

Electronic Supplementary Information

Glaser-Eglinton-Hay sp-sp coupling and macrocyclization: construction of a new class of polyether macrocycles having a 1,3-diyne unit

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Table 1 (ESI). Single crystal data and the results of the structure refinement details.

Good quality single crystals were grown by using a combination of dichloromethane or hexanes or methanol or ethyl acetate by slow solvent evaporation technique at 25 °C and the single crystal X-ray diffraction data for all the compounds were collected using four circle CCD diffractometer (monochromatic Mo K α radiation). Data collection and unit cell refinement for the data sets were done using APEX-II suit,¹ data reduction and integration were performed by SAINT V7.685A,¹ and absorption corrections and scaling were done using SADABS V2008/1.¹ The crystal structures were solved using Olex2² or WinGx³ packages using SHELXS97,⁴ and the structures were refined using SHELXL97.⁴ All the hydrogen atoms have been fixed at their geometrically ideal positions and refined using the riding model available within SHELXS97. All figures including the packing and interaction diagrams have been generated using Mercury.⁵ Geometric calculations including the least-squares plane angle calculations have been done using PARST⁶ and PLATON.⁷

Sample code	5a	5f	5g	5h	5i
Empirical formula	C ₂₂ H ₂₀ O ₄	C ₂₈ H ₂₄ O ₄	C ₂₄ H ₂₂ O ₄	C ₂₄ H ₂₀ O ₄	C ₂₈ H ₂₀ O ₆
Formula weight	348.38	424.47	374.41	372.42	452.44
Temperature (K)	298	298	298	298	298
Crystal system	monoclinic	monoclinic	monoclinic	monoclinic	monoclinic
Space group	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> 2 ₁ / <i>c</i>
<i>a</i> /Å	17.4674(16)	8.867(3)	8.903(4)	4.856(3)	11.383(3)
<i>b</i> /Å	22.482(2)	16.757(5)	23.110(10)	53.09(4)	11.543(3)
<i>c</i> /Å	9.5117(9)	14.800(5)	10.380(5)	7.510(5)	17.587(5)
α /°	90	90	90	90	90
β /°	90.222(6)	91.210(7)	108.380(10)	93.902(18)	97.626(13)
γ /°	90	90	90	90	90
Volume/Å ³	3735.20(6)	2198.5(12)	2026.7(16)	1932(2)	2290.2(11)
<i>Z</i>	8	4	4	4	4
<i>Z'</i>	2	1	1	1	1
ρ_{calc} /mg/mm ³	1.239	1.282	1.227	1.2805	1.312
<i>m</i> /mm ⁻¹	0.085	0.085	0.083	0.087	0.092
<i>F</i> (000)	1472.0	896	792	784.0	944.0
Crystal size/mm ³	0.2 × 0.2 × 0.1	0.3 × 0.2 × 0.2	0.3 × 0.2 × 0.2	0.3 × 0.2 × 0.1	0.3 × 0.2 × 0.1
2 $\theta_{\text{min, max}}$	2.3, 50.0°	3.6, 50.0°	3.5, 50.4°	1.5, 50.0°	3.6, 50.2°
<i>h</i> min, <i>h</i> max; <i>k</i> min, <i>k</i> max; <i>l</i> min, <i>l</i> max	-20, 19; -26, 21; -11, 11	-10, 10; 0, 19; 0, 17	-10, 10; -27, 17; -9, 12	-2, 5; -61, 63; -7, 8	-13, 13; -13, 13; -20, 20
No. of reflections	26640	3845	9134	10743	18779
No. of independant reflections	6589	3845	3649	3345	4022
<i>R</i> (int)	0.040	0	0.054	0.082	0.049
Data/restraints /parameters	6589/0/470	3845/0/290	3649/0/254	3345/0/252	4022/0/307
Goodness-of-fit on <i>F</i> ²	1.040	1.086	1.015	1.128	1.018
<i>R</i> _{obs}	0.059	0.038	0.046	0.080	0.039
<i>wR</i> ₂ (obs)	0.145	0.108	0.113	0.236	0.097
$\Delta\rho_{\text{max, min}}$ / e Å ⁻³	0.19/-0.17	0.34/-0.16	0.18/-0.15	0.48/-0.35	0.15/-0.19

Continued.....

Table 1 (ESI). Single crystal data and the results of the structure refinement details.

Sample code	5k	6a	6c	6g	7b
Empirical formula	C ₂₄ H ₂₄ O ₄	C ₂₄ H ₂₄ O ₅	C ₂₆ H ₂₈ O ₆	C ₂₆ H ₂₄ O ₈	C ₃₀ H ₃₂ O ₄
Formula weight	376.43	392.43	436.48	464.45	456.56
Temperature (K)	298	298	298	298	298
Crystal system	triclinic	monoclinic	orthorhombic	orthorhombic	monoclinic
Space group	<i>P</i> $\bar{1}$	<i>P</i> 2 ₁ / <i>c</i>	<i>Pbcn</i>	<i>P</i> 2 ₁ 2 ₁ 2 ₁	<i>P</i> 2 ₁ / <i>c</i>
<i>a</i> /Å	5.975(7)	20.0985(11)	18.8390(8)	8.9306(5)	15.205(6)
<i>b</i> /Å	10.756(13)	8.3800(4)	13.5066(6)	13.0866(8)	16.280(7)
<i>c</i> /Å	16.56(2)	12.1731(6)	9.0088(3)	19.8566(11)	10.936(5)
α /°	73.702(14)	90	90	90	90
β /°	87.212(14)	91.639(2)	90	90	107.697(9)
γ /°	83.25(2)	90	90	90	90
Volume/Å ³	1014(2)	2049.42(18)	2292.30(16)	2320.7(2)	2578.9(19)
<i>Z</i>	2	4	4	4	4
<i>Z'</i>	1	1	1	1	1
ρ_{calc} /mg/mm ³	1.232	1.272	1.265	1.329	1.176
μ /mm ⁻¹	0.083	0.089	0.089	0.099	0.077
<i>F</i> (000)	400	832	928	976	976
Crystal size/mm ³	0.3 × 0.2 × 0.2	0.3 × 0.2 × 0.1	0.3 × 0.3 × 0.2	0.3 × 0.2 × 0.2	0.3 × 0.2 × 0.1
2 $\theta_{\text{min, max}}$	4.0, 50.0°	5.2, 49.7°	3.7, 50.0	3.7, 50.0°	2.8, 50.0°
<i>h</i> min, <i>h</i> max; <i>k</i> min, <i>k</i> max; <i>l</i> min, <i>l</i> max	-3, 7; -10, 12; -19, 19	-23, 18; -9, 9; -13, 14	-22, 22; -16, 13; -10, 7	-10, 10; -15, 14; -22, 22	-18, 18; -19, 14; -10, 12
No. of reflections	6191	7127	9702	12355	12584
No. of independant reflections	3543	3040	2031	4008	4539
<i>R</i> (int)	0.143	0.050	0.069	0.034	0.045
Data/restraints/parameters	3543/6/284	3040/0/263	2031/6/158	4008/0/323	4539/0/308
Goodness-of-fit on <i>F</i> ²	1.005	1.575	1.065	1.018	1.009
<i>R</i> _{obs}	0.082	0.110	0.057	0.047	0.053
<i>wR</i> ₂ (obs)	0.220	0.358	0.156	0.107	0.130
$\Delta\rho_{\text{max, min}}$ / e Å ⁻³	0.37/ -0.44	1.27/ -0.57	0.30/-0.37	0.28/-0.18	0.30/-0.18

Continued.....

Table 1 (ESI). Single crystal data and the results of the structure refinement details.

Sample code	7d	8a	9a	9c
Empirical formula	C ₃₀ H ₃₂ O ₅	C ₂₂ H ₂₃ NO ₅	C ₂₄ H ₂₆ O ₅ S	C ₂₆ H ₃₀ O ₄ S
Formula weight	472.56	381.41	426.51	438.56
Temperature (K)	298	298	298	298
Crystal system	triclinic	monoclinic	orthorhombic	triclinic
Space group	<i>P</i> $\bar{1}$	<i>P</i> 2 ₁ / <i>c</i>	<i>Pnma</i>	<i>P</i> $\bar{1}$
<i>a</i> /Å	8.6914(9)	10.4541(10)	9.5532(5)	8.9442(9)
<i>b</i> /Å	12.4846(14)	17.4437(17)	24.2453(13)	9.7734(10)
<i>c</i> /Å	25.241(3)	11.0894(10)	9.5232(4)	14.2491(12)
α /°	77.626(3)	90	90	85.521(5)
β /°	87.992(4)	104.244(5)	90	75.471(5)
γ /°	79.519(4)	90	90	75.776(5)
Volume/Å ³	2630.6(5)	1960.1(3)	2205.77(19)	1168.6(2)
<i>Z</i>	4	4	4	2
<i>Z'</i>	2	1	0.5	1
ρ_{calc} /mg/mm ³	1.193	1.293	1.284	1.246
μ /mm ⁻¹	0.08	0.092	0.179	0.168
<i>F</i> (000)	1008	808	904	468
Crystal size/mm ³	0.3 × 0.3 × 0.2	0.3 × 0.2 × 0.2	0.3 × 0.2 × 0.2	0.4 × 0.2 × 0.2
2 $\Theta_{\text{min, max}}$	1.7, 51.0°	4.0, 50.2°	3.4, 50.0°	3.0, 50.1°
<i>h</i> min, <i>h</i> max; <i>k</i> min, <i>k</i> max; <i>l</i> min, <i>l</i> max	-8, 10; -14, 14; -30, 30	-12, 12; -17, 20; -13, 13	-11, 7; -28, 25; -11, 11	-10, 10; -11, 11; -16, 16
No. of reflections	26776	14425	9531	7592
No. of independant reflections	9406	3493	1973	4126
<i>R</i> (int)	0.118	0.0159	0.0762	0.0306
Data/restraints/parameters	9406/0/631	3493/0/265	1973/0/140	4126/0/274
Goodness-of-fit on <i>F</i> ²	0.906	1.067	0.959	1.06
<i>R</i> _{obs}	0.071	0.063	0.052	0.104
<i>wR</i> ₂ (obs)	0.142	0.168	0.094	0.303
$\Delta\rho_{\text{max,min}}$ / e Å ⁻³	0.18/-0.19	0.53/-0.30	0.17/-0.18	0.60/-0.50

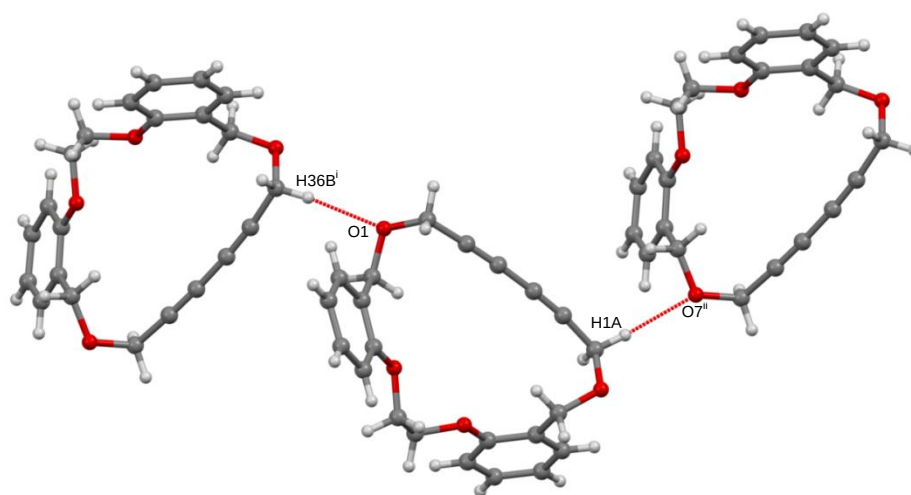


Figure 1. C–H...O hydrogen bonds in the X-ray structure of the compound **5a** with symmetry Codes: (i) $x, \frac{3}{2}-y, -\frac{1}{2}+z$, (ii) $1+x, \frac{3}{2}-y, -\frac{1}{2}+z$.

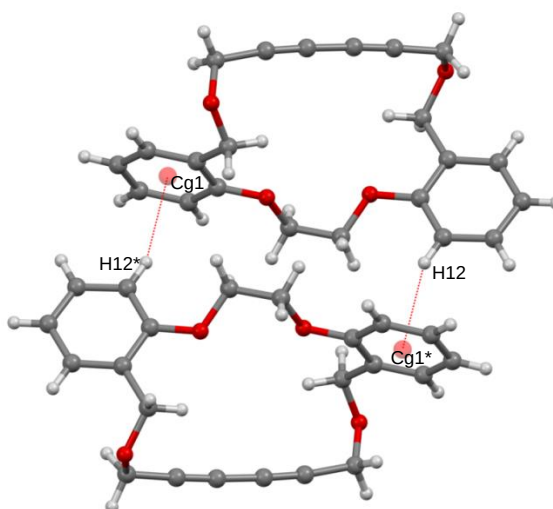


Figure 2. C–H... π interactions in the X-ray structure of the compound **5a** with symmetry Codes: (*) $2-x, 2-y, -z$.

Table 5a_1: Details of C–H...O intermolecular interactions of compounds **5a**

Code	C–H...O	$D(C\cdots O/\text{\AA})$	$d(H\cdots O/\text{\AA})$	$\theta(\angle C-H\cdots O/^\circ)$
5a	C36–H36B...O1	3.337(2)	2.37	173
	C1–H1A...O7	3.312(3)	2.34	174

Table 5a_2: Details of C–H... π intermolecular interactions of compounds **5a**

Code	C–H... π	$C\cdots \pi(\text{\AA})$	$H\cdots \pi(\text{\AA})$	$\theta(\angle C-H\cdots \pi/^\circ)$
5a	C12–H12...Cg1	3.676(4)	2.79	159

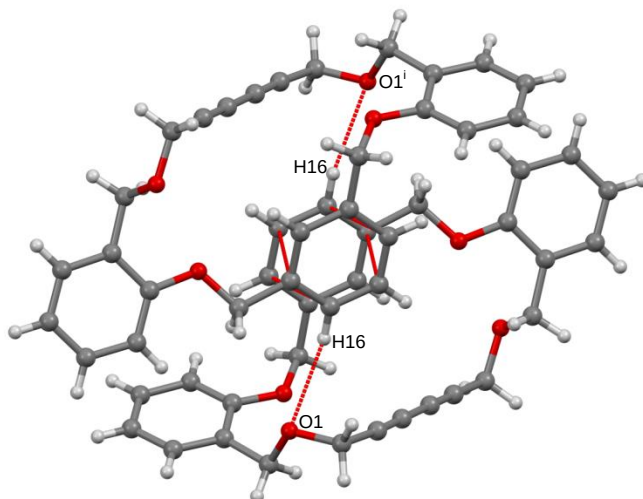


Figure 3. C–H $\cdots$ O hydrogen bonds and $\pi\cdots\pi$ interactions in the X-ray structure of the compound **5f** with Symmetry Codes: (i) 2-x, -y, -z.

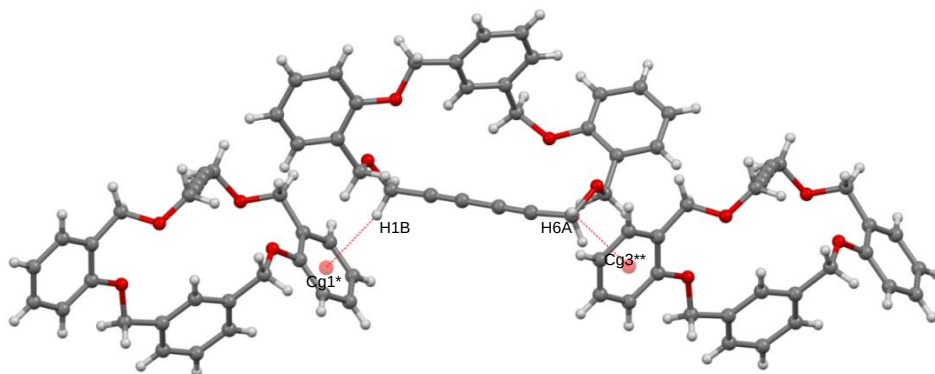


Figure 4. C–H $\cdots\pi$ interactions in the X-ray structure of the compound **5f** with symmetry codes: (*) 2-x, $-1/2+y$, $1/2-z$, (**) 1-x, $1/2+y$, $1/2-z$.

Table 5f_1: Details of C–H $\cdots$ O intermolecular interactions of compounds **5f**

Code	C–H $\cdots$ O	D(C $\cdots$ O/Å)	d (H $\cdots$ O/Å)	$\theta(\angle\text{C–H}\cdots\text{O}/^\circ)$
5f	C16–H16 $\cdots$ O1	3.379(3)	2.56	148

Table 5f_2: Details of $\pi\cdots\pi$ interactions of the compounds **5f**

Code	$\pi\cdots\pi$	Cg1 $\cdots$ Cg2 (Å)	(Cg1 $\cdots$ Per) (Å)	(Cg2 $\cdots$ Per) (Å)	Slippage	Symmetry Code
5f	Cg1 $\cdots$ Cg2	3.592(2)	3.2527(6)	3.2527(6)	1.523	2-x, -y, -z

Table 5f_3: Details of C–H $\cdots\pi$ interactions of the compounds **5f**

Code	C–H $\cdots\pi$	C $\cdots\pi$ (Å)	H $\cdots\pi$ (Å)	$\theta(\angle\text{C–H}\cdots\pi/^\circ)$
5f	C1–H1B $\cdots$ Cg1	3.389(2)	2.92	111
	C6–H6A $\cdots$ Cg3	3.662(2)	2.82	147

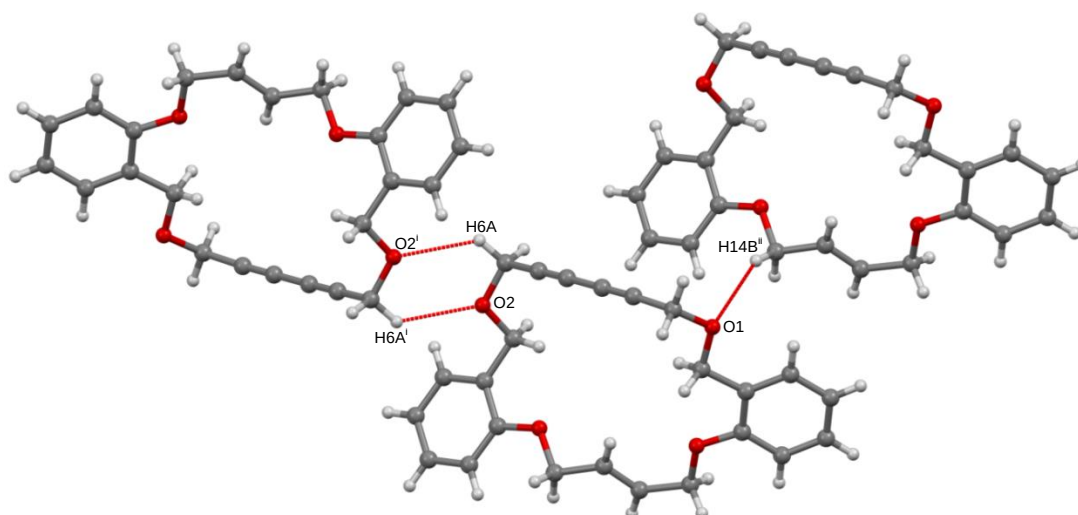


Figure 5. C–H···O hydrogen bonds in the X-ray structure of the compound **5g** with symmetry codes: (i) -x, 1-y, -z (ii) 1+x, y, z .

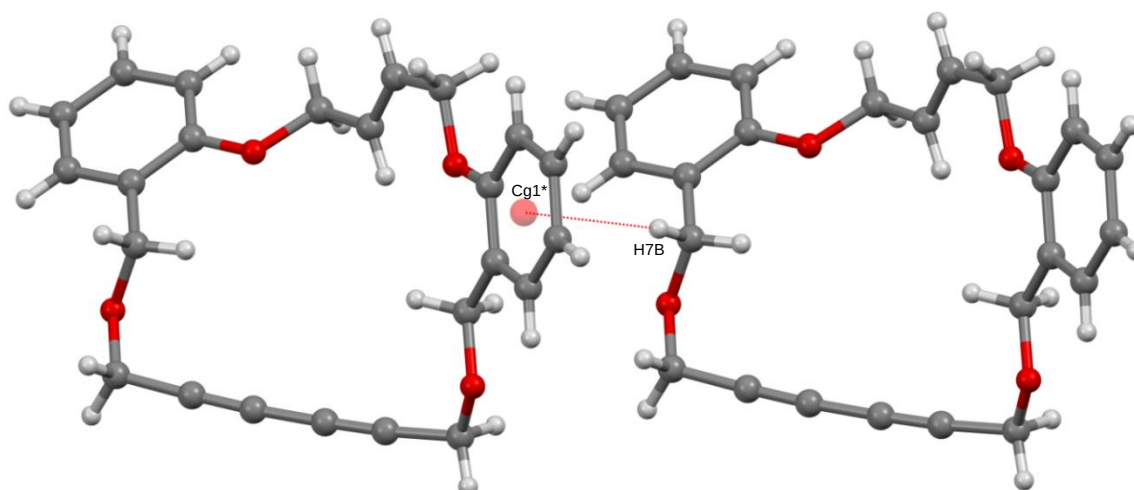


Figure 6. C–H··· π interactions in the X-ray structure of the compound **5g** with symmetry codes: (*) -x, -y, $\frac{1}{2}$ -z.

Table 5g_1: Details of C–H···O intermolecular interactions of compounds **5g**

Code	C–H···O	D(C···O/Å)	<i>d</i> (H···O/Å)	$\theta(\angle\text{C–H}\cdots\text{O}/^\circ)$
5g	C16–H6A···O2	3.240(4)	2.51	129
	C14–H14B···O1	3.496(5)	2.62	150

Table 5g_2: Details of C–H··· π interactions of the compounds **5g**

Code	C–H··· π	C··· π (Å)	H··· π (Å)	$\theta(\angle\text{C–H}\cdots\pi/^\circ)$
5g	C7–H7B···Cg1	3.369(3)	2.82	109

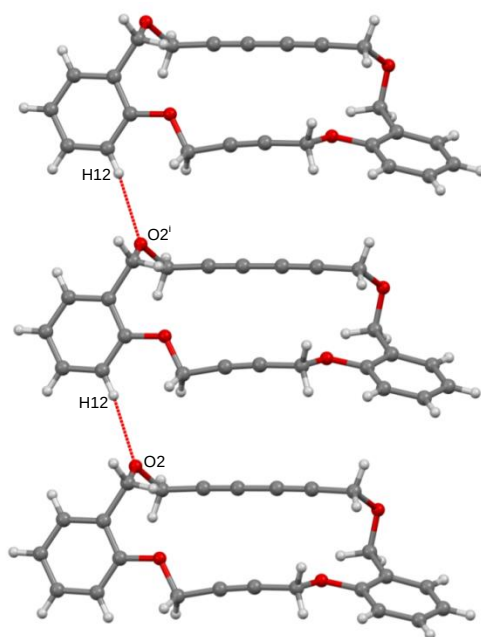


Figure 7. C–H $\cdots$ O hydrogen bonds in the X-ray structure of the compound **5h** with symmetry codes: (i) x, y, -1+z .

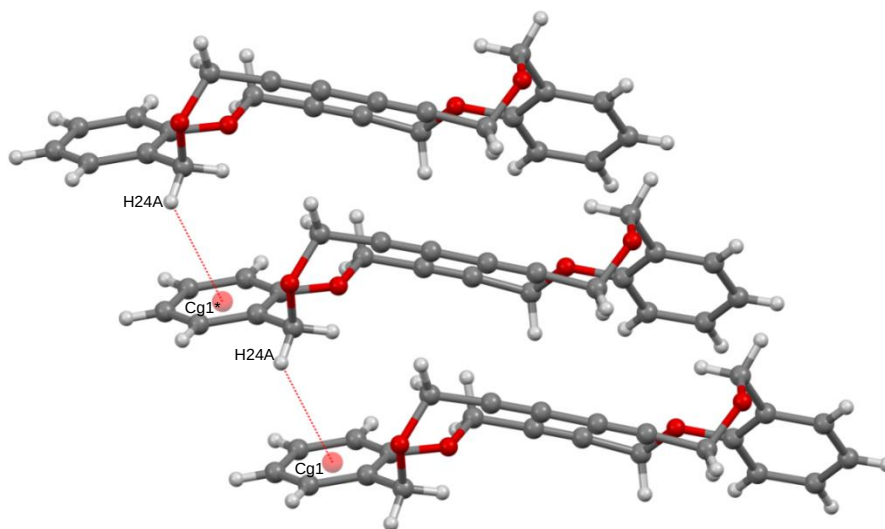


Figure 8. C–H $\cdots$ π interactions in the X-ray structure of the compound **5h** with symmetry codes: (*) 1+x, y, z.

Table 5h_1: Details of C–H $\cdots$ O intermolecular interactions of compounds **5h**

Code	C–H $\cdots$ O	D (C $\cdots$ O/Å)	d (H $\cdots$ O/Å)	θ ($\angle$ C–H $\cdots$ O/ $^\circ$)
5h	C12–H12 $\cdots$ O2	3.385(4)	2.50	158

Table 5h_2: Details of C–H $\cdots$ π interactions of the compounds **5h**

Code	C–H $\cdots$ π	C $\cdots$ π (Å)	H $\cdots$ π (Å)	θ ($\angle$ C–H $\cdots$ π / $^\circ$)
5h	C24–H24A $\cdots$ Cg1	3.431(2)	2.91	114

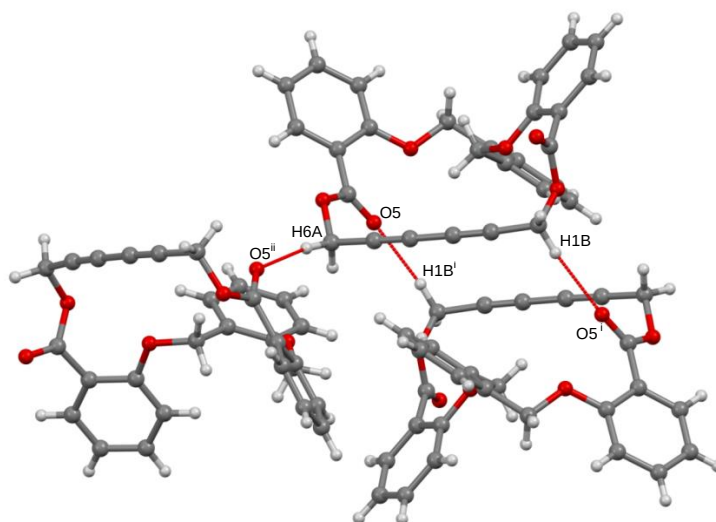


Figure 9. C–H···O hydrogen bonds in the X-ray structure of the compound **5i** with symmetry codes: (i) 1-x, -y, -z (ii) 1-x, -1/2+y, 1/2-z.

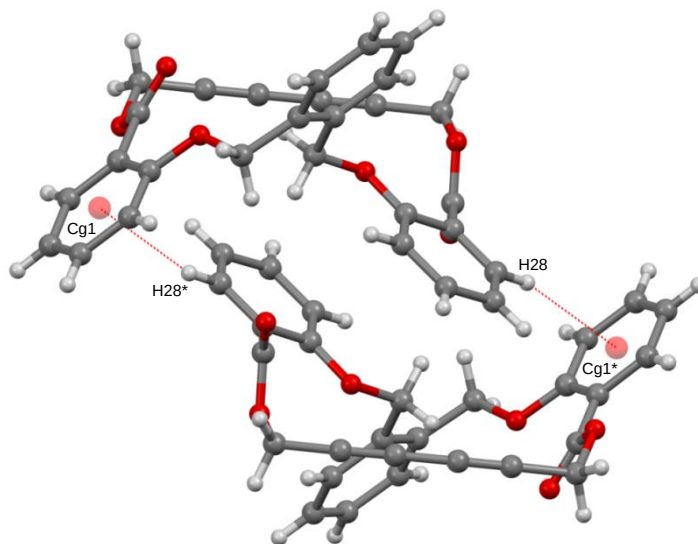


Figure 10. C–H··· π interactions in the X-ray structure of the compound **5i** with symmetry codes: (*) -x, -y, -z.

Table 5i_1: Details of C–H···O intermolecular interactions of compounds **5i**

Code	C–H···O	D (C···O/Å)	d (H···O/Å)	θ ($\angle$ C–H···O/ $^\circ$)
5i	C1–H1B···O5	3.262(3)	2.35	156
	C6–H6A···O5	3.315(4)	2.36	169

Table 5i_2: Details of C–H··· π interactions of the compounds **5i**

Code	C–H··· π	C··· π (Å)	H··· π (Å)	θ ($\angle$ C–H··· π / $^\circ$)
5i	C28–H28···Cg1	3.564(3)	2.68	159

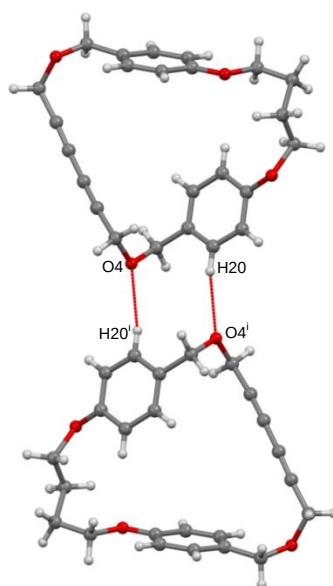


Figure 11. C–H $\cdots$ O hydrogen bonds in the X-ray structure of the compound **5k** with symmetry code: (i) 2-x, 1-y, -z.

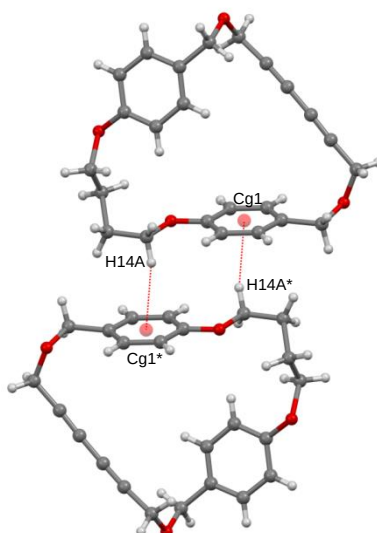


Figure 12. C–H $\cdots$ π interactions in the X-ray structure of the compound **5k** with symmetry code: (*) -x, -y, 1-z.

Table 5k_1: Details of C–H $\cdots$ O intermolecular interactions of compounds **5k**

Code	C–H $\cdots$ O	D (C $\cdots$ O/Å)	d (H $\cdots$ O/Å)	θ ($\angle$ C–H $\cdots$ O/ $^\circ$)
5k	C20–H20 $\cdots$ O4	3.436(4)	2.59	152

Table 5k_2: Details of C–H $\cdots$ π interactions of the compounds **5k**

Code	C–H $\cdots$ π	C $\cdots$ π (Å)	H $\cdots$ π (Å)	θ ($\angle$ C–H $\cdots$ π / $^\circ$)
5k	C14–H14A $\cdots$ Cg1	3.711(4)	2.97	134

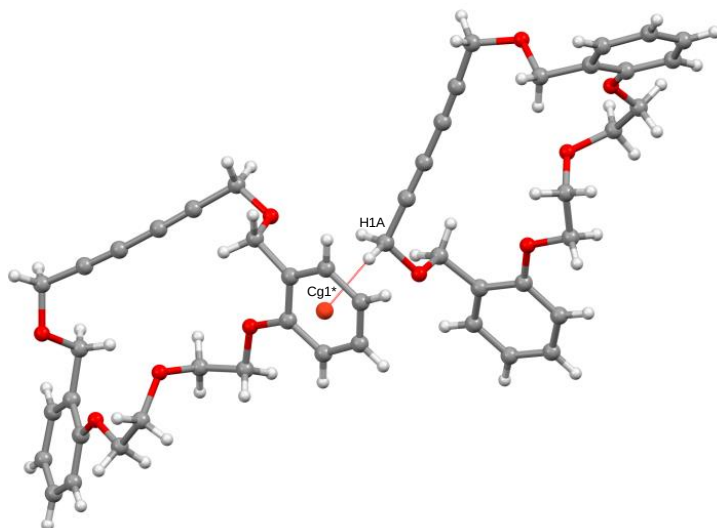


Figure 13. C–H $\cdots\pi$ interactions in the X-ray structure of the compound **6a** with symmetry code: (*) 2-x, $\frac{1}{2}$ +y, $\frac{1}{2}$ -z.

Table 6a: Details of C–H $\cdots\pi$ interactions of the compounds **6a**

Code	C–H $\cdots\pi$	C $\cdots\pi$ (Å)	H $\cdots\pi$ (Å)	θ ($\angle$ C–H $\cdots\pi$ / $^\circ$)
6a	C1–H1A $\cdots$ Cg1	3.739(5)	2.77	154

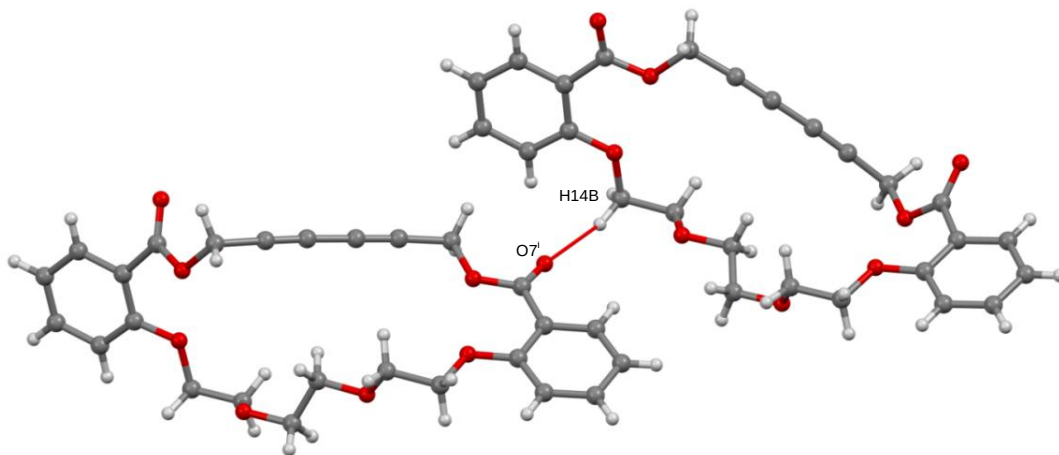


Figure 14. C–H $\cdots$ O hydrogen bonds in the X-ray structure of the compound **6g** with symmetry code: (i) 2-x, $-\frac{1}{2}$ +y, $\frac{1}{2}$ -z.

Table 6g: Details of C–H $\cdots$ O intermolecular interactions of compounds **6g**

Code	C–H $\cdots$ O	D(C $\cdots$ O/Å)	<i>d</i> (H $\cdots$ O/Å)	θ ($\angle$ C–H $\cdots$ O/ $^\circ$)
6g	C14–H14B $\cdots$ O7	3.288(4)	2.37	158

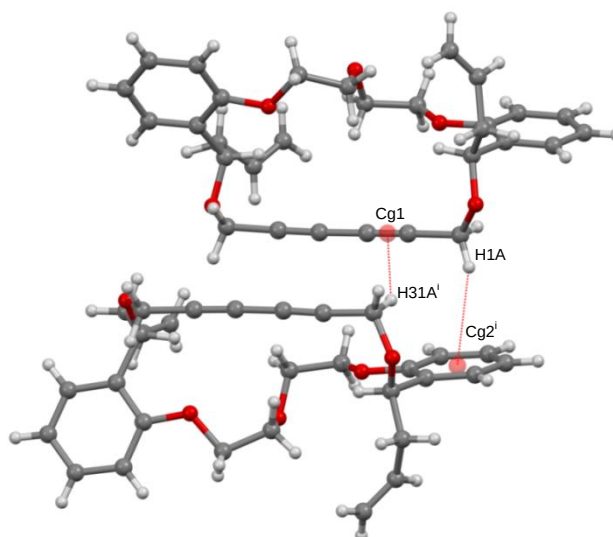


Figure 15. C–H $\cdots\pi$ interactions within both the asymmetric units of the compound **7d** with symmetry code: (i) x, y, z.

Table 7d: Details of C–H $\cdots\pi$ interactions of the compounds **7d**

Code	C–H $\cdots\pi$	C $\cdots\pi$ (Å)	H $\cdots\pi$ (Å)	θ ($\angle$ C–H $\cdots\pi$ / $^\circ$)
7d	C1–H1A $\cdots$ Cg2	3.711(6)	2.86	147
	C31–H31A $\cdots$ Cg1	3.728(4)	2.90	144

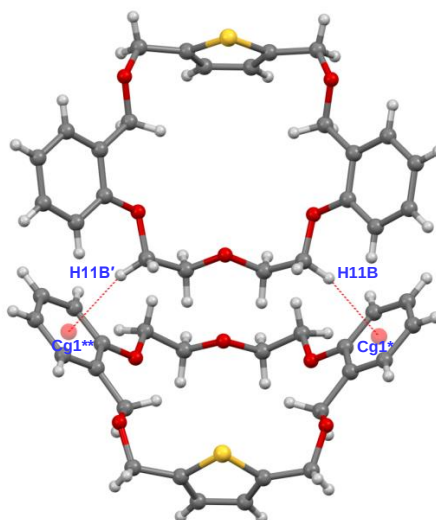


Figure 16. C–H $\cdots\pi$ interactions in the X-ray structure of the compound **9a** with symmetry code: (*) $\frac{1}{2}+x, y, \frac{3}{2}-z$; (') x, $\frac{3}{2}-y, z$; (**) $\frac{1}{2}+x, \frac{3}{2}-y, \frac{3}{2}-z$.

Table 9a: Details of C–H $\cdots\pi$ interactions of the compounds **9a**

Code	C–H $\cdots\pi$	C $\cdots\pi$ (Å)	H $\cdots\pi$ (Å)	θ ($\angle$ C–H $\cdots\pi$ / $^\circ$)
9a	C11–H11B $\cdots$ Cg1	3.508(3)	2.57	164

References

- (a) G. Kaur and A. R. Choudhury, *Cryst. Growth Des.*, 2014, DOI: 10.1021/cg401573d.
- (b) P. A. Wood, F. H. Allen and E. Pidcock, *CrystEngComm*, 2009, **11**, 1563.

All Experimental Procedures and Characterization Data of all Compounds

Typical procedure for the synthesis of *bis*-aldehydes 2 (Procedure A). To a flame dried round-bottom flask was sequentially added the corresponding phenol derivative (12 mmol) dry DMF (20 mL), and anhydrous K₂CO₃ (20 mmol, 2.780 g). The reaction mixture was stirred at 80 °C for 15 min. After 15 min, the temperature of the reaction bath was increased to 110 °C and the corresponding alkyl dibromide or alkyl dichloride (5 mmol) was added in one portion to the hot reaction mixture. The resulting reaction mixture was stirred at 110 °C for 12 h and after this period, the reaction mixture was allowed to attain the room temperature, which was then added to ice flakes (15-25 g). The resulting solid compound (*bis*-aldehyde) was filtered through a filtration funnel and used in the next step without further purification. In case, if the *bis*-aldehyde is liquid; then the reaction mixture (after the treatment with ice flakes/cold water) was extracted using ethyl acetate (3 X 10 mL). The combined organic layers were dried over anhydrous Na₂SO₄, evaporated under vacuum and used as such (without further purification) in the next step.

The compounds **2a**,⁸ **2b**,⁸ **2c**,⁹ **2d**,¹⁰ **2e**,¹¹ **2f**,¹¹ **2g**,¹² **2h**,¹³ **2i**,¹⁴ **2j**,¹⁵ **2k**,¹⁶ **2l**,¹⁷ **2n**,¹⁸ **2o**,¹⁹ **2p**,²⁰ **2r**,²⁵ **3a**,²¹ **3b**,²² **3c**,²² **3f**,²³ **3g**,¹² **3j**,²⁴ are reported in the literature.

6,6'-((Oxybis(ethane-2,1-diyl))bis(oxy))bis(3-bromobenzaldehyde) (2m): Following the general procedure A, **2m** was obtained after filtration through a filtration funnel as brown a solid mp: 141-143 °C; Yield: 4.127 g, 88%; IR (CH₂Cl₂): 2949, 1681, 1591, 1478, 1241, 1123, 808 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 10.41 (s, 2H), 7.93 (d, *J* = 2.6 Hz, 2H), 7.62 (dd, *J* = 6.24, 2.6 Hz, 2H), 6.91 (d, *J* = 8.8 Hz, 2H), 4.27 (t, *J* = 5.5 Hz, 4H), 3.99 (t, *J* = 4.56 Hz, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 188.2, 159.9, 138.3, 131.1, 126.3, 114.9, 114.0, 69.8, 68.6; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₁₈H₁₆Br₂O₅Na: 492.9262; found 492.9256.

General procedure for the synthesis of *bis*-alcohols 3a-p (Procedure B). To a mixture of *bis*-aldehyde **2** (3 mmol) in ethanol (7 mL) was added NaBH₄ (10 mmol) at room temperature. The resulting mixture was stirred at room temperature for 30 min. After this period, the reaction mixture was poured on to cold water (20 mL) or crushed ice (20 g). Then, the resulting solid compound was filtered through a filtration funnel and used without further purification. In case, if the *bis*-alcohol **3** is liquid; then the reaction mixture (after the treatment with ice flakes/cold water) was extracted using ethyl acetate (3 X 10 mL). The combined organic layers were dried over anhydrous Na₂SO₄, evaporated under vacuum and used as such (without further purification) in the next step.

((Ethane-1,2-diylbis(oxy))bis(2,1-phenylene))dimethanol (3a): Following the general procedure B, **3a** was obtained after filtration through a filtration funnel as a white solid, mp: 125-127 °C ; Yield: 0.674 g, 82%; IR (CH₂Cl₂): 3399, 2233, 1600, 1490, 1028 and 730 cm⁻¹; ¹H NMR (400 MHz, CDCl₃ and DMSO): δ 7.36-7.32 (m, 2H), 7.24-7.19 (m, 2H), 6.98-6.87 (m, 4H), 4.61 (s, 4H), 4.34 (s, 4H), ¹³C NMR (100 MHz, CDCl₃ and DMSO): δ 155.9, 130.2, 128.5, 128.3, 121.0, 111.4, 67.7, 60.2; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₁₆H₁₈O₄Na: 297.1103; found 297.1115. (In ¹H NMR OH protons could not be detected).

((Butane-1,4-diylbis(oxy))bis(2,1-phenylene))dimethanol (3b): Following the general procedure B, **3b** was obtained after filtration through a filtration funnel as a white solid, mp: 91-93 °C ; Yield: 1.11 g, 92%; IR (CH₂Cl₂): 3398, 2231, 1601, 1491, 1027 and 730 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.28-7.22 (m, 4H), 6.93 (t, *J* = 7.3 Hz, 2H), 6.86 (d, *J* = 8.1 Hz, 2H), 4.65 (s, 4H), 4.09 (s, 4H), 2.49 (brs, 2H), 2.01 (brs, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 155.7, 129.2, 128.9, 128.8, 120.8, 111.1, 67.4, 61.9, 26.2; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₁₈H₂₂O₄Na: 325.1416; found 325.1427.

((Hexane-1,6-diylbis(oxy))bis(2,1-phenylene))dimethanol (3c): Following the general procedure B, **3c** was obtained after filtration through filtration funnel as a brown solid, mp: 60-62 °C ; Yield: 1.036 g, 80%; IR (CH₂Cl₂): 3396, 2231, 1602, 1493, 1030 and 730 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.26-7.22 (m, 4H), 6.91 (t, *J* = 7.4 Hz, 2H), 6.85 (d, *J* = 8.1 Hz, 2H), 4.65 (s, 4H), 4.02 (t, *J* = 6.2 Hz, 4H), 2.44 (brs, 2H), 1.86-1.83 (m, 4H), 1.57-1.54 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 156.8, 129.2, 128.9, 128.7, 120.6, 111.1, 67.7, 62.2, 29.2, 25.9; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₂₀H₂₆O₄Na: 353.1729; found 353.1737.

((Butane-1,4-diylbis(oxy))bis(naphthalene-2,1-diyl))dimethanol (3d): Following the general procedure B, **3d** was obtained after filtration through a filtration funnel as a brown solid, mp: 161-163 °C ; Yield: 1.025 g, 85%; IR (CH₂Cl₂): 3364, 2921, 1602, 1589, 1128, 1491 and 730 cm⁻¹; ¹H NMR (400 MHz, CDCl₃ and DMSO): δ 8.17 (s, 2H), 7.97 (s, 4H), 7.50-7.28 (m, 6H), 5.14 (s, 4H), 4.24 (s, 4H), 2.12 (s, 4H); ¹³C NMR (100 MHz, CDCl₃ and DMSO): δ 154.2, 133.1, 129.6, 129.0, 128.1, 126.6, 123.6, 123.5, 122.1, 114.7, 69.2, 54.8, 26.3. HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₂₆H₂₆O₄Na: 425.1729; found 425.1734. (In ¹H NMR OH protons could not be detected may be it is merged with the residual DMSO signal).

((((1,2-Phenylenebis(methylene))bis(oxy))bis(2,1-phenylene))dimethanol (3e): Following the general procedure B, **3e** was obtained after filtration through a filtration funnel as a brown solid, mp: 95-97 °C; Yield: 0.903 g, 88%; IR (CH₂Cl₂): 3364, 3067, 2877, 1602, 1589 and 730 cm⁻¹; ¹H NMR (400 MHz, CDCl₃ and DMSO): δ 7.52-7.50 (m, 2H), 7.38-7.36 (m, 4H), 7.22 (t, *J* = 7.7 Hz, 2H), 6.97-6.92 (m, 4H), 5.19 (s, 4H), 4.66 (s, 4H); ¹³C NMR (100 MHz, CDCl₃ and DMSO): δ 155.9, 134.9, 129.9, 128.9, 128.5, 128.4, 120.9, 111.4, 67.8, 60.5. HRMS (ESI): *m/z* [M + H]⁺ calcd for C₂₂H₂₃O₄: 351.1596; found 351.1602. (In ¹H NMR OH protons could not be detected may be it is merged with the residual DMSO signal).

((((1,3-Phenylenebis(methylene))bis(oxy))bis(2,1-phenylene))dimethanol (3f): Following the general procedure B, **3f** was obtained after filtration through a filtration funnel as a brown solid, mp: 82-84 °C; Yield: 0.882 g, 84%; IR (CH₂Cl₂): 3364, 2921, 1602, 1589, 1128, 1491 and 730 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.52 (s, 1H), 7.40-7.30 (m, 5H), 7.26-7.22 (m, 2H), 6.97-6.90 (m, 4H), 5.12 (s, 4H), 4.71 (s, 4H), 2.69 (brs, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 156.3, 137.4, 129.4, 128.9, 128.9, 126.8, 125.7, 121.1, 111.6, 69.7, 61.8. HRMS (ESI): *m/z* [M + H]⁺ calcd for C₂₂H₂₂O₄Na: 373.1416; found 373.1425.

(E)-((But-2-ene-1,4-diylbis(oxy))bis(2,1-phenylene))dimethanol (3g): Following the general procedure B, **3g** was obtained after filtration through a filtration funnel as a brown solid, mp: 95-96 °C; Yield: 0.774 g, 86%; IR (CH₂Cl₂): 3353, 2918, 1602, 1489 and 752 cm⁻¹; ¹H NMR (400 MHz, CDCl₃ and DMSO): δ 7.37-6.82 (m, 8H), 6.05 (s, 2H), 4.62 (d, *J* = 3.1 Hz, 8H), 3.98 (brs,

2H); ^{13}C NMR (100 MHz, CDCl_3 and DMSO): δ 160.4, 135.0, 132.9, 132.8, 132.7, 125.5, 116.1, 72.4, 64.8. HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{18}\text{H}_{20}\text{O}_4\text{Na}$: 323.1259; found 323.1270. (In ^1H NMR OH protons could not be detected may be it is merged with the residual DMSO signal).

((But-2-yne-1,4-diylbis(oxy))bis(2,1-phenylene))dimethanol (3h): Following the general procedure B, **3h** was obtained after filtration through a filtration funnel as a brown solid, mp: 96-98 °C; Yield: 0.795 g, 89%; IR (CH_2Cl_2): 3381, 2926, 1622, 1465 and 738 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 and DMSO): δ 7.24 (d, J = 7.3 Hz, 2H), 7.12 (t, J = 7.8 Hz, 2H), 6.89 (t, J = 7.4 Hz, 2H), 6.81 (d, J = 8.6 Hz, 2H), 4.66 (s, 4H), 4.57 (s, 4H); ^{13}C NMR (100 MHz, CDCl_3 and DMSO): δ 155.2, 130.0, 128.8, 128.6, 121.6, 111.9, 82.4, 61.2, 56.2. HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{18}\text{H}_{18}\text{O}_4\text{Na}$: 321.1103; found 321.1117. (In ^1H NMR OH protons could not be detected may be it is merged with the residual DMSO signal).

((Ethane-1,2-diylbis(oxy))bis(3,1-phenylene))dimethanol (3j): Following the general procedure B, **3j** was obtained after filtration through filtration a funnel as a brown solid, mp: 128-130 °C; Yield: 0.674 g, 82%; IR (CH_2Cl_2): 3398, 2231, 1601, 1491, 1027 and 730 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 and DMSO): δ 7.29-6.74 (m, 8H), 4.54 (s, 4H), 4.25 (s, 4H); ^{13}C NMR (100 MHz, CDCl_3 and DMSO): δ 158.7, 143.5, 129.3, 119.4, 113.5, 112.8, 66.4, 64.3; HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{16}\text{H}_{18}\text{O}_4\text{Na}$: 297.1103; found 297.1111. (In ^1H NMR OH protons could not be detected may be it is merged with the residual DMSO signal).

((Butane-1,4-diylbis(oxy))bis(4,1-phenylene))dimethanol (3k): Following the general procedure B, **3k** was obtained after filtration through a filtration funnel as a brown solid, mp: 160-162 °C ; Yield: 1.063 g, 88%; IR (CH_2Cl_2): 3398, 2231, 1601, 1491, 1027 and 730 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 and DMSO): δ 7.15 (d, J = 8.5 Hz, 4H), 6.73 (d, J = 8.5 Hz, 4H), 4.44 (d, J = 5.1Hz, 4H), 3.90 (s, 4H), 3.82 (t, J = 5.5 Hz, 2H), 1.84-1.82 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3 and DMSO): δ 158.1, 133.9, 128.4, 114.2, 67.4, 64.1, 25.9; HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{18}\text{H}_{22}\text{O}_4\text{Na}$: 325.1416; found 325.1428. (In ^1H NMR OH protons could not be detected may be it is merged with the residual DMSO signal).

((((Oxybis(ethane-2,1-diyl))bis(oxy))bis(2,1-phenylene))dimethanol (3l): Following the general procedure B, **3l** was obtained after filtration through a filtration funnel as a brown solid, mp: 52-54 °C; Yield: 0.858 g, 90%; IR (CH_2Cl_2): 3405, 2939, 2365, 1687, 1492 and 754 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.25-7.20 (m, 4H), 6.92 (t, J = 7.4 Hz, 2H), 6.84 (d, J = 8.8 Hz, 2H), 4.61 (s, 4H), 4.20-4.18 (m, 4H), 4.03 (brs, 2H), 3.89-3.86 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ 156.9, 130.2, 129.7, 129.0, 121.3, 112.2, 69.8, 67.8, 61.8. HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{18}\text{H}_{22}\text{O}_5\text{Na}$: 341.1365; found 341.1374.

((((Oxybis(ethane-2,1-diyl))bis(oxy))bis(3-bromo-6,1-phenylene))dimethanol (3m): Following the general procedure B, **3m** was obtained after filtration through a filtration funnel as a brown solid, mp: 88-90 °C; Yield: 1.248 g, 88%; IR (CH_2Cl_2): 3401, 2931, 1453, 1242, and 730 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 and DMSO): δ 7.28-7.12 (m, 4H), 6.59-6.54 (m, 2H), 4.42 (s, 4H), 4.10 (s, 2H), 3.98 (s, 4H), 3.72 (s, 4H); ^{13}C NMR (100 MHz, CDCl_3 and DMSO): δ 155.4, 132.6, 131.4, 130.9, 113.6, 113.3, 69.6, 67.9, 60.2; HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{18}\text{H}_{20}\text{Br}_2\text{O}_5\text{Na}$: 496.9575; found 496.9576. (In ^1H NMR OH protons could not be detected may be it is merged with the residual DMSO signal).

(((Ethane-1,2-diylbis(oxy))bis(ethane-2,1-diyl))bis(oxy))bis(2,1-phenylene))dimethanol

(3n): Following the general procedure B, **3n** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 40:60) as colourless liquid; Yield: 0.955 g, 88%; IR (CH₂Cl₂): 3414, 2874, 1602, 1492 and 753 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.28-7.24 (m, 4H), 6.95 (t, *J* = 7.4 Hz, 2H), 6.88 (d, *J* = 8.4 Hz, 2H), 4.64 (s, 4H), 4.20-4.18 (m, 4H), 3.87-3.85 (m, 4H), 3.73 (s, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 157.1, 130.2, 129.3, 128.9, 121.2, 112.3, 70.7, 69.6, 67.8, 62.1; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₂₀H₂₆O₆Na: 385.1627; found 385.1639. (In ¹H NMR OH protons could not be detected).

(((Oxybis(ethane-2,1-diyl))bis(oxy))bis(ethane-2,1-diyl))bis(oxy))bis(2,1-phenylene))dimethanol (3o):

Following the general procedure B, **3o** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 50:50) as a colorless liquid; Yield: 1.12 g, 92%; IR (CH₂Cl₂): 3402, 2935, 1455, 1242, and 730 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.28-7.24 (m, 4H), 6.97-6.87 (m, 4H), 4.65 (s, 4H), 4.20-4.18 (m, 4H), 3.86-3.84 (m, 4H), 3.72-3.66 (m, 8H); ¹³C NMR (100 MHz, CDCl₃): δ 156.9, 130.2, 129.3, 128.9, 121.2, 112.2, 70.6, 70.5, 69.6, 67.8, 61.9; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₂₂H₃₀O₇Na: 429.1889; found 429.1894. (In ¹H NMR OH protons could not be detected).

(((Oxybis(ethane-2,1-diyl))bis(oxy))bis(naphthalene-2,1-diyl))dimethanol (3p): Following the general procedure B, **3p** was obtained after filtration through a filtration funnel as a brown solid, mp: 124-126 °C; Yield: 1.065 g, 85%; IR (CH₂Cl₂): 3401, 2934, 1454, 1240, and 728 cm⁻¹; ¹H NMR (400 MHz, CDCl₃ and DMSO): δ 8.11 (d, *J* = 8.5 Hz, 2H), 7.77 (d, *J* = 9.1 Hz, 4H), 7.48 (t, *J* = 8.1 Hz, 2H), 7.35 (t, *J* = 7.8 Hz, 2H), 7.21 (d, *J* = 9.1 Hz, 2H), 5.11 (s, 4H), 4.35-4.33 (m, 4H), 3.95-3.93 (m, 4H); ¹³C NMR (100 MHz, CDCl₃ and DMSO): δ 154.2, 132.8, 129.8, 129.6, 128.3, 126.9, 123.9, 123.4, 123.3, 114.9, 70.1, 69.1, 55.1; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₂₆H₂₆O₅Na: 441.1678; found 441.1680. (In ¹H NMR OH protons could not be detected may be it is merged with the residual DMSO signal).

General procedure for the syntheses of bis-alcohols 3s-w (Procedure C). To a mixture of corresponding bis-aldehyde **3** (3 mmol) and allyl bromide (7 equiv) in THF (7 mL) was added saturated NH₄Cl solution (18 mL) and Zn metal (5 equiv) successively at room temperature. The resulting mixture was stirred at room temperature for 30 h. After this period, the reaction mixture was extracted by using ethyl acetate (3 X 7 mL). The combined organic layers were dried over anhydrous Na₂SO₄, the organic phase was concentrated and the resulting crude reaction mixture was purified by silica gel column chromatography (EtOAc : hexanes = 30:70) which afforded the corresponding products **3s-w**.

1,1'-((Ethane-1,2-diylbis(oxy))bis(2,1-phenylene))bis(but-3-en-1-ol) (3s): Following the general procedure C, **3s** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 30:70) as a white solid, mp: 66-68 °C; Yield: 0.849 g, 80%; IR (CH₂Cl₂): 3413, 2937, 1600, 1453 and 751 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.38-7.35 (m, 2H), 7.24 (t, *J* = 7.8 Hz, 2H), 6.99 (t, *J* = 7.5 Hz, 2H), 6.91 (d, *J* = 8.2 Hz, 2H), 5.81-5.71 (m, 2H), 5.08-4.96 (m, 6H), 4.40-4.38 (m, 4H), 2.90 (brs, 2H), 2.58-2.46 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 155.3, 135.1, 132.4, 128.3, 127.1, 121.4, 117.7, 111.5, 68.7, 66.9, 41.7; HRMS (ESI): *m/z* [M +

$\text{Na}]^+$ calcd for $\text{C}_{22}\text{H}_{26}\text{O}_4\text{Na}$: 377.1729; found 377.1740. (Isolated as a 1:1 mixture of diastereomers and ^{13}C values given here for one isomer).

1,1'-((Butane-1,4-diylbis(oxy))bis(2,1-phenylene))bis(but-3-en-1-ol) (3t): Following the general procedure C, **3t** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 30:70) as a white solid, mp: 57-59 °C; Yield: 1.008 g, 88%; IR (CH_2Cl_2): 3413, 3073, 2875, 1588 and 754 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.34 (dd, J = 5.9, 1.56 Hz, 2H), 7.23-7.19 (m, 2H), 6.95 (t, J = 7.4 Hz, 2H), 6.85 (d, J = 7.6 Hz, 2H), 5.86-5.75 (m, 2H), 5.14-5.06 (m, 4H), 4.99-4.96 (m, 2H), 4.09-4.07 (m, 4H), 2.57-2.47 (m, 6H), 2.02-1.99 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ 155.5, 135.2, 131.9, 128.3, 126.8, 120.8, 117.6, 111.1, 69.4, 67.3, 42.0, 26.3; HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{24}\text{H}_{30}\text{O}_4\text{Na}$: 405.2042; found 405.2043. (In ^1H NMR OH protons could not be detected. This compound was isolated as a 1:1 mixture of diastereomers and ^{13}C values given here for one isomer).

1,1'-(((1,3-Phenylenebis(methylene))bis(oxy))bis(2,1-phenylene))bis(but-3-en-1-ol) (3u): Following the general procedure C, **3u** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 30:70) as a brown liquid; Yield: 1.057 g, 82%; IR (CH_2Cl_2): 3397, 3073, 2917, 1640, and 752 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.54 (s, 1H), 7.47-7.41 (m, 5H), 7.26 (t, J = 7.6 Hz, 2H), 7.04-6.94 (m, 4H), 5.89-5.85 (m, 2H), 5.30-5.09 (m, 12H), 2.92 (brs, 2H), 2.66-2.53 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ 155.2, 137.5, 135.2, 132.3, 129.1, 128.3, 126.9, 126.9, 126.7, 125.7, 121.1, 117.7, 111.7, 69.7, 69.1, 42.1; HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{28}\text{H}_{30}\text{O}_4\text{Na}$: 453.2041; found 453.2044. (Isolated as a 1:1 mixture of diastereomers and ^{13}C values given here for one isomer).

1,1'-(((Oxybis(ethane-2,1-diyl))bis(oxy))bis(2,1-phenylene))bis(but-3-en-1-ol) (3v): Following the general procedure C, **3v** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 50:50) as a colourless liquid; Yield: 0.955 g, 80%; IR (CH_2Cl_2): 3416, 3073, 1640, 1601 and 753 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.28-7.17 (m, 4H), 6.94 (t, J = 7.4 Hz, 2H), 6.86 (d, J = 8.1 Hz, 2H), 5.82-5.71 (m, 2H), 5.07-4.99 (m, 4H), 4.92-4.87 (m, 2H), 4.21-4.15 (m, 4H), 3.90 (t, J = 4.5 Hz, 4H), 3.33 (t, J = 5.1 Hz, 2H), 2.57-2.53 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ 155.8, 135.4, 132.3, 128.3, 127.3, 121.2, 117.2, 112.1, 70.3, 69.8, 67.6, 41.5; HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{24}\text{H}_{30}\text{O}_5\text{Na}$: 421.1991; found 421.1997. (In ^1H NMR OH protons could not be detected. This compound was isolated as a 1:1 mixture of diastereomers and ^{13}C values given here for one isomer).

1,1'-((((Ethane-1,2-diylbis(oxy))bis(ethane-2,1-diyl))bis(oxy))bis(2,1-phenylene))bis(but-3-en-1-ol) (3w): Following the general procedure C, **3w** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 50:50) as a colourless liquid; Yield: 1.169 g, 82%; IR (CH_2Cl_2): 3416, 2876, 1640, 1587 and 751 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.31-7.23 (m, 4H), 6.97-6.87 (m, 4H), 5.90-5.79 (m, 2H), 5.13-5.05 (m, 4H), 4.92-4.88 (m, 2H), 4.19-4.17 (m, 4H), 3.88-3.85 (m, 4H), 3.73 (s, 4H), 3.39 (brs, 2H), 2.63-2.59 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ 155.9, 135.2, 132.4, 132.3, 128.3, 127.3, 121.1, 117.1, 112.2, 112.2, 70.8, 70.6, 69.7, 67.7, 67.6, 41.5; HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{26}\text{H}_{34}\text{O}_6\text{Na}$: 465.2253; found 465.2257. (This compound was isolated as a 1:1 mixture of diastereomers and ^{13}C values given here for one isomer).

General procedure for the syntheses of compounds 4a-h, 4j-p and 4s-w (Procedure D). To a solution of corresponding *bis*-alcohol **3** (1 mmol) (synthesized in the previous steps by using the procedure B or C) in dry THF (3 mL) was added NaH (4 mmol, 55-60 % suspension in mineral oil) at room temperature. The mixture was stirred at room temperature for 10 min and then propargyl bromide (5 mmol, 80 wt% in toluene) was added. The resulting mixture was stirred for 20 h at room temperature. After this period, few drops of EtOH was added and stirred for 10 min and then the resulting mixture was poured on to water (20 mL) and was extracted by using ethyl acetate (3 X 10 mL). The combined organic layers were dried over anhydrous Na₂SO₄ and the organic phase was concentrated and the resulting crude reaction mixture was purified by silica gel column chromatography (EtOAc : Hexanes = 20:80) to give the corresponding product **4**.

1,2-Bis(2-((prop-2-yn-1-yloxy)methyl)phenoxy)ethane (4a): Following the general procedure D, **4a** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a white solid, mp: 64-66 °C; Yield: 0.262 g, 75%; IR (CH₂Cl₂): 2943, 2883, 1602, 1493, 1241, 1081 and 754 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.42 (d, *J* = 7.4 Hz, 2H), 7.31 (t, *J* = 8 Hz, 2H), 7.01 (t, *J* = 7.4 Hz, 2H), 6.96 (d, *J* = 8.2 Hz, 2H), 4.66 (s, 4H), 4.40 (s, 4H), 4.21 (d, *J* = 2.4 Hz, 4H), 2.38 (t, *J* = 2.3 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 156.4, 129.5, 129.0, 126.3, 121.0, 111.6, 79.9, 74.3, 66.9, 66.6, 57.5. HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₂₂H₂₂O₄Na: 373.1416; found 373.1425.

1,4-Bis(2-((prop-2-yn-1-yloxy)methyl)phenoxy)butane (4b): Following the general procedure D, **4b** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a red liquid; Yield: 0.321 g, 85%; IR (CH₂Cl₂): 2943, 2883, 1602, 1493, 1241, 1081 and 754 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.40 (d, *J* = 7.4 Hz, 2H), 7.29 (t, *J* = 7.8 Hz, 2H), 6.98 (t, *J* = 7.8 Hz, 2H), 6.90 (d, *J* = 8.0 Hz, 2H), 4.68 (s, 4H), 4.24 (d, *J* = 2.4 Hz, 4H), 4.09 (brs, 4H), 2.48 (t, *J* = 2.4 Hz, 2H), 2.07-2.04 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 156.7, 129.6, 129.1, 125.8, 120.4, 111.2, 80.0, 74.4, 67.5, 66.6, 57.5, 26.1. HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₂₄H₂₆O₄Na: 401.1729; found 401.1736.

1,6-Bis(2-((prop-2-yn-1-yloxy)methyl)phenoxy)hexane (4c): Following the general procedure D, **4c** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a brown liquid; Yield: 0.324 g, 80%; IR (CH₂Cl₂): 3291, 2940, 2862, 1602, 1590, 1494, 1455, 1048 and 754 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.35 (d, *J* = 7.4 Hz, 2H), 7.24 (t, *J* = 7.8 Hz, 2H), 6.92 (t, *J* = 7.4 Hz, 2H), 6.85 (d, *J* = 8.2 Hz, 2H), 4.64 (s, 4H), 4.20 (d, *J* = 2.3 Hz, 4H), 3.98 (t, *J* = 6.3 Hz, 4H), 2.43 (t, *J* = 2.3 Hz, 2H), 1.83 (t, *J* = 6.3 Hz, 4H), 1.57-1.53 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 156.8, 129.5, 129.0, 125.9, 120.3, 111.3, 80.1, 74.3, 67.9, 66.7, 57.5, 29.3, 25.9; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₂₆H₃₀O₄Na: 429.2042; found 429.2048.

1,4-Bis((1-((prop-2-yn-1-yloxy)methyl)naphthalen-2-yl)oxy)butane (4d): Following the general procedure D, **4d** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 20:80) as a brown solid, mp: 86-88 °C; Yield: 0.429 g, 92%; IR (CH₂Cl₂): 2946, 1513, 1464, 1264, 1023 and 748 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.12 (d, *J* = 8.4 Hz, 2H), 7.81-7.75 (m, 4H), 7.50 (t, *J* = 7.7 Hz, 2H), 7.34 (t, *J* = 7.4 Hz, 2H), 7.24 (d, *J* = 9.1 Hz, 2H), 5.14 (s, 4H), 4.22-4.18 (m, 8H), 2.49 (t, *J* = 2.3 Hz, 2H), 2.10 (t, *J* = 2.7 Hz, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 155.2, 133.9, 130.7, 129.2, 128.3, 127.0, 123.8, 118.1, 114.4, 80.4, 74.4,

69.2, 61.9, 57.3, 26.5. HRMS (ESI): m/z $[M + Na]^+$ calcd for $C_{32}H_{30}O_4Na$: 501.2042; found 501.2037.

1,2-Bis(2-((prop-2-yn-1-yloxy)methyl)phenoxy)methylbenzene (4e): Following the general procedure D, **4e** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a colourless liquid; Yield: 0.319 g, 75%; IR (CH_2Cl_2): 2929, 2851, 1603, 1590, 1493 and 754 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$): δ 7.58-7.56 (m, 2H), 7.41-7.37 (m, 4H), 7.29-7.24 (m, 2H), 7.01-6.95 (m, 4H), 5.22 (s, 4H), 4.68 (s, 4H), 4.16 (d, $J = 2.4\text{ Hz}$, 4H), 2.38 (t, $J = 2.3\text{ Hz}$, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.4, 135.1, 129.7, 129.2, 128.7, 128.3, 126.1, 120.9, 111.7, 79.9, 74.5, 68.0, 66.7, 57.5. HRMS (ESI): m/z $[M + Na]^+$ calcd for $C_{28}H_{26}O_4Na$: 449.1729; found 449.1731.

1,3-Bis(2-((prop-2-yn-1-yloxy)methyl)phenoxy)methylbenzene (4f): Following the general procedure D, **4f** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a white solid, mp: 62-64 °C; Yield: 0.332 g, 78%; IR (CH_2Cl_2): 2929, 2851, 1603, 1590, 1493 and 754 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$): δ 7.55 (s, 1H), 7.45 (s, 5H), 7.29 (t, $J = 7.7\text{ Hz}$, 2H), 7.03-6.95 (m, 4H), 5.16 (s, 4H), 4.76 (s, 4H), 4.24 (s, 4H), 2.44 (brs, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.4, 137.5, 129.6, 129.1, 128.8, 128.8, 126.8, 126.2, 126.0, 120.9, 111.9, 80.0, 74.5, 69.9, 66.8, 57.6. HRMS (ESI): m/z $[M + Na]^+$ calcd for $C_{28}H_{26}O_4Na$: 449.1729; found 449.1724.

(E)-1,4-Bis(2-((prop-2-yn-1-yloxy)methyl)phenoxy)but-2-ene (4g): Following the general procedure D, **4g** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a red solid, mp: 73-75 °C; Yield: 0.263 g, 70%; IR (CH_2Cl_2): 2929, 1625, 1595, 1513, 1148 and 748 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$): δ 7.33 (d, $J = 7.4\text{ Hz}$, 2H), 7.19 (t, $J = 7.8\text{ Hz}$, 2H), 6.91 (t, $J = 7.4\text{ Hz}$, 2H), 6.81 (d, $J = 8.2\text{ Hz}$, 2H), 6.05-6.04 (m, 2H), 4.63 (s, 4H), 4.56-4.55 (m, 4H), 4.16 (d, $J = 2.4\text{ Hz}$, 4H), 2.40 (t, $J = 2.3\text{ Hz}$, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.3, 129.6, 129.6, 127.9, 126.2, 120.8, 111.7, 80.0, 74.5, 67.9, 66.6, 57.5. HRMS (ESI): m/z $[M + Na]^+$ calcd for $C_{24}H_{24}O_4Na$: 399.1572; found 399.1578.

1,4-Bis(2-((prop-2-yn-1-yloxy)methyl)phenoxy)but-2-yne (4h): Following the general procedure D, **4h** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as red a liquid; Yield: 0.187 g, 50%; IR (CH_2Cl_2): 2929, 1625, 1595, 1513, 1148 and 748 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$): δ 7.33 (d, $J = 7.4\text{ Hz}$, 2H), 7.19-7.14 (m, 2H), 6.93 (t, $J = 7.4\text{ Hz}$, 2H), 6.86 (d, $J = 8.2\text{ Hz}$, 2H), 4.68 (s, 4H), 4.58 (s, 4H), 4.14 (d, $J = 2.4\text{ Hz}$, 4H), 2.39 (t, $J = 2.3\text{ Hz}$, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.3, 129.6, 128.9, 126.5, 121.5, 112.2, 82.4, 79.9, 74.4, 65.5, 57.5, 56.3; MS (CI): m/z (%) 374 ($[M]^+$, 20).

1,2-Bis(3-((prop-2-yn-1-yloxy)methyl)phenoxy)ethane (4j): Following the general procedure D, **4j** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a red liquid; Yield: 0.175 g, 50%; IR (CH_2Cl_2): 2929, 2851, 1602, 1492 and 753 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$): δ 7.32 (t, $J = 7.7\text{ Hz}$, 2H), 7.0-6.92 (m, 6H), 4.62 (s, 4H), 4.35 (s, 4H), 4.20 (d, $J = 2.4\text{ Hz}$, 4H), 2.50 (t, $J = 2.4\text{ Hz}$, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.8, 138.9, 129.5, 120.7, 114.4, 114.1, 79.6, 74.7, 71.3, 66.5, 57.1. HRMS (ESI): m/z $[M + Na]^+$ calcd for $C_{22}H_{22}O_4Na$: 373.1416; found 373.1427.

1,4-Bis(4-((prop-2-yn-1-yloxy)methyl)phenoxy)butane (4k): Following the general procedure D, **4k** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a white solid; mp: 86-88 °C; Yield: 0.189 g, 50%; IR (CH₂Cl₂): 2920, 2851, 1611, 1584, 1454 and 818 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.30 (d, *J* = 8.6 Hz, 4H), 6.90 (d, *J* = 8.6 Hz, 4H), 4.56 (s, 4H), 4.16 (d, *J* = 2.4 Hz, 4H), 4.05 (brs, 4H), 2.49 (t, *J* = 2.4 Hz, 2H), 2.01-1.98 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 158.8, 129.9, 129.2, 114.4, 79.8, 74.5, 71.2, 67.5, 56.7, 25.9. HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₂₄H₂₆O₄Na: 401.1729; found 401.1735.

2,2'-(((Oxybis(ethane-2,1-diyl))bis(oxy))bis(((prop-2-yn-1-yloxy)methyl)benzene) (4l): Following the general procedure D, **4l** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a red liquid; Yield: 0.275 g, 70%; IR (CH₂Cl₂): 2930, 2875, 1603, 1494, 1083 and 755 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.38 (d, *J* = 7.4 Hz, 2H), 7.26 (t, *J* = 7.8 Hz, 2H), 6.97 (t, *J* = 7.4 Hz, 2H), 6.88 (d, *J* = 7.6 Hz, 2H), 4.66 (s, 4H), 4.20-4.17 (m, 8H), 3.98-3.95 (m, 4H), 2.44 (t, *J* = 2.3 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 156.5, 129.6, 129.0, 126.2, 120.9, 111.6, 80.1, 74.4, 70.0, 68.0, 66.7, 57.5; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₂₄H₂₆O₅Na: 417.1678; found 417.1689.

4,4'-(((Oxybis(ethane-2,1-diyl))bis(oxy))bis(1-bromo-3-((prop-2-yn-1-yloxy)methyl)benzene) (4m): Following the general procedure D, **4m** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a red liquid; Yield: 0.247 g, 45%; IR (CH₂Cl₂): 2931, 2874, 1603, 1590, 1494, 1288, 1083 and 755 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.42 (d, *J* = 2.5 Hz, 2H), 7.26 (dd, *J* = 6.2, 2.5 Hz, 2H), 6.67 (d, *J* = 8.7 Hz, 2H), 4.52 (s, 4H), 4.12 (d, *J* = 2.3 Hz, 4H), 4.06 (t, *J* = 4.7 Hz, 4H), 3.86-3.83 (m, 4H), 2.37 (t, *J* = 2.3 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 155.3, 131.8, 131.4, 128.6, 113.3, 113.3, 79.7, 74.6, 69.9, 68.3, 66.0, 57.8; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₂₄H₂₄Br₂O₅Na: 572.9888; found 572.9886.

1,2-Bis(2-(2-((prop-2-yn-1-yloxy)methyl)phenoxy)ethoxy)ethane (4n): Following the general procedure D, **4n** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 30:70) as a red liquid; Yield: 0.35 g, 80%; IR (CH₂Cl₂): 2926, 2874, 1590, 1494, 1247 and 755 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.36 (d, *J* = 7.4 Hz, 2H), 7.24 (t, *J* = 7.9 Hz, 2H), 6.94 (t, *J* = 7.4 Hz, 2H), 6.85 (d, *J* = 8.1 Hz, 2H), 4.65 (s, 4H), 4.20 (d, *J* = 2.3, 4H), 4.14 (t, *J* = 4.9 Hz, 4H), 3.87 (t, *J* = 4.9 Hz, 4H), 3.75 (s, 4H), 2.46 (t, *J* = 2.3 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 156.5, 129.5, 129.0, 126.1, 120.8, 111.6, 80.1, 74.4, 71.0, 69.8, 67.9, 66.7, 57.5; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₂₆H₃₀O₆Na: 461.1940; found 461.1949.

2,2'-((((Oxybis(ethane-2,1-diyl))bis(oxy))bis(ethane-2,1-diyl))bis(oxy))bis(((prop-2-yn-1-yloxy)methyl)benzene) (4o): Following the general procedure D, **4o** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 40:60) as a colourless liquid; Yield: 0.462 g, 96%; IR (CH₂Cl₂): 2874, 1603, 1590, 1494, 1121 and 755 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.36 (d, *J* = 7.4 Hz, 2H), 7.24 (t, *J* = 7 Hz, 2H), 6.95 (t, *J* = 7.4 Hz, 2H), 6.84 (d, *J* = 7.9 Hz, 2H), 4.65 (s, 4H), 4.21 (d, *J* = 2.4 Hz, 4H), 4.14 (d, *J* = 4.9 Hz, 4H), 3.86 (t, *J* = 4.9 Hz, 4H), 3.75-3.72 (m, 4H), 3.69-3.66 (m, 4H), 2.47 (t, *J* = 2.3 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 156.5, 129.5, 129.0, 126.1, 120.7, 111.6, 80.1, 74.4, 70.9, 70.7, 69.7, 67.8, 66.6, 57.5. HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₂₈H₃₄O₇Na: 505.2202; found 505.2206.

2,2'-((Oxybis(ethane-2,1-diyl))bis(oxy))bis(1-((prop-2-yn-1-yloxy)methyl)naphthalene) (4p):

Following the general procedure D, **4p** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 20:80) as a colourless liquid; Yield: 0.395 g, 80%; IR (CH₂Cl₂): 2879, 1596, 1513, 1135, 1074 and 736 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.16 (d, *J* = 8.5 Hz, 2H), 7.85-7.80 (m, 4H), 7.55 (t, *J* = 8.4 Hz, 2H), 7.40 (t, *J* = 8.1 Hz, 2H), 7.30 (d, *J* = 8.9 Hz, 2H), 5.1 (s, 4H), 4.35 (t, *J* = 4.8 Hz, 4H), 4.25 (d, *J* = 2.4 Hz, 4H), 4.04 (t, *J* = 4.8 Hz, 4H), 2.51 (t, *J* = 2.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 155.1, 133.8, 130.7, 129.5, 128.3, 127.1, 123.9, 123.9, 118.7, 115.0, 80.5, 74.4, 70.3, 69.7, 62.0, 57.3; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₃₂H₃₀O₅Na: 517.1991; found 517.1992.

1,2-Bis(2-(1-(prop-2-yn-1-yloxy)but-3-en-1-yl)phenoxy)ethane (4s): Following the general procedure described above, **4s** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a red liquid; Yield: 0.352 g, 82%; IR (CH₂Cl₂): 2929, 2851, 1602, 1492 and 753 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.43-7.27 (m, 4H), 7.04 (t, *J* = 7.2 Hz, 2H), 6.96 (d, *J* = 8.2 Hz, 2H), 5.86-5.79 (m, 2H), 5.07-4.98 (m, 6H), 4.36 (s, 4H), 4.14 (d, *J* = 2.4 Hz, 1H), 4.10 (d, *J* = 2.4 Hz, 1H), 3.95-3.90 (m, 2H), 2.53-2.49 (m, 4H), 2.36 (t, *J* = 2.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 156.1, 134.8, 129.4, 128.5, 127.0, 121.3, 116.7, 111.5, 80.2, 74.1, 73.9, 66.9, 56.0, 40.8. HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₂₈H₃₀O₄Na: 453.2042; found 453.2051.

1,4-Bis(2-(1-(prop-2-yn-1-yloxy)but-3-en-1-yl)phenoxy)butane (4t): Following the general procedure D, **4t** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a white solid, mp 85-87 °C; Yield: 0.389 g, 85%; IR (CH₂Cl₂): 2929, 2851, 1602, 1492 and 753 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.40 (dd, *J* = 5.8, 1.6 Hz, 2H), 7.28-7.24 (m, 2H), 7.01 (t, *J* = 7.4 Hz, 2H), 6.90 (d, *J* = 8.2 Hz, 2H), 5.92-5.82 (m, 2H), 5.11-5.02 (m, 6H), 4.17-3.92 (m, 8H), 2.54-2.49 (m, 4H), 2.40-2.39 (m, 2H), 2.05 (t, *J* = 2.7 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 156.4, 135.1, 129.1, 128.5, 126.7, 120.7, 116.6, 111.1, 80.2, 74.1, 73.9, 67.4, 55.9, 40.9, 26.2. HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₃₀H₃₄O₄Na: 481.2355; found 481.2364.

1,3-Bis((2-(1-(prop-2-yn-1-yloxy)but-3-en-1-yl)phenoxy)methyl)benzene (4u): Following the general procedure D, **4u** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a colourless liquid; Yield: 0.253 g, 50%; IR (CH₂Cl₂): 3073, 2924, 2855, 1601, 1588, 1048 and 754 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.51 (s, 1H), 7.44-7.42 (m, 5H), 7.28-7.24 (m, 2H), 7.03 (t, *J* = 7.2 Hz, 2H), 6.95 (d, *J* = 8.1 Hz, 4H), 5.92-5.85 (m, 2H), 5.15 (s, 6H), 5.10-5.02 (m, 4H), 3.97 (d, *J* = 2.3 Hz, 1H), 3.93 (d, *J* = 2.3 Hz, 1H), 2.57-2.53 (m, 4H), 2.38-2.37 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 156.1, 137.5, 135.0, 129.5, 128.8, 128.5, 126.9, 126.6, 125.7, 121.2, 116.6, 111.9, 80.2, 74.3, 73.9, 69.8, 56.0, 41.0; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₃₄H₃₄O₄Na: 529.2355; found 529.2357.

2,2'-((Oxybis(ethane-2,1-diyl))bis(oxy))bis((1-(prop-2-yn-1-yloxy)but-3-en-1-yl)benzene) (4v):

Following the general procedure D, **4v** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a red liquid; Yield: 0.402 g, 85%; IR (CH₂Cl₂): 2927, 1600, 1588, 1489, 1285, 1241, 1082 and 755 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.42-7.24 (m, 4H), 7.02-6.89 (m, 4H), 5.93-5.83 (m, 2H), 5.10-5.01 (m, 6H), 4.20-3.93 (m, 12H), 2.55-2.53 (m, 4H), 2.38 (t, *J* = 2.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 156.2, 135.1,

129.4, 128.5, 126.8, 121.1, 116.5, 111.6, 80.3, 74.4, 74.3, 73.8, 70.1, 67.9, 50.0, 40.9. HRMS (ESI): m/z $[M + Na]^+$ calcd for $C_{30}H_{34}O_5Na$: 497.2304; found 497.2318.

1,2-Bis(2-(2-(1-(prop-2-yn-1-yloxy)but-3-en-1-yl)phenoxy)ethoxy)ethane (4w): Following the general procedure D, **4w** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a red liquid; Yield: 0.450 g, 87%; IR (CH_2Cl_2): 2927, 1600, 1588, 1489, 1285, 1241, 1082 and 755 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$): δ 7.41-7.23 (m, 4H), 7.01-6.87 (m, 4H), 5.92-5.83 (m, 2H), 5.11-5.02 (m, 6H), 4.18-3.88 (m, 12H), 3.77 (s, 4H), 2.54-2.51 (m, 4H), 2.41 (t, $J = 2.4\text{ Hz}$, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.2, 135.1, 129.3, 128.5, 126.8, 121.0, 116.5, 111.6, 80.3, 74.3, 73.9, 71.0, 69.9, 67.7, 56.0, 40.8. HRMS (ESI): m/z $[M + Na]^+$ calcd for $C_{32}H_{38}O_6Na$: 541.2566; found 541.2567.

Di(prop-2-yn-1-yl) 2,2'-((1,2-phenylenebis(methylene))bis(oxy))dibenzoate (4i): To a solution of propargyl alcohol (1.5 mmol), 2,2'-((1,2-phenylenebis(methylene))bis(oxy))dibenzoic acid (**2i**, 0.5 mmol) and 4-(dimethylamino)pyridine (DMAP, 0.80 mmol) in CH_2Cl_2 (2.5 mL) was added 1,3-dicyclohexylcarbodiimide (1.5 mmol) in small fractions. The reaction mixture was stirred at room temperature for 2 h. The resulting pale yellow suspension was filtered through filtration funnel. The filtrate was concentrated and purified by silica gel column chromatography (EtOAc : Hexanes = 10:90) which afforded the product **4i** as a red solid, mp 104-106 °C; Yield: 0.181 g, 80%; IR (CH_2Cl_2): 2933, 1782, 1600, 1489, 1244 and 701 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$): δ 7.90-7.87 (m, 2H), 7.69-7.67 (m, 2H), 7.51-7.39 (m, 4H), 7.14-6.99 (m, 4H), 5.37 (s, 4H), 4.88 (m, 4H), 2.49 (t, $J = 2.4\text{ Hz}$, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.2, 158.3, 134.5, 134.1, 132.2, 128.6, 128.3, 120.5, 119.3, 113.5, 77.8, 75.0, 68.9, 52.3. HRMS (ESI): m/z $[M + Na]^+$ calcd for $C_{28}H_{22}O_6Na$: 477.1314; found 477.1311.

1,12-Bis(2-(prop-2-yn-1-yloxy)phenyl)-5,8-dioxa-2,11-dithiadodecane (4q): To a suspension of 2,2'-(ethane-1,2-diylbis(oxy))diethanethiol (2 mmol) in ethanol (10 mL) at room temperature was added KOH (4mmol) and the reaction mixture was stirred at room temperature for 1 h. Then, to this solution, a solution of 1-(chloromethyl)-2-(prop-2-yn-1-yloxy)benzene(4 mmol) in benzene (5 mL) was added drop wise. The resulting mixture was stirred for an additional 1 h and then the reaction mixture was filtered. The resulted filtrate was added with DCM (20 mL) and the combined layers were washed with H_2O (3 X 10 mL). After this, the organic were dried over anhydrous Na_2SO_4 , concentrated under vacuum and the resulting crude reaction mixture was purified by silica gel column chromatography (EtOAc : hexanes = 10:90), which afforded the product **4q** as a pale yellow solid, mp 76-78 °C; Yield: 0.705 g, 75%; IR (CH_2Cl_2): 2929, 1732, 1600, 1297 and 753 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$): δ 7.25-7.15 (m, 4H), 6.94-6.88 (m, 4H), 4.69 (d, $J = 2.4\text{ Hz}$, 2H), 3.74 (s, 4H), 3.57 (t, $J = 7\text{ Hz}$, 4H), 3.54 (s, 4H), 2.62 (t, $J = 7\text{ Hz}$, 4H), 2.47 (t, $J = 2.4\text{ Hz}$, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.3, 130.6, 128.2, 127.7, 121.5, 112.3, 78.7, 75.6, 70.8, 70.2, 56.1, 30.8, 30.5. HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{26}H_{32}O_4S_2$: 471.1664; found 471.1659.

Di(prop-2-yn-1-yl) 2,2'-(((ethane-1,2-diylbis(oxy))bis(ethane-2,1-diyl))bis(oxy))dibenzoate (4r): To a solution of propargyl alcohol (1.5 mmol), 2,2'-(((ethane-1,2-diylbis(oxy))bis(ethane-2,1-diyl))bis(oxy))dibenzoic acid (**2r**, 0.5 mmol) and 4-(dimethylamino)pyridine (DMAP, 0.80 mmol) in CH_2Cl_2 (2.5 mL) was added 1,3-dicyclohexylcarbodiimide (1.5 mmol) in small fractions. The reaction mixture was stirred at room temperature for 2 h. The resulting pale yellow

suspension was filtered through filtration funnel. The filtrate was concentrated and purified by silica gel column chromatography (EtOAc : Hexanes = 10:90) which afforded the product **4r** as red sticky liquid; Yield: 0.202 g, 87%; IR (CH₂Cl₂): 2935, 2876, 1590, 1603, 1494, 1248 and 739 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.86-7.83 (m, 2H), 7.48-7.44 (m, 2H), 6.99 (d, *J* = 8 Hz, 4H), 4.88 (d, *J* = 2.4 Hz, 4H), 4.21 (t, *J* = 4.7 Hz, 4H), 3.93 (t, *J* = 5.16 Hz, 4H), 3.81 (s, 4H), 2.53 (t, *J* = 2.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 165.2, 158.7, 133.9, 131.9, 120.5, 119.6, 113.7, 77.9, 74.9, 71.2, 69.5, 68.9, 52.2. HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₂₆H₂₆O₈Na: 489.1525; found 489.1530.

General procedure for the syntheses of macrocycles 5a-k, 6a-g and 7a-7e (Procedure E). A mixture of **4a** (0.20 mmol), Cu(OAc)₂·H₂O (30 mol % or 1 equiv as mentioned the respective Scheme/Table/Fig.) and DMSO (2 mL) was taken in a vial (10 mL capacity) or round bottom flask (10 or 20 mL capacity). The reaction mixture was stirred at 110 °C under open air atmosphere for 4 h. After this period, the resulting mixture was cooled to room temperature and diluted with water (4 mL). The mixture was filtered through a filtration funnel and the washed with ethyl acetate (4 x 5 mL). The combined layers were extracted using ethyl acetate (3 x 5 mL) and the combined organic layers were dried over anhydrous Na₂SO₄. The solvent was removed under reduced pressure and the residue was purified by silica gel chromatography (EtOAc : Hexane) which gave the crown/polyether-type macrocycles **5a-k**, **6a-g** and **7a-7e**. See the corresponding Schemes 3 and 4 and Fig. 1 for specific examples.

5a: Following the general procedure E, **5a** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a white solid, mp: 142-144 °C; Yield: 0.061 g, 70%; IR (CH₂Cl₂): 3031, 1702, 1599, 1486 and 1027 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.37 (d, *J* = 5.8 Hz, 2H), 7.26 (t, *J* = 6.9 Hz, 2H), 6.98 (t, *J* = 7.0 Hz, 2H), 6.88 (d, *J* = 7.6 Hz, 2H), 4.81 (s, 4H), 4.41 (s, 4H), 4.28 (s, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 156.6, 129.3, 128.9, 126.1, 121.1, 111.7, 76.5, 72.1, 67.6, 64.5, 57.3; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₂₀H₂₀O₄Na: 371.1259; found 371.1263. This compound was crystallized using a mixture of EtOAc and Hexanes and confirmed by single crystal X-ray structure analysis.

5b: Following the general procedure E, **5b** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a white solid mp: 131-133 °C; Yield: 0.049 g, 52%; IR (CH₂Cl₂): 2942, 2883, 1599, 1343 and 891cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.36 (d, *J* = 5.9 Hz, 2H), 7.29-7.26 (m, 2H), 6.96 (t, *J* = 7.0 Hz, 2H), 6.89 (d, *J* = 8.1 Hz, 2H), 4.76 (s, 4H), 4.27 (s, 4H), 4.05(brs, 4H), 2.06 (brs, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 156.9, 129.6, 129.1, 125.7, 120.7, 111.4, 75.4, 70.9, 68.4, 64.9, 57.2, 26.5; HRMS (ESI): *m/z* [M + H]⁺ calcd for C₂₄H₂₅O₄: 377.1752; found 377.1729.

5c: Following the general procedure E, **5c** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a white solid, mp: 134-136 °C; Yield: 0.105 g, 52%; IR (CH₂Cl₂): 2936, 1602, 1424, 1248 and 732 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.31-7.24 (m, 4H), 6.91 (t, *J* = 7.0 Hz, 2H), 6.85 (d, *J* = 8.2 Hz, 2H), 4.65 (s, 4H), 4.27 (s, 4H), 3.99 (t, *J* = 5.9 Hz, 4H), 1.89-1.86 (m, 4H), 1.66-1.62(m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 157.4, 130.5, 129.5, 125.7, 120.4, 111.4, 75.6, 70.3, 68.3, 66.3, 57.6, 29.6, 26.7; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₂₆H₂₈O₄Na: 427.1885; found 427.1899.

5d: Following the general procedure E, **5d** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 20:80) as a white solid, mp: 185-187 °C; Yield: 0.167 g, 70%; IR (CH₂Cl₂): 2925, 2867, 1601, 1493, 1248 and 755 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.10 (d, *J* = 8.2 Hz, 2H), 7.83 (d, *J* = 9 Hz, 2H), 7.78 (d, *J* = 80 Hz, 2H), 7.51 (t, *J* = 71 Hz, 2H), 7.37 (t, *J* = 6.9 Hz, 2H), 7.30 (d, *J* = 9.0 Hz, 2H), 5.28 (s, 4H), 4.21-4.19 (m, 8H), 2.22-2.20 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 155.9, 133.8, 130.7, 129.4, 128.3, 126.9, 124.0, 123.9, 117.9, 115.0, 75.8, 70.9, 70.4, 60.8, 56.2, 27.3; HRMS (ESI): *m/z* [M + H]⁺ calcd for C₃₂H₂₉O₄: 477.2066; found 477.2058.

5e: Following the general procedure E, **5e** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a white solid, mp: 95-97 °C; Yield: 0.091 g, 43%; IR (CH₂Cl₂): 2873, 1603, 1344, 1492 and 1017 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.48-7.45 (m, 2H), 7.36-7.29 (m, 4H), 7.21 (t, *J* = 7.8 Hz, 2H), 6.94 (t, *J* = 6.9 Hz, 2H), 6.80 (d, *J* = 7.8 Hz, 2H), 5.34 (s, 4H), 4.75 (s, 4H), 4.27 (s, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 156.2, 135.1, 129.6, 129.2, 128.4, 128.2, 126.1, 120.9, 111.6, 75.7, 70.8, 68.1, 66.2, 57.8; HRMS (ESI): *m/z* [M + H]⁺ calcd for C₂₈H₂₅O₄: 425.1753; found 425.1747.

5f: Following the general procedure E, **5f** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a white solid, mp: 92-94 °C; Yield: 0.095 g, 45%; IR (CH₂Cl₂): 3048, 2872, 1590, 1373 and 753 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.56 (s, 1H), 7.35-7.29 (m, 5H), 7.24-7.17 (m, 2H), 6.96-6.91 (m, 4H), 5.01 (s, 4H), 4.64 (s, 4H), 4.22 (s, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 156.7, 137.4, 129.2, 129.0, 128.6, 128.3, 128.1, 126.6, 121.2, 112.4, 75.4, 70.9, 70.7, 65.9, 57.8; HRMS (ESI): *m/z* [M + H]⁺ calcd for C₂₈H₂₅O₄: 425.1753; found 425.1749. This compound was crystallized using a mixture of EtOAc and Hexanes and confirmed by single crystal X-ray structure analysis.

5g: Following the general procedure E, **5g** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a white solid, mp: 110-112 °C; Yield: 0.081 g, 43%; IR (CH₂Cl₂): 2878, 1602, 1493, 1239 and 603 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.32 (d, *J* = 7.4 Hz, 2H), 7.20 (t, *J* = 7.9 Hz, 2H), 6.92 (t, *J* = 7.3 Hz, 2H), 6.83 (d, *J* = 7.8 Hz, 2H), 6.15 (s, 2H), 4.74 (s, 4H), 4.51 (s, 4H), 4.21 (s, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 156.4, 129.4, 129.0, 127.7, 125.9, 121.1, 111.7, 75.4, 71.0, 68.2, 64.8, 57.3; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₂₄H₂₂O₄Na: 397.1416; found 397.1409. This compound was crystallized using a mixture of EtOAc and Hexanes and confirmed by single crystal X-ray structure analysis.

5h: Following the general procedure E, **5h** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a white solid, mp: 145-147 °C; Yield: 0.065 g, 35%; IR (KBr): 2943, 1600, 1493, 1061 and 753 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.41 (d, *J* = 7.4 Hz, 2H), 7.31-7.27 (m, 2H), 7.04 (t, *J* = 7 Hz, 2H), 6.89 (d, *J* = 8.2 Hz, 2H), 4.79 (s, 4H), 4.78 (s, 4H), 4.33 (s, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 155.6, 129.4, 128.9, 126.8, 121.8, 112.3, 81.7, 75.3, 71.0, 65.4, 57.6, 57.2; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₂₄H₂₀O₄Na: 395.1259; found 395.1253. This compound was crystallized using a mixture of EtOAc and Hexanes and confirmed by single crystal X-ray structure analysis.

5i: Following the general procedure E, **5i** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a white solid, mp: 215-217 °C; Yield:

0.014 g, 25%; IR (KBr): 2929, 1732, 1600, 1297 and 753 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.67 (dd, J = 5.8, 1.7 Hz, 2H), 7.52-7.50 (m, 2H), 7.42-7.34 (m, 4H), 6.99 (t, J = 5.5 Hz, 2H), 6.85 (d, J = 8.0 Hz, 2H), 5.33 (s, 4H), 4.92 (s, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ 166.4, 157.3, 134.4, 133.2, 130.6, 128.4, 128.3, 120.6, 113.0, 74.7, 70.7, 68.5, 52.8. HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{28}\text{H}_{20}\text{O}_6$: 475.1158; found 475.1156. This compound was crystallized using a mixture of DCM and Hexanes and confirmed by single crystal X-ray structure analysis.

5j: Following the general procedure E, **5j** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a white solid, mp: 120-122 $^\circ\text{C}$; Yield: 0.022 g, 25%; IR (KBr): 2925, 2851, 1731, 1595 and 786 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.31-7.27 (m, 2H), 7.14 (s, 2H), 6.97-6.93 (m, 4H), 4.67 (s, 4H), 4.45 (s, 4H), 4.24 (s, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ 158.9, 138.7, 129.7, 120.9, 116.1, 114.8, 75.9, 71.3, 70.9, 67.3, 57.3; HRMS (ESI): m/z $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{22}\text{H}_{21}\text{O}_4$: 349.1439; found 349.1434.

5k: Following the general procedure E, **5k** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a white solid, mp: 83-85 $^\circ\text{C}$; Yield: 0.047 g, 25%; IR (CH_2Cl_2): 2920, 2851, 1611, 1584, 1454 and 818 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.30 (d, J = 8.6 Hz, 4H), 6.89 (d, J = 8.6 Hz, 4H), 4.63 (s, 4H), 4.24 (s, 4H), 4.17 (brs, 4H), 1.96 (brs, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ 158.4, 129.9, 129.6, 114.6, 76.3, 71.7, 67.1, 57.9, 24.4; HRMS (ESI): m/z $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{24}\text{H}_{25}\text{O}_4$: 377.1752; found 377.1747. This compound was crystallized using a mixture of DCM and Hexanes and confirmed by single crystal X-ray structure analysis.

6a: Following the general procedure E, **6a** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a white solid, mp: 99-101 $^\circ\text{C}$; Yield: 0.061 g, 52%; IR (CH_2Cl_2): 2935, 2876, 1603, 1494, 1246 and 704 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.33 (d, J = 5.8 Hz, 2H), 7.29-7.25 (m, 2H), 6.96 (t, J = 7.0 Hz, 2H), 6.89 (d, J = 8.2 Hz, 2H), 4.73 (s, 4H), 4.27 (s, 4H), 4.18-4.09 (m, 8H); ^{13}C NMR (100 MHz, CDCl_3): δ 157.1, 130.5, 129.5, 125.9, 121.1, 112.2, 75.8, 70.9, 70.6, 69.1, 65.7, 57.4; HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{24}\text{H}_{24}\text{O}_5\text{Na}$: 415.1521; found 415.1557. This compound was crystallized using a mixture of EtOAc and Hexanes and confirmed by single crystal X-ray structure analysis.

6b: Following the general procedure E, **6b** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a white solid, mp: 132-134 $^\circ\text{C}$; Yield: 0.082 g, 30%; IR (CH_2Cl_2): 2932, 2874, 1603, 1454, 1249 and 737 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.50 (d, J = 2.5 Hz, 2H), 7.40-7.37 (m, 2H), 6.79 (d, J = 8.7 Hz, 2H), 4.70 (s, 4H), 4.31 (s, 4H), 4.18-4.16 (m, 4H), 4.09-4.06 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ 155.9, 132.6, 131.9, 128.3, 113.8, 113.4, 75.5, 70.9, 70.7, 69.3, 65.1, 57.6; HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{24}\text{H}_{22}\text{Br}_2\text{O}_5\text{Na}$: 570.9731; found 570.9714.

6c: Following the general procedure E, **6c** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a white solid, mp: 131-133 $^\circ\text{C}$; Yield: 0.045 g, 52%; IR (CH_2Cl_2): 2938, 1624, 1512, 1435, 1246 and 748 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.35 (d, J = 7.4 Hz, 2H), 7.31-7.27 (m, 2H), 6.98 (t, J = 7.4 Hz, 2H), 6.88 (d, J = 7.9 Hz, 2H), 4.69 (s, 4H), 4.35 (s, 4H), 4.20-4.18 (m, 4H), 3.99-3.97 (m, 4H), 3.92 (s, 4H); ^{13}C NMR

(100 MHz, CDCl₃): δ 156.7, 130.1, 129.4, 125.9, 120.8, 111.5, 75.9, 71.4, 70.4, 69.9, 68.7, 66.7, 58.0; HRMS (ESI): m/z [M + Na]⁺ calcd for C₂₆H₂₈O₆Na: 459.1783; found 459.1794.

6d: Following the general procedure E, **6d** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 40:60) as a colourless liquid; Yield: 0.108 g, 45%; IR (CH₂Cl₂): 2874, 1603, 1590, 1494, 1288 and 755 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.32 (d, J = 7.4 Hz, 2H), 7.25 (t, J = 6.9 Hz, 2H), 6.94 (t, J = 7.4 Hz, 2H), 6.84 (d, J = 8.1 Hz, 2H), 4.65 (s, 4H), 4.31 (s, 4H), 4.14-4.12 (m, 4H), 3.90-3.72 (m, 12H); ¹³C NMR (100 MHz, CDCl₃): δ 156.7, 130.1, 129.3, 126.0, 111.6, 75.9, 71.1, 70.7, 70.4, 69.7, 68.2, 66.9, 58.1; HRMS (ESI): m/z [M + Na]⁺ calcd for C₂₈H₃₂O₇Na: 503.2046; found 503.2032.

6e: Following the general procedure E, **6e** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 20:80) as a white solid, mp: 163-165 °C; Yield: 0.071 g, 48%; IR (CH₂Cl₂): 2874, 1623, 1590, 1345, 1288 and 742 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.08 (d, J = 8.4 Hz, 2H), 7.84 (d, J = 9.0 Hz, 2H), 7.79 (d, J = 8.1 Hz, 2H), 7.51 (t, J = 7.1 Hz, 2H), 7.37 (t, J = 7.0 Hz, 2H), 7.28-7.24 (m, 2H), 5.27 (s, 4H), 4.33-4.25 (m, 8H), 4.24 (s, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 155.5, 133.8, 130.8, 129.4, 128.4, 127.1, 123.9, 123.8, 117.7, 114.4, 76.3, 71.5, 70.5, 70.4, 61.1, 56.6; HRMS (ESI): m/z [M + Na]⁺ calcd for C₃₂H₂₈O₅Na: 515.1834; found 515.1853. This compound was crystallized using a mixture of EtOAc and Hexanes and confirmed by single crystal X-ray structure analysis.

6f: Following the general procedure E, **6f** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a white solid, mp: 119-121 °C; Yield: 0.028 g, 30%; IR (CH₂Cl₂): 2929, 1732, 1600, 1297 and 753 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.38 (d, J = 6.2 Hz, 2H), 7.24 (t, J = 7.2 Hz, 2H), 7.02 (t, J = 7.4 Hz, 2H), 6.89 (d, J = 7.8 Hz, 2H), 4.81 (s, 4H), 3.80 (s, 4H), 3.63-3.59 (m, 8H), 2.65 (t, J = 7.2 Hz, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 155.1, 131.1, 128.2, 128.1, 122.2, 111.6, 74.6, 70.9, 70.8, 70.1, 56.5, 30.1, 29.3; HRMS (ESI): m/z [M + Na]⁺ calcd for C₂₆H₂₈O₄S₂Na: 491.1327; found 491.1361.

6g: Following the general procedure E, **6g** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 60:40) as a white solid, mp: 157-159 °C; Yield: 0.049 g, 35%; IR (CH₂Cl₂): 2935, 2876, 1590, 1603, 1494, 1248 and 739 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.84 (dd, J = 5.96, 1.76 Hz, 2H), 7.51-7.47 (m, 2H), 7.01 (t, J = 7.8 Hz, 2H), 6.95 (d, J = 8.4 Hz, 2H), 5.01 (s, 4H), 4.23-4.21 (m, 4H), 4.01-3.98 (m, 4H), 3.95 (s, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 166.4, 158.4, 134.2, 132.4, 120.5, 119.1, 112.9, 73.9, 71.6, 70.4, 69.6, 69.5, 52.8; HRMS (ESI): m/z [M + H]⁺ calcd for C₂₆H₂₅O₈: 465.1549; found 465.1541. This compound was crystallized using a mixture of DCM and Hexanes and confirmed by single crystal X-ray structure analysis.

7a: Following the general procedure E, **7a** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a semi solid; Yield: 0.322 g, 75%; IR (CH₂Cl₂): 2931, 1718, 1599, 1489 and 753 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.31-7.22 (m, 4H), 7.04-6.87 (m, 4H), 5.87-5.74 (m, 2H), 5.59-5.51 (m, 2H), 5.10-4.98 (m, 4H), 4.49-4.07 (m, 8H), 2.57-2.44 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 156.6, 156.5, 134.8, 134.6, 129.2, 128.5, 128.5, 128.4, 127.1, 126.8, 121.3, 121.3, 116.8, 116.7, 112.3, 111.8, 75.5, 75.3, 71.1, 71.9, 70.9, 70.4, 68.1, 67.5, 55.3, 55.1, 41.5, 41.6; HRMS (ESI): m/z [M + Na]⁺ calcd for C₂₈H₂₈O₄Na: 471.1783; found 471.1794.

451.1885; found 451.1868. Isolated as a mixture of isomers (*dr* = 60:40) and NMR values given for both the isomers.

7b: Following the general procedure E, **7b** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as white solid, mp: 110-112 °C; Yield: 0.329 g, 72%; IR (CH₂Cl₂): 2953, 2876, 1602, 1491 and 754 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.30 (d, *J* = 5.8 Hz, 2H), 7.25-7.21 (m, 2H), 6.96 (t, *J* = 7.2 Hz, 2H), 6.86 (d, *J* = 7.6 Hz, 2H), 5.82-5.76 (m, 2H), 5.45-5.42 (m, 2H), 5.06-4.97 (m, 4H), 4.24 (d, *J* = 16.56 Hz, 2H), 4.04 (t, *J* = 5.82 Hz, 4H), 3.94 (d, *J* = 16.6 Hz, 2H), 2.48-2.42 (m, 4H), 2.10-1.98 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 156.8, 134.8, 128.6, 128.5, 126.5, 120.8, 116.6, 110.9, 75.3, 70.9, 70.6, 67.9, 55.4, 41.2, 26.7; HRMS (ESI): *m/z* [M + H]⁺ calcd for C₃₀H₃₃O₄: 457.2378; found 457.2365. This reaction gave a mixture of isomers (*dr* = 60:40) however, we got one of the isomer in pure form and NMR values given for one of the pure isomers. This compound was crystallized using a mixture of EtOAc and Hexanes and confirmed by single crystal X-ray structure analysis.

7c: Following the general procedure E, **7c** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a semi solid; Yield: 0.035 g, 35%; IR (CH₂Cl₂): 2874, 1488, 1248, 1017 and 805 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.41 (s, 1H), 7.39-7.28 (m, 7H), 7.13-7.05 (m, 4H), 5.84-5.38 (m, 2H), 5.38-5.15 (m, 2H), 5.15-5.01 (m, 8H), 4.31-4.27 (m, 2H), 4.08-4.01 (m, 2H), 2.52-2.48 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 156.7, 156.6, 137.6, 137.4, 134.9, 134.8, 129.6, 129.5, 128.7, 128.6, 128.6, 128.5, 128.4, 127.5, 126.9, 126.8, 126.6, 121.5, 121.4, 116.7, 116.6, 112.8, 112.6, 75.4, 75.2, 72.2, 71.1, 71.0, 70.5, 70.3, 55.7, 55.6, 41.1, 40.9; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₃₄H₃₂O₄Na: 527.2198; found 527.2189. Isolated as a mixture of isomers (*dr* = 60:40) and NMR values given for both the isomers.

7d: Following the general procedure E, **7d** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a white solid, mp: 82-84 °C; Yield: 0.042 g, 45%; IR (CH₂Cl₂): 2930, 1641, 1600, 1489 and 755 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.35-7.26 (m, 4H), 7.06-6.91 (m, 4H), 5.92-5.42 (m, 2H), 5.18-5.13 (m, 2H), 5.12-5.07 (m, 4H), 4.31-3.96 (m, 12H), 2.57-2.54 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 156.9, 156.7, 134.9, 134.9, 129.3, 128.8, 128.7, 128.6, 126.8, 126.8, 121.5, 121.3, 116.7, 116.6, 112.7, 111.6, 75.5, 75.4, 72.1, 71.7, 71.1, 70.6, 70.3, 69.9, 69.4, 55.6, 55.4, 41.0, 41.0; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₃₀H₃₂O₅Na: 495.2147; found 495.2139. Isolated as a mixture of isomers (*dr* = 60:40) and NMR values given for both the isomers. This compound was crystallized using a mixture of EtOAc and Hexanes and confirmed by single crystal X-ray structure analysis.

7e: Following the general procedure E, **7e** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a colourless liquid; Yield: 0.061 g, 60%; IR (CH₂Cl₂): 3076, 2870, 1598, 1489 and 754 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.36-7.25 (m, 4H), 7.03-6.87 (m, 4H), 5.87-5.83 (m, 2H), 5.27-5.24 (m, 2H), 5.13-5.03 (m, 4H), 4.33-4.17 (m, 6H), 4.02-3.86 (m, 10H), 2.53-2.51 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 156.4, 134.9, 129.1, 128.7, 126.8, 121.1, 116.6, 111.4, 75.8, 72.8, 71.5, 69.9, 69.8, 68.8, 56.1, 40.8; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₃₂H₃₆O₆Na: 539.2409; found 539.2404. This reaction gave a mixture of isomers (*dr* = 60:40) however, we got one of the isomer in pure form and NMR values given for one of the pure isomer.

General procedure for the syntheses of macrocycles 8a-f (Procedure F). A mixture of **5a** (0.20 mmol), $\text{NH}_2\text{OH}\cdot\text{HCl}$ (5 equiv), Et_3N (6 equiv) and DMSO (1 mL) was taken in a vial (10 mL capacity). The reaction mixture was sealed using a vial cap and stirred at 110 °C for 24 h. After this period, the vial was cooled to room temperature. Then the resulting mixture was diluted with water (4 mL). The mixture was filtered through a filtration funnel and the washed with ethyl acetate (4 x 5 mL). The combined layers were extracted using ethyl acetate (3 x 5 mL) and the combined organic layers were dried over anhydrous Na_2SO_4 . The solvent was removed under reduced pressure and the residue was purified by silica gel chromatography (EtOAc : Hexane) which gave the crown/polyether macrocycle **8**. See the corresponding Scheme 5 for specific examples.

8a: Following the general procedure F, **8a** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 50:50) as a white solid, mp: 141-143 °C; Yield: 0.062 g, 90%; IR (CH_2Cl_2): 2925, 2875, 1603, 1495 and 752 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.35-7.33 (m, 1H), 7.28-7.21 (m, 3H), 6.95-6.89 (m, 2H), 6.86-6.83 (m, 2H), 6.30 (s, 1H), 4.60 (s, 2H), 4.50 (s, 2H), 4.54 (s, 2H), 4.33-4.27 (m, 4H), 3.79 (t, J = 5.08 Hz, 2H), 2.92 (t, J = 5.20 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 171.9, 161.9, 156.9, 156.6, 130.9, 129.9, 129.5, 129.2, 126.6, 126.2, 121.2, 120.9, 111.0, 111.8, 101.5, 69.0, 67.6, 67.5, 67.3, 67.2, 63.5, 28.1; HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{22}\text{H}_{23}\text{NO}_5\text{Na}$: 404.1474; found 404.1483. This compound was crystallized using a mixture of MeOH, DCM and Hexanes and confirmed by single crystal X-ray structure analysis.

8b: Following the general procedure F, **8b** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 50:50) as a white solid, mp: 120-121 °C; Yield: 0.089 g, 88%; IR (CH_2Cl_2): 2943, 2884, 1599, 1470 and 754 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.33-7.25 (m, 4H), 6.95-6.85 (m, 4H), 6.34 (s, 1H), 4.62 (s, 2H), 4.54 (d, J = 2.6 Hz, 4H), 4.08-4.04 (m, 4H), 3.81 (t, J = 5.9 Hz, 2H), 2.99 (t, J = 5.8 Hz, 2H), 2.09-1.97 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ 171.7, 161.6, 157.5, 157.4, 131.7, 131.1, 129.8, 129.7, 125.7, 125.5, 120.5, 120.4, 111.4, 111.1, 101.1, 68.9, 68.9, 67.9, 67.4, 67.1, 64.1, 27.8, 26.4, 26.1; HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{24}\text{H}_{27}\text{NO}_5\text{Na}$: 432.1769; found 432.1769.

8c: Following the general procedure F, **8c** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 50:50) as a white solid, mp: 77-79 °C; Yield: 0.051 g, 45%; IR (KBr): 2923, 2865, 1604, 1495 and 754 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.50-7.37 (m, 2H), 7.31-7.26 (m, 2H), 7.15-7.03 (m, 2H), 6.92-6.88 (m, 2H), 6.16 (s, 1H), 5.82-5.71 (m, 2H), 5.01-4.90 (m, 5H), 4.69-4.24 (m, 7H), 3.99-3.87 (m, 1H), 3.66-3.38 (m, 1H), 3.14-2.81 (m, 2H), 2.53-2.47 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ 172.2, 171.8, 161.9, 161.7, 156.9, 156.2, 156.1, 155.9, 135.0, 134.9, 134.5, 133.9, 130.6, 130.3, 129.9, 128.7, 128.4, 128.3, 128.3, 127.5, 127.4, 127.3, 126.8, 122.5, 121.3, 121.2, 120.8, 117.1, 116.8, 116.7, 116.4, 115.2, 112.7, 111.1, 110.8, 102.2, 100.6, 72.5, 71.1, 69.3, 67.9, 67.6, 66.6, 66.6, 65.7, 60.8, 60.1, 41.5, 40.9, 40.9, 40.3, 29.4, 28.9; HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{28}\text{H}_{31}\text{NO}_5\text{Na}$: 484.2099; found 484.2092. Reaction was carried out from mixture of isomers of **7a** (dr = 60:40) and the compound **8c** was isolated as a mixture of isomers (dr = 60:40) and ^{13}C NMR values given for both the isomers. ^1H NMR values given for one of the isomer.

8d: Following the general procedure F, **8d** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 50:50) as a white solid, mp: 92-94 °C; Yield: 0.079 g, 65%; IR (CH₂Cl₂): 2938, 2873, 1600, 1490 and 804 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.35 (d, *J* = 7.56 Hz, 1H), 7.30 (d, *J* = 7.30 Hz, 1H), 7.19-7.16 (m, 2H), 6.92 (q, *J* = 7.60 Hz, 2H), 6.79 (t, *J* = 7.02 Hz, 2H), 6.17 (s, 1H), 5.69-5.60 (m, 2H), 4.97-4.87 (m, 5H), 4.71 (t, *J* = 6.38 Hz, 1H), 4.56-4.52 (m, 1H), 4.32-4.22 (m, 1H), 3.98-3.90 (m, 4H), 3.76-3.71 (m, 1H), 3.49-3.42 (m, 1H), 3.03-2.96 (m, 1H), 2.79-2.74 (m, 1H), 2.52-2.36 (m, 4H), 1.91-1.82 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 171.5, 171.2, 161.8, 161.6, 156.9, 156.3, 135.2, 134.9, 134.5, 129.6, 129.6, 128.7, 128.6, 128.5, 128.5, 127.5, 127.4, 127.3, 127.2, 120.8, 120.7, 120.6, 120.5, 116.8, 116.7, 116.6, 116.4, 111.4, 111.1, 110.9, 101.8, 100.9, 75.5, 73.2, 72.1, 67.8, 67.6, 67.5, 67.1, 66.1, 60.5, 42.4, 41.2, 41.0, 40.8, 29.7, 28.5, 28.3, 26.9, 26.7, 26.7; HRMS (ESI): *m/z* [M + H]⁺ calcd for C₃₀H₃₆NO₅: 490.2593; found 490.2581. Reaction was carried out from mixture of isomers of **7b** (*dr* = 60:40) the compound **8d** was isolated as a mixture of isomers (*dr* = 60:40) and ¹³C NMR values given for both the isomers. ¹H NMR values given for one of the isomer.

8e: Following the general procedure F, **8e** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 50:50) as a colourless liquid; Yield: 0.05 g, 66%; IR (CH₂Cl₂): 2926, 2871, 1602, 1453 and 756 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.39-7.28 (m, 4H), 7.00-6.96 (m, 2H), 6.90 (t, *J* = 8.8 Hz, 2H), 6.35 (s, 1H), 4.66 (s, 2H), 4.60 (s, 2H), 4.56 (s, 2H), 4.20-4.12 (m, 4H), 4.0-3.97 (m, 4H), 3.83 (t, *J* = 6.1 Hz, 2H), 3.03 (t, *J* = 6.0 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 171.5, 161.9, 157.4, 156.9, 131.2, 130.9, 129.7, 129.6, 126.4, 126.1, 121.0, 120.9, 112.5, 111.9, 101.3, 70.3, 70.2, 68.6, 68.5, 68.3, 68.1, 66.9, 63.9, 27.7; HRMS (ESI): *m/z* [M + H]⁺ calcd for C₂₄H₂₈NO₆: 426.1916; found 426.1933.

8f: Following the general procedure F, **8f** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 50:50) as a colourless liquid; Yield: 0.074 g, 88%; IR (CH₂Cl₂): 3042, 2871, 1603, 1494 and 754 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.31-7.18 (m, 4H), 6.92-6.86 (m, 2H), 6.82 (d, *J* = 8.1 Hz, 2H), 6.18 (s, 1H), 4.57 (s, 2H), 4.52 (s, 2H), 4.51 (s, 2H), 4.06 (t, *J* = 4.3 Hz, 4H), 3.79-3.73 (m, 6H), 3.64 (s, 4H), 3.01 (t, *J* = 6.5 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 171.2, 161.9, 157.1, 156.9, 130.7, 130.6, 129.5, 129.4, 126.3, 126.0, 120.9, 120.8, 111.9, 111.6, 101.2, 70.9, 70.8, 69.8, 69.7, 68.3, 68.0, 67.6, 67.1, 63.6, 27.5; HRMS (ESI): *m/z* [M + Na]⁺ calcd for C₂₆H₃₁NO₇Na: 492.1998; found 492.2006.

8g: Following the general procedure F, **8g** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 50:50) as a colourless liquid; Yield: 0.073 g, 76%; IR (CH₂Cl₂): 3076, 2865, 1600, 1484 and 753 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.53 (s, 1H), 7.47-7.23 (m, 8H), 7.11-6.92 (m, 3H), 5.91 (s, 1H), 5.17-4.77 (m, 10H), 4.60-4.49 (m, 1H), 4.34 (d, *J* = 13.9 Hz, 1H), 4.54-3.42 (m, 1H), 3.32-3.26 (m, 1H), 2.93-2.74 (m, 2H), 2.62-2.40 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 170.8, 170.5, 161.5, 161.5, 156.6, 156.3, 156.3, 137.6, 137.4, 137.3, 137.2, 135.2, 135.0, 134.7, 130.8, 130.4, 129.7, 129.6, 128.8, 128.8, 128.7, 128.7, 128.7, 128.2, 127.8, 127.8, 127.8, 127.4, 127.4, 127.3, 127.2, 126.8, 121.8, 121.7, 121.5, 116.8, 116.7, 116.7, 116.6, 113.8, 112.9, 112.8, 112.5, 101.2, 100.9, 74.7, 74.6, 73.8, 73.8, 71.6, 71.1, 70.8, 70.4, 66.1, 65.7, 61.1, 60.9, 40.8, 40.7, 40.6, 40.6, 27.5, 27.4; HRMS (ESI): *m/z* [M + H]⁺ calcd for C₃₄H₃₆NO₅: 538.2593; found 538.2596. Reaction was carried out from mixture of isomers of **7c** (*dr* = 60:40) and the compound **8g** was isolated as a mixture of isomers (*dr* =

60:40) and ^{13}C NMR values given for both the isomers. ^1H NMR values given for one of the isomer.

8h: Following the general procedure F, **8h** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 50:50) as a white solid, mp: 150-152 °C; Yield: 0.028 g, 55 %; IR (KBr): 2929, 2851, 1602, 1492 and 753 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.25-7.20 (m, 7H), 7.09-7.05 (m, 1H), 6.92-6.78 (m, 3H), 6.62-6.54 (m, 1H), 6.28 (s, 1H), 5.38 (s, 2H), 5.15 (s, 2H), 4.53 (d, J = 6.5 Hz, 4H), 4.41 (s, 2H), 3.72 (t, J = 5.4 Hz, 2H), 2.89 (t, J = 5.2 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 171.9, 161.8, 156.9, 137.2, 133.1, 132.1, 131.7, 130.0, 129.9, 129.4, 128.9, 127.3, 126.8, 125.6, 125.3, 120.7, 120.4, 112.0, 111.4, 101.2, 69.7, 69.4, 69.2, 66.9, 66.8, 64.0, 27.5; HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{28}\text{H}_{27}\text{NO}_5\text{Na}$: 480.1787; found 480.1816.

8i: Following the general procedure F, **8i** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 50:50) as a colourless liquid; Yield: 0.017 g, 35 %; IR (KBr): 2929, 2856, 1602, 1495 and 756 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.48 (s, 1H), 7.37-7.22 (m, 7H), 6.95-6.90 (m, 4H), 5.85 (s, 1H), 5.01 (s, 2H), 4.97 (s, 2H), 4.52 (s, 2H), 4.47 (d, J = 7.1 Hz, 4H), 3.59 (t, J = 6.6 Hz, 2H), 2.77 (t, J = 6.6 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 171.0, 161.8, 157.4, 157.1, 137.5, 137.4, 131.0, 130.9, 129.7, 129.6, 128.8, 128.1, 127.8, 127.5, 127.1, 126.7, 126.3, 121.2, 121.1, 112.6, 112.3, 101.0, 71.0, 70.4, 68.4, 68.3, 67.3, 63.9, 27.5; HRMS (ESI): m/z $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{28}\text{H}_{28}\text{NO}_5$: 458.1967; found 458.1961.

General procedure for the syntheses of macrocycles 9a-d (Procedure G). A mixture of **6f** (0.06 mmol), $\text{Na}_2\text{S} \cdot x\text{H}_2\text{O}$ (70 mg), CuI (10 mol %), 1,10-phen (15 mol %) and DMF (0.5 mL) was stirred at 90 °C for 6h under open air atmosphere. After this period, the vial was cooled to room temperature. Then the resulting mixture was diluted with water (4 mL). The mixture was filtered through a filtration funnel and the washed with ethyl acetate (4 x 5 mL). The combined layers were extracted using ethyl acetate (3 x 5 mL) and the combined organic layers were dried over anhydrous Na_2SO_4 . The solvent was removed under reduced pressure and the residue was purified by silica gel chromatography (EtOAc : Hexane) which gave the crown/polyether macrocycle **9**. See the corresponding Scheme 6 for specific examples.

9a: Following the general procedure G, **9a** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 50:50) as a white solid, mp: 90-92 °C; Yield: 0.018 g, 17%; IR (CH_2Cl_2): 2872, 1602, 1493, 1358 and 754 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.34 (d, J = 7.5 Hz, 2H), 7.14 (t, J = 7.3 Hz, 2H), 6.90 (t, J = 7.4 Hz, 2H), 6.80 (s, 2H), 6.76 (d, J = 8.2 Hz, 2H), 4.67 (s, 4H), 4.47 (s, 4H), 4.01 (t, J = 4.7 Hz, 4H), 3.82 (t, J = 4.7 Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ 156.6, 141.6, 129.6, 128.9, 126.8, 126.5, 121.1, 111.9, 70.5, 68.8, 66.6, 65.8; HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{24}\text{H}_{26}\text{O}_5\text{SNa}$: 449.1399; found 449.1408. This compound was crystallized using a mixture of EtOAc and Hexanes and confirmed by single crystal X-ray structure analysis.

9b: Following the general procedure G, **9b** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 50:50) as a colourless; Yield: 0.058 g, 29%; IR (CH_2Cl_2): 2871, 1602, 1493, 1248 and 735 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.33 (d, J = 7.4 Hz, 2H), 7.19 (t, J = 7.8 Hz, 2H), 6.90 (t, J = 7.4 Hz, 2H), 6.83 (s, 2H), 6.78 (d, J = 8.1 Hz, 2H),

4.66 (s, 4H), 4.55 (s, 4H), 4.06 (t, $J = 4.3$ Hz, 4H), 3.76 (t, $J = 4.3$ Hz, 4H), 3.61-3.59 (m, 4H), 3.53-3.51 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ 156.7, 141.8, 130.0, 129.0, 126.8, 126.1, 120.8, 111.6, 70.9, 70.7, 69.7, 68.2, 66.9, 66.6; HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{28}\text{H}_{34}\text{O}_7\text{SNa}$: 537.1923; found 537.1927.

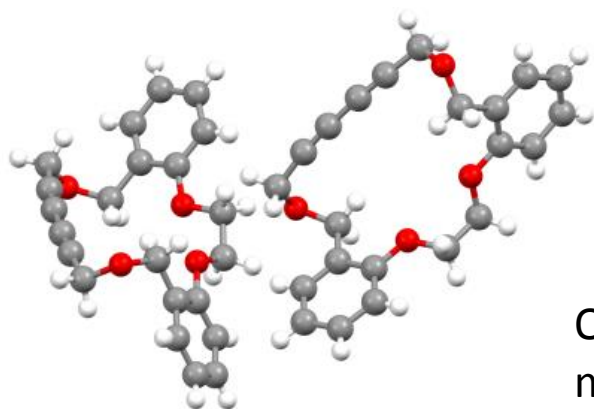
9c: Following the general procedure G, **9c** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 10:90) as a white solid, mp: 84-86 $^\circ\text{C}$; Yield: 0.068 g, 52%; IR (CH_2Cl_2): 2936, 2858, 1603, 1494, 1248 and 753 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.38 (d, $J = 7.4$ Hz, 2H), 7.28 (dd, $J = 13.9, 1.7$ Hz, 2H), 6.95 (t, $J = 7.3$ Hz, 2H), 6.90 (s, 2H), 6.88 (d, $J = 7.7$ Hz, 2H), 4.74 (s, 4H), 4.60 (s, 4H), 4.01 (t, $J = 5.98$ Hz, 4H), 1.84-1.80 (m, 4H), 1.59-1.55 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ 157.4, 141.7, 130.4, 129.2, 126.4, 126.0, 120.3, 111.6, 67.9, 67.2, 67.1, 29.3, 25.9; HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{26}\text{H}_{30}\text{O}_4\text{SNa}$: 461.1762; found 461.1765. This compound was crystallized using a mixture of EtOAc and Hexanes and confirmed by single crystal X-ray structure analysis.

9d: Following the general procedure G, **9d** was obtained after purification by column chromatography on silica gel (EtOAc:Hexanes = 50:50) as a yellow liquid; Yield: 0.017 g, 55%; IR (CH_2Cl_2): 2918, 1599, 1453, 1290 and 753 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.36 (d, $J = 6.2$ Hz, 2H), 7.30 (s, 2H), 7.25 (t, $J = 7.8$ Hz, 2H), 7.03-6.97 (m, 4H), 5.25 (s, 4H), 3.87 (s, 4H), 3.66 (t, $J = 6.6$ Hz, 4H), 3.56 (s, 4H), 2.70 (t, $J = 6.6$ Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ 155.9, 140.5, 130.7, 128.2, 128.0, 125.9, 121.5, 112.1, 70.9, 70.3, 65.8, 31.4, 30.4; HRMS (ESI): m/z $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{26}\text{H}_{30}\text{O}_4\text{S}_3\text{Na}$: 525.1203; found 525.1196.

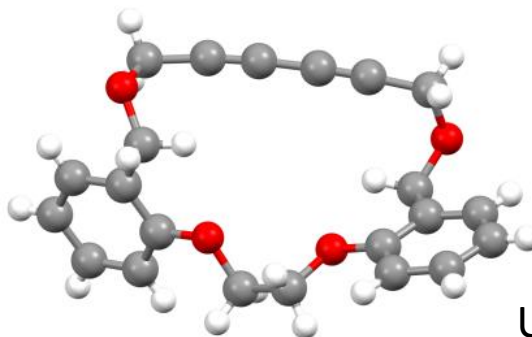
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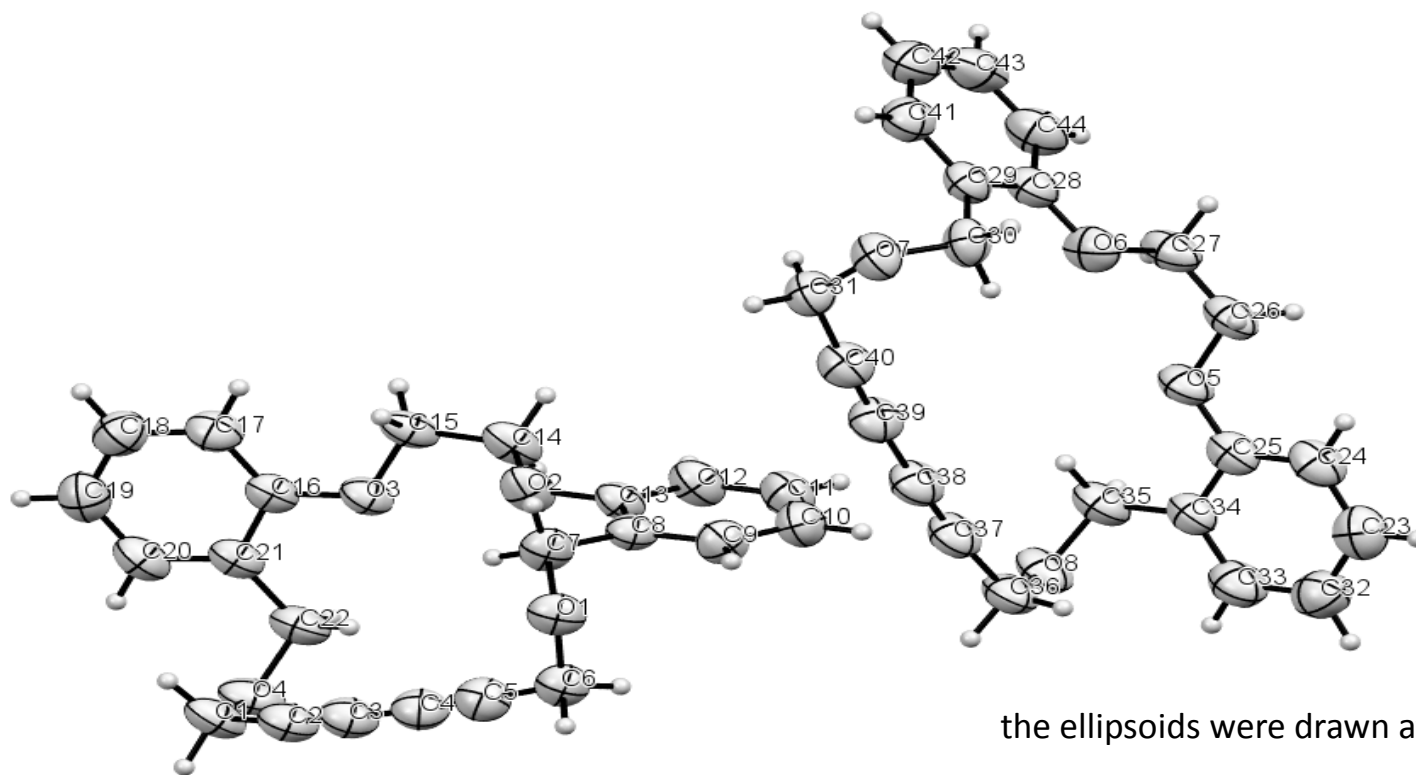
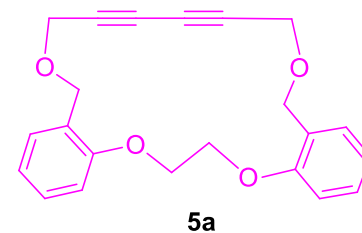
X-ray structure of Compound 5a



One of the two
molecules in unit cell

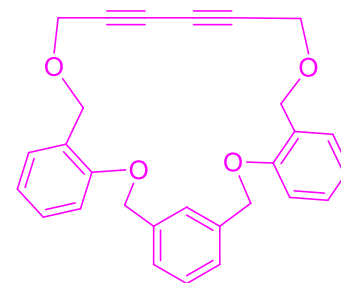
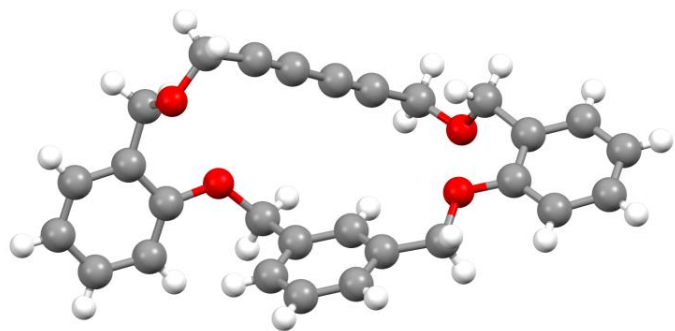


Unit cell contains two molecules
in the asymmetric unit

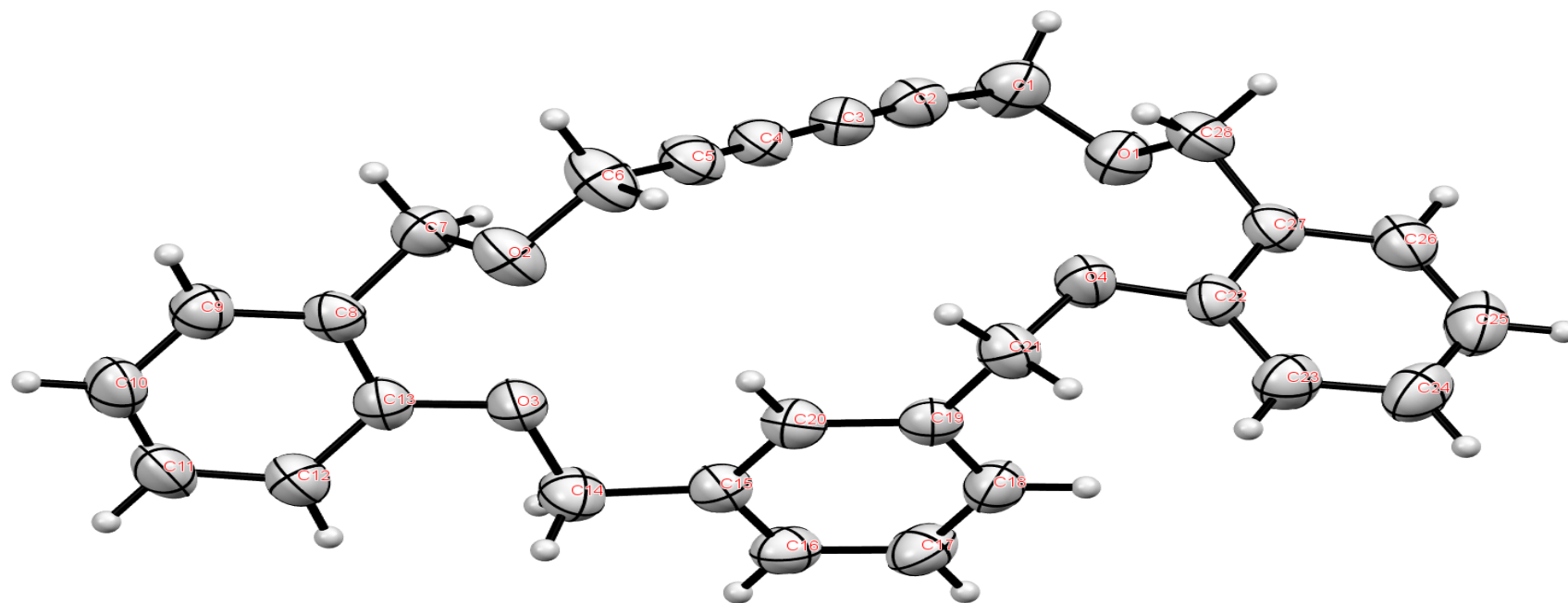


the ellipsoids were drawn at 50% probability level

X-ray structure of Compound **5f**

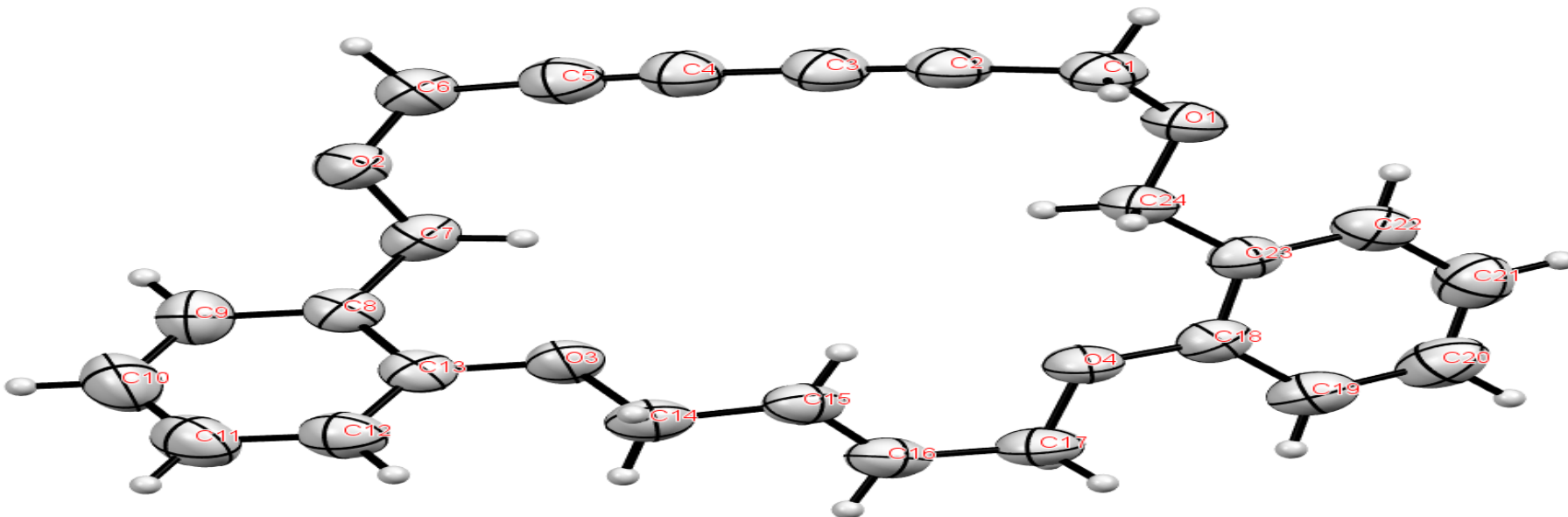
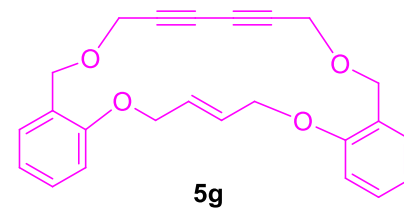
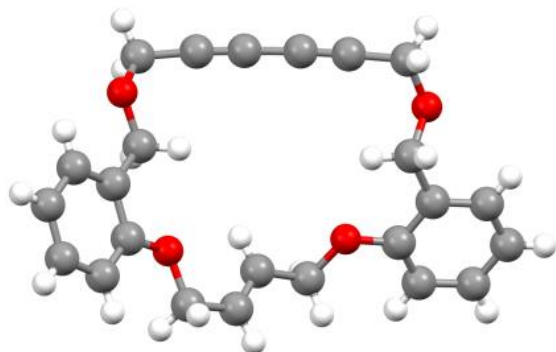


5f



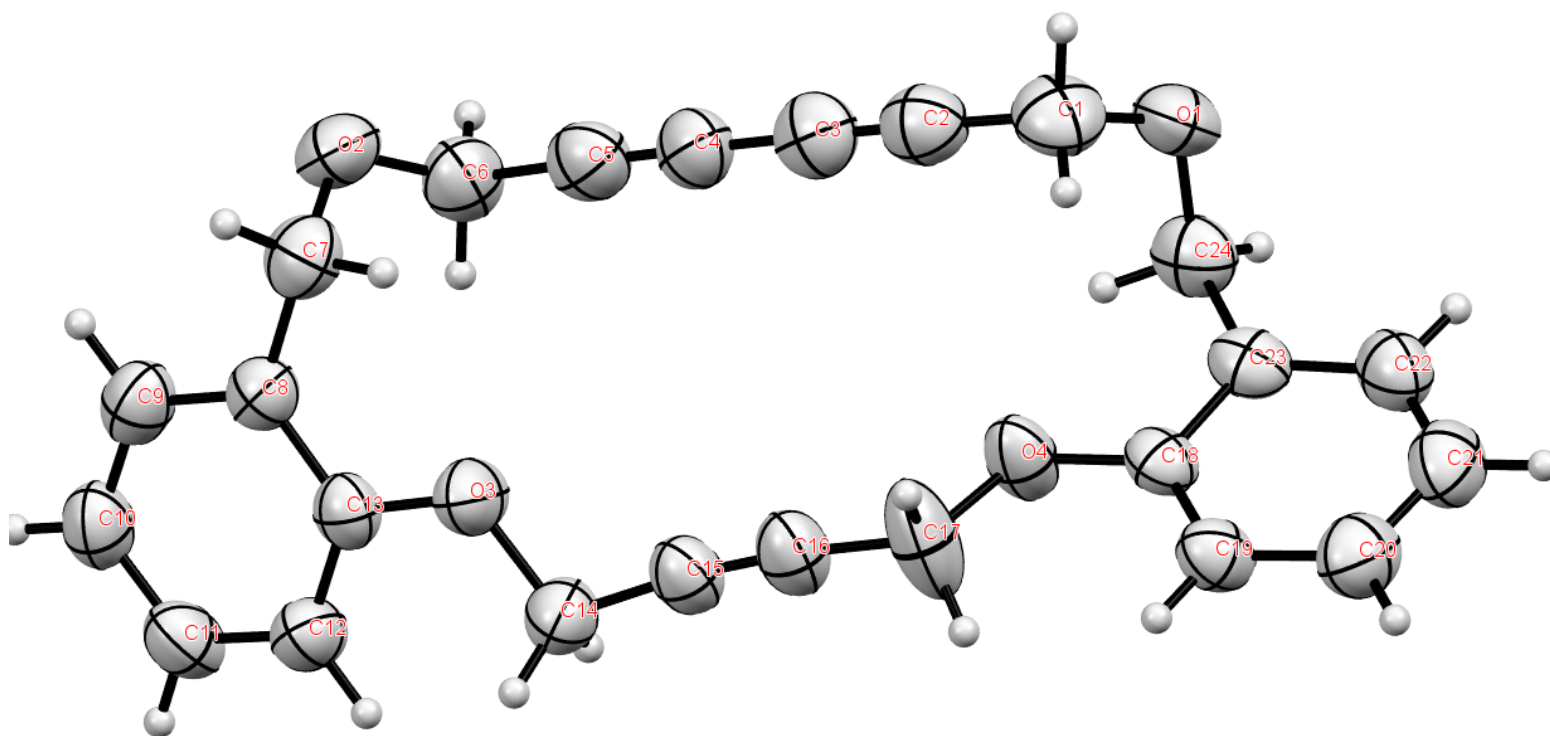
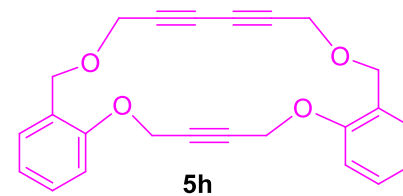
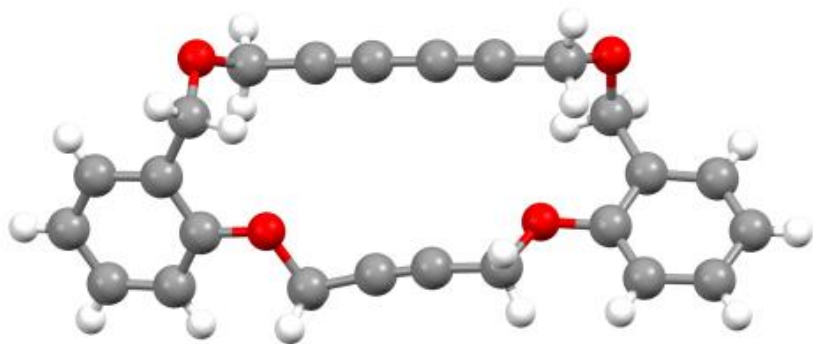
the ellipsoids were drawn at 50% probability level

X-ray structure of Compound **5g**



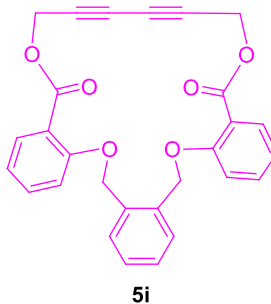
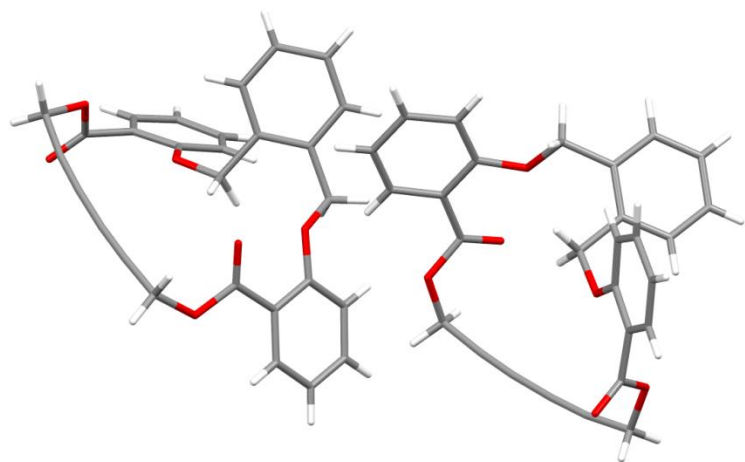
the ellipsoids were drawn at 50% probability level

X-ray structure of compound **5h**

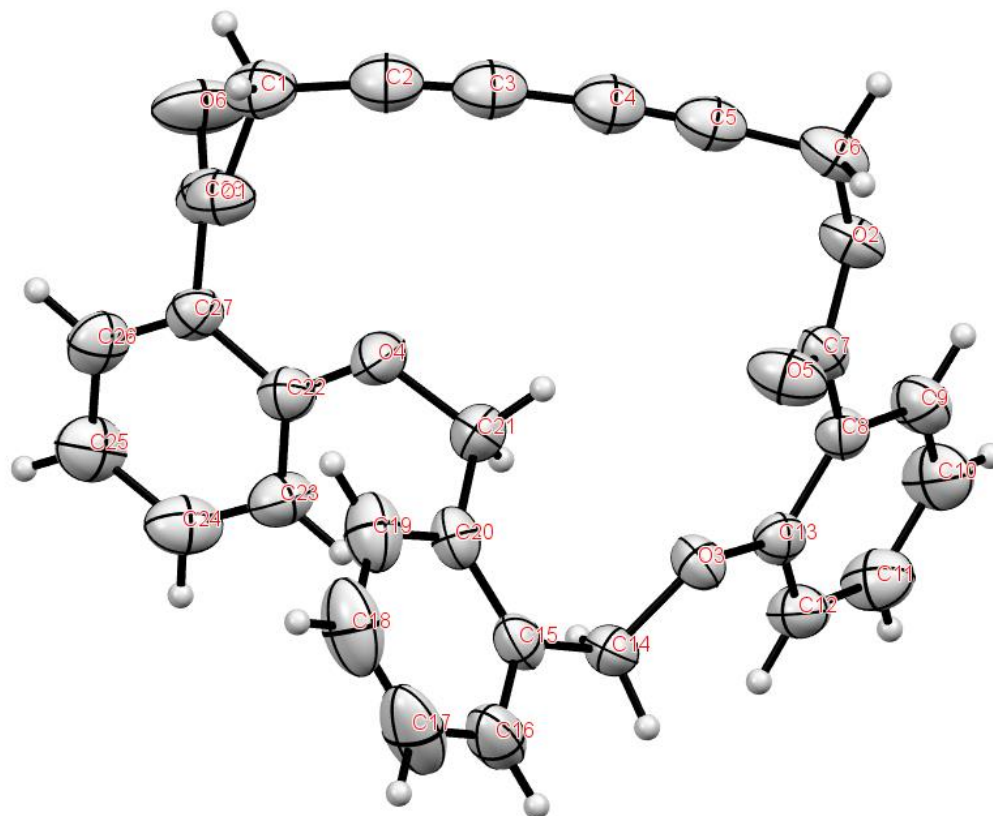


the ellipsoids were drawn at 50% probability level

X-ray structure of compound **5i**

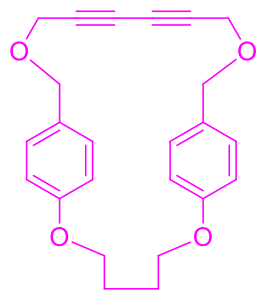


Unit cell contains two molecules in the asymmetric unit
Only one molecule is shown in article

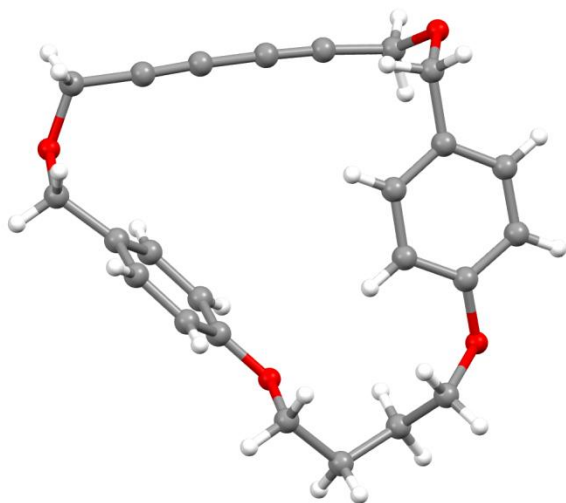


the ellipsoids were drawn at 50% probability level

X-ray structure of compound **5k**

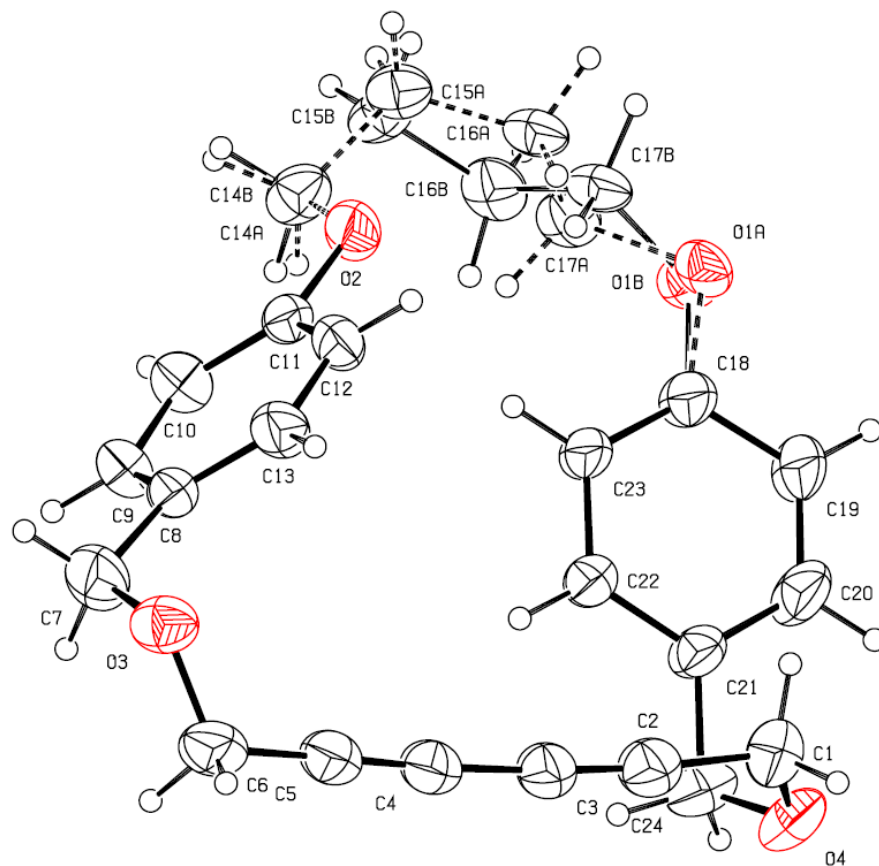


5k



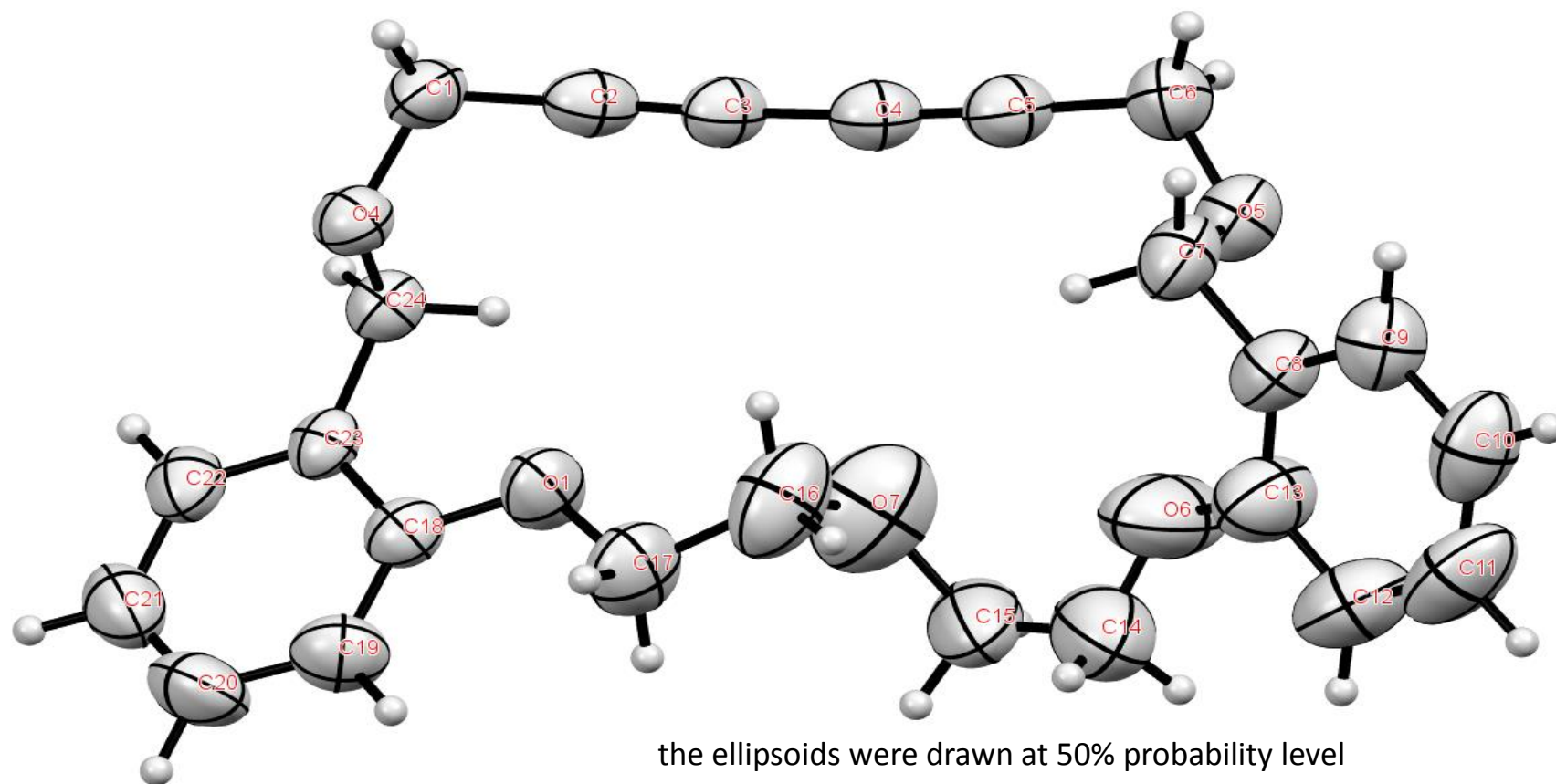
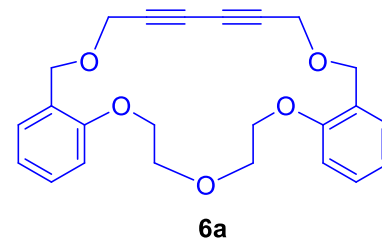
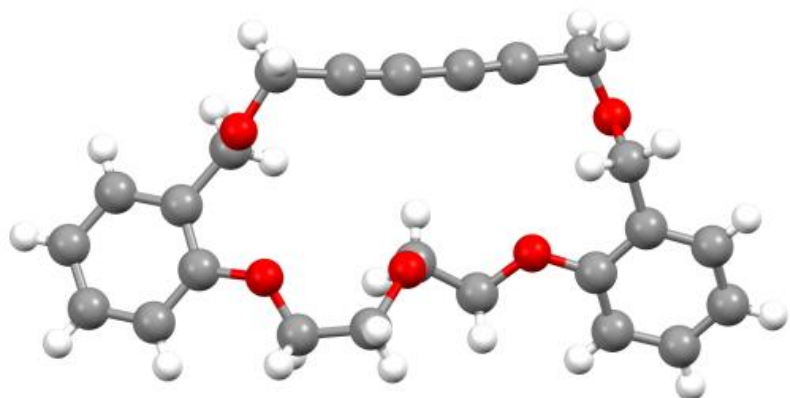
the structure of one of the parts

This molecule has disorder at C14A (C14B), C15A (C15B), C16A (C16B), C17A (C17B) and O1A (O1B) (perhaps due to one more Conformation). In the article, the structure of one of the parts is shown



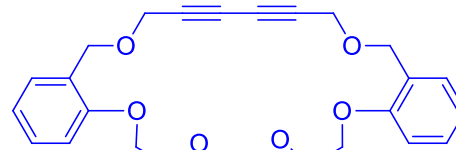
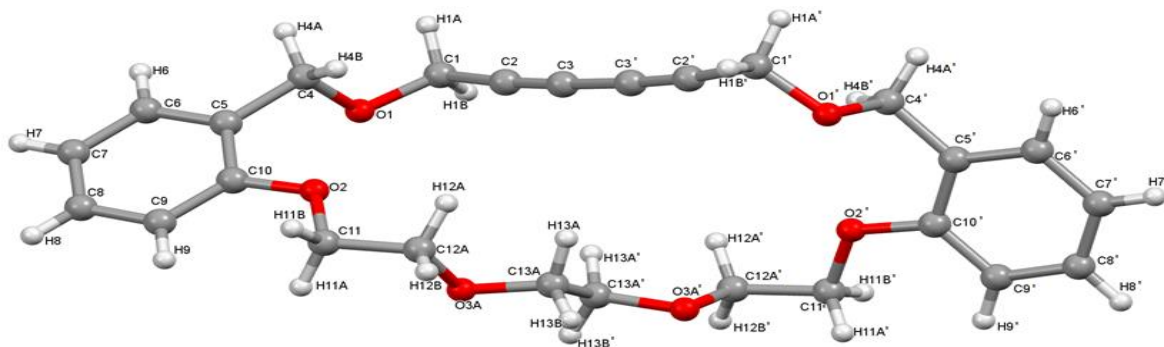
the ellipsoids were drawn at 30% probability level

X-ray structure of compound 6a



the ellipsoids were drawn at 50% probability level

X-ray structure of compound **6c**

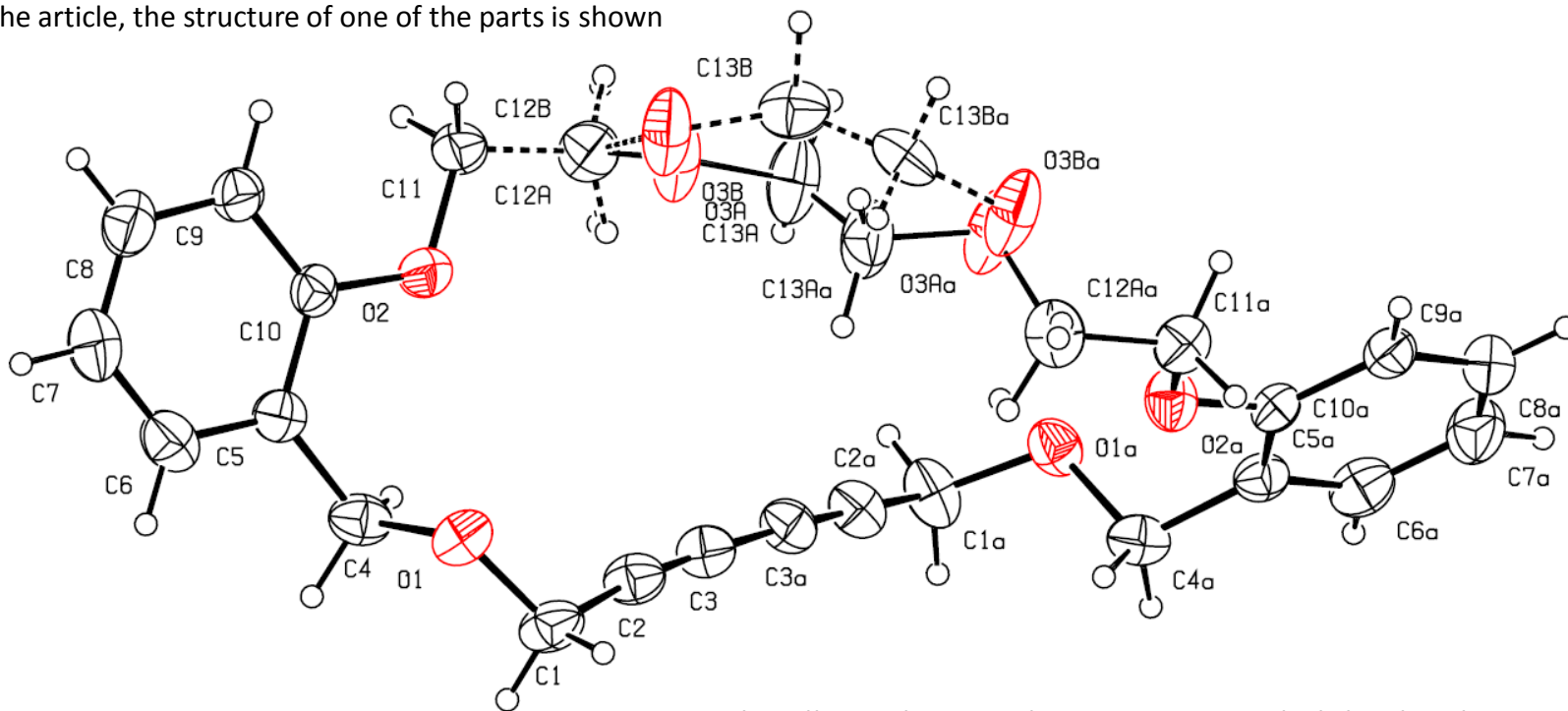


6c

Only half of the molecule is present in the asymmetric unit of the X-ray structure of the compound **6c**.

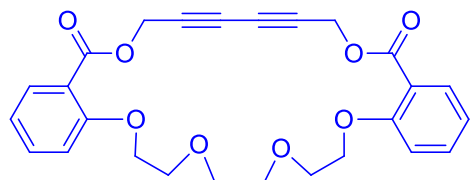
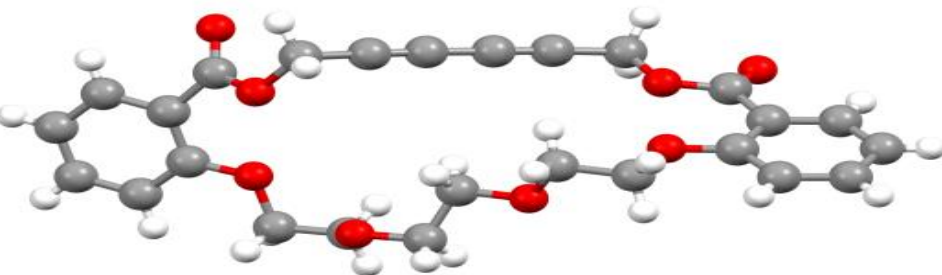
The structure of one of the parts

This molecule has disorder at C12A (C12B), O3A (O3B), C13A (C13B), C13Aa (C13Ba) AND O3Aa (O3Ba) (perhaps due to one more Conformation).
In the article, the structure of one of the parts is shown

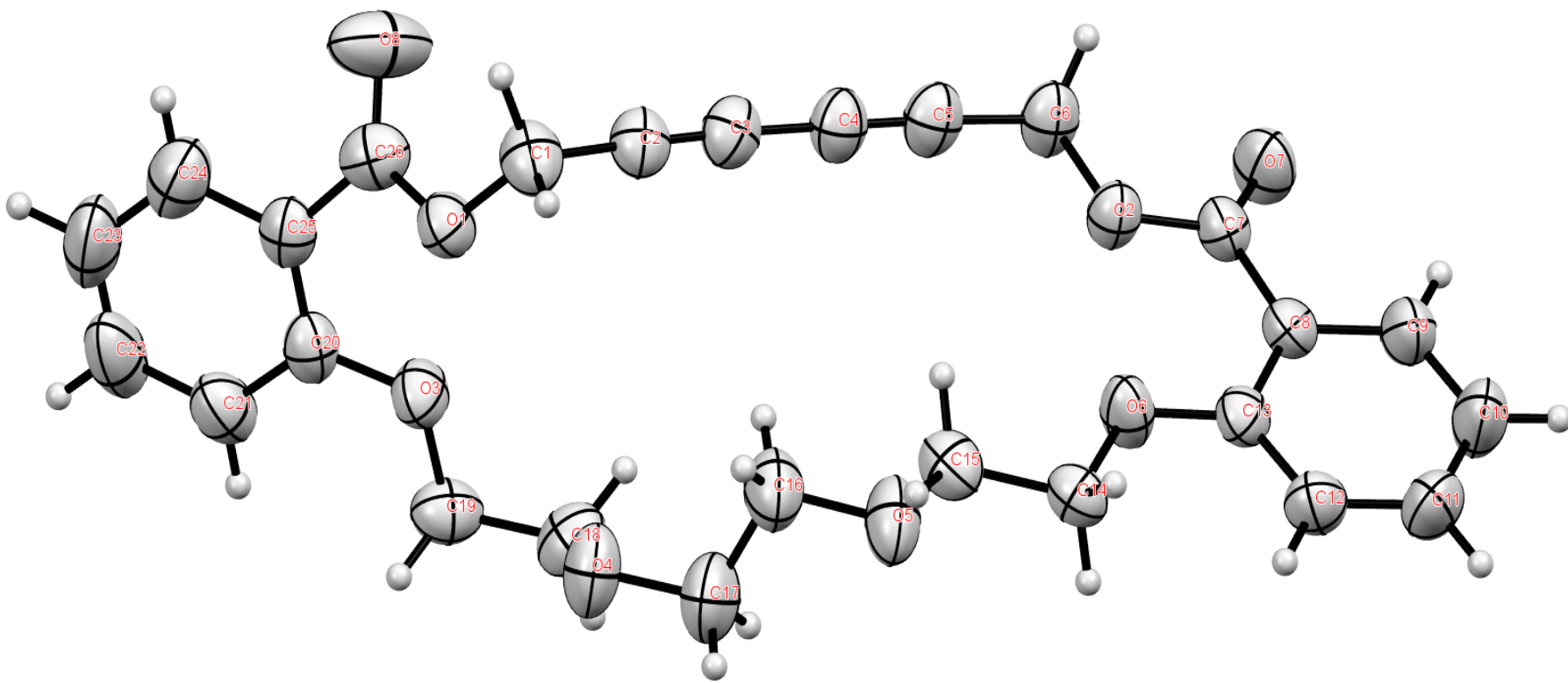


the ellipsoids were drawn at 30% probability level

X-ray structure of compound **6g**

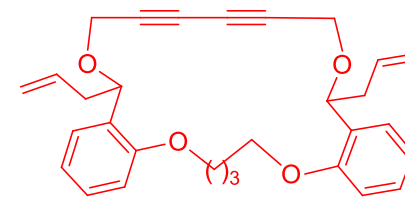
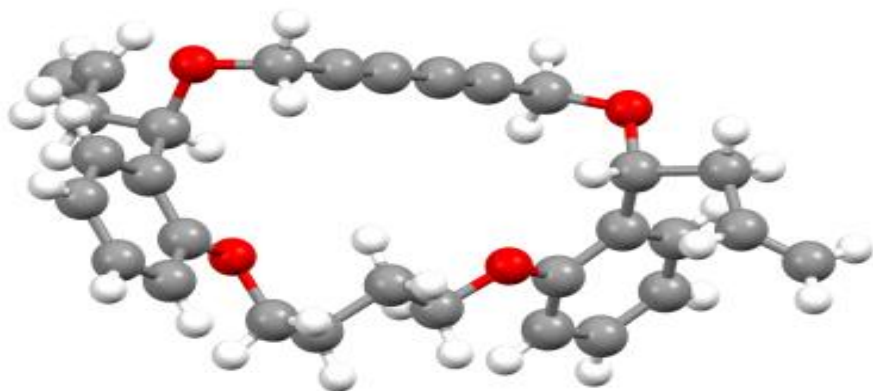


6g

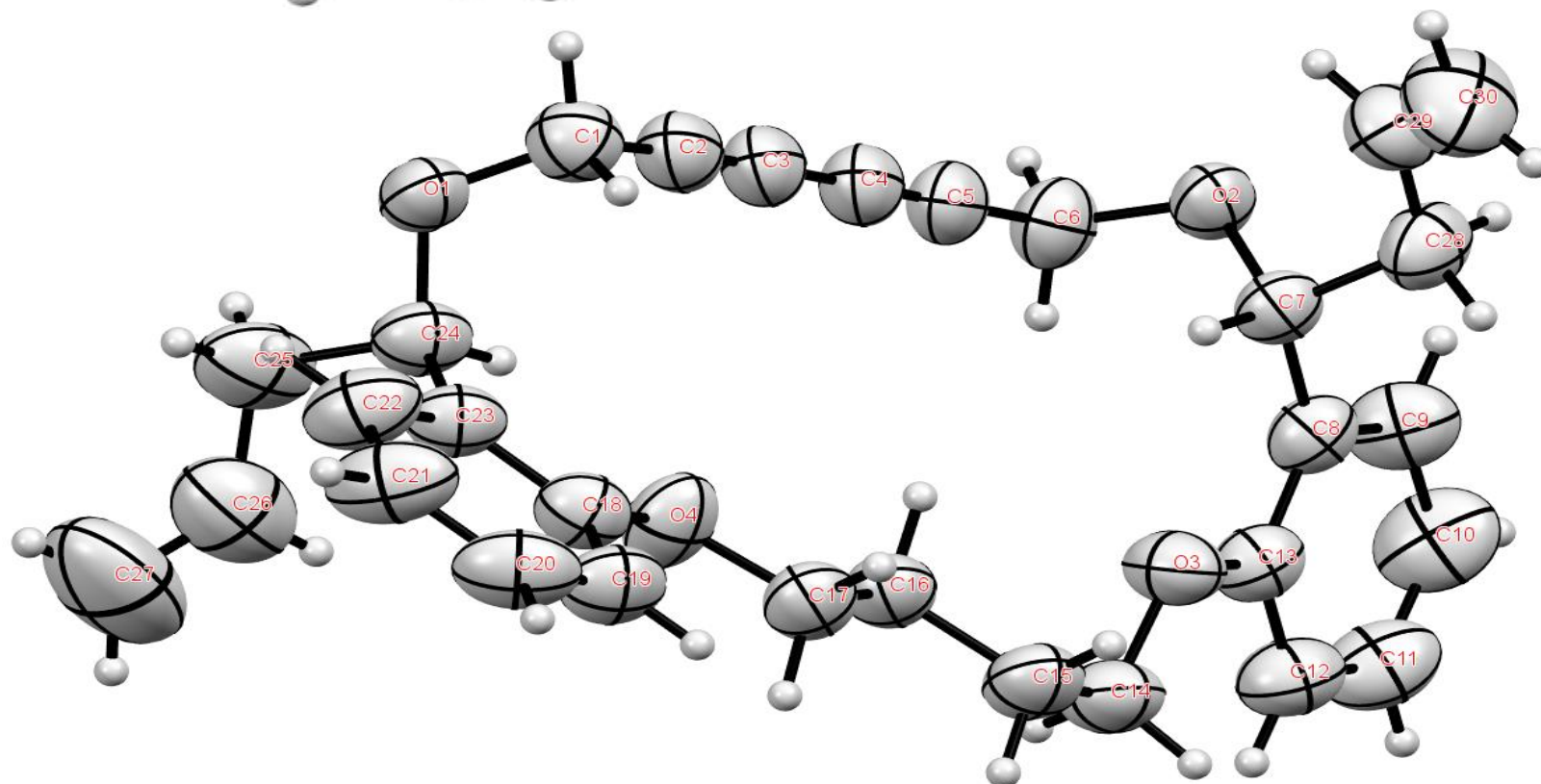


the ellipsoids were drawn at 50% probability level

X-ray structure of Compound **7b**

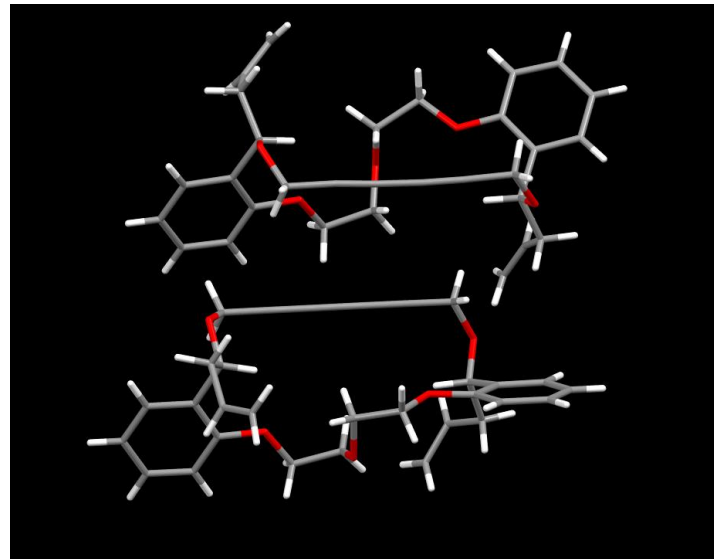
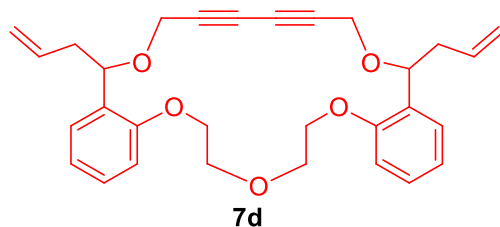
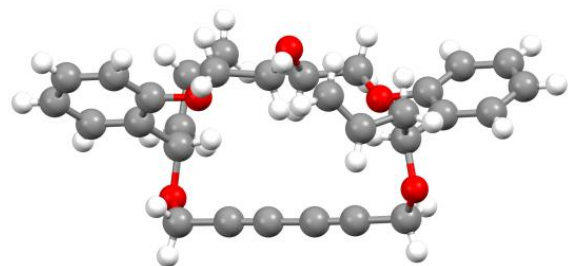


7b



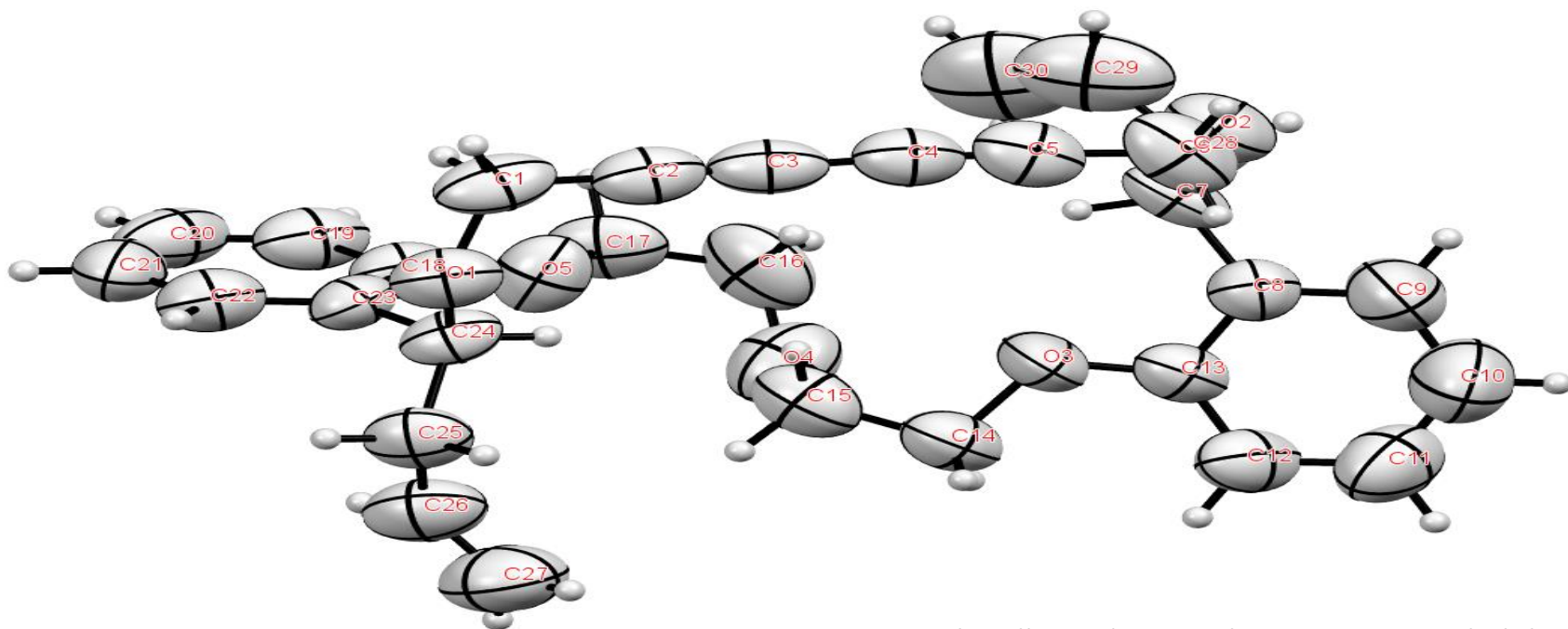
the ellipsoids were drawn at 50% probability level

X-ray structure of compound **7d**



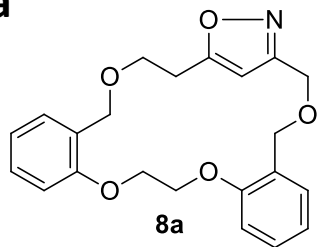
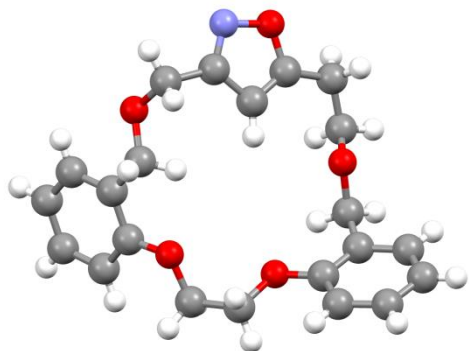
One of the molecules present in Unit cell

Unit cell contains two molecules
in the asymmetric unit



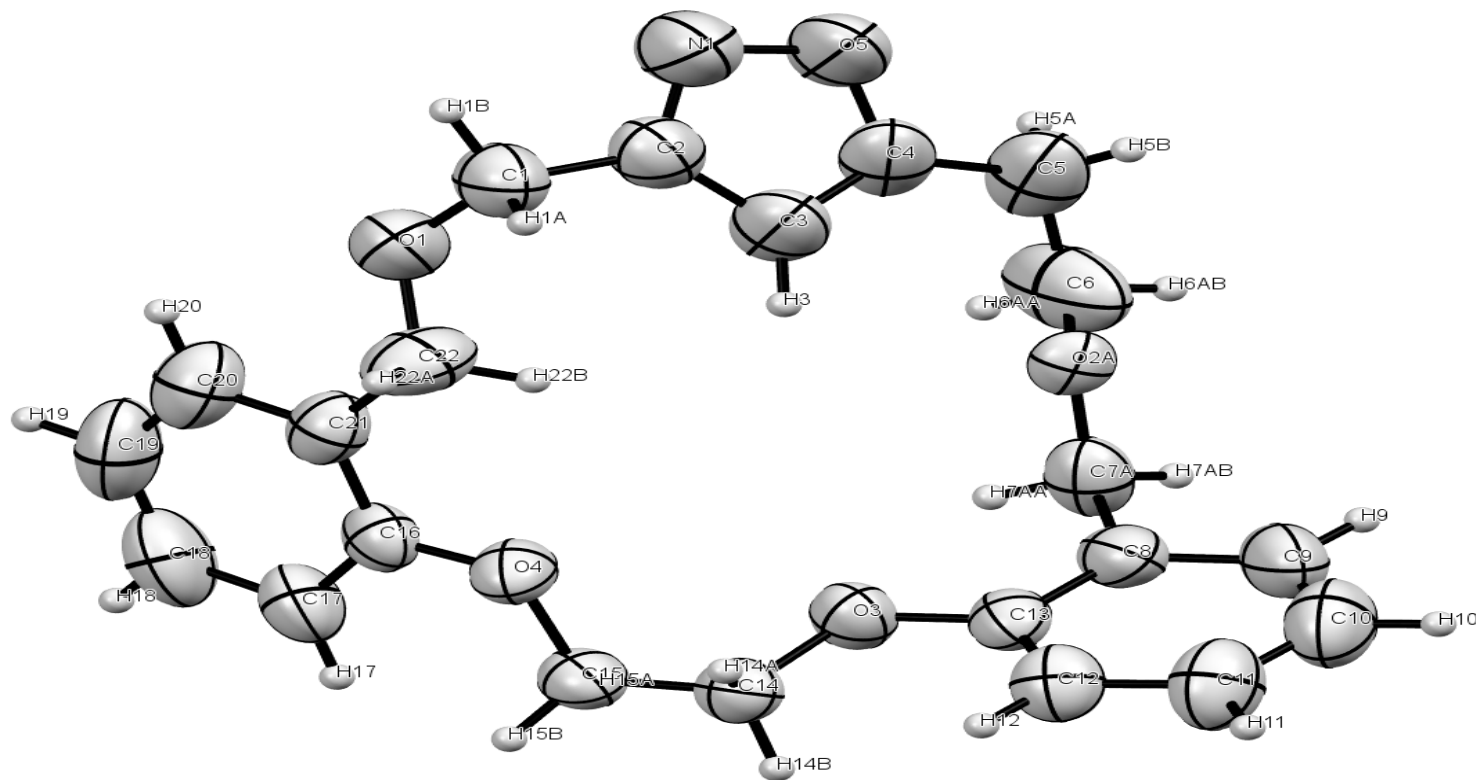
the ellipsoids were drawn at 50% probability level

X-ray structure of compound **8a**



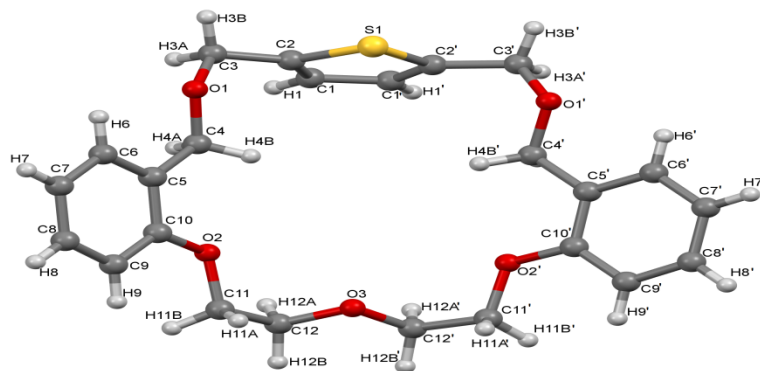
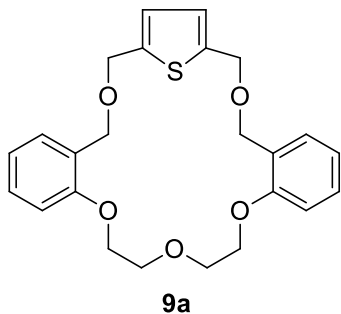
This molecule has disorder at C6, C7 and O2 (perhaps due to one more Conformation)

In the article as well as here the structure of one of the parts is shown

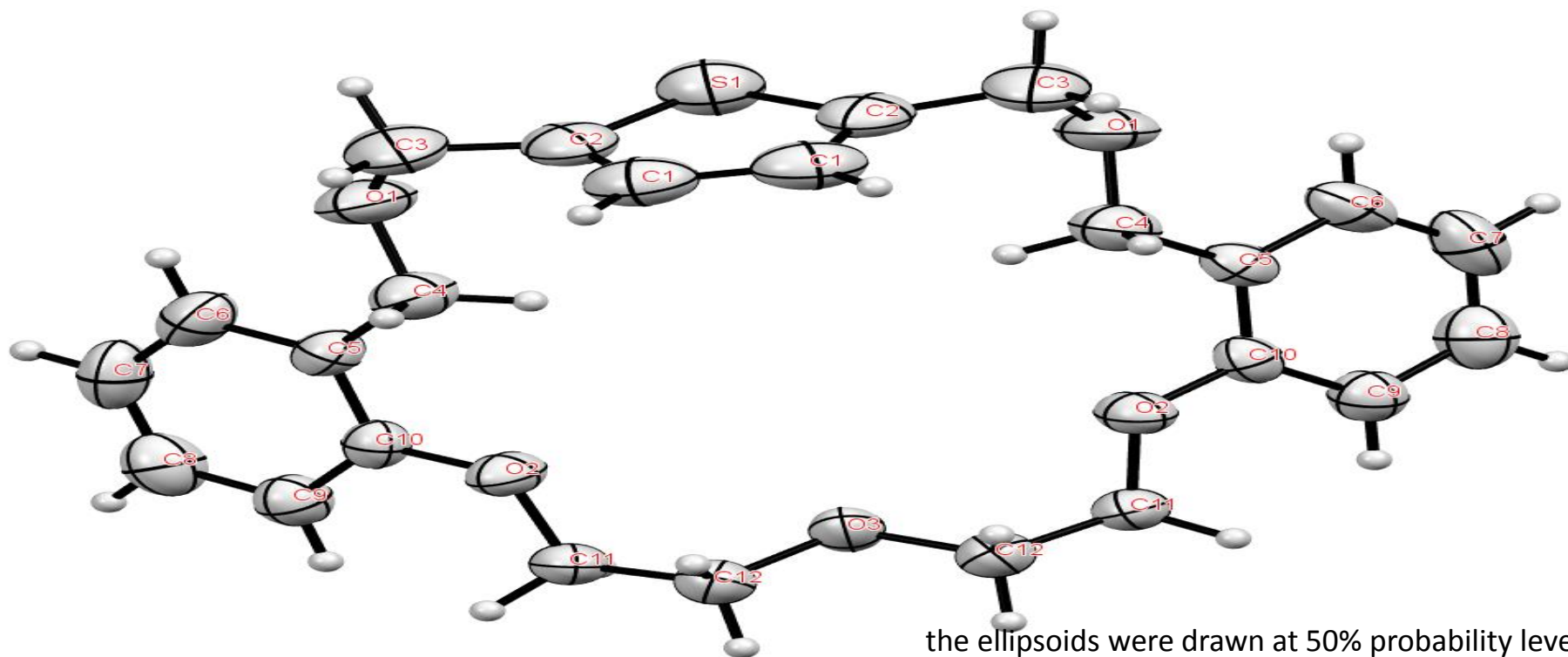


the ellipsoids were drawn at 50% probability level

X-ray structure of compound **9a**

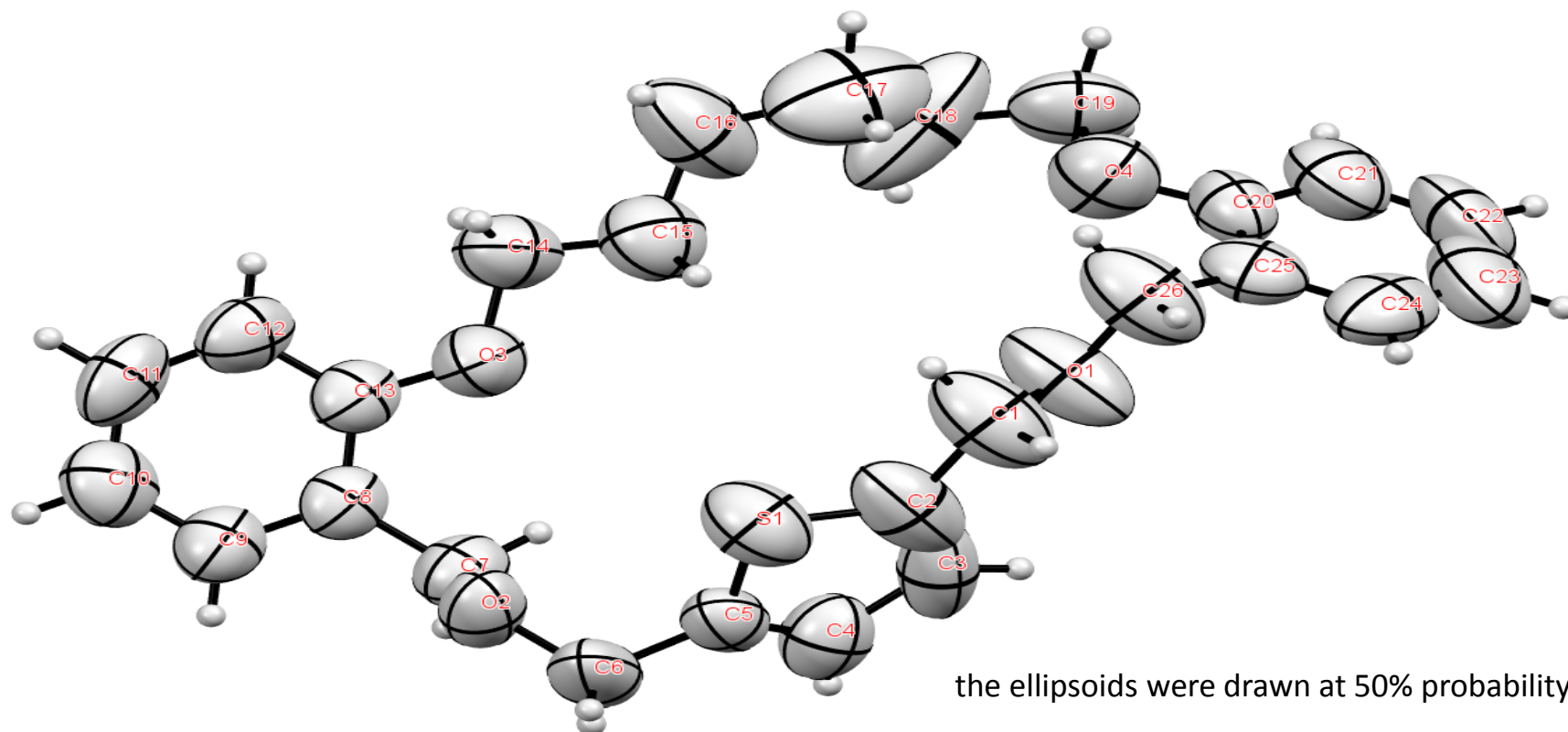
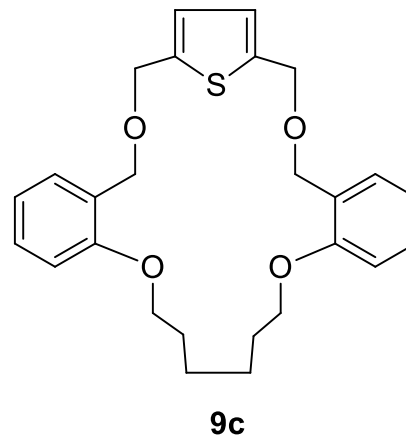
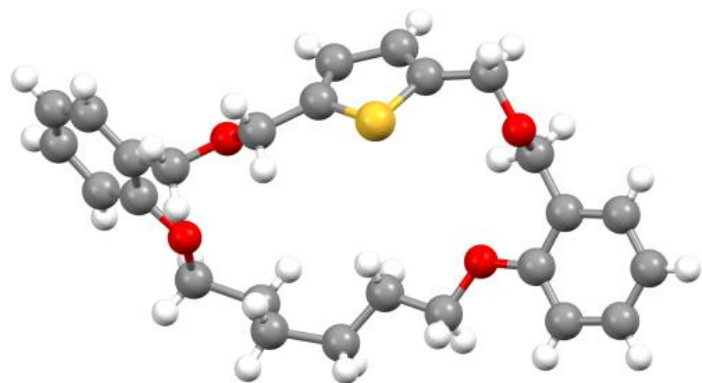


Only half of the molecule is present in the asymmetric unit of the X-ray structure of the compound **9a**. Therefore, the atoms at the right hand side of X-ray structure of **9a** (shown with a prime (') label) are at equivalent position $(x, 3/2-y, z)$ with respect to the atoms on the left hand side



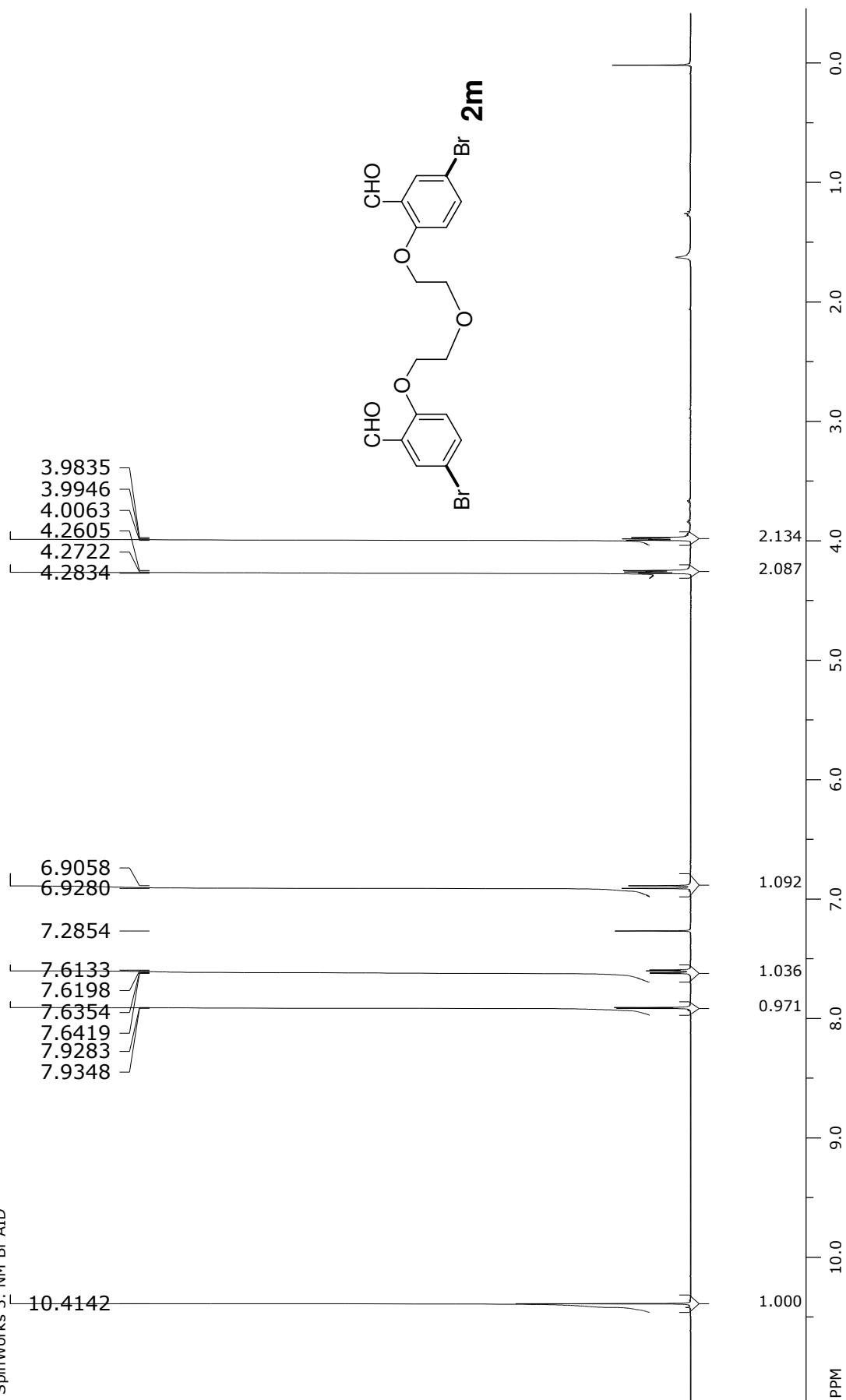
the ellipsoids were drawn at 50% probability level

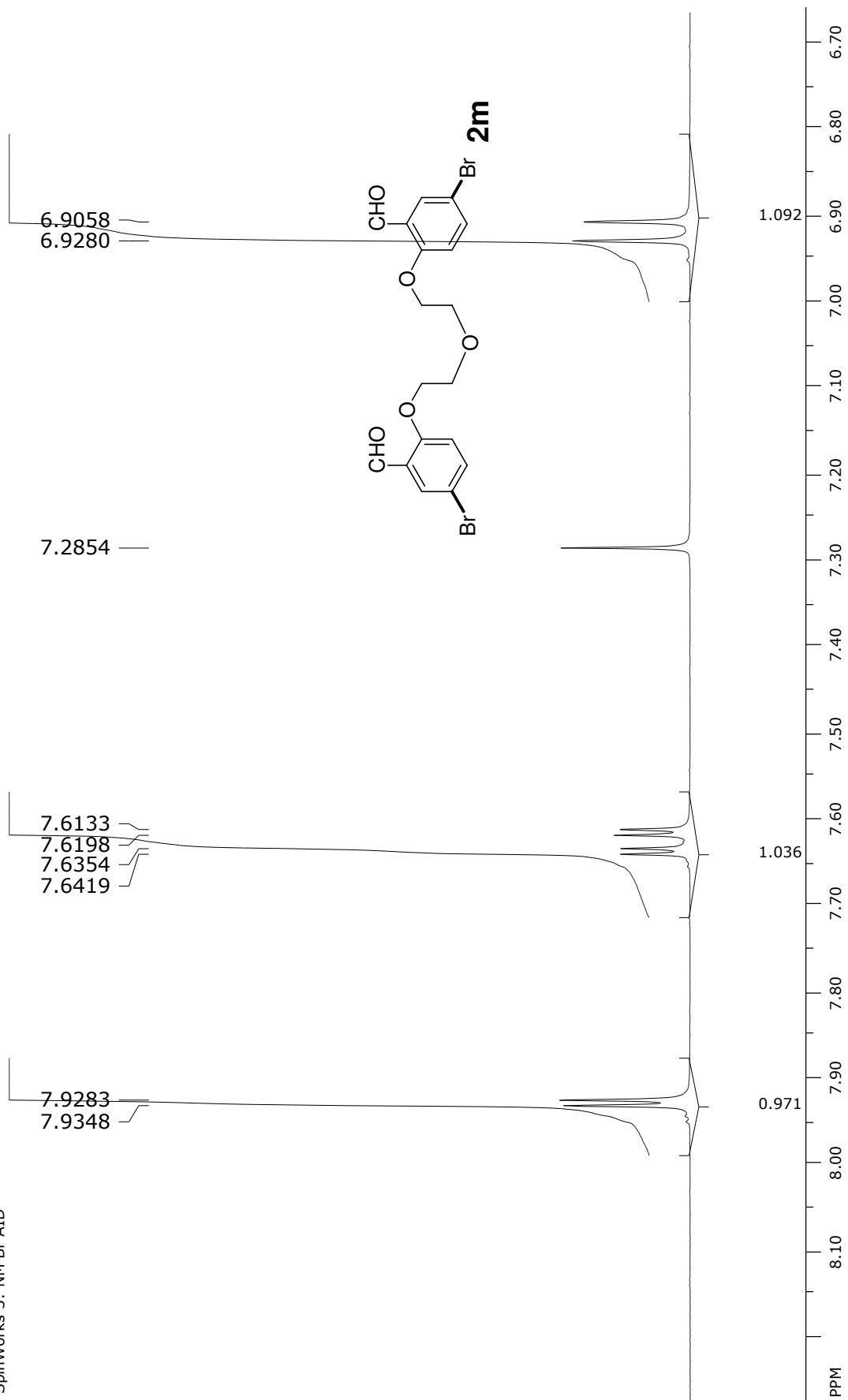
X-ray structure of compound **9c**



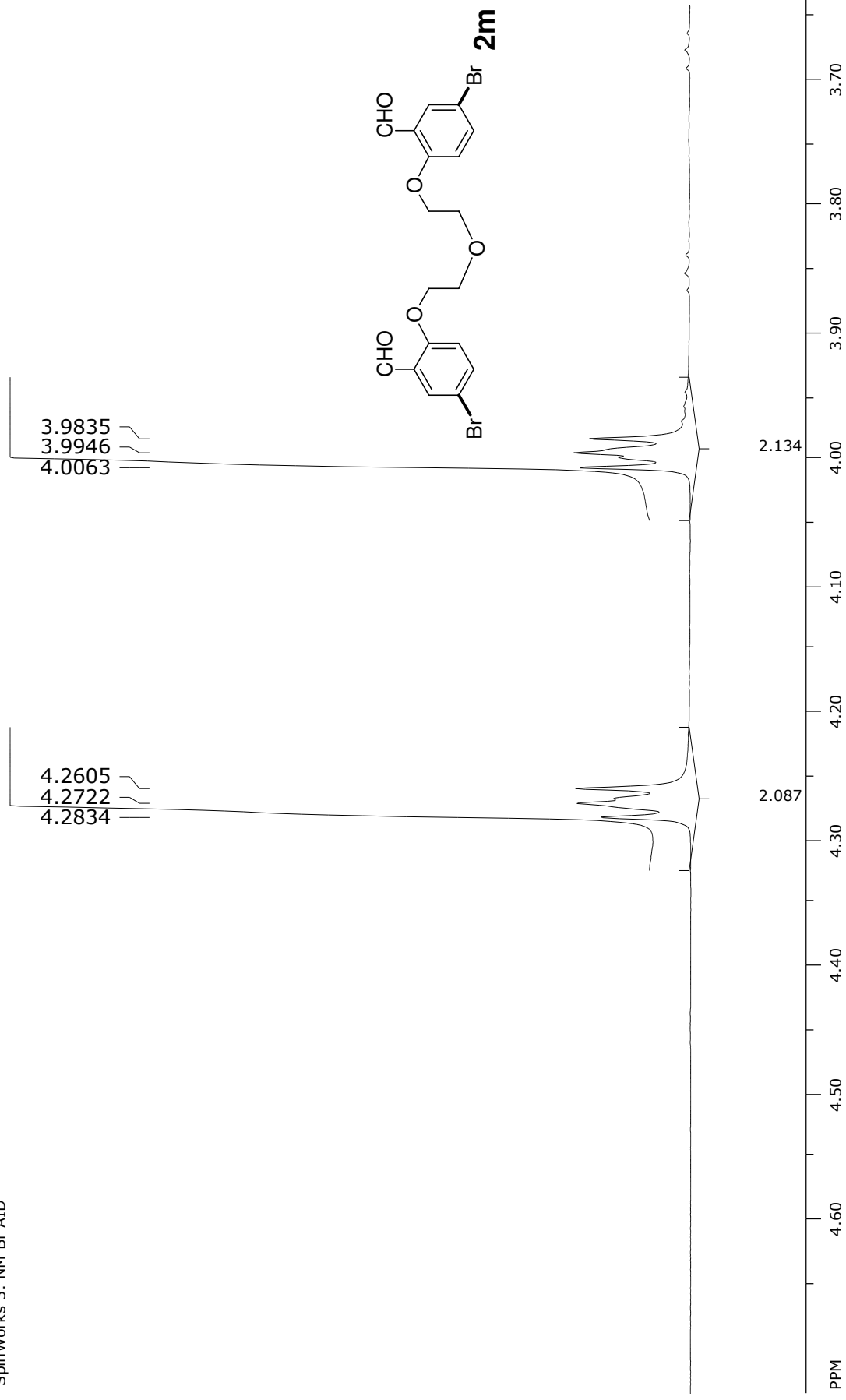
the ellipsoids were drawn at 50% probability level

SpinWorks 3: NM Br AID





SpinWorks 3: NM Br AID



SpinWorks 3: NM Br AID

188.2193 —

159.9381 —

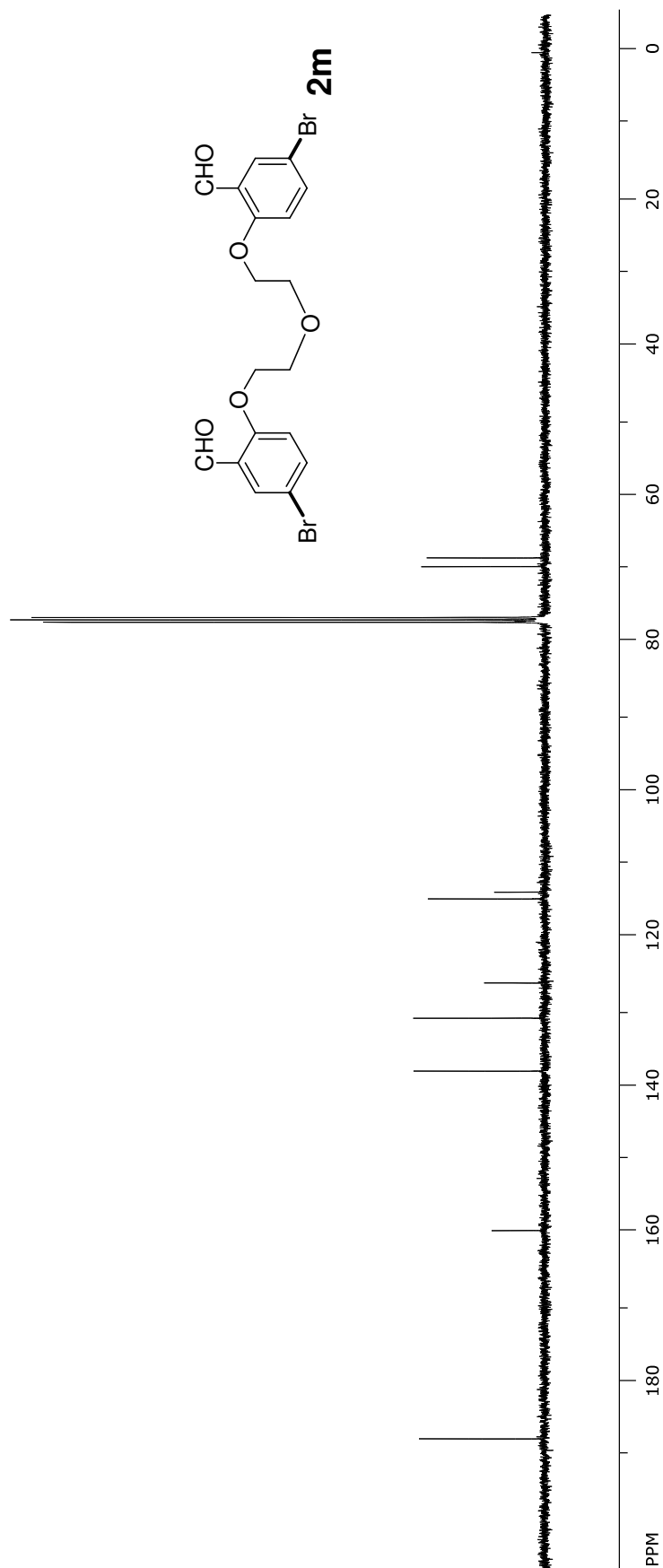
138.3032 —

131.1078 —

126.3318 —

114.0001 —
114.9179 —

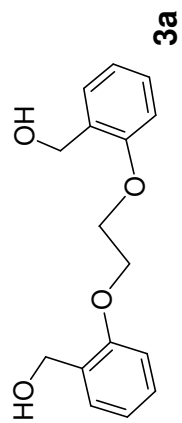
68.6281 —
69.8030 —
76.7233 —
77.0413 —
77.2454 —
77.3585 —



SpinWorks 3: Nm297 Proton test

6.8780
6.8986
6.9049
6.9176
6.9256
6.9358
6.9443
6.9546
6.9629
6.9815
7.1973
7.2174
7.2215
7.2411
7.3241
7.3434
7.3482
7.3670

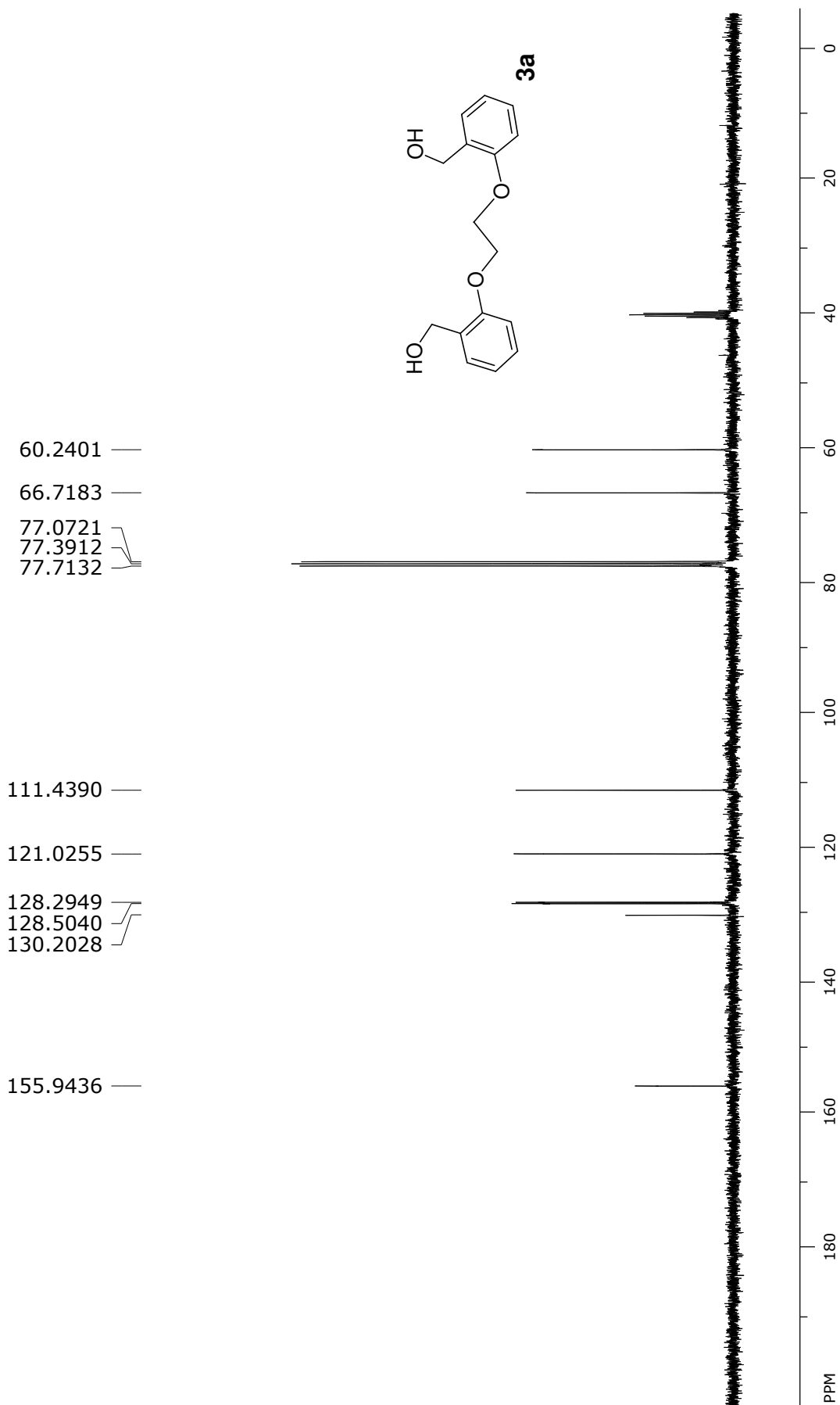
4.3460
4.6145



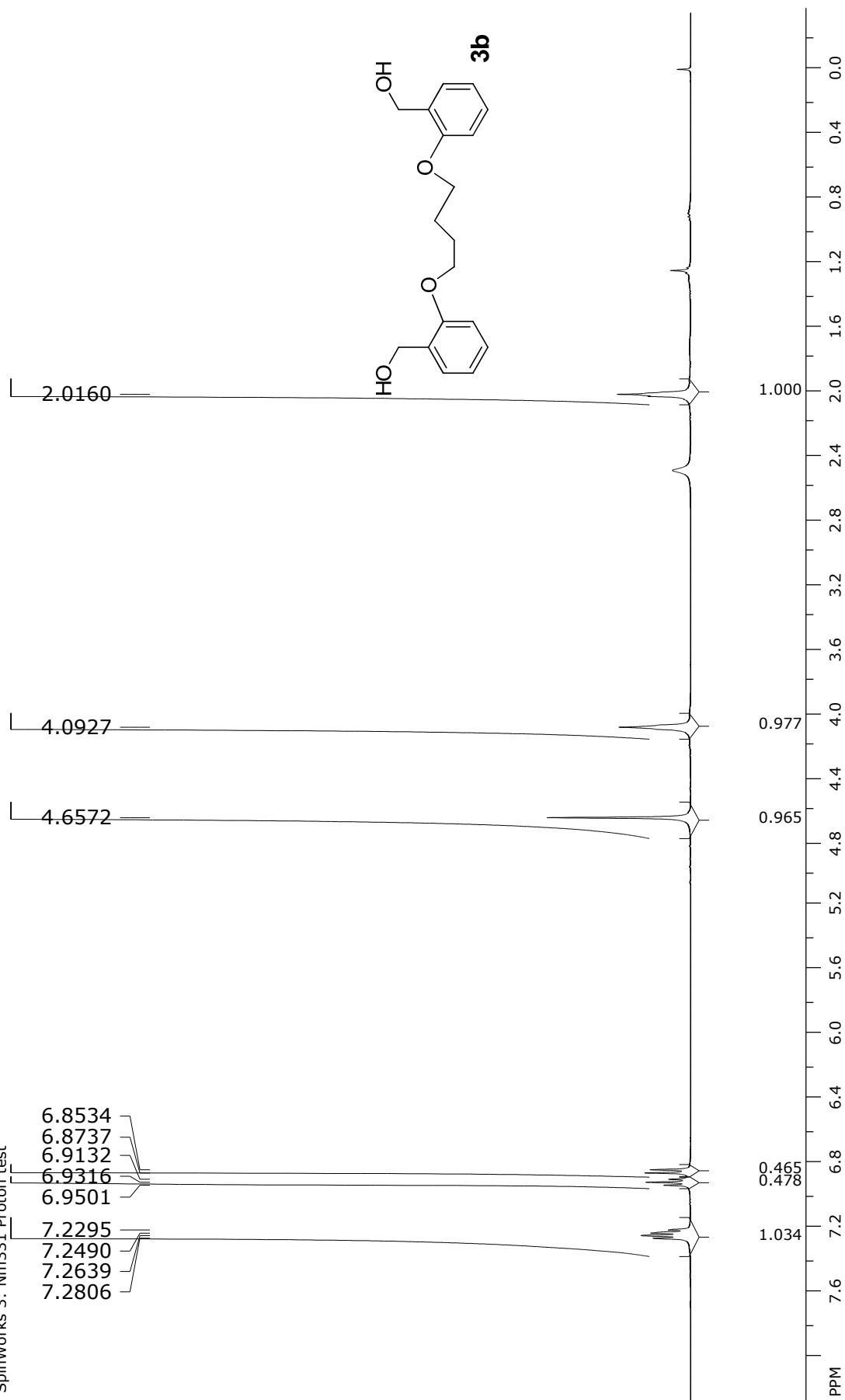
0.998
1.014

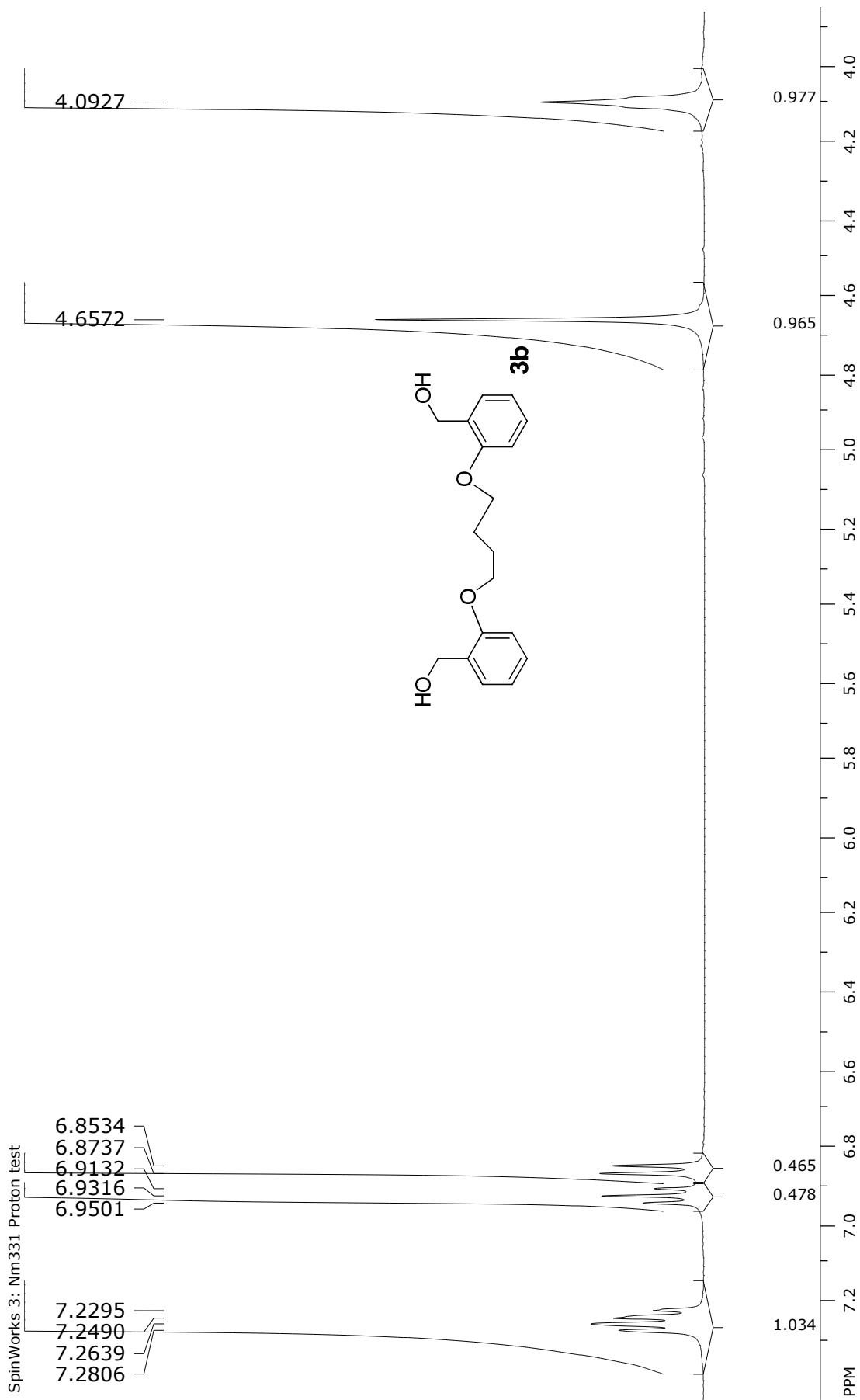
1.025
0.510
0.504

PPM 8.4 8.0 7.6 7.2 6.8 6.4 6.0 5.6 5.2 4.8 4.4 4.0 3.6 3.2 2.8 2.4 2.0 1.6 1.2 0.8 0.4 0.0



SpinWorks 3: Nm331 Proton test





26.2092 —

61.9679 —

67.3894 —

76.7370 —

77.0548 —

77.3723 —

111.0949 —

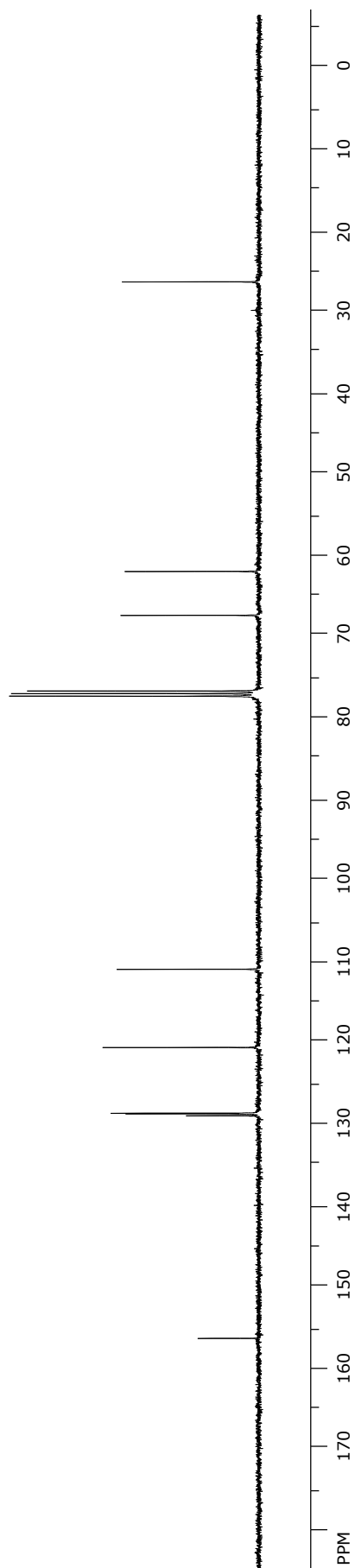
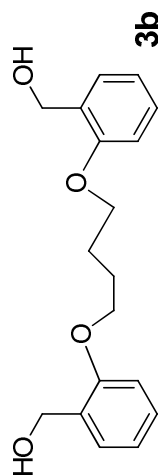
120.7609 —

128.8720 —

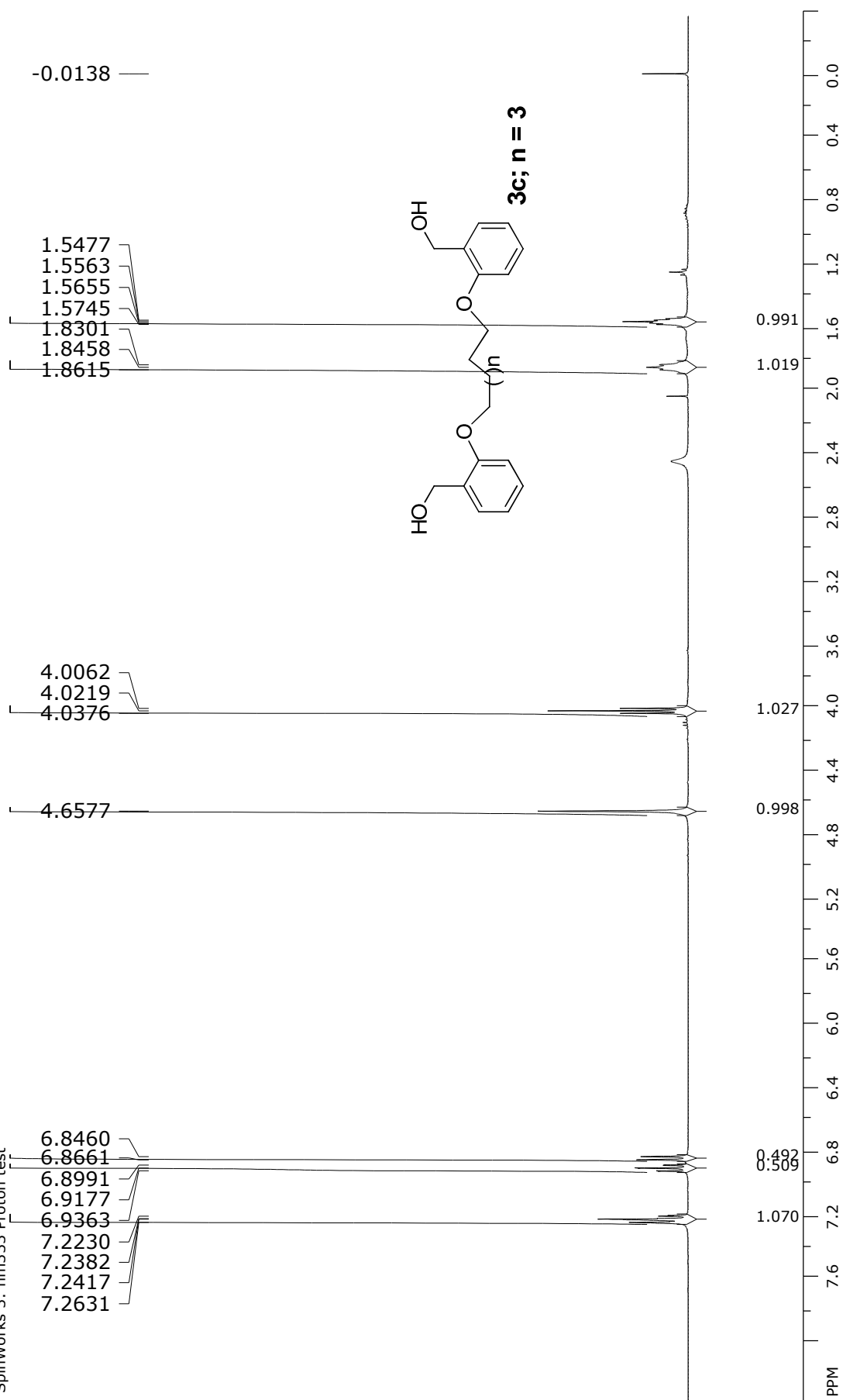
128.9486 —

129.1817 —

156.6758 —



SpinWorks 3: nm333 Proton test

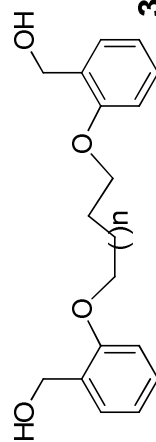


SpinWorks 3: nm333 Proton test

7.2230
7.2382
7.2417
7.2631

6.8460
6.8661
6.8991
6.9177
6.9363

4.6577



3c; n = 3

1.070

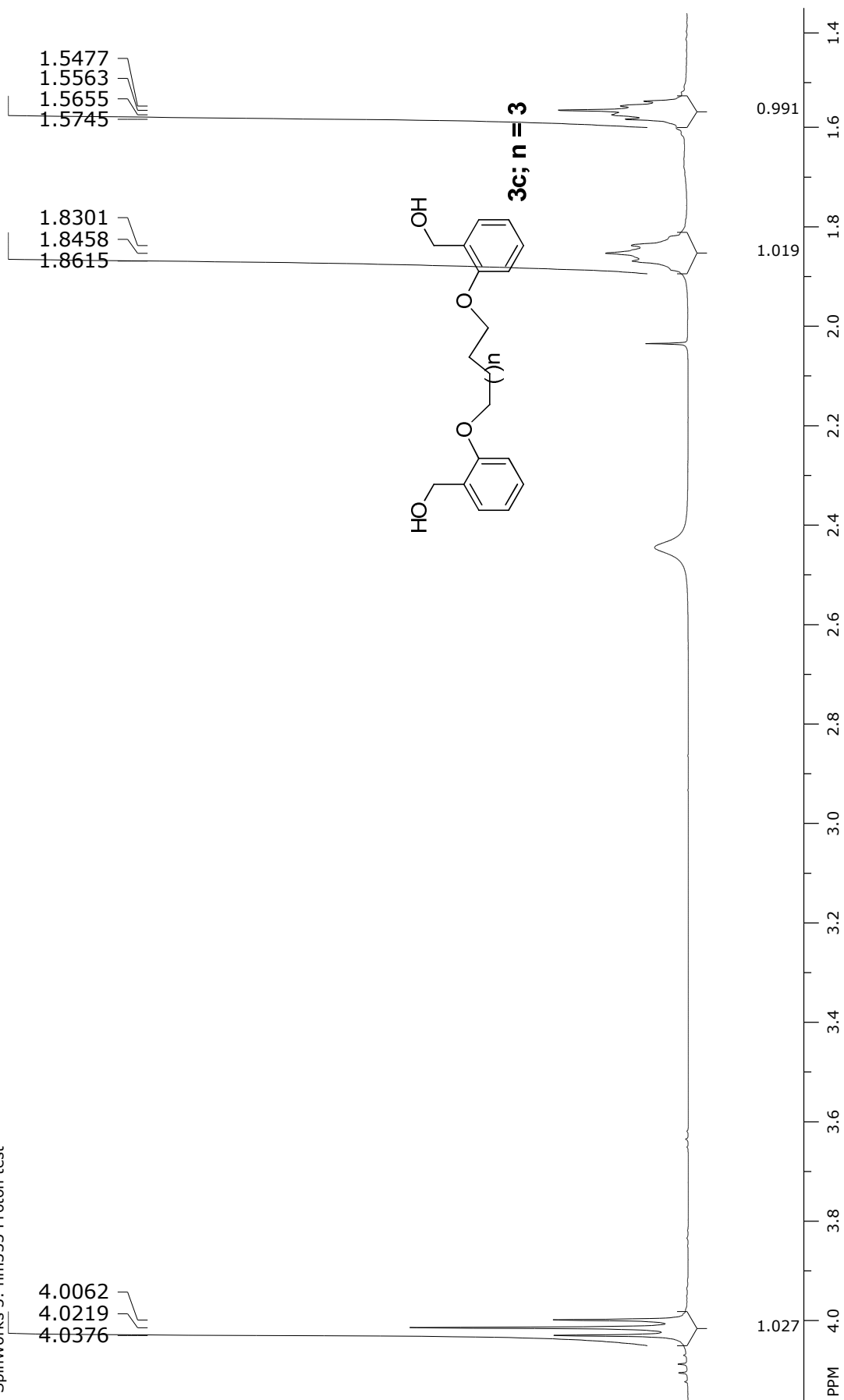
0.509

0.492

0.998

PPM

SpinWorks 3: nm333 Proton test



25.9751 —
29.2140 —

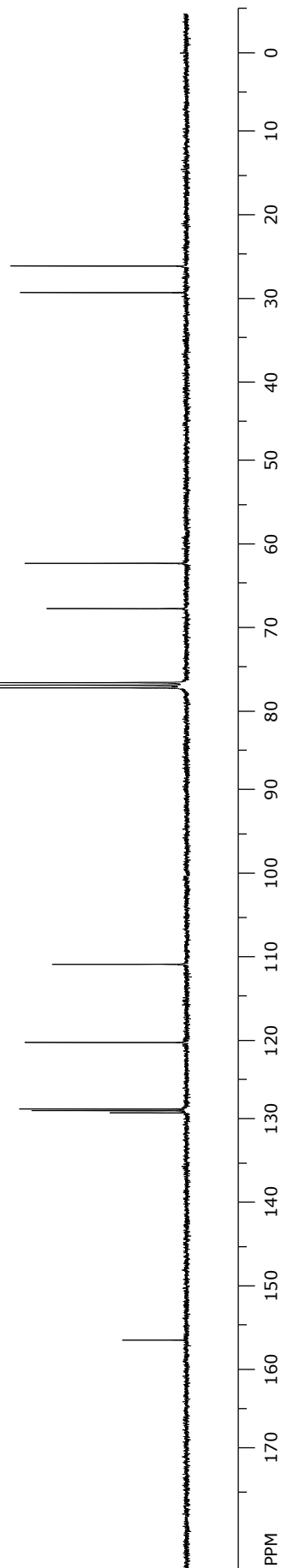
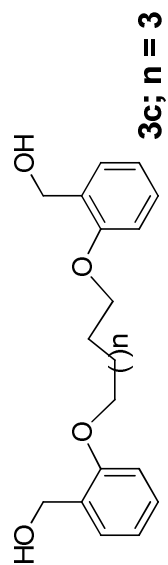
62.2064 —
67.7218 —
76.7380 —
77.0557 —
77.3730 —

111.0650 —

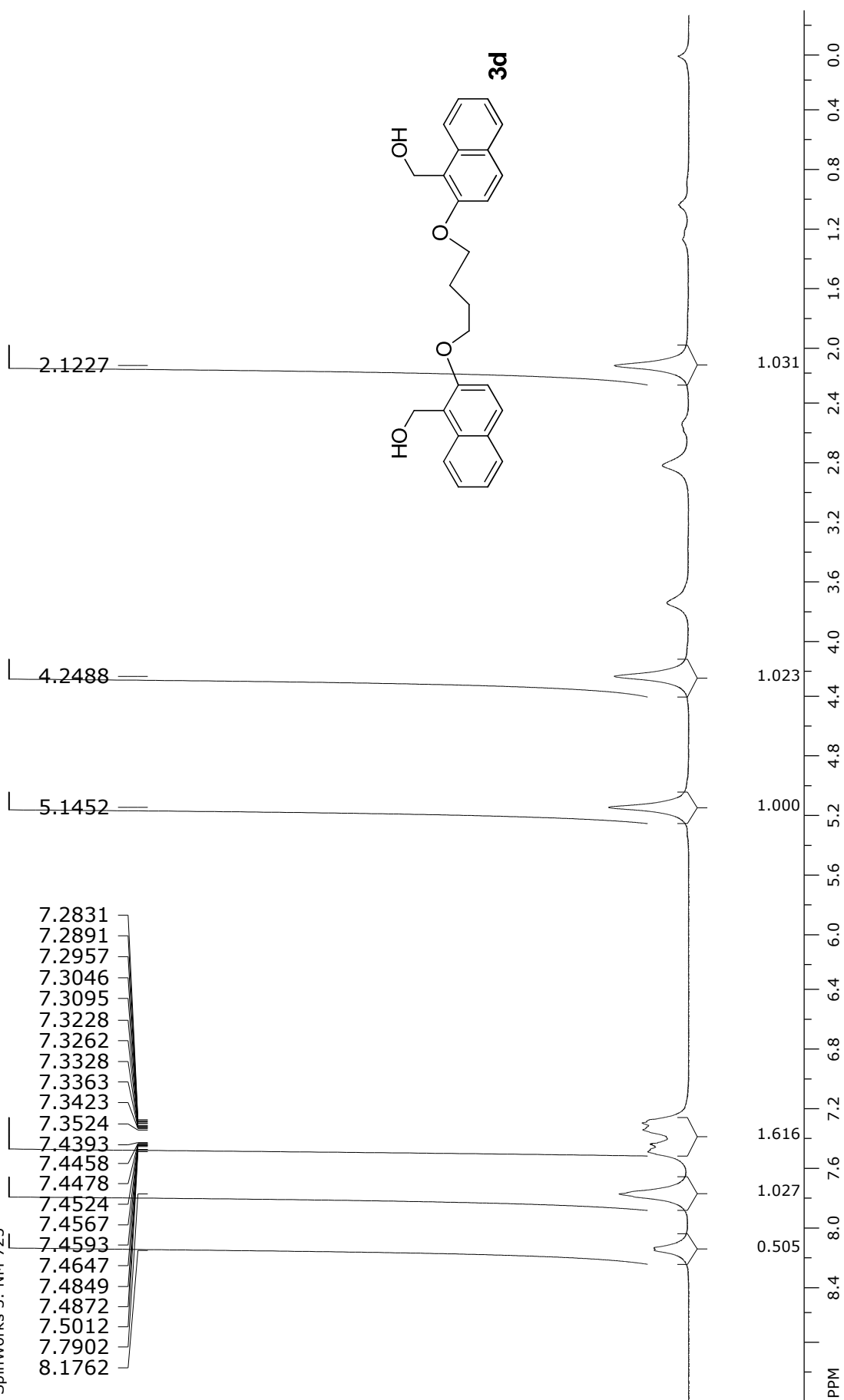
120.5963 —

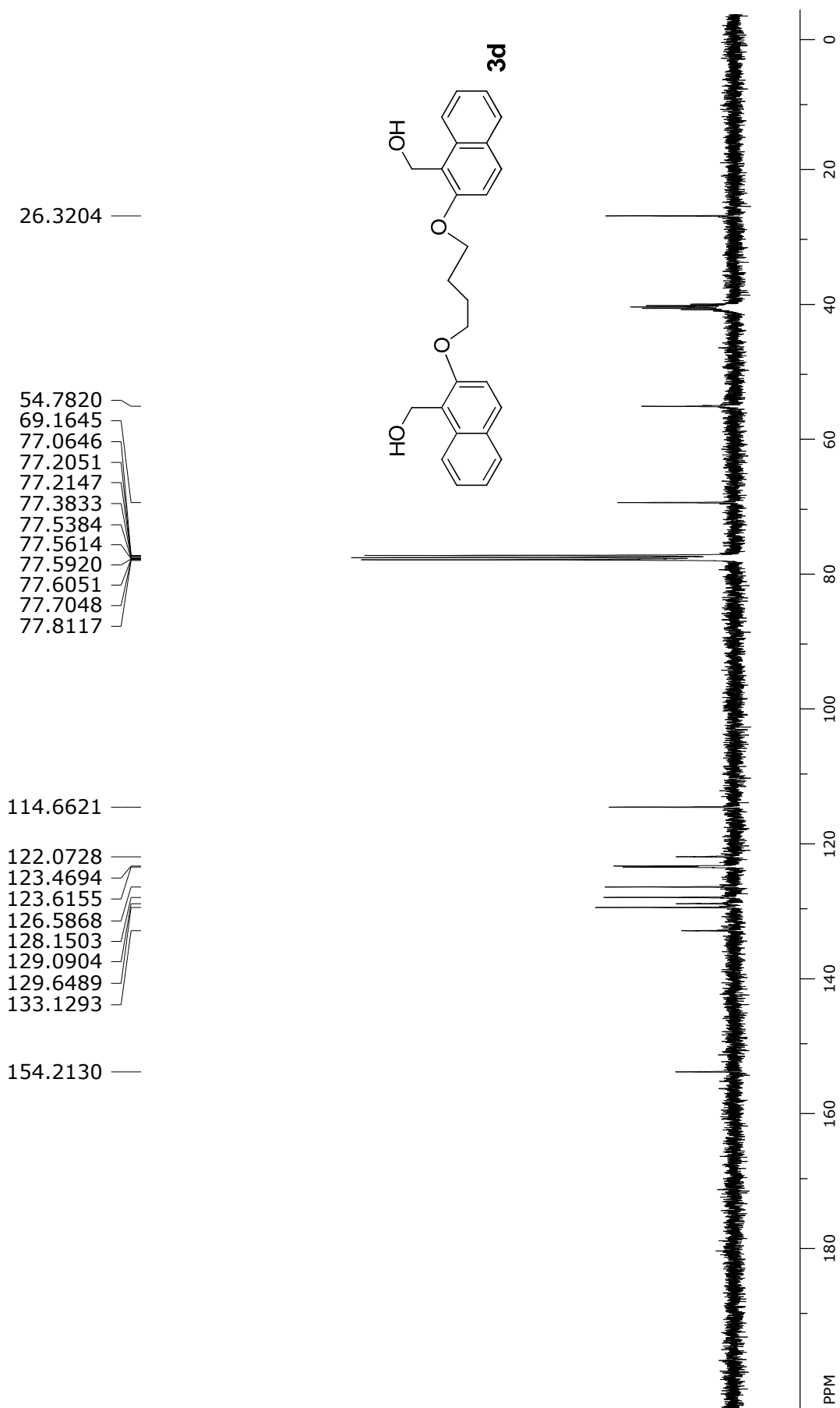
128.6995 —
128.8988 —
129.1664 —

156.8530 —



SpinWorks 3: NM-725





SpinWorks 3: NM-725

133.1293 —

129.0904 —

129.6489 —

128.1503 —

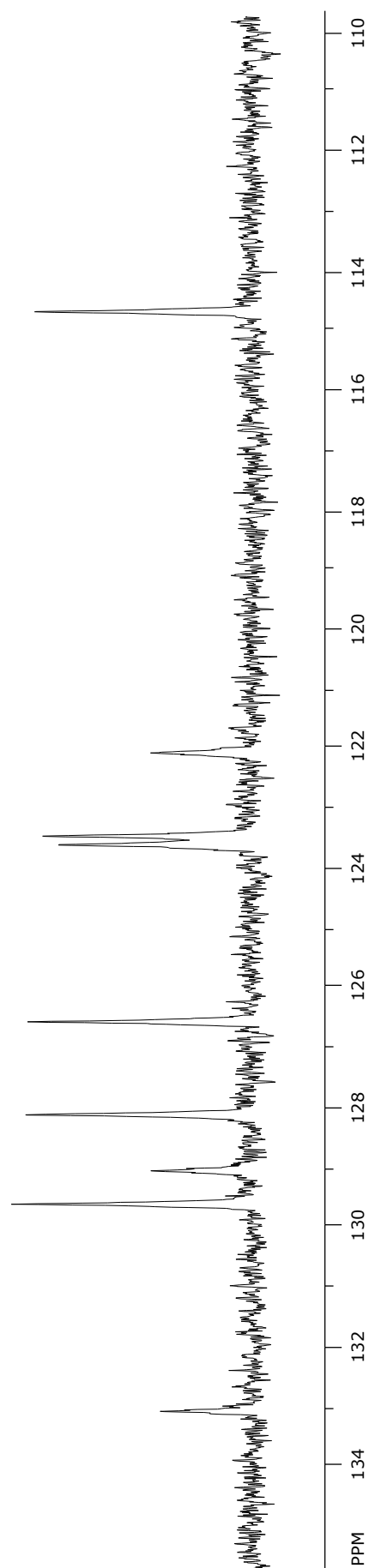
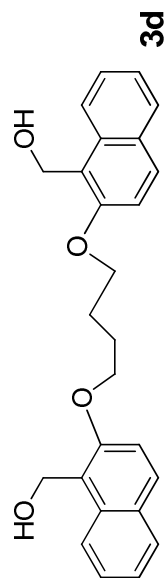
126.5868 —

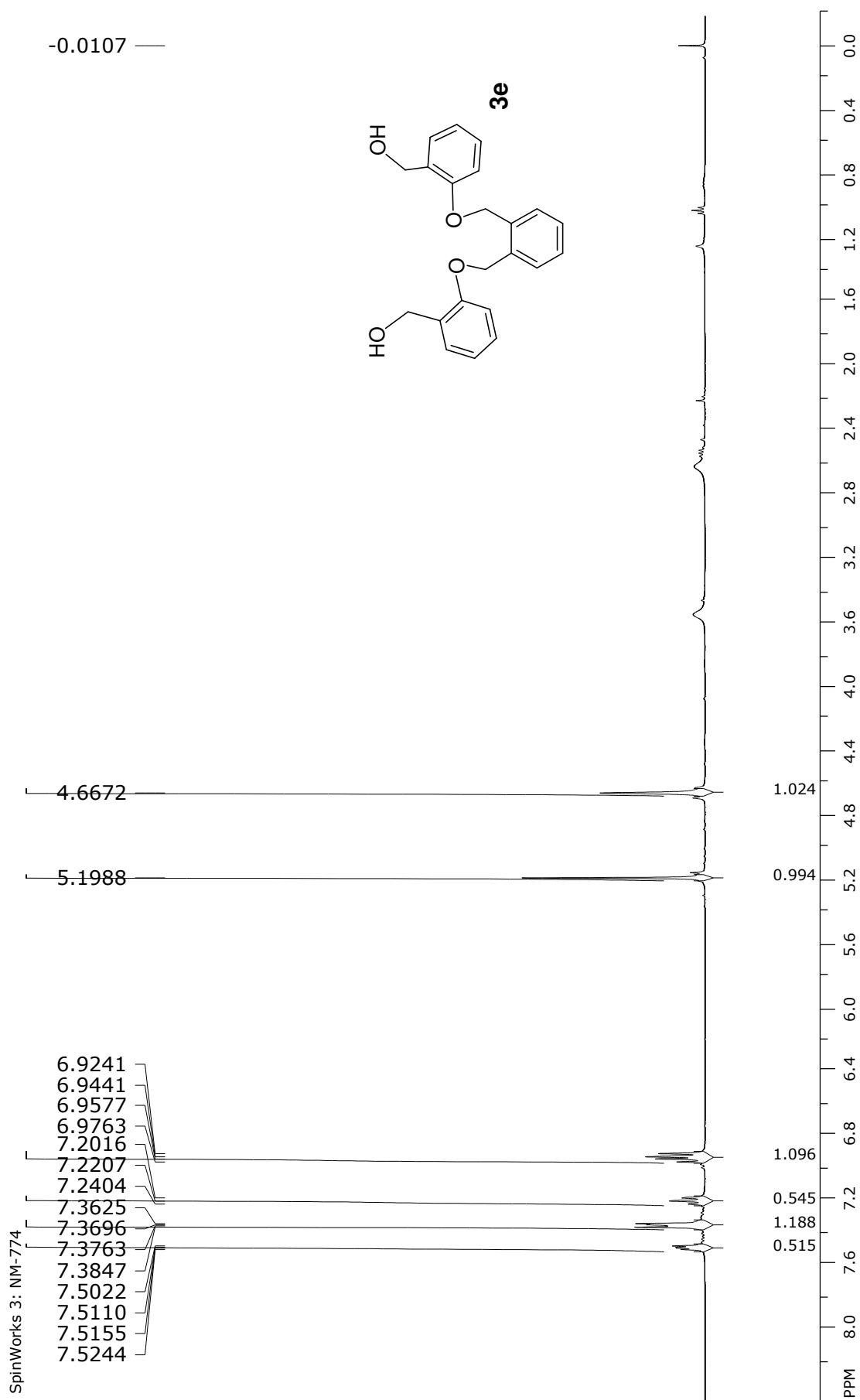
123.4694 —

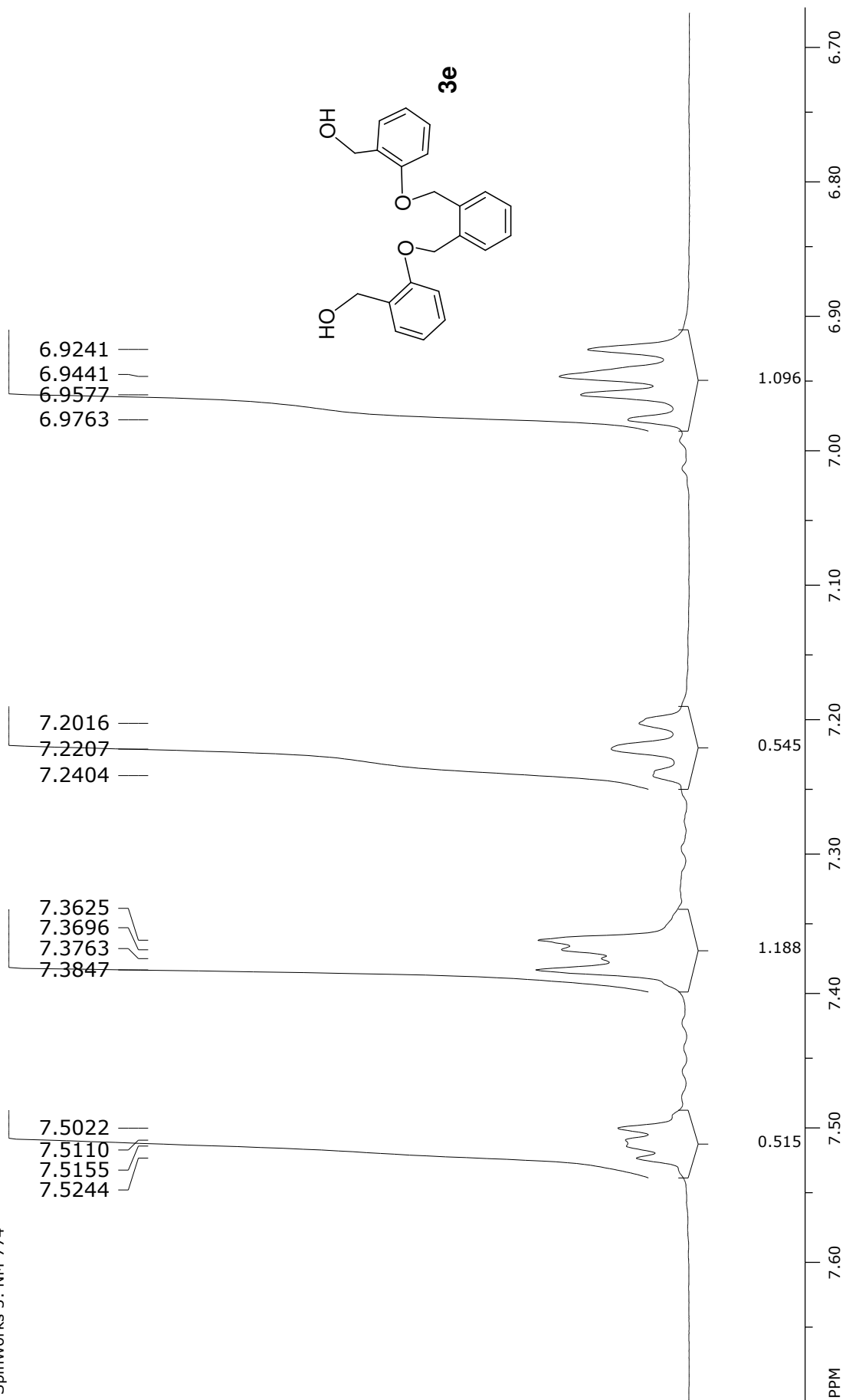
123.6155 —

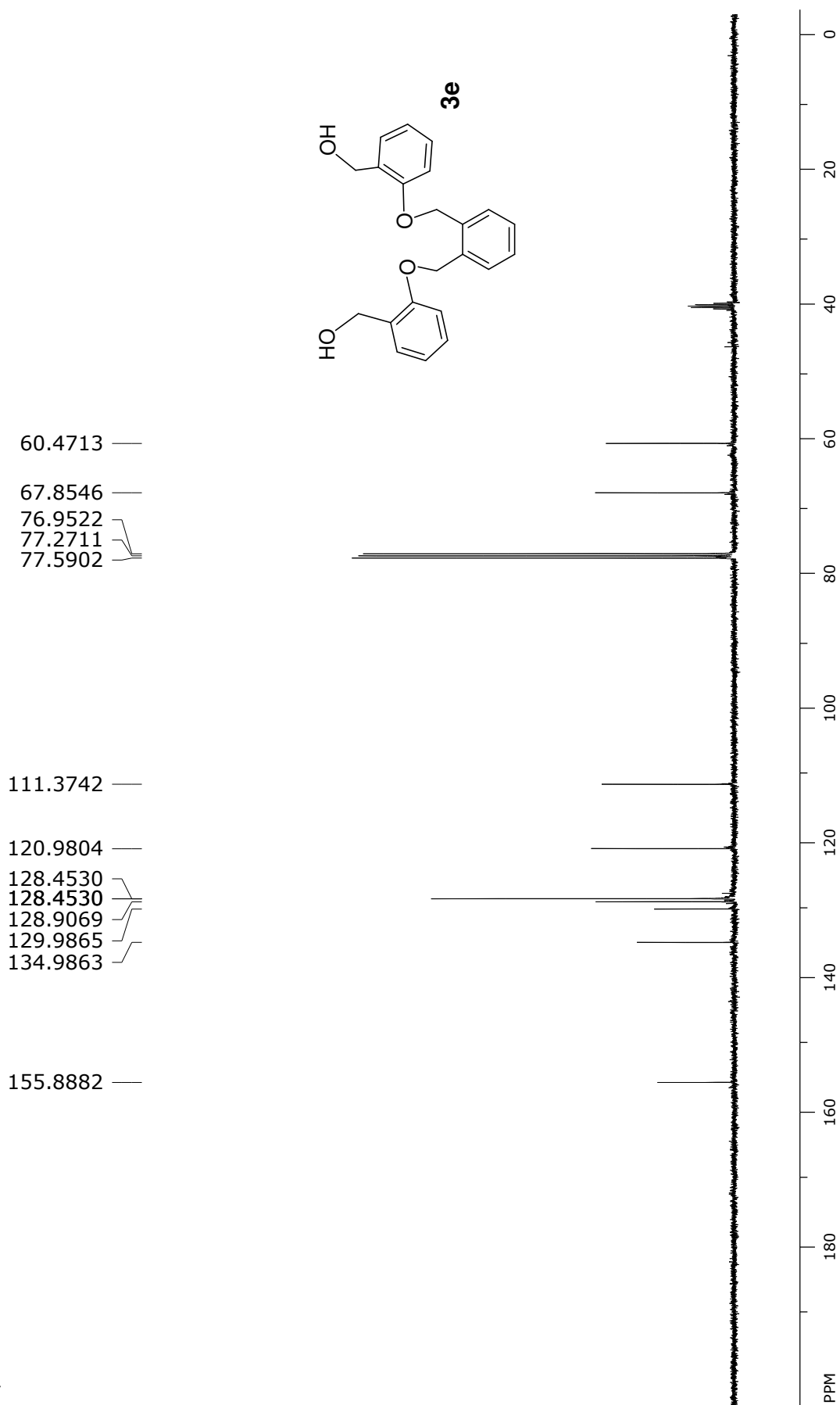
122.0728 —

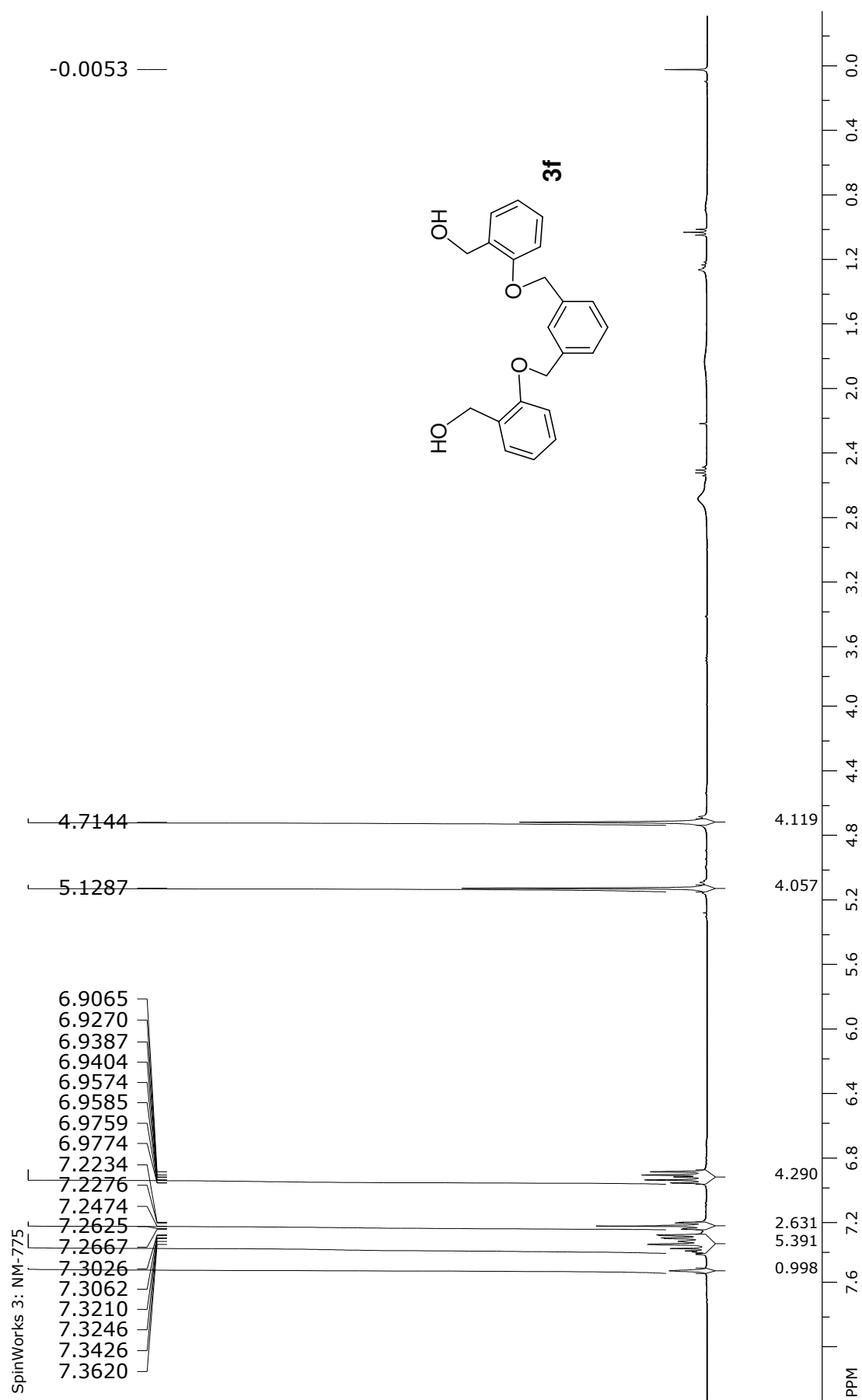
114.6621 —

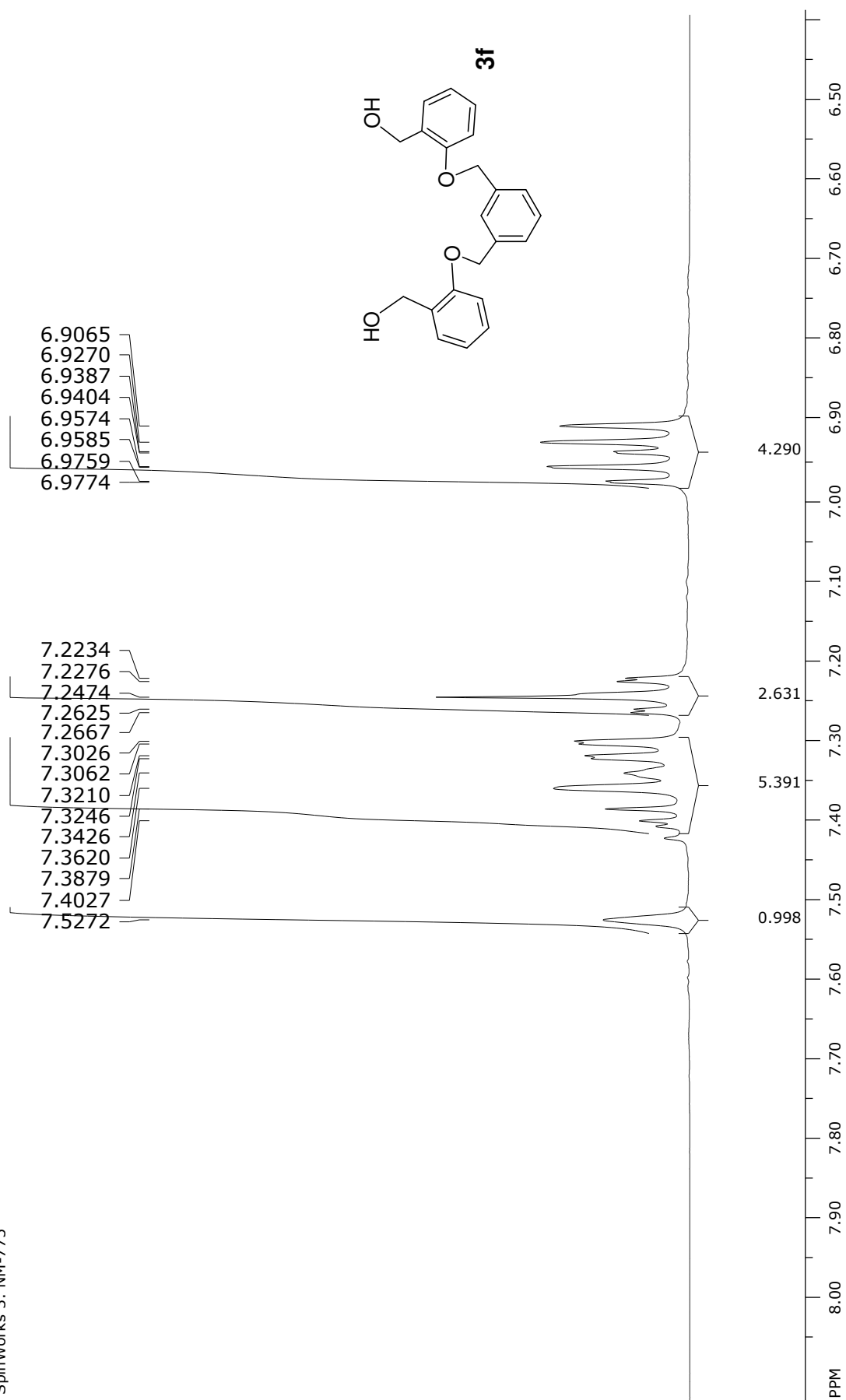


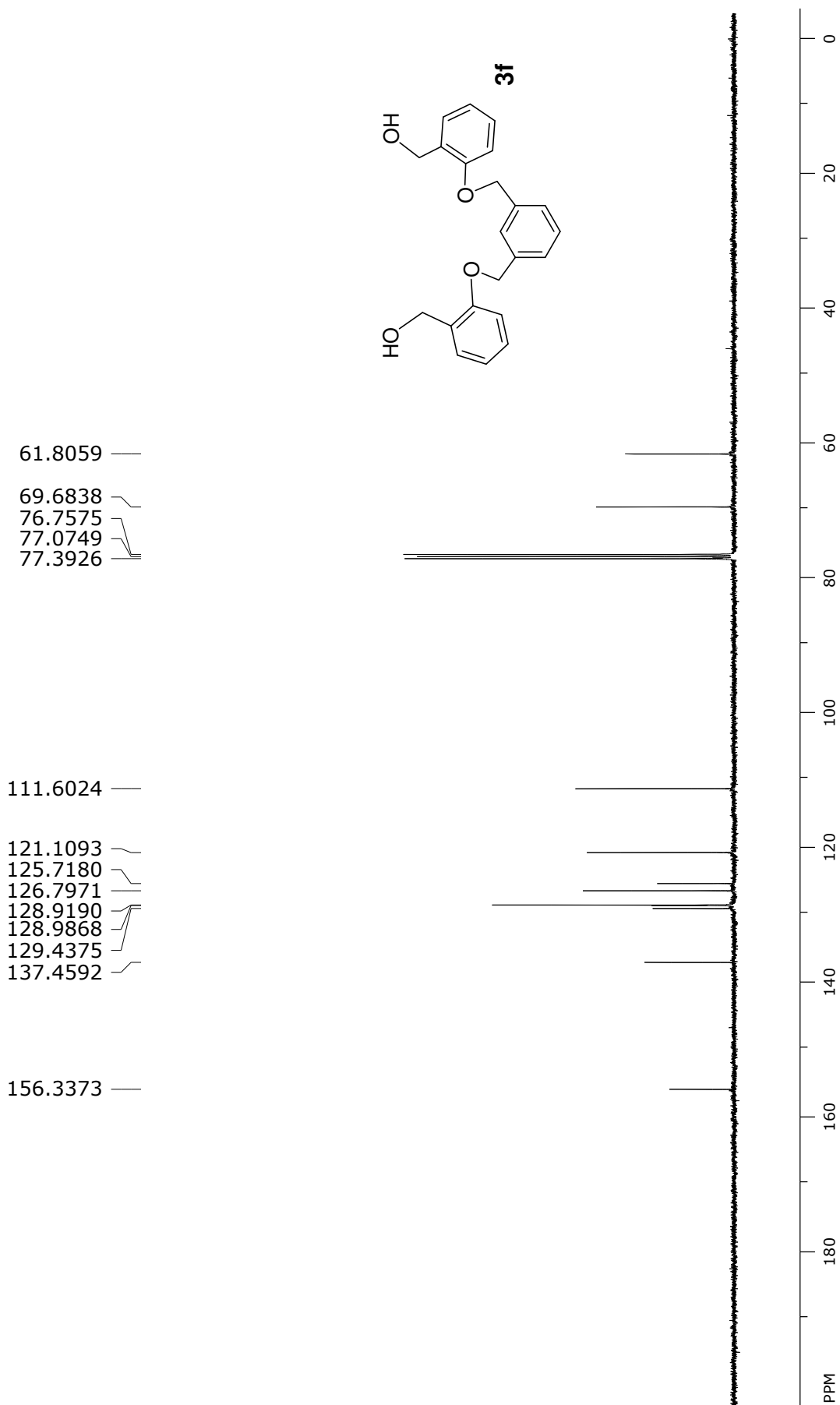












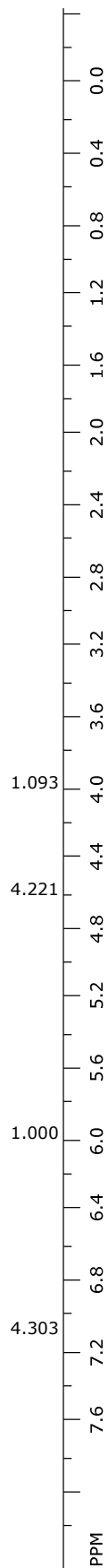
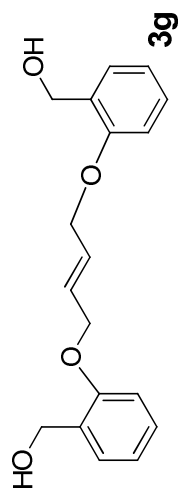
SpinWorks 3: NM1108 Proton test

6.8218 —
6.9177 —
7.1827 —
7.3729 —

6.0567

4.5836 —
4.6617 —

3.9845

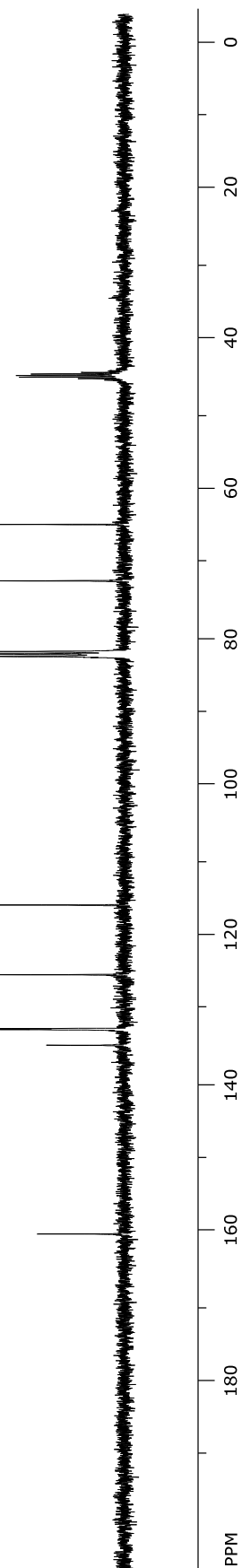
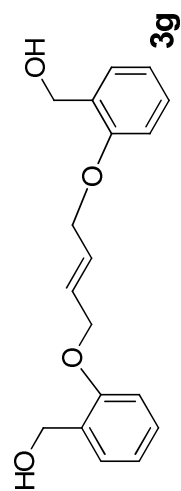


SpinWorks 3: Nm1108 Carbon test

64.8409 —
72.4354 —
81.9797 —
82.3009 —
82.6219 —

116.1211 —
125.5107 —
132.7753 —
132.8446 —
132.9079 —
135.0282 —

160.4517 —

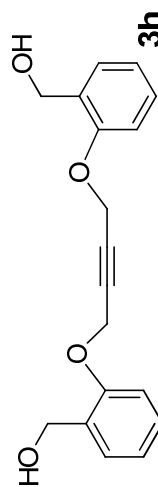


SpinWorks 3: NM1109 Proton test

-0.0985

6.8018
6.8223
6.8733
6.8919
6.9105
7.1092
7.1288
7.1484
7.2313
7.2497

4.5706
4.6653



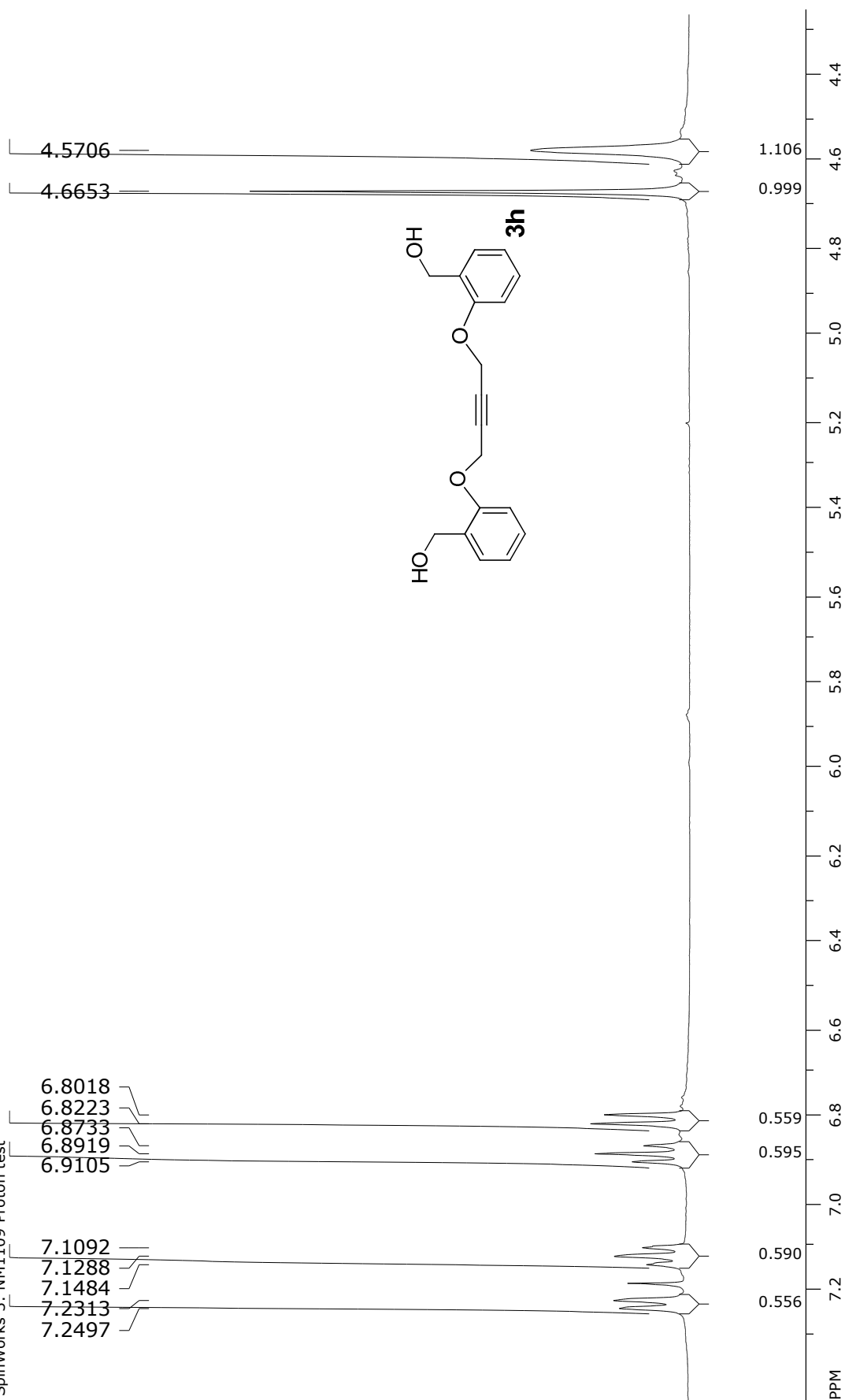
1.106
0.999

0.559
0.595

0.590
0.556

PPM

-0.4 0.0 0.4 0.8 1.2 1.6 2.0 2.4 2.8 3.2 3.6 4.0 4.4 4.8 5.2 5.6 6.0 6.4 6.8 7.2 7.6



56.2099 —

61.1981 —

76.7936 —

77.1131 —

77.4299 —

82.4333 —

111.9941 —

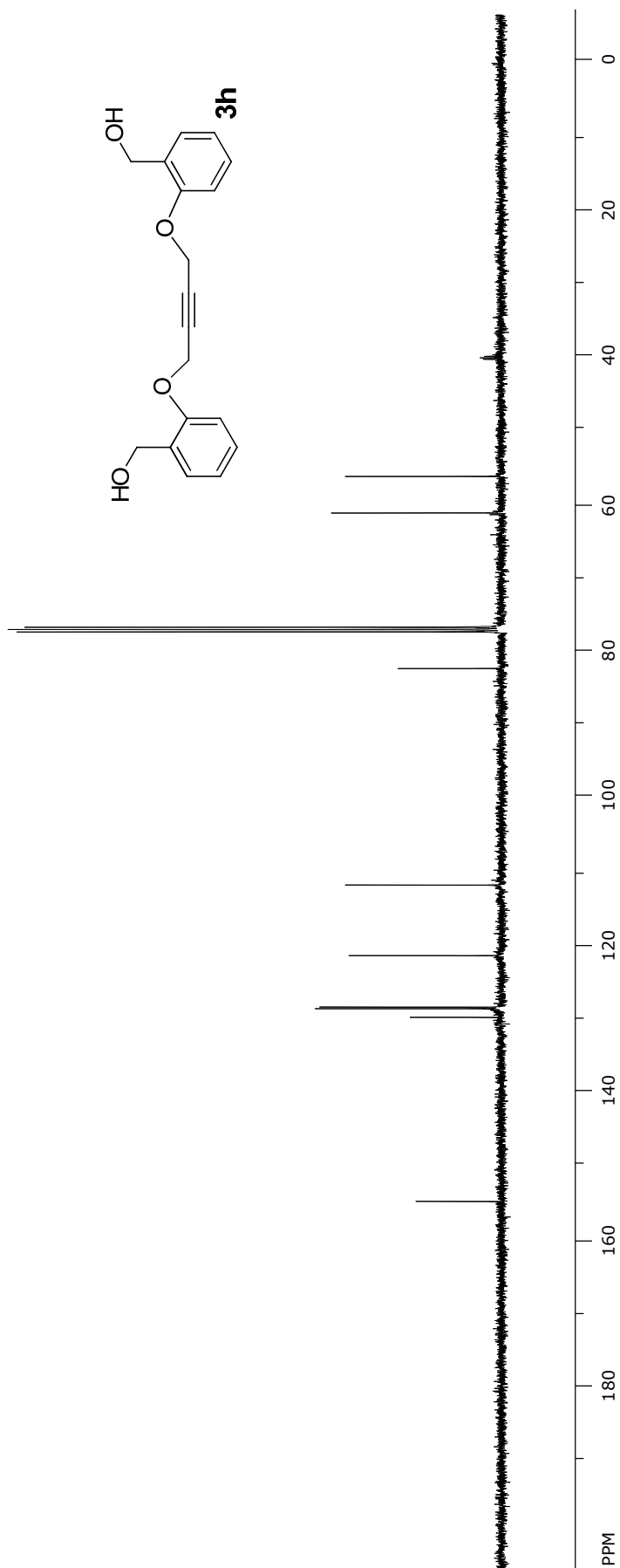
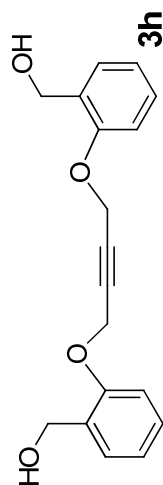
121.6257 —

128.6606 —

128.8626 —

130.0540 —

155.1897 —

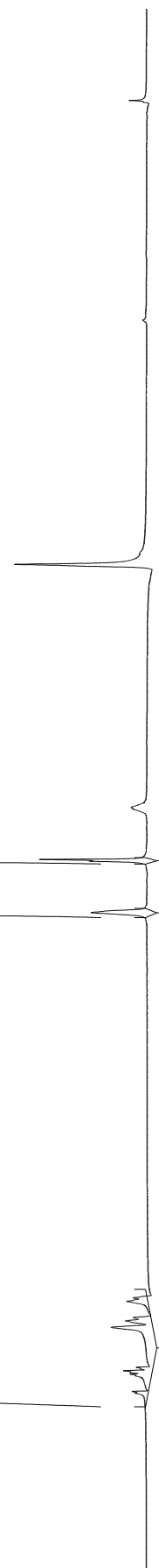
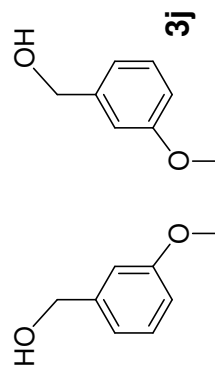


SpinWorks 3: NM 378

6.7488
6.7621
6.7692
6.8617
6.8802
6.9178
7.1454
7.1653
7.1743
7.1843
7.1937
7.2832
7.2847
7.2932

4.2451

4.5487



0.968

0.999

2.479

PPM

-0.4

0.0

0.4

0.8

1.2

1.6

2.0

2.4

2.8

3.2

3.6

4.0

4.4

4.8

5.2

5.6

6.0

6.4

6.8

7.2

7.6

39.7346
39.9417
40.1513

64.3496
66.4167

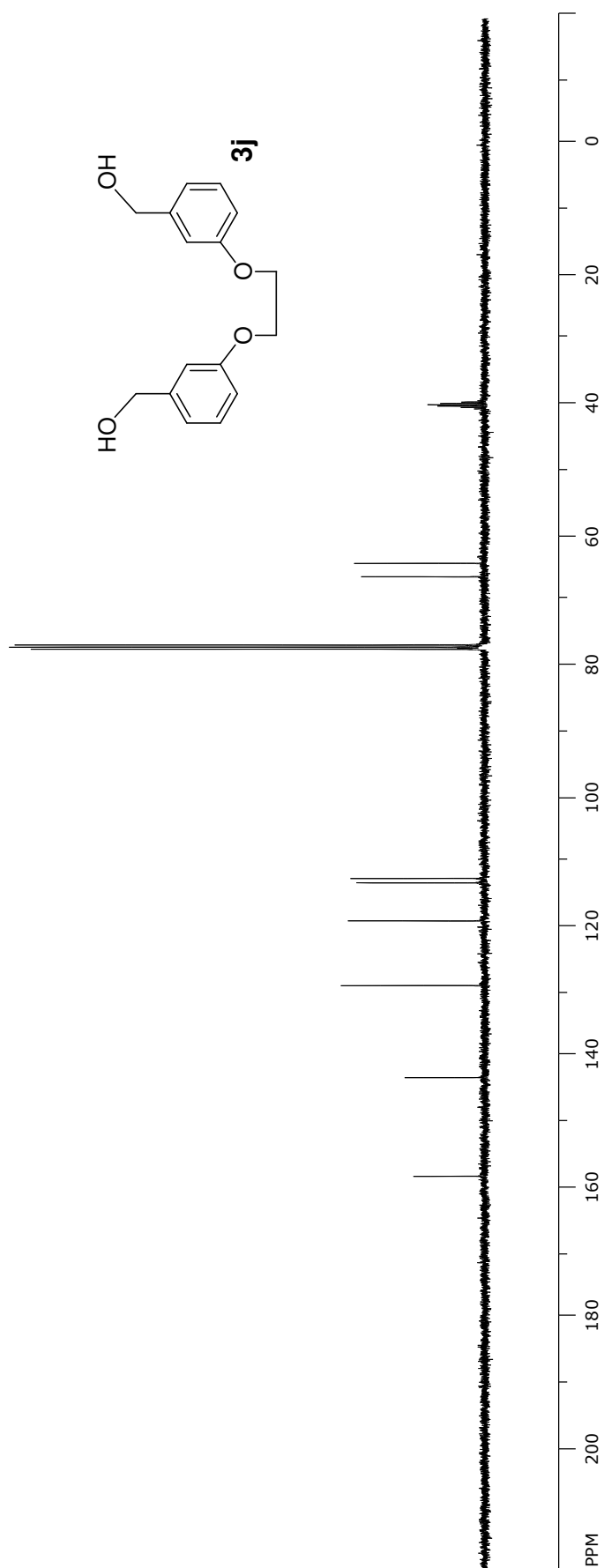
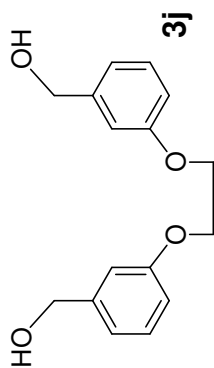
76.9189
77.2382
77.5575

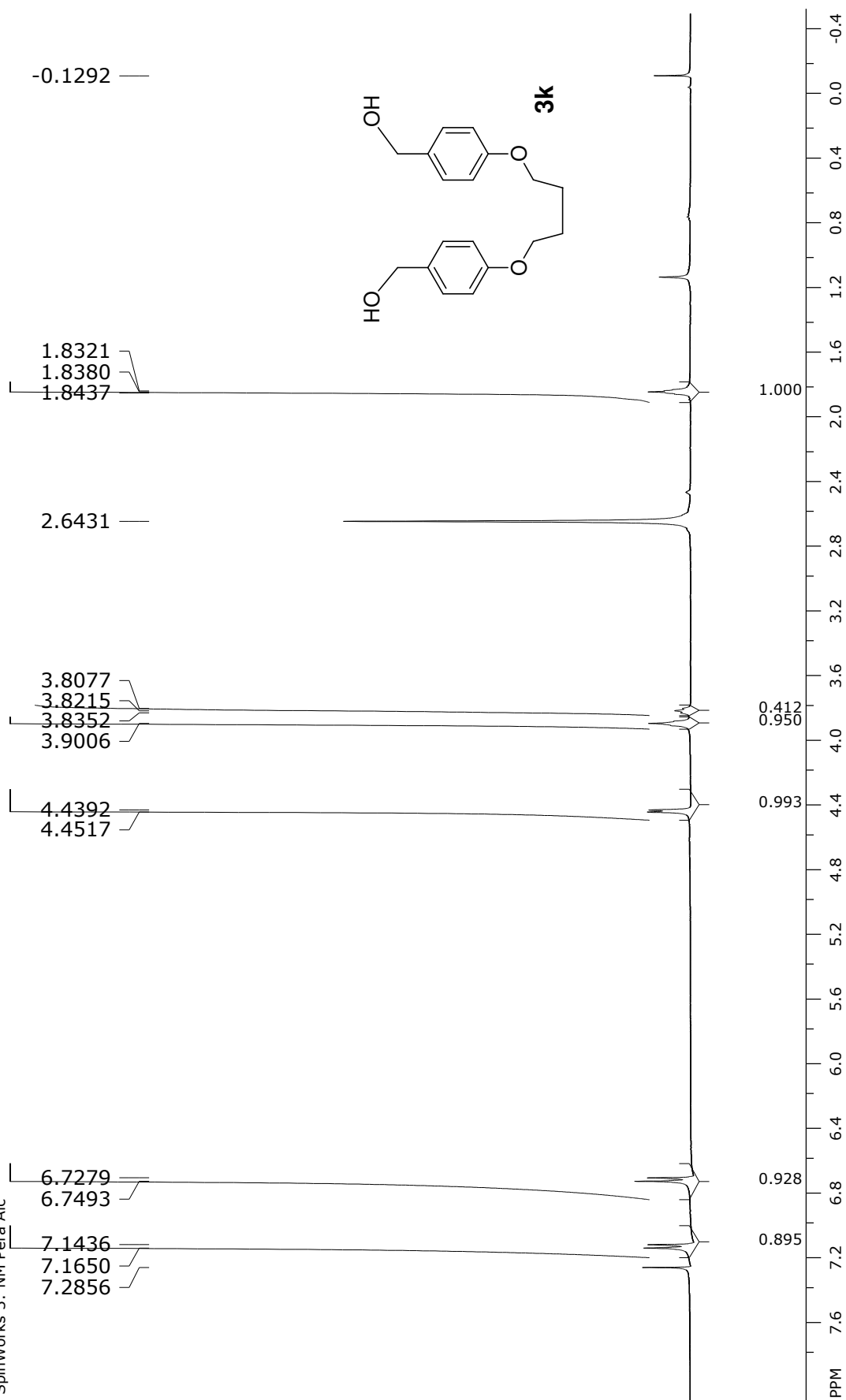
112.8427
113.5303
119.3757

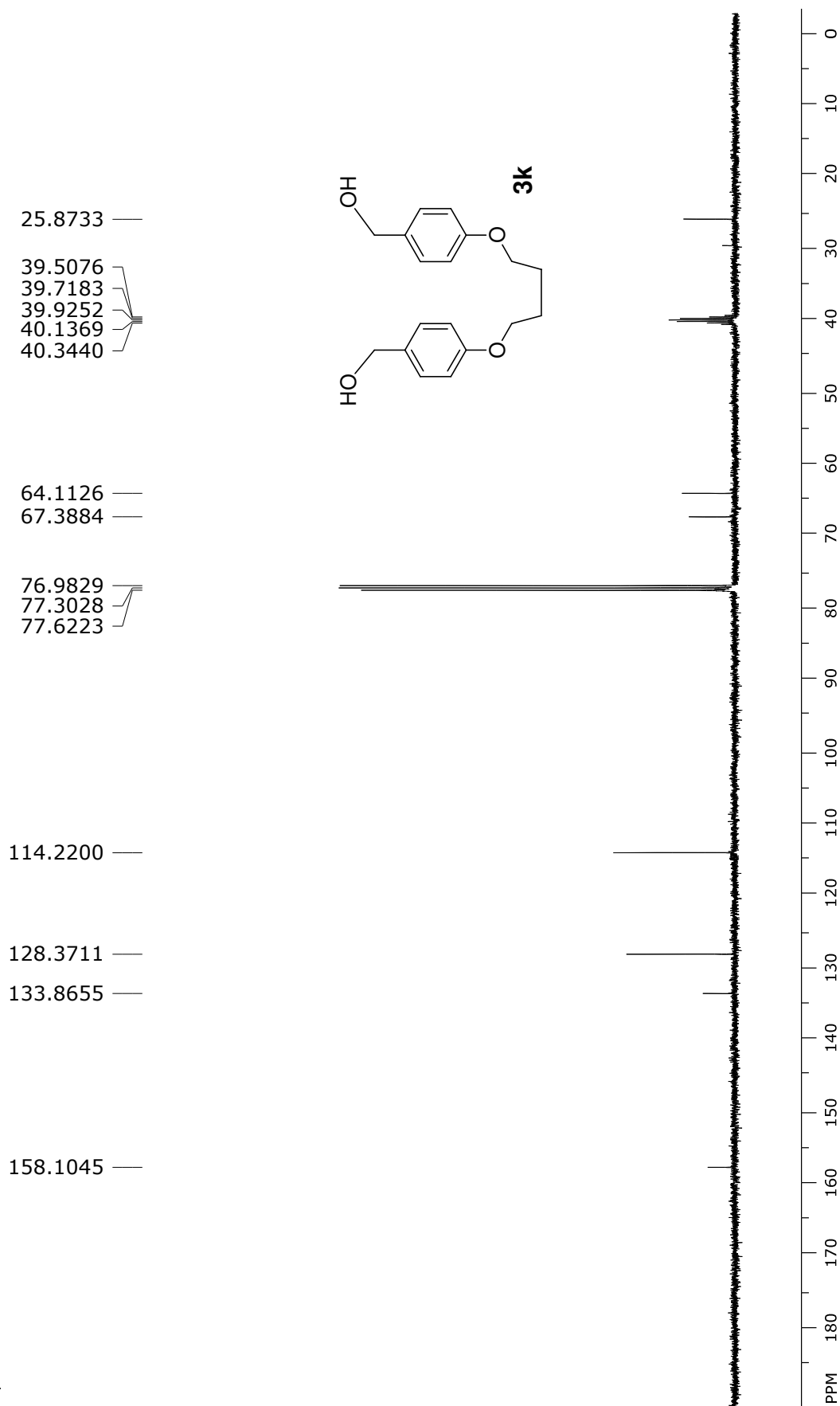
129.3367

143.5040

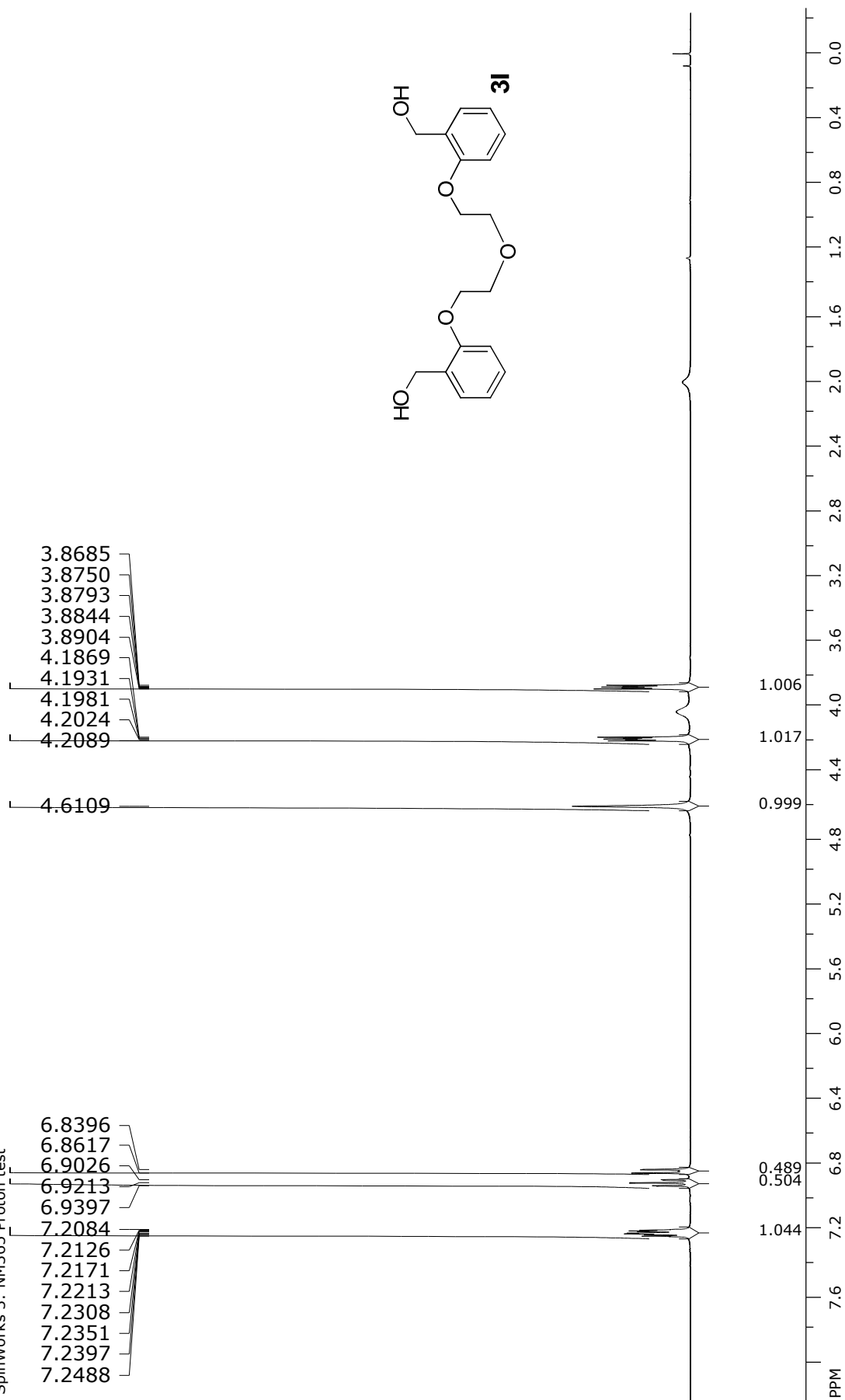
158.6813

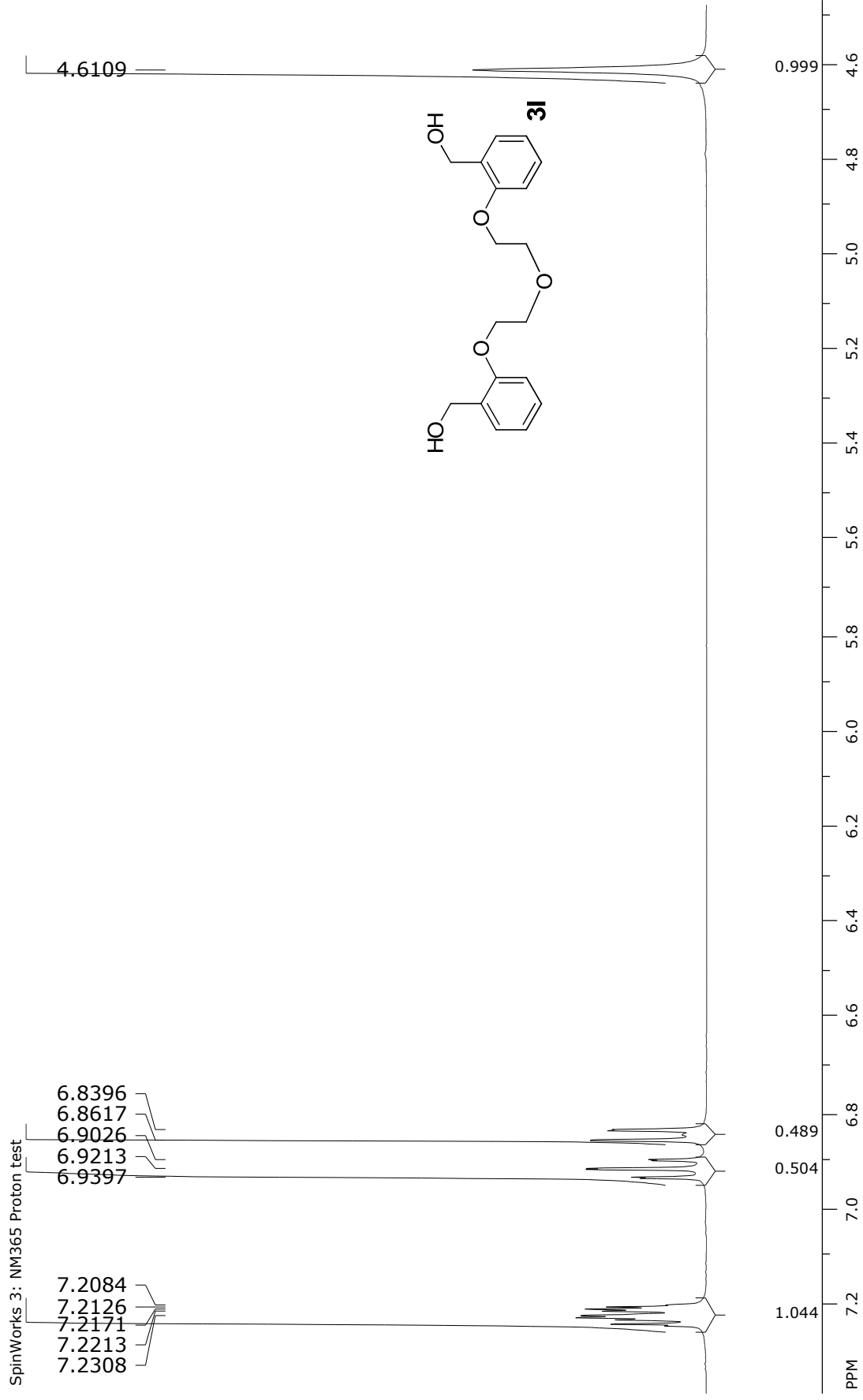




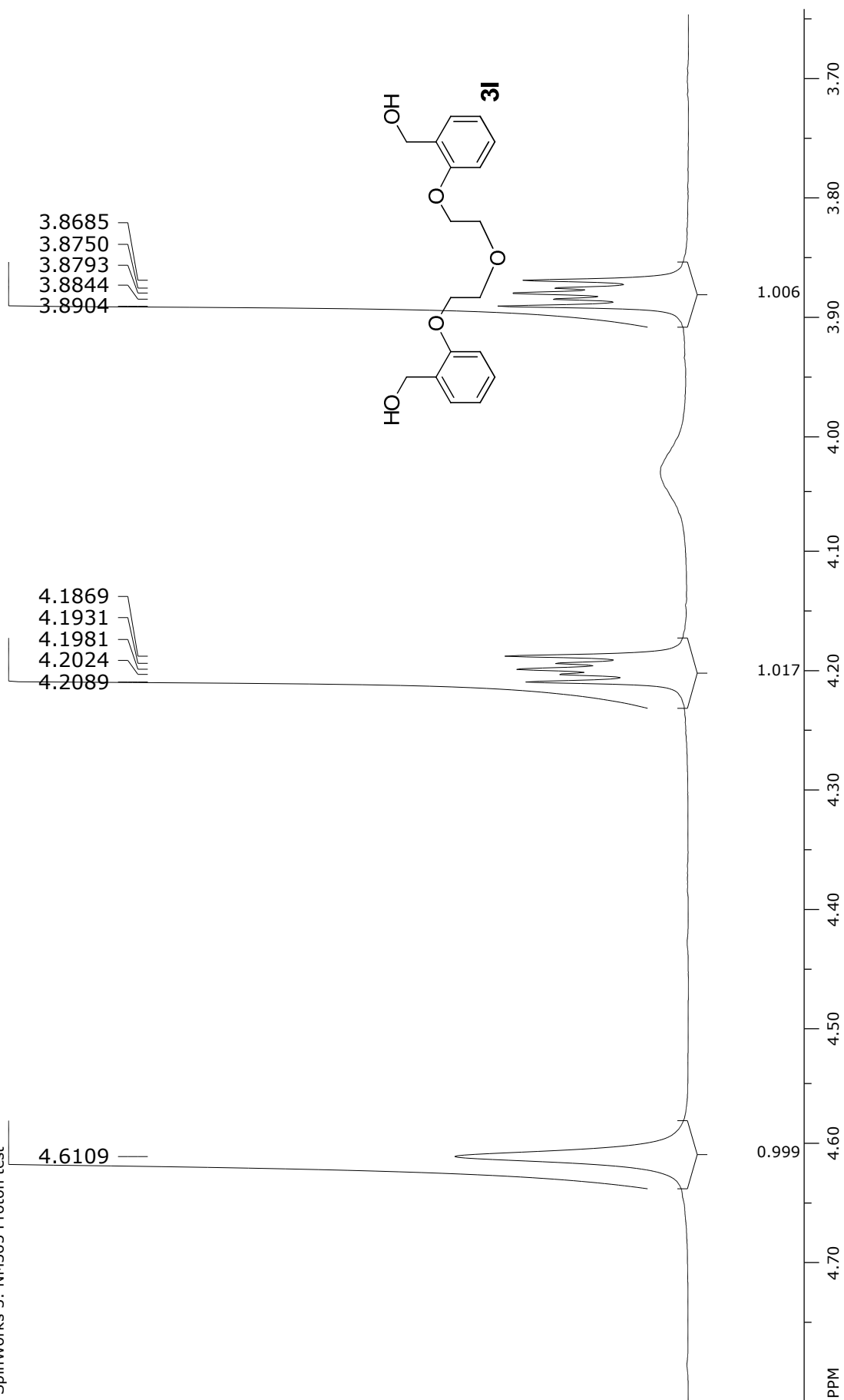


SpinWorks 3: NM365 Proton test





SpinWorks 3: NM365 Proton test



SpinWorks 3: NM365 Carbon test

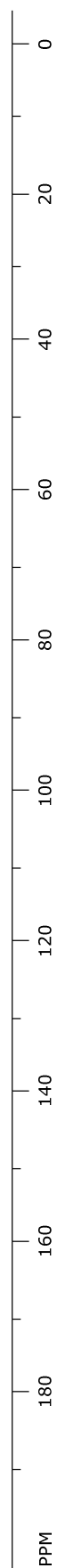
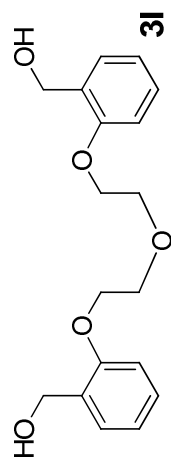
61.8169 —
67.7689 —
69.8388 —
76.7763 —
77.0946 —
77.4121 —

112.2186 —

121.2908 —

129.0243 —
129.7061 —
130.1605 —

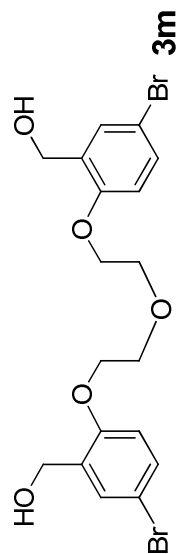
156.9761 —



SpinWorks 3: NM1110 Proton test

7.1211
7.1409
7.1608
7.2689
7.2869

6.5447
6.5683
6.5904
4.4267
4.1027
3.9833
3.7207



0.760
1.134
0.766

0.367
0.822

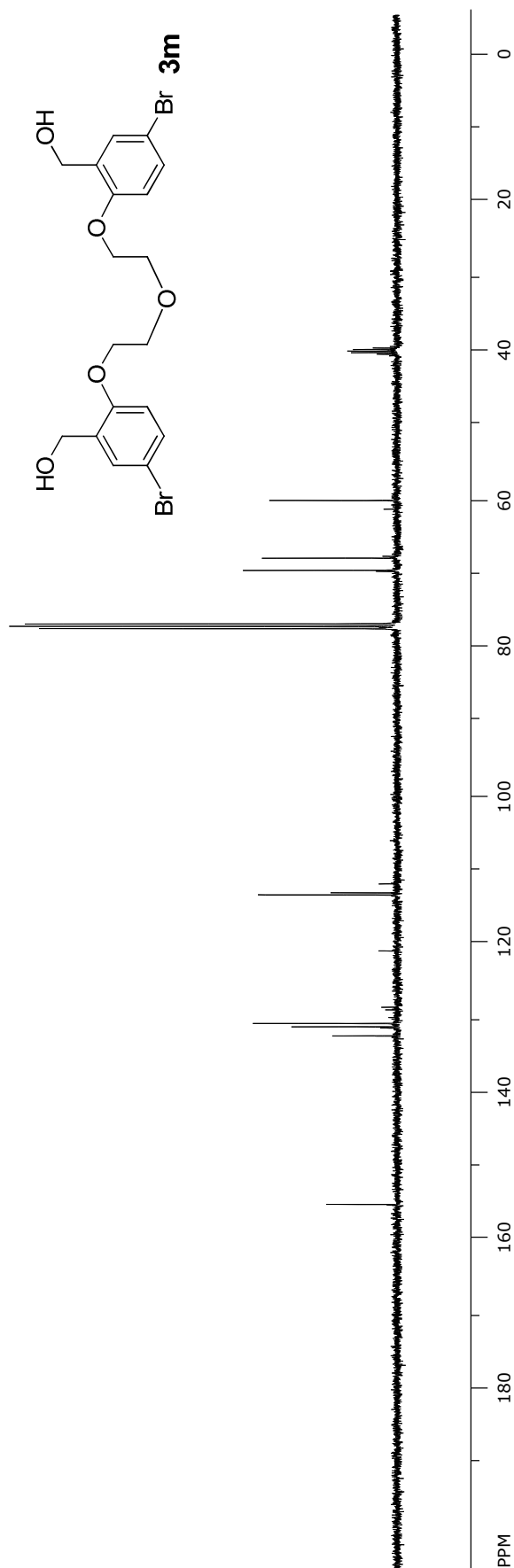
PPM -0.4 0.0 0.4 0.8 1.2 1.6 2.0 2.4 2.8 3.2 3.6 4.0 4.4 4.8 5.2 5.6 6.0 6.4 6.8 7.2 7.6 8.0

60.1923 —
67.9988 —
69.6436 —
76.8955 —
77.2153 —
77.5337 —

113.2873 —
113.5679 —

130.9556 —
131.4131 —
132.6409 —

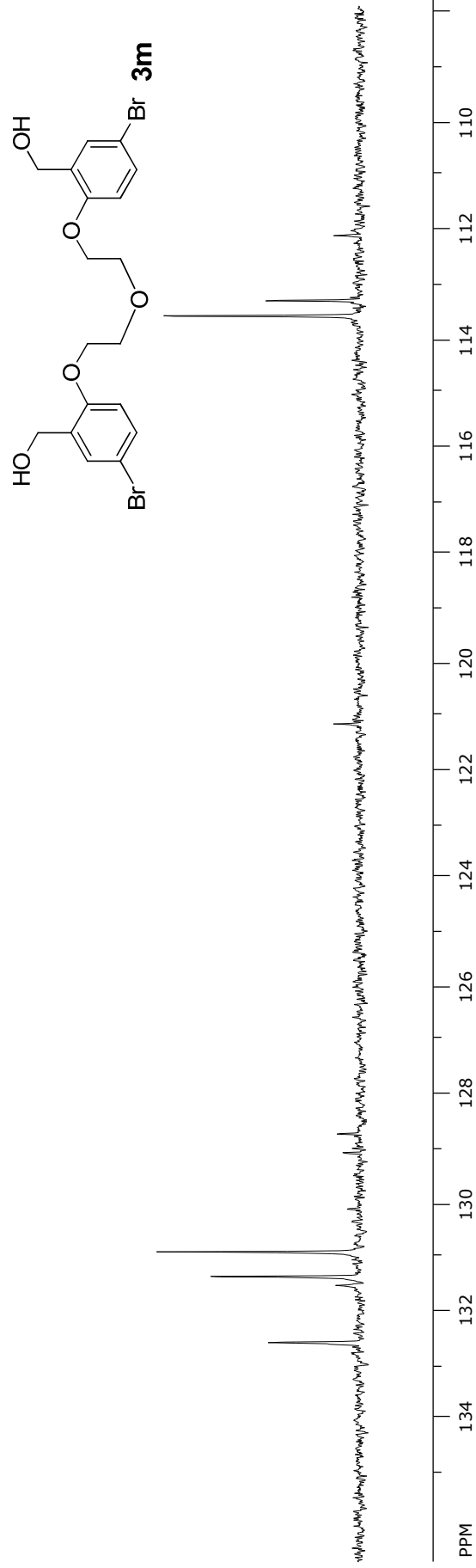
155.4523 —



SpinWorks 3: NM1110 Carbon test

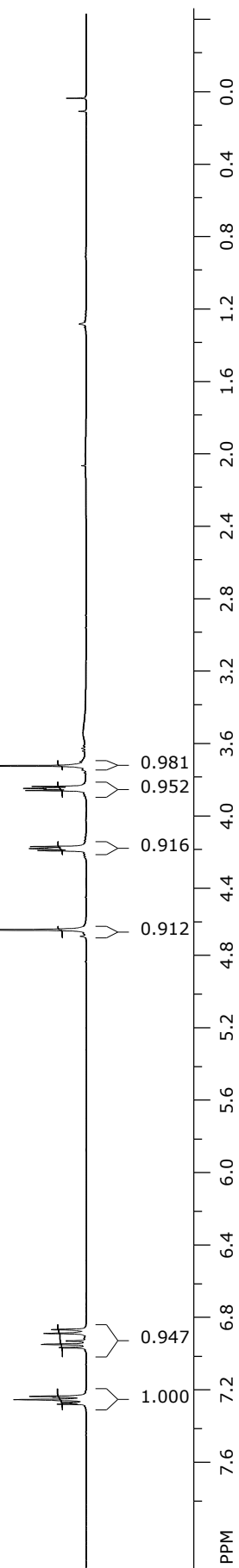
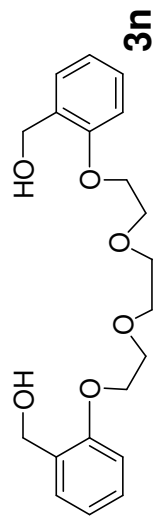
130.9556 —
131.4131 —
132.6409 —

113.2873 —
113.5679 —



6.9347
~~6.9504~~
~~6.9504~~
~~6.9517~~
7.2415
7.2461
7.2598
7.2642
7.2762
7.2852

3.7360
3.8507
3.8587
3.8618
3.8661
3.8733
4.1847
4.1919
4.1961
4.2073
4.6478



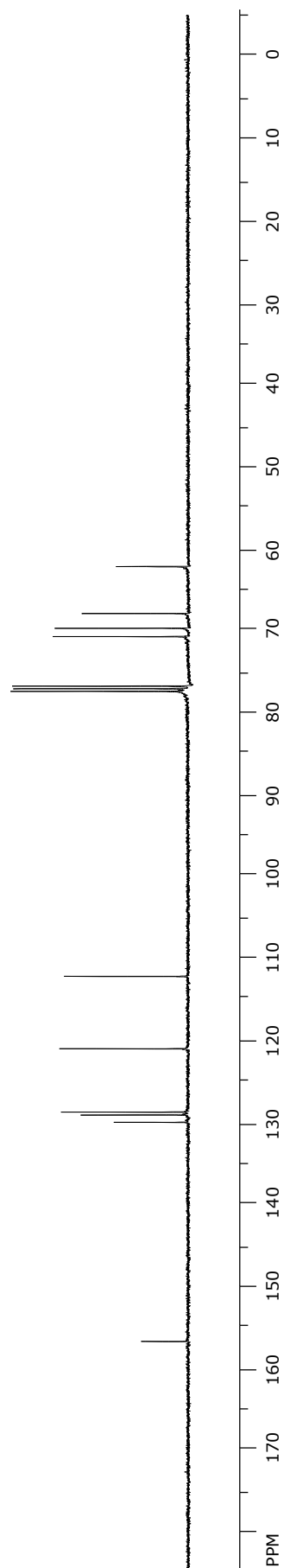
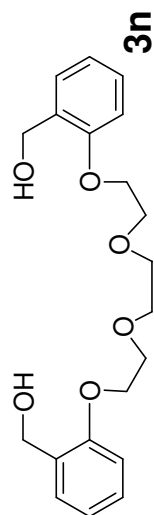
62.1047 —
67.8591 —
69.6647 —
70.6911 —
76.7581 —
77.0761 —
77.3936 —

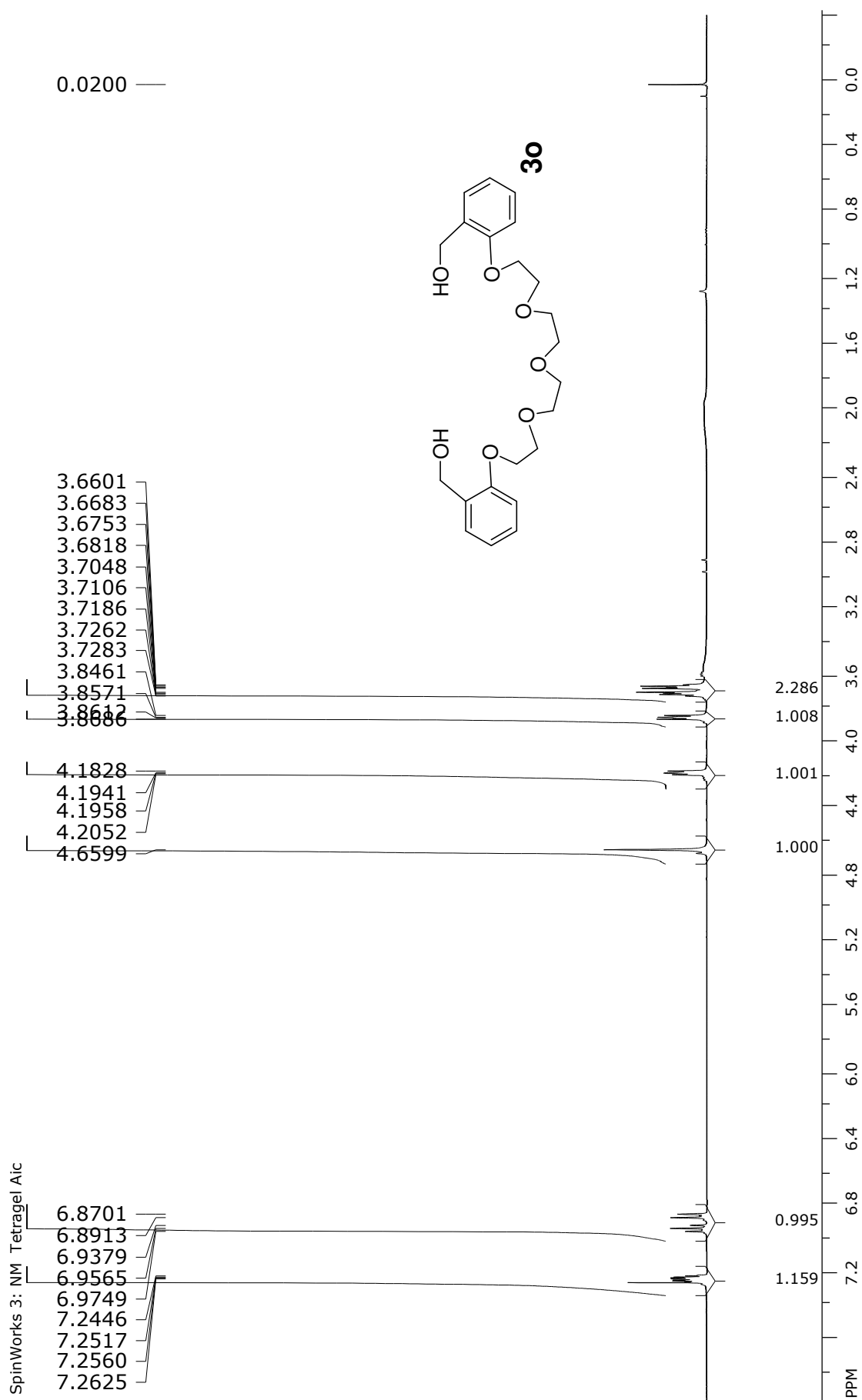
112.3112 —

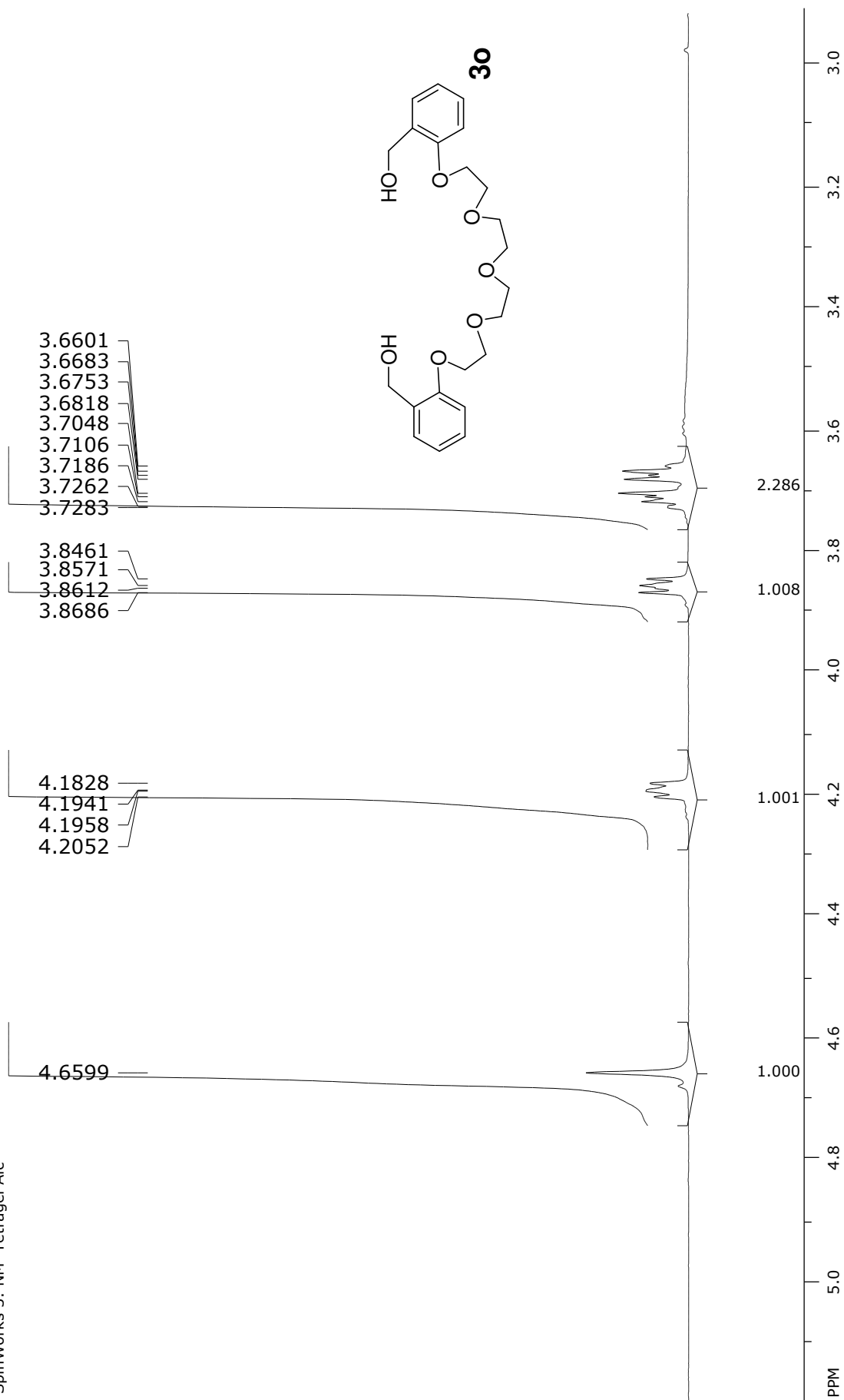
121.1778 —

128.9350 —
129.2936 —
130.1885 —

157.0303 —







61.9860
67.8106
69.5614
70.5612
70.6477

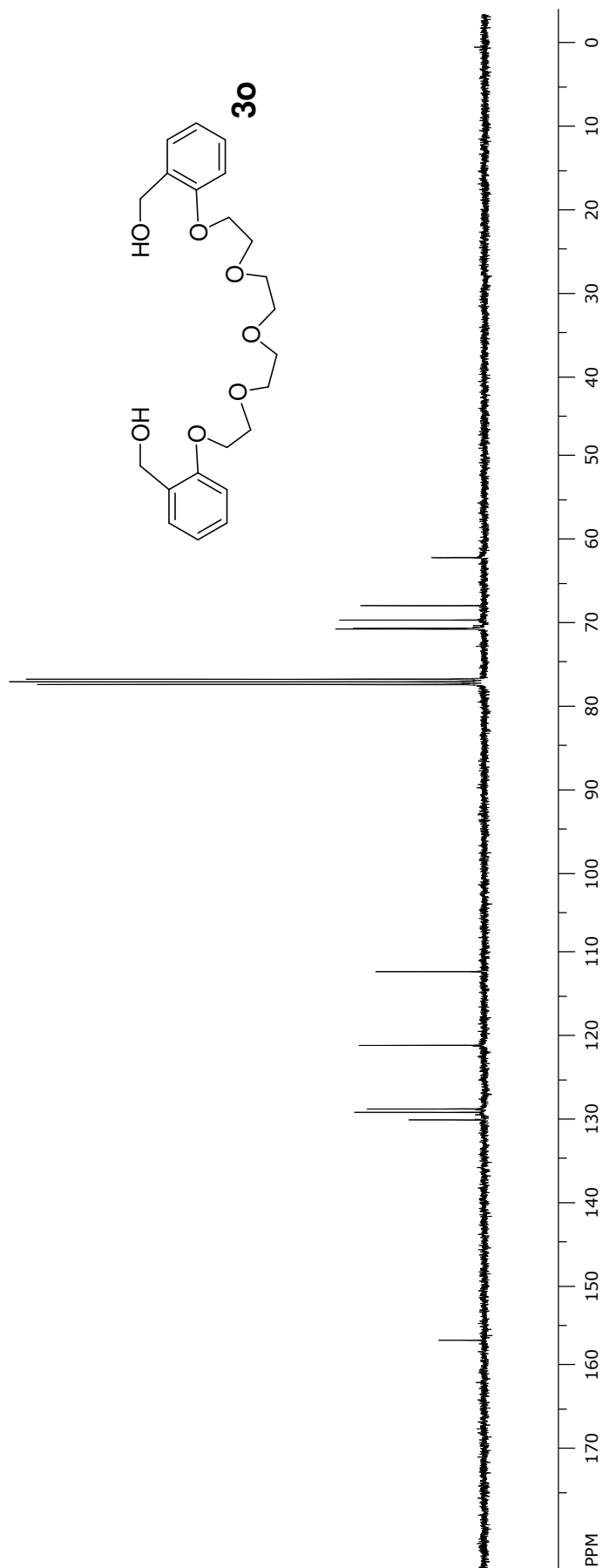
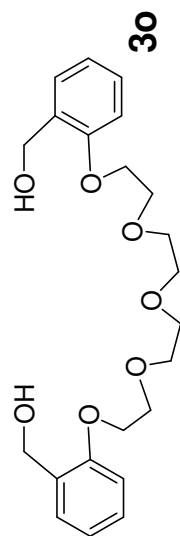
76.7387
77.0567
77.3742

112.2338 —

121.1741 —

128.9016
129.3120
130.2319

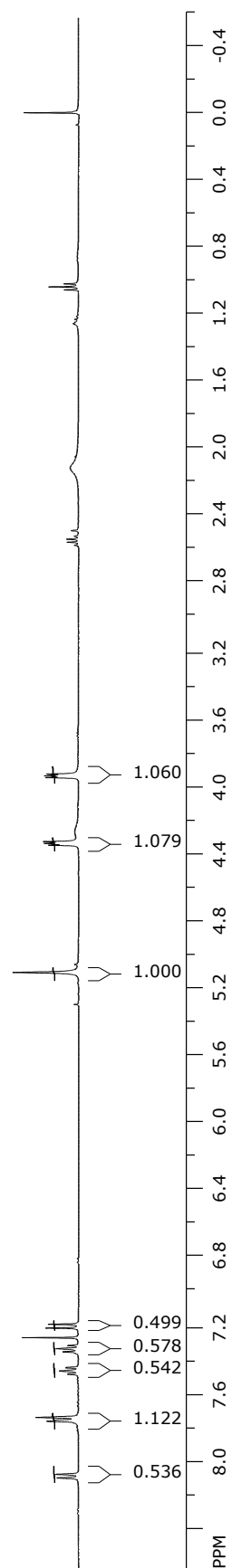
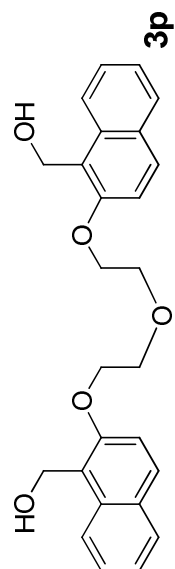
156.9725 —

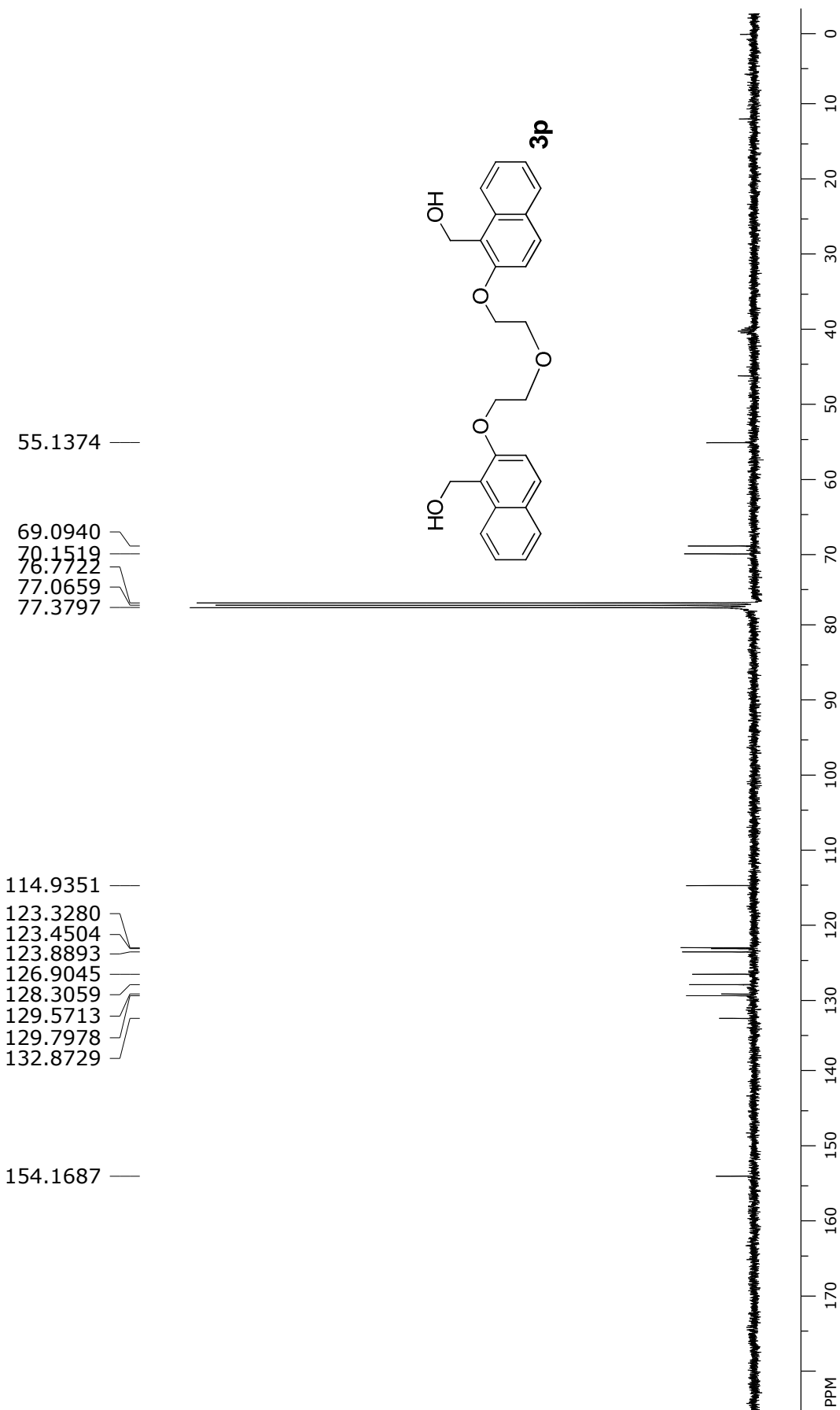


7.3314
7.3057
7.2782
7.2855
7.4620
7.4828
7.5032
7.7600
7.7829
8.0984
8.1198

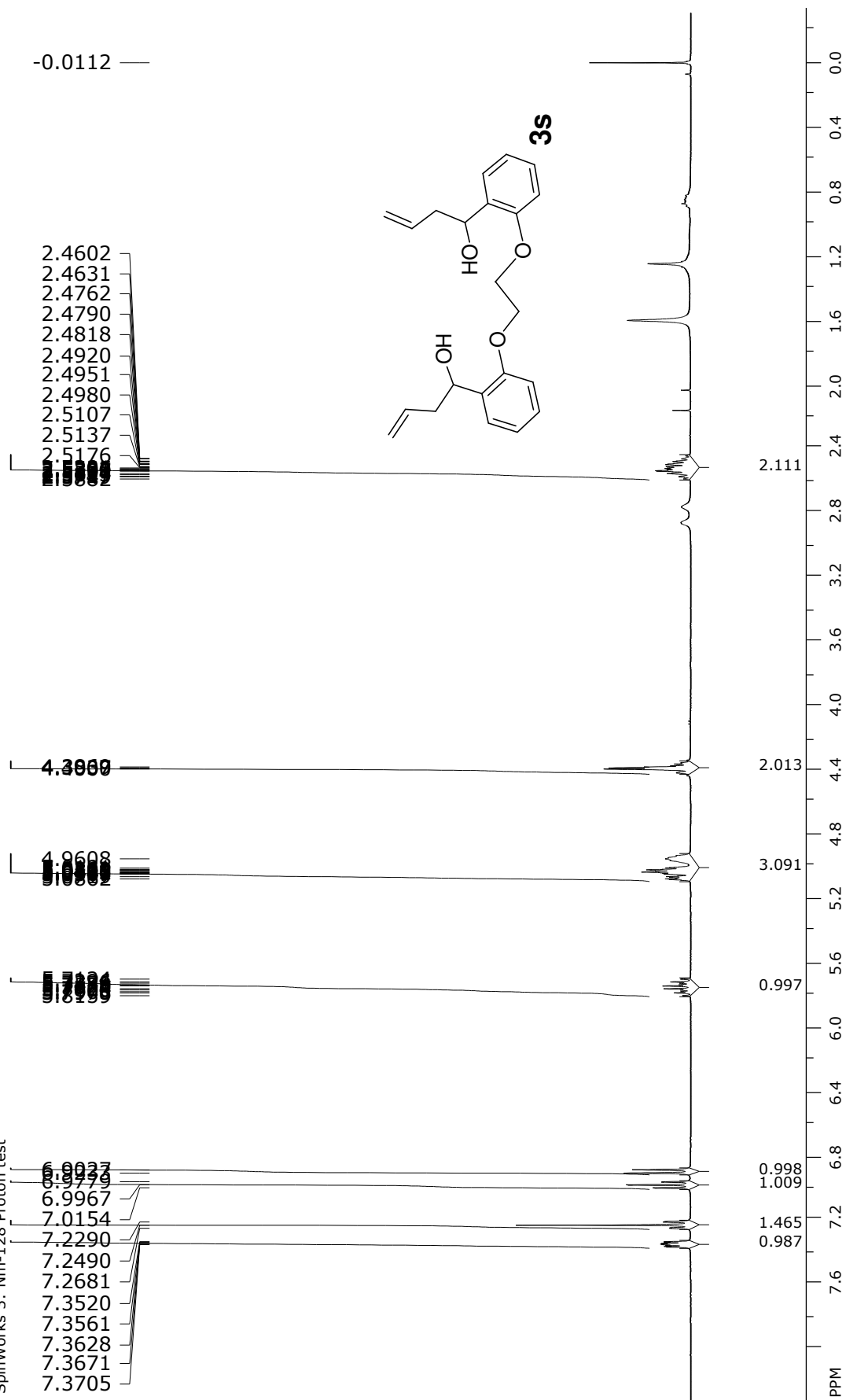
3.9323
3.9383
3.9429
3.9481
3.9537
4.3343
4.3399
4.3451
4.3496
4.3557

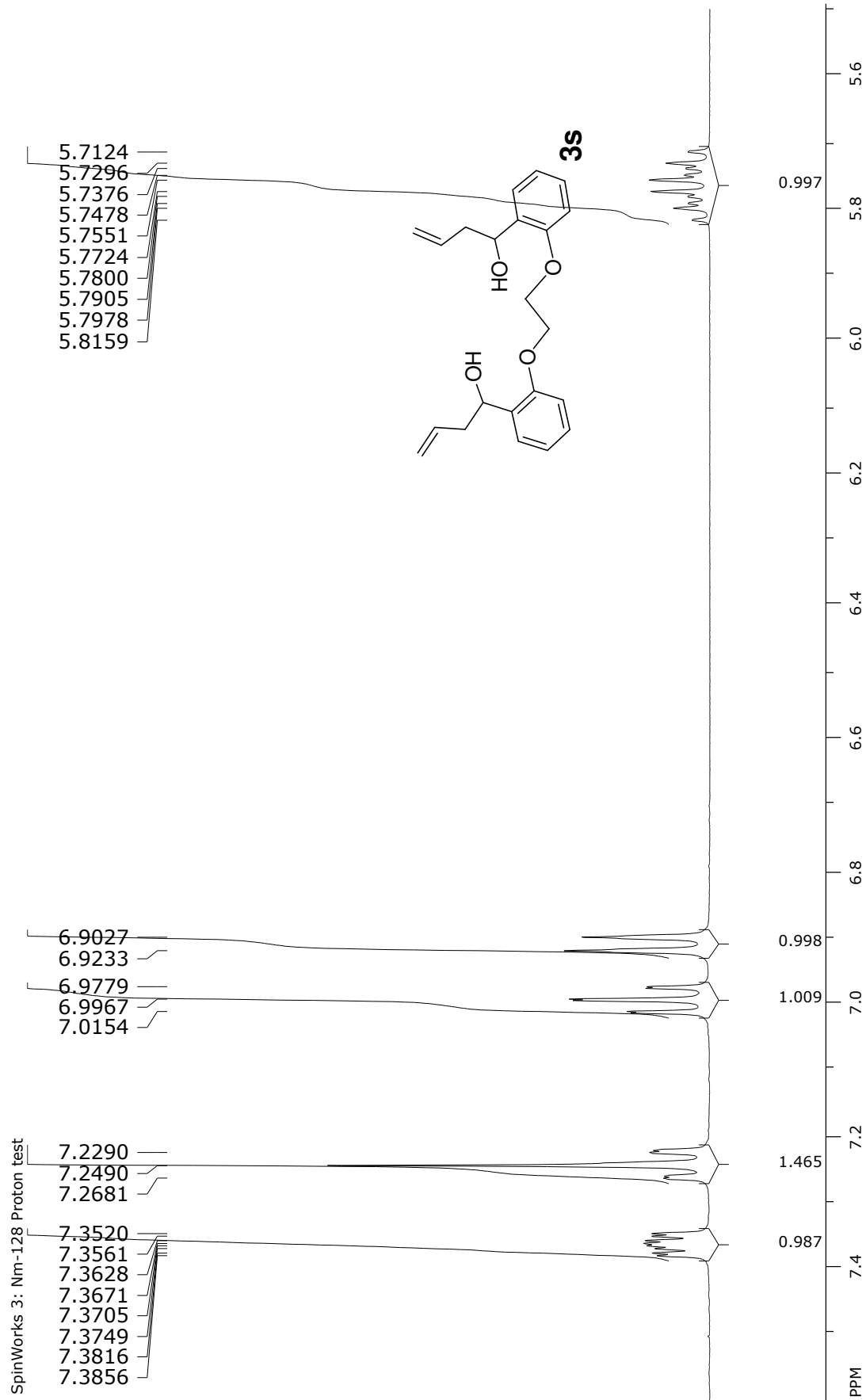
5.1125



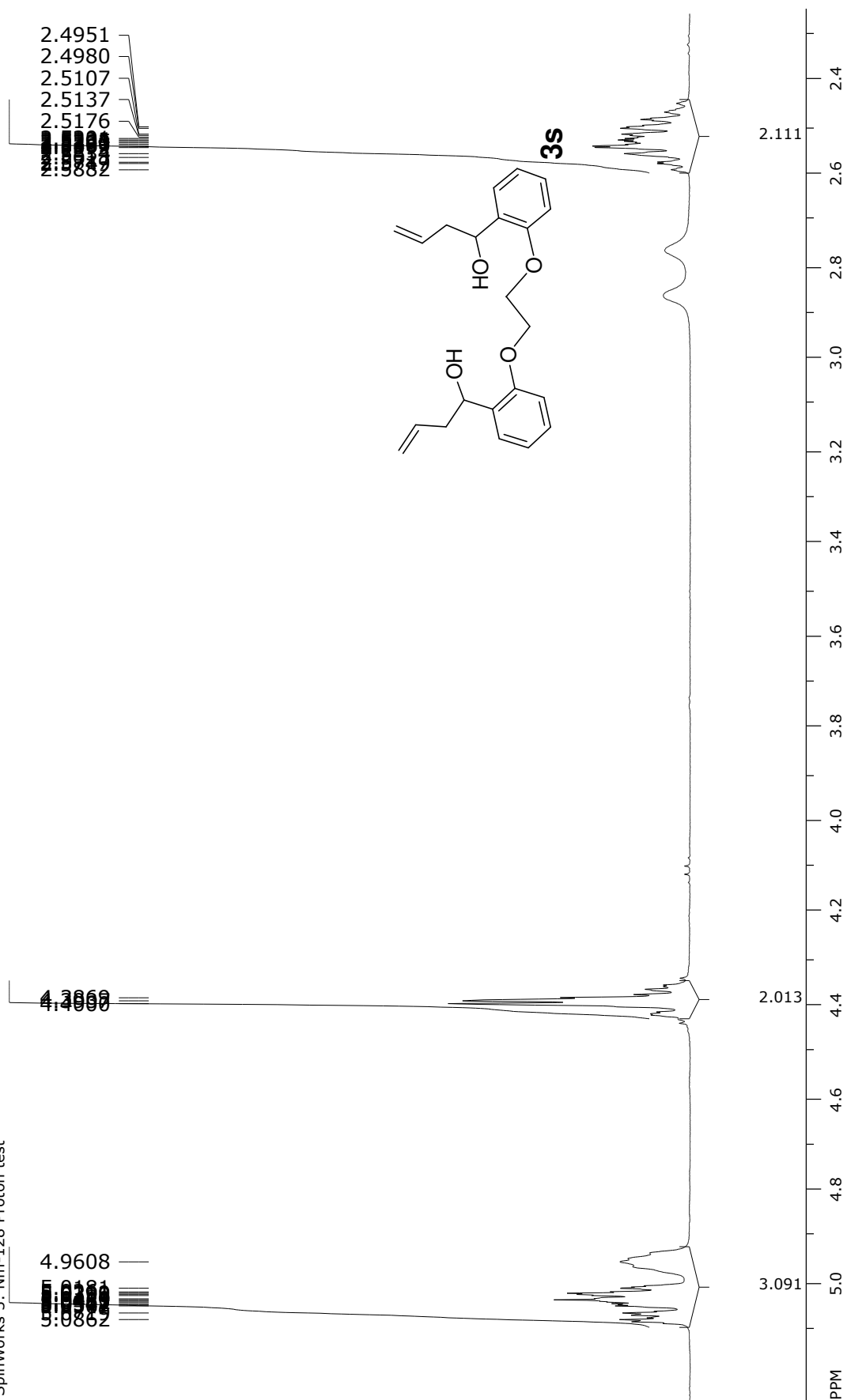


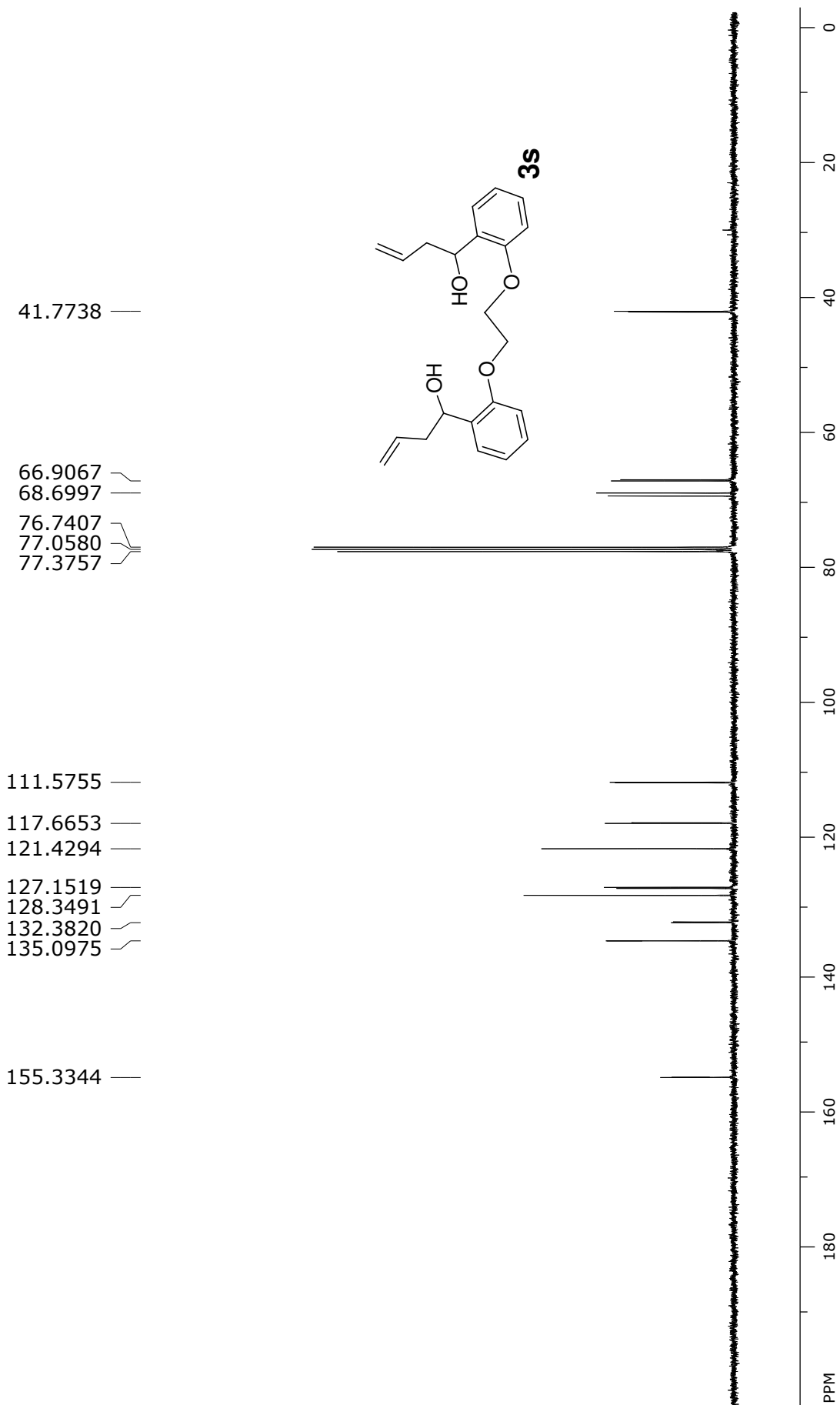
SpinWorks 3: Nm-128 Proton test

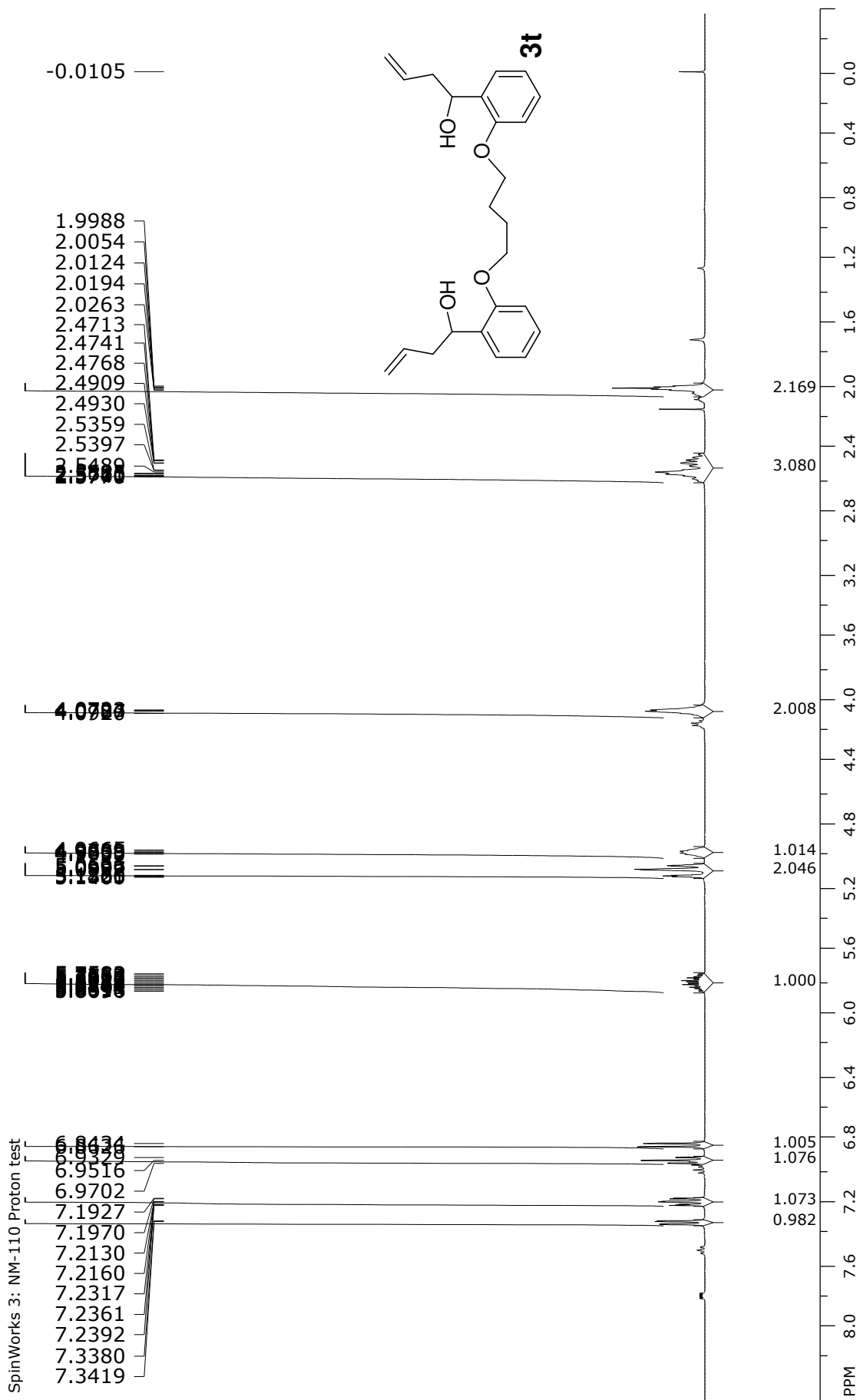




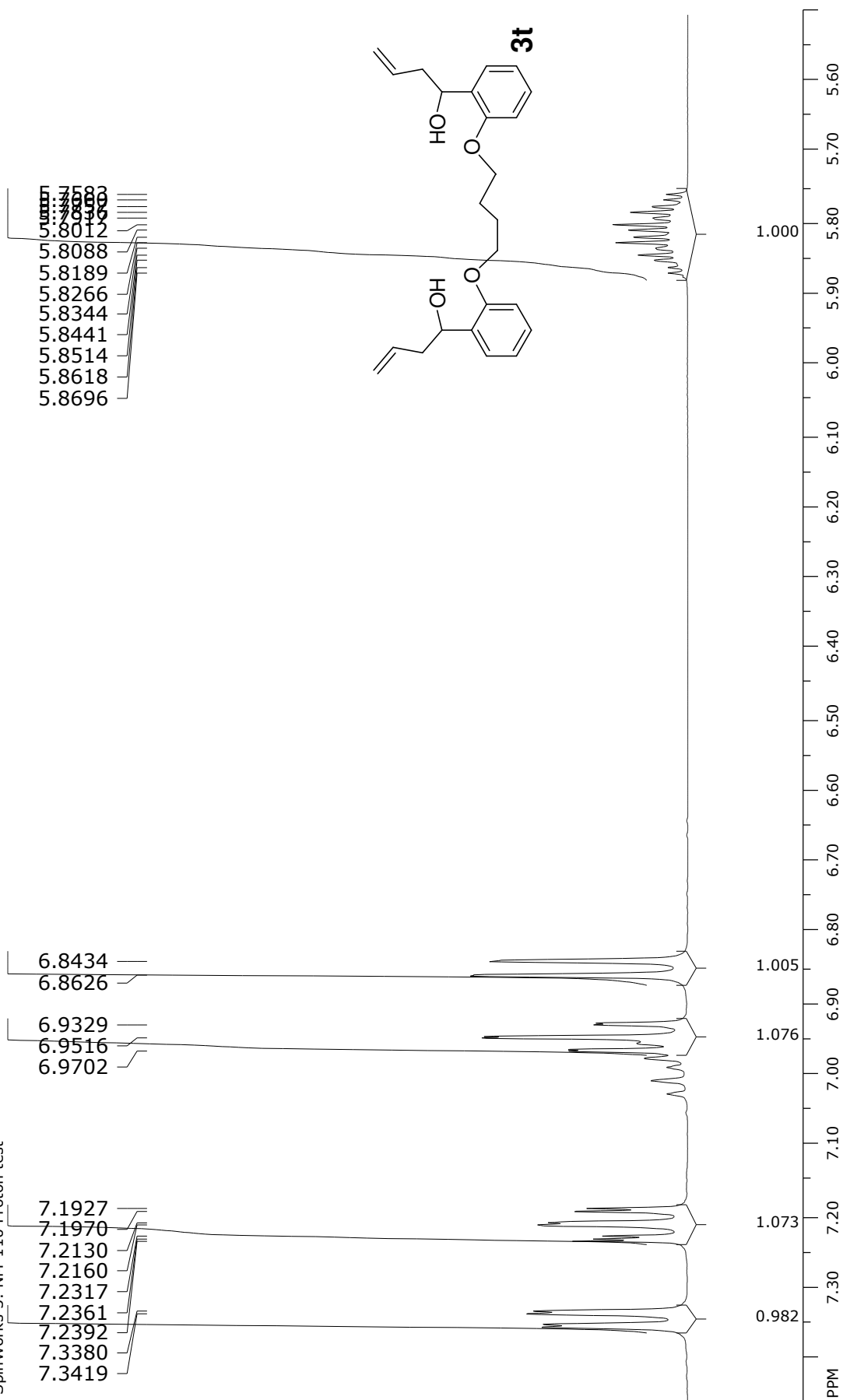
SpinWorks 3: Nm-128 Proton test



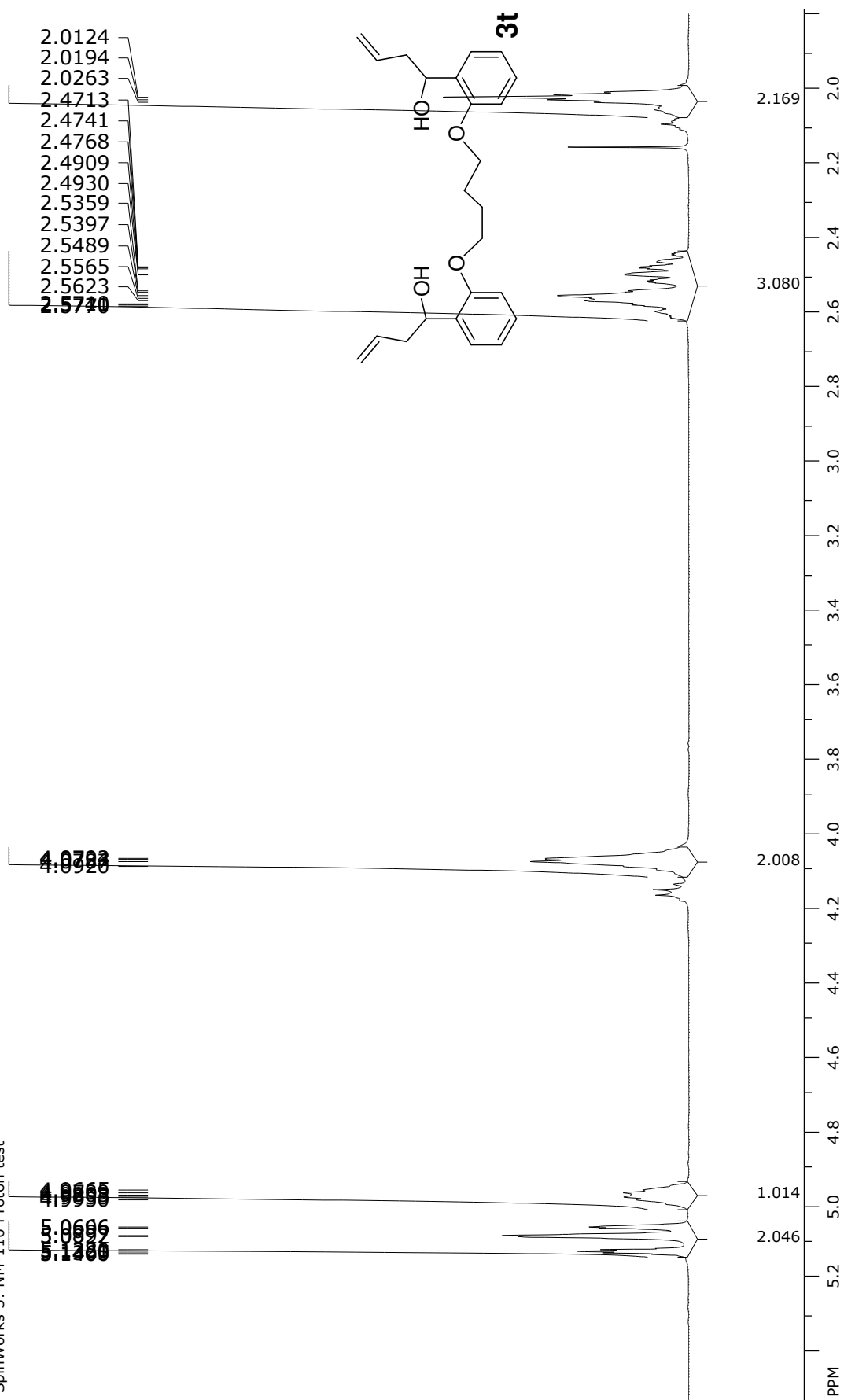




SpinWorks 3: NM-110 Proton test



SpinWorks 3: NM-110 Proton test



155.5441 —

126.7807 —

128.2954 —

131.9158 —

135.1681 —

111.1414 —

117.6386 —

120.7644 —

67.3440 —

69.4113 —

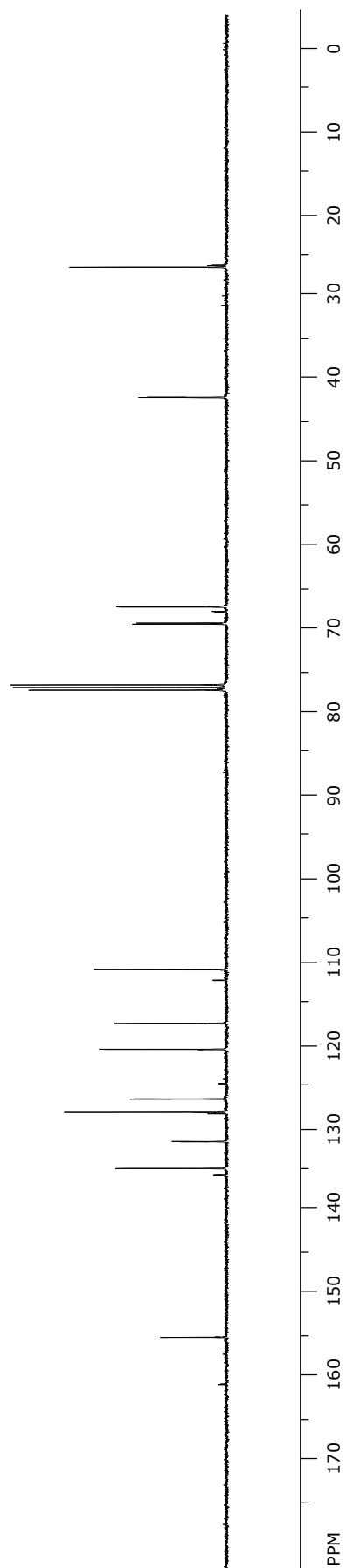
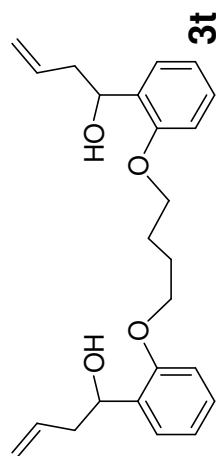
76.7571 —

77.0747 —

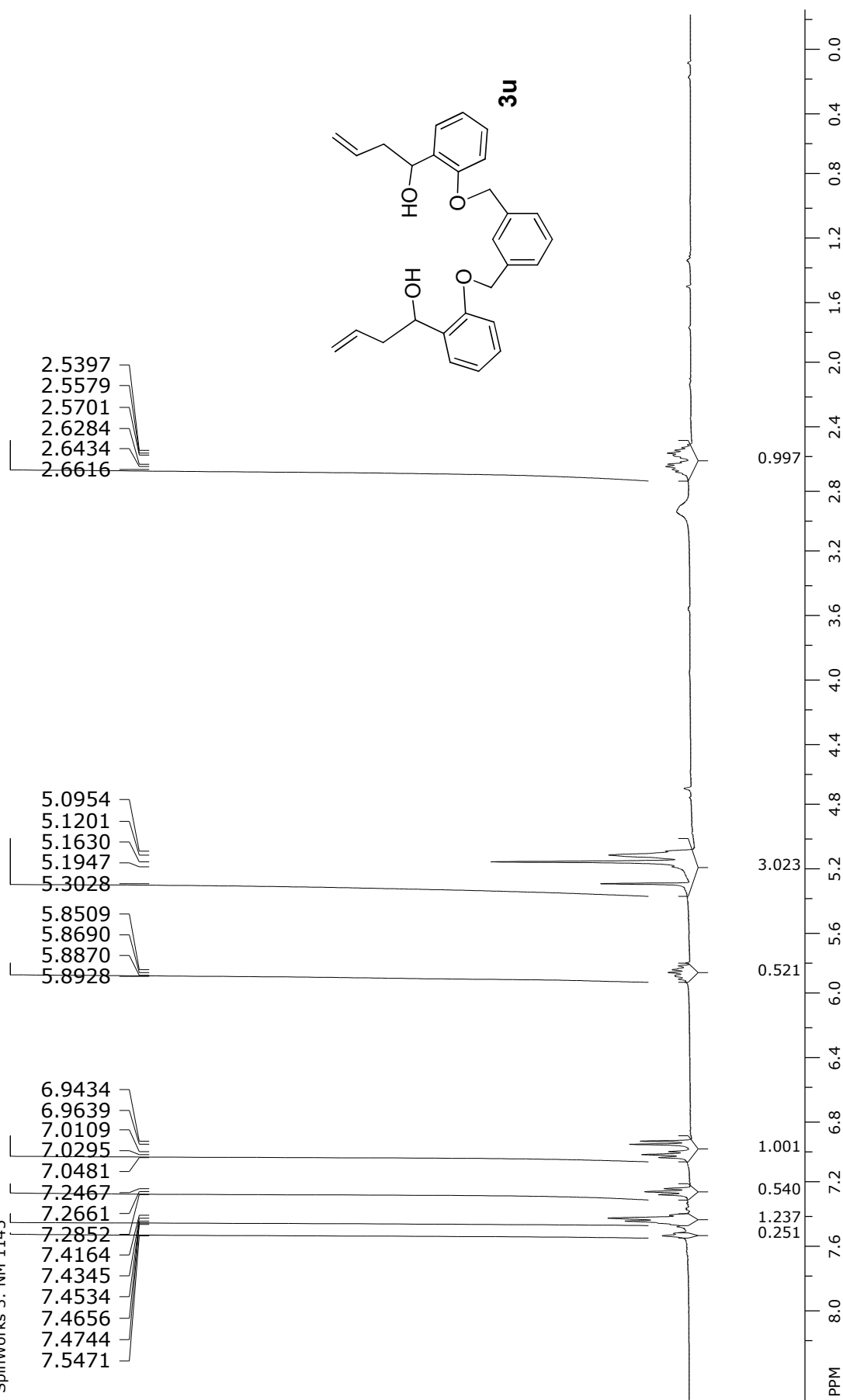
77.3930 —

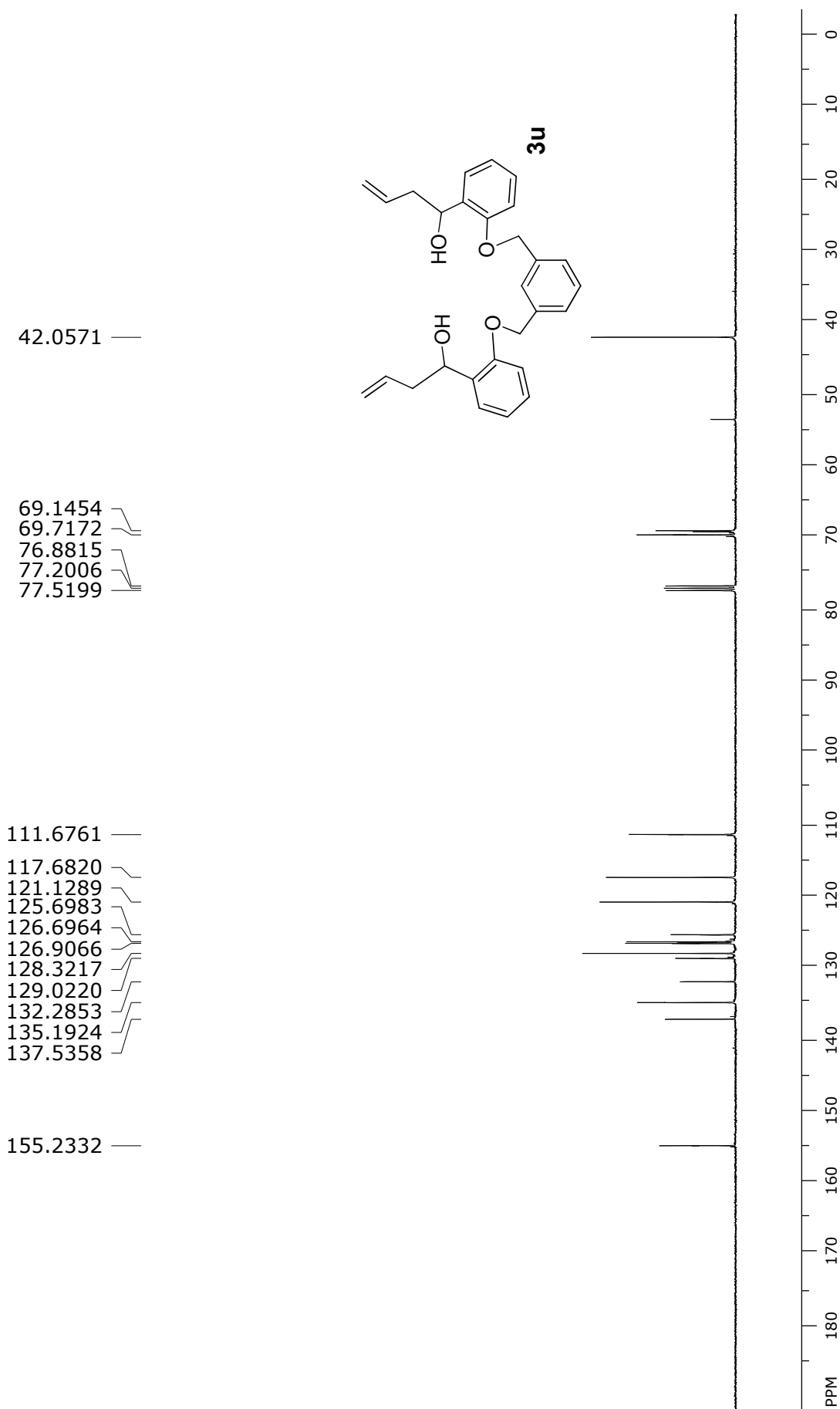
42.0356 —

26.3065 —

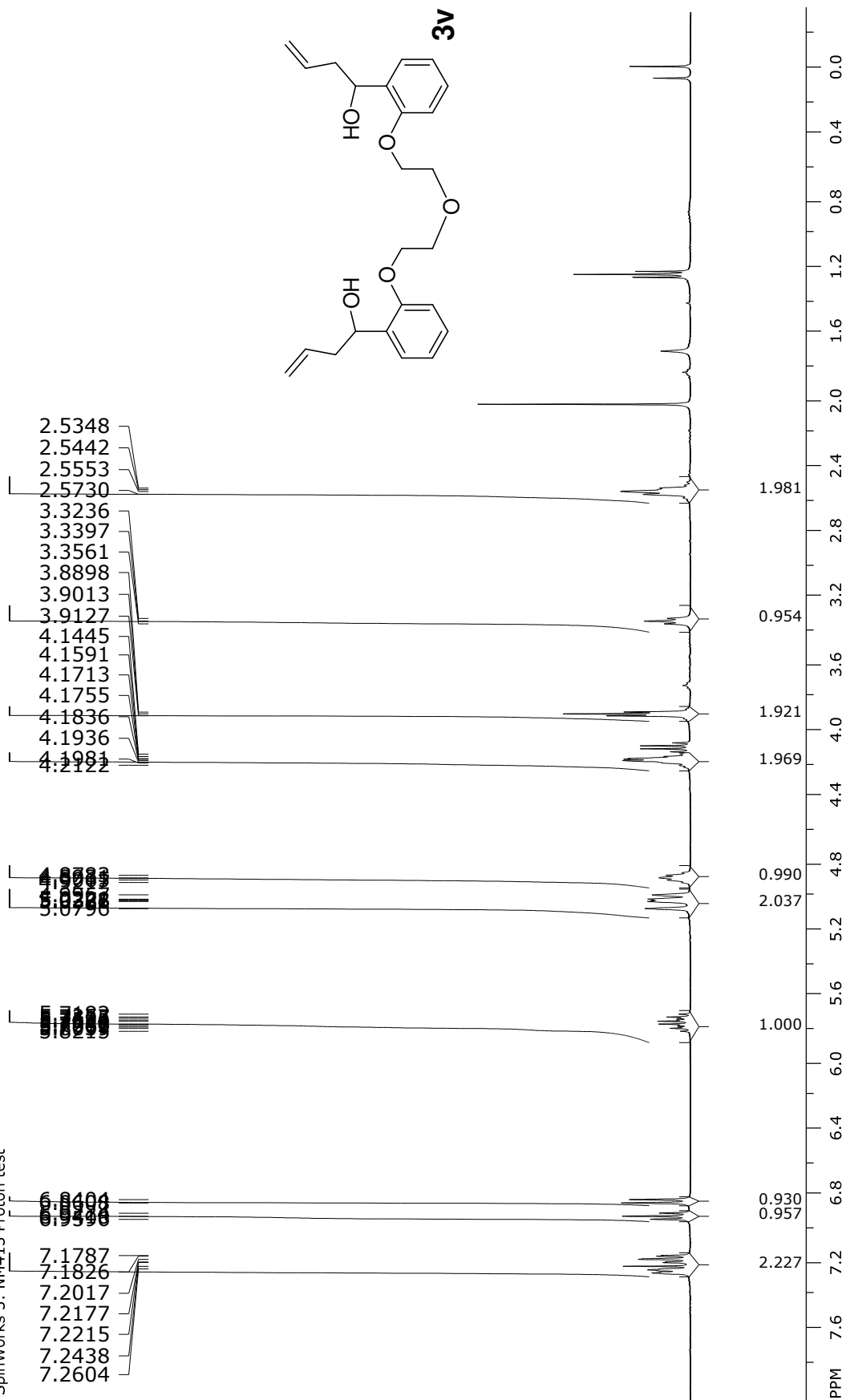


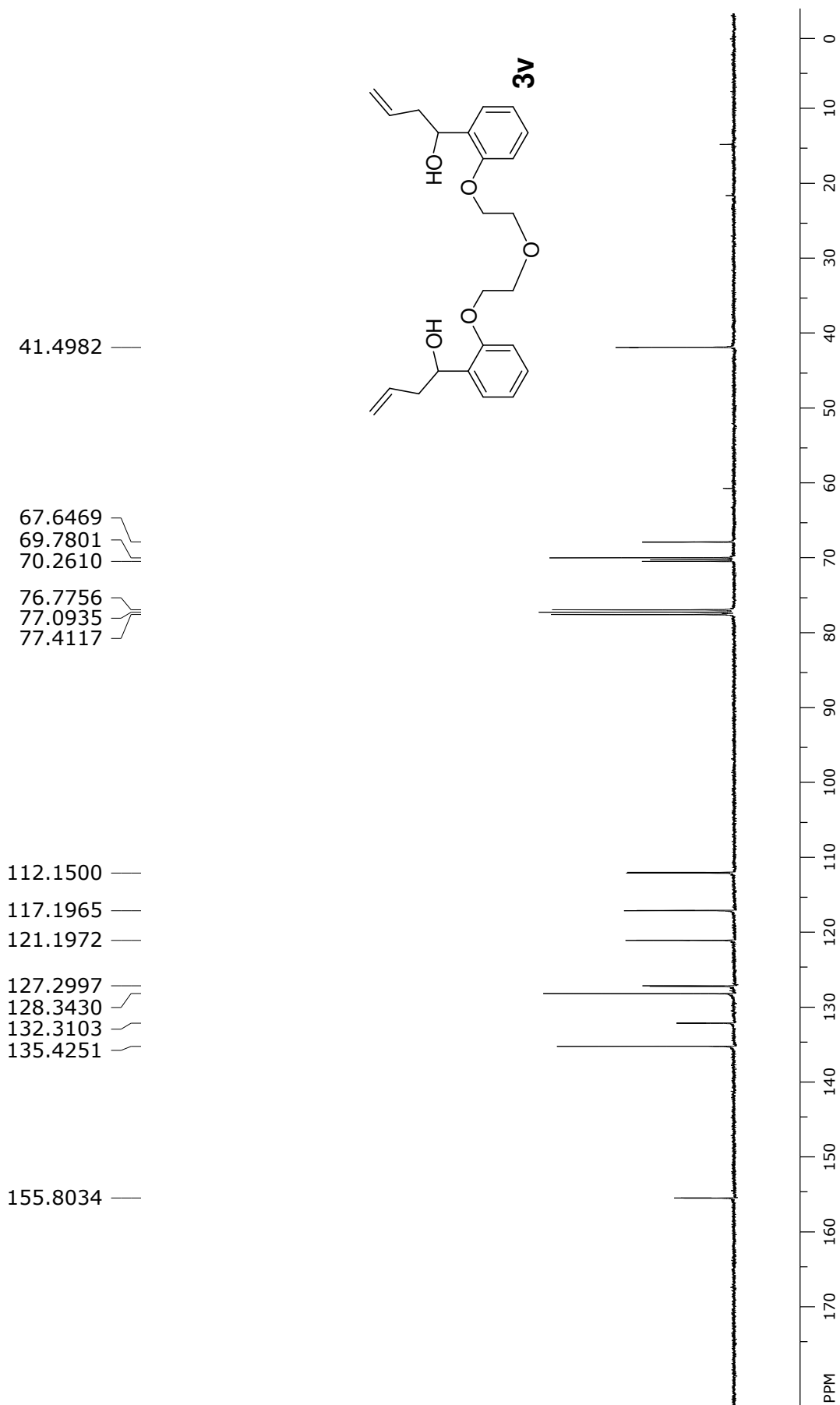
SpinWorks 3: NM 1145

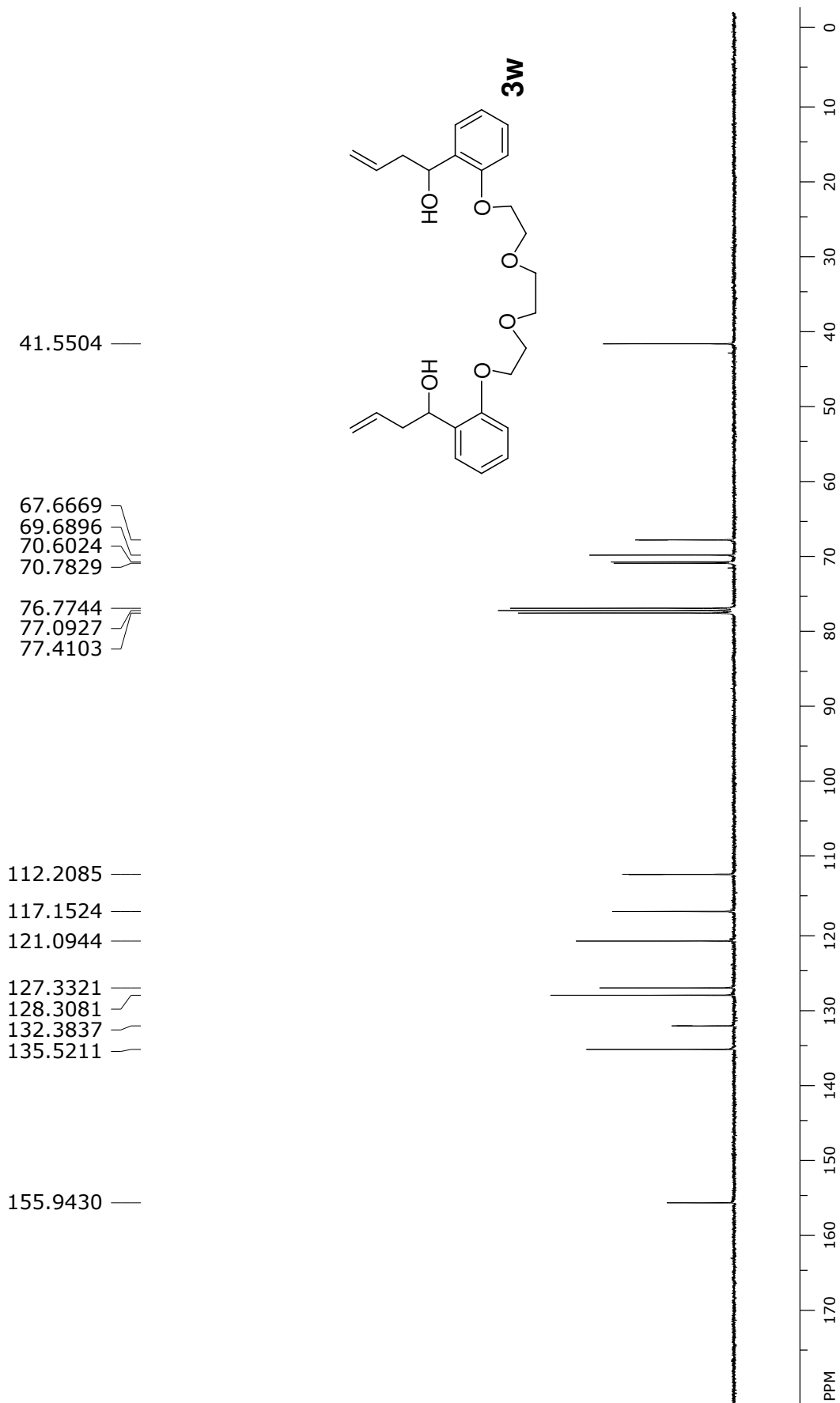




SpinWorks 3: NM413 Proton test





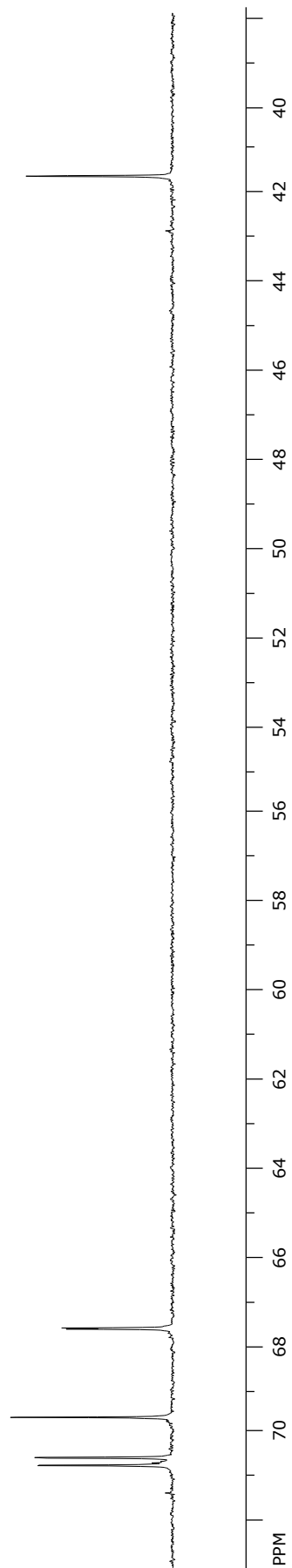
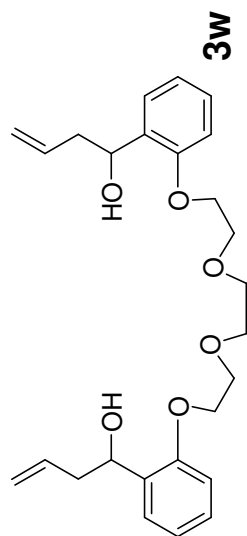


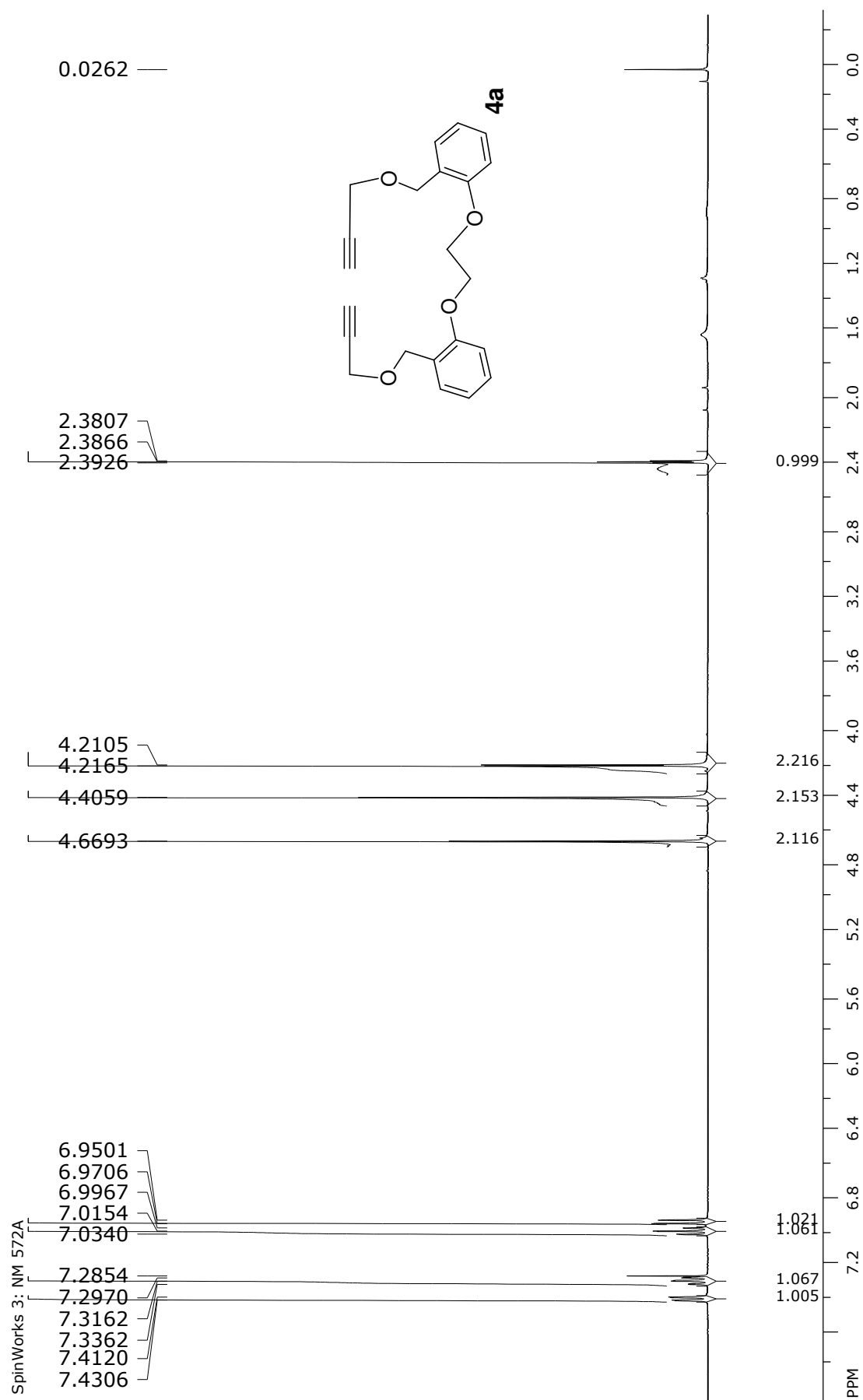
SpinWorks 3: NM 417

67.6669 —

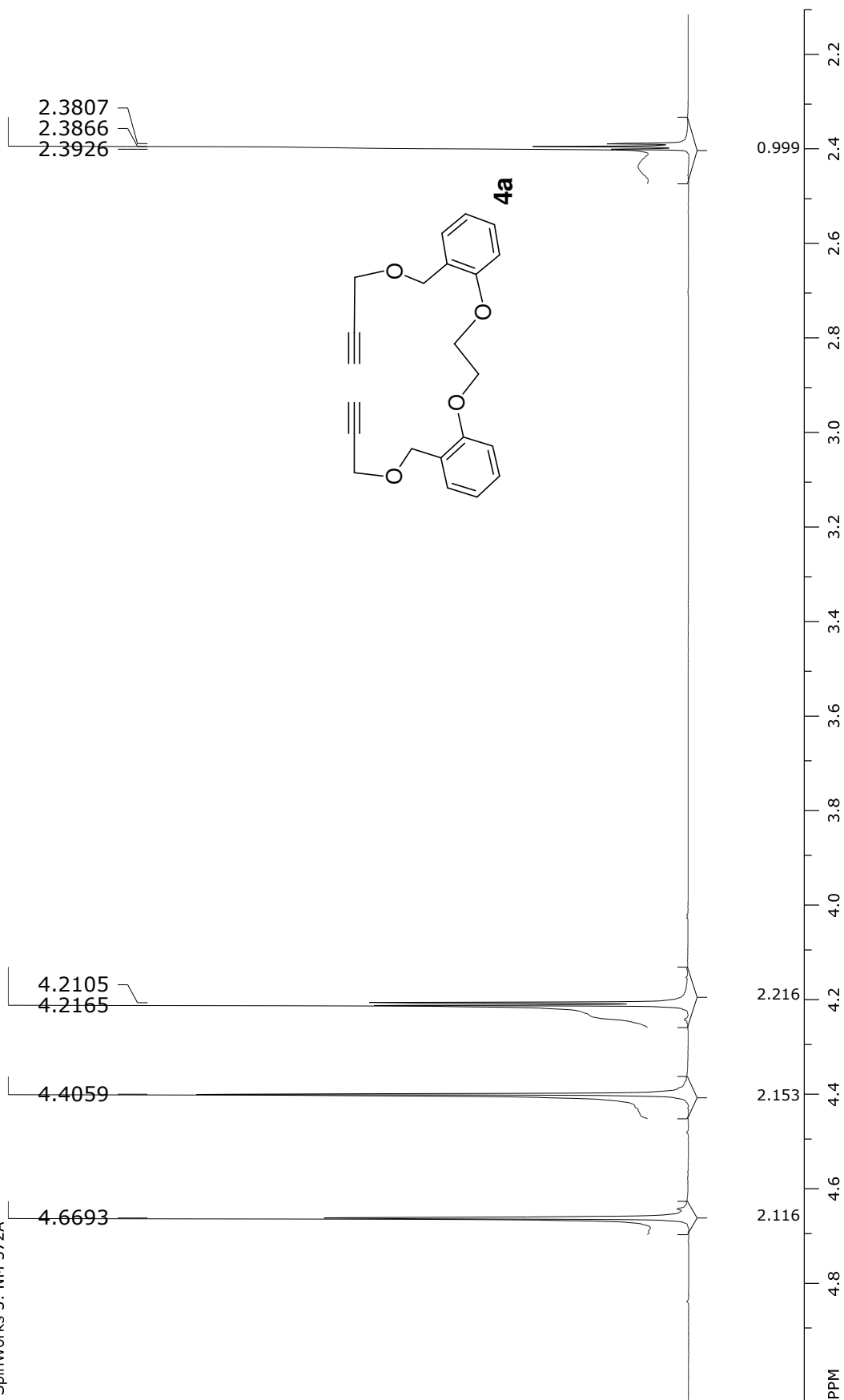
69.6896 —
70.6024 —
70.7829 —

41.5504 —





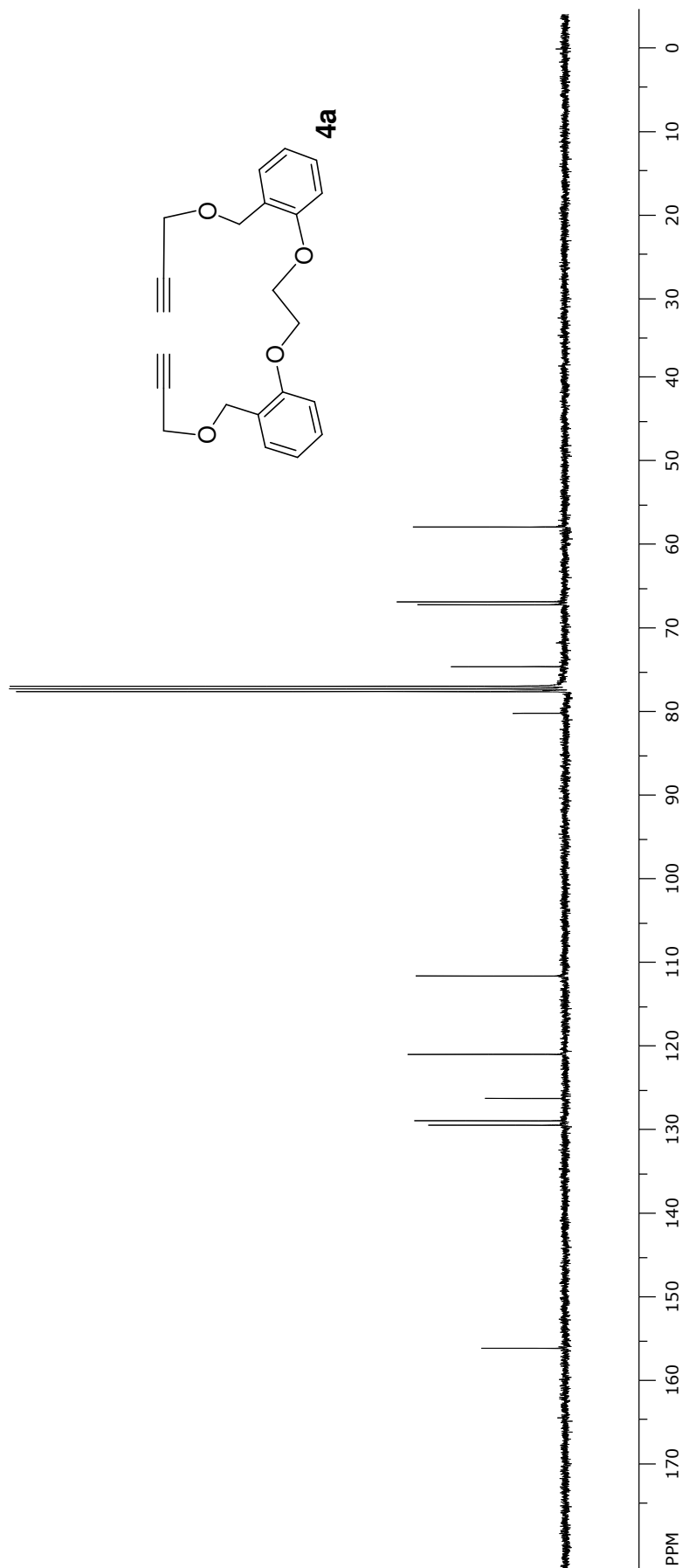
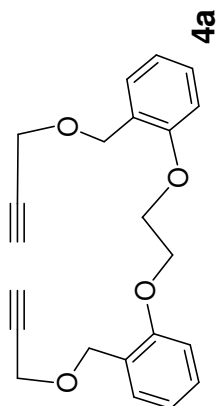
SpinWorks 3: NM 572A

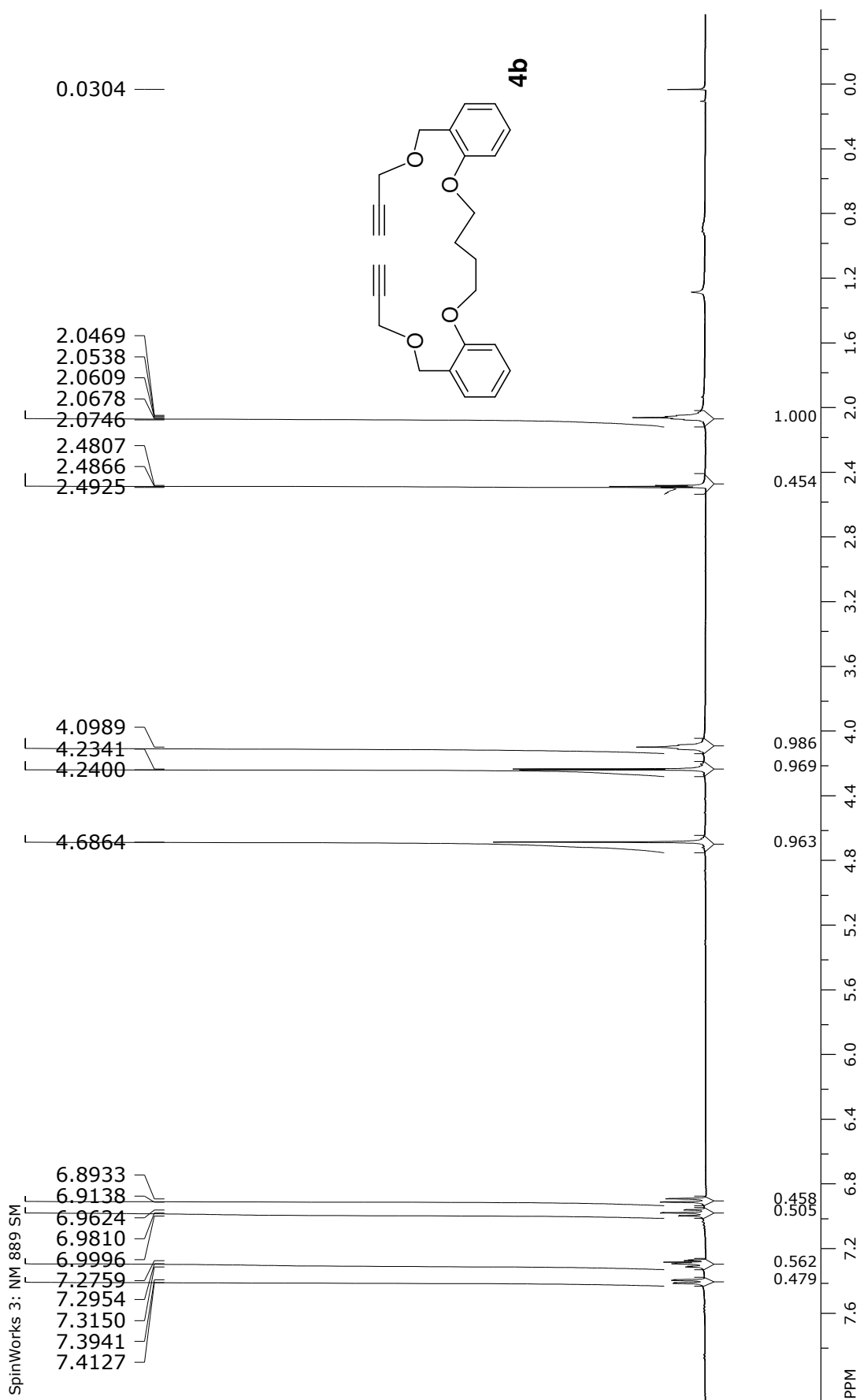


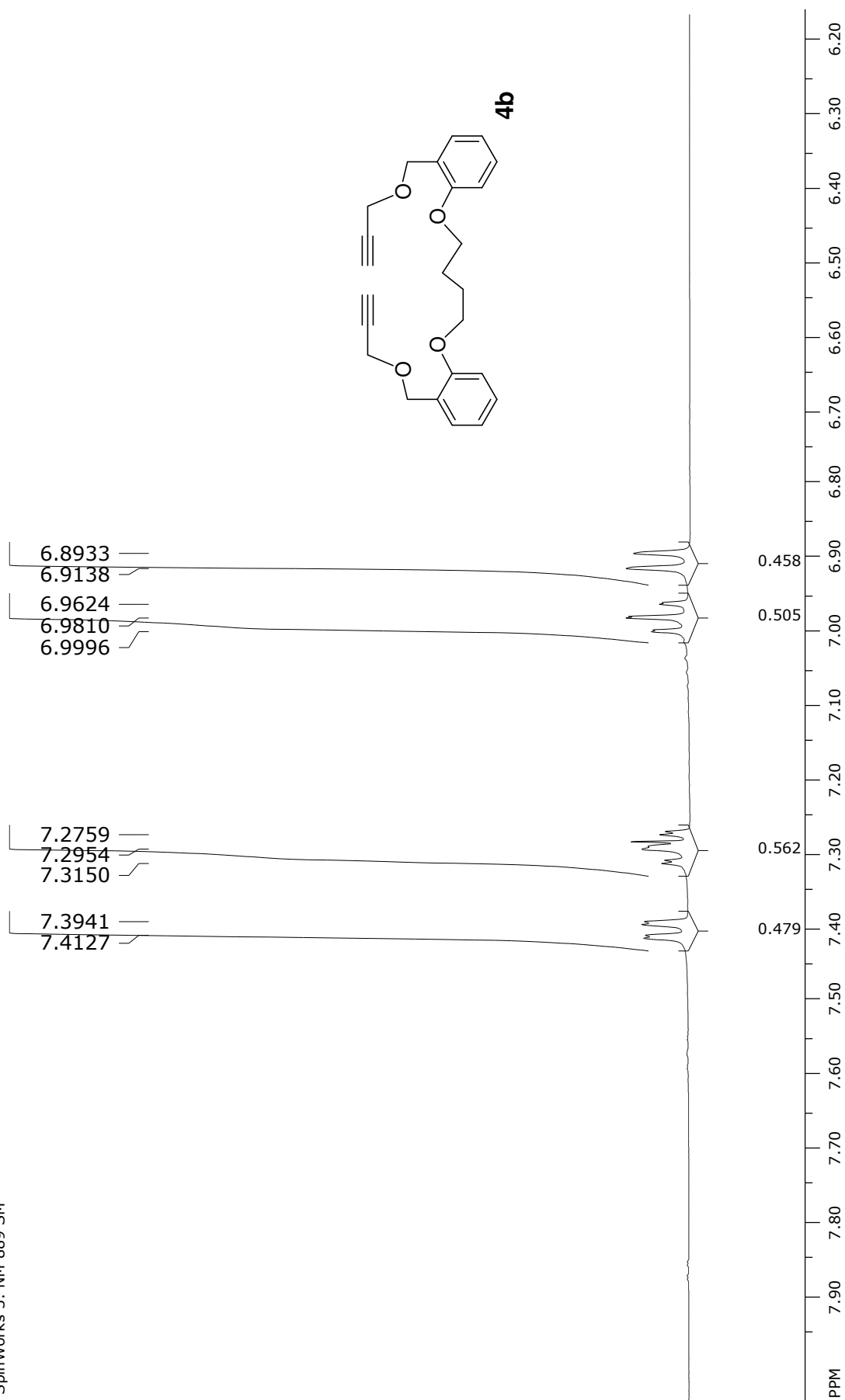
57.5598 —
66.5649 —
66.9218 —
74.3759 —
76.6266 —
76.7344 —
77.0514 —
77.2548 —
77.3694 —
79.9787 —

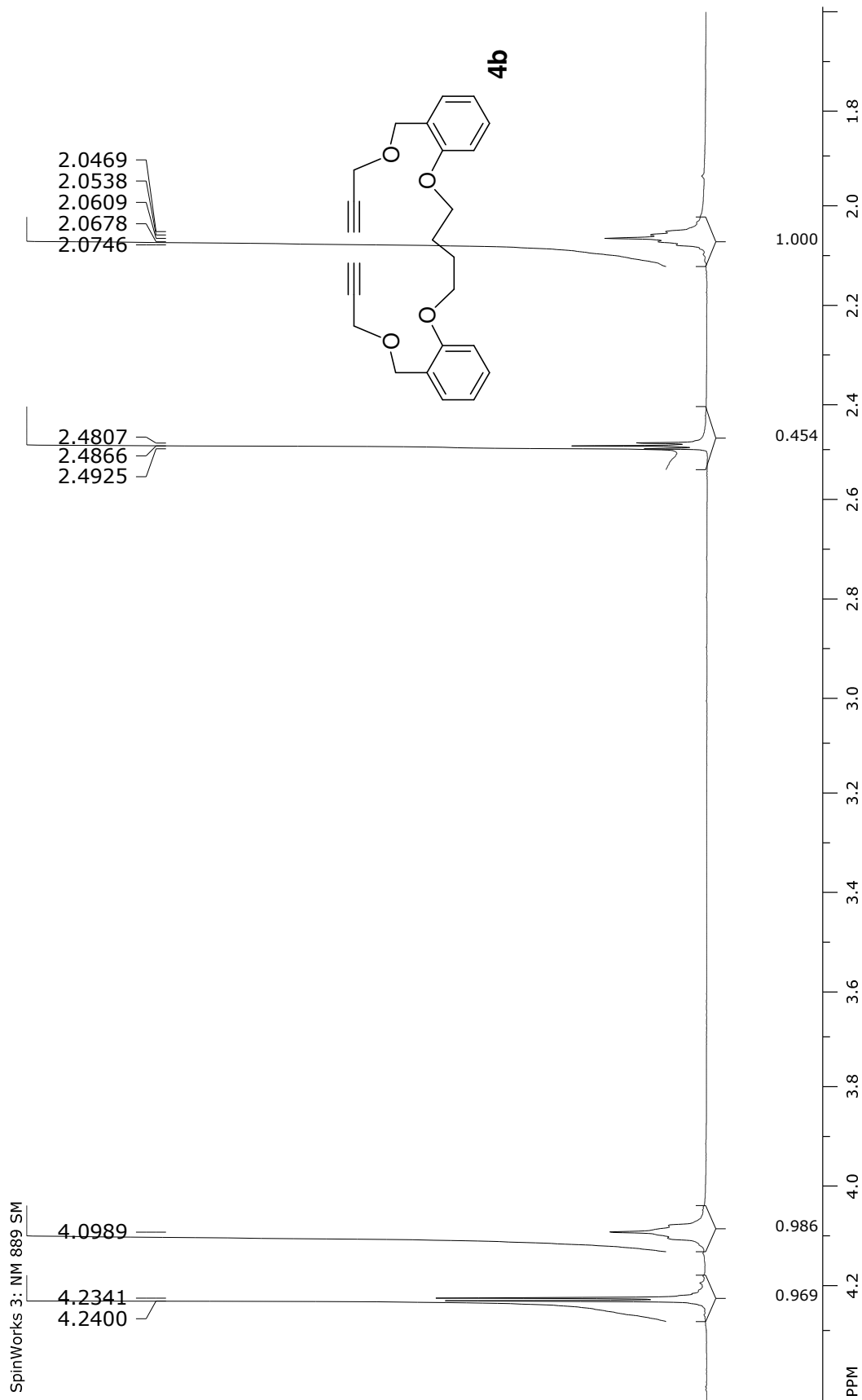
111.6034 —
121.0307 —
126.3393 —
129.0229 —
129.5560 —

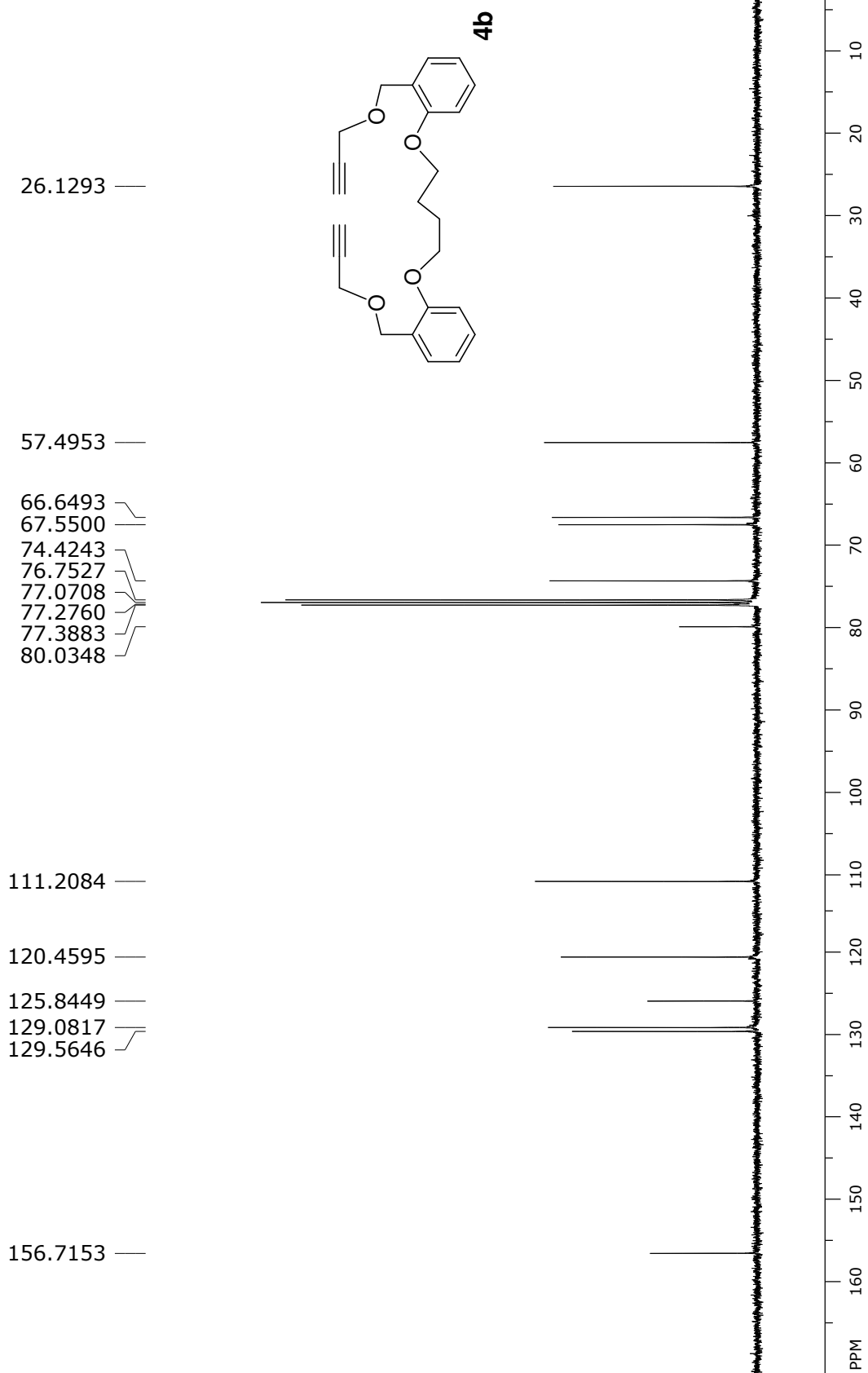
156.4078 —



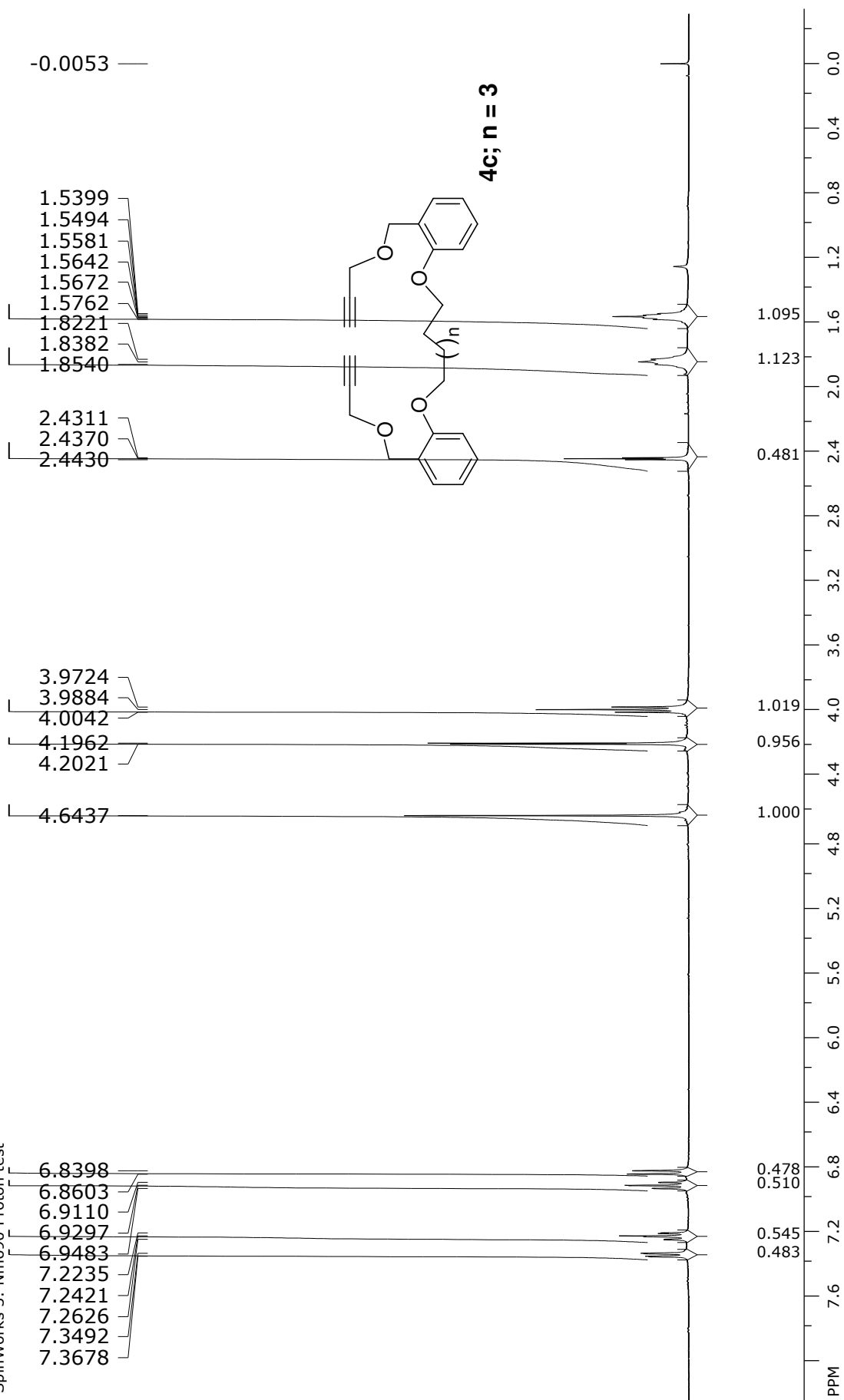




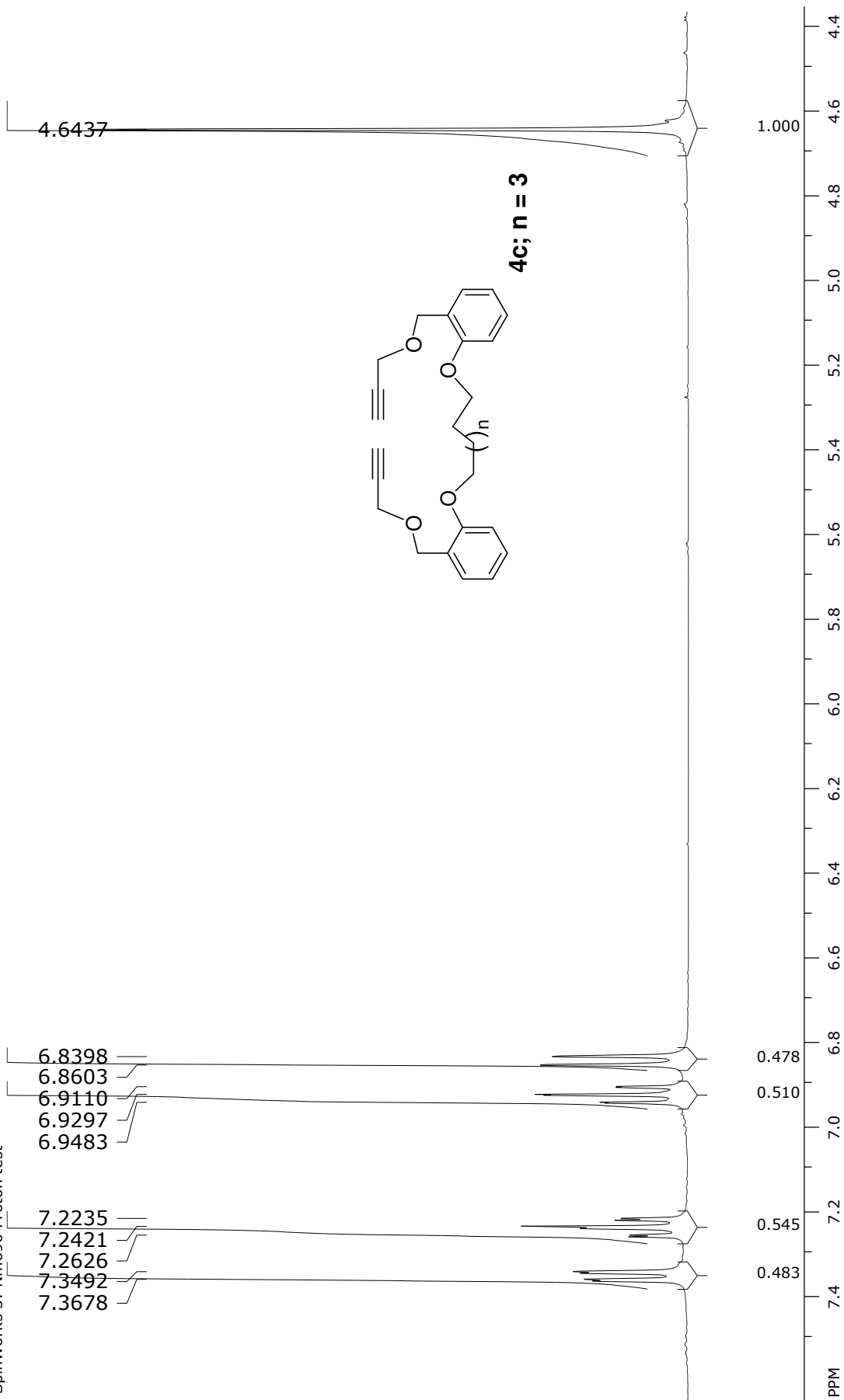




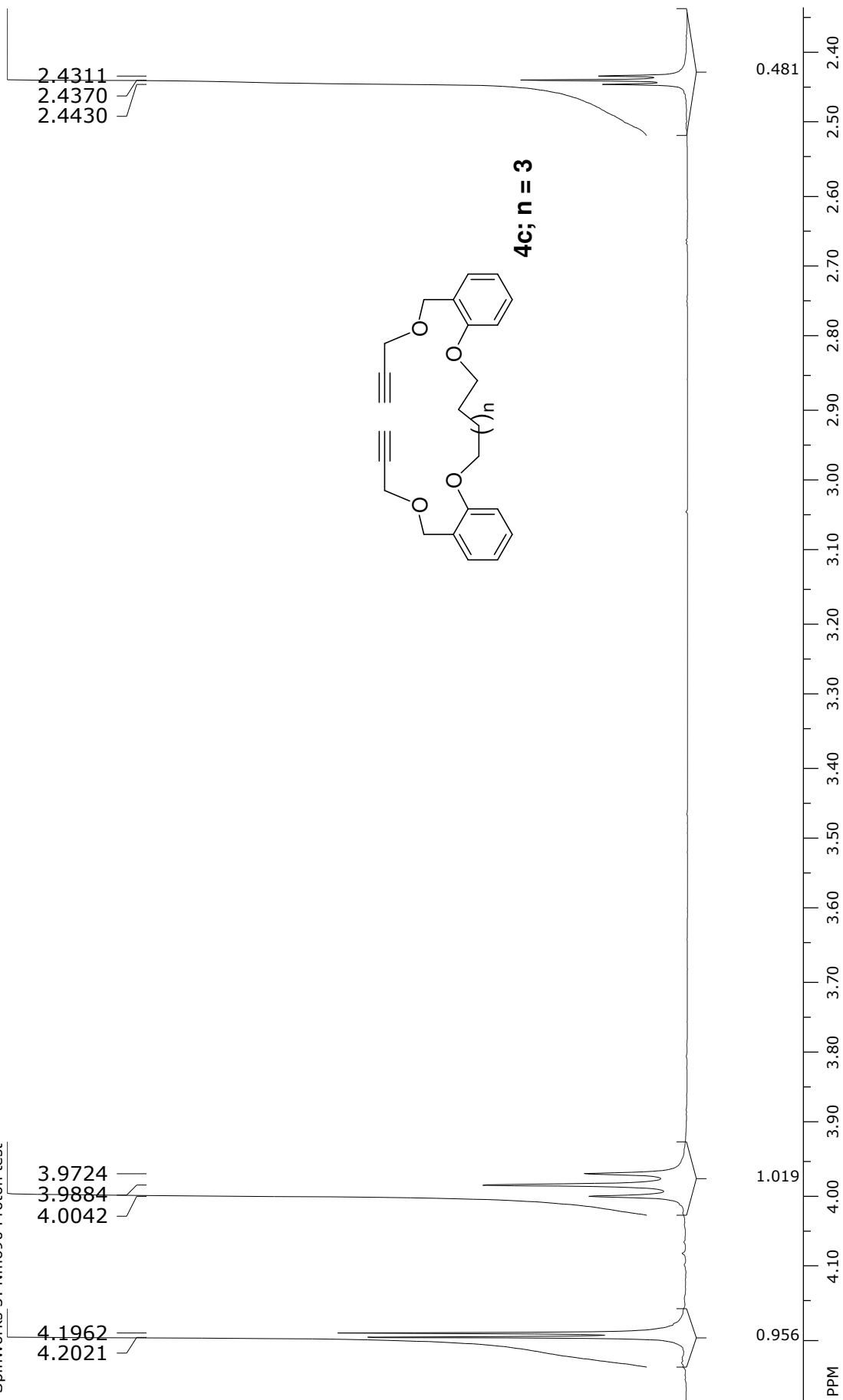
SpinWorks 3: Nm690 Proton test

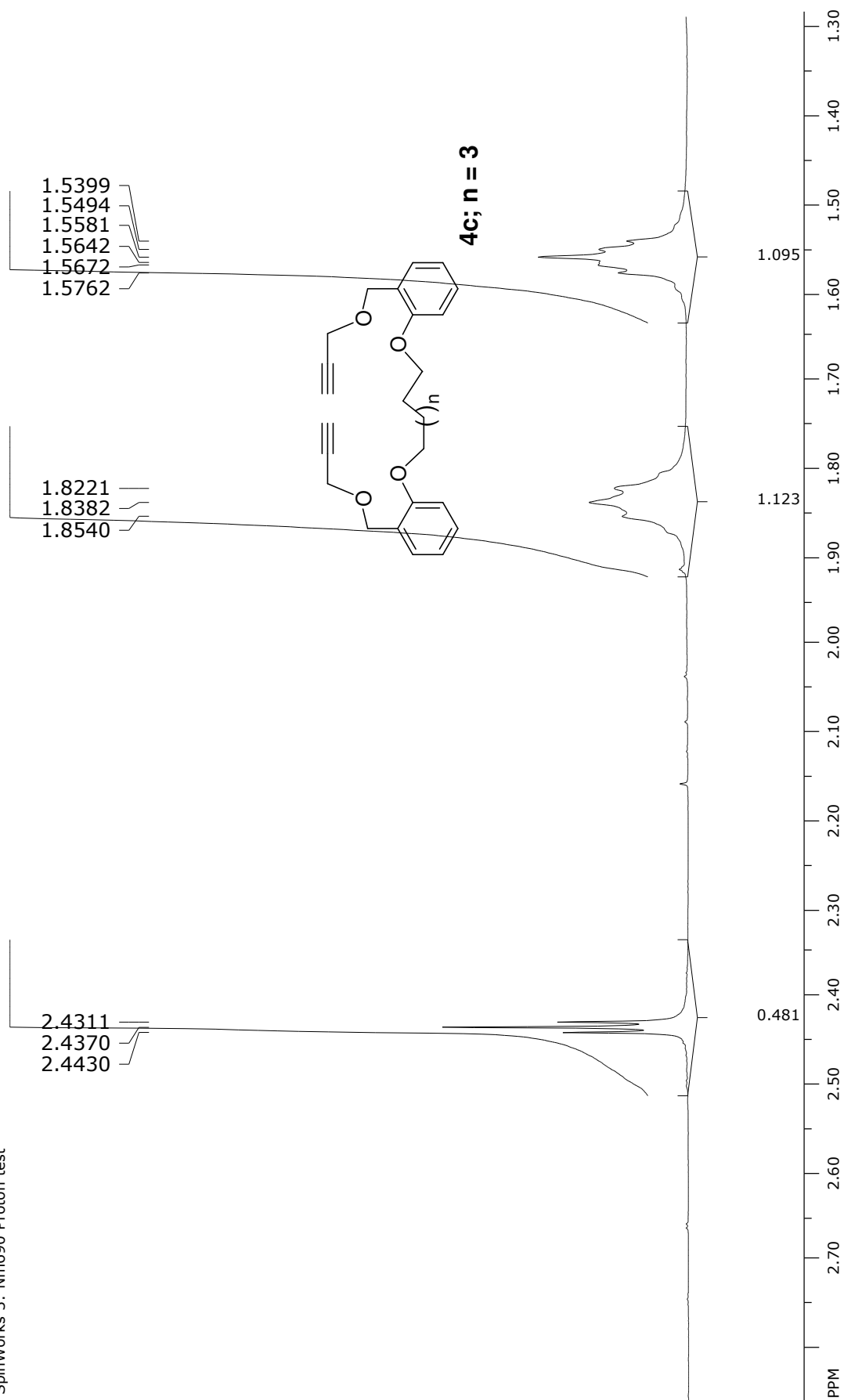


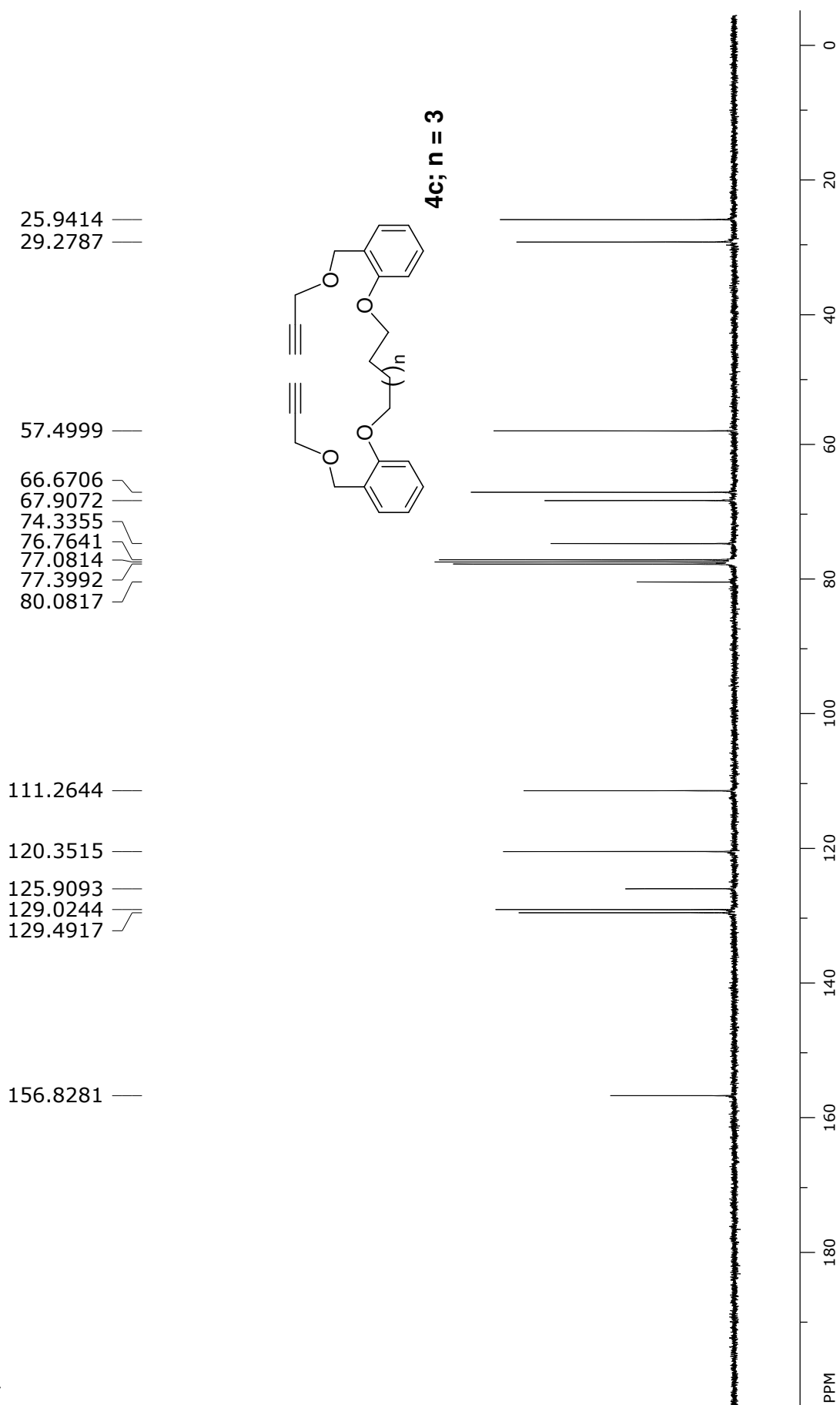
SpinWorks 3: Nm690 Proton test

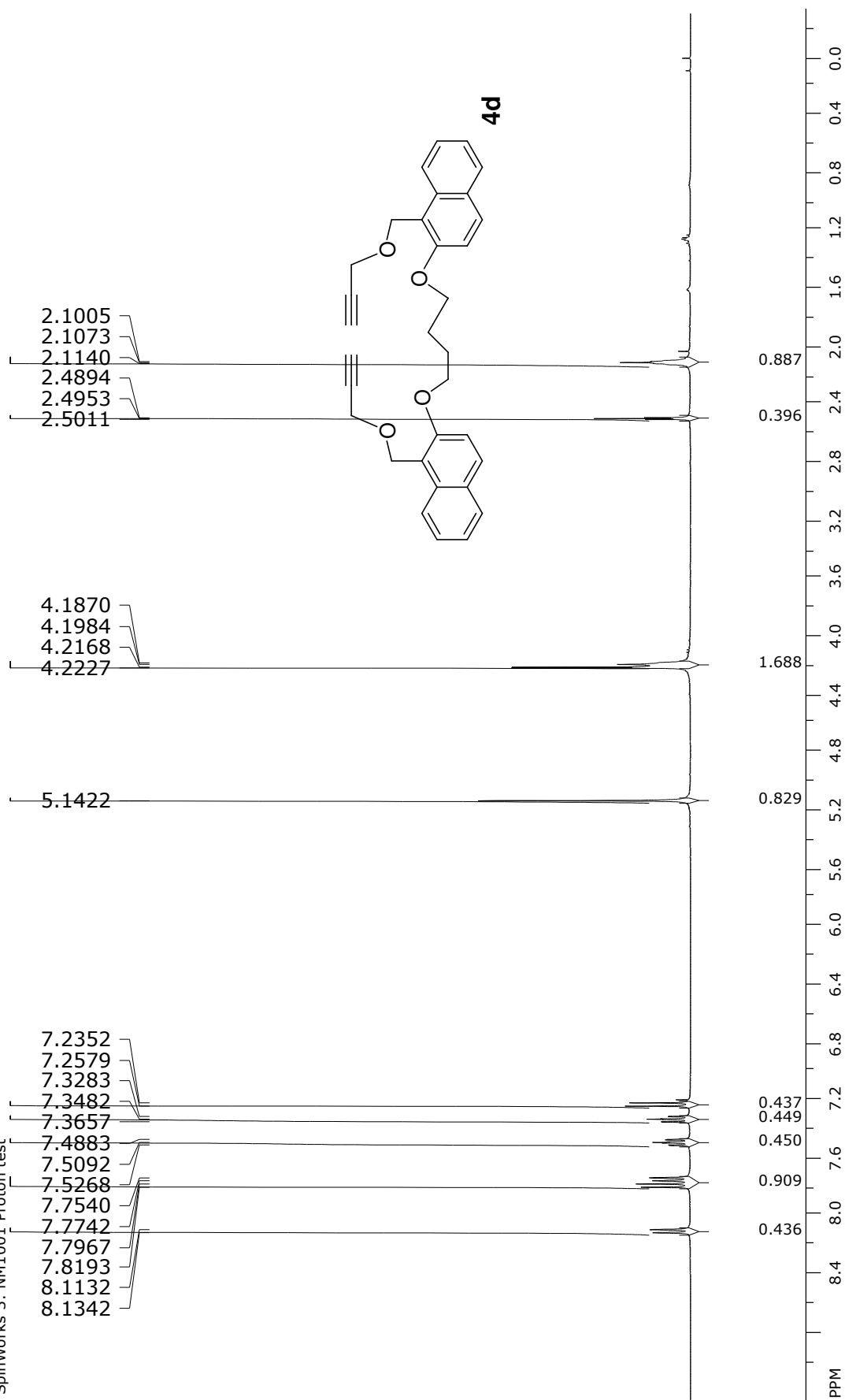


SpinWorks 3: Nm690 Proton test

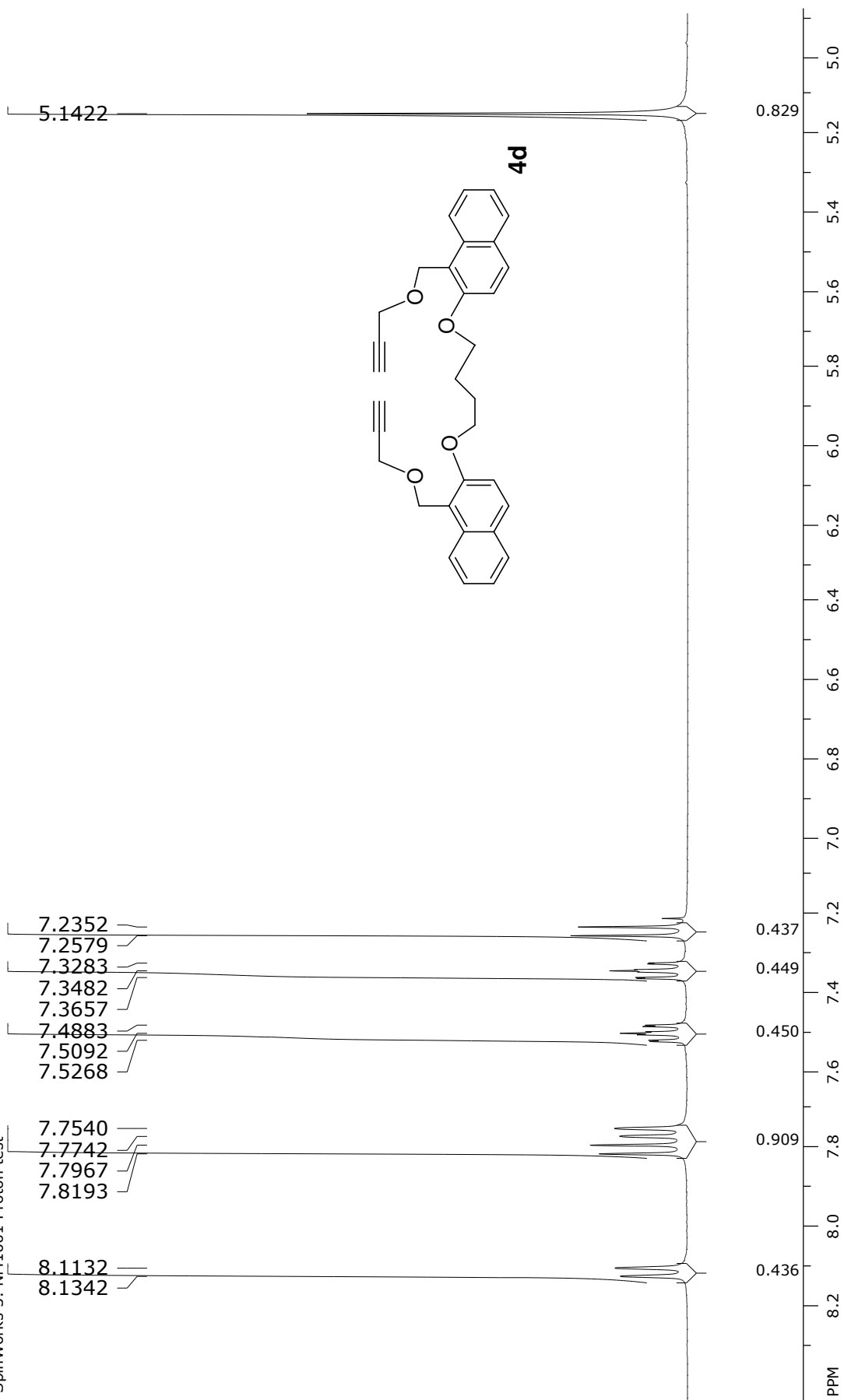




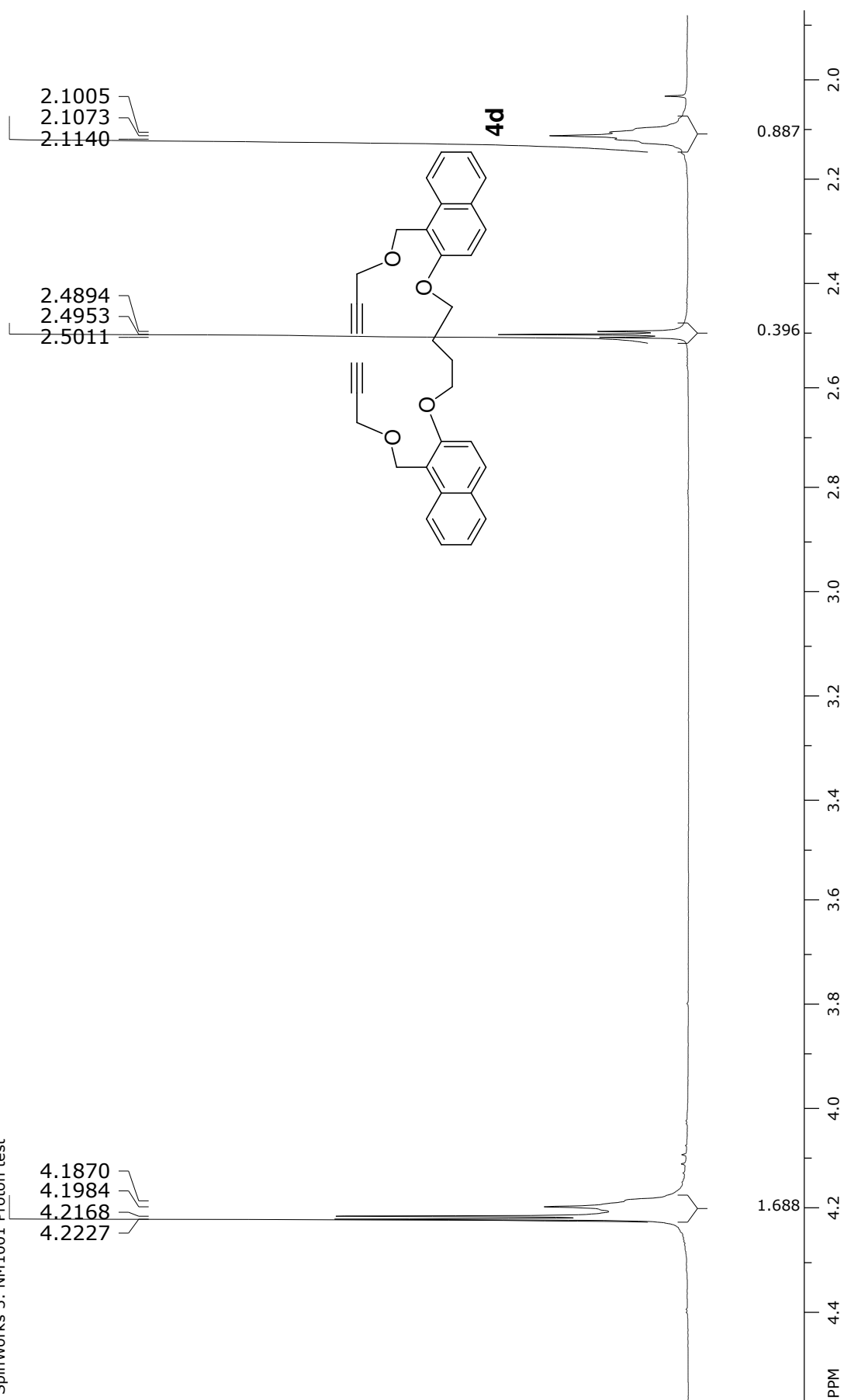


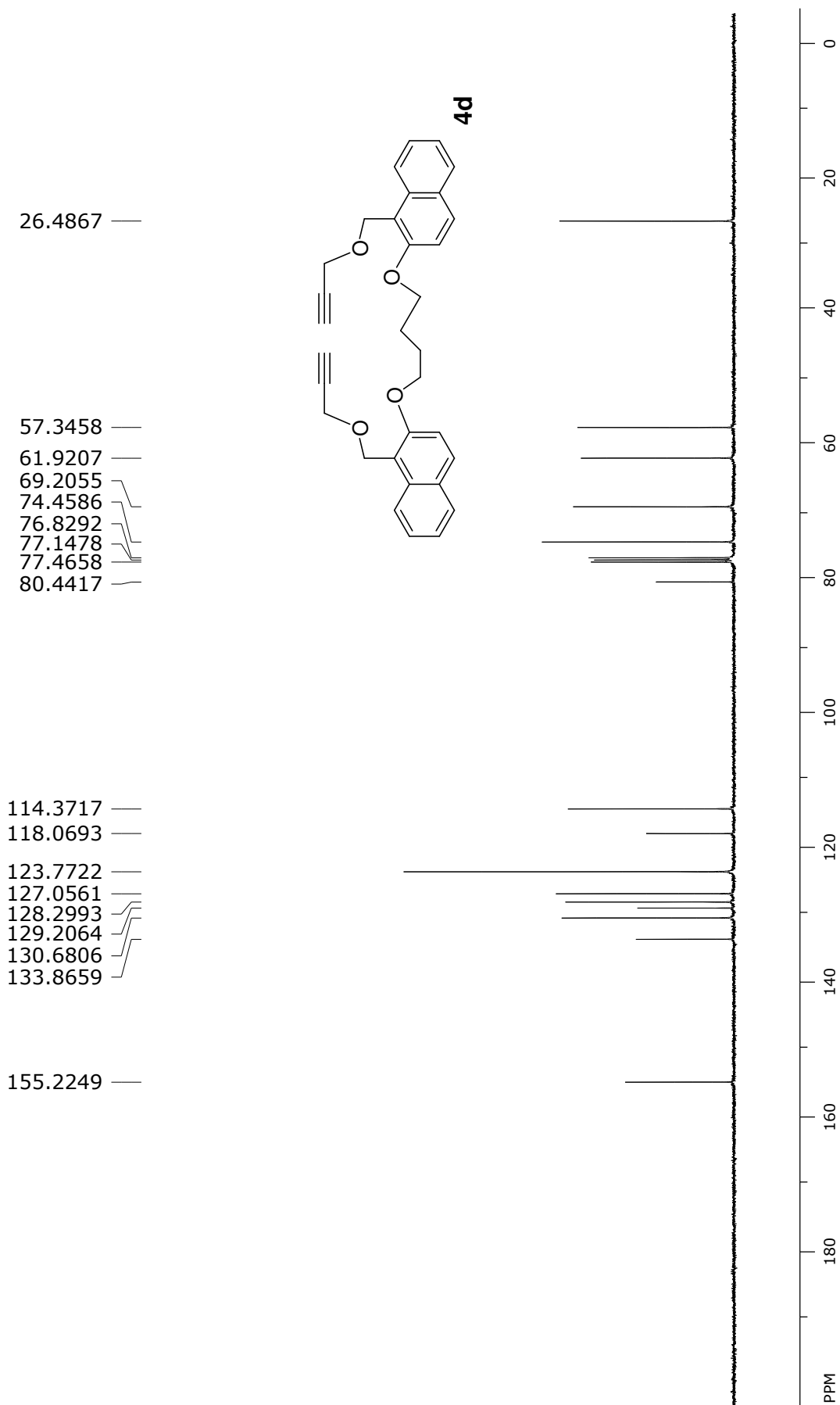


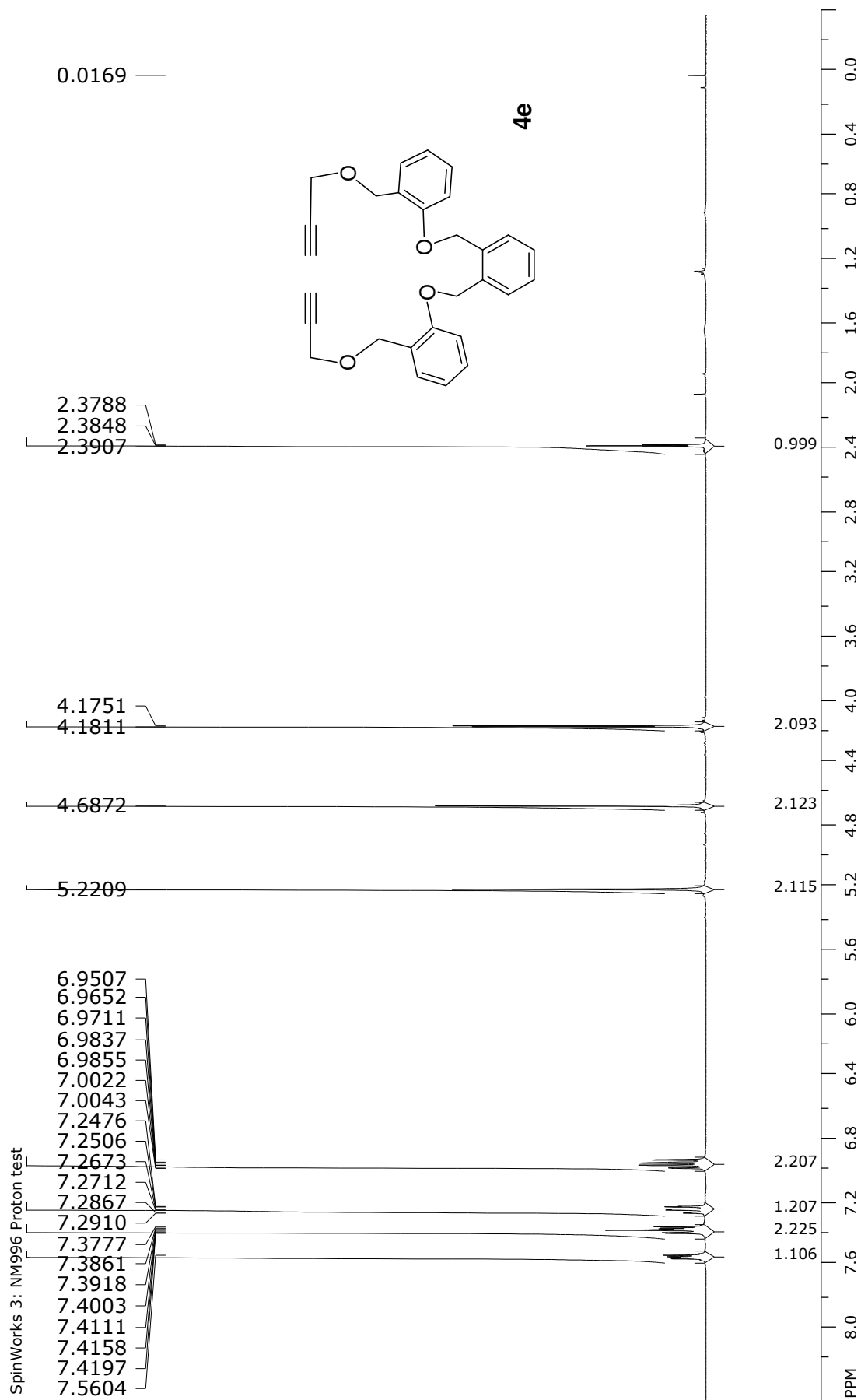
SpinWorks 3: NM1001 Proton test



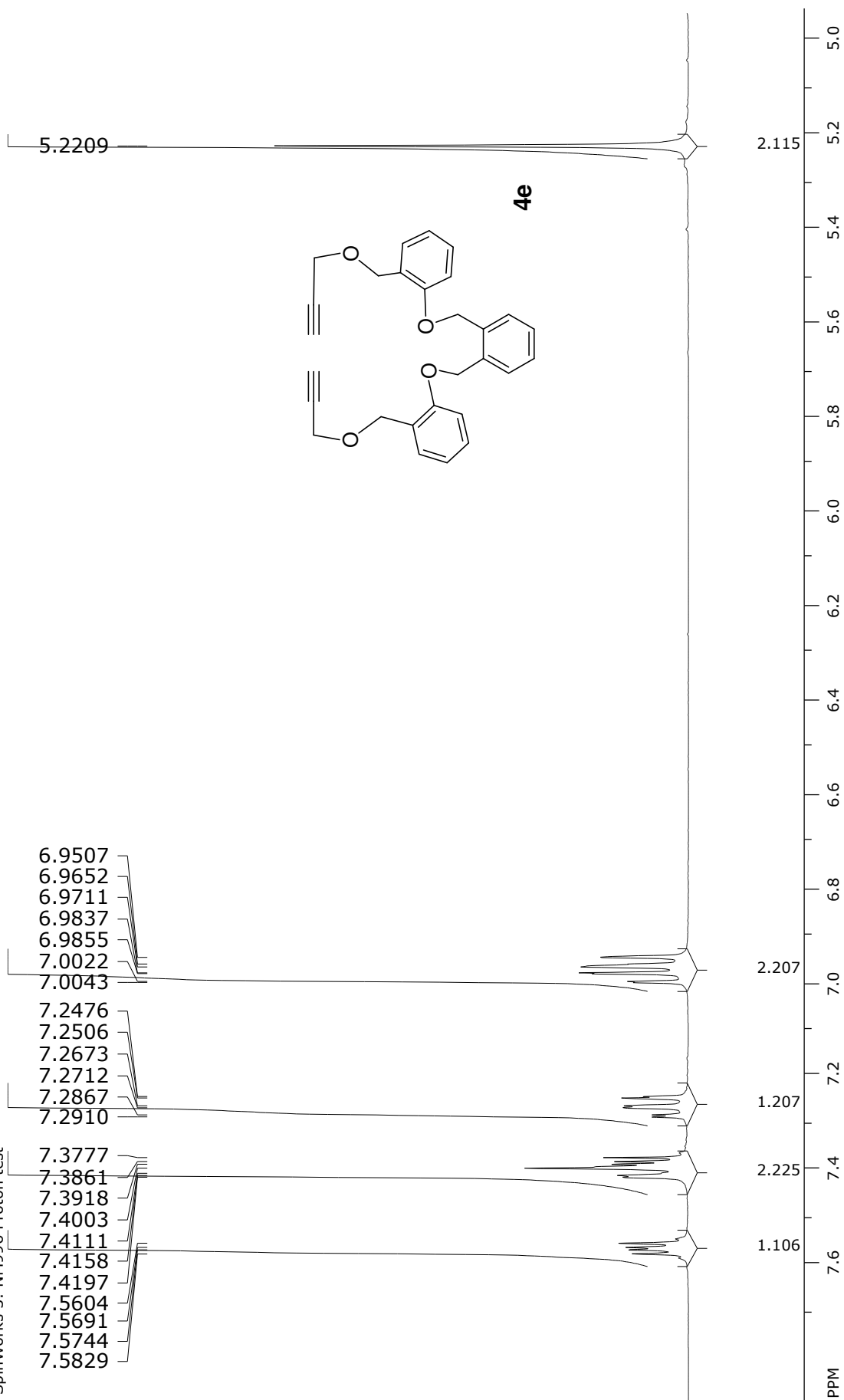
SpinWorks 3: NM1001 Proton test



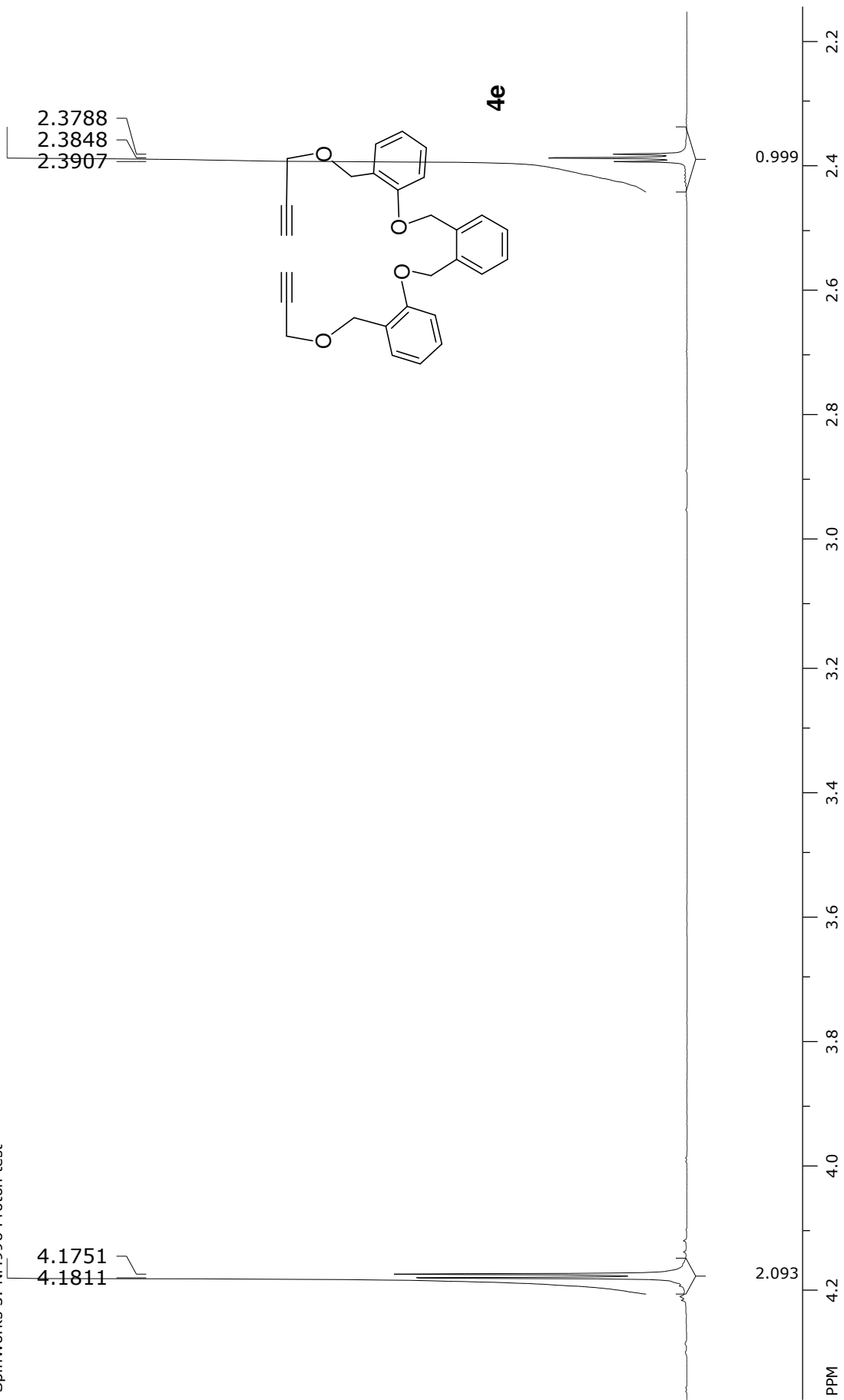


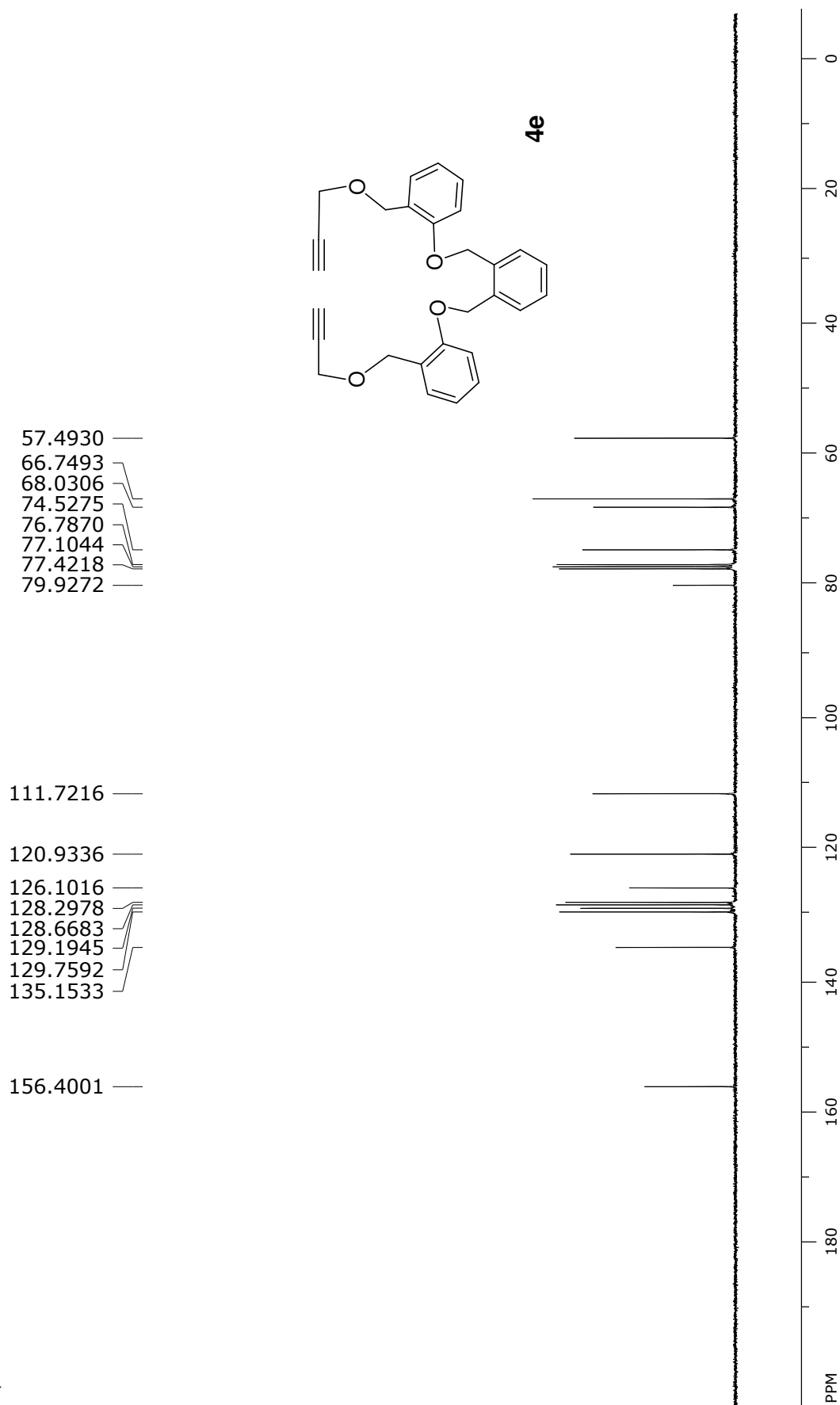


SpinWorks 3: NM996 Proton test

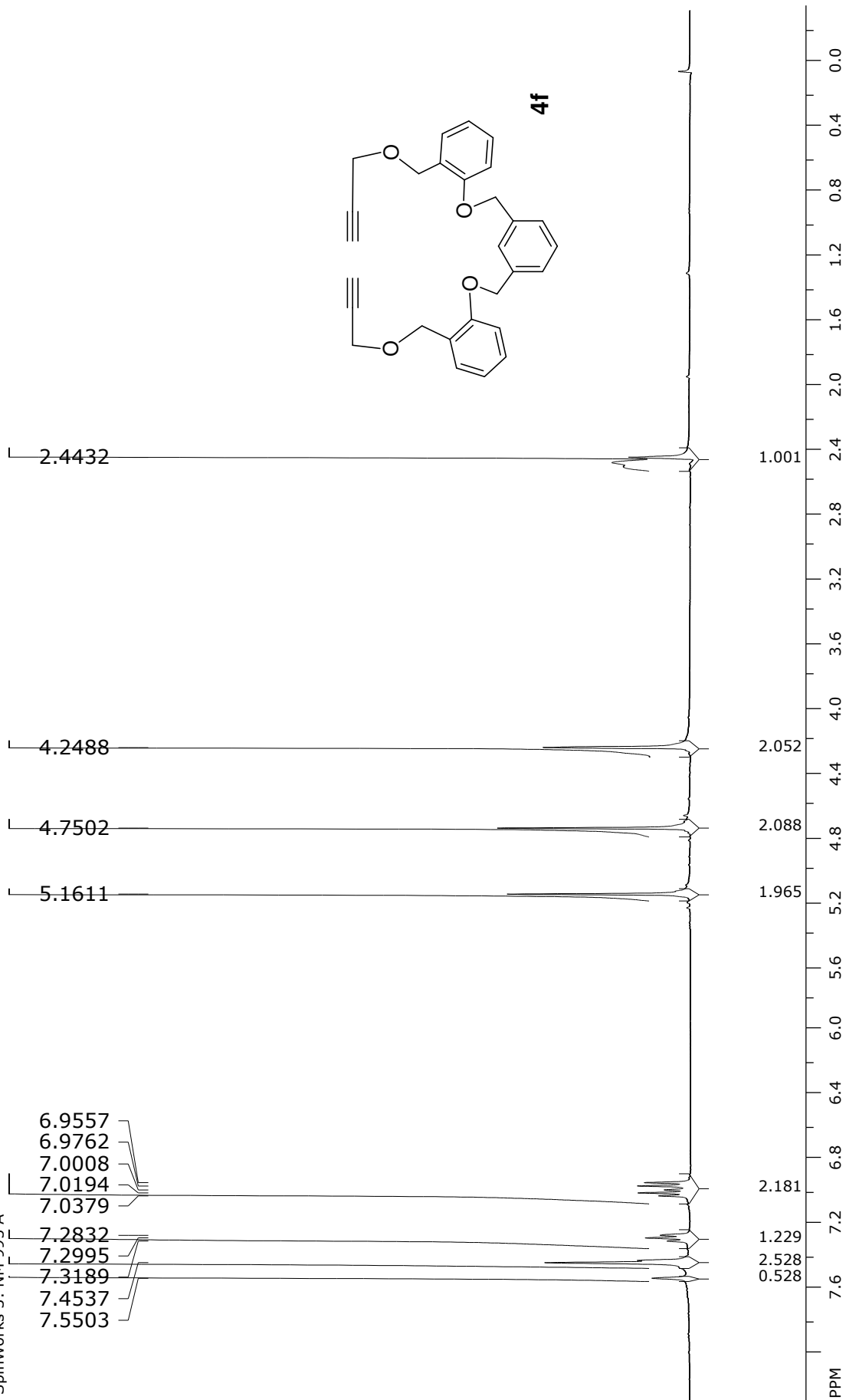


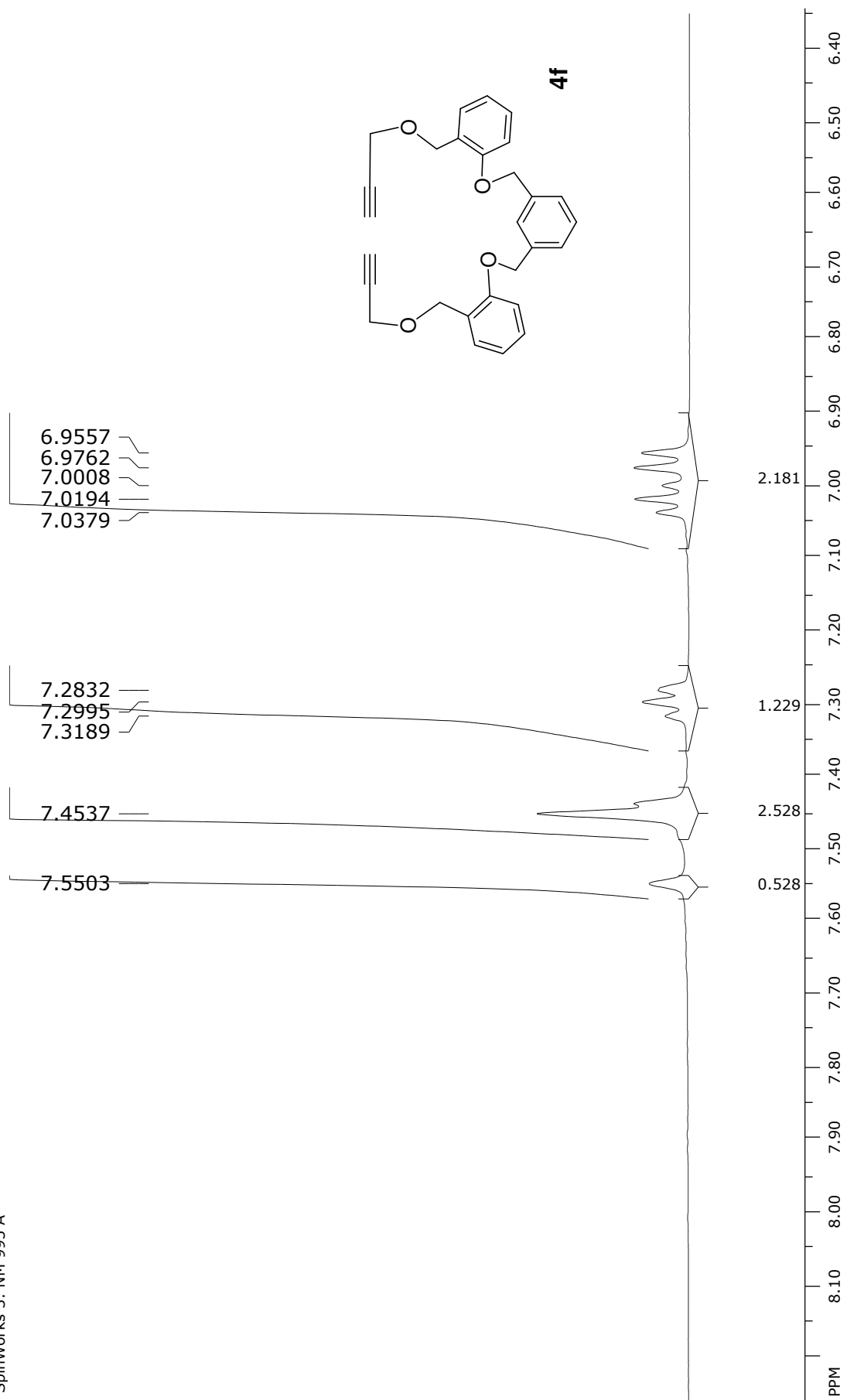
SpinWorks 3: NM996 Proton test





SpinWorks 3: NM 995 A

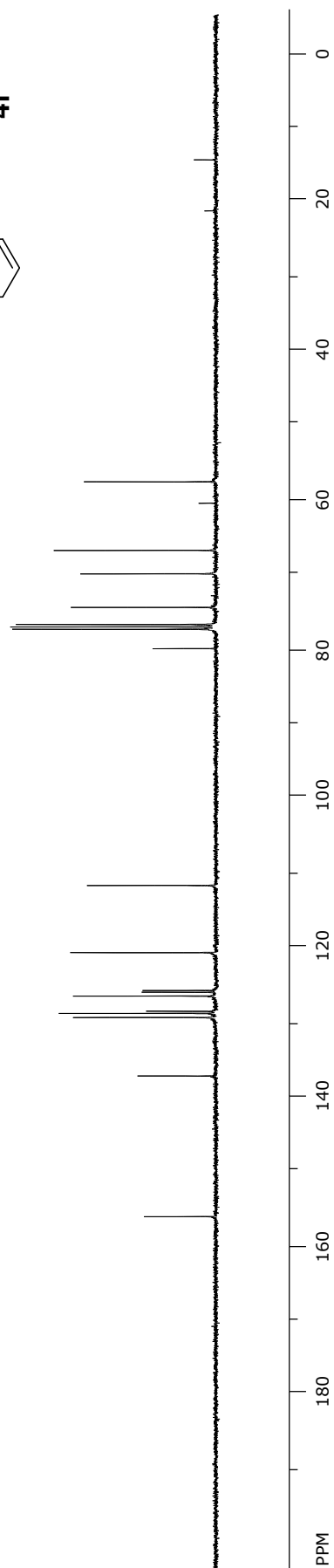
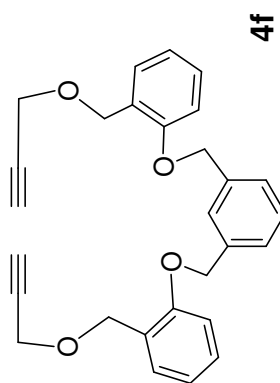




57.5668 —
66.7942 —
69.9507 —
74.4683 —
76.7741 —
77.0912 —
77.4097 —
80.0159 —

111.8839 —
120.9236 —
126.0176 —
126.2566 —
126.7659 —
128.8121 —
129.0906 —
129.6497 —
137.5205 —

156.4465 —



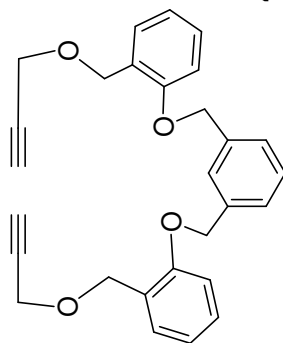
SpinWorks 3: NM995 Carbon test

128.8121 —
129.0906 —
129.6497 —

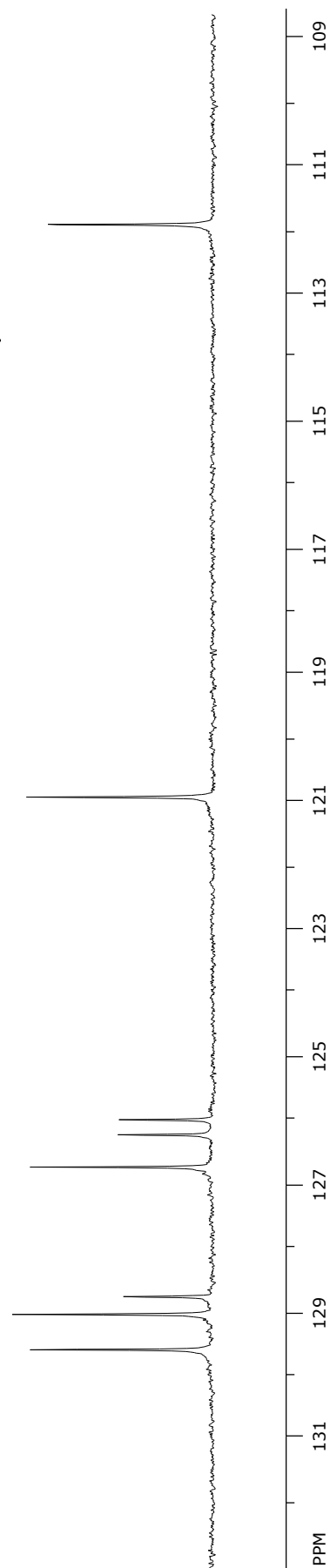
126.0176 —
126.2566 —
126.7659 —

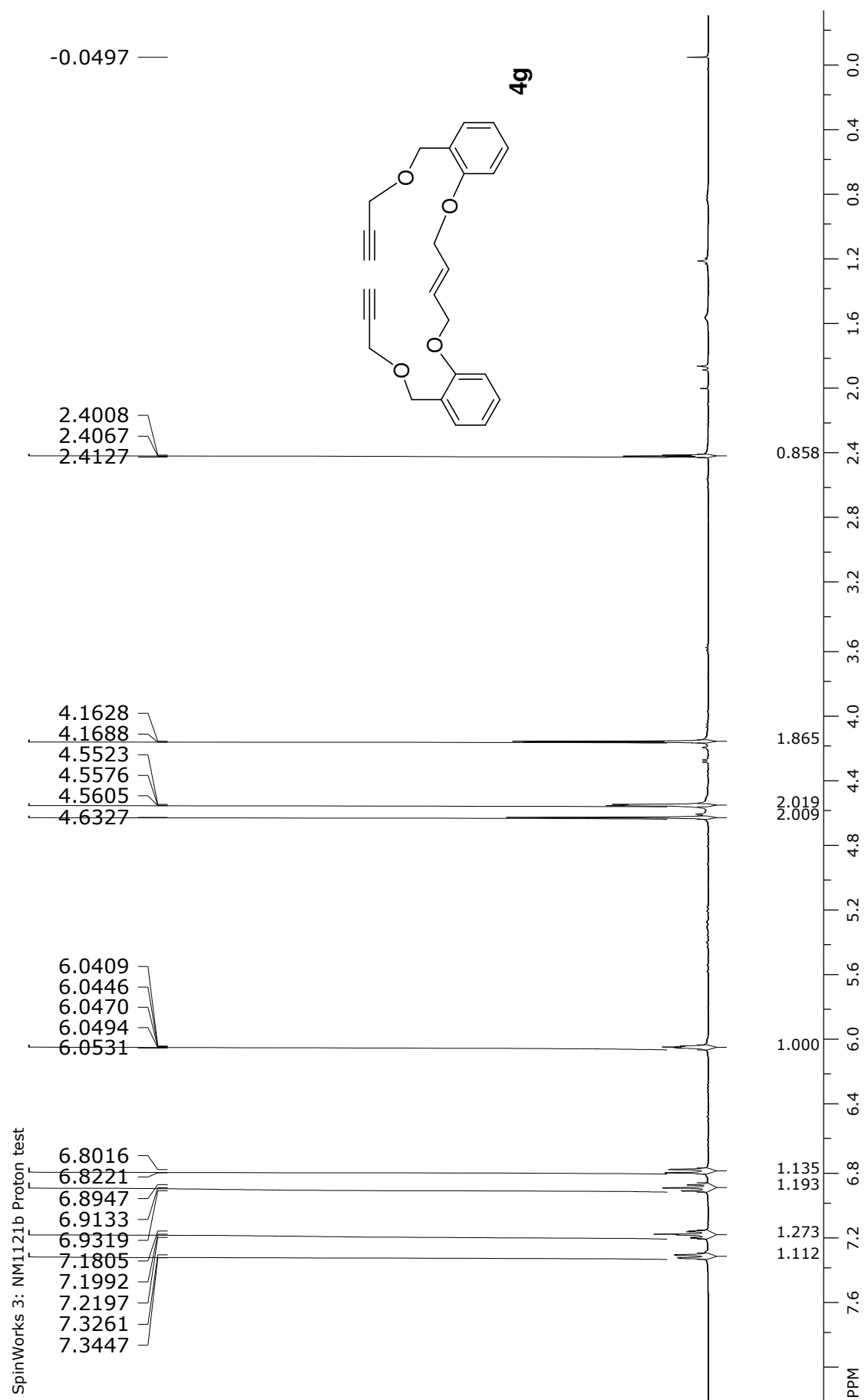
120.9236 —

111.8839 —

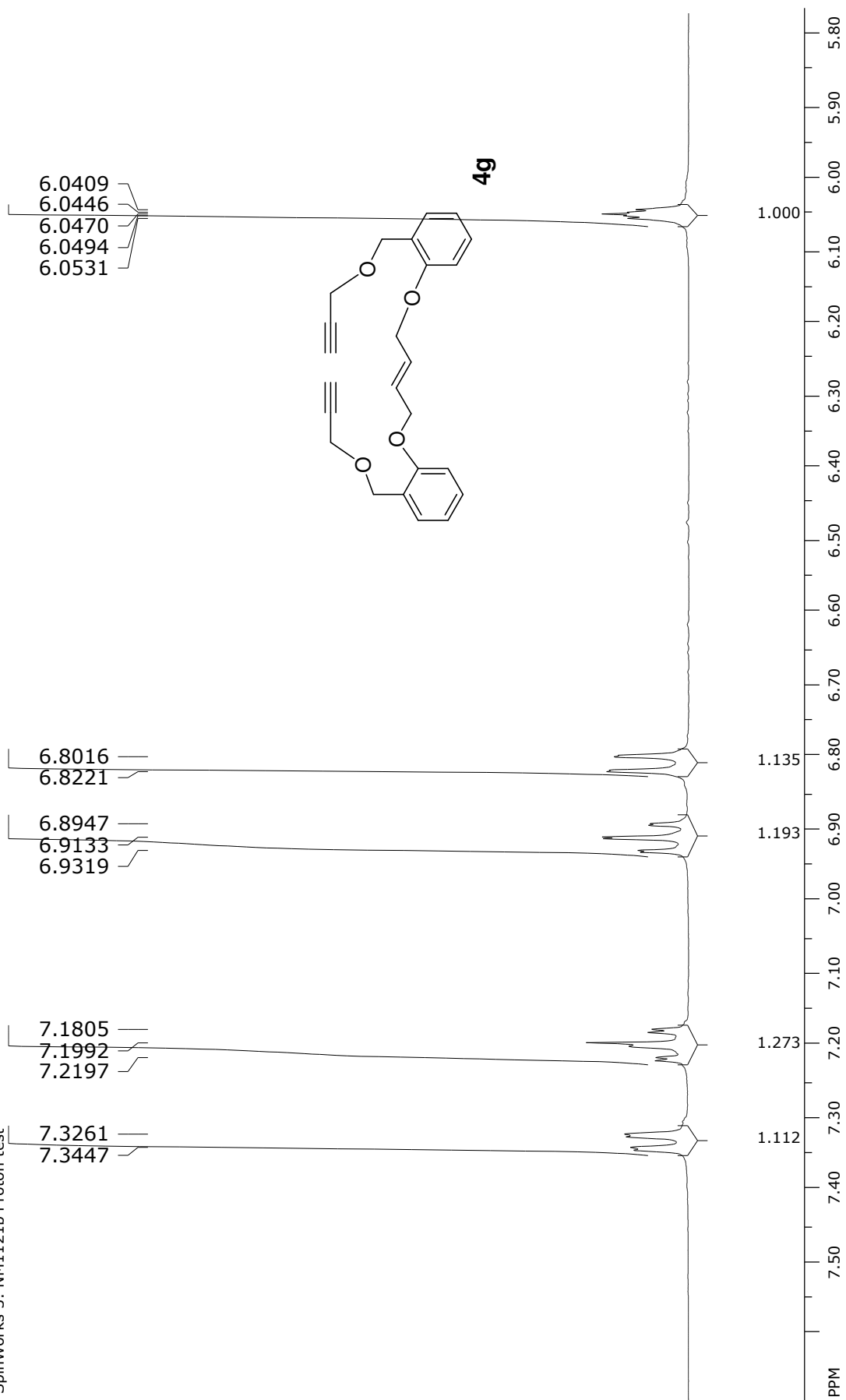


4f

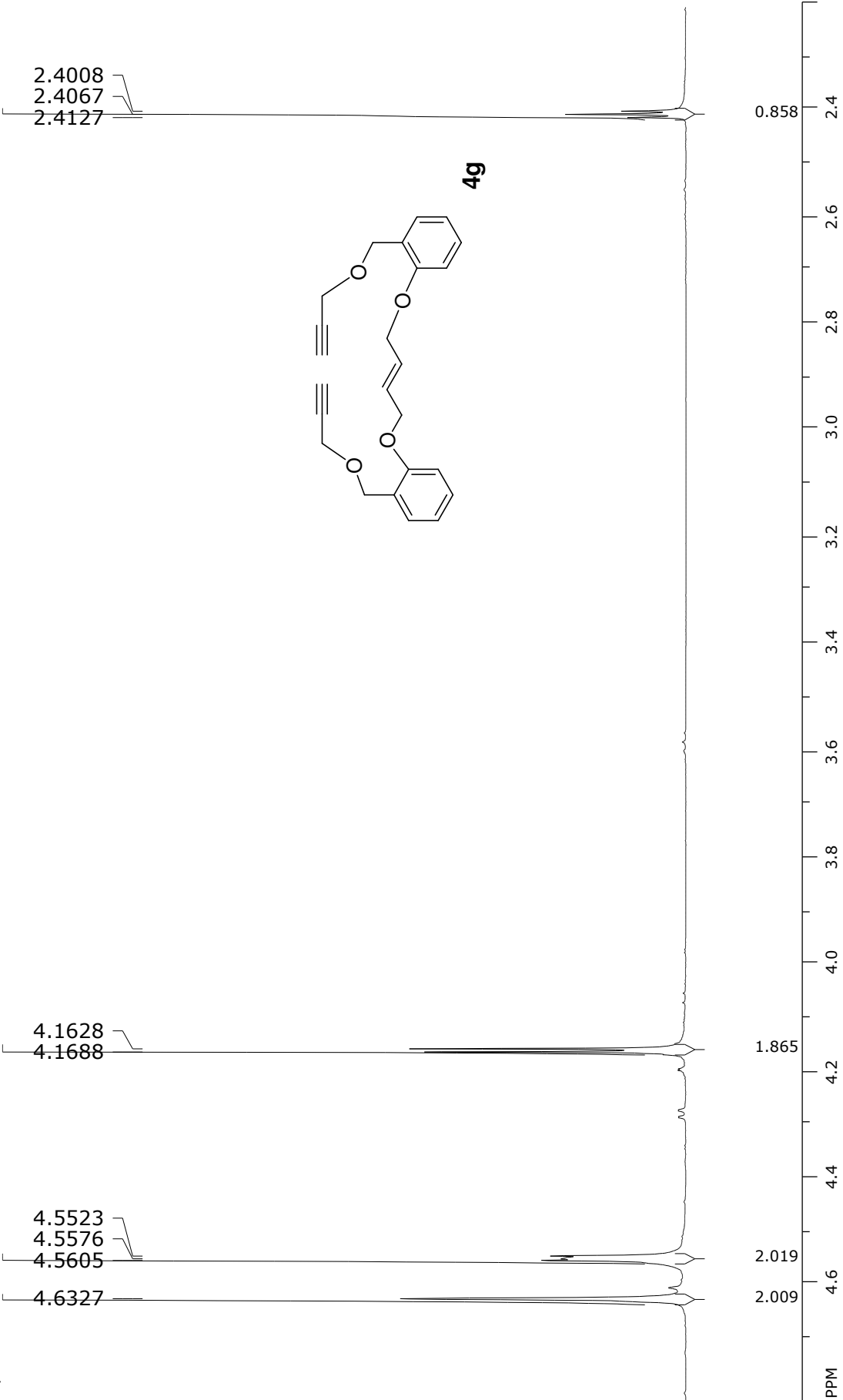




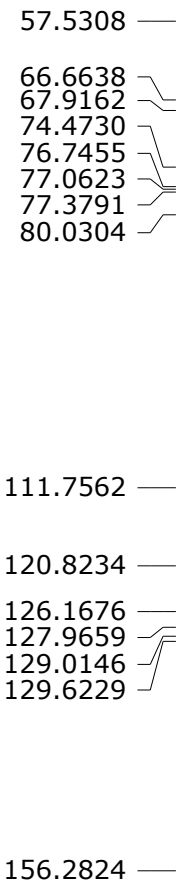
SpinWorks 3: NM1121b Proton test



SpinWorks 3: NM1121b Proton test



SpinWorks 3: Nm1121 Carbon test



SpinWorks 3: Nm1121 Carbon test

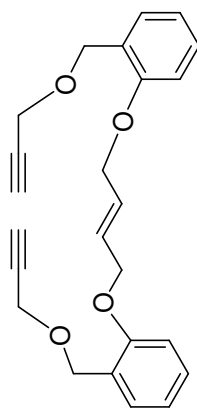
129.0146 —
129.6229 —

127.9659 —

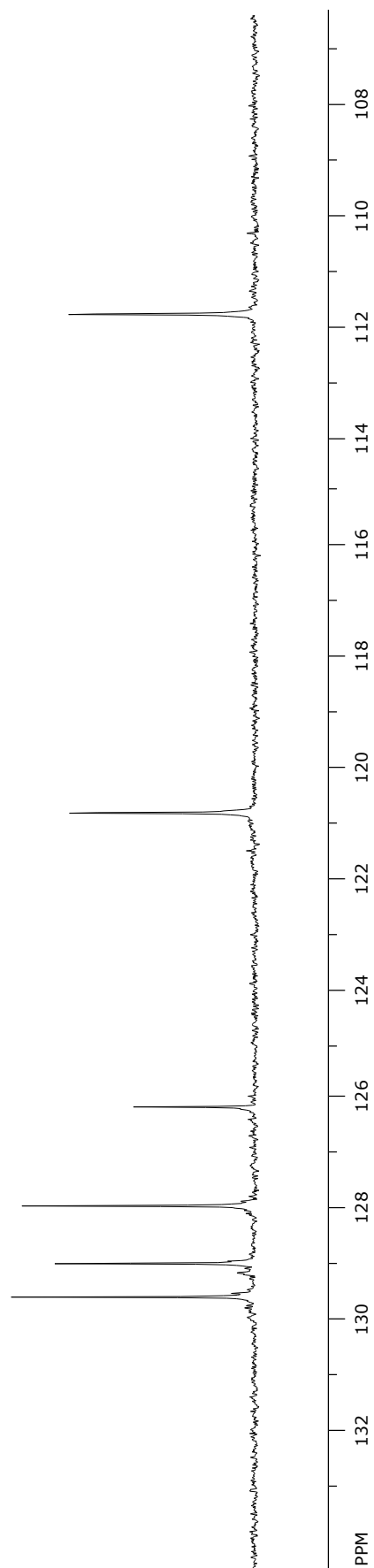
126.1676 —

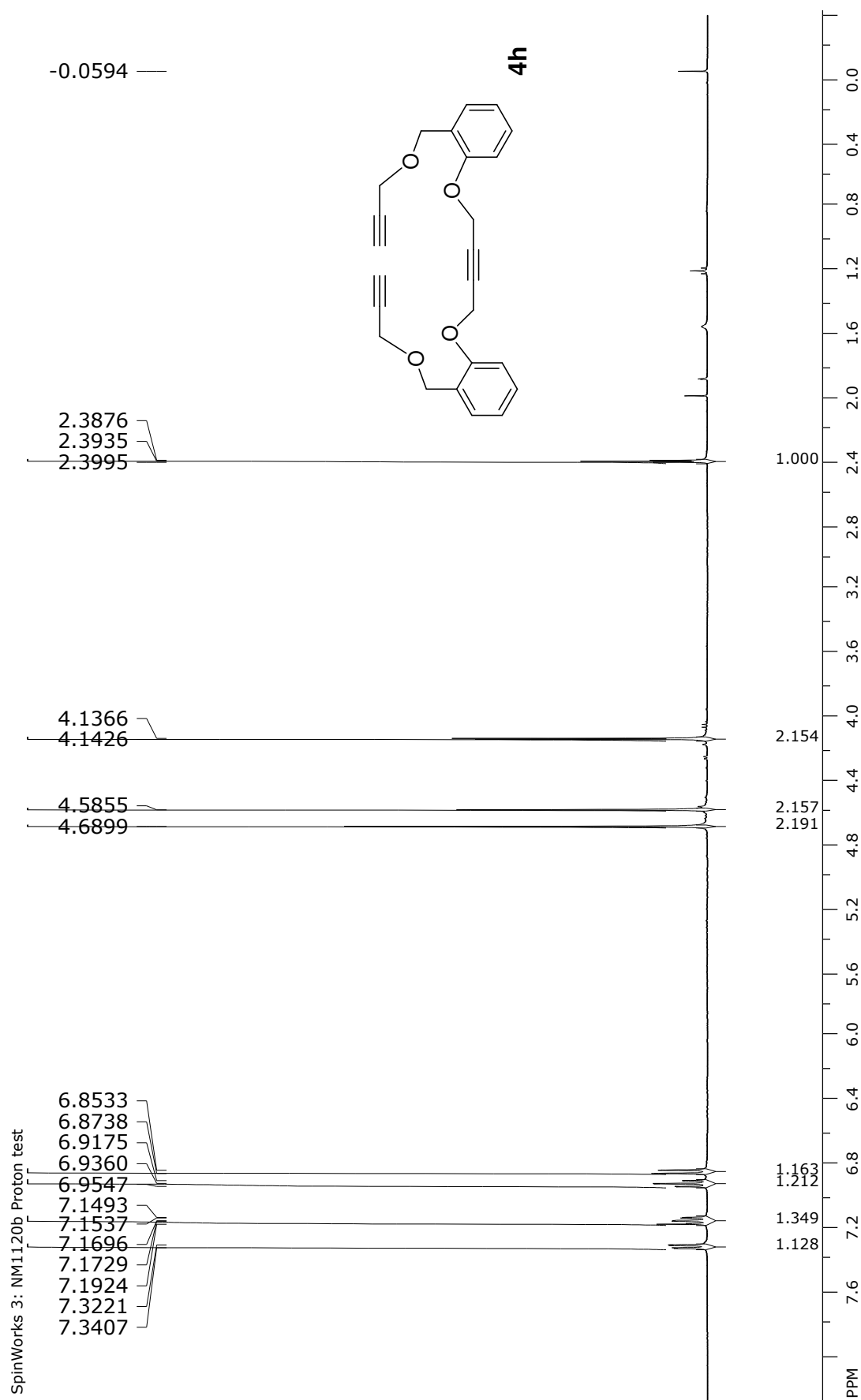
120.8234 —

111.7562 —

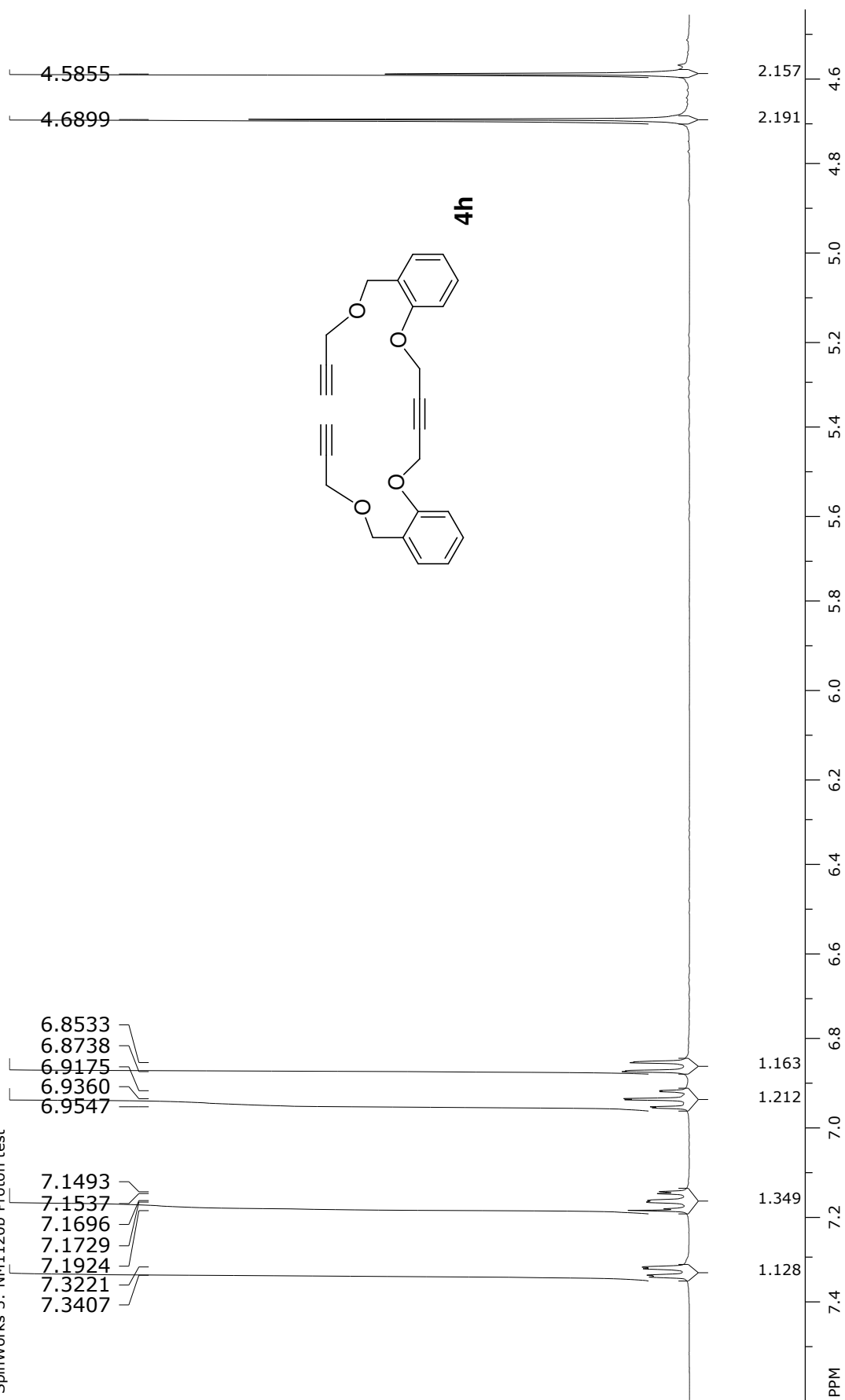


4g

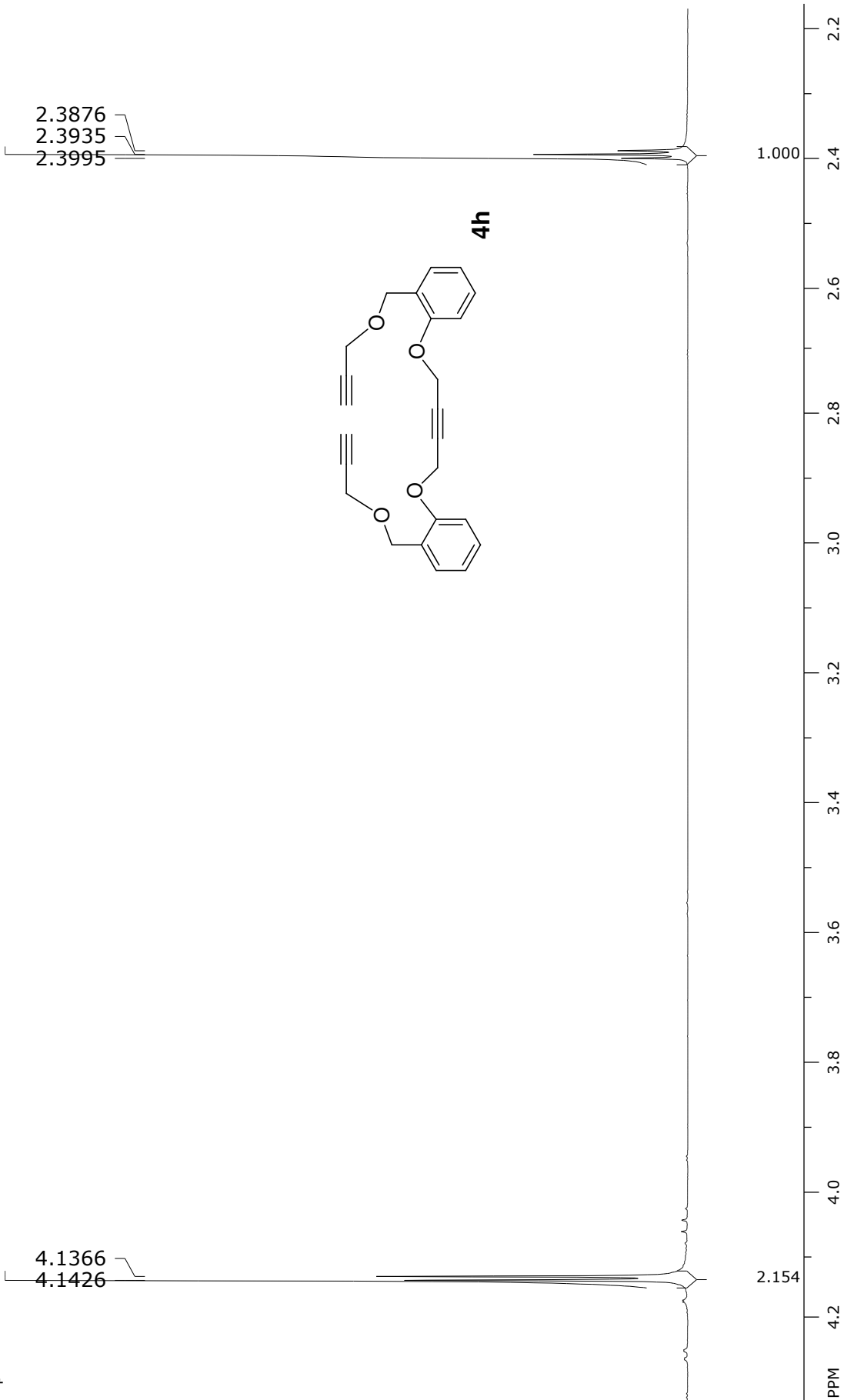


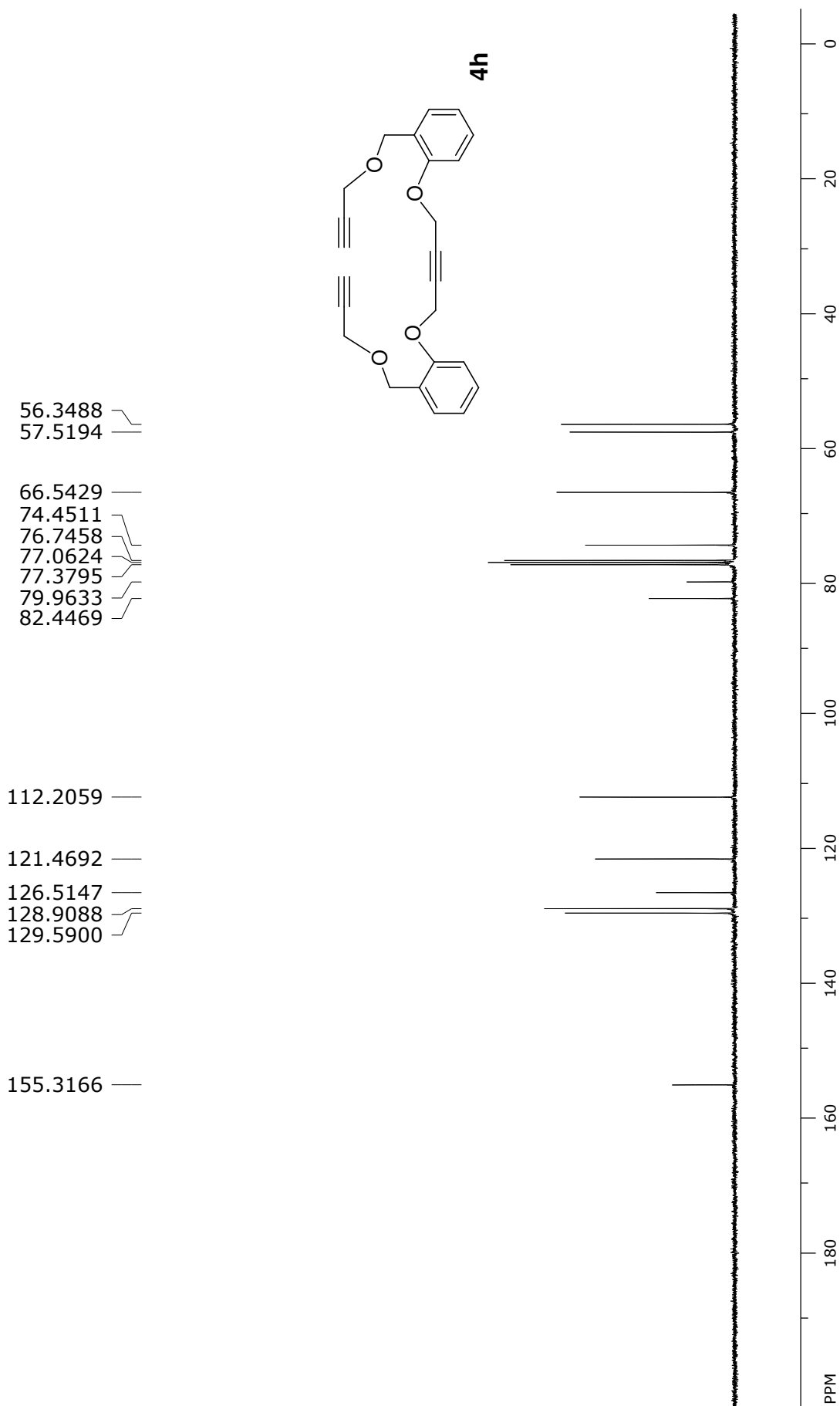


SpinWorks 3: NM1120b Proton test

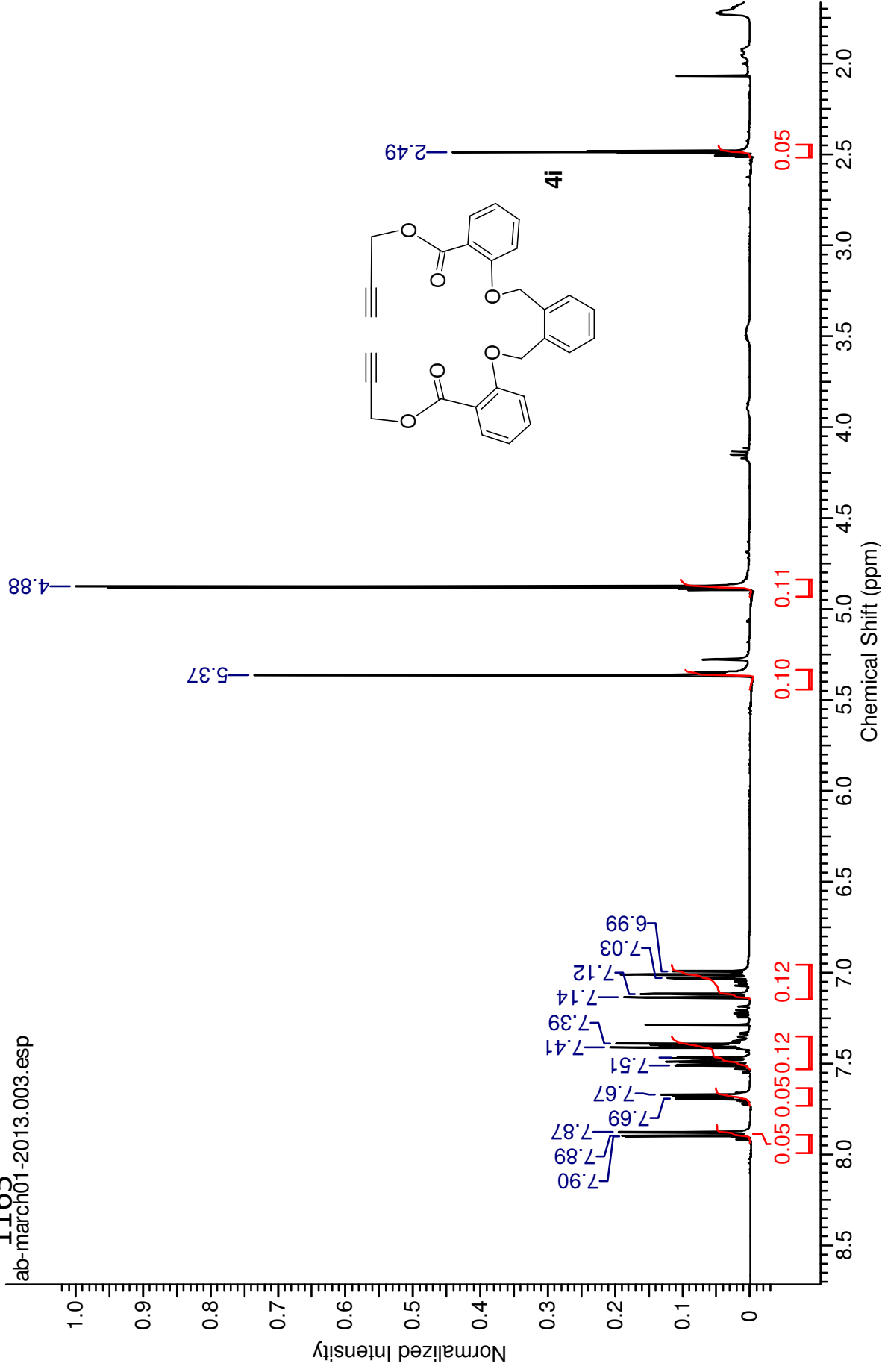


SpinWorks 3: NM1120b Proton test





1165
ab-march01-2013.003.esp



52.3117 —

68.9607
75.0448
76.7700
77.0871
77.4056
77.7875

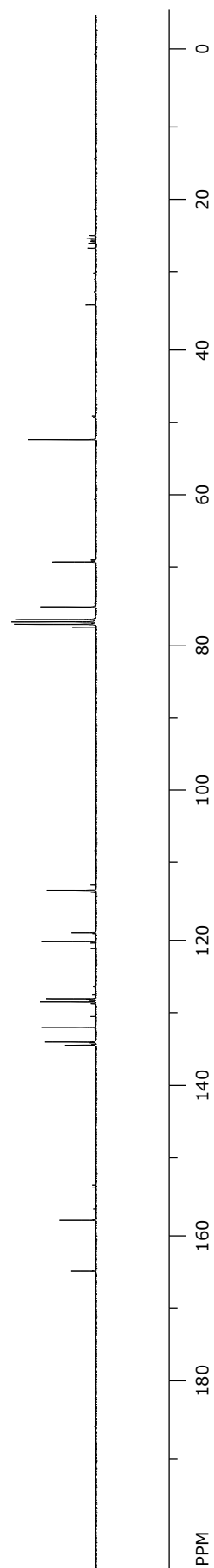
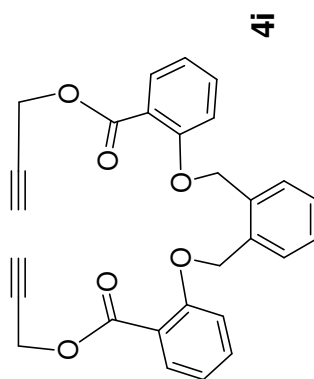
113.5111 —

119.2674 —
120.4822 —

128.2951 —
128.6199 —
132.1661 —
134.1165 —
134.5446 —

158.3328 —

165.2169 —



SpinWorks 3: nm1165a

113.5111 —

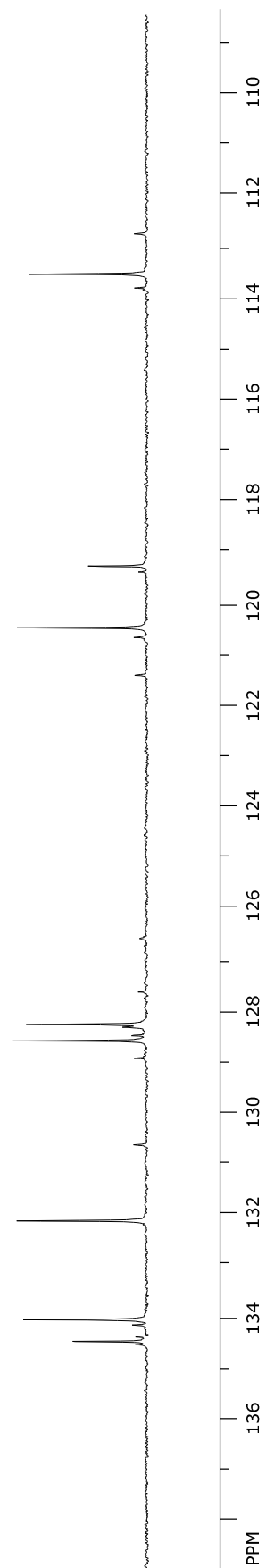
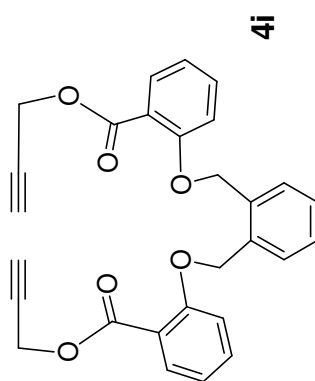
119.2674 —

120.4822 —

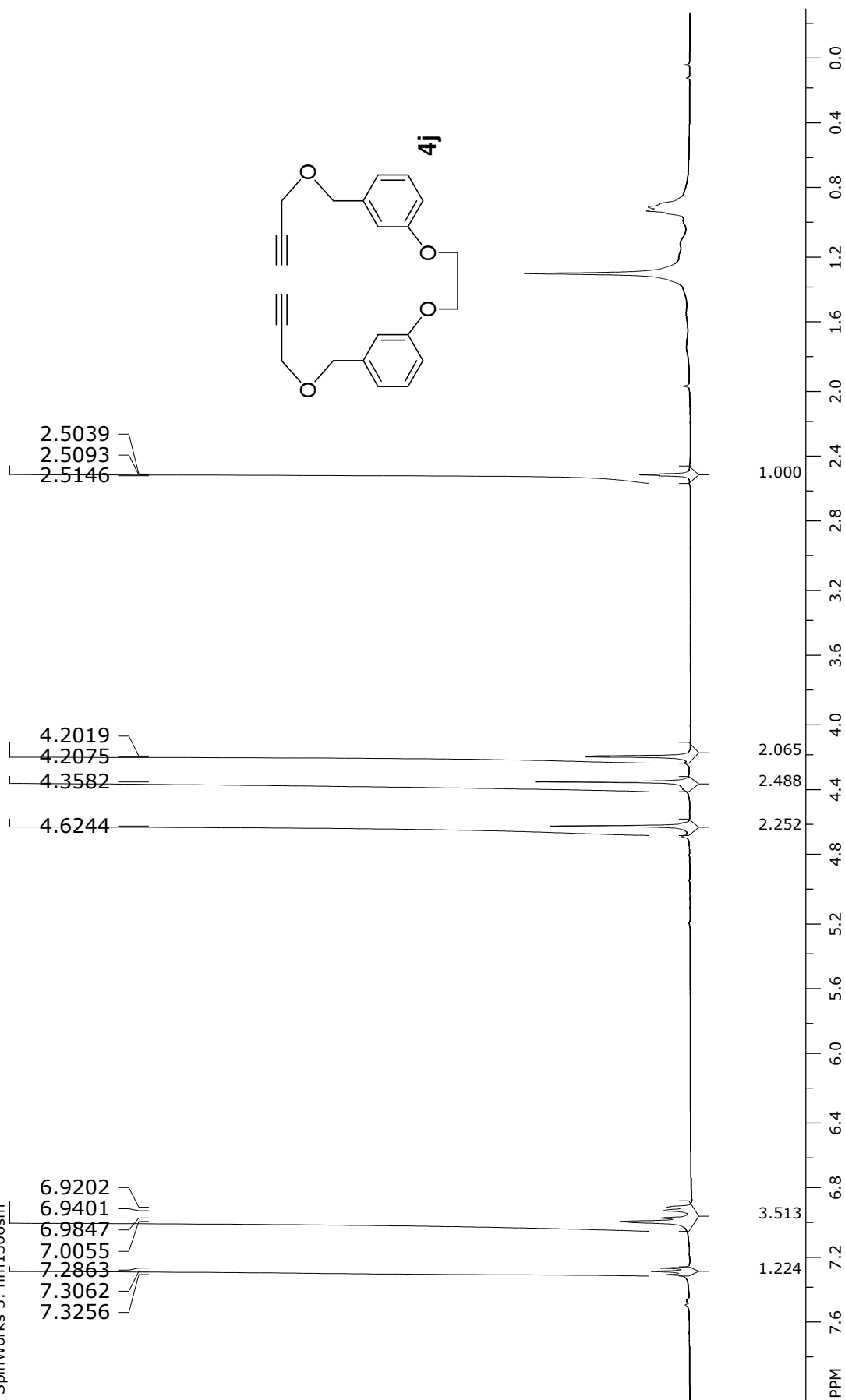
128.2951 —
128.6199 —

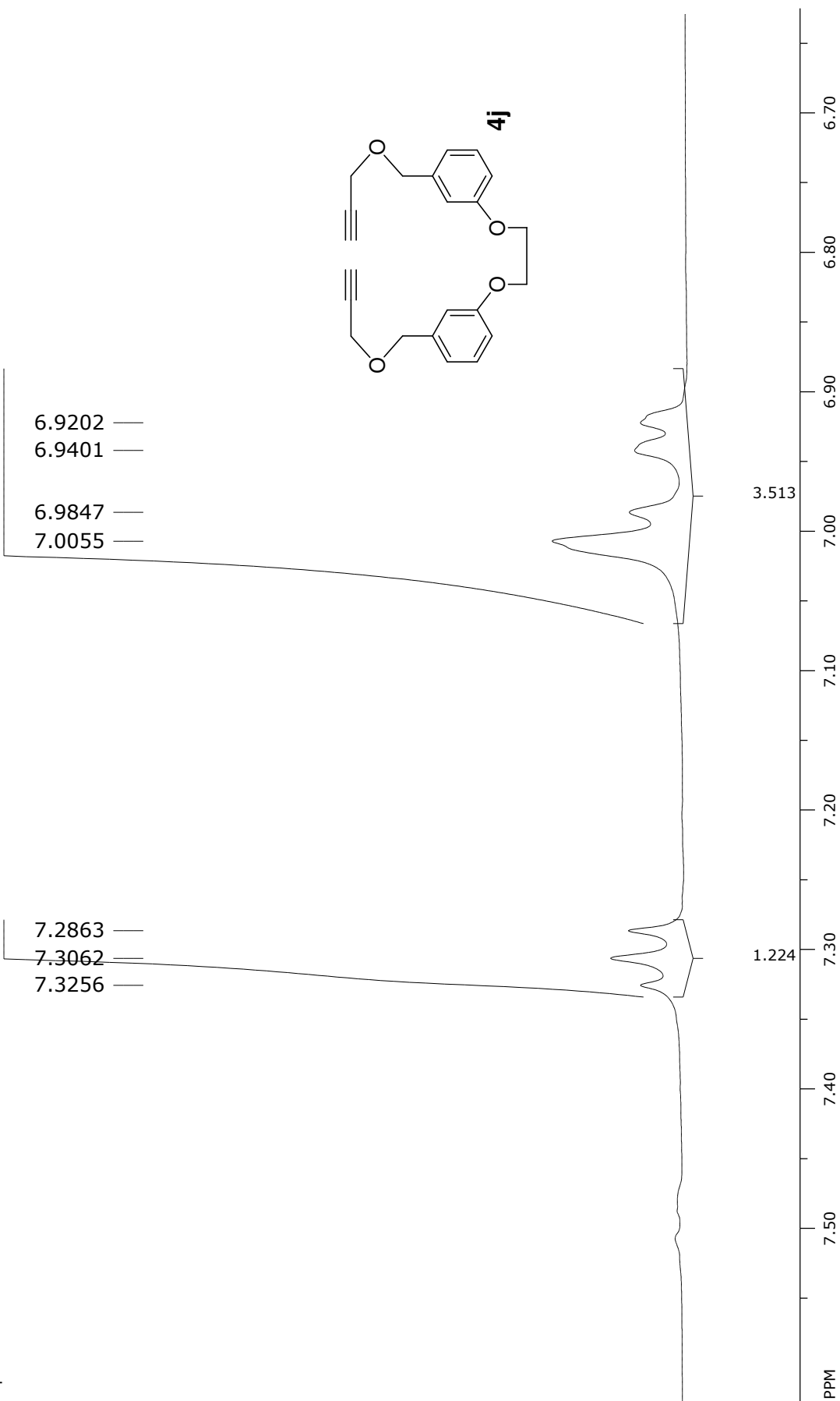
132.1661 —

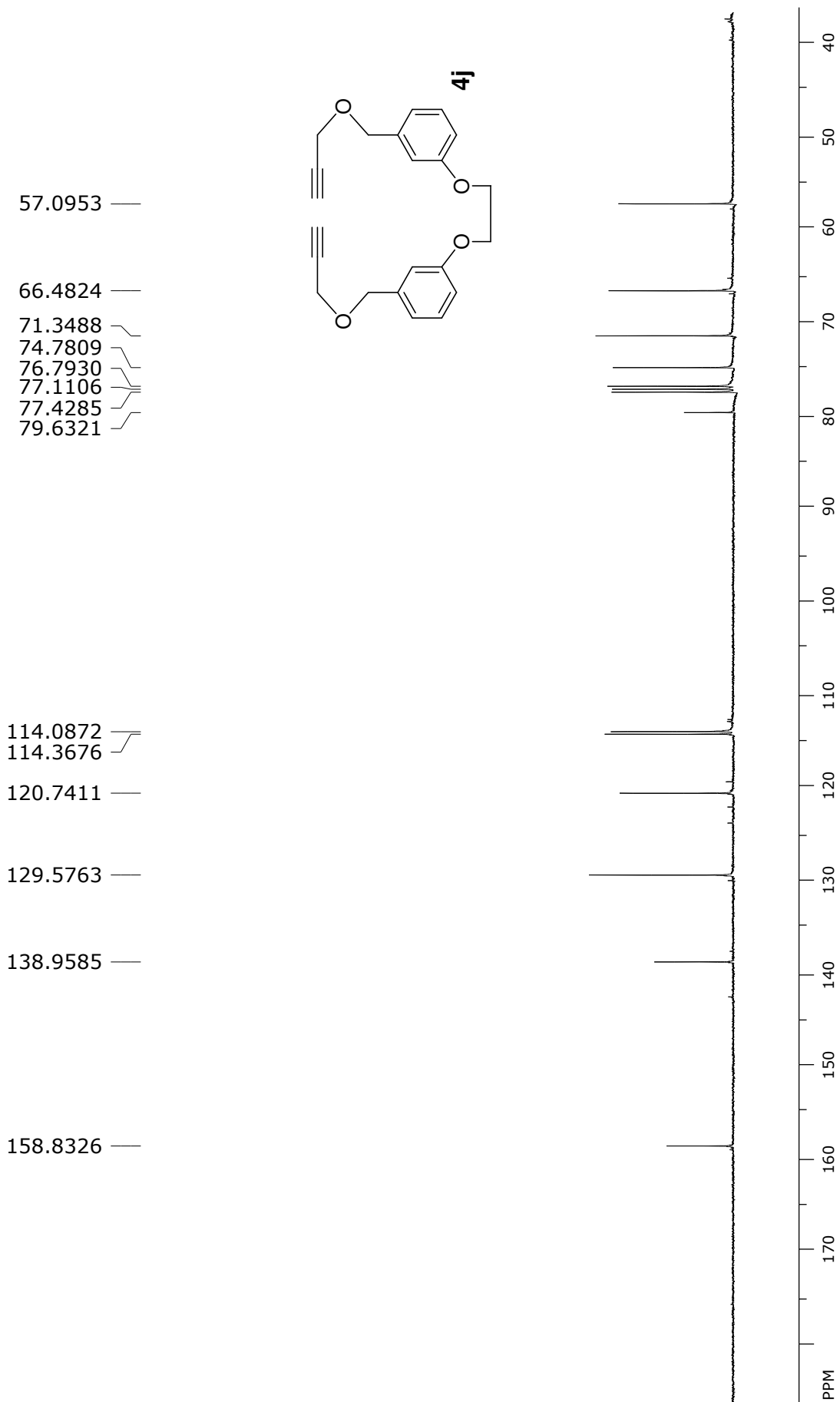
134.1165 —
134.5446 —



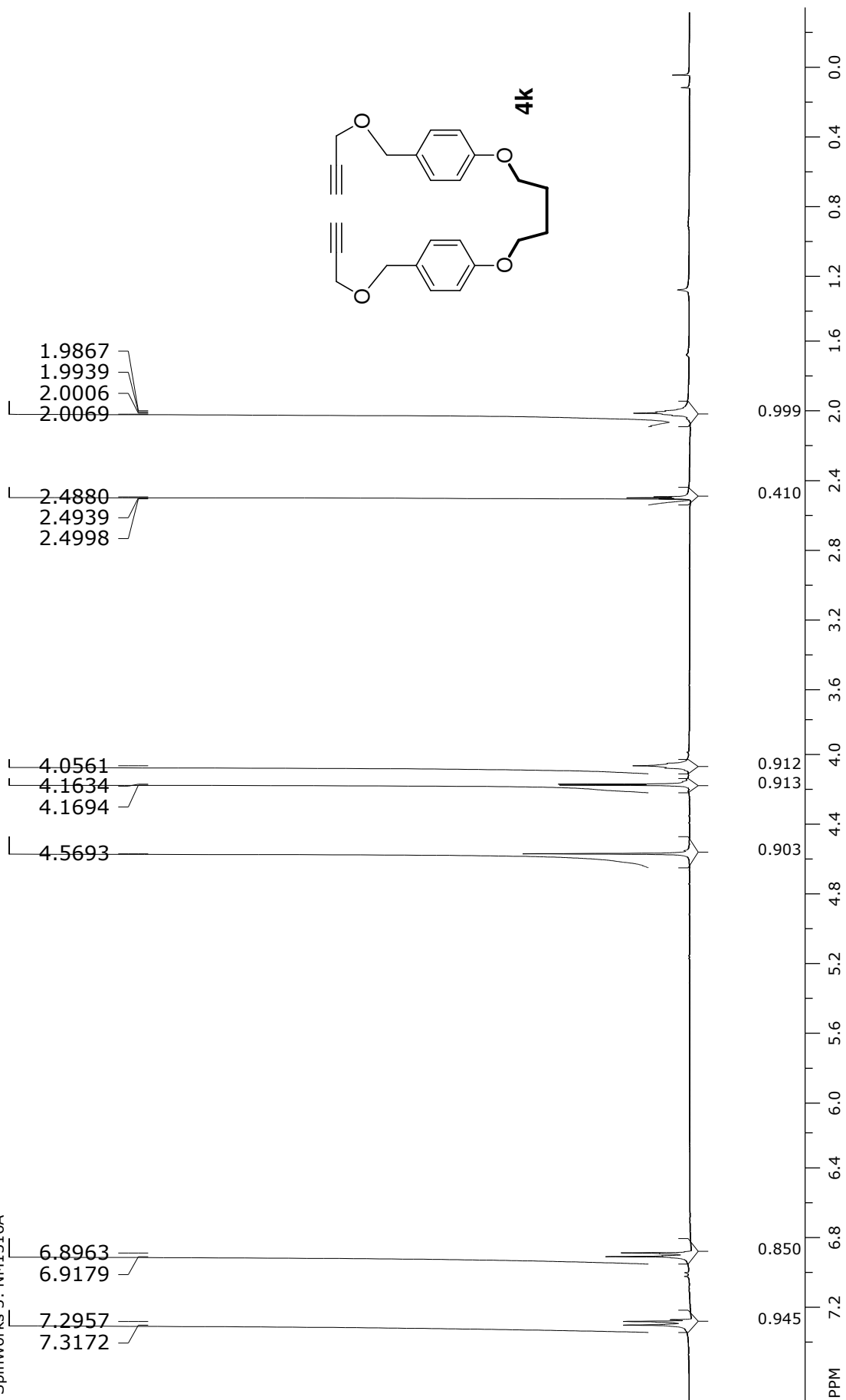
SpinWorks 3: nm1306sm

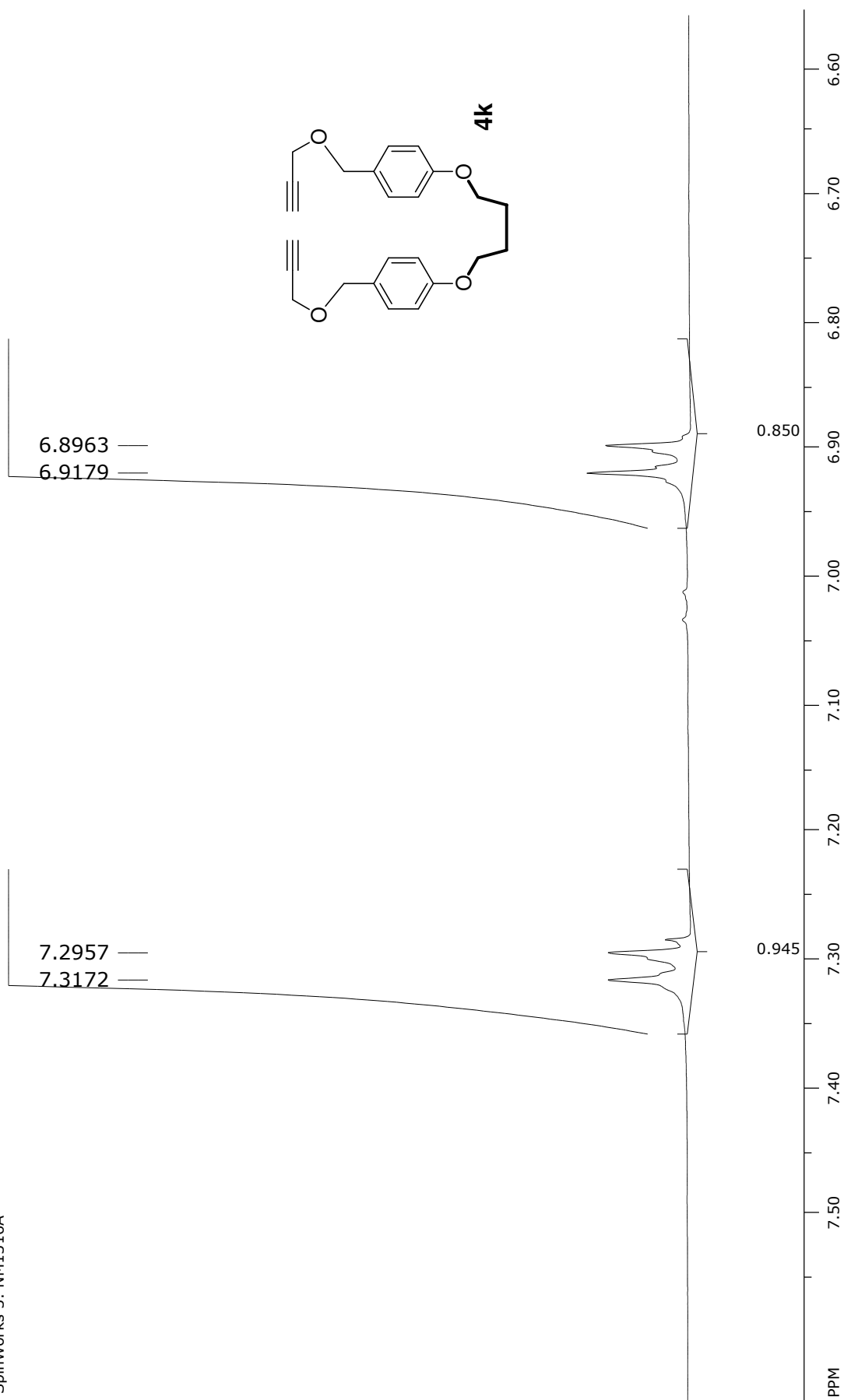




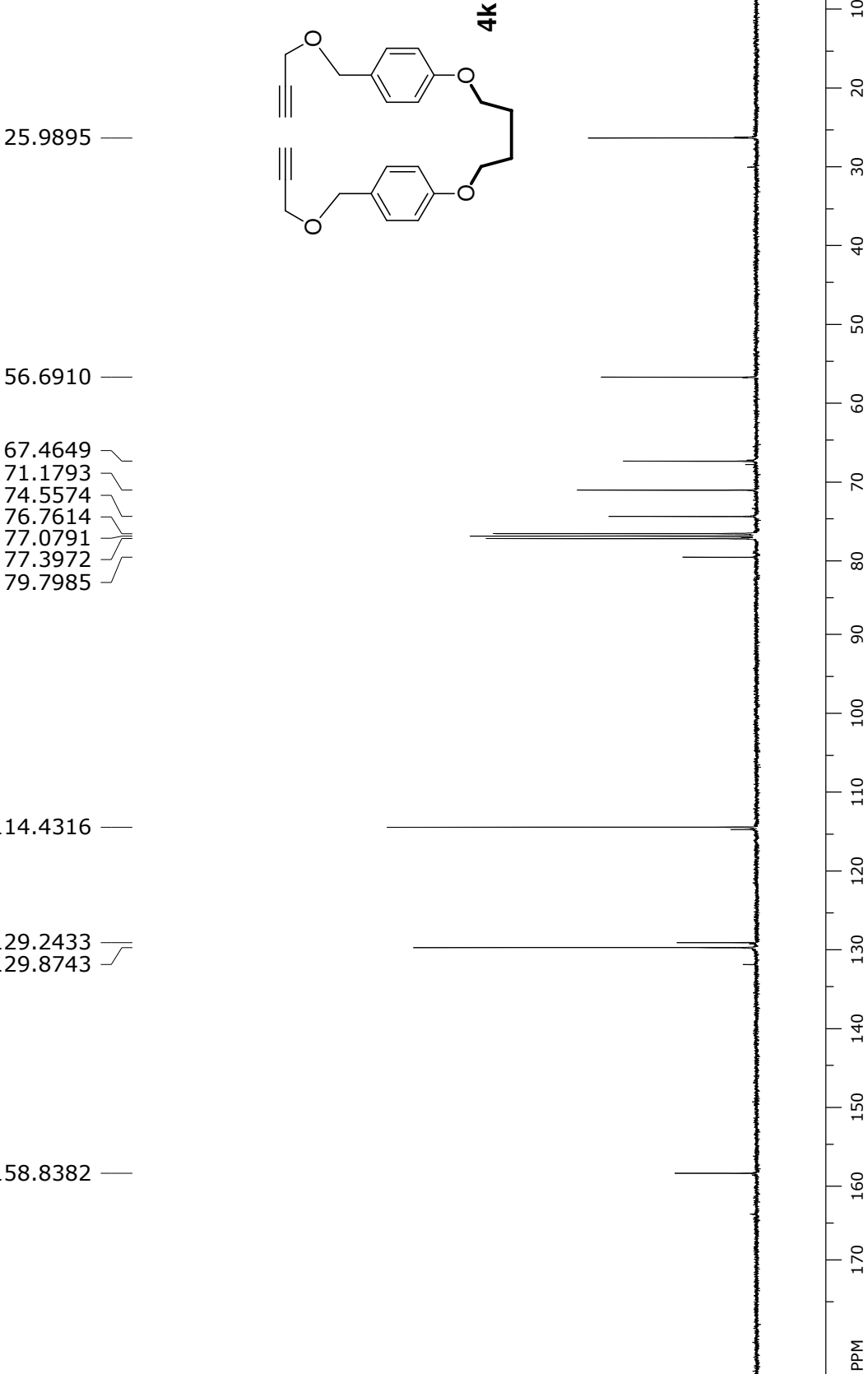


SpinWorks 3: NM1316A

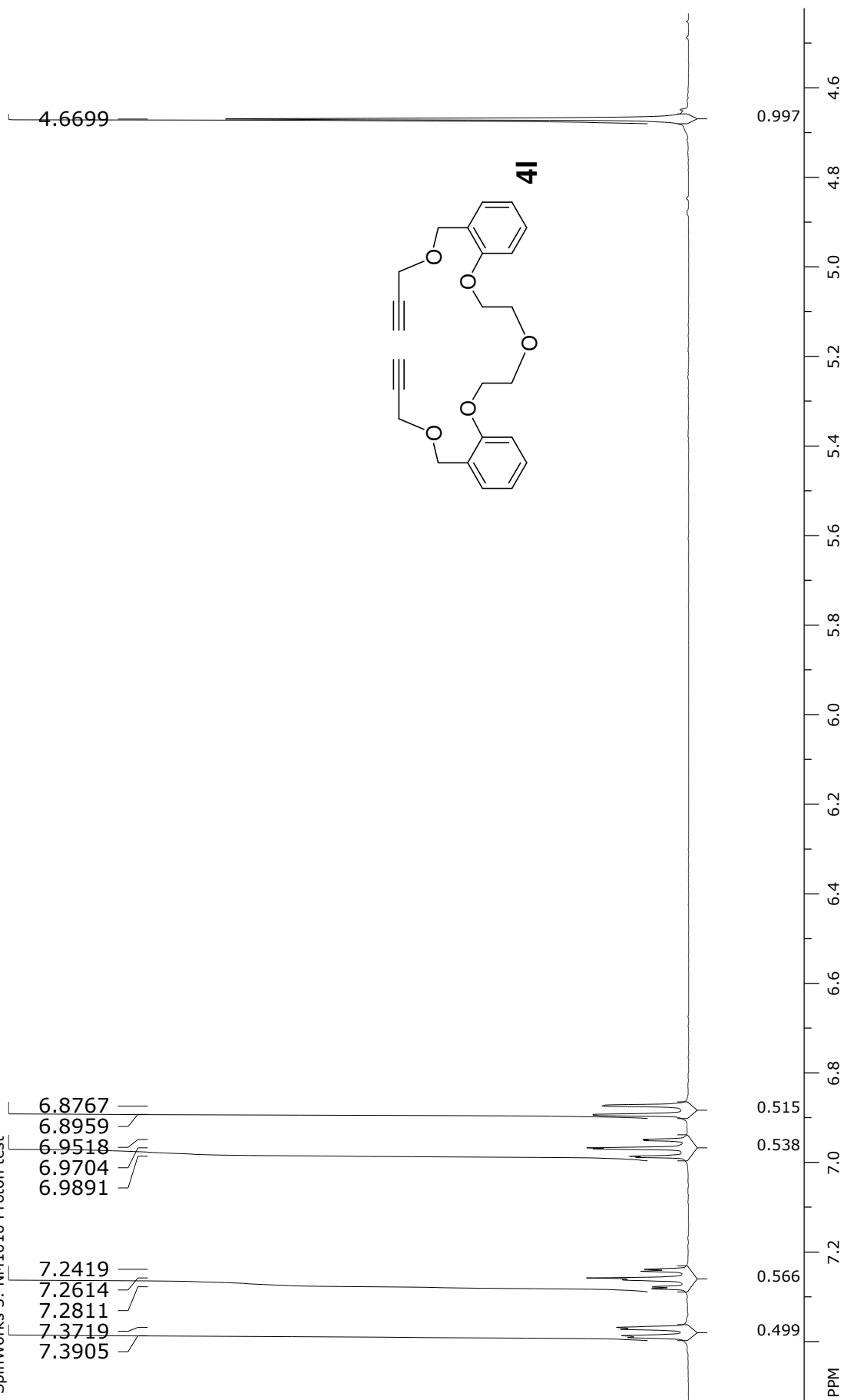




SpinWorks 3: NM1316A



SpinWorks 3: NM1010 Proton test

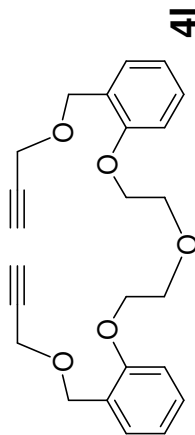


SpinWorks 3: NM1010 Proton test

2.4346
2.4405
2.4465

3.9595
3.9711
3.9741
3.9837

4.1704
4.1831
4.1947
4.2016
4.2075



0.455

1.025

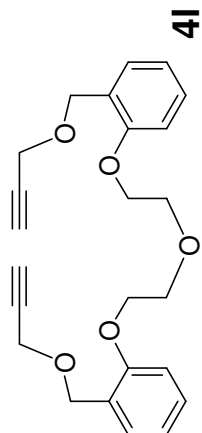
2.057

PPM

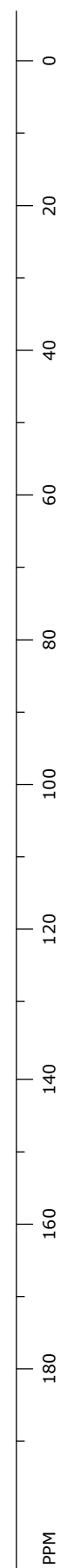
57.5204 —
 66.6774 —
 68.0356 —
 70.0268 —
 74.3808 —
 76.7788 —
 77.0962 —
 77.4136 —
 80.1055 —

111.6543 —
 120.8794 —
 126.1723 —
 129.0535 —
 129.5740 —

156.5432 —



4l



SpinWorks 3: NM1010 Carbon test

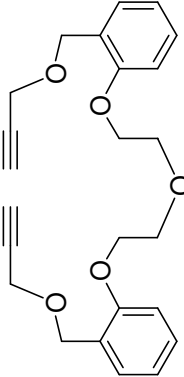
156.5432 —

126.1723 —

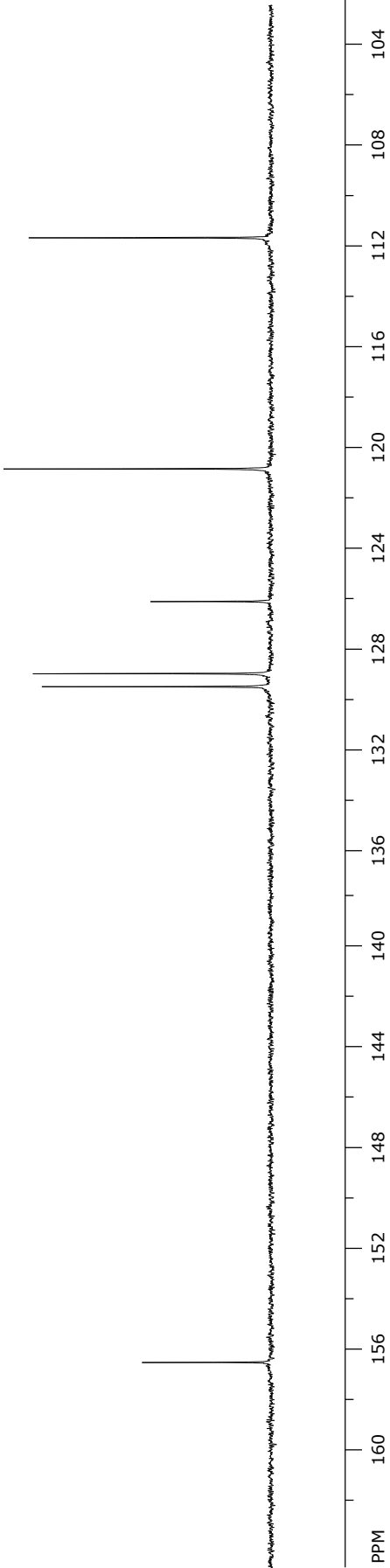
129.0535 —
129.5740 —

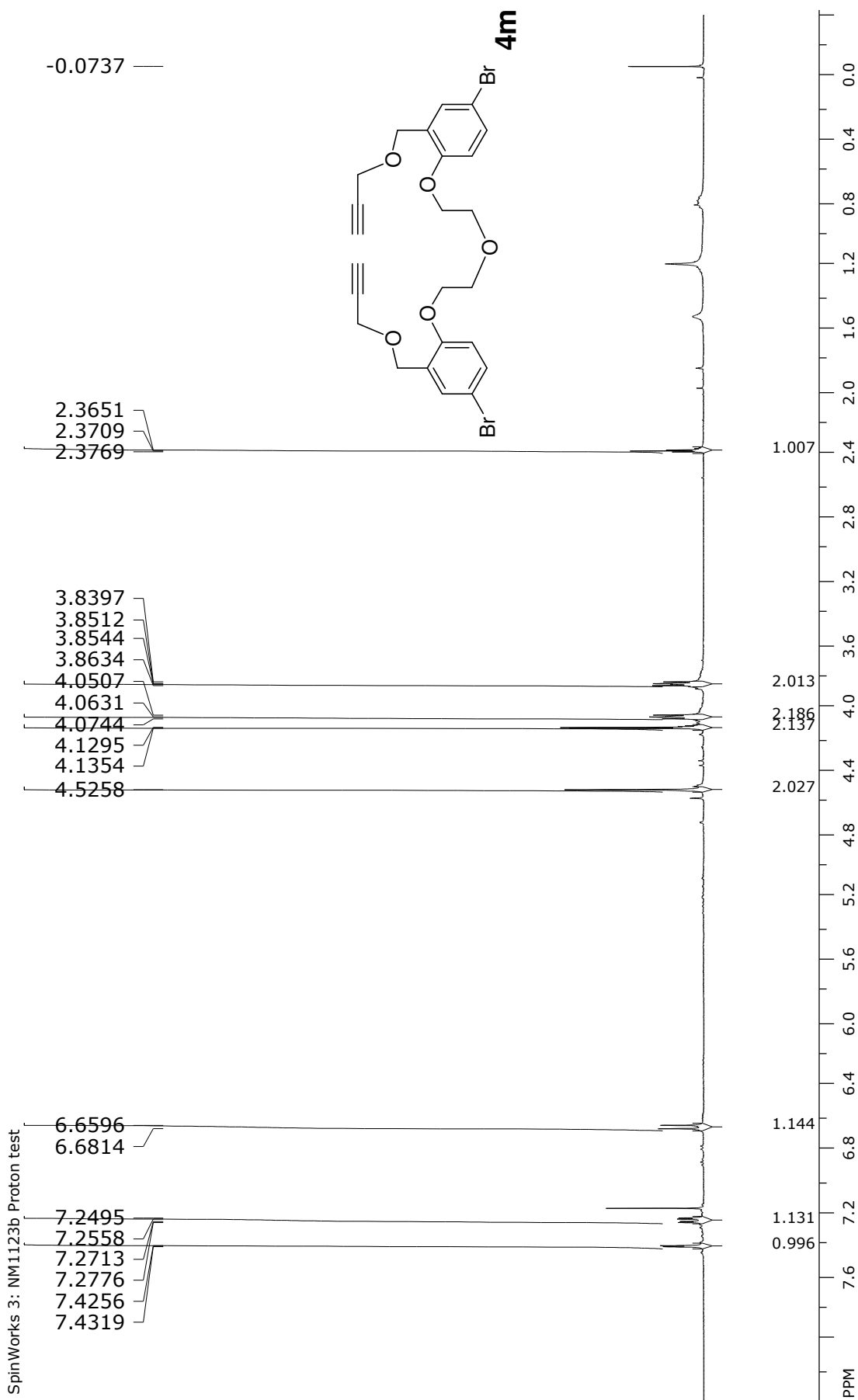
120.8794 —

111.6543 —

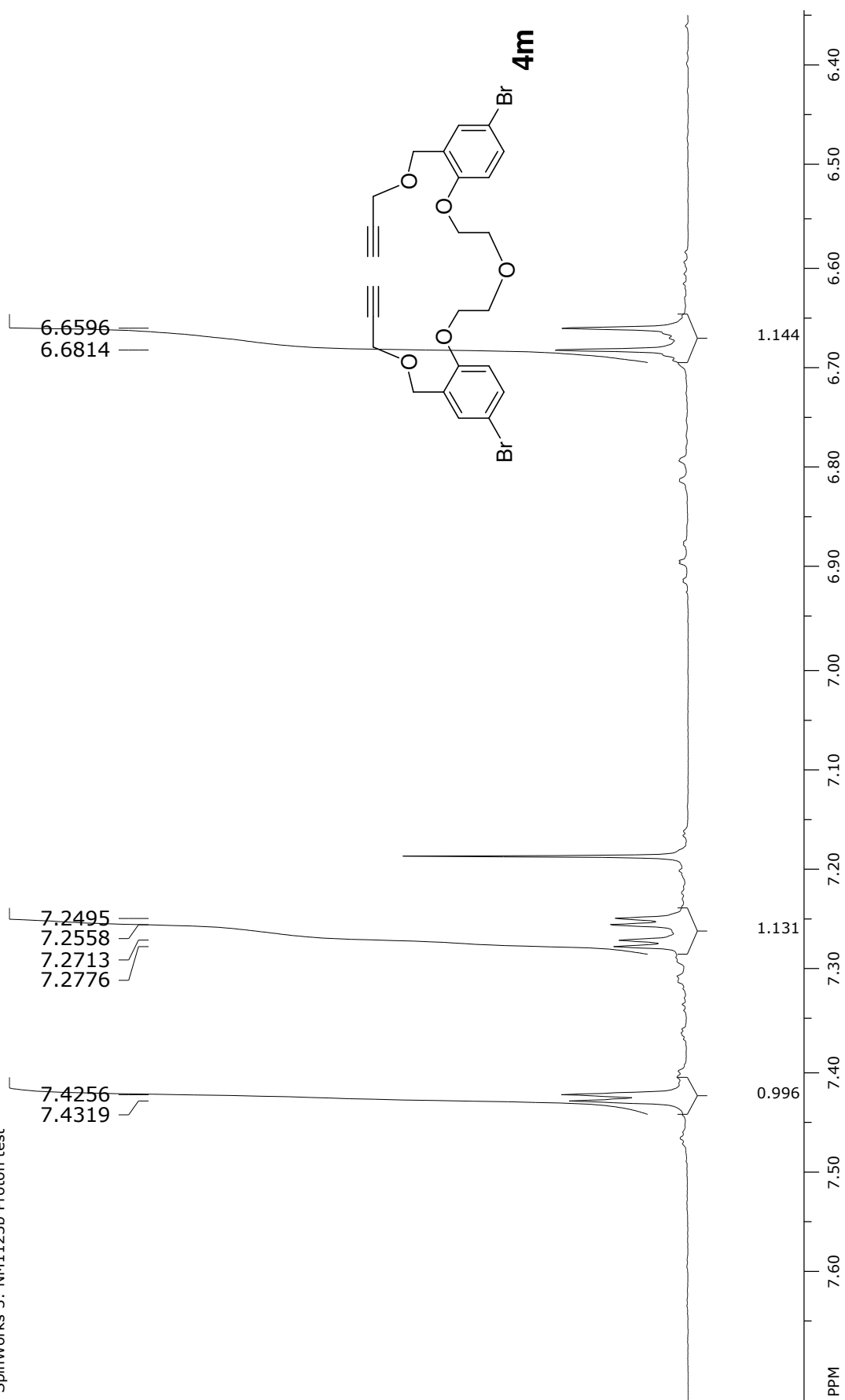


41

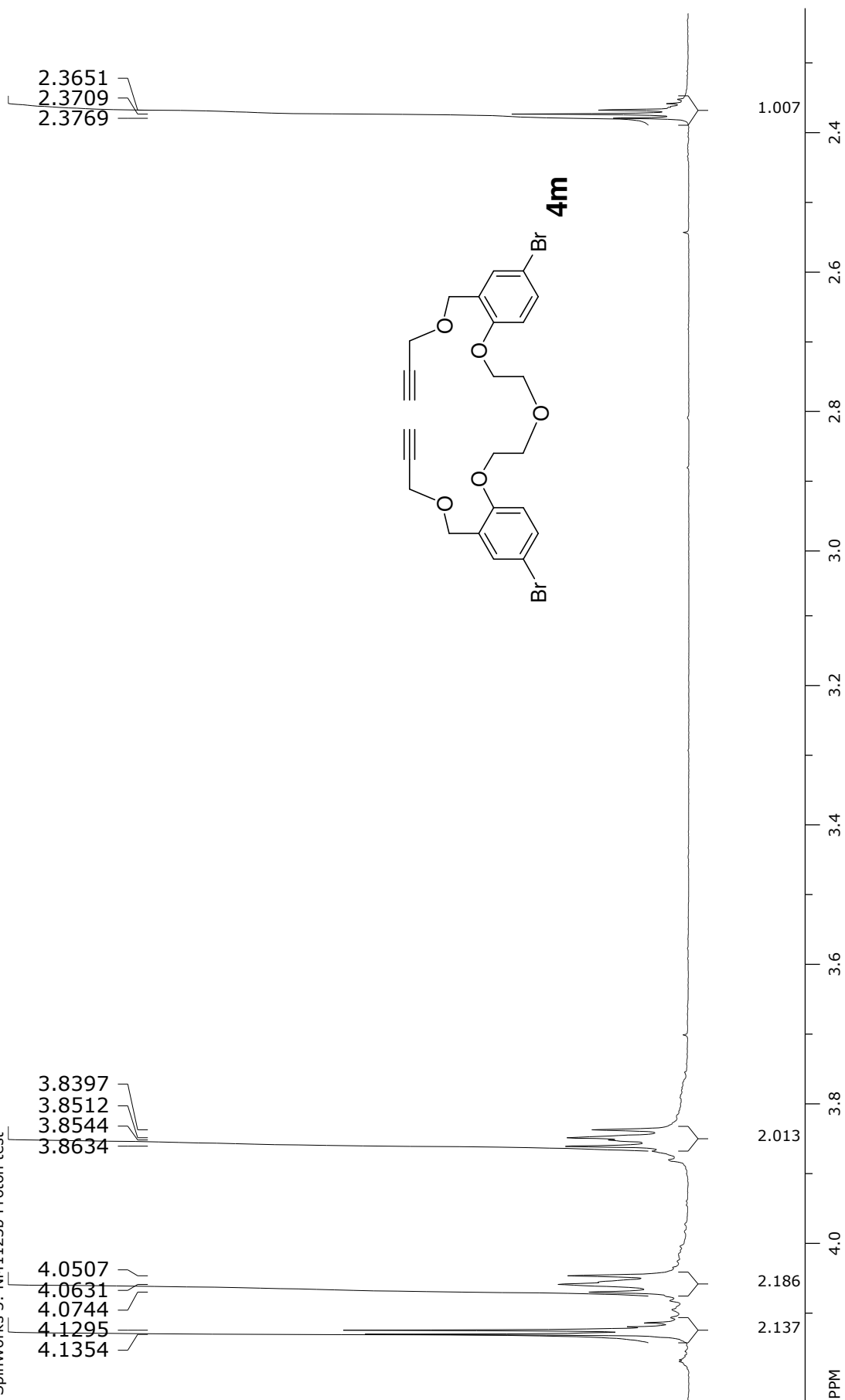


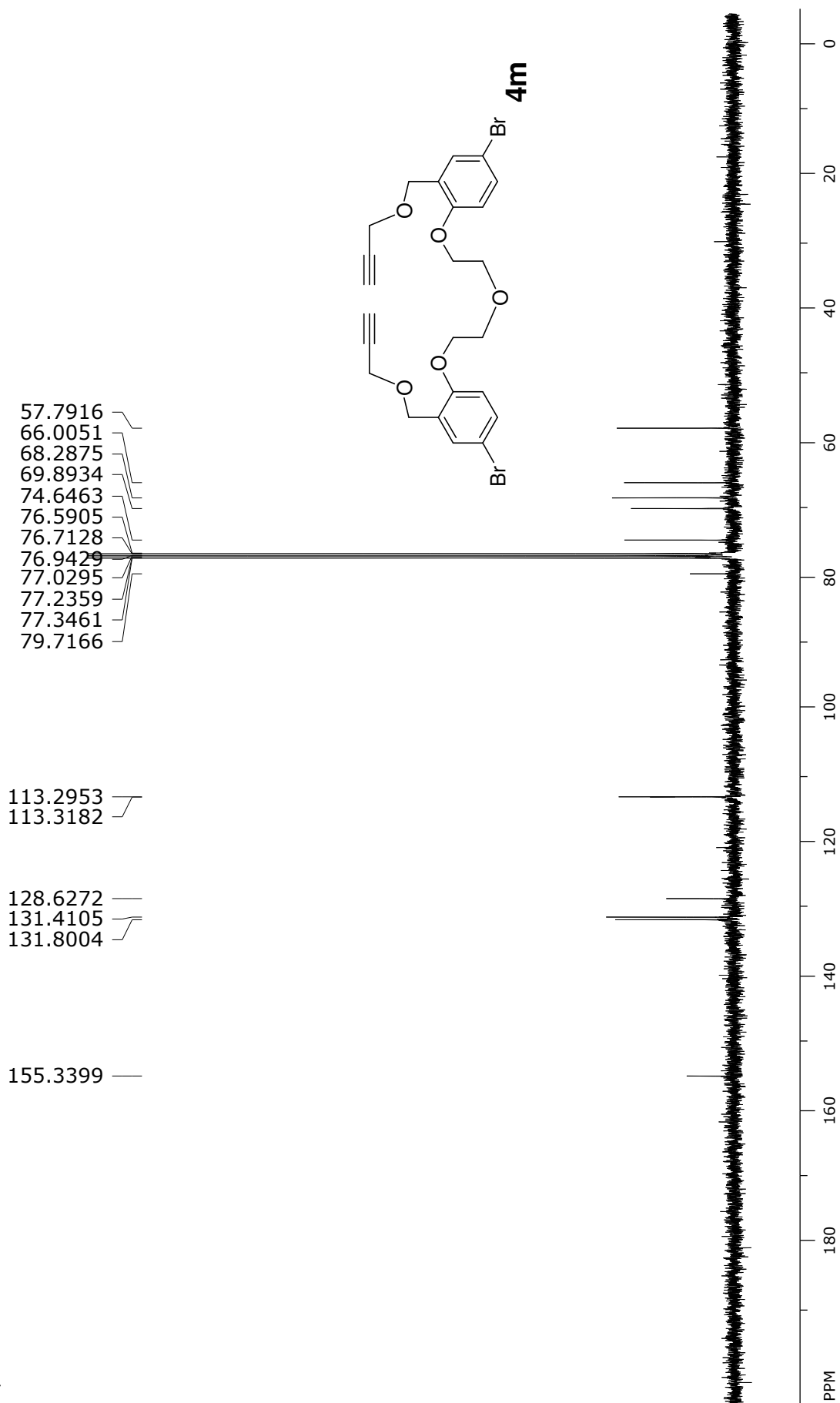


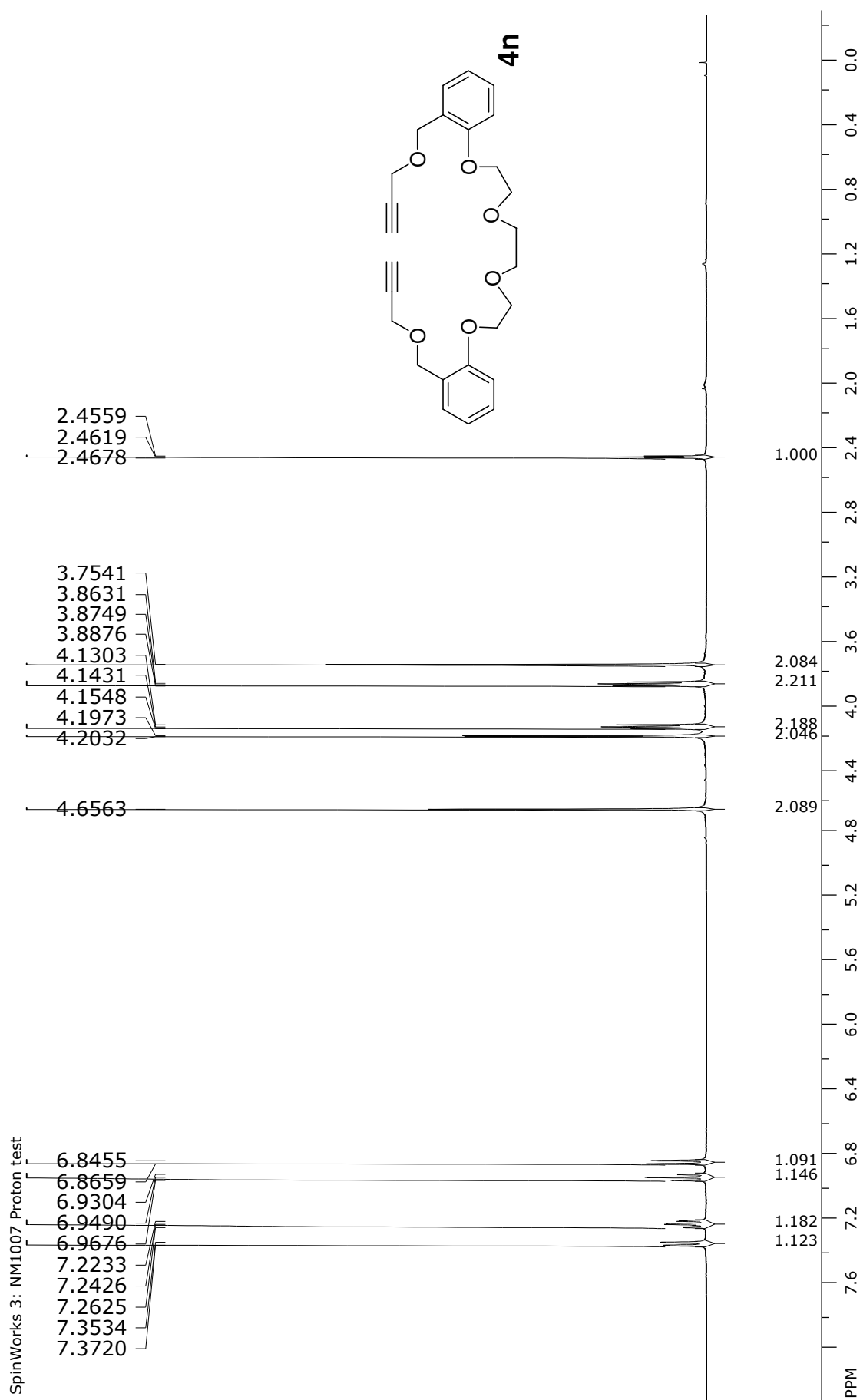
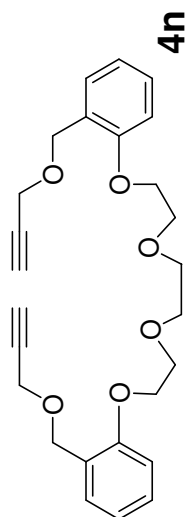
SpinWorks 3: NM1123b Proton test



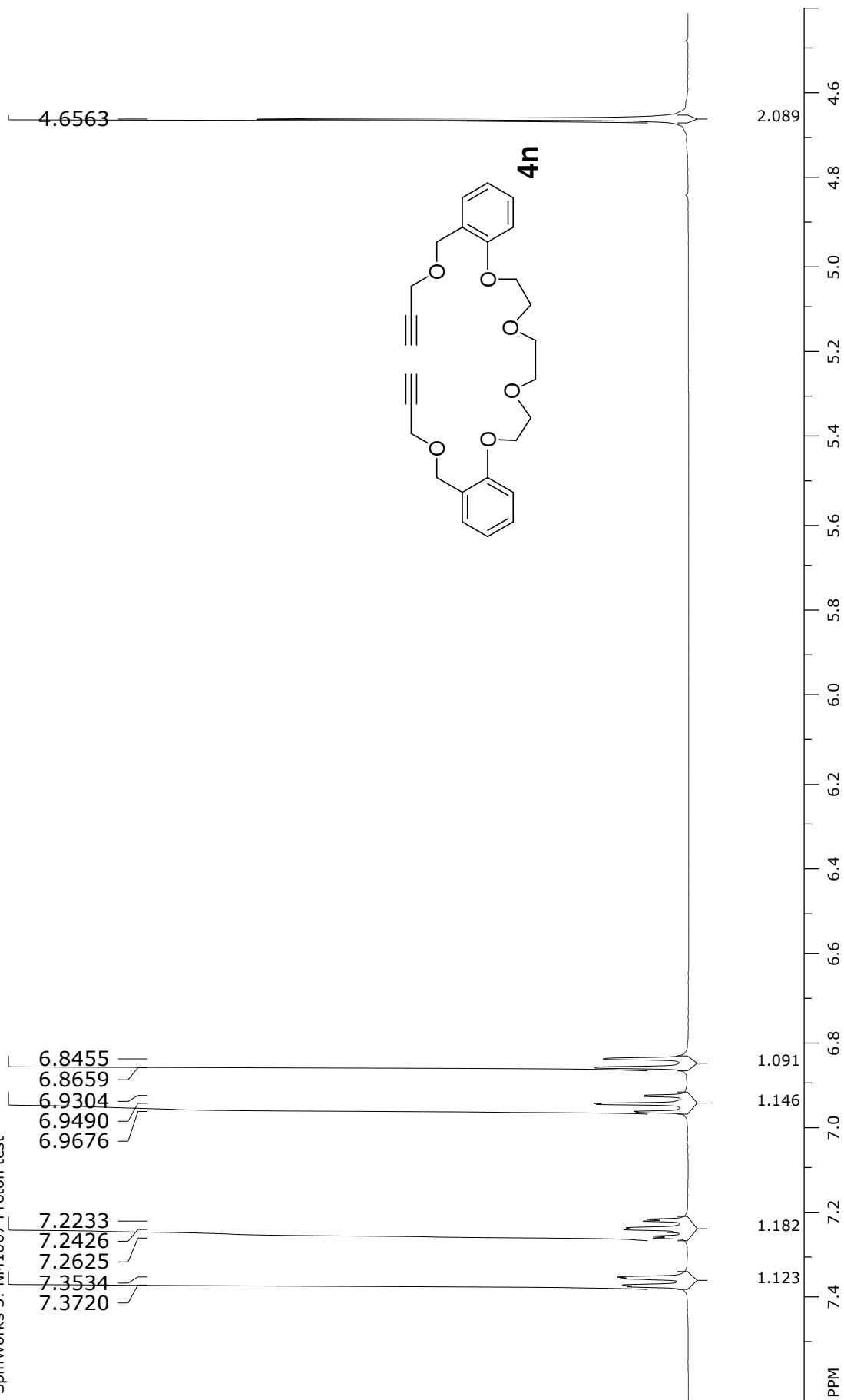
SpinWorks 3: NM1123b Proton test



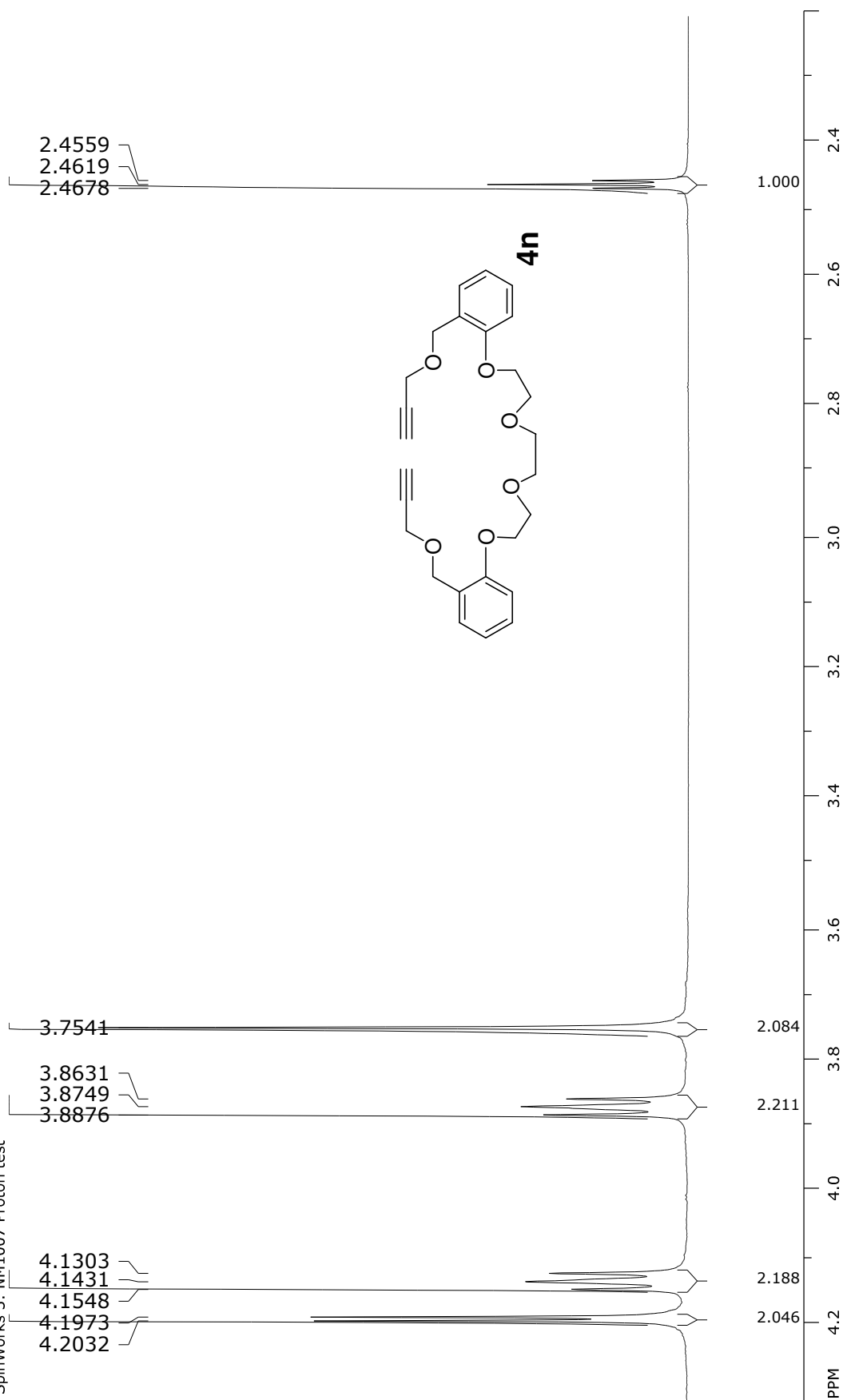




SpinWorks 3: NM1007 Proton test



SpinWorks 3: NM1007 Proton test



57.5226
66.6773
67.9052
69.8198
71.0087
74.4731
76.8768
77.1961
77.5141
80.1499

111.6394

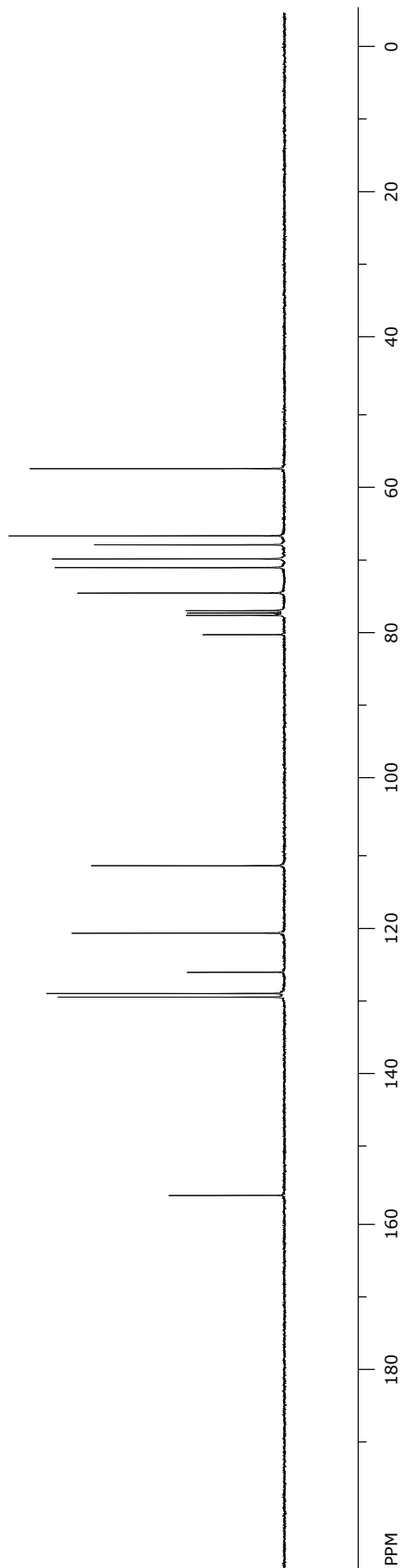
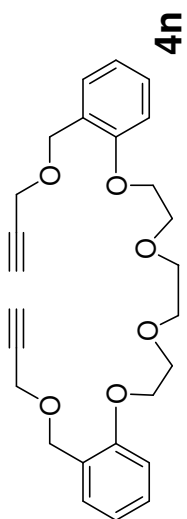
120.8139

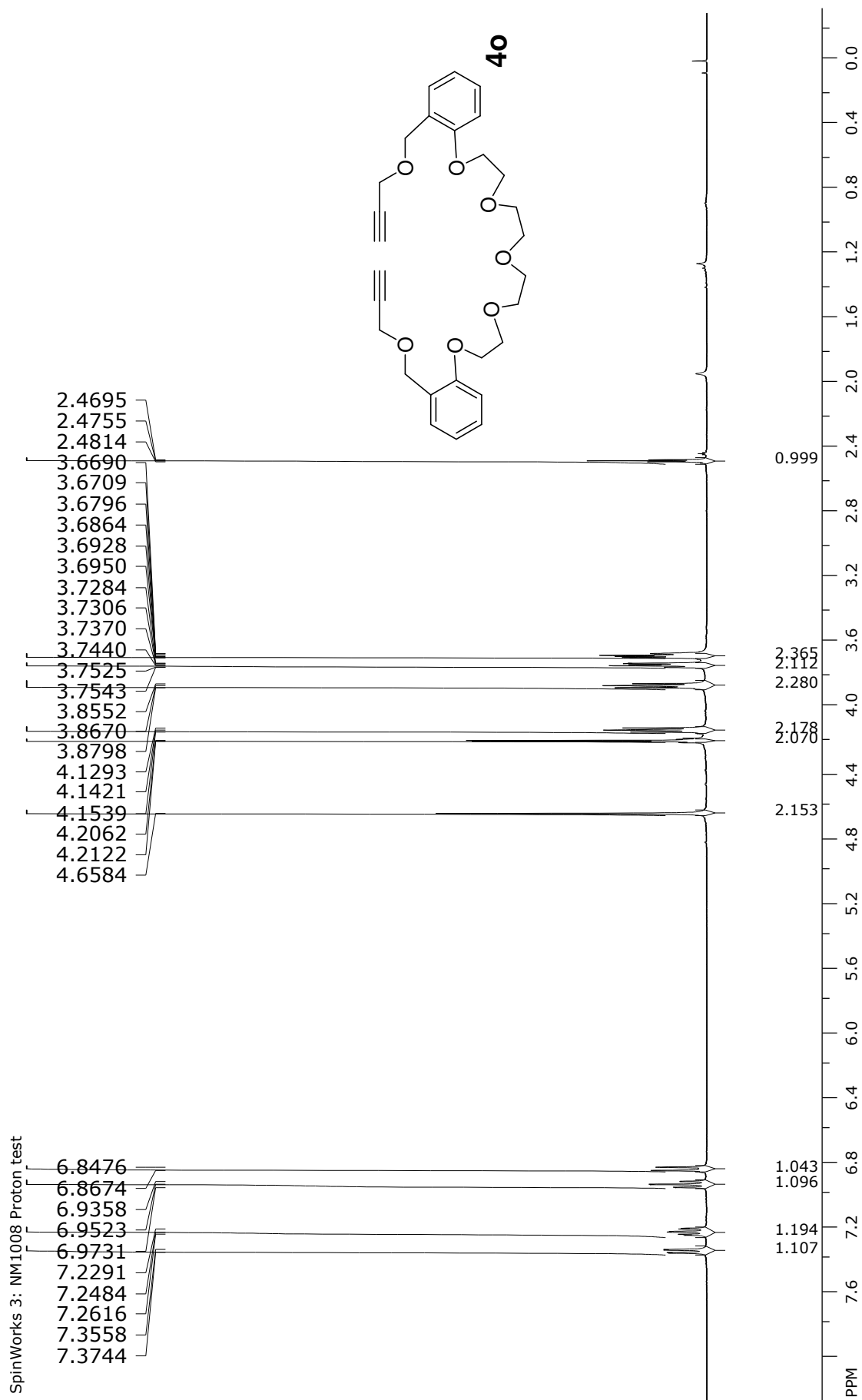
126.1487

129.0471

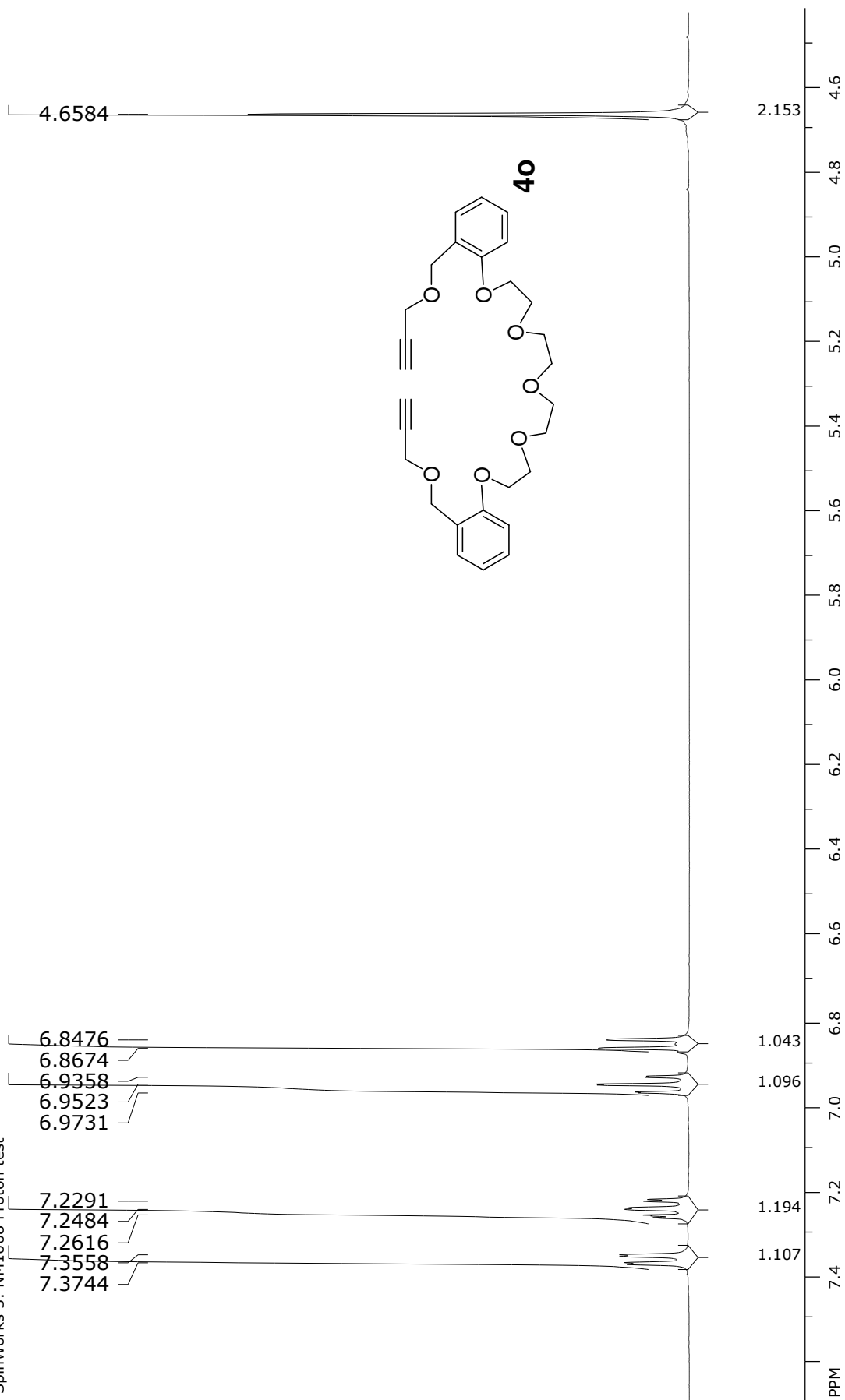
129.5389

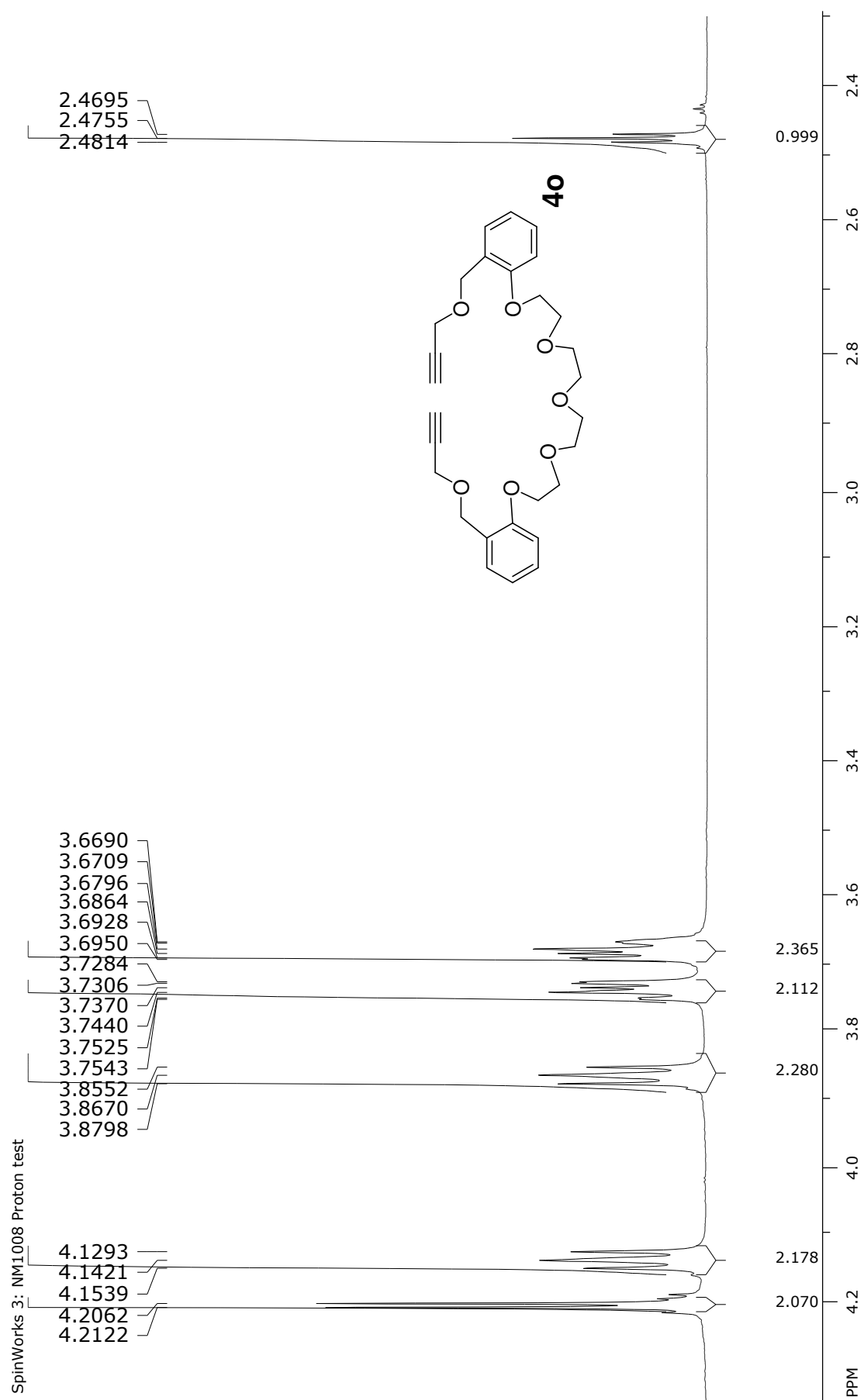
156.5580





SpinWorks 3: NM1008 Proton test





57.5156
66.6530
67.8792
69.7569
70.7507
70.9148
74.4406
76.7935
77.1112
77.4288
80.1143

111.6069

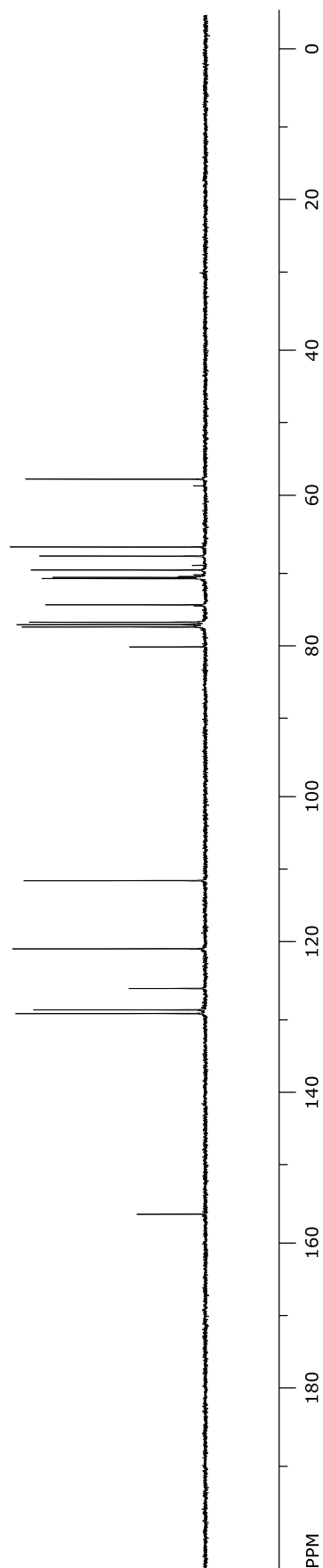
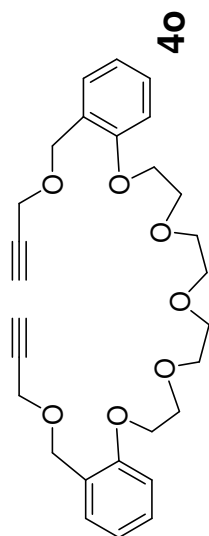
120.7926

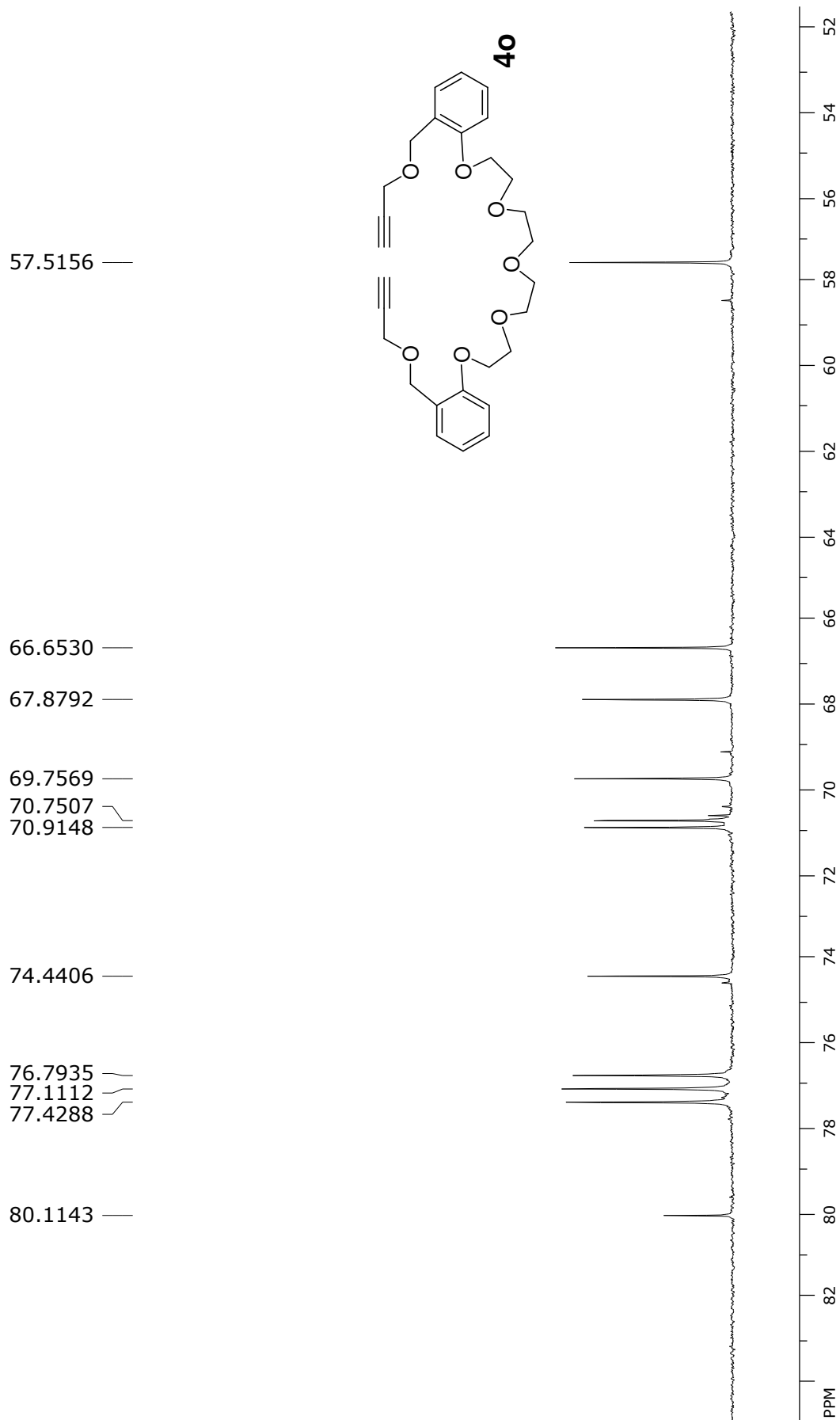
126.1350

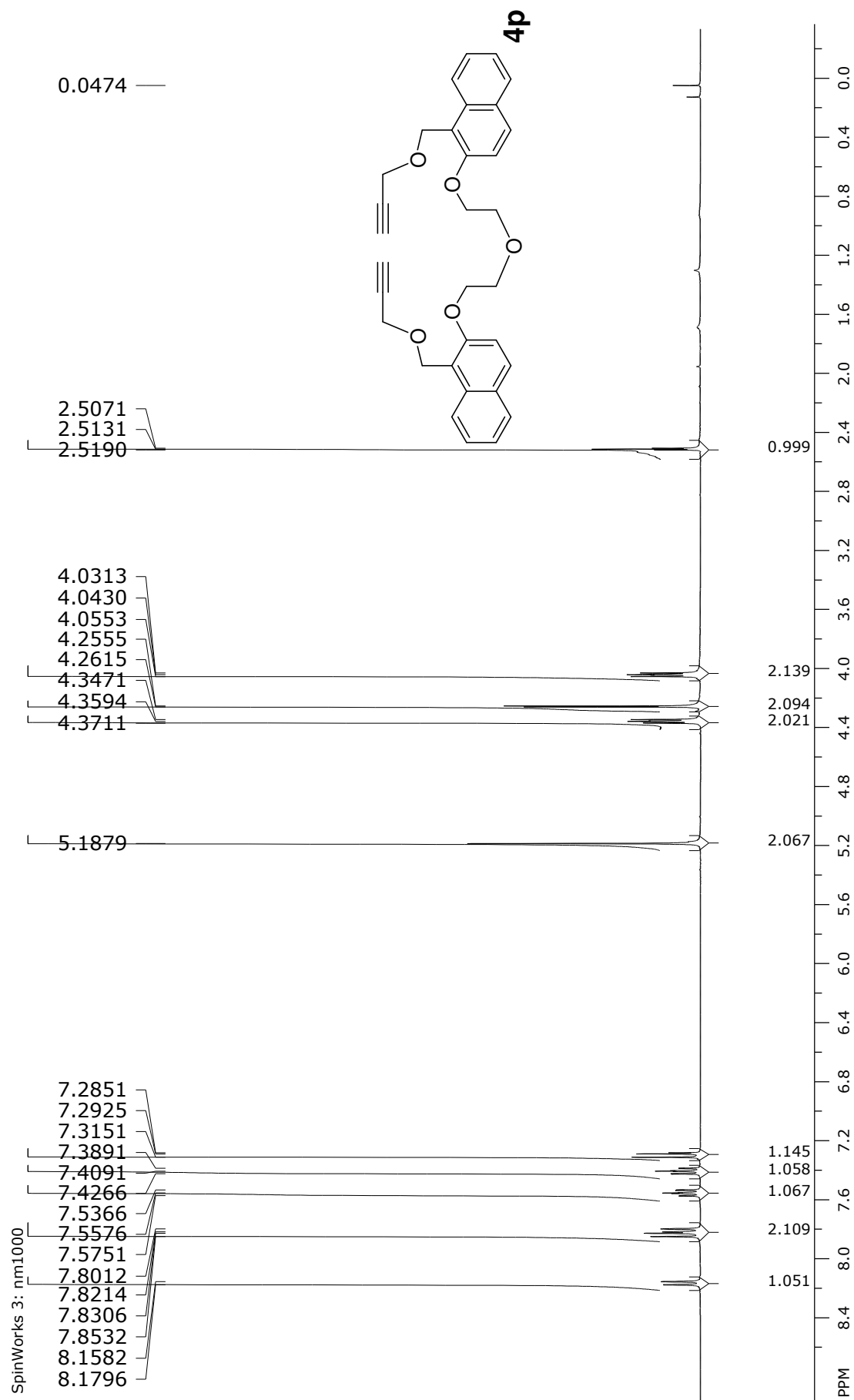
129.0177

129.5140

156.5476



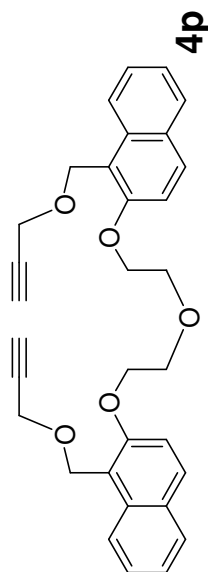




57.3575
62.0418
69.7184
70.2850
74.4556
76.9199
77.2379
77.5557
80.5394

115.0591
118.7560
123.9174
123.9875
127.0674
128.3255
129.4899
130.7065
133.8247

155.1443



SpinWorks 3: NM1000 Carbon test

133.8247 —

130.7065 —

129.4899 —

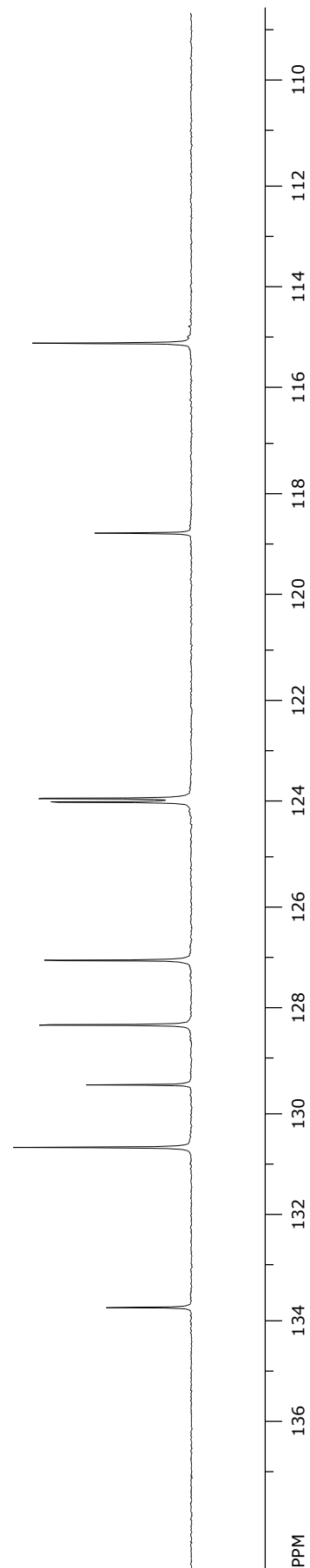
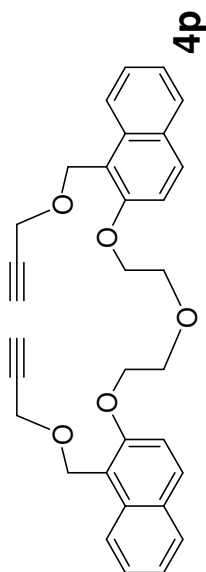
128.3255 —

127.0674 —

123.9174 —
123.9875 —

118.7560 —

115.0591 —



69.7184 —

70.2850 —

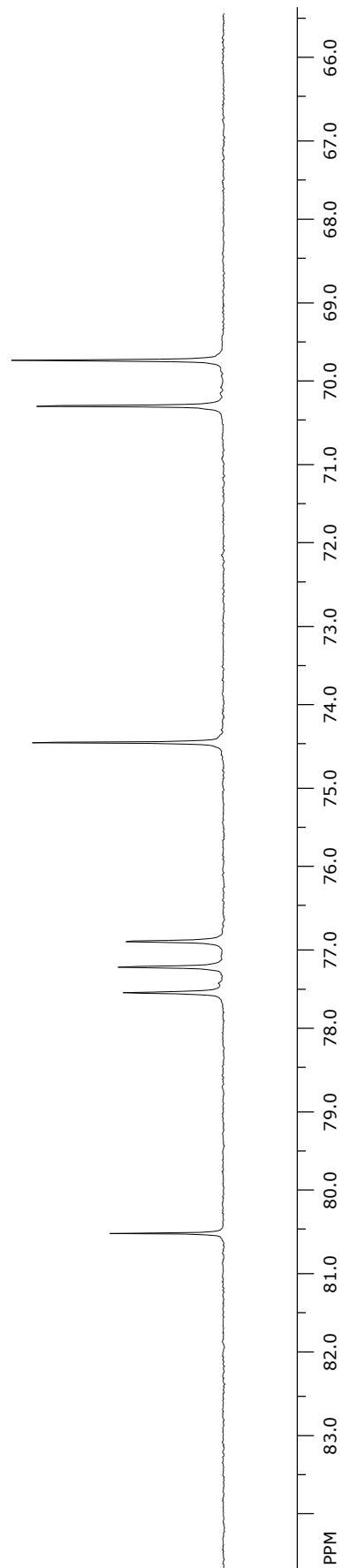
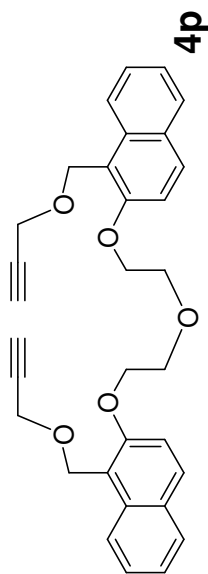
74.4556 —

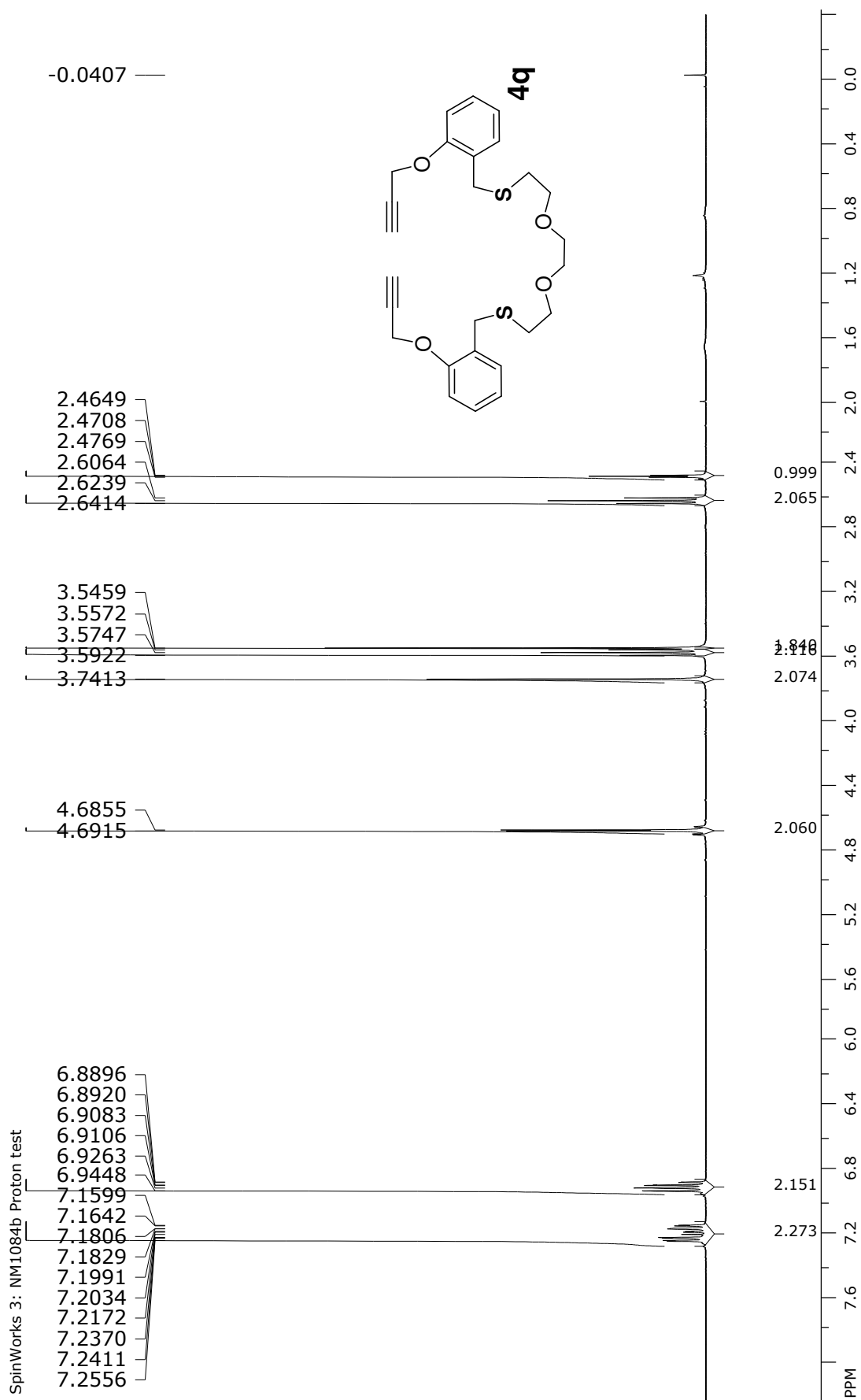
76.9199 —

77.2379 —

77.5557 —

80.5394 —

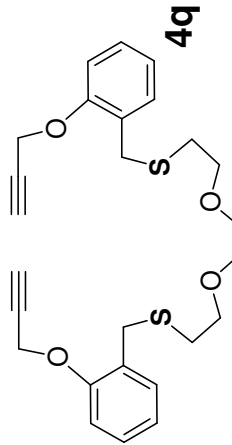




SpinWorks 3: NM1084b Proton test

7.2411
7.2370
7.2172
7.2034
7.1991
7.1829
7.1806
7.1642
7.1599
6.9448
6.9263
6.9106
6.9083
6.8920
6.8896

4.6915
4.6855



2.151

2.273

2.060

PPM

4.4

4.6

4.8

5.0

5.2

5.4

5.6

5.8

6.0

6.2

6.4

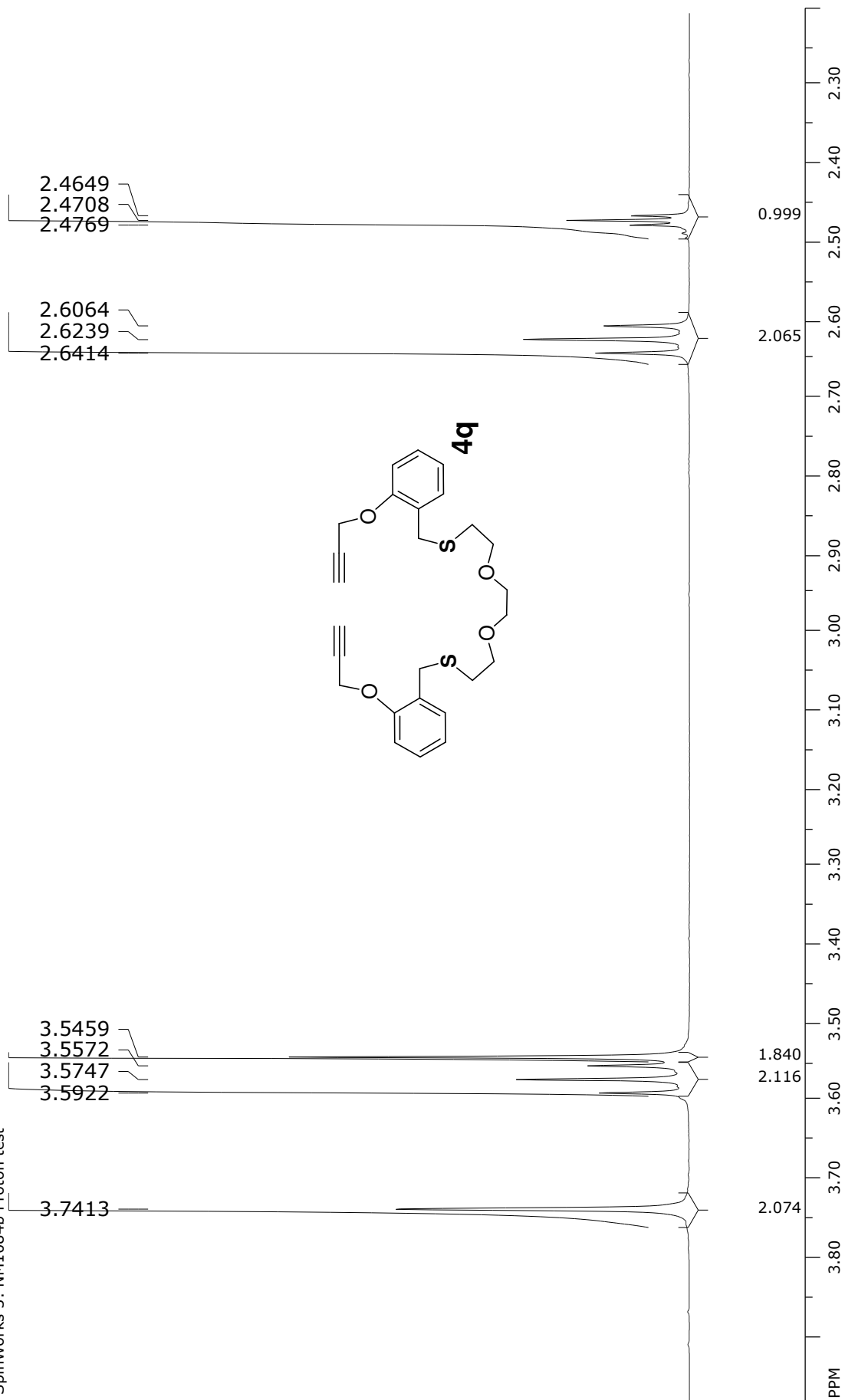
6.6

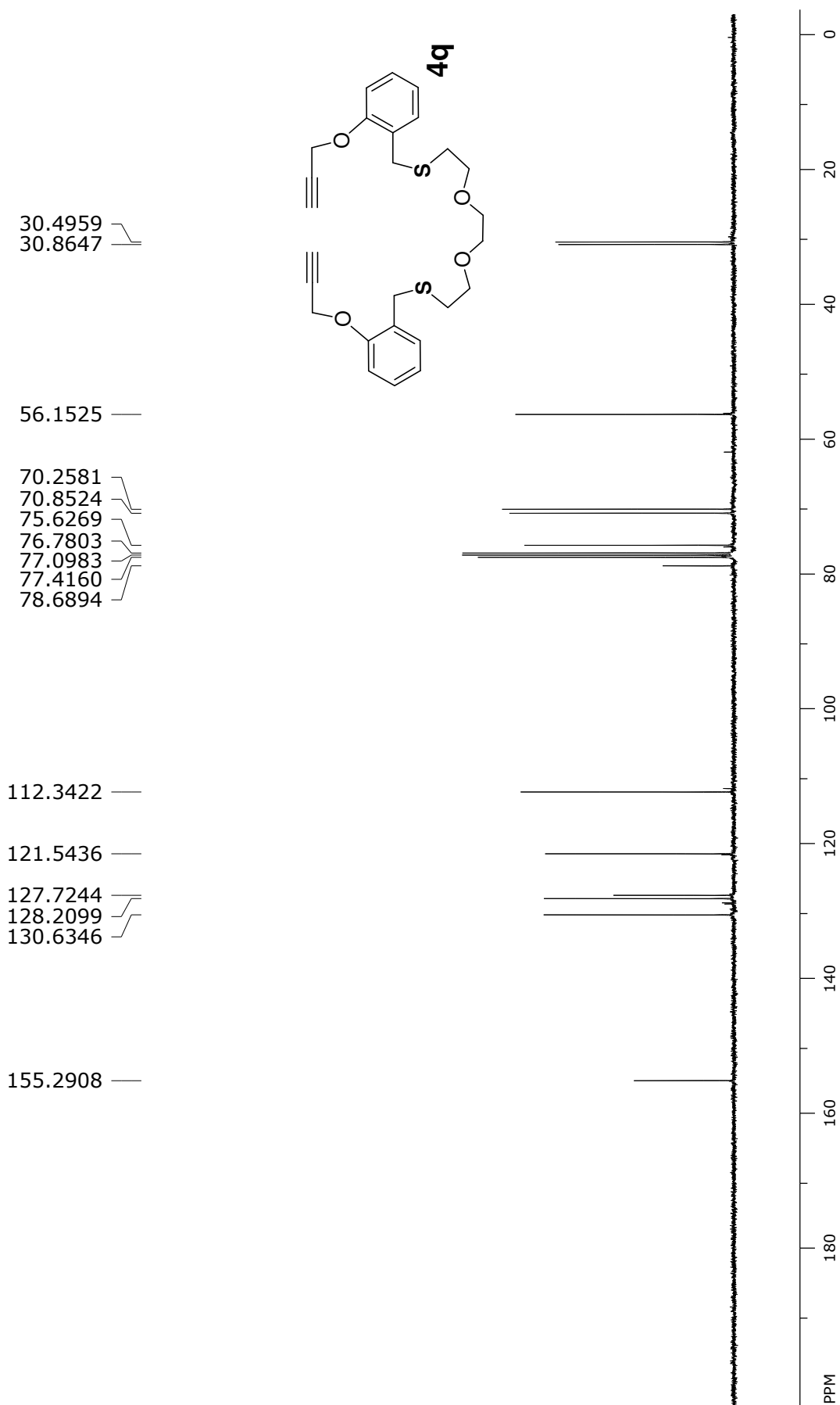
6.8

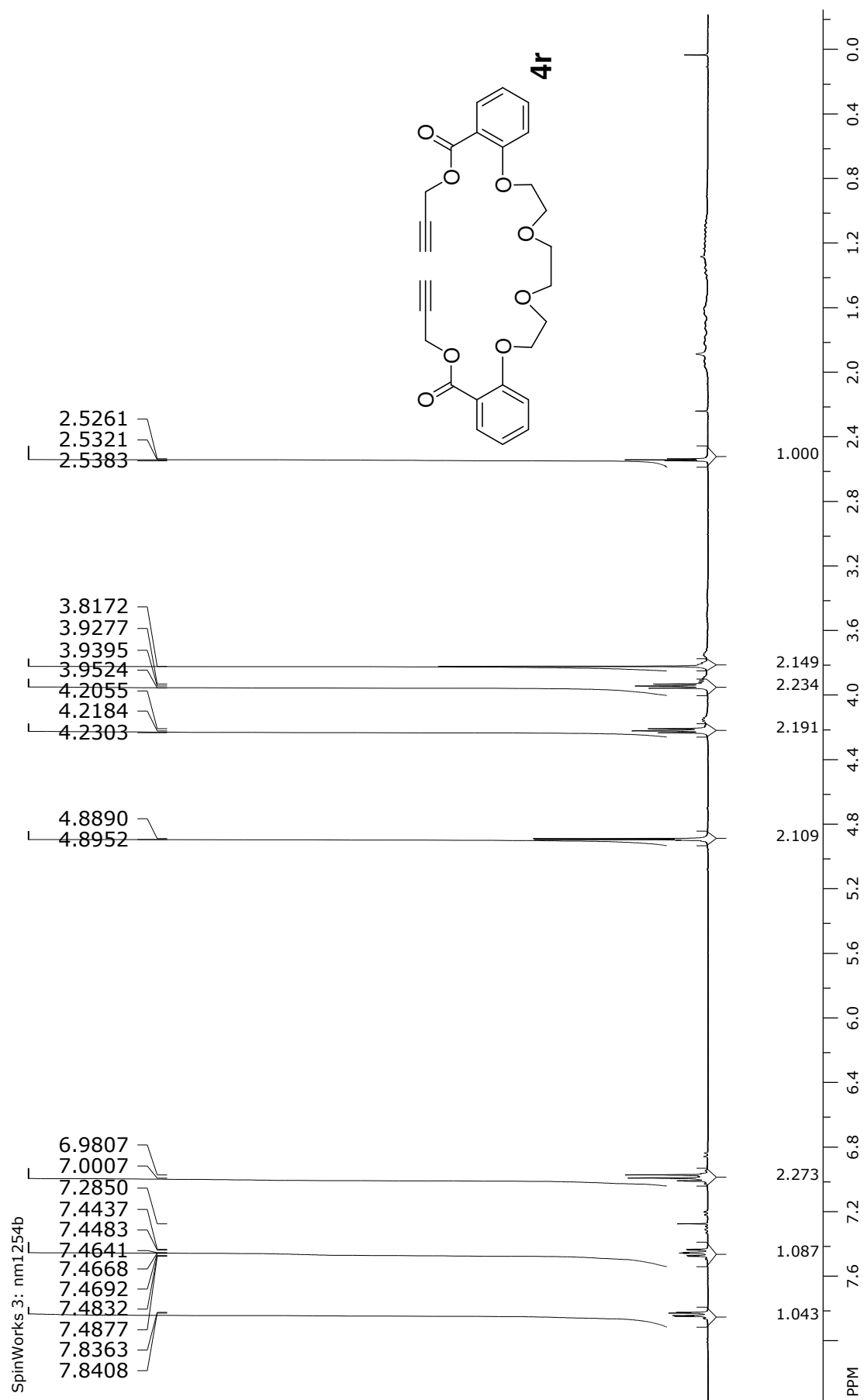
7.0

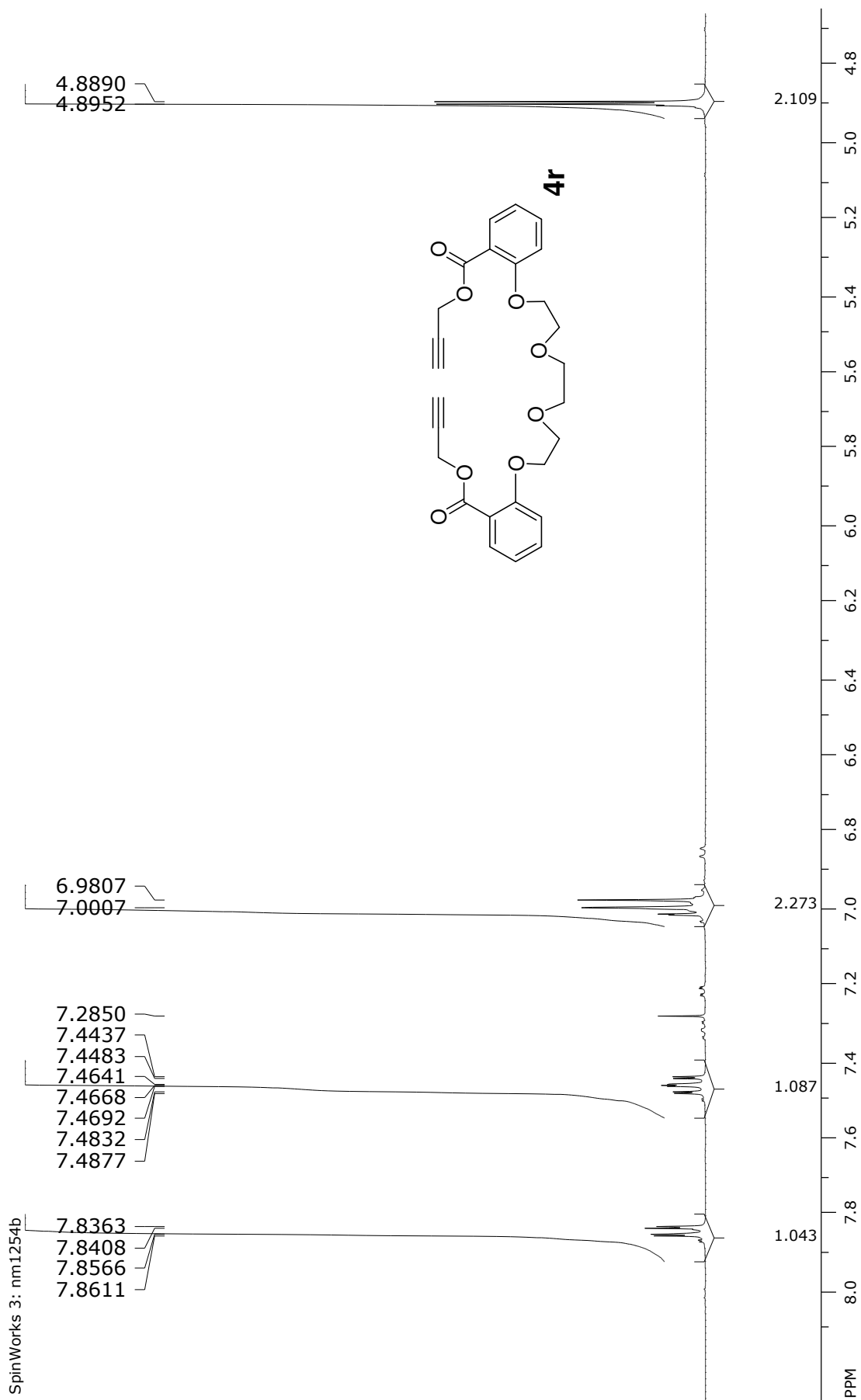
7.2

SpinWorks 3: NM1084b Proton test

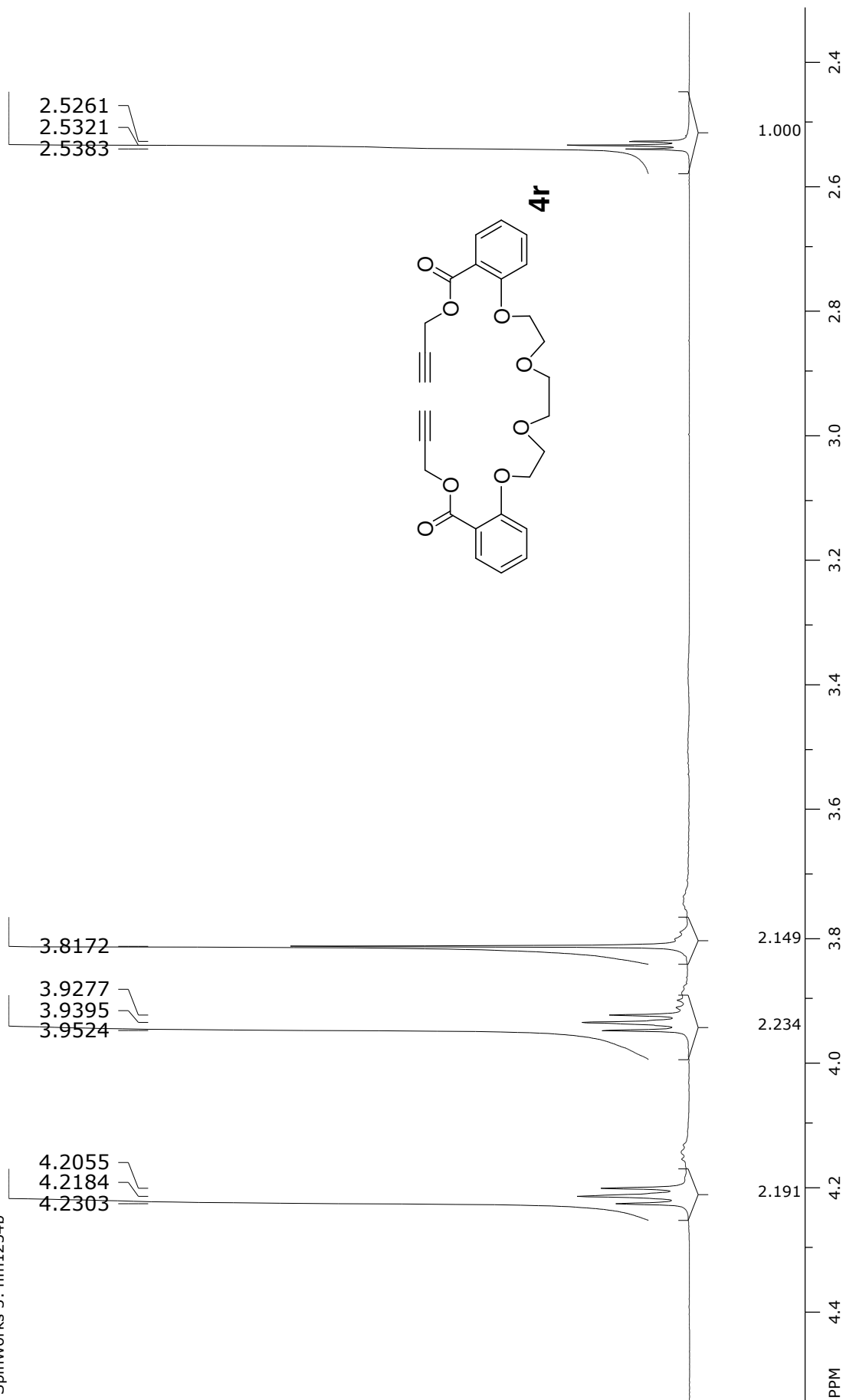








SpinWorks 3: nm1254b



SpinWorks 3: nm-1254b

158.7049 —

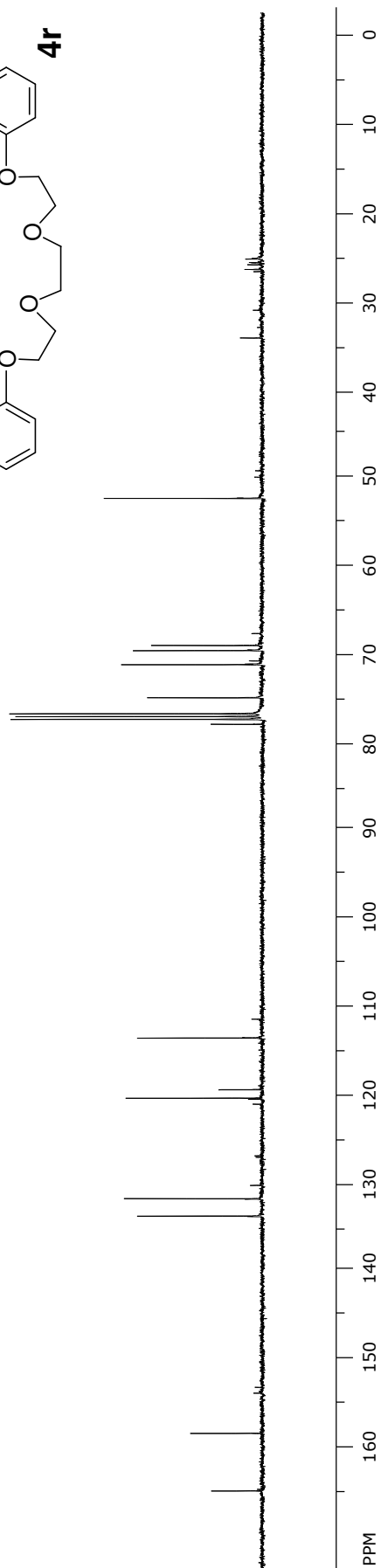
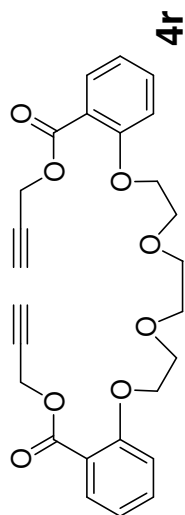
165.2590 —

131.9819 —
133.9595 —

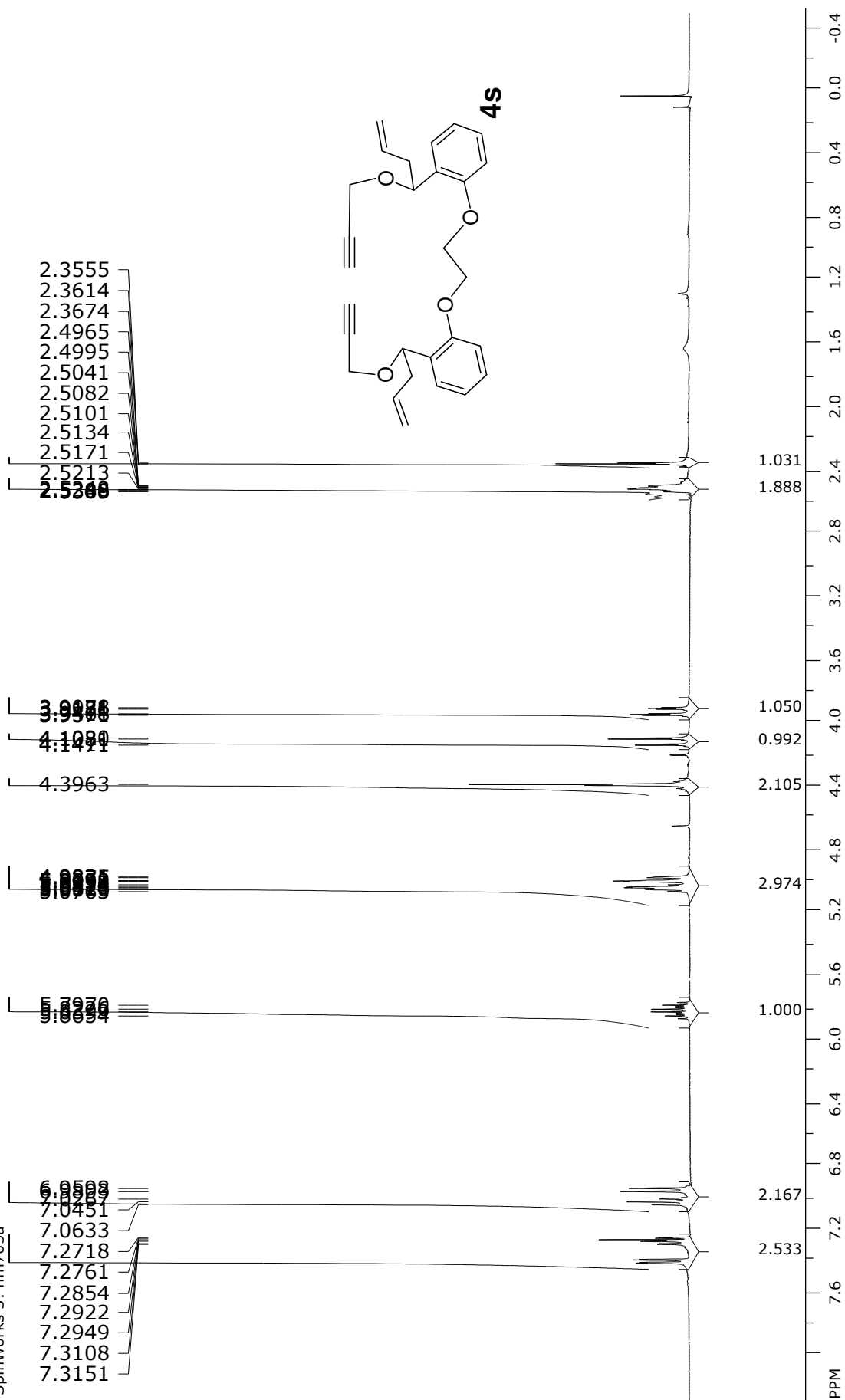
113.6804 —
119.5694 —
120.5345 —

68.9796 —
69.5617 —
71.1653 —
74.9325 —
76.7565 —
77.0738 —
77.3919 —
77.9617 —

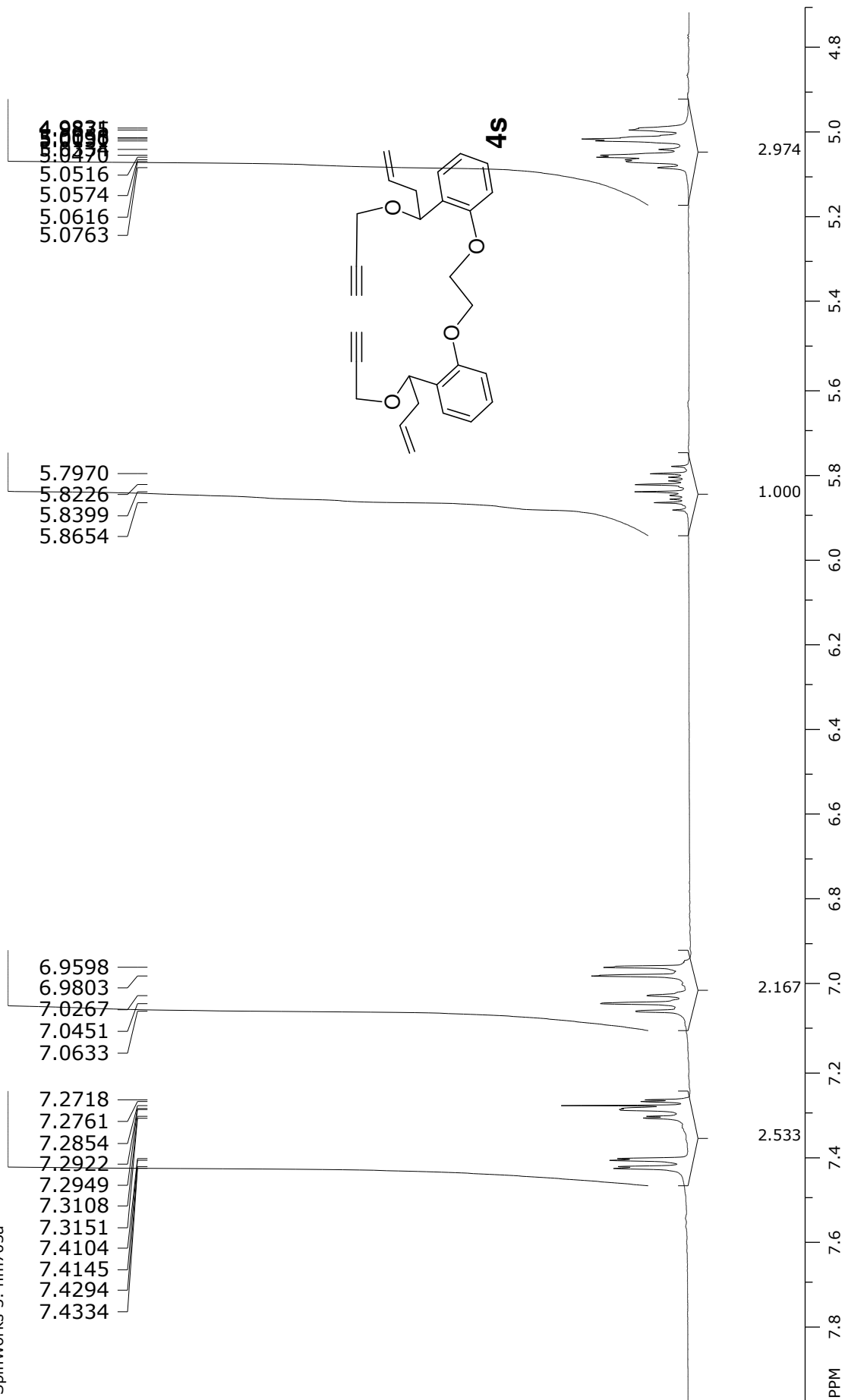
52.2297 —



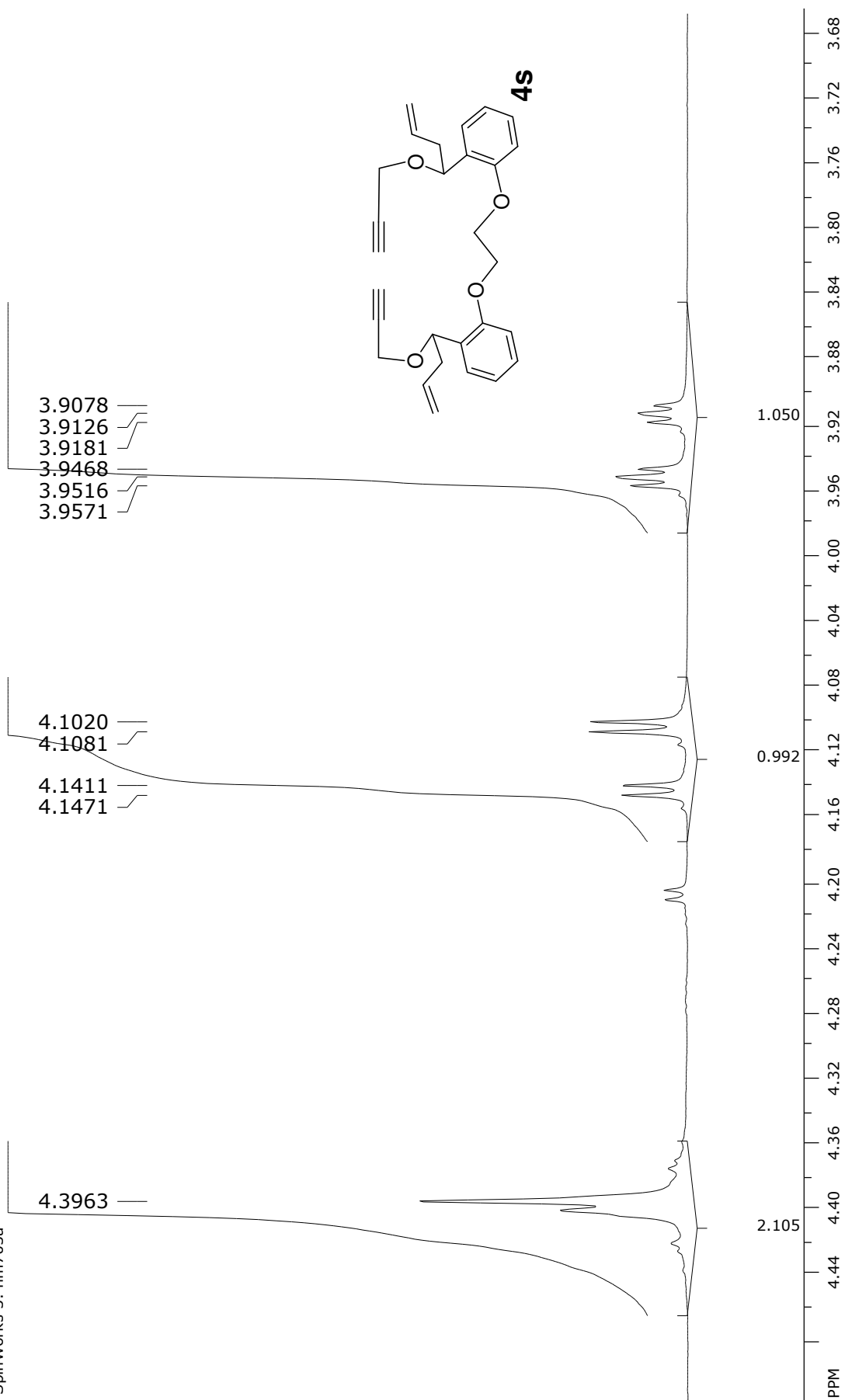
SpinWorks 3: nm703a



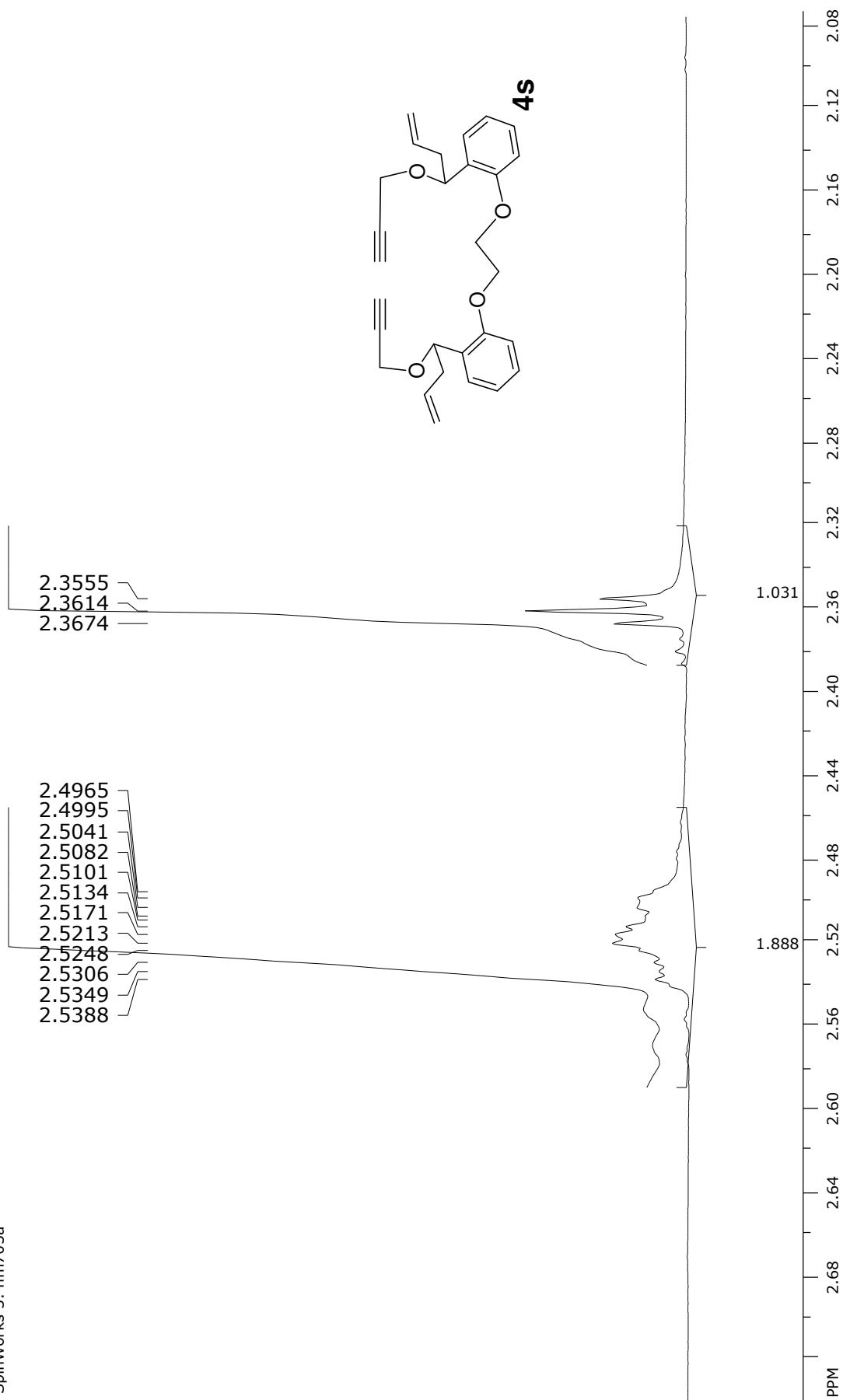
SpinWorks 3: nm703a



SpinWorks 3: nm703a



SpinWorks 3: nm703a



SpinWorks 3: nm-703-a

156.1229 —

134.8595 —

129.4020 —

128.5464 —

127.0367 —

121.3168 —

116.7101 —

111.5668 —

80.2270 —

77.3716 —

76.7365 —

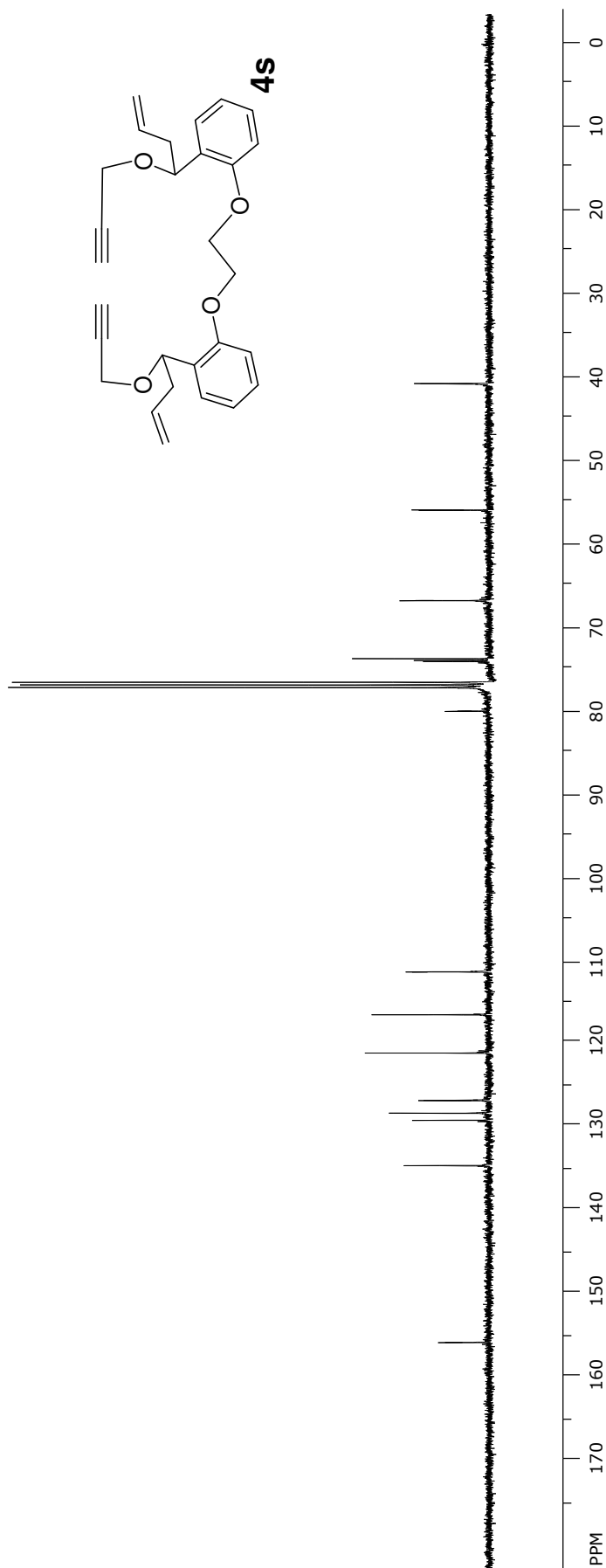
74.1178 —

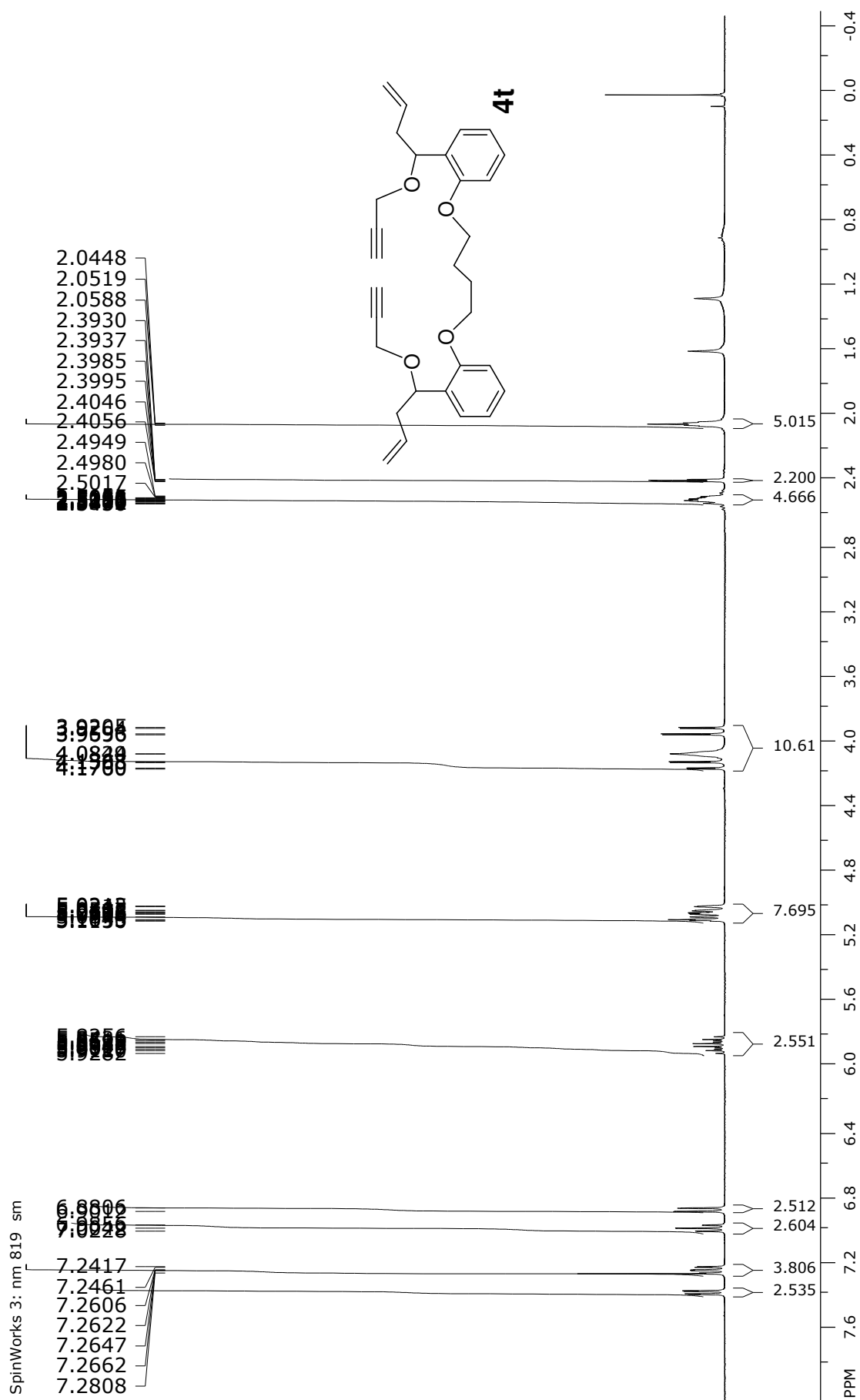
73.9061 —

66.9216 —

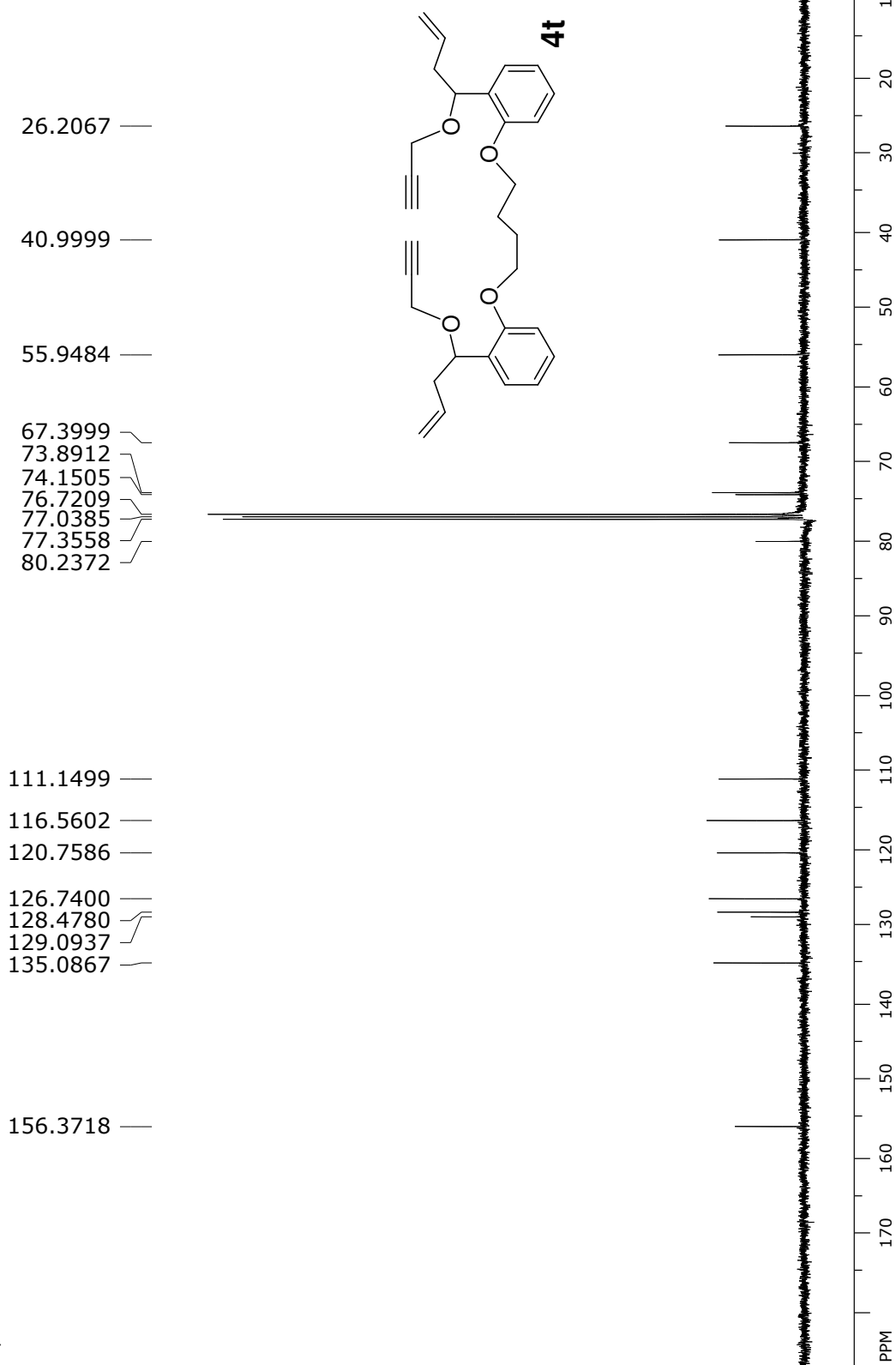
56.0220 —

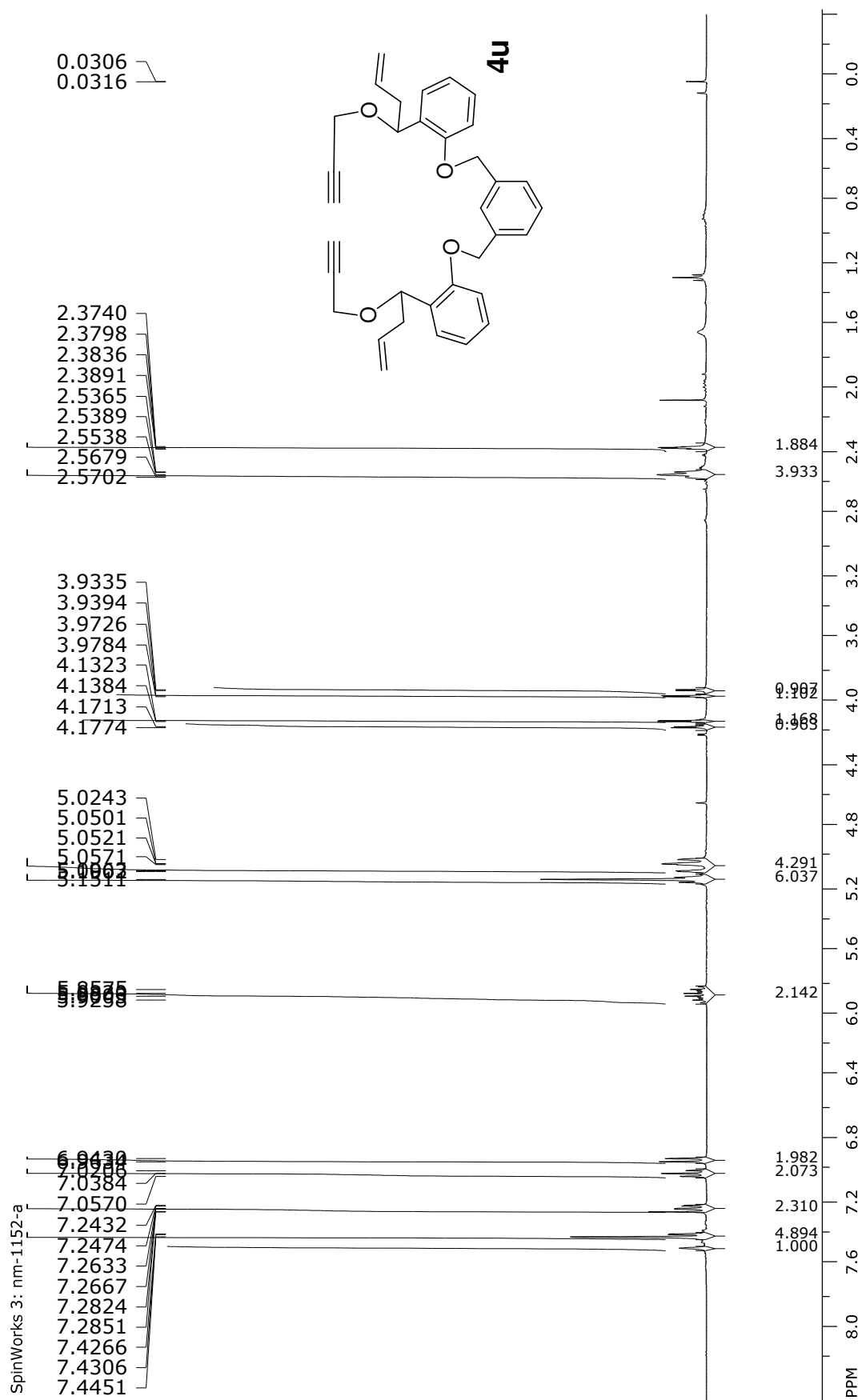
40.8129 —



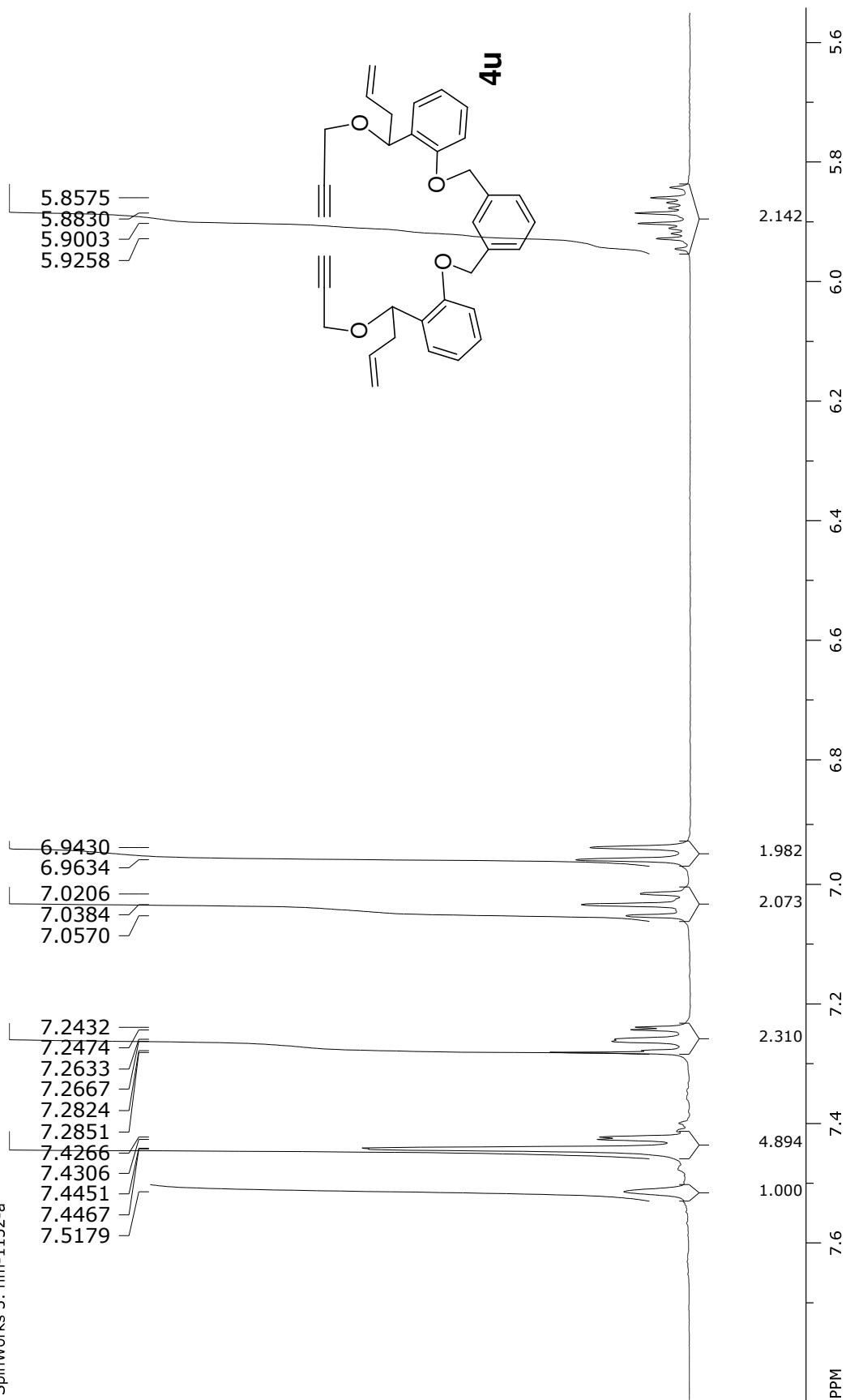


SpinWorks 3: nm 819 sm

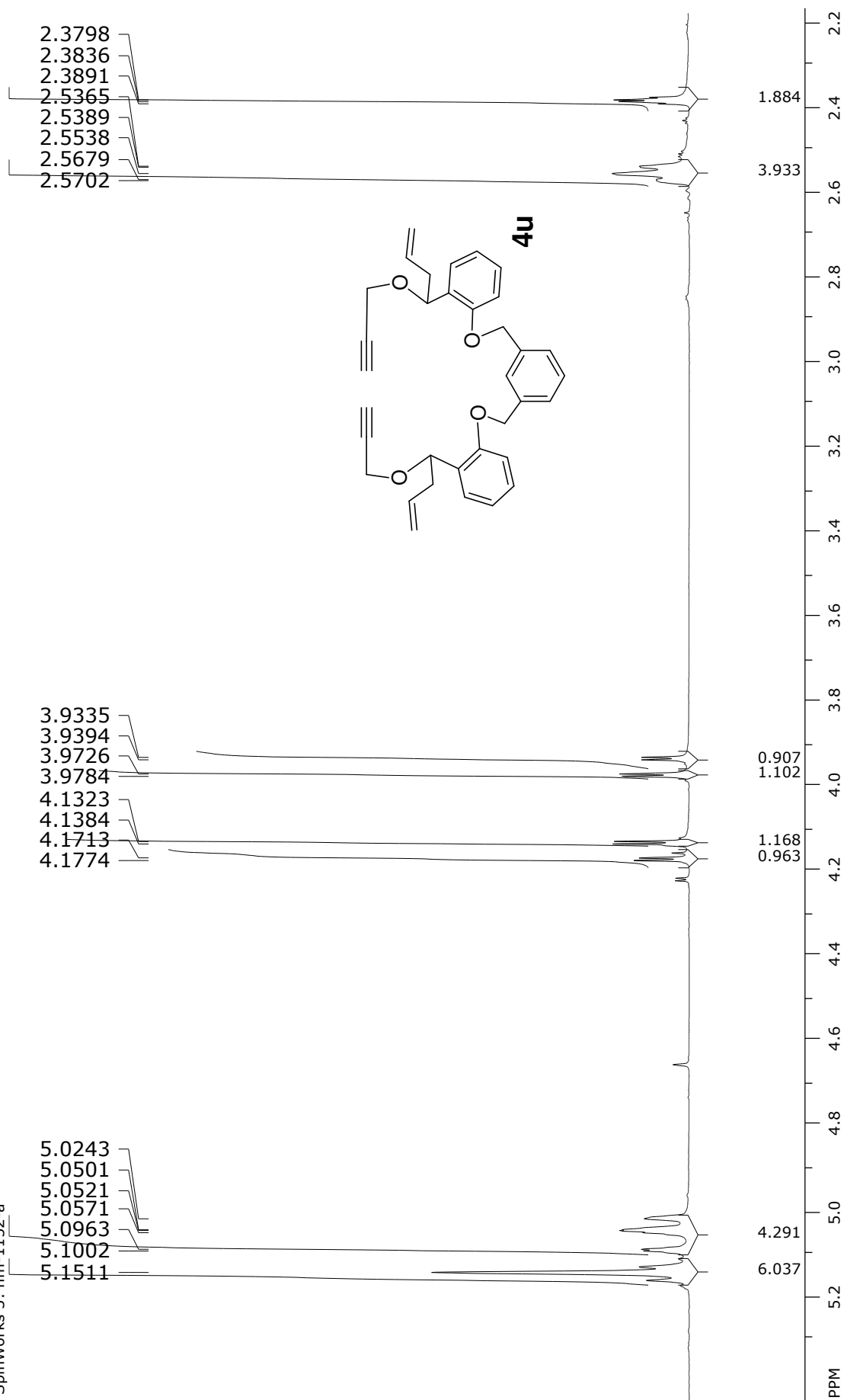


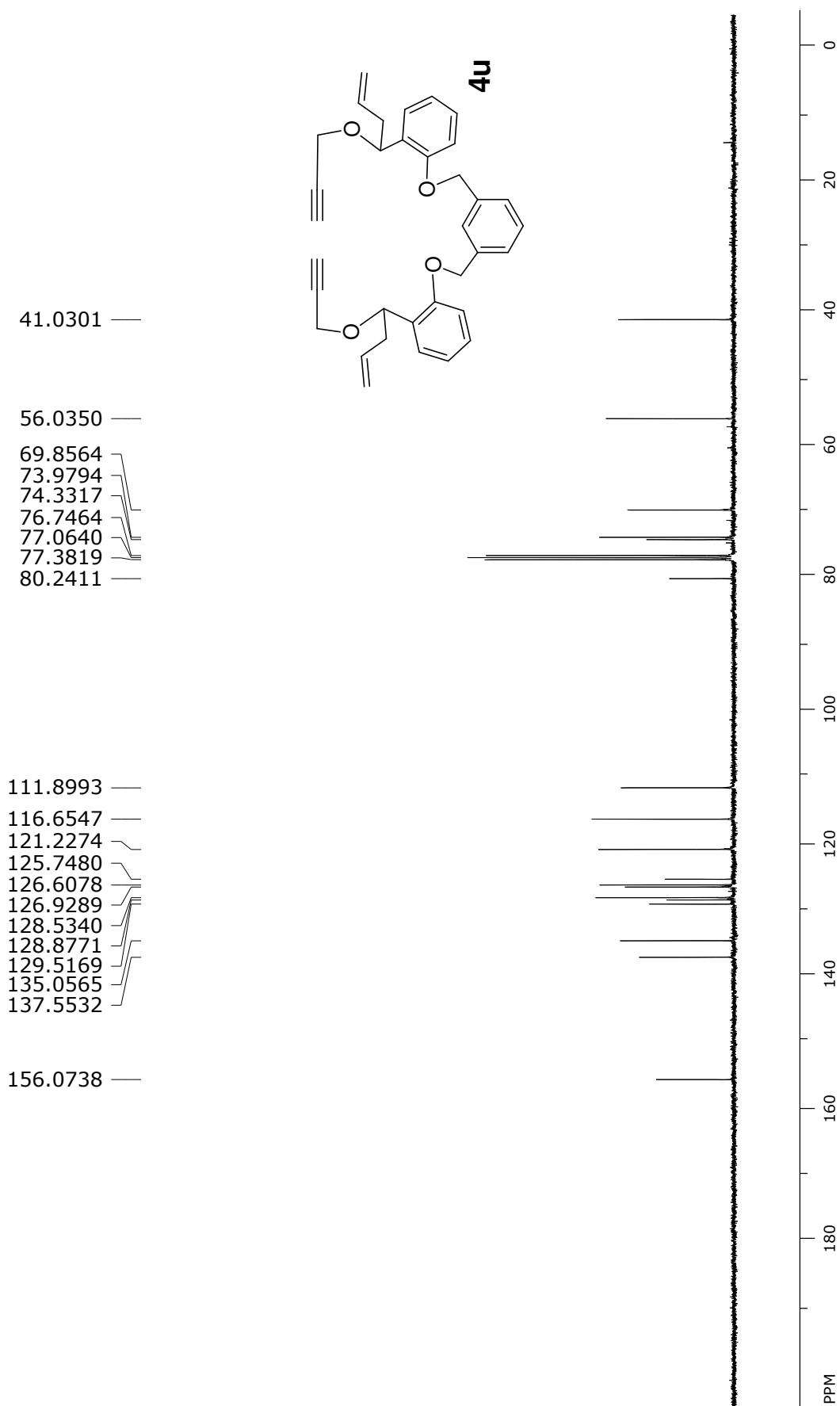


SpinWorks 3: nm-1152-a

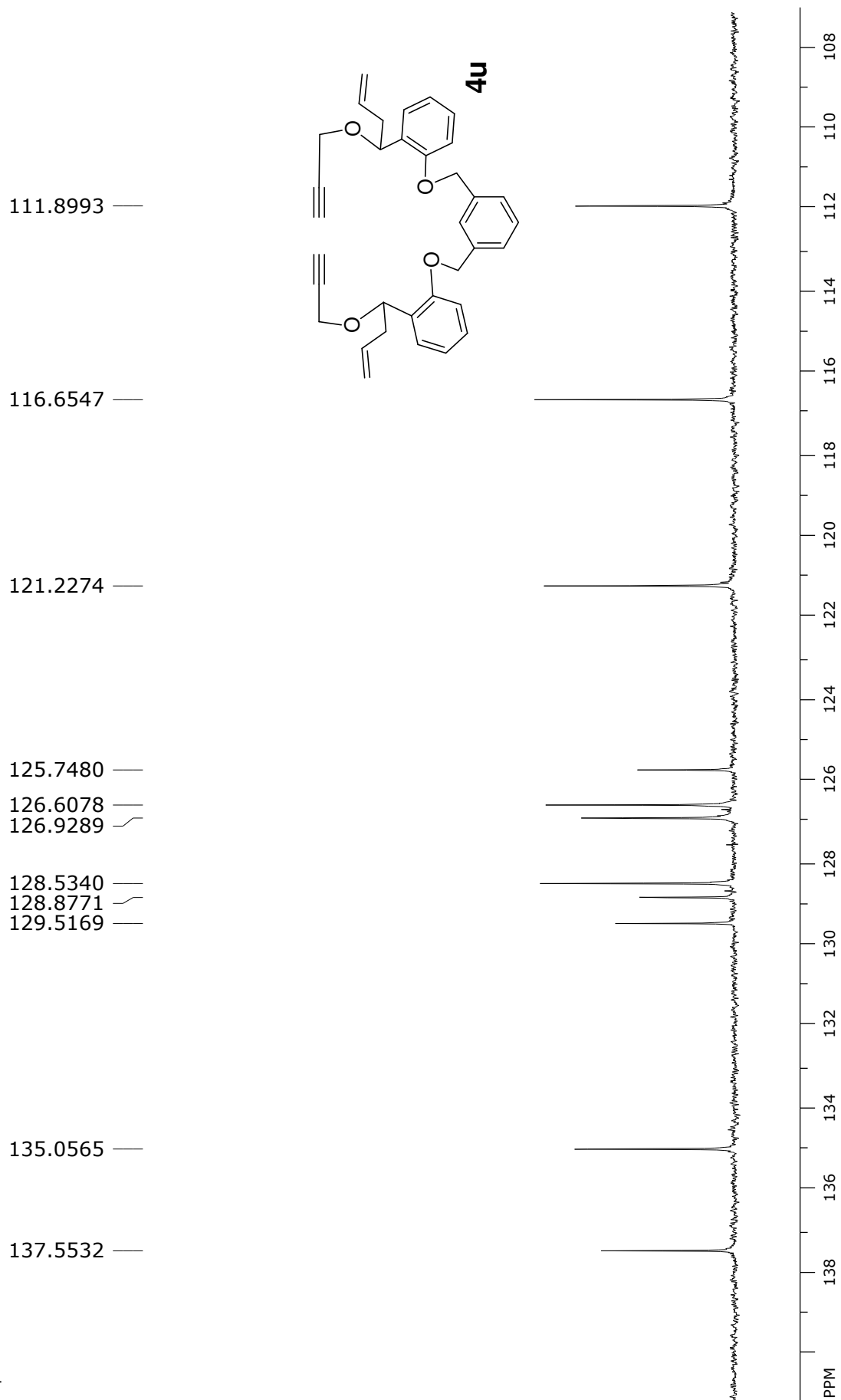


SpinWorks 3: nm-1152-a

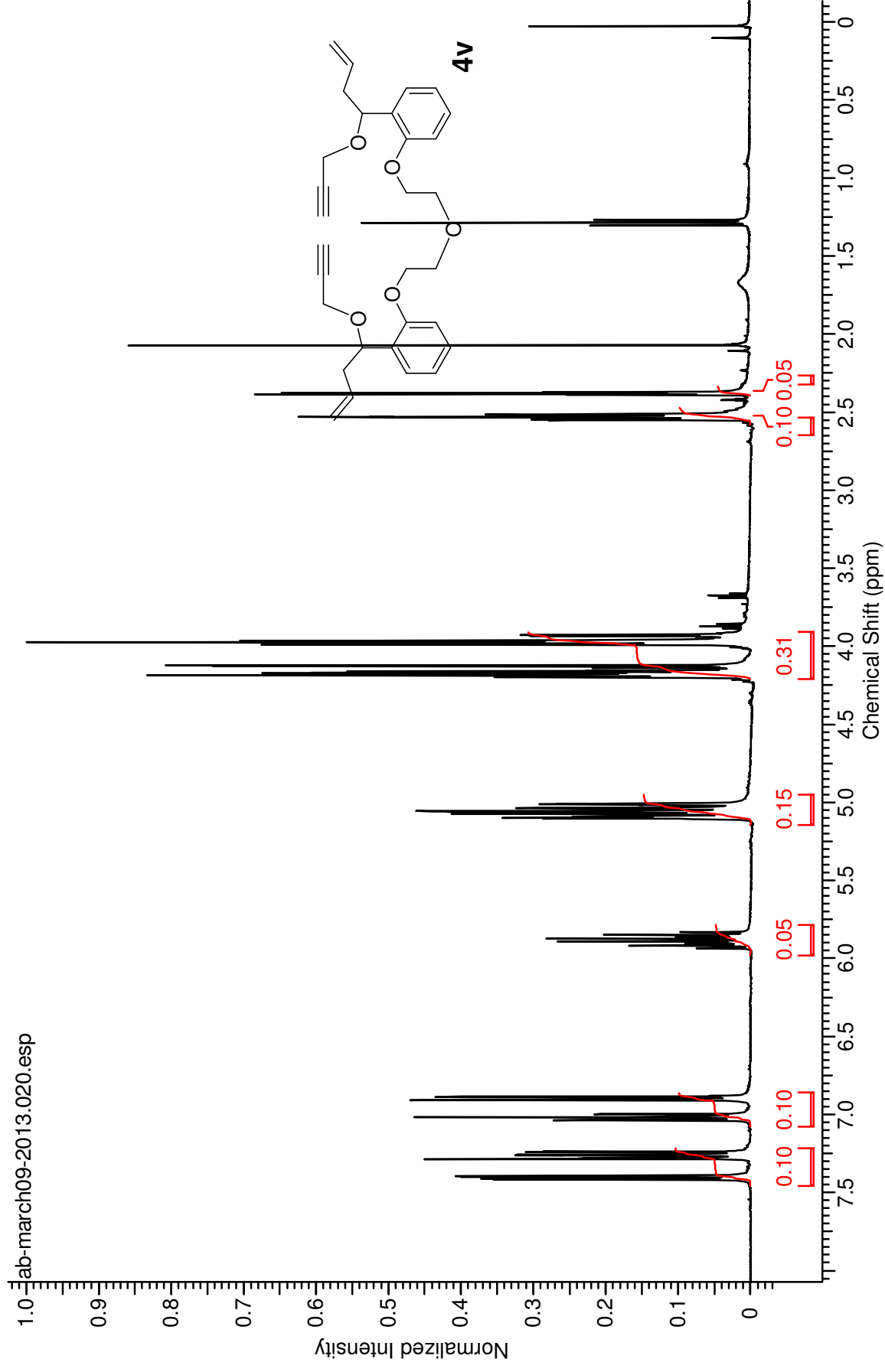


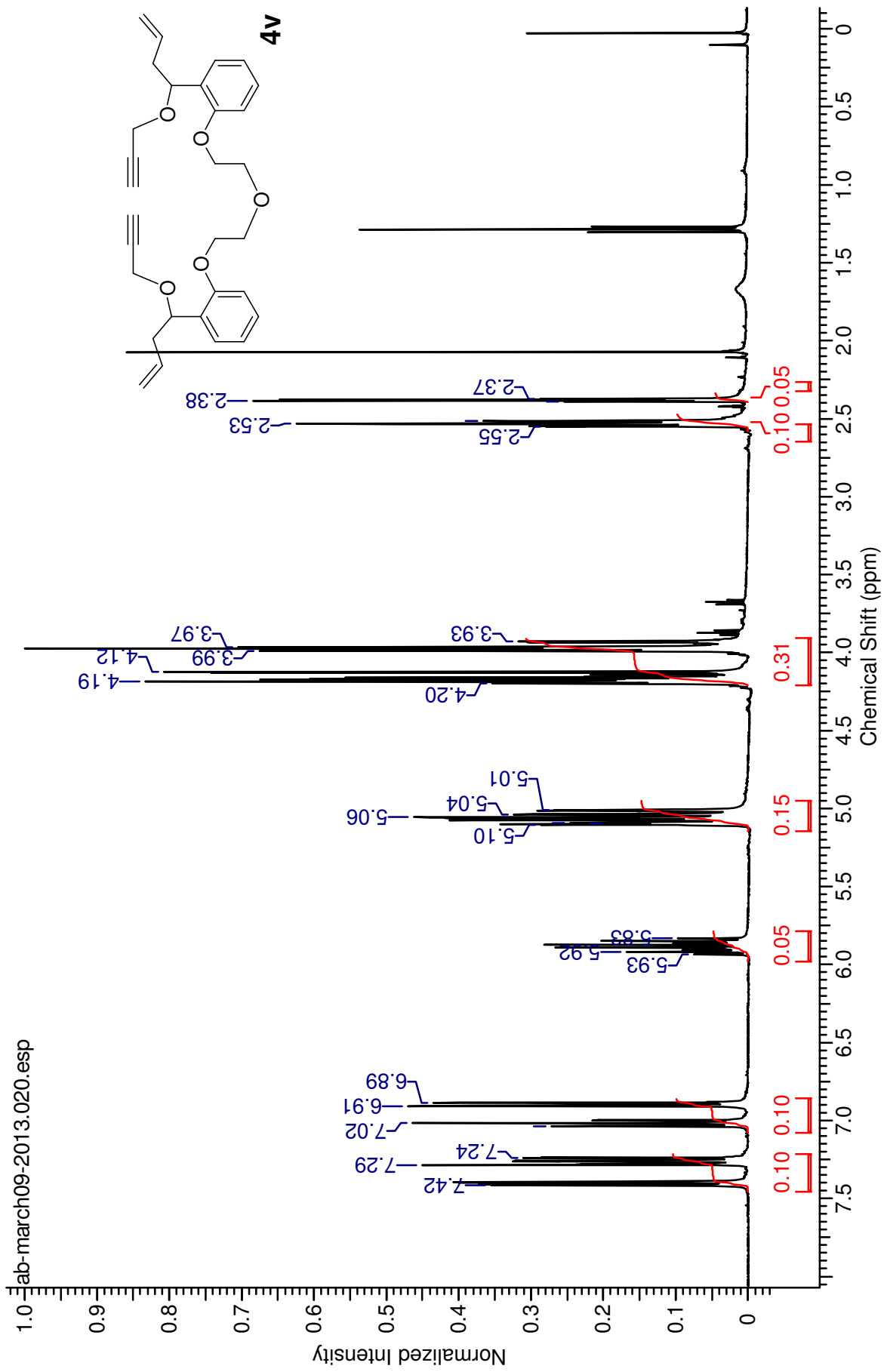


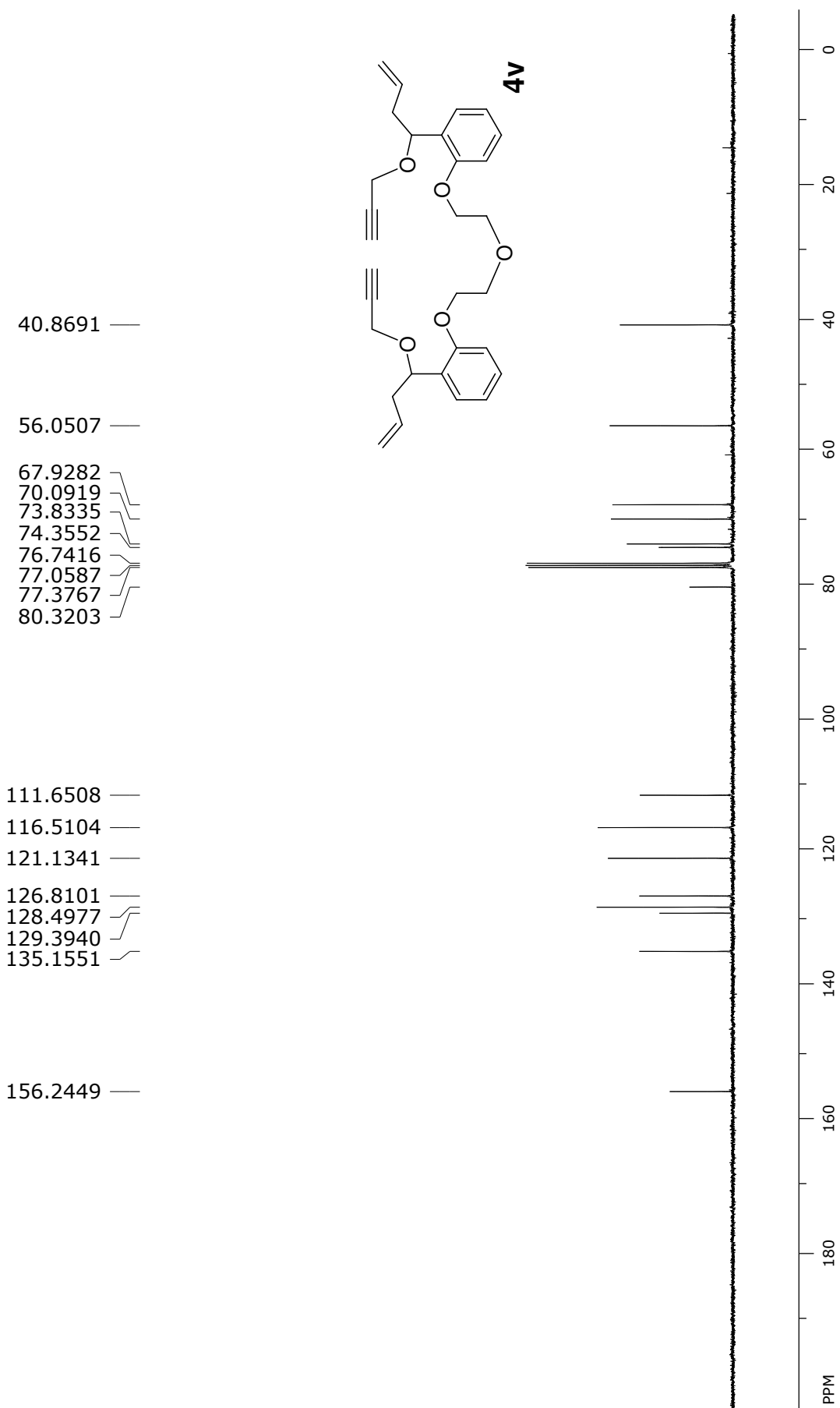
SpinWorks 3: nm1152a



1.0-ab-march09-2013.020.esp



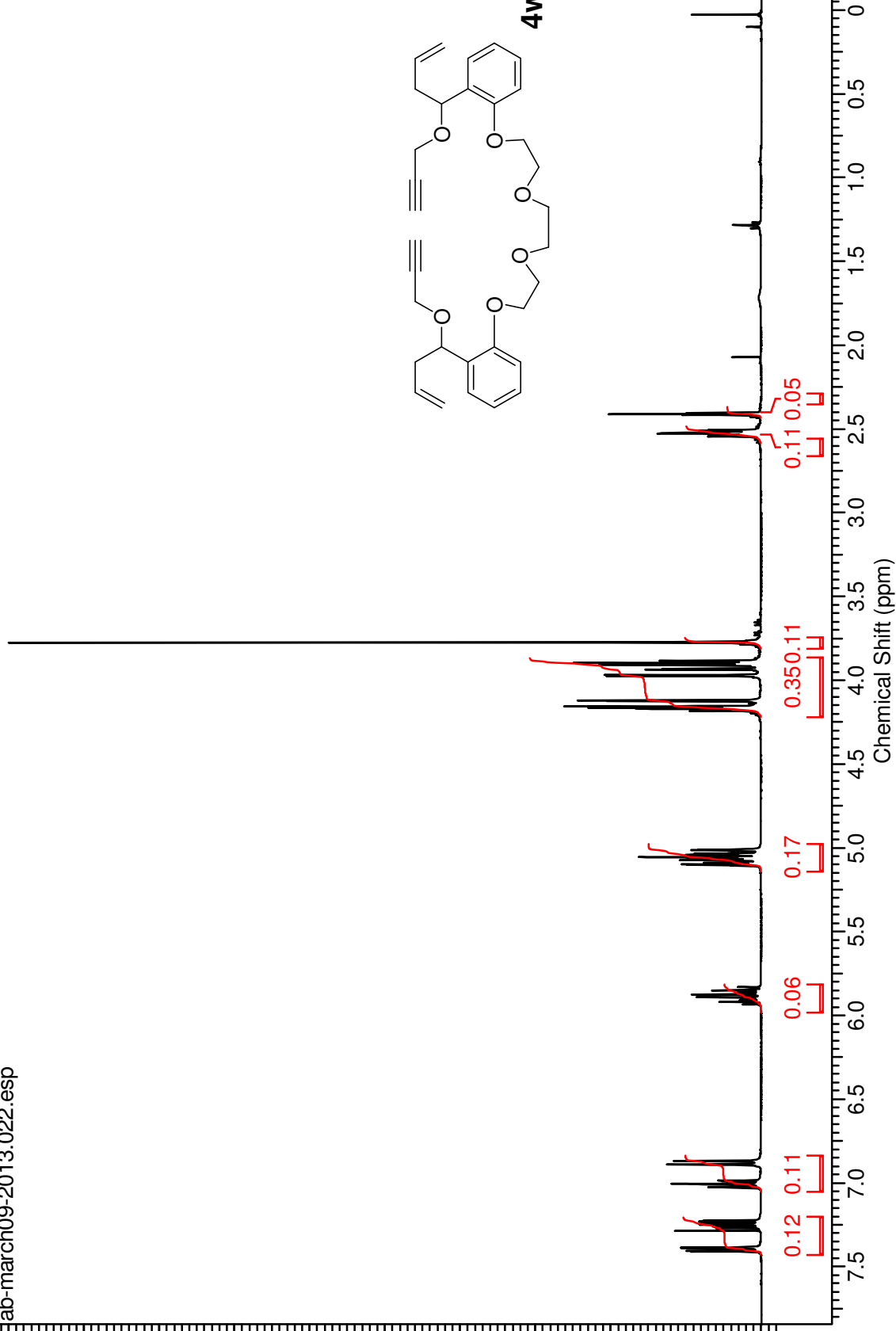


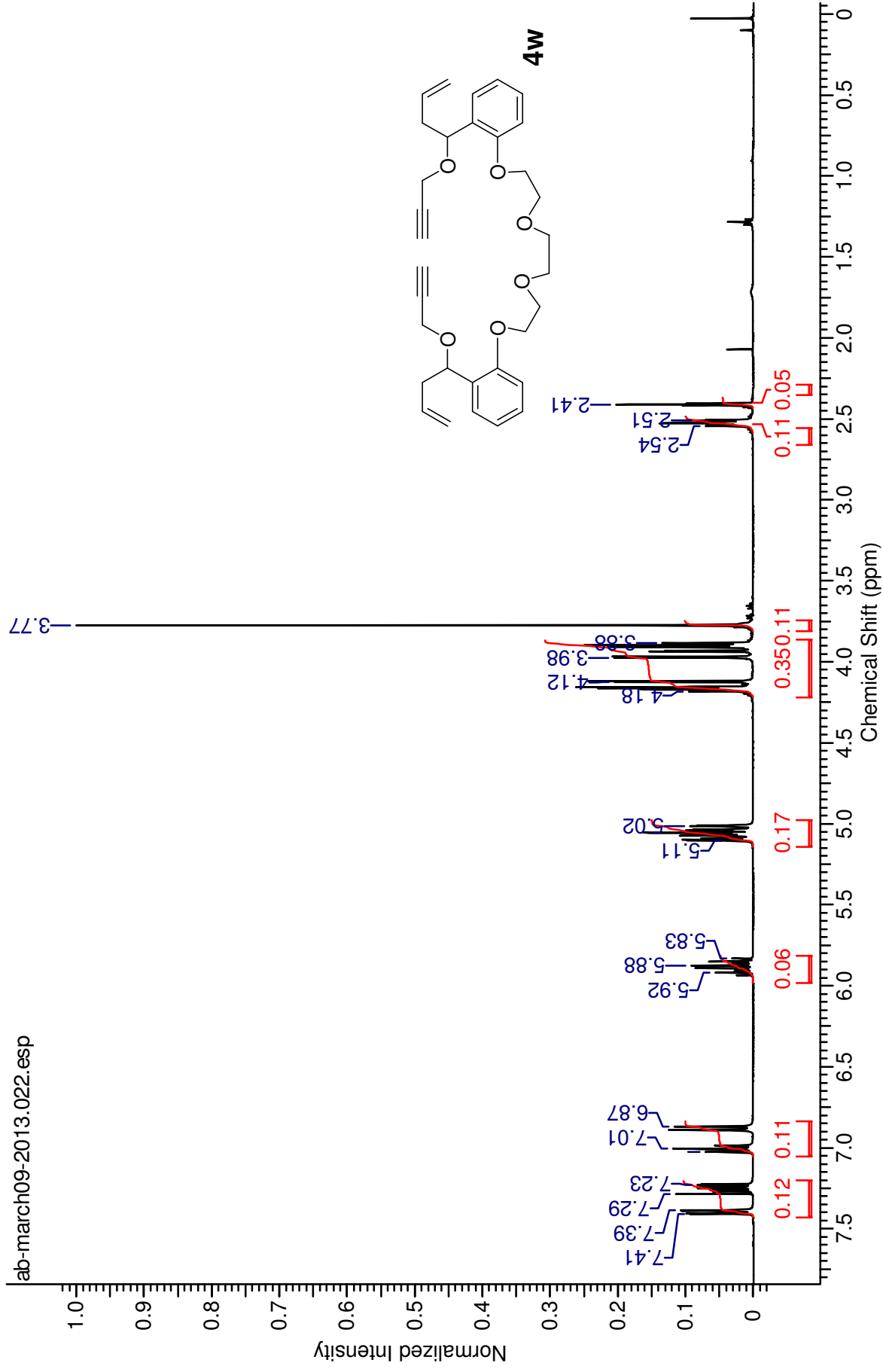


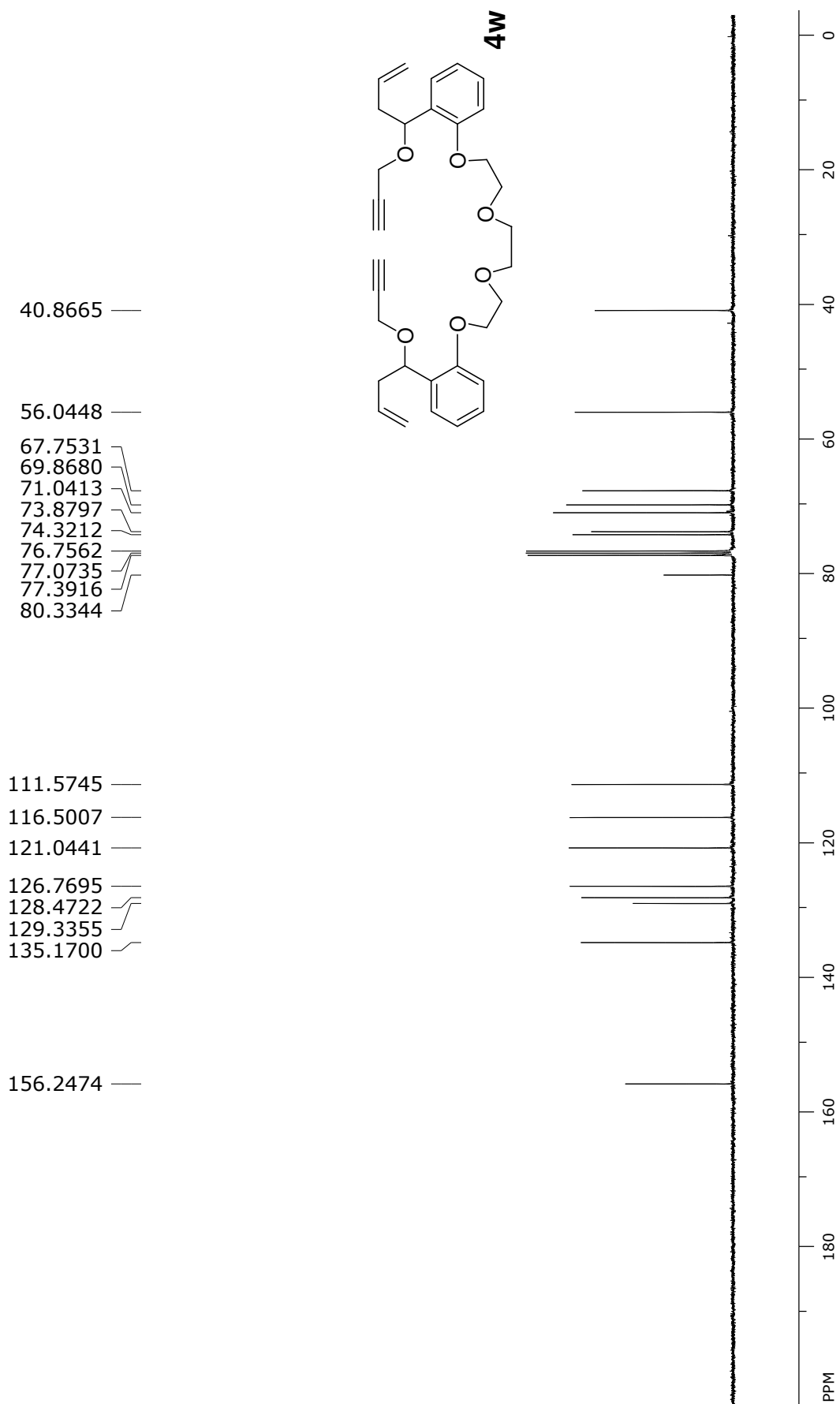
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0

lab-march09-2013.022.esp

Normalized Intensity

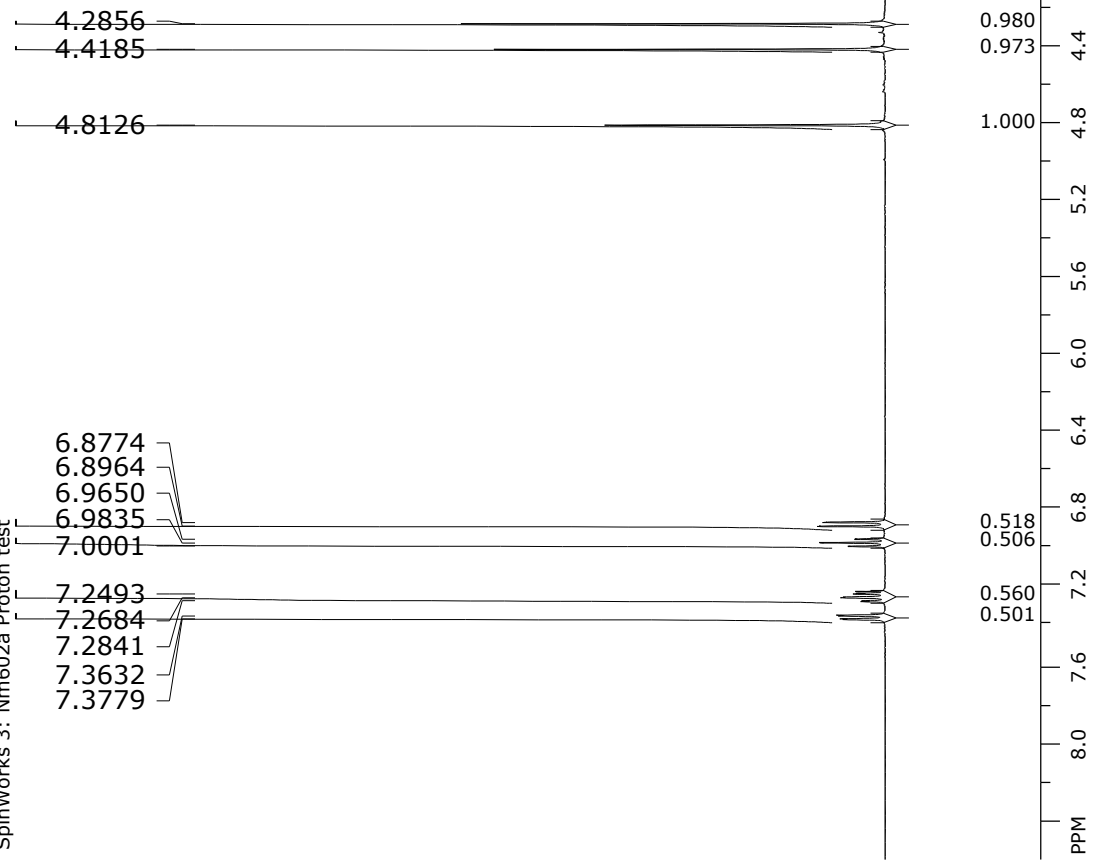
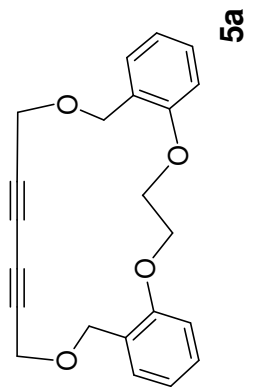


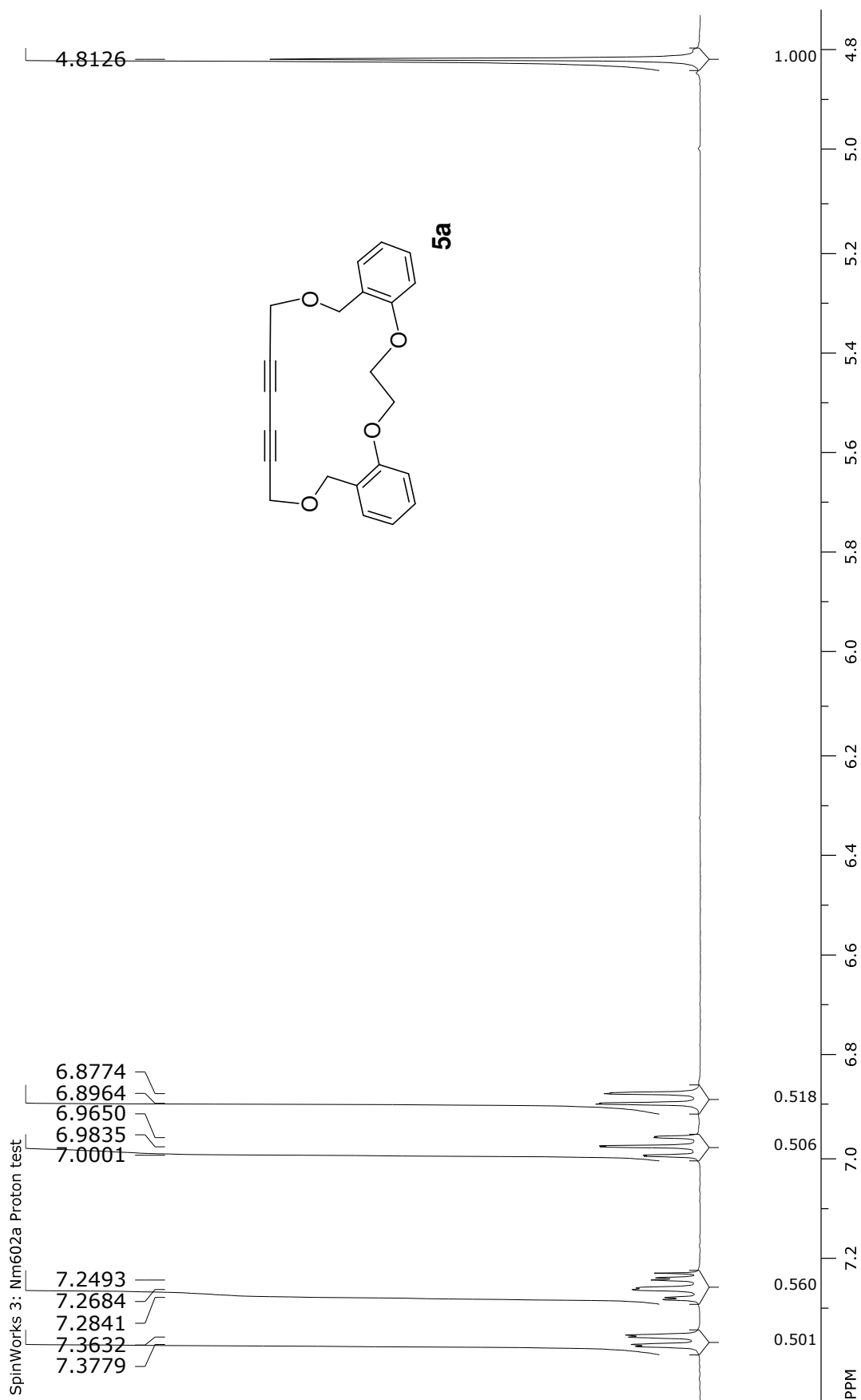




SpinWorks 3: Nm602a Proton test

-0.0112 —

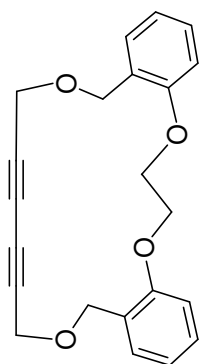
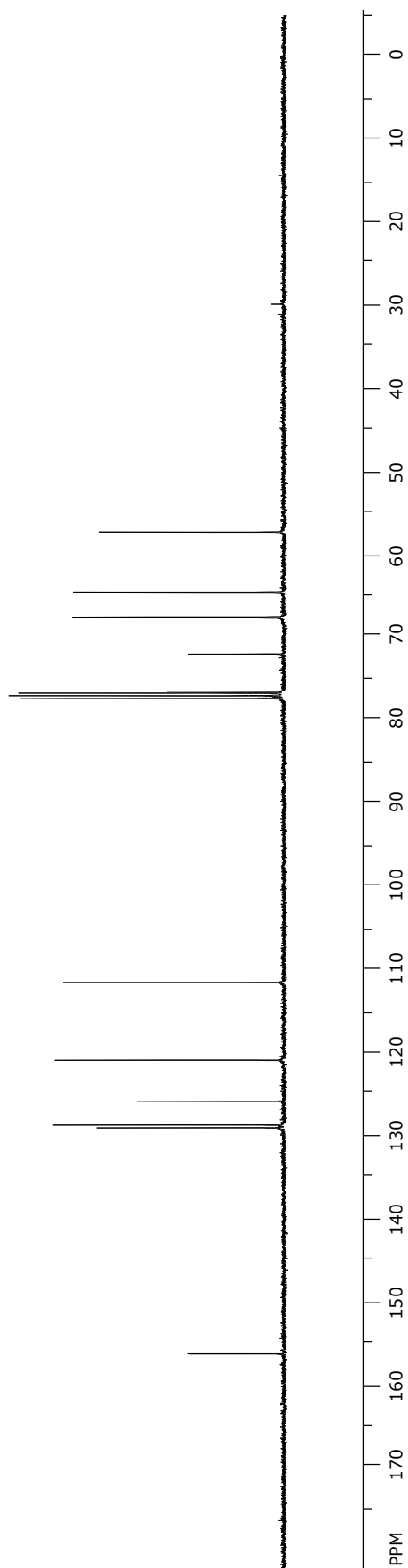




57.2842 —
64.5510 —
67.6328 —
72.1113 —
76.5289 —
76.7552 —
77.0725 —
77.3899 —

111.7332 —
121.1406 —
126.1087 —
128.9840 —
129.3285 —

156.5865 —

**5a**

SpinWorks 3: Nm602b Carbon test

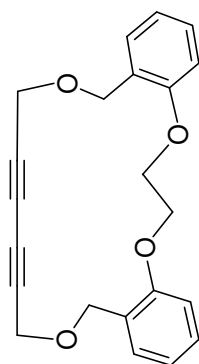
76.5289
76.7552
77.0725
77.3899

72.1113

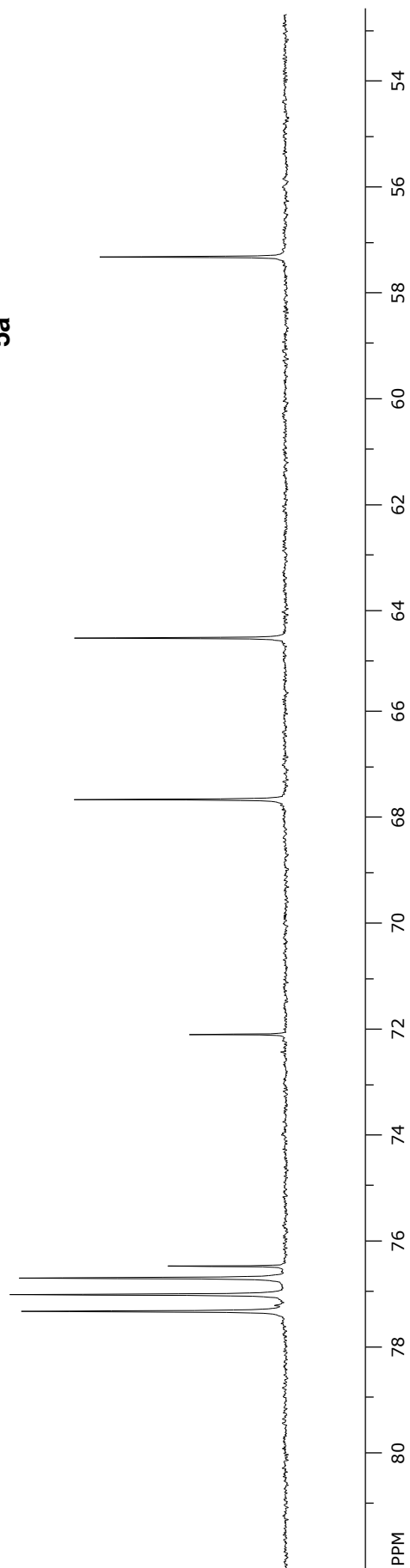
67.6328

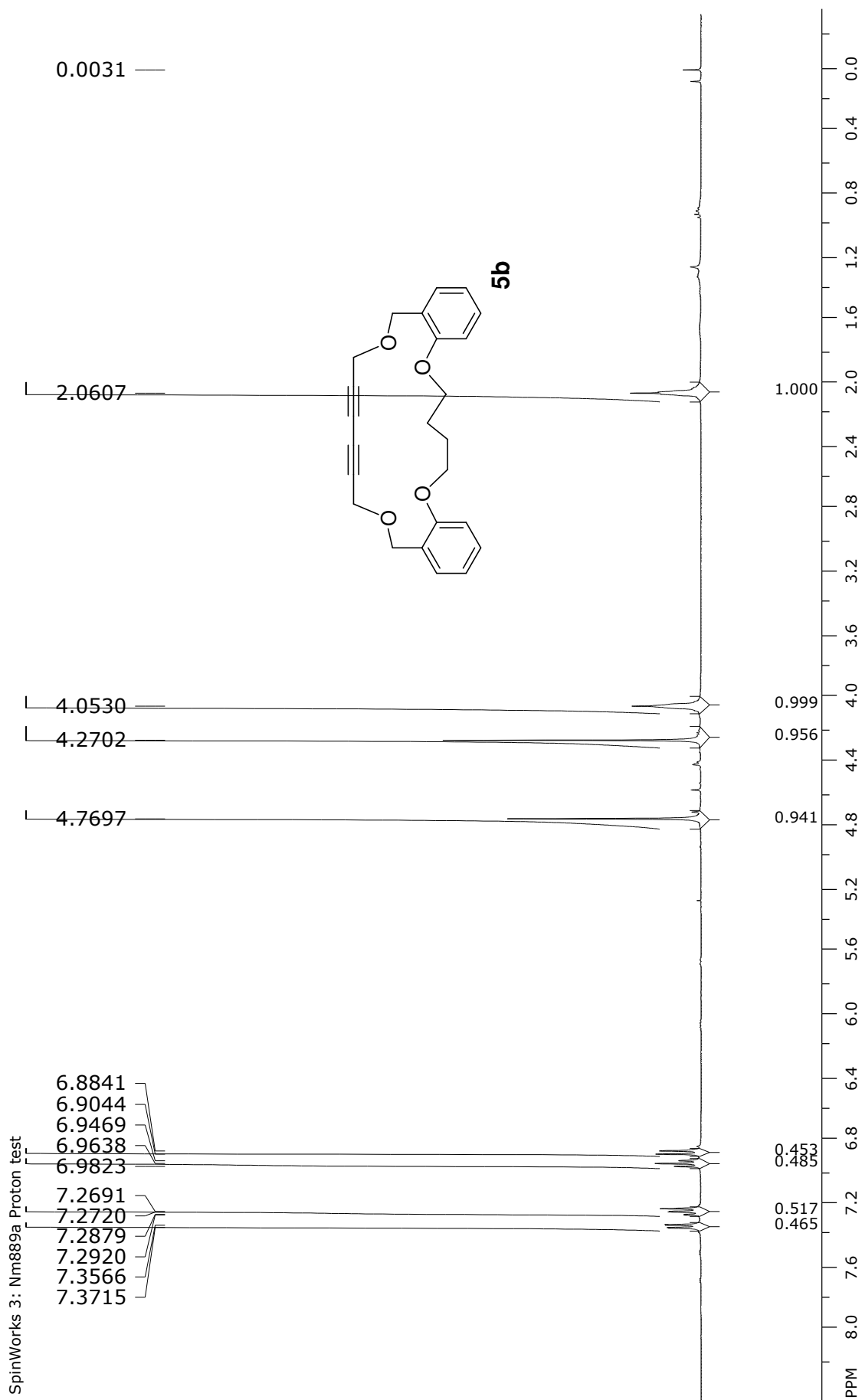
64.5510

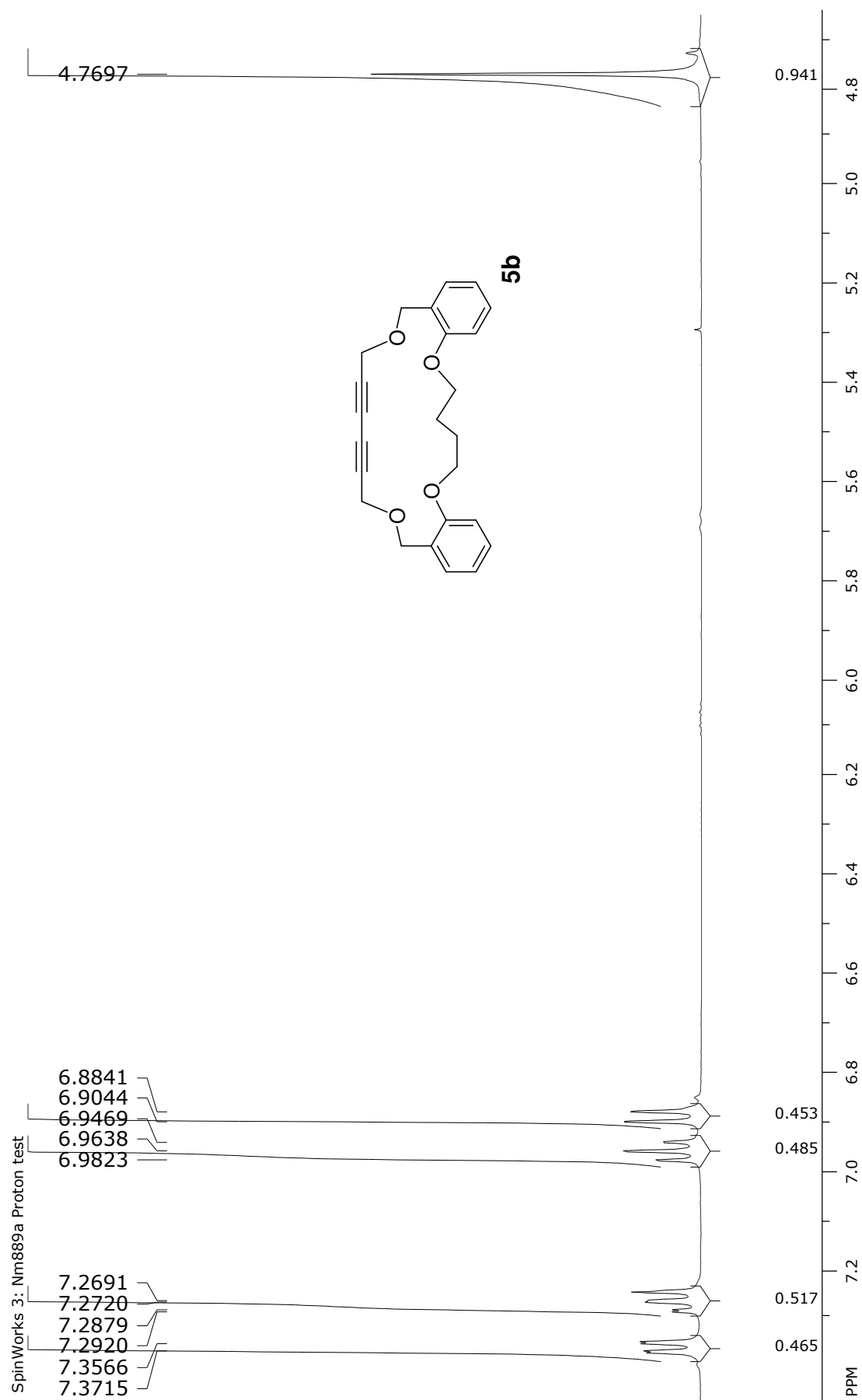
57.2842



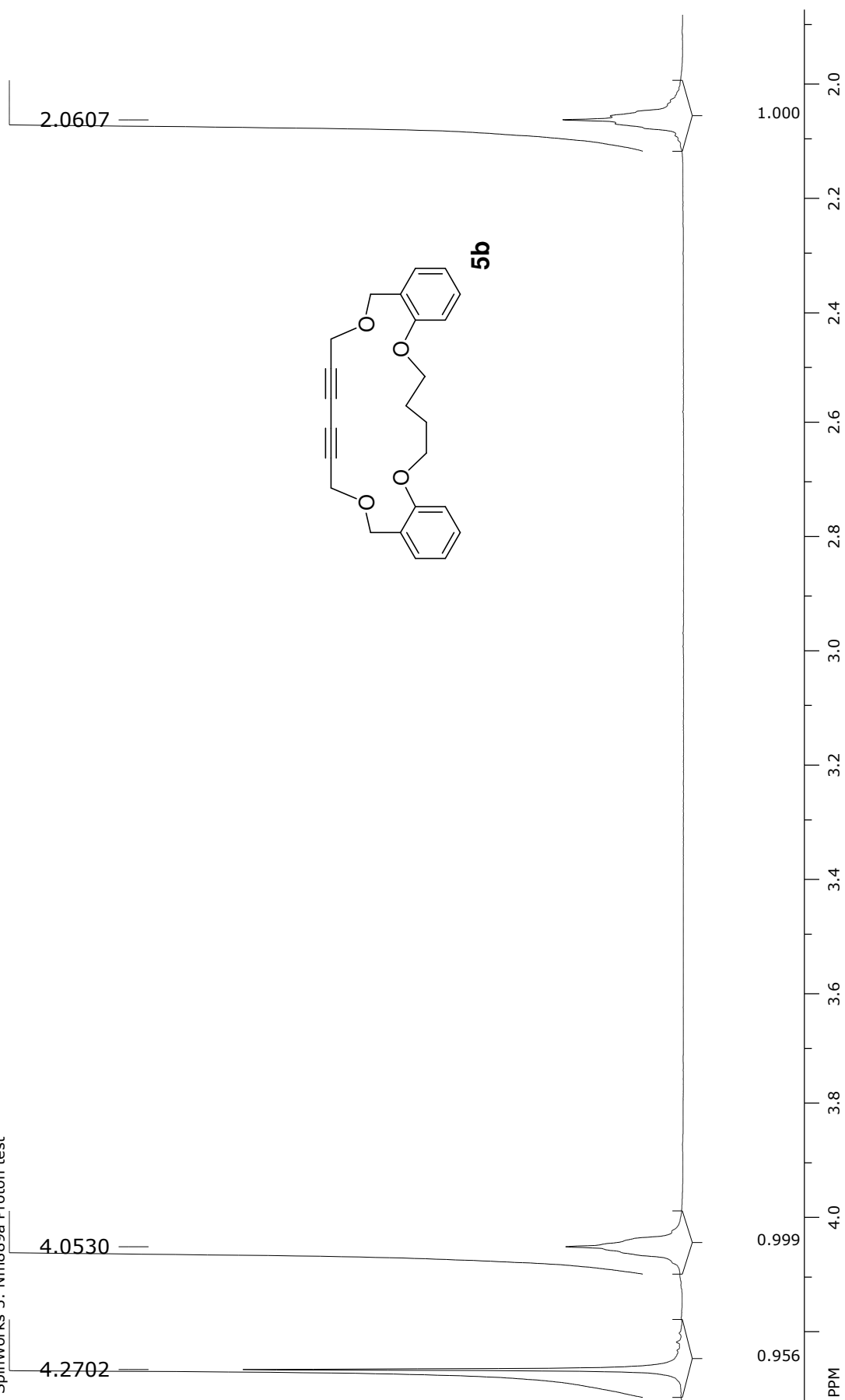
5a







SpinWorks 3: Nm889a Proton test



26.5018 —

57.1684 —

64.9459 —

68.4025 —

70.8657 —

75.3853 —

76.7623 —

77.0799 —

77.3973 —

111.4407 —

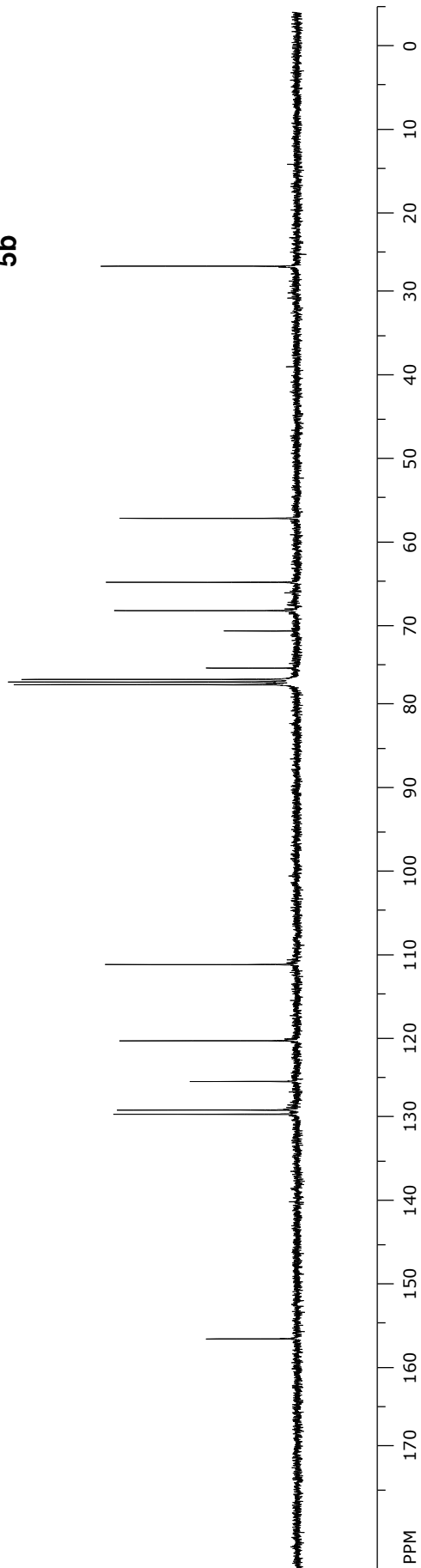
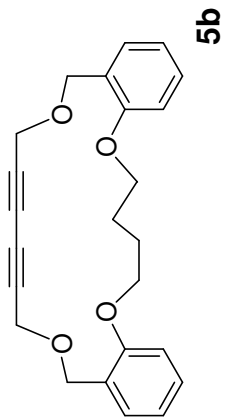
120.7099 —

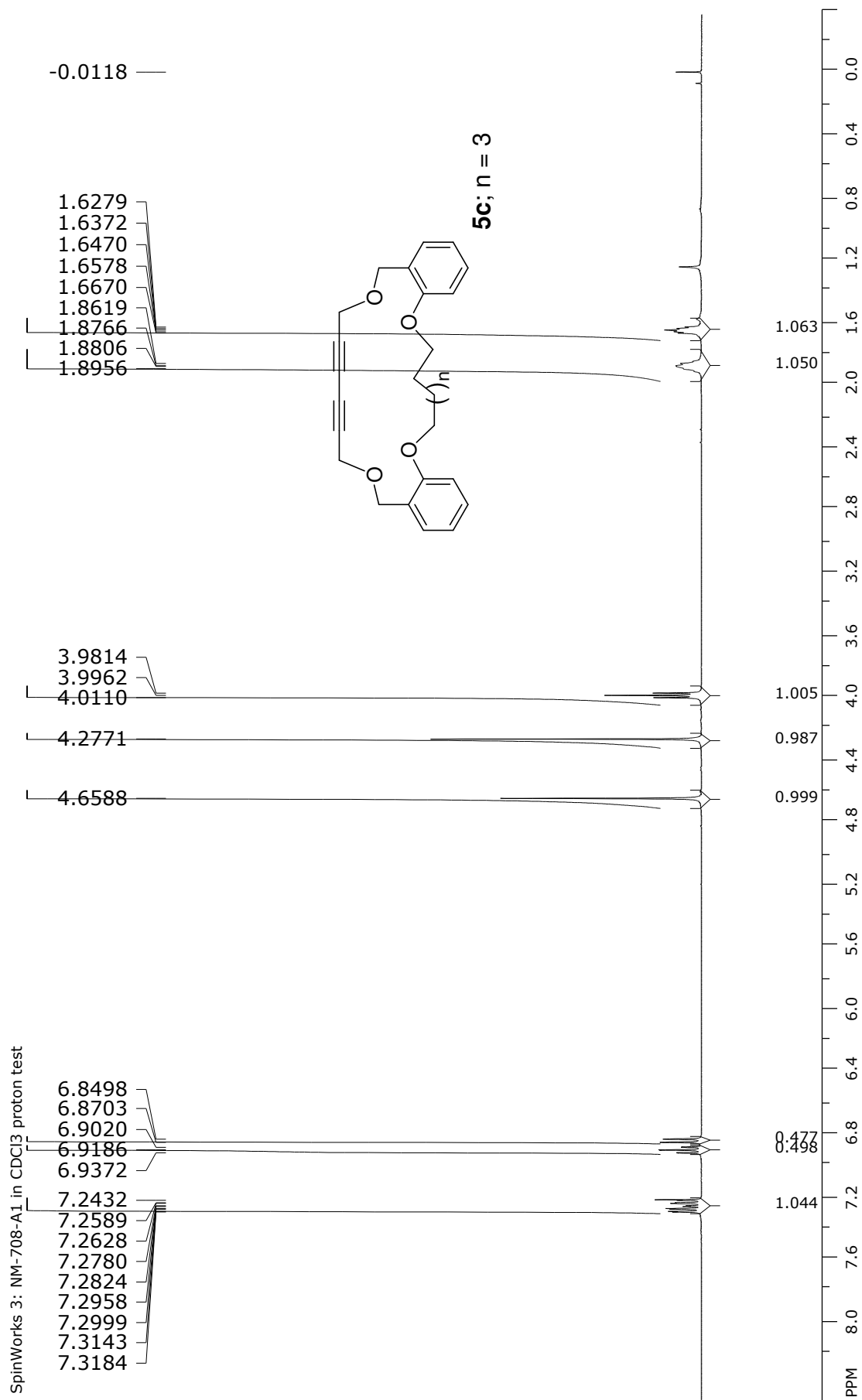
125.6660 —

129.1401 —

129.6448 —

156.9682 —



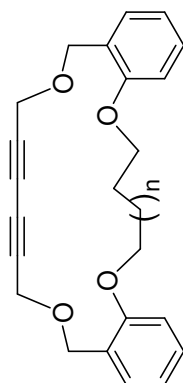


SpinWorks 3: NM-708-A1 in CDCl3 proton test

7.2432
7.2589
7.2628
7.2780
7.2824
7.2958
7.2999

6.8498
6.8703
6.9020
6.9186
6.9372

4.6588



5c; n = 3

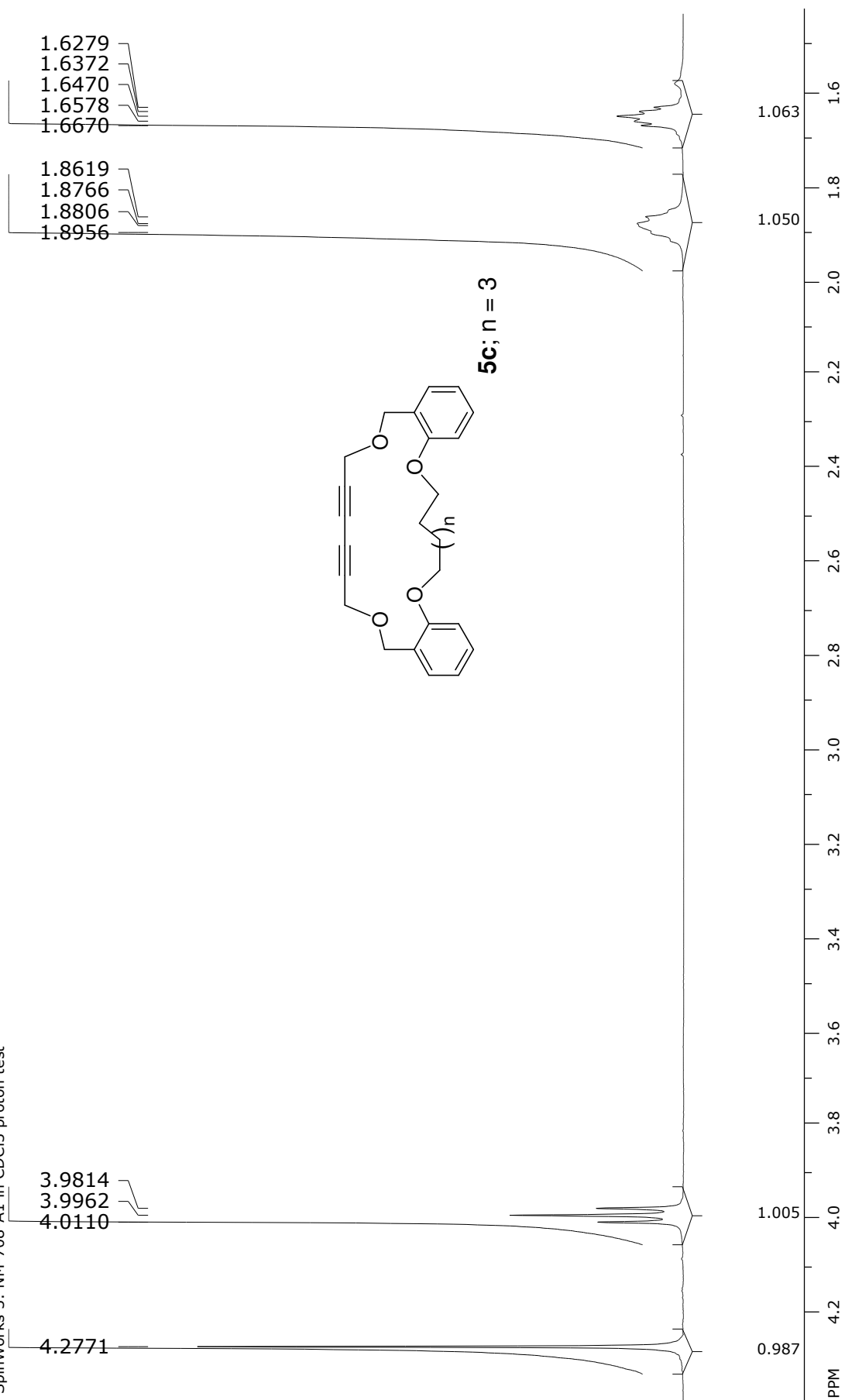
0.477
0.498

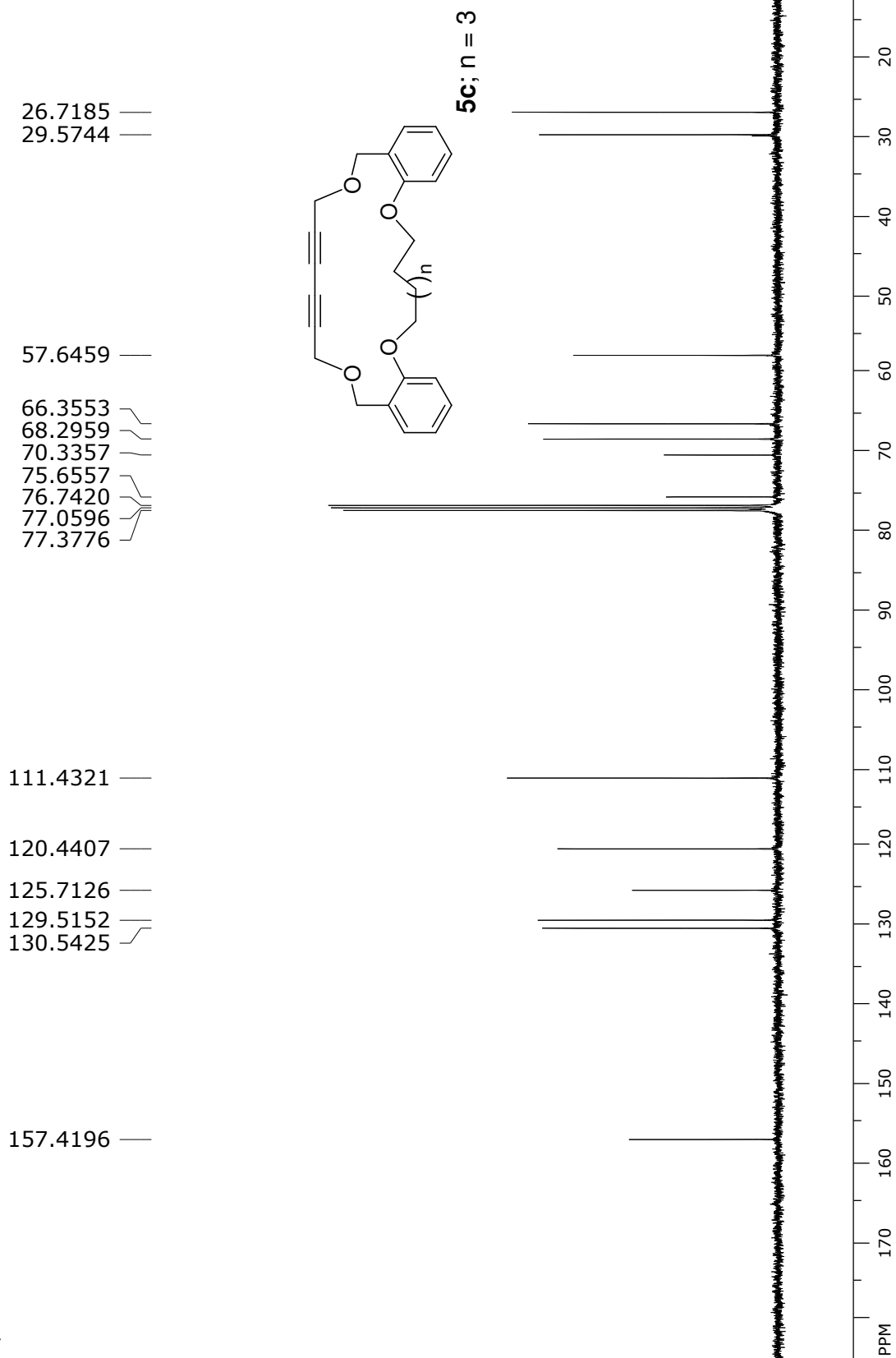
1.044

0.999

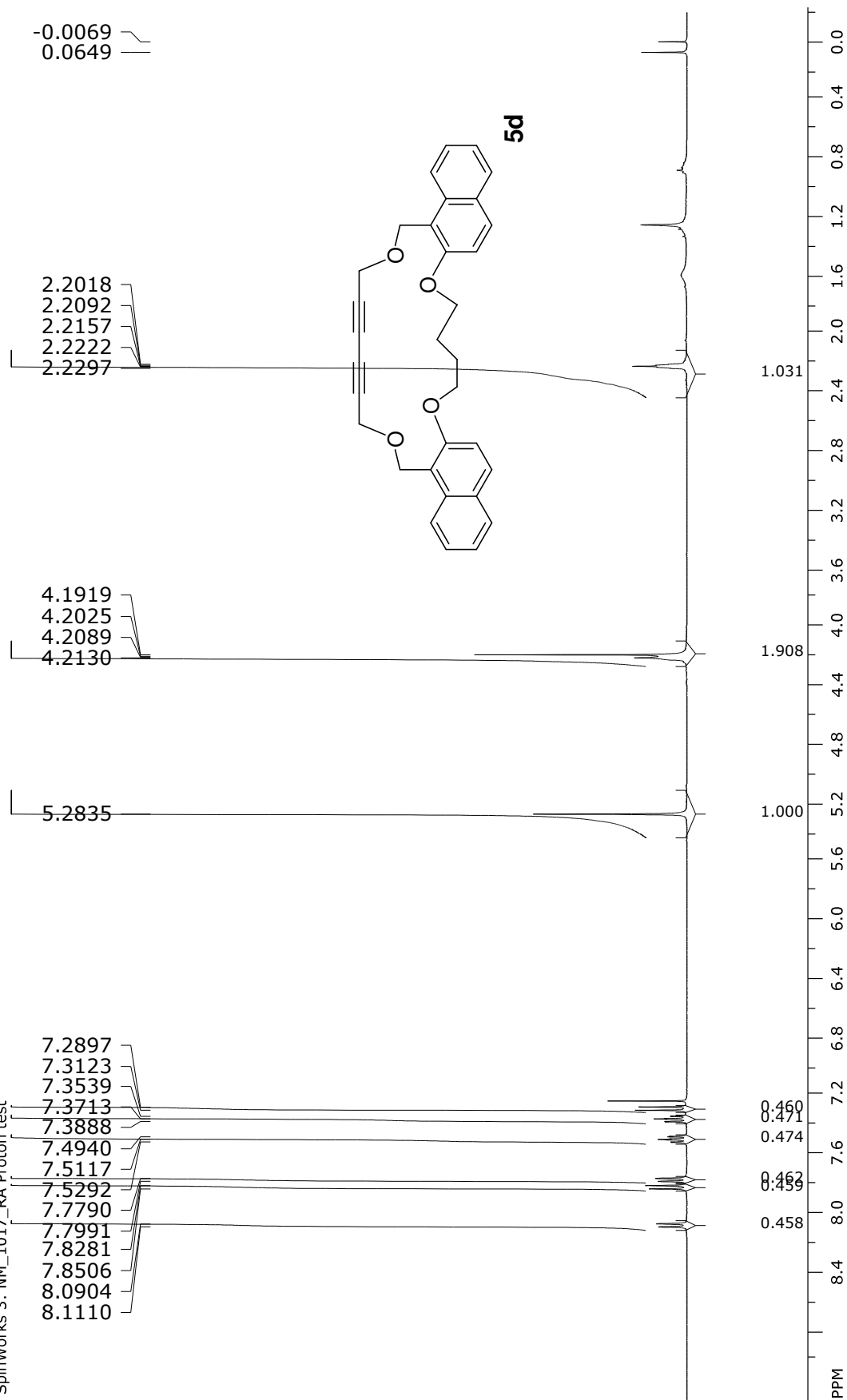
PPM

SpinWorks 3: NM-708-A1 in CDCl3 proton test

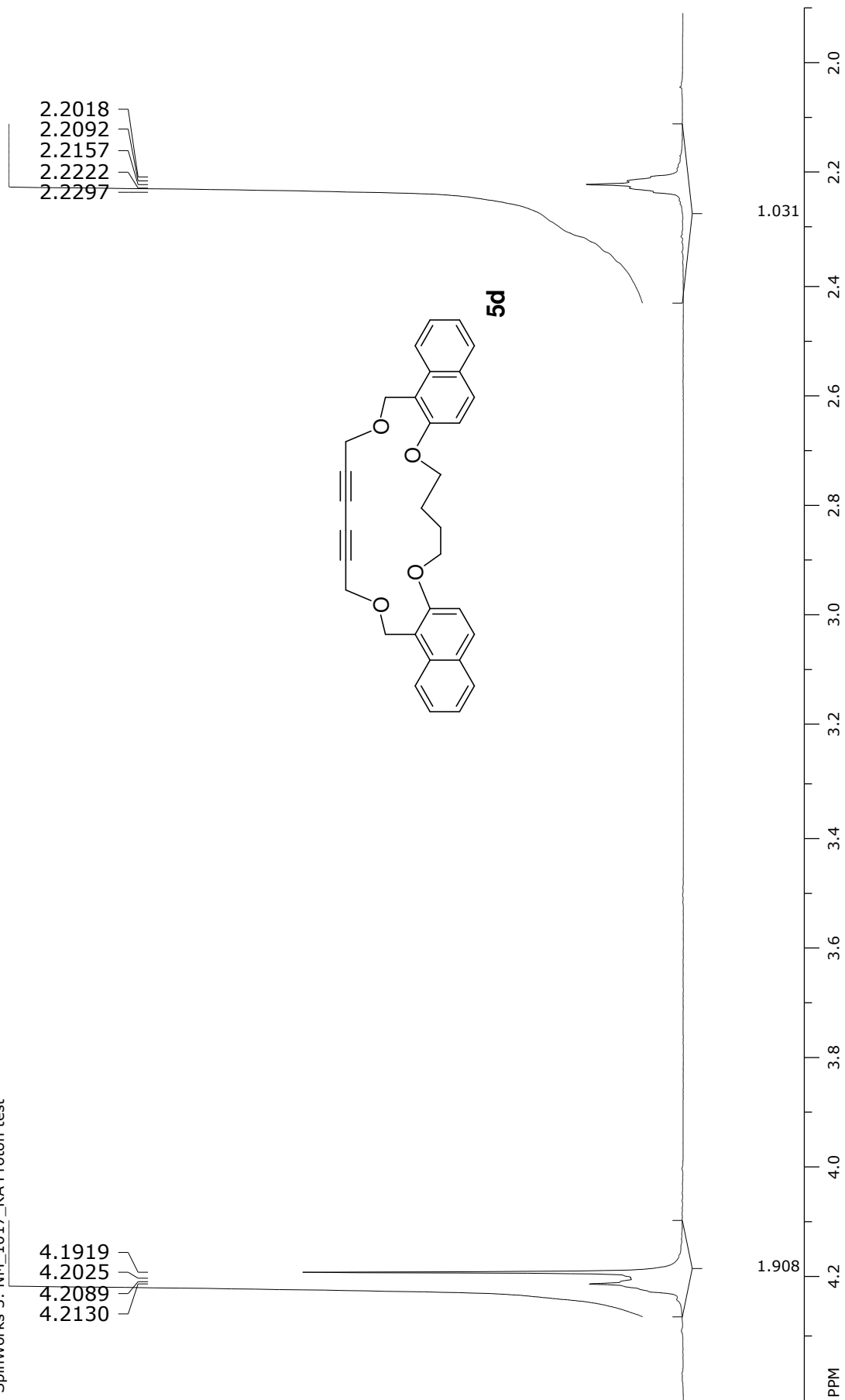




SpinWorks 3: NM_1017_RA Proton test



SpinWorks 3: NM_1017_RA Proton test



SpinWorks 3: NM 1017 RA C13 test

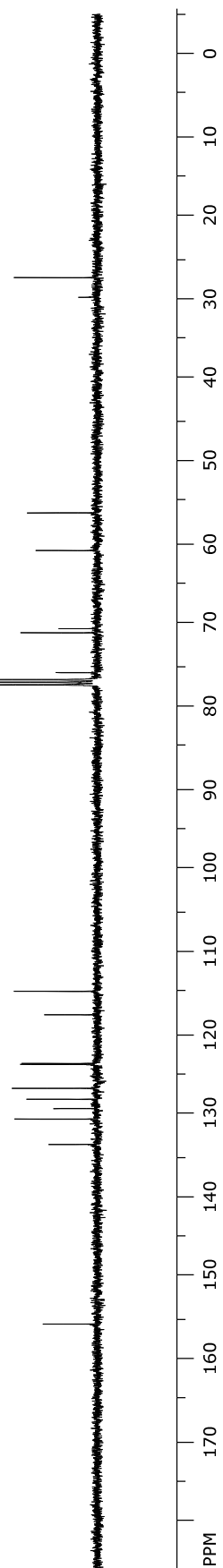
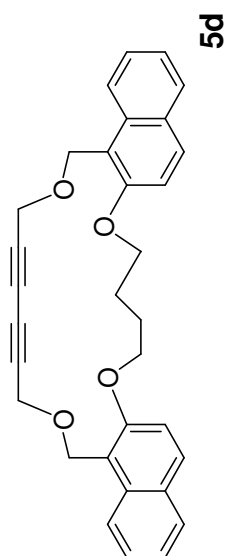
155.9126

115.0219
117.9047

123.8907
124.0078
126.9471
128.2885
129.4262
130.7239
133.8380

56.2206
60.8517
70.4469
70.9559
75.8565
76.7239
77.0410
77.3590

27.3134



SpinWorks 3: NM 1017 RA C13 test

133.8380 —

130.7239 —

129.4262 —

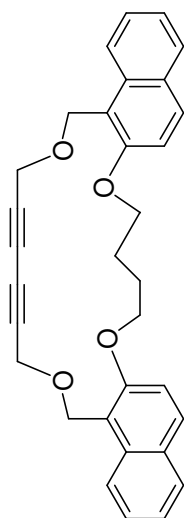
128.2885 —

126.9471 —

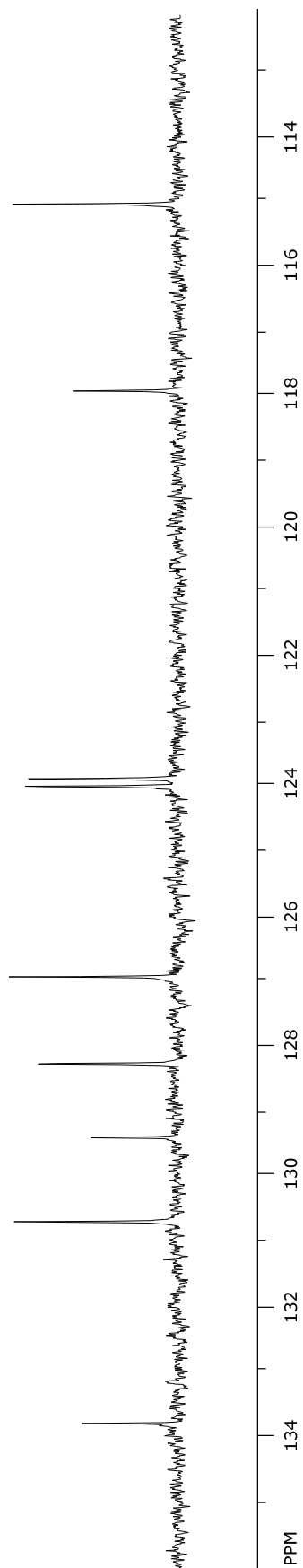
124.0078 —
123.8907 —

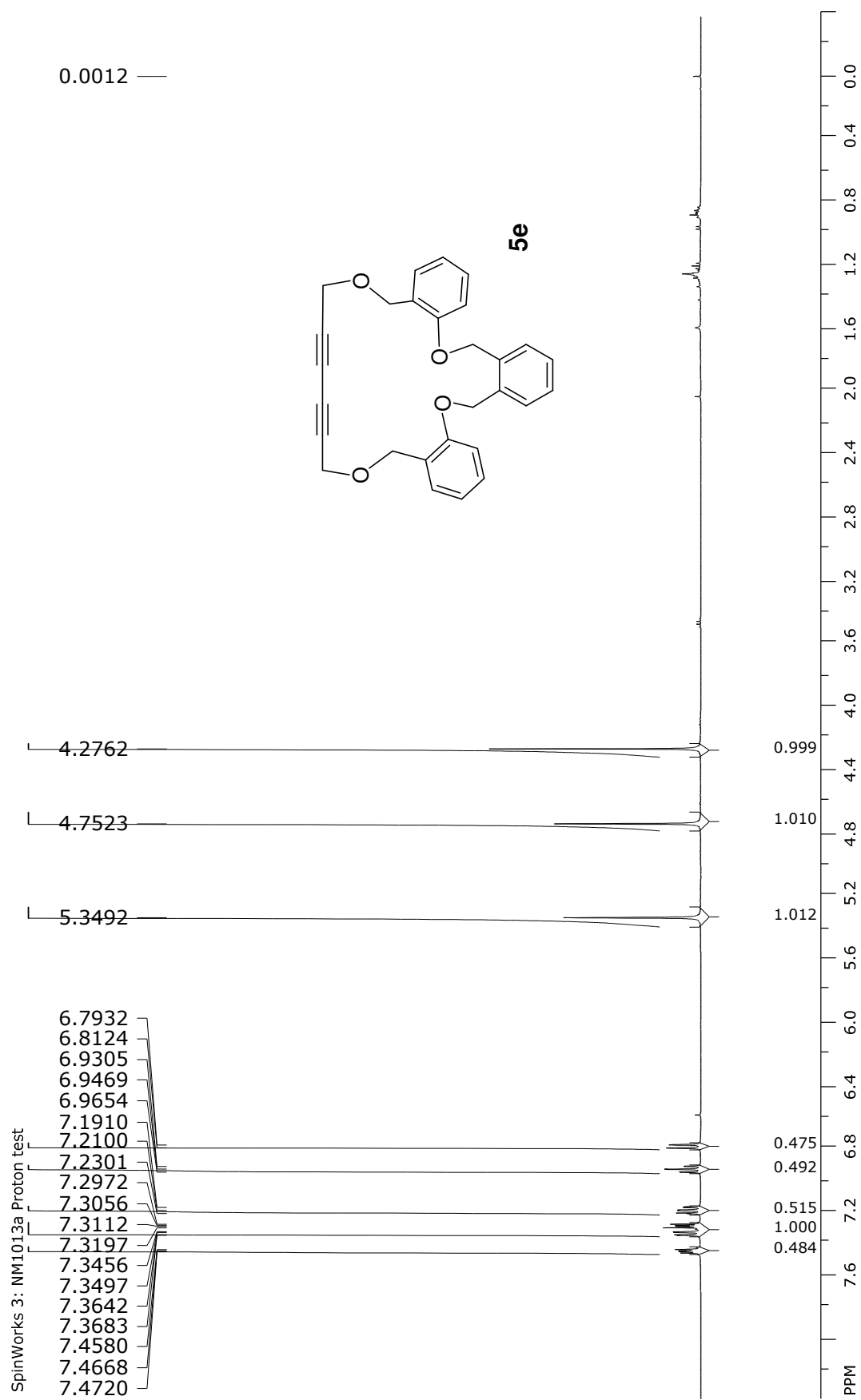
117.9047 —

115.0219 —



5d





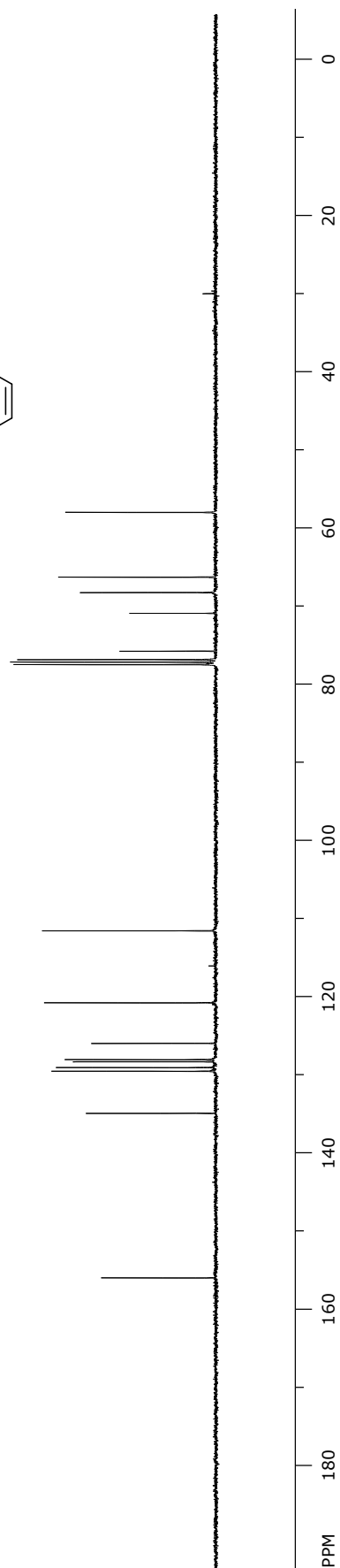
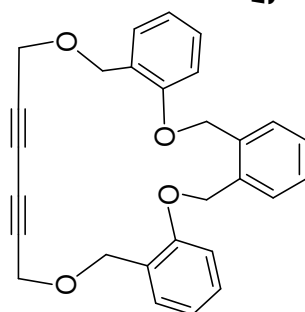
57.8526
66.1749
68.1491
70.8172
75.6876
76.7745
77.0914
77.4098

111.6166

120.8643

126.0737
128.1696
128.4357
129.1832
129.6582
135.0666

156.2074



111.6166 —

120.8643 —

126.0737 —

128.1696 —

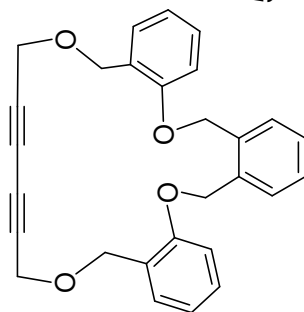
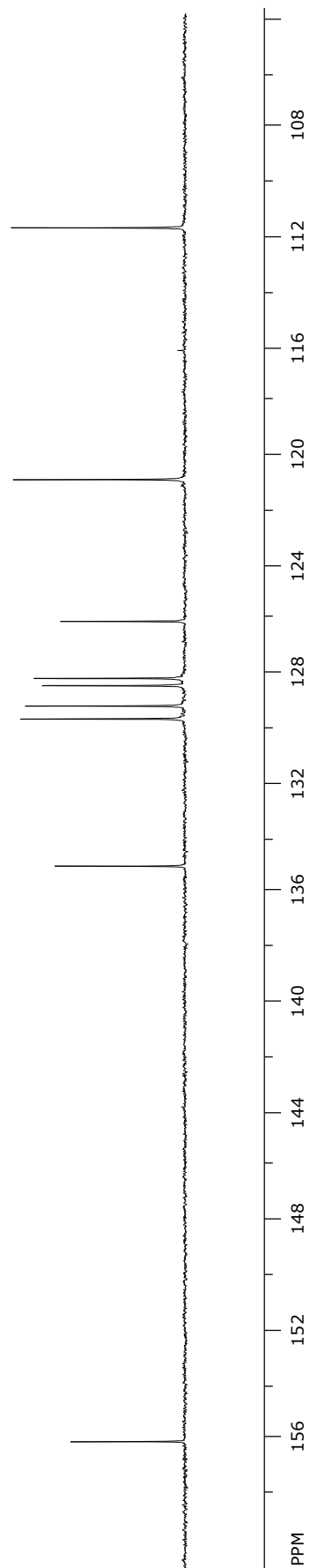
128.4357 —

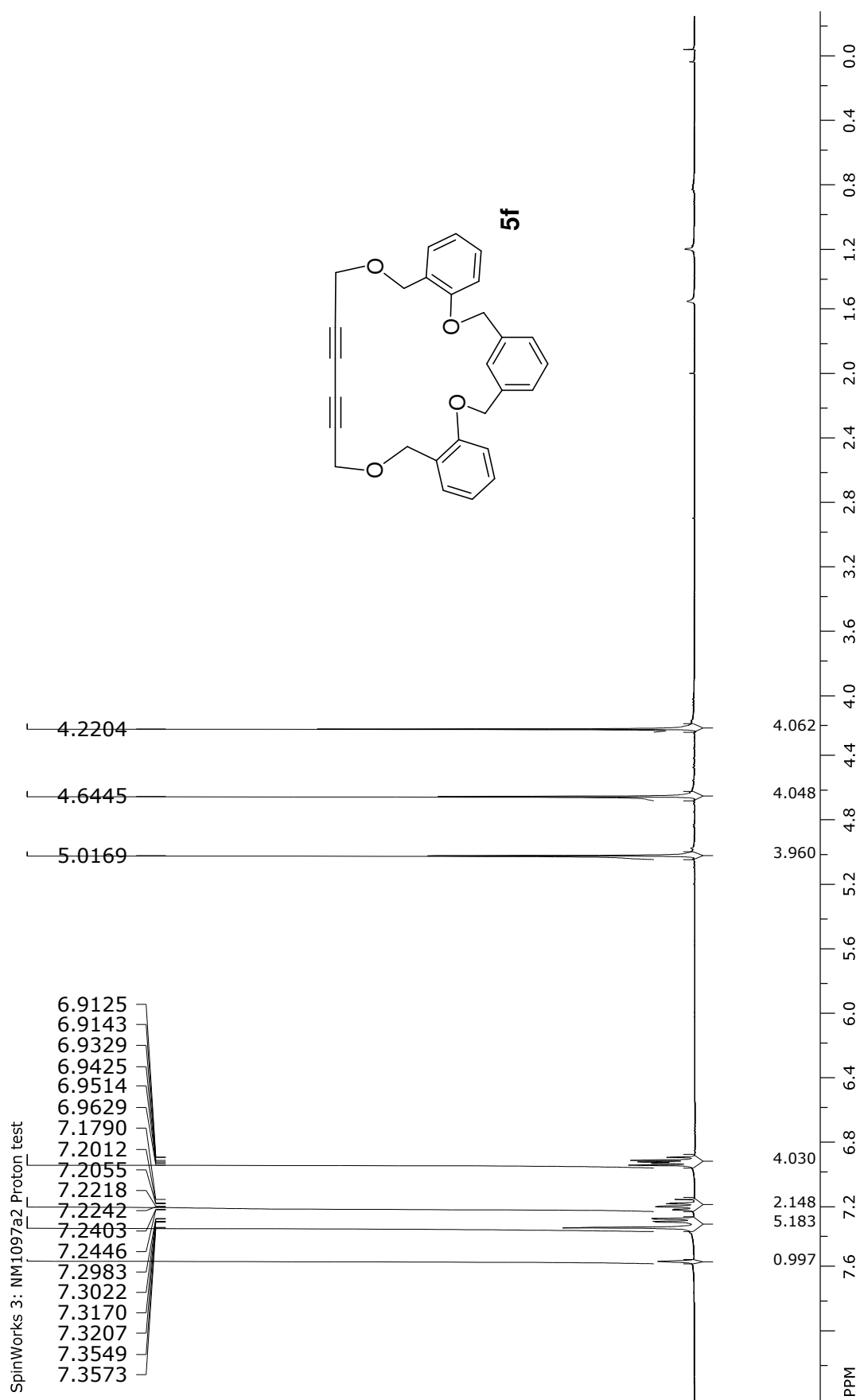
129.1832 —

129.6582 —

135.0666 —

156.2074 —

**5e**



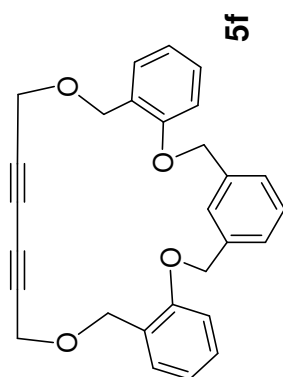
57.7730
65.9166
70.7573
70.9813
75.4884
76.7775
77.0951
77.4134

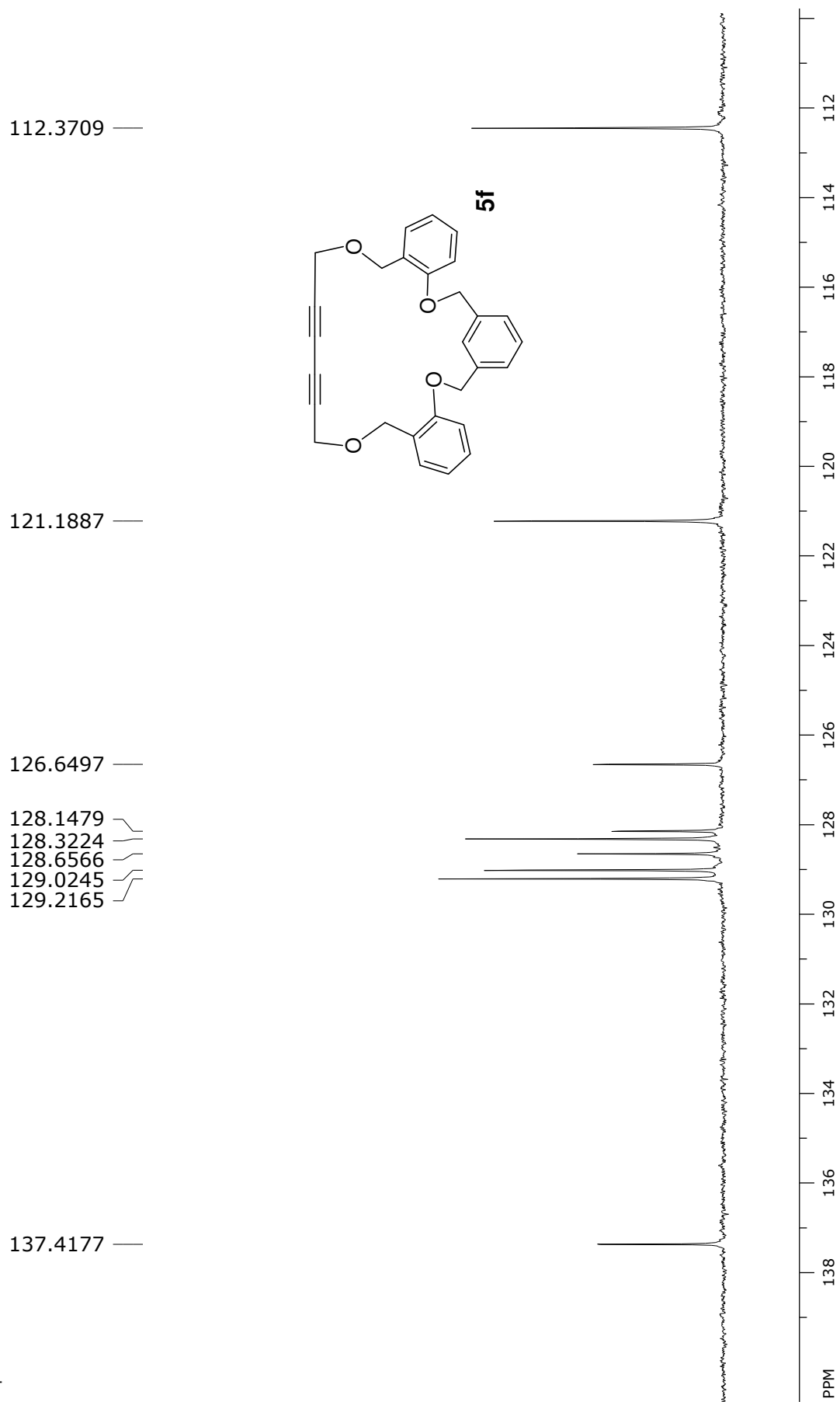
112.3709

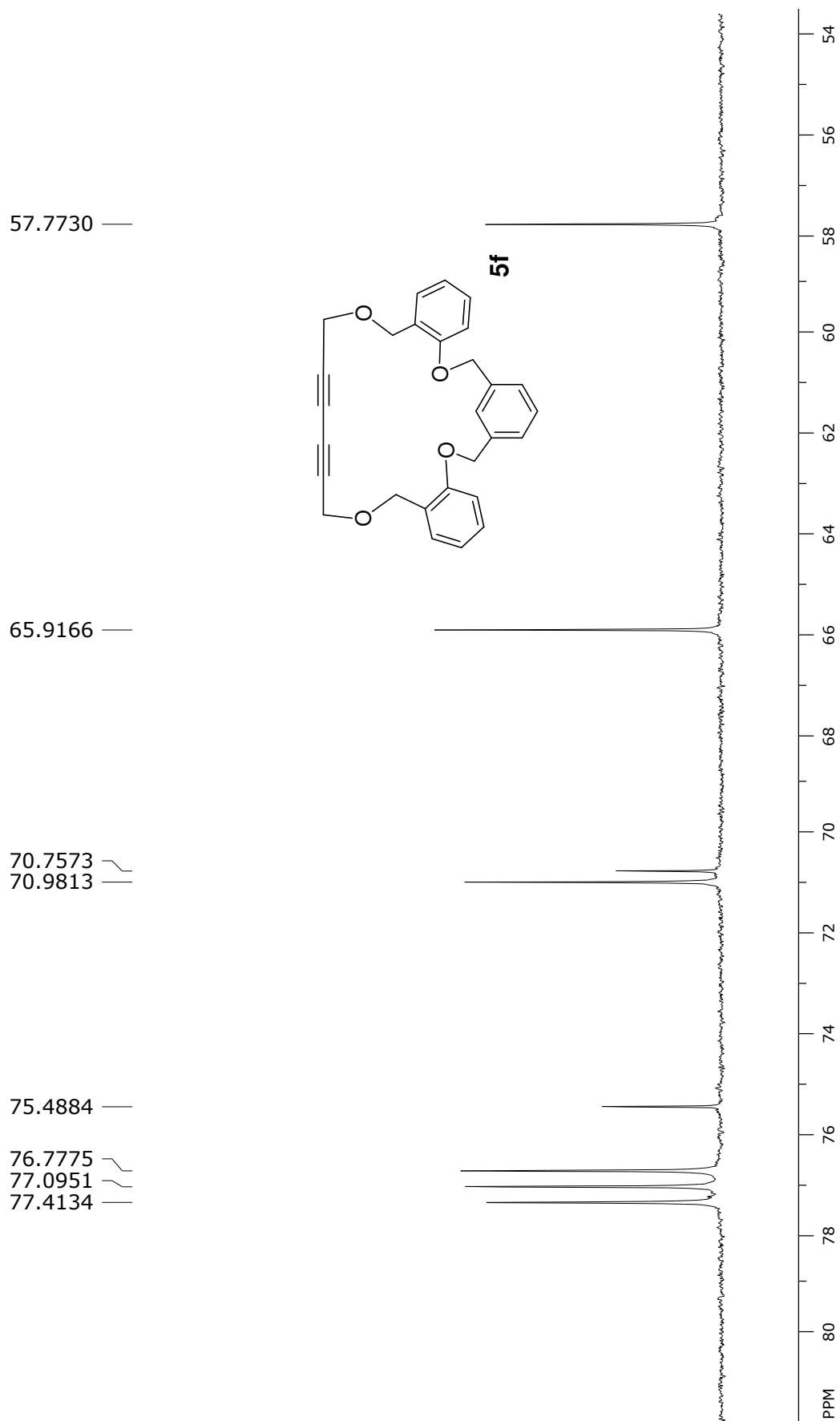
121.1887

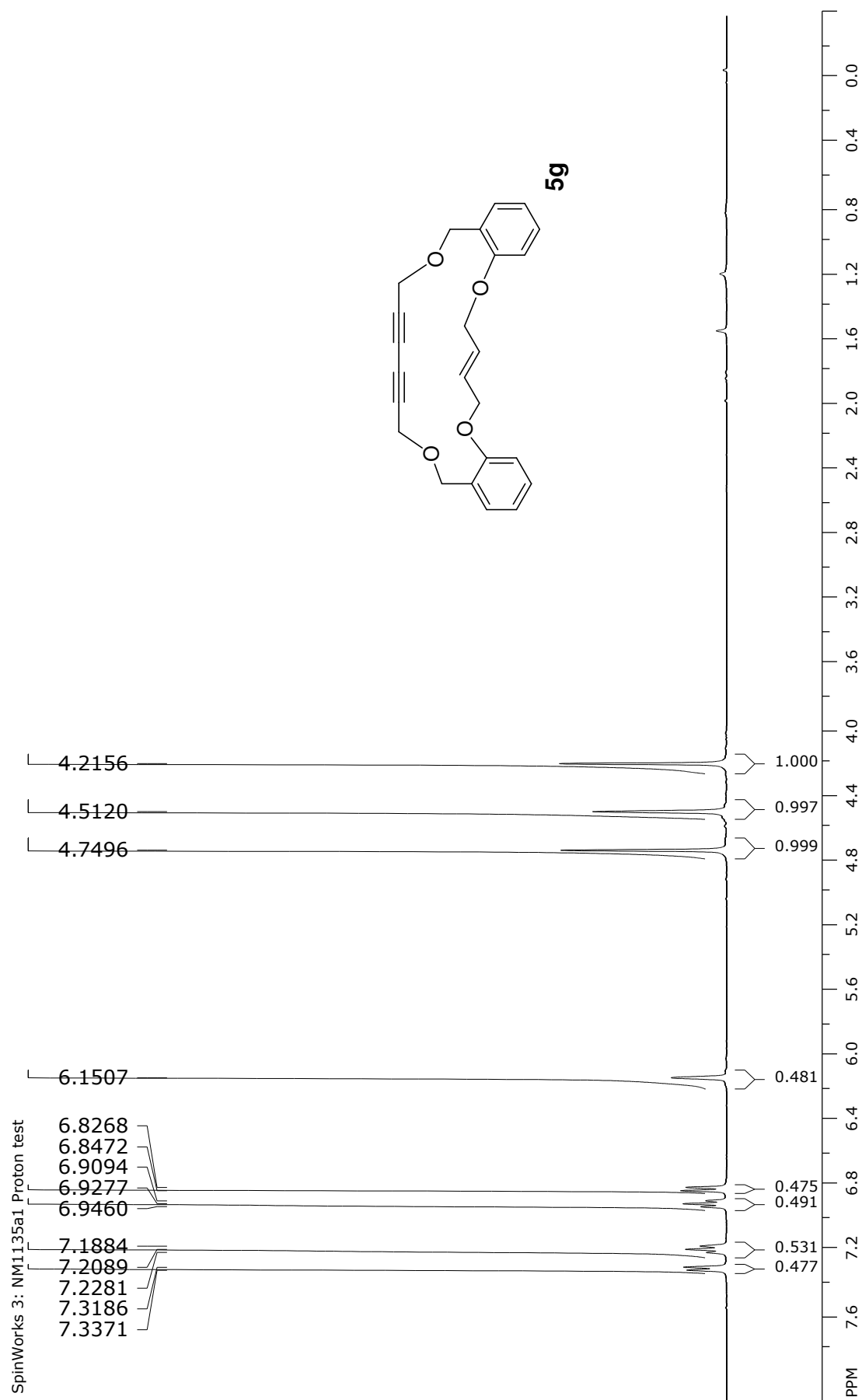
126.6497
128.1479
128.3224
128.6566
129.0245
129.2165
137.4177

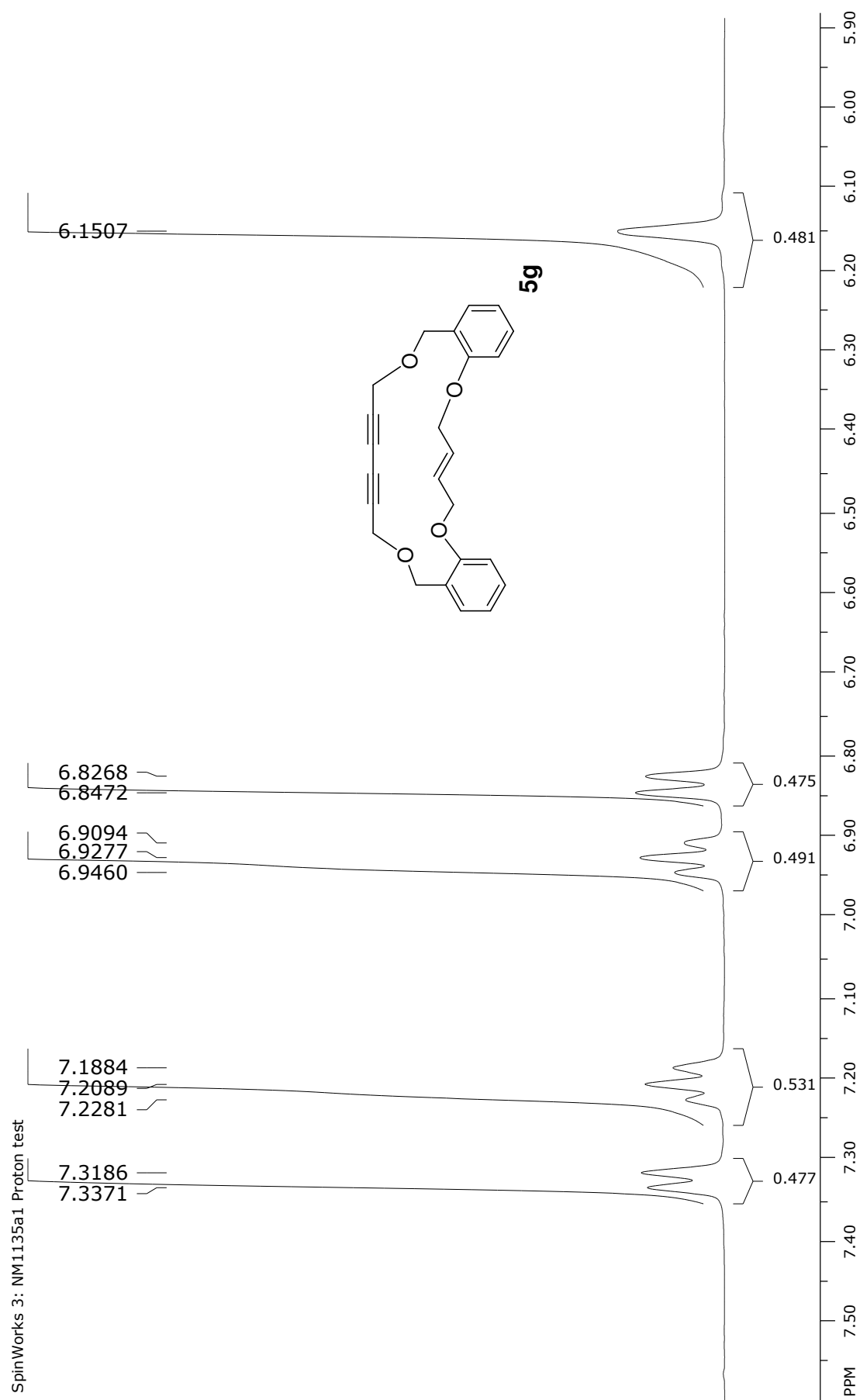
156.7501



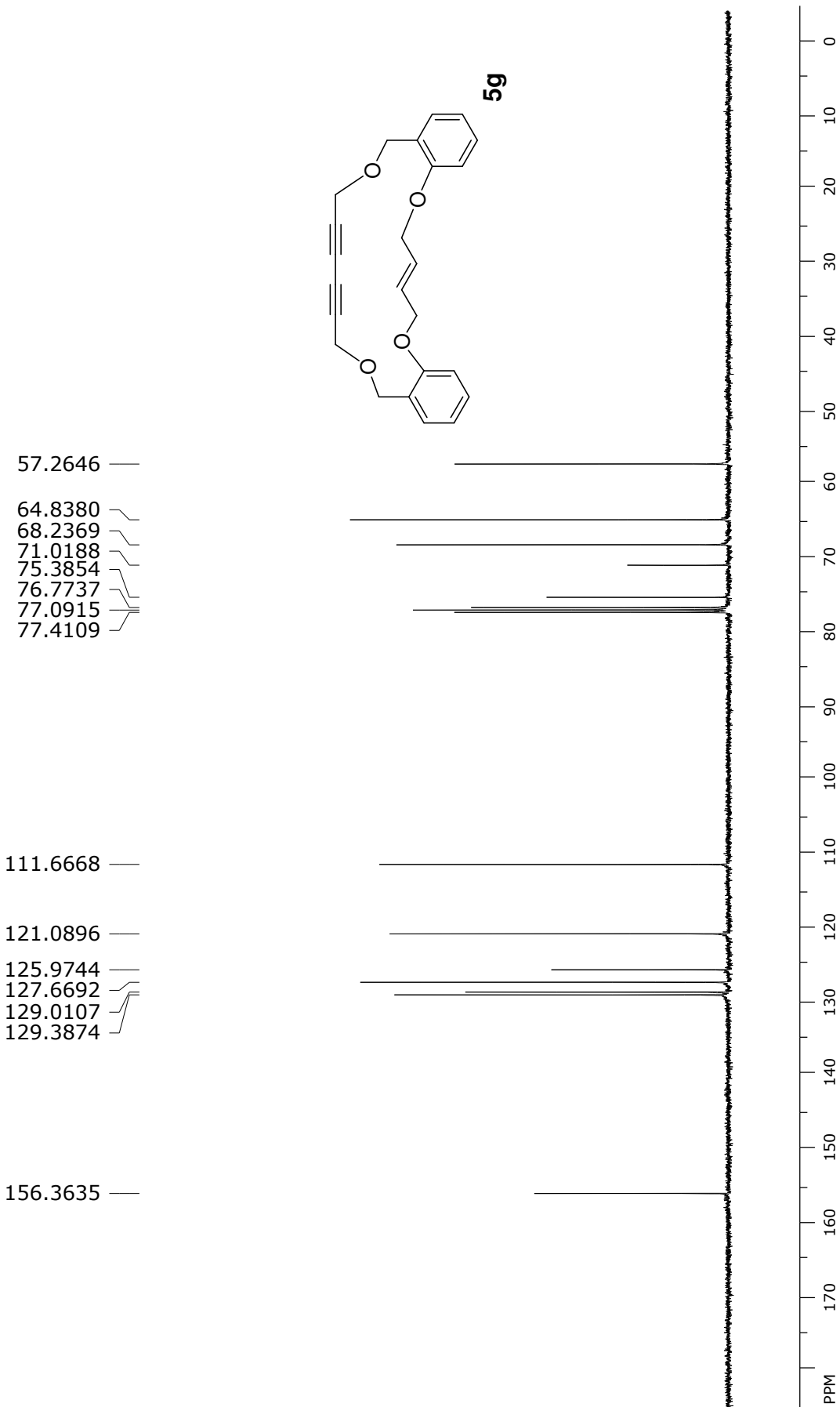








SpinWorks 3: NM1135a1 Carbon test



6.8829
6.9034
7.0304
7.0490
7.0657
7.2745
7.2788
7.2852
7.2948
7.2980
7.3137
7.3180
7.4078
7.4265

4.7842

4.7842

4.7951

6.8829
6.9034
7.0304
7.0490
7.0657
7.2745
7.2788
7.2852
7.2948
7.2980
7.3137
7.3180
7.4078
7.4265

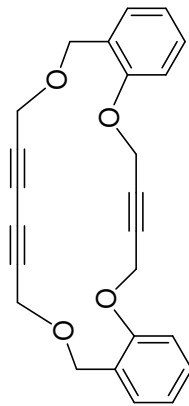
0.426

0.214

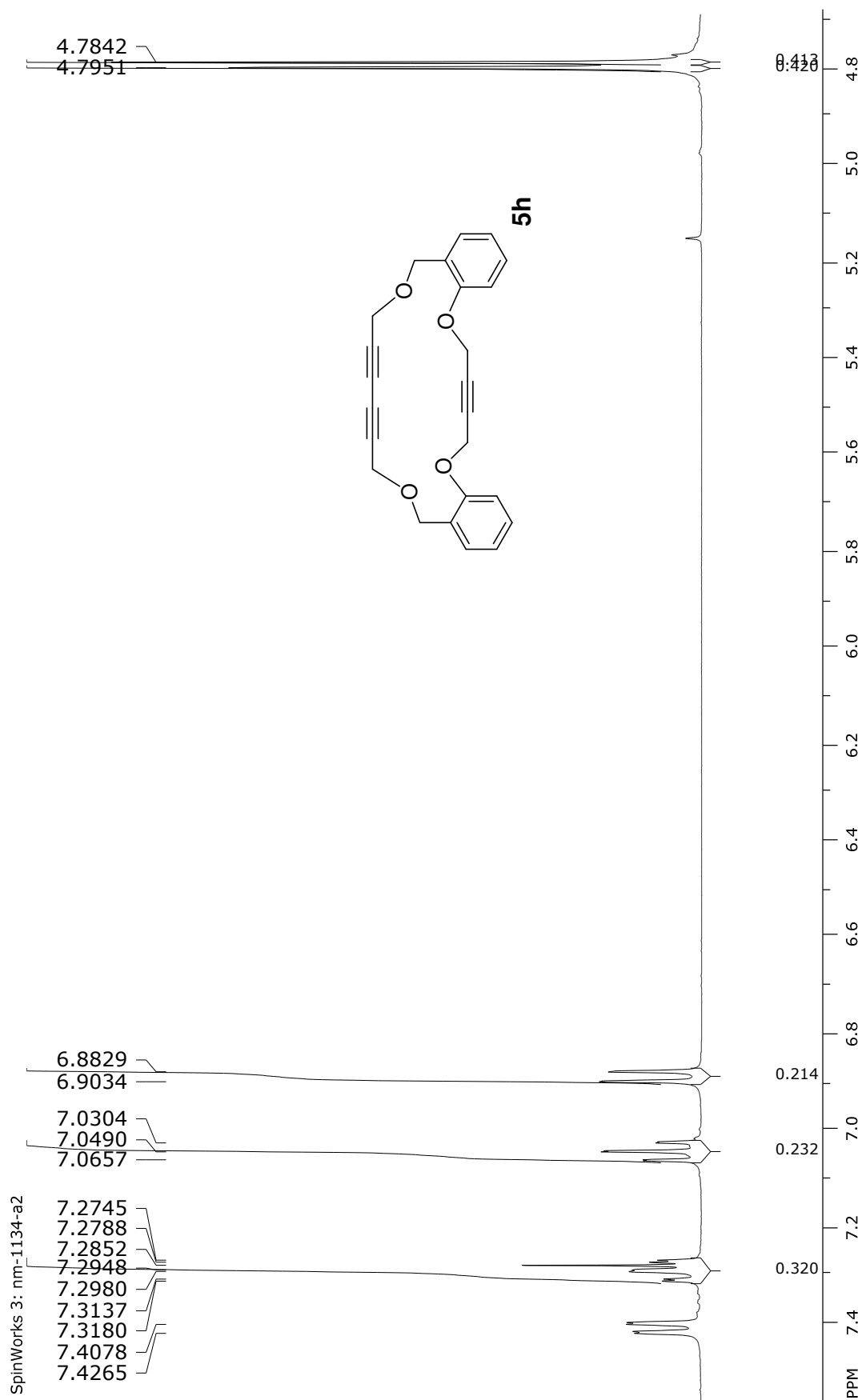
0.232

0.320

PPM	8.0	7.6	7.2	6.8	6.4	6.0	5.6	5.2	4.8	4.4	4.0	3.6	3.2	2.8	2.4	2.0	1.6	1.2	0.8	0.4	0.0
-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----

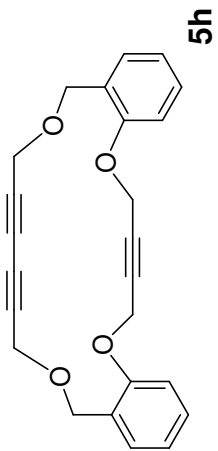


5h



SpinWorks 3: nm-1134-a2

4.7842

~~4.7951~~

5h

4.3353

0.411

0.413

0.420

ppm

4.76

4.68

4.60

4.52

4.44

4.36

4.28

SpinWorks 3: nm-1034-a2

155.6269 —

129.4086

128.9714

126.8436 —

121.8825 —

112.3101 —

81.7308 —

77.3543 —//

77.2394 —
77.2716 —

77.0301
77.2394

76.7192
77.0361

75.3204
76.7192

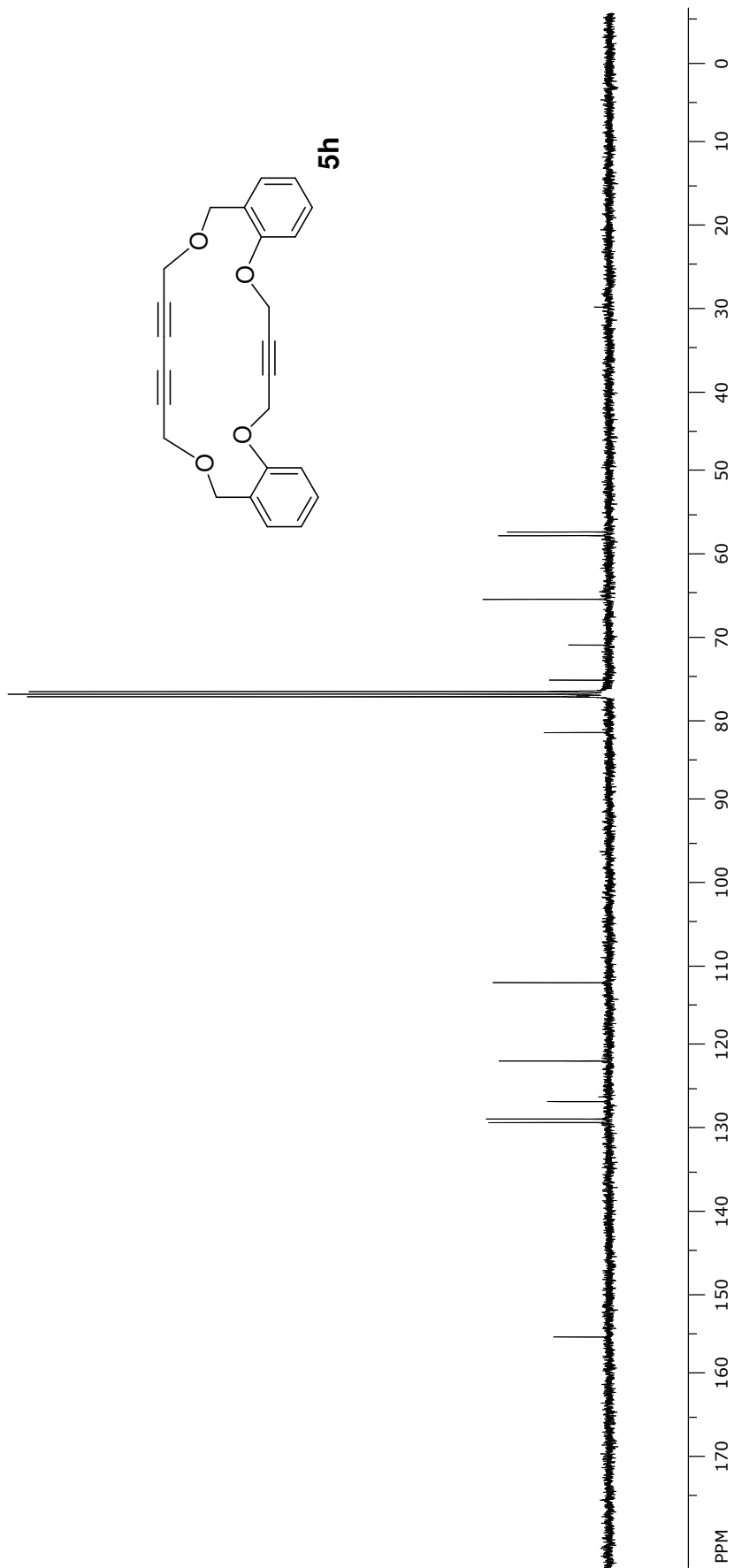
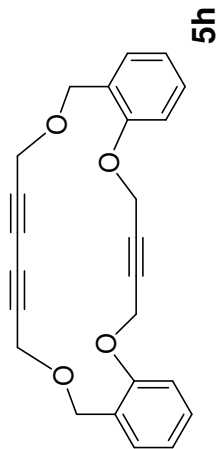
71.0308
75.3204

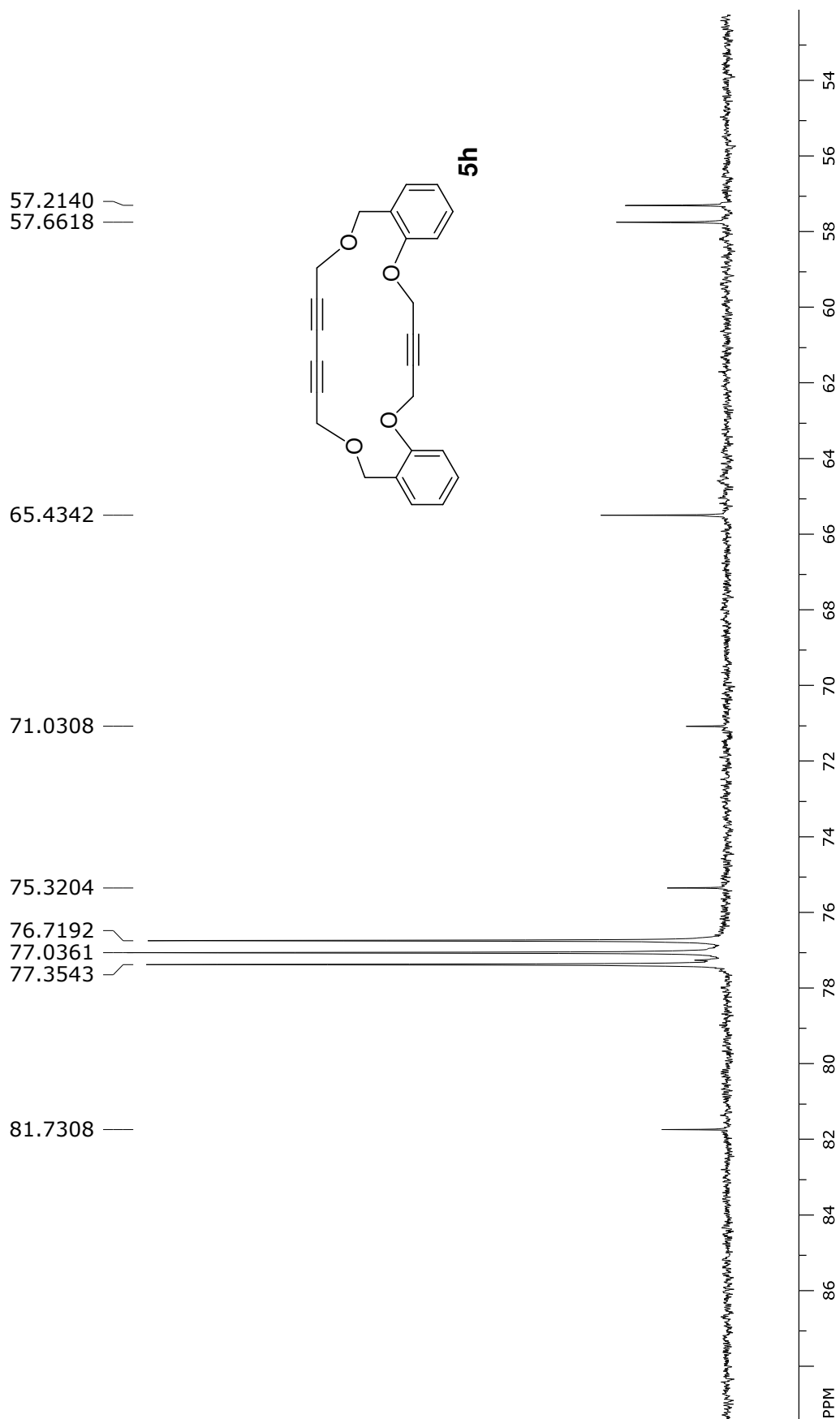
65.4342 

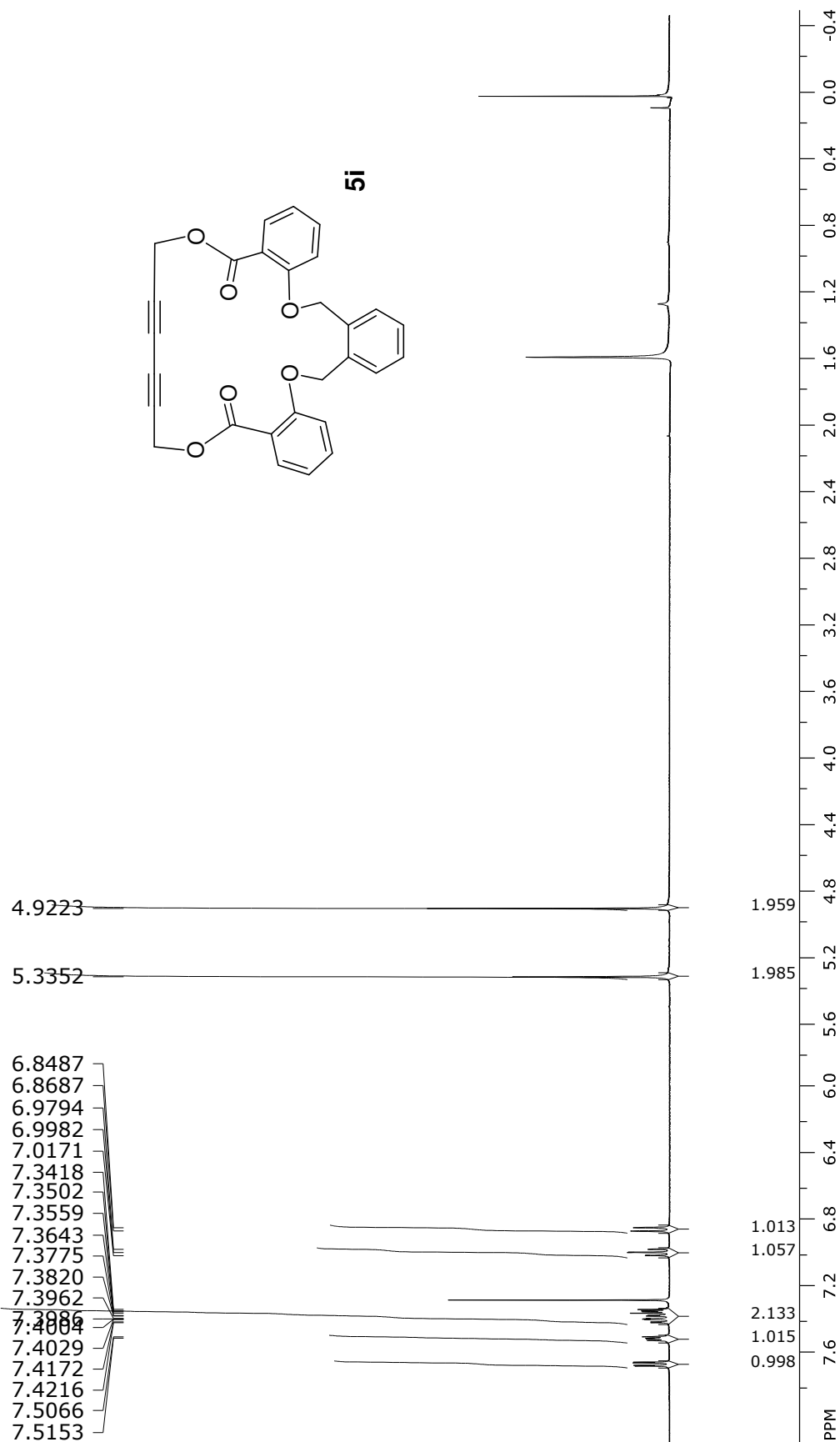
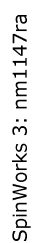
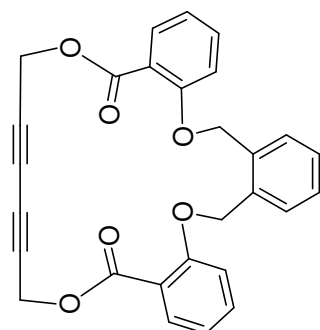
71.0308

65 4343

57.6618 —







SpinWorks 3: nm1147ra

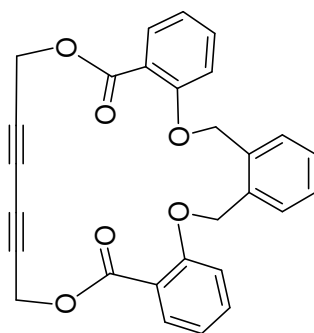
7.6598
7.6642
7.6789
7.6833

7.5066
7.5153
7.5206
7.5291

7.3418
7.3502
7.3559
7.3643
7.3775
7.3820
7.3962
7.3986
7.4004
7.4029
7.4172
7.4216

6.9794
6.9982
7.0171

6.8487
6.8687



0.998

1.015

2.133

1.057

1.013

PPM

7.70

7.60

7.50

7.40

7.30

7.20

7.10

7.00

6.90

6.80

6.70

SpinWorks 3: nm1147ra

166.3749 —

157.2769 —

134.4225 —
133.1724 —
130.6300 —
128.3945 —
128.3261 —

120.5708 —

113.0398 —

77.3561 —

77.0382 —

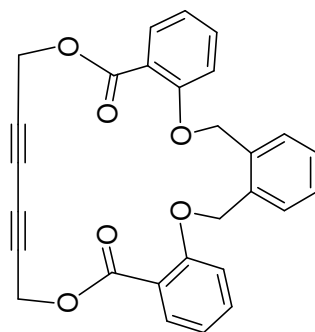
76.7212 —

74.6803 —

70.6741 —

68.5079 —

52.7771 —



5i

PPM

180

170

160

150

140

130

120

110

100

90

80

70

60

50

40

30

20

10

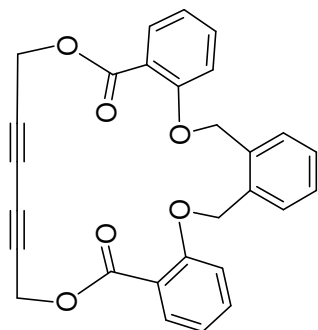
0

128.3261 —
128.3945 —

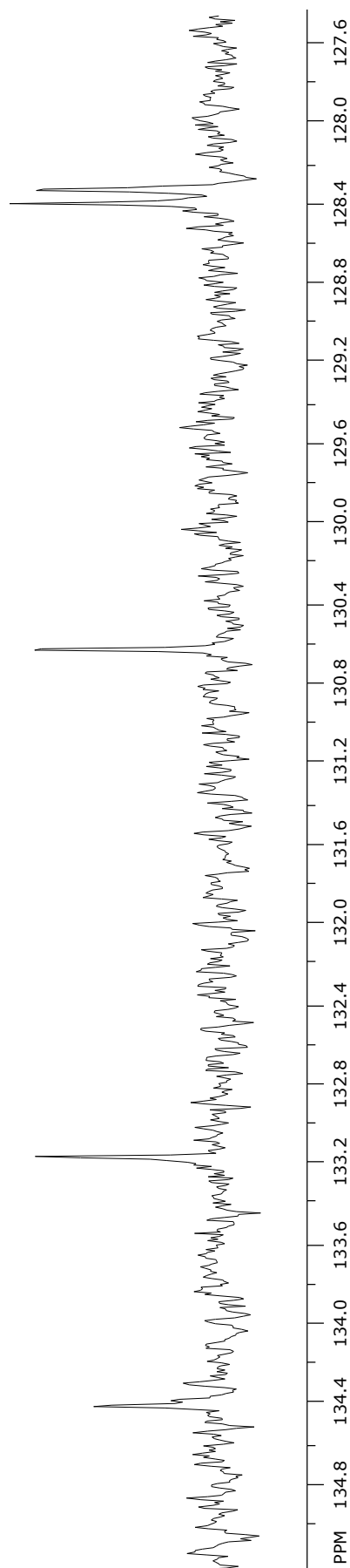
130.6300 —

133.1724 —

134.4225 —



5i



SpinWorks 3: nm1147ra

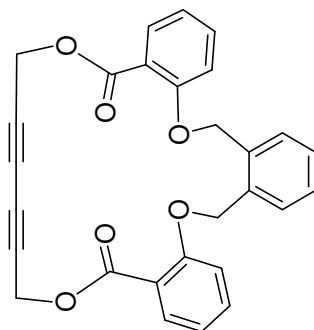
76.7212
77.0382
77.3561

74.6803 —

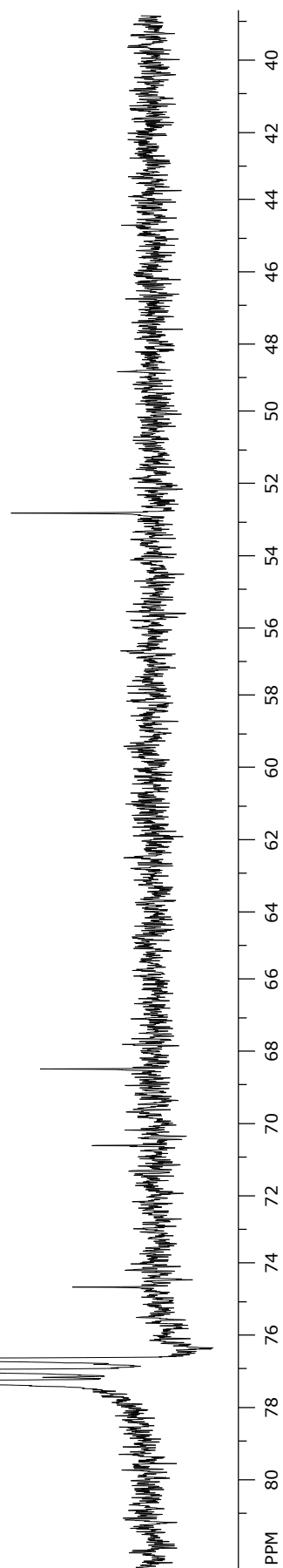
70.6741 —

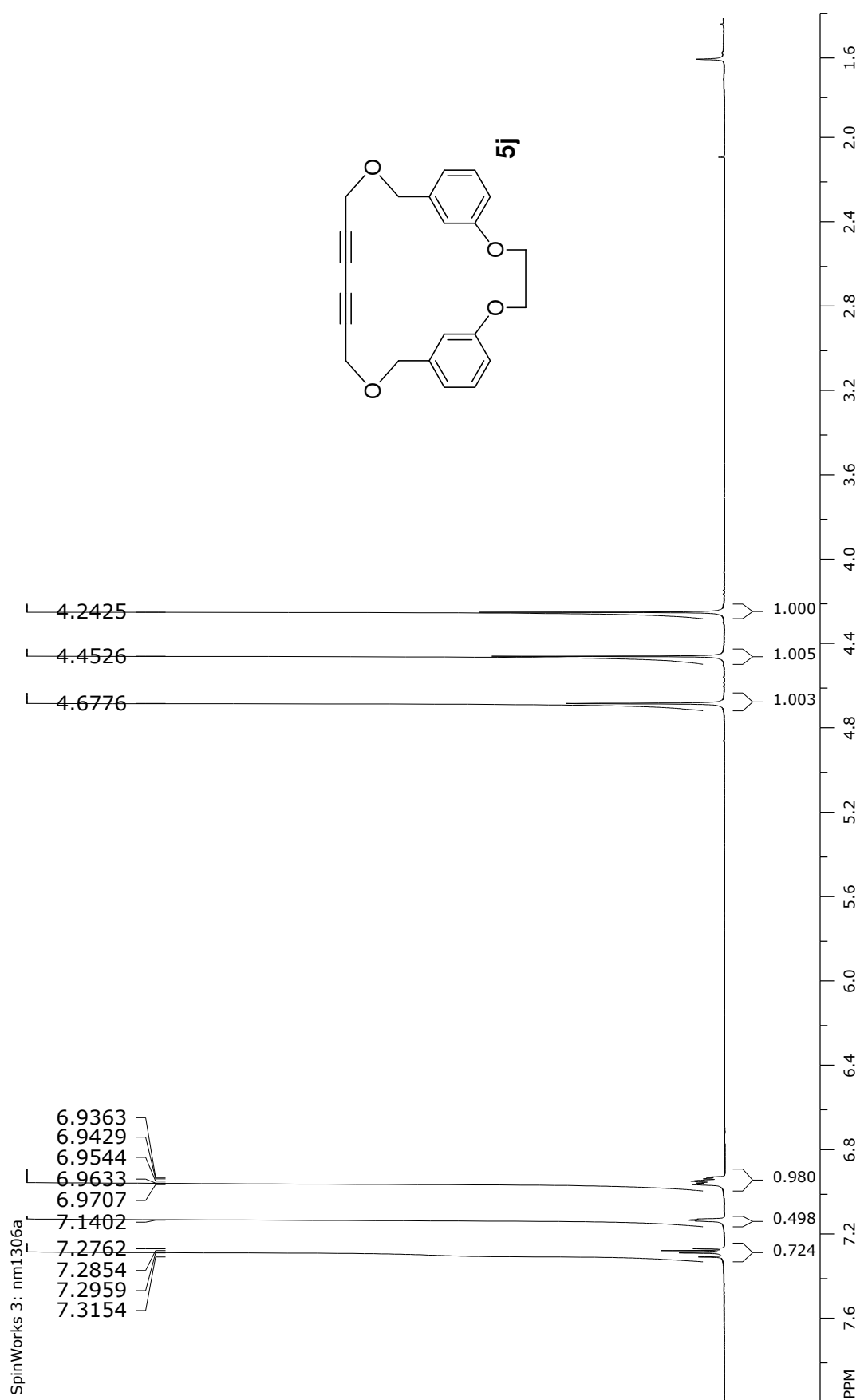
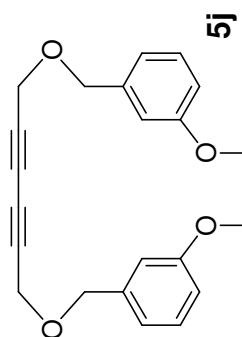
68.5079 —

52.7771 —

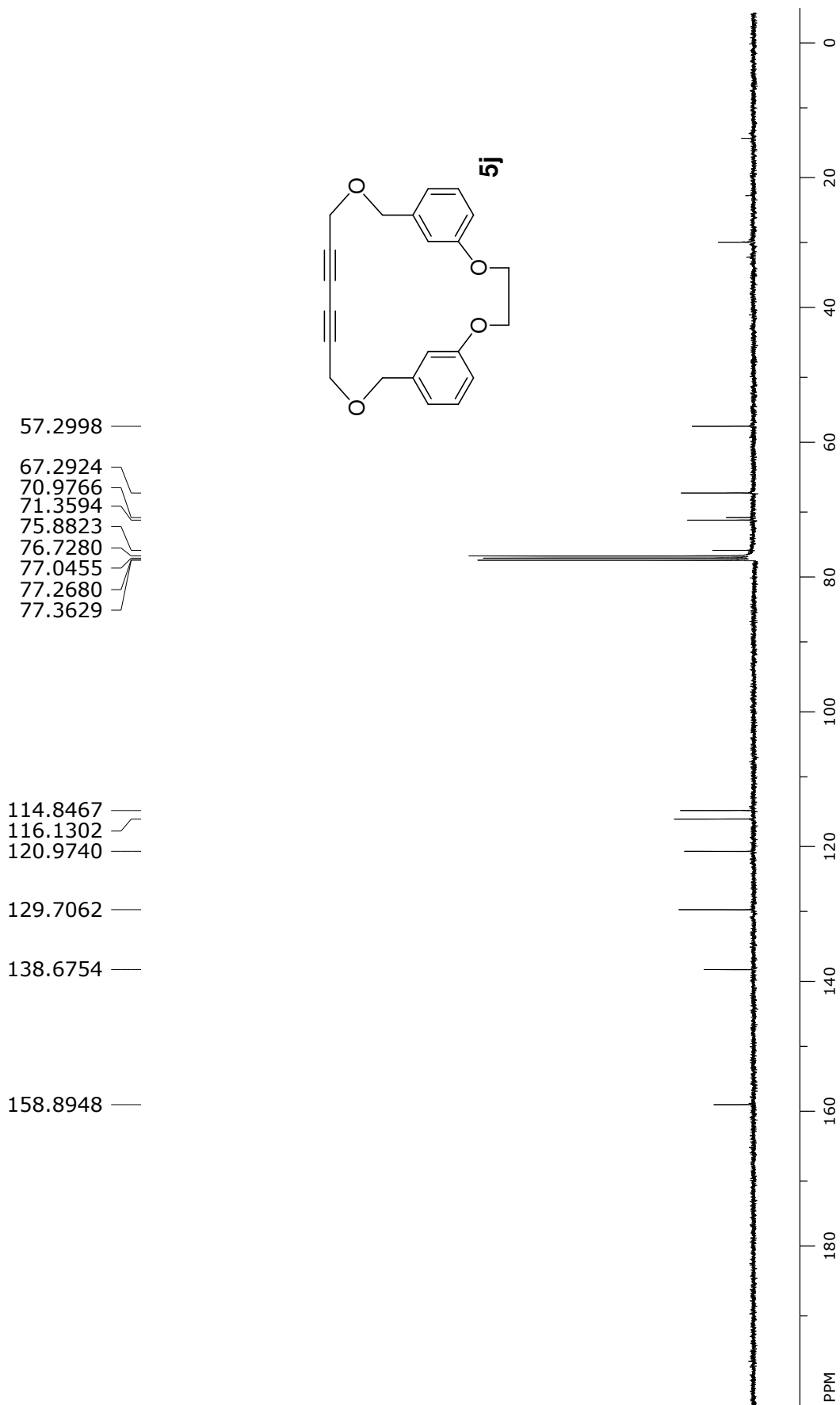


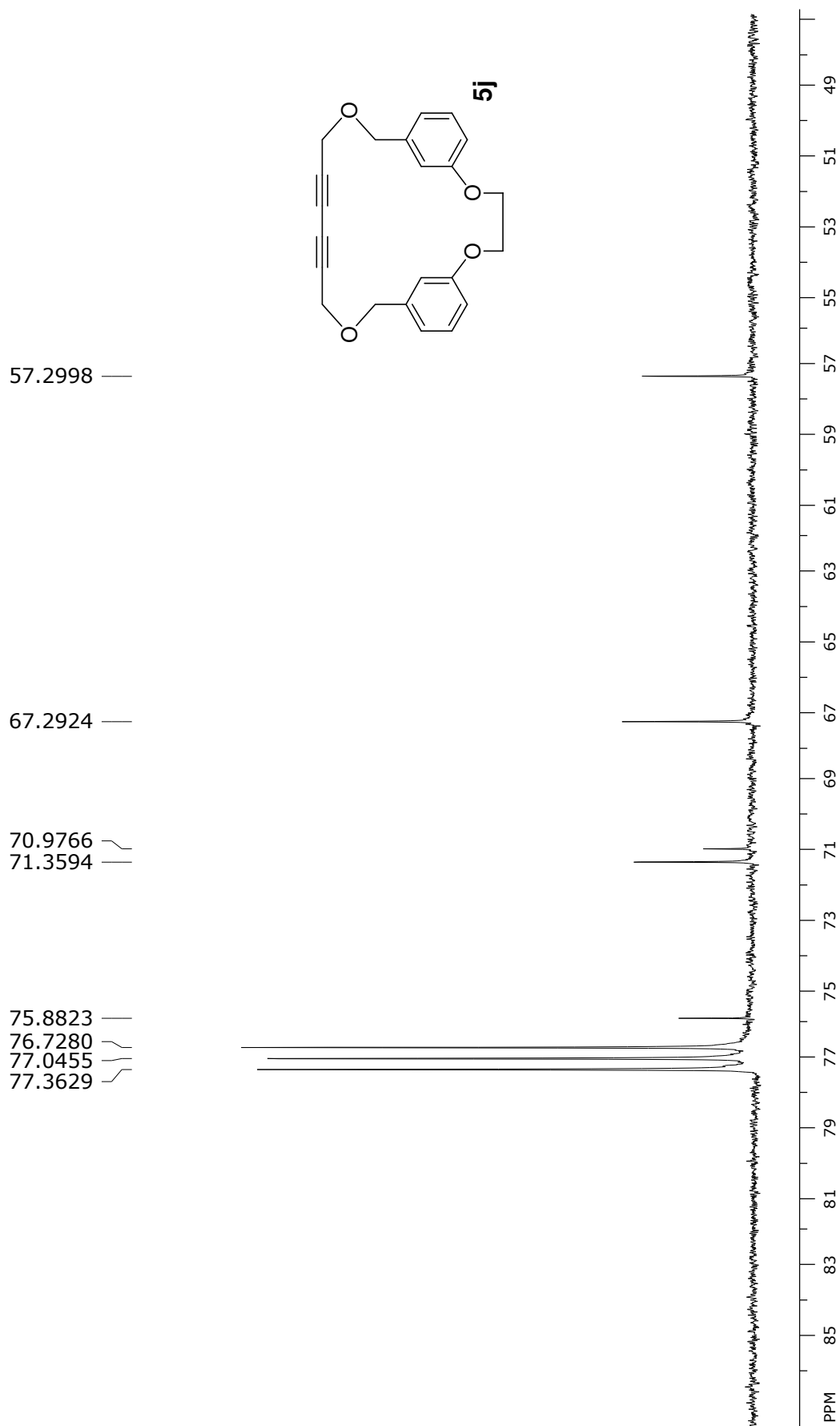
5i

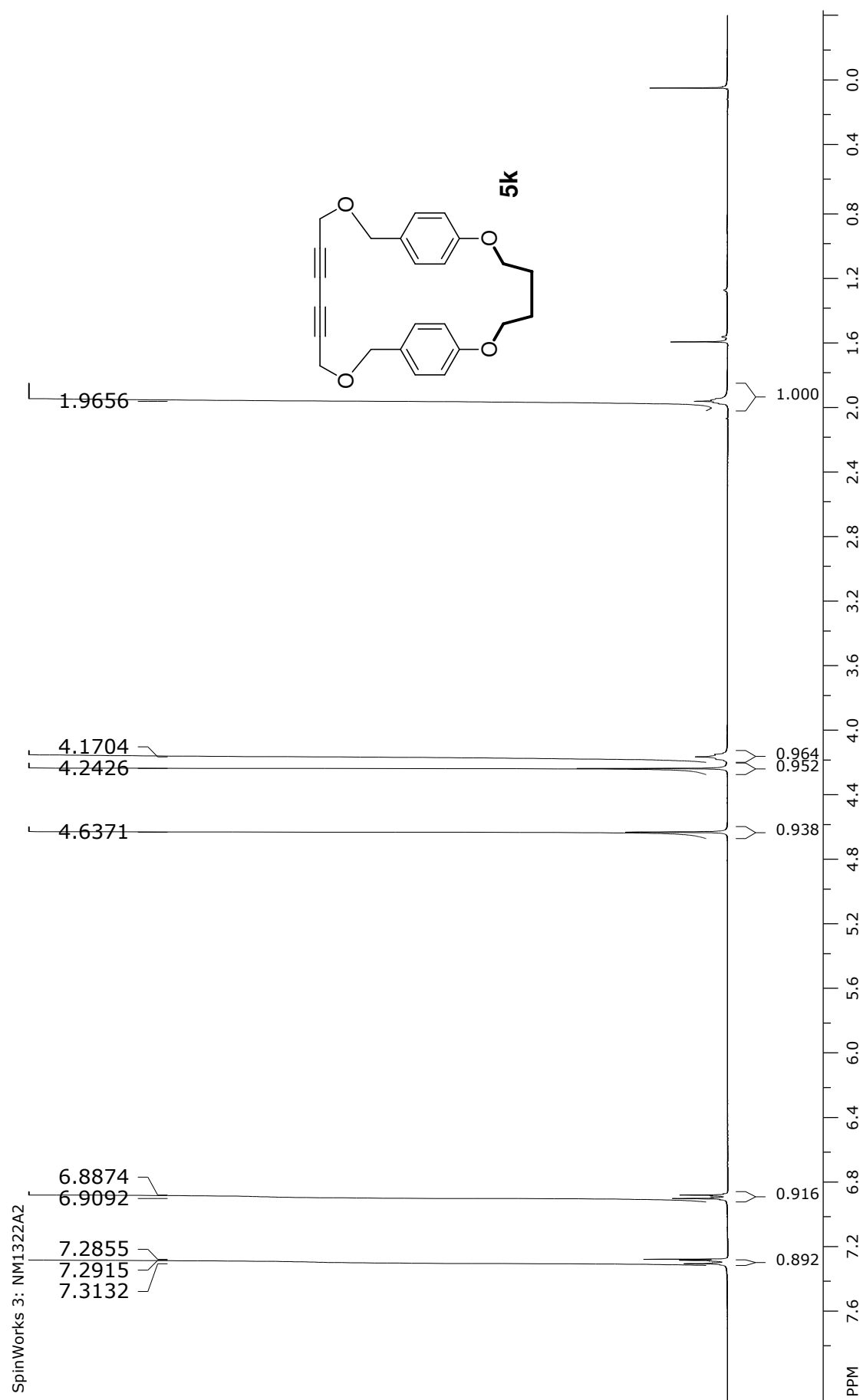




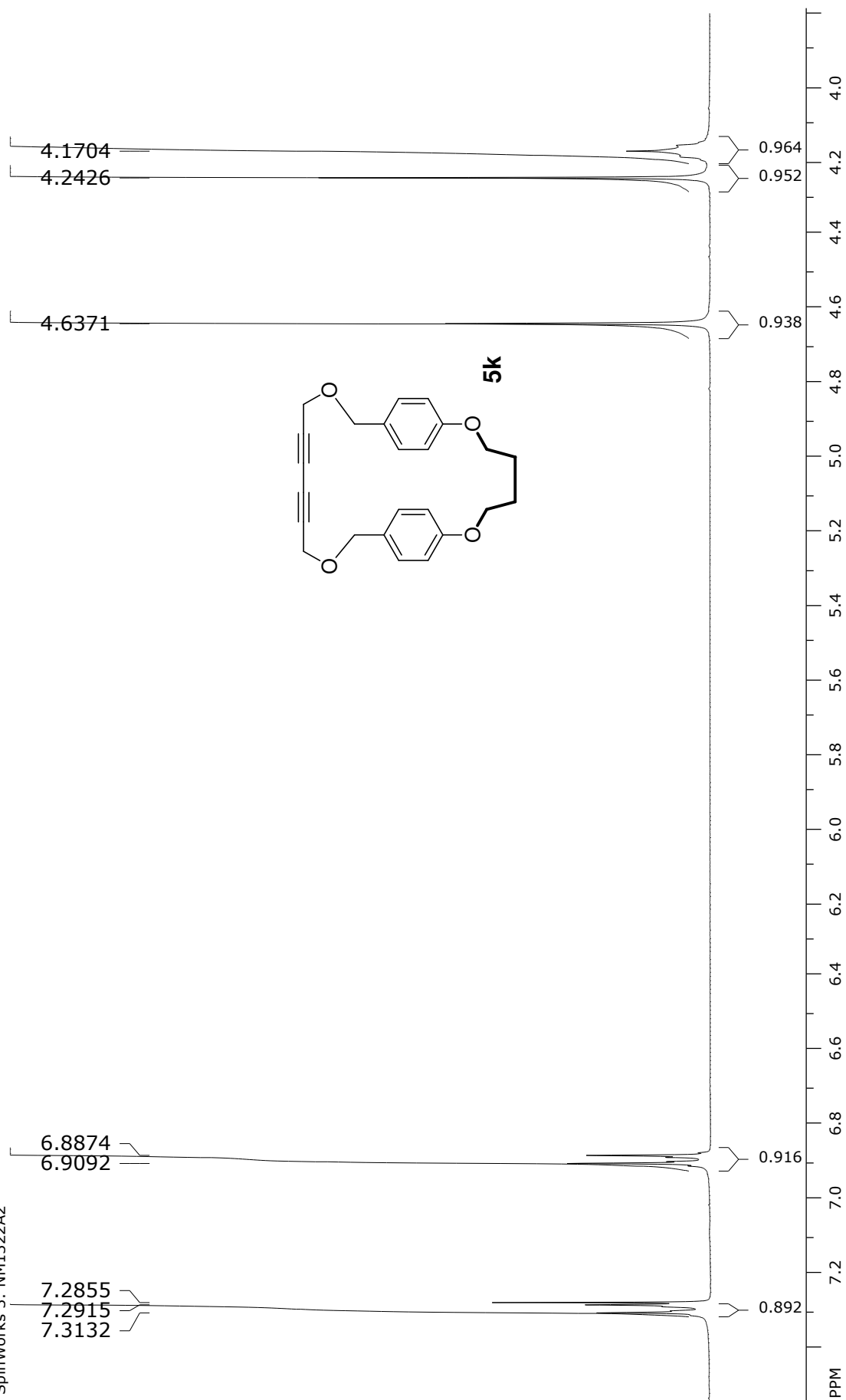
SpinWorks 3: NM1306A

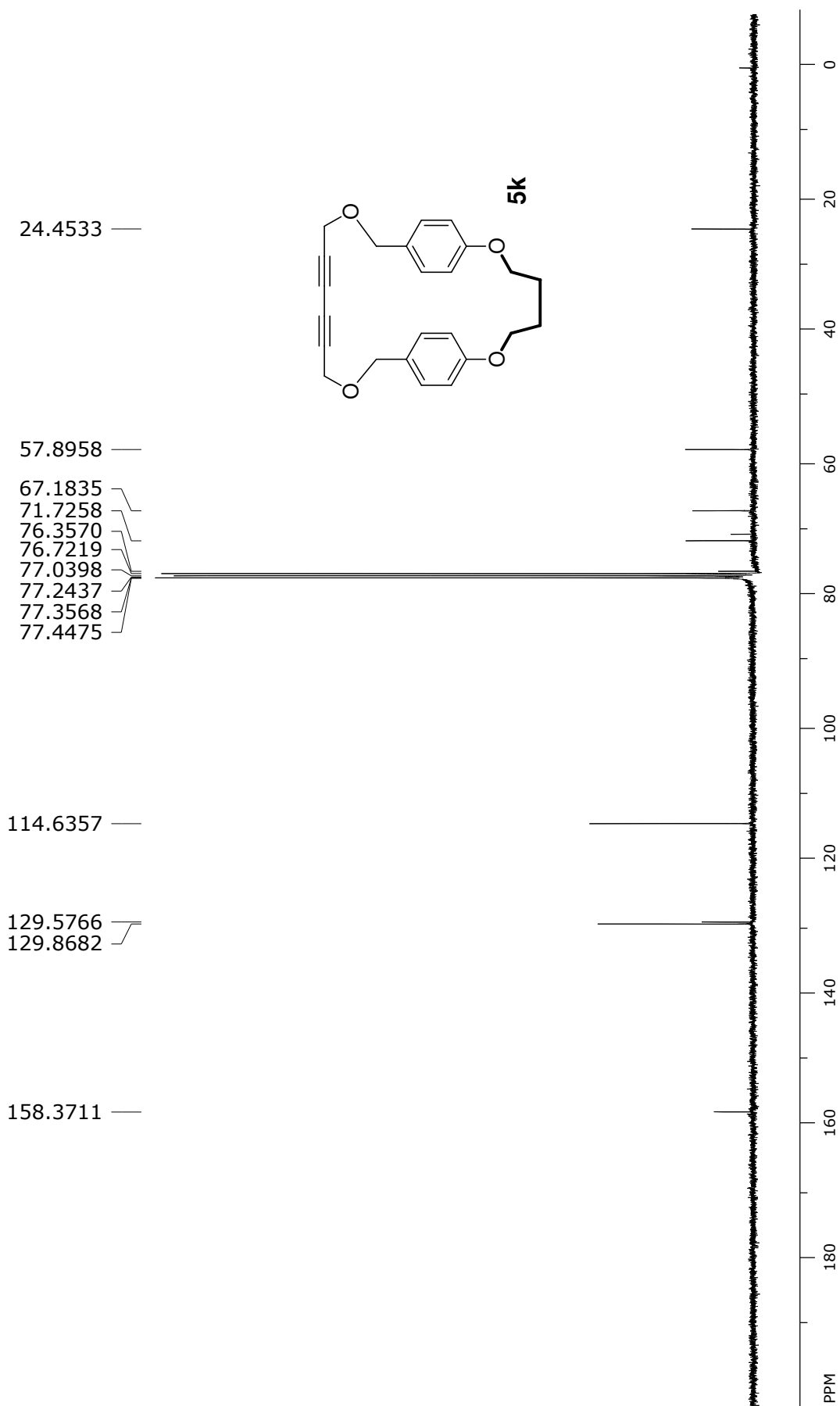






SpinWorks 3: NM1322A2



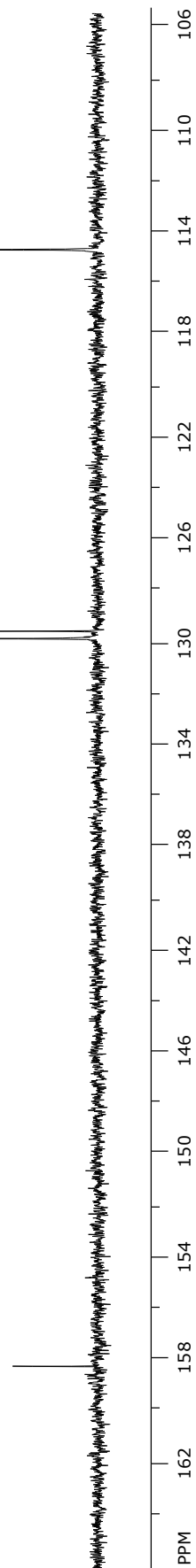
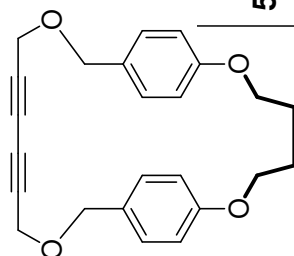


SpinWorks 3: NM1322A2

158.3711 —

129.5766 —
129.8682 —

114.6357 —



SpinWorks 3: NM1322A2

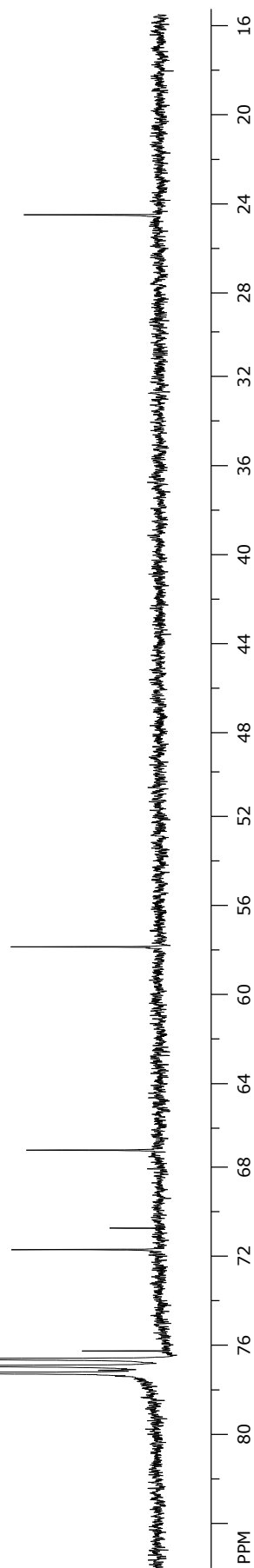
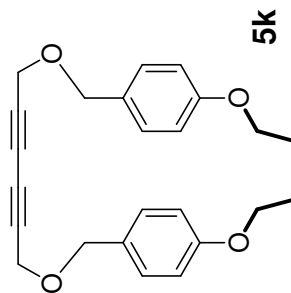
76.3570
76.7219
77.0398
77.3568

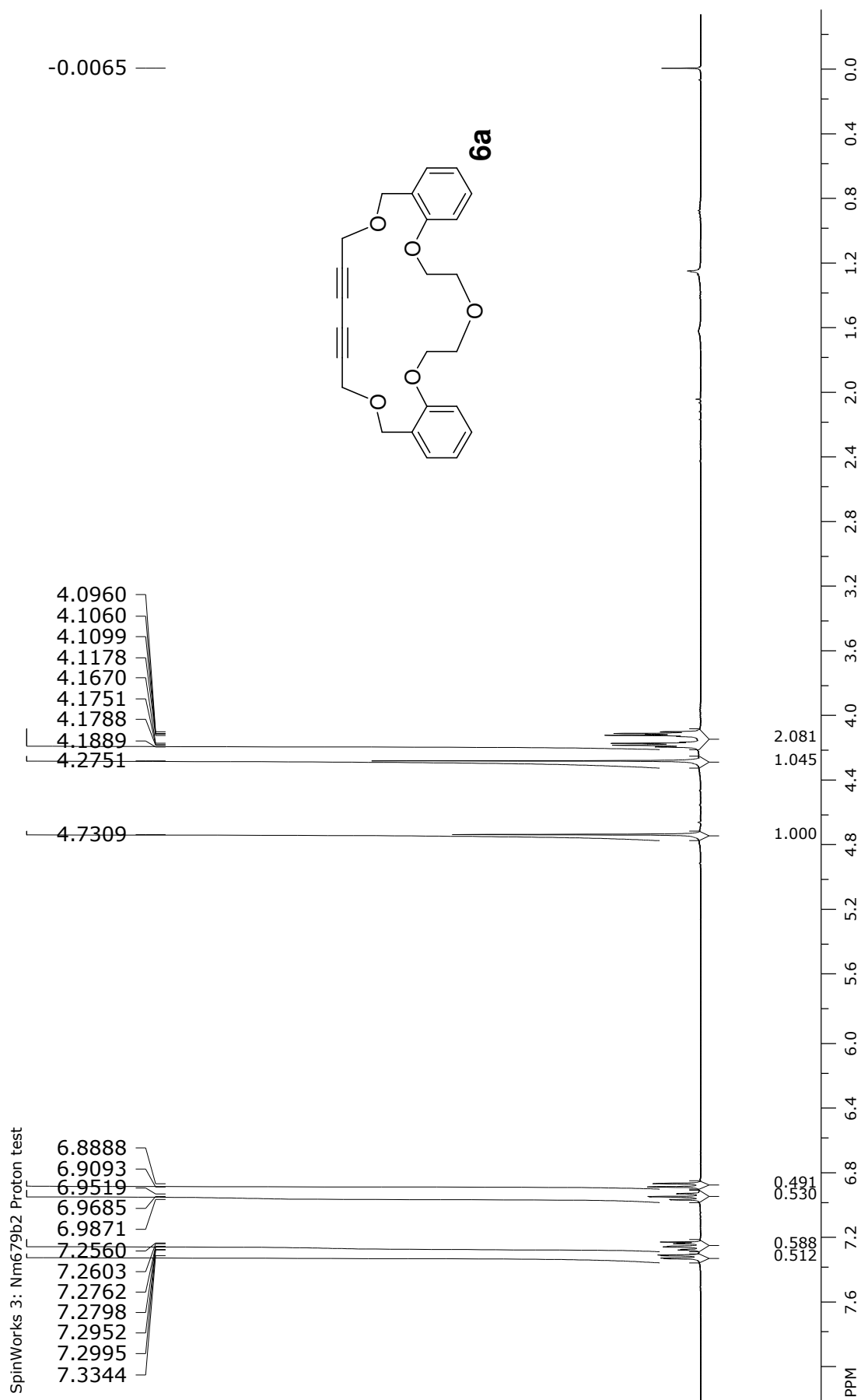
71.7258

67.1835

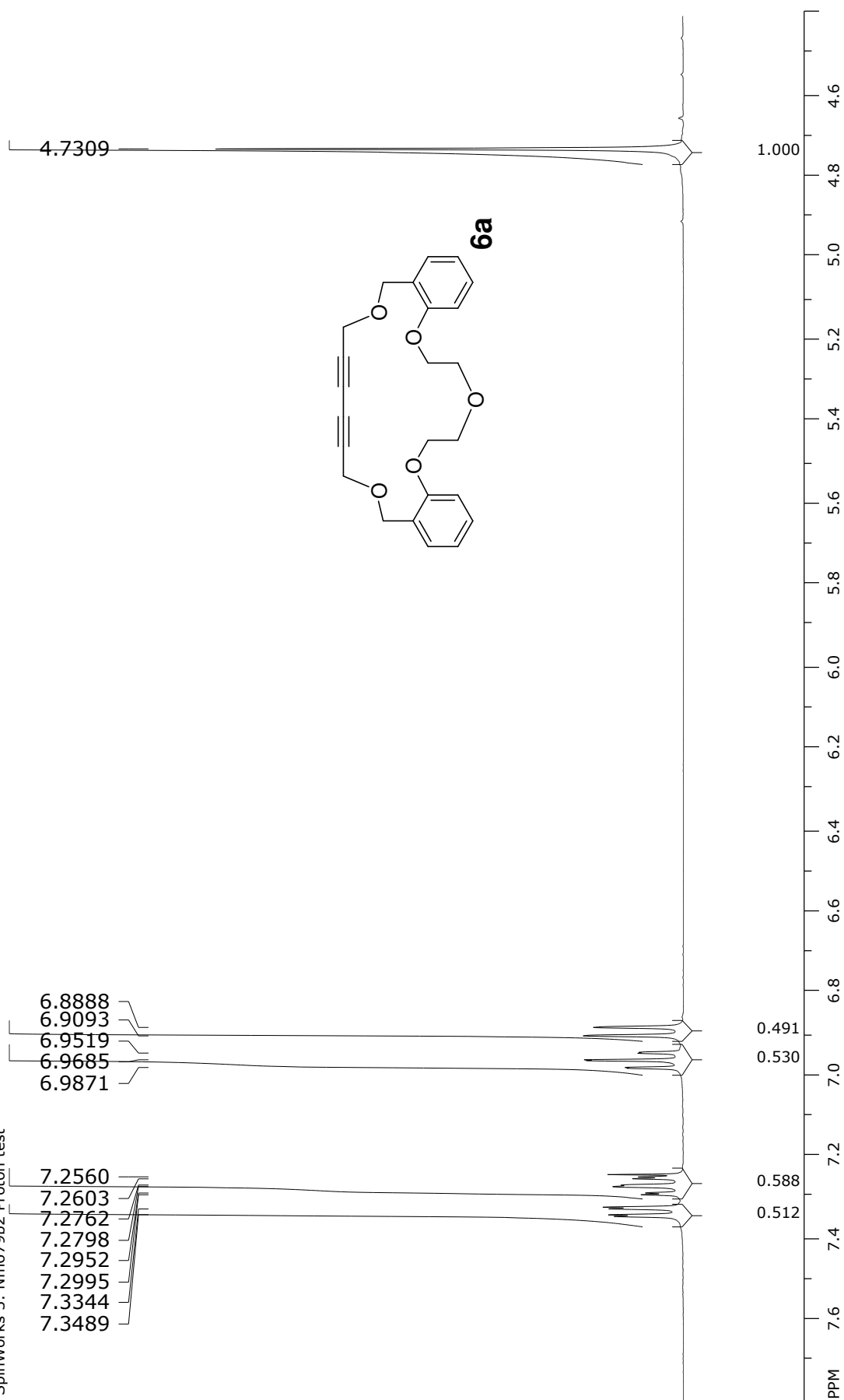
57.8958

24.4533





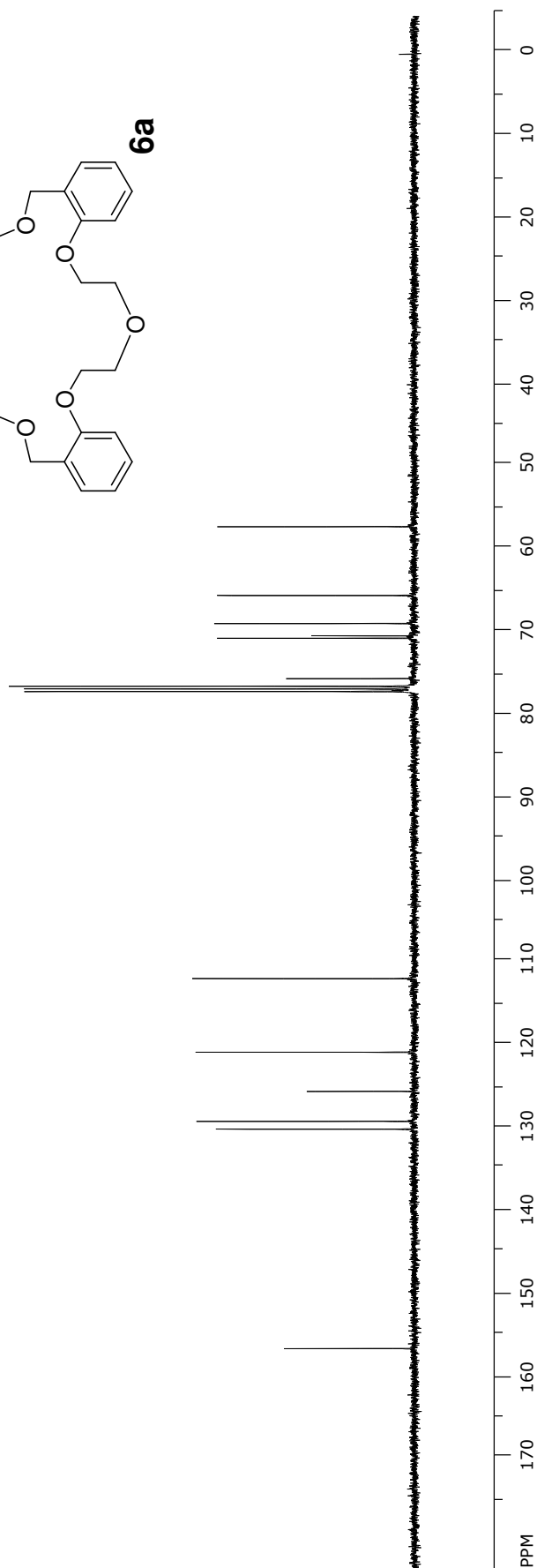
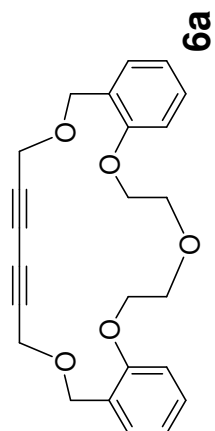
SpinWorks 3: Nm679b2 Proton test



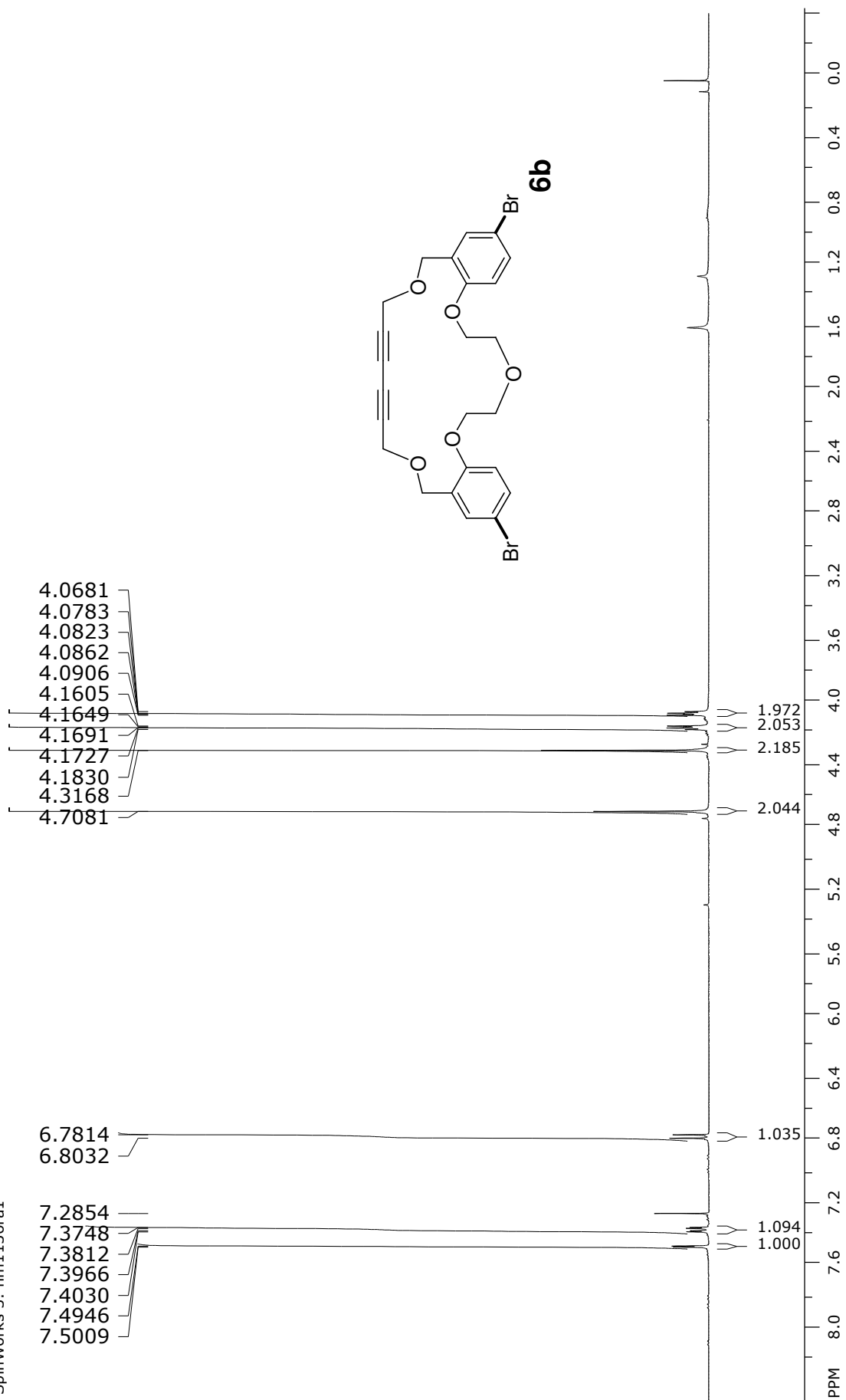
57.3739 —
65.7251 —
69.1282 —
70.6197 —
70.9139 —
75.8092 —
76.7422 —
77.0600 —
77.3783 —

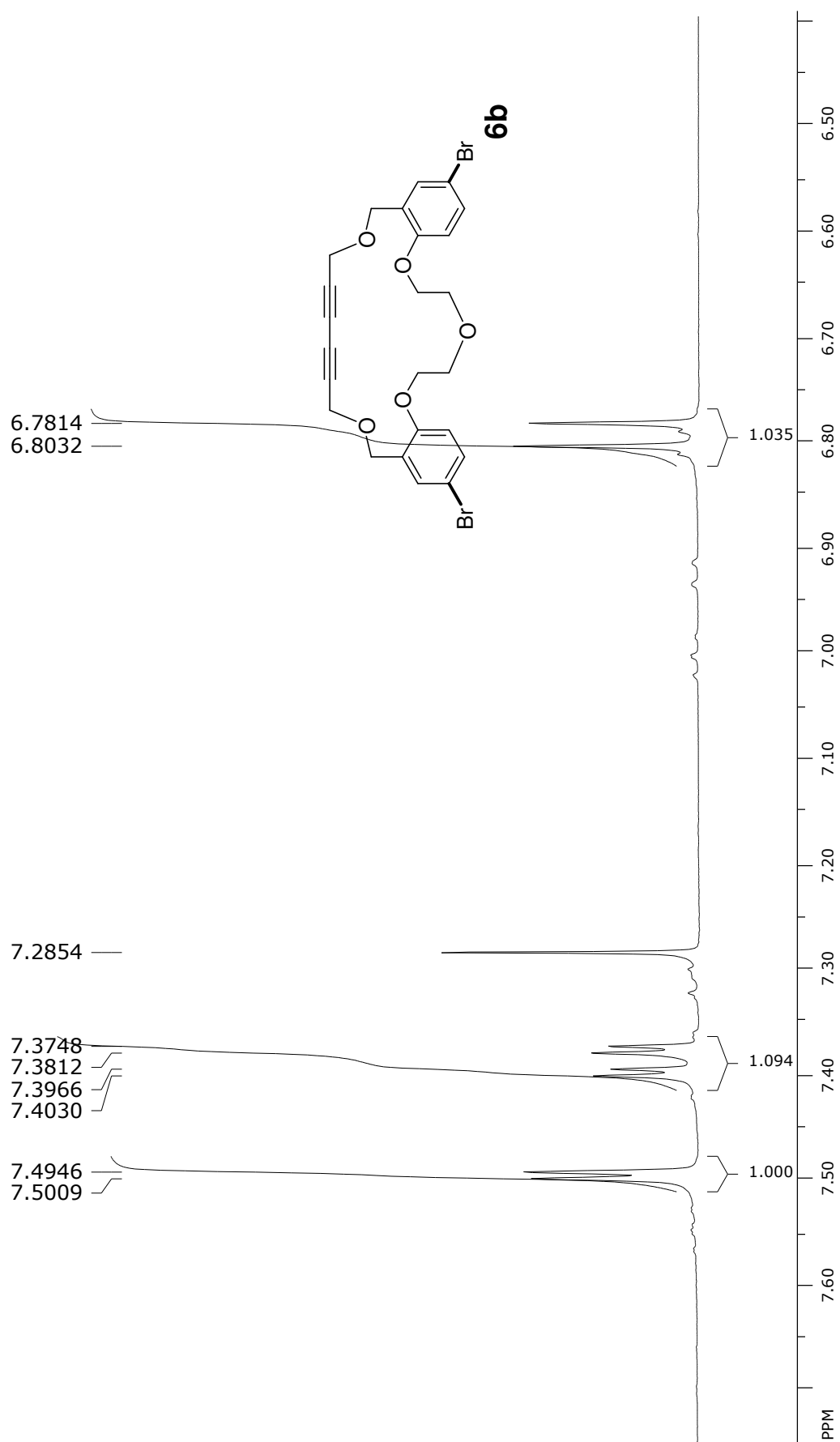
112.2234 —
121.1573 —
125.9163 —
129.5461 —
130.4952 —

157.1283 —

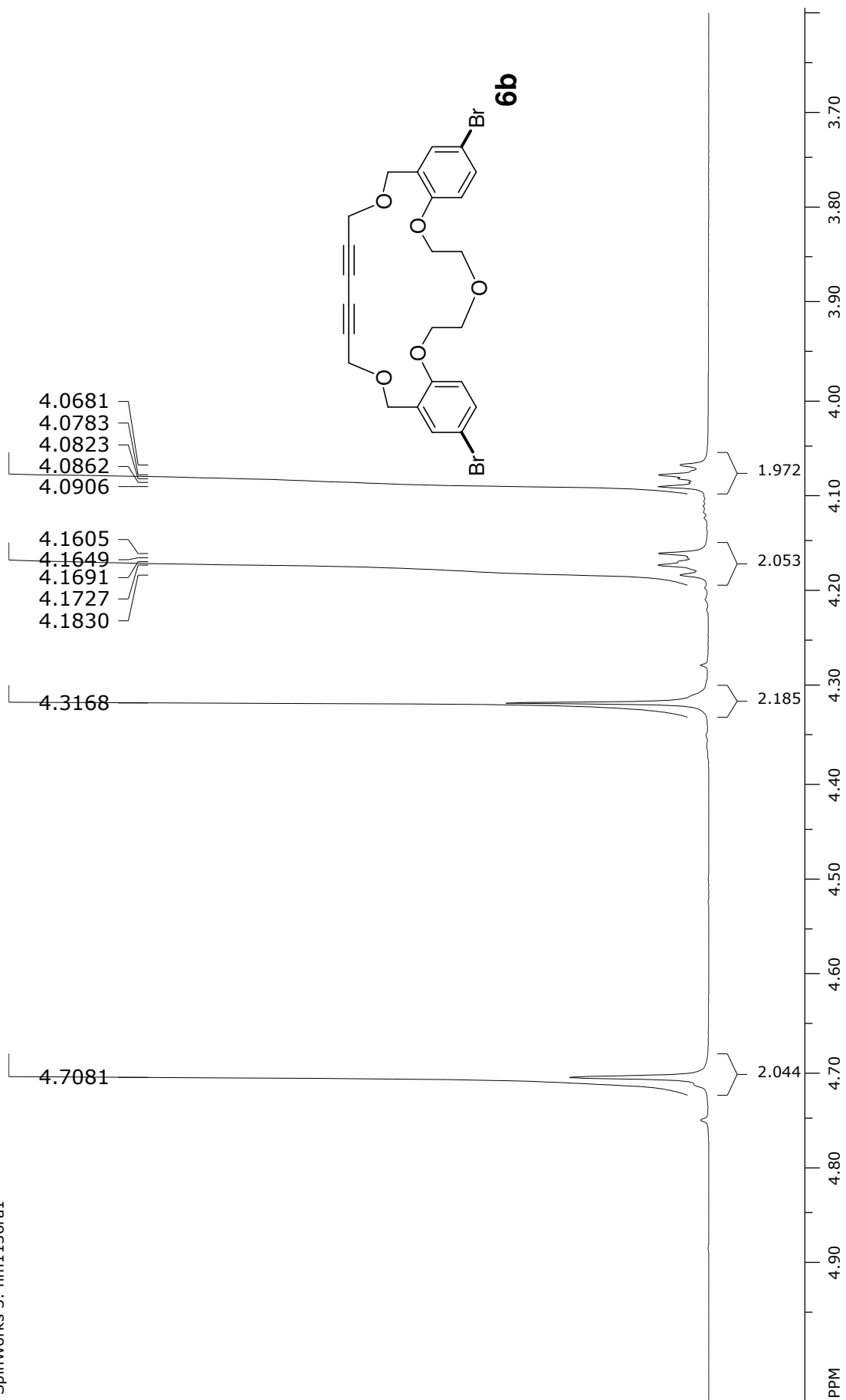


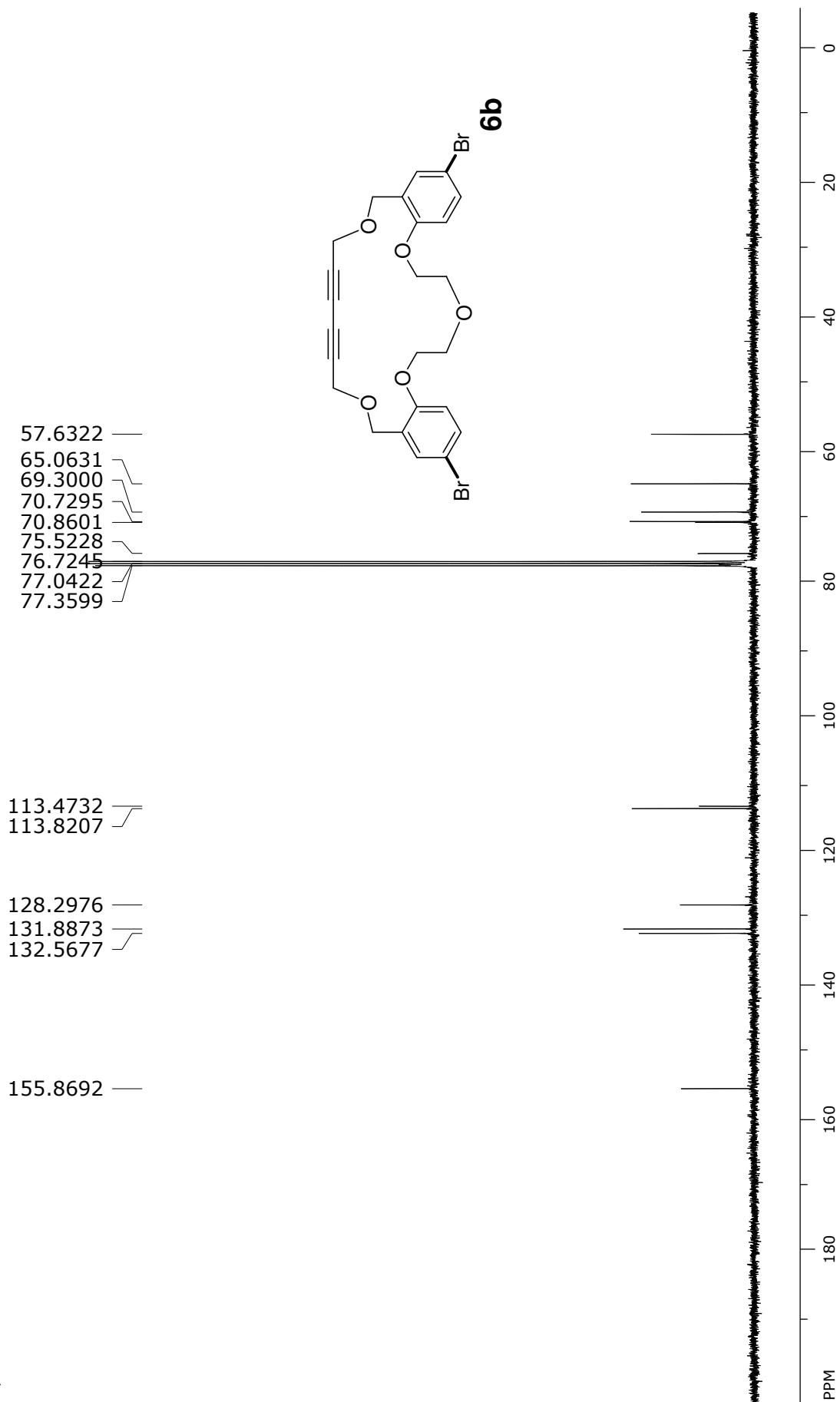
SpinWorks 3: nm1136ra1





SpinWorks 3: nm1136ra1





SpinWorks 3: nm-1136ra1

75.5228 —

76.7245 —

77.0422 —

77.3599 —

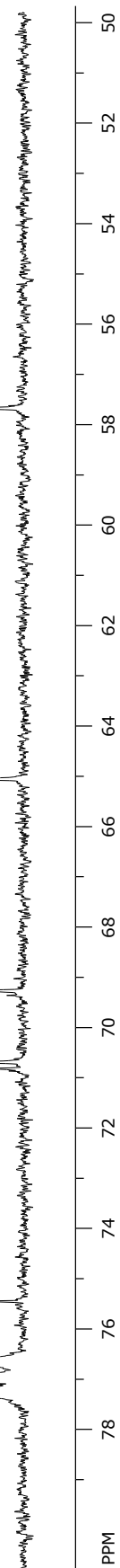
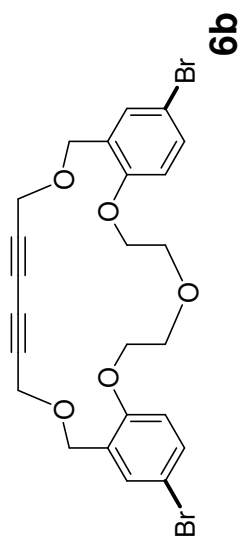
70.7295 —

70.8601 —

69.3000 —

65.0631 —

57.6322 —



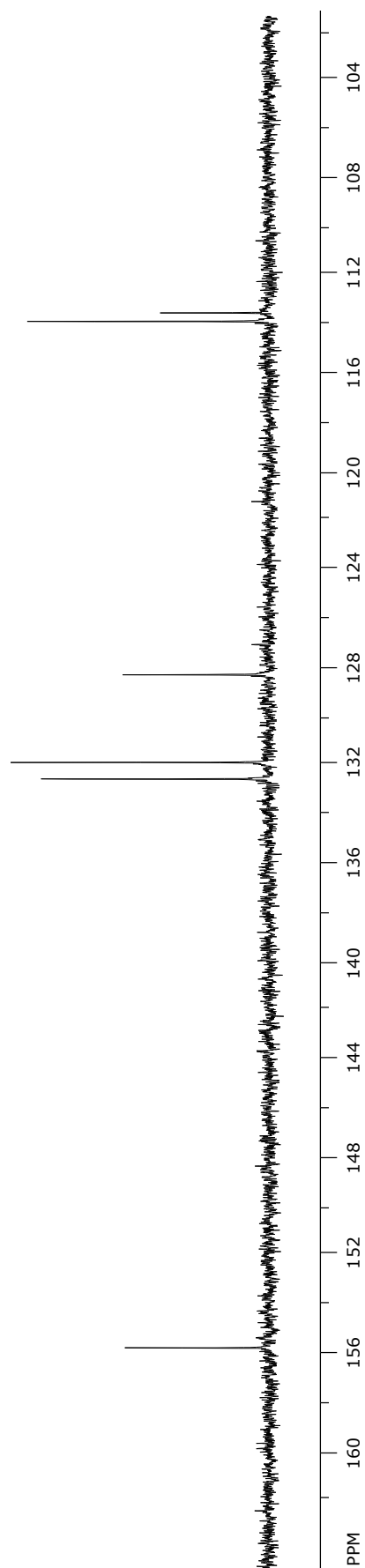
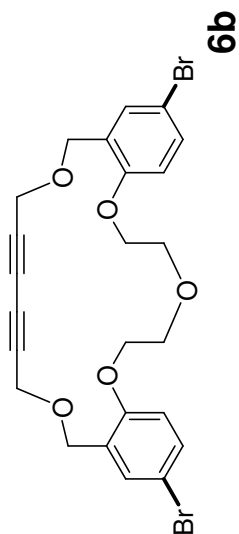
SpinWorks 3: nm-1136ra1

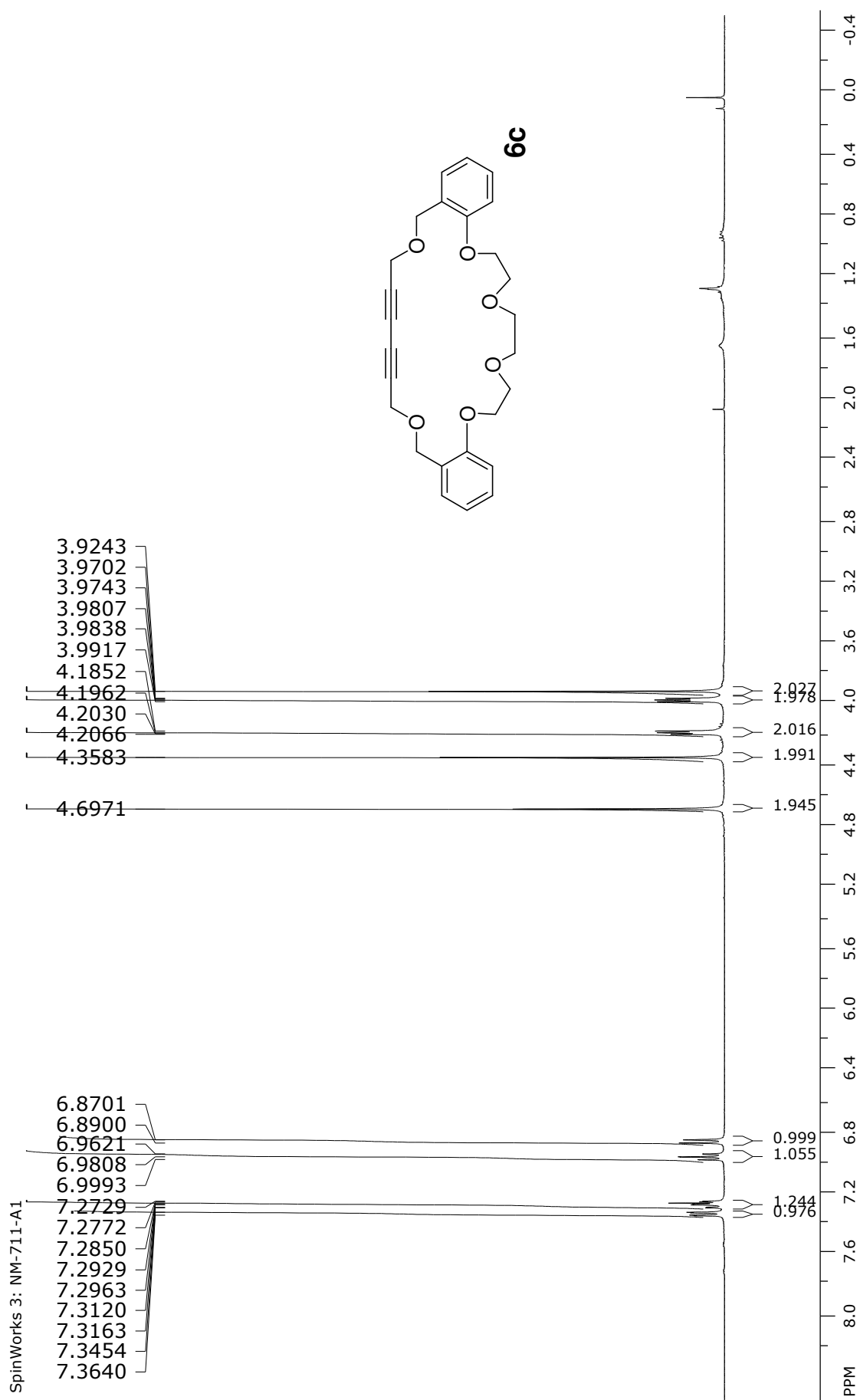
155.8692 —

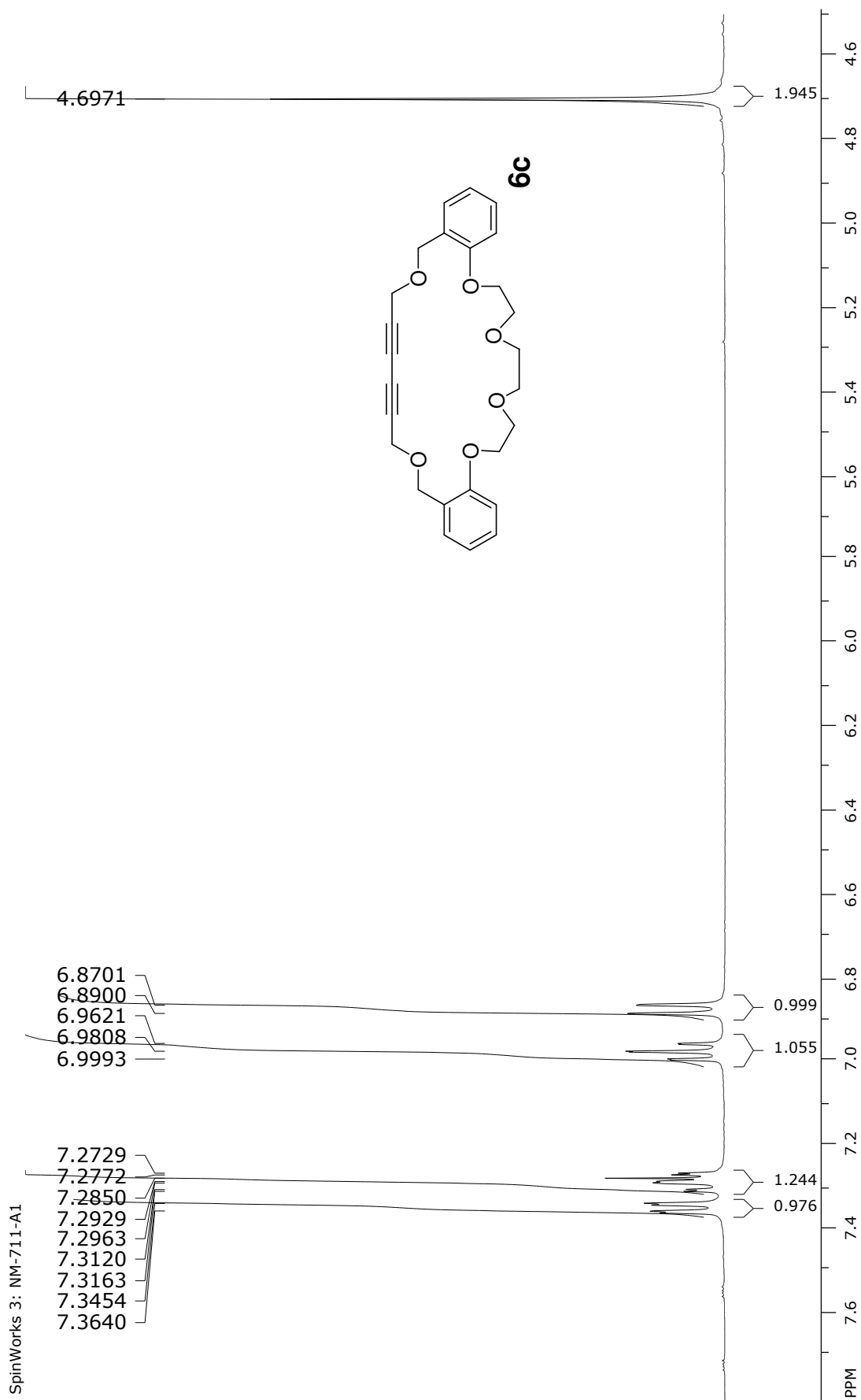
131.8873 —
132.5677 —

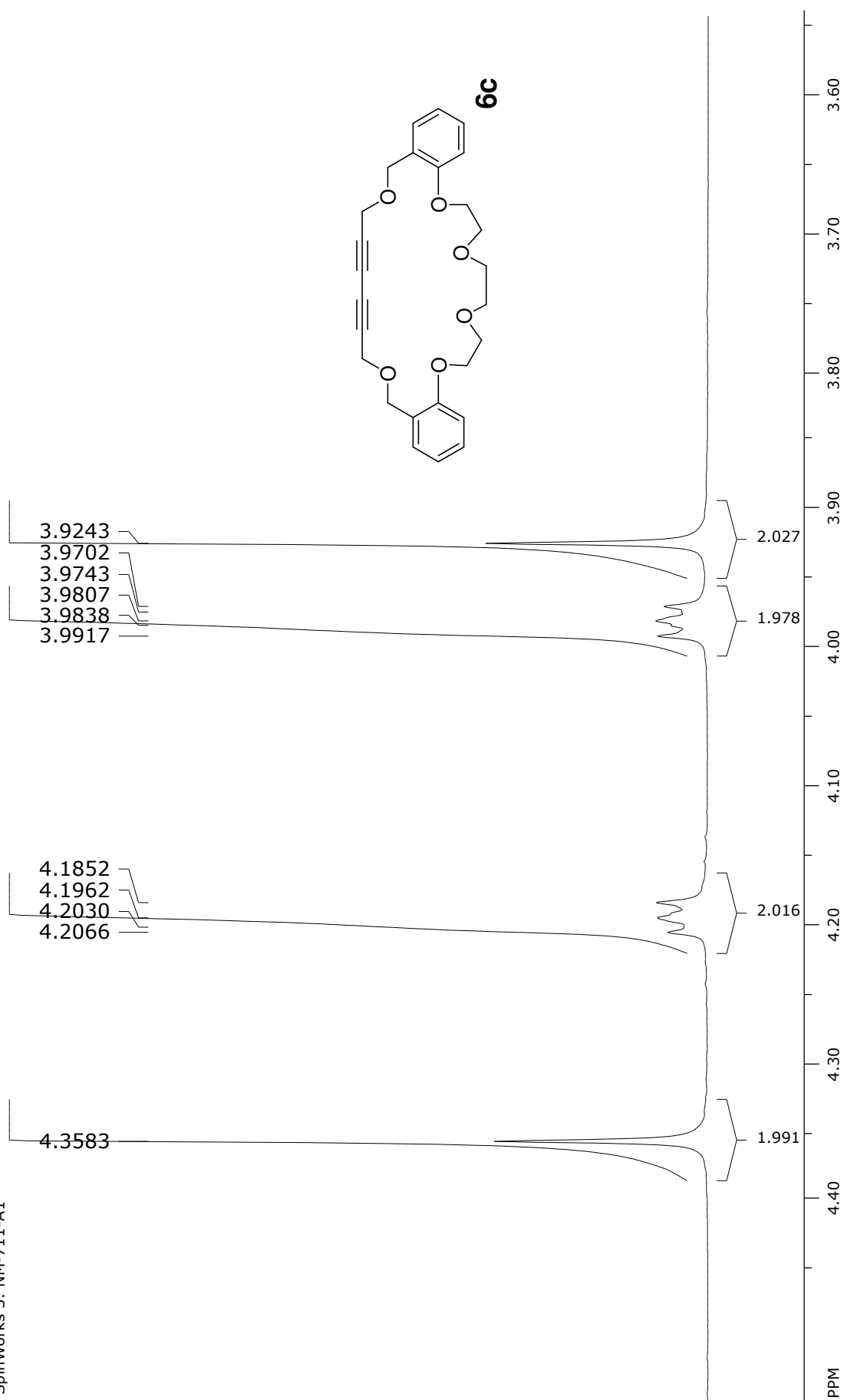
128.2976 —

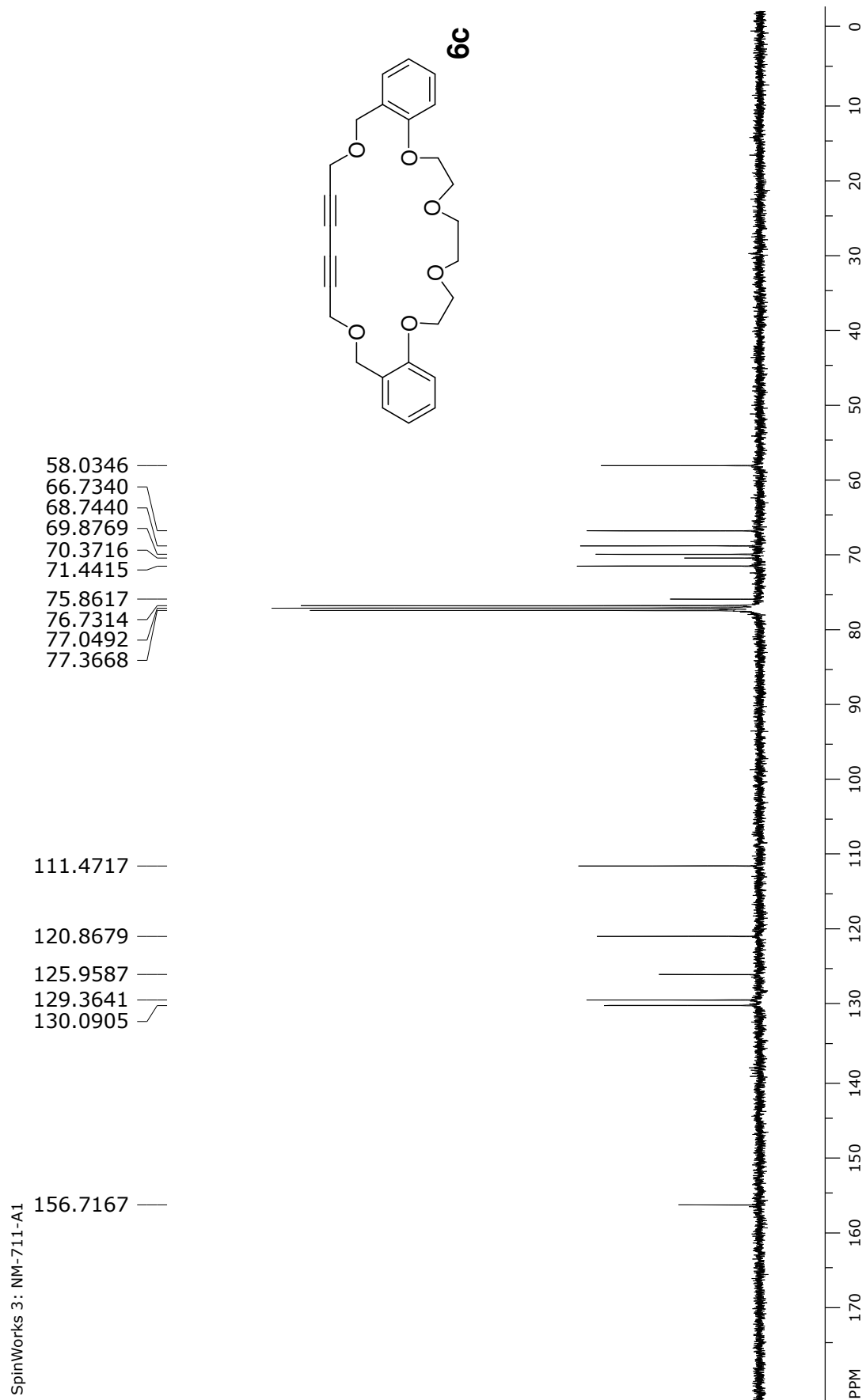
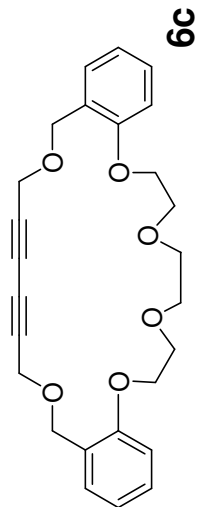
113.4732 —
113.8207 —

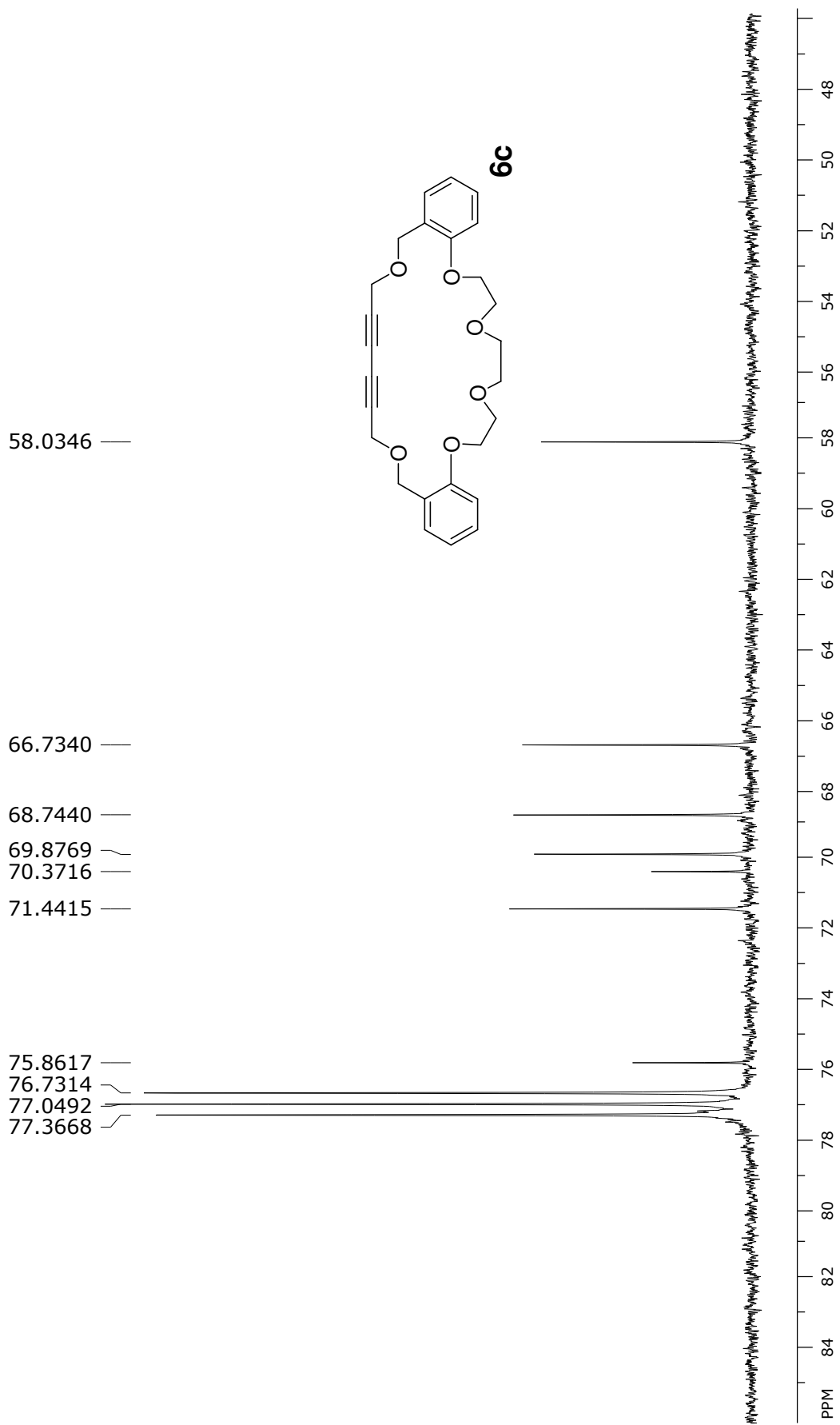




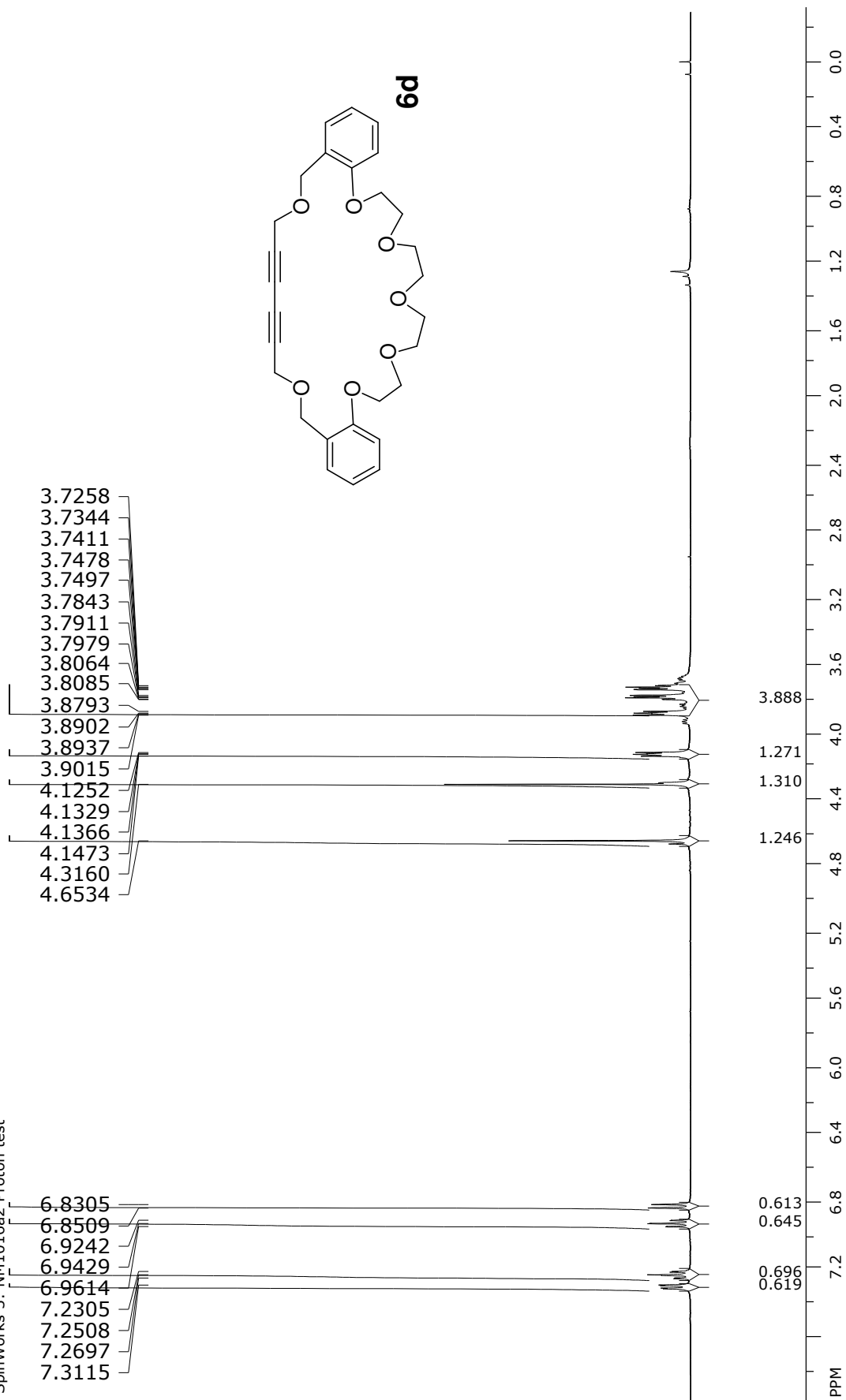




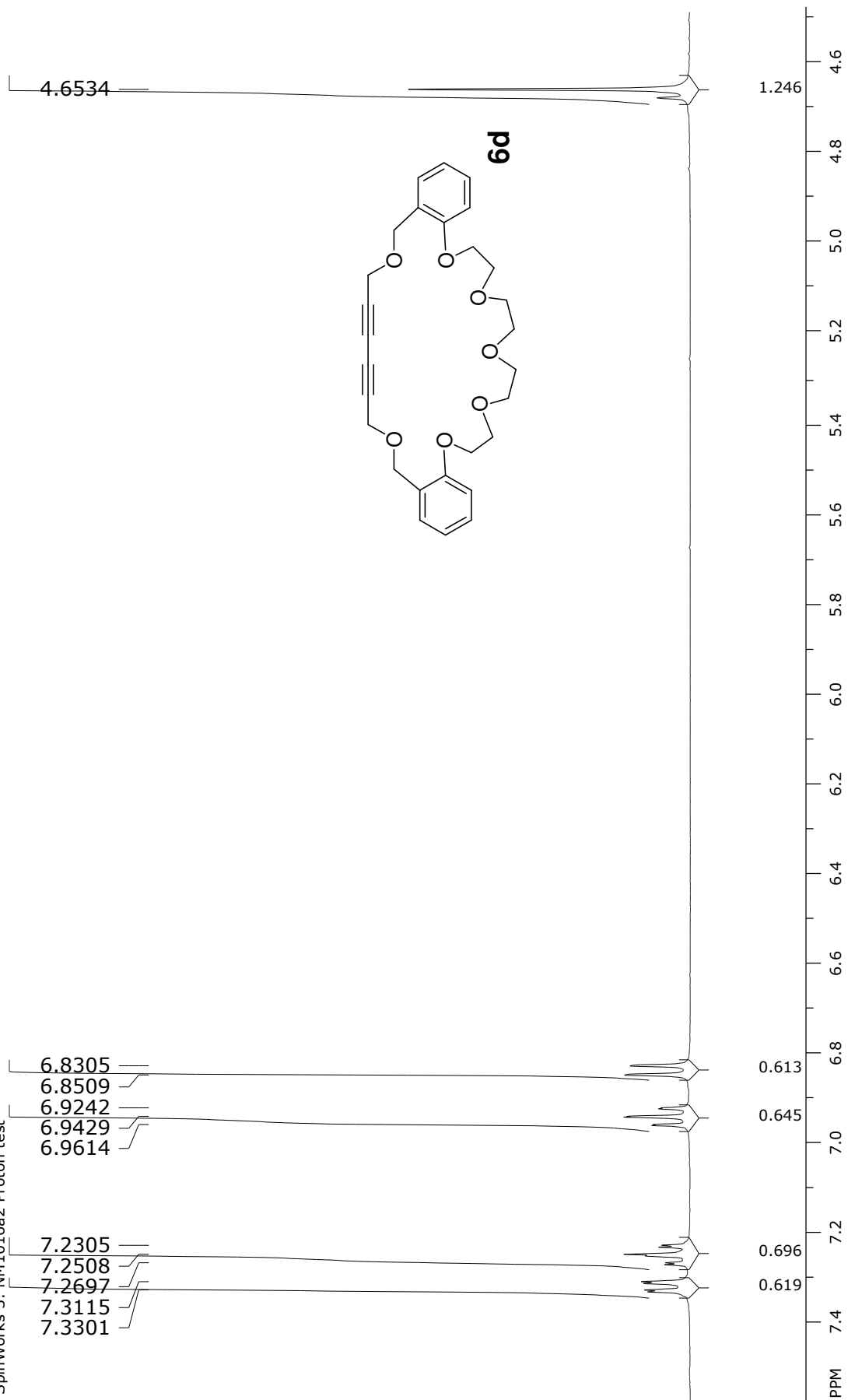


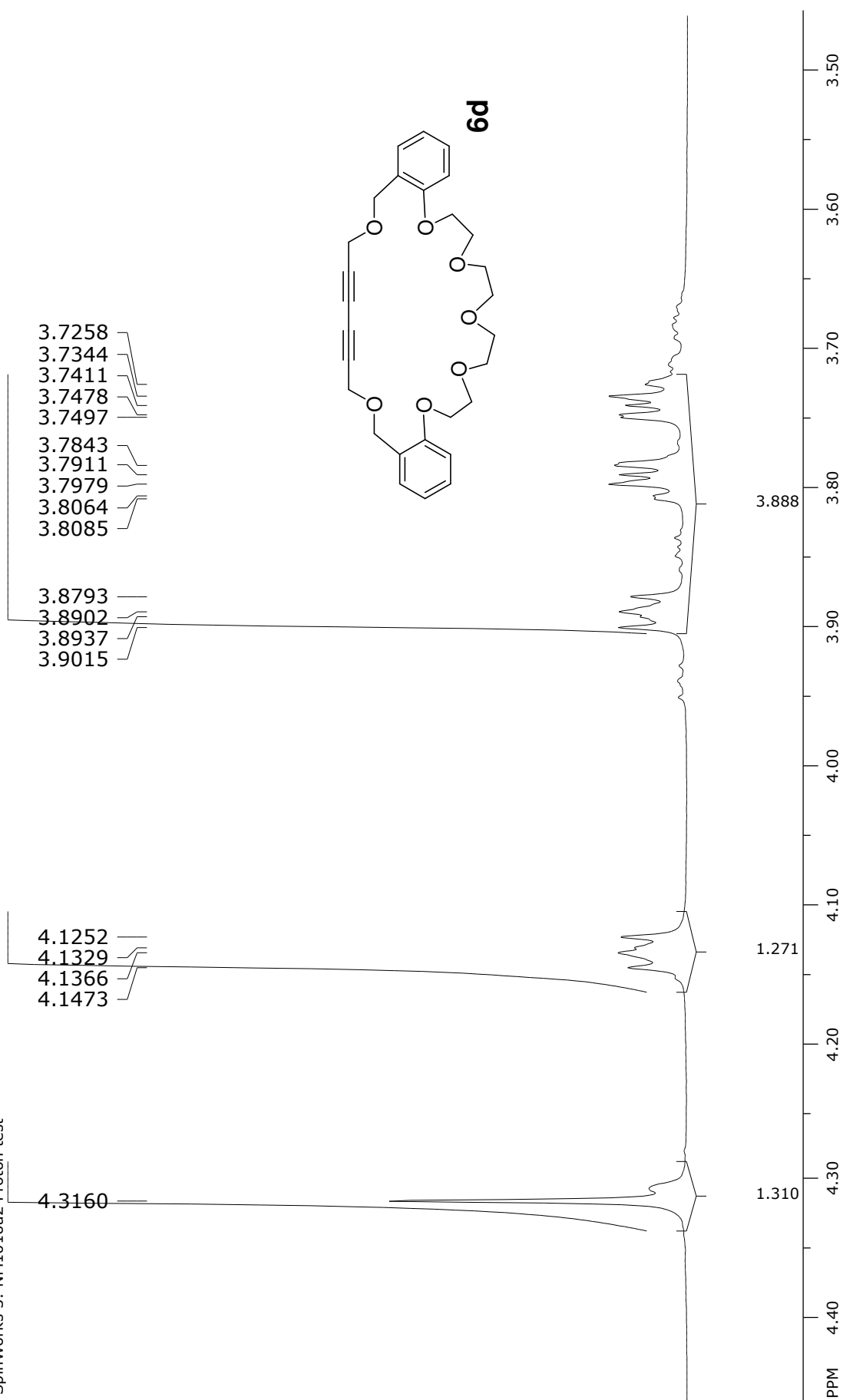


SpinWorks 3: NM1016a2 Proton test



SpinWorks 3: NM1016a2 Proton test





SpinWorks 3: NM1016a2 Carbon test

58.1308
66.8654
68.2133
69.6640
70.3724
70.7046
71.0723
75.9195
76.7990
77.1162
77.4349

111.5653 —

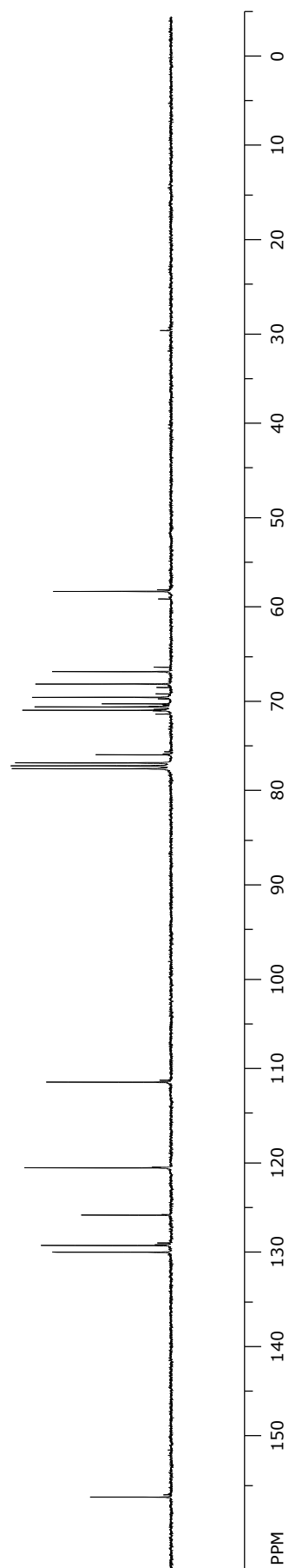
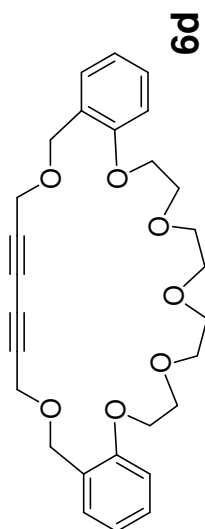
120.8893 —

126.0407 —

129.3447 —

130.0752 —

156.7429 —



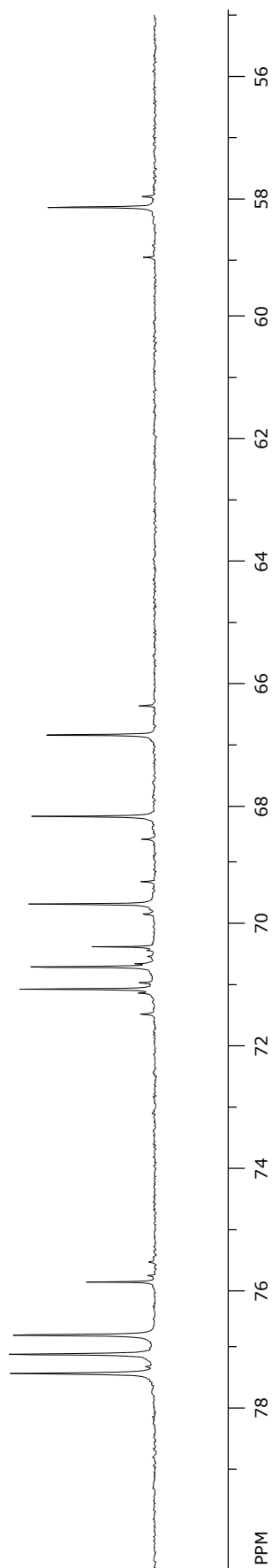
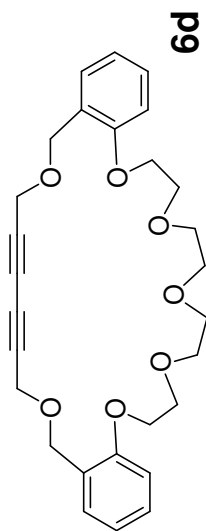
SpinWorks 3: NM1016a2 Carbon test

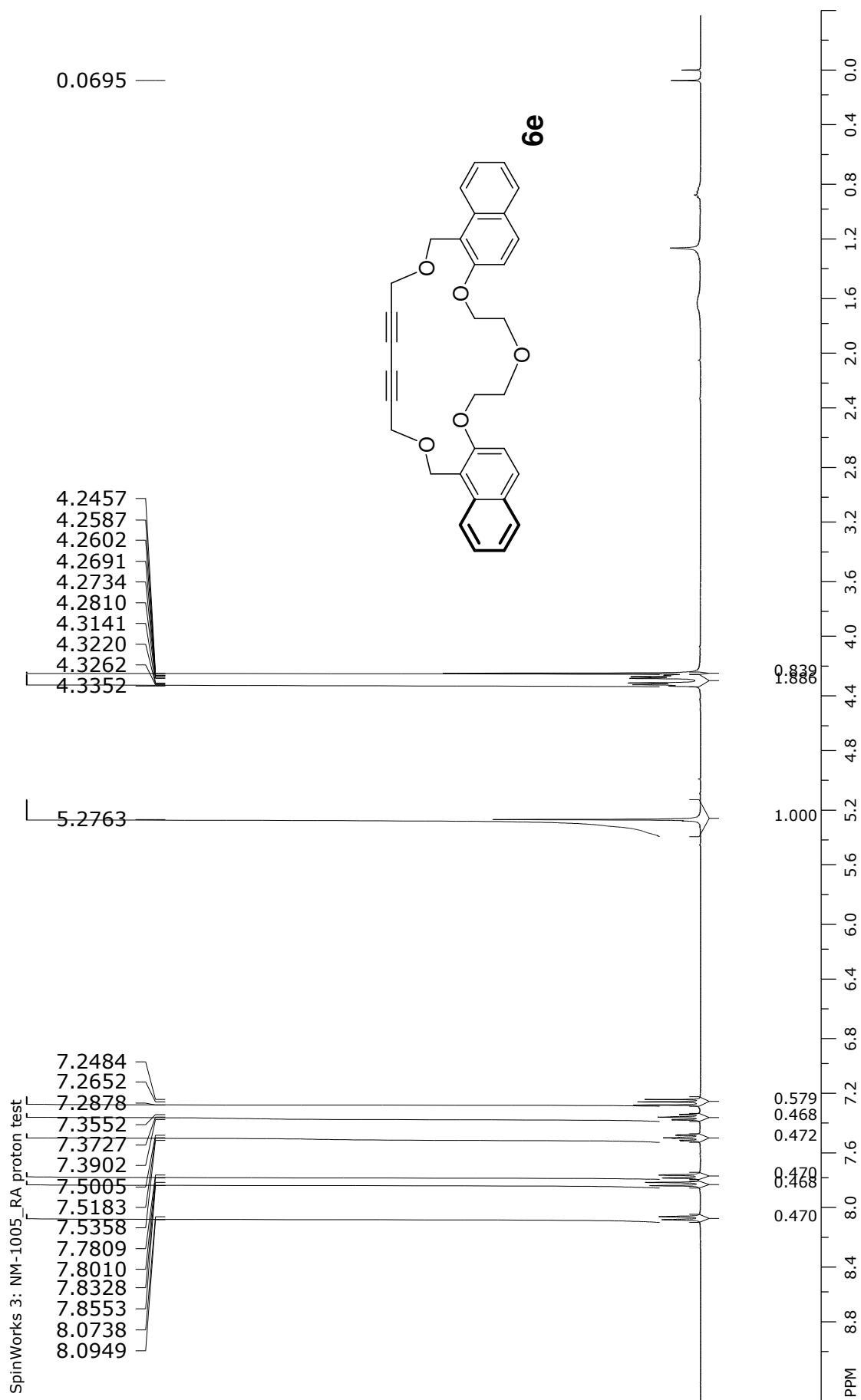
75.9195 —
76.7990 —
77.1162 —
77.4349 —

70.3724 —
70.7046 —
71.0723 —

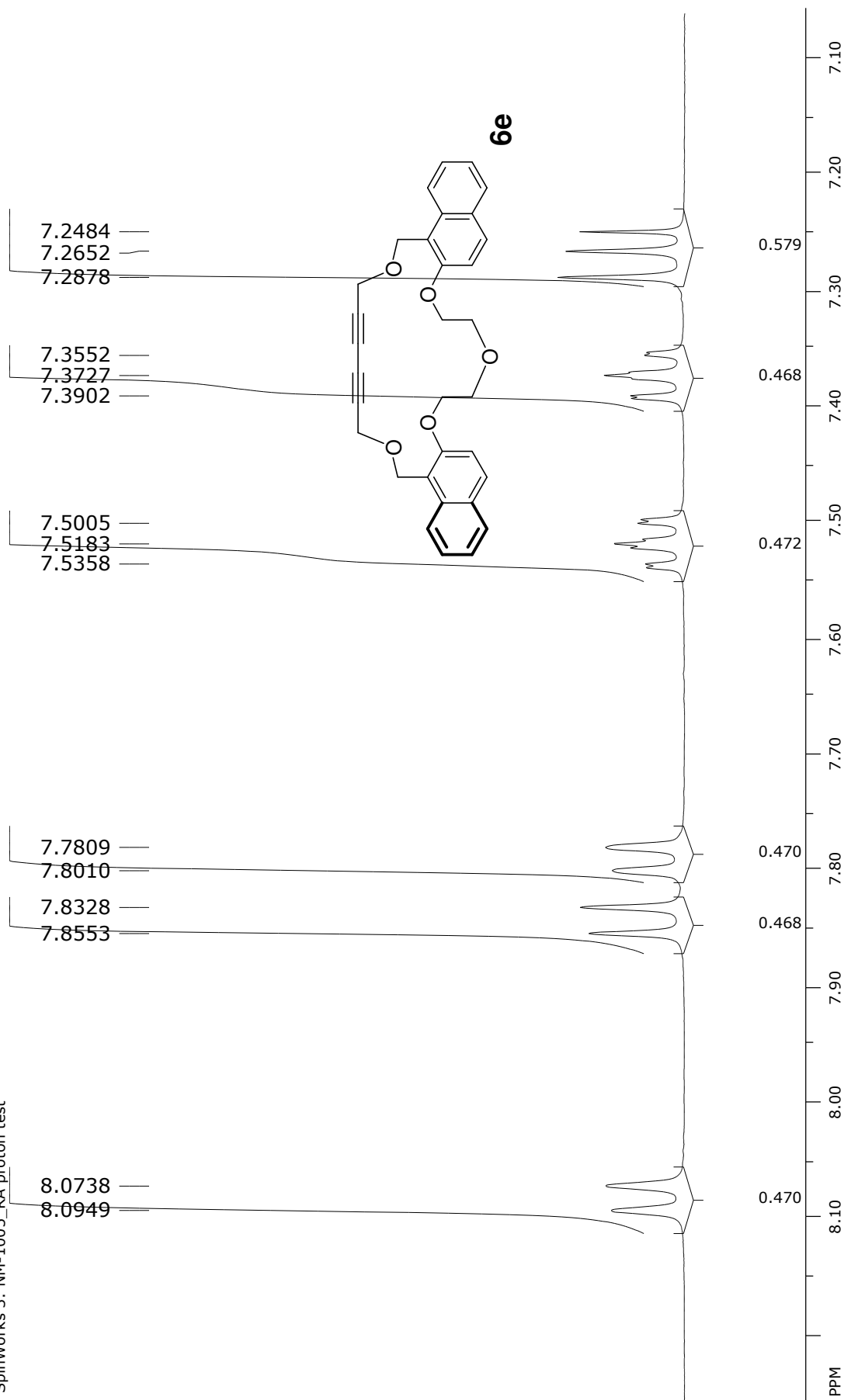
68.2133 —
66.8654 —

58.1308 —

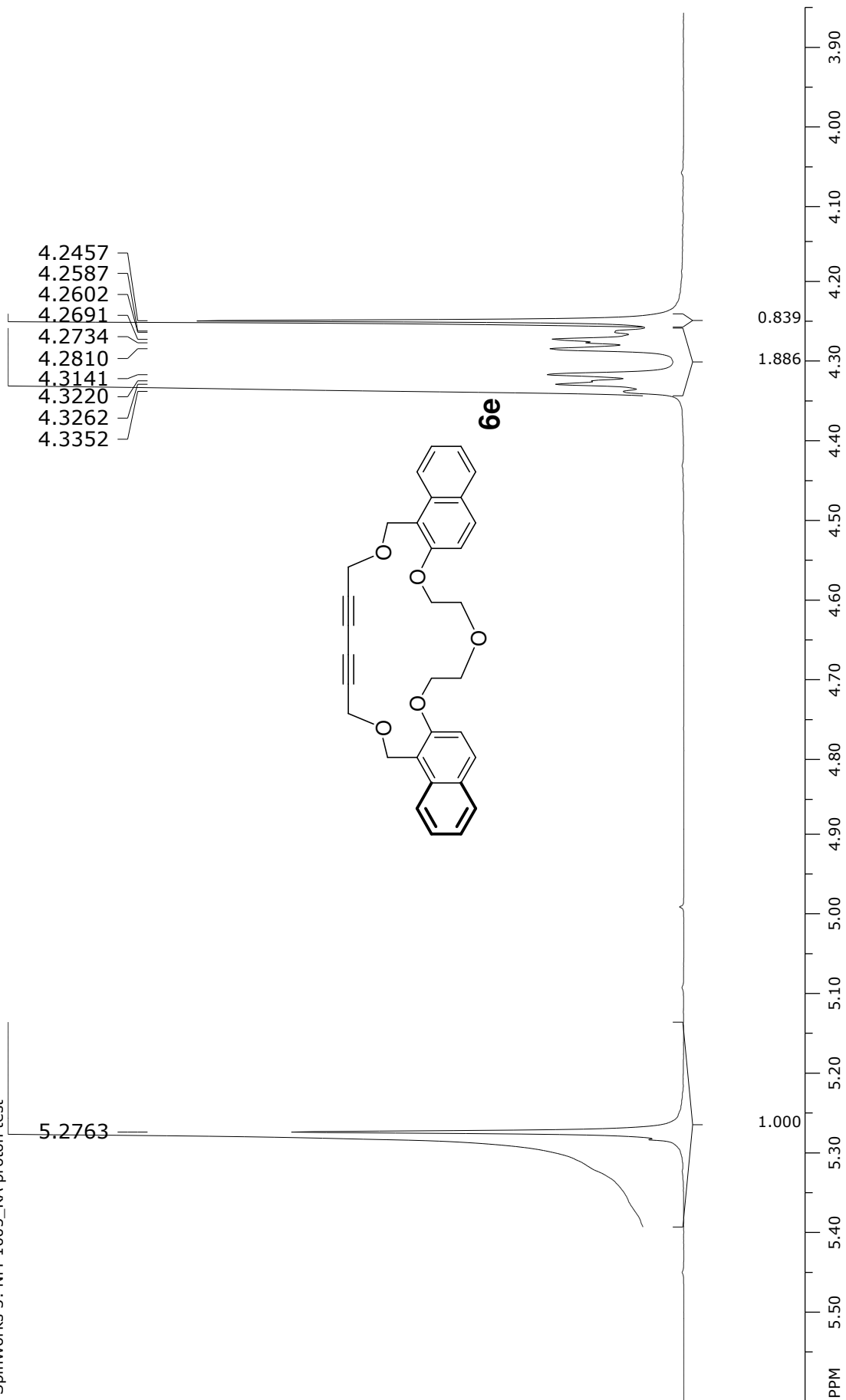




SpinWorks 3: NM-1005_RA proton test



SpinWorks 3: NM-1005_RA proton test



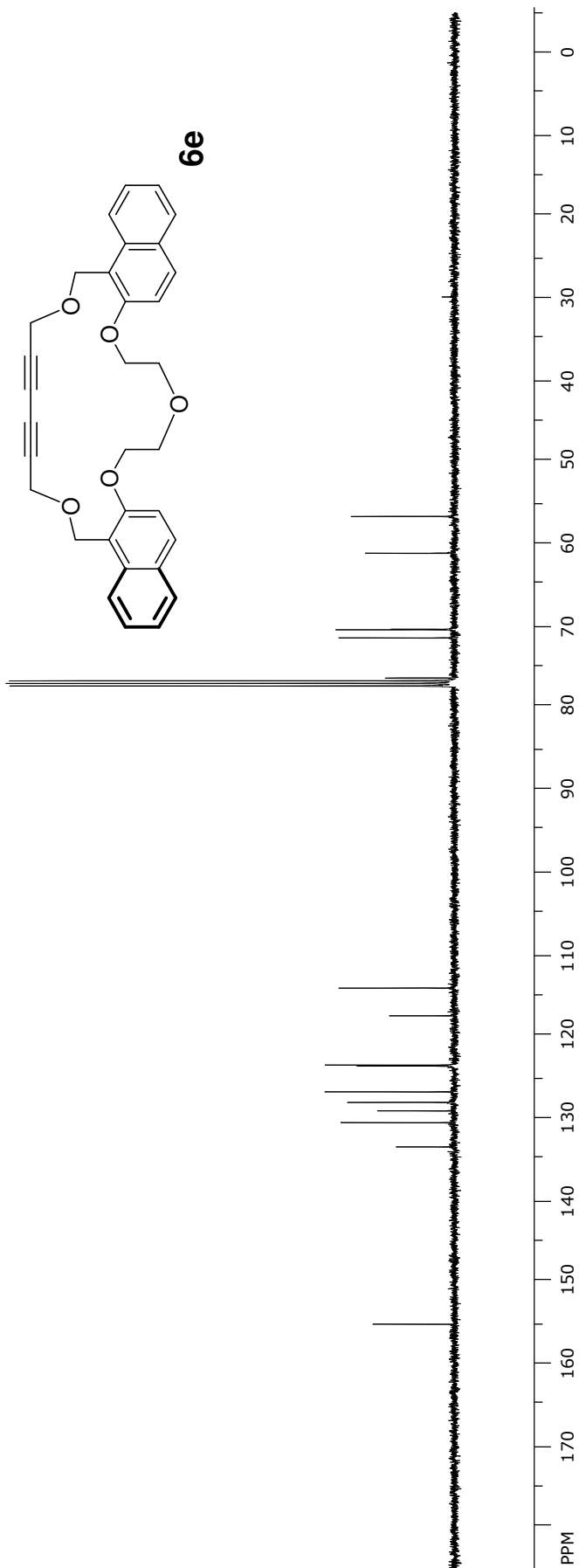
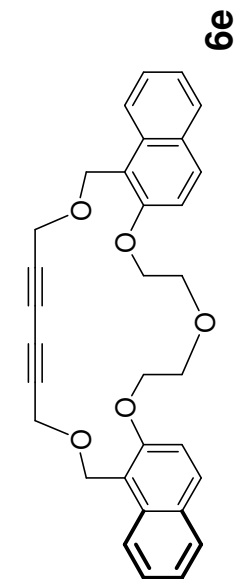
SpinWorks 3: NM 1005 RA C13 test

alpha	Final Number of Nodes (approx.)
0.0	56.6073
0.1	61.1127
0.2	70.4208
0.3	70.4807
0.4	71.4865
0.5	76.3955
0.6	76.7339
0.7	77.0506
0.8	77.3690

114.3609 —
117.7540 —

123.7904
123.9113
127.0714
128.3618
129.3965
130.8444
133.8108

155.5235 —



SpinWorks 3: NM 1005 RA C13 test

76.3955 —

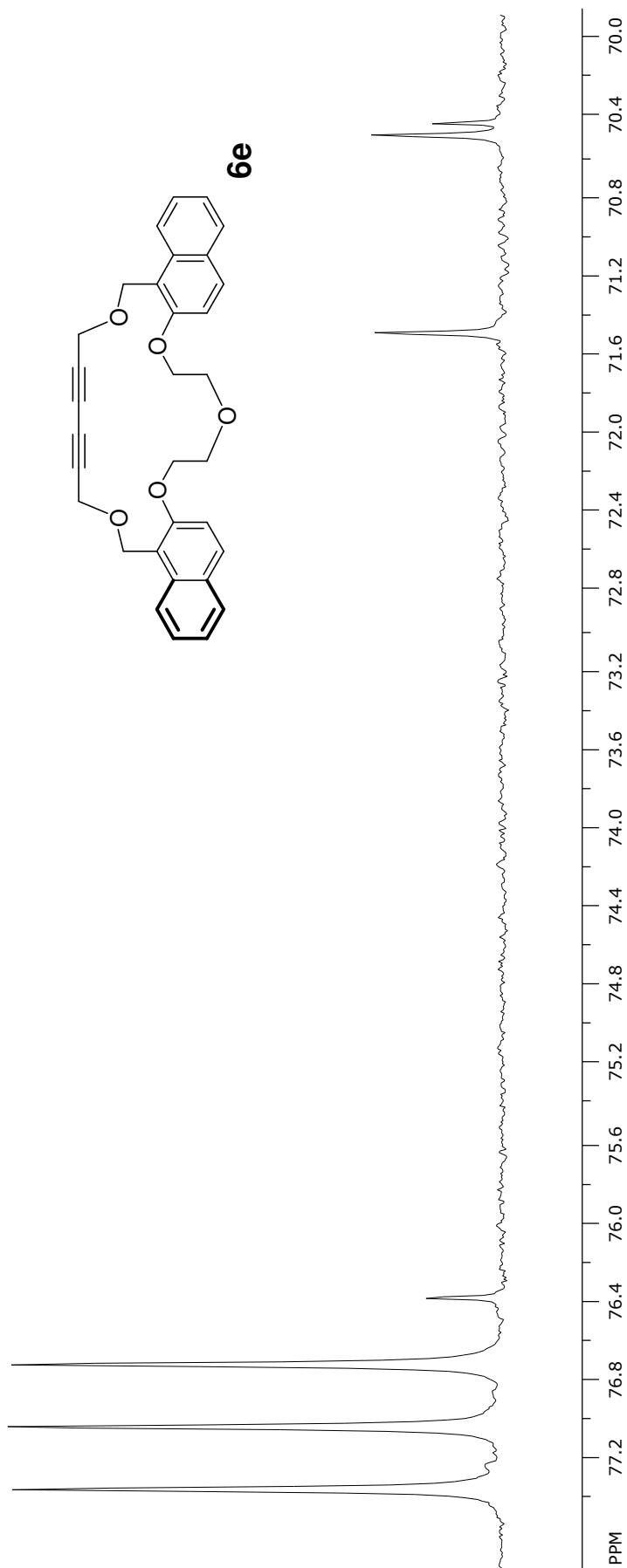
76.7339 —

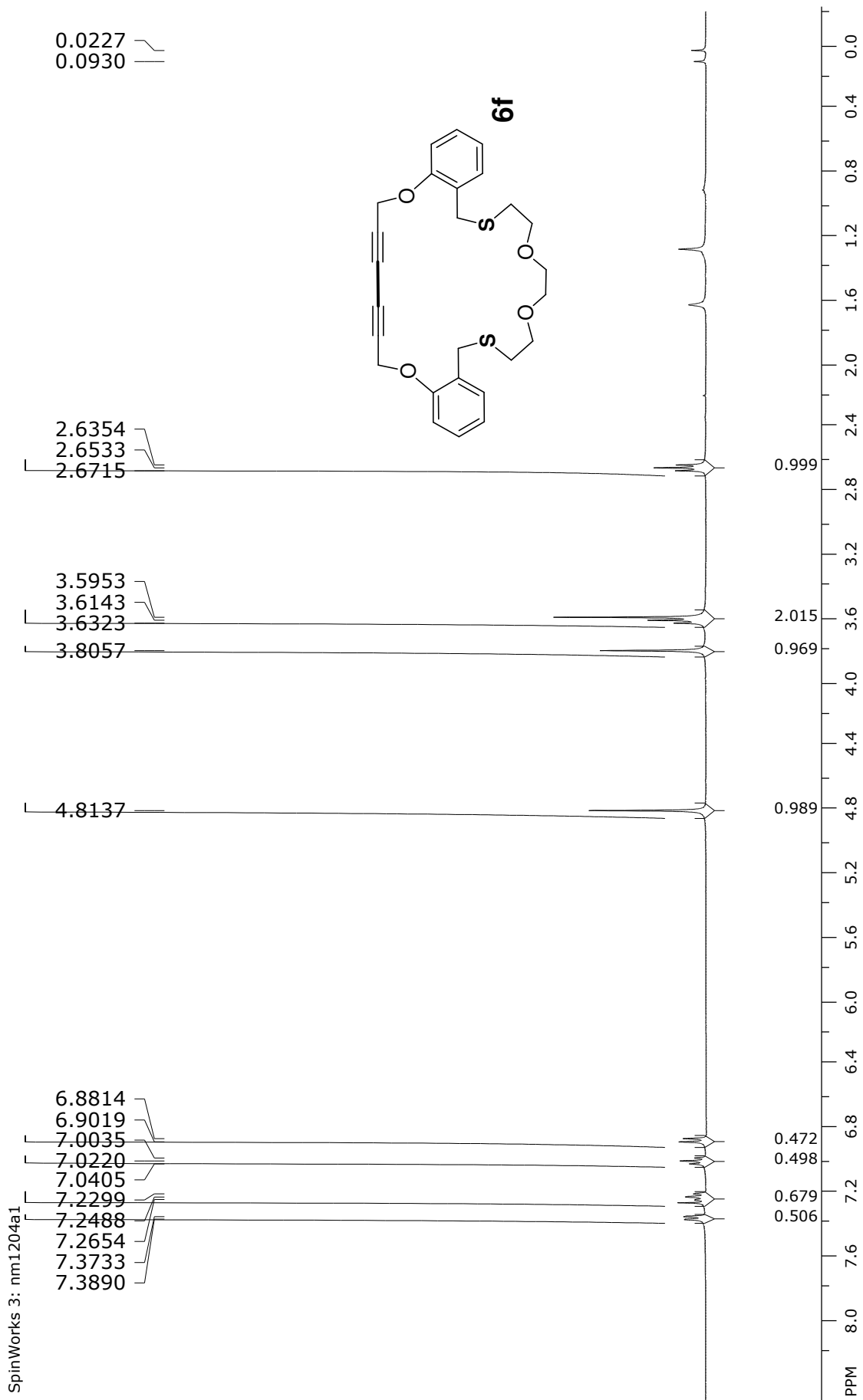
77.0506 —

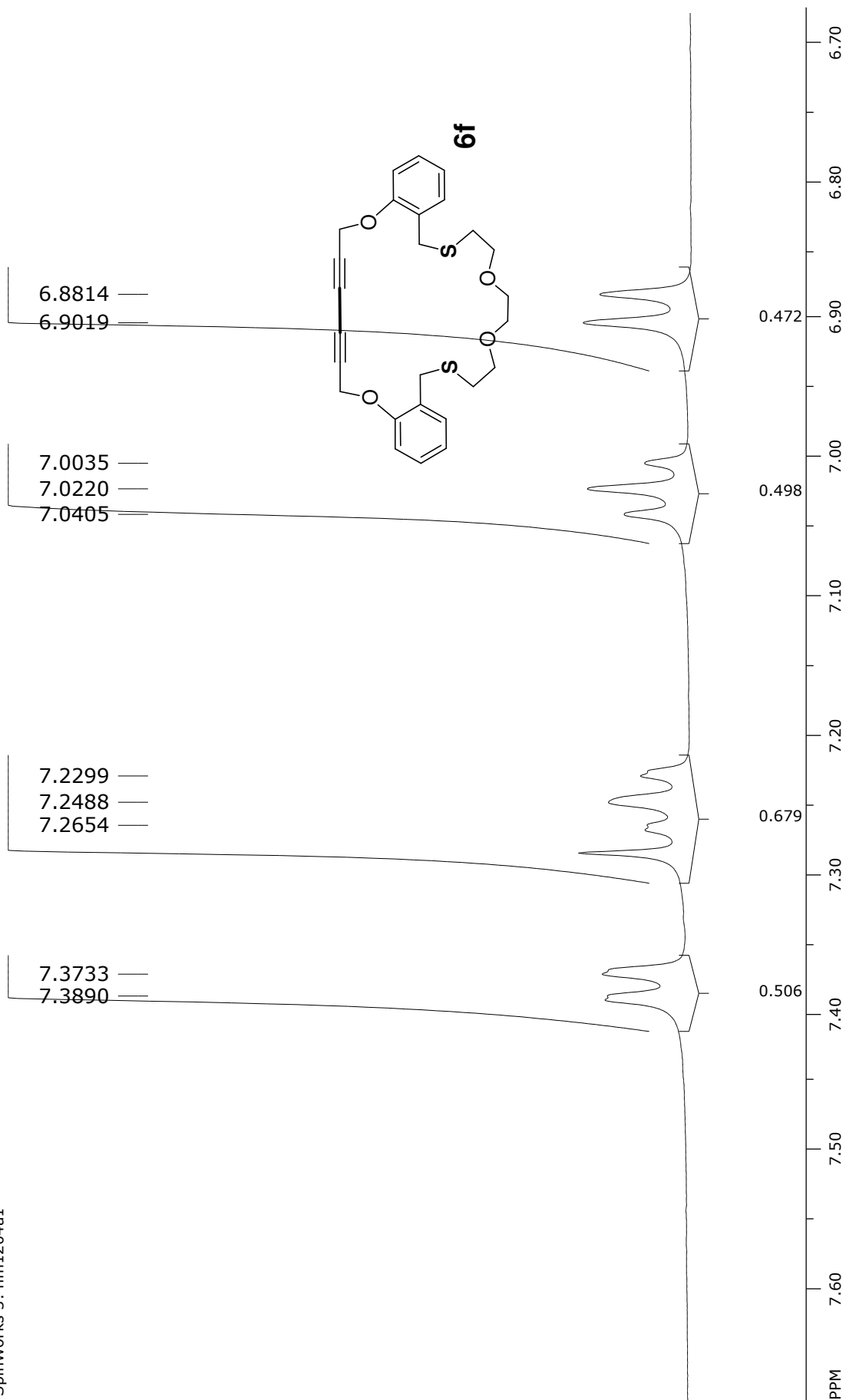
77.3690 —

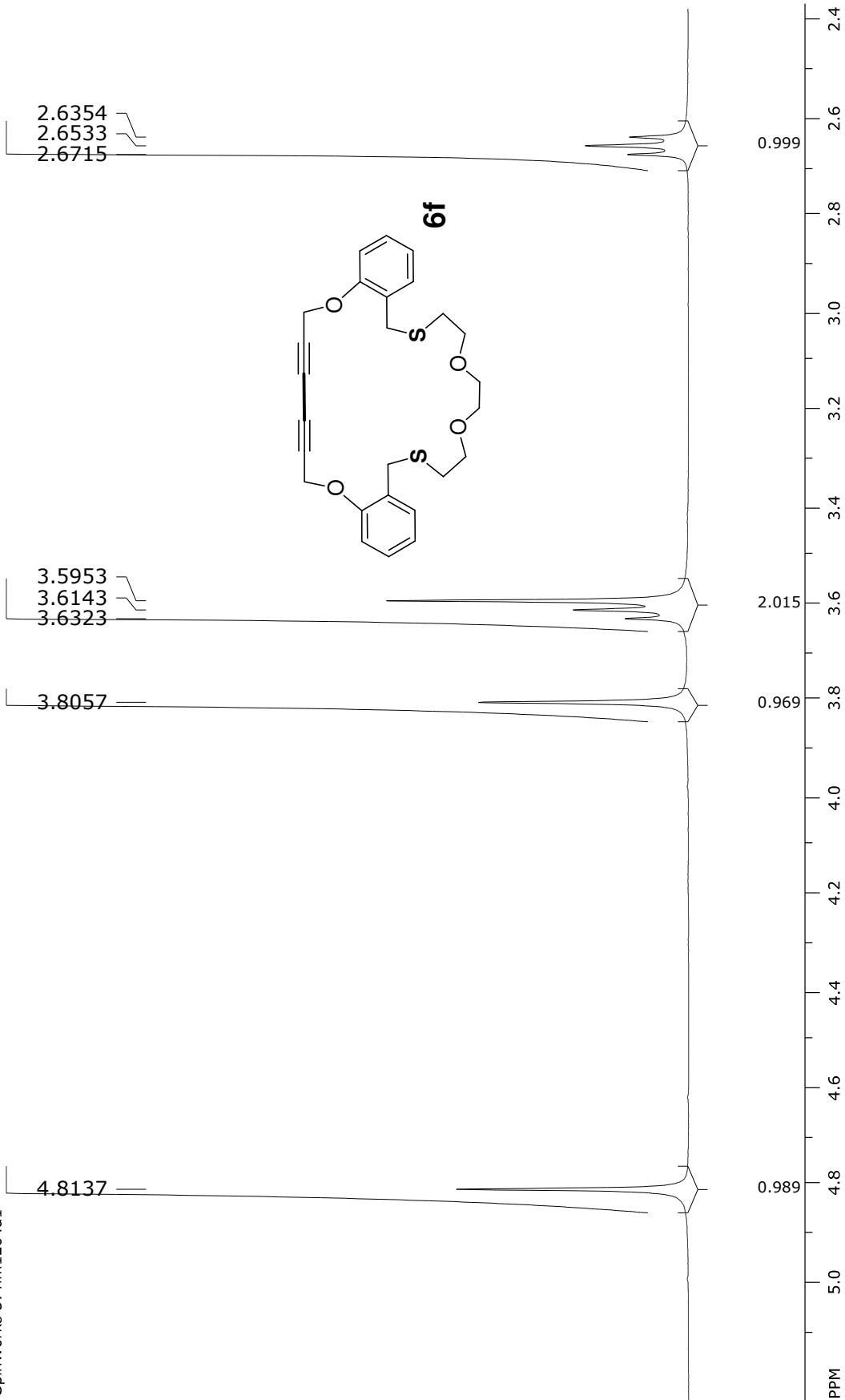
71.4865 —

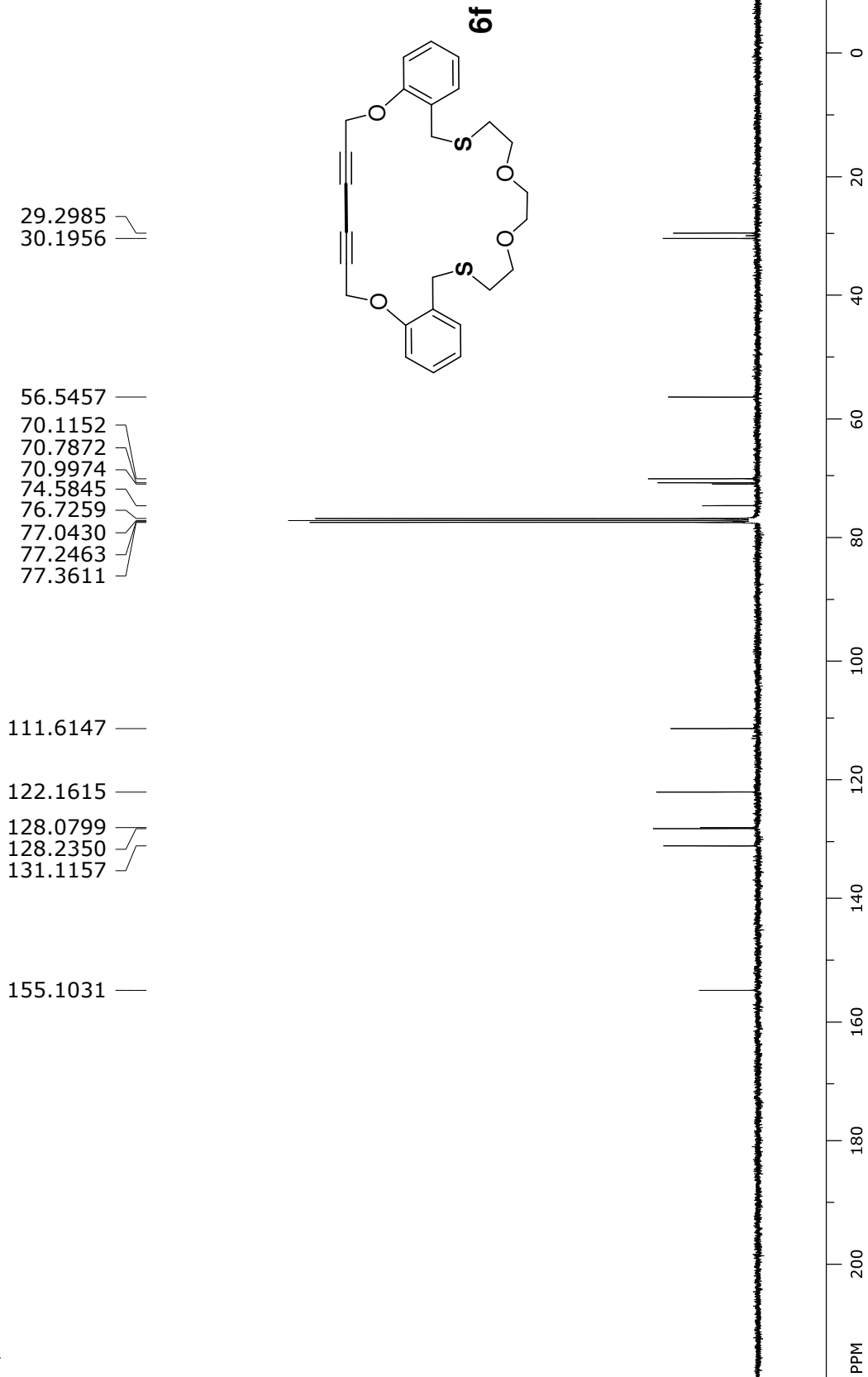
70.4208 —
70.4807 —









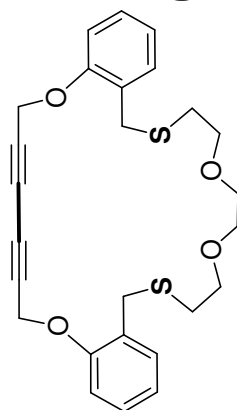
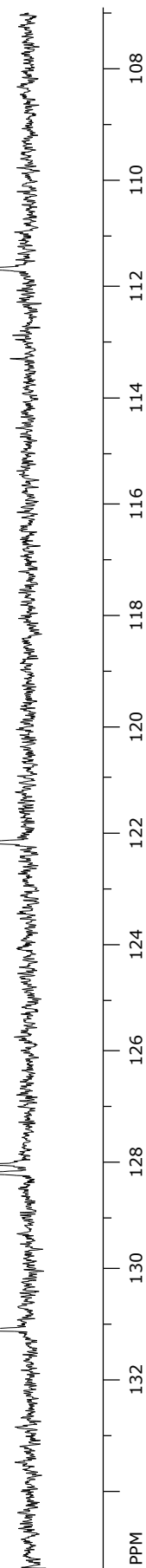


131.1157 —

128.0799 —
128.2350 —

122.1615 —

111.6147 —

**6f**

SpinWorks 3: nm-1204-a1

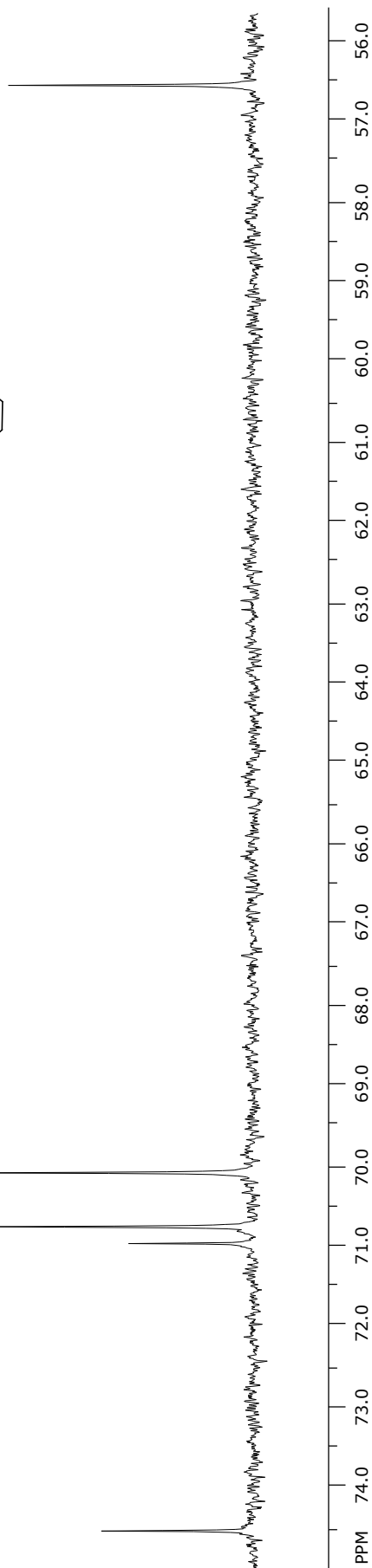
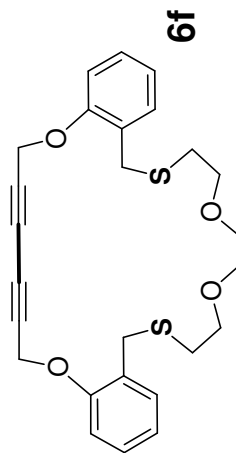
74.5845 —

70.1152 —

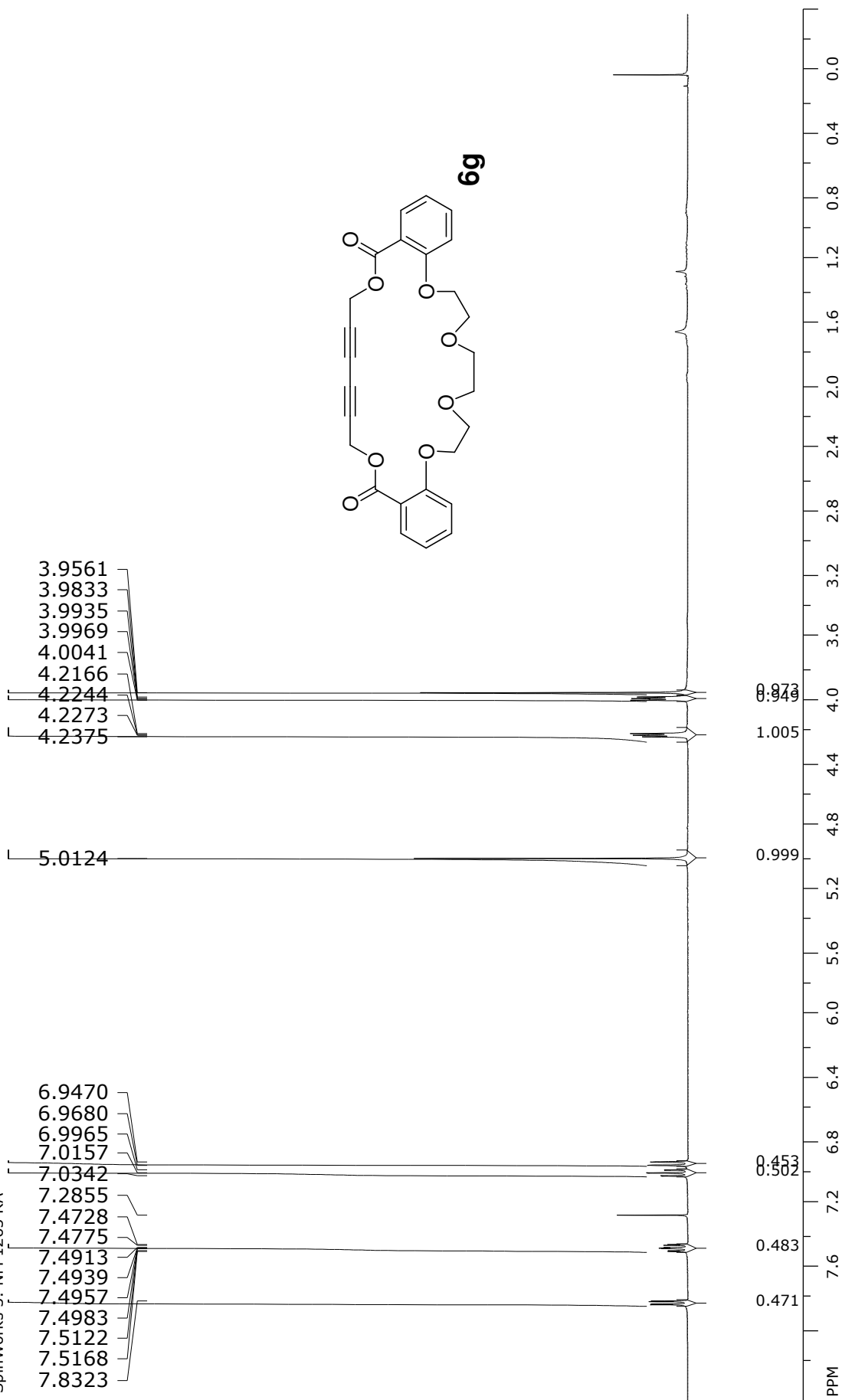
70.7872 —

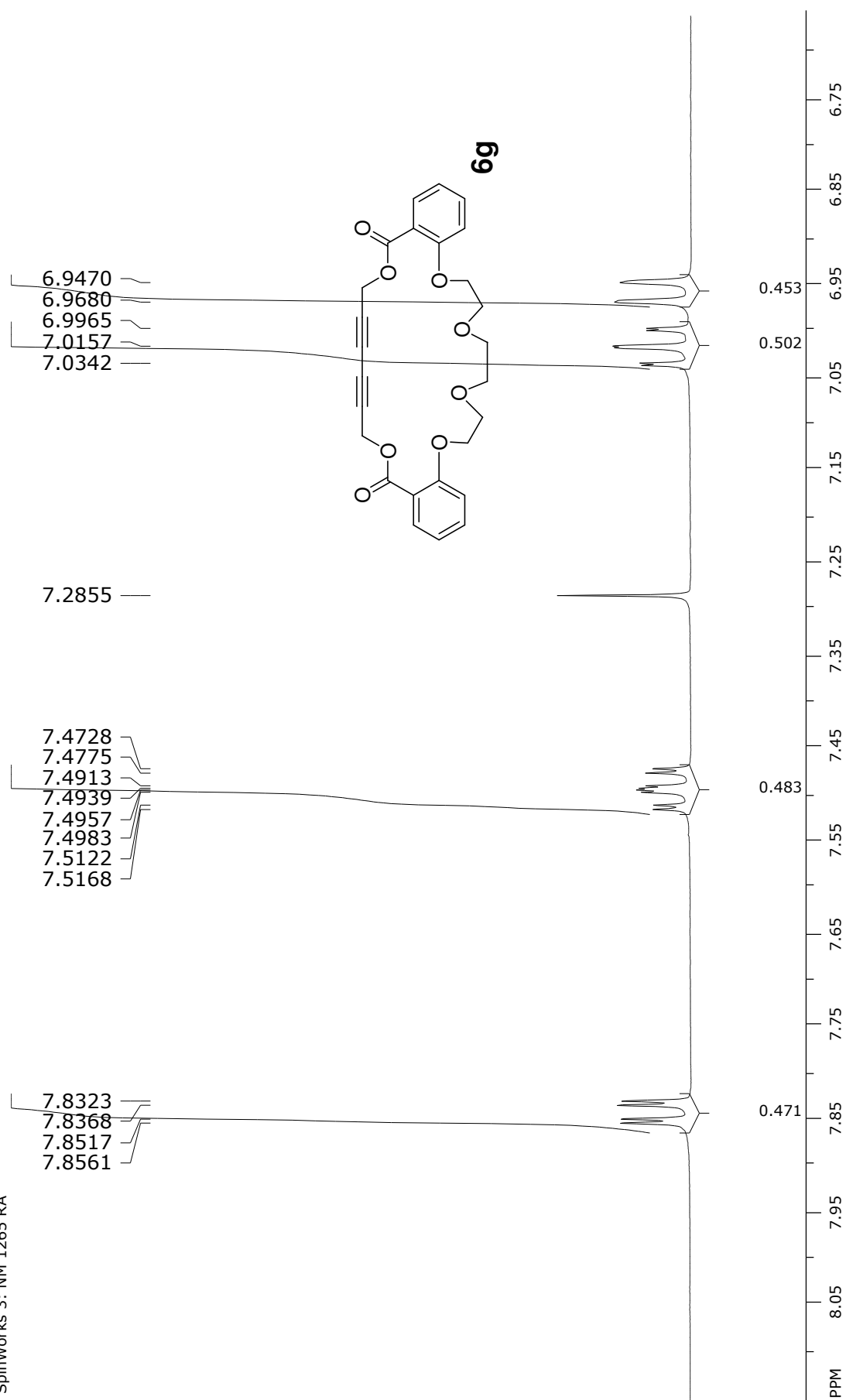
70.9974 —

56.5457 —

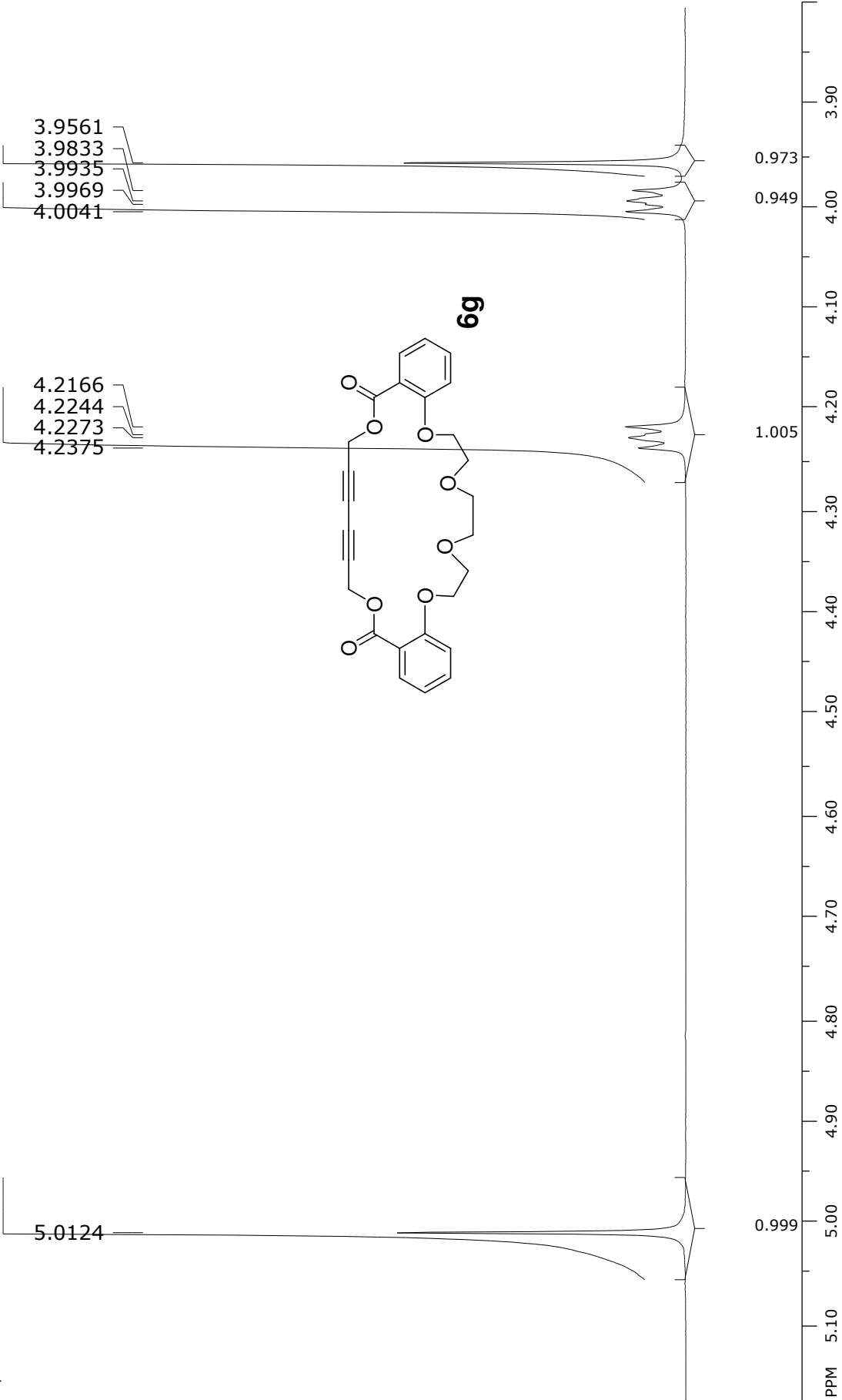


SpinWorks 3: NM 1265 RA





SpinWorks 3: NM 1265 RA



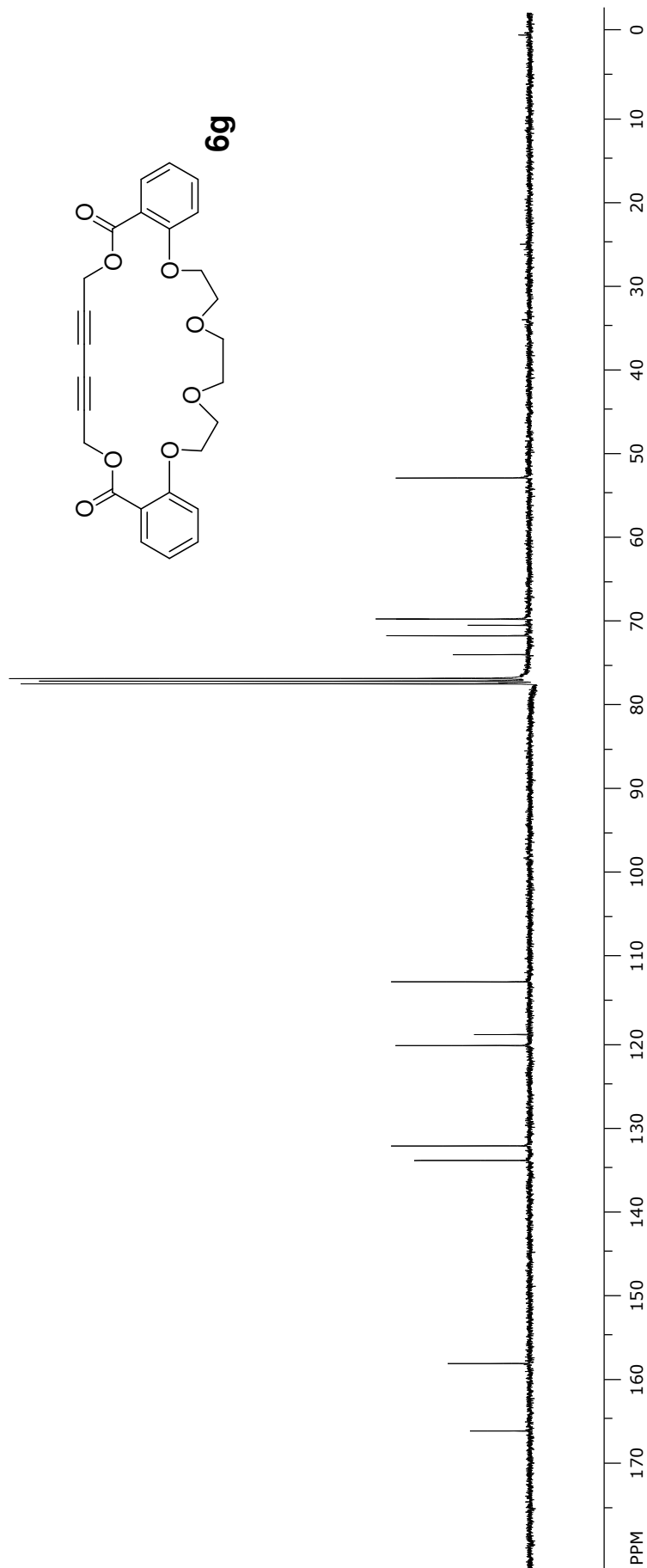
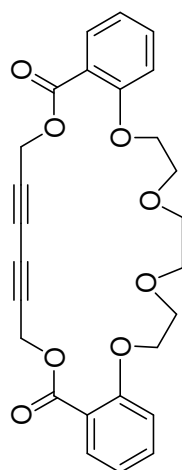
SpinWorks 3: NM 1265 RA

52.8212 —
69.6350
69.6553
70.3858
71.6386
73.8847
76.7280
77.0456
77.2491
77.3631

112.8857 —
119.1737 —
120.4841

132.4482 —
134.1811

158.3906 —
166.4275 —



SpinWorks 3: NM 1265 RA

76.7280
77.0456
77.2491
77.3631

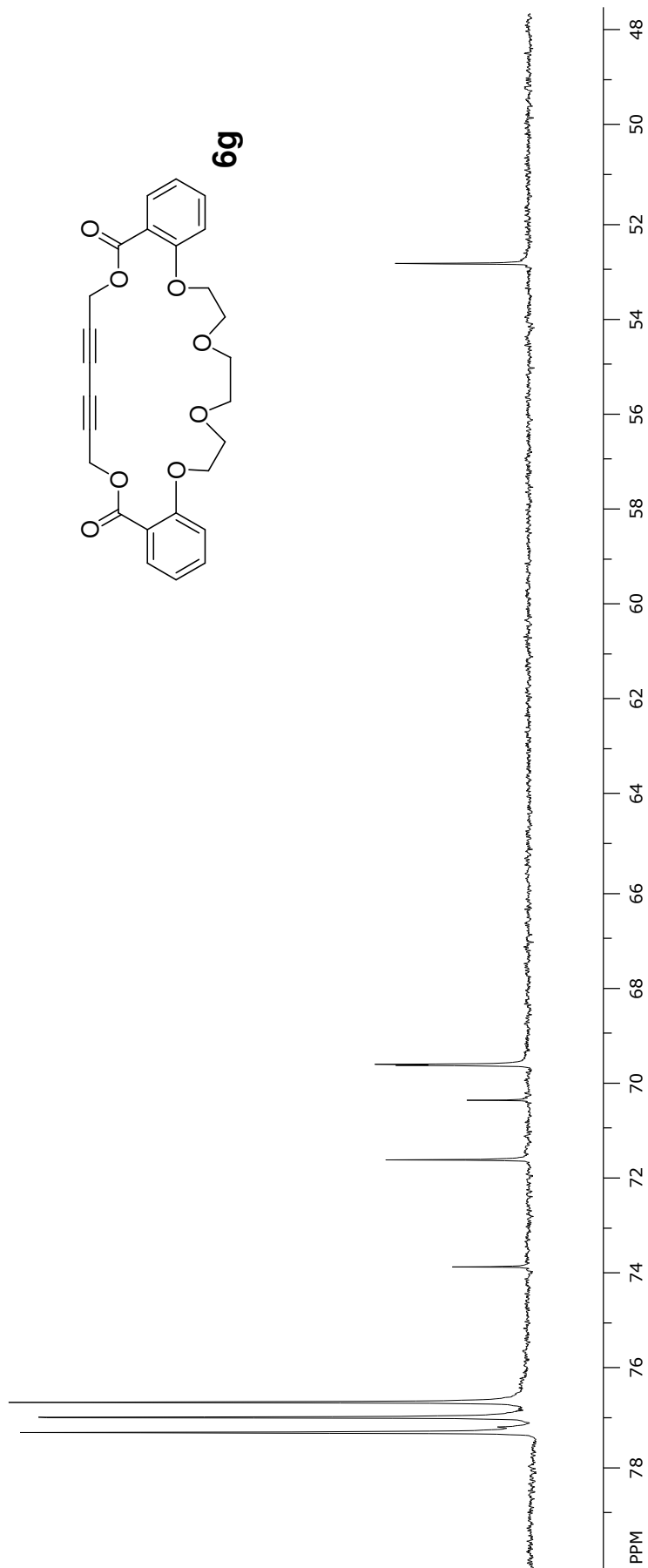
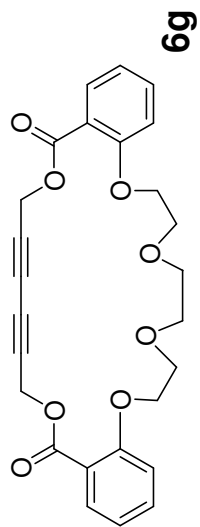
73.8847

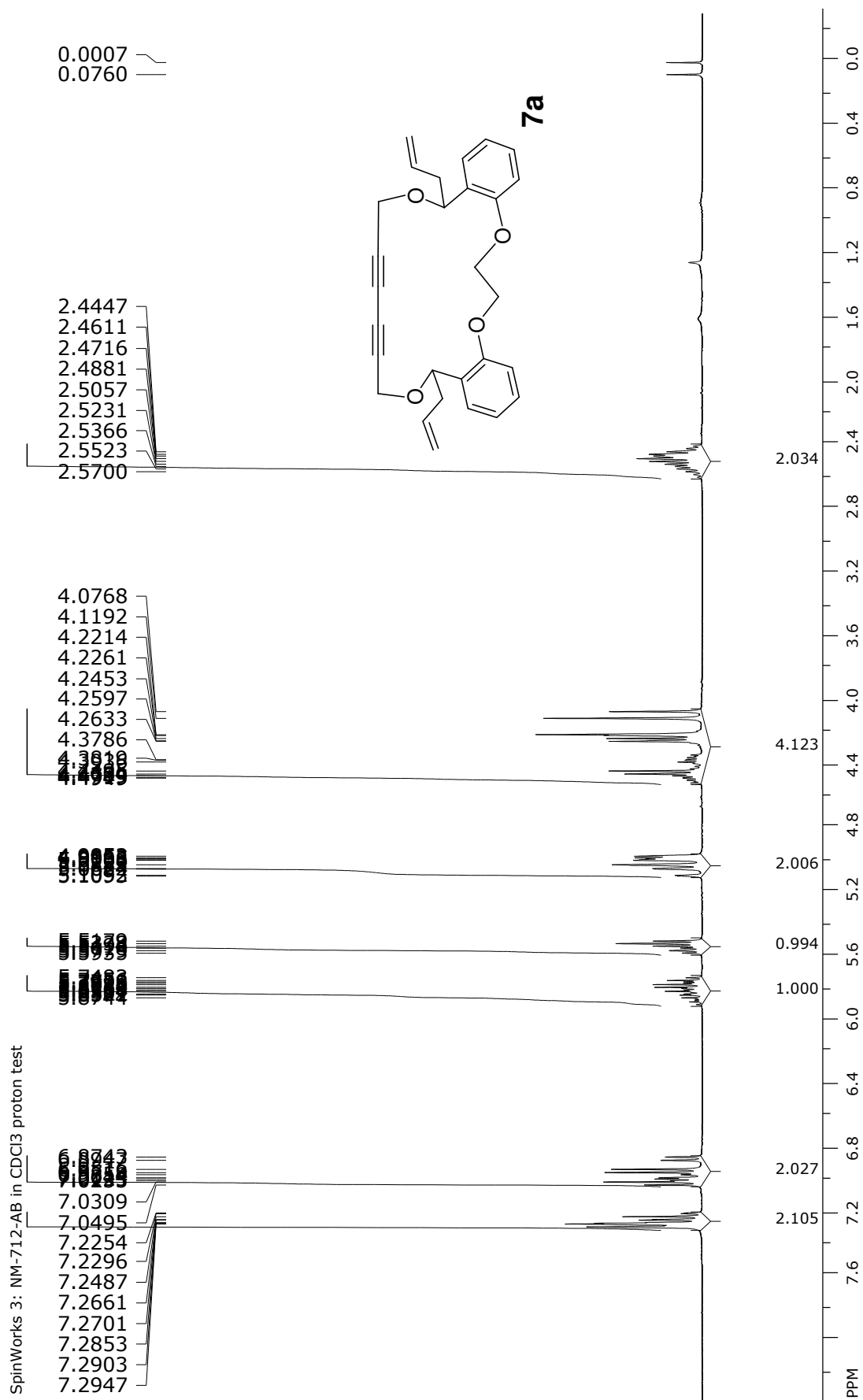
71.6386

70.3858

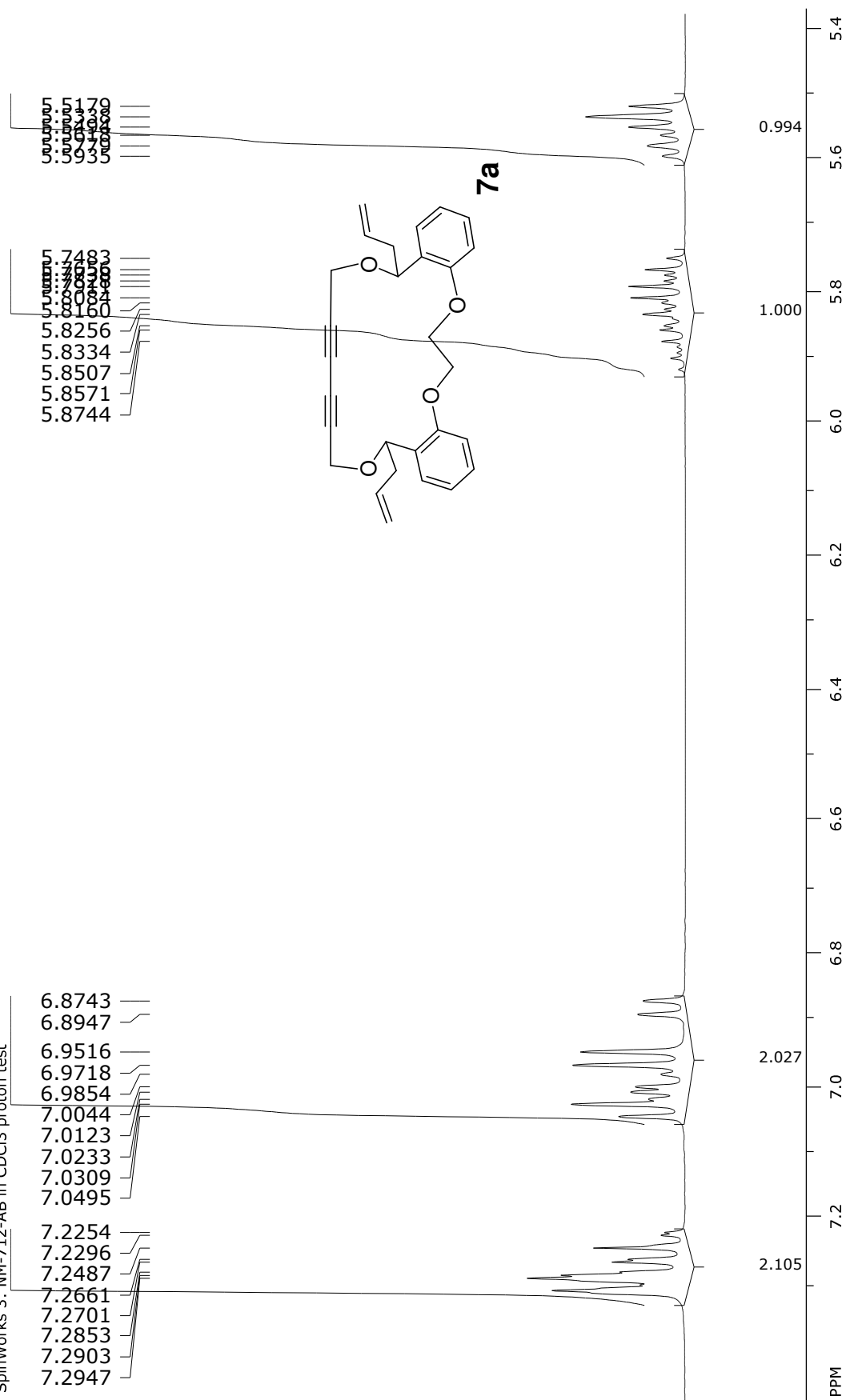
69.6350
69.6553

52.8212

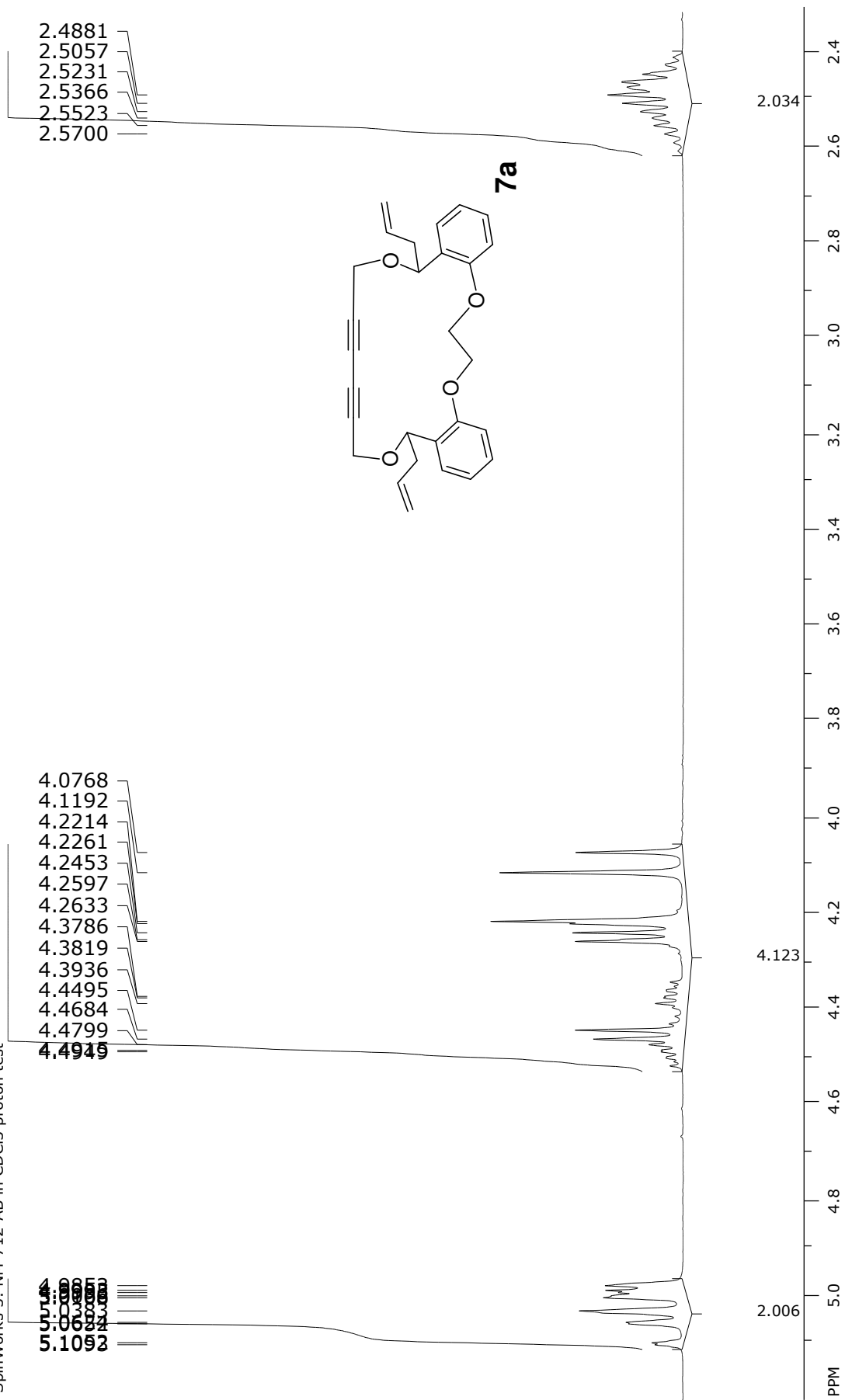




SpinWorks 3: NM-712-AB in CDCl3 proton test



SpinWorks 3: NM-712-AB in CDCl3 proton test

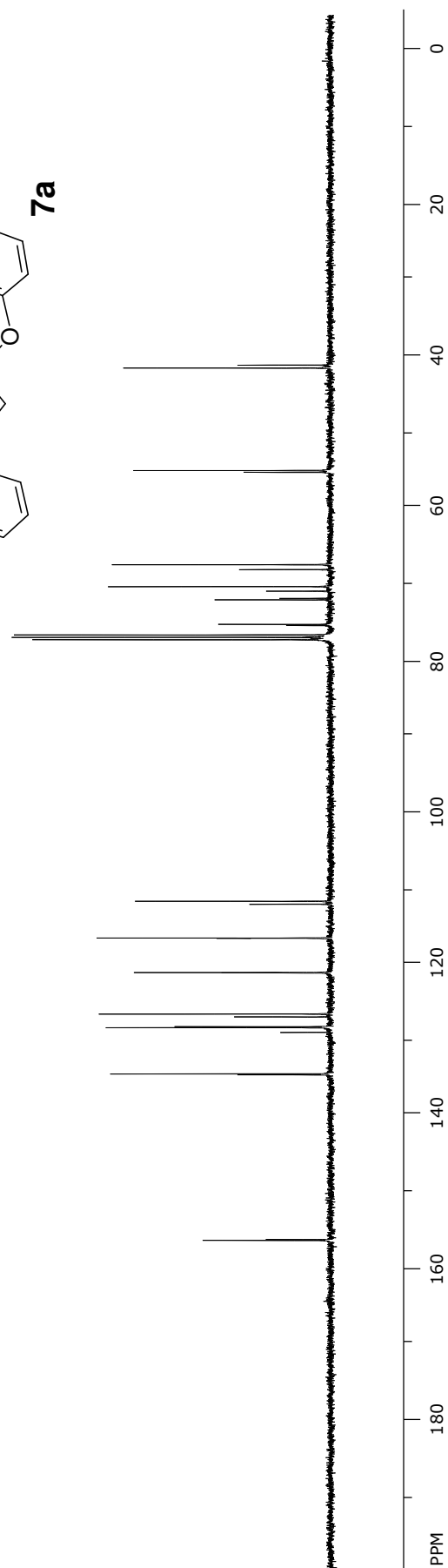
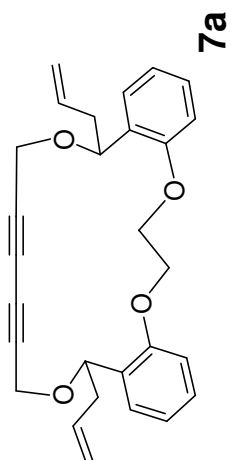


SpinWorks 3: Nm712ab Carbon test

41.1640
41.5574
55.0744
55.3114
67.4912
68.1381
70.3830
70.9829
71.9305
72.1283
75.3713
75.5142
76.7573
77.0747
77.3931

111.8857
112.2722
116.7348
116.7661
121.2867
121.3294
126.7561
127.1434
128.4308
128.5272
128.5518
129.1773
134.6580
134.7790

156.5119
156.6170



SpinWorks 3: Nm712ab Carbon test

156.5119
156.6170

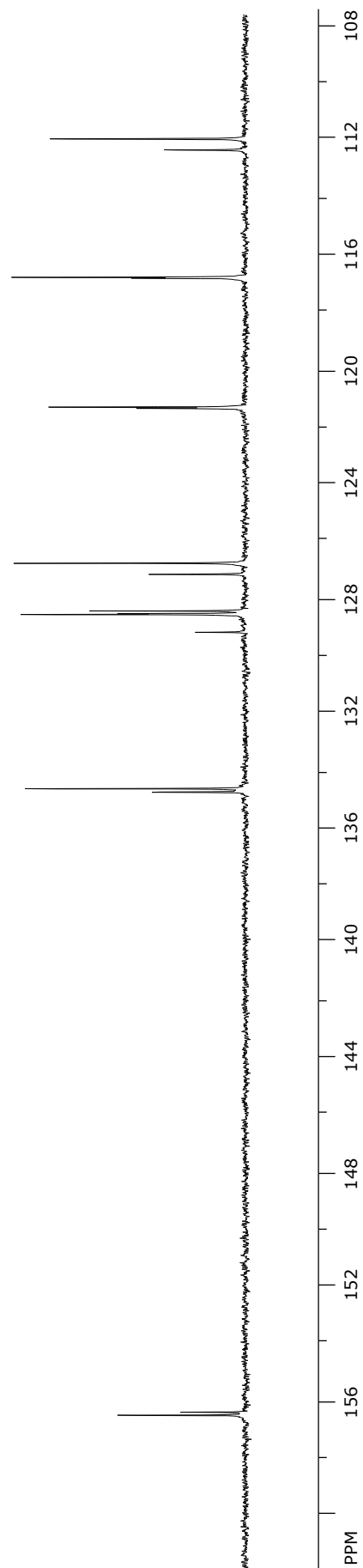
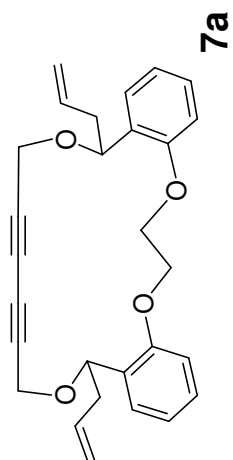
134.6580
134.7790

129.1773
128.5518
128.5272
128.4308
127.1434
126.7561

121.3294
121.2867

116.7661
116.7348

112.2722
111.8857



SpinWorks 3: Nm712ab Carbon test

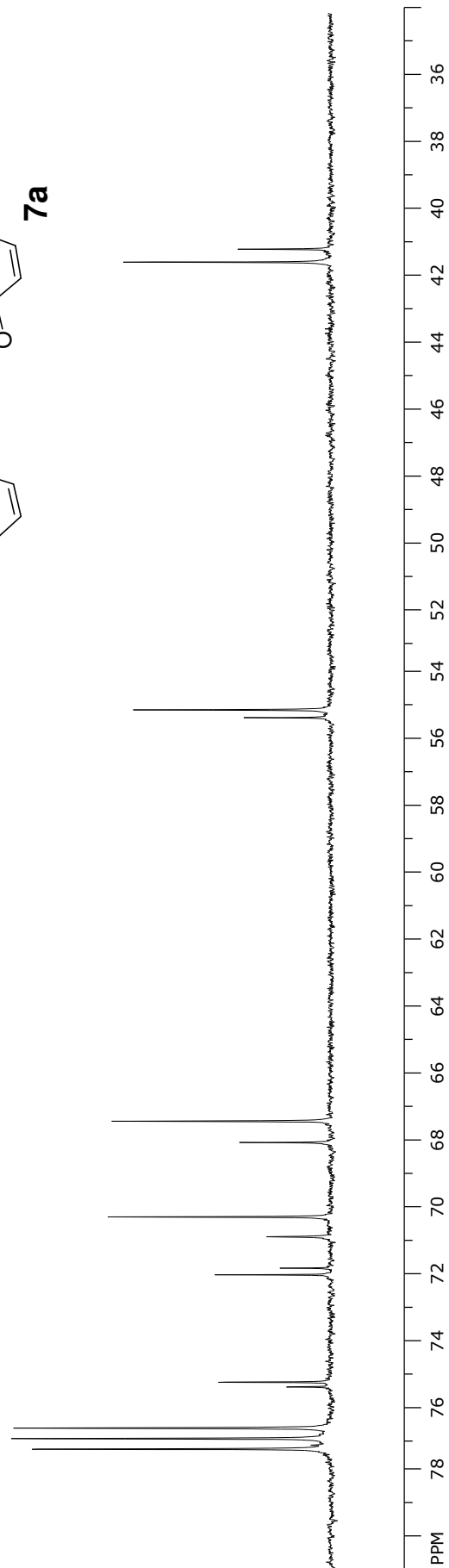
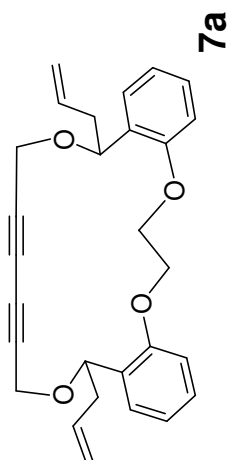
41.1640
41.5574

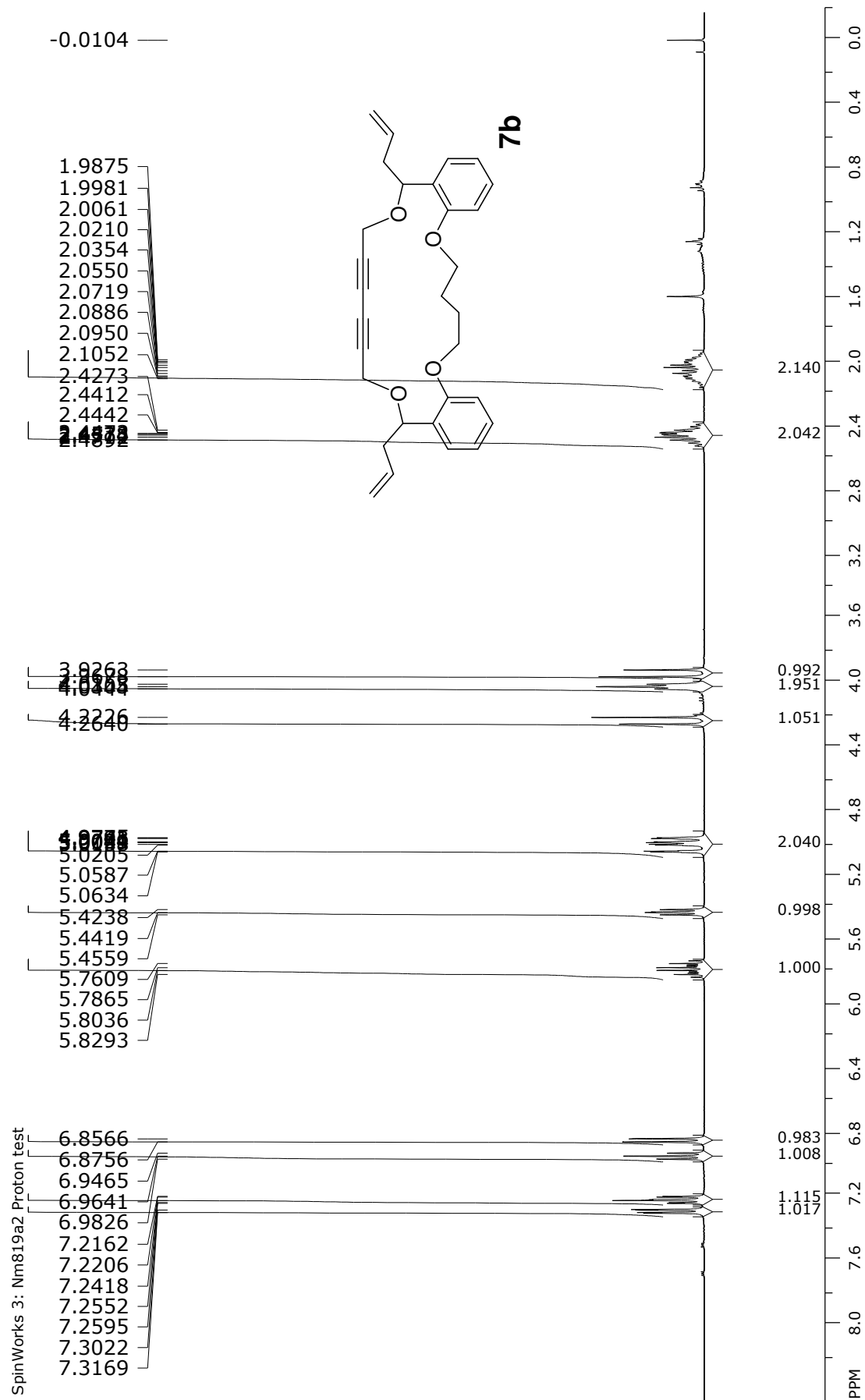
55.0744
55.3114

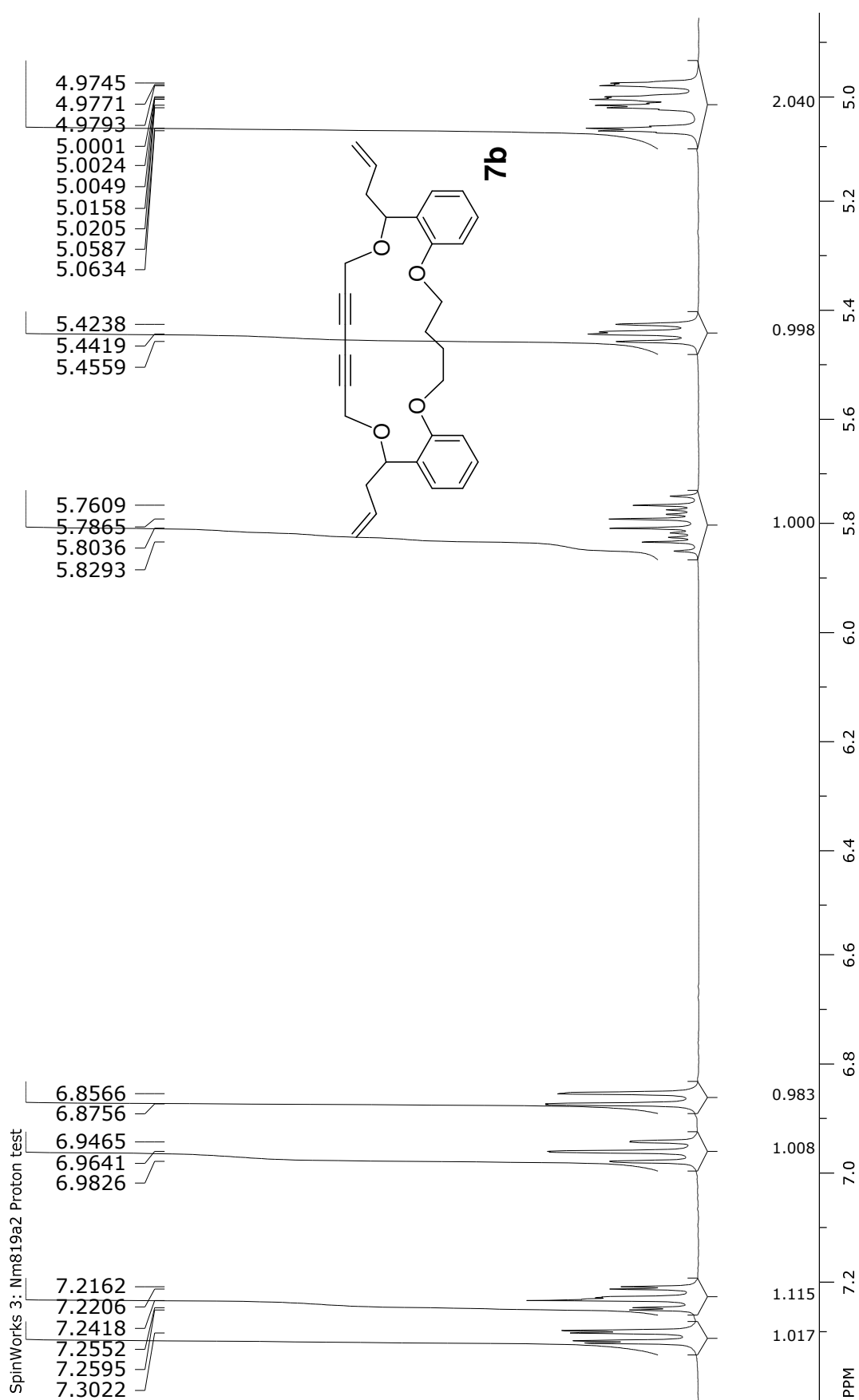
67.4912
68.1381

70.3830
70.9829
71.9305
72.1283

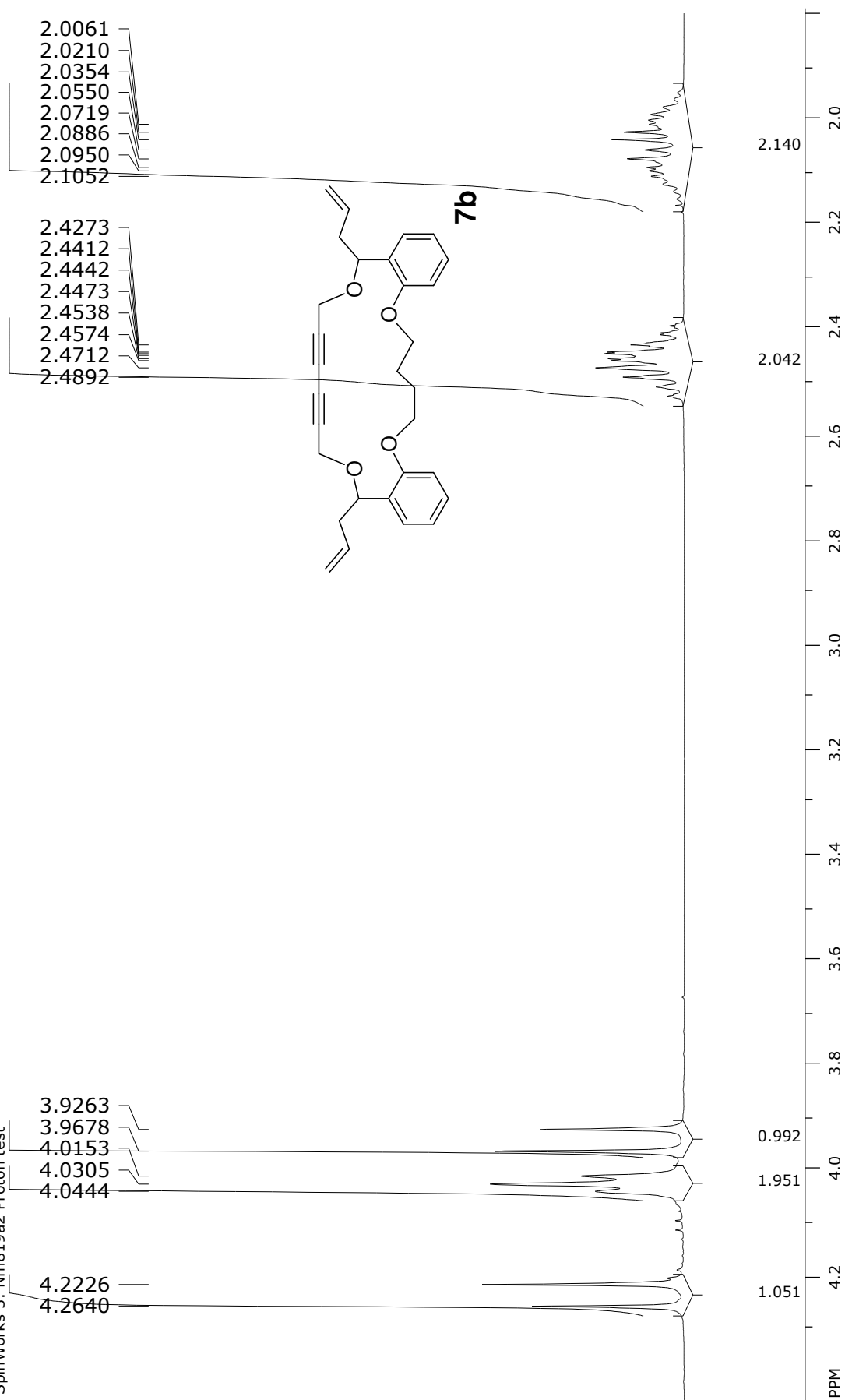
75.3713
75.5142
76.7573
77.0747
77.3931

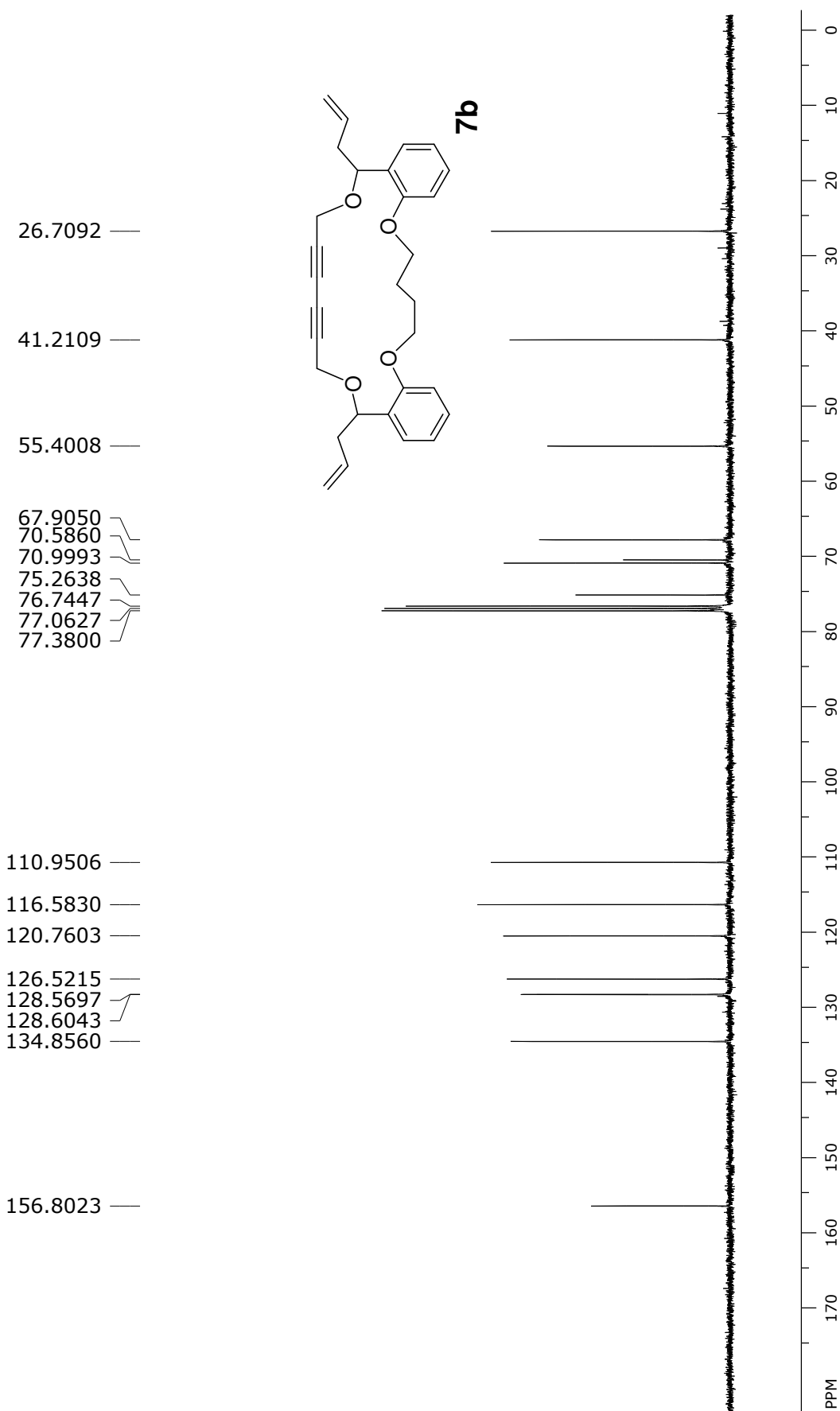






SpinWorks 3: Nm819a2 Proton test





SpinWorks 3: Nm819a2 Carbon test

26.7092 —

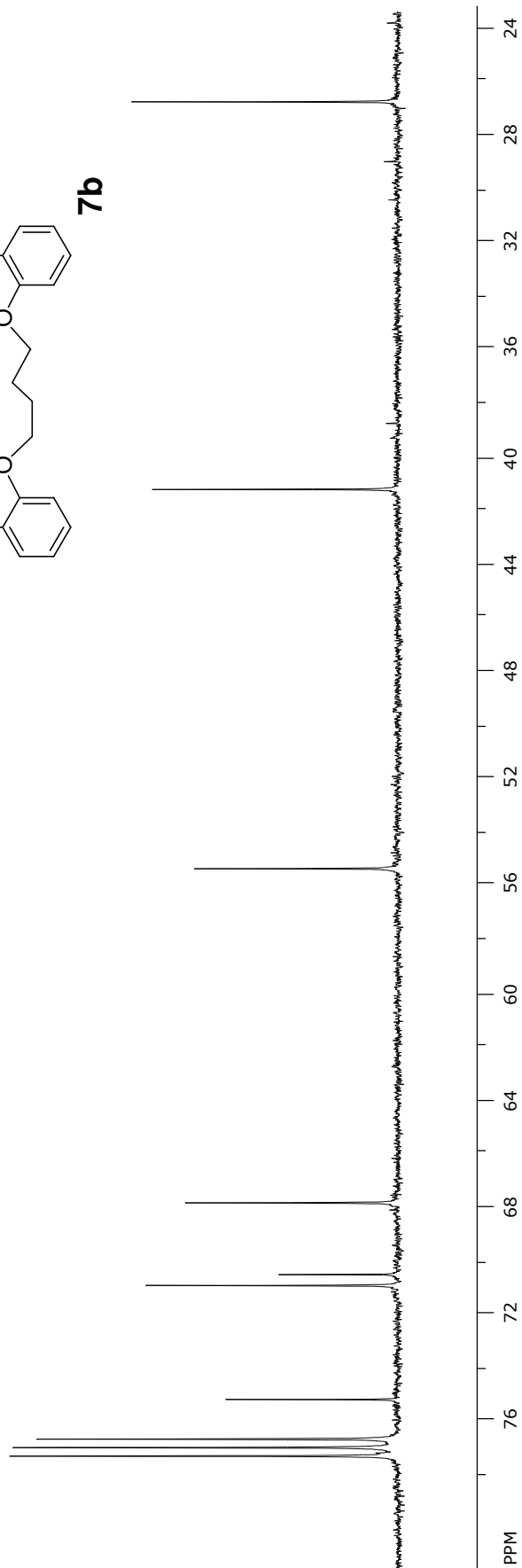
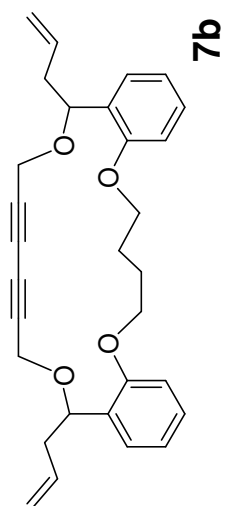
41.2109 —

55.4008 —

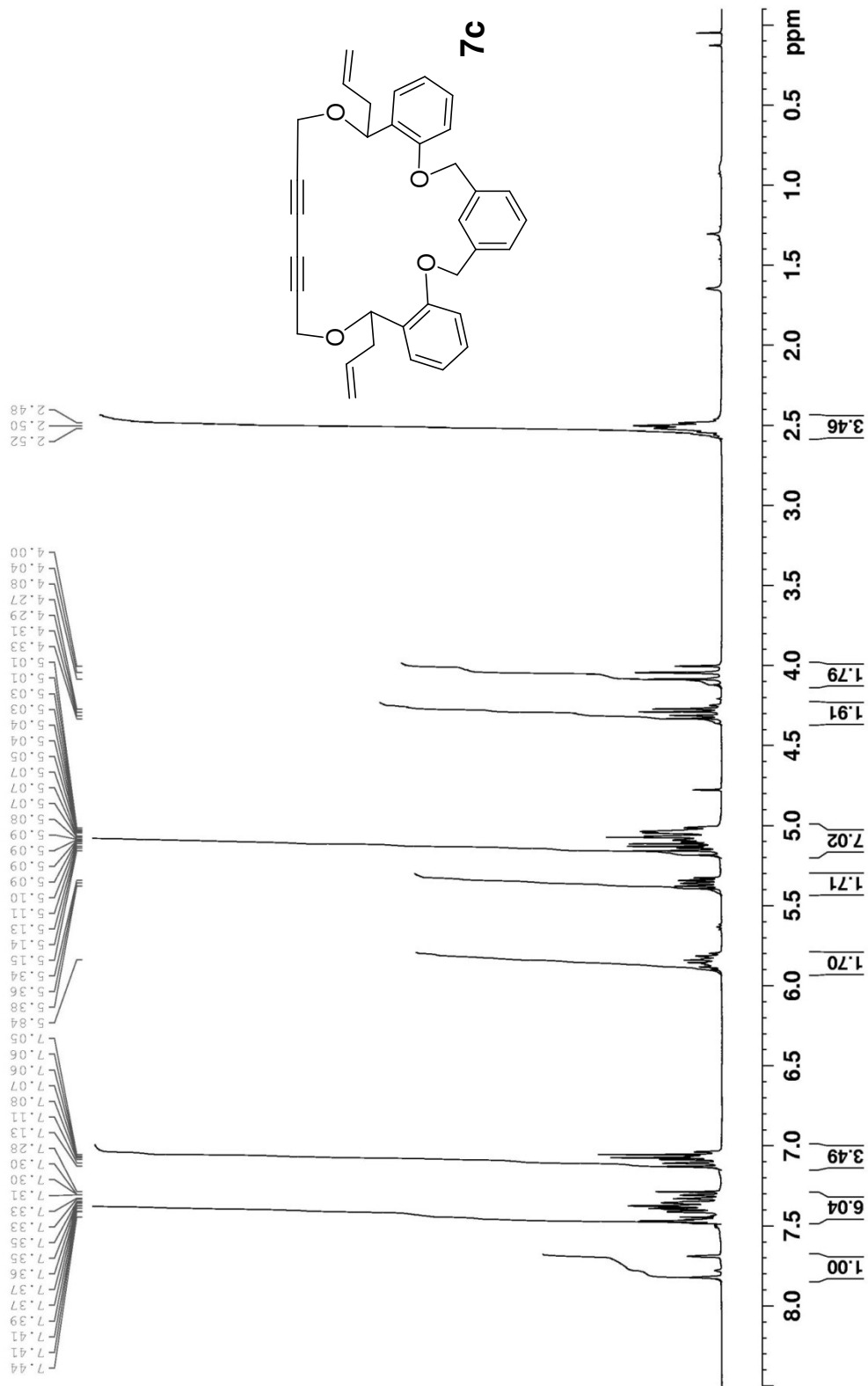
67.9050 —

70.5860 —
70.9993 —

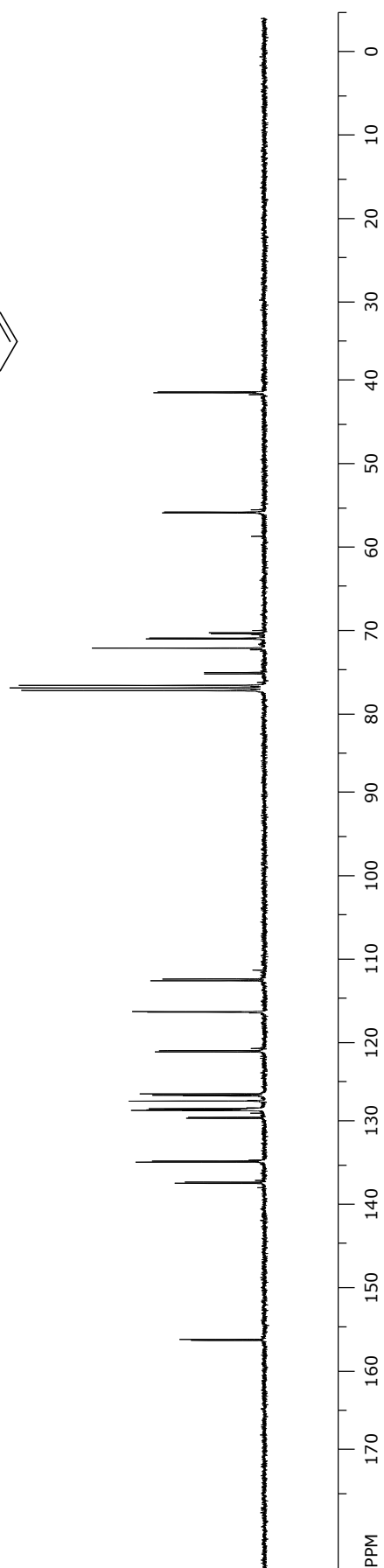
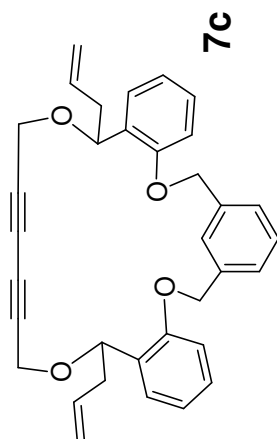
75.2638 —
76.7447 —
77.0627 —
77.3800 —



nm1185a2
 PROTON CDCl3 /opt/topspin nmrsu 57



112.6191
112.8346
116.6054
116.6663
121.3634
121.5146
126.6377
126.8078
126.8847
127.5124
128.4401
128.5211
128.5897
128.6490
128.6855
129.5019
129.6256
134.8438
134.9628
137.4157
137.5608
156.6126
156.7304



156.6126 —
156.7304 —

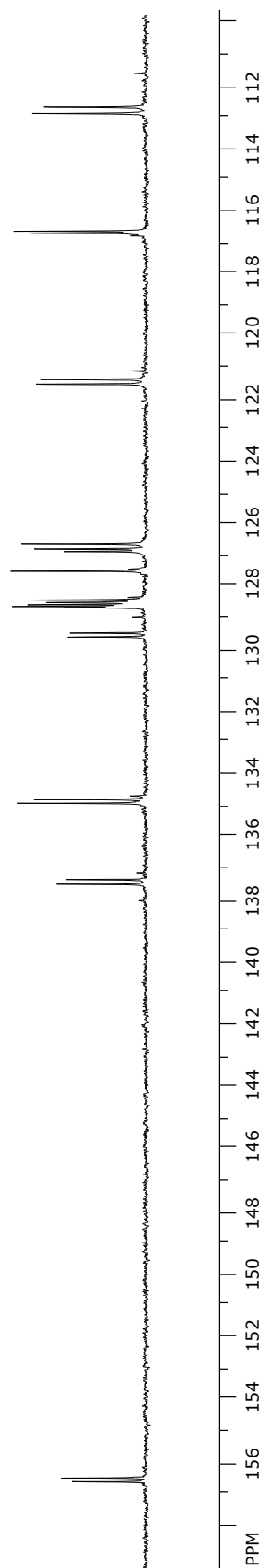
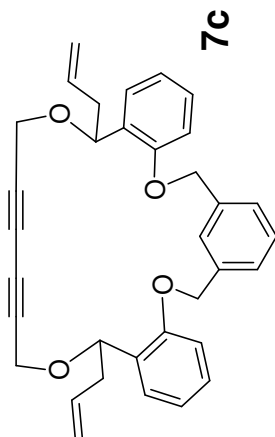
116.6054
116.6663

121.3634 —
121.5146 —

126.6377
126.8078
126.8847
127.5124
128.4401
128.5211
128.5897
128.6490
128.6855
129.5019
129.6256

134.8438
134.9628

137.4157 —
137.5608 —



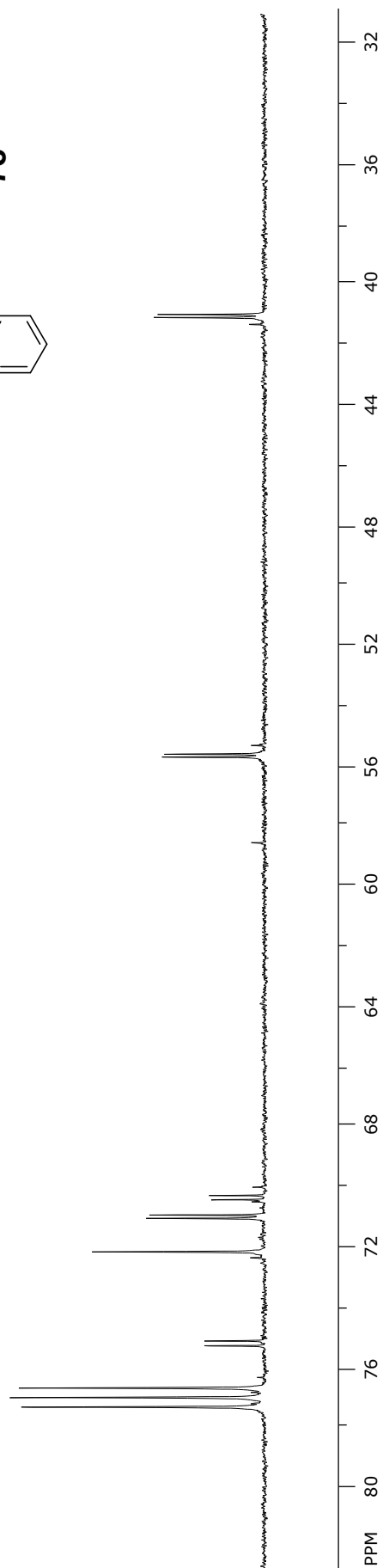
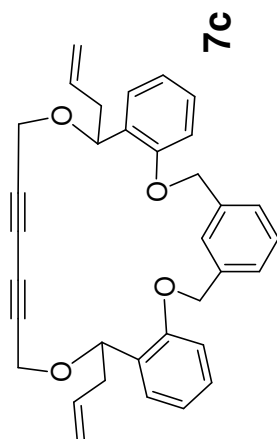
SpinWorks 3: nm1185a2

40.9566
41.0618

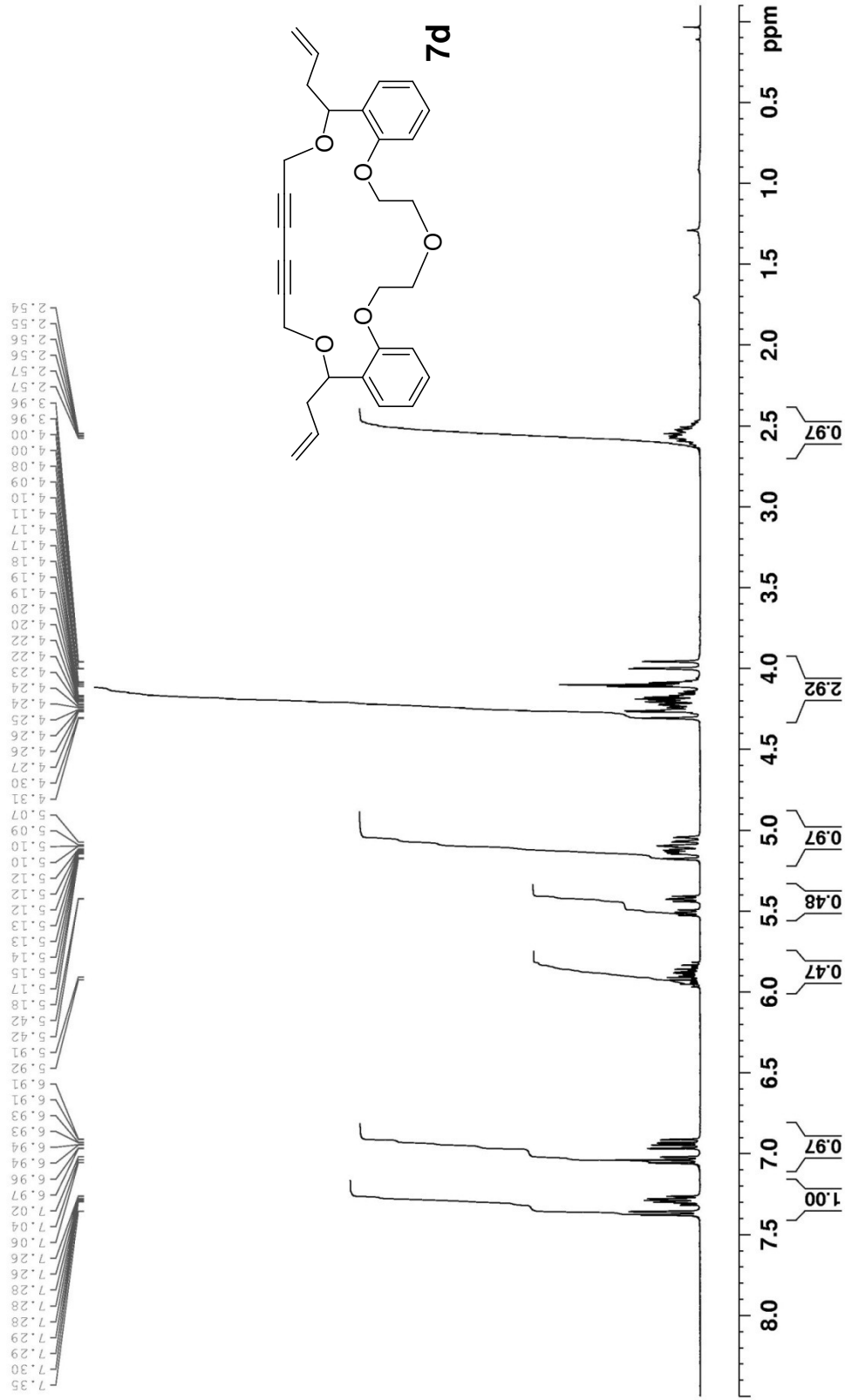
55.6261
55.7243

70.3497
70.4923
71.0044
71.1146
72.2323

75.2037
75.3681
76.7741
77.0920
77.4095



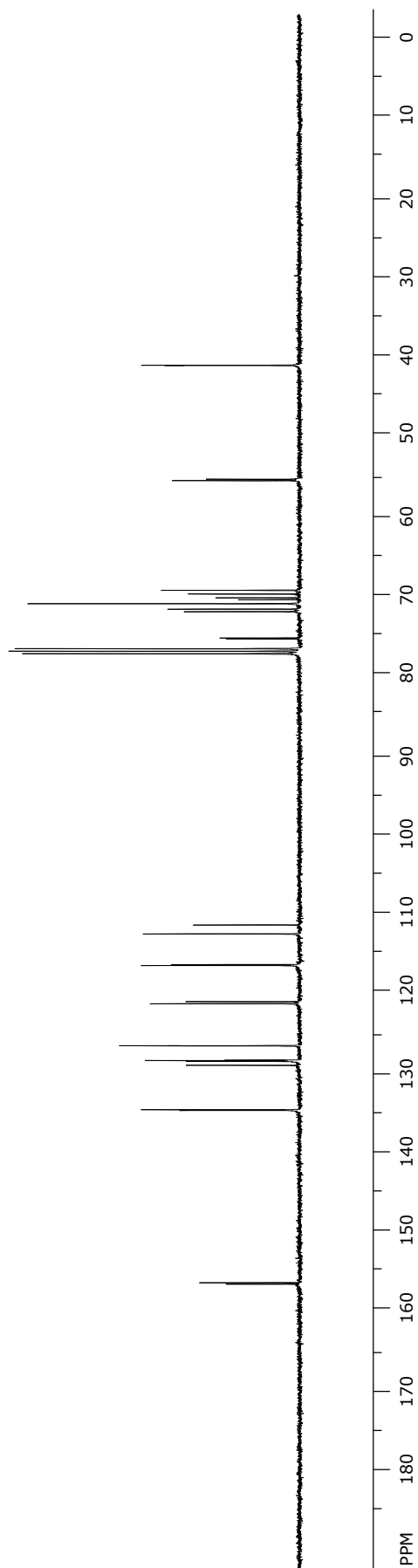
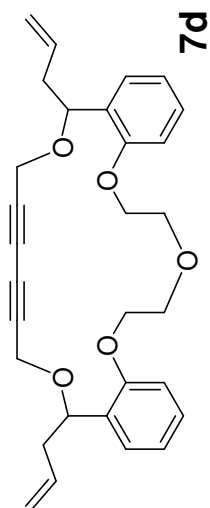
nm1186ab
PROTON CDCl3 /opt/topspin nmrsu 48



41.0236
41.0438
55.3925
55.5628
69.3960
69.8683
70.3488
70.5885
71.0858
71.7776
72.0917
75.4137
75.5345
76.7664
77.0847
77.4018

111.6071
112.7323
116.6053
116.7078
121.2731
121.5334
126.8225
126.8415
128.6463
128.7090
128.7987
129.2999
134.9065
134.9841

156.7204
156.9009



SpinWorks 3: nm1186ab

156.7204
156.9009

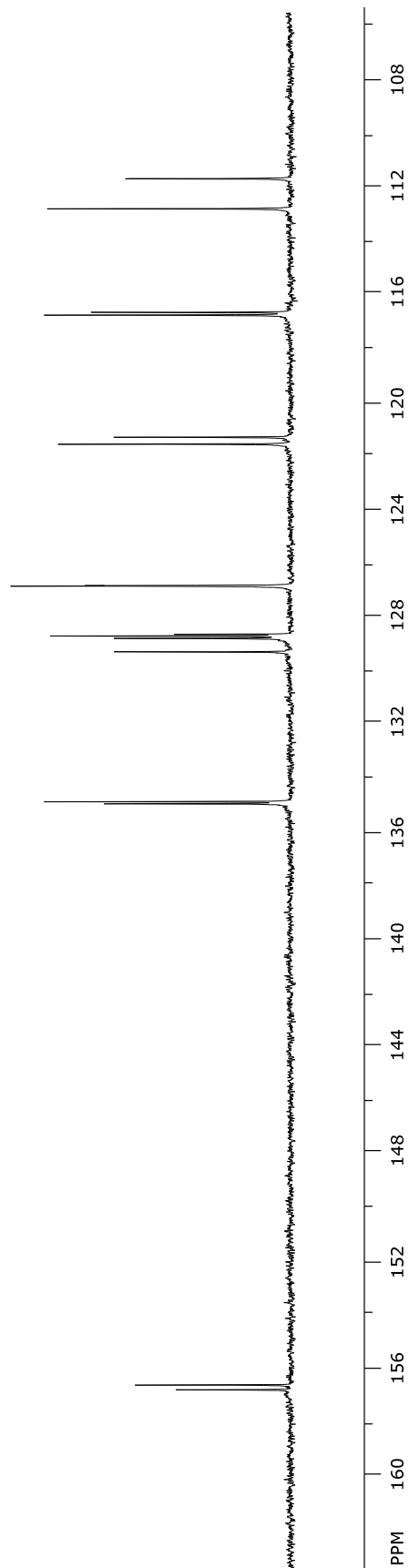
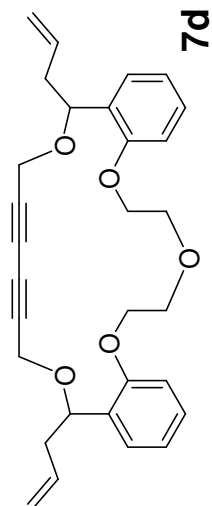
134.9065
134.9841

126.8225
126.8415
128.6463
128.7090
128.7987
129.2999

121.2731
121.5334

116.6053
116.7078

111.6071
112.7323



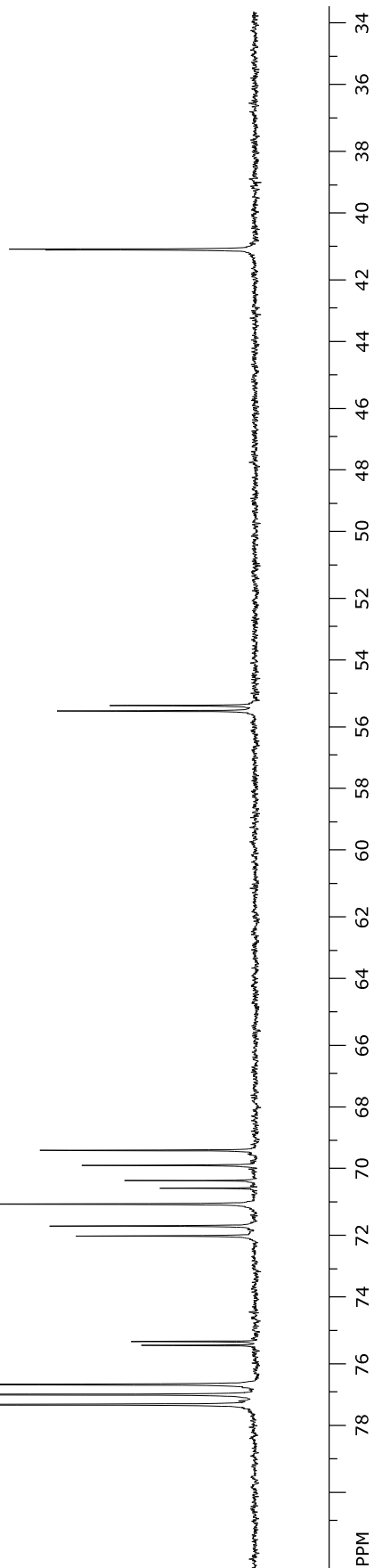
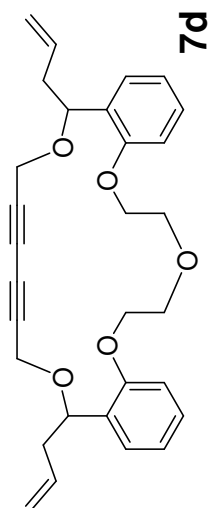
SpinWorks 3: nm1186ab

41.0236
41.0438

55.3925
55.5628

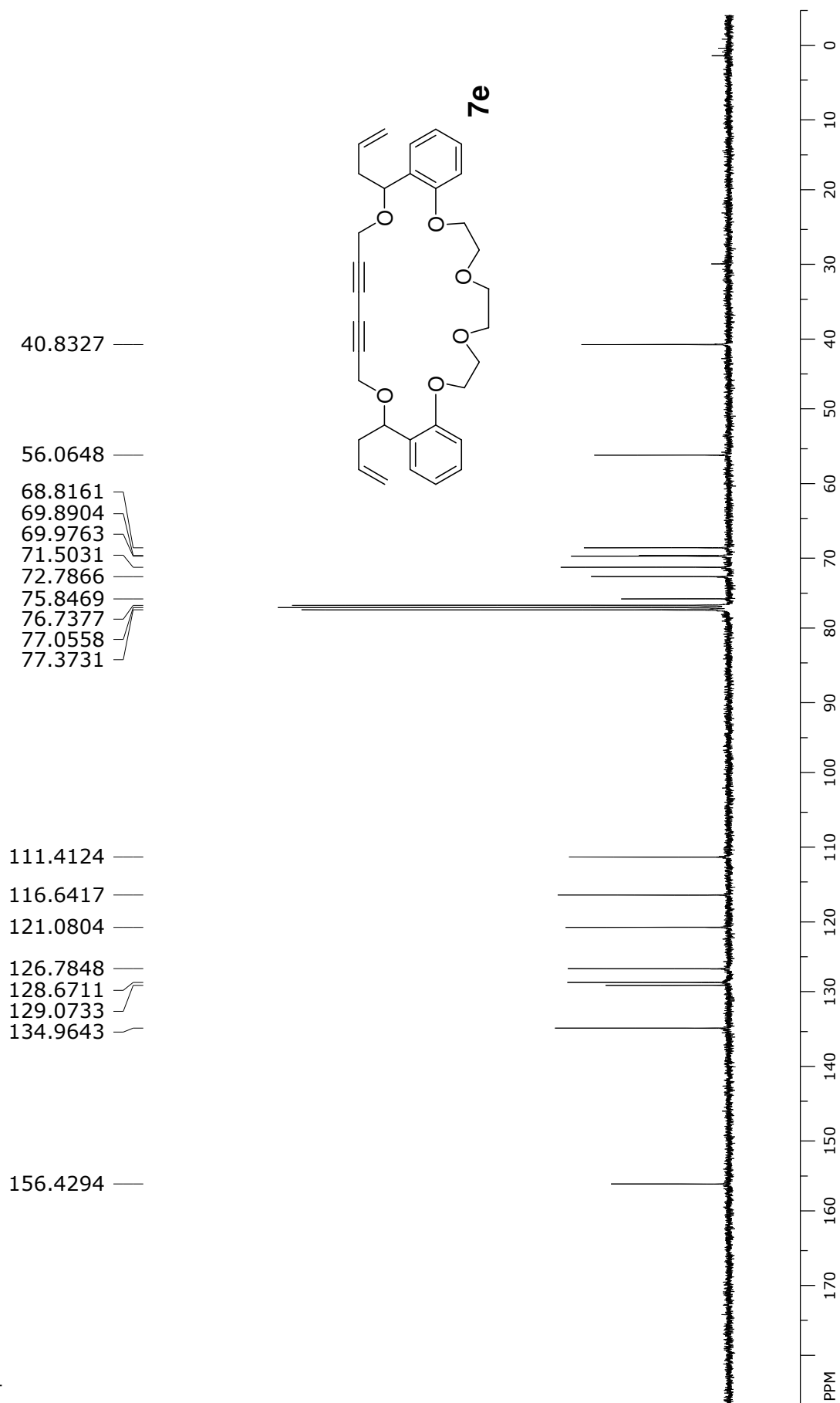
69.3960
69.8683
70.3488
70.5885
71.0858
71.7776
72.0917

75.4137
75.5345
76.7664
77.0847
77.4018



PROTON CDC13 /opt/topspin nmrsu 6





SpinWorks 3: nm1187a1

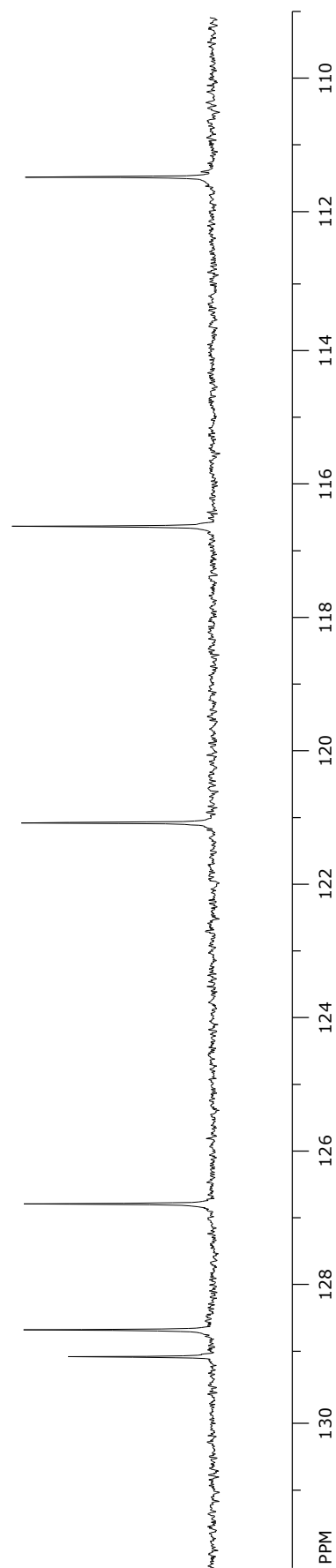
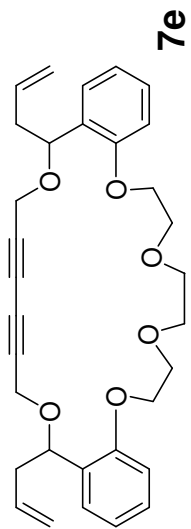
128.6711 —
129.0733 —

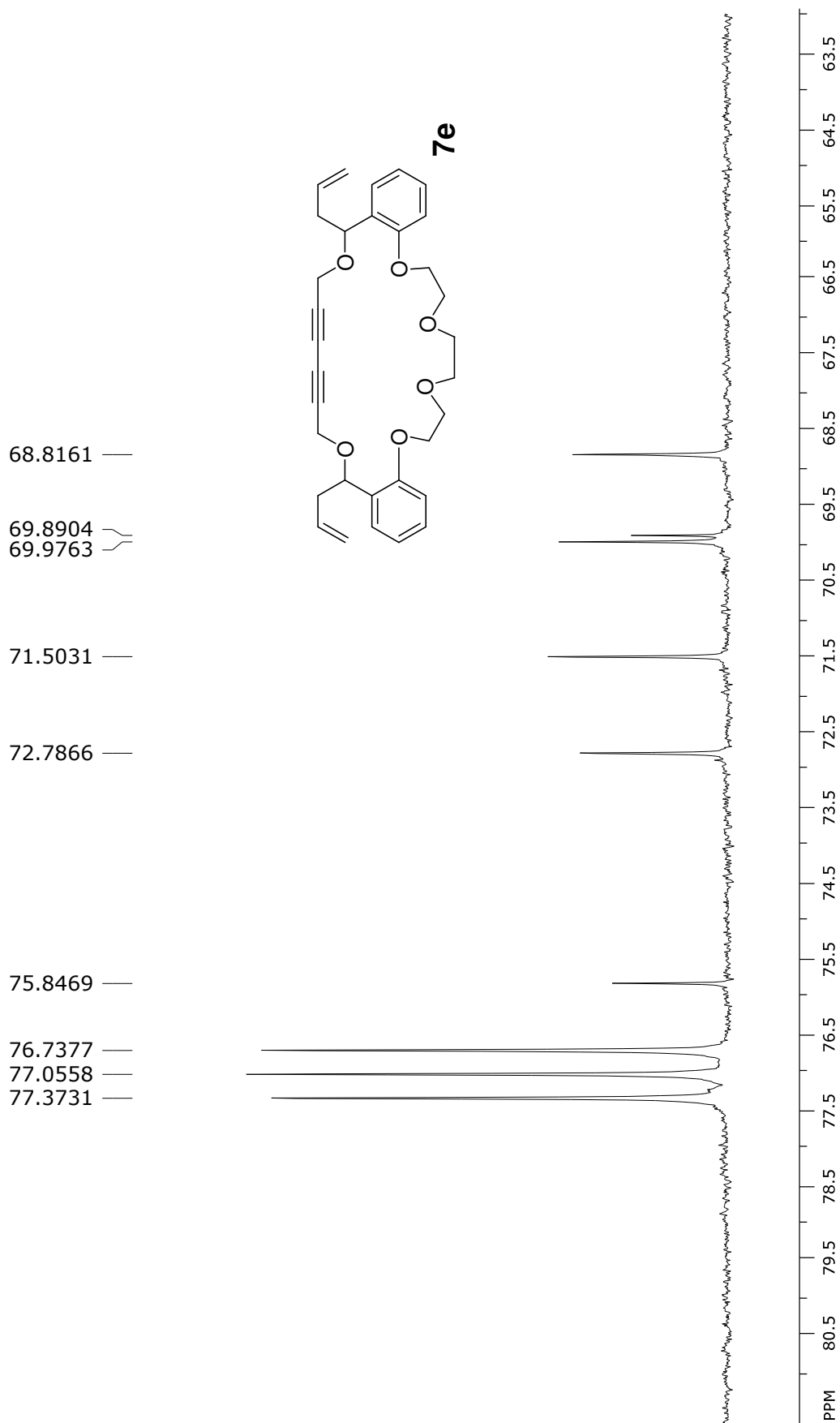
126.7848 —

121.0804 —

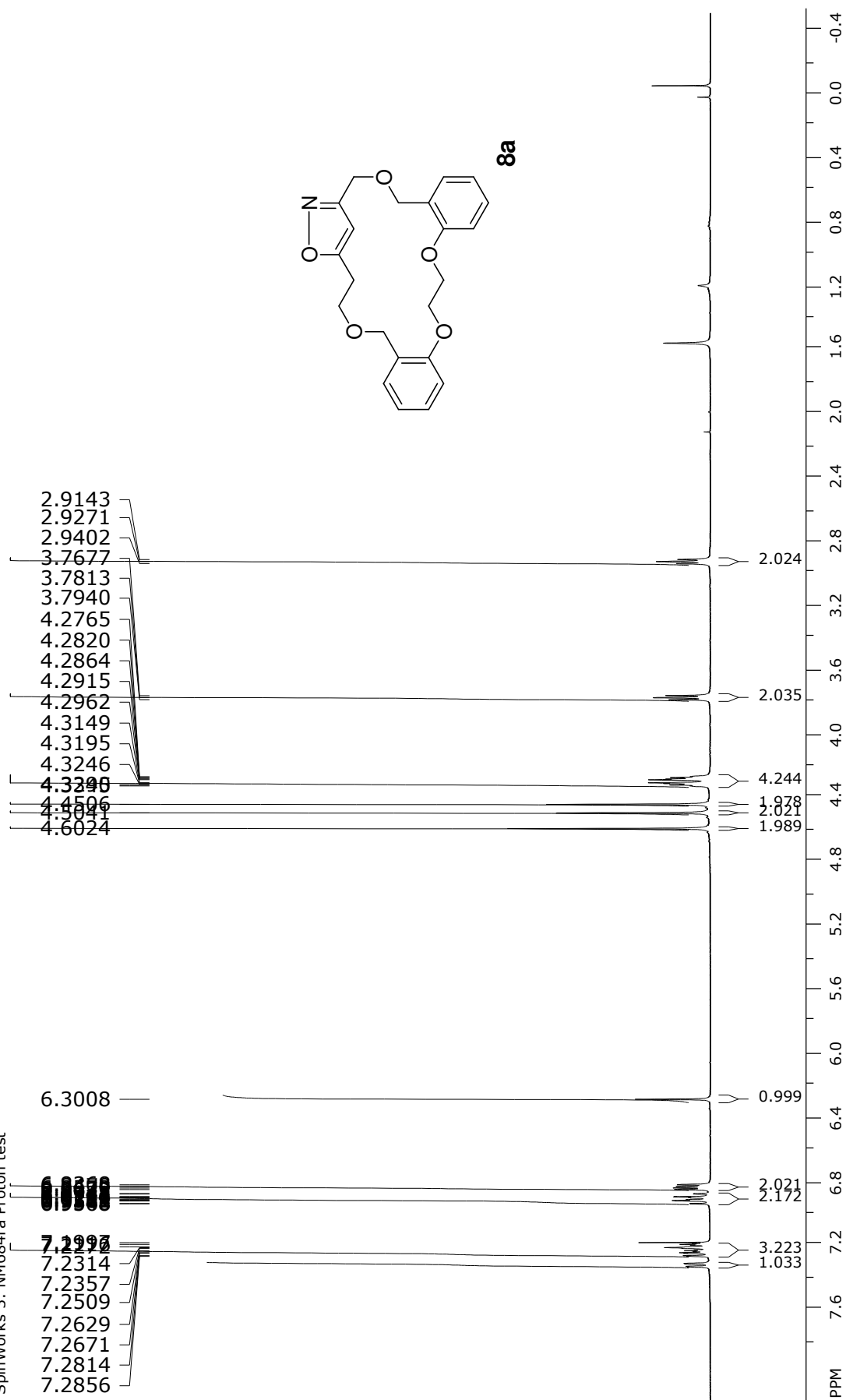
116.6417 —

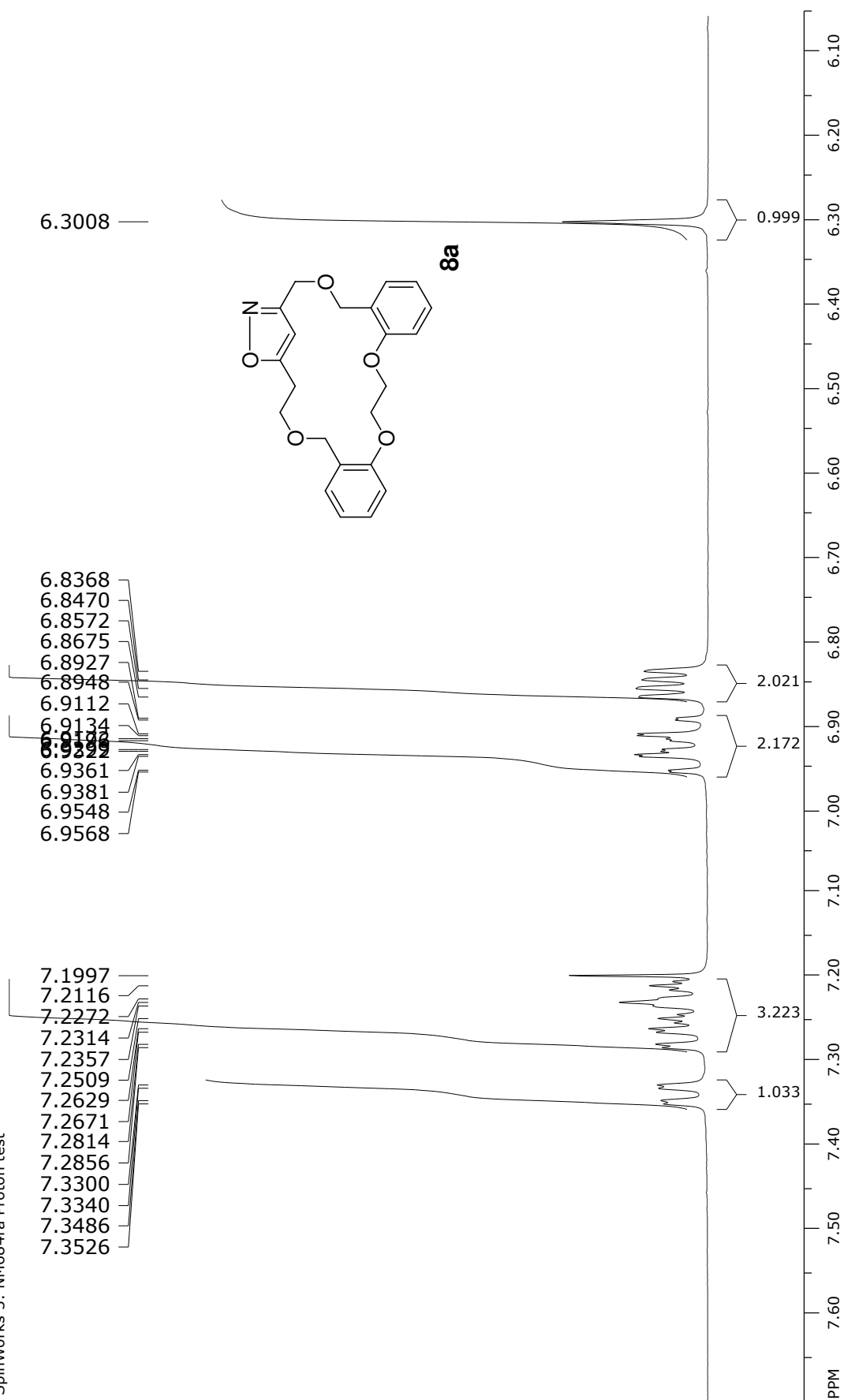
111.4124 —

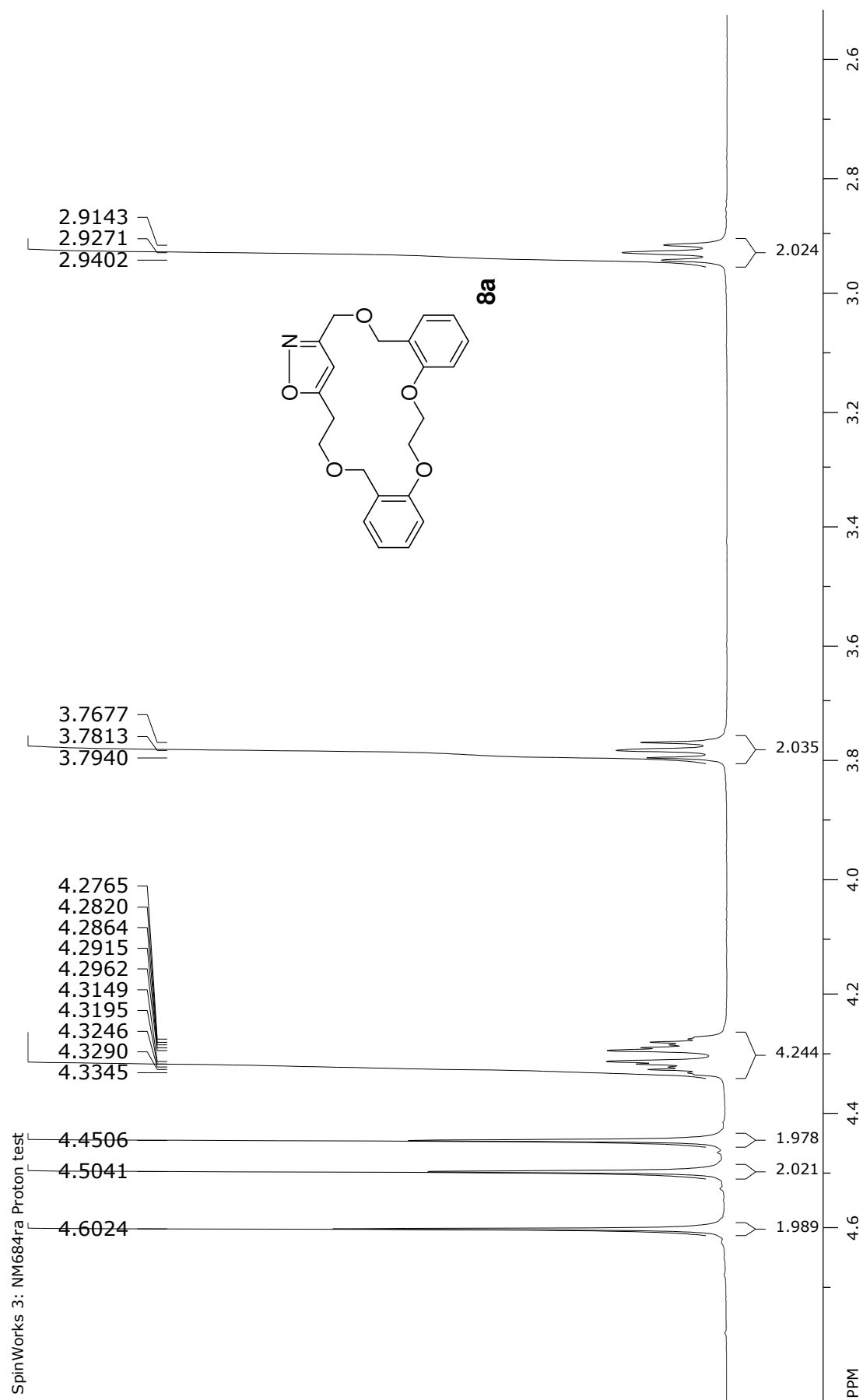


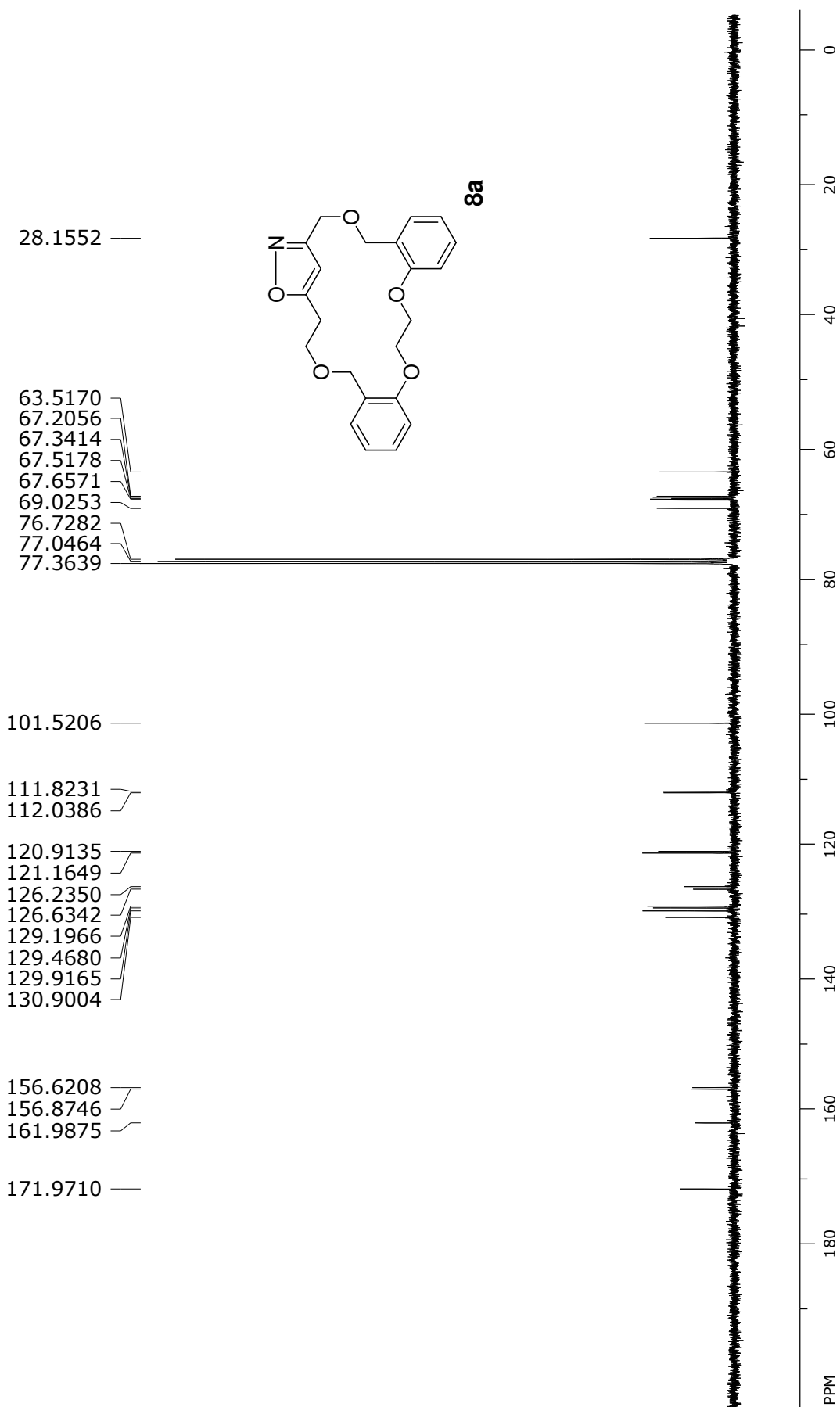


SpinWorks 3: NM684ra Proton test









SpinWorks 3: NM684ra Carbon test

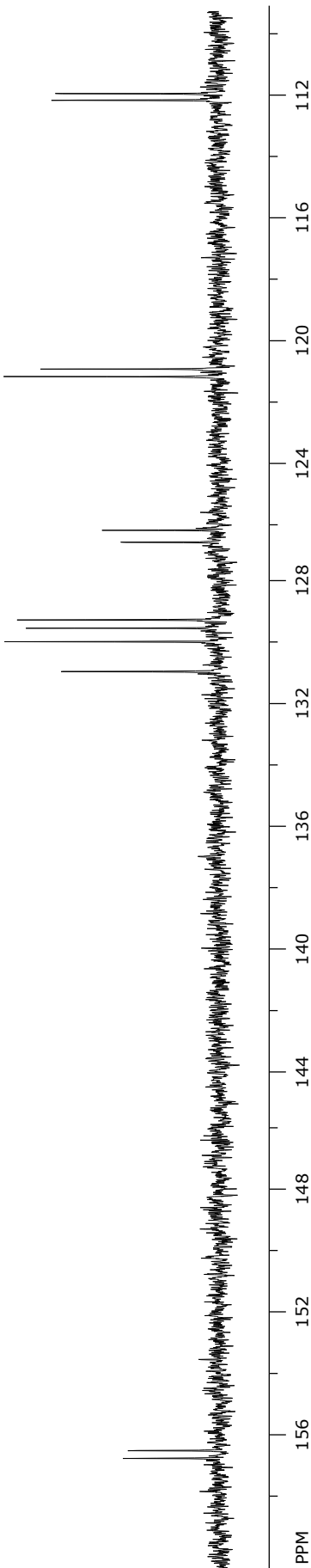
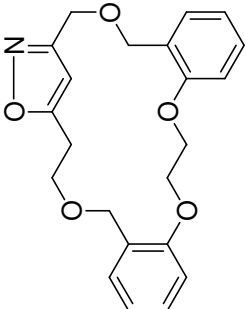
156.6195 
156.8739

129.1964 —
129.4678 —
129.9164 —
130.8998 —

126.2349 —
126.6337 —

120.9135 —
121.1648 —

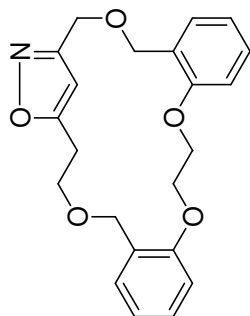
111.8234 —
112.0388 —



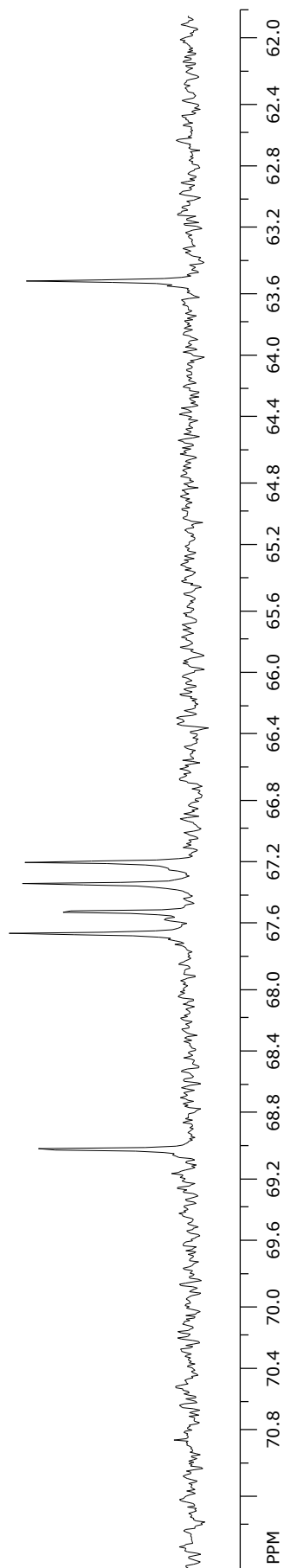
63.5180 —

67.2066 —
67.3422 —
67.5198 —
67.6580 —

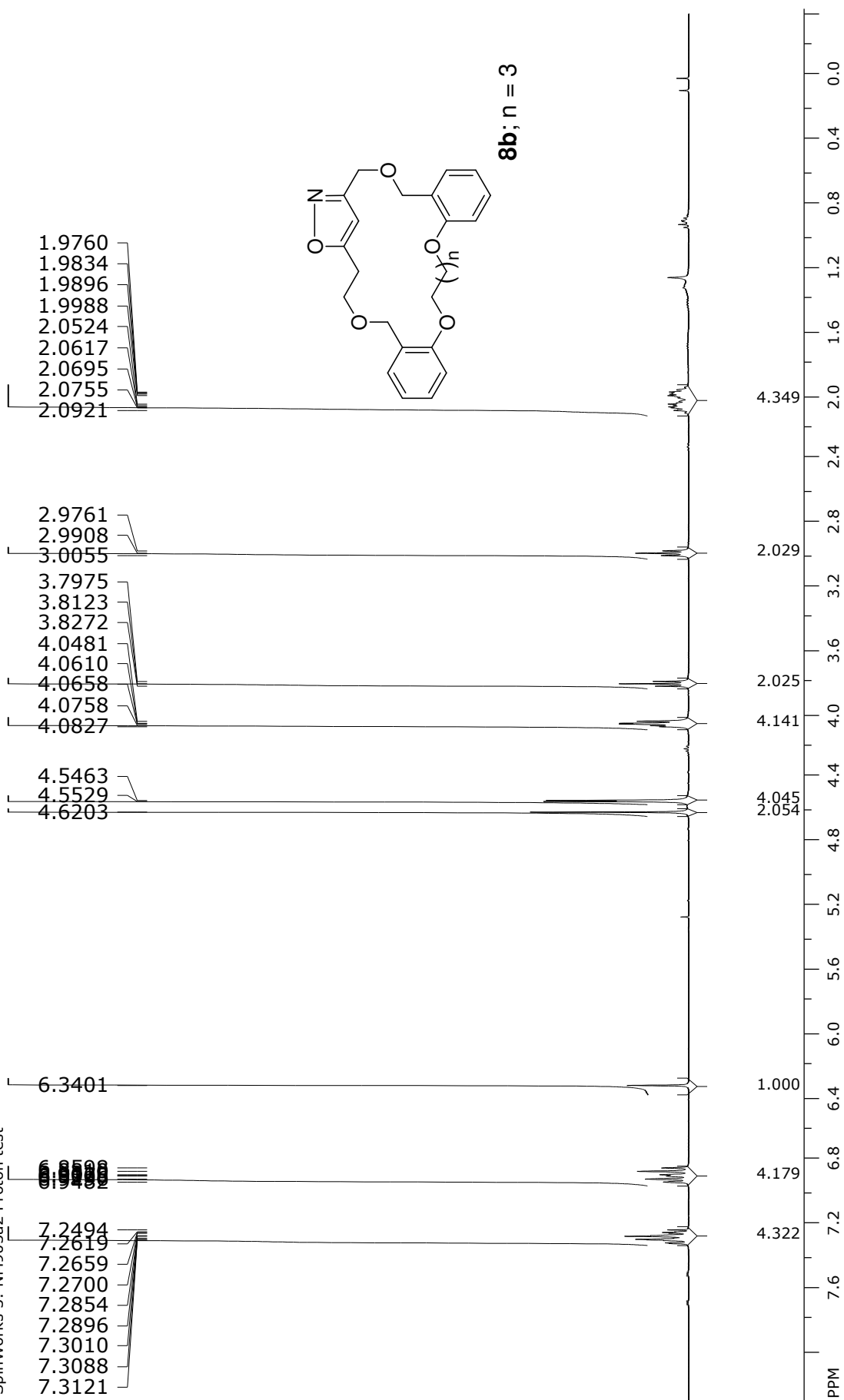
69.0267 —

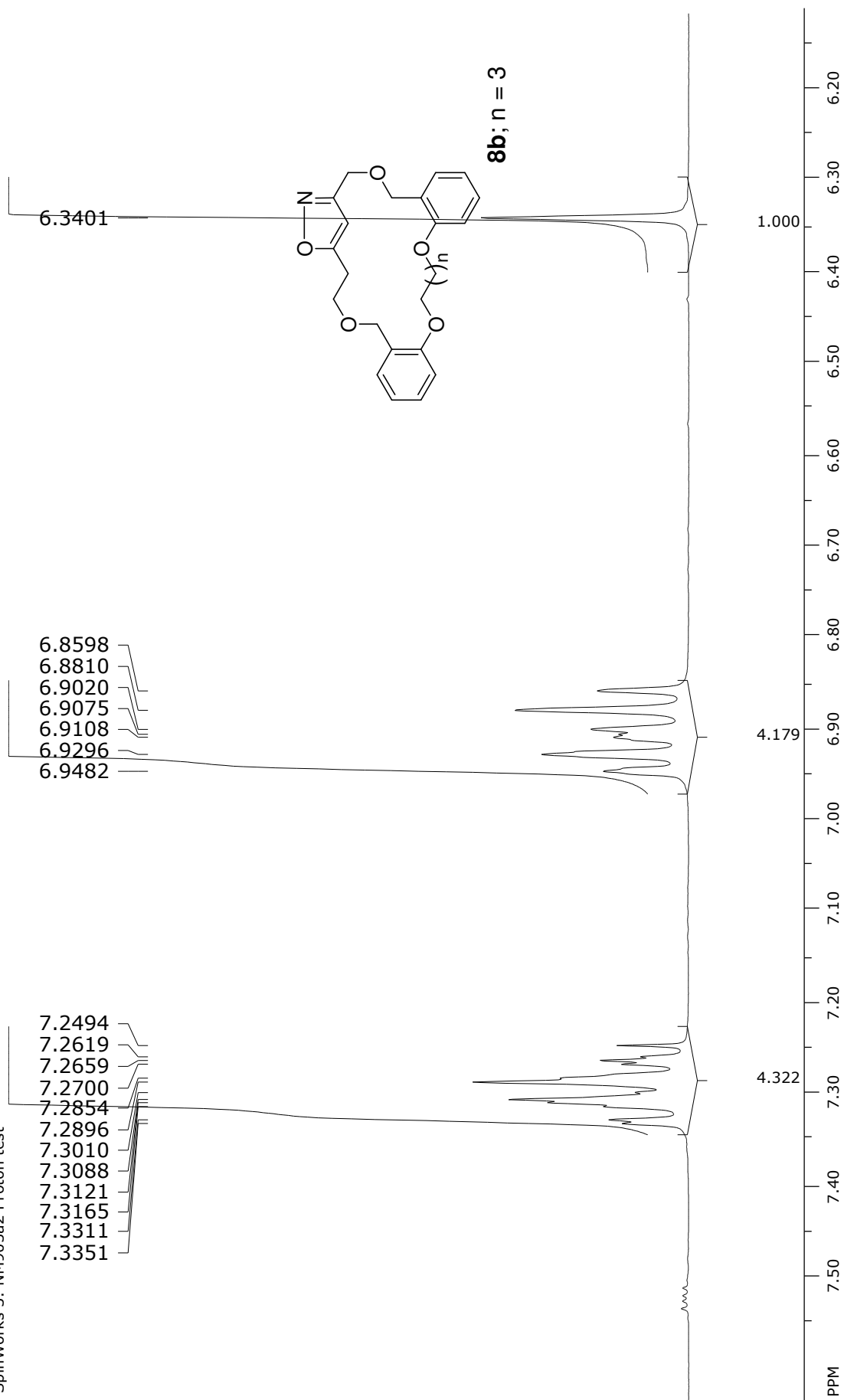


8a

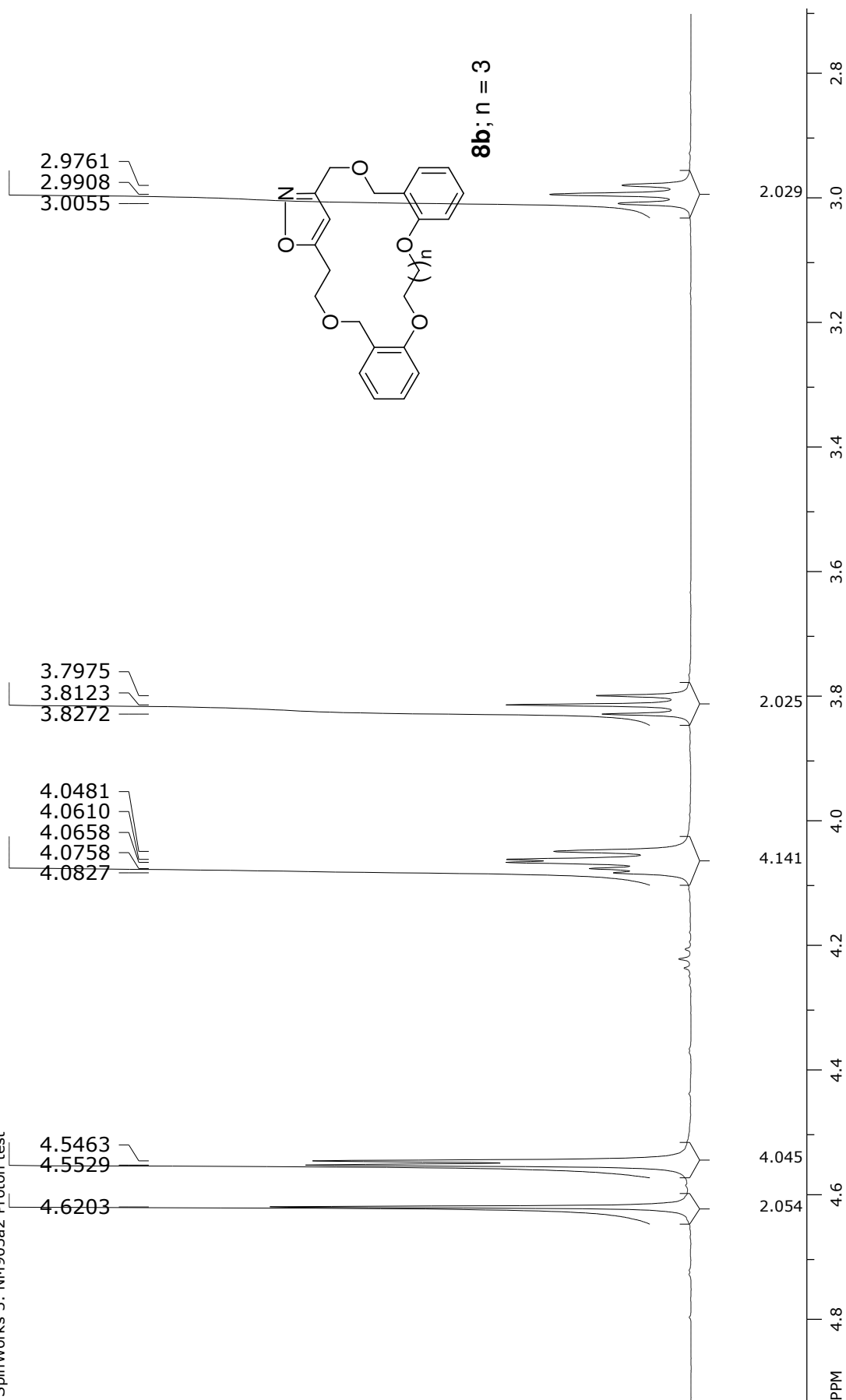


SpinWorks 3: NM903a2 Proton test





SpinWorks 3: NM903a2 Proton test



SpinWorks 3: NM-903A2 in cdcl3 carbon test

26.1315
26.4395
27.7853

64.0711
67.1503
67.4573
67.9133
68.8834
68.9310

76.7853
77.1031
77.4205

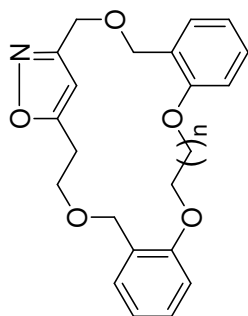
101.1379

111.1109
111.4474

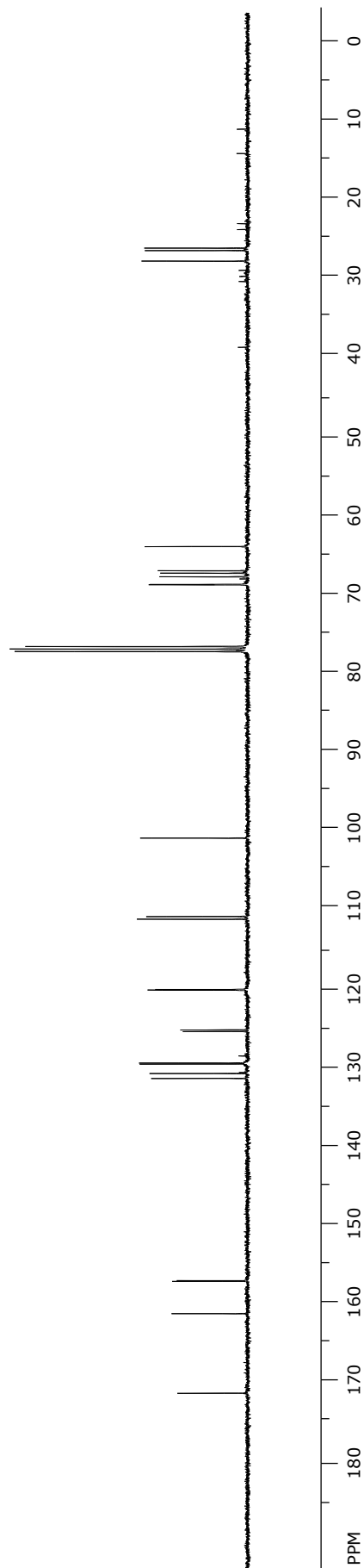
120.3865
120.4636
125.5233
125.7230
129.7354
129.8607
131.0637
131.6915

157.4036
157.4832
161.5983

171.7142



8b; $n = 3$



SpinW 157.4036

157.4036
157.4832

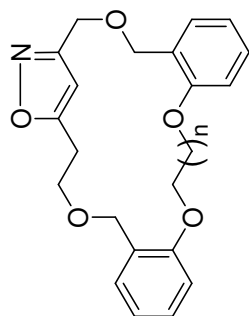
129.7354
129.8607
131.0637
131.6915

125.5233 
125.7230 

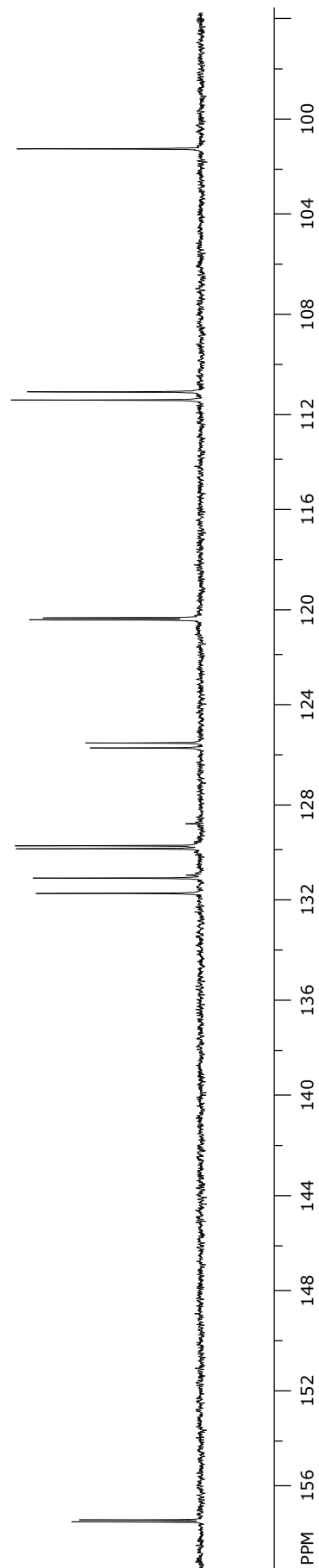
120.3865
120.4636

111.1109 —
111.4474 —

101.1379 —

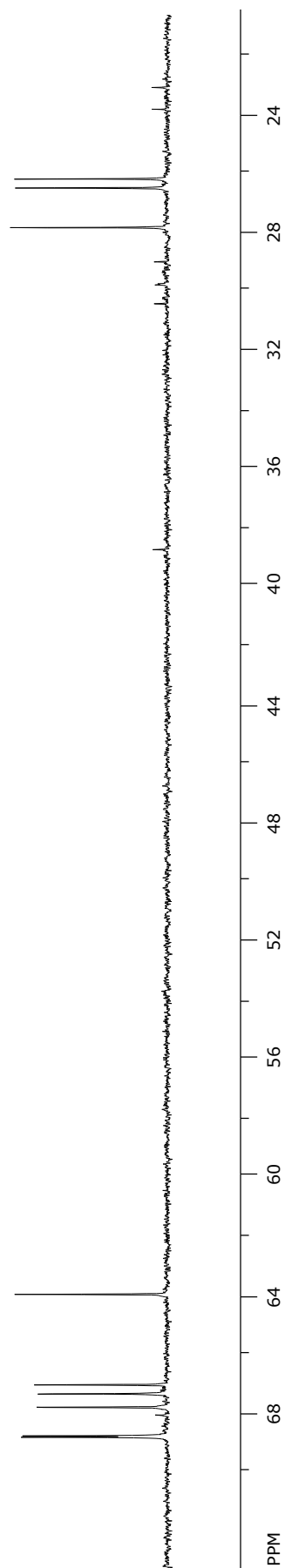
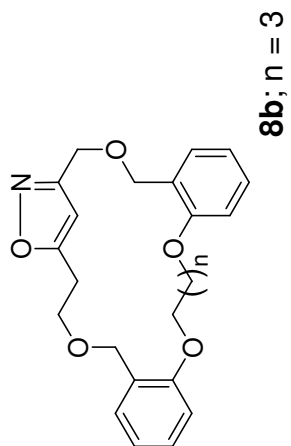


8b; $n = 3$



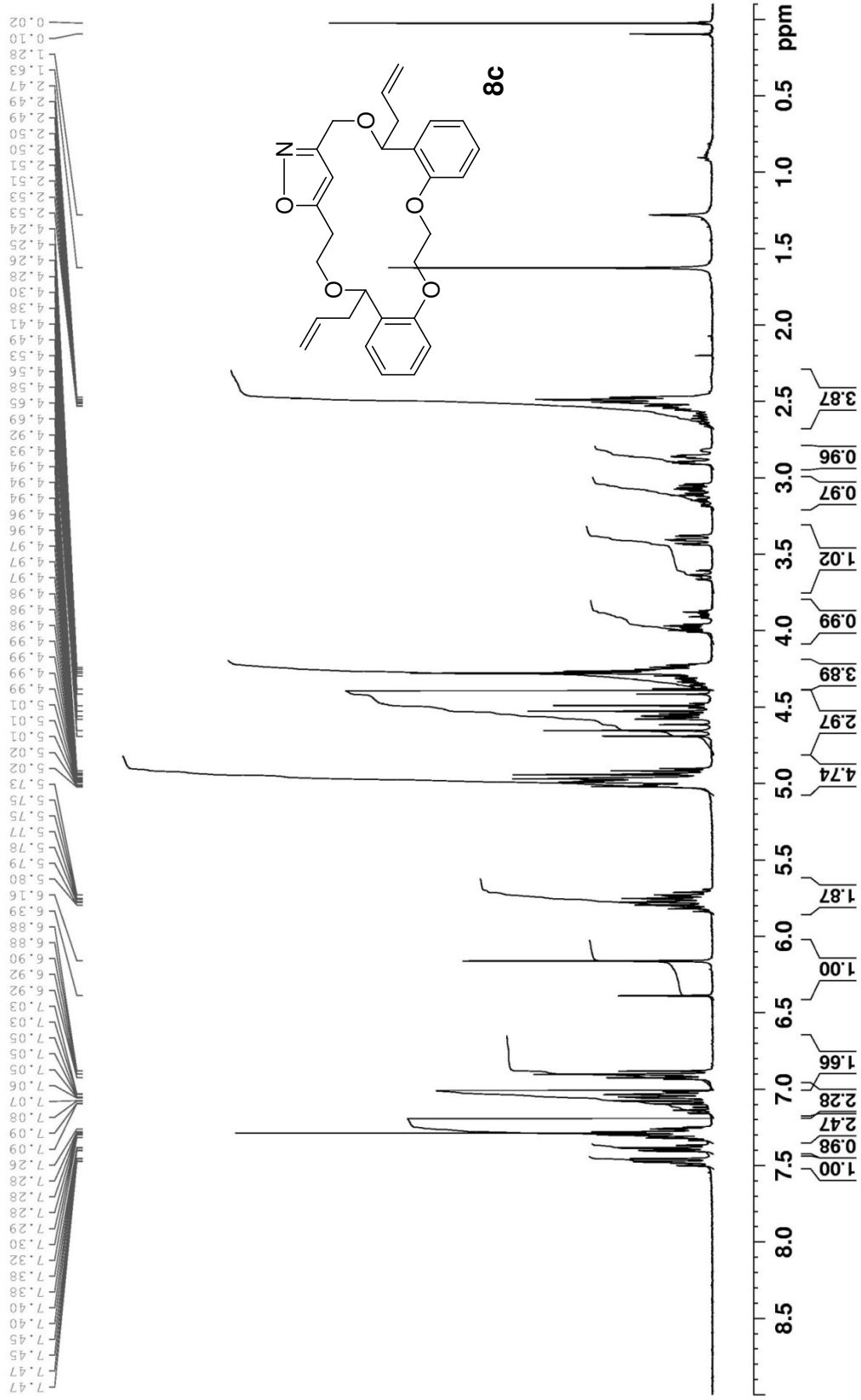
64.0711 —
67.1503 —
67.4573 —
67.9133 —
68.8834 —
68.9310 —

26.1315 —
26.4395 —
27.7853 —



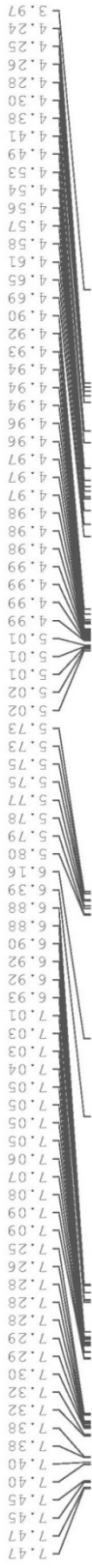
nm1147ra1

PROTON CDC13 /opt/topspin nmrsu 46



nm1147ra1

PROTON CDC13 /opt/topspin nmrsu 46



28.9911
29.4061
40.3225
40.9462
40.9902
41.5419
60.1516
60.7745
65.7577
66.6166
66.6368
66.6799
67.5635
67.9162
69.3530
71.1232
72.4869
76.7446
77.9608

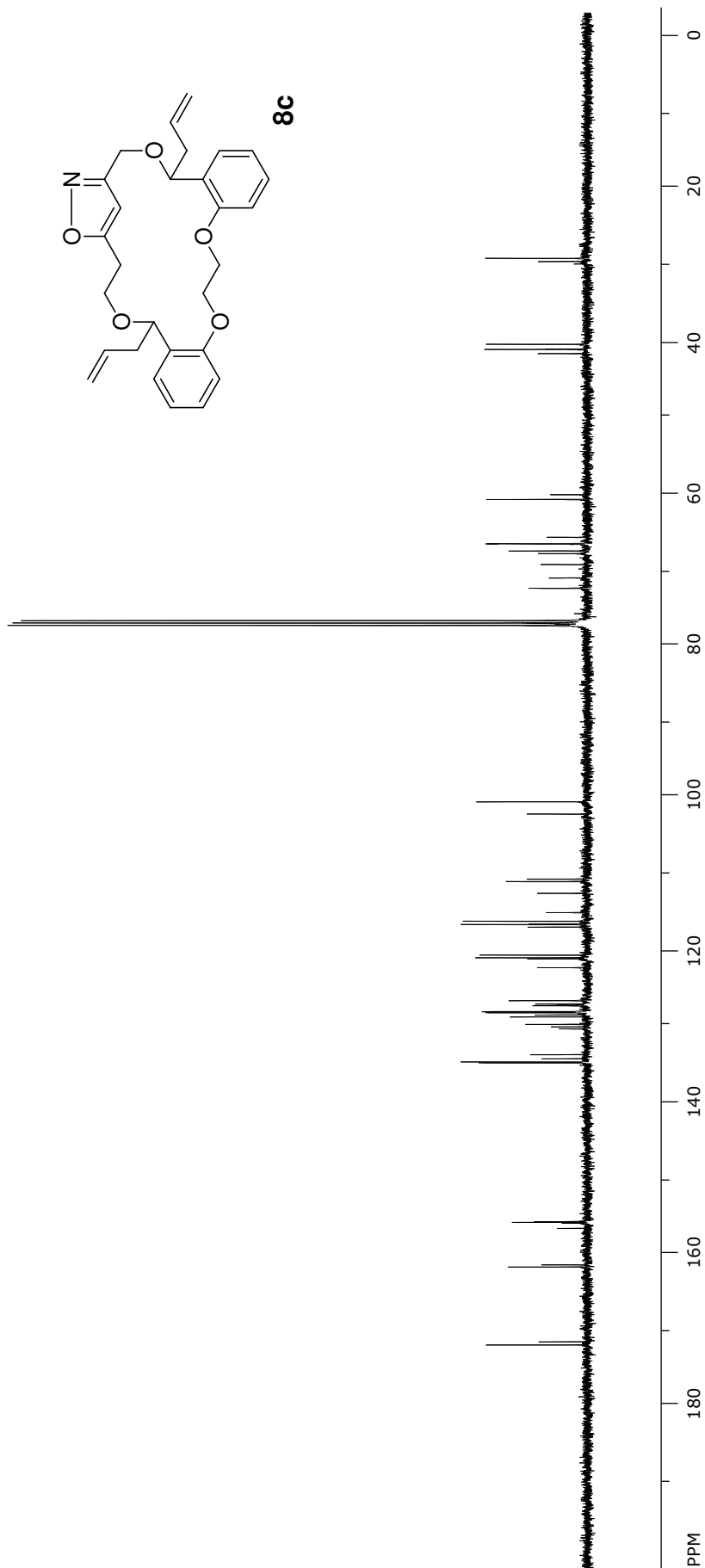
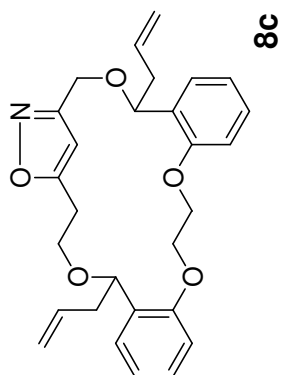
100.6361
102.2554

110.8536
112.7043
110.8164
110.8192

120.8504
122.5109

128.8506
128.8506

128.7497
128.9920
129.9889
130.3176
130.5843
133.9811
134.5224
134.9426
135.0545
155.9818
156.0793
156.1929
156.8859
161.6868
161.9728
171.8446
172.2517

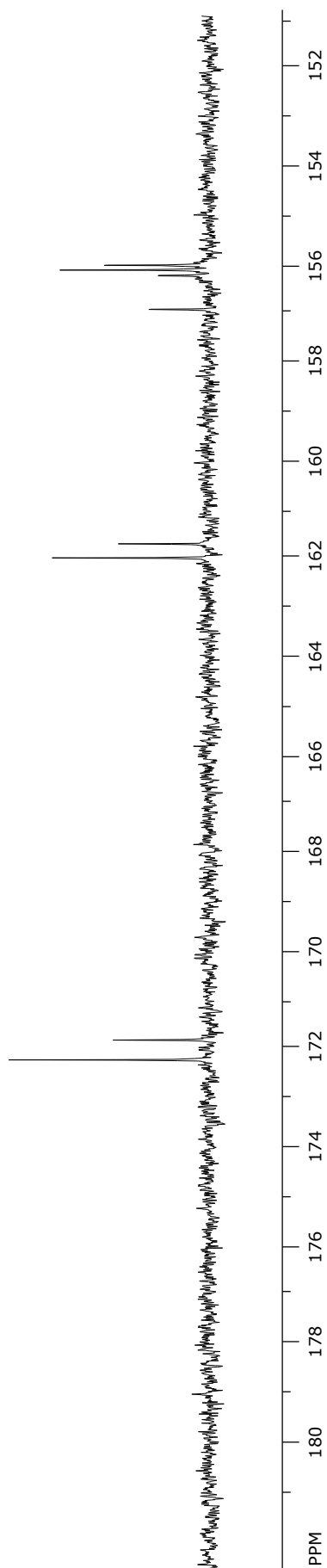
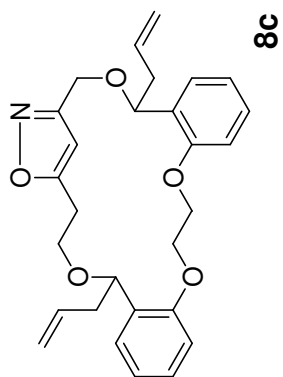


SpinWorks 3: nm-1147-a1

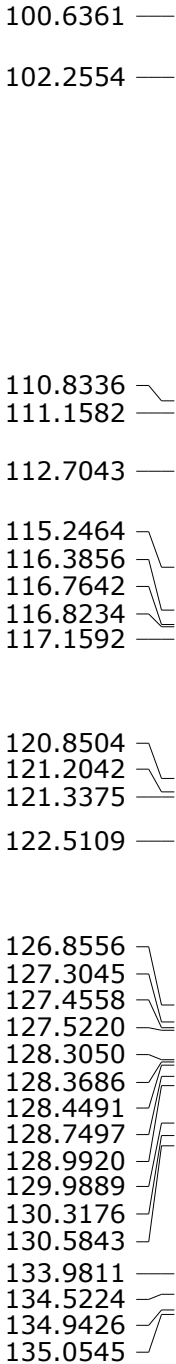
155.9818
156.0793
156.1929
156.8859

161.6868
161.9728

171.8446
172.2517



SpinWorks 3: nm-1147-a1



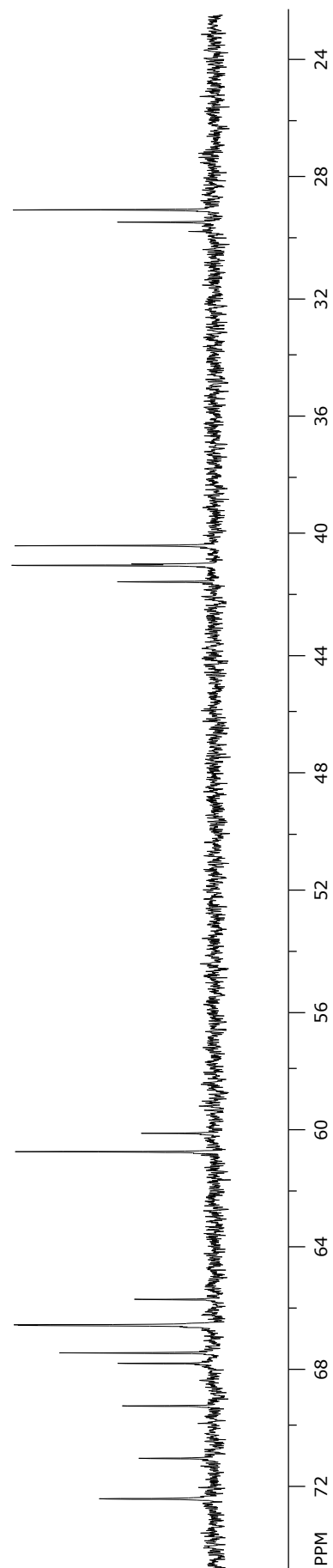
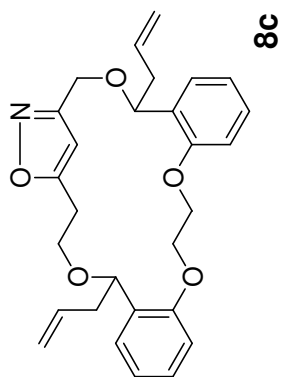
28.9911

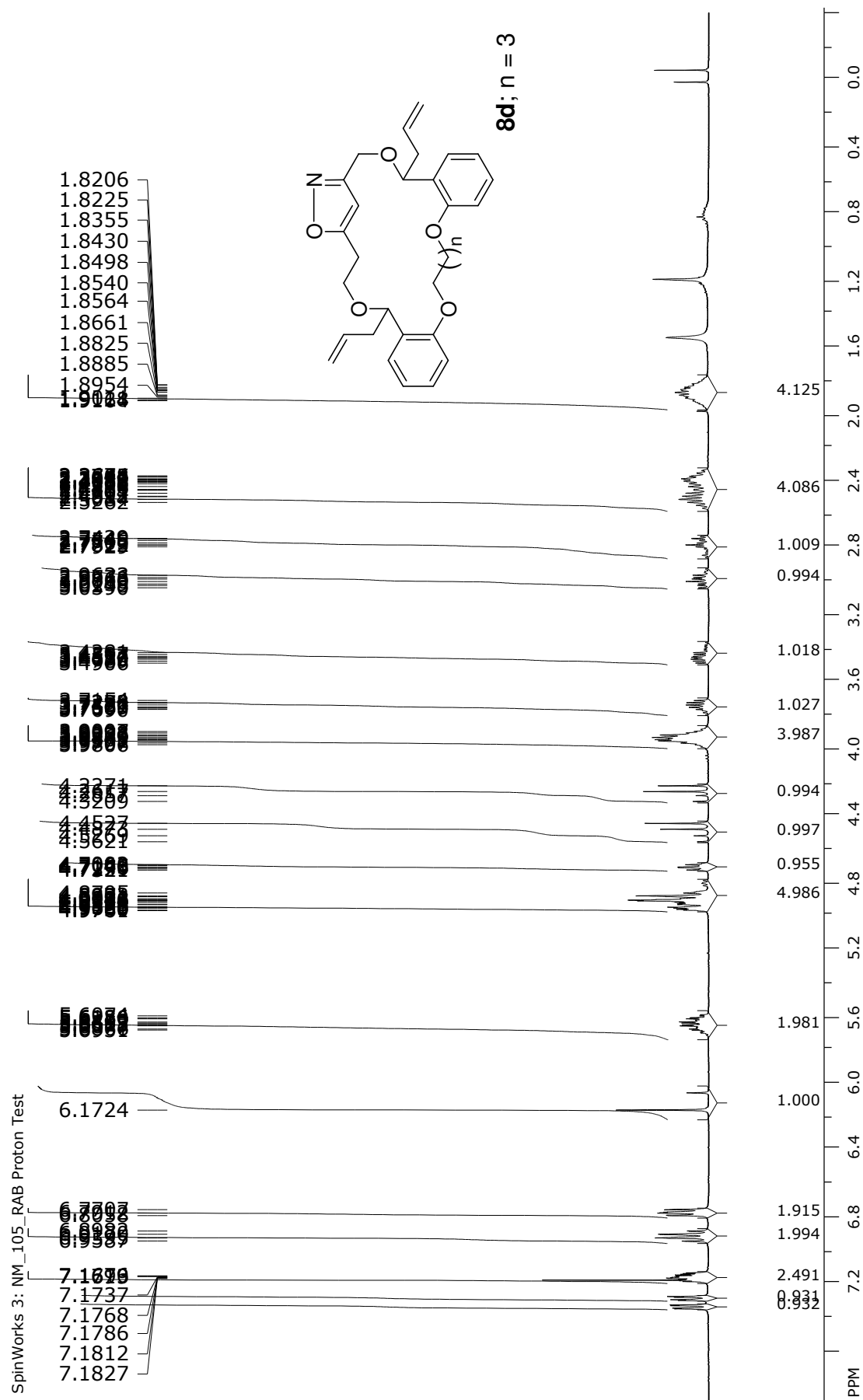
29.4061 

40.3225
40.9462
40.9902
41.5419

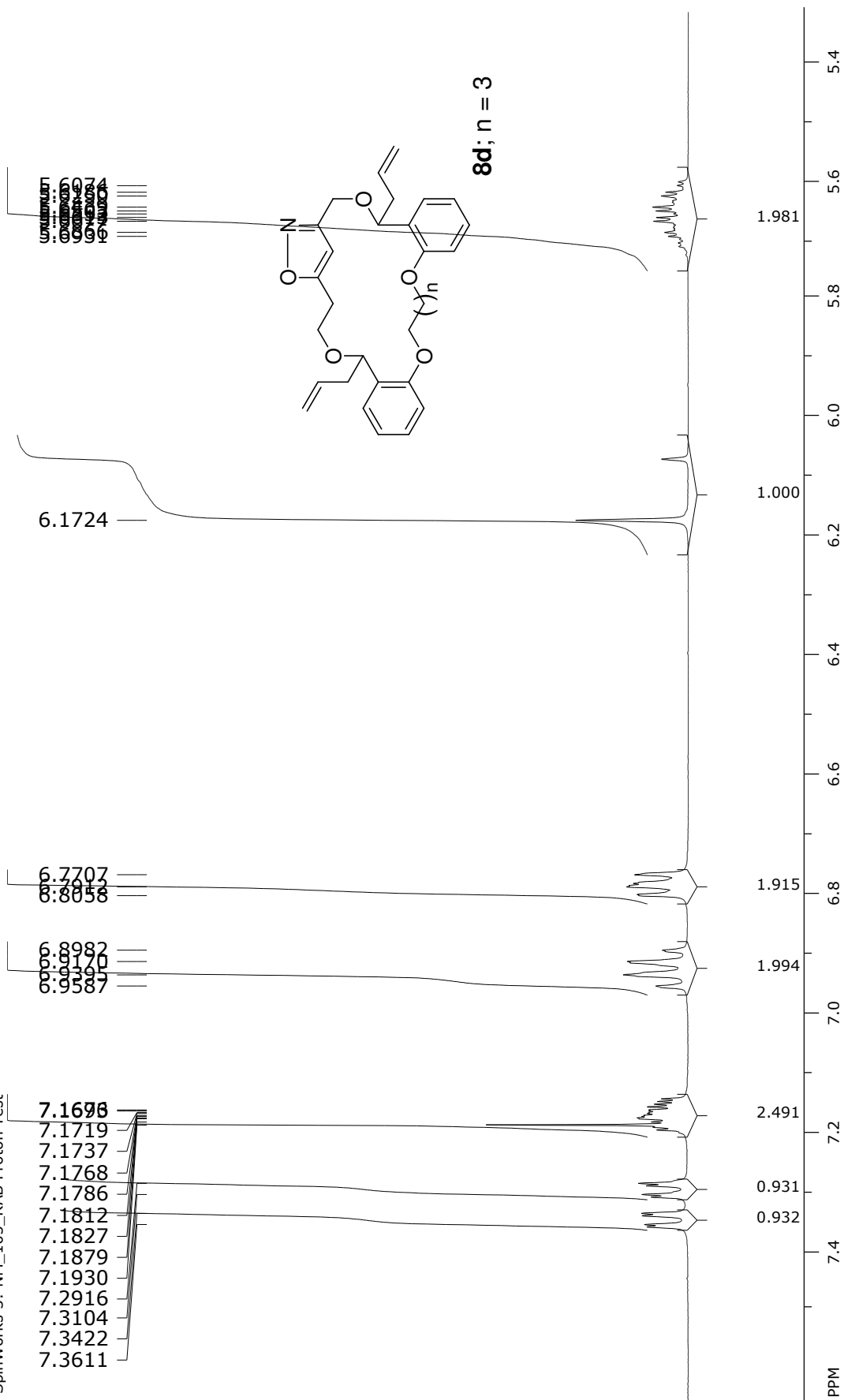
60.1516 —
60.7745 —

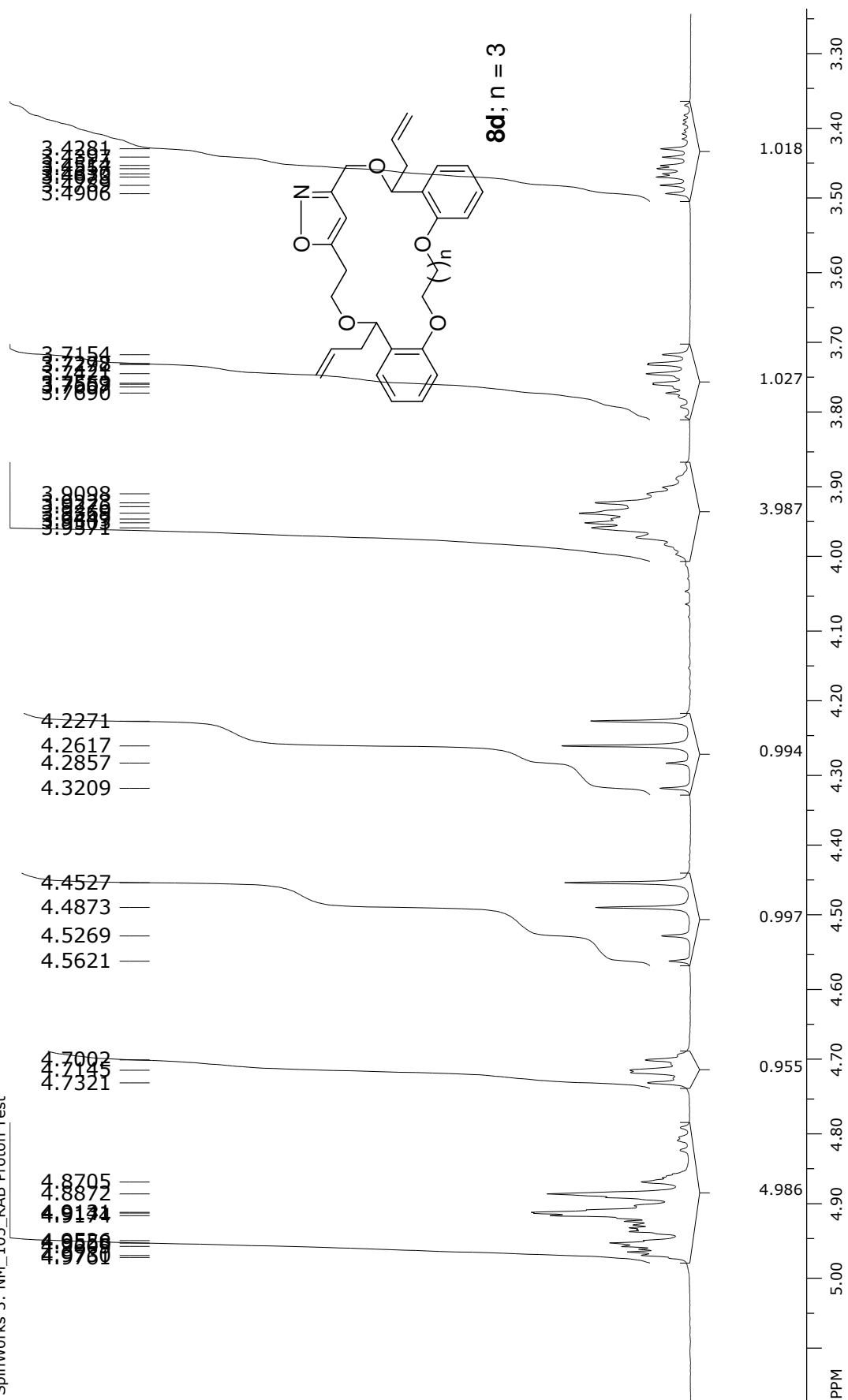
65.7577	—
66.6166	—
66.6368	—
67.5635	—
67.9162	—
69.3530	—
71.1232	—
72.4869	—

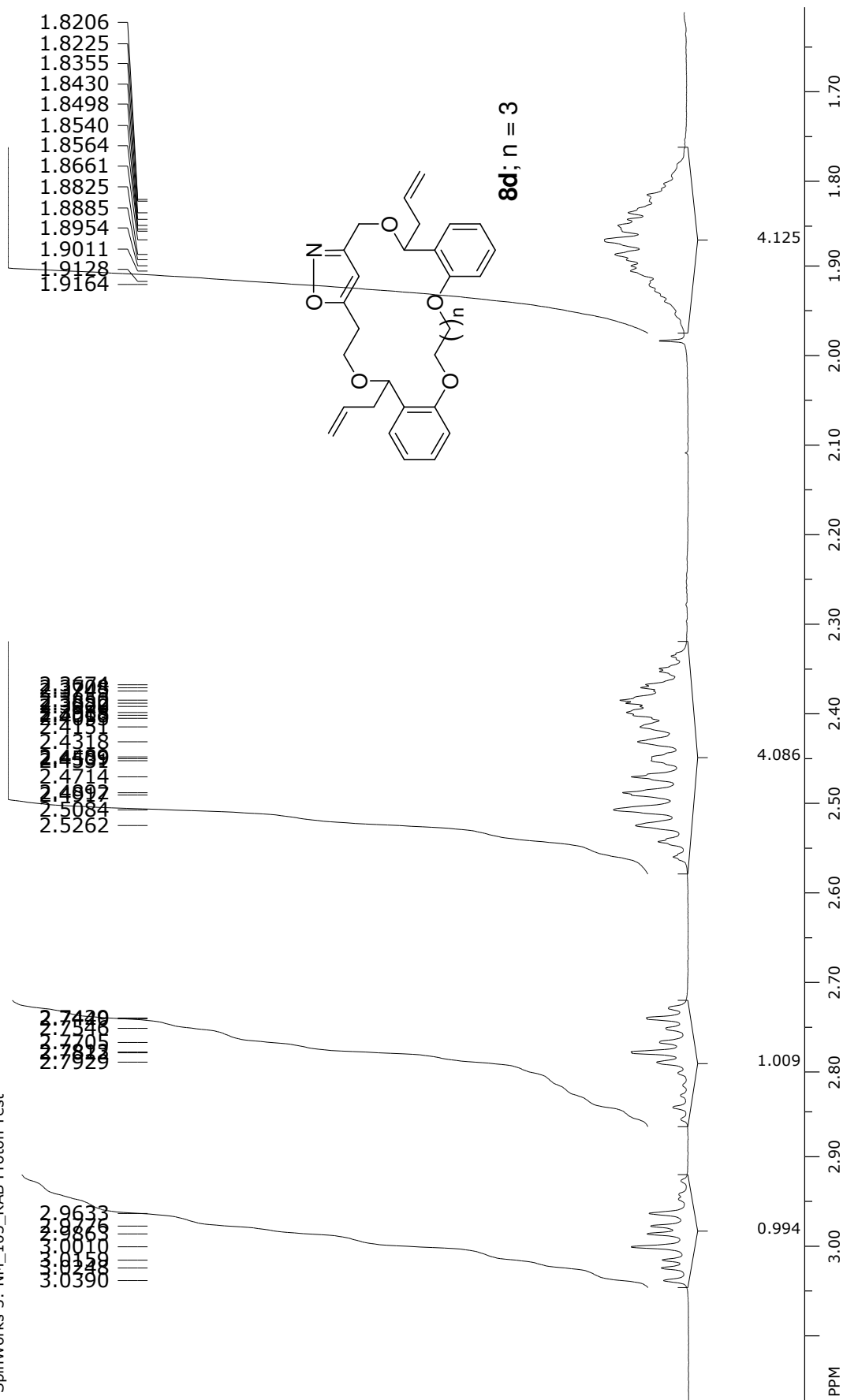




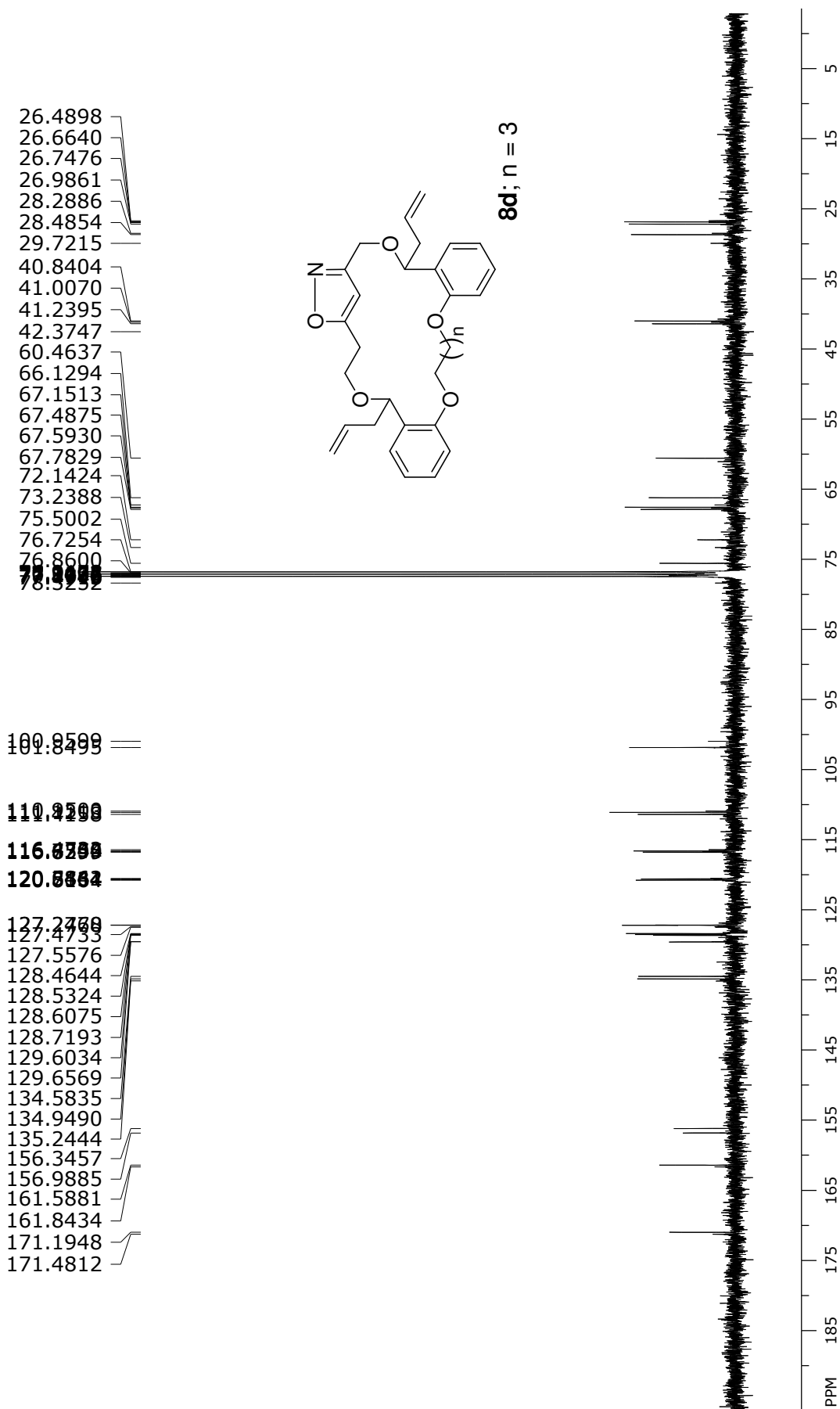
SpinWorks 3: NM_105_RAB Proton Test







1
1
1
1
1
1
1
1



161.5881 —
161.8434 —

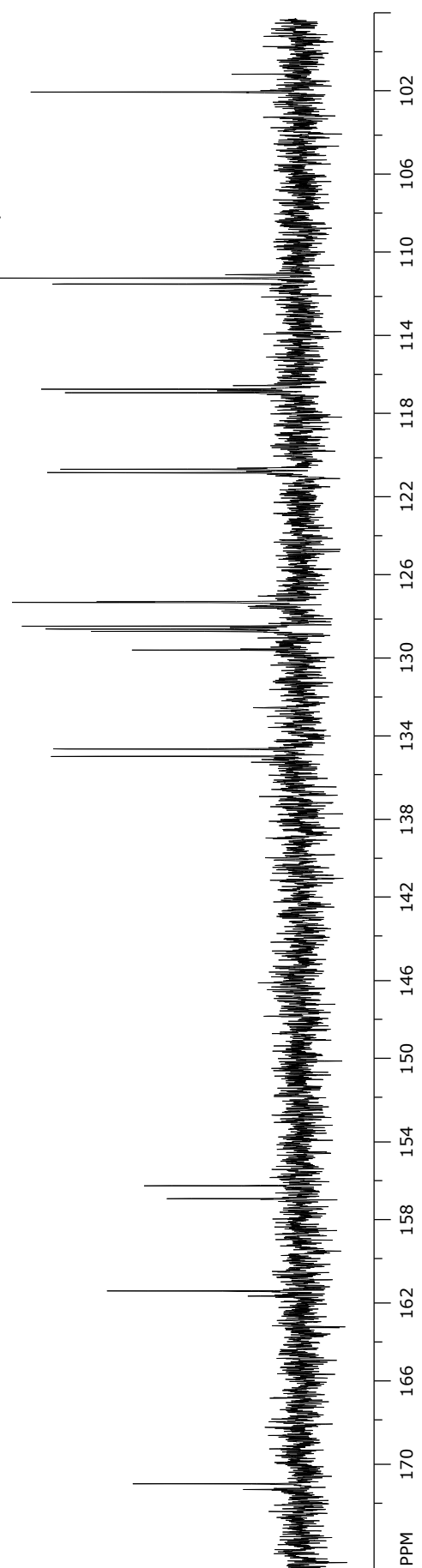
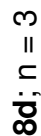
171.1948 —
171.4812 —

156.3457 —
156.9885 —

127.2468
127.2770
127.4733
127.5576
128.4644
128.5324
128.6075
128.7193
129.6034
129.6569
134.5835
134.9490
135.2444

110.9500
111.1213
111.4158
116.4732
116.6544
116.7355
116.8299
120.5842
120.6461
120.7534
120.8164

100.9599 —
101.8495 —



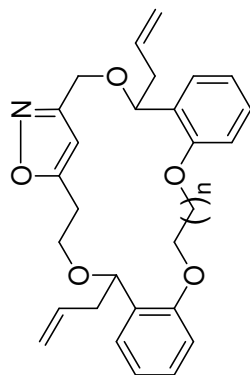
26.6640
26.7476
26.9861
28.2886
28.4854
29.7215

40.8404
41.0070
41.2395
42.3747

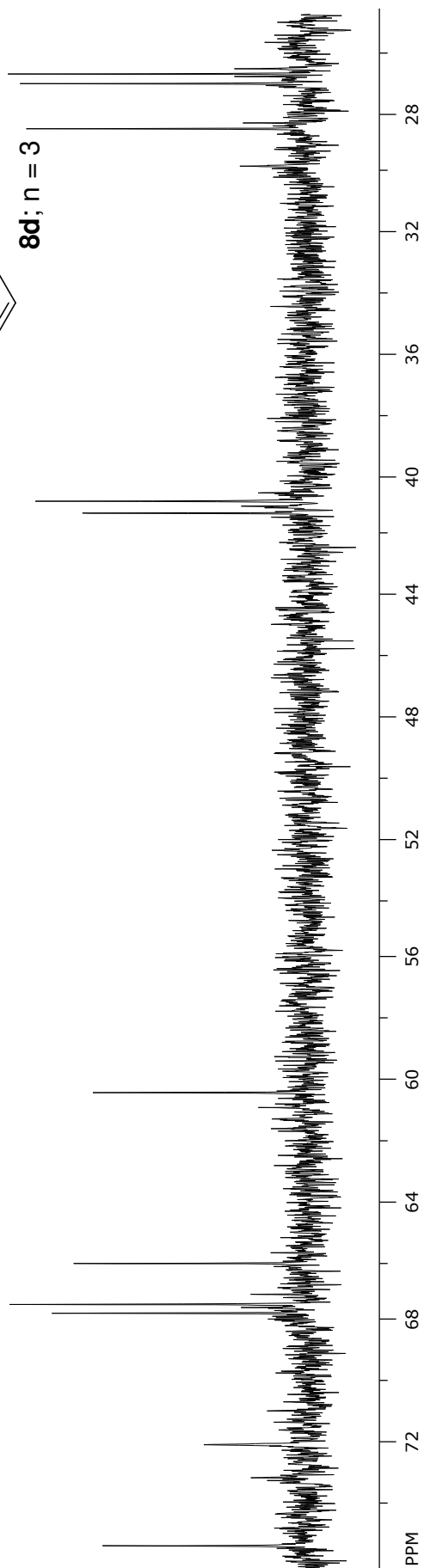
60.4637

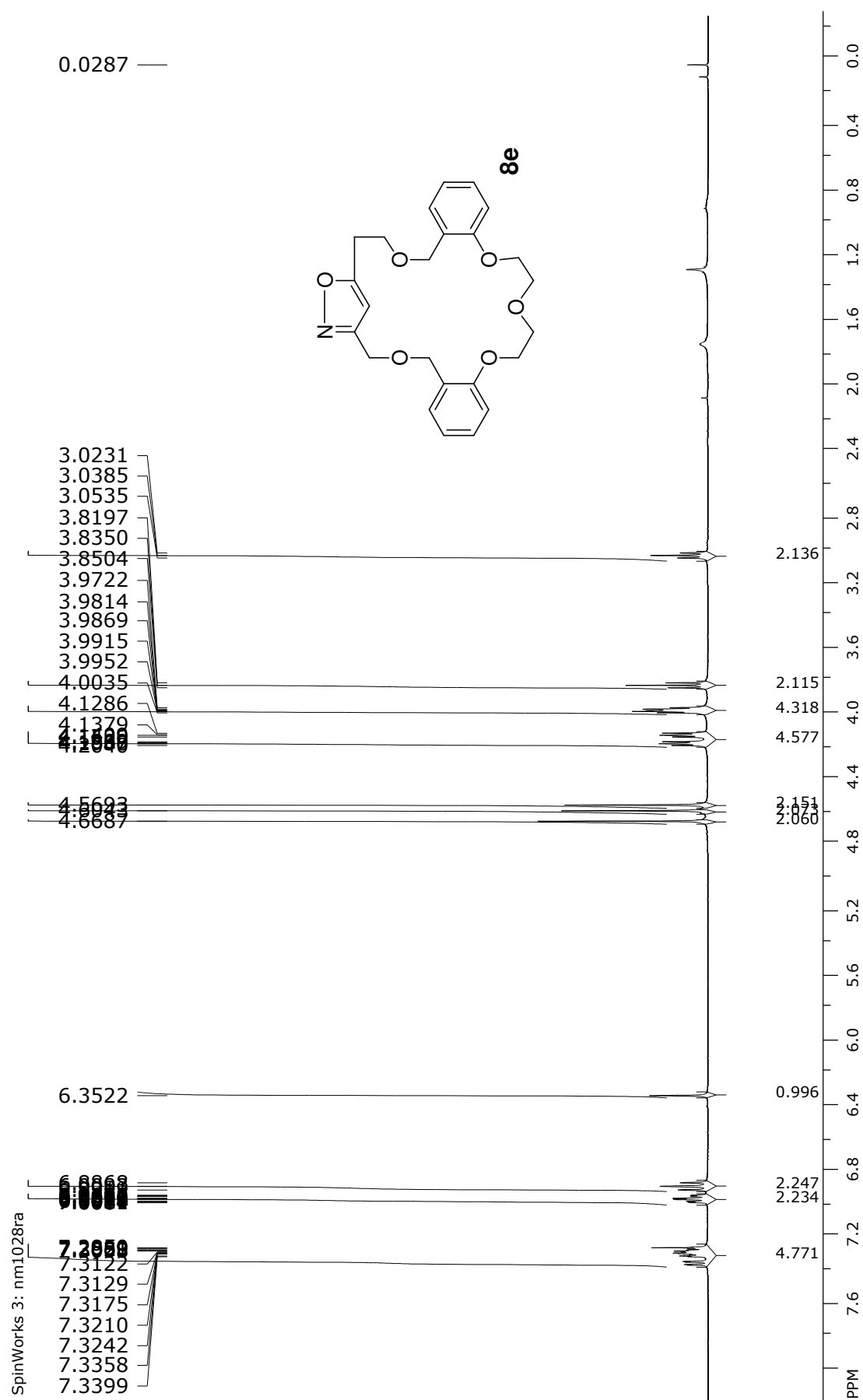
66.1294
67.1513
67.4875
67.5930
67.7829

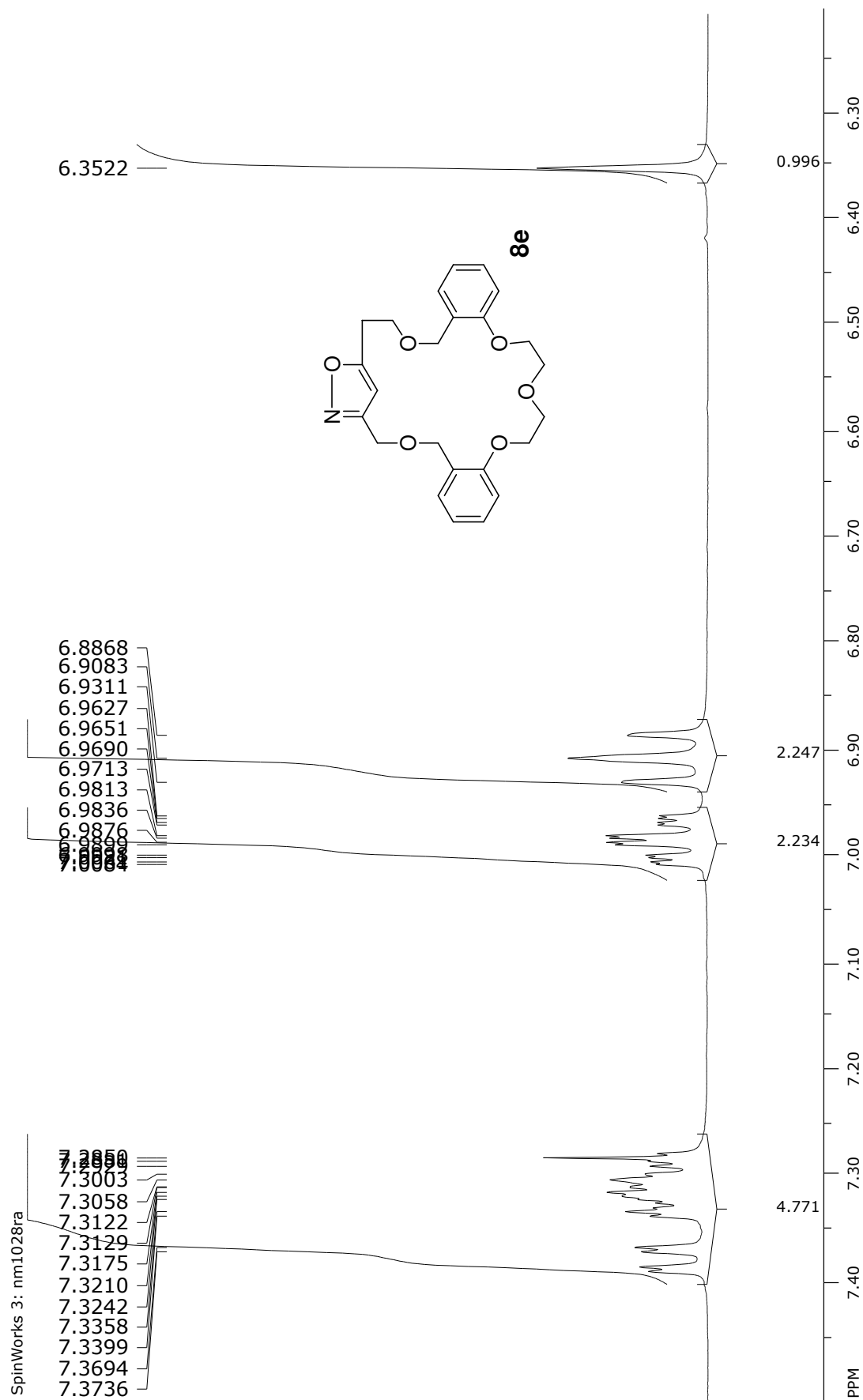
72.1424
73.2388
75.5002

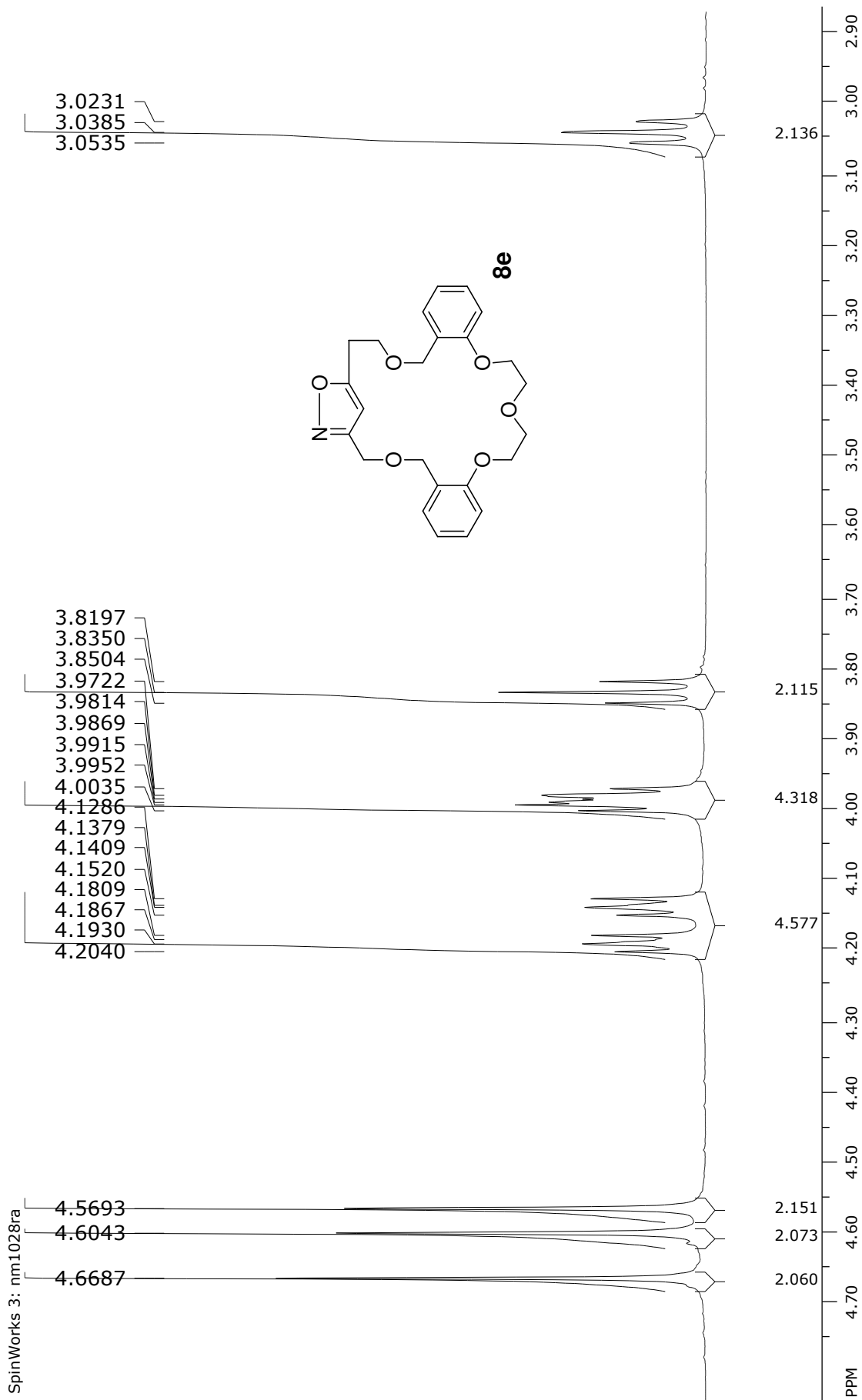


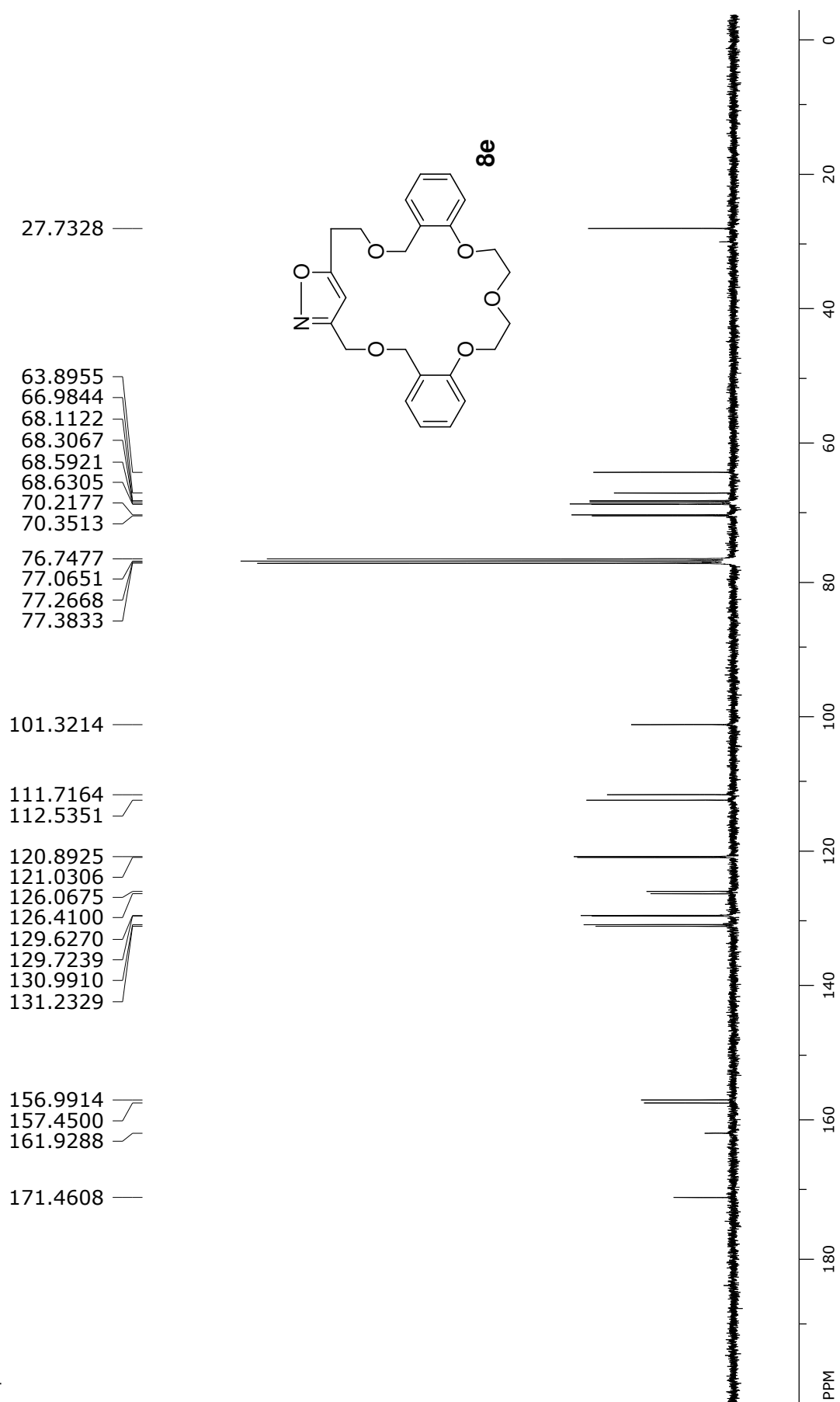
8d; $n = 3$









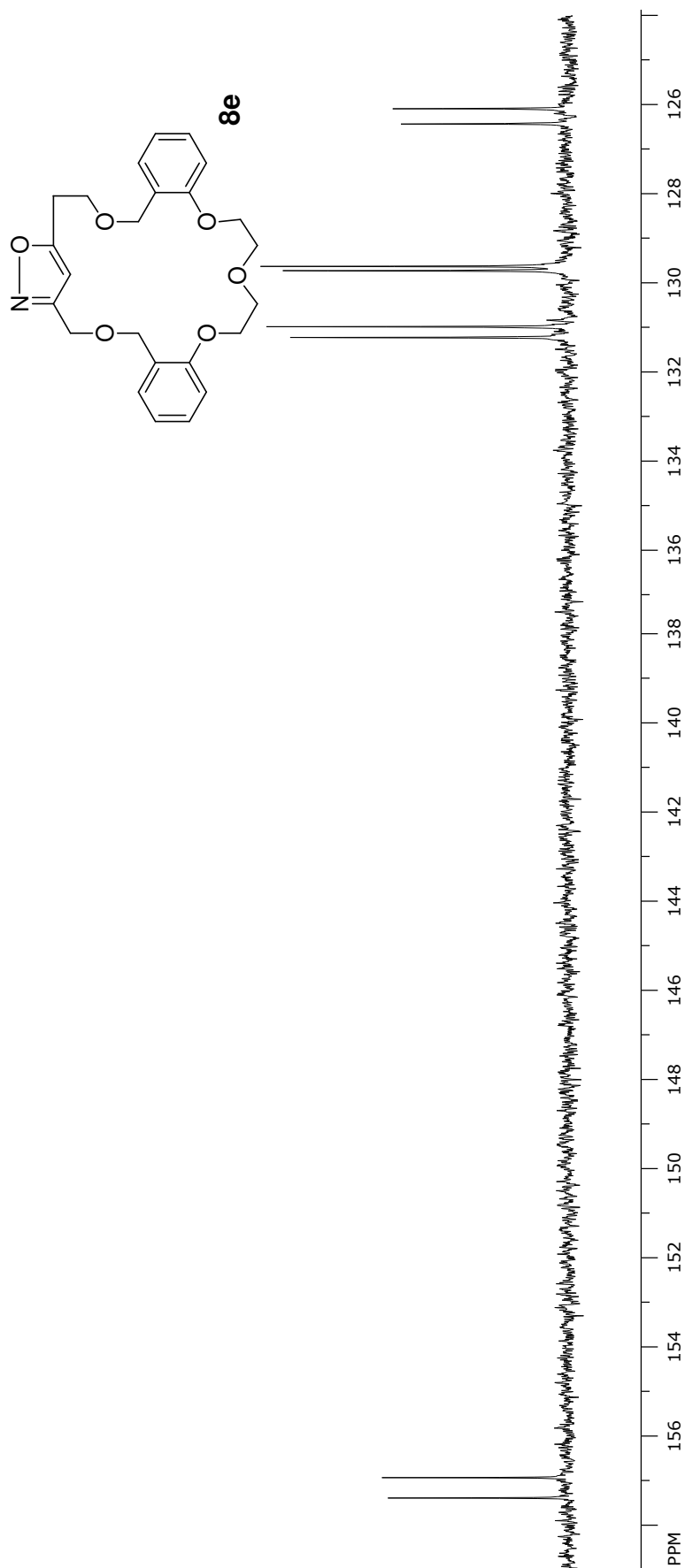


SpinWorks 3: nm1028ra

156.9914 —
157.4500 —

129.6270 —
129.7239 —
130.9910 —
131.2329 —

126.0675 —
126.4100 —



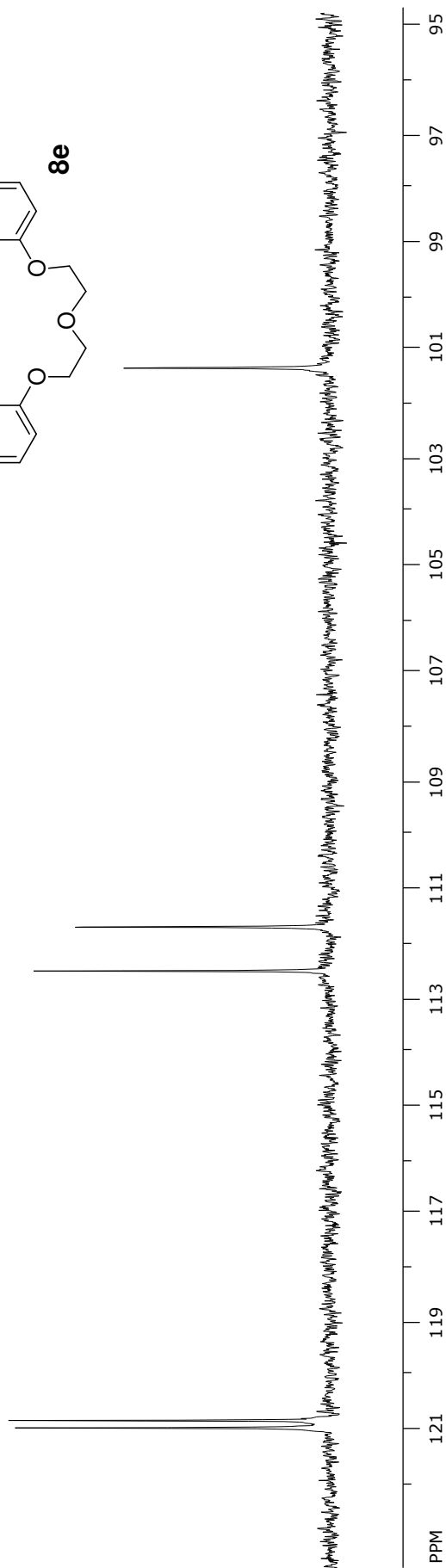
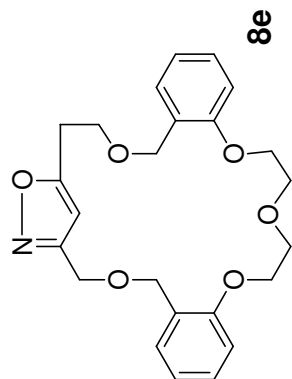
SpinWorks 3: nm1028ra

120.8925
121.0306

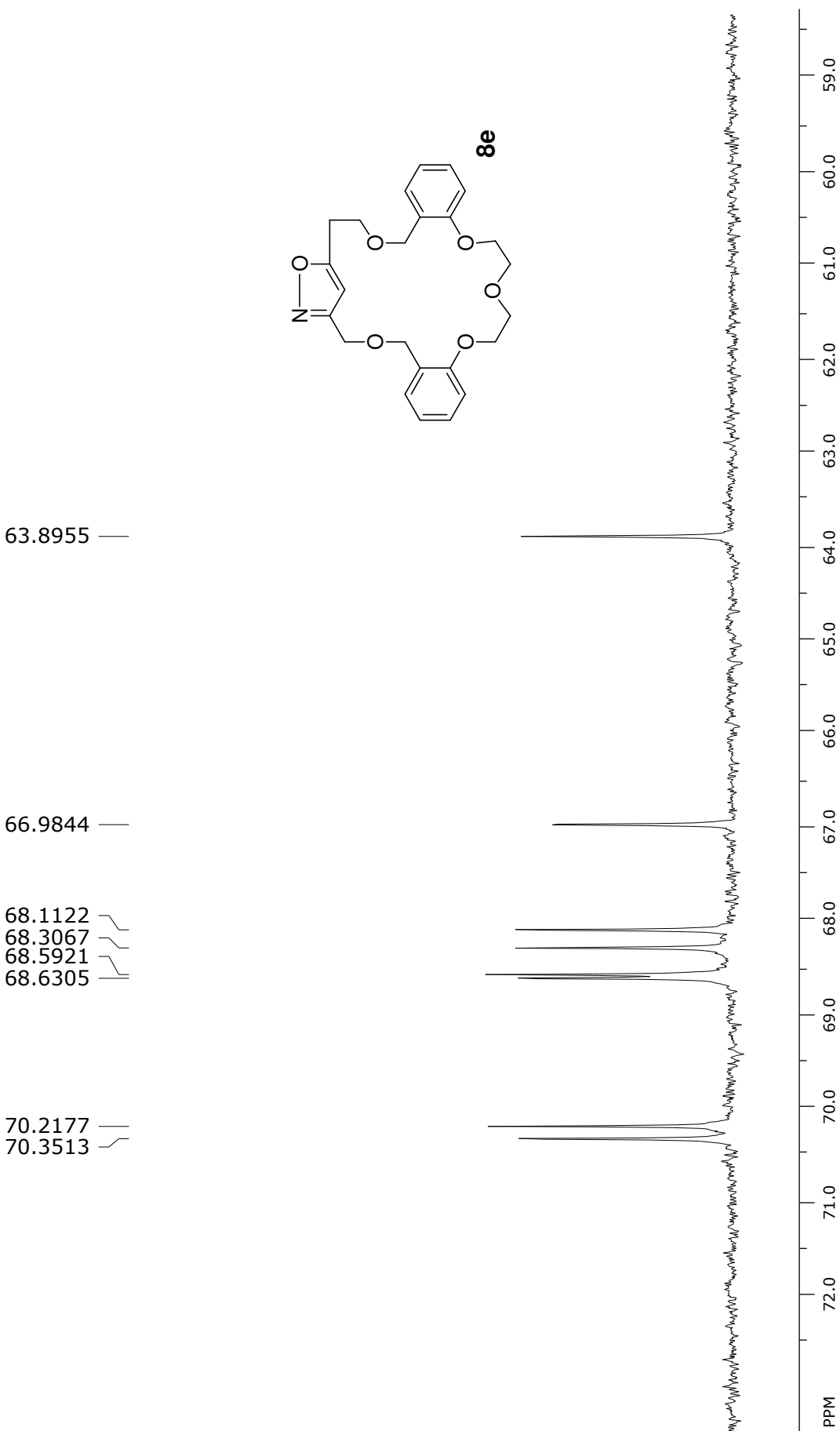
111.7164

112.5351

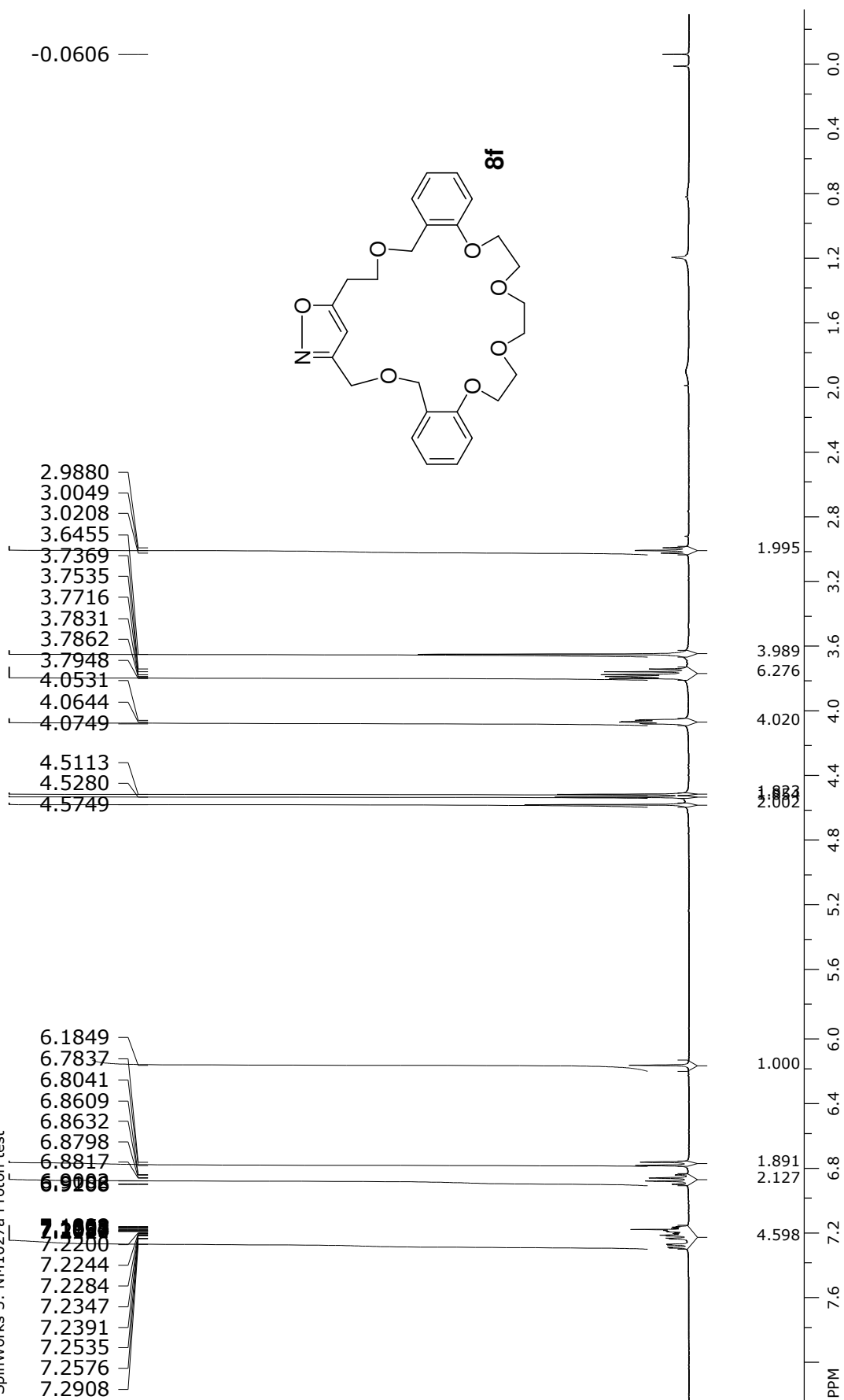
101.3214



SpinWorks 3: nm1028ra



SpinWorks 3: NM1027a Proton test

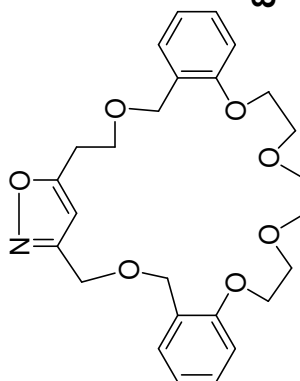


SpinWorks 3: NM1027a Proton test

7.1808
7.1852
7.1893
7.1937
7.1994
7.2055
7.2080
7.2091
7.2127
7.2200
7.2244
7.2284
7.2347
7.2391
7.2535
7.2576
7.2908
7.2949
7.3093

6.9208
6.9186
6.9002
6.8817
6.8798
6.8632
6.8609
6.8041
6.7837

6.1849



4.598

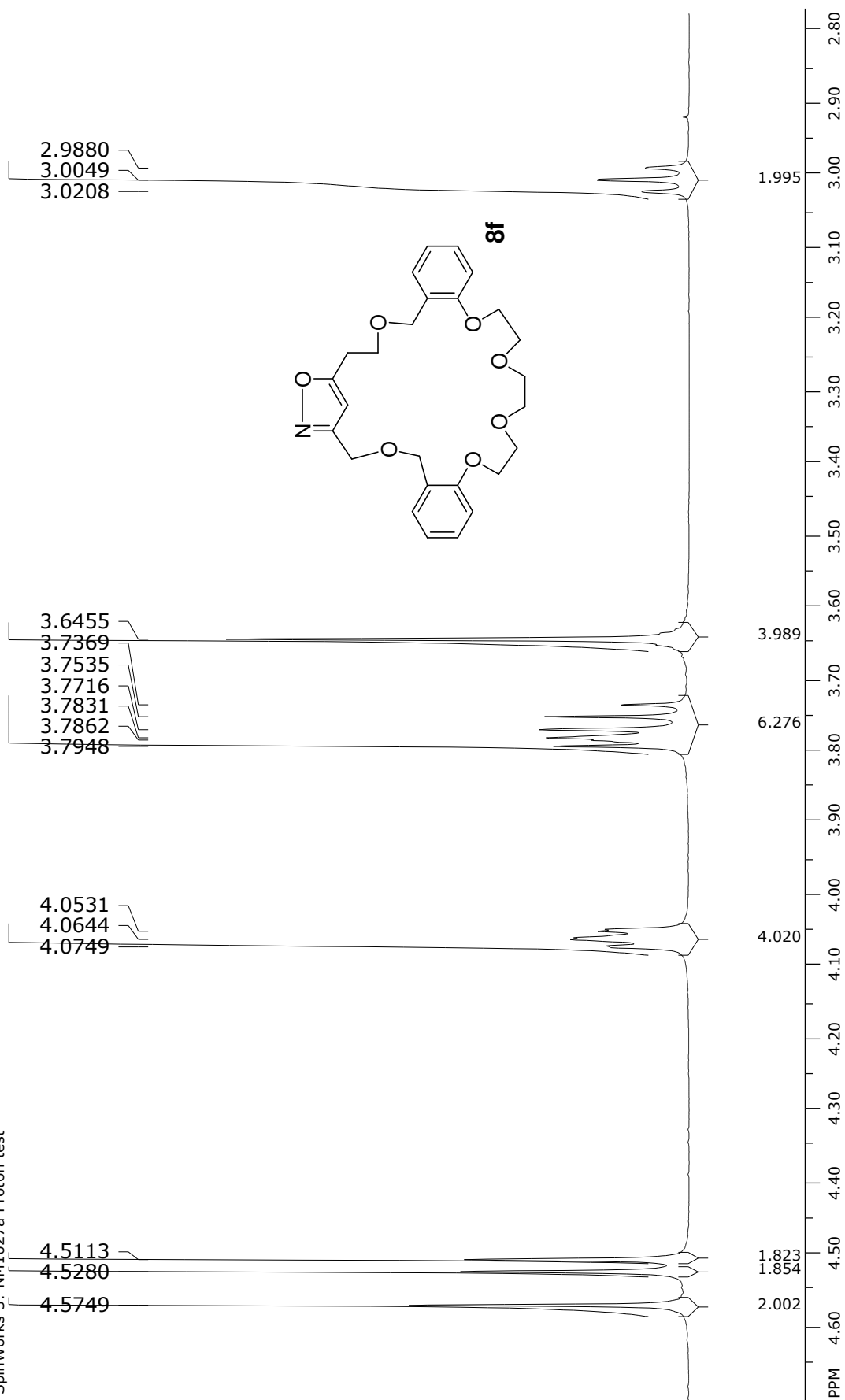
2.127

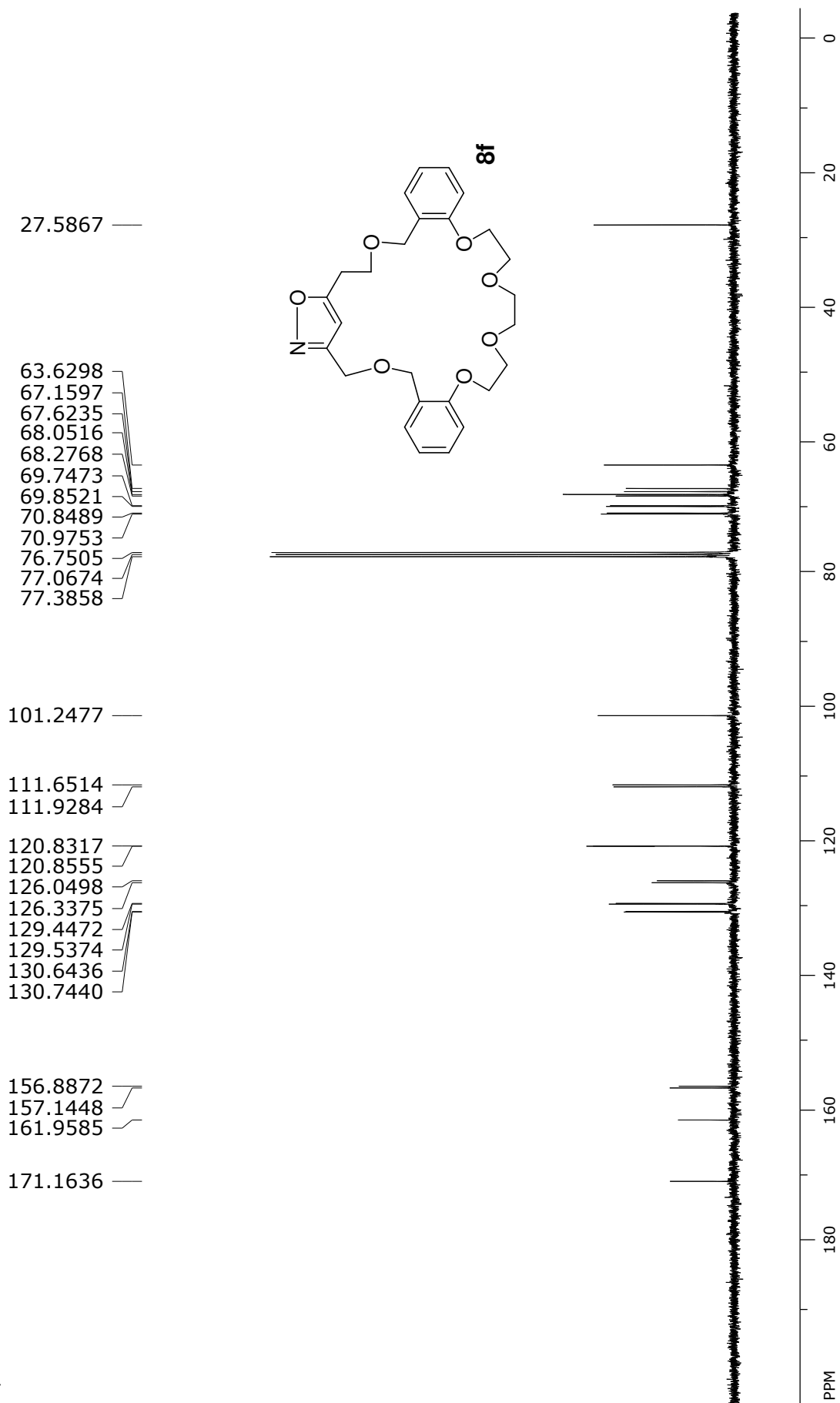
1.891

1.000

PPM

SpinWorks 3: NM1027a Proton test



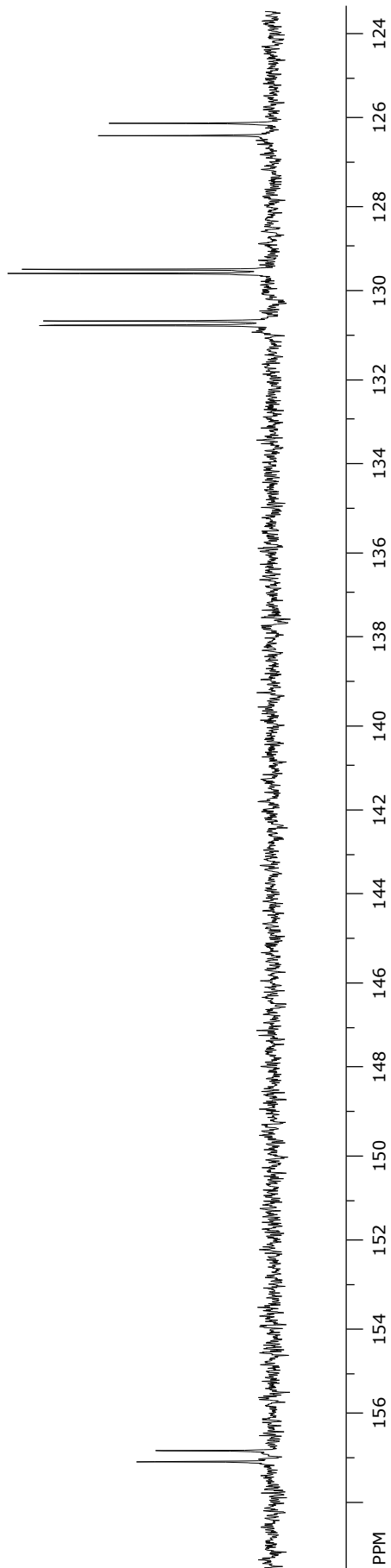
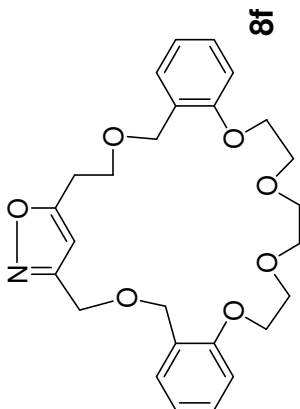


SpinWorks 3: NM1027a Carbon test

156.8872 —
157.1448 —

126.0498 —
126.3375 —

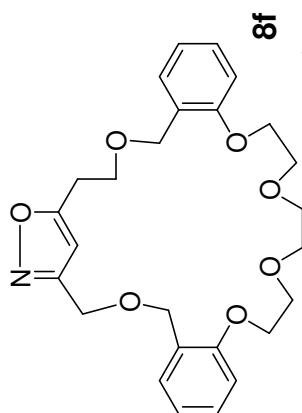
129.4472 —
129.5374 —
130.6436 —
130.7440 —



SpinWorks 3: NM1027a Carbon test

120.8317
120.8555

111.6514
111.9284



SpinWorks 3: NM1027a Carbon test

70.8489 —
70.9753 —

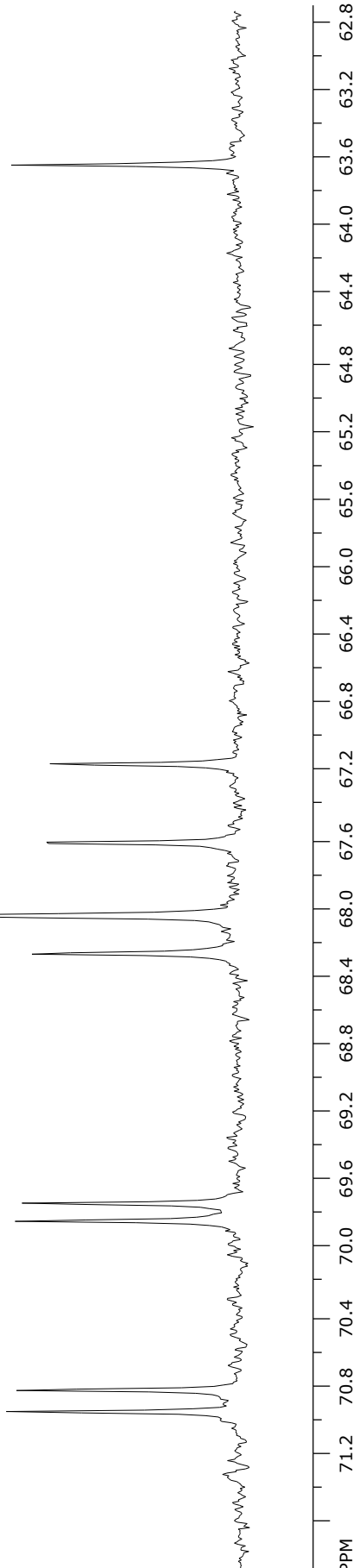
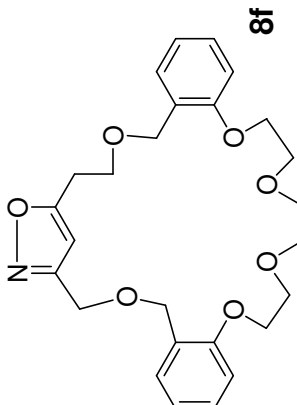
69.7473 —
69.8521 —

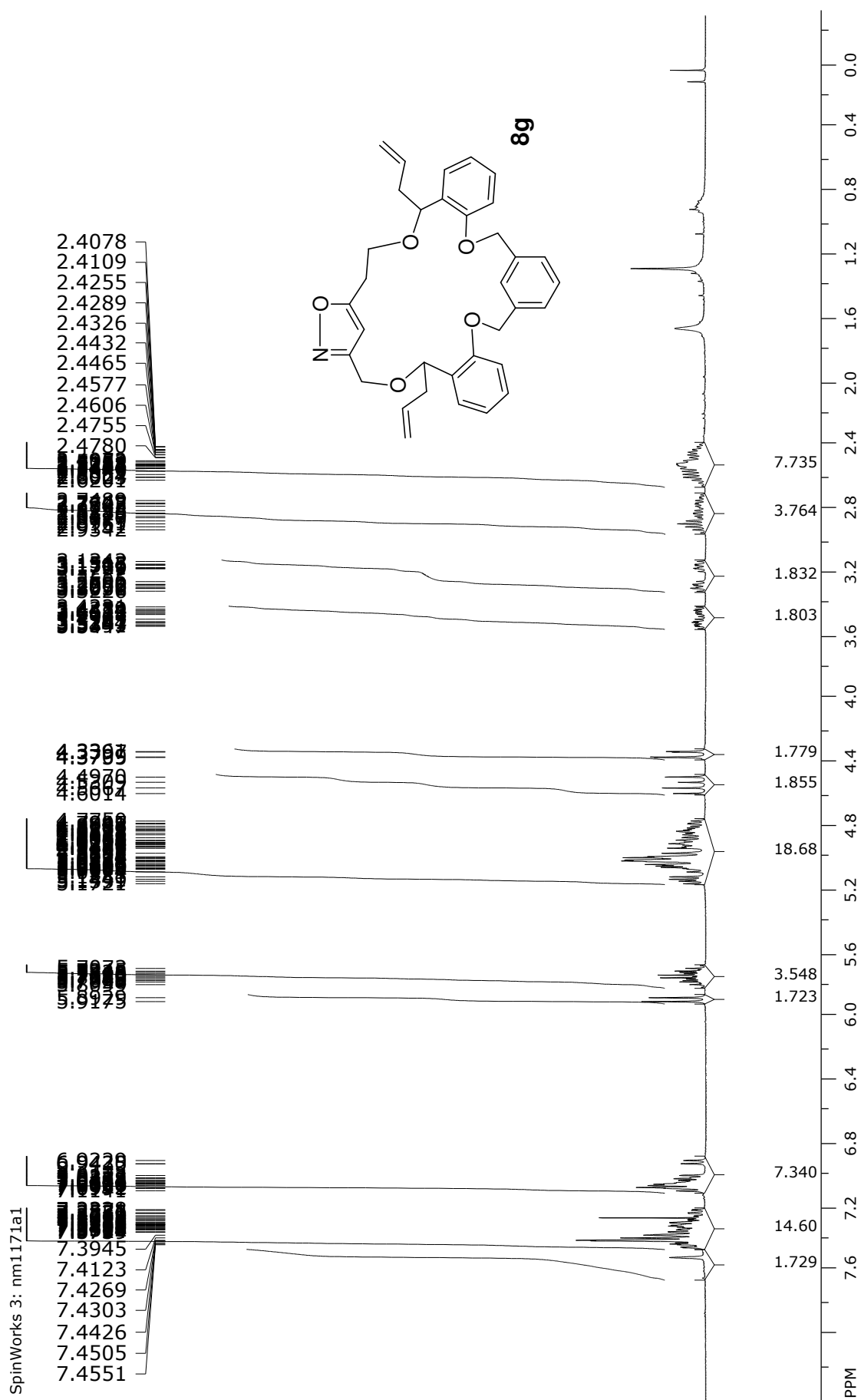
68.0516 —
68.2768 —

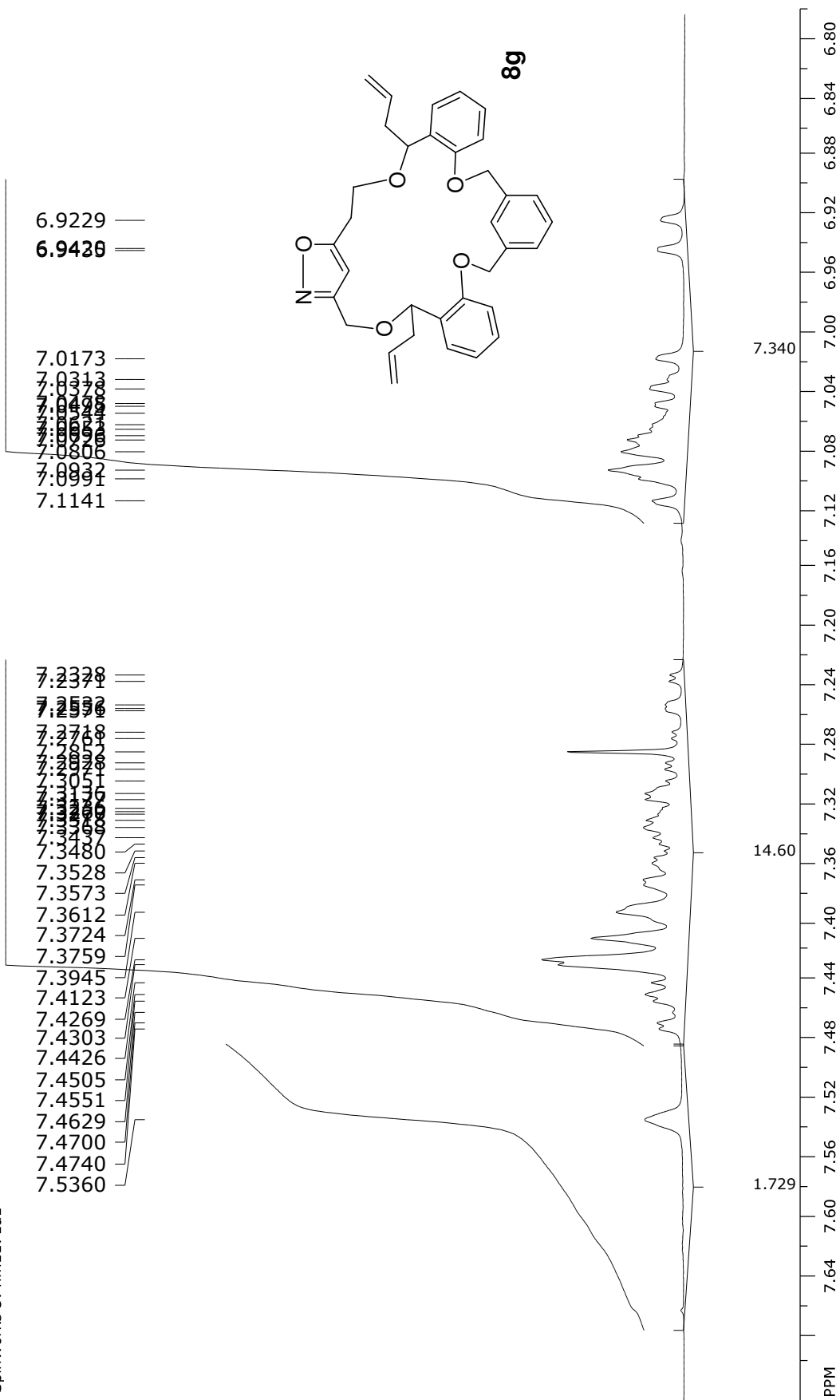
67.6235 —

67.1597 —

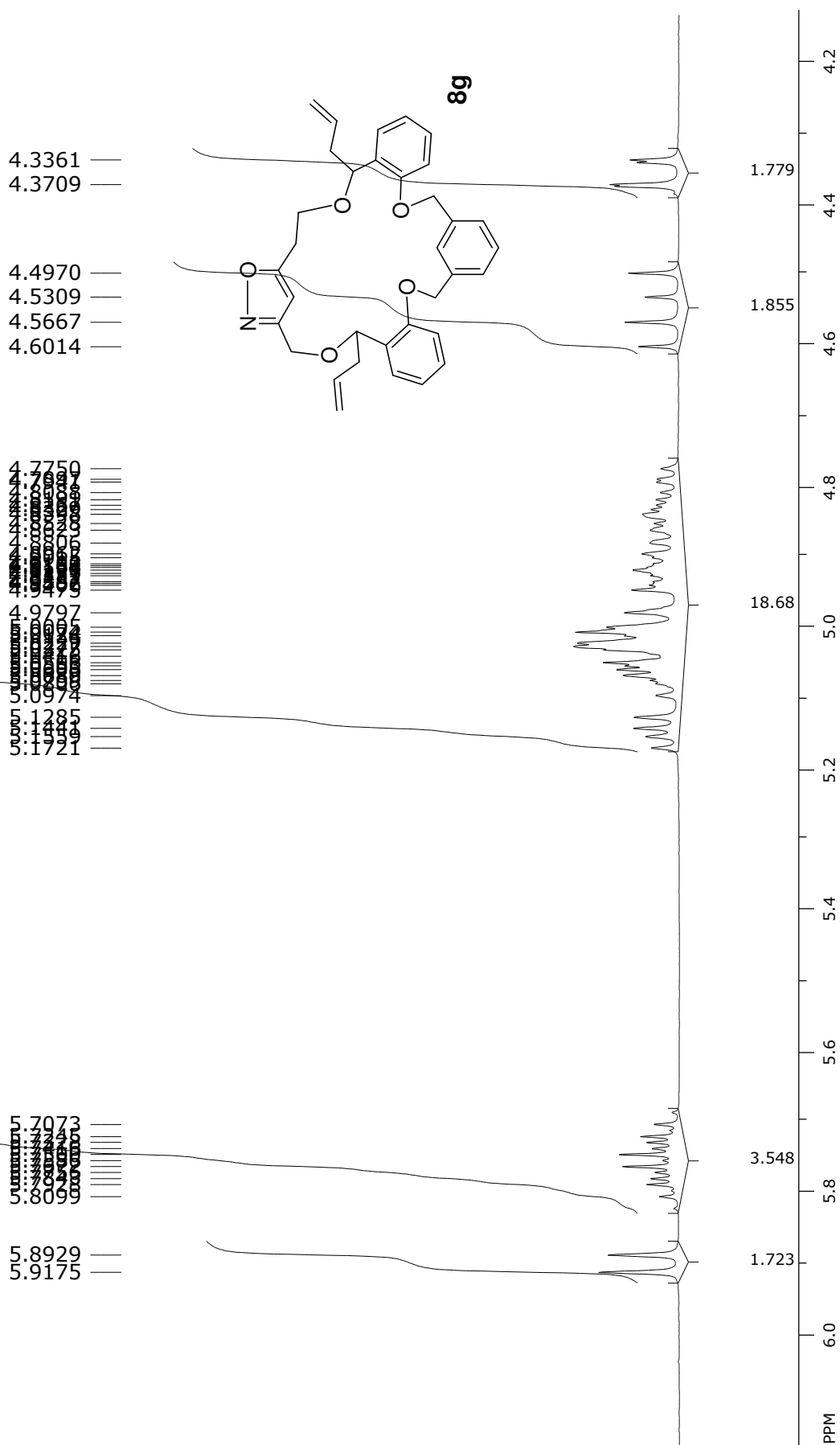
63.6298 —

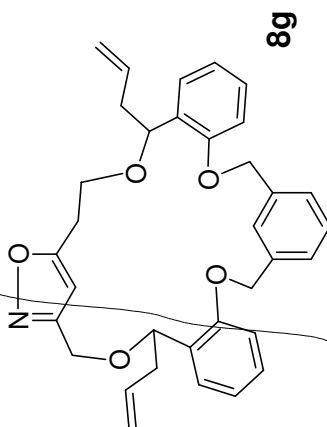




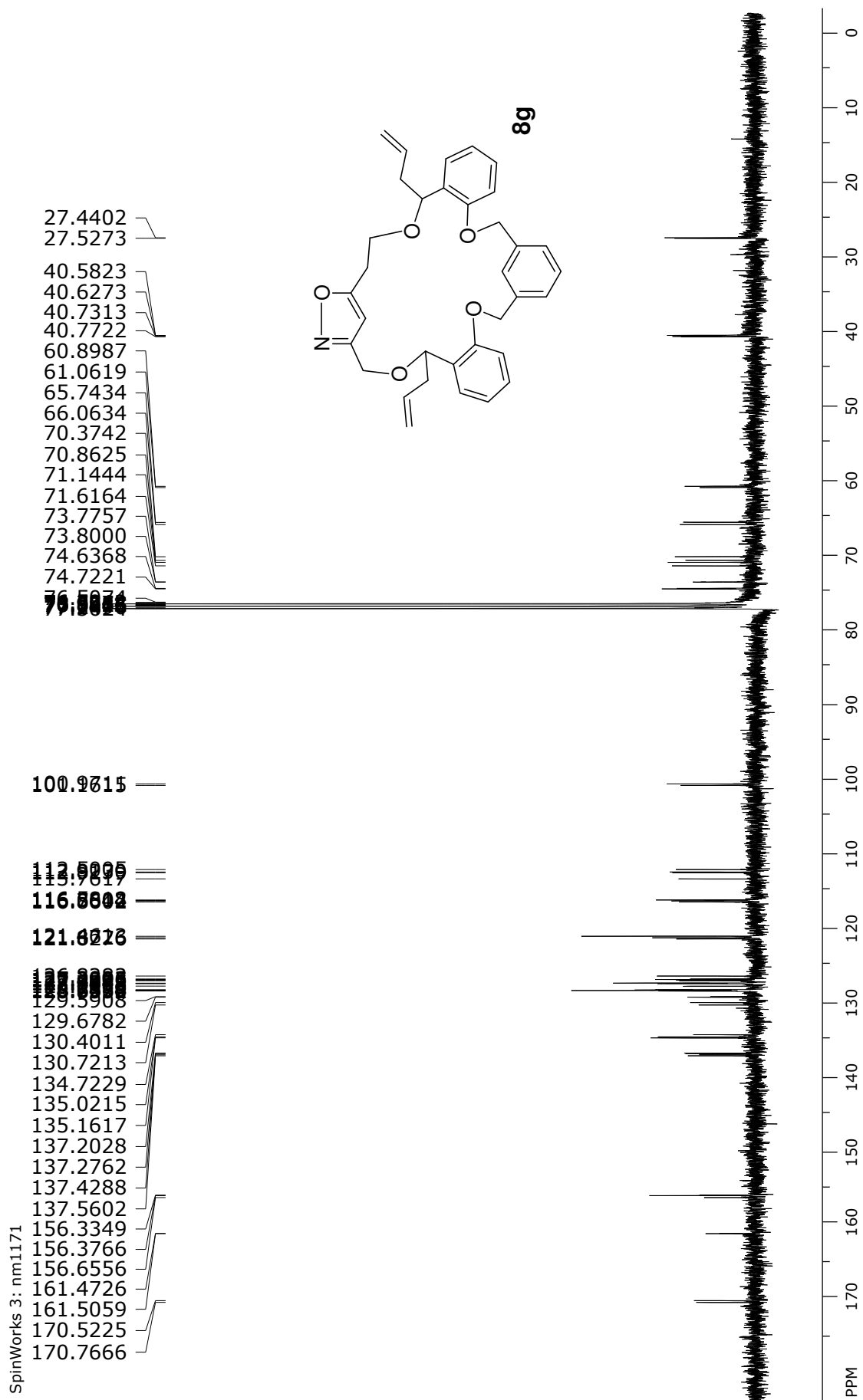


5
7
8
9
0
1
2
3

[illegible]



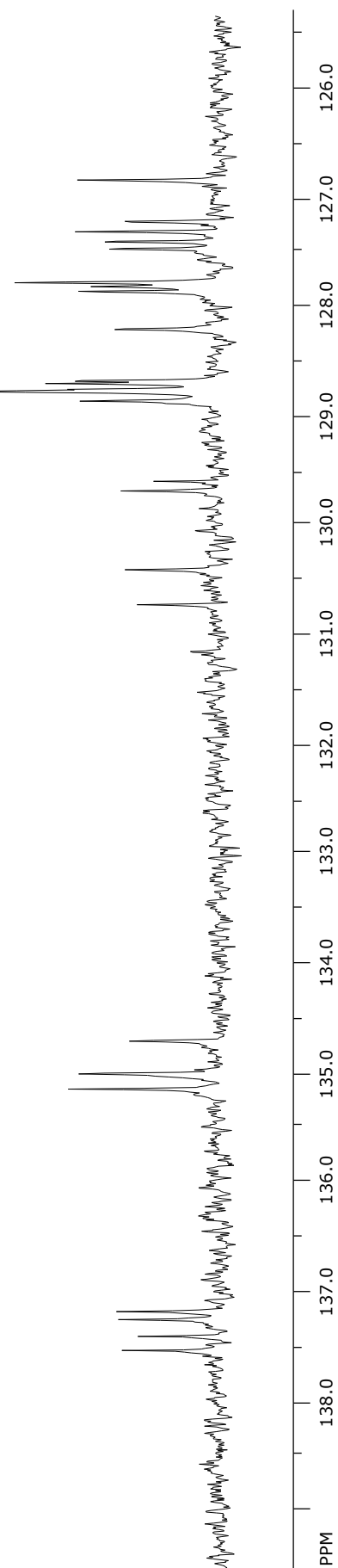
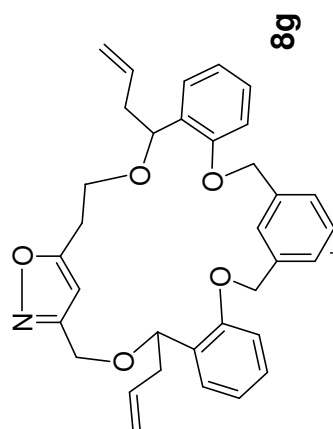
89



137.2028 —
137.2762 —
137.4288 —
137.5602 —

134.7229 —
135.0215 —
135.1617 —

126.8283 —
127.2092 —
127.3006 —
127.3935 —
127.4595 —
127.7675 —
127.8042 —
127.8498 —
128.1955 —
128.6690 —
128.6939 —
128.7478 —
128.7664 —
128.8532 —
129.5908 —
129.6782 —
130.4011 —
130.7213 —



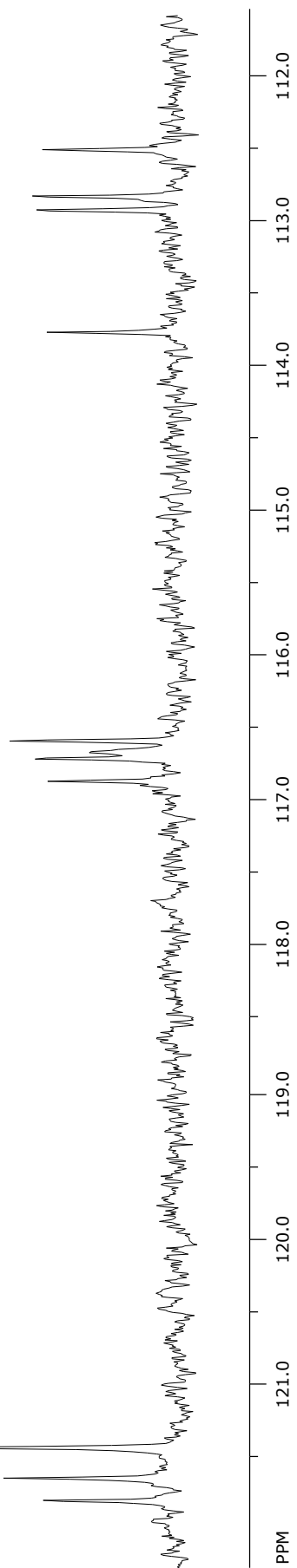
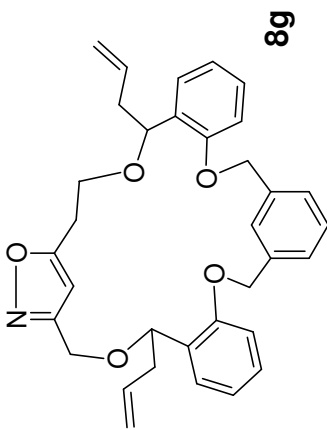
SpinWorks 3: nm1171

121.4613 —
121.6726 —
121.8276 —

116.5818 —
116.6602 —
116.7044 —
116.8602 —

113.7617 —

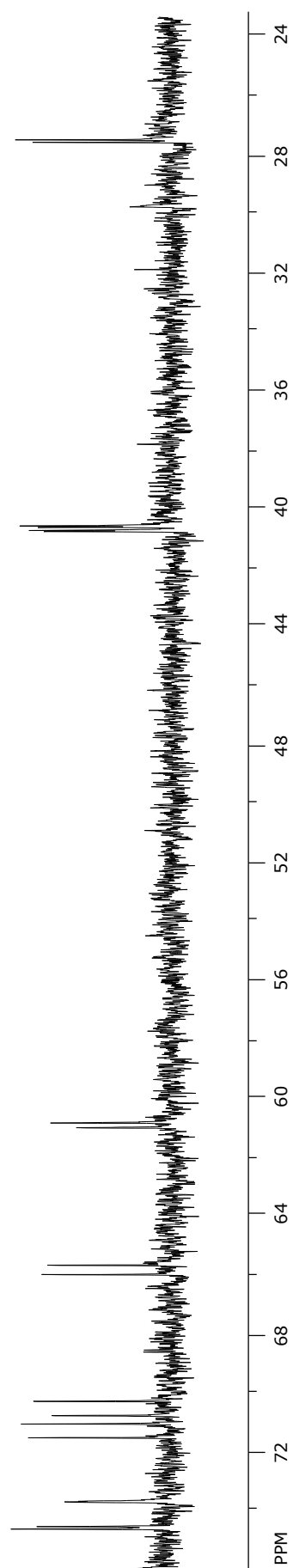
112.5005 —
112.8230 —
112.9179 —

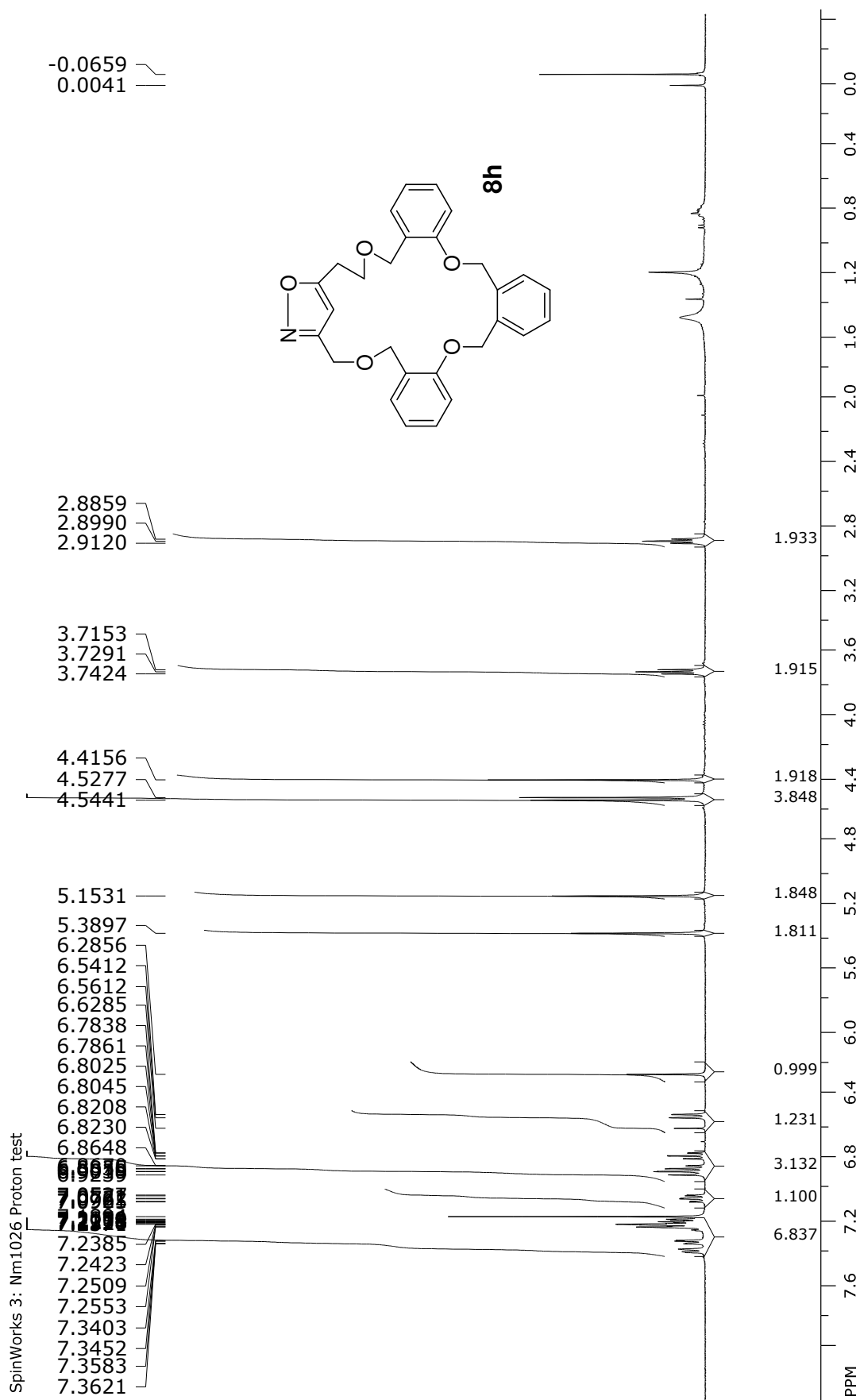


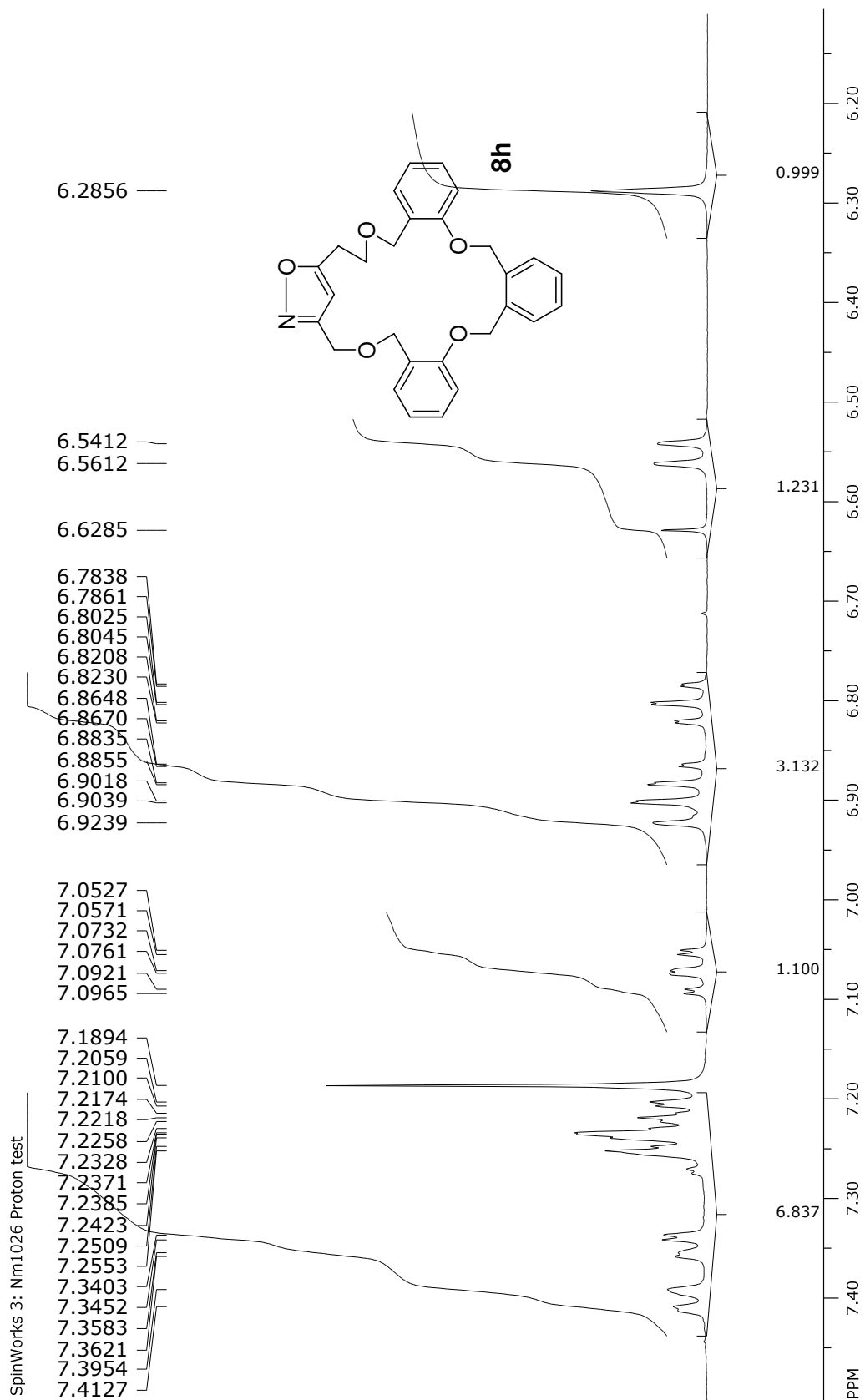
70.3742
70.8625
71.1444
71.6164
73.7757
73.8000
74.6368
74.7221

60.8987
61.0619

27.4402 —
27.5273 —



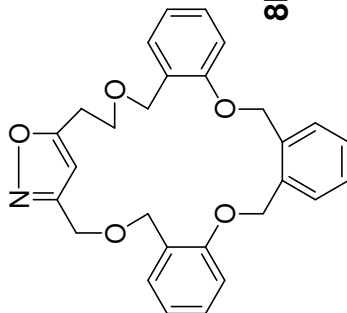






2.8859
2.8990
2.9120

8h



3.7153
3.7291
3.7424

4.4156

4.5277
4.5441

5.1531

5.3897

1.933

1.915

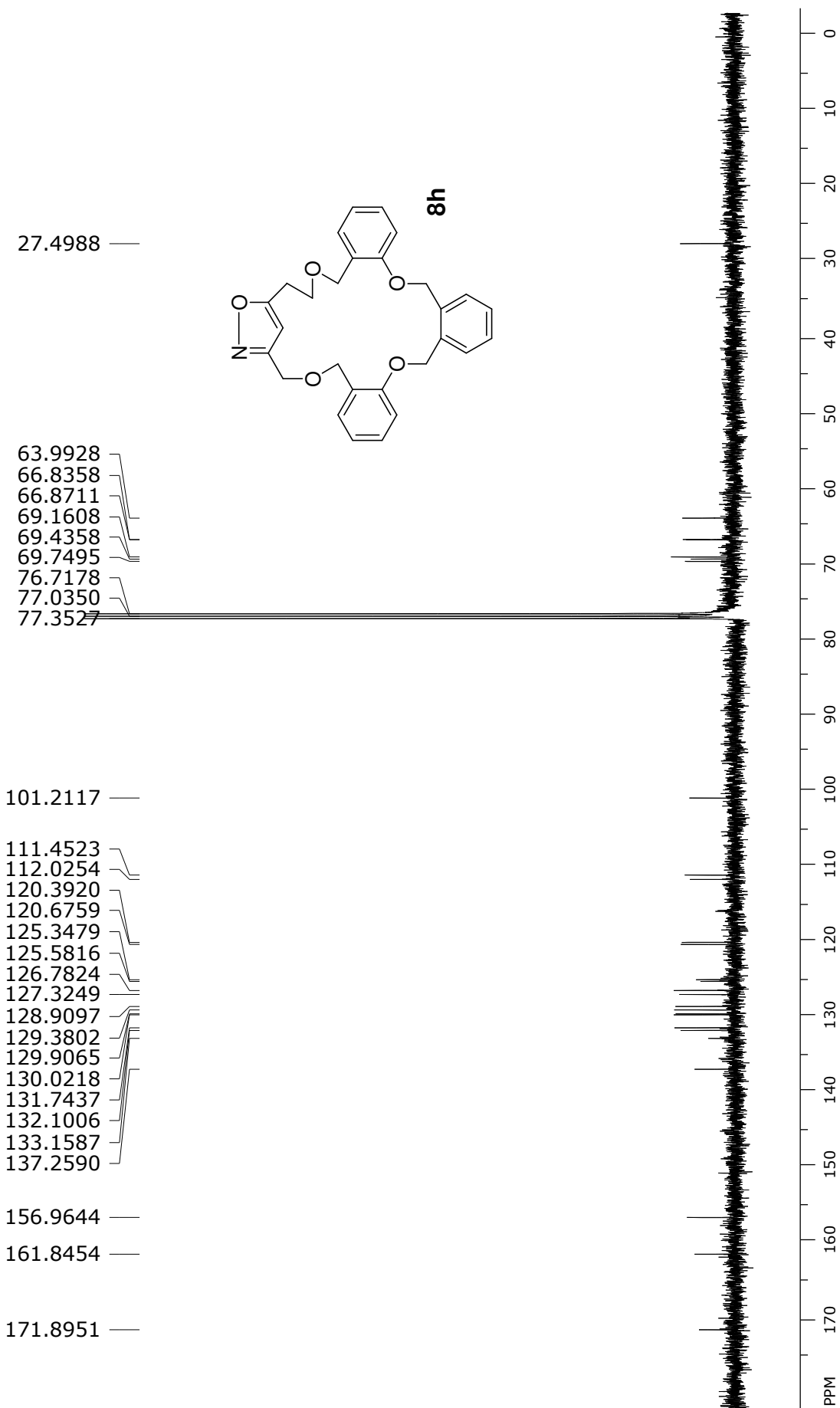
1.918

3.848

1.848

1.811

PPM



SpinWorks 3: NM1026a Carbon test

131.7437 —

132.1006 —

133.1587 —

128.9097 —

129.3802 —

129.9065 —

130.0218

126.7824 —

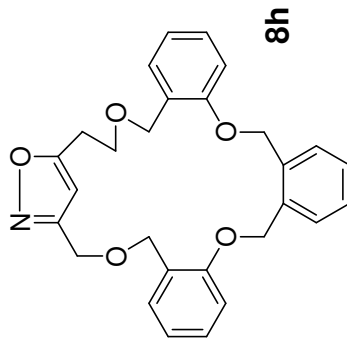
127.3249 —

125.3479 —

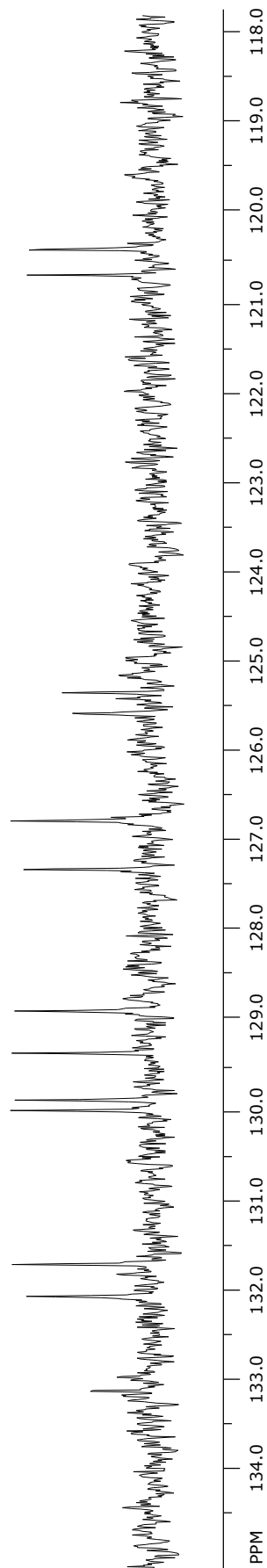
125.5816 —

120.3920 —

120.6759 —



48



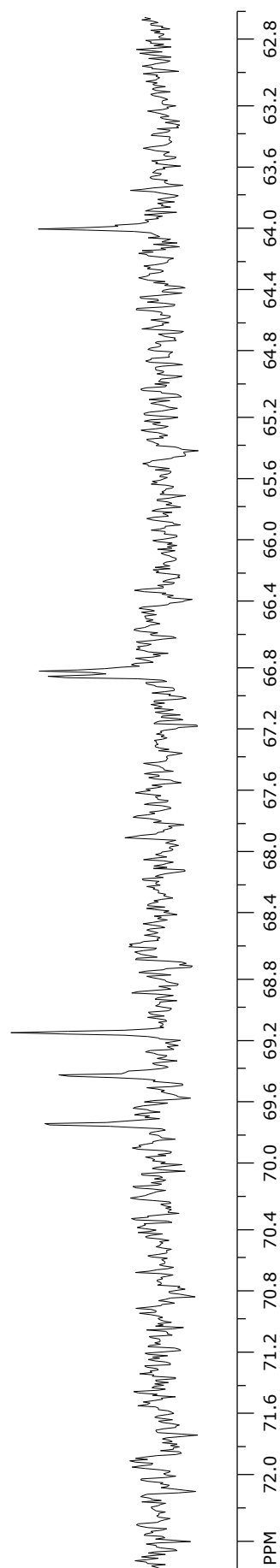
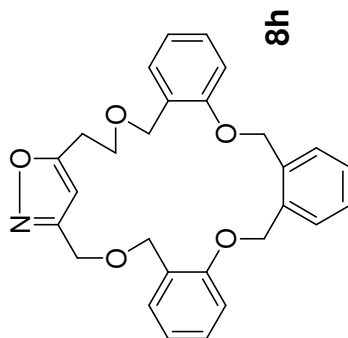
63.9928 —

66.8358 —
66.8711 —

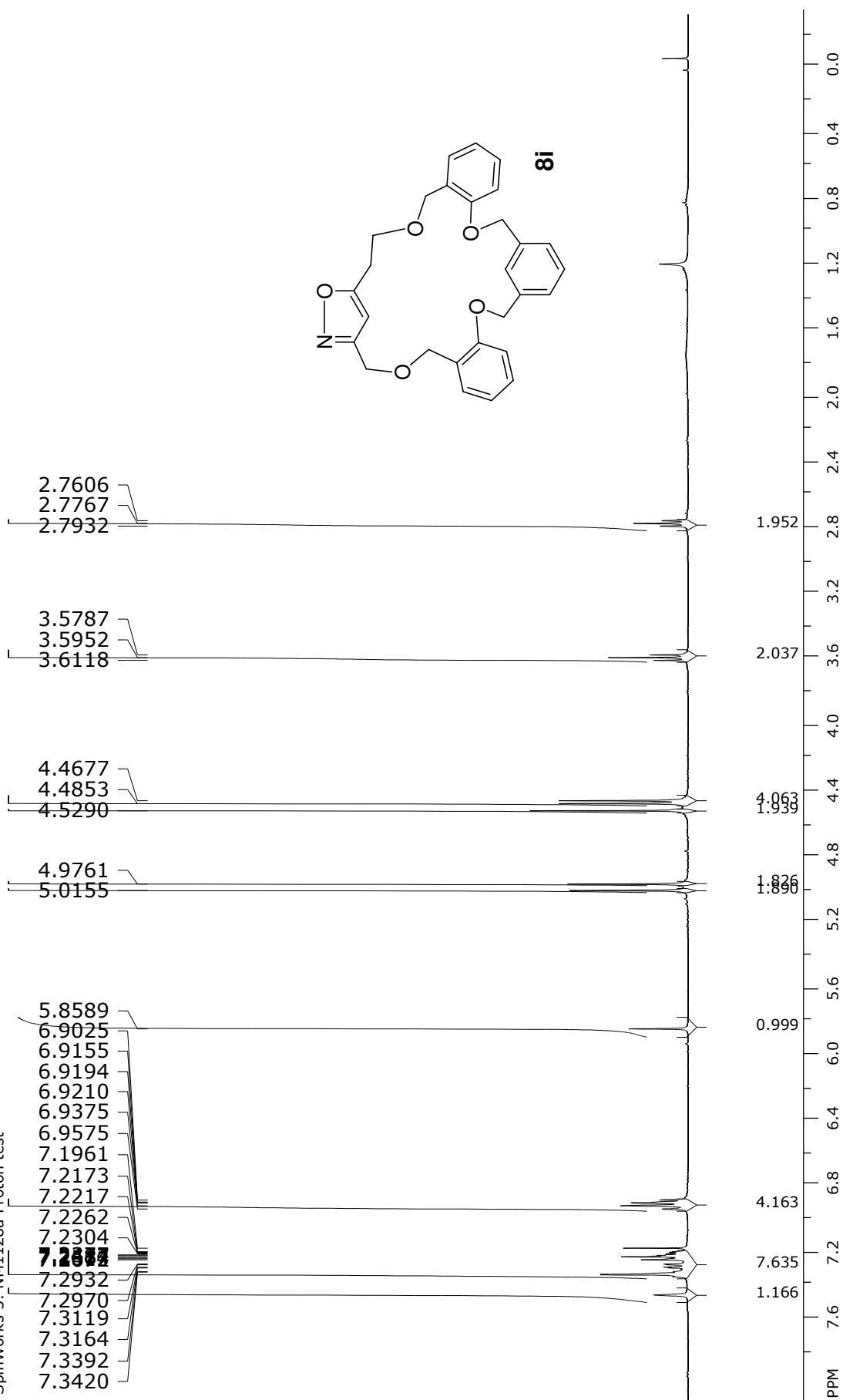
69.1608 —

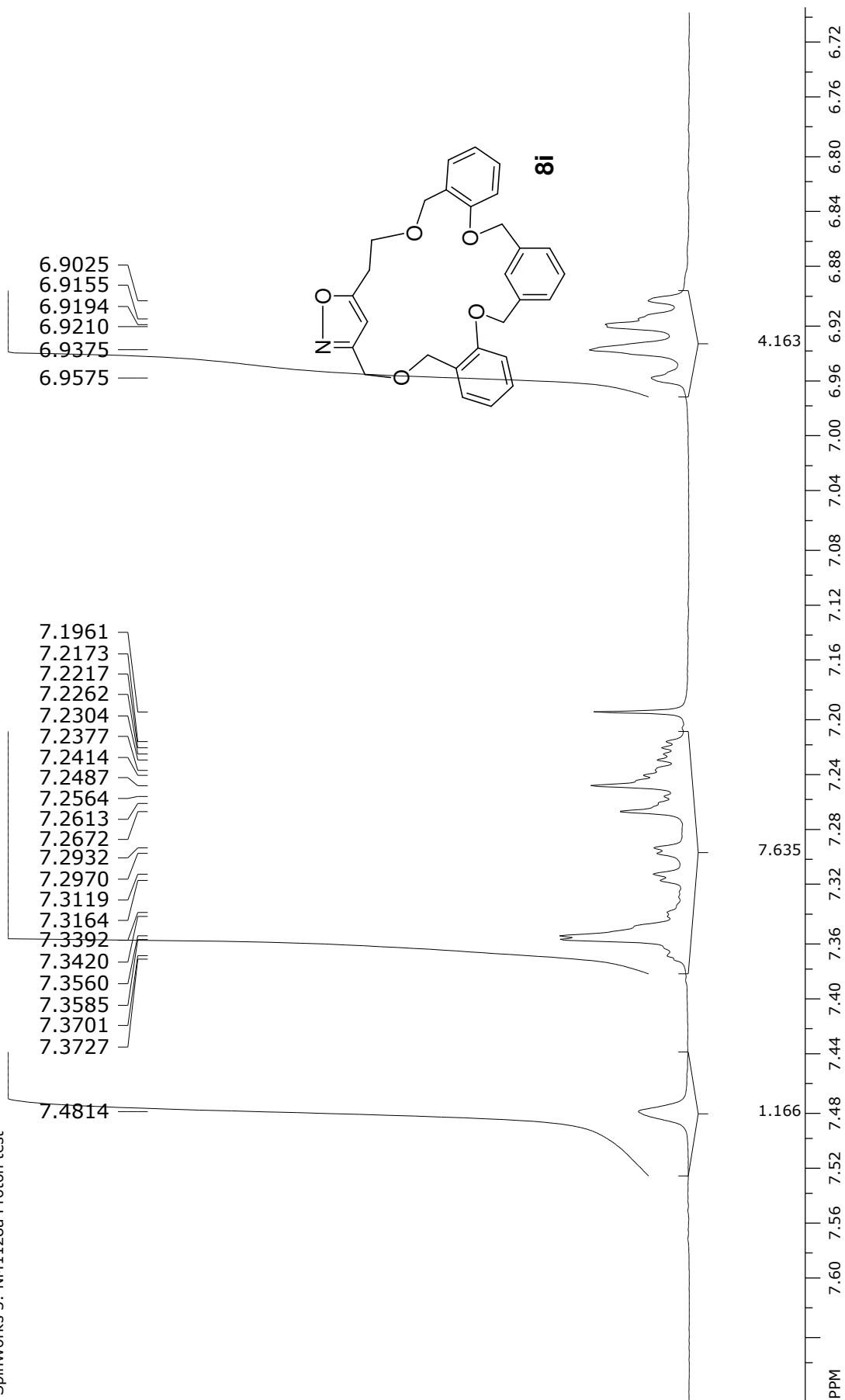
69.4358 —

69.7495 —

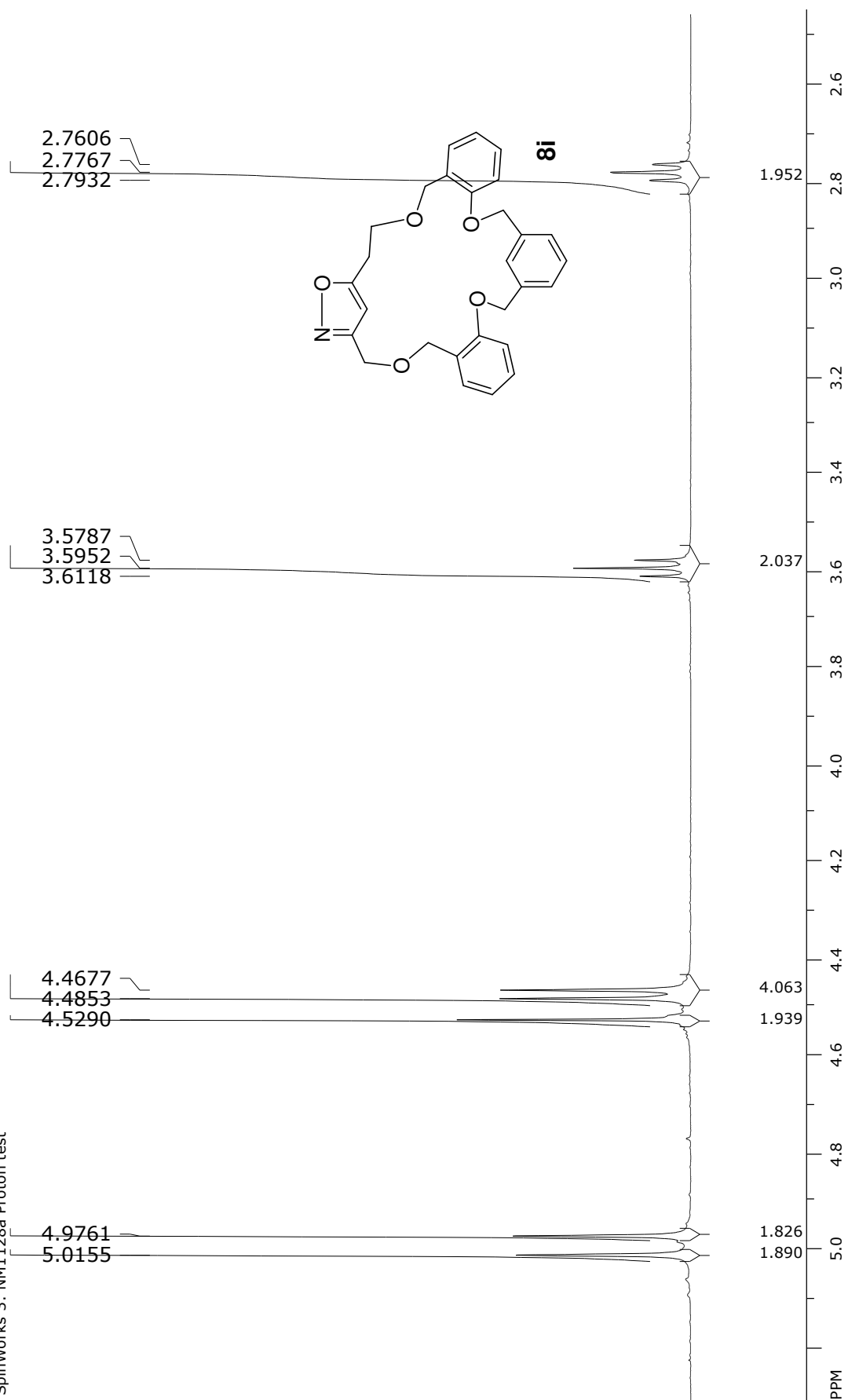


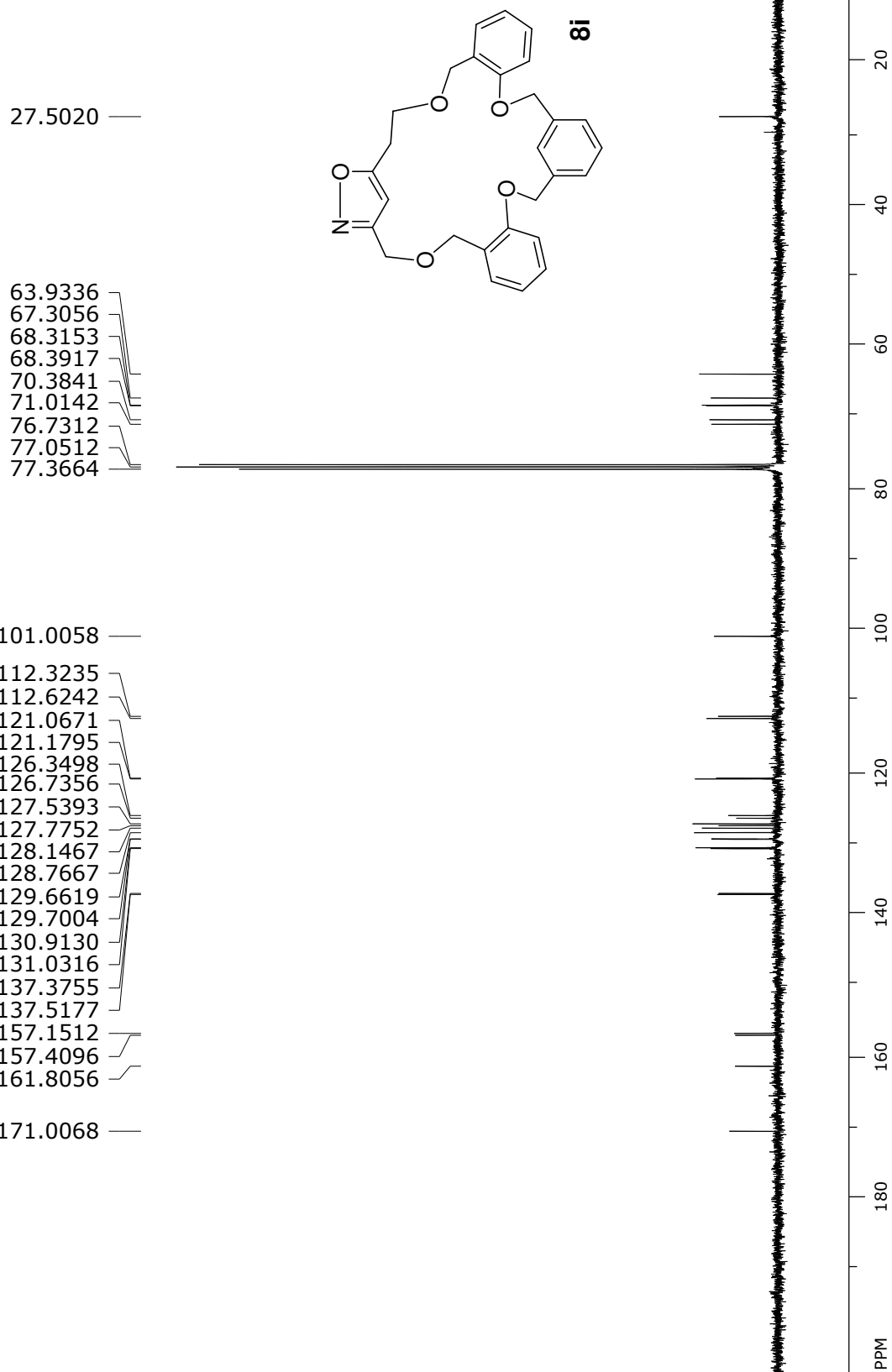
SpinWorks 3: NM1128a Proton test





SpinWorks 3: NM1128a Proton test

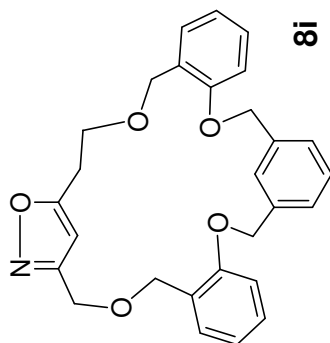




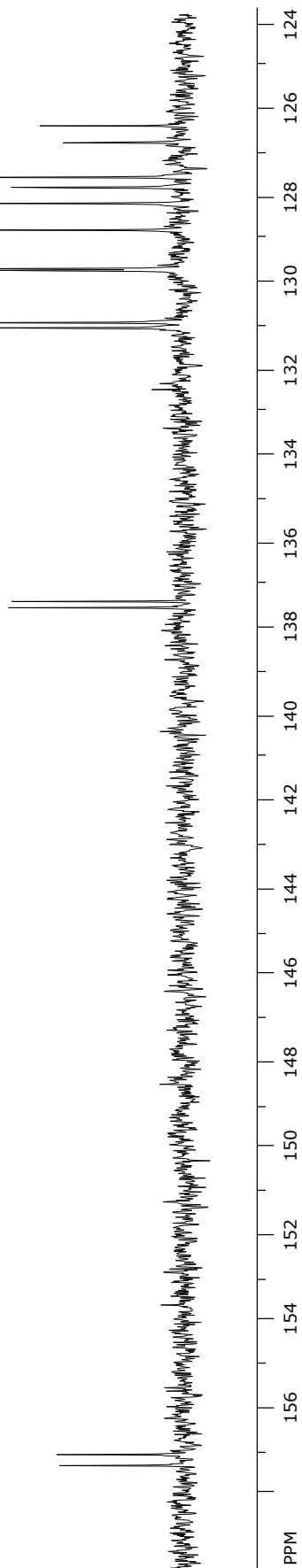
SpinWorks 3: NM1128a Carbon test

157.1512
157.4096

126.3498
126.7356
127.5393
127.7752
128.1467
128.7667
129.6619
129.7004
130.9130
131.0316



8i

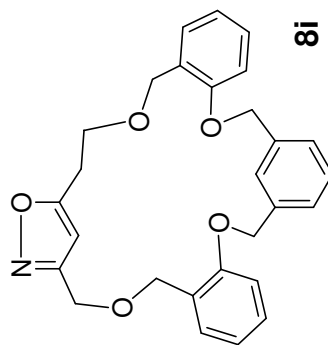


SpinWorks 3: NM1128a Carbon test

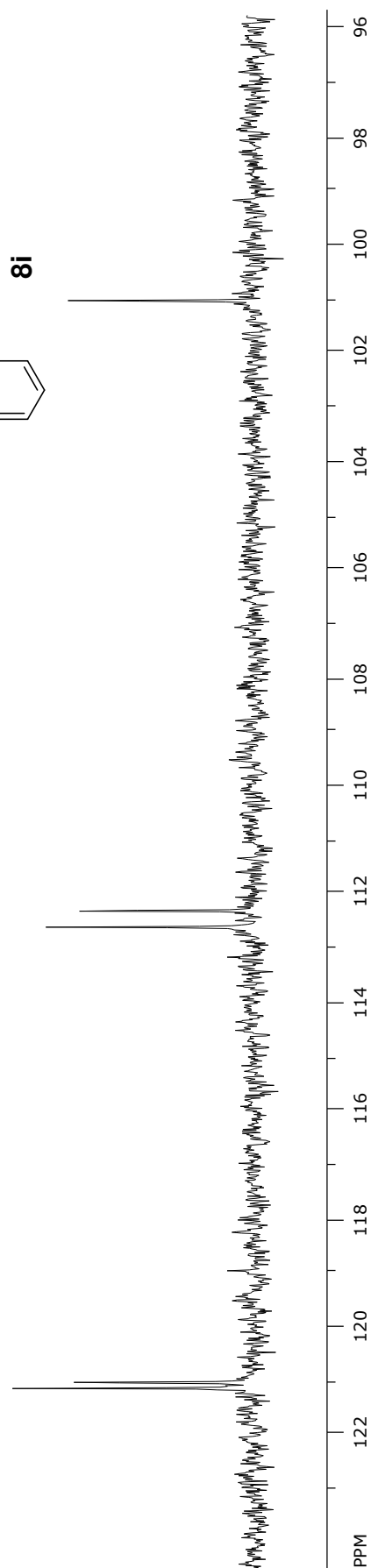
121.0671
121.1795

112.3235
112.6242

101.0058



8i



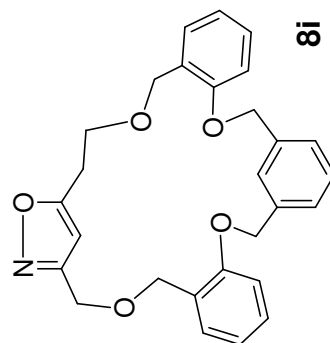
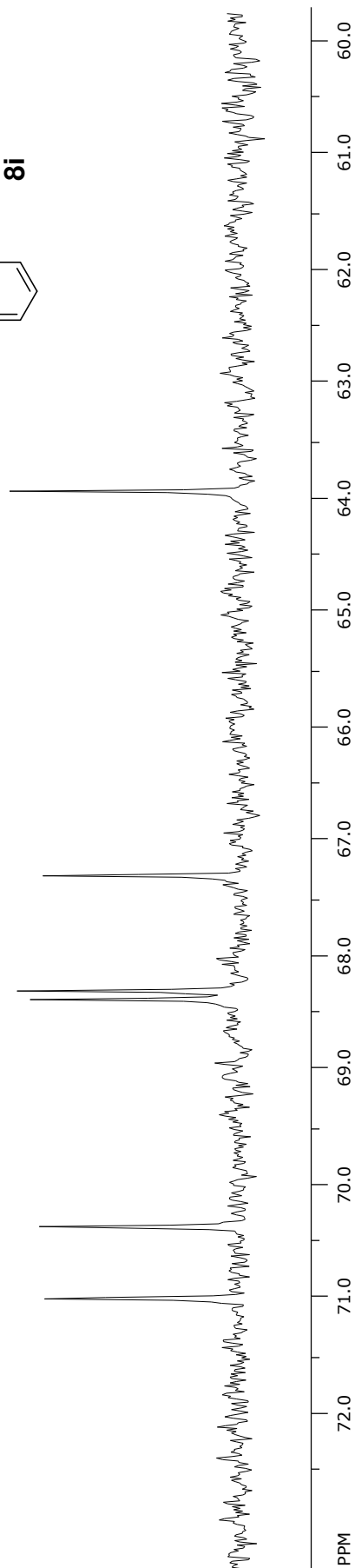
63.9336 —

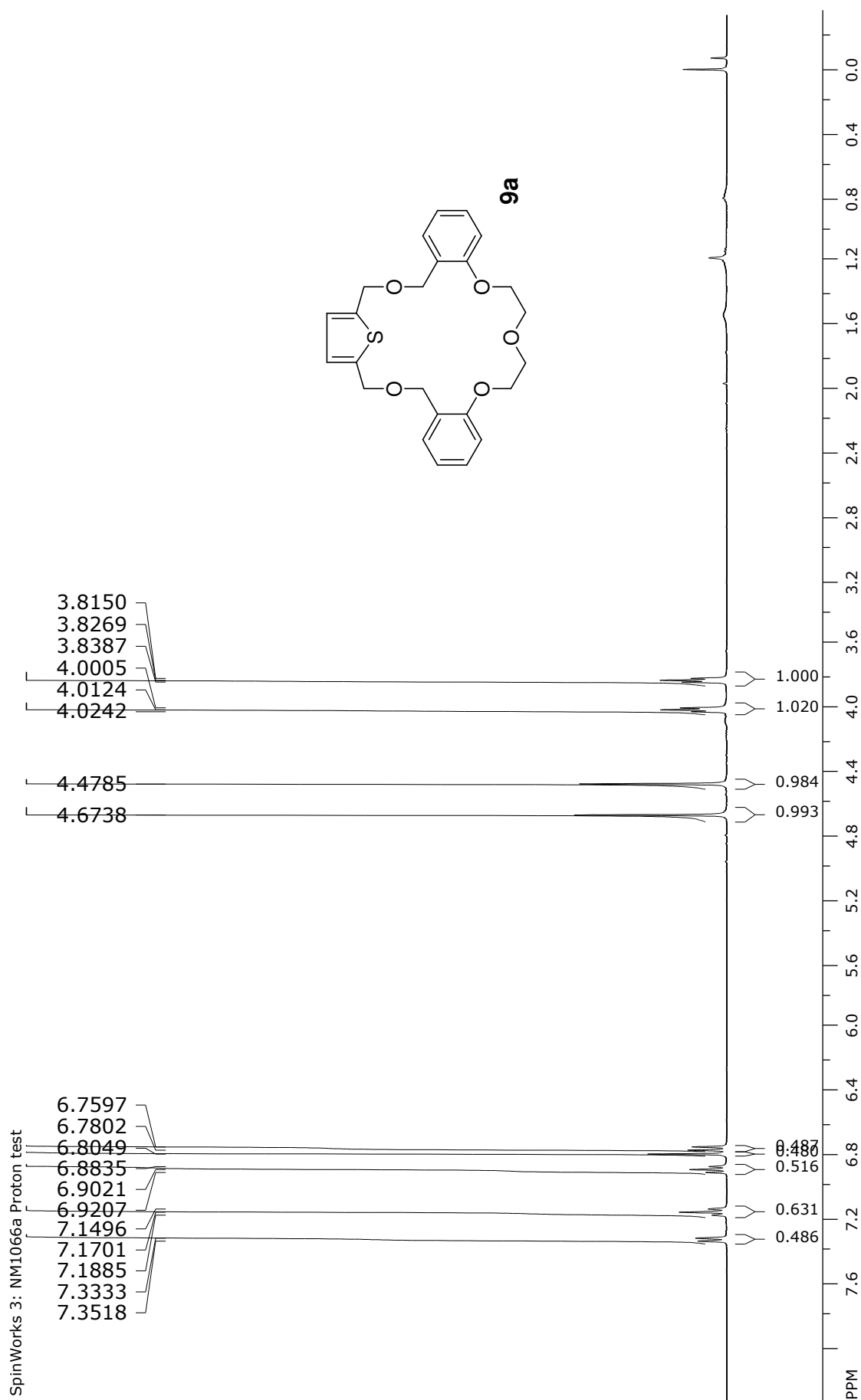
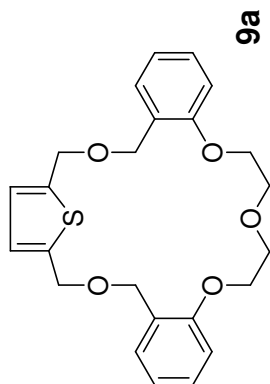
67.3056 —

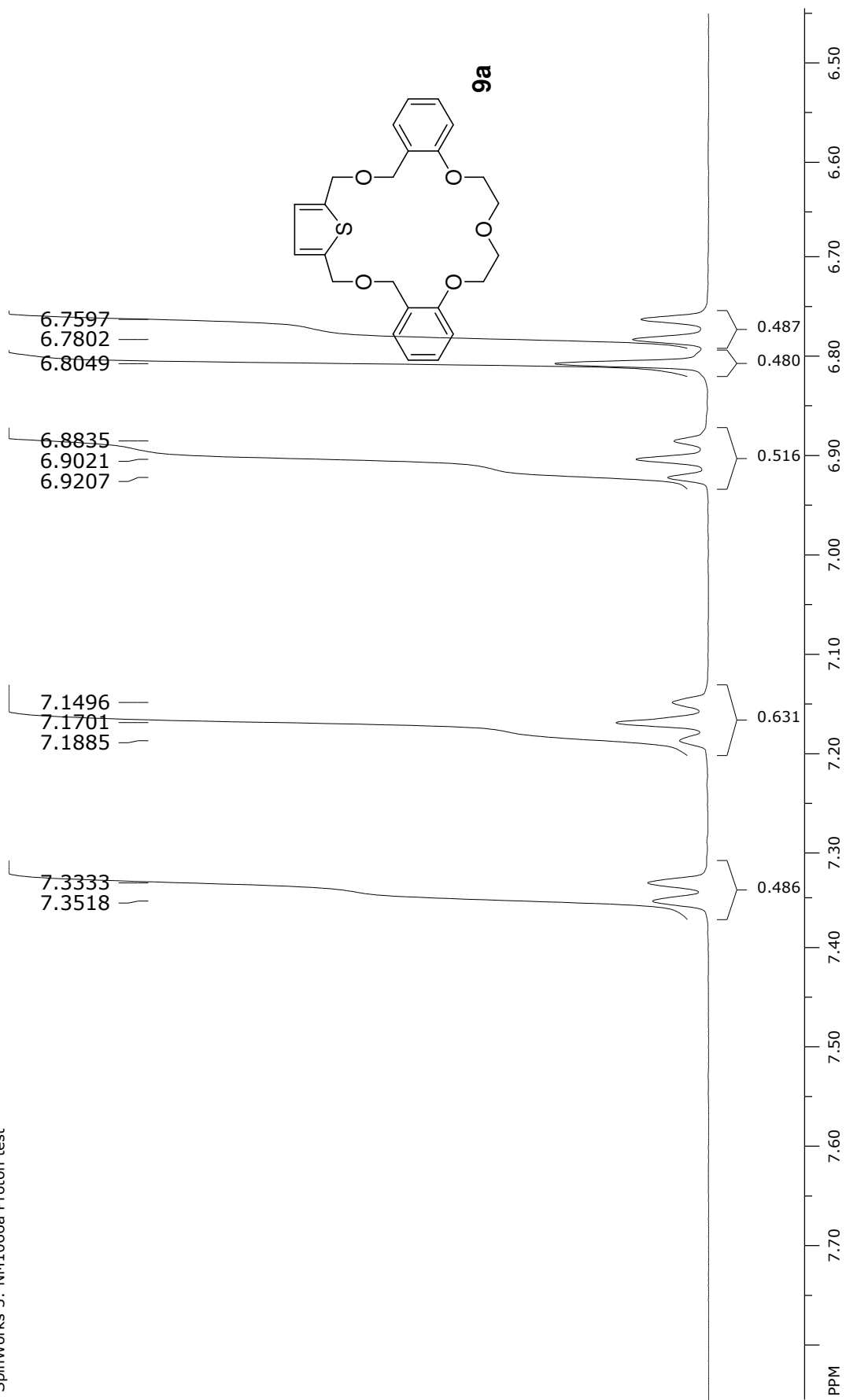
68.3153 —
68.3917 —

70.3841 —

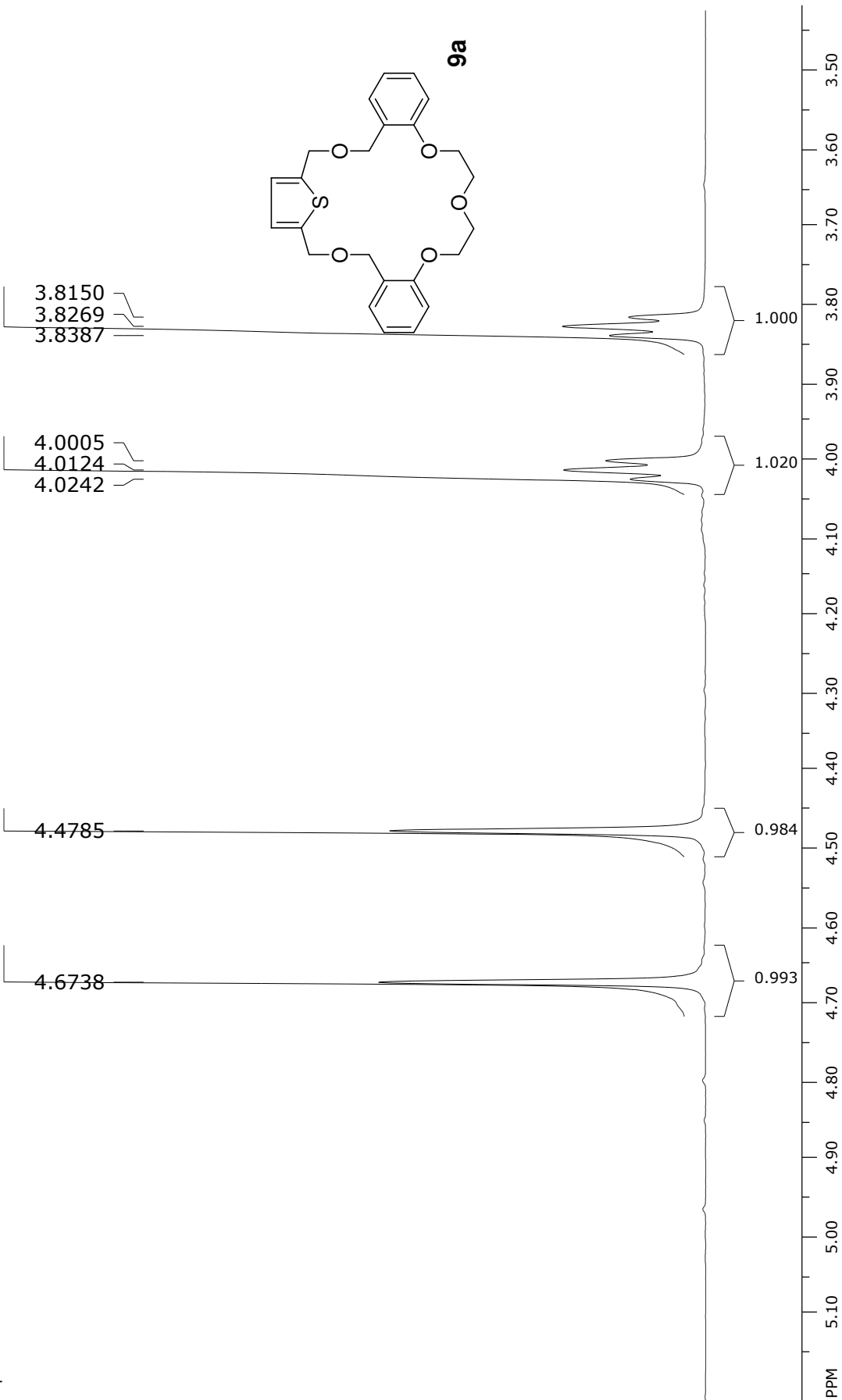
71.0142 —

**8i**

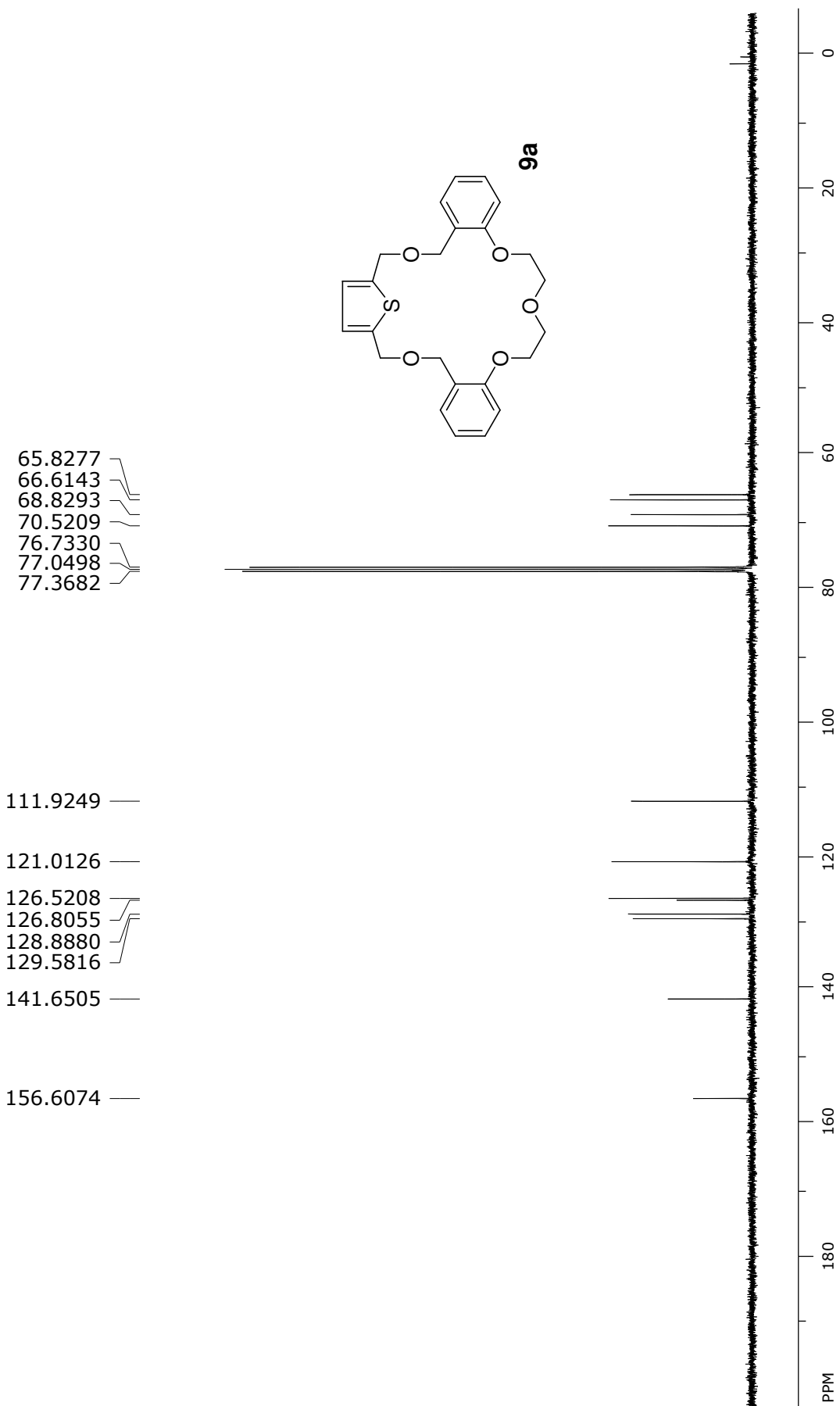




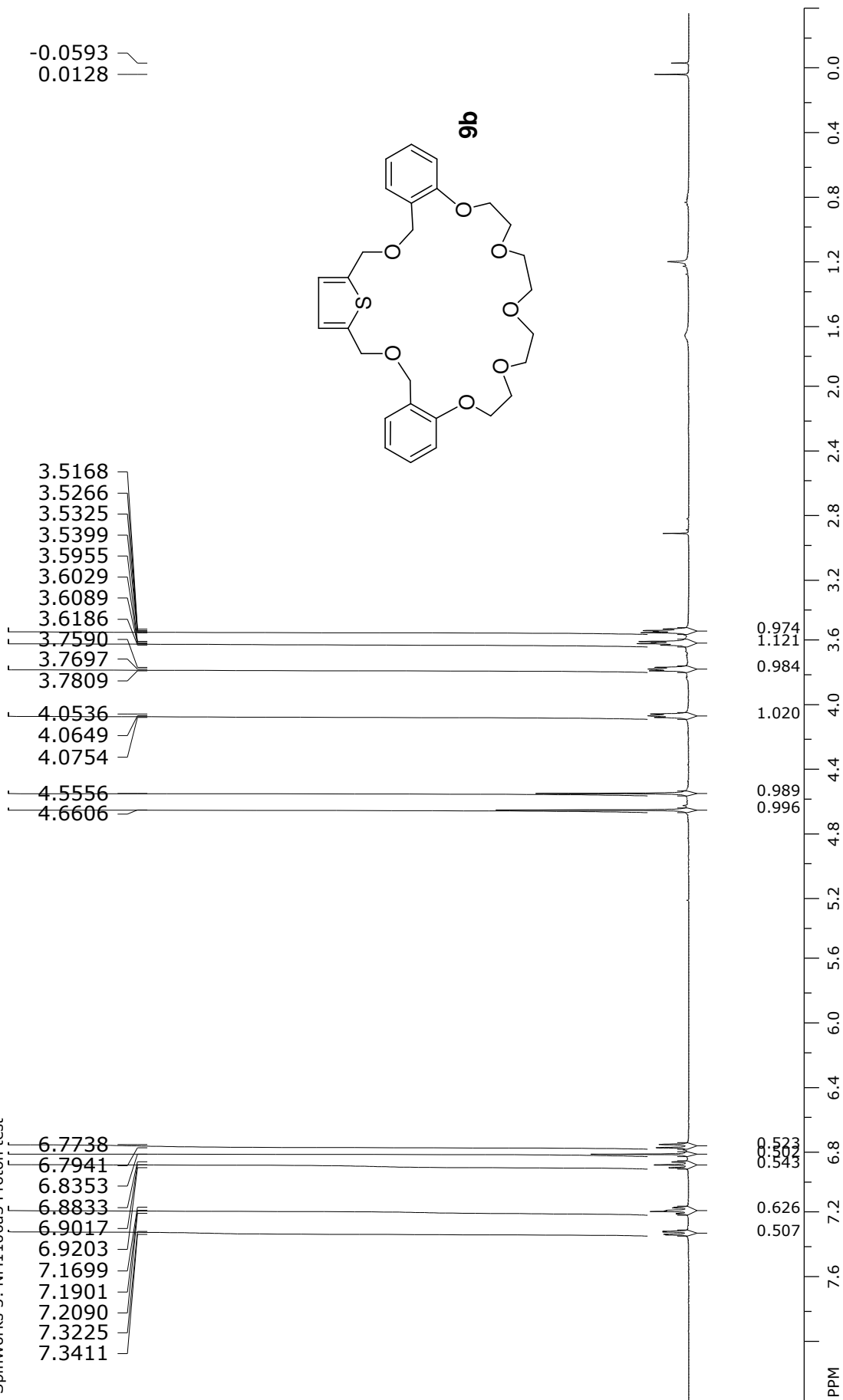
SpinWorks 3: NM1066a Proton test



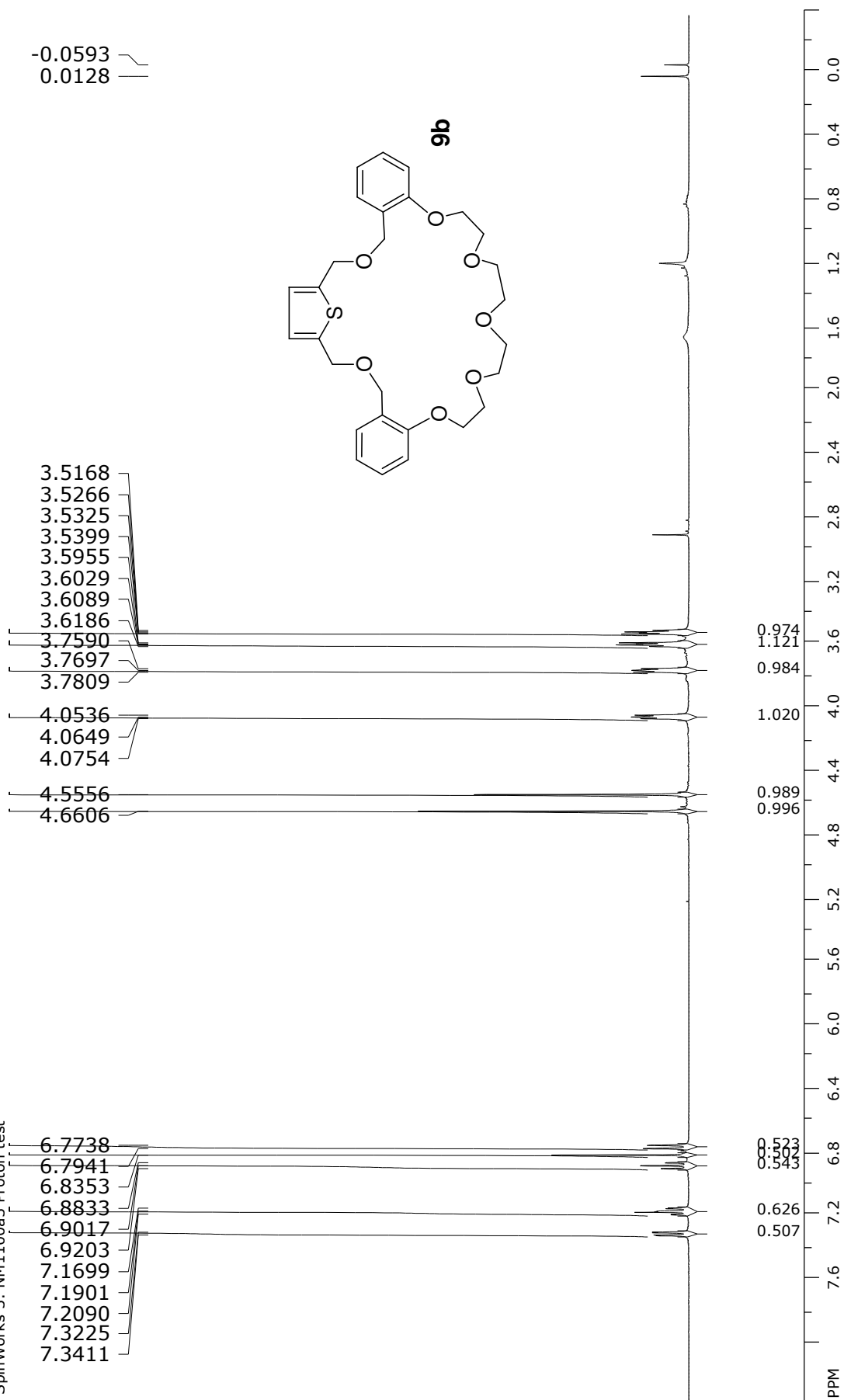
SpinWorks 3: NM1066a Carbon test



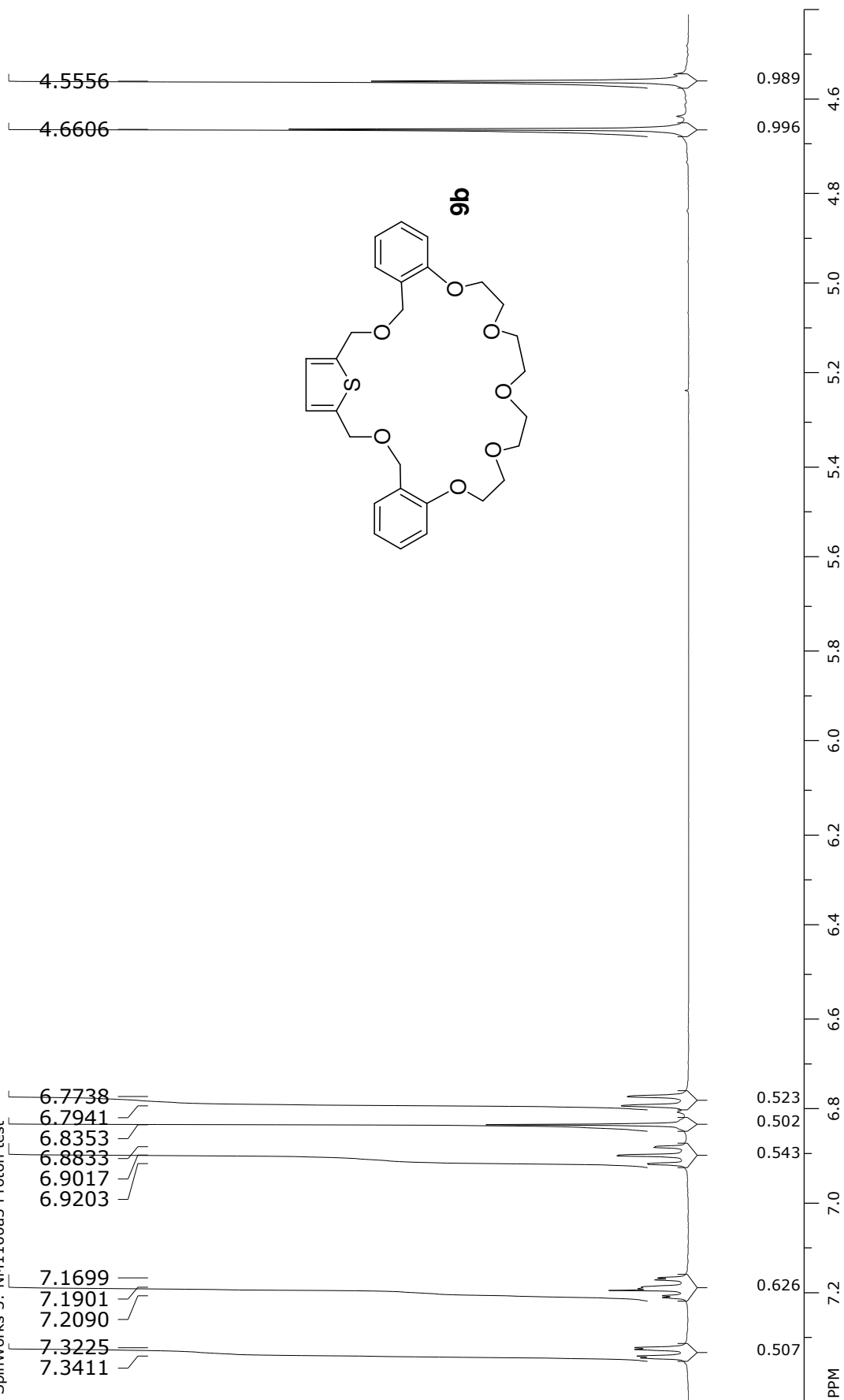
SpinWorks 3: NM1100a3 Proton test

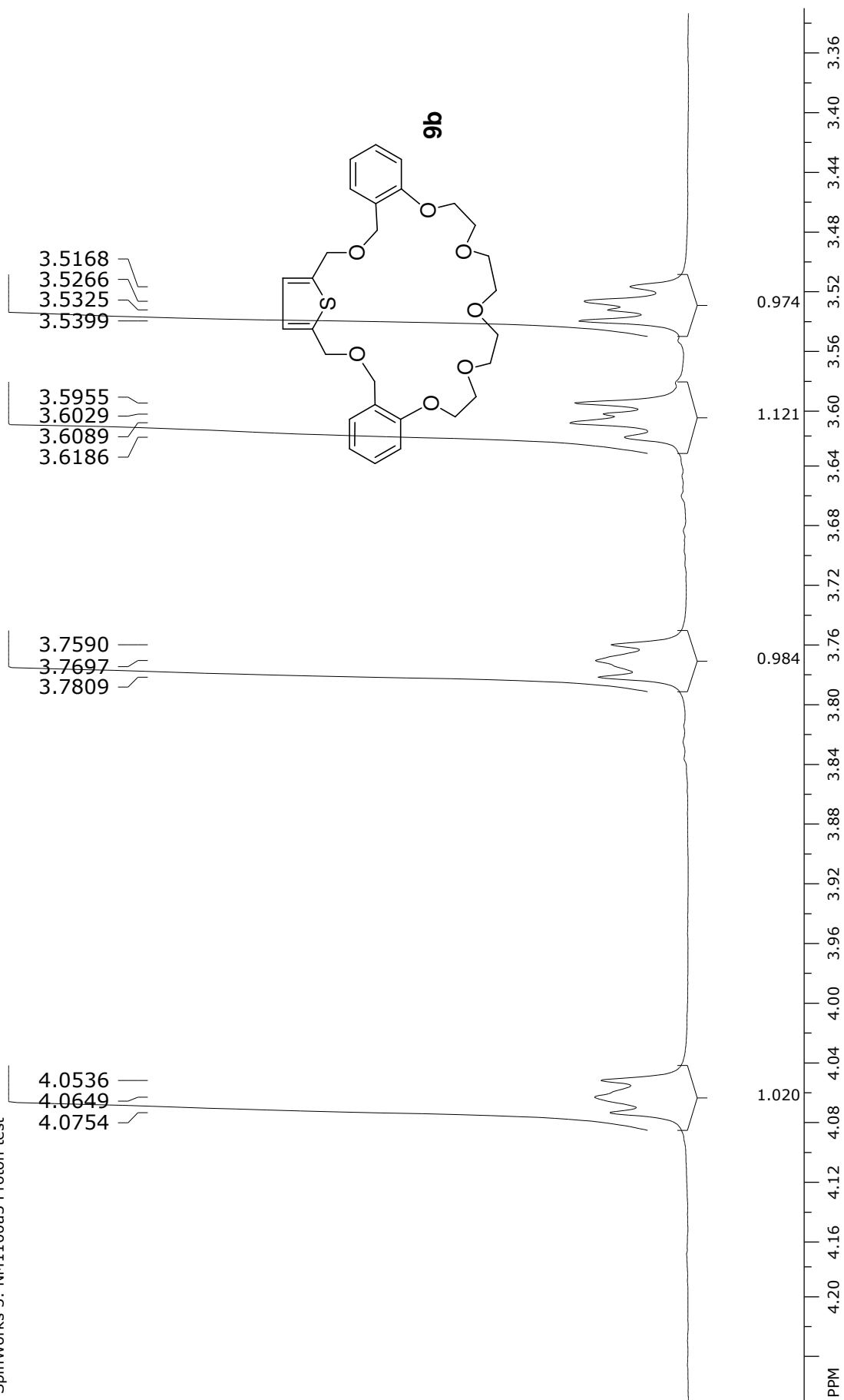


SpinWorks 3: NM1100a3 Proton test

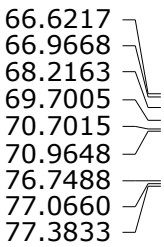


SpinWorks 3: NM1100a3 Proton test

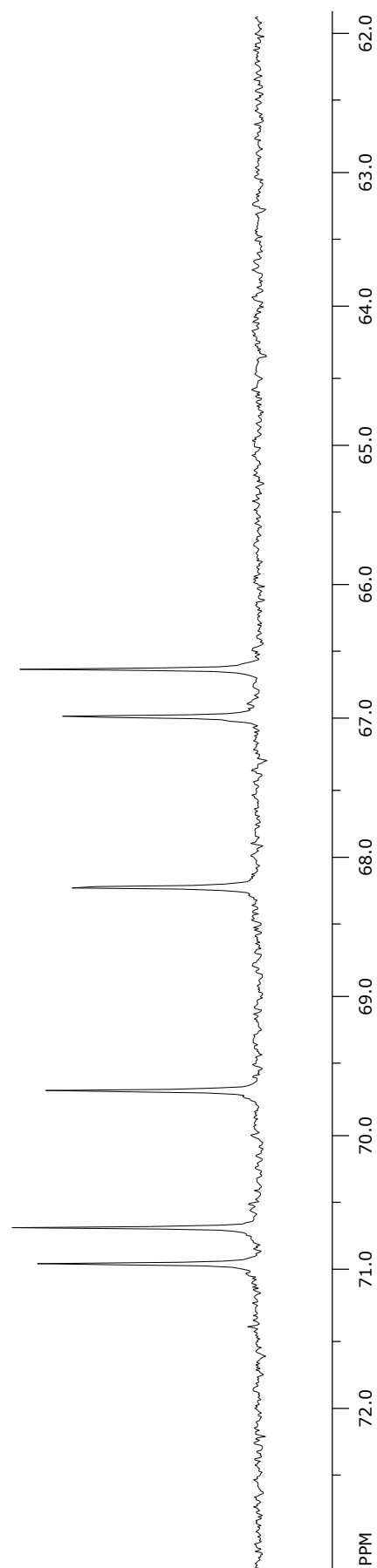
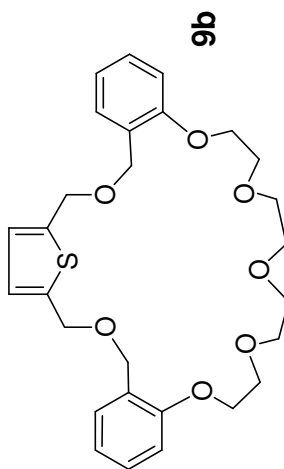
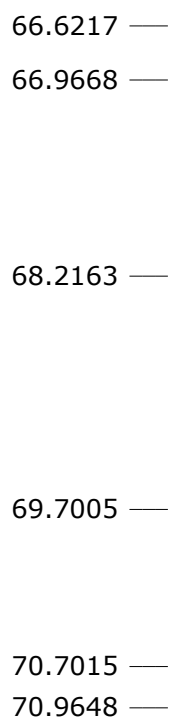


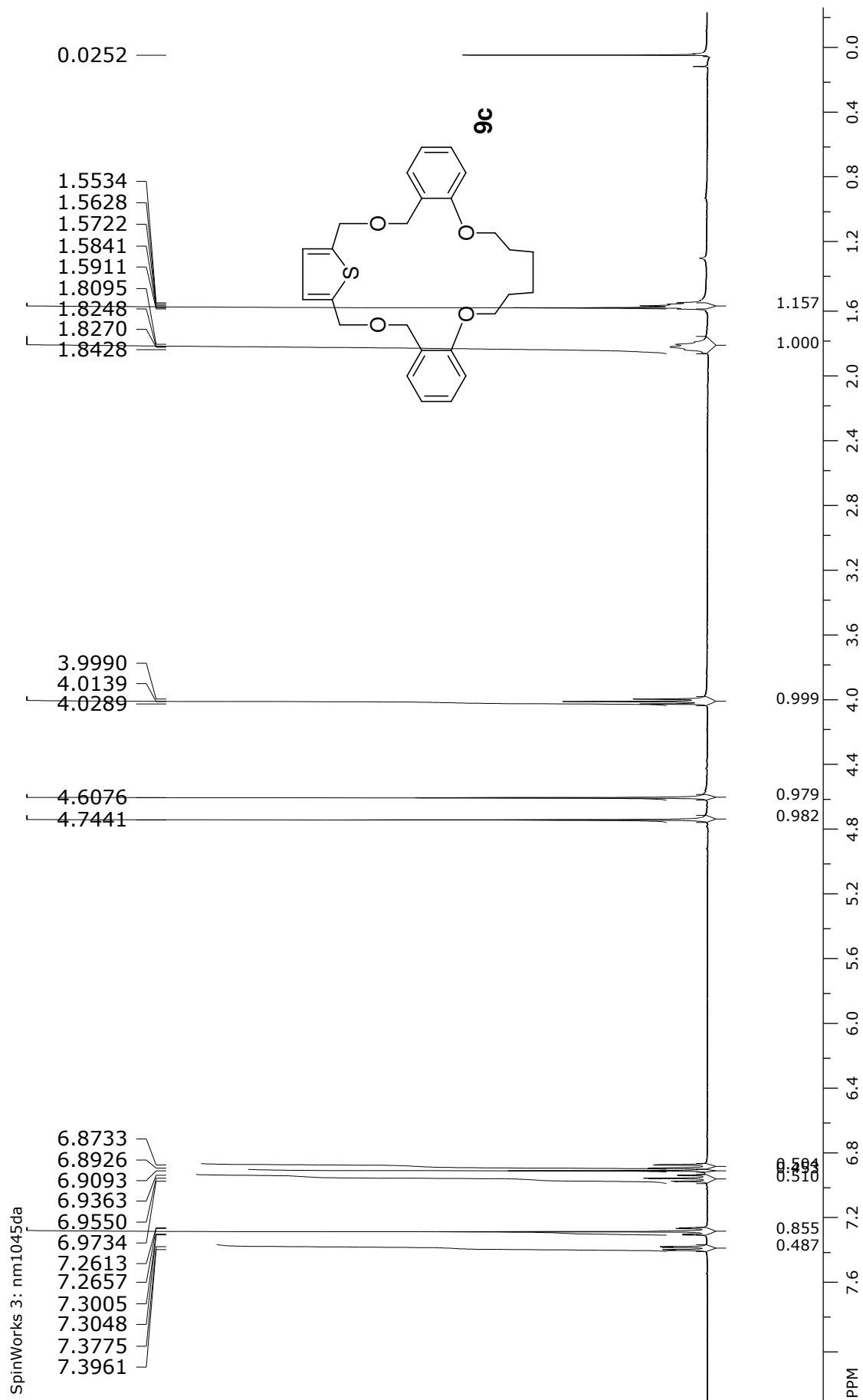


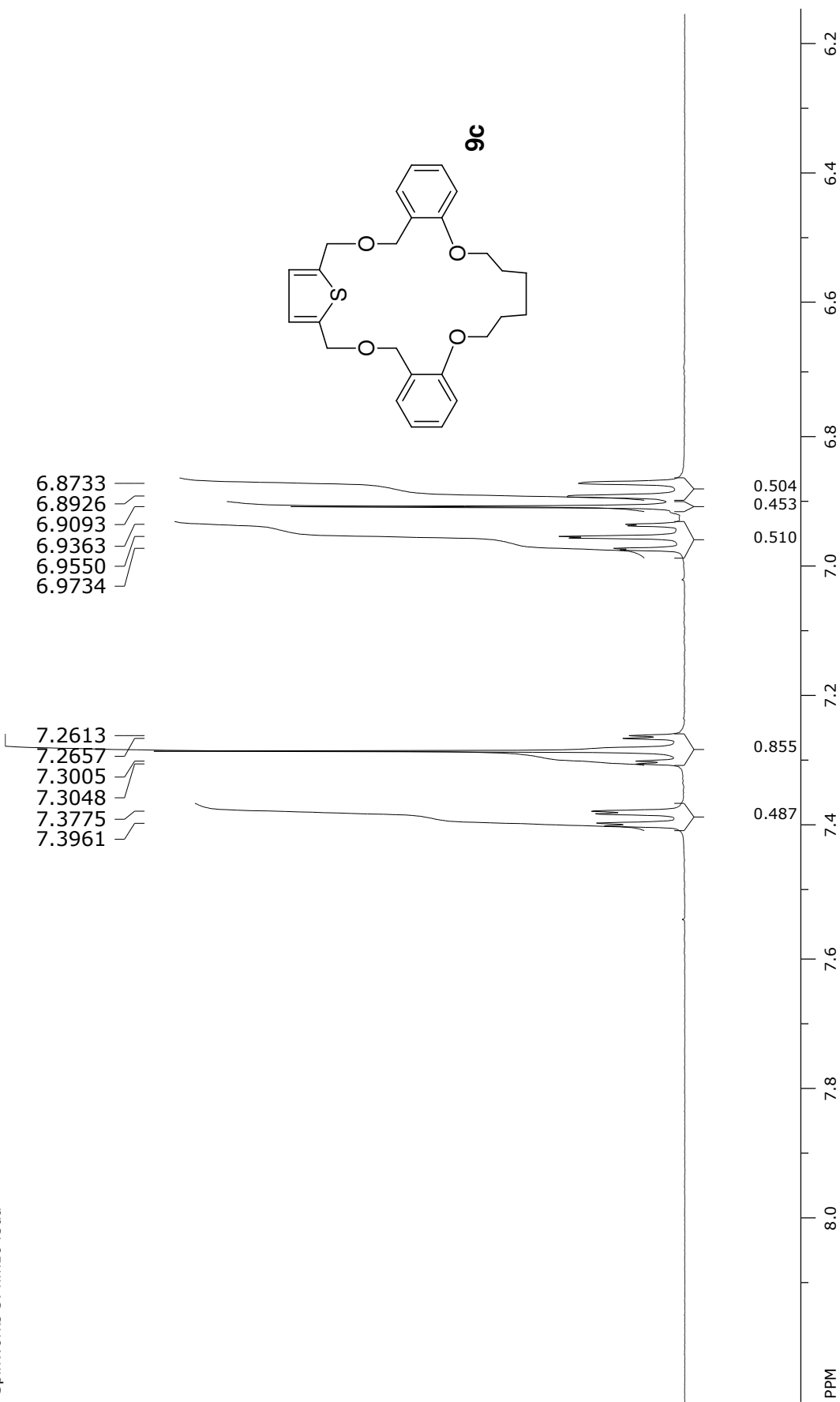
SpinWorks 3: NM1100a3 carbon test

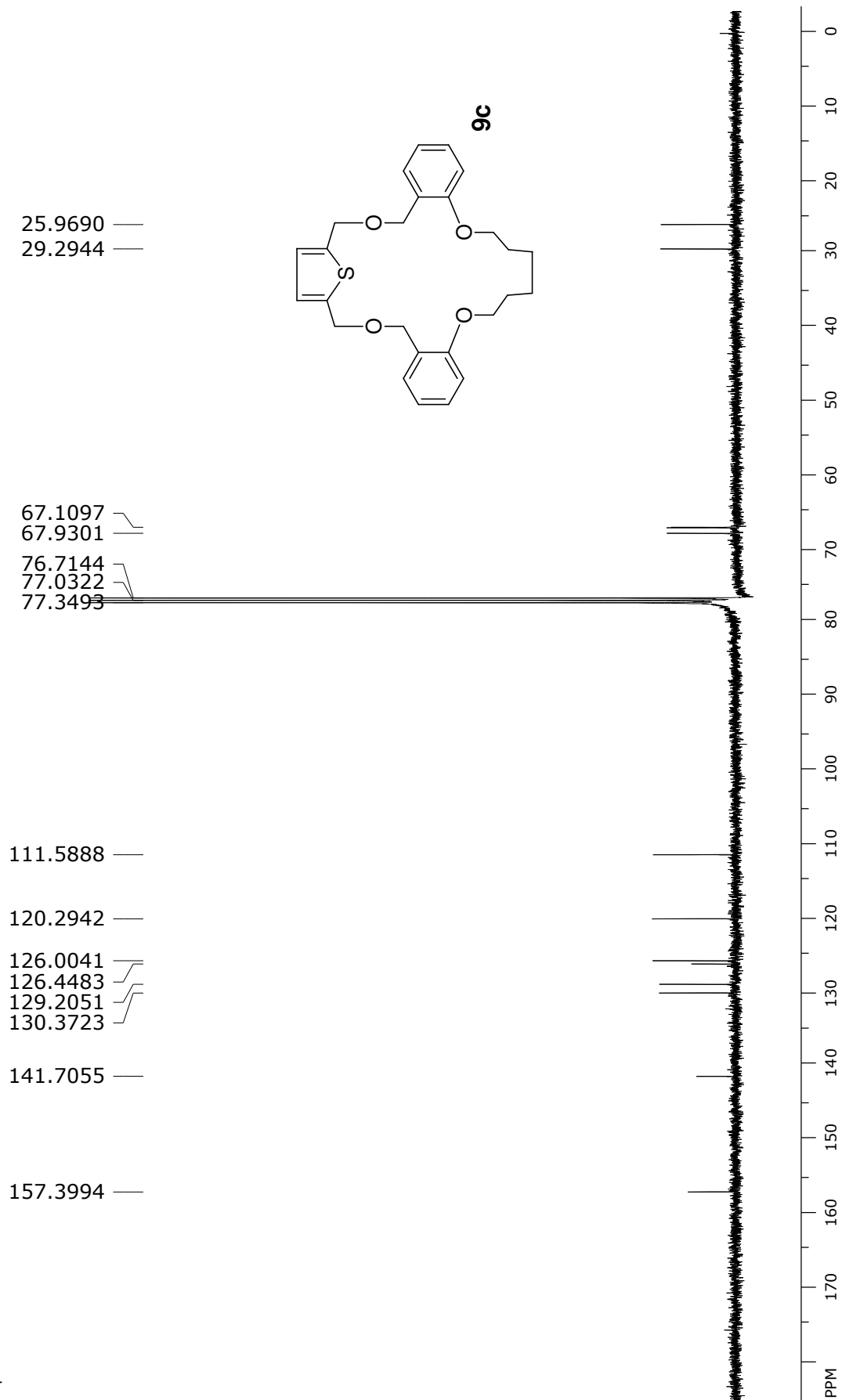


SpinWorks 3: NM1100a3 carbon test









SpinWorks 3: nm1045ra

130.3723 —

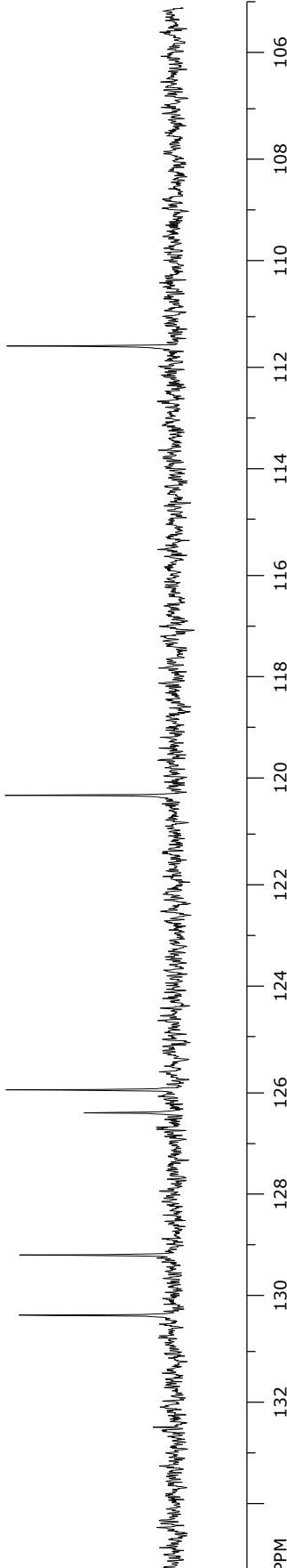
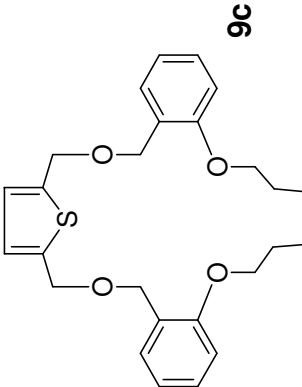
129.2051 —

126.0041 —

126.4483 —

120.2942 —

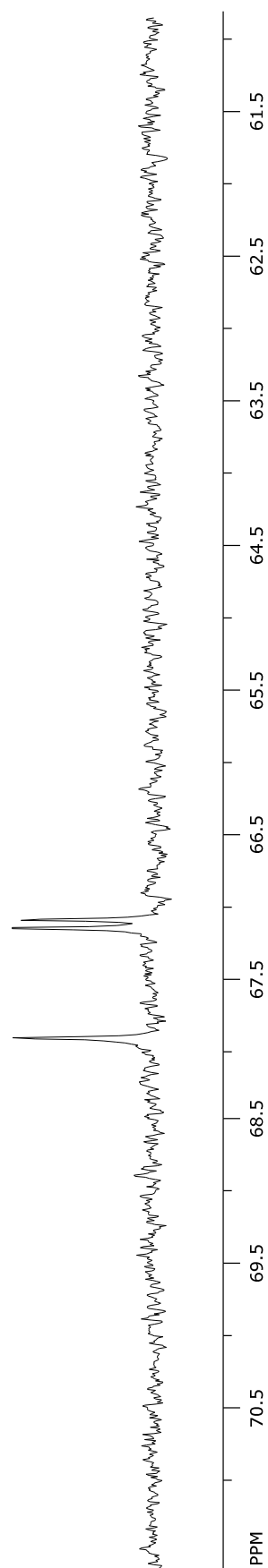
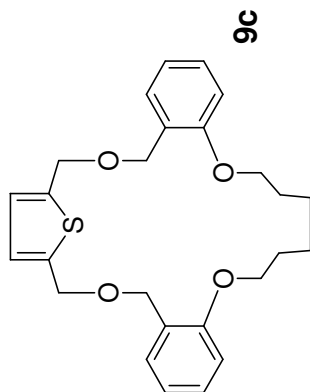
111.5888 —

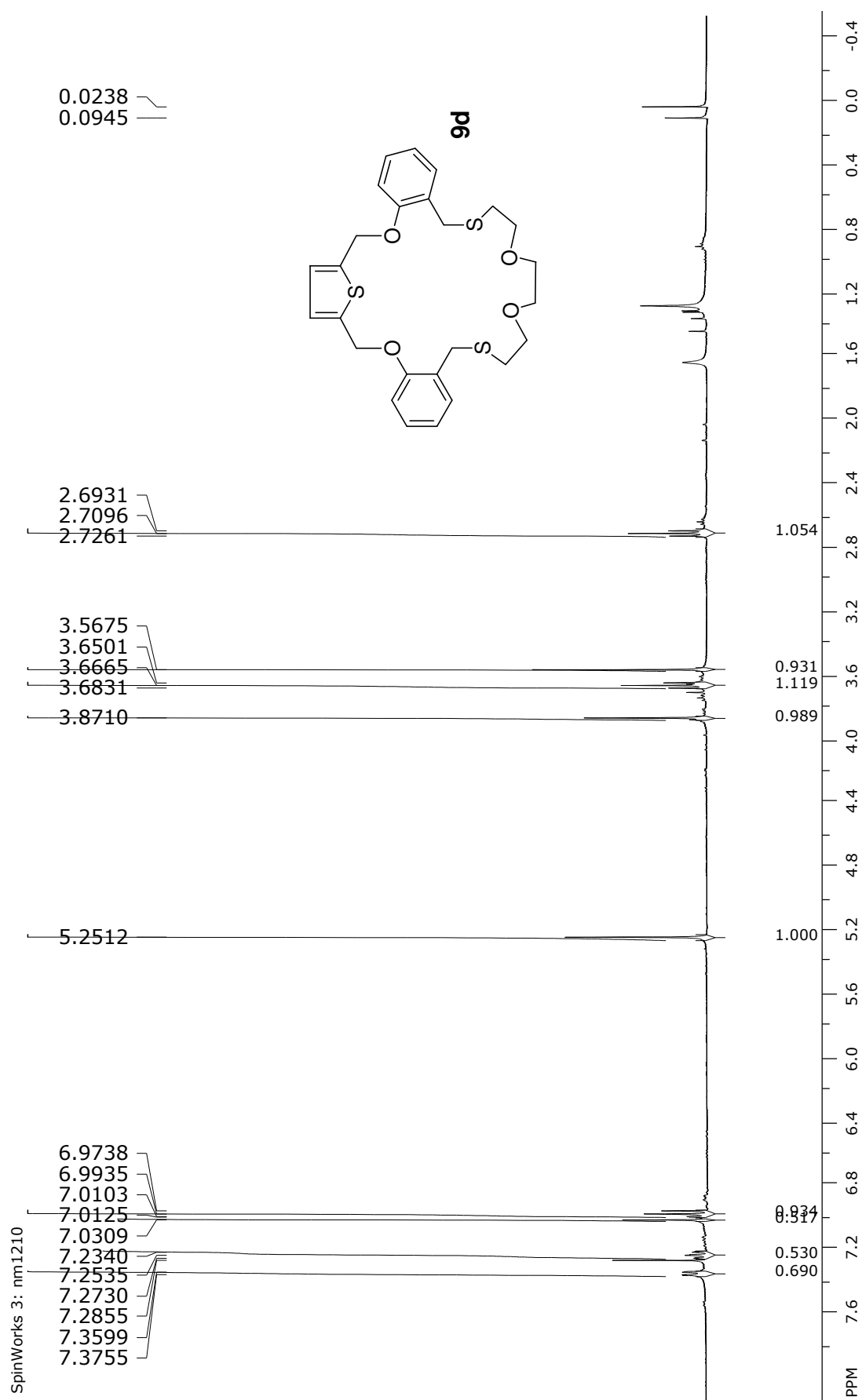


SpinWorks 3: nm1045ra

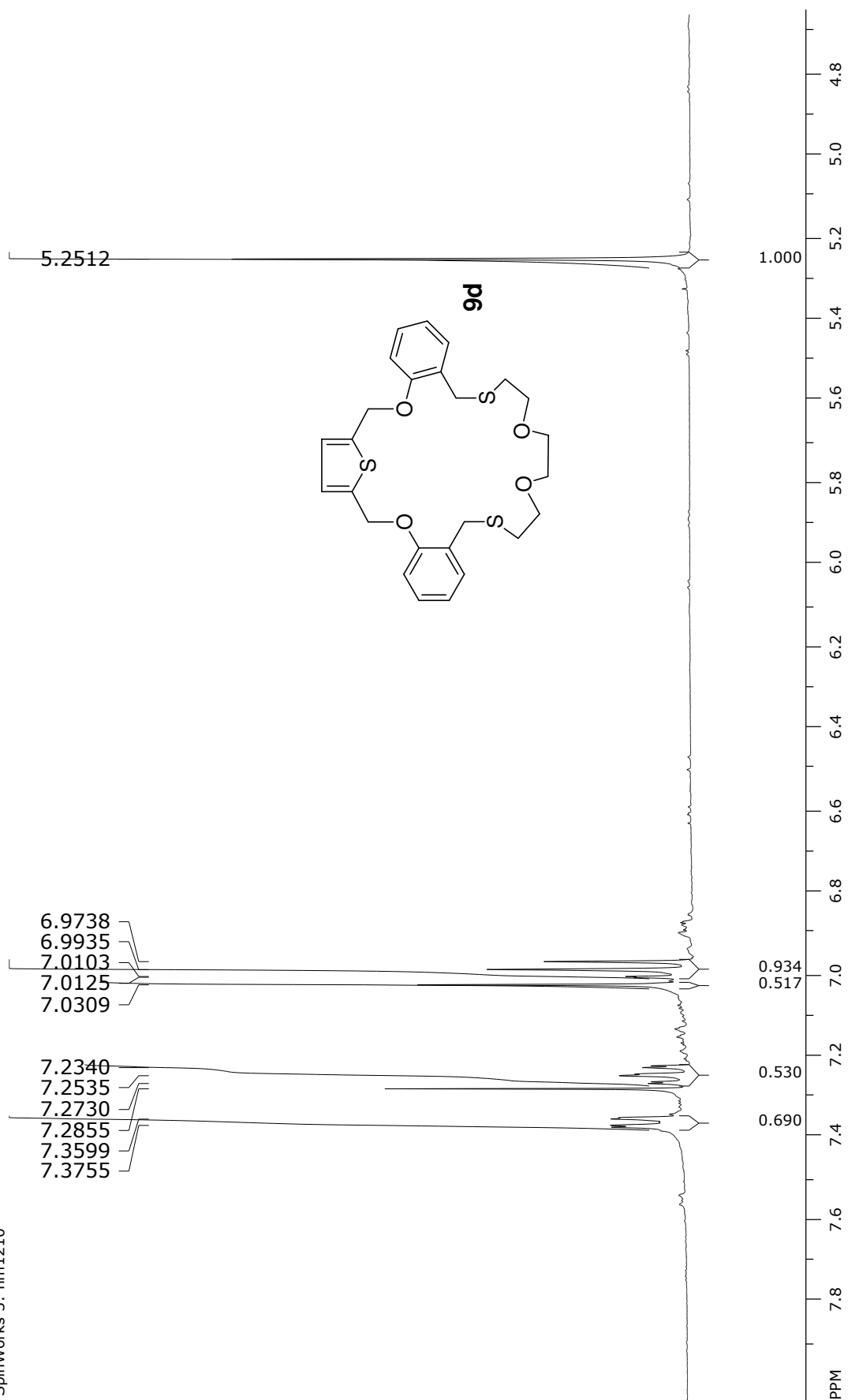
67.1097 —

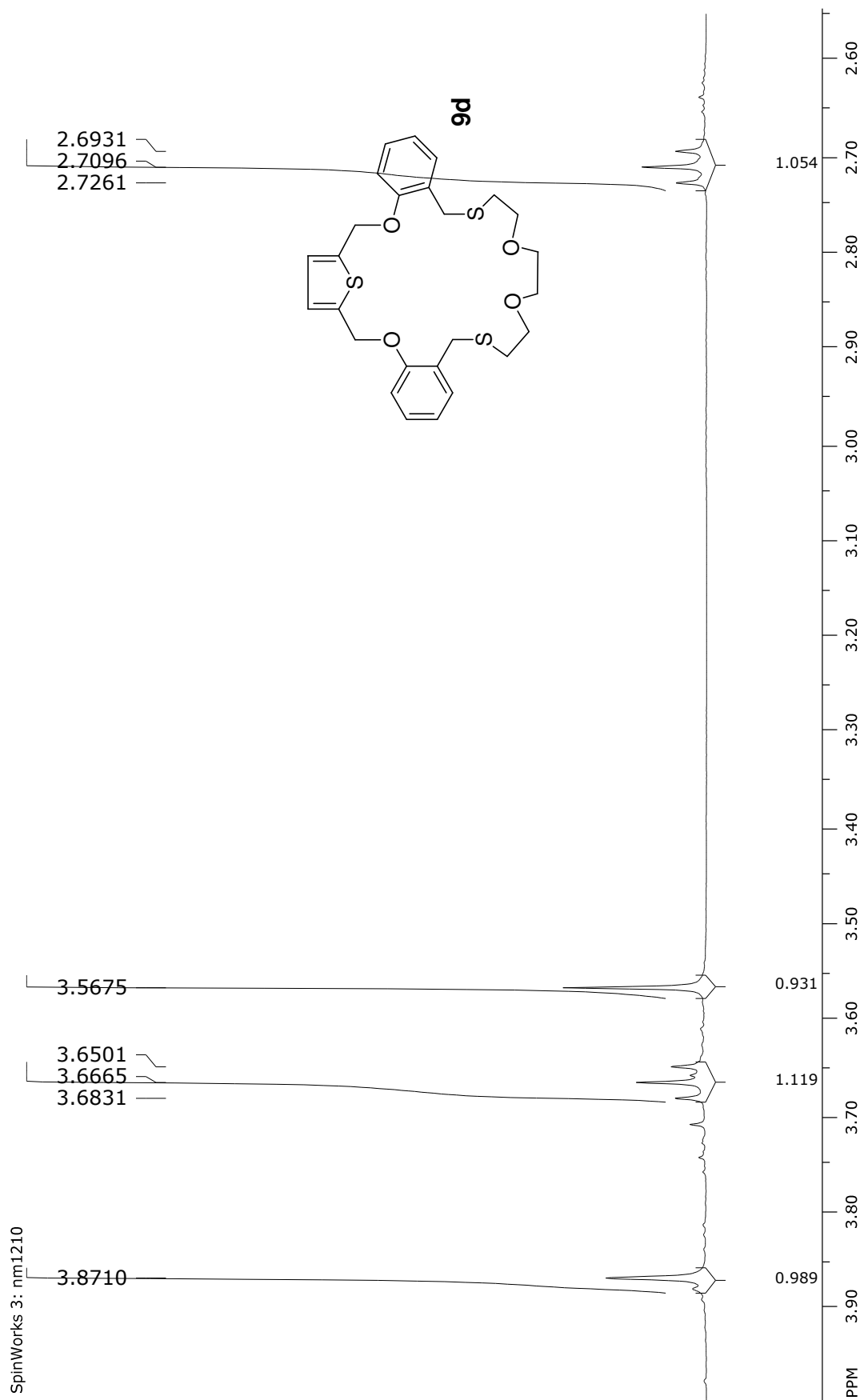
67.9301 —



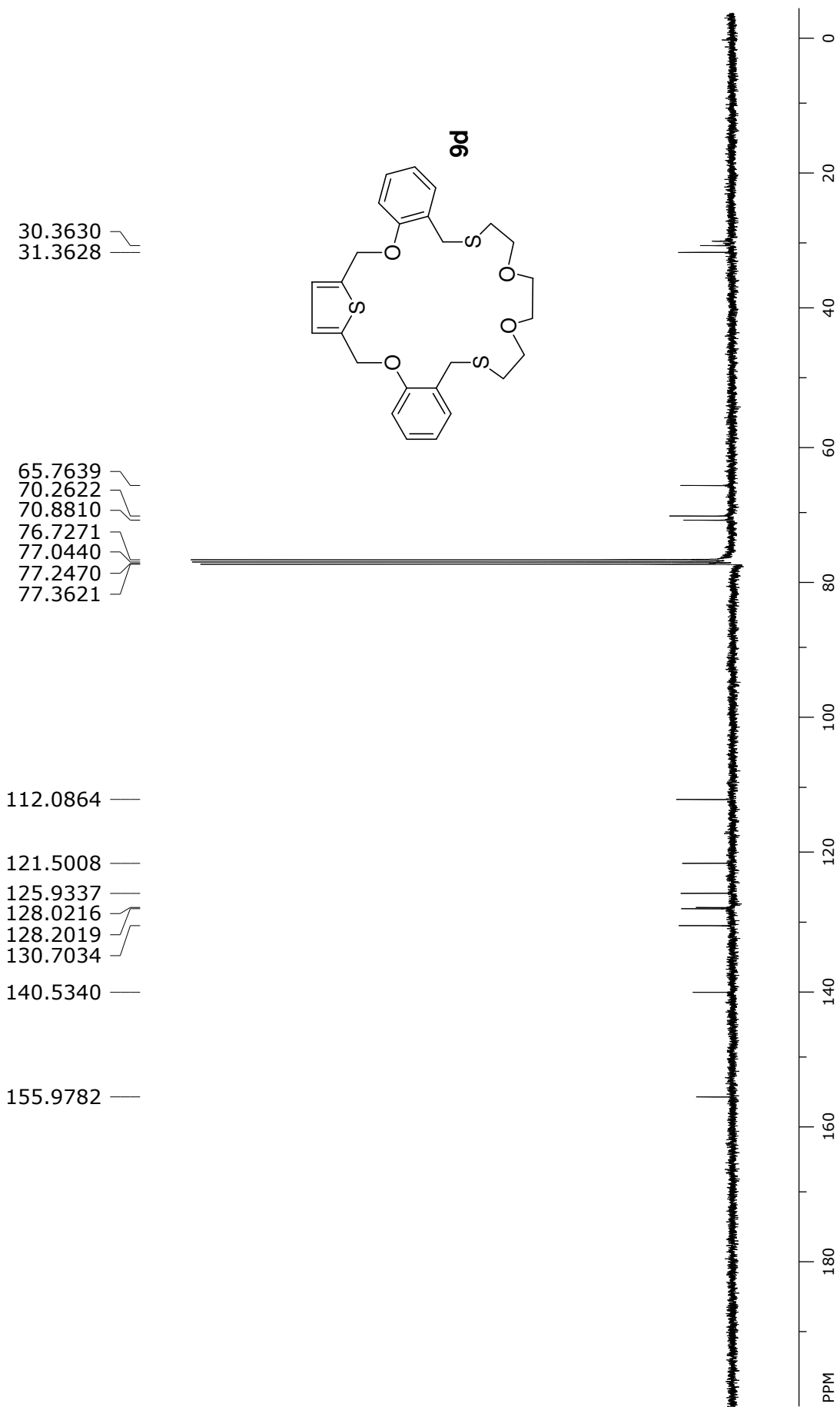


SpinWorks 3: nm1210

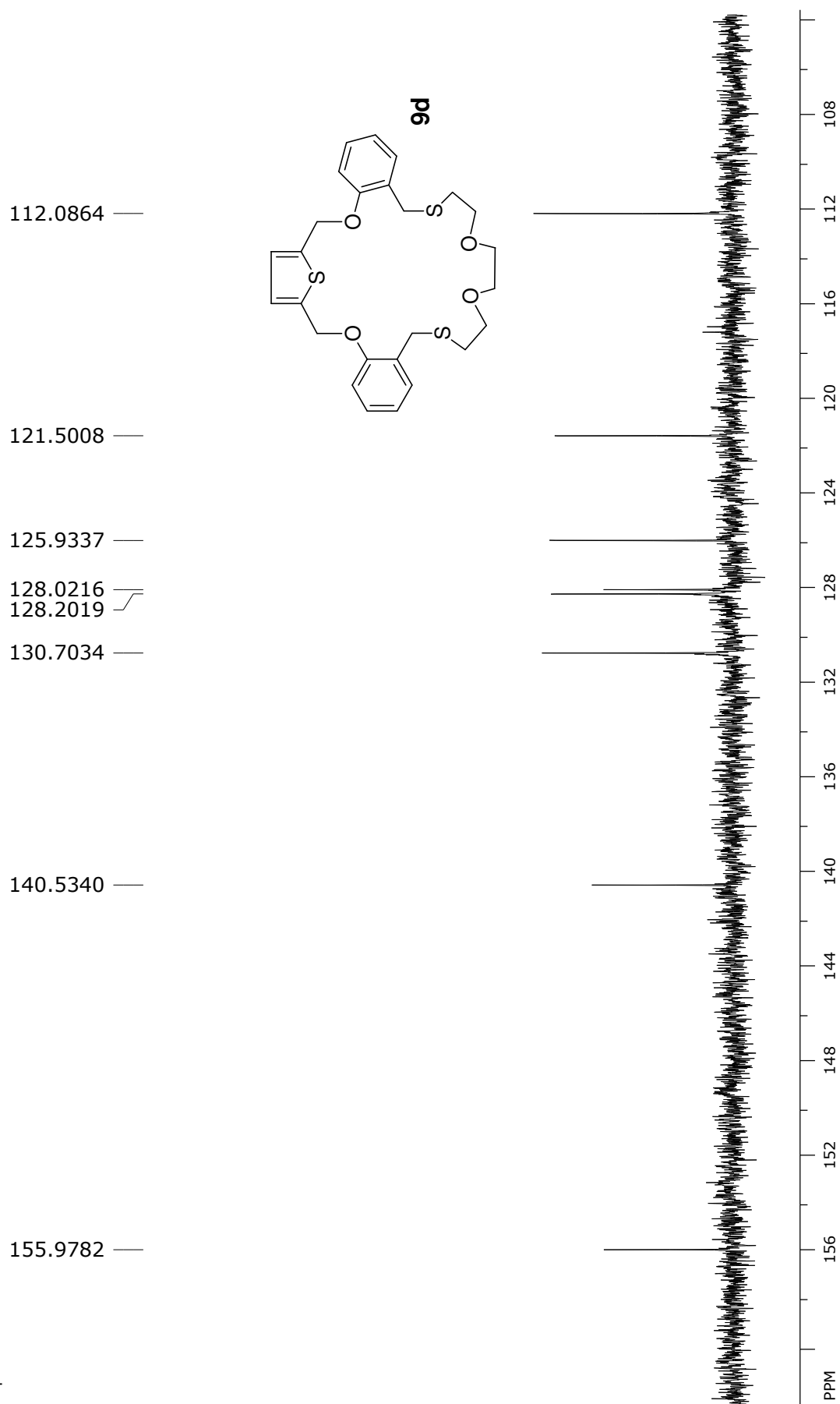




SpinWorks 3: nm1210



SpinWorks 3: nm1210



SpinWorks 3: nm1210

65.7639 —

70.2622 —

70.8810 —

30.3630 —

31.3628 —

