Supplementary Information for Green Chemistry

A Practical Improvement of Odorless Corey-Kim and Swern Oxidations

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<Contents>

Procedure for the preparation of 6-morpholinohexan-1-ol (1a). A mixture of morpholine (5.04 g, 57.85 mmol) and 1-chloro-6-hexanol (7.90 g, 57.85 mmol) was heated at 120°C for 5 h. The solid cake was dissolved in warm methanol (10 mL), cooled and triturated with hexane (15 mL). The light pink crystals were filtered and washed with methanol and hexane (1:5) to yield HCl salt of **1a**[1)] (12.60 g, 97%): mp 164-165°C (methanol/ether); IR (KBr): 3415, 3395, 2945, 2925, 2710, 1430, 1310 cm^{-1}; ^1H NMR (CD_3OD, 400 MHz): δ 4.81 (br s, 1H, OH), 4.03 (br d, J = 12.1 Hz, 2H), 3.82 (br t, J = 12.1 Hz, 2H), 3.55 (t, J = 6.2 Hz, 2H), 3.49 (br d, J = 12.3 Hz, 2H), 3.17-3.10 (m, 4H), 1.83-1.75 (m, 2H), 1.58-1.52 (m, 2H), 1.46-1.40 (m, 4H); ^{13}C NMR (CD_3OD, 100 MHz): δ 65.0, 62.6, 58.4, 53.1, 33.3, 27.4, 26.4, 24.6; MS (EI): *m/z* 187 (M^+, 2.5), 157 (3.4), 100 (100), 88 (2.8); Anal. Calcd for $C_{10}H_{22}NO_2Cl$: C, 53.68; H, 9.91; N, 6.26. Found: C, 53.42; H, 9.96; N, 6.51.

Typical procedure for the preparation of 6-morpholinohexan-1-thiol (2a). To a solution of morpholino alcohol **1a** (10.5 g, 56.06 mmol), thiourea (4.69 g, 61.66 mmol) in water (20 mL) was added 48% HBr (27.22 g, 0.34 mol) slowly. The reaction mixture was refluxed with stirring at 120°C for 8 h under N_2 before being brought to rt. A solution of NaOH (20 g) in water (200 mL) was poured into the reaction mixture and kept refluxing under N_2 without stirring for another 2 h. Cooled and the pink colored solution was extracted with diethyl ether (5 × 75 mL). The ethereal layer was dried ($MgSO_4$) and the solvent was evaporated under reduced pressure to give crude material (9.72 g) as thiol **2a** (R_f = 0.4, 10% MeOH in hexane as eluents) along with little amount of the corresponding disulfide (R_f = 0.1, 10% MeOH in hexane as eluents). Silica gel column chromatography using hexane and ethyl acetate (1:1) or kuegelruhr distillation (130°C, 1.5 mmHg) afforded pure thiol **2a** as colorless oil (8.34 g, 73%). bp 130°C (1.5 mmHg); IR ($CHCl_3$): 3030, 2920, 2860, 2770, 2490, 1605, 1450, 1360 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ 3.70-3.68 (m, 4H), 2.50 (q, J = 7.1 Hz, 2H), 2.40 (br s, 4H), 2.29 (t, J = 7.7 Hz, 2H), 1.59 (quint, J = 7.1 Hz, 2H), 1.50-1.20 (m, 7H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 66.9, 59.0, 53.8, 33.9, 28.2, 26.9, 26.4, 24.5; MS (EI): *m/z* 203 (M^+, 2.5), 170 (26.1), 100 (100), 87 (15.8); HRMS calcd for $C_{10}H_{21}NOS$: 203.1340, found 203.1344.

5-Morpholinopentan-1-thiol (entry 3 in Table 1): Colorless oil (130°C, 1.5 mmHg); IR ($CHCl_3$): 3015, 2940, 2860, 2815, 1460, 1450, 1225, 1115 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ 3.71 (dd, J = 4.9, 4.6 Hz, 4H), 2.53 (q, J = 7.3 Hz, 2H), 2.43 (br dd, J = 4.2, 4.0 Hz, 4H), 2.33 (dd, J = 7.3, 5.8 Hz, 2H), 1.64 (q, J = 7.3 Hz, 2H), 1.54-1.37 (m, 4H), 1.35 (t, J = 7.3 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 66.9, 58.8, 53.7, 33.8, 26.1, 25.9, 24.5; MS (CI): *m/z* 190 (M^++H, 5.2), 156 (49), 100 (100), 87 (4.1); HRMS calcd for $C_9H_{20}NOS$ (M^++H): 190.1266, found 190.1259.

4-Morpholinobutan-1-thiol (2b) was prepared similarly from **1b**[1)] (8.75 g, 54.95 mmol), thiourea (4.60 g, 60.45 mmol) and 48% HBr (26.67 g, 0.33 mol) in 74% yield (7.12 g). Colorless oil (130°C, 1.5 mmHg); IR ($CHCl_3$): 3005, 2940, 2860, 2815, 1600, 1460, 1360 cm^{-1}; ^1H NMR ($CDCl_3$, 200 MHz): δ 3.72 (t, J = 4.6 Hz, 4H), 2.55 (q, J = 7.8 Hz, 2H), 2.43 (dd, J = 4.9, 4.6 Hz, 4H), 2.34 (t, J = 7.3 Hz, 2H), 1.72-1.50 (m, 4H), 1.36 (t, J = 7.8 Hz, 1H); ^{13}C NMR ($CDCl_3$, 50 MHz): δ 66.9, 58.4, 53.7, 31.9, 25.3, 24.6; MS (EI): *m/z* 175 (M^+, 25.9), 174 (75.1), 142 (13.7), 100 (100), 87 (62.9); HRMS calcd for $C_8H_{17}NOS$: 175.2943, found 175.2940.

3-Morpholinopropan-1-thiol (entry 1 in Table 1)[2)]: Colorless oil (110°C, 1.5 mmHg); IR ($CHCl_3$): 3005, 2965, 2945, 1460, 1260, 1115 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ 3.71 (dd, J = 4.9, 4.6 Hz, 4H), 2.59 (q, J = 7.1 Hz, 2H), 2.48-2.40 (m, 6H), 1.81 (q, J = 7.1 Hz, 2H), 1.43 (t, J = 7.9 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 67.0, 57.2, 53.7, 30.6, 22.6; MS (EI): *m/z* 161 (M^+, 73.9), 146 (3.6), 127 (12.5), 113 (9.4), 100 (100); HRMS calcd for $C_7H_{15}NOS$: 161.0874, found 161.0871.

References

1) Anderson, G. W.; Pollard, C. B. *J. Am. Chem. Soc.* **1939**, *61*, 3440.
2) Clinton, R. O.; Salvador, U. J.; Laskowski, S. C. *J. Am. Chem. Soc.* **1949**, *71*, 3366.

Table 1. The Odor Scale for Alkanethiols

Entry	Thiols	Carbon Length	Odor Scale [a] A	Odor Scale [a] B	Bp (°C)
1	CH_3CH_2SH	2	5	5	35
2	$CH_3(CH_2)_2SH$	3	5	5	67
3	$CH_3(CH_2)_3SH$	4	5	4	98
4	$CH_3(CH_2)_4SH$	5	4	3	126
5	$CH_3(CH_2)_5SH$	6	3	3	150
6	$CH_3(CH_2)_6SH$	7	4	3	173
7	$CH_3(CH_2)_7SH$	8	2	1	197
8	$CH_3(CH_2)_8SH$	9	2	1	220
9	$CH_3(CH_2)_9SH$	10	1	1	114/ 13mmHg
10	$CH_3(CH_2)_{10}SH$	11	1	0	103/ 3mmHg
11	$CH_3(CH_2)_{11}SH$	12	0	0	266
12	$CH_3(CH_2)_{13}SH$	14	0	1	298
13	$CH_3(CH_2)_{15}SH$	16	0	1	184/ 7mmHg

[a] Odor Scale: stench 5 ← 0 odorless

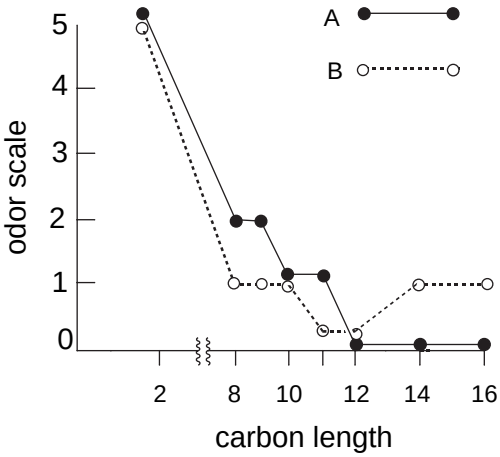
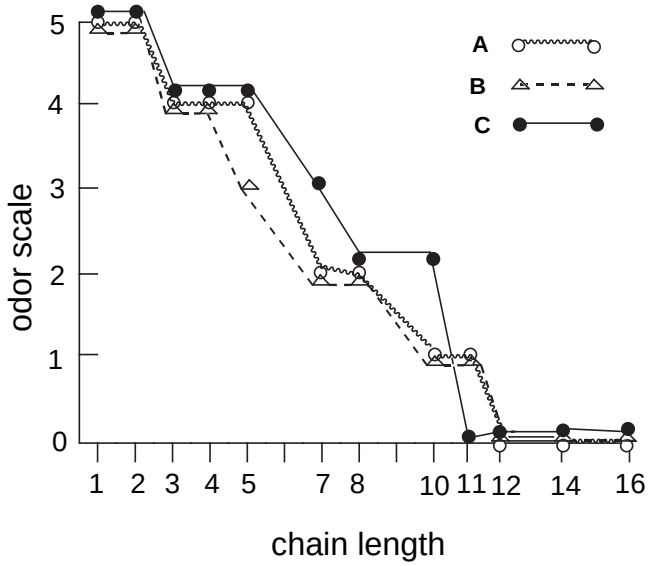
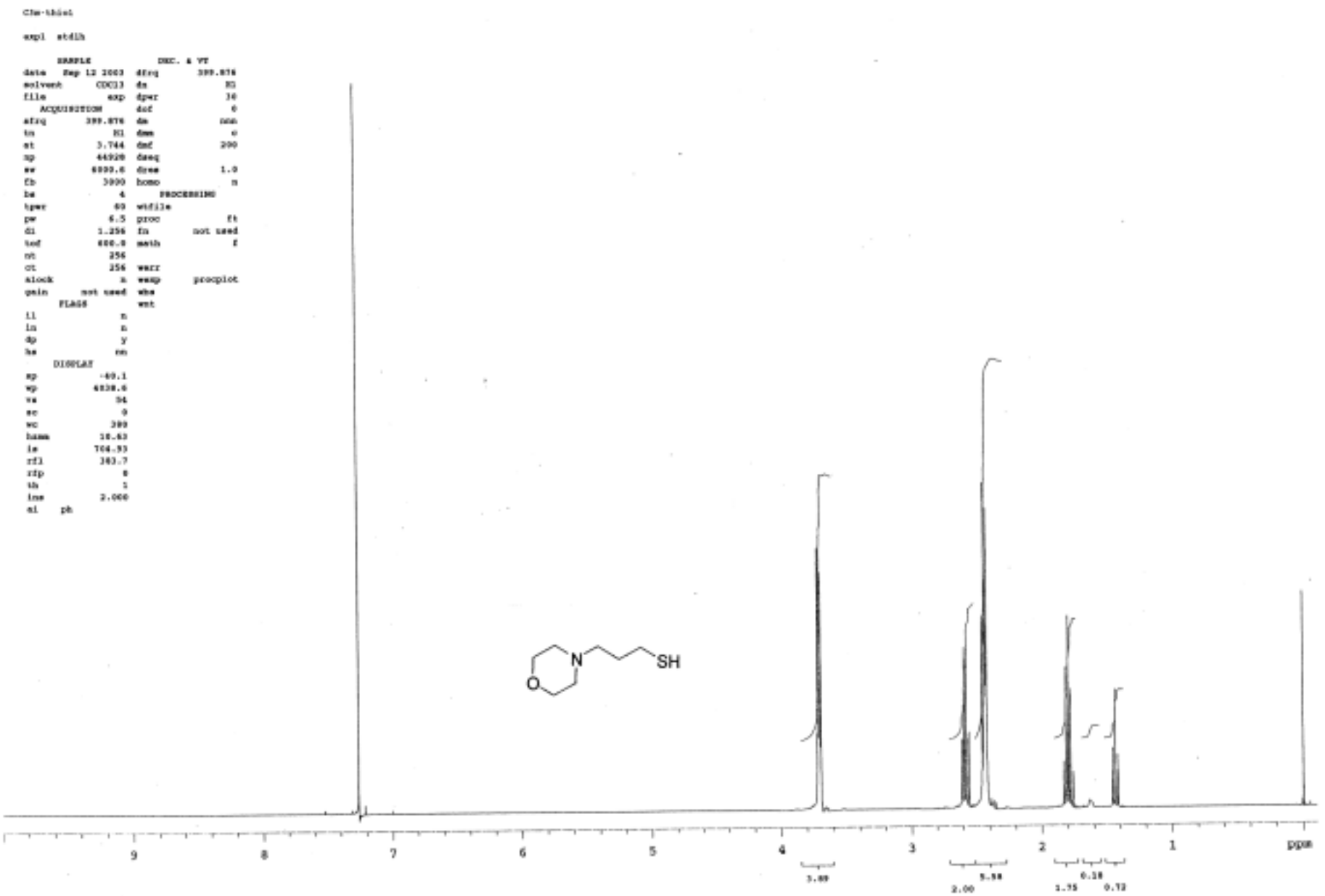


Table 2. The Odor Scale for Alkyl Methyl Sulfides

Entry	Sulfides	Carbon length	Odor Scale A	Odor Scale B	Odor Scale C
1	CH_3SCH_3	1	5	5	5
2	$CH_3CH_2SCH_3$	2	5	5	5
3	$CH_3(CH_2)_2SCH_3$	3	4	4	4
4	$CH_3(CH_2)_3SCH_3$	4	4	4	4
5	$CH_3(CH_2)_4SCH_3$	5	4	3	4
6	$CH_3(CH_2)_6SCH_3$	7	2	2	3
7	$CH_3(CH_2)_7SCH_3$	8	2	2	2
8	$CH_3(CH_2)_9SCH_3$	10	1	1	2
9	$CH_3(CH_2)_{10}SCH_3$	11	1	1	0
10	$CH_3(CH_2)_{11}SCH_3$	12	0	0	0
11	$CH_3(CH_2)_{13}SCH_3$	14	0	0	0
12	$CH_3(CH_2)_{15}SCH_3$	16	0	0	0
13	$C_6H_{13}SC_6H_{13}$	6 + 6	1	1	1

odor scale : stench 5 ← 0 odorless

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fb 3000
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ct 256
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in n
dp y
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dp               y
hs              ns
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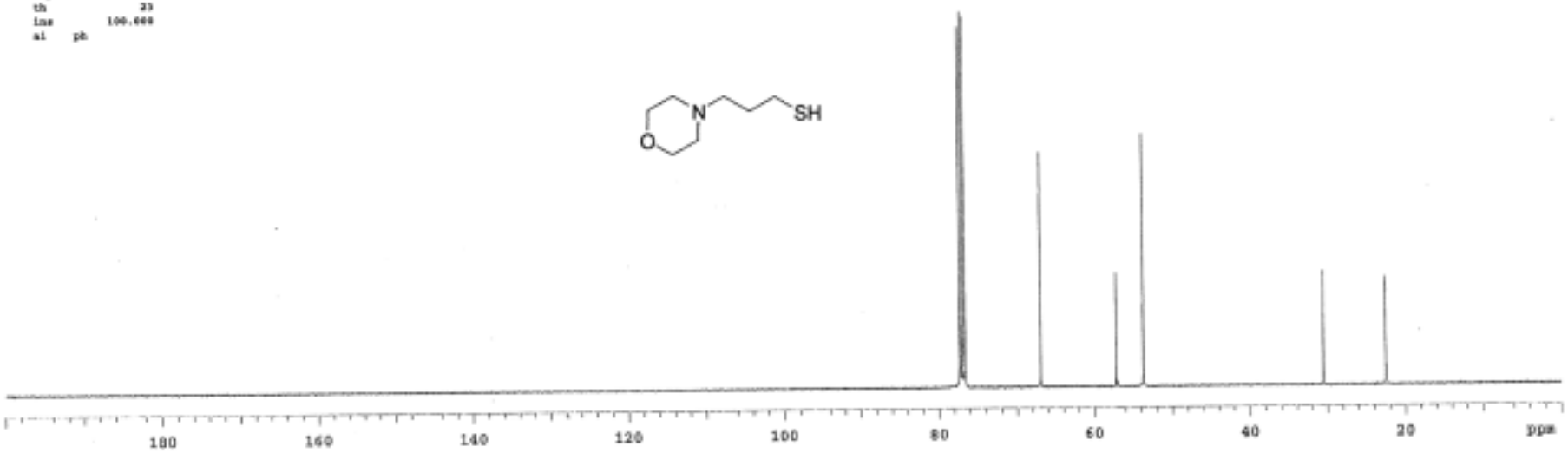

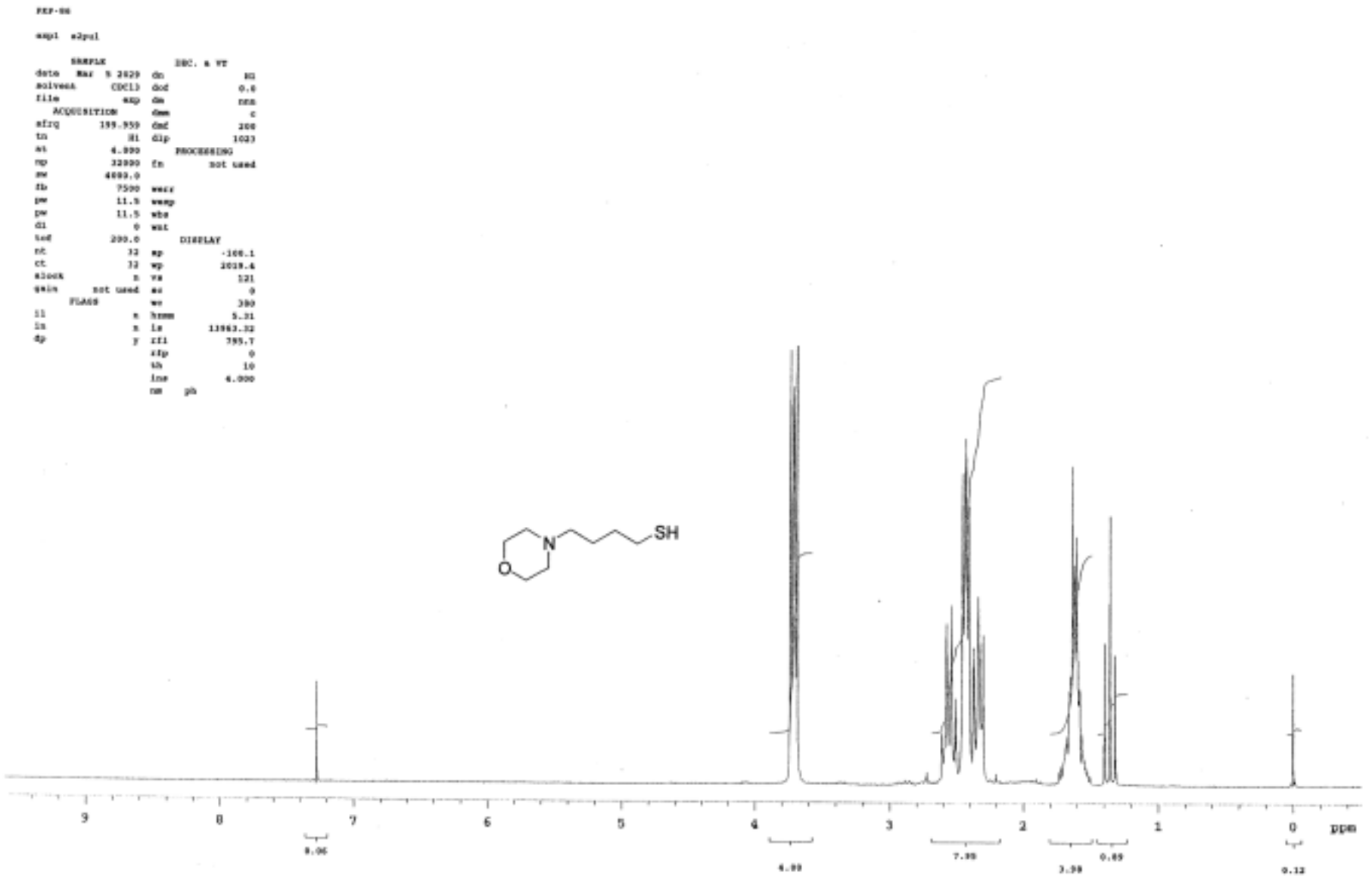
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13C SENSITIVITY
ASTM SAMPLE

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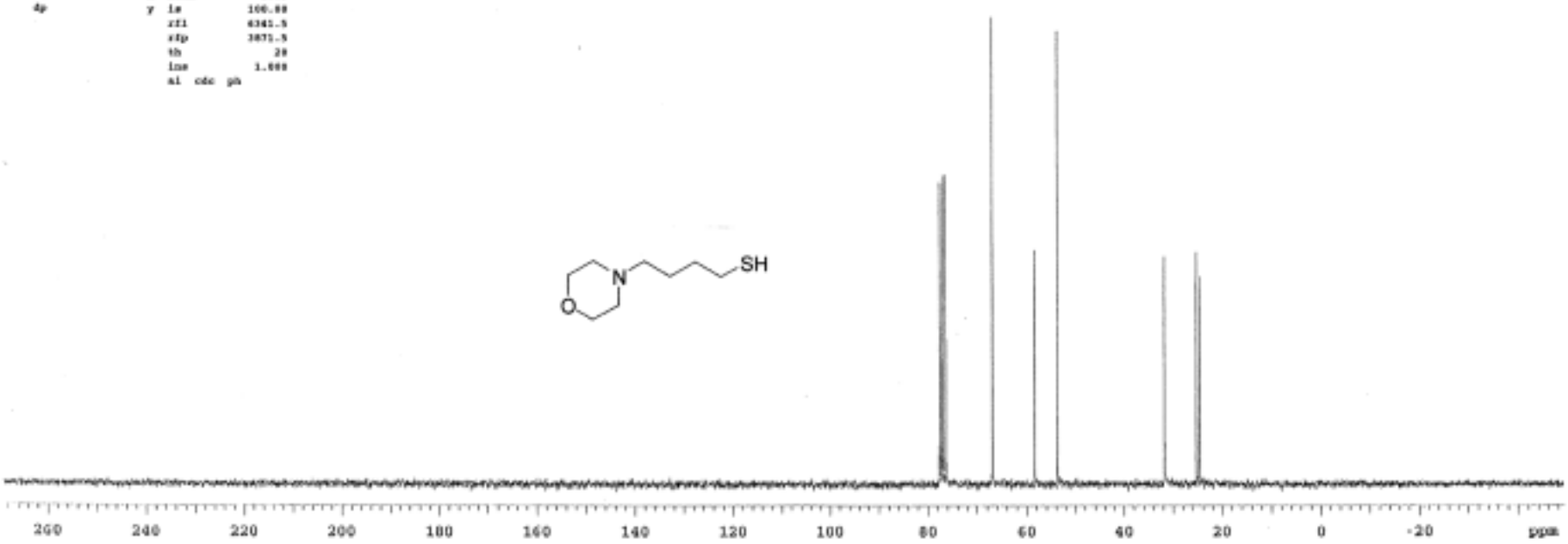

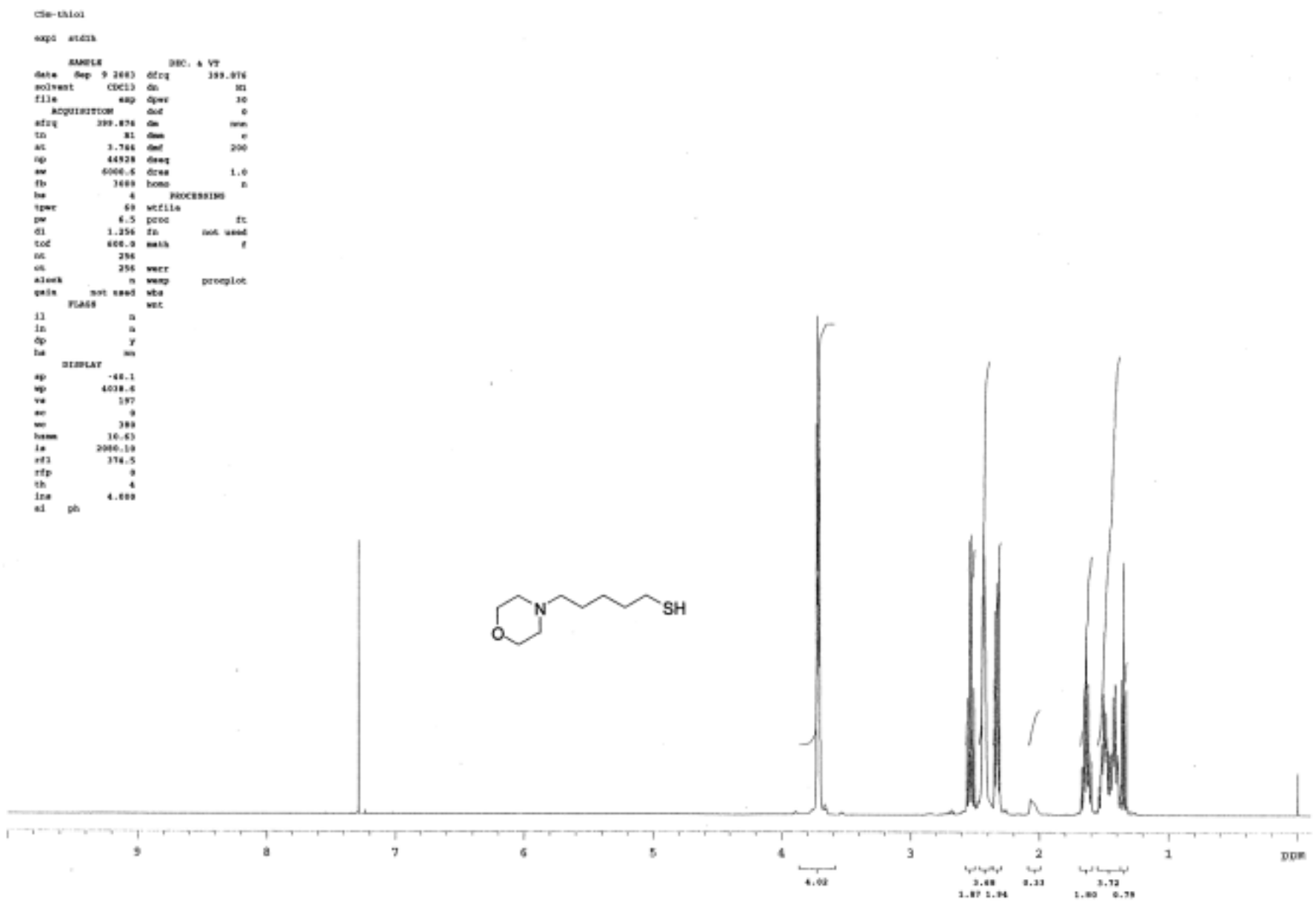
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C5n-thiol

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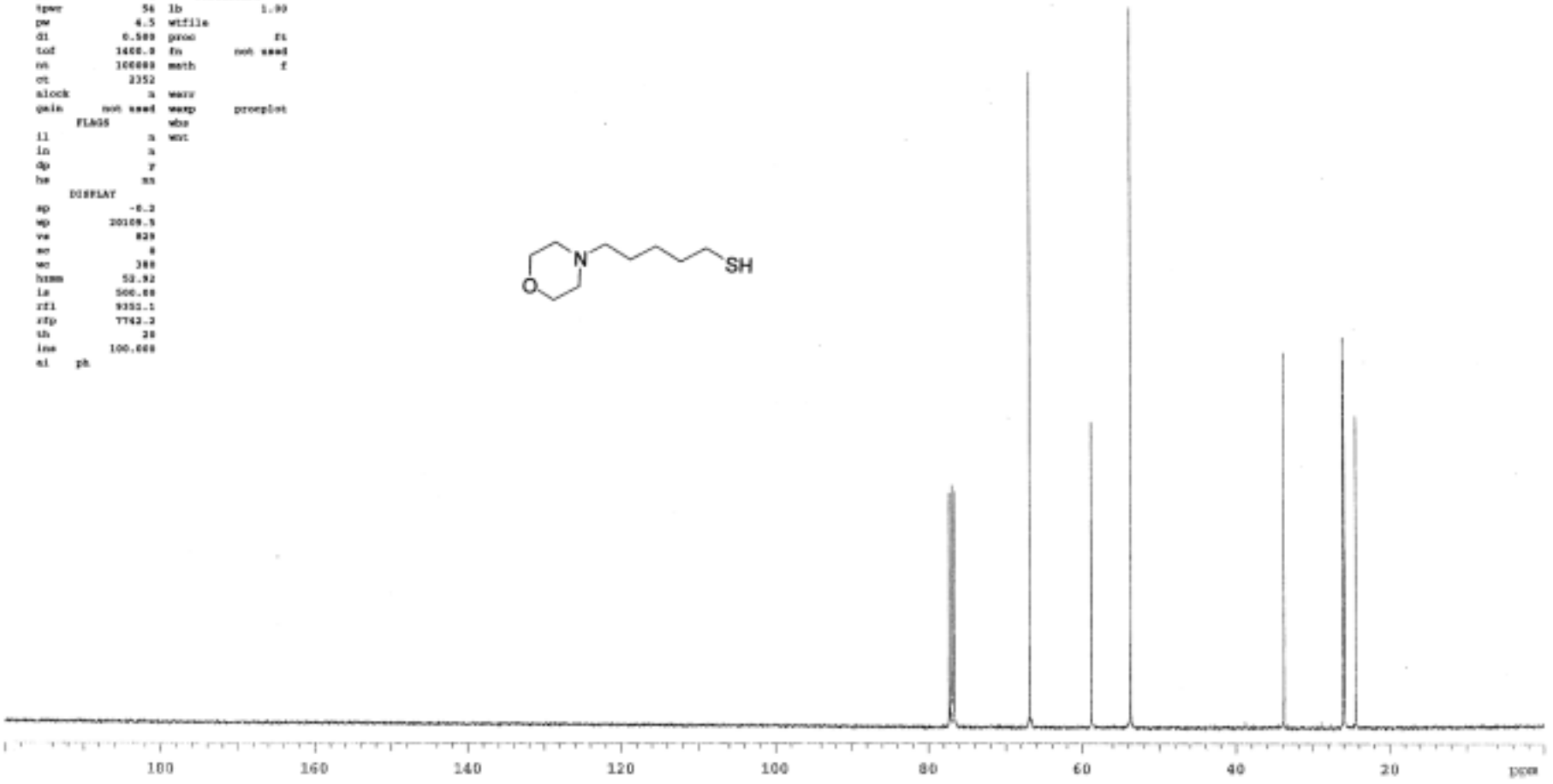

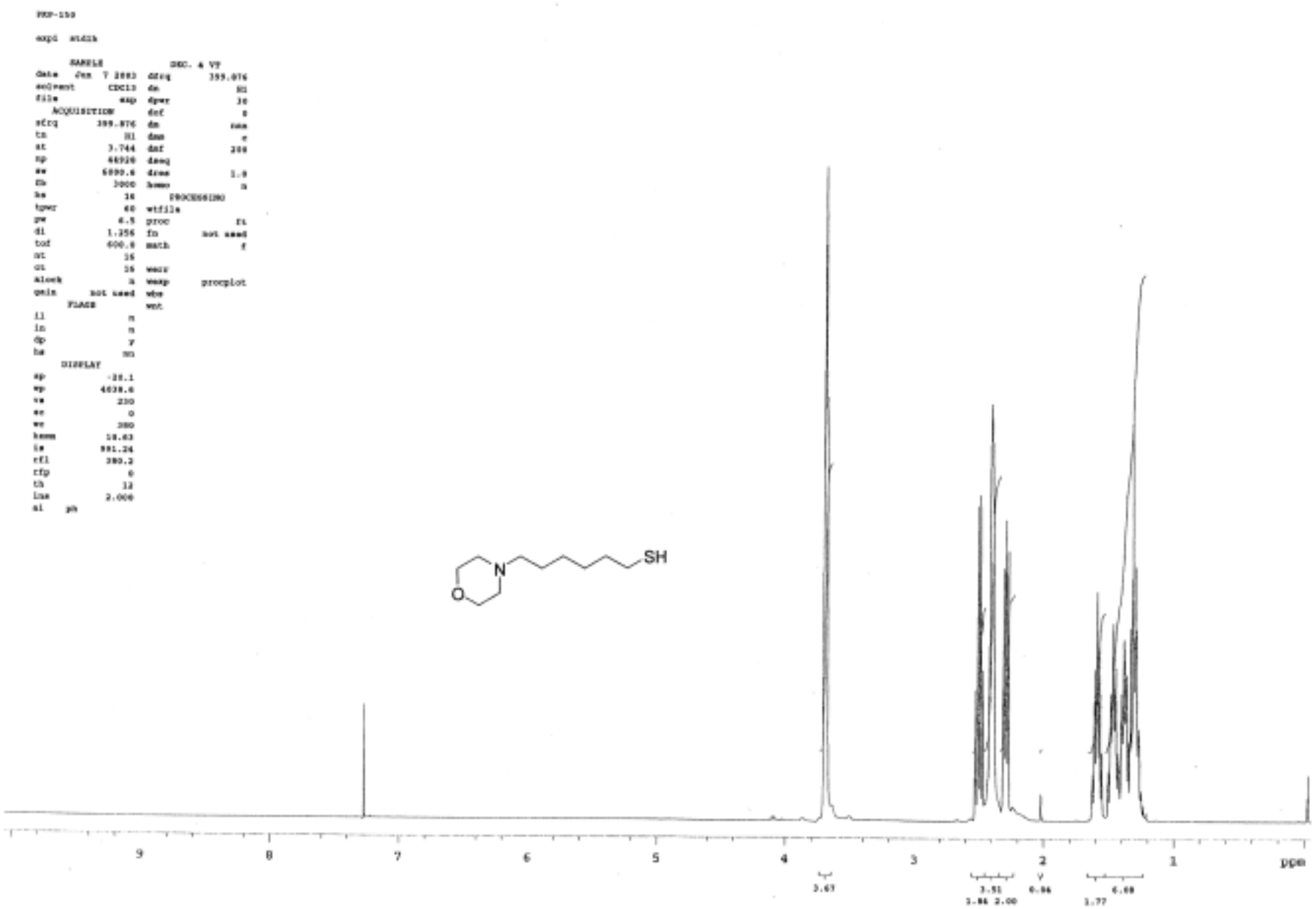
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PEP-150

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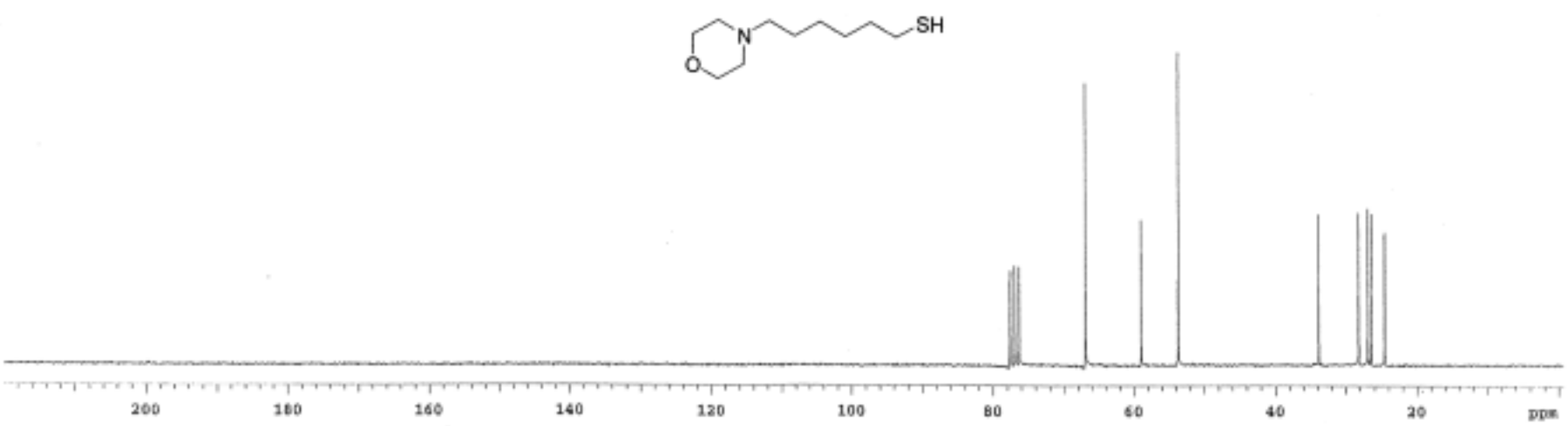

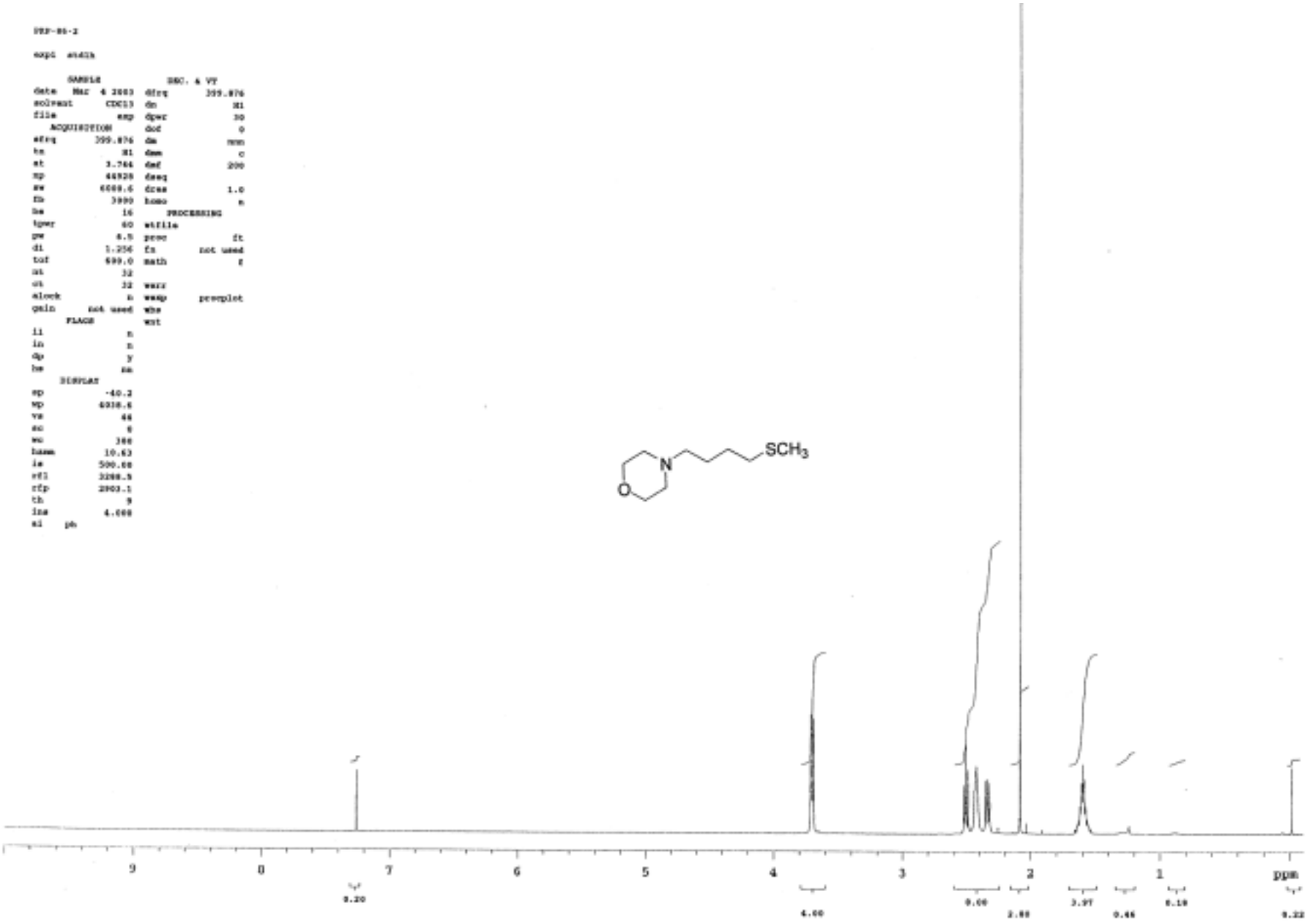
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13C OBSERVE

PKP-86

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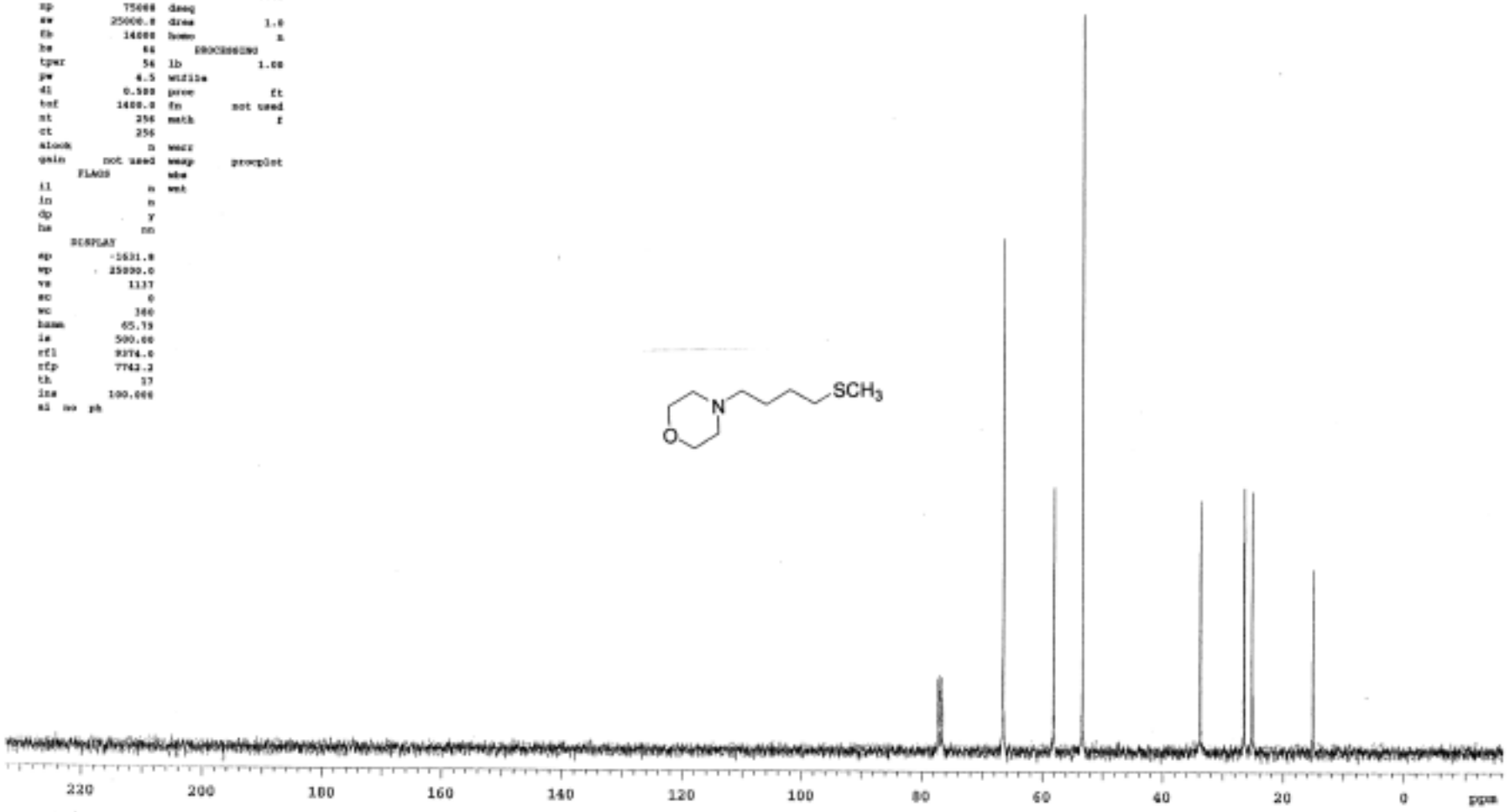
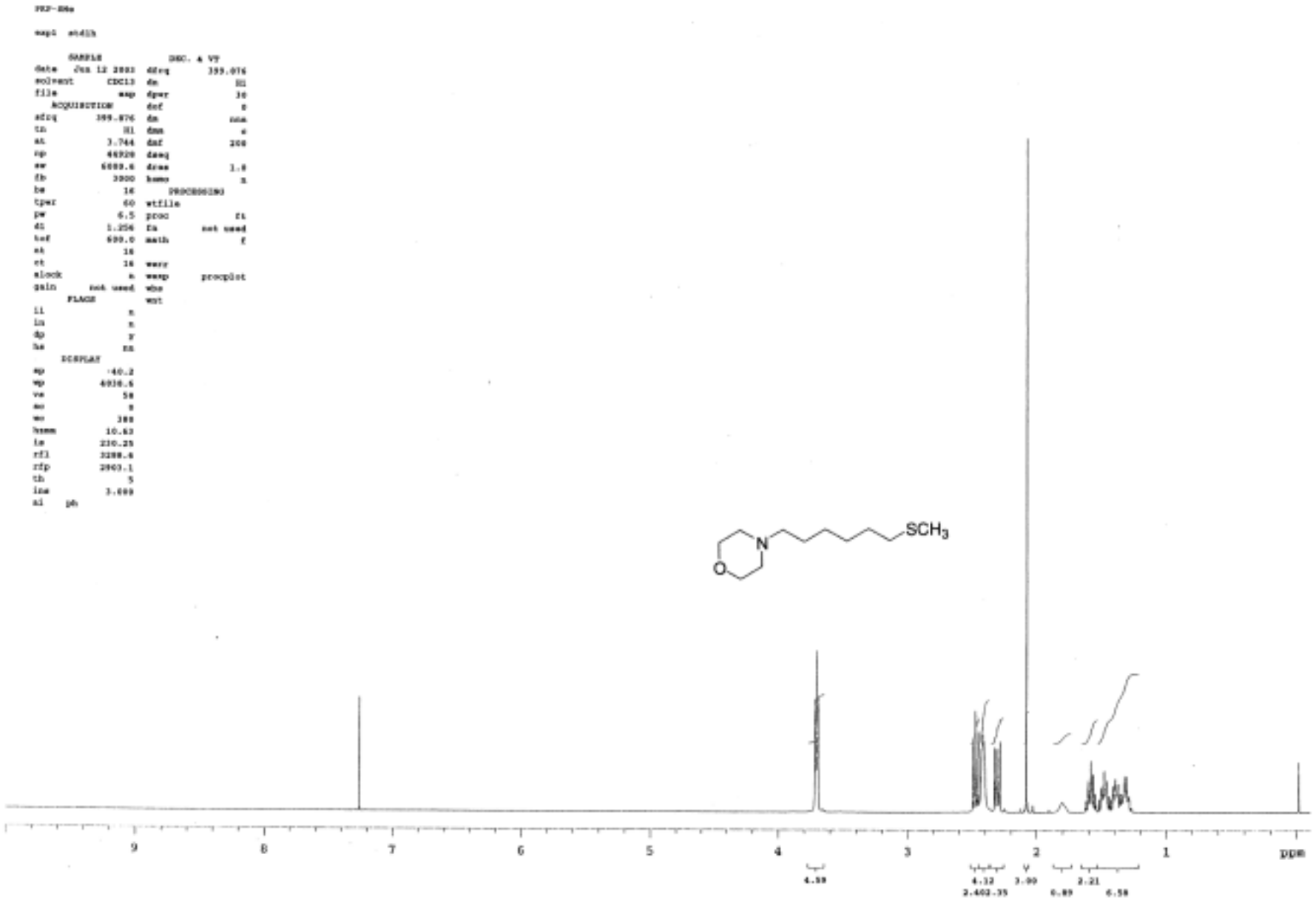
s1 ns ph

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wapl anslh
SAMPLE
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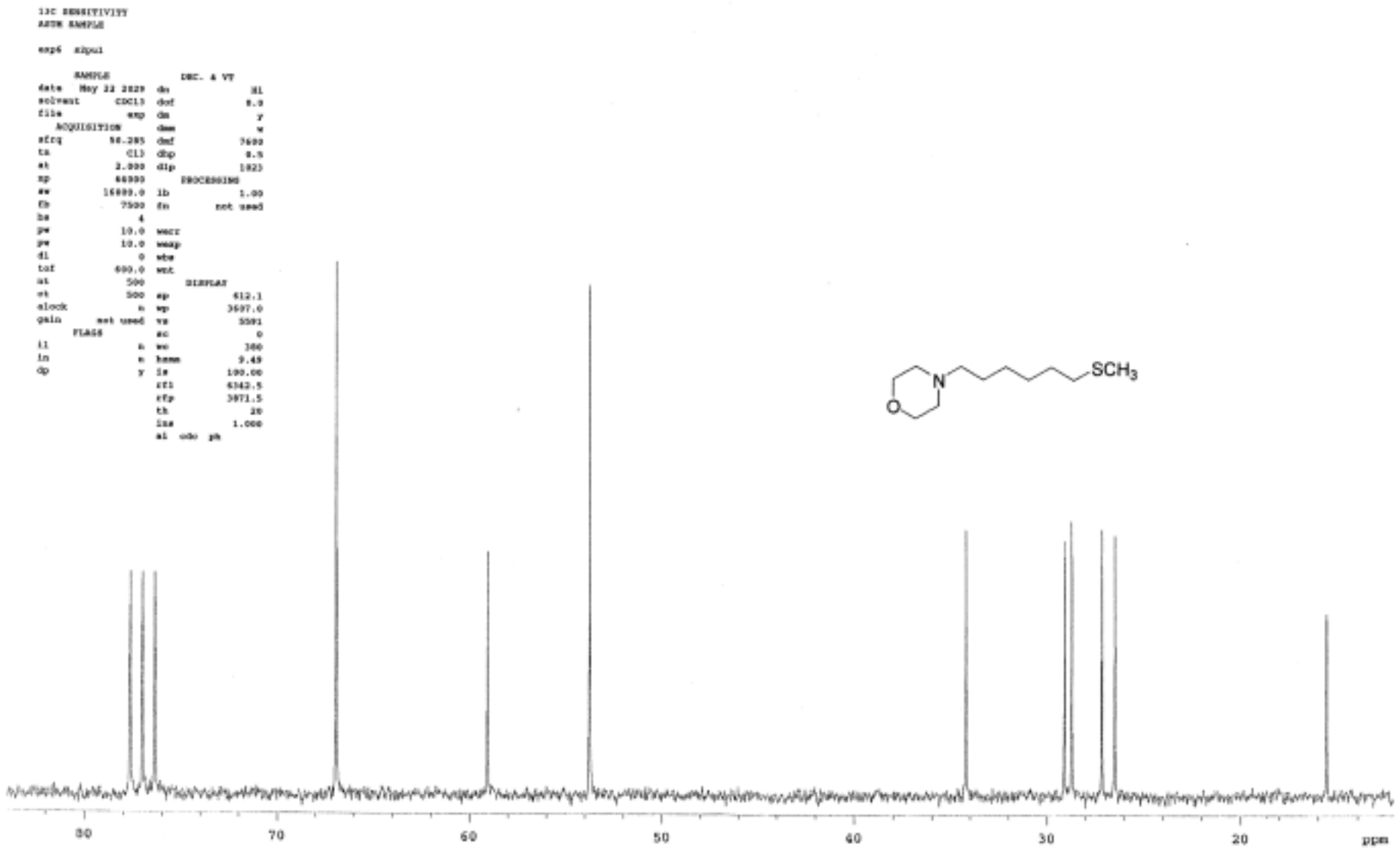
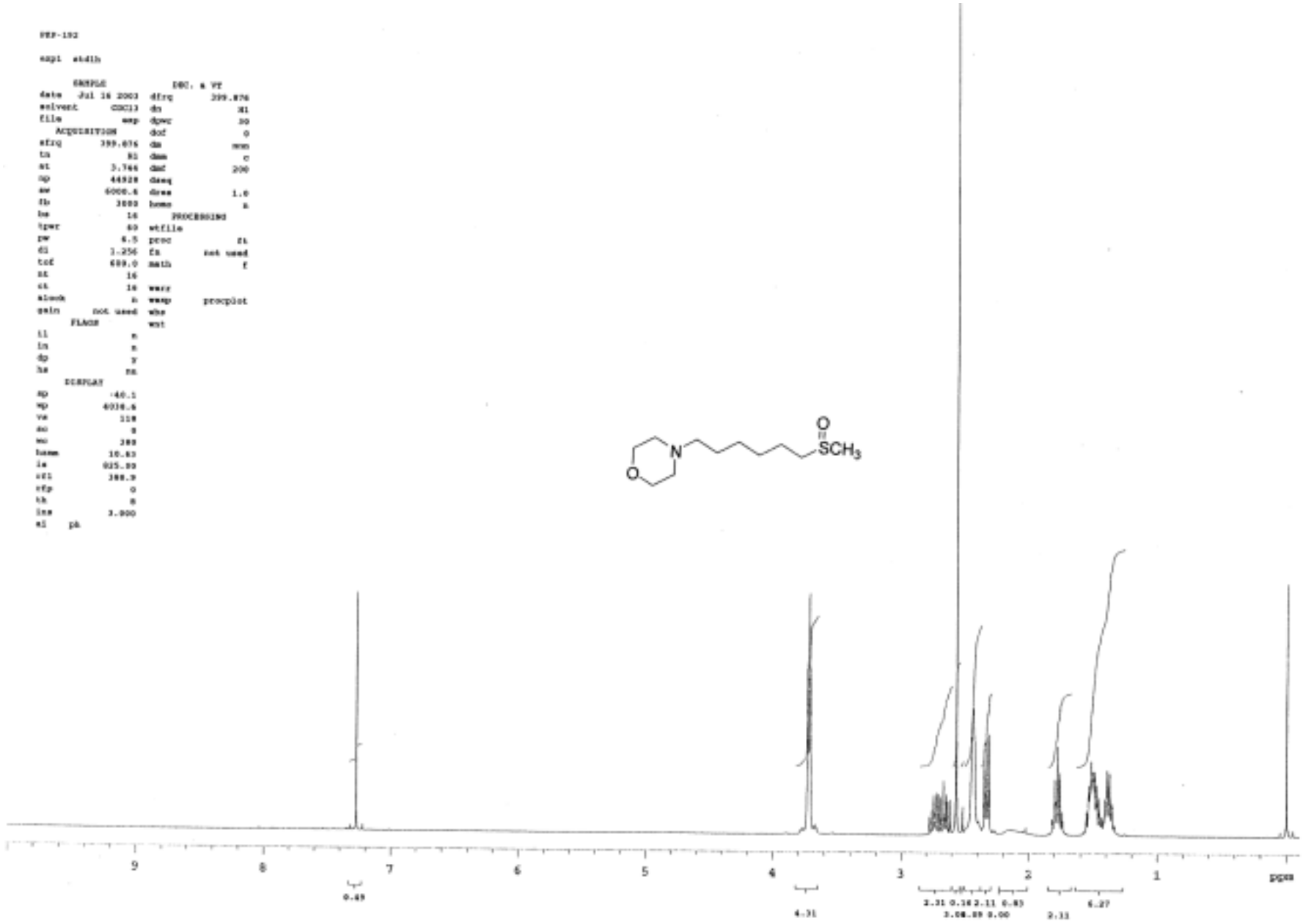
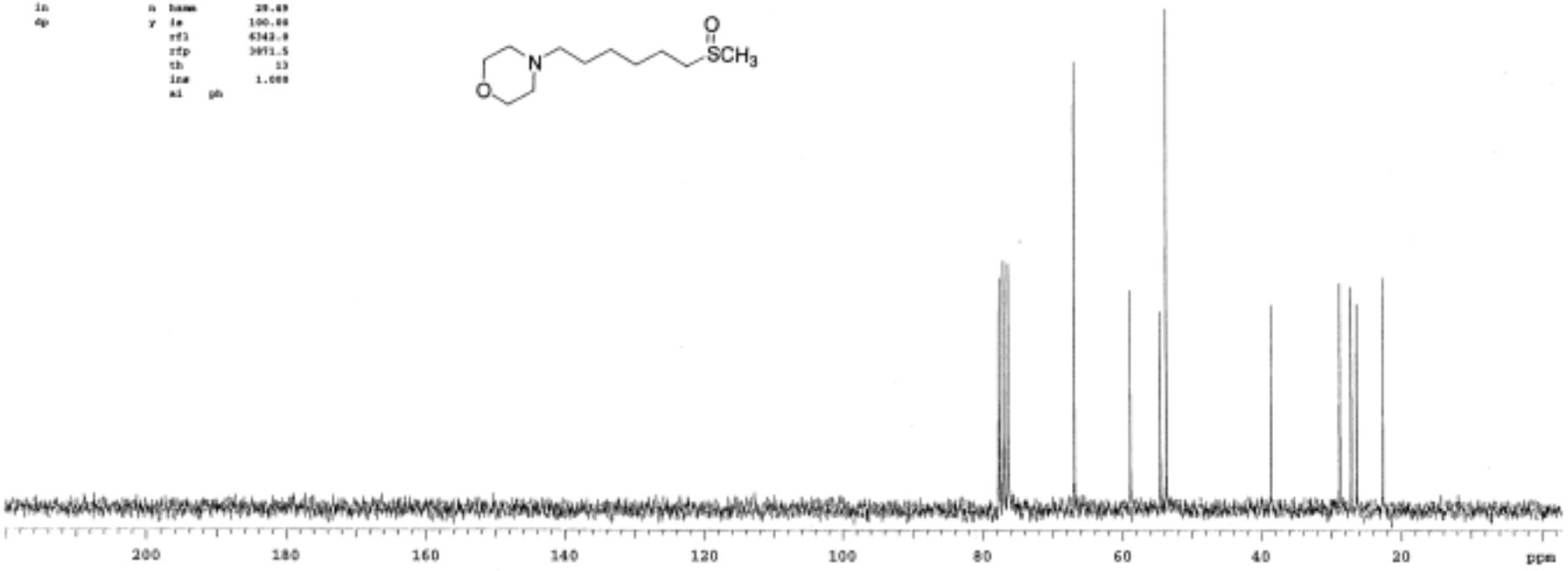
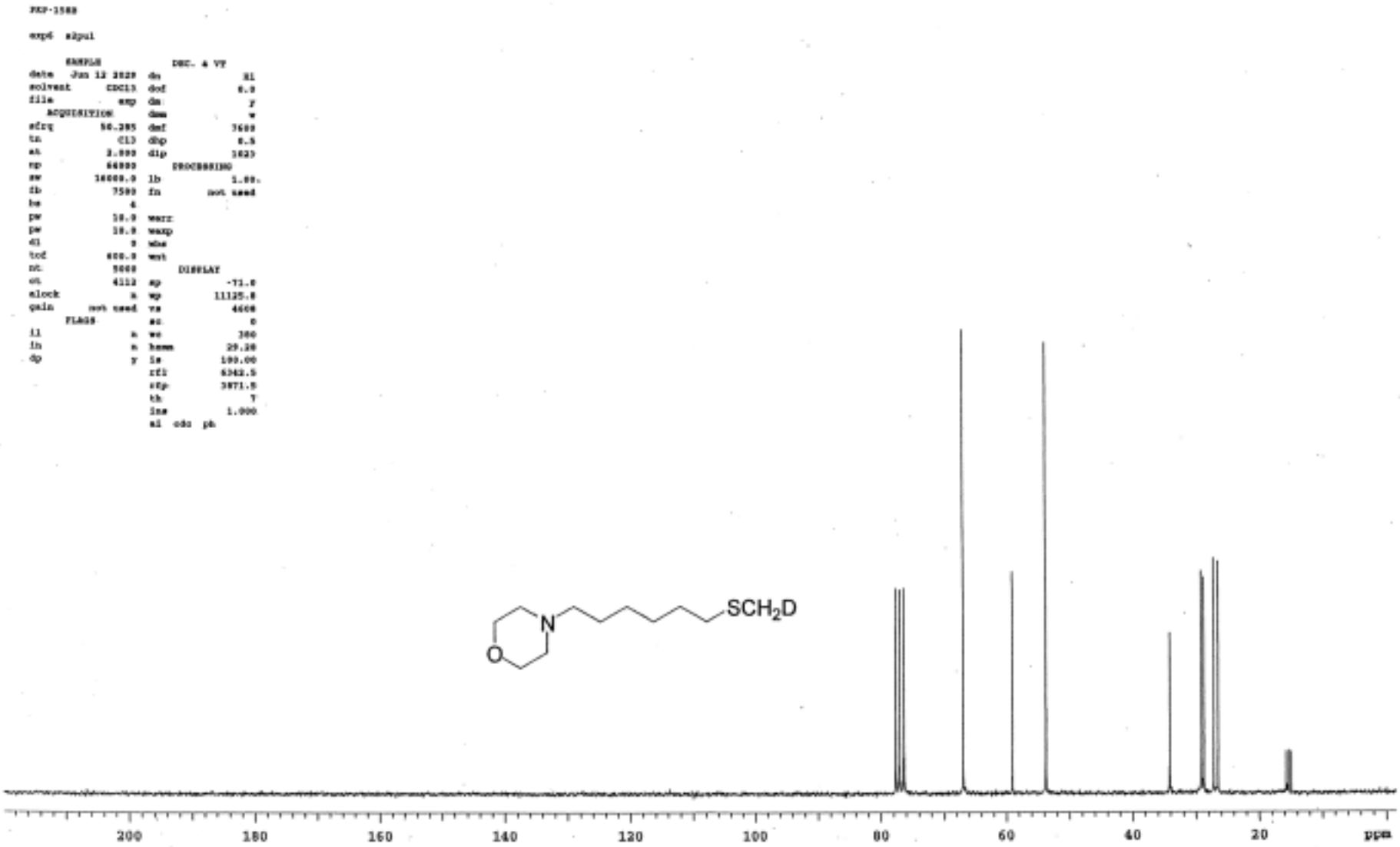
DISPLAY
sp -40.2
wp 4938.5
vs 58
sc 0
wc 288
hzmm 10.63
is 230.25
rfl 3288.6
rfp 2963.1
th 5
ins 1.000
ai ph
DEC. & VT
dfrq 399.876
dn H1
dpwr 36
dof 0
dm nnn
dmm c
dmf 200
dmq
dres 1.8
homo n
PROTON01
wtfile
proc lt
ta not used
math f
wexp
wexp procplot
who
wnt
SCH3
N
O
9
8
7
6
5
4
3
2
1
ppm
4.59
4.12
2.402.35
V
3.00
2.21
6.89
6.58

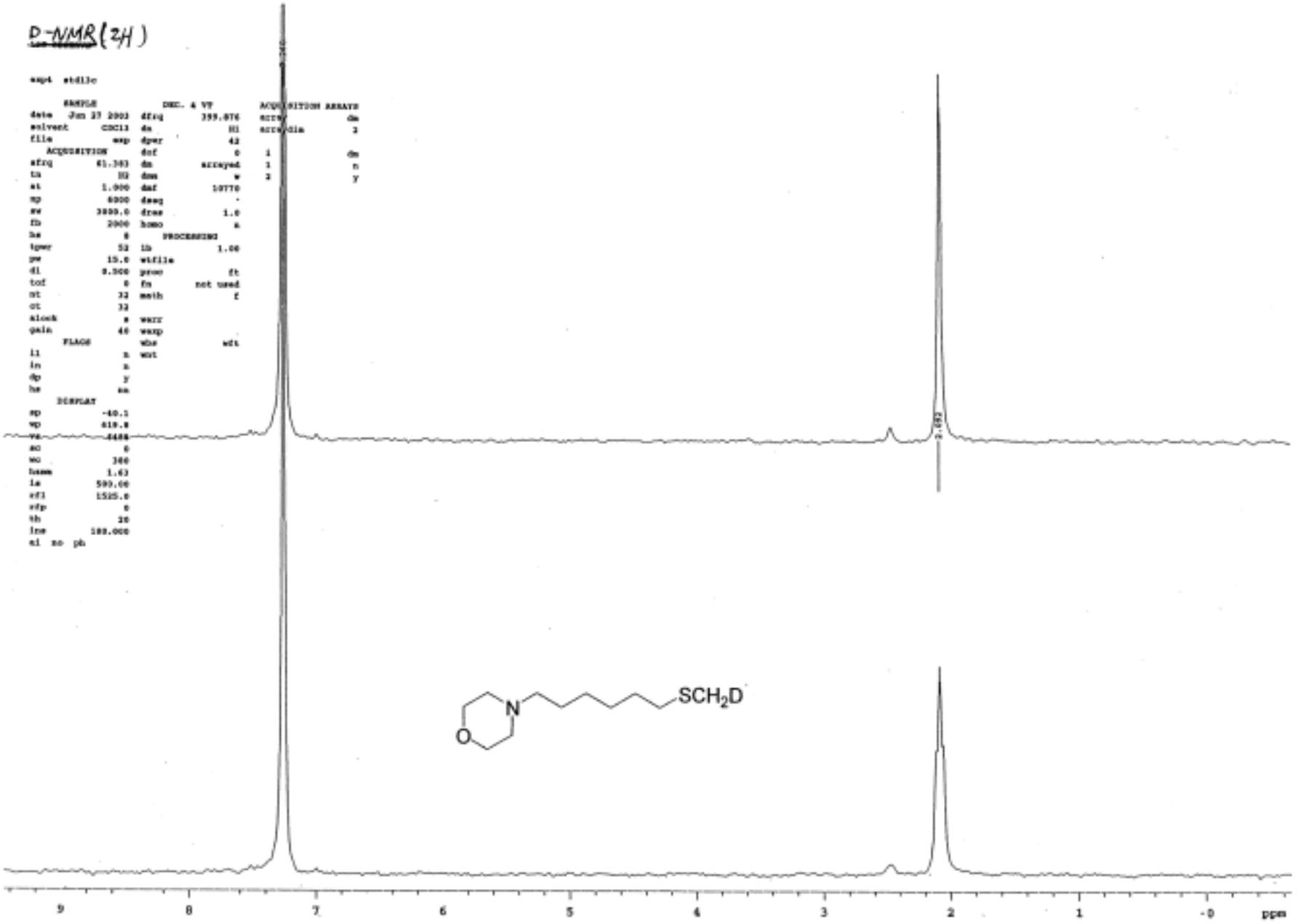

13C SENSITIVITY
AUTH SAMPLE
exp6 xlpul
SAMPLE
date May 22 2020
solvent CDCl3
file exp
ACQUISITION
sfrq 58.285
tn C13
at 2.000
np 64000
sw 16889.0
fb 7500
bs 4
pw 10.0
pw 10.0
dl 0
tof 600.0
at 500
rt 500
alock n
gain not used
FLAGS
il n
in n
dp y
DEC. & VT
dn H1
dof 0.0
dm y
dmm w
dmf 7600
dbp 8.5
dlp 1821
PROCESSING
lb 1.00
fn not used
wscr
wexp
wbs
wnt
DISPLAY
sp 612.1
wp 3597.0
vs 5591
sc 0
wc 380
hzmm 9.49
is 100.00
rfl 6342.5
rfp 3871.5
th 20
lvs 1.000
al cdc ph
SCH3
N
O
80
70
60
50
40
30
20
ppm

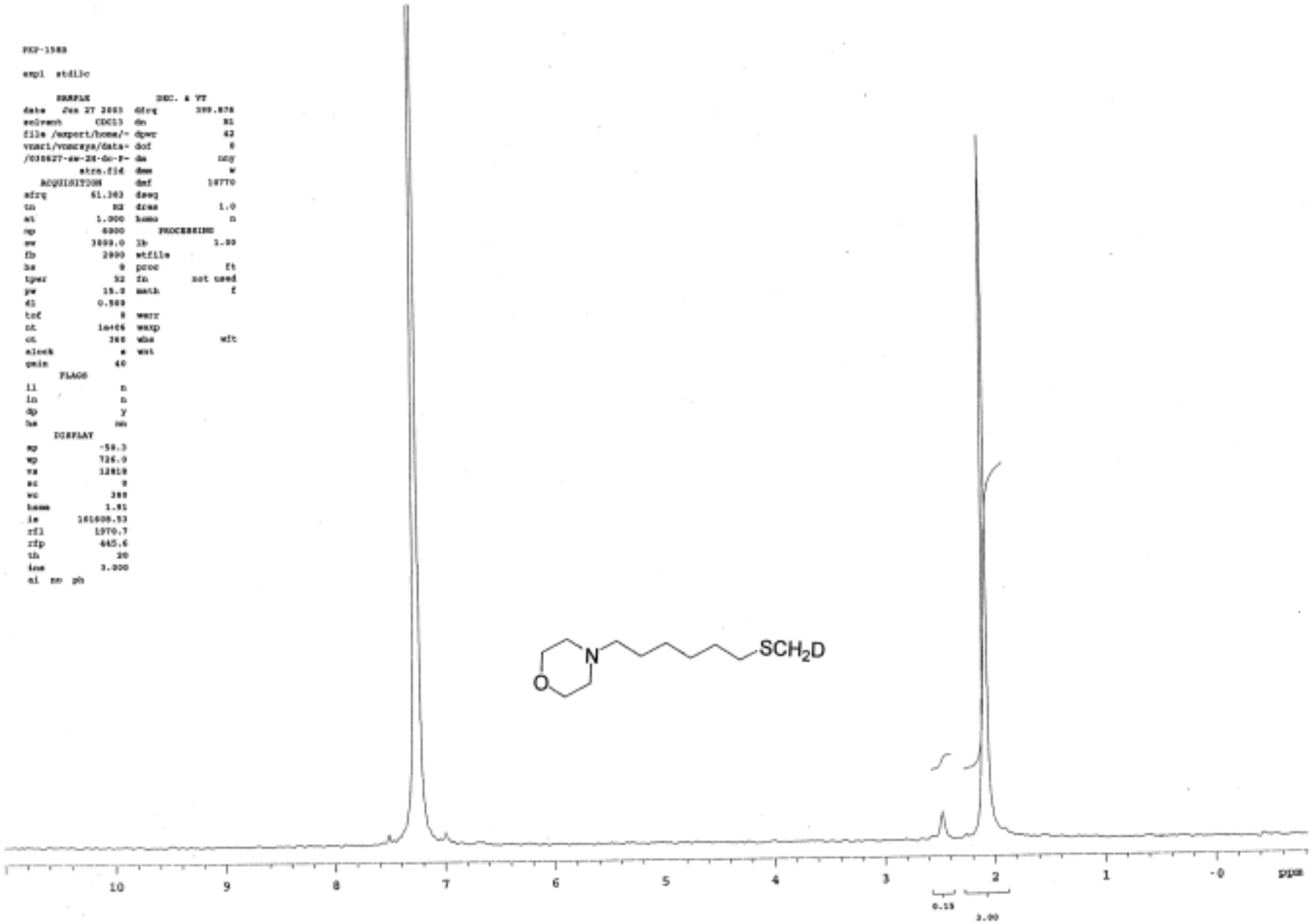

PEP-192
sapl stdih
SAMPLE
date Jul 16 2003
solvent CDCl3
file sap
ACQUISITION
sfrq 399.876
tn H1
at 3.744
np 44928
sw 6000.6
fb 3000
bs 16
tpwr 60
pw 6.5
dl 1.256
tof 699.0
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -40.1
wp 4038.6
vs 118
sc 0
wc 288
hzmm 10.63
is 925.99
rfl 388.9
rfp 0
th 8
ins 3.000
ai pk
DEC. & PP
dfrq 399.876
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
drea 1.6
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
wnrr
wnsp procplot
wbs
wnt
O
SCH3
N
O
9
8
7
6
5
4
3
2
1
ppm
0.69
4.31
2.31 0.16 2.11 0.83
3.08 0.00
2.13
6.27

```
PRP-161

exp6  s2pul

      SAMPLE              DEC. & VT
date   Jun 18 2020  dn              H1
solvent      CDCl3  dof             0.0
file          exp   dm              y
   ACQUISITION      dmm             w
sfrq        58.285  dmf          7680
tn             C13  dbp           6.5
at           2.000  dlp          1823
np          64000      PROCESSING
sw        16880.0  lb             1.00
fb           7500  fn       not used
bs              4
pw             10.0  wexr
pw1            10.0  wexp
d1              0    wba
tof          699.0  wnt
nt              750       DISPLAY
ct              750  sp          -136.9
alock             n  wp         11305.9
gain      not used  vs            7159
      FLAGS         sc               8
il                n  wc             388
in                n  hzmm         28.69
dp                y  is           100.00
                     rfl           6343.8
                     rfp           3971.5
                     th              13
                     ins           1.000
                     ai    ph
```


PKP-1588
exp6 s2pul
SAMPLE
date Jun 12 2020
solvent CDCl3
file exp
ACQUISITION
sfrq 50.285
tn C13
at 2.000
np 64000
sw 16000.0
fb 7500
bs 4
pw 10.0
pw 10.0
dl 2
tof 400.0
nt 5000
ct 4112
alock n
gain not used
FLAGS
il n
in n
dp y
DEC. & VT
dn H1
dof 0.0
dm y
dmm w
dmf 7600
dhp 0.5
dlp 1623
PROCESSING
lb 1.00
fn not used
wexr
wexp
wbs
wzt
DISPLAY
sp -71.8
wp 11125.8
vs 4600
sc 0
wc 380
hzmm 29.38
is 100.00
rfl 6362.5
rfp 3871.5
th 7
ins 1.000
ai cdc ph
N
O
SCH2D
200
180
160
140
120
100
80
60
40
20
ppm


D-NMR(2H)
wapt stdlic
SAMPLE
date Jun 27 2003
solvent CDCl3
file wsp
ACQUISITION
sfrq 61.361
tn H2
at 1.000
np 6000
sw 3000.0
fb 2000
bs 8
tpwr 52
pw 15.0
dl 0.500
tof 0
nt 32
ct 32
alock n
gain 46
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -40.1
wp 619.8
vs 4488
sc 0
wc 386
hzmm 1.63
is 500.00
rfl 1525.0
rfp 0
th 26
lvl 188.000
el no ph
DEC. & VT
dfrq 399.876
dn H1
dpwr 42
dof 0
dm arrayed
dmm w
dmf 10770
dseq -
drss 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
wexp
wbs wts
wnt
ACQUISITION ARRAYS
array dm
arrayelm 2
1 dm
1 n
2 y
2.349
2.193
N
O
SCH2D
9
8
7
6
5
4
3
2
1
0
ppm


PKP-1588
expl stdlic
SAMPLE
date Jun 27 2001
solvent CDCl3
file /export/home/~
vnmri/vnmrsys/data~
/030627-am-28-dc-P~
shns.fid
ACQUISITION
sfrq 61.383
tn H2
at 1.000
np 6000
sw 3889.0
fb 2000
bs 8
tpwr 52
pw 15.0
dl 0.500
tof 0
rt 1e+06
ct 368
alock a
gain 40
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -58.3
wp 726.9
vs 12810
sc 0
wc 388
hzmm 1.91
is 161698.53
rfl 1970.7
rfp 445.6
th 20
ins 3.000
ai nn ph
DEC. & VT
dfrq 399.878
dn H1
dpwr 42
dof 0
da nny
dmm w
dmf 10770
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs wft
wnt
N
O
SCH2D
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.59
3.00