Total Synthesis of Xanthohumol

Rahul S. Khupse[†] and Paul W. Erhardt*

Center for Drug Design and Development
The University of Toledo College of Pharmacy
Toledo, Ohio 43606-3390

*Telephone: (419) 530-2167
Fax: (419) 530-1994
Email: paul.erhardt@utoledo.edu

[†] Present address: Lexington Pharmaceutical Labs, LLC
Lexington, Kentucky 40511

Supplementary Information

S1 Title

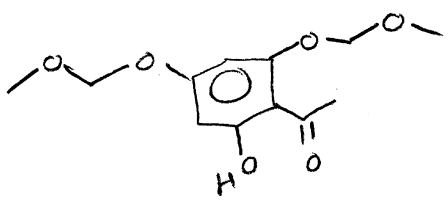
S2 NMR Spectra (^{1}H, COSY, ^{13}C)

S 2

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 30.9 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8723053 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



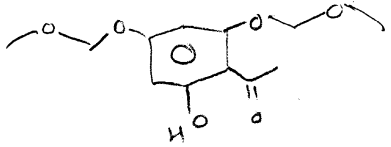
4

RKX(1)

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
480 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



4

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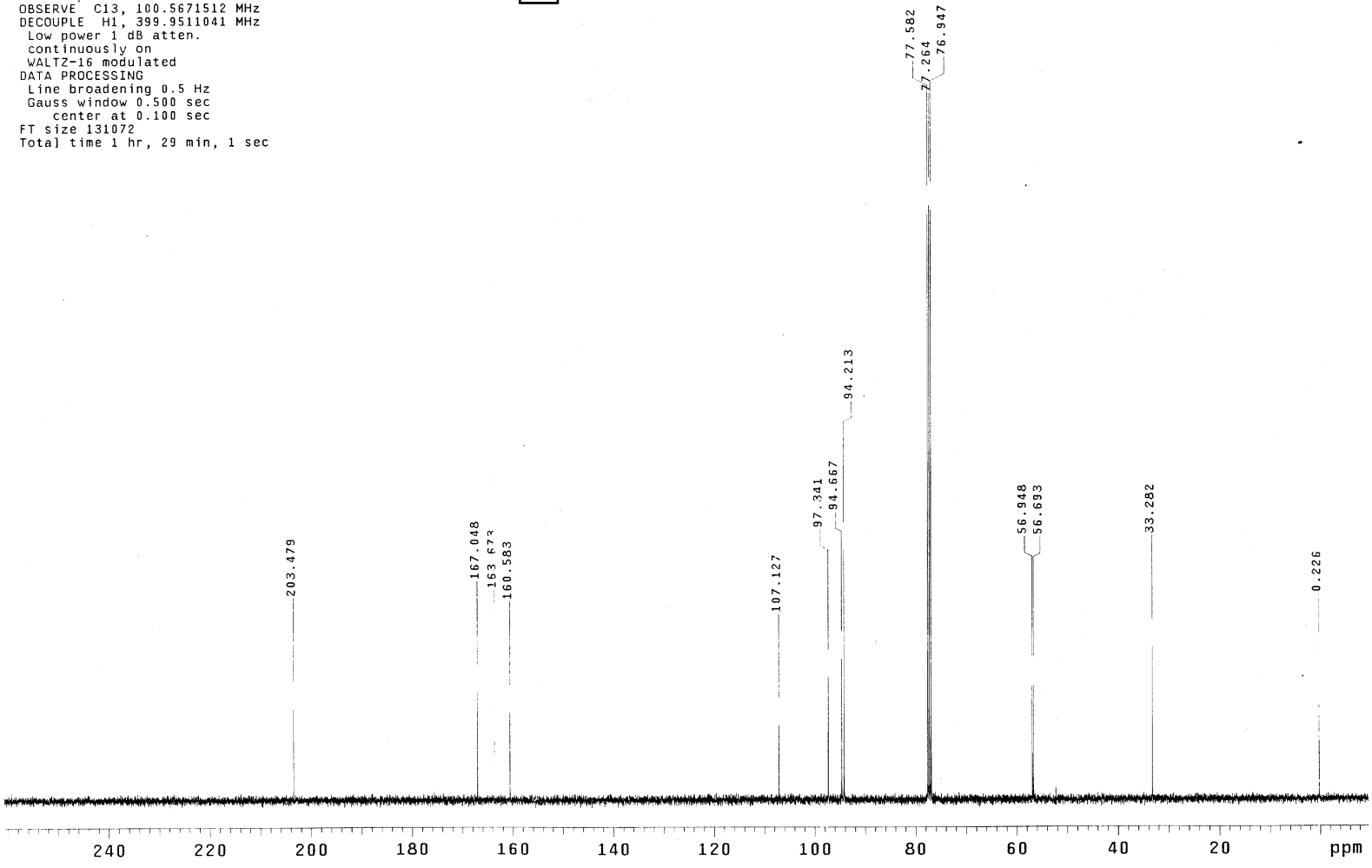
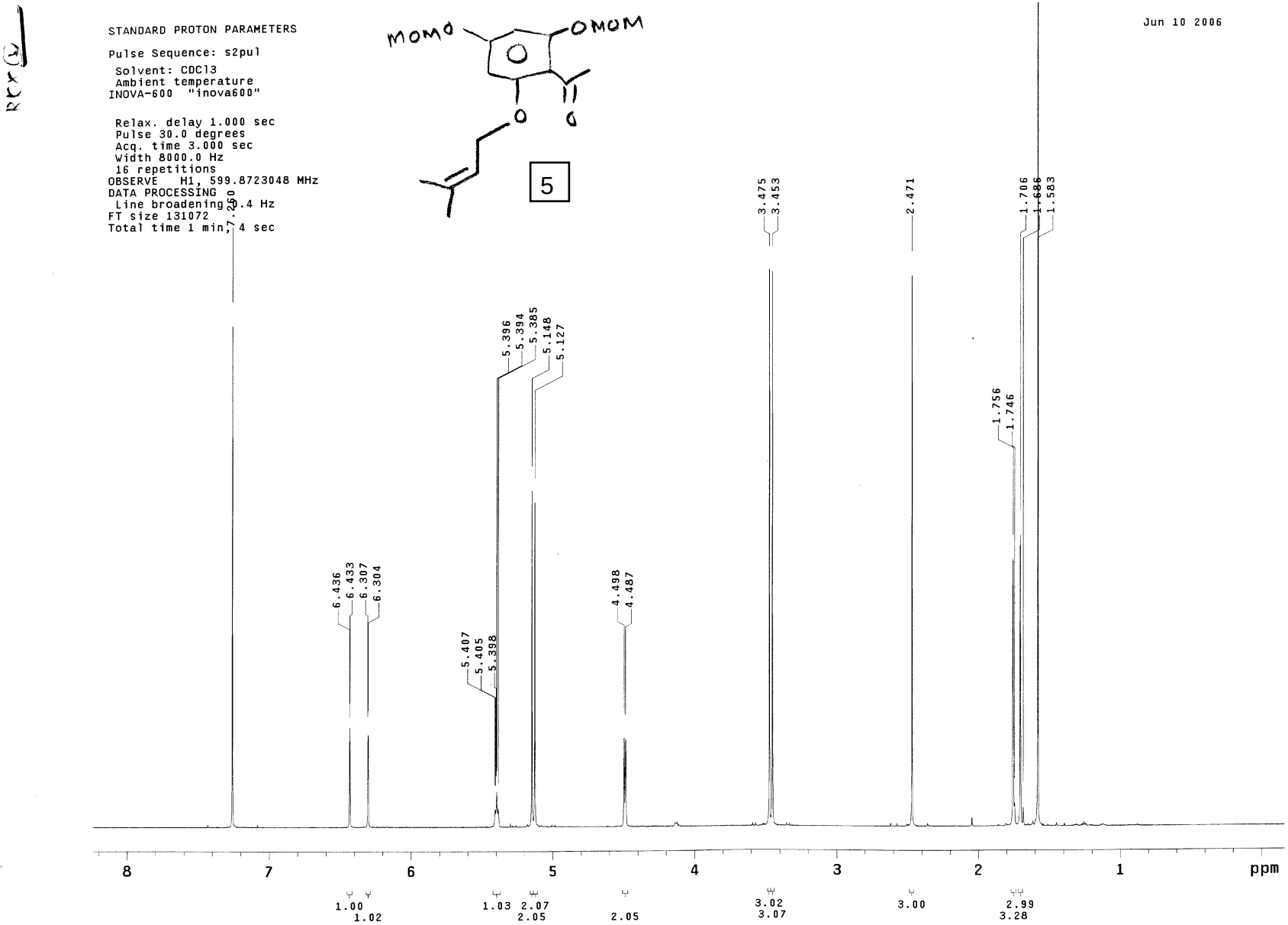
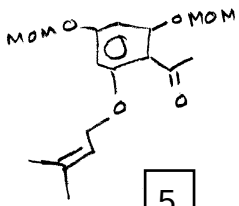



STANDARD PROTON PARAMETERS
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-600 "inova600"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8723048 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec
Jun 10 2006
MOMO
OMOM
5
7.260
6.436
6.433
6.307
6.304
5.407
5.405
5.398
5.396
5.394
5.385
5.148
5.127
4.498
4.487
3.475
3.453
2.471
1.756
1.746
1.706
1.686
1.583
8
7
6
5
4
3
2
1
ppm
1.00
1.02
1.03
2.07
2.05
2.05
3.02
3.07
3.00
2.99
3.28

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul
Solvent: CDCl3
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
912 repetitions
OBSERVE C13, 150.8379775 MHz
DECOUPLE H1, 599.8753144 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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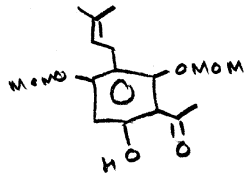
RKX(4)

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul
Solvent: CDCl3
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, 599.8723066 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



6

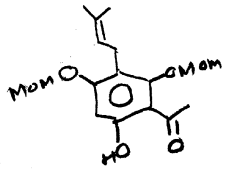
Sep 7 2006

RKX④

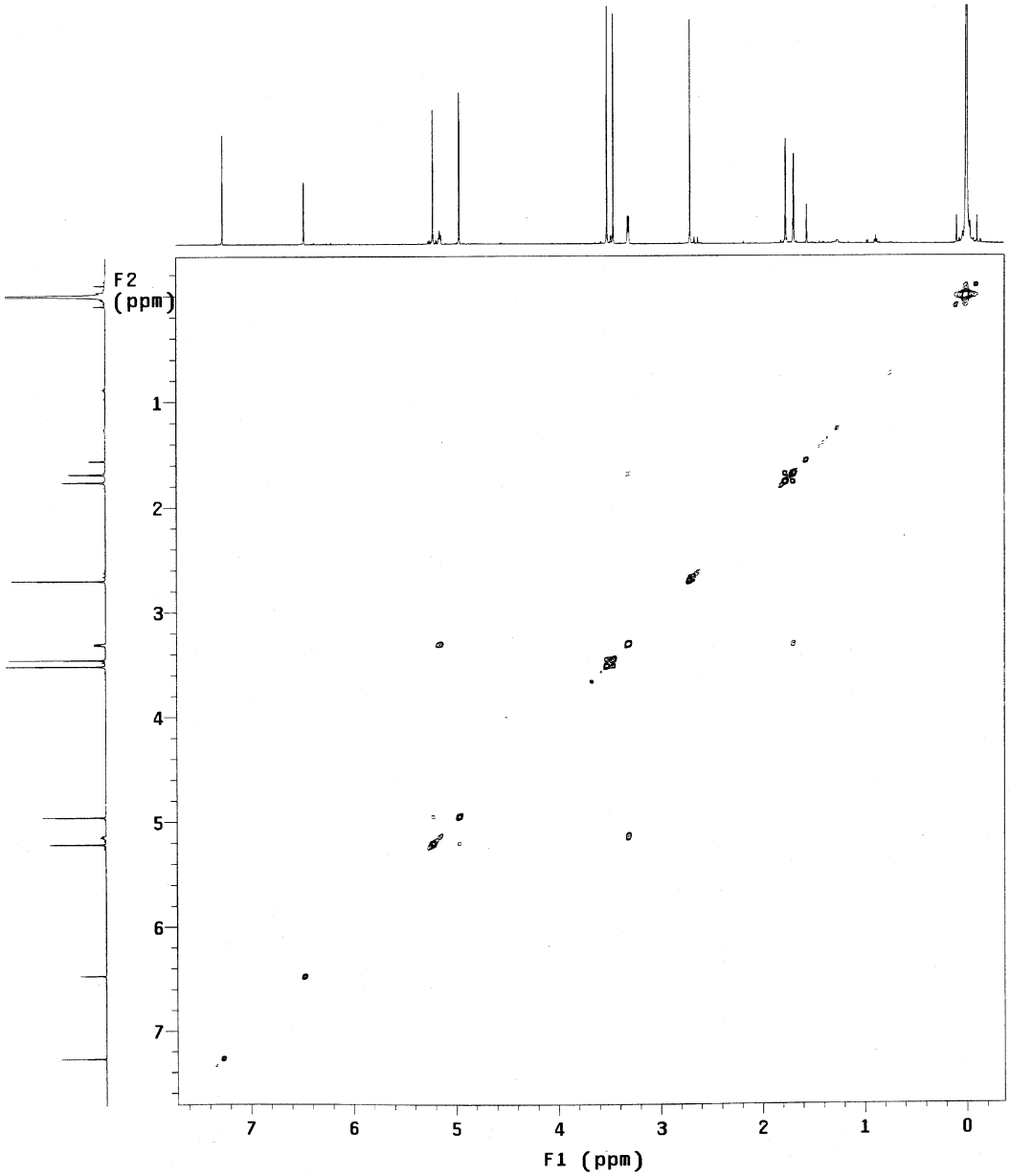
STANDARD PROTON PARAMETERS

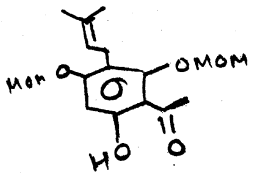
Pulse Sequence: gcosy
Solvent: CDCl3
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.211 sec
Width 4850.2 Hz
2D Width 4850.2 Hz
Single scan
512 increments
OBSERVE H1, 599.8723066 MHz
DATA PROCESSING
Sine bell 0.106 sec
F1 DATA PROCESSING
Sine bell 0.053 sec
FT size 2048 x 2048
Total time 10 min, 56 sec



6





6

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul
Solvent: CDCl3
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
224 repetitions
OBSERVE C13, 150.8378775 MHz
DECOUPLE H1, 599.8753144 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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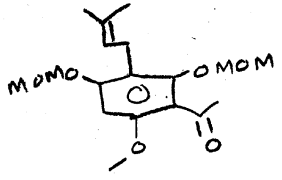
S 9

STANDARD PROTON PARAMETERS

Pulse Sequence: presat
Solvent: CDCl3
Ambient temperature
File: RKX5H1
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, 599.8723064 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 39 sec

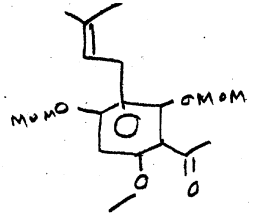


7

STANDARD PROTON PARAMETERS

Pulse Sequence: gcosy
Solvent: CDCl3
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.211 sec
Width 4843.5 Hz
2D Width 4843.5 Hz
Single scan
512 increments
OBSERVE H1, 599.8723061 MHz
DATA PROCESSING
Sine bell 0.106 sec
F1 DATA PROCESSING
Sine bell 0.053 sec
FT size 2048 x 2048
Total time 10 min, 56 sec



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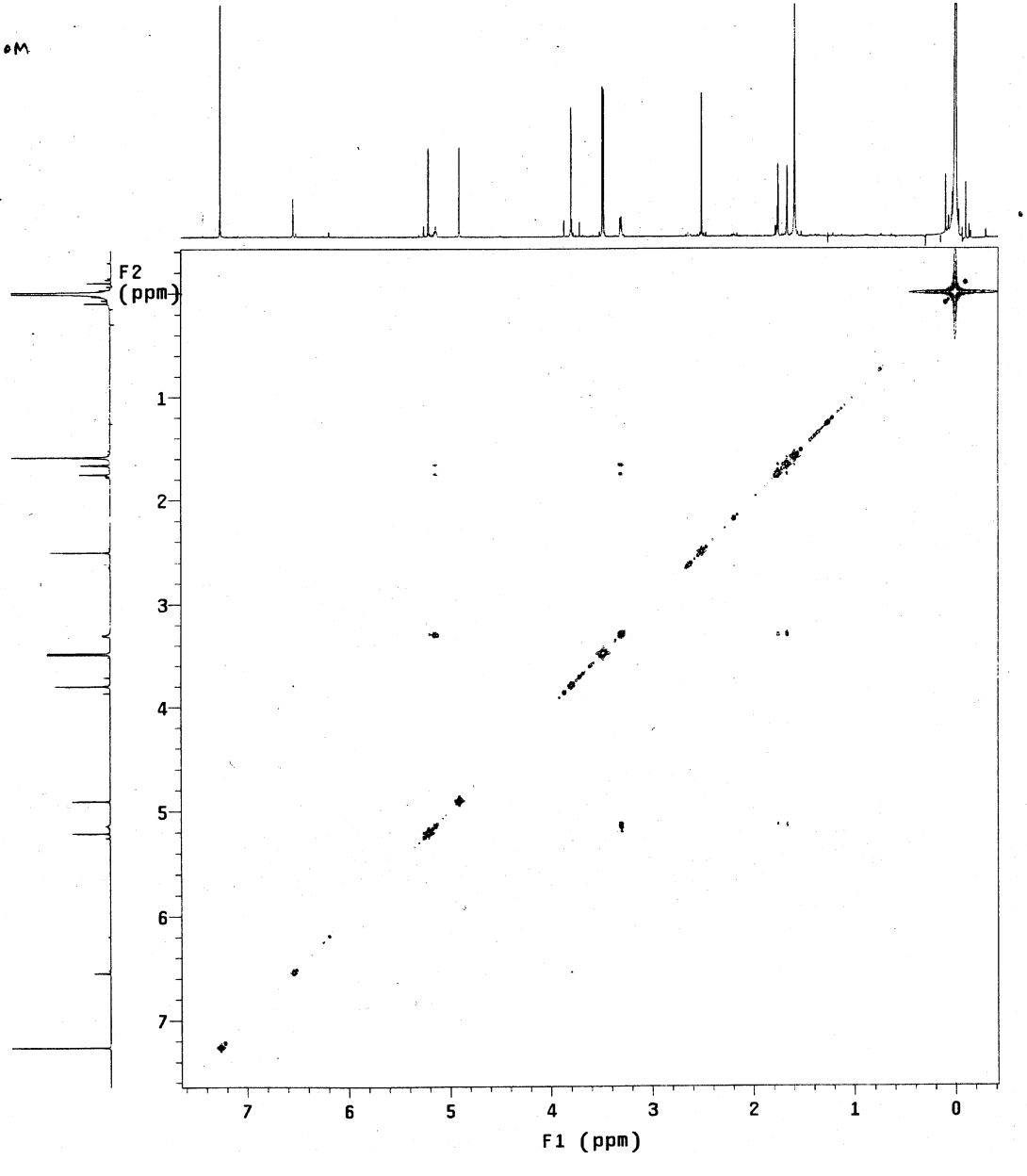
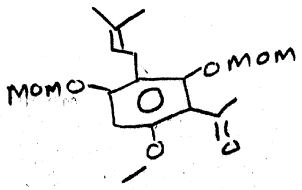


RK-45

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: RKX5c13
INOVA-500 "vxrs-wkst"

Relax. delay 0.100 sec
Pulse 61.4 degrees
Acq. time 1.205 sec
Width 27192.4 Hz
352 repetitions
OBSERVE C13, 100.5671667 MHz
DECOUPLE H1, 399.9511041 MHz
Power 30 dB
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 22 min, 25 sec



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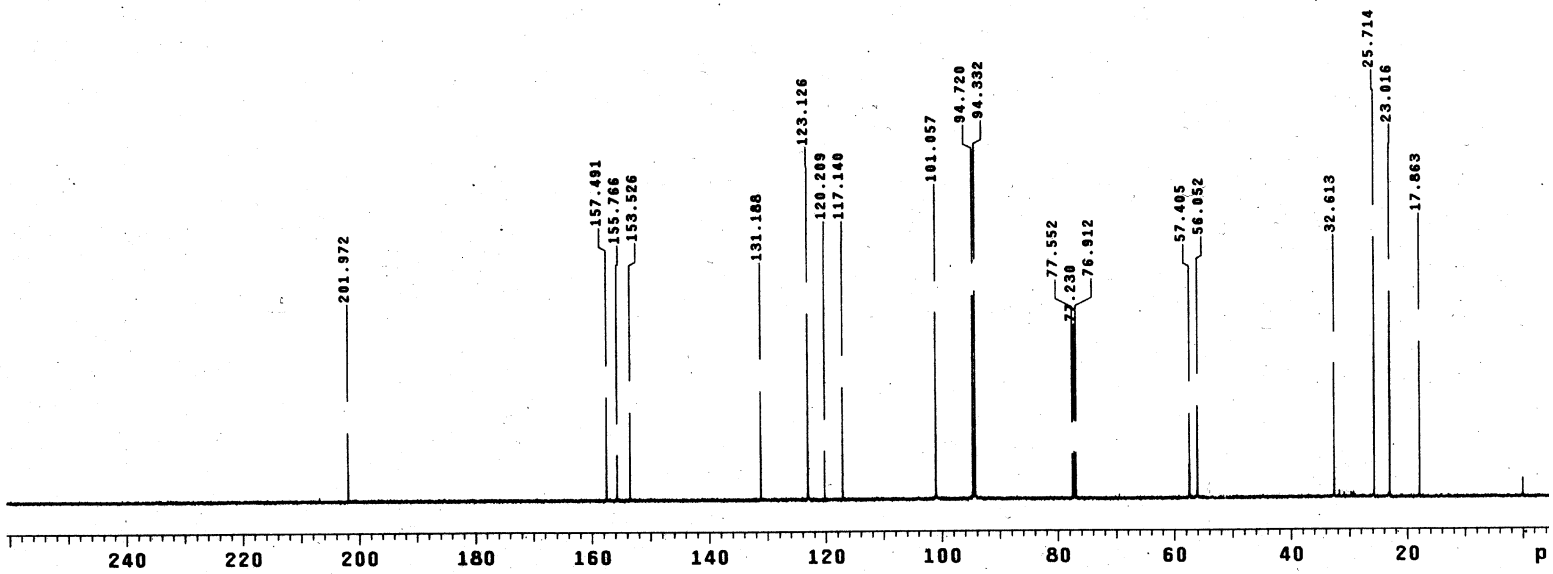
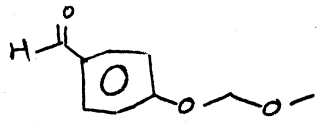


AKX2

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 30.9 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8723050 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



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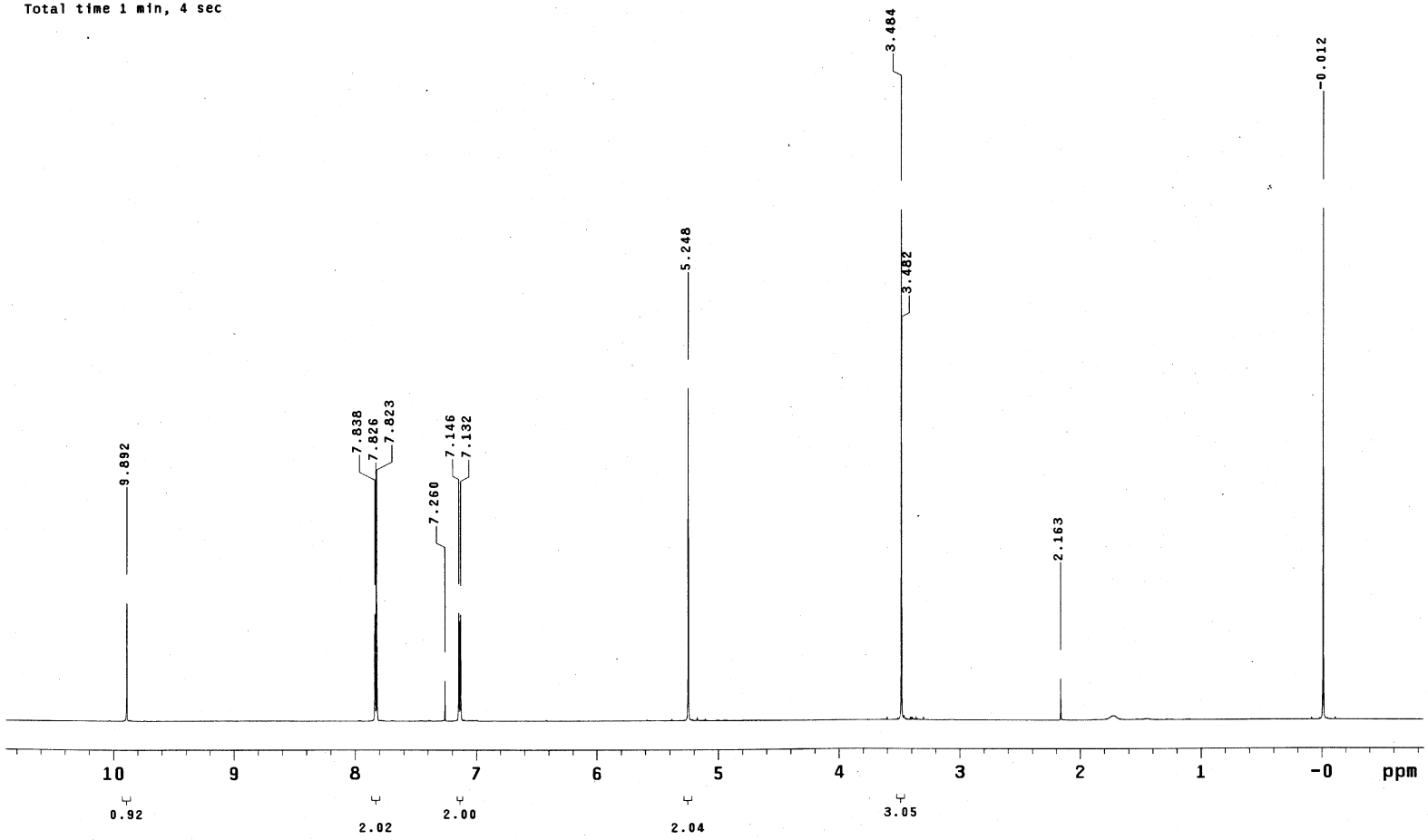

RK-2

13C OBSERVE

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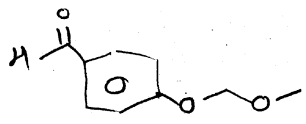
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
UNITY-400 "vxrs400"

Relax. delay 4.000 sec
Pulse 29.2 degrees
Acq. time 1.205 sec
Width 27192.4 Hz
64 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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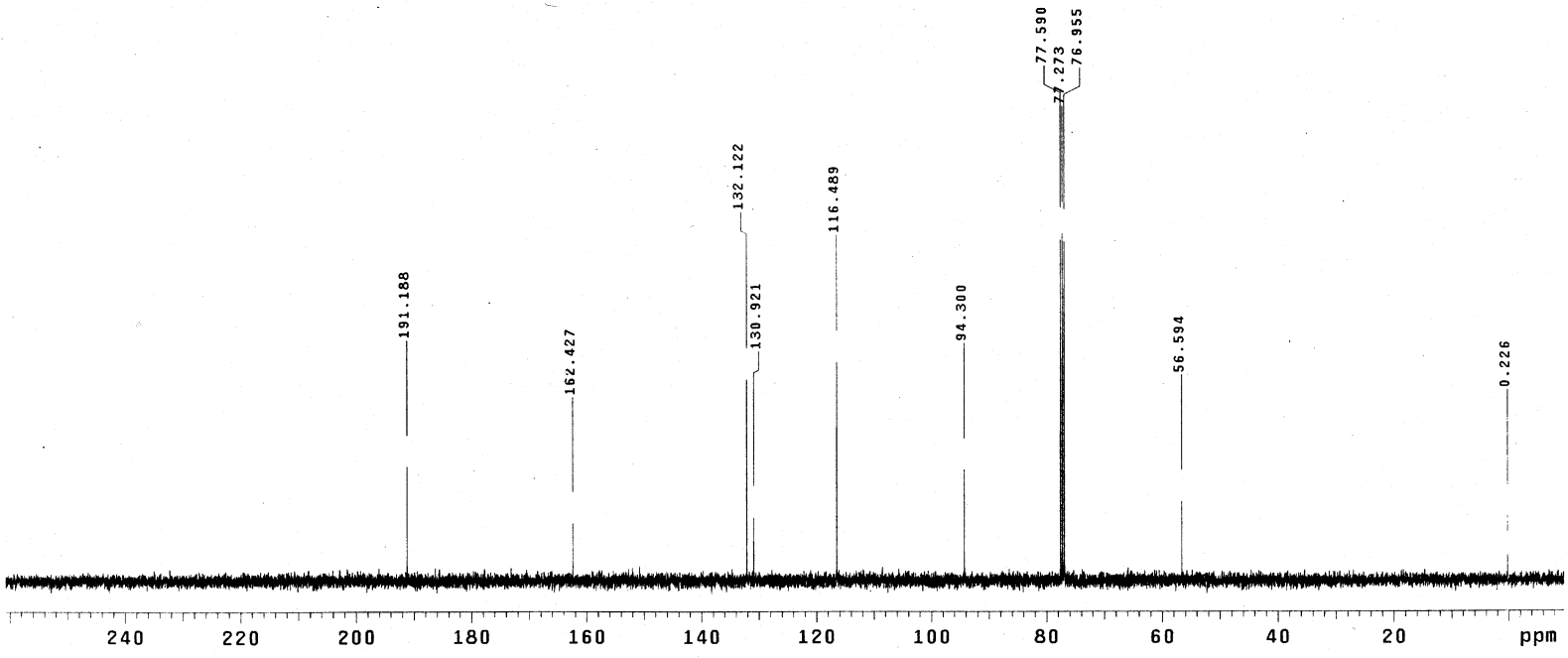
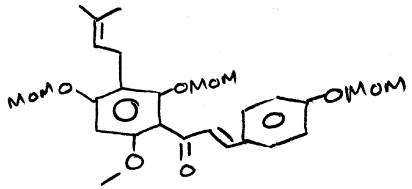


RKY6

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul
Solvent: CDCl3
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, 599.8723066 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



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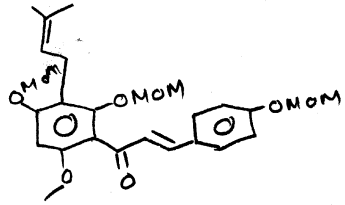
RK X(6)

STANDARD PROTON PARAMETERS

Pulse Sequence: gcosy
Solvent: CDCl3
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.209 sec
Width 4896.6 Hz
2D Width 4896.6 Hz
Single scan
512 increments
OBSERVE H1, 599.8723148 MHz
DATA PROCESSING
Sine bell 0.105 sec
F1 DATA PROCESSING
Sine bell 0.052 sec
FT size 2048 x 2048
Total time 10 min, 55 sec



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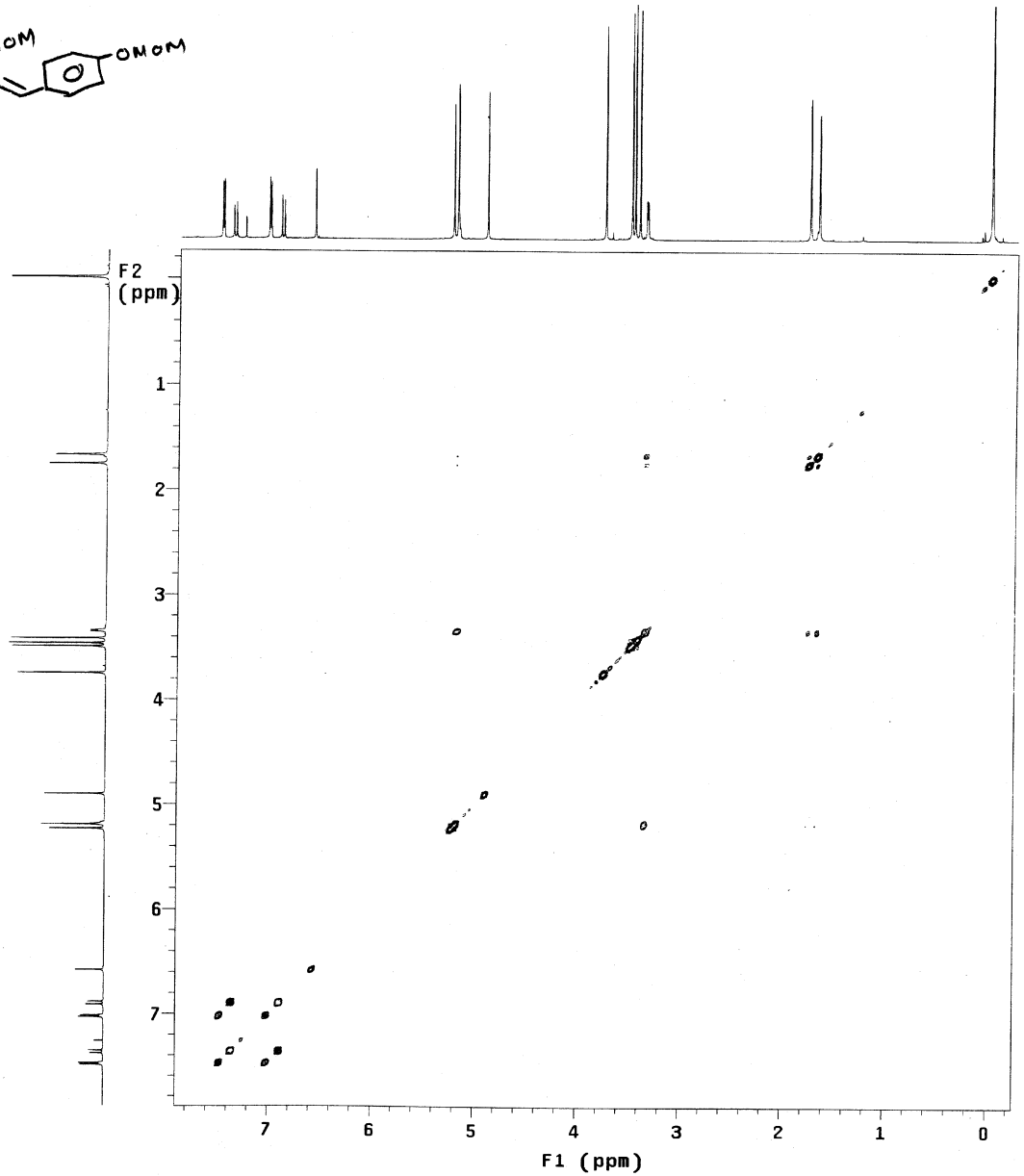
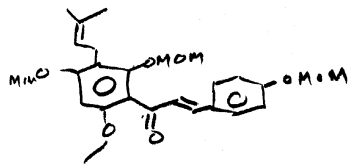


RK X(6)

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul
Solvent: CDCl3
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
384 repetitions
OBSERVE C13, 150.8379775 MHz
DECOUPLE H1, 599.8753144 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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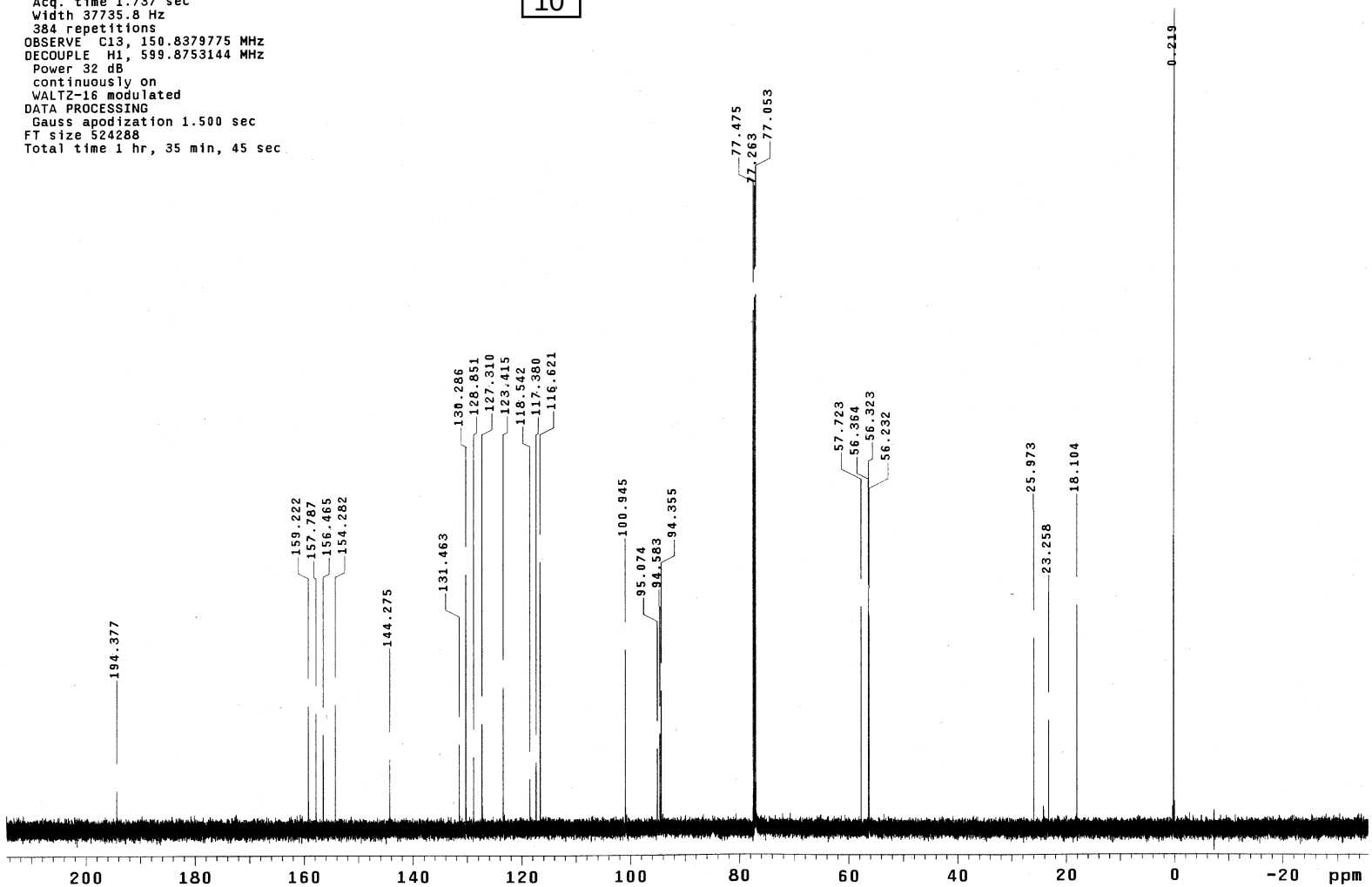
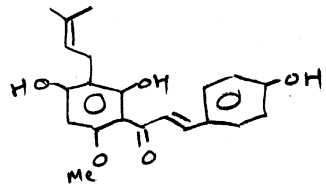


1H H1

STANDARD PROTON PARAMETERS

Pulse Sequence: presat
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, 599.8746735 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 39 sec



1

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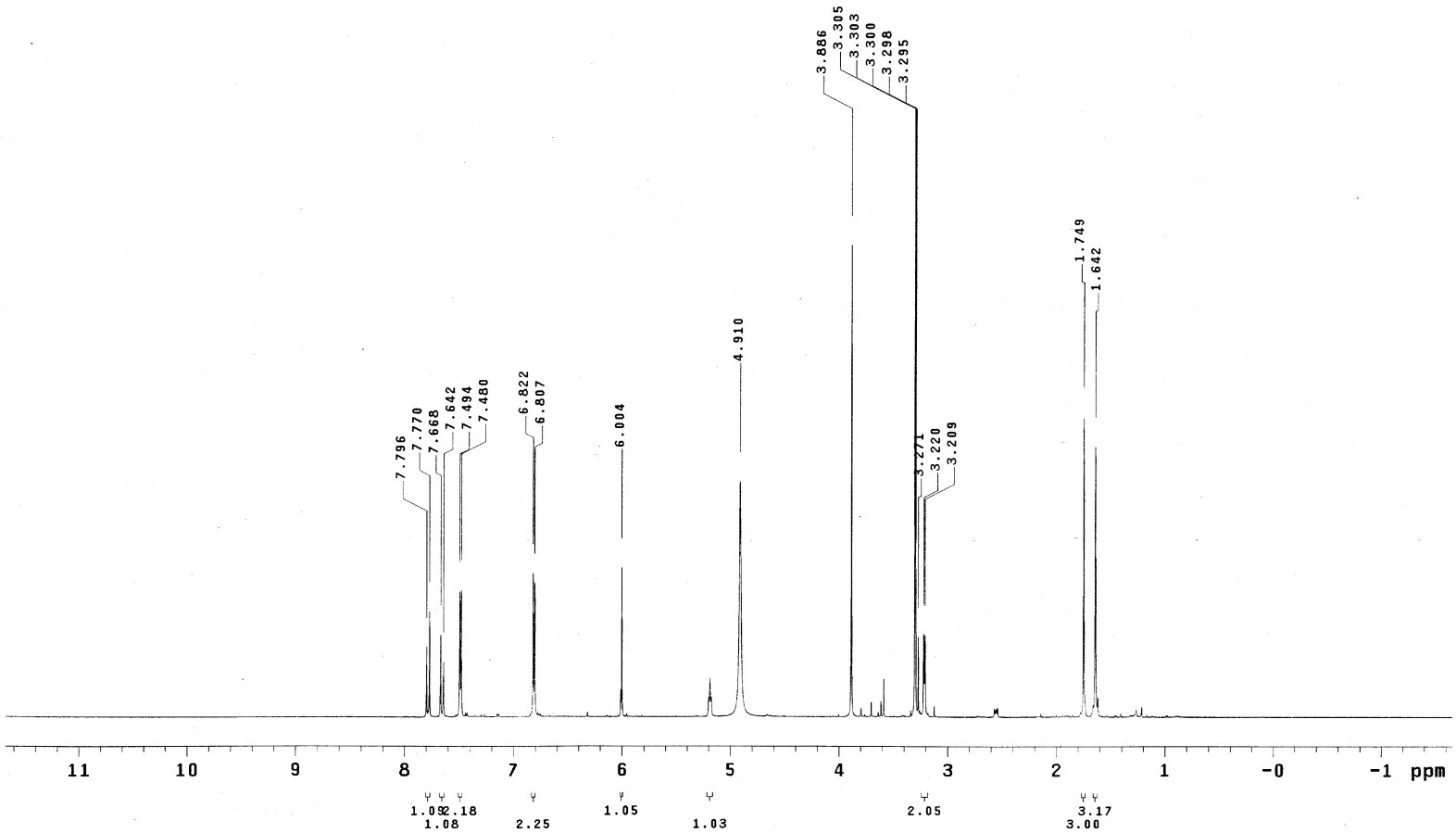
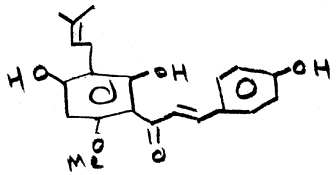


STANDARD PROTON PARAMETERS

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

Relax. delay 1.000 sec
Acq. time 0.199 sec
Width 5148.0 Hz
2D Width 5148.0 Hz
Single scan
512 increments
OBSERVE H1, 599.8746783 MHz
DATA PROCESSING
Sine bell 0.099 sec
F1 DATA PROCESSING
Sine bell 0.050 sec
FT size 2048 x 2048
Total time 10 min, 48 sec



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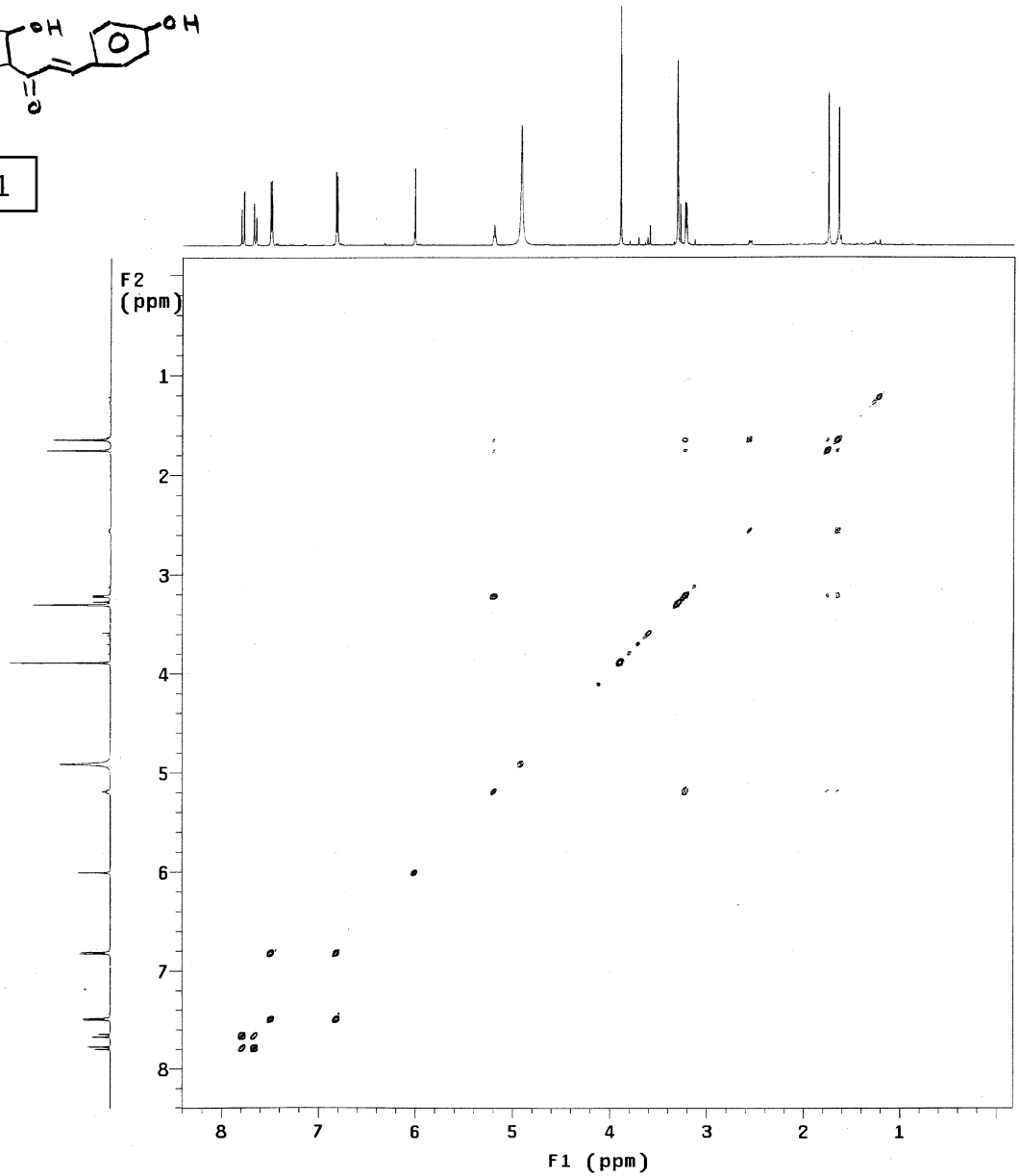
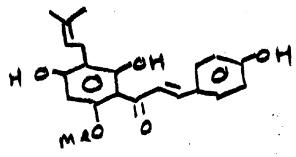


13C OBSERVE

Pulse Sequence: s2pul
Solvent: CD3OD
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, 100.5674131 MHz
DECOUPLE H1, 399.9526799 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



1

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