Cyclization of Oxa-Bicyclic Alkenes with β-Iodo-(Z)-propenoates and *o*-Iodobenzoate Catalyzed by Nickel Complexes: A Simple Efficient Route to Annulated Coumarins

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Supporting Information

General Procedure for the Cyclization of 7-Oxabenzonorbornadienes (1) with Methyl 2-Iodobenzoate (2). A round-bottom side-arm flask (25 mL) containing **1** (2.00 mmol), $NiBr_2$(dppe) (0.0500 mmol) and zinc powder (0.180 g, 2.75 mmol) was evacuated and purged with nitrogen gas three times. Freshly distilled CH_3CN (3.0 mL) and **2** (1.0 mmol) were added. The system was heated with stirring at 80 °C for 12 h and was then cooled to ambient temperature. After dilution with dichloromethane (15 mL), the reaction mixture was stirred in the air for 15 min, filtered through a thin Celite and silica gel pad and washed with dichloromethane several times. The filtrate was concentrated and the residue was purified on a silica gel column using hexane-ethyl acetate as eluent to afford the desired cyclization products **3**. Important spectral data of these products are listed below.

6*H*-Dibenzo[*c*,*h*]chromen-6-one (3a). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.52-7.58 (3 H, m), 7.67 (1 H, d, *J* = 9 Hz), 7.78 (1 H, d, *J* = 6 Hz), 7.79 (1 H, d, *J* = 7.5 Hz), 7.95 (1 H, d, *J* = 9 Hz), 8.08 (1H, d, *J* = 8 Hz), 8.38 (1H, d, *J* = 7.5 Hz), 8.49 (1H, d, J = 7.5 Hz); ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ =112.8, 119.0, 121.0, 121.9, 122.1, 123.7, 124.3, 126.9, 127.5, 127.7, 128.4, 130.4, 134.1, 134.8, 135.2, 147.0, 161.1; HRMS (m/e): calcd for $C_{17}H_{10}O_2$ 246.0681, found 246.0678

2,3-Dimethoxy-6*H*-dibenzo[*c*,*h*]chromen-6-one (3b). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 4.01 (3H, s), 4.08 (3H, s), 7.13 (1H, s), 7.55 (1H, td, *J* = 7 Hz, *J* = 1 Hz), 7.60 (1H, d, *J* = 8.5 Hz), 7.78 (1H, s), 7.84 (1H, td, *J* = 8 Hz, J=1 Hz), 7.89 (1H, d, *J* = 8.5 Hz), 8.14 (1H, d, *J* = 7.5 Hz), 8.43 (1H, dd, *J* = 4Hz, *J* = 1 Hz); ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ = 55.9, 56.3, 101.0, 106.4, 111.9, 117.5, 119.0 120.6, 121.7, 122.9, 128.1, 130.4, 130.5, 134.7, 146.4, 150.2, 150.9, 161.5; HRMS (m/e): calcd for $C_{19}H_{14}O_4$ 306.0892, found 306.0894.

12-Methyl-6*H*-dibenzo[*c*,*h*]chromen-6-one (3c). ^{1}H NMR (500 MHz, $CDCl_3$): δ

= 2.64 (3H, s), 7.50 (1H, td, *J* = 1.0 Hz, *J* = 8.5 Hz), 7.54-7.58 (2H, m), 7.70 (1H, s), 7.76 (1H, td, *J* = 1.0 Hz, *J* = 8.0 Hz), 7.86-7.89 (1H, m), 8.04 (1H, d, *J* = 8.5 Hz), 8.35 (1H, dd, *J* = 1.0 Hz, *J* = 7.5 Hz), 8.47-8.50 (1H, m); $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$): δ = 19.3, 112.2, 119.0, 121.0, 121.7, 122.5, 123.7, 124.0, 126.5, 127.5, 128.3, 130.4, 130.6, 133.2, 134.7, 135.1, 145.7, 161.2; HRMS (m/e): calcd for $C_{18}H_{12}O_2$ 260.0837, found 260.0832.

14*H*-Benzo[*c*]naphtho[2,3-*h*]chromen-14-one (3e). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.55-7.52 (2H, m), 7.60 (1H, *t*, *J* = 7.2 Hz), 7.91-7.85 (2H, m), 8.00 (2H, t, *J* = 7 Hz), 8.12-8.09 (1H, m), 8.18 (1H, d, *J* = 8 Hz), 8.41 (1H, s), 8.48 (1H, d, *J* = 7.6 Hz), 9.16 (1H, s); $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$): δ = 111.4, 118.3, 121.0, 121.7, 121.9, 122.3, 124.9, 126.1, 126.5, 127.9, 128.4, 128.8, 130.5, 131.4, 131.9, 132.5, 134.9, 135.3, 147.3, 161.3; HRMS (m/e): calcd for $C_{21}H_{12}O_2$ 296.0837, found 296.0841.

2*H*-Benzo[*h*]chromen-2-one (3g). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 6.45 (1H, d, *J* = 9 Hz), 7.38 (1H, d, *J* = 8 Hz), 7.62-7.57 (3H, m), 7.75 (1H, *J* = 8 Hz), 7.81 (1H, dd, *J* = 4.5 Hz, *J* = 1 Hz), 8.46 (1H, dd, *J* = 4.5 Hz, J = *1* Hz); $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$): δ = 114.1, 11.5, 122.7, 122.9, 123.4,127.0, 127.7, 128.6, 134.7, 144.1, 151.1, 160.8; HRMS (m/e): calcd for $C_{13}H_8O_2$ 196.0524, found 196.0521

8,9-Di(methyloxy)-2*H*-benzo[*h*]chromen-2-one (3h). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 4.01 (3H, s), 4.05 (3H, s), 6.42 (1H, d, *J* = 10.0 Hz), 7.13 (1H, s), 7.30 (1H, d, *J* = 8.0 Hz), 7.51 (1H, d, *J* = 8.0 Hz) 7.74 (1H, s), 7.78(1H, d, *J* = 9.0 Hz); $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$): δ = 56.0, 56.3, 100.9, 106.5, 113.3, 114.5, 118.2, 122.0, 122.8, 131.3, 144.6, 150.3, 150.4, 151.5, 161.4; HRMS (m/e): calcd for $C_{15}H_{12}O_4$ 256.0736, found 256.0735.

4-Methyl-2*H*-benzo[*h*]chromen-2-one (3i). ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.54 (3H, d, *J* = 1.6 Hz), 6.39(1H, d, *J* = 1.2 Hz), 7.62-7.66(4H, m), 7.87(1H, dd, *J* = 2.4

Hz, J = 4.4 Hz), 8.58 (1H, dd, J = 2.0 Hz, J = 6.4 Hz); $^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$): δ 19.13, 114.27, 115.10, 120.22, 122.57, 123.09, 124.04, 127.04, 127.56, 128.51, 134.70, 150.52, 153.29, 160.76; HRMS (m/e): calcd for $C_{14}H_{10}O_2$ 210.0680, found 210.0679.

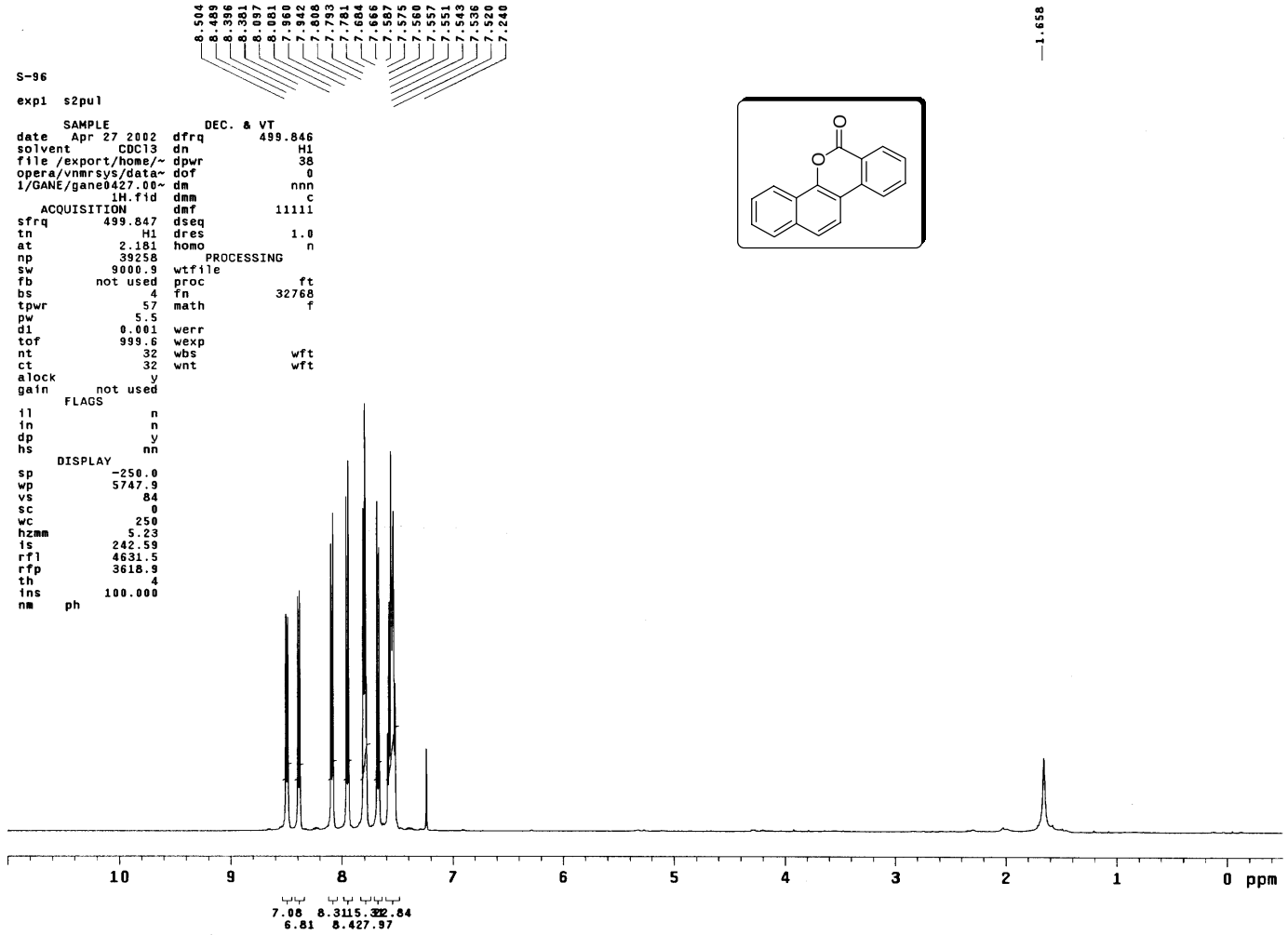
8,9-Dimethoxy-4-methyl-2*H*-benzo[*h*]chromen-2-one (3j). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.47 (3H, d, J = 1.2 Hz,), 4.00 (3H, s), 4.04 (3H, s), 6.27 (1H, d, J = 1.6 Hz,), 7.10 (1H, s), 7.41 (1H, d, J = 8.4 Hz,), 7.50 (1H, d, J = 8.4 Hz,), 7.72 (1H, s); $^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$): δ = 19.09, 55.93, 56.28, 101.26, 106.31, 113.15, 114.16, 118.28, 118.71, 122.50, 131.17, 149.68, 150.29, 151.50, 153.67, 161.22; HRMS (m/e): calcd for $C_{16}H_{14}O_4$ 270.0892, found 270.0893.

9-Methyl-6-oxaheptacyclo[14.6.6.$0^{2,15}$.$0^{4,13}$.$0^{5,10}$.$0^{17,22}$.$0^{23,28}$]octacosa-2(15),3,5(10), 8,11,13,17(22),18,20,23(28),24,26-dodecaen-7-one (3l). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.44 (3H, d, J = 0.8 Hz), 5.55 (1H, s), 5.60 (1H, s), 6.28 (1H, d, J = 0.8 Hz), 7.00-7.05 (4H, m), 7.41-7.47 (5H, m), 7.53 (1H, d, J = 8.8 Hz), 7.76 (1H, s), 8.50 (1H, s); $^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$): δ = 18.8, 53.6, 53.7, 113.6, 114.9, 116.3, 119.9, 120.7, 121.4, 123.4, 123.7, 123.9, 125.6, 125.7, 133.1, 143.4, 143.9, 144.1, 144.9, 150.0, 153.3, 160.7; HRMS (m/e): calcd for $C_{28}H_{18}O_2$ 386.1307, found 386.1302.

5,6-Dihydrobenzo[*c*]phenanthridin-6-one (4). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 3.89-3.87 (1H, m), 4.86(1H, d, J = 6 Hz), 5.50 (1H, s), 5.77-5.50 (1H, m), 7.16 (1H, d, J = 7.5 Hz), 7.26-7.24 (2H, m), 7.34-7.30 (2H, m), 7.39 (1H, td, J = 7 Hz, J = 1 Hz), 7.52 (1H, td, J = 7 Hz, J = 1 Hz), 8.09 (1H, d, J = 7.5 Hz); $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$): δ = 38.8, 52.2, 127.1, 127.2, 127.6, 127.7, 128.0, 128.3, 128.6, 129.3, 131.7, 132.7, 133.0, 139.6; HRMS (m/e): calcd for $C_{17}H_{13}NO$ 247.0997, found 247.0997.

(3*R,4*S**,4a*S**,10b*R**)-3,4-Di((methyloxy)methyl)-4,4a,6,10b-tetrahydro-3*H*-b**

enzo[*c*]chromen-6-one (6). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.07-3.10 (1H, m), 3.18-3.19 (1H, m), 3.29 (3H, s), 3.34-3.35 (1H, m), 3.37 (1H, s), 3.52 (1H, d, *J* = 6.0 Hz), 3.53-3.57 (2H, m), 3.63-3.68 (2H, m), 5.80 (1H, dd, *J* = 2.8 Hz, *J* = 9.2 Hz), 6.59 (1H, dd, *J* = 2.8 Hz, *J* = 9.2 Hz), 7.47 (1H, t, *J* = 7.6 Hz), 7.58 (1H, d, *J* = 8.0 Hz), 7.73 (1H, t, *J* = 7.6 Hz), 8.30 (1H, d, *J* = 8.0 Hz); HRMS (m/e): calcd for $C_{17}H_{20}O_4$ 288.1362, found 288.1359.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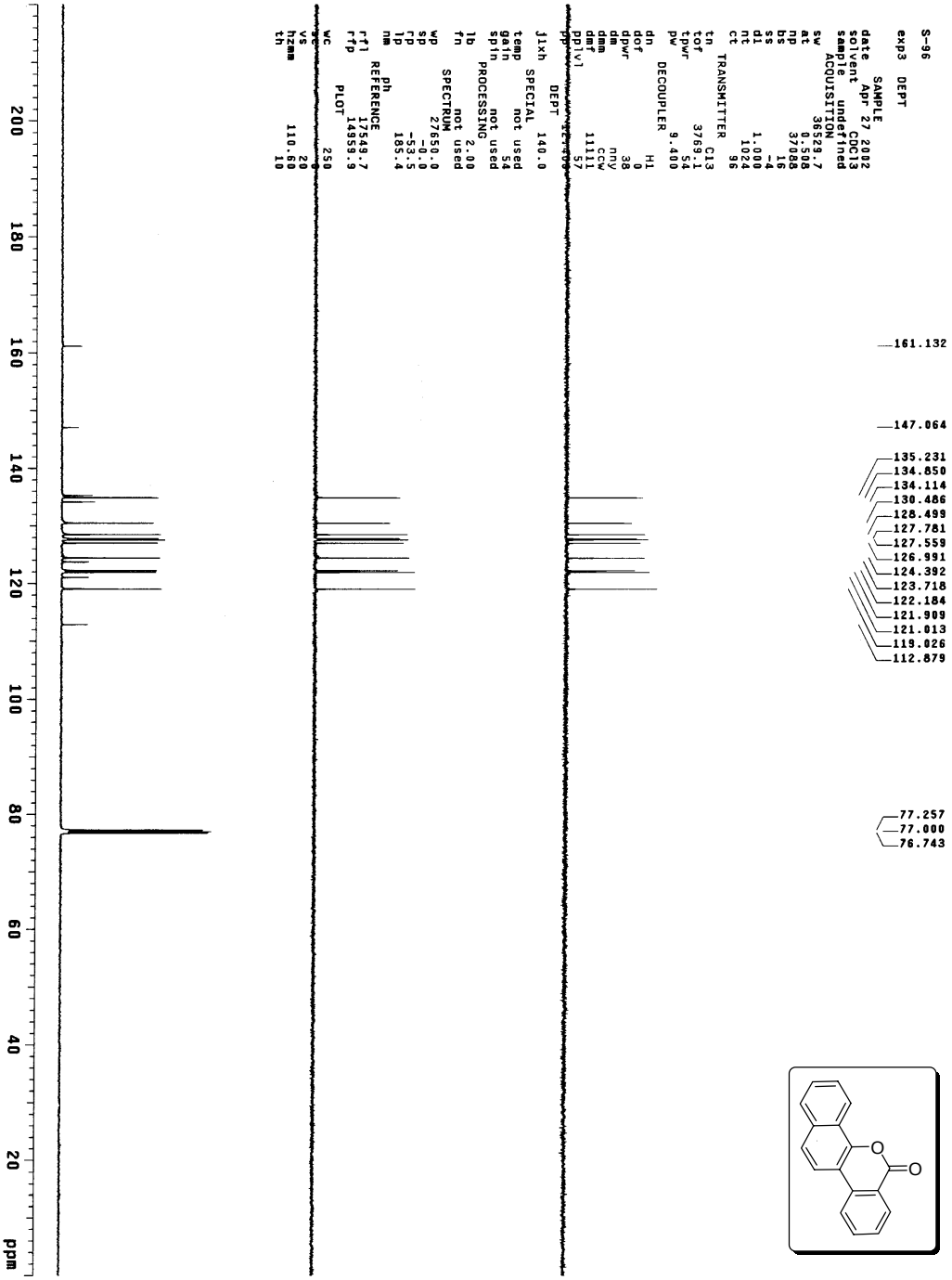
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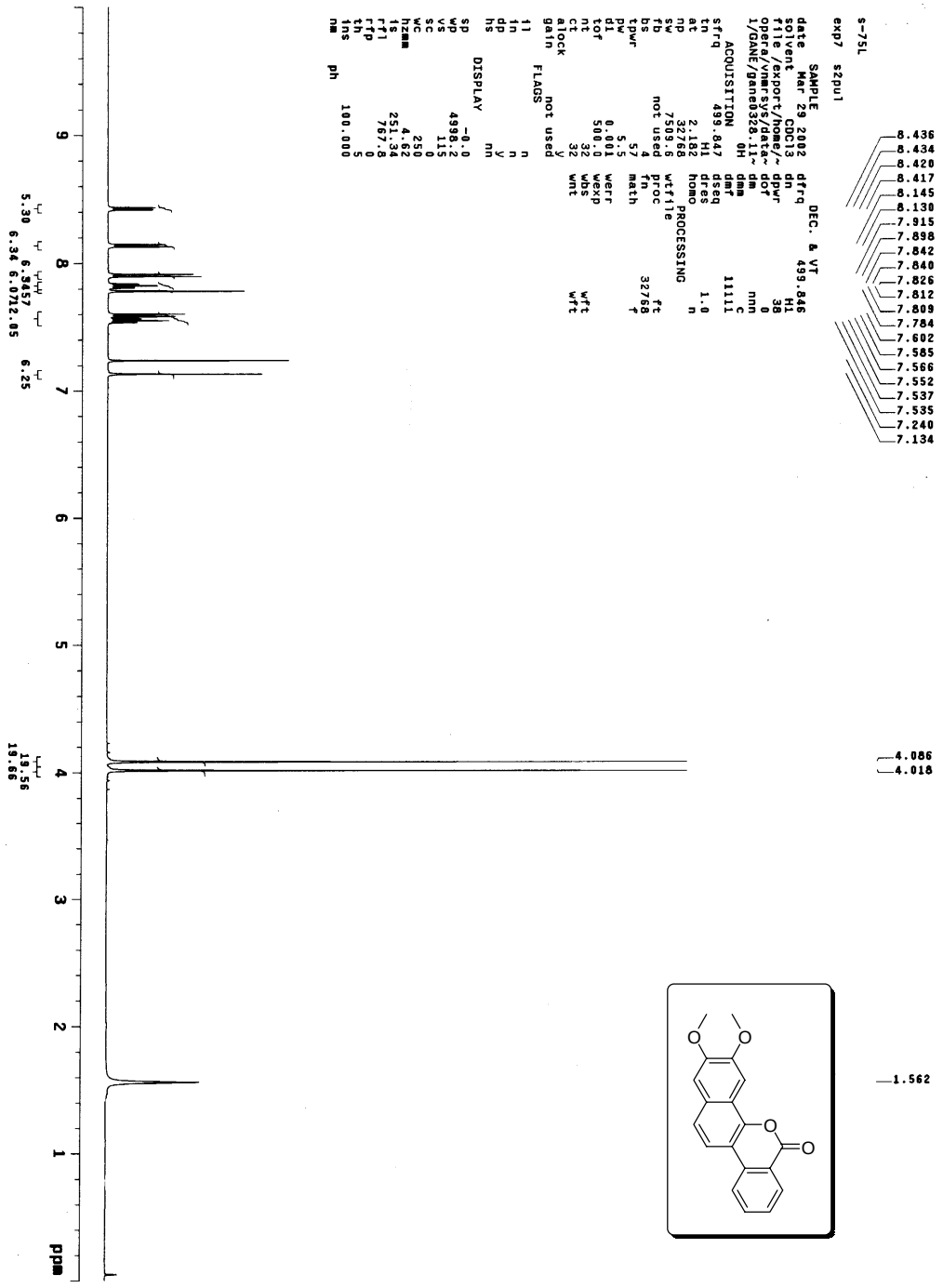
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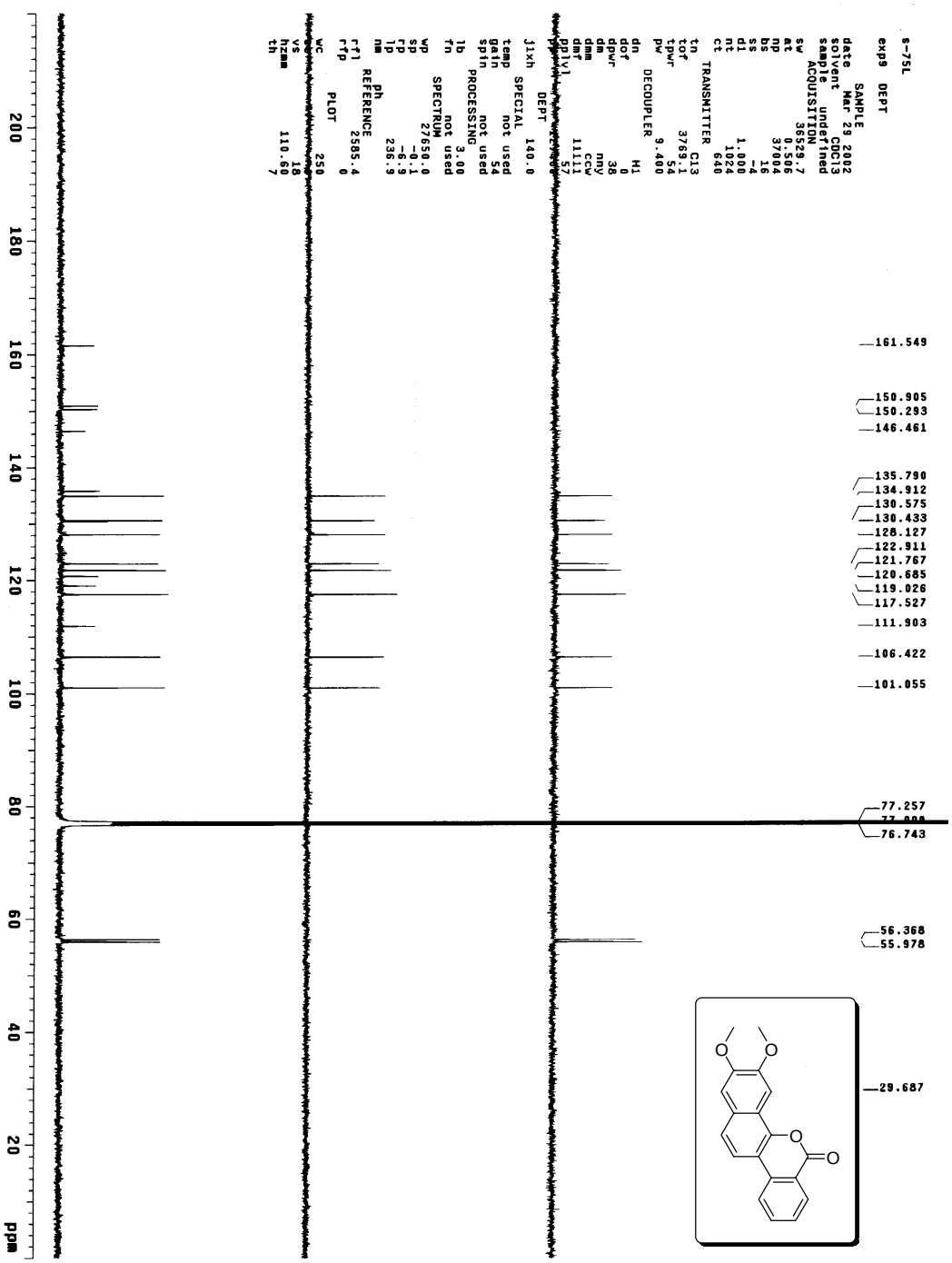
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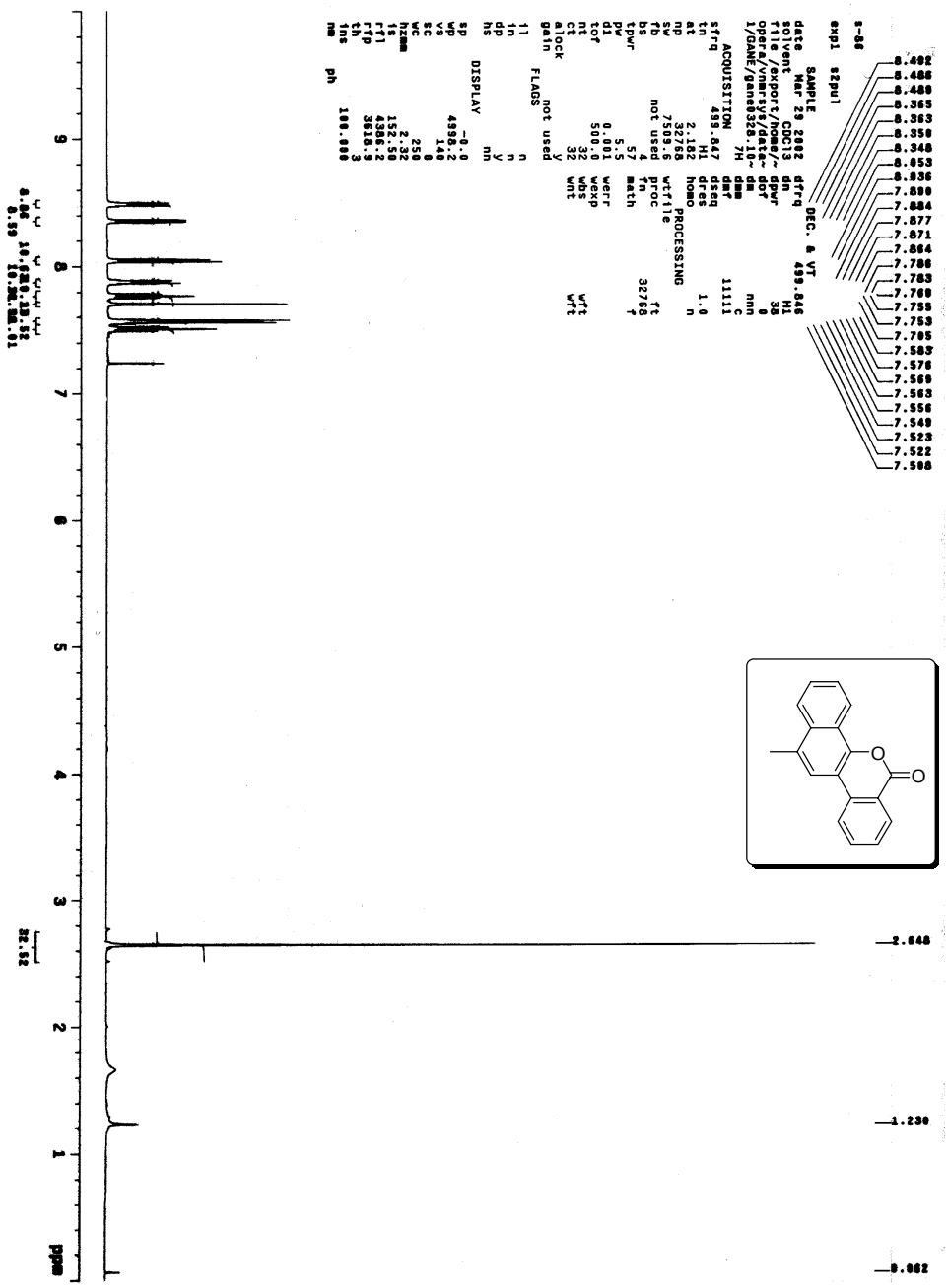
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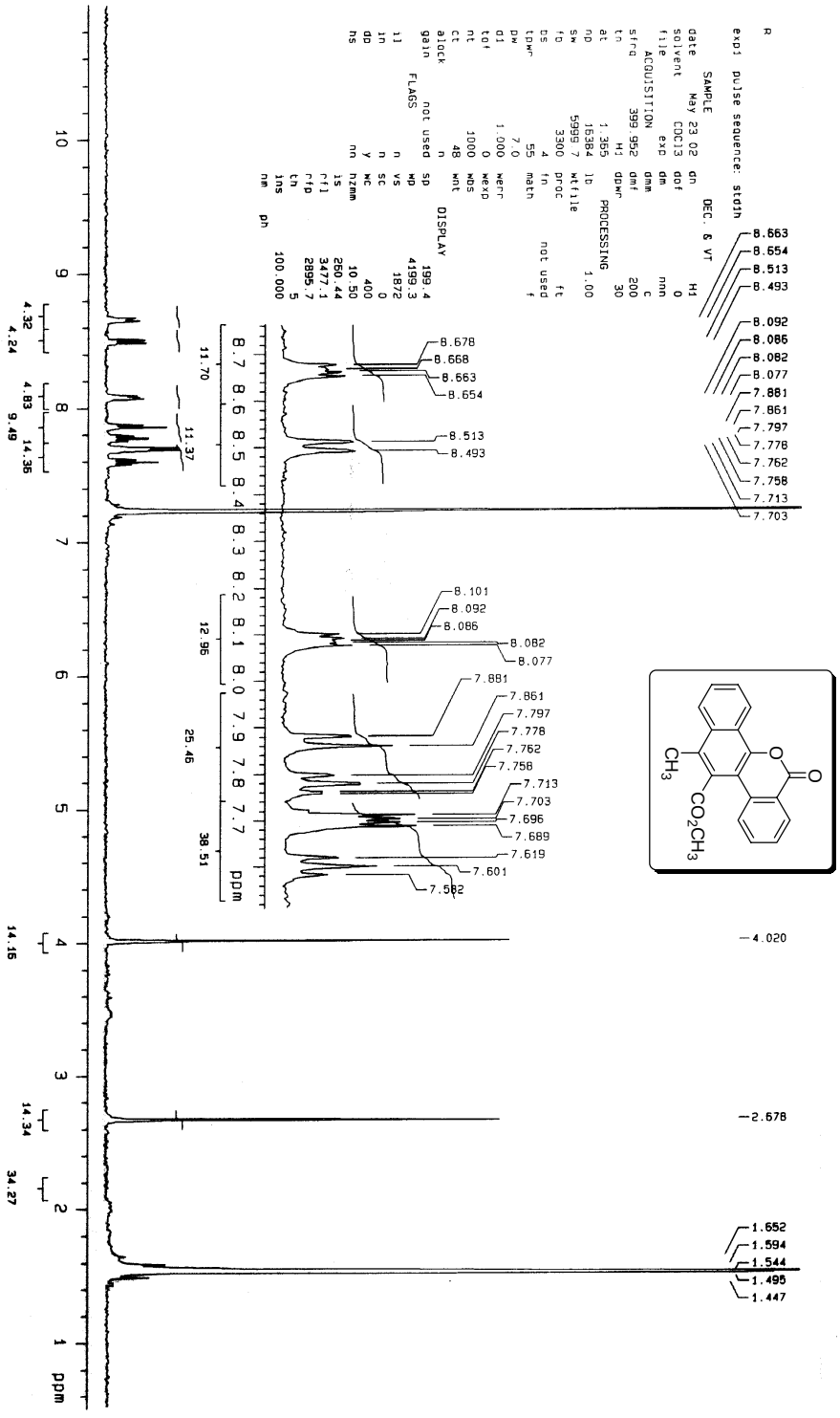
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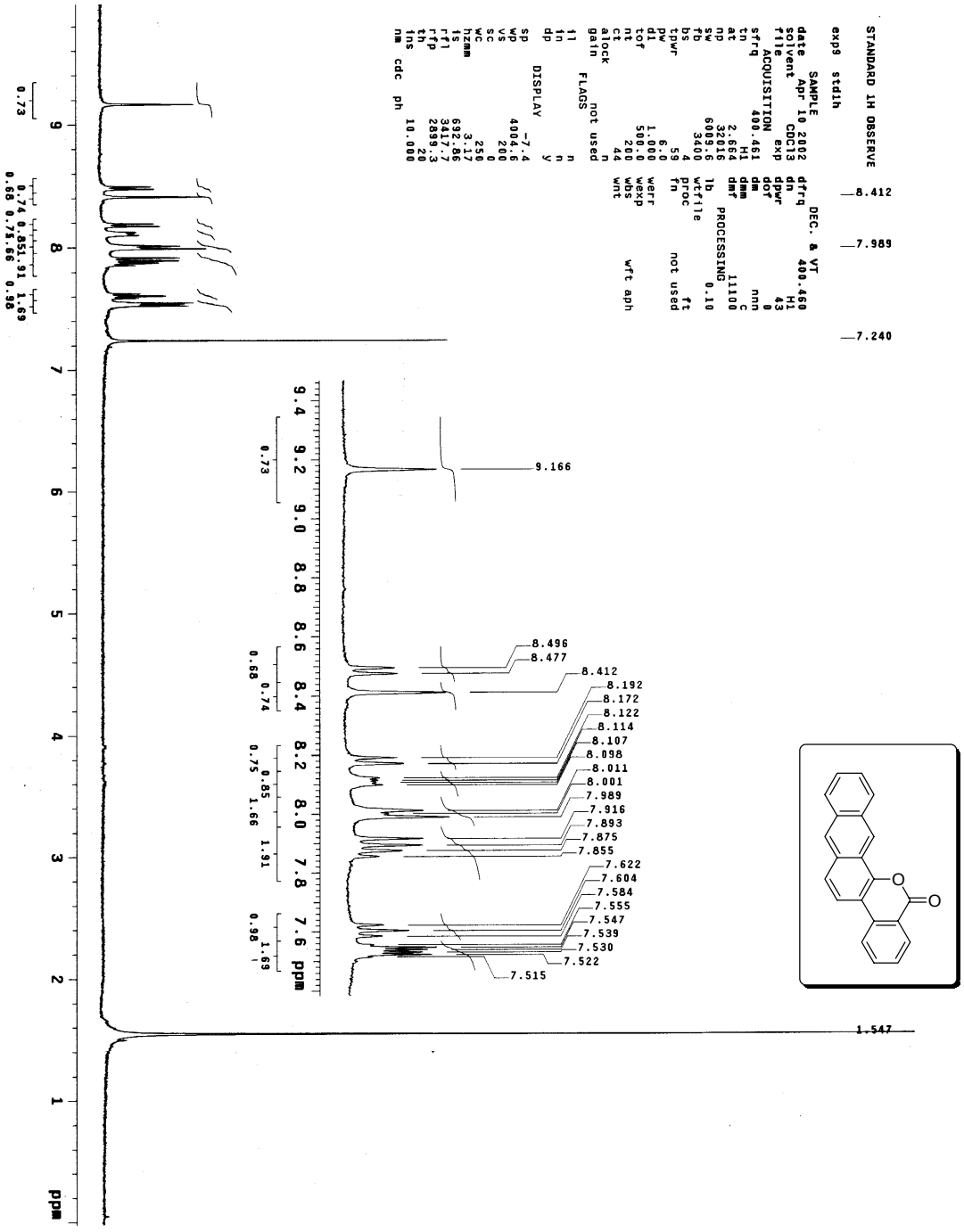
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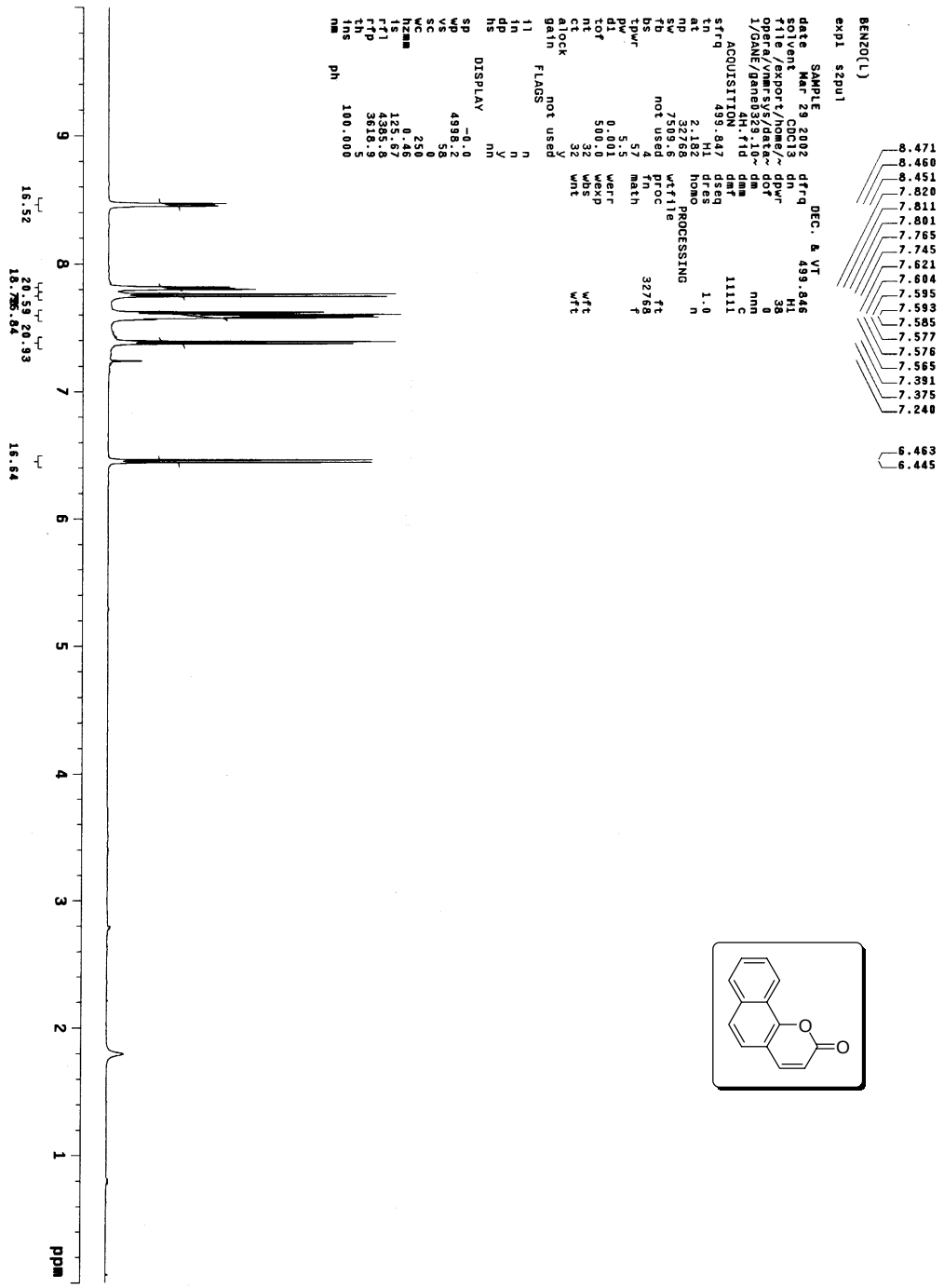
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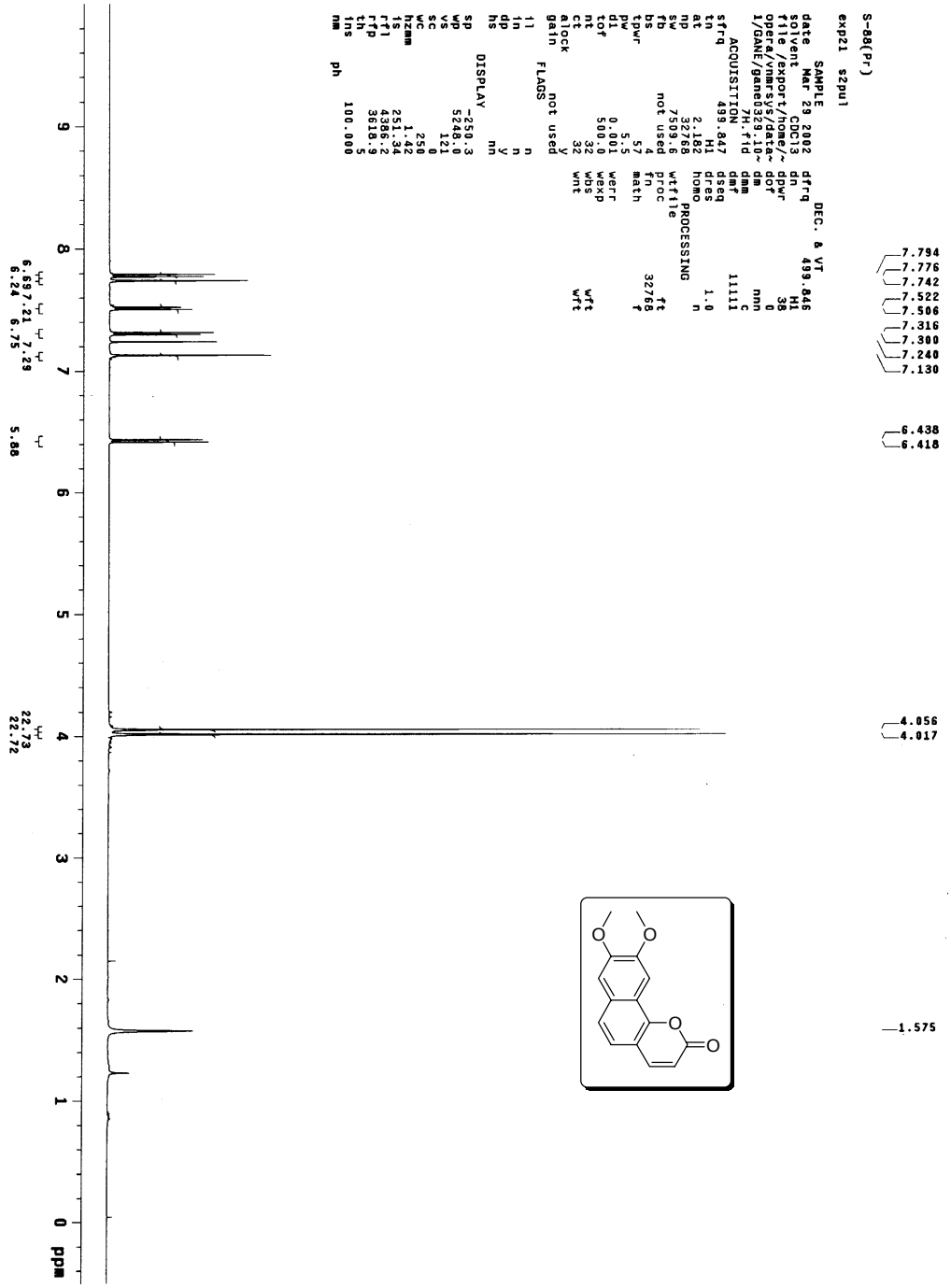
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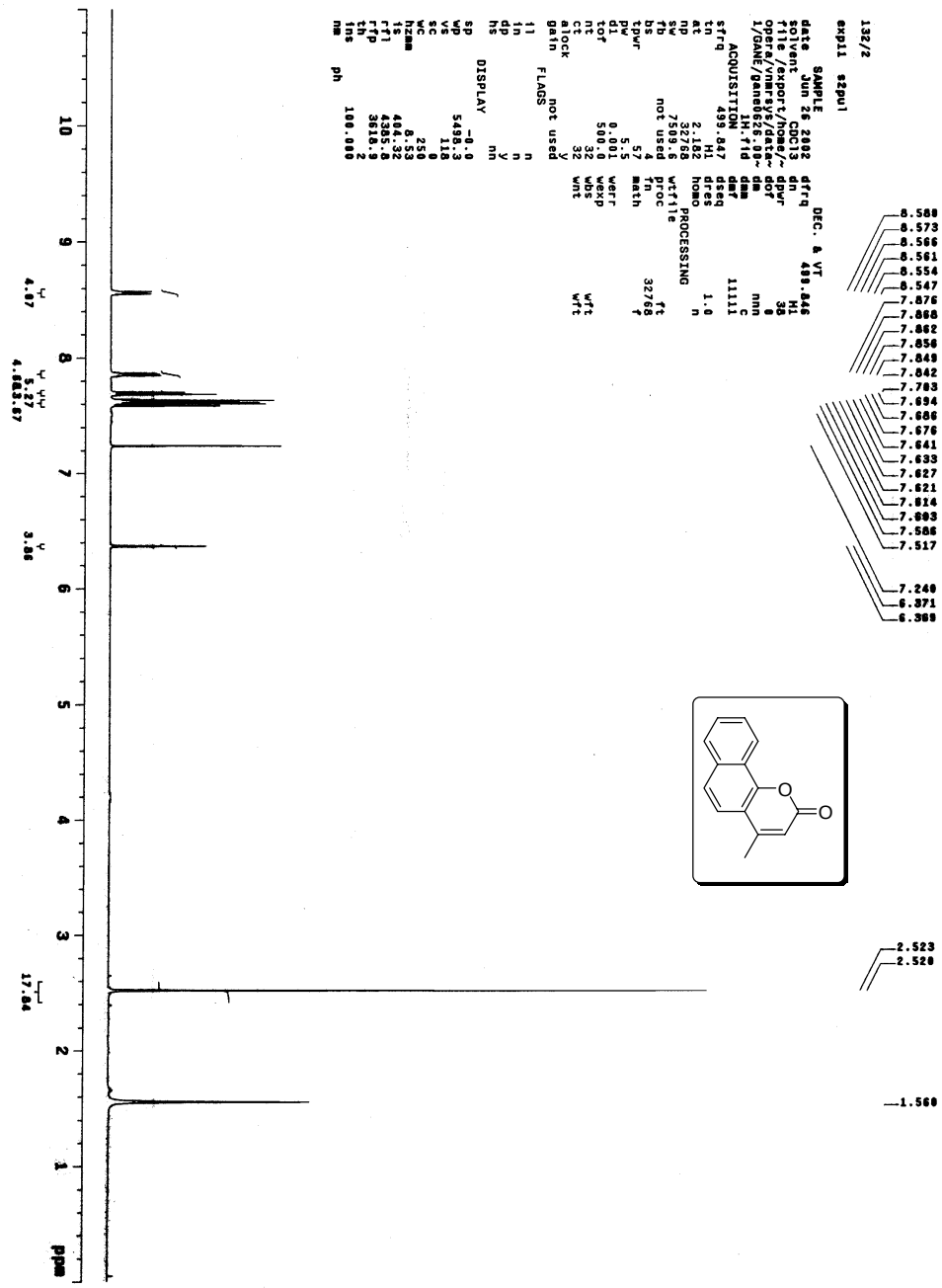
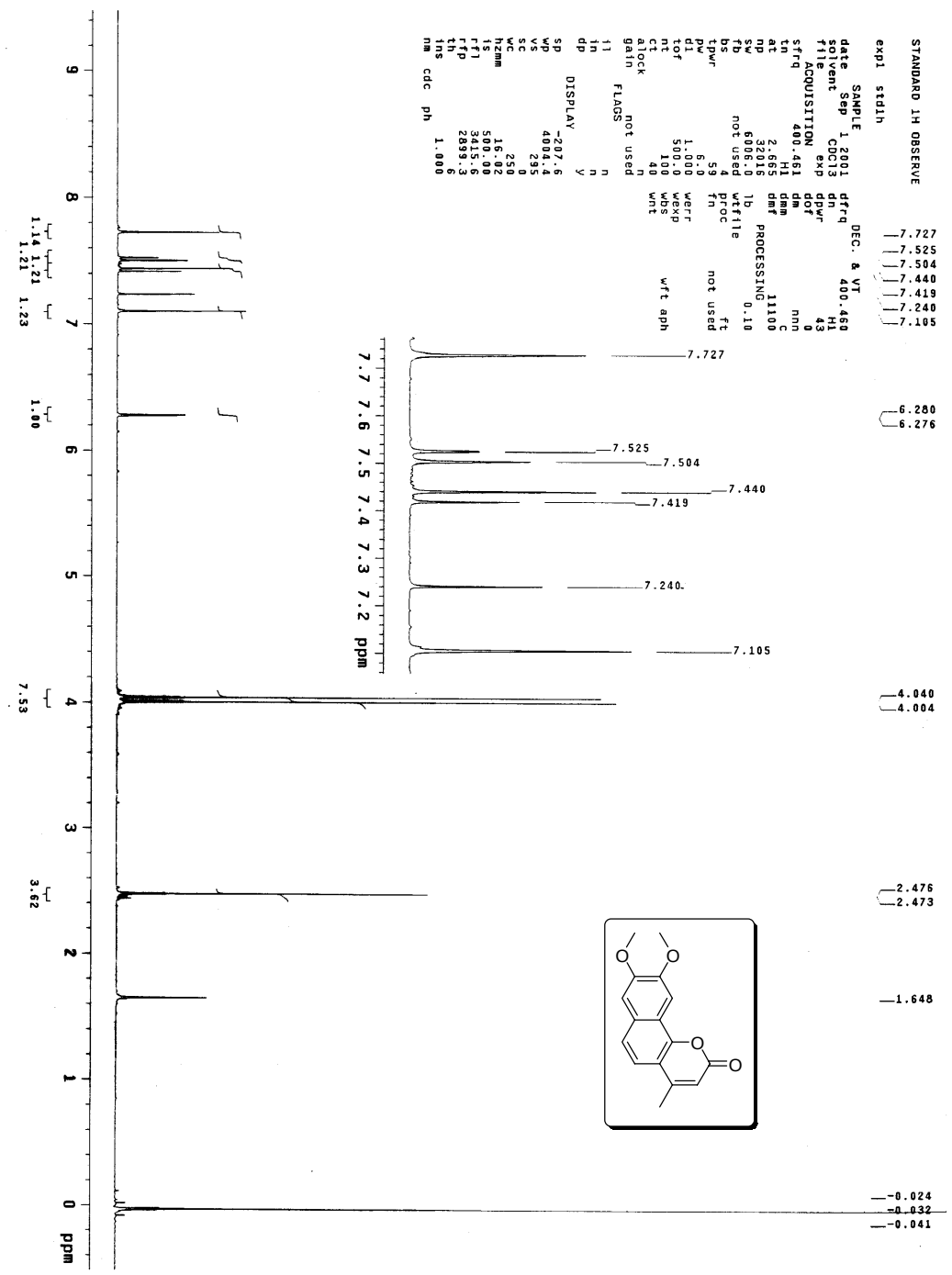
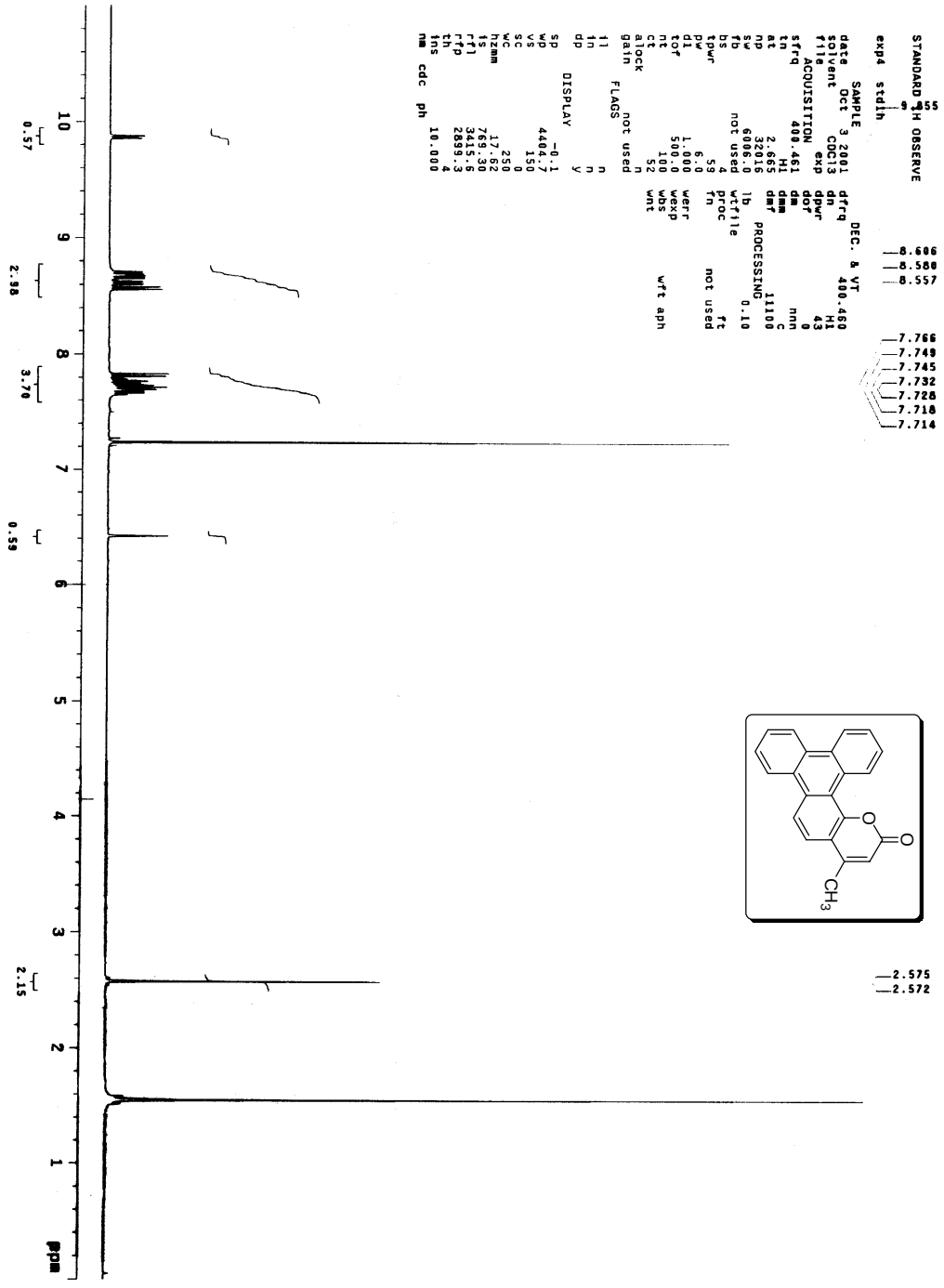
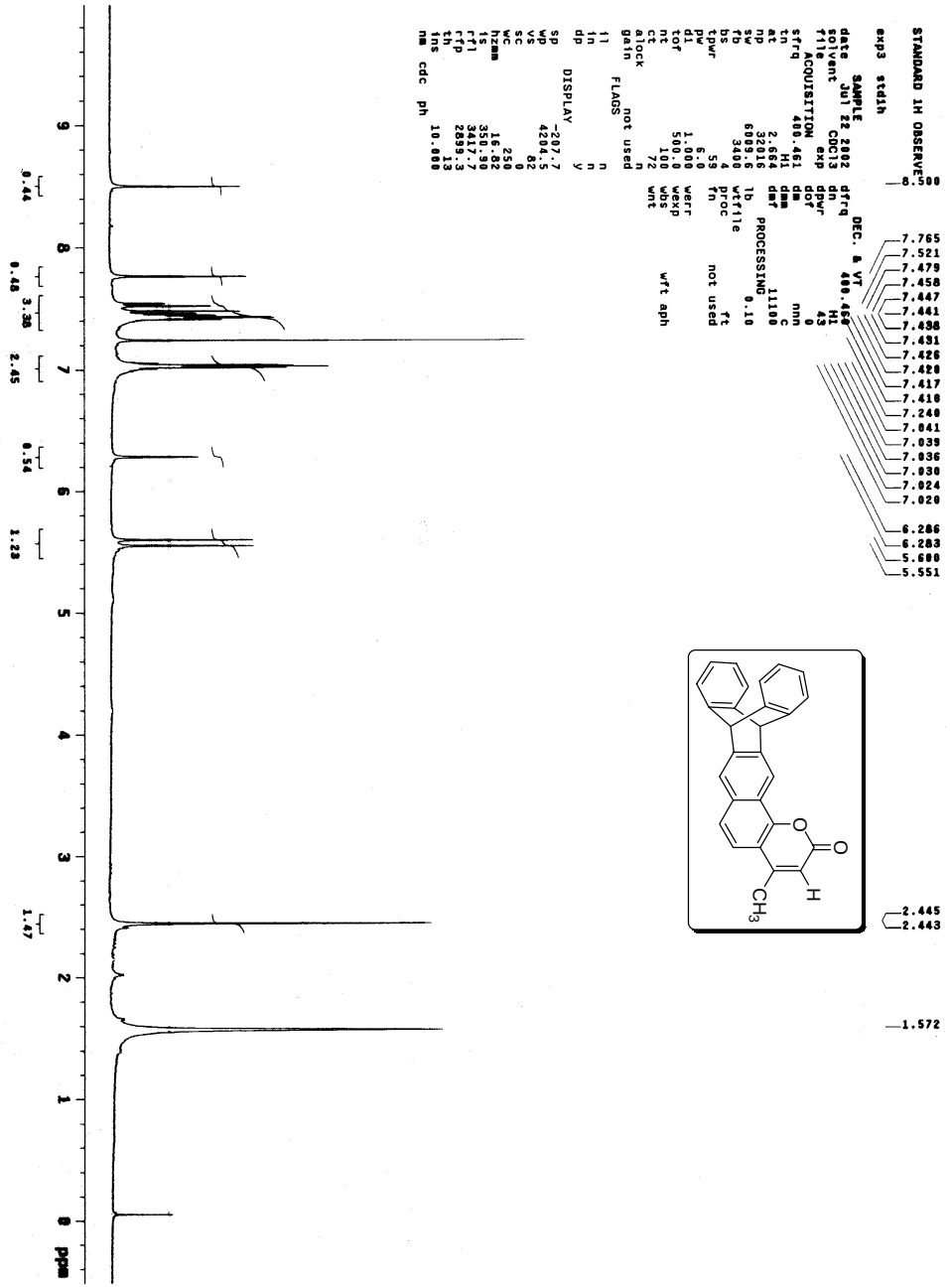
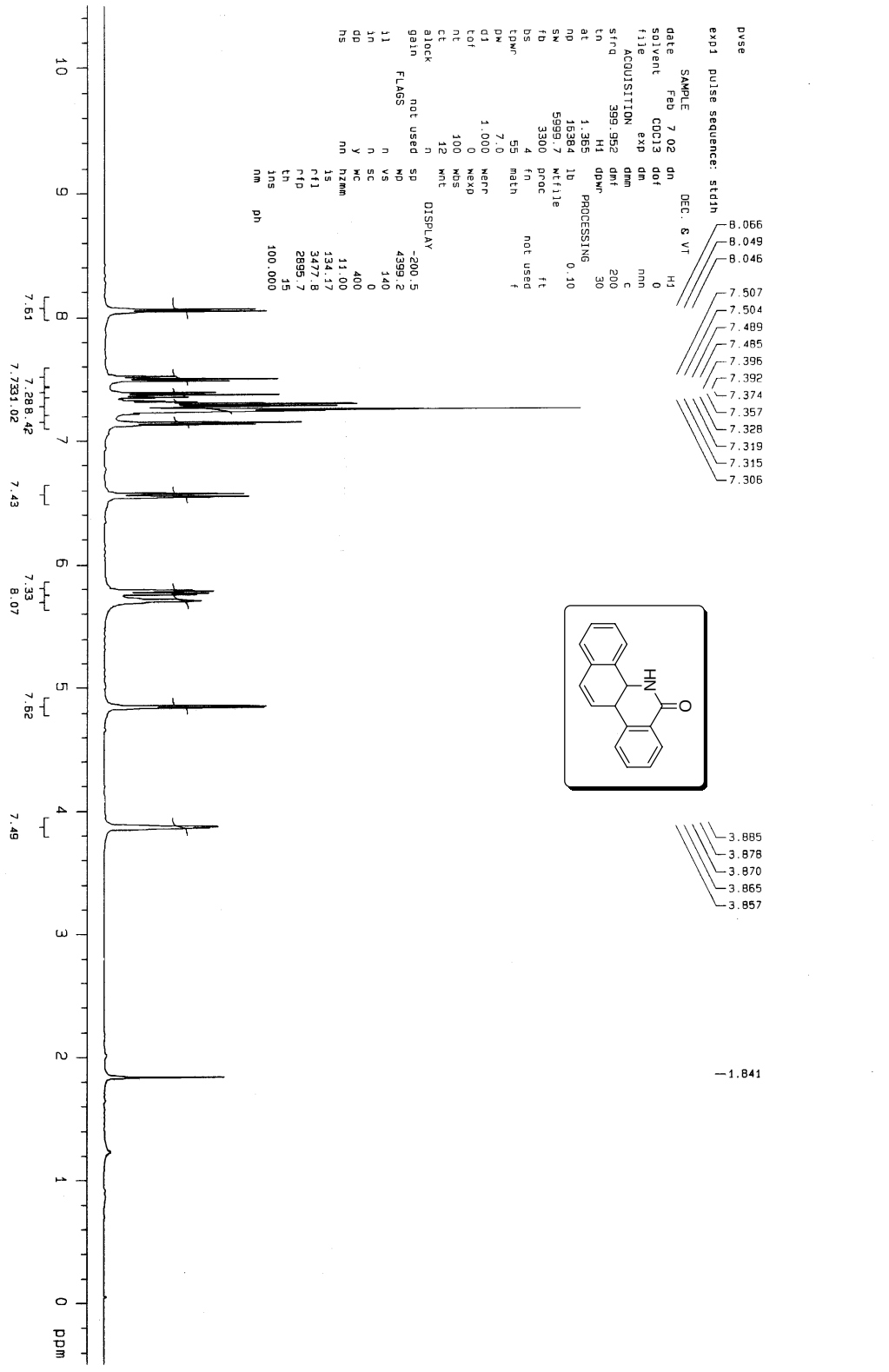
operator/vnmrsys/data~
1/SAME/gene03/7H.fid
ACQUISITION
sfrq 499.847
tn H1
at 2.182
np 32768
sw 7509.6
fb not used
bs 4
tpwr 57
pw 5.5
d1 0.001
tof 500.0
nt 32
ct 32
alock y
gain not used
FLAGS
DISPLAY
sp -250.3
wp 5248.1
vs 12
sc 0
wc 250
hzmm 1.42
is 251.34
rfl 4386.2
rfp 3618.9
th 5
ins 100.000
nm ph
DEC. & VT
dfrq 499.846
dn H1
dpwr 38
dof 0
dmm nnc
dmf 11111
seq
res
homo
1.0 n
PROCESSING
wtfile
proc ft
fn 32768
math f
wft
wft
wft
7.794
7.776
7.742
7.522
7.506
7.316
7.300
7.240
7.130
6.438
6.418
4.056
4.017
1.575
7.29
7.21
6.75
6.69
6.24
5.88
22.73
22.72
ppm

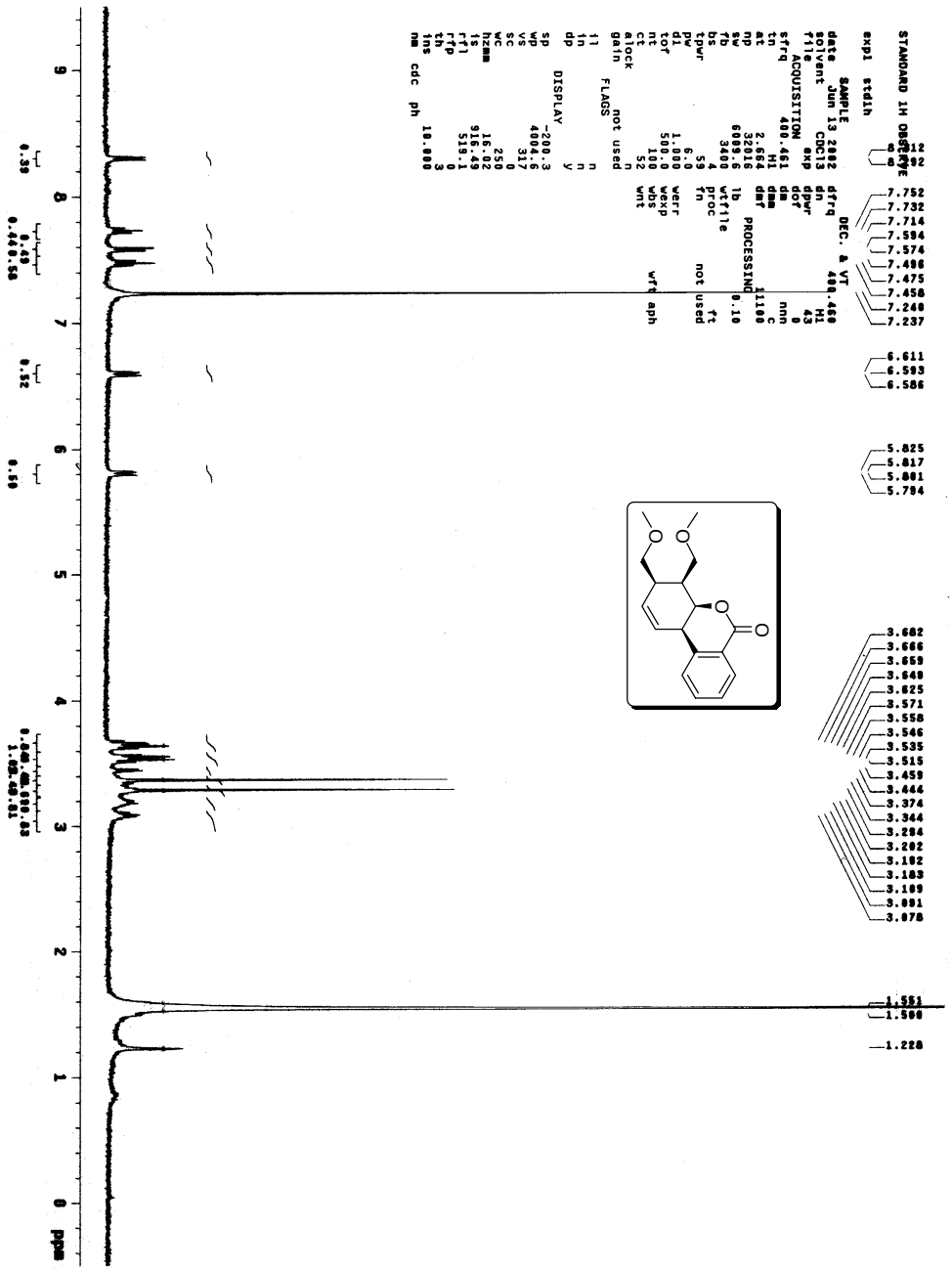

8.588
8.573
8.566
8.561
8.554
8.547
7.876
7.868
7.862
7.856
7.849
7.842
7.703
7.694
7.686
7.676
7.641
7.633
7.627
7.621
7.614
7.603
7.586
7.517
7.240
6.371
6.368
2.523
2.520
1.560
4.07
5.27
4.68
3.87
3.86
17.04
ppm


STANDARD 1H OBSERVE
SAMPLE
date Sep 1 2001
solvent CDCl3
file exp
ACQUISITION
sfrq 400.461 MHz
tn H1
at 2.665
np 32016
sw 6006.0
fb not used
bs 4
tpwr 59
pw 6.0
d1 1.000
tof 500.0
nt 100
ct 40
alock not used
gain
FLAGS
il n
in n
dp y
DISPLAY
sp -207.6
wp 4004.4
vs 295
sc 0
wc 250
hzmm 16.02
is 500.00
rfl 3415.9
rfp 2899.3
th 6
ins 1.000
nm cdc ph
dfrq
dn
dpwr
dof
dm
dmm
dmf
PROCESSING
lb
wtfile
proc
fn
not used
wft aph
DEC. & VT
400.460
H1
43
nnn
11106
0.10
ft
not used
7.727
7.525
7.504
7.440
7.419
7.240
7.105
6.280
6.276
4.040
4.004
2.476
2.473
1.648
-0.024
-0.032
-0.041
1.14
1.21
1.21
1.23
1.00
7.53
3.62
ppm


STANDARD 1H OBSERVE
exp4 std1h
SAMPLE
date Oct 3 2001
solvent CDCl3
file
ACQUISITION
sfrq 400.461
tn H1
at 2.665
np 32016
sw 6006.0
fb
bs
tpwr
pw 6.0
d1 1.000
tof 500.0
nt 100
ct 52
alock
gain
FLAGS
il n
in n
dp y
DISPLAY
sp -0.1
wp 4404.7
vs 150
sc 0
wc 250
hzmm 17.62
is 7631.30
rf1 3415.9
rfp 2839.3
th 4
ins
nm cdc ph
10.000
DEC. & VT
dfrq 400.460
dn H1
dpwr 43
dof 0
dm nnn
dmf 11110
PROCESSING
lb 0.10
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt wft aph
9.455
8.606
8.580
8.557
7.766
7.749
7.745
7.732
7.728
7.718
7.714
2.575
2.572
0.57
2.98
3.70
0.59
2.15
O
O
CH3
ppm


STANDARD 1H OBSERVE
8.500
7.765
7.521
7.479
7.458
7.447
7.441
7.438
7.431
7.426
7.420
7.417
7.410
7.240
7.041
7.039
7.036
7.030
7.024
7.020
6.286
6.283
5.600
5.551
2.445
2.443
1.572
O
O
H
CH3
0.44
0.48
3.38
2.45
0.54
1.28
1.47
ppm


pvse
exp1 pulse sequence: std1h
SAMPLE
date Feb 7 02
solvent cdcl3
file exp
ACQUISITION
sfrq 399.952
tn H1
at 1.365
np 16384
sw 5999.7
fb 3300
bs 4
tdwr 55
pw 7.0
d1 1.000
tof 0
nt 100
ct 12
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DEC. & VT
dn H1
dof 0
dm nnn
dmm c
dmf 200
dpwr 30
PROCESSING
lb 0.10
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
DISPLAY
sp -200.5
wp 4399.2
vs 140
sc 0
wc 400
hzmm 11.00
is 134.17
rfl 3477.8
rfp 2895.7
th 15
ins 100.000
nm ph
8.066
8.049
8.046
7.507
7.504
7.489
7.485
7.396
7.392
7.374
7.357
7.328
7.319
7.315
7.306
3.885
3.878
3.870
3.865
3.857
1.841
7.51
7.28
8.42
7.733
1.02
7.43
7.33
8.07
7.52
7.49
10
9
8
7
6
5
4
3
2
1
0
ppm


7.752
7.732
7.714
7.594
7.574
7.498
7.475
7.458
7.240
7.237
6.611
6.593
6.586
5.825
5.817
5.801
5.794
3.682
3.666
3.659
3.648
3.625
3.571
3.558
3.546
3.535
3.515
3.459
3.444
3.374
3.344
3.294
3.202
3.192
3.183
3.109
3.091
3.078
1.551
1.500
1.228
ppm