**Total Syntheses of Racemic, Natural (-) and Unnatural (+) Glyceollin I**

*Rahul S. Khupse and Paul W. Erhardt**

Department of Medicinal and Biological Chemistry
Center for Drug Design and Development (CD3)
The University of Toledo
2801 West Bancroft Street
Toledo, OH 43606-3390 (USA)
Fax: (+1) 419-530-1994
E-mail: paul.erhardt@utoledo.edu

# **Supporting Information (vi)** List of Spectra

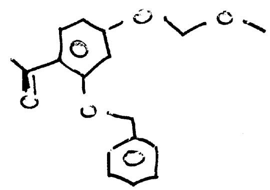
STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8723048 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec

May 3 2006



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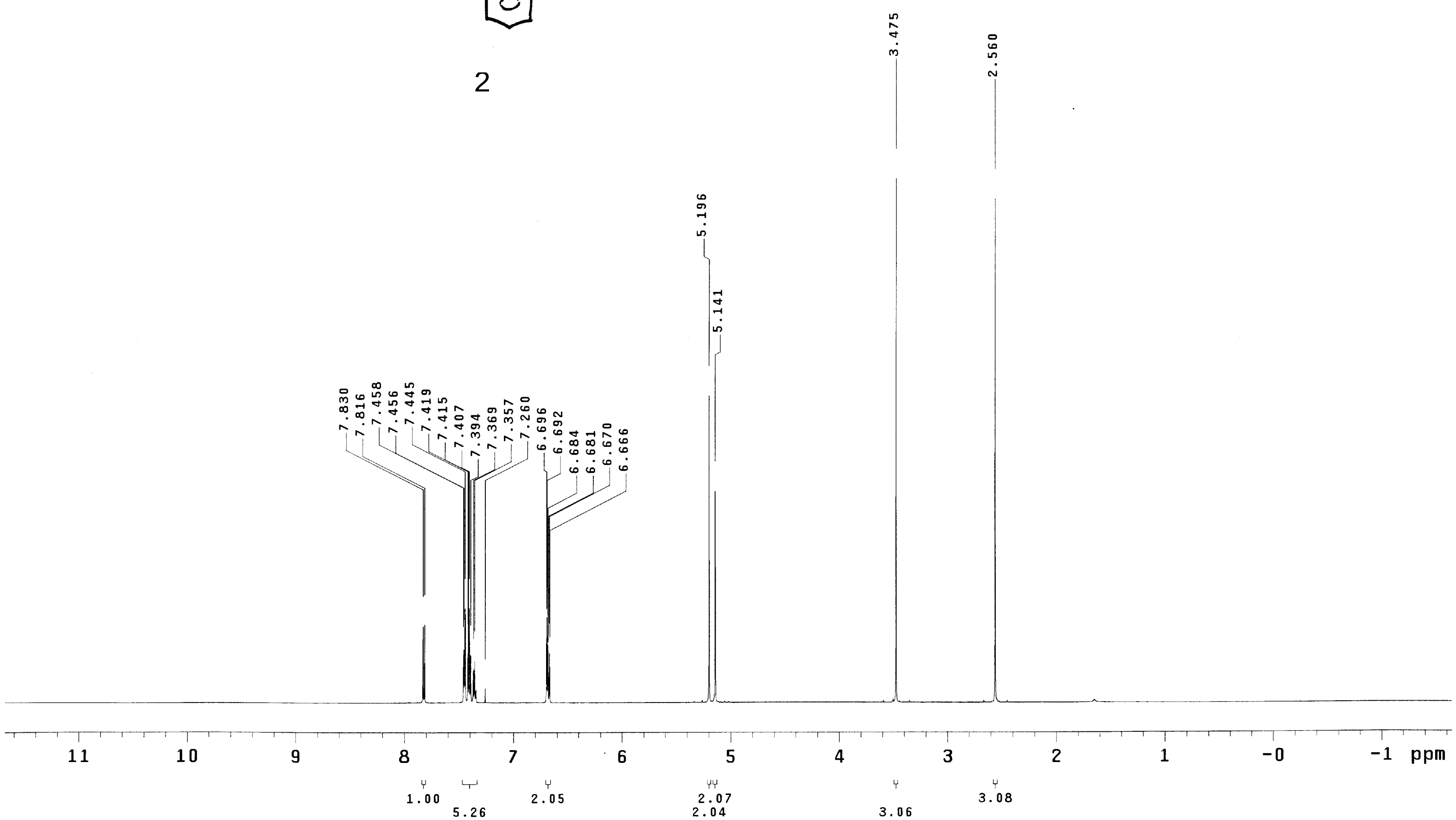
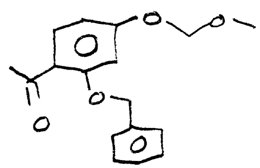


STANDARD PROTON PARAMETERS

Pulse Sequence: gcosy

Solvent: CDCl3
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.203 sec
Width 5042.2 Hz
2D Width 5042.2 Hz
Single scan
512 increments
OBSERVE H1, 599.8723048 MHz
DATA PROCESSING
Sine bell 0.102 sec
F1 DATA PROCESSING
Sine bell 0.051 sec
FT size 2048 x 2048
Total time 10 min, 51 sec



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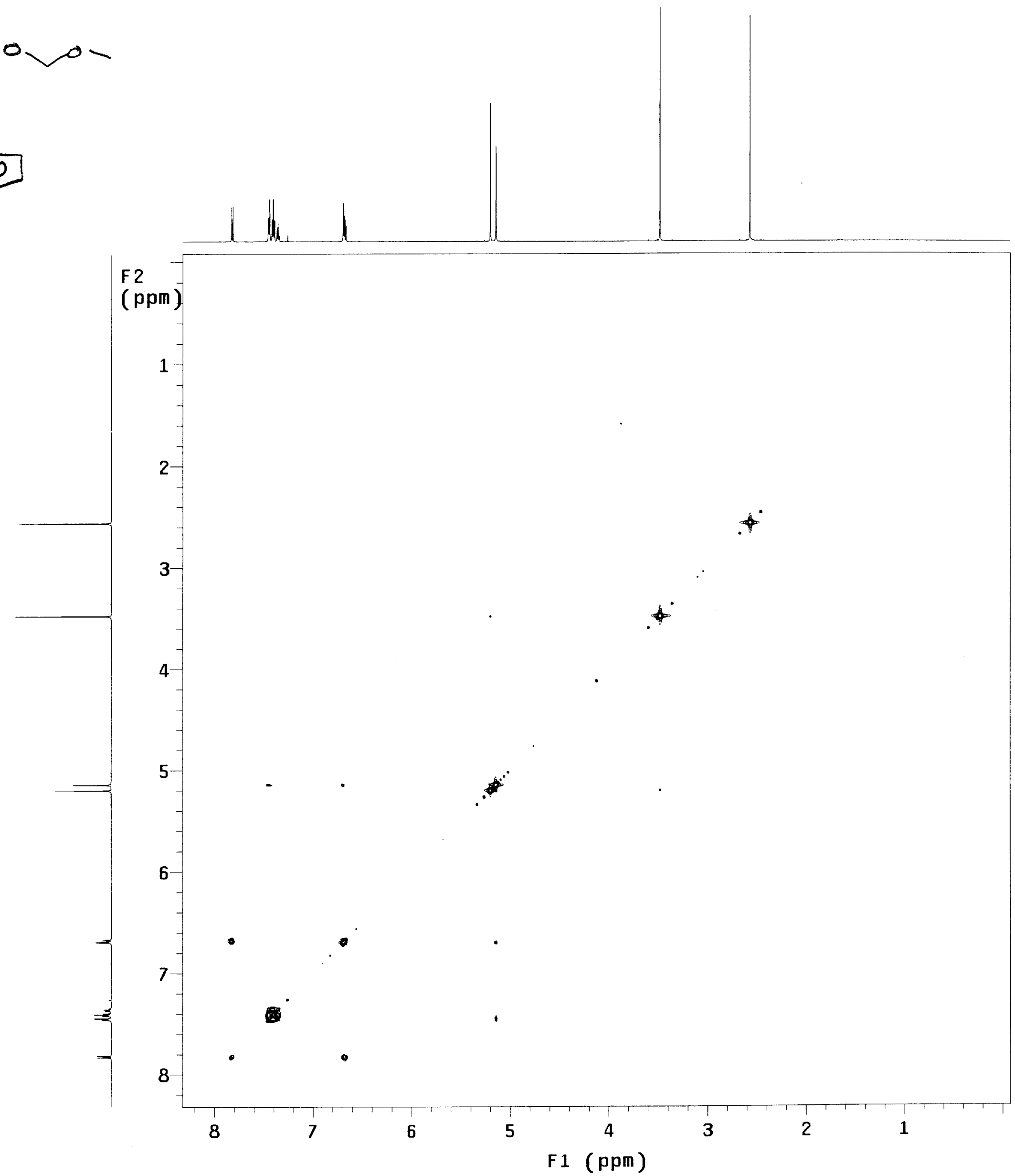
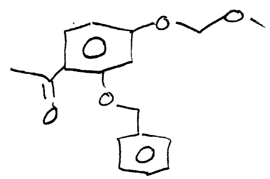


13C OBSERVE

May 4 2006

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
UNITY-400 "vxrs400"

Relax. delay 4.000 sec
Pulse 29.2 degrees
Acq. time 1.205 sec
Width 27192.4 Hz
384 repetitions
OBSERVE C13, 100.5671512 MHz
DECOUPLE H1, 399.9511041 MHz
Low power 1 dB atten.
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
Gauss window 0.500 sec
center at 0.100 sec
FT size 131072
Total time 1 hr, 29 min, 1 sec



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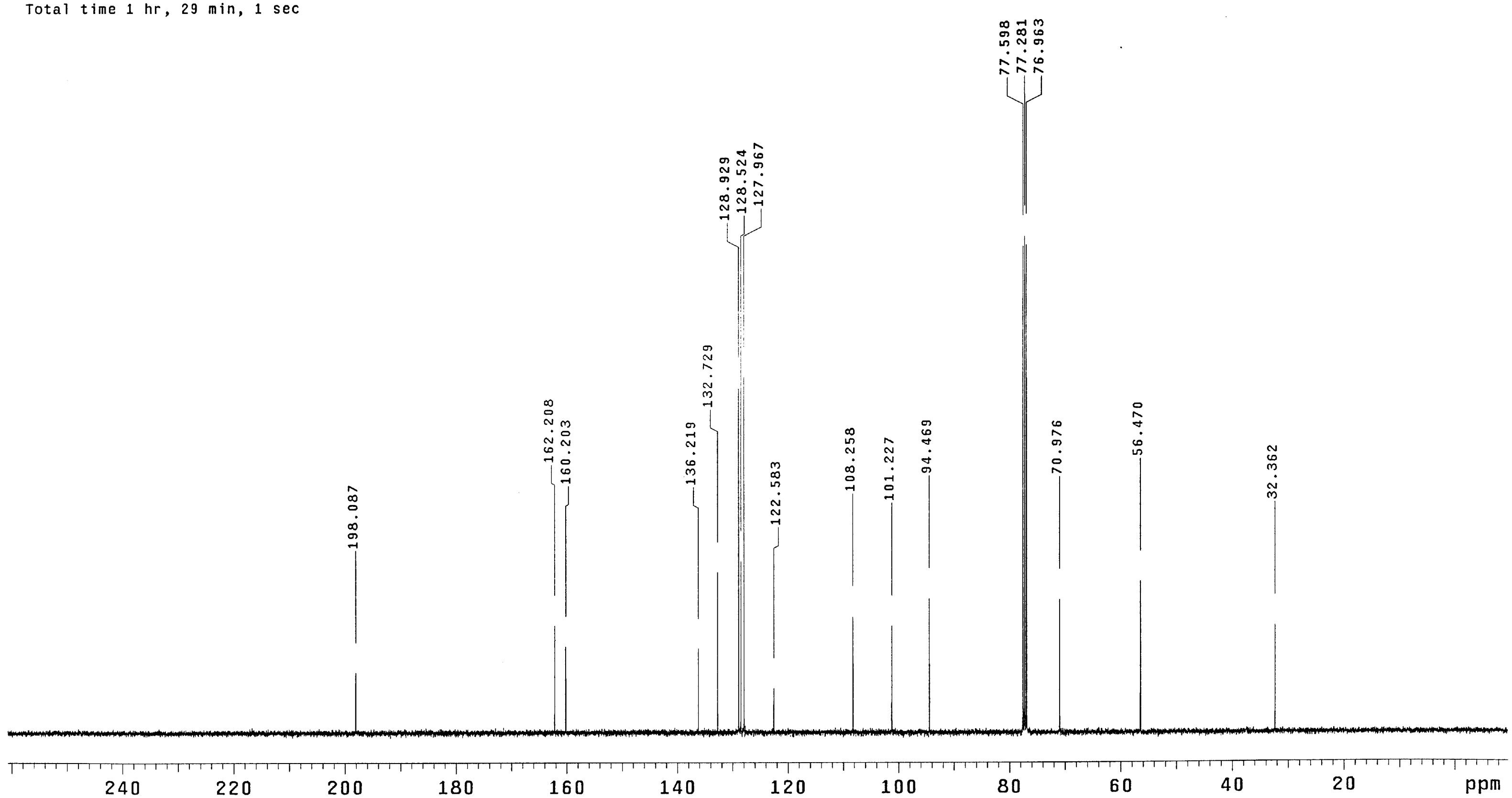
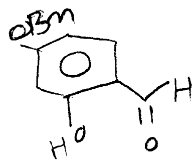


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: Acetone
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8754282 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec

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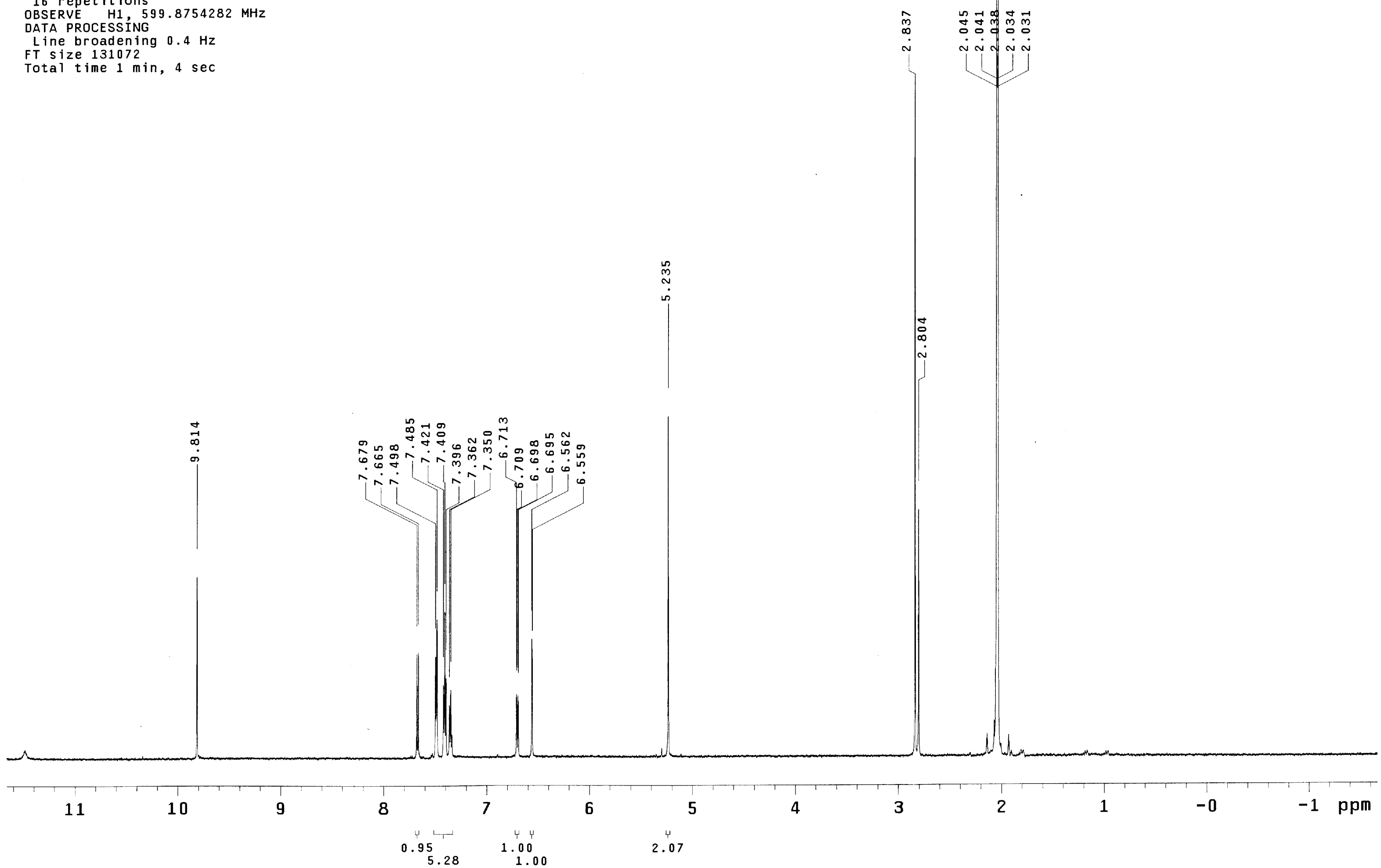
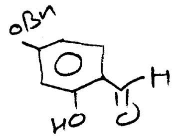


STANDARD PROTON PARAMETERS

Pulse Sequence: gcosy

Solvent: CDCl3
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.146 sec
Width 7001.0 Hz
2D Width 7001.0 Hz
Single scan
512 increments
OBSERVE H1, 599.8723148 MHz
DATA PROCESSING
Sine bell 0.073 sec
F1 DATA PROCESSING
Sine bell 0.037 sec
FT size 2048 x 2048
Total time 10 min, 15 sec

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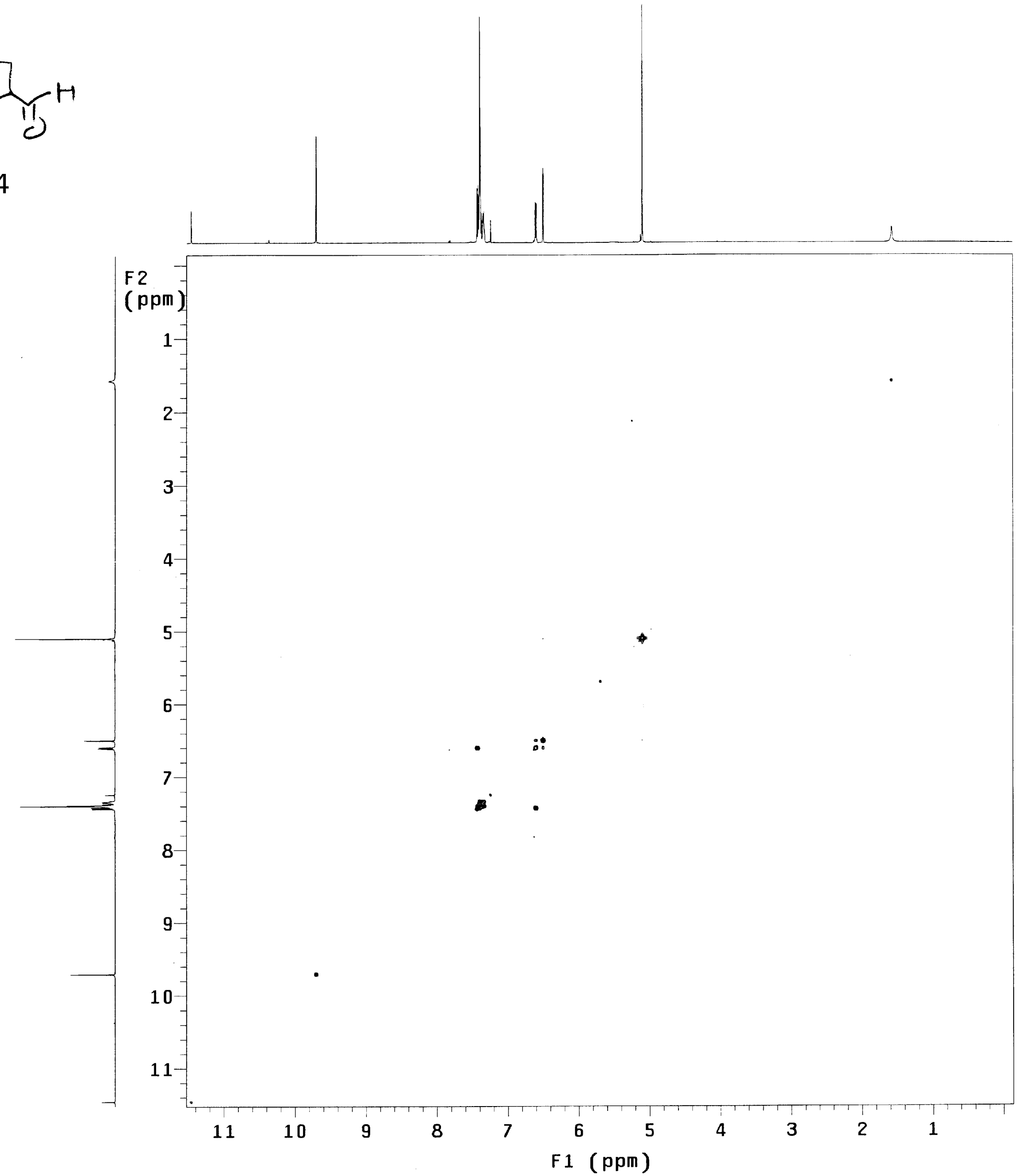
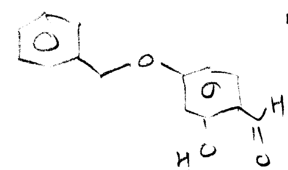


13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
UNITY-400 "vxrs400"

Relax. delay 4.000 sec
Pulse 29.2 degrees
Acq. time 1.205 sec
Width 27192.4 Hz
288 repetitions
OBSERVE C13, 100.5671512 MHz
DECOUPLE H1, 399.9511041 MHz
Low power 1 dB atten.
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
Gauss window 0.500 sec
center at 0.100 sec
FT size 131072
Total time 1 hr, 29 min, 1 sec

May 4 2006



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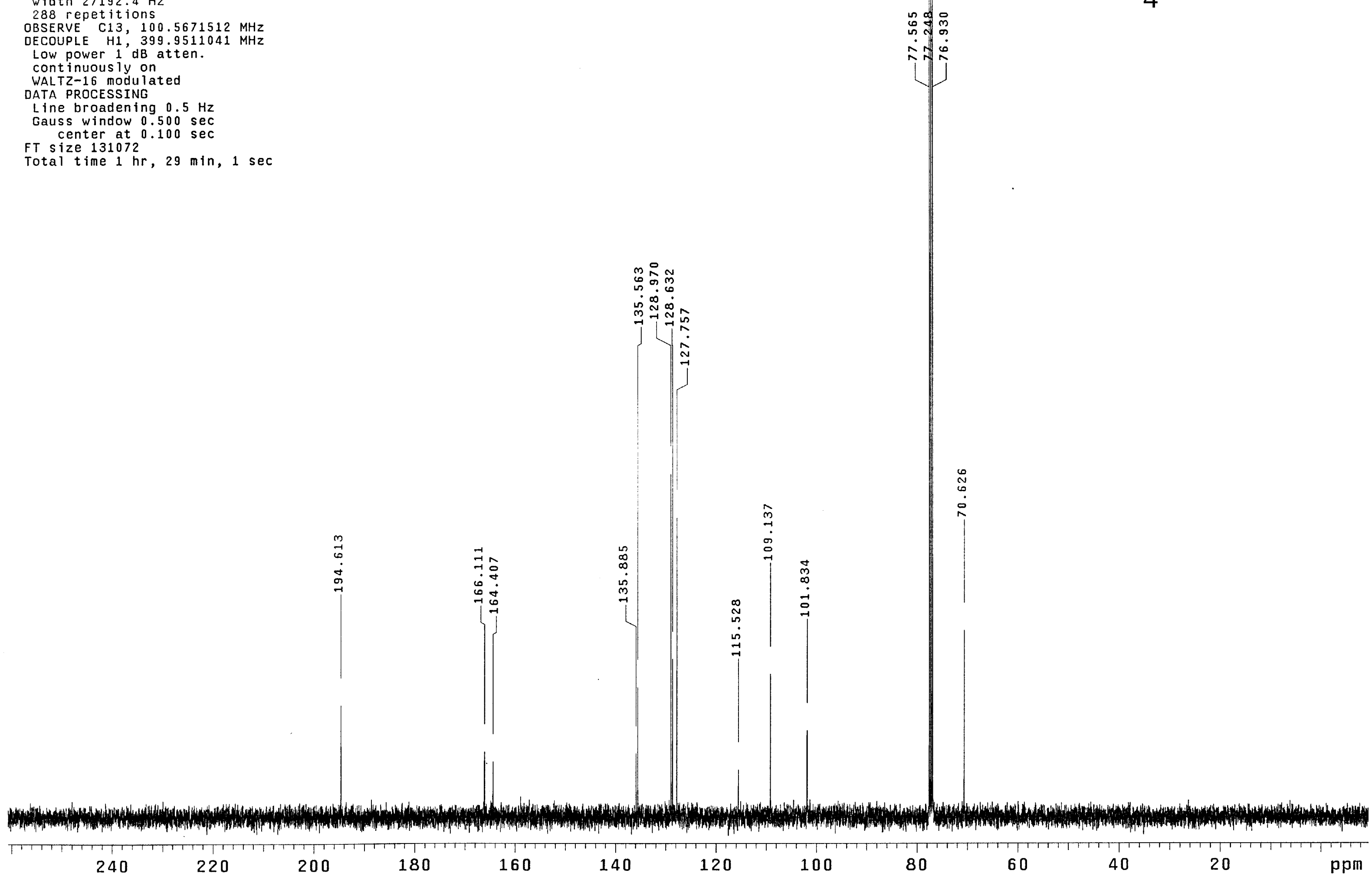


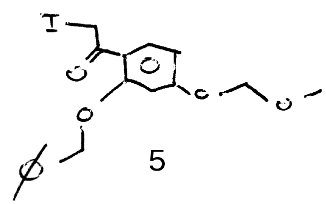
STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8723048 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec

May 3 2006



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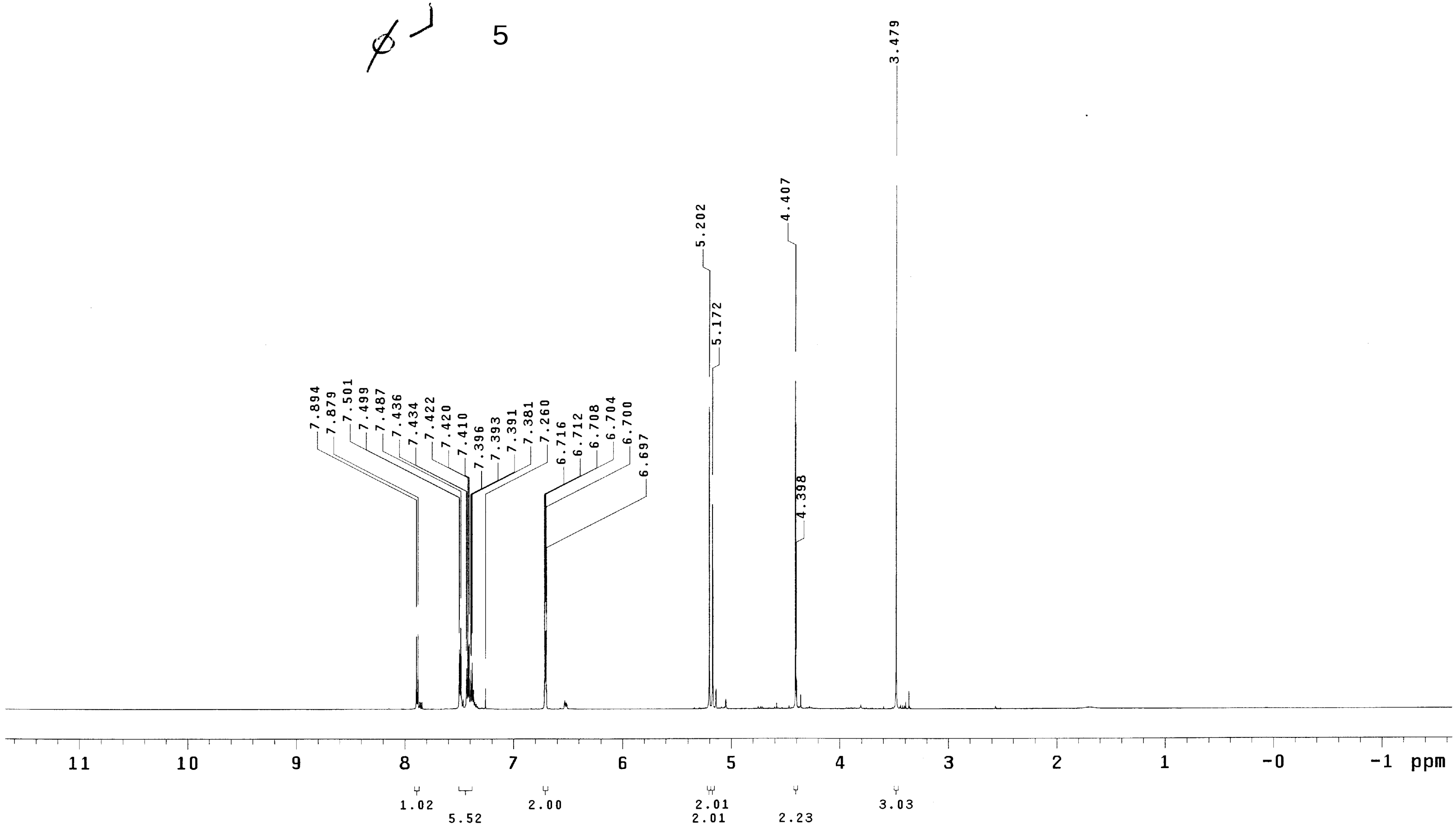
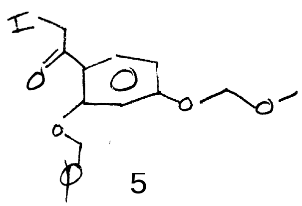


STANDARD PROTON PARAMETERS

Pulse Sequence: gcosy

Solvent: CDCl3
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.203 sec
Width 5035.6 Hz
2D Width 5035.6 Hz
Single scan
512 increments
OBSERVE H1, 599.8723048 MHz
DATA PROCESSING
Sine bell 0.102 sec
F1 DATA PROCESSING
Sine bell 0.051 sec
FT size 2048 x 2048
Total time 10 min, 51 sec



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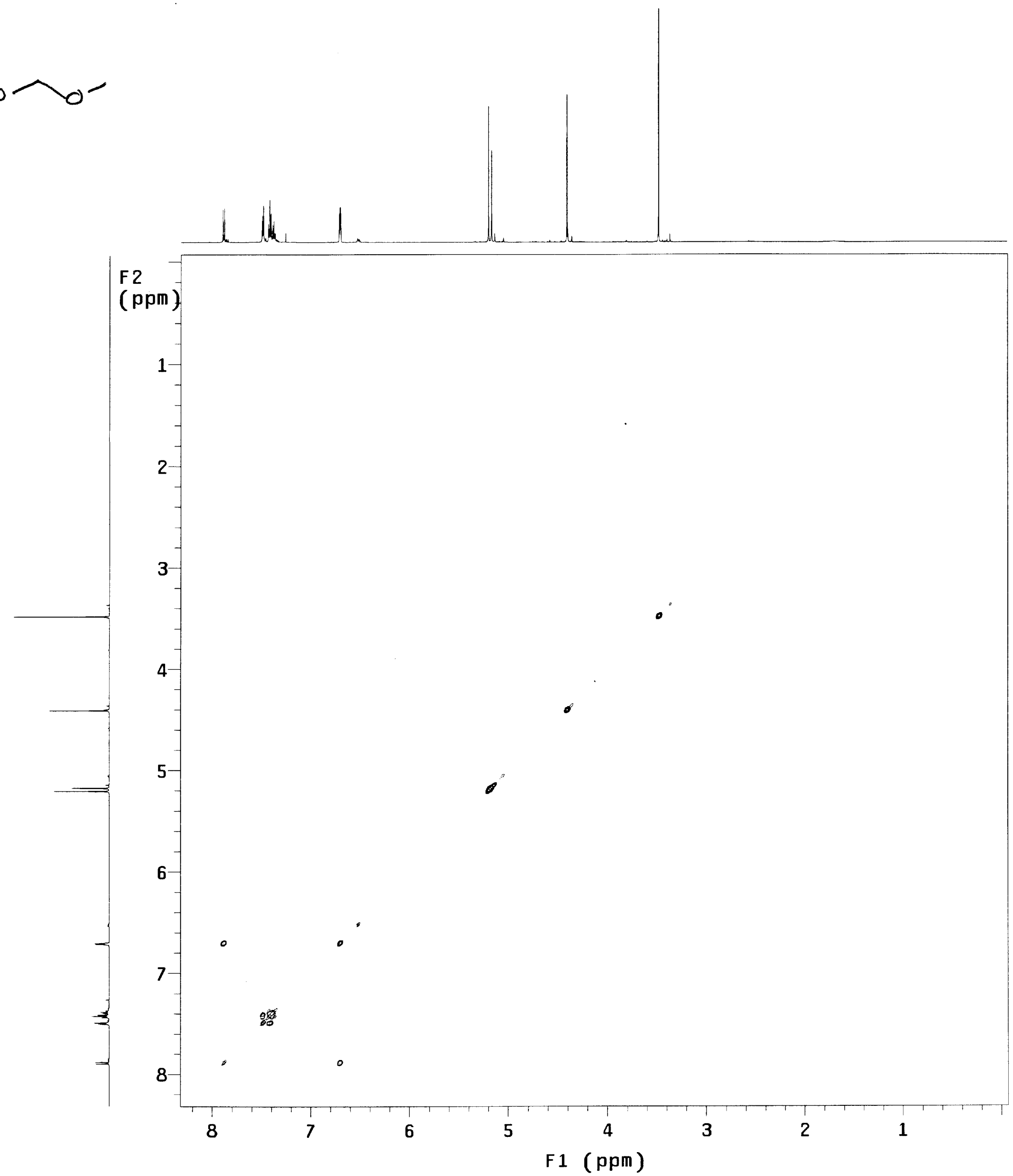
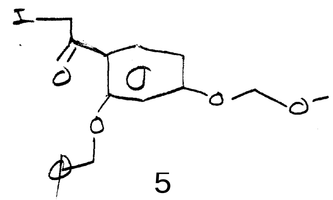


13C OBSERVE

May 4 2006

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
UNITY-400 "vxrs400"

Relax. delay 4.000 sec
Pulse 29.2 degrees
Acq. time 1.205 sec
Width 27192.4 Hz
1024 repetitions
OBSERVE C13, 100.5671530 MHz
DECOUPLE H1, 399.9511041 MHz
Low power 1 dB atten.
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
Gauss window 0.500 sec
center at 0.100 sec
FT size 131072
Total time 1 hr, 29 min, 1 sec



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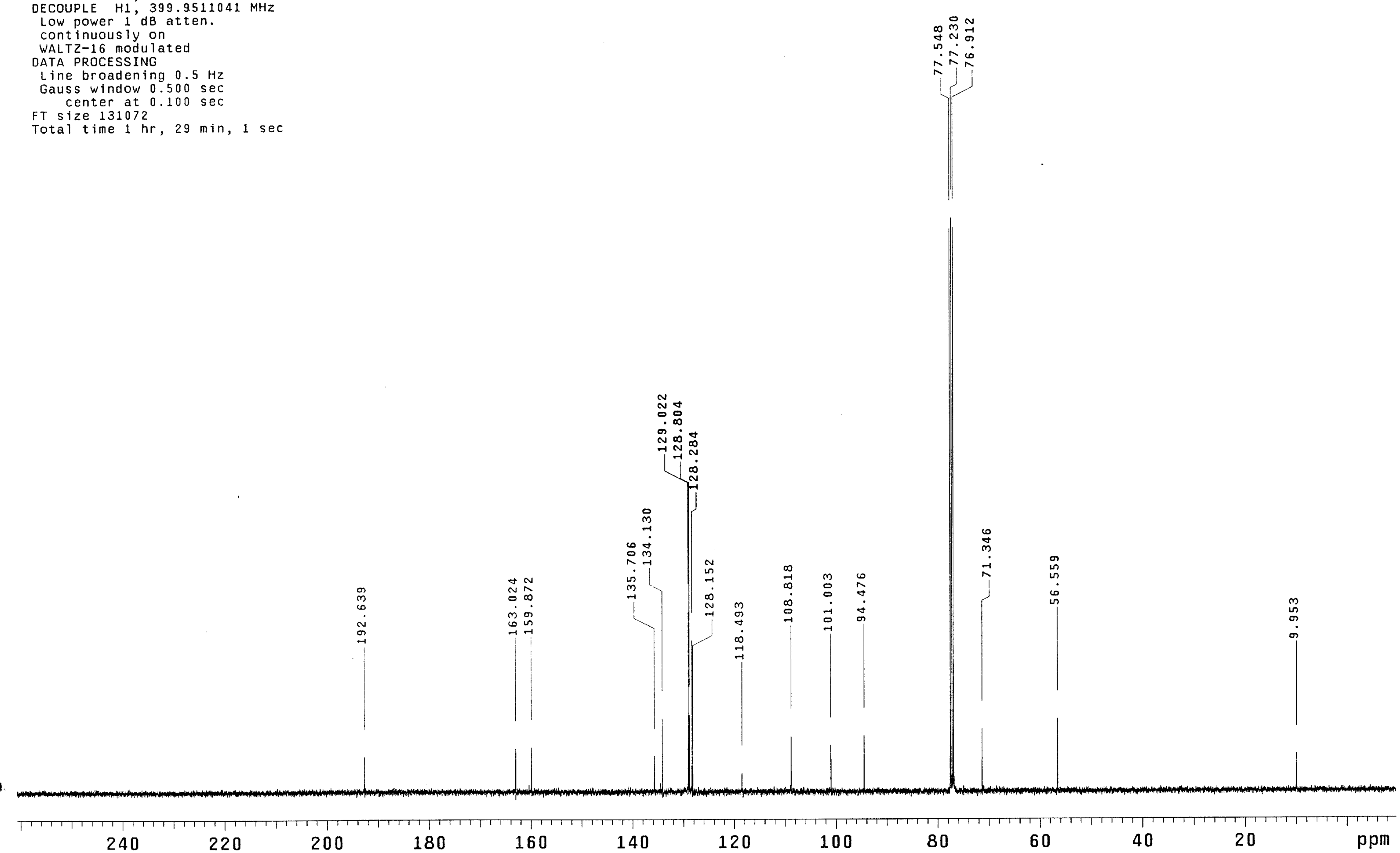
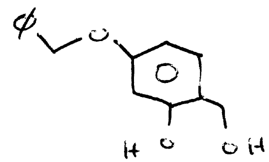


STANDARD PROTON PARAMETERS

May 3 2006

Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8751457 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



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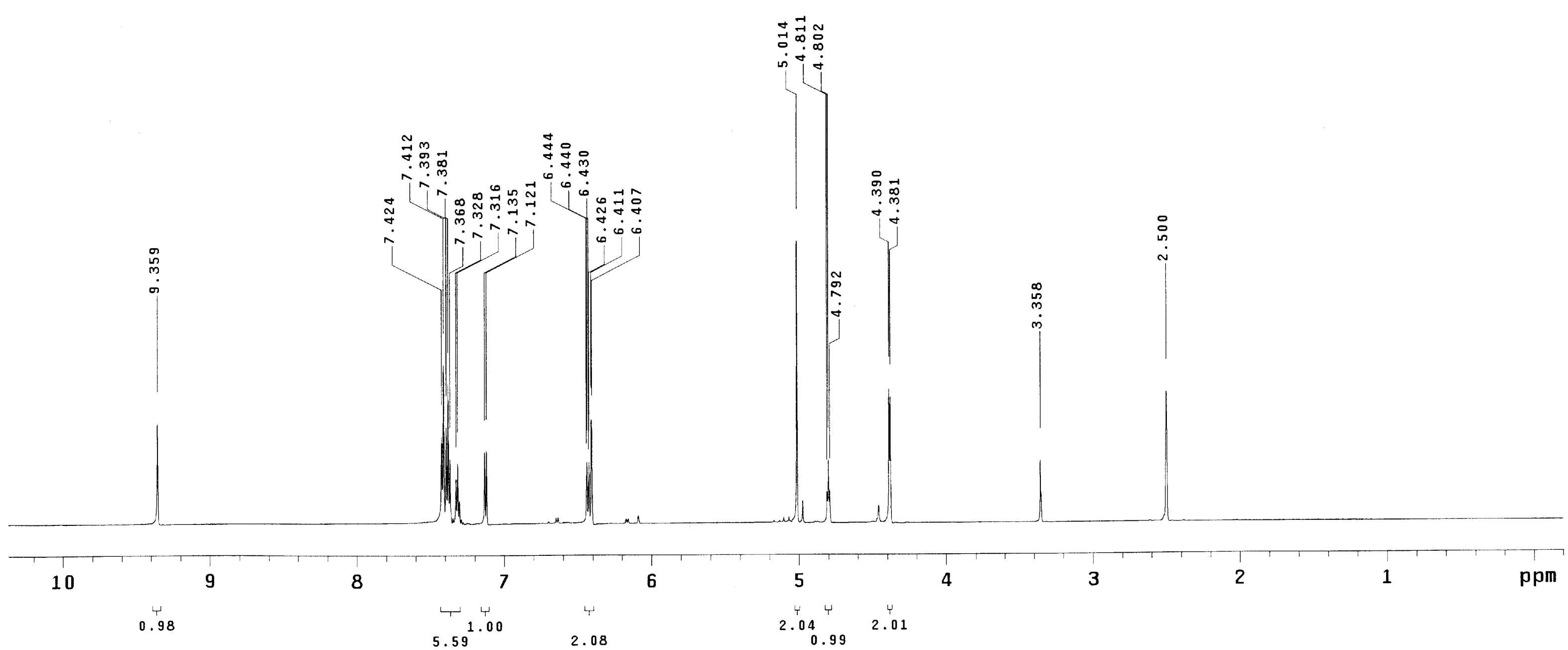
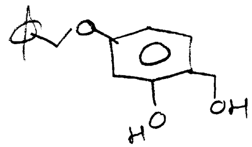


STANDARD PROTON PARAMETERS

Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.169 sec
Width 6047.8 Hz
2D Width 6047.8 Hz
Single scan
512 increments
OBSERVE H1, 599.8751642 MHz
DATA PROCESSING
Sine bell 0.085 sec
F1 DATA PROCESSING
Sine bell 0.042 sec
FT size 2048 x 2048
Total time 10 min, 29 sec

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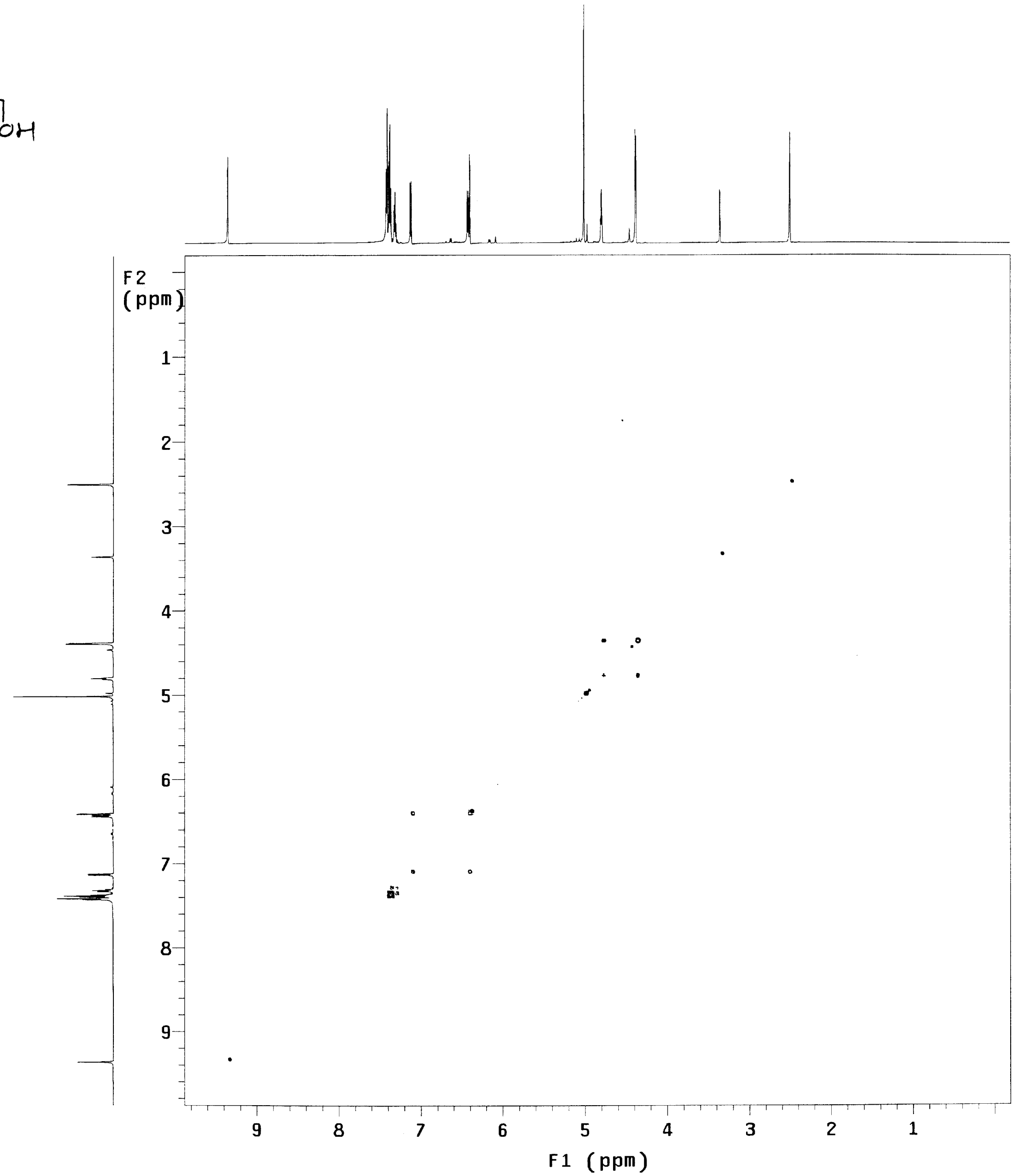
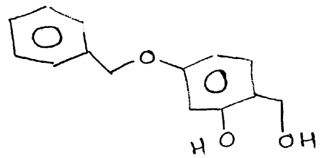


STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
User: 1-14-87
INOVA-600 "inova600"

Relax. delay 4.000 sec
Pulse 32.3 degrees
Acq. time 1.737 sec
Width 37735.8 Hz
208 repetitions
OBSERVE C13, 150.8387896 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss apodization 1.500 sec
FT size 524288
Total time 1 hr, 35 min, 45 sec

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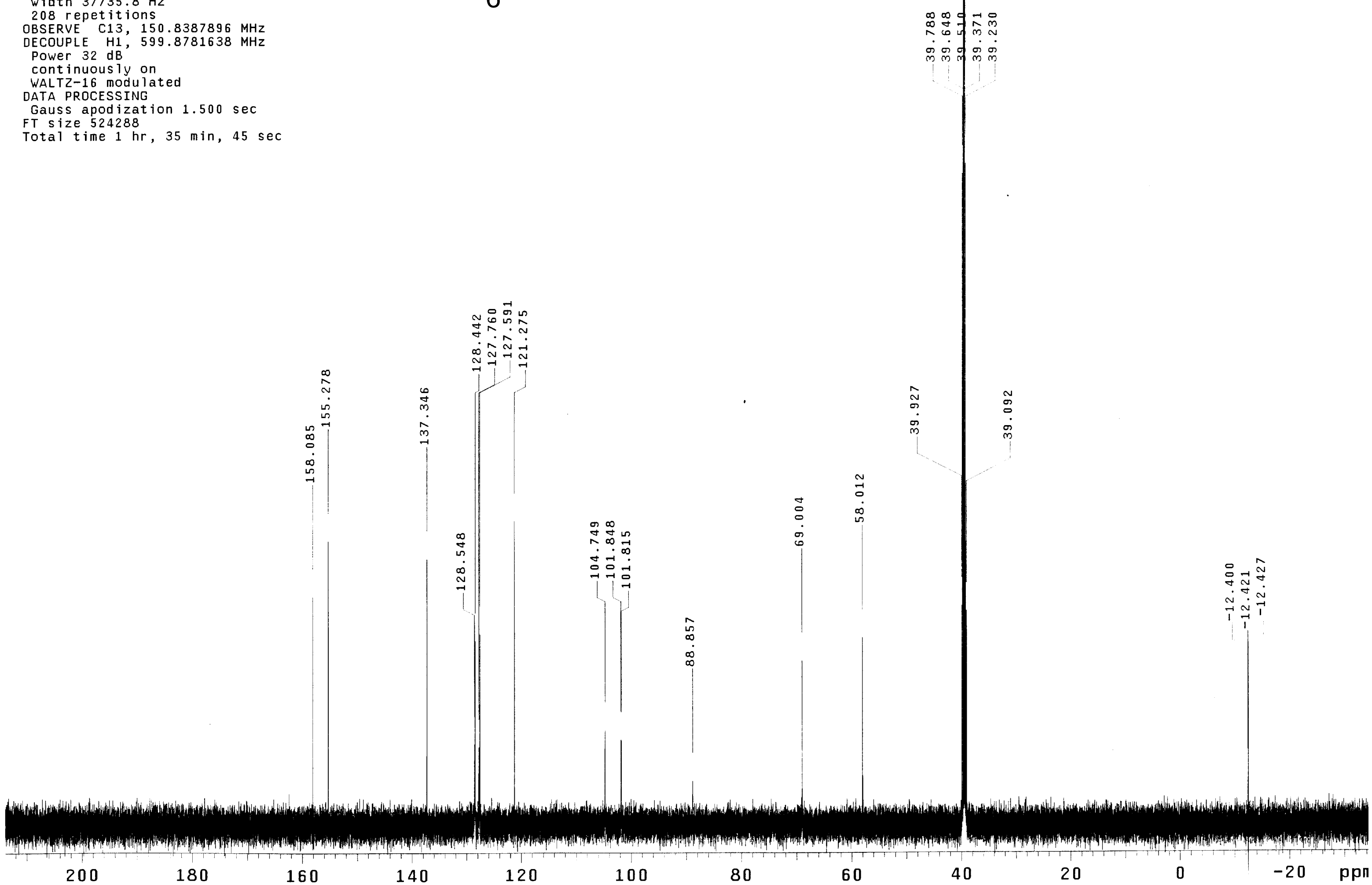
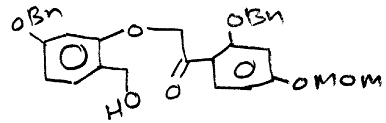


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: Acetone
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8754282 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec

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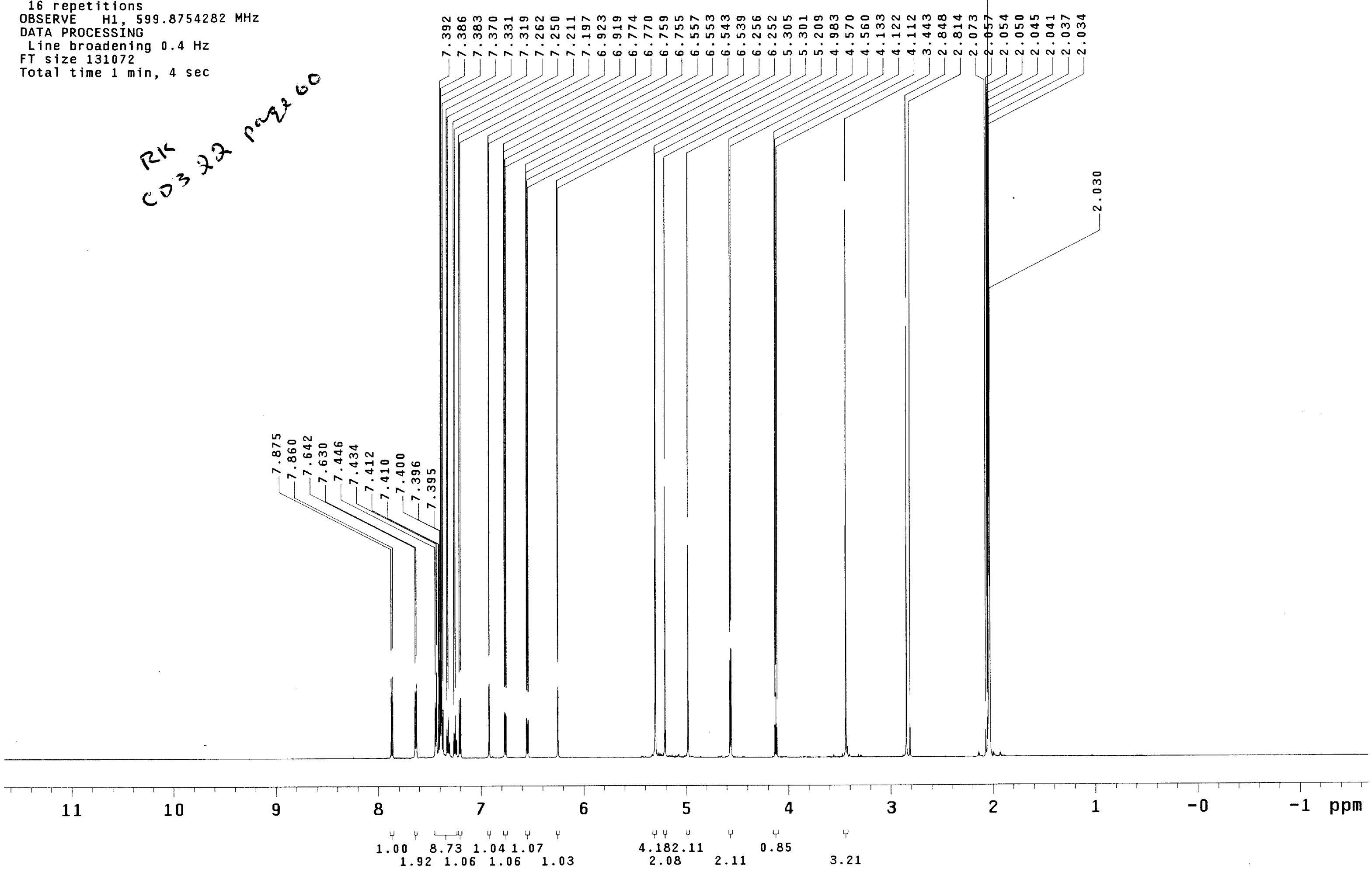
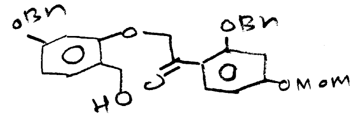


STANDARD PROTON PARAMETERS

Pulse Sequence: gcosy

Solvent: Acetone
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.197 sec
Width 5187.7 Hz
2D Width 5187.7 Hz
Single scan
512 increments
OBSERVE H1, 599.8754282 MHz
DATA PROCESSING
Sine bell 0.099 sec
F1 DATA PROCESSING
Sine bell 0.049 sec
FT size 2048 x 2048
Total time 10 min, 47 sec

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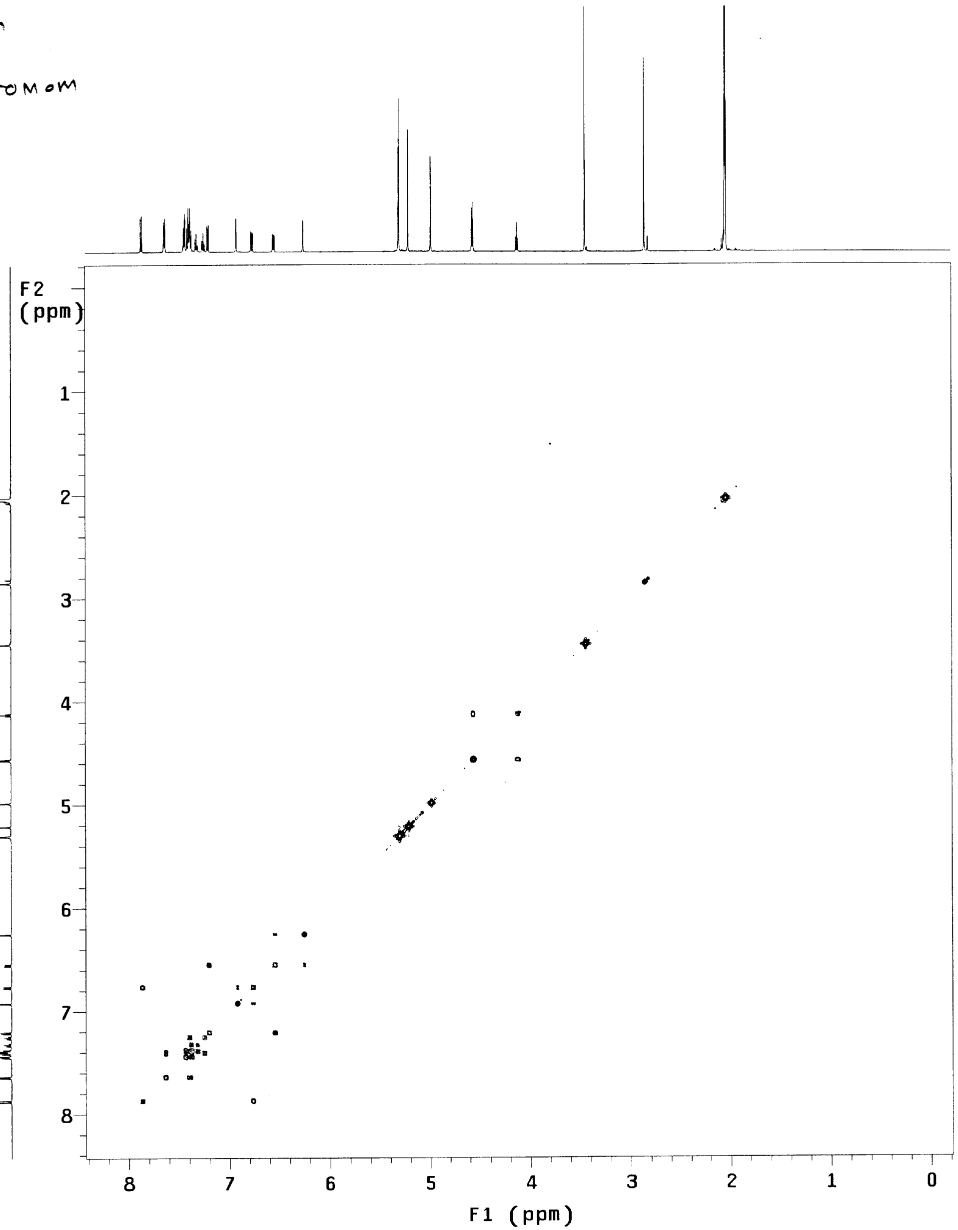
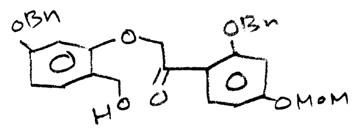


13C OBSERVE

Pulse Sequence: s2pul
Solvent: Acetone
Ambient temperature
UNITY-400 "vxrs400"

Relax. delay 4.000 sec
Pulse 29.2 degrees
Acq. time 1.205 sec
Width 27192.4 Hz
672 repetitions
OBSERVE C13, 100.5675533 MHz
DECOUPLE H1, 399.9531799 MHz
Low power 1 dB atten.
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
Gauss window 0.500 sec
center at 0.100 sec
FT size 131072
Total time 1 hr, 29 min, 1 sec

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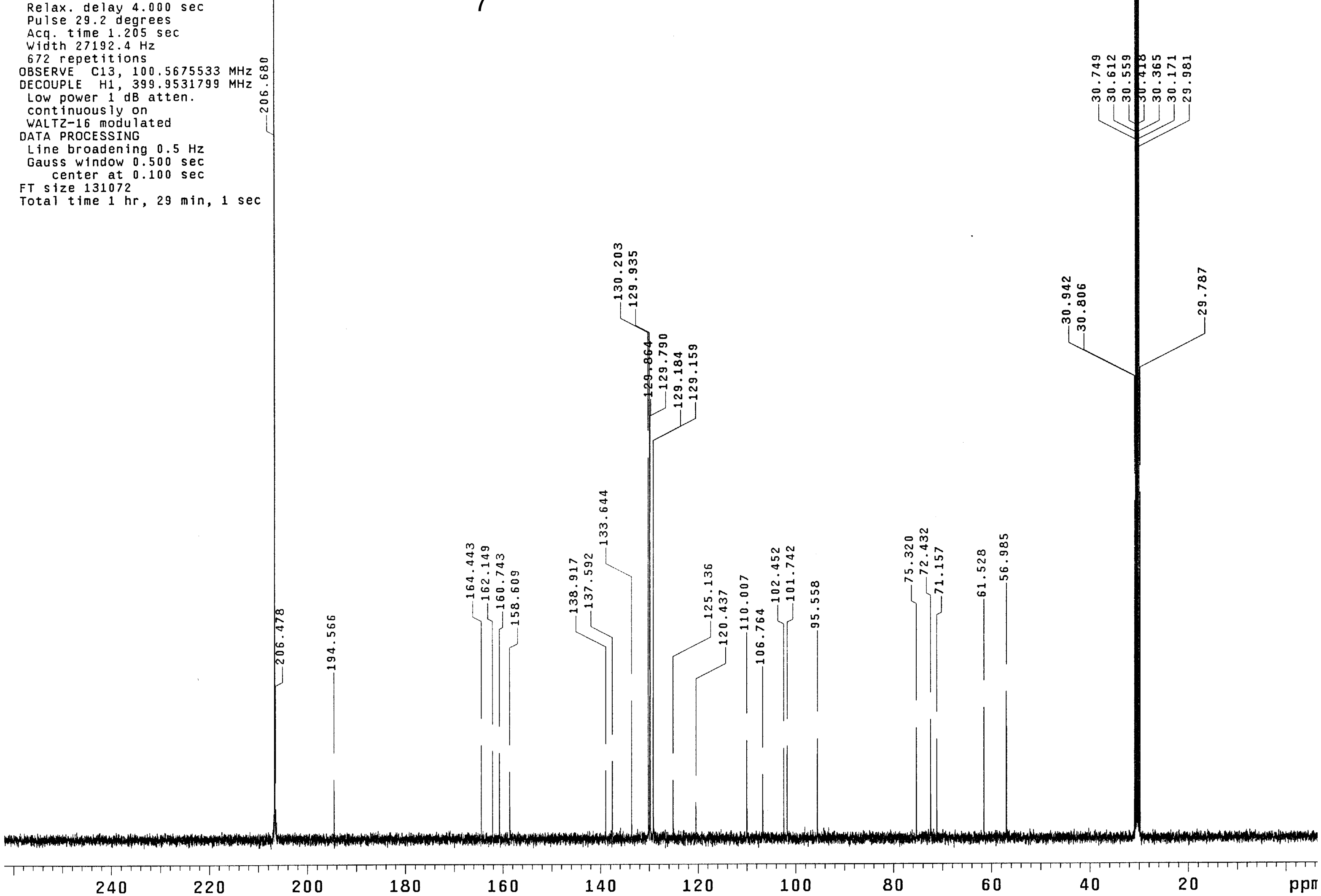
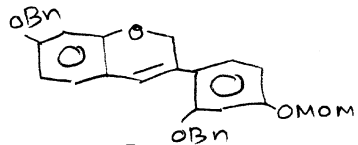


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul
Solvent: Acetone
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8754282 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec

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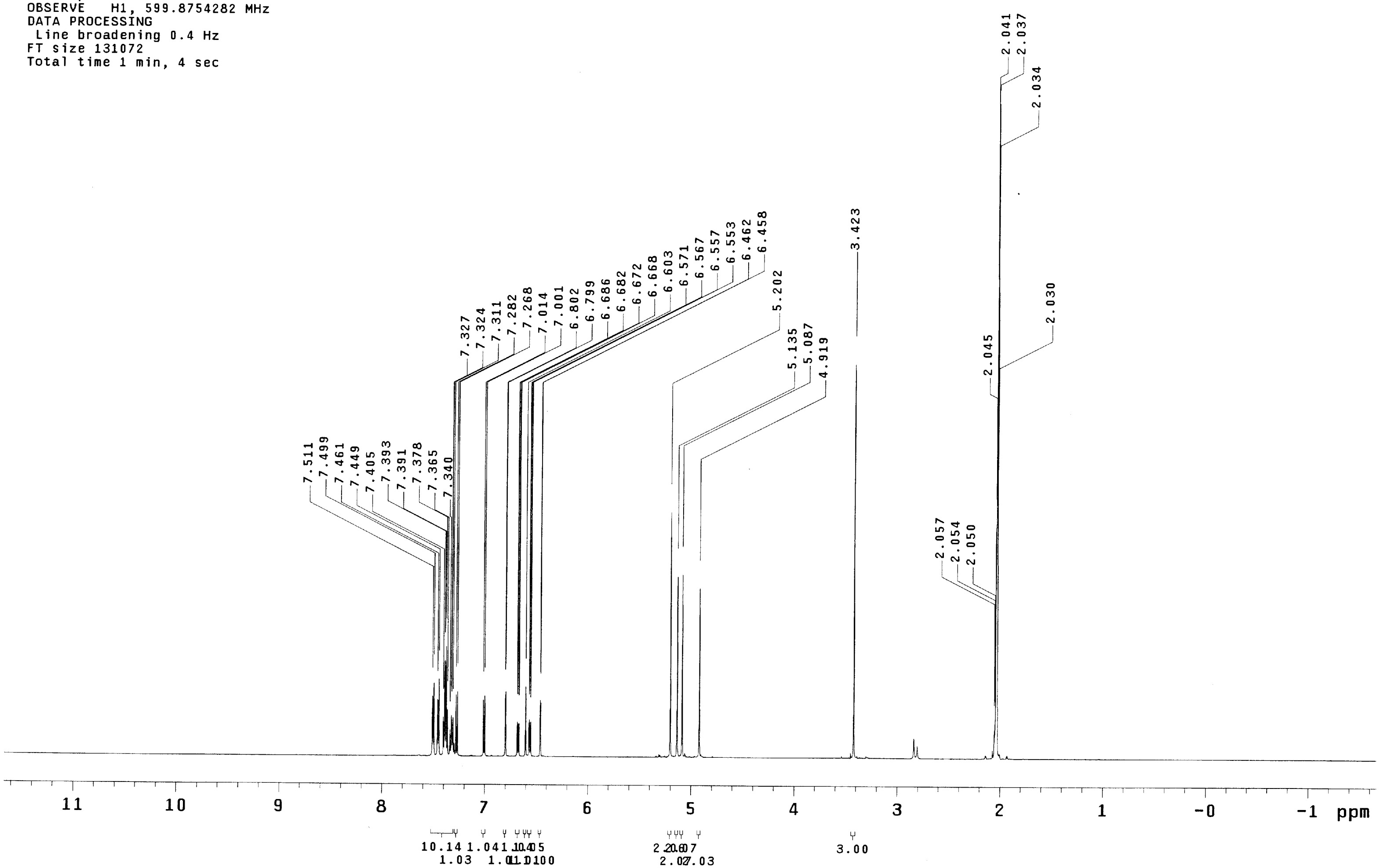
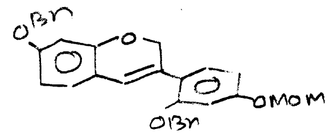


STANDARD PROTON PARAMETERS

Pulse Sequence: gcosy

Solvent: Acetone
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.128 sec
Width 8000.0 Hz
2D Width 8000.0 Hz
Single scan
512 increments
OBSERVE H1, 599.8754282 MHz
DATA PROCESSING
Sine bell 0.064 sec
F1 DATA PROCESSING
Sine bell 0.032 sec
FT size 2048 x 2048
Total time 10 min, 3 sec

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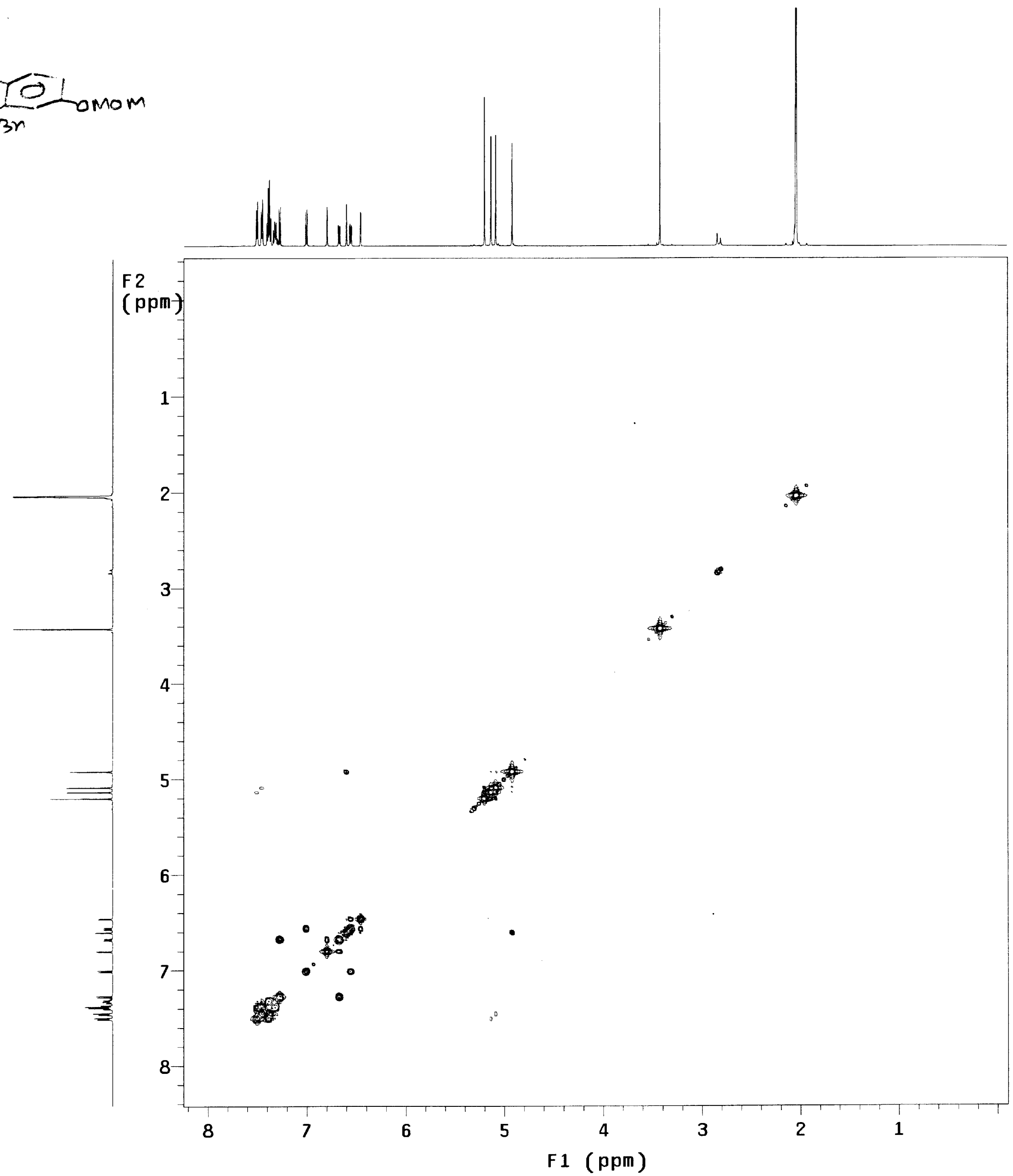

13C OBSERVE

Pulse Sequence: s2pul

Solvent: Acetone
Ambient temperature
File: Nb7C13
UNITY-400 "vxrs400"

Relax. delay 4.000 sec
Pulse 29.2 degrees
Acq. time 1.205 sec
Width 27192.4 Hz
992 repetitions
OBSERVE C13, 100.5675459 MHz
DECOUPLE H1, 399.9531799 MHz
Low power 1 dB atten.
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
Gauss window 0.500 sec
center at 0.100 sec
FT size 131072
Total time 1 hr, 29 min, 1 sec

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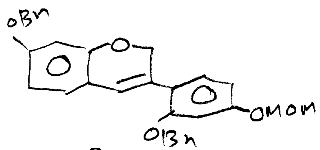


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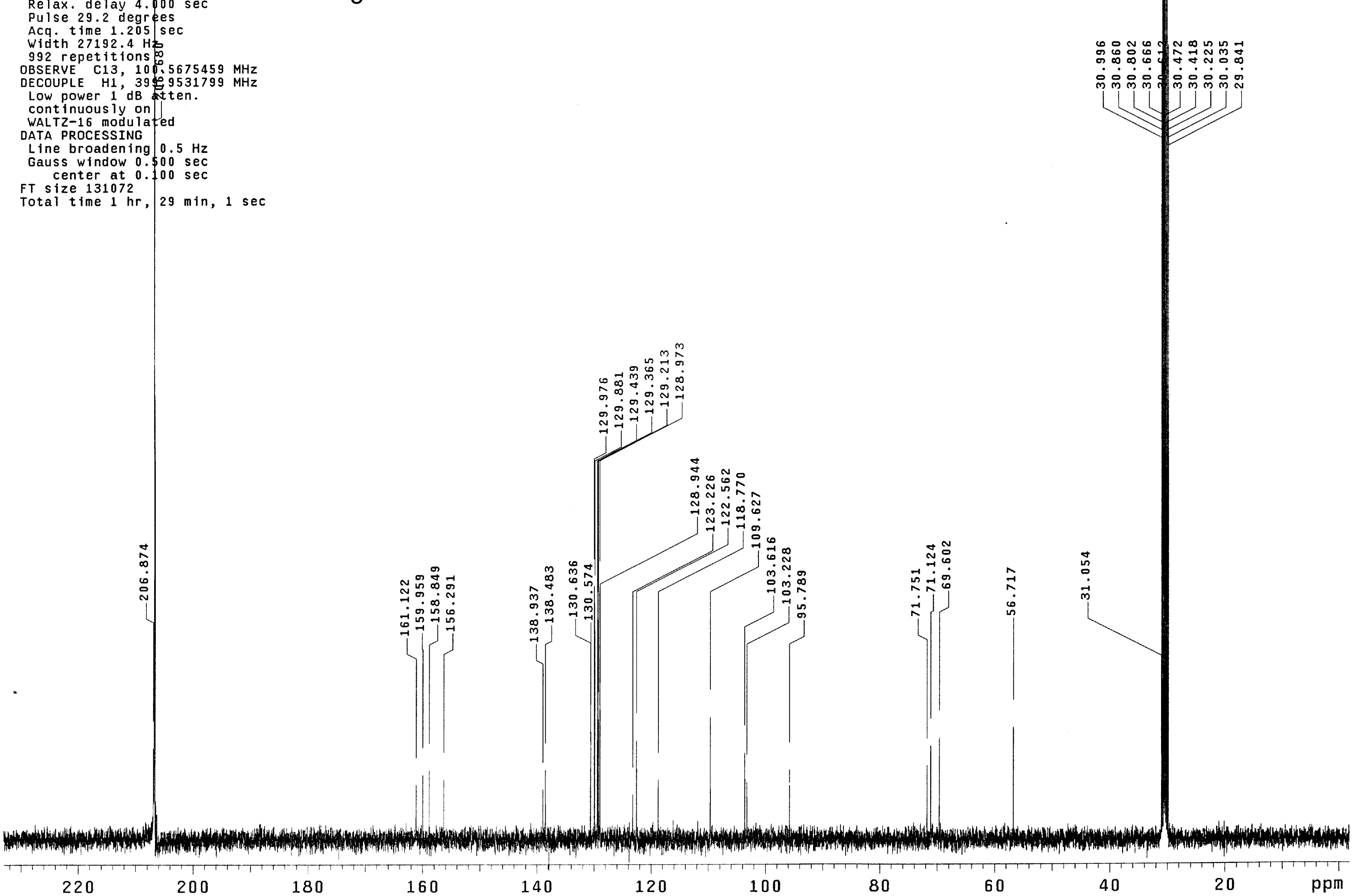

STANDARD 1H OBSERVE

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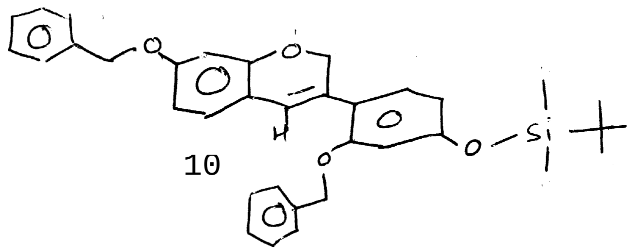
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
UNITY-400 "vxrs400"

Relax. delay 2.000 sec
Pulse 29.5 degrees
Acq. time 2.731 sec
Width 5999.7 Hz
32 repetitions
OBSERVE H1, 399.9505713 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 2 min, 31 sec

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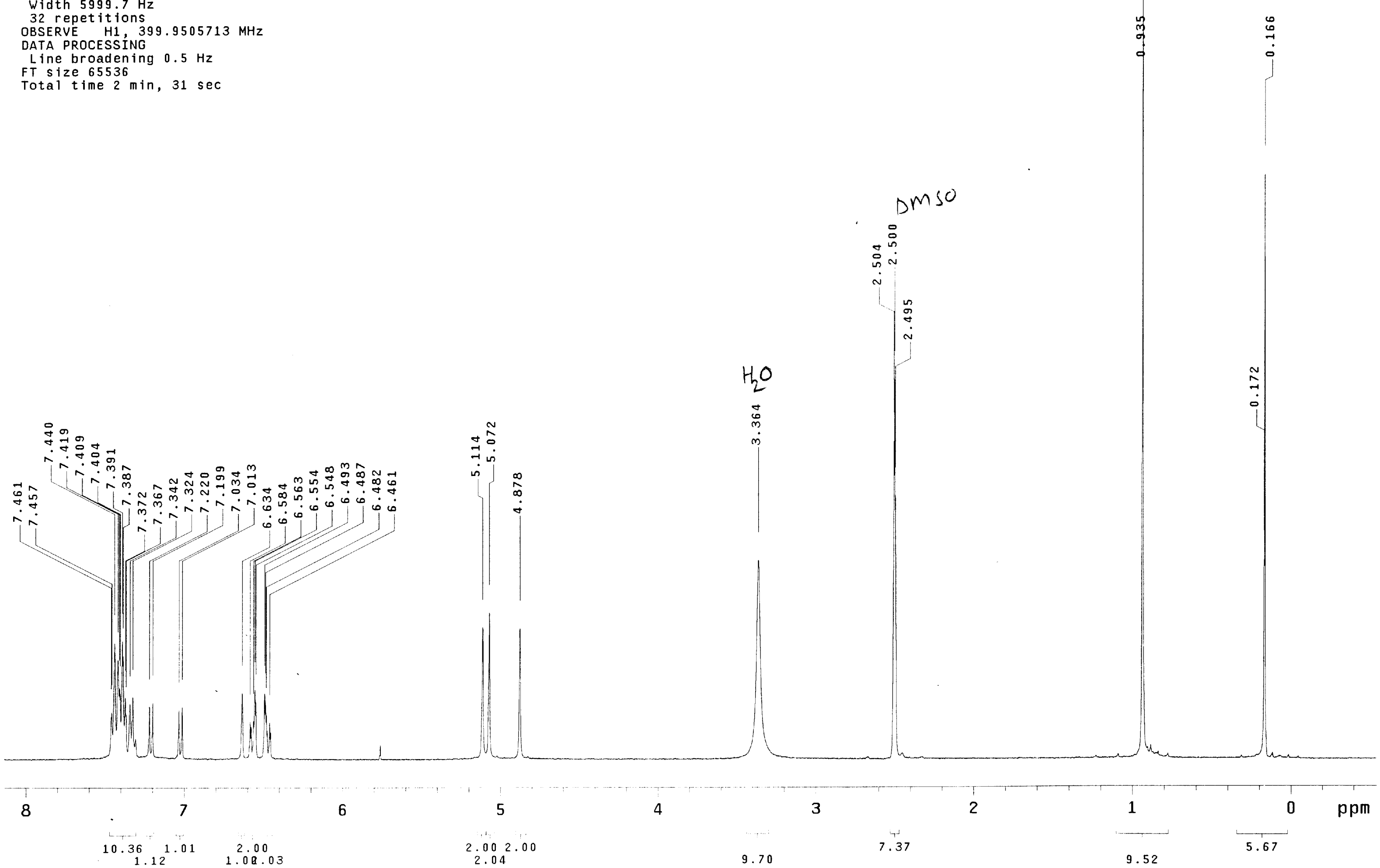
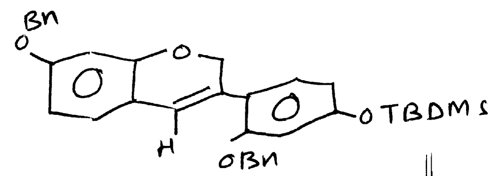


STANDARD 1H OBSERVE

Pulse Sequence: relayh
Solvent: DMSO
Ambient temperature
UNITY-400 "vxrs400"

Relax. delay 1.000 sec
COSY 90-90
Acq. time 0.152 sec
Width 3379.2 Hz
2D Width 3379.2 Hz
4 repetitions
256 increments
OBSERVE H1, 399.9505713 MHz
DATA PROCESSING
Sine bell 0.076 sec
F1 DATA PROCESSING
Sine bell 0.038 sec
FT size 1024 x 1024
Total time 20 min, 32 sec

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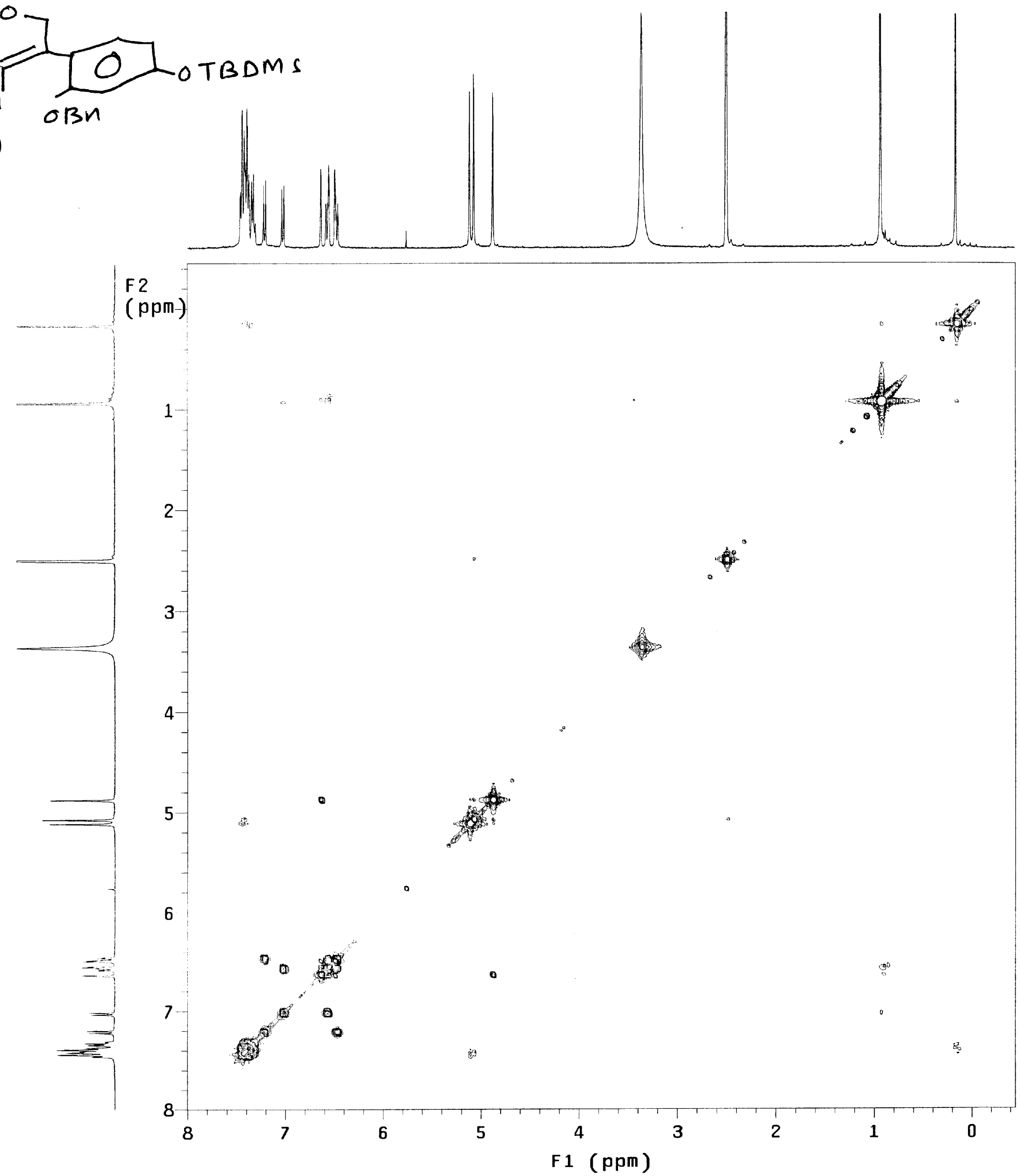
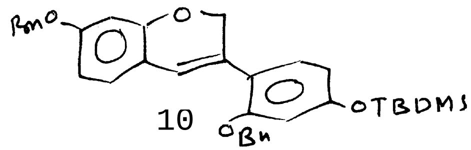


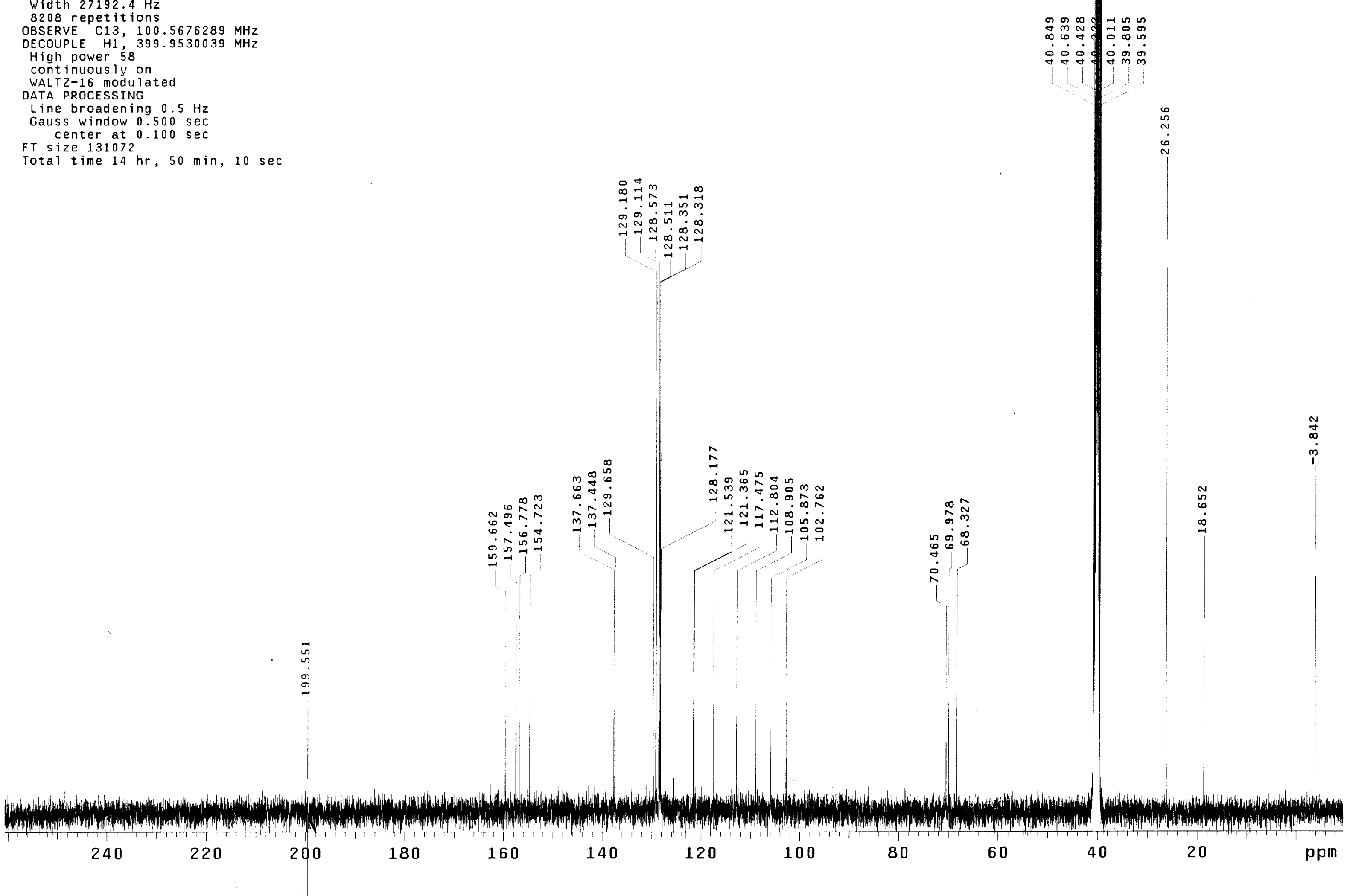
13C OBSERVE

Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
UNITY-400 "vxrs400"

Relax. delay 4.000 sec
Pulse 39.8 degrees
Acq. time 1.205 sec
Width 27192.4 Hz
8208 repetitions
OBSERVE C13, 100.5676289 MHz
DECOUPLE H1, 399.9530039 MHz
High power 58
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
Gauss window 0.500 sec
center at 0.100 sec
FT size 131072
Total time 14 hr, 50 min, 10 sec




Mar 21 2005



STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: Acetone
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8754282 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec

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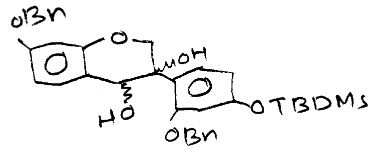


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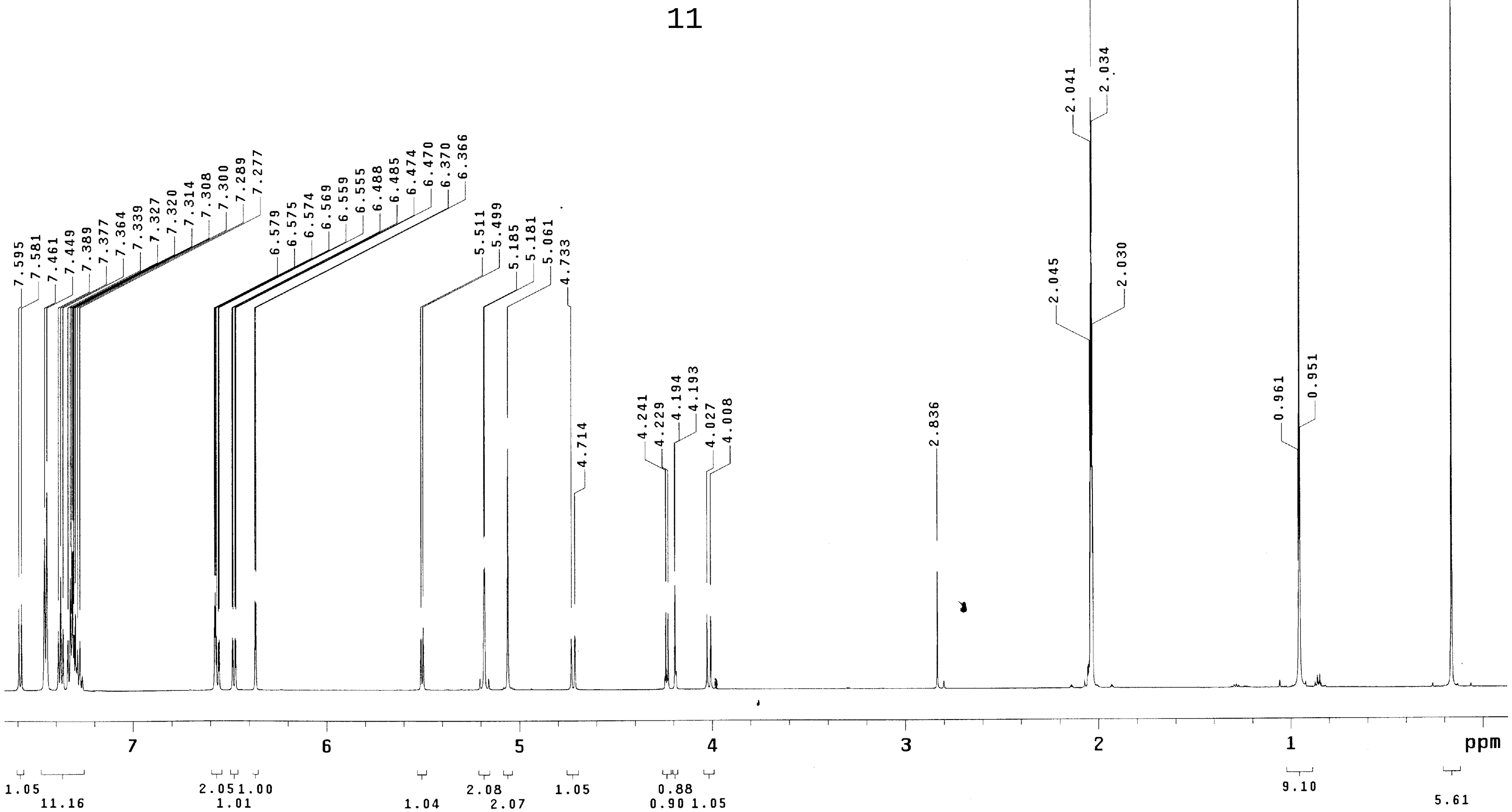
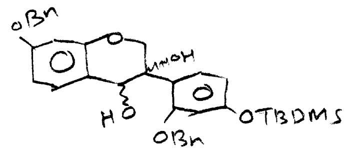


STANDARD PROTON PARAMETERS

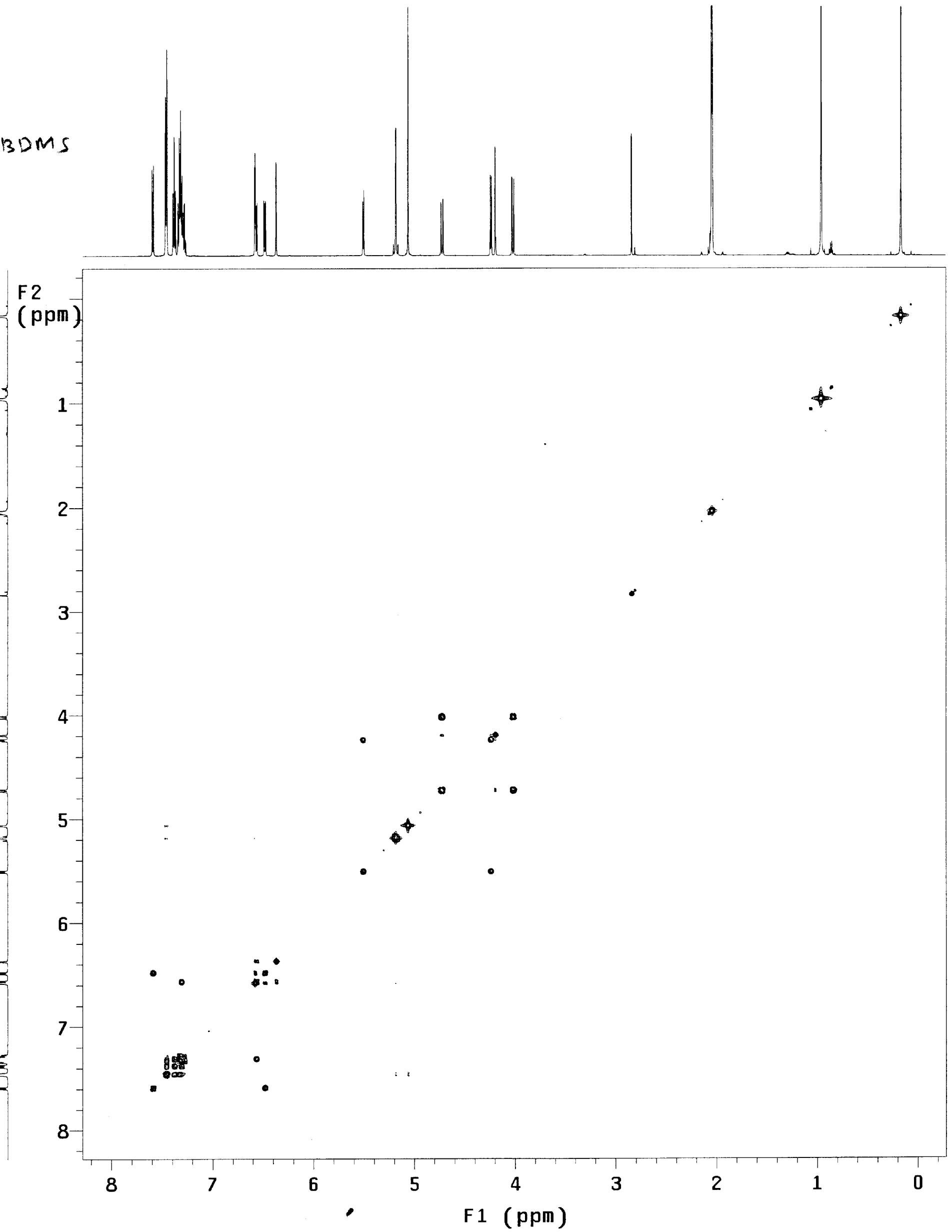
Pulse Sequence: gcosy

Solvent: Acetone
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.199 sec
Width 5141.4 Hz
2D Width 5141.4 Hz
Single scan
512 increments
OBSERVE H1, 599.8754282 MHz
DATA PROCESSING
Sine bell 0.100 sec
F1 DATA PROCESSING
Sine bell 0.050 sec
FT size 2048 x 2048
Total time 10 min, 48 sec




11

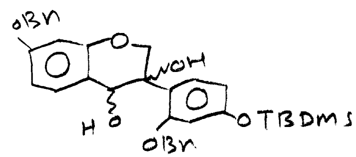


13C OBSERVE

Pulse Sequence: s2pul

Solvent: Acetone
Ambient temperature
File: nbdiolc13
UNITY-400 "vxrs400"

Relax. delay 4.000 sec
Pulse 29.2 degrees
Acq. time 1.205 sec
Width 27192.4 Hz
2048 repetitions
OBSERVE C13, 100.5675467 MHz
DECOUPLE H1, 399.9531799 MHz
Low power 1 dB atten.
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
Gauss window 0.500 sec
center at 0.100 sec
FT size 131072
Total time 2 hr, 58 min, 2 sec




11

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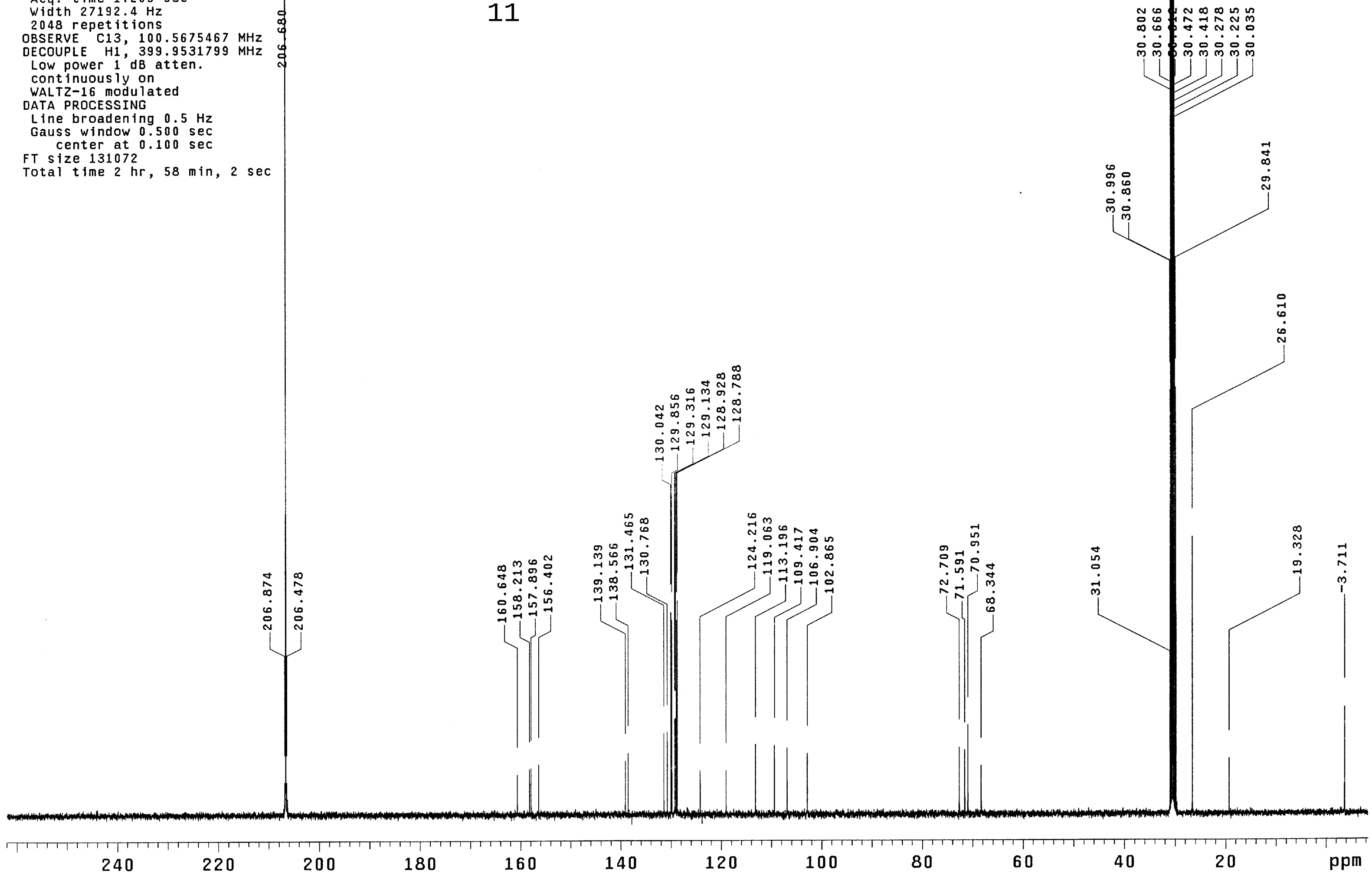
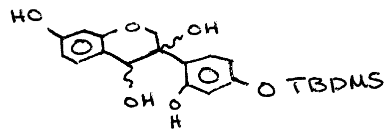
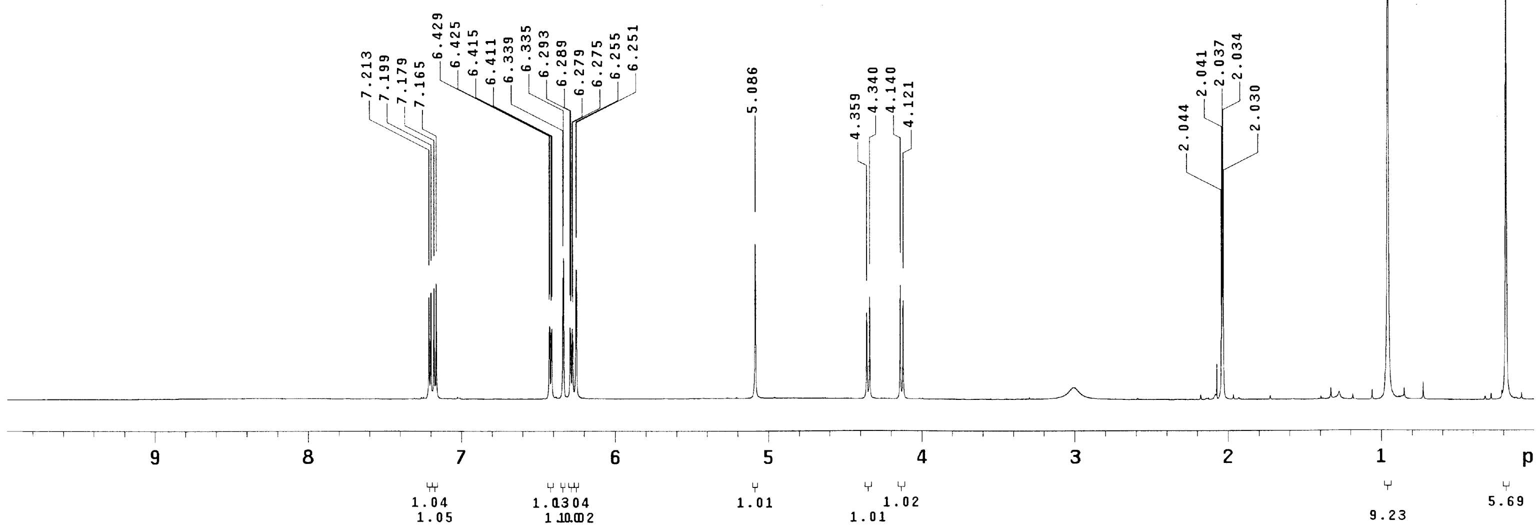


Pulse Sequence: presat
Solvent: Acetone
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8754282 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 39 sec



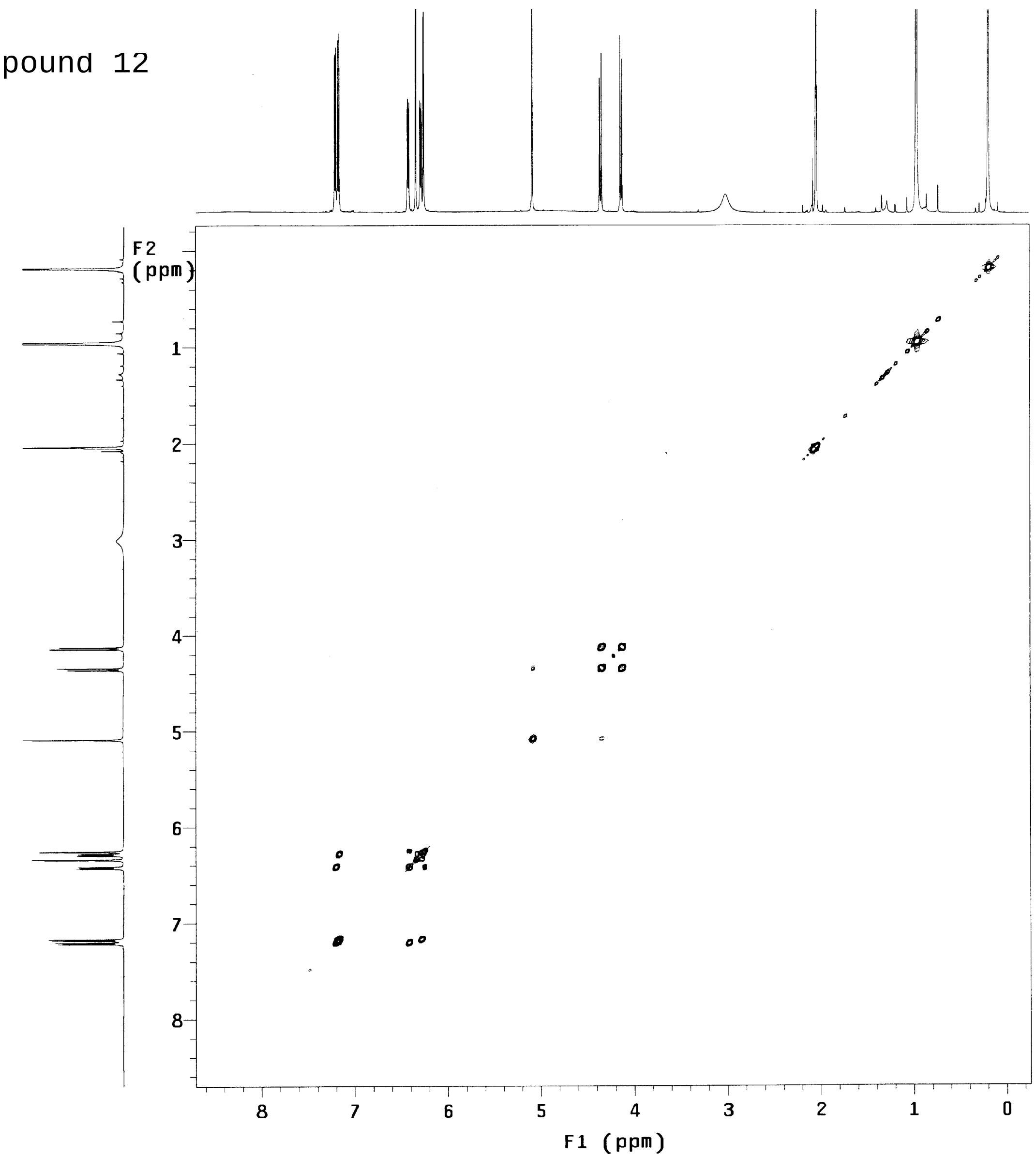

12



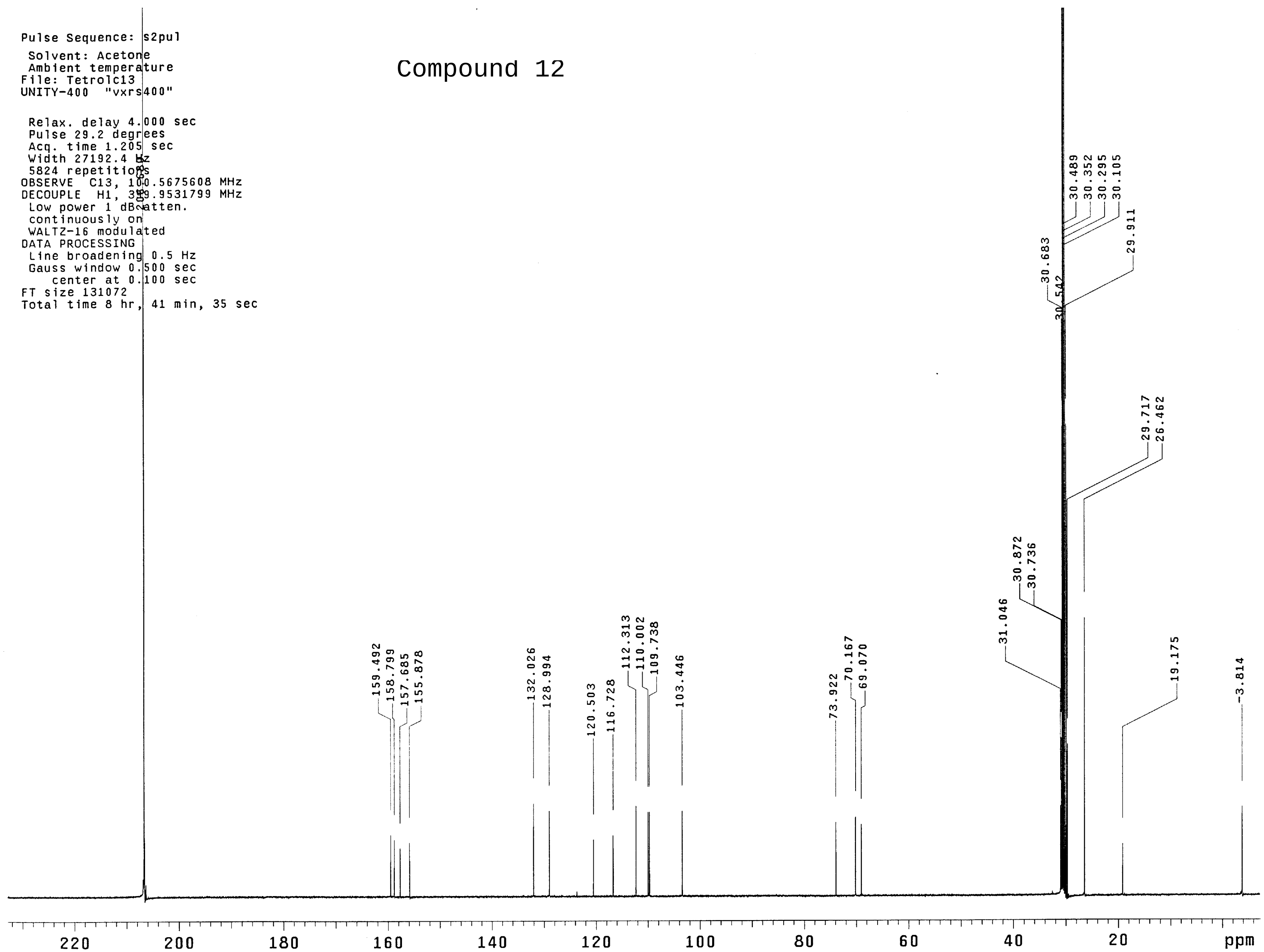
```
Pulse Sequence: gcosy
 Solvent: Acetone
 Ambient temperature
INOVA-600  "inova600"

 Relax. delay 1.000 sec
 Acq. time 0.190 sec
 Width     5379.6 Hz
 2D Width 5379.6 Hz
 Single scan
 512 increments
OBSERVE   H1, 599.8754282 MHz
DATA PROCESSING
 Sine bell 0.095 sec
F1 DATA PROCESSING
 Sine bell 0.048 sec
FT size 2048 x 2048
Total time 10 min, 43 sec
```

Compound 12



Compound 12


Pulse Sequence: s2pul
Solvent: Acetone
Ambient temperature
File: Tetrolc13
UNITY-400 "vxrs400"
Relax. delay 4.000 sec
Pulse 29.2 degrees
Acq. time 1.205 sec
Width 27192.4 Hz
5824 repetitions
OBSERVE C13, 100.5675608 MHz
DECOUPLE H1, 399.9531799 MHz
Low power 1 dB atten.
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
Gauss window 0.500 sec
center at 0.100 sec
FT size 131072
Total time 8 hr, 41 min, 35 sec
159.492
158.799
157.685
155.878
132.026
128.994
120.503
116.728
112.313
110.002
109.738
103.446
73.922
70.167
69.070
31.046
30.872
30.736
30.683
30.542
30.489
30.352
30.295
30.105
29.911
29.717
26.462
19.175
-3.814
220
200
180
160
140
120
100
80
60
40
20
ppm

GH01 1

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: Acetone
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8754282 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec

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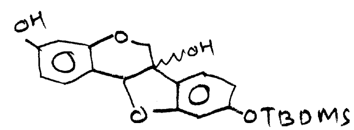
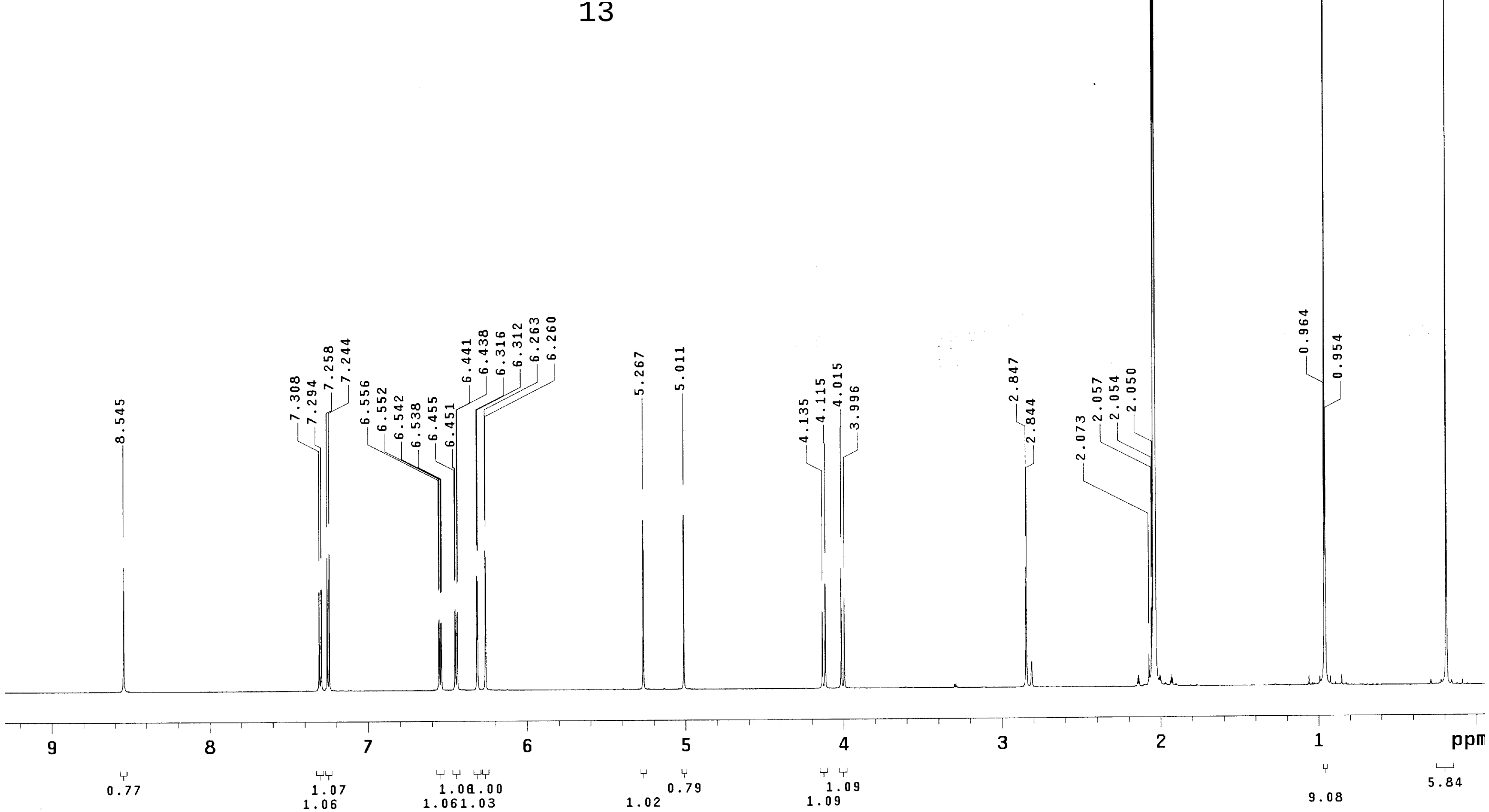



13

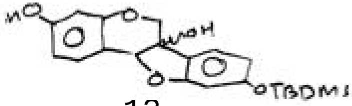


STANDARD PROTON PARAMETERS

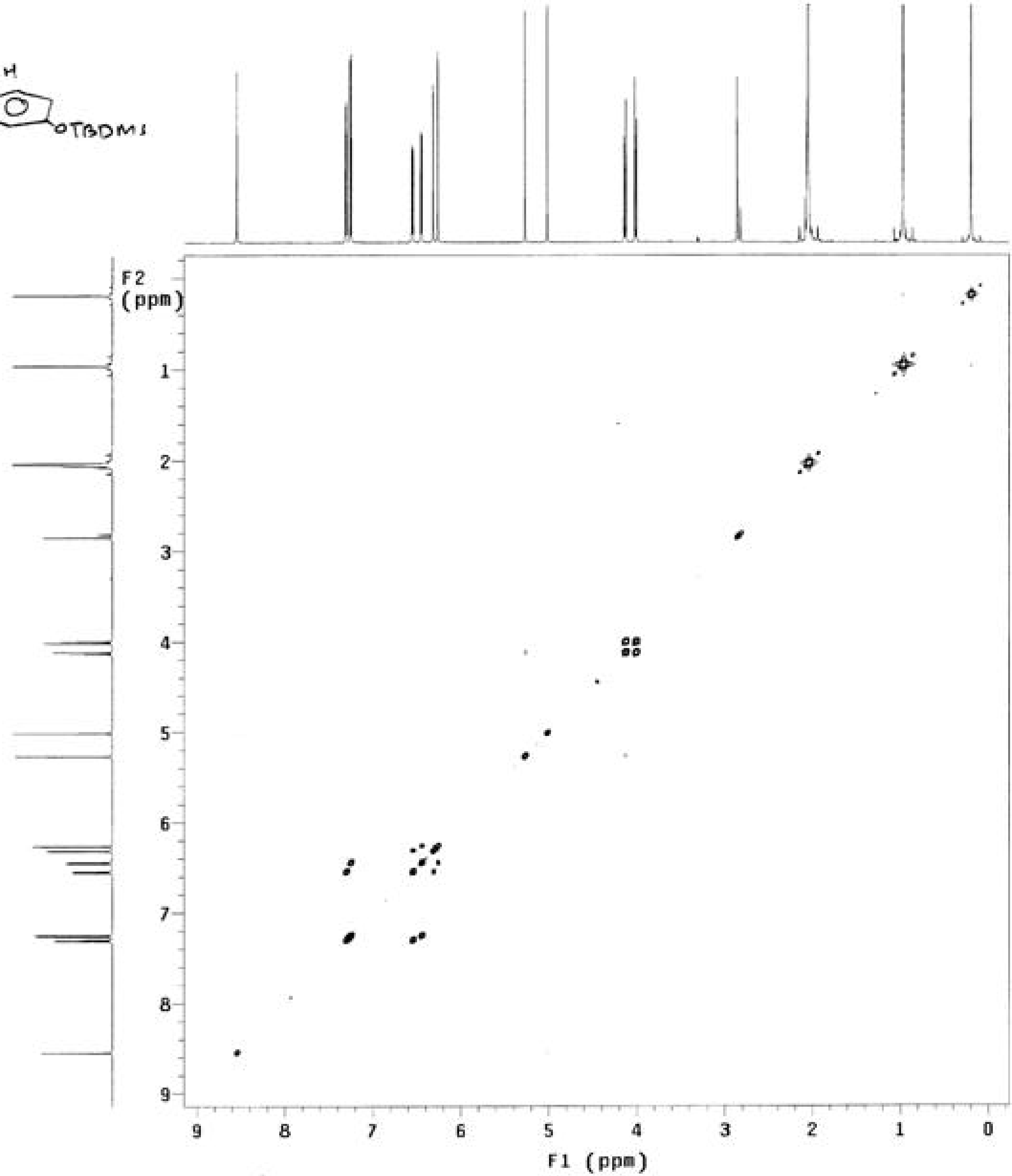
Pulse Sequence: gcosy

Solvent: Acetone
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.182 sec
Width 5637.8 Hz
2D Width 5637.8 Hz
Single scan
512 increments
OBSERVE H1, 599.8754282 MHz
DATA PROCESSING
Sine bell 0.091 sec
F1 DATA PROCESSING
Sine bell 0.045 sec
FT size 2048 x 2048
Total time 10 min, 37 sec




13



13C OBSERVE

Pulse Sequence: s2pul
Solvent: Acetone
Ambient temperature
File: GnolTc13
UNITY-400 "vxrs400"

Relax. delay 4.000 sec
Pulse 29.2 degrees
Acq. time 1.205 sec
Width 27192.4 Hz
7000 repetitions
OBSERVE C13, 100.5675455 MHz
DECOUPLE H1, 399.9531799 MHz
Low power 1 dB atten.
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
Gauss window 0.500 sec
center at 0.100 sec
FT size 131072
Total time 10 hr, 8 min, 31 sec

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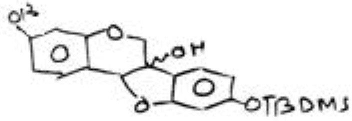
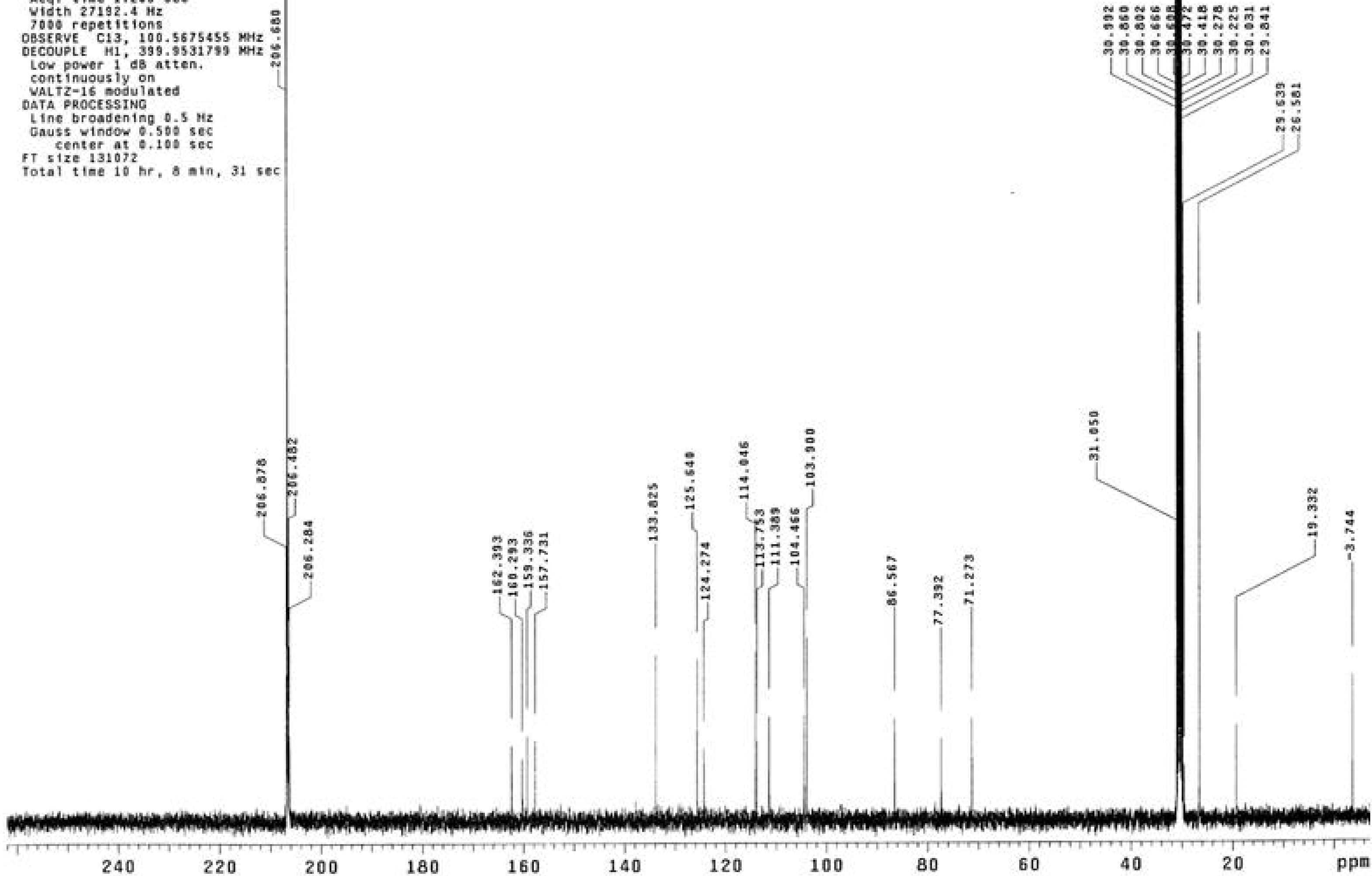



13



STANDARD PROTON PARAMETERS

Pulse Sequence: presat
Solvent: Acetone
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
64 repetitions
OBSERVE H1, 599.8754282 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 6 min, 3 sec

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CD3 22
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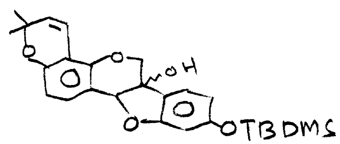
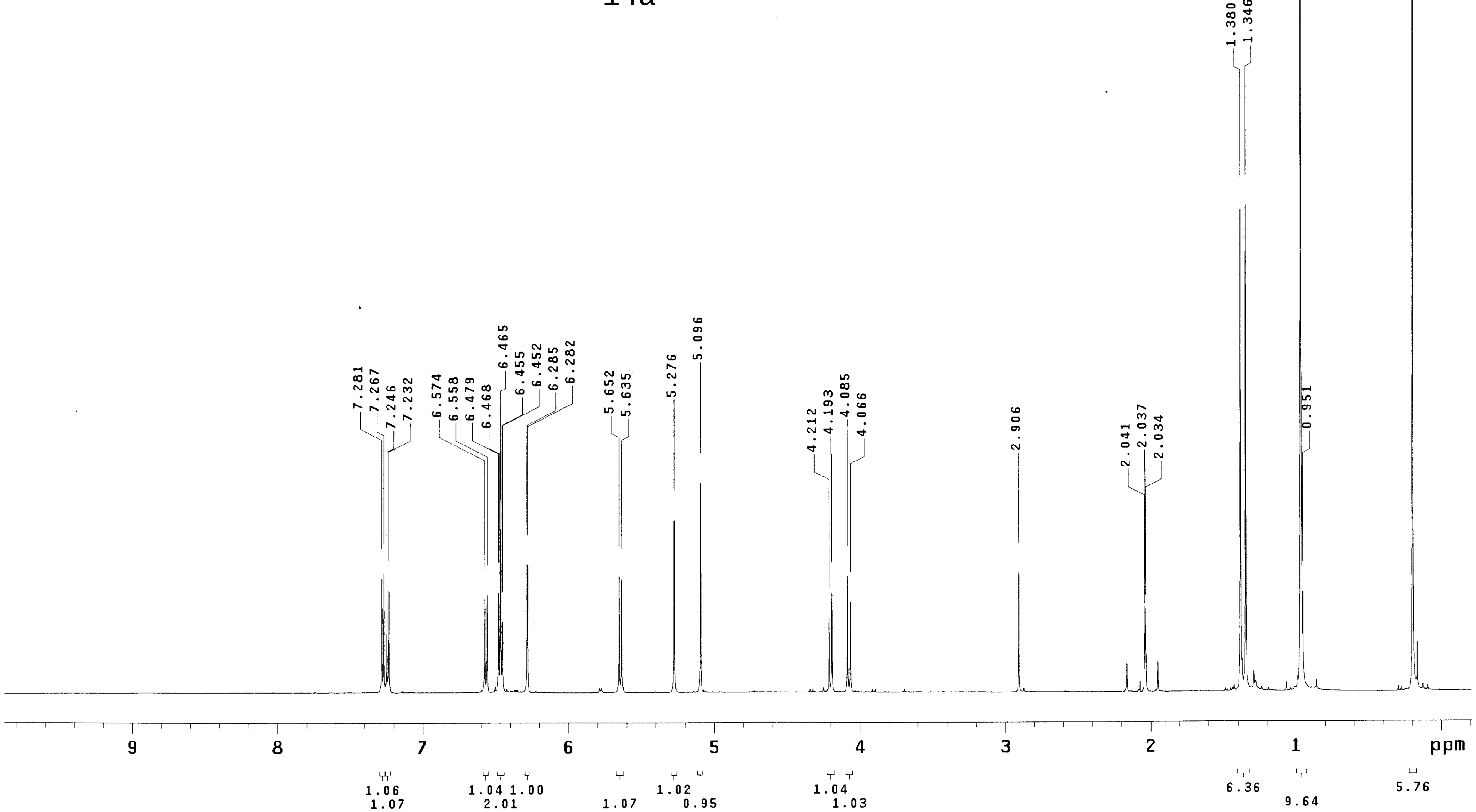



14a

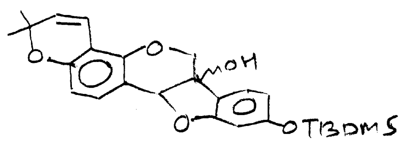


STANDARD PROTON PARAMETERS

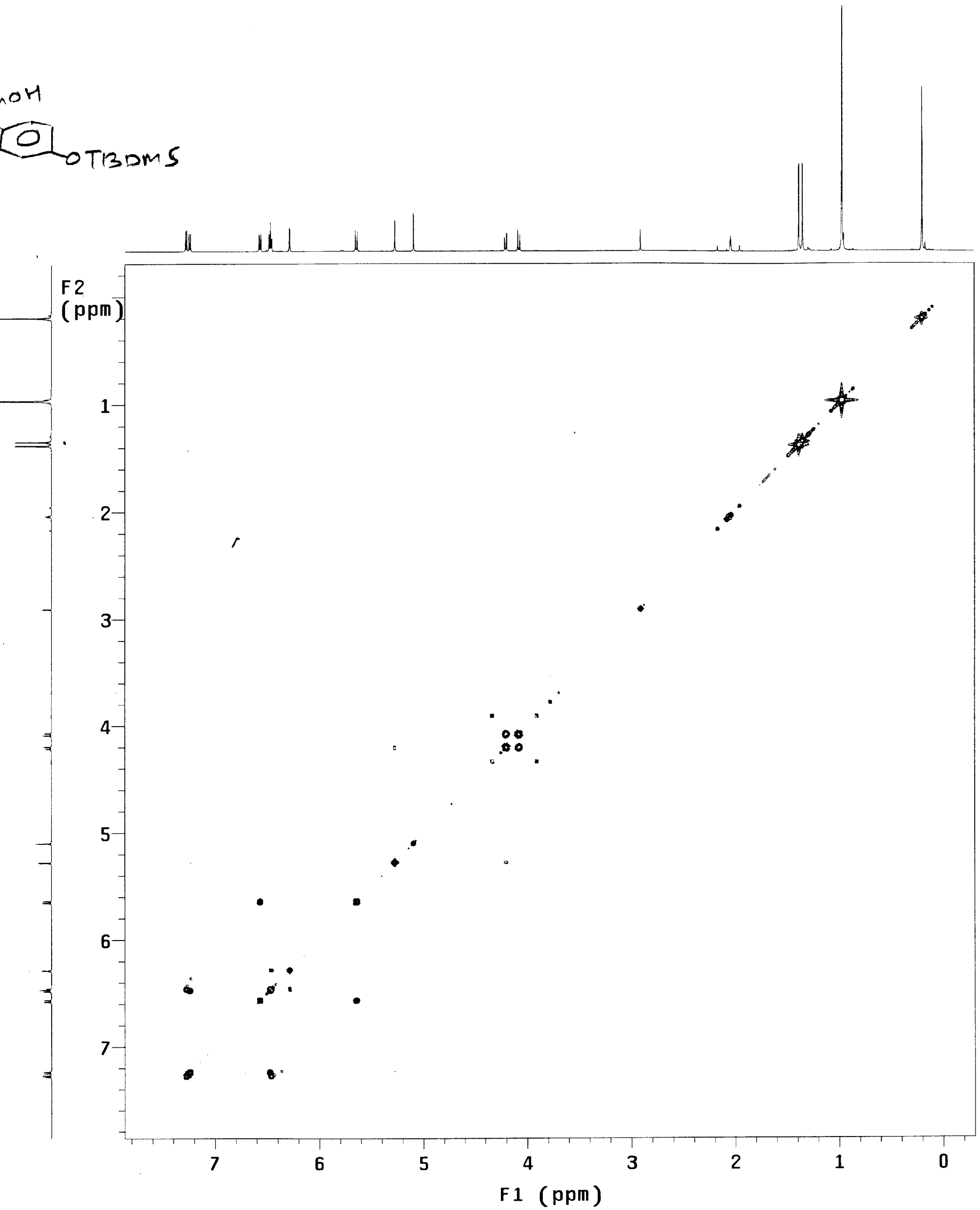
Pulse Sequence: gcosy

Solvent: Acetone
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.209 sec
Width 4903.2 Hz
2D Width 4903.2 Hz
Single scan
512 increments
OBSERVE H1, 599.8754282 MHz
DATA PROCESSING
Sine bell 0.104 sec
F1 DATA PROCESSING
Sine bell 0.052 sec
FT size 2048 x 2048
Total time 10 min, 55 sec




14a

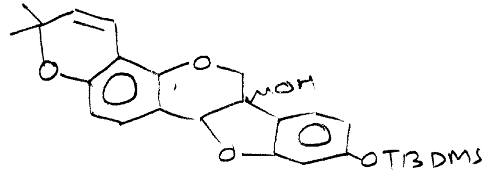


13C OBSERVE

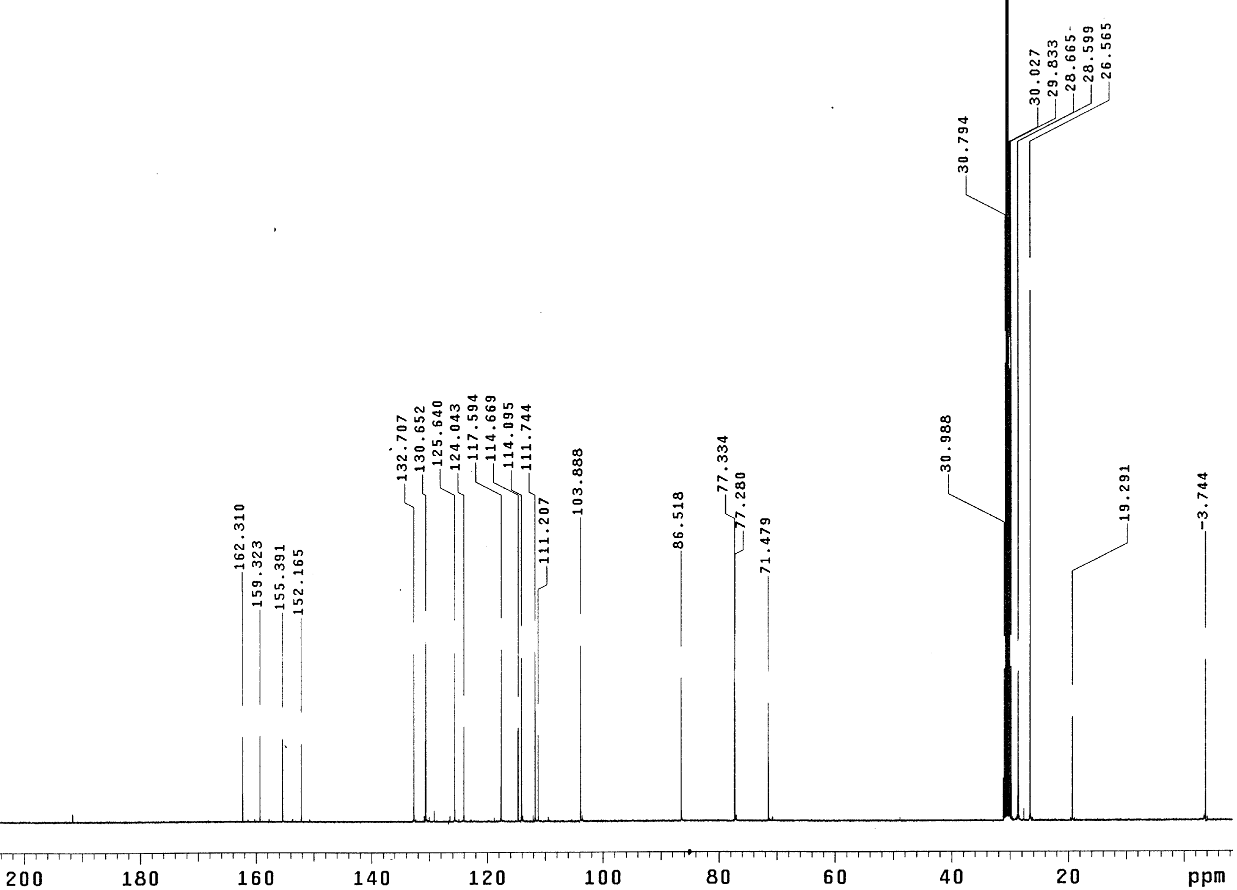
May 2 2006

Pulse Sequence: s2pul
Solvent: Acetone
Ambient temperature
UNITY-400 "vxrs400"

Relax. delay 4.000 sec
Pulse 29.2 degrees
Acq. time 1.205 sec
Width 27192.4 Hz
7904 repetitions
OBSERVE C13, 100.5675525 MHz
DECOUPLE H1, 399.9531799 MHz
Low power 1 dB atten.
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
Gauss window 0.500 sec
center at 0.100 sec
FT size 131072
Total time 11 hr, 35 min, 27 sec




14a



STANDARD PROTON PARAMETERS

Pulse Sequence: presat
Solvent: CD3OD
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
64 repetitions
OBSERVE H1, 599.8746729 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 6 min, 3 sec

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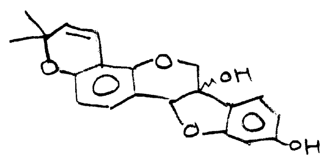


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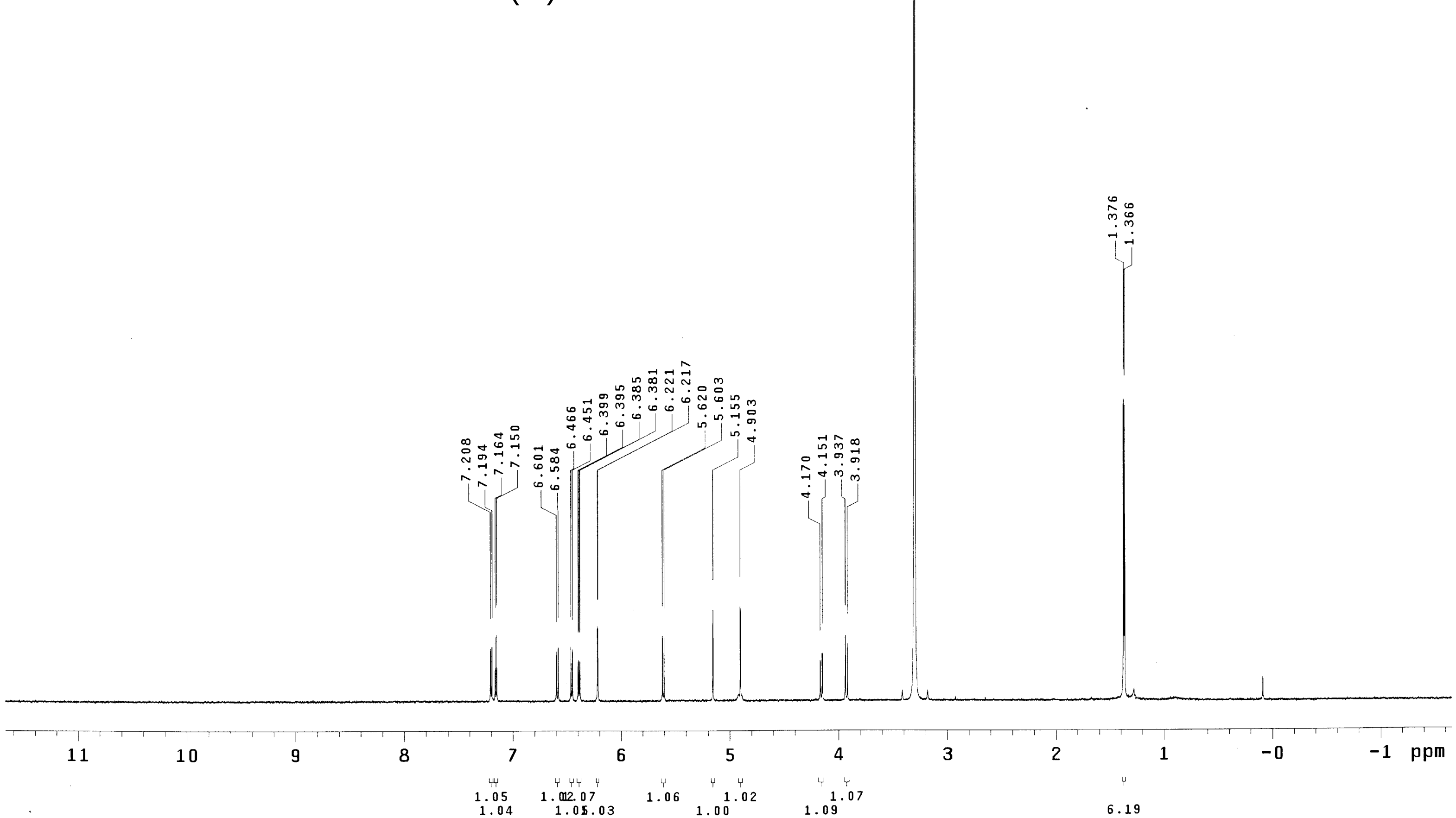
Apr 23 2006

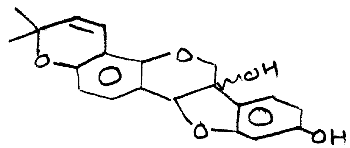


STANDARD PROTON PARAMETERS

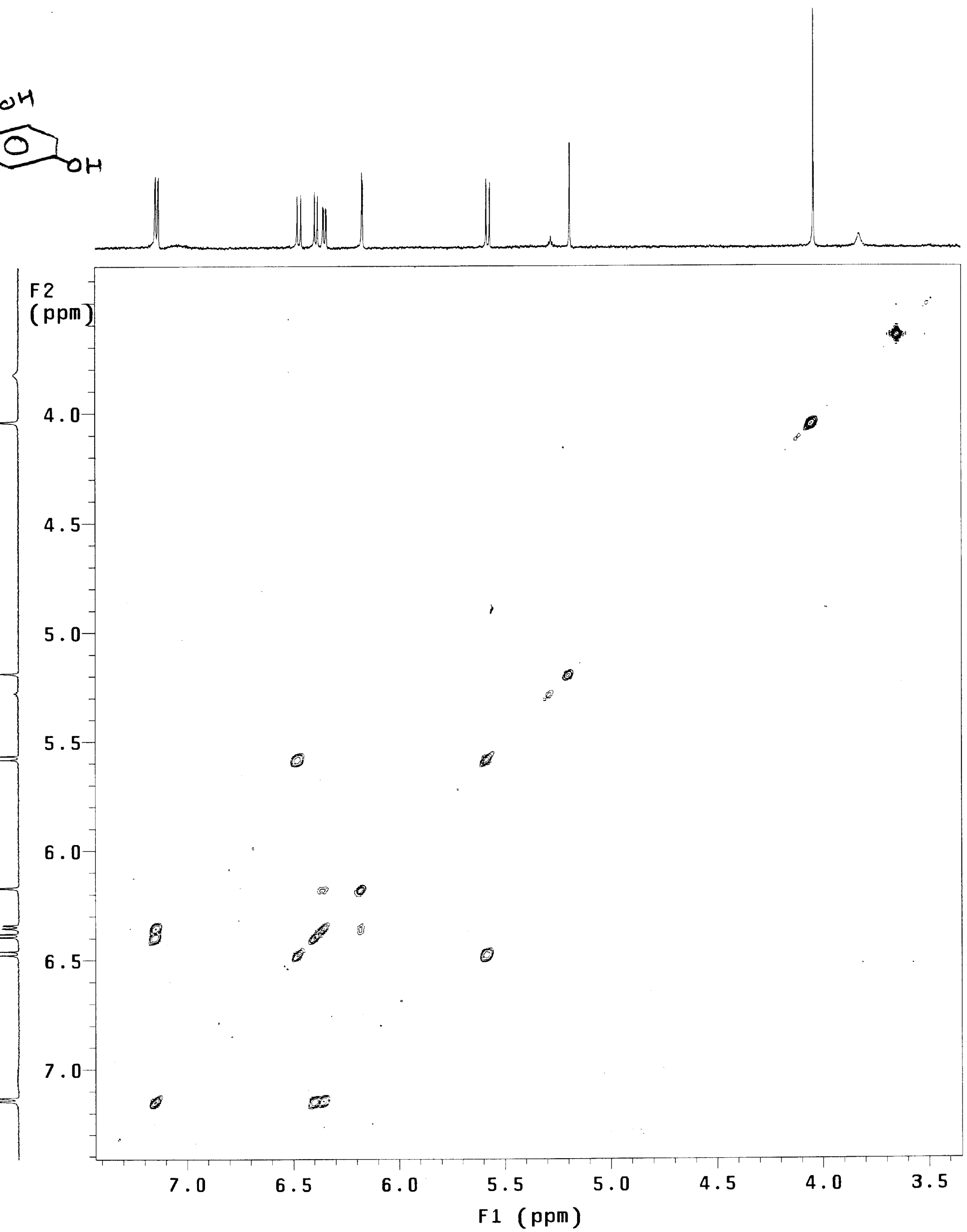
Pulse Sequence: gcosy

Solvent: CD3CN
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.225 sec
Width 4545.7 Hz
2D Width 4545.7 Hz
Single scan
512 increments
OBSERVE H1, 599.8755390 MHz
DATA PROCESSING
Sine bell 0.113 sec
F1 DATA PROCESSING
Sine bell 0.056 sec
FT size 2048 x 2048
Total time 11 min, 5 sec



(±)15



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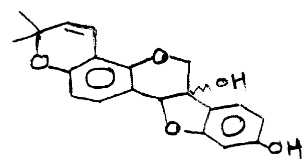
STANDARD CARBON PARAMETERS

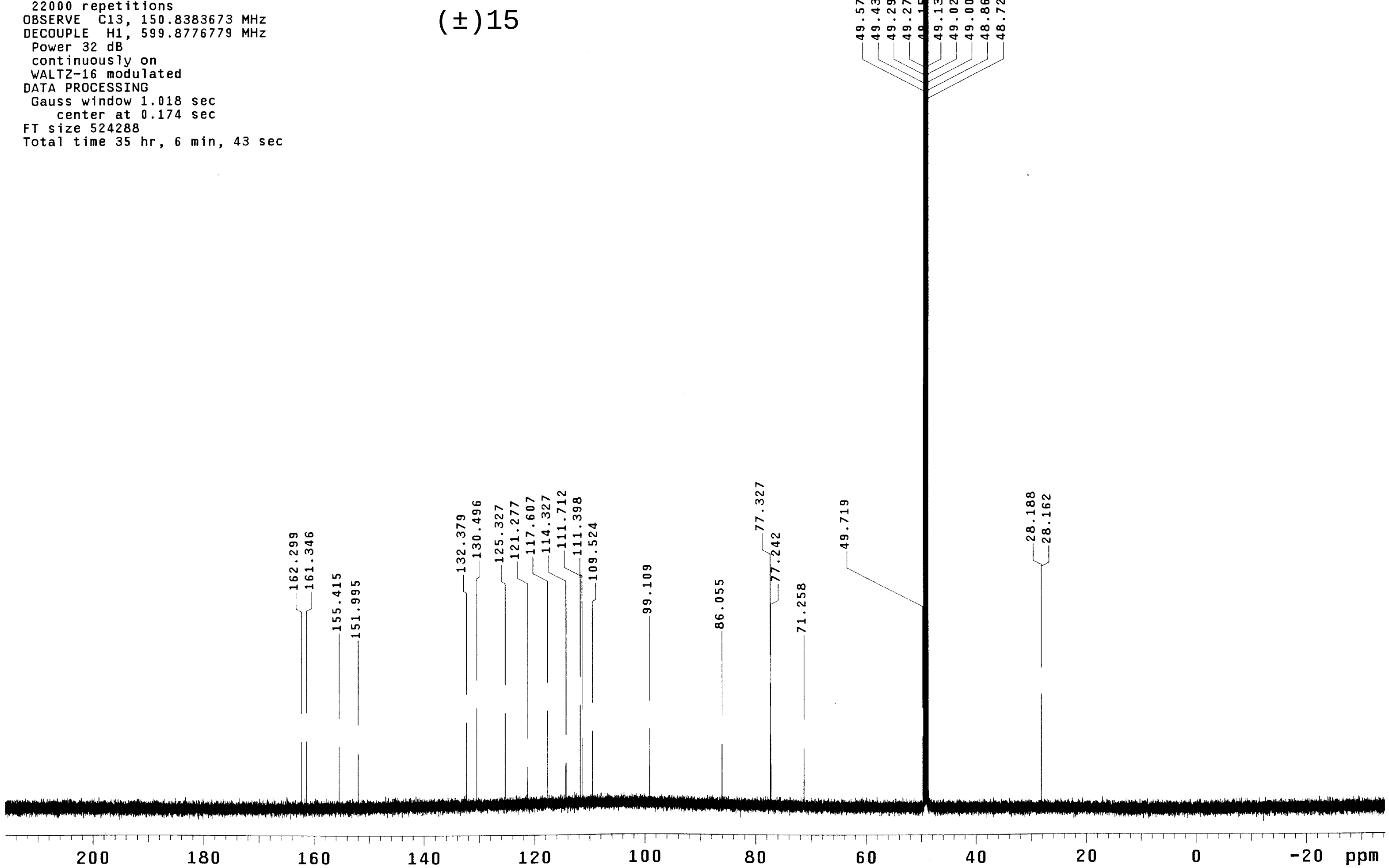
Pulse Sequence: s2pul
Solvent: CD3OD
Ambient temperature
User: 1-14-87
File: G1C13
INOVA-600 "inova600"

Relax. delay 4.000 sec
Pulse 33.7 degrees
Acq. time 1.737 sec
Width 37735.8 Hz
22000 repetitions
OBSERVE C13, 150.8383673 MHz
DECOUPLE H1, 599.8776779 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 1.018 sec
center at 0.174 sec
FT size 524288
Total time 35 hr, 6 min, 43 sec




(±)15

May 5 2006

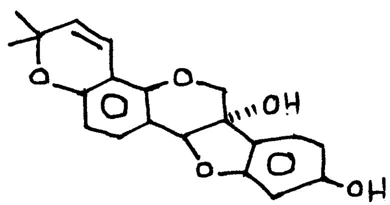


STANDARD PROTON PARAMETERS

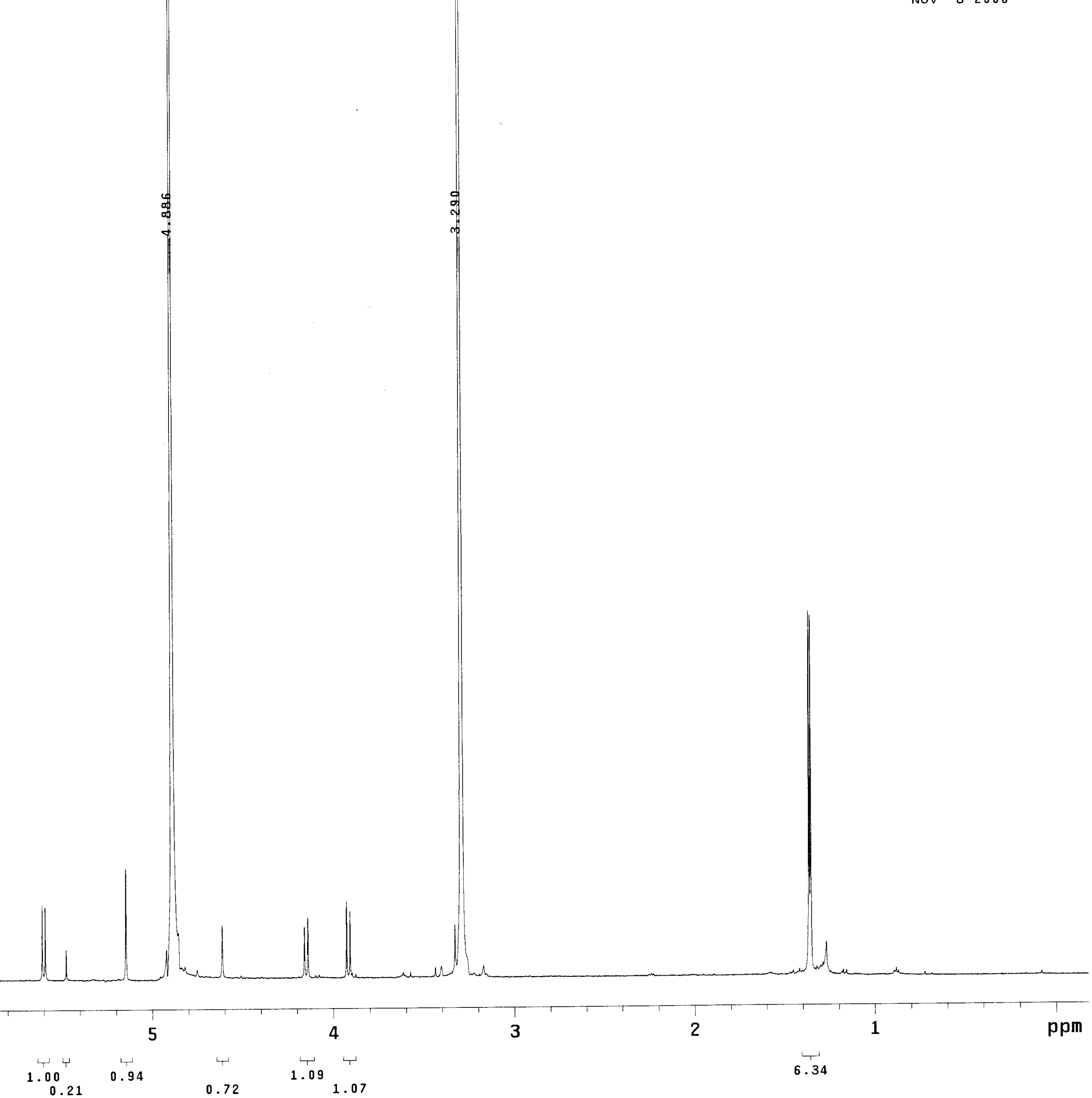
Pulse Sequence: s2pul

Solvent: CD3OD
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 31.3 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8746783 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec




15



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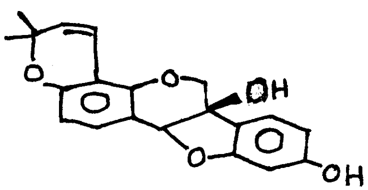
STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: CD3OD
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 31.3 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8746723 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec




(+)15