



Supporting Information

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Supporting Information

**Efficient Iridium-catalyzed
C-H Functionalization/Silylation of Heteroarenes**

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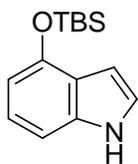
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Experimental

General. All reactions were carried out under an argon atmosphere. Anhydrous solvents were freshly distilled from sodium benzophenone ketyl, except for CH₂Cl₂, which was distilled from CaH₂. Extracts were dried over anhydrous Na₂SO₄ and then filtered prior to removal of all volatiles under reduced pressure. Unless otherwise noted, commercially available materials were used without further purification. [Ir(OMe)(COD)]₂ was purchased from Alfa Aesar. Flash chromatography (FC) was performed using *E Merck* silica gel 60 (240–400 mesh). Thin layer chromatography was performed using precoated plates purchased from *E. Merck* (silica gel 60 PF254, 0.25 mm).

Nuclear magnetic resonance (NMR) spectra were recorded on a *Varian* 300 spectrometer at operating frequencies of 300 MHz (¹H NMR) or 75 MHz (¹³C NMR). Chemical shifts (δ) are given in ppm relative to residual solvent (usually chloroform δ 7.27 for ¹H NMR or δ 77.23 for proton decoupled ¹³C NMR), and coupling constants (*J*) in Hz. Multiplicity is tabulated as s for singlet, d for doublet, t for triplet, q for quadruplet, and m for multiplet, whereby the prefix *app* is applied in cases where the true multiplicity is unresolved, and *br* when the signal in question is broadened. The Michigan State University Mass Spectroscopy Facility provided high-resolution mass spectral analyses.



4-(*tert*-Butyldimethylsilyloxy)indole (5)^[1]

To a solution of 4-hydroxyindole (40 mg, 0.3 mmol) in dry DMF (0.5 mL) was added imidazole (37 mg, 0.54 mmol) and TBSCl (52 mg, 0.32 mmol). The mixture was stirred at room temperature for 3 h. EtOAc (5 mL) and H₂O (5 mL) were added. The organic layer was separated and the aqueous layer was extracted with EtOAc (3 × 3 mL). The combined organic extracts were dried with Na₂SO₄ and concentrated under reduced pressure. The residue was purified by flash chromatography using silica gel to give 78

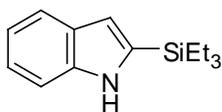
mg (100%) of 4-(*tert*-butyldimethylsilyloxy)indole.

^1H NMR (300 MHz, CDCl_3) δ 8.09 (br, 1H), 7.10-7.01 (m, 3H), 6.60 (s, 1H), 6.54 (d, J = 6.0 Hz, 1H), 1.08 (s, 9H), 0.25 (s, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 149.3, 138.0, 122.9 (2 \times C), 121.9, 108.9, 104.9, 100.6, 26.0, 18.6, -4.1.

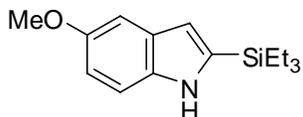
General procedure for the 2-silylation of heteroarenes with triethylsilane. A flamed-dried Schlenk tube was charged with heteroarene (0.2 mmol), $[\text{Ir}(\text{OMe})(\text{COD})]_2$ (6.6 mg, 0.01 mmol) and dtpy (5.4 mg, 0.02 mmol), then evacuated and flushed with argon three times. Under a positive flow of argon, 2-norbornene (56 mg, 0.6 mmol) and dry THF (1 mL) were added. After stirring for 5 min, triethylsilane (98 μL , 0.6 mmol) was added dropwise. The reaction mixture was heated at 80°C for 24 h or indicated time. The solvent was concentrated under reduced pressure. The residue was purified by flash chromatography using silica gel to give 2-(triethylsilyl)heteroarene.

For 1-tosyl-indole, the 3-silylation product was obtained using above general procedure.

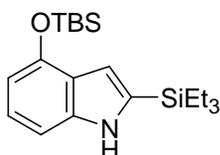
In case of furan (0.2 mmol) and thiophene (0.2 mmol), 2-norbornene (75 mg, 0.8mmol) and triethylsilane (130 μL , 0.6 mmol) were employed.



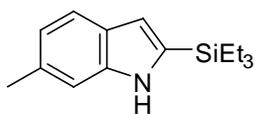
2-(Triethylsilyl)indole (2). 87% yield; ^1H NMR (300 MHz, CDCl_3) δ 8.10 (br, 1H), 7.66-7.64 (m, 1H), 7.41 (dd, J = 8.1 Hz, 0.9 Hz, 1H), 7.22-7.16 (m, 1H), 7.13-7.07 (m, 1H), 6.76-6.75 (m, 1H), 1.07-1.01 (m, 9H), 0.89-0.81 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 138.8, 135.8, 128.9, 122.4, 120.7, 119.8, 112.6, 111.0, 7.7, 3.8. HRMS (ESI) for $\text{C}_{14}\text{H}_{22}\text{NSi}$ [MH^+] Calc 232.1522, Found 232.1517.



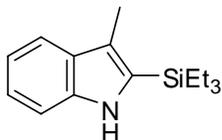
5-Methoxyl-2-triethylsilyl-indole (4). 90% yield; ^1H NMR (300 MHz, CDCl_3) δ 8.05 (br, 1H), 7.30 (d, $J = 9.0$ Hz, 1H), 7.11 (s, 1H), 6.88 (dd, $J = 8.4$ Hz, 2.1 Hz, 1H), 6.69 (s, 1H), 3.87 (s, 3H), 1.07-1.02 (m, 9H), 0.89-0.81 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 154.3, 136.7, 134.2, 129.3, 113.0, 112.2, 111.7, 101.9, 56.1, 7.7, 3.8. HRMS (ESI) for $\text{C}_{15}\text{H}_{24}\text{NOSi}$ [MH^+] Calc 262.1627 Found 262.1620.



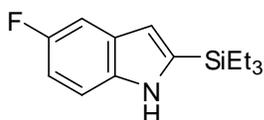
4-(*tert*-Butyldimethylsilyloxy)-2-(triethylsilyl)-indole (6). 82% yield; m.p 62-63 $^\circ\text{C}$; ^1H NMR (300 MHz, CDCl_3) δ 8.03 (br, 1H), 7.02 (s, 1H), 7.01 (d, $J = 1.8$ Hz, 1H), 6.74 (d, $J = 2.4$ Hz, 1H), 6.48 (dd, $J = 4.8$ Hz, 3.0 Hz, 1H), 1.06 (s, 9H), 1.05-1.00 (m, 9H), 0.87-0.79 (m, 6H), 0.24 (s, 6H); ^{13}C NMR (75 MHz, CDCl_3): δ 149.0, 140.9, 134.1, 123.1, 122.8, 110.2, 108.5, 104.6, 26.1, 18.6, 7.7, 3.8, -4.0. HRMS (ESI) for $\text{C}_{20}\text{H}_{36}\text{NOSi}_2$ [MH^+] Calc 362.2335 Found 362.2338.



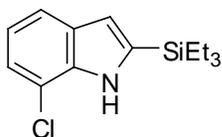
6-Methyl-2-triethylsilylindole (8). 86% yield; ^1H NMR (300 MHz, CDCl_3) δ 7.97 (br, 1H), 7.52 (d, $J = 7.5$ Hz, 1H), 7.20 (s, 1H), 6.93 (d, $J = 8.1$ Hz, 1H), 6.70 (s, 1H), 2.47 (s, 3H), 1.06-1.00 (m, 9H), 0.88-0.80 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 139.4, 135.0, 132.2, 126.8, 121.6, 120.3, 112.4, 110.9, 22.0, 7.7, 3.8. HRMS (ESI) for $\text{C}_{15}\text{H}_{24}\text{NSi}$ [MH^+] Calc 246.1678 Found 246.1682.



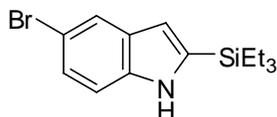
3-Methyl-2-triethylsilylindole (10). 55% yield; ^1H NMR (300 MHz, CDCl_3) δ 7.87 (br, 1H), 7.62-7.58 (m, 1H), 7.38-7.35 (m, 1H), 7.22-7.16 (m, 1H), 7.14-7.09 (m, 1H), 2.42 (s, 3H), 1.06-1.01 (m, 9H), 0.95-0.87 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 138.5, 130.9, 129.7, 122.3, 121.1, 119.1, 118.8, 110.9, 10.8, 7.7, 3.9. HRMS (ESI) for $\text{C}_{15}\text{H}_{23}\text{NSi}$ [M^+] Calc 245.1600 Found 245.1606.



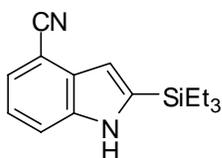
5-Fluoro-2-triethylsilylindole (12). 76% yield; ^1H NMR (300 MHz, CDCl_3) δ 8.11 (br, 1H), 7.34-7.26 (m, 2H), 6.99-6.92 (m, 1H), 6.72-6.71 (m, 1H), 1.08-1.02 (m, 9H), 0.90-0.82 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 158.1 ($J = 233.0$ Hz), 138.2, 135.5, 129.2 ($J = 9.7$ Hz), 112.5 ($J = 4.9$ Hz), 111.4 ($J = 9.7$ Hz), 110.8 ($J = 26.0$ Hz), 105.2 ($J = 22.4$ Hz), 7.7, 3.7; HRMS (ESI) for $\text{C}_{14}\text{H}_{21}\text{NFSi}$ [MH^+] Calc 250.1427 Found 250.1425.



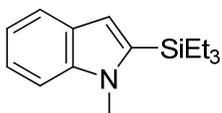
7-Chloro-2-triethylsilylindole (14). 64% yield; ^1H NMR (300 MHz, CDCl_3) δ 8.21 (br, 1H), 7.54 (dd, $J = 8.1$ Hz, 0.9 Hz, 1H), 7.18 (dd, $J = 7.8$ Hz, 0.9 Hz, 1H), 7.03 (t, $J = 7.5$ Hz, 1H), 6.78 (d, $J = 2.1$ Hz, 1H), 1.08-1.02 (m, 9H), 0.91-0.83 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 137.0, 136.1, 130.4, 121.6, 120.6, 119.3, 116.5, 113.5, 7.7, 3.7; HRMS (ESI) for $\text{C}_{14}\text{H}_{21}\text{NSiCl}$ [MH^+] Calc 266.1132 Found 266.1133.



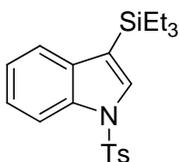
6-Bromo-2-triethylsilylindole (16). 52% yield; ^1H NMR (300 MHz, CDCl_3) δ 8.12 (br, 1H), 7.75 (s, 1H), 7.26-7.25 (m, 2H), 6.66 (s, 1H), 1.02-0.96 (m, 9H), 0.88-0.80 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 137.6, 137.4, 130.7, 125.1, 123.1, 113.0, 112.3, 112.0, 7.7, 3.7; HRMS (ESI) for $\text{C}_{14}\text{H}_{21}\text{NSiBr}$ [MH^+] Calc 310.0627 Found 310.0624.



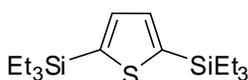
4-Nitro-2-triethylsilylindole (18). 41% yield; m.p 131-133 $^\circ\text{C}$; ^1H NMR (300 MHz, CDCl_3) δ 8.51 (br, 1H), 7.64 (dd, $J = 8.4$ Hz, 0.4 Hz, 1H), 7.46 (d, $J = 7.5$ Hz, 1H), 7.23-7.18 (m, 1H), 6.93-6.92 (m, 1H), 1.06-1.01 (m, 9H), 0.92-0.84 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 140.0, 138.6, 130.1, 125.3, 121.8, 119.3, 115.8, 111.0, 102.7, 7.6, 3.6. HRMS (ESI) for $\text{C}_{15}\text{H}_{21}\text{N}_2\text{Si}$ [MH^+] Calc 257.1474 Found 257.1470.



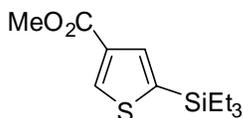
N-Methyl-2-triethylsilylindole (20). 49% yield; ^1H NMR (300 MHz, CDCl_3) δ 7.62 (d, $J = 7.8$ Hz, 1H), 7.34 (d, $J = 8.1$ Hz, 1H), 7.25-7.20 (m, 1H), 7.09 (t, $J = 7.2$ Hz, 1H), 6.73 (s, 1H), 3.84 (s, 3H), 1.04-0.99 (m, 9H), 0.95-0.88 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 140.4, 138.5, 128.7, 122.0, 120.7, 119.2, 113.1, 109.2, 33.2, 7.8, 4.2; HRMS (ESI) for $\text{C}_{15}\text{H}_{24}\text{NSi}$ [MH^+] Calc 246.1678 Found 246.1676.



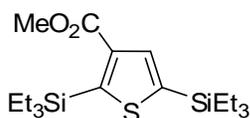
N-Tosyl-3-triethylsilylindole (22). 70% yield; ^1H NMR (300 MHz, CDCl_3) δ 7.97-7.94 (m, 1H), 7.76 (d, $J = 7.8$ Hz, 2H), 7.60-7.56 (m, 1H), 7.55 (s, 1H), 7.30-7.18 (m, 4H), 2.33 (s, 3H), 0.99-0.94 (m, 9H), 0.90-0.82 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 145.1, 136.1, 135.5, 135.3, 132.8, 130.1, 127.0, 124.4, 123.3, 122.7, 115.4, 113.7, 21.8, 7.7, 3.9. HRMS (ESI) for $\text{C}_{21}\text{H}_{28}\text{NO}_2\text{SSi}$ [MH^+] Calc 386.1610 Found 386.1607.



2,5-Bis(triethylsilyl)thiophene (24). 98% yield; ^1H NMR (300 MHz, CDCl_3) δ 7.33 (s, 2H), 1.04-0.98 (m, 18H), 0.85-0.77 (m, 12H); ^{13}C NMR (75 MHz, CDCl_3) δ 142.5, 135.6, 7.7, 4.8. HRMS (EI) for $\text{C}_{16}\text{H}_{32}\text{SSi}_2$ [M^+] Calc 312.1763 Found 312.1762.

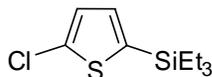


Methyl 5-(triethylsilyl)thiophene-3-carboxylate (26). 67% yield; ^1H NMR (300 MHz, CDCl_3) δ 8.32 (d, $J = 0.9$ Hz, 1H), 7.64 (d, $J = 0.9$ Hz, 1H), 3.86 (s, 3H), 1.02-0.96 (m, 9H), 0.85-0.77 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 163.8, 138.6, 138.0, 135.6, 135.0, 52.0, 7.5, 4.4. HRMS (ESI) for $\text{C}_{12}\text{H}_{21}\text{O}_2\text{SiS}$ [MH^+] Calc 257.1032 Found 257.1035.

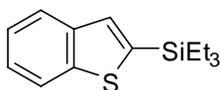


Methyl 2,5-bis(triethylsilyl)thiophene-3-carboxylate 33% yield; ^1H NMR (300 MHz, CDCl_3) δ 7.77 (s, 1H), 3.86 (s, 3H), 1.02-0.94 (m, 24H), 0.81 (t, $J = 7.5$ Hz, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 164.6, 152.2, 142.7, 140.0, 138.5, 51.7, 7.9, 7.6, 4.6, 4.1.

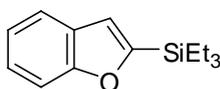
HRMS (ESI) for $C_{18}H_{38}O_2Si_2S$ [MH^+] Calc 371.1896 Found 371.1894.



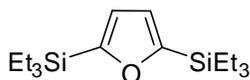
5-Chloro-2-triethylsilylthiophene (28). 91% yield; 1H NMR (300 MHz, $CDCl_3$) δ 7.00 (d, $J = 3.6$ Hz, 1H), 6.97 (d, $J = 3.6$ Hz, 1H), 1.02-0.96 (m, 9H), 0.81-0.73 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.0, 134.6, 134.3, 127.6, 7.5, 4.4. HRMS (EI) for $C_{10}H_{17}SSiCl$ [M^+] Calc 232.0509 Found 232.0508.



2-Triethylsilyl benzothiophene (30). 99% yield; 1H NMR (300 MHz, $CDCl_3$) δ 7.91-7.88 (m, 1H), 7.84-7.81 (m, 1H), 7.47 (s, 1H), 7.34-7.31 (m, 2H), 1.07-1.01 (m, 9H), 0.91-0.83 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.8, 141.2, 139.2, 131.8, 124.3, 124.1, 123.6, 122.4, 7.6, 4.5. HRMS (EI) for $C_{14}H_{20}SSi$ [M^+] Calc 248.1055 Found 248.1048.

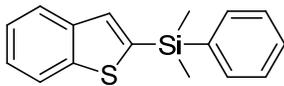


2-Triethylsilyl benzofuran (32). 83% yield; 1H NMR (300 MHz, $CDCl_3$) δ 7.60-7.57 (m, 1H), 7.53-7.50 (m, 1H), 7.30-7.16 (m, 2H), 6.99 (s, 1H), 1.07-1.01 (m, 9H), 0.88-0.81 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 161.6, 158.1, 128.0, 124.1, 122.2, 120.9, 117.2, 111.3, 7.3, 3.1. HRMS (EI) for $C_{14}H_{20}OSi$ [M^+] Calc 232.1283 Found 232.1283 .



2,5-Bis(triethylsilyl)furan (34).^[2] 93% yield; 1H NMR (300 MHz, $CDCl_3$) δ 6.62 (s, 2H), 1.02-0.96 (m, 18H), 0.80-0.71 (m, 12H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.8, 120.1, 7.6,

3.6.



2-Dimethyl(phenyl)silyl benzothiophene (35). 92% yield; ^1H NMR (300 MHz, CDCl_3) δ 7.90-7.80 (m, 2H), 7.64-7.61 (m, 2H), 7.50 (s, 1H), 7.42-7.31 (m, 5H); 0.67 (s, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 144.1, 141.2, 140.3, 137.4, 134.2, 132.5, 129.8, 128.2, 124.5, 124.3, 123.8, 122.4, -1.4. HRMS (EI) for $\text{C}_{16}\text{H}_{16}\text{SSi}$ [M^+] Calc 268.0742 Found 268.0741.

References

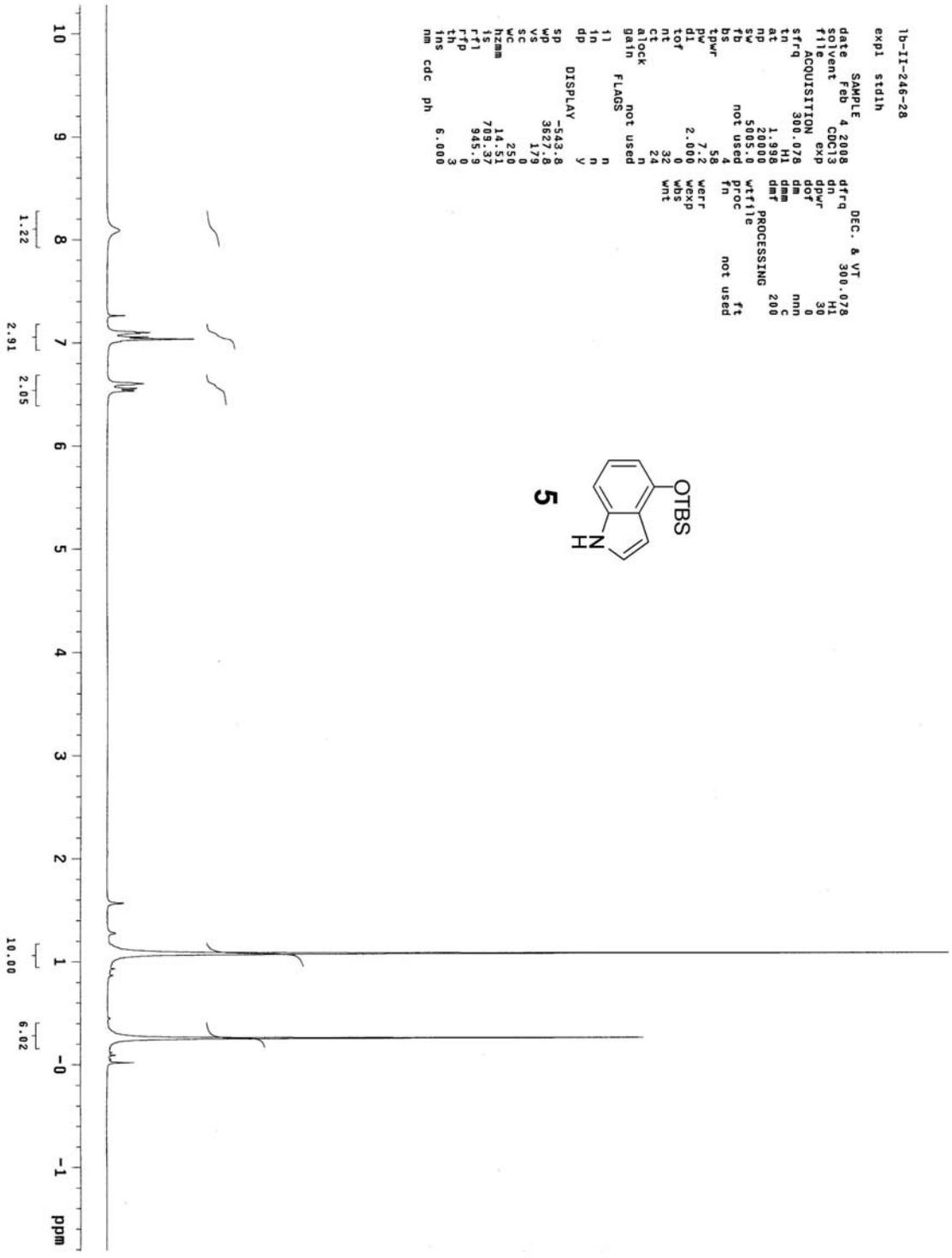
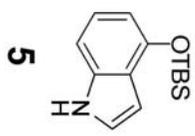
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- [2] J-P. Bouillon, D. Saleur, C. Portella, *Synthesis*, **2000**, 843.

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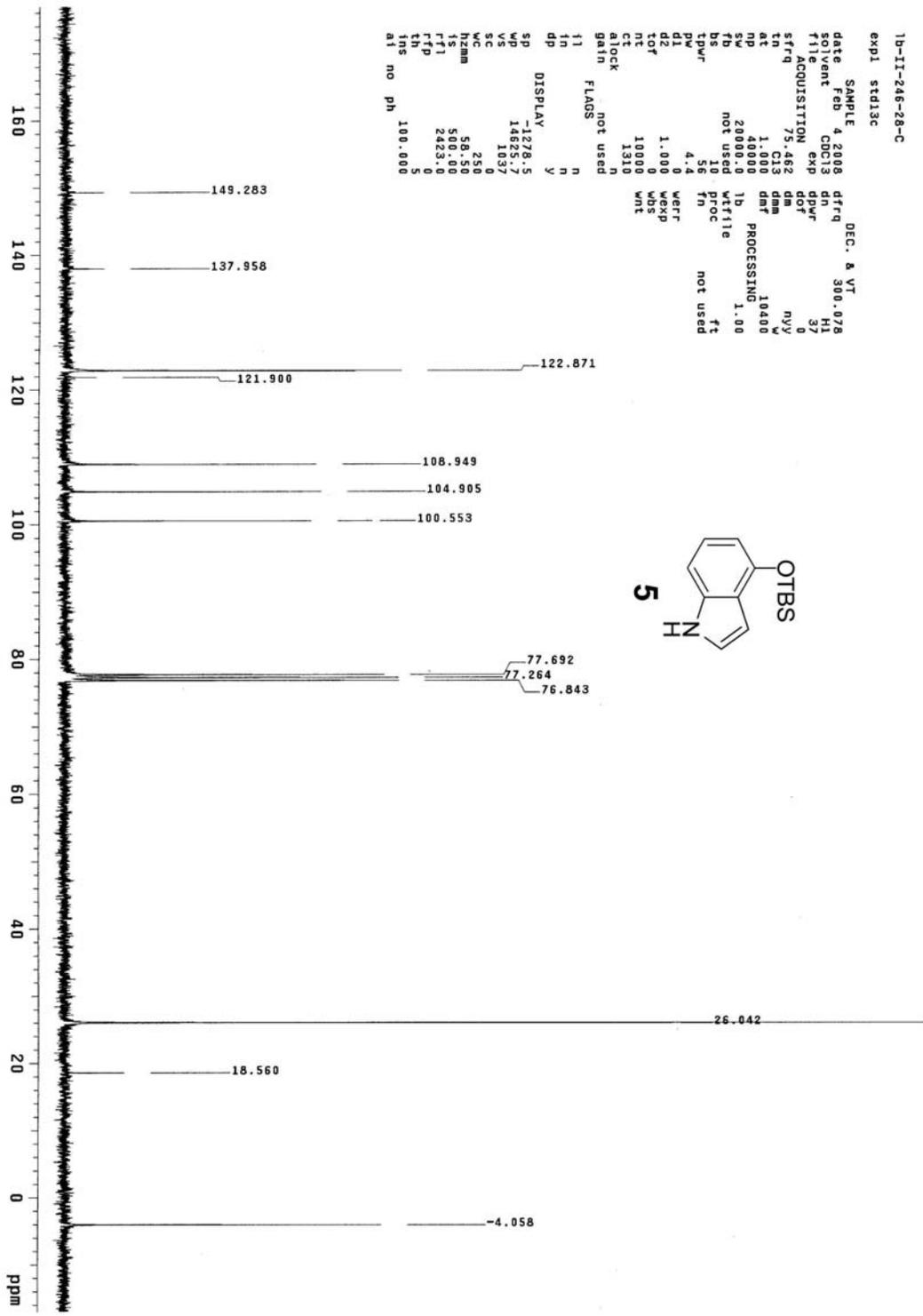
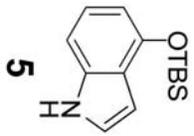


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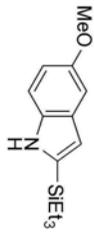
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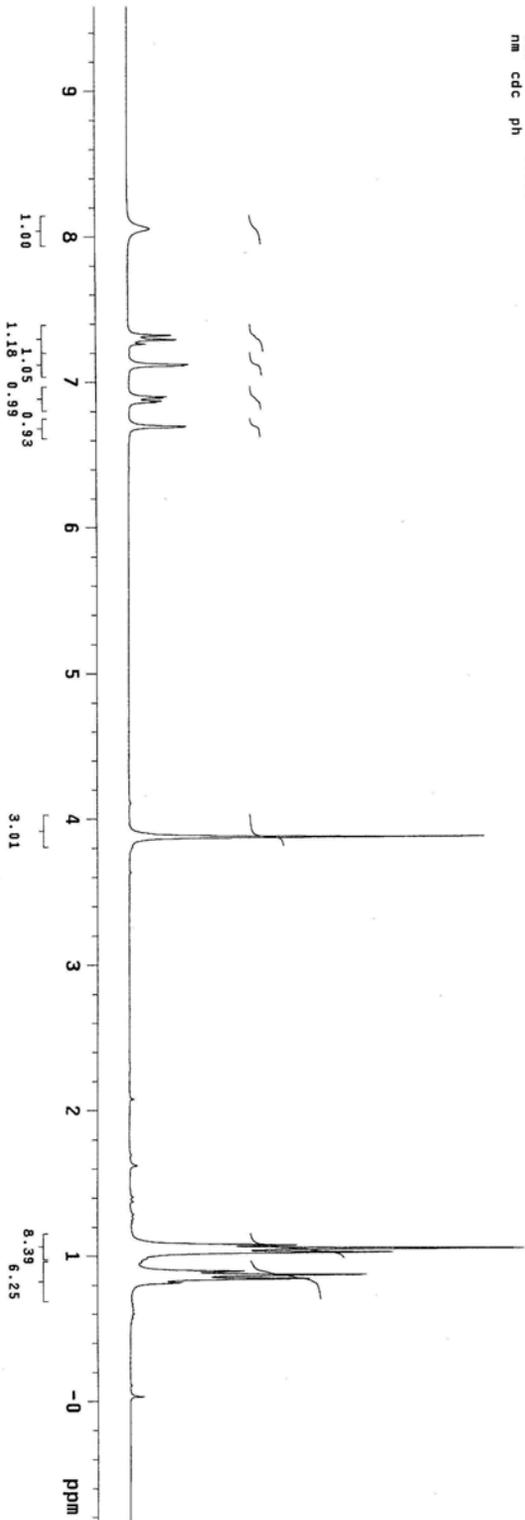
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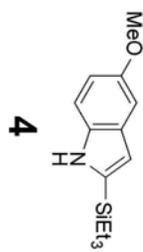
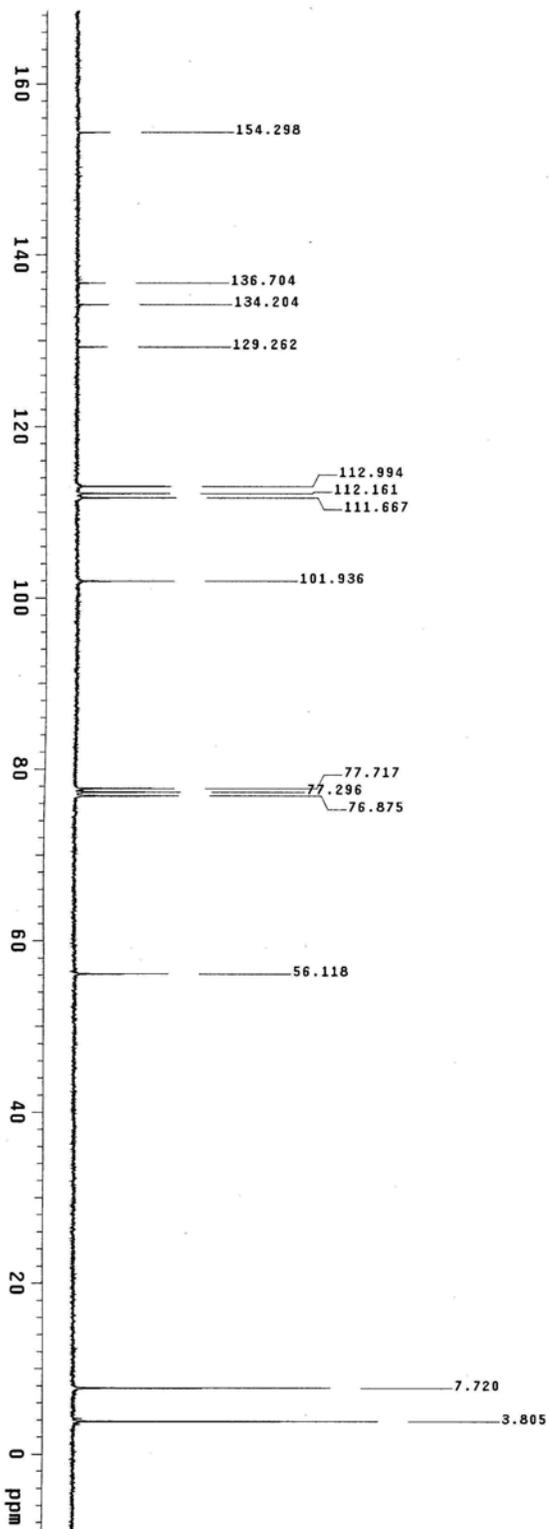
1b-II-220-20
expt std1h
SAMPLE DEC. & VT
date Jan 17 2008 dfreq 300.078
solvent CDCl3 dn H1
file ACQUISITION exp dpwr 30
file ACQUISITION exp dot 30
sfreq 300.078 dmz nnd
st 1.996 dmf C
sw 20000 wtfile PROCESSING 200
sb 5005.0 not used ft
bs not used fn not used
tpwr 58 werr
pw 7.2 wexp
di 2.000 wds
fof 32 wnt
ct 16
alock not used
gain not used
flags
l1 n
l2 n
l3 n
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4

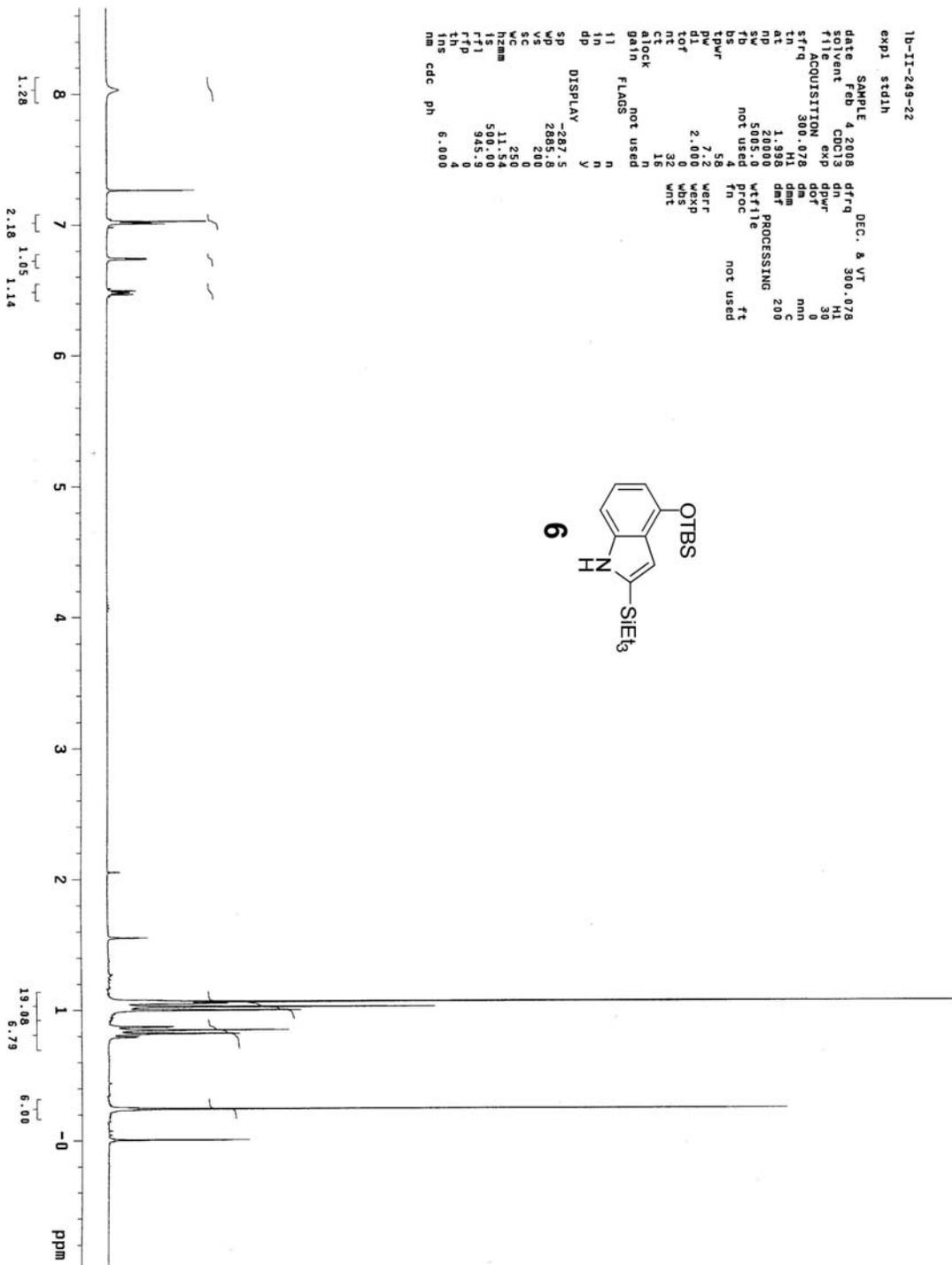
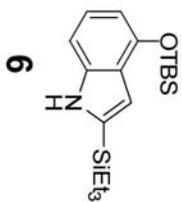




```

1b-II-249-22
expt std1h
SAMPLE DEC. 8 VT
date Feb 4 2008 dfrq 300.078
solvent CDCl3 dn H1
file ACQUISITION exp dpwr 30
sfrq 300.078 ddr 0
at 1.998 ddf mm
np 20000 wifile PROCESSED 200
sw 5005.0 wifile ft
td not used proc not used
ds 54 fn
dpwr 7.2 warr
d1 2.000 wexp
tot 0 wbs
nt 32 wnt
ct 18
atlock not used
gain not used
fl n
fn n
dp DISPLAY y
SP -287.5
VP 2688.5
SC 200
WC 250
hzmm 11.54
fs 500.00
fwd 343.0
th 4
ins nm cdc ph 6.000

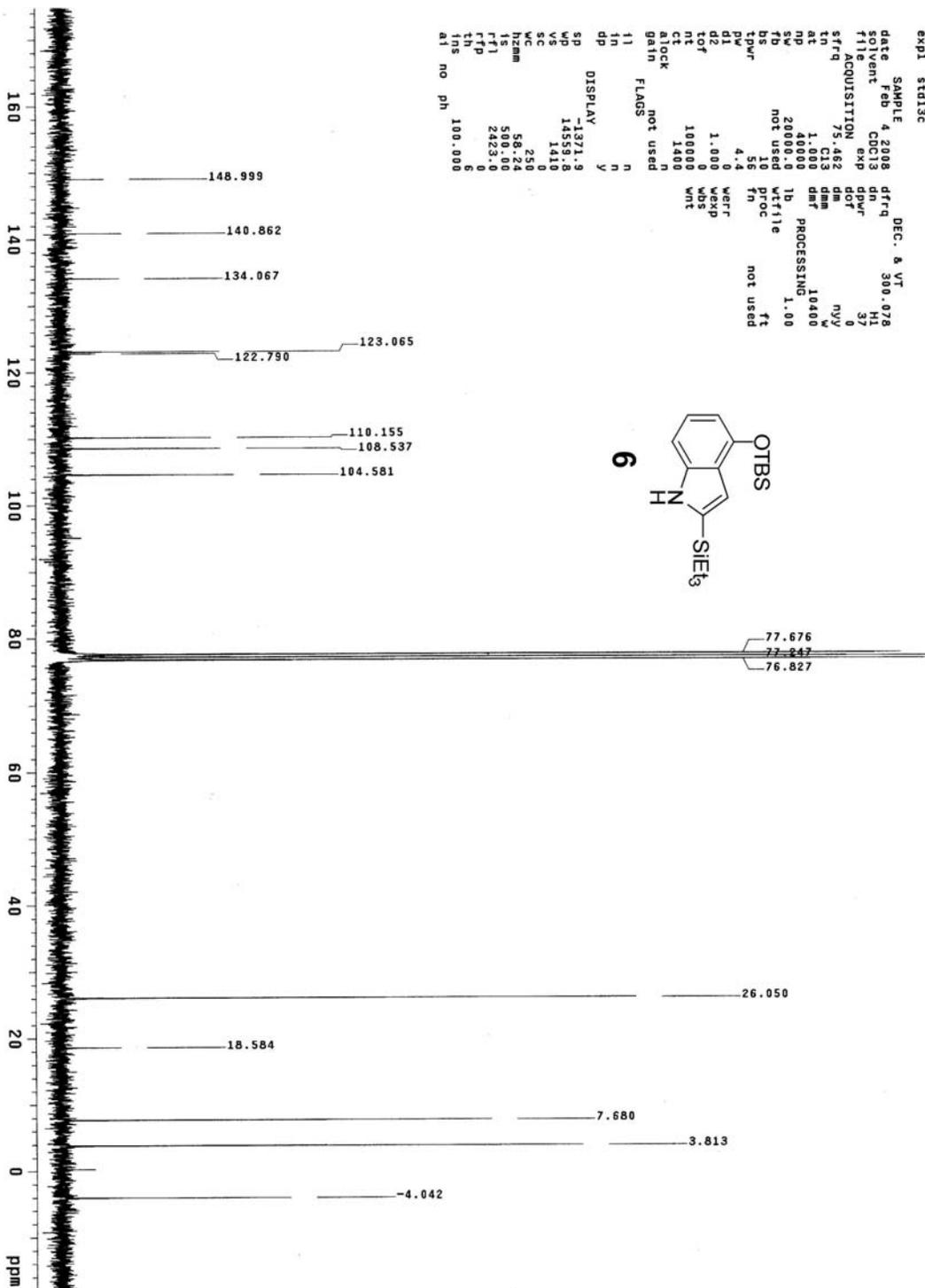
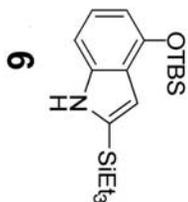
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1b-II-249-22-C

exptl stid13c

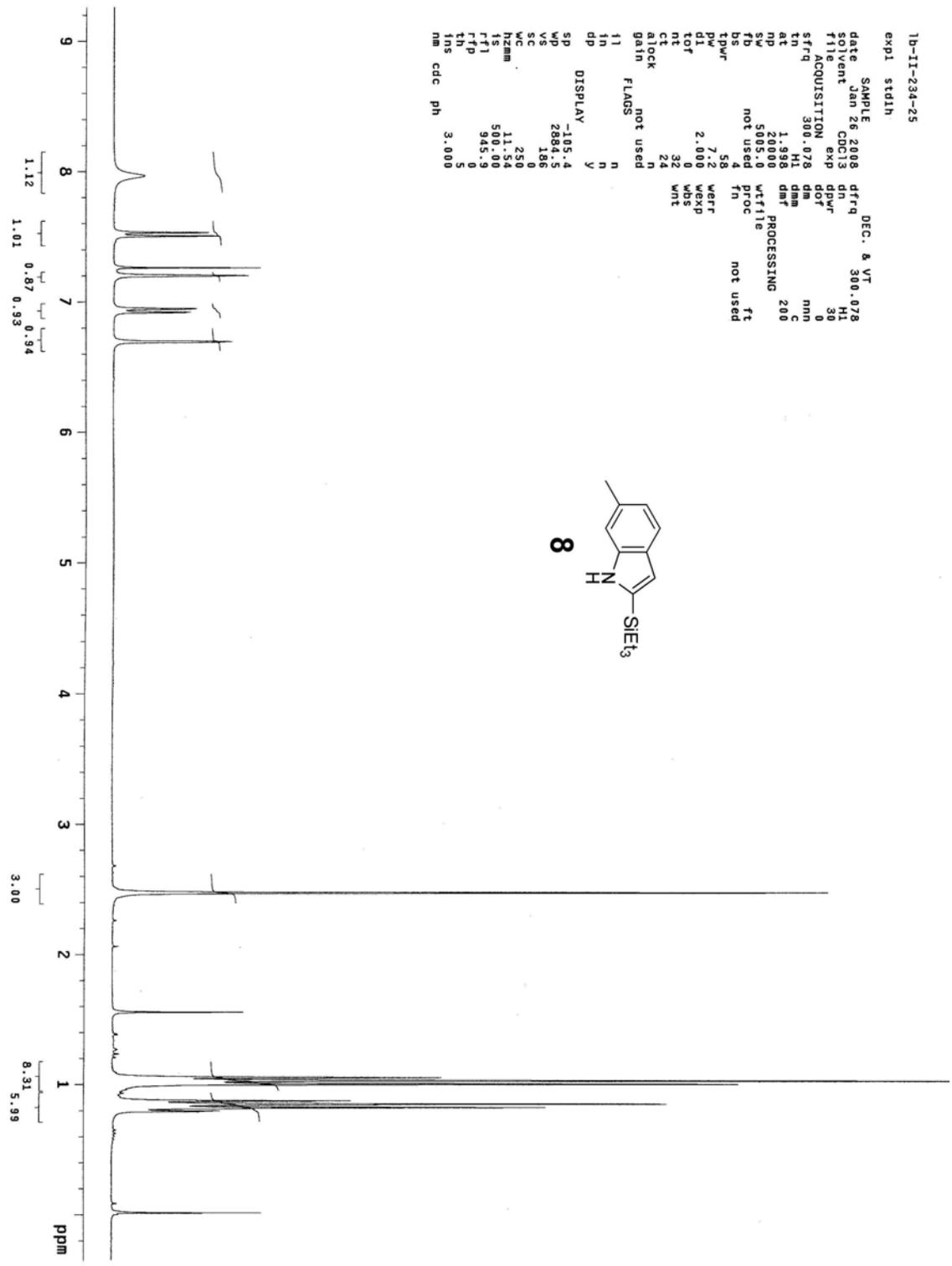
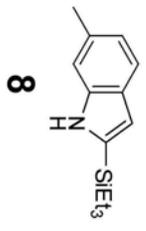
SAMPLE DEC. & VT
date Feb 4 2008 dfrq 300.078
solvent CDCl3 dh dppm 37
file ACQUISITION exp dppm 0
strq 75.462 dm dof 0
tn C13 dam nvy
at 1.000 dar 10400
np 20000 lb PROCESSING 1.00
fb not used wifile
bs 10 proc fi
tpwr 56 fn not used
pv 4.4
dl 0 werr
dc 1.000 wexp
tcf 10000 wos
ct 1400 wnt
alock n
gain not used
flags
11 n
1n n
dp DISPLAY y
SP -1371.9
WP 14559.8
VS 1410
SC 0
WC 250
Hzmm 58.424
rfj 2423.0
rfp 0
th 6
at no ph 100.000



1b-II-234-25
 expl stidih

SAMPLE DEC. & VT
 date Jan 26 2008 dfrq 300.078
 solvent CDCl3 dnm 14
 T1 ACQUISITION exp dcf 0
 sfrq 300.078 dm nmn
 tn H1 dam c
 at 1.998 daf PROCESSING 200
 np 20000 wtitle
 sw 5005.0 wtitle
 fb not used proc ft
 ds 58 tn not used
 pwr 72 werr
 dl 2.000 wexp
 tof 0 wbs
 nt 32 wnt
 ct 24
 alock not used
 gain not used
 fl n
 l1 n
 l1 n
 dp y

DISPLAY
 sp -105.4
 wp 2884.5
 vs 186
 sc 0
 wc 250
 hznm 11.34
 f2 500.00
 f3 385.0
 rfp 5
 th 5
 ins 3.000
 nm cdc ph

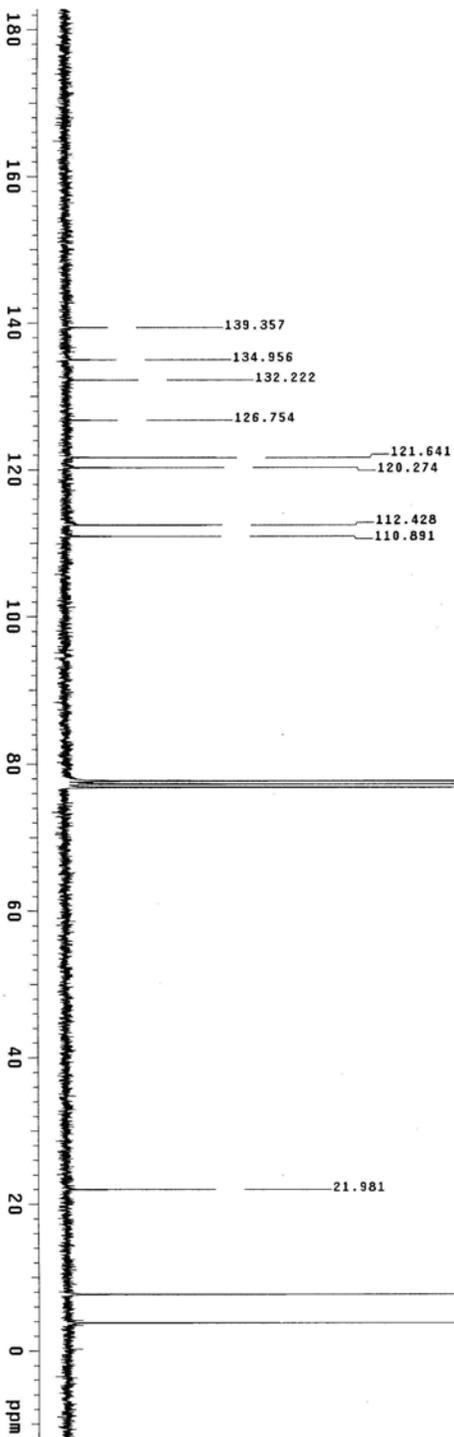
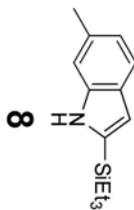


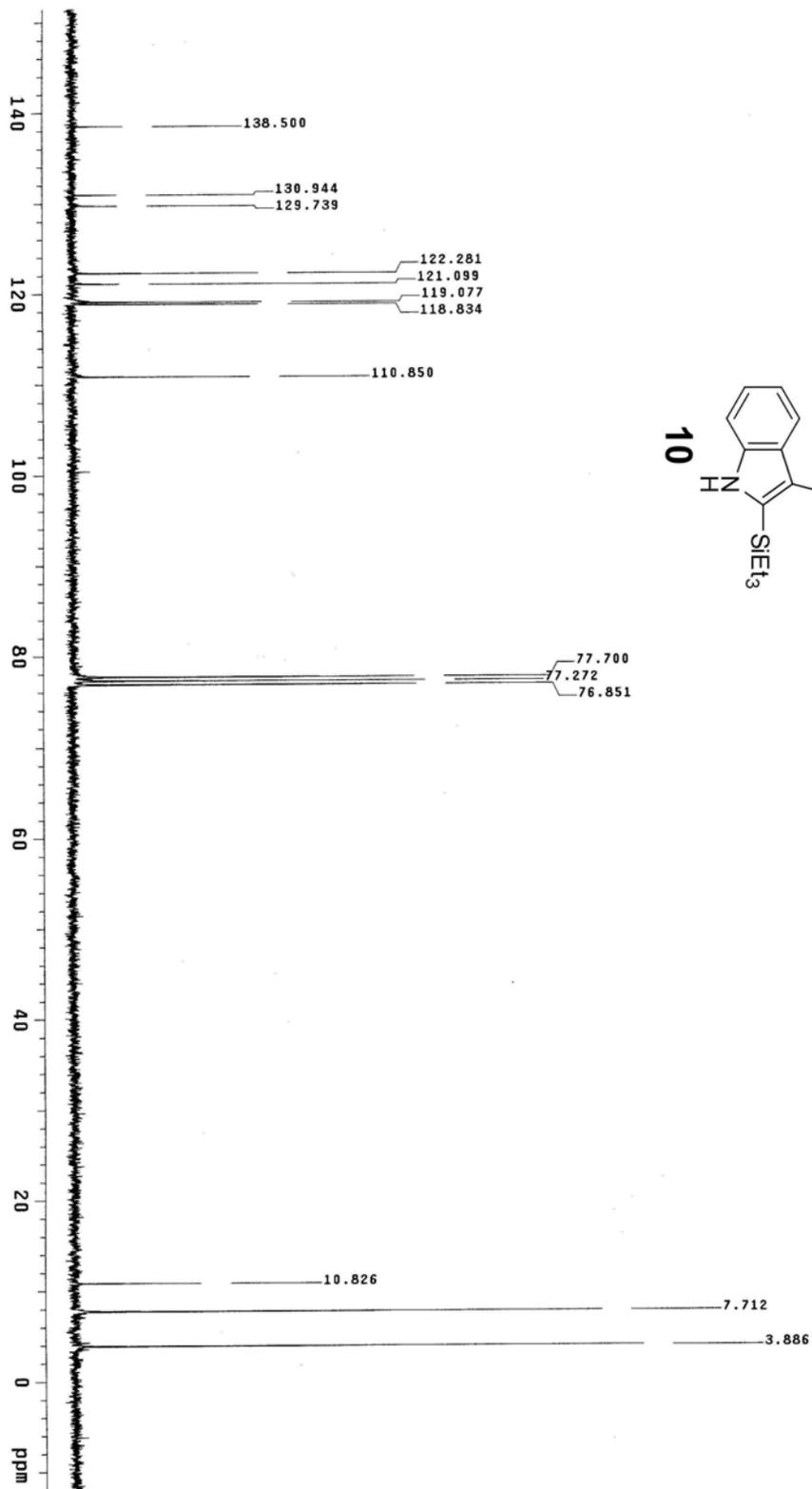
1b-II-234-25-C

exptl std13c

SAMPLE DEC. & VT
date Jan 26 2008 dfrq 300.078
client C013 d 37
file ACQUISITION exp dpwr 0
ACQUISITION exp dof 0
sfrq 75.482 dm nvy
tn C13 dm 10400
at 1.000 dmf W
sp 20000 1b PROCESSING 1.00
fb not used wfile ft
bs 10 proc not used
tpwr 56 fn
pw 4.4 weff
d1 1.000 d2f weff
d2f 1.000 weff
nt 100000 MDS
ct 910 MFL

gain not used
alock n
flags not used
l1 n
l2 n
l3 n
dp y
DISPLAY -925.1
sp 14717.2
wp 851
vs 851
sc 250
mc 250
hmm 58.29
ls 500.00
rfl 2423.0
th 0
tms 100.000
at no ph

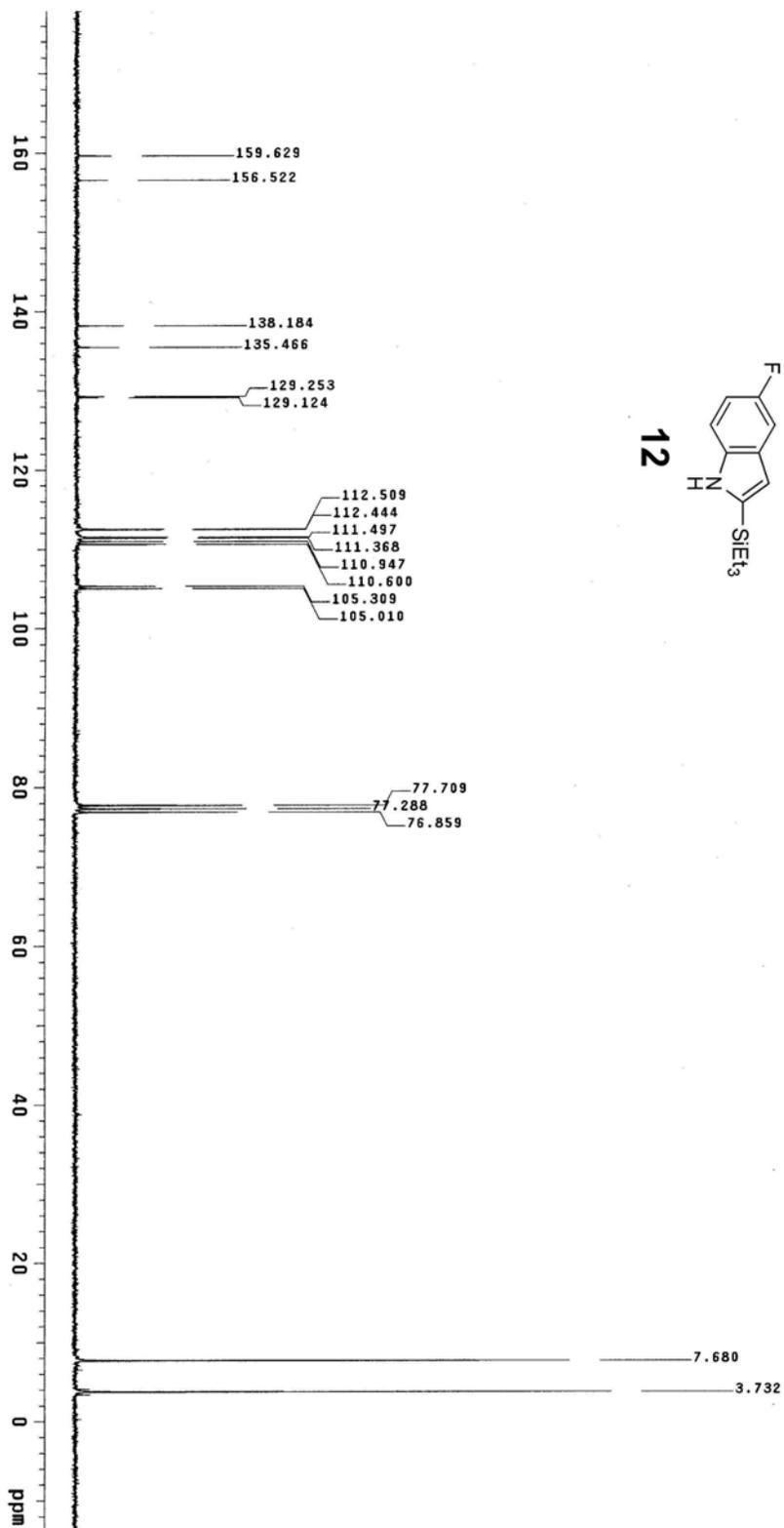




1b-II-215-26

expt stdih

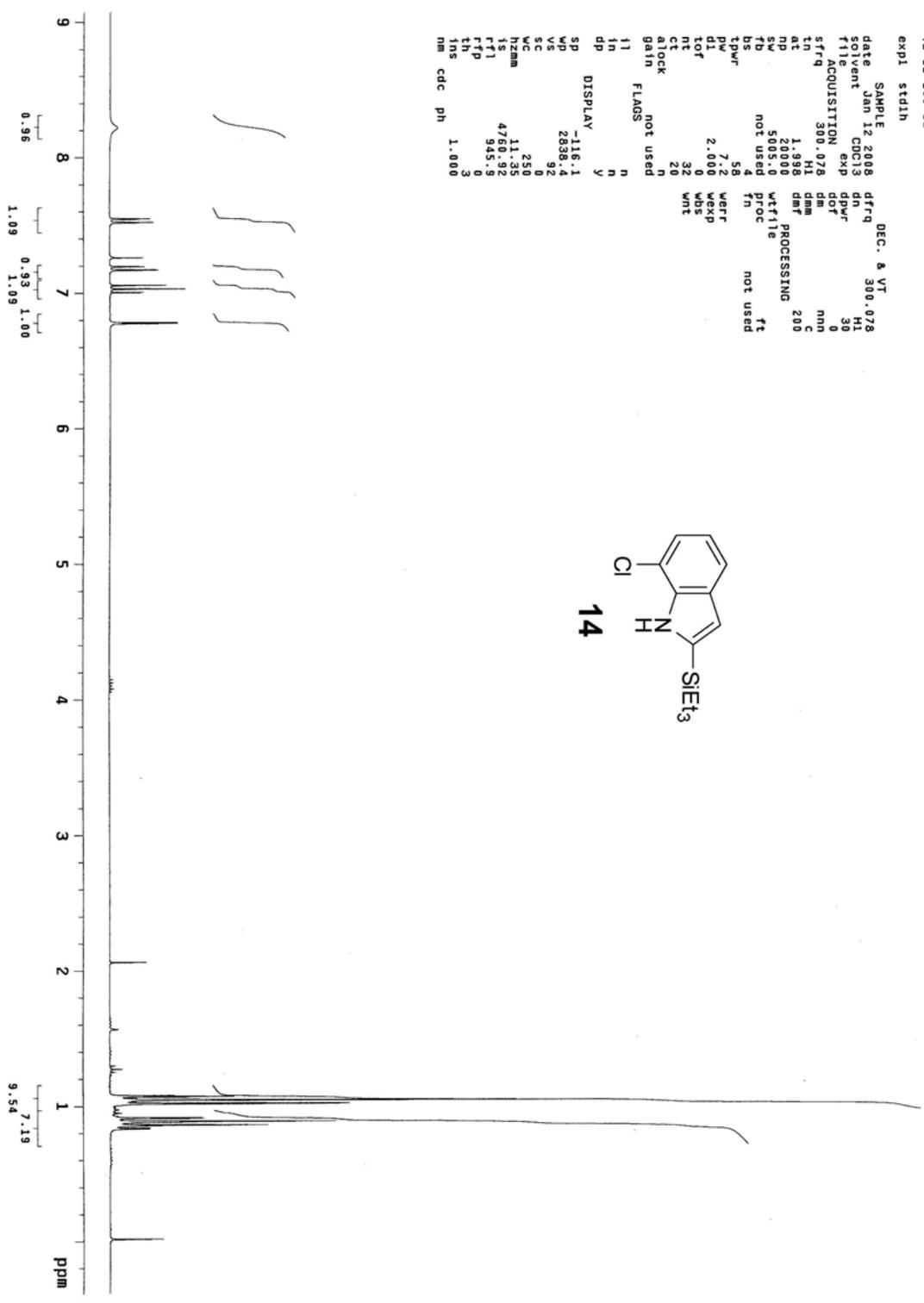
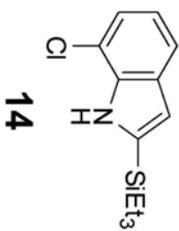
SAMPLE DEC. & VT
 date Jan 15 2008 dtrq 300.078
 solvent CDCl3 dh H1
 file exp dpwr 30
 ACQUISITION dof 0
 sfrq 300.078 H1 dm
 at 1.998 dmf
 np 20000 wifile PROCESSING 200
 sw 5005.0 wifile ft
 fb not used proc not used
 bs 4 fn
 tpwr 58 werr
 pw 7.12 wexp
 dl 2.000 wexp
 nr 32 wnt
 ct 16
 atlock not used
 gain not used
 flags not used
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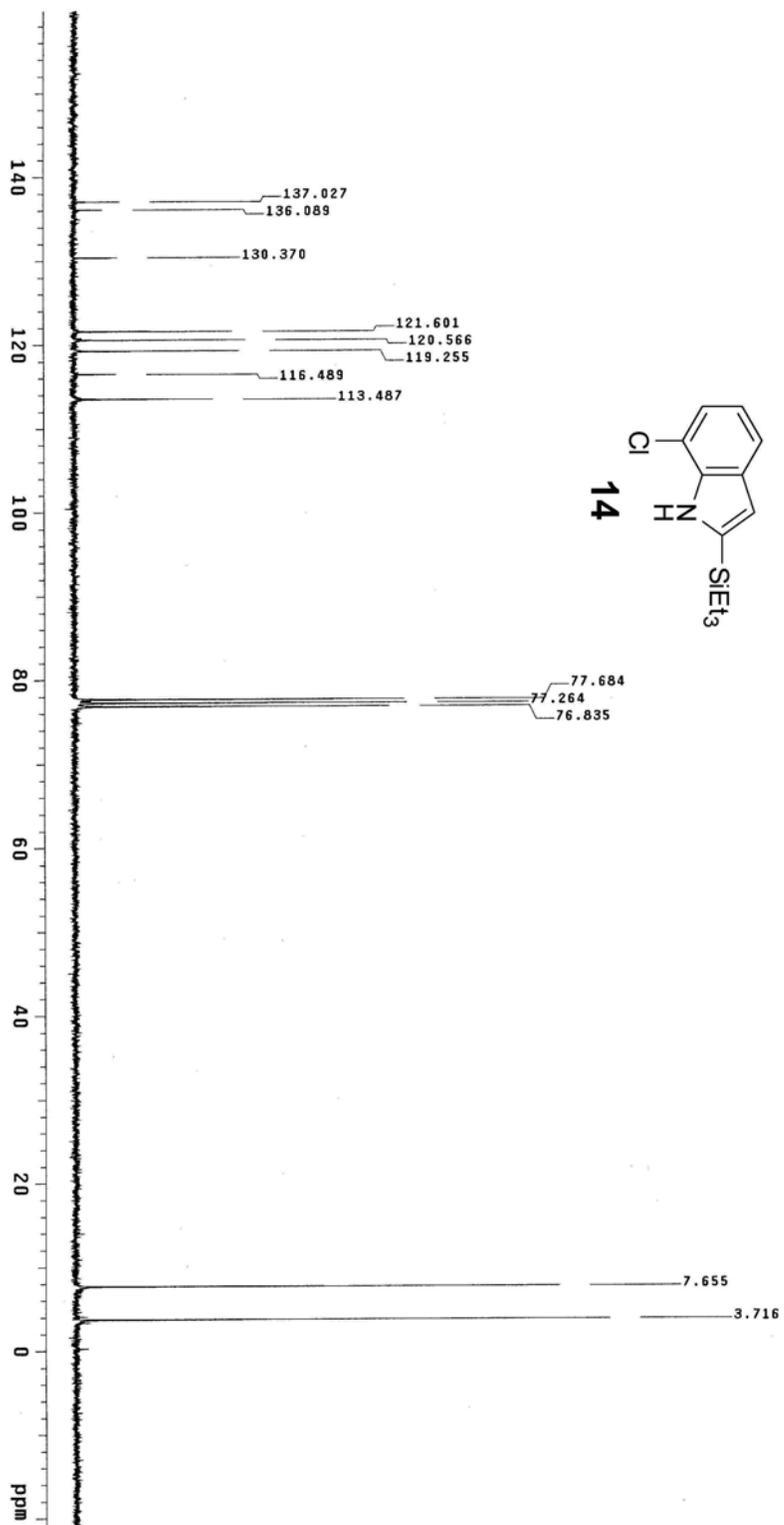


1b-II-209-22

expt stdinh

date	Jan 12 2008	DEC. & VI	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION	exp	dot	0
sfreq	300.078	dmf	nmn
ti	H1	dmf	C
at	1.998	PROCESsing	200
np	20000	wtfile	ft
sw	5005.0	proc	not used
fb	not used	fn	not used
bs	4		
tpwr	58	usrf	
pw	7.00	MEXP	
nt	2.00	WBS	
ct	32	Wnt	
atlock	20		
gain	not used		
FLAGS	not used		
l1	n		
l2	n		
l3	n		
l4	n		
l5	n		
l6	n		
l7	n		
l8	n		
l9	n		
l10	n		
l11	n		
l12	n		
l13	n		
l14	n		
l15	n		
l16	n		
l17	n		
l18	n		
l19	n		
l20	n		
l21	n		
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l27	n		
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l32	n		
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l37	n		
l38	n		
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l89	n		
l90	n		
l91	n		
l92	n		
l93	n		
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l95	n		
l96	n		
l97	n		
l98	n		
l99	n		
l100	n		

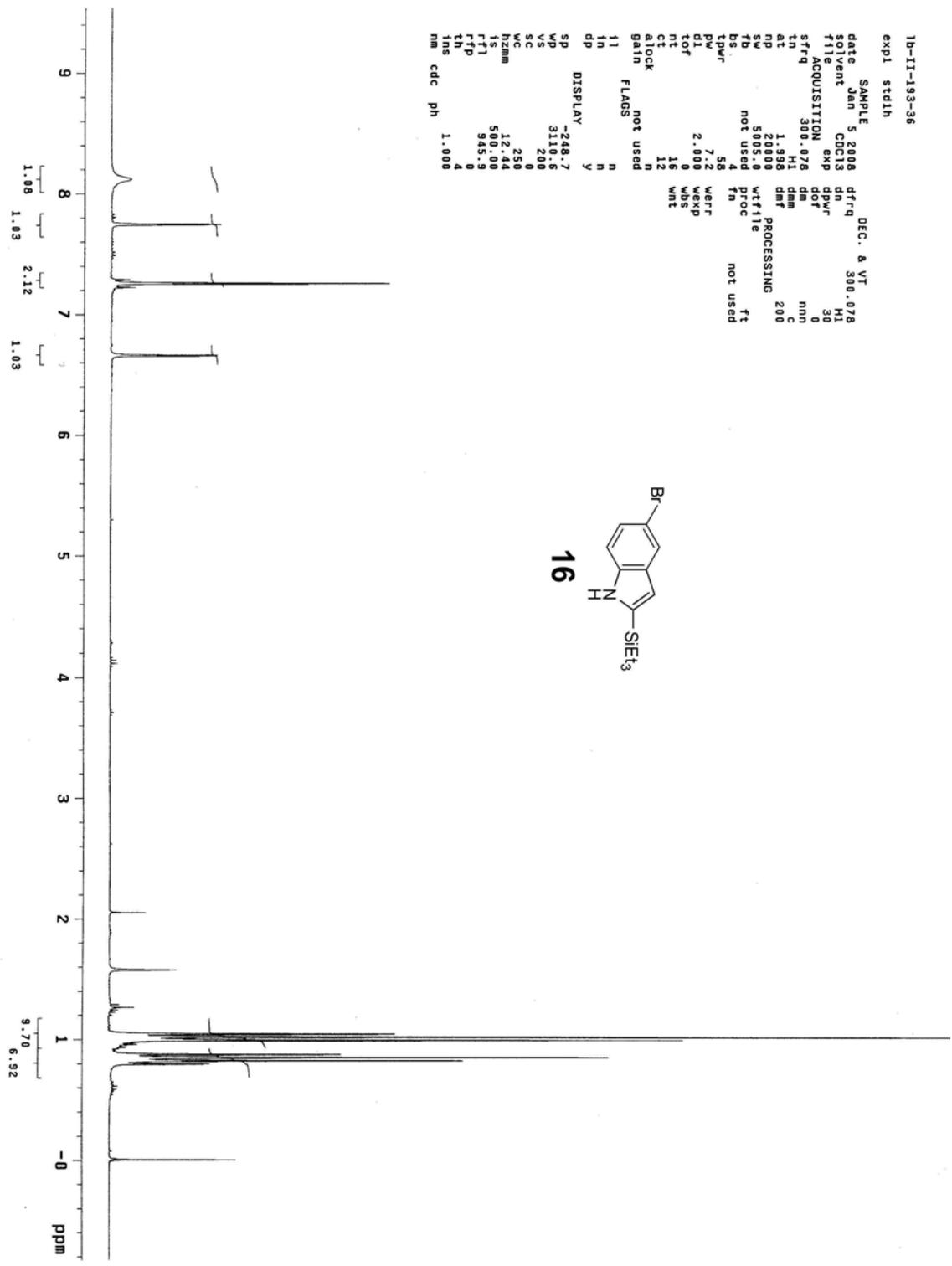
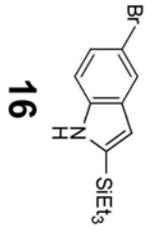


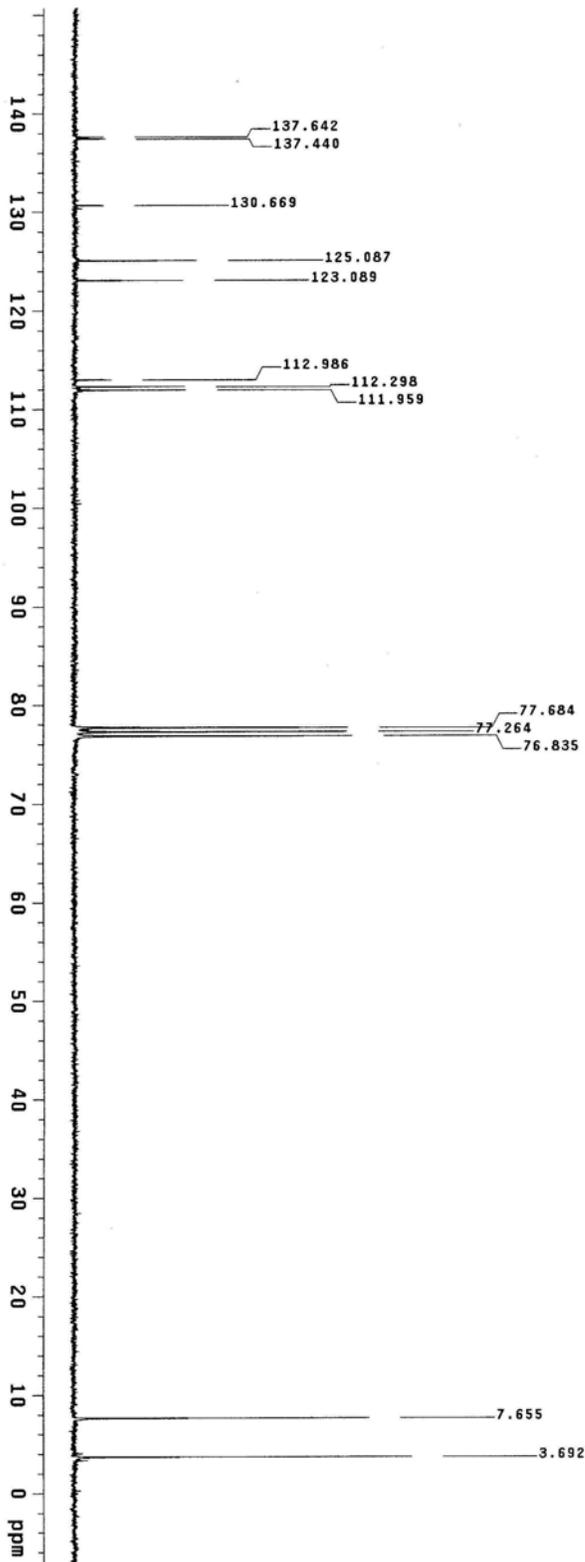
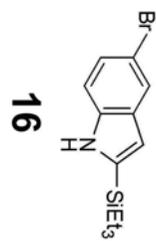


```

1b-II-193-36
expt stdih
SAMPLE DEC. & VT
date Jan 5 2008 dfrq 300.078
solvent CDCl3 dn H1
FILE ACQUISITION exp dpwr 30
sfrq 300.078 dot 0
tn H1 dnm n/c
at 1.998 dmf PROCESSING 200
np 2000 wtf1le ft
sw 5005.0 not used proc fn
bs not used 4
tpwr 56 werr not used
pw 7.72 wexp
nt 2.000 wps
ct 16 wnt
atlock 12
gain not used n
flags not used
11 n
1n n
dp DISPLAY y
SP -248.7
WP 3110.6
VS 200
SC 0
WC 250
hzkm 12.44
IS 500.00
FT1 945.9
TFD 4
ins 1.000
nm cdc ph

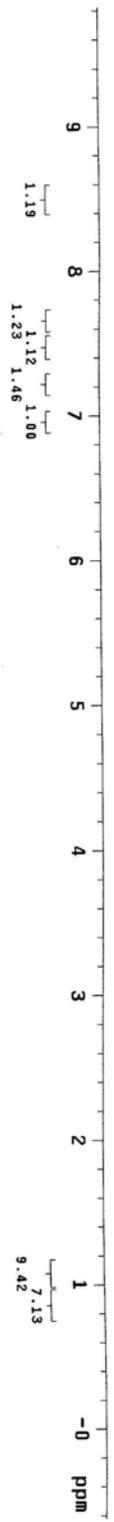
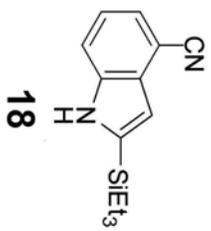
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1b-II-240-31
expt st01h

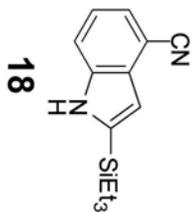
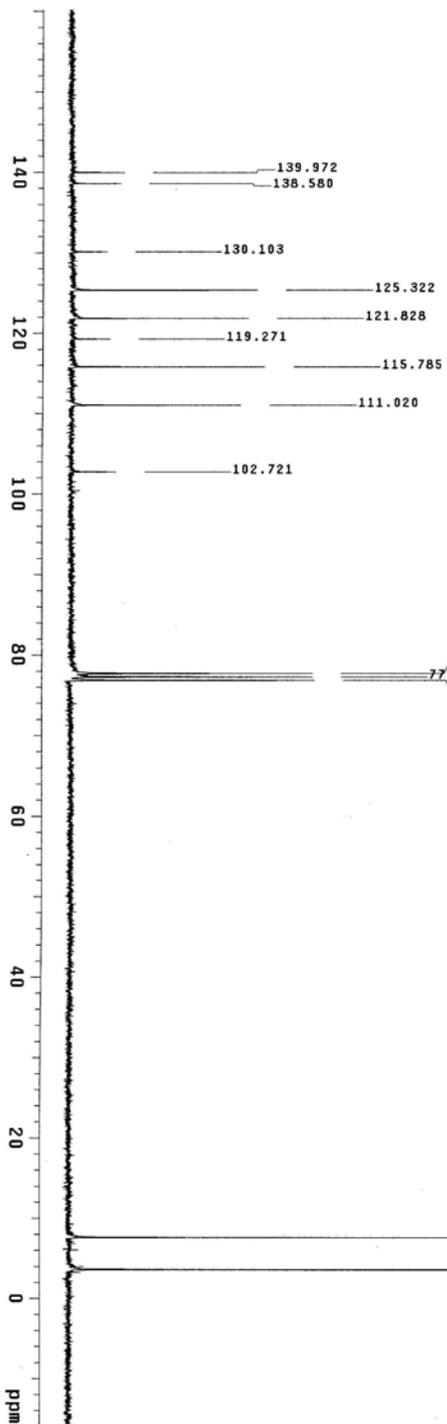
SAMPLE DEC. & VT
date Jan 31 2008 dfrq 300.078
solvent CDCl3 dn HI
file exp dpwr 30
ACQUISITION dof 0
sfrq 300.078 dm nmh
tn HI dm
at 1.898 dmf PROCESSING 200
nd 2.890 wfile
sp 5005.0 wfile
fb not used fn
bs 4 not used
tdwr 58
pw 7.2 weff
d1 2.000 wexp
tof 0 wds
nt 32 wnt
ct 24
atlock not used
gain
fls
f1 n
in n
dp y
DISPLAY
sp -187.0
wp 3134.1
vs 114
sc 250
hsc 12.54
h2mm 500.00
f1 945.9
r1p 0
th 6
nm cdc ph 1.000



1b-II-240-31-C

expt std13c

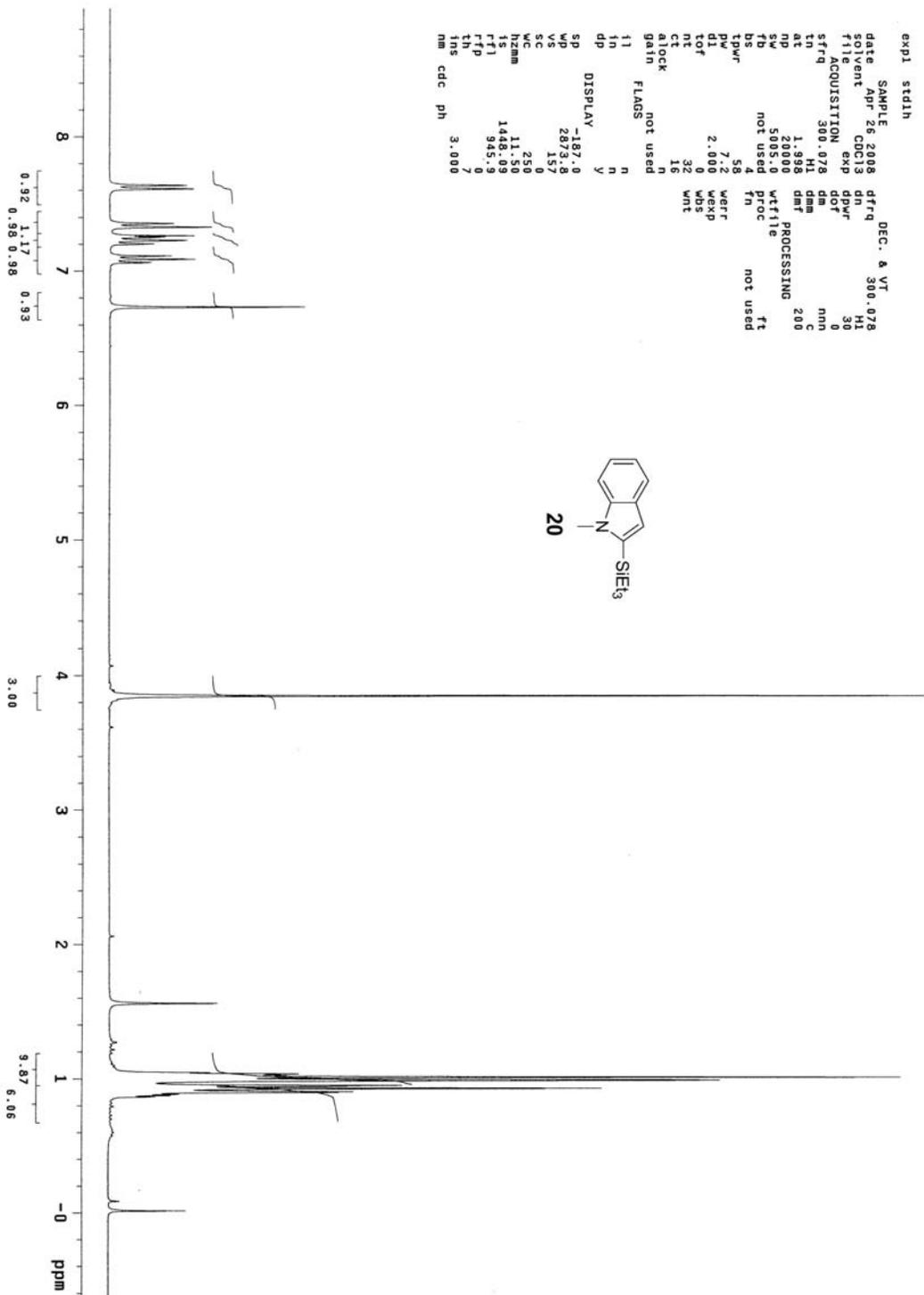
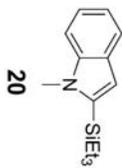
SAMPLE DEC. & VT
date Jan 51 2008 dfrq 300.078
solvent CDCl3 dn H1
file CDC13 exp dpvr 37
ACQUISITION dot 0
sfrq 75.462 dm nvy
an 1.013 dm 10400
ap 1.010 dm 10400
sw 20000.0 lb wtfile
fb not used proc ft
ds 10 not used
tpwr 4.8 fn
d1 0 werr
d2 1.000 wexp
tof 0 wbs
nt 10000 wnt
ct 1370
atlock not used
gain not used
flags
i1 n
in n
dp y
DISPLAY
sp 1344.7
up 13824.4
vs 371
sc 0
wc 250
hzm 53.30
fs 500.00
ft 2423.0
th 5
ins 100.000
at no ph



1b-III-65-31

exptl std1h

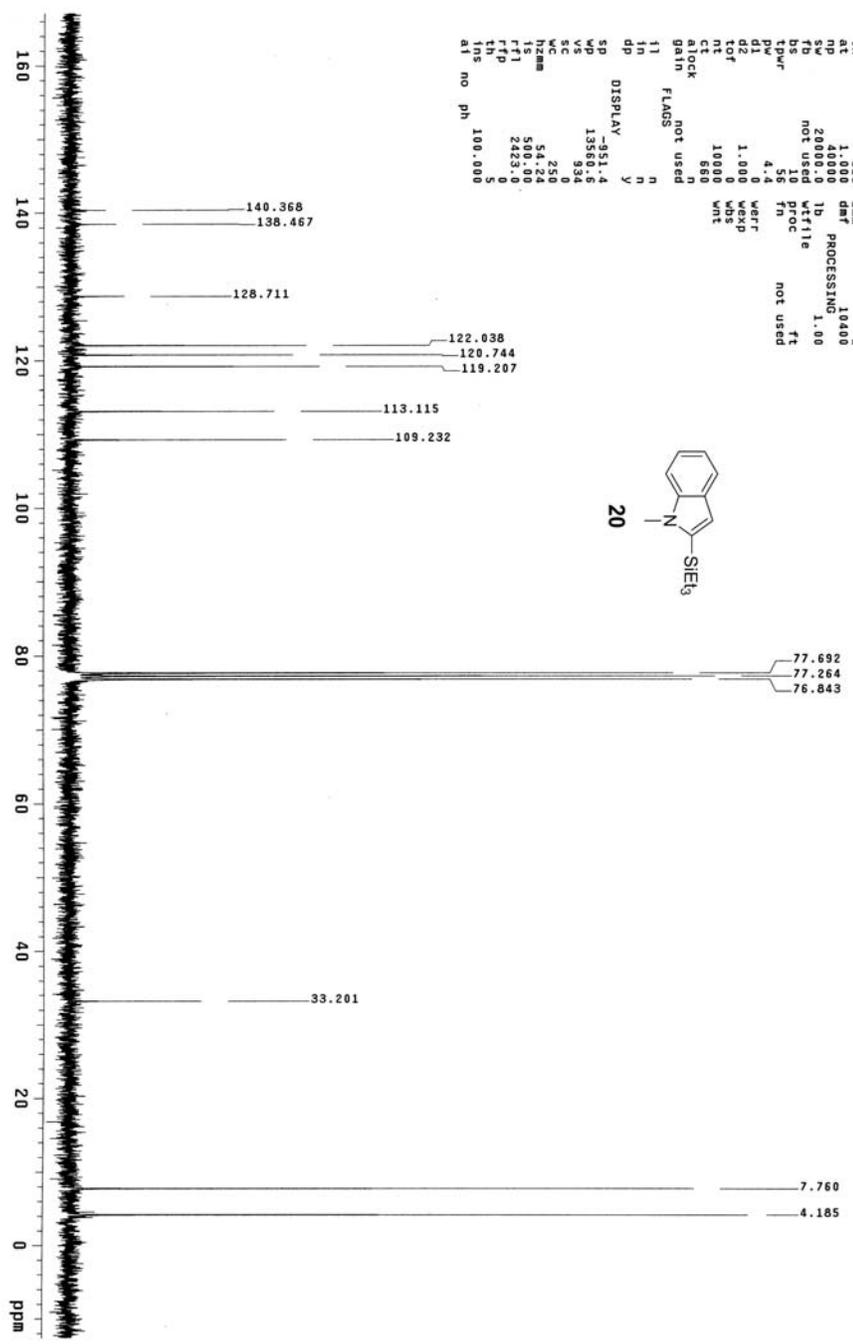
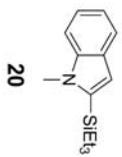
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SAMPLE DEC. & VT
date Apr 26 2008 dfrq 300.078
solvent CDCl3 dn HI
file C0013 exp dpr 30
ACQUISITION exp dot 0
sfrq 300.078 dm nnn
tn HI dmm
at 1.998 dmt PROCESSING 200
np 5005.0 wffile
fb not used proc fn
bs 4
tpwr 58
pw 7.2 weff
dl 2.000 wexp
tor 0 wds
ct 32 wnt
atlock n
gain not used
FLAGS
11 n
1n n
dp y
DISPLAY -187.0
SP 2873.8
VS 157
SC 0
WC 250
hzmm 11.50
rfi 144.50
rfp 945.9
th 0
nm cdc ph 3.000
```



```

1b-III-65-31-C
expi std13c
SAMPLE DEC. 8 VT
date 26 2008 dfrq 300.078
solvent CDCl3 dn 37
file C0013 exp dpr 0
ACQUISITION dot
tfrq 75.452 dm nny
at 1.000 dmf 10000
np 40000 lb file 1.00
sw 20000.0 pfile
ds not used pproc not used
tpwr 56 fn
pv 4.4 werr
d1 1.000 wexp
tcf 100000 wnt
nt 10000
ct 660
alock not used
gain 660
l1 n
l2 n
l3 n
l4 n
l5 n
l6 n
l7 n
l8 n
l9 n
l10 n
l11 n
l12 n
l13 n
l14 n
l15 n
l16 n
l17 n
l18 n
l19 n
l20 n
l21 n
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l23 n
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l25 n
l26 n
l27 n
l28 n
l29 n
l30 n
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l32 n
l33 n
l34 n
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l99 n
l100 n
at no ph 100.000

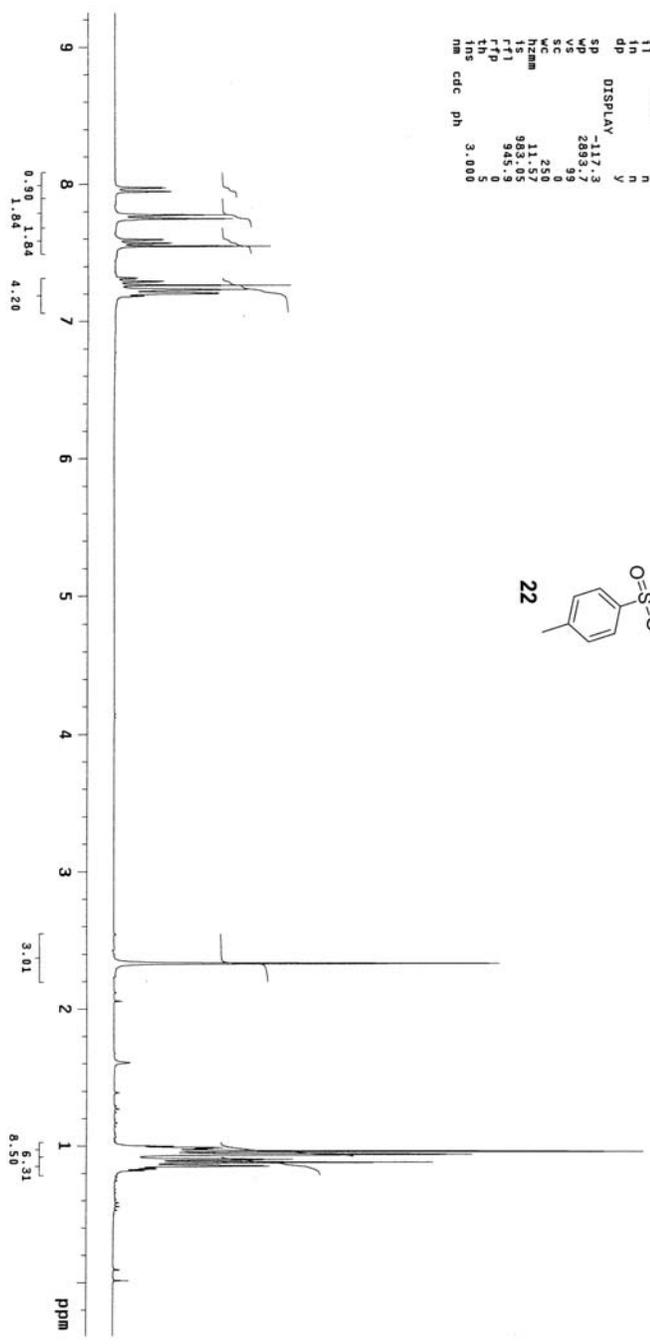
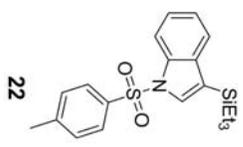
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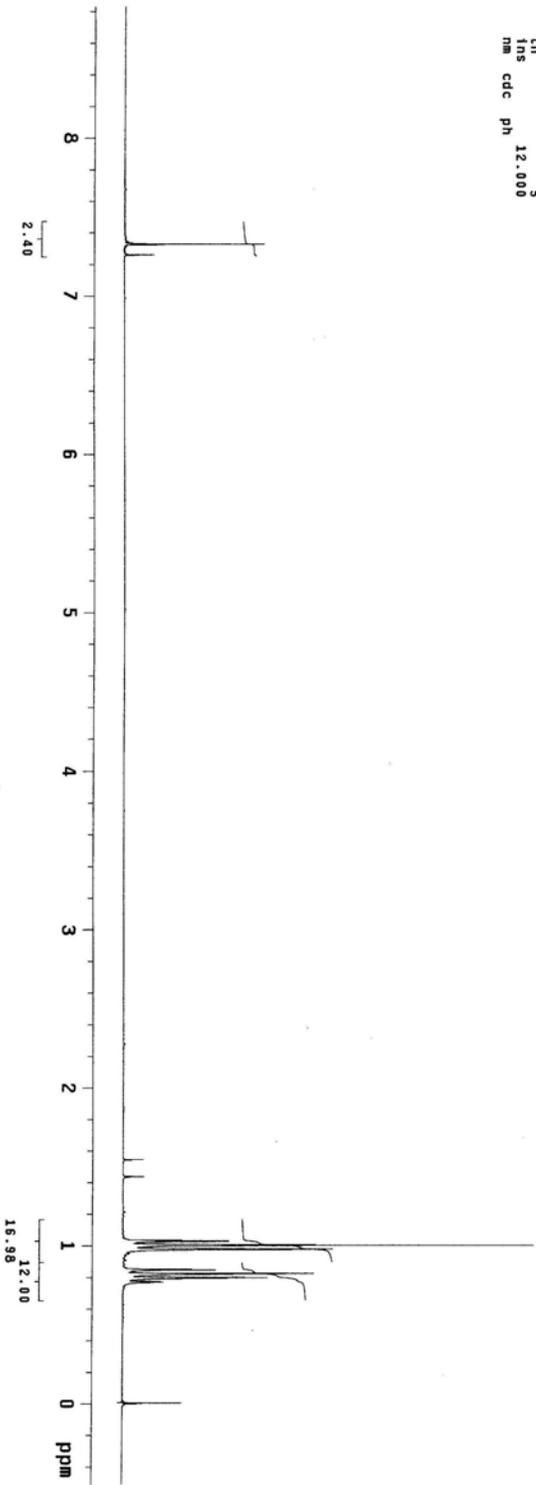
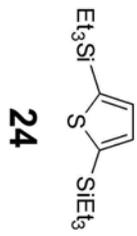
1b-III-81-31
expt stdIn
SAMPLE
date May 5 2008 dfrq DEC. & VT 300.078
solent CDCl3 dm 31
file C03 DM 31
ACQUISITION exp DM 0
sfrq 300.078 dm 0
in 1.914 dm 0
nd 20000 dm 0
sw 5005.0 wft1le PROCESSING 200
fb not used proc ft
ds 58 not used tn
pwr 7.2 warr
di 2.000 wexp
to 0
nt 20 wts
rt 24
atlock not used n
gain not used n
flags not used n
in n
dp y
DISPLAY
sp -112.3
vd 2839.92
sc 0
wc 250
hzmm 81.52
rf1 945.9
rfp 0
th 5
ms cdc ph 3.000

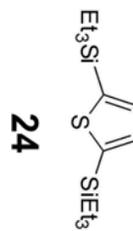
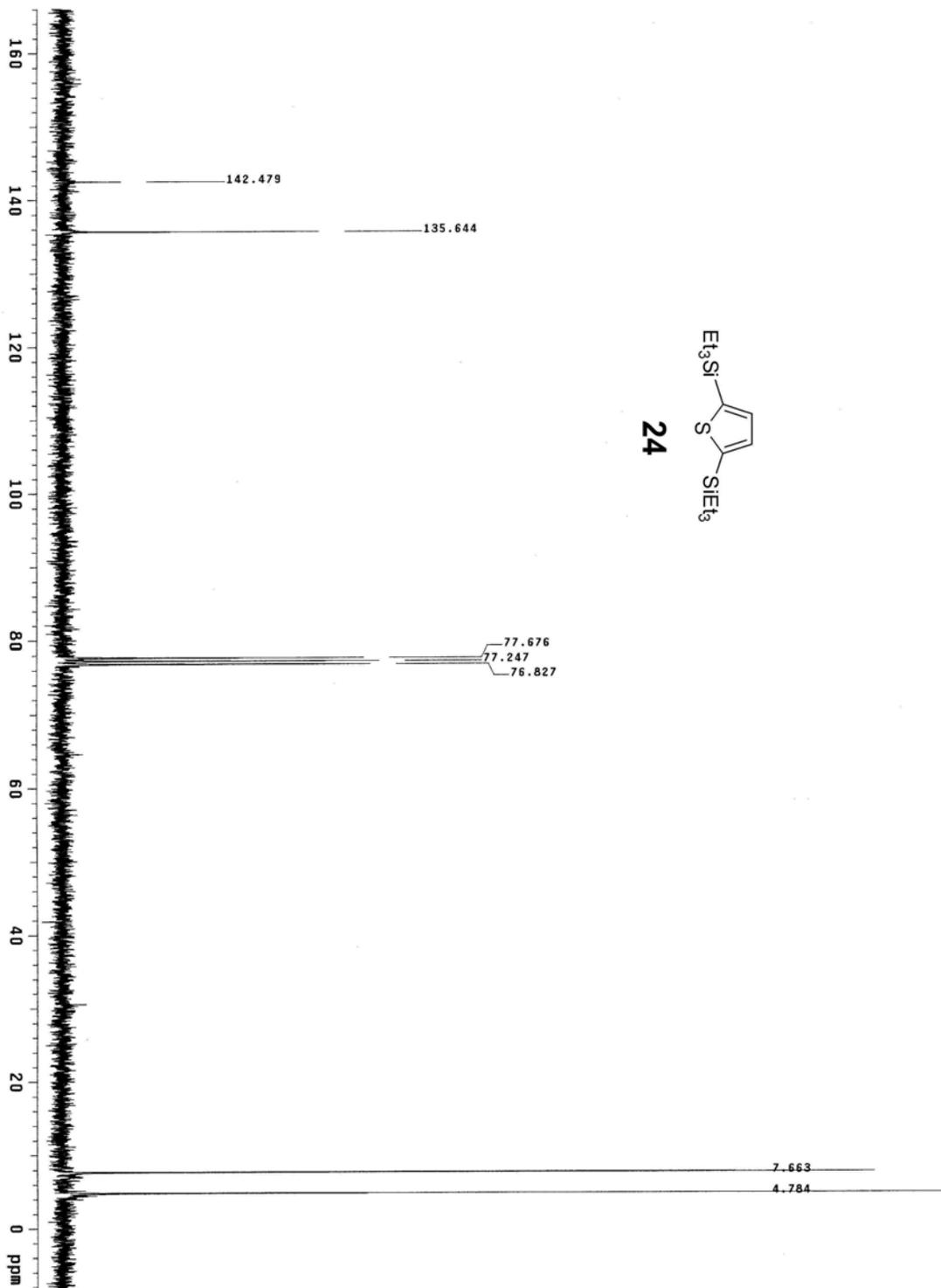
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1b-11-14-30

expt stidh
SAMPLE 7 2008 DEC. 8 VT
date Jan 7 CDC13 dn 300.078 H1
solvent CDCl3 exp ddwr 30 H1
file ACQUISITION dof 0 nnn
sfrq 300.078 dm dam nnn
in H1 dm dam 200 C
at 1.998 dmf PROCESSING
np 5.000 wfile
fb not used proc ft
bs 4 fn not used
tpwr 58
pw 7.2 werr
dl 2.000 wexp
tof 0 wds
nt 32 wnt
z 24
slock
gain not used
flags
l1 n
l2 n
l3 n
l4 n
l5 n
l6 n
l7 n
l8 n
l9 n
l10 n
l11 n
l12 n
l13 n
l14 n
l15 n
nm cdc ph 12.000

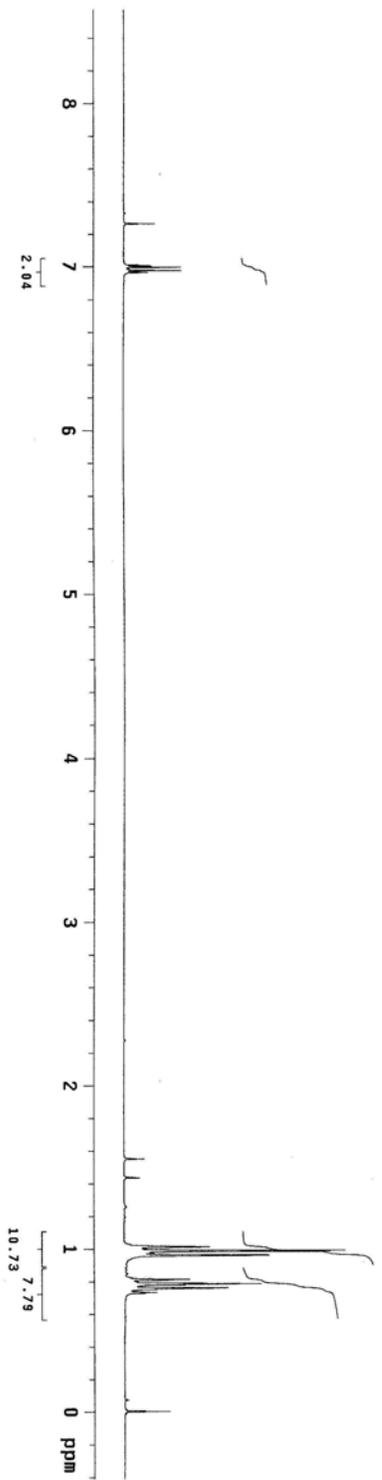
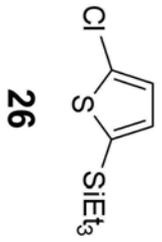




1b-II-227-26

expt stdIn

date	Jan 22 2008	dfreq	300.078	DEC. & VT	300.078
solvent	CDCl3	dn	H1		
file	exp	dpwr	30		
sfreq	300.078	dm	nm		
at	1.998	H1 dmf	200		
np	20000	wtfile	PROCESSING		
sw	5005.0	proc	not used		
fb	not used	fn			
bs	54				
dpwr	7.2	werr			
dl	2.000	wexp			
tot	0	wbs			
nt	32	wnt			
ct	32				
gatin	not used				
lock	not used				
flags	not used				
in	n				
in	n				
dp	y				
DISPLAY					
sp	-123.8				
wp	2696.3				
vs	37				
sc	250				
ns	0				
hz	10.73				
ts	500.00				
rfl	945.9				
rfd	0				
th	0				
ins	6				
nm	cdc	ph	2.000		

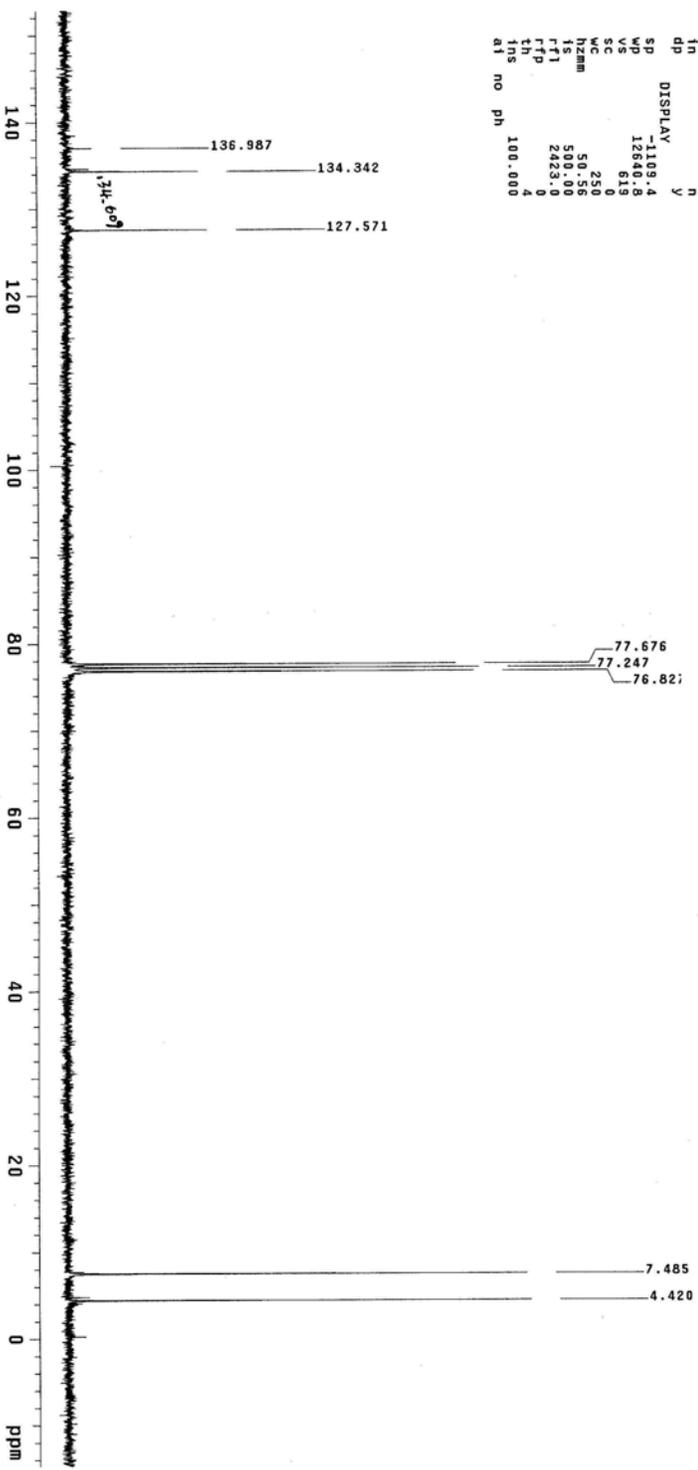
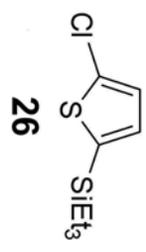


1b-II-227-26-c

exp1 std13c

SAMPLE DEC. & VI
date Jan 22 2008 dfrq 300.078
solvent CDCl3 dn H1
file CXC13 exp 37
ACQUISITION exp 37
sfrq 75.482 dn nvy
tn 75.482 dmf 10400
at 1.000 dmf
sw 40000 lb PROCESSING 1.00
fd 20000.0 wfile
not used wfile ft
ds 5.0 Proc not used
bpuw 4.4
d1 1.000 werr
d2 1.000 wexp
tof 0 wbs
nt 10000 wnt
ct 800
alock not used
gain not used
ll flags n
in n
dp y

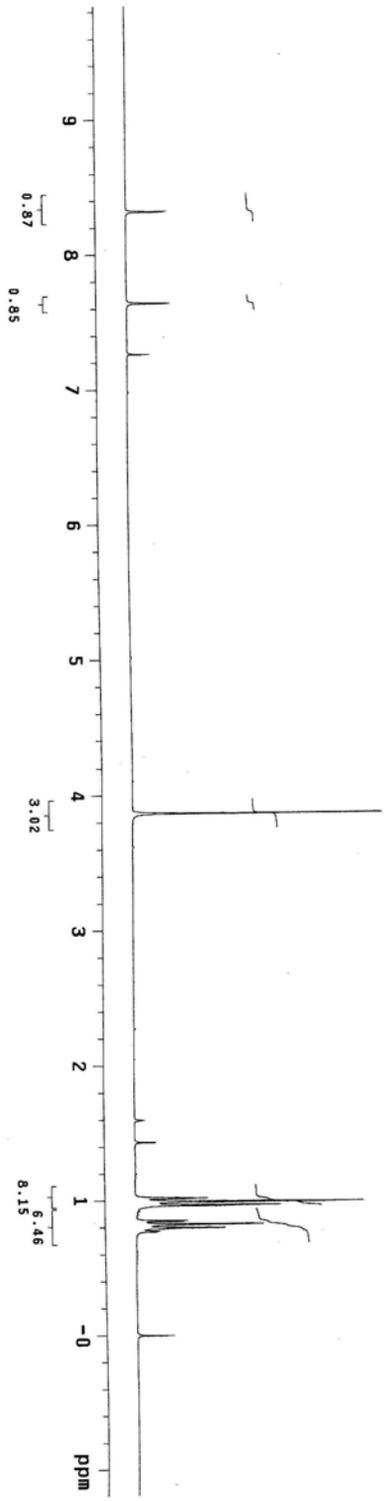
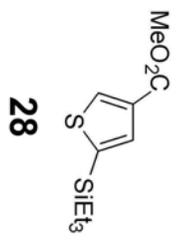
DISPLAY
sp -1199.4
wp 12690.8
sc 81.0
wc 250
hzmm 50.56
fs 500.00
fft 2423.0
ft 0
th 0
ns no
at ph 100.000

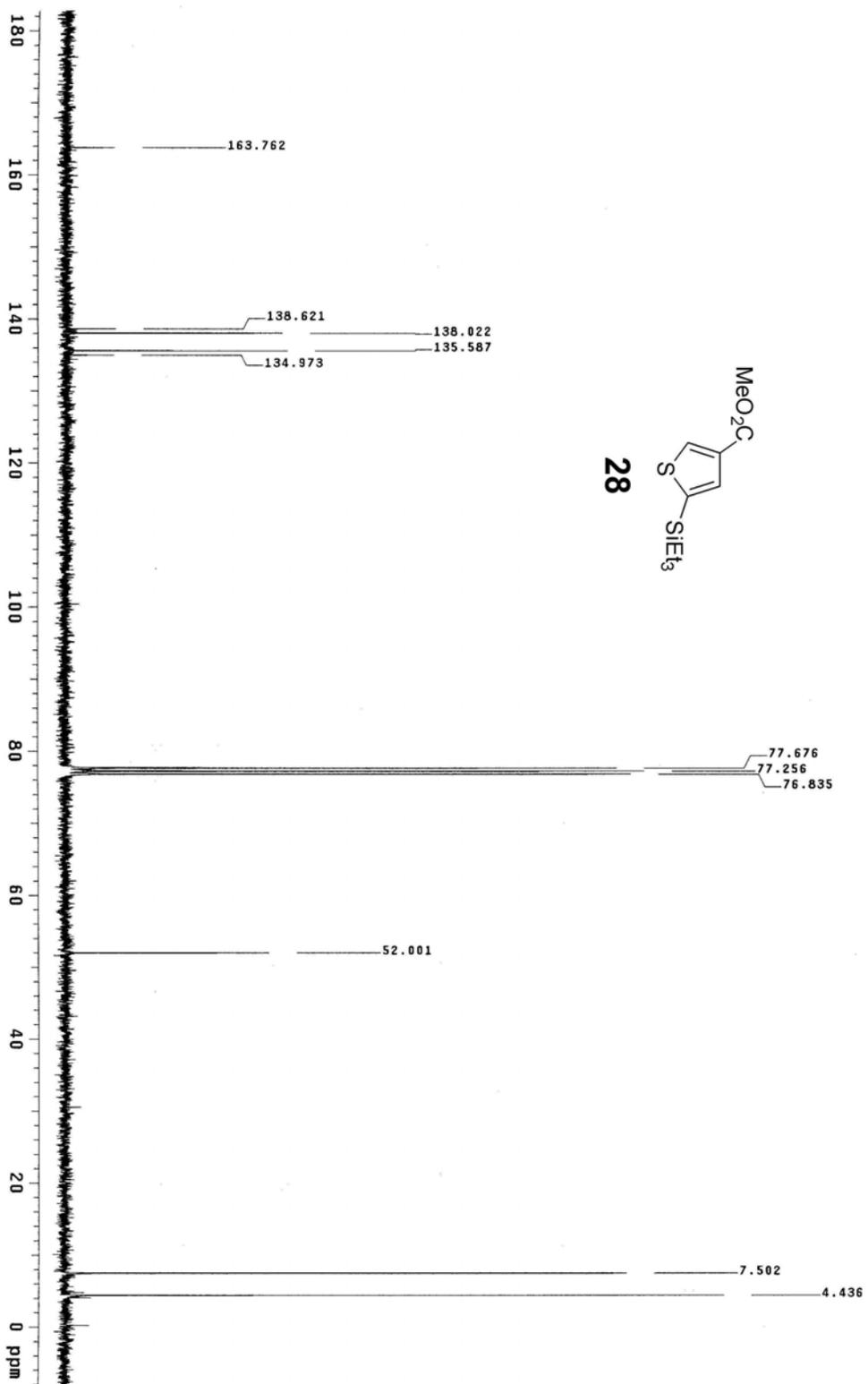


```

1b-11-224-33
expt1 std1h
SAMPLE Jan 19 2008 dfreq DEC. & VT 300.078
date Jan 19 2008 dn CDC13
solvent CDC13 exp dppv H1
file ACQUISITION dot ddt 30
sfrq 300.078 dmz nmn
sfrq 1.996 dmz dmf 200
at 20000 wtrfile PROCESsing ft
sw 5005.0 not used proc fn not used
fb not used
bs 4
tpwr 58 WERR
pw 7.2 WERR
pd 2.000 WERR
qd 0 WBS
nr 32 WBS
ct 24 WNT
ct alock n
gain not used
flags not used
11 n
1n n
dn y
DISPLAY -358.4
SP 3311.6
VD 41
VS 0
WC 250
SC 13.25
hzemm 580.00
IS 945.9
FTF 8
fns 3.000
nm cdc ph

```

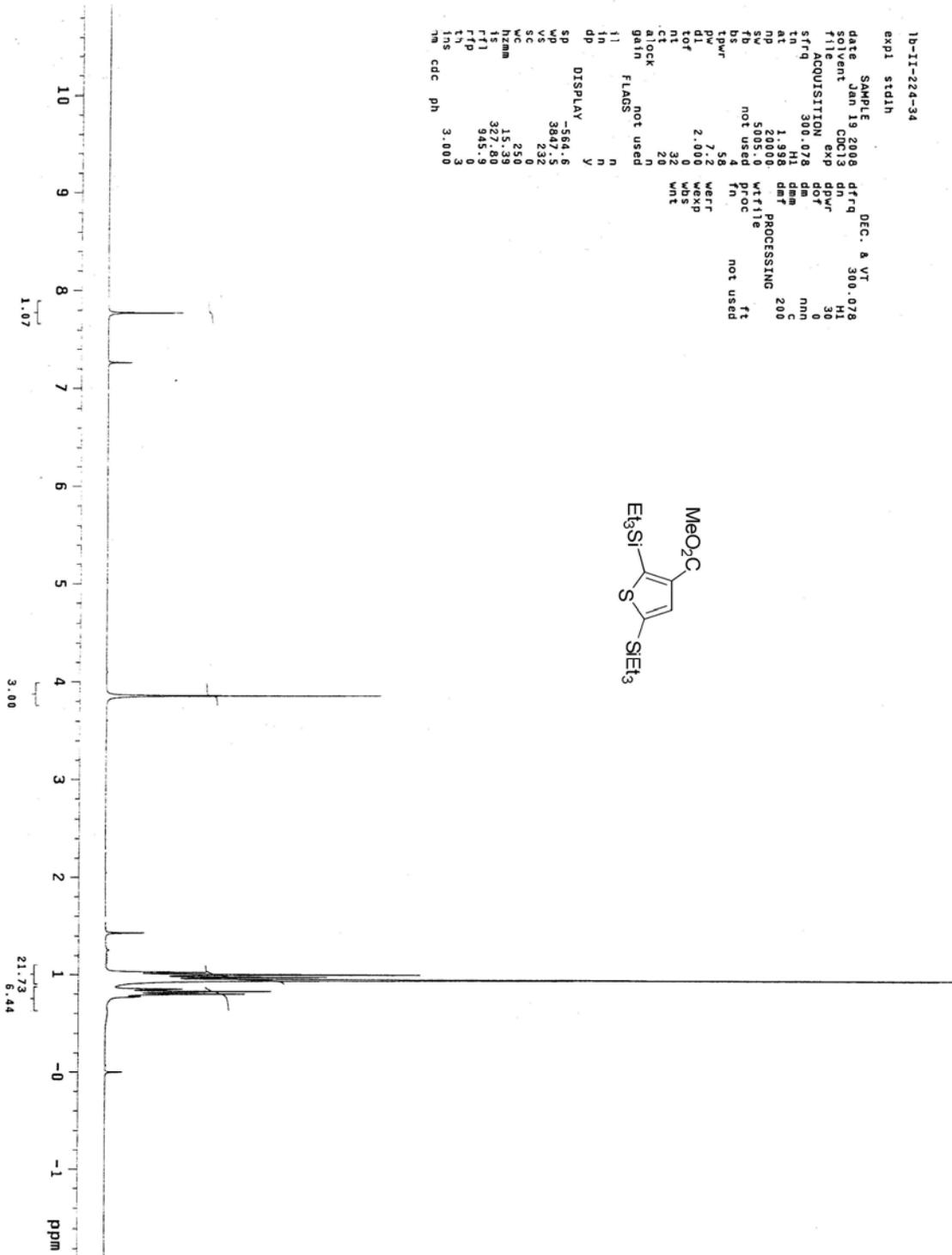
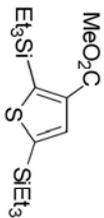




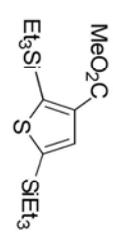
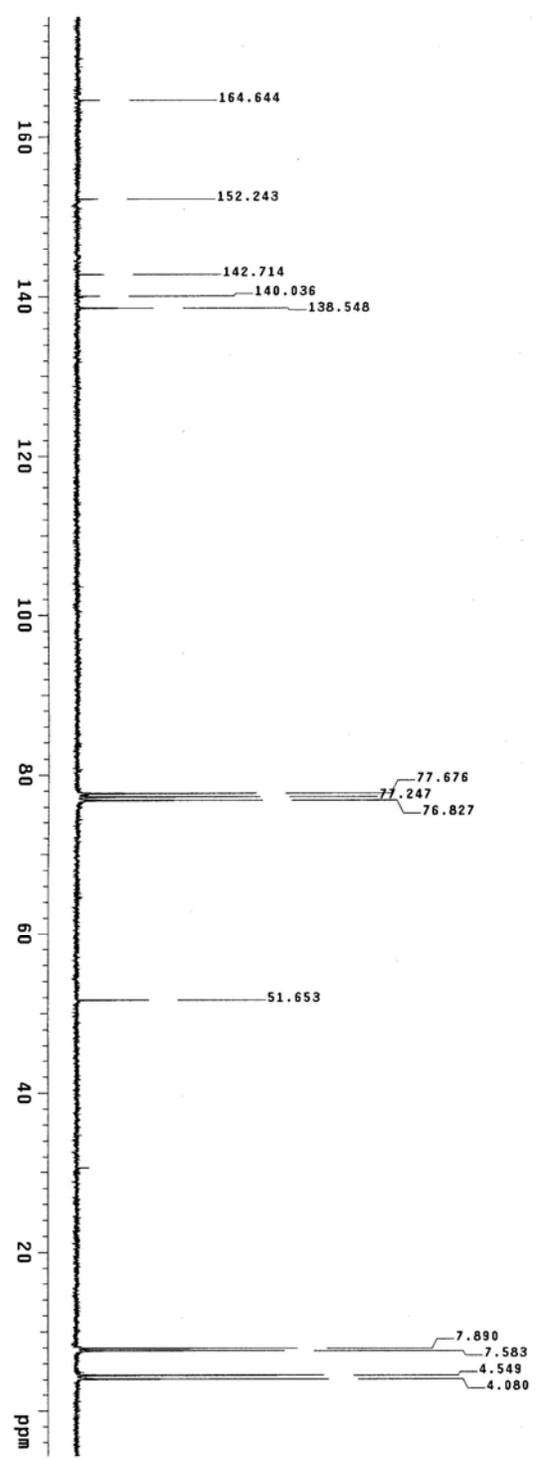
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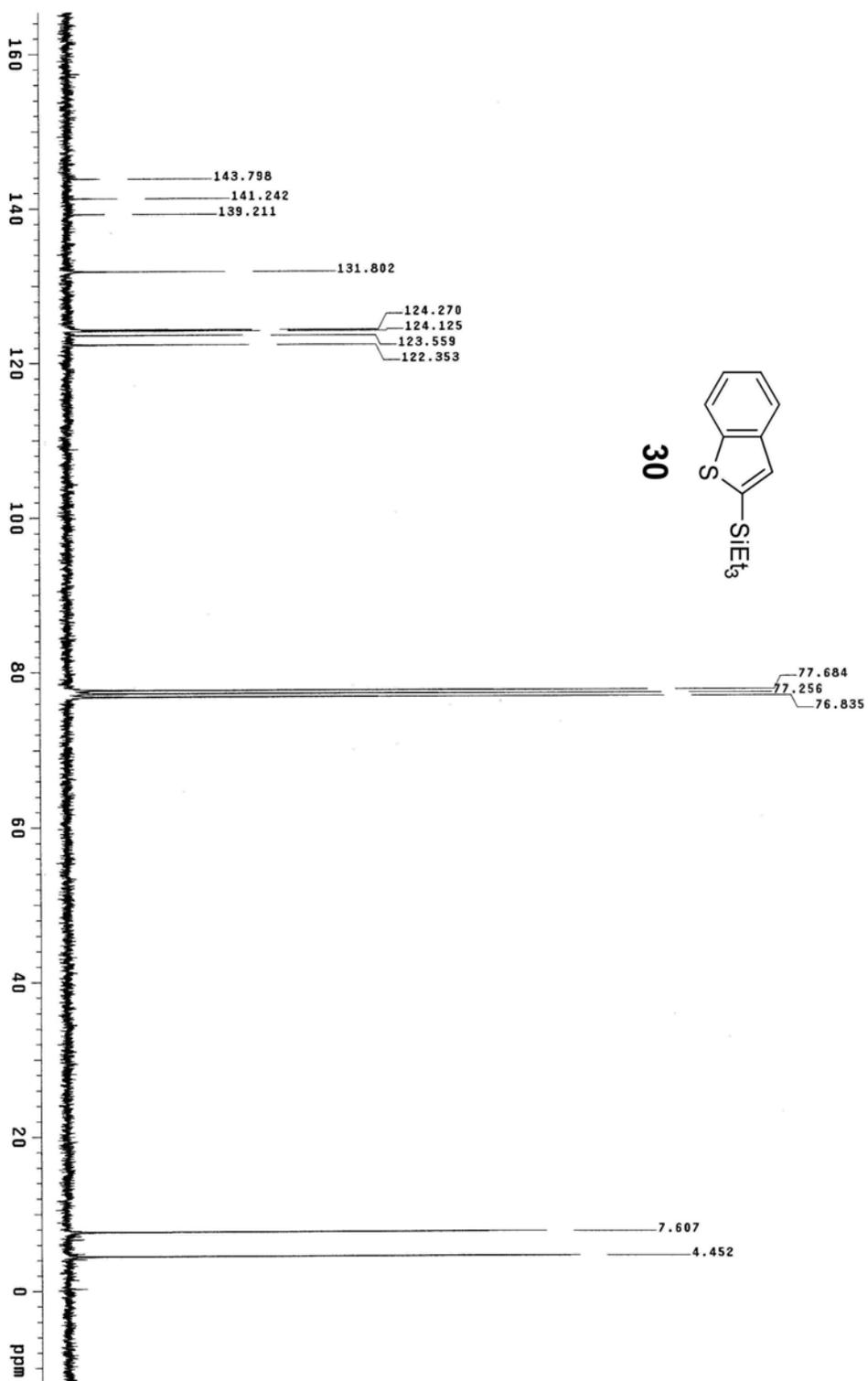
1b-II-224-34
expt stdIn
SAMPLE
date Jan 19 2008 dtrq DEC. 8 VT
solvent CDCl3 dn 300.078
file ACQUISITION exp dpwr H1
sfreq 300.078 dm 30
at 1.998 dmf nmh
np 20000 wtfile PROCESSING 200
sw 5005.0 proc ft
fb not used fn not used
tpwr 56 wprt
dv 7.02 wexp
d1 2.000 wbp
tof 32 wnt
ct 20
gain not used
alock not used
flags not used
ii n
in n
dp y
DISPLAY
sp -564.6
wp 3847.5
vs 232
sc 0
pc 250
hnum 15.30
rs 327.80
rf1 945.9
rfp 0
th 3
ins 3.000
tm cdc ph

```



1b-II-224-34-c
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 Mercury-30088 "pele300"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.000 sec
 SFO 300.13610 MHz
 SFO 75.26535 MHz
 OBSERVE C13 75.4541519 MHz
 DECOUPLE H1 300.0775584 MHz
 Power 37 dB
 on during acquisition
 off during delay
 WALTZ16 Reculated
 DUMPSOLRESSON
 Line broadening 1.0 Hz
 FT size 65536
 Total time 9 hr, 8 min, 38 sec

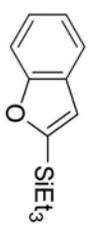




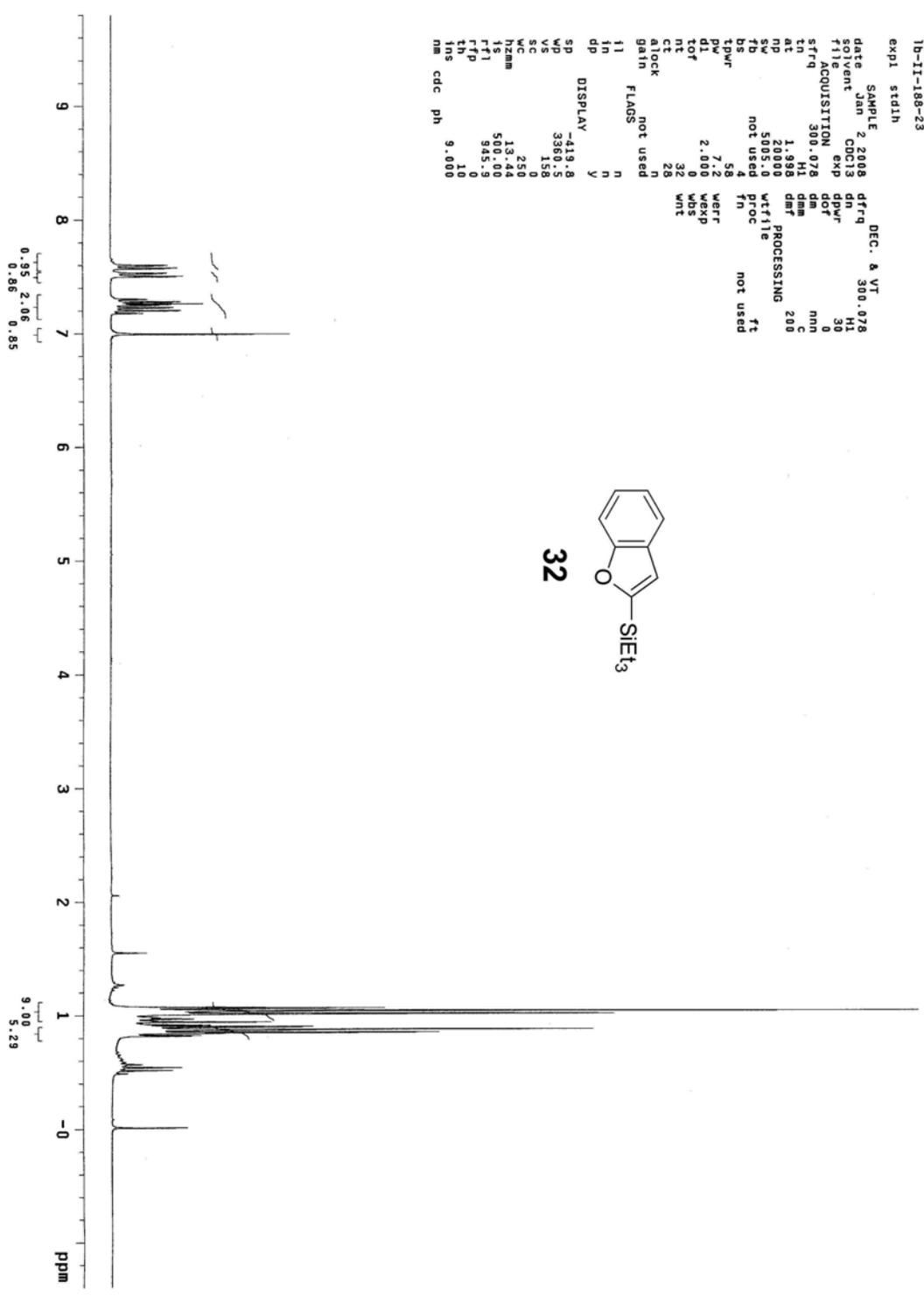
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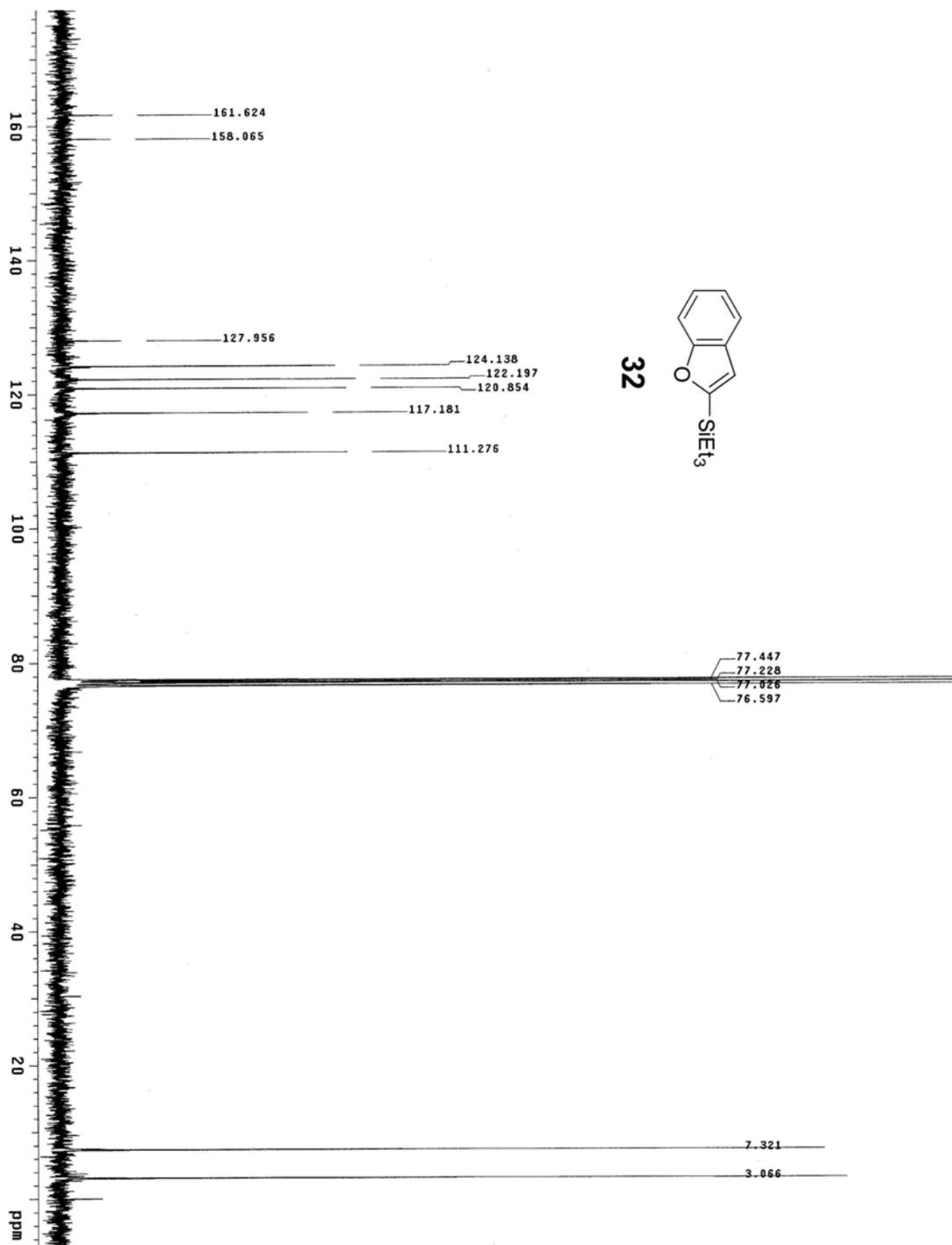
1b-II-188-23
expt stidh
SAMPLE 2 2008 DEC. 8 VT
date Jan 2 CDC13 dfrq 300.078
file ACQUISITION exp dpmr HI
ACQUISITION exp dot 30
sfrq 300.078 dm nm
fn HI dm nm
ac 1.000 dfr 200
sv 5005.0 wifile
fb not used proc ft
bs 4 fn not used
tpwr 58
pwr 7.2 werr
dl 2.000 wexp
tof 32
ct 28
alock not used
gain not used
flags
11 n
1n n
dp y
DISPLAY -419.8
SP 3380.5
V5 158
V6 0
WC 250
WZMM 18.44
F1 94.00
F2 95.00
FFP 0
th 10
ns cdc ph 9.000

```



32

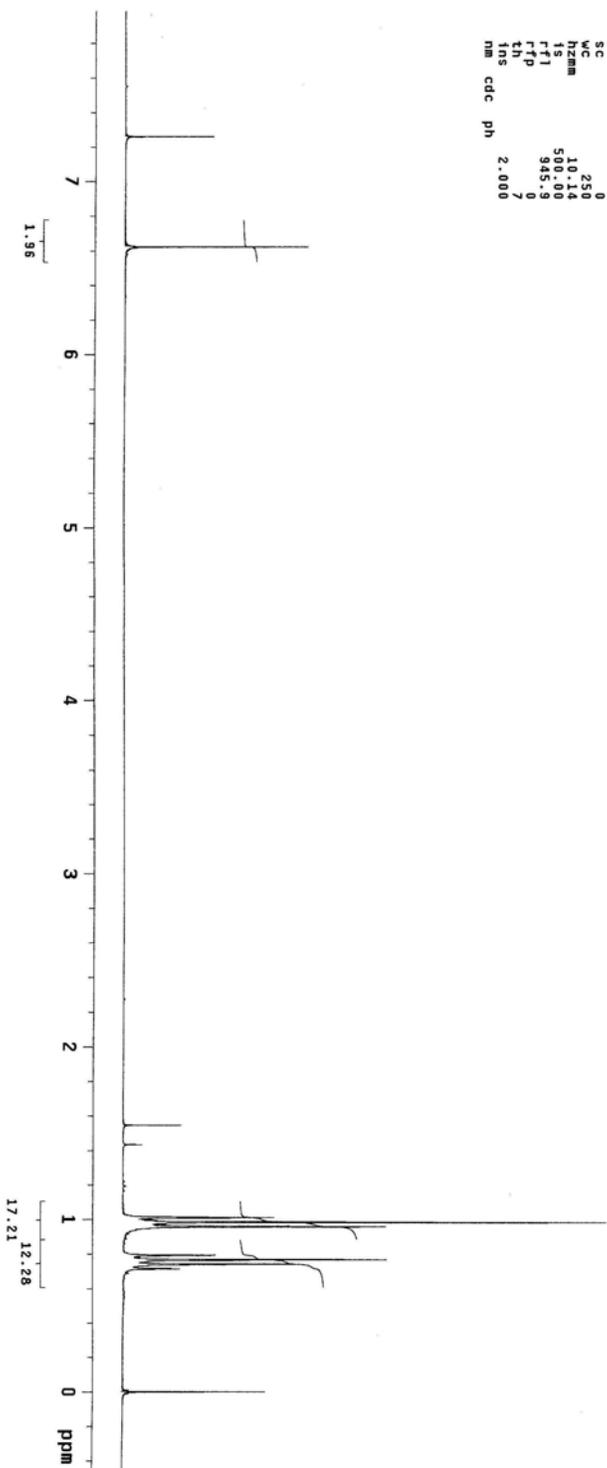
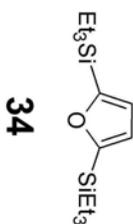


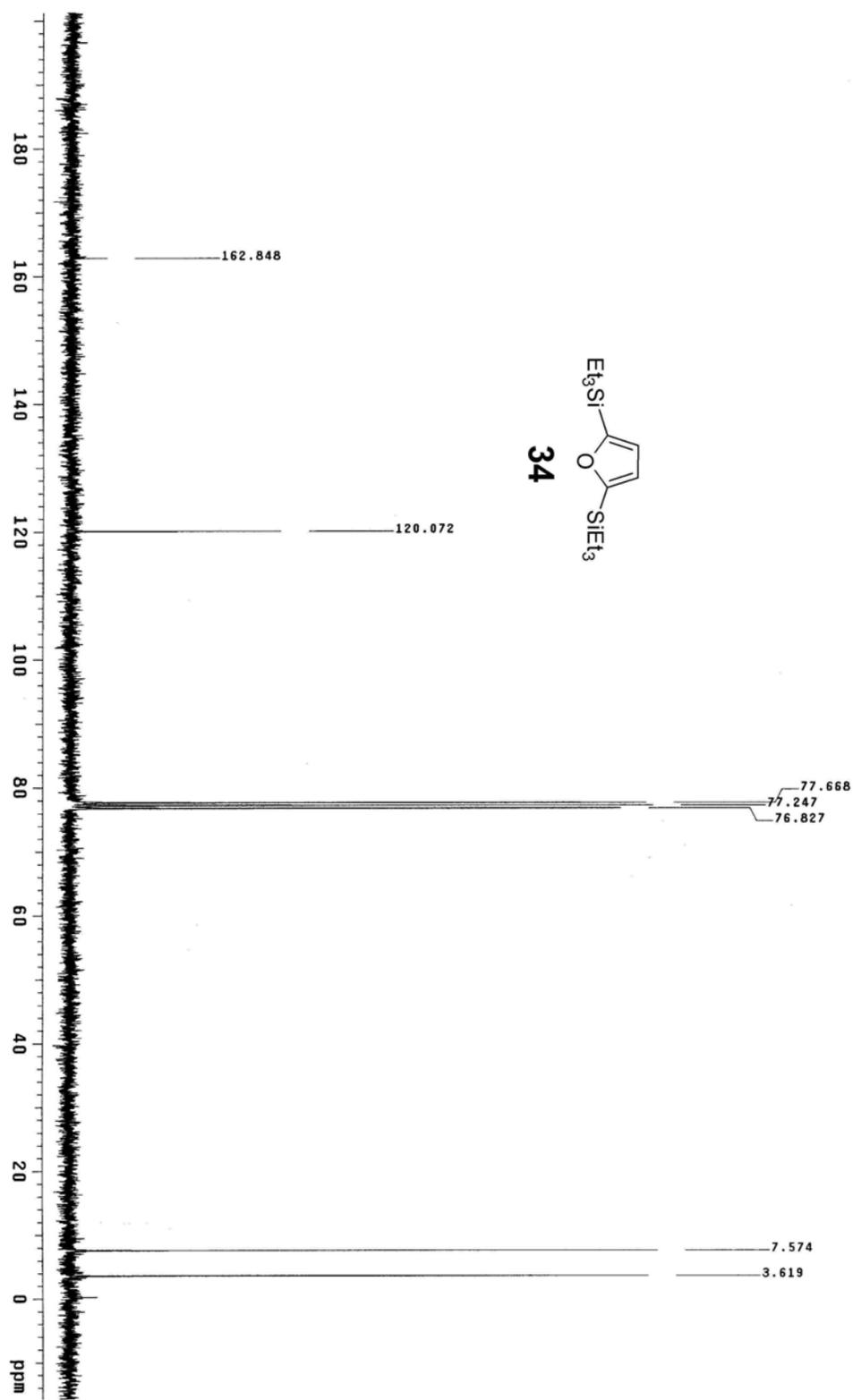


STANDARD 1H OBSERVE

```

expt stdIn
SAMPLE DEC. & VT
date Jan 11 2008 dfrq 300.078
solvent CDCl3 dn H1
F1ACQUISITION exp dpr 30
sfrq 300.078 dm nmn
tn H1 dm C
at 1.998 H1 dmf PROCESSING
np 20000 wtfile ft
sw 5005.0 not used fn not used
ts 58
tswr 58
d1 72 weff
dl 2.000 wexp
nt 0 wbs
ct 32 wnt
atloc not used
gain not used
ll n
in n
dp n
DISPLAY y
SP -137.8
WP 2534.7
VC 82
WC 250
hzmm 10.14
fs 500.00
rf1 945.9
th 0
ims 7
nm cdc ph 2.000
  
```





1b-III-144
expt std1h

SAMPLE DEC. 8 VI 300.078
date Jun 21 2008 dfrq
solvent CDCl3 dn H1
file /data/faic/L~ dpvr 30
ubiao/1b-III-144.f~ dot 0
id dm nnn
dm dmm c

ACQUISITION
sfrq 300.078 dmf PROCESSING 200
n 1/1
at 1.994 wffile
np 20000 proc
sw 5005.0 fn
fb not used
bs 4 werr
tpvr 58 wexp
pw 7.2 wds
dla 2.000 wnt
tc 0
ct 32
atlock n
gain not used

FLAGS not used
11 n
in n
dp y
SD DISPLAY-413.4
wd 3459.2
vs 104
sc 0
wc 250
hzmm 13.84
ls 3814.70
rf1 945.9
rf2 0
rf3 4
ins 4
nm cdc ph 6.000

