Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

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 A          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	

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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
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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

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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
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Alert level G

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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
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```
  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

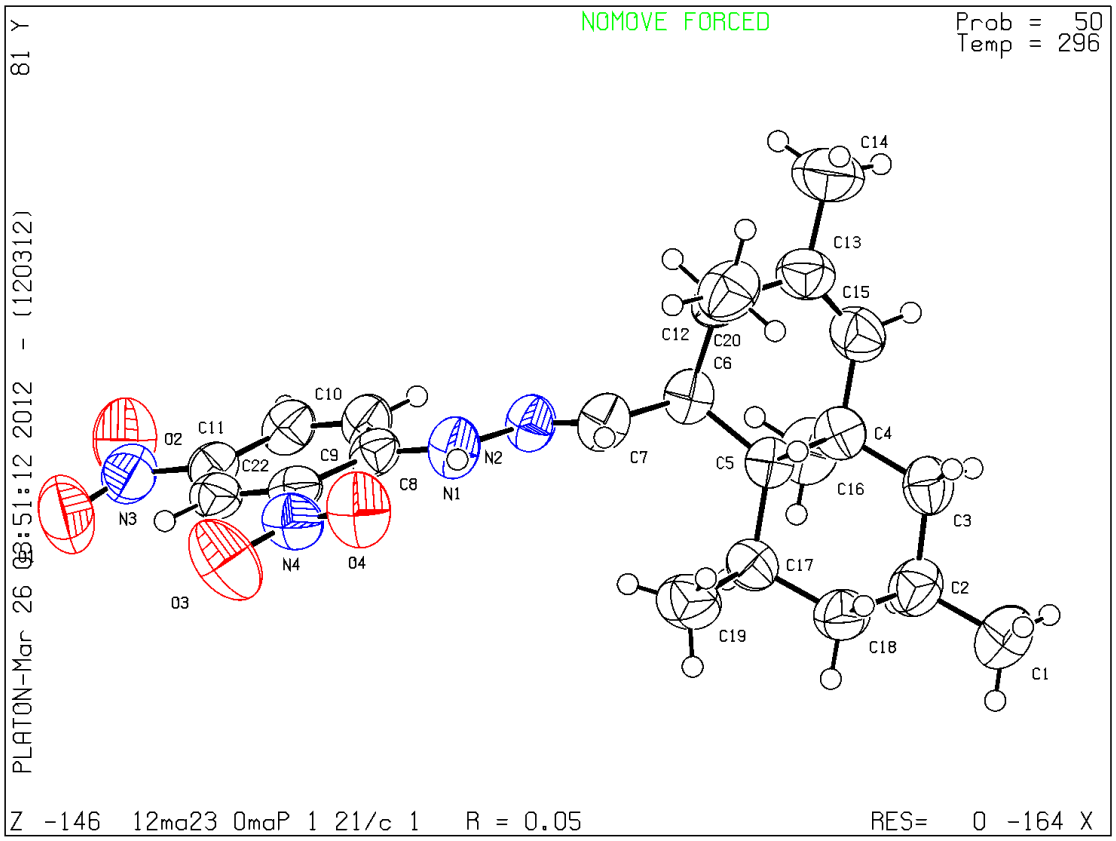
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section 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

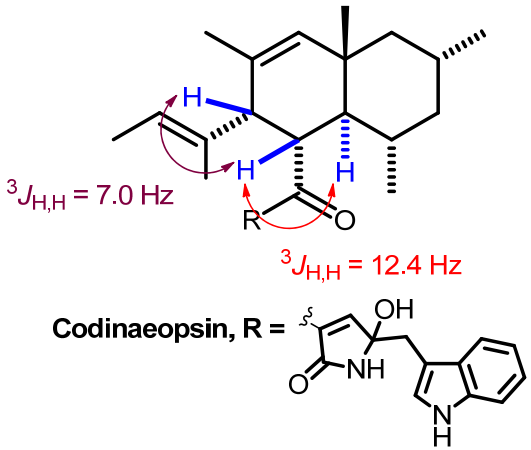
Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

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

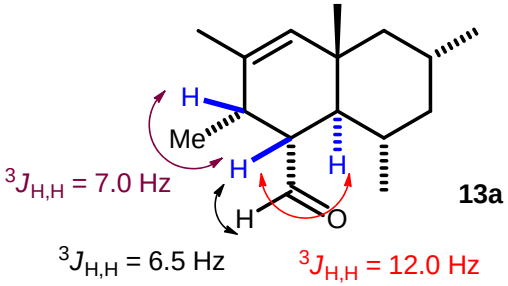
Datablock 12ma23_0ma - ellipsoid plot



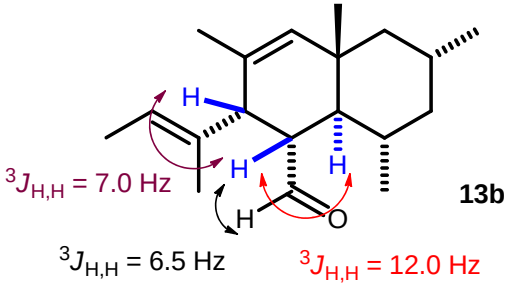
Observed coupling constants of *exo* and *endo* IMDA cycloadducts.



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



13a



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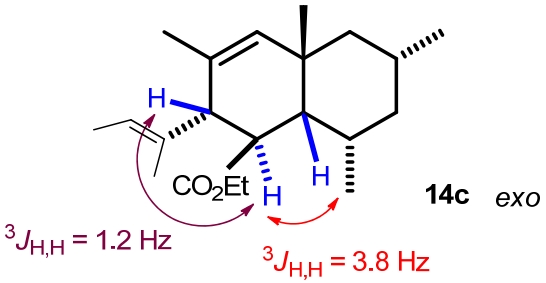
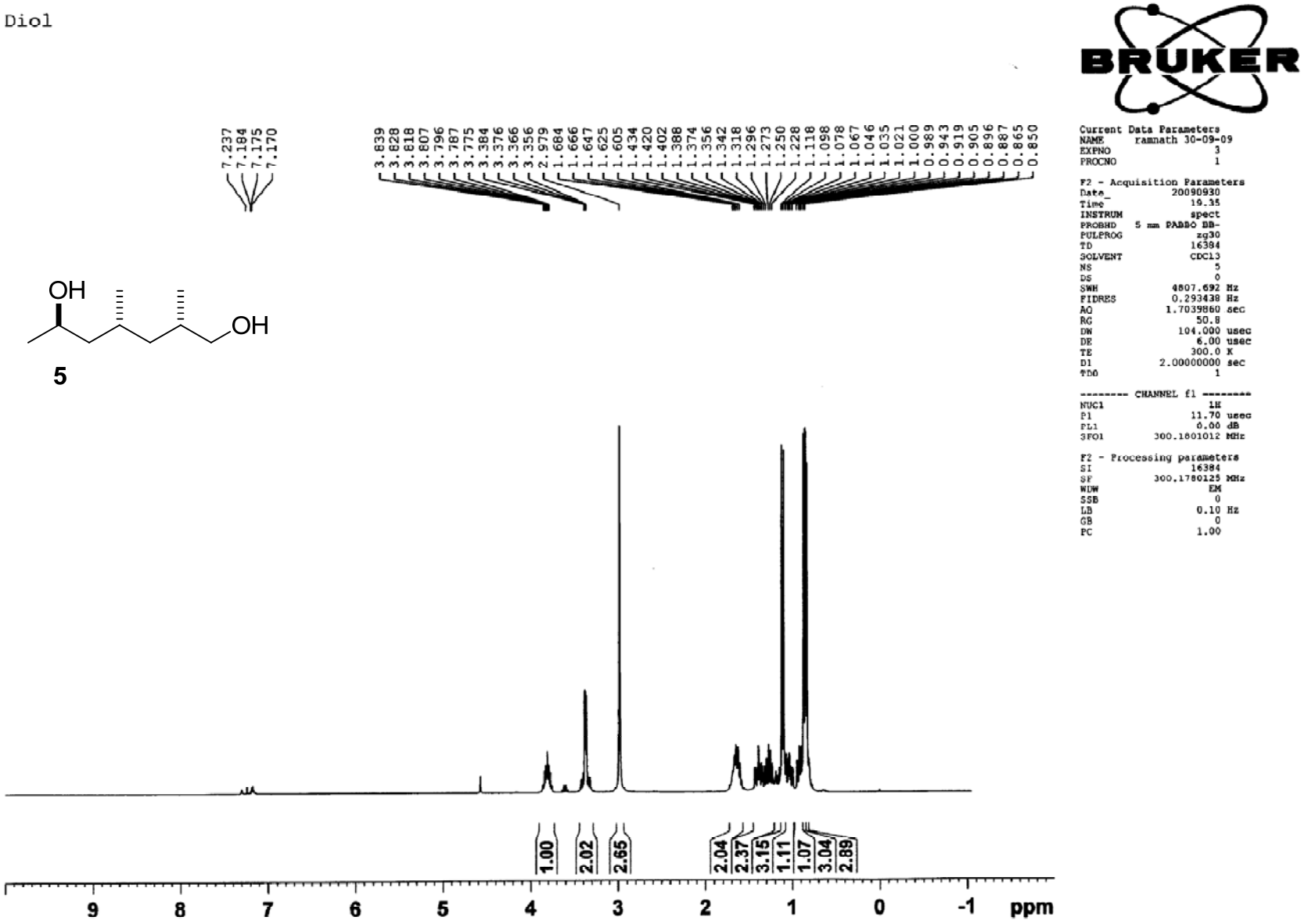


14c *exo*



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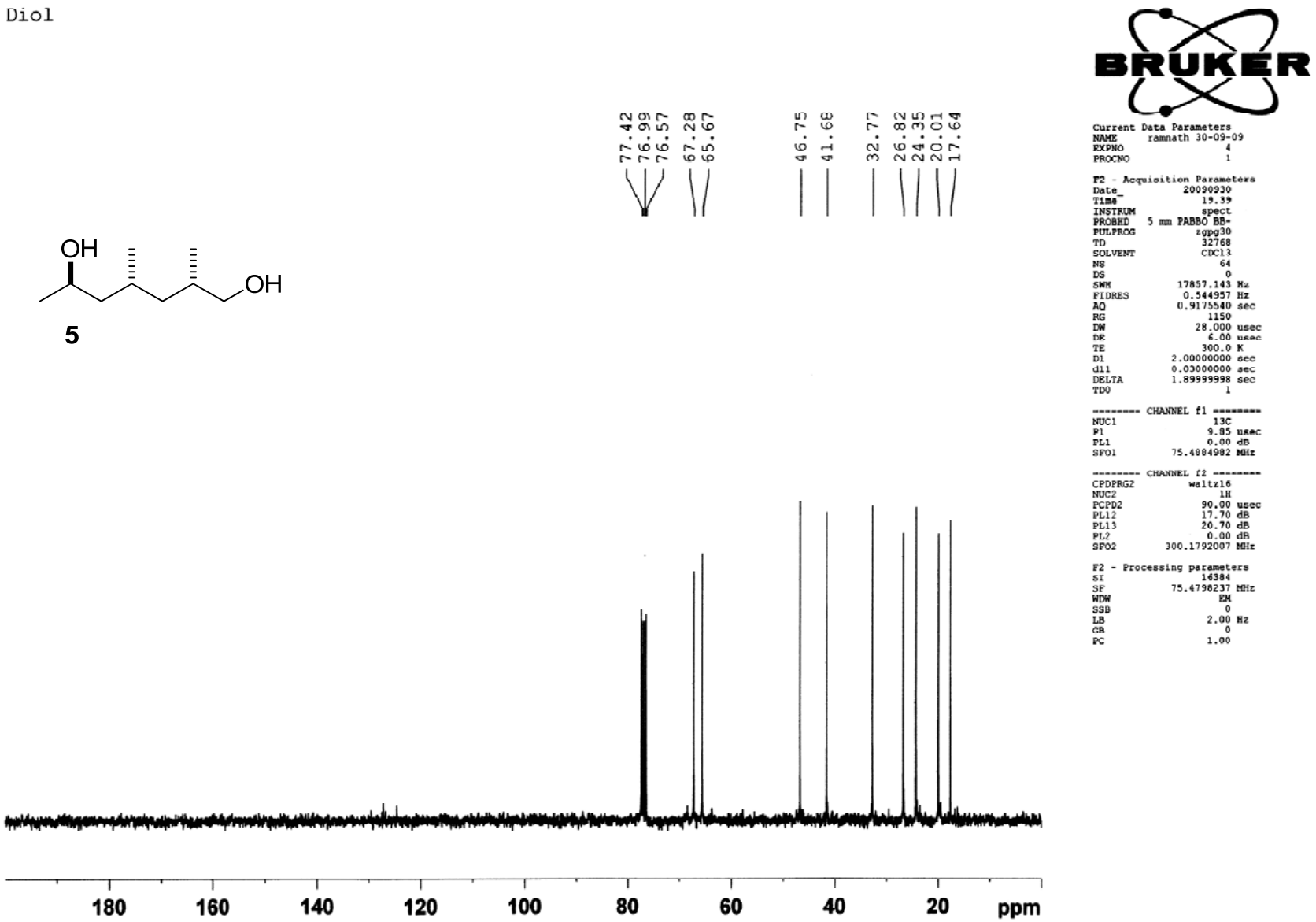

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d11 0.03000000 sec
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SFO1 75.4884902 MHz

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PCPD2 90.00 usec
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PL2 0.00 dB
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F2 - Processing parameters
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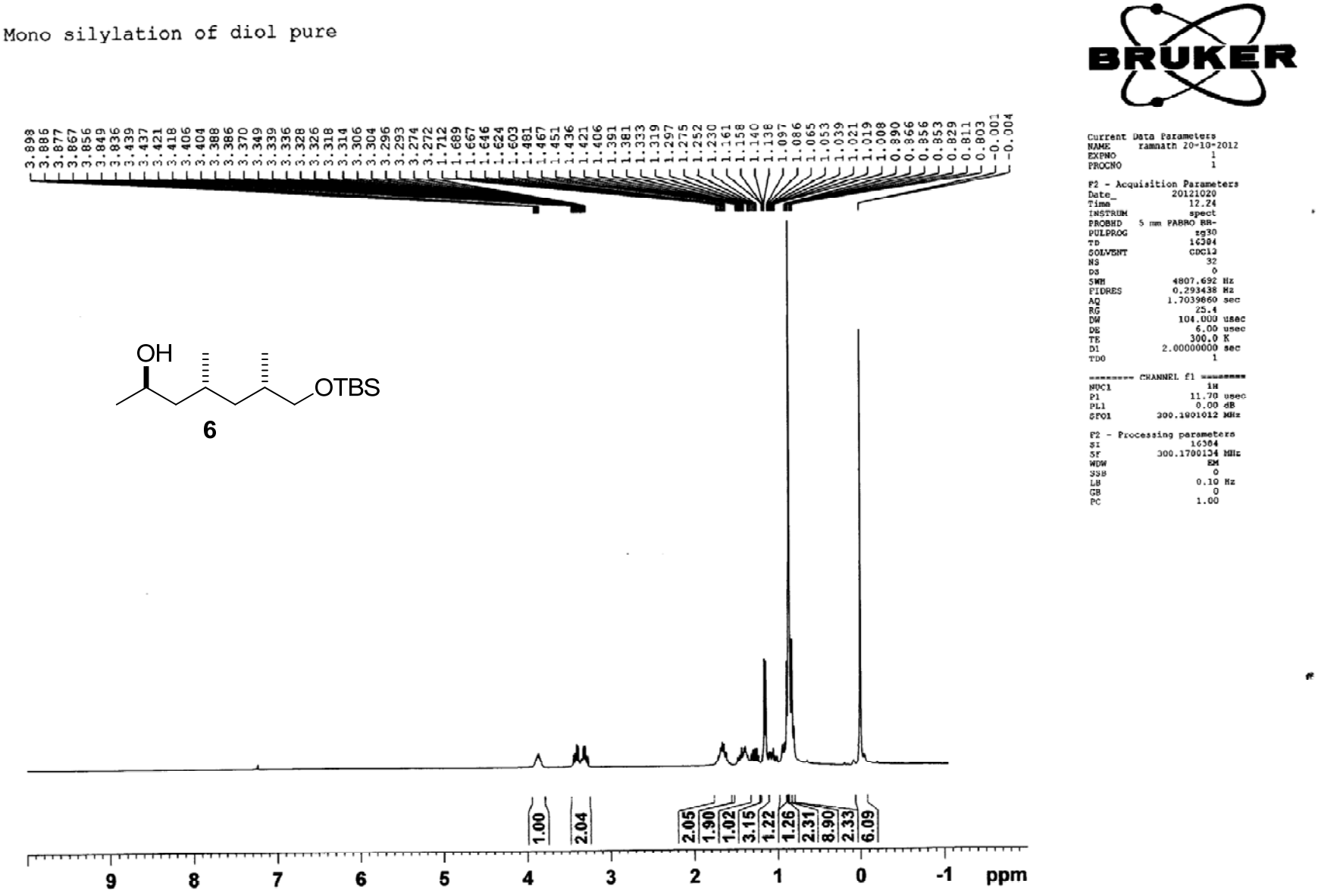

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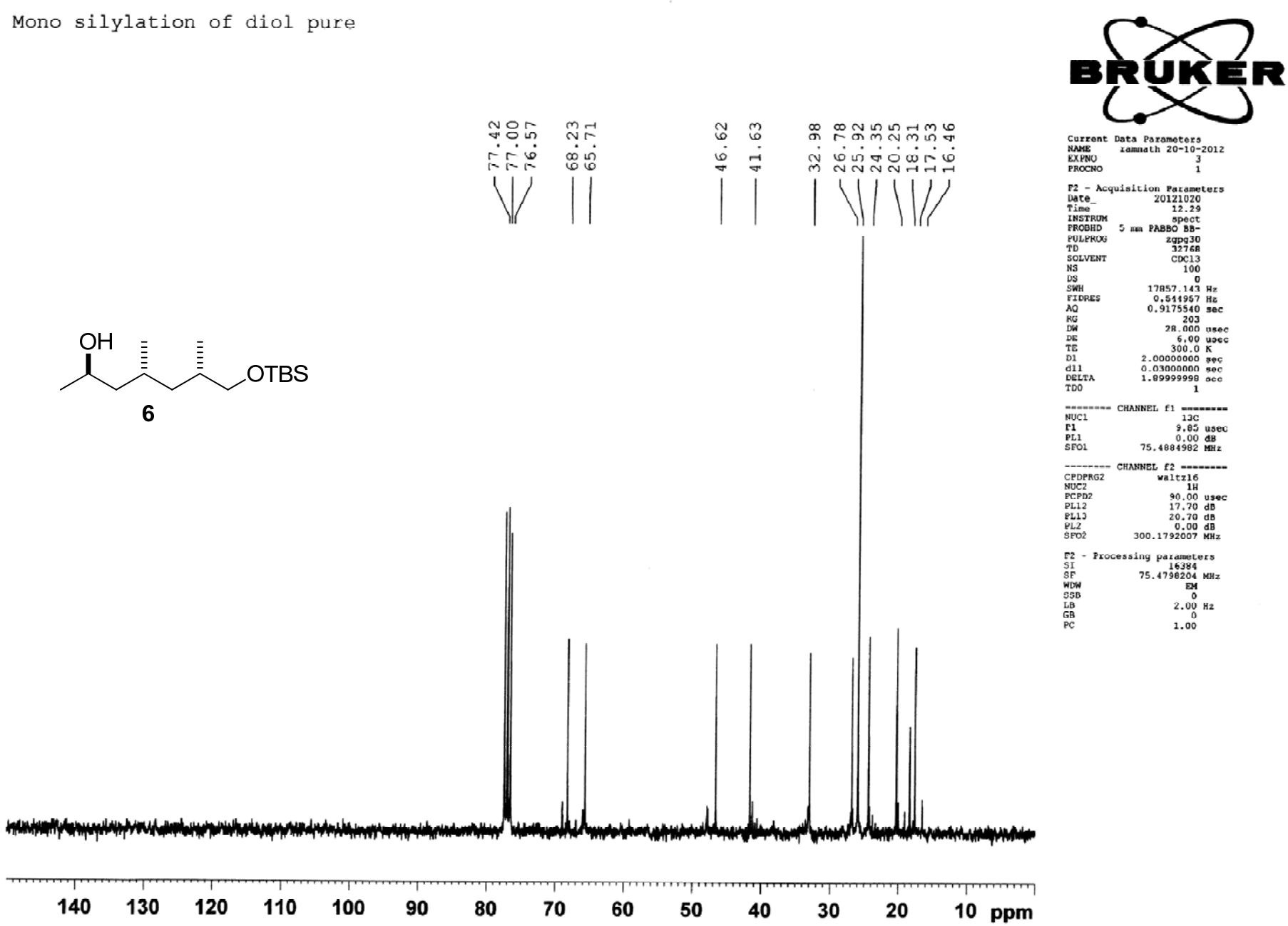

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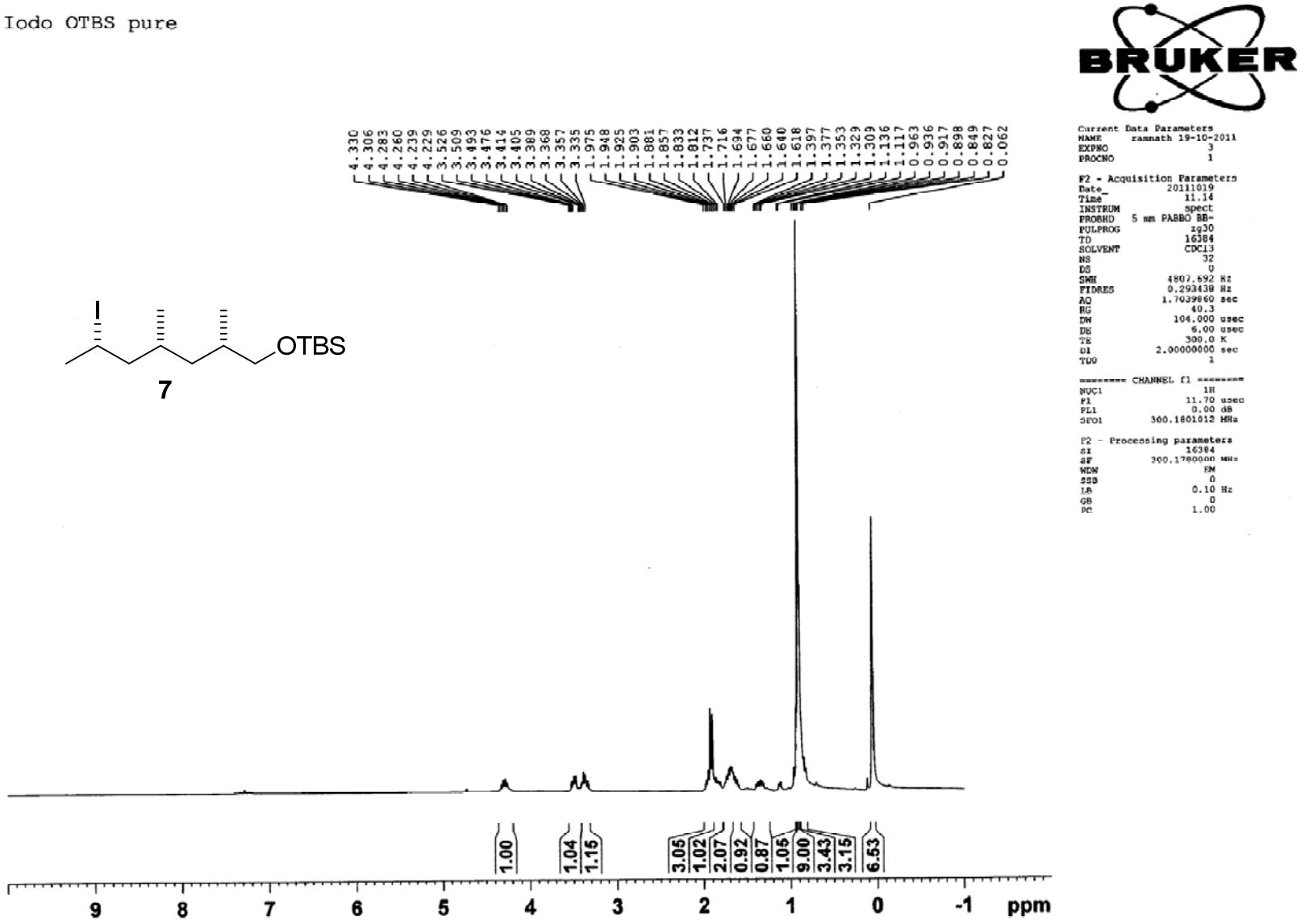

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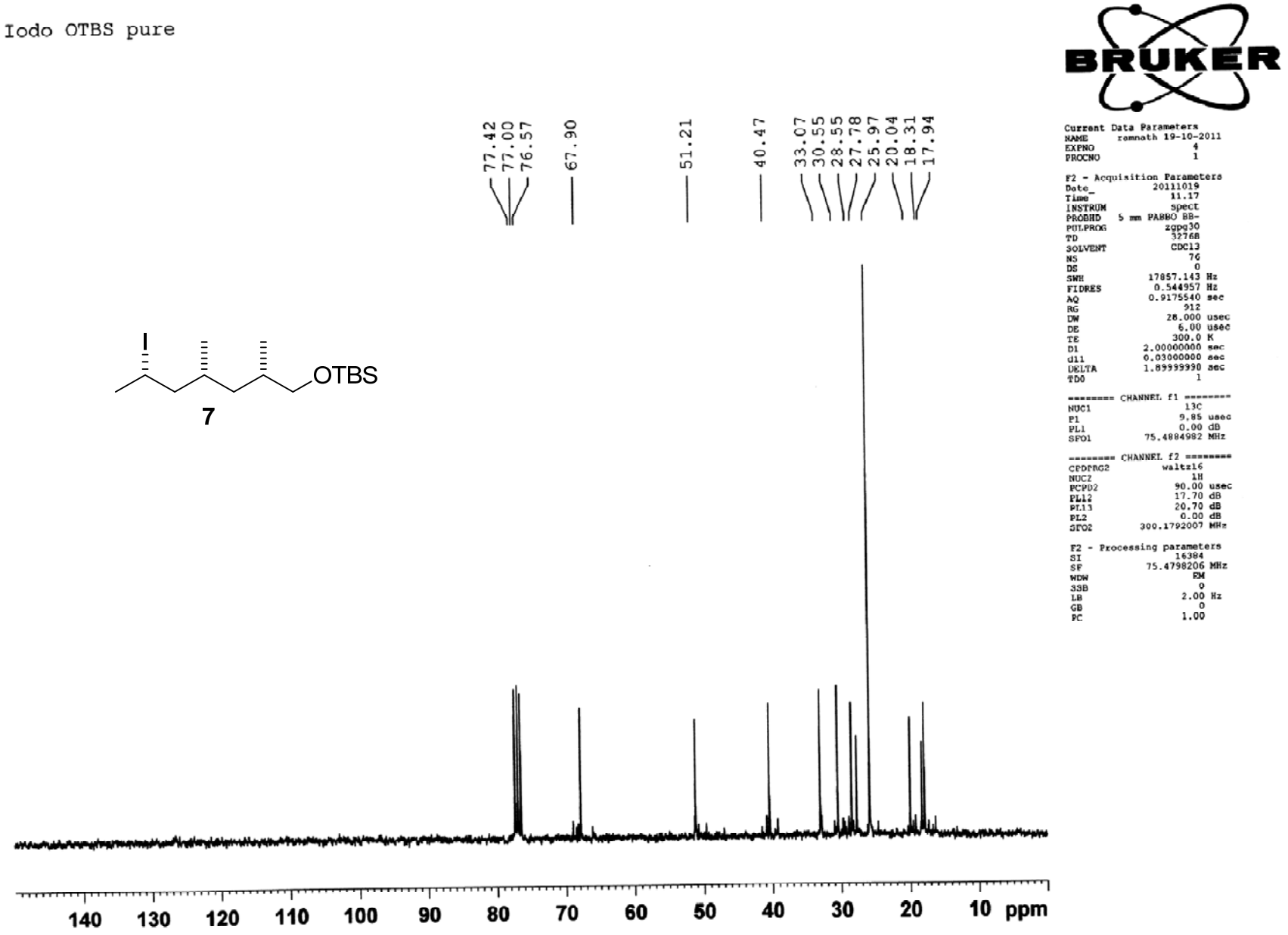

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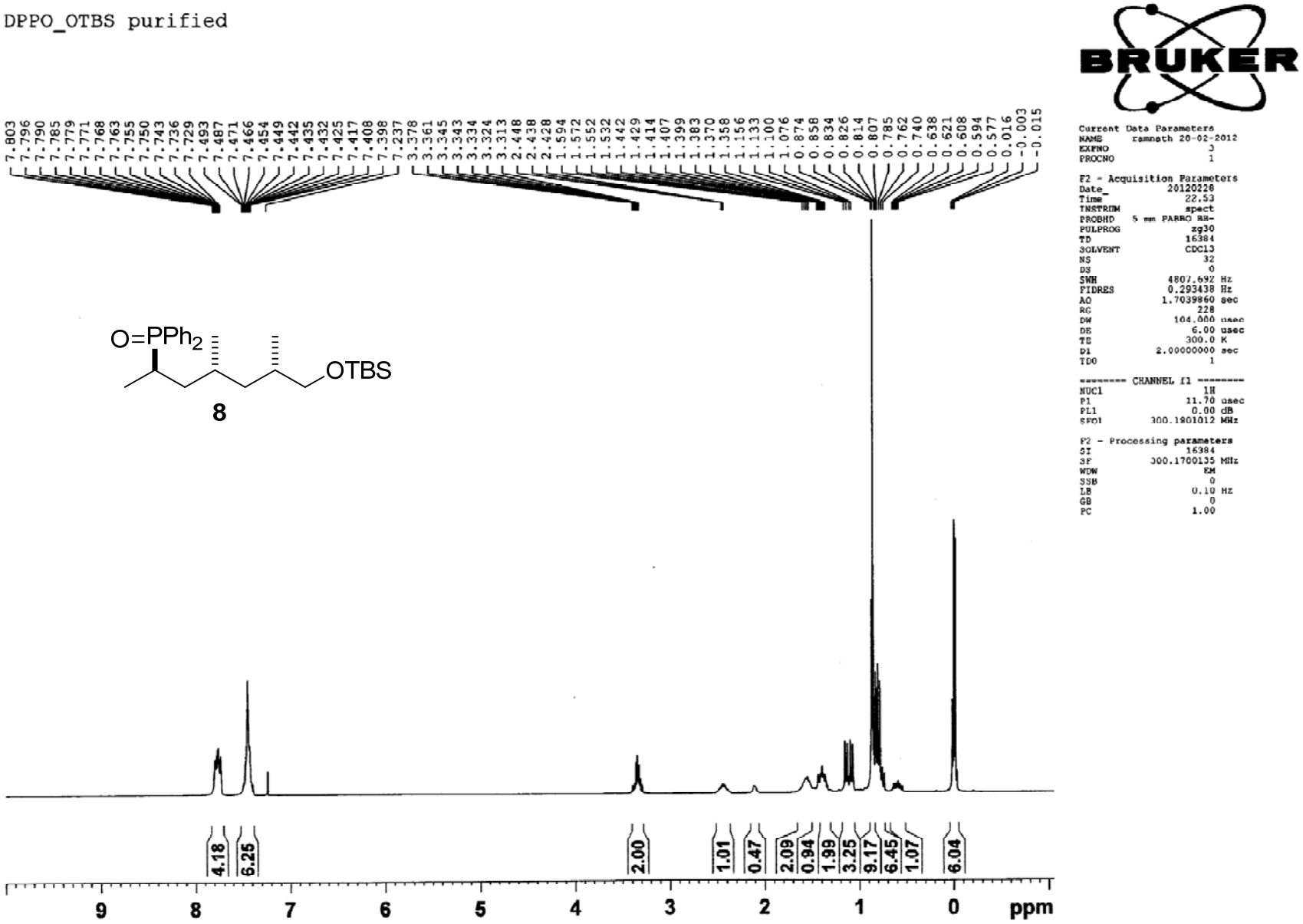

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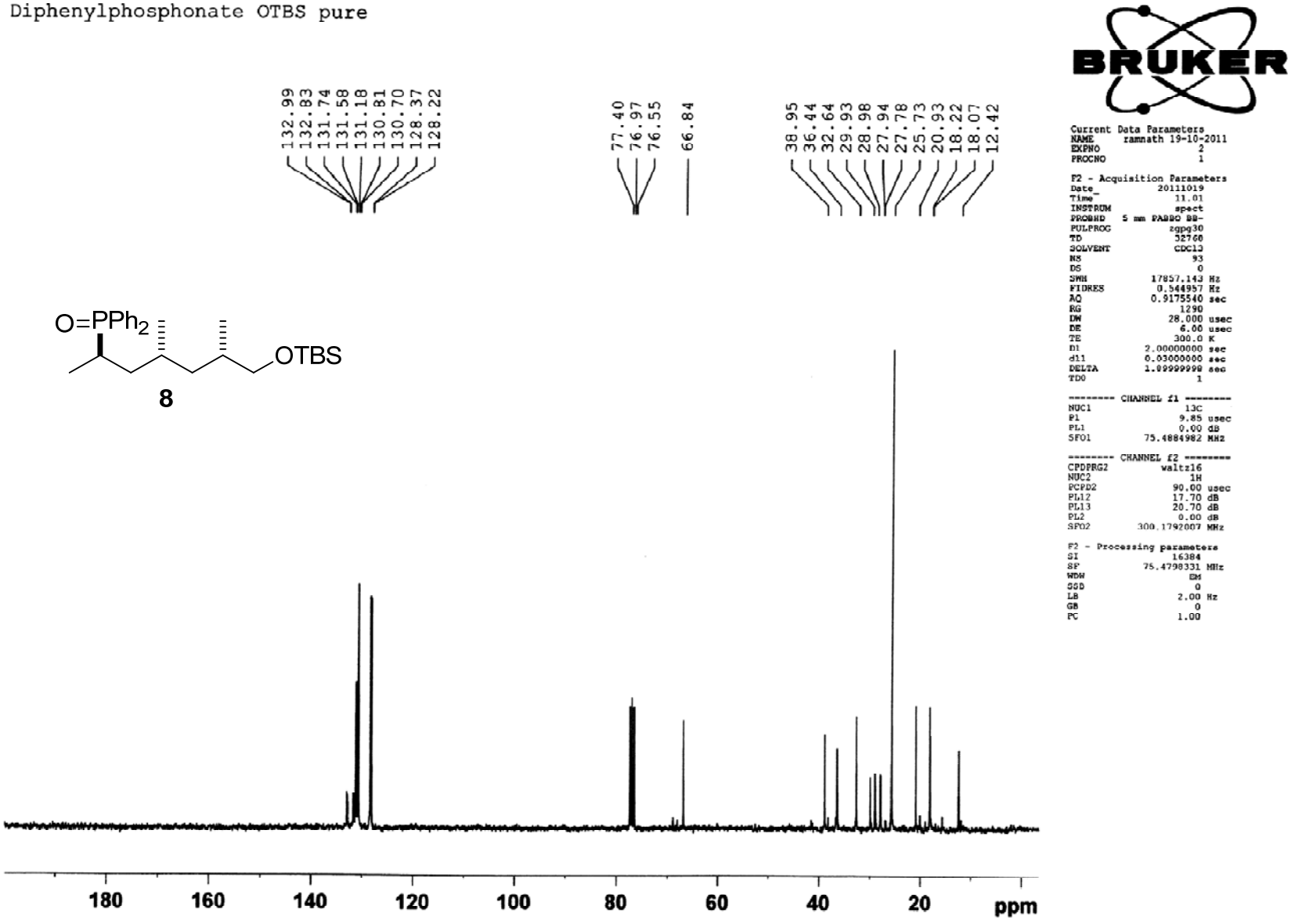

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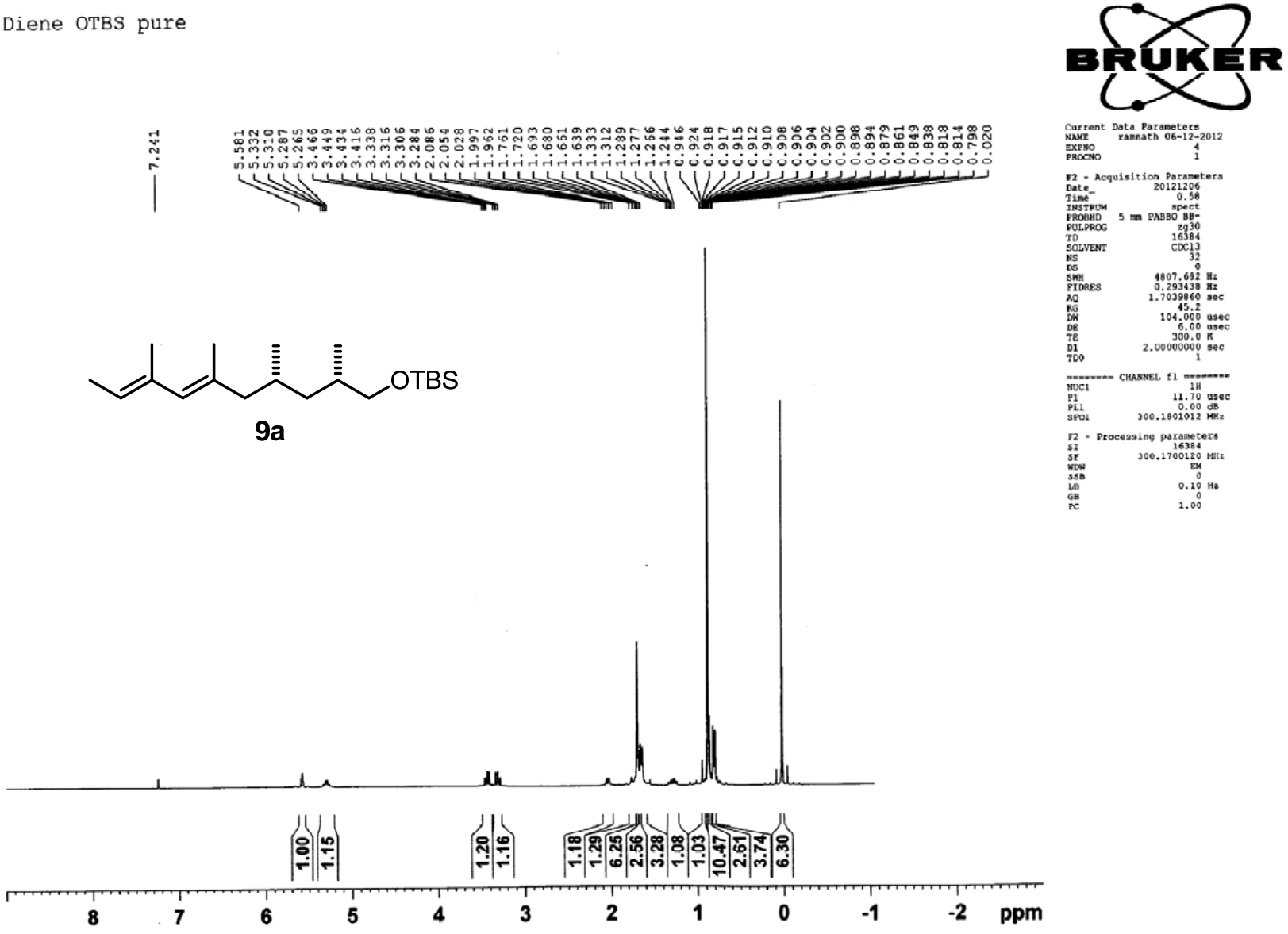

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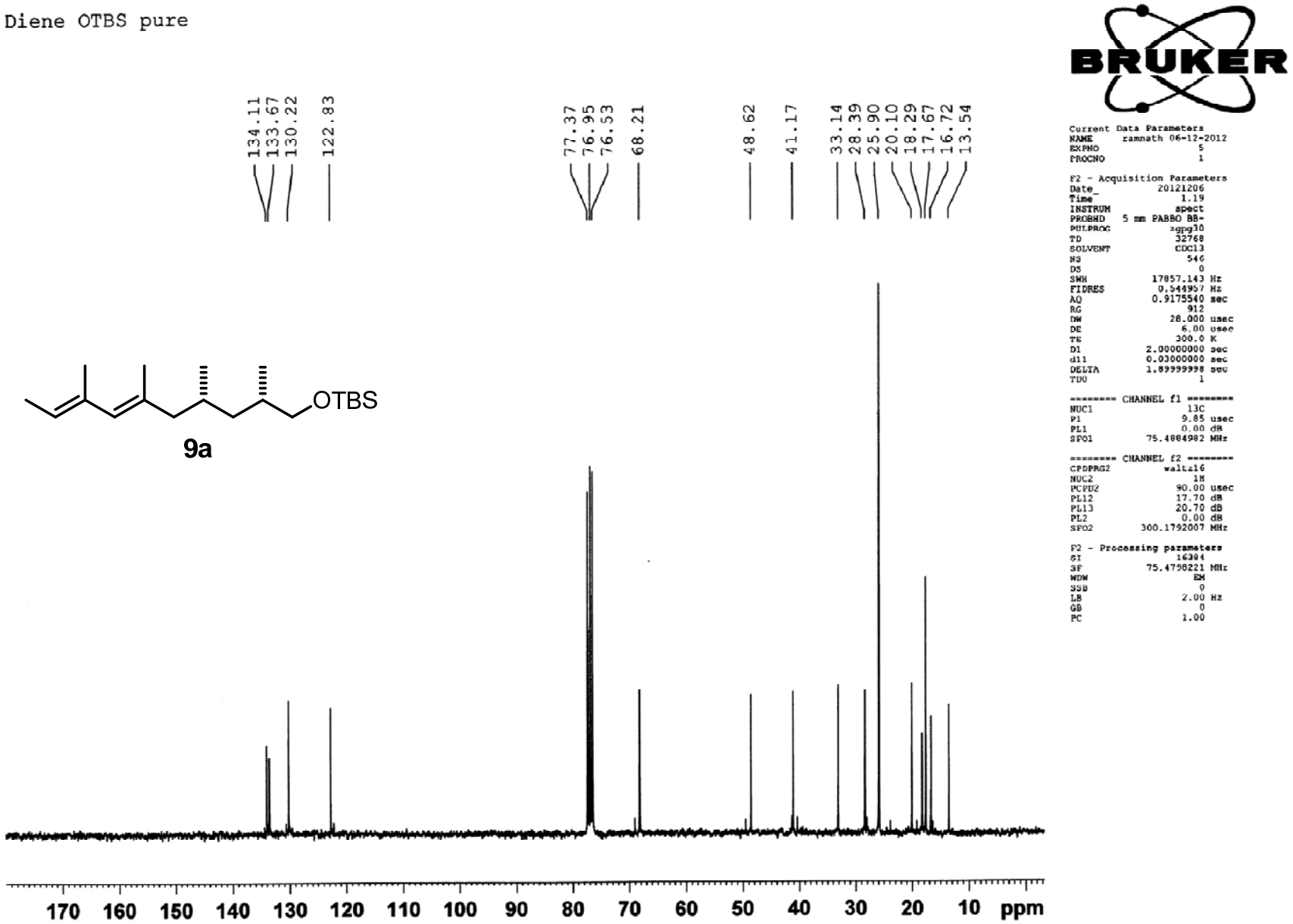

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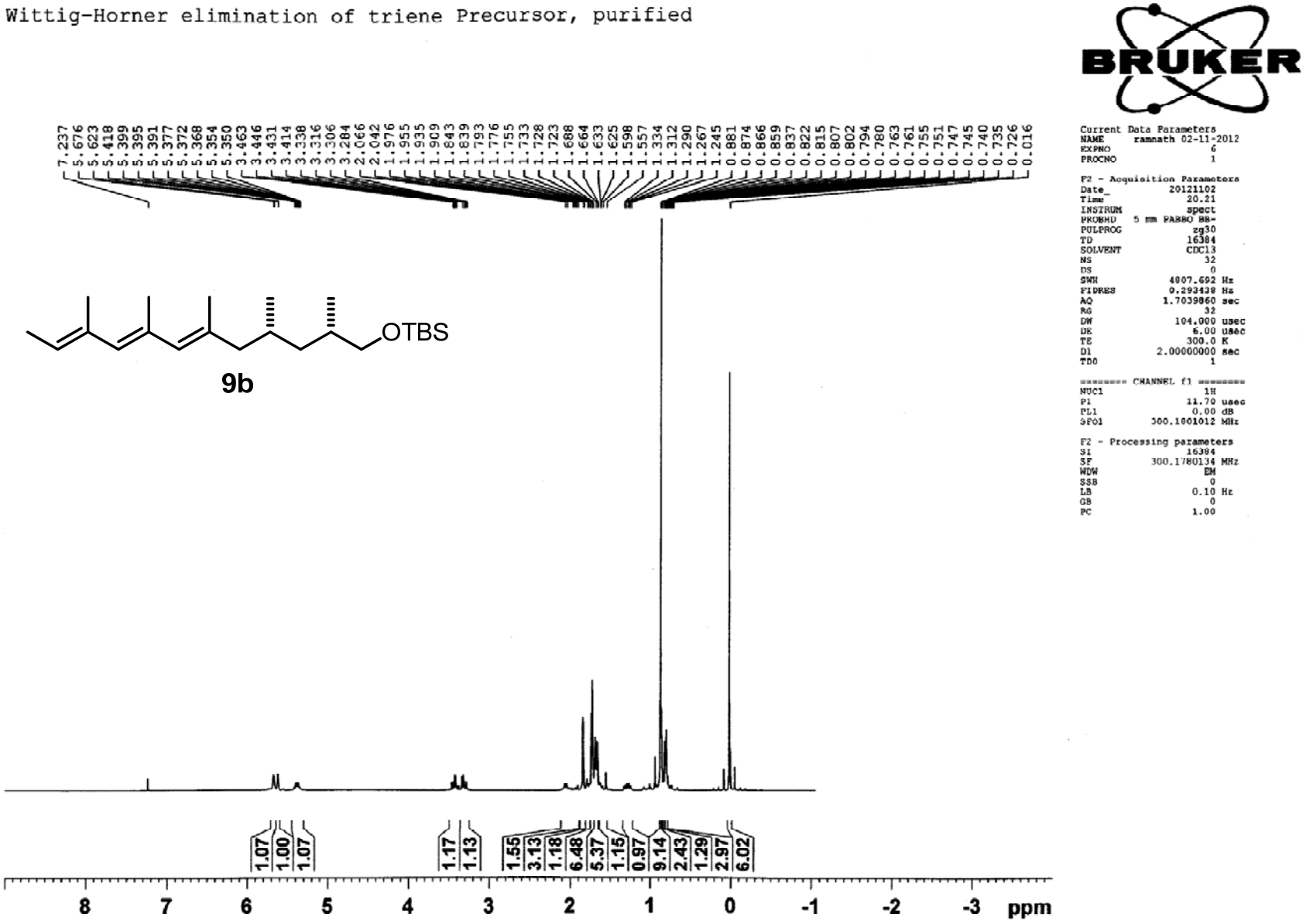

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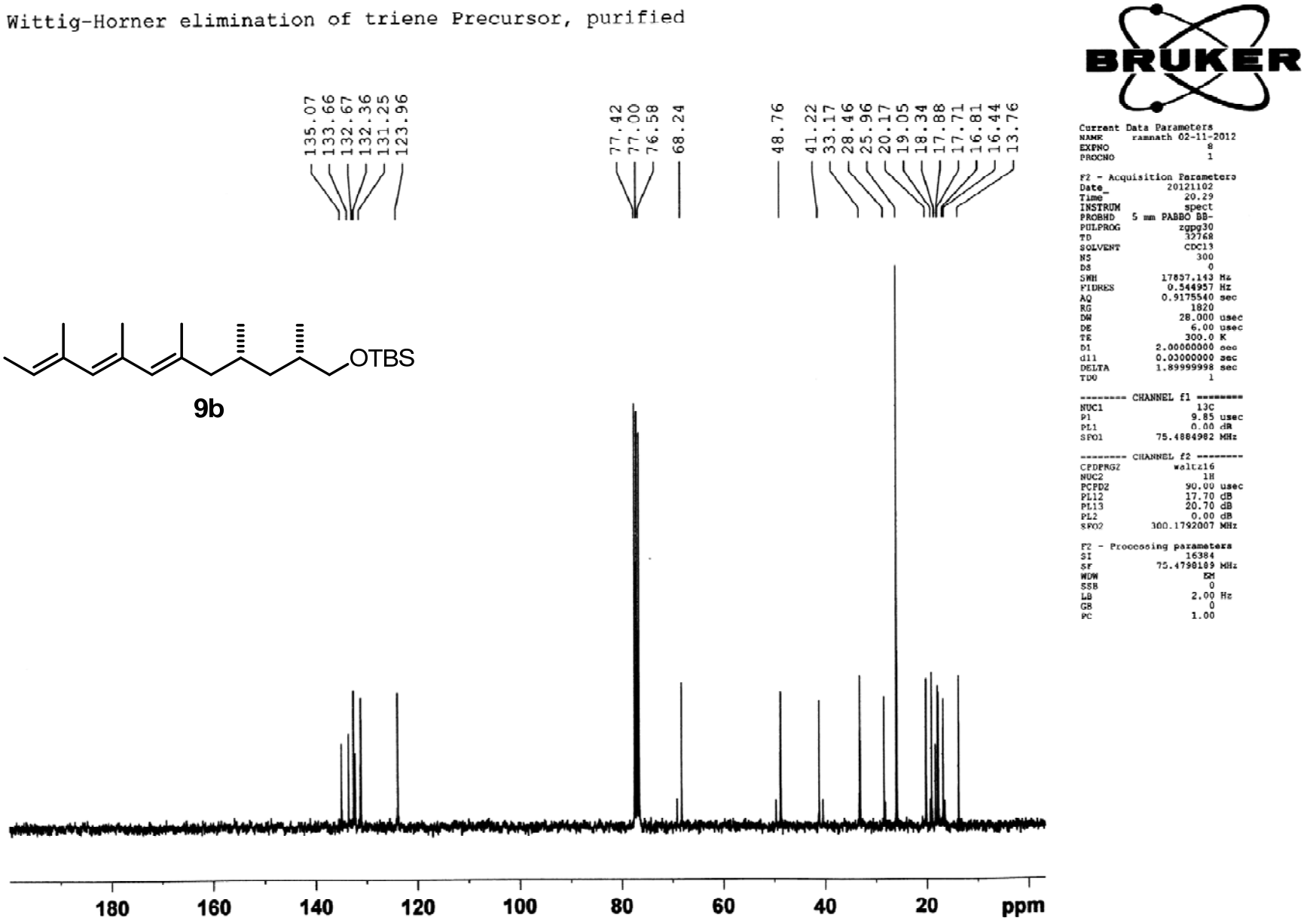

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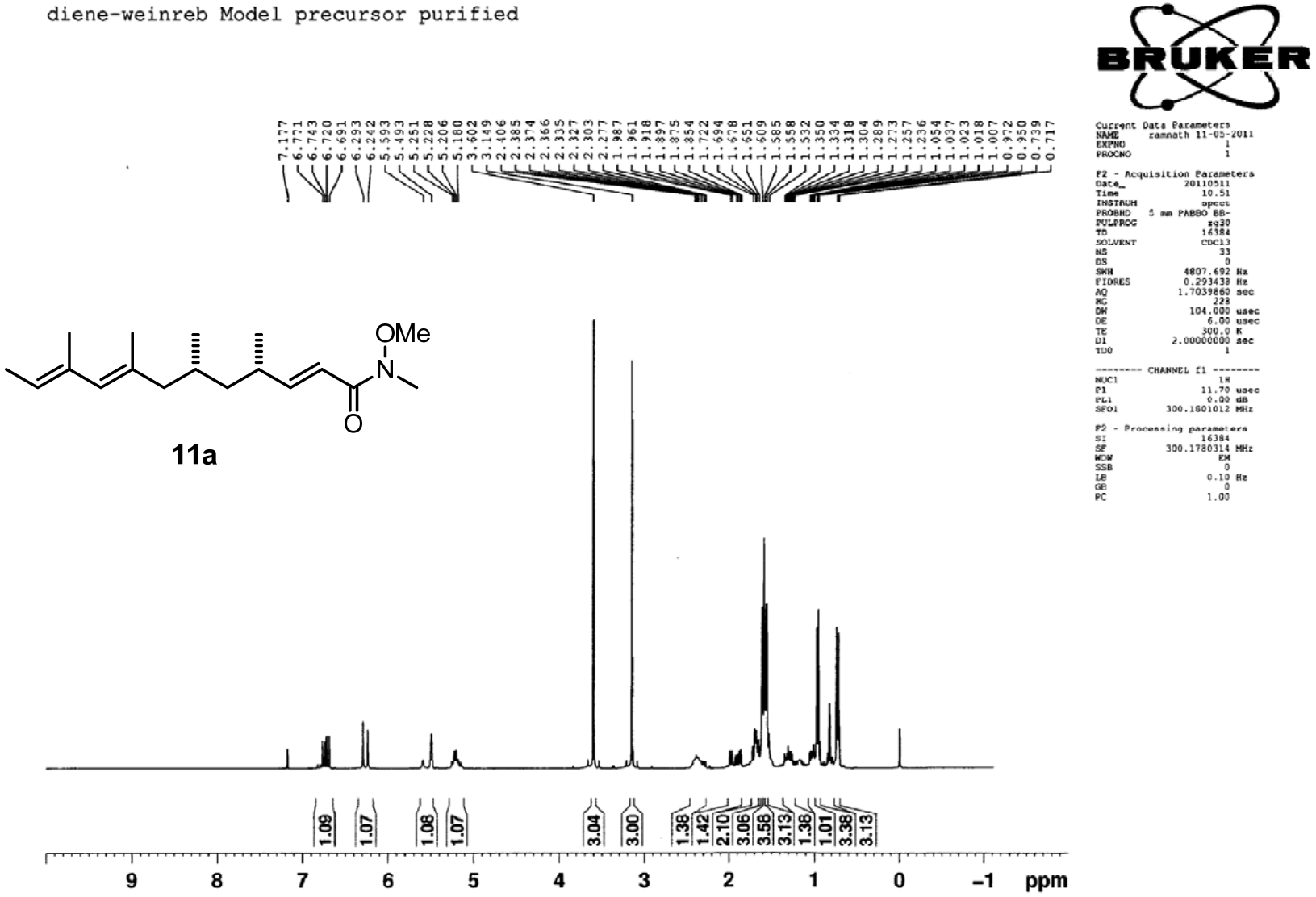

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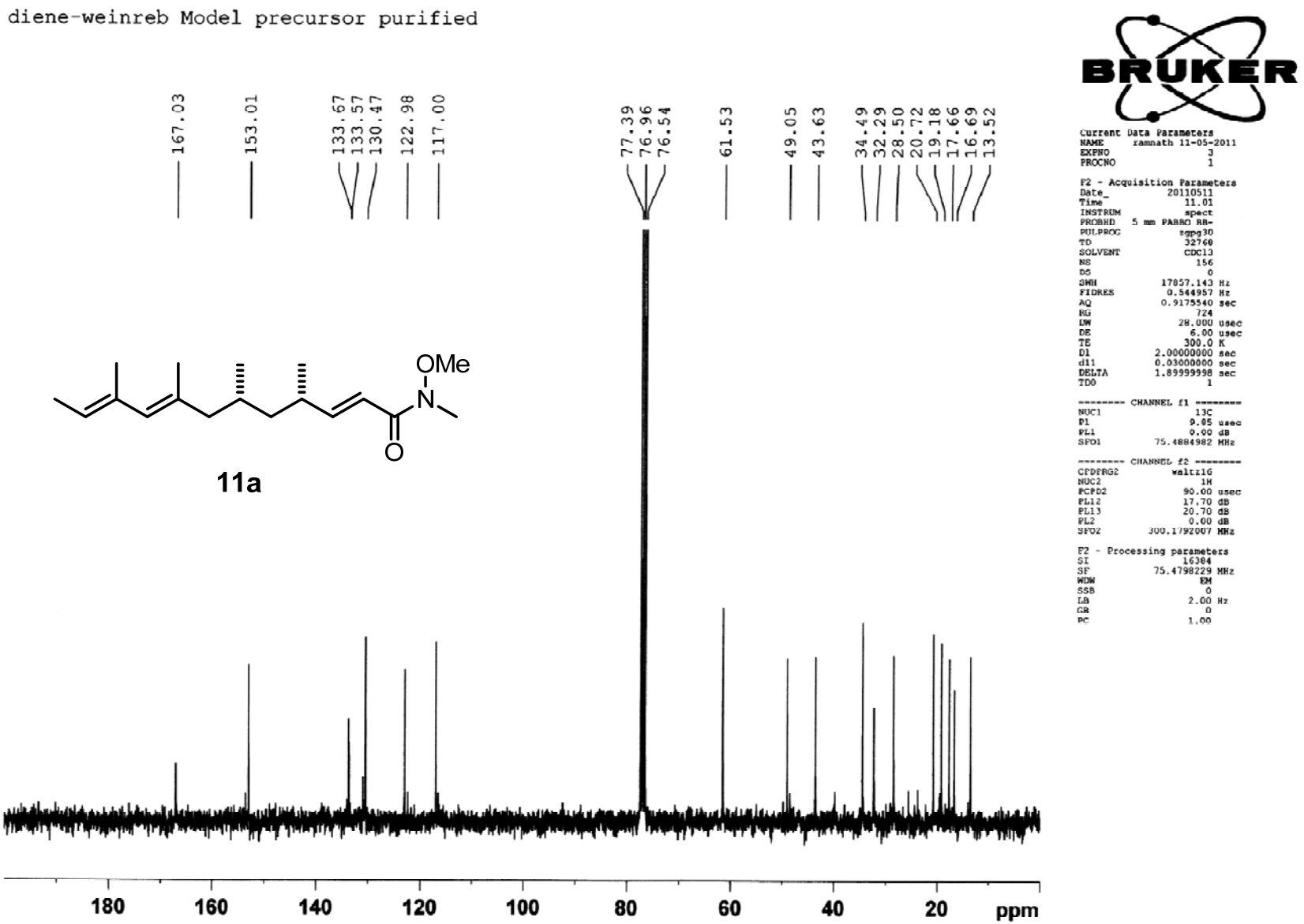

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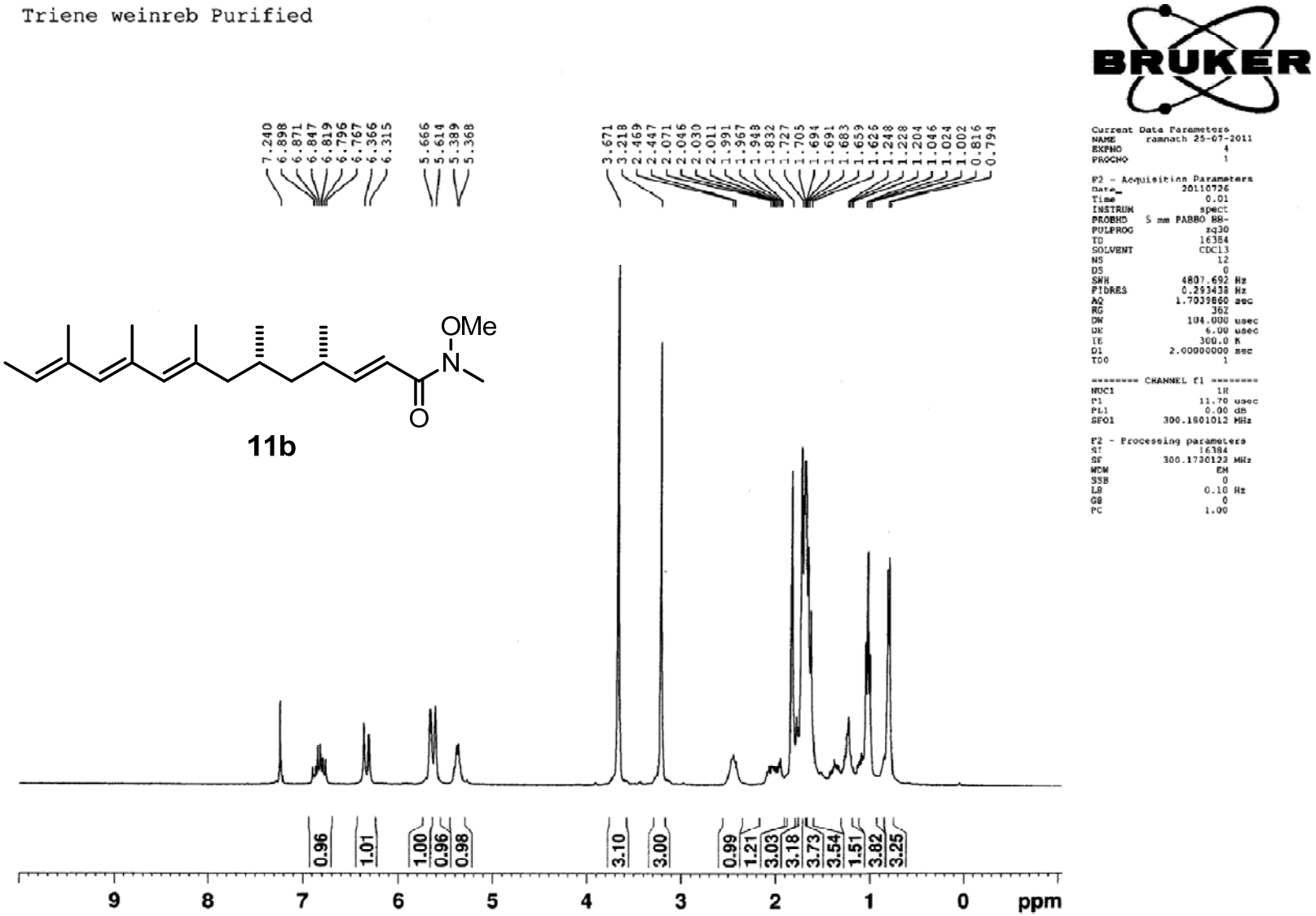

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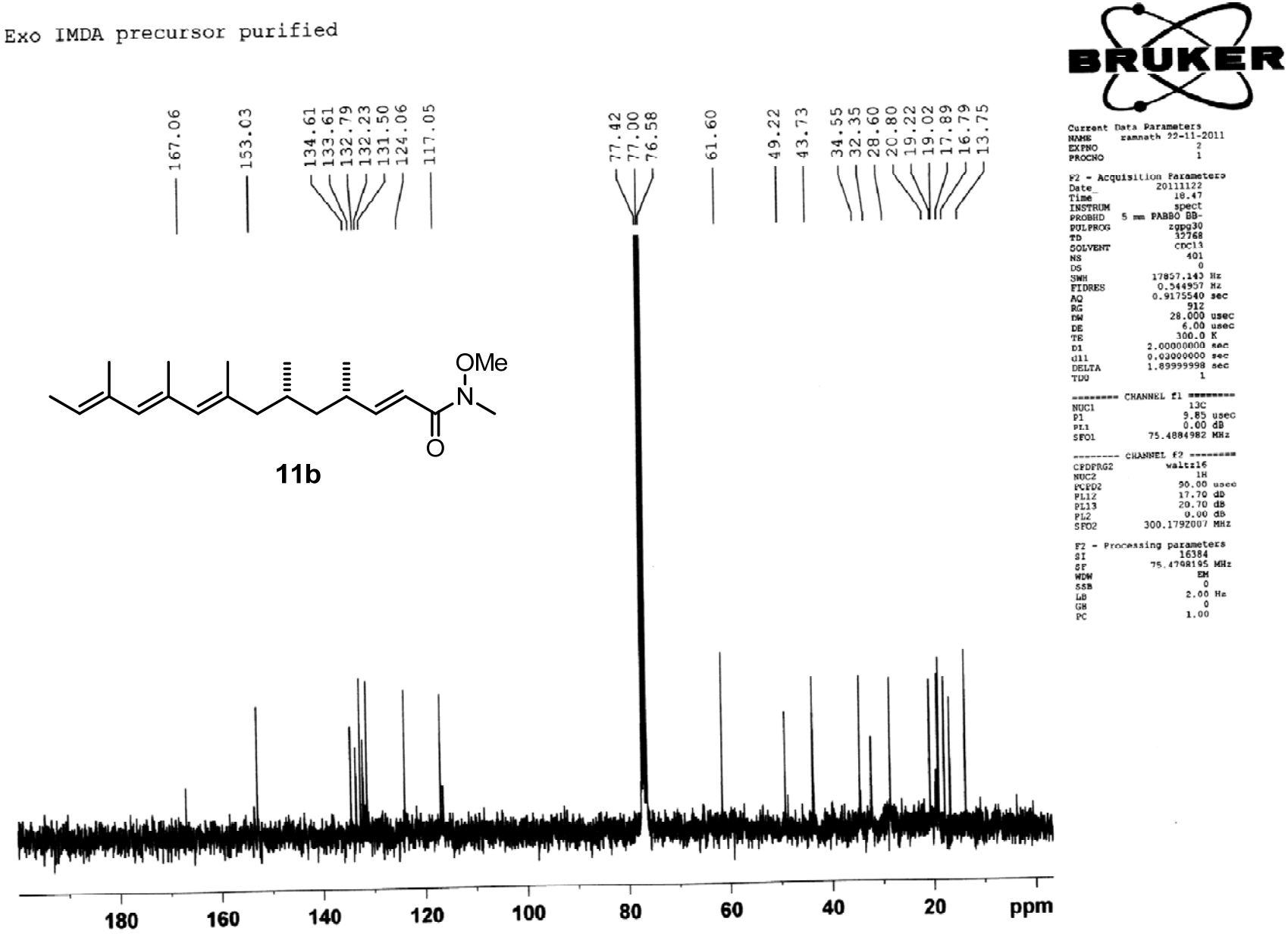

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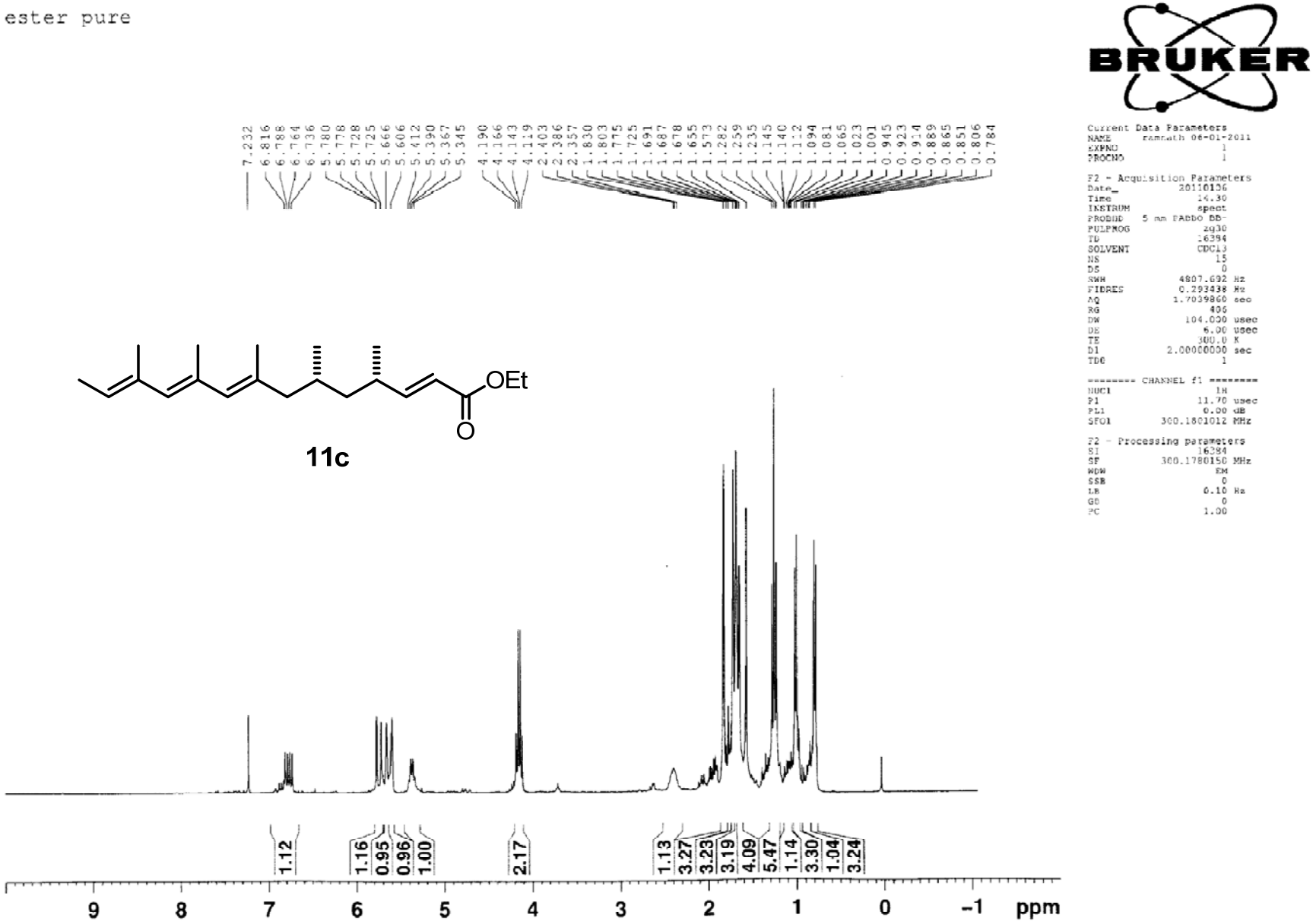

BRUKER

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PROCNO                     1

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TE                   300.0 K
D1              2.00000000 sec
TD0                       1

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PL1                    0.00 dB
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F2 - Processing parameters
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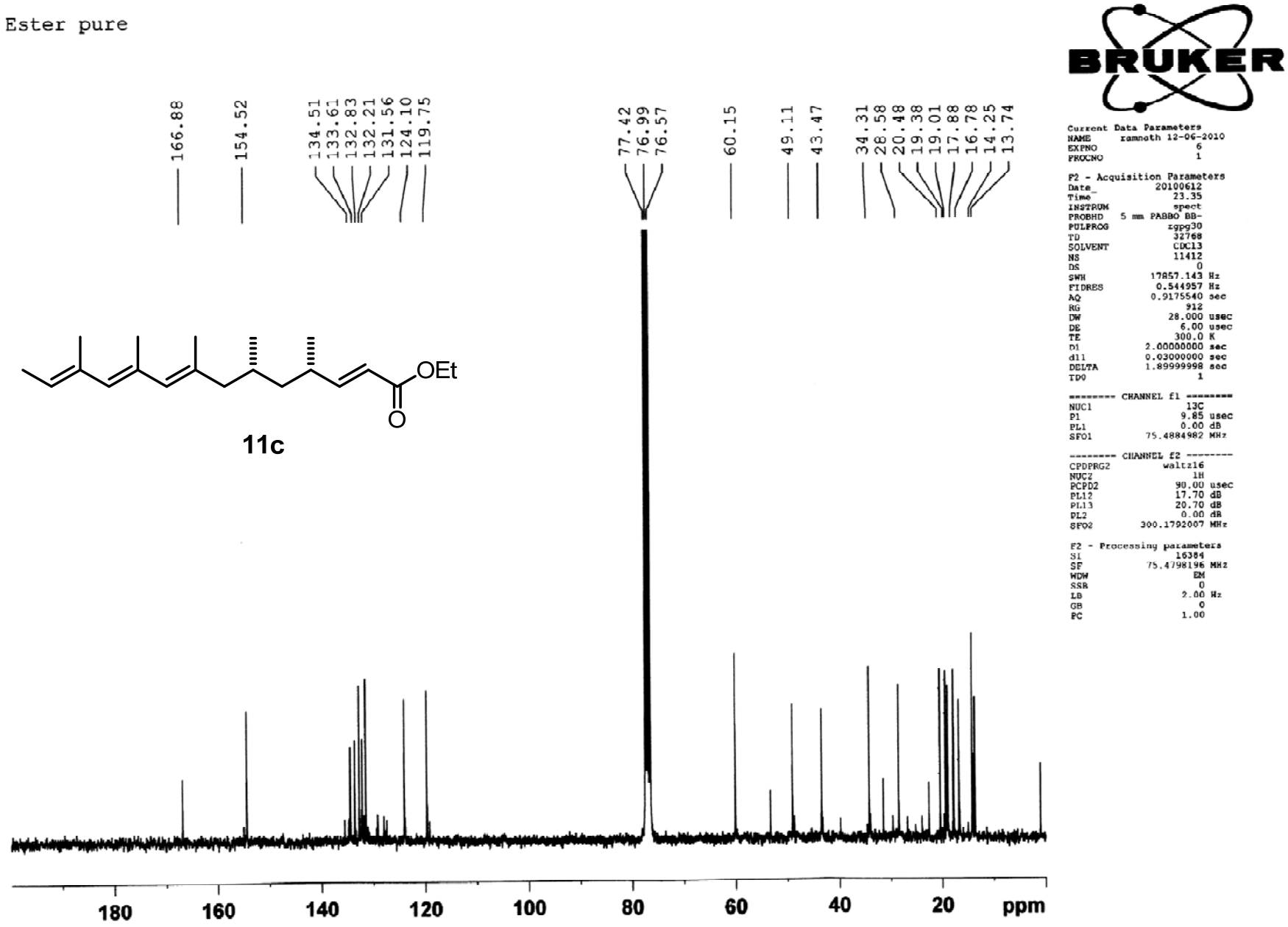

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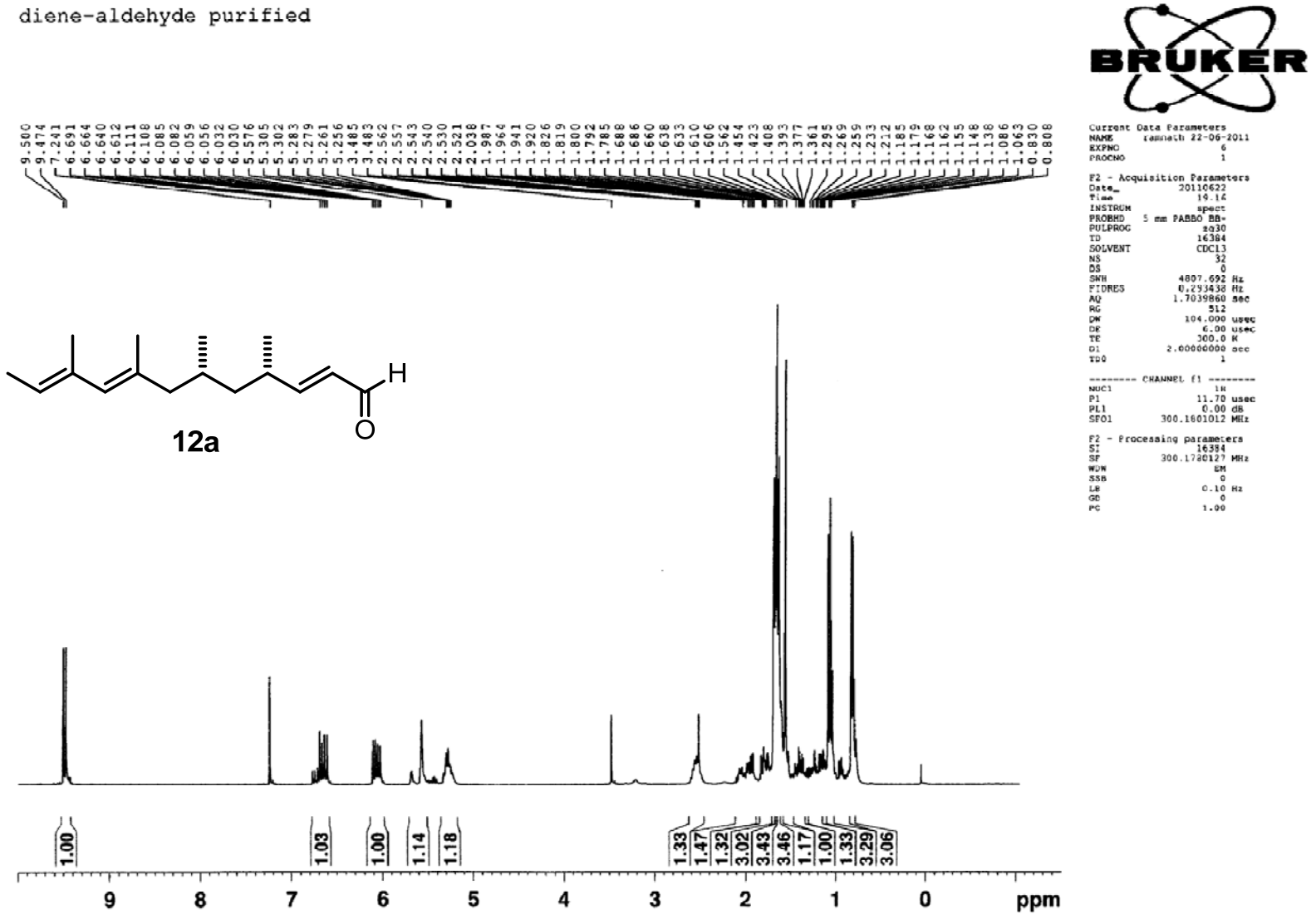

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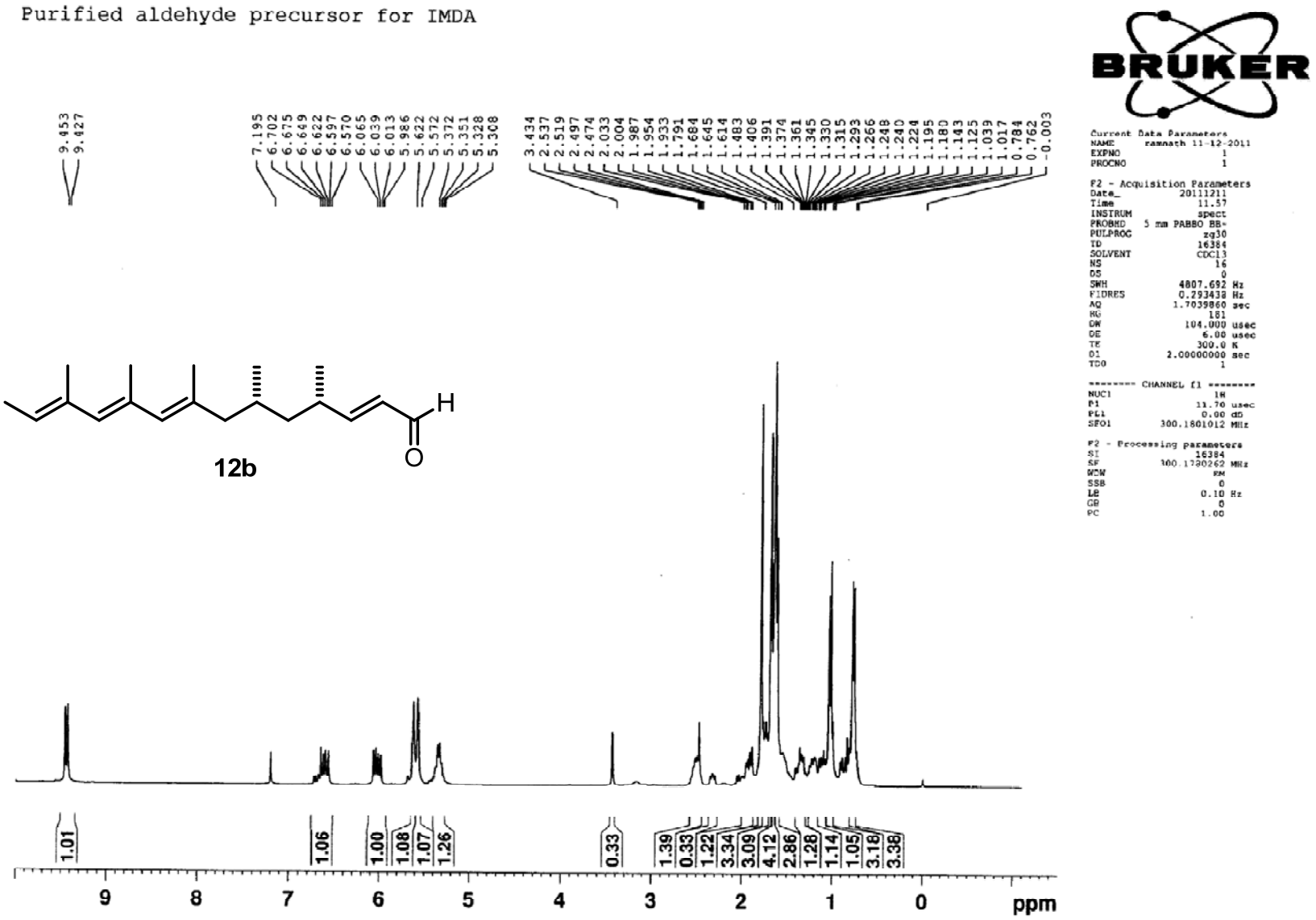

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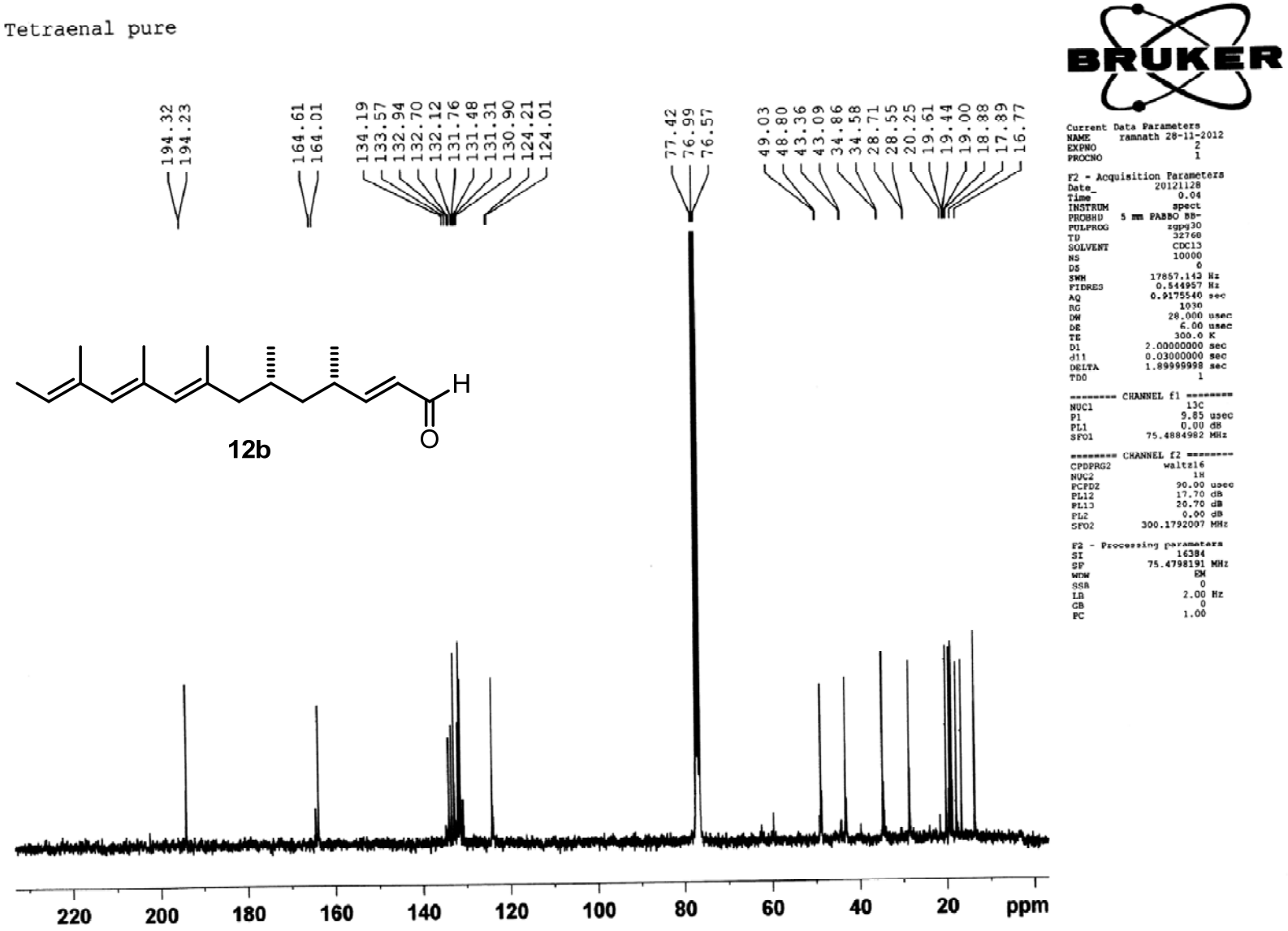

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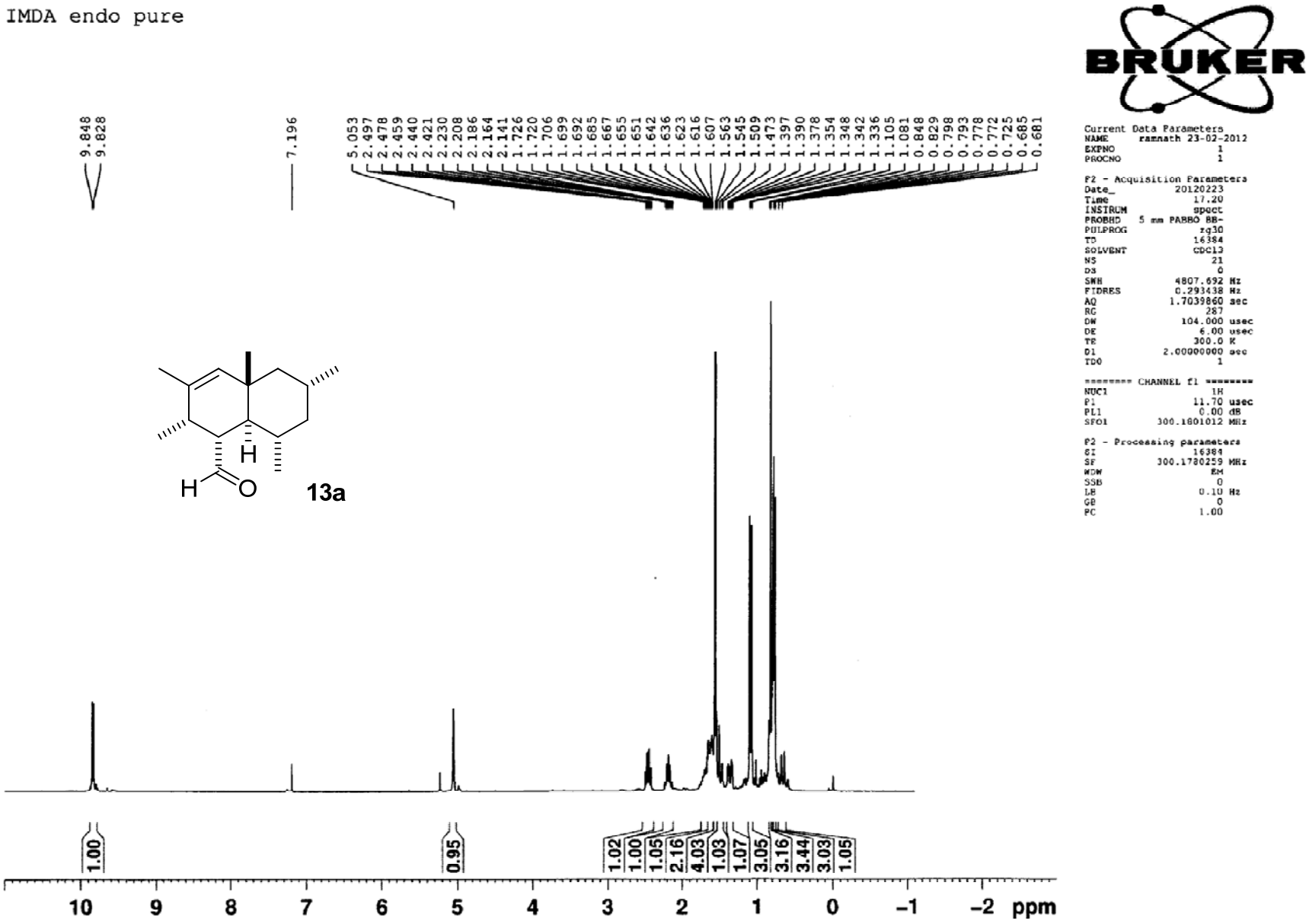

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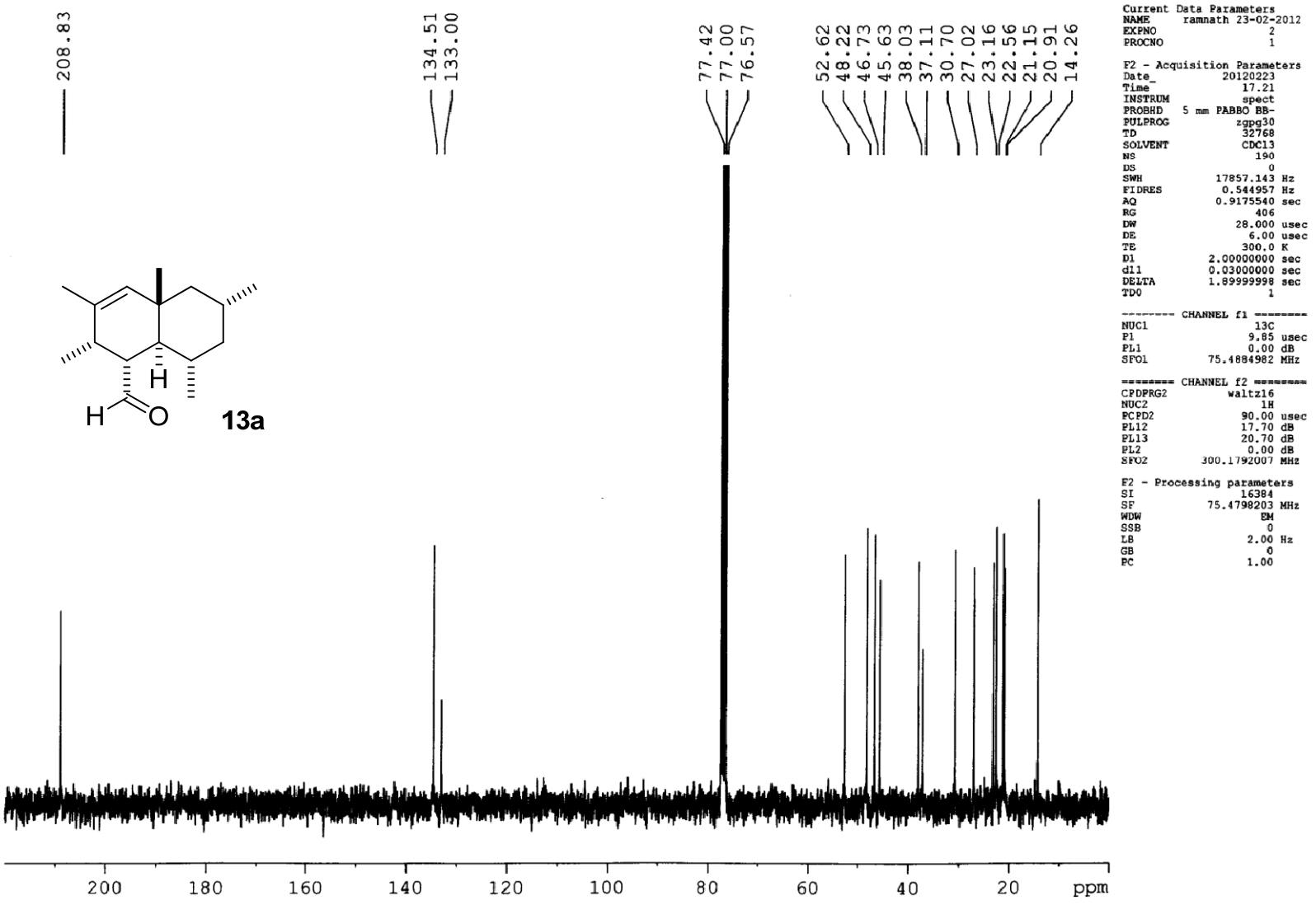
IMDA endo pure



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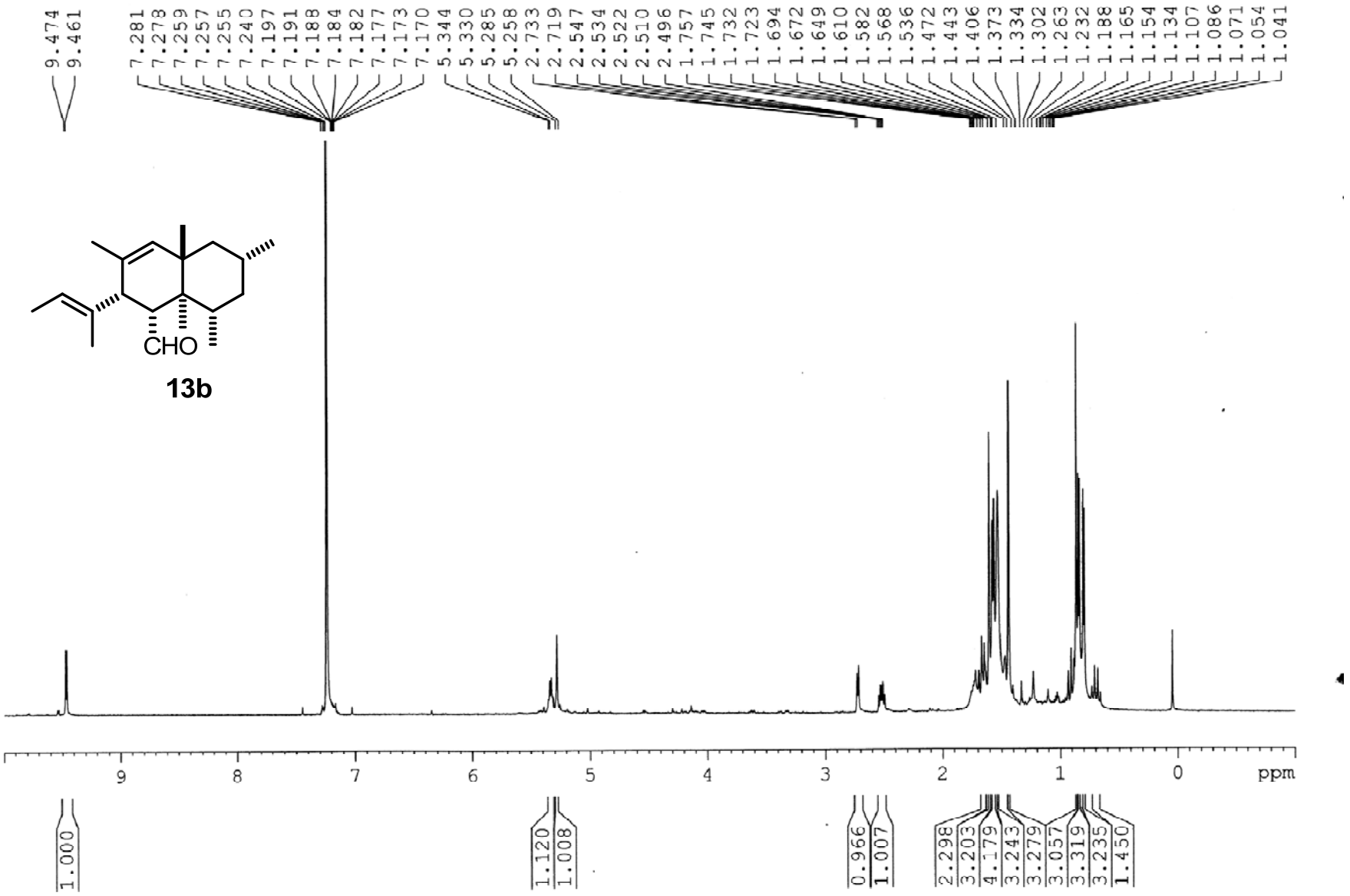

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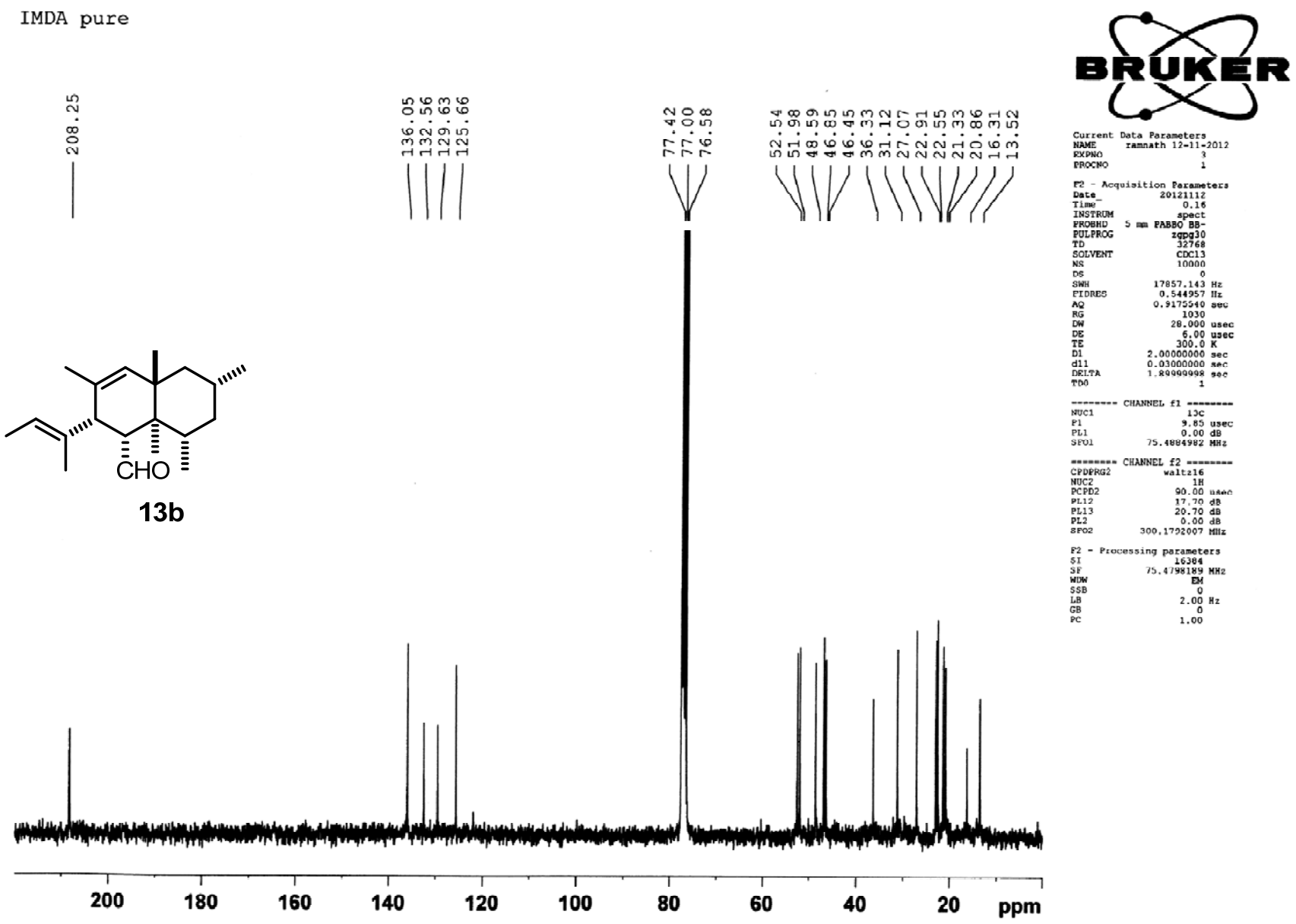

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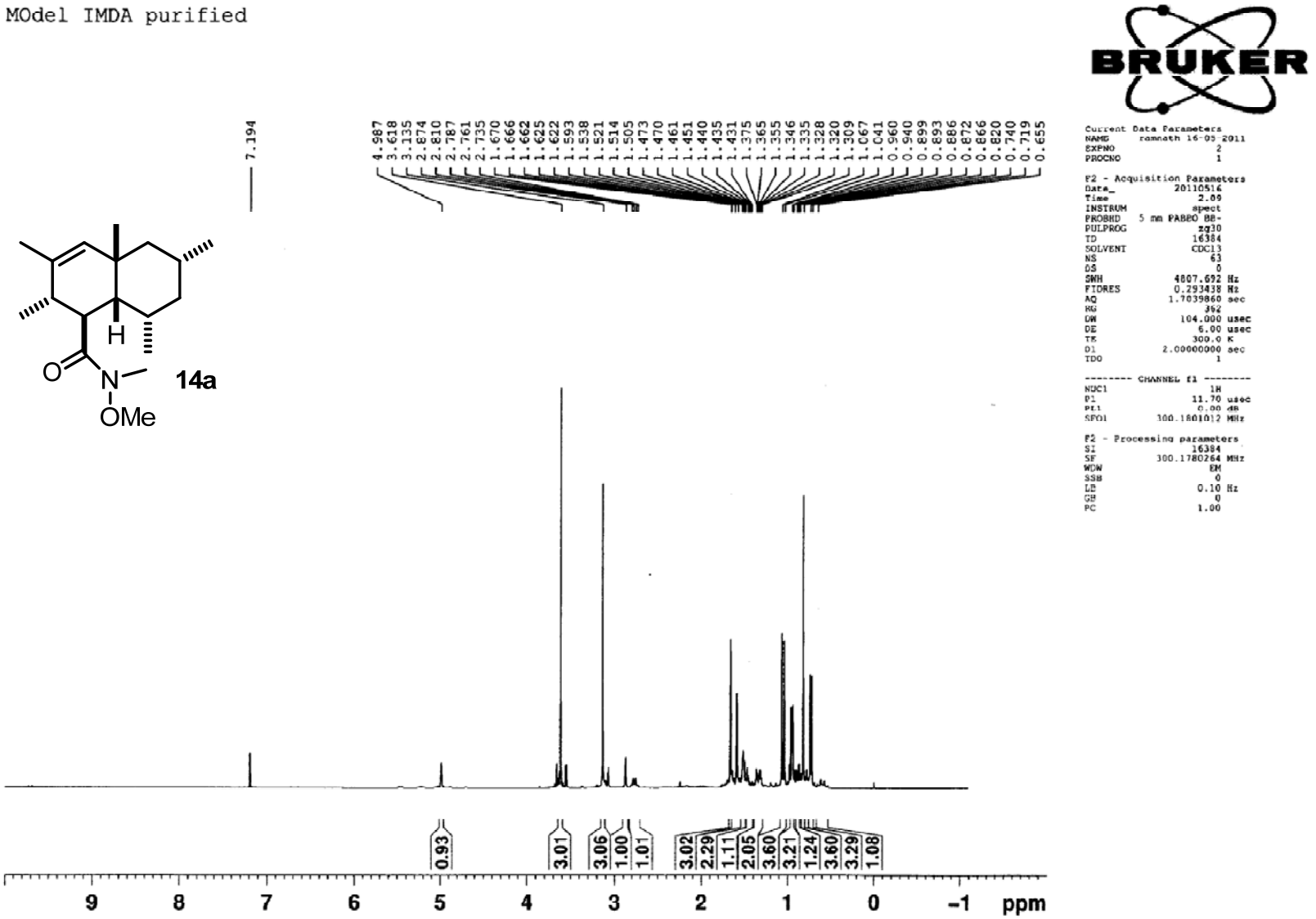

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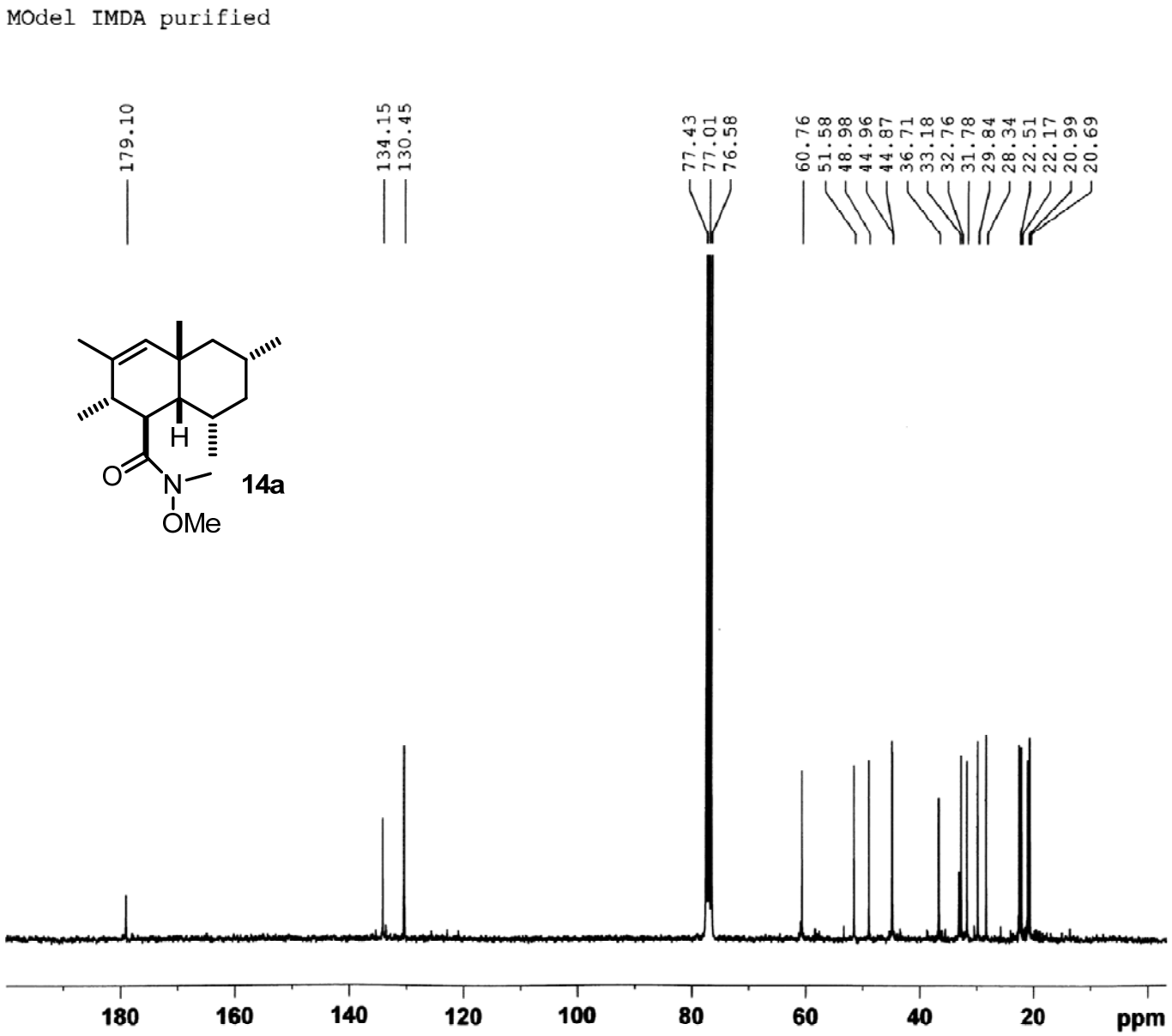


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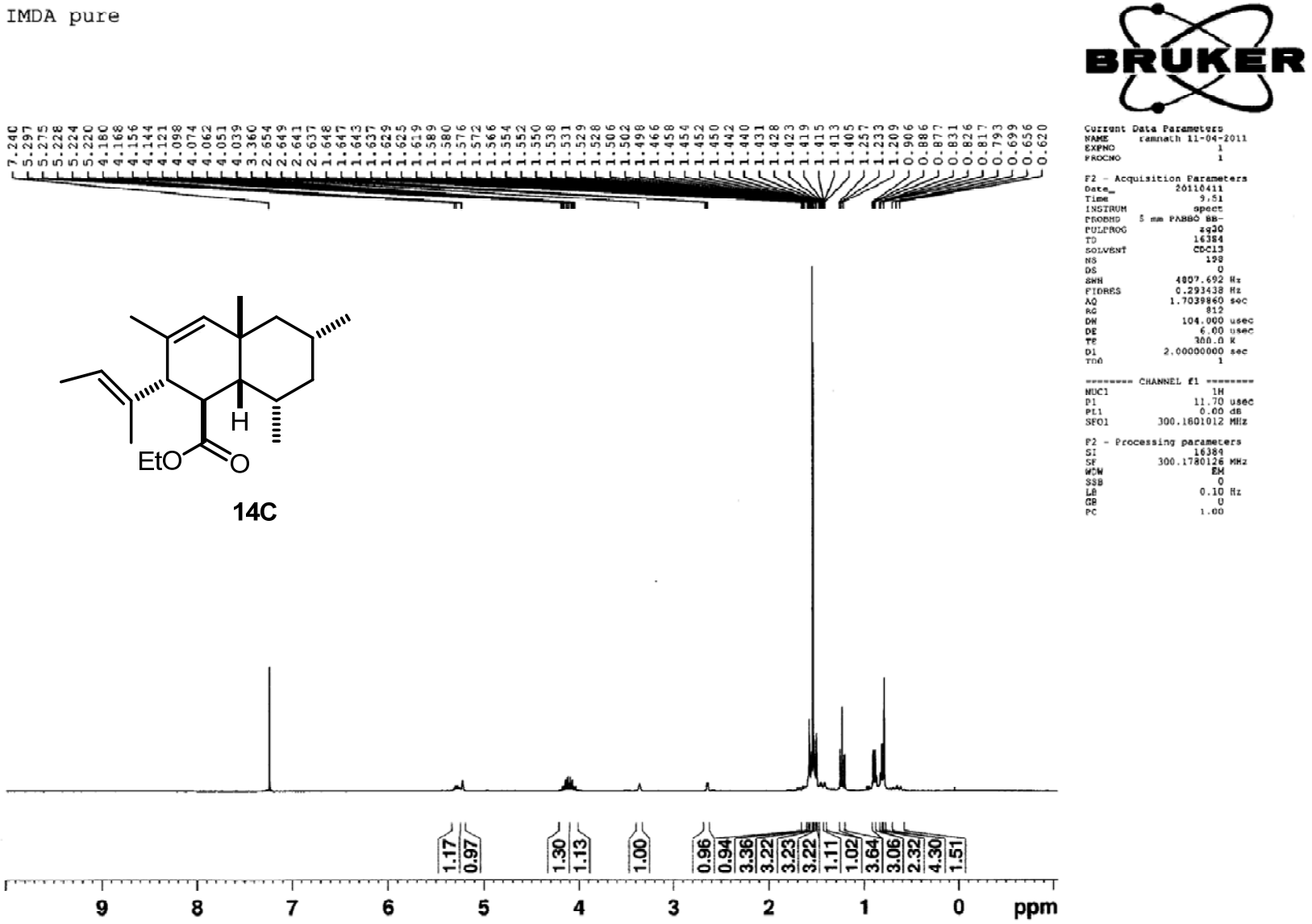

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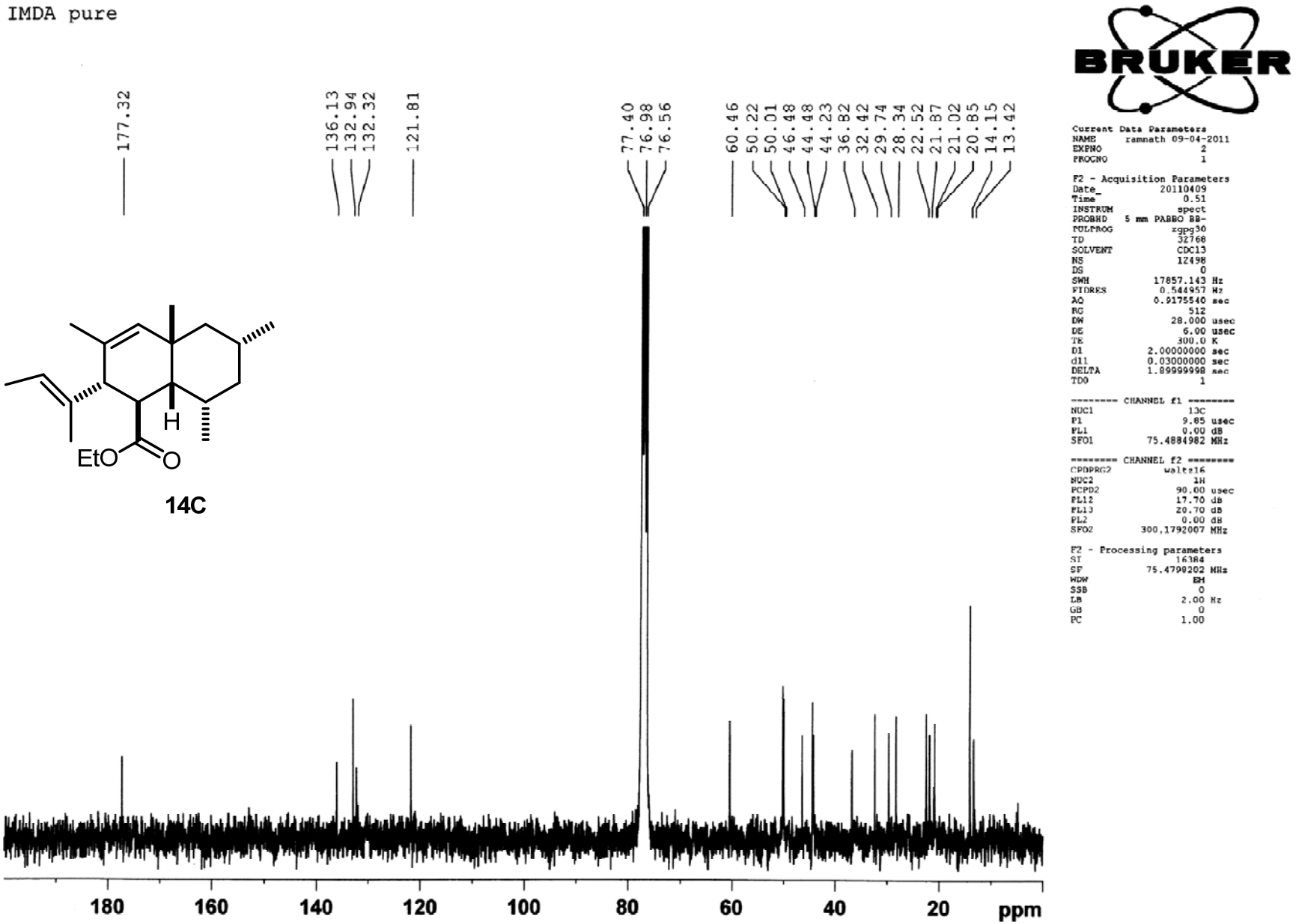

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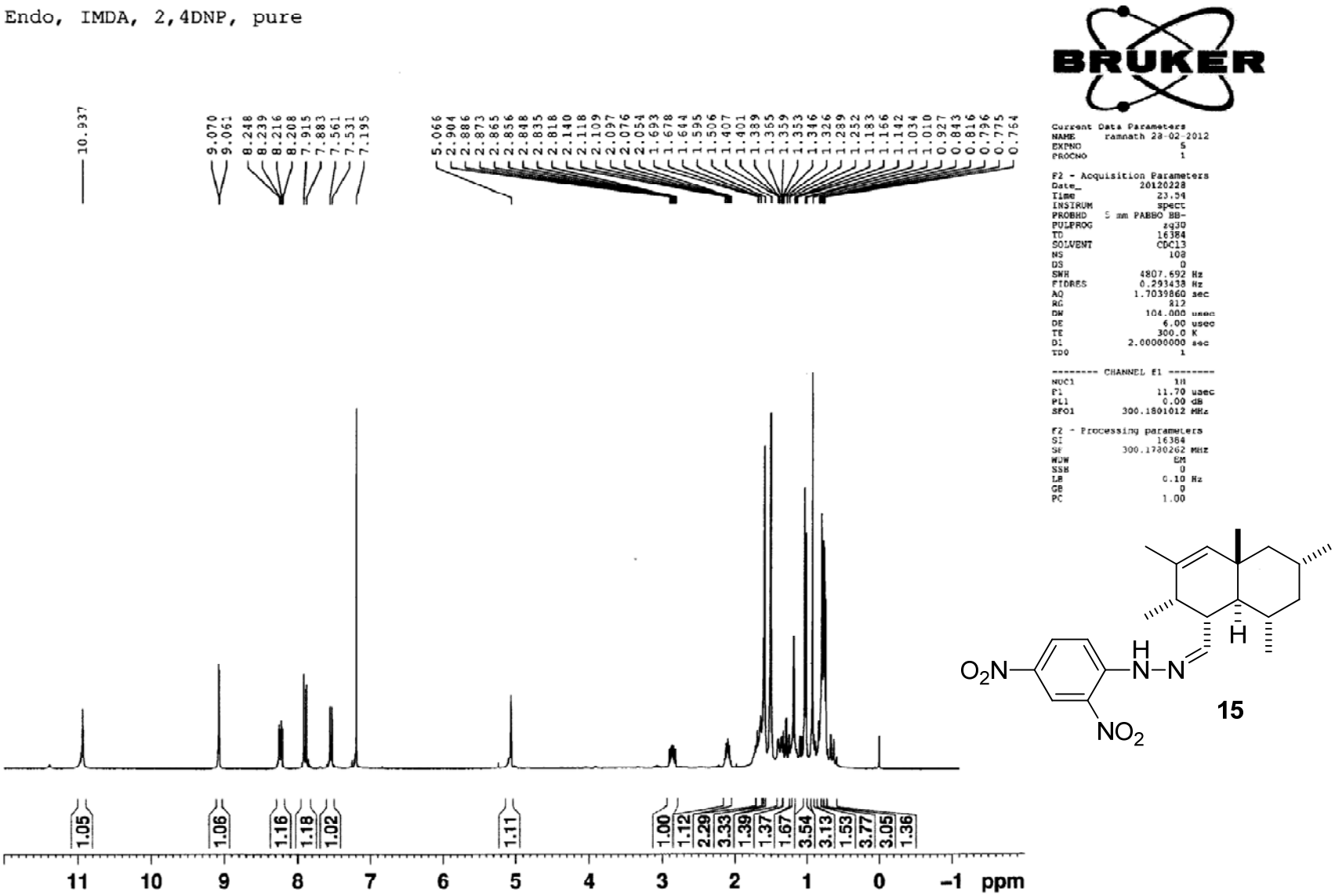

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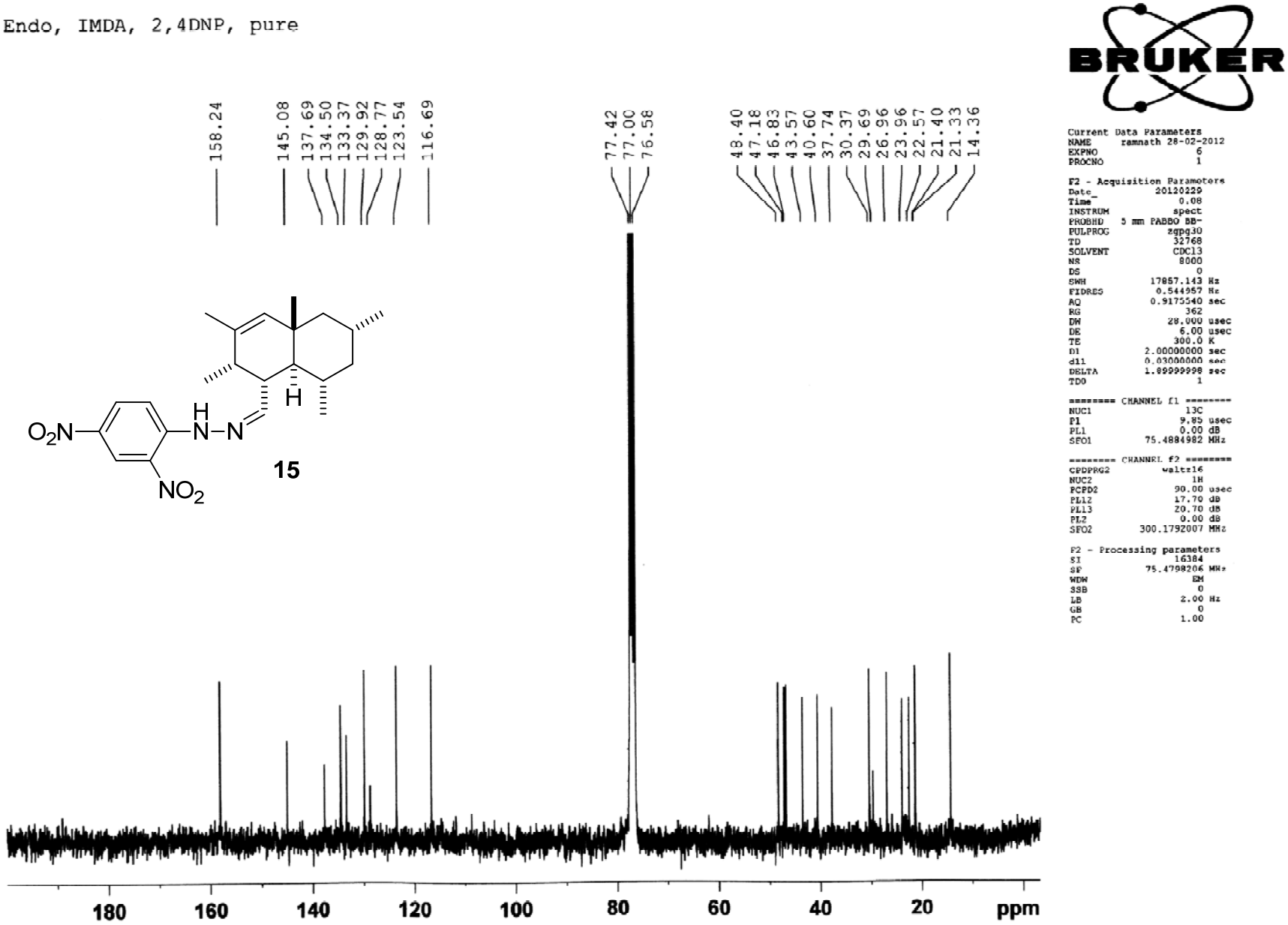

BRUKER

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PROCNO                       1

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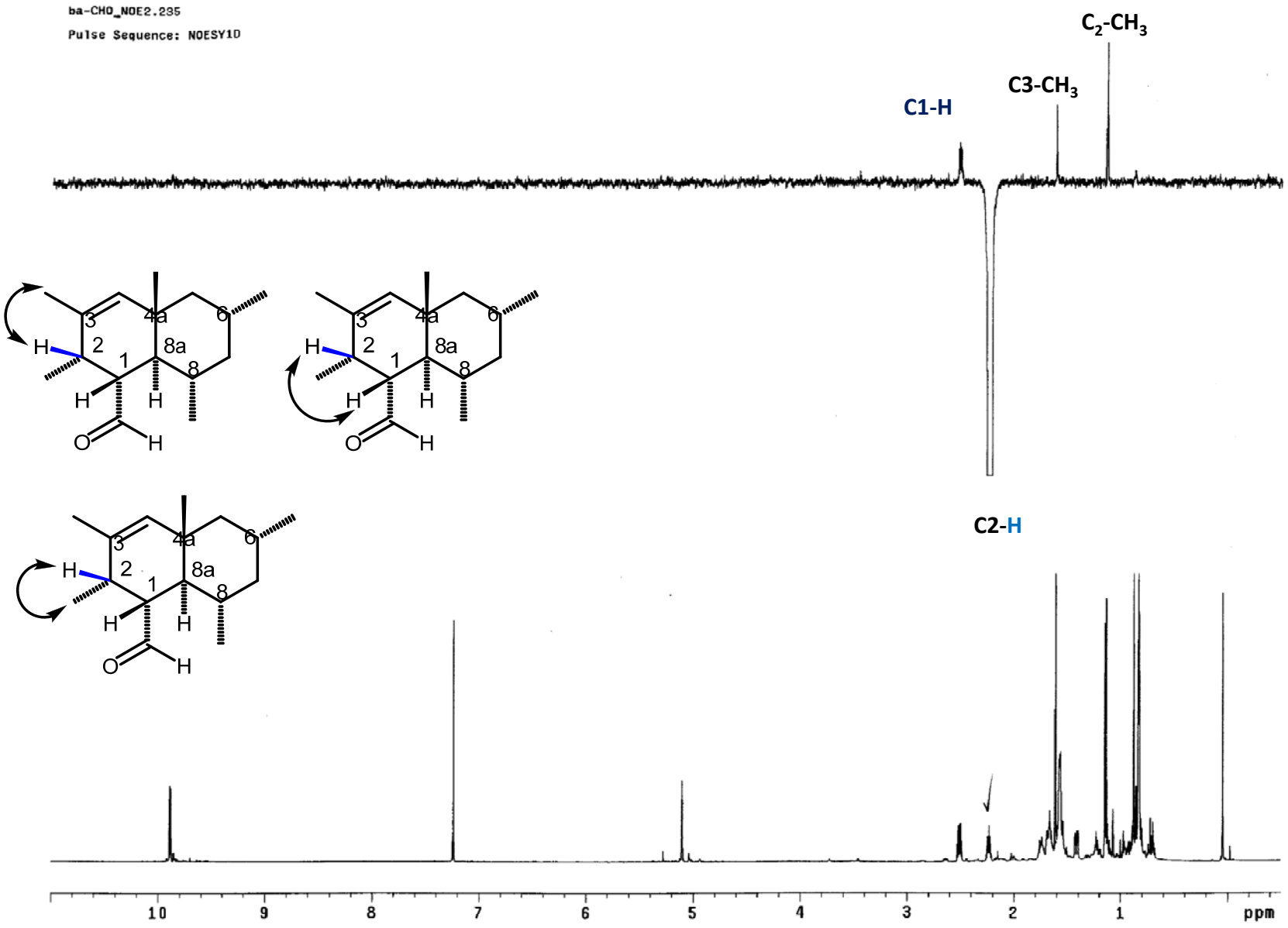
DS                          0
SWH                17857.143 Hz
FIDRES             0.544957 Hz
AQ                 0.9175540 sec
RG                        362
DW                   28.000 usec
DE                    6.00 usec
TE                    300.0 K
D1                2.00000000 sec
d11               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

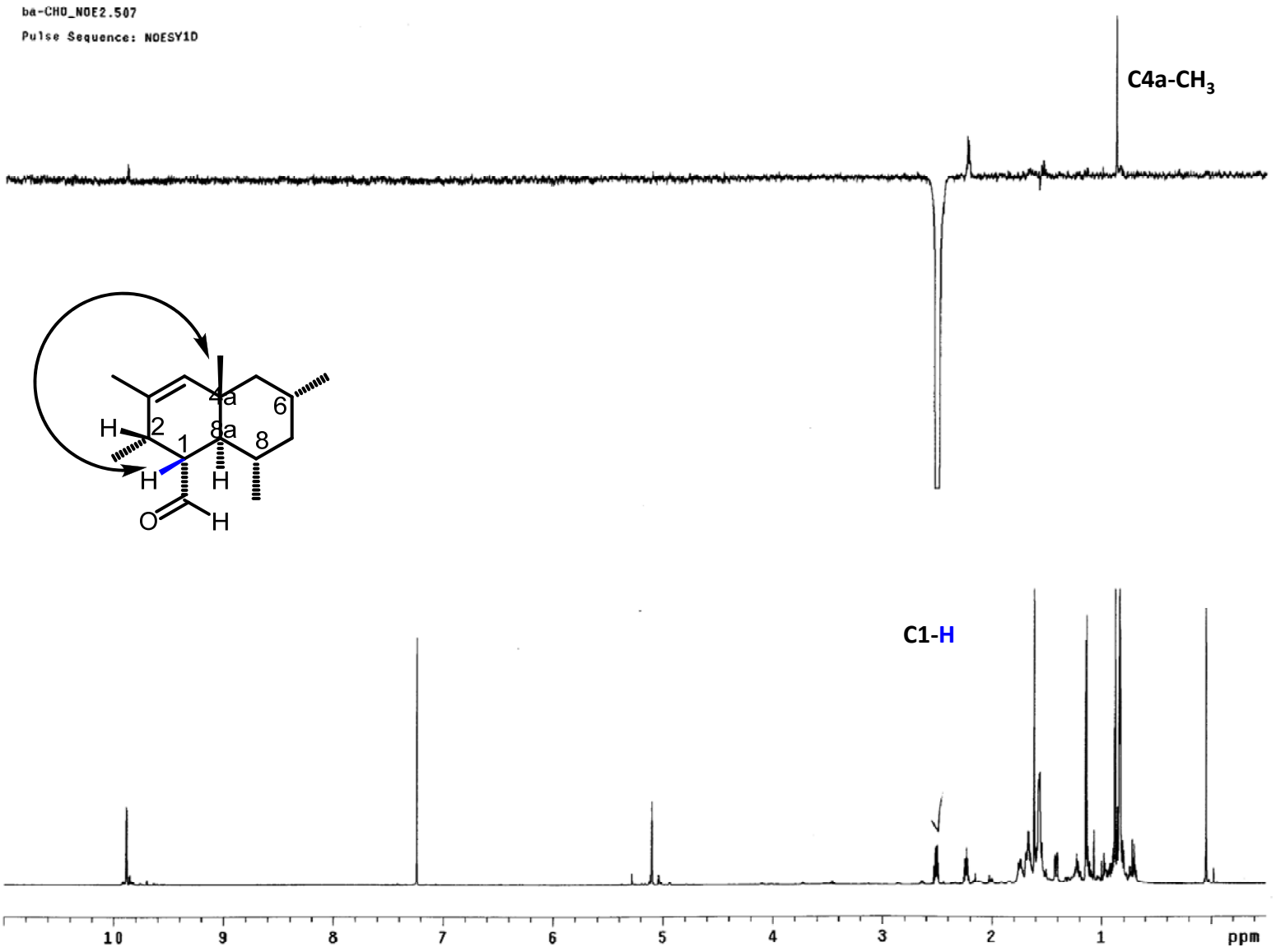
======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 90.00 usec
PL12                  17.70 dB
PL13                  20.70 dB
PL2                    0.00 dB
SFO2              300.1792007 MHz

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

^{13}C NMR (75 MHz, $CDCl_3$) of Compound **15**



NOE of 13a



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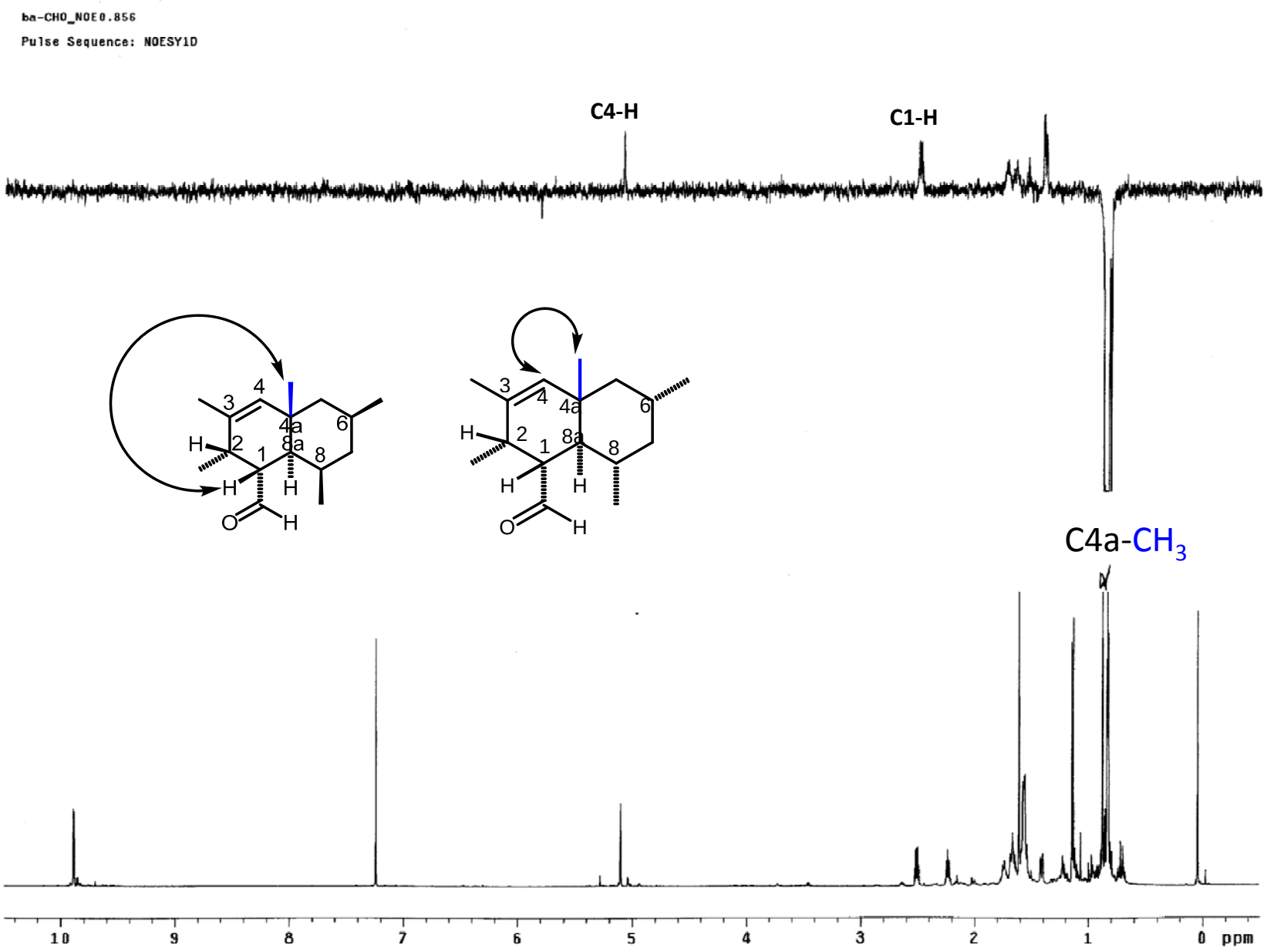

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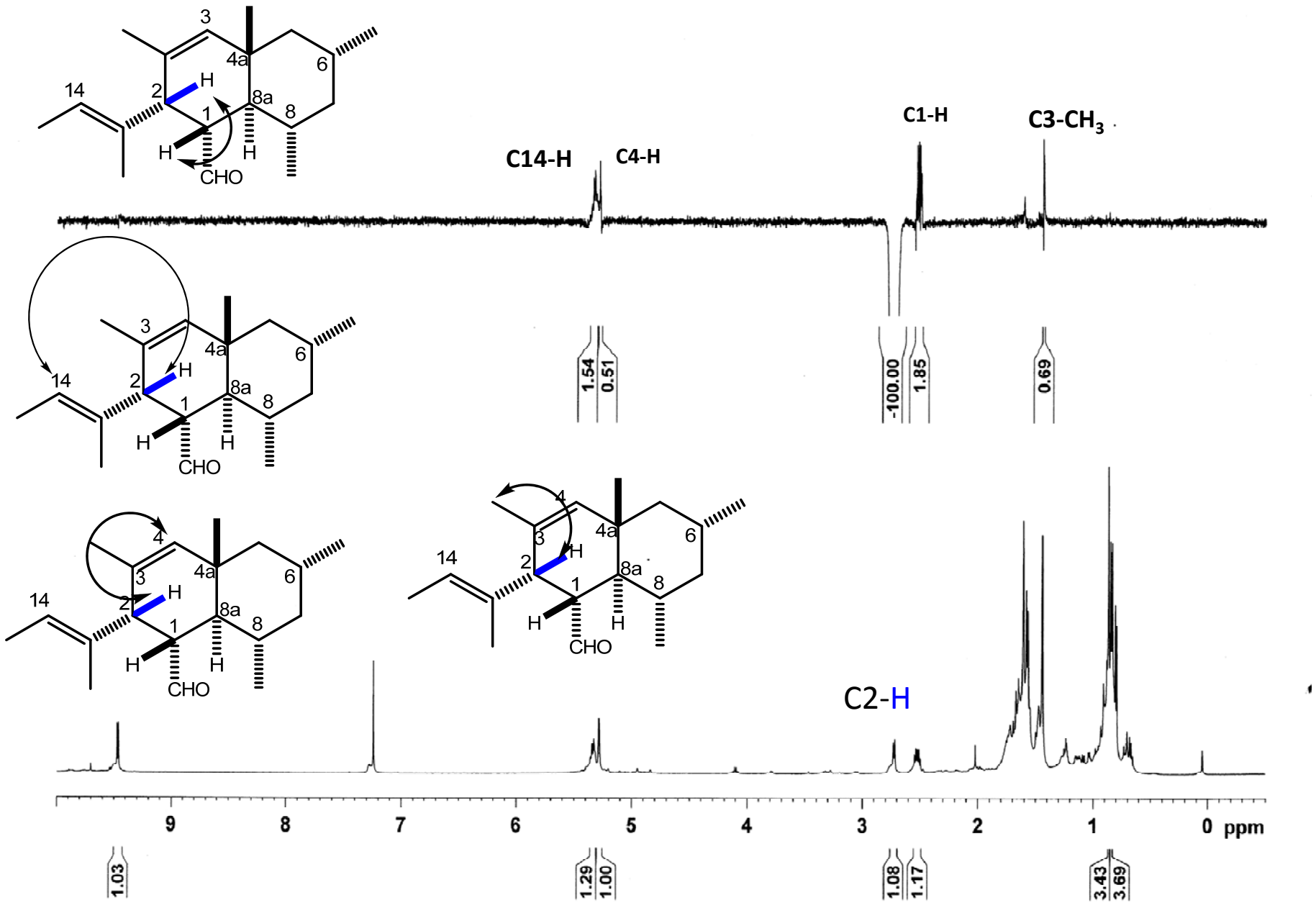

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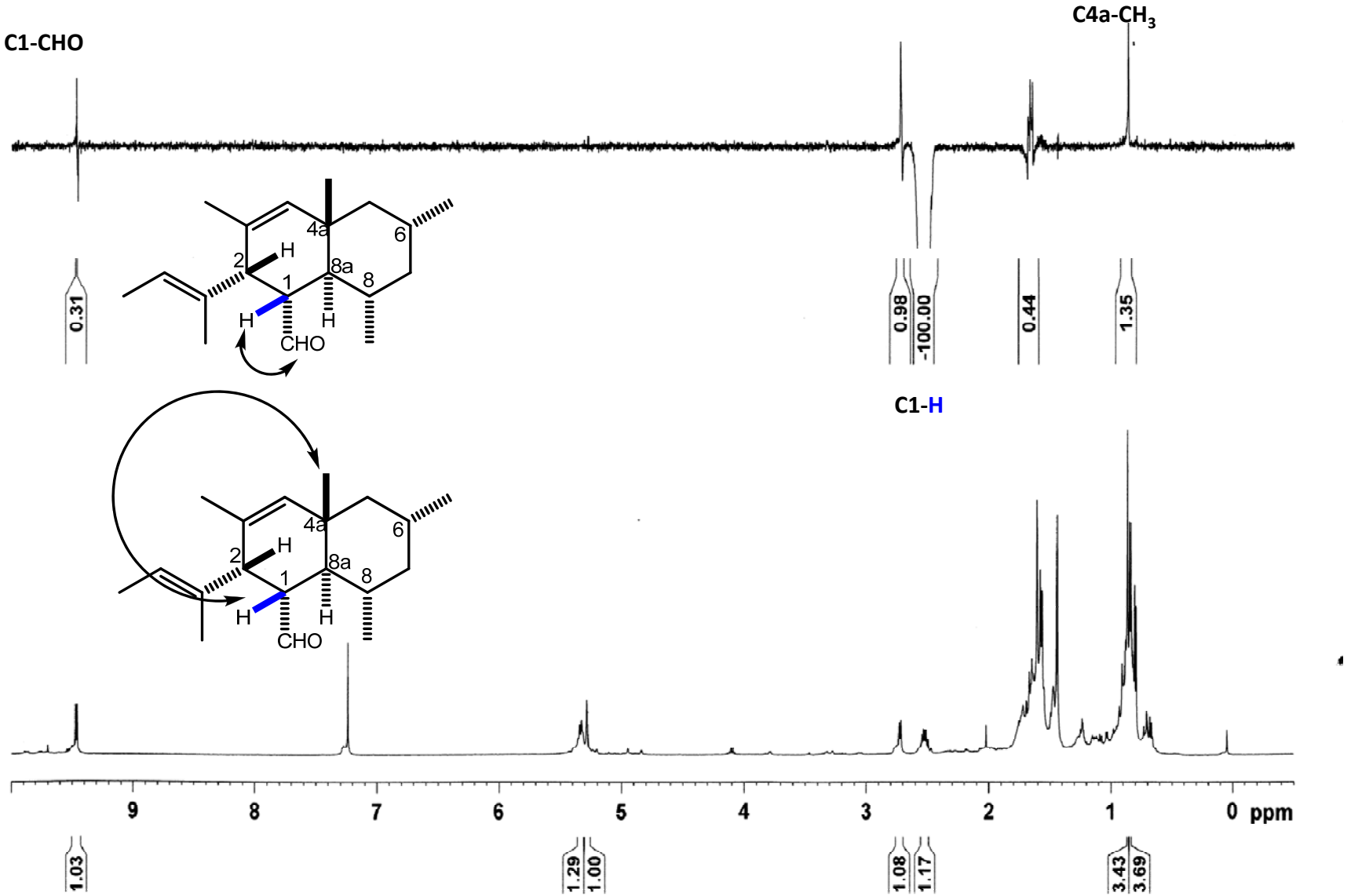

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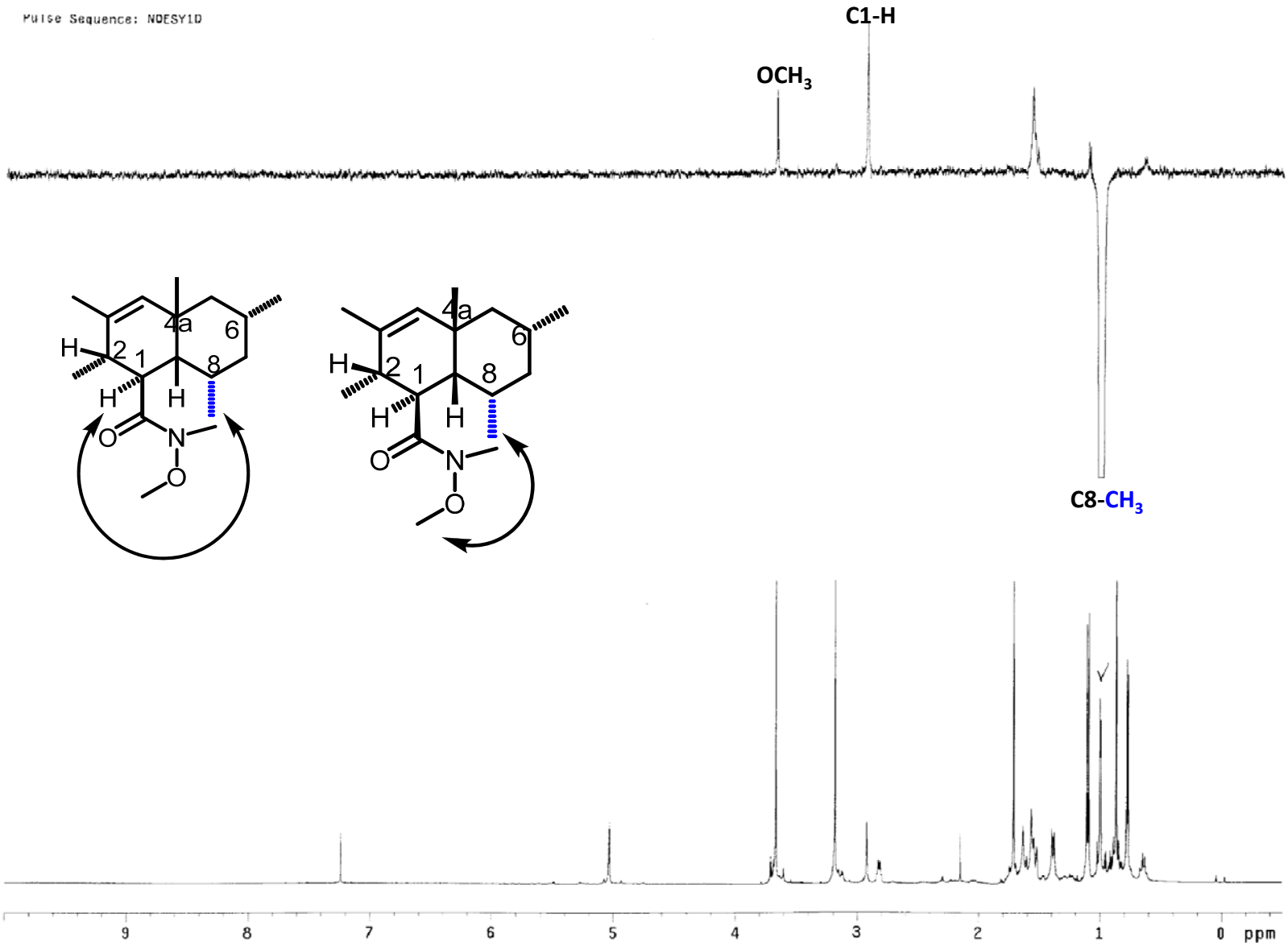

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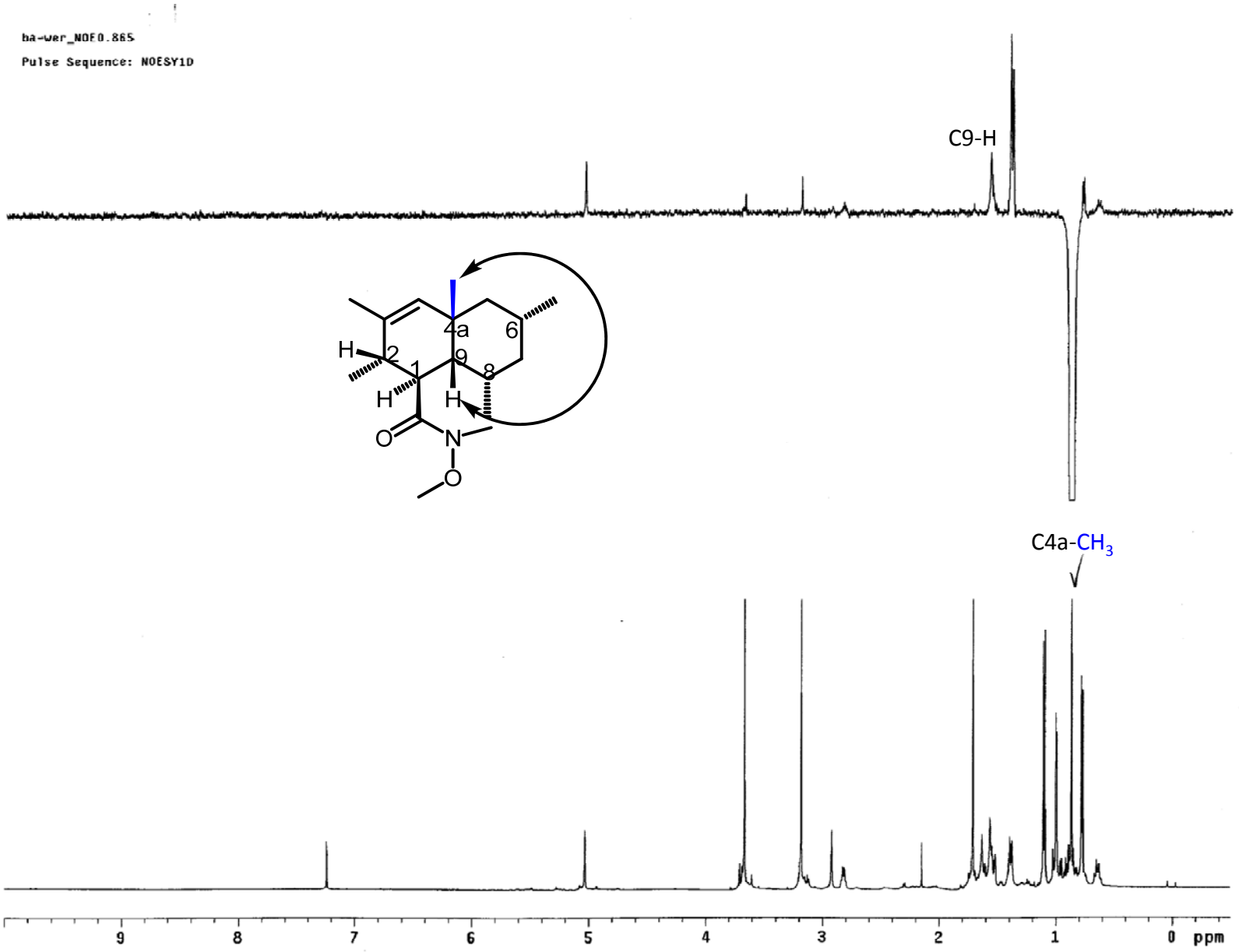

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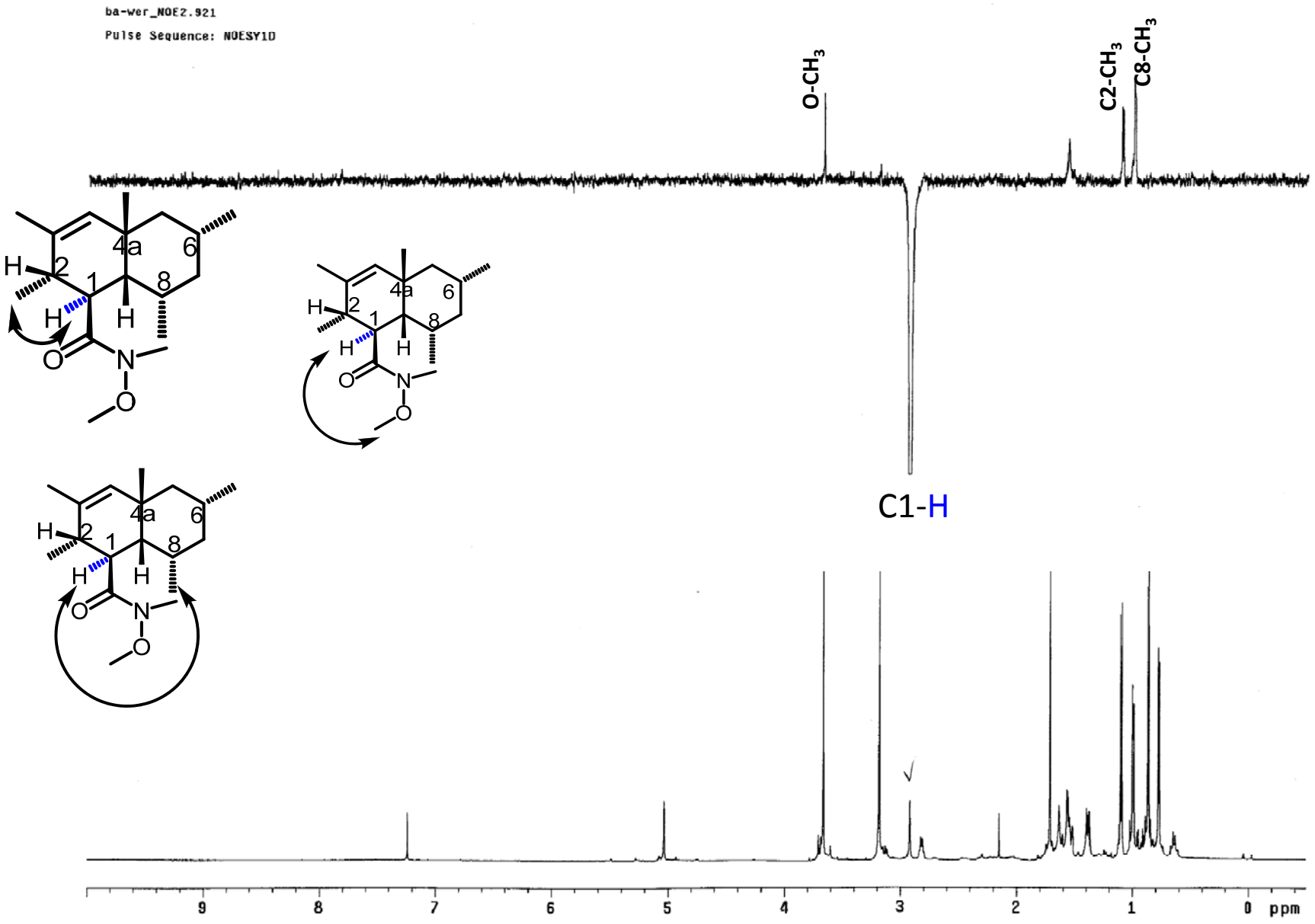

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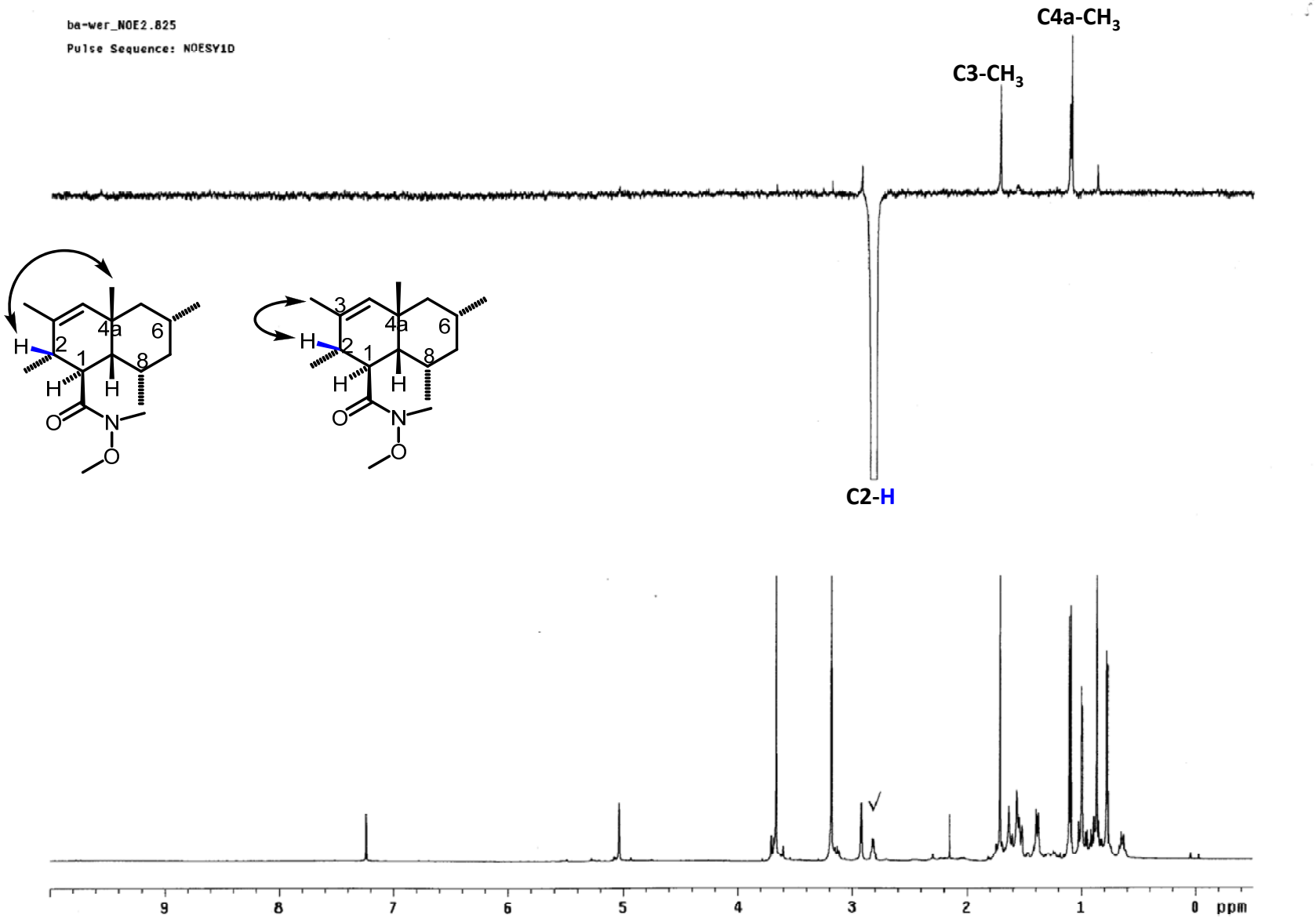

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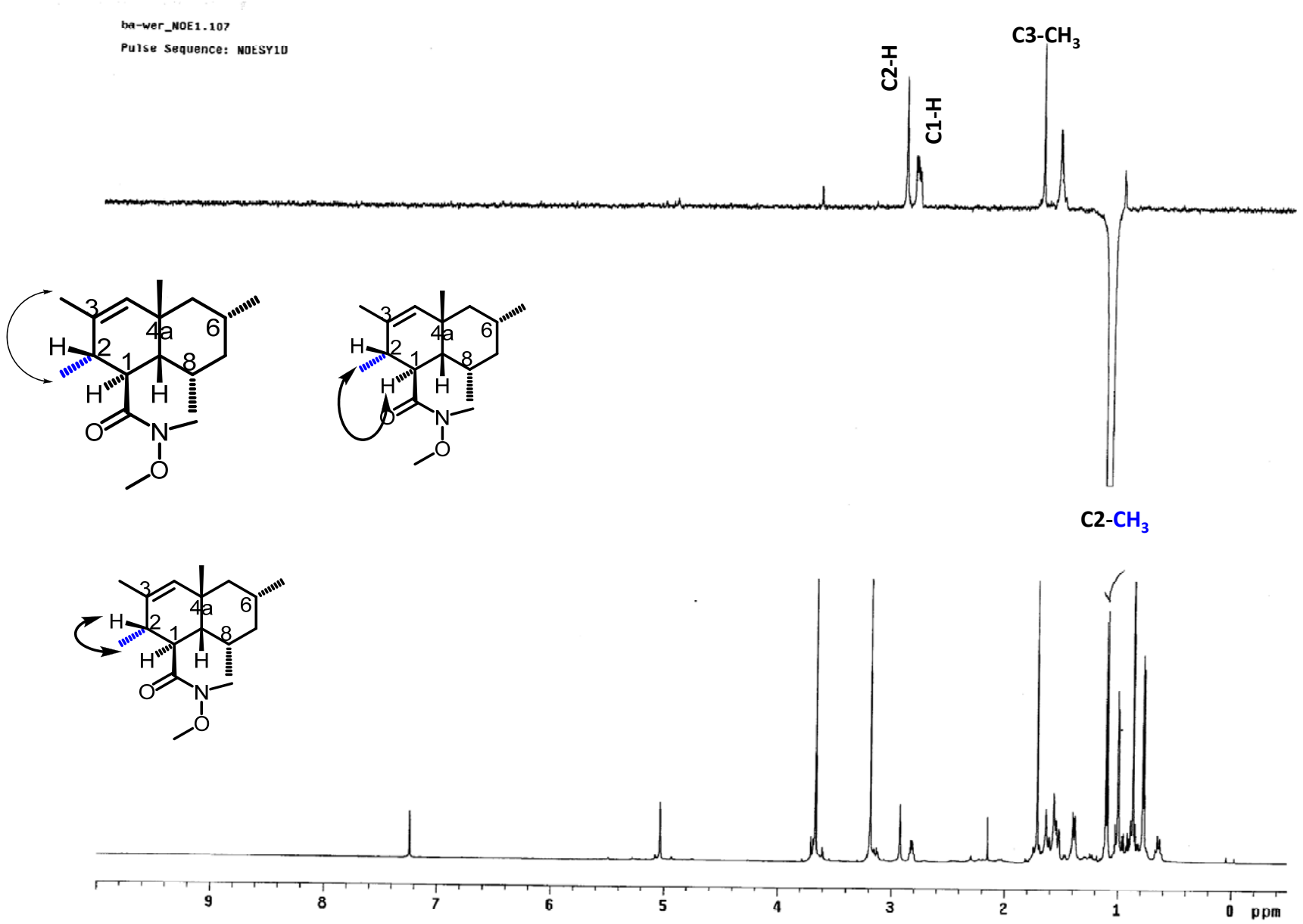

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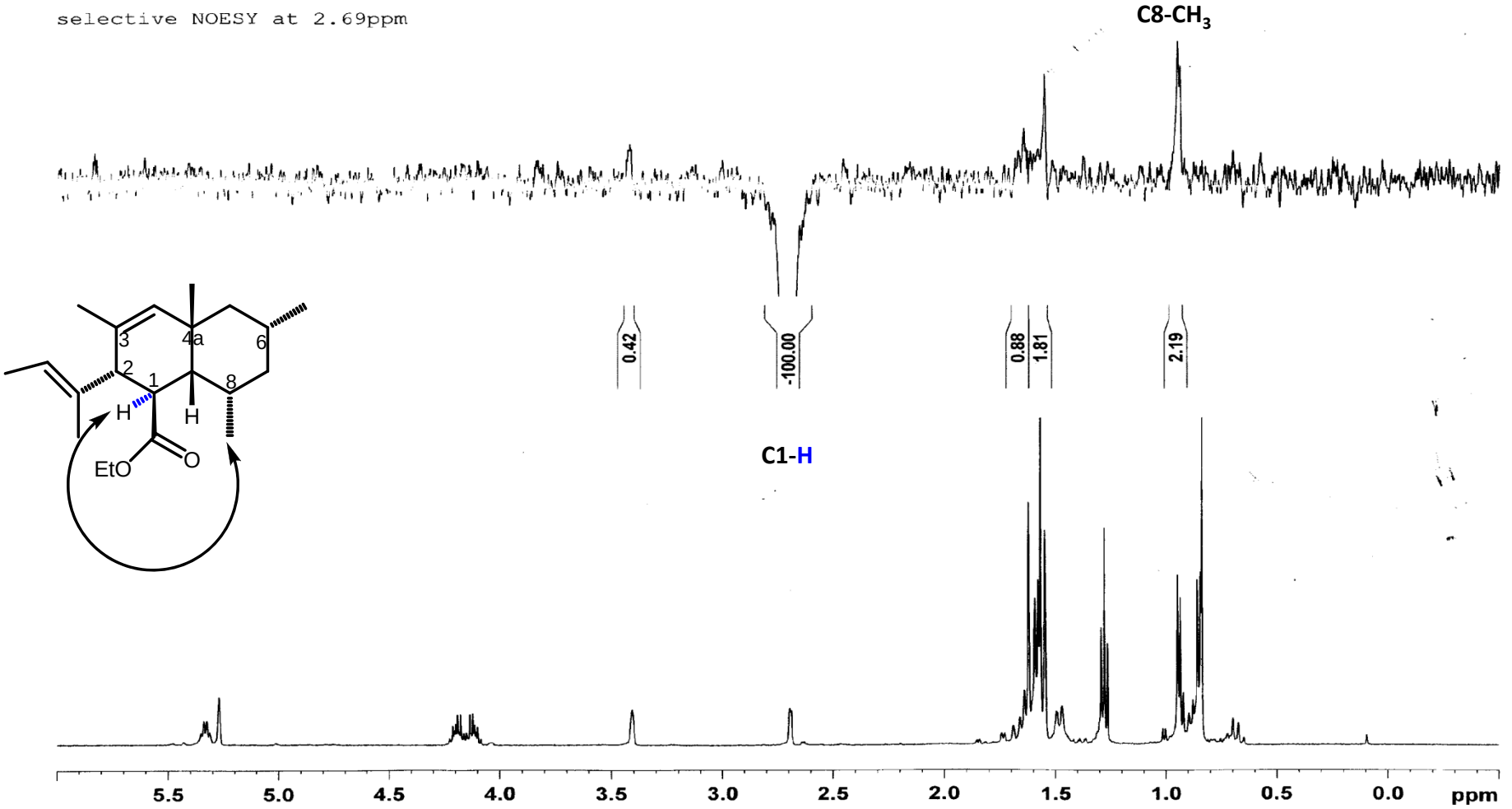

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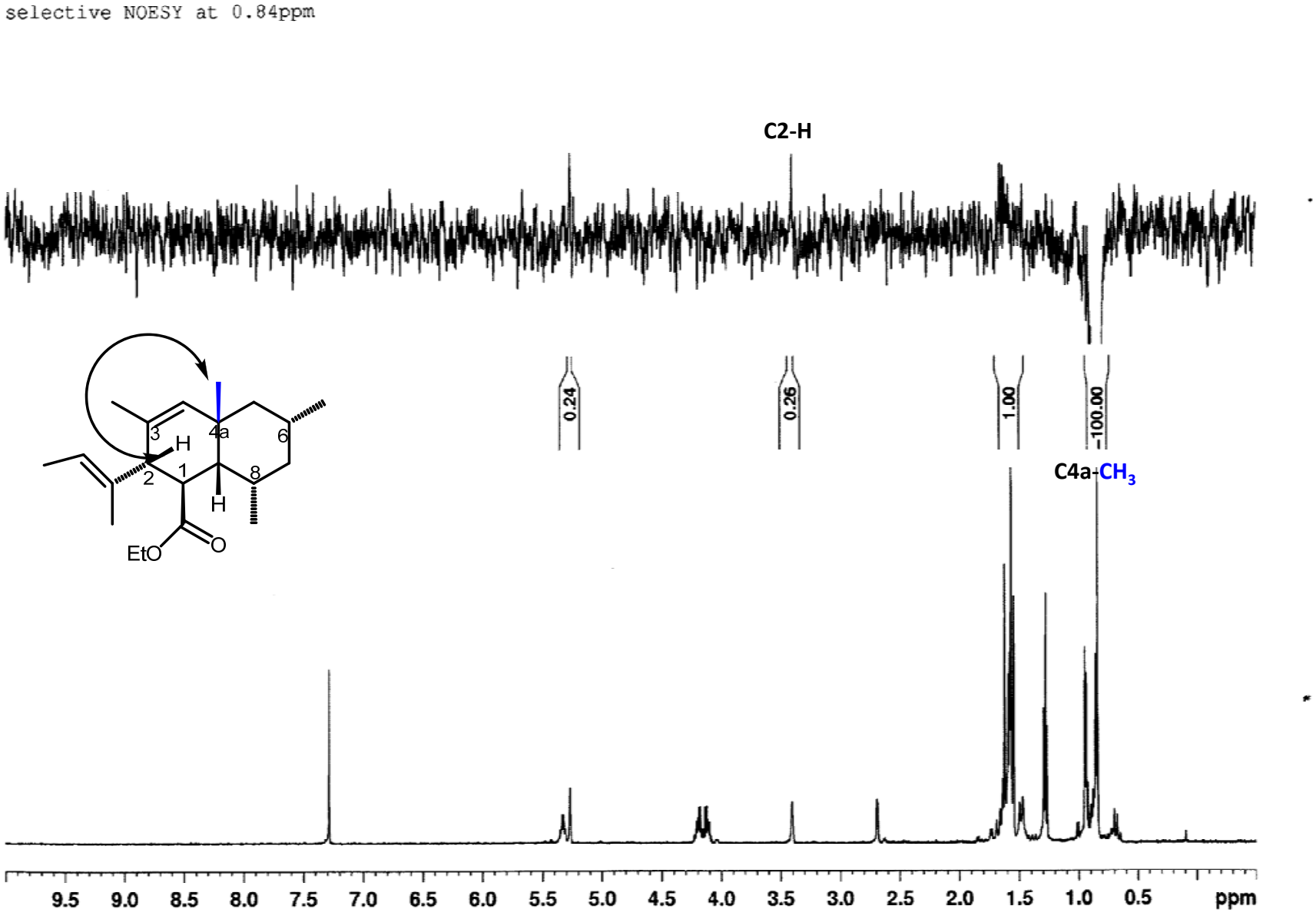

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