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# Supporting Information



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***Supporting Information***

# Synthesis of Enantioenriched α-(Hydroxyalkyl)-tri-n-butylstannanes

Anyu He and J. R. Falck*

*Departments of Biochemistry and Pharmacology, University of Texas Southwestern Medical Center, Dallas, Texas 75390 USA*

*E-mail*: j.falck@UTSouthwestern.edu

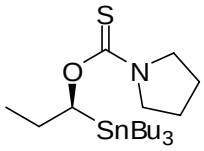
**General.** All reactions were maintained under an argon atmosphere and covered with aluminum foil. Anhydrous solvents were freshly distilled from sodium benzophenone ketyl, except for $CH_2Cl_2$, which was distilled from $CaH_2$. Extracts were dried over anhydrous $Na_2SO_4$ and then filtered prior to removal of all volatiles under reduced pressure. Unless otherwise noted, commercially available materials were used without further purification. 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 or 400 spectrometer at operating frequencies of 300/400 MHz ($^1$H) or 75/100 MHz ($^{13}$C). Chemical shifts (δ) are given in ppm relative to residual solvent (usually chloroform δ = 7.27 for $^1$H NMR or δ = 77.23 for proton decoupled $^{13}$C NMR), and coupling constants (*J*) in Hz. Optical rotations were measured at room temperature (24°C) and corrected to 20 °C on a Rudolph Research Analytical Autopol® IV polarimeter. The Michigan State University Mass Spectroscopy Facility provided high-resolution mass spectral analyses. Racemic products were prepared using 1,2-Bis(diphenylphosphino)ethane (dppe) as ligand. The enantiomeric excess, expressed as %ee, were determined by HPLC analysis as specified in the individual experimental descriptions and verified using the appropriate racemic mixtures. The absolute configuration of compound **9** was established by comparisons of the optical rotation of the MOM protected derivative with literature values. All other absolute configurations were assigned by analogy.

**General experimental procedure.** *n*-$Bu_3SnH$ (1.06 mL, 4 mmol) was added dropwise to a stirring, -78 ºC solution of $Et_2Zn$ (4 mL, 1M in hexane) in anhydrous DME (10 mL) under an argon atmosphere. After 5 min, the reaction mixture was warmed to 4 ºC and kept at this temperature for 1 day. Upon dilution with more DME (27 mL), the reaction mixture was re-cooled -78 ºC and the catalyst (0.2 mmol) in DME (2 mL) and aldehyde (1 mmol) in DME (1 mL) were added sequentially. After 5 min, the temperature was raised to the level indicated in the Table and maintained using a cryogenic cooler. After complete reaction, typically 3-6 h, AcCl (0.2 mL) was added and the reaction mixture was warmed to rt over 0.5 h. After another 2 h, the reaction mixture was subjected to extractive isolation using $CH_2Cl_2$ and the crude product was purified by $SiO_2$ column chromatography.

For the thiocarbamate, the reaction mixture was quenched with saturated aq. $NH_4Cl$, extracted with $CH_2Cl_2$ (3 × 50 mL), and the combined organic extracts were washed with brine, dried over $Na_2SO_4$, and concentrated in *vacuo*. The crude α-hydroxyalkylstannane was re-dissolved in $CH_2Cl_2$ (10 mL) to which was added $Im_2C(S)$ (2 mmol) and DMAP (10 mol%) at rt. After ~ 2 h, the reaction mixture was filtered through a short pad of silica gel. The silica gel pad was rinsed with hexane (40 mL) first to remove the non-polar tin byproduct, then with hexane/EtOAc (1:1, 100 mL). The combined filtrates were concentrated *in vacuo*. The residue was immediately dissolved in neat, anhydrous pyrrolidine (2 mL) at rt. Evaporation of the pyrrolidine after 1 h and purification of the residue by $SiO_2$ column chromatography affords alpha-thiocarbamoyl protected stannane.

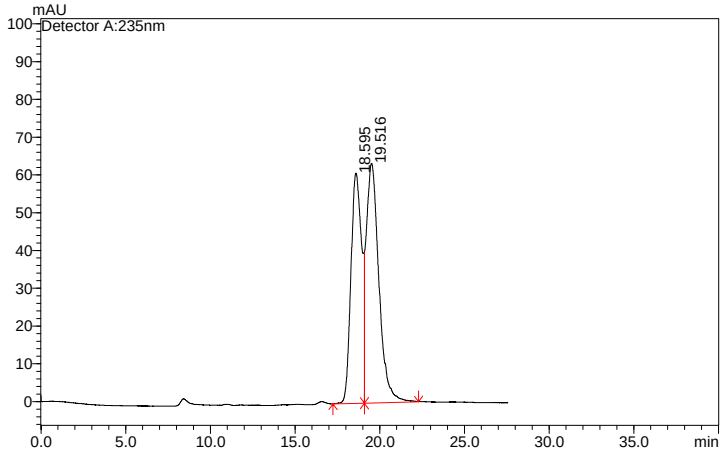
For the 4-nitrobenzoate (**29**), the reaction mixture was quenched with saturated aq. $NH_4Cl$, extracted with $CH_2Cl_2$ (3 × 15 mL). The combined organic extracts were washed with brine, dried over $Na_2SO_4$, and concentrated in *vacuo*. The crude α-hydroxyalkylstannane was protected directly by stirred with 4-nitrobenzoyl chloride (0.5 mmol), Py (0.2 mL) in $CH_2Cl_2$ (10 mL) at rt. After ~ 4 h, the reaction was quenched with water, extracted with $CH_2Cl_2$. The combined organic extracts were washed with brine, dried over $Na_2SO_4$, and concentrated in *vacuo*. The crude product was purified by $SiO_2$ column chromatography.

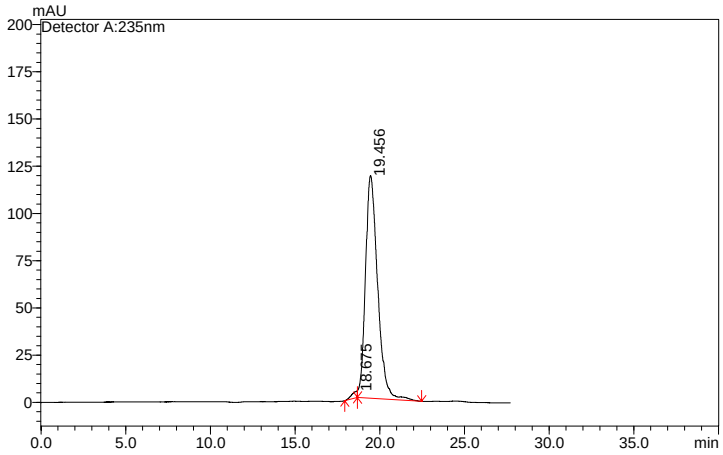
**Characterization data for products and chiral HPLC chromatograms.**




**(S)-O-1-(Tributylstannyl)propyl pyrrolidine-1-carbothioate (9).** (58% y, 97% e.e.). TLC ($SiO_2$): EtOAc/hexane (1:4), Rf ~ 0.66; $^1$H NMR (400 MHz) δ 5.49 (1H, dd, *J* = 7.6, 7.5 Hz), 3.70 (2H, t, *J* = 6.8 Hz), 3.55-3.38 (2H, m), 2.02-1.84 (6H, m), 1.56-1.40 (6H, m), 1.34-1.22 (6H, m), 0.98-0.82 (18H, m); $^{13}$C NMR (100 MHz) δ 185.6, 80.9, 52.0, 47.7, 29.3, 27.9, 27.7,25.9, 24.8, 13.9, 12.3, 10.3; $[\alpha]_{20}$ = +51.6 (c 2.1, $CHCl_3$). HRMS (FAB, NBA) Calcd.for $C_{20}H_{41}NOSSn$ $[MH]^+$ 464.2009, found 464.2011.

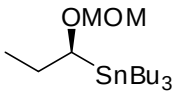
HPLC: Chiralpak AD, 250mm×4.6mm, 205nm, hexane, 0.5 mL/min



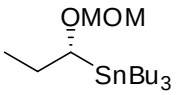

| Peak # | Retention time (min) | Area% |
|---|---|---|
| 1 | 18.7 | 1.4 |
| 2 | 19.5 | 98.6 |

The absolute configuration was determined by converting a small portion of the α-hydroxyalkylstannane to MOM protected derivative, and comparing the optical rotation with literature value.[1]



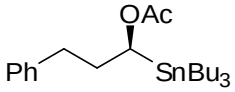

(S)

$[\alpha]^{20}_D$ = +40.0 (c 0.25, $CHCl_3$)



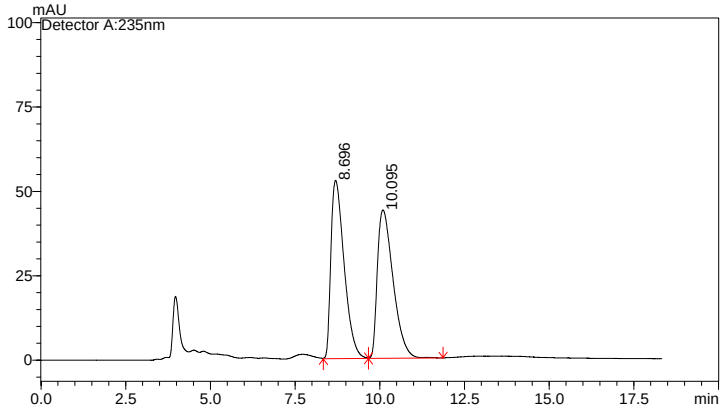

literature value: 98%ee (R)

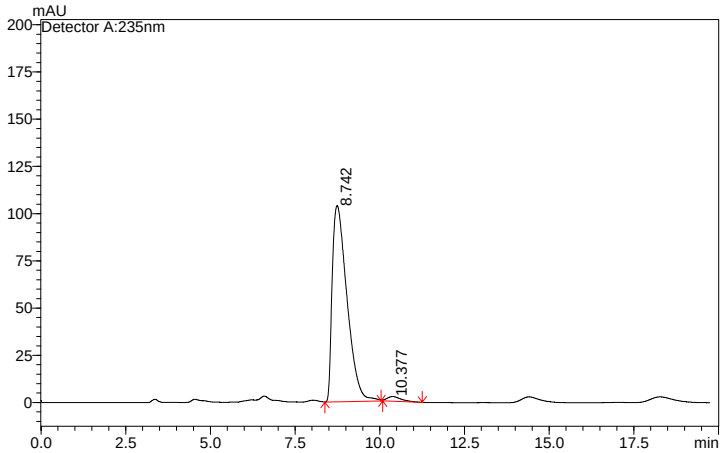
$[\alpha]^{20}_D$ = -38.0 (c 1.3, $CHCl_3$)



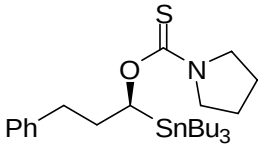

**(*S*)-3-Phenyl-1-(tributylstannyl)propyl acetate (11).**[2] (66% y, 96% e.e.). $[\alpha]^{20}_D$ = +60.1 (c 1.35, $CHCl_3$).

HPLC: Chiralcel OD, 250mm×4.6mm, 235nm, hexane, 1 mL/min



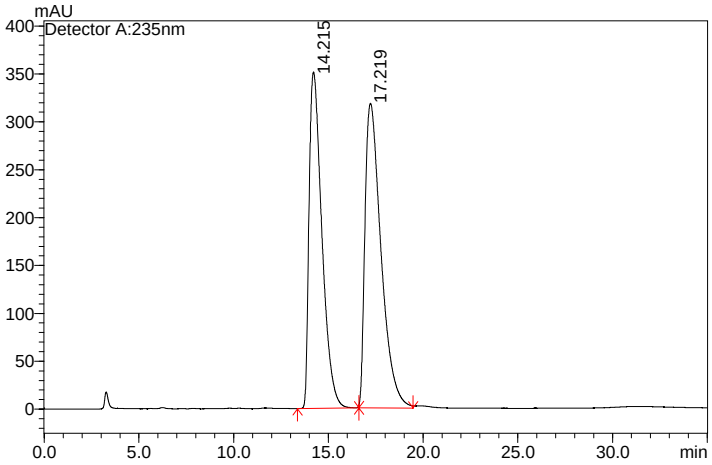



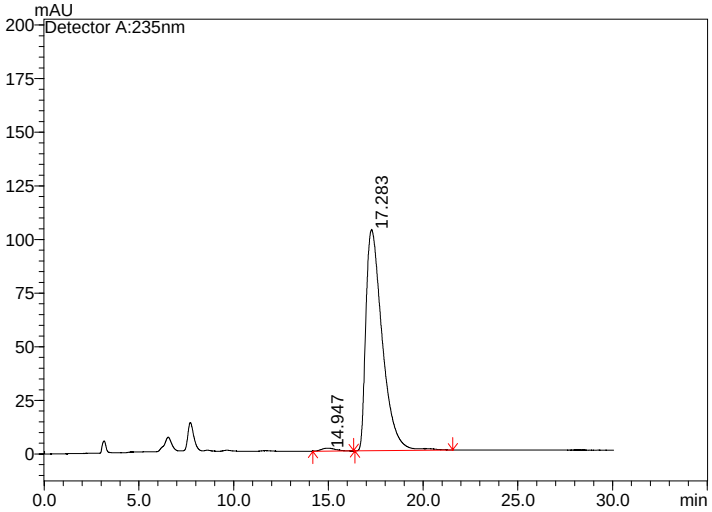

| Peak # | Retention time (min) | Area% |
|---|---|---|
| 1 | 8.7 | 97.8 |
| 2 | 10.4 | 2.2 |



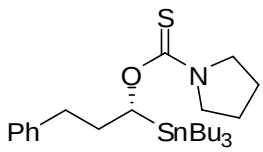

**(*S*)-O-3-Phenyl-1-(tributylstannyl)propyl pyrrolidine-1-carbothioate (12).**[2,3] (69% y, 98% e.e.). $[\alpha]^{20}_D$ = +45.8 (c 1.65, $CHCl_3$).

HPLC: Chiralcel OD, 250mm×4.6mm, 235nm, hexane, 1 mL/min



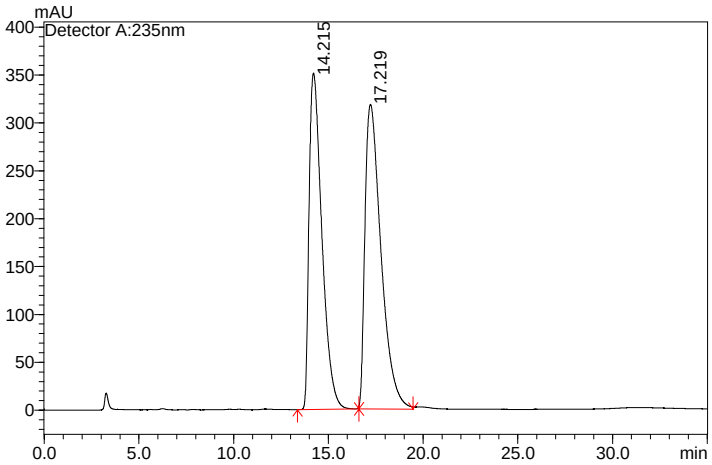



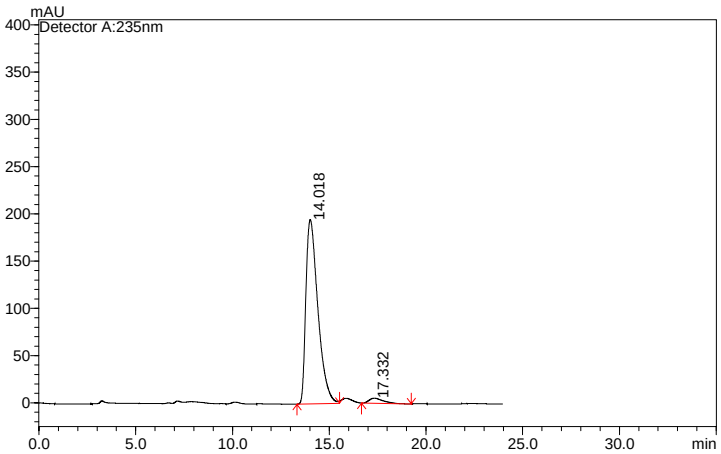

| Peak # | Retention time (min) | Area% |
|---|---|---|
| 1 | 14.9 | 0.9 |
| 2 | 17.3 | 99.1 |



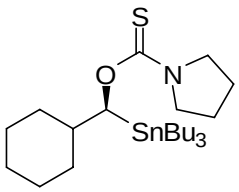

**(*R*)-O-3-Phenyl-1-(tributylstannyl)propyl pyrrolidine-1-carbothioate (13).**[2,3] (68% y, 93% e.e.). $[\alpha]^{20}_{D}$ = -42.9 (c 1.03, $CHCl_3$).

HPLC: Chiralcel OD, 250mm×4.6mm, 235nm, hexane, 1 mL/min

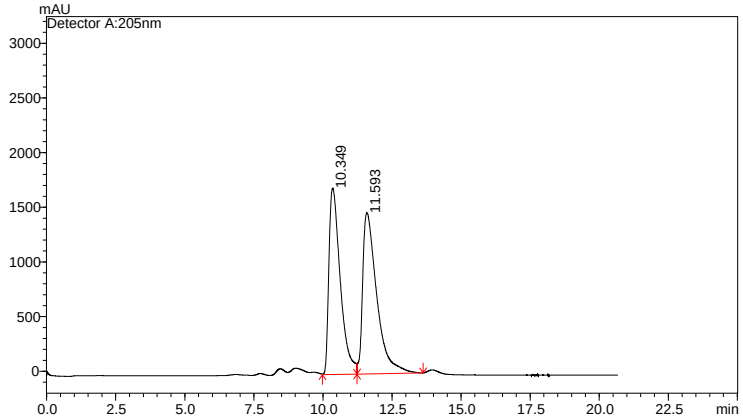


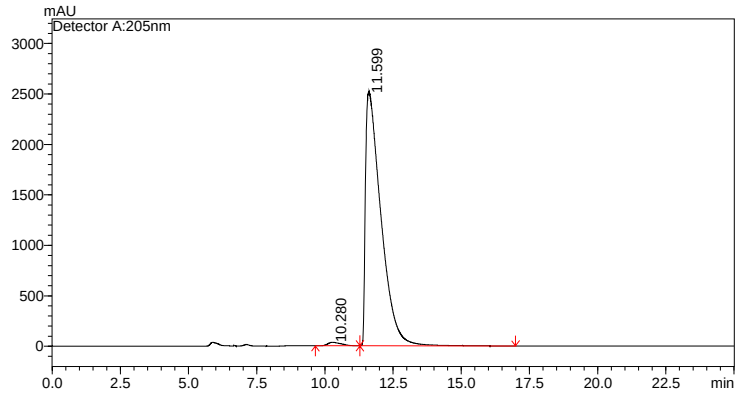




| Peak # | Retention time (min) | Area% |
|---|---|---|
| 1 | 14.0 | 96.9 |
| 2 | 17.3 | 3.1 |

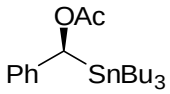



**(*S*)-O-Cyclohexyl(tributylstannyl)methyl pyrrolidine-1-carbothioate (15).** (70% y, 98% e.e.). TLC ($SiO_2$) hexane/EtOAc (6:1), $R_f \approx 0.58$; $^1$H NMR (300 MHz, $CDCl_3$) δ 5.53 (1H, d, *J* = 6 Hz), 3.69 (2H, t, *J* = 6 Hz), 3.53-4.43 (2H, m), 1.972-1.84 (5H, m), 1.75-1.68 (4H, m), 1.53-1.43 (6H, m), 1.35–0.97 (12H, m), 0.91–0.84 (15H, m); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 185.5, 85.1, 52.0, 47.7, 42.6, 31.5, 30.8, 29.3, 27.7, 26.7, 26.39, 26.36, 25.9, 24.8, 13.9, 10.8. $[\alpha]^{20}_D = +35.5$ (c 1.5, $CHCl_3$). HRMS (ESI) Calcd. for $C_{24}H_{48}NOSSn$ [$MH^+$] *m/z* 518.2479, found 518.2486. HPLC: Chiralpak AD, 250mm×4.6mm, 205nm, hexane, 0.5 mL/min



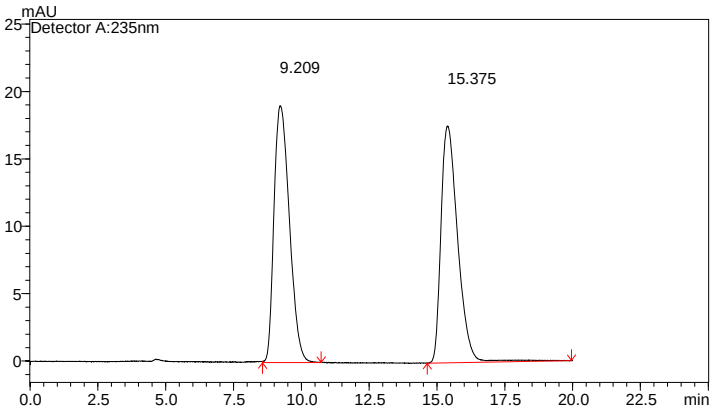



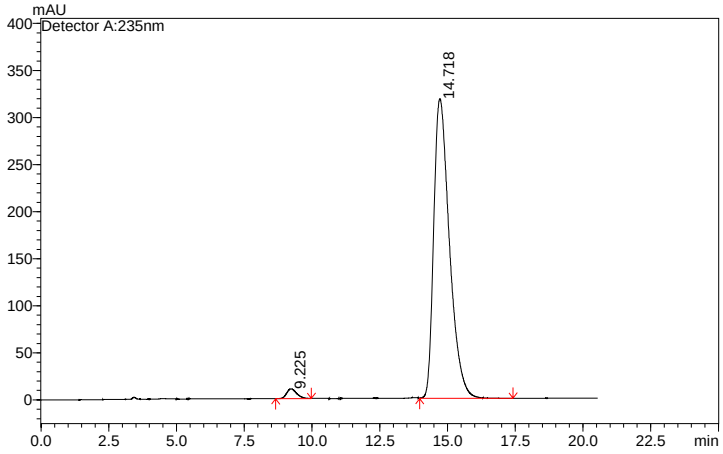

| Peak # | Retention time (min) | Area% |
|---|---|---|
| 1 | 10.3 | 0.8 |
| 2 | 11.6 | 99.2 |



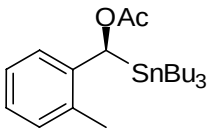

**(*S*)-Phenyl(tributylstannyl)methyl acetate (4).**[4] (60% y, 96% e.e.). $[\alpha]^{20}_{D}$ = +3.8 (c 1.15, $CHCl_3$).

HPLC: Chiralcel OD, 250mm×4.6mm, 235nm, hexane, 1 mL/min



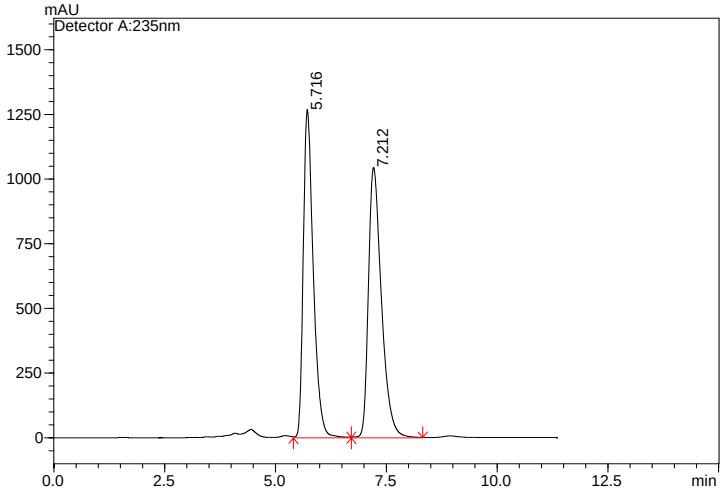


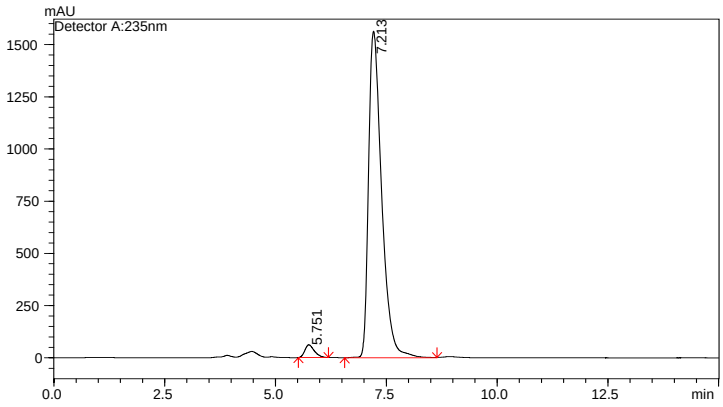

| Peak # | Retention time (min) | Area% |
|---|---|---|
| 1 | 9.2 | 2.0 |
| 2 | 14.7 | 98.0 |



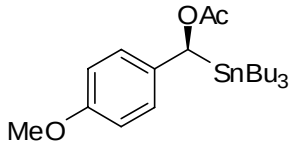

**(*S*)-o-Tolyl(tributylstannyl)methyl acetate (17).** (54% y, 95% e.e.). TLC ($SiO_2$) hexane/EtOAc (6:1), $R_f \approx 0.55$; $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.26 (1H, dd, *J* = 8, 1 Hz), 7.17 (1H, dt, *J* = 7, 1 Hz), 7.09 (1H, dd, *J* = 7, 1 Hz), 7.02 (1H, dt, *J* = 8, 1 Hz), 5.92 (1H, s), 2.20 (3H, s), 2.14 (3H, s), 1.42-1.32 (6H, m), 1.29–1.17 (6H, m), 0.86–0.77 (15H, m); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 171.0, 140.8, 131.4, 130.3, 126.4, 125.2, 124.2. $[\alpha]^{20}_D$ = -14.0 (c 0.8, $CHCl_3$). HRMS (FAB, TEGDME) Calcd. for $C_{18}H_{29}O_2O_2Sn$ [$M^+$-Bu] *m/z* 397.1190, found 397.1195.

HPLC: Chiralcel OD, 250mm×4.6mm, 235nm, hexane, 1 mL/min



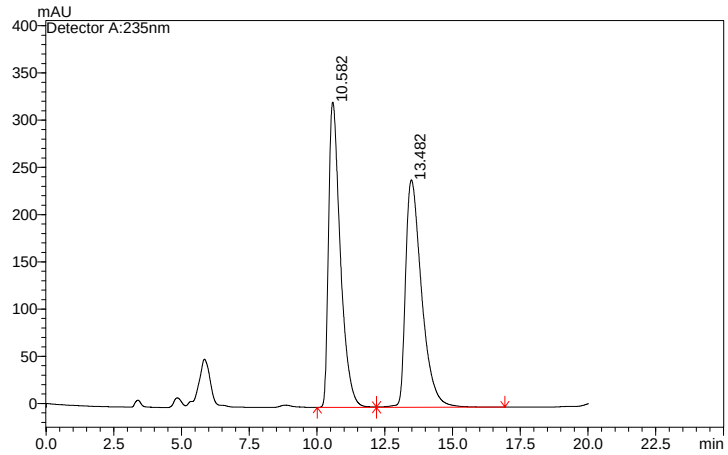


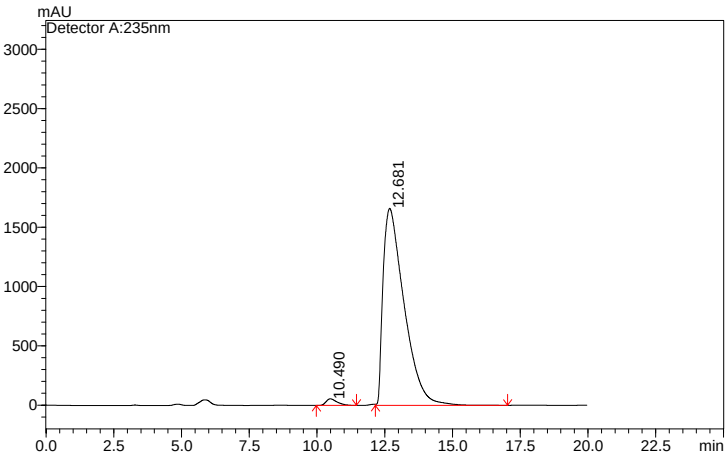

| Peak # | Retention time (min) | Area% |
|---|---|---|
| 1 | 5.8 | 2.6 |
| 2 | 7.2 | 97.4 |




**(*S*)-(4-Methoxyphenyl)(tributylstannyl)methyl acetate (19).**[5] (85% y, 97% e.e.). $[\alpha]^{20}_{D}$ = -6.4 (c 1.0, $CHCl_3$).

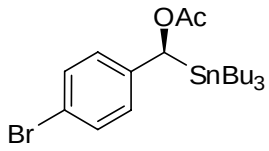
HPLC: Chiralcel OD, 250mm×4.6mm, 235nm, hexane, 1 mL/min



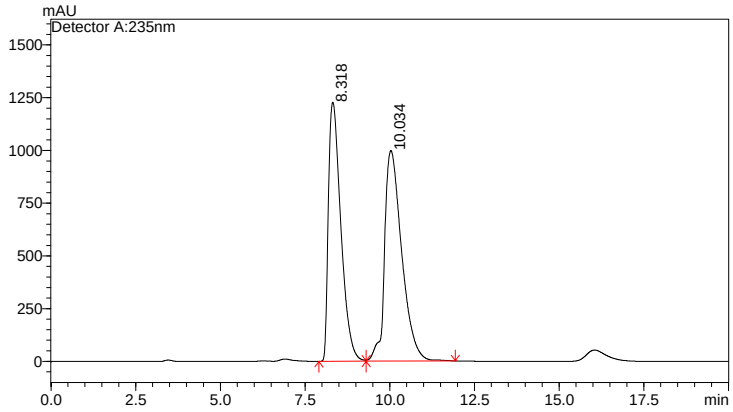

| Peak # | Retention time (min) | Area% |
|---|---|---|
| 1 | 10.5 | 1.7 |

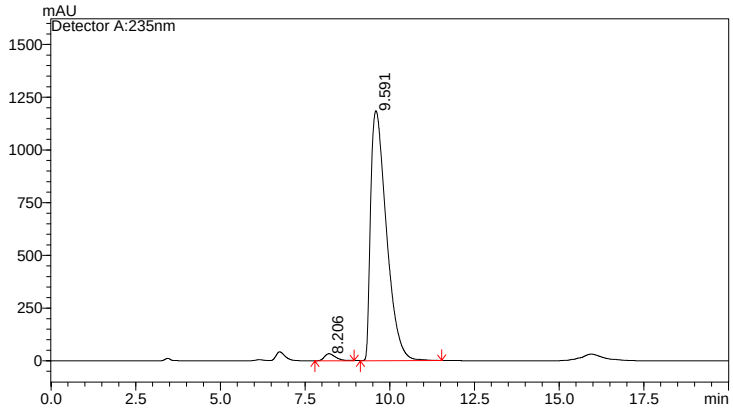
| | | |
|---|---|---|
| 2 | 12.7 | 98.3 |



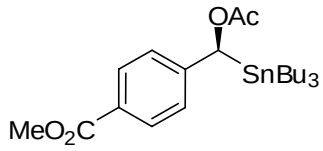

**(*S*)-(4-Bromophenyl)(tributylstannyl)methyl acetate (21).** (60% y, 96% e.e.). TLC ($SiO_2$) hexane/EtOAc (2:1), $R_f$ ≈ 0.66; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.39 (2H, d, *J* = 8 Hz), 6.98 (2H, d, *J* = 8 Hz), 5.86 (1H, s), 2.13 (3H, s), 1.42-1.34 (6H, m), 1.30–1.18 (6H, m), 0.88–0.80 (15H, m); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 171.0, 142.3, 131.6, 125.5, 118.5, 72.8, 29.0, 27.5, 21.2, 13.9, 10.2. $[\alpha]^{20}_D$ = -26.1 (c 1.05, $CHCl_3$). HRMS (FAB, NPOE) Calcd. for $C_{17}H_{26}O_2SnBr$ [$M^+$-Bu] *m/z* 461.0138, found 461.0134.

HPLC: Chiralcel OD, 250mm×4.6mm, 235nm, hexane, 1 mL/min



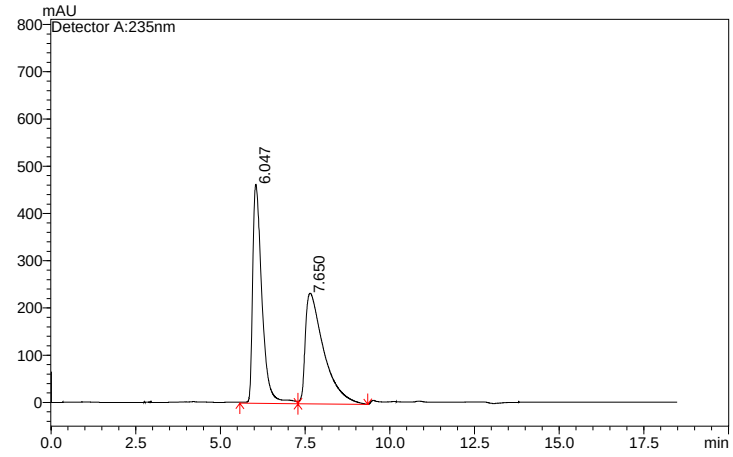



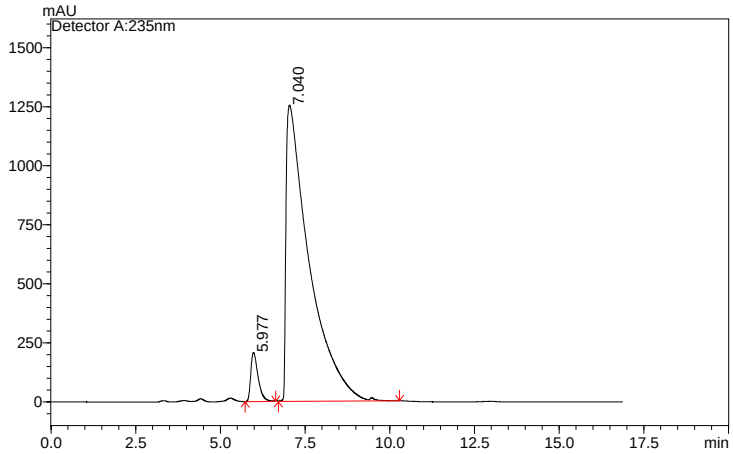

| Peak # | Retention time (min) | Area% |
|---|---|---|
| 1 | 8.2 | 2.0 |
| 2 | 9.6 | 98.0 |



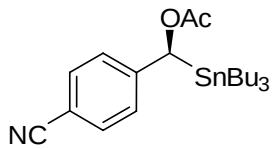
**(*S*)-Methyl 4-(acetoxy(tributylstannyl)methyl)benzoate (23).** (80% y, 87% e.e.). TLC ($SiO_2$) hexane/EtOAc (6:1), $R_f \approx 0.34$; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.96 (2H, d, *J* = 8 Hz), 7.14 (2H, d, *J* = 8 Hz), 5.99 (1H, s), 3.90 (3H, s), 2.16 (3H, s), 1.45-1.35 (6H, m), 1.30–1.18 (6H, m), 0.88–0.81 (15H, m); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 170.9, 167.3, 148.9, 130.2, 126.8, 123.1, 73.2, 52.2, 28.9, 27.5, 21.2, 13.8, 10.3. $[\alpha]^{20}_D$ = -60.3 (c 0.7, $CHCl_3$). HRMS (FAB, NBA) Calcd. for $C_{23}H_{38}O_4Sn$ [$M^+$] *m/z* 498.1792, found 498.1791.

HPLC: Chiralpak AD, 250mm×4.6mm, 235nm, hexane/$^i$PrOH/AcOH 100:0.2:0.1, 1 mL/min



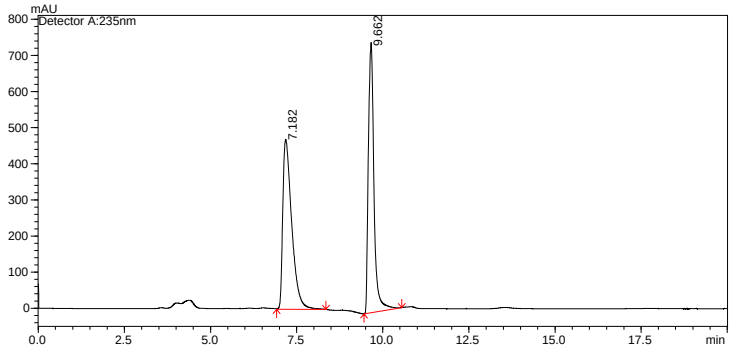



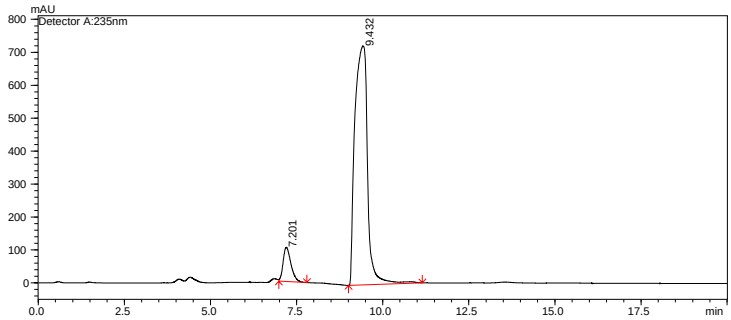

| Peak # | Retention time (min) | Area% |
|---|---|---|
| 1 | 6.0 | 5.0 |
| 2 | 7.0 | 95.0 |



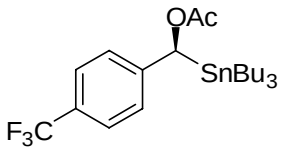

**(*S*)-(4-Cyanophenyl)(tributylstannyl)methyl acetate (25).** (86% y, 84% e.e.)**.** TLC ($SiO_2$) hexane/EtOAc (6:1), $R_f \approx 0.32$; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.57 (2H, d, *J* = 8 Hz), 7.17 (2H, dd, *J* = 8, 1 Hz), 5.96 (1H, s), 2.16 (3H, s), 1.45-1.34 (6H, m), 1.30–1.18 (6H, m), 0.88–0.82 (15H, m); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 170.7, 149.2, 132.5, 123.7, 119.4, 108.3, 72.8, 28.9, 27.5, 21.1, 13.8, 10.4. $[\alpha]^{20}_D$ = -58.0 (c 1.2, $CHCl_3$). HRMS (FAB, NBA) Calcd. for $C_{22}H_{35}O_2SnN$ [$M^+$] *m/z* 465.1690, found 465.1688.

HPLC: Chiralpak AD, 250mm×4.6mm, 235nm, hexane/$^i$PrOH/AcOH 100:0.2:0.1, 1 mL/min



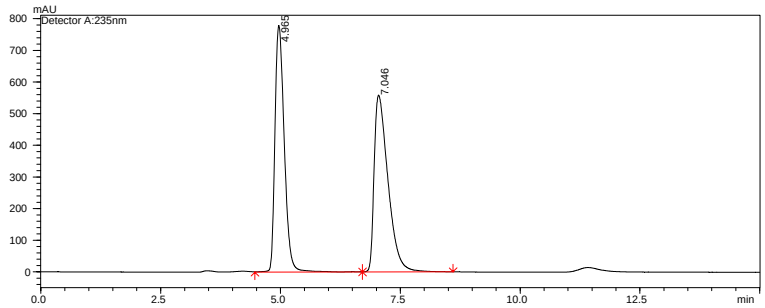



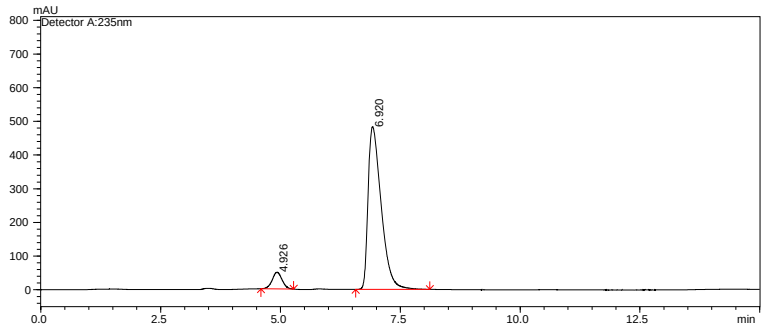

| Peak # | Retention time (min) | Area% |
|---|---|---|
| 1 | 7.2 | 8.2 |
| 2 | 9.4 | 91.8 |



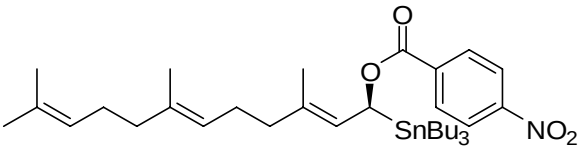

**(*S*)-(Tributylstannyl)(4-(trifluoromethyl)phenyl)methyl acetate (27).** (72% y, 86% e.e.). TLC ($SiO_2$) hexane/EtOAc (6:1), $R_f \approx 0.52$; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.53 (2H, d, $J$ = 8 Hz), 7.19 (2H, d, $J$ = 8 Hz), 5.97 (1H, s), 2.16 (3H, s), 1.42-1.34 (6H, m), 1.28–1.19 (6H, m), 0.87–0.83 (15H, m); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 170.9, 147.5, 125.7, 125.6, 123.5, 72.9, 28.9, 27.5, 21.2, 13.8, 10.3. $[\alpha]^{20}_D$ = -24.9 (c 1.4, $CHCl_3$). HRMS (FAB, NBA) Calcd. for $C_{18}H_{26}F_3O_2Sn$ [$M^+$-Bu] *m/z* 451.0907, found 451.0910.

HPLC: Chiralpak AD, 250mm×4.6mm, 235nm, hexane, 1 mL/min



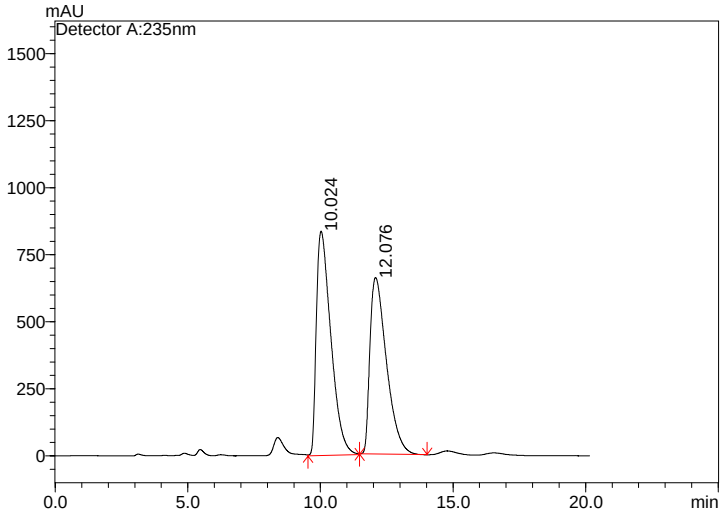


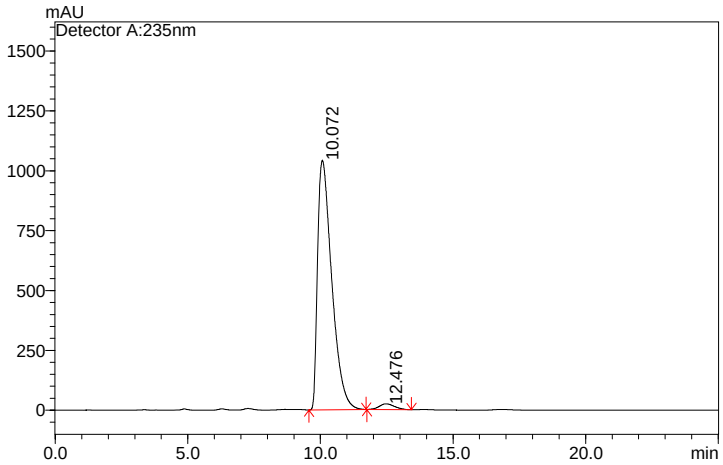

| Peak # | Retention time (min) | Area% |
|---|---|---|
| 1 | 4.9 | 7.1 |
| 2 | 6.9 | 92.9 |



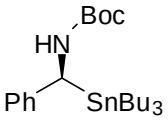

**(2*E*,6*E*)-3,7,11-Trimethyl-1-(tributylstannyl)dodeca-2,6,10-trienyl 4-nitrobenzoate (29).** (56% y, 95% e.e.). TLC ($SiO_2$) hexane/EtOAc (6:1), $R_f \approx 0.66$; $^1$H NMR (300 MHz, $CDCl_3$) δ 8.30-8.26 (2H, m), 8.21-8.16 (2H, m), 5.89 (1H, d, *J* = 11 Hz), 5.65 (1H, dd, *J* = 11, 1 Hz), 5.14-5.06 (2H, m), 2.16-1.95 (8H, m), 1.70 (3H, d, *J* = 1 Hz), 1.68 (3H, s), 1.62 (3H, s), 1.59 (3H, s), 1.53–1.45 (6H, m), 1.34–1.22 (6H, m), 0.96–0.84 (15H, m); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 164.8, 150.5, 136.3, 135.6, 133.1, 131.6, 1330.6, 124.5, 124.5, 124.1, 123.71, 123.65. $[\alpha]^{20}_D$ = -9.4 (c 2.7, $CHCl_3$). HRMS (FAB, NPOE) Calcd. for $C_{27}H_{51}OSn$ [$M^+$-(4-nitrobenzoyl)] *m/z* 511.2962, found 511.2966.

HPLC: Chiralcel OD, 250mm×4.6mm, 235nm, hexane, 1 mL/min



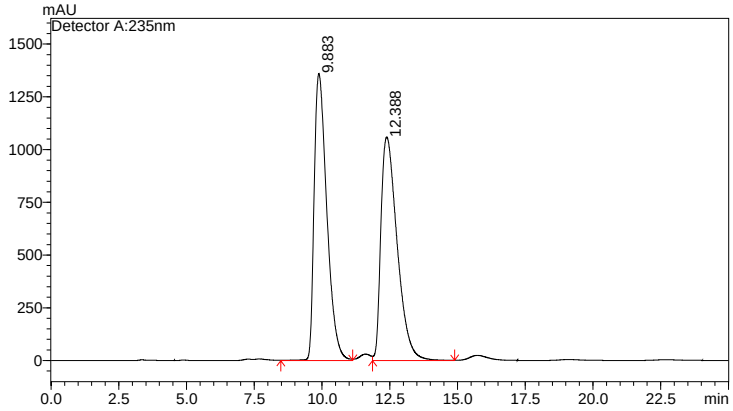


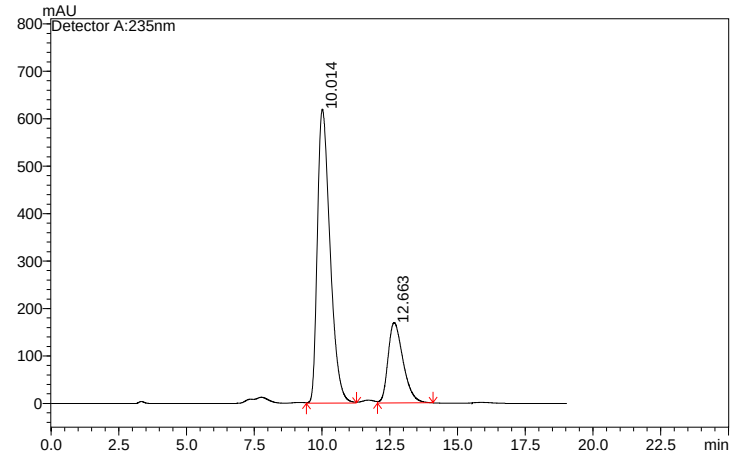

| Peak # | Retention time (min) | Area% |
|---|---|---|
| 1 | 10.1 | 97.6 |
| 2 | 12.5 | 2.4 |




**(S)-tert-Butyl phenyl(tributylstannyl)methylcarbamate.** Prepared as general procedure. After reaction is complete, the reaction mixture was quenched with saturated aq. $NH_4Cl$, extracted with $CH_2Cl_2$ (3 × 15 mL). The combined organic extracts were washed with brine, dried over $Na_2SO_4$, and concentrated in *vacuo*. The crude product was purified by $SiO_2$ column chromatography. (81% y, 51% e.e.). TLC ($SiO_2$) hexane/EtOAc (6:1), $R_f \approx 0.33$; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.23 (2H, t, $J$ = 8 Hz), 7.07-7.00 (3H, m), 5.16 (1H, d, $J$ = 5 Hz), 4.18 (1H, d, $J$ = 5 Hz), 1.46 (9H, s), 1.43–1.33 (6H, m), 1.29–1.17 (6H, m), 0.87–0.77 (15H, m); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 157.1, 145.8, 128.6, 124.6, 124.4, 79.4, 45.9, 29.1, 28.6, 27.7, 13.9, 11.2. $[\alpha]^{20}_D$ -17.4 (c 1.65, $CHCl_3$). HRMS (FAB, Gly) Calcd. for $C_{20}H_{34}O_2O_2SnN$ [$M^+$-Bu] *m/z* 440.1612, found 440.1615.

HPLC: Chiralcel OD, 250mm×4.6mm, 235nm, hexane, 1 mL/min




| Peak # | Retention time (min) | Area% |
|---|---|---|
| 1 | 10.0 | 75.5 |
| 2 | 12.7 | 24.5 |

**References**

(1) Chong, J. M.; Mar. E. K. *Tetrahedron* **1989**, *45*, 7709.

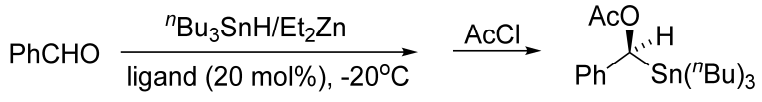
(2) (a) Itoh, T; Ohta, T. *Tetrahedron Lett.* **1990**, *31*, 6407. (b) Falck, J. R.; Bhatt, R. K.; Ye, J. *J. Am. Chem. Soc.* **1995**, *117*, 5973.

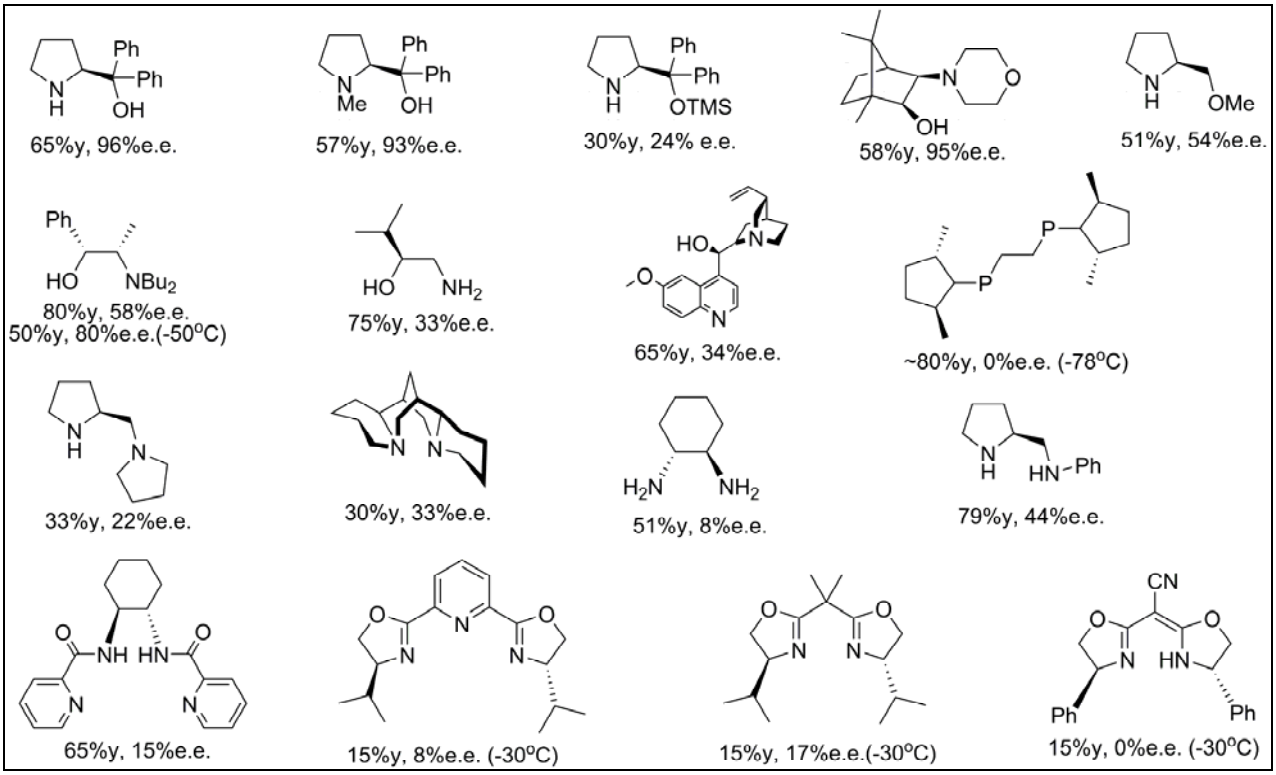
(3) Bhatt, R. K.; Ye, J.; Falck, J. R. *Tetrahedron Lett.* **1996**, *37*, 3811.

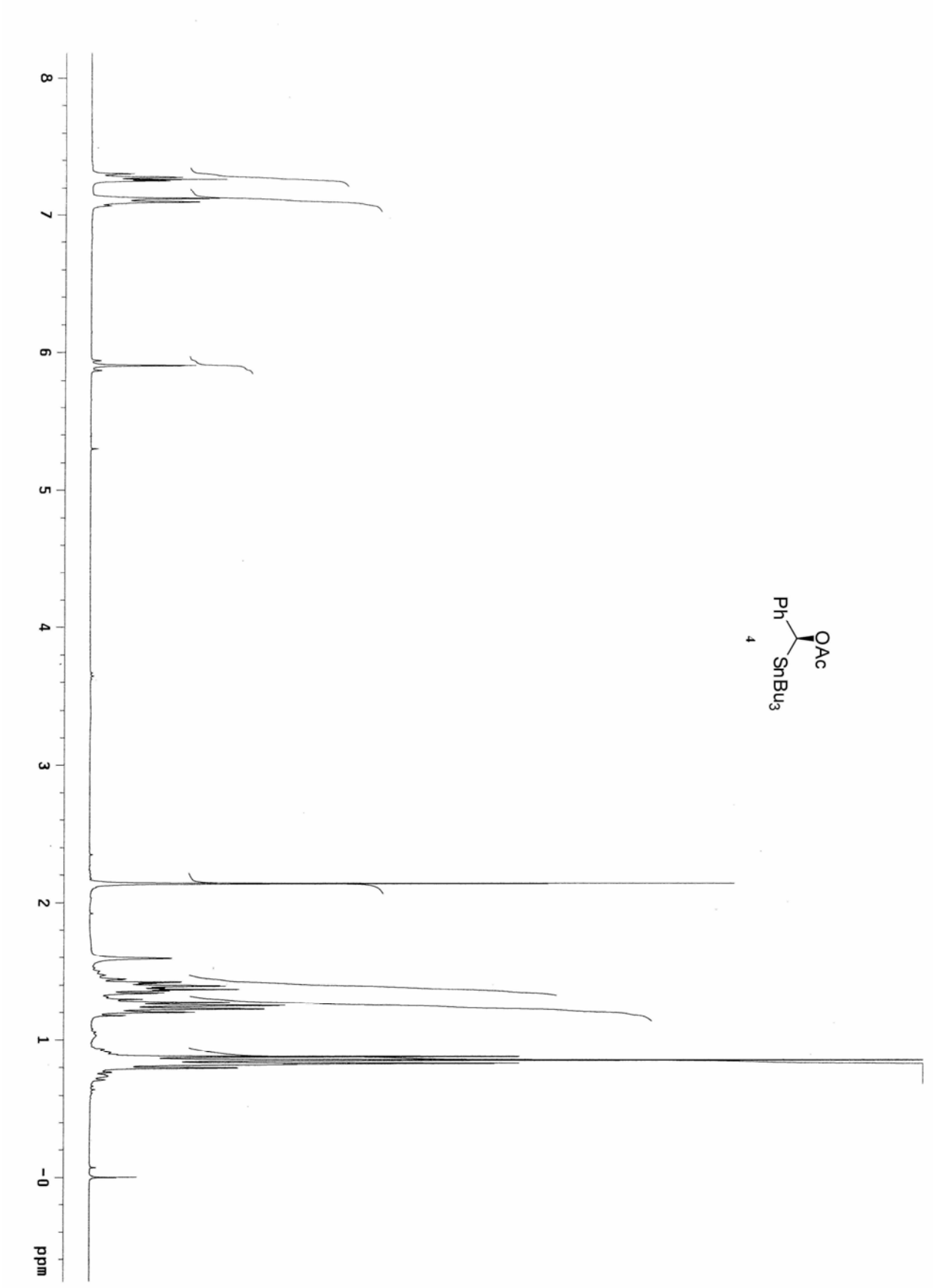
(4) (a) Ye, J.; Bhatt, R. K.; Falck, J. R. *J. Am. Chem. Soc.* **1994**, *116*, 1. (b) Yoshida, J.; Morita, Y.; Ishichi, Y.; Isoe, S. *Tetrahedron Lett.* **1994**, *35*, 5247.

(5) Sugawara, M.; Yoshida, J. *J. Org. Chem.* **2000**, *65*, 3135.

**Chiral ligands screening.**

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ppm

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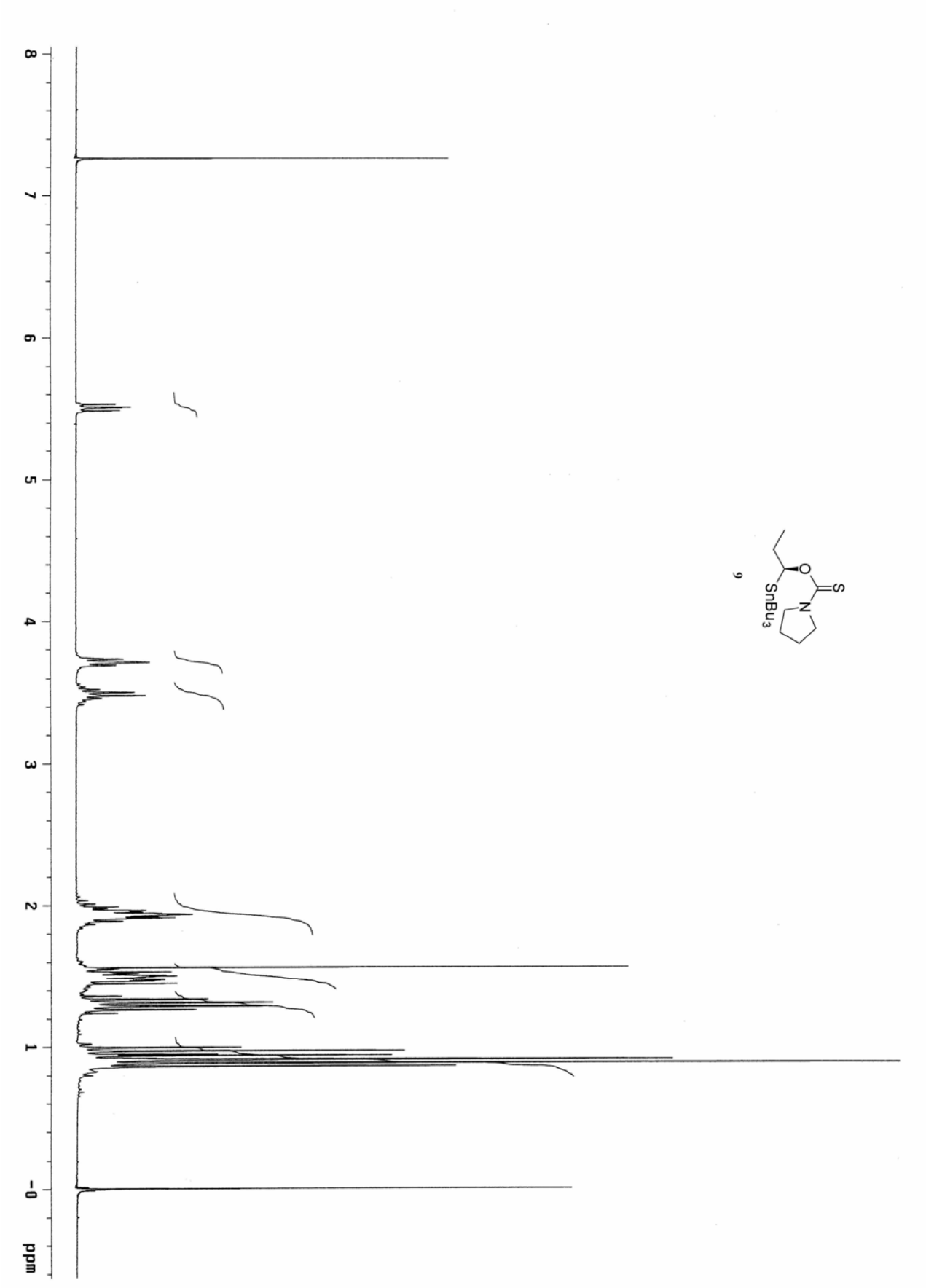
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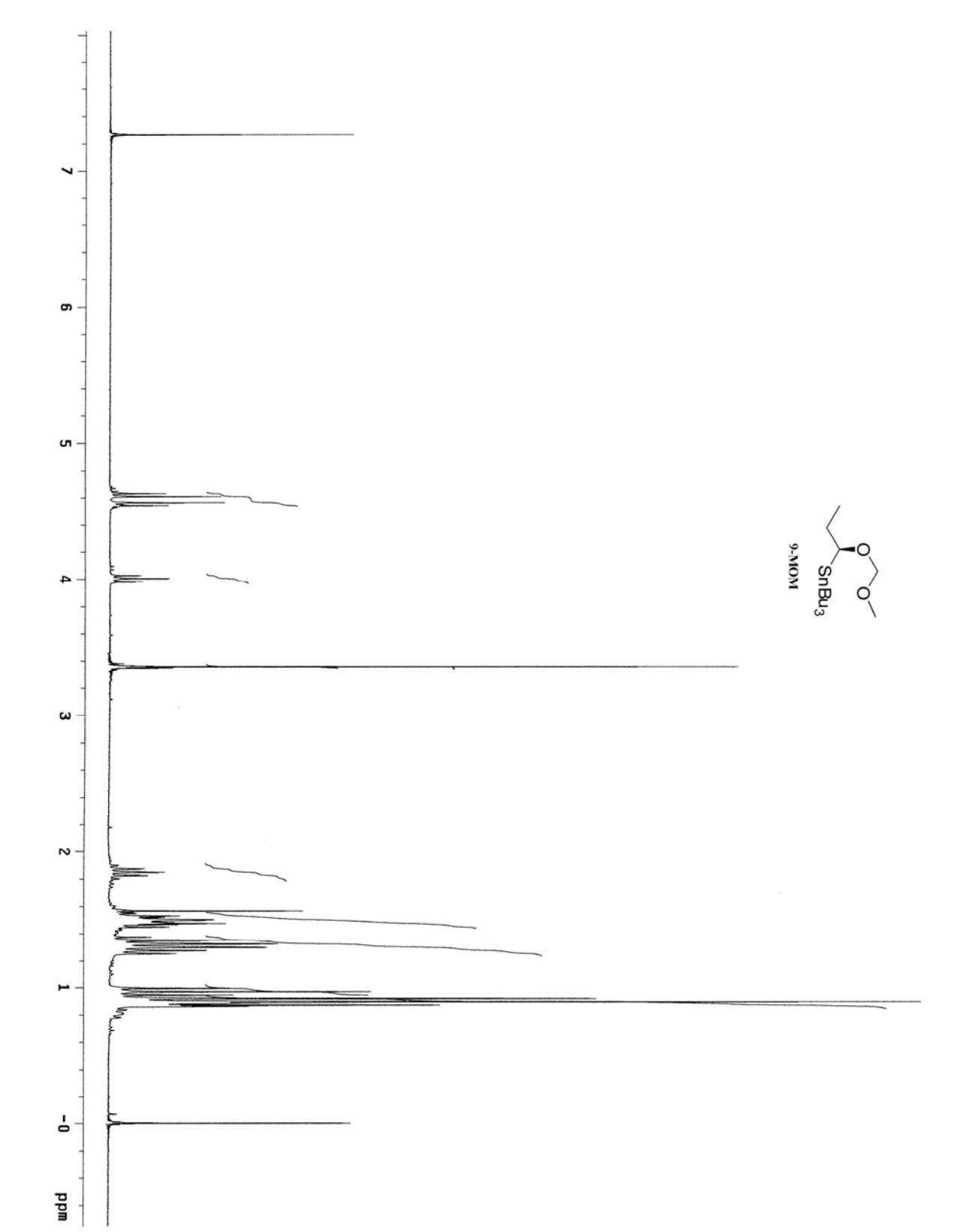
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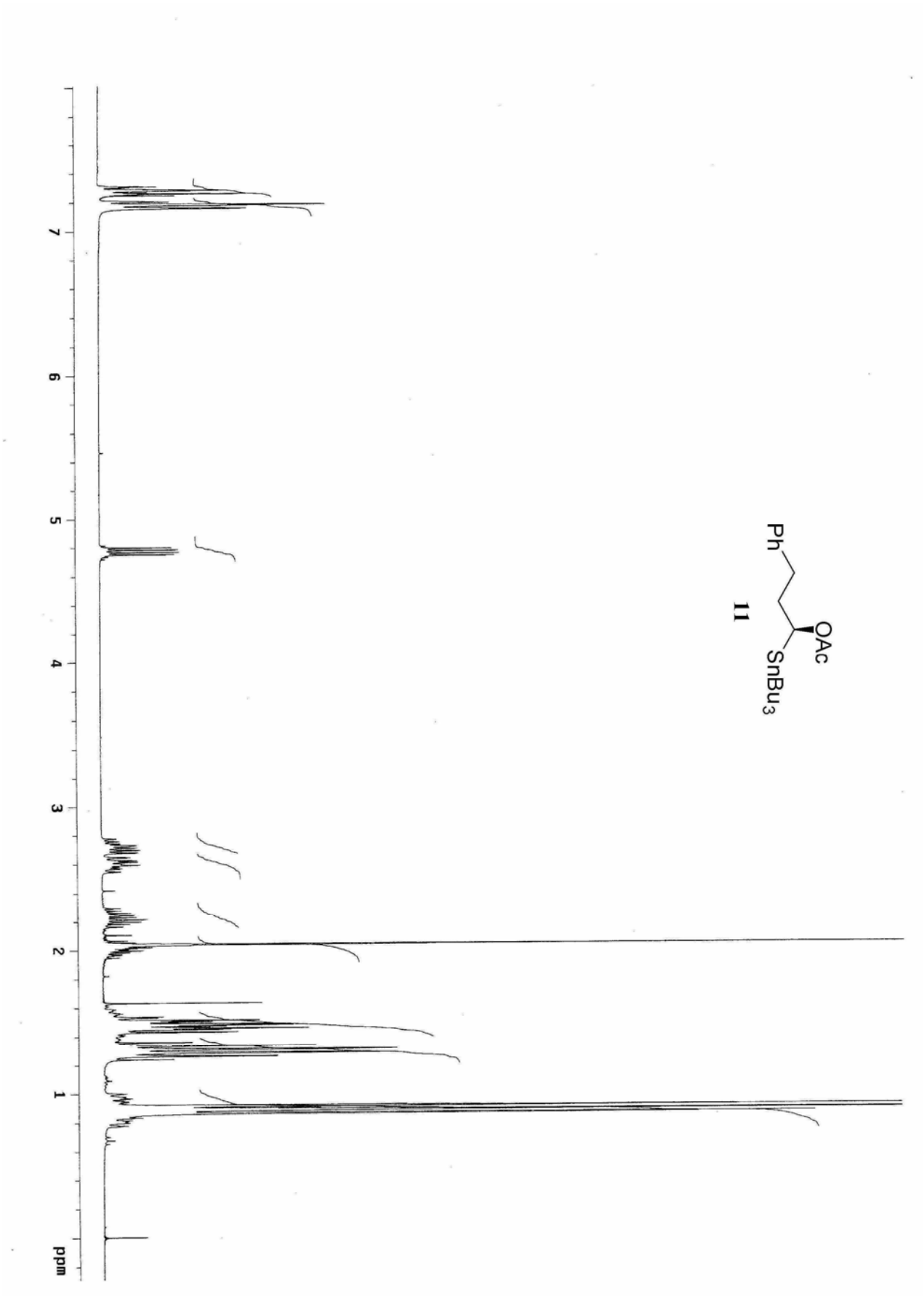
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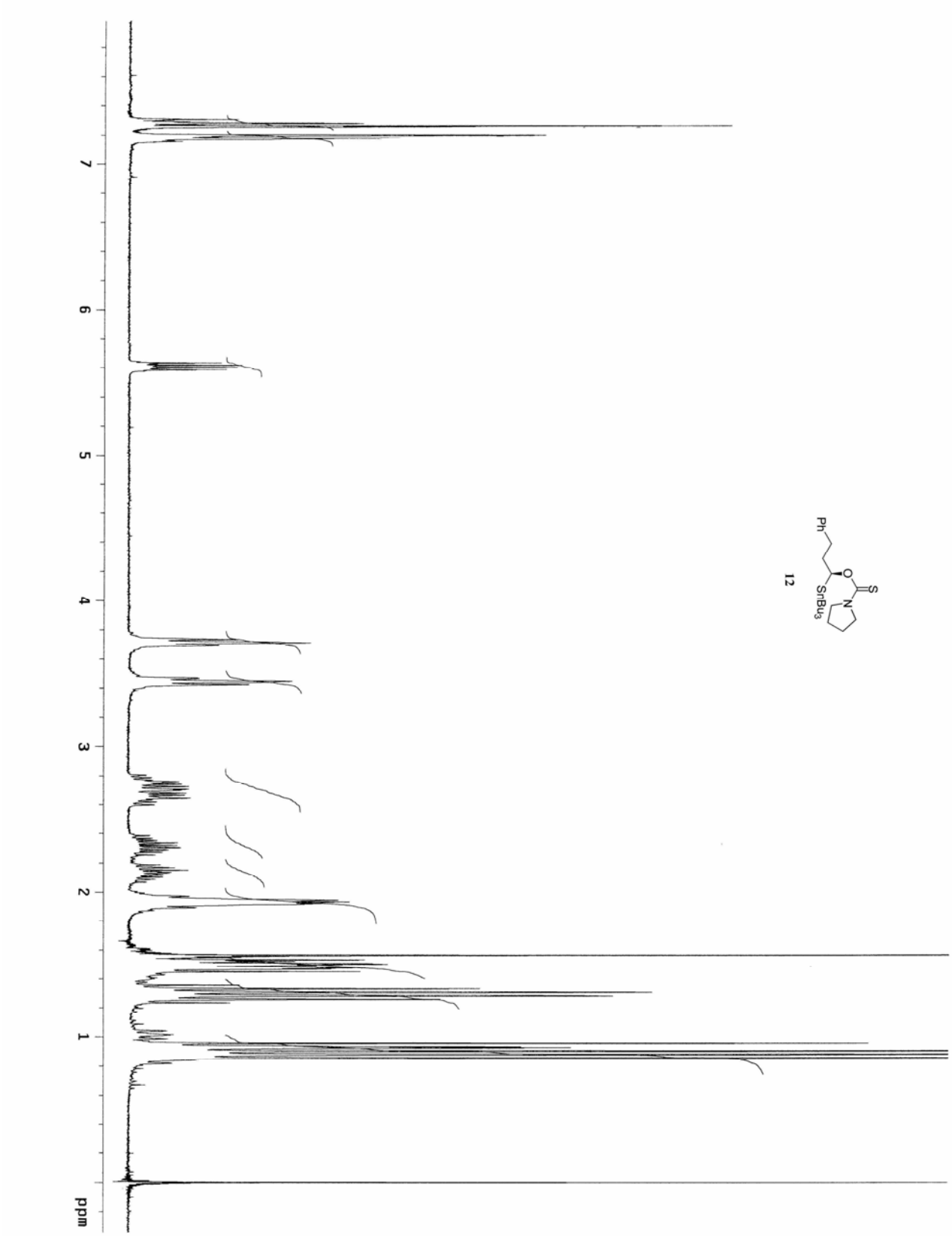
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S
N
SnBu3
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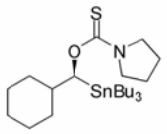


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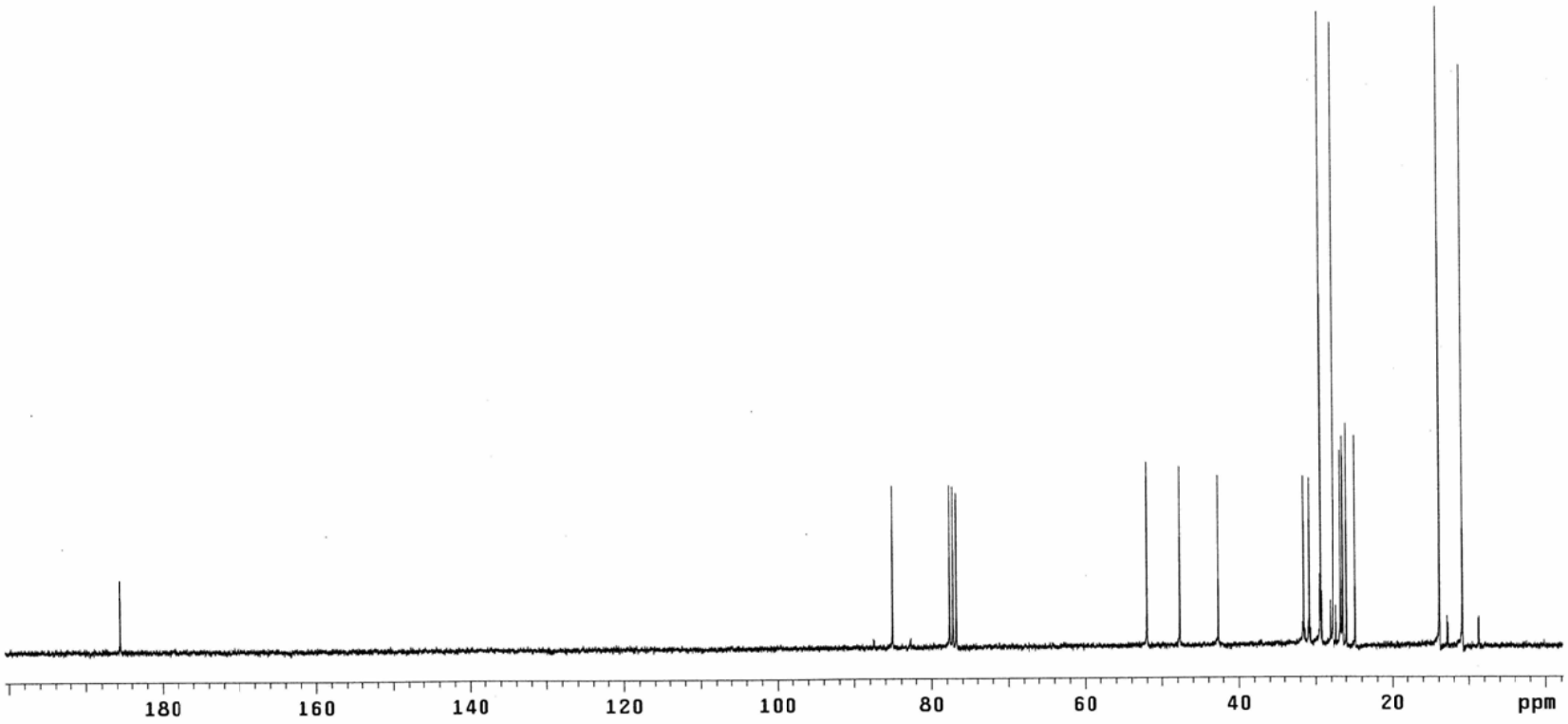

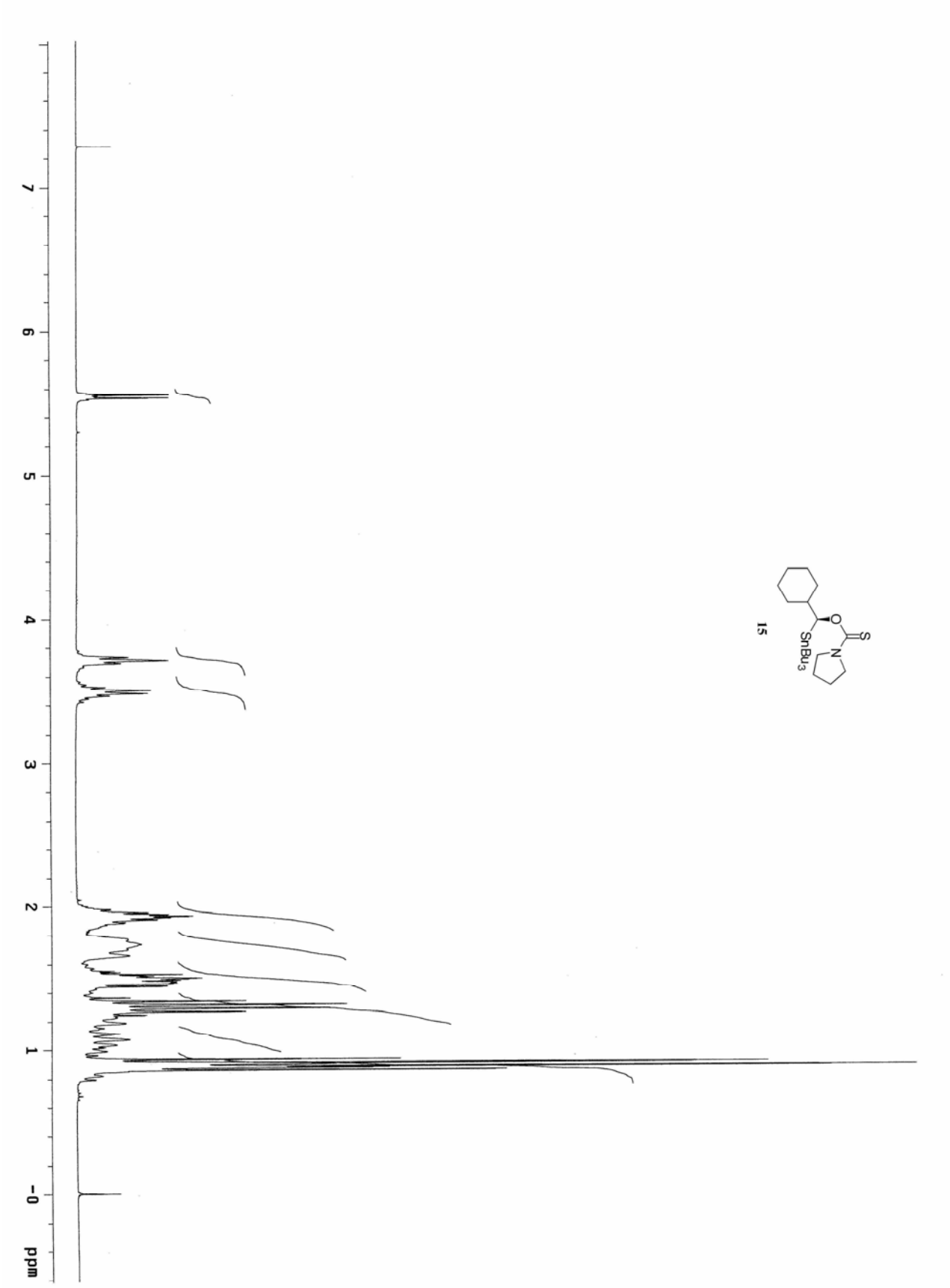
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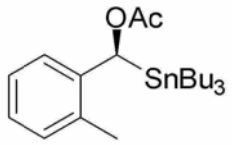
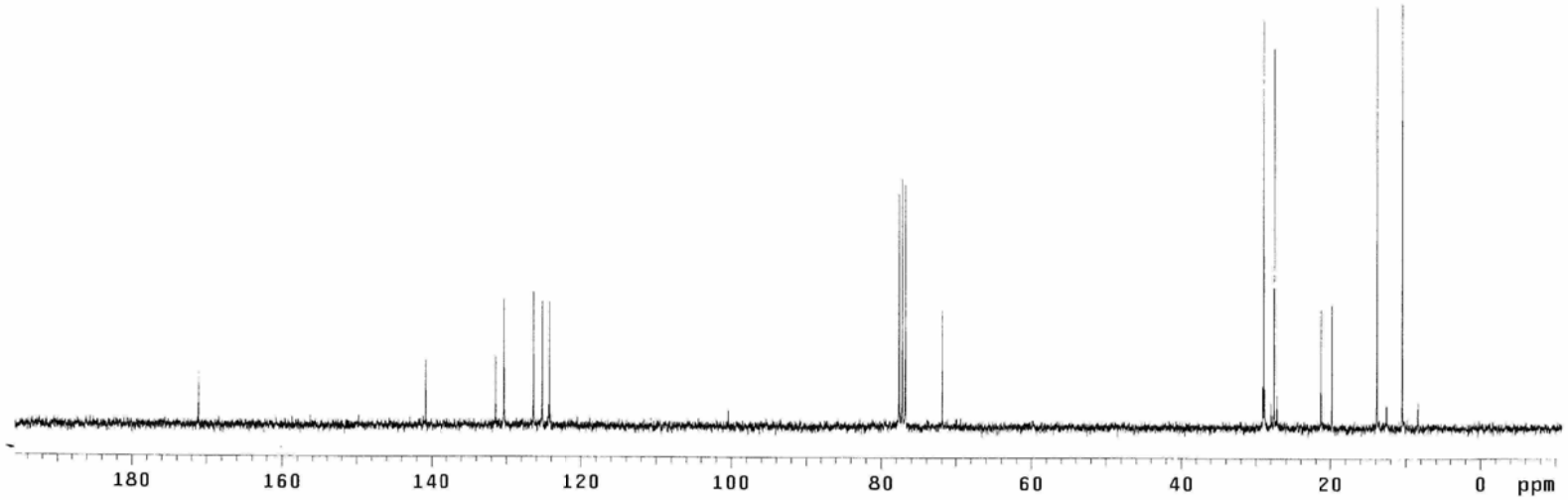
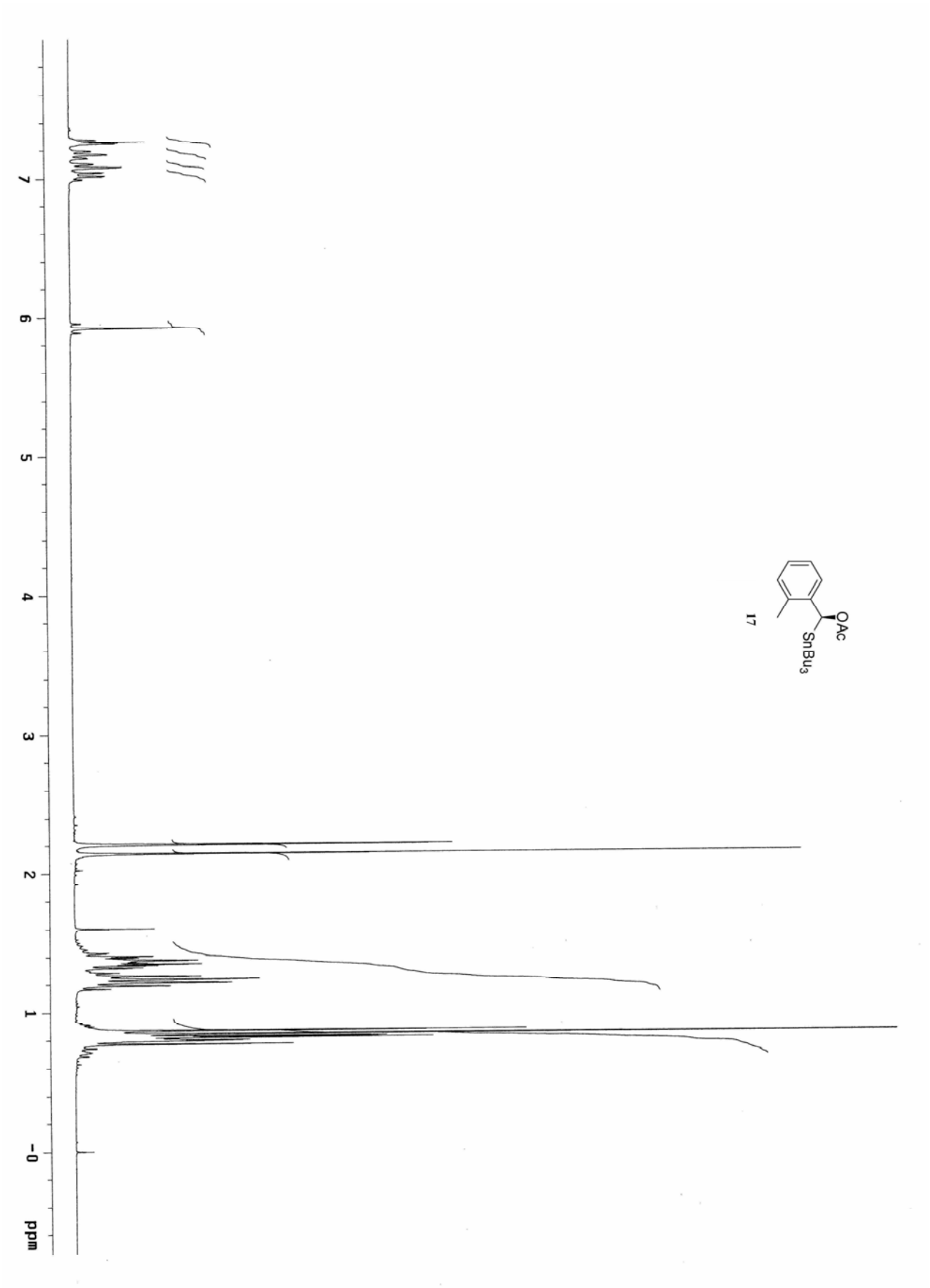
SnBu3
O
S
N




**17**

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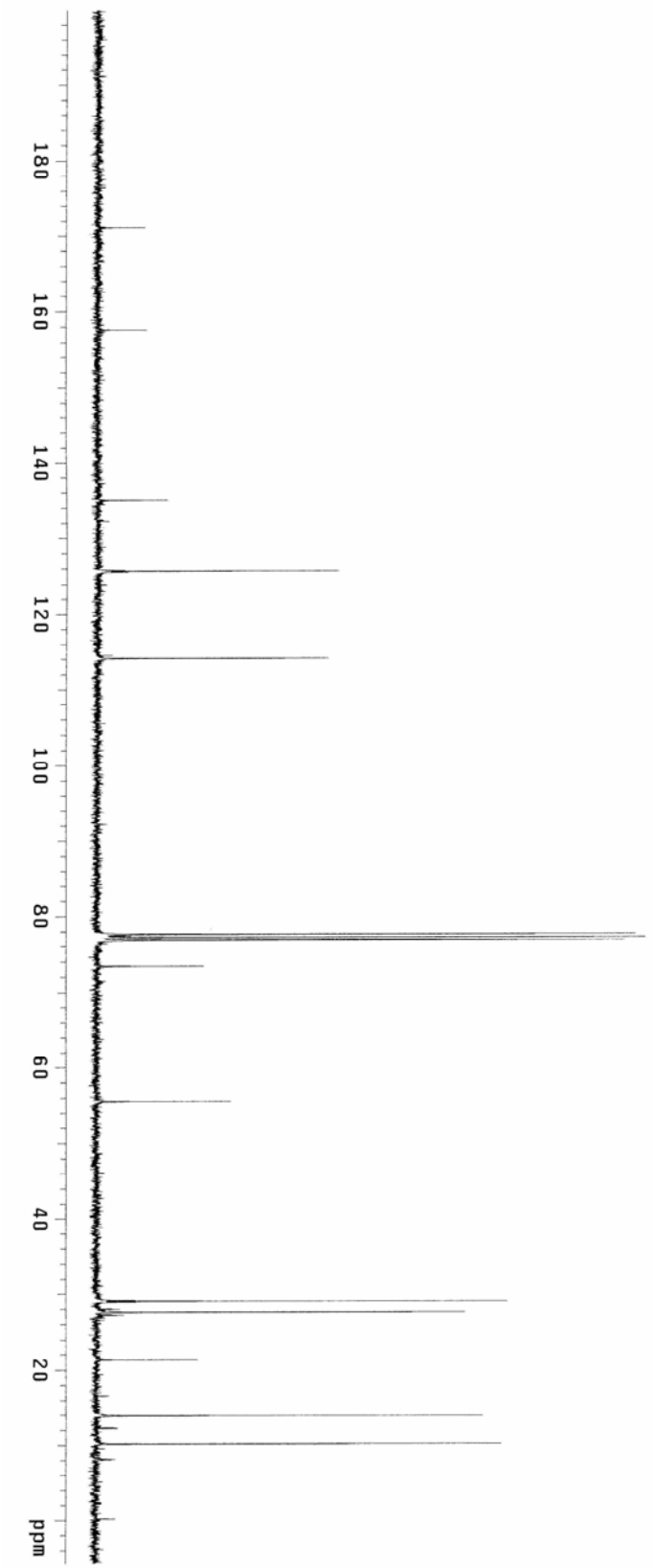


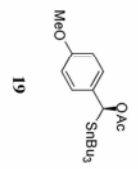


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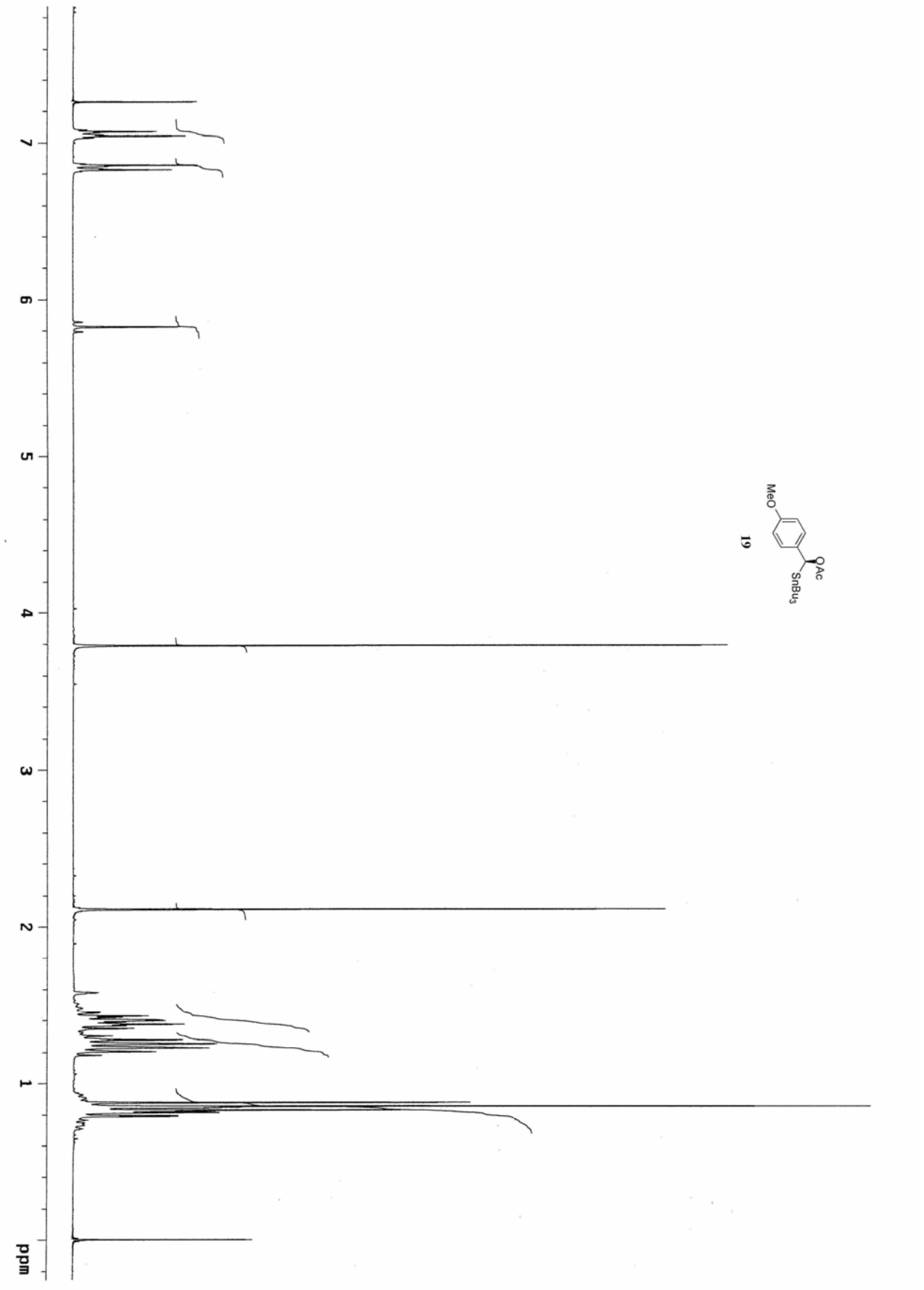
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3
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1
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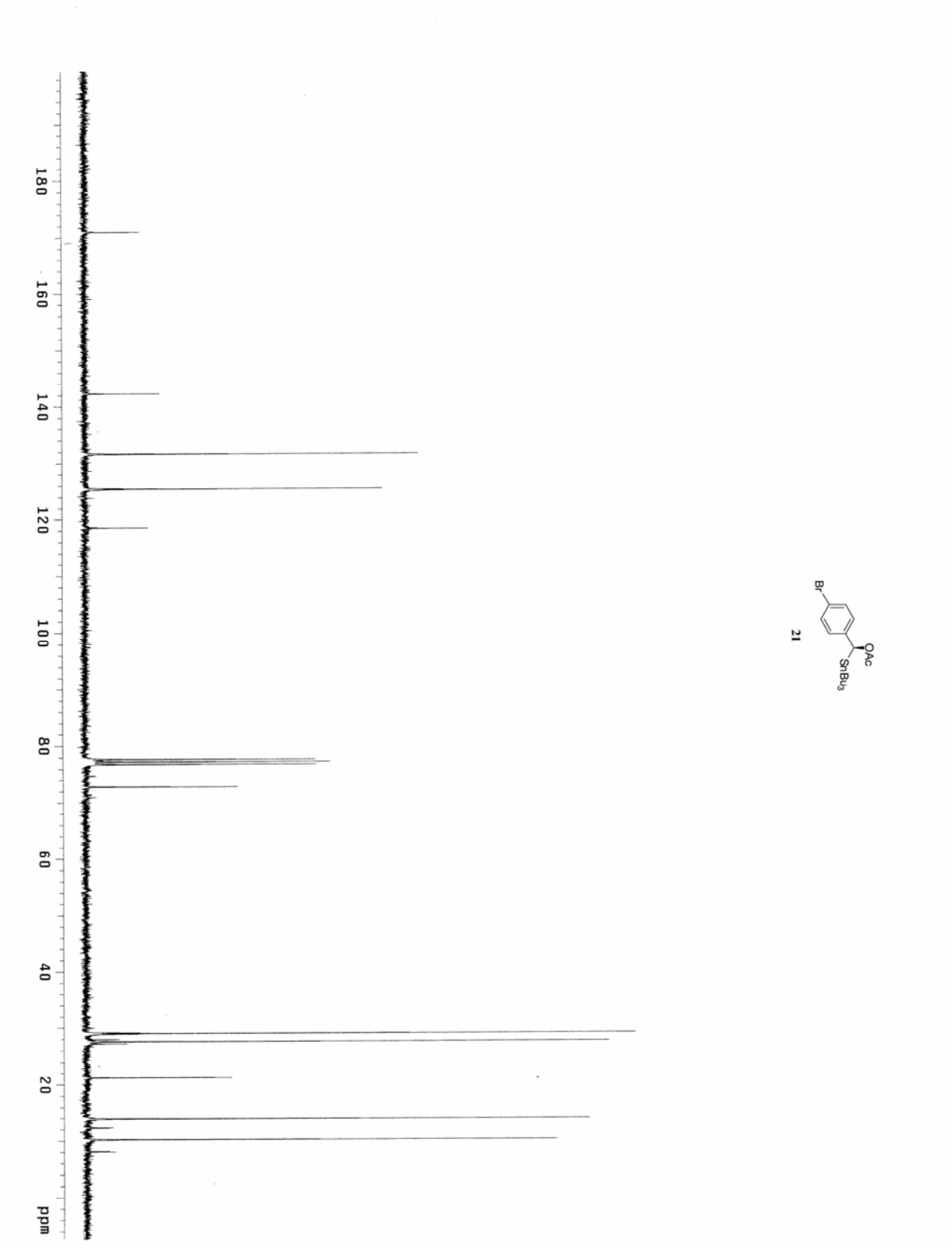
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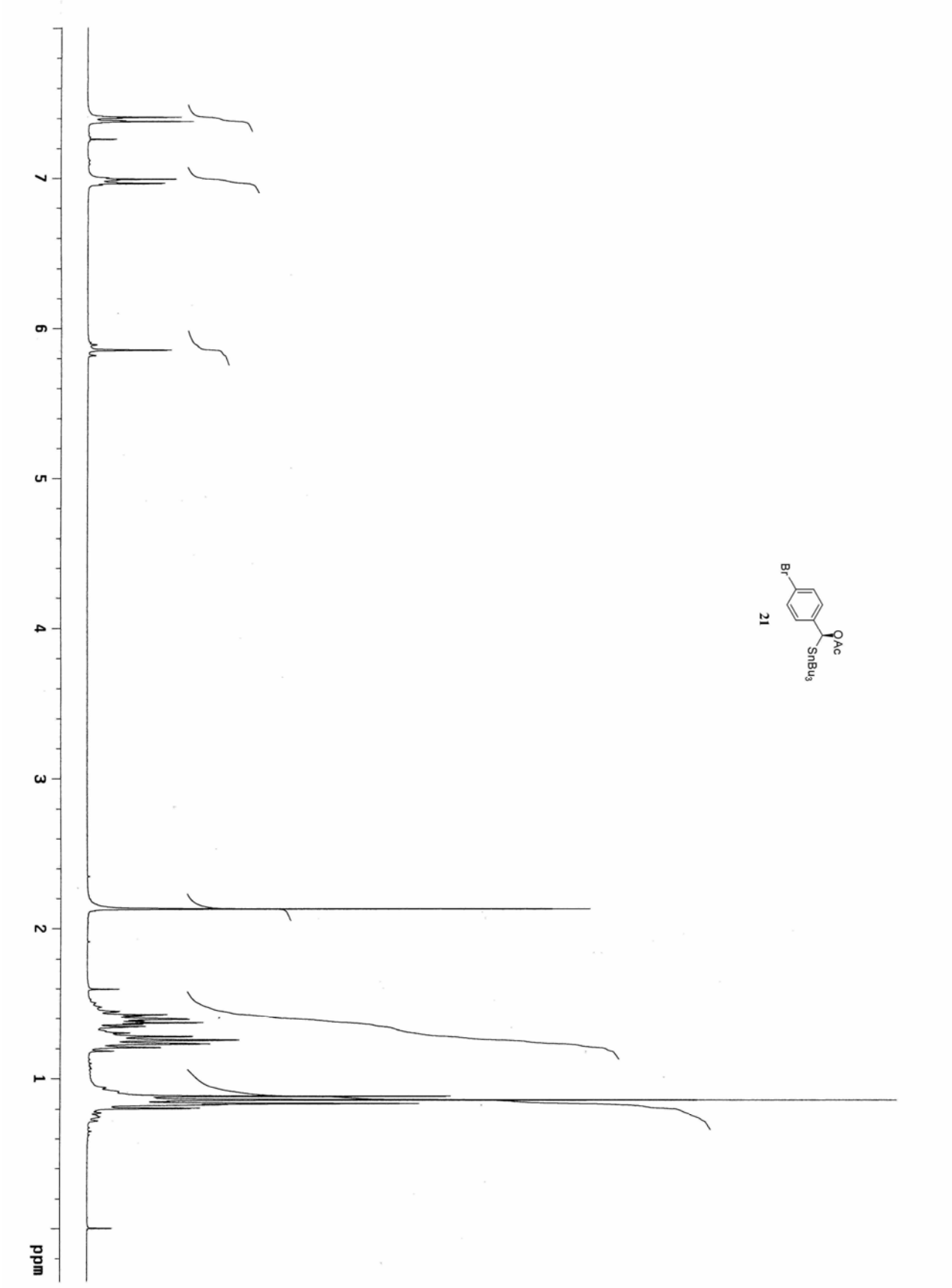
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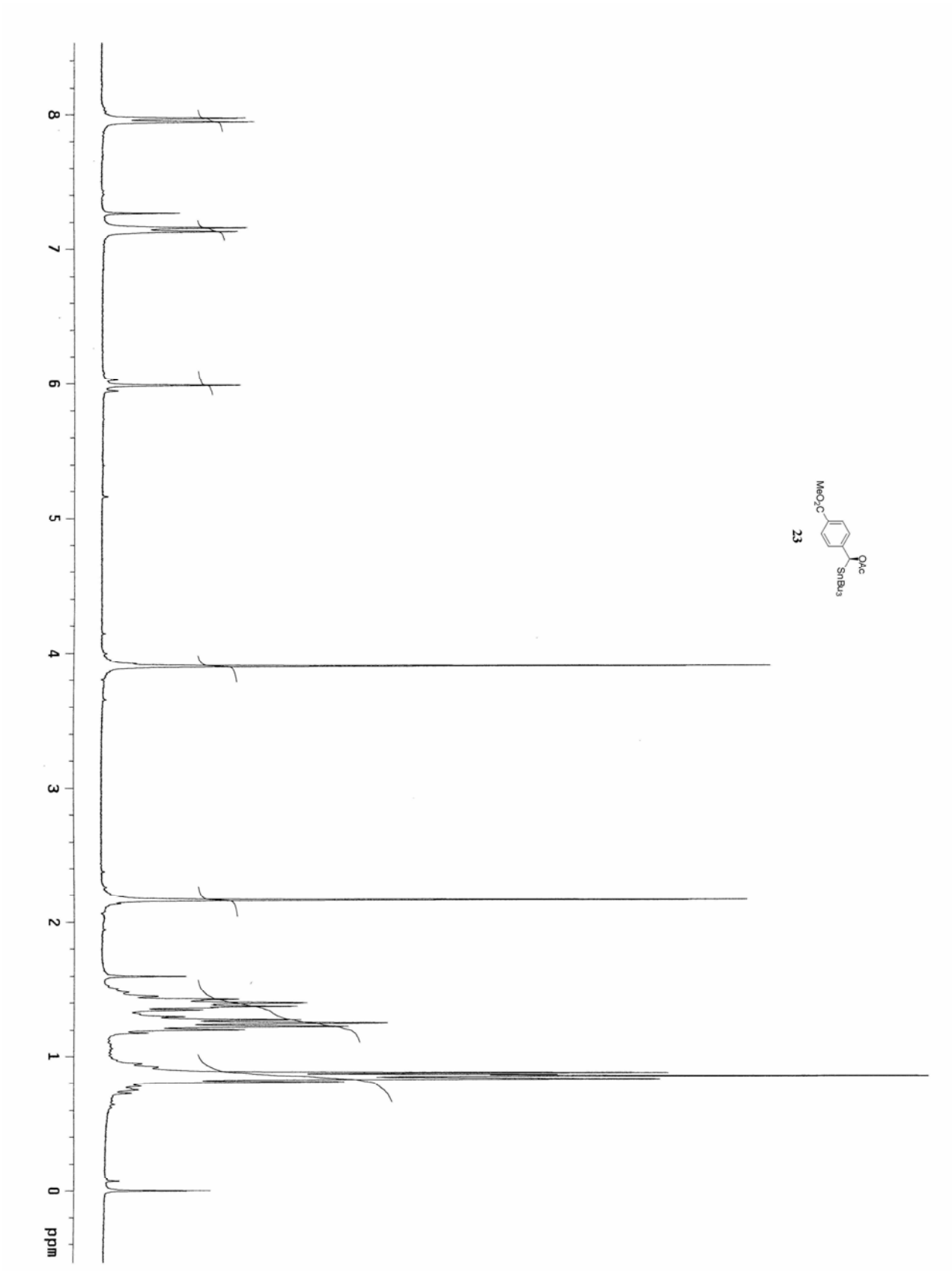
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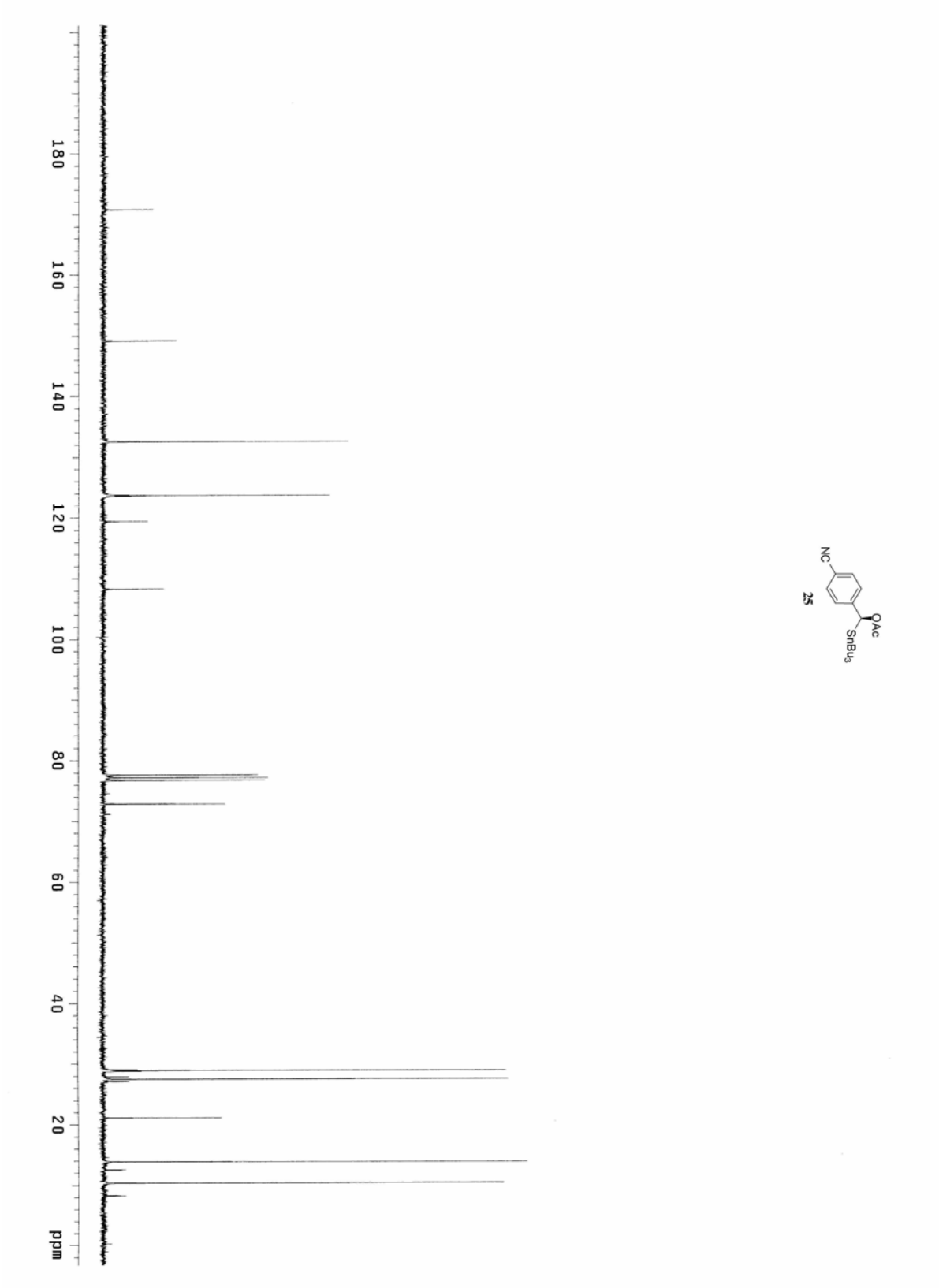
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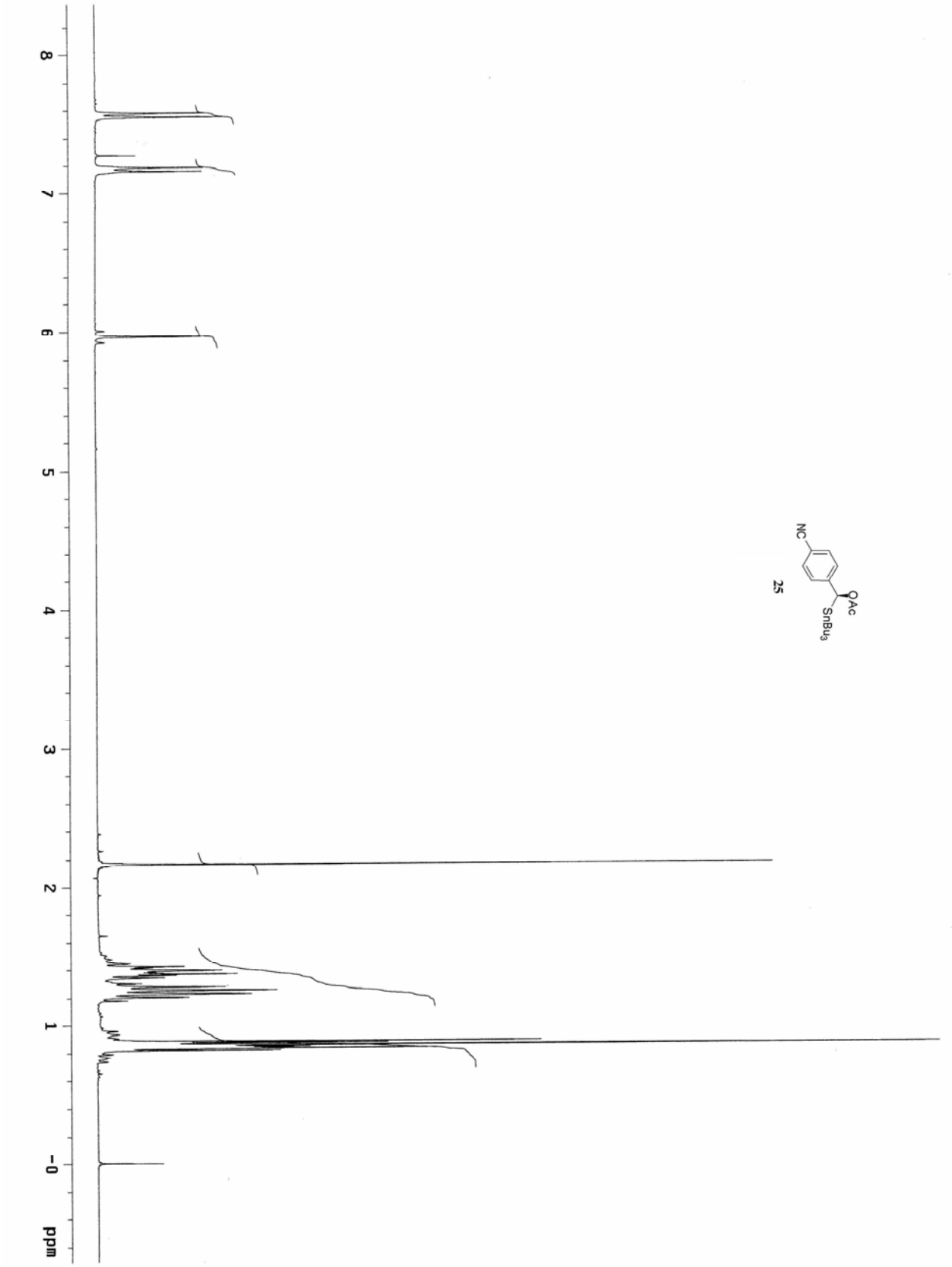
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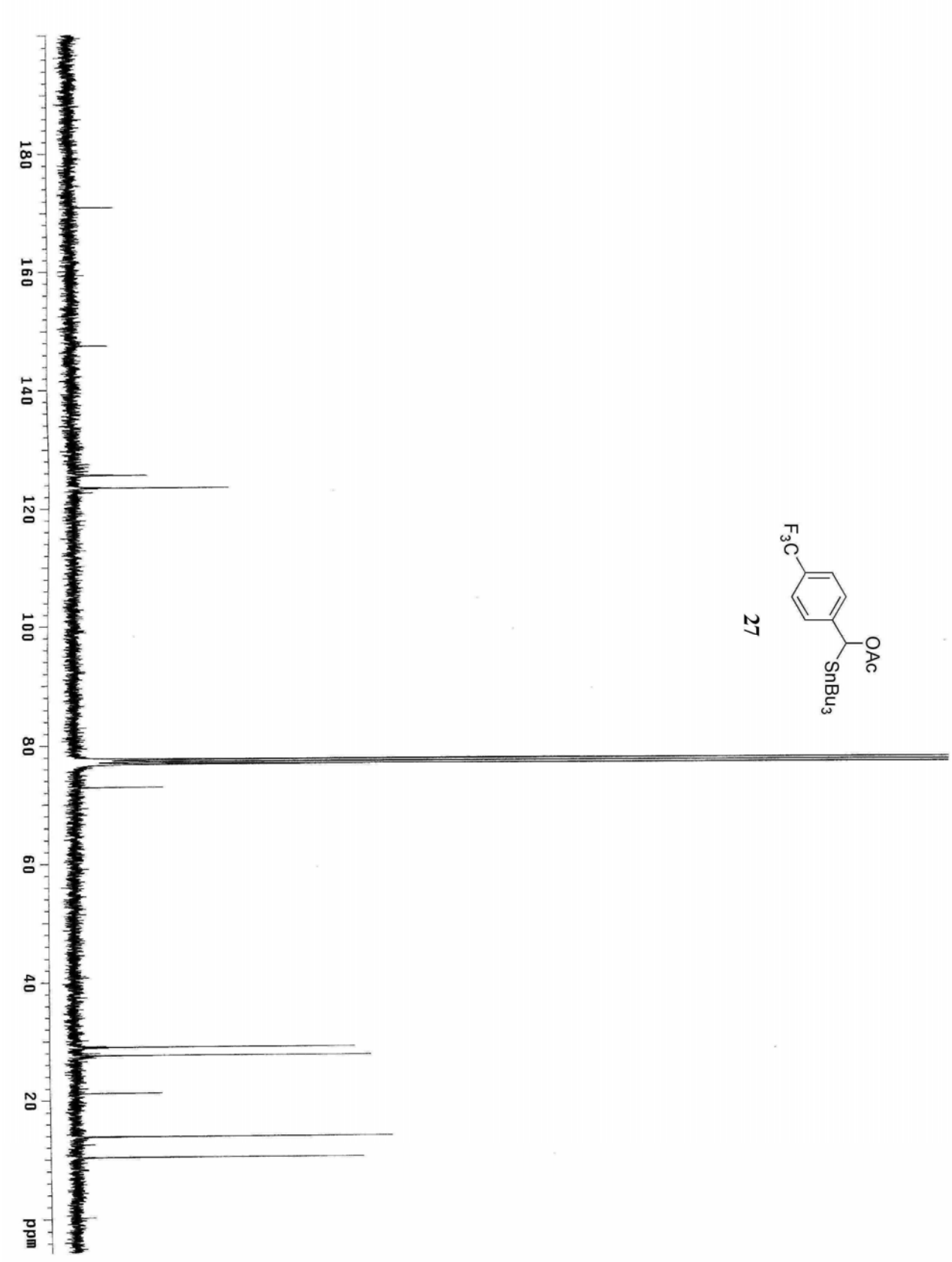
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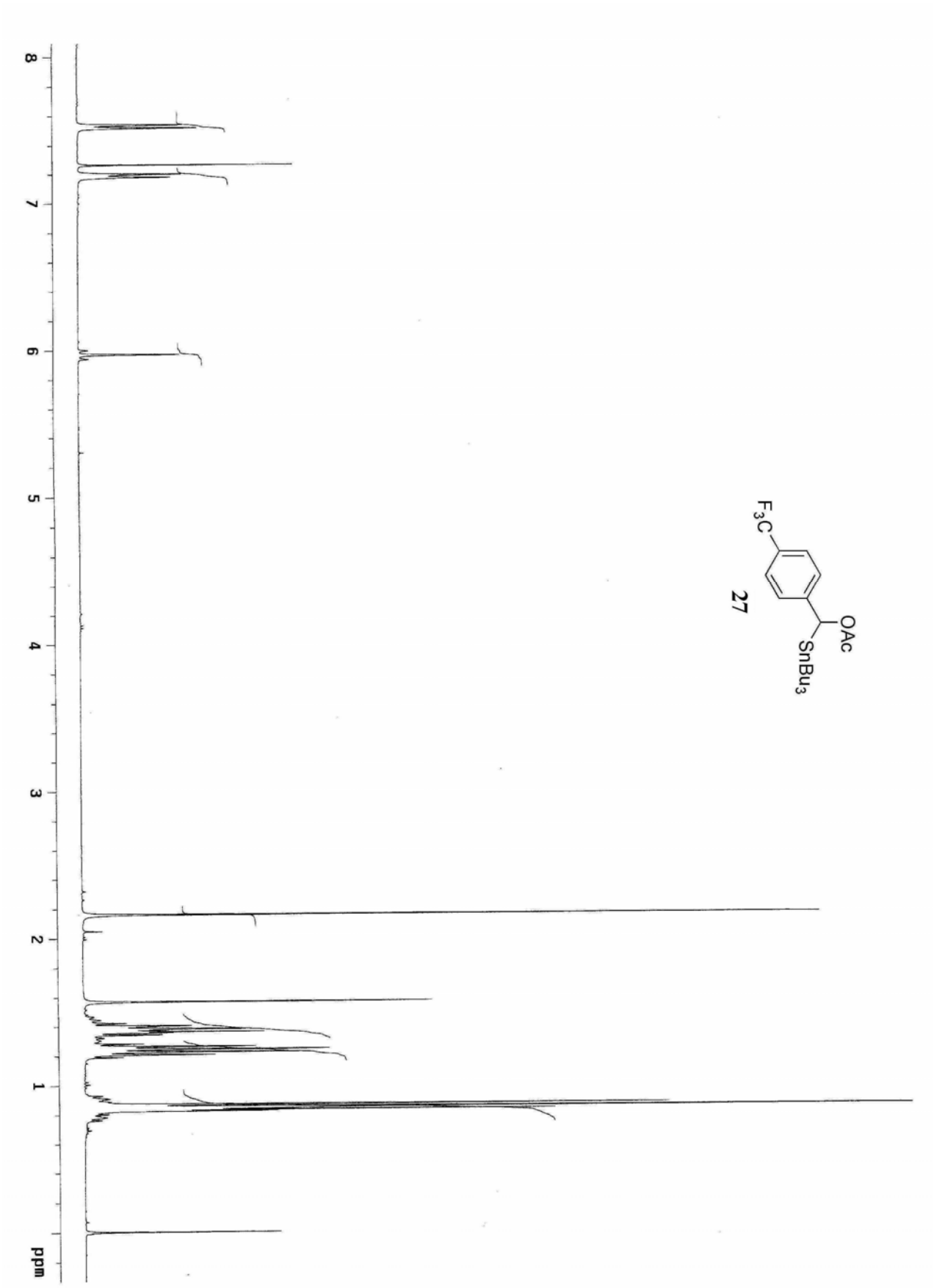
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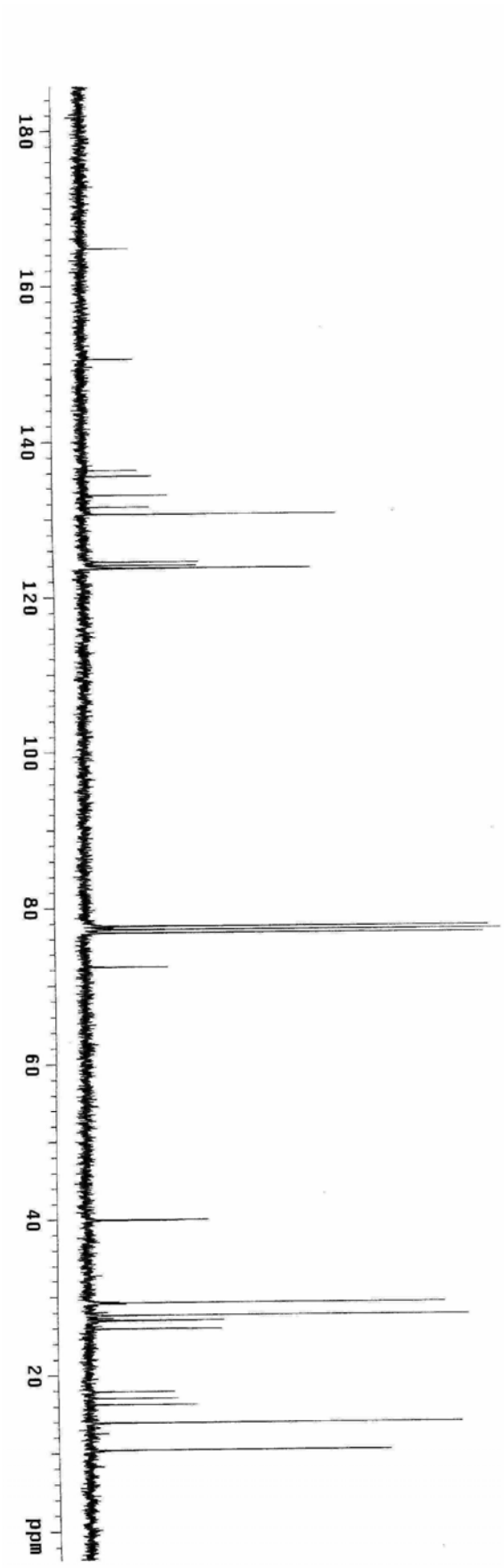


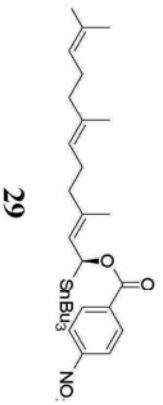


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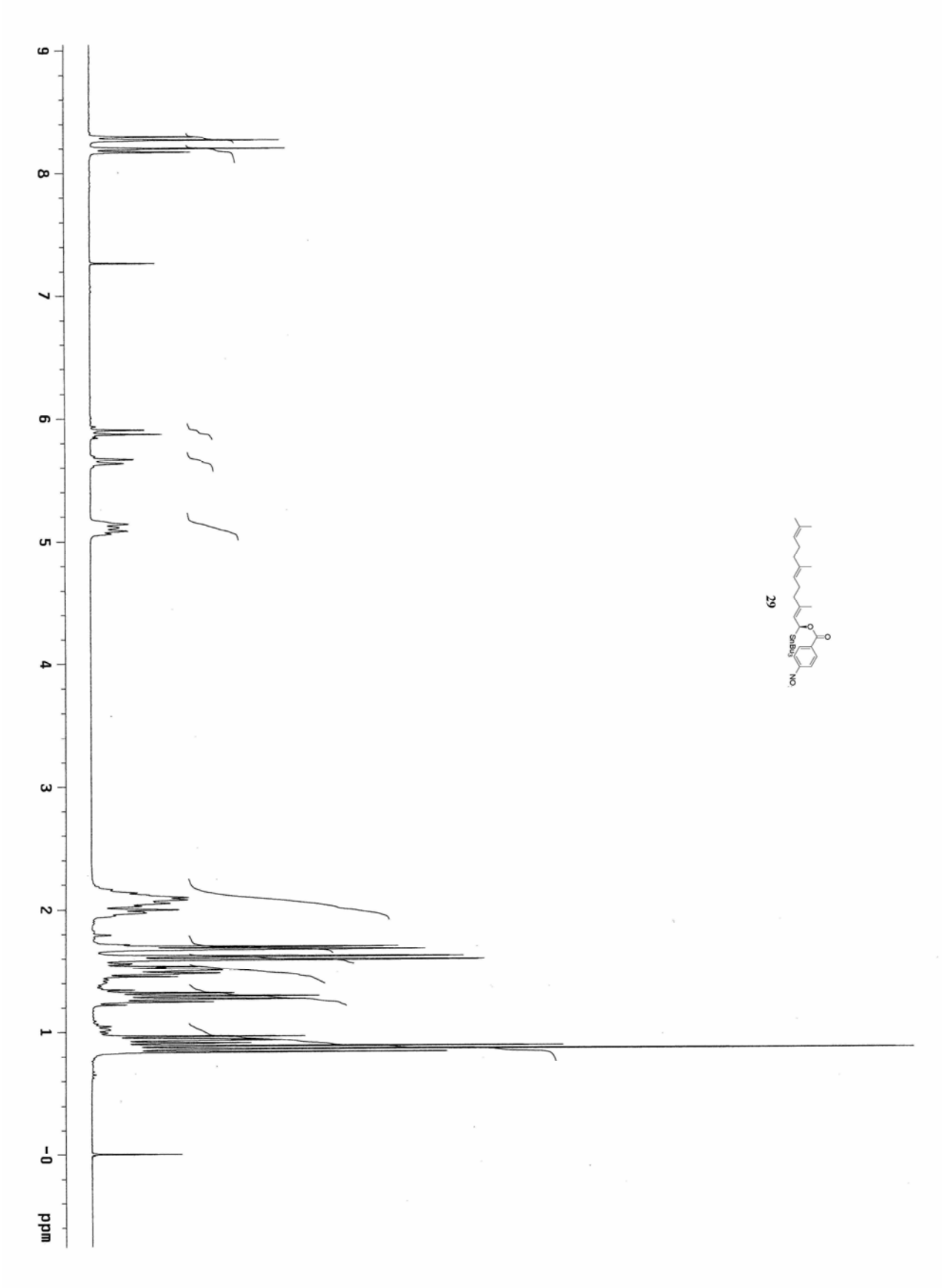
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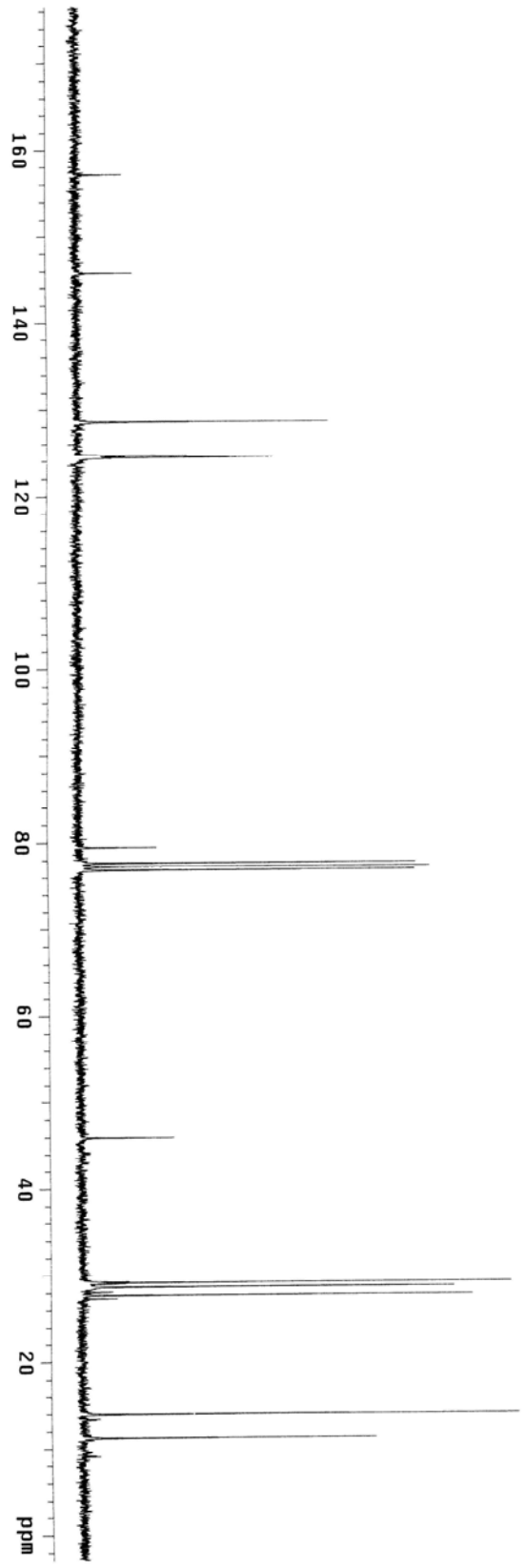
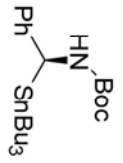
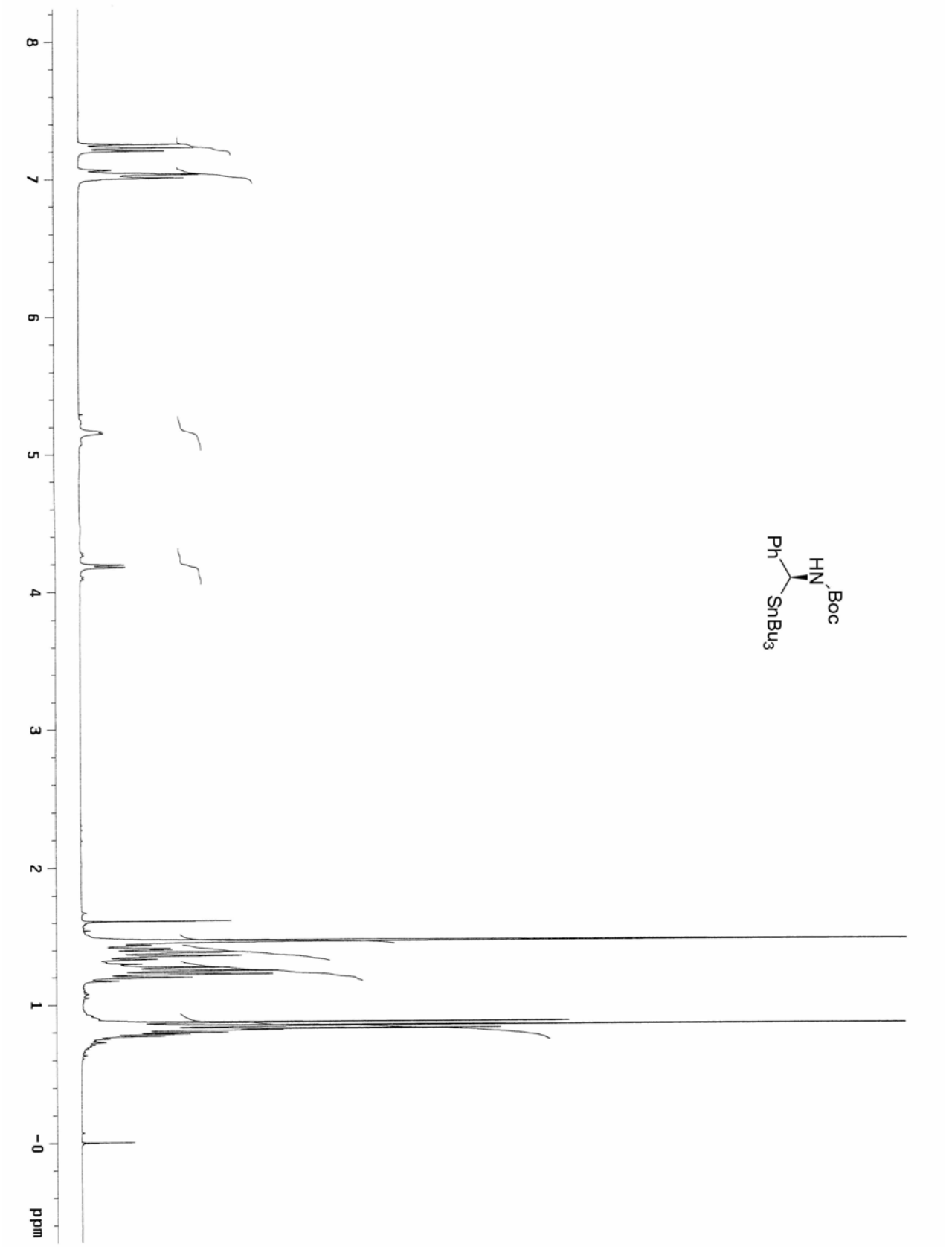
ppm
29
NO2


160
140
120
100
80
60
40
20
ppm


Ph
HN
Boc
SnBu3


8
7
6
5
4
3
2
1
0
ppm
HN
Boc
Ph
SnBu3