Supporting Information for:

Synthesis of Cyclopentanyl Carbocyclic 5-Fluorocytosine ((-)-5-Fluoro-Carbodine) Using a Facially Selective Hydrogenation Approach

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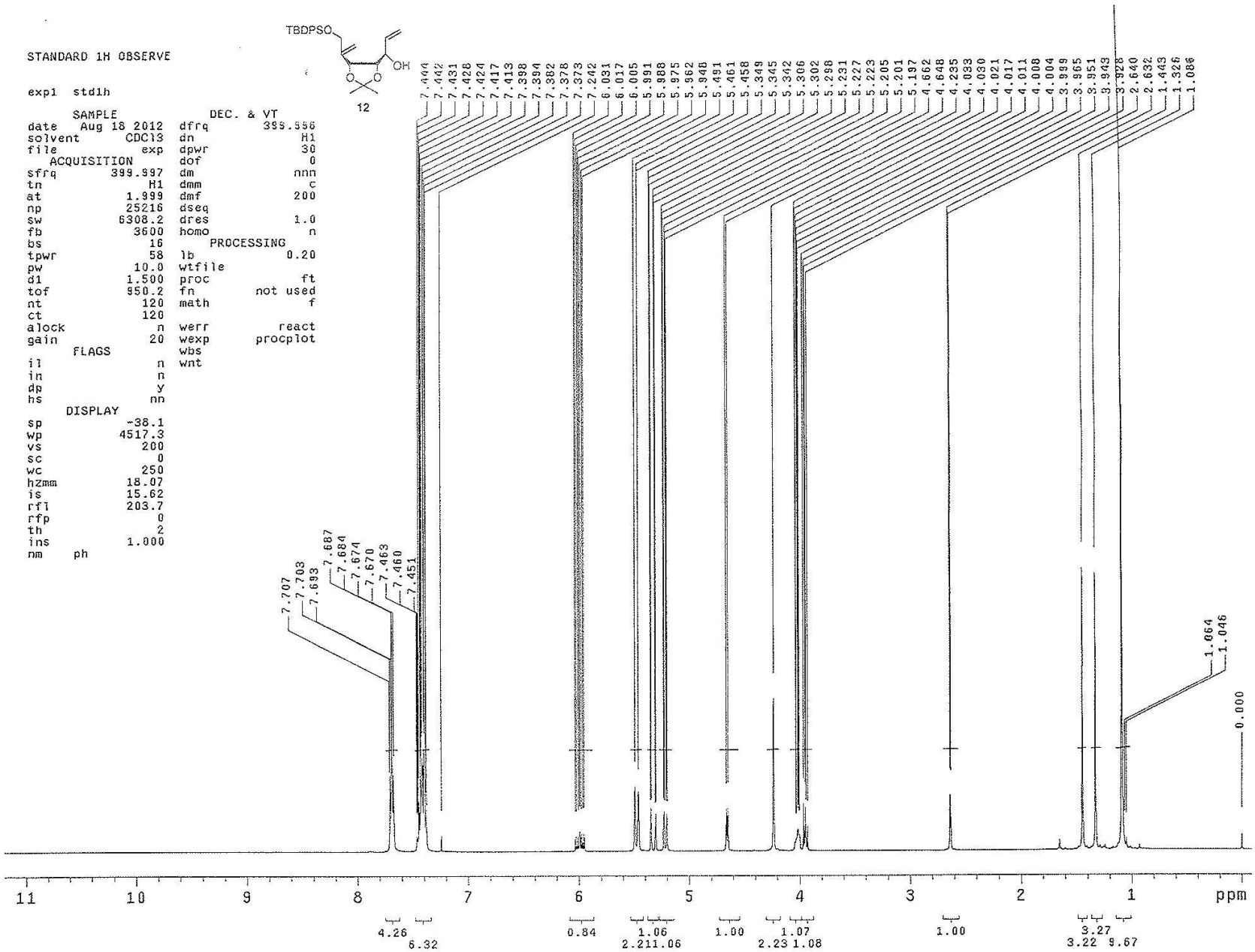
Table 1. Condition Optimization for the Formation of **18**

Entry	Reaction conditions	4-pentenoyl chloride	Yield (%)
1	Pyridine, rt, 24 h	4.0 equiv	15
2	DMAP, Pyridine,rt, 12 h	3.0 equiv	24
3	Et_3N, Pyridine,rt, 12 h	3.0 equiv	28
4	Et_3N, DMAP, CH_2Cl_2, rt, 12 h	4.0 equiv	56
5	Et_3N, CH_2Cl_2, 0 °C, 3 h	4.0 equiv	97
6	Et_3N, CH_2Cl_2, 0 °C, 4 h	3.0 equiv	97

Table 2. Condition Optimization for the Formation of Compound **19** from **18** using 2nd Generation Grubbs' Catalyst **D**

Entry	Solvent[a]	Reaction conditions	RCM % yield (recovered SM %)[b]
1	CH_2Cl_2	5.0 mol% **D**, rt, 48 h	25 (45)
2	CH_2Cl_2	1.0 mol% **D**, rt, 72 h	30 (40)
3	CH_2Cl_2	2.5 mol% **D**, 40 °C, 12 h	43 (32)
4	$Cl(CH_2)_2Cl$	2.5 mol% **D**, 40 °C, 24 h	35 (30)
4	Toluene	5.0 mol% **D**, rt, 24 h	32 (40)
5	Toluene	5.0 mol% **D**, 40 °C, 6 h	70 (15)
6	Toluene	5.0 mol% **D**, 80 °C, 0.5 h	87
7	Toluene	2.0 mol% **D**, 80 °C, 1 h	90
8	Toluene	2.0 mol% **D**, 80 °C, 6 h	78 (5)

[a]1mM concentration. [b]Yields of isolated compounds **18** and **19** after column chromatography.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STANDARD 1H OBSERVE

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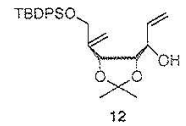

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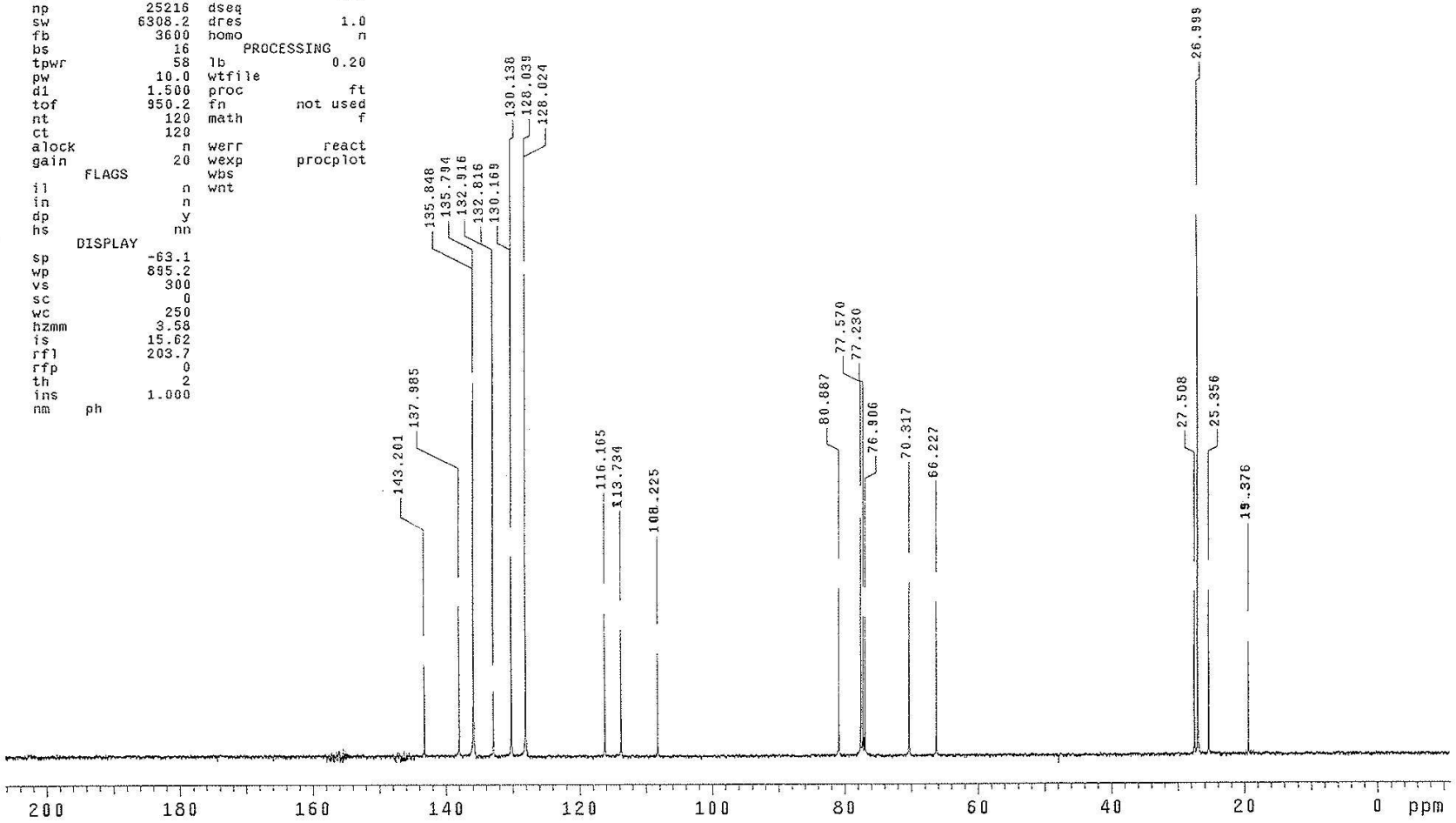

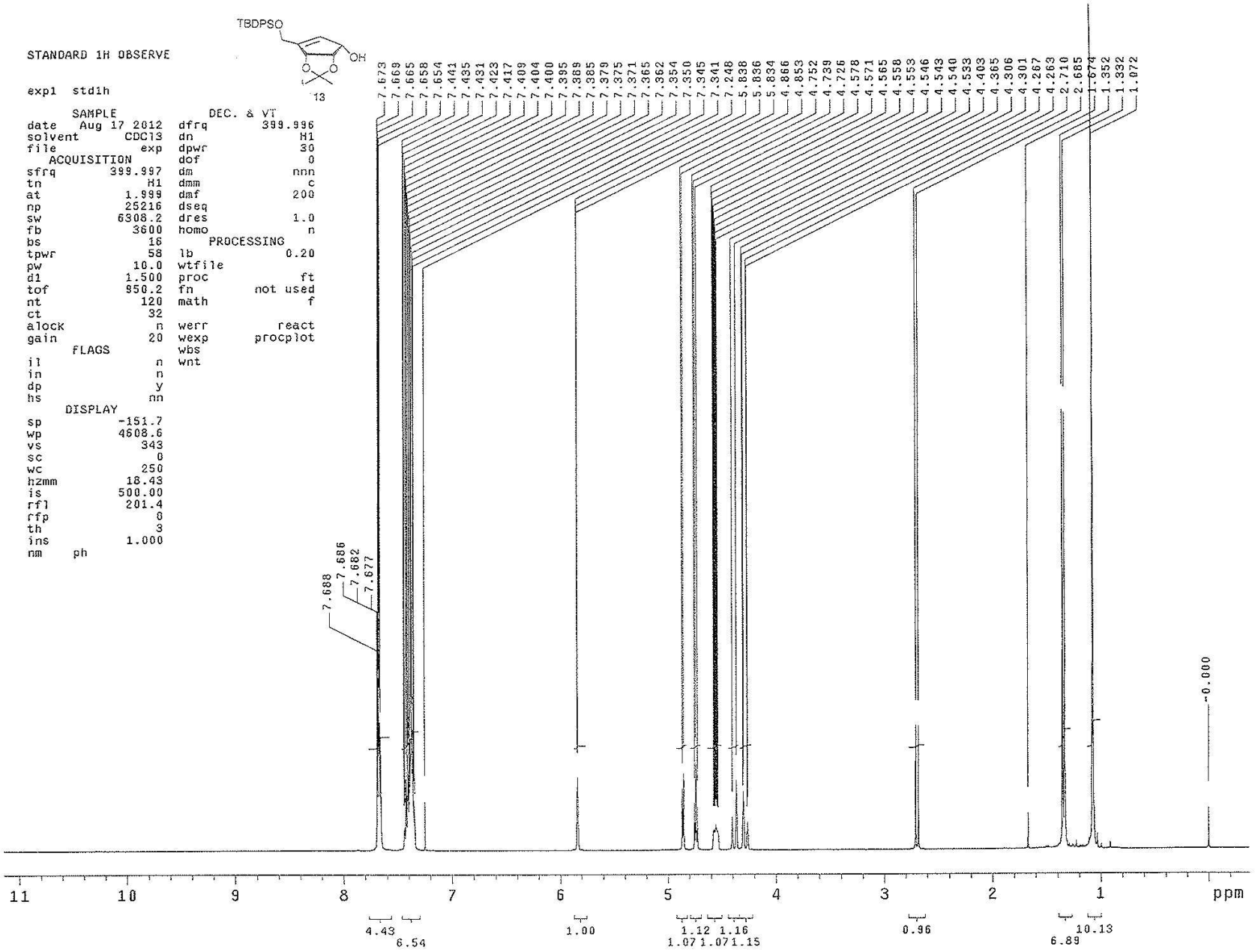
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STANDARD 1H OBSERVE

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gain	20	wexp	procplot
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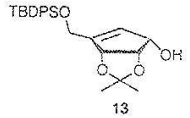

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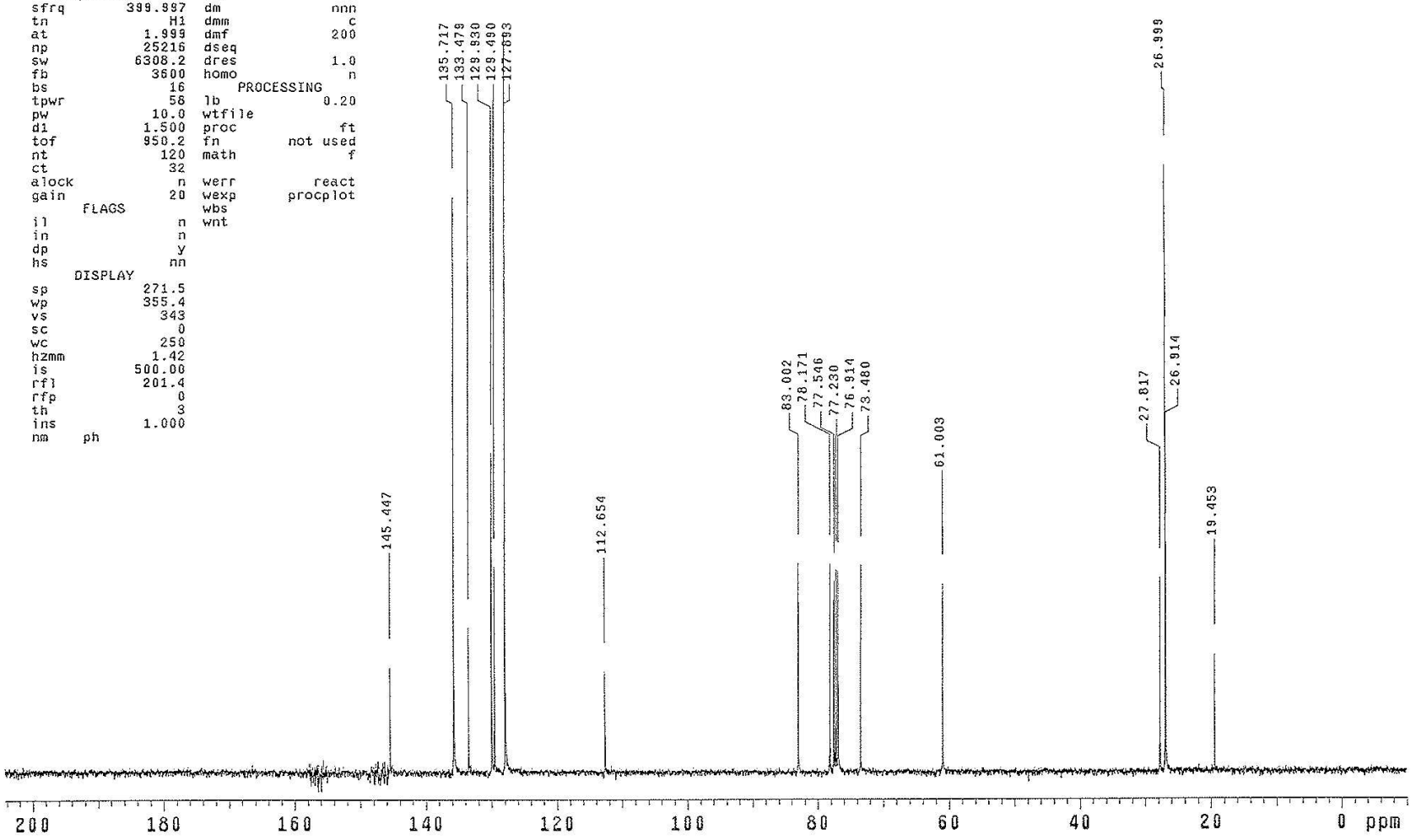

STANDARD 1H OBSERVE

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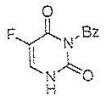

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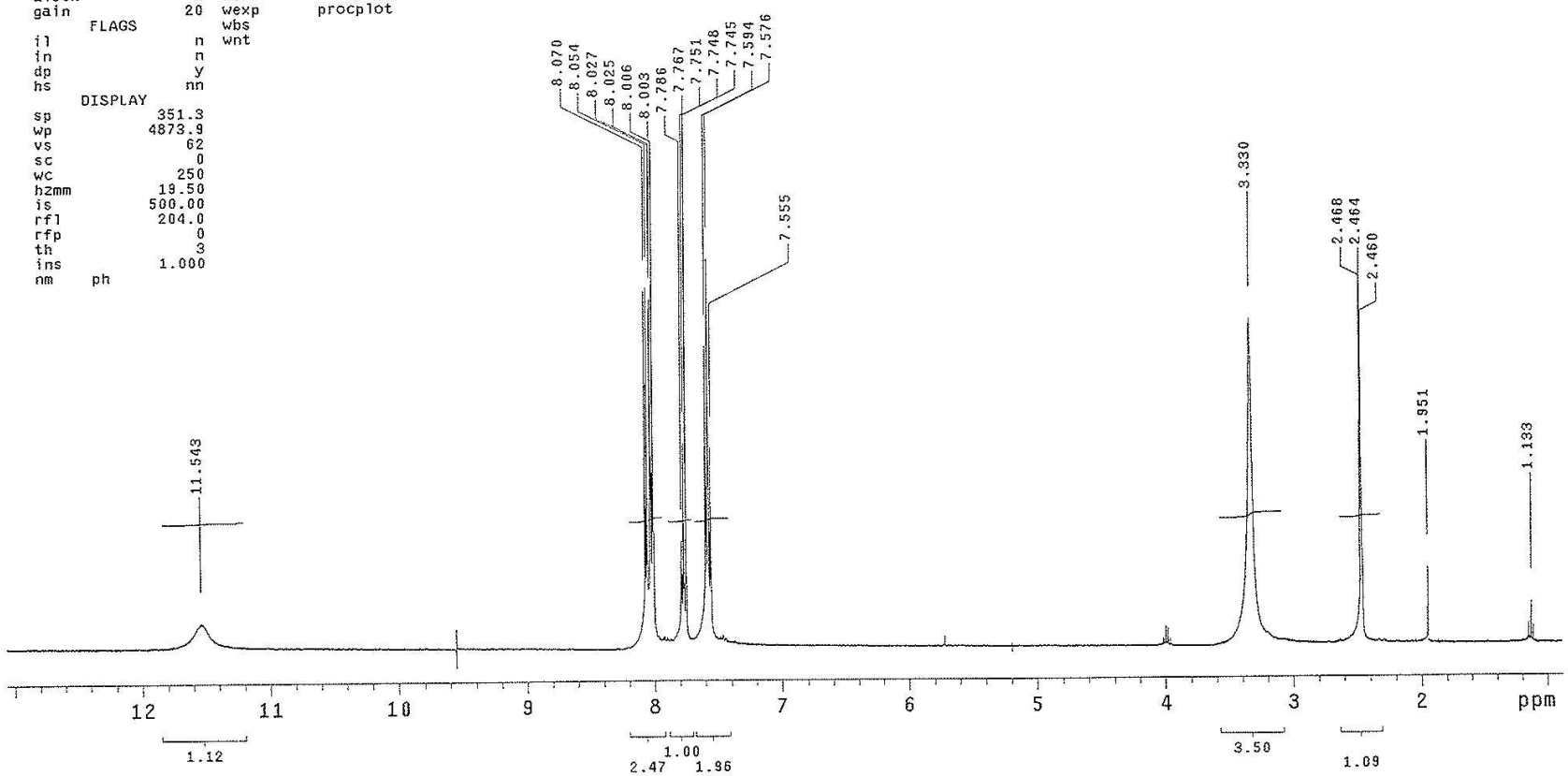

STANDARD 1H OBSERVE

exp1 std1h

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sw	6308.2	dres	1.0
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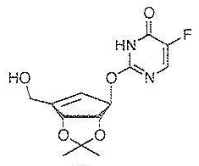

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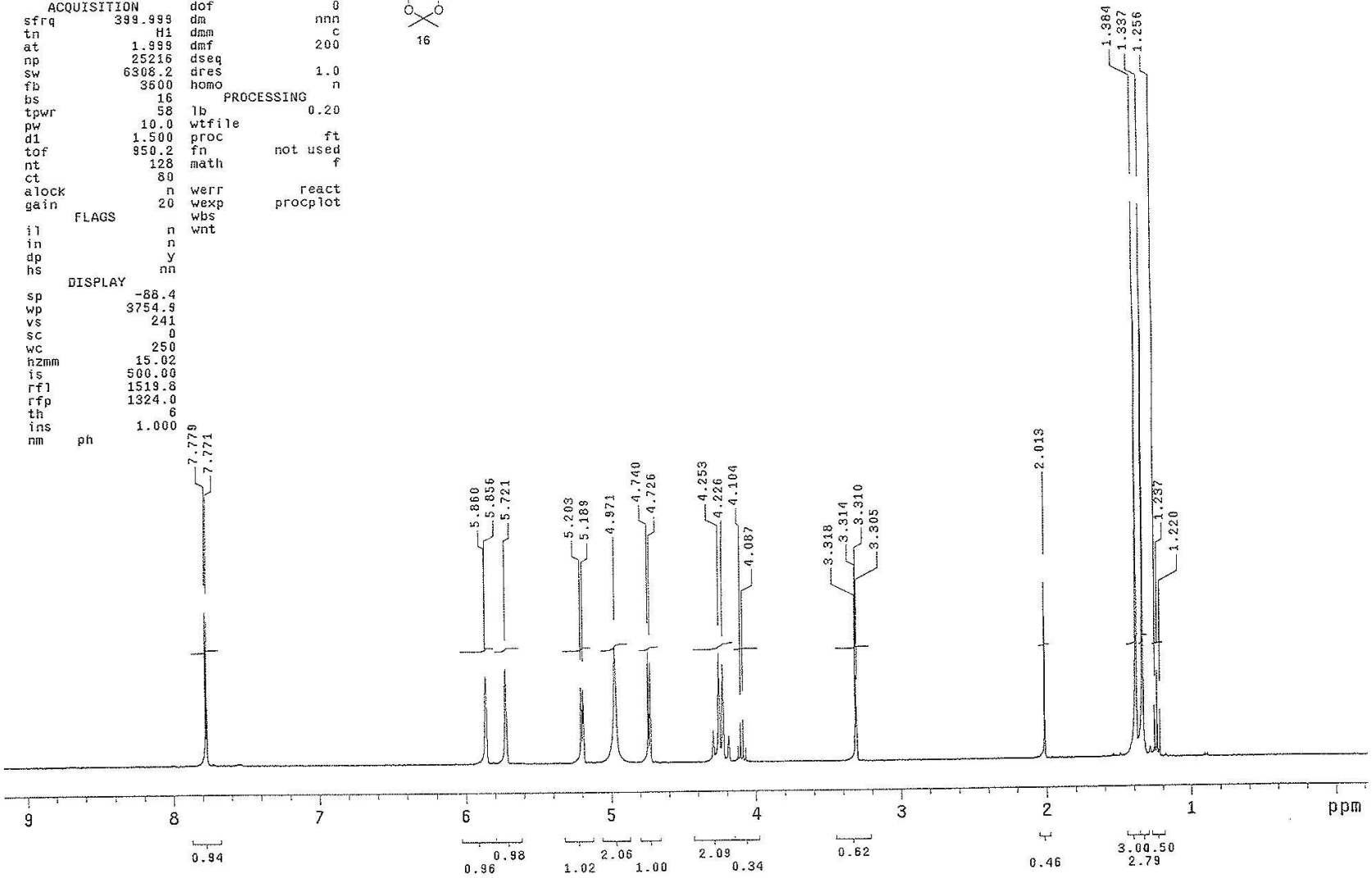

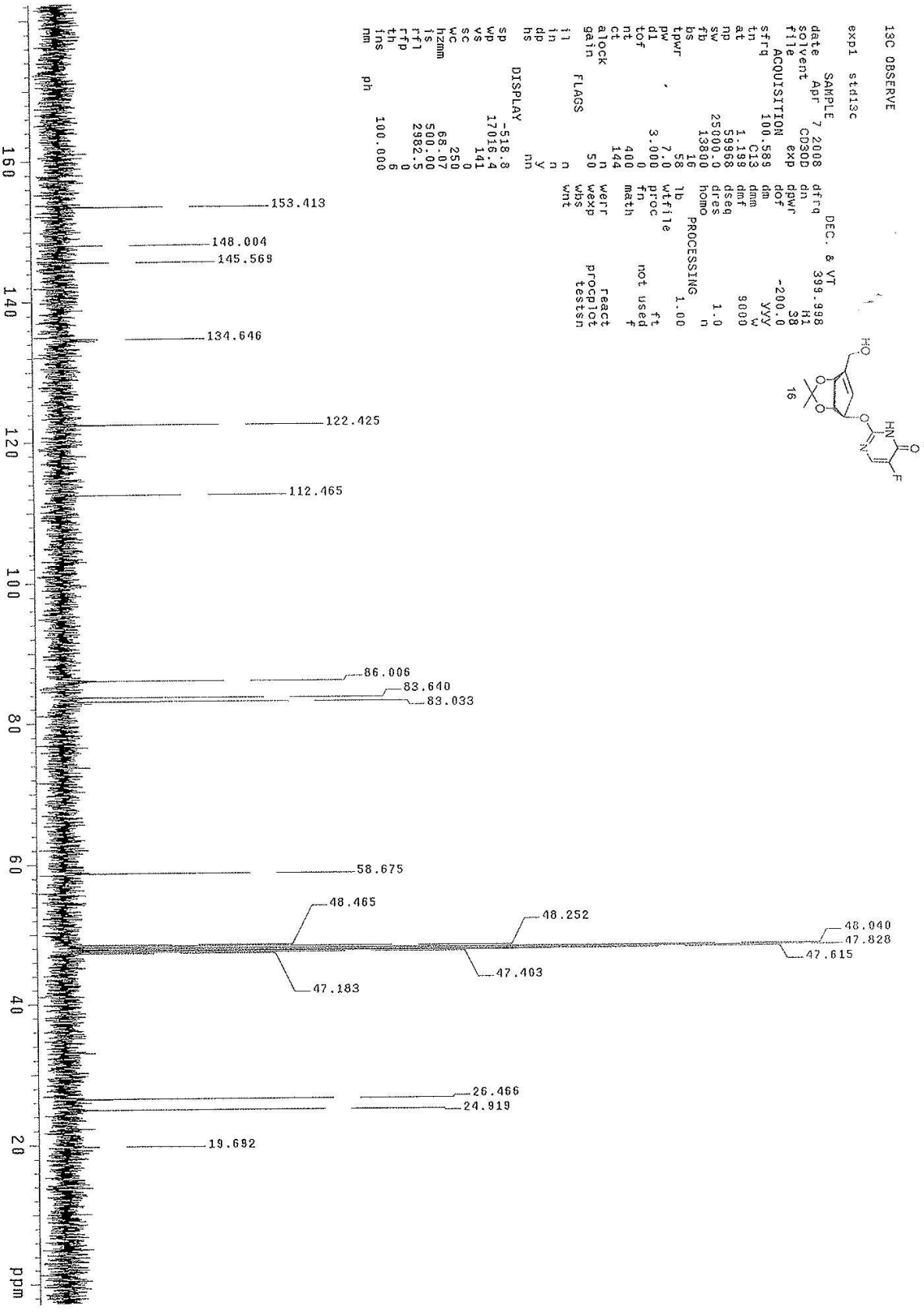
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STANDARD 1H OBSERVE

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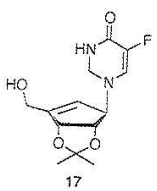

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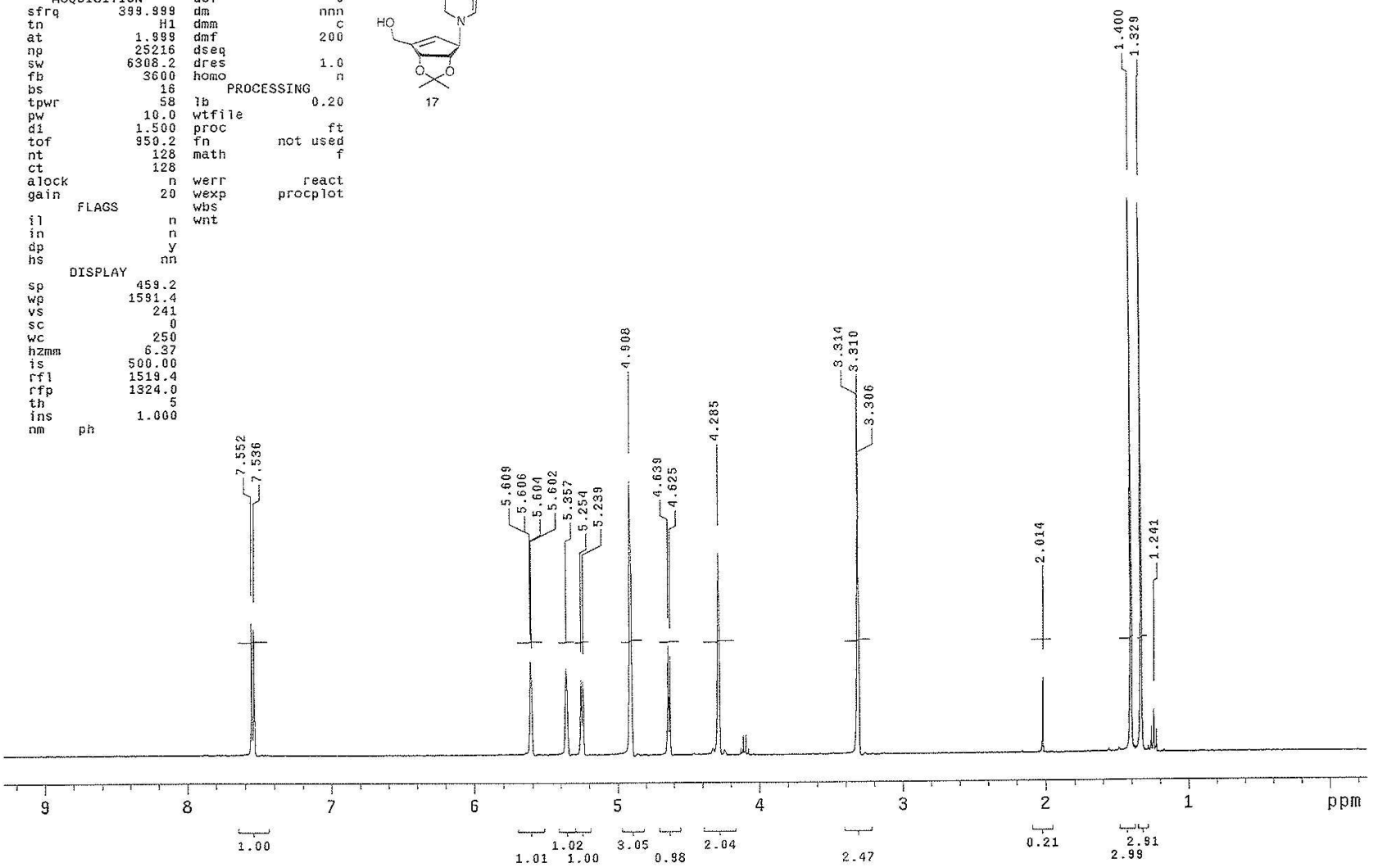

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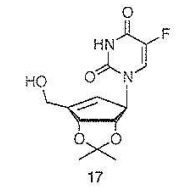

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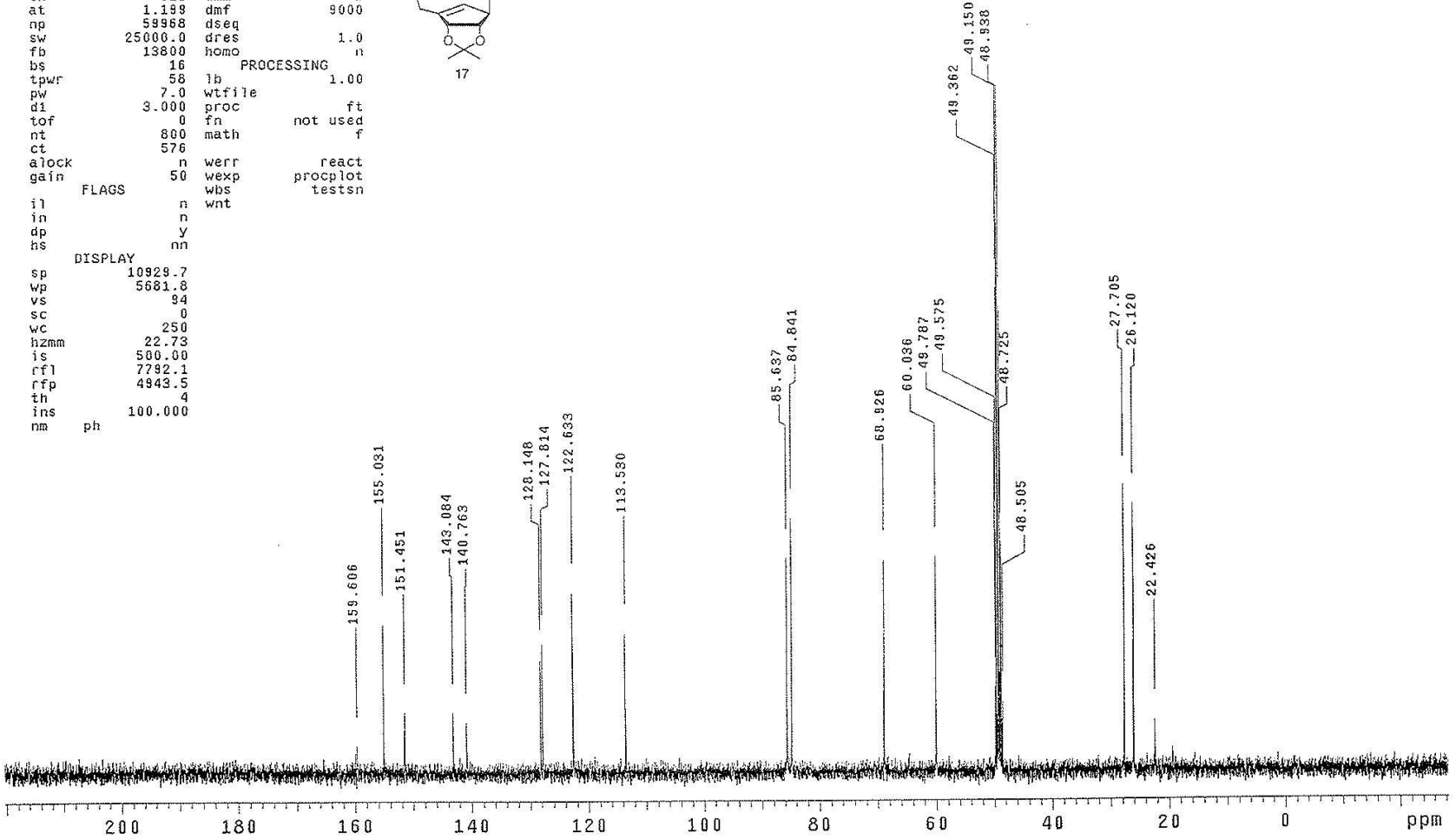

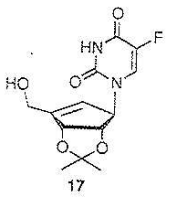

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F19_STD

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hs	nn		
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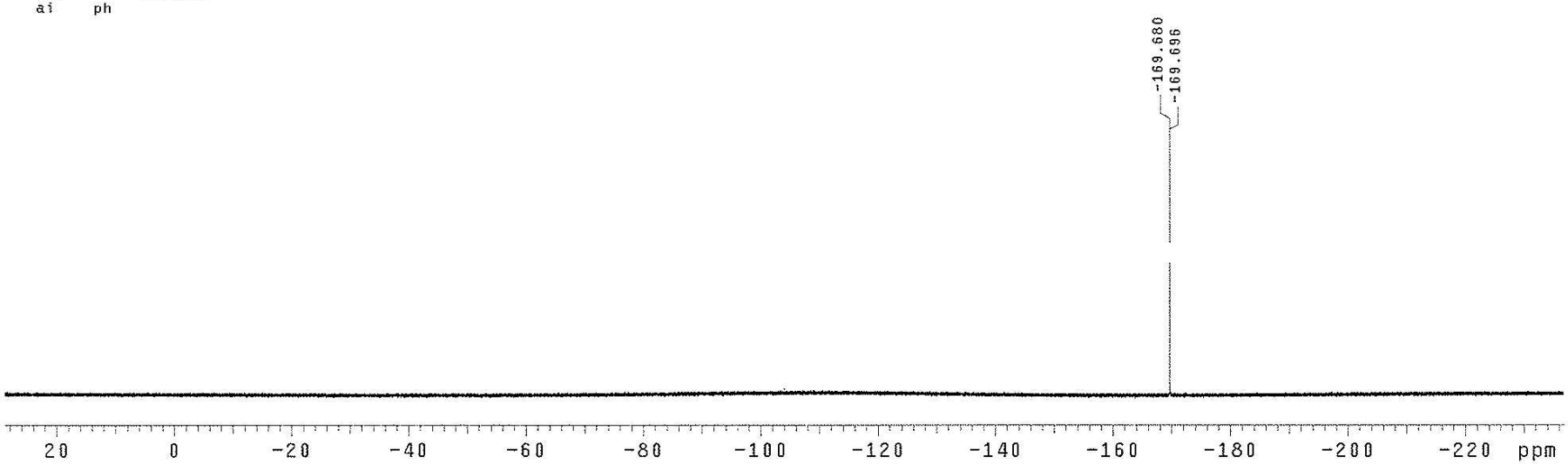
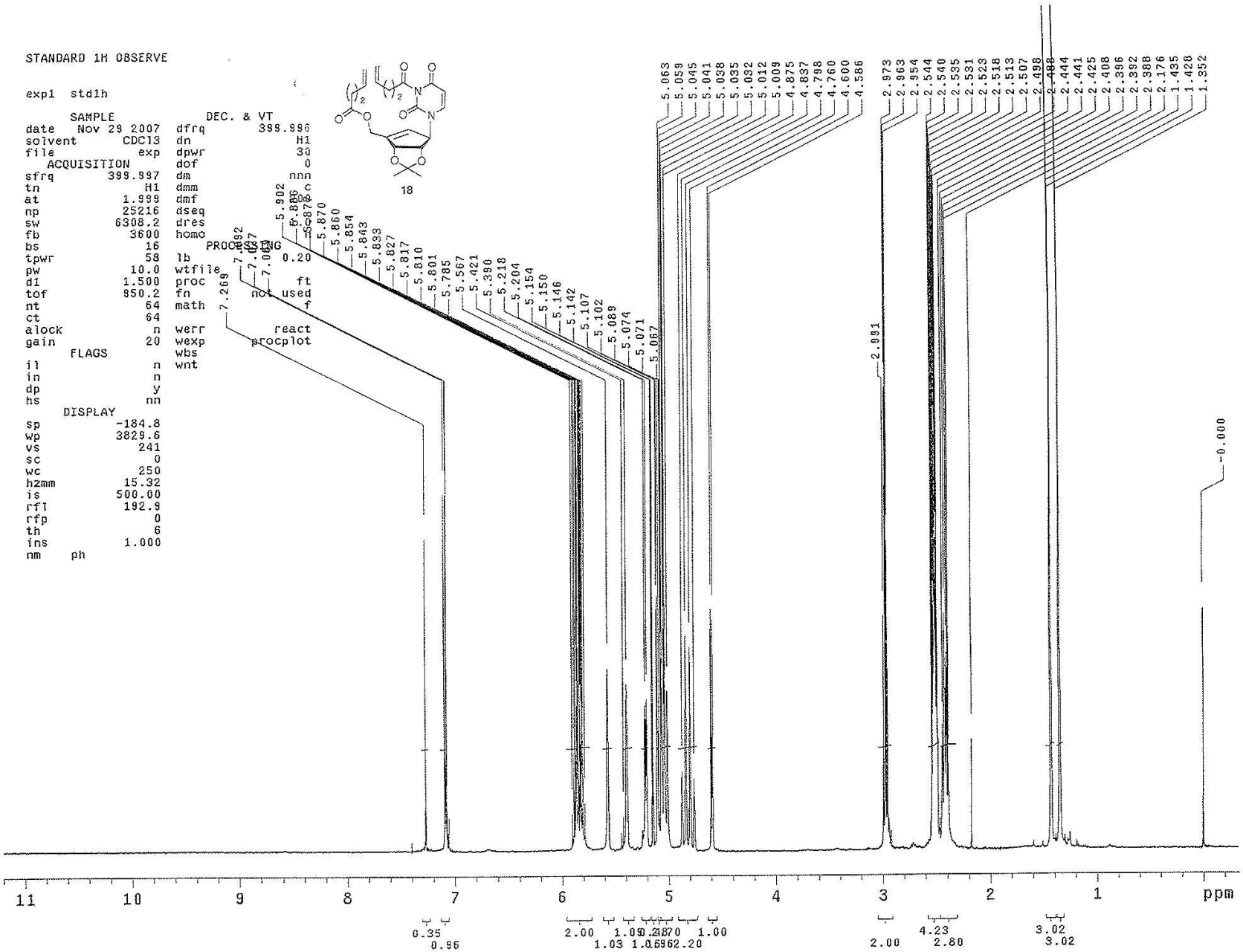
ai ph			

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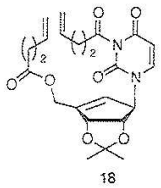
dm nnn
dmm c
dmf
dseq
dres
homo
PROCESSING
lb 0.20
wtfile
proc ft
fn not used
math f
werr react
wexp procplot
wbs
wnt
18
ppm
0.35
0.96
2.00
1.03
1.00
2.00
4.23
2.80
3.02
3.02

STANDARD 1H OBSERVE

exp1 std1h

SAMPLE		DEC. & VT	
date	Apr 8 2008	dfrq	399.996
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	399.997	dm	nnn
tn	H1	dmm	c
at	1.998	dmf	200
np	25216	dseq	
sw	6308.2	dres	1.0
fb	3600	homo	n
bs	16	PROCESSING	
tpwr	58	lb	0.20
pw	10.0	wtfile	
d1	1.500	proc	ft
tof	950.2	fn	not used
nt	32	math	f
ct	32		
alock	n	werr	react
gain	20	wexp	procplot
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-177.0		
wp	4650.2		
vs	241		
sc	0		
wc	250		
hzmm	18.60		
is	500.00		
rfl	3101.5		
rfp	2908.0		
th	6		
ins	1.000		
nm	ph		



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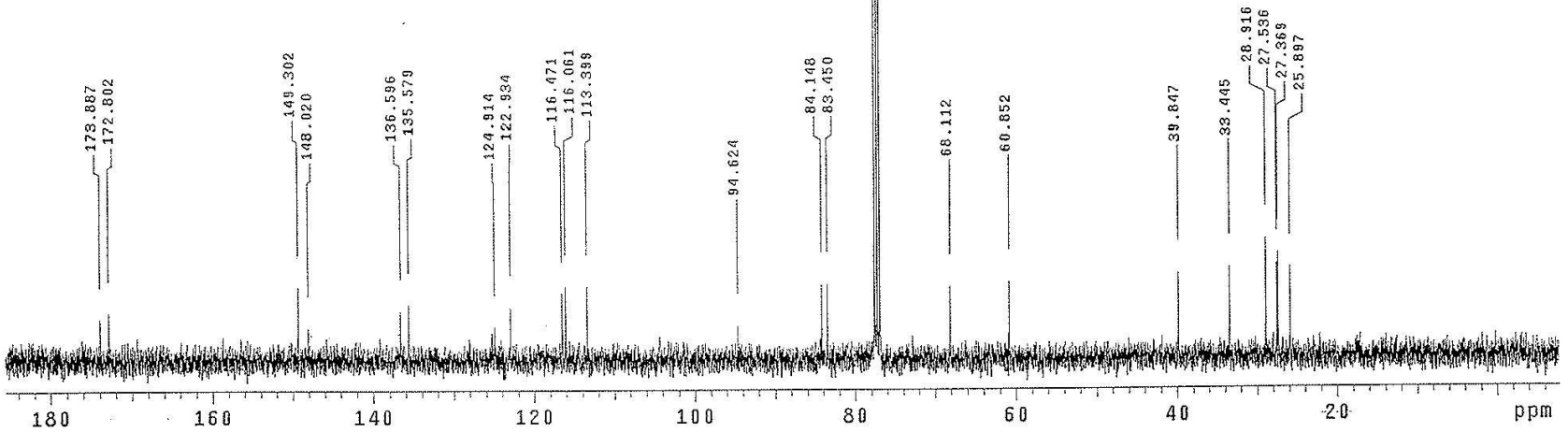
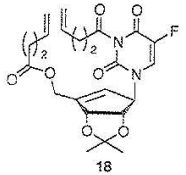


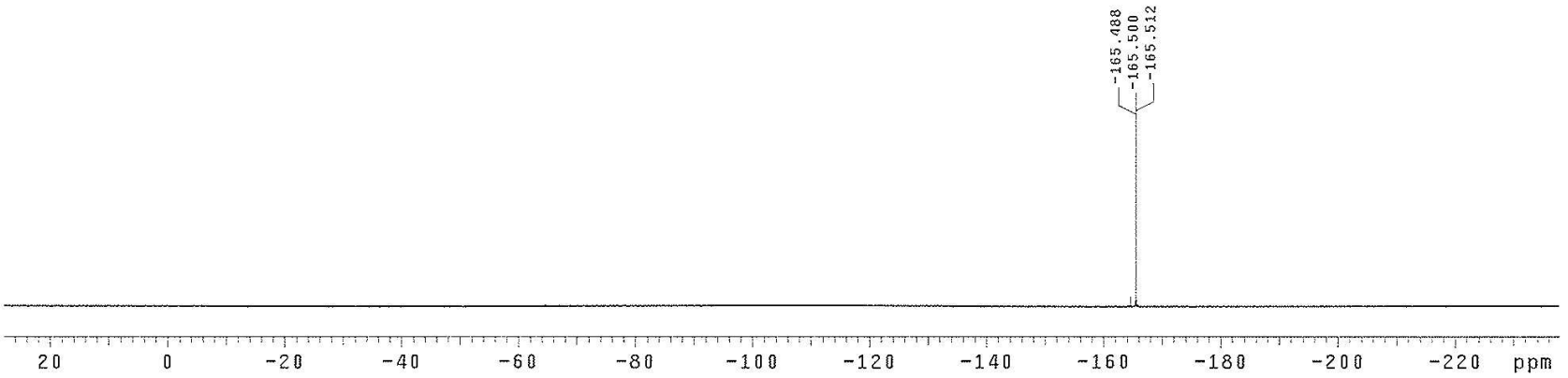


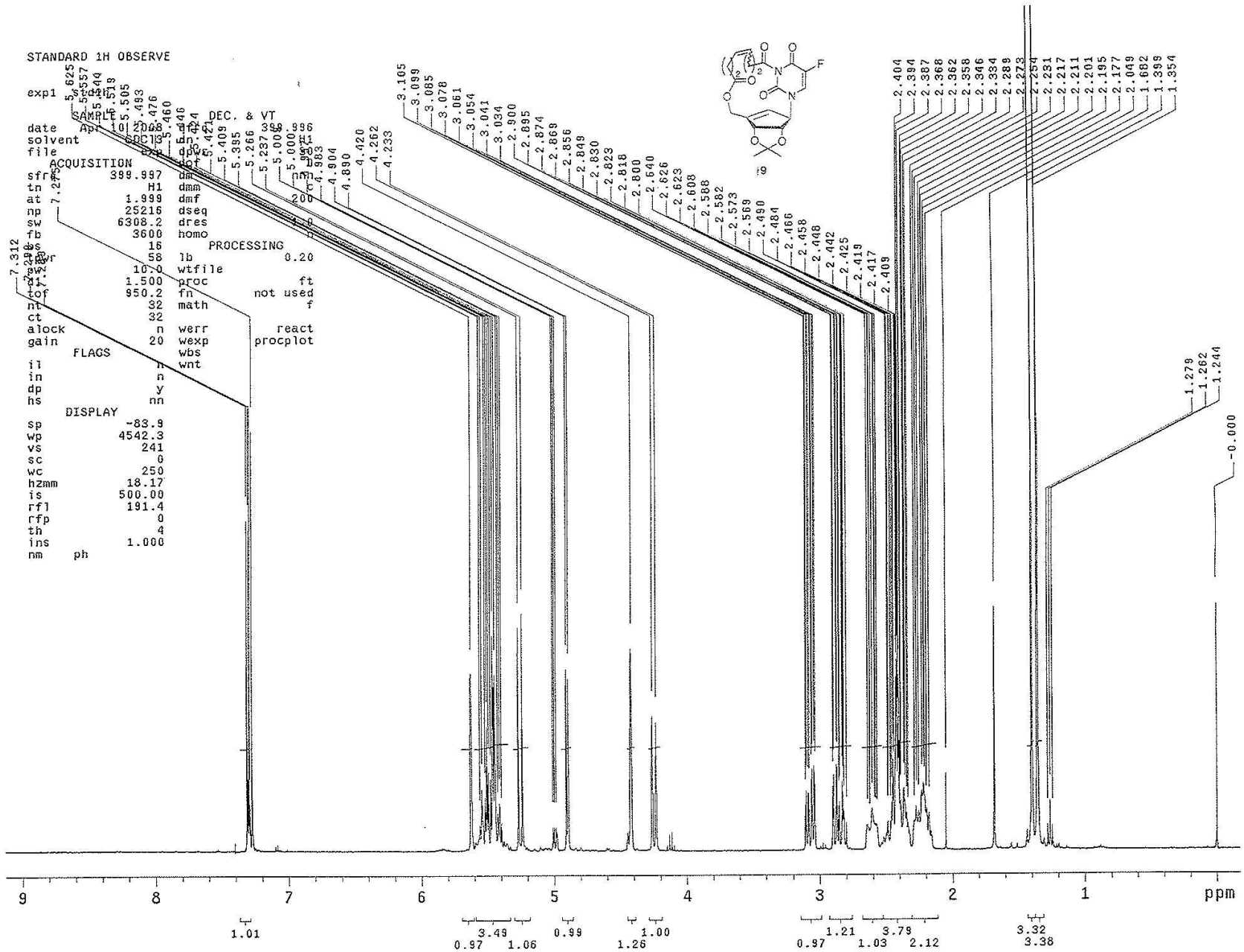
18

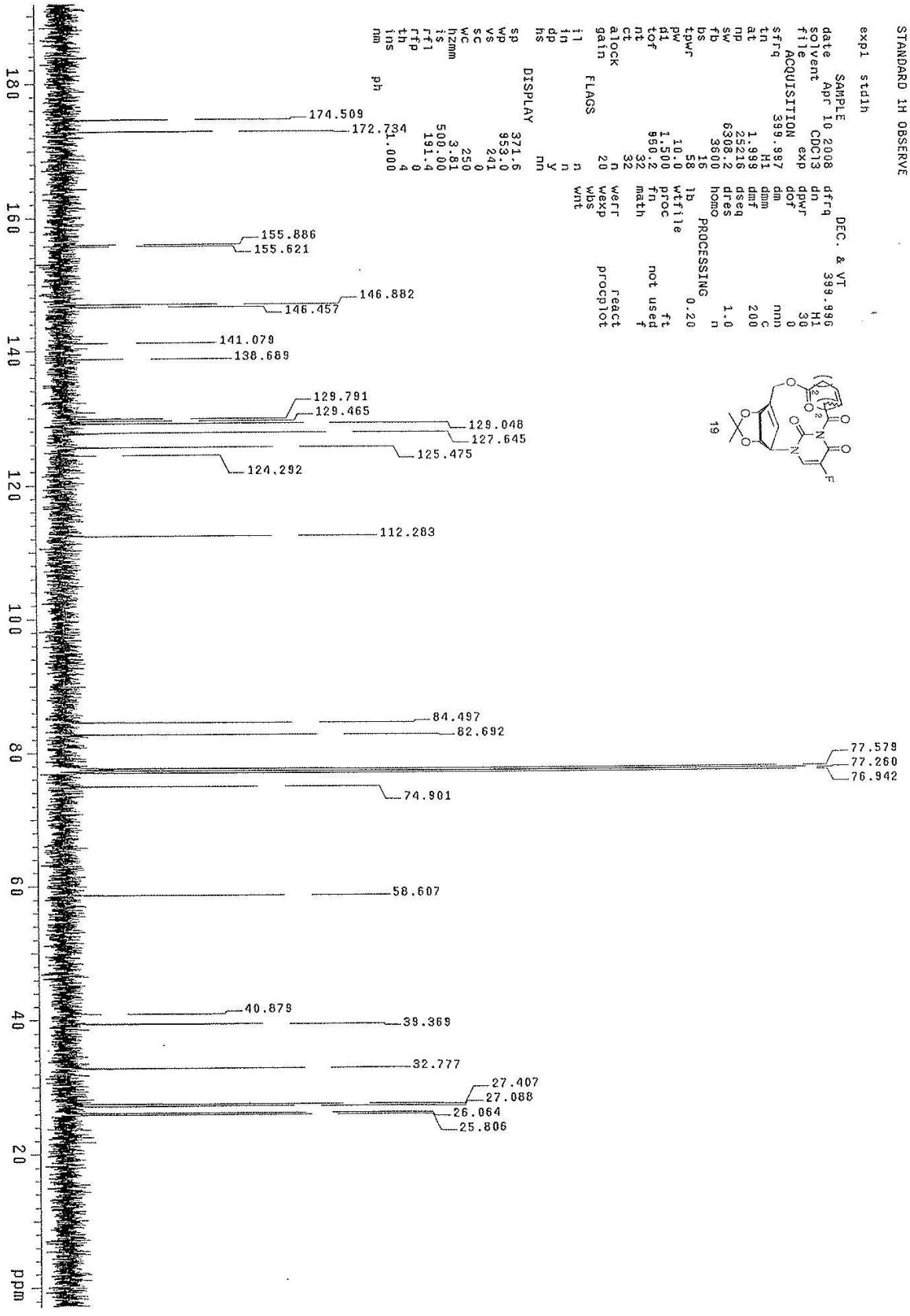
F19_STD

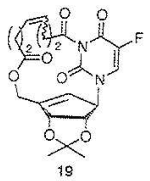
exp1 std1h

SAMPLE		DEC. & VT	
date	Nov 5 2012	dfrq	399.996
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	-208.0
sfrq	376.332	dm	nnn
tn	F19	dmm	c
at	0.640	dmf	200
np	128000	dseq	
sw	100000.0	dres	1.0
fb	51200	homo	n
bs	16	PROCESSING	
tpwr	58	lb	0.34
pw	12.0	wtfile	
d1	2.000	proc	ft
tof	950.2	fn	not used
nt	120	math	f
ct	48		
alock	n	werr	react
gain	20	wexp	procplot
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-89508.5		
wp	100000.0		
vs	1258		
sc	0		
wc	250		
hzmm	400.00		
is	500.00		
rfl	27219.0		
rfp	-62289.4		
th	5		
ins	100.000		
ai ph			




STANDARD 1H OBSERVE
exp1 stdn
SAMPLE DEC. & VT
date Apr 10 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 399.997
tn H1
at 1.999
np 25216
sw 6308.2
fb 3600
bs 16
pwr 58
pw 10.0
d1 1.500
tof 950.2
nt 32
ct 32
alock n
gain 20
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -83.9
wp 4542.3
vs 241
sc 0
wc 250
hzmm 18.17
is 500.00
rfl 191.4
rfp 0
th 4
ins 1.000
nm ph
PROCESSING
lb 0.20
wtfile
proc ft
fn not used
math f
werr react
wexp procplot
wbs
wnt
ppm
1.01
3.49
0.97
1.06
0.99
1.00
1.26
1.21
3.79
0.97
1.03
2.12
3.32
3.38


STANDARD 1H OBSERVE
exp1 std1h
SAMPLE
date Apr 10 2008
solvent CDCl3
file
ACQUISITION
sfrq 399.987
tn H1
at 1.999
np 25216
sw 6308.2
fb 3600
bs 16
tpwr 58
pw 10.0
d1 1.500
tof 950.2
nt 32
ct 32
alock n
gain 20
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 371.6
wp 953.0
vs 241
sc 0
wc 250
hzmm 3.81
is 500.00
rfl 191.4
rfp 0
th 4
ins 1.000
nm
ph
DEC. & VT
dfrq 399.995
dn H1
dpwr 30
dof 0
dm nnn
tn c
at 200
dmm
dmf
dseq
dres 1.0
homo n
PROCESSING
lb 0.20
wtfile
proc ft
fn not used
math f
werr
wexp react
wbs procplot
wnt
19
174.509
172.734
155.886
155.621
146.882
146.457
141.079
138.689
129.791
129.465
129.048
127.645
125.475
124.292
112.283
84.497
82.692
77.579
77.260
76.942
74.901
58.607
40.879
39.369
32.777
27.407
27.088
26.064
25.806
ppm



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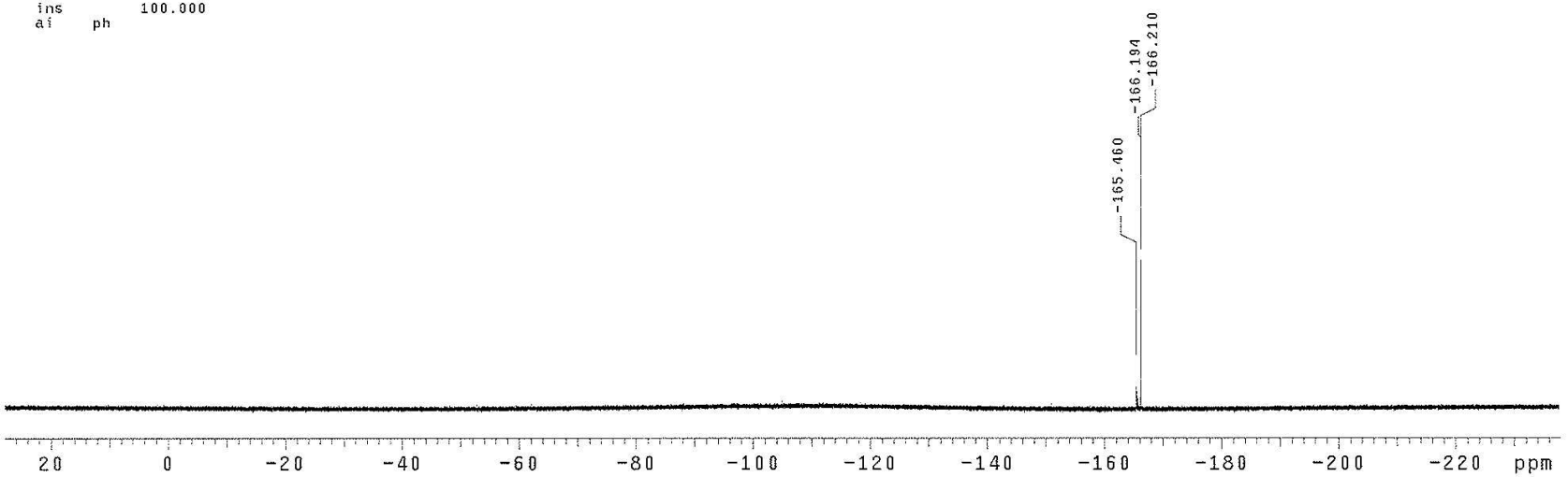
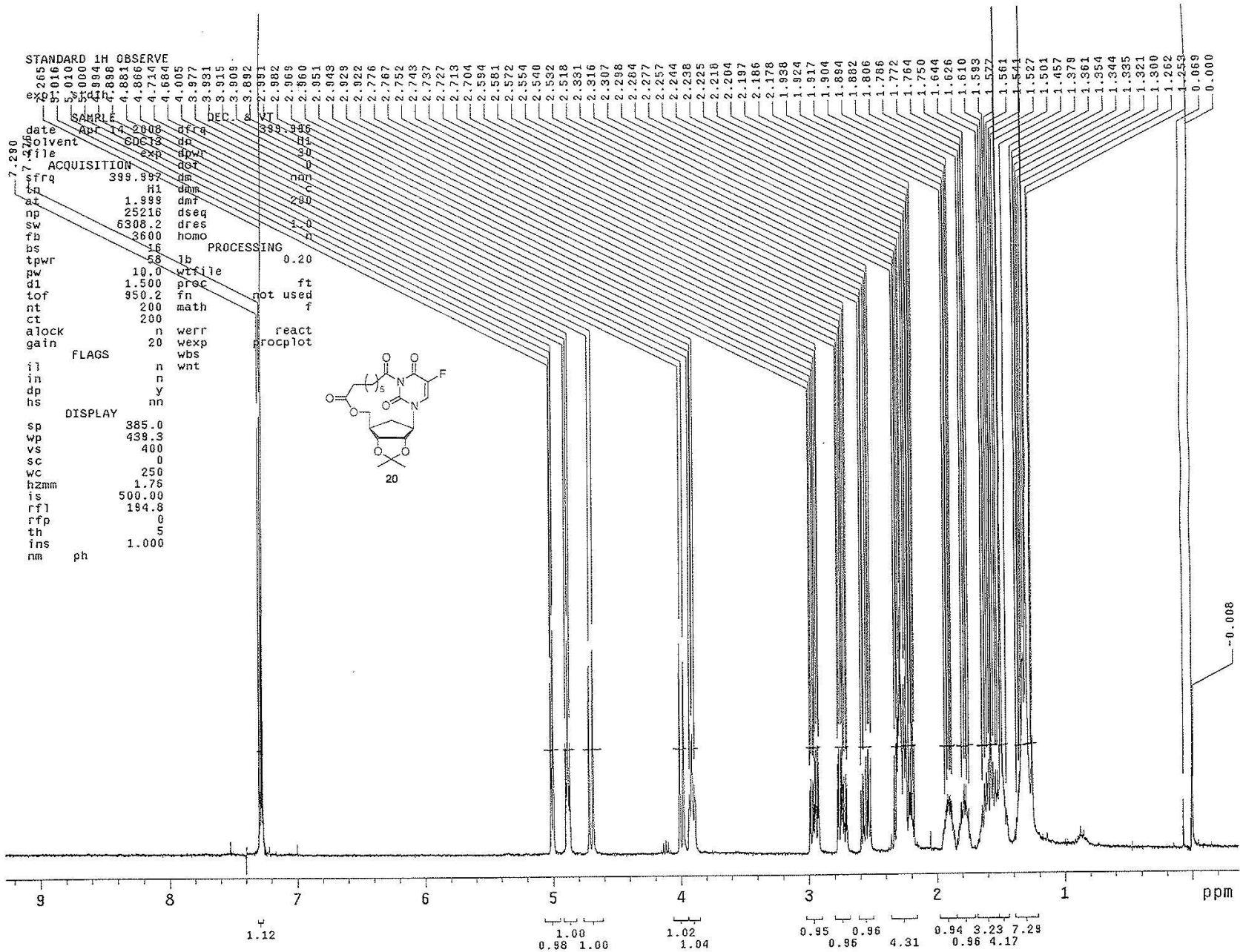
F19_STD

exp1 std1h

SAMPLE		DEC. & VT	
date	Nov 5 2012	dfrq	399.996
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	-208.0
sfrq	376.332	dm	nnn
tn	F19	dmm	c
at	0.640	dmf	200
np	128000	dseq	
sw	100000.0	dres	1.0
fb	51200	homo	n
bs	16	PROCESSING	
tpwr	58	lb	0.34
pw	12.0	wtfile	
d1	2.000	proc	ft
tof	950.2	fn	not used
nt	240	math	f
ct	32		
alock	n	werr	react
gain	20	wexp	procplot
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-89504.1		
wp	100000.0		
vs	3593		
sc	0		
wc	250		
hzmm	400.00		
is	500.00		
rfl	26947.4		
rfp	-62556.6		
th	2		
ins	100.000		
ai ph			



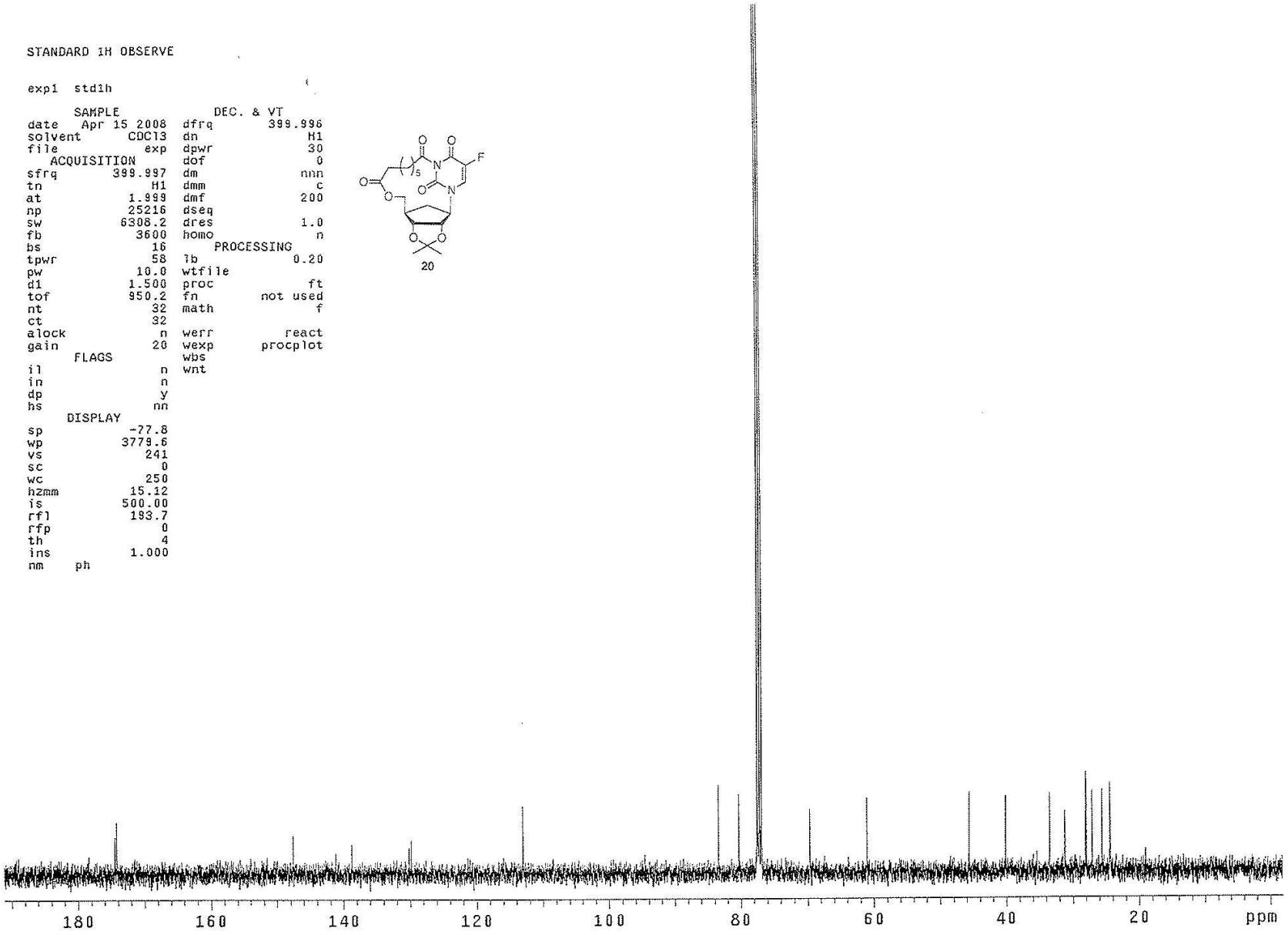

STANDARD 1H OBSERVE
SAMPLE
date Apr 14 2008
Solvent CDCl3
file exp
ACQUISITION
sfrq 399.997
tn H1
at 1.999
np 25216
sw 6308.2
fb 3600
bs 16
tpwr 58
pw 10.0
d1 1.500
tof 950.2
nt 200
ct 200
alock n
gain 20
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 385.0
wp 439.3
vs 400
sc 0
wc 250
hzmm 1.76
is 500.00
rfl 194.8
rfp 0
th 5
ins 1.000
nm ph
DEC. & VT
dfrq 399.996
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
lb 0.20
wtfile
proc ft
fn not used
math f
werr react
wexp procplot
wbs
wnt
20
ppm
1.12
1.00
0.98 1.00
1.02
1.04
0.95 0.96
0.96 4.31
0.94 3.23 7.29
0.96 4.17

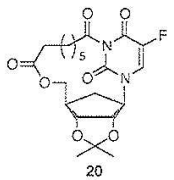
STANDARD 1H OBSERVE

exp1 std1h

SAMPLE		DEC. & VT	
date	Apr 15 2008	dfrq	399.996
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	399.997	dm	nnn
tn	H1	dmm	c
at	1.999	dmf	200
np	25216	dseq	
sw	6308.2	dres	1.0
fb	3600	homo	n
bs	16	PROCESSING	
tpwr	58	lb	0.20
pw	10.0	wtfile	
d1	1.500	proc	ft
tof	950.2	fn	not used
nt	32	math	f
ct	32		
alock	n	werr	react
gain	20	wexp	procplot
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-77.8		
wp	3779.6		
vs	241		
sc	0		
wc	250		
hzmm	15.12		
is	500.00		
rfl	193.7		
rfp	0		
th	4		
ins	1.000		
nm	ph		

20





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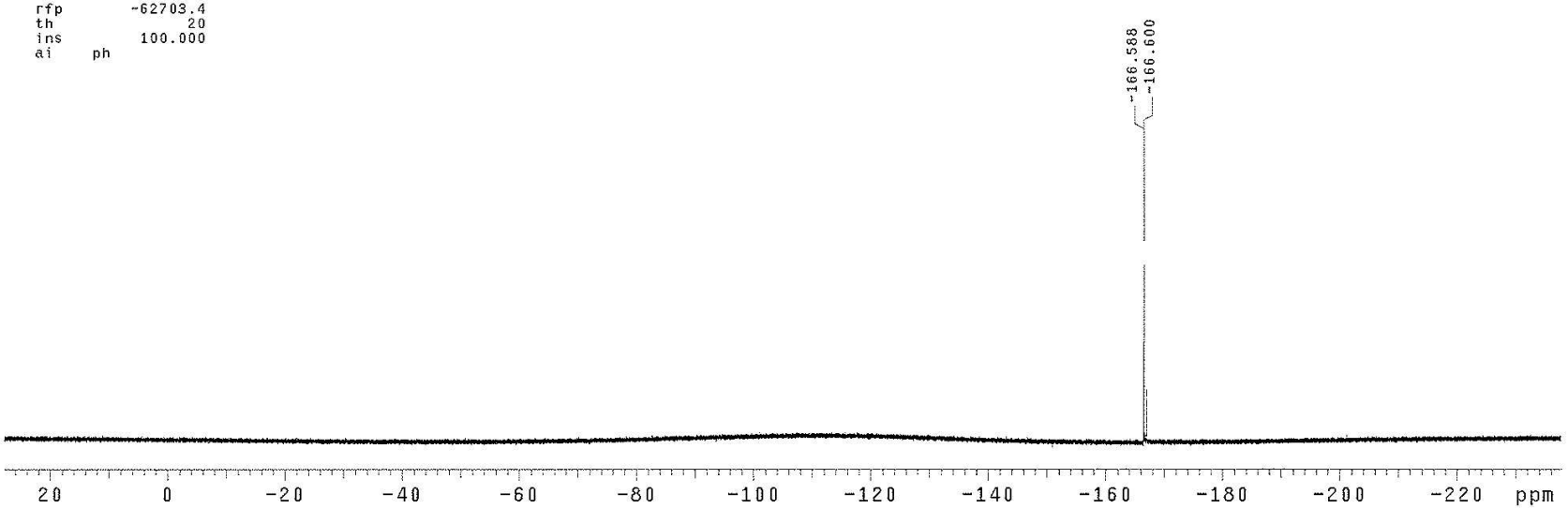
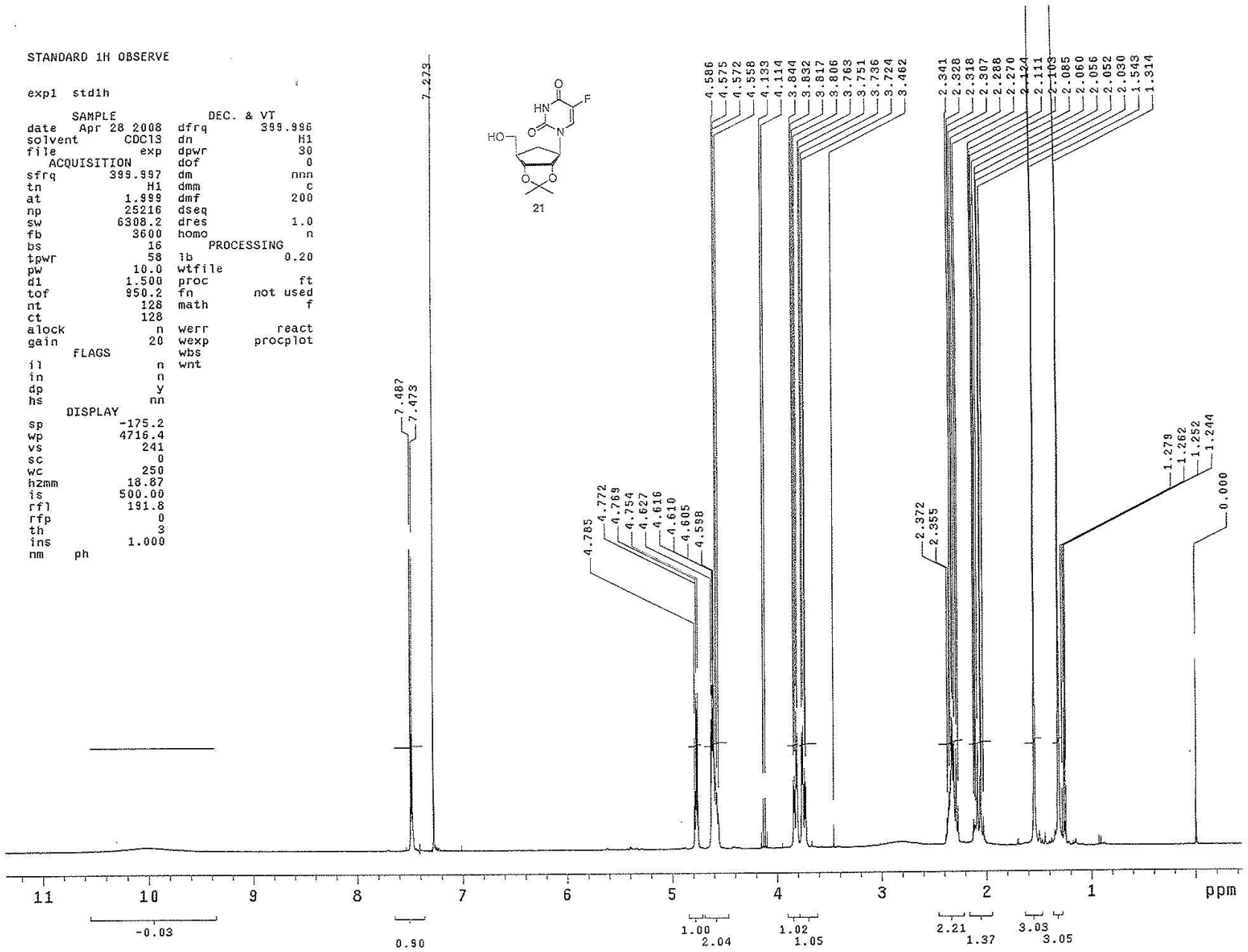
F19_STD

exp1 std1h

SAMPLE		DEC. & VT	
date	Nov 5 2012	dfrq	399.996
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	-208.0
sfrq	376.332	dm	nnn
tn	F19	dmm	c
at	0.640	dmf	200
np	128000	dseq	
sw	100000.0	dres	1.0
fb	51200	homo	n
bs	16	PROCESSING	
tpwr	58	lb	0.34
pw	12.0	wtfile	
d1	2.000	proc	ft
tof	950.2	fn	not used
nt	360	math	f
ct	112		
alock	n	werr	react
gain	20	wexp	procplot
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-89505.9		
wp	100000.0		
vs	7082		
sc	0		
wc	250		
hzmm	400.00		
is	500.00		
rfl	26802.5		
rfp	-62703.4		
th	20		
ins	100.000		
ai ph			

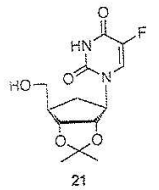



STANDARD 1H OBSERVE
exp1 std1h
SAMPLE
date Apr 28 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 399.997
tn H1
at 1.999
np 25216
sw 6308.2
fb 3600
bs 16
tpwr 58
pw 10.0
d1 1.500
tof 950.2
nt 128
ct 128
alock n
gain 20
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -175.2
wp 4716.4
vs 241
sc 0
wc 250
hzmm 18.87
is 500.00
rfl 191.8
rfp 0
th 3
ins 1.000
nm ph
DEC. & VT
dfrq 399.996
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
lb 0.20
wtfile
proc ft
fn not used
math f
werr react
wexp procplot
wbs
wnt
21
7.487
7.473
7.273
4.785
4.772
4.769
4.754
4.627
4.616
4.610
4.605
4.588
4.586
4.575
4.572
4.558
4.133
4.114
3.844
3.832
3.817
3.806
3.763
3.751
3.736
3.724
3.462
2.372
2.355
2.341
2.328
2.318
2.307
2.288
2.270
2.124
2.111
2.103
2.085
2.060
2.056
2.052
2.030
1.543
1.314
1.279
1.262
1.252
1.244
0.000
11
10
9
8
7
6
5
4
3
2
1
ppm
-0.03
0.90
1.00
2.04
1.02
1.05
2.21
1.37
3.03
3.05

13C OBSERVE

exp1 std13c

SAMPLE		DEC. & VT	
date	Apr 28 2008	dfrq	399.996
solvent	CDCl3	dn	H1
file	exp	dpwr	38
ACQUISITION		dof	-200.0
sfrq	100.588	dm	yyy
tn	C13	dmm	w
at	1.199	dmf	9000
np	59968	dseq	
sw	25000.0	dres	1.0
fb	13800	homo	n
bs	16	PROCESSING	
tpwr	58	lb	1.00
pw	7.0	wtfile	
d1	3.000	proc	ft
tof	0	fn	not used
nt	800	math	f
ct	720		
alock	n	werr	react
gain	50	wexp	procplot
FLAGS		wbs	testsn
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	2040.9		
wp	2923.7		
vs	88		
sc	0		
wc	250		
hzmm	11.69		
is	500.00		
rfl	10752.4		
rfp	7767.7		
th	4		
ins	100.000		
nm	ph		



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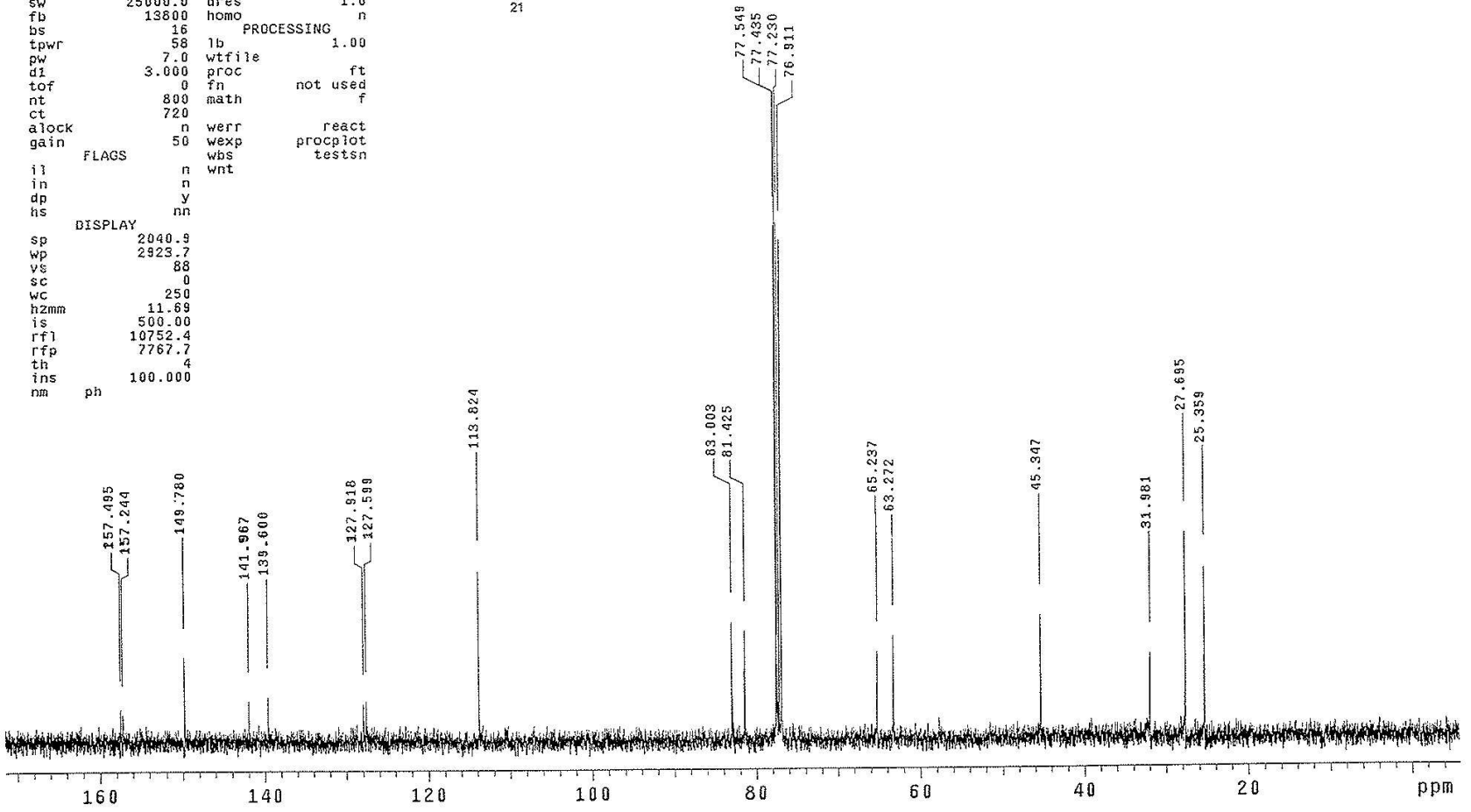

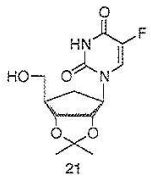

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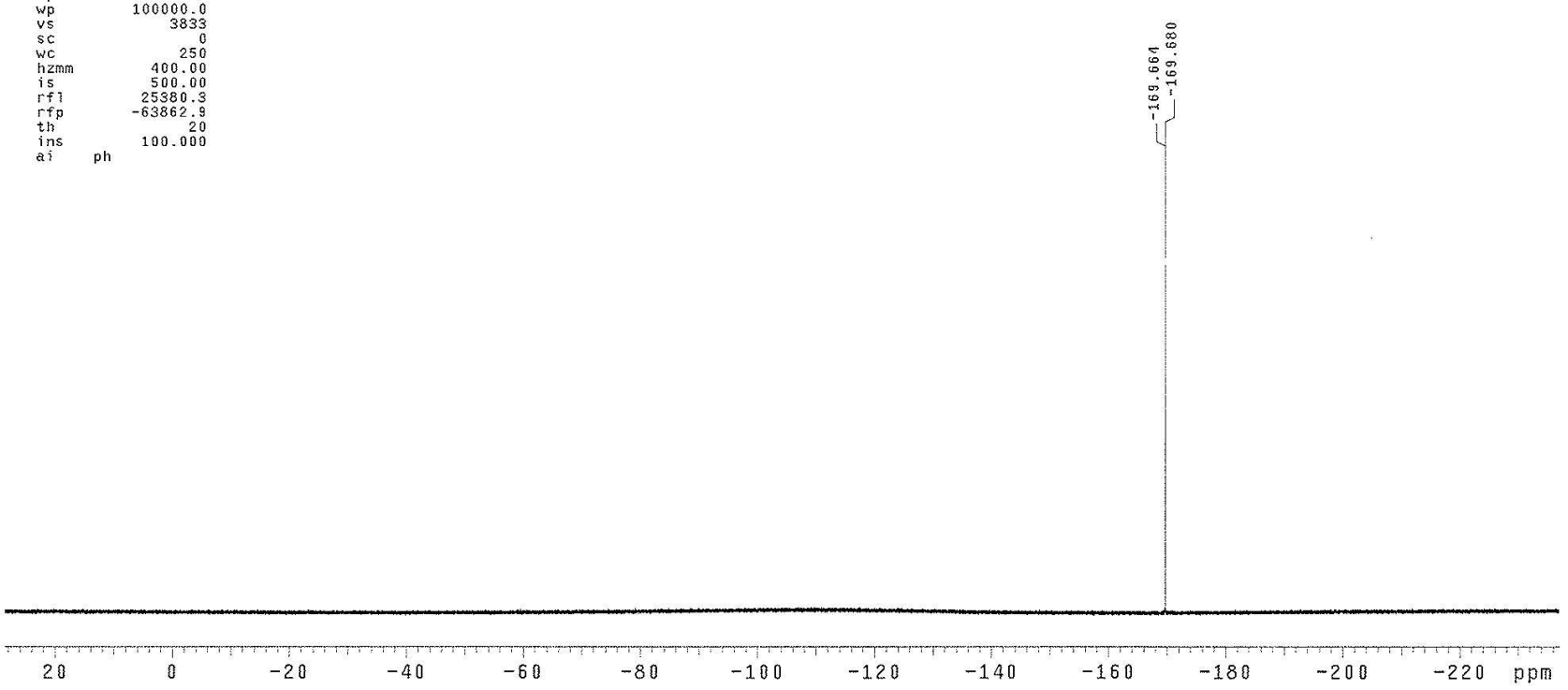
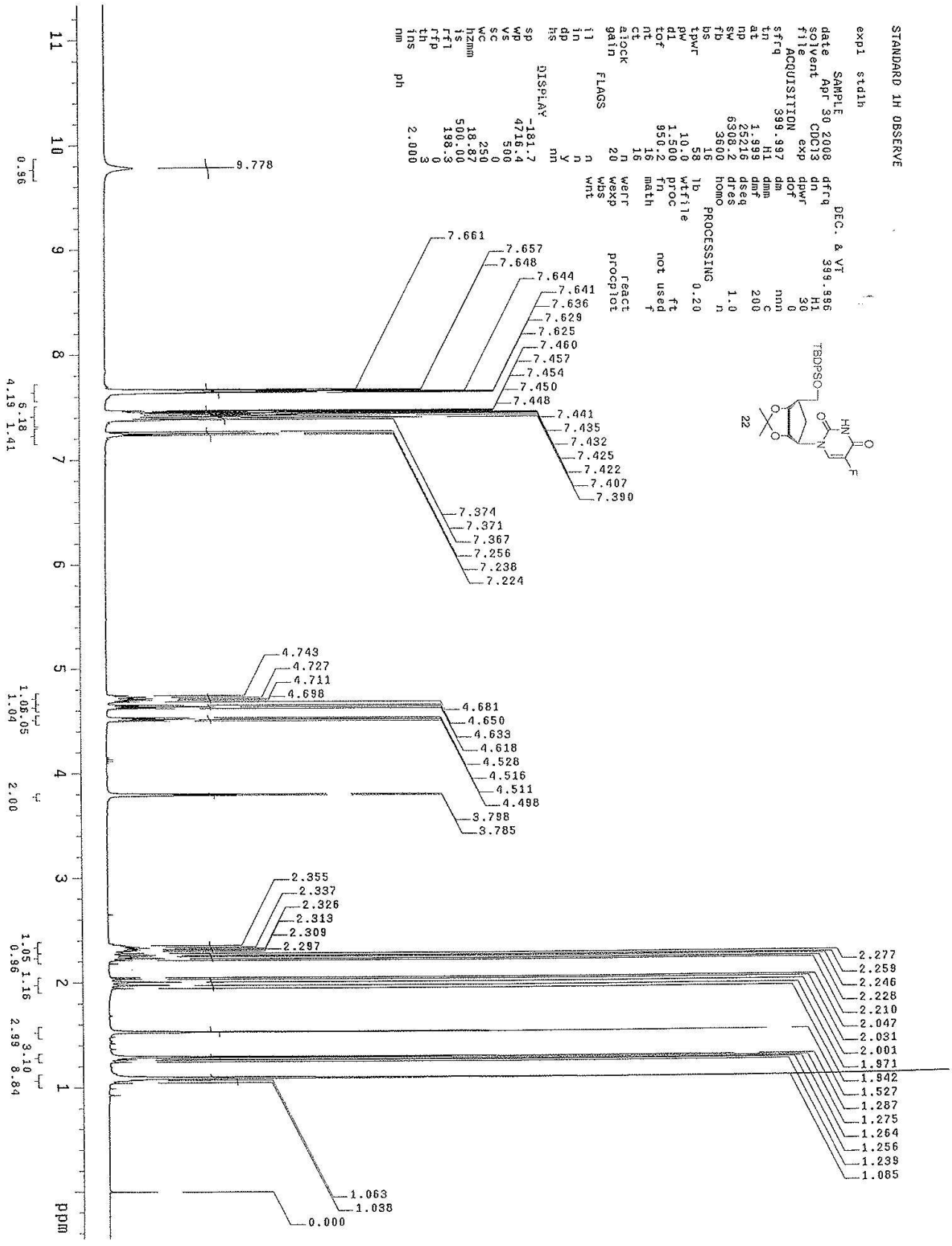
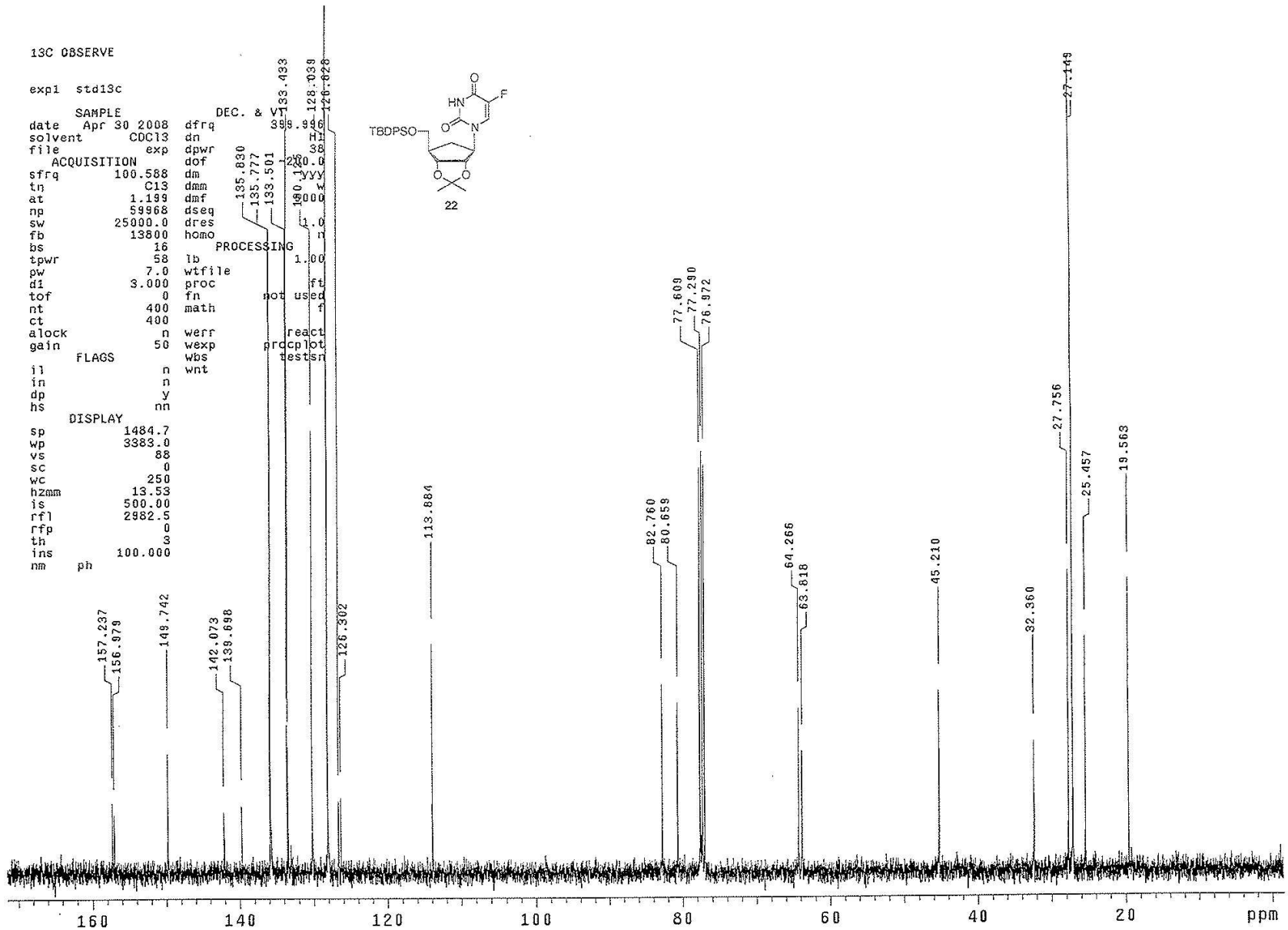
F19_STD

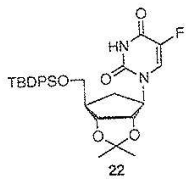
exp1 std1h

SAMPLE		DEC. & VT	
date	Nov 5 2012	dfrq	399.998
solvent	CD3OD	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	-208.0
sfrq	376.333	dm	nnn
tn	F19	dmm	c
at	0.640	dmf	200
np	128000	dseq	
sw	100000.0	dres	1.0
fb	51200	homo	n
bs	16	PROCESSING	
tpwr	58	lb	0.34
pw	12.0	wtfile	
d1	2.000	proc	ft
tof	950.2	fn	not used
nt	48	math	f
ct	48		
alock	n	werr	react
gain	20	wexp	procplot
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-89243.2		
wp	100000.0		
vs	3833		
sc	0		
wc	250		
hzmm	400.00		
is	500.00		
rfl	25380.3		
rfp	-63862.9		
th	20		
ins	100.000		
ai ph			




STANDARD 1H OBSERVE
expl stdih
SAMPLE
date Apr 30 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 399.997
tn H1
at 1.999
np 25216
sw 6308.2
fb 3600
bs 16
tpwr 58
pw 10.0
d1 1.500
tof 950.2
nt 16
ct 16
alock n
gain 20
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -181.7
wp 4716.4
vs 500
sc 0
wc 250
hzmm 18.87
is 500.00
rfl 198.3
rfp 0
th 3
ins 2.000
nm
ph
DEC. & VT
dfrq 399.996
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
lb 0.20
wtfile
proc ft
fn not used
math f
werr
wexp react
wbs procplot
wnt
TBDPSO
HN
O
O
O
N
F
22
9.778
7.661
7.657
7.648
7.644
7.641
7.636
7.629
7.625
7.460
7.457
7.454
7.450
7.448
7.441
7.435
7.432
7.425
7.422
7.407
7.390
7.374
7.371
7.367
7.256
7.238
7.224
4.743
4.727
4.711
4.698
4.681
4.650
4.633
4.618
4.528
4.516
4.511
4.498
3.798
3.785
2.355
2.337
2.326
2.313
2.309
2.297
2.277
2.259
2.246
2.228
2.210
2.047
2.031
2.001
1.971
1.942
1.527
1.287
1.275
1.264
1.256
1.239
1.085
1.063
1.038
0.000
0.96
6.18
4.19 1.41
1.05 1.05
1.04
2.00
1.05 1.16
0.96
2.99 3.10 8.84
ppm


13C OBSERVE
exp1 std13c
SAMPLE
date Apr 30 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 100.588
tn C13
at 1.199
np 59968
sw 25000.0
fb 13800
bs 16
tpwr 58
pw 7.0
d1 3.000
tof 0
nt 400
ct 400
alock n
gain 50
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 1484.7
wp 3383.0
vs 88
sc 0
wc 250
hzmm 13.53
is 500.00
rfl 2982.5
rfp 0
th 3
ins 100.000
nm ph
DEC. & VT
dfrq 399.996
dn H1
dpwr 38
dof -200.0
dm yyy
dmm w
dmf 1000
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
werr react
wexp procplot
wbs testsn
wnt
TBDPSO
22
157.237
156.979
149.742
142.073
139.698
135.830
135.777
133.501
133.433
128.039
126.828
126.302
113.884
82.760
80.659
77.609
77.290
76.972
64.266
63.818
45.210
32.360
27.756
27.149
25.457
19.563
160
140
120
100
80
60
40
20
ppm



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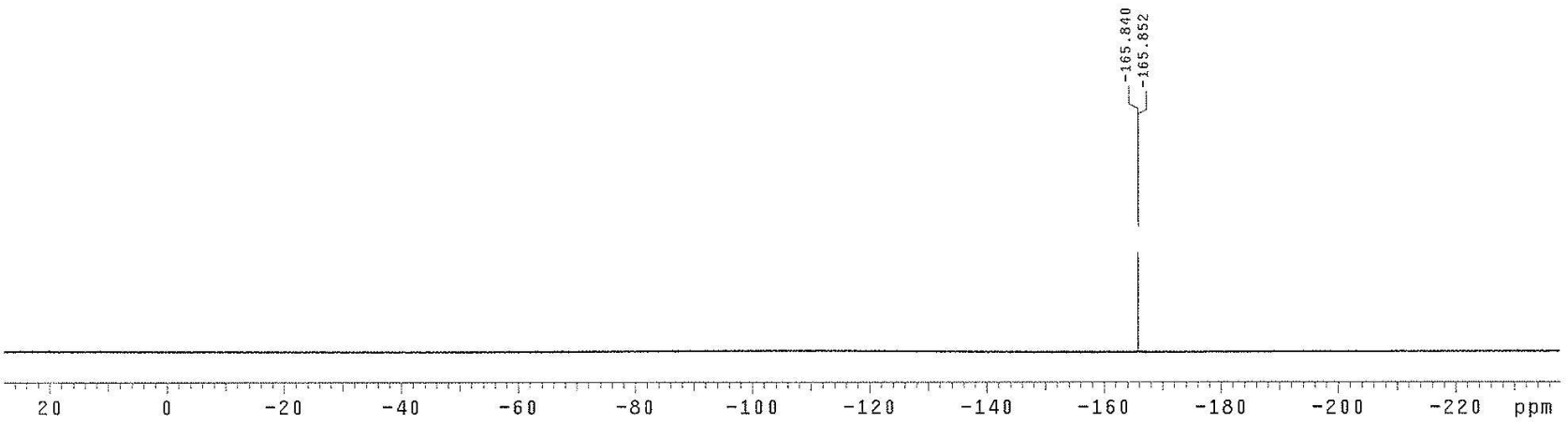
F19_STD

exp1 std1h

SAMPLE		DEC. & VT	
date	Nov 5 2012	dfrq	399.996
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	-208.0
sfrq	376.332	dm	nnn
tn	F19	dmm	c
at	0.640	dmf	200
np	128000	dseq	
sw	100000.0	dres	1.0
fb	51200	homo	n
bs	16	PROCESSING	
tpwr	58	lb	0.34
pw	12.0	wtfile	
d1	2.000	proc	ft
tof	950.2	fn	not used
nt	120	math	f
ct	48		
alock	n	werr	react
gain	20	wexp	procplot
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-89512.8		
wp	100000.0		
vs	1691		
sc	0		
wc	250		
hzmm	400.00		
is	500.00		
rfl	27095.4		
rfp	-62417.4		
th	5		
ins	100.000		
ai ph			

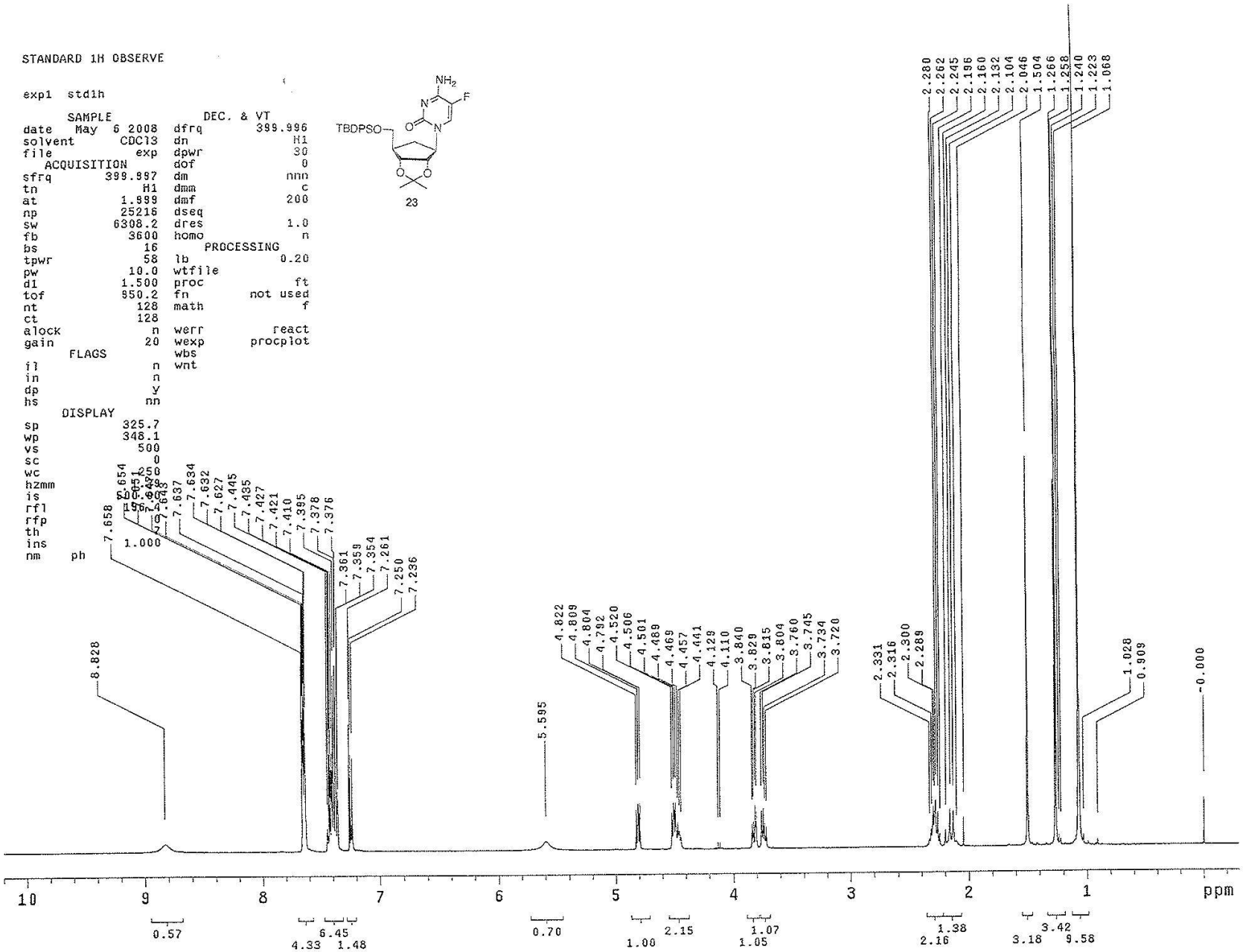


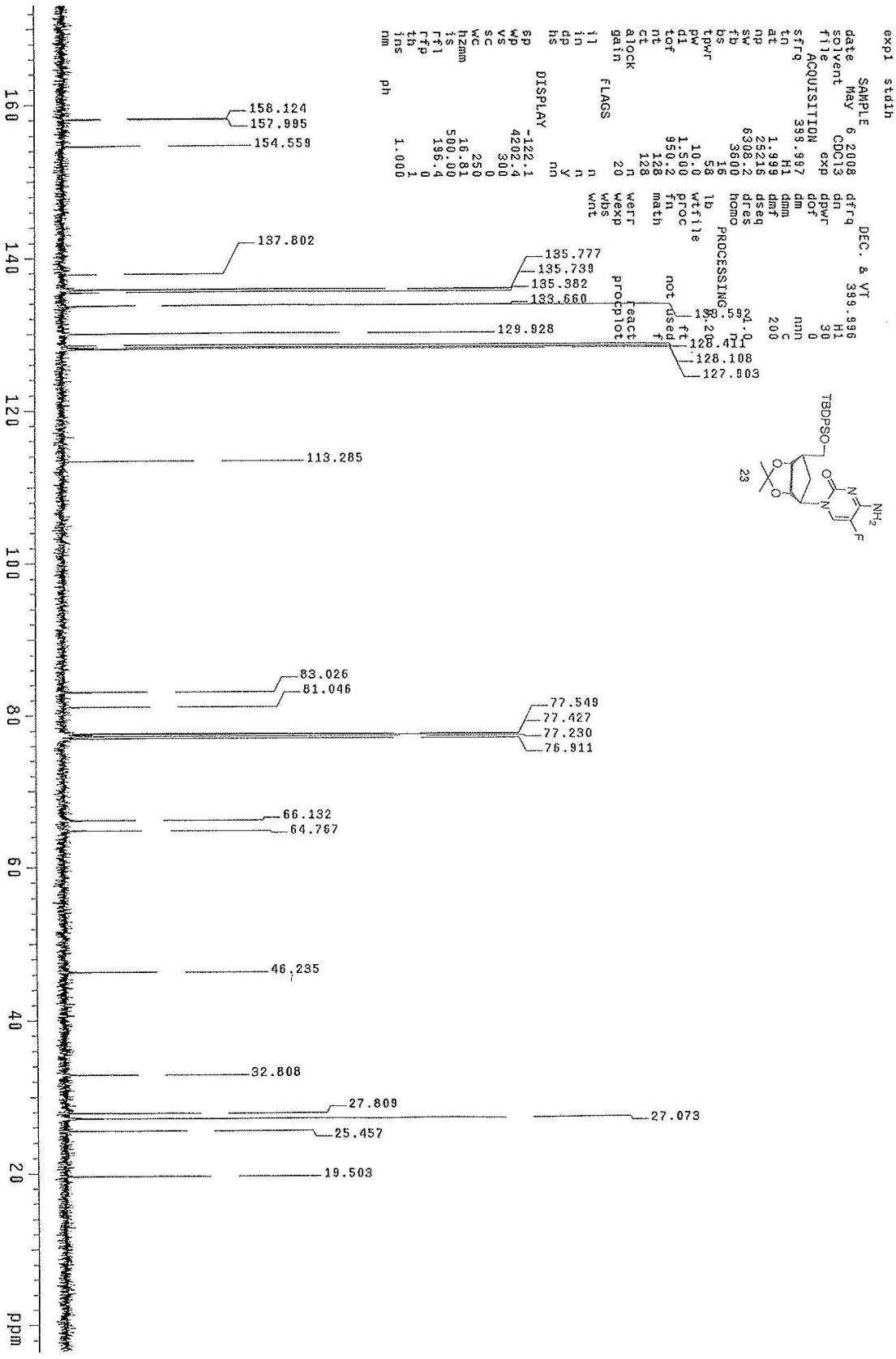
STANDARD 1H OBSERVE

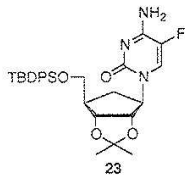
exp1 std1h

SAMPLE		DEC. & VT	
date	May 6 2008	dfrq	399.996
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	399.997	dm	nnn
tn	H1	dmm	c
at	1.999	dmf	200
np	25216	dseq	
sw	6308.2	dres	1.0
fb	3600	homo	n
bs	16	PROCESSING	
tpwr	58	lb	0.20
pw	10.0	wtfile	
d1	1.500	proc	ft
tof	950.2	fn	not used
nt	128	math	f
ct	128		
alock	n	werr	react
gain	20	wexp	procplot
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	325.7		
wp	348.1		
vs	500		
sc	0		
wc	250		
hzmm			
is			
rfl			
rfp			
th			
ins	1.000		
nm	ph		

23




STANDARD 1H OBSERVE
exp1 stdih
SAMPLE
date May 6 2008
solvent CDCl3
file
ACQUISITION
sfrq 399.997
tn H1
at 1.999
np 25216
sw 6308.2
fb 3600
bs 16
tpwr 58
pw 10.0
d1 1.500
tof 950.2
nt 128
ct 128
alock n
gain 20
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -122.1
wp 4202.4
vs 300
sc 0
wc 250
hzmm 16.81
is 500.00
rfl 196.4
rfp 0
th 1
ins 1.000
nm
ph
dfrq DEC. & VT
dn 399.996
dpwr H1
dof 30
dm 0
tn nnn
at c
np 200
dmf
dseq
dres
homo
PROCESSING
lb
wtfile
proc
fn
math
werr
wexp
wbs
wnt
proplot
not used
ft
f
158.124
157.995
154.559
137.802
135.777
135.739
135.382
133.660
129.928
113.285
83.026
81.046
77.549
77.427
77.230
76.911
66.132
64.767
46.235
32.808
27.809
27.073
25.457
19.503
TBDPSO
23
NH2
F
160
140
120
100
80
60
40
20
ppm



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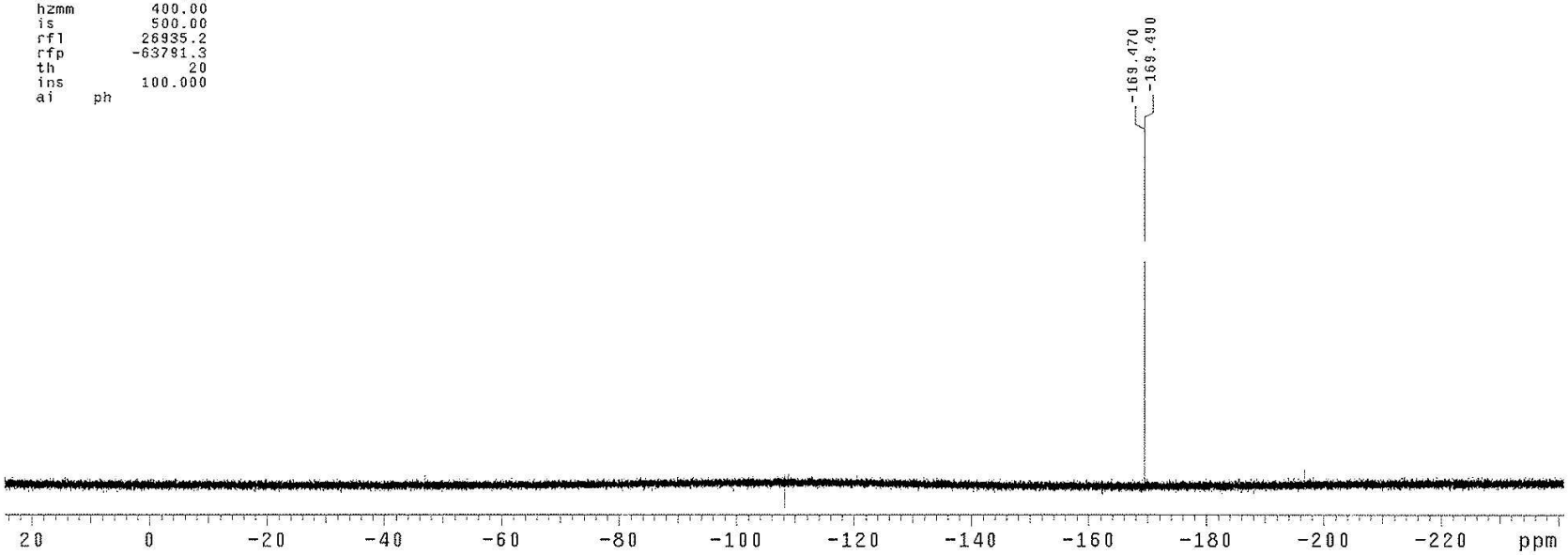
F19_STD

exp1 std1h

SAMPLE		DEC. & VT	
date	Nov 5 2012	dfrq	399.996
solvent	cdcl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	-208.0
sfrq	376.332	dm	nnn
tn	F19	dmm	c
at	0.640	dmf	200
np	128000	dseq	
sw	100000.0	dres	1.0
fb	51200	homo	n
bs	16	PROCESSING	
tpwr	58	lb	0.34
pw	12.0	wtfile	
d1	2.000	proc	ft
tof	950.2	fn	not used
nt	120	math	f
ct	120		
alock	n	werr	react
gain	2	wexp	procplot
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-90726.6		
wp	100000.0		
vs	36081		
sc	0		
wc	250		
hzmm	400.00		
is	500.00		
rfl	26935.2		
rfp	-63791.3		
th	20		
ins	100.000		
ai ph			



STANDARD 1H OBSERVE

exp1 std1h

SAMPLE		DEC. & VT	
date	May 8 2008	dfrq	399.988
solvent	DMSO	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	399.989	dm	nnn
tn	H1	dmm	c
at	1.999	dmf	200
np	25216	dseq	
sw	6308.2	dres	1.0
fb	3600	homo	n
bs	16	PROCESSING	
tpwr	58	lb	0.20
pw	10.0	wtfile	
d1	1.500	proc	ft
tof	950.2	fn	not used
nt	64	math	f
ct	64		
alock	n	werr	react
gain	20	wexp	procplot
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-48.5		
wp	4558.9		
vs	129		
sc	0		
wc	250		
hzmm	18.24		
is	500.00		
rfl	1189.0		
rfp	1000.0		
th	5		
ins	1.000		
nm ph			

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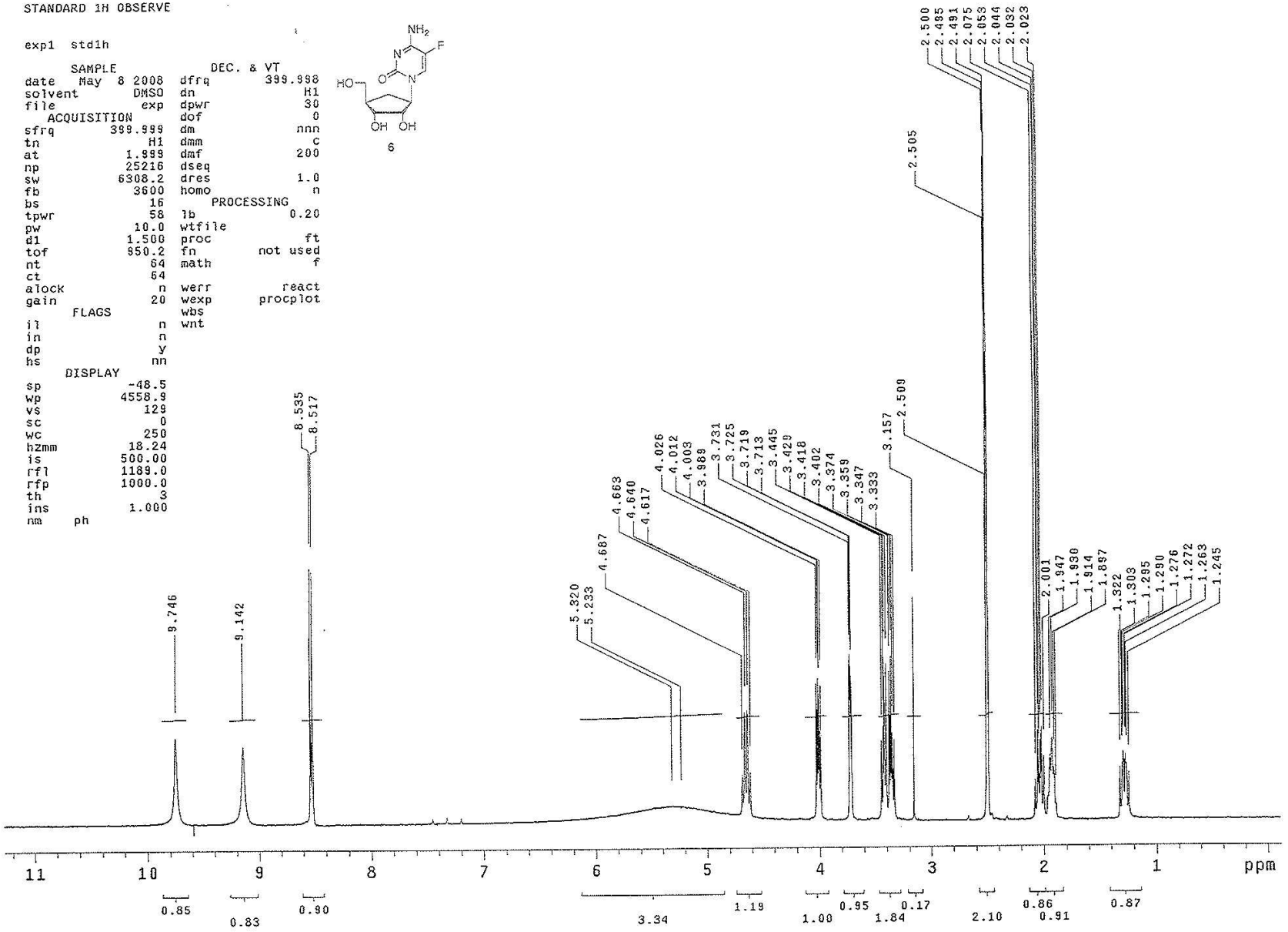

13C OBSERVE

exp1 std13c

SAMPLE		DEC. & VT	
date	May 8 2008	dfrq	399.998
solvent	DMSO	dn	H1
file	exp	dpwr	38
ACQUISITION		dof	-200.0
sfrq	100.589	dm	yyy
tn	C13	dmm	w
at	1.199	dmf	9000
np	59968	dseq	
sw	25000.0	dres	1.0
fb	13800	homo	n
bs	16	PROCESSING	
tpwr	58	lb	1.00
pw	7.0	wtfile	
d1	3.000	proc	ft
tof	0	fn	not used
nt	12000	math	f
ct	12000		
alock	n	werr	react
gain	50	wexp	procplot
FLAGS		wbs	testsn
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-190.7		
wp	16688.3		
vs	225		
sc	0		
wc	250		
hzmm	66.75		
is	500.00		
rfl	2982.4		
rfp	0		
th	4		
ins	100.000		
nm	ph		

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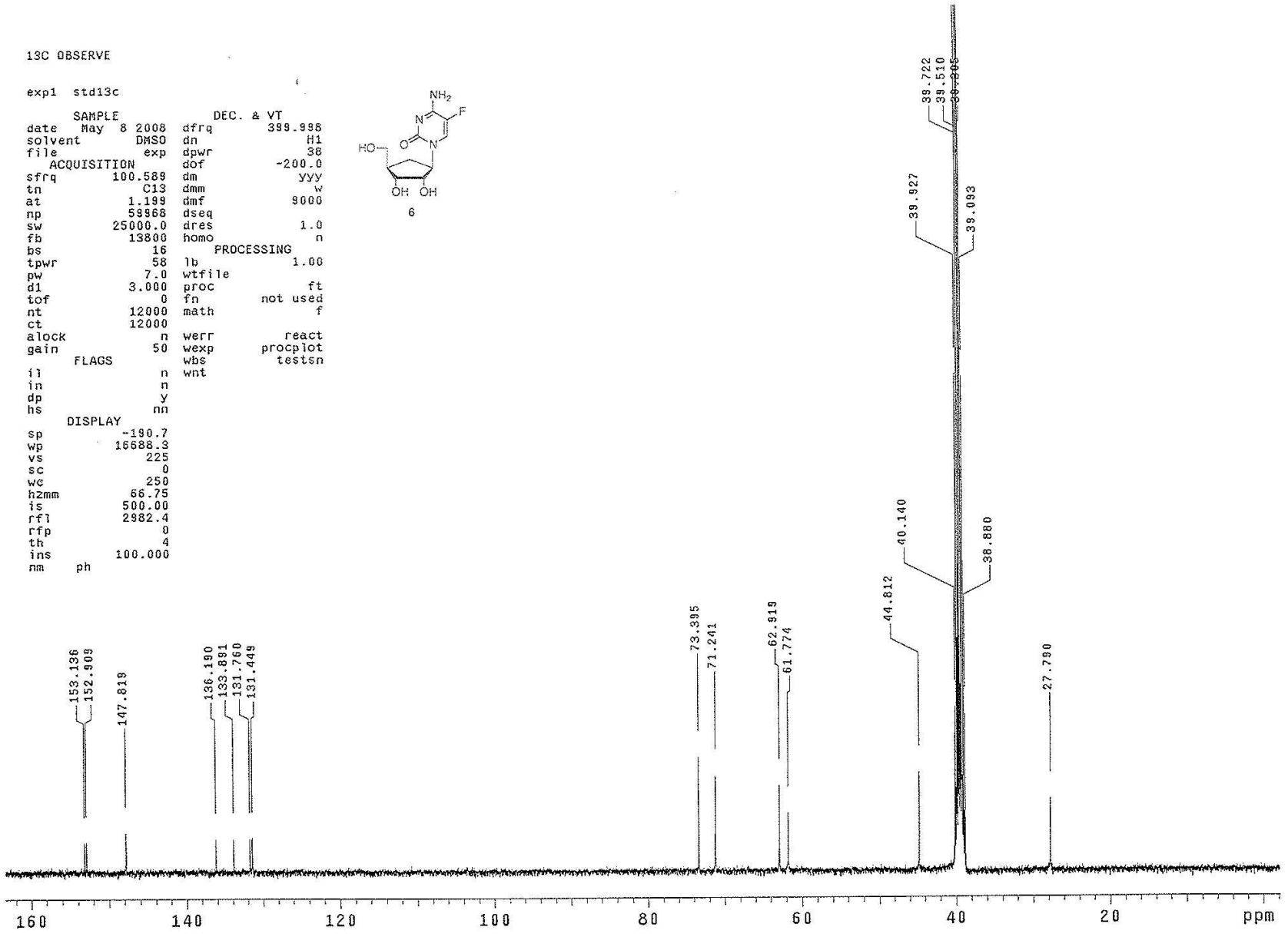
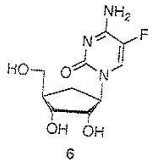
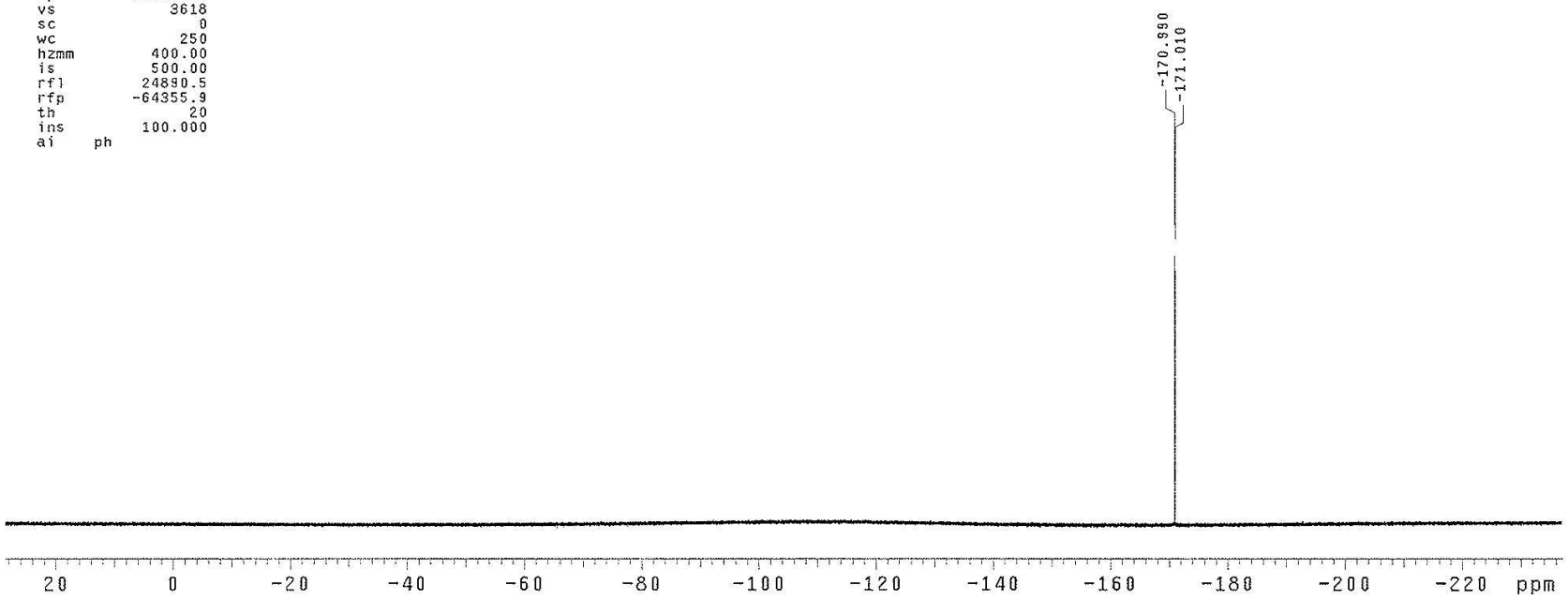


F19_STD

exp1 std1h

SAMPLE		DEC. & VT	
date	Nov 5 2012	dfrq	399.998
solvent	CD3OD	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	-208.0
sfrq	376.333	dm	nnn
tn	F19	dmm	c
at	0.640	dmf	200
np	128000	dseq	
sw	100000.0	dres	1.0
fb	51200	homo	n
bs	16	PROCESSING	
tpwr	58	lb	0.34
pw	12.0	wtfile	
d1	2.000	proc	ft
tof	950.2	fn	not used
nt	48	math	f
ct	48		
alock	n	werr	react
gain	20	wexp	procplot
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-89246.4		
wp	100000.0		
vs	3618		
sc	0		
wc	250		
hzmm	400.00		
is	500.00		
rfl	24890.5		
rfp	-64355.9		
th	20		
ins	100.000		
ai ph			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