

Supporting Information

Synthetic Studies toward the Total Synthesis of Tautomycetin

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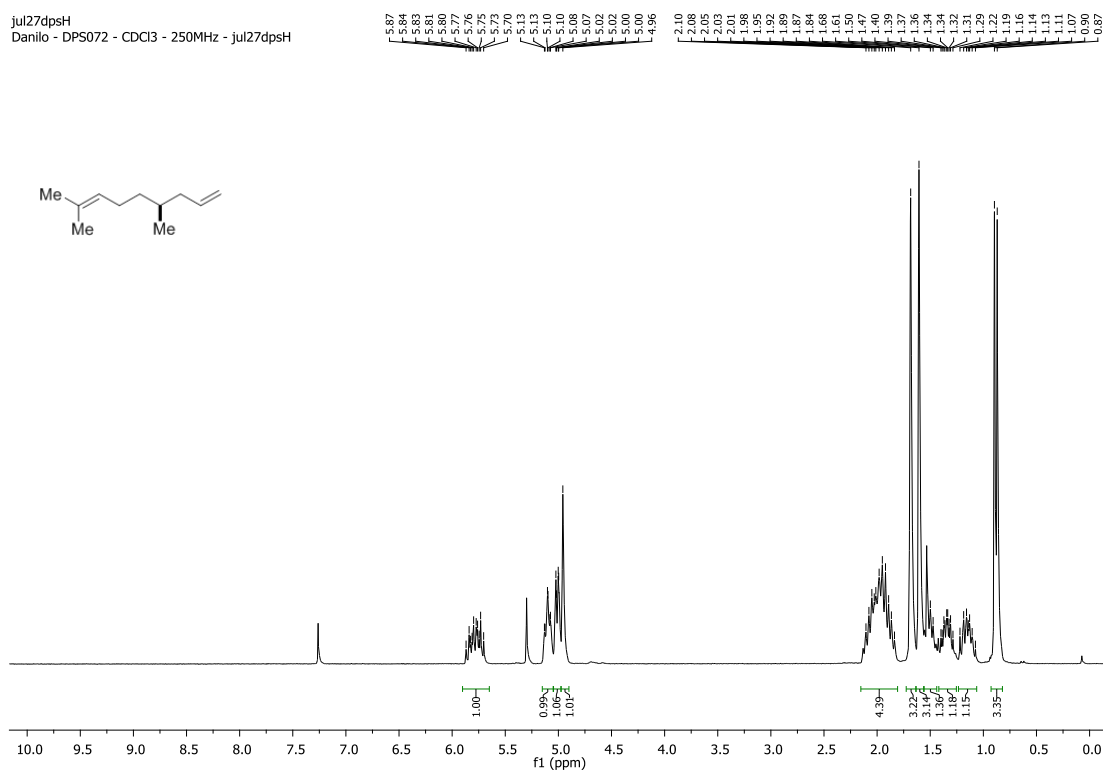
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renata.marcia_de_figueiredo@enscm.fr

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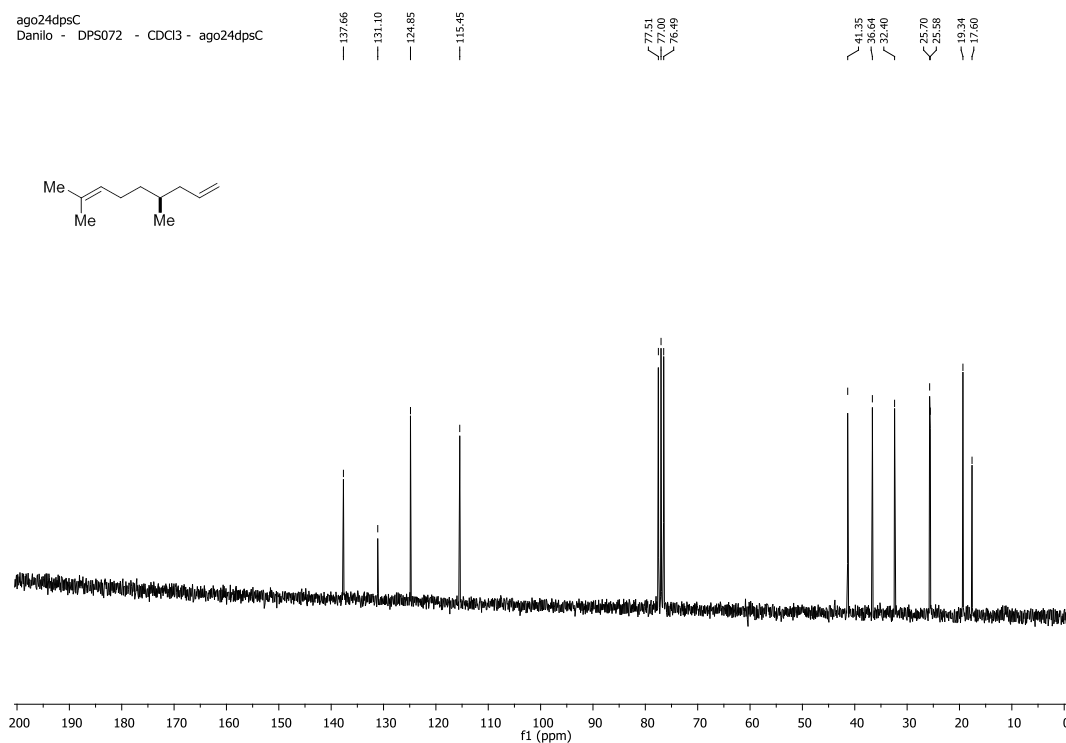
SPECTRA, MOSHER ANALYSIS and HPLC profiles

jul27dpsH
Danilo - DPS072 - CDCl₃ - 250MHz - jul27dpsH



¹H NMR of **14** (CDCl₃, 250 MHz)

ago24dpsC
Danilo - DPS072 - CDCl₃ - ago24dpsC



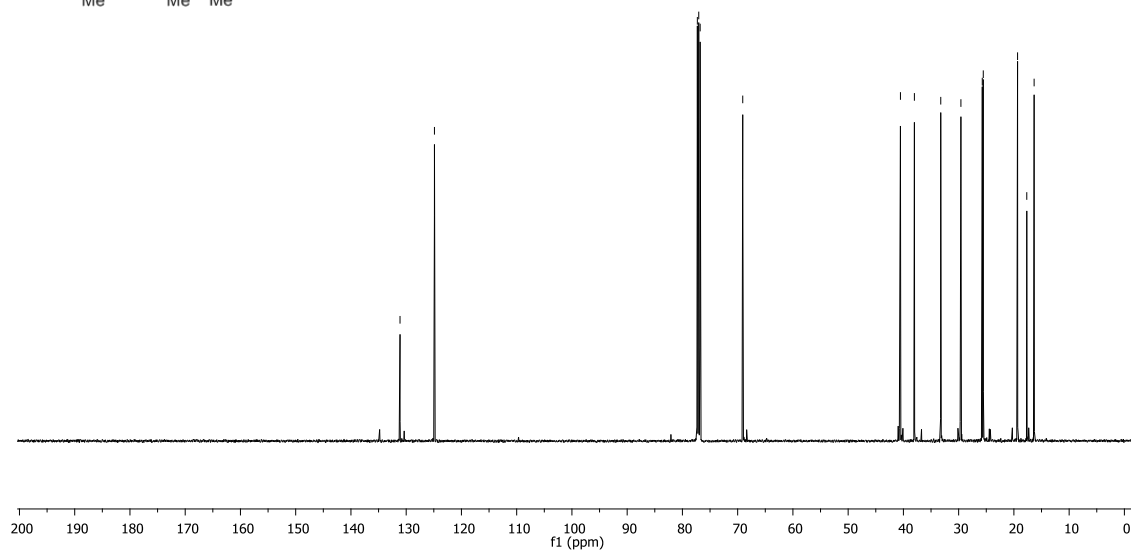
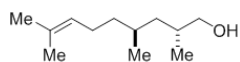
¹³C NMR of **14** (CDCl₃, 62.5 MHz)

mar24dpsH1
 Danilo - DPSA - CDCl₃ - Av 500 MHz - mar24dpsH1

131.1
 124.9

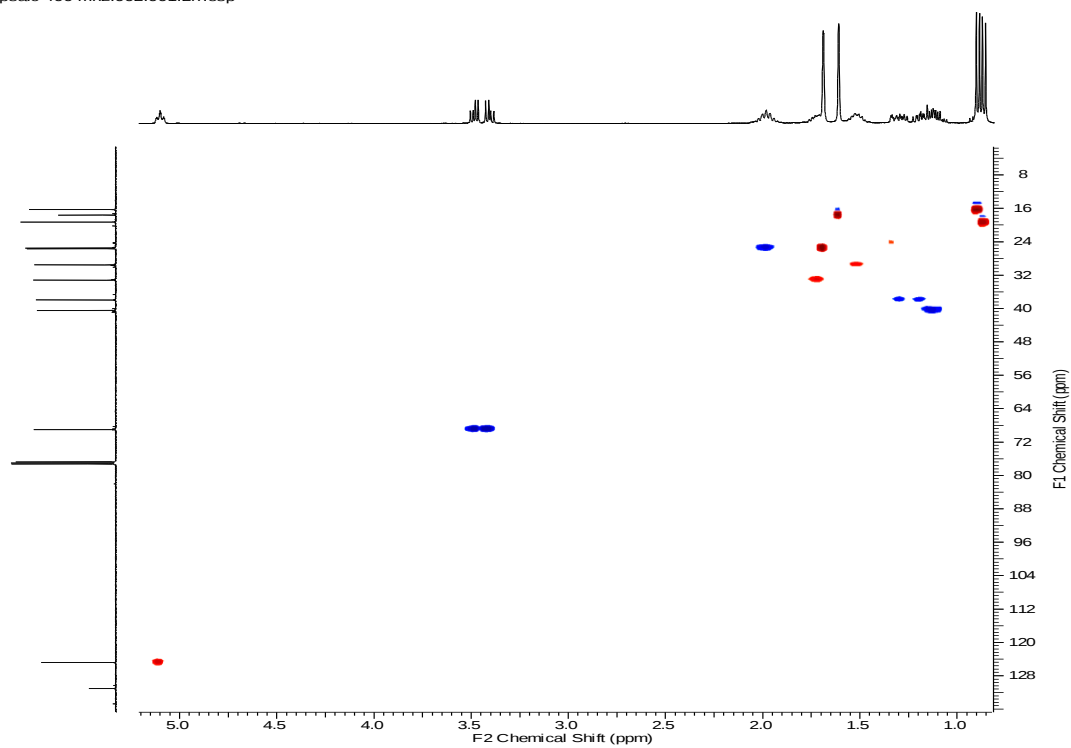
77.3
 77.0
 76.8
 69.1

40.5
 38.0
 33.2
 29.6
 25.7
 25.5
 19.3
 17.7
 16.3

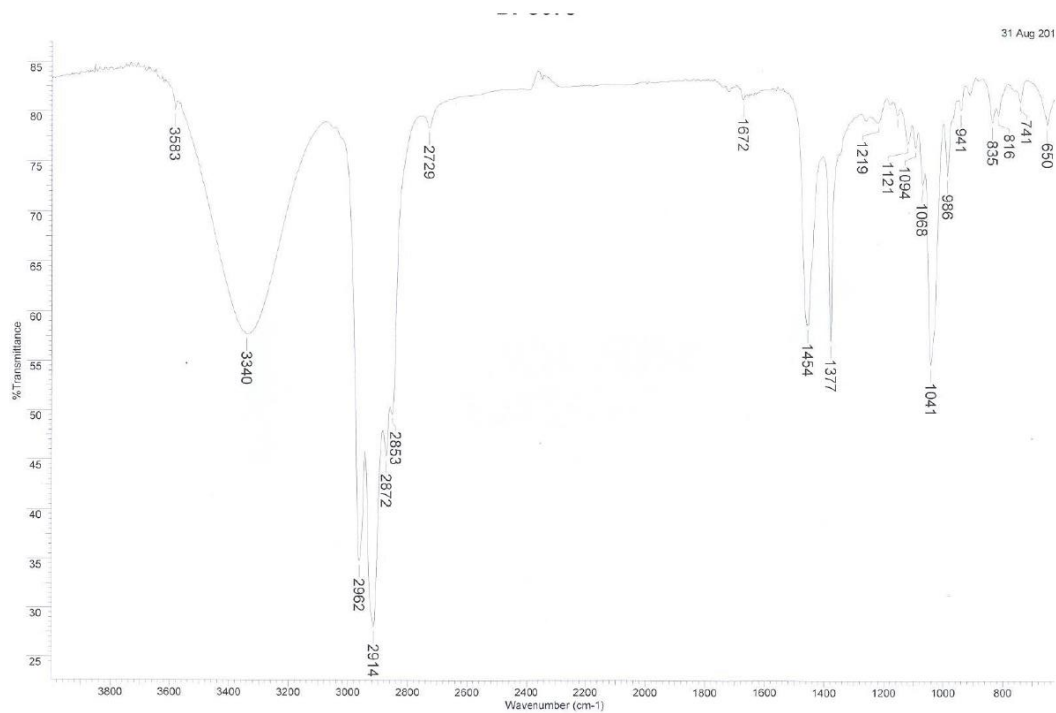


¹³C NMR of **15** (CDCl₃, 125 MHz)

dpsalc 400.mhz.002.001.2rr.esp

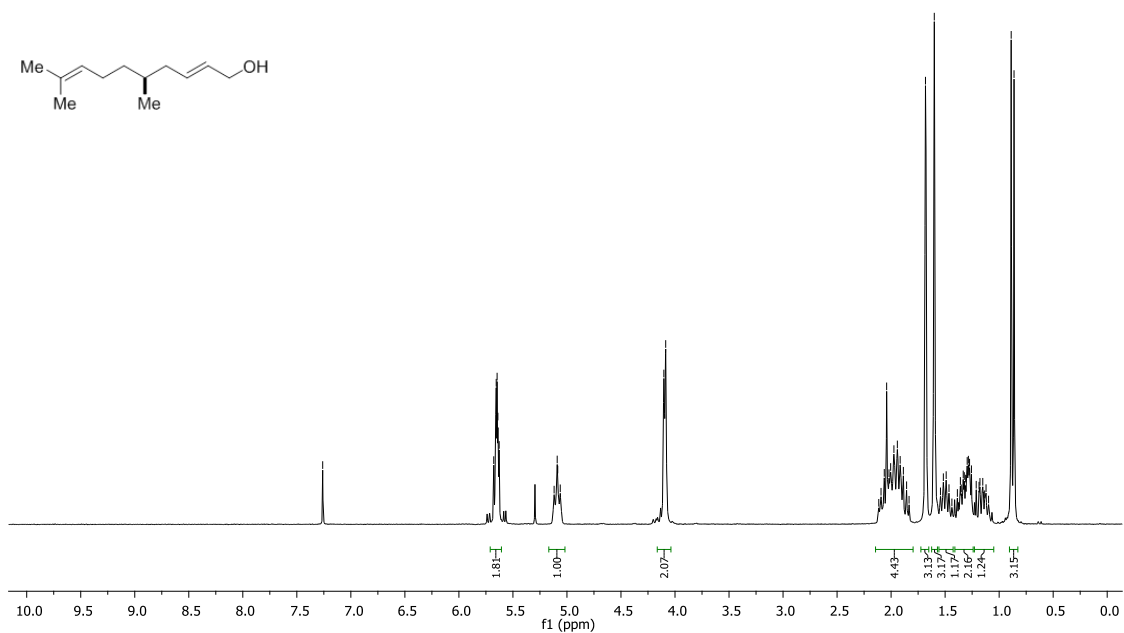
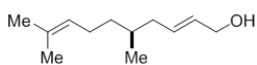
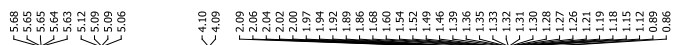


HSQC of **15**



IR of 15

jun30dpsH
Danilo - DPS064 - CDCl3 - 250MHz - jun30dpsH

¹H NMR of **16** (CDCl₃, 250 MHz)

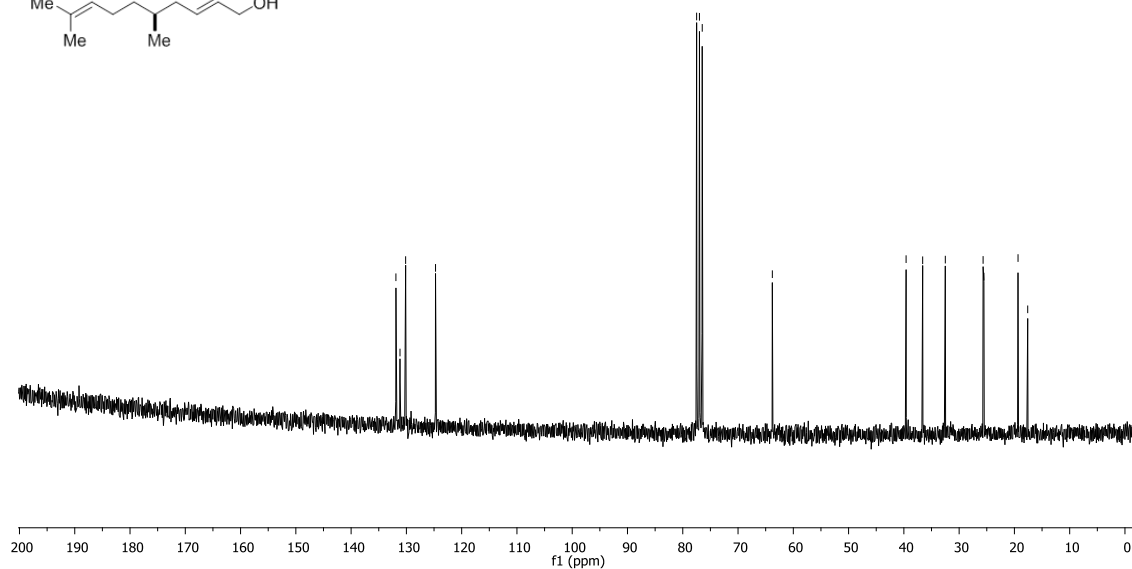
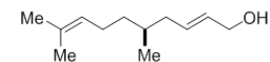
ago26dpsC1
Danilo - DPS64 - CDCl3/250mhz - ago26dpsC1

62.421
41.061
31.116
06.161

77.77
76.48
75.00

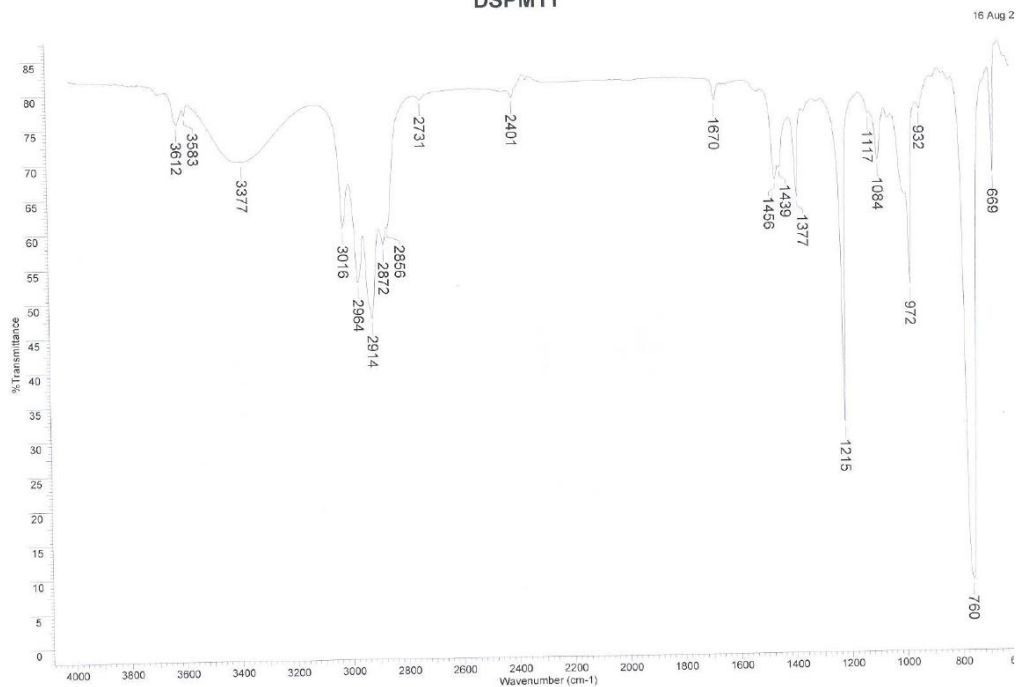
62.69

55.26
49.36
39.06
35.35
25.52
14.61
11.59



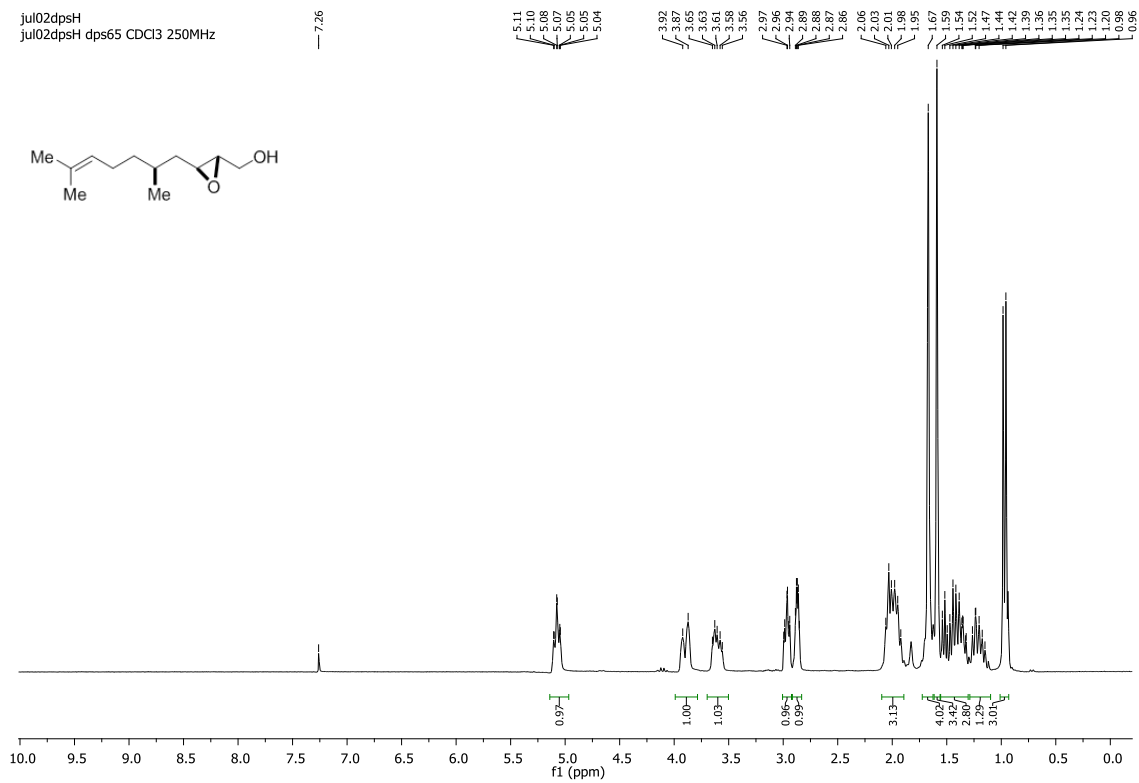
¹³C NMR of **16** (CDCl₃, 62.5 MHz)

DSPM11



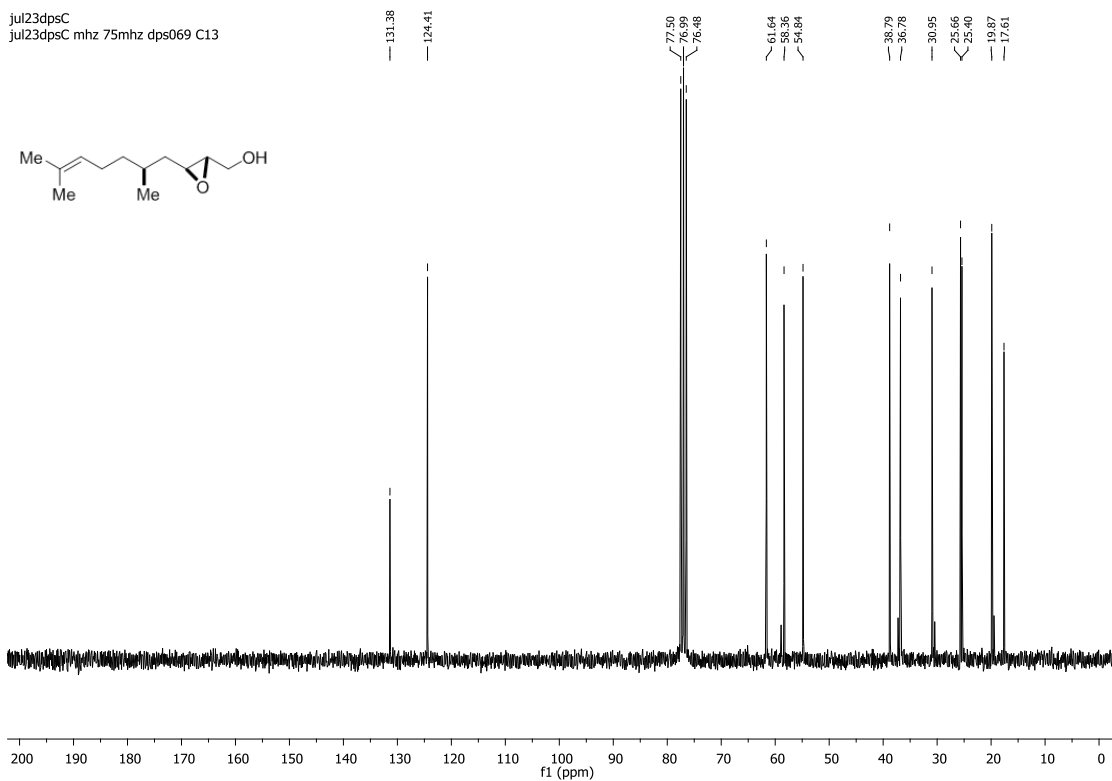
IR of **16**

jul02dpsH
jul02dpsH dps65 CDCl3 250MHz

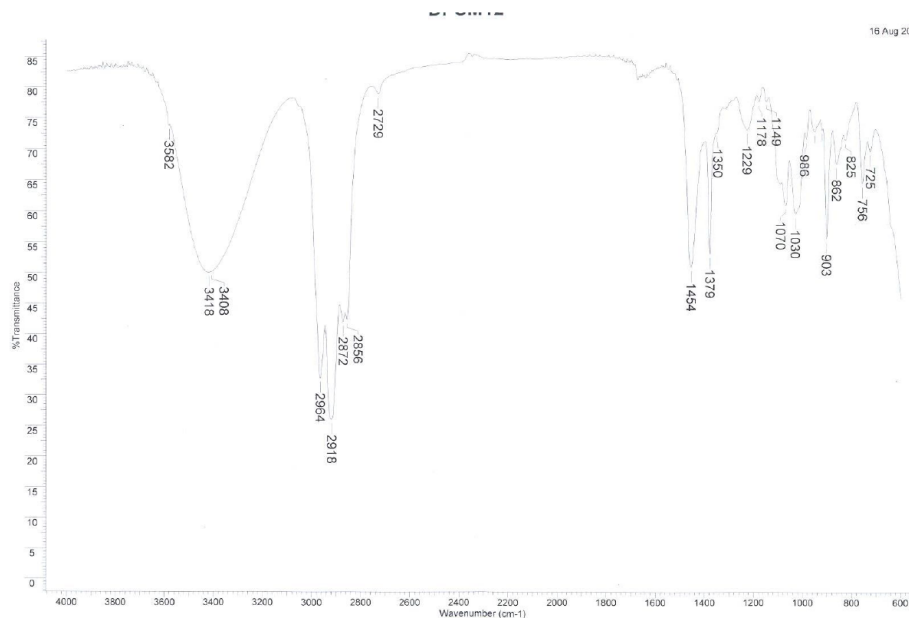


¹H NMR of **17** (CDCl₃, 250 MHz)

jul23dpsC
jul23dpsC mhz 75mhz dps069 C13



¹³C NMR of **17** (CDCl₃, 62.5 MHz)



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Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 3.0 mDa / DBE: min = -10.0, max = 100.0

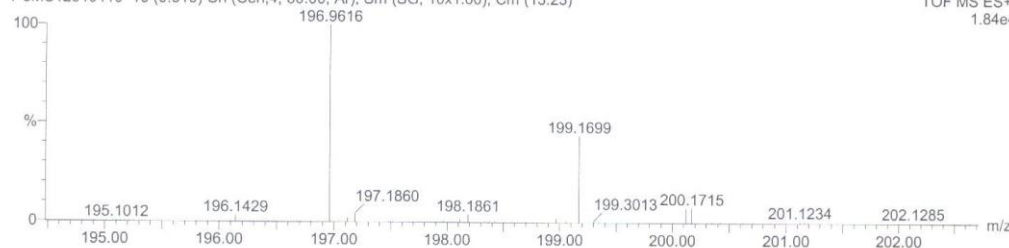
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Even Electron Ions

35 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Q-TOF RMF DS01 / Mmono = 198,1620
Y-JMC12040410 16 (0.310) Cn (Cen,4, 80.00, Ar); Sm (SG, 10x1.00); Cm (13:23)

4-APR-2012
TOF MS ES+
1.84e4

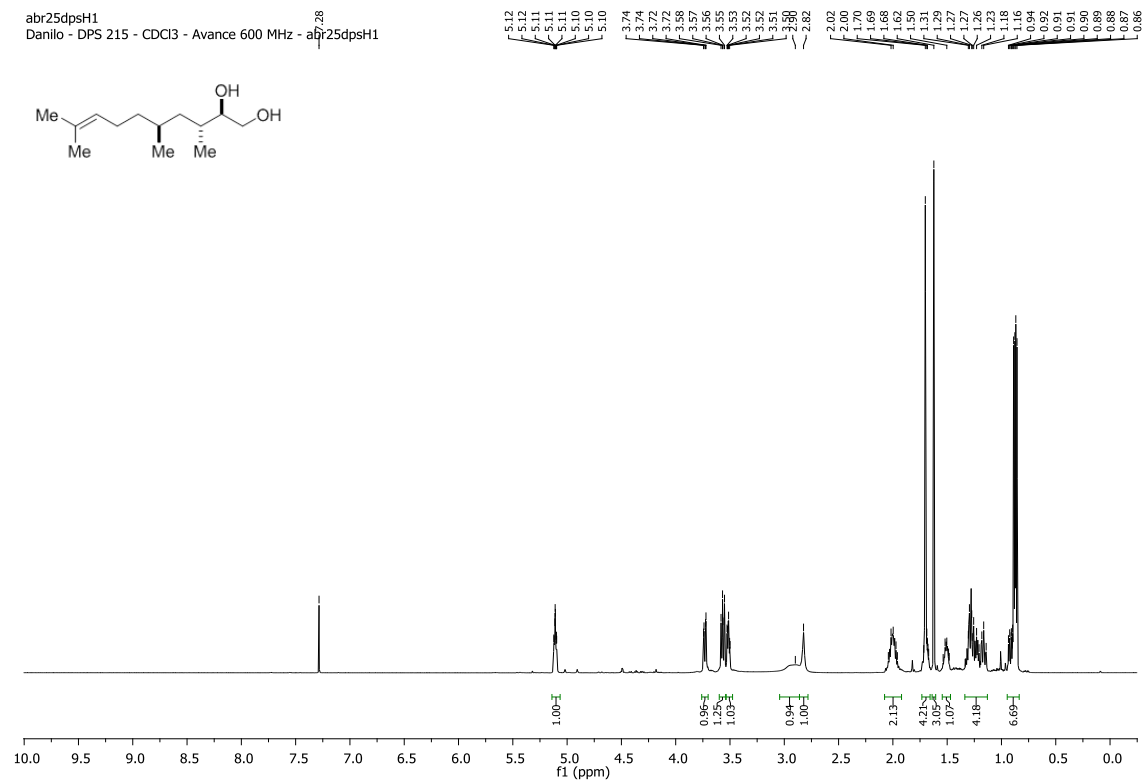


Minimum:
Maximum:

Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
199.1699	199.1698	0.1	0.5	1.5	1	C12 H23 O2

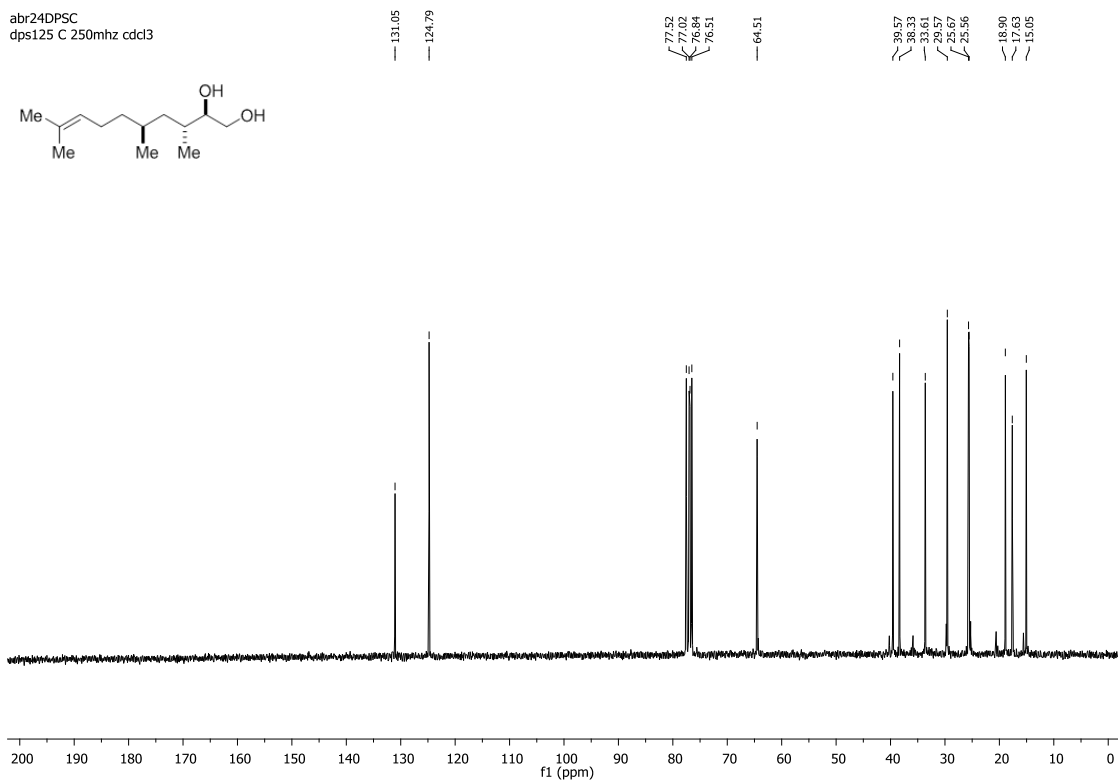
HRMS (ESI+) m/z calculated for $C_{12}H_{23}O_2$ $[M+H]^+$ 199.1698

abr25dpsH1
Danilo - DPS 215 - CDCl₃ - Avance 600 MHz - abr25dpsH1

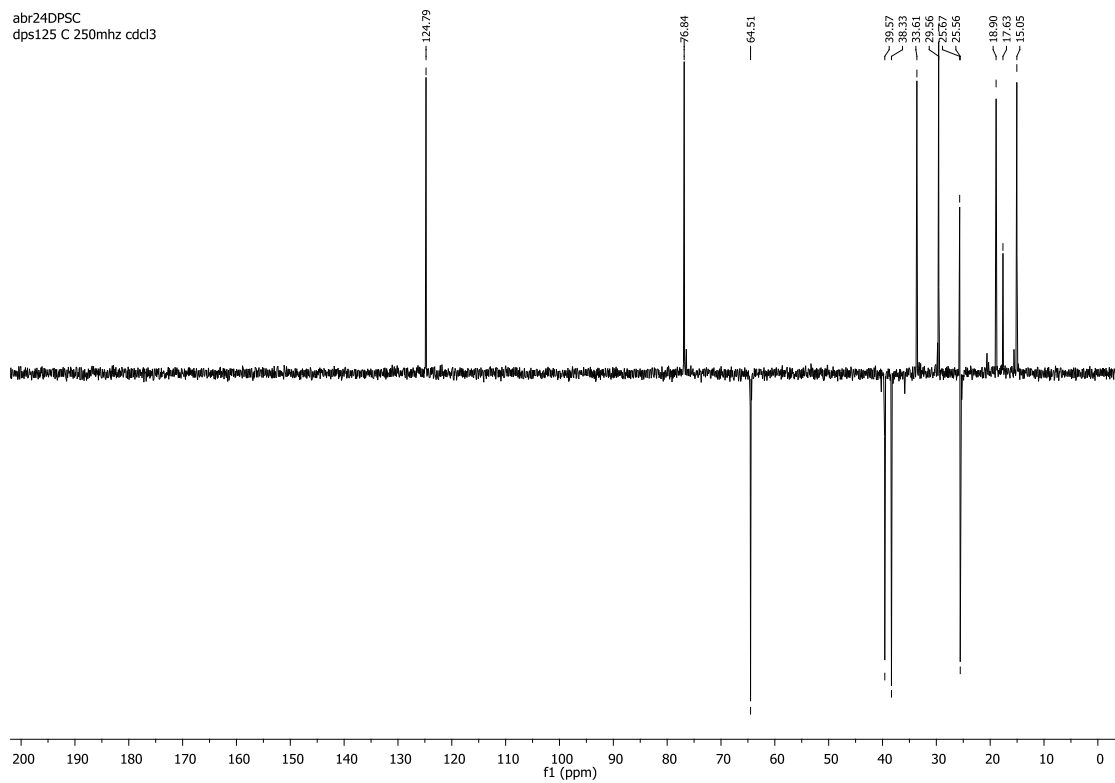


¹H NMR of **18** (CDCl₃, 600 MHz)

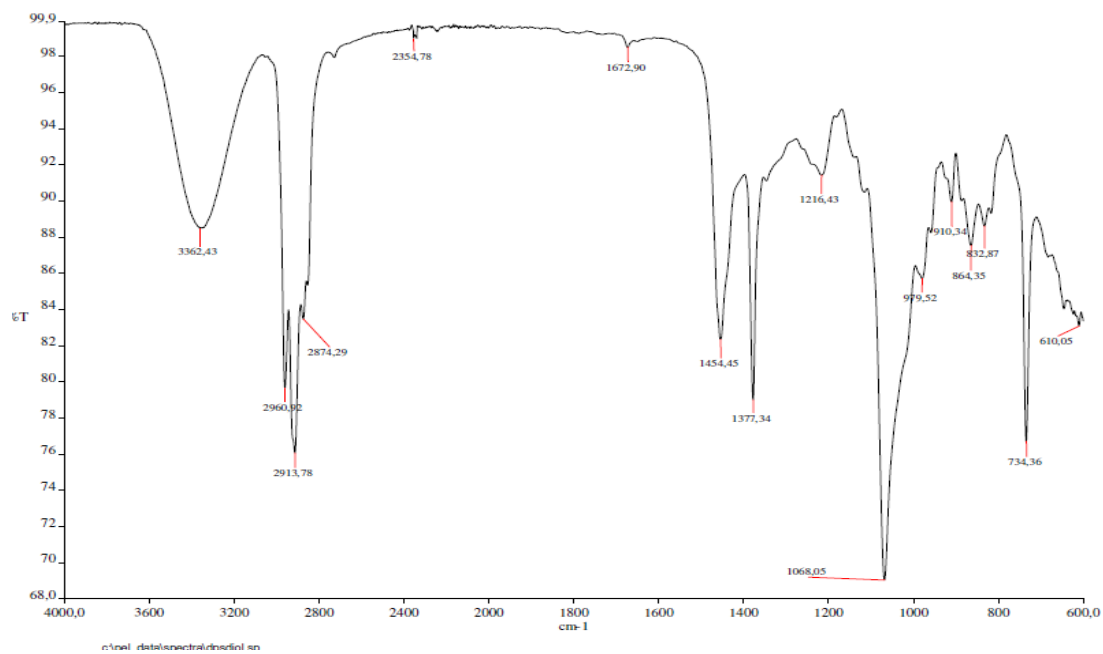
abr24DPSC
dps125 C 250mhz cdcl3



¹³C NMR of **18** (CDCl₃, 62.5 MHz)



DEPT 135 of **18** (CDCl₃, 62.5 MHz)



IR of **18**

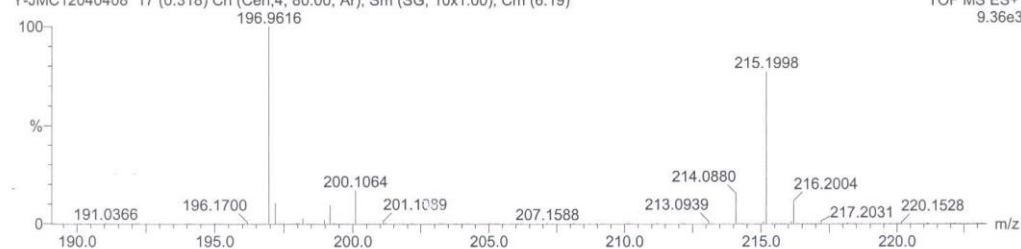
Single Mass Analysis

Tolerance = 3.0 mDa / DBE: min = -10.0, max = 100.0

Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Even Electron Ions

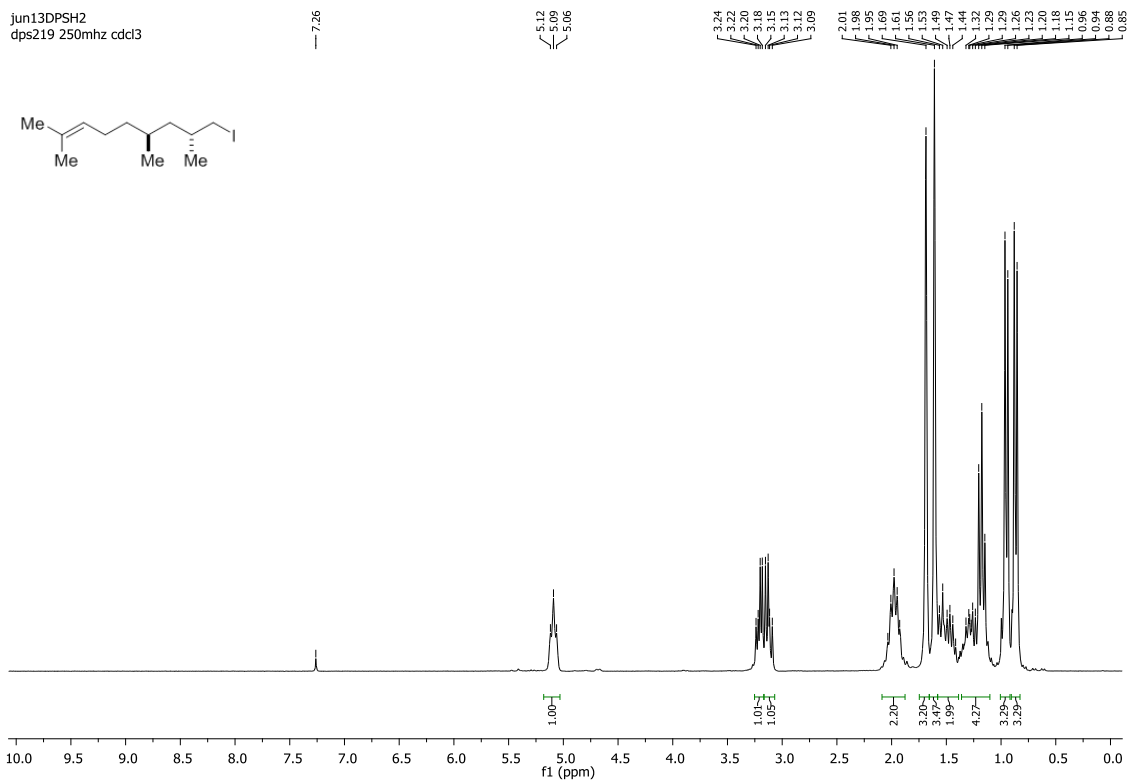
35 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Q-TOF RMF DS02 / Mmono = 214.1933
Y-JMC12040408 17 (0.318) Cn (Gen,4, 80.00, Ar); Sm (SG, 10x1.00); Cm (6:19)4-APR-2012
TOF MS ES+
9.36e3Minimum: -10.0
Maximum: 3.0 10.0 100.0

Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
215.1998	215.2011	-1.3	-6.1	0.5	1	C13 H27 O2

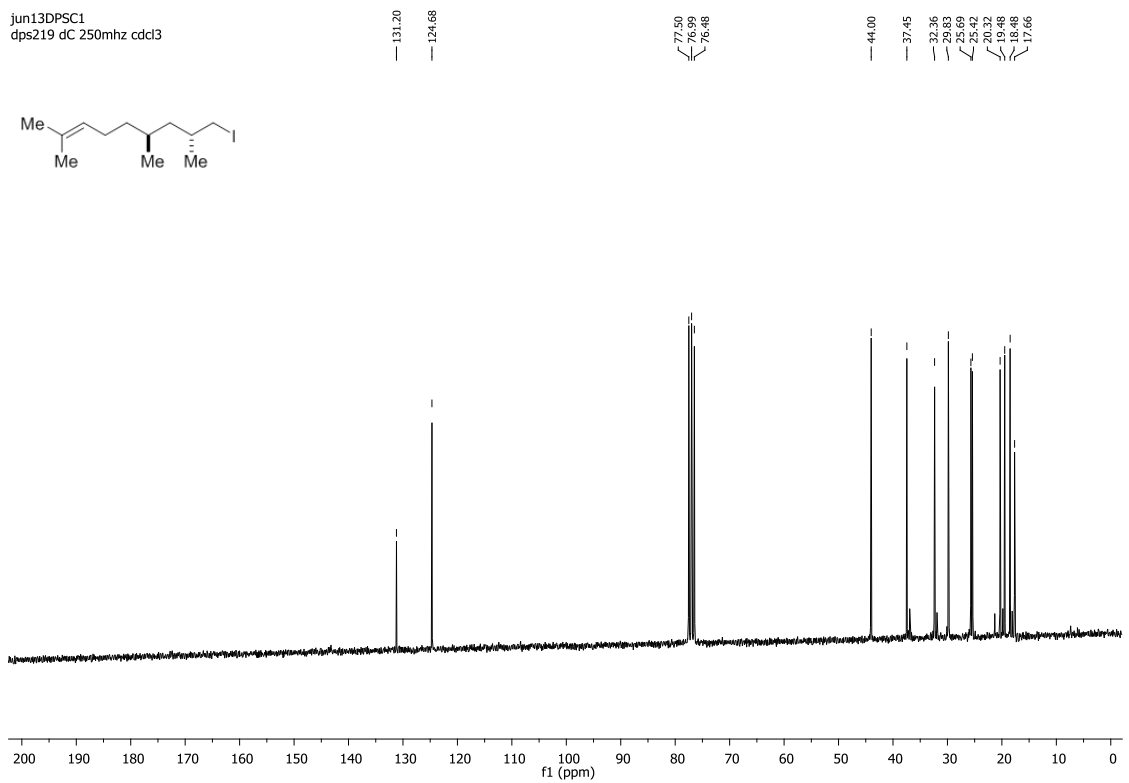
HRMS (ESI+) m/z calculated for $C_{13}H_{27}O_2$ $[M+H]^+$ 215.2011

jun13DPSh2
dps219 250mhz cdcl3

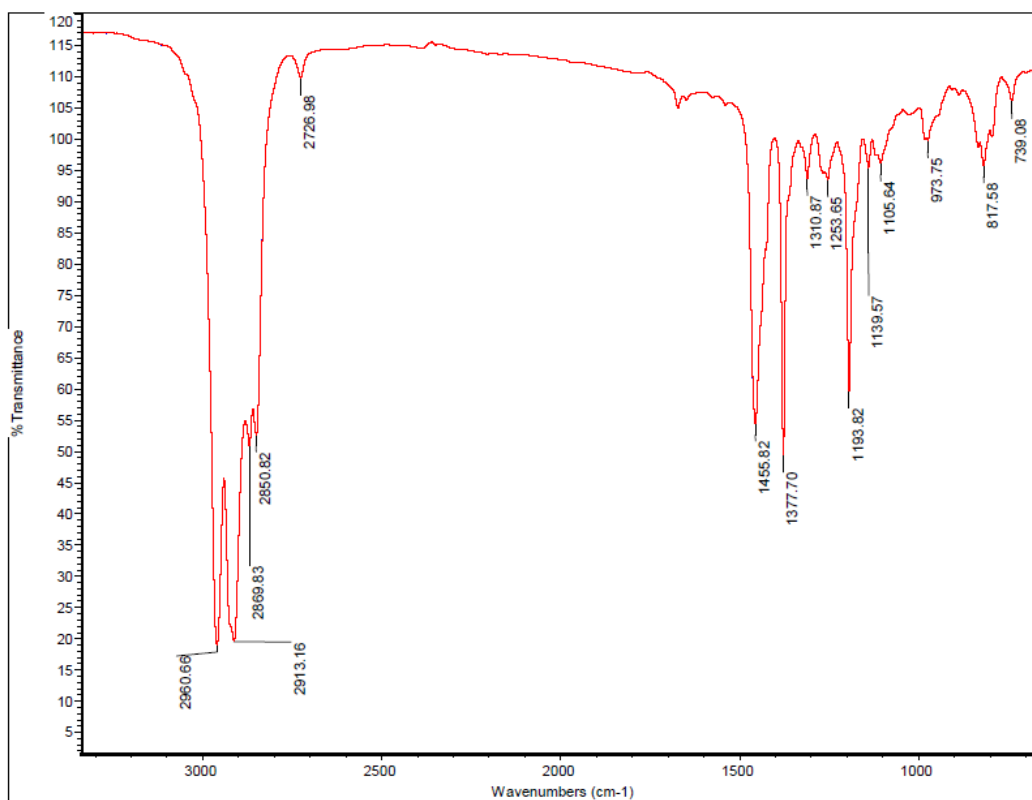


¹H NMR 7 (CDCl₃, 250 MHz)

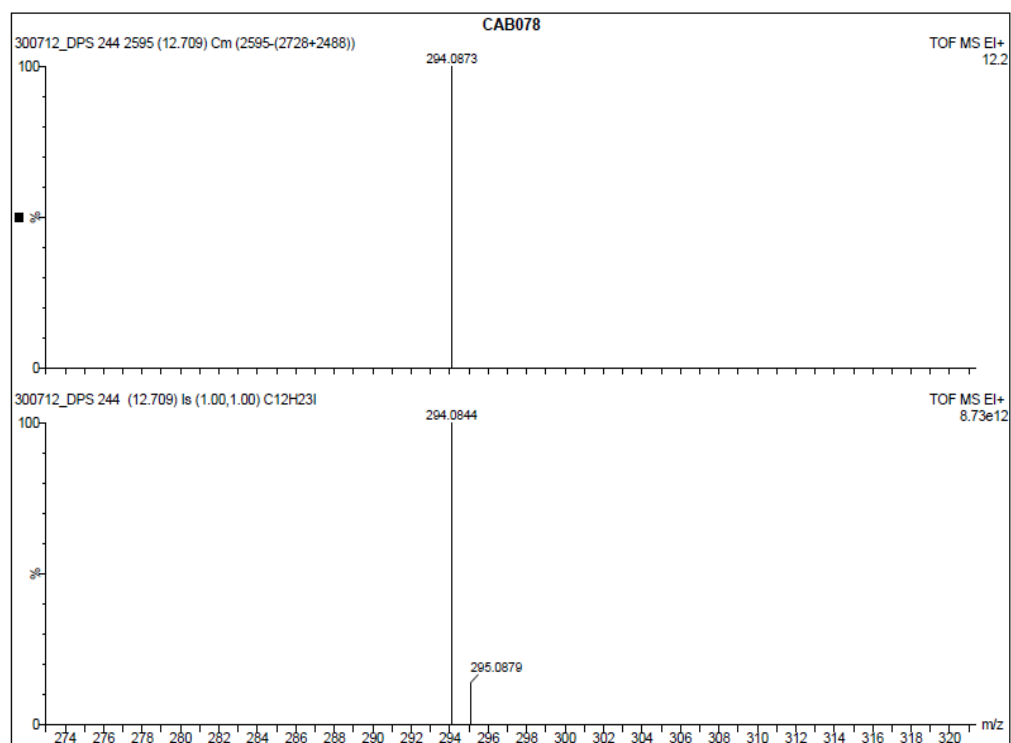
jun13DPSC1
dps219 dC 250mhz cdcl3



¹³C NMR 7 (CDCl₃, 62.5 MHz)

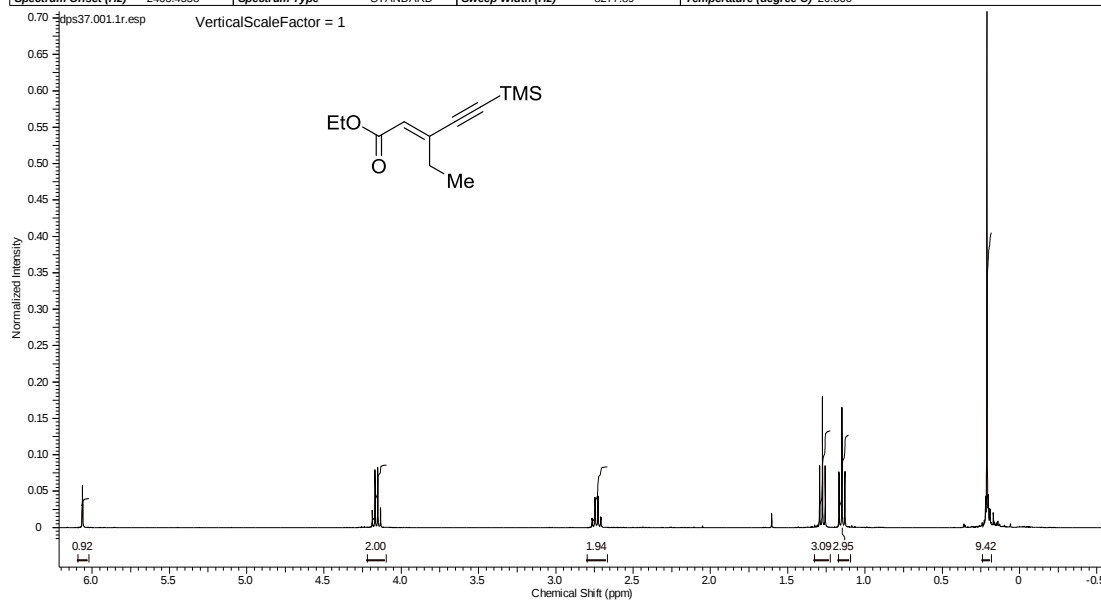


IR of 7



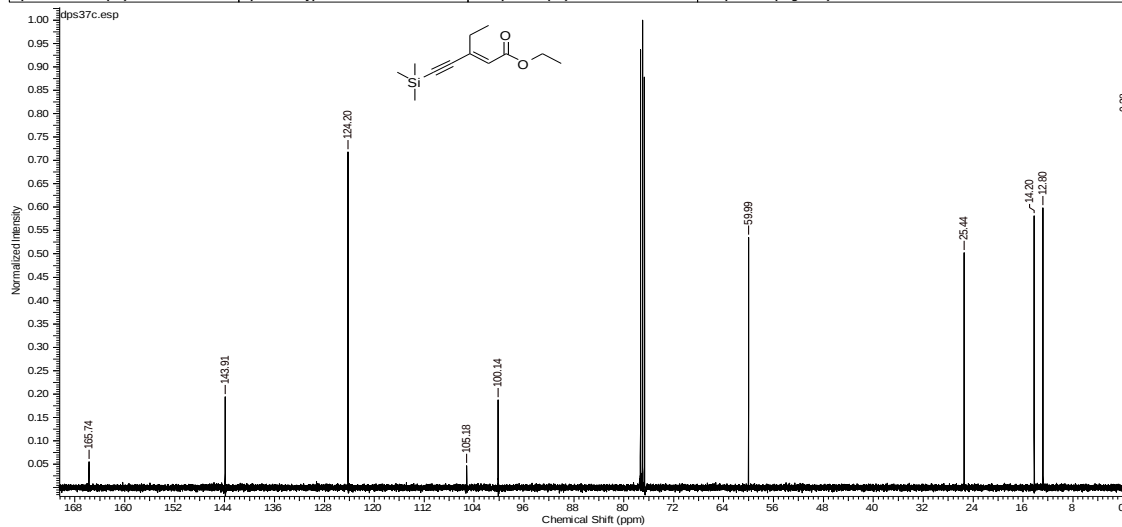
HRMS (ESI+) m/z calculated for C₁₂H₂₄I [M+H]⁺ 295.0879

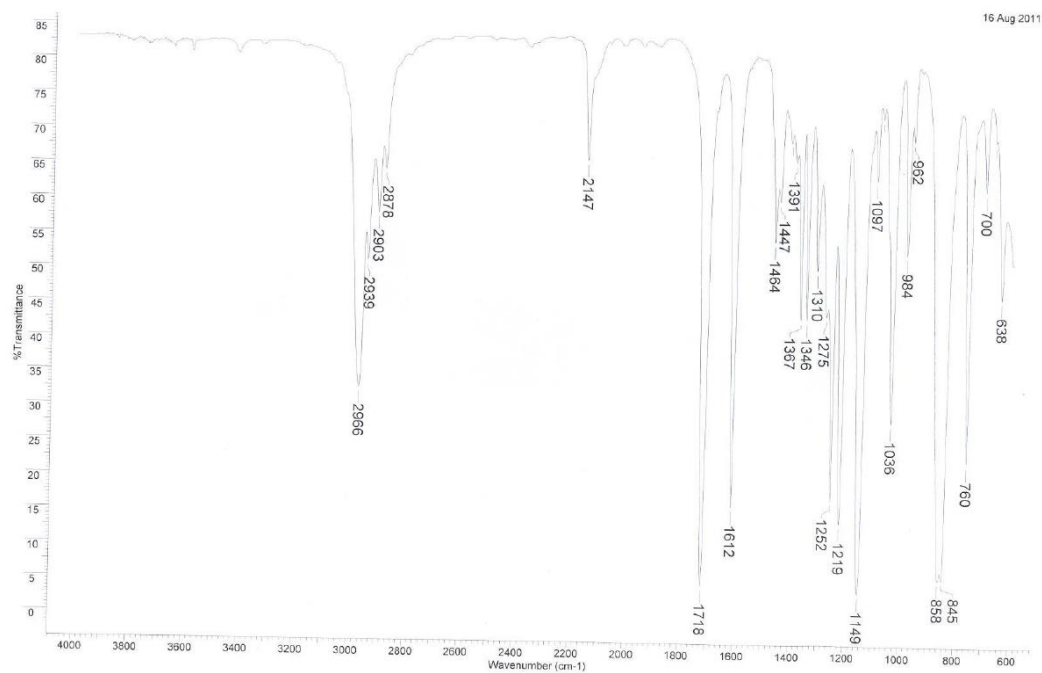
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Date Stamp	27 Jan 2011 12:52:32	File Name	C:\Users\Danilo\Documents\tautomicetinal\fid\dps37pdata\111r		
Frequency (MHz)	400.13	Nucleus	¹ H	Number of Transients	16
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Receiver Gain	90.50	SW(cyclical) (Hz)	8278.15	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2465.4558	Spectrum Type	STANDARD	Sweep Width (Hz)	8277.89
				Temperature (degree C)	20.360

¹H NMR of **19** (CDCl₃, 400 MHz)

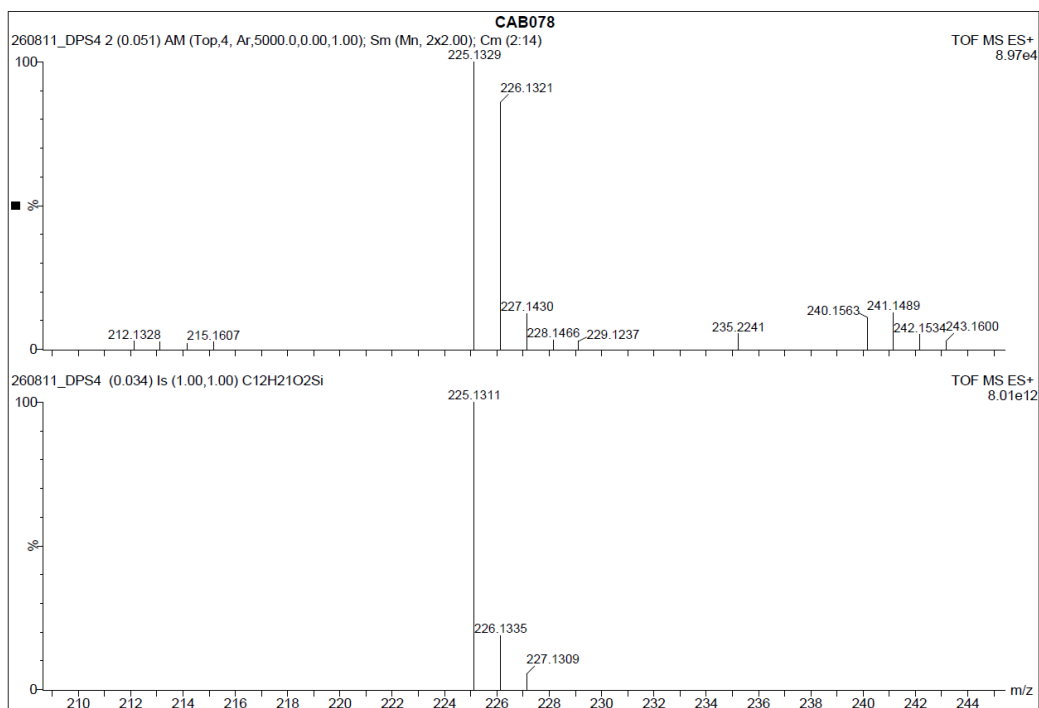
pent-2-inoato de etila

Acquisition Time (sec)	1.3664	Comment	dps037 C13CPD CDCl3 opt/topspin am2n3 48	Date	29 Jan 2011 04:46:08
Date Stamp	29 Jan 2011 04:46:08	File Name	C:\Users\Danilo\Documents\tautomicetinal\fid\dps37c\fid		
Frequency (MHz)	100.61	Nucleus	¹³ C	Number of Transients	2048
Original Points Count	32768	Owner	auto	Points Count	32768
Receiver Gain	456.10	SW(cyclical) (Hz)	23980.81	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	10057.2227	Spectrum Type	STANDARD	Sweep Width (Hz)	23980.08
				Temperature (degree C)	20.860

¹³C NMR of **19** (CDCl₃, 100 MHz)

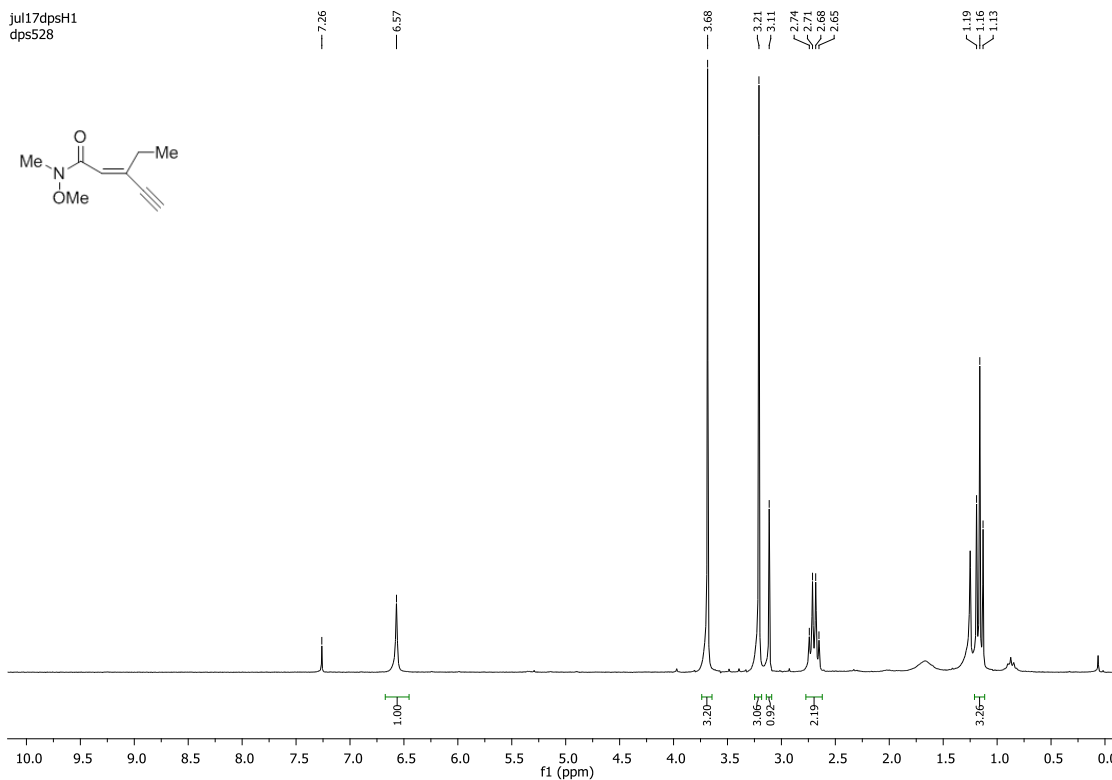
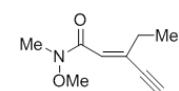


IR of **19**



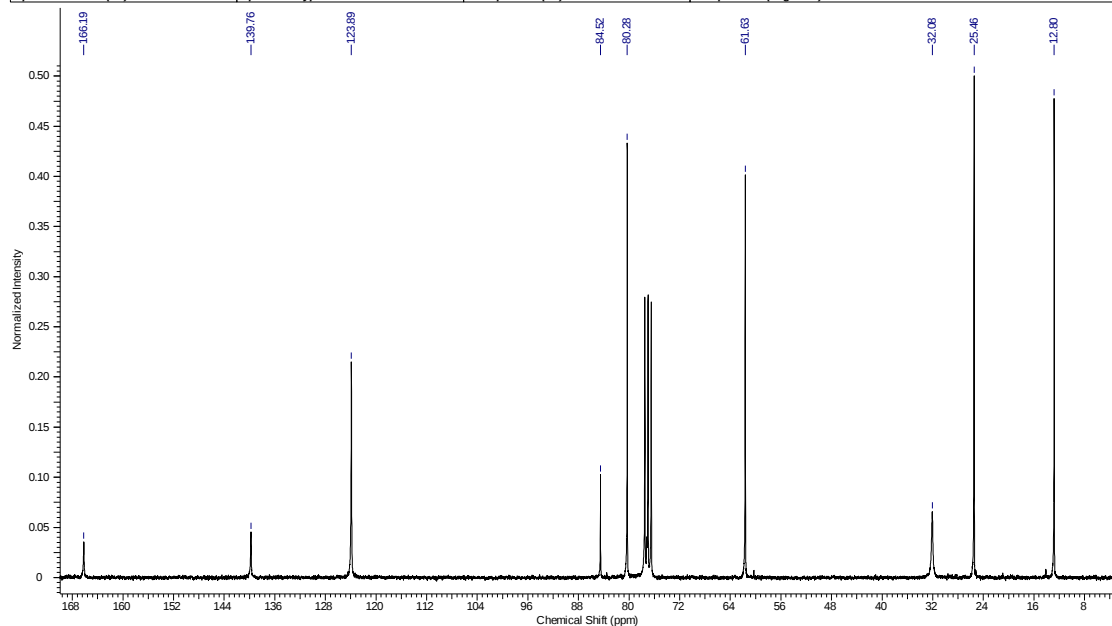
HRMS (ESI+) m/z calculated for $[M+H]^+$ 225.1311

jul17dpsH1
dps528



^1H NMR of **20** (CDCl_3 , 250 MHz)

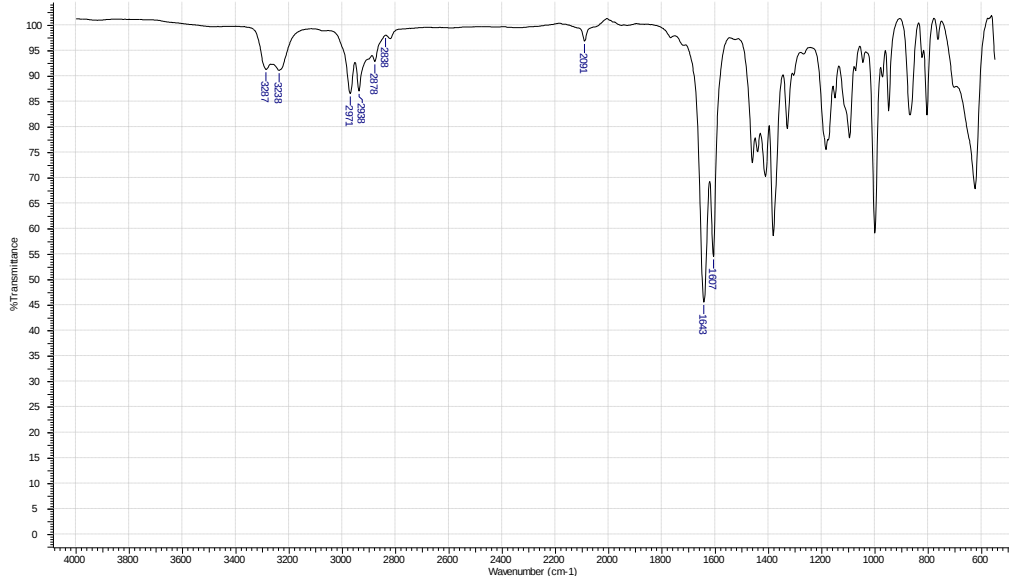
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Date Stamp	02 Aug 2013 07:25:04	File Name	C:\Users\Danilo P. Sant'\AnalDocuments\tautomicetina\fid\dps500-ldps537 C\2\data\111r	Number of Transients	14336
Frequency (MHz)	62.90	Nucleus	^{13}C	Origin	spect
Original Points Count	8192	Owner	roct	Points Count	32768
Receiver Gain	362.00	SW(cyclical) (Hz)	15060.24	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	6287.6929	Spectrum Type	STANDARD	Solvent	CHLOROFORM-d
		Sweep Width (Hz)	15059.78	Temperature (degree C)	25.160



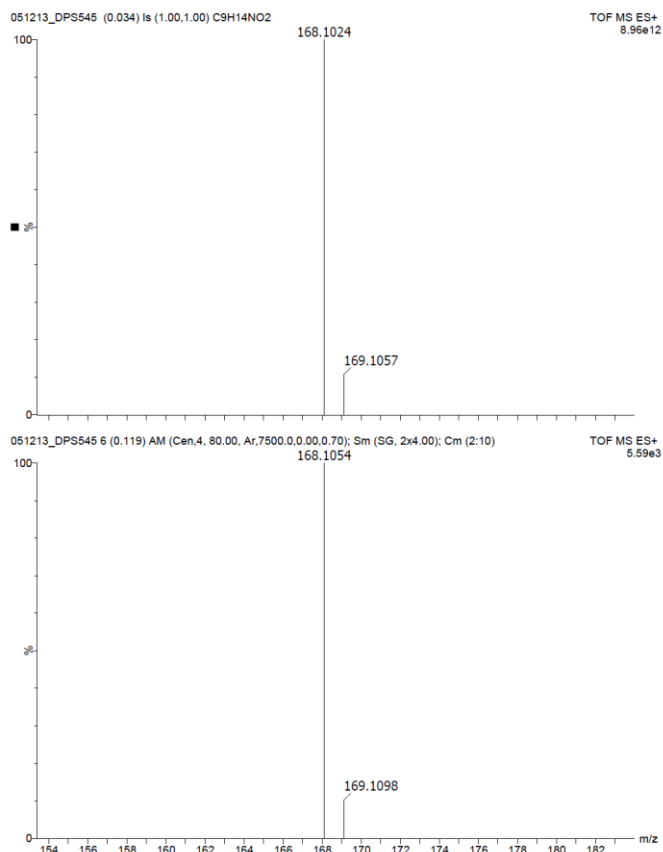
^{13}C NMR of **20** (CDCl_3 , 62.5 MHz)

10 Jan 2014

Title	brasileiro de verão (GMT-2:00)	Comment	Amostra 006 por LQOS data sexta-feira, dezembro 13 2013	
Origin	brasileiro de verão (GMT-2:00)	File Name	C:\USERS\DANILO P SANT'ANA\DOCUMENTS\T\AUTOMICETINA\AMIDA ACETILENO 2_1.SP	
Date Stamp	fri dec 13 16:50:00 2013 Horário brasileiro de verão (GMT-2:00)	Date	fri dec 13 16:50:10 2013 Horário brasileiro de verão (GMT-2:00)	
Technique	Infrared	Instrument	Spectrum Two	Spectral Region IR
X Axis	Wavenumber (cm-1)	Y Axis	%Transmittance	Spectrum Range 550.0000 - 4000.0000
Points Count	3451	Data Spacing	1.0000	

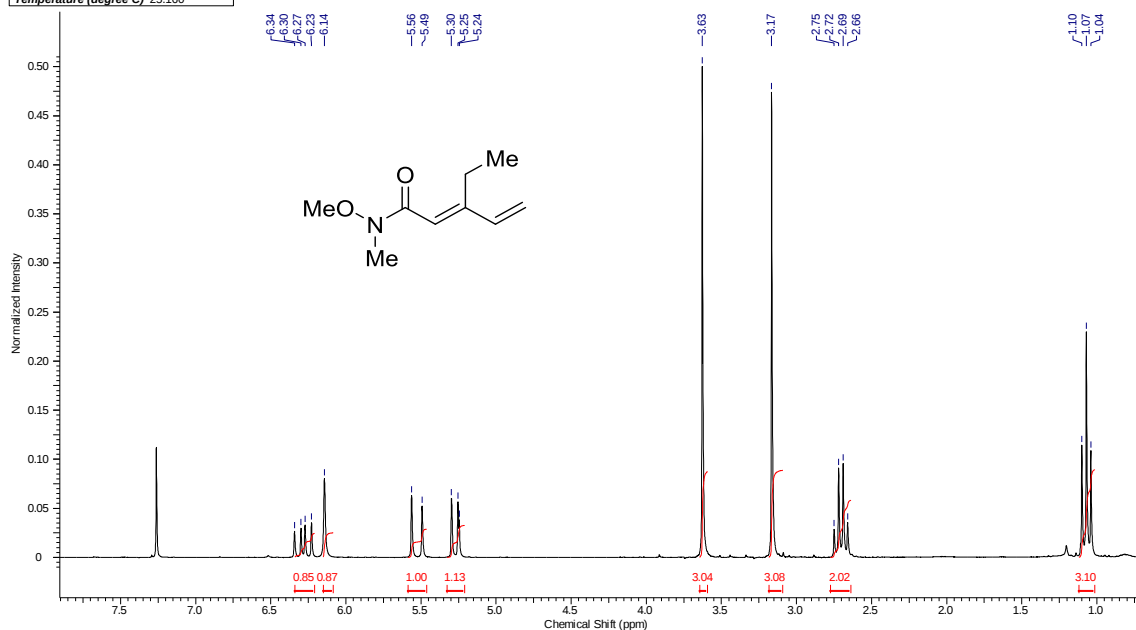


IR of 20

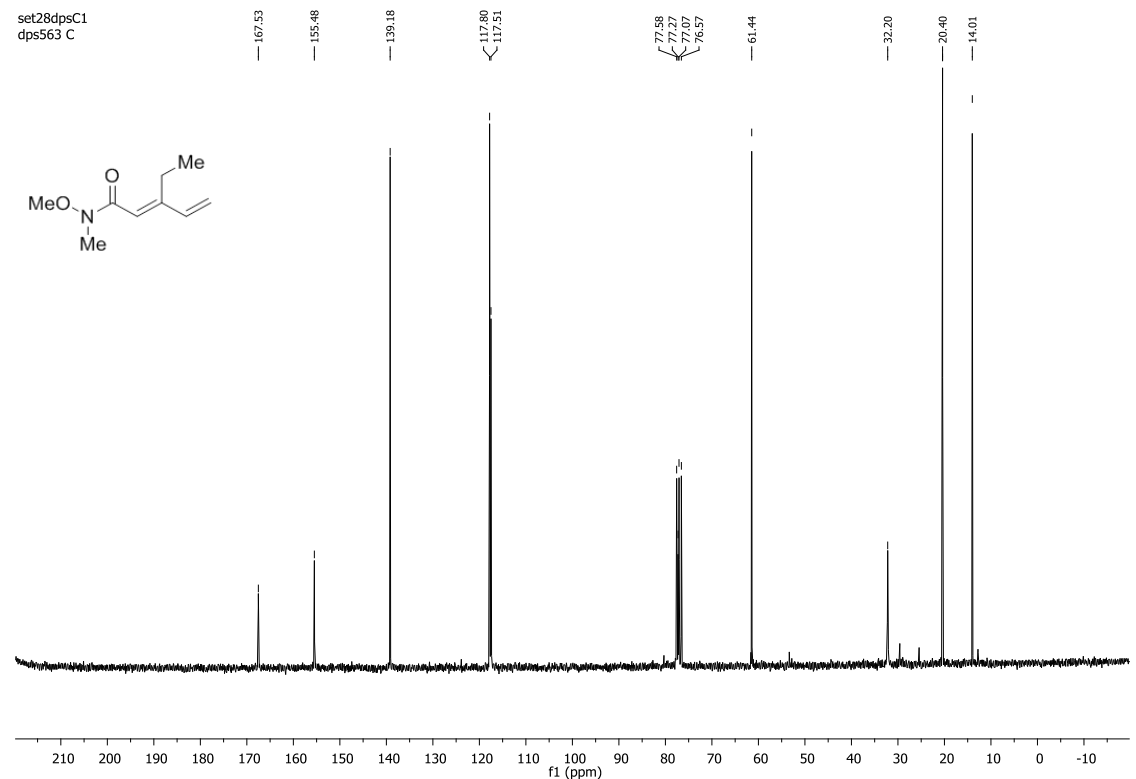


HRMS (ESI+) m/z calculated for $C_9H_{14}NO_2$ $[M+H]^+$ 168.1024

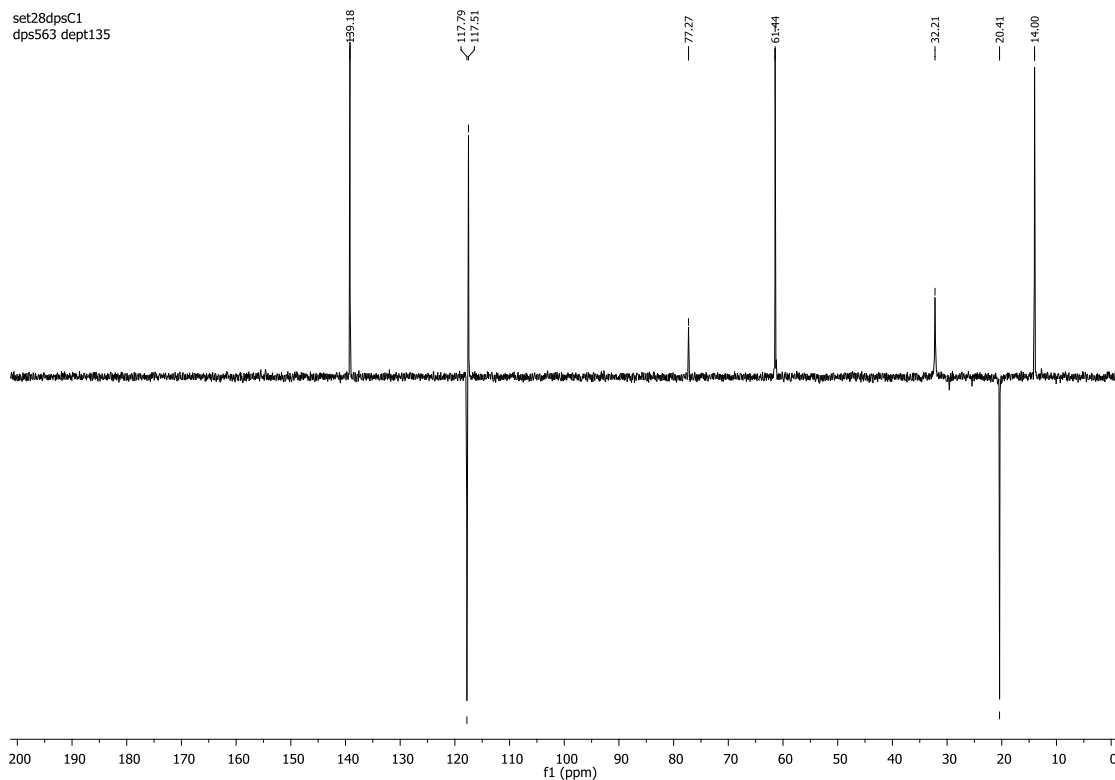
Acquisition Time (sec)	3.1654	Comment	dps563	Date	28 Sep 2013 16:07:44
Date Stamp	28 Sep 2013 16:07:44	File Name	C:\Users\Danilo P Sant\Ana\Documents\tautomicetinal\fid\dps500\dps563\1\data\1\1r		
Frequency (MHz)	250.13	Nucleus	¹ H	Number of Transients	1
Owner	root	Points Count	32768	Receiver Gain	128.00
Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	1750.9110	Spectrum Type	STANDARD
Temperature (degree C)	25.160			Original Points Count	16384
				SW(cyclical) (Hz)	5175.98
				Sweep Width (Hz)	5175.83



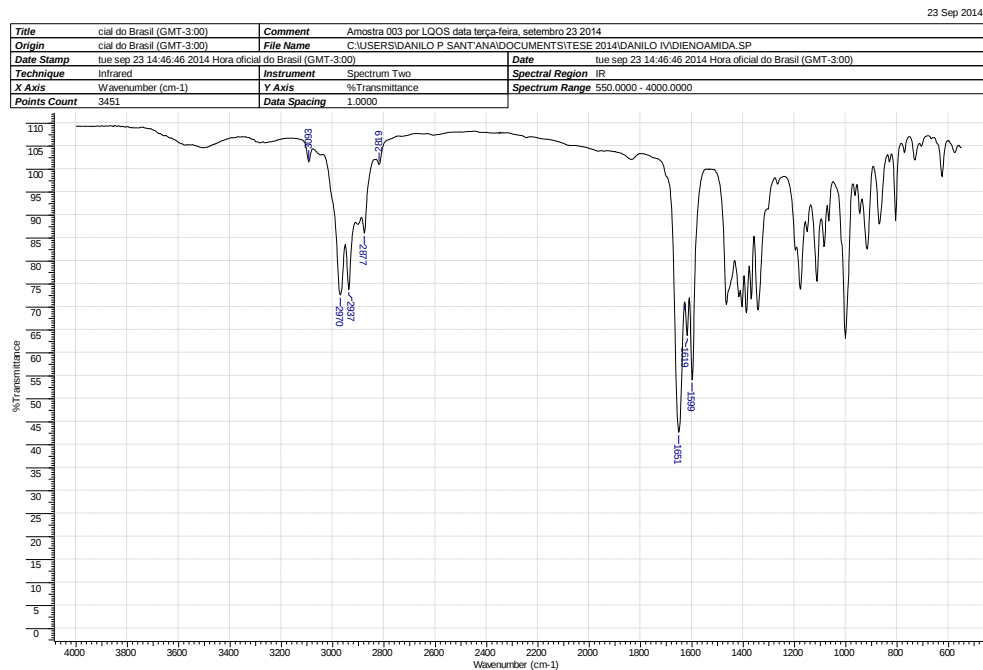
¹H NMR of **10** (CDCl₃, 250 MHz)



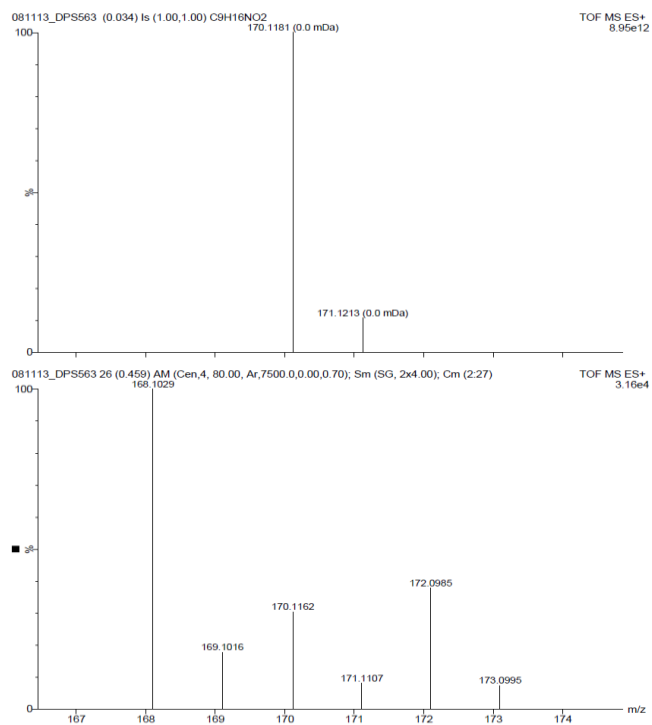
¹³C NMR of **10** (CDCl₃, 62.5 MHz)



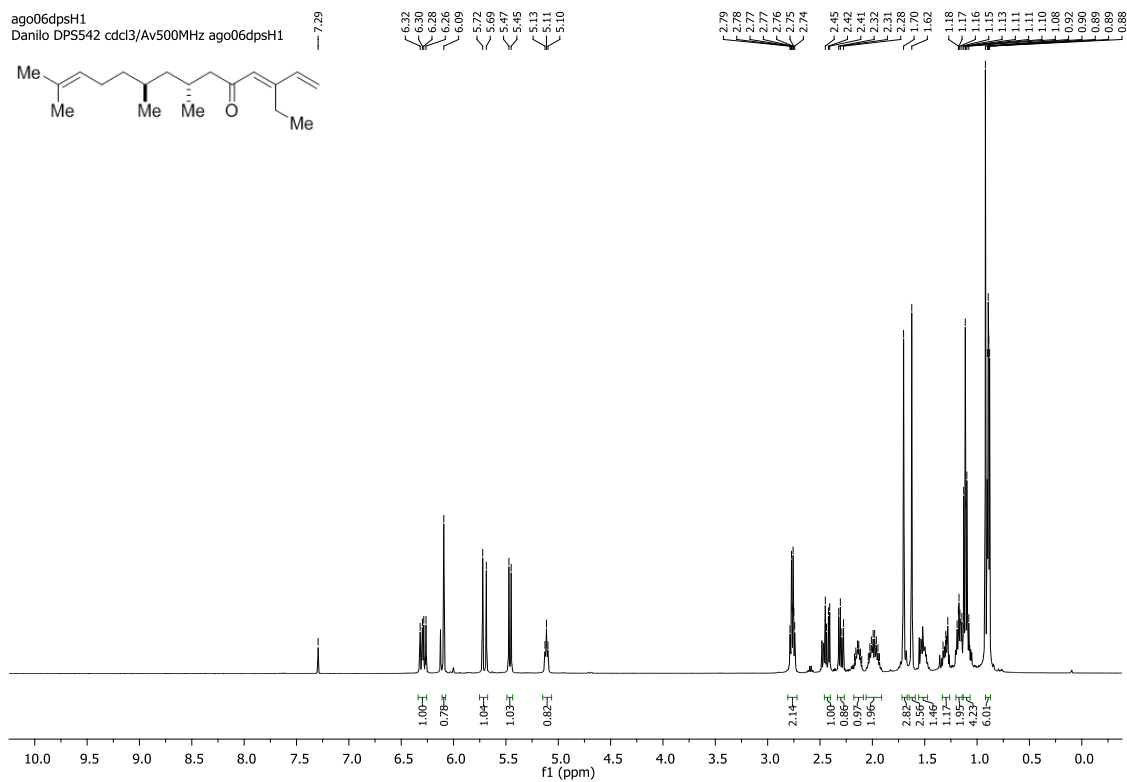
DEPT 135 of **10** (CDCl₃, 62.5 MHz)



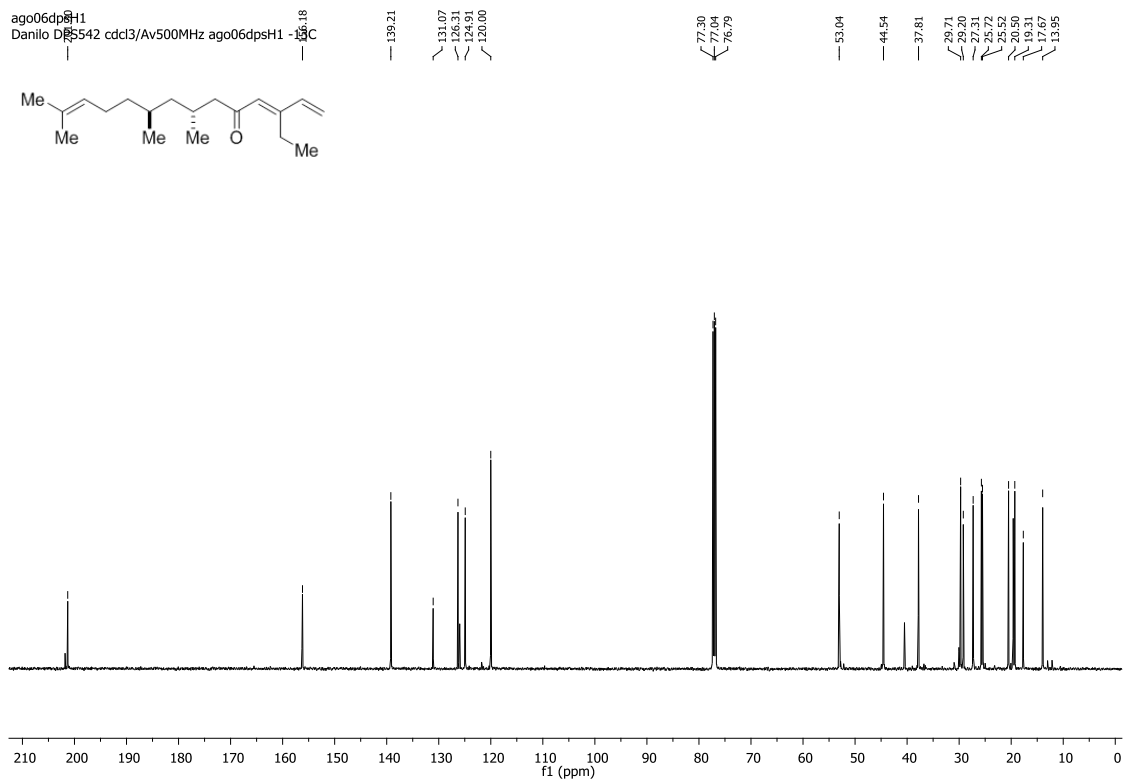
IR of **10**



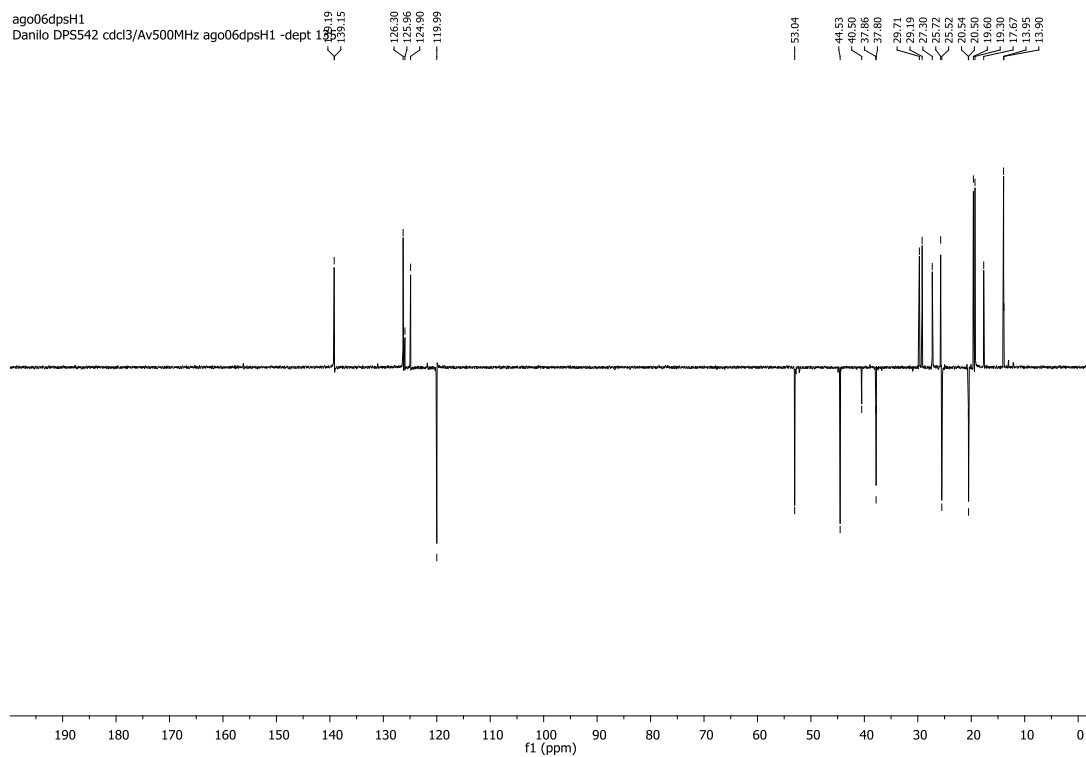
HRMS (ESI+) m/z calculated for C₉H₁₆NO₂ [M+H]⁺ 170.1181



¹H NMR of **22** (CDCl₃, 500 MHz)

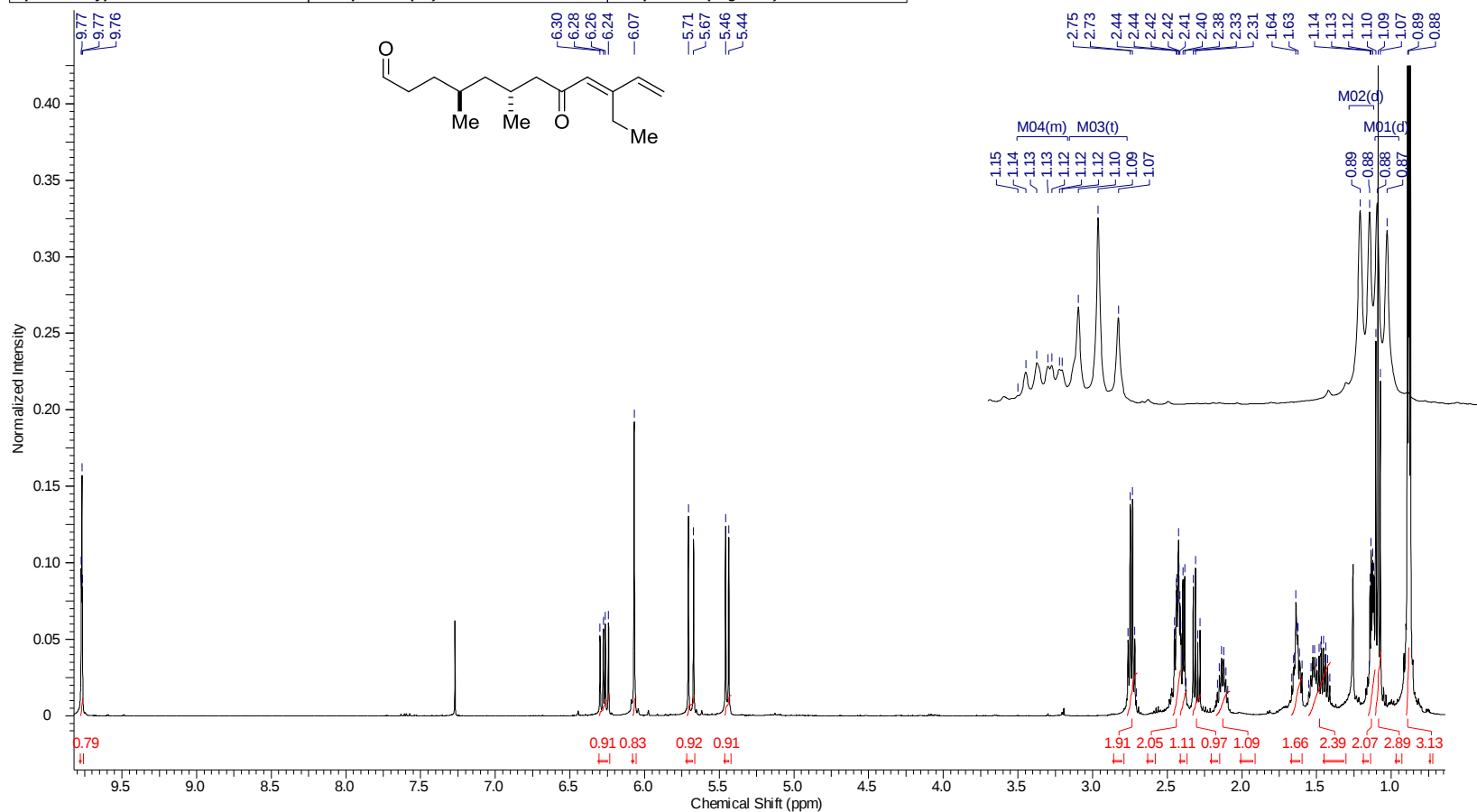


¹³C NMR of **22** (CDCl₃, 125 MHz)

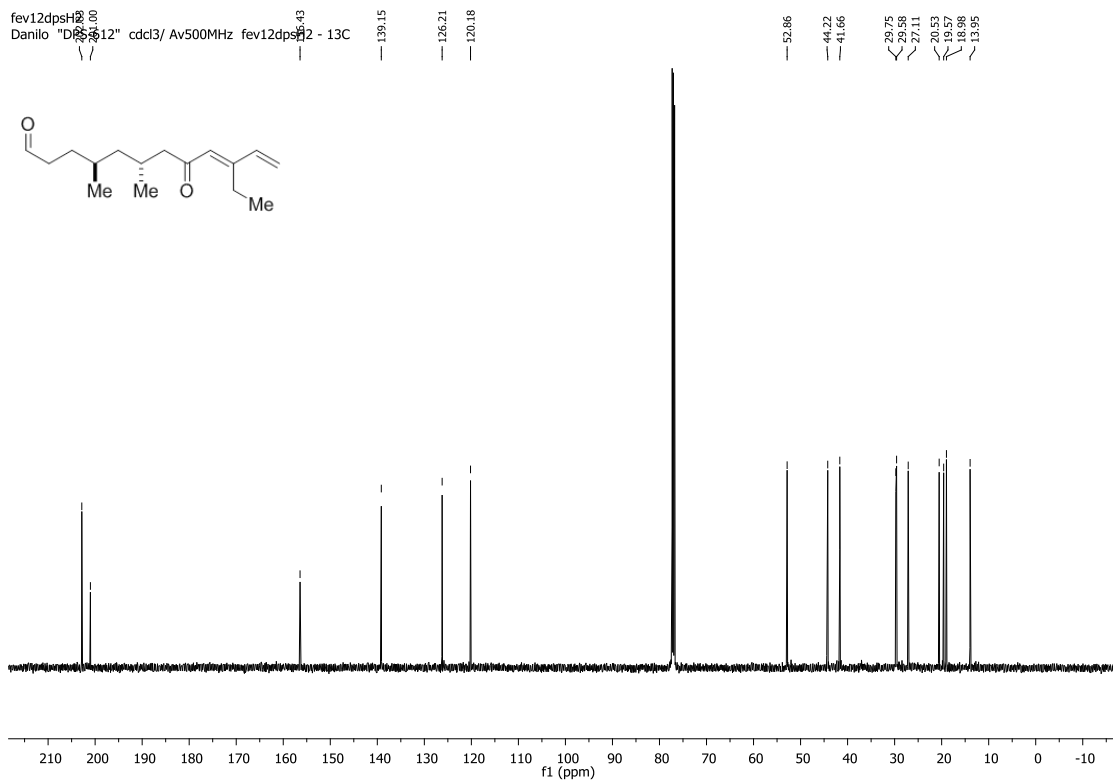


DEPT 135 of **22** (CDCl₃, 125 MHz)

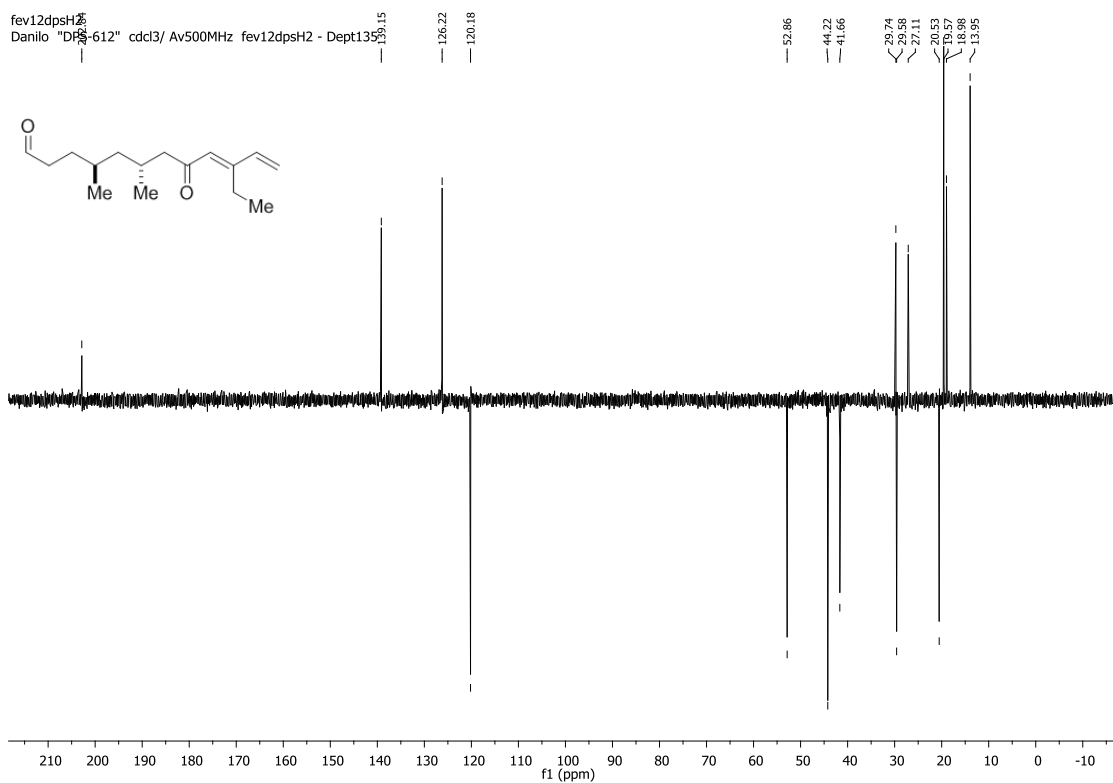
Acquisition Time (sec)	1.6384	Comment	Danilo "DPS-612" cdcl3/ Av500MHz fev12dpsH2	Date	12 Feb 2014 17:01:20
Date Stamp	12 Feb 2014 17:01:20		File Name	C:\Users\Danilo P Sant\Ana\Documents\tautomicetina\fid\dps600-\dps612\1\data\1\1r	
Frequency (MHz)	499.87	Nucleus	¹ H	Number of Transients	16
Original Points Count	16384	Owner	nmrsu	Points Count	65536
Receiver Gain	80.60	SW(cyclical) (Hz)	10000.00	Solvent	CDCl3
Spectrum Type	STANDARD	Sweep Width (Hz)	9999.85	Temperature (degree C)	25.142
				Pulse Sequence	zg30
				Spectrum Offset (Hz)	3074.8962



¹H NMR of fragment C1-C12 (CDCl₃, 500 MHz)

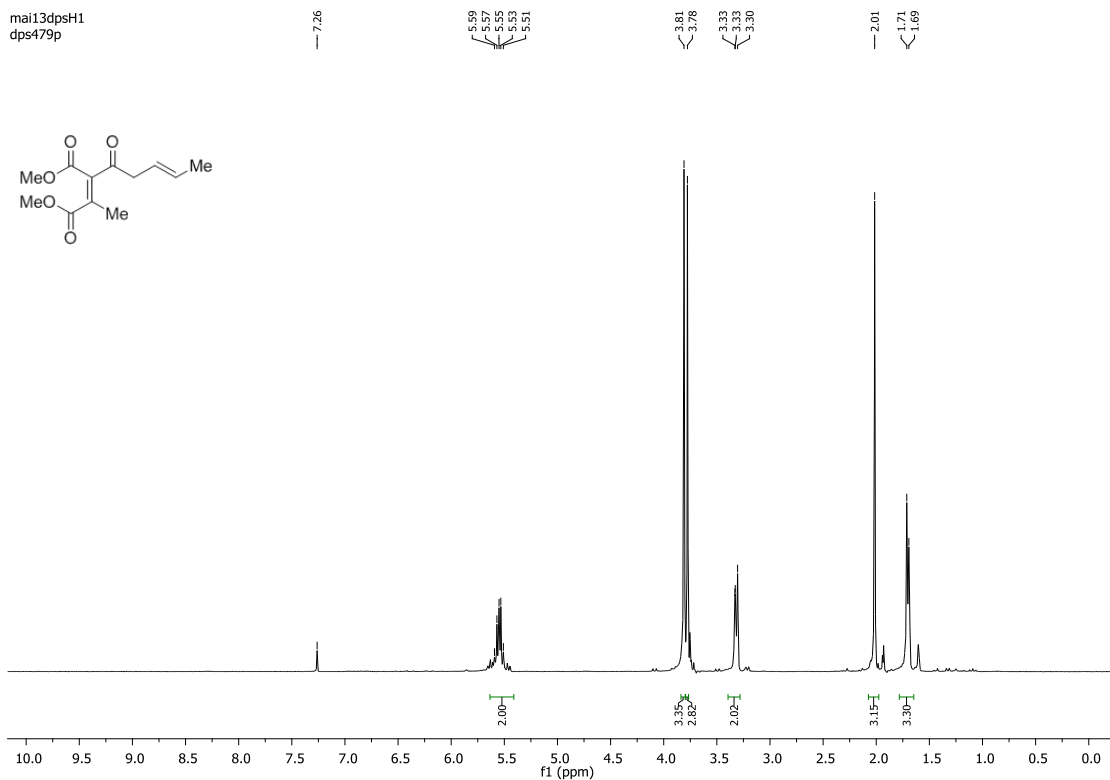


^{13}C NMR of fragment C1-C12 (CDCl_3 , 125 MHz)



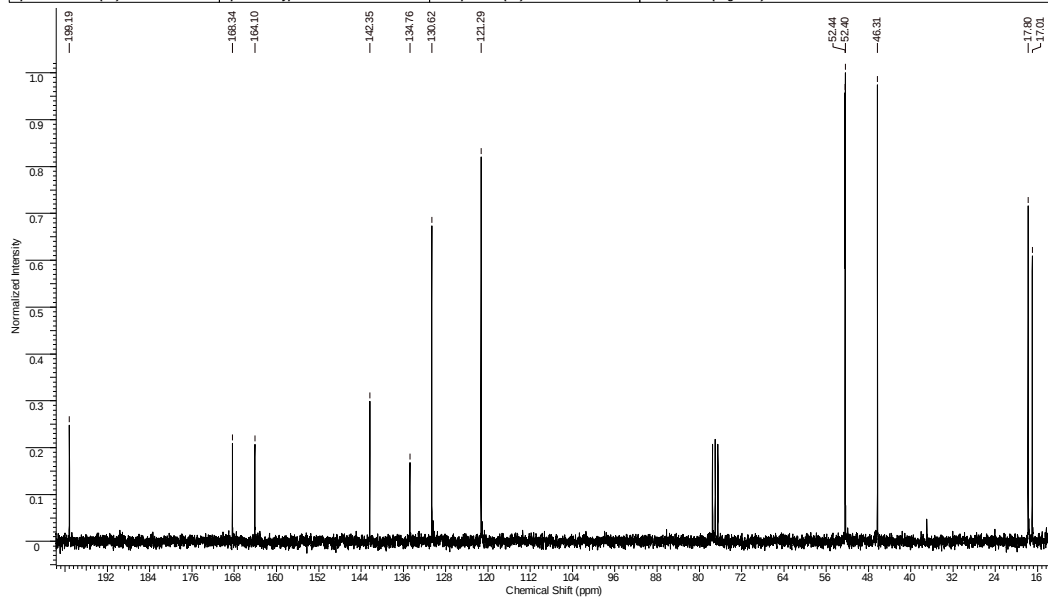
DEPT 135 of fragment C1-C12 (CDCl_3 , 125 MHz)

mai13dpsH1
dps479p

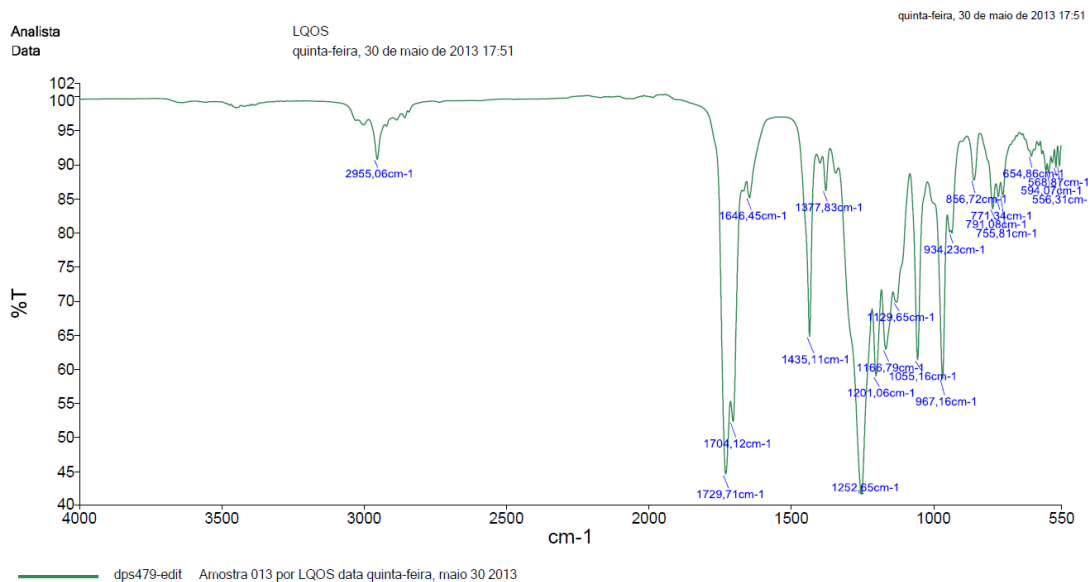


¹H NMR of **31** (CDCl₃, 250 MHz)

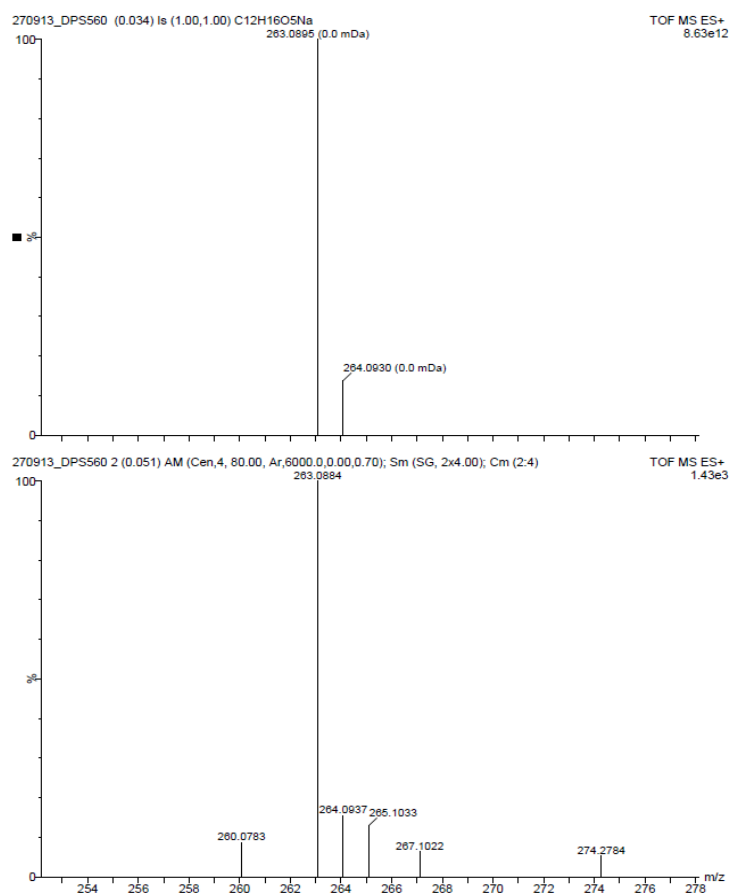
Acquisition Time (sec)	0.5439	Comment	dps479p 13C	Date	12 May 2013 17:52:16
Date Stamp	12 May 2013 17:52:16	Nucleus	13C	File Name	C:\Users\Danilo\Documents\stautomicetna\fid\dps401-499\dps479 C1\pdata1\1r
Frequency (MHz)	62.90	Owner	root	Number of Transients	153
Original Points Count	8192	SW(cyclical) (Hz)	15060.24	Points Count	32768
Receiver Gain	322.50	Spectrum Type	STANDARD	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	6282.6372			Sweep Width (Hz)	15059.78
				Temperature (degree C)	25.160



¹³C NMR of **31** (CDCl₃, 62.5 MHz)

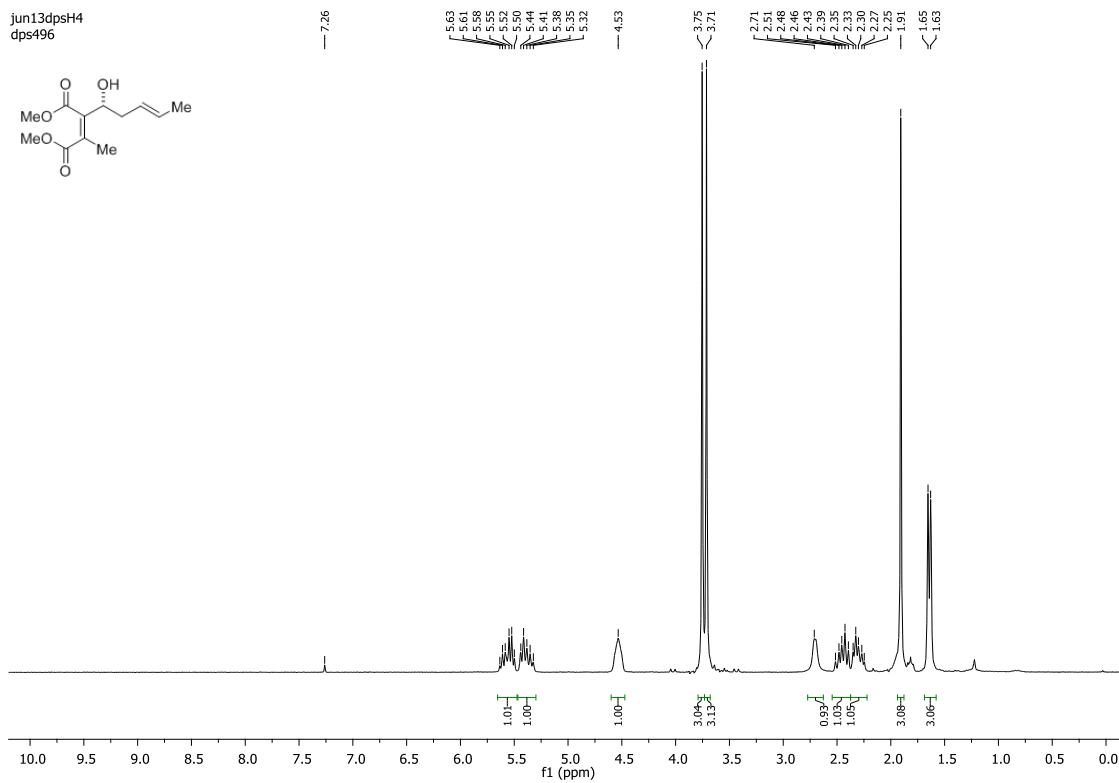
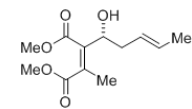


IR of 31



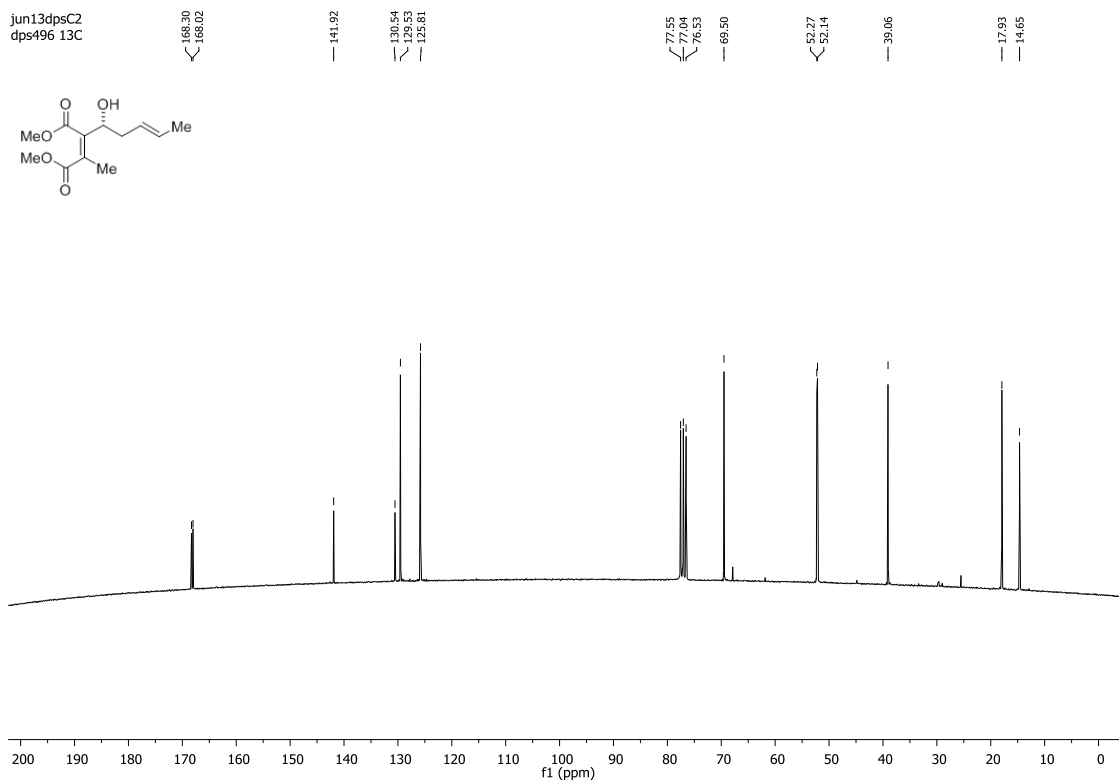
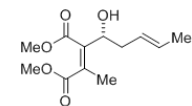
HRMS (ESI+) m/z calculated for C₁₂H₁₆O₅Na [M+Na]⁺ 263.0895

jun13dpsH4
dps496



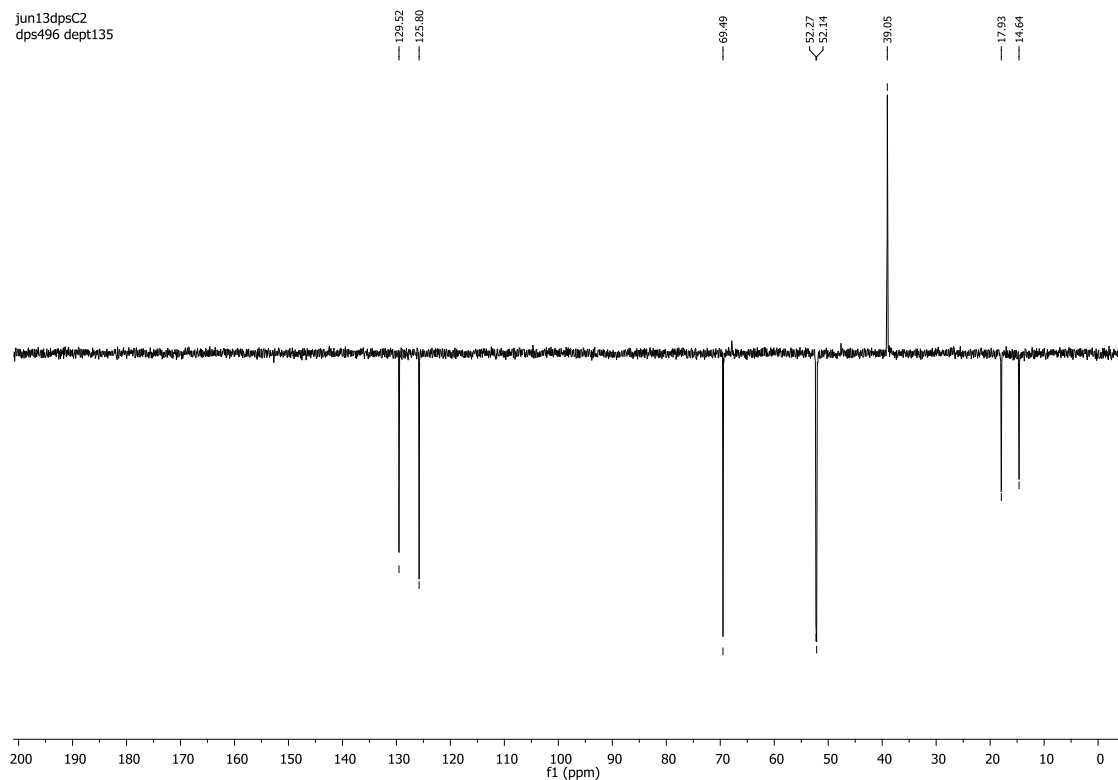
^1H NMR of **32** (CDCl_3 , 250 MHz)

jun13dpsC2
dps496 13C



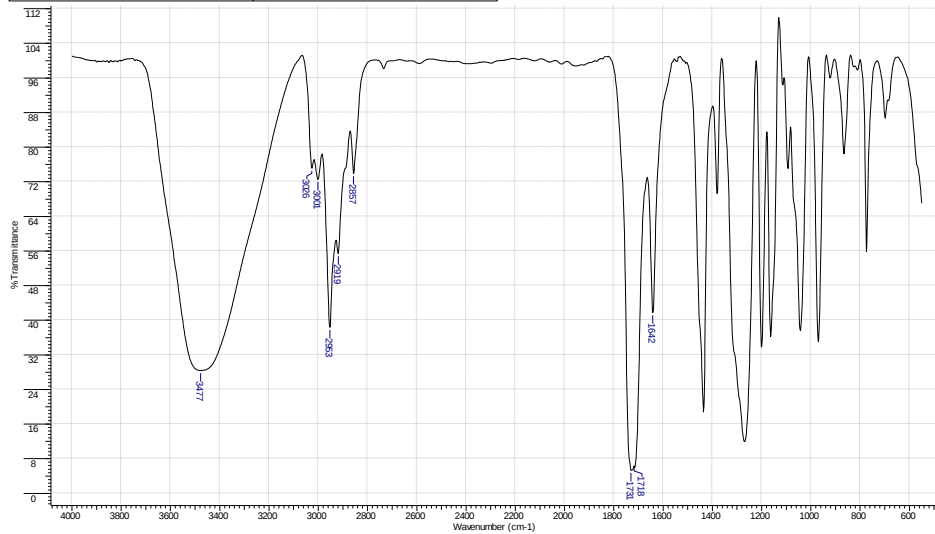
^{13}C NMR de **32** (CDCl_3 , 62.5 MHz)

jun13dpsC2
dps496 dept135

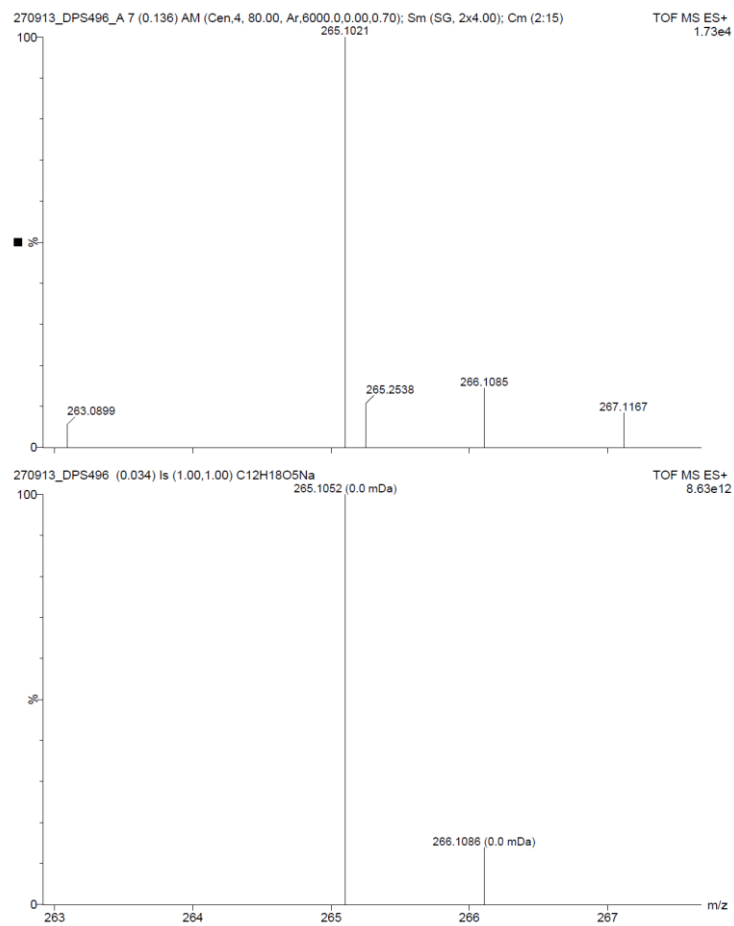


DEPT 135 of **32** (CDCl₃, 62.5 MHz)

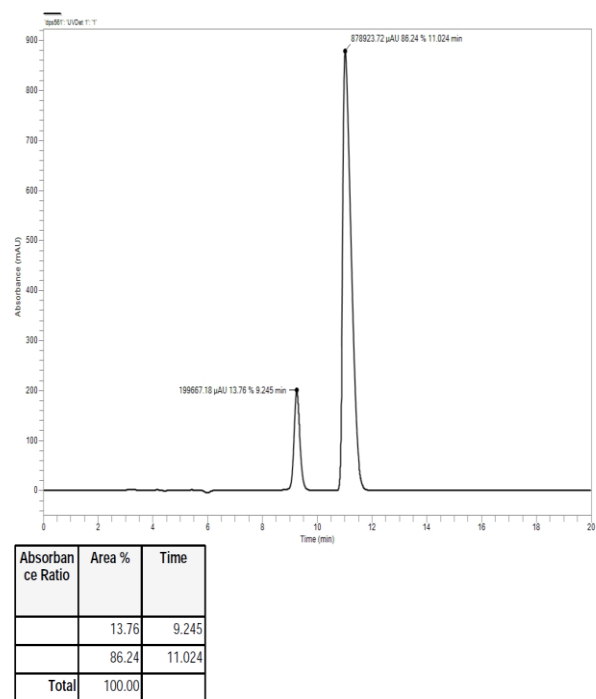
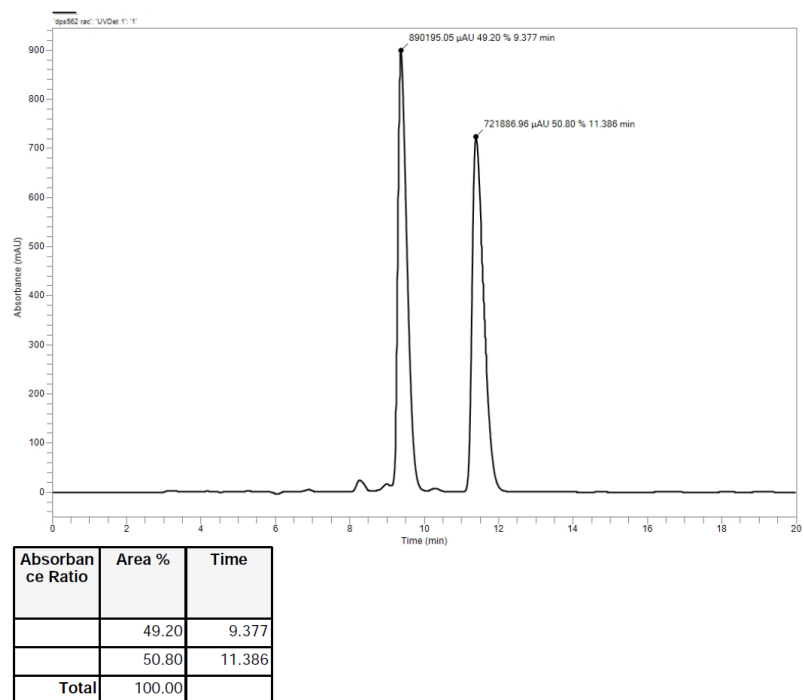
Title	brasileiro de verão (GMT-2:00)	Comment	Amostra 000 por LQOS data sexta-feira, dezembro 13 2013
Origin	brasileiro de verão (GMT-2:00)	File Name	C:\USERS\DANILO P SANT'ANA\DOCUMENTS\1AUTOMICETINA\VIDPS562_1.SP
Date Stamp	fri dec 13 15:32:33 2013 Horário brasileiro de verão (GMT-2:00)	Date	fri dec 13 15:34:37 2013 Horário brasileiro de verão (GMT-2:00)
Technique	Infrared	Instrument	Spectrum Two
X Axis	Wavenumber (cm-1)	Y Axis	%Transmittance
Points Count	3451	Data Spacing	1.0000
		Spectral Region	IR
		Spectrum Range	550.0000 - 4000.0000



IR of **32**

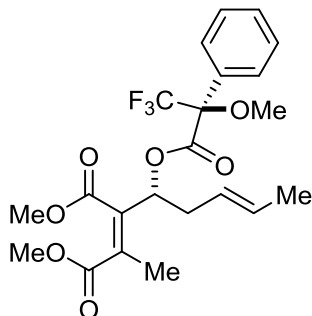


HRMS (ESI+) m/z calculated for C₁₂H₁₈O₅Na [M+Na]⁺ 265.1052



HPLC profile of **32** (Chiralpak IC[®], *n*hexane/*i*PrOH 8:2, 1.0 mL/min, 254 nm)

(S)-MTPA 32: (*R*)-(-)-MTPA-Cl (0.93 mL, 0.495 mmol), Et₃N (0.064 mL, 0.460 mmol) and



DMAP (1 mg) were added to a solution of **32** (16 mg, 0.066 mmol) in CH₂Cl₂ (3.3 mL). The reaction was stirred for 30 minutes After which the mixture was filtered through a plug of silica gel eluted with CH₂Cl₂.

The solvent was removed under reduced pressure to provide **(S)-**

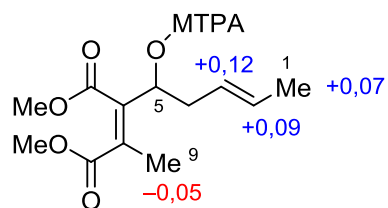
MTPA 32 (29 mg, 96%)

R_f = 0.27 (hexane:AcOEt 4:1). [α]_D²⁵ = -32 (c 0.7, CHCl₃). ¹H NMR

Major diastereomer (CDCl₃, 500 MHz) δ 7.53 (dd, *J* = 11.7, 9.6 Hz, 2H), 7.46 – 7.39 (m, 3H), 5.85 (dd, *J* = 9.1, 5.7 Hz, 1H), 5.70-5.63 (m, 1H), 5.45 – 5.36 (m, 1H), 3.79 (s, 3H), 3.60 (s, 3H), 3.57 (d, *J* = 0.9 Hz, 3H), 2.85 – 2.75 (m, 1H), 2.63 – 2.54 (m, 1H), 2.02 (s, 3H), 1.69 (d, *J* = 6.4 Hz, 3H). ¹³C{¹H} NMR **Major diastereomer (CDCl₃, 125 MHz)** δ 15.2, 18.0, 29.7, 36.1, 52.1, 54.5, 55.6, 73.7, 124.6, 127.4, 128.4, 129.6, 130.5, 132.1, 134.7, 136.2, 166.0, 166.5, 166.5, 168.0. **IR (film):** 3029, 3001, 2954, 2923, 2853, 1754, 1740, 1731 cm⁻¹. HRMS (ESI+) *m/z* calculated for C₂₂H₂₅F₃O₇Na [M+Na⁺] 481.1450, found 481.1412.

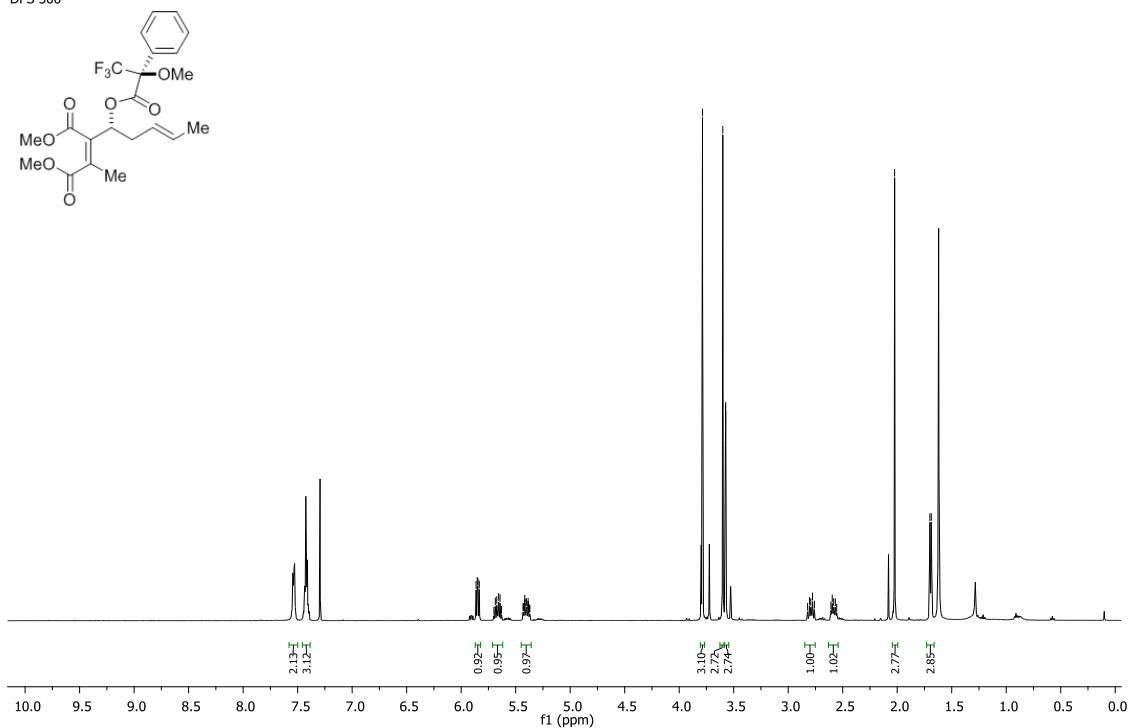
(R)-MTPA 32: (*S*)-(+)-MTPA-Cl (0.43 mL, 0.277 mmol), Et₃N (0.09 mL, 0.636 mmol) and DMAP (1 mg) were added to a solution of **32** (22 mg, 0.091 mmol) in CH₂Cl₂ (0.9 mL). The reaction was stirred for 30 minutes After which the mixture was filtered through a plug of silica gel eluted with CH₂Cl₂. The solvent was removed under reduced pressure to provide **(R)-**

MTPA 32 (30 mg, 72%)
R_f = 0.27 (hexane:AcOEt 80:20). [α]_D²⁵ = +23 (c 1.5, CHCl₃). ¹H NMR **Major diastereomer (CDCl₃, 500 MHz)** δ 7.58 – 7.49 (m, 2H), 7.49 – 7.41 (m, 3H), 5.96 – 5.87 (m, 1H), 5.60-5.55 (m, 1H), 5.34 – 5.22 (m, 1H), 3.80 (s, 3H), 3.72 (s, 3H), 3.53 (s, 3H), 2.73-2.67 (m, 1H), 2.57 – 2.46 (m, 1H), 2.08 (s, 3H), 1.62 (d, *J* = 6.4 Hz, 3H). ¹³C{¹H} NMR **Major diastereomer (CDCl₃, 125 MHz)** δ 167.81, 166.78, 165.95, 136.84, 133.95, 131.84, 130.30, 129.64, 128.38, 127.60, 124.27, 73.53, 55.38, 52.53, 52.22, 36.27, 29.72, 17.92, 15.18. **IR (film)** 3064, 3027, 2954, 2924, 2853, 1748, 1743, 1731, cm⁻¹. HRMS (ESI+) *m/z* calculated for C₂₂H₂₆F₃O₇ [M+H⁺] 459.1631, found 459.1638.



(S)-MTPA X			(R)-MTPA X			
Position	δ H (ppm)	Mult. <i>J</i>	Position	δ H (ppm)	Mult. <i>J</i>	$\Delta\delta^{SR}$
1	1.67	d (<i>J</i> 6.4)	1	1.60	d (<i>J</i> 6.4)	+ 0.07
2	5.64	dd (1H, <i>J</i> 15.2 e 6.5 Hz),	2	5.55	dd (1H, <i>J</i> 15.1 e 6.6 Hz)	+ 0.09
3	5.38	m	3	5.26	m	+ 0.12
9	2.01	s	9	2.06	s	– 0.05

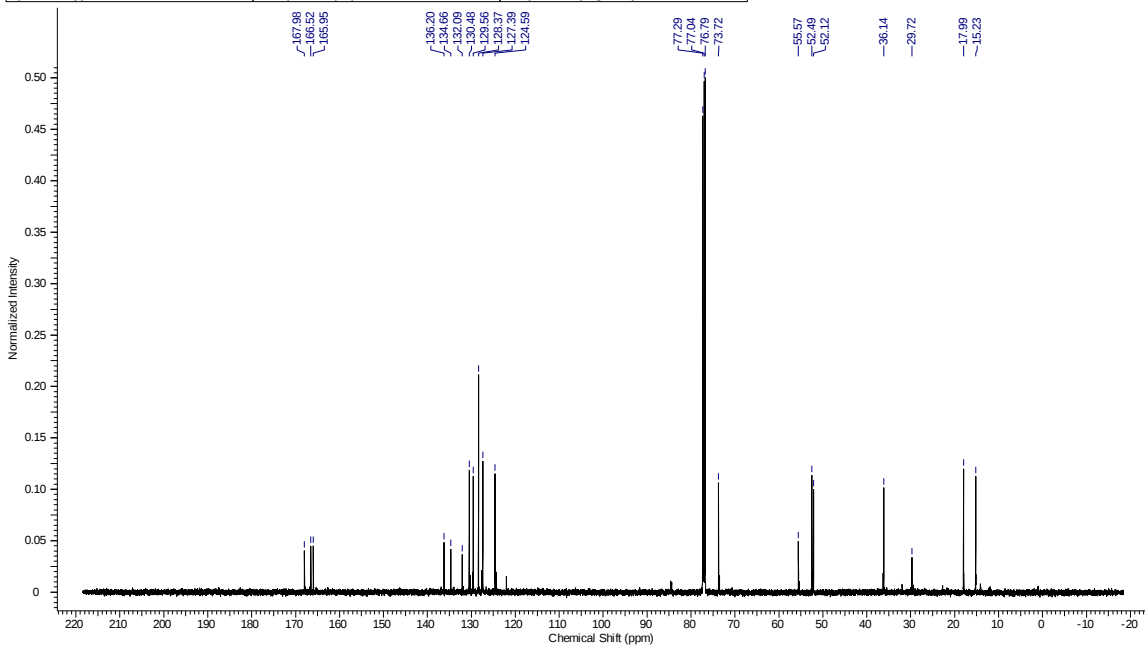
jun07dpsH2
jun07dpsH2 2013 500MHz
DPS 500



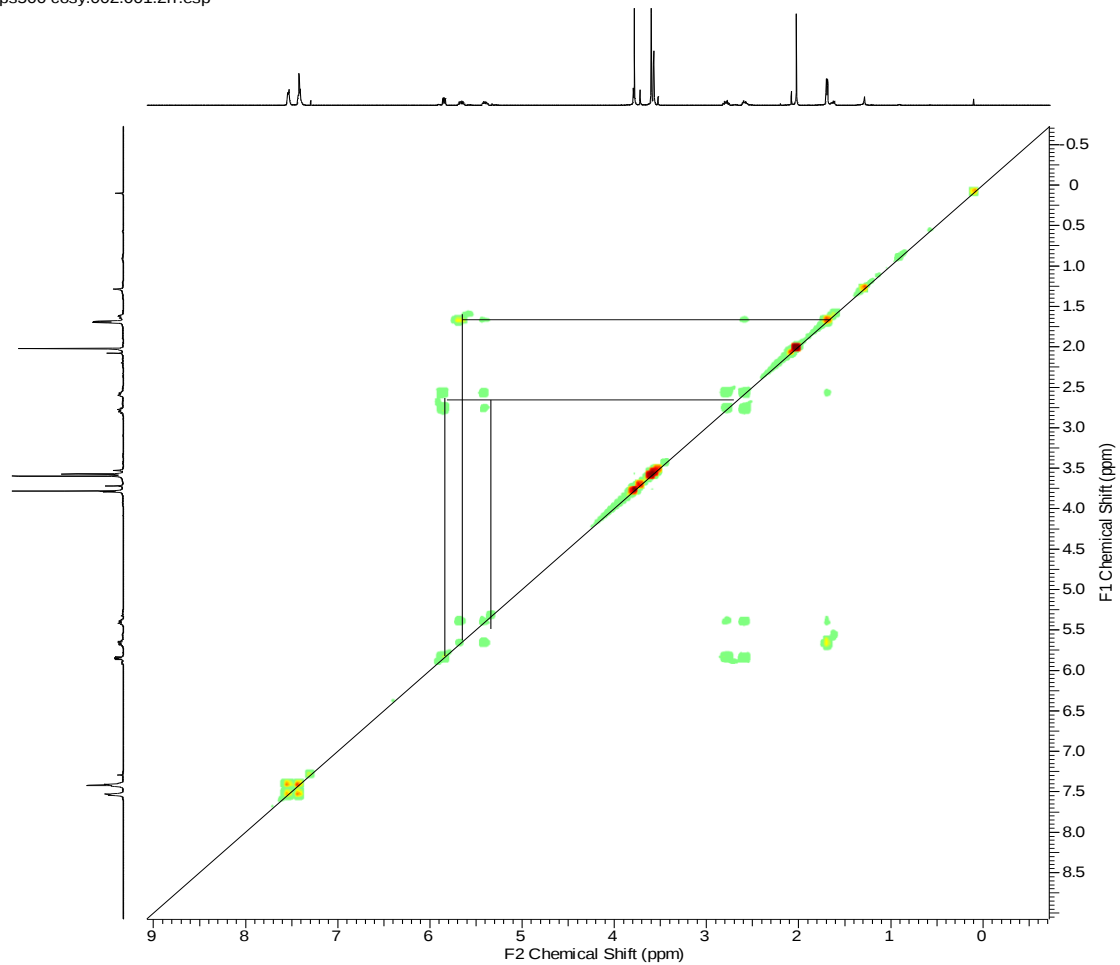
¹H NMR of (S)-MTPA 32 (CDCl₃, 500 MHz)

17/12/2013 17:13:11

Acquisition Time (sec)	0.5505	Comment	Danilo - DPS 500 - CDCl ₃ - AV 500MHz - dez12dpsC1	Date	12 Dec 2013 10:45:52
Date Stamp	12 Dec 2013 10:45:52	File Name	C:\Users\Danilo P. Sant'Ana\Documents\tautomicetinal\fid\dps500-ldps500 C\1\data\1\1r	Origin	spect
Frequency (MHz)	125.69	Nucleus	13C	Number of Transients	512
Original Points Count	16384	Owner	nmrsv	Points Count	32768
Receiver Gain	2050.00	SW(cyclical) (Hz)	29761.90	Solvent	CHLOROFORM-d
Spectrum Type	STANDARD	Sweep Width (Hz)	29761.00	Temperature (degree C)	25.154
				Spectrum Offset (Hz)	12569.1270

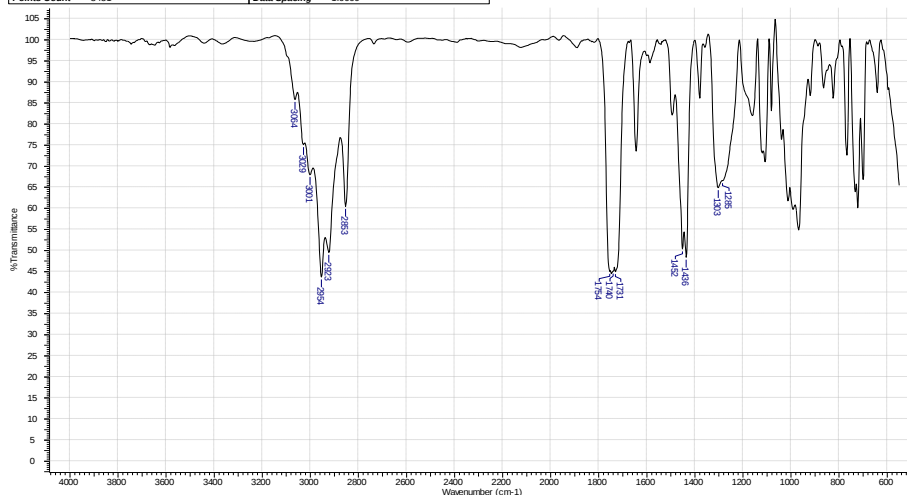


¹³C NMR of (S)-MTPA 32 (CDCl₃, 125 MHz)

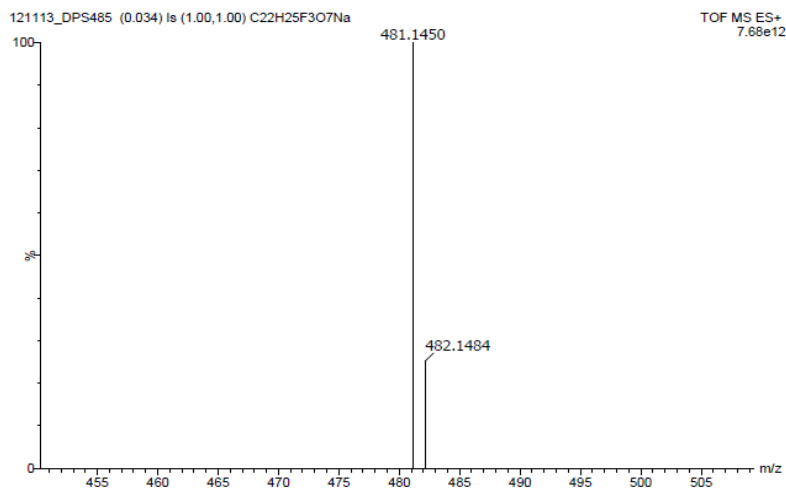
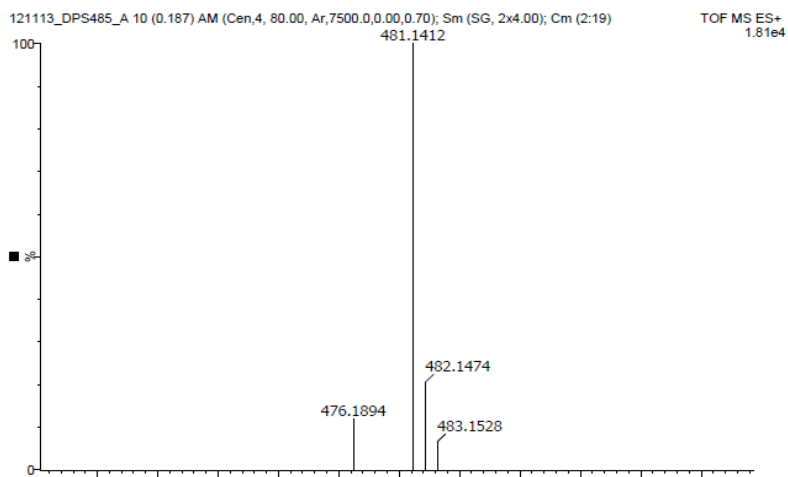


COSY of (S)-MTPA 32

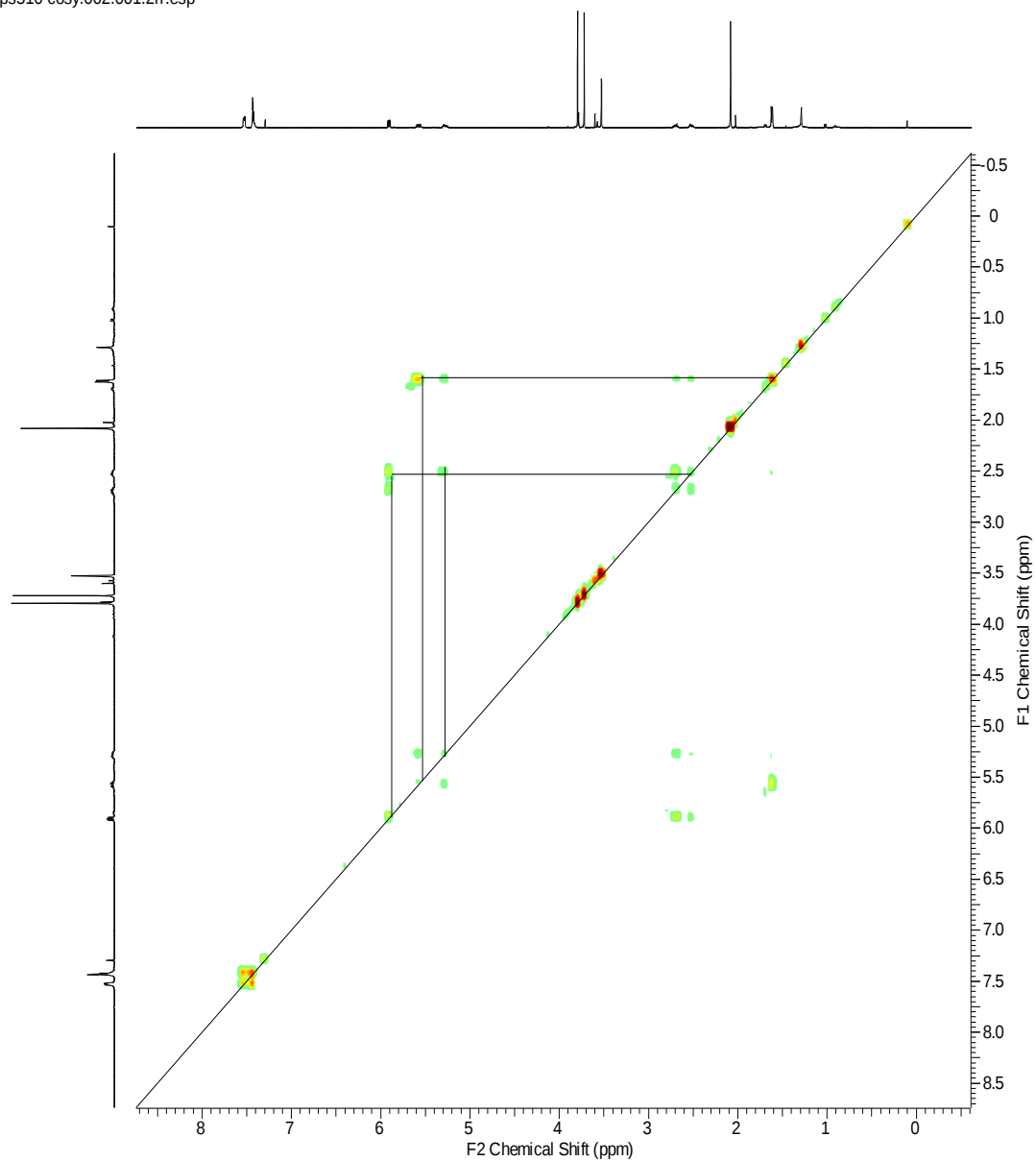
Title	brasileiro de verão (GMT-2:00)	Comment	Amostra 004 por LQOS data quarta-feira, dezembro 11 2013
Origin	brasileiro de verão (GMT-2:00)	File Name	C:\USERS\DAULO P SANTANA\DOCUMENTS\15AUTOMICETINA\VIDPS500 (2).SP
Date Stamp	wed dec 11 18:11:04 2013 Horário brasileiro de verão (GMT-2:00)	Date	wed dec 11 18:13:25 2013 Horário brasileiro de verão (GMT-2:00)
Technique	Infrared	Instrument	Spectrum Two
X Axis	Wavenumber (cm-1)	Y Axis	%Transmittance
Points Count	3451	Data Spacing	1.0000
		Spectral Region	IR
		Spectrum Range	550.0000 - 4000.0000



IR of (S)-MTPA 32

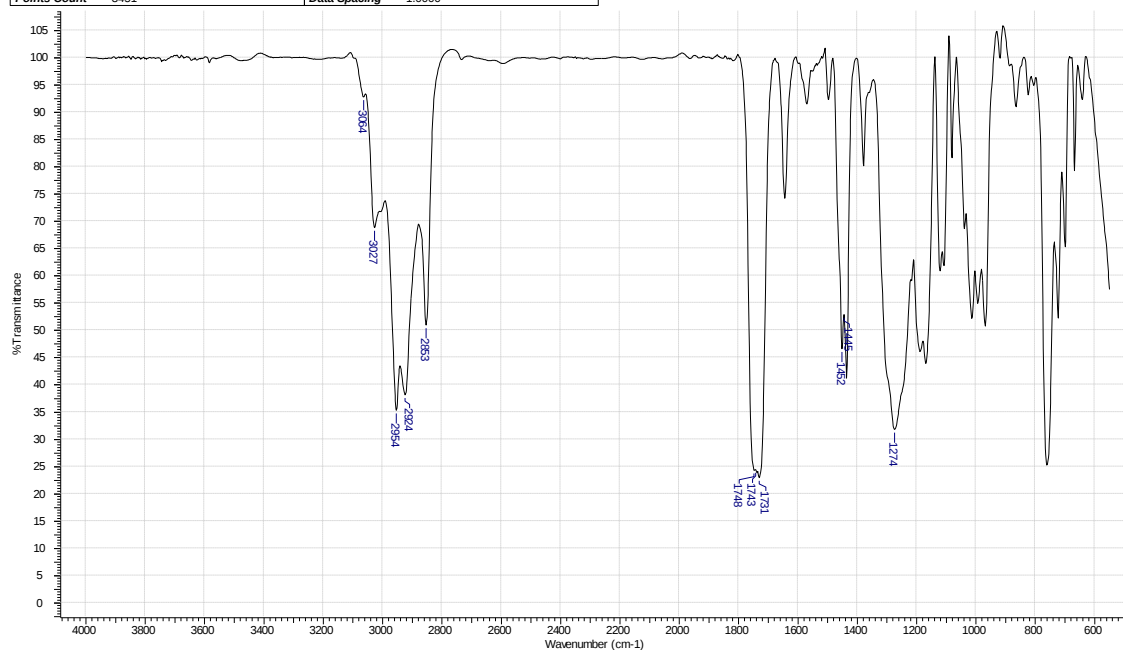


HRMS (ESI+) m/z calculated for $C_{22}H_{25}F_3O_7Na$ $[M+Na]^+$ 481.1450

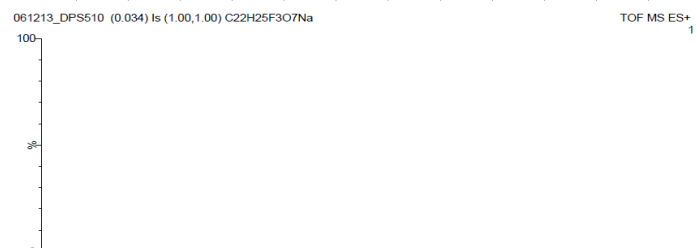
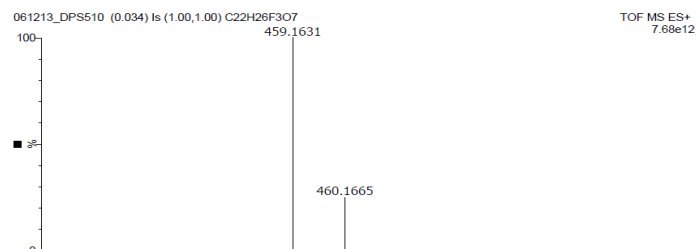


COSY of (*R*)-MTPA 32

Title	brasileiro de verão (GMT-2:00)	Comment	Amostra 001 por LQOS data sexta-feira, dezembro 13 2013
Origin	brasileiro de verão (GMT-2:00)	File Name	C:\USERS\IDANILO P SANT'ANA\DOCUMENTS\TAUTOMICETINA\IVDPSS10_1.SP
Date Stamp	fri dec 13 15:43:35 2013 Horário brasileiro de verão (GMT-2:00)	Date	fri dec 13 15:45:52 2013 Horário brasileiro de verão (GMT-2:00)
Technique	Infrared	Instrument	Spectrum Two
X Axis	Wavenumber (cm-1)	Y Axis	%Transmittance
Points Count	3451	Data Spacing	1.0000
		Spectral Region	IR
		Spectrum Range	550.0000 - 4000.0000

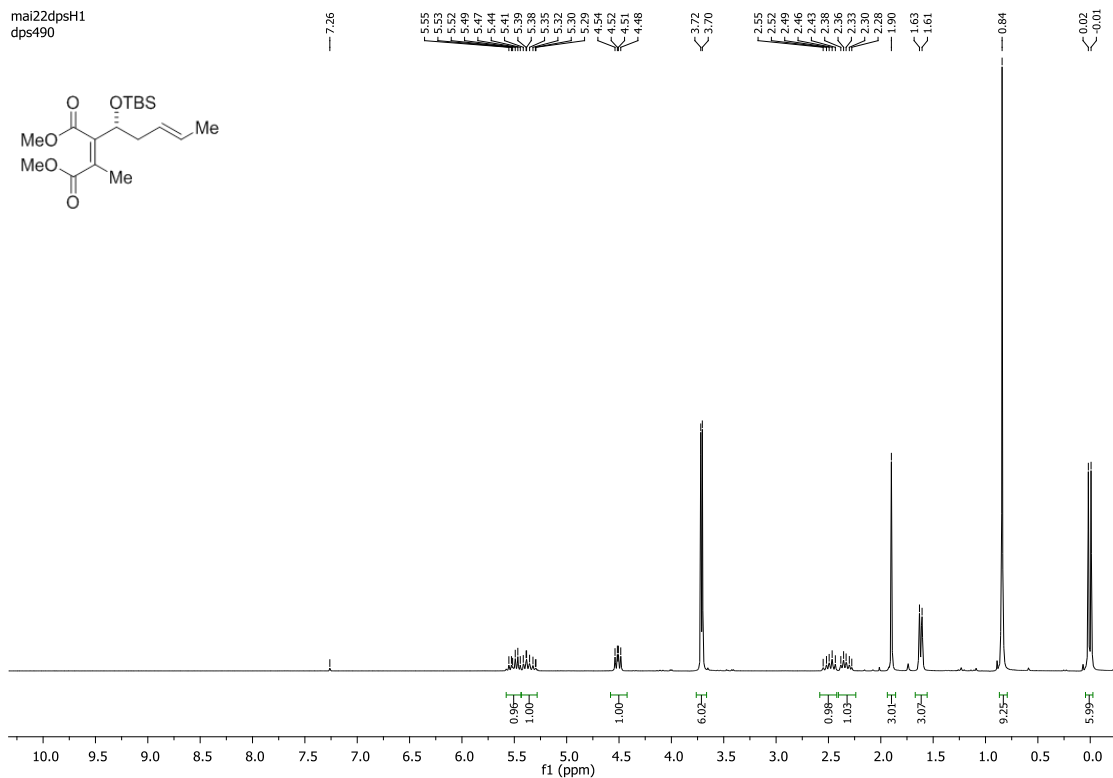
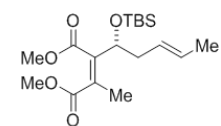


IR of (R)-MTPA 32



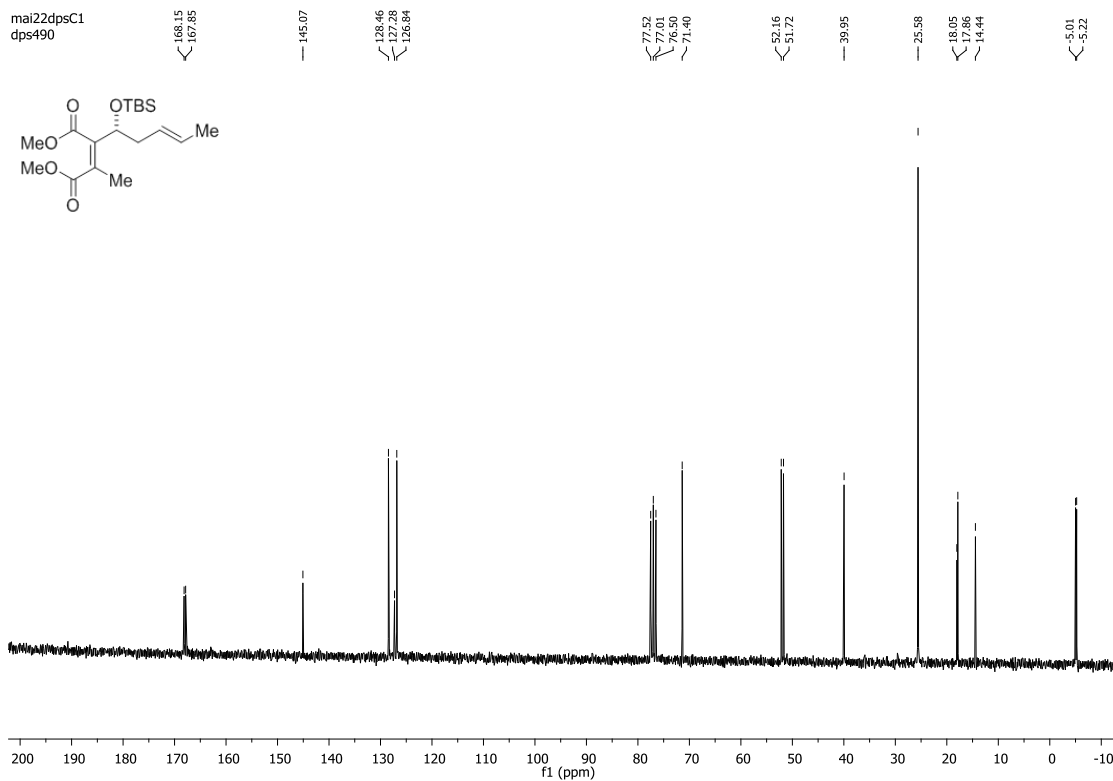
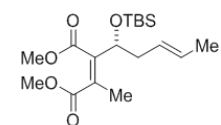
HRMS (ESI+) m/z calculated for $C_{22}H_{26}F_3O_7$ $[M+H]^+$ 459.1631

mai22dpsH1
dps490



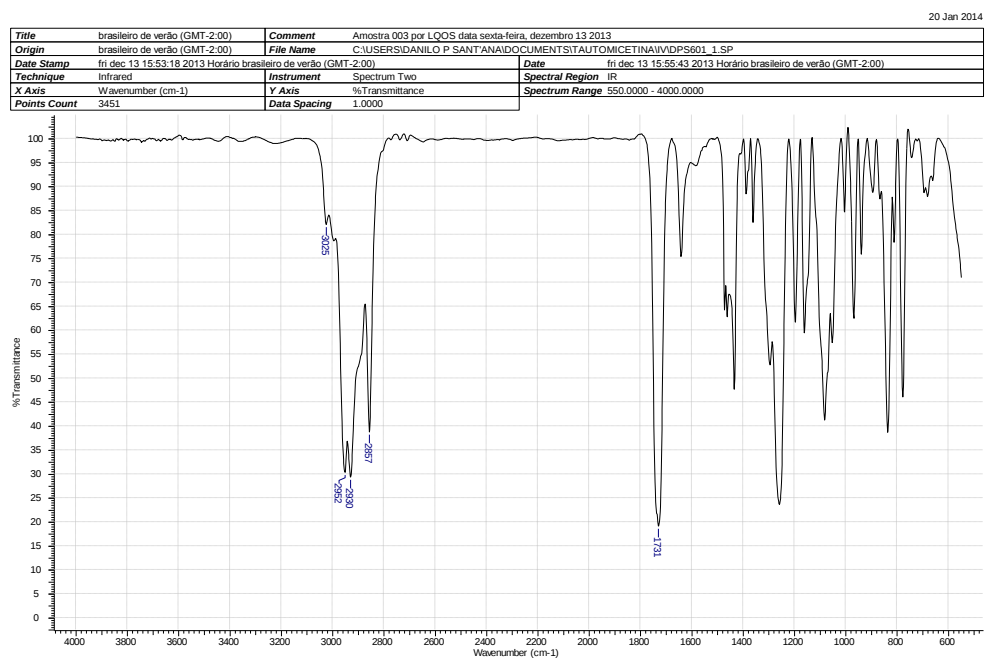
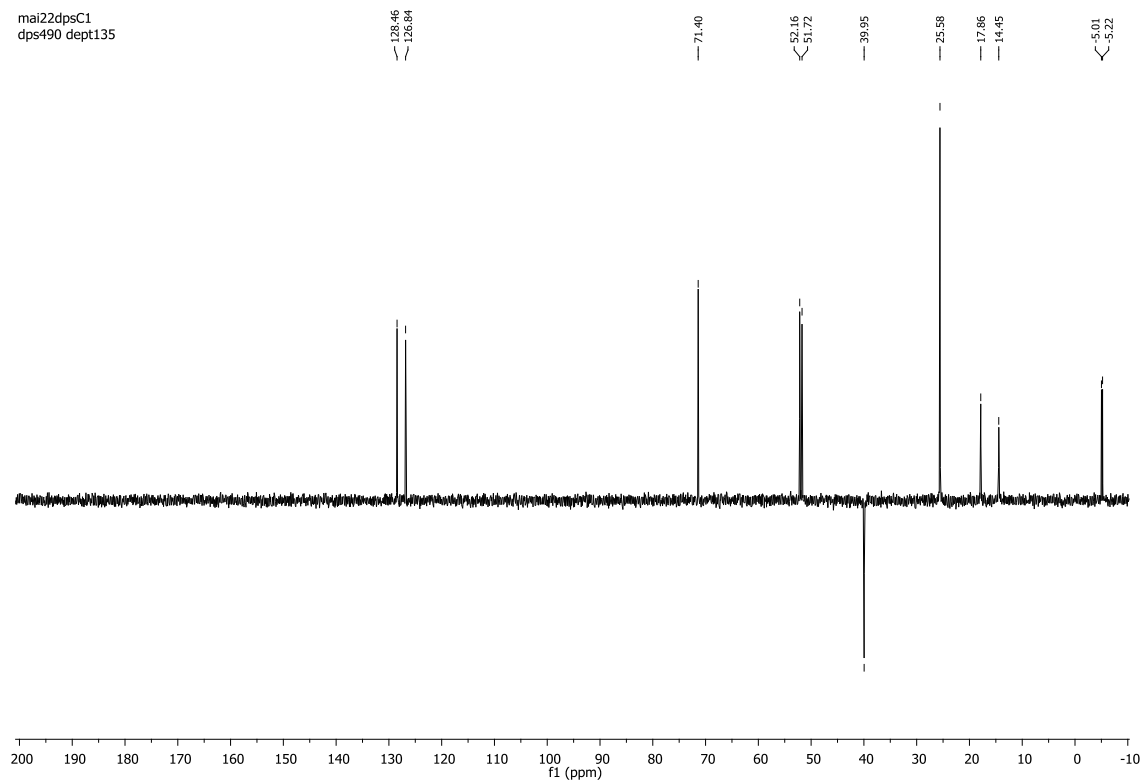
^1H NMR of **33** (CDCl_3 , 250 MHz)

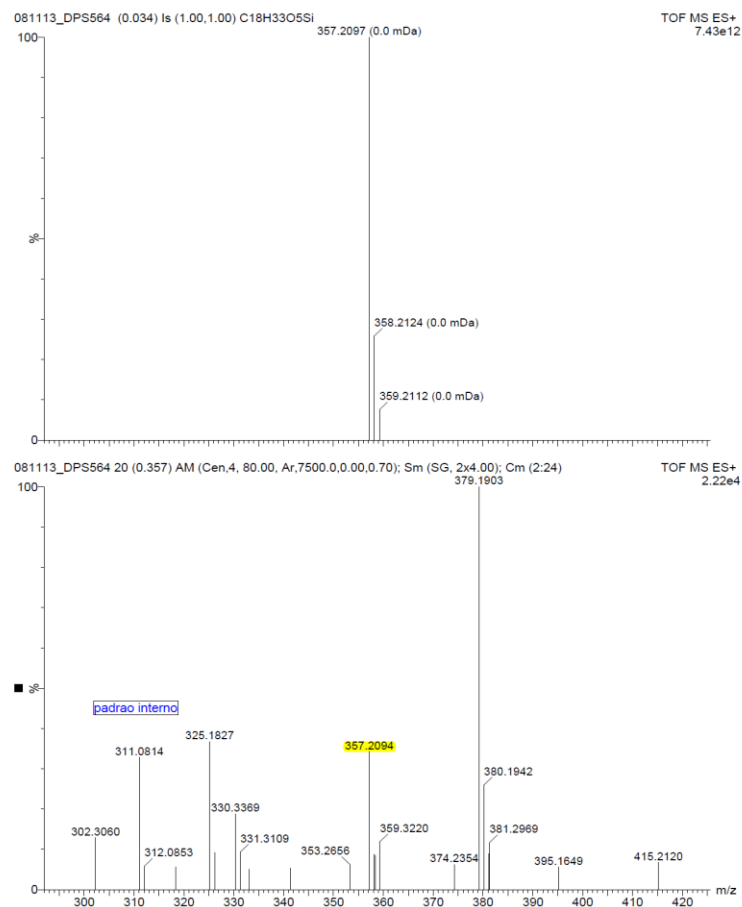
mai22dpsC1
dps490



^{13}C NMR **33** (CDCl_3 , 62.5 MHz)

mai22dpsC1
dps490 dept135





HRMS (ESI+) m/z calculated for C₁₈H₃₃O₅Si [M+H]⁺ 357.2097

Chemical structure of the compound is shown, with R¹ = Me, R² = SEM and R¹ = SEM, R² = Me. The structure features a substituted cyclohexene ring with an OTBS group, a methyl group, and a side chain containing a double bond and a methyl group.

¹H NMR spectrum (400 MHz, CDCl₃) showing peaks from 0.00 to 7.26 ppm. Integration values are provided below the peaks.

Chemical Shift (ppm)	Integration
7.26	—
5.57, 5.51, 5.49, 5.33	4.12
4.57, 4.55, 4.54, 4.53, 4.52, 4.51	1.00
3.75, 3.73, 3.72, 3.72, 3.68	5.06
2.57, 2.54, 2.51, 2.49, 2.45, 2.40, 2.38, 2.35, 2.32, 2.30	0.95, 1.02
1.93	3.08
1.65, 1.63	3.46
1.01, 0.99, 0.94, 0.93, 0.86	2.12, 9.22
0.04, 0.02	15.22

dez02dpsH1
Danilo DPS 595 - CDCl₃ - 500 MHz - dez02dpsH1 - 14

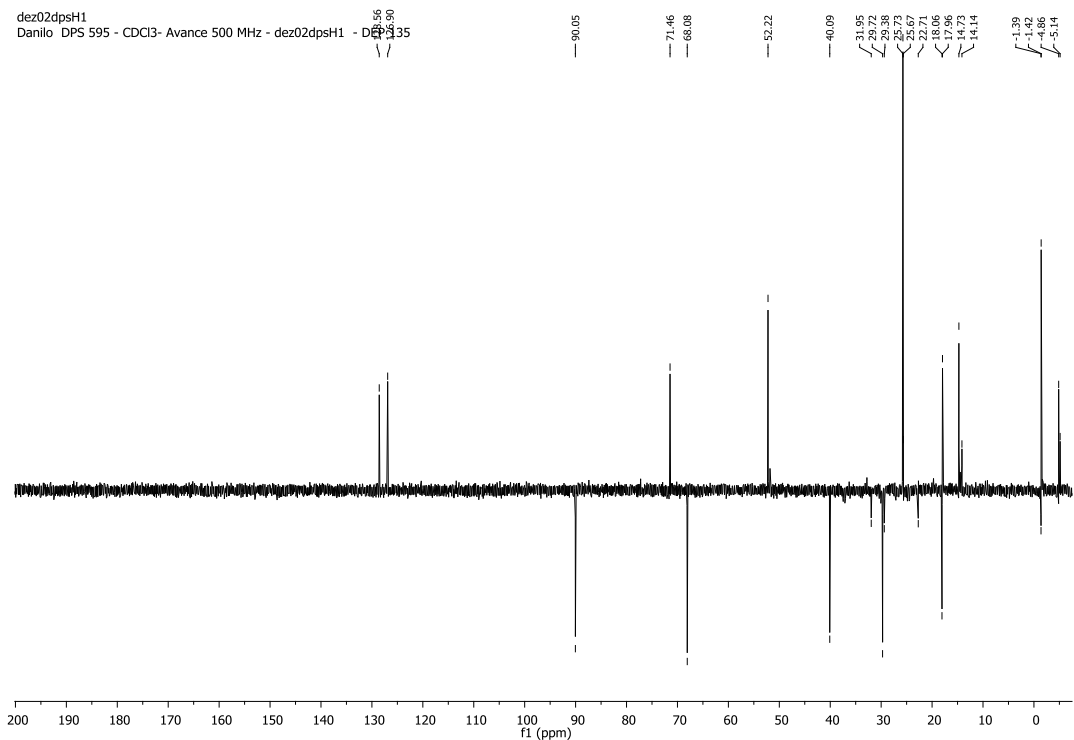
Chemical structure: CC(=C(C(=O)OR1)C(=O)OR2)C(=O)O[Si](C)(C)C/C=C/C

R¹: Me; R²: SEM
R¹: SEM; R²: Me

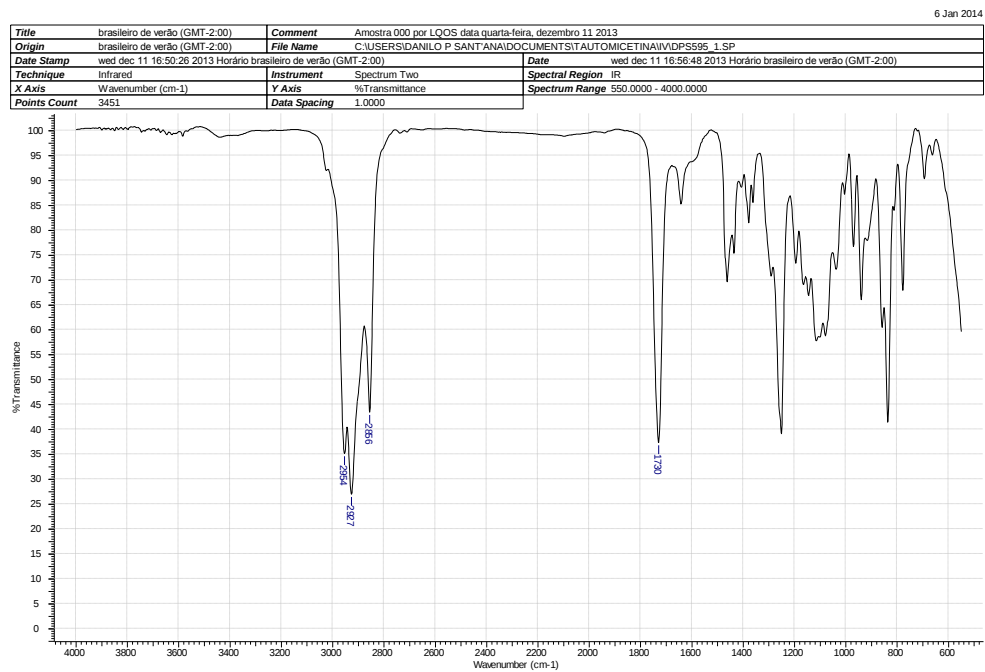
142.05, 137.27, 136.36, 135.56, 126.90, 90.05, 77.28, 77.03, 76.78, 71.45, 68.08, 52.22, 40.09, 31.95, 29.72, 25.73, 18.06, 17.96, 14.74, -1.42, -0.65, -5.14

f1 (ppm)

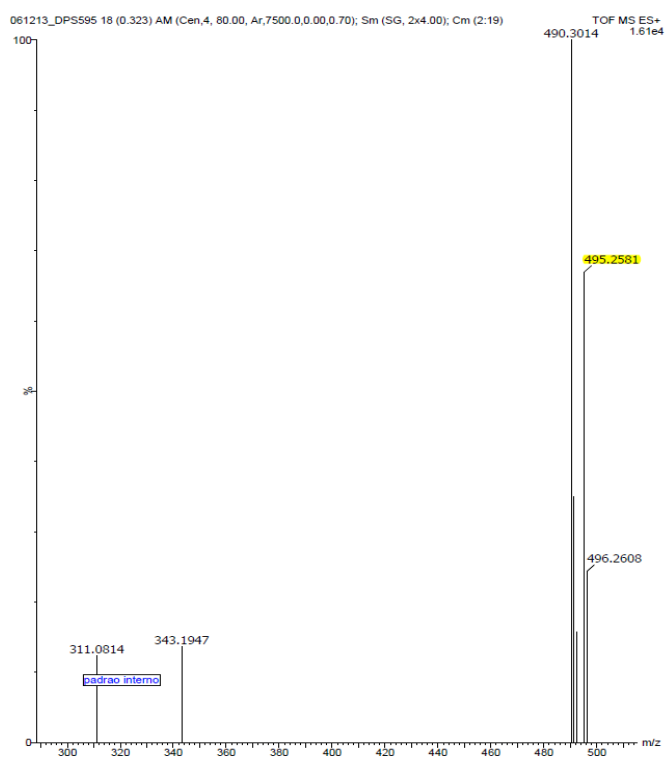
S42



DEPT 135 of **34** (CDCl₃, 100 MHz)

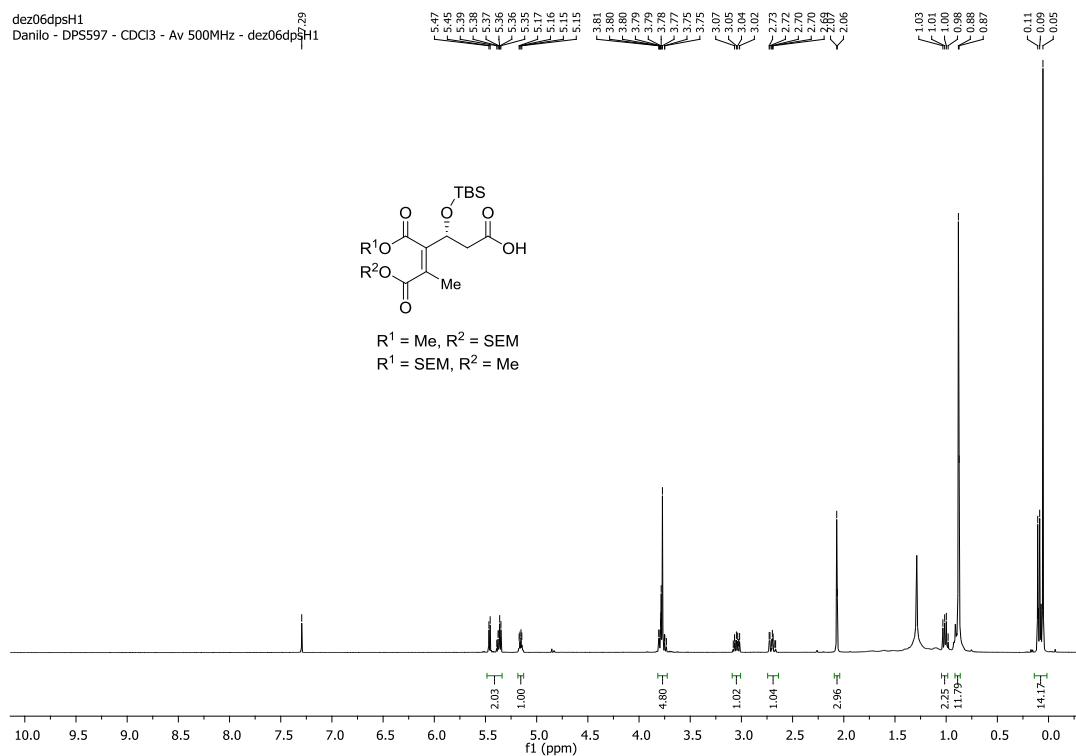


IR of **34**

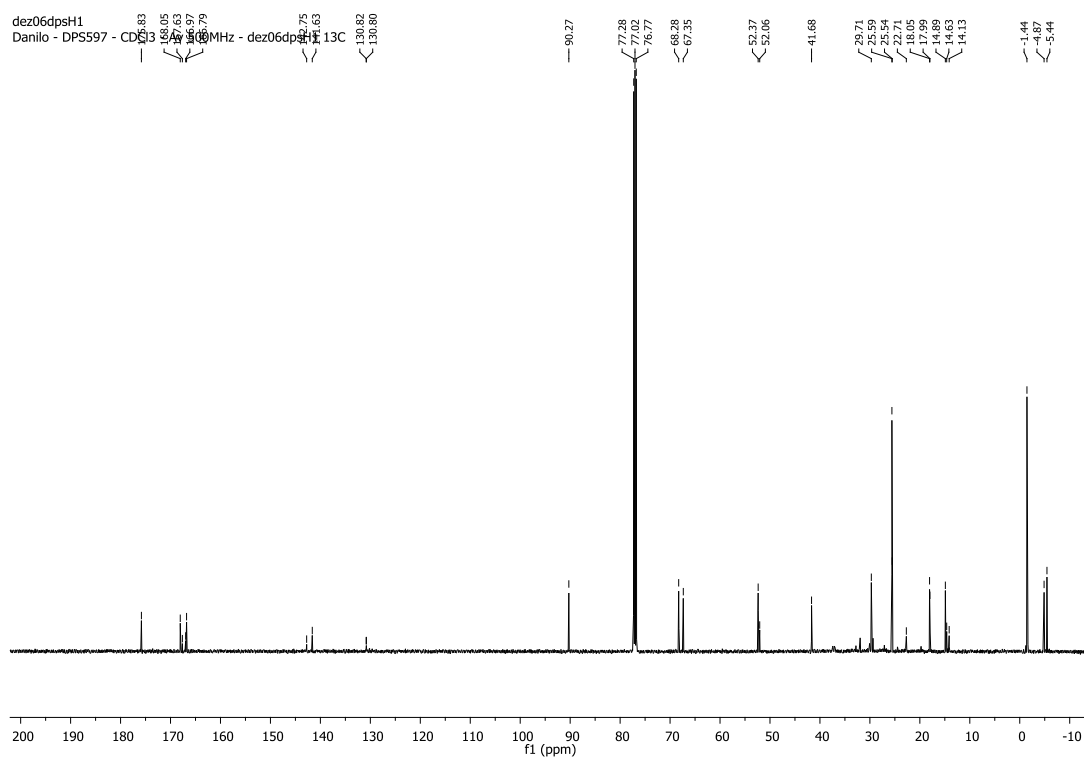


HRMS (ESI+) m/z calculated for $C_{23}H_{44}NaO_6Si_2 [M+Na]^+$ 495.2574

dez06dpsH1
 Danilo - DPS597 - CDCl3 - Av 500MHz - dez06dpsH1

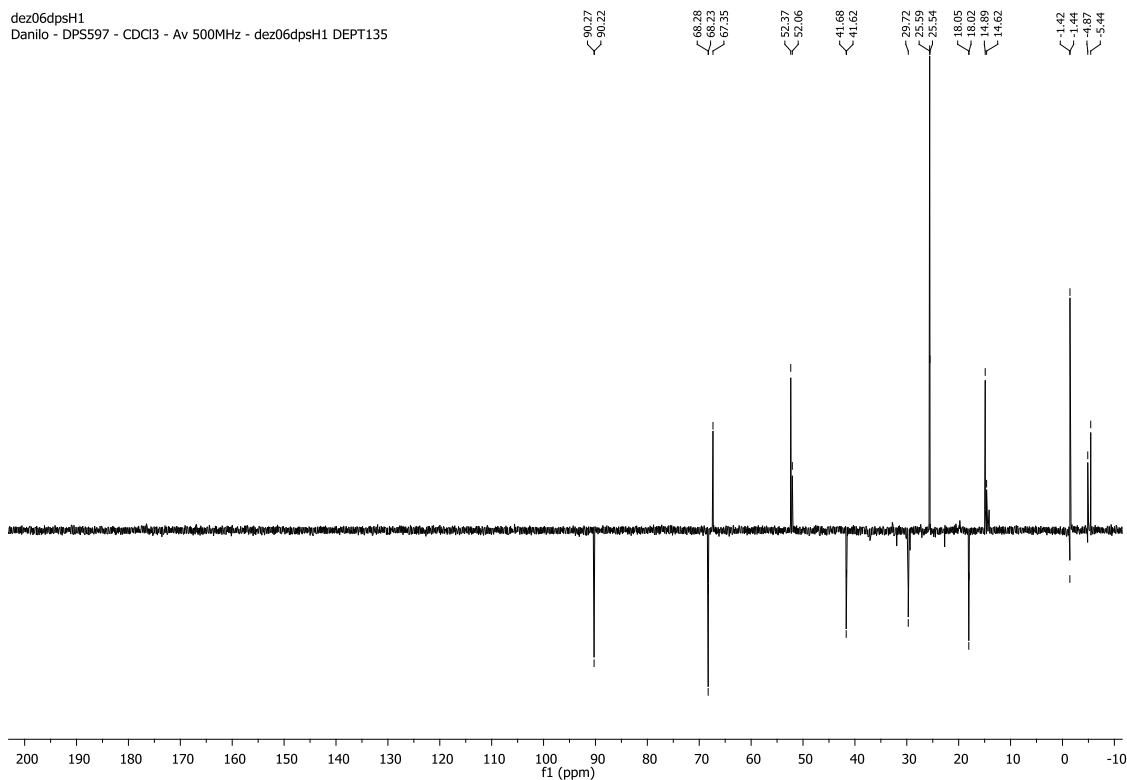


¹H NMR de **11** (CDCl₃, 250 MHz)



¹³C NMR of **11**(CDCl₃, 100 MHz)

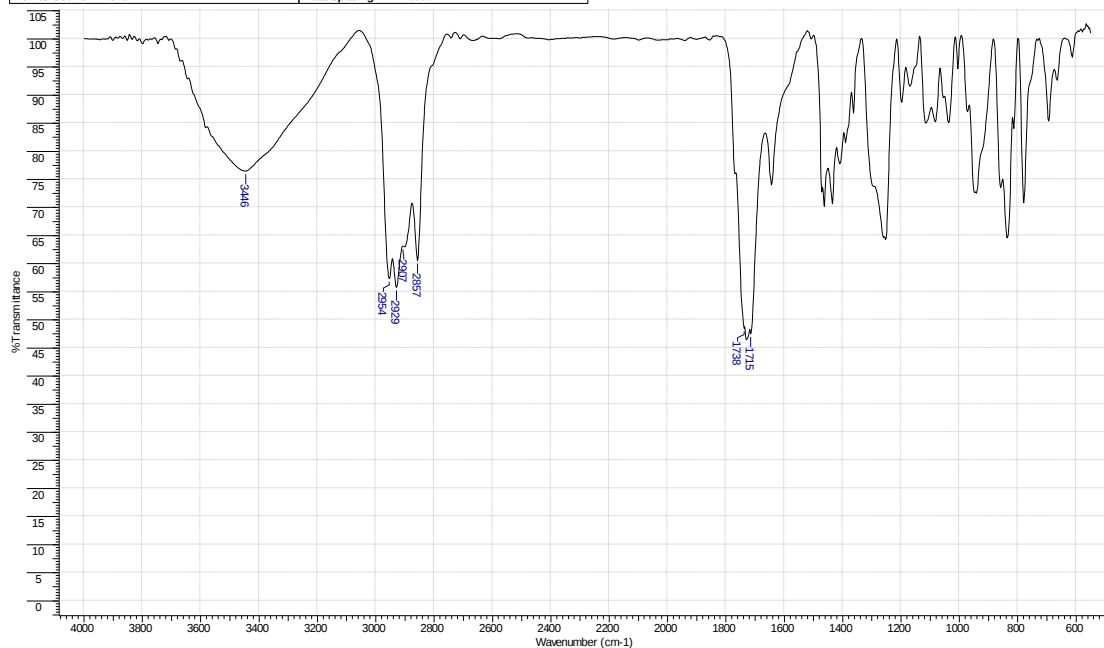
dez06dpsH1
 Danilo - DPS597 - CDCl₃ - Av 500MHz - dez06dpsH1 DEPT135



DEPT 135 of **11** (CDCl₃, 100 MHz)

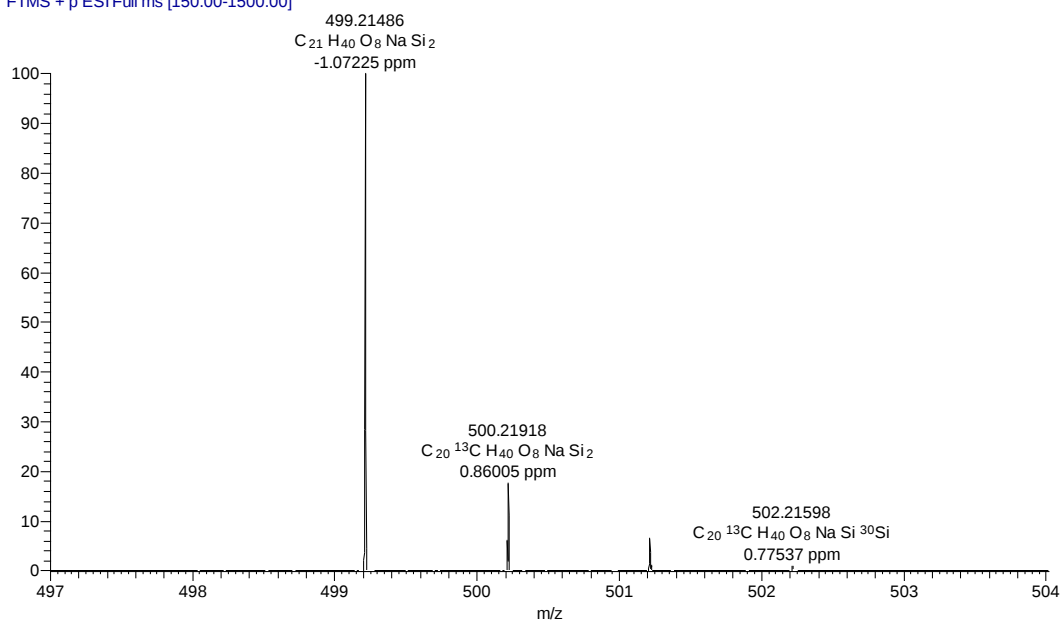
6 Jan 2014

Title	brasileiro de verão (GMT-2:00)	Comment	Amostra 004 por LQOS data sexta-feira, dezembro 13 2013
Origin	brasileiro de verão (GMT-2:00)	File Name	C:\USERS\DANILO P SANTANA\DOCUMENTS\TITAUTOMICETINA\VIDPSS97_1.SP
Date Stamp	fri dec 13 16:04:07 2013 Horário brasileiro de verão (GMT-2:00)	Date	fri dec 13 16:06:20 2013 Horário brasileiro de verão (GMT-2:00)
Technique	Infrared	Instrument	Spectrum Two
X Axis	Wavenumber (cm-1)	Y Axis	%Transmittance
Points Count	3451	Data Spacing	1.0000
		Spectral Region	IR
		Spectrum Range	550.0000 - 4000.0000



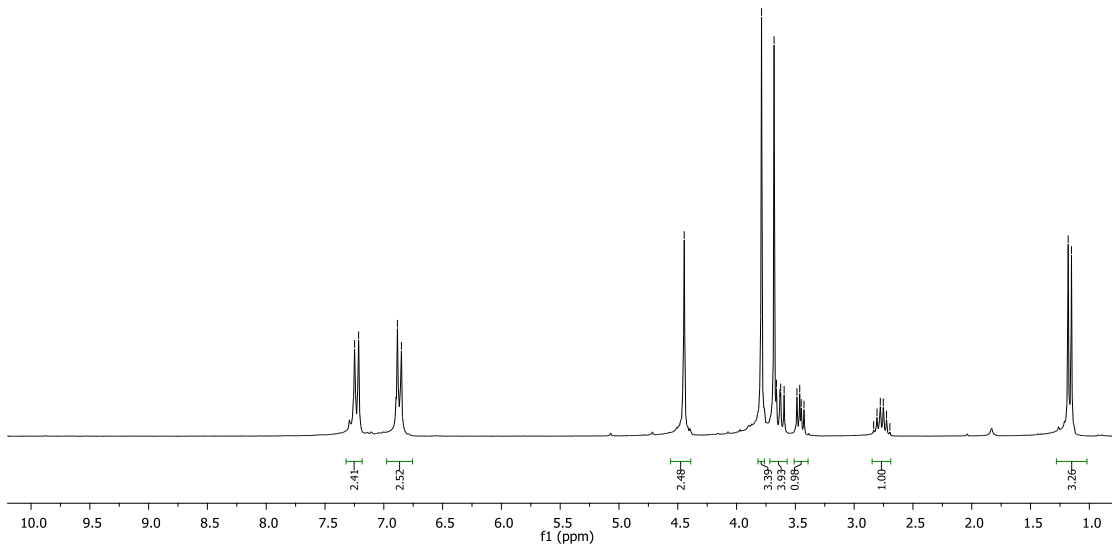
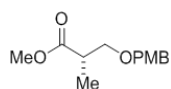
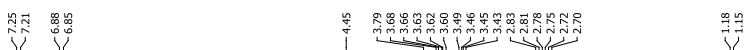
IR of **11**

D003 #54 RT: 0.82 AV: 1 NL: 1.07E7
T: FTMS + p ESI Full ms [150.00-1500.00]



HRMS (ESI+) m/z calculated for $C_{21}H_{40}O_8NaSi_2$ $[M+Na]^+$ 499.2159

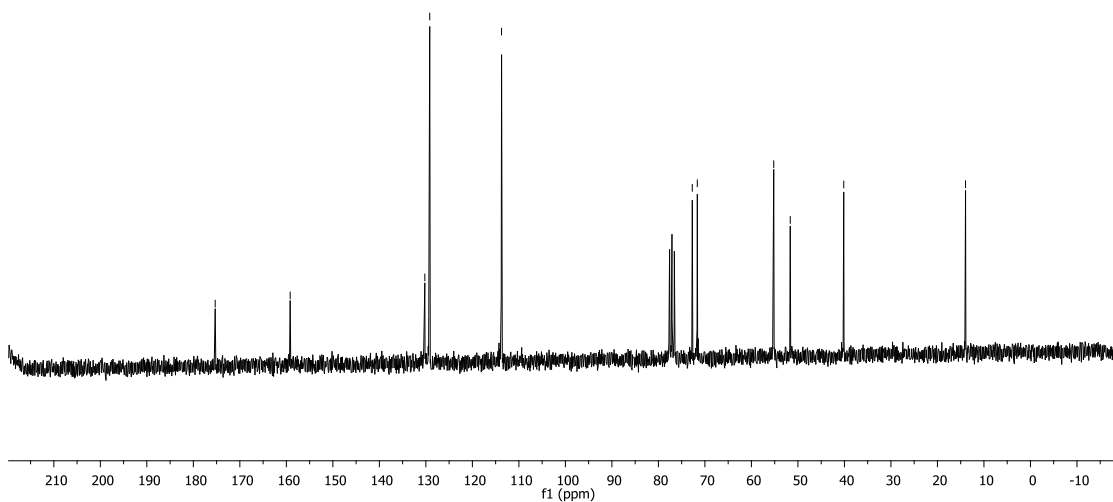
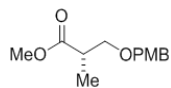
mai11dpsH1
mai11dpsH1 dpsRE



1H NMR of **36** ($CDCl_3$, 250 MHz)

mai11dpsH1
mai11dpsH1 dpsRE 13C

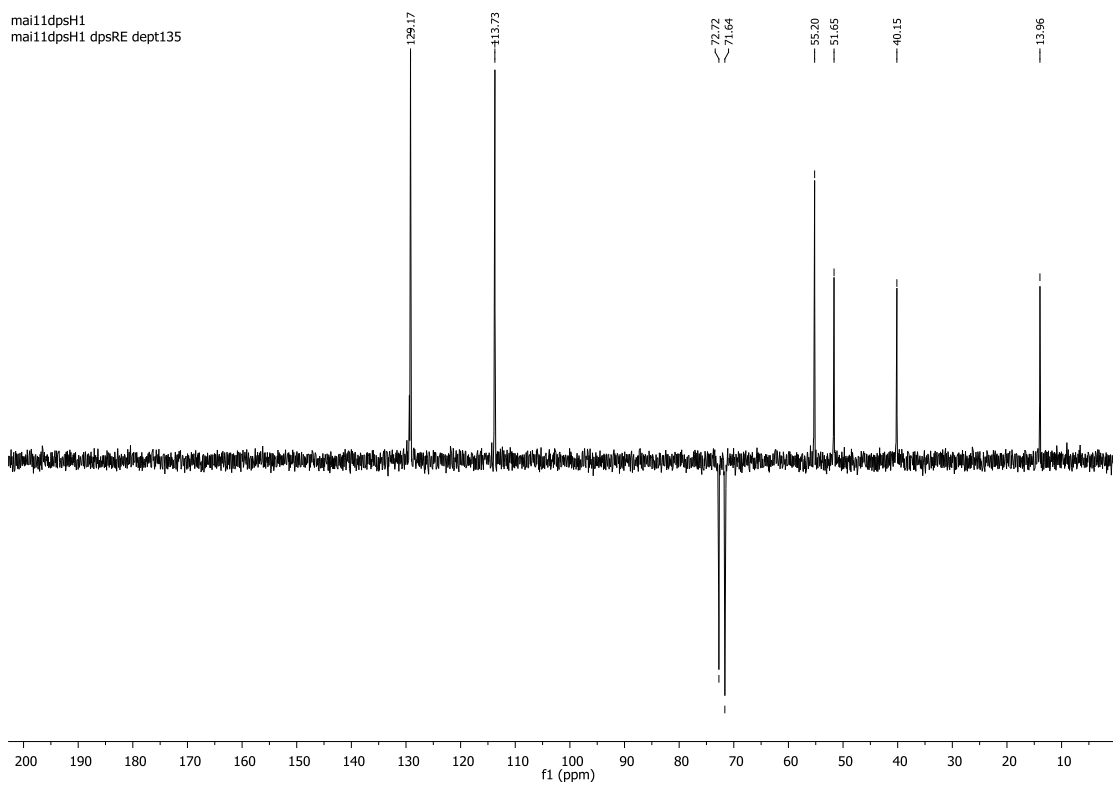
175.30 159.18 130.23 128.17 113.73 72.72 71.64 55.20 51.65 40.15 13.96



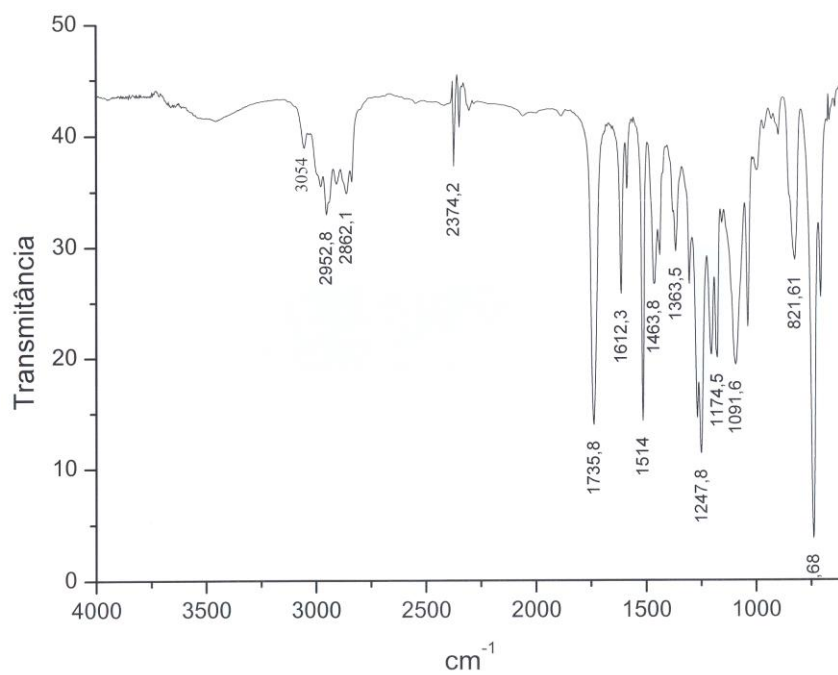
^{13}C NMR of **36** (CDCl₃, 62.5 MHz)

mai11dpsH1
mai11dpsH1 dpsRE dept135

128.17 113.73 72.72 71.64 55.20 51.65 40.15 13.96

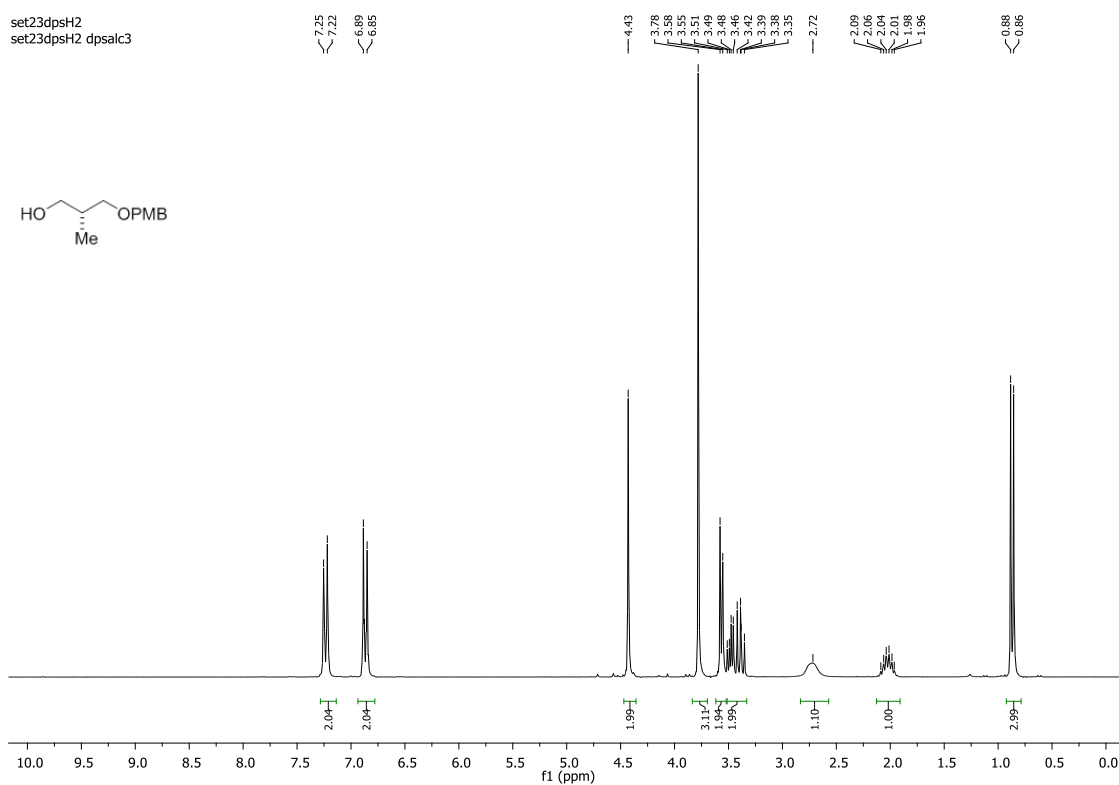


DEPT 135 of **36** (CDCl₃, 62.5 MHz)

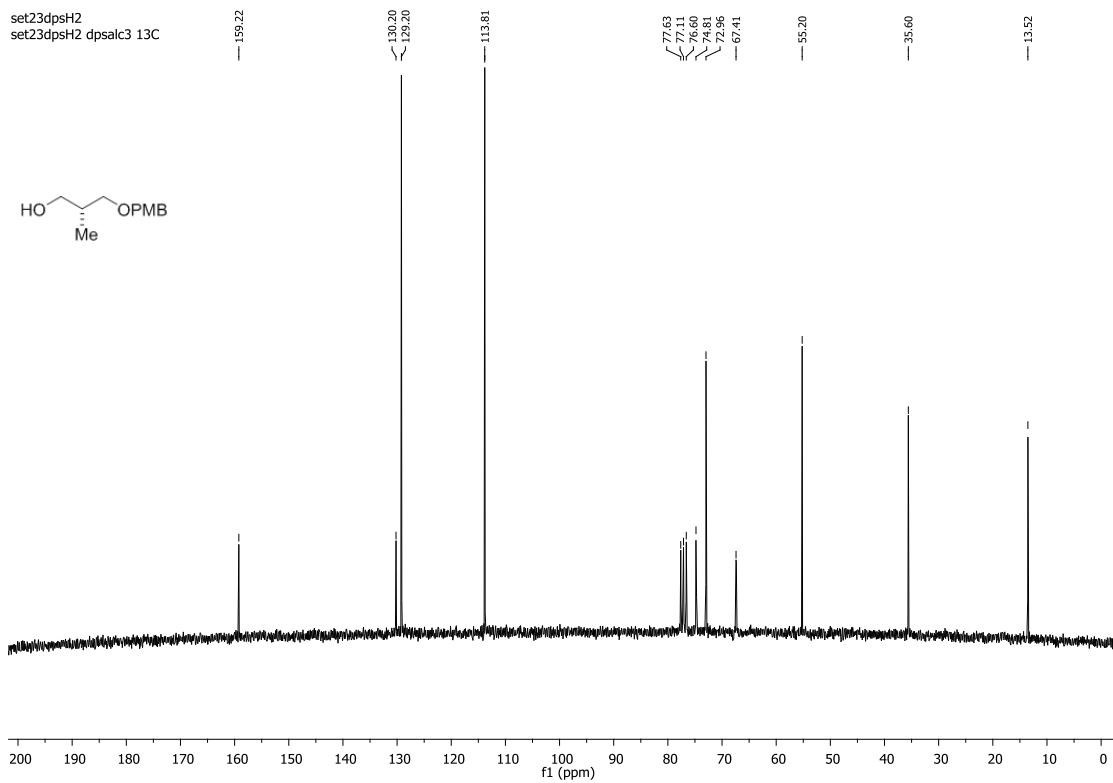


IR of **36**

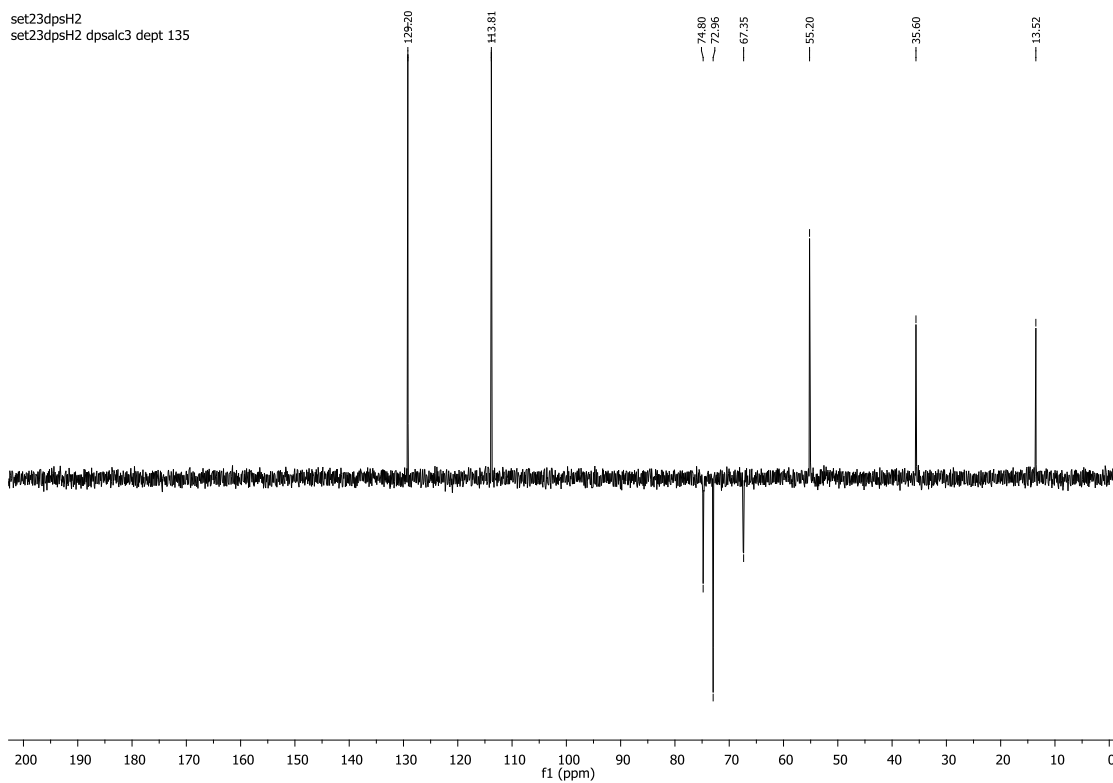
set23dpsH2
set23dpsH2 dpsalc3



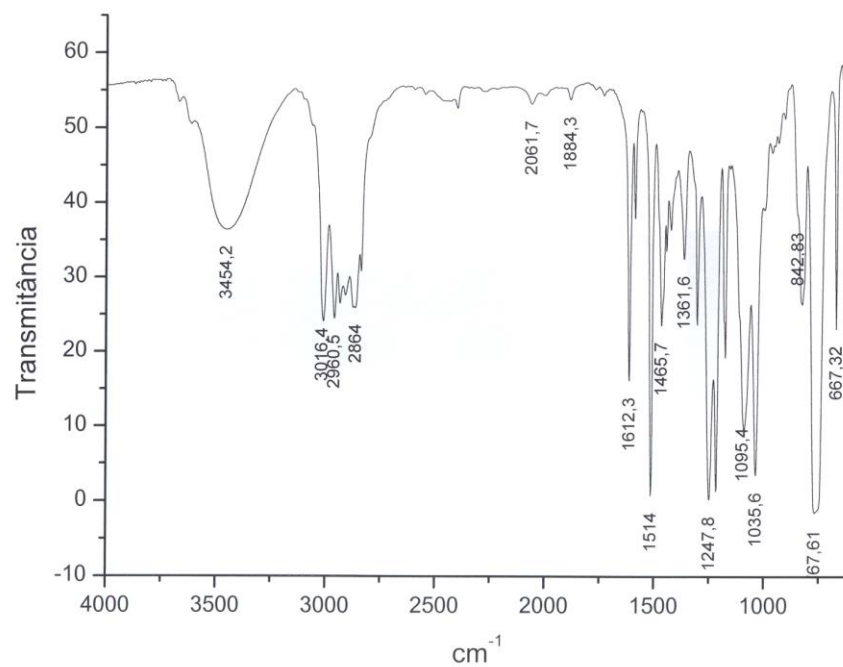
^1H NMR of **37** (CDCl_3 , 250 MHz)



¹³C NMR of **37** (CDCl₃, 62.5 MHz)

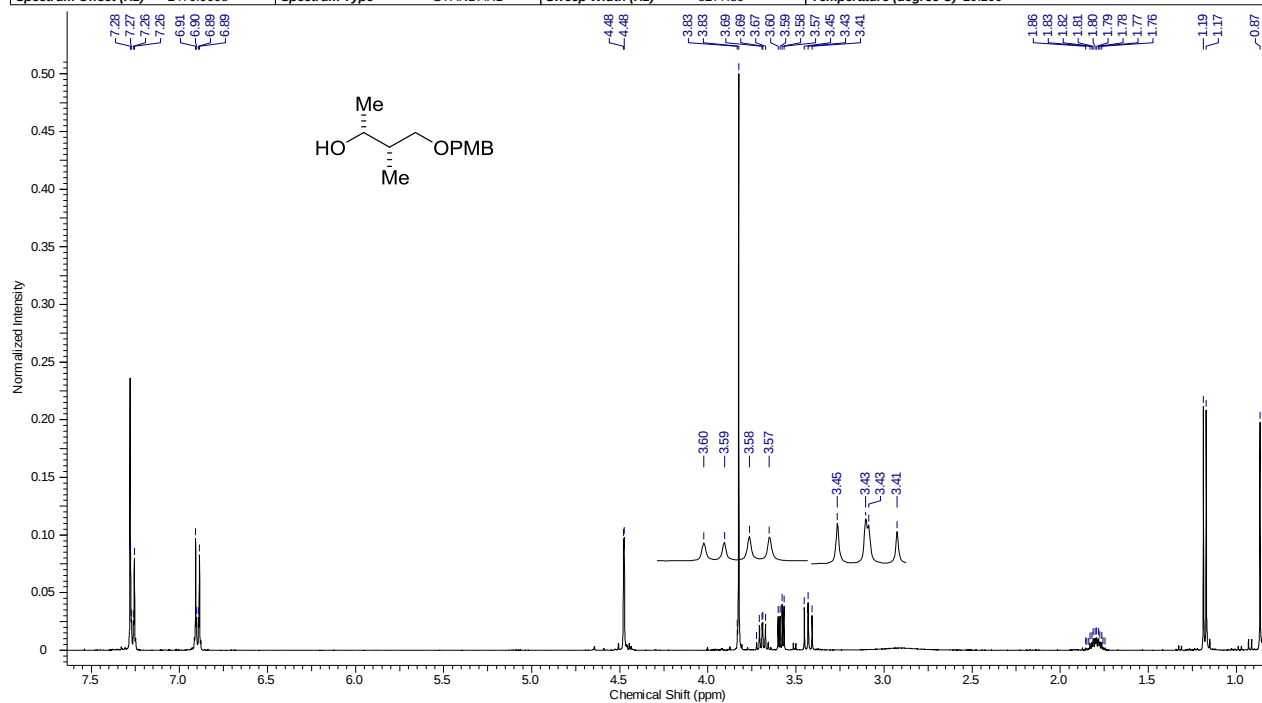


DEPT 135 of **37** (CDCl₃, 62.5 MHz)



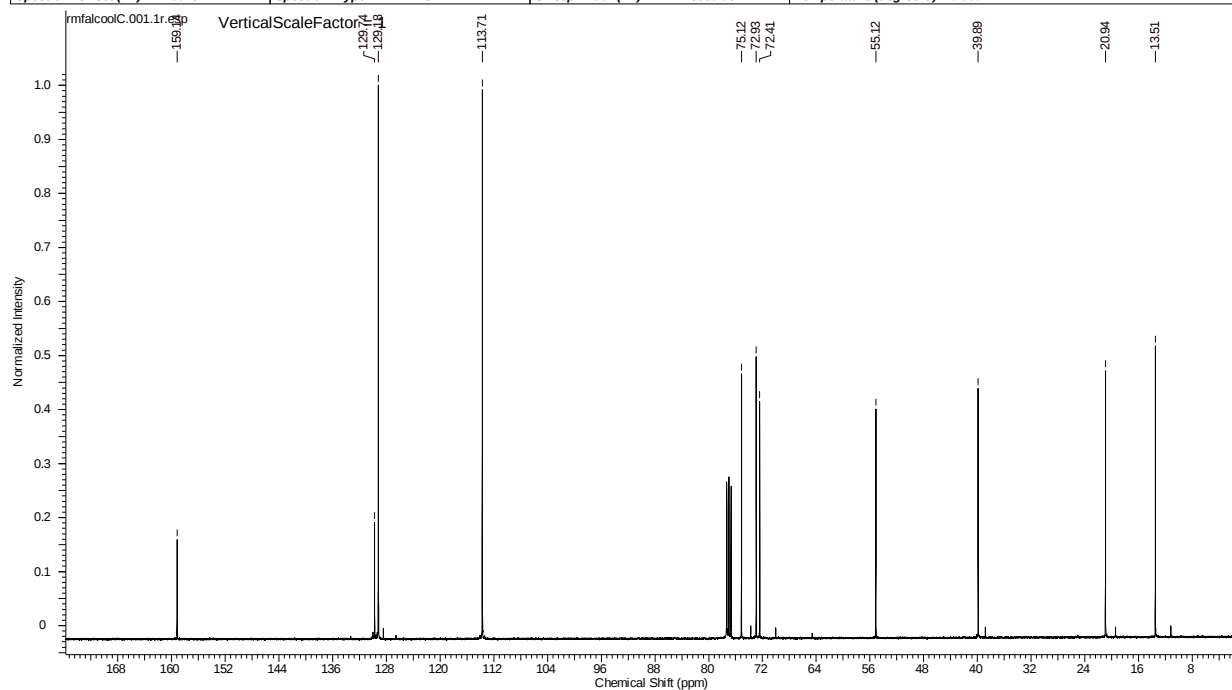
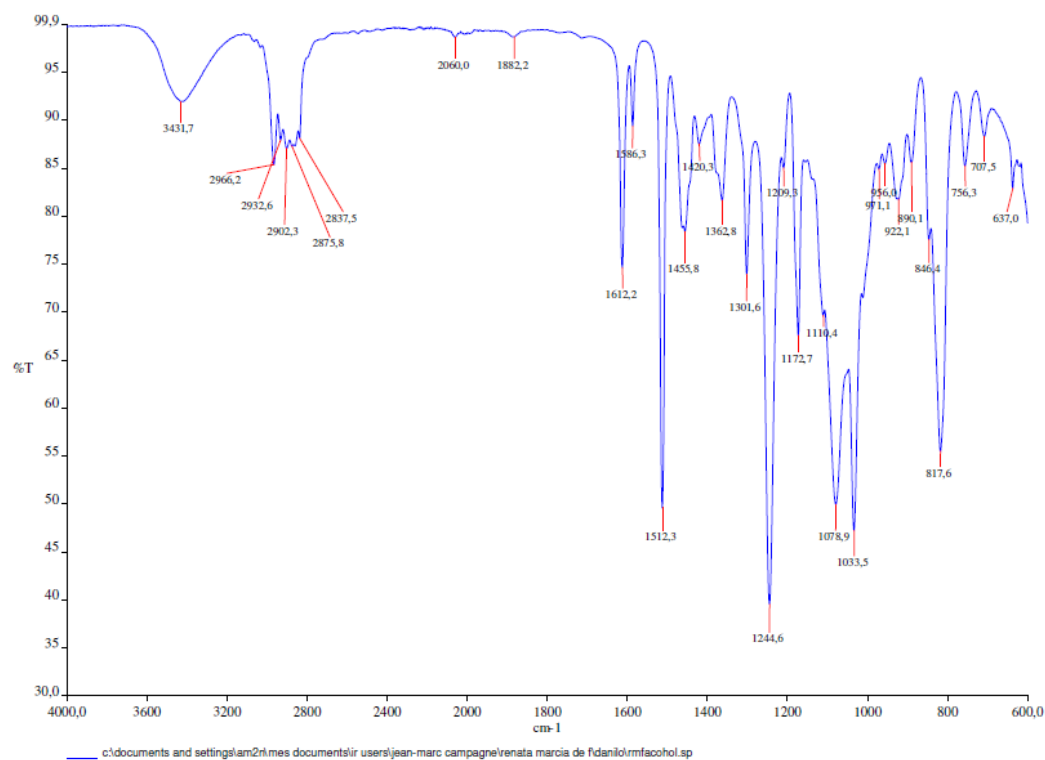
IR of **37**

Acquisition Time (sec)	3.9584	Comment	remalcool PROTON CDCl3 /opt/topspin am2n3 32	Date	13 Feb 2013 03:22:56
Date Stamp	13 Feb 2013 03:22:56	File Name	C:\Users\Danilo P Sant\Ana\Documents\tautomicetinalfidrenata\rmfalcool\data\11r	Origin	spect
Frequency (MHz)	400.13	Nucleus	1H	Number of Transients	8
Original Points Count	32768	Owner	auto	Points Count	32768
Receiver Gain	322.50	SW(cyclical) (Hz)	8278.15	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	2470.9668	Spectrum Type	STANDARD	Sweep Width (Hz)	8277.89
				Temperature (degree C)	20.260

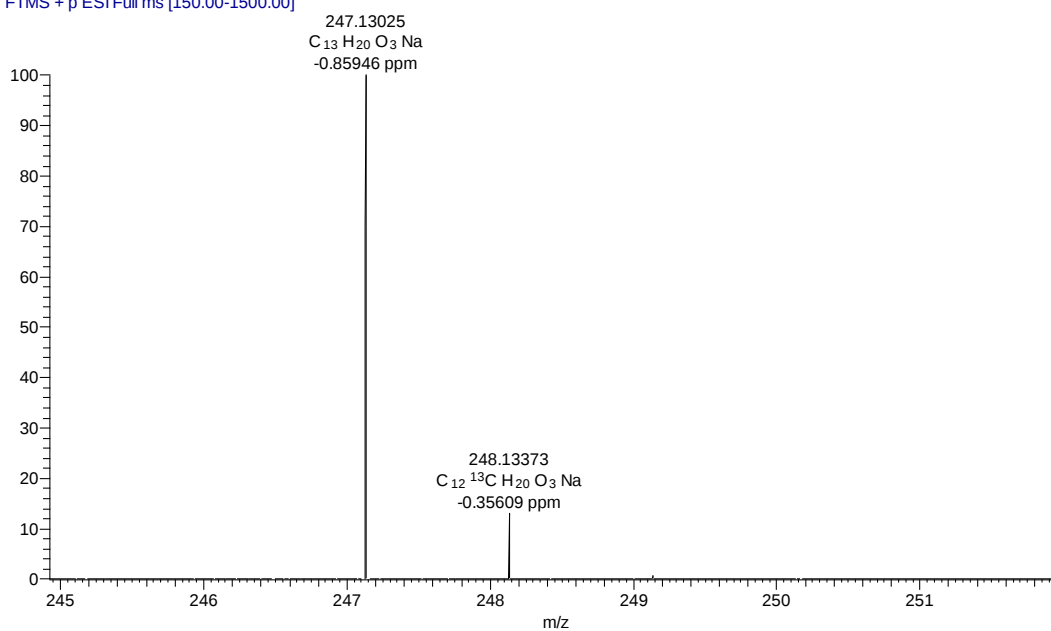


¹H NMR of **12** (CDCl₃, 400 MHz)

Acquisition Time (sec)	1.3664	Comment	RMFALCOOL C C13CPD CDCI3 /opt/topspin am2n3 60			Date	12 Feb 2013 23:41:04
Date Stamp	12 Feb 2013 23:41:04		File Name	C:\Users\Danilo\Documents\tautomicetina\fid\renata\rmfalcool\C\data\111r			
Frequency (MHz)	100.61	Nucleus	13C	Number of Transients	1024	Origin	spect
Original Points Count	32768	Owner	auto	Points Count	32768	Pulse Sequence	zgpg30
Receiver Gain	812.70	SW(cyclical) (Hz)	23980.81	Solvent	CHLOROFORM-d		
Spectrum Offset (Hz)	10048.4424	Spectrum Type	STANDARD	Sweep Width (Hz)	23980.08	Temperature (degree C)	20.960

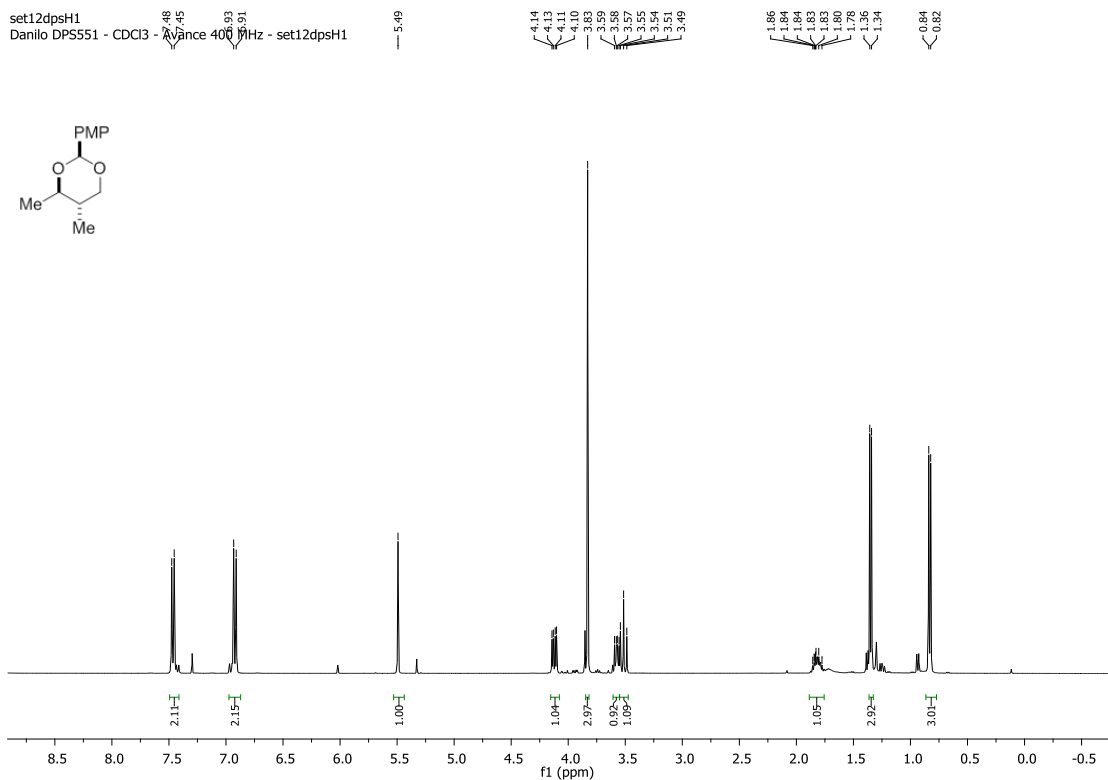
¹³C NMR of **12** (CDCl₃, 100 MHz)IR of **12**

D013 #54 RT: 0.81 AV: 1 NL: 7.35E6
T: FTMS + p ESI Full ms [150.00-1500.00]



HRMS (ESI+) m/z calculated for $C_{13}H_{20}O_3Na$ $[M+Na]^+$ 247.1310

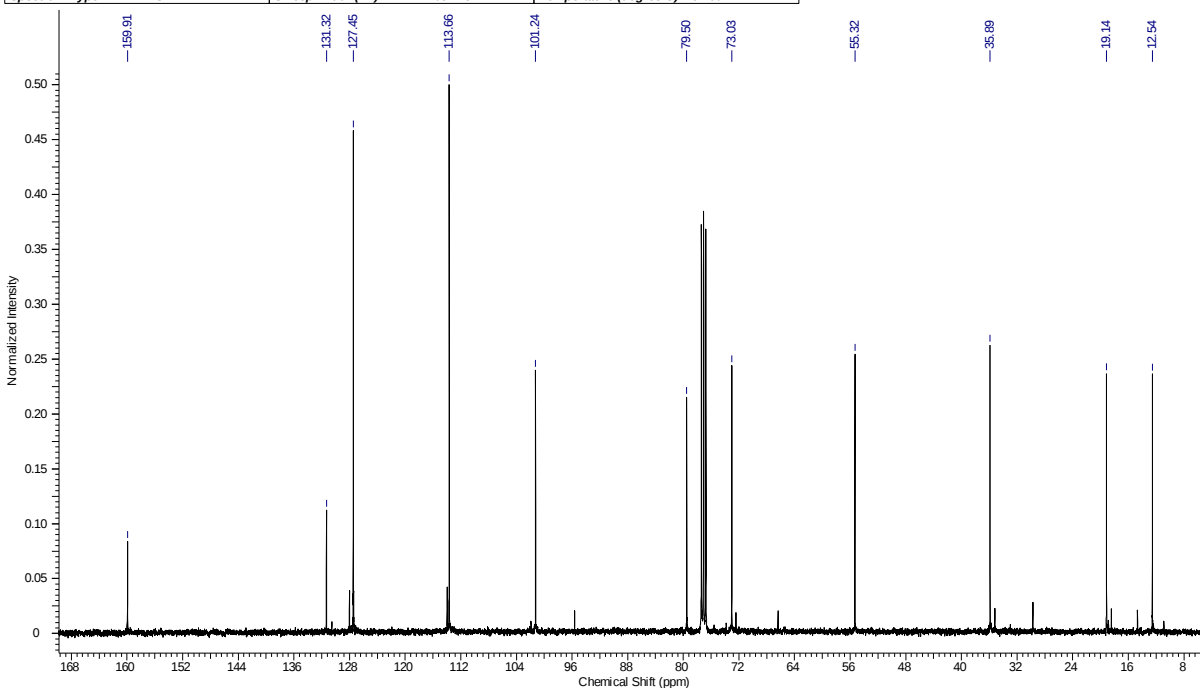
set12dpsH1
Danilo DPS551 - CDCl₃ - Avance 400 MHz - set12dpsH1



¹H NMR of **39** (CDCl₃, 400 MHz)

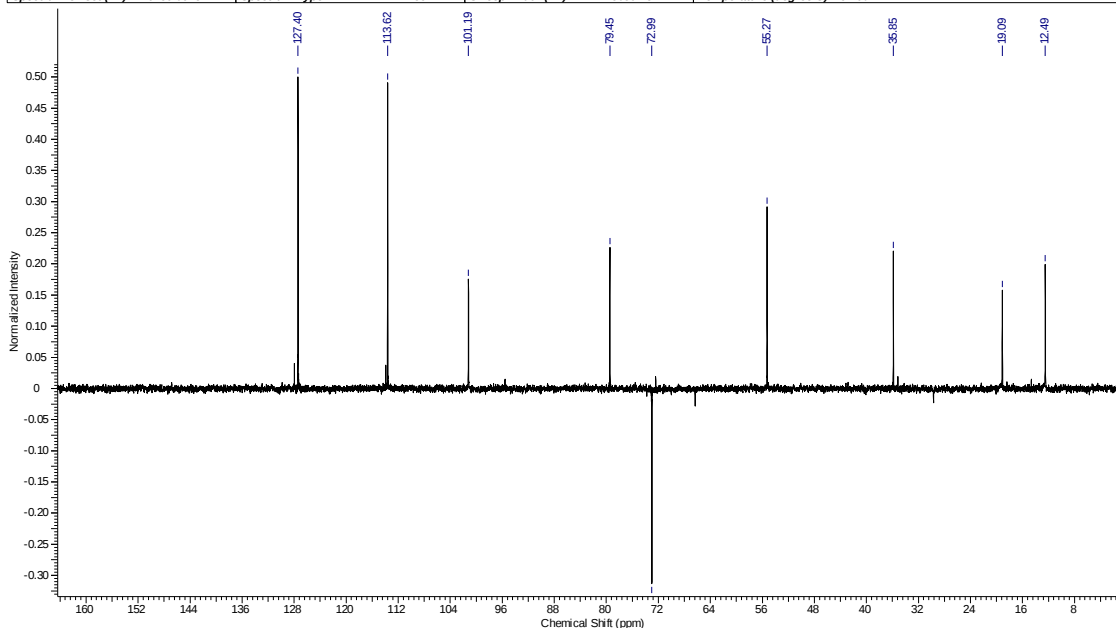
10/01/2014 15:18:34

Acquisition Time (sec)	0.6816	Comment	Danilo DPS551 - CDCl ₃ - Avance 400 MHz - set12dpsH1- 13C	Date	12 Sep 2013 13:00:00
Date Stamp	12 Sep 2013 13:00:00			File Name	C:\Users\Danilo P Sant\Ana\Documents\tautomicetina\fid\dps500-ldps551 noel6\data\11r
Frequency (MHz)	100.63	Nucleus	¹³ C	Number of Transients	3200
Original Points Count	16384	Owner	avance	Points Count	32768
Receiver Gain	203.00	SW(cyclical) (Hz)	24038.46	Solvent	CHLOROFORM-d
Spectrum Type	STANDARD	Sweep Width (Hz)	24037.73	Temperature (degree C)	25.160
				Spectrum Offset (Hz)	10061.5625

¹³C NMR of **39** (CDCl₃, 100 MHz)

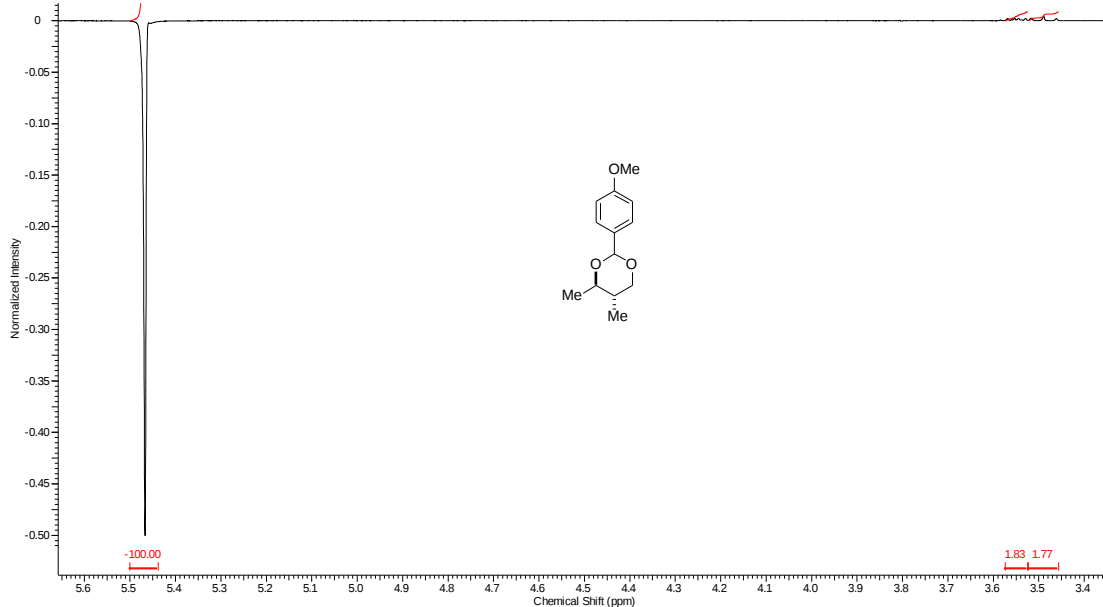
10/01/2014 15:26:04

Acquisition Time (sec)	0.5439	Comment	dps551 dept135	Date	12 Sep 2013 20:42:56
Date Stamp	12 Sep 2013 20:42:56			File Name	C:\Users\Danilo P Sant\Ana\Documents\tautomicetina\fid\dps500-ldps551 dept1\data\11r
Frequency (MHz)	62.90	Nucleus	¹³ C	Number of Transients	1
Original Points Count	8192	Owner	root	Points Count	32768
Receiver Gain	1149.40	SW(cyclical) (Hz)	15060.24	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	6289.0518	Spectrum Type	DEPT135	Sweep Width (Hz)	15059.78
				Temperature (degree C)	25.160

DEPT 135 of **39** (CDCl₃, 62.5 MHz)

10/01/2014 17:06:12

Acquisition Time (sec)	4.1943	Comment	Danilo DPS551 - CDCI3 - Avance 400 MHz - set12dpsH1 - NOE-Seleto		
Date	12 Sep 2013 11:19:44	Date Stamp	12 Sep 2013 11:19:44		
File Name	C:\Users\Danilo P Sant\Ana\Documents\tautomicetina\fid\dp5500\dp551 noe4\data1\1r	Frequency (MHz)	400.18		
Nucleus	¹ H	Number of Transients	320	Origin	spect
Owner	avance	Points Count	32768	Pulse Sequence	selhnp
SW(cyclical) (Hz)	3906.25	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	1603.5298
Temperature (degree C)	25.160	Sweep Width (Hz)	3906.13		



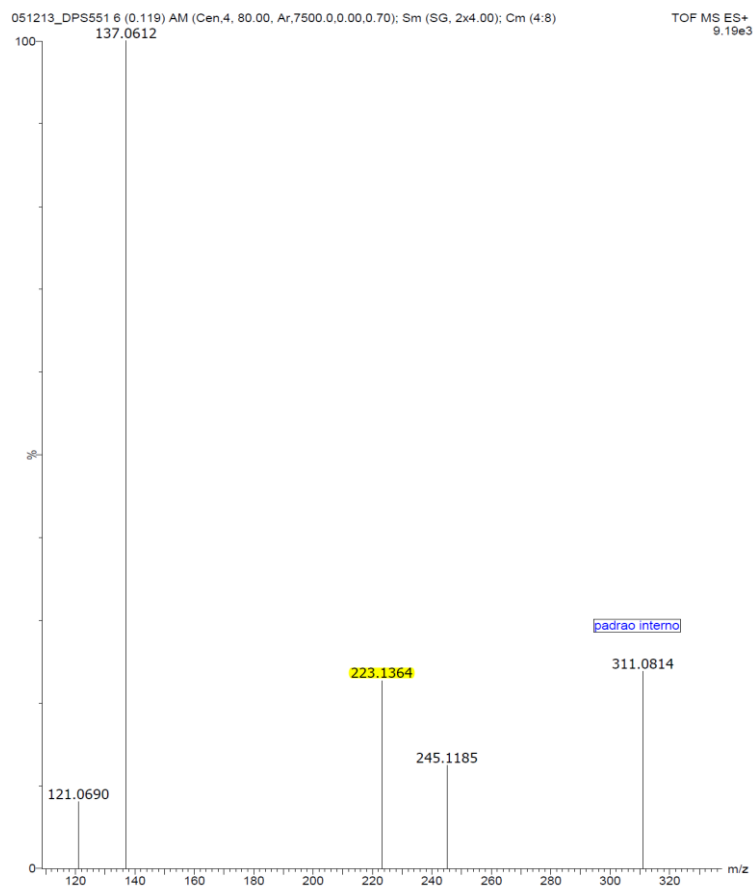
Selective Noe of 39

10 Jan 2014

Title	brasileiro de verão (GMT-2:00)	Comment	Amostra 001 por LQOS data quarta-feira, dezembro 11 2013		
Origin	brasileiro de verão (GMT-2:00)	File Name	C:\USERS\DANILO P SANT\ANADOCUMENTS\TAUTOMICETINA\VIDP5551_1.SP		
Date Stamp	wed dec 11 17:45:57 2013 Horário brasileiro de verão (GMT-2:00)	Date	wed dec 11 17:47:25 2013 Horário brasileiro de verão (GMT-2:00)		
Technique	Infrared	Instrument	Spectrum Two	Spectral Region	IR
X Axis	Wavenumber (cm-1)	Y Axis	%Transmittance	Spectrum Range 550.0000 - 4000.0000	
Points Count	3451	Data Spacing	1.0000		

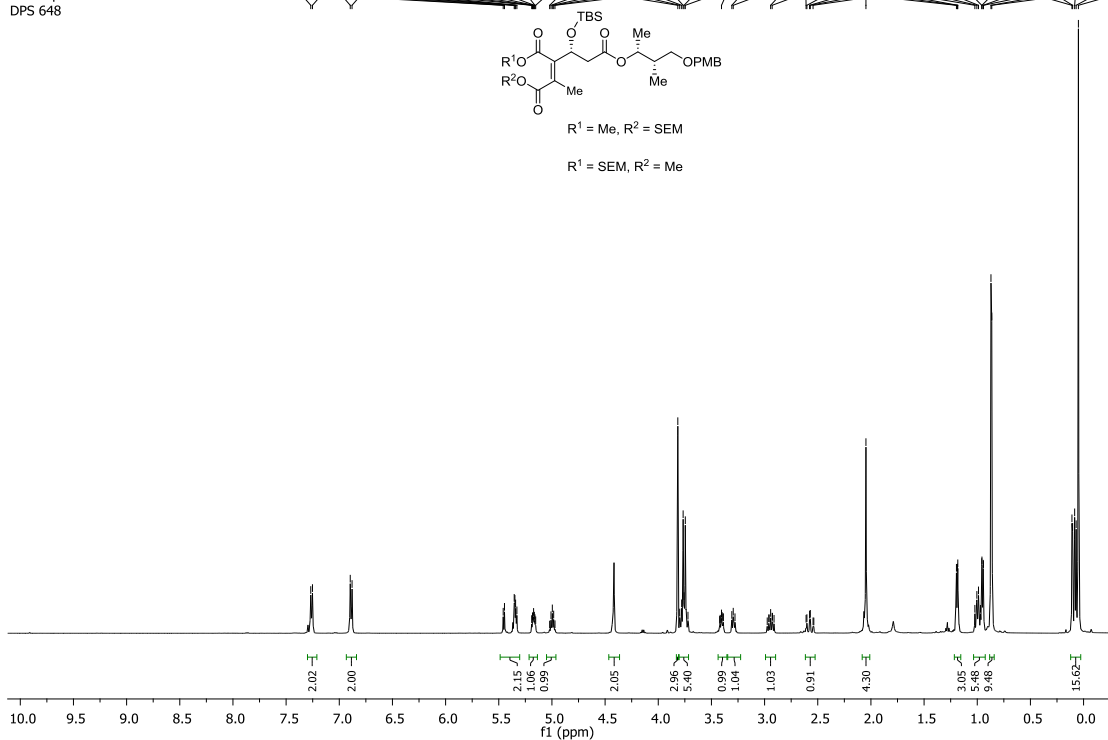
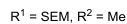
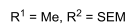


IR of 39



HRMS (ESI+) m/z calculated for $[M+H]^+$ 223.1334

Figure 1 displays 12 line graphs showing the variation of TBS (Tensile Bond Strength) in MPa versus the thickness of the adhesive layer (mm) for different combinations of primer and adhesive. The graphs are arranged in three rows of four. Each graph has a y-axis labeled 'TBS (MPa)' and an x-axis labeled 'TPS (mm)'. The data points are connected by lines, and some points are highlighted with circles. The values generally decrease as the thickness of the adhesive layer increases, with some variations depending on the primer and adhesive combination.

¹H NMR **41** (CDCl₃, 500 MHz)

50.38
50.34
48.05
47
167.72
166.98
166.89
— 159.12

143.61	130.57
142.50	129.18

— 113.74

 $\begin{matrix} 90.15 \\ 90.11 \end{matrix}$

✓ 77.07
✓ 76.81

72.50
71.73

67.43

51.94

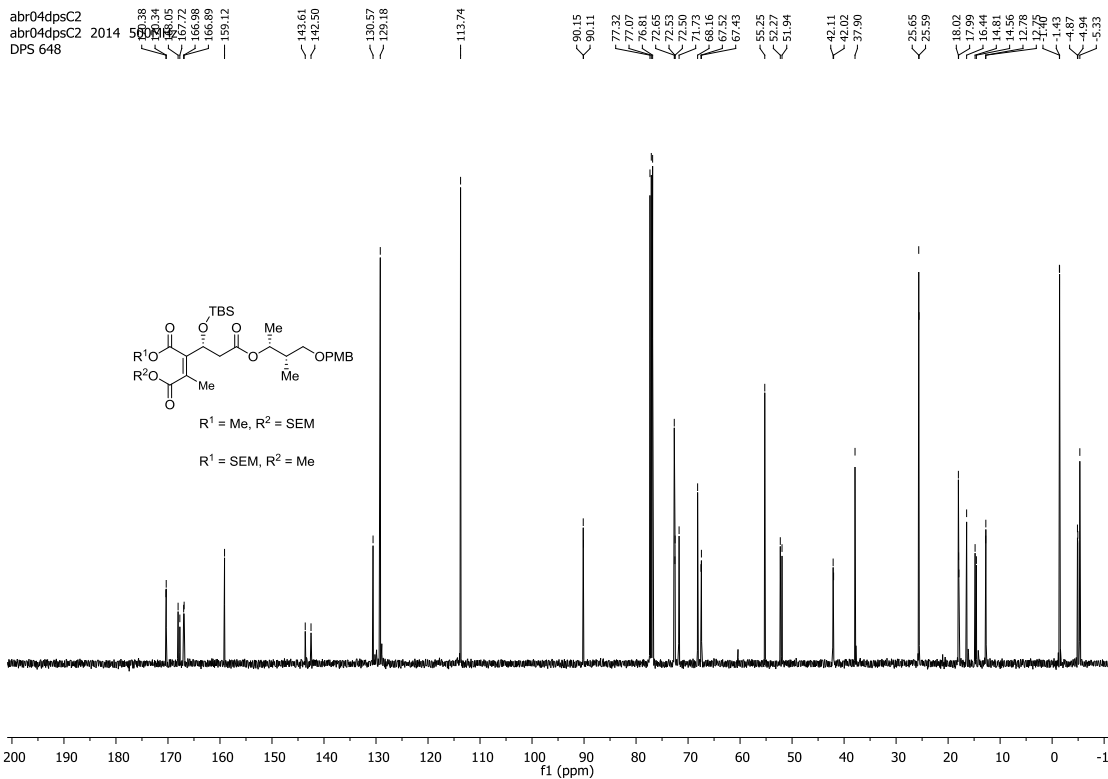
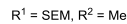
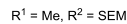
42.02
37.90

✓ 25.65

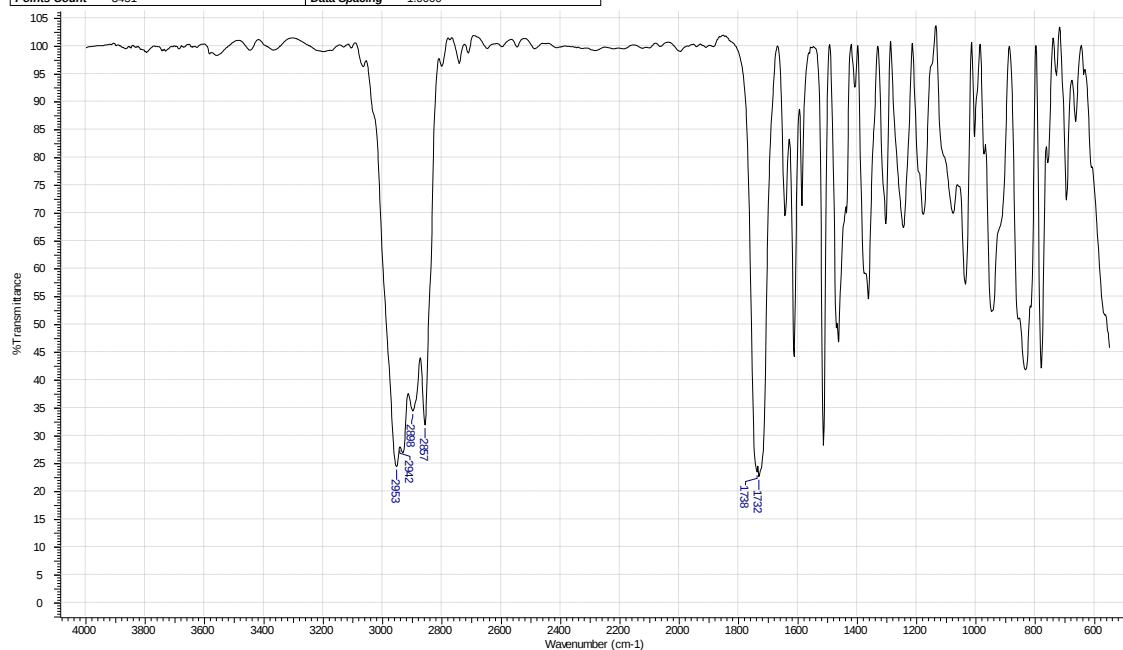
18.02
17.99

14.56
12.78

4-1.43
4-4.87
4-1.01

 ^{13}C NMR **41** (CDCl_3 , 125 MHz)

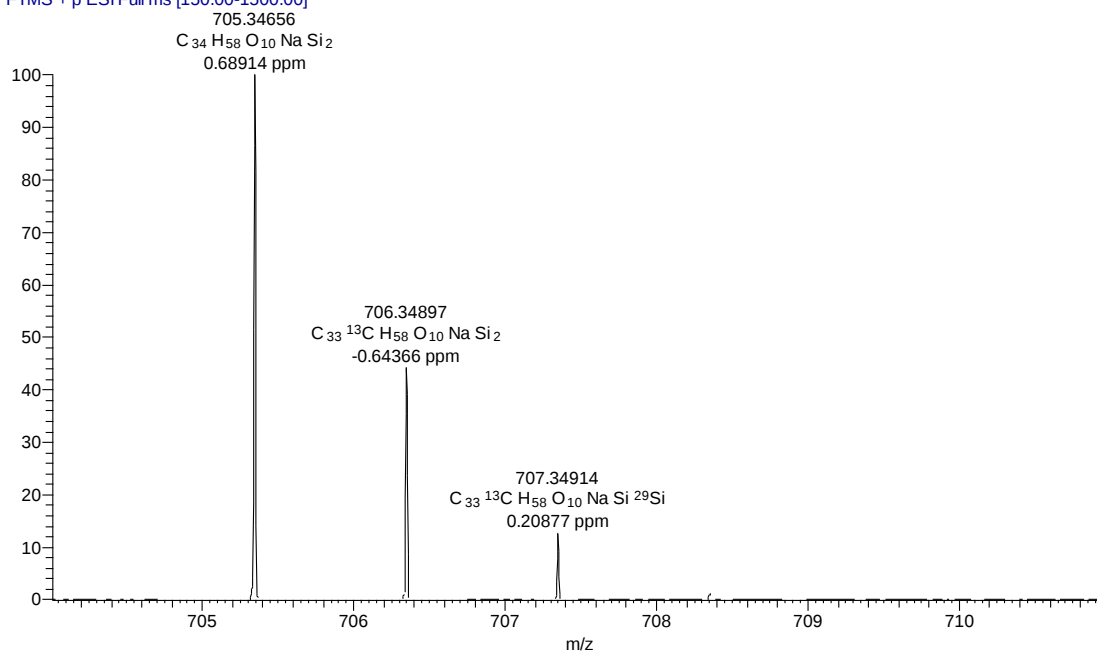
Title	brasileiro de verão (GMT-2:00)	Comment	Amostra 004 por LQOS data quarta-feira, dezembro 11 2013
Origin	brasileiro de verão (GMT-2:00)	File Name	C:\USERS\IDANILO P SANTANA\DOCUMENTS\1TAUTOMICETINA\IVDPS598.SP
Date Stamp	wed dec 11 18:25:32 2013 Horário brasileiro de verão (GMT-2:00)	Date	wed dec 11 18:29:23 2013 Horário brasileiro de verão (GMT-2:00)
Technique	Infrared	Instrument	Spectrum Two
X Axis	Wavenumber (cm-1)	Y Axis	% Transmittance
Points Count	3451	Data Spacing	1.0000
		Spectral Region	IR
		Spectrum Range	550.0000 - 4000.0000



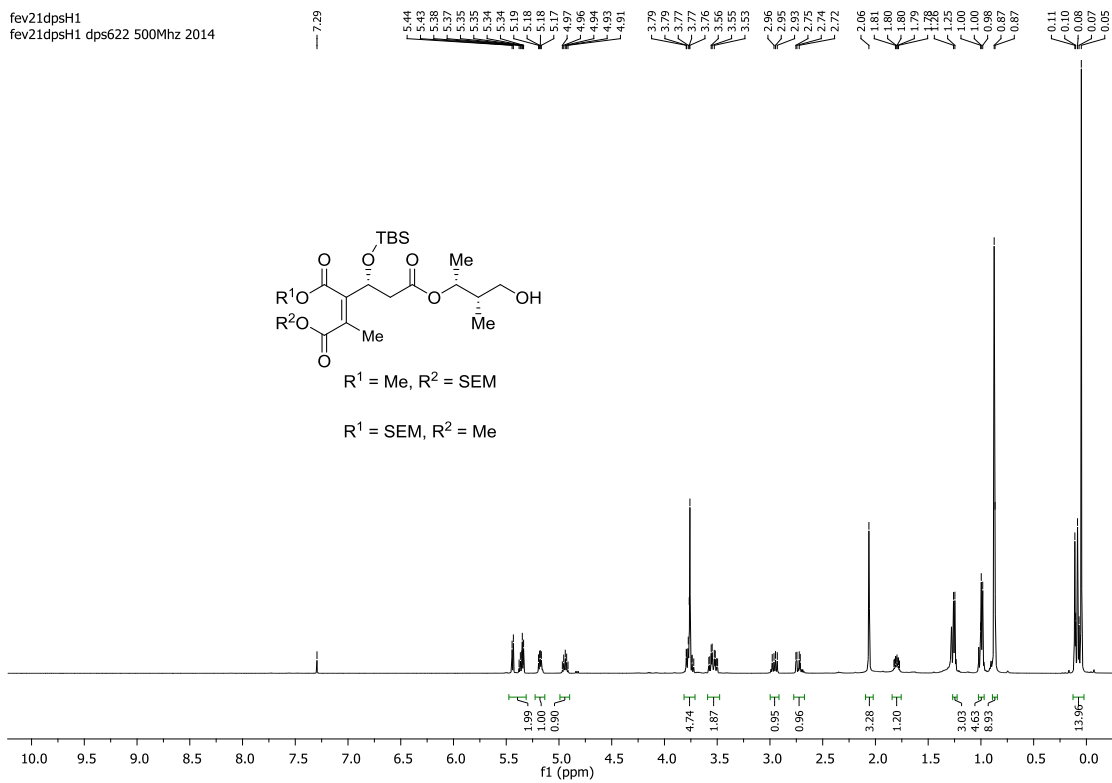
IR of 41

D007 #11 RT: 0.15 AV: 1 NL: 8.80E7

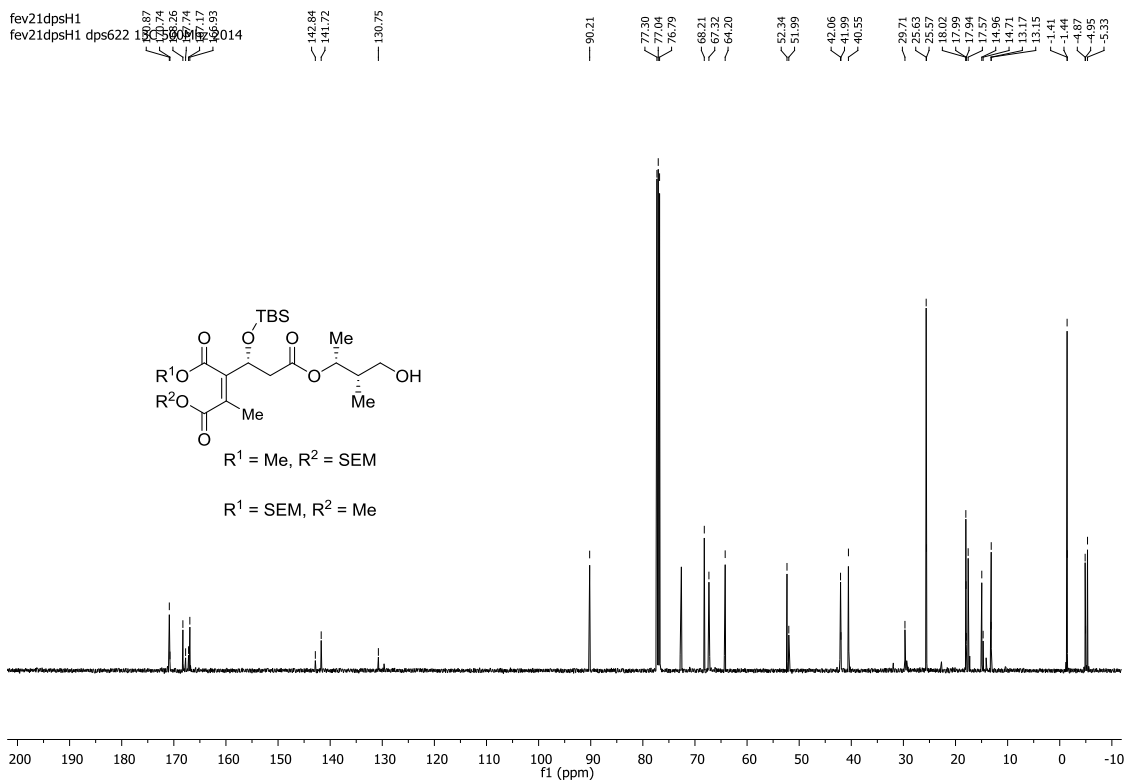
T: FTMS + p ESI Full ms [150.00-1500.00]

HRMS (ESI+) m/z calculated for $C_{34}H_{58}O_{10}NaSi_2$ $[M+Na]^+$ 705.3466

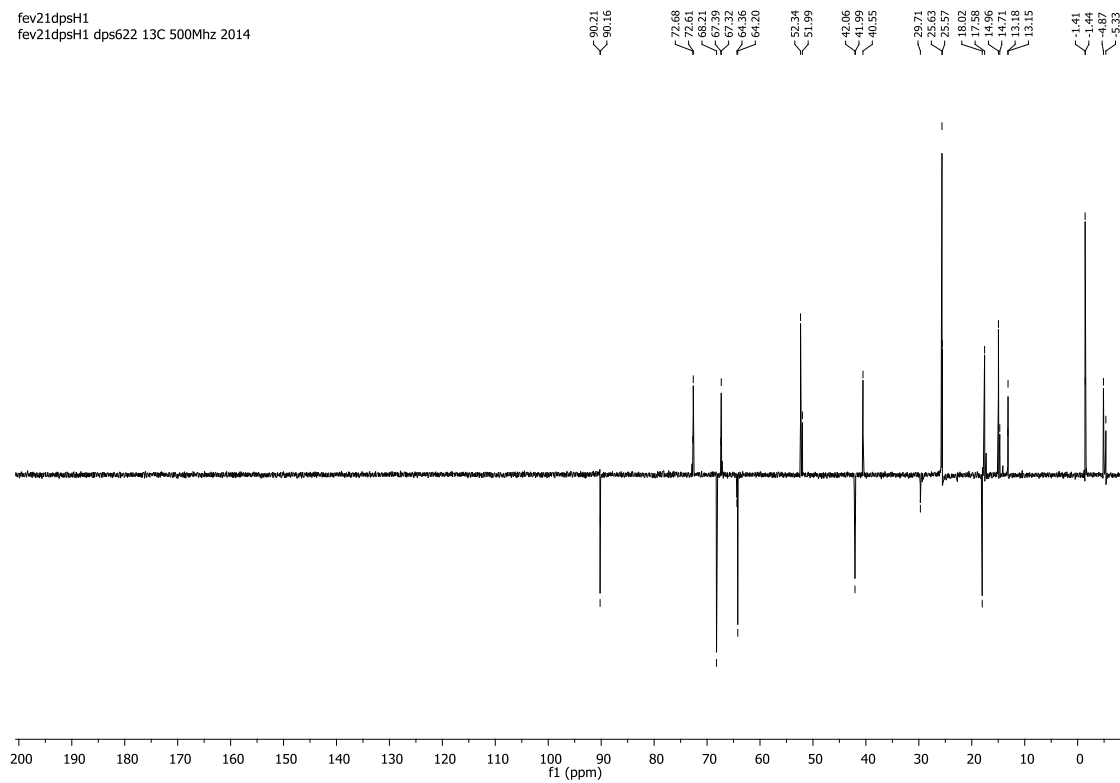
fev21dpsH1
fev21dpsH1 dps622 500Mhz 2014



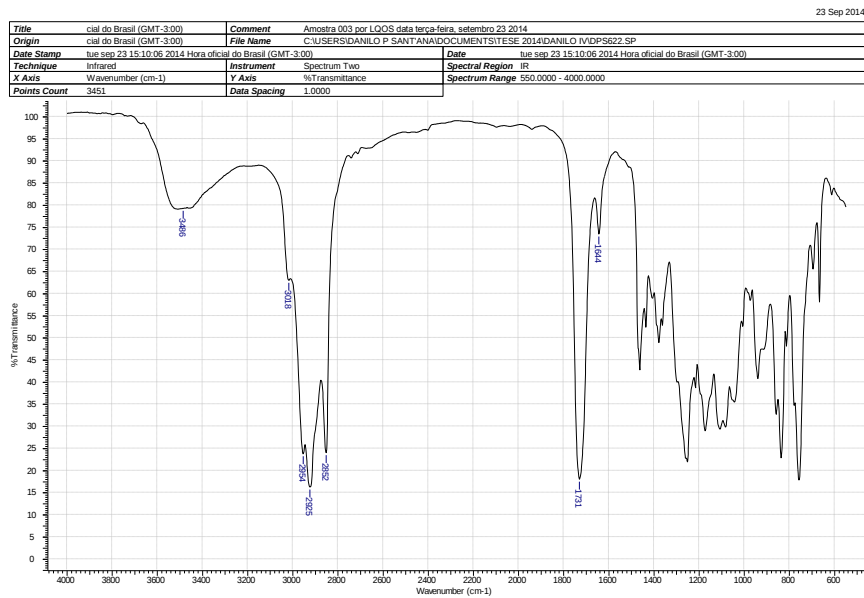
fev21dpsH1
fev21dpsH1 dps622 125Mhz 2014



fev21dpsH1
fev21dpsH1 dps622 13C 500Mhz 2014

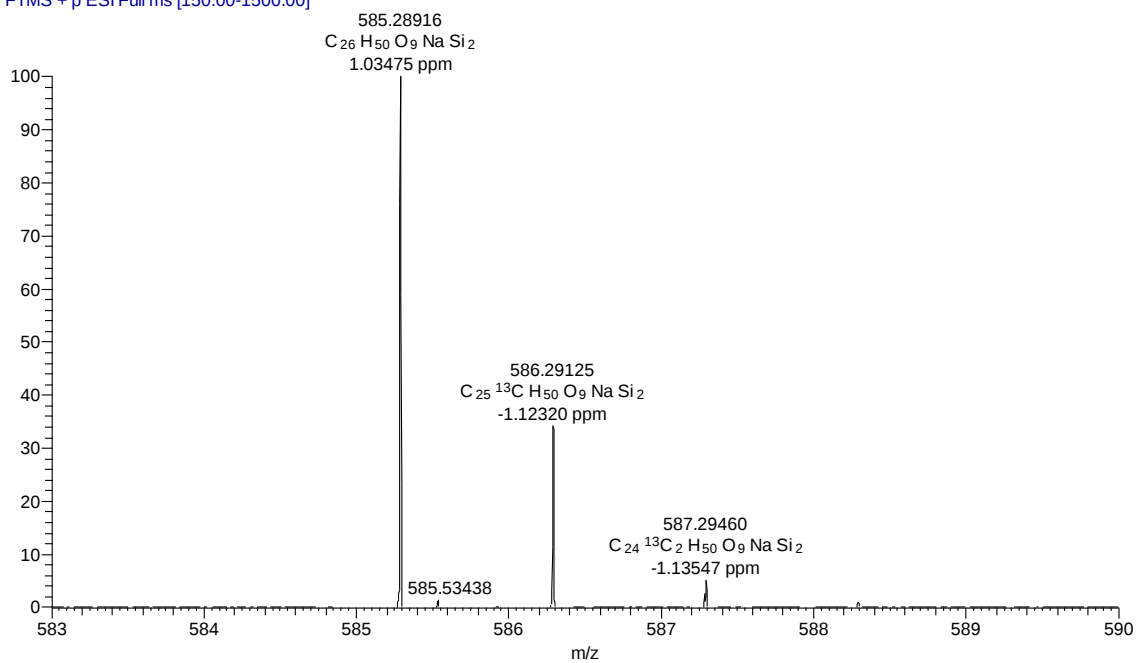


DEPT 135 of **42** (CDCl₃, 125 MHz)

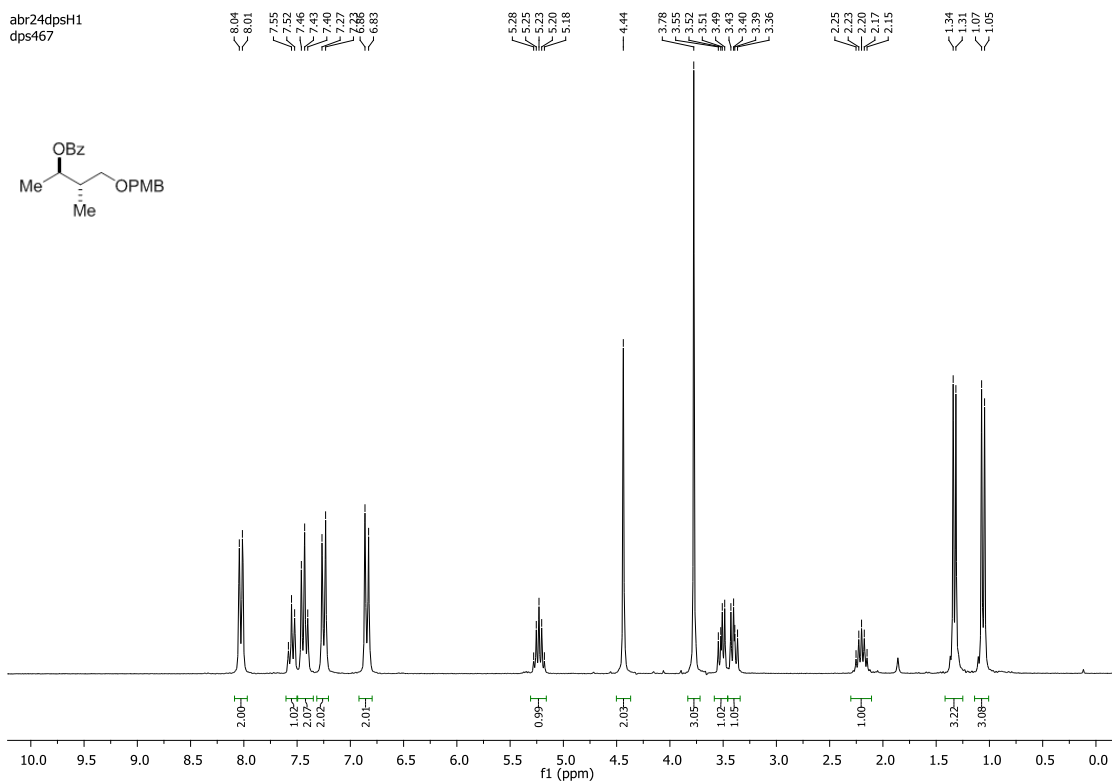


IR of **42**

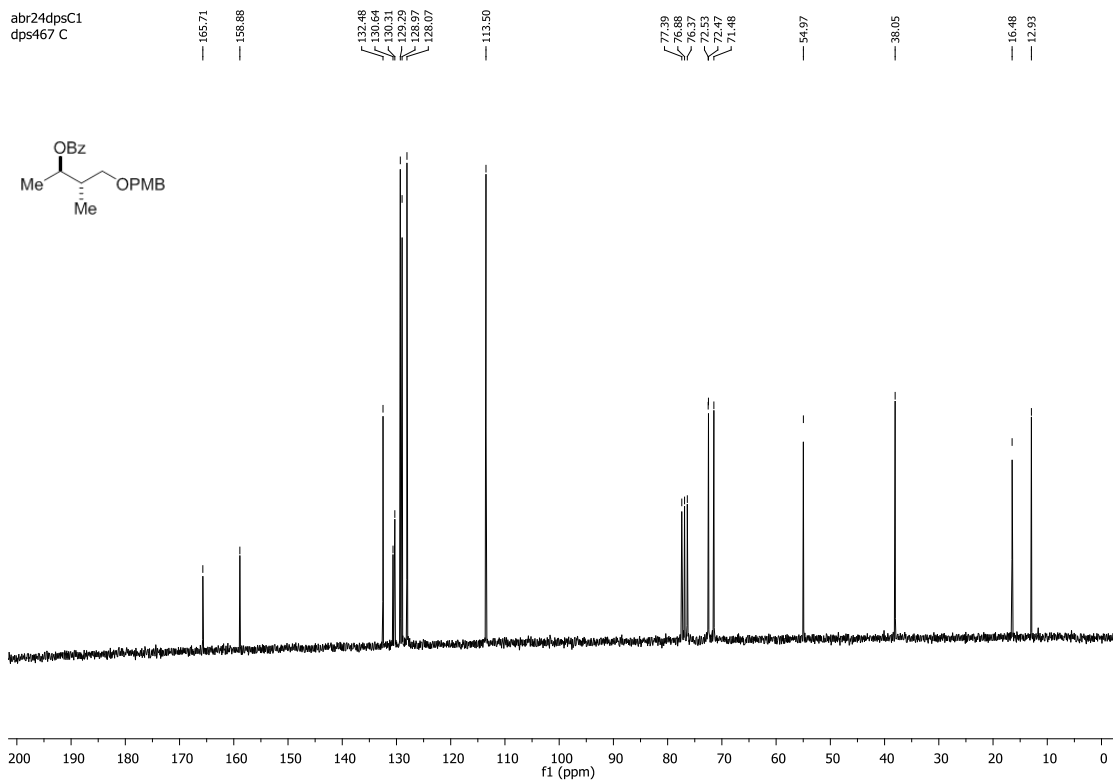
D001 #7 RT: 0.10 AV: 1 NL: 6.68E7
T: FTMS + p ESI Full ms [150.00-1500.00]



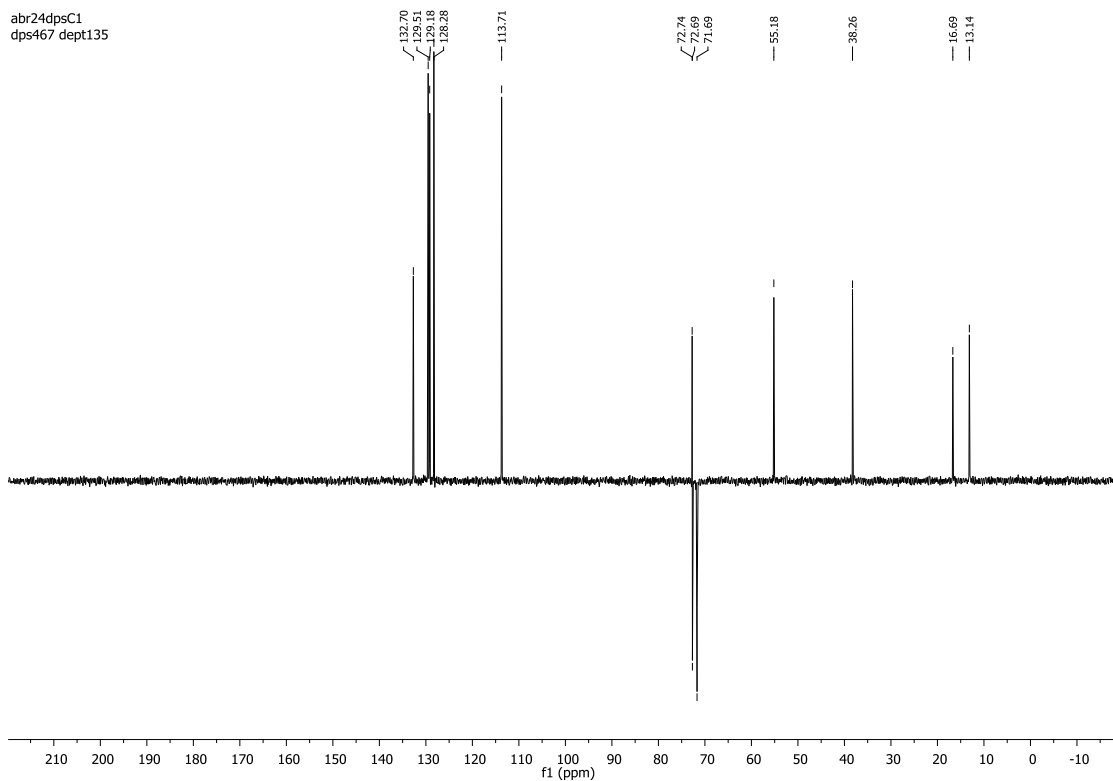
HRMS (ESI+) *m/z* calculated for C₂₆H₅₀O₉NaSi₂ [M+Na]⁺ 585.2891



¹H NMR of **43** (CDCl₃, 250 MHz)



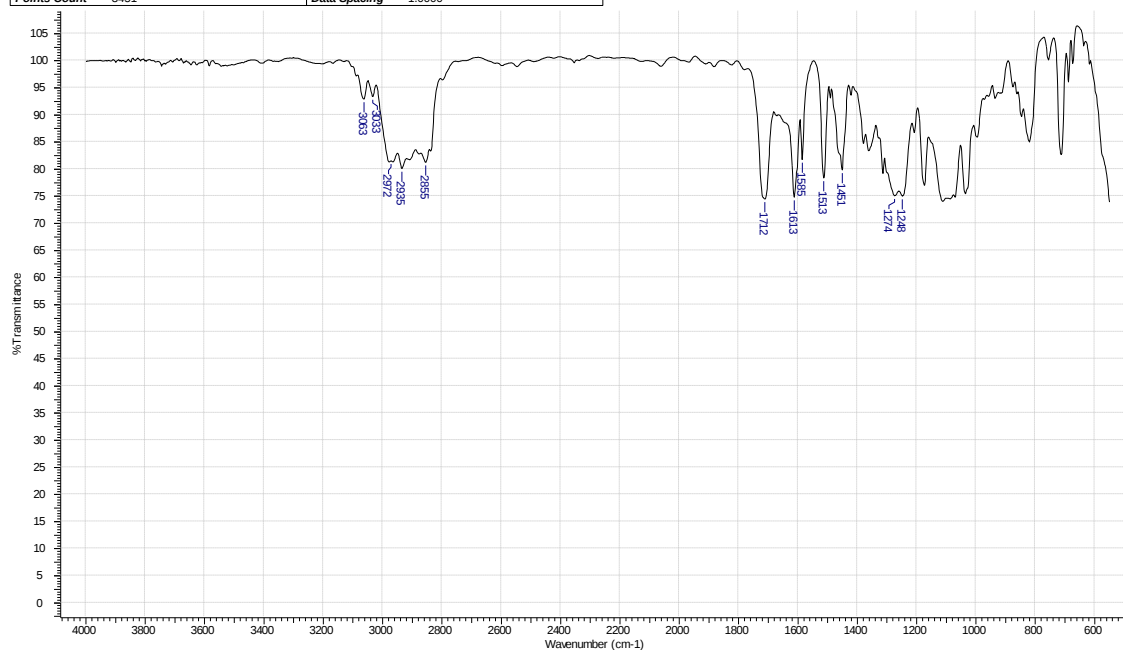
¹³C NMR of **43** (CDCl₃, 62.5 MHz)



DEPT 135 of **43** (CDCl₃, 62.5 MHz)

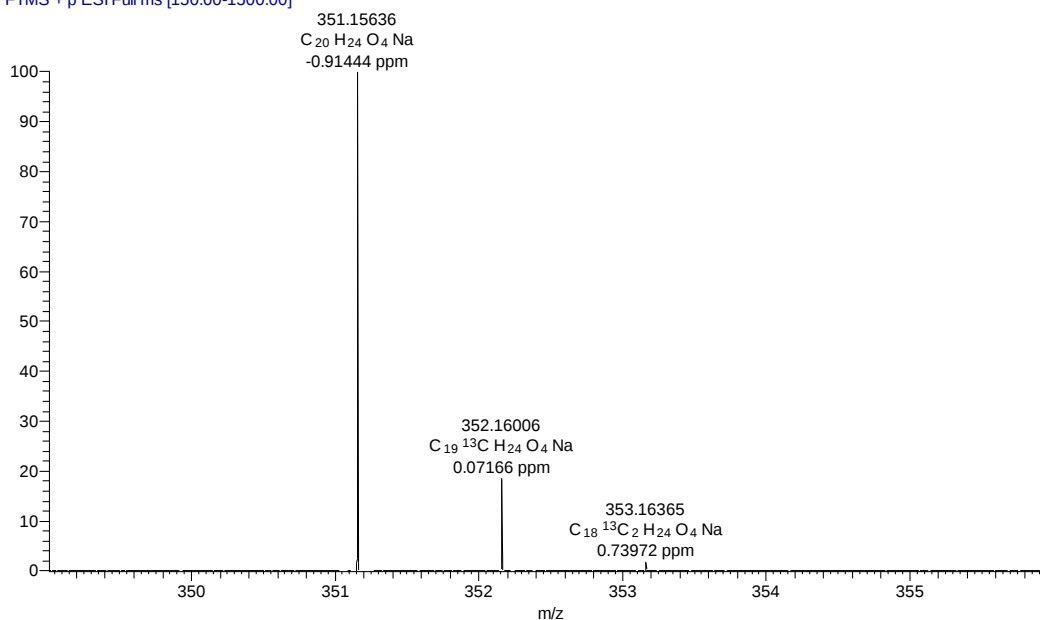
8 Jan 2014

Title	brasileiro de verão (GMT-2:00)	Comment	Amostra 002 por LQOS data quarta-feira, dezembro 11 2013
Origin	brasileiro de verão (GMT-2:00)	File Name	C:\USERS\IDANILO P. SANTANA\DOCUMENTS\ITAUTOMICETINA\VBZMOD_1.SP
Date Stamp	wed dec 11 17:27:05 2013 Horário brasileiro de verão (GMT-2:00)	Date	wed dec 11 17:29:17 2013 Horário brasileiro de verão (GMT-2:00)
Technique	Infrared	Instrument	Spectrum Two
X Axis	Wavenumber (cm-1)	Y Axis	%Transmittance
Points Count	3451	Data Spacing	1.0000
		Spectral Region	IR
		Spectrum Range	550.0000 - 4000.0000



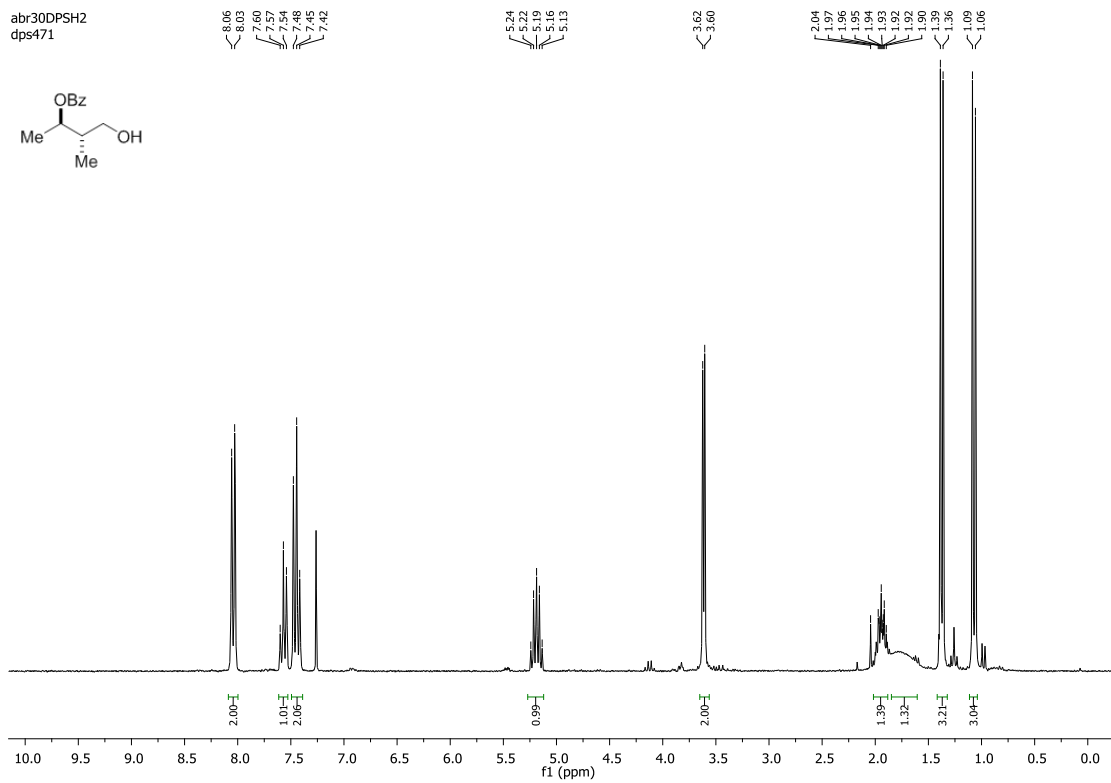
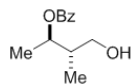
IR of 43

D010 #54 RT: 0.81 AV: 1 NL: 2.43E7
T: FTMS + p ESI Full ms [150.00-1500.00]



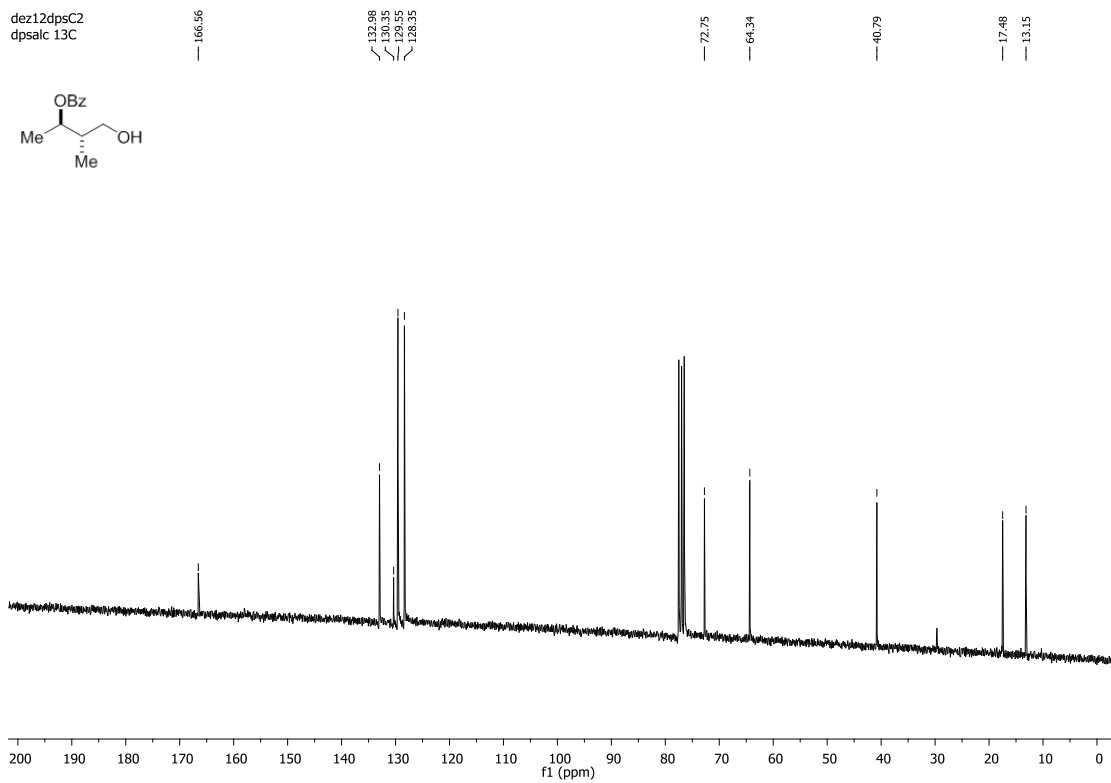
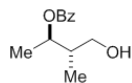
HRMS (ESI+) m/z calculated for $C_{20}H_{24}O_4Na$ $[M+Na]^+$ 351.1572

abr30DPSH2
dps471



¹H NMR of **44** (CDCl₃, 250 MHz)

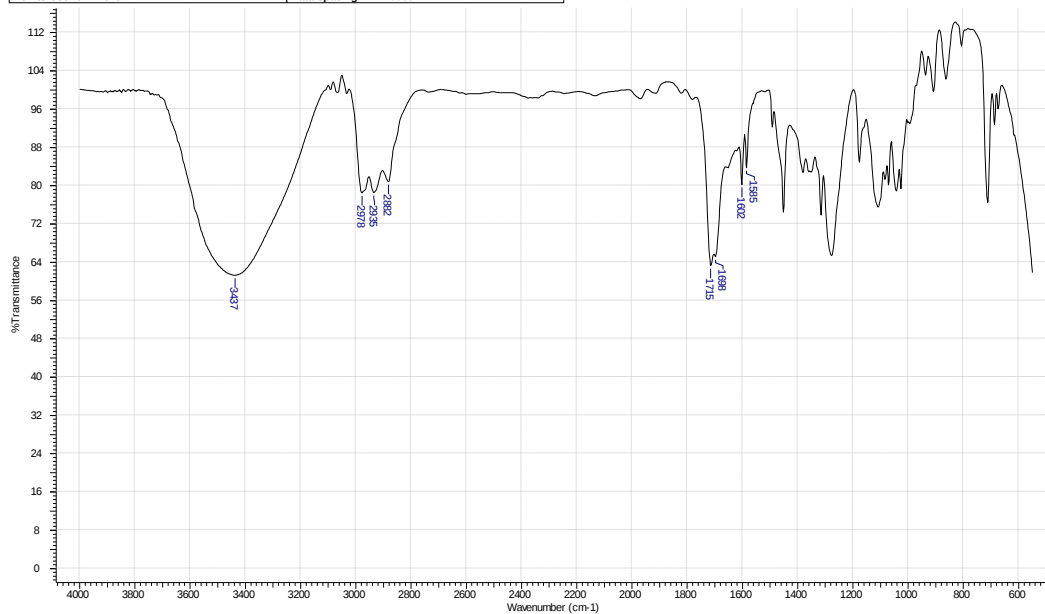
dez12dpsC2
dpsalc 13C



¹³C NMR **44** (CDCl₃, 62.5 MHz)

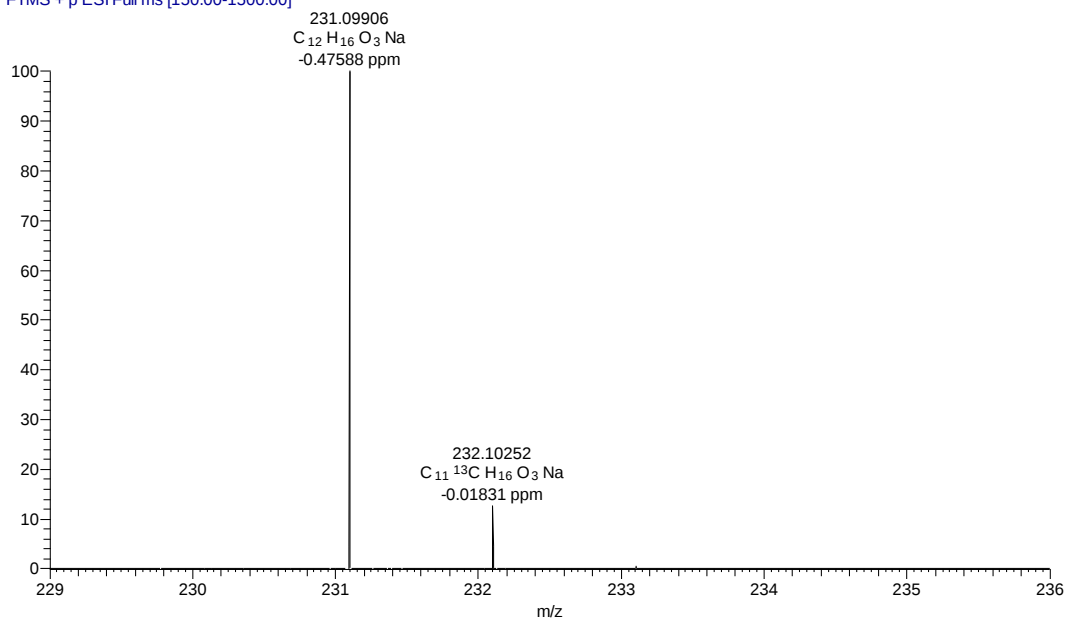
8 Jan 2014

Title	brasileiro de verão (GMT-2:00)	Comment	Amostra 001 por LQOS data quarta-feira, dezembro 11, 2013
Origin	brasileiro de verão (GMT-2:00)	File Name	C:\USERS\DANILO P SANTANA\DOCUMENTS\TSAUTOMICETINA\VALCOOLMOD.SP
Date Stamp	wed dec 11 17:19:18 2013 Horário brasileiro de verão (GMT-2:00)	Date	wed dec 11 17:20:42 2013 Horário brasileiro de verão (GMT-2:00)
Technique	Infrared	Instrument	Spectrum Two
X Axis	Wavenumber (cm-1)	Y Axis	%Transmittance
Points Count	3451	Data Spacing	1.0000
		Spectral Region	IR
		Spectrum Range	550.0000 - 4000.0000



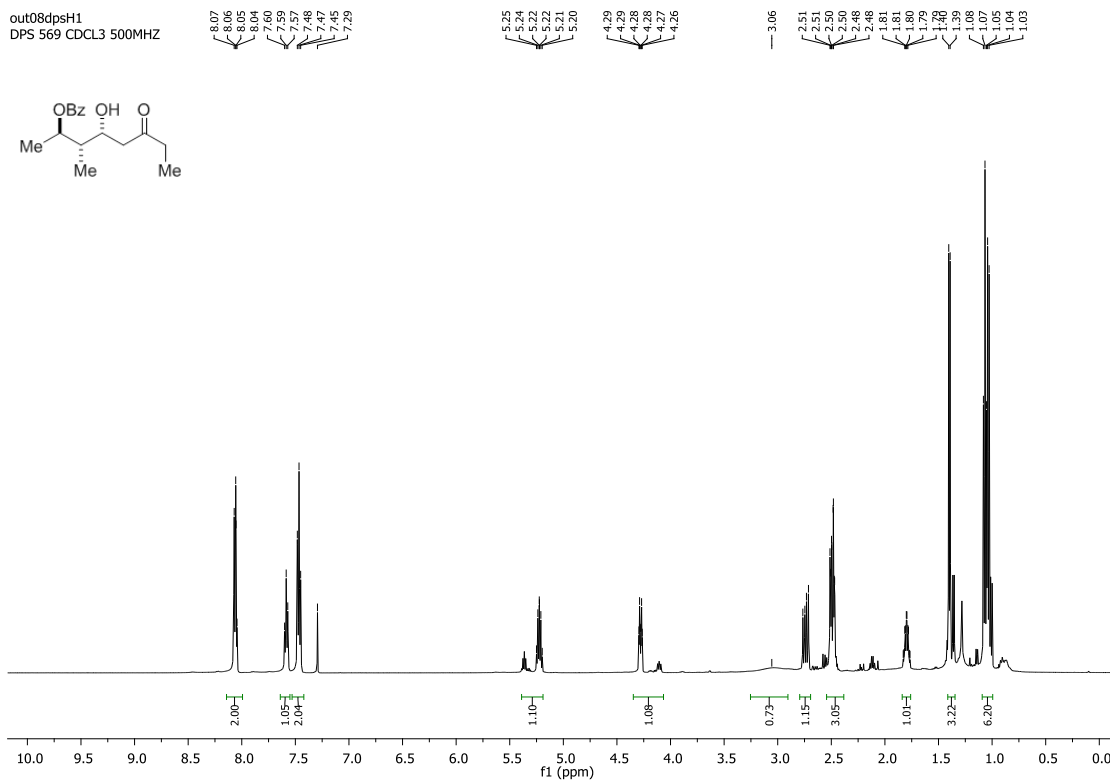
IR of 44

D011 #53 RT: 0.80 AV: 1 NL: 7.90E6
T: FTMS + p ESI Full ms [150.00-1500.00]



HRMS (ESI+) m/z calculated for $C_{12}H_{16}O_3Na$ $[M+Na]^+$ 231.0997

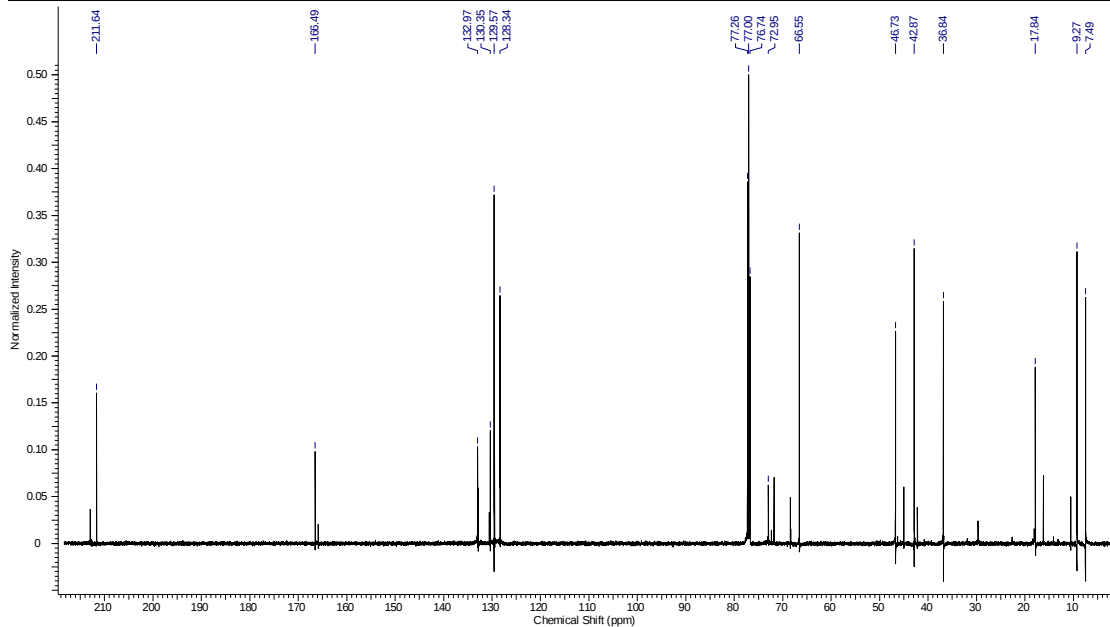
out08dpsH1
DPS 569 CDCL3 500MHZ



^1H NMR of **47** (CDCl_3 , 500 MHz)

08/01/2014 19:53:37

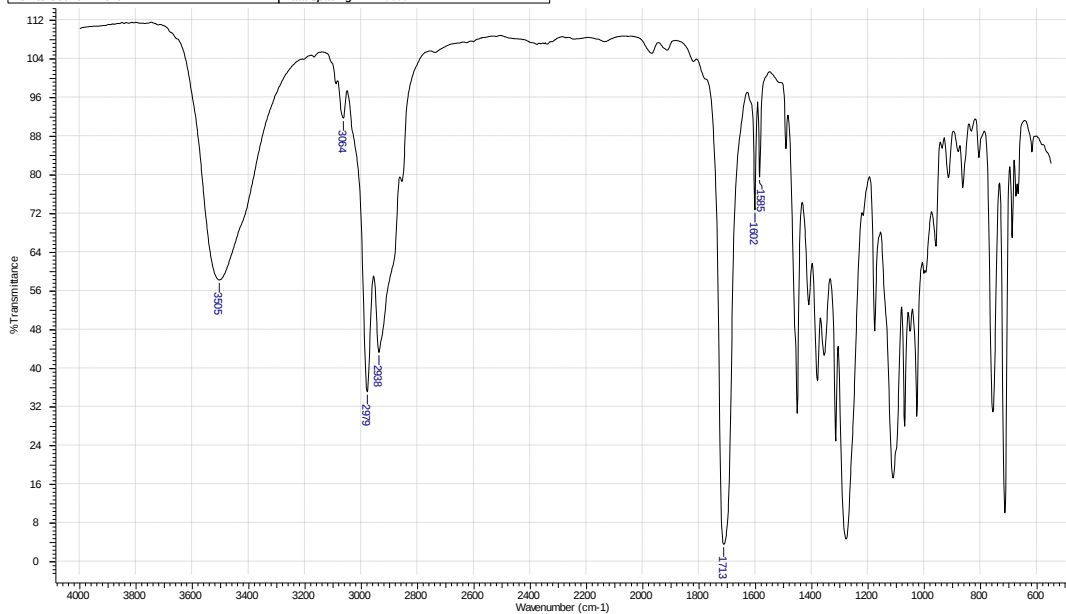
Acquisition Time (sec)	0.5505	Comment	DPS 569CDCL3 500MHz	Date	09 Oct 2013 03:24:00
Date Stamp	09 Oct 2013 03:24:00	File Name	C:\Users\Danilo P Sant\Ana\Documents\tautomicetina\fid\dps500\dps569 C 500mhz1.fid	Origin	spect
Frequency (MHz)	125.70	Nucleus	^{13}C	Number of Transients	2048
Original Points Count	16384	Owner	nmrsu	Points Count	16384
Receiver Gain	2050.00	SW(cyclical) (Hz)	29761.90	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	12561.1963	Spectrum Type	STANDARD	Sweep Width (Hz)	29760.09
				Temperature (degree C)	25.151



^{13}C NMR of **47** (CDCl_3 , 125 MHz)

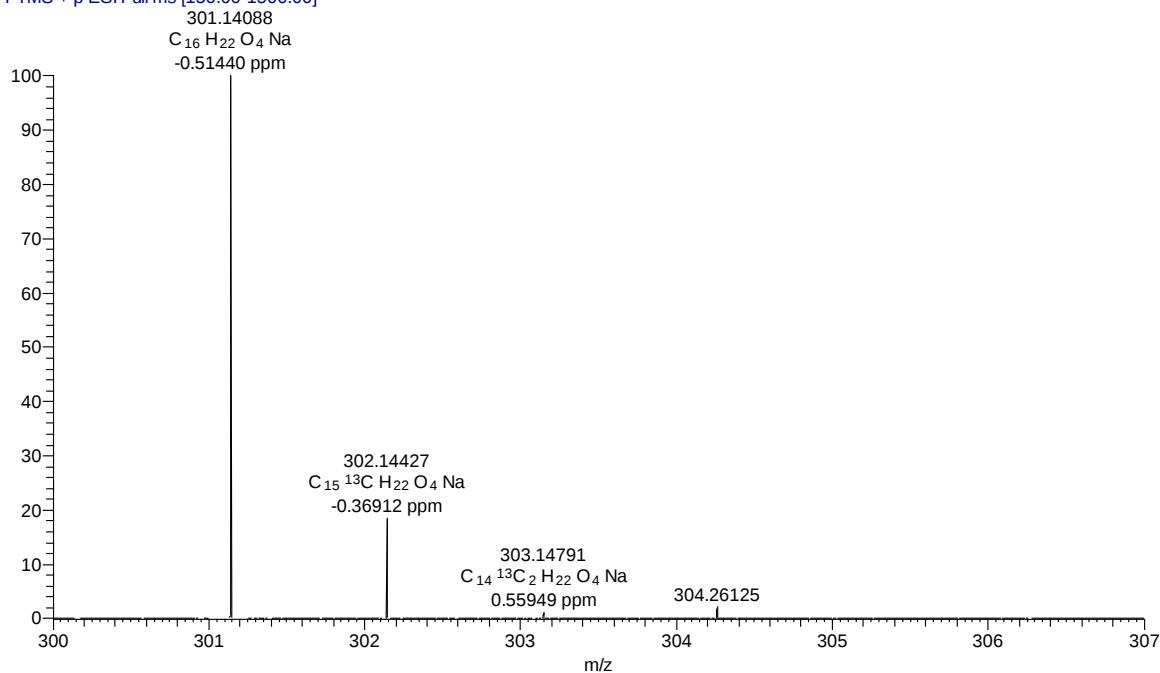
24 Sep 2014

Title	cial do Brasil (GMT-3:00)	Comment	Amostra 004 por LOQS data terça-feira, setembro 23 2014
Origin	cial do Brasil (GMT-3:00)	File Name	C:\USERS\DANILO P SANT'ANA\DOCUMENTS\ITESE 2014\DAI\LO\ADPSMUK.SP
Date Stamp	tue sep 23 14:57:37 2014 Hora oficial do Brasil (GMT-3:00)	Date	tue sep 23 14:57:37 2014 Hora oficial do Brasil (GMT-3:00)
Technique	Infrared	Instrument	Spectrum Two
X Axis	Wavenumber (cm-1)	Y Axis	%Transmittance
Points Count	3451	Data Spacing	1.0000
		Spectral Region	IR
		Spectrum Range	550.0000 - 4000.0000



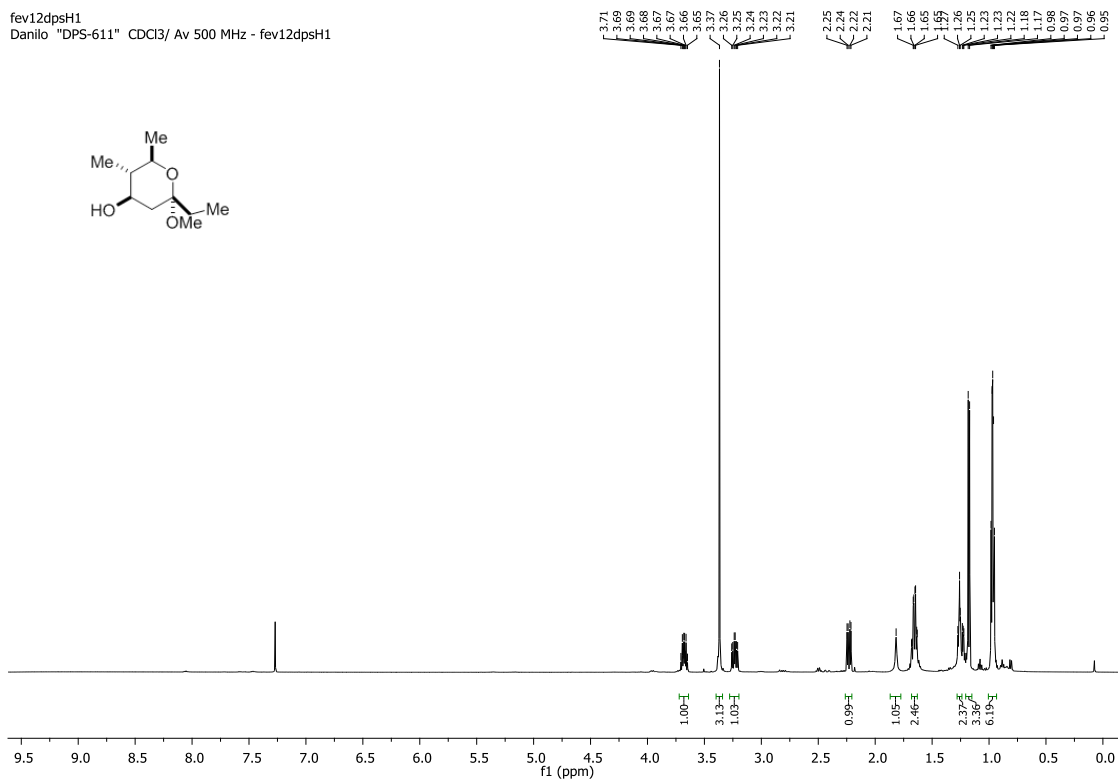
IR of 47

D008 #53 RT: 0.81 AV: 1 NL: 4.47E7
T: FTMS + p ESI Full ms [150.00-1500.00]



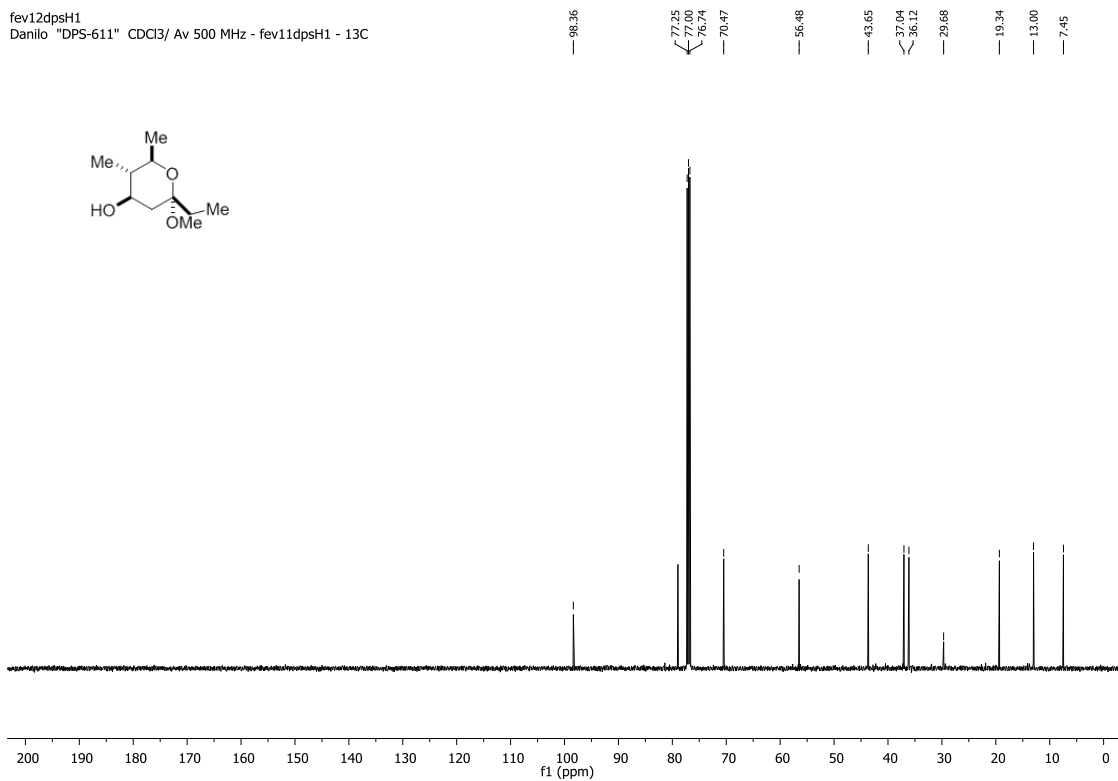
HRMS (ESI+) m/z calculated for $C_{16}H_{22}O_4Na$ $[M+Na]^+$ 301.1416

fev12dpsH1
 Danilo "DPS-611" CDCl₃/ Av 500 MHz - fev12dpsH1



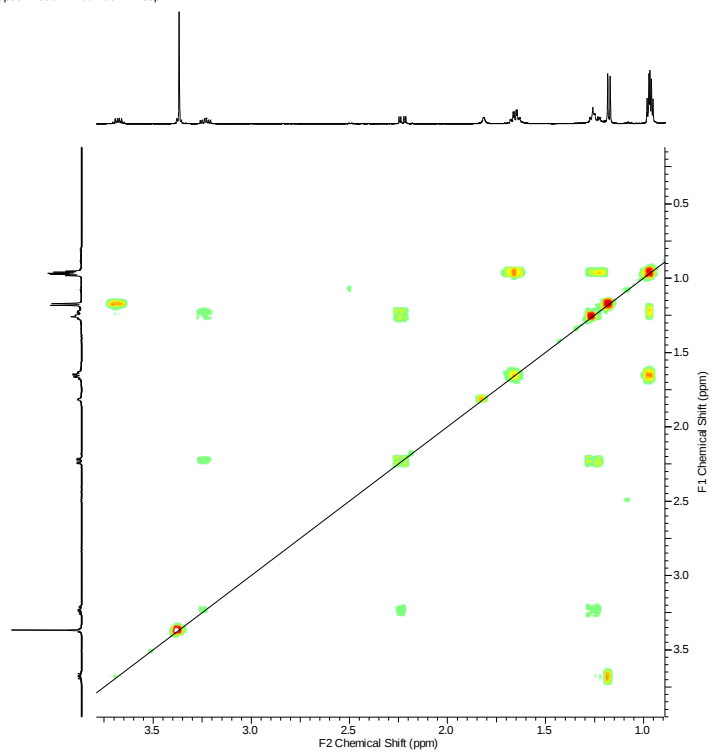
¹H NMR of **48** (CDCl₃, 500 MHz)

fev12dpsH1
 Danilo "DPS-611" CDCl₃/ Av 500 MHz - fev11dpsH1 - 13C



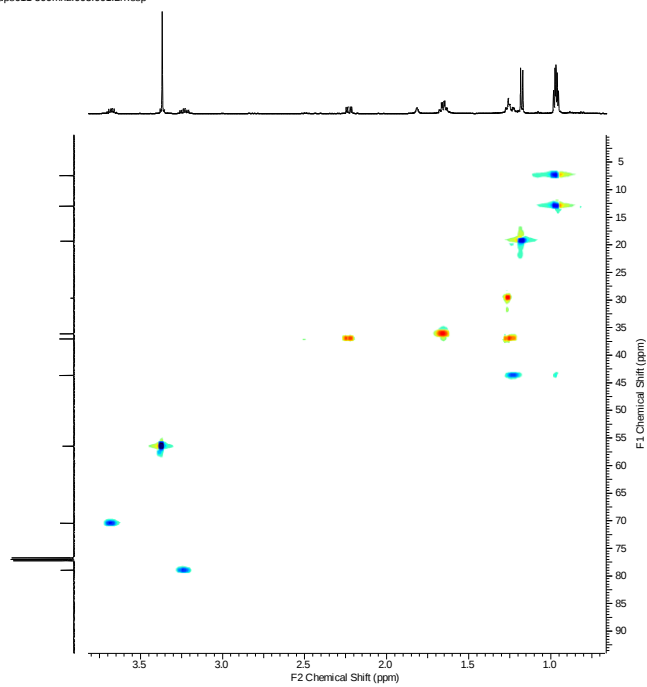
¹³C NMR of **48** (CDCl₃, 125 MHz)

dps611 500mhz.002.001.2rr.esp



COSY of **48**

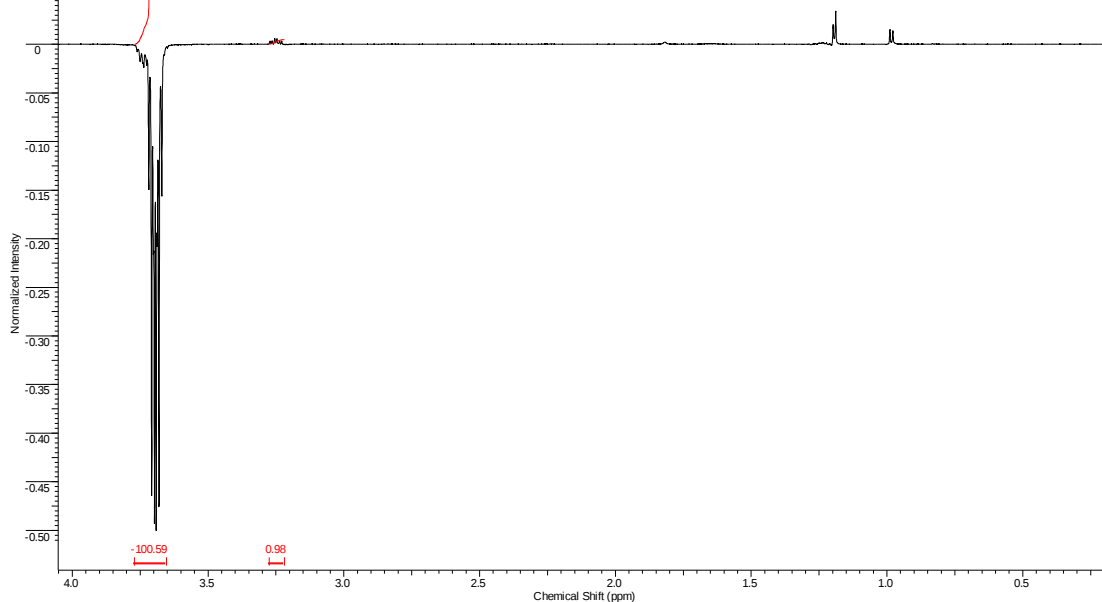
dps611 500mhz.003.001.2rr.esp



HSQC of **48**

24/09/2014 16:06:16

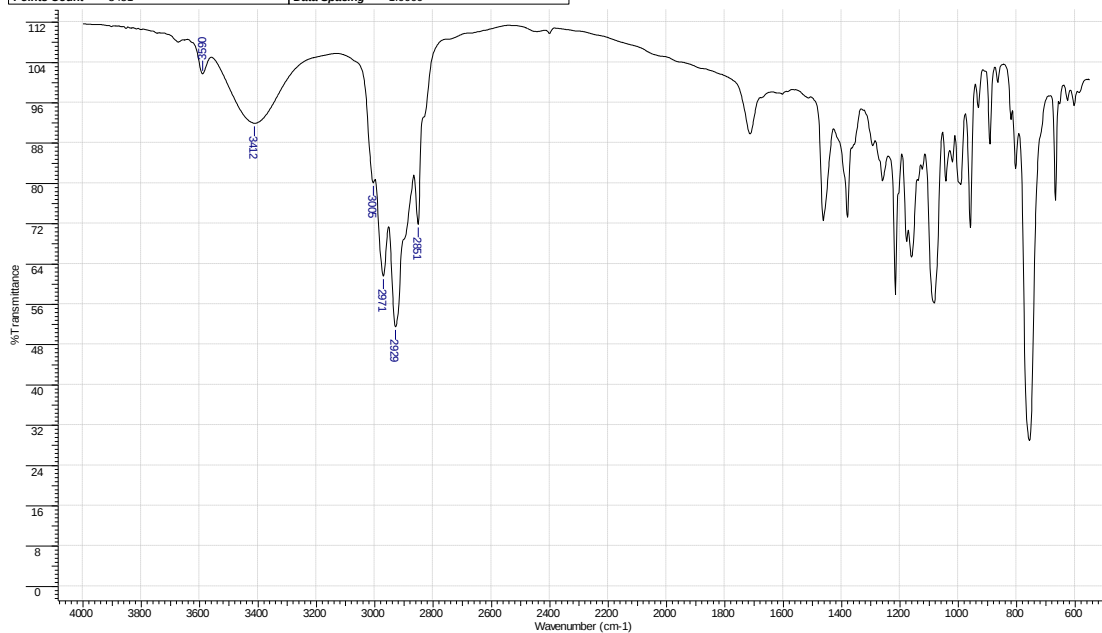
Acquisition Time (sec)	5.0725	Comment	Danilo "DPS-611" cdc13/ Av 600 MHz - mar11dpsH1 NOE seletivo em 3.37ppm		
Date	19 Mar 2014 15:31:28	Date Stamp	19 Mar 2014 15:31:28		
File Name	C:\Users\Danilo P Sant\Ana\Documents\tautomicetina\fid\dps600\dps611 2 600mhz\3\data\1\1r	Frequency (MHz)	600.17		
Nucleus	¹ H	Number of Transients	512	Origin	spect
Owner	nmrsu	Points Count	65536	Pulse Sequence	selhpgp
SW(cyclical) (Hz)	6459.95	Solvent	CDC13	Spectrum Offset (Hz)	2647.7092
Temperature (degree C)	25.151	Receiver Gain	90.50		
		Sweep Width (Hz)	6459.85		



NOE of 48

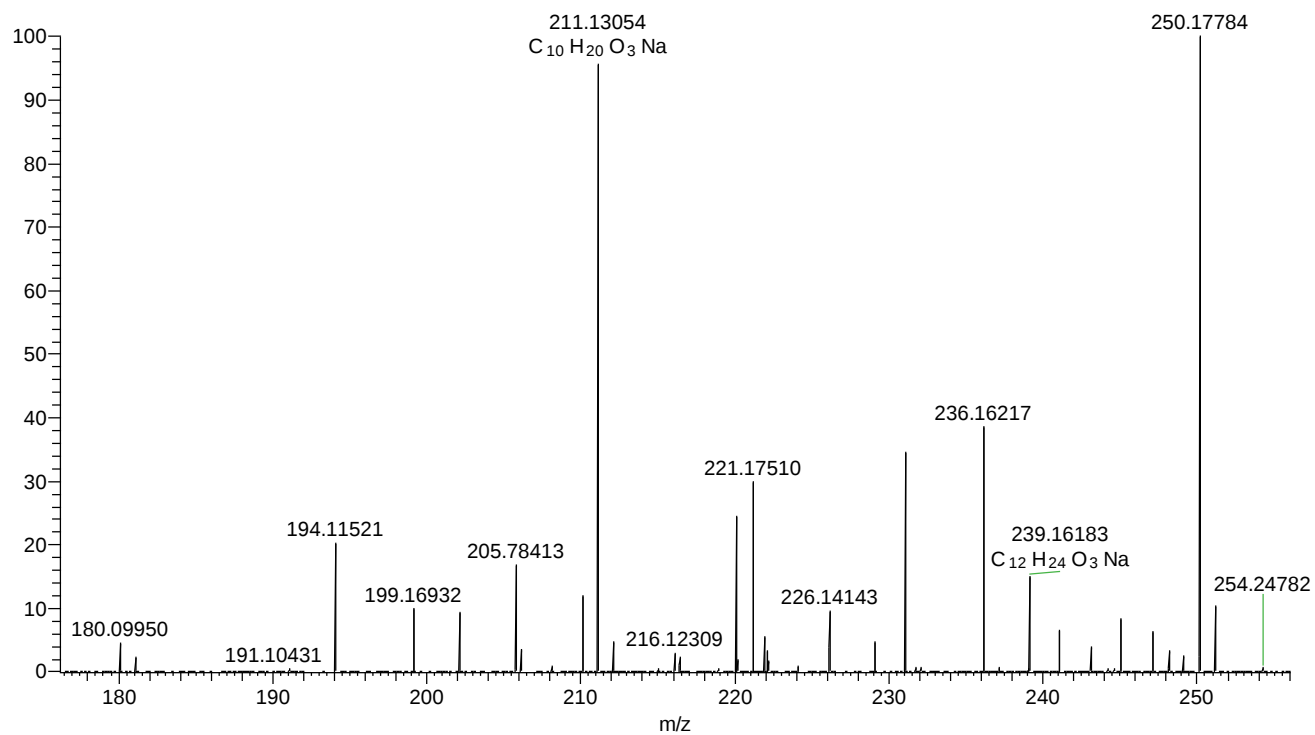
24 Sep 2014

Title	cial do Brasil (GMT-3:00)	Comment	Amostra 004 por LQOS data terça-feira, setembro 23 2014		
Origin	cial do Brasil (GMT-3:00)	File Name	C:\USERS\DANILO P SANT\ANA\DOCUMENTS\ITSE 2014\IDANILO IVDPS611.SP		
Date Stamp	tue sep 23 15:21:13 2014 Hora oficial do Brasil (GMT-3:00)	Date	tue sep 23 15:21:13 2014 Hora oficial do Brasil (GMT-3:00)		
Technique	Infrared	Instrument	Spectrum Two	Spectral Region	IR
X Axis	Wavenumber (cm-1)	Y Axis	%Transmittance	Spectrum Range	550.0000 - 4000.0000
Points Count	3451	Data Spacing	1.0000		



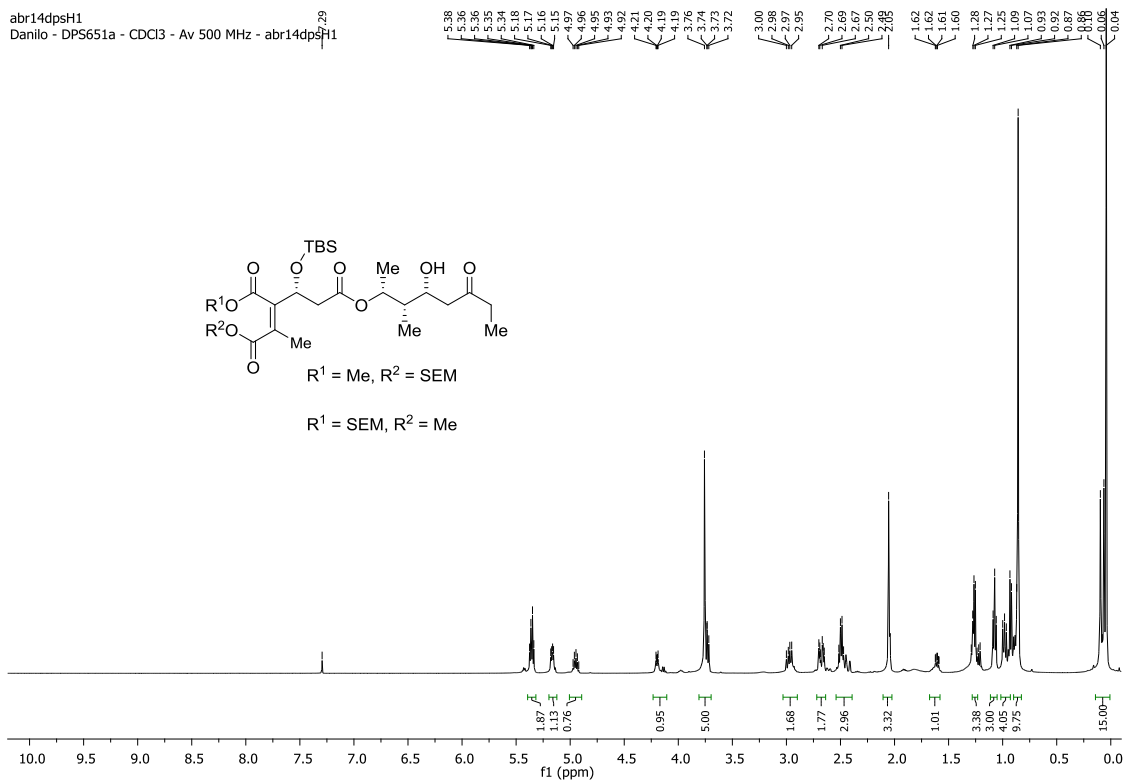
IR of 48

D012 #7-65 RT: 0.10-0.98 AV: 59 NL: 7.38E4
T: FTMS + p ESI Full ms [150.00-1500.00]



HRMS (ESI+) m/z calculated for $C_{10}H_{20}O_3Na$ $[M+Na]^+$ 211.1310

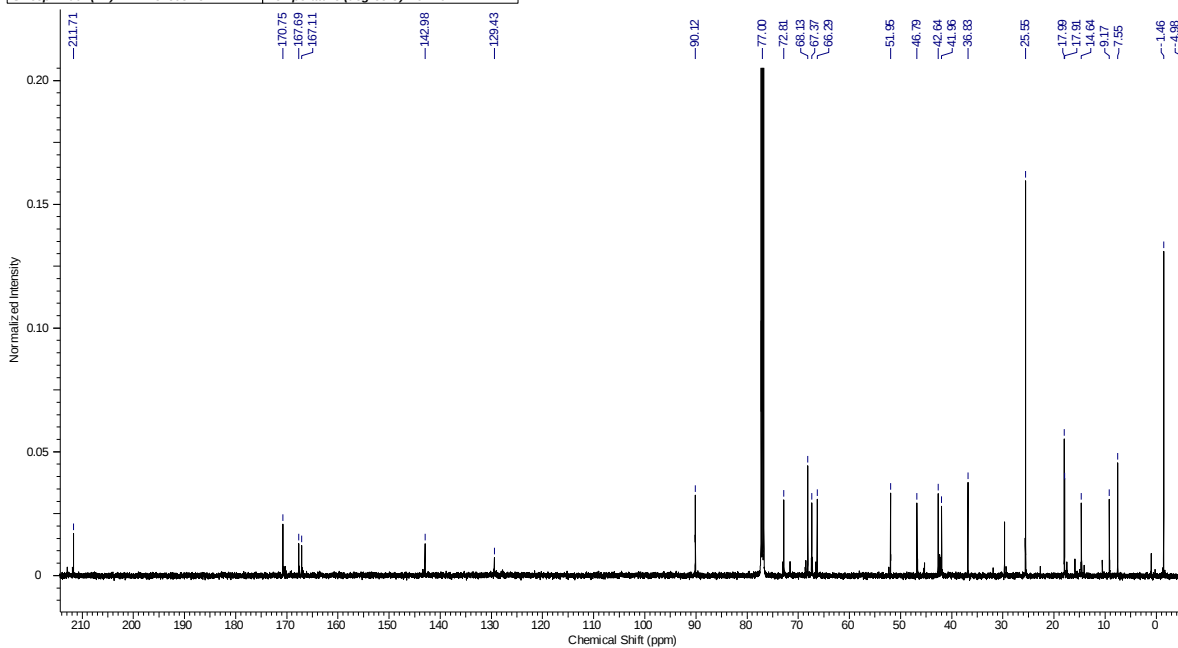
abr14dpsH1
Danilo - DPS651a - CDCl₃ - Av 500 MHz - abr14dpsH1



¹H NMR **50** (CDCl₃, 500 MHz)

25/09/2014 11:57:10

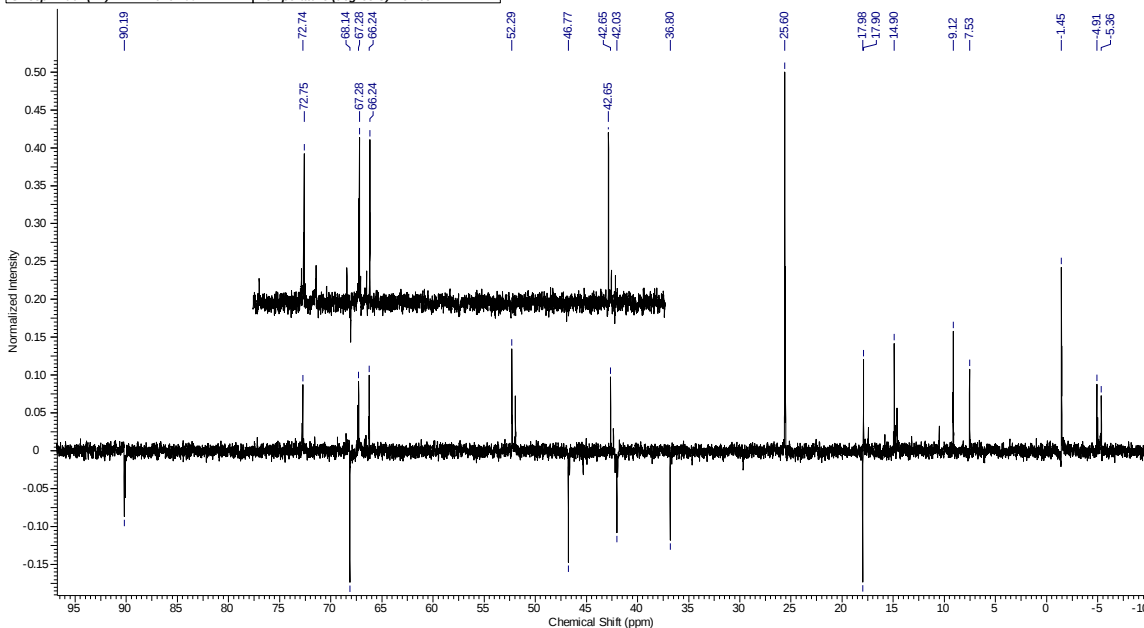
Acquisition Time (sec)	0.4981	Comment	Danilo - DPS 672a - CDCl ₃ - Avance 500 MHz - mai16dpsH1 - 13C
Date	16 May 2014 09:22:24	Date Stamp	16 May 2014 09:22:24
File Name	C:\Users\Danilo P Sant\Ana\Documents\tautomicetinal\ddps600-vdps672\2\data\111r	Frequency (MHz)	125.69
Nucleus	13C	Number of Transients	3072
Owner	nmrsu	Points Count	32768
SW(cyclical) (Hz)	32894.74	Solvent	CDCl ₃
Sweep Width (Hz)	32893.73	Temperature (degree C)	25.149
		Pulse Sequence	zgpg30
		Spectrum Offset (Hz)	13821.7793
		Receiver Gain	2050.00
		Spectrum Type	STANDARD



¹³C NMR of **50** (CDCl₃, 125 MHz)

24/09/2014 21:45:22

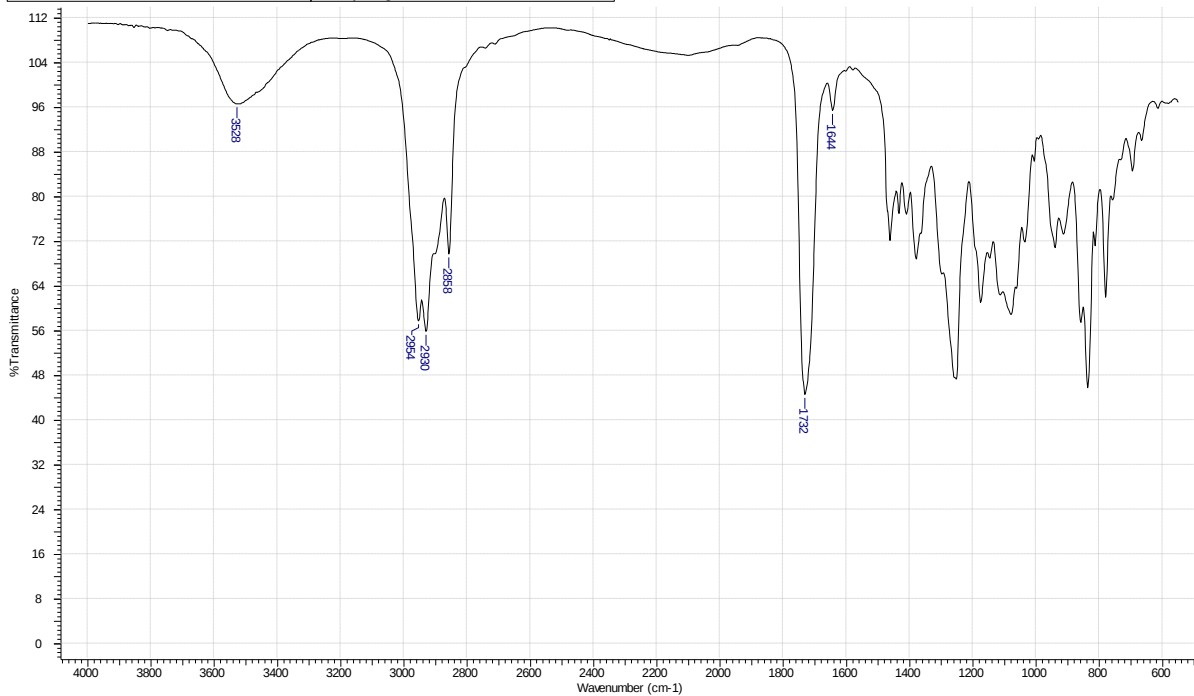
Acquisition Time (sec)	0.5505	Comment Danilo - DPS 624 - CDCI3 - Avance 500 MHz - fev26dpsH1 - DEPT 135					
Date	26 Feb 2014 09:35:12	Date Stamp	26 Feb 2014 09:35:12				
File Name	C:\Users\Danilo P Sant Ana\Documents\1automicetina\fidhdps600\1dps624\3\data\111r					Frequency (MHz)	125.69
Nucleus	13C	Number of Transients	360	Origin	spect	Original Points Count	16384
Owner	nmrsu	Points Count	32768	Pulse Sequence	dept135	Receiver Gain	2050.00
SW(cyclical) (Hz)	29761.90	Solvent	CDCI3	Spectrum Offset (Hz)	12564.1885	Spectrum Type	DEPT135
Sweep Width (Hz)	29761.00	Temperature (degree C)	25.163				



DEPT 90 and 135 of 50

24 Sep 2014

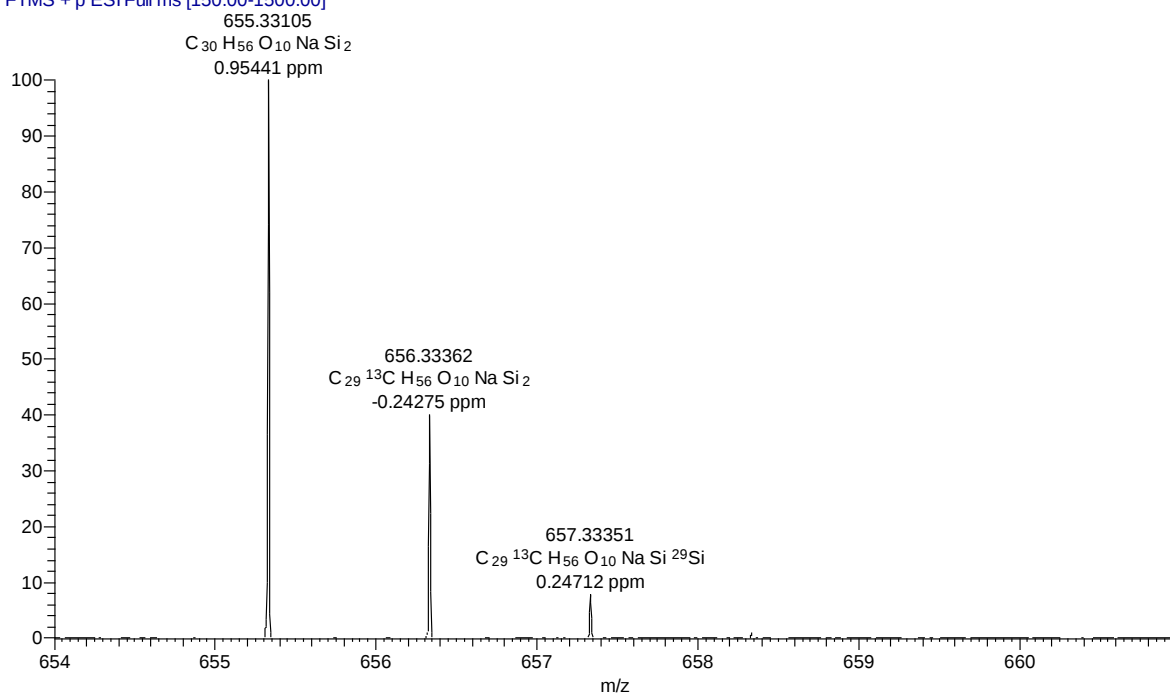
Title	cial do Brasil (GMT-3:00)	Comment	Amostra 005 por LQOS data terça-feira, setembro 23 2014		
Origin	cial do Brasil (GMT-3:00)	File Name	C:\USERS\DANILO P SANT'ANA\DOCUMENTS\SITESE 2014\1DANILO IVDPS651.SP		
Date Stamp	tue sep 23 15:32:09 2014 Hora oficial do Brasil (GMT-3:00)	Date	tue sep 23 15:32:09 2014 Hora oficial do Brasil (GMT-3:00)		
Technique	Infrared	Instrument	Spectrum Two	Spectral Region	IR
X Axis	Wavenumber (cm-1)	Y Axis	%Transmittance	Spectrum Range	550.0000 - 4000.0000
Points Count	3451	Data Spacing	1.0000		



IR of 50

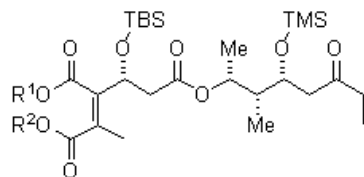
S73

D002 #17 RT: 0.25 AV: 1 NL: 7.73E7
T: FTMS + p ESI Full ms [150.00-1500.00]

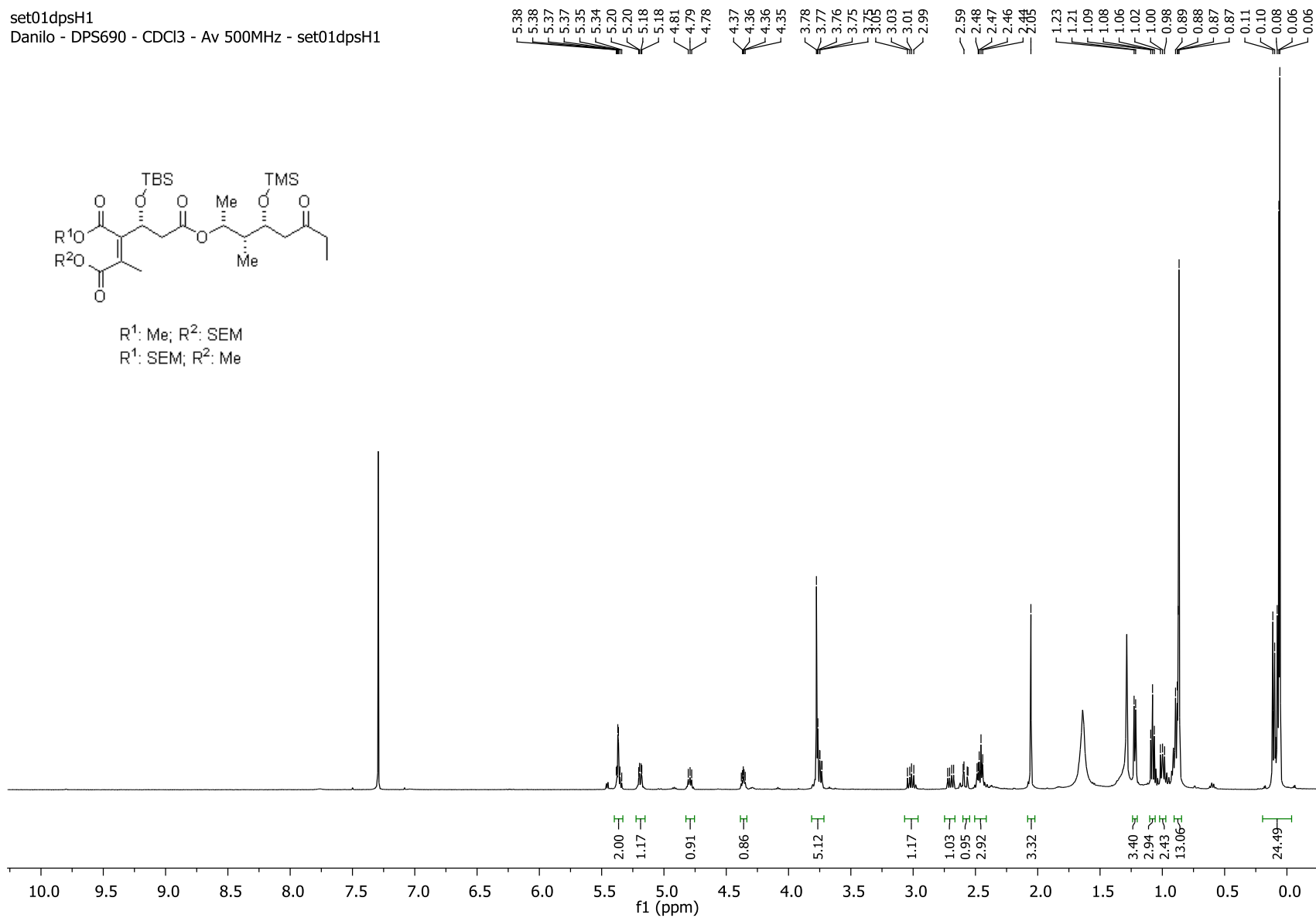


HRMS (ESI+) m/z calculated for $C_{30}H_{56}O_{10}NaSi_2$ $[M+Na]^+$ 655.3310

set01dpsH1
 Danilo - DPS690 - CDCl3 - Av 500MHz - set01dpsH1

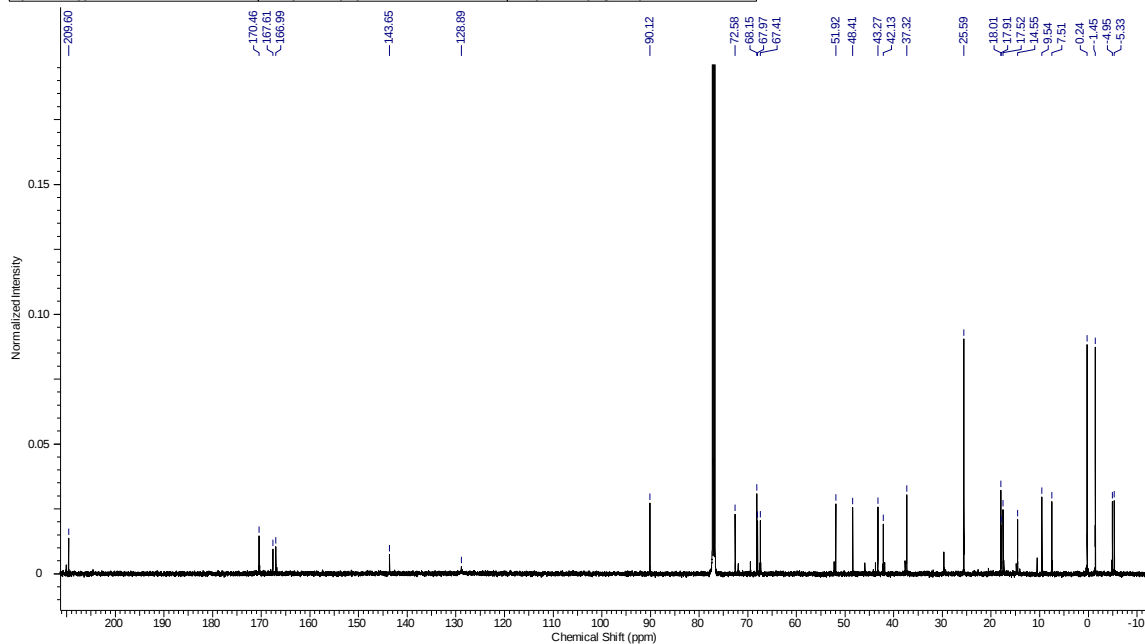


R¹: Me; R²: SEM
 R¹: SEM; R²: Me



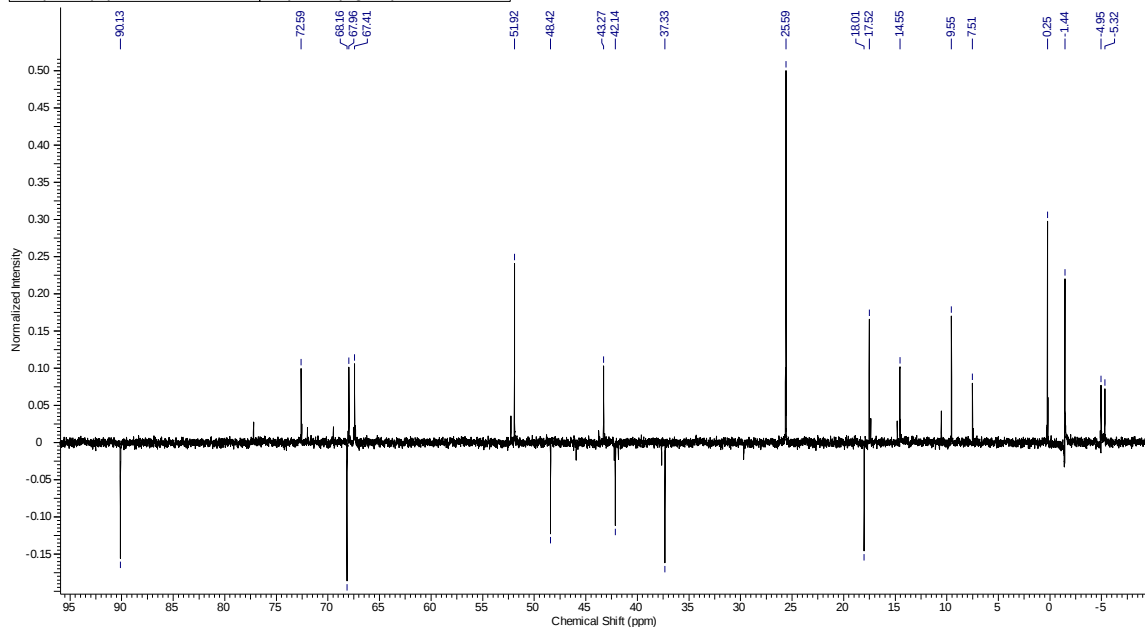
¹H NMR of fragment C13-C25 (CDCl₃, 500 MHz)
 S75

Acquisition Time (sec)	0.5505	Comment	Danilo - DPS 642 - CDCl ₃ - Avance 500 MHz - ago25dpsH1 - 13C	Date	25 Aug 2014 13:00:00
Date Stamp	25 Aug 2014 13:00:00	File Name	C:\Users\Danilo P Sant\Ana\Documents\tautomicetinal\fid\dps600-ldps642 500mhz\2\pdata1\1r	Frequency (MHz)	125.69
Frequency (MHz)	125.69	Nucleus	13C	Number of Transients	5120
Original Points Count	16384	Owner	nmrsu	Points Count	32768
Receiver Gain	2050.00	SW(cyclical) (Hz)	29761.90	Solvent	CDCl ₃
Spectrum Type	STANDARD	Sweep Width (Hz)	29761.00	Temperature (degree C)	25.151
				Pulse Sequence	zgpg30
				Spectrum Offset (Hz)	12566.1318



¹³C NMR of fragment C13-C25 (CDCl₃, 125 MHz)

Acquisition Time (sec)	0.5505	Comment	Danilo - DPS 642 - CDCl ₃ - Avance 500 MHz - ago25dpsH1 - DEPT 135	Date Stamp	25 Aug 2014 16:29:04
Date	25 Aug 2014 16:29:04	File Name	C:\Users\Danilo P Sant\Ana\Documents\tautomicetinal\fid\dps600-ldps642 500mhz\3\pdata1\1r	Frequency (MHz)	125.69
File Name	C:\Users\Danilo P Sant\Ana\Documents\tautomicetinal\fid\dps600-ldps642 500mhz\3\pdata1\1r	Nucleus	13C	Number of Transients	1024
Nucleus	13C	Owner	nmrsu	Points Count	32768
Original Points Count	16384	SW(cyclical) (Hz)	29761.90	Solvent	CDCl ₃
Receiver Gain	2050.00	Spectrum Offset (Hz)	12567.4727	Spectrum Type	DEPT135



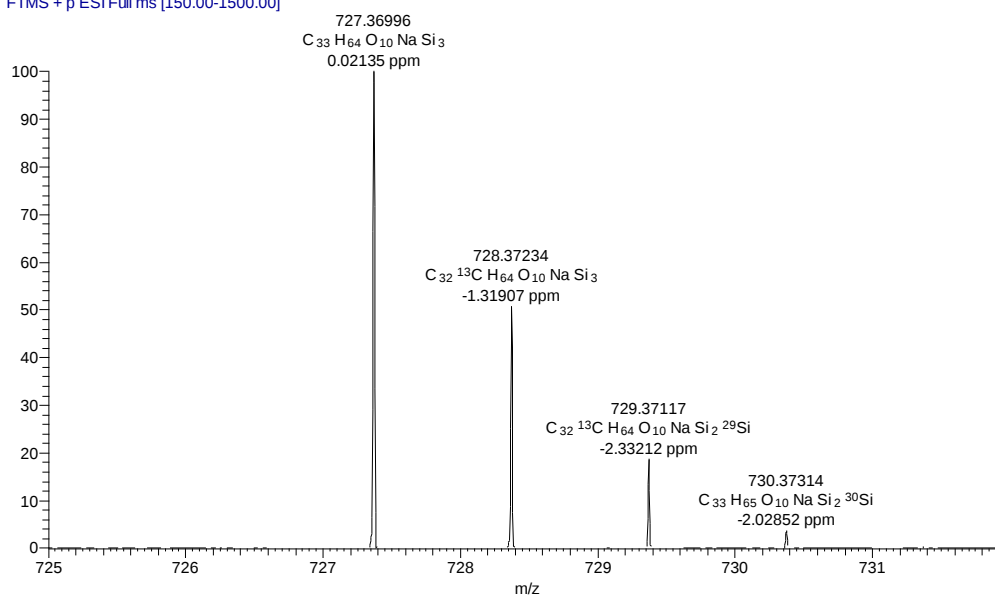
DEPT 135 of fragment C13-C25 (CDCl₃, 125 MHz)

Title	cial do Brasil (GMT-3:00)	Comment	Amostra 006 por LQOS data terça-feira, setembro 23 2014
Origin	cial do Brasil (GMT-3:00)	File Name	C:\USERS\DANILO P. SANT'ANA\DOCUMENTS\ITESE 2014\DANILO IV\DP672.SP
Date Stamp	tue sep 23 15:43:19 2014 Hora oficial do Brasil (GMT-3:00)	Date	tue sep 23 15:43:19 2014 Hora oficial do Brasil (GMT-3:00)
Technique	Infrared	Instrument	Spectrum Two
X Axis	Wavenumber (cm-1)	Y Axis	%Transmittance
Points Count	3451	Data Spacing	1.0000
		Spectral Region	IR
		Spectrum Range	550.0000 - 4000.0000



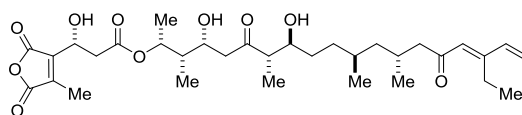
IR of fragment C13-C25

D015 #60 RT: 0.90 AV: 1 NL: 1.59E8
T: FTMS + p ESI Full ms [150.00-1500.00]



HRMS (ESI+) m/z calculated for $C_{33}H_{64}O_{10}NaSi_3$ $[M+Na]^+$ 727.3705

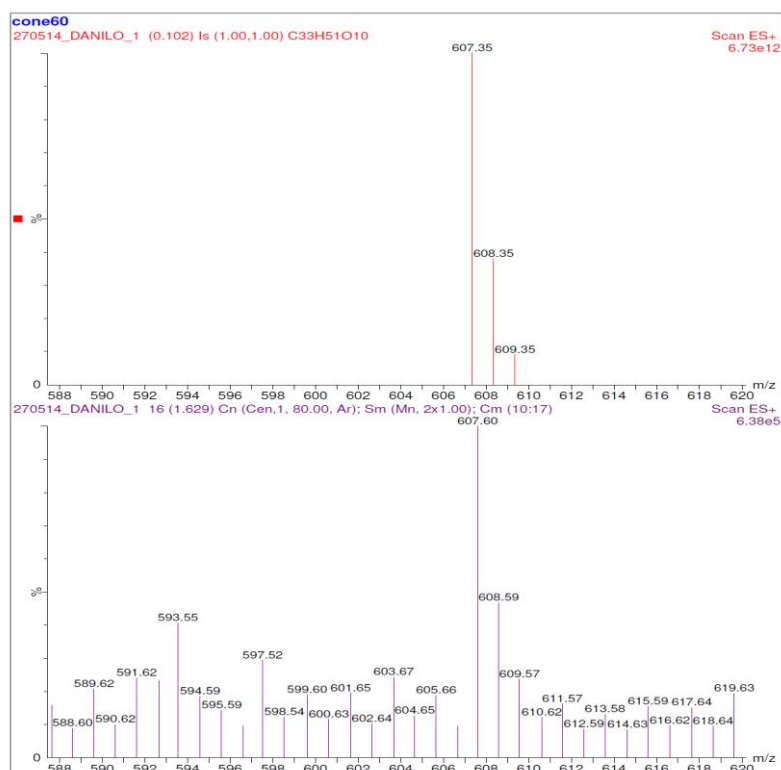
Some evidences concerning the formation of TTN



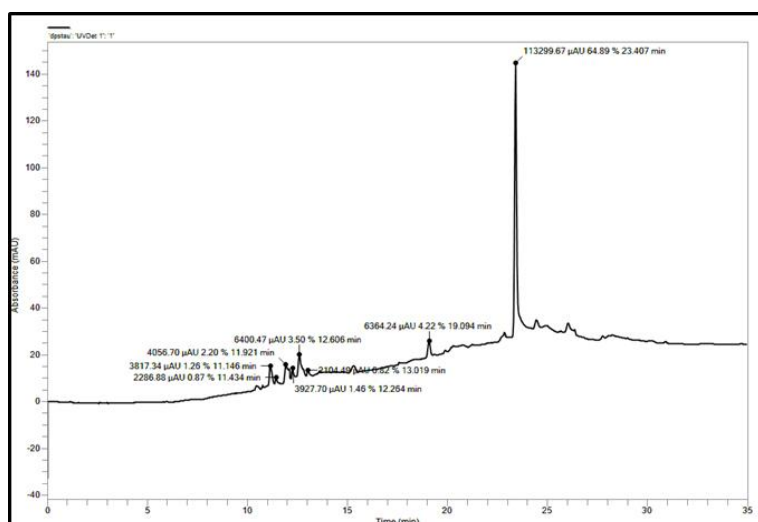
TTN (1)

Chemical Formula: $C_{33}H_{50}O_{10}$

Exact Mass: 606,3404



Mass Spectra of crude material



HPLC profile; for comparison see literature data¹

¹ Yang, D.; Li, W.; Huang, S.-X.; Shen, B. Functional Characterization of *ttnI* Completing the Tailoring Steps for Tautomycetin Biosynthesis in *Streptomyces griseochromogenes*. *Org. Lett.* **2012**, *14*, 1302-1305.