

Supporting Information

Reactions of Mono- and Bicyclic Enol Ethers

with the I₂ – Hydroperoxide System

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Ashot V. Arzumanyan, and Gennady I. Nikishin**

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47 Leninsky prosp., 119991 Moscow, Russian Federation

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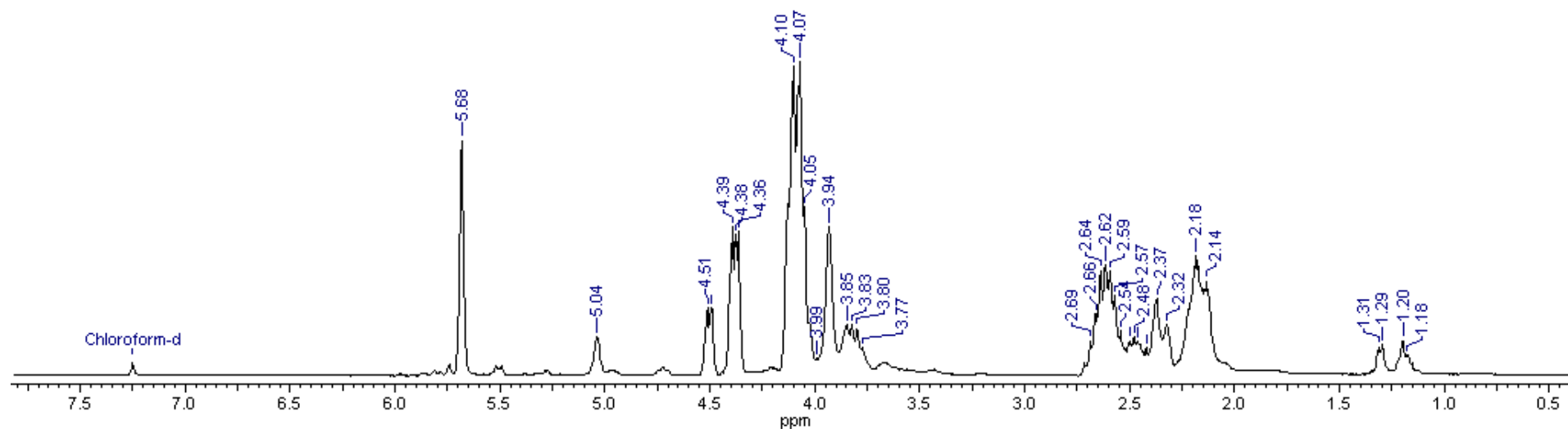
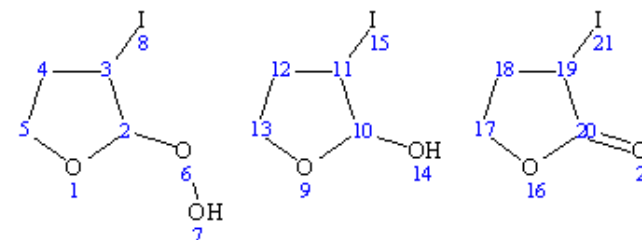
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¹H NMR Mixture of products 7a-9a (reaction of 3,4-dihydrofuran 4a with I₂/H₂O₂ system)

4 Jul 2013

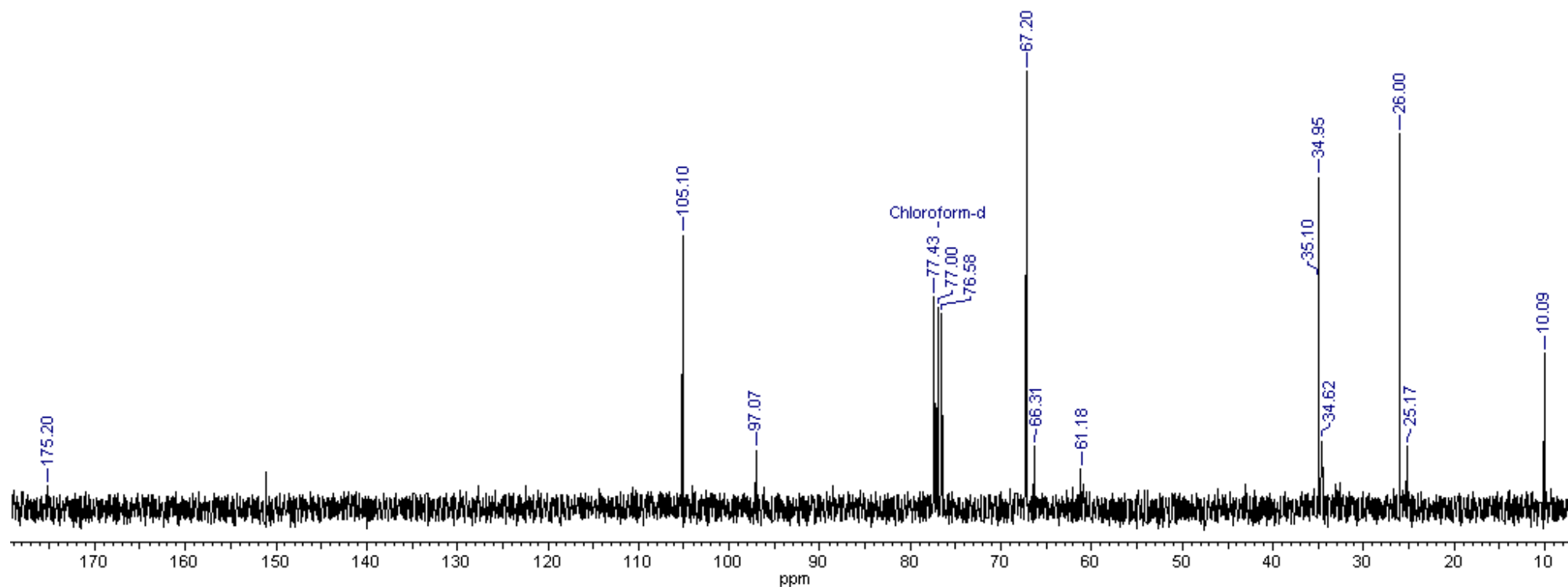
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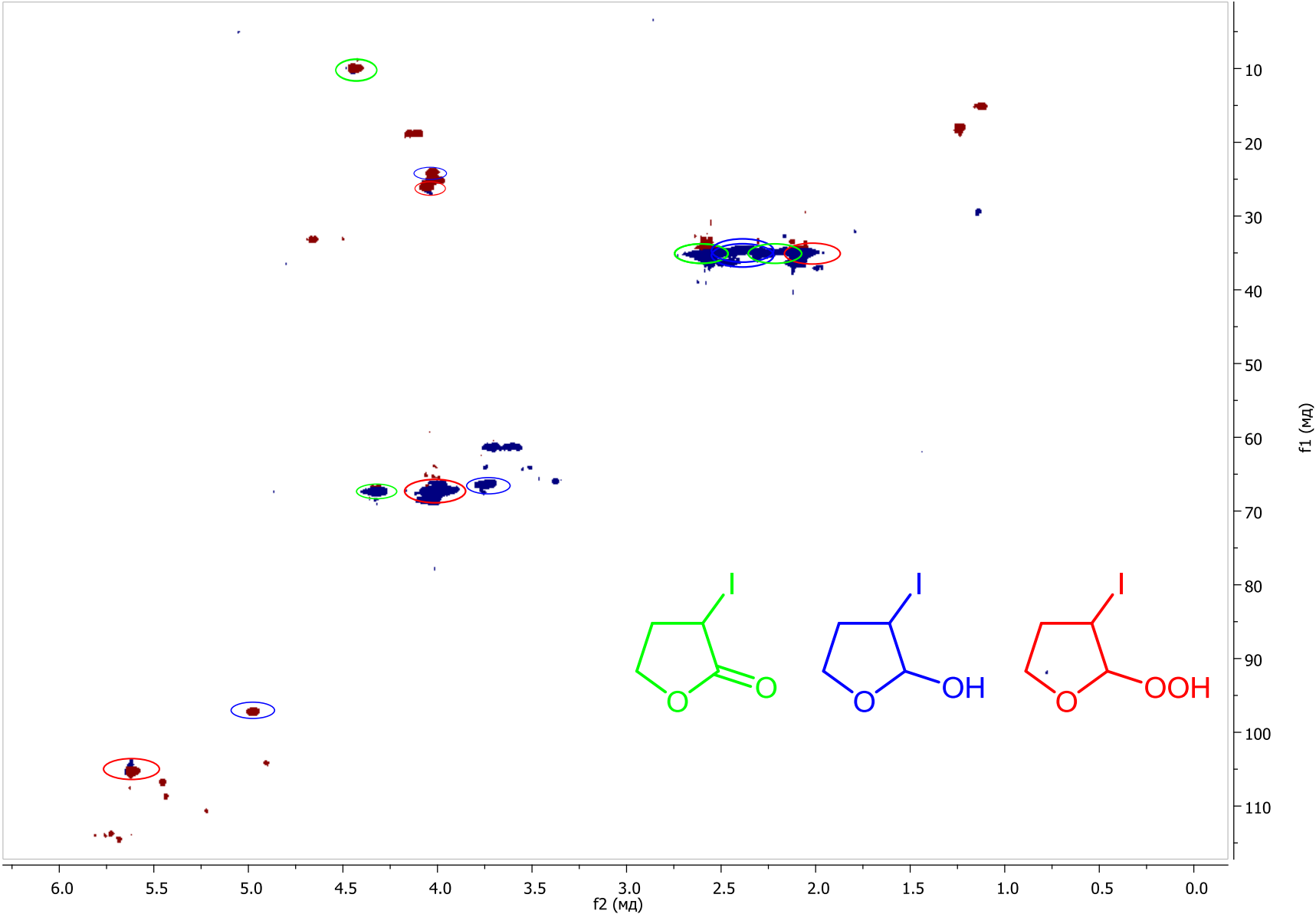
¹³C NMR Mixture of products 7a-9a (reaction of 3,4-dihydrofuran 4a with I₂/H₂O₂ system)

4 Jul 2013

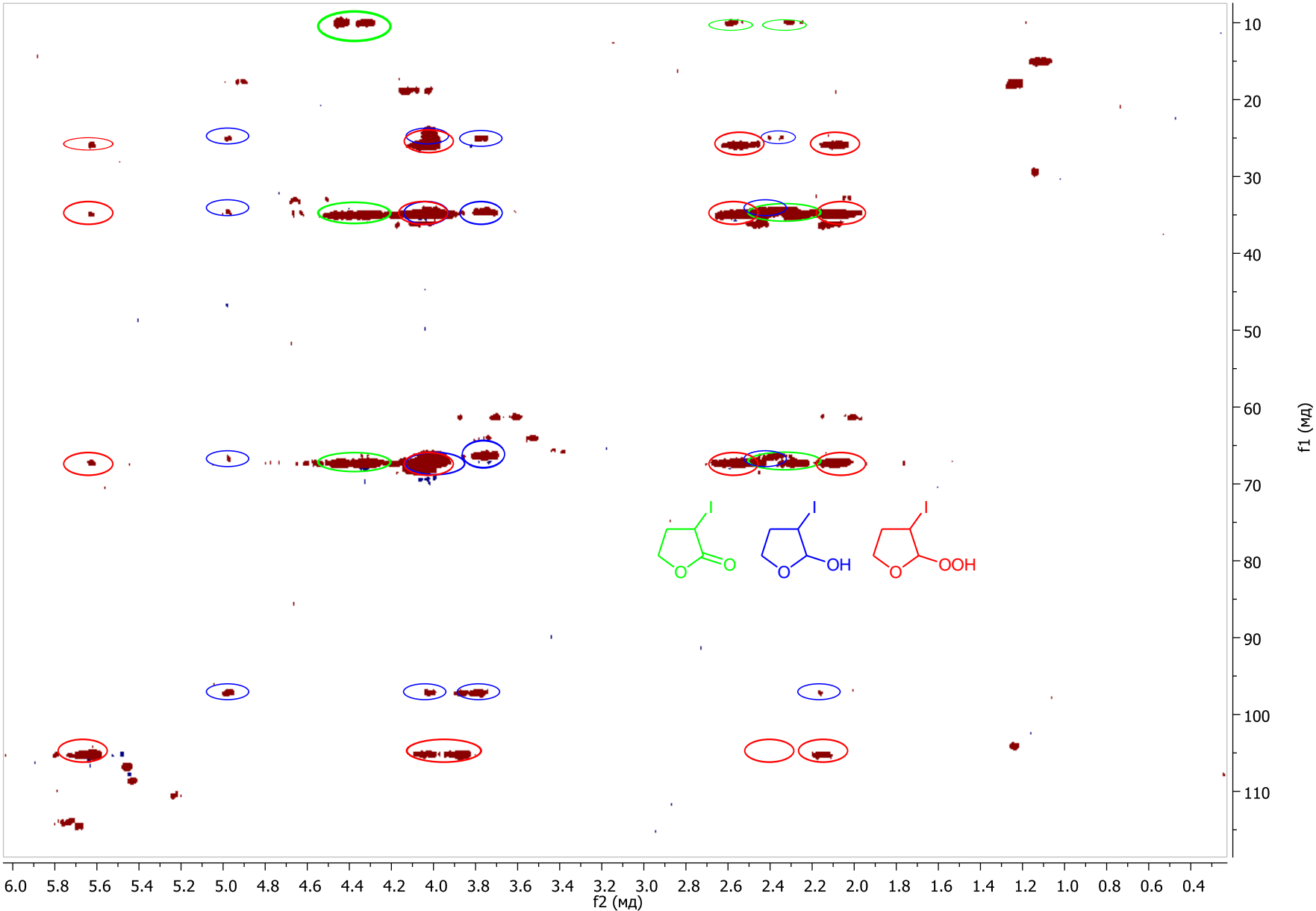
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Solvent	CHLOROFORM-D	Sweep Width (Hz)	18115.94	Temperature (degree C)	30.100



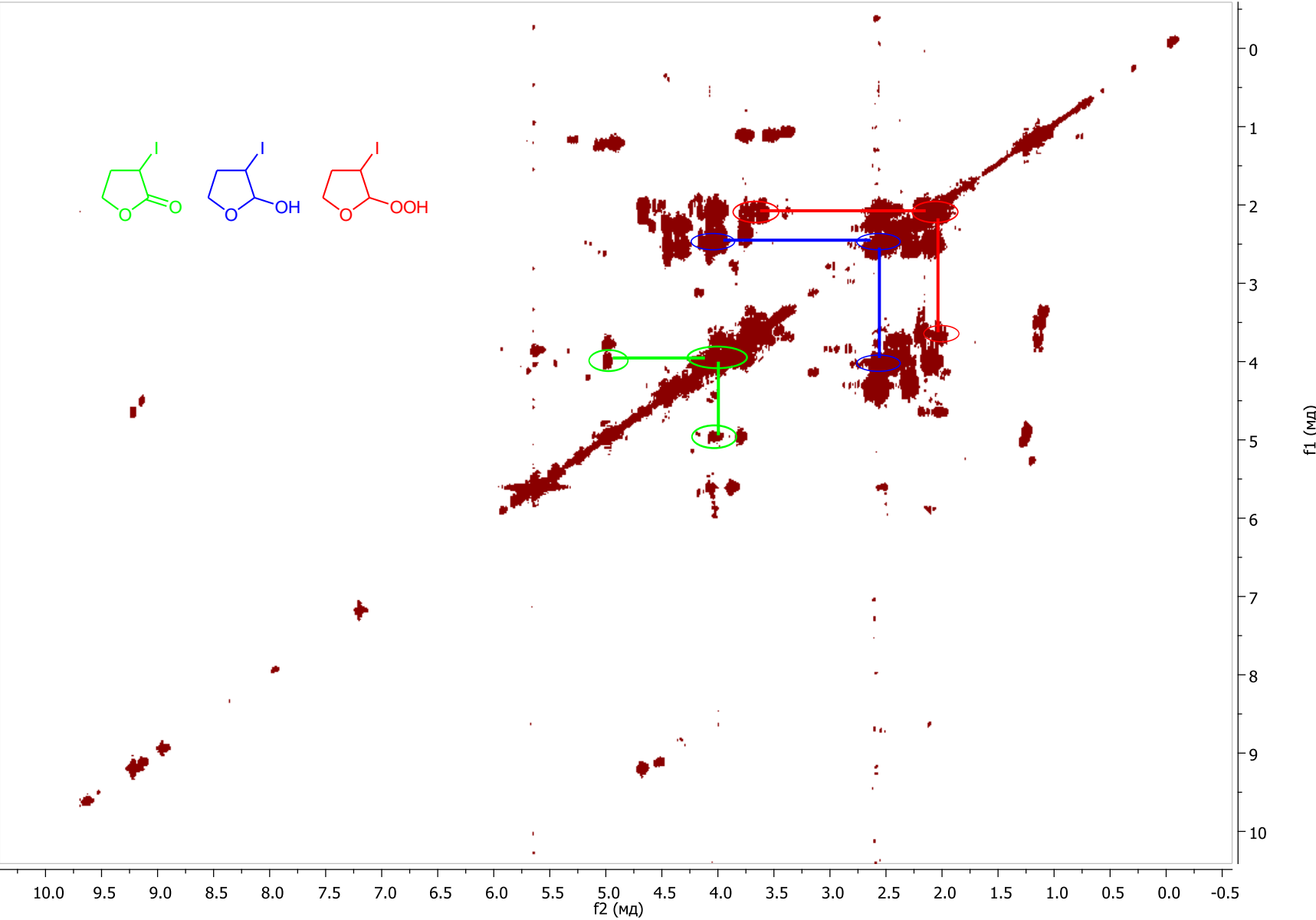
HSQC Mixture of products 7a-9a (reaction of 3,4-dihydrofuran 4a with I₂/H₂O₂ system)



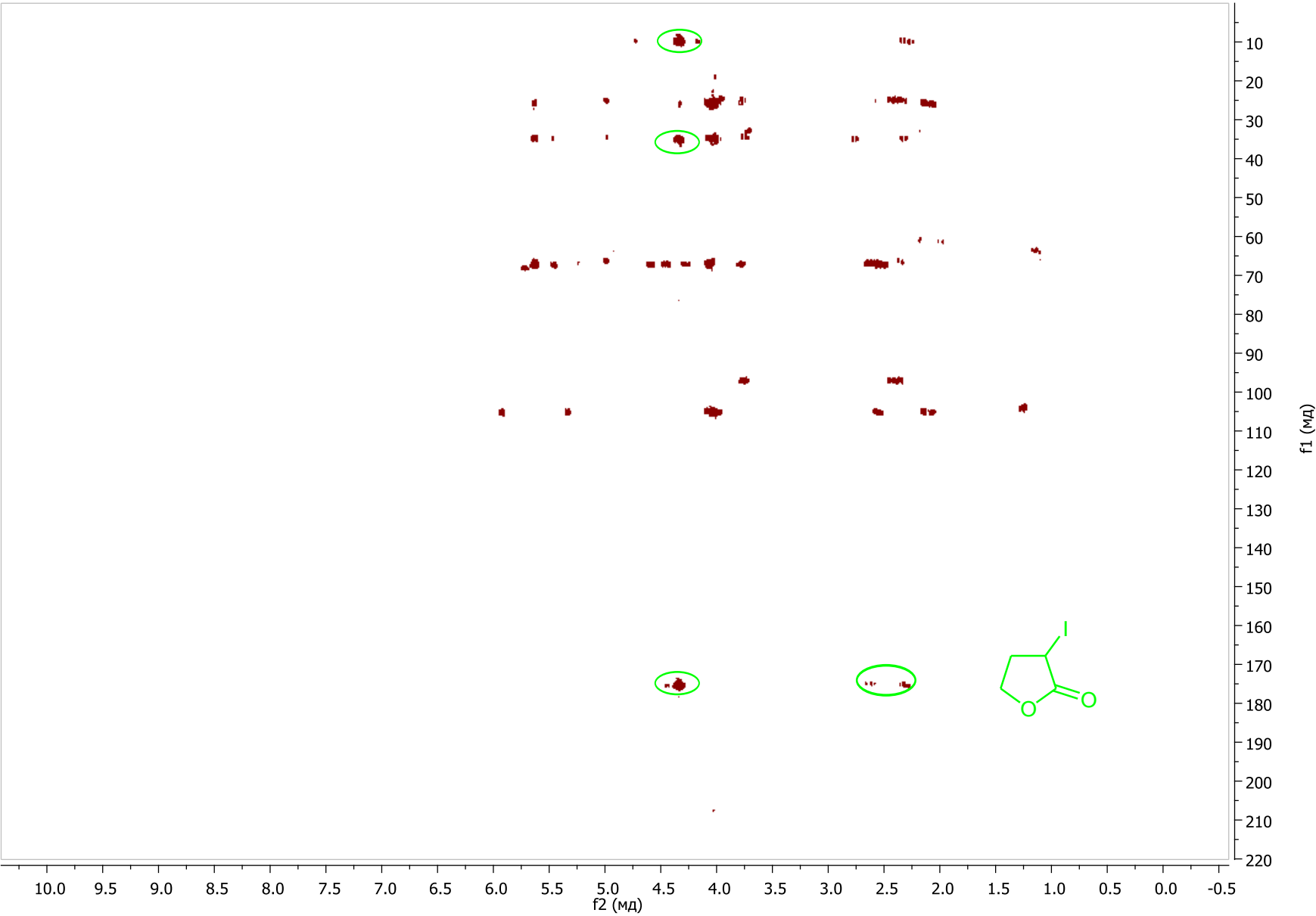
HSQC-TOCSY Mixture of products 7a-9a (reaction of 3,4-dihydrofuran 4a with I₂/H₂O₂ system)



COSY Mixture of products 7a-9a (reaction of 3,4-dihydrofuran 4a with I₂/H₂O₂ system)



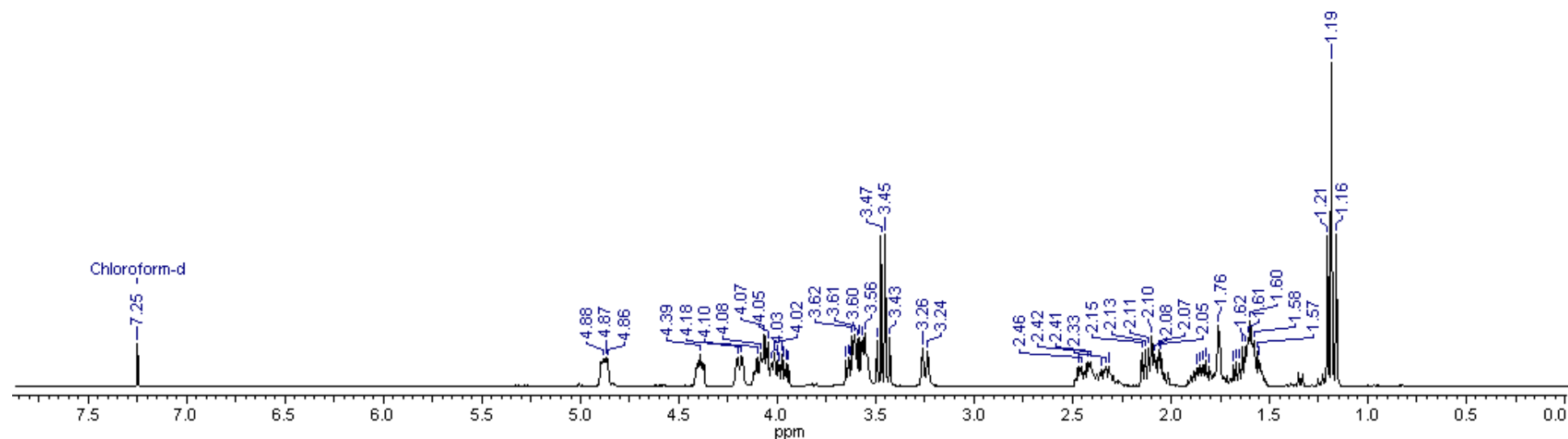
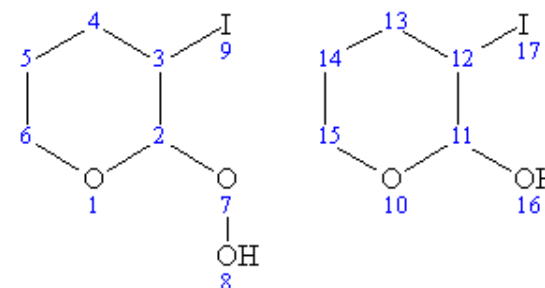
HMBC Mixture of products 7a-9a (reaction of 3,4-dihydrofuran 4a with I₂/H₂O₂ system)



¹H NMR Mixture of products 7b-8b (reaction of 3,4-dihydropyran 4b with I₂/H₂O₂ system)

4 Jul 2013

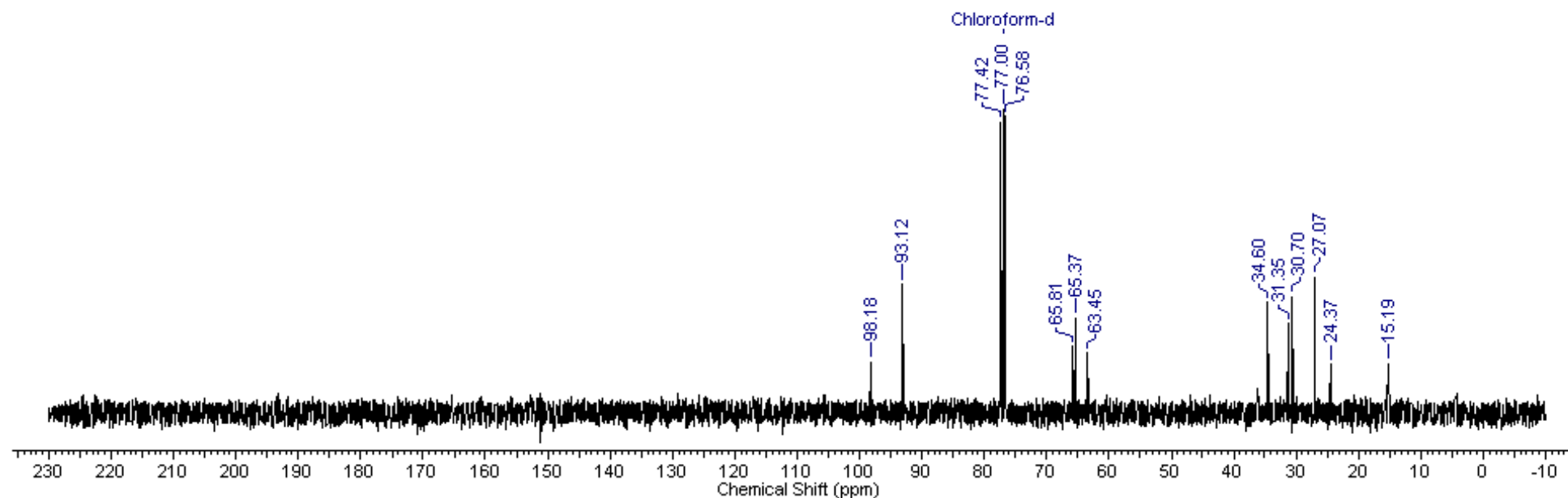
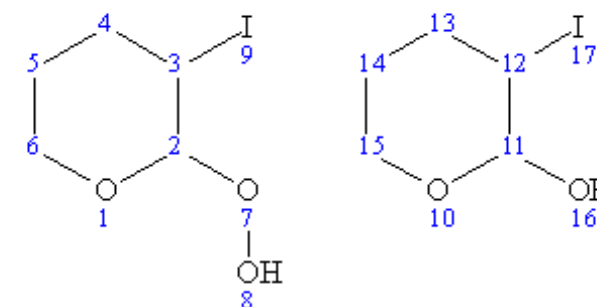
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				Temperature (degree C)	30.000



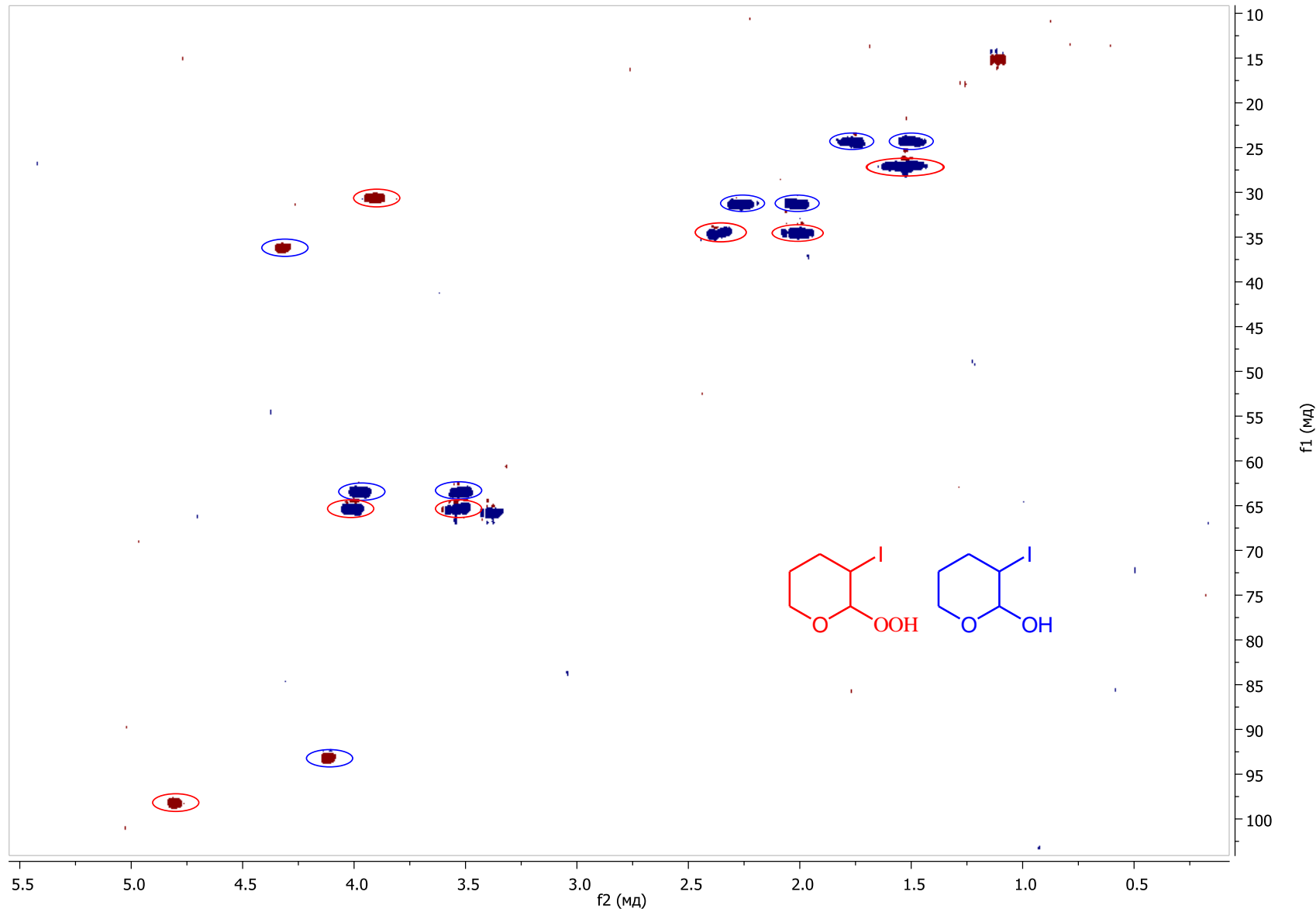
¹³C NMR Mixture of products 7b-8b (reaction of 3,4-dihydropyran 4b with I₂/H₂O₂ system)

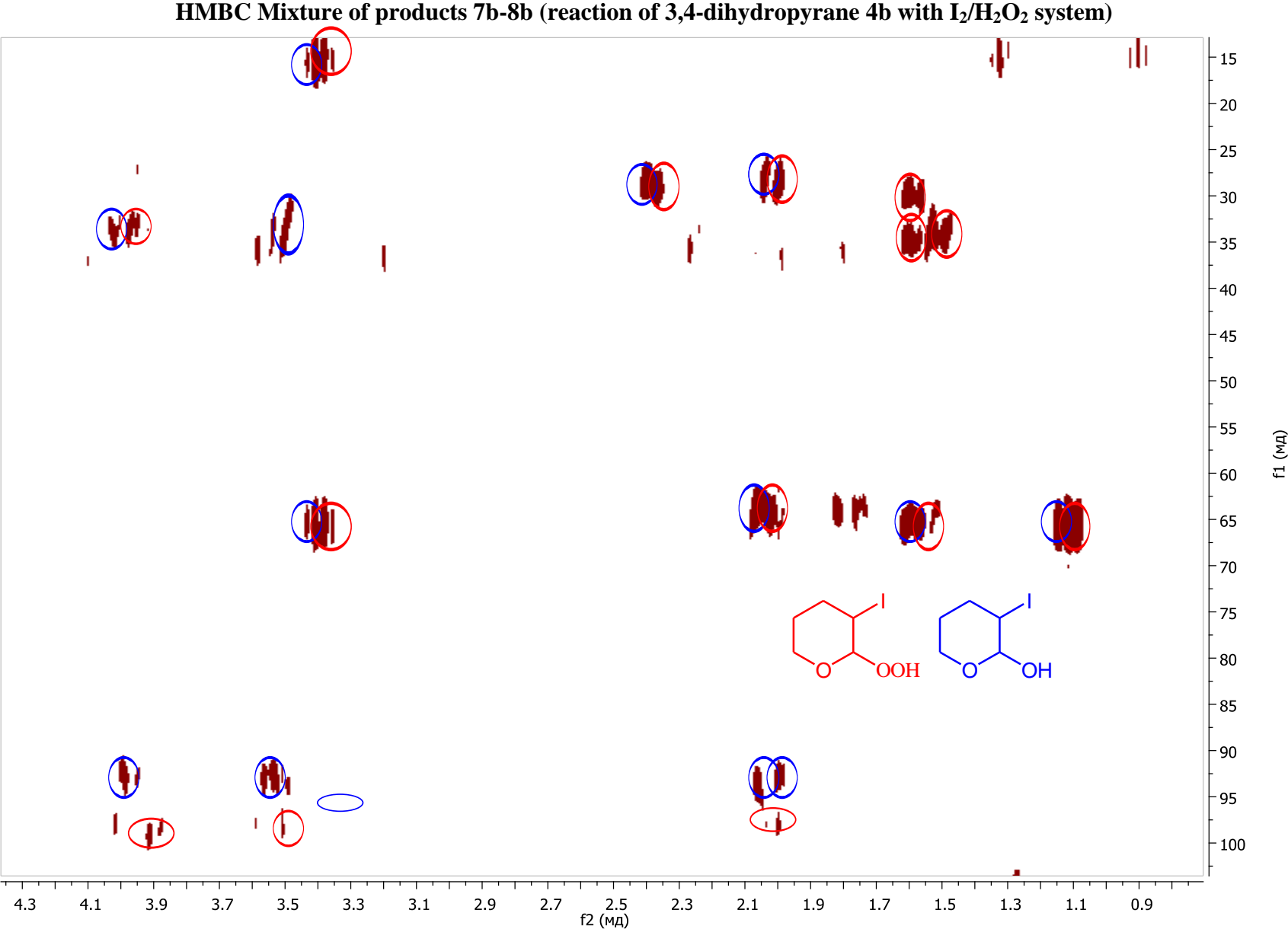
26 Sep 2013

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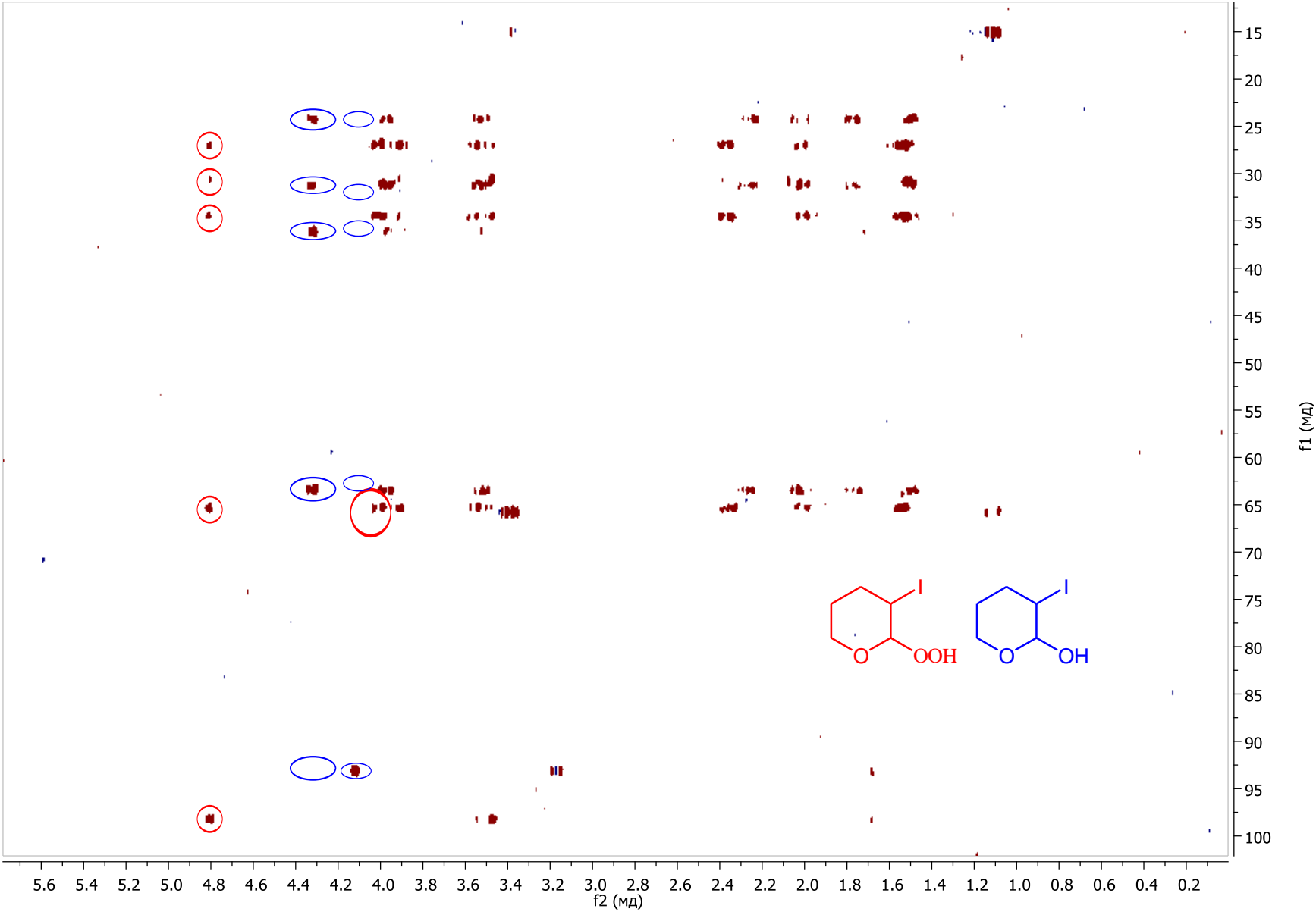


HSQC Mixture of products 7b-8b (reaction of 3,4-dihydropyran 4b with I₂/H₂O₂ system)

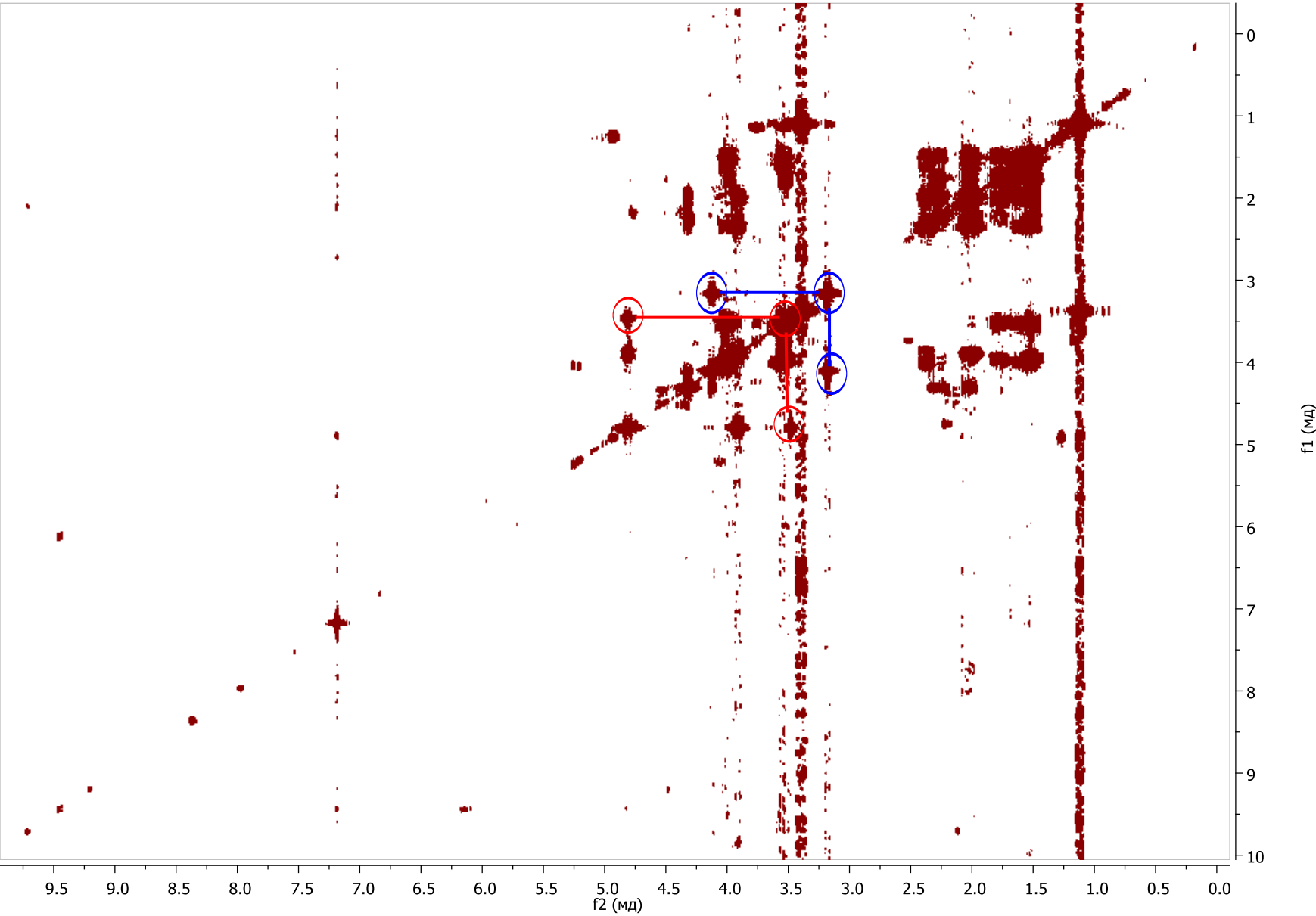




HSQC-TOCSY Mixture of products 7b-8b (reaction of 3,4-dihydropyran 4b with I₂/H₂O₂ system)



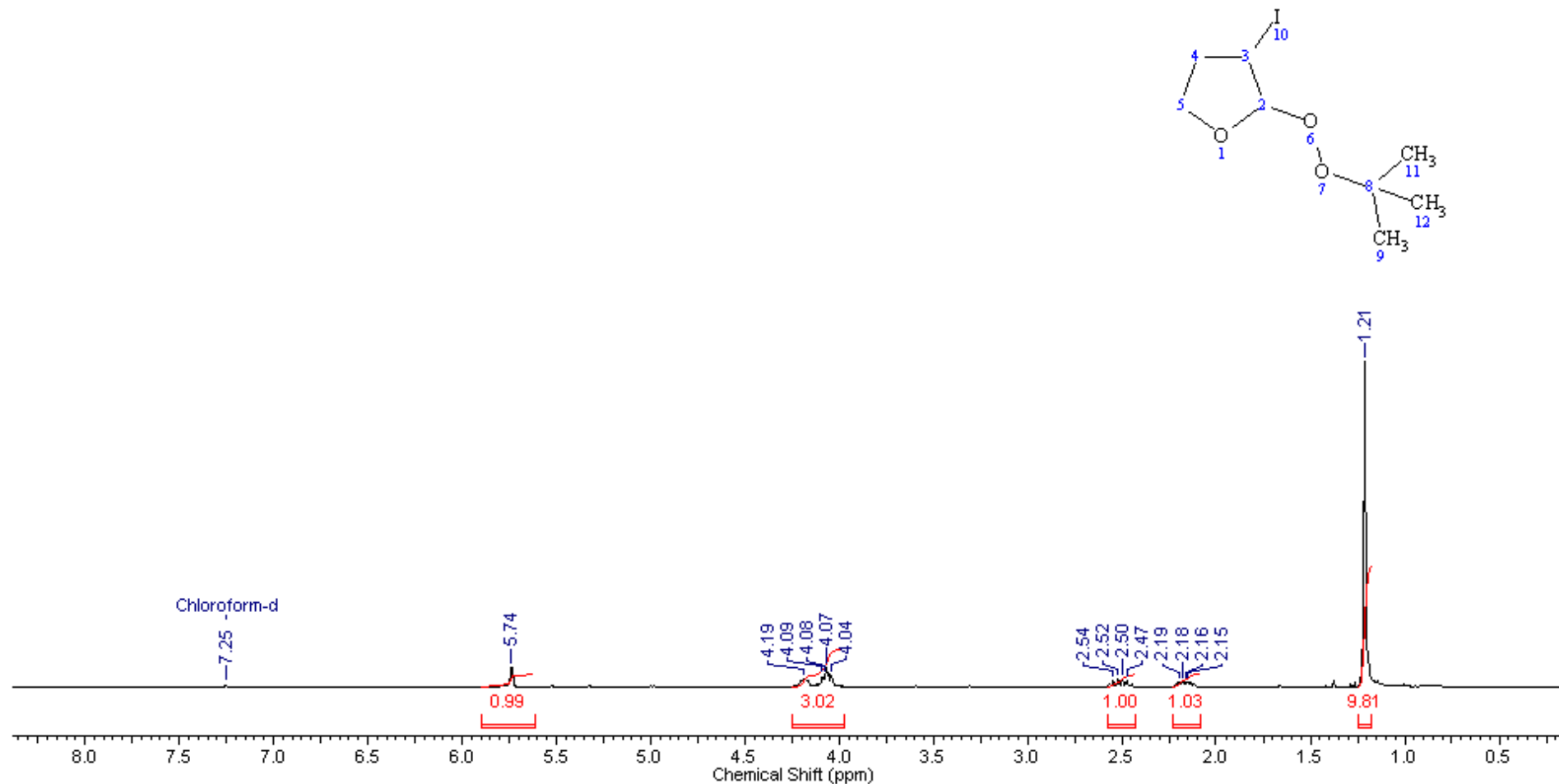
COSY Mixture of products 7b-8b (reaction of 3,4-dihydropyrane 4b with I₂/H₂O₂ system)



¹H NMR 2-(*tert*-Butylperoxy)-3-iodotetrahydrofuran (5a)

25 Sep 2013

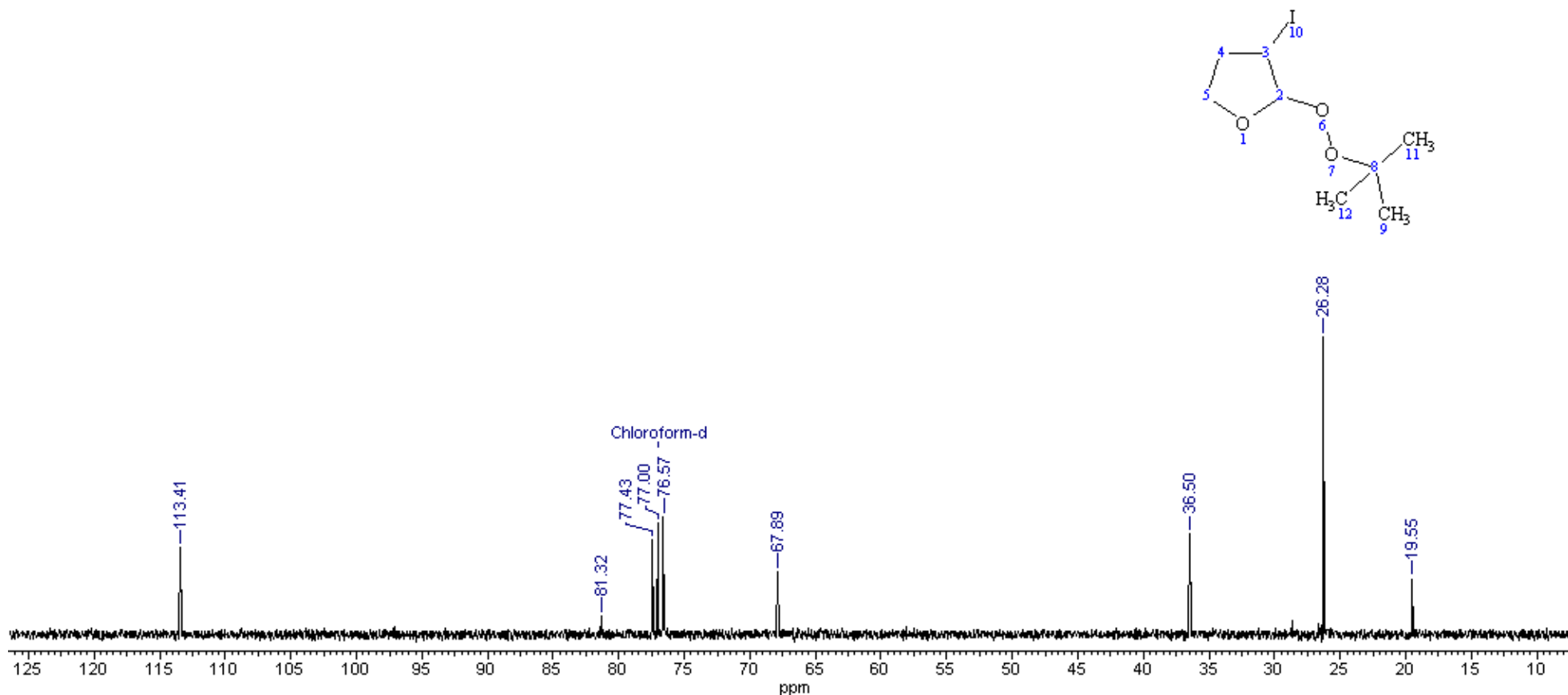
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Pulse Sequence	zg	Solvent	CHLOROFORM-D	Sweep Width (Hz)	6009.62
				Points Count	8192
				Temperature (degree C)	27.500



¹³C NMR 2-(*tert*-Butylperoxy)-3-iodotetrahydrofuran (5a)

2 Jul 2013

Acquisition Time (sec)	0.4322	Comment	Avance-300, C-13, CDCl ₃	Date	16 Aug 2012 09:48:48
Frequency (MHz)	75.48	Nucleus	13C	Number of Transients	99
Points Count	16384	Pulse Sequence	zgpg60base	Solvent	CHLOROFORM-D
Temperature (degree C)	28.400			Original Points Count	16308
				Sweep Width (Hz)	18867.92



HRMS 2-(*tert*-Butylperoxy)-3-iodotetrahydrofuran (5a)

Display Report

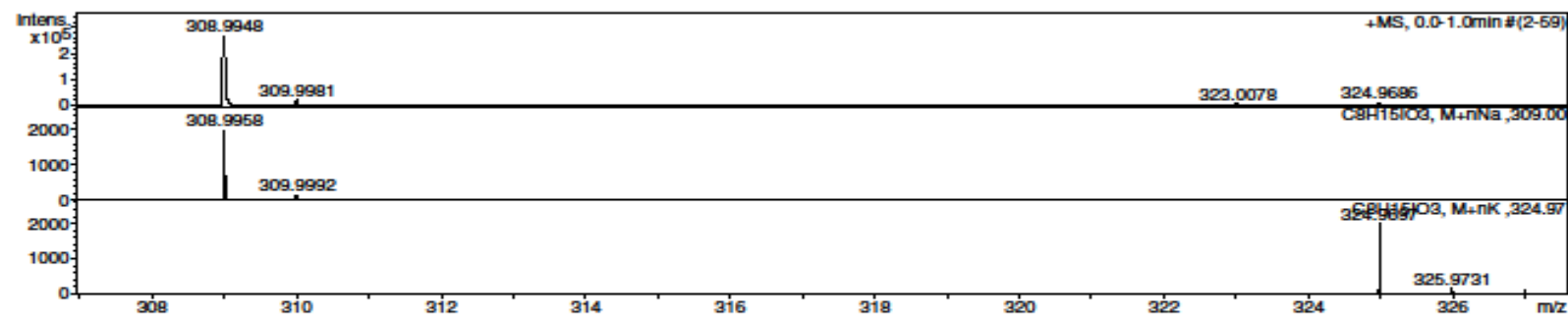
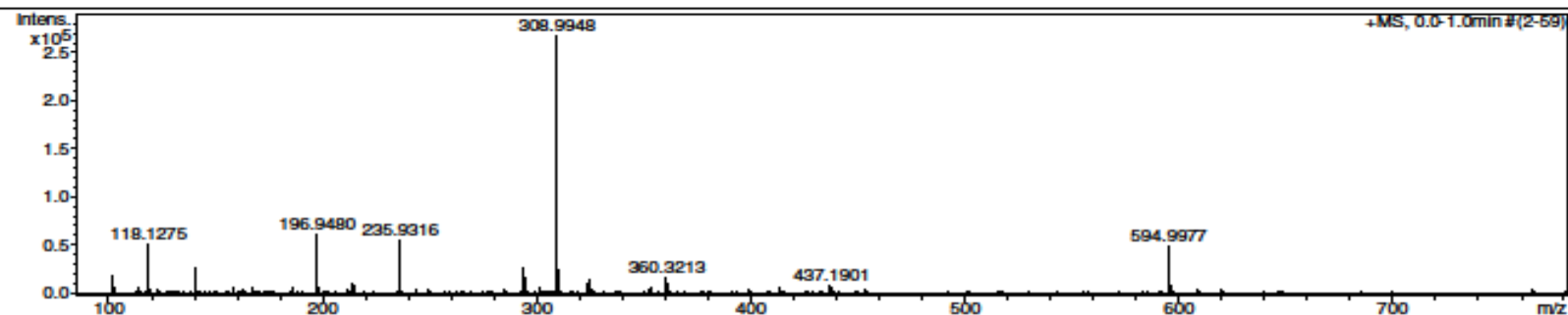
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Method tune_low.m
Sample Name /TERN ZD4
Comment MeOH no clb added

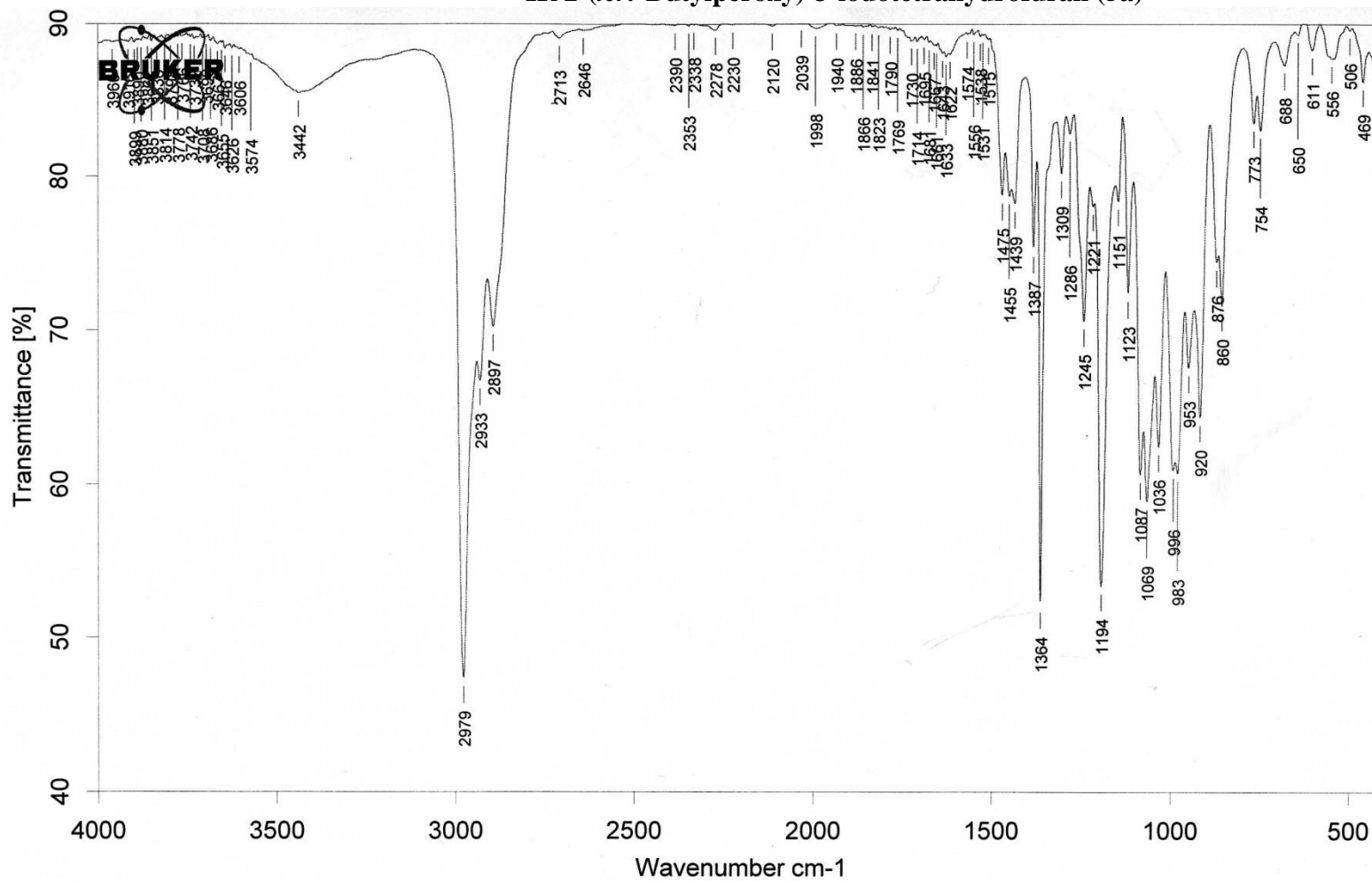
Acquisition Date 8/13/2012 5:05:38 PM
Operator BDAL@DE
Instrument / Ser# micrOTOF 10248

Acquisition Parameter

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Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
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IR 2-(*tert*-Butylperoxy)-3-iodotetrahydrofuran (5a)



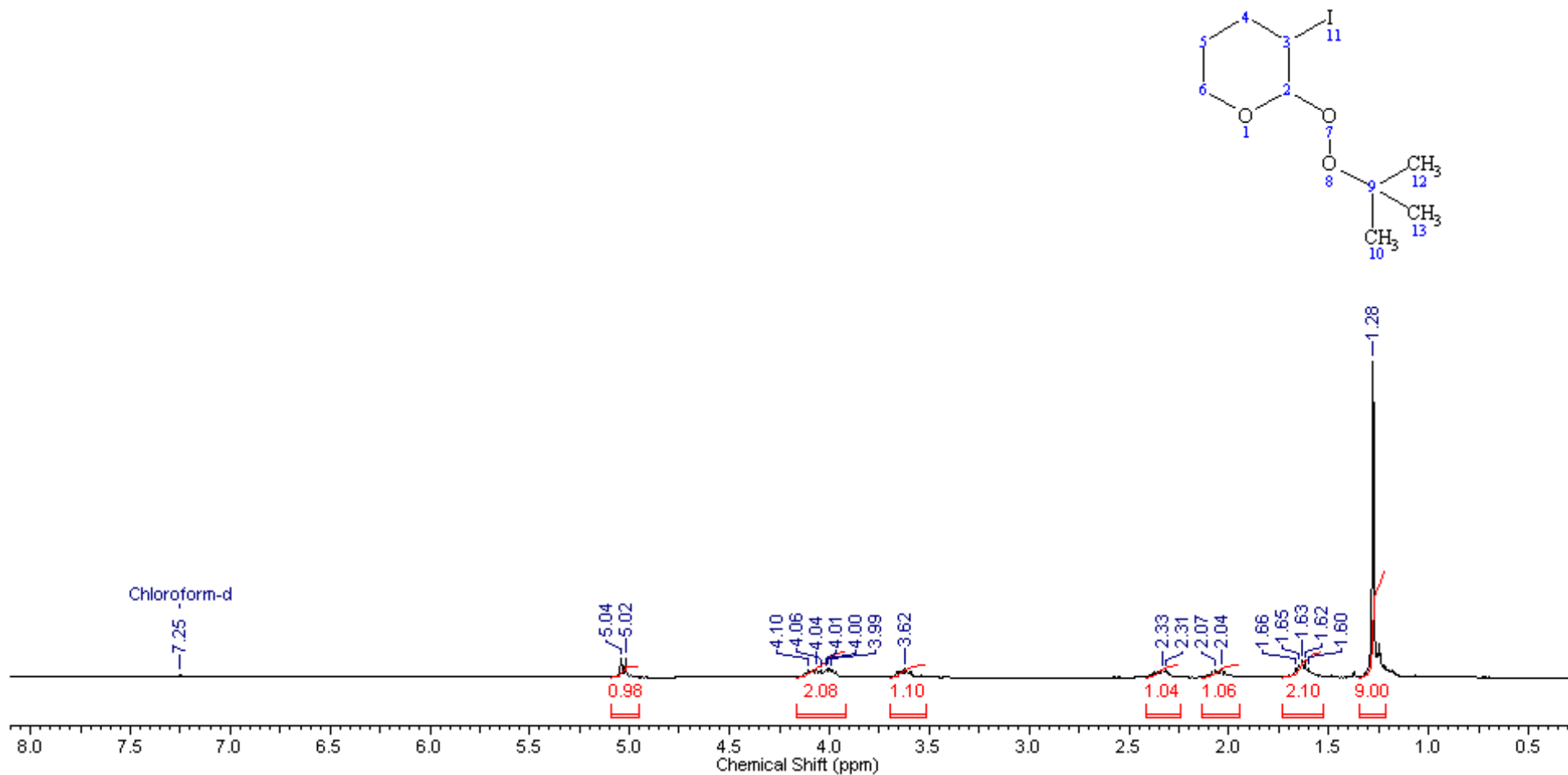
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17/10/2012

¹H NMR 2-(*tert*-Butylperoxy)-3-iodotetrahydro-2*H*-pyran (5b)

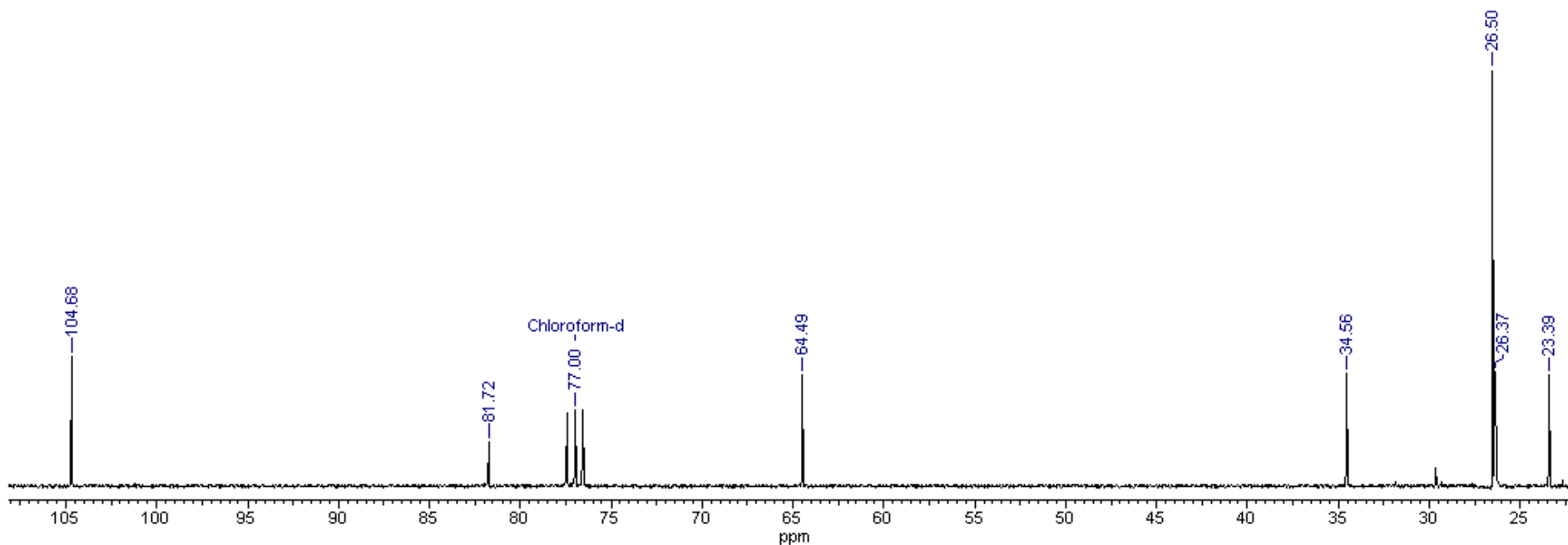
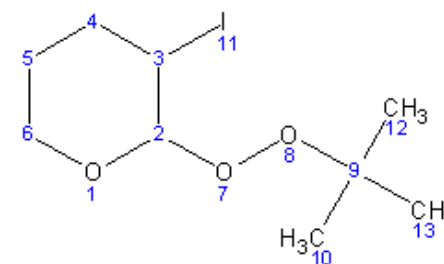
24 Sep 2013

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Solvent	CHLOROFORM-D	Sweep Width (Hz)	6009.62
		Number of Transients	1
		Pulse Sequence	zg
		Temperature (degree C)	27.700



¹³C NMR 2-(*tert*-Butylperoxy)-3-iodotetrahydro-2*H*-pyran (5b)

Acquisition Time (sec)	0.4501	Comment	Avance-300, C-13, CDCl3	Date	05 May 2008 12:37:20
File Name					
Frequency (MHz)	75.48	Nucleus	13C	Number of Transients	2444
Original Points Count	16308	Points Count	16384	Pulse Sequence	zgpg30base
Solvent	CHLOROFORM-D	Sweep Width (Hz)	18115.94	Temperature (degree C)	24.100



HRMS 2-(*tert*-Butylperoxy)-3-iodotetrahydro-2*H*-pyran (5b)

Display Report

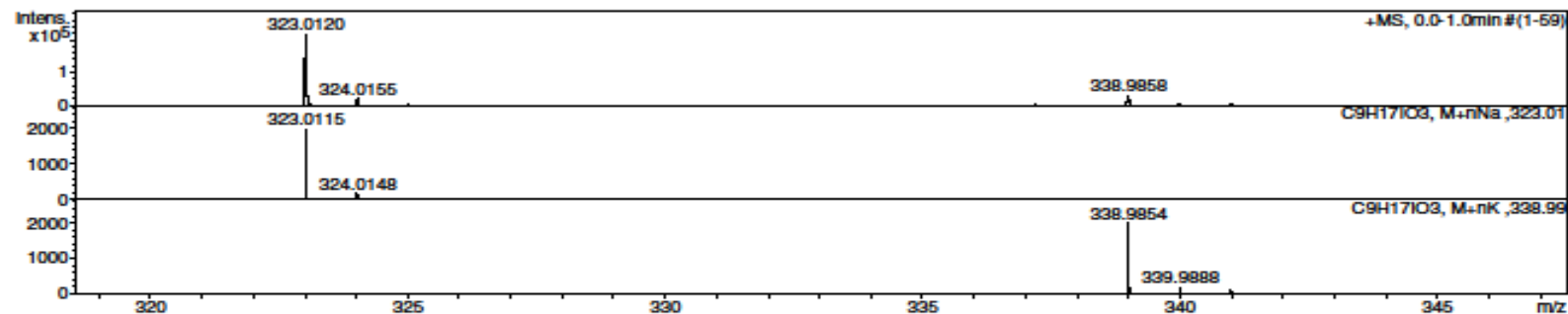
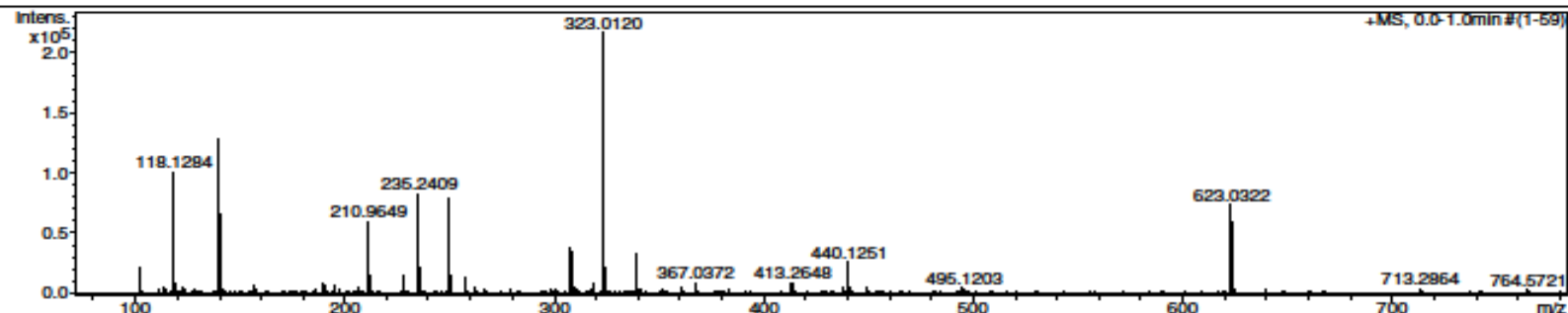
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Sample Name /TERN ZD12
Comment MeOH no clb added

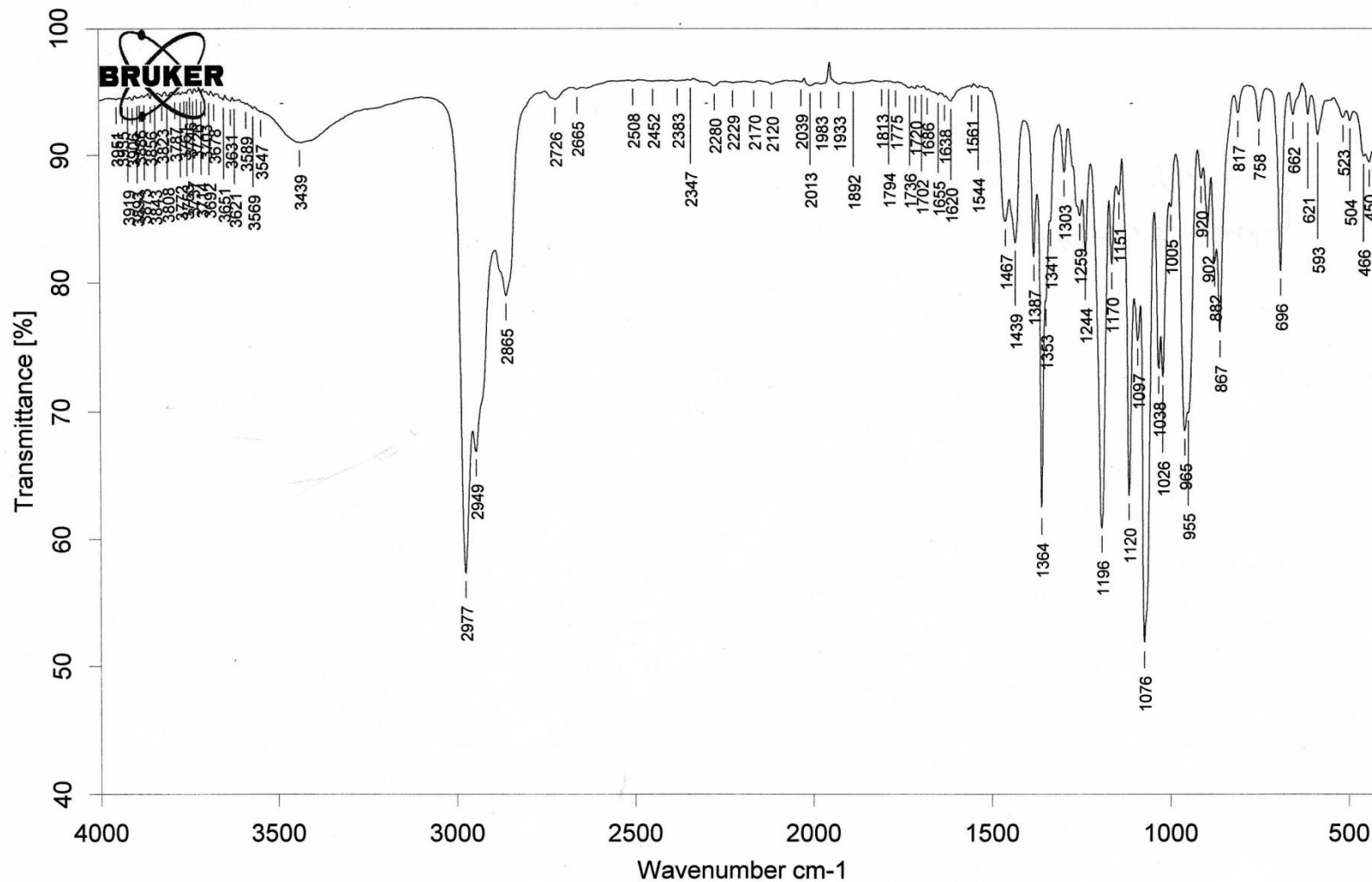
Acquisition Date 8/13/2012 4:51:02 PM
Operator BDAL@DE
Instrument / Ser# micrOTOF 10248

Acquisition Parameter

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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



IR 2-(*tert*-Butylperoxy)-3-iodotetrahydro-2*H*-pyran (5b)



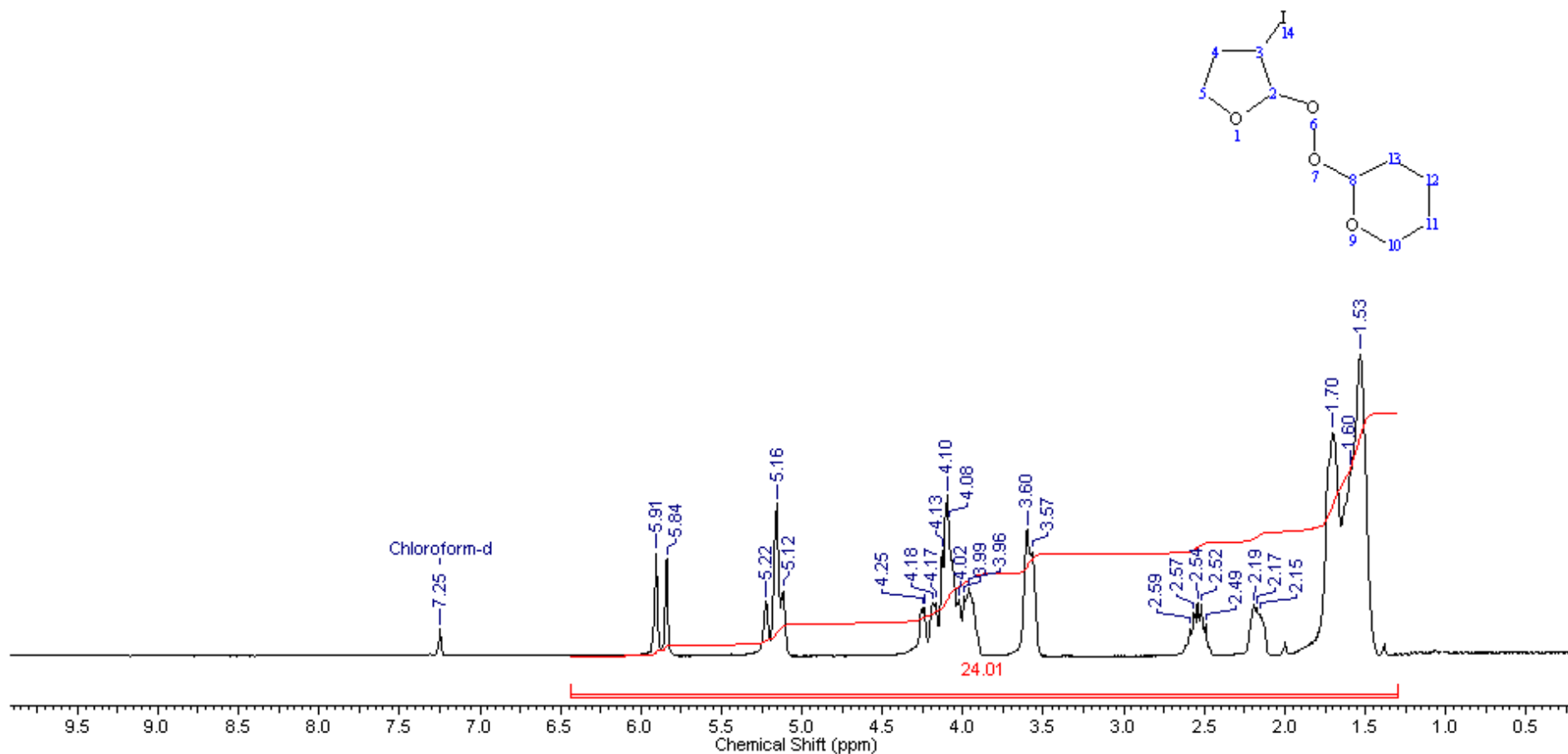
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17/10/2012

¹H NMR 2-[(3-Iodotetrahydrofuran-2-yl)peroxy]tetrahydro-2H-pyran (6a)

25 Sep 2013

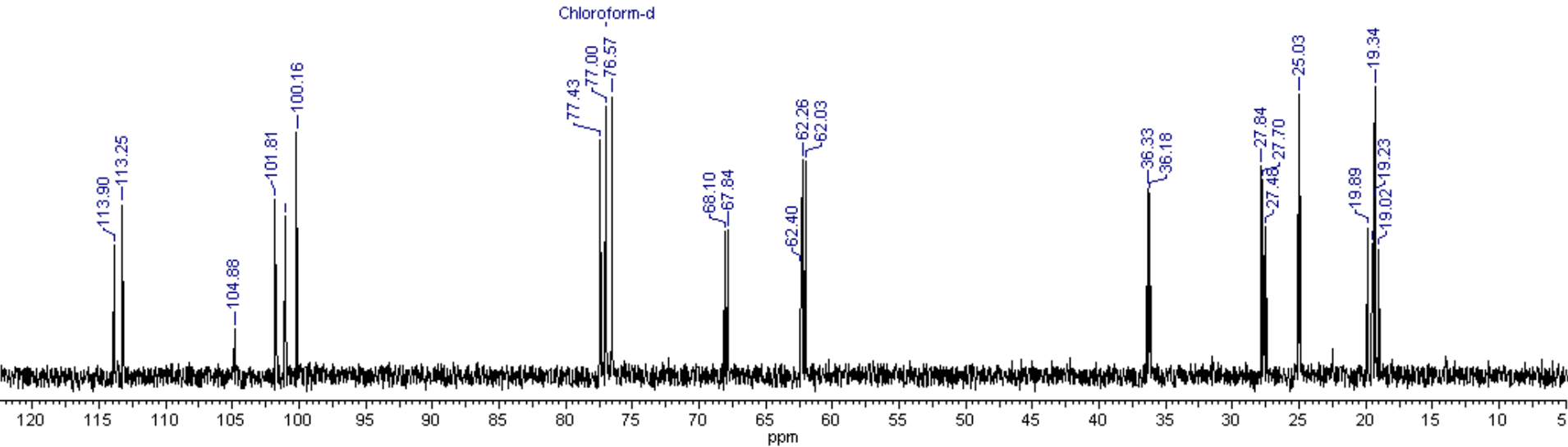
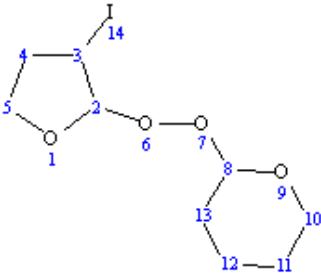
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Original Points Count	8124	Points Count	8192	Pulse Sequence zgpg30
Solvent	CHLOROFORM-D	Sweep Width (Hz)	6009.62	Temperature (degree C) 27.000



¹³C NMR 2-[(3-Iodotetrahydrofuran-2-yl)peroxy]tetrahydro-2H-pyran (6a)

2 Jul 2013

Acquisition Time (sec)	0.4322	Comment	NMR/50075242	Date	10 Nov 2012 09:18:56
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Points Count	16384	Pulse Sequence	zgpg60base	Solvent	CHLOROFORM-D
Temperature (degree C)	27.800			Original Points Count	16308
				Sweep Width (Hz)	18867.92



No.	Annotation	(ppm)
1	Chloroform-d	77.00

HRMS 2-[(3-Iodotetrahydrofuran-2-yl)peroxy]tetrahydro-2H-pyran (6a)

Display Report

Analysis Info

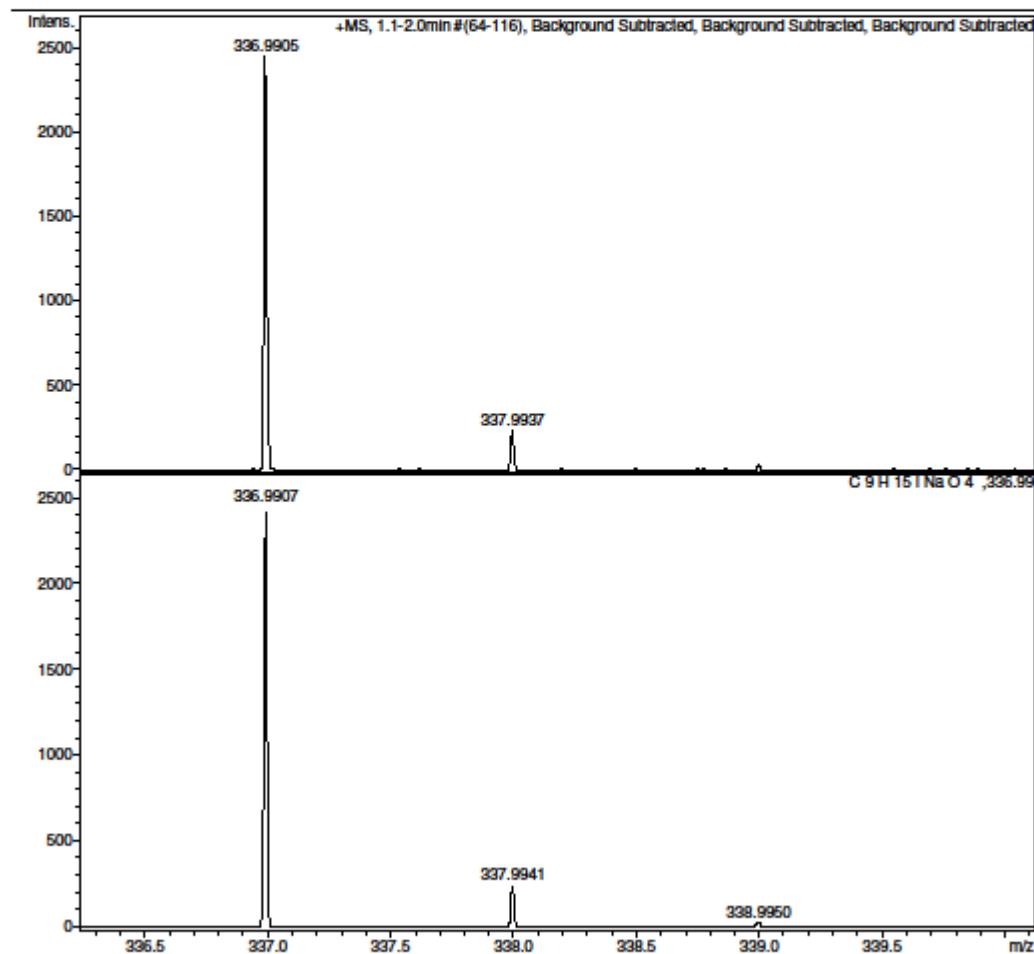
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Method tune_wide.m
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Comment Sample name: zd-120
Sample owner: Zdvigkov
Solvent: MeCN
Conc.: approx. 0.18 ng/ml

Acquisition Date 11/23/2012 12:01:50 PM

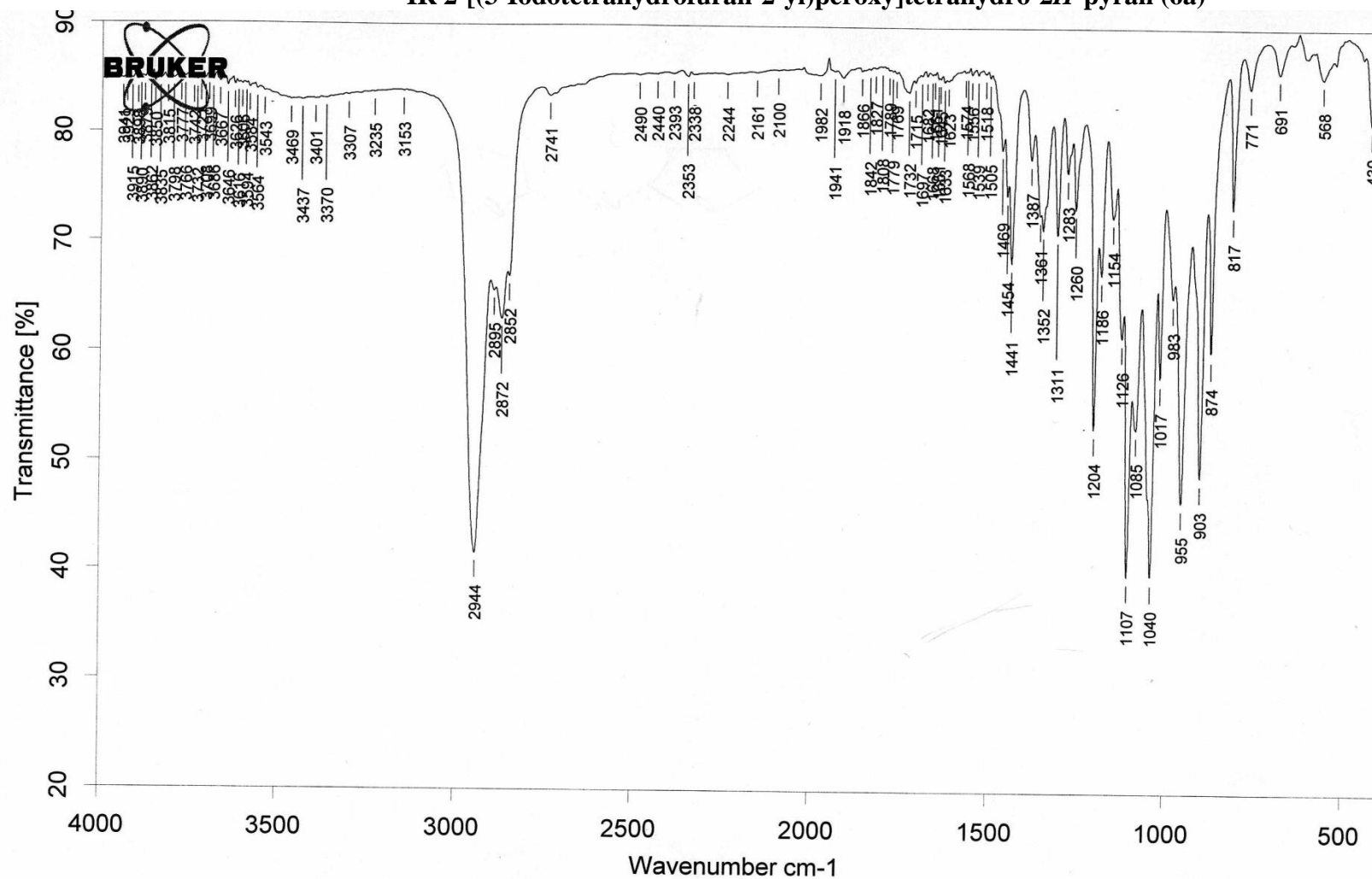
Operator BDAL@DE
Instrument / Ser# maXis 43

Acquisition Parameter

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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



IR 2-[(3-Iodotetrahydrofuran-2-yl)peroxy]tetrahydro-2H-pyran (6a)



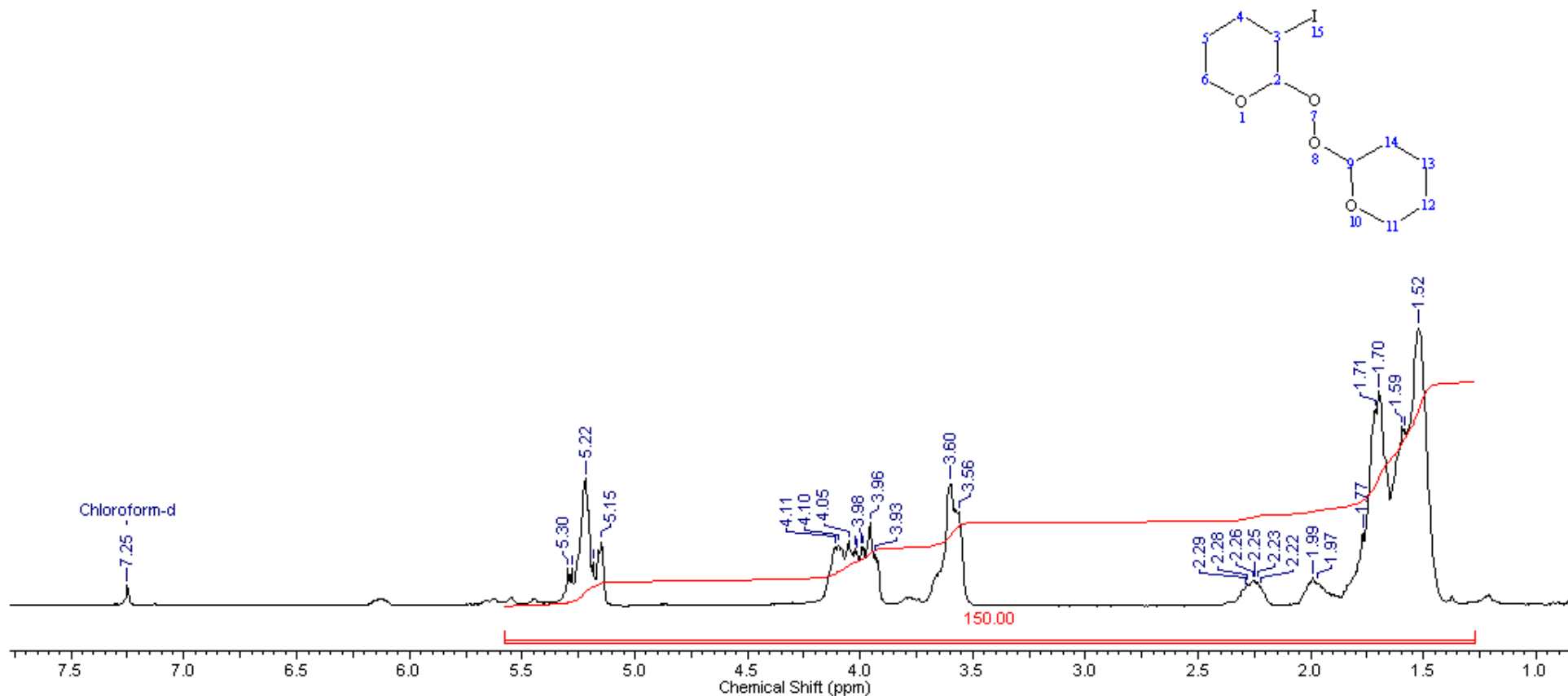
C:\Data\EDL\ZD-120.0 ZDVIZHKOV. ZD-120, thin layer on KBr.

11/12/2012

¹H NMR 3-Iodo-2-(tetrahydro-2H-pyran-2-ylperoxy)tetrahydro-2H-pyran (6b)

4 Jan 2014

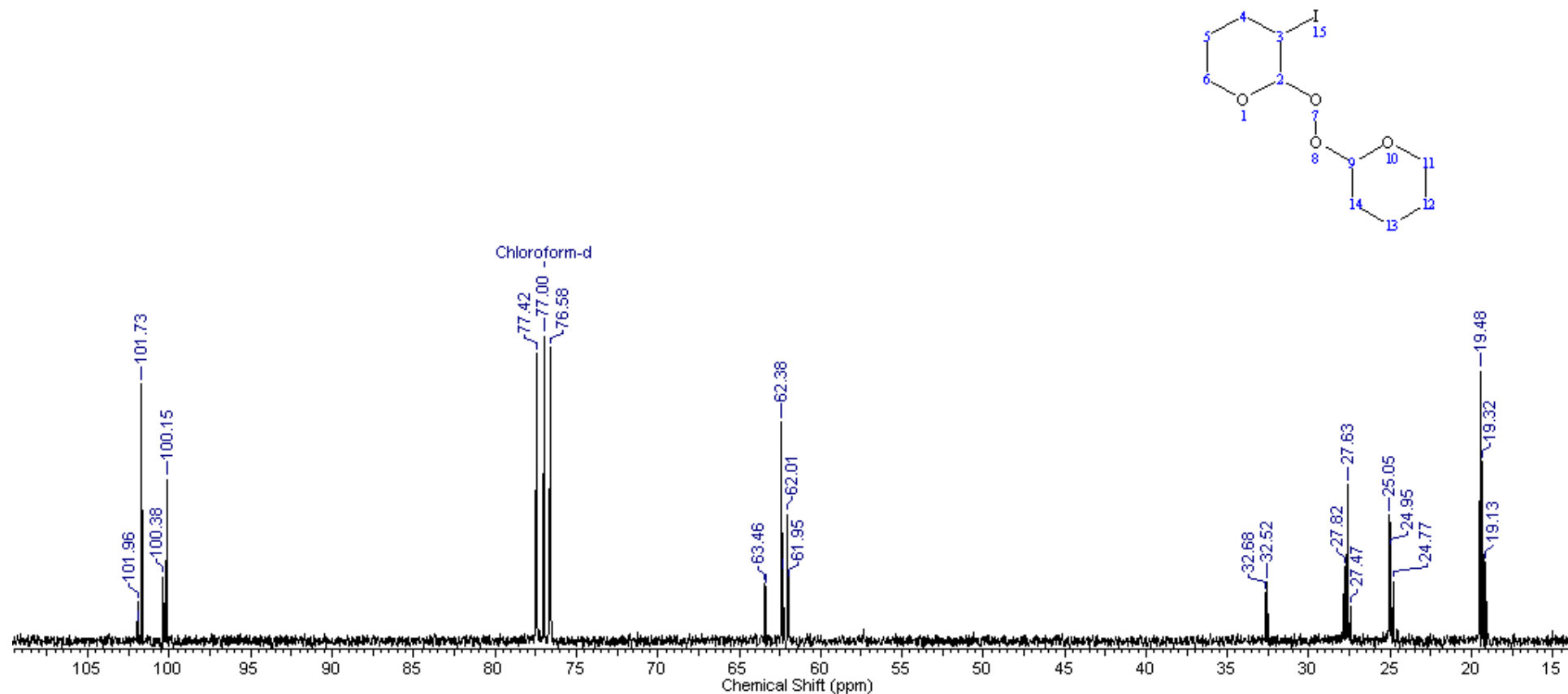
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Points Count	8192	Pulse Sequence	zg	Solvent	CHLOROFORM-D
Temperature (degree C)	27.200			Original Points Count	8124
				Sweep Width (Hz)	6009.62



¹³C NMR 3-Iodo-2-(tetrahydro-2*H*-pyran-2-ylperoxy)tetrahydro-2*H*-pyran (6b)

4 Jan 2014

Acquisition Time (sec)	0.9002	Comment	Avance-300, C-13, CDCl ₃	Date	12 Nov 2012 16:27:44
File Name					
Frequency (MHz)	75.48	Nucleus	13C	Number of Transients	321
Original Points Count	16308	Points Count	16384	Pulse Sequence	zgpg30base
Solvent	CHLOROFORM-D	Sweep Width (Hz)	18115.94	Temperature (degree C)	28.000



HRMS 3-Iodo-2-(tetrahydro-2H-pyran-2-ylperoxy)tetrahydro-2H-pyran (6b)

Display Report

Analysis Info

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Method tune_wide.m
Sample Name MeCN&zd-152
Comment Sample name: zd-152
Sample owner: Zdvigkov
Solvent: MeCN
Conc.: 0.18 ng/ml

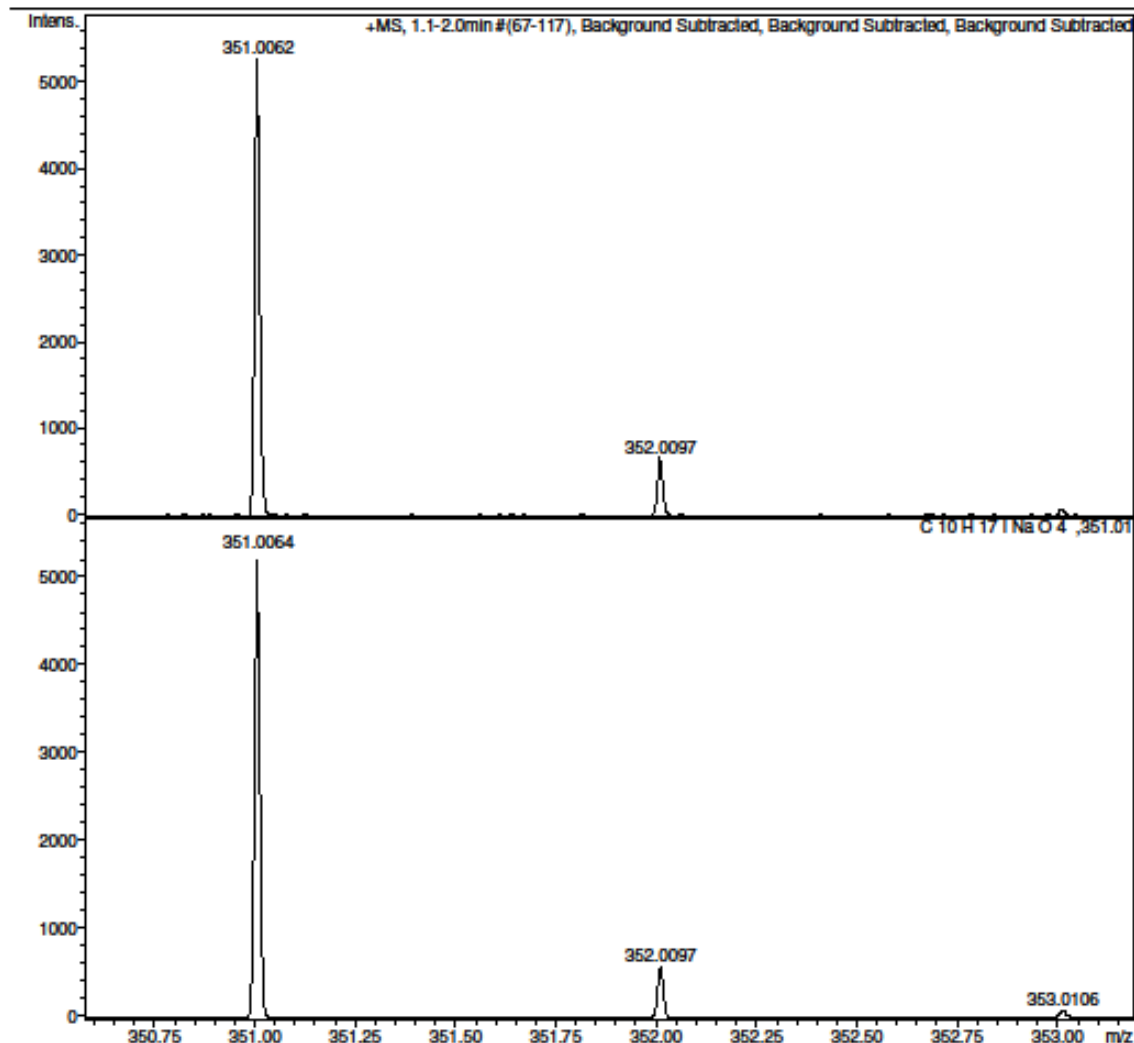
Acquisition Date 11/23/2012 11:48:35 AM

Operator BDAL@DE

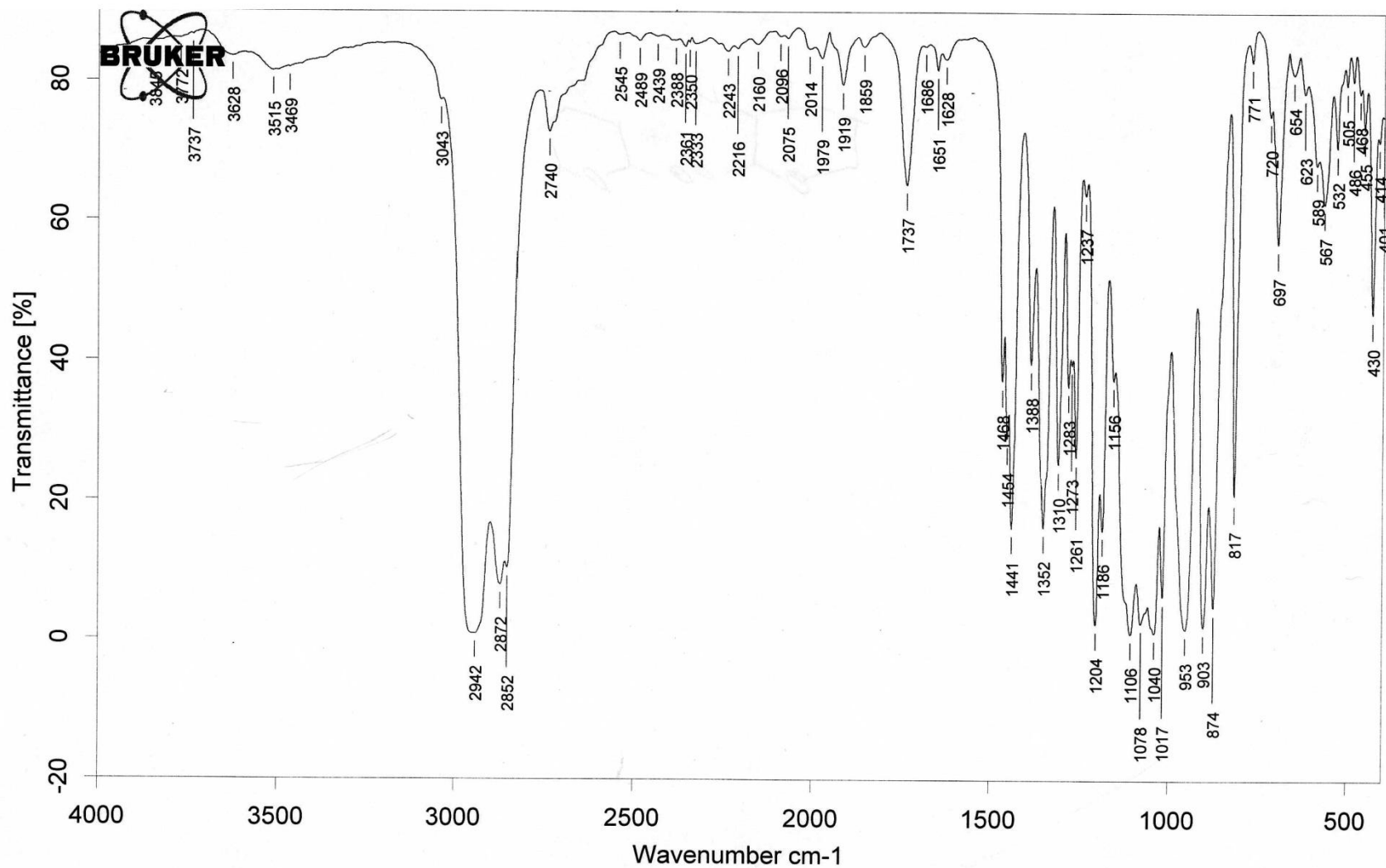
Instrument / Ser# maXis 43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
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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



IR 3-Iodo-2-(tetrahydro-2*H*-pyran-2-ylperoxy)tetrahydro-2*H*-pyran (6b)



C:\Data\EDL\ZD-152.0

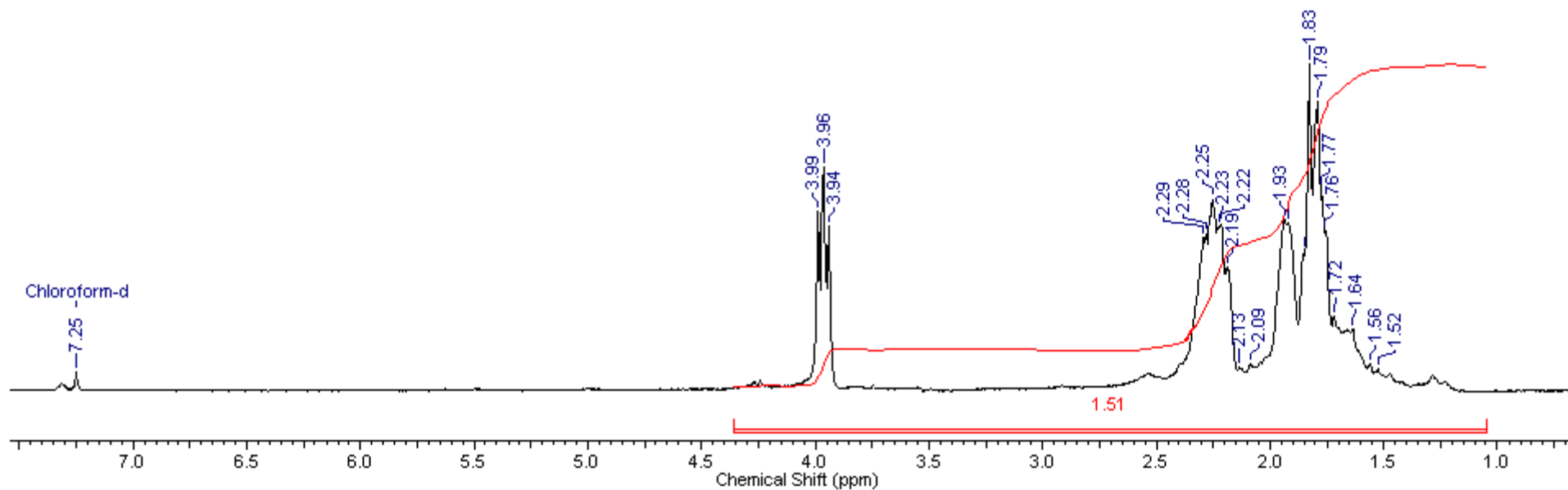
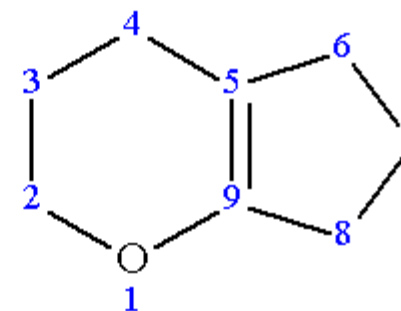
ZDVIZHKOV. ZD-152, thin layer on KBr.

11/12/2012

¹H NMR 2,3,4,5,6,7-Hexahydrocyclopenta[b]pyran (10a)

23 Jul 2013

Acquisition Time (sec)	1.4418	Comment	/TERN_ARZDV576.001 □Opr:Gvozdev V.D.;Solv:CDCl3;		Date	22/07/2013 21:06:35	
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Number of Transients	8	Original Points Count	4096	Points Count	8192	Solvent	CHLOROFORM-D
Sweep Width (Hz)	2840.91	Temperature (degree C)	24.000				

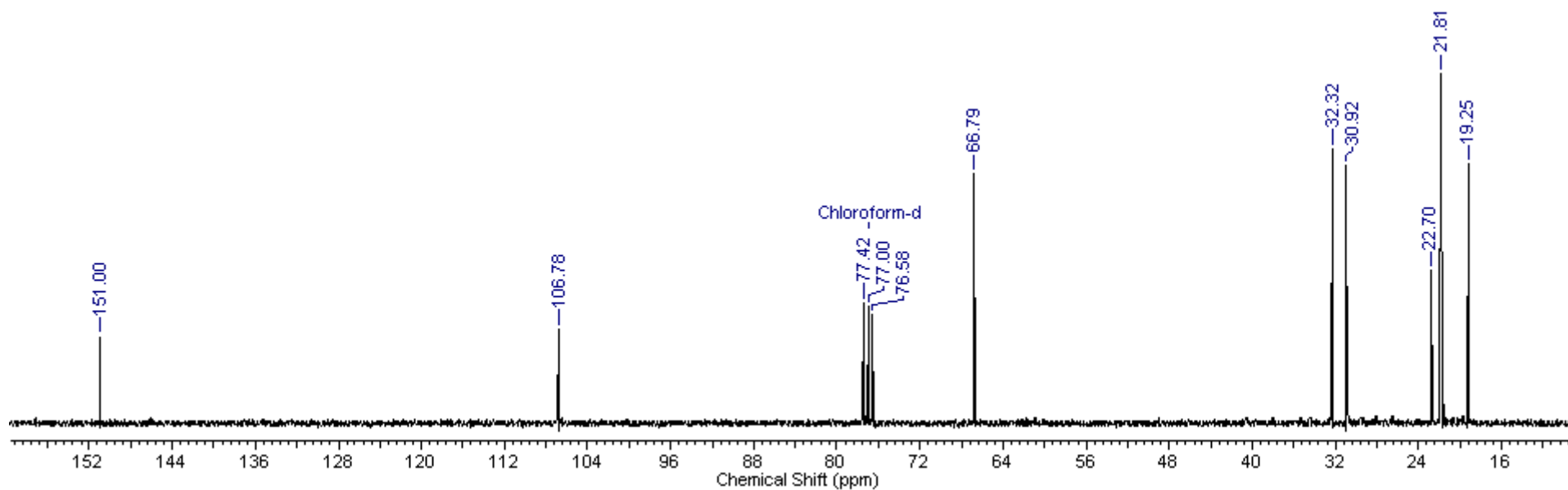
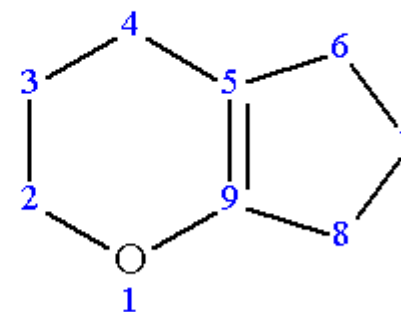


No.	Annotation	(ppm)	No.	(ppm)	Value	Absolute Value
1	Chloroform-d	7.25	1	[1.04 .. 4.36]	1.506	4.75305e+8

¹³C NMR 2,3,4,5,6,7-Hexahydrocyclopenta[*b*]pyran (10a)

25 Jul 2013

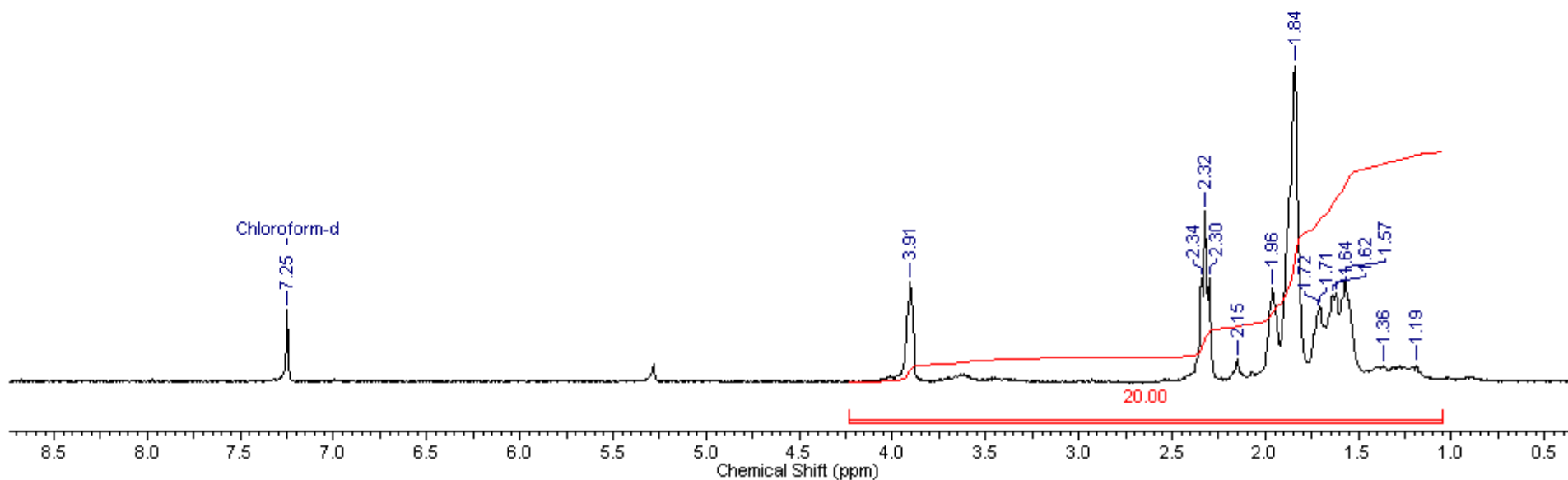
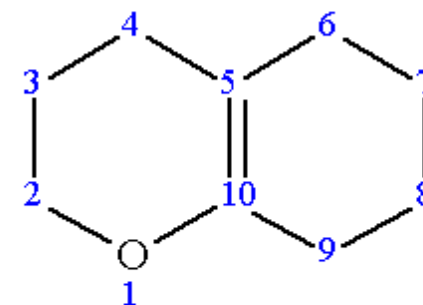
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¹H NMR 3,4,5,6,7,8-Hexahydro-2H-chromene (10b)

26 Sep 2013

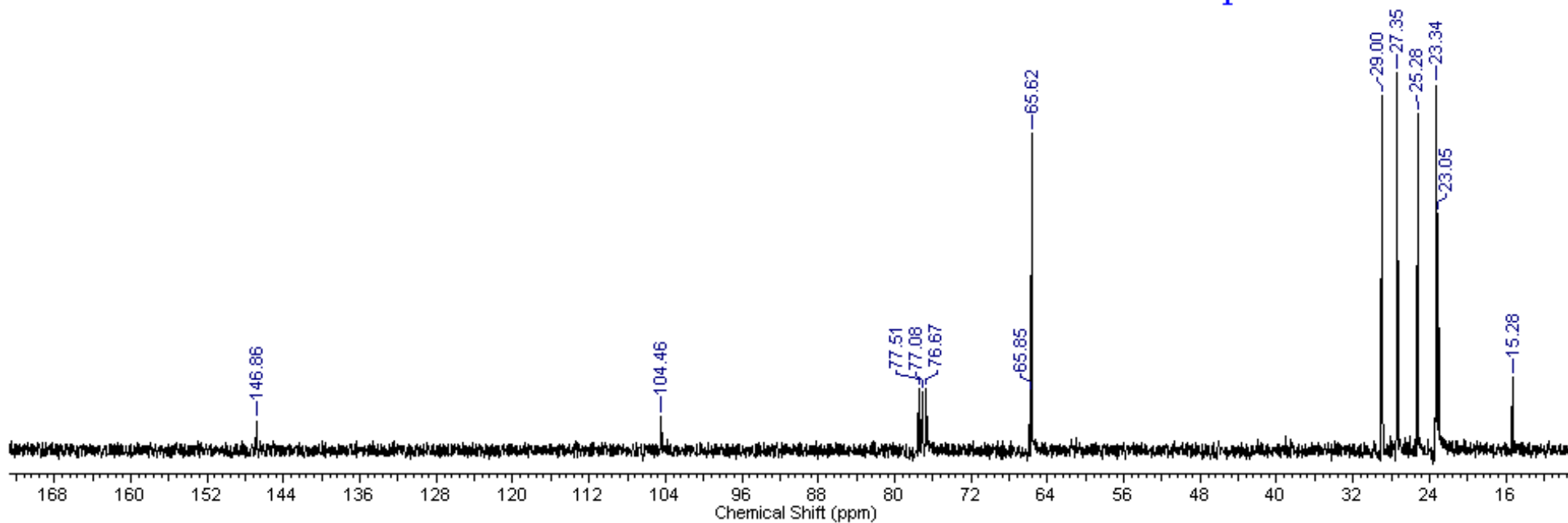
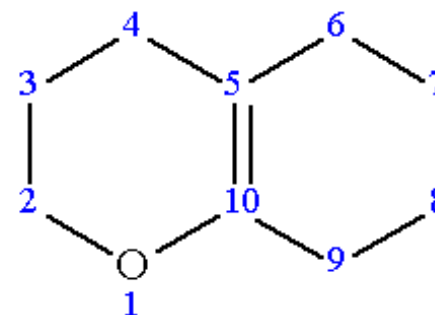
08 Dec 2012 08:12:43	Avance-300, 1H, CDCl ₃		1.351
300.1			File Name
819	8124	Number of Transients	1
6009.6	CHLOROFORM-d		Pulse Sequence
			27.800



¹³C NMR 3,4,5,6,7,8-Hexahydro-2H-chromene (10b)

26 Sep 2013

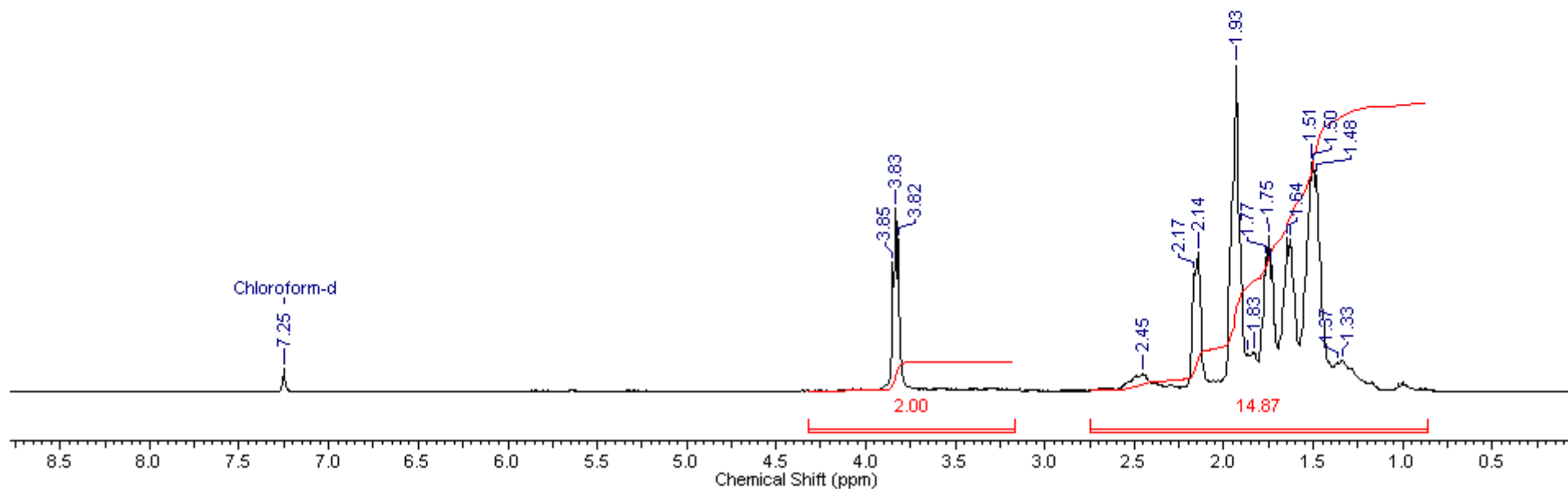
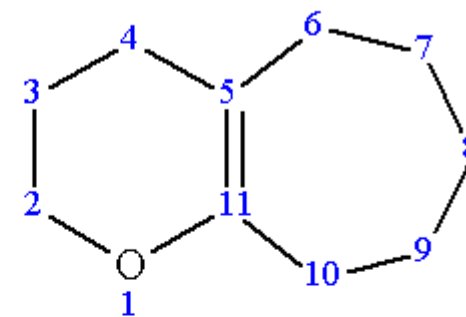
	08 Dec 2012 12:41:36	Date	NMR/5007524	Comment	0.432	Acquisition Time (sec)
75.48	Frequency (MHz)					File Name
16384	Points Count	16384	Original Points Count	240	Number of Transients	130
28.30	Temperature (degree C)	18867.9	Sweep Width (Hz)	CHLOROFORM-D	Solvent	zpgpg60bas
						Pulse Sequence



¹H NMR 2,3,4,5,6,7,8,9-Octahydrocyclohepta[b]pyran (10c)

26 Sep 2013

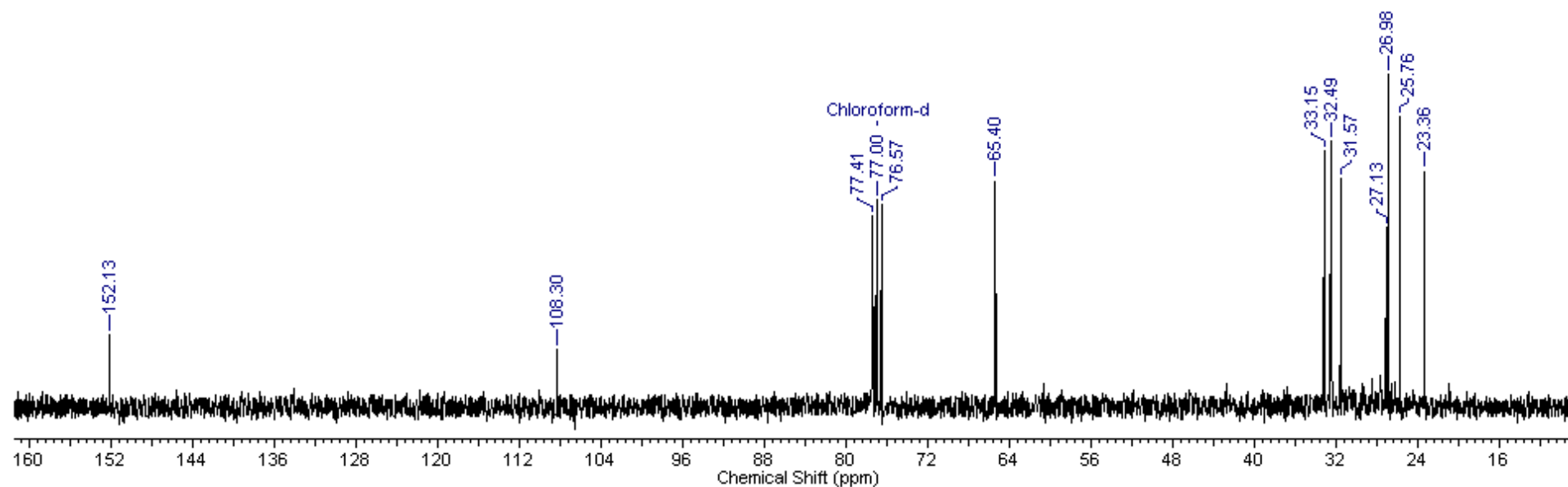
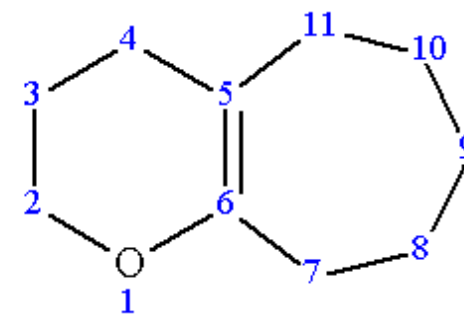
Acquisition Time (sec)	0.6759	Comment	Avance-300, 1H CDCl3	Date	16 Nov 2012 08:17:04
File Name				Frequency (MHz)	300.13
Nucleus	1H	Number of Transients	1	Original Points Count	8124
Pulse Sequence	zg	Solvent	CHLOROFORM-D	Sweep Width (Hz)	6009.62
				Points Count	8192
				Temperature (degree C)	26.800



¹³C NMR 2,3,4,5,6,7,8,9-Octahydrocyclohepta[*b*]pyran (10c)

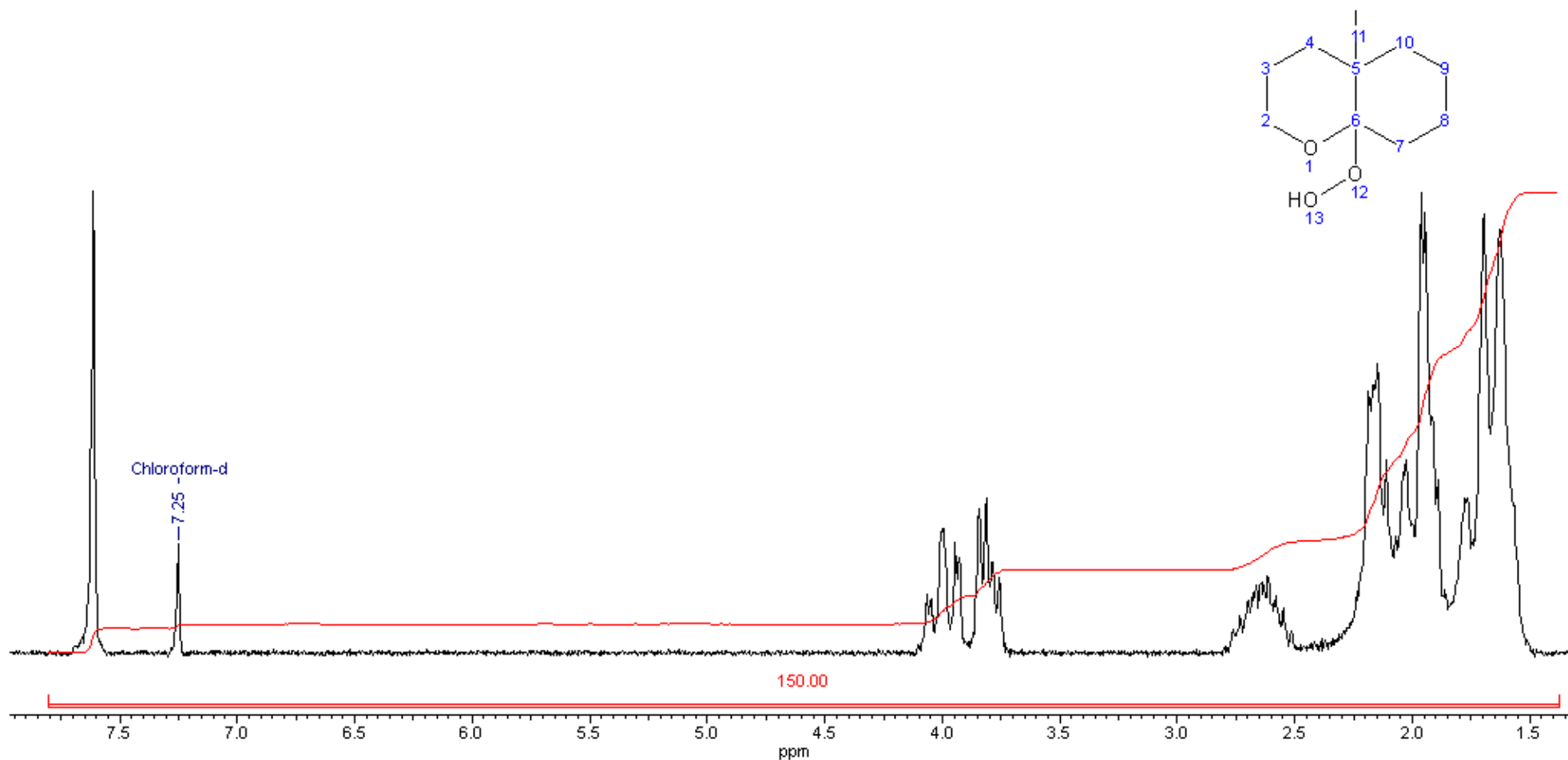
23 Jul 2013

Acquisition Time (sec) 0.8643		Date 17 Nov 2012 09:38:08				
File Name					Frequency (MHz)	75.48
Nucleus 13C		Number of Transients 216		Original Points Count 16308	Points Count	16384
Pulse Sequence zgpg30base		Solvent CHLOROFORM-D		Sweep Width (Hz) 18867.92	Temperature (degree C) 27.700	



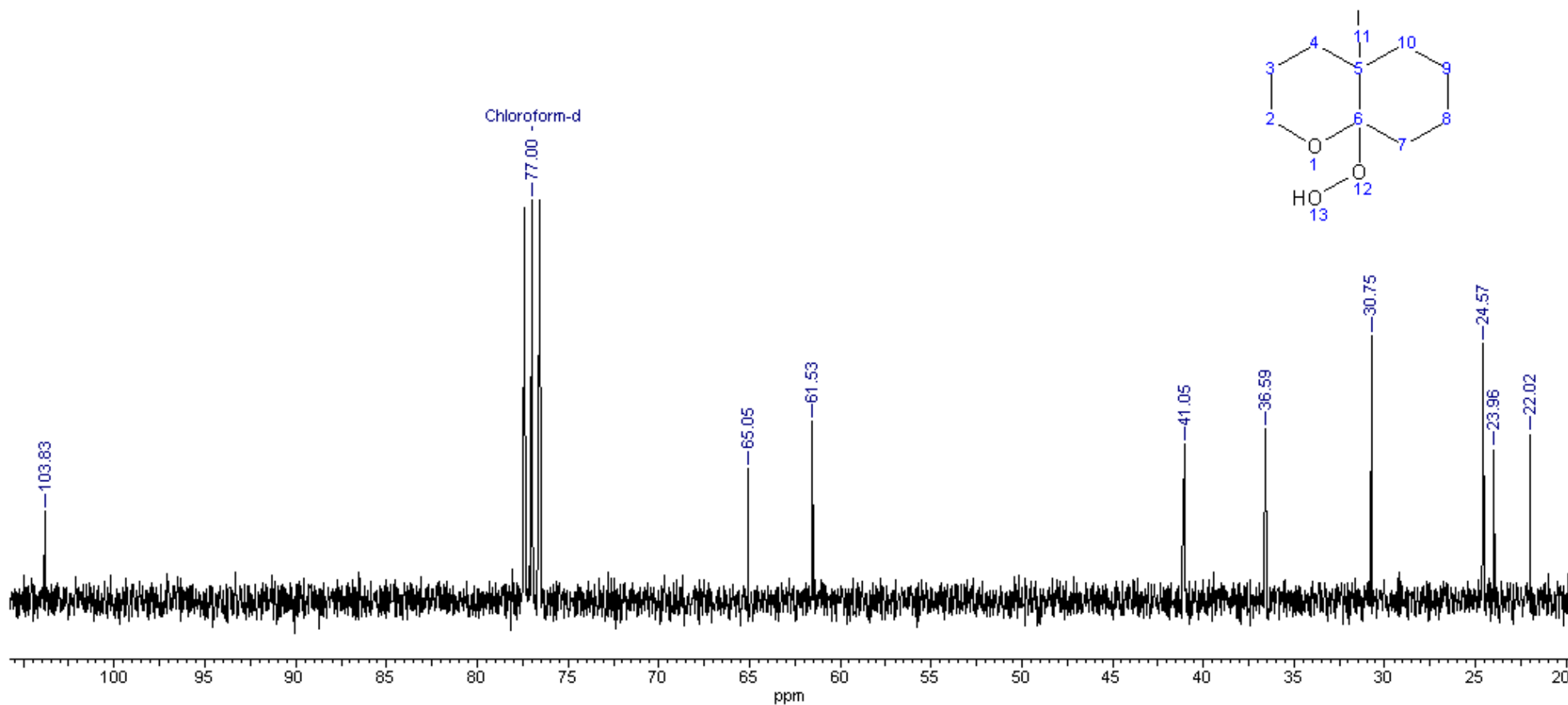
¹H NMR 4a-Iodoctahydrocyclohepta[b]pyran-9a(2H)-yl hydroperoxide (11b)

Acquisition Time (sec)	1.0240	Comment		Date	25/03/2008 15:16:27
File Name					
Frequency (MHz)	200.13	Nucleus	1H	Number of Transients	12
Points Count	8192	Solvent	CHLOROFORM-D	Original Points Count	8192
				Sweep Width (Hz)	4000.00
				Temperature (degree C)	24.000



¹³C NMR 4a-Iodoctahydrocyclohepta[b]pyran-9a(2H)-yl hydroperoxide (11b)

Acquisition Time (sec)	0.4501	Comment	Avance-300, cdcl3;tem	Date	25 Mar 2008 15:23:44
File Name					
Frequency (MHz)	75.48	Nucleus	13C	Number of Transients	267
Original Points Count	16308	Points Count	16384	Pulse Sequence	zgpg30base
Solvent	CHLOROFORM-D	Sweep Width (Hz)	18115.94	Temperature (degree C)	21.200



HRMS 4a-Iodoctahydrocyclohepta[b]pyran-9a(2H)-yl hydroperoxide (11b)

Display Report

Analysis Info

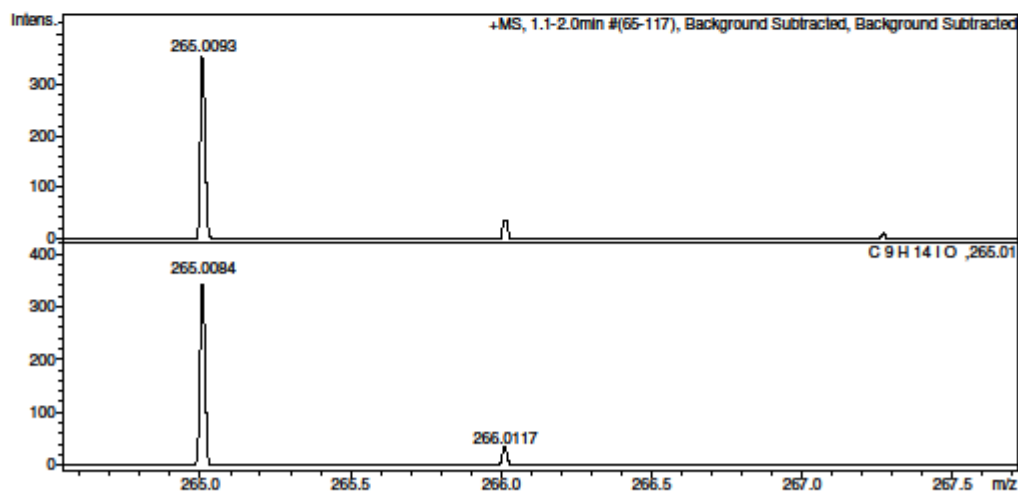
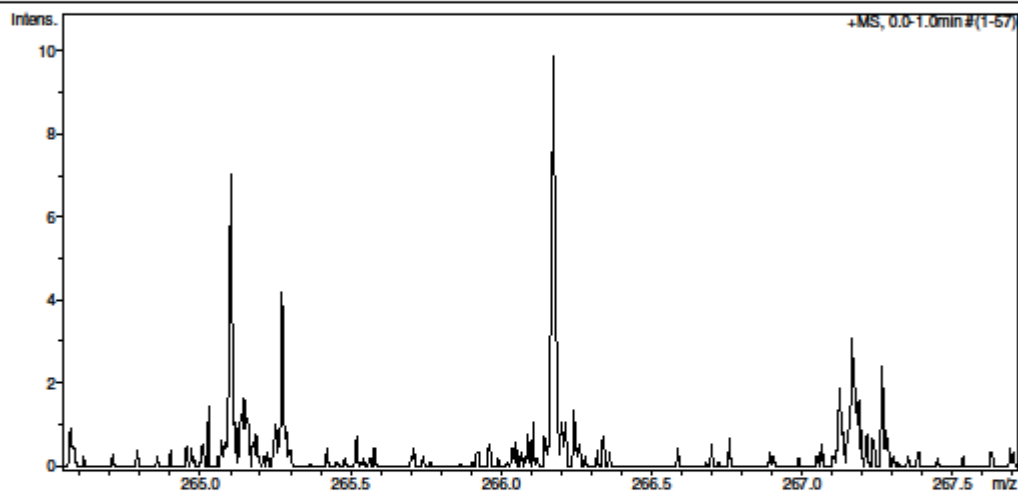
Analysis Name D:\Data\chemchyan\daily\MeCN&zd-96_1_low.d
Method tune_low.m
Sample Name MeCN&zd-96_1_low
Comment Samplename: zd-96_1
Solvent: MeCN

Acquisition Date 10/5/2012 7:12:12 PM

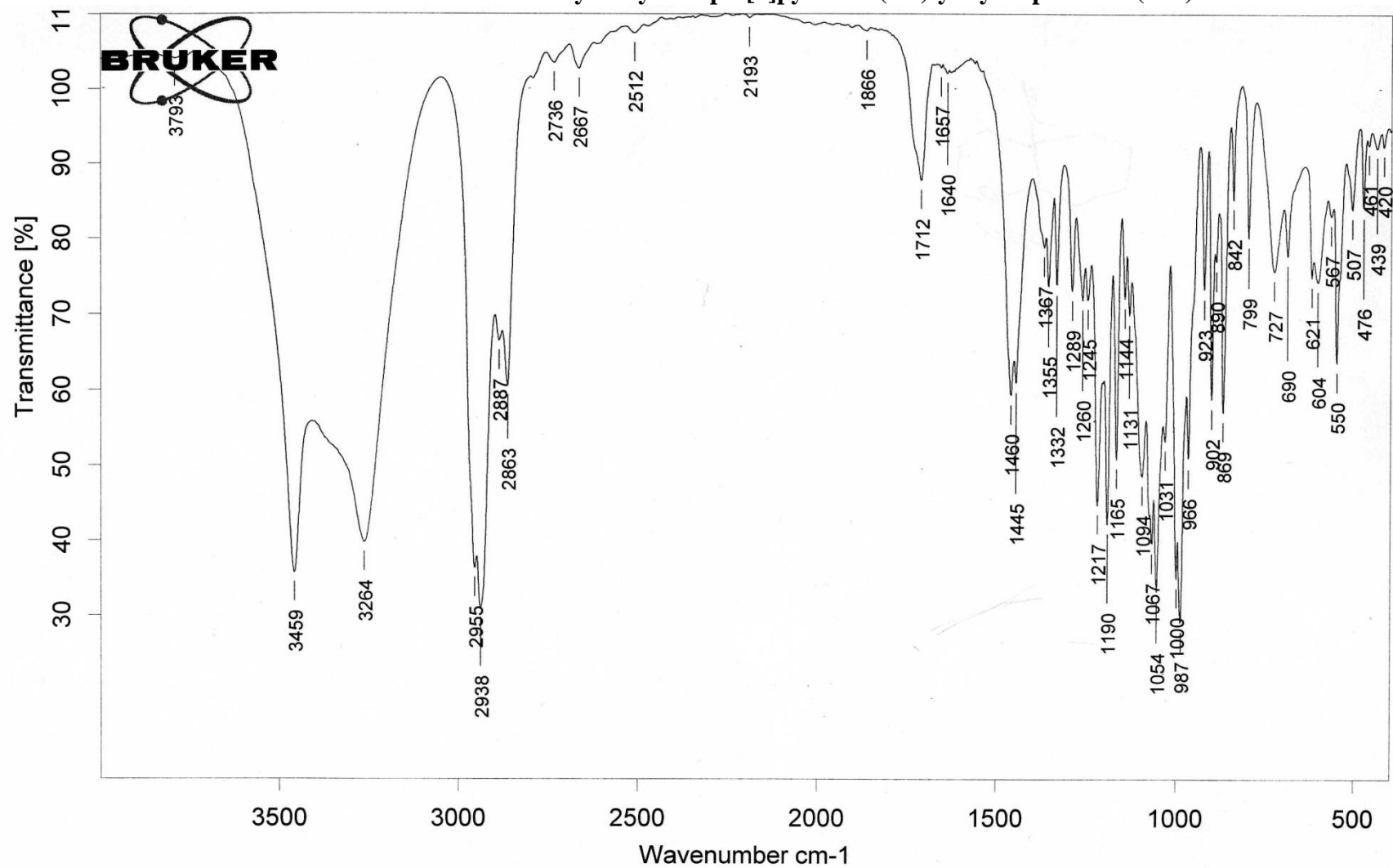
Operator BDAL@DE
Instrument maXis 43

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



IR 4a-Iodoctahydrocyclohepta[*b*]pyran-9a(2*H*)-yl hydroperoxide (11b)



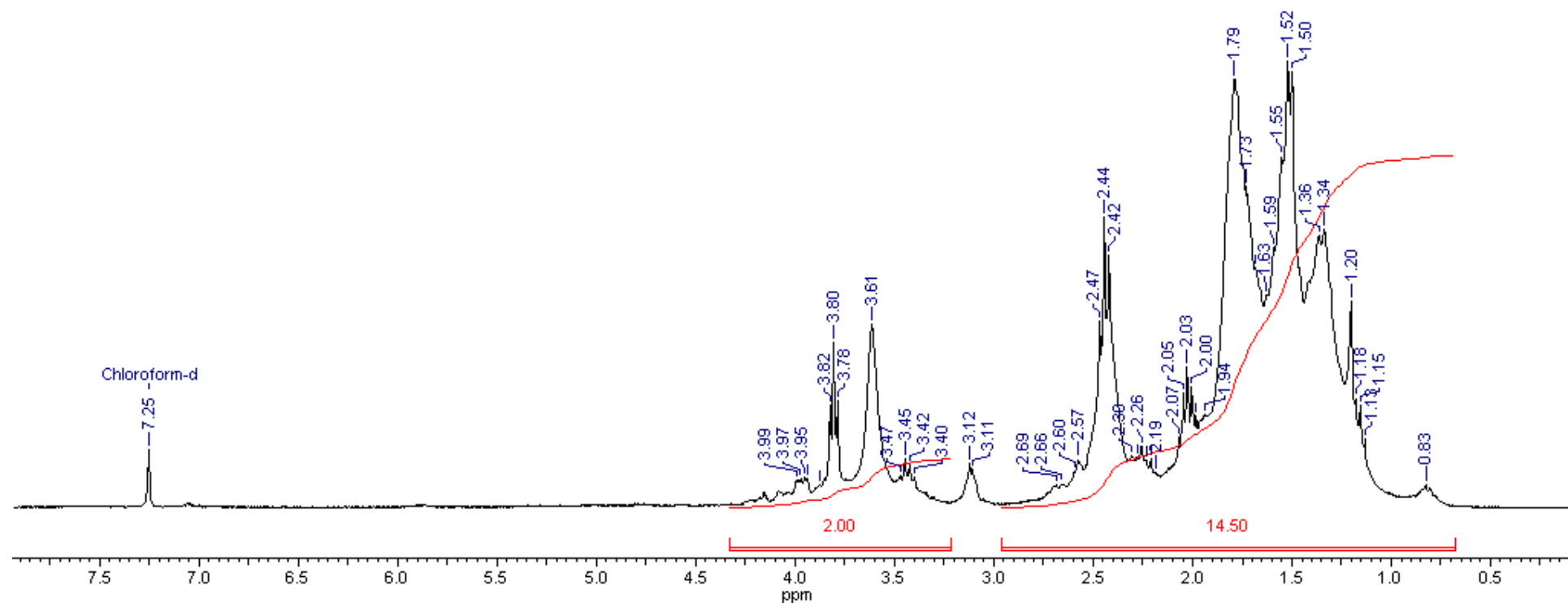
D:\EDL\Zd-96.0 ЗДВИЖКОВ. Zd-96, прессовка KBr, 1/200.

19.02.2013

¹H NMR 4a-Iodoctahydrocyclohepta[b]pyran-9a(2H)-yl hydroperoxide (11c)

4 Jul 2013

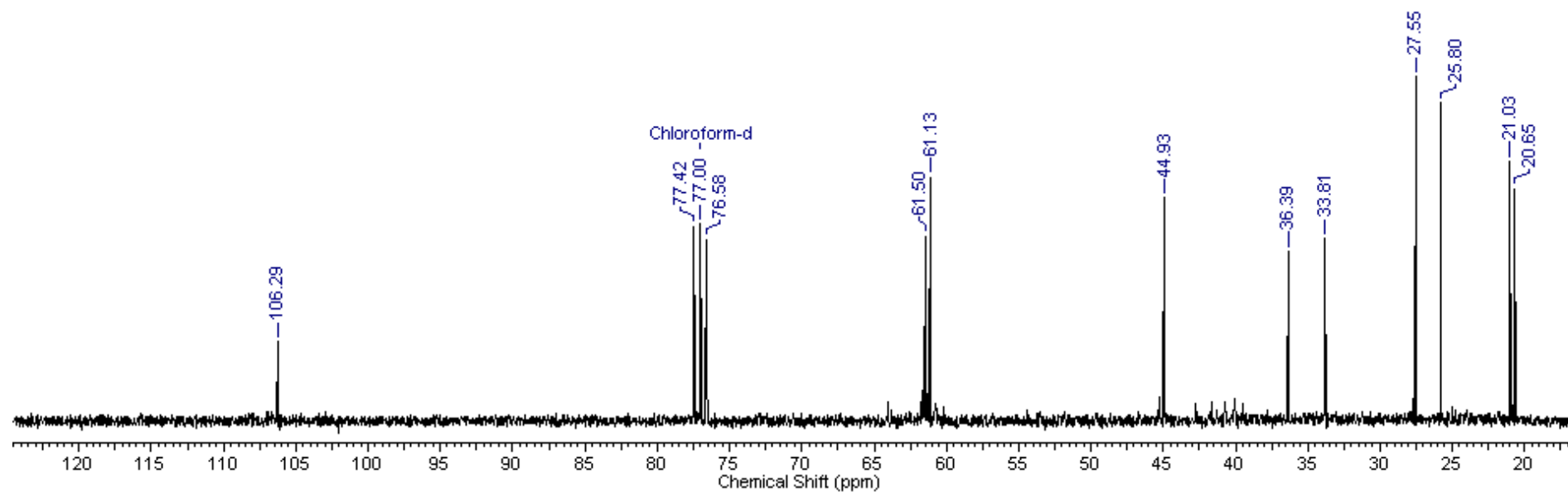
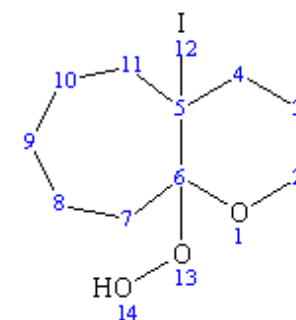
Acquisition Time (sec)	0.6759	Comment	/TERN ARZDV114	Date	22 Nov 2012 13:45:36
File Name				Frequency (MHz)	300.13
Nucleus	1H	Number of Transients	1	Original Points Count	8124
				Points Count	8192
Pulse Sequence	zg	Solvent	CHLOROFORM-D	Sweep Width (Hz)	6009.62
				Temperature (degree C)	27.000



¹³C NMR 4a-Iodoctahydrocyclohepta[b]pyran-9a(2H)-yl hydroperoxide (11c)

5 Nov 2013

Acquisition Time (sec)	0.9002	Comment	Avance-300, C-13, DMSO-d6	Date	
File Name				Frequency (MHz)	75.48
Nucleus	13C	Number of Transients	405	Original Points Count	16308
Pulse Sequence	zgpg baseopt	Solvent	CHLOROFORM-D	Sweep Width (Hz)	18115.94
				Points Count	16384
				Temperature (degree C)	30.000



HRMS 4a-Iodoctahydrocyclohepta[b]pyran-9a(2H)-yl hydroperoxide (11c)

Display Report

Analysis Info

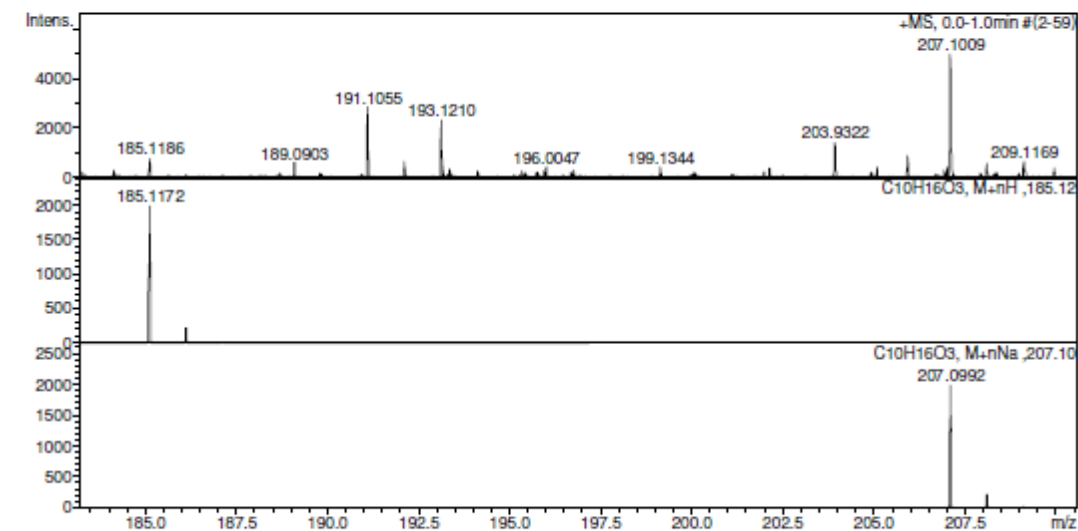
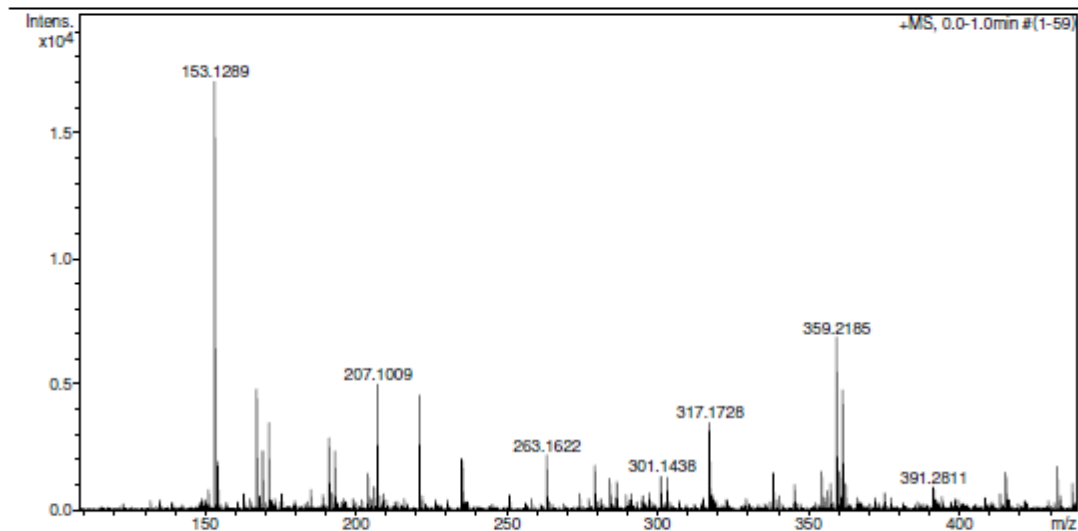
Analysis Name D:\Data\Ivanova\ZD-363low.d
Method tune_low.m
Sample Name /TERN ZD-363low
Comment CH3CN 100 %, dil. 1000, calibrant added

Acquisition Date 01.11.2013 19:32:55

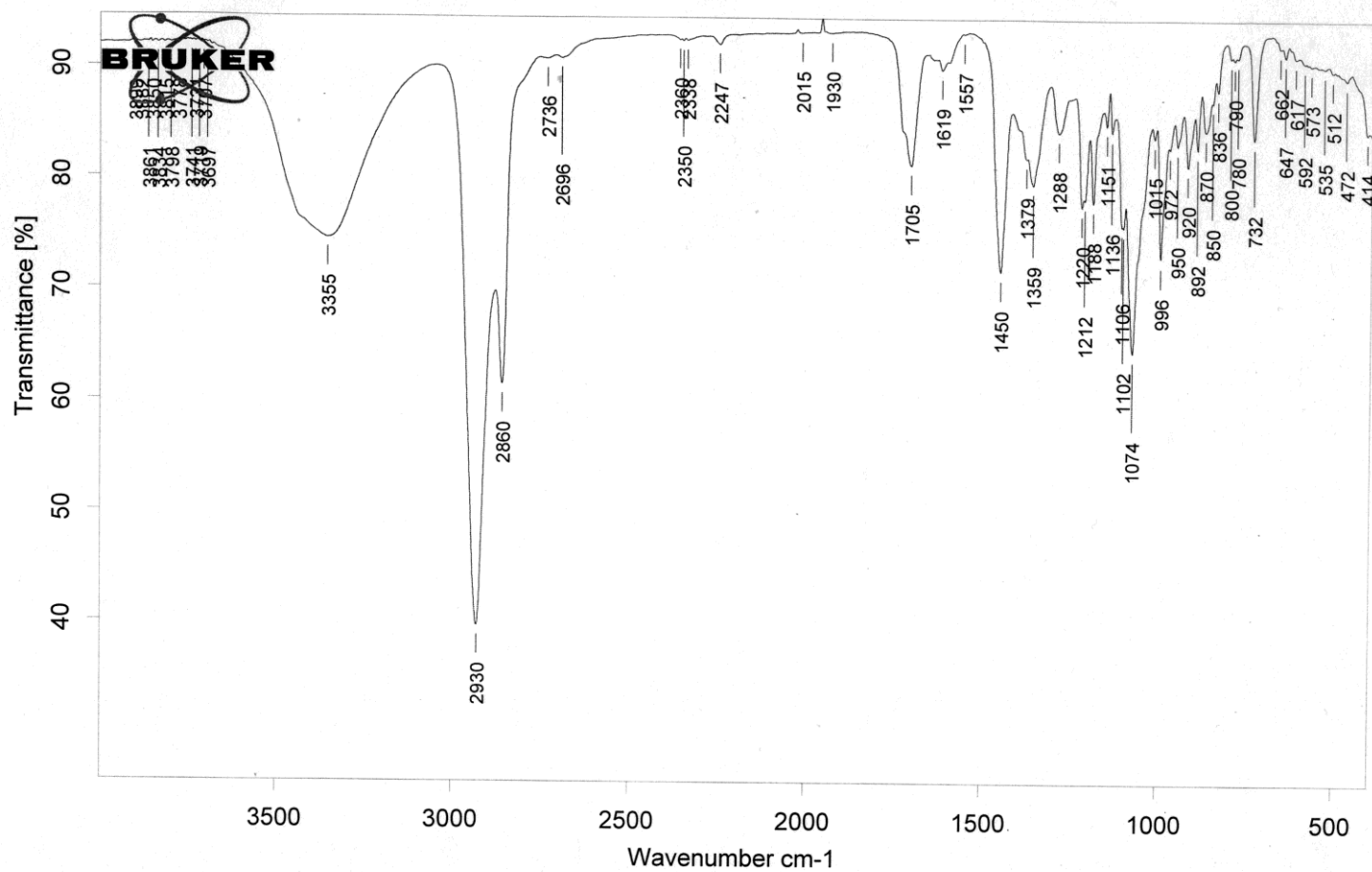
Operator BDAL@DE
Instrument / Ser# microTOF 10248

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



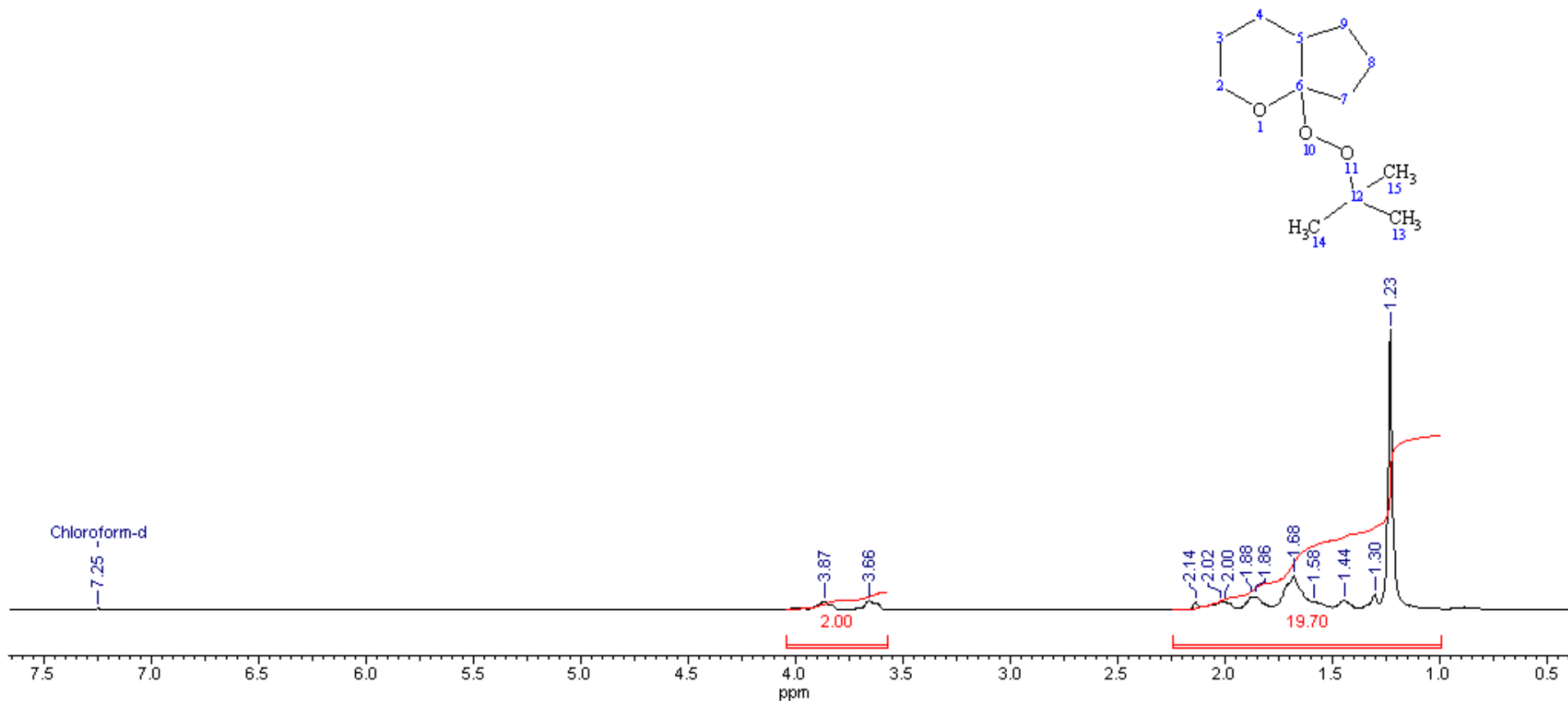
IR 4a-Iodoctahydrocyclohepta[*b*]pyran-9a(2*H*)-yl hydroperoxide (11c)



¹H NMR 7a-(*tert*-Butylperoxy)octahydrocyclopenta[*b*]pyran (12a)

2 Jul 2013

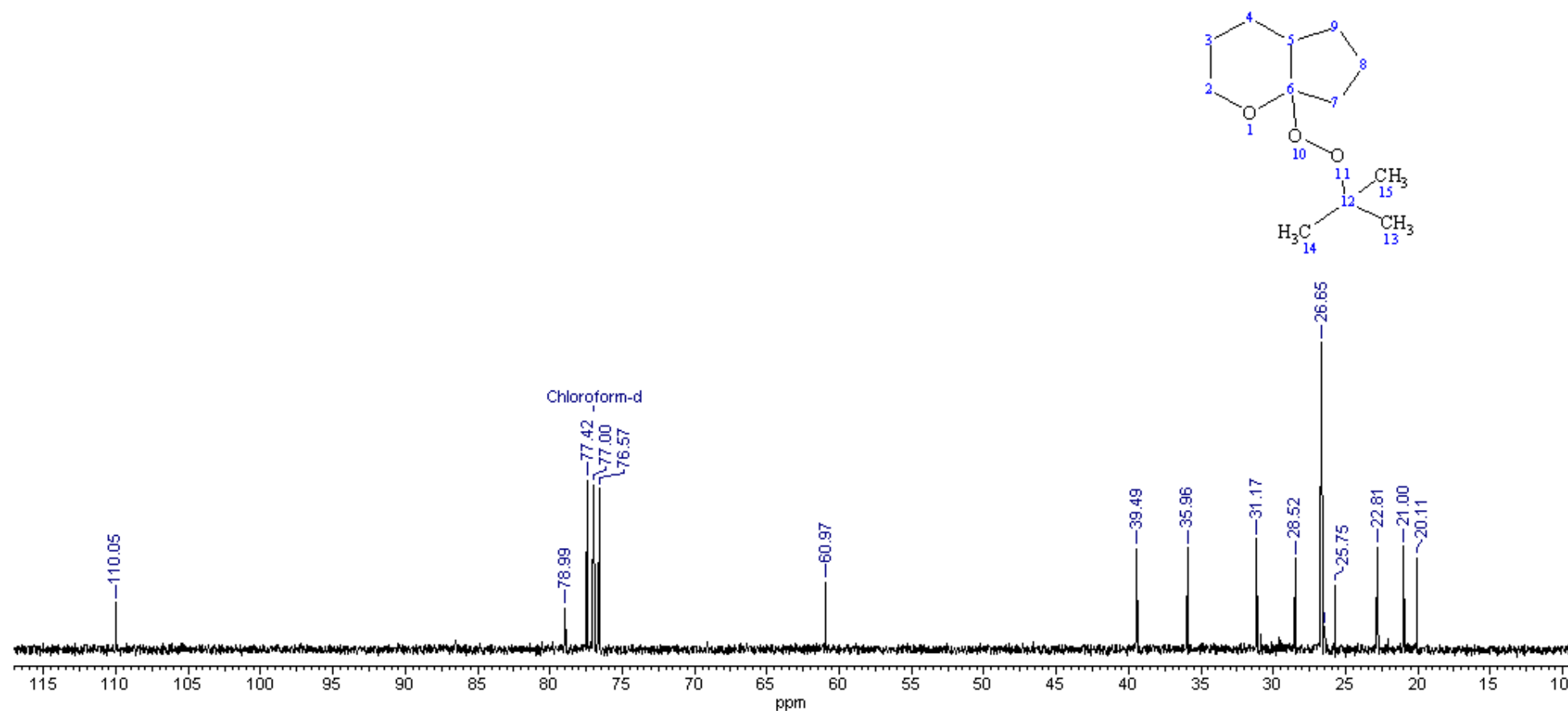
Acquisition Time (sec)	0.6759	Comment	NMR/50075654	Date	07 Nov 2012 09:29:36
File Name					
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	1
Points Count	8192	Pulse Sequence	zg	Original Points Count	8124
Sweep Width (Hz)	6009.62	Temperature (degree C)	27.300	Solvent	CHLOROFORM-D



¹³C NMR 7a-(*tert*-Butylperoxy)octahydrocyclopenta[*b*]pyran (12a)

2 Jul 2013

Acquisition Time (sec)	0.4501	Comment	Avance-300, C-13, CDCl ₃	Date	07 Nov 2012 09:33:52
File Name				Frequency (MHz)	75.48
Nucleus	13C	Number of Transients	481	Original Points Count	16308
Pulse Sequence	zgpg30base	Solvent	CHLOROFORM-D	Sweep Width (Hz)	18115.94
				Points Count	16384
				Temperature (degree C)	27.500



HRMS 7a-(*tert*-Butylperoxy)octahydrocyclopenta[*b*]pyran (12a)

Display Report

Analysis Info

Analysis Name D:\Data\khemchyan\MS service @ IOC\zdvigkov\14_11_2012\MeCN&zd-147_low.d
Method tune_low.m
Sample Name MeCN&zd-147_low
Comment Sample name: zd-147
Sample owner: Zdvigkov A
Solvent: MeCN
Dil : 500 times

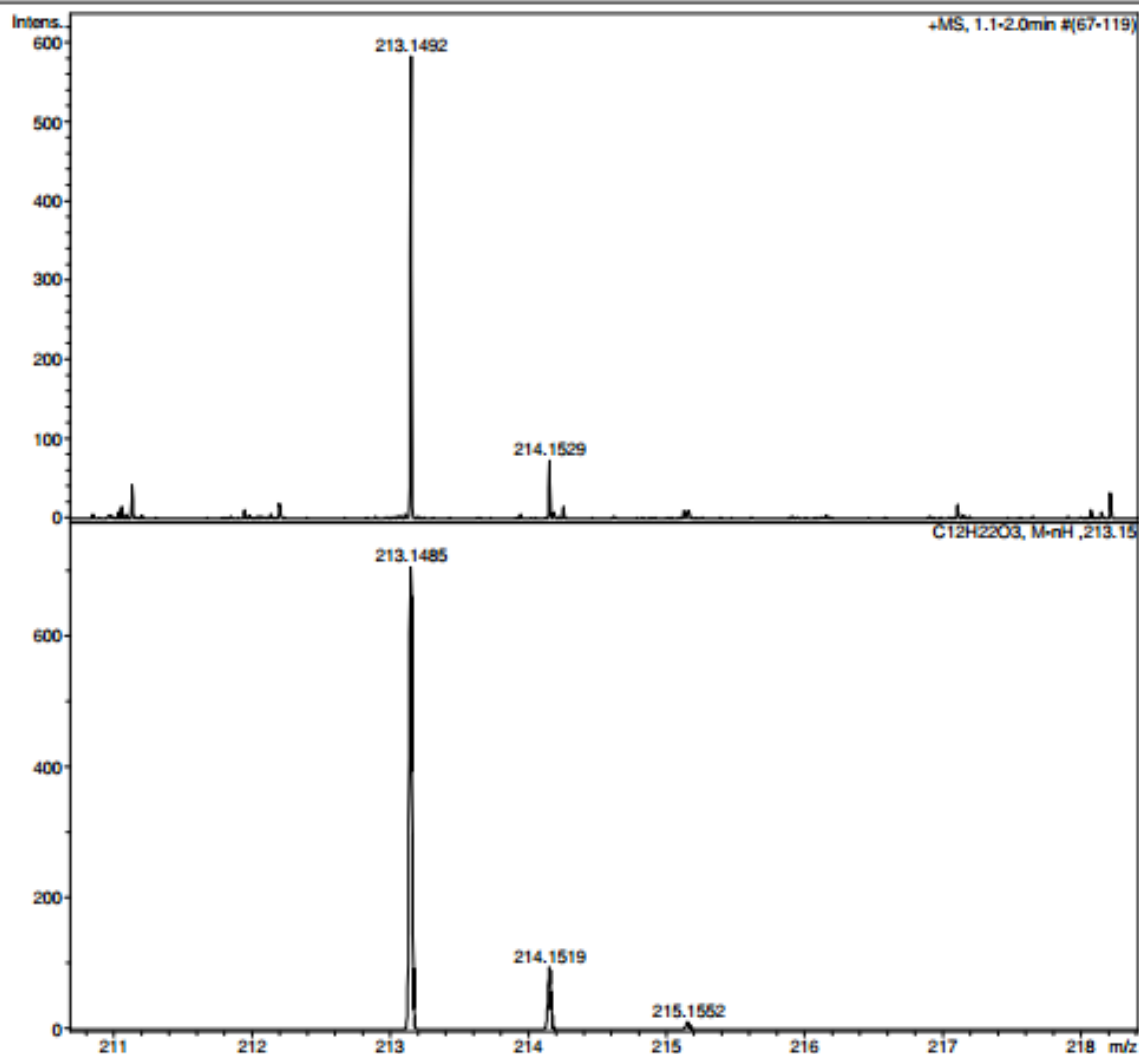
Acquisition Date 14.11.2012 18:45:29

Operator BDAL@DE

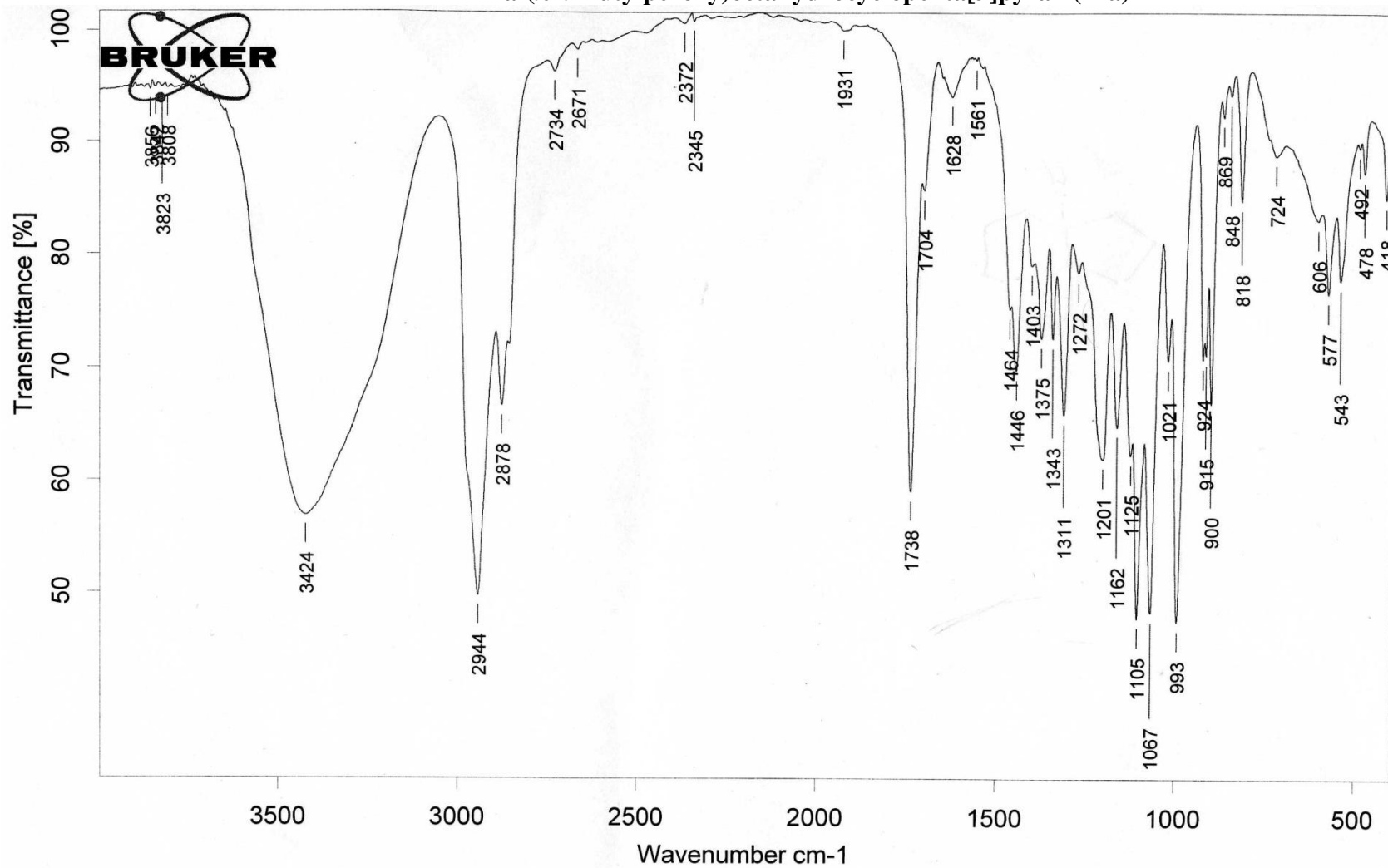
Instrument maXis 43

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



IR 7a-(*tert*-Butylperoxy)octahydrocyclopenta[*b*]pyran (12a)



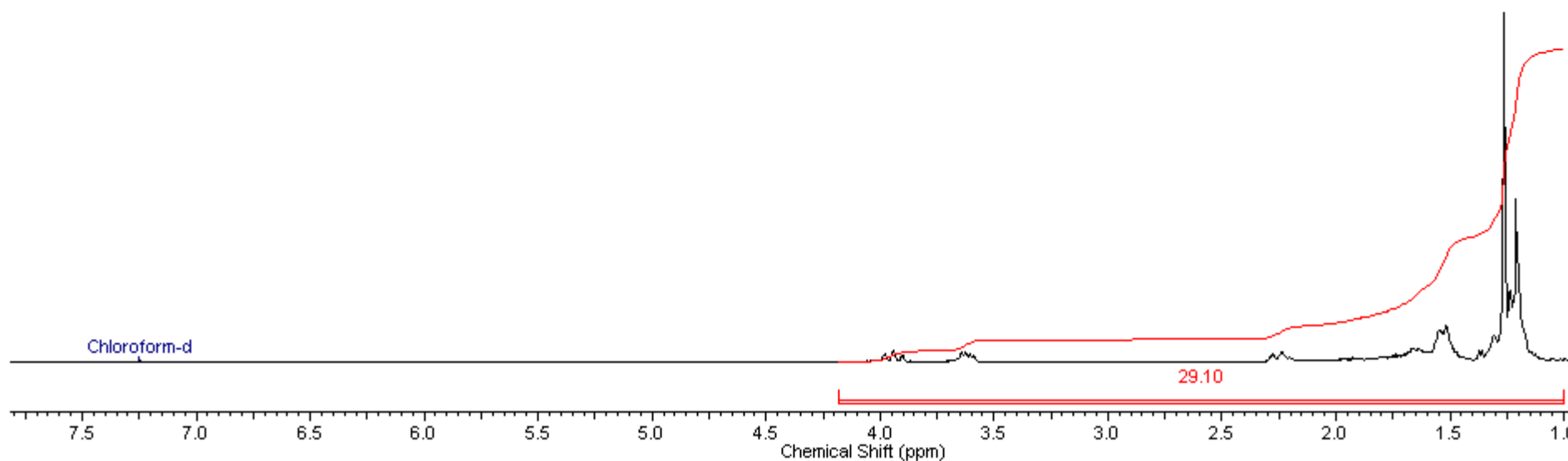
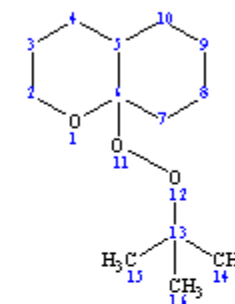
D:\EDL\Zd-170.0 ЗДВИЖКОВ. Zd-170 , прессовка KBr, 1/200.

19.02.2013

¹H NMR 8a-(*tert*-Butylperoxy)octahydro-2*H*-chromene (12b)

26 Sep 2013

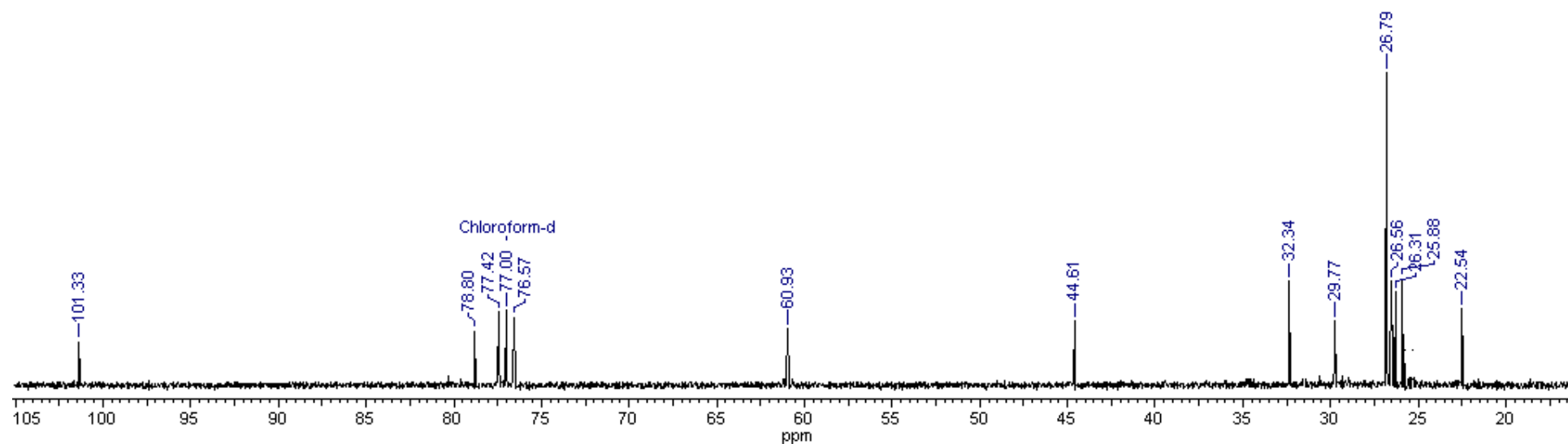
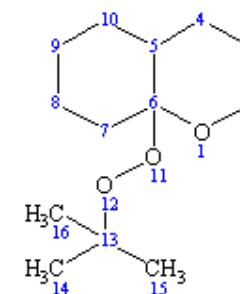
Acquisition Time (sec)	0.6759	Comment	MGOR NH 0051	Date	11 Oct 2012 12:50:08
File Name					
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	1
Points Count	8192	Pulse Sequence	zgq	Solvent	CHLOROFORM-D
Temperature (degree C)	27.200			Sweep Width (Hz)	6009.62



¹³C NMR 8a-(*tert*-Butylperoxy)octahydro-2*H*-chromene (12b)

2 Jul 2013

Acquisition Time (sec)	0.4501	Comment	Avance-300, C-13, CDCl3		Date	11 Oct 2012 12:50:08	
File Name					Frequency (MHz)	75.48	
Nucleus	13C	Number of Transients	99	Original Points Count	16308	Points Count	16384
Pulse Sequence	zgpg30base	Solvent	CHLOROFORM-D	Sweep Width (Hz)	18115.94	Temperature (degree C)	27.400



HRMS 8a-(*tert*-Butylperoxy)octahydro-2*H*-chromene (12b)

Display Report

Analysis Info

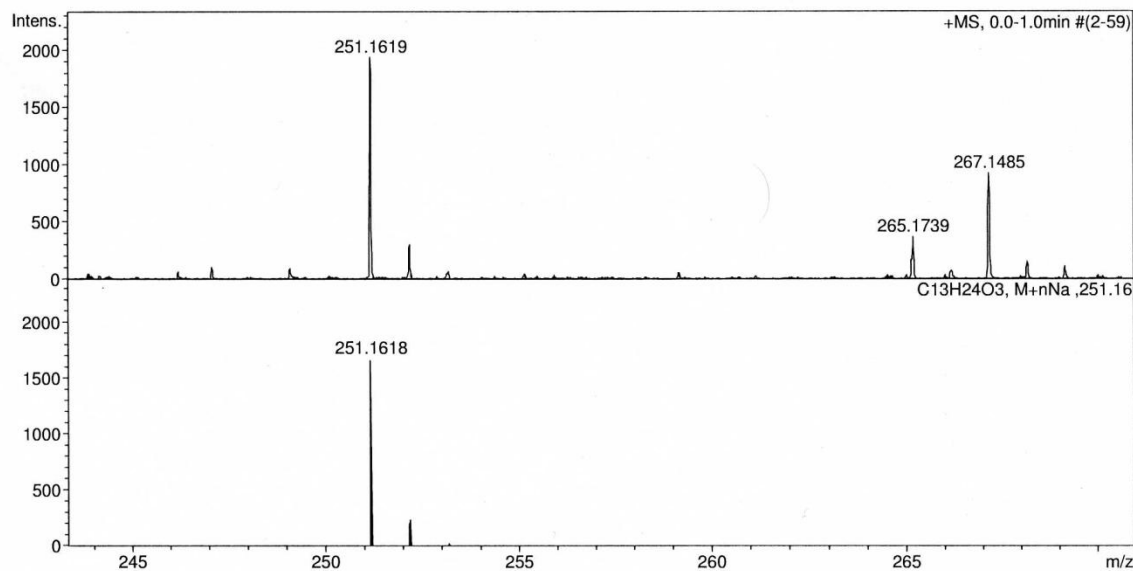
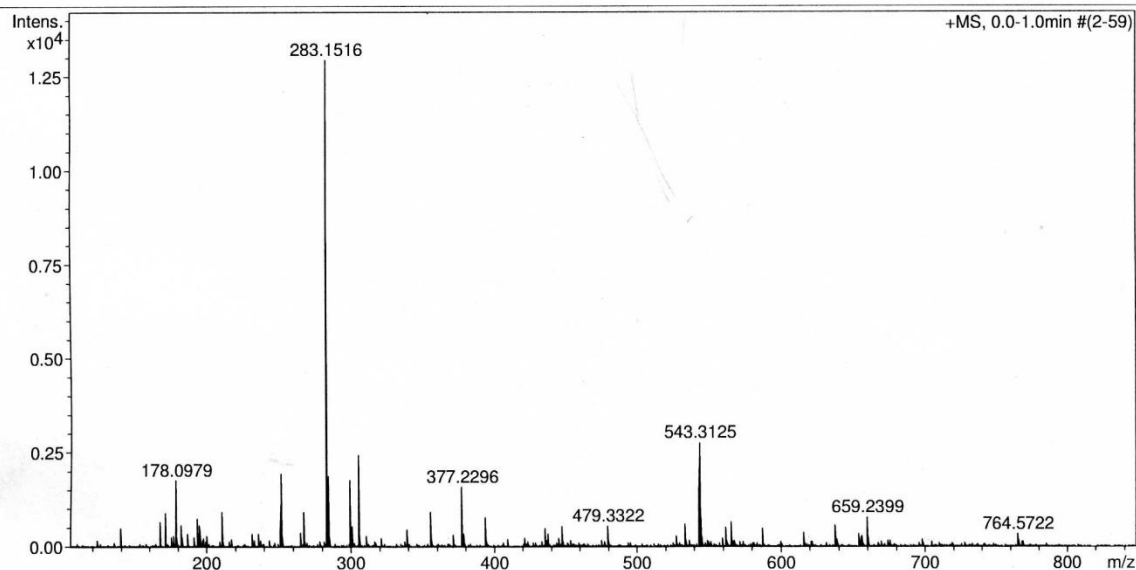
Analysis Name D:\Data\Ivanova\ZD-192.d
Method tune_low.m
Sample Name /TERN ZD-192
Comment CH3CN 100%, dil 1000

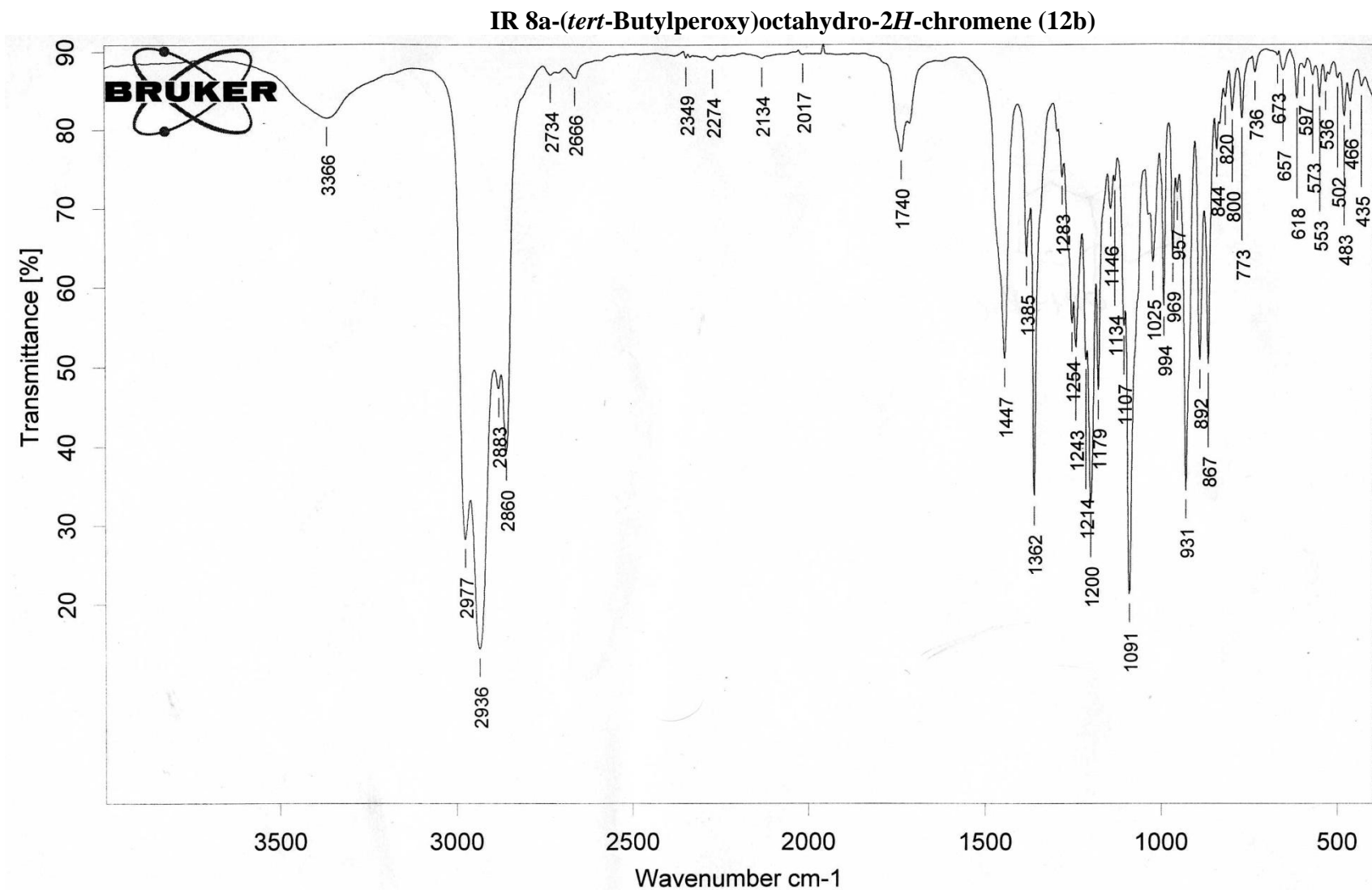
Acquisition Date 17-Jan-13 17:38:17

Operator BDAL@DE
Instrument / Ser# micrOTOF 10248

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





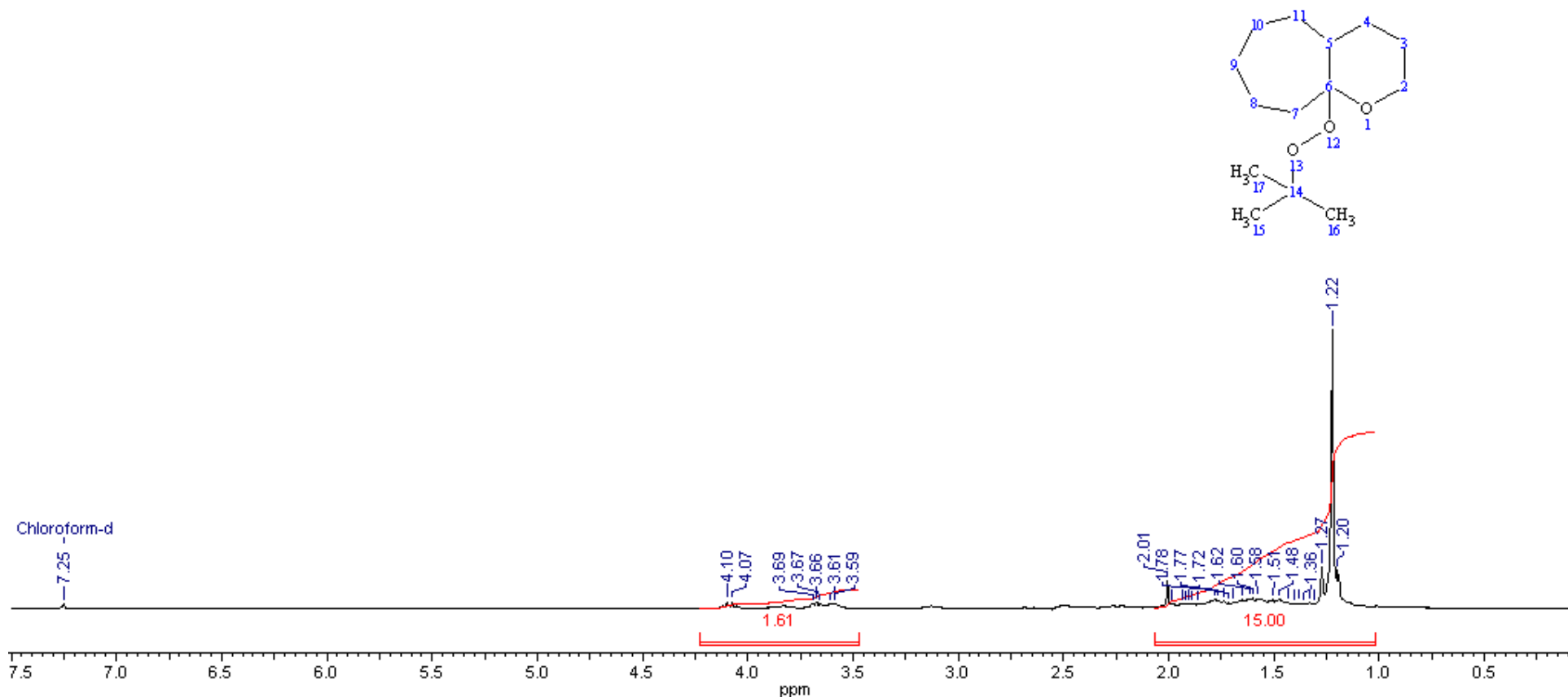
D:\EDL\Zd-108.0 ЗДВИЖКОВ. Zd-108 тонкий слой, пл. KBr

19.02.2013

¹H NMR 9a-(*tert*-Butylperoxy)decahydrocyclohepta[*b*]pyran (12c)

2 Jul 2013

Acquisition Time (sec)	0.6759	Comment	MVTS 00195	Date	25 Oct 2012 10:31:28
File Name				Frequency (MHz)	300.13
Nucleus	1H	Number of Transients	1	Original Points Count	8124
Pulse Sequence	zg	Solvent	CHLOROFORM-D	Points Count	8192
Temperature (degree C)	28.200			Sweep Width (Hz)	6009.62

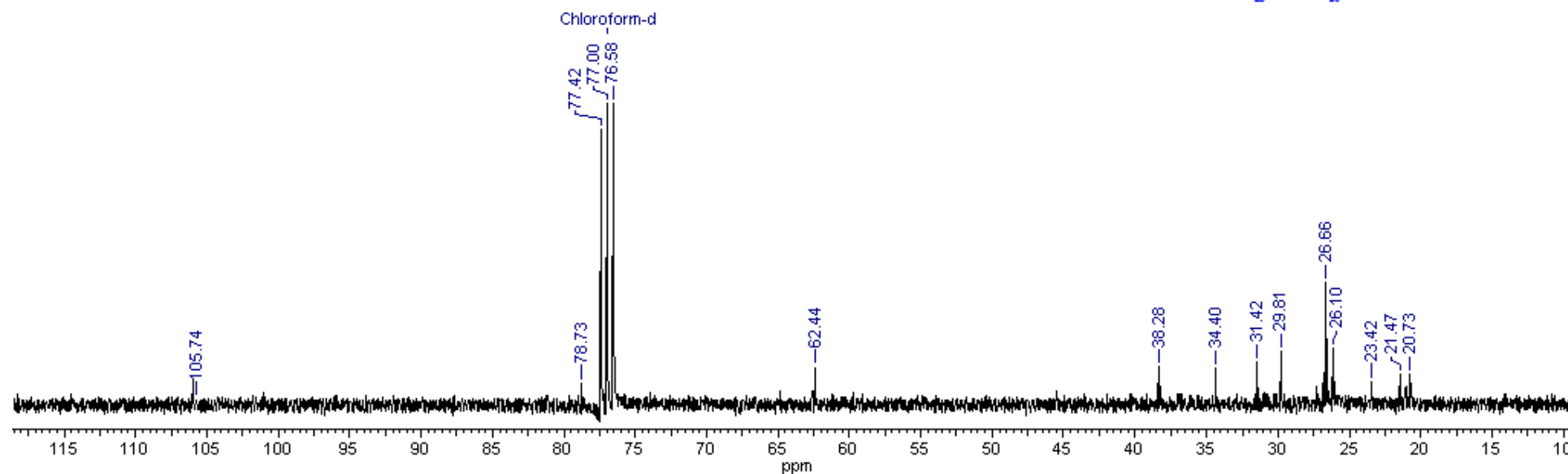
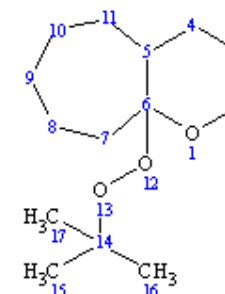


No.	Annotation	(ppm)
1	Chloroform-d	7.25

¹³C NMR 9a-(*tert*-Butylperoxy)decahydrocyclohepta[*b*]pyran (12c)

2 Jul 2013

Acquisition Time (sec)	0.4501	Comment	Avance-300, C-13, CDCl3		Date	25 Oct 2012 10:33:36	
File Name					Frequency (MHz)	75.48	
Nucleus	13C	Number of Transients	207	Original Points Count	16308	Points Count	16384
Pulse Sequence	zgpg30base	Solvent	CHLOROFORM-D	Sweep Width (Hz)	18115.94	Temperature (degree C)	28.400



HRMS 9a-(*tert*-Butylperoxy)decahydrocyclohepta[*b*]pyran (12c)

Display Report

Analysis Info

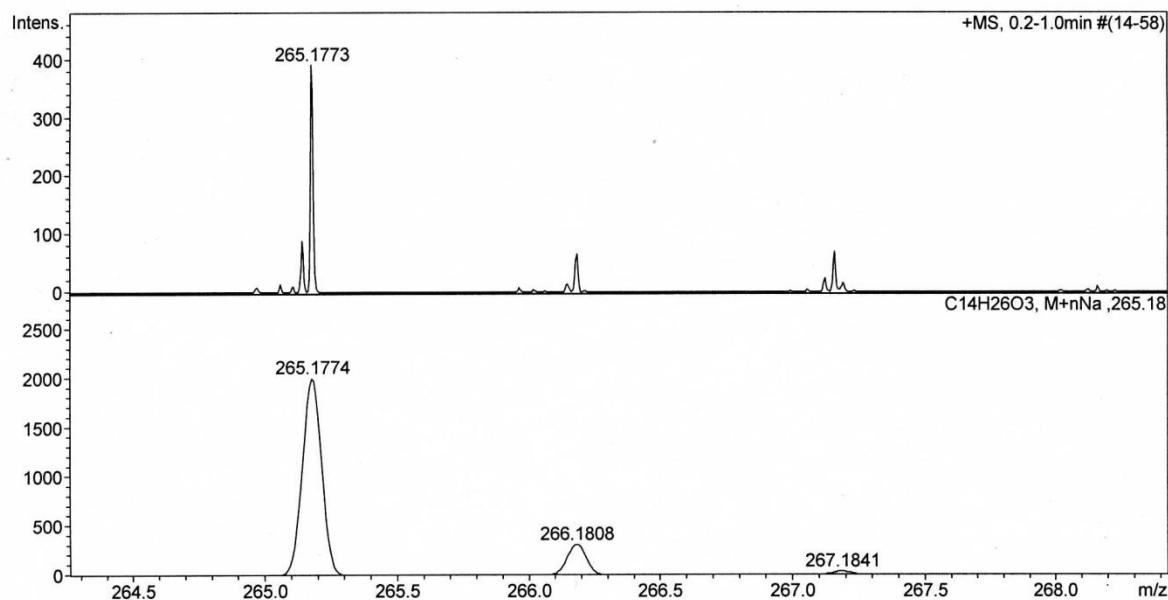
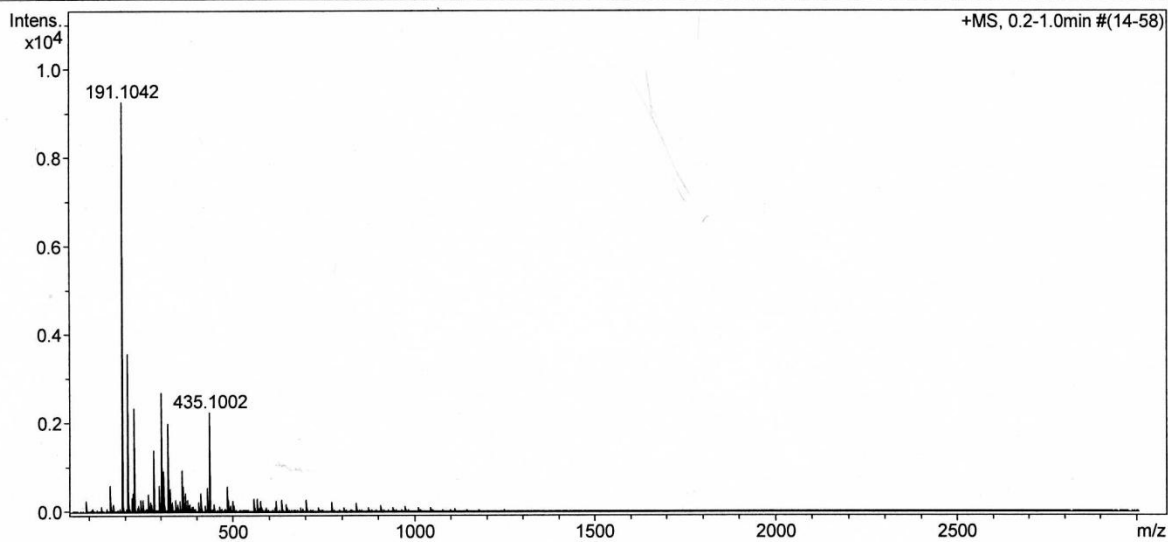
Analysis Name D:\Data\Ivanova\ZD-130&clb.d
Method tune_low.m
Sample Name /TERN ZD-130&clb
Comment CH3CN dill 200, clb added

Acquisition Date 28.02.2013 20:00:45

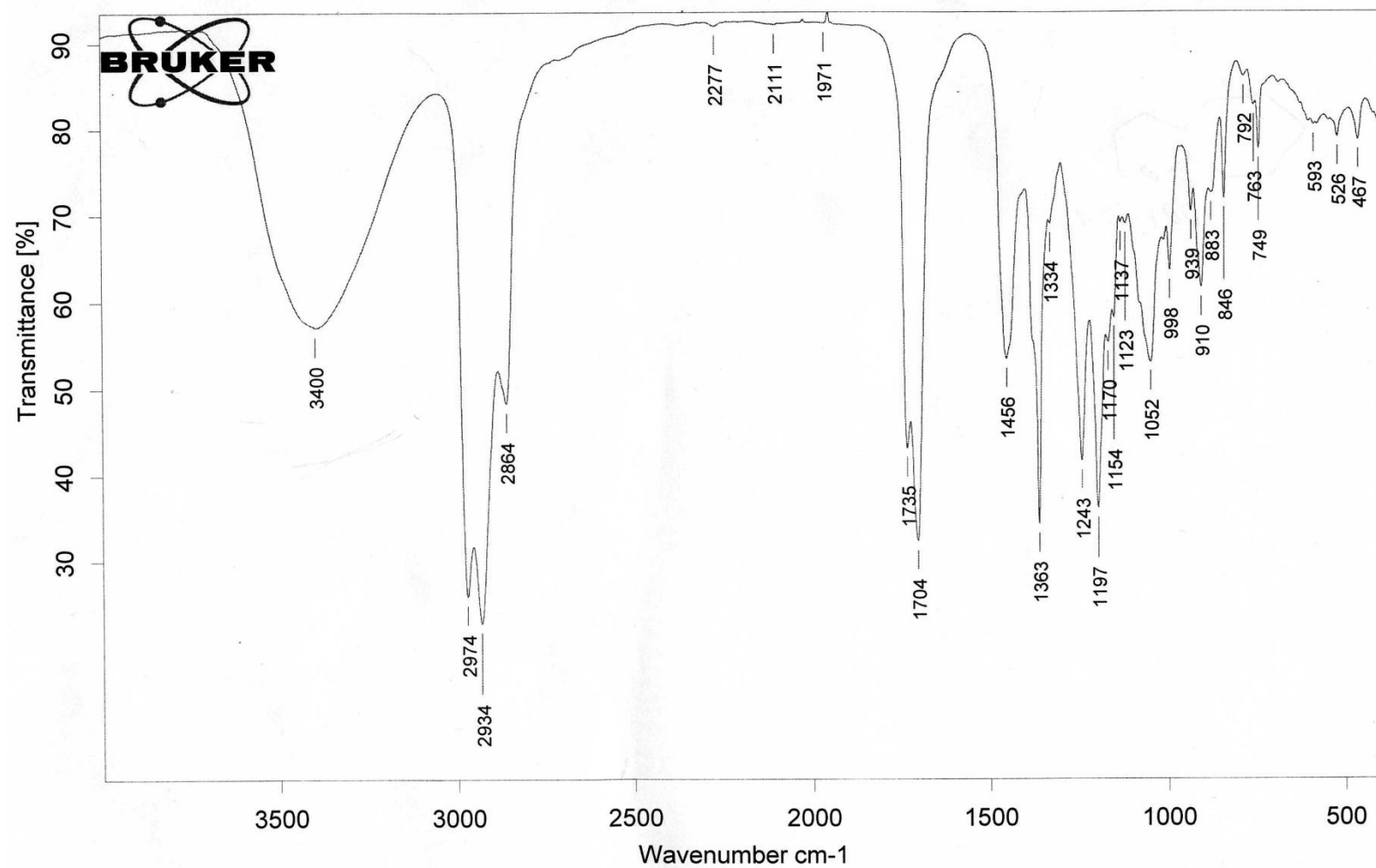
Operator BDAL@DE
Instrument maXis 43

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



IR 9a-(*tert*-Butylperoxy)decahydrocyclohepta[*b*]pyran (12c)



D:\EDL\Zd-130.0

ЗДВИЖКОВ. Zd-130 , тонкий слой, пл. KBr

19.02.2013