Supplementary Material

Synthesis of 4-alkoxypyridines as intermediates for zwitterionic liquid crystals

Anas N. Hajhussein,[a] Loay S. Abuzahra,[a] Anna Pietrzak,[a,b] Mary F. Sadek,[a] Muhammad O. Ali, [a] Jakub Wojciechowski,[a] Andrienne C. Friedli,* [a] and Piotr Kaszyński* [a,c,d]

[a] Organic Materials Research Group, Department of Chemistry, Middle Tennessee State University, Murfreesboro, TN 37132, USA

[b] Łódź University of Technology, 90 924 Łódź, Poland

[c] Centre of Molecular and Macromolecular Studies, Polish Academy of Sciences, 90-363 Łódź, Poland

[d] Faculty of Chemistry, University of Łódź, 91 403 Łódź, Poland

Email: Piotr.kaszynski@mtsu.edu

Table of Contents

1. Single crystal XRD data collection and refinement

X-Ray data collection. Single-crystal X-ray diffraction measurements for **1[13]** and **1[16]** were performed with a Rigaku XtaLAB SuperNova, Pilatus 200K diffractometer whereas for **2[13]** they were performed with Rigaku XtaLAB Synergy, Dualflex, Pilatus 300K diffractometer. The crystals were kept at 100.0(1) K during data collection. The data were integrated using CrysAlisPro program. Intensities for absorption were corrected using SCALE3 ABSPACK scaling algorithm implemented in CrysAlisPro program.[1] All structures were solved with the ShelXT[2] structure solution program using Intrinsic Phasing and refined by the full-matrix least-squares minimization on F^2 with the ShelXL[3] refinement package. All non-hydrogen atoms were refined anisotropically. C−H and B–H hydrogens were generated geometrically using the HFIX command as in ShelXL. Hydrogen atoms were refined isotropically using a riding model with U_{iso}(H) = 1.2U_{eq}(C,B).[4] The crystal data and structure refinement descriptors are presented in Table S1.

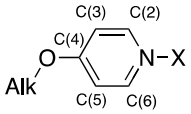
CCDC 1828911, 1857238 and 1857240 contain supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via https://www.ccdc.cam.ac.uk/structures/

A comparison of the pyridine ring geometry for free pyridines **1[13]** and **1[16]** and zwitterion **2[13]** is provided in Table S2.

Table S1. Crystal data and refinement details for **1[13]**, **1[16]** and **2[13]**

	1[13] [CCDC1857238]	**1[16]** [CCDC1828911]	**2[13]** [CCDC1857240]
Formula	$C_{18}H_{31}NO$	$C_{21}H_{37}NO$	$C_{36}H_{70}B_{10}N_2O_2$
Formula weight	277.44	319.51	671.04
Crystal system	Monoclinic	Triclinic	Triclinic
Space group	$P2_1/n$	$P\bar{1}$	$P\bar{1}$
a/Å	29.001(3)	7.4609(2)	8.6840(1)
b/Å	10.9308(9)	9.3722(3)	14.8169(2)
c/Å	5.4815(5)	14.3022(4)	18.0004(2)
α/°	90	81.184(2)	66.839(1)
β/°	100.978(11)	84.566(2)	76.644(1)
γ/°	90	85.573(3)	87.352(1)
Volume/Å^3	1705.9(3)	981.83(5)	2069.26(5)
Z	4	2	2
2ϑ range for data collection/°	6.208 to 134.132	9.57 to 135.382	10.028 to 140.146
Index ranges	-34 ≤ h ≤ 34, -13 ≤ k ≤ 8, -6 ≤ l ≤ 6	-8 ≤ h ≤ 8, -11 ≤ k ≤ 11, -17 ≤ l ≤ 17	-10 ≤ h ≤ 10, -17 ≤ k ≤ 18, -20 ≤ l ≤ 21
No. of measured, independent and observed reflections	12671, 3045, 1757 {I>2σ(I)}	21595, 3538, 3227 {I>2σ(I)}	53758, 7860, 7127 {I>2σ(I)}
R_{int}	0.067	0.031	0.059
$R[F^2>2\sigma(F^2)]$, $wR(F^2)$, S	0.076, 0.236, 1.05	0.034, 0.105, 1.09	0.041, 0.126, 1.06
No. of reflections	3045	3638	7860
No. of parameters	182	209	453
Largest diff. peak/hole/e Å^{-3}	0.29, -0.26	0.20, -0.18	0.30, -0.24

Table S2. Selected interatomic distances and angles for **1[13]**, **1[16]** and **2[13]**.



parameter	**1[16]**	**1[13]**	**2[13]** [a]	**2[13]-1[13]**	**2[13]-1[16]**
distances	*d* /Å	*d* /Å	*d* /Å	Δ*d* /Å	Δ*d* /Å
N(1)-C(2)	1.3440	1.3450	1.3540	0.009	0.010
C(2)-C(3)	1.3790	1.3750	1.3690	-0.006	-0.010
C(3)-C(4)	1.3970	1.3830	1.3970	0.014	0.000
C(4)-C(5)	1.3820	1.3910	1.3970	0.006	0.015
C(5)-C(6)	1.3890	1.3830	1.3730	-0.010	-0.016
C(6)-N(1)	1.3410	1.3340	1.3490	0.015	0.008
C(4)-O	1.3590	1.3620	1.3390	-0.023	-0.020
O-Alk	1.4430	1.4380	1.4535	0.0155	0.0105
angles	α/°	α/°	α/°	Δα/°	Δα/°
C(2)-N(1)-C(6)	115.50	115.30	118.10	2.80	2.60
C(3)-C(4)-C(5)	118.50	118.50	118.40	-0.10	-0.10

[a] Average values for two pyridinium groups.

2. General procedure and characterization details for 1[n].

General

Reactions were carried out under Ar and subsequent manipulations were conducted in air. NMR spectra were obtained at 500 MHz (^{1}H) and 125 MHz (^{13}C) fields. Chemical shifts were referenced to the solvent (^{1}H and ^{13}C: 7.26 ppm and 77.16 ppm for $CDCl_3$).[5] All reagents and solvents: 1-alkanols (**6[n]**), 4-chloropyridine hydrochloride (**5•HCl**) and DMSO, were obtained from Sigma-Aldrich and used as received. Finely divided NaOH (sand) was obtained from Loudwolf Industrial and Scientific and stored in a desiccator.

General Procedure for Synthesis of 4-Alkoxypyridines (1[n])

A 100 mL round bottom flask was flushed with argon and reagents added *via* funnel in the following order: finely divided sodium hydroxide (2.00 g, 50.0 mmol), alcohol **6[n]** (10.0 mmol), and reagent grade DMSO (12 mL). The mixture was heated with stirring to 80°C under argon and 4-chloropyridine hydrochloride (**5•HCl**, 1.50 g, 10.0 mmol) was added with DMSO rinse (8 mL). After approximately 30 minutes, an additional portion of **5•HCl** (0.300 g, 2.0 mmol) was added. The reaction was stirred and heated overnight. Water was added (20 mL) and the mixture was extracted with ethyl acetate/hexane (1:1, 20 mL, 2x). The organic layer was separated, dried (Na_2SO_4), and solvent was removed under reduced pressure. Lower members of the series **1[n]** were purified from traces of impurities and unreacted alcohol by short-path distillation ($n \leq 9$) or Kugel-Rohr distillation (n = 10, 11, 12), while higher members of the series **1[n]** (n > 12) were purified by column chromatography using silica gel with gradient EtOAc/hexanes mixtures (starting from 5% up to 25% EtOAc) as eluent. Typical yields of isolated and purified products as colorless liquids or solids are in a range 75-80%. All 4-alkoxypyridines **1[n]** exhibit the following characteristic IR vibrations: (neat) *v* 1595, 1571, 1473, 1286, 1216, 1022, 820 cm^{-1}.

4-Pentyloxypyridine 1[5]:[6] bp 72-75 °C/ 0.25 mm Hg (lit.[7] 109-110 °C/ 1 mm); ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.93 (t, *J* = 7.4 Hz, 3H), 1.35-1.48 (m, 4H), 1.80 (quint, *J* = 7.0 Hz, 2H), 3.99 (t, *J* = 6.6 Hz, 2H), 6.78 (dd, J_1 = 4.8 Hz, J_2 = 1.5 Hz, 2H), 8.40 (dd, J_1 = 4.8 Hz, J_2 =1.6 Hz, 2H) [lit.[6]]; ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.7, 22.1, 27.8, 28.3, 67.6, 110.0, 150.7, 164.8. Anal. Calcd. for $C_{10}H_{15}NO$: C, 72.69; H, 9.15; N, 8.48. Found: C, 72.44; H, 9.34; N, 8.35.

4-Hexyloxypyridine 1[6]: bp 78-80 °C/ 0.35 mm Hg; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.90 (t, *J* = 7.2 Hz, 3H), 1.25-1.40 (m, 4H), 1.45 (quint, *J* = 7.3 Hz, 2H), 1.79 (quint, *J* = 7.2 Hz, 2H), 3.99 (t, *J* = 6.6 Hz, 2H), 6.78 (dd, J_1 = 5.1 Hz, J_2 =1.7 Hz, 2H), 8.40 (dd, J_1 = 4.8 Hz, J_2 =1.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.0, 22.5, 25.6, 28.8, 31.5, 67.9, 110.3, 151.0, 165.1; EI-MS, *m/z* 179(11, M), 96(100), 95(42), 78(24, M-OR). Anal. Calcd. for $C_{11}H_{17}NO$: C, 73.70; H, 9.56; N, 7.81. Found: C, 73.24; H, 9.65; N, 7.69.

4-Heptyloxypyridine 1[7]:[8] bp 84-85 °C/ 0.25 mm Hg (lit.[8] 96-98 °C/ 0.2 mm Hg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.90 (t, *J* = 6.9 Hz, 3H), 1.45 (quint, *J* = 7.4 Hz, 2H), 1.25-1.35 (m, 6H), 1.80 (quint, *J* = 7.0 Hz, 2H), 3.99 (t, *J* = 6.6 Hz, 2H), 6.78 (dd, J_1 = 4.8 Hz, J_2 = 1.5 Hz, 2H), 8.40 (dd, J_1 = 4.8 Hz, J_2 =1.5 Hz, 2H) [lit.[6,8]]; ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.0, 22.5, 25.8, 28.8, 28.9, 31.6, 67.8, 110.2, 150.8, 165.0 [lit.[8]]; EI-MS, *m/z* 193 (1, M), 96(100), 95(40), 78(24, M-OR) [lit.[8]]. Anal. lit.[8]

4-Octyloxypyridine 1[8]:[9,10] bp 90-93 °C/ 0.35 mm Hg (lit.[9] 118-120 °C/ 0.2 mm Hg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.89 (t, *J* = 6.9 Hz, 3H), 1.24–1.40 (m, 8H), 1.45 (quint, *J* = 7.3 Hz, 2H), 1.79 (quint, *J* = 7.2 Hz, 2H), 3.99 (t, *J* = 6.6 Hz, 2H), 6.78 (dd, J_1 = 4.6 Hz, J_2 = 1.5 Hz, 2H), 8.40 (dd, J_1 = 4.8 Hz, J_2 =1.5 Hz, 2H) [lit.[10]]; ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.1, 22.7, 25.9, 28.9, 29.2, 29.3, 31.8, 67.9, 110.2, 151.0, 165.1 [lit.[10]]; EI-MS, *m/z* 207 (0.7, M), 96(100), 95(56), 78(22, M-OR). Anal. Calcd. for $C_{13}H_{21}NO$: C, 75.32; H, 10.21; N, 6.76. Found: C, 75.04; H, 10.48; N, 6.69. [lit. [9,10]]

4-Nonyloxypyridine 1[9]:[6] bp 103-105 °C/ 0.35 mm Hg (lit.[6] 125 °C/ 0.5 mm Hg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.88 (t, *J* = 7.2 Hz, 3H), 1.24-1.37 (m, 10H), 1.45 (quint, *J* = 7.4 Hz, 2H), 1.80 (quint, *J* = 7.0 Hz, 2H), 3.99 (t, *J* = 6.6 Hz, 2H), 6.78 (dd, J_1 = 4.8 Hz, J_2 = 1.5 Hz, 2H), 8.40 (dd, J_1 = 4.8 Hz, J_2 =1.5 Hz, 2H) [lit.[6]]; ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.0, 22.6, 25.8, 28.8, 29.15, 29.24, 29.4, 31.8, 67.8, 110.2, 150.9, 165.0; EI-MS, *m/z* 221 (0.7, M), 96(100), 95(73), 78(22, M-OR). Anal. lit.[6]

4-Decyloxypyridine 1[10]:[11,12] isolated by Kugel-Rohr distillation: 125-130 °C/ 1.5 mm Hg; mp 30-32 °C [lit.[11] mp 30 °C]; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.89 (t, *J* = 6.9 Hz, 3H), 1.24-1.34 (m, 12H), 1.45 (quint, *J* = 7.3 Hz, 2H), 1.79 (quint, *J* = 7.0 Hz, 2H), 3.99 (t, *J* = 6.6 Hz, 2H), 6.78 (dd, J_1 = 4.6 Hz, J_2 = 1.7 Hz, 2H), 8.40 (dd, J_1 = 4.8 Hz, J_2 =1.5 Hz, 2H) [lit.[12]]; ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.2, 22.8, 26.0, 29.0, 29.4, 29.62, 29.67, 29.69, 32.0, 68.0, 110.4, 151.1, 165.2 [lit.[12]]; EI-MS, *m/z* 235 (0.5, M), 96(100), 95(72), 78(19, M-OR) [lit.[12]]. Anal. Calcd. for $C_{15}H_{25}NO$: C, 76.55; H, 10.71; N, 5.95. Found: C, 76.30; H, 11.03; N, 5.76.

4-Undecyloxypyridine 1[11]: bp 106-108 °C/ 0.25 mm Hg; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.88 (t, *J* = 7.2 Hz, 3H), 1.24-1.36 (m, 14H), 1.45 (quint, *J* = 7.5 Hz, 2H), 1.79 (quint, *J* = 7.2 Hz, 2H), 4.00 (t, *J* = 6.6 Hz, 2H), 6.79 (dd, J_1 = 4.6 Hz, J_2 = 1.7 Hz, 2H), 8.40 (dd, J_1 = 4.7 Hz, J_2 = 1.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.0, 22.6, 25.8, 28.8, 29.2, 29.42, 29.47, 29.49(2C), 31.8, 67.7, 110.1, 150.8, 164.9; EI-MS, *m/z* EI-MS, *m/z* 249 (0.5, M), 96(100), 95(72), 78(15, M-OR). Anal. Calcd. for $C_{16}H_{27}NO$: C, 77.06; H, 10.91; N, 5.62. Found: C, 77.06; H, 11.17; N, 5.69.

4-Dodecyloxypyridine 1[12]:[10] mp 34-36 °C (lit.[13] mp 35-36 °C); ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.88 (t, *J* = 7.2 Hz, 3H), 1.24-1.38 (m, 16H), 1.45 (quint, *J* = 7.4 Hz, 2H), 1.79 (quint, *J* = 7.0 Hz, 2H), 4.00 (t, *J* = 6.6 Hz, 2H), 6.79 (dd, J_1 = 4.8 Hz, J_2 = 1.6 Hz, 2H), 8.41 (dd, J_1 = 4.8 Hz, J_2 = 1.5 Hz, 2H) [lit.[10]]; ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.2, 22.8, 26.0, 29.0, 29.42, 29.44, 29.63, 29.67, 29.73, 29.74, 32.0, 68.0, 110.3, 151.1, 165.2 [lit.[10]]; EI-MS, *m/z* 263 (0.3, M), 96(100), 95(63), 78(13, M-OR). Anal. Lit.[10]

4-Tridecyloxypyridine 1[13]: mp 34-36 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.9 Hz, 3H), 1.24-1.35 (m, 18H), 1.45 (quint, *J* = 7.4 Hz, 2H), 1.79 (quint, *J* = 7.2 Hz, 2H), 3.99 (t, *J* = 6.6 Hz, 2H), 6.79 (dd, J_1 = 4.8 Hz, J_2 = 1.5 Hz, 2H), 8.40-8.42 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.2, 22.8, 26.0, 28.9, 29.38, 29.42, 29.60, 29.62, 29.71(2C), 29.74, 32.0, 67.9, 110.3, 151.1, 165.1. Anal. Calcd. for $C_{18}H_{31}NO$: C, 77.92; H, 11.26; N, 5.05. Found: C, 77.86; H, 11.40; N, 4.97.

4-Tetradecyloxypyridine 1[14]: mp 38-41 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.9 Hz, 3H), 1.24-1.35 (m, 20H), 1.45 (quint, *J* = 7.5 Hz, 3H), 1.79 (quint, *J* = 7.2 Hz, 2H), 3.99 (t, *J* = 6.6 Hz, 2H), 6.79 (dd, J_1 = 4.8 Hz, J_2 = 1.4 Hz, 2H), 8.40-8.42 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.2, 22.8, 26.0, 28.9, 29.39, 29.44, 29.61, 29.65, 29.73(3C), 32.0, 68.0, 110.43 151.1, 165.1; EI-MS, *m/z* 291 (0.3, M), 96(100), 95(52), 78(13, M-OR). Anal. Calcd. for $C_{19}H_{33}NO$: C, 78.29; H, 11.41; N, 4.81. Found: C, 78.57; H, 11.51; N, 4.70.

4-Hexadecyloxypyridine 1[16]:[10] mp 53-55 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.6 Hz, 3H), 1.20-1.35 (m, 26H), 1.45 (quint, *J* = 7.3 Hz, 2H), 1.79 (quint, *J* = 7.0 Hz, 2H), 4.00 (t, *J* = 6.6 Hz, 2H), 6.79 (dd, J_1 = 4.6 Hz, J_2 = 1.7 Hz, 2H), 8.41 (dd, J_1 = 4.8 Hz, J_2 = 1.5 Hz, 2H) [lit.[10,14]]; ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.2, 22.8, 26.0, 29.0, 29.42, 29.47, 29.64, 29.68, 29.77, 29.79 (5C), 32.0, 68.0, 110.4, 151.1, 165.1 [lit.[10]]; EI-MS, *m/z* 319 (0.3, M), 96(100), 95(44), 78(7, M-OR). Anal. lit.[10]

4-Octadecyloxypyridine 1[18]: mp 57-60 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.9 Hz, 3H), 1.24-1.35 (m, 26H), 1.45 (quint, *J* = 7.3 Hz, 2H), 1.79 (quint, *J* = 7.2 Hz, 2H), 3.99 (t, *J* = 6.6 Hz, 2H), 6.78 (dd, J_1 = 4.8 Hz, J_2 = 1.5 Hz, 2H), 8.41 (dd, J_1 = 4.8 Hz, J_2 = 1.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.2, 22.8, 26.0, 29.0, 29.42, 29.46, 29.63, 29.67, 29.77, 29.80(7C), 32.0, 67.9, 110.3, 151.1, 165.1; EI-MS, *m/z* 347 (0.4, M), 96(100), 95(39), 78(5, M-OR). Anal. Calcd. for $C_{23}H_{41}NO$: C, 79.48; H, 11.89; N, 4.03. Found: C, 79.46; H, 12.19; N, 3.97.

3. ^{1}H and ^{13}C NMR spectra for 1[n]

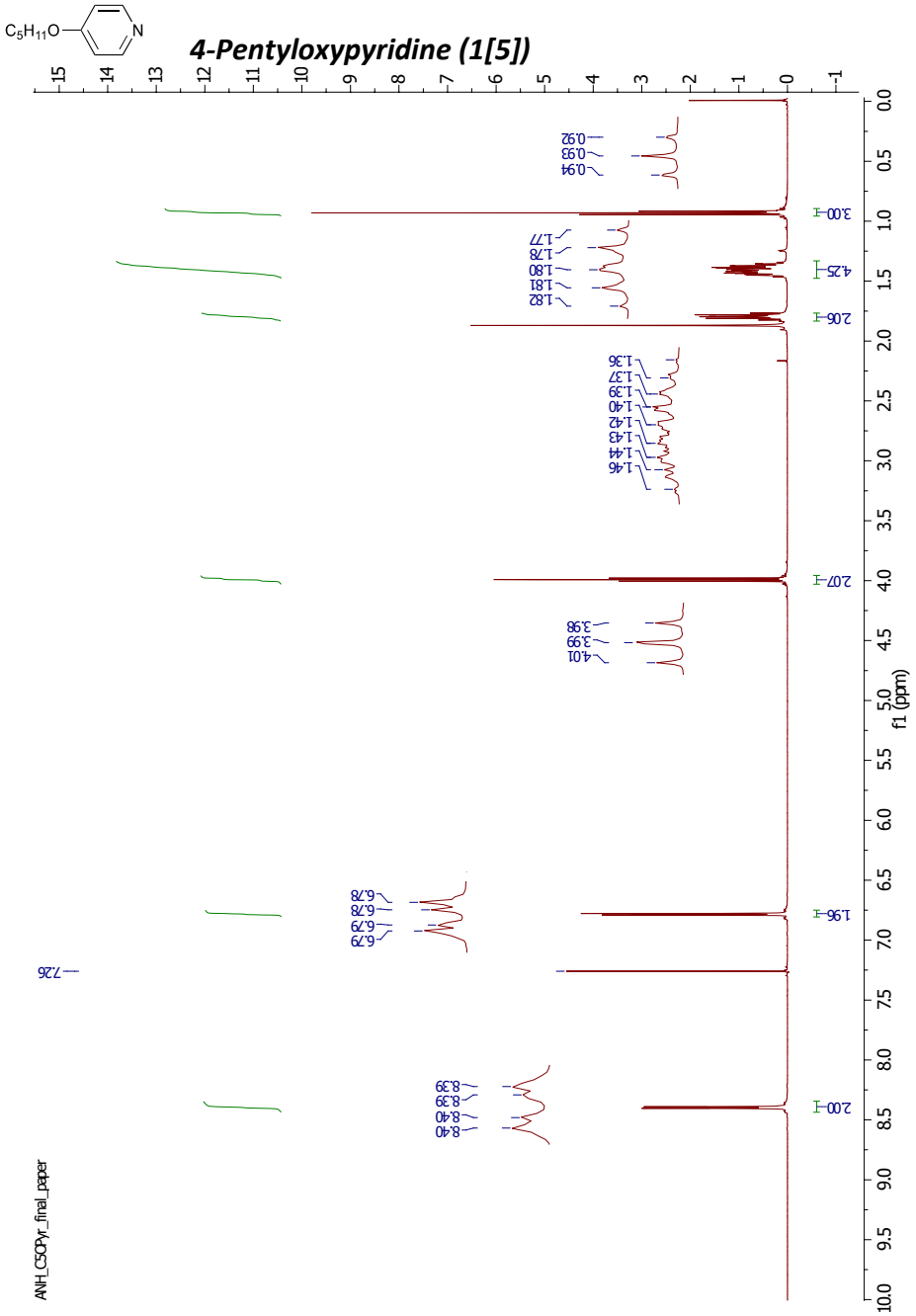


Figure S1. ^{1}H NMR of **1[5]** ($CDCl_3$, 500 MHz).

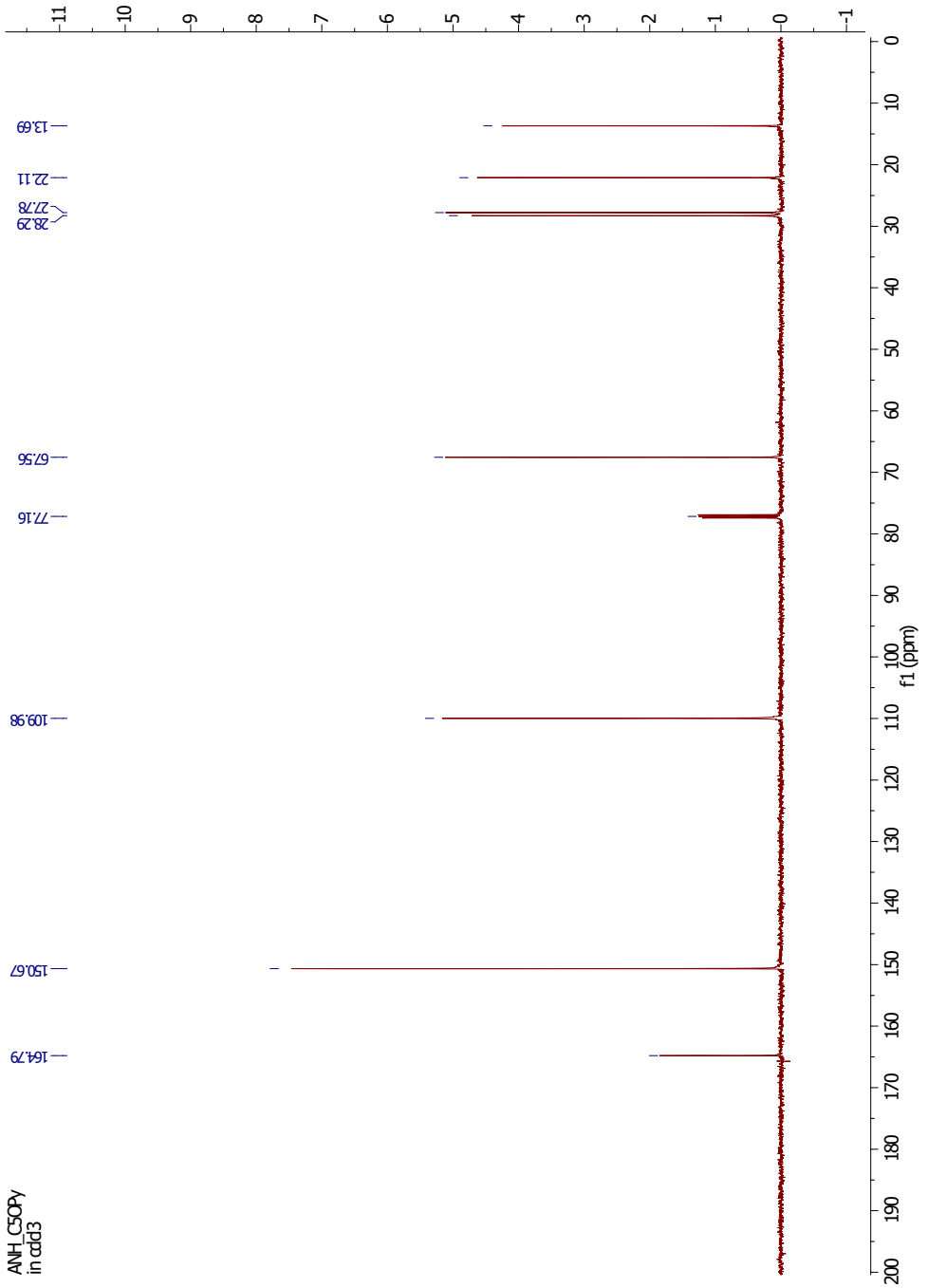


Figure S2. ^{13}C NMR of **1[5]** ($CDCl_3$, 125 MHz).

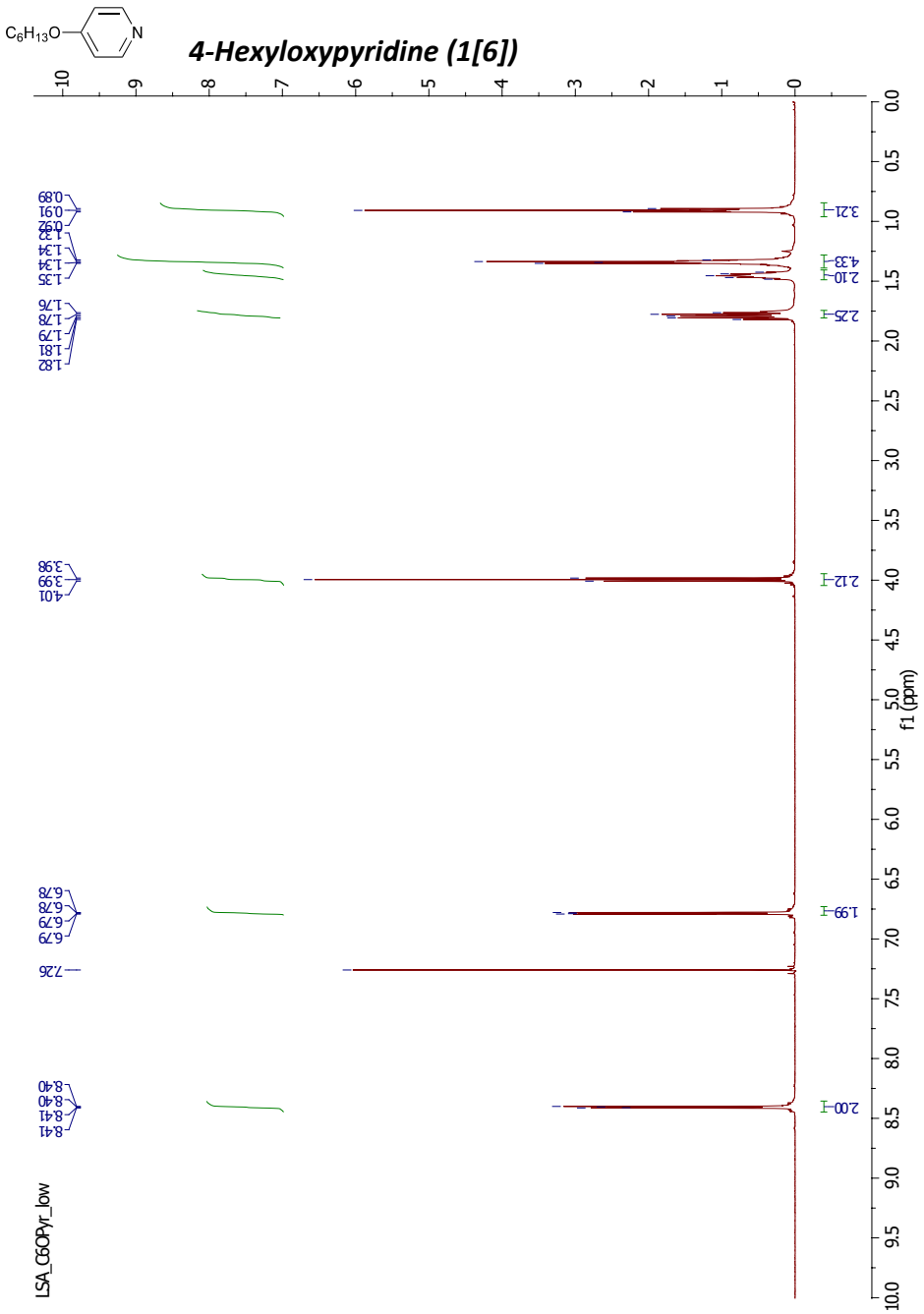


Figure S3. ^{1}H NMR of **1[6]** ($CDCl_3$, 500 MHz).

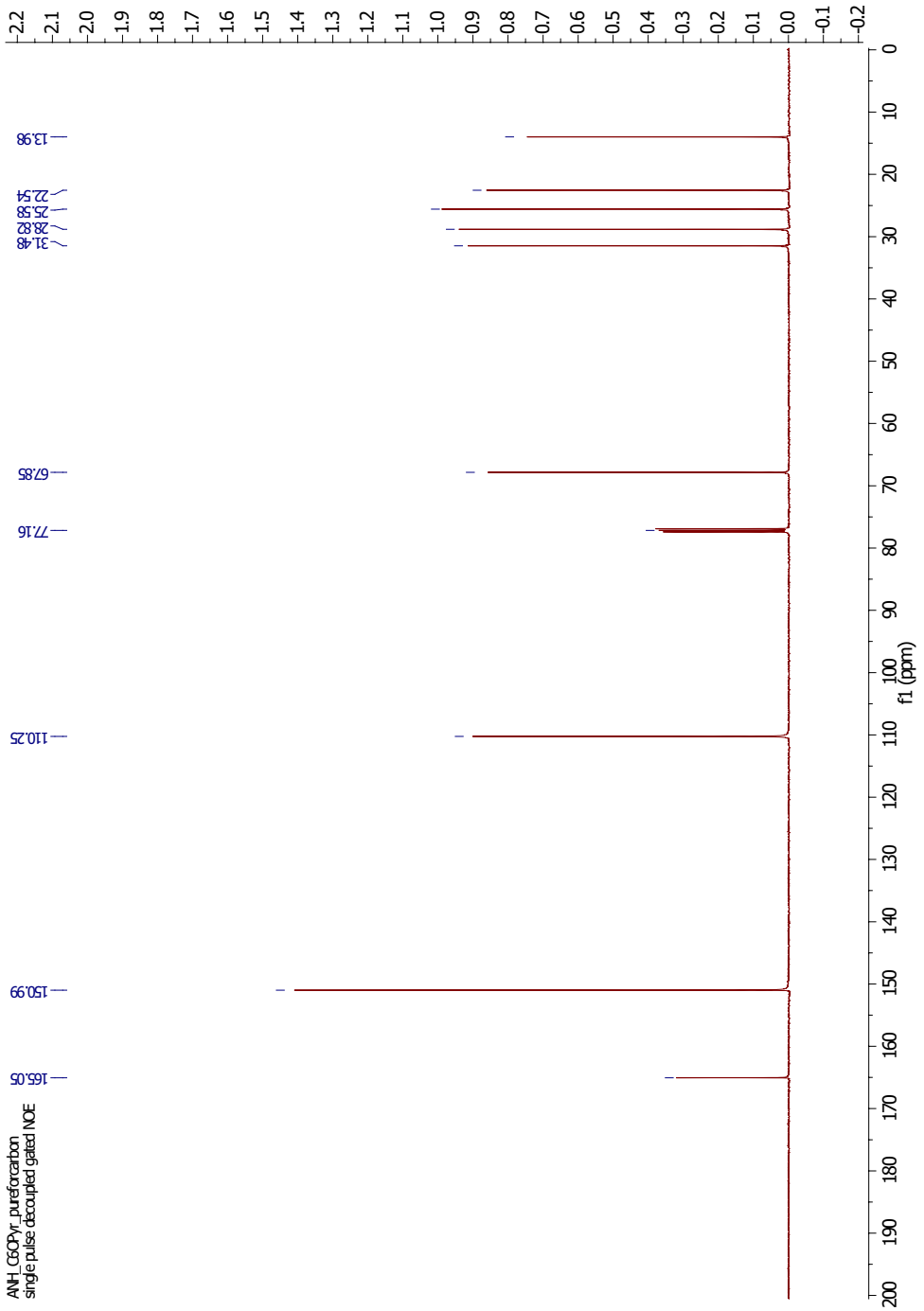


Figure S4. ^{13}C NMR of **1[6]** ($CDCl_3$, 125 MHz).

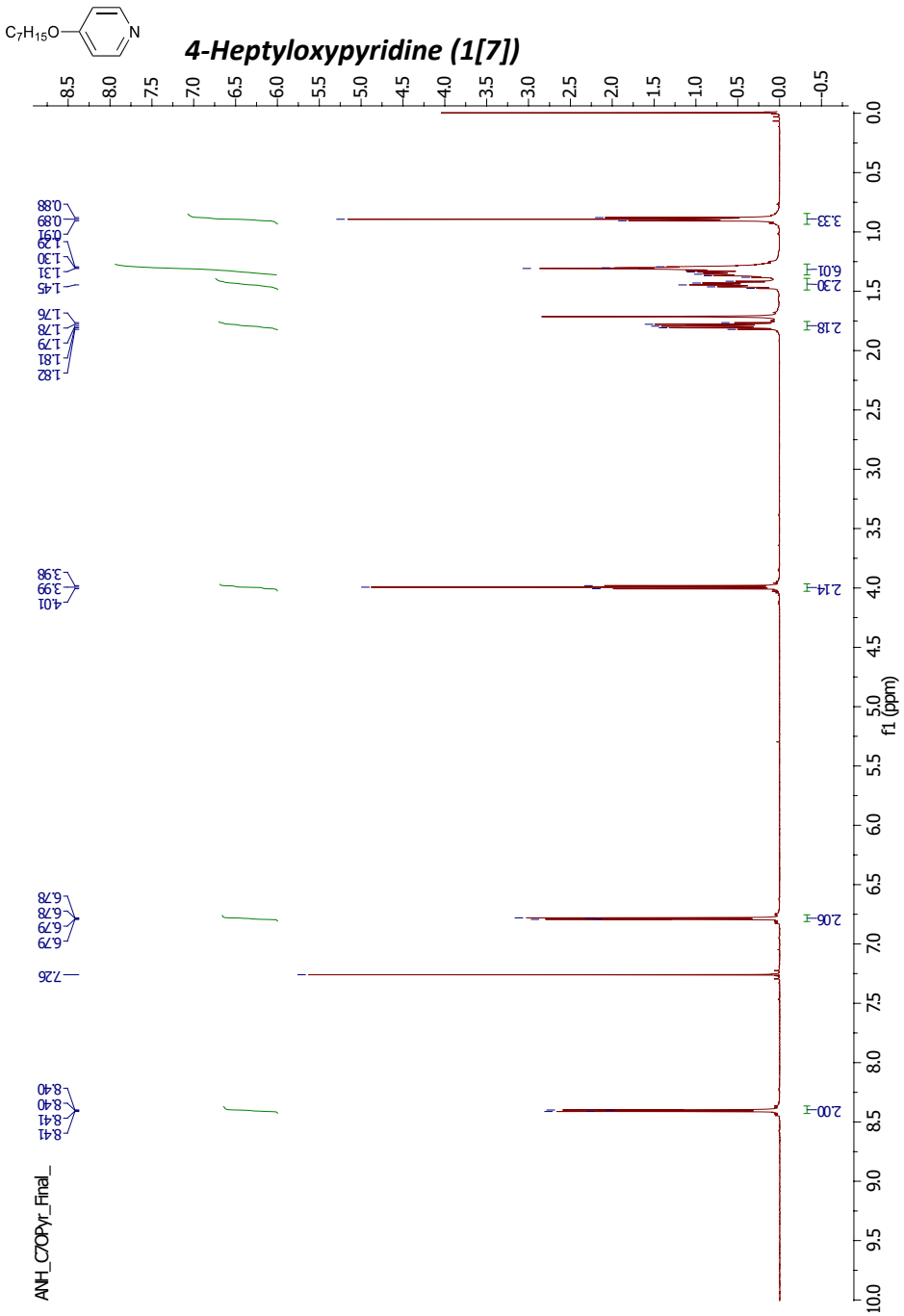


Figure S5. ^{1}H NMR of **1[7]** ($CDCl_3$, 500 MHz).

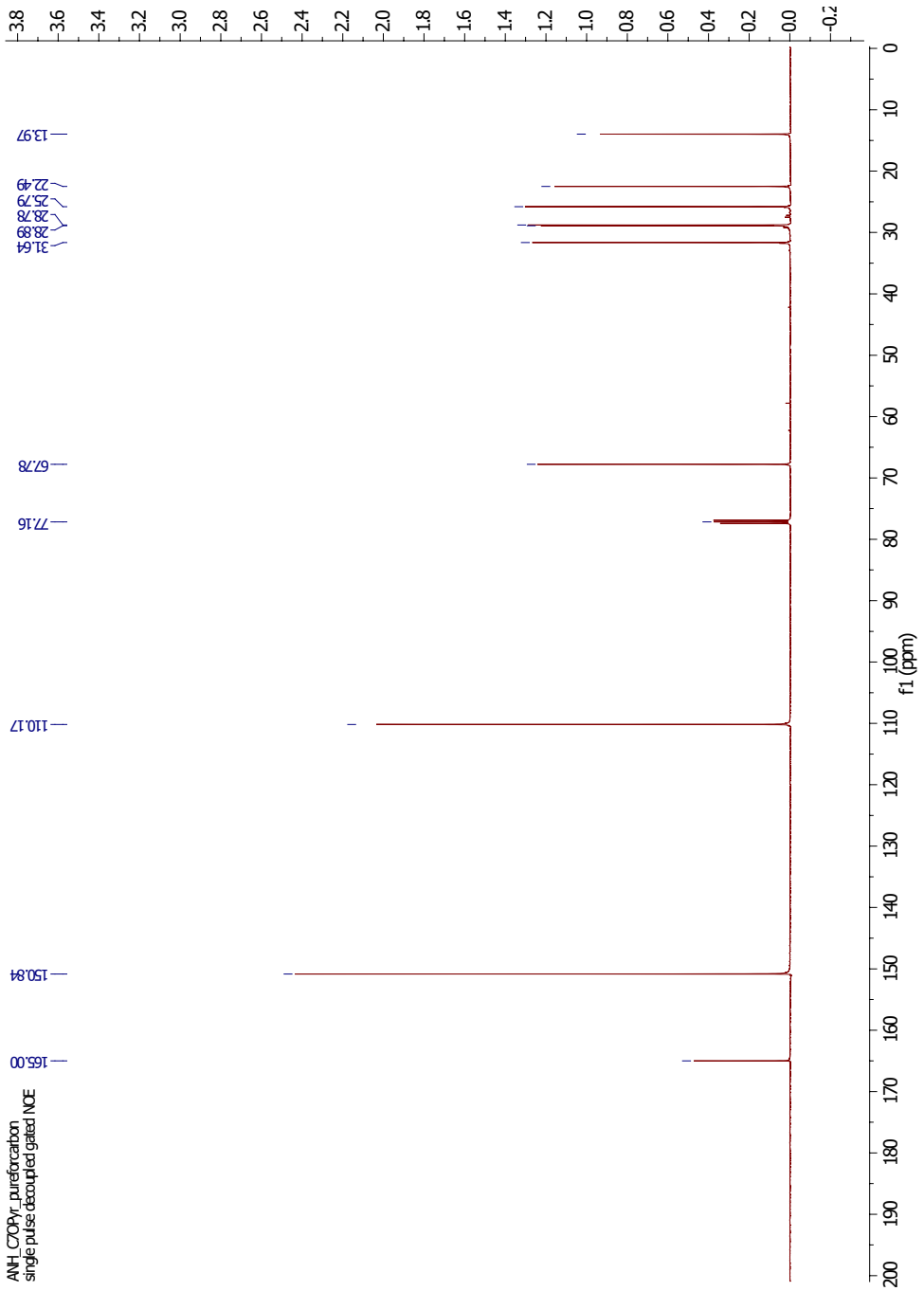


Figure S6. ^{13}C NMR of **1[7]** ($CDCl_3$, 125 MHz).

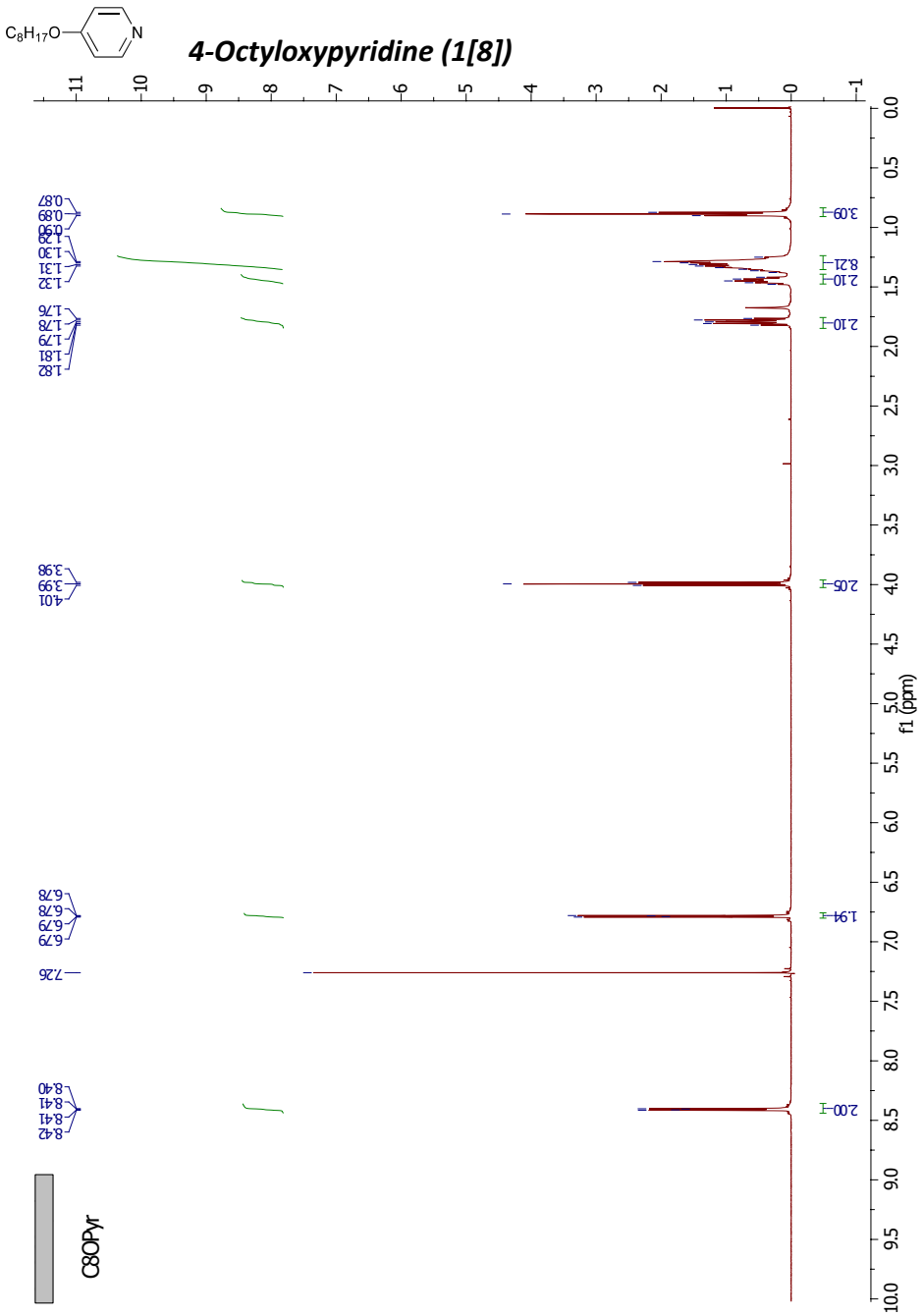


Figure S7. ^{1}H NMR of **1[8]** ($CDCl_3$, 500 MHz).

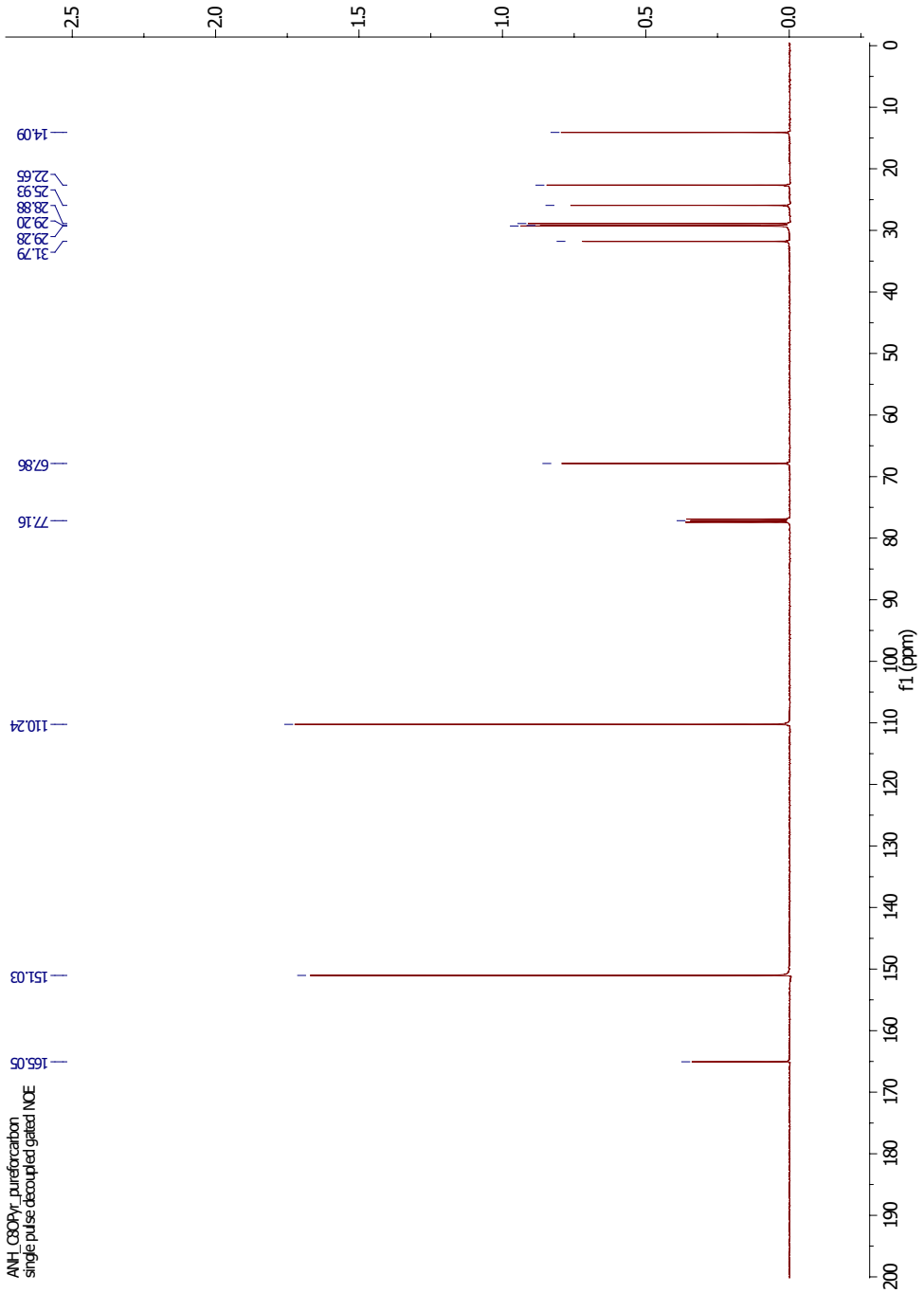


Figure S8. ^{13}C NMR of **1[8]** ($CDCl_3$, 125 MHz).

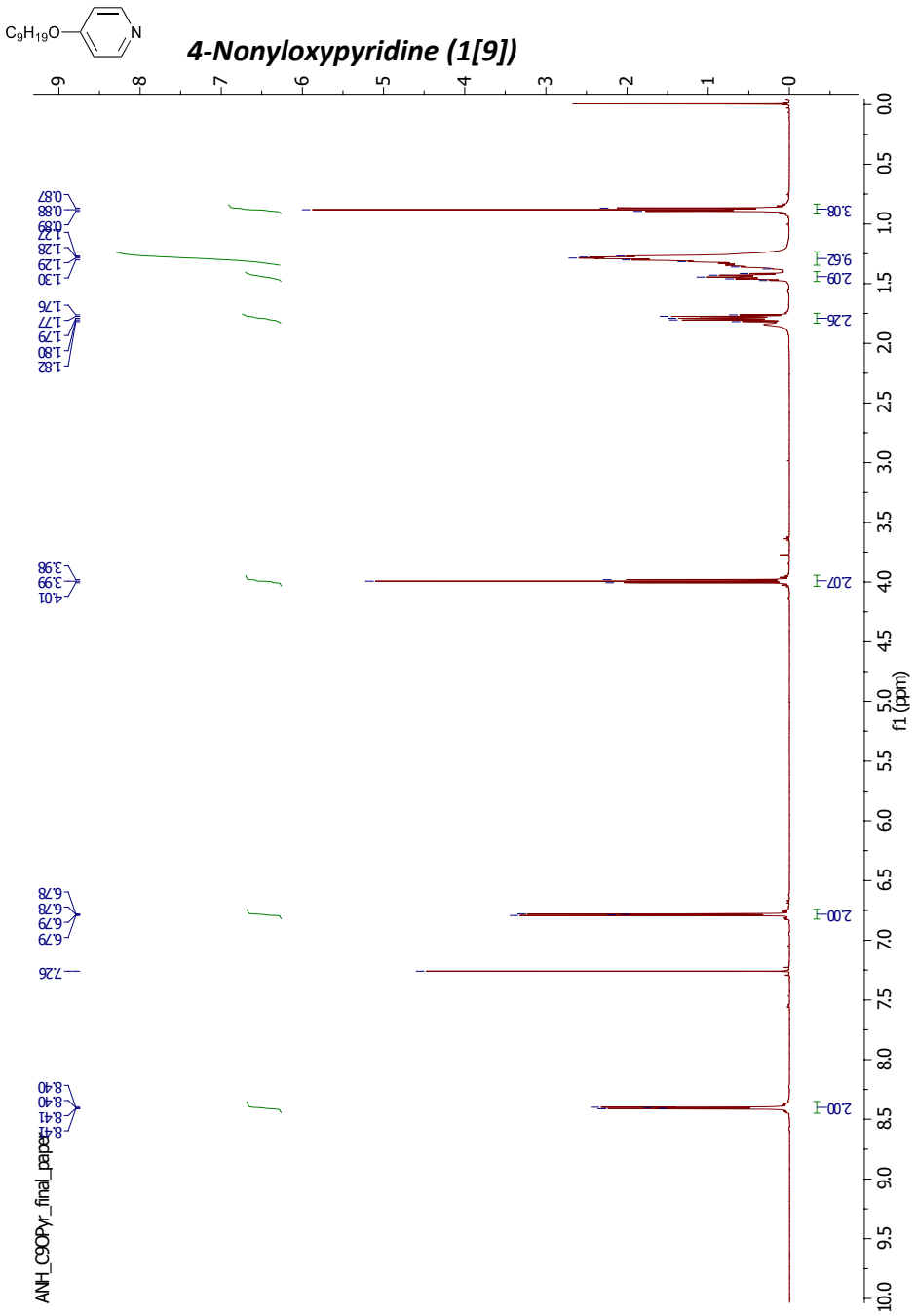


Figure S9. ^{1}H NMR of **1[9]** ($CDCl_3$, 500 MHz).

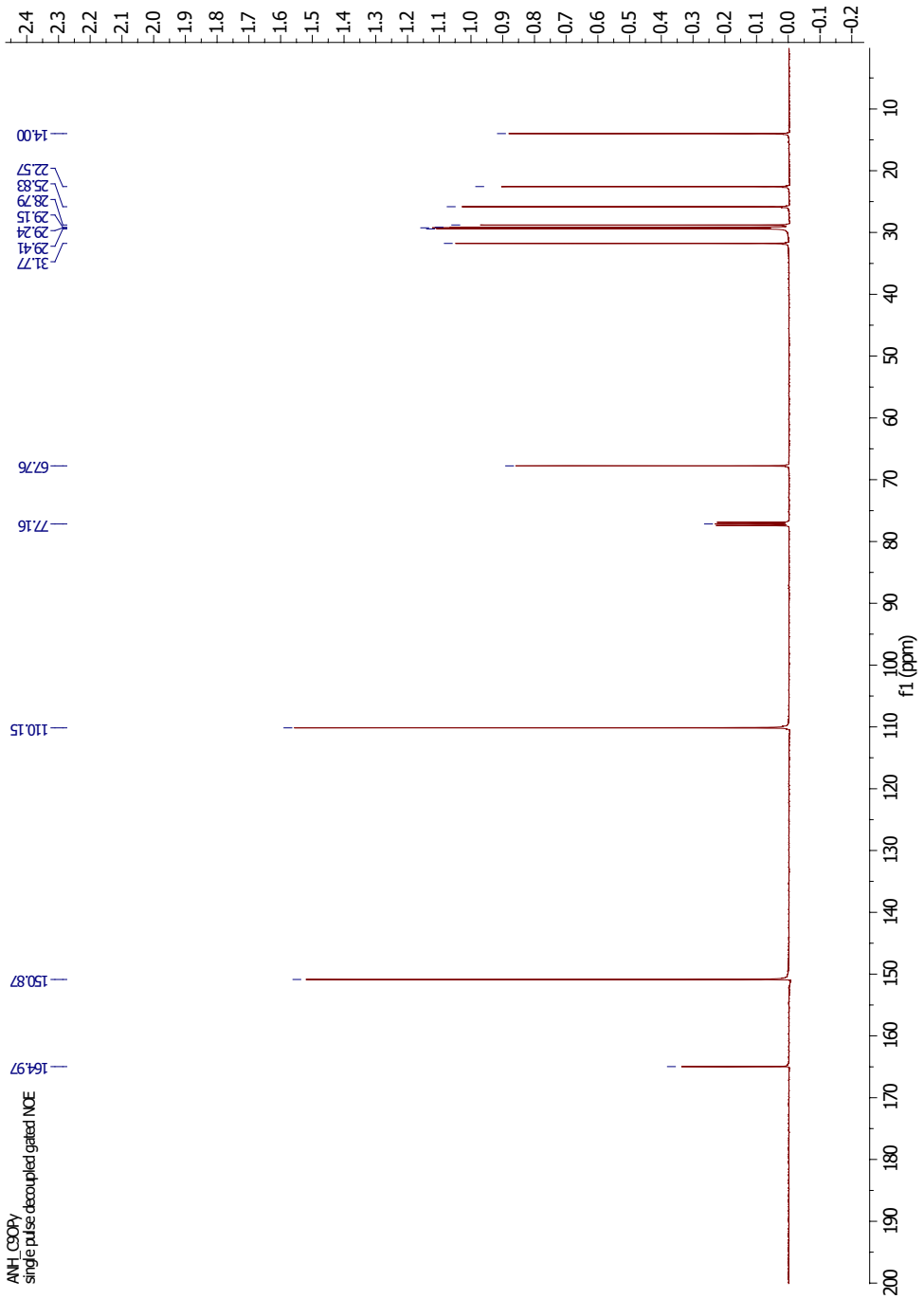


Figure S10. ^{13}C NMR of **1[9]** ($CDCl_3$, 125 MHz).

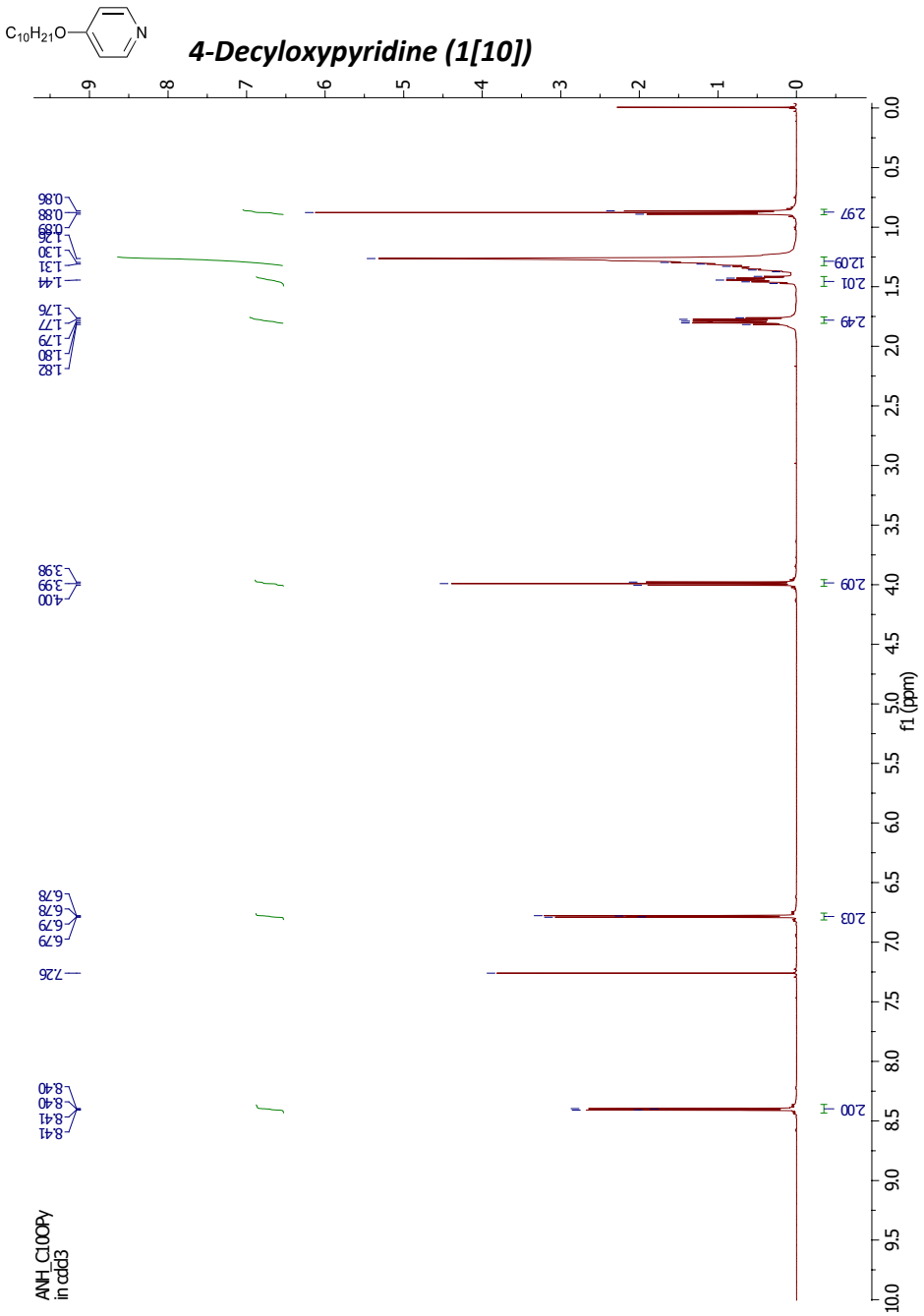


Figure S11. ^{1}H NMR of **1[10]** ($CDCl_3$, 500 MHz).

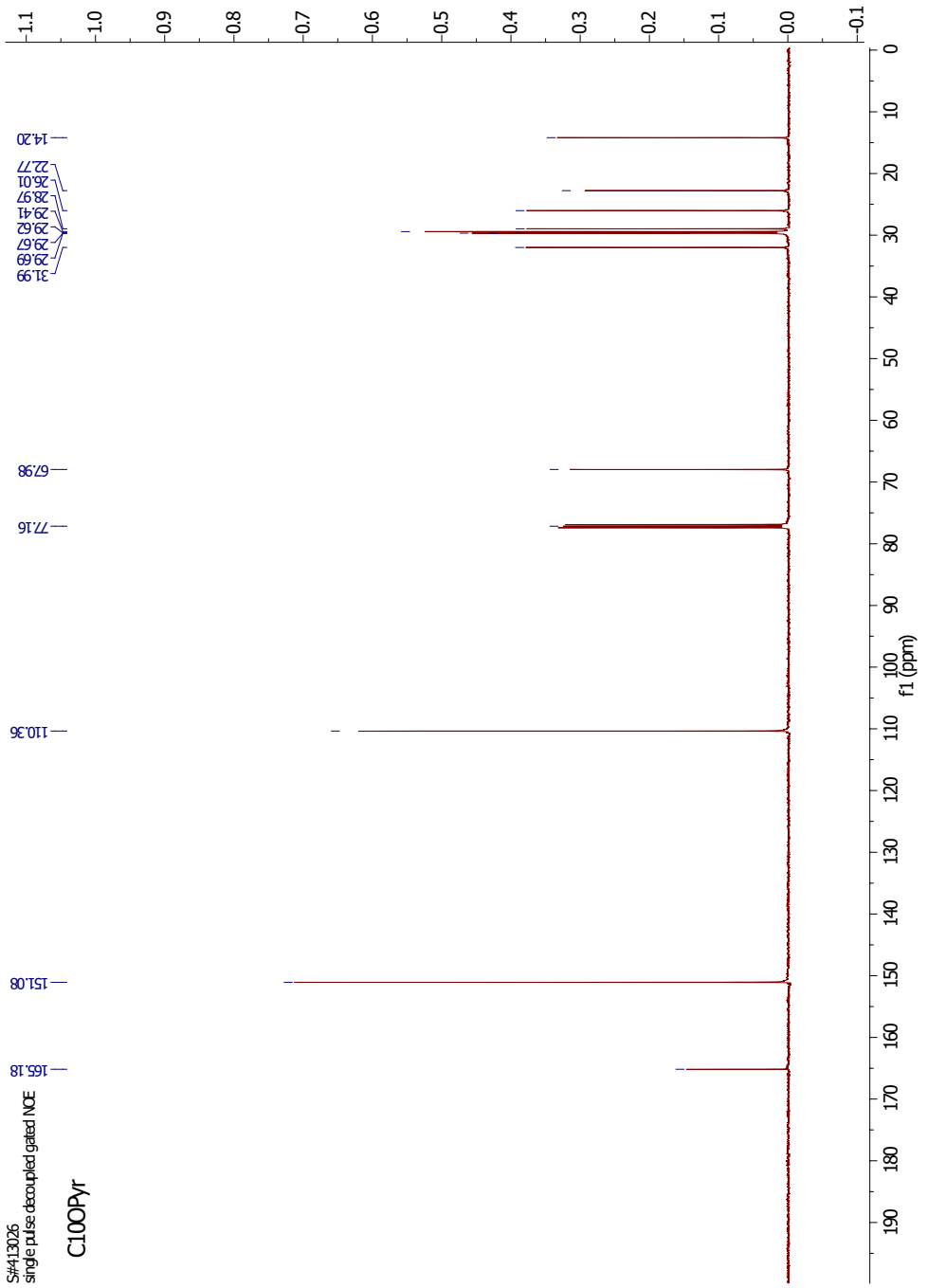


Figure S12. ^{13}C NMR of **1[10]** ($CDCl_3$, 125 MHz).

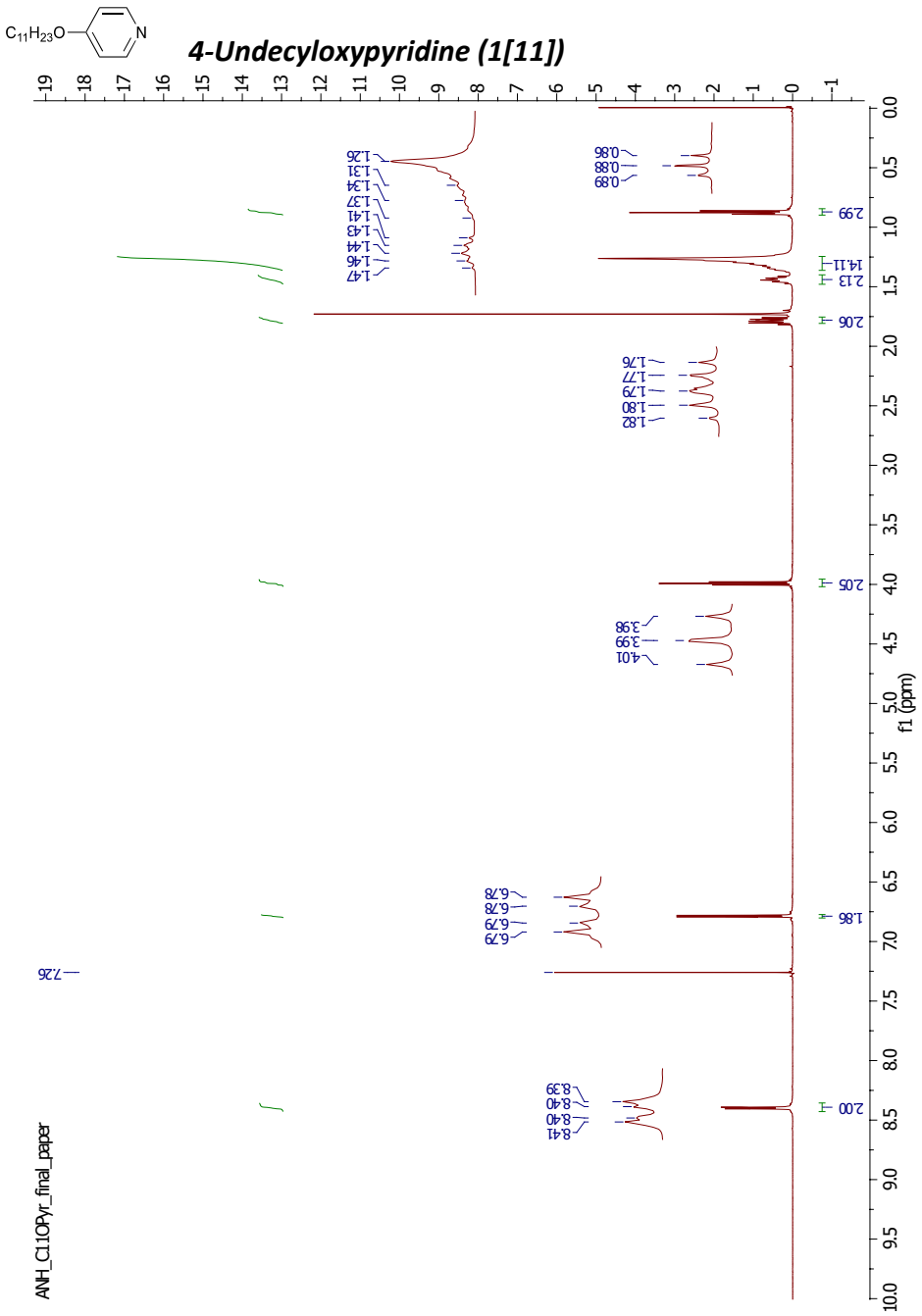


Figure S13. ^{1}H NMR of **1[11]** ($CDCl_3$, 500 MHz).

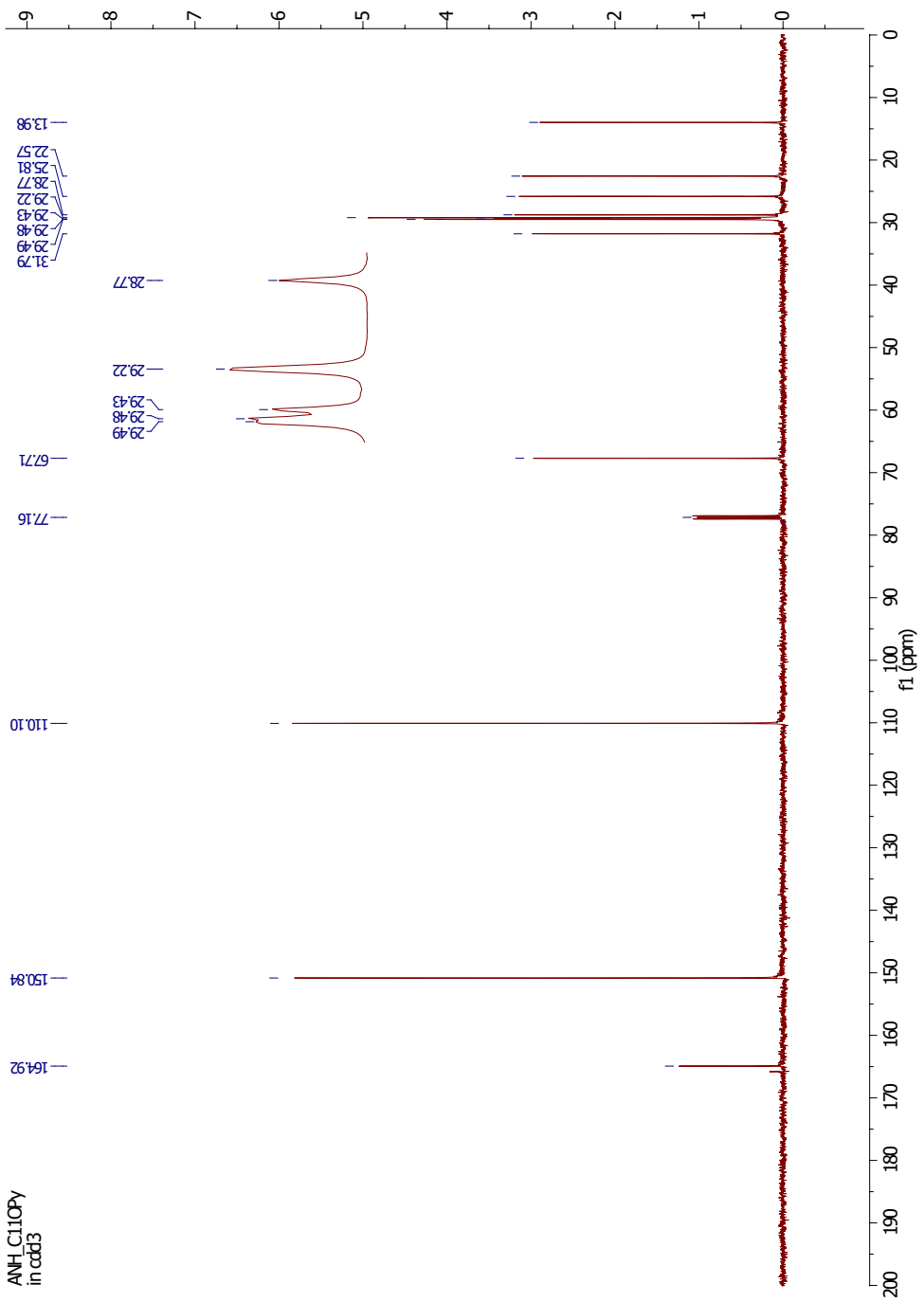


Figure S14. ^{13}C NMR of **1[11]** ($CDCl_3$, 125 MHz).

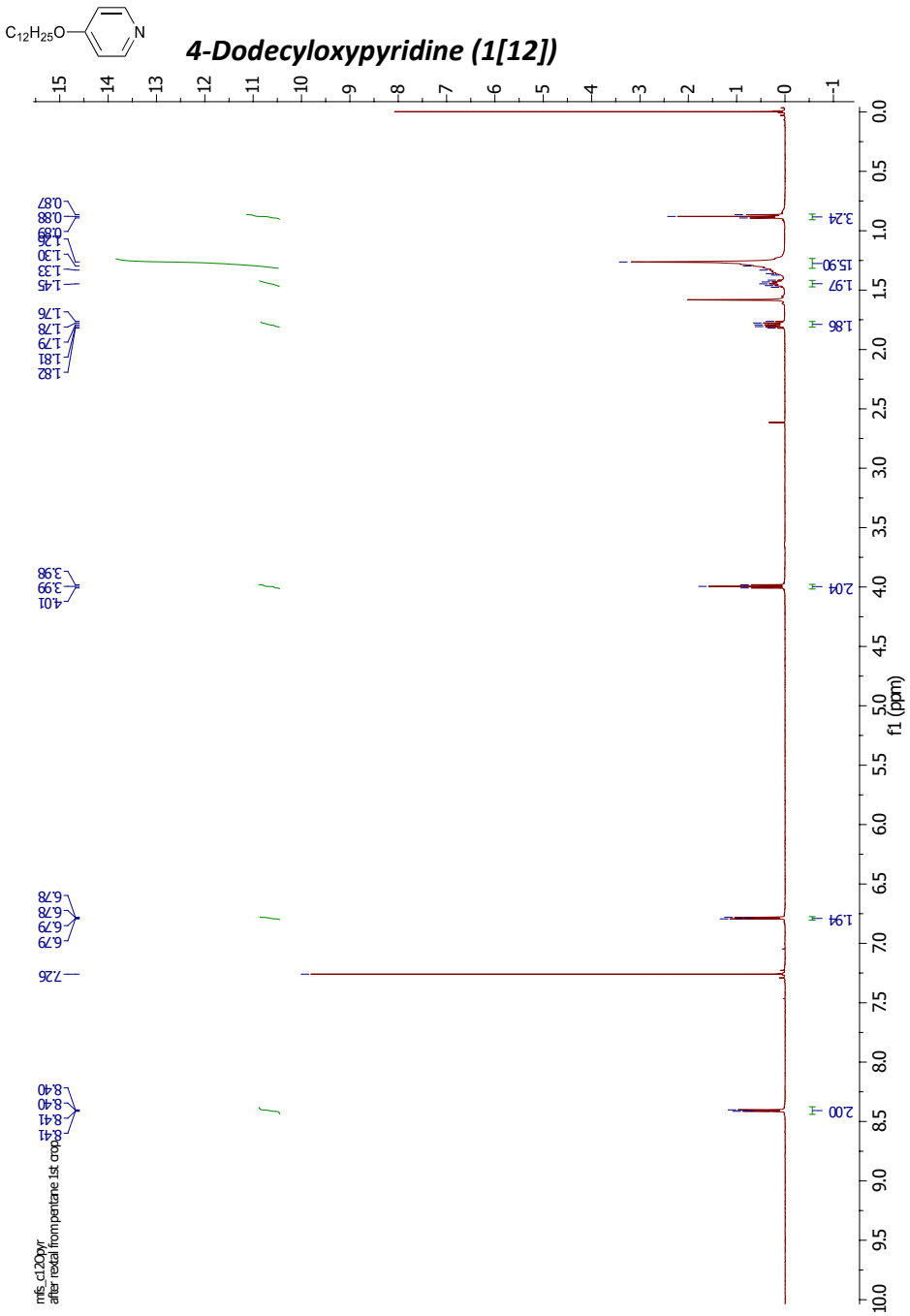


Figure S15. ^{1}H NMR of **1[12]** ($CDCl_3$, 500 MHz).

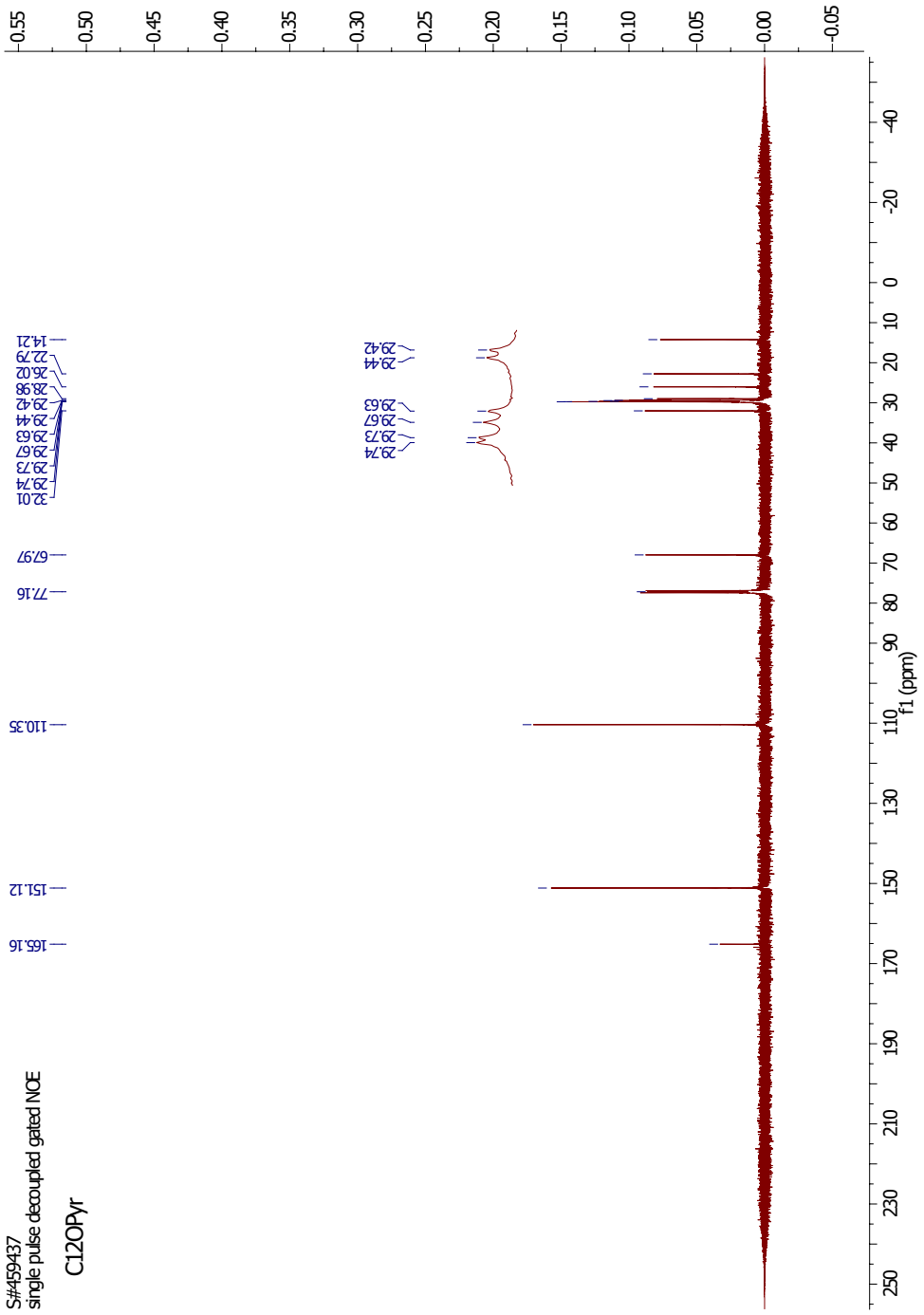


Figure S16. ^{13}C NMR of **1[12]** ($CDCl_3$, 125 MHz).

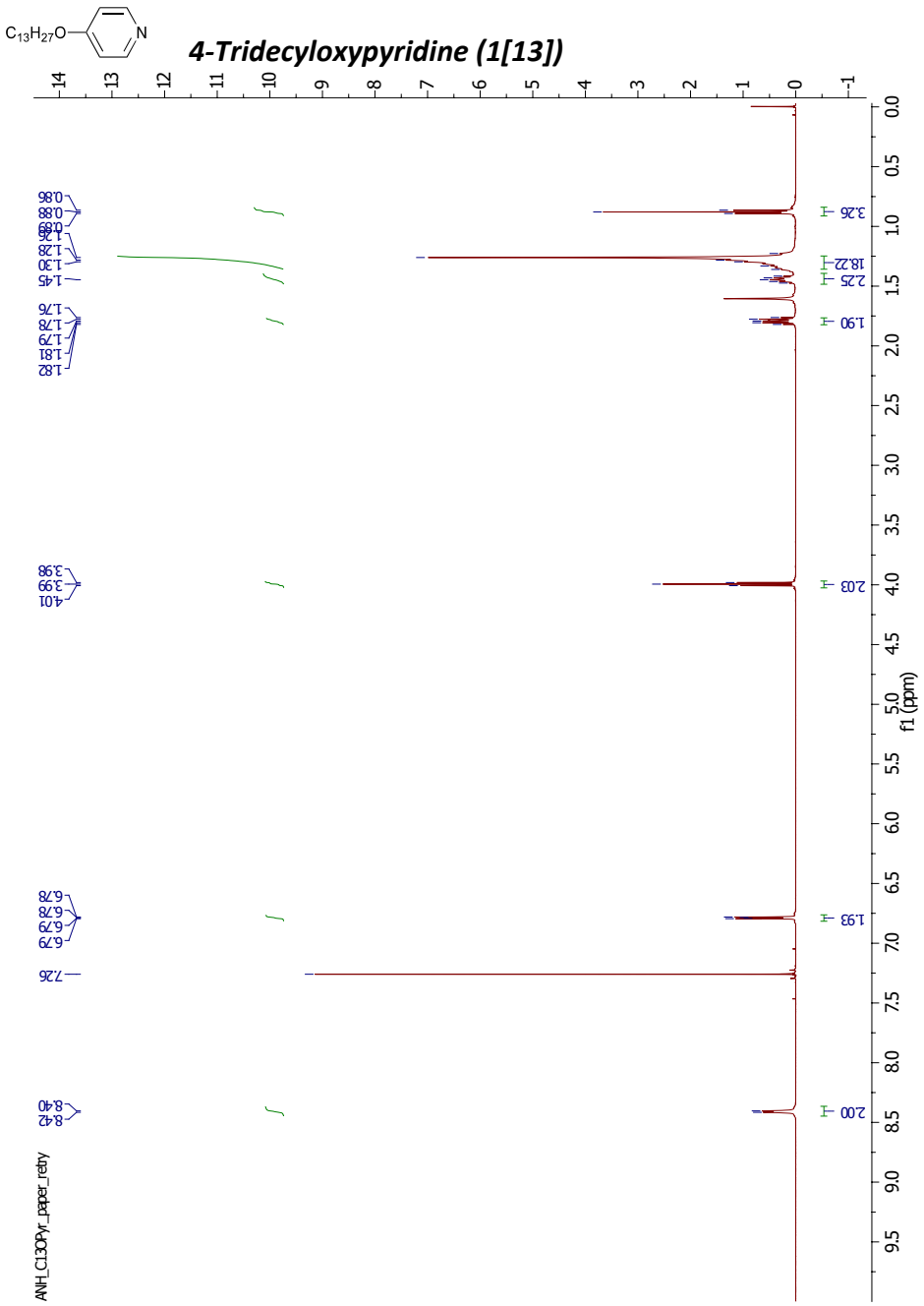


Figure S17. ^{1}H NMR of **1[13]** ($CDCl_3$, 500 MHz).

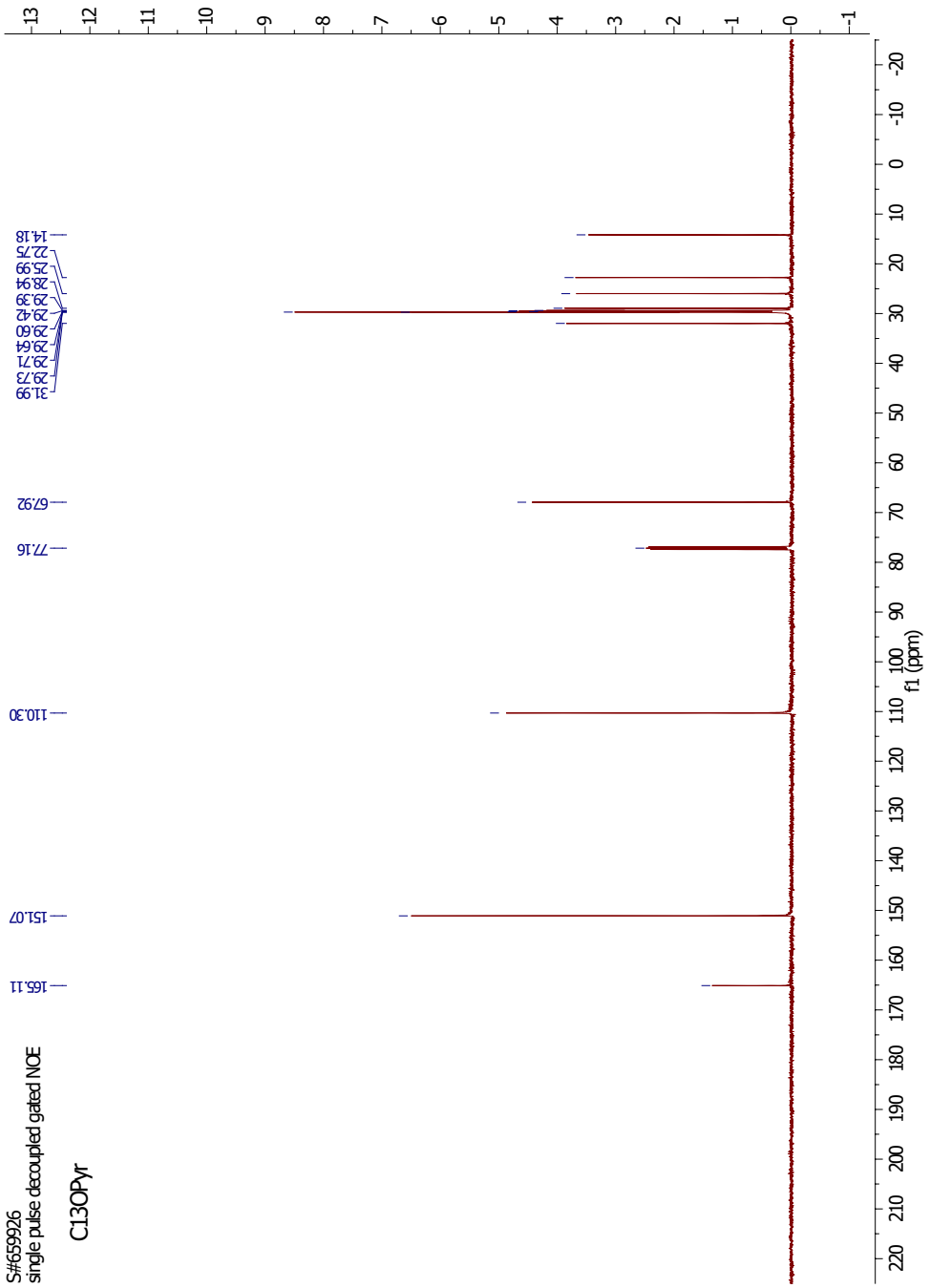


Figure S18. ^{13}C NMR of **1[13]** ($CDCl_3$, 125 MHz).

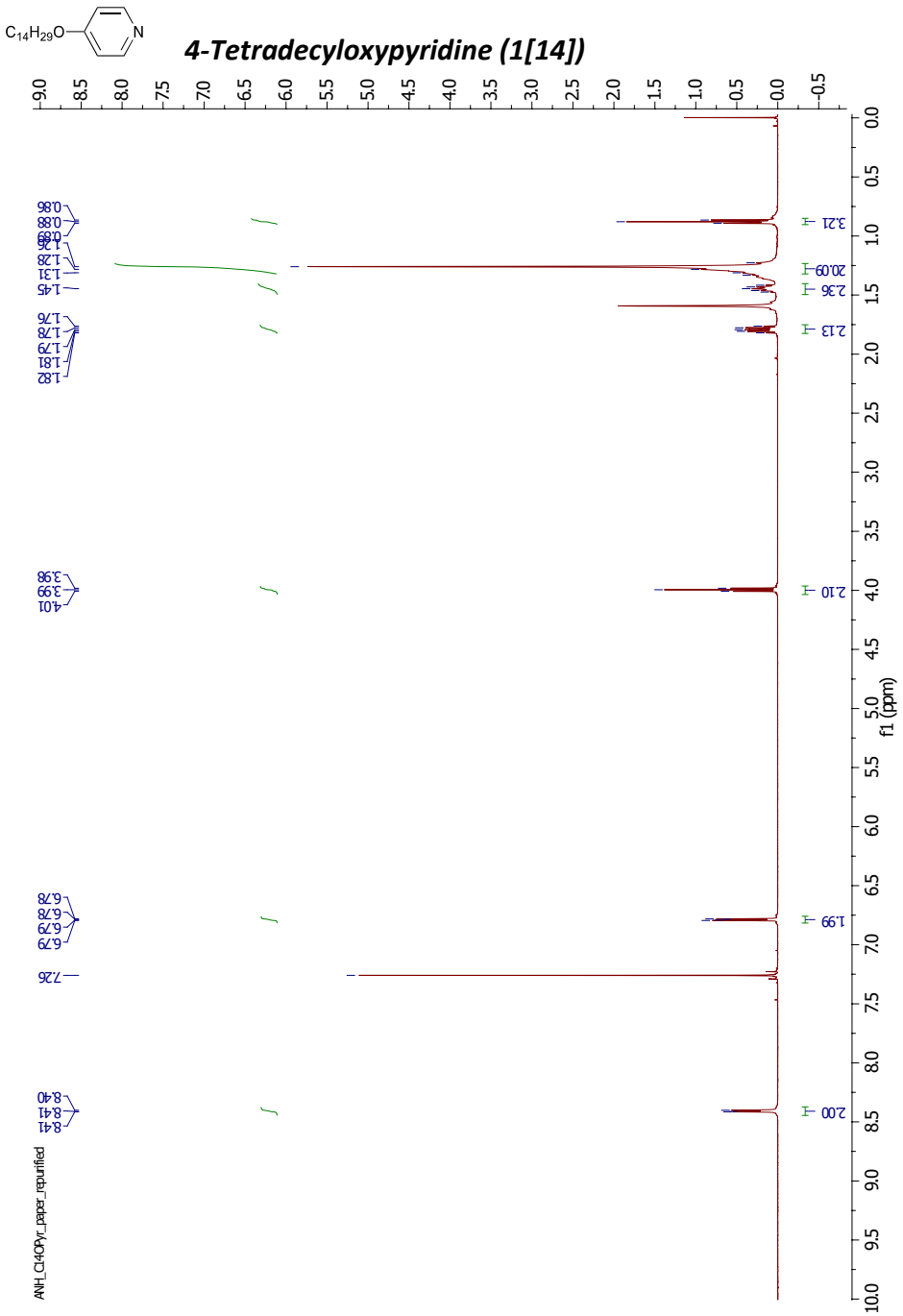


Figure S19. ^{1}H NMR of **1[14]** ($CDCl_3$, 500 MHz).

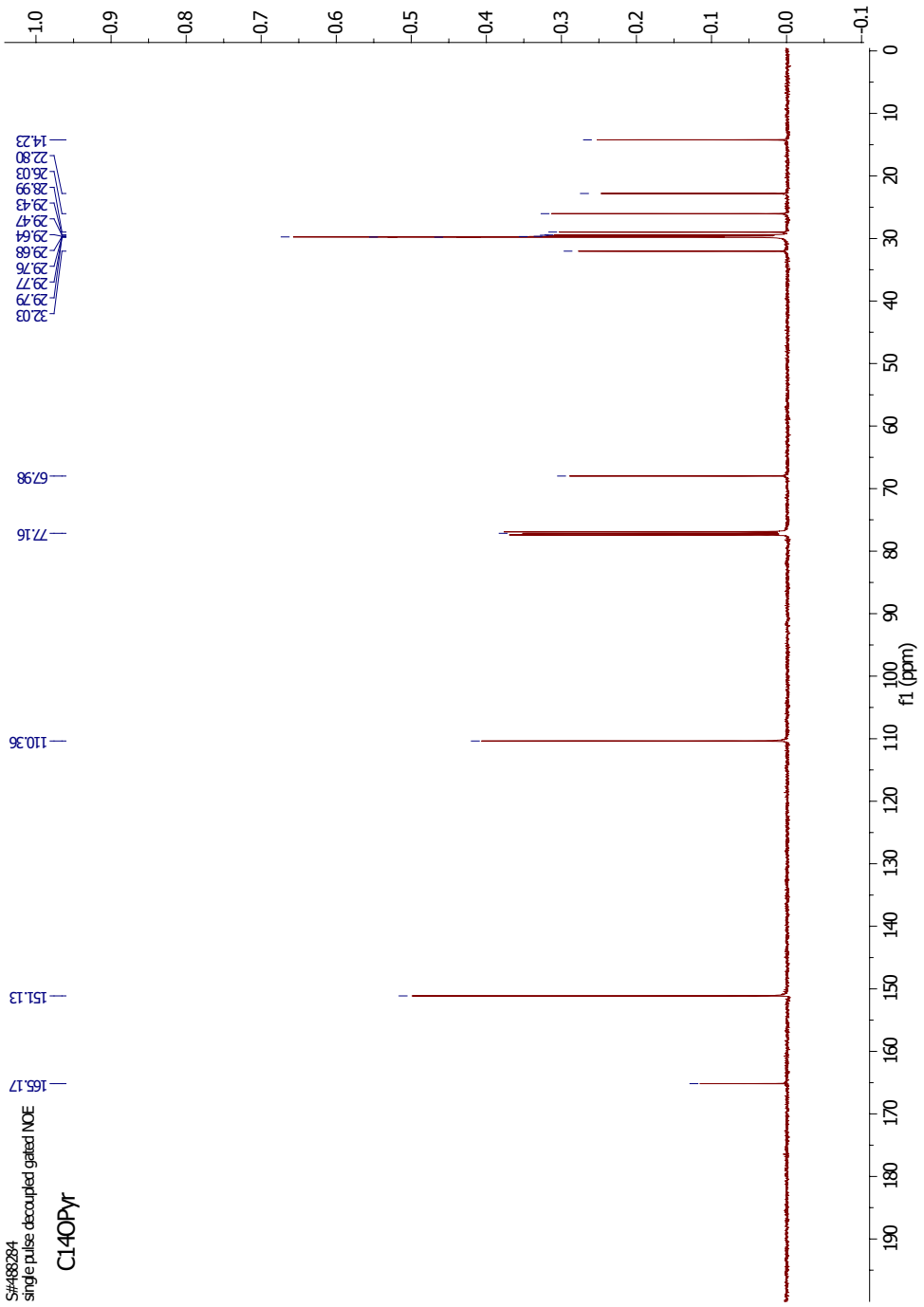


Figure S20. ^{13}C NMR of **1[14]** ($CDCl_3$, 125 MHz).

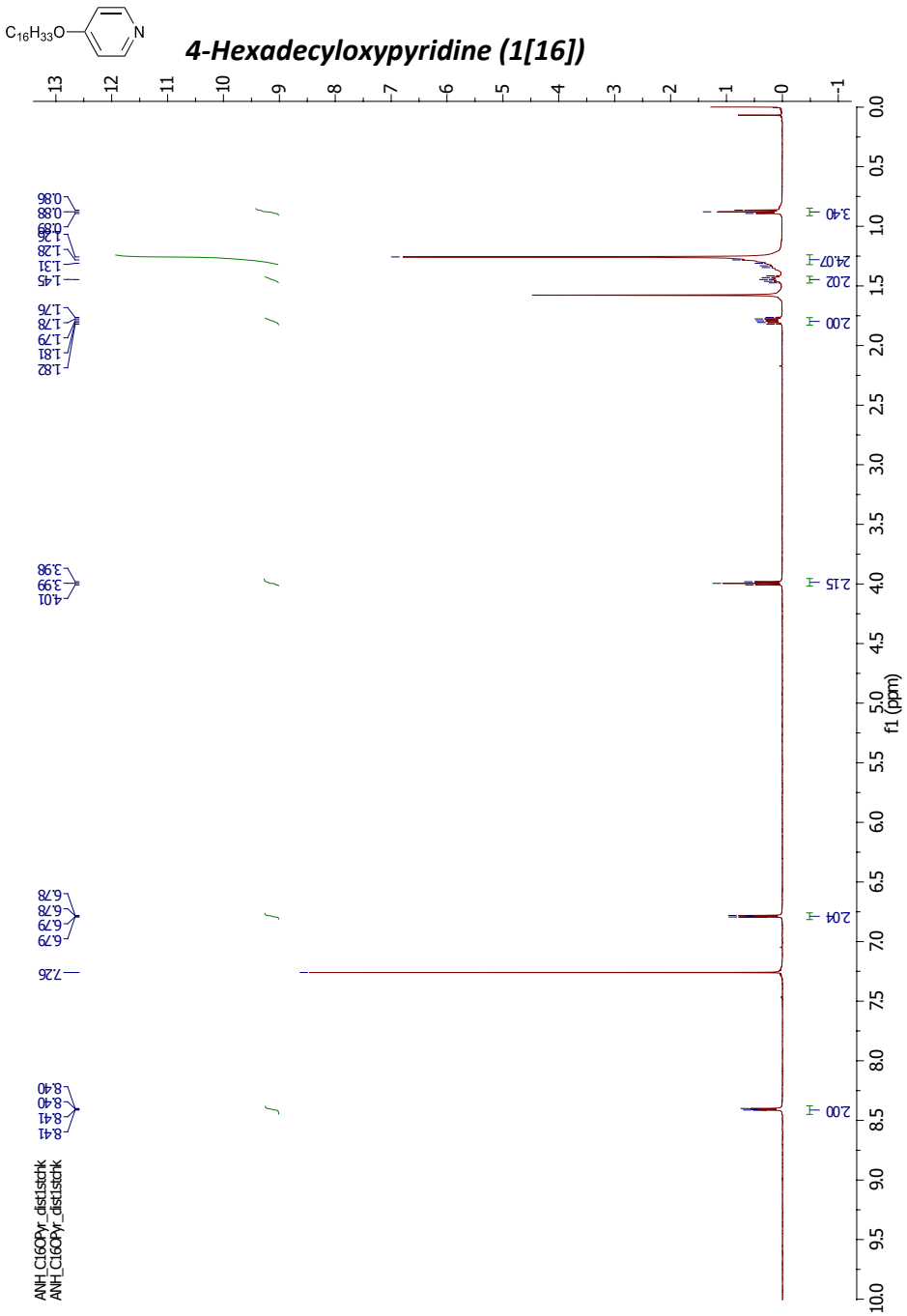


Figure S21. ^{1}H NMR of **1[16]** ($CDCl_3$, 500 MHz).

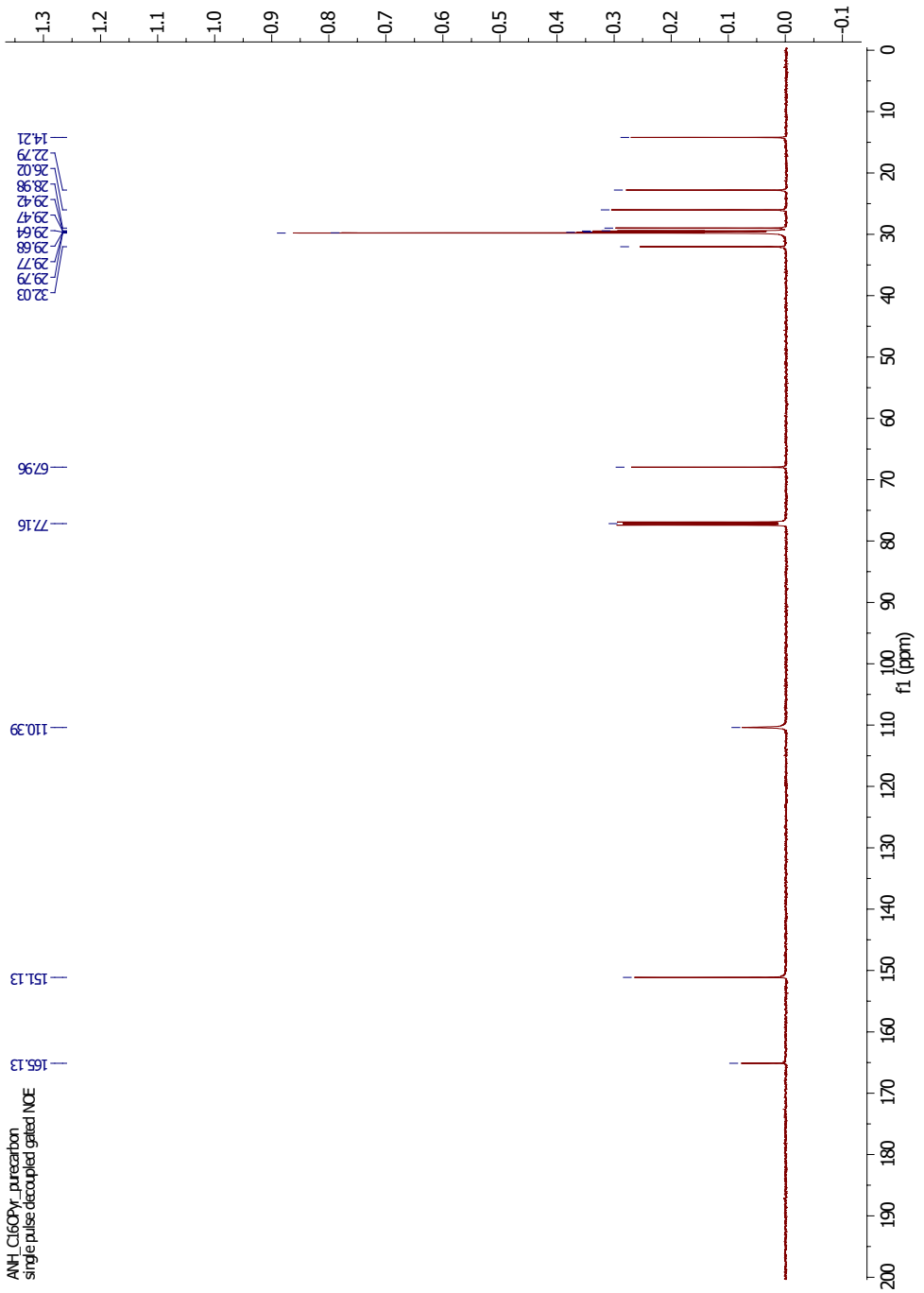


Figure S22. ^{13}C NMR of **1[16]** ($CDCl_3$, 125 MHz).

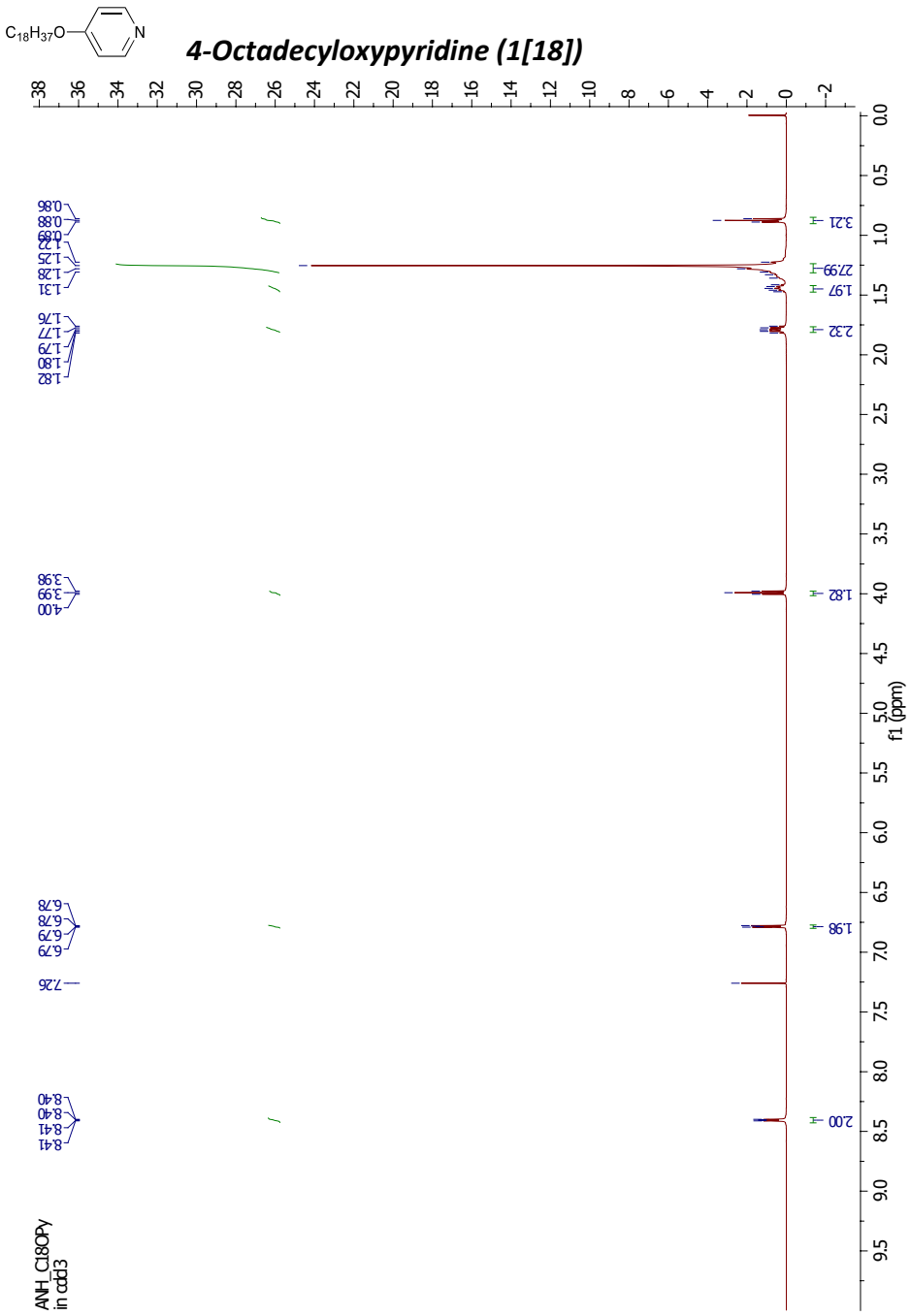


Figure S23. ^{1}H NMR of **1[18]** ($CDCl_3$, 500 MHz).

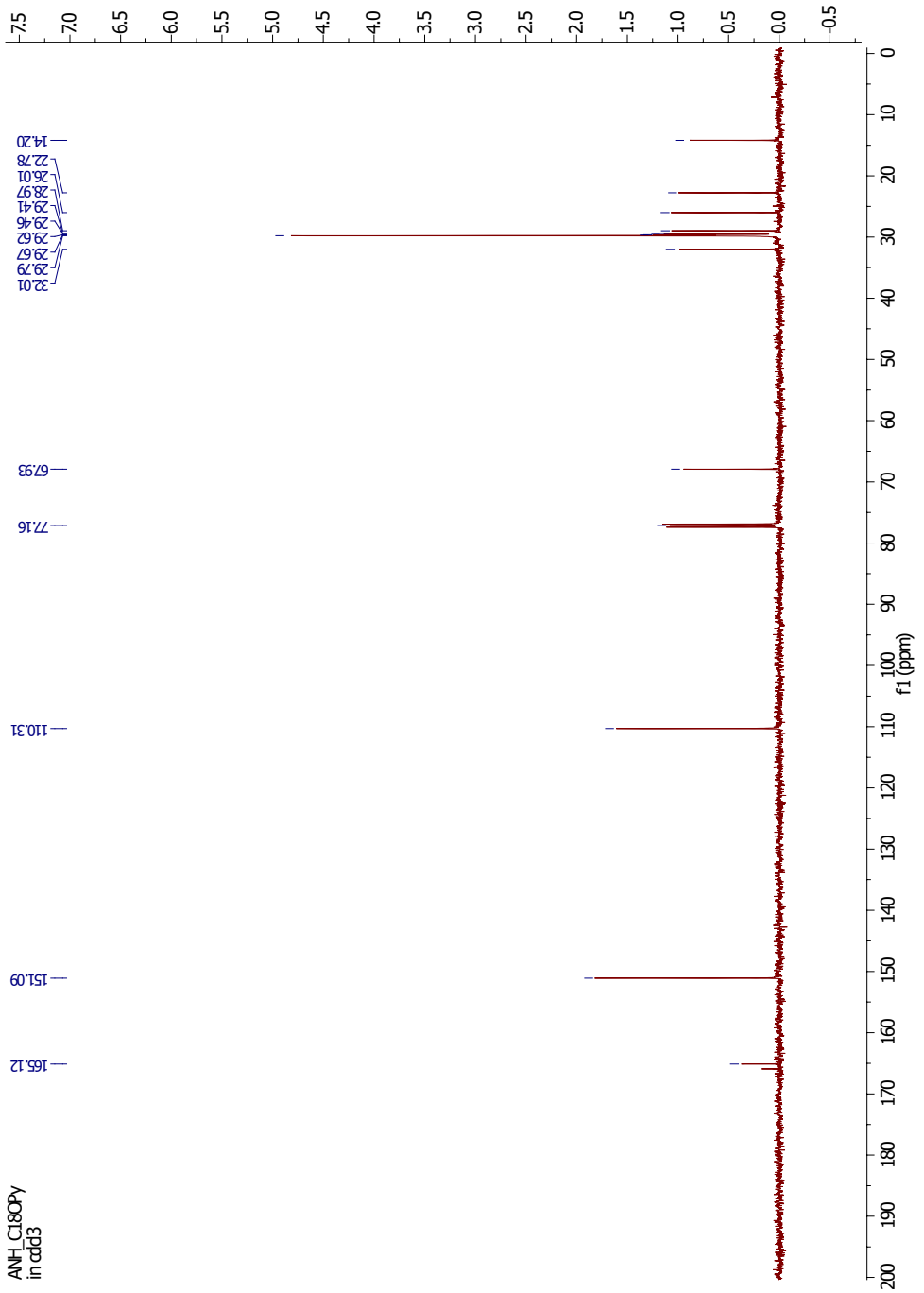


Figure S24. ^{13}C NMR of **1[18]** ($CDCl_3$, 125 MHz).

4. References

(1) CrysAlisPro 1.171.39.44a (Rigaku Oxford Diffraction, **2018**).

(2) Sheldrick, G. M. *Acta Crystallogr., Sect. A* **2015**, *A71*, 3-8.

(3) Sheldrick, G. M. *Acta Crystallogr., Sect. C* **2015**, *C71*, 3-8.

(4) Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. *J. Appl. Cryst.* **2009**, *42*, 339-341.

(5) Fulmer, G. R.; Miller, A. J. M.; Sherden, N. H.; Gottlieb, H. E.; Nudelman, A.; Stoltz, B. M.; Bercaw, J. E.; Goldberg, K. I. *Organometallics* **2010**, *29*, 2176–2179.

(6) Jankowiak, A.; Baliński, A.; Harvey, J. E.; Mason, K.; Januszko, A.; Kaszyński, P.; Young, V. G., Jr.; Persoons, A. *J. Mater. Chem. C* **2013**, *1*, 1144-1159.
(7) Butler, D. E.; Bass, P.; Nordin, I. C.; Hauck, F. P., Jr.; L'Italien, Y. J. *J. Med. Chem.* **1971**, *14*, 575-579.
(8) Kaszynski, P.; Huang, J.; Jenkins, G. S.; Bairamov, K. A.; Lipiak, D. *Mol. Cryst. Liq. Cryst.* **1995**, *260*, 315-332.
(9) Schmid, G. H.; Wolkoff, A. W. *Can. J. Chem.* **1972**, *50*, 1181-1187.
(10) Lin, H.-C.; Shiaw, J.-M.; Liu, R.-C.; Tsai, C.; Tso, H.-H. *Liq. Cryst.* **1998**, *25*, 277-283.
(11) Alcalde, E.; Pérez-García, L.; Dinarés, I.; Frigola, J. *J. Org. Chem.* **1991**, *56*, 6516-6521.
(12) Gonzalez, L.; Gimeno, N.; Tejedor, R. M.; Polo, V.; Ros, M. B.; Uriel, S.; Serrano, J. L. *Chem. Mater.* **2013**, *25*, 4503-4510.
(13) Vompe, A. F.; Monich, N. V.; Meskhi, L. M. *Russ. J. Org. Chem.* **1974**, *10*, 1301-1306.
(14) Abdullah, N.; Sharmin, N.; Ozair, L. N.; Nordin, A. R.; Mohd Nasir, W. S. N.; Mohamadin, M. I. *J. Coord. Chem.* **2015**, *68*, 1347-1360.

