

Supporting Information

Synthesis of the decalin core of codinaeopsin via an intramolecular Diels-Alder reaction

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checkCIF/PLATON report

You have not supplied any structure factors. As a result the full set of tests cannot be run.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 12ma23_0ma

Bond precision: C-C = 0.0027 Å

Wavelength=0.71073

Cell: a=14.0891(6) b=12.3368(5) c=14.0775(6)
 alpha=90 beta=117.268(1) gamma=90
Temperature: 296 K

	Calculated	Reported
Volume	2174.96(16)	2174.96(16)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C22 H30 N4 O4	?
Sum formula	C22 H30 N4 O4	C22 H30 N4 O4
Mr	414.50	414.50
Dx, g cm ⁻³	1.266	1.266
Z	4	4
Mu (mm ⁻¹)	0.088	0.088
F000	888.0	888.0
F000'	888.39	
h, k, lmax	18, 16, 18	18, 16, 18
Nref	5492	5405
Tmin, Tmax	0.987, 0.991	0.984, 0.991
Tmin'	0.984	

Correction method= MULTI-SCAN

Data completeness= 0.984

Theta(max)= 28.460

R(reflections)= 0.0482(3025)

wR2(reflections)= 0.1413(5405)

S = 1.026

Npar= 276

The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.

Alert level C

PLAT230_ALERT_2_C	Hirshfeld Test	Diff for N3 -- C11 ..	5.9 su
PLAT242_ALERT_2_C	Check Low	Ueq as Compared to Neighbors for	N3
PLAT242_ALERT_2_C	Check Low	Ueq as Compared to Neighbors for	N4

● Alert level G

PLAT005_ALERT_5_G	No _iucr_refine_instructions_details in CIF	?
PLAT007_ALERT_5_G	Note: Number of Unrefined D-H Atoms	1
PLAT793_ALERT_4_G	The Model has Chirality at C2 (Verify)	S
PLAT793_ALERT_4_G	The Model has Chirality at C4 (Verify)	S
PLAT793_ALERT_4_G	The Model has Chirality at C5 (Verify)	R
PLAT793_ALERT_4_G	The Model has Chirality at C6 (Verify)	R
PLAT793_ALERT_4_G	The Model has Chirality at C12 (Verify)	R
PLAT793_ALERT_4_G	The Model has Chirality at C17 (Verify)	R

-
- 0 **ALERT level A** = Most likely a serious problem - resolve or explain
 - 0 **ALERT level B** = A potentially serious problem, consider carefully
 - 3 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
 - 8 **ALERT level G** = General information/check it is not something unexpected

- 0 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
 - 3 ALERT type 2 Indicator that the structure model may be wrong or deficient
 - 0 ALERT type 3 Indicator that the structure quality may be low
 - 6 ALERT type 4 Improvement, methodology, query or suggestion
 - 2 ALERT type 5 Informative message, check
-

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

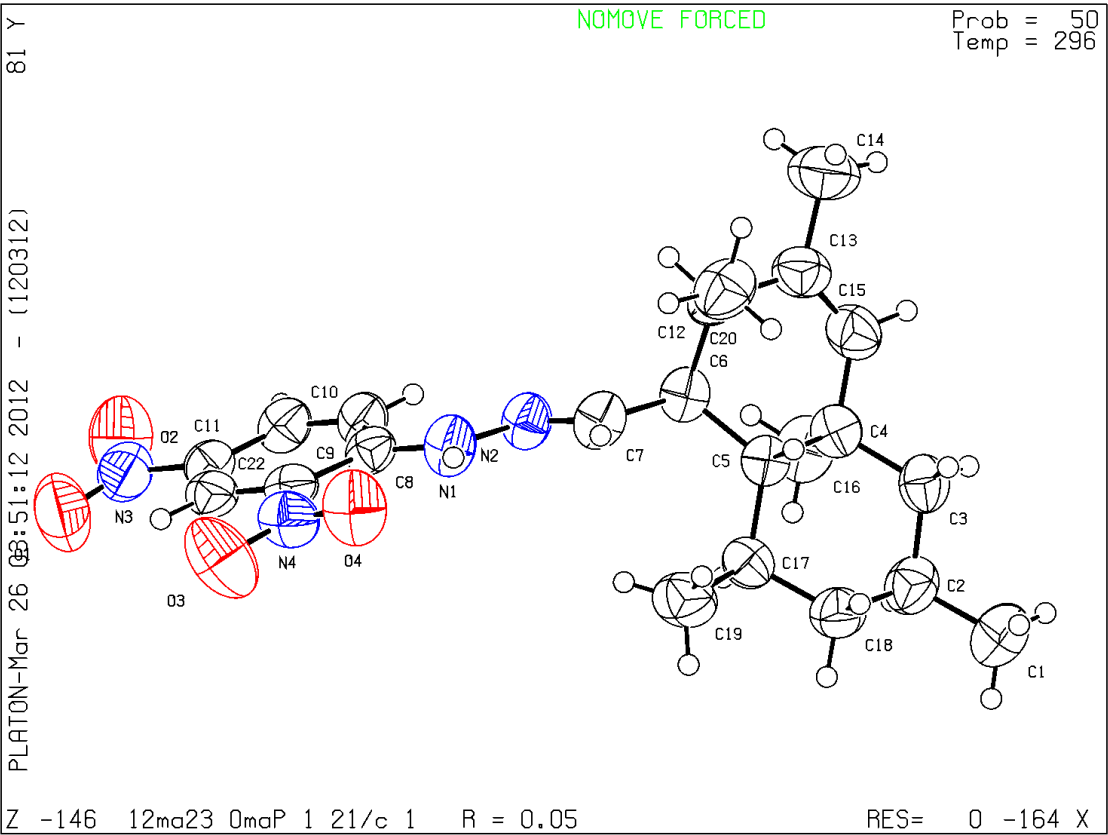
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

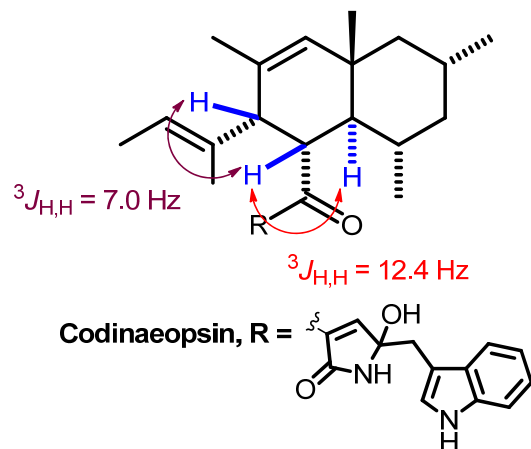
Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 12/03/2012; check.def file version of 10/02/2012

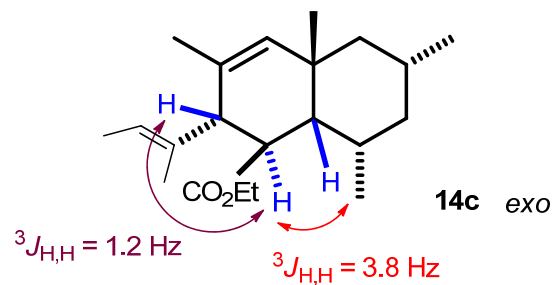
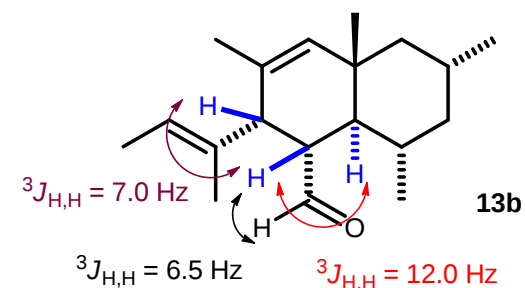
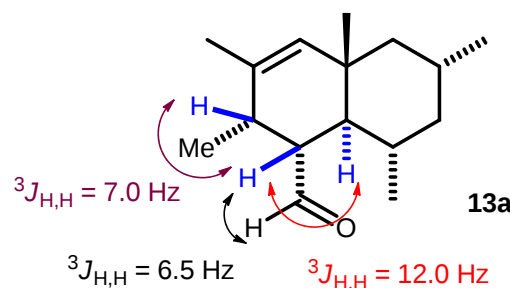
Datablock 12ma23_0ma - ellipsoid plot



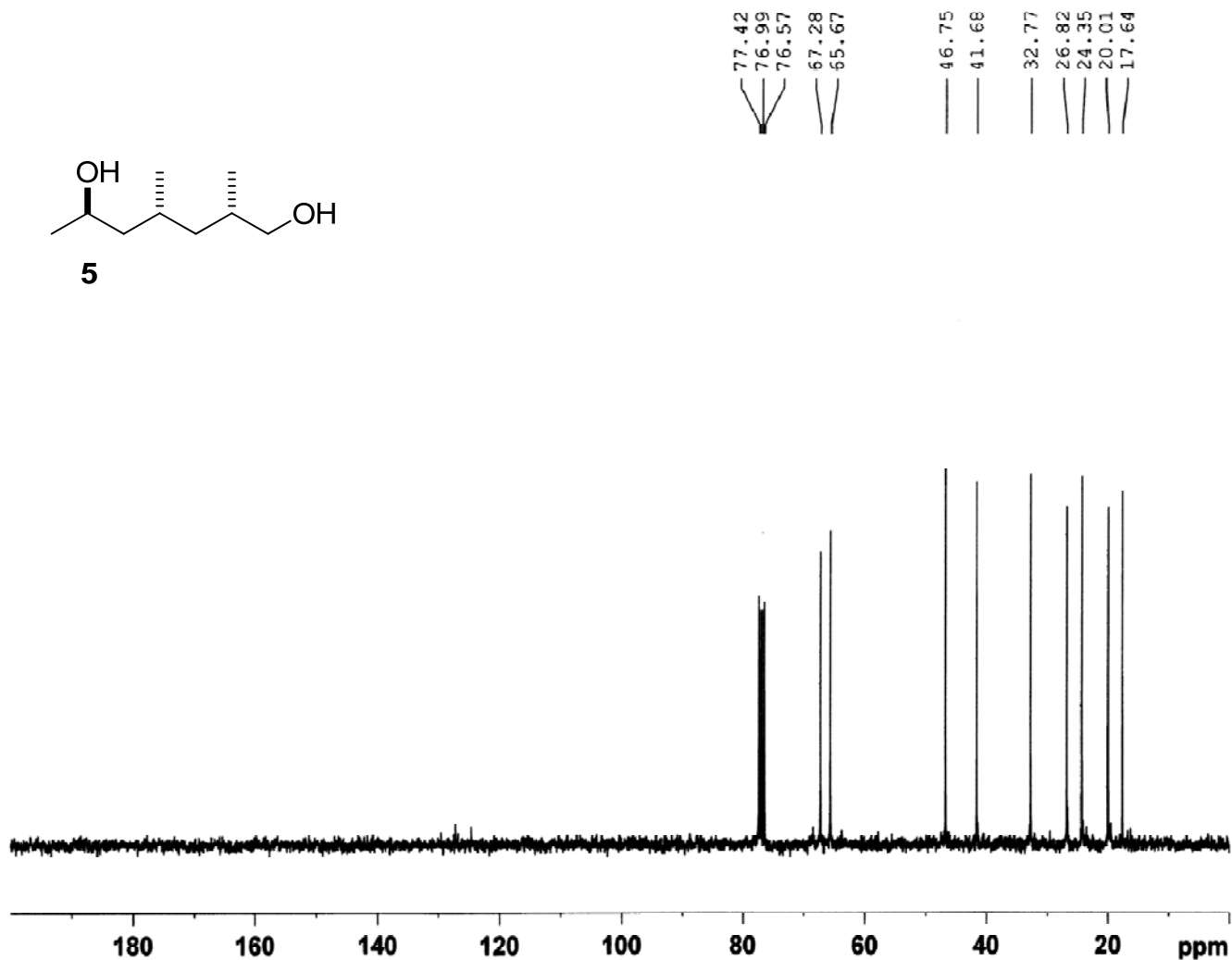
Observed coupling constants of *exo* and *endo* IMDA cycloadducts.



Kontnik. R.; Clardy. J. *Org.Lett.* **2008**,10, 4149



Diol



Current Data Parameters
NAME ramnath 30-09-09
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090930
Time 19.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl₃
NS 64
DS 0
SWH 17857.143 Hz
FIDRES 0.344937 Hz
AQ 0.9175540 sec
RG 1150
RW 28.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

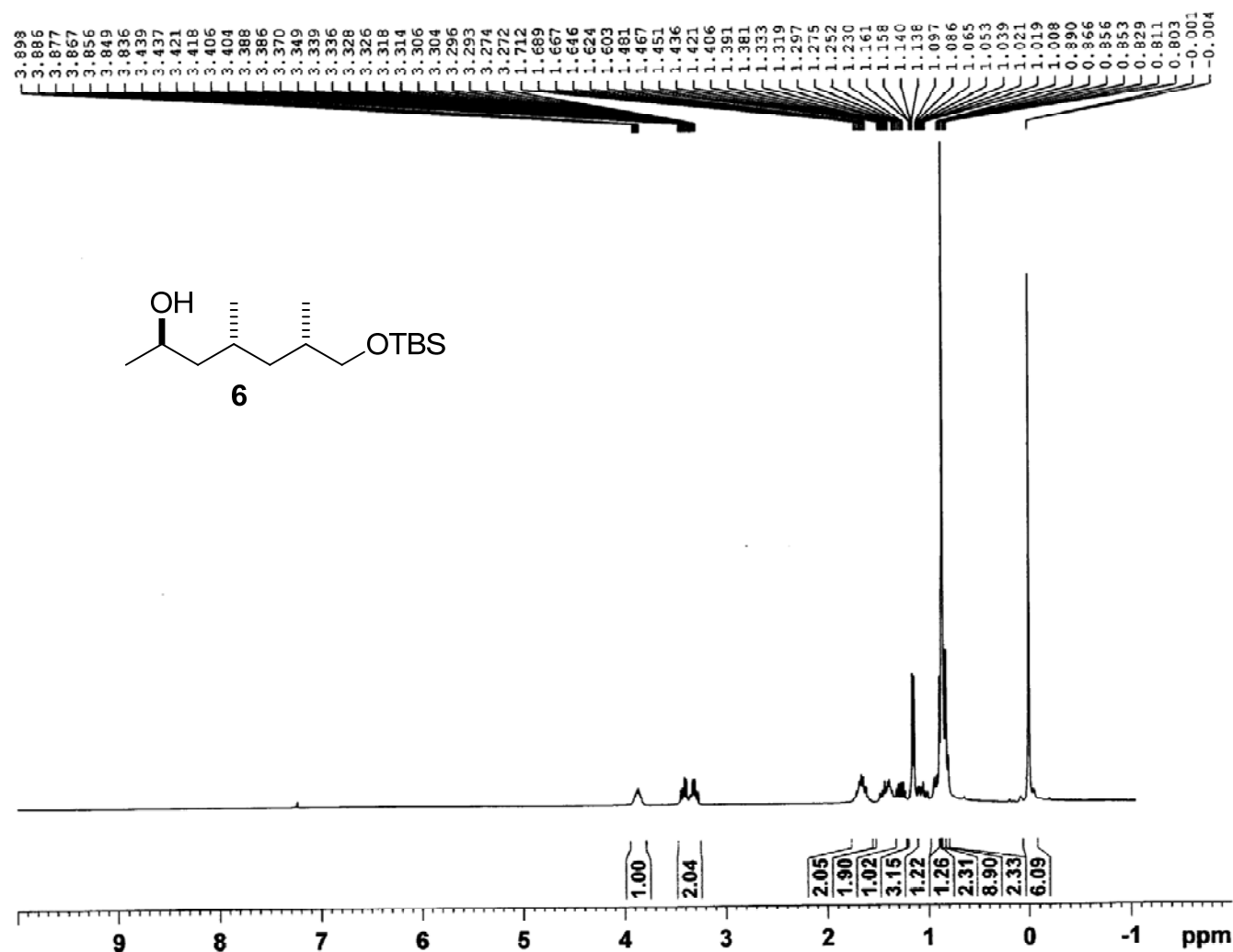
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P1 9.85 usec
PL1 0.00 dB
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NUC2 1H
PCPD2 90.00 usec
PL12 17.70 dB
PL13 20.70 dB
PL2 0.00 dB
SFO2 300.1792007 MHz

F2 - Processing parameters
SI 16384
SF 75.4798237 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

¹³C NMR (75 MHz, CDCl₃) of Compound 5

Mono silylation of diol pure



Current Data Parameters
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EXPNO 1
PROCNO 1

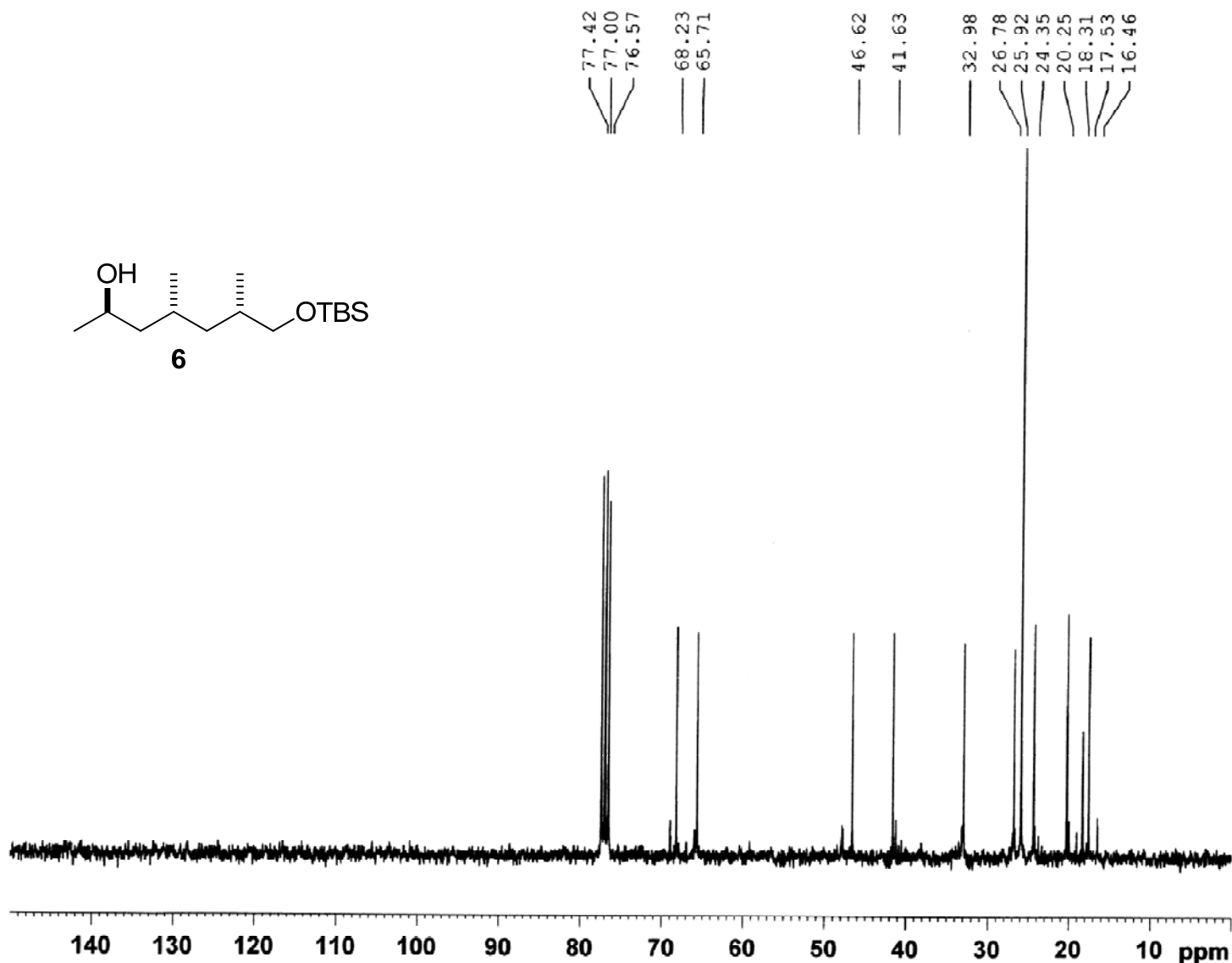
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Time 12.24
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PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 32
DS 0
SWH 4807.692 Hz
FIDRES 0.293438 Hz
AQ 1.7039860 sec
RG 25.4
DM 104.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
PT01 200.1901012 MHz

F2 - Processing parameters
SI 16384
SF 300.1700134 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

¹H NMR (300 MHz, CDCl₃) of Compound 6

Mono silylation of diol pure



Current Data Parameters
NAME iammeth 20-10-2012
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121020
Time 12.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 100
DS 0
SWH 17857.143 Hz
FIDRES 0.541957 Hz
AQ 0.9175540 sec
RG 203
DW 28.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.99999999 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.85 usec
PL1 0.00 dB
SFO1 75.4884982 MHz

===== CHANNEL f2 =====
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NUC2 1H
PCPD2 90.00 usec
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PL13 20.70 dB
PL2 0.00 dB
SFO2 300.1792007 MHz

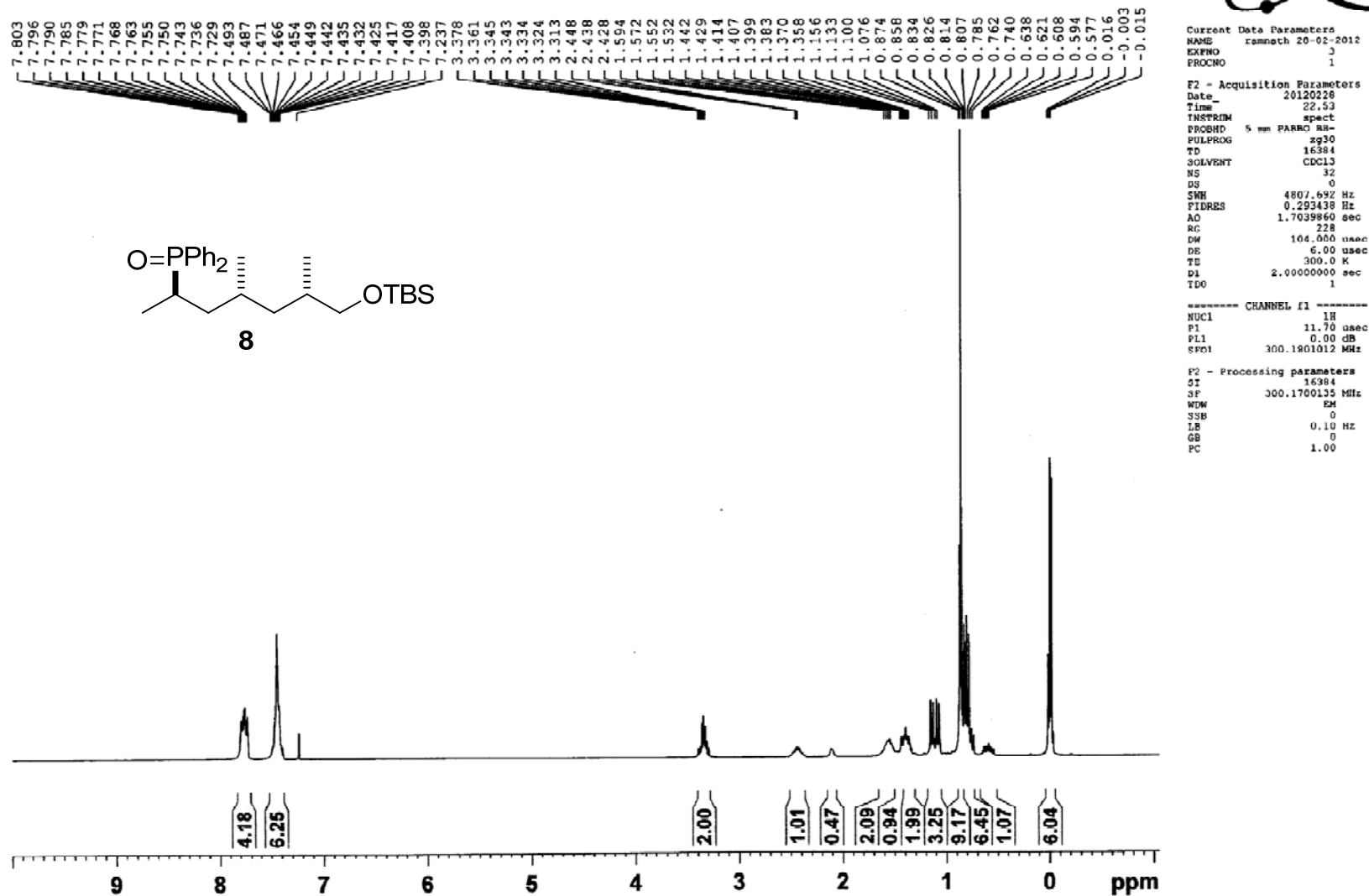
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WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00



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F2 - Processing parameters
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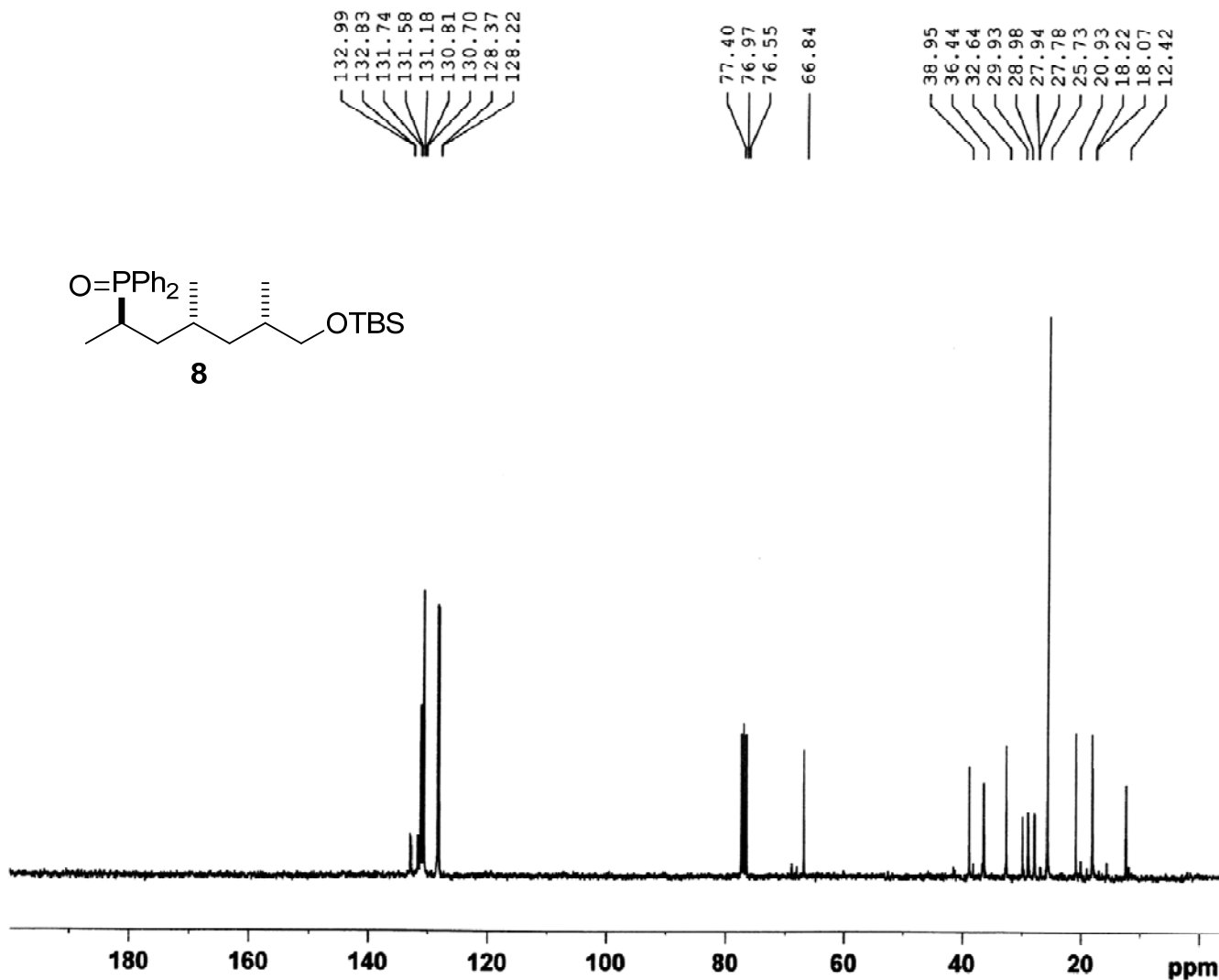


DPPO_OTBS purified



¹H NMR (300 MHz, CDCl₃) of Compound 8

Diphenylphosphonate OTBS pure



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PROCNO 1

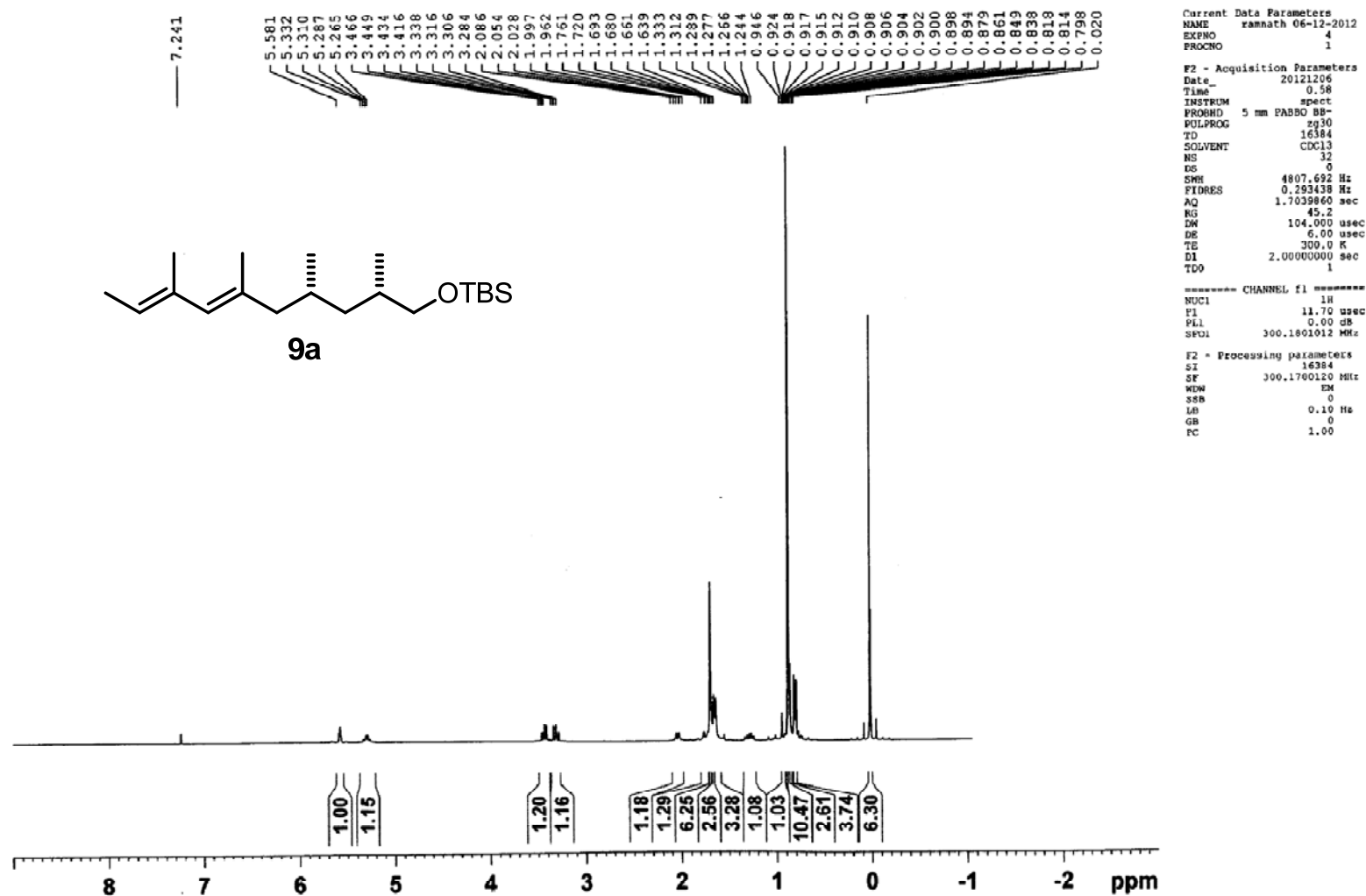
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PULPROG zgpg30
TD 32768
SOLVENT CDCl₃
NS 93
DS 0
SWH 17857.143 Hz
FIDRES 0.344957 Hz
AQ 0.9175540 sec
RG 1290
DW 28.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.99999999 sec
TD0 1

----- CHANNEL f1 -----
NUC1 13C
P1 9.85 usec
PL1 0.00 dB
SFO1 75.4884982 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL12 17.70 dB
PL13 20.70 dB
PL2 0.00 dB
SFO2 300.1742007 MHz

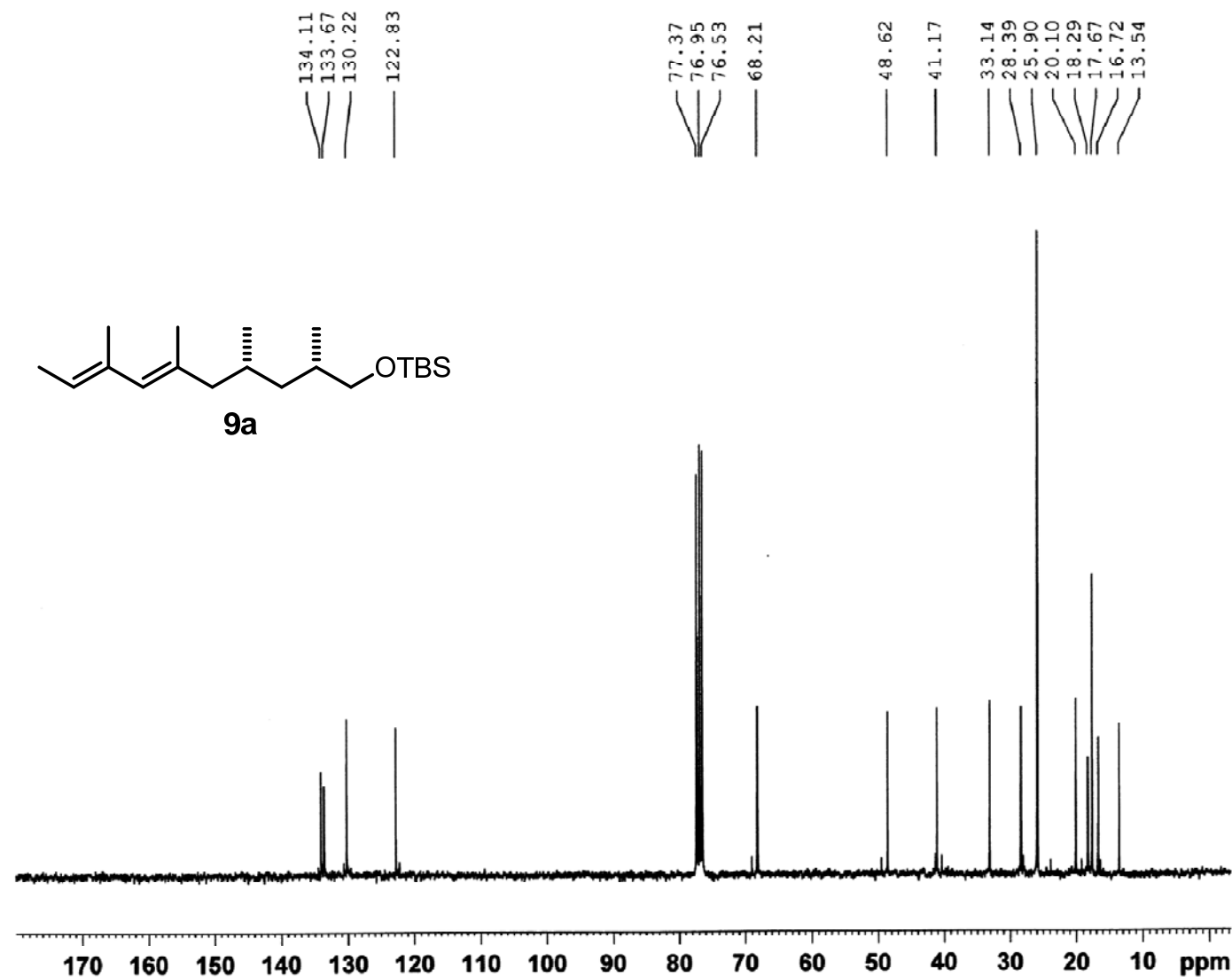
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SI 16384
SF 75.4798331 MHz
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LB 2.00 Hz
GB 0
PC 1.00

Diene OTBS pure



¹H NMR (300 MHz, CDCl₃) of Compound 9a

Diene OTBS pure



Current Data Parameters
NAME rannath 06-12-2012
EXPNO 5
PROCNO 1

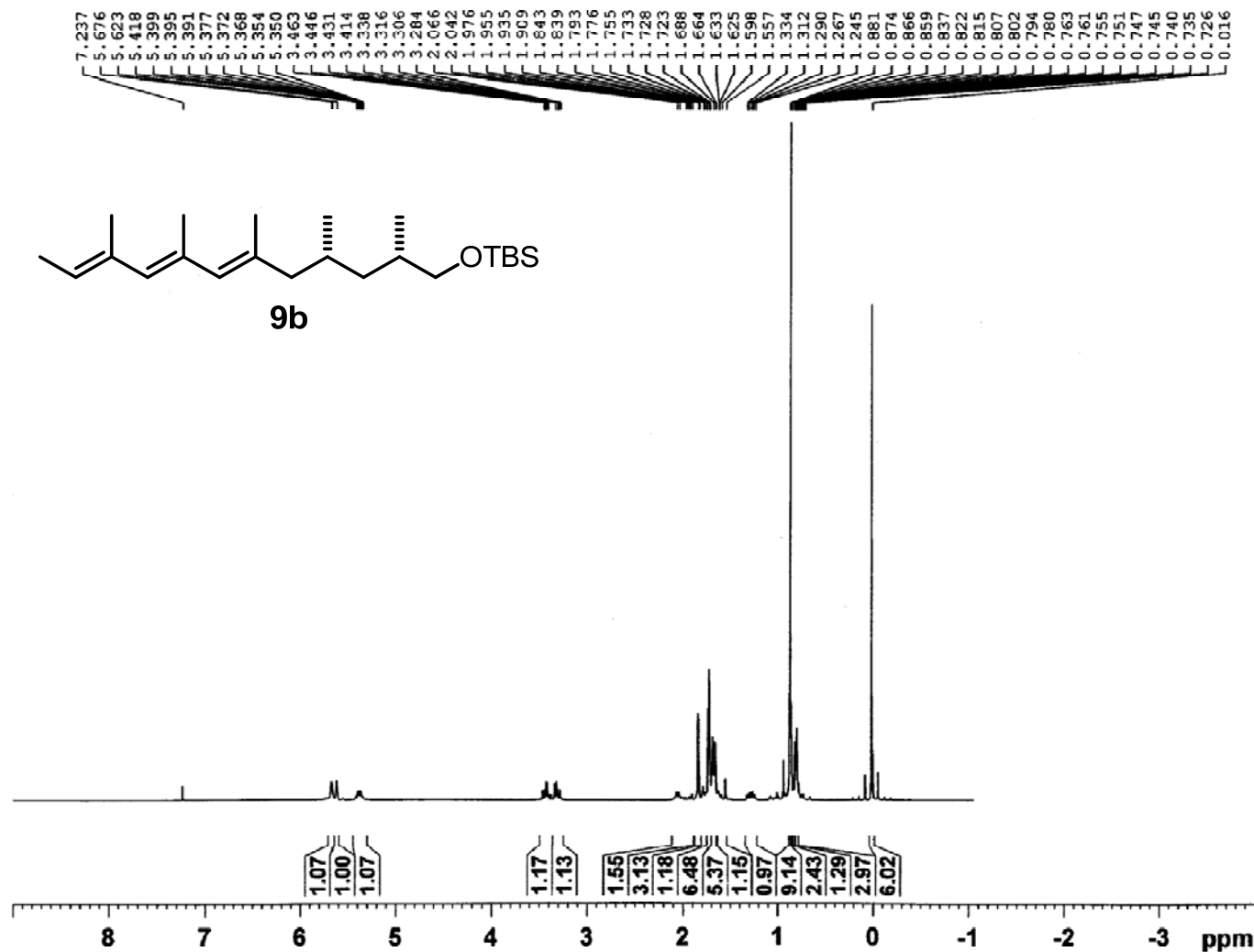
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Date_ 20121206
Time 1.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 546
DS 0
SWH 17857.143 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 912
DM 28.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999999 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.85 usec
PL1 0.00 dB
SFO1 75.4884082 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPDZ 90.00 usec
PL12 17.70 dB
PL13 20.70 dB
PL2 0.00 dB
SFO2 300.1792007 MHz

F2 - Processing parameters
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SF 75.4798221 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

Wittig-Horner elimination of triene Precursor, purified



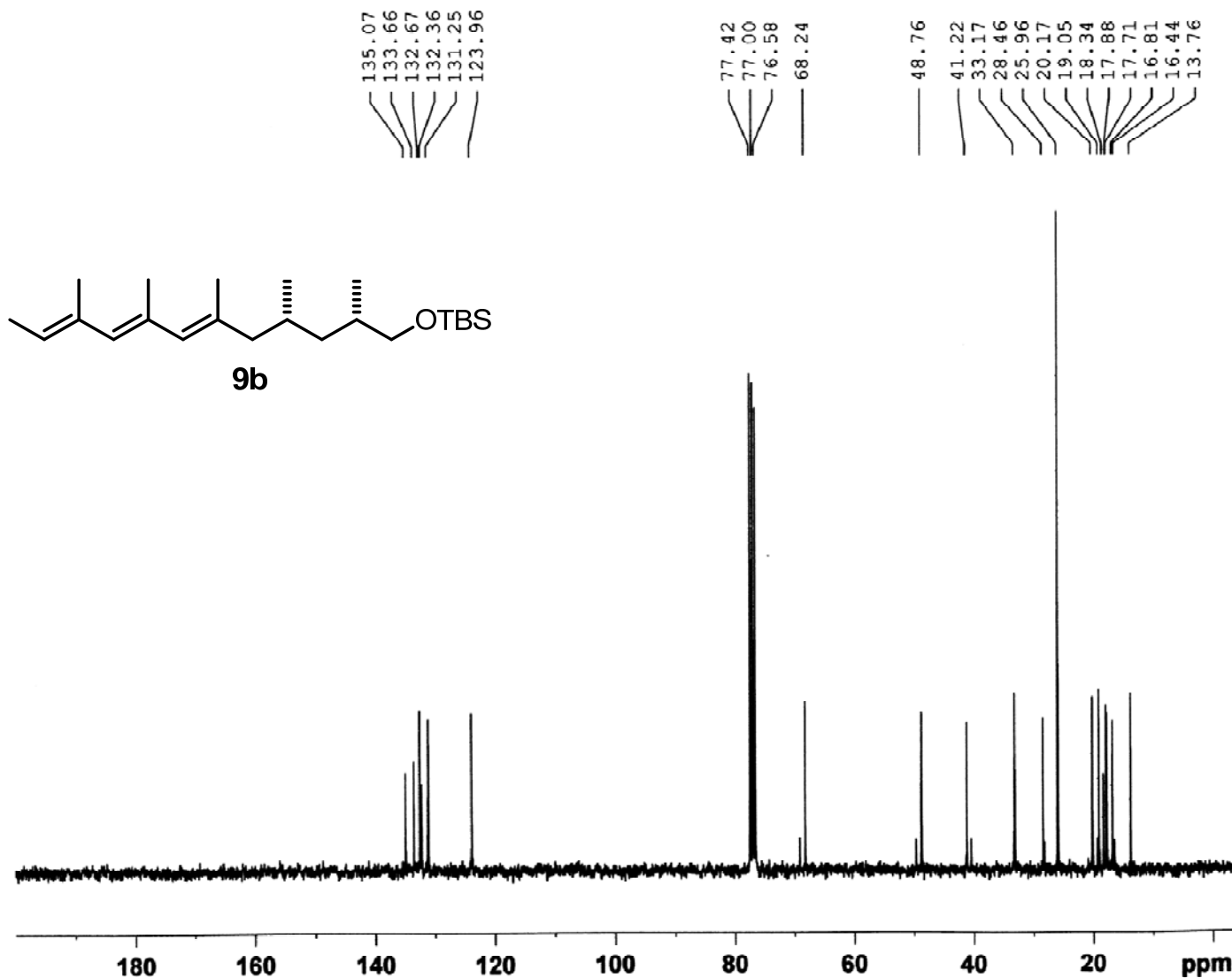
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NAME ramnath 02-11-2012
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
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PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 32
DS 0
SWH 4007.692 Hz
FIDRES 0.293439 Hz
AQ 1.7039860 sec
RG 32
DW 104.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SFO1 300.1001012 MHz

F2 - Processing parameters
SI 16384
SF 300.1780134 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Wittig-Horner elimination of triene Precursor, purified



Current Data Parameters
NAME ramnath 02-11-2012
EXPNO 8
PROCNO 1

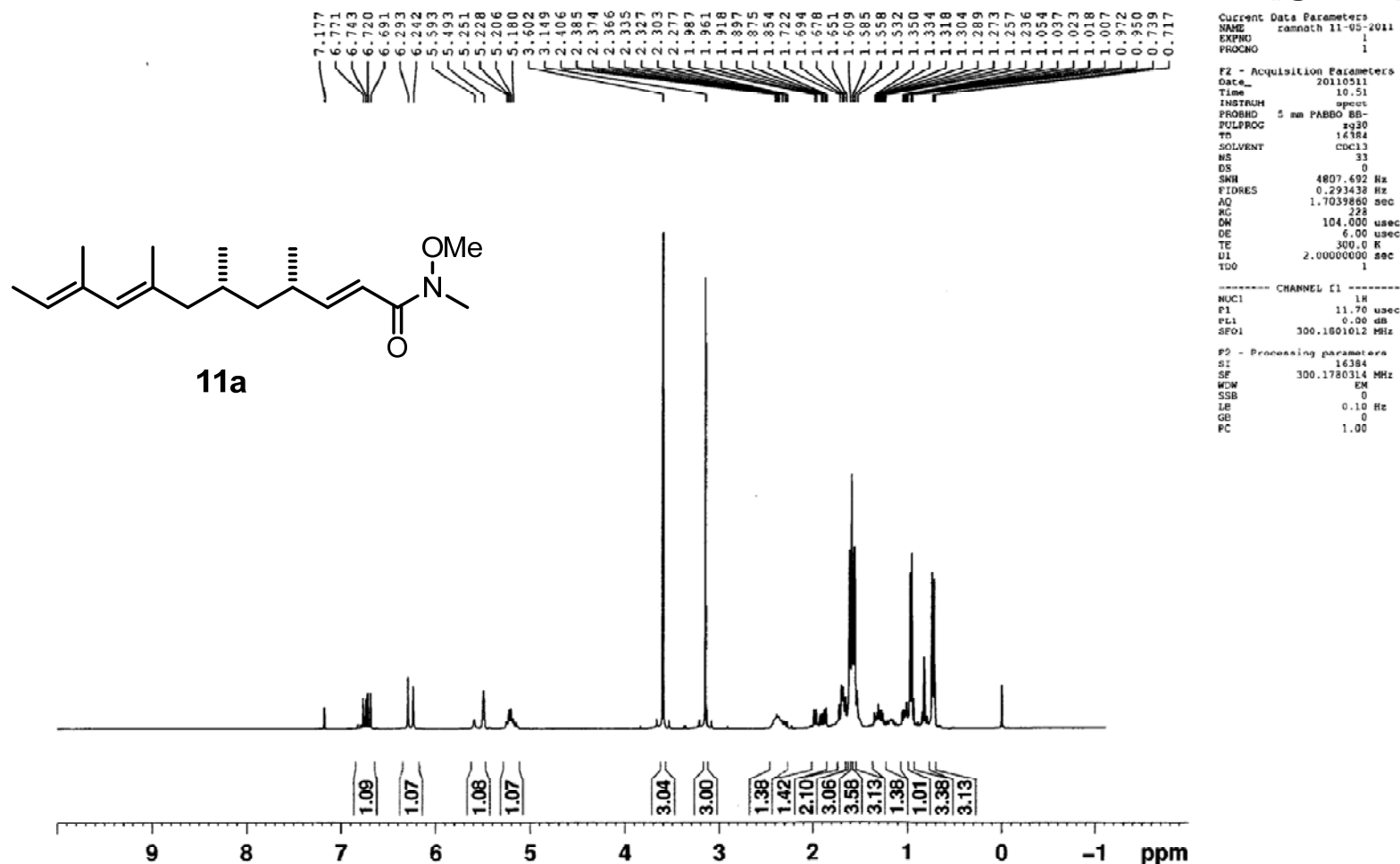
F2 - Acquisition Parameters
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Time 20.29
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TD 12768
SOLVENT CDCl3
NS 300
DS 0
SWH 17857.143 Hz
FIDRES 0.344957 Hz
AQ 0.9175540 sec
RG 1820
DM 28.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

----- CHANNEL f1 -----
NUC1 13C
P1 9.85 usec
PL1 0.00 dB
SFO1 75.4884982 MHz

----- CHANNEL f2 -----
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NUC2 1H
PCPDZ 90.00 usec
PL12 17.70 dB
PL13 20.70 dB
PL2 0.00 dB
SFO2 300.1792007 MHz

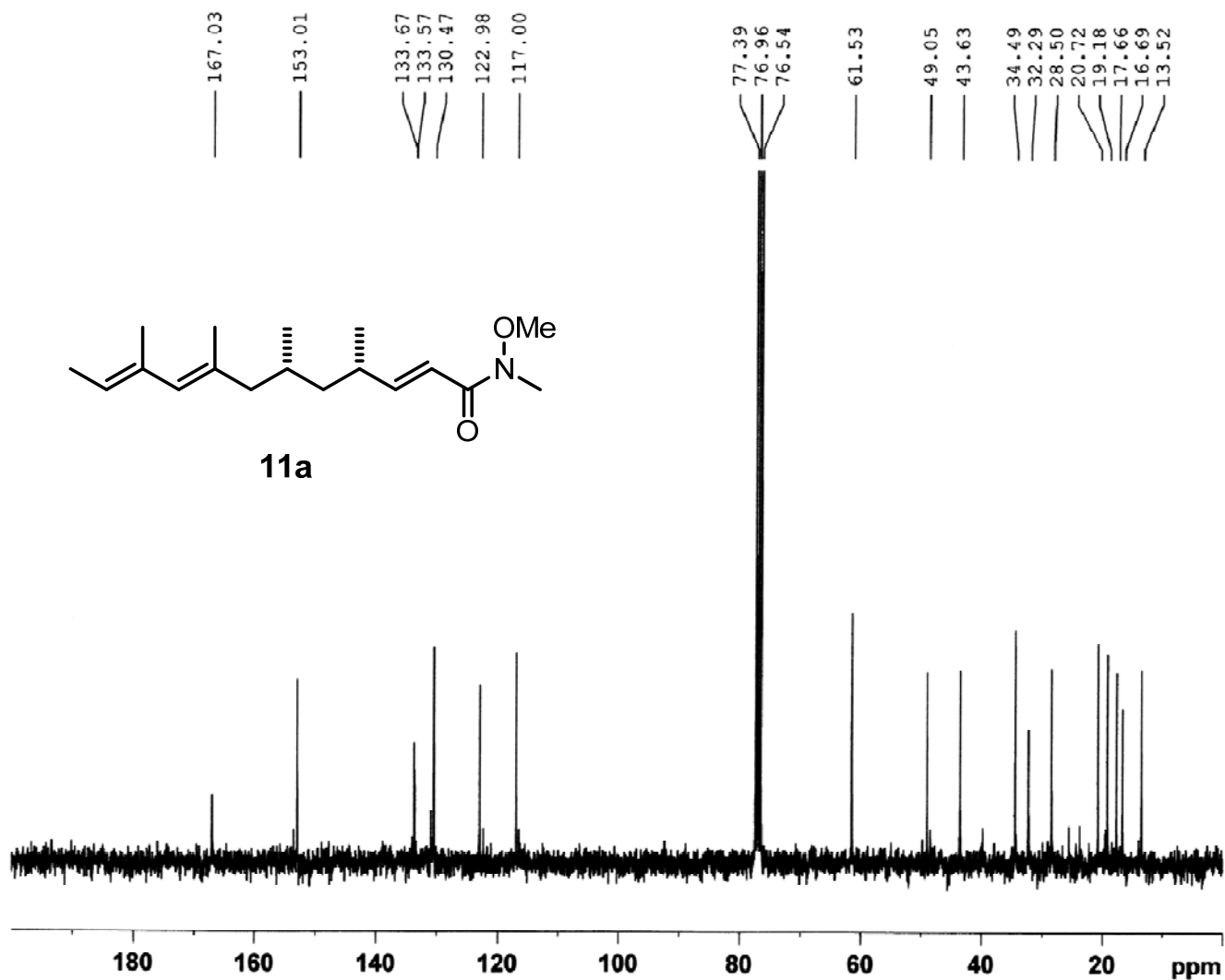
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LB 2.00 Hz
GB 0
PC 1.00

diene-weinreb Model precursor purified



^1H NMR (300 MHz, CDCl_3) of Compound **11a**

diene-weinreb Model precursor purified



Current Data Parameters
NAME ramnath 11-05-2011
EXPERO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110511
Time_ 11.01
INSTRUM spect
PROBHD 5 mm PARRC
PULPROG zgpg30
TD 32768
SOLVENT CDCl_3
NS 156
DS 0
SWH 17657.143 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 724
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DE 6.00 usec
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d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

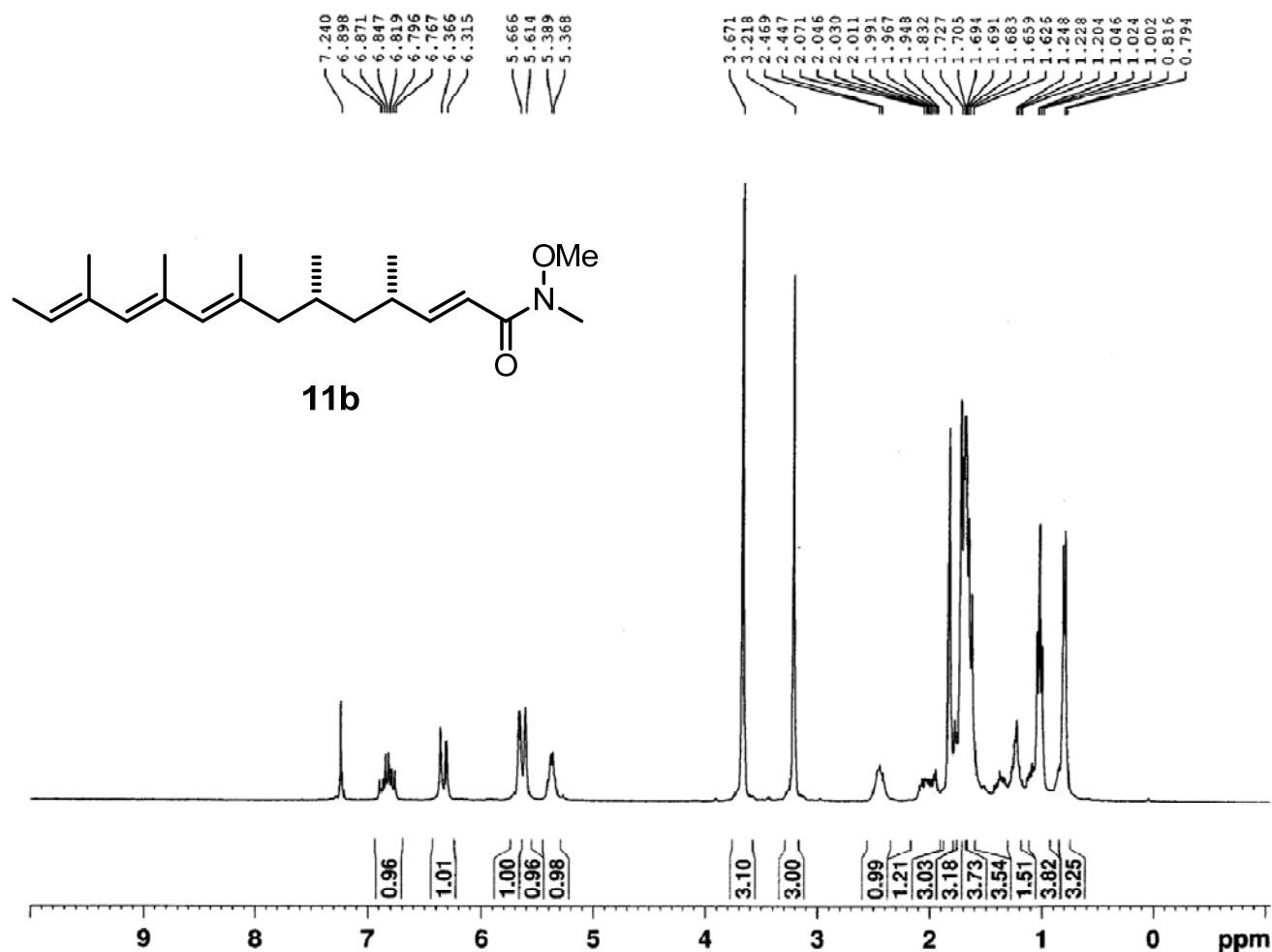
----- CHANNEL f1 -----
NUC1 ^{13}C
P1 0.05 usec
PL1 0.00 dB
SFO1 75.4884582 MHz

----- CHANNEL f2 -----
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NUC2 ^1H
PCPD2 90.00 usec
PL12 17.70 dB
PL13 20.70 dB
PL2 0.00 dB
SFO2 300.1360007 MHz

F2 - Processing parameters
SI 16384
SF 75.4798229 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

^{13}C NMR (75 MHz, CDCl_3) of Compound **11a**

Triene weinreb Purified



Current Data Parameters
NAME ramnath 25-07-2011
EXPNO 4
PROCNO 1

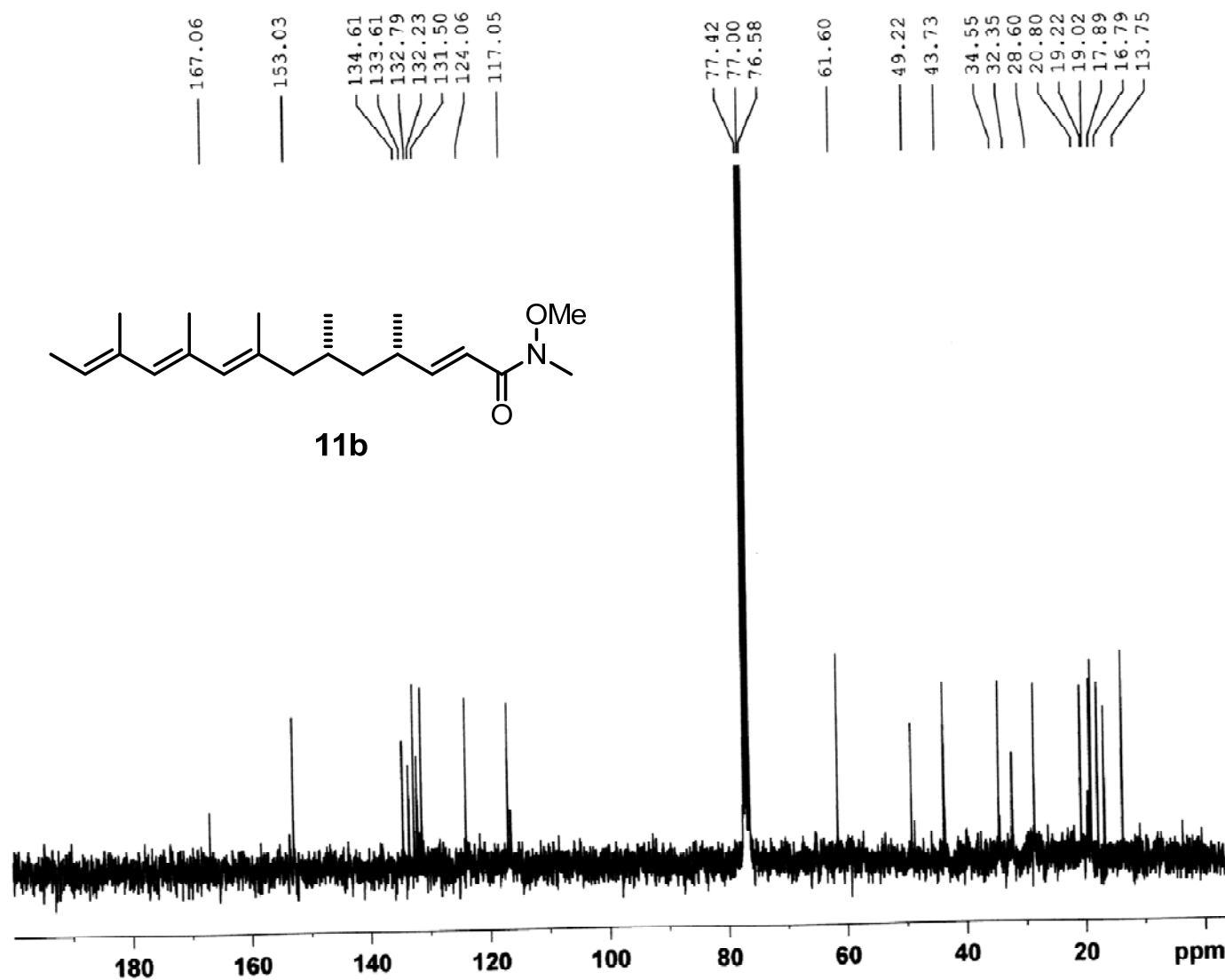
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PULPROG zg30
TD 16384
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NS 12
DS 0
SWH 4807.692 Hz
FIDRES 0.293428 Hz
AQ 1.7039860 sec
RG 362
DN 104.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SFO1 300.1801012 MHz

F2 - Processing parameters
SI 16384
SF 300.1720122 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

¹H NMR (300 MHz, CDCl₃) of Compound **11b**

Exo IMDA precursor purified



Current Data Parameters
NAME ramnath 22-11-2011
EXPRO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111122
Time 18.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT cdcl3
NS 401
DS 0
SWH 17857.143 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 912
RG 28.000 usec
RW 6.00 usec
DE 300.0 K
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

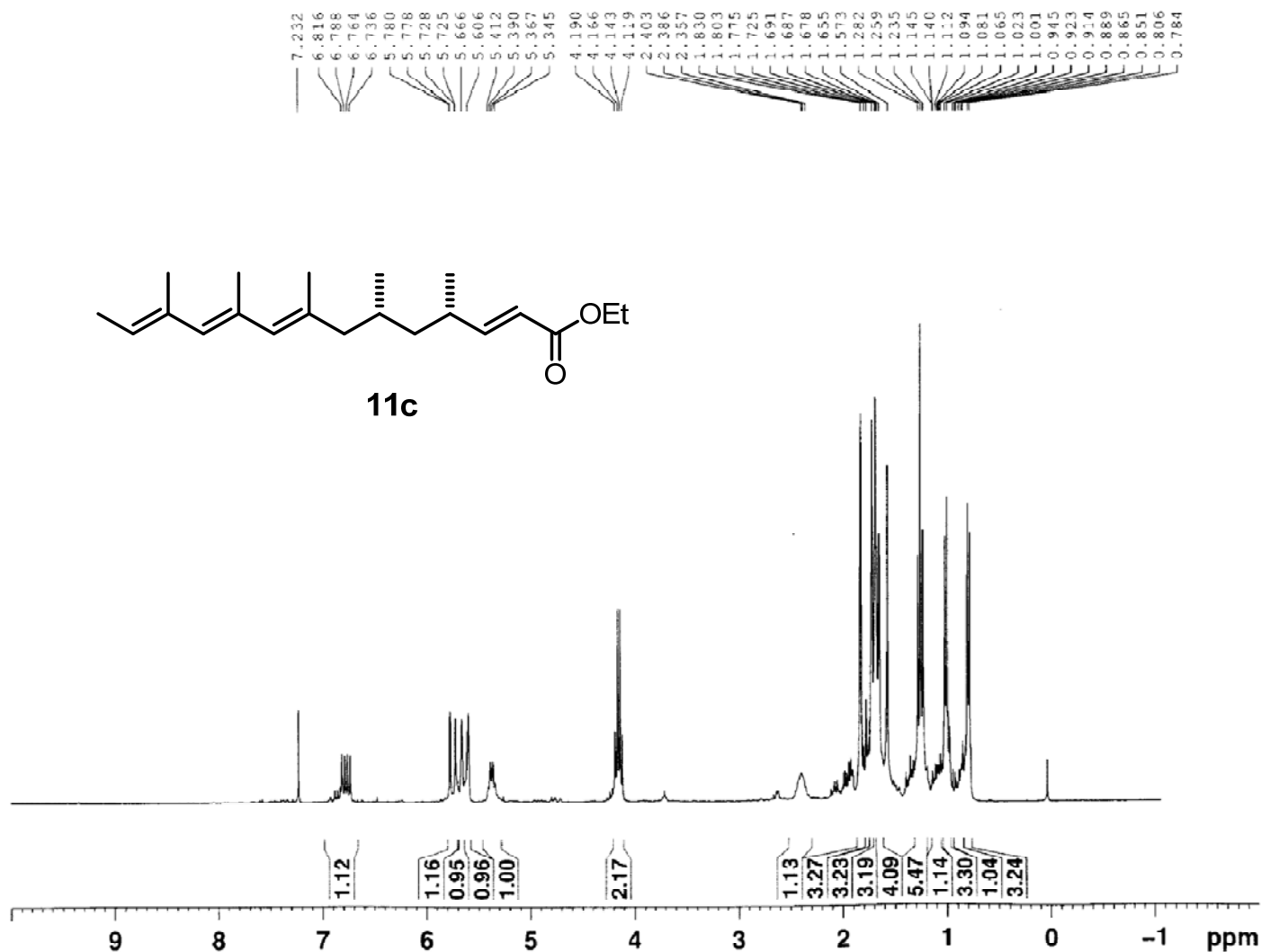
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NUC1 13C
P1 9.85 usec
PL1 0.00 dB
SFO1 75.4884982 MHz

===== CHANNEL f2 =====
CFDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL12 17.70 dB
PL13 20.70 dB
PL2 0.00 dB
SFO2 300.1792007 MHz

F2 - Processing parameters
SI 16384
SF 75.4768195 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

¹³C NMR (75 MHz, CDCl₃) of Compound **11b**

ester pure



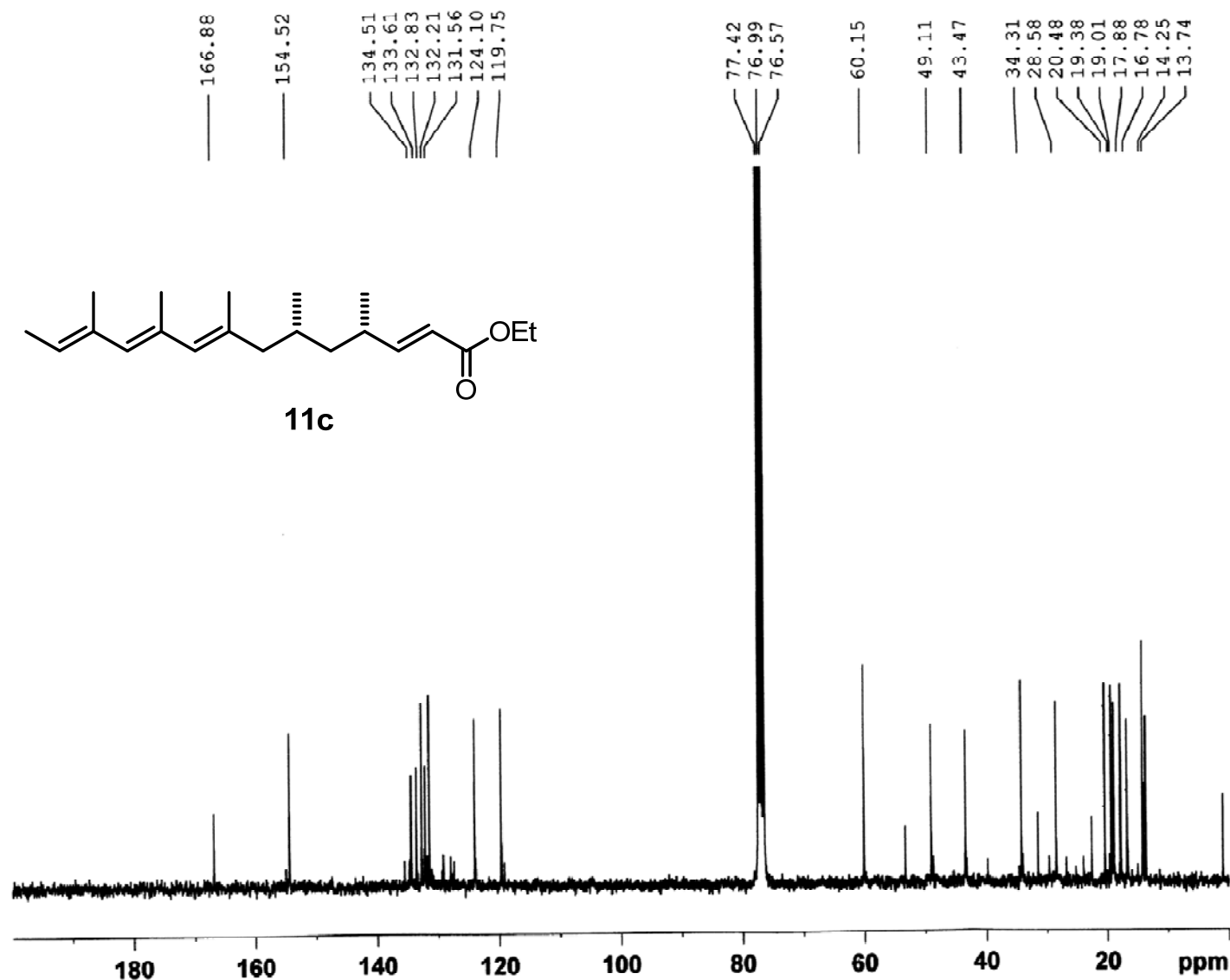
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NAME ramath 06-01-2011
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110136
Time 14.30
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PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 15
DS 0
SWH 4807.632 Hz
FIDRES 0.293438 Hz
AQ 1.7039860 sec
RG 405
DW 104.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SFO1 300.1801012 MHz

F2 - Processing parameters
SI 16384
SF 300.1780150 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Ester pure



Current Data Parameters
NAME ramoth 12-06-2010
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100612
Time 23.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 11412
DS 0
SWH 17857.143 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 912
DW 28.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

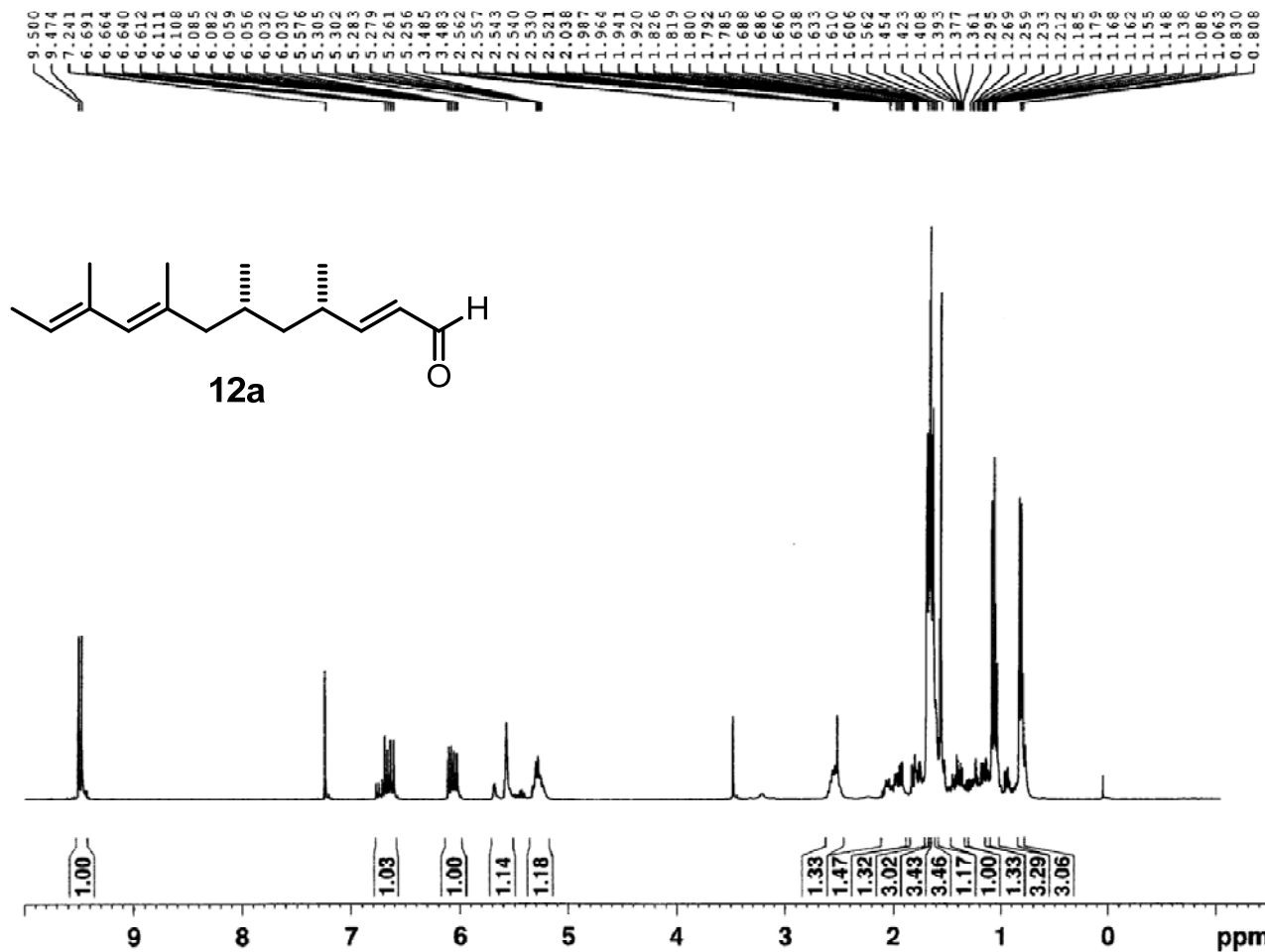
----- CHANNEL f1 -----
NUC1 13C
P1 9.85 usec
PL1 0.00 dB
SFO1 75.4884982 MHz

----- CHANNEL f2 -----
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NUC2 1H
PCPD2 90.00 usec
PL12 17.70 dB
PL13 20.70 dB
PL2 0.00 dB
SFO2 300.1792007 MHz

F2 - Processing parameters
SI 16384
SF 75.4798196 MHz
WDW EM
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GB 0
PC 1.00

¹³C NMR (75 MHz, CDCl₃) of Compound **11c**

diene-aldehyde purified



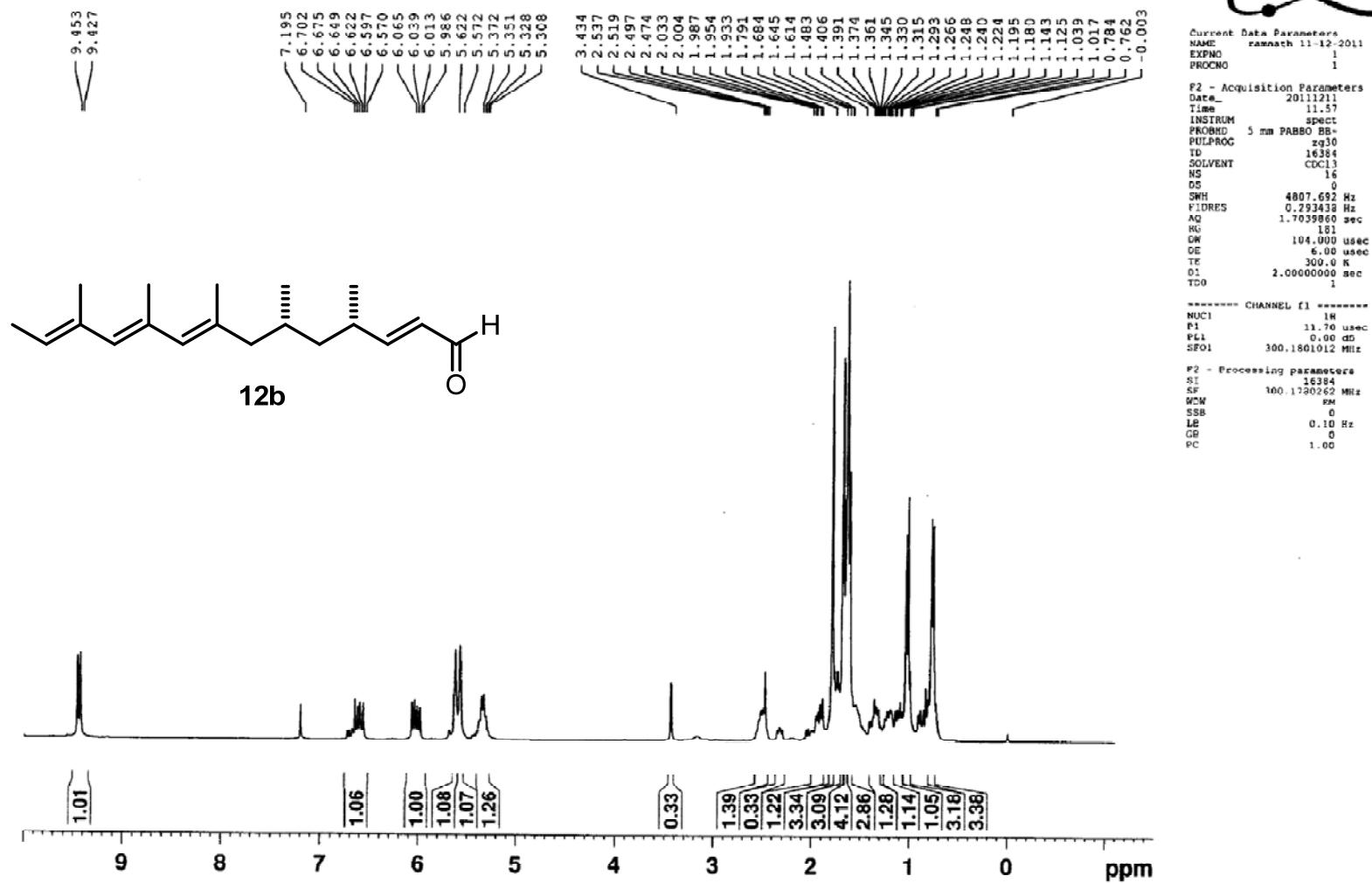
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NAME rsmach 22-06-2011
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
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Time 19.14
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PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 32
DS 0
SWH 4807.692 Hz
FIDRES 0.293438 Hz
AQ 1.7039860 sec
RG 512
QM 104.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.6006000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SFO1 300.1601012 MHz

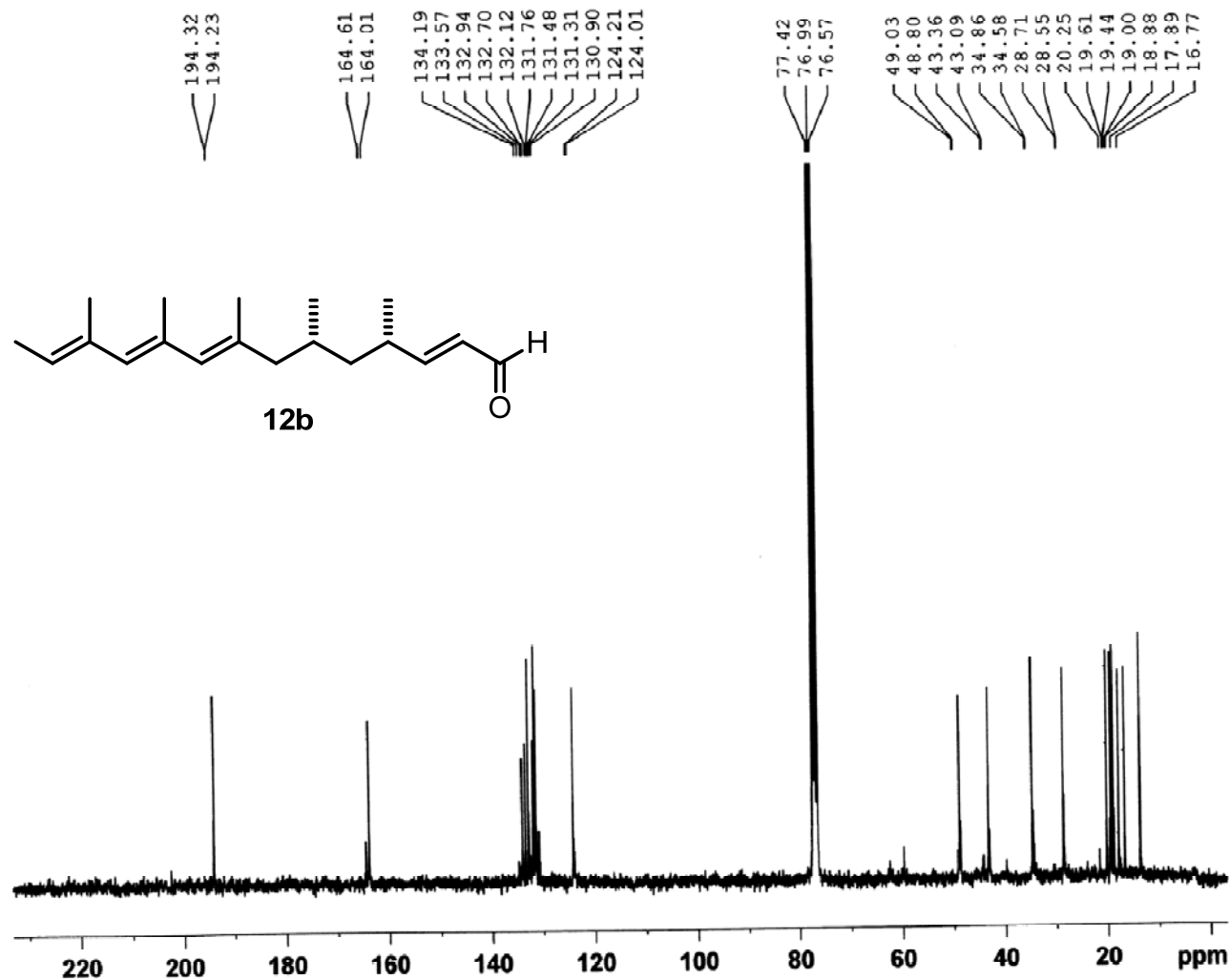
F2 - Processing parameters
SI 16384
SF 300.1780127 MHz
WOM 5M
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Purified aldehyde precursor for IMDA



¹H NMR (300 MHz, CDCl₃) of Compound 12b

Tetraenal pure



Current Data Parameters
NAME ramnath 28-11-2012
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121128
Time 0.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 10000
DS 0
SWH 17857.143 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 1030
DW 28.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

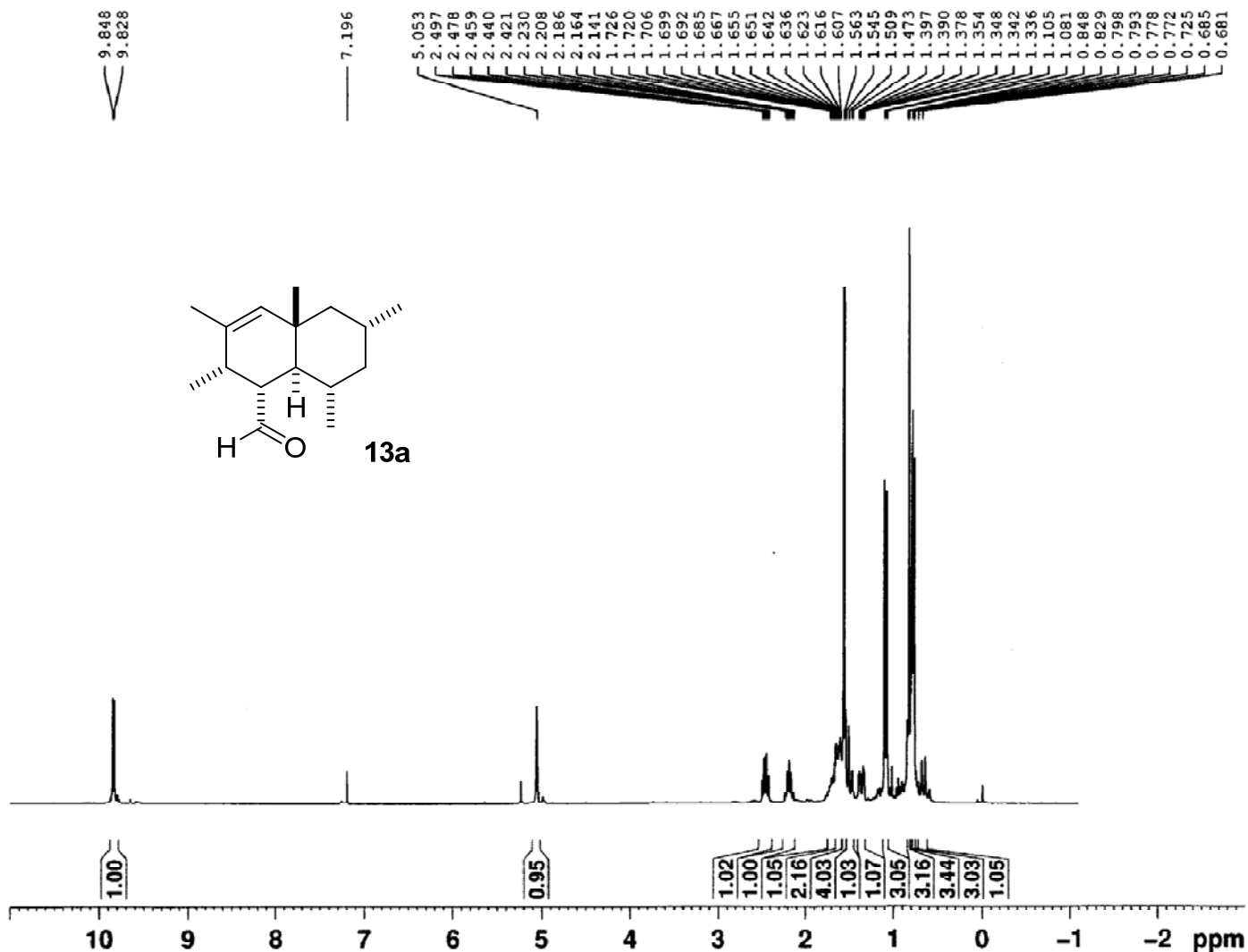
===== CHANNEL f1 =====
NUC1 13C
P1 9.85 usec
PL1 0.00 dB
SFO1 75.4884982 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPDZ 90.00 usec
PL12 17.70 dB
PL13 20.70 dB
PL2 0.00 dB
SFO2 300.1792007 MHz

F2 - Processing parameters
SI 16384
SF 75.4798191 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

¹³C NMR (75 MHz, CDCl₃) of Compound **12b**

IMDA endo pure



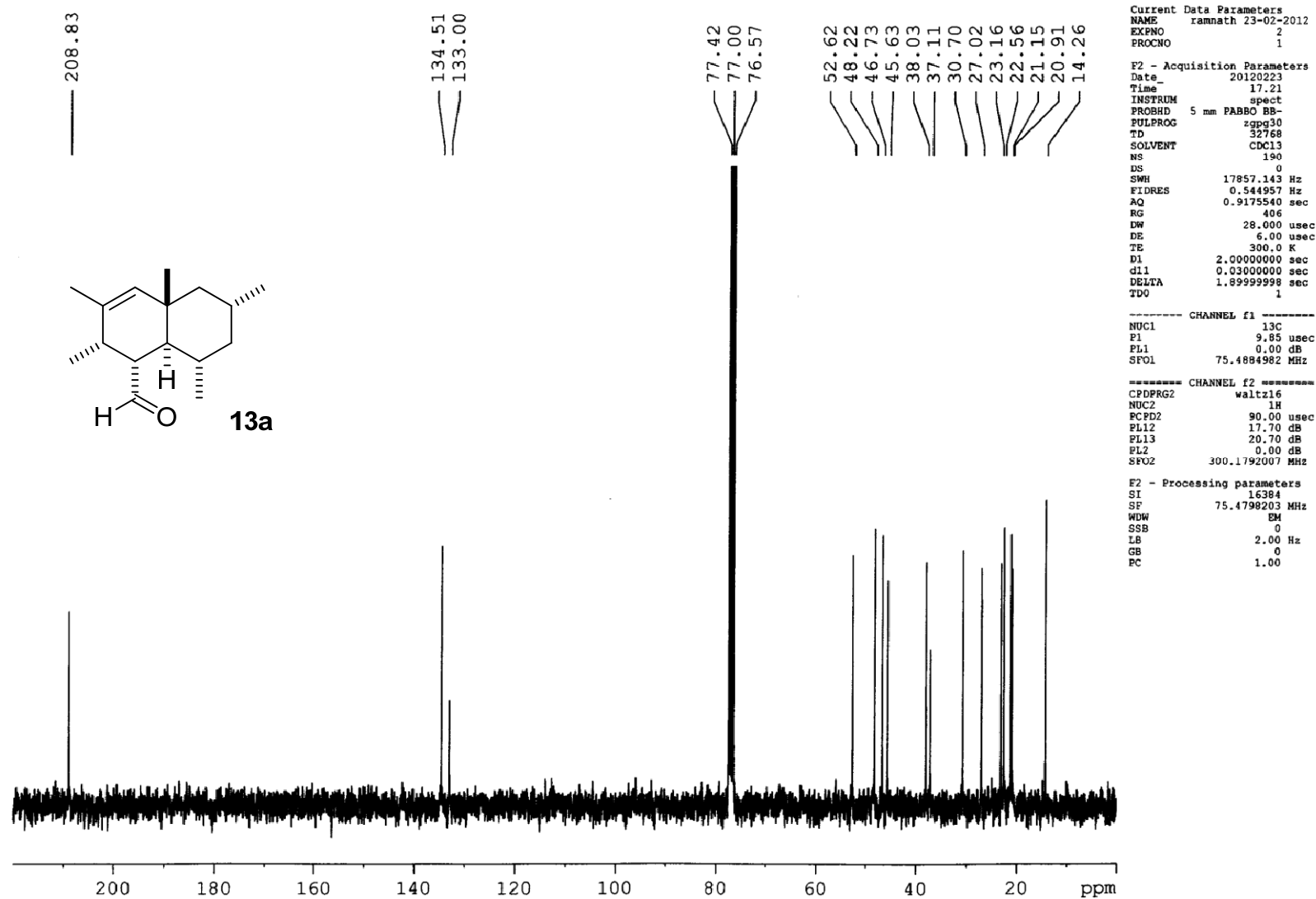
Current Data Parameters
NAME ramnath 23-02-2012
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120223
Time 17.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 21
DS 0
SWH 4807.692 Hz
FIDRES 0.291438 Hz
AQ 1.7039860 sec
RG 287
DW 104.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

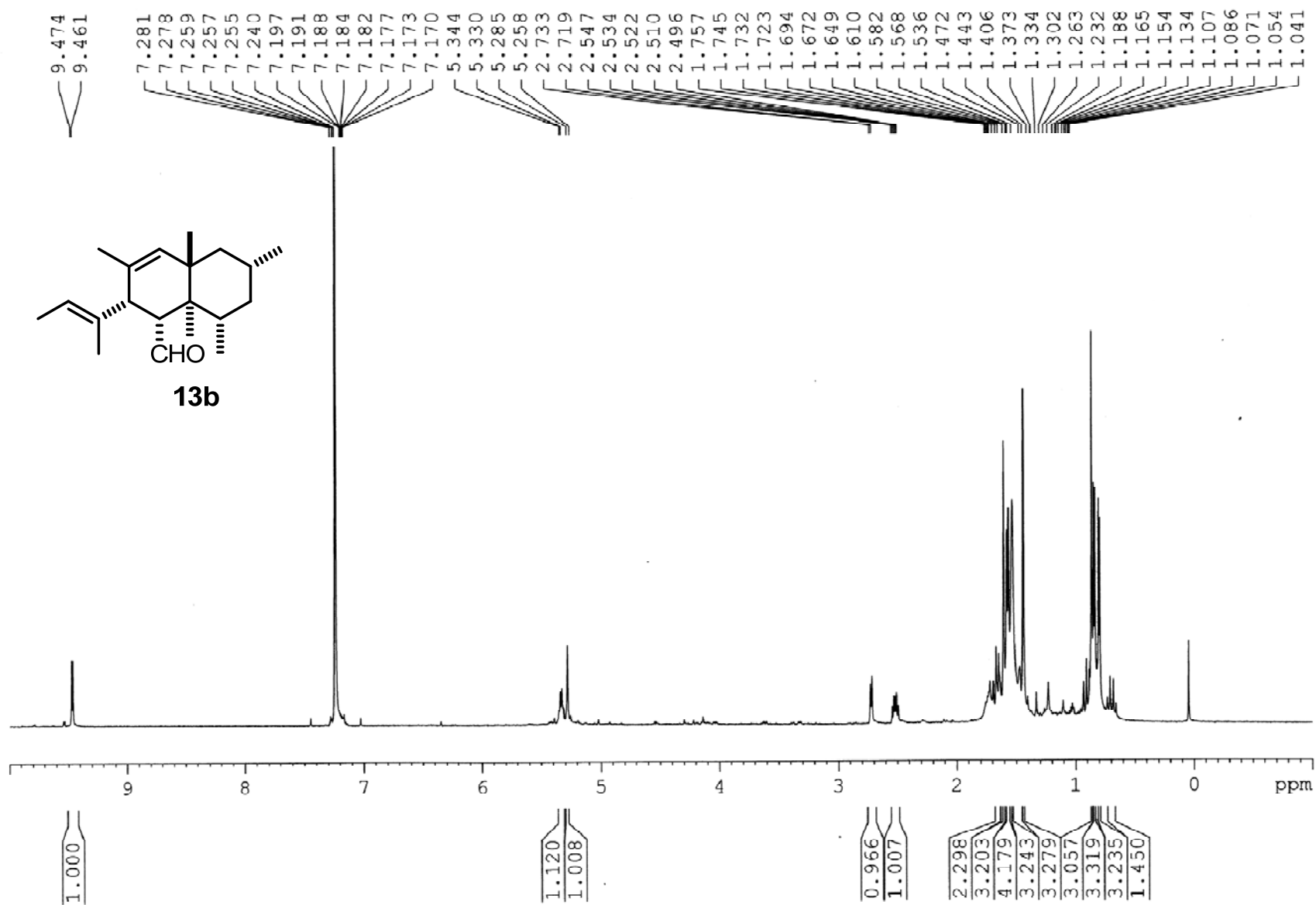
***** CHANNEL f1 *****
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SFO1 300.1601012 MHz

F2 - Processing parameters
SI 16384
SF 300.1780259 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

IMDA endo pure

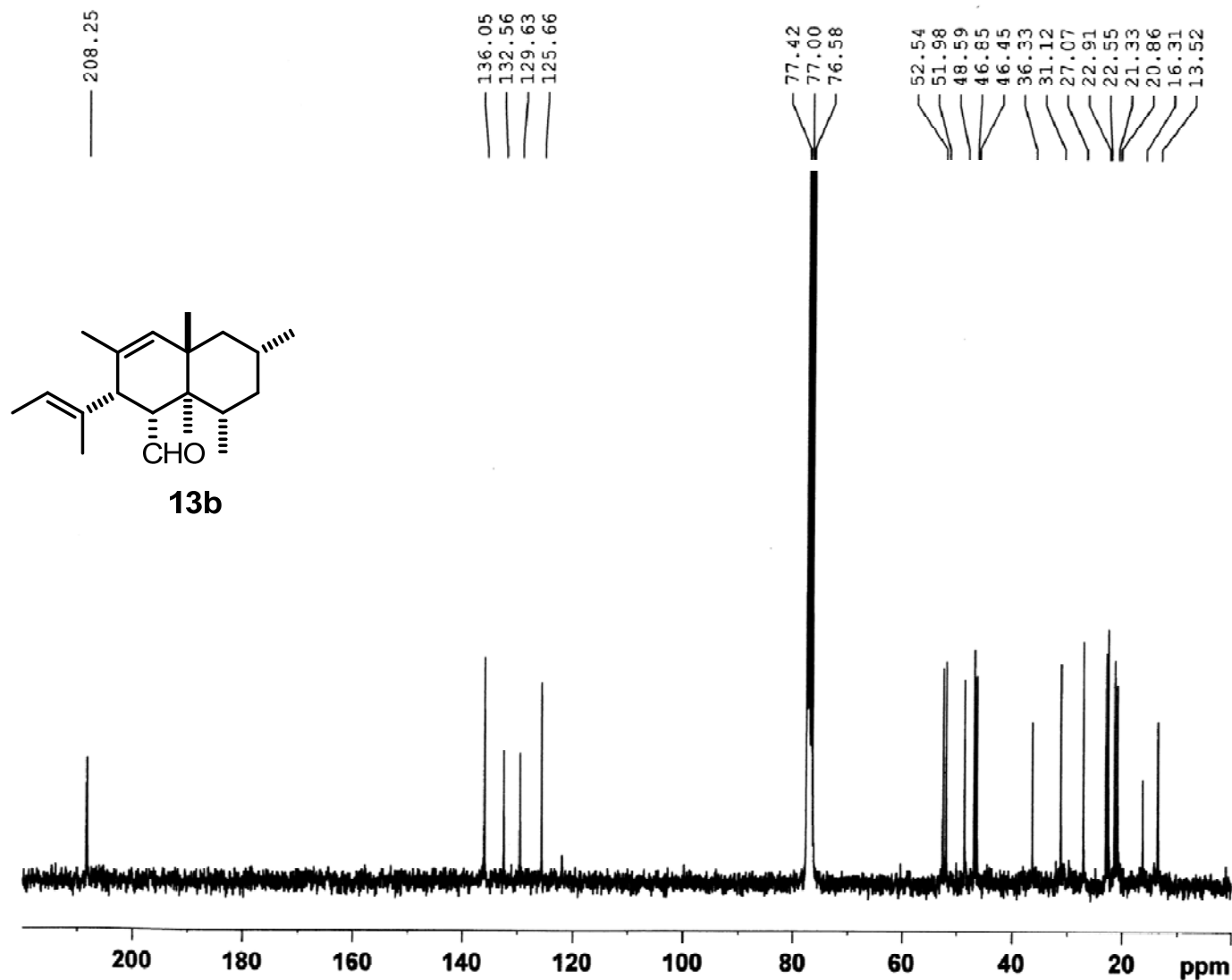


¹³C NMR (75 MHz, CDCl₃) of Compound 13a



¹H NMR (500 MHz, CDCl₃) of Compound **13b**

IMDA pure



Current Data Parameters
NAME ramnath 12-11-2012
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121112
Time 0.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl_3
NS 10000
DS 0
SWH 17857.143 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 1030
DM 28.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.80000000 sec
TD0 1

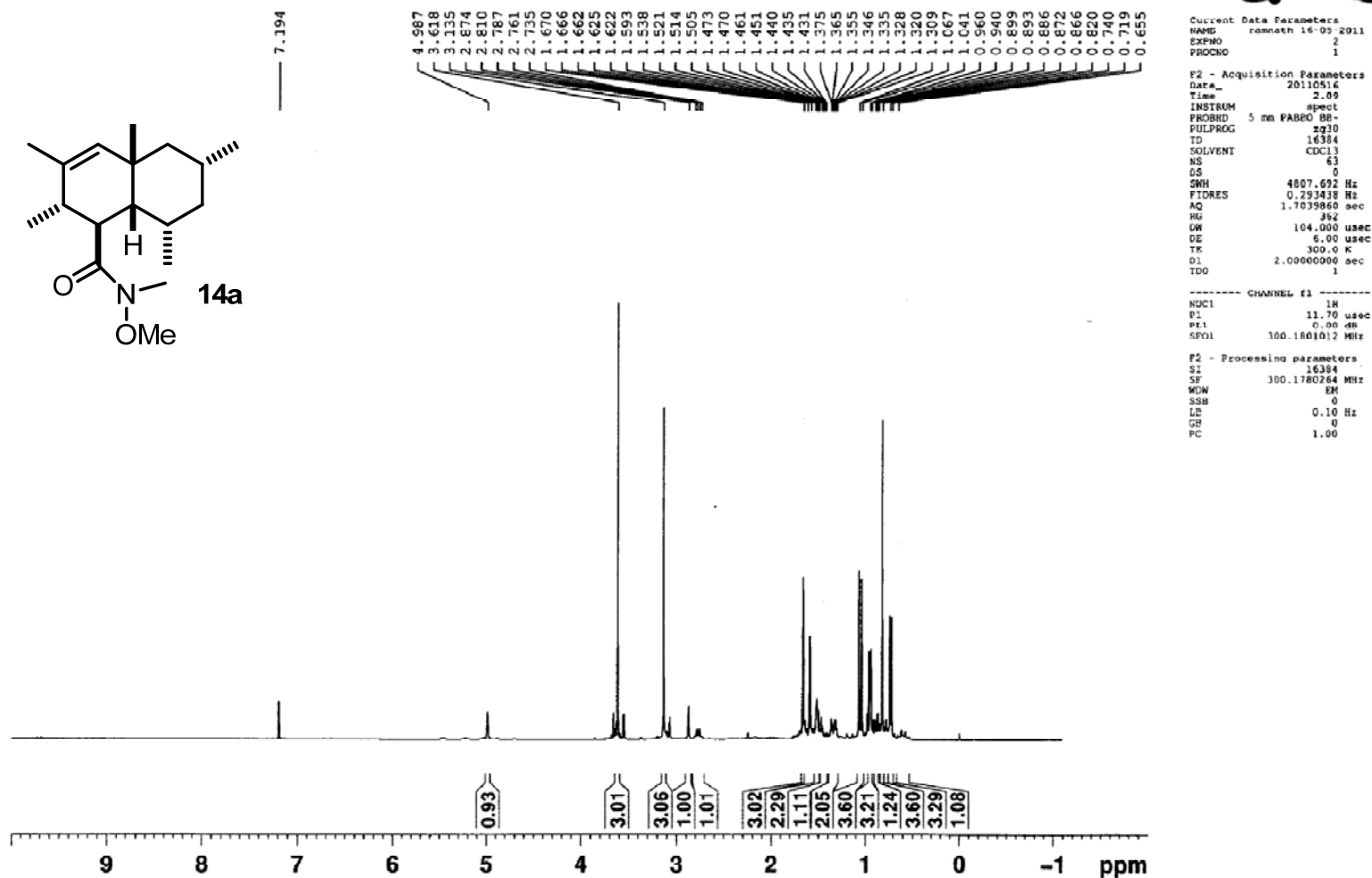
===== CHANNEL f1 =====
NUC1 ^{13}C
P1 9.85 usec
PL1 0.00 dB
SFO1 75.4804982 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 90.00 usec
PL12 17.70 dB
PL13 20.70 dB
PL2 0.00 dB
SFO2 300.172007 MHz

F2 - Processing parameters
SI 16384
SF 75.4798189 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

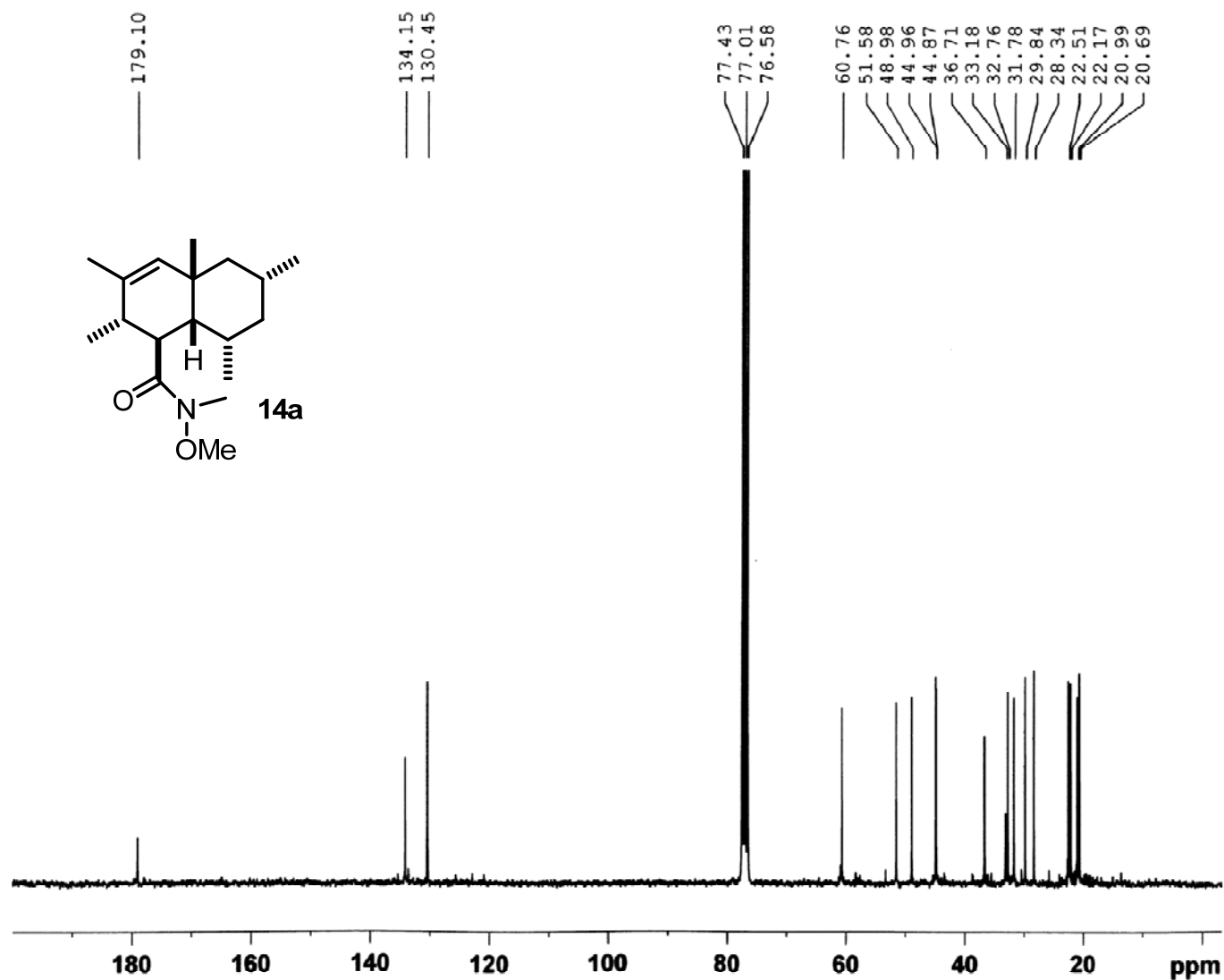
^{13}C NMR (75 MHz, CDCl_3) of Compound **13b**

Model IMDA purified



¹H NMR (300 MHz, CDCl₃) of Compound **14a**

Model IMDA purified



Current Data Parameters
NAME ramnath 16-05-2011
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110516
Time 2.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl₃
NS 8183
DS 0
SWH 17857.143 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 575
DM 28.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999999 sec
TEO 1

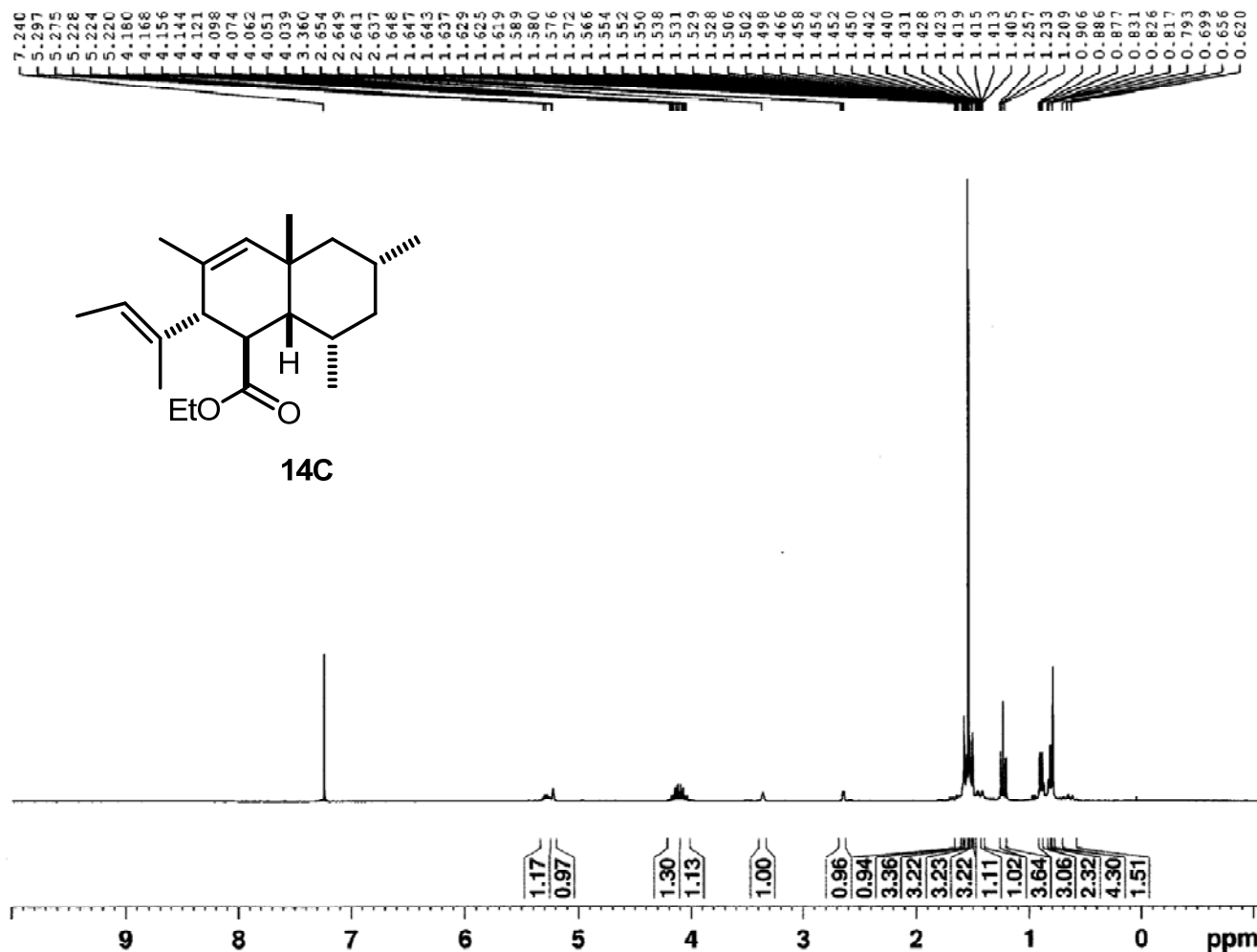
===== CHANNEL f1 =====
NUC1 13C
P1 9.85 usec
PL1 0.00 dB
SFO1 75.4884982 MHz

===== CHANNEL f2 =====
CEDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 17.70 dB
PL13 20.70 dB
PL2 0.00 dB
SFO2 300.1792007 MHz

F2 - Processing parameters
SI 16384
SF 75.4798191 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

¹³C NMR (75 MHz, CDCl₃) of Compound 14a

IMDA pure



Current Data Parameters
NAME ksmnath 11-04-2011
EXPNO 1
PROCNO 1

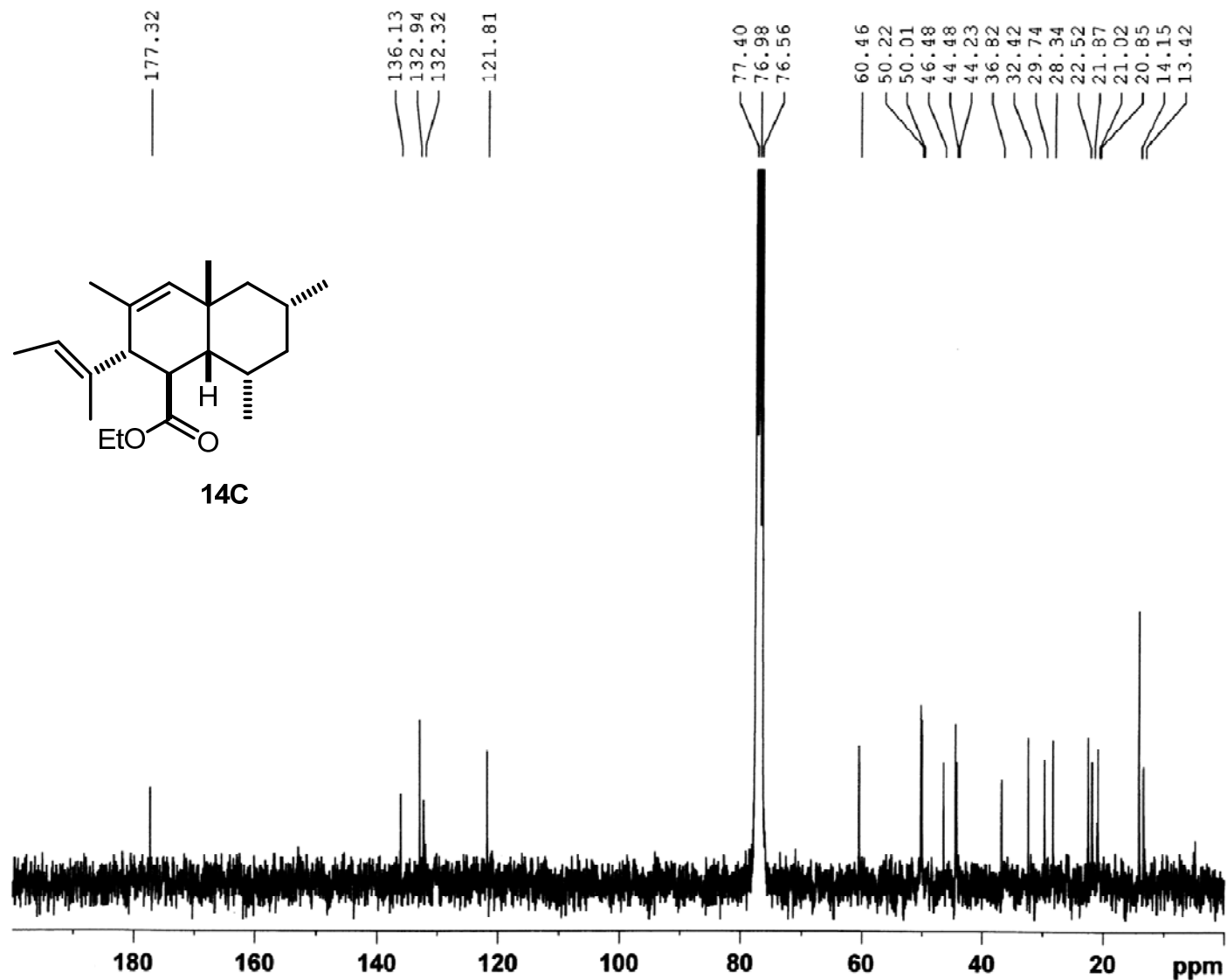
F2 - Acquisition Parameters
Date_ 20110411
Time 9.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 198
DS 0
SWH 4807.692 Hz
FIDRES 0.291438 Hz
AQ 1.7039860 sec
RG 812
DM 104.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SFO1 300.1601012 MHz

F2 - Processing parameters
SI 16384
SF 300.1780126 MHz
WDM EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

¹H NMR (300 MHz, CDCl₃) of Compound **14c**

IMDA pure



Current Data Parameters
NAME ramnath 09-04-2011
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110409
Time 0.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 12498
DS 0
SWH 17857.143 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 512
DW 28.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

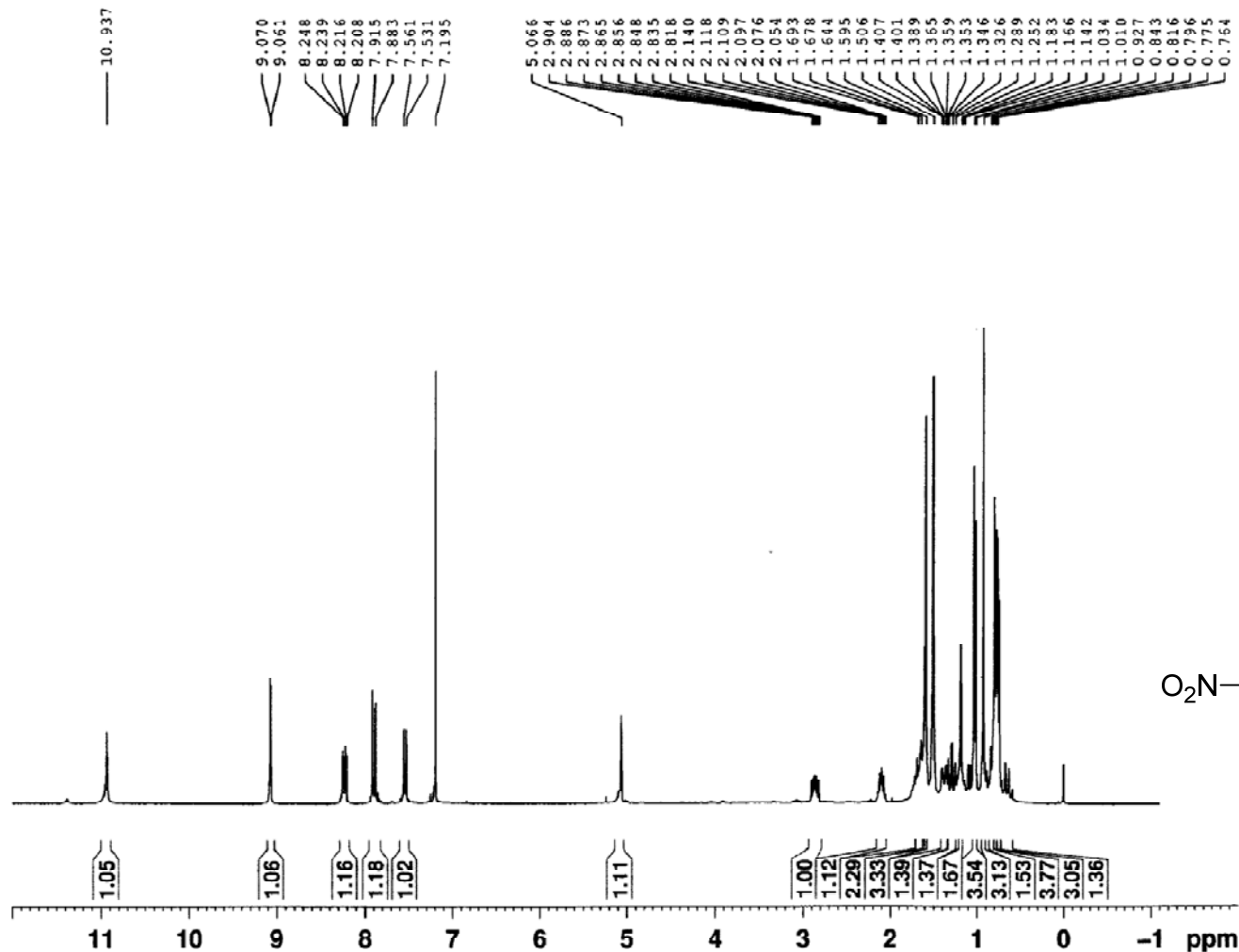
===== CHANNEL f1 =====
NUC1 13C
P1 9.85 usec
PL1 0.00 dB
SFO1 75.4884982 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL12 17.70 dB
PL13 20.70 dB
PL2 0.00 dB
SFO2 300.1792007 MHz

F2 - Processing parameters
SI 16384
SF 75.4799202 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

¹³C NMR (75 MHz, CDCl₃) of Compound **14c**

Endo, IMDA, 2,4DNP, pure

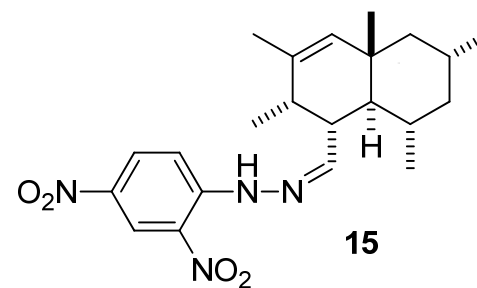


Current Data Parameters
NAME: ramnath 23-02-2012
EXPNO: 5
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20120228
Time: 23.24
INSTRUM: spect
PROBHD: 5 mm PABBO BB-
PULPROG: zg30
TD: 16384
SOLVENT: CDCl3
NS: 108
DS: 0
SWH: 4807.692 Hz
FIDRES: 0.293438 Hz
AQ: 1.7039860 sec
RG: 312
DW: 104.000 usec
DE: 6.00 usec
TE: 300.0 K
D1: 2.00000000 sec
TD0: 1

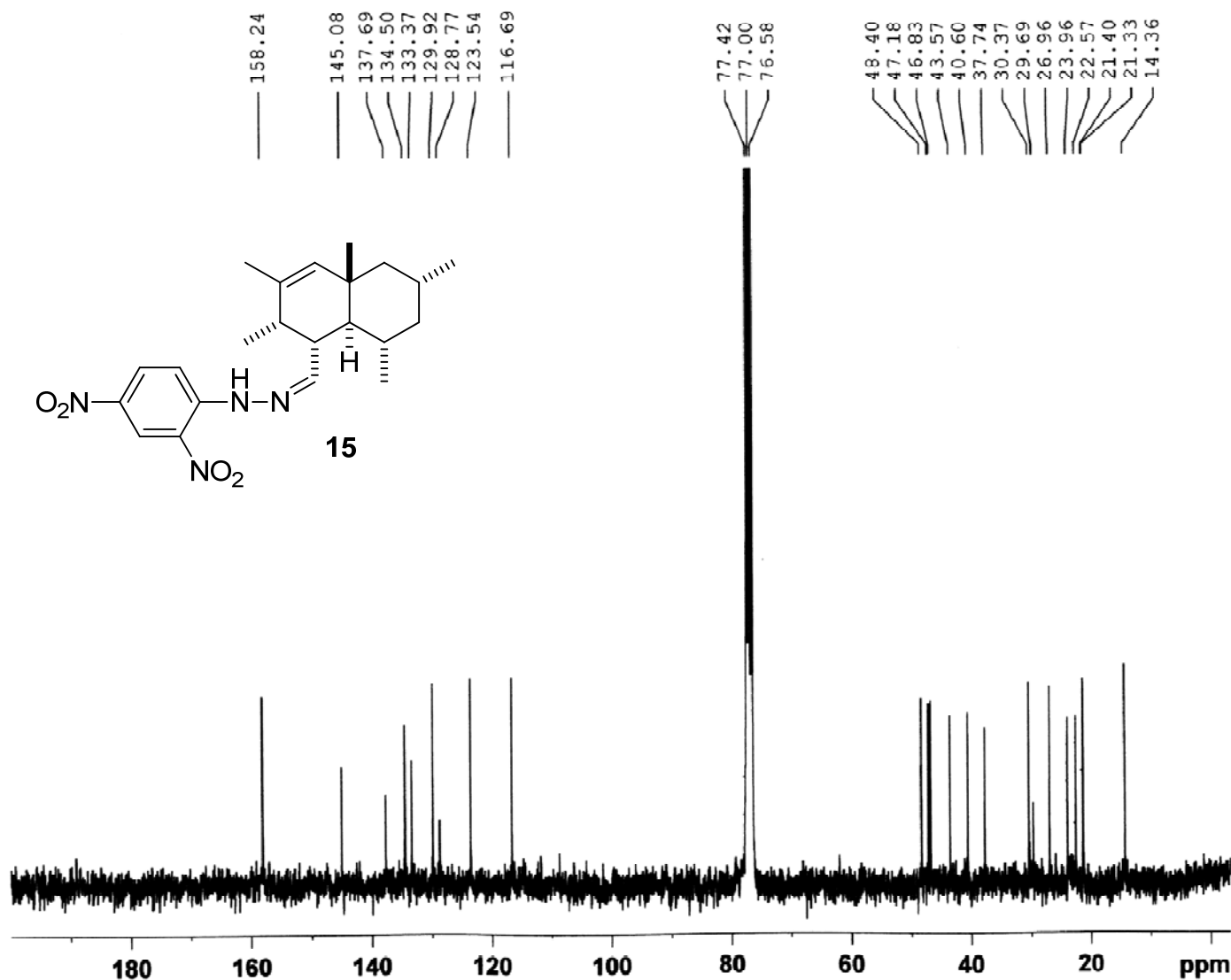
----- CHANNEL F1 -----
NUC1: 1H
P1: 11.70 usec
PL1: 0.00 dB
SFO1: 300.1801012 MHz

F2 - Processing parameters
SI: 16384
SF: 300.1730262 MHz
WDW: EM
SSB: 0
LB: 0.10 Hz
GB: 0
PC: 1.00



^1H NMR (300 MHz, CDCl_3) of Compound 15

Endo, IMDA, 2,4DNP, pure



Current Data Parameters
NAME ramnath 28-02-2012
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date 20120229
Time 0.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 8000
DS 0
SWH 17857.143 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 362
DN 28.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.09999998 sec
TDO 1

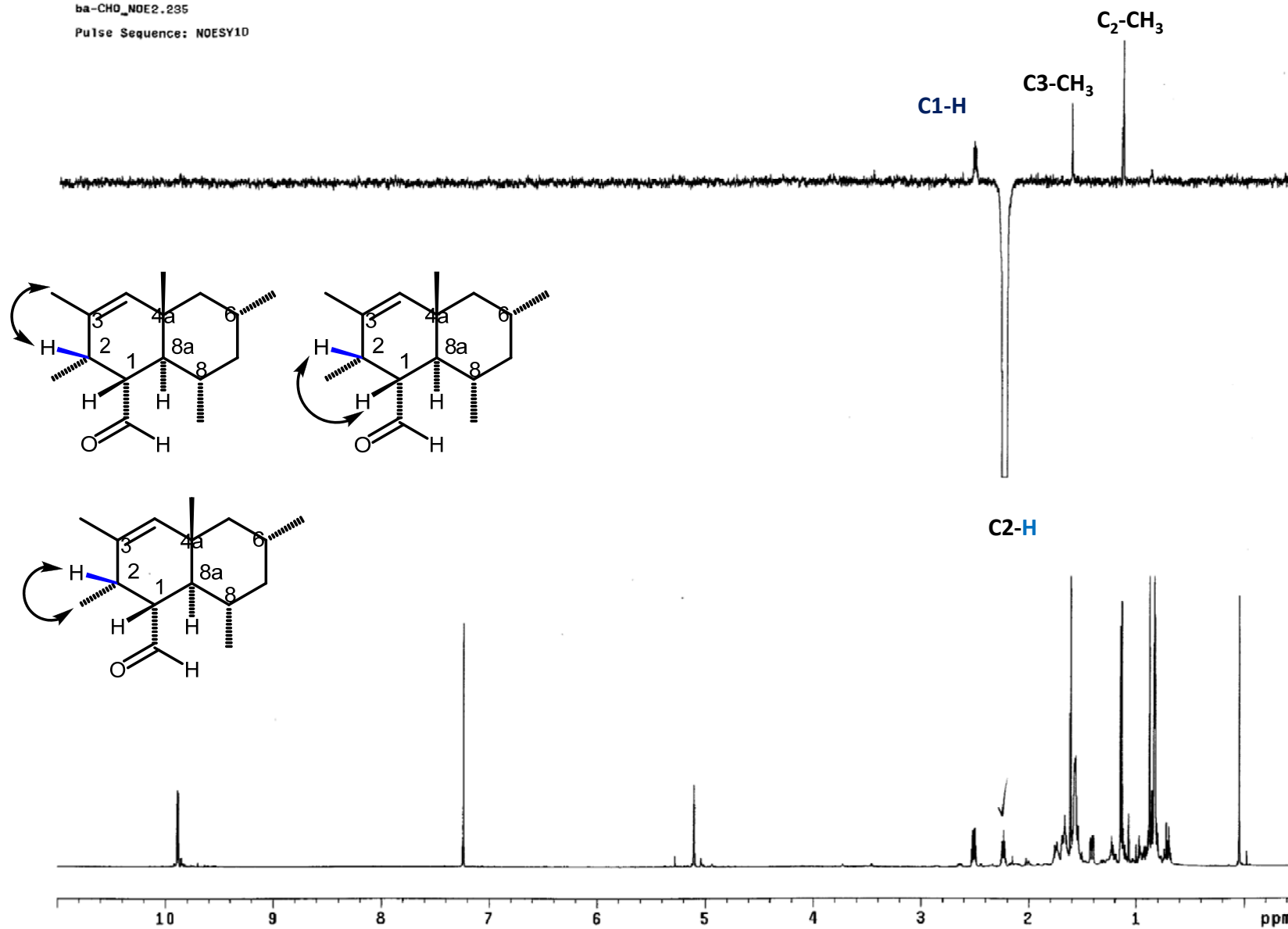
===== CHANNEL f1 =====
NUC1 13C
P1 9.85 usec
PL1 0.00 dB
SFO1 75.4884982 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL12 17.70 dB
PL13 20.70 dB
PL2 0.00 dB
SFO2 300.1792007 MHz

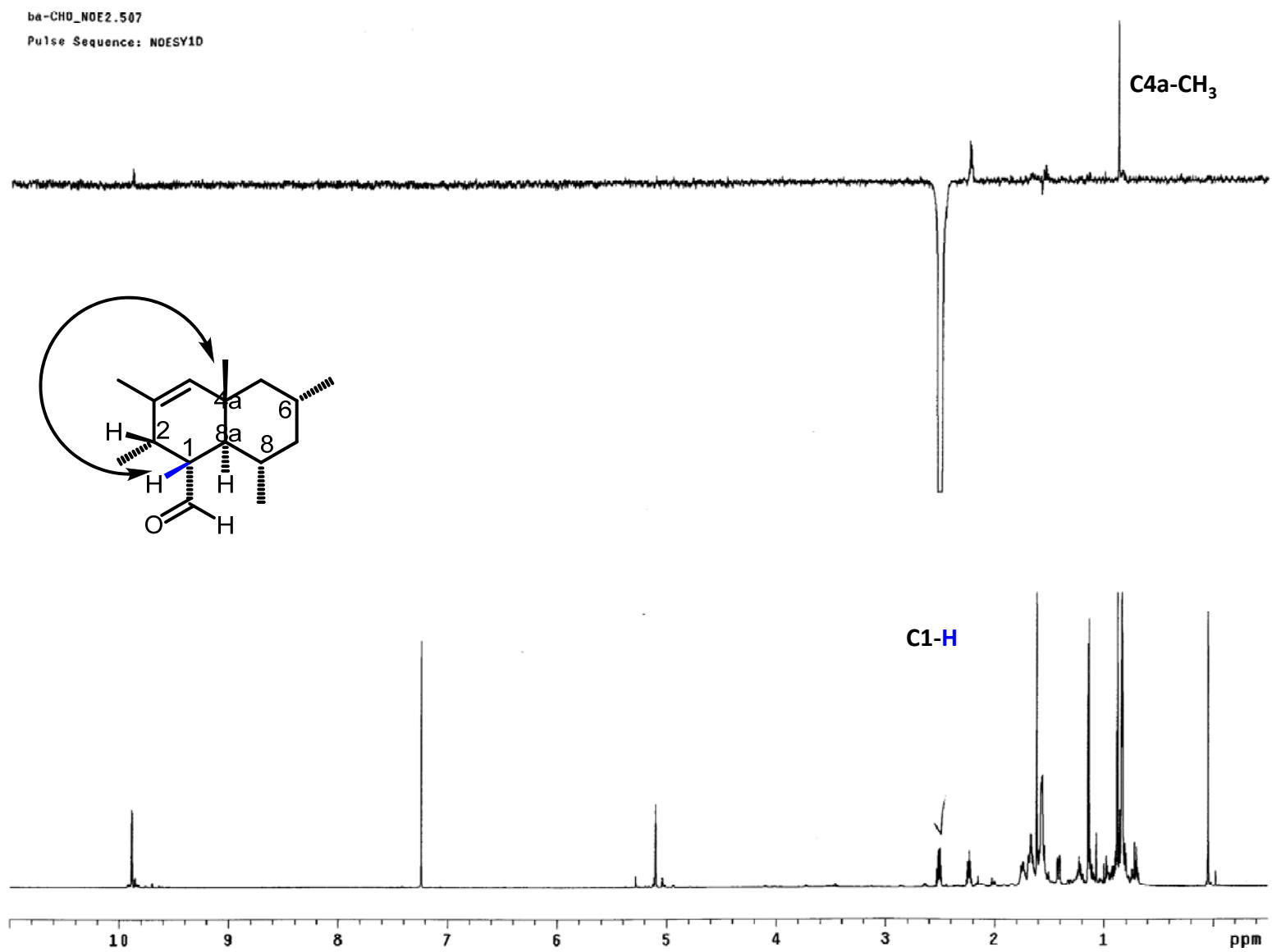
F2 - Processing parameters
SI 16384
SF 75.4798206 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

¹³C NMR (75 MHz, CDCl₃) of Compound 15

ba-CHO_NOE2.235
Pulse Sequence: NOESY1D

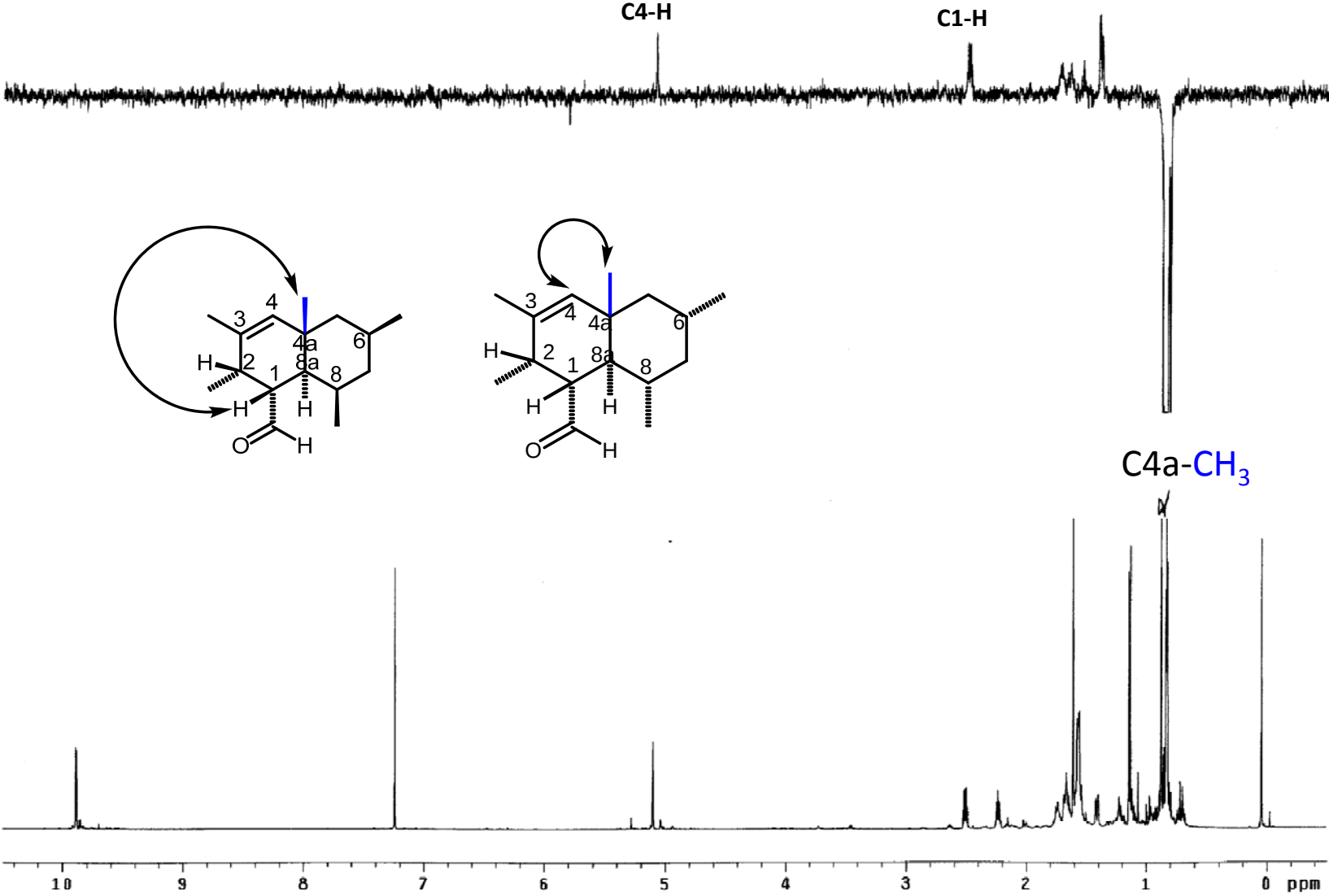


NOE of 13a

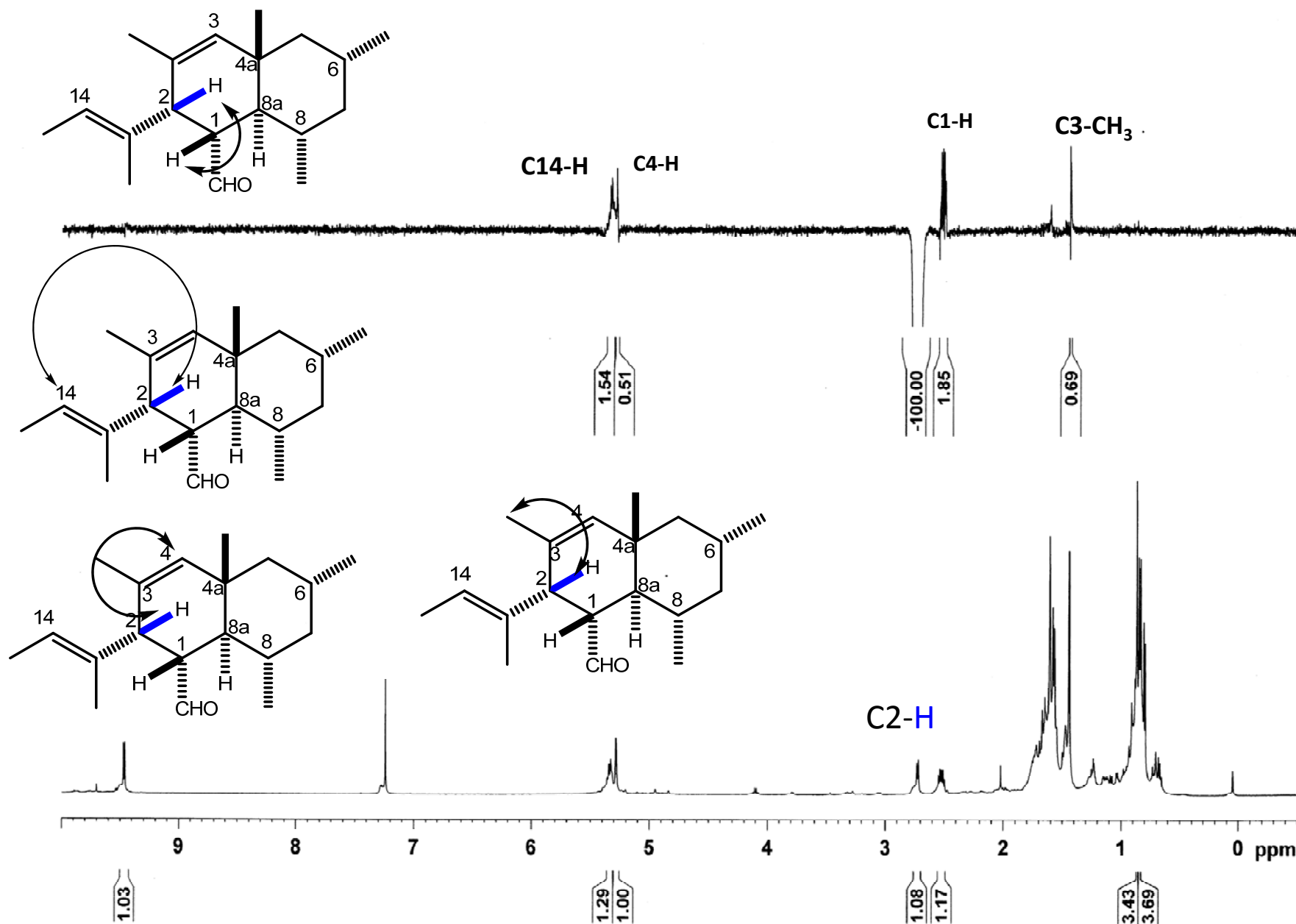


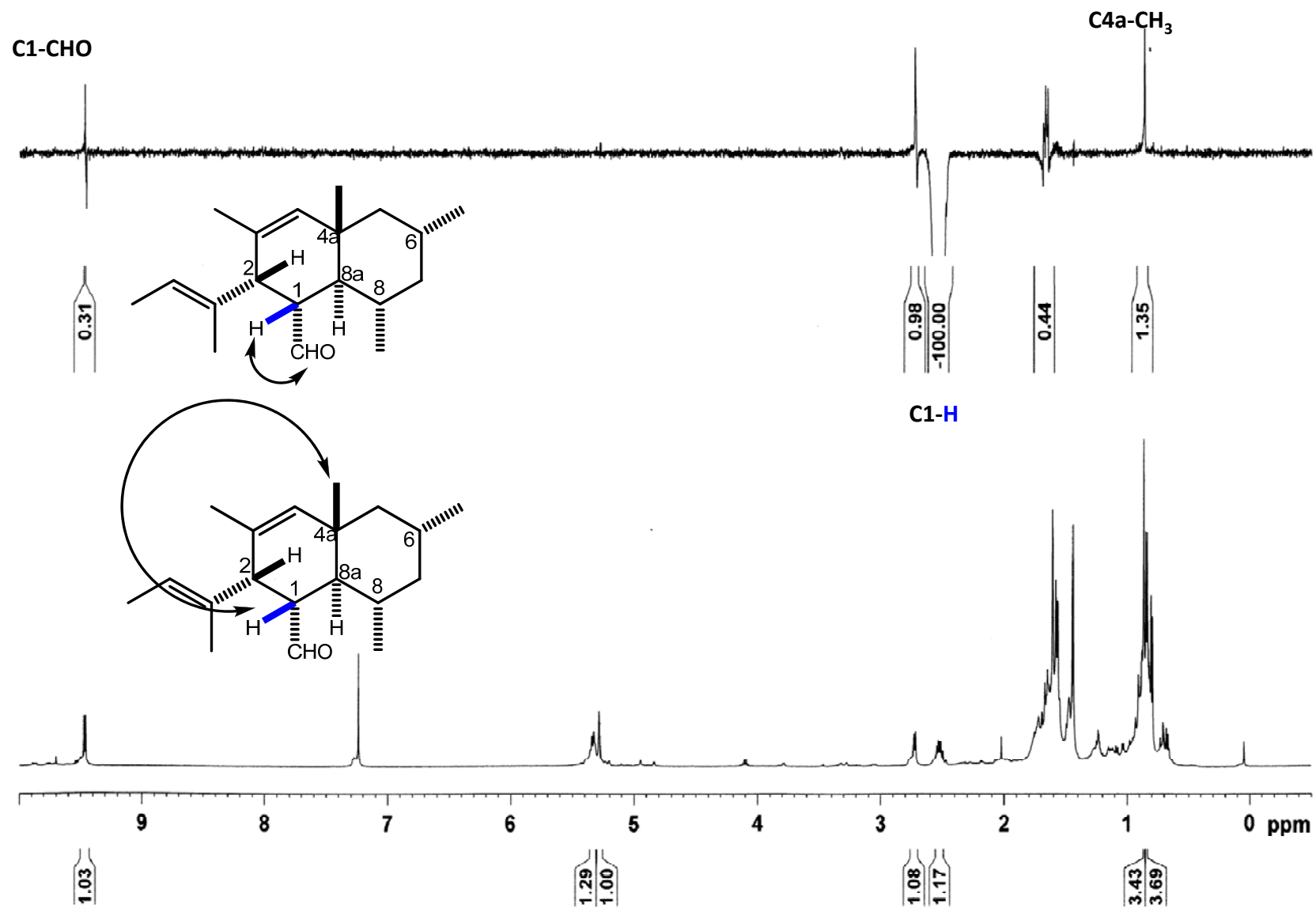
NOE of 13a

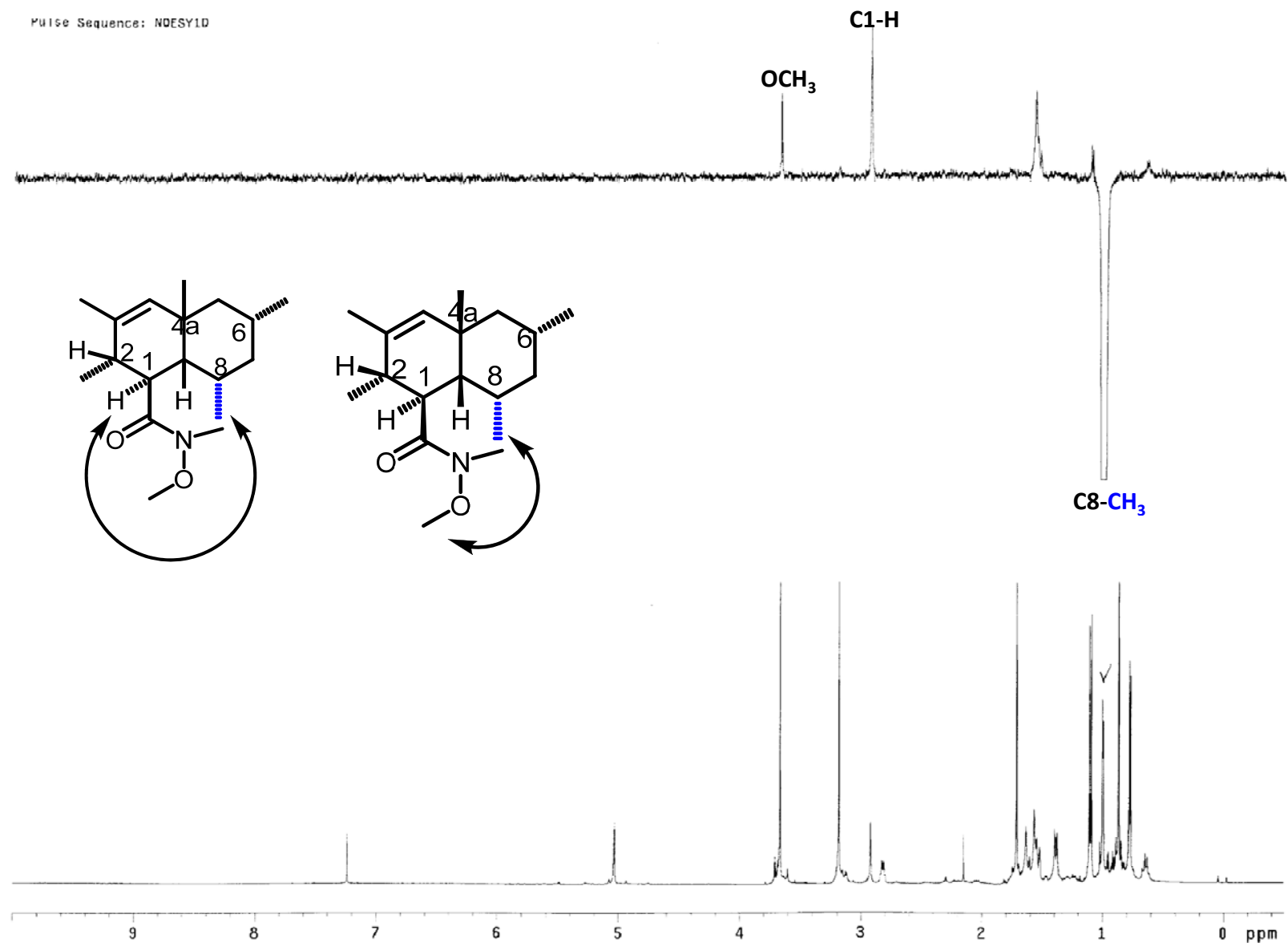
ba-CHO_NOE0.856
Pulse Sequence: NOESY1D



NOE of 13a



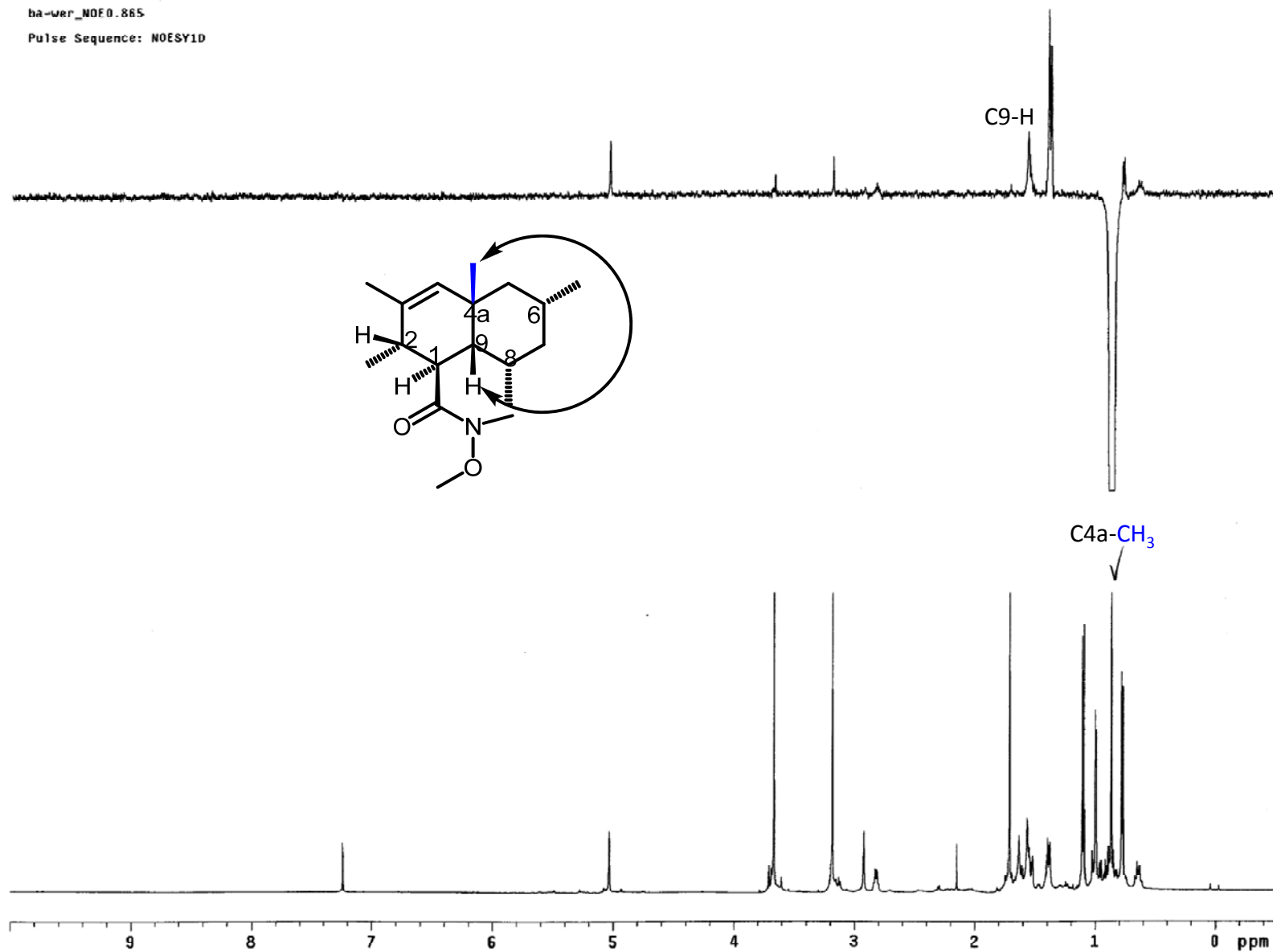




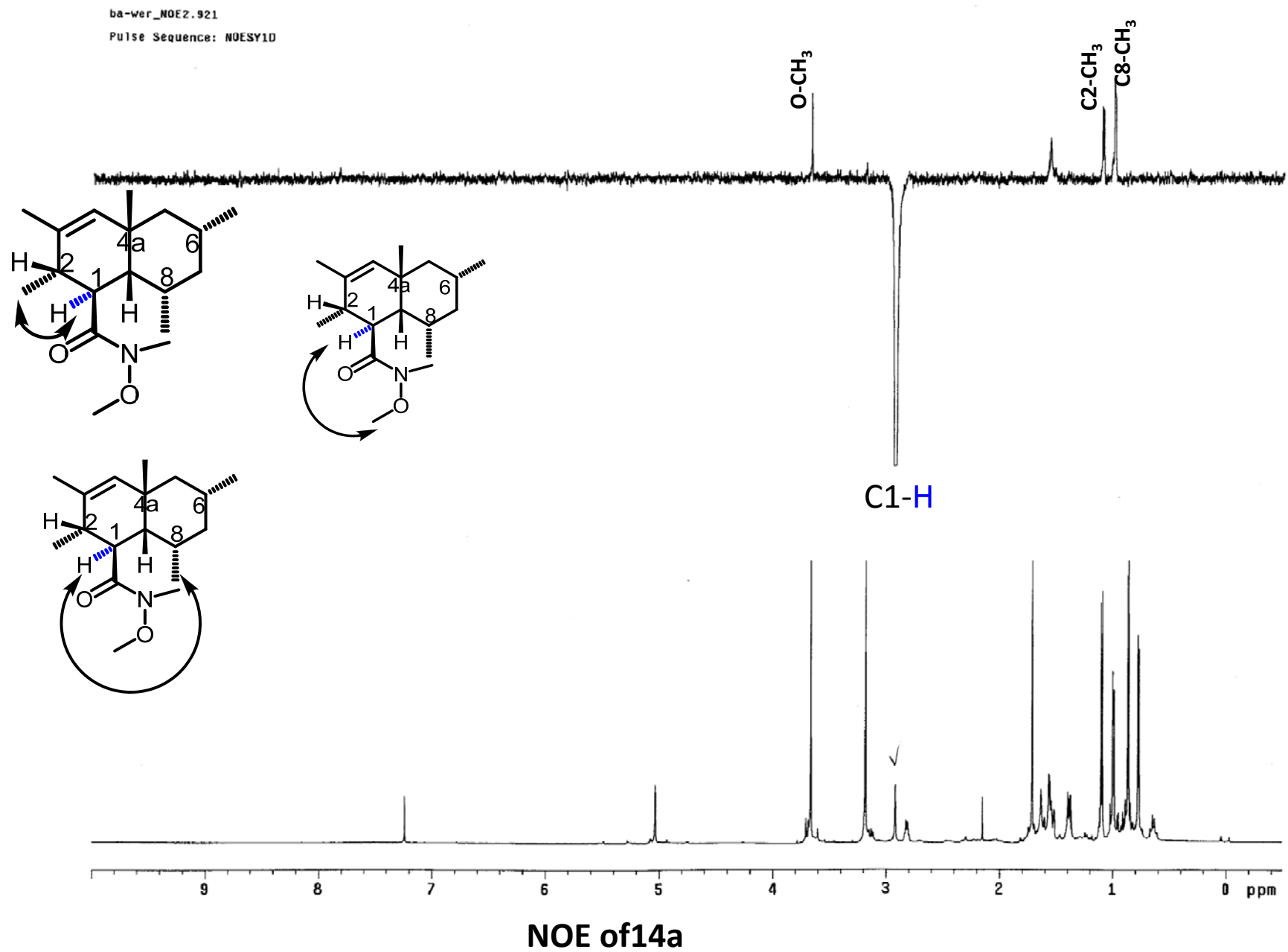
NOE of 14a

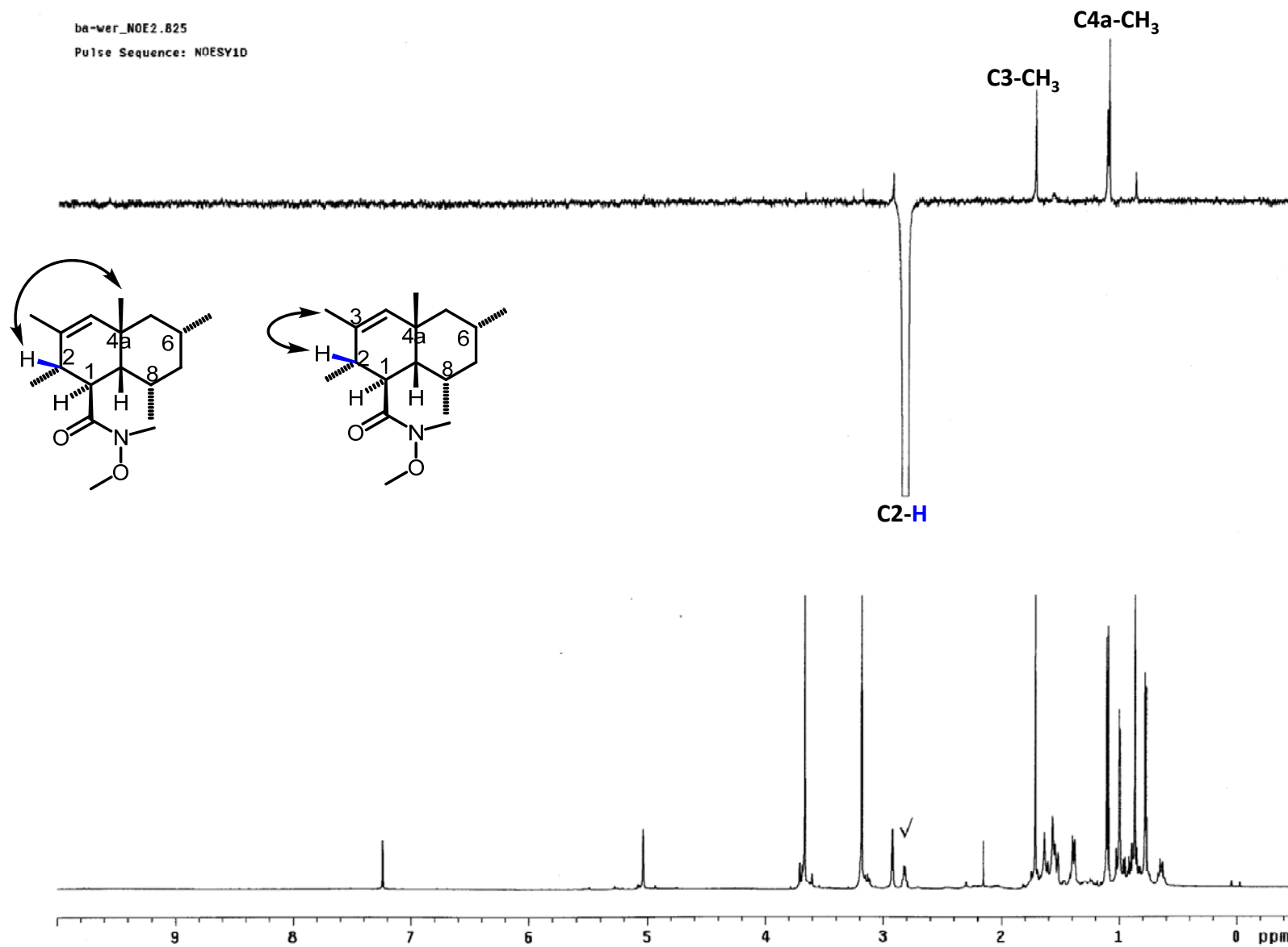
ba-wer_NOF0.865

Pulse Sequence: NOESY1D

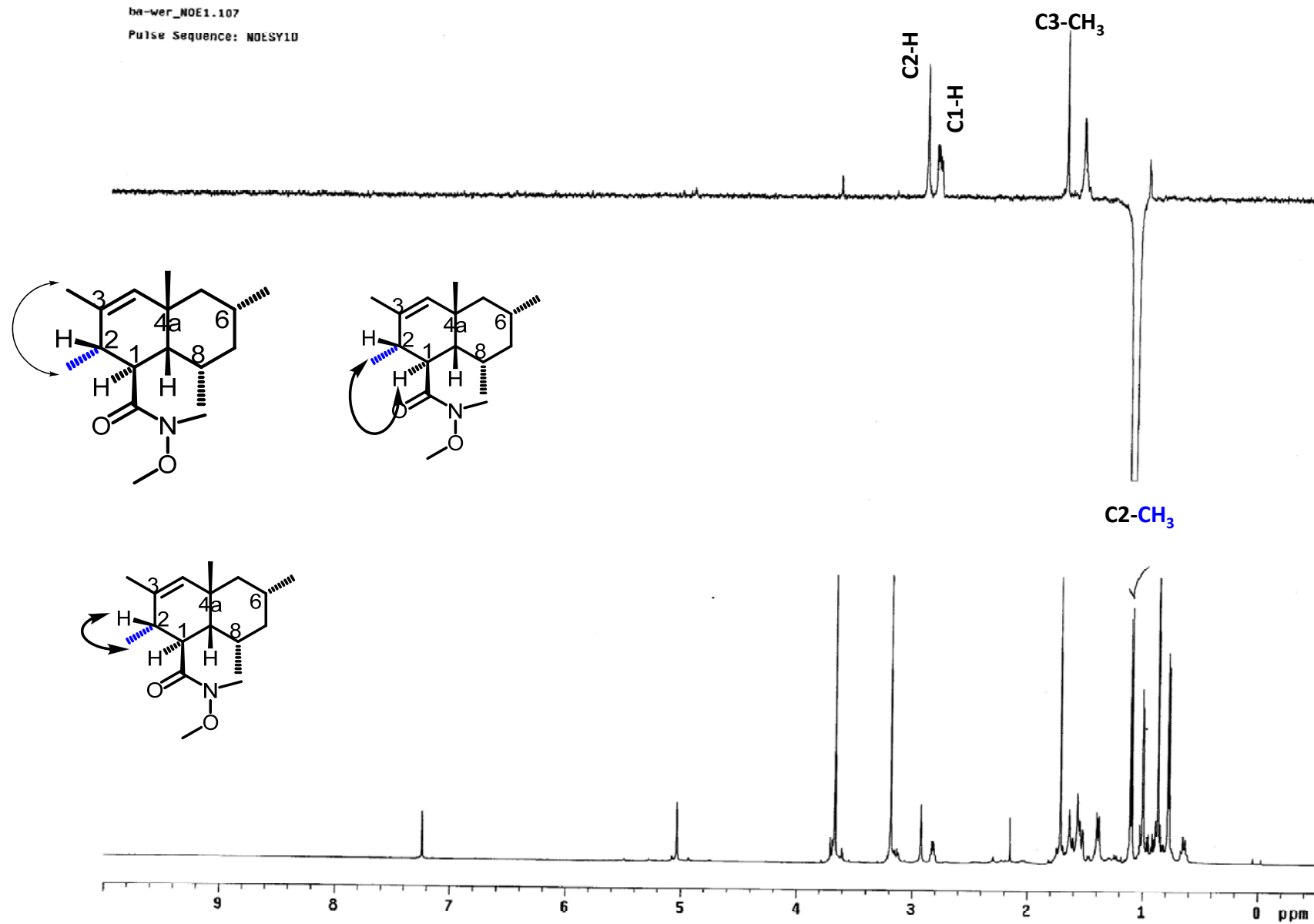


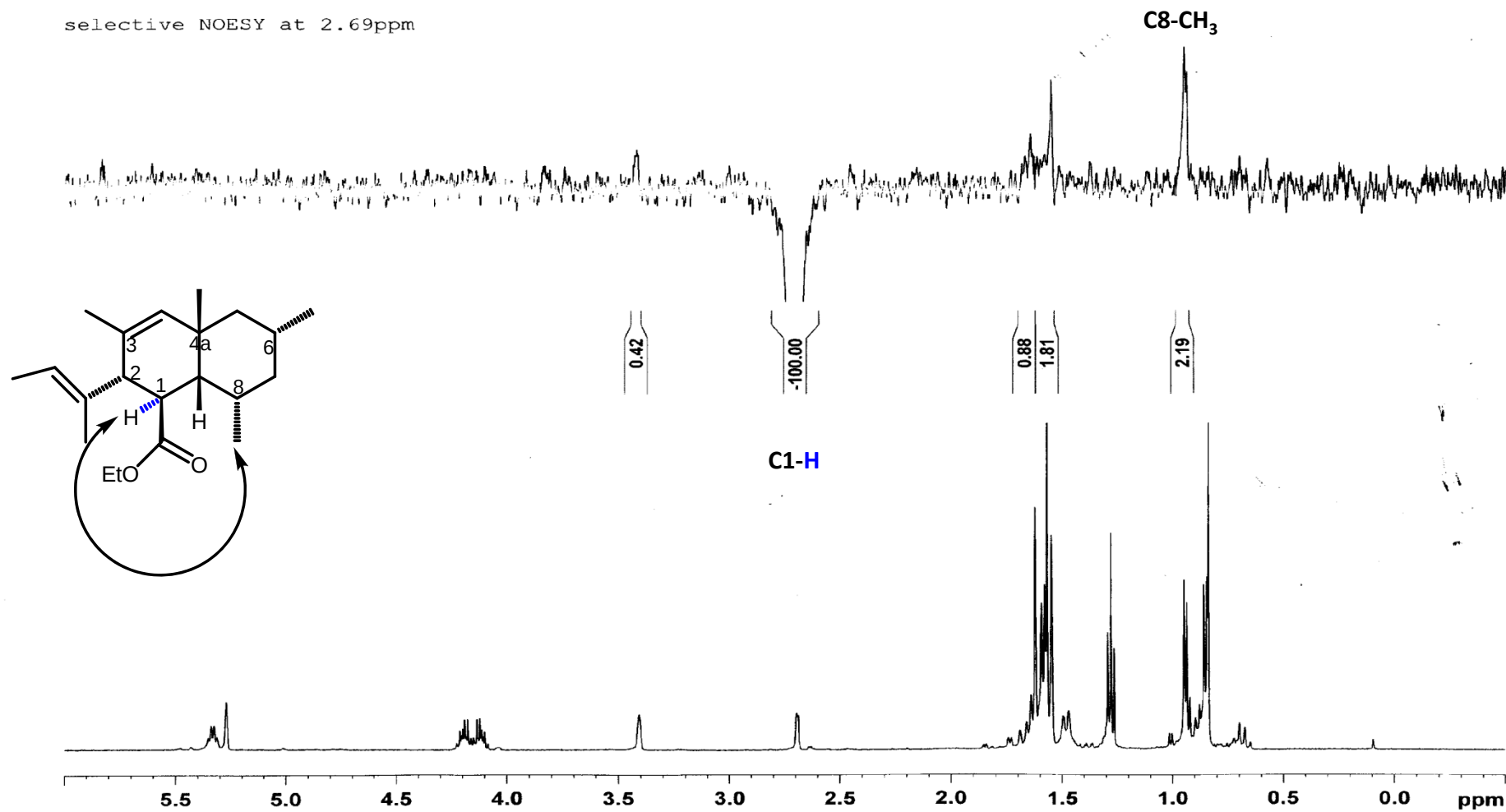
NOE of 14a





NOE of 14a





NOE of 14c

selective NOESY at 0.84ppm

