

Design, Synthesis and Estrogenic Activity of a Novel Estrogen Receptor Modulator - a Hybrid Structure of 17 β -Estradiol and Vitamin E in Hippocampal Neurons

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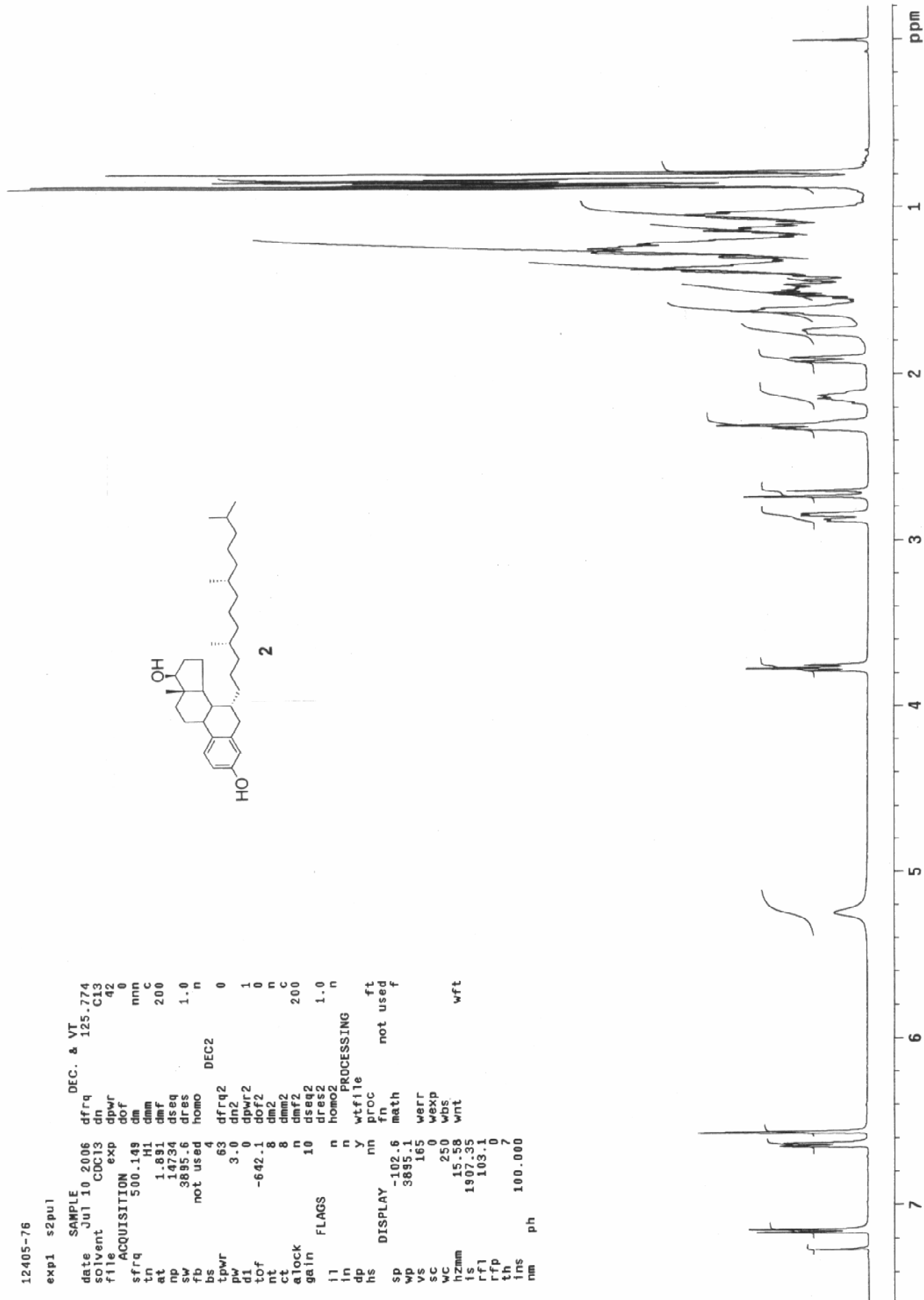
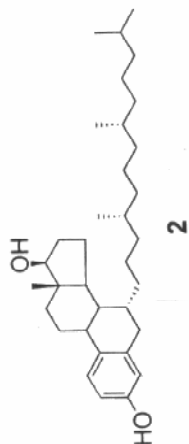
Supporting Information

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NMR spectra (¹ H, ¹³ C, gHSQC, gHMBC, gCOSY and ROESY) of 2	S2
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exp1 s2pul

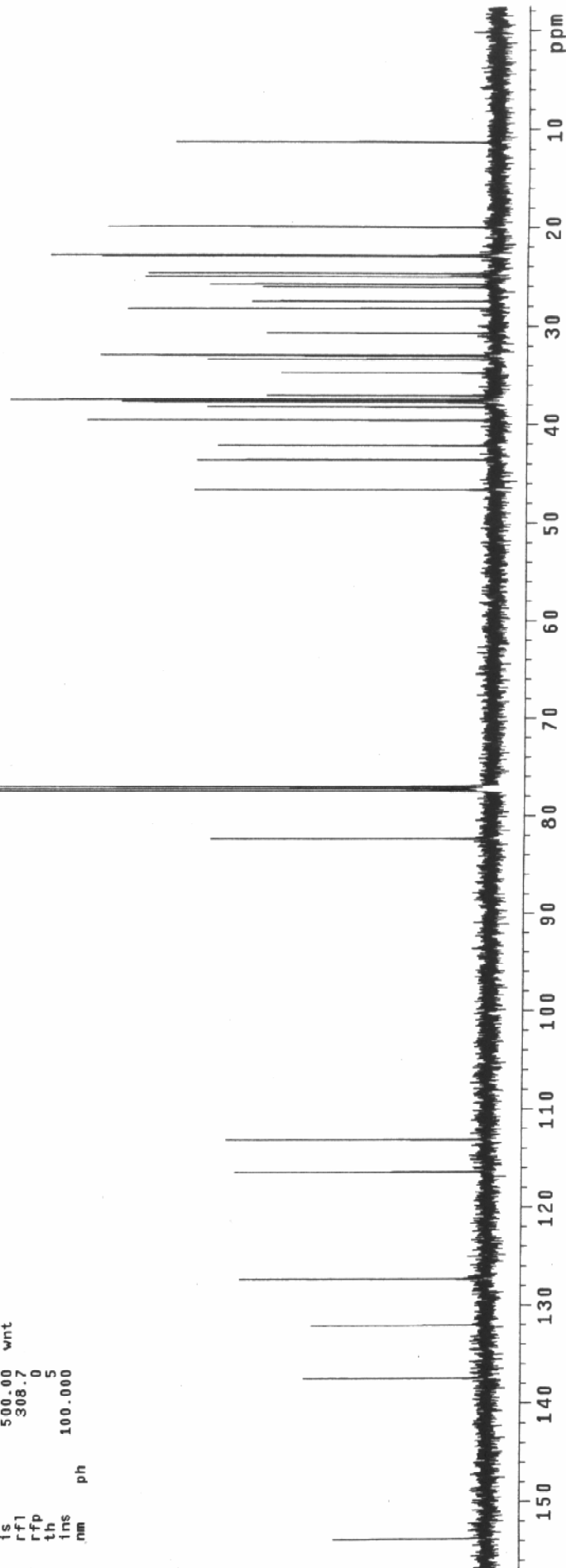
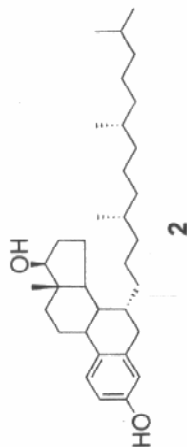
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tn	H1	dmm	c
at	1.851	dmf	200
np	14734	dseq	1.
sw	3895.6	dres	n
fb	not used	homo	
bs	4	DEC2	
tpwr	63	dfrq2	0
pw	3.0	dn2	1
d1	0	dpr2	0
tof	-642.1	dm2	n
nt	8	dmf2	c
ct	8	dmm2	200
alock	n	dses2	1.0
gain	10	dres2	n
FLAGS		homo2	
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in	n	wtfle	ft
dp	y	proc	not used
hs	nn	fn	f
DISPLAY		math	
sp	-102.6	werr	
wp	3895.1	wexp	
vs	165	wbs	
sc	0	wnt	wft
wc	250		
hzmm	15.58		
ls	1907.35		
rfl	103.1		
rff	0		
th	7		
ins	100.000		
nm	ph		



12405-76

exp2 s2pul

SAMPLE		DEC. & VT	
date	Jul 10 2006	dfrq	500.150
solvent	CDCl3	dn	H1
file	exp	dpwr	38
ACQUISITION		dof	0
sfrq	125.772	dm	YYY
tn	C13	dm	W
at	1.303	dmf	12048
np	5254	dseq	
sw	20050.1	dres	1.0
fb	not used	homo	n
bs	4	DEC2	
tpwr	60	dfrq2	0
pw	3.0	dn2	
d1	0	dpwr2	1
tof	-2221.7	dof2	0
nt	1000	dm2	n
ct	1000	dm2	c
alock	n	dmf2	10000
gain	20	dseq2	
FLAGS		dres2	1.0
ll	n	homo2	n
in	n	PROCESSING	
dp	nn	lb	1.00
hs	y	wf1	
DISPLAY		proc	ft
sp	-308.0	fn	not used
wp	20048.5	math	f
vs	200	werr	
sc	0	wexp	
wc	250	wbs	
hzm	80.20	wnt	
is	500.00		
rfl	308.7		
rff	0		
th	5		
ins	100.000		
nm	ph		



12405-76

Pulse Sequence: gHSQC

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

INOVA-500 "tesla"

Relax. delay 1.000 sec

Acq. time 0.131 sec

Width 3885.6 Hz

2D Width 20050.1 Hz

32 repetitions

2 x 128 increments

OBSERVE H1, 500.1472535 MHz

DECOUPLE C13, 125.7718205 MHz

Power 43 dB

on during acquisition

off during delay

W40_1d 8182 modulated

DATA PROCESSING

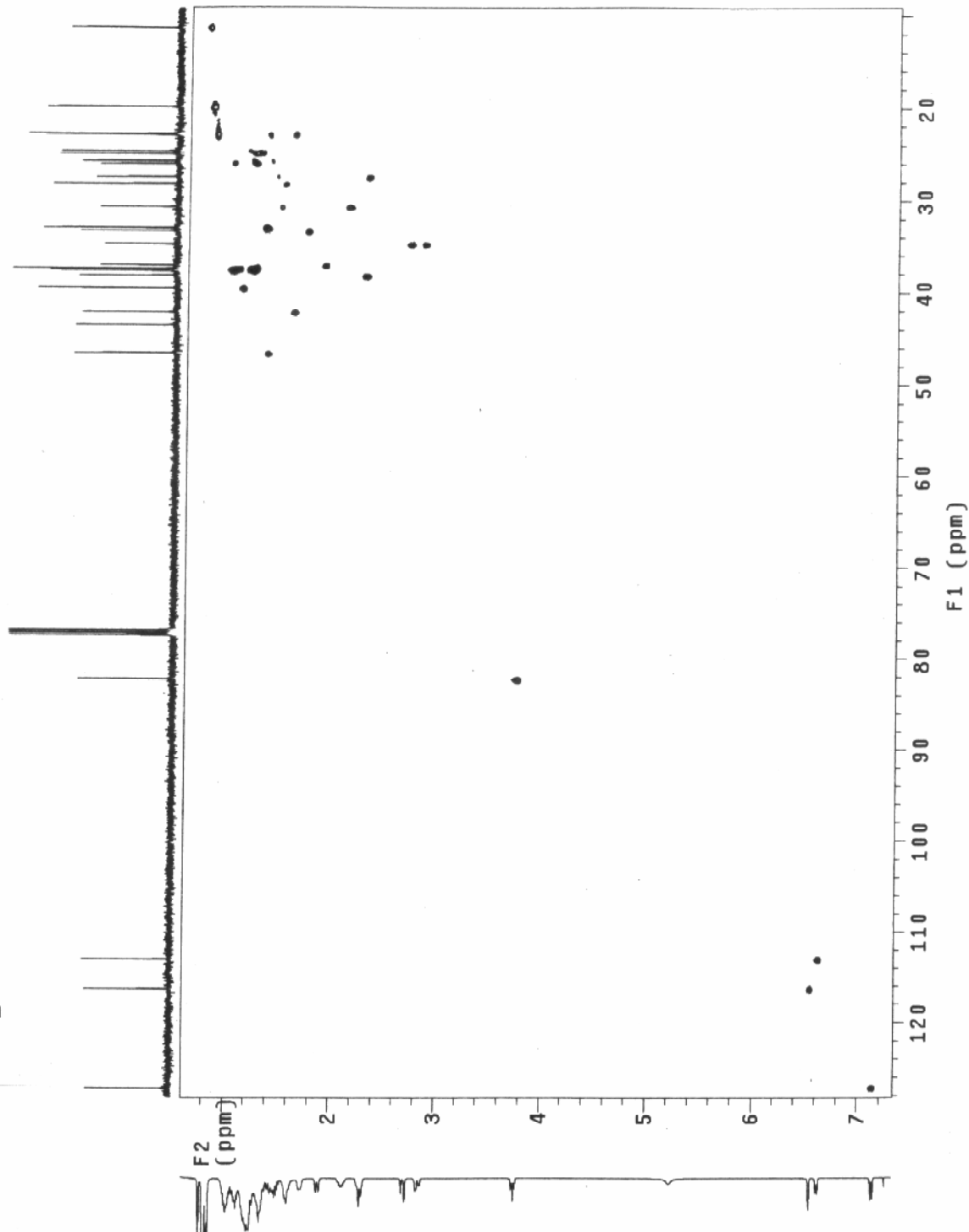
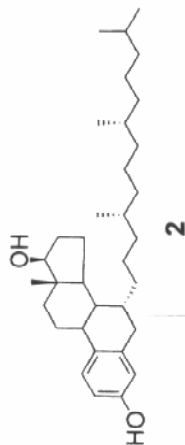
Gauss apodization 0.061 sec

F1 DATA PROCESSING

Gauss apodization 0.011 sec

FT size 1024 x 2048

Total time 2 hr, 44 min, 53 sec



12405-76

Pulse Sequence: gHMBC

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

INOVA-500 "tesla"

Relax. delay 1.000 sec

Acq. time 0.131 sec

Width 3895.6 Hz

20 Width 20050.1 Hz

64 repetitions

400 increments

OBSERVE H1, 500.1472538 MHz

DATA PROCESSING

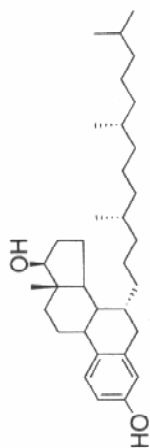
Sine bell 0.066 sec

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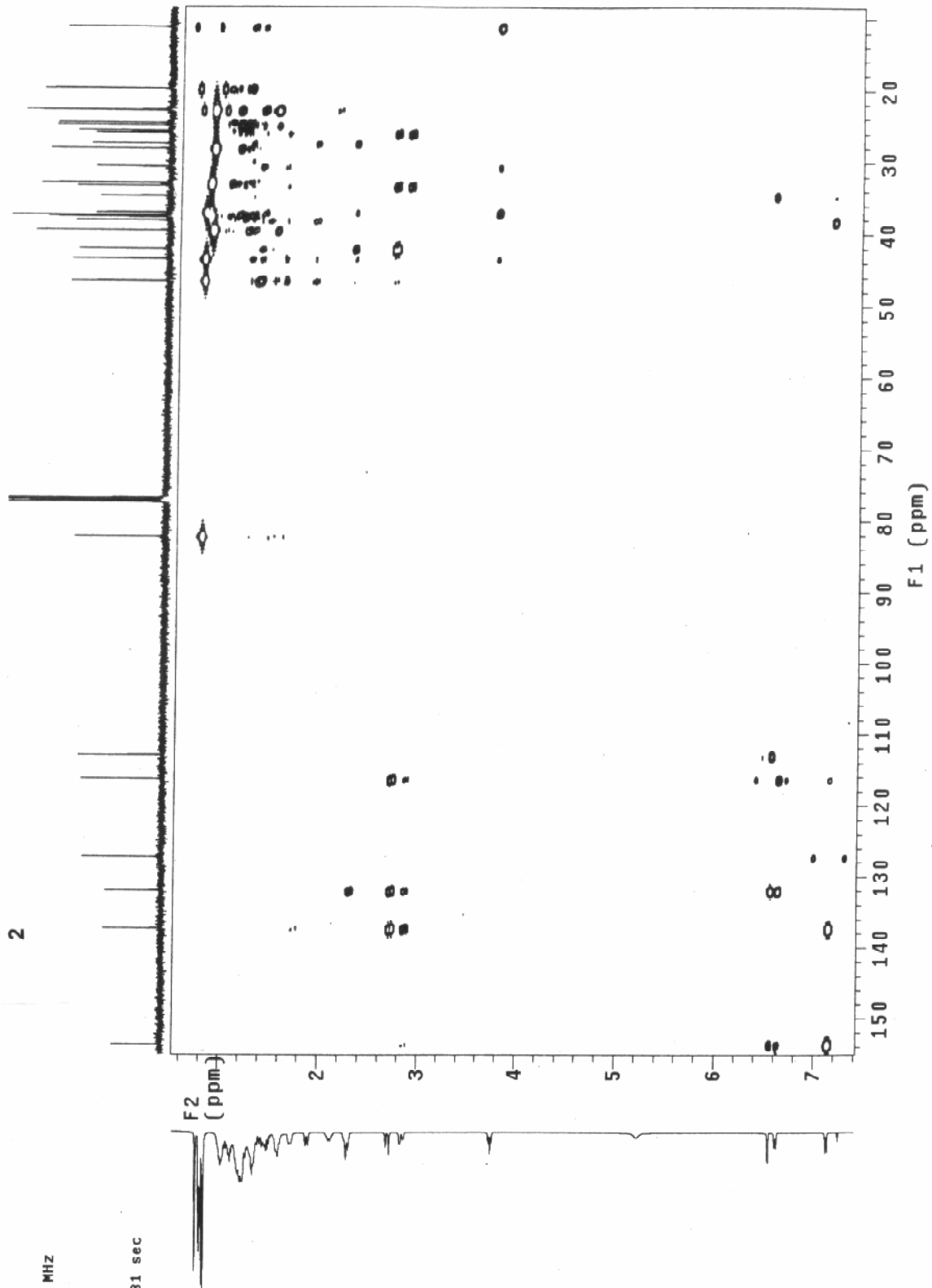
Sine bell 0.007 sec

FI size 1024 x 2048

Total time 8 hr, 42 min, 31 sec



2



12405-76

Pulse Sequence: gCOSY

Solvent: CDCl₃

Ambient temperature

INOVA-500 "tesla"

Relax. delay 1.000 sec

Acq. time 0.131 sec

Width 3895.6 Hz

2D Width 3895.6 Hz

16 repetitions

128 increments

OBSERVE H1 500.1472543 MHz

DATA PROCESSING

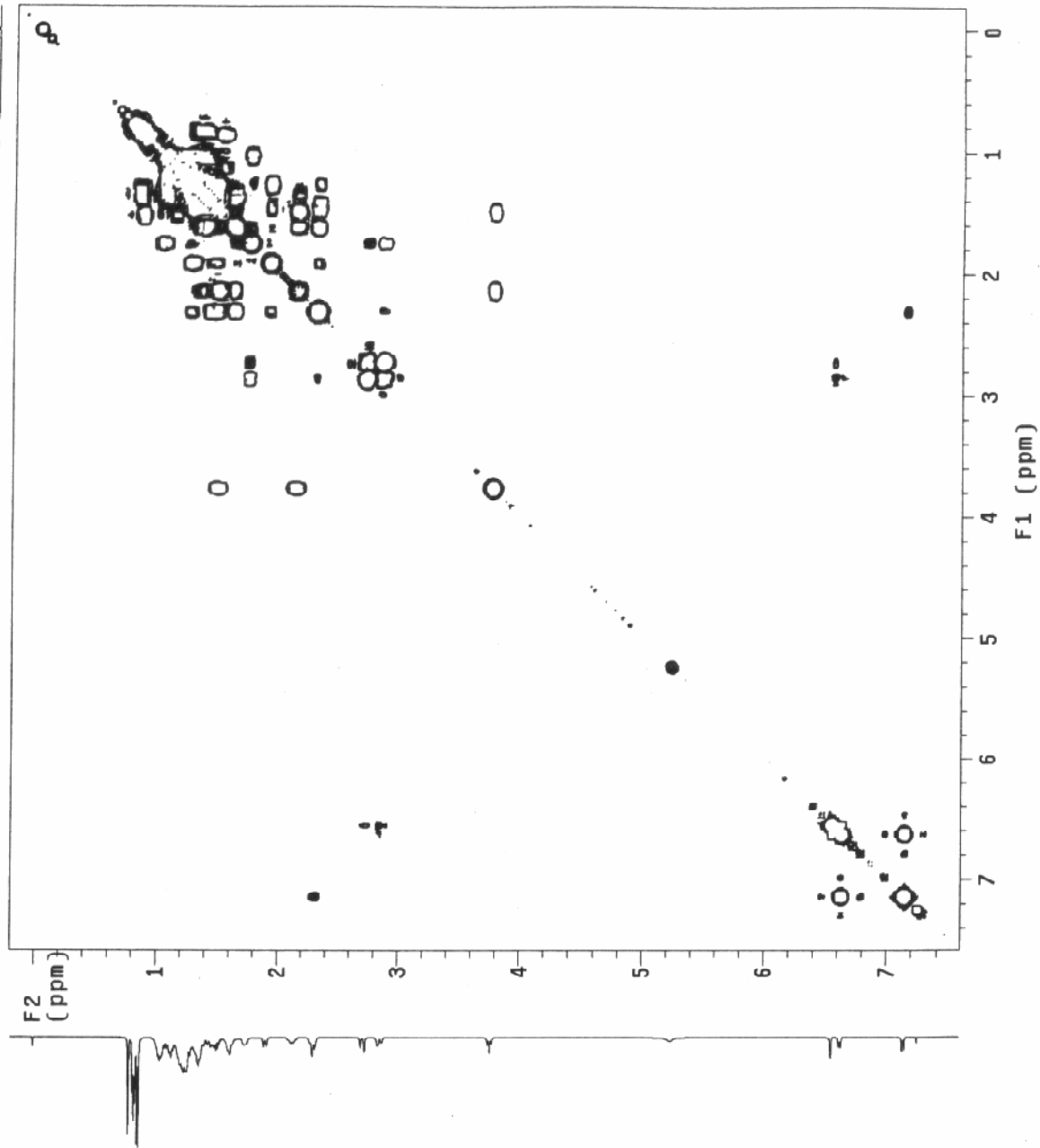
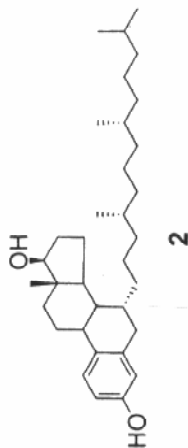
Sq. sine bell 0.066 sec

F1 DATA PROCESSING

Sq. sine bell 0.033 sec

FT size 1024 x 1024

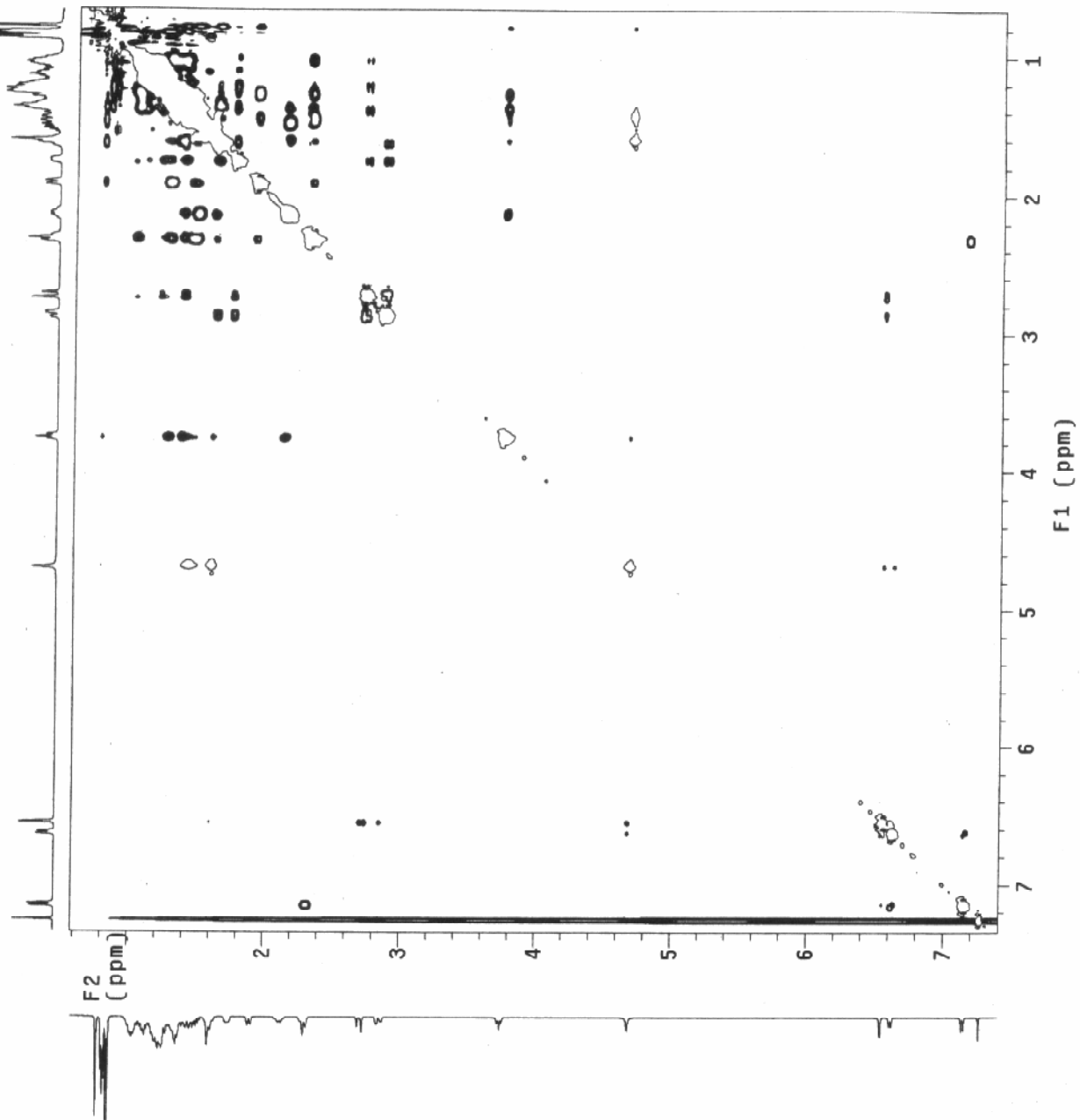
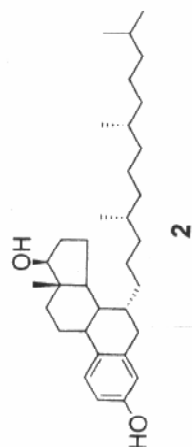
Total time 39 min, 55 sec



12405-76 ROESY

Pulse Sequence: ROESY
Solvent: CDCl₃
Ambient temperature
INOVA-500 "tesla"

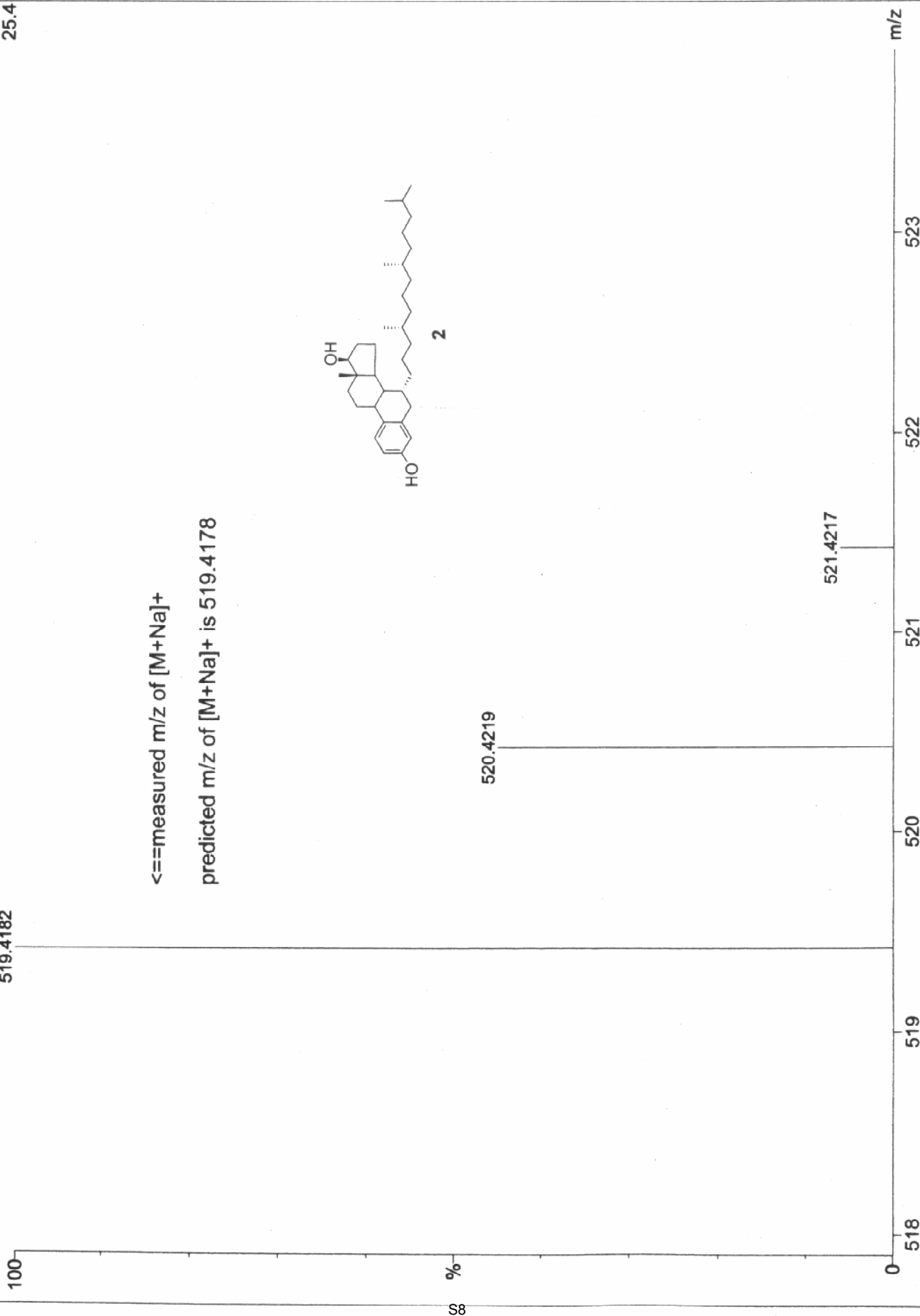
Relax. delay 1.000 sec
Mixing 0.400 sec
Acq. time 0.131 sec
Width 3895.6 Hz
20 Width 3895.6 Hz
64 repetitions
2 x 200 increments
OBSERVE H1, 500.1472510 MHz
DATA PROCESSING
Gauss apodization 0.061 sec
F1 DATA PROCESSING
Gauss apodization 0.047 sec
FT size 2048 x 2048
Total time 11 hr, 12 min, 20 sec



RTI-12405-76 Electrospray with Na+ added

RTI-12405-76_Na_HR 16 (0.294) Cn (Cen,7, 50.00, Ht); Sb (15,40.00); Sm (SG, 3x7.00); Cm (1:115)
519.4182

Voltage ES+
25.4

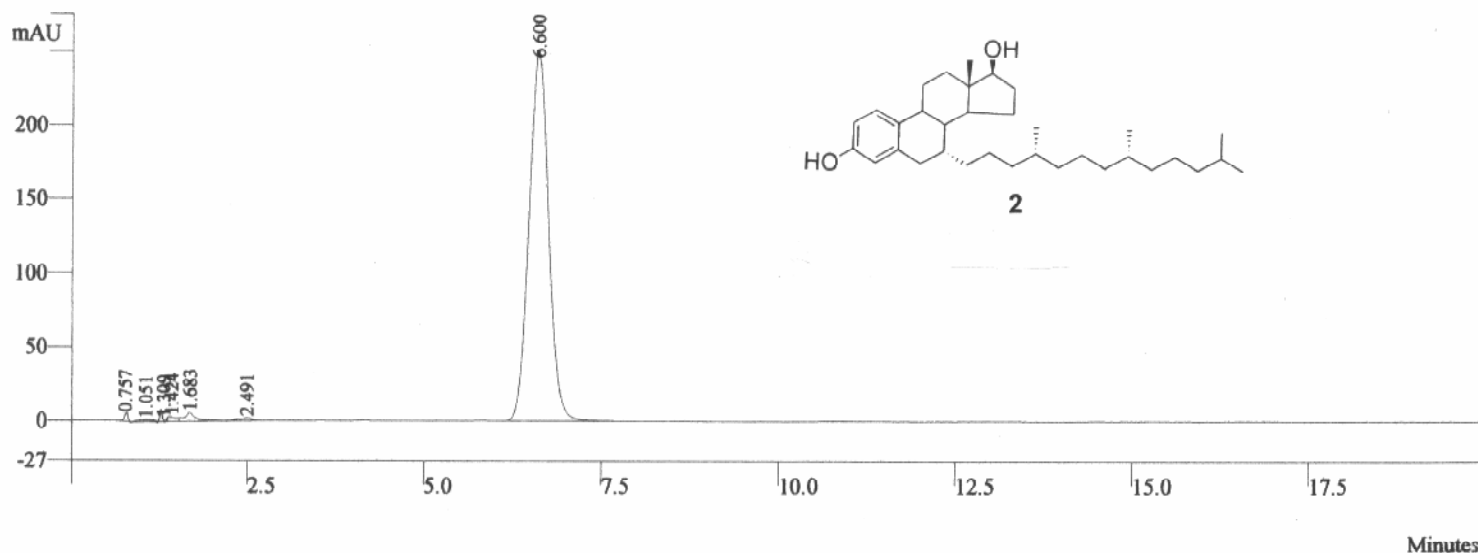


Method Notes

X Terra MS C-18, 5 um, 3.9x150 mm column

90% MeOH - 10% Water

1 ml/min



Peak No	Result (%)	Ret Time (min)	Rel Ret Time	Sep. Code	Width 1/2 (sec)	Peak Area (counts)	Peak Height (counts)
1	0.3963	0.757	0.00	BP	1.6	100220	24744
2	0.3345	1.051	0.00	PV	0.0	84594	5340
3	0.4255	1.309	0.00	VV	8.3	107619	9401
4	0.4922	1.424	0.00	VV	0.0	124483	15429
5	1.3364	1.683	0.00	VV	7.0	337969	27454
6	0.4632	2.491	0.00	VB	7.7	117148	9775
7	96.5518	6.600	0.00	BB	17.8	24417822	1278387
99.9999						25289856	1370530

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Run Time (min): 20.000
Workstation: W98
Instrument (Inj): Star HPLC system 1

Operator (Calc): CJIN
Calc Date: 07/17/06 03:43:21 PM
Times Calculated: 1
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Instrument (Calc): Star HPLC system 1
Run Mode: Analysis
Peak Measurement: Peak Area
Calculation Type: Percent
Calibration Level: N/A
Verification Tolerance: N/A

Elemental analysis of **2**.

Compound	Formula	Calculated		Found	
		C	H	C	H
2	$\text{C}_{34}\text{H}_{56}\text{O}_2 \cdot 0.25\text{H}_2\text{O}$	81.46	11.36	81.51	11.35