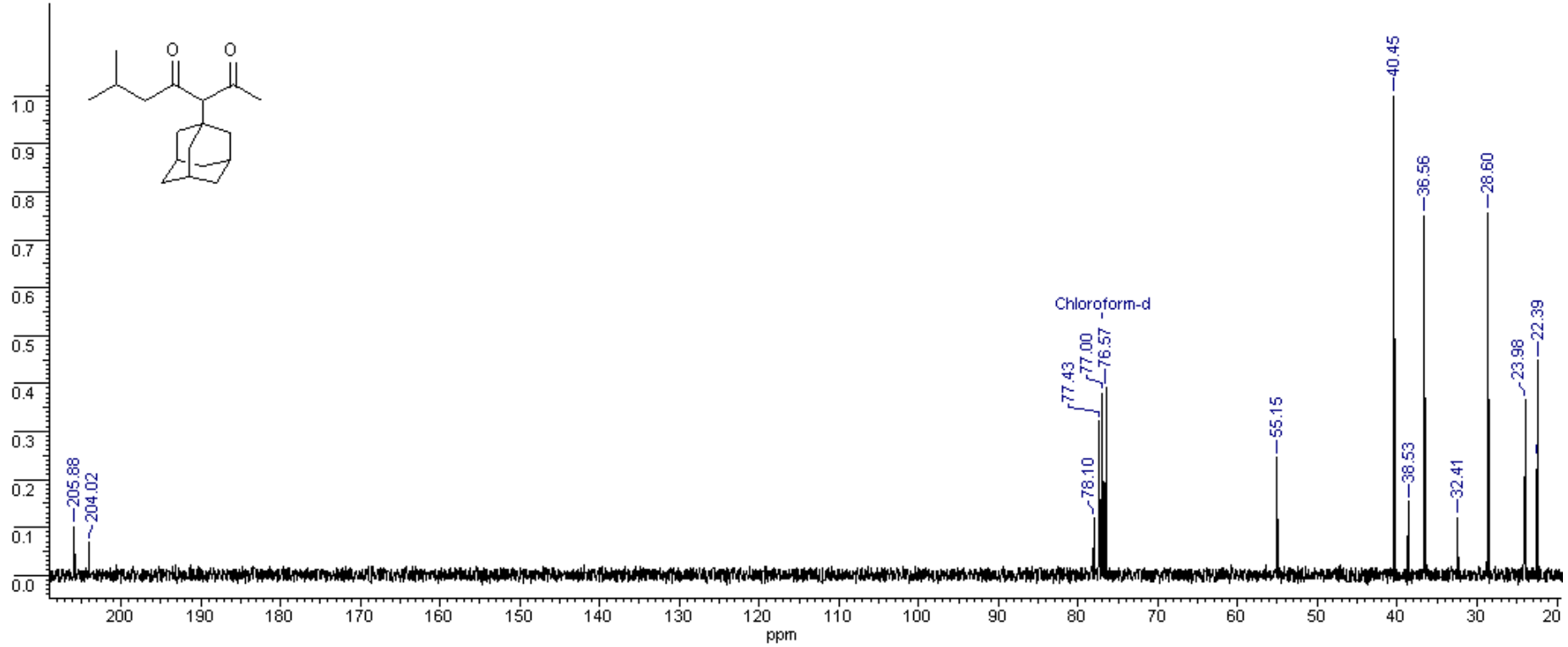
Acquisition Time (sec)	0.6759	***Comment***	NMR/50059953	***Date***	10 May 2012 09:57:20		
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	24.900						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

3-(1-Adamantyl)-6-methylheptane-2,4-dione (1j)

Acquisition Time (sec)	0.4322	***Comment***	Avance-300, C-13, CDCl3			***Date***	04 May 2012 16:04:16
File Name						***Frequency (MHz)***	75.48
Nucleus	13C	***Number of Transients***	125	***Original Points Count***	16308	***Points Count***	16384
Pulse Sequence	zgpg60base	***Solvent***	CHLOROFORM-D	***Sweep Width (Hz)***	18867.92	***Temperature (degree C)***	27.600



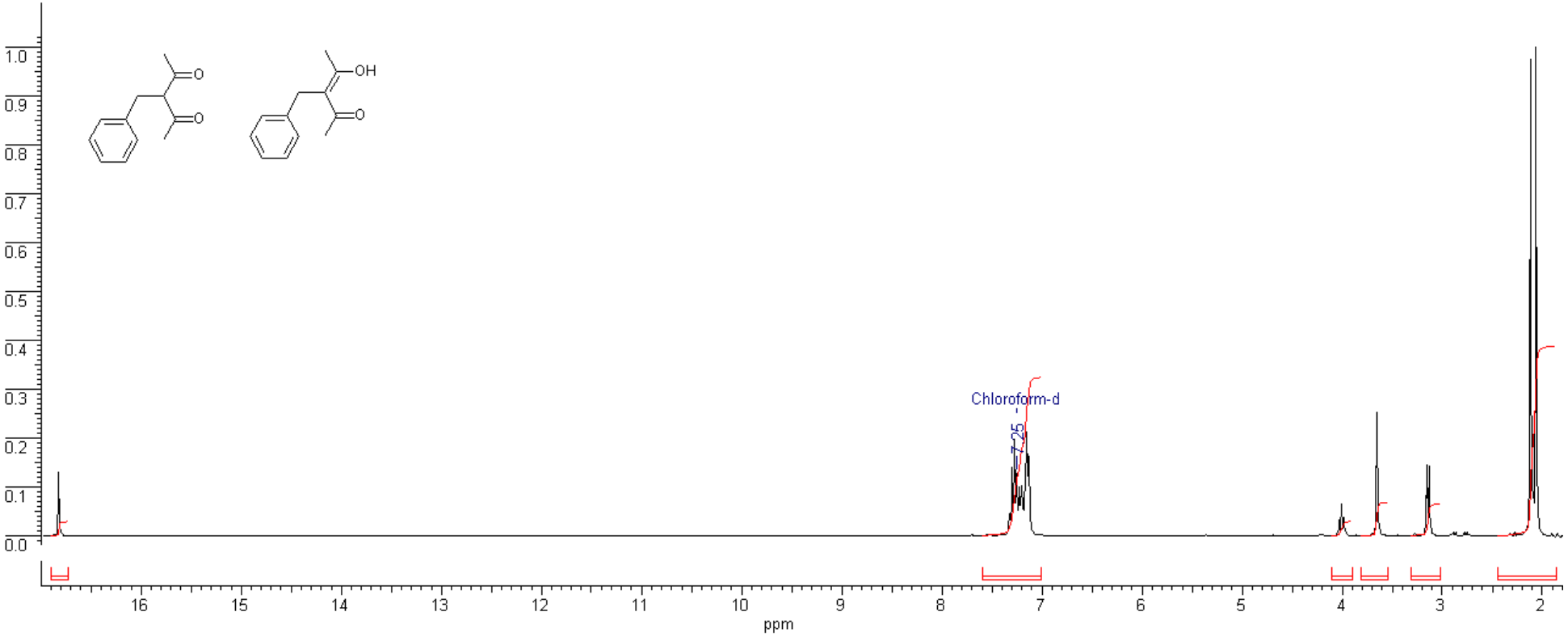
No.	(ppm)	(Hz)	Height
1	22.39	1689.8	0.4491
2	22.46	1695.5	0.2440
3	23.98	1809.6	0.3651
4	28.60	2158.5	0.7563
5	32.41	2446.4	0.1208
6	36.56	2759.7	0.7505
7	38.53	2908.3	0.1529
8	40.45	3053.4	1.0000

No.	(ppm)	(Hz)	Height
9	55.15	4162.4	0.2453
10	76.57	5779.4	0.3915
11	77.00	5811.6	0.3797
12	77.43	5843.9	0.3202
13	78.10	5894.6	0.1189
14	204.02	15398.2	0.0681
15	205.88	15538.7	0.1008

No.	Annotation	(ppm)
1	Chloroform-d	77.00

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

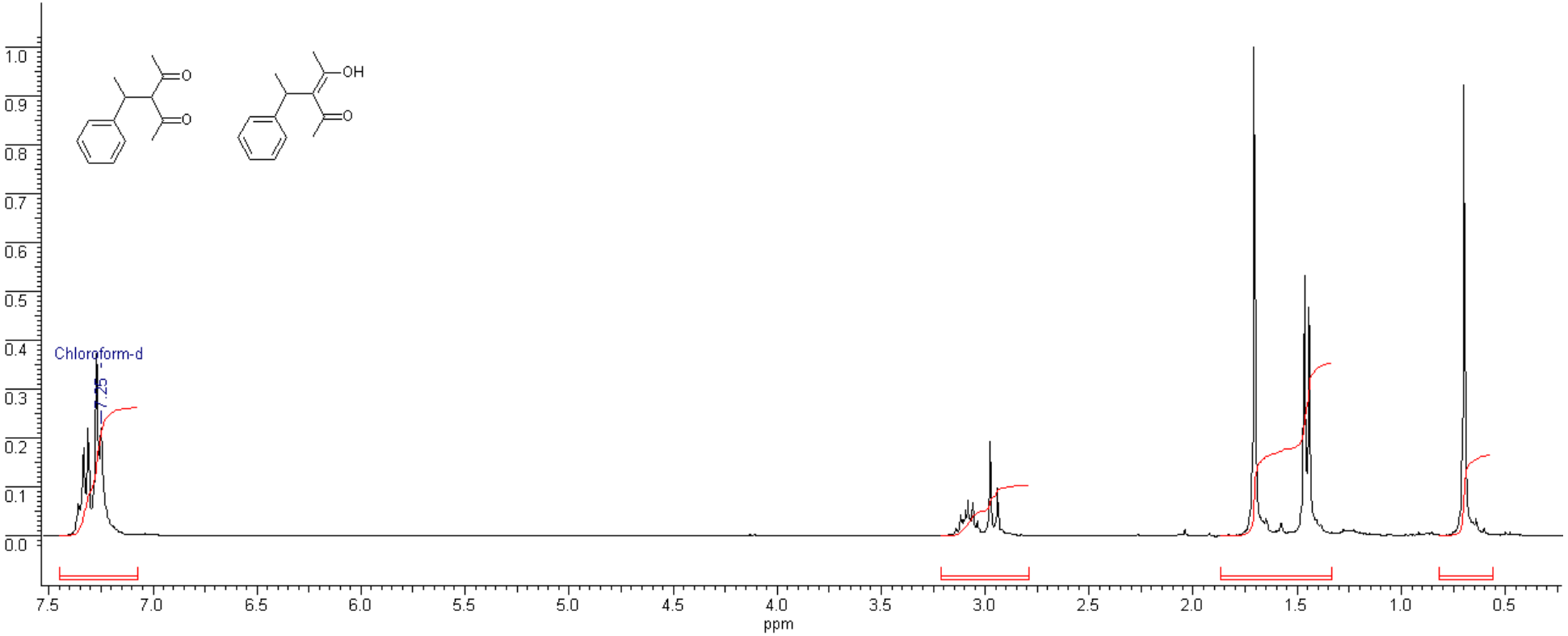
Acquisition Time (sec)	0.6759	***Comment***	Avance-300, CDCl3			***Date***	18 May 2011 12:43:44
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	22.500						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

3-(1-Phenylethyl)pentane-2,4-dione (1l)

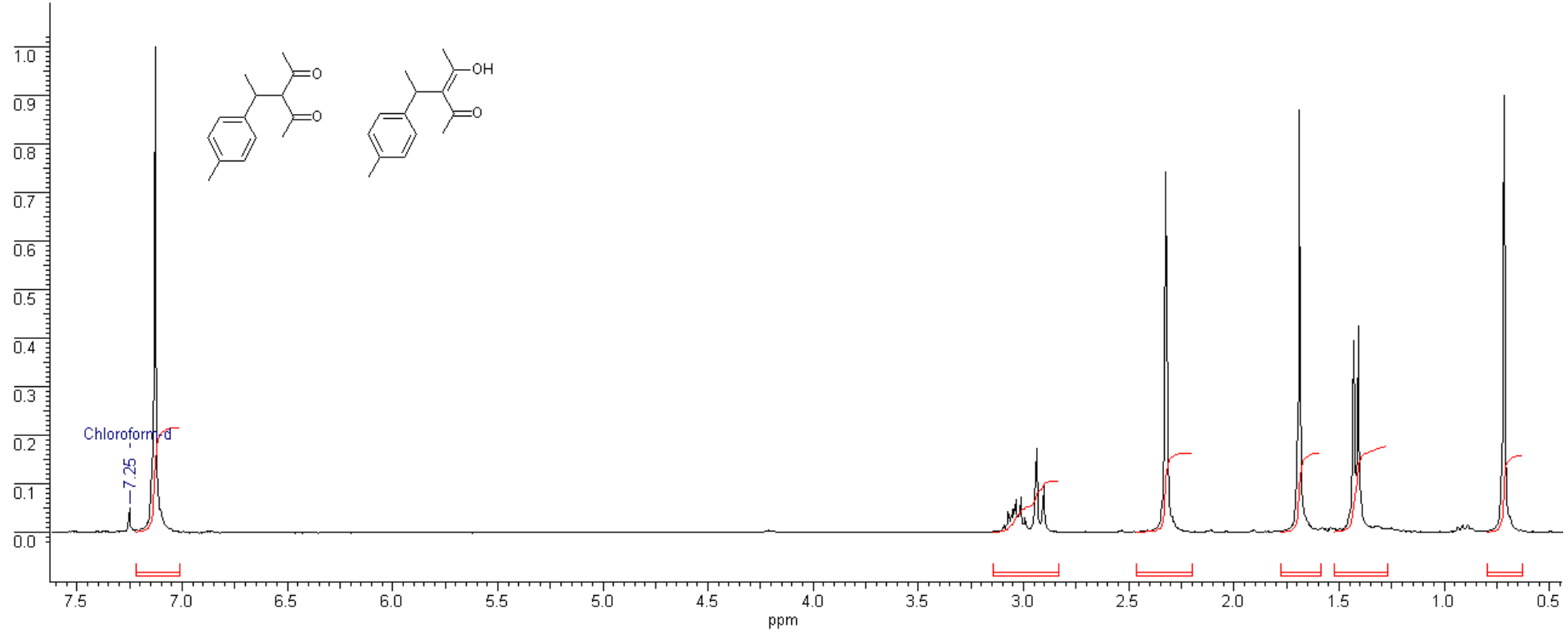
Acquisition Time (sec)	0.5416	***Comment***	Avance-300, CDCl3			***Date***	03 Jun 2011 14:36:48
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	7500.00
Temperature (degree C)	25.300						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

3-[1-(4-Methylphenyl)ethyl]pentane-2,4-dione (1m)

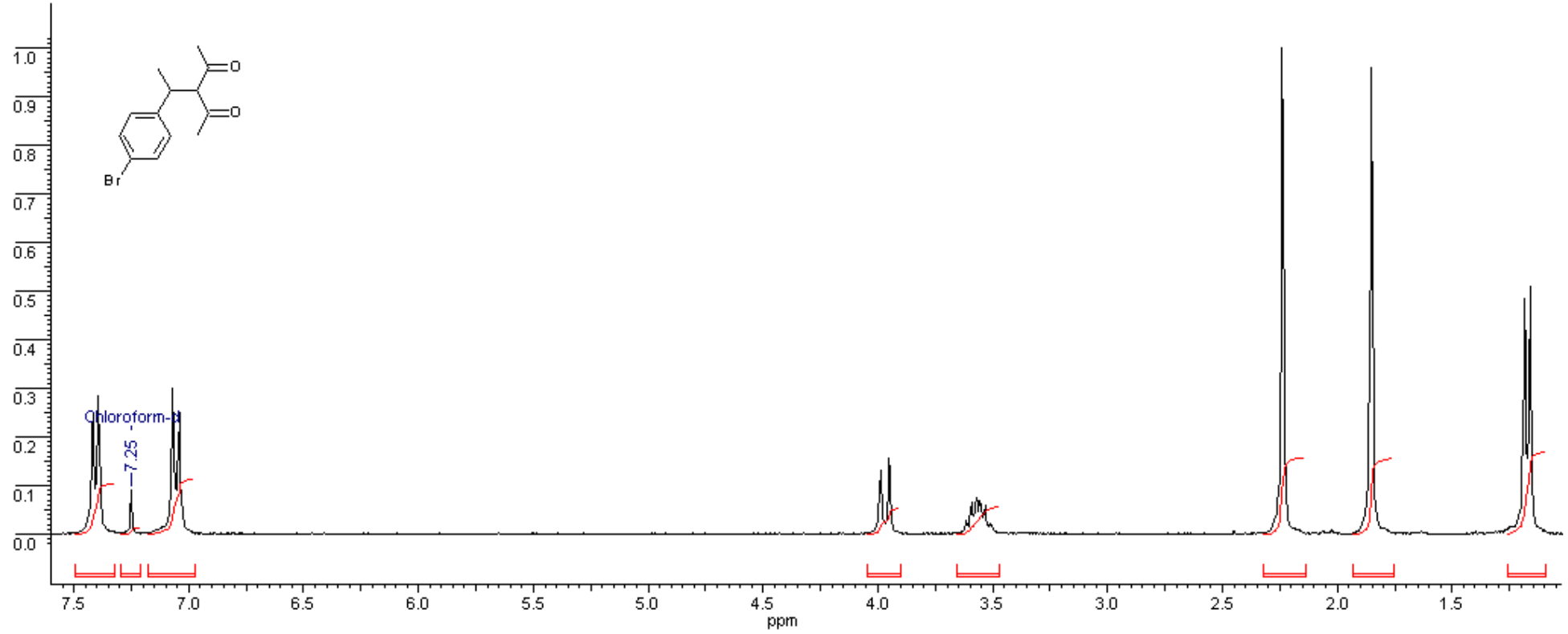
Acquisition Time (sec)	0.6759	***Comment***	Avance-300, CDCl3			***Date***	21 Jun 2011 09:55:12
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	25.700						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

3-[1-(4-Bromophenyl)ethyl]pentane-2,4-dione (1n)

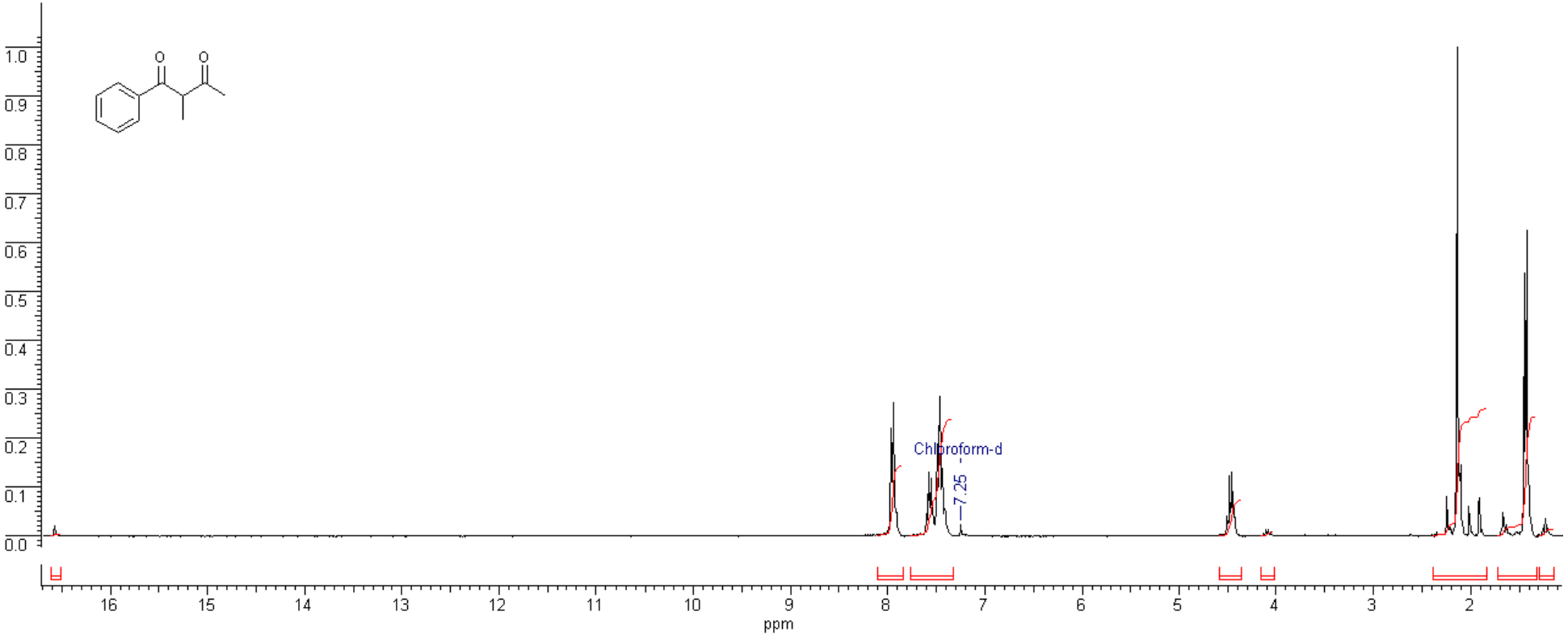
Acquisition Time (sec)	0.6759	***Comment***	Avance-300, CDCl3	***Date***	14 Jun 2011 14:49:36
File Name				***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count*** 8124	***Points Count*** 8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D		***Sweep Width (Hz)*** 6009.62
Temperature (degree C)	34.900				



No.	Annotation	(ppm)
1	Chloroform-d	7.25

2-Methyl-1-phenylbutane-1,3-dione (1o)

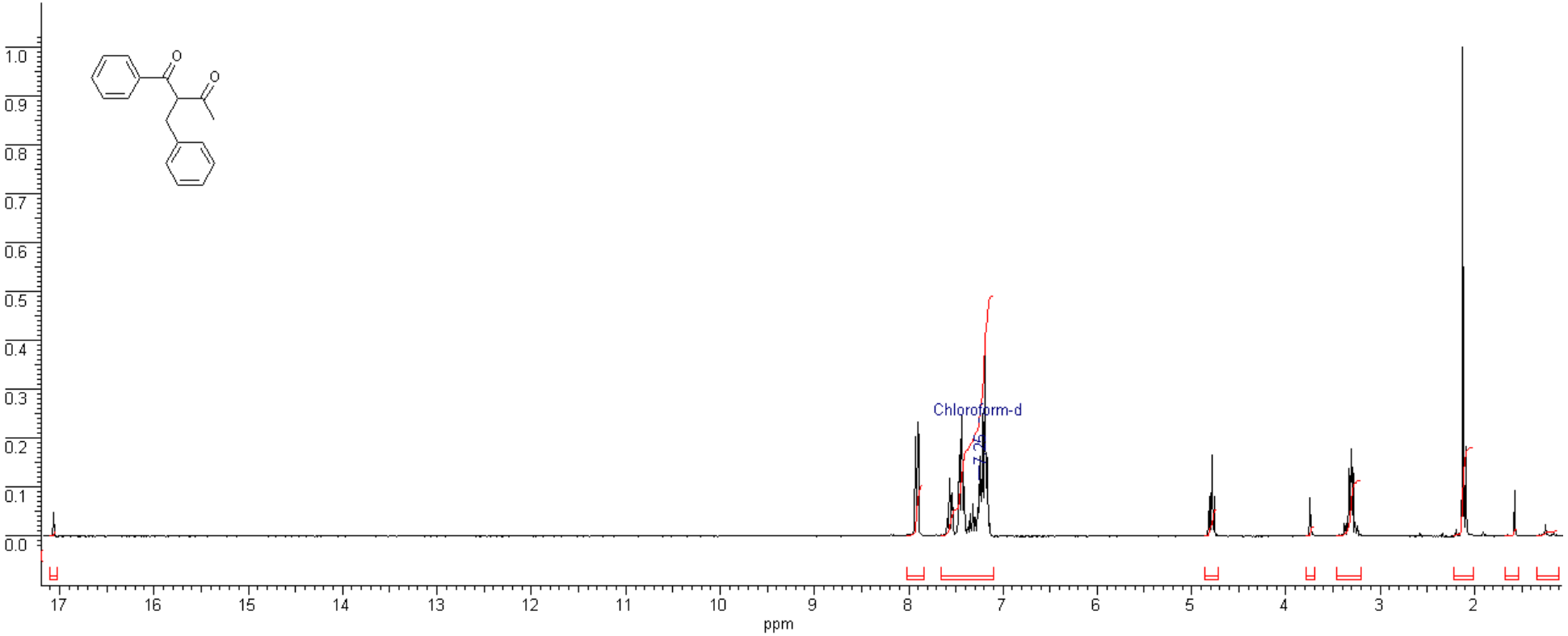
Acquisition Time (sec)	0.6759	***Comment***	/TSFC mk1324.32	***Date***	14 Jul 2012 08:25:36		
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	28.700						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

2-Benzyl-1-phenylbutane-1,3-dione (1p)

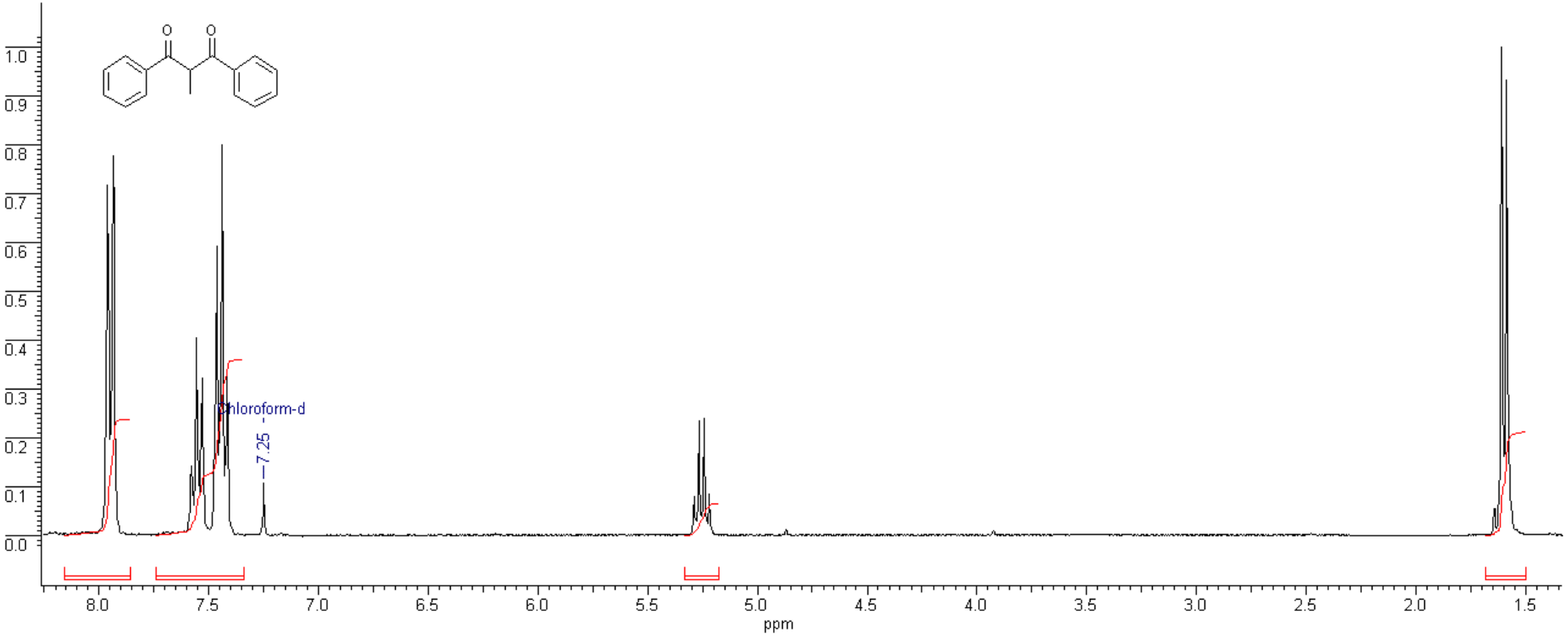
Acquisition Time (sec)	0.6759	***Comment***	NMR/50059953	***Date***	29 May 2012 10:05:52		
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	26.100						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

2-Methyl-1,3-diphenylpropane-1,3-dione (1q)

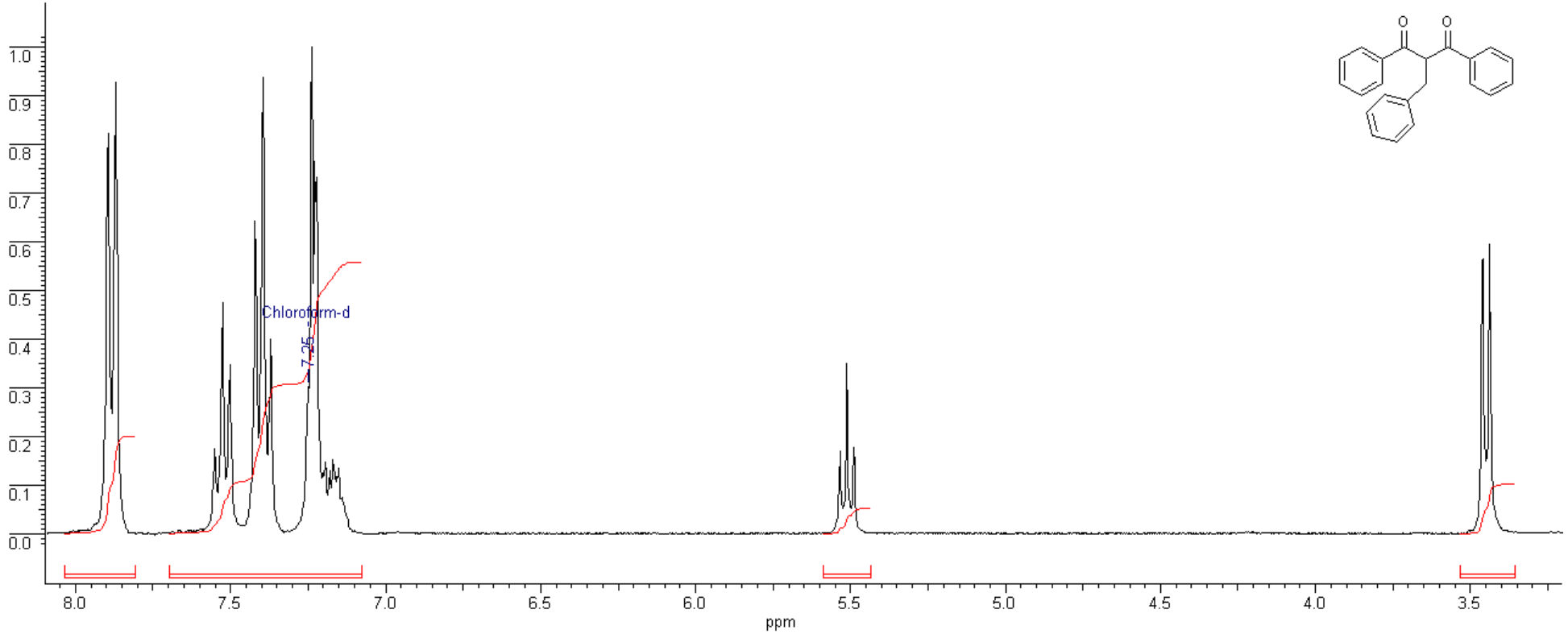
Acquisition Time (sec)	0.6759	***Comment***	NMR/50059953	***Date***	16 Jul 2012 14:00:32		
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	28.700						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

2-Benzyl-1,3-diphenylpropane-1,3-dione (1r)

Acquisition Time (sec)	0.6759	***Comment***	IGOR NH0023	***Date***	25 Jul 2012 10:14:24		
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	27.500						

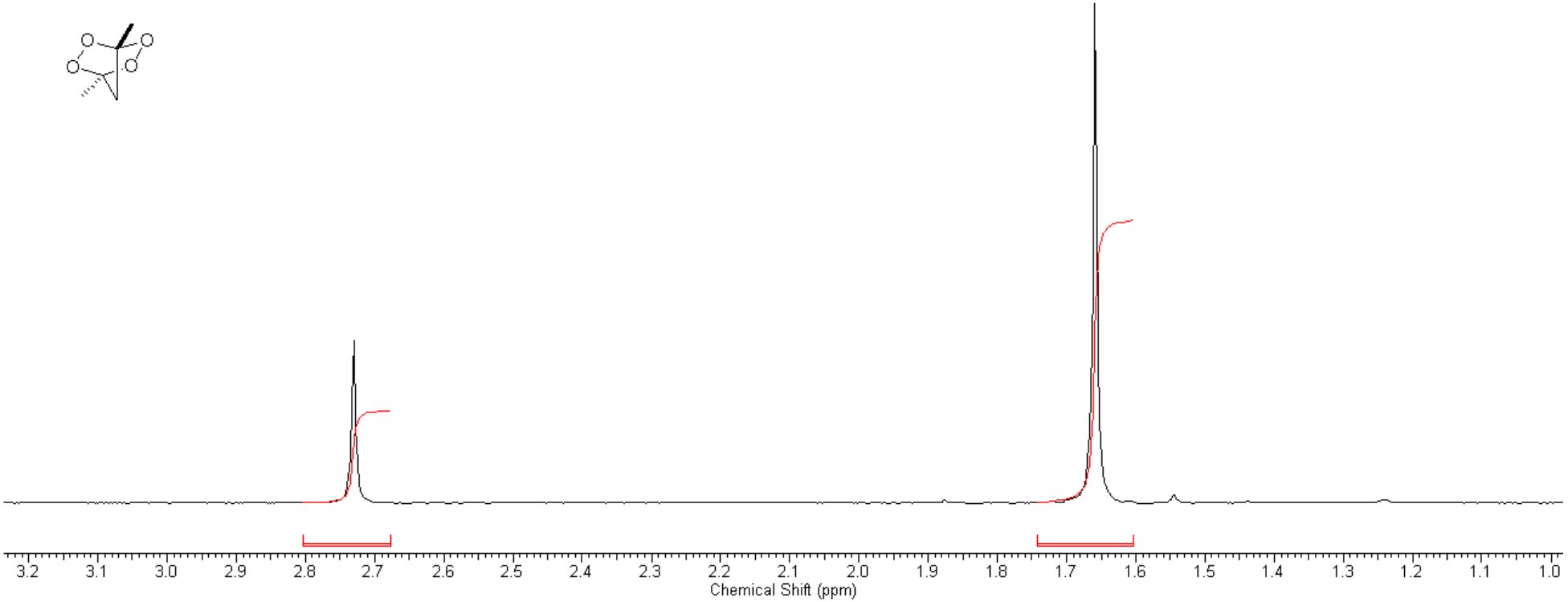


No.	Annotation	(ppm)
1	Chloroform-d	7.25

NMR spectra of tetraoxanes

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

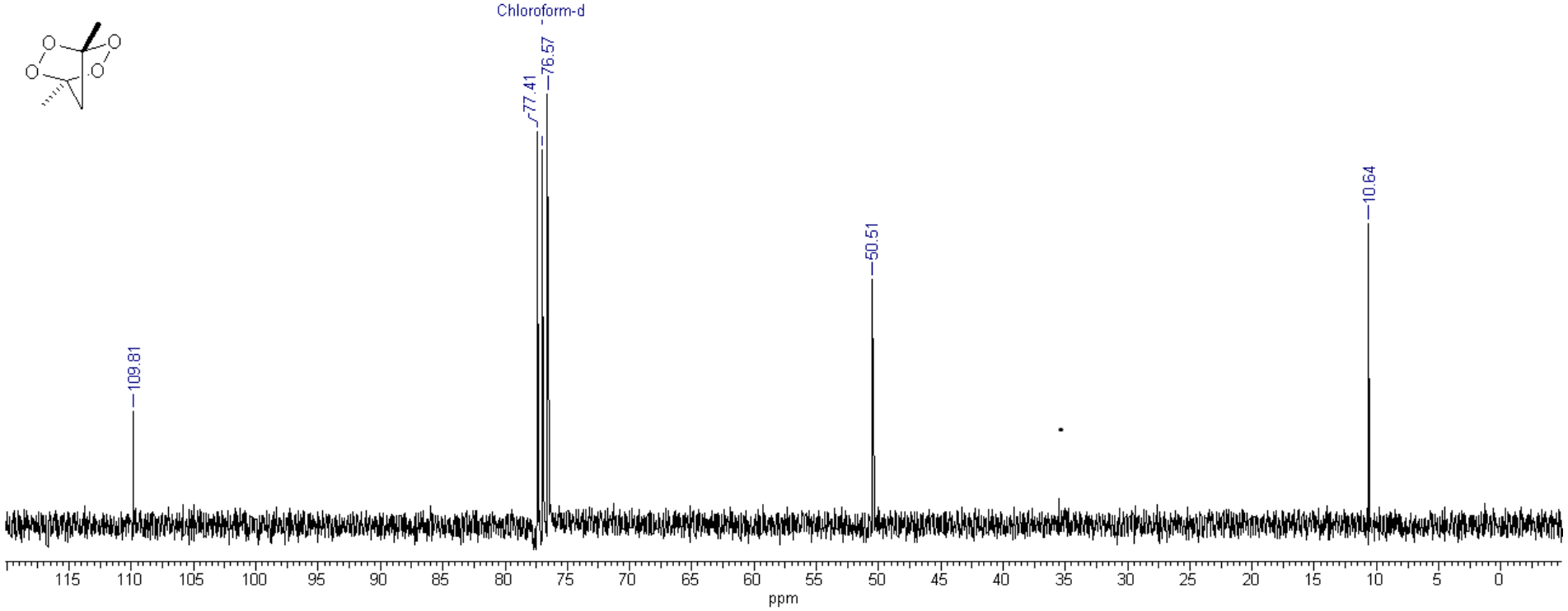
Acquisition Time (sec)	1.3518	***Date***	06 Apr 2012 13:52:00				
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	26.300						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

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

Acquisition Time (sec)	0.4322	***Comment***	Avance-300, C-13, CDCl3			***Date***	06 Apr 2012 14:04:48
File Name						***Frequency (MHz)***	75.48
Nucleus	13C	***Number of Transients***	114	***Original Points Count***	16308	***Points Count***	16384
Pulse Sequence	zgpg60base	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	18867.92
Temperature (degree C)	26.800						

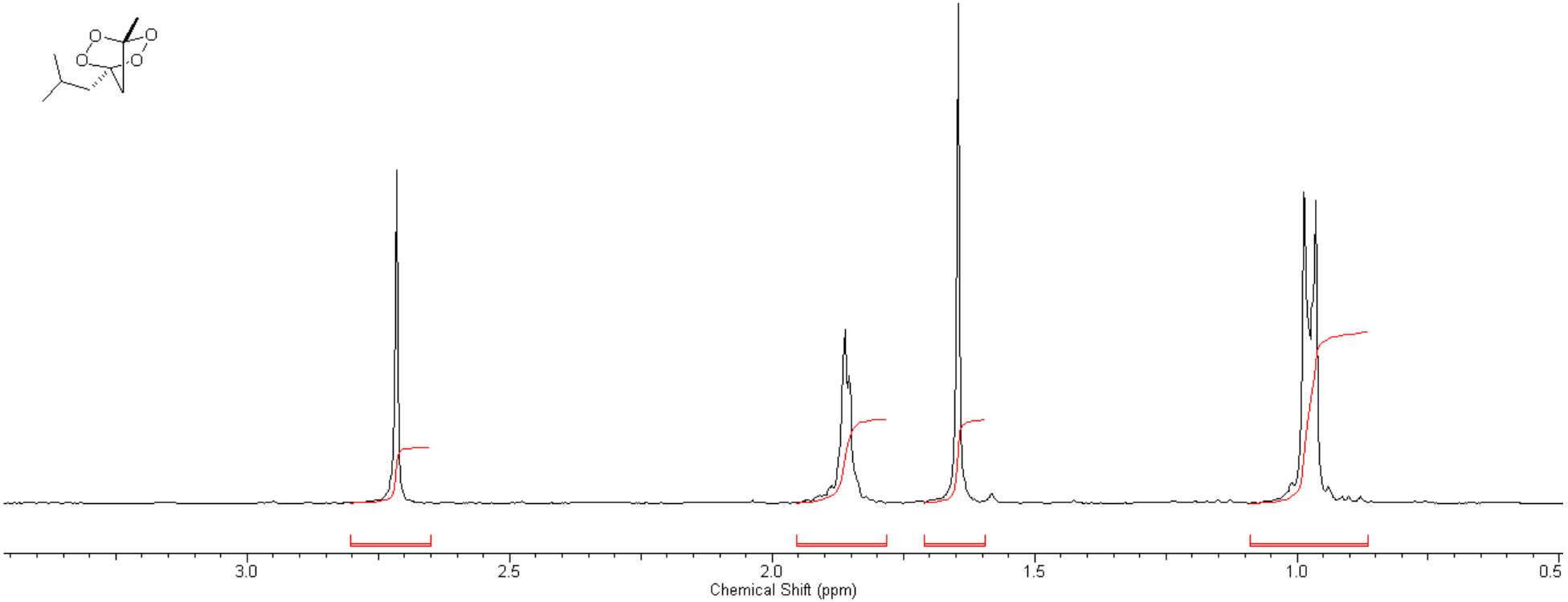


No.	(ppm)	(Hz)	Height
1	10.64	803.0	0.6986
2	50.51	3812.3	0.5685
3	76.57	5779.4	1.0000
4	77.00	5811.6	0.8690
5	77.41	5842.7	0.9122
6	109.81	8287.7	0.2611

No.	Annotation	(ppm)
1	Chloroform-d	77.00

1-Isobutyl-4-methyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2b)

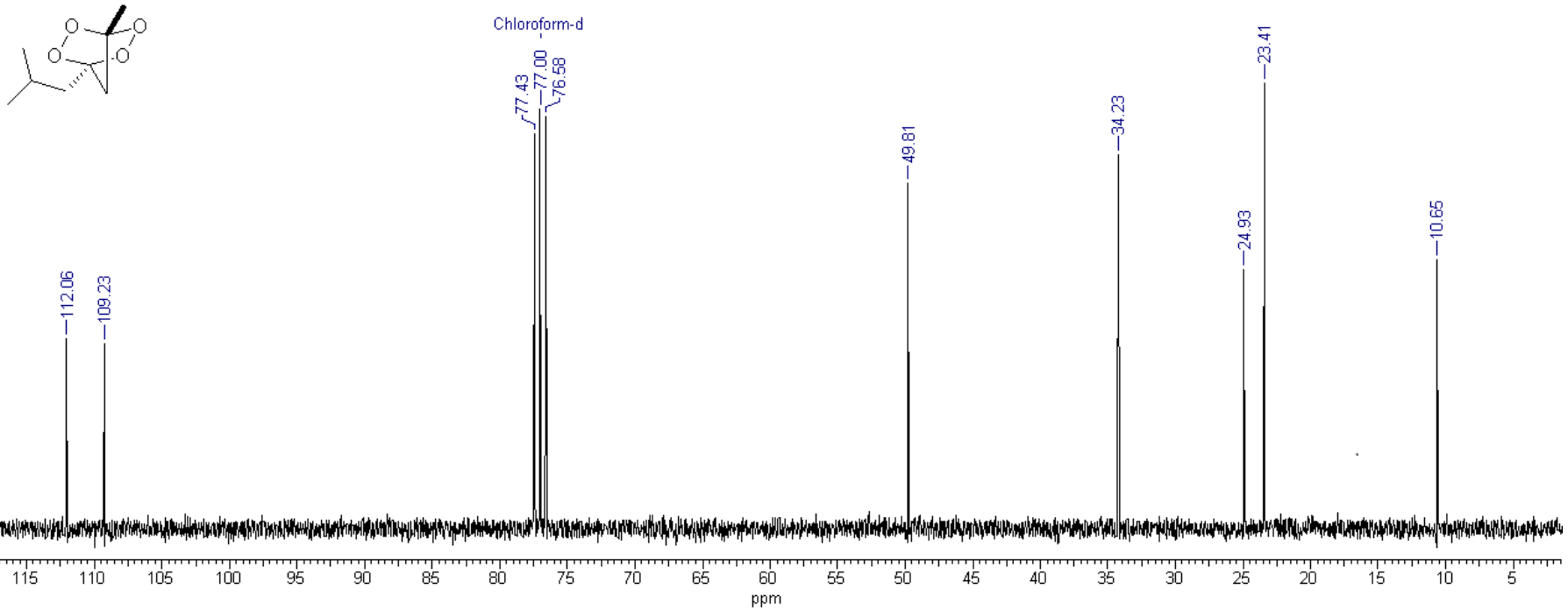
Acquisition Time (sec)	1.3518	***Date***	19 Apr 2012 14:32:32				
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	26.800						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

1-Isobutyl-4-methyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2b)

Acquisition Time (sec)	0.4501	***Comment***	Avance-300, C-13, CDCl3			***Date***	19 Apr 2012 14:32:32
File Name						***Frequency (MHz)***	75.48
Nucleus	13C	***Number of Transients***	138	***Original Points Count***	16308	***Points Count***	16384
Pulse Sequence	zgpg30base	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	18115.94
Temperature (degree C)	26.900						

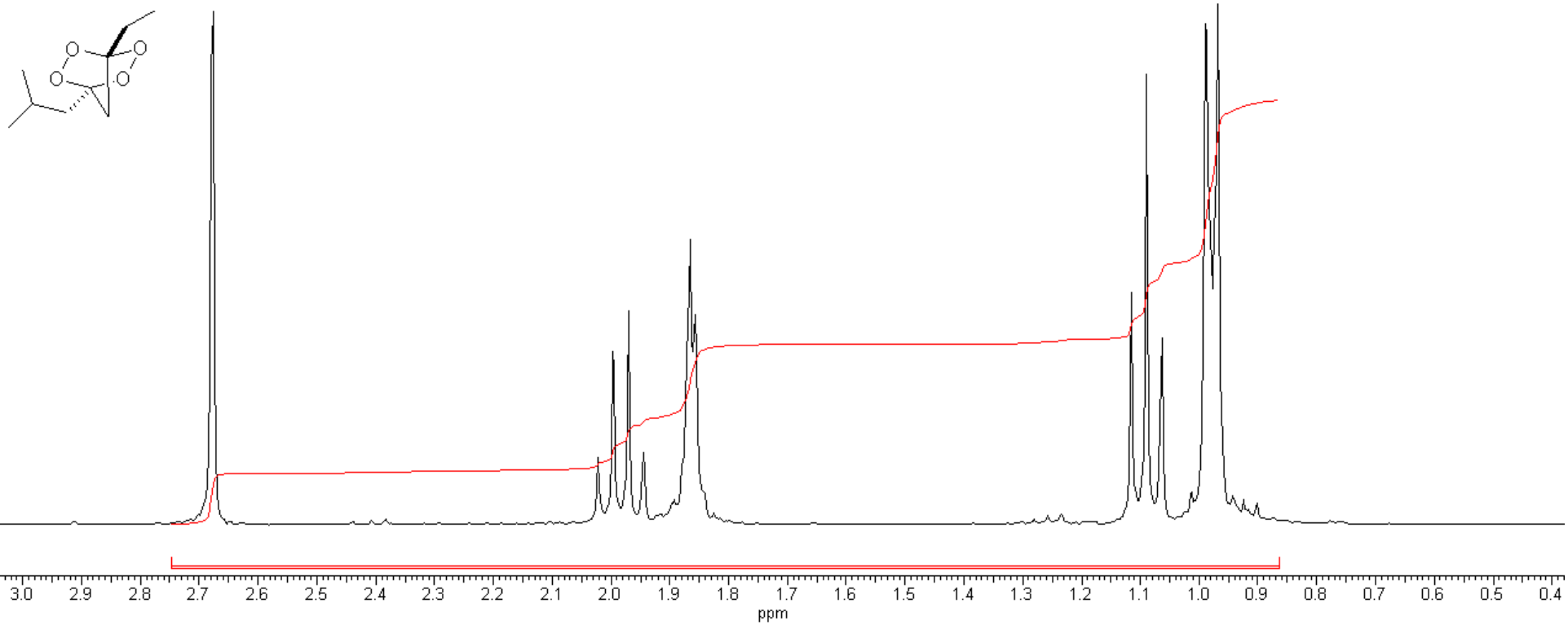


No.	(ppm)	(Hz)	Height
1	10.65	803.6	0.6038
2	23.41	1766.7	1.0000
3	24.93	1881.7	0.5817
4	34.23	2583.9	0.8385
5	49.81	3759.4	0.7739
6	76.58	5779.6	0.9251
7	77.00	5811.7	0.9419
8	77.43	5843.7	0.8852
9	109.23	8244.4	0.4161
10	112.06	8457.8	0.4270

No.	Annotation	(ppm)
1	Chloroform-d	77.00

1-Ethyl-4-isobutyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2c)

Acquisition Time (sec)	0.6759	***Comment***	NMR/50059953			***Date***	19 Apr 2012 14:53:52
File Name						***Frequency (MHz)***	300.13
Number of Transients	1	***Original Points Count***	8124	***Points Count***	8192	***Pulse Sequence***	zg
Solvent	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62	***Temperature (degree C)***	27.200



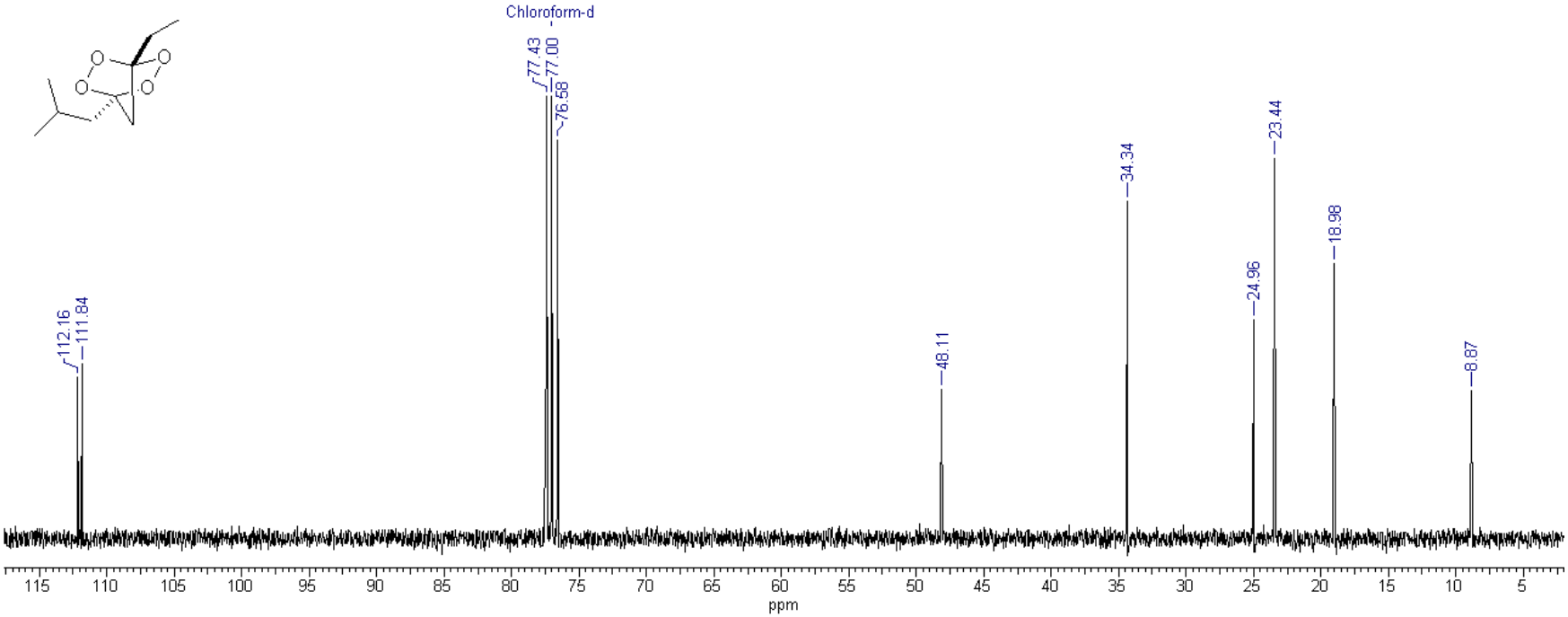
No.	(ppm)	(Hz)	Height
1	7.25	2176.1	0.0262

No.	Annotation	(ppm)
1	Chloroform-d	7.25

No.	(ppm)	Value
1	.86 .. 2.7	150.000

1-Ethyl-4-isobutyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2c)

Acquisition Time (sec)	0.4501	***Comment***	Avance-300, C-13, CDCl3			***Date***	19 Apr 2012 14:56:00
File Name						***Frequency (MHz)***	75.48
Nucleus	13C	***Number of Transients***	201	***Original Points Count***	16308	***Points Count***	16384
Pulse Sequence	zgpg30base	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	18115.94
Temperature (degree C)	27.100						



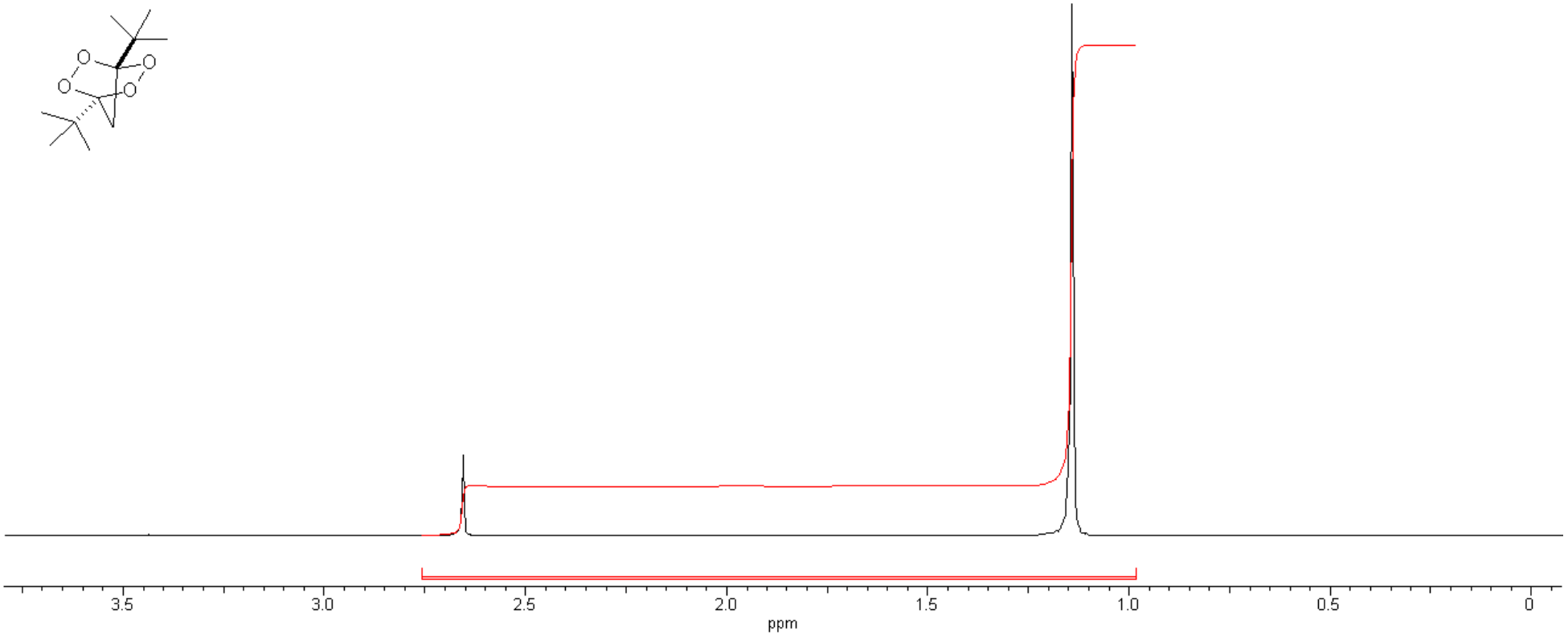
No.	(ppm)	(Hz)	Height
1	8.87	669.8	0.3348
2	18.98	1432.8	0.6199
3	23.44	1769.0	0.8576
4	24.96	1884.0	0.4939
5	34.34	2591.7	0.7627
6	48.11	3631.1	0.3371
7	76.58	5779.6	0.8994
8	77.00	5811.7	1.0000
9	77.43	5843.8	0.9991
10	111.84	8441.2	0.3935
11	112.16	8465.5	0.3653

No.	Annotation	(ppm)
1	Chloroform-d	77.00

1,4-Di-*tert*-butyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2d)

Acquisition Time (sec)	0.6759	***Comment***	NMR/50059953			***Date***	18 Jun 2012 10:42:08
File Name						***Frequency (MHz)***	300.13
Number of Transients	1	***Original Points Count***	8124	***Points Count***	8192	***Pulse Sequence***	zg
Solvent	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62	***Temperature (degree C)***	26.300

Nucleus 1H



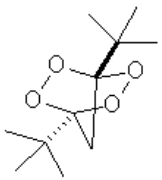
No.	(ppm)	(Hz)	Height
1	7.25	2176.1	0.0097

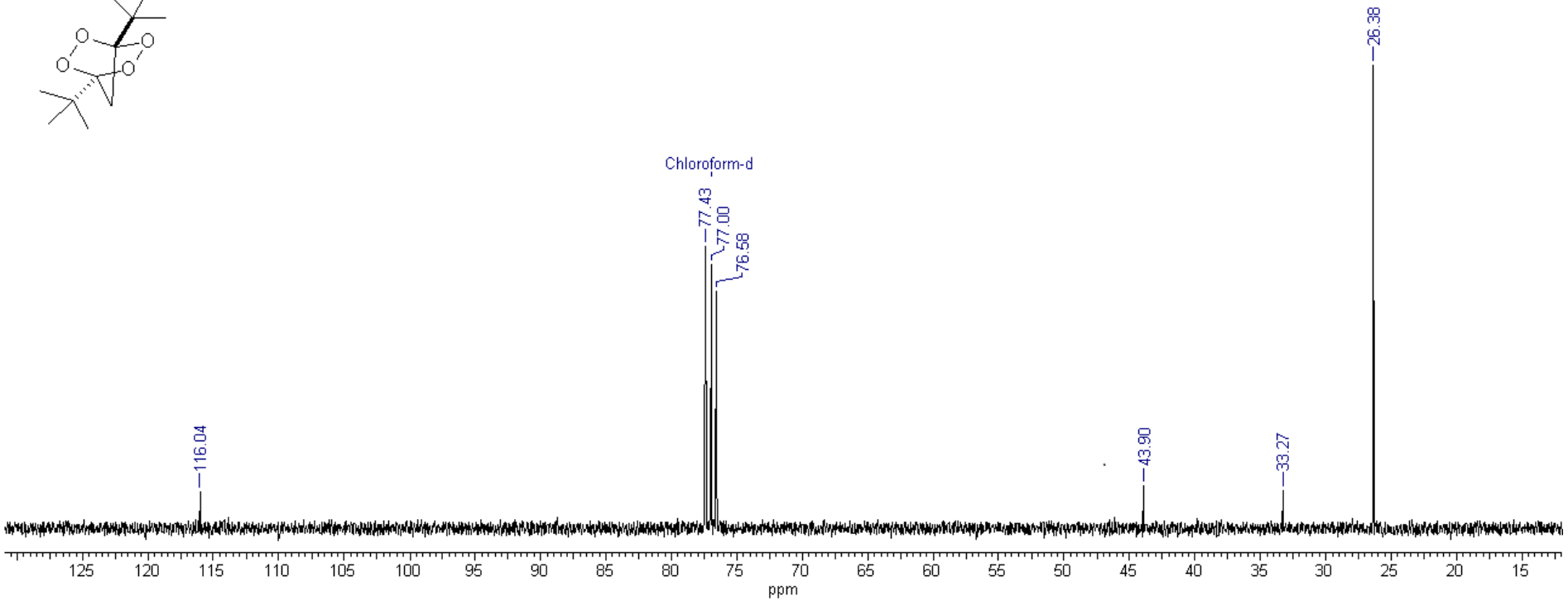
No.	Annotation	(ppm)
1	Chloroform-d	7.25

No.	(ppm)	Value
1	.98 .. 2.7	50.000

1,4-Di-*tert*-butyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2d)

Acquisition Time (sec)	0.4501	***Comment***	Avance-300, C-13, CDCl3			***Date***	18 Jun 2012 10:44:16
File Name						***Frequency (MHz)***	75.48
Nucleus	13C	***Number of Transients***	139	***Original Points Count***	16308	***Points Count***	16384
Pulse Sequence	zgpg30base	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	18115.94
Temperature (degree C)	26.400						



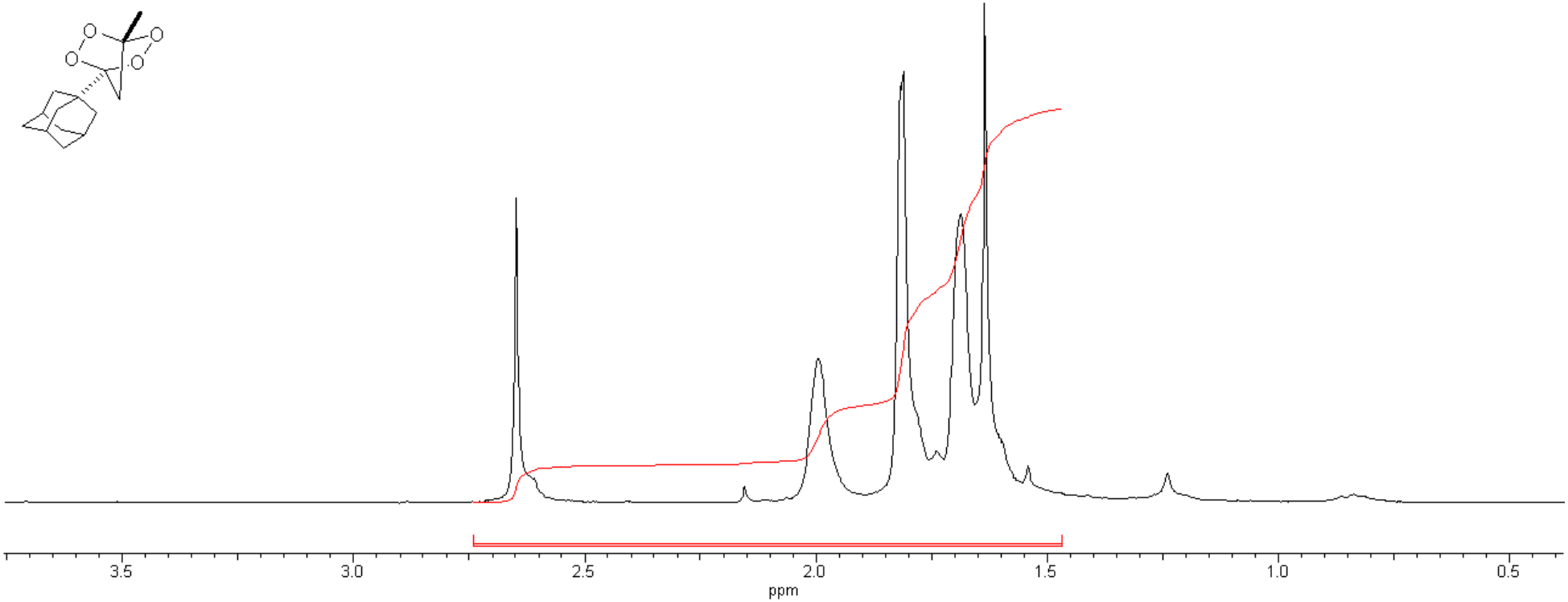


No.	(ppm)	(Hz)	Height
1	26.38	1991.2	1.0000
2	33.27	2510.9	0.0813
3	43.90	3313.7	0.0905
4	76.58	5779.6	0.5130
5	77.00	5811.7	0.5690
6	77.43	5843.8	0.6084
7	116.04	8758.6	0.0790

No.	Annotation	(ppm)
1	Chloroform-d	77.00

1-(1-Adamantyl)-4-methyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2e)

Acquisition Time (sec)	0.6759	*Comment*	/LB58 ZSS130	*Date*	07 Dec 2011 14:38:56		
File Name						*Frequency (MHz)*	300.13
Nucleus	1H	*Number of Transients*	1	*Original Points Count*	8124	*Points Count*	8192
Pulse Sequence	zg	*Solvent*	CHLOROFORM-D			*Sweep Width (Hz)*	6009.62
Temperature (degree C)	26.900						



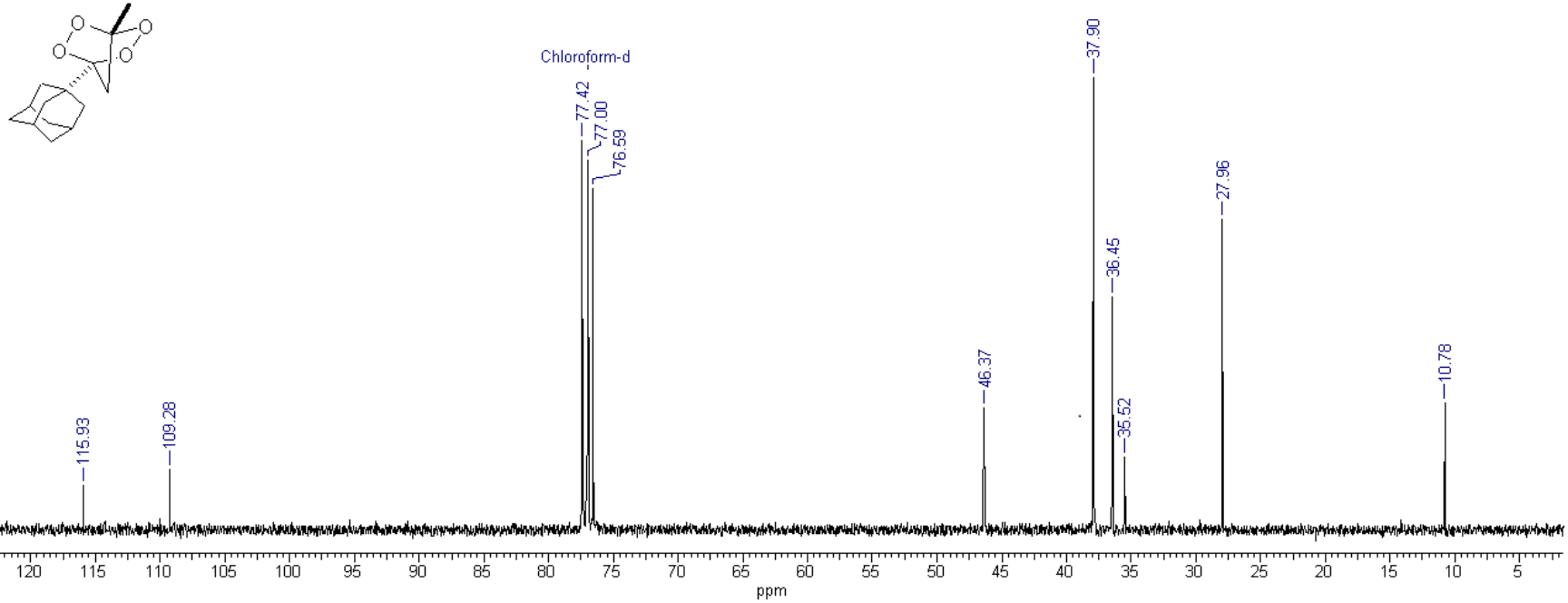
No.	(ppm)	(Hz)	Height
1	7.25	2176.0	0.0547

No.	Annotation	(ppm)
1	Chloroform-d	7.25

No.	(ppm)	Value
1	.47 .. 2.7	11.317

1-(1-Adamantyl)-4-methyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2e)

Acquisition Time (sec)	0.4501	***Comment***	Avance-300, C-13, CDCB			***Date***	07 Dec 2011 14:41:04
File Name						***Frequency (MHz)***	75.48
Nucleus	13C	***Number of Transients***	1154	***Original Points Count***	16308	***Points Count***	16384
Pulse Sequence	zgpg30base	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	18115.94
Temperature (degree C)	26.900						

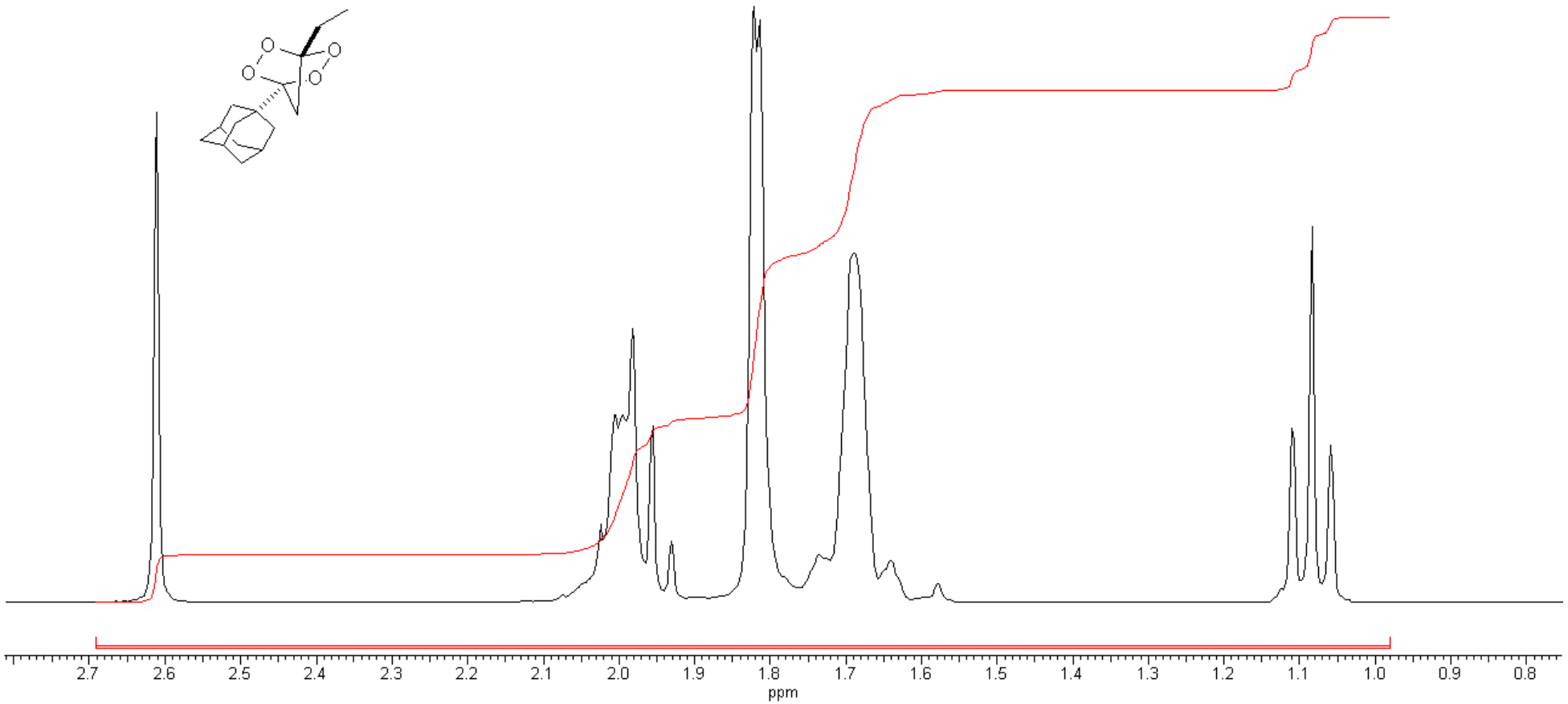


No.	(ppm)	(Hz)	Height
1	10.78	813.5	0.2808
2	27.96	2110.6	0.6854
3	35.52	2681.2	0.1613
4	36.45	2750.9	0.5148
5	37.90	2860.3	1.0000
6	46.37	3499.5	0.2682
7	76.59	5780.7	0.7546
8	77.00	5811.7	0.8163
9	77.42	5843.7	0.8593
10	109.28	8247.7	0.1345
11	115.93	8749.7	0.0972

No.	Annotation	(ppm)
1	Chloroform-d	77.00

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

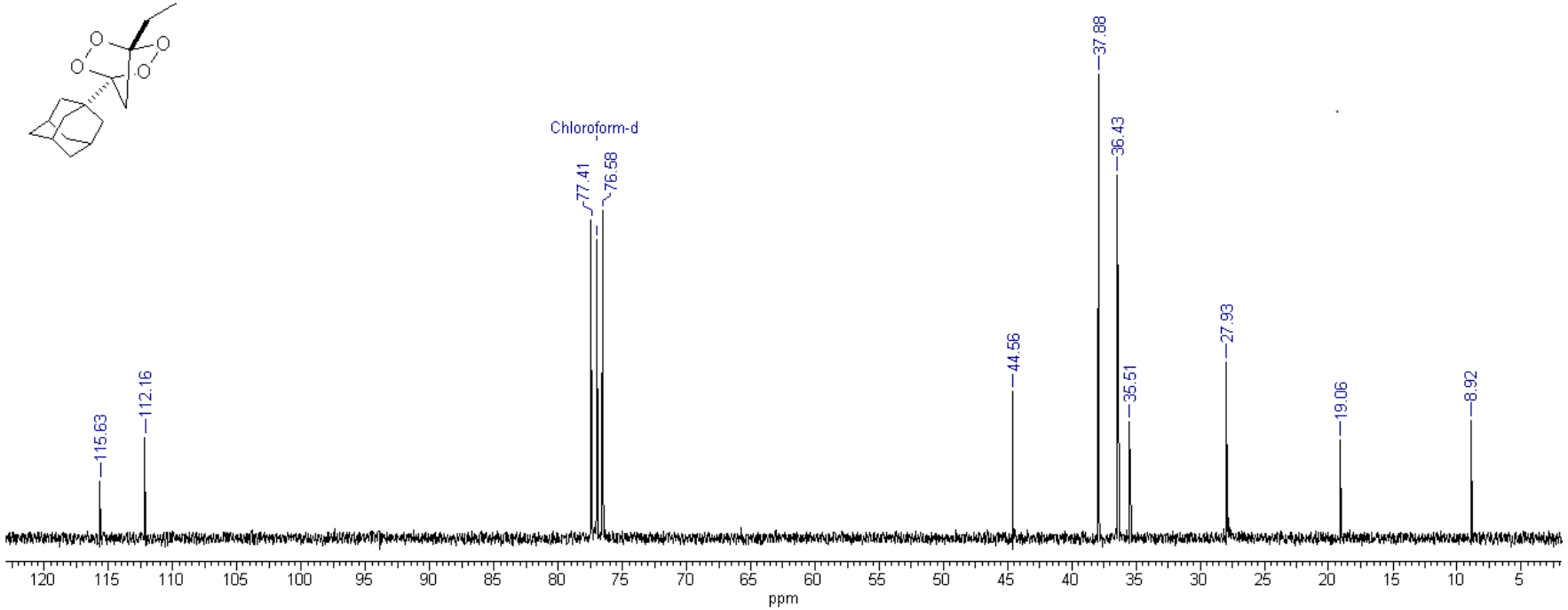
Acquisition Time (sec)	0.6759	***Comment***	NMR/50059953			***Date***	18 Jan 2012 15:13:04
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	27.000						



No.	(ppm)	Value
1	1.98 .. 2.6	150.000

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

Acquisition Time (sec)	0.4501	***Comment***	Avance-300, C-13, CDCl3			***Date***	18 Jan 2012 15:15:12
File Name						***Frequency (MHz)***	75.48
Nucleus	13C	***Number of Transients***	231	***Original Points Count***	16308	***Points Count***	16384
Pulse Sequence	zgpg30base	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	18115.94
Temperature (degree C)	26.200						



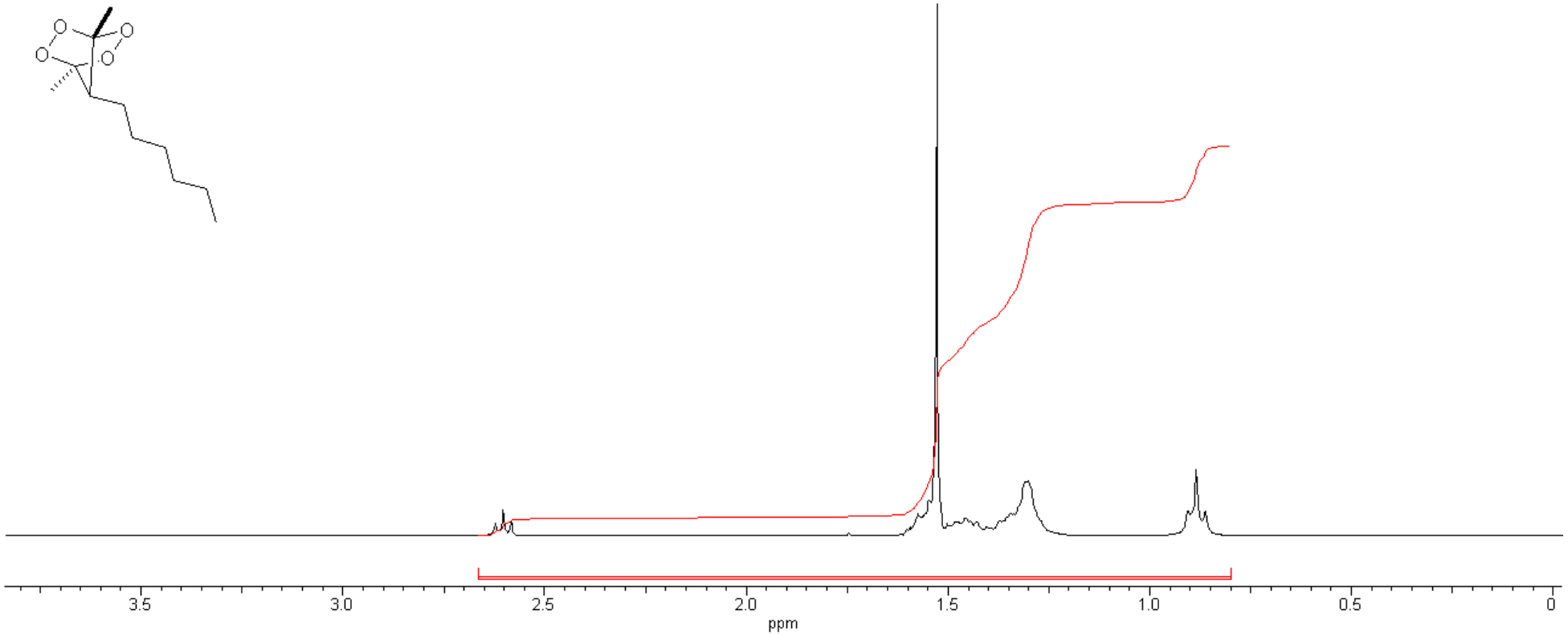
No.	(ppm)	(Hz)	Height
1	8.92	673.1	0.2548
2	19.06	1438.3	0.2121
3	27.93	2108.4	0.3807
4	35.51	2680.1	0.2502
5	36.43	2749.8	0.7835
6	37.88	2859.2	1.0000
7	44.56	3363.5	0.3150
8	76.58	5779.6	0.7055
9	77.00	5811.7	0.6429
10	77.41	5842.6	0.6864
11	112.16	8465.5	0.2182
12	115.63	8727.6	0.1220

No.	Annotation	(ppm)
1	Chloroform-d	77.00

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

Acquisition Time (sec)	0.6759	*Comment*	NMR/50059953			*Date*	28 May 2012 12:56:32
File Name						*Frequency (MHz)*	300.13
Number of Transients	1	*Original Points Count*	8124	*Points Count*	8192	*Pulse Sequence*	zg
Solvent	CHLOROFORM-D			*Sweep Width (Hz)*	6009.62	*Temperature (degree C)*	26.200

Nucleus	1H



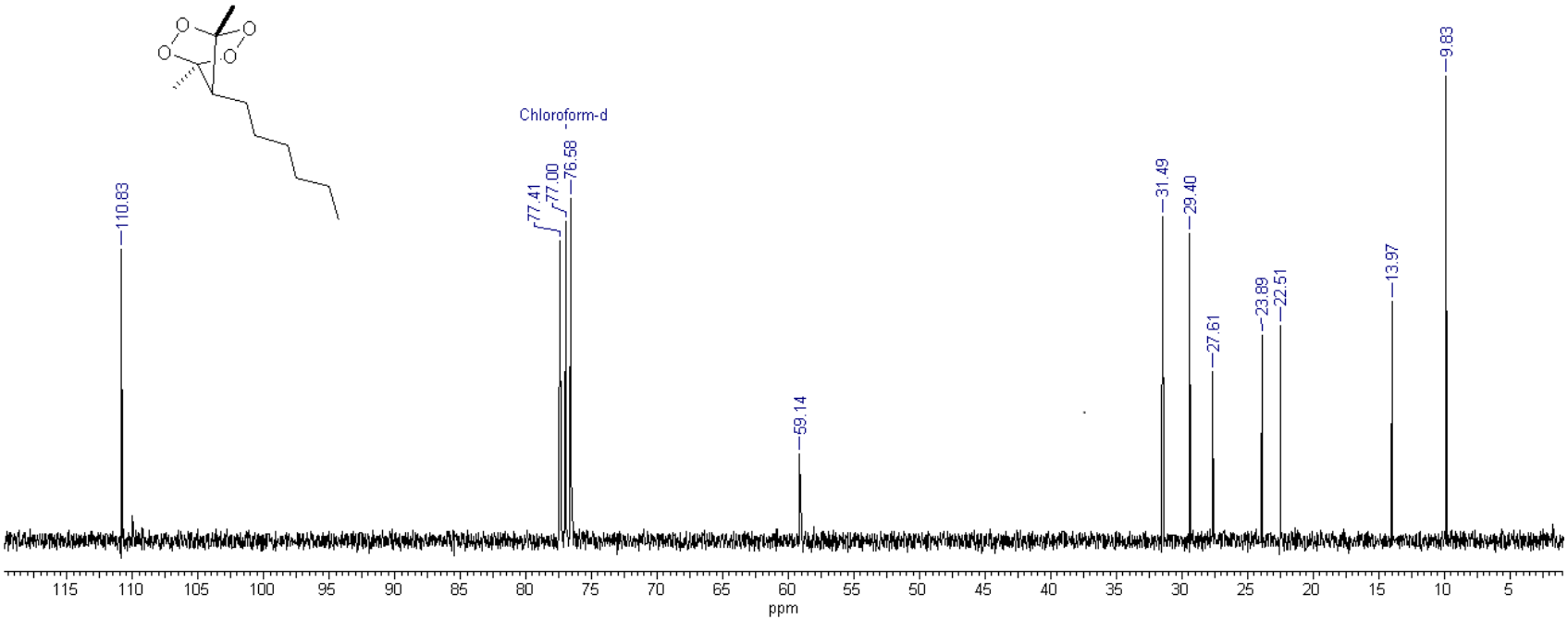
No.	(ppm)	(Hz)	Height
1	7.25	2176.1	0.0064

No.	Annotation	(ppm)
1	Chloroform-d	7.25

No.	(ppm)	Value
1	.80 .. 2.6	150.000

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

Acquisition Time (sec)	0.4501	***Comment***	Avance-300, C-13, CDCl3			***Date***	28 May 2012 12:58:40
File Name						***Frequency (MHz)***	75.48
Nucleus	13C	***Number of Transients***	157	***Original Points Count***	16308	***Points Count***	16384
Pulse Sequence	zgpg30base	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	18115.94
Temperature (degree C)	26.300						

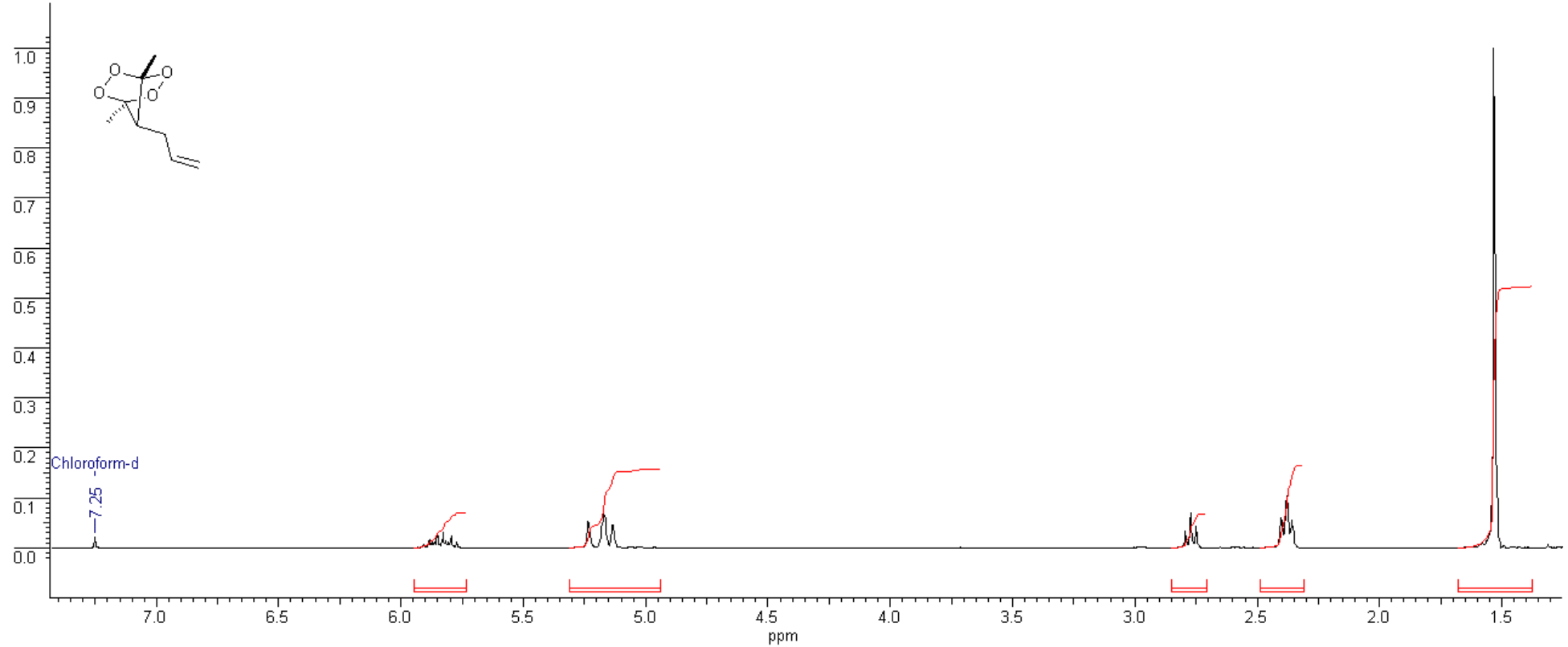


No.	(ppm)	(Hz)	Height
1	9.83	741.7	1.0000
2	13.97	1054.6	0.5154
3	22.51	1699.3	0.4640
4	23.89	1803.2	0.4416
5	27.61	2084.1	0.3651
6	29.40	2219.0	0.6601
7	31.49	2377.1	0.6974
8	59.14	4463.7	0.1854
9	76.58	5779.6	0.7381
10	77.00	5811.7	0.6893
11	77.41	5842.6	0.6469
12	110.83	8364.9	0.6285

No.	Annotation	(ppm)
1	Chloroform-d	77.00

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

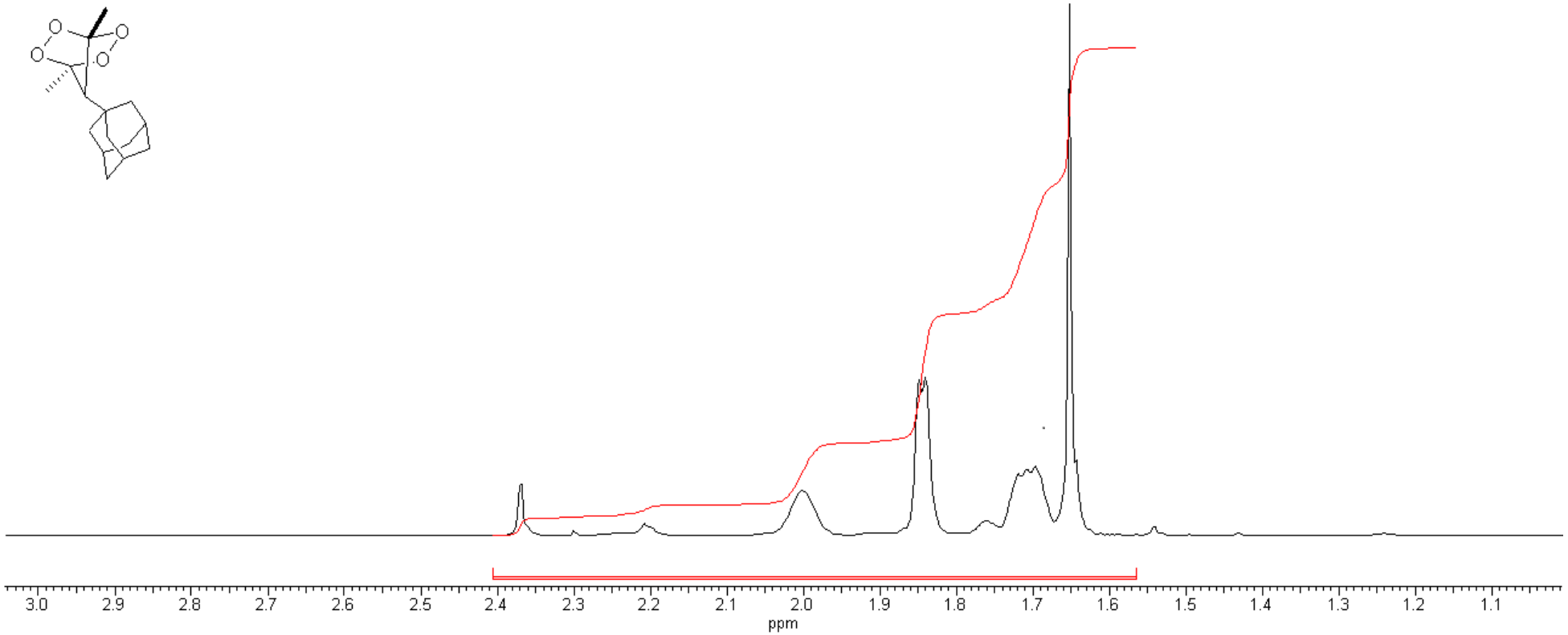
Acquisition Time (sec)	0.6759	***Comment***	NMR/50059953			***Date***	06 Feb 2012 10:18:40
File Name						***Frequency (MHz)***	300.13
Number of Transients	1	***Original Points Count***	8124	***Points Count***	8192	***Pulse Sequence***	zg
Solvent	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62	***Temperature (degree C)***	23.800



No.	Annotation	(ppm)
1	Chloroform-d	7.25

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

Acquisition Time (sec)	0.6759	***Comment***	NMR/50059953			***Date***	05 Mar 2012 10:27:12
File Name						***Frequency (MHz)***	300.13
Number of Transients	1	***Original Points Count***	8124	***Points Count***	8192	***Pulse Sequence***	zg
Solvent	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62	***Temperature (degree C)***	27.000



No.	(ppm)	(Hz)	Height
1	7.25	2176.1	0.0183

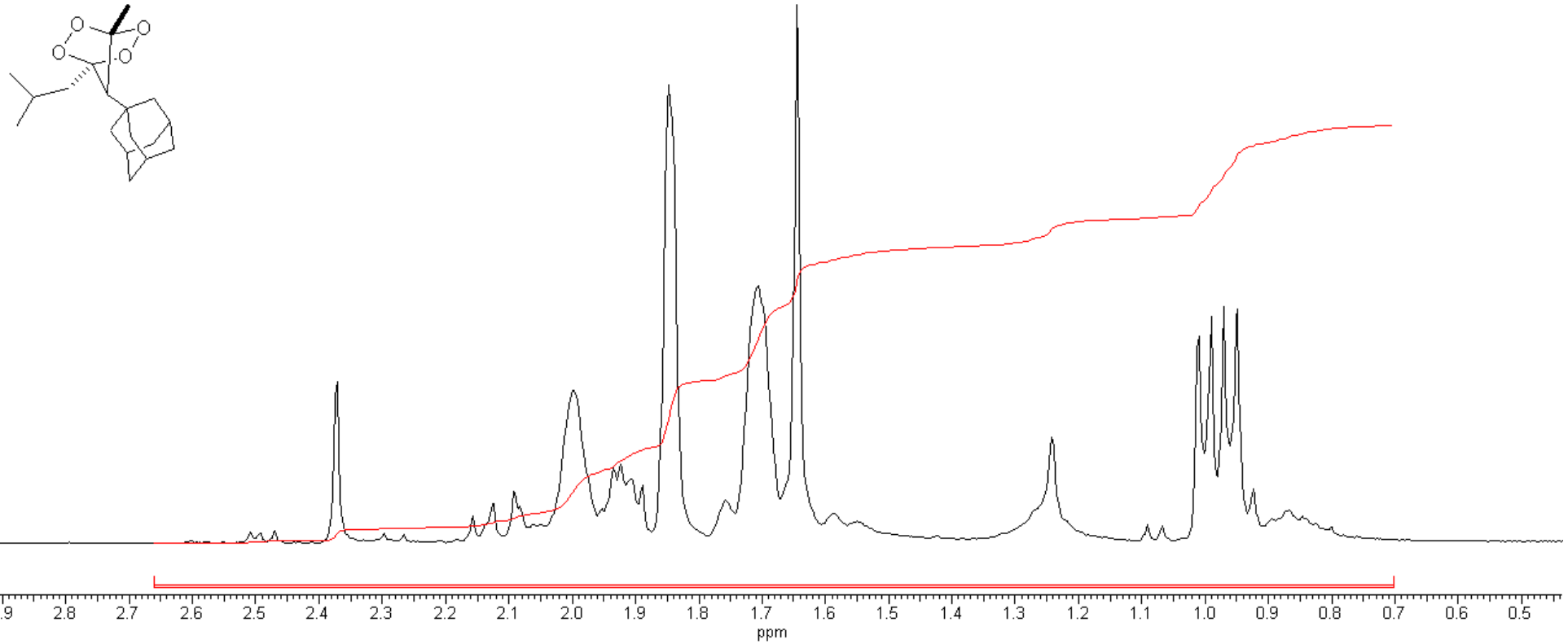
No.	Annotation	(ppm)
1	Chloroform-d	7.25

No.	(ppm)	Value
1	.56 .. 2.4	150.000

7-(1-Adamantyl)-1-isobutyl-4-methyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2j)

Acquisition Time (sec)	0.6759	*Comment*	NMR/50059953			*Date*	14 May 2012 11:01:20
File Name						*Frequency (MHz)*	300.13
Number of Transients	1	*Original Points Count*	8124	*Points Count*	8192	*Pulse Sequence*	zg
Solvent	CHLOROFORM-D			*Sweep Width (Hz)*	6009.62	*Temperature (degree C)*	27.000

Nucleus	1H



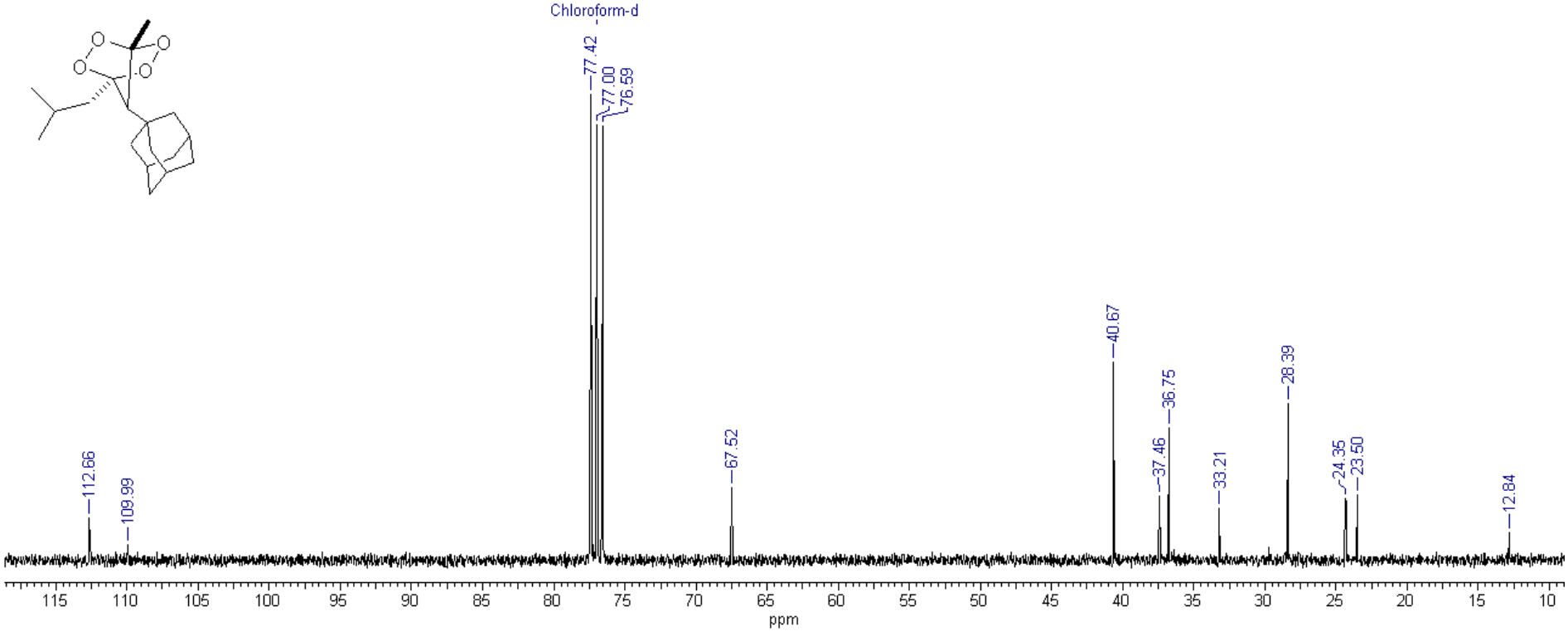
No.	(ppm)	(Hz)	Height
1	7.25	2176.1	0.1199

No.	Annotation	(ppm)
1	Chloroform-d	7.25

No.	(ppm)	Value
1	.70 .. 2.6	150.000

7-(1-Adamantyl)-1-isobutyl-4-methyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2j)

Acquisition Time (sec)	0.4501	***Comment***	Avance-300, C-13, CDCl3			***Date***	14 May 2012 11:01:20
File Name						***Frequency (MHz)***	75.48
Nucleus	13C	***Number of Transients***	493	***Original Points Count***	16308	***Points Count***	16384
Pulse Sequence	zgpg30base	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	18115.94
Temperature (degree C)	25.600						



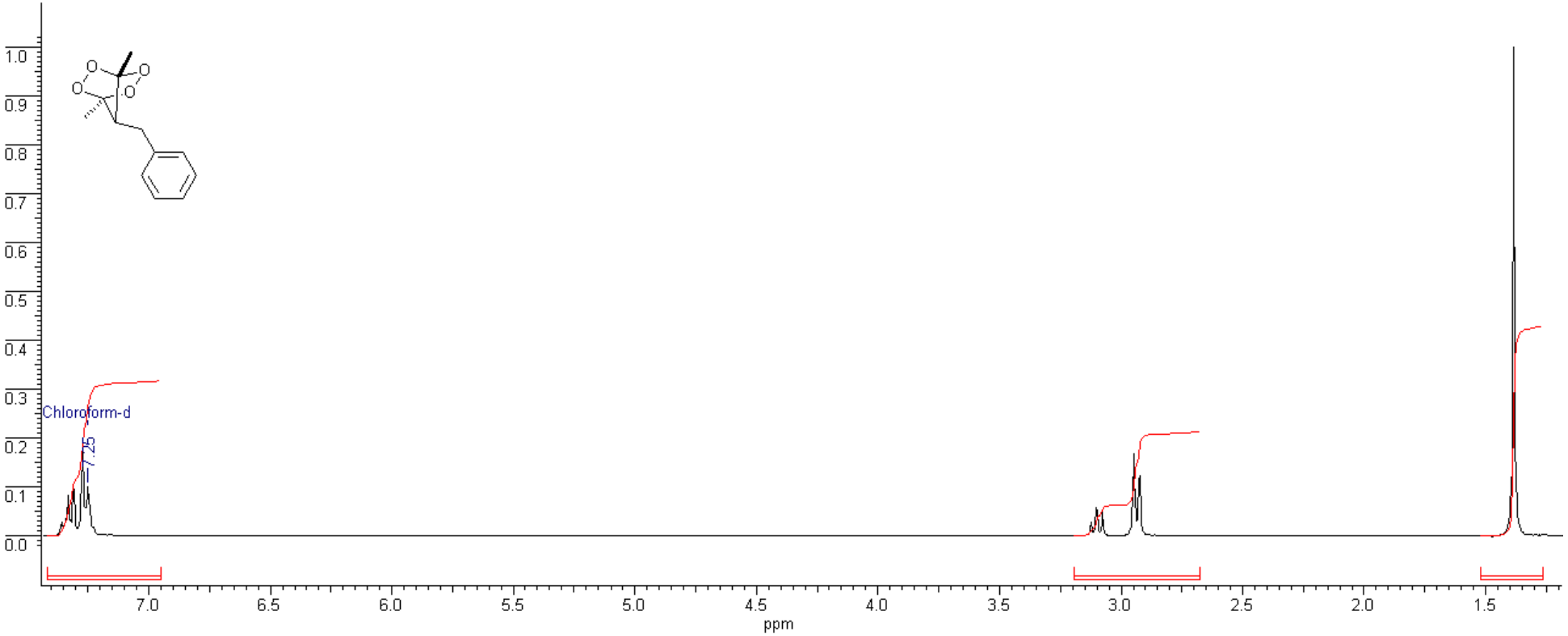
No.	(ppm)	(Hz)	Height
1	12.84	969.4	0.0590
2	23.50	1773.3	0.1395
3	24.35	1837.5	0.1339
4	28.39	2142.7	0.3356
5	33.21	2506.5	0.1107
6	36.75	2774.1	0.2847
7	37.46	2827.1	0.1373

No.	(ppm)	(Hz)	Height
8	40.67	3069.3	0.4264
9	67.52	5096.2	0.1551
10	76.59	5780.7	0.9321
11	77.00	5811.6	0.9344
12	77.42	5843.7	1.0000
13	109.99	8301.8	0.0326
14	112.66	8503.1	0.0903

No.	Annotation	(ppm)
1	Chloroform-d	77.00

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

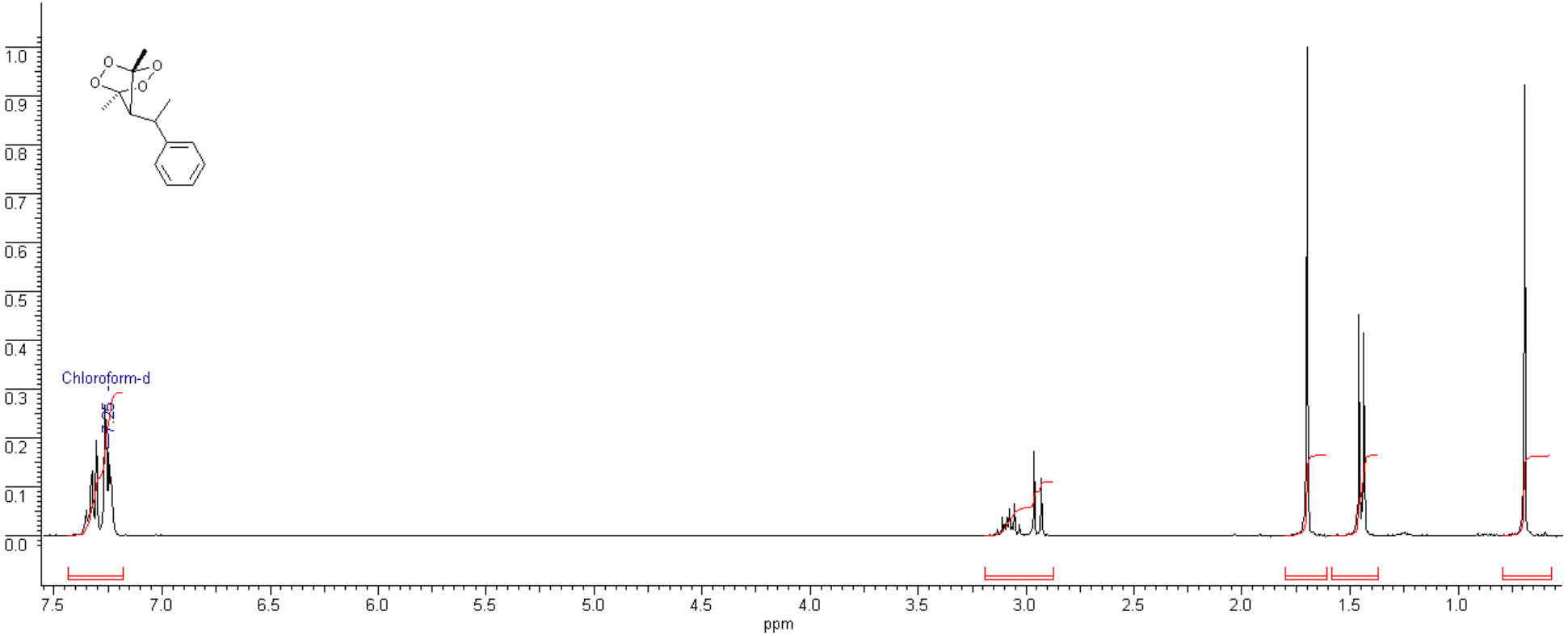
Acquisition Time (sec)	0.6759	***Comment***	NMR/50059953	***Date***	10 Jul 2012 12:07:28		
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	27.900						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

1,4-Dimethyl-7-(1-phenylethyl)-2,3,5,6-tetraoxabicyclo[2.2.1]heptanes (2l)

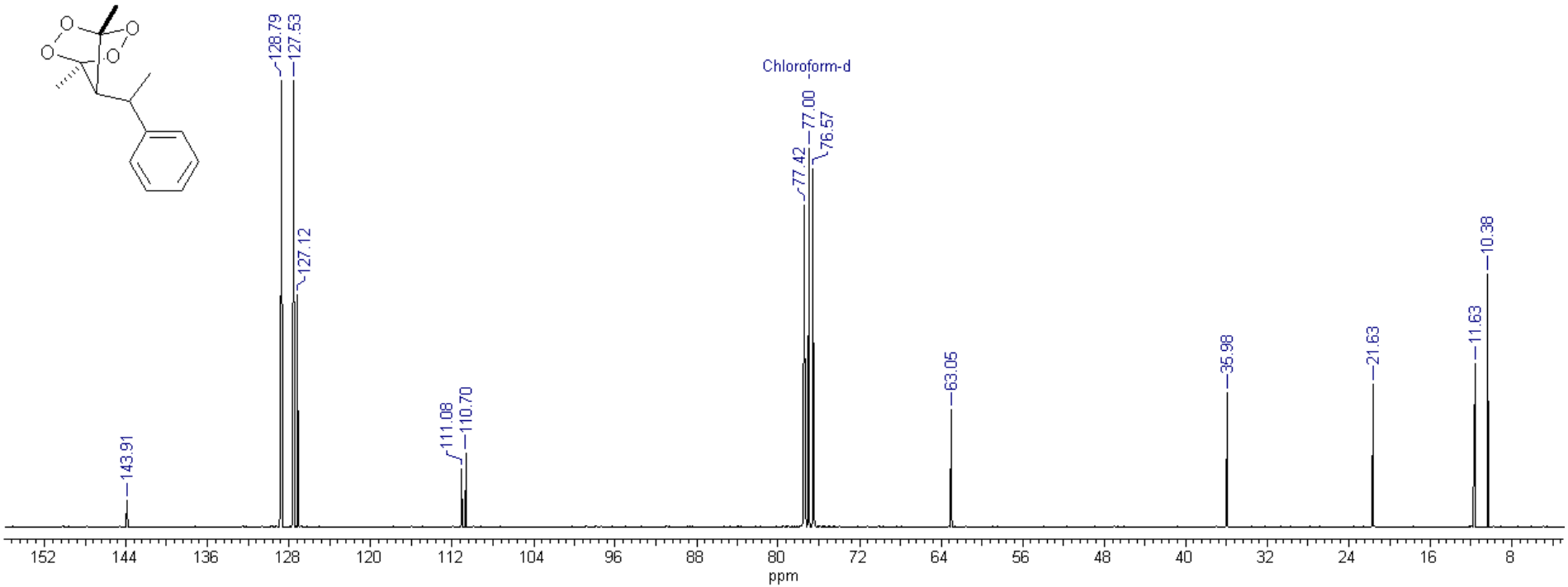
Acquisition Time (sec)	0.6759	***Comment***	NMR/50059953	***Date***	19 Apr 2012 14:24:00		
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	27.000						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

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

Acquisition Time (sec)	0.4501	***Comment***	Avance-300, C-13, CDCl3			***Date***	18 Jan 2012 14:43:12
File Name						***Frequency (MHz)***	75.48
Nucleus	13C	***Number of Transients***	92	***Original Points Count***	16308	***Points Count***	16384
Pulse Sequence	zgpg30base	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	18115.94
Temperature (degree C)	27.000						



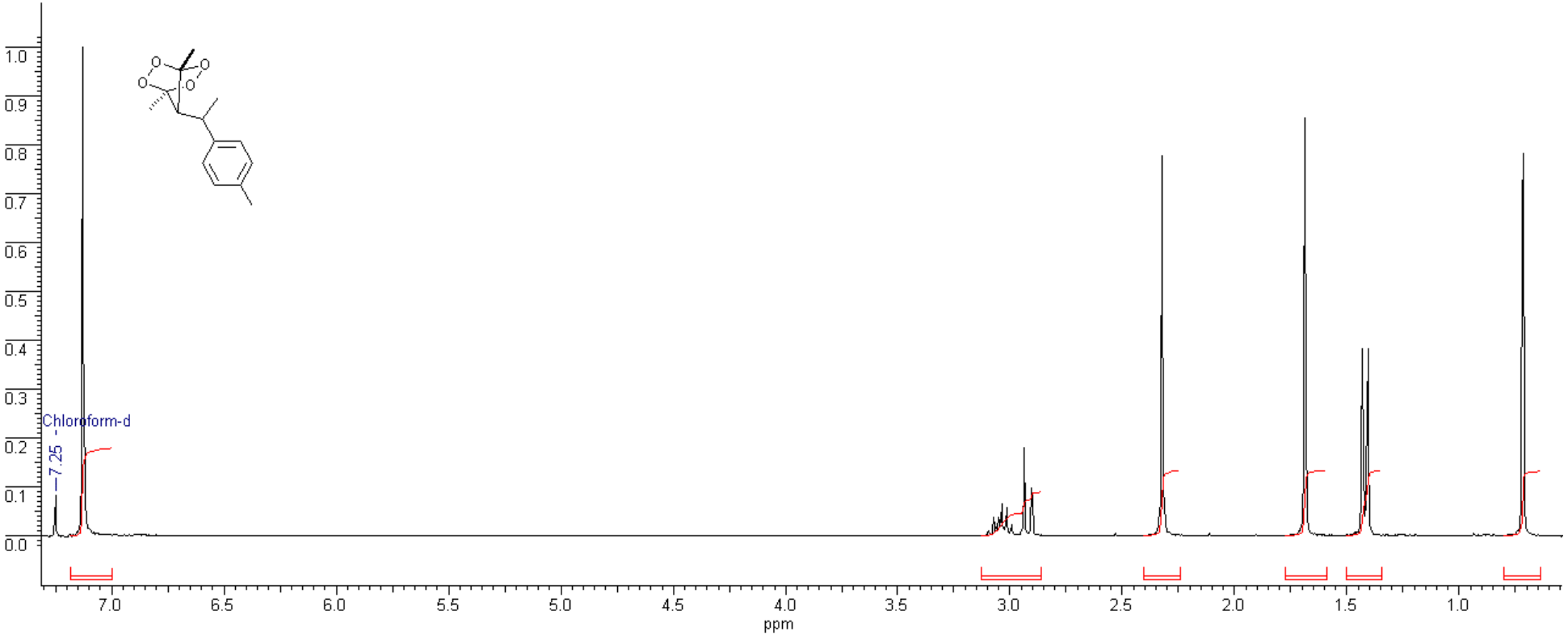
No.	(ppm)	(Hz)	Height
1	10.38	783.7	0.2908
2	11.63	877.7	0.1873
3	21.63	1632.9	0.1641
4	35.98	2715.5	0.1547
5	63.05	4758.9	0.1349
6	76.57	5779.6	0.4110
7	77.00	5811.6	0.4348

No.	(ppm)	(Hz)	Height
8	77.42	5843.7	0.3690
9	110.70	8354.9	0.0849
10	111.08	8383.7	0.0671
11	127.12	9594.5	0.2677
12	127.53	9625.5	1.0000
13	128.79	9720.6	0.9970
14	143.91	10861.7	0.0303

No.	Annotation	(ppm)
1	Chloroform-d	77.00

1,4-Dimethyl-7-[1-(4-methylphenyl)ethyl]-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2m)

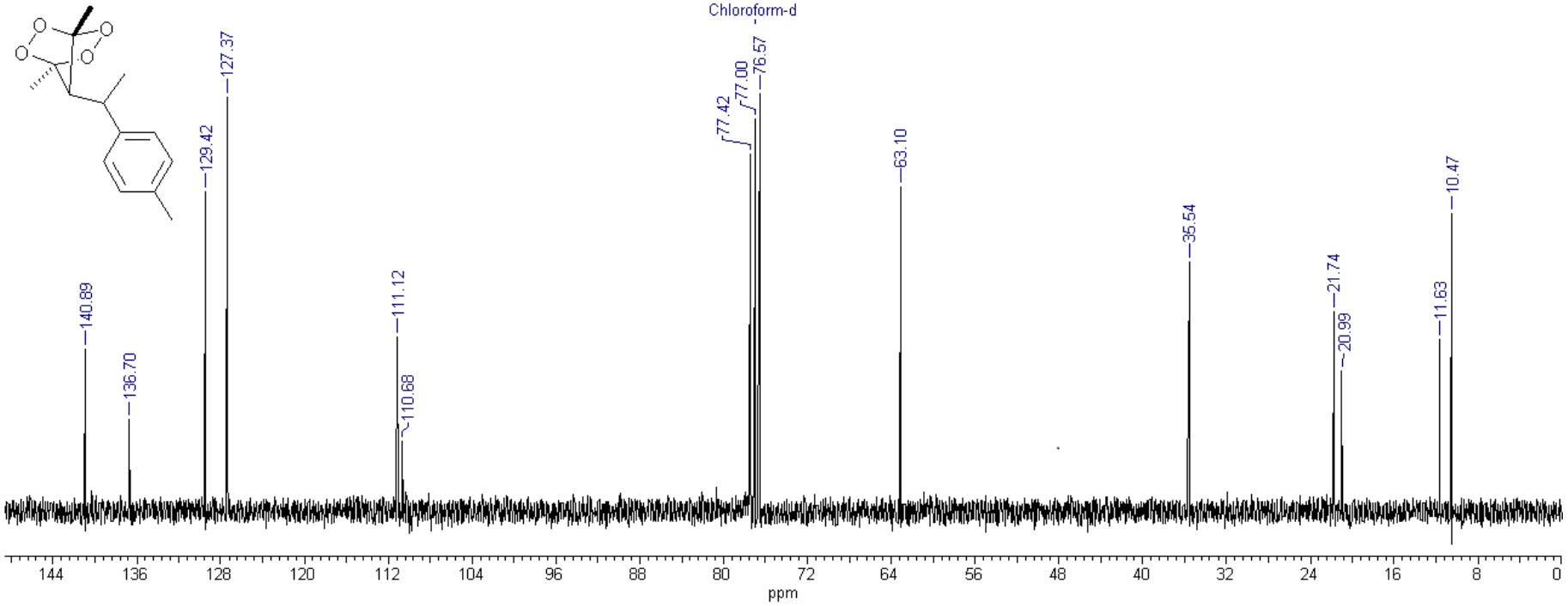
Acquisition Time (sec)	0.6759	***Comment***	NMR/50059953	***Date***	19 Apr 2012 14:28:16		
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	26.900						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

1,4-Dimethyl-7-[1-(4-methylphenyl)ethyl]-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2m)

Acquisition Time (sec)	0.4501	***Comment***	Avance-300, C-13, CDCl3			***Date***	18 Jan 2012 13:17:52
File Name						***Frequency (MHz)***	75.48
Nucleus	13C	***Number of Transients***	101	***Original Points Count***	16308	***Points Count***	16384
Pulse Sequence	zgpg30base	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	18115.94
Temperature (degree C)	26.500						



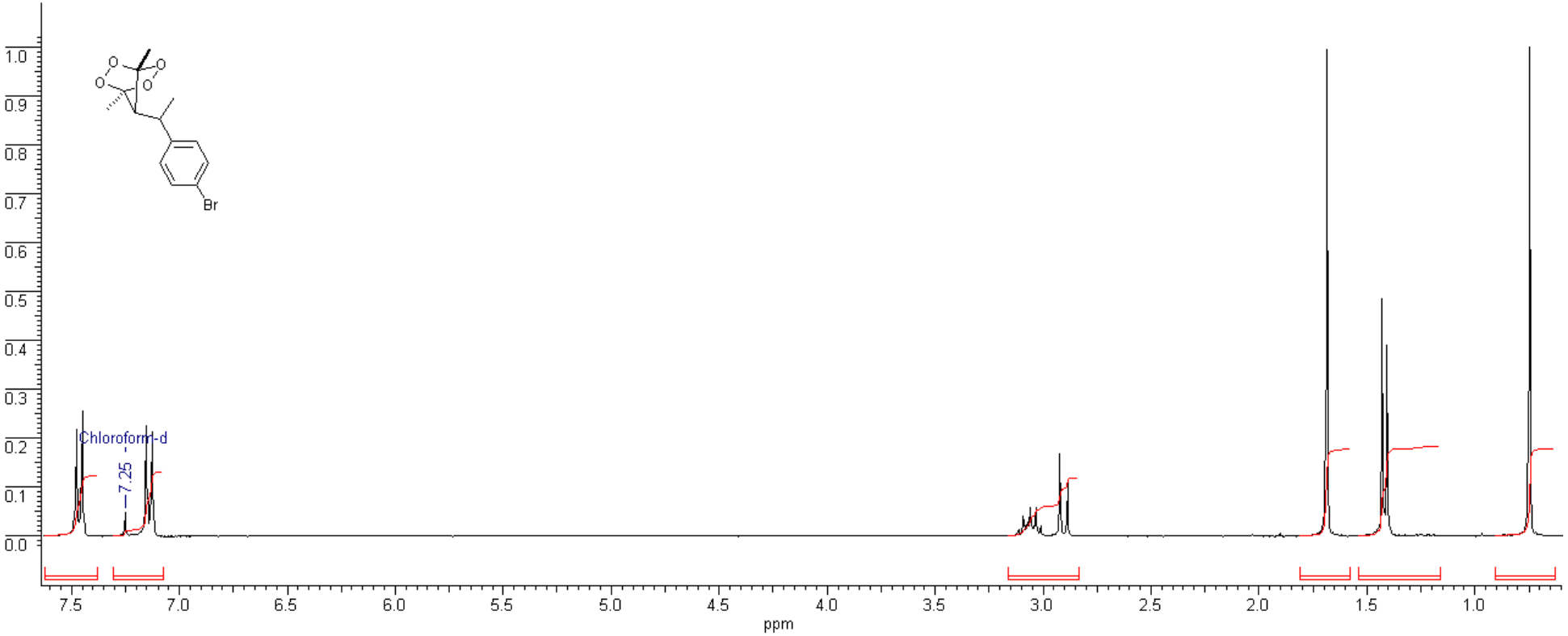
No.	(ppm)	(Hz)	Height
1	10.47	790.3	0.7108
2	11.63	877.6	0.4126
3	20.99	1584.2	0.3350
4	21.74	1640.6	0.4780
5	35.54	2682.3	0.5962
6	63.10	4762.2	0.7774
7	76.57	5779.6	1.0000
8	77.00	5811.6	0.9393

No.	(ppm)	(Hz)	Height
9	77.42	5843.7	0.8542
10	110.68	8353.8	0.1694
11	111.12	8387.0	0.4186
12	127.37	9613.3	0.9908
13	129.42	9768.1	0.7639
14	136.70	10317.7	0.2192
15	140.89	10633.9	0.3886

No.	Annotation	(ppm)
1	Chloroform-d	77.00

7-[1-(4-Bromophenyl)ethyl]-1,4-dimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2n)

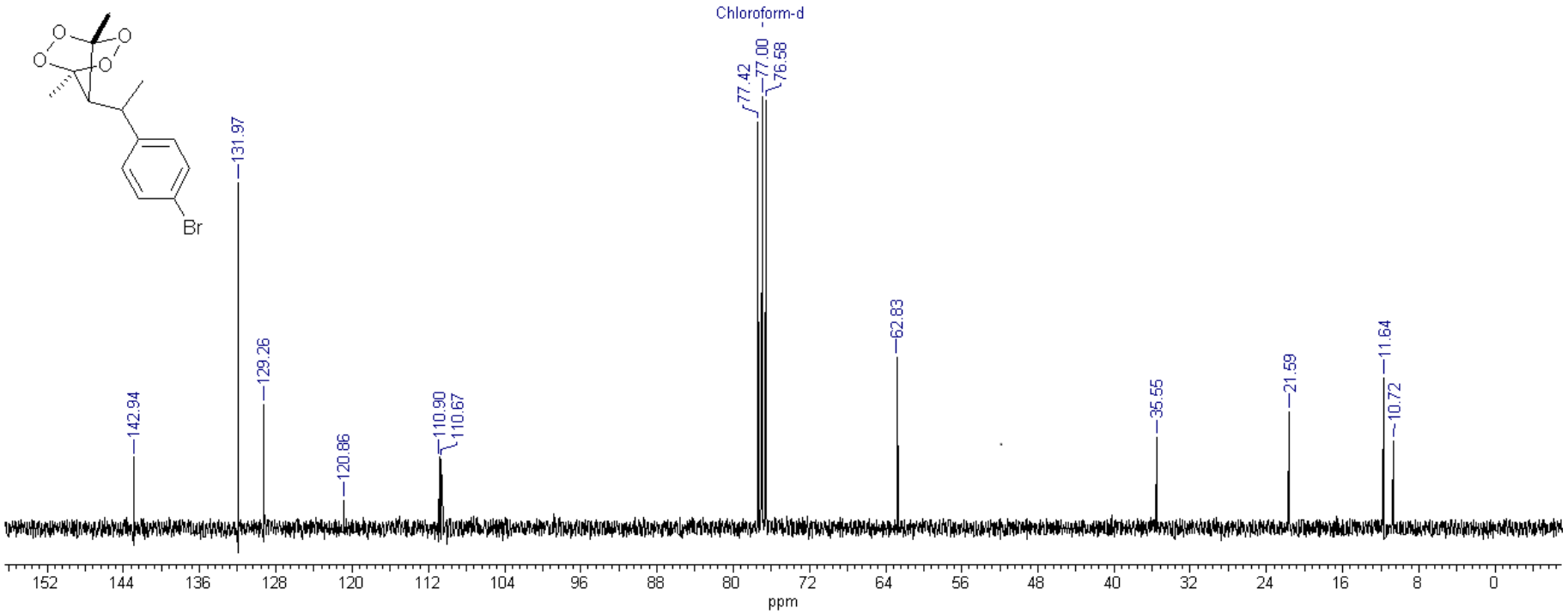
Acquisition Time (sec)	0.6759	***Comment***	NMR/50059953	***Date***	19 Apr 2012 14:41:04		
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	27.000						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

7-[1-(4-Bromophenyl)ethyl]-1,4-dimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptanes (2n)

Acquisition Time (sec)	0.4501	***Comment***	Avance-300, C-13, CDCB			***Date***	19 Apr 2012 14:43:12
File Name						***Frequency (MHz)***	75.48
Nucleus	13C	***Number of Transients***	218	***Original Points Count***	16308	***Points Count***	16384
Pulse Sequence	zgpg30base	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	18115.94
Temperature (degree C)	27.000						



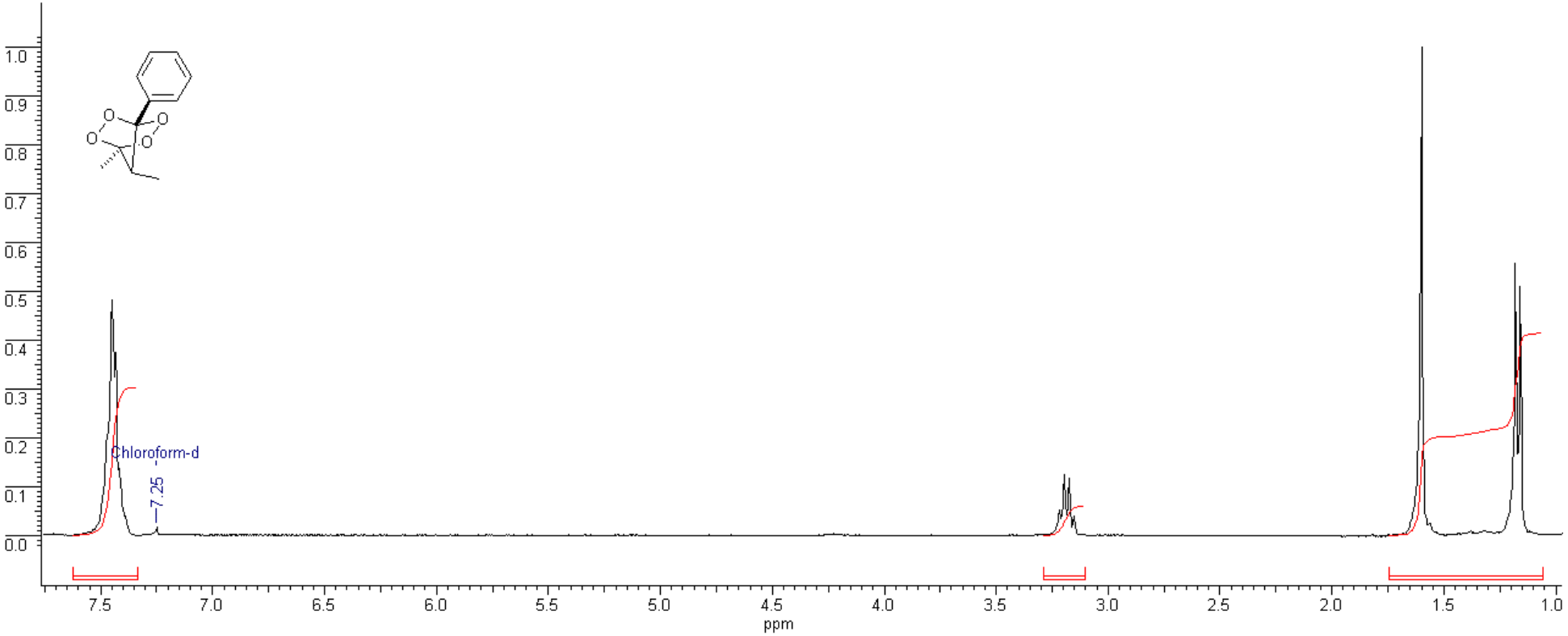
No.	(ppm)	(Hz)	Height
1	10.72	809.1	0.1999
2	11.64	878.8	0.3489
3	21.59	1629.6	0.2702
4	35.55	2683.4	0.2093
5	62.83	4742.4	0.3952
6	76.58	5779.6	0.9898
7	77.00	5811.7	1.0000

No.	(ppm)	(Hz)	Height
8	77.42	5843.7	0.9386
9	110.67	8352.7	0.1591
10	110.90	8370.4	0.1630
11	120.86	9122.4	0.0640
12	129.26	9756.0	0.2871
13	131.97	9960.5	0.7994
14	142.94	10788.8	0.1628

No.	Annotation	(ppm)
1	Chloroform-d	77.00

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

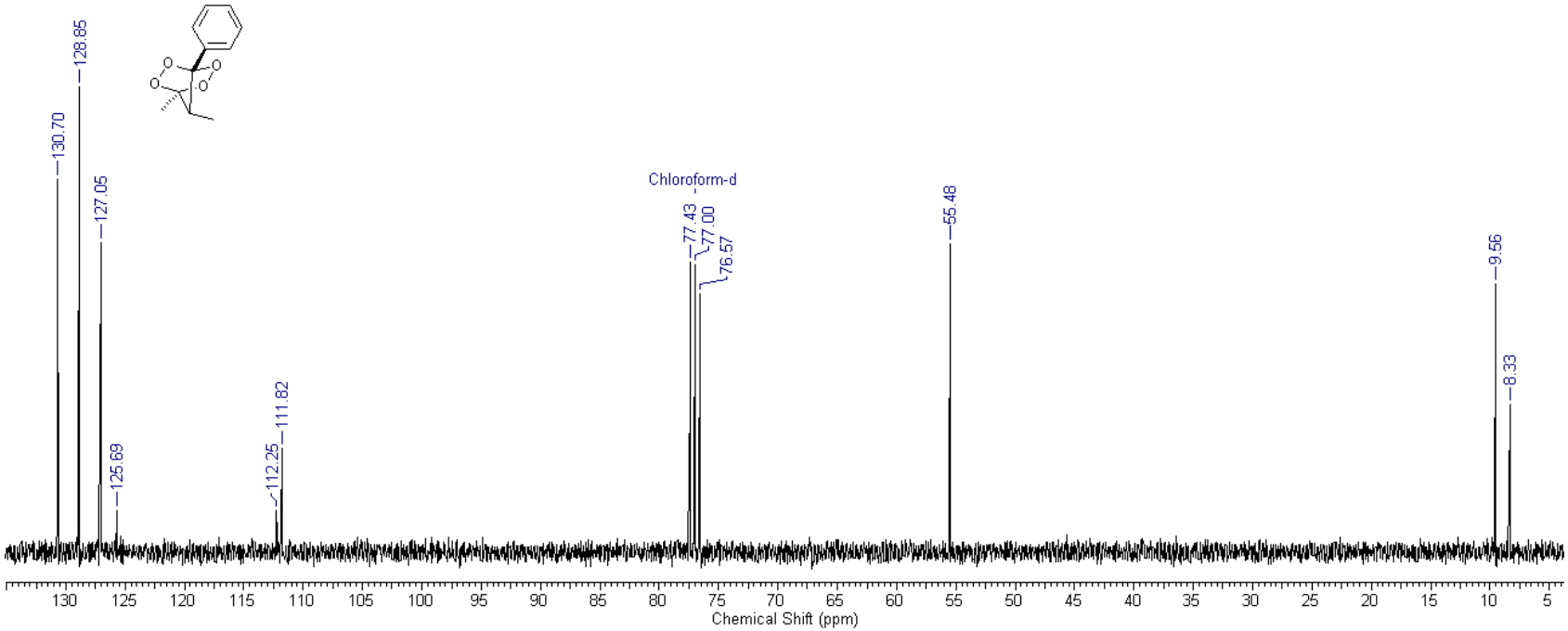
Acquisition Time (sec)	0.6759	***Comment***	/TSFC mk1324.32	***Date***	20 Jul 2012 12:13:52		
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	30.000						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

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

Acquisition Time (sec)	0.8643	***Comment***	Avance-300, C-13, CDCl3			***Date***	20 Jul 2012 12:18:08
File Name						***Frequency (MHz)***	75.48
Nucleus	13C	***Number of Transients***	128	***Original Points Count***	16308	***Points Count***	16384
Pulse Sequence	zgpg60base	***Solvent***	CHLOROFORM-D	***Sweep Width (Hz)***	18867.92	***Temperature (degree C)***	30.000

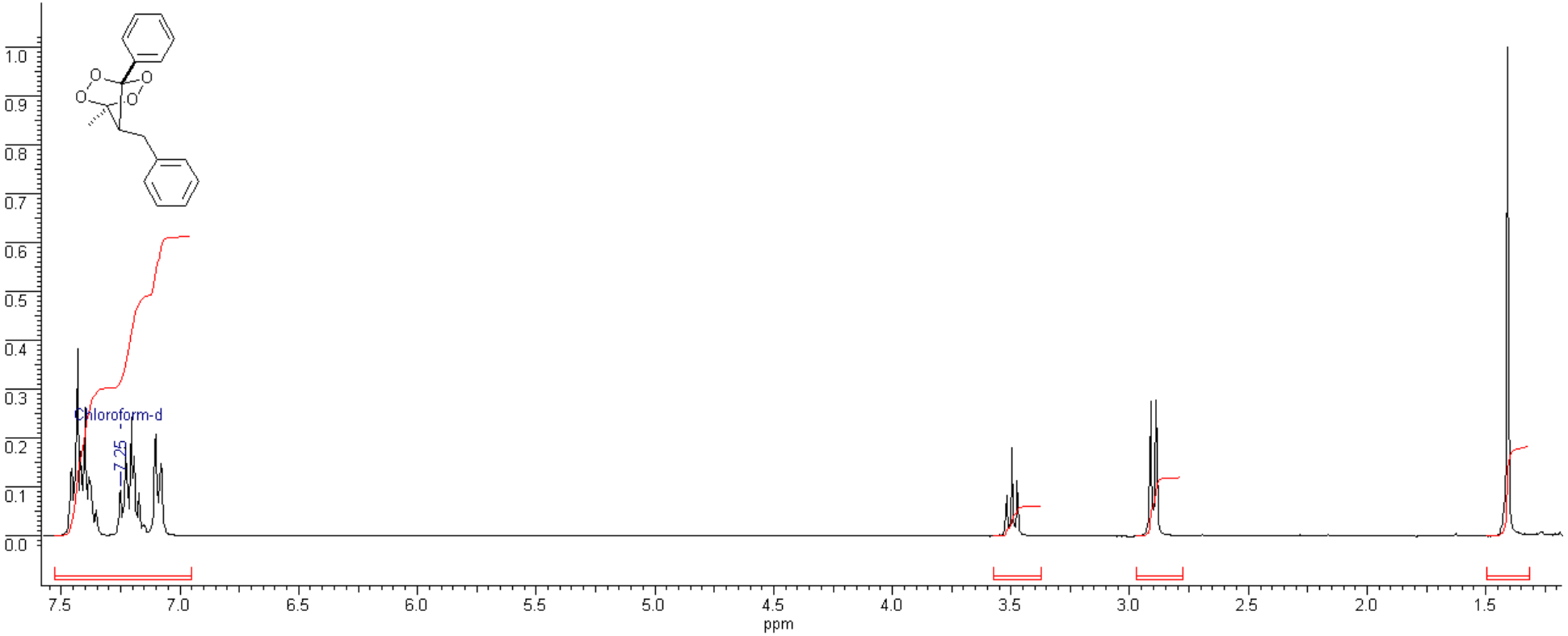


No.	(ppm)	(Hz)	Height
1	8.33	629.1	0.3163
2	9.56	721.2	0.5757
3	55.48	4187.8	0.6632
4	76.57	5779.4	0.5547
5	77.00	5811.6	0.6193
6	77.43	5843.9	0.6224
7	111.82	8439.8	0.2216
8	112.25	8472.0	0.0877
9	125.69	9486.6	0.0900
10	127.05	9589.1	0.6653
11	128.85	9725.0	1.0000
12	130.70	9864.4	0.8021

No.	Annotation	(ppm)
1	Chloroform-d	77.00

7-Benzyl-1-methyl-4-phenyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2p)

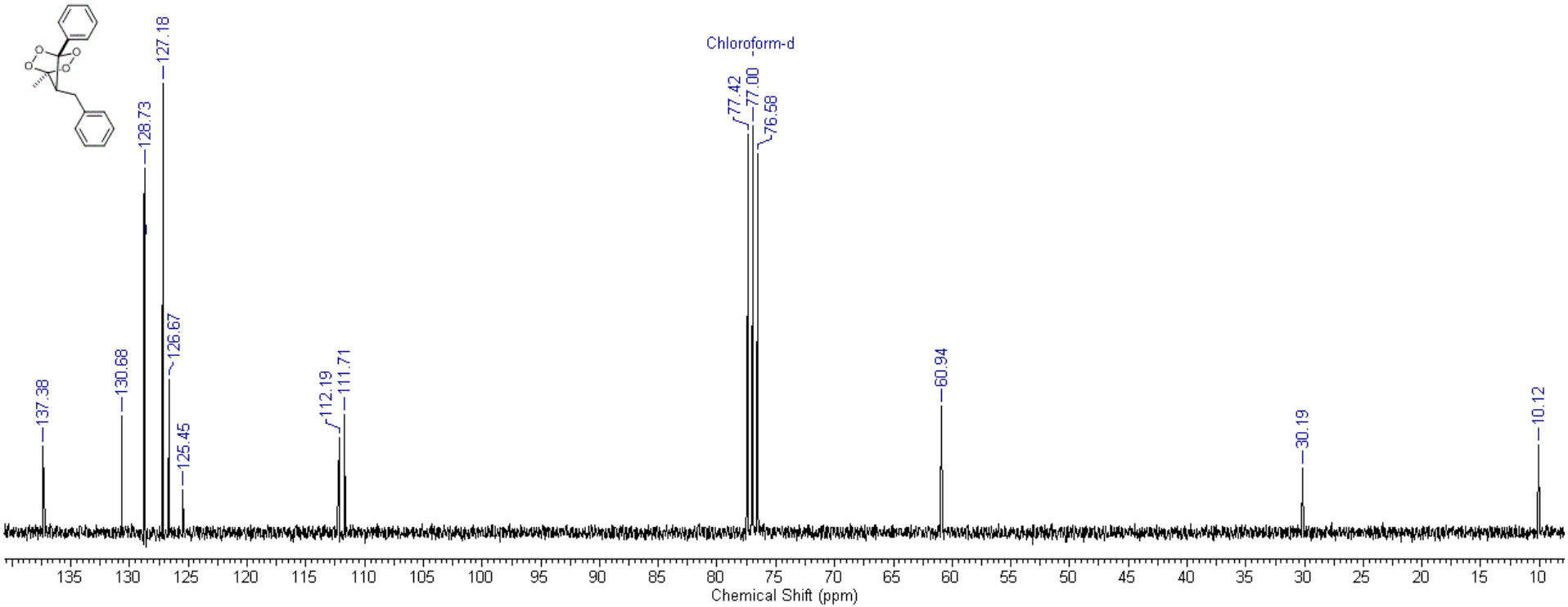
Acquisition Time (sec)	0.6759	*Comment*	NMR/50059953	*Date*	30 May 2012 15:36:32		
File Name						*Frequency (MHz)*	300.13
Nucleus	1H	*Number of Transients*	1	*Original Points Count*	8124	*Points Count*	8192
Pulse Sequence	zg	*Solvent*	CHLOROFORM-D			*Sweep Width (Hz)*	6009.62
Temperature (degree C)	26.700						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

7-Benzyl-1-methyl-4-phenyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2p)

Acquisition Time (sec)	0.9002	***Comment***	Avance-300, C-13, CDCl3			***Date***	30 May 2012 15:38:40
File Name						***Frequency (MHz)***	75.48
Nucleus	13C	***Number of Transients***	245	***Original Points Count***	16308	***Points Count***	16384
Pulse Sequence	zgpg30base	***Solvent***	CHLOROFORM-D	***Sweep Width (Hz)***	18115.94	***Temperature (degree C)***	26.800



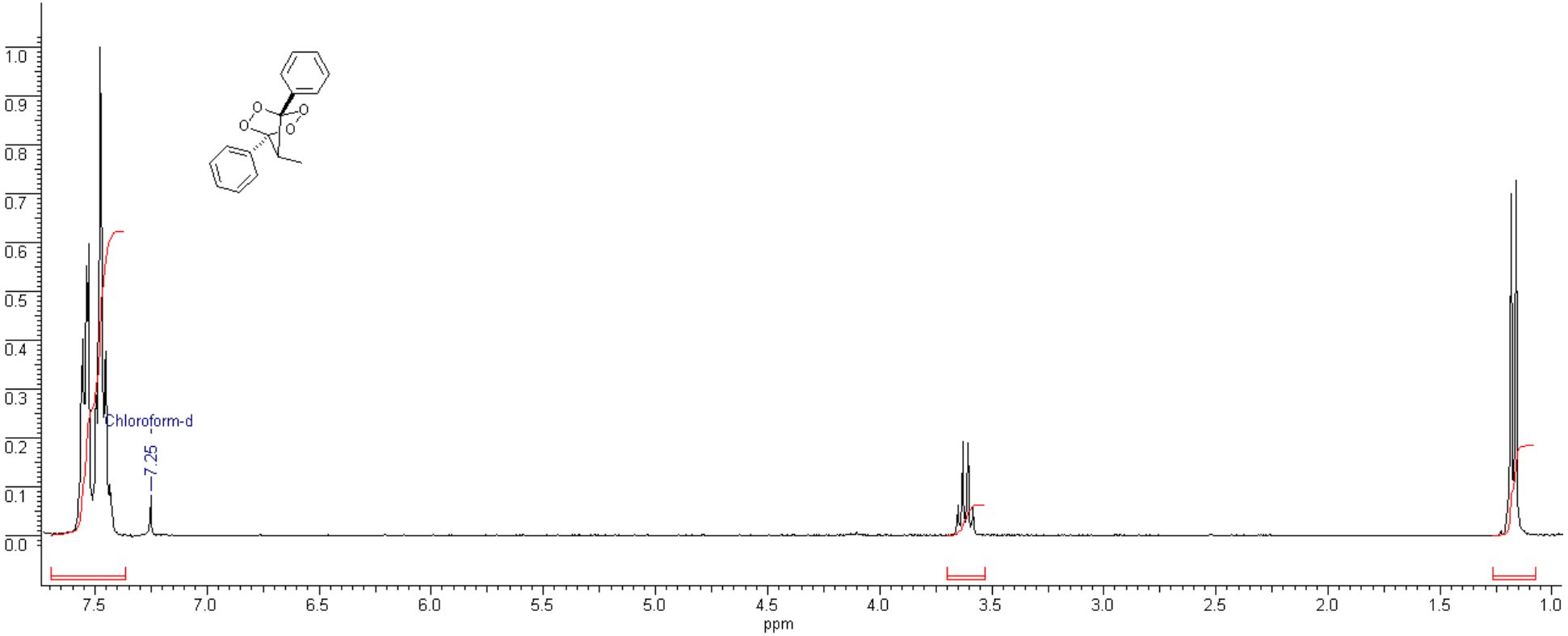
No.	(ppm)	(Hz)	Height
1	10.12	763.8	0.1954
2	30.19	2278.7	0.1434
3	60.94	4599.7	0.2812
4	76.58	5779.6	0.8442
5	77.00	5811.7	0.9062
6	77.42	5843.7	0.8861
7	111.71	8431.2	0.2623
8	112.19	8467.7	0.2120

No.	(ppm)	(Hz)	Height
9	125.45	9468.5	0.0962
10	126.67	9560.2	0.3411
11	127.18	9598.9	1.0000
12	128.61	9707.3	0.6548
13	128.73	9716.2	0.8115
14	128.79	9720.6	0.6982
15	130.68	9863.2	0.2606
16	137.38	10368.6	0.1924

No.	Annotation	(ppm)
1	Chloroform-d	77.00

1,4-Diphenyl-7-methyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2q)

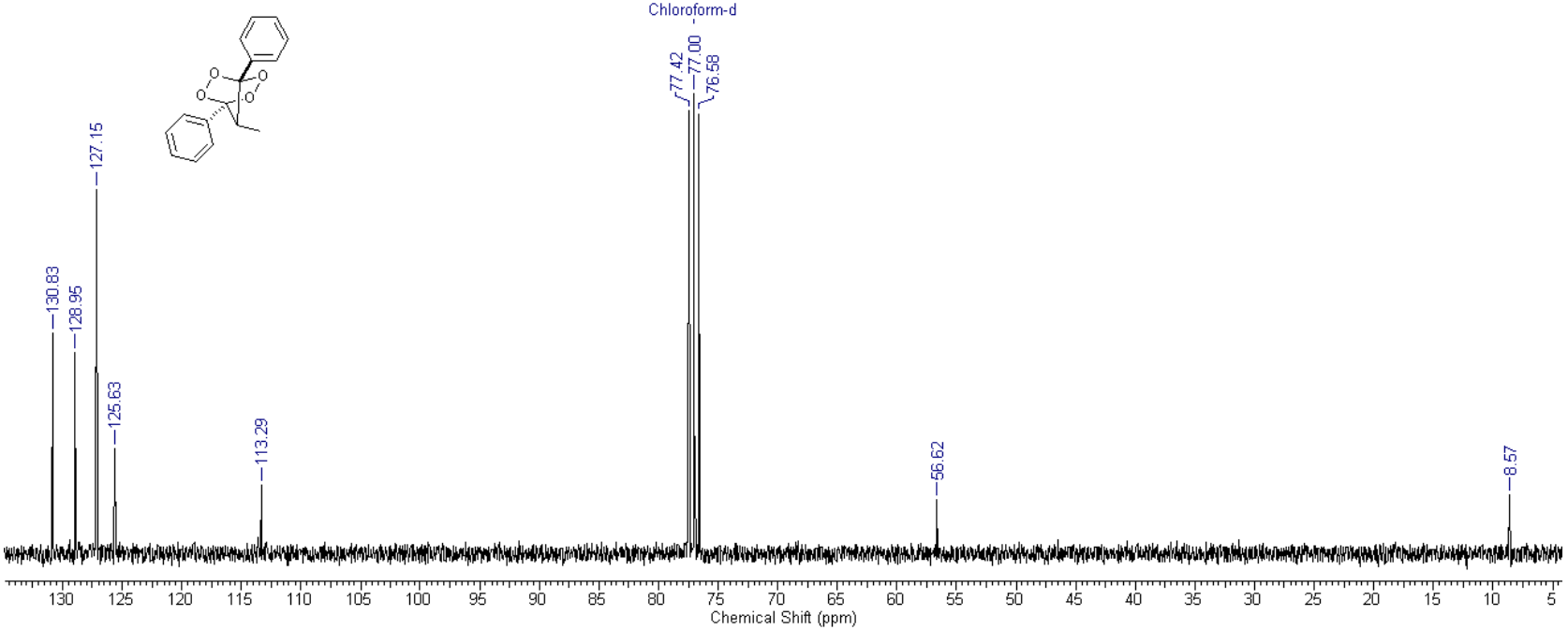
Acquisition Time (sec)	0.6759	***Comment***	IGOR NH0023	***Date***	19 Jul 2012 14:09:04		
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	27.600						



No.	Annotation	(ppm)
1	Chloroform-d	7.25

1,4-Diphenyl-7-methyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2q)

Acquisition Time (sec)	0.9002	***Comment***	Avance-300, C-13, CDCB	***Date***	19 Jul 2012 14:09:04
File Name				***Frequency (MHz)***	75.48
Nucleus	13C	***Number of Transients***	226	***Original Points Count***	16308	***Points Count***	16384
Pulse Sequence	zgpg30base	***Solvent***	CHLOROFORM-D	***Sweep Width (Hz)***	18115.94
Temperature (degree C)	27.600				

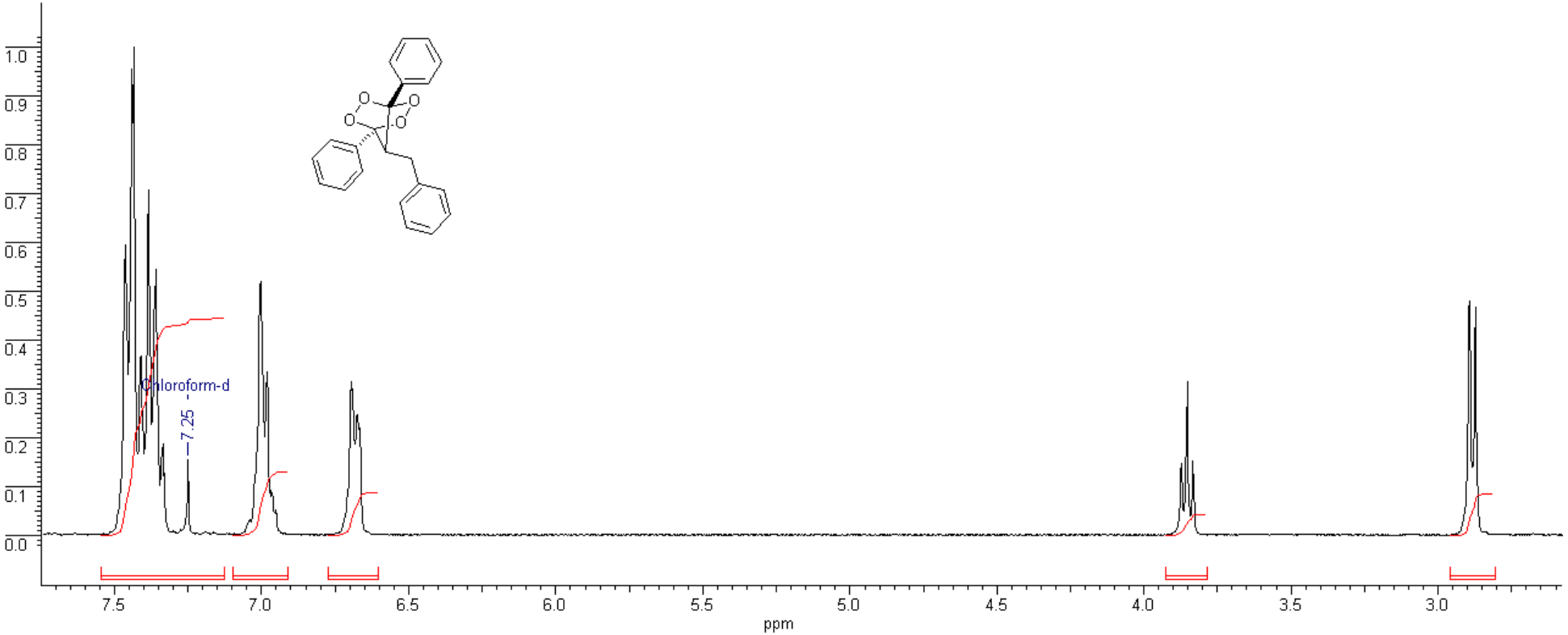


No.	(ppm)	(Hz)	Height
1	8.57	646.6	0.1273
2	56.62	4273.5	0.1169
3	76.58	5779.6	0.9565
4	77.00	5811.7	1.0000
5	77.42	5843.7	0.9634
6	113.29	8550.7	0.1487
7	125.63	9481.7	0.2292
8	127.15	9596.7	0.7896
9	128.95	9732.7	0.4370
10	130.83	9874.3	0.4798

No.	Annotation	(ppm)
1	Chloroform-d	77.00

7-Benzyl-1,4-diphenyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2r)

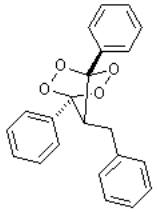
Acquisition Time (sec)	0.6759	*Comment*	IGOR NH0023	*Date*	30 Jul 2012 09:44:32		
File Name						*Frequency (MHz)*	300.13
Nucleus	1H	*Number of Transients*	1	*Original Points Count*	8124	*Points Count*	8192
Pulse Sequence	zg	*Solvent*	CHLOROFORM-D			*Sweep Width (Hz)*	6009.62
Temperature (degree C)	27.000						

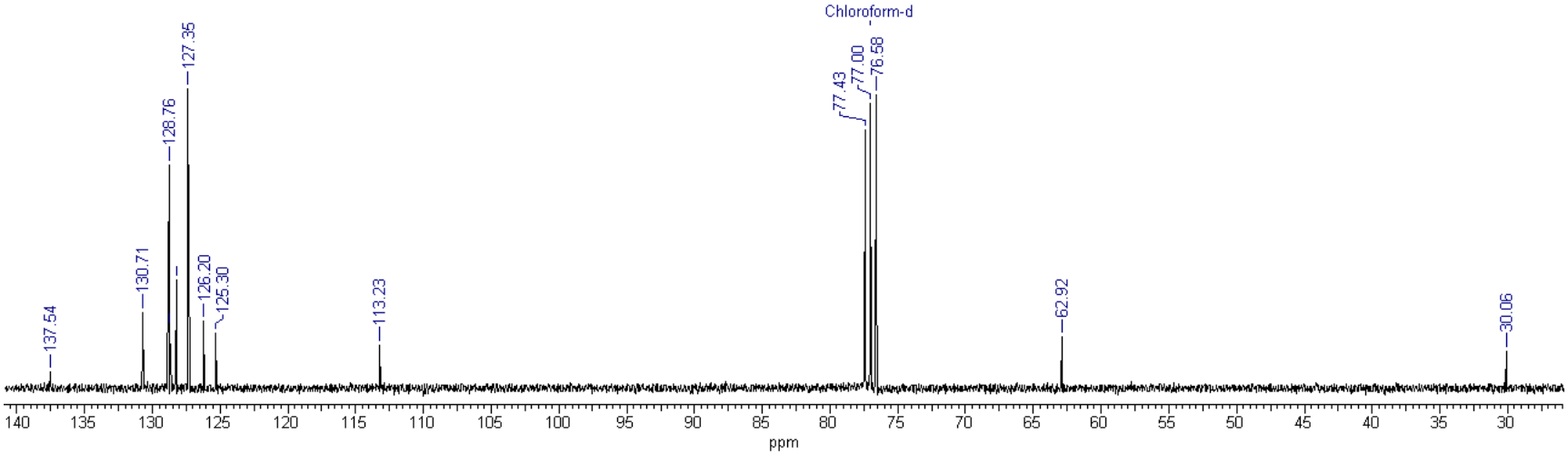


No.	Annotation	(ppm)
1	Chloroform-d	7.25

7-Benzyl-1,4-diphenyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2r)

Acquisition Time (sec)	0.4501	***Comment***	Avance-300, C-13, CDCl3			***Date***	30 Jul 2012 09:50:56
File Name						***Frequency (MHz)***	75.48
Nucleus	13C	***Number of Transients***	367	***Original Points Count***	16308	***Points Count***	16384
Pulse Sequence	zgpg30base	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	18115.94
Temperature (degree C)	28.000						





No.	(ppm)	(Hz)	Height
1	30.06	2268.8	0.1239
2	62.92	4749.0	0.1710
3	76.58	5779.6	0.9821
4	77.00	5811.7	0.9541
5	77.43	5843.7	0.8646
6	113.23	8546.3	0.1461
7	125.30	9457.4	0.1844

No.	(ppm)	(Hz)	Height
8	126.20	9524.9	0.2227
9	127.35	9612.2	1.0000
10	128.19	9675.3	0.3617
11	128.69	9712.9	0.2052
12	128.76	9718.4	0.7473
13	130.71	9865.5	0.2547
14	137.54	10380.7	0.0528

No.	Annotation	(ppm)
1	Chloroform-d	77.00

HRMS of β-diketone

3-(1-Adamantyl)-6-methylheptane-2,4-dione (1j)

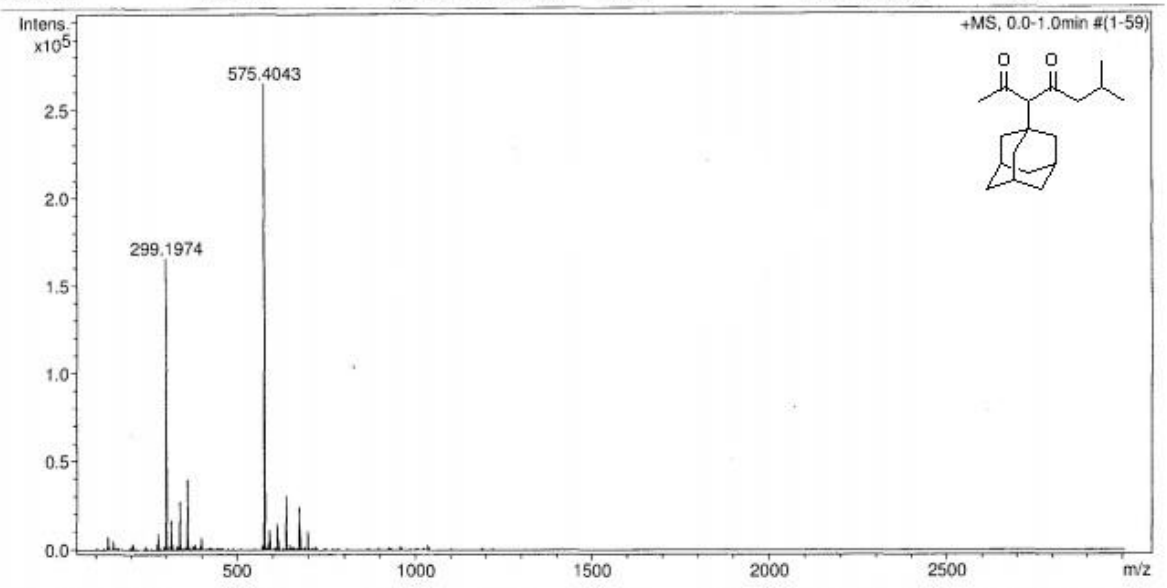
Display Report

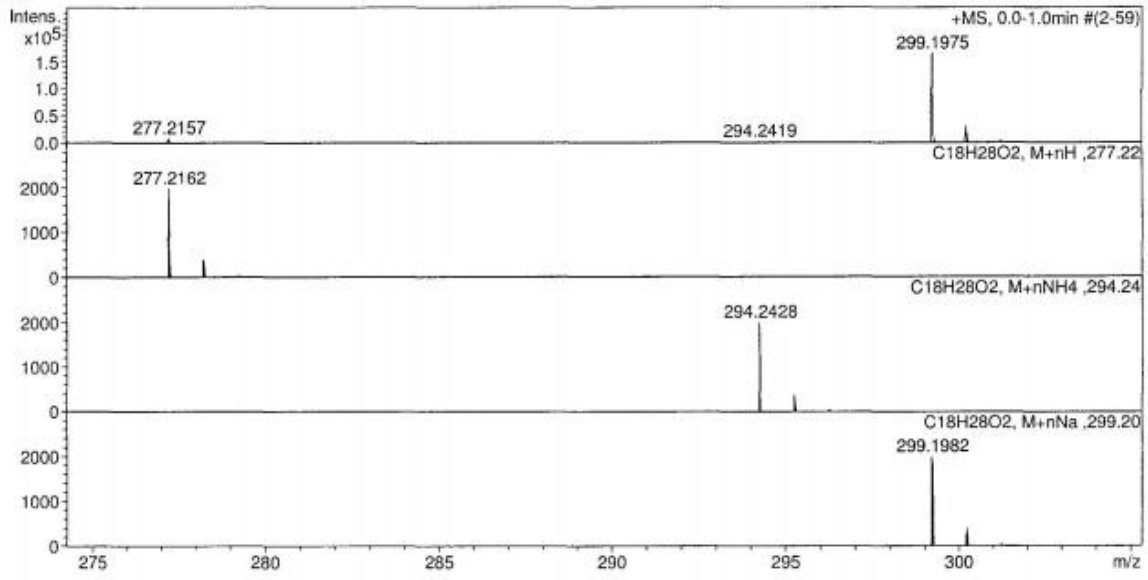
Analysis Info

Analysis Name	D:\Data\Chizhov\Nikishin\Terentiev\Yaremenko\b-426_low.d	Acquisition Date	6/4/2012 4:21:43 PM
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN B-426	Instrument / Ser#	micrOTOF 10248
Comment	CH3CN 100 %, dil. 200, no calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste





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HRMS of tetraoxanes

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

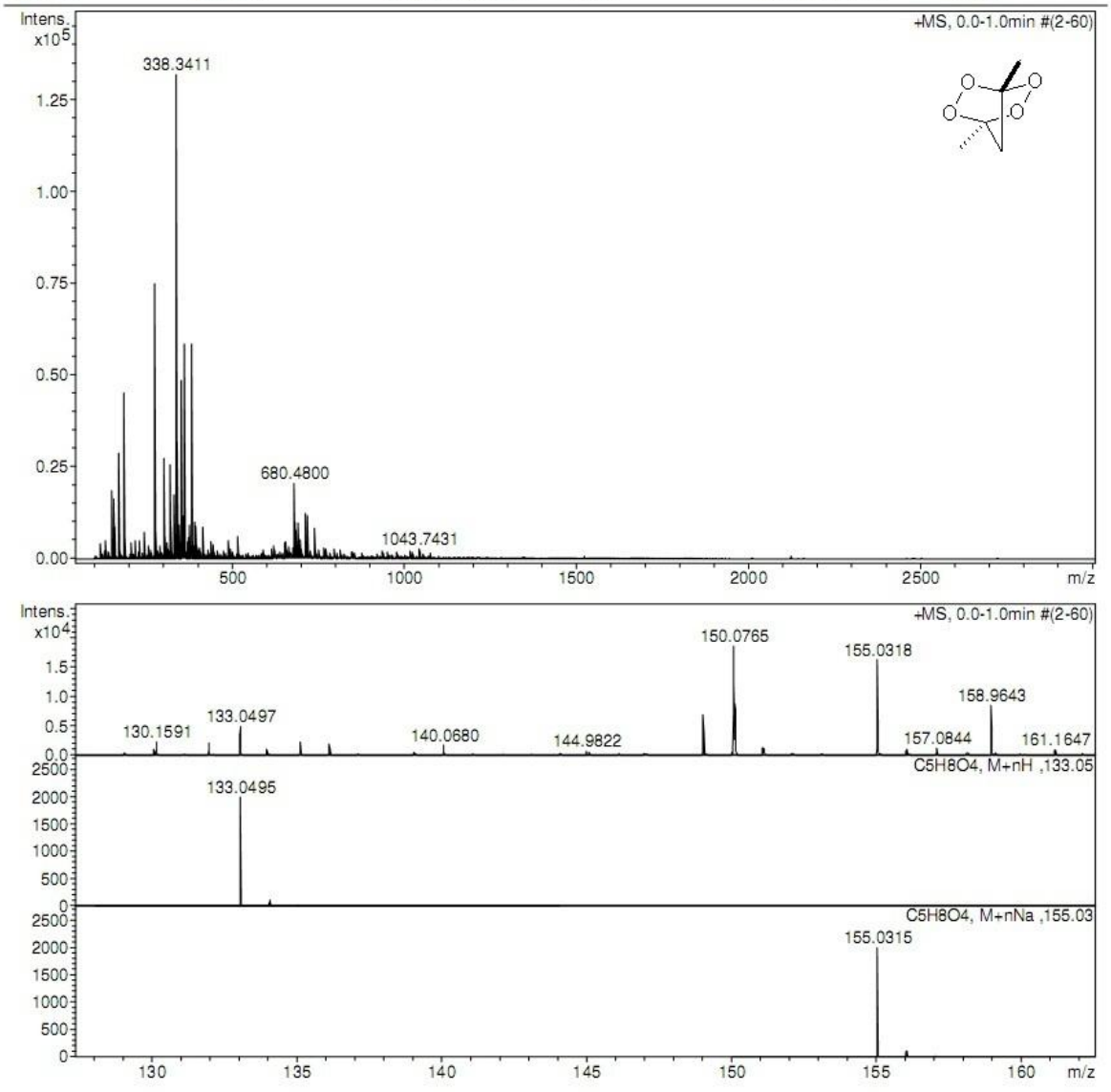
Display Report

Analysis Info

Analysis Name	D:\Data\Chizhov\Nikishin\Terentiev\Yaremenko\bb201&clblow.d	Acquisition Date	5/11/2012 2:57:00 PM
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN BB201	Instrument / Ser#	micrOTOF 10248
Comment	CH3CN 100 %, dil. > 1, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Bruker Compass DataAnalysis 4.0 printed: 5/11/2012 2:59:36 PM Page 1 of 1

1-Isobutyl-4-methyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2b)

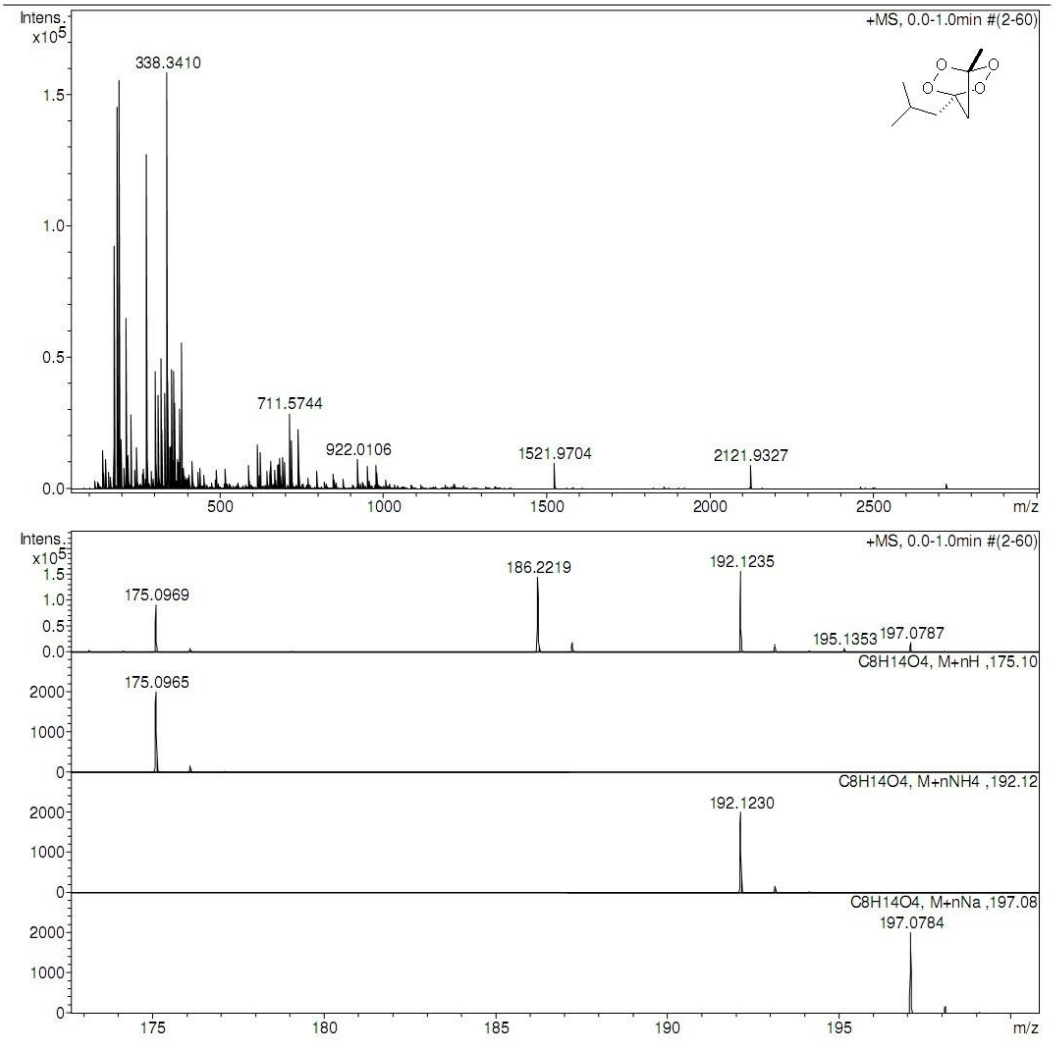
Display Report

Analysis Info

Analysis Name	D:\Data\Chizhov\Nikishin\Terentiev\Yaremenko\b419_&clblow.d	Acquisition Date	4/23/2012 5:16:47 PM
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN B-419	Instrument / Ser#	micrOTOF 10248
Comment	CH3CN 100 %, dil. 200, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Bruker Compass DataAnalysis 4.0 printed: 4/23/2012 5:21:16 PM Page 1 of 1

1-Ethyl-4-isobutyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2c)

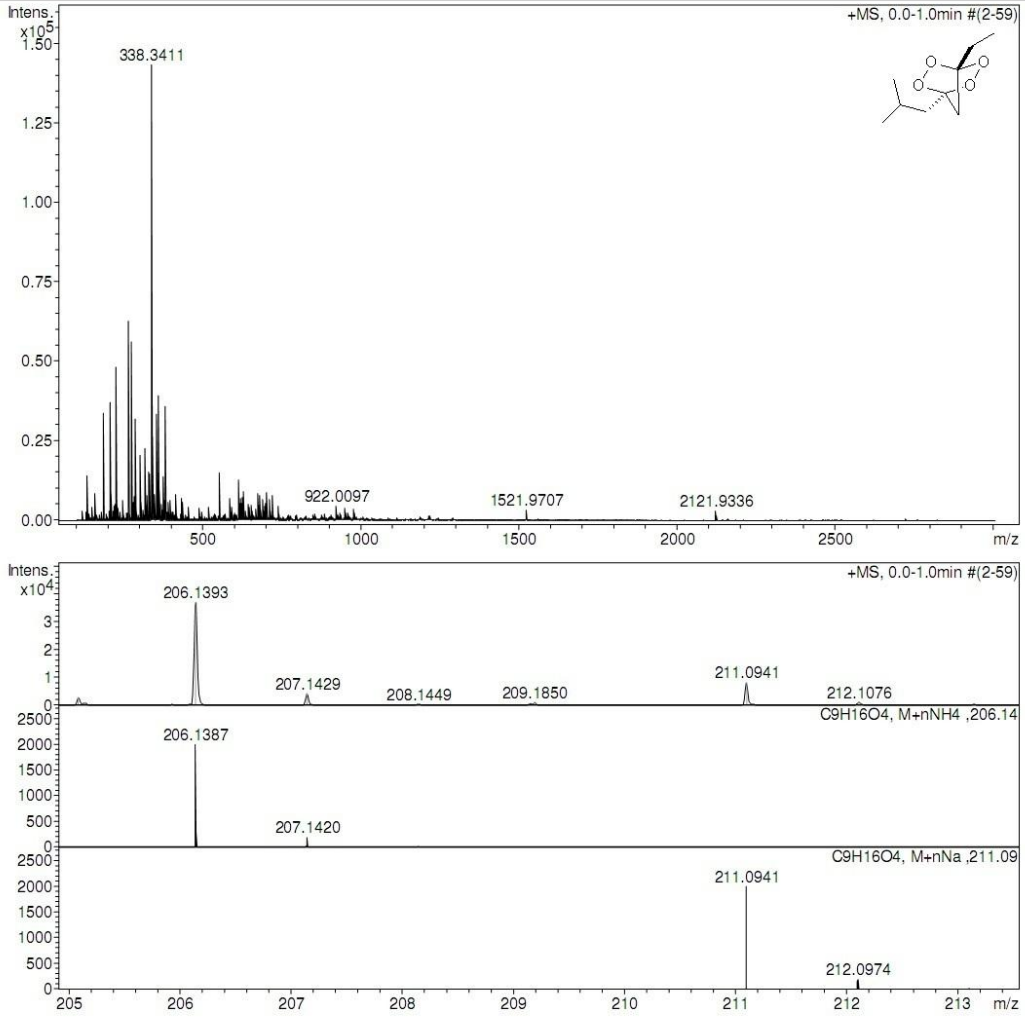
Display Report

Analysis Info

Analysis Name	D:\Data\Chizhov\Nikishin\Terentiev\Yaremenko\bb204_&clblow.d	Acquisition Date	5/11/2012 3:38:04 PM
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN BB204	Instrument / Ser#	micrOTOF 10248
Comment	CH3CN 100 %, dil. 200, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Bruker Compass DataAnalysis 4.0 printed: 5/11/2012 3:42:04 PM Page 1 of 1

1,4-Di-*tert*-butyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2d)

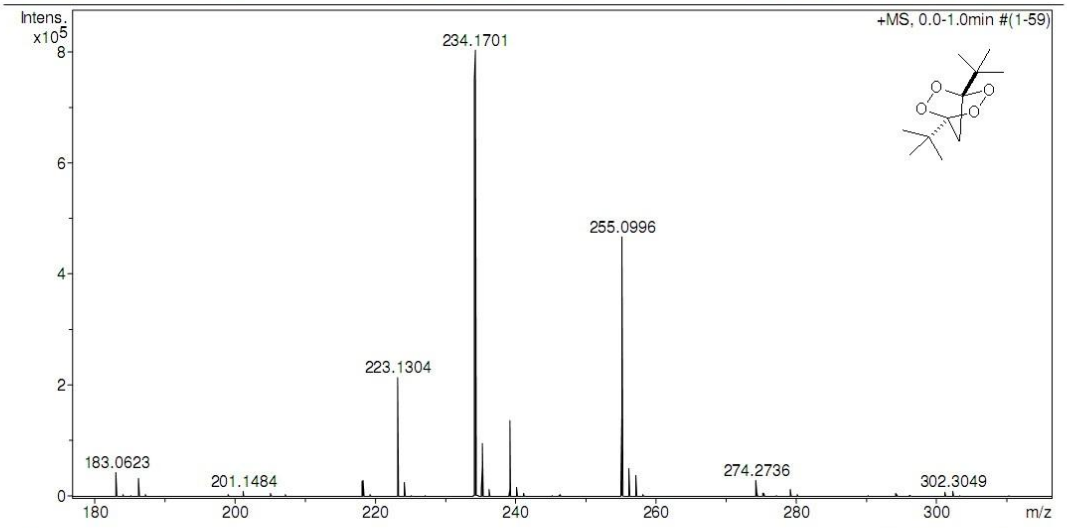
Display Report

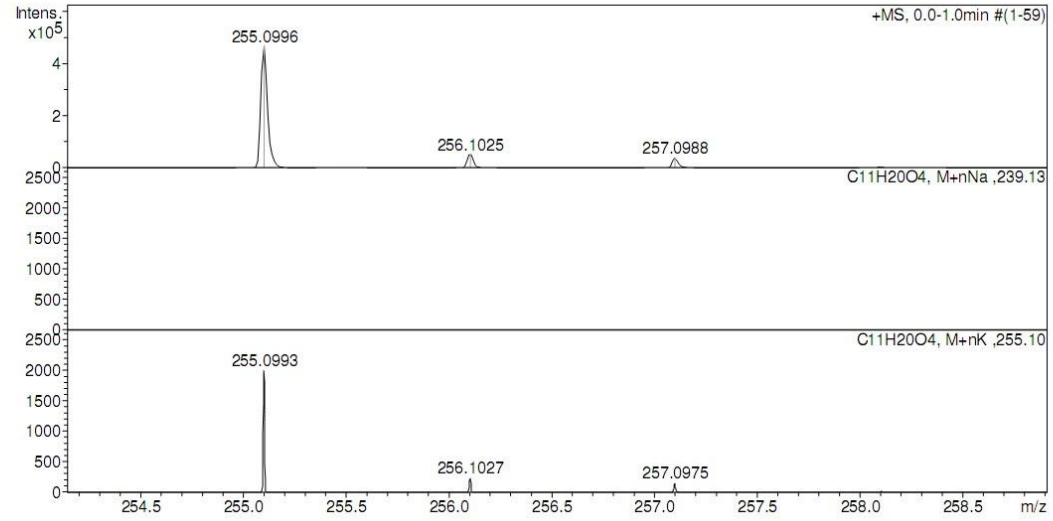
Analysis Info

Analysis Name	D:\Data\Chizhov\Nikishin\Terentiev\Yaremenko\bb-226&clblow.d	Acquisition Date	6/18/2012 5:41:58 PM
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN BB-226	Instrument / Ser#	micrOTOF 10248
Comment	CH3CN 100 %, dil. 2, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste





Bruker Compass DataAnalysis 4.0 printed: 6/18/2012 5:47:44 PM Page 1 of 1

1-(1-Adamantyl)-4-methyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2e)

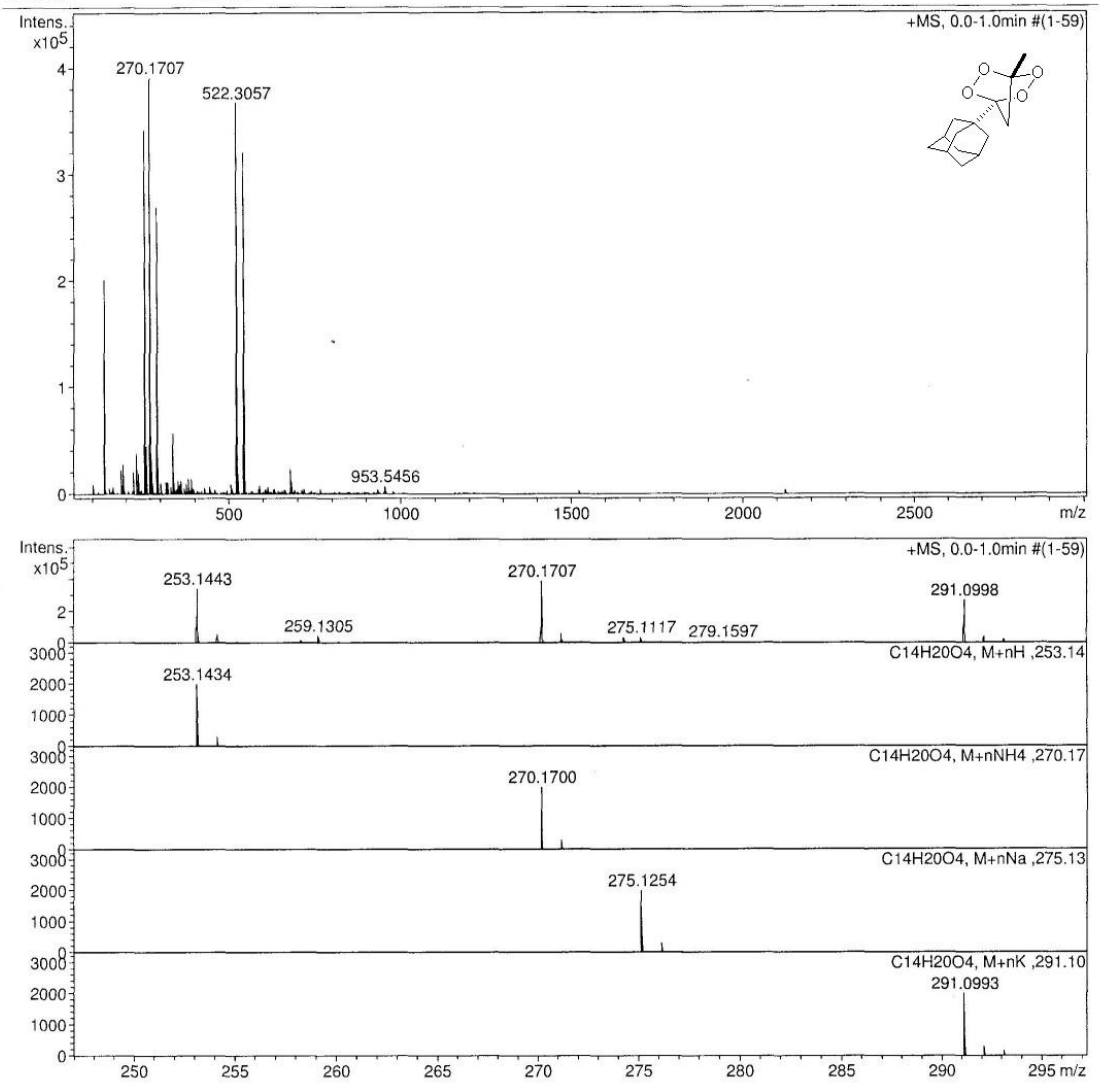
Display Report

Analysis Info

Analysis Name	D:\Data\Chizhov\Nikishin\Terentiev\b364&clb$low.d	Acquisition Date	1/16/2012 11:22:44 AM
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN B-364	Instrument / Ser#	micrOTOF 10248
Comment	CH3CN 100 %, dil. 2, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Bruker Compass DataAnalysis 4.0 printed: 1/16/2012 11:30:33 AM Page 1 of 1

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

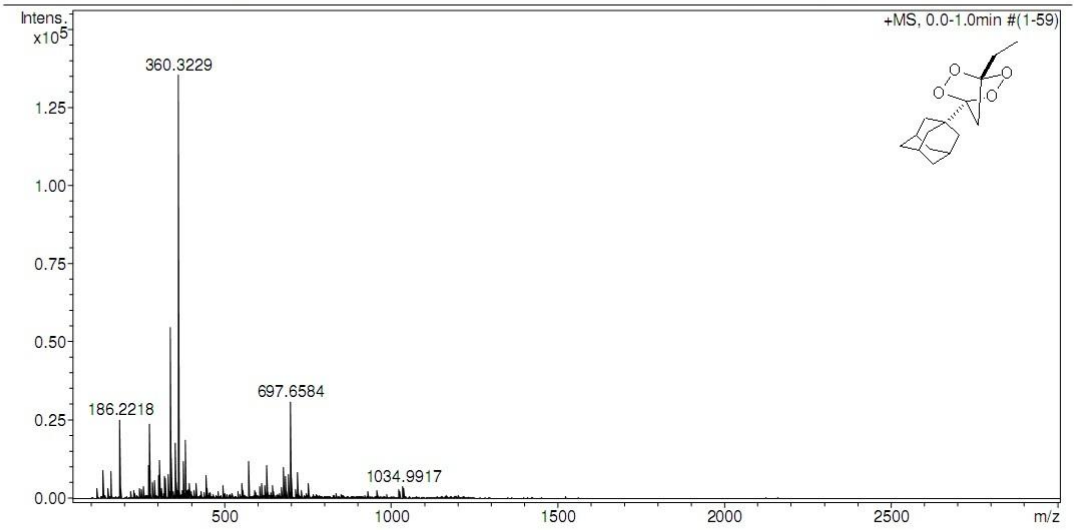
Display Report

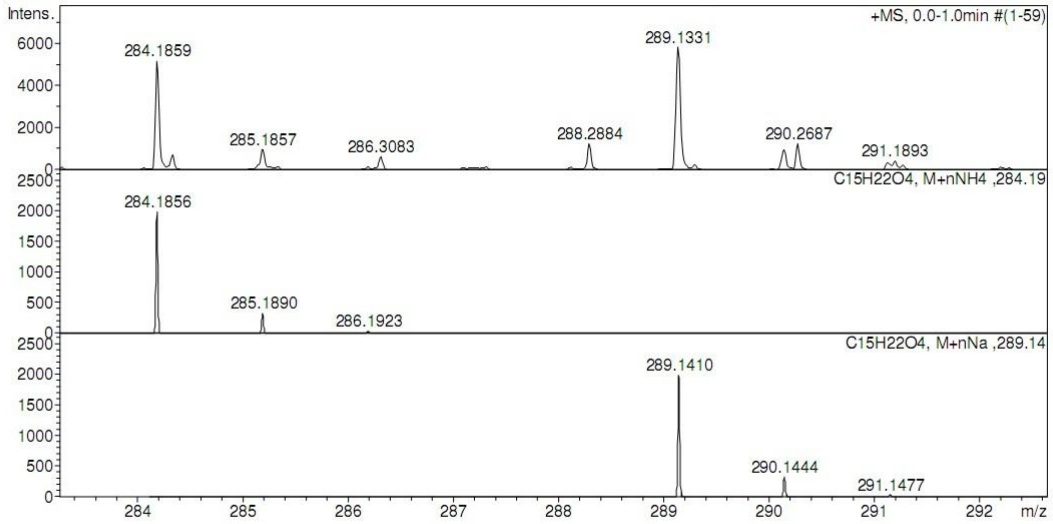
Analysis Info

Acquisition Date	5/21/2012 2:43:07 PM		
Analysis Name	D:\Data\Chizhov\Nikishin\Terentiev\Yaremenko\bb-149_&clblow.d		
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN BB-149	Instrument / Ser#	micrOTOF 10248
Comment	CH3CN 100 %, dil. 200, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste





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

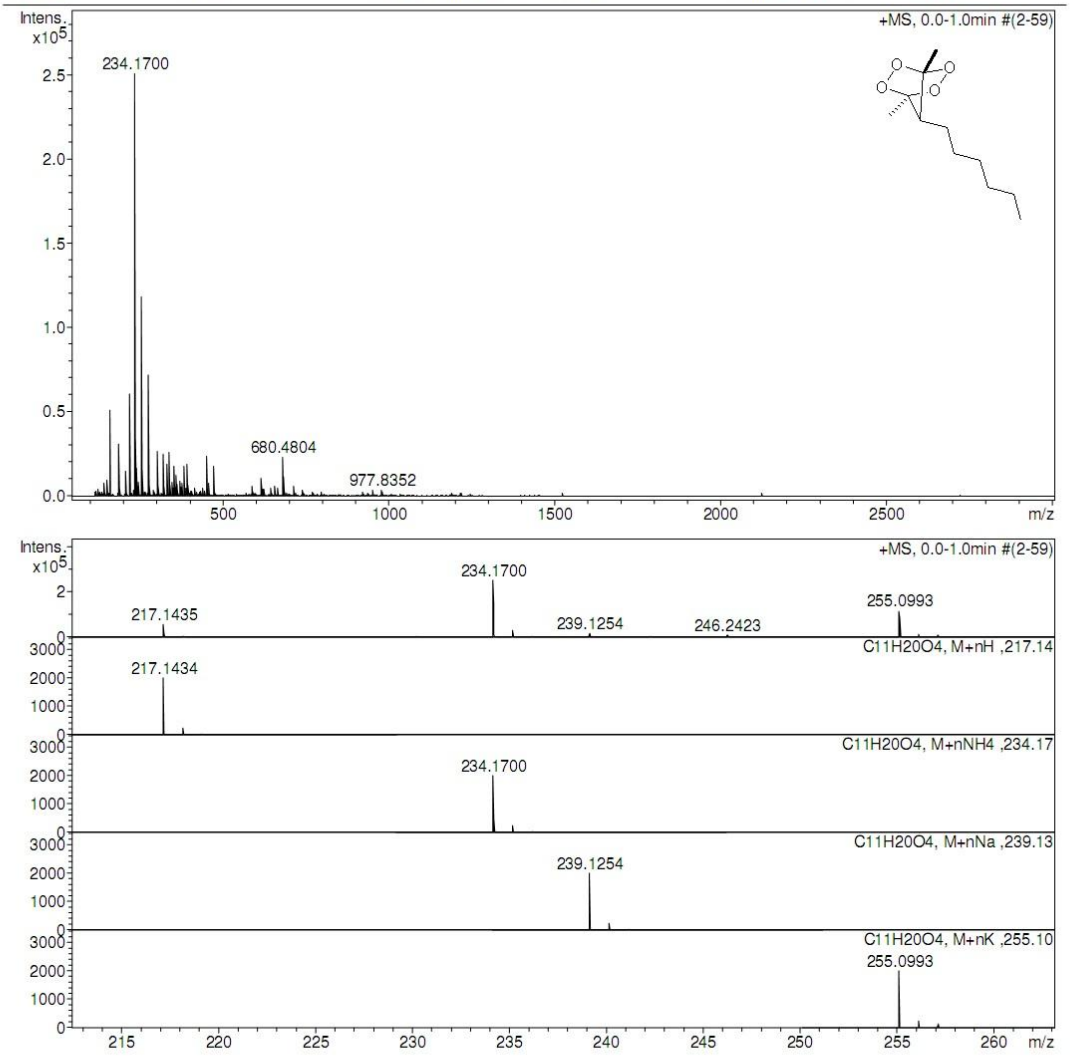
Display Report

Analysis Info

Analysis Name	D:\Data\Chizhov\Nikishin\Terentiev\Yaremenko\bb207_&clblow.d	Acquisition Date	5/25/2012 4:55:49 PM
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN BB207	Instrument / Ser#	micrOTOF 10248
Comment	CH3CN 100 %, dil. 200, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Bruker Compass DataAnalysis 4.0 printed: 5/25/2012 4:58:31 PM Page 1 of 1

7-(1-Adamantyl)-1-isobutyl-4-methyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2j)

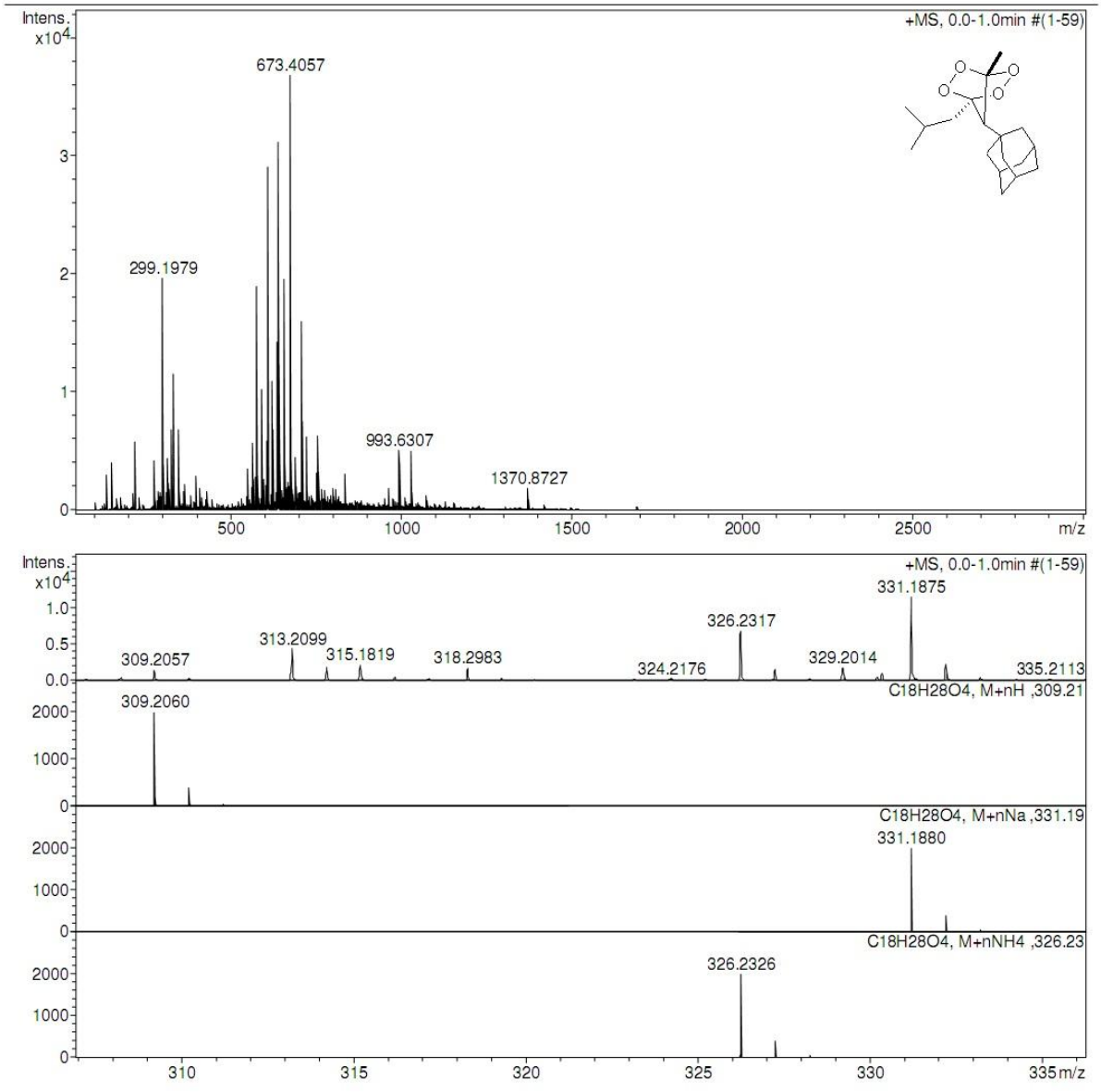
Display Report

Analysis Info

Analysis Name	D:\Data\Chizhov\Nikishin\Terentiev\Yaremenko\bb-208low.d	Acquisition Date	6/4/2012 3:10:53 PM
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN BB-208	Instrument / Ser#	micrOTOF 10248
Comment	CH3CN 100 %, dil. 1, no calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



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

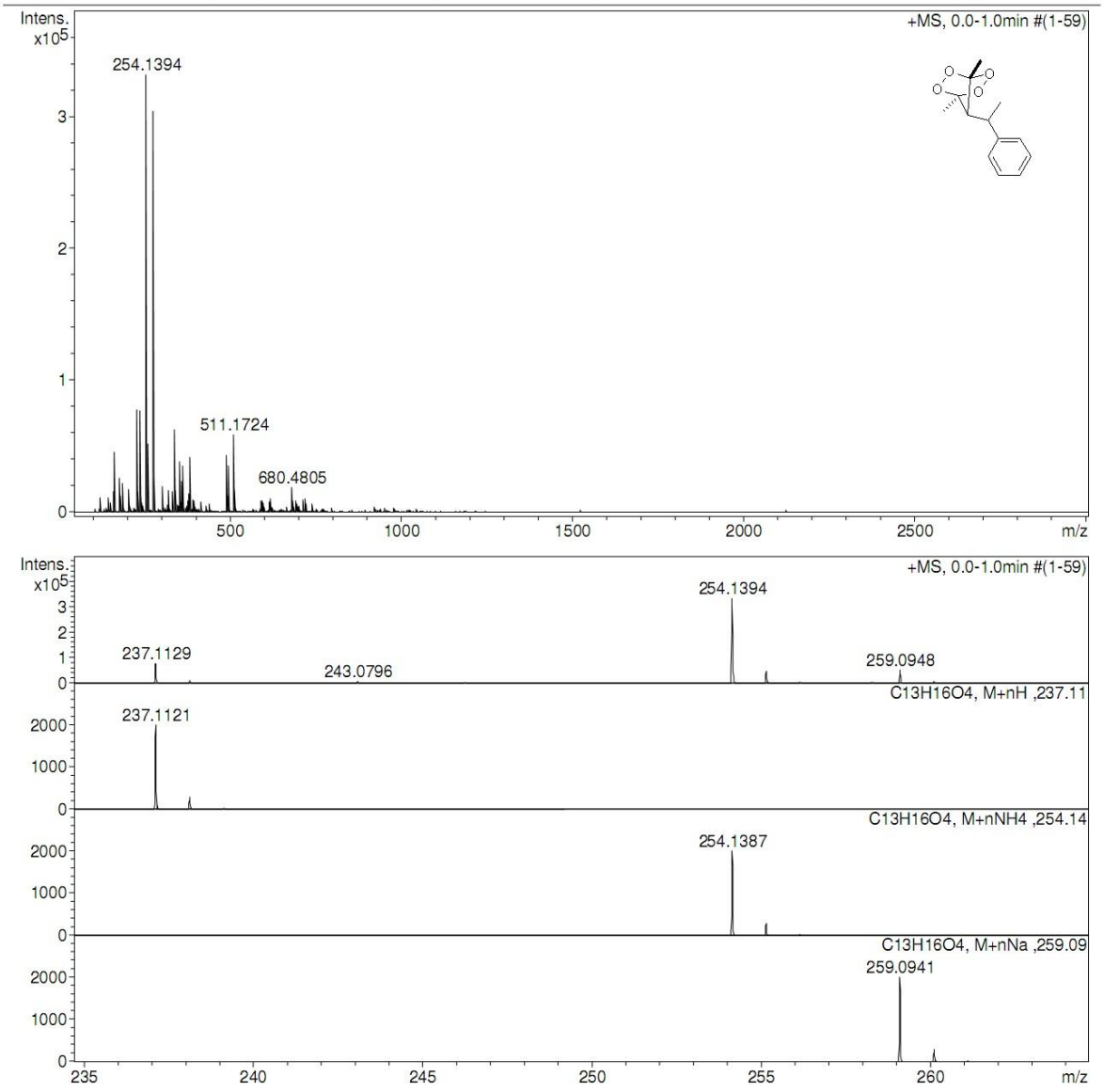
Display Report

Analysis Info

Analysis Name	D:\Data\Chizhov\Nikishin\Terentiev\Yaremenko\b416_&clblow.d	Acquisition Date	4/23/2012 4:54:42 PM
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN B-416	Instrument / Ser#	micrOTOF 10248
Comment	CH3CN 100 %, dil. 200, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



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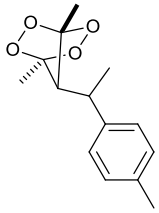
1,4-Dimethyl-7-[1-(4-methylphenyl)ethyl]-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2m)



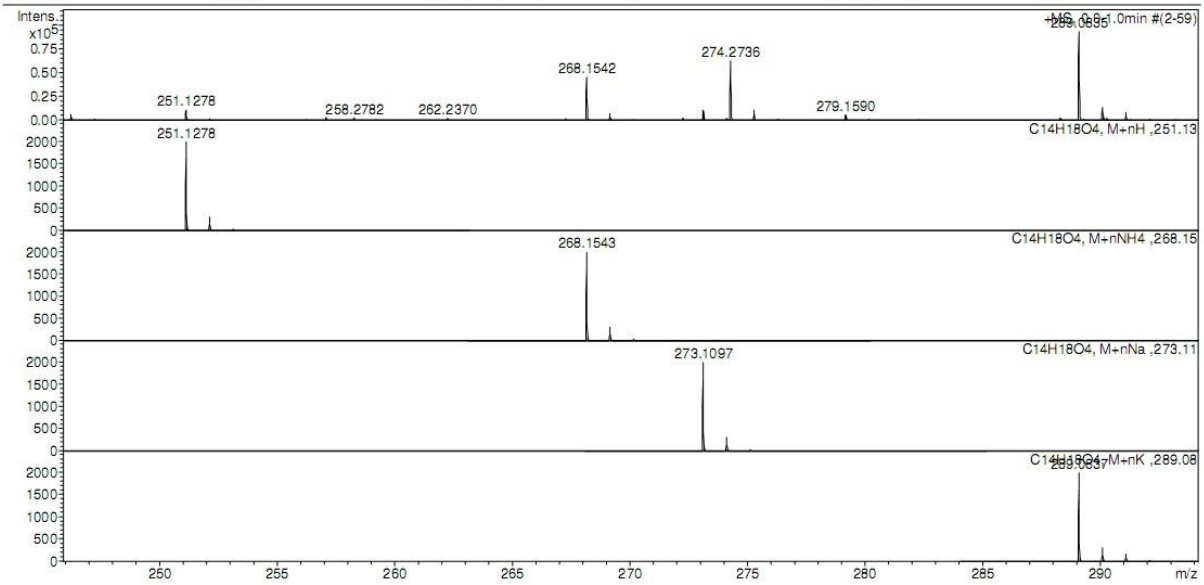
Display Report

Analysis Info

Analysis Name	D:\Data\Chizhov\Nikishin\Terentiev\Yaremenko\b421_&clblow.d	Acquisition Date	4/23/2012 5:40:14 PM
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN B-421	Instrument / Ser#	micrOTOF 10248
Comment	CH3CN 100 %, dil. 200, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



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7-[1-(4-Bromophenyl)ethyl]-1,4-dimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2n)

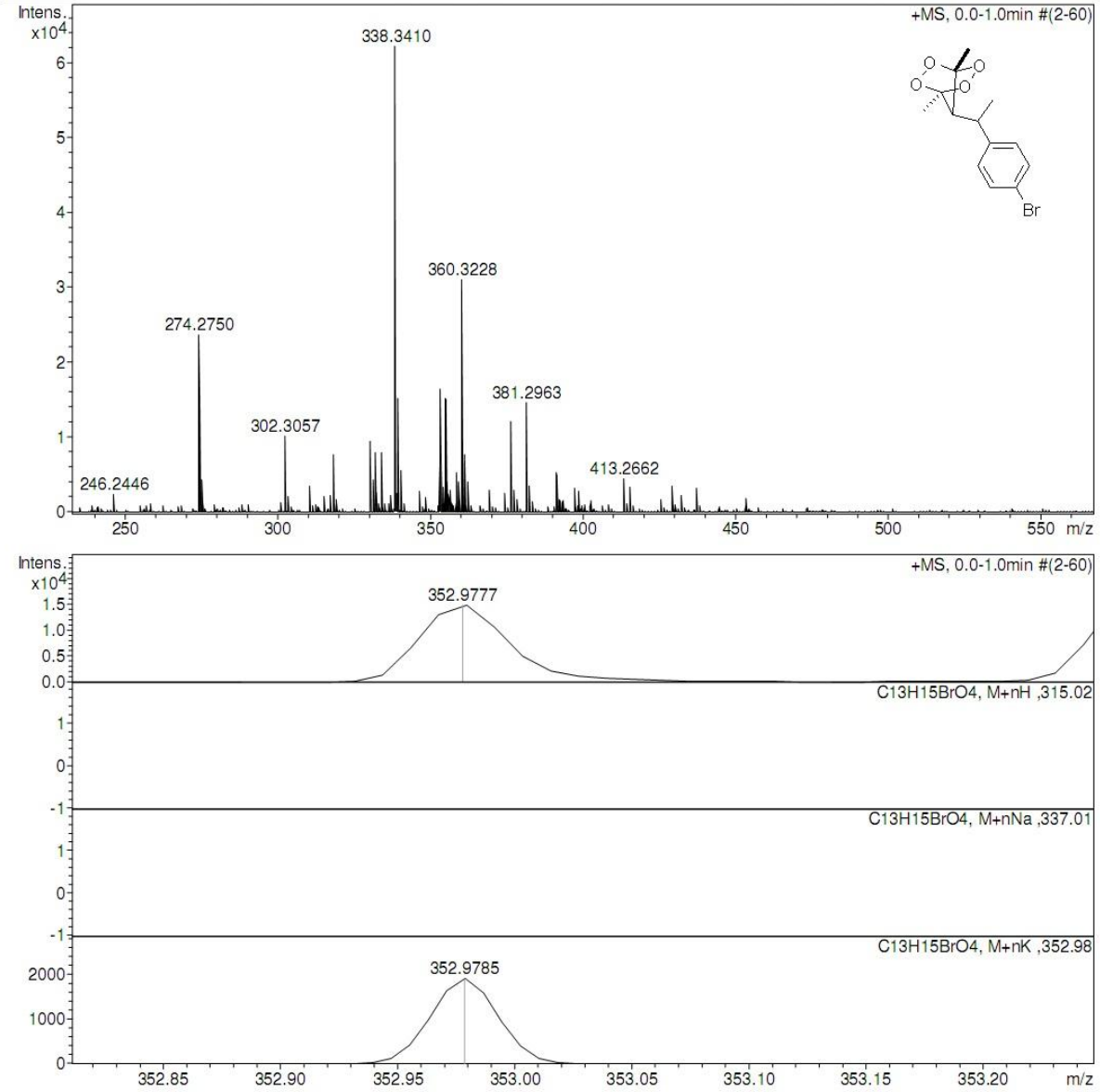
Display Report

Analysis Info

Analysis Name	D:\Data\Chizhov\Nikishin\Terentiev\Yaremenko\b423_low.d	Acquisition Date	4/23/2012 6:00:25 PM
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN B-423	Instrument / Ser#	micrOTOF 10248
Comment	CH3CN 100 %, dil. 200, no calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



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

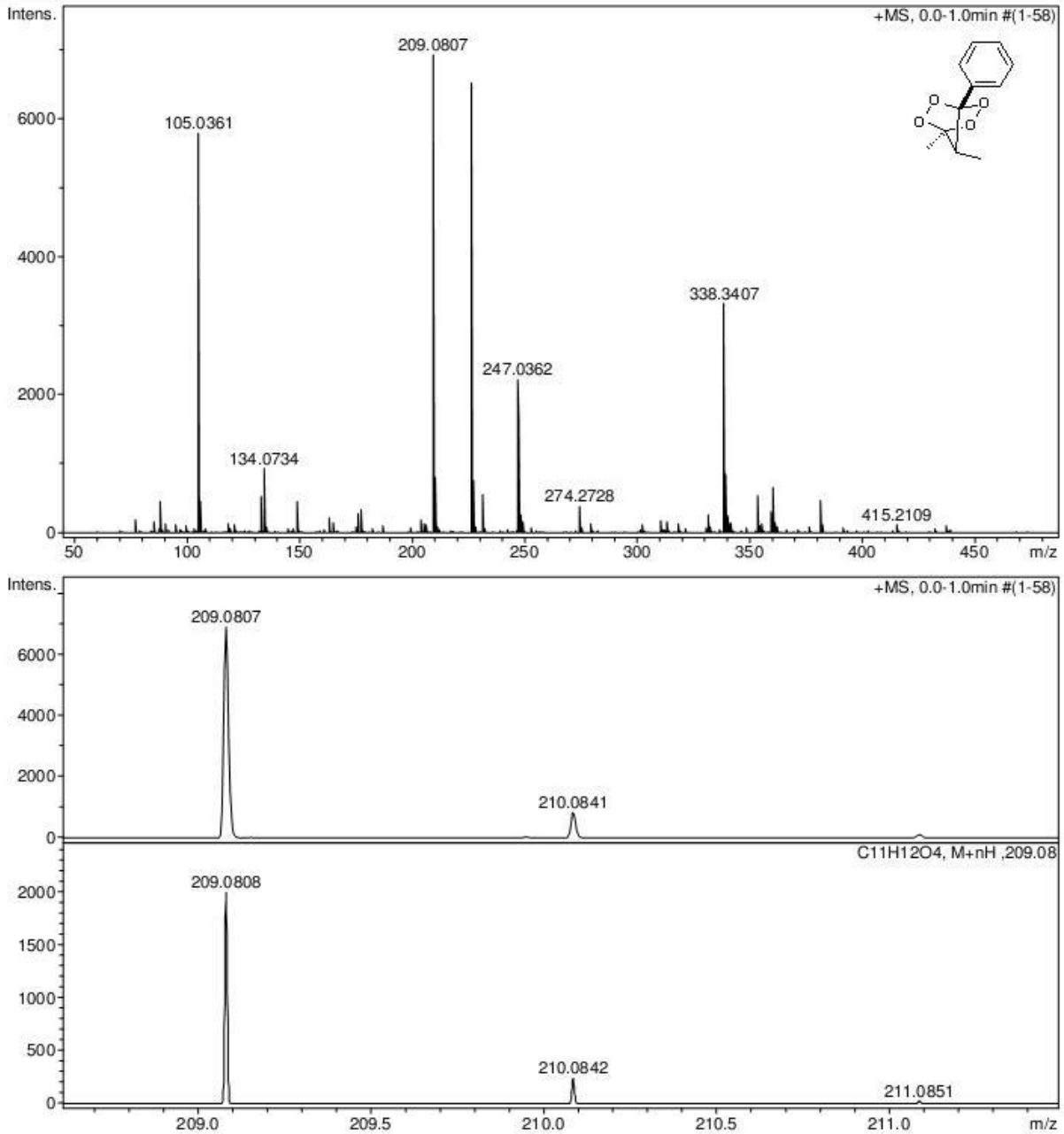
Display Report

Analysis Info

Analysis Name	D:\Data\khemchyan\daily\249.d	Acquisition Date	7/25/2012 6:37:39 PM
Method	tune_low.m	Operator	BDAL@DE
Sample Name	249	Instrument	maXis 43
Comment	Solvent: MeCN Yaremenko I		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Source



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7-Benzyl-1-methyl-4-phenyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2p)

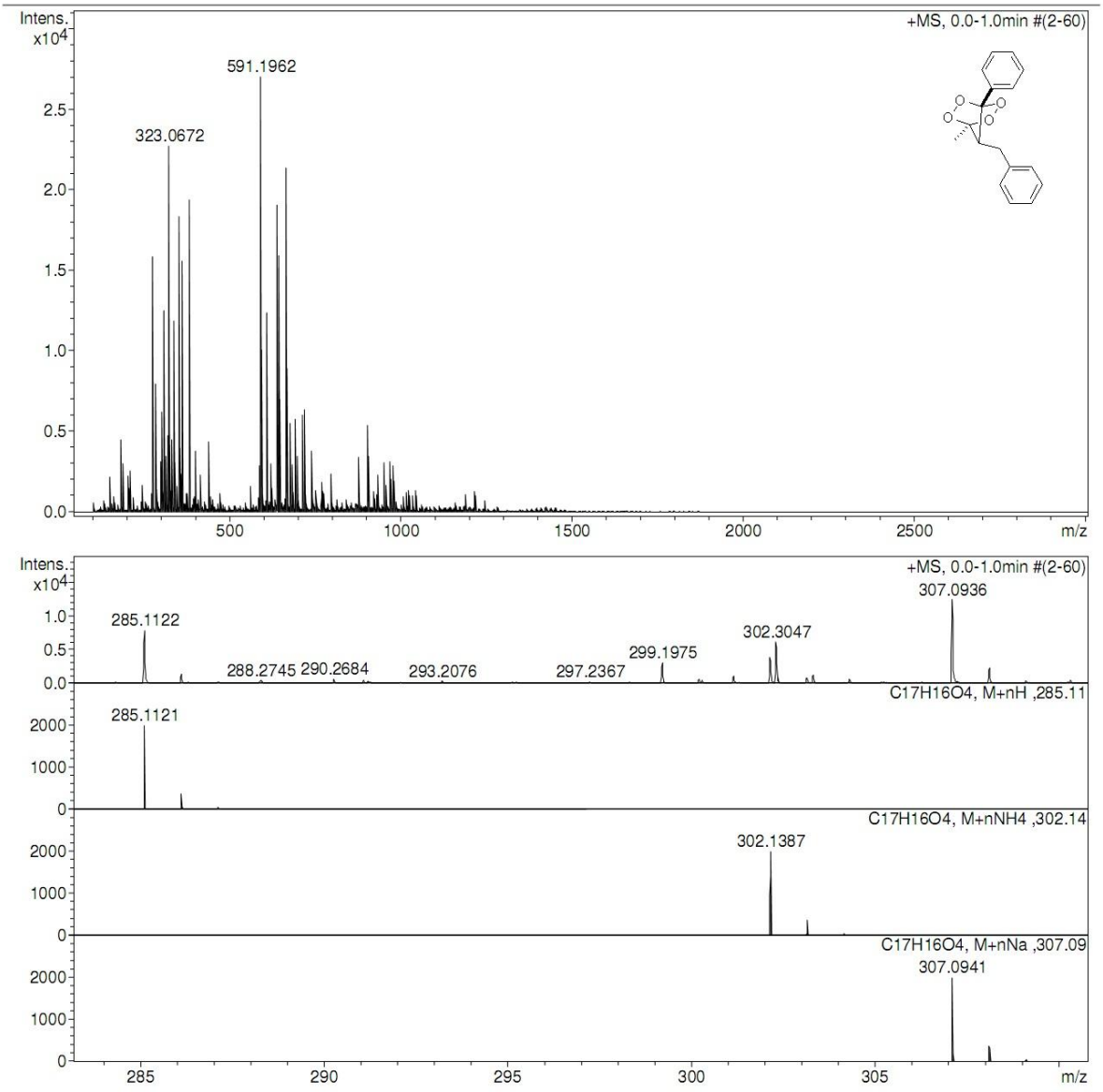
Display Report

Analysis Info

Analysis Name	D:\Data\Chizhov\Nikishin\Terentiev\Yaremenko\bb-215_low.d	Acquisition Date	6/4/2012 4:06:52 PM
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN BB-215	Instrument / Ser#	micrOTOF 10248
Comment	CH3CN 100 %, dil. 200, no calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



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1,4-Diphenyl-7-methyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2q)

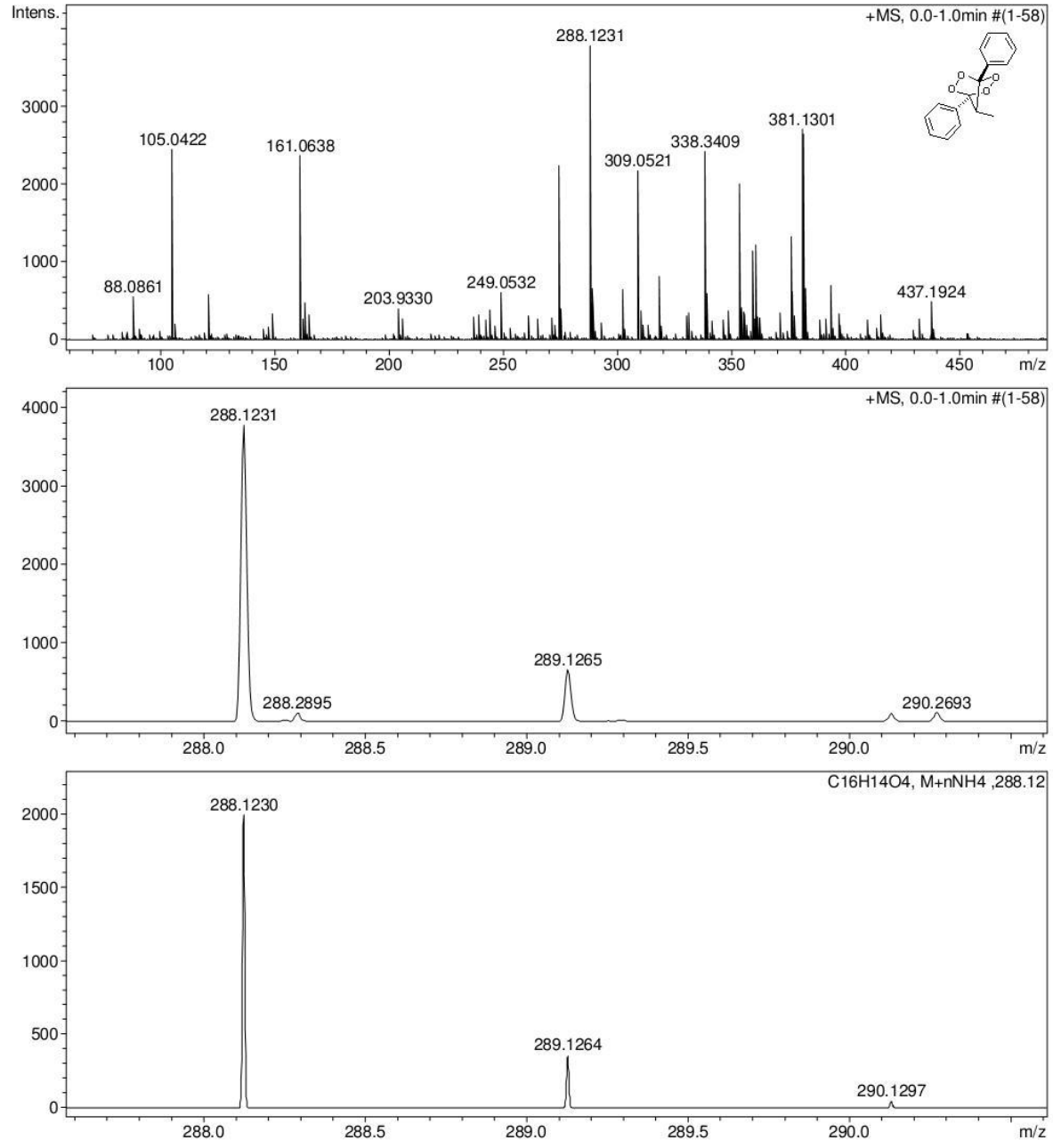
Display Report

Analysis Info

Analysis Name	D:\Data\khemchyan\MS service @ IOC\Yaremenko I\31_07_2012\252.d	Acquisition Date	7/31/2012 11:40:22 AM
Method	tune_low.m	Operator	BDAL@DE
Sample Name	252	Instrument	maXis 43
Comment	Yaremenko MeCN		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Source



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7-Benzyl-1,4-diphenyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2r)

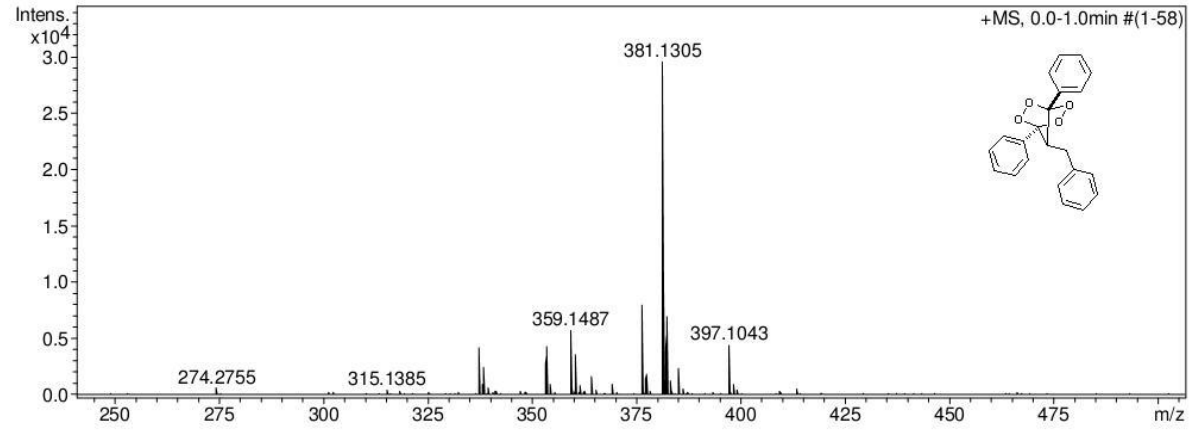
Display Report

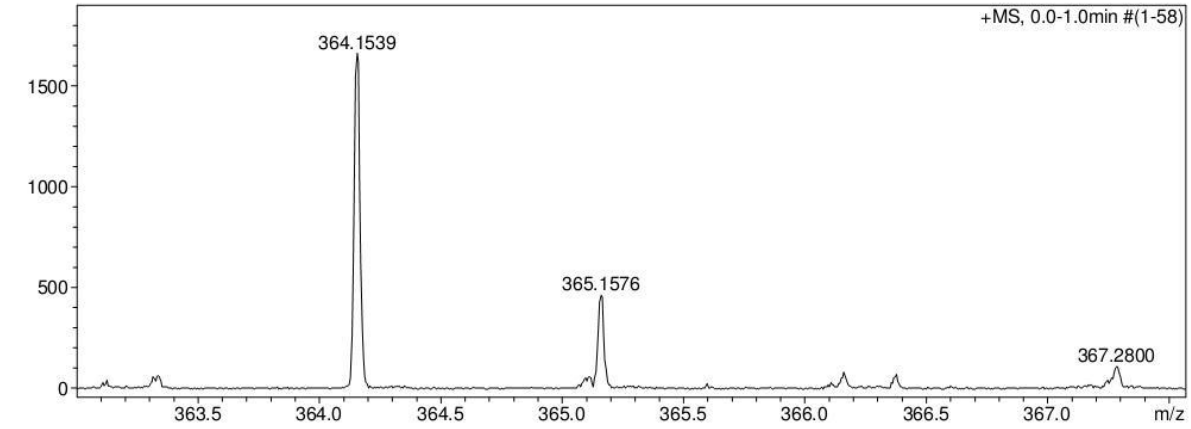
Analysis Info

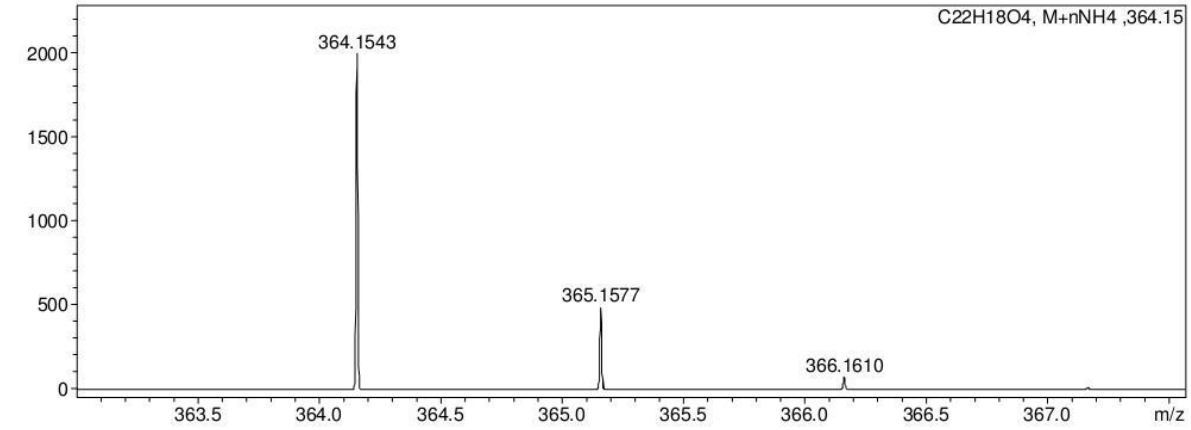
Analysis Name	D:\Data\khemchyan\MS service @ IOC\Yaremenko I\31_07_2012\254.d	Acquisition Date	7/31/2012 11:54:45 AM
Method	tune_wide.m	Operator	BDAL@DE
Sample Name	254	Instrument	maXis 43
Comment	Yaremenko MeCN		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	1200.0 Vpp	Set Divert Valve	Waste







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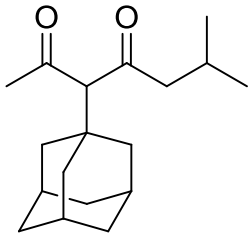
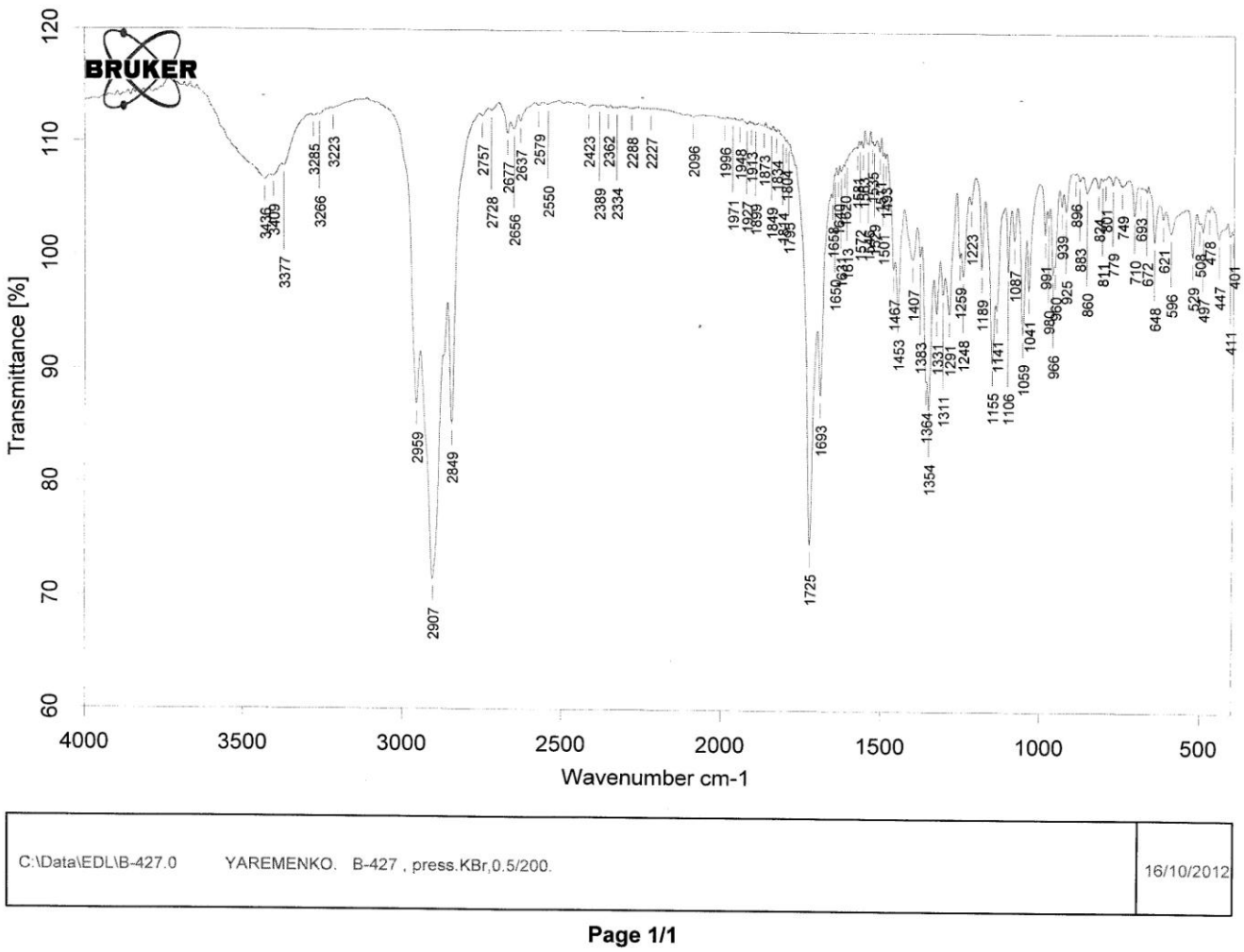
IR spectrum of diketone

3-(1-Adamantyl)-6-methylheptane-2,4-dione (1j)





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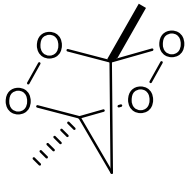
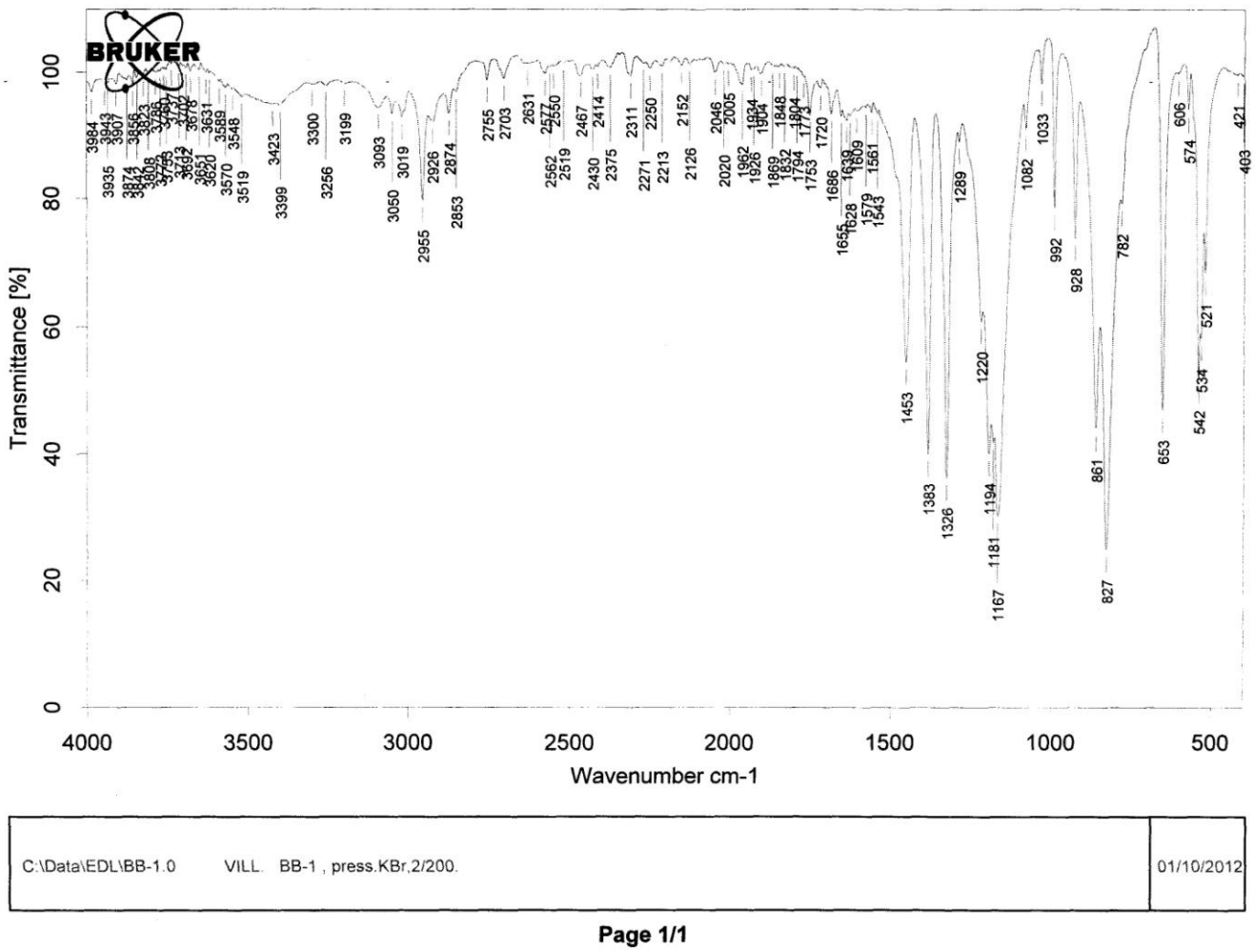
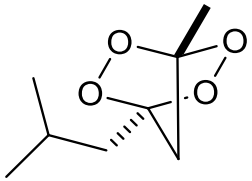
IR spectra of tetraoxanes

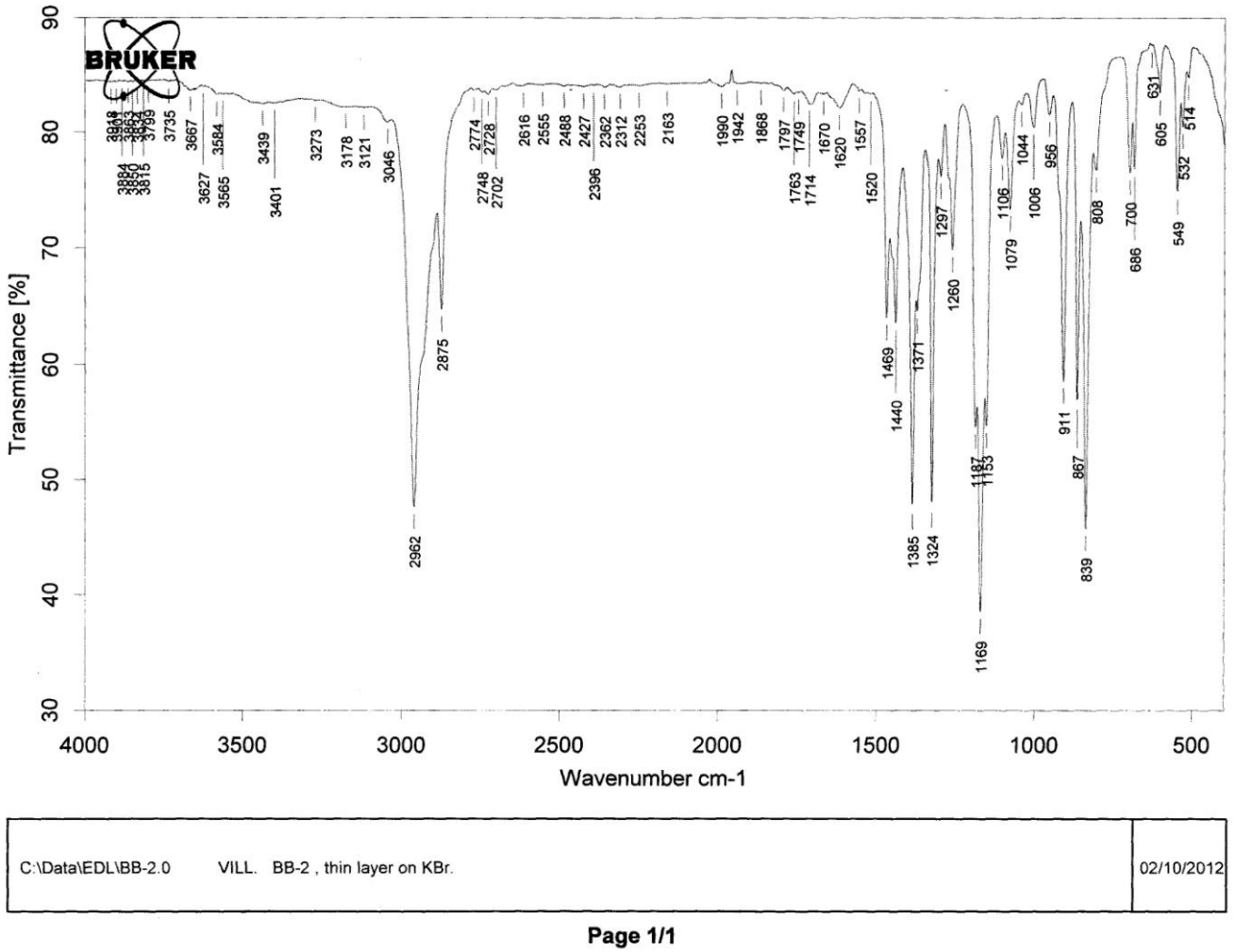
1,4-Dimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2a)





1-Isobutyl-4-methyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2b)





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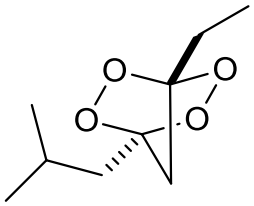
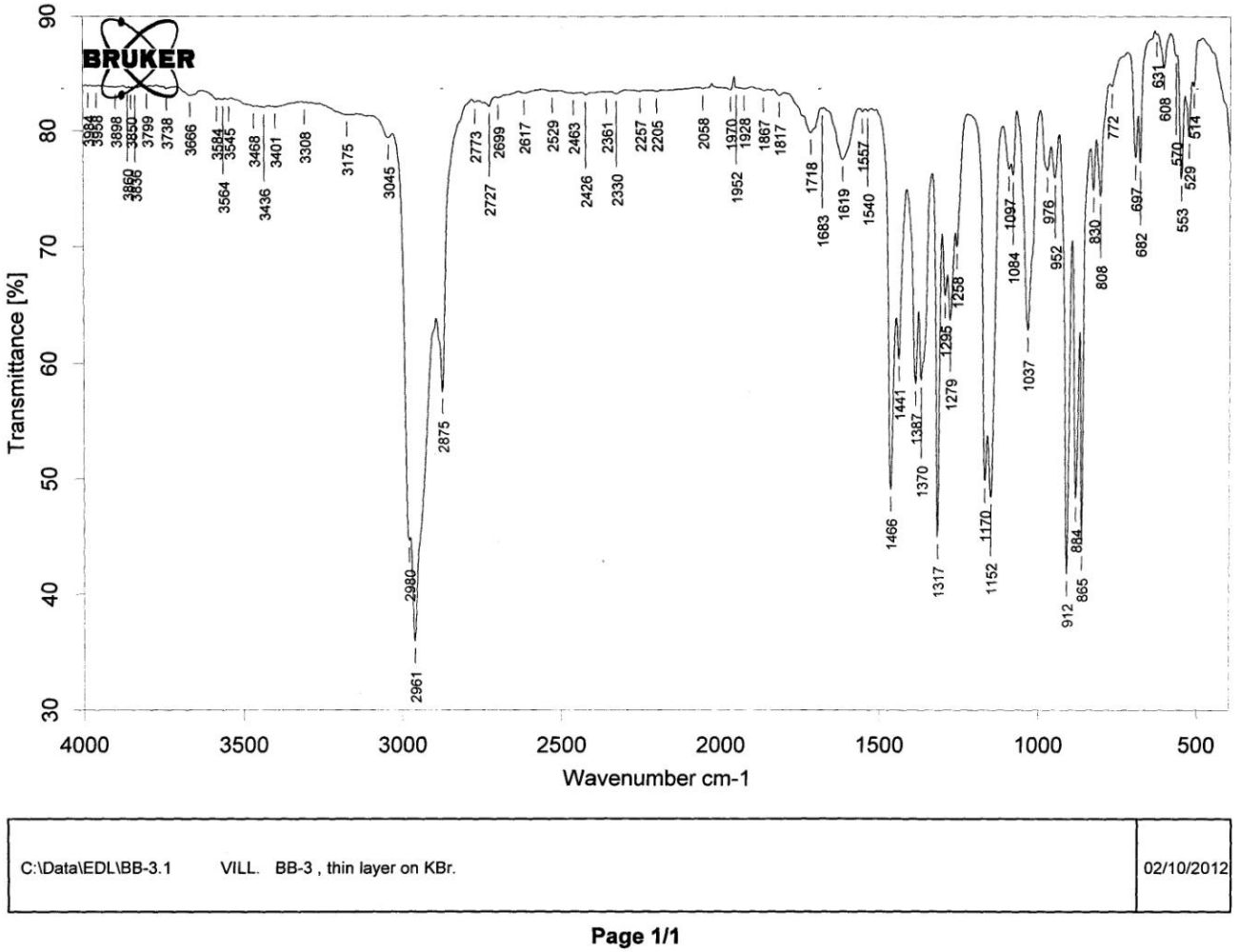
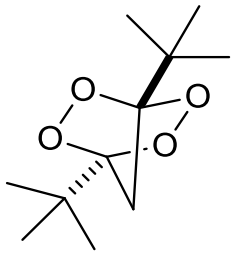
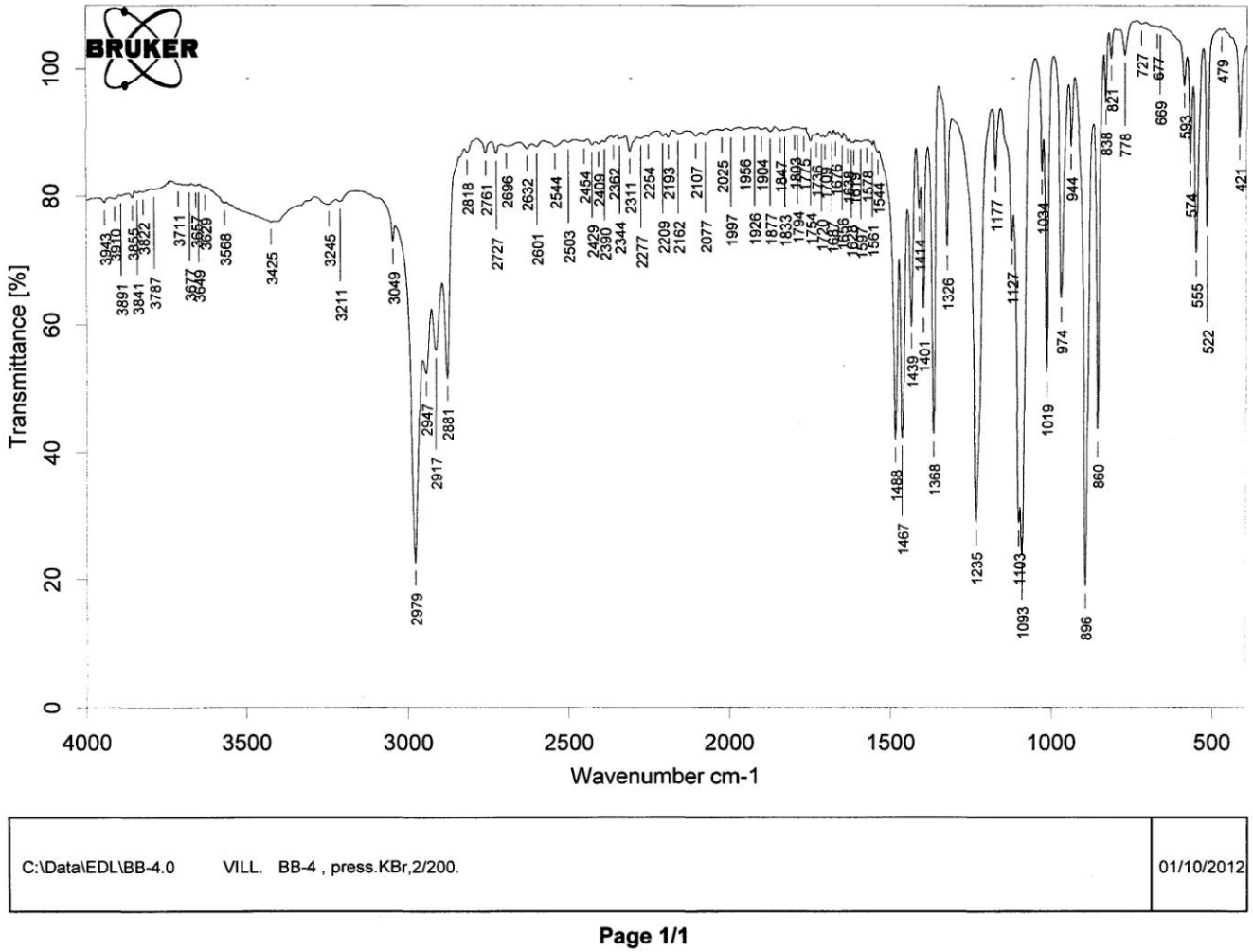
1-Ethyl-4-isobutyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2c)



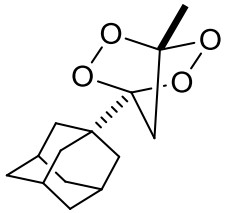


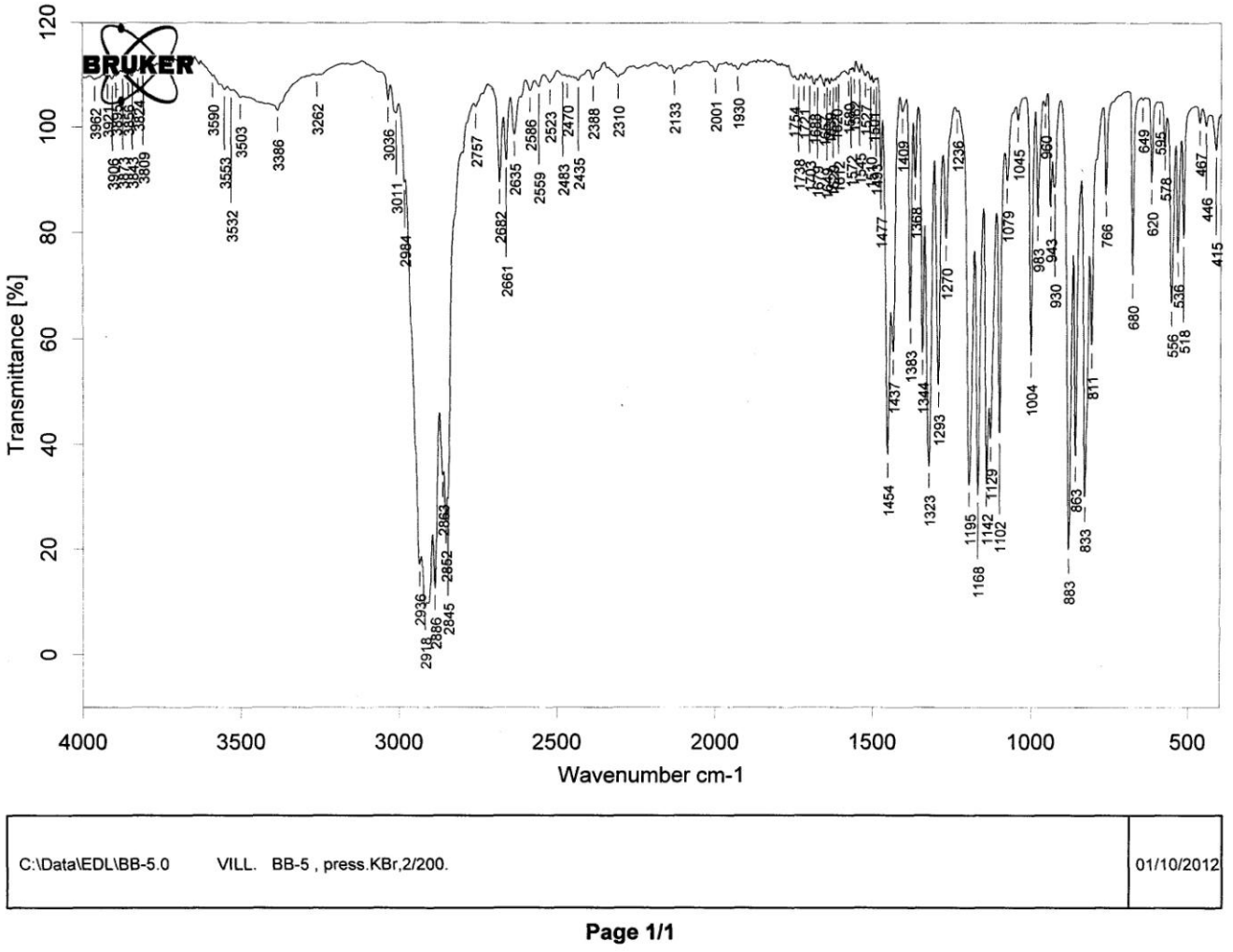
1,4-Di-*tert*-butyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2d)



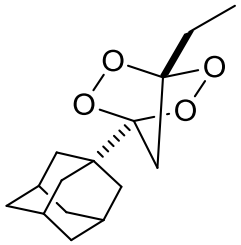


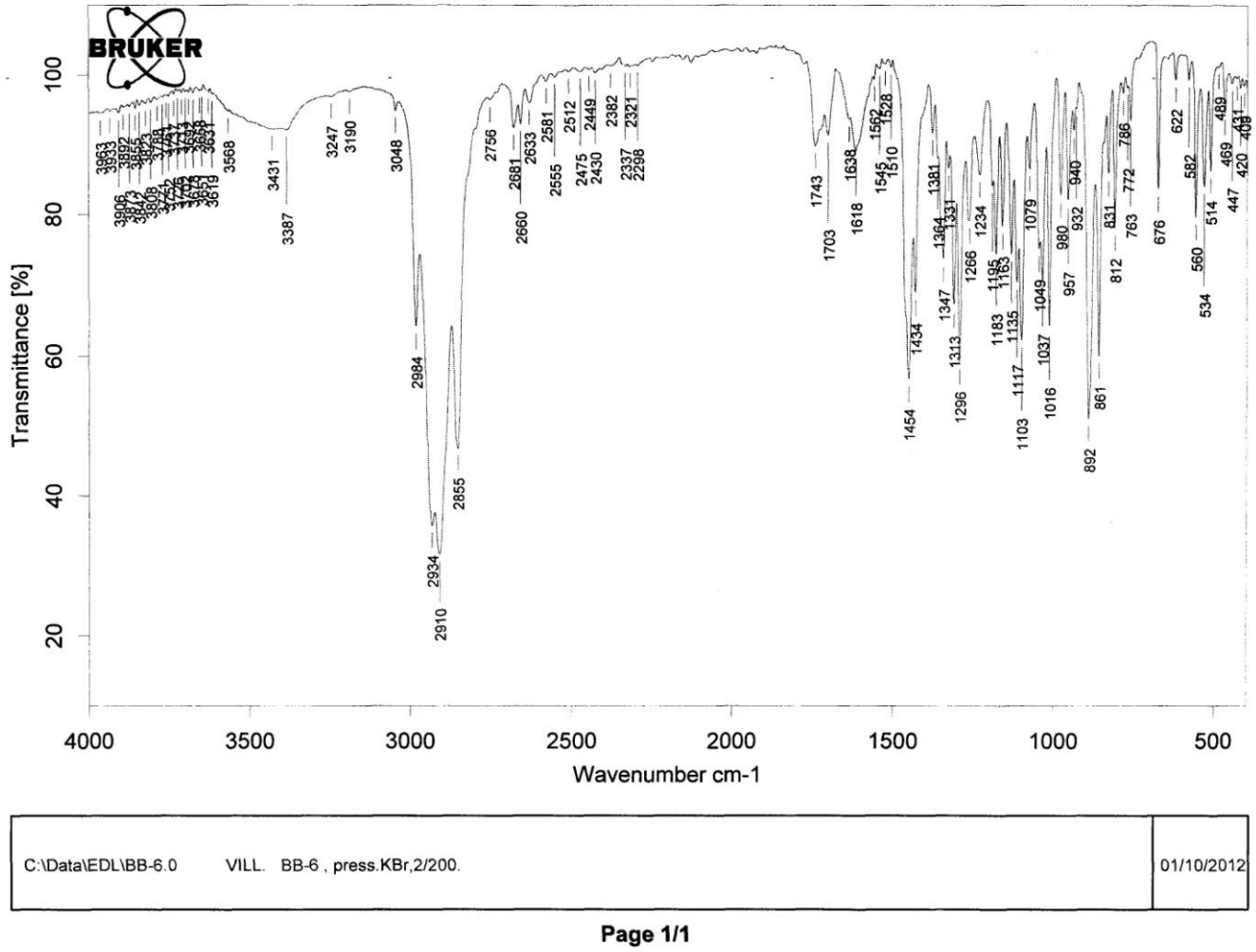
1-(1-Adamantyl)-4-methyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2e)



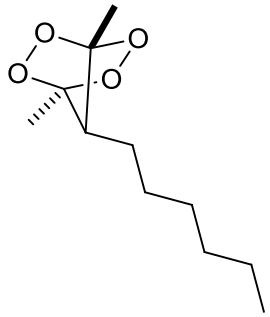


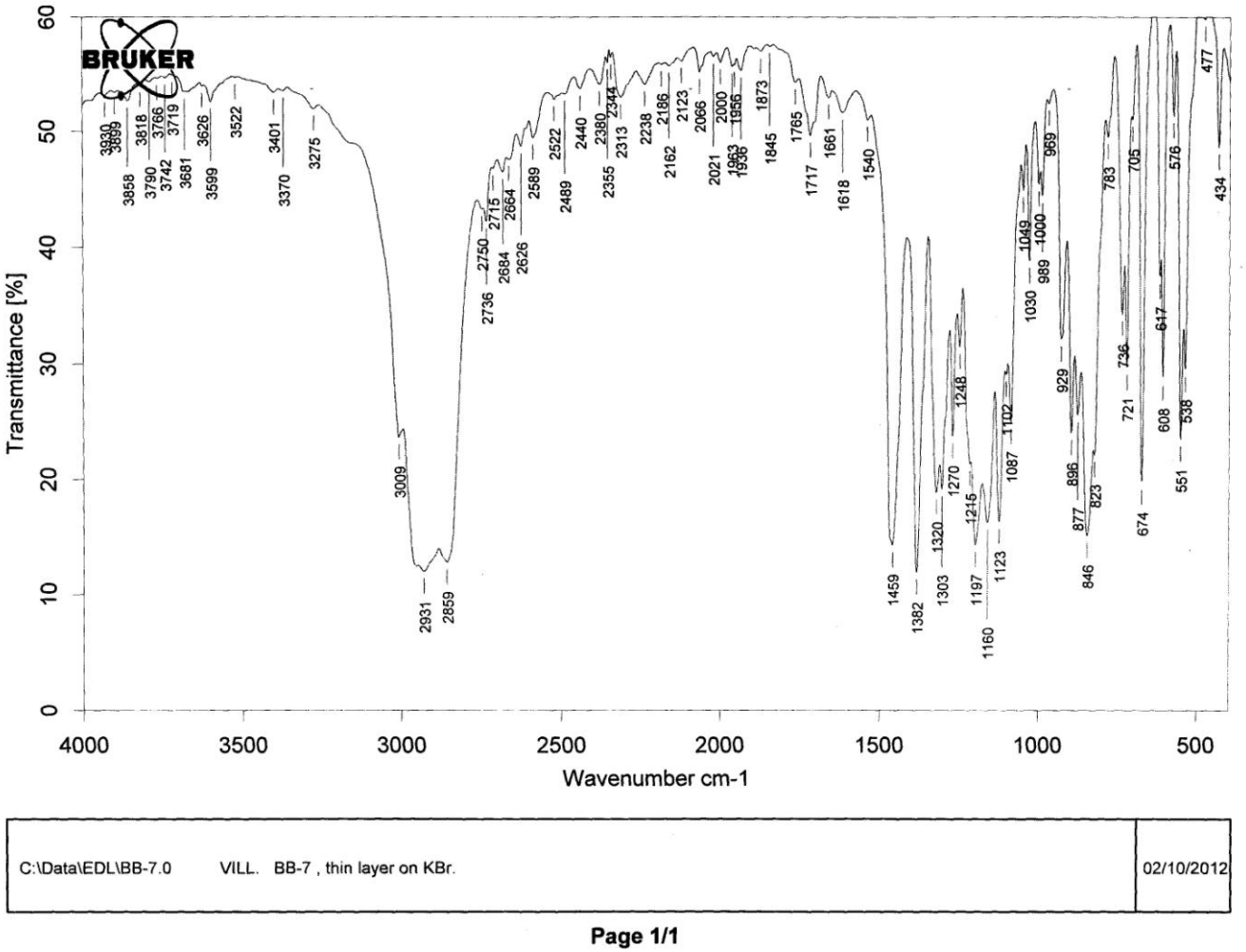
1-(1-Adamantyl)-4-ethyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2f)





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





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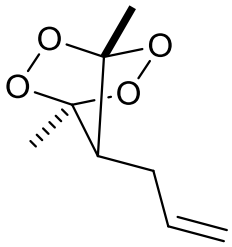
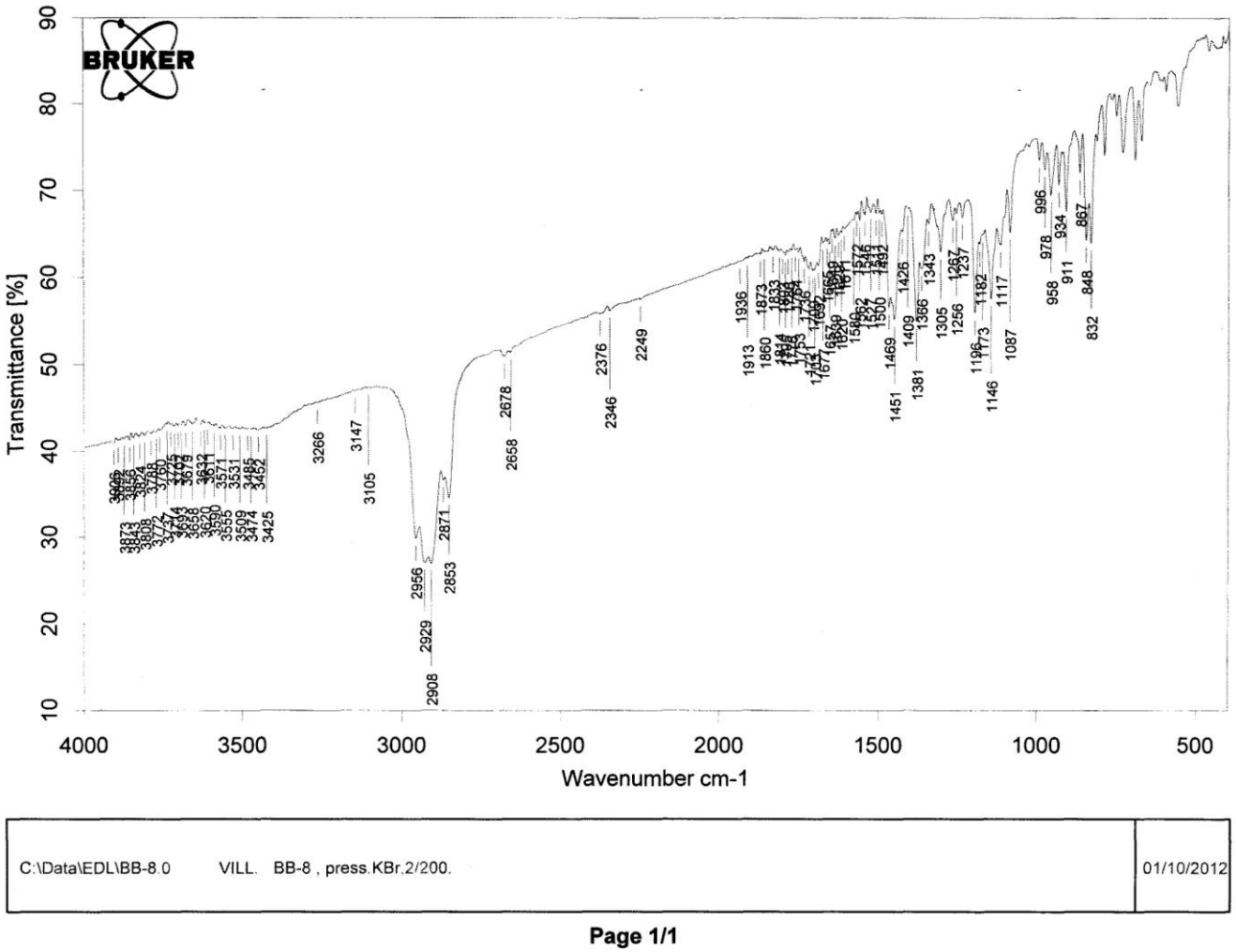
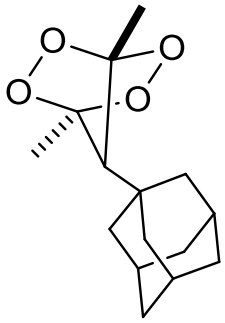
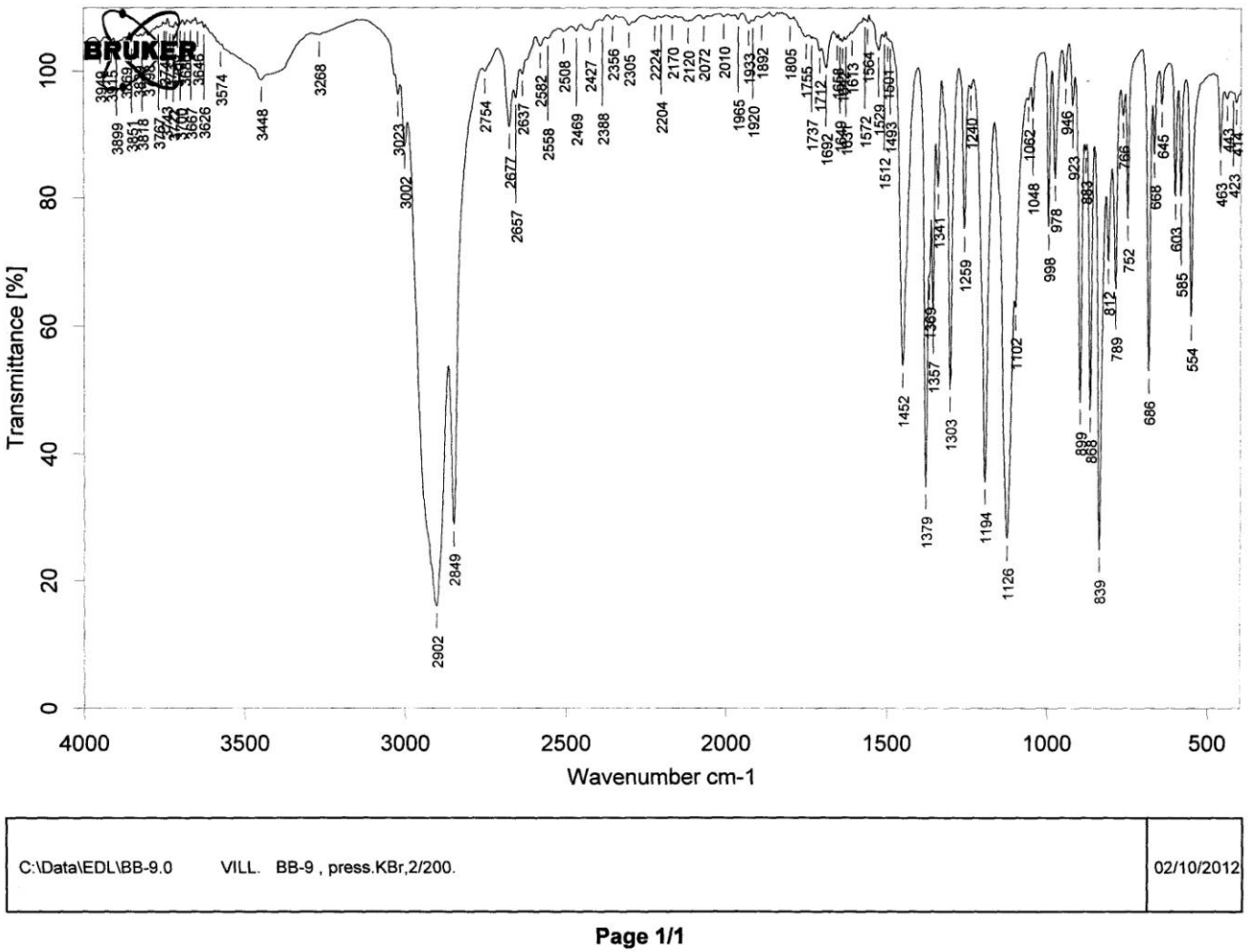
7-Allyl-1,4-dimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2h)



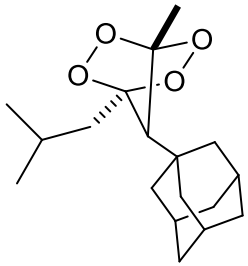


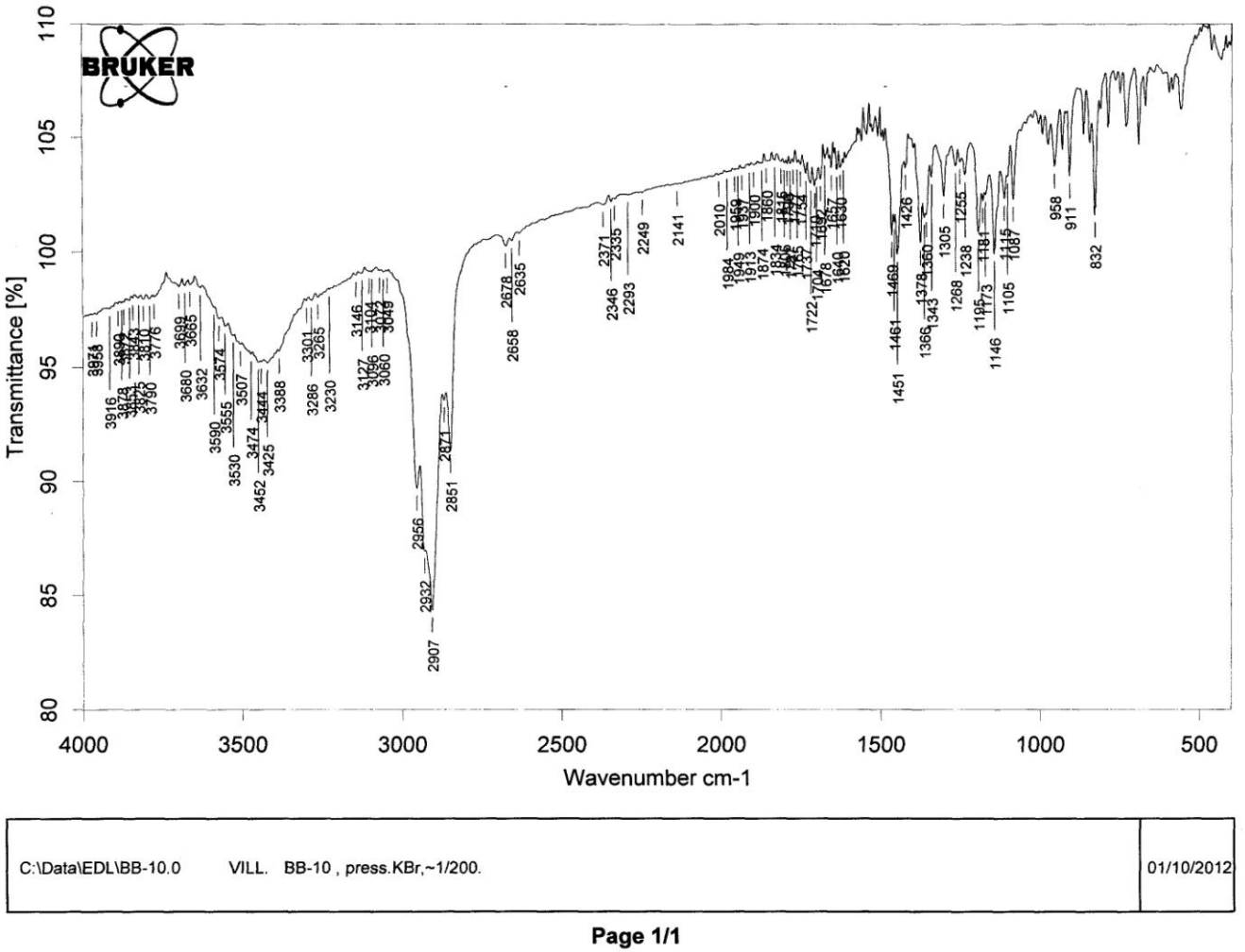
7-(1-Adamantyl)-1,4-dimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2i)



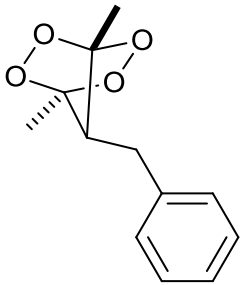


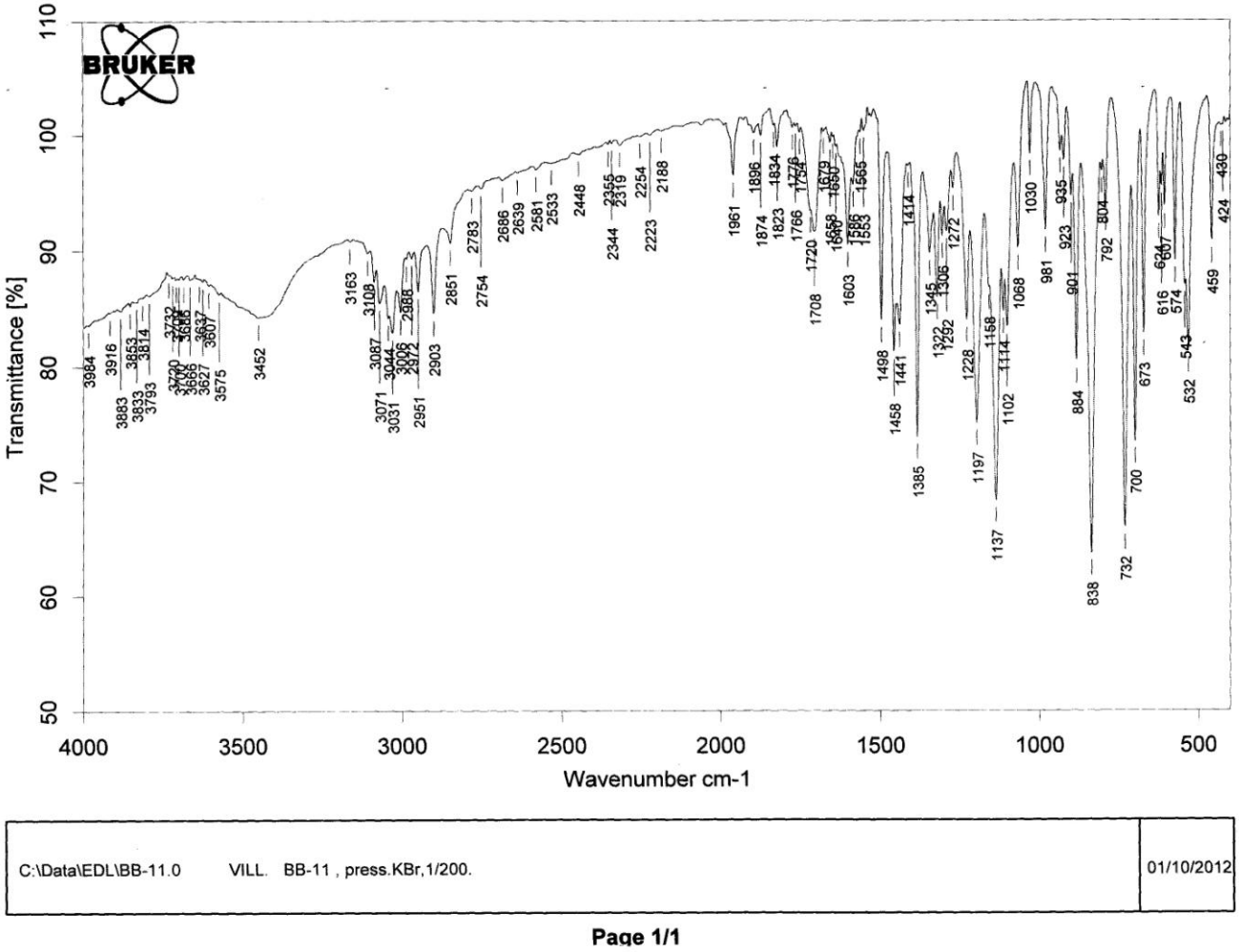
7-(1-Adamantyl)-1-isobutyl-4-methyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2j)



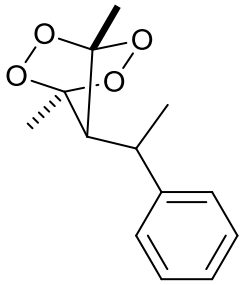


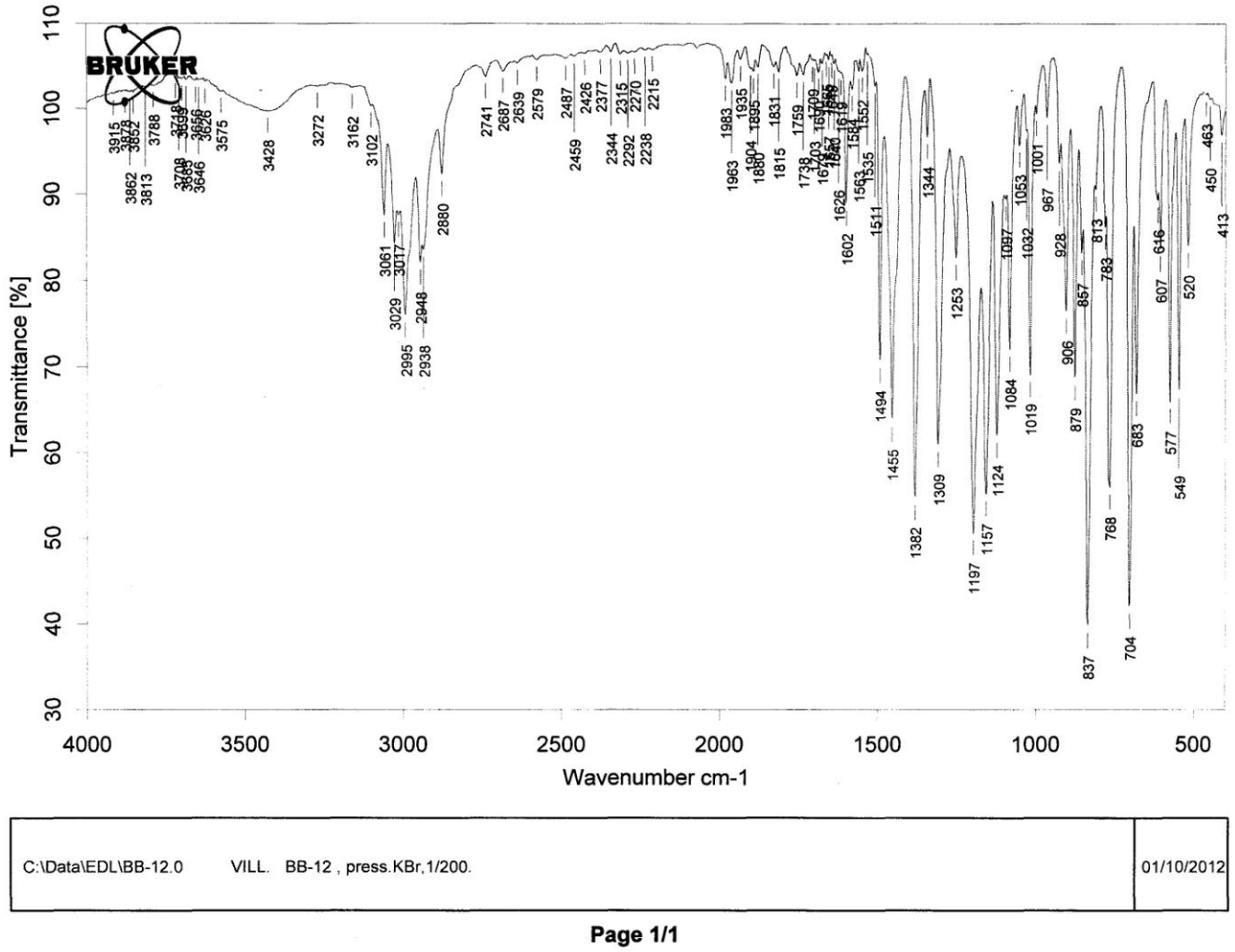
7-Benzyl-1,4-dimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2k)



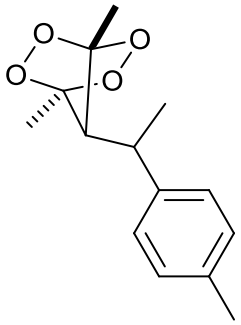


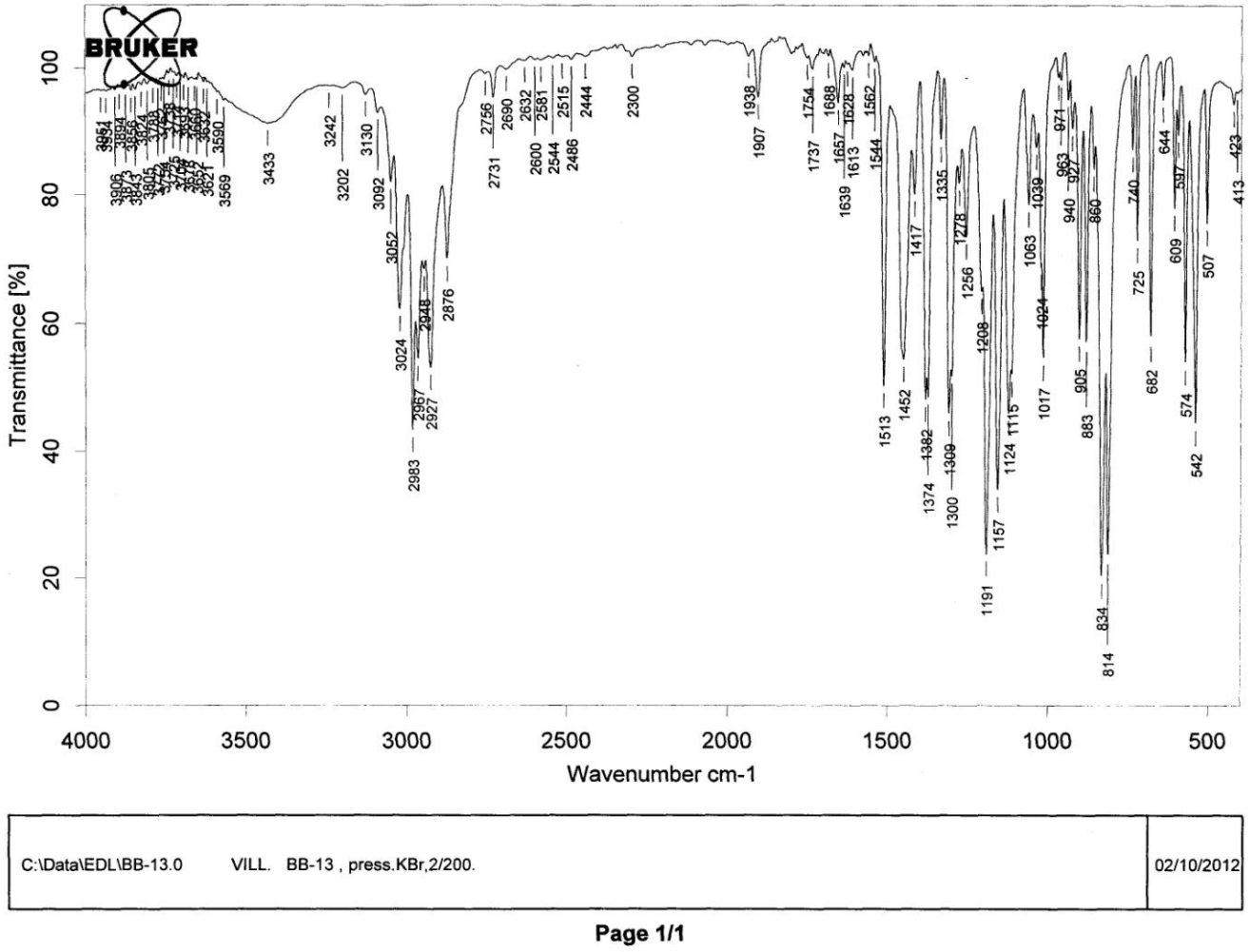
1,4-Dimethyl-7-(1-phenylethyl)-2,3,5,6-tetraoxabicyclo[2.2.1]heptanes (2l)



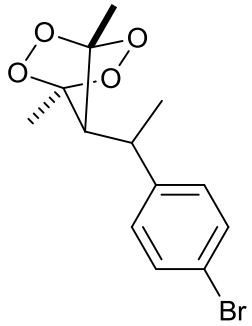


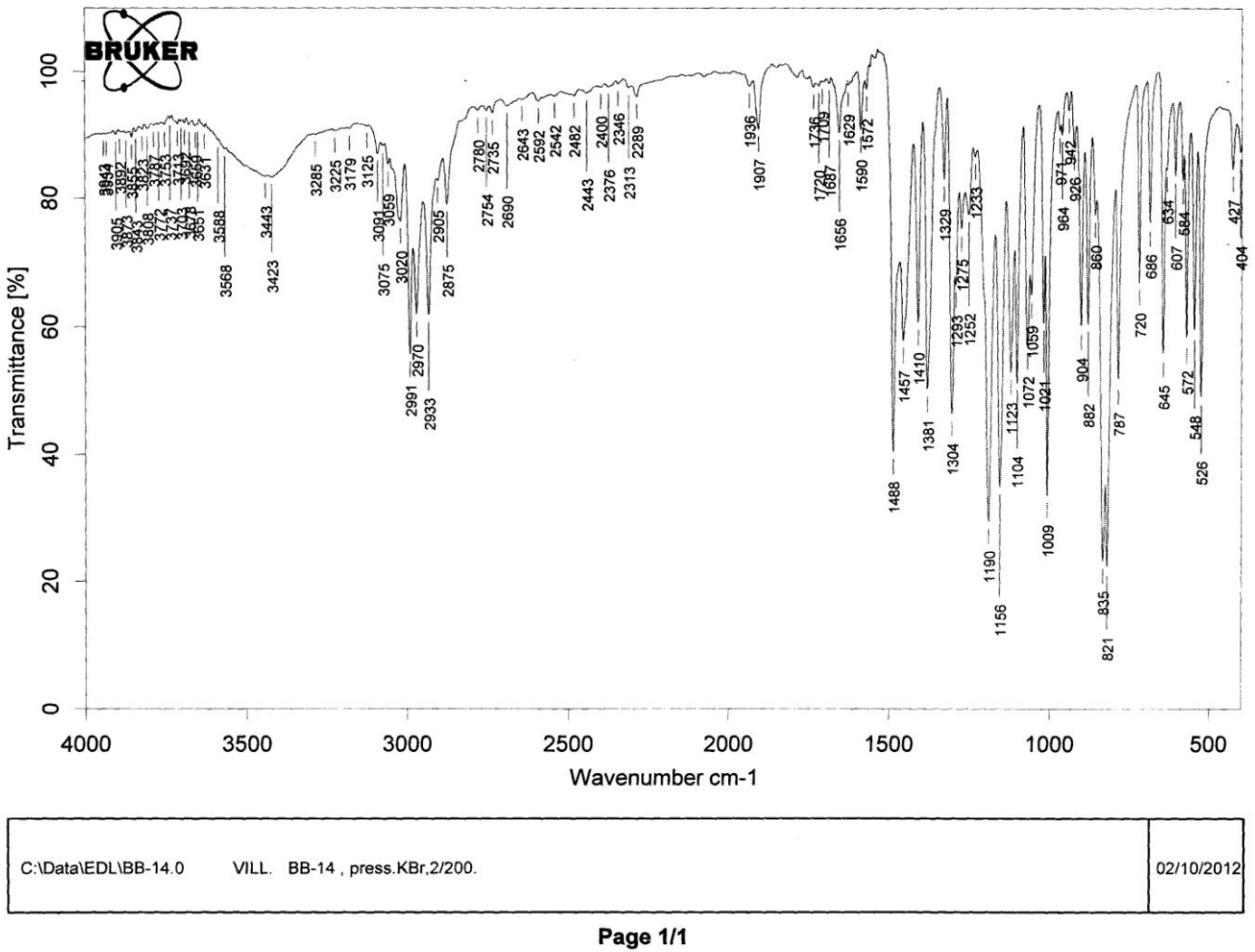
1,4-Dimethyl-7-[1-(4-methylphenyl)ethyl]-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2m)





7-[1-(4-Bromophenyl)ethyl]-1,4-dimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2n)





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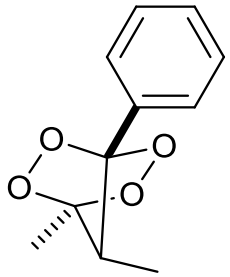
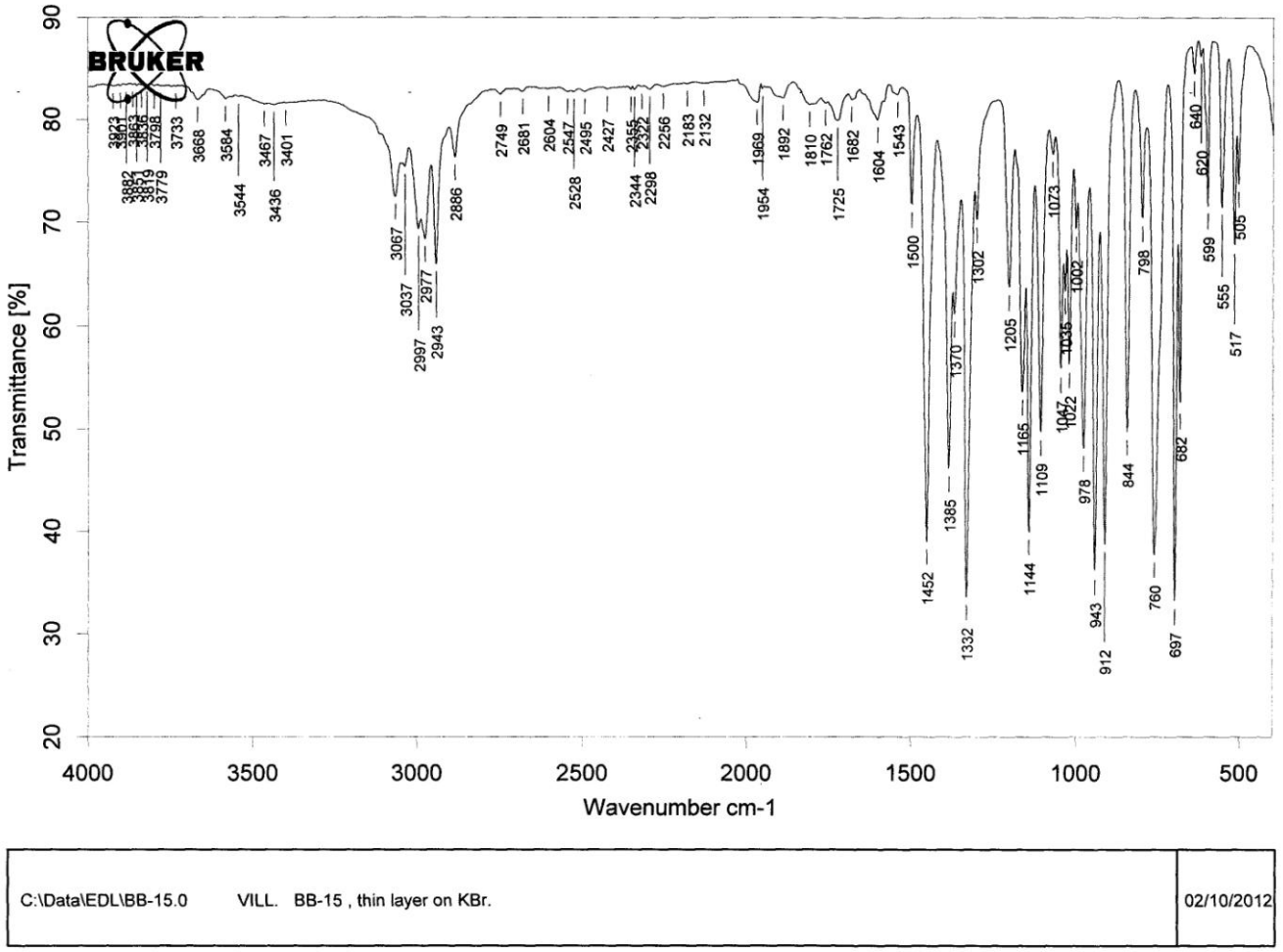
1,7-Dimethyl-4-phenyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2o)





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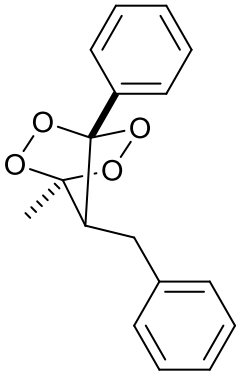
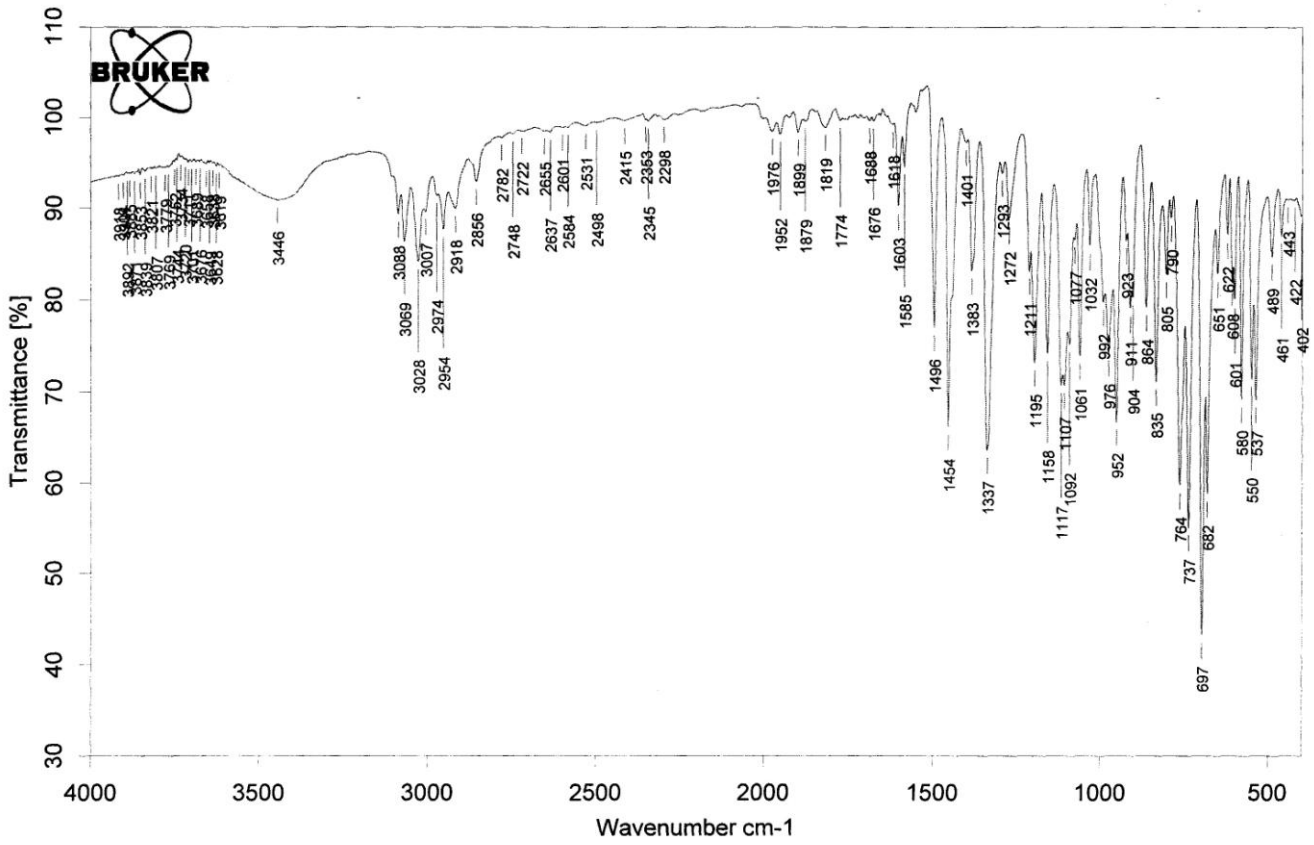
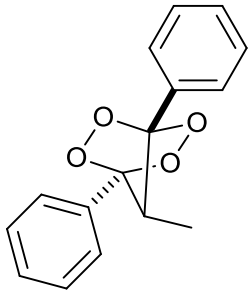
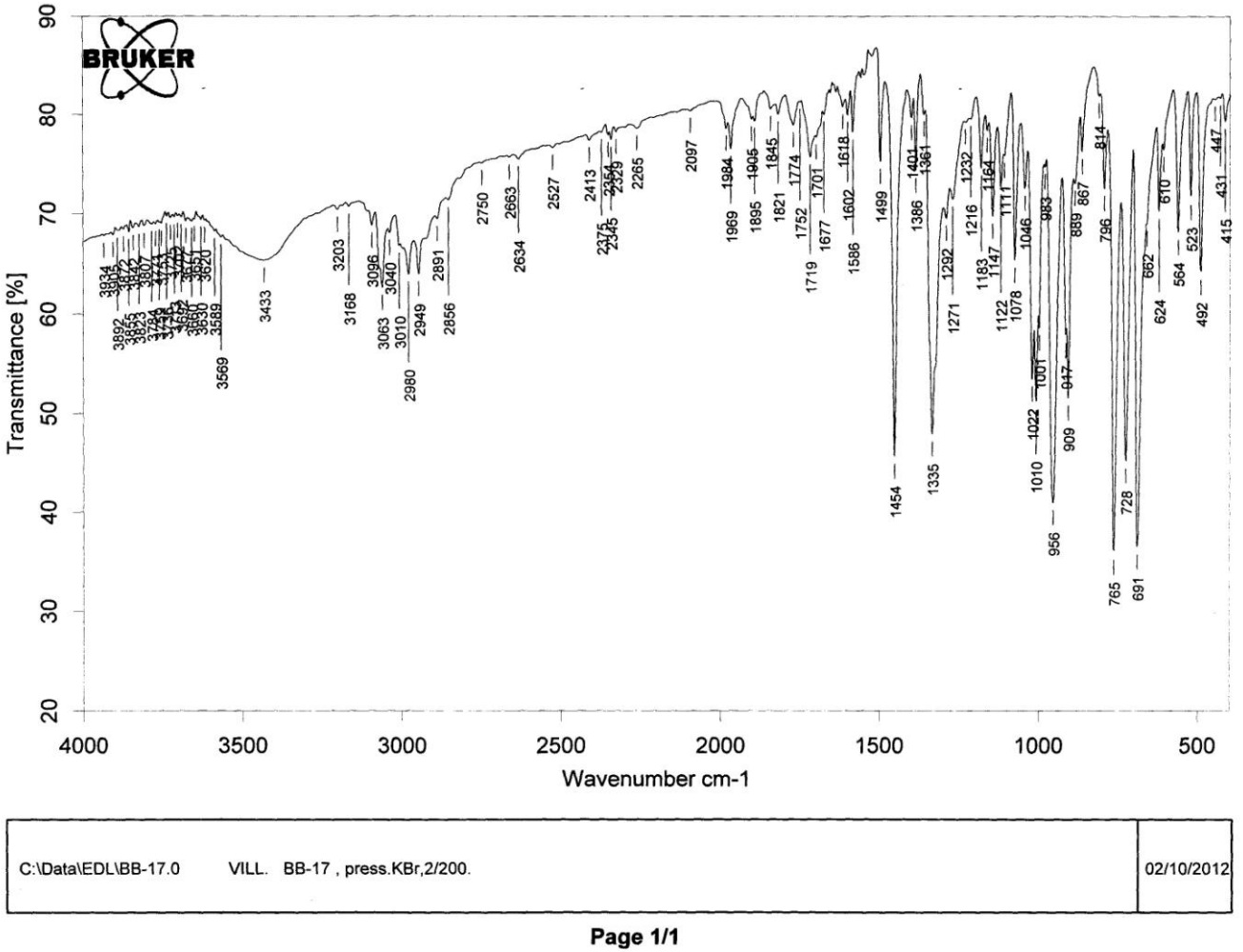
7-Benzyl-1-methyl-4-phenyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2p)



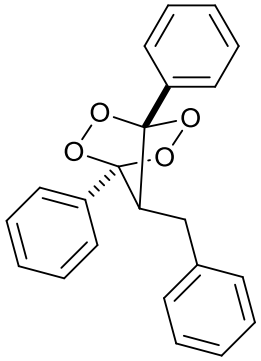


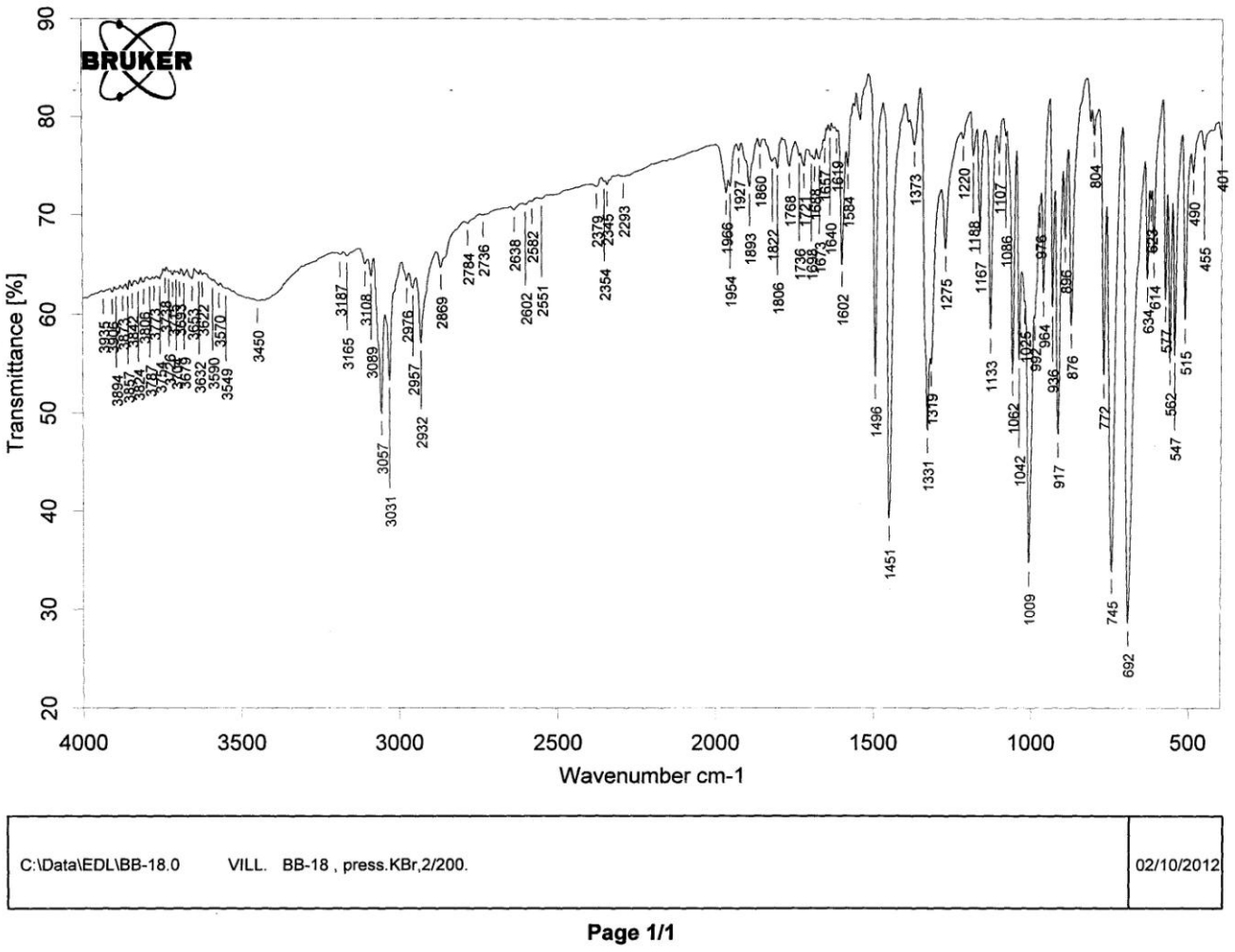
1,4-Diphenyl-7-methyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2q)



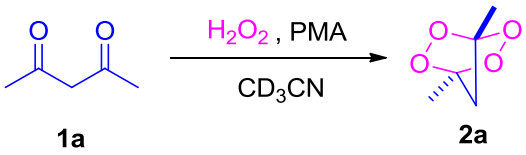


7-Benzyl-1,4-diphenyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane (2r)





Monitoring of the PMA-catalyzed reaction of pentane-2,4-dione 1a with H_2O_2

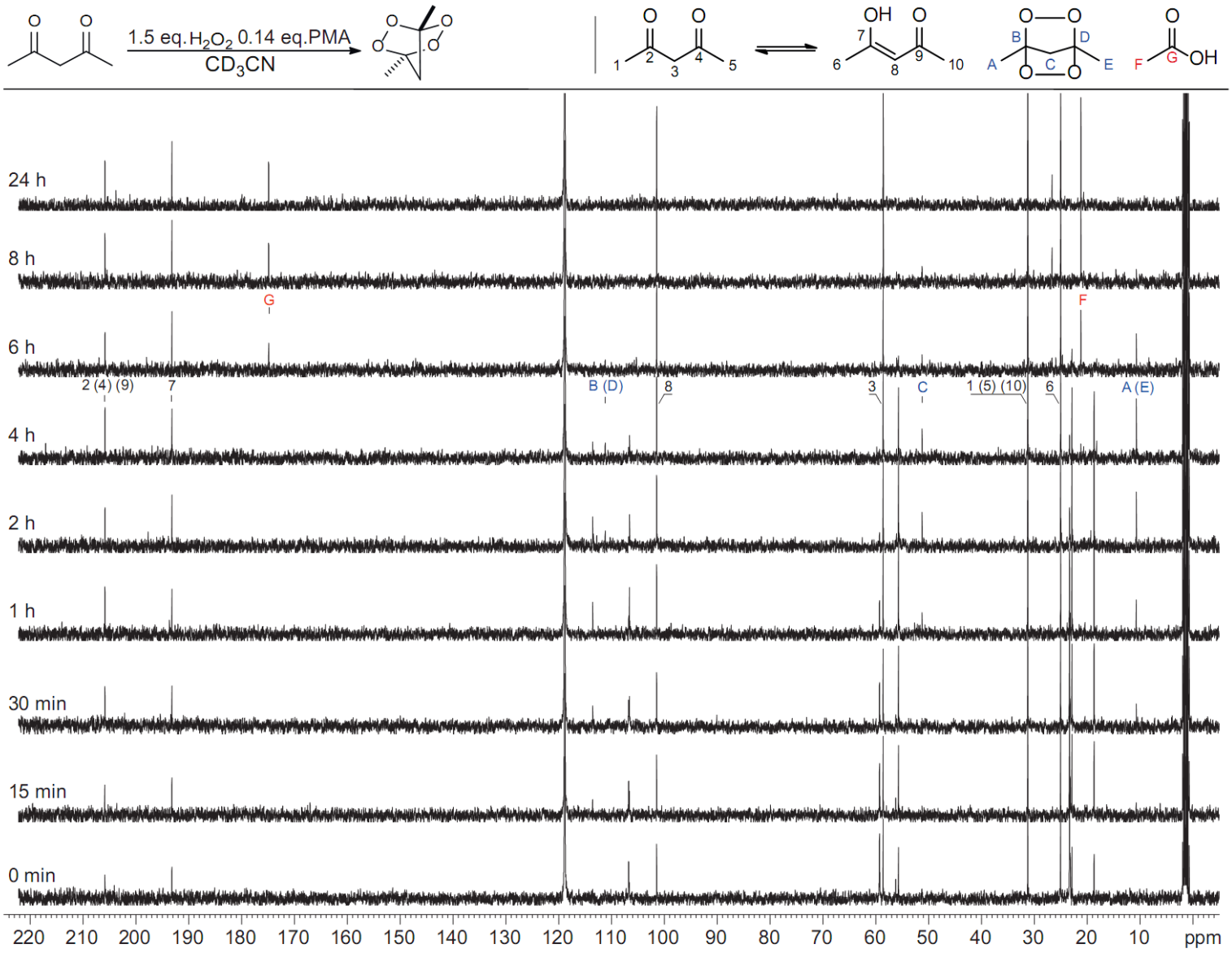


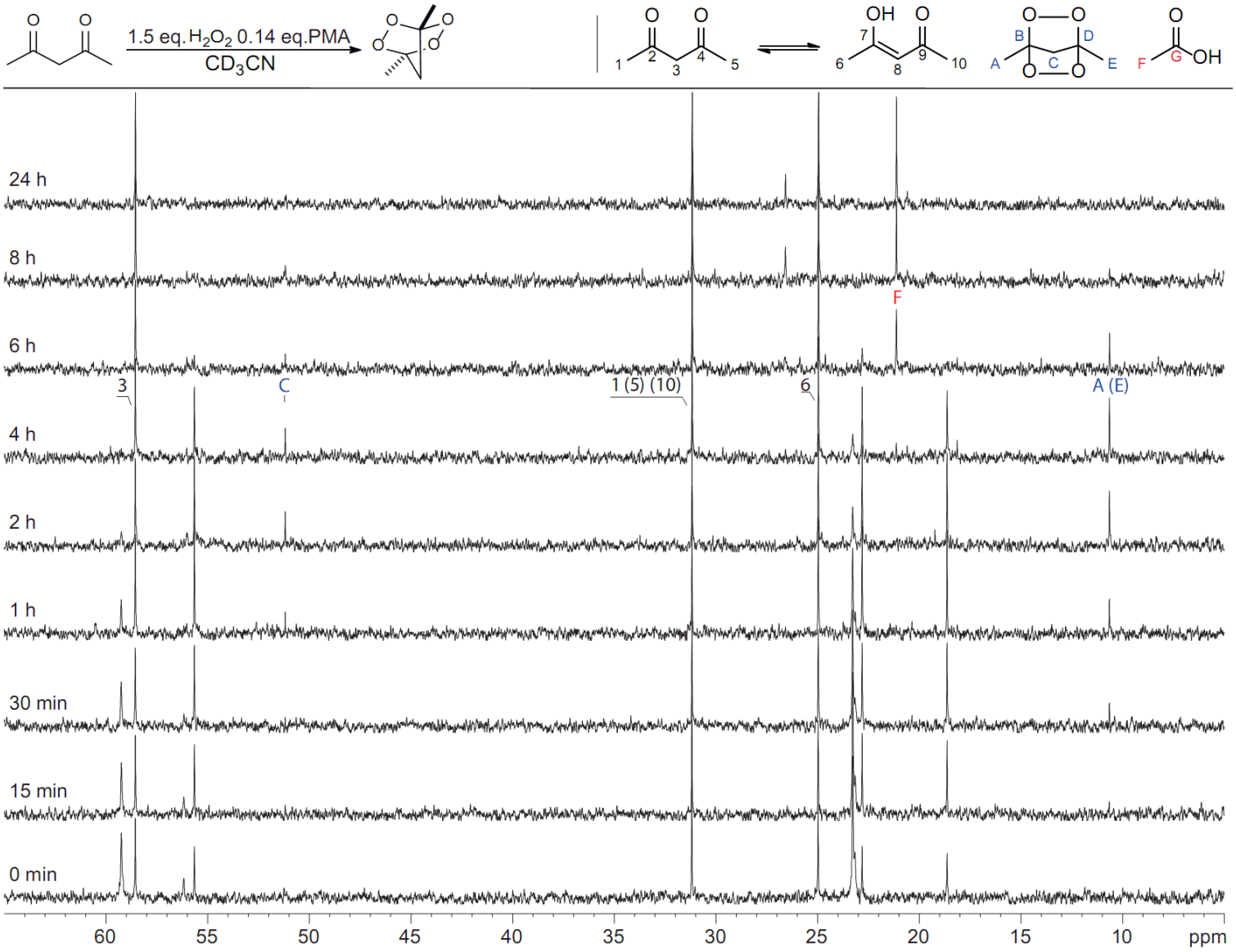
1a **2a**

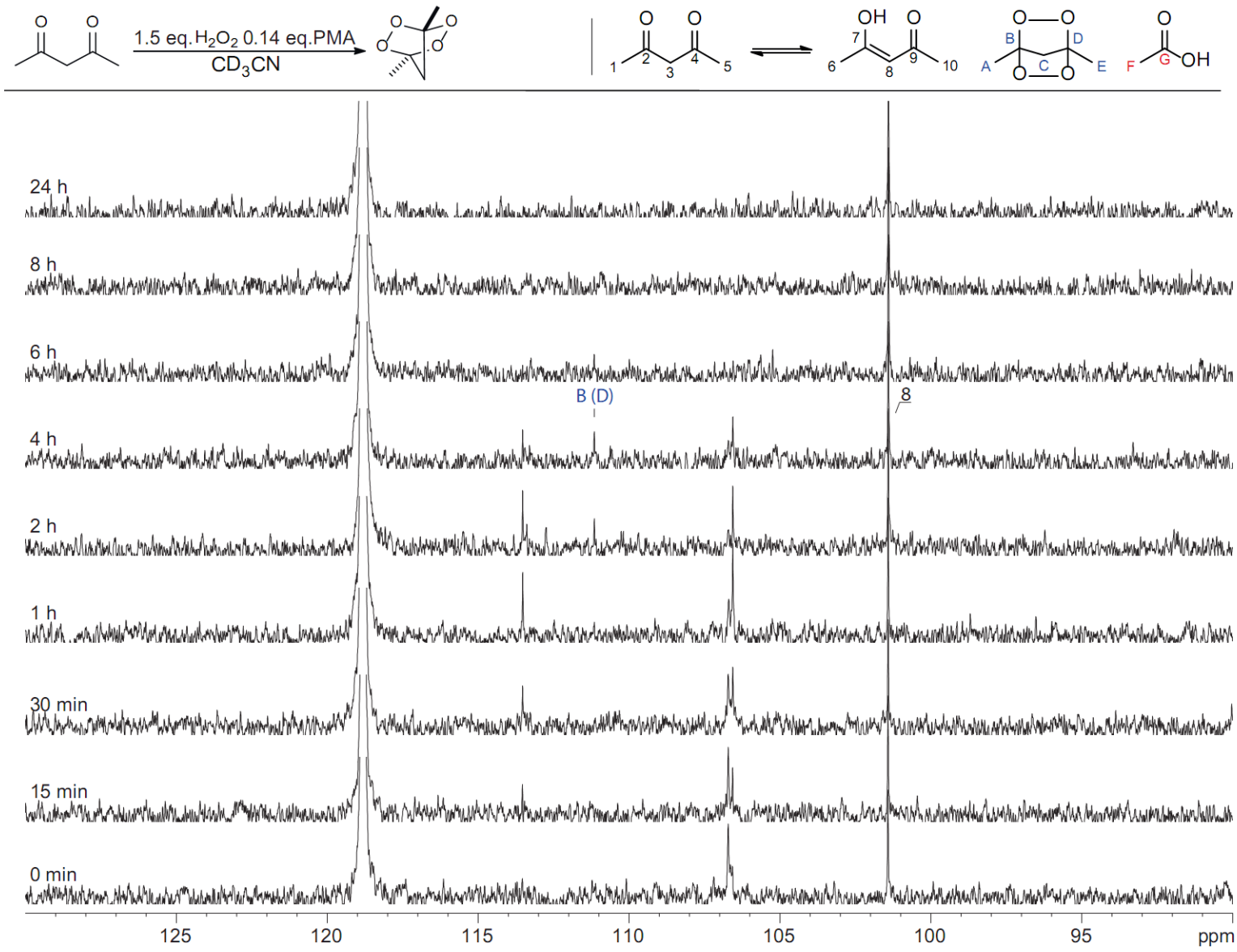
A 37% aqueous H_2O_2 solution (115.8 mg, 1.26 mmol) and phosphomolybdic acid (PMA) (133.2 mg, 0.06 mmol) were successively added to a solution of diketone **1a** (42 mg, 0.42 mmol) in CD_3CN (0.7 mL) at room temperature in an NMR tube. The course of the reaction was monitored in an NMR tube.

Pentane-2,4-dione **1a**: ^{13}C NMR (75.48 MHz, CD_3CN), δ: 24.9 (CH_3), 31.0 (CH_3), 58.7 (CH_2), 101.1 (CH), 192.7(C(O)), 204.0 (C(O)). CH_3COOH: ^{13}C NMR (75.48 MHz, CD_3CN), δ: 20.73 (CH3), 173.21 (CO). ^{13}C NMR for acetic acid [31].

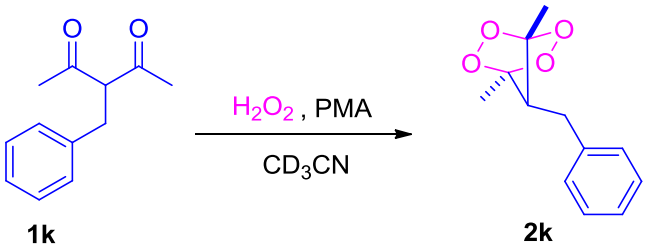
1,4-Dimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane **2a**: ^{13}C NMR (75.48 MHz, CD_3CN), δ: 10.7 (CH_3), 51.2 (CH_2), 111.0 (OCO).







Monitoring of the PMA-catalyzed reaction of 3-benzylpentane-2,4-dione 1k with H_2O_2



1k **2k**

A 37% aqueous H_2O_2 solution (55.1 mg, 0.60 mmol) and phosphomolybdic acid (PMA) (63.4 mg, 0.027 mmol) were successively added to a solution of diketone **1k** (38.2 mg, 0.20 mmol) in CD_3CN (0.64 mL) at room temperature in an NMR tube. The course of the reaction was monitored in an NMR tube.

3-Benzylpentane-2,4-dione **1k**: ^{13}C NMR (75.48 MHz, CD_3CN), δ: 23.6 (CH_3^{enol}), 30.4(CH_3), 33.3 (CH_2^{enol}), 34.5 (CH_2), 69.4 (CH), 109.6 (C^{enol}), 127.1, 127.4, 128.5, 129.5, 129.6, 129.7 (CH_{ar}), 139.8(C_{ar}), 141.3 (C_{ar}^{enol}), 193.2 (CO^{enol}), 204.9 (CO).

(1,4)-7-Benzyl-1,4-dimethyl-2,3,5,6-tetraoxabicyclo[2.2.1]heptane **2k:** ^{13}C NMR (75.48 MHz, CD_3CN), δ: 10.0 (CH_3), 30.8 (CH_2), 60.0 (CH), 111.7 (OCO), 118.24, 127.6, 129.6, 130.0 (CH_{ar}), 138.9 (C_{ar}).