

Supplementary Materials

Figure S1. Compound **8**: ^1H -NMR, CD_3OD , 298 K, 600 MHz.

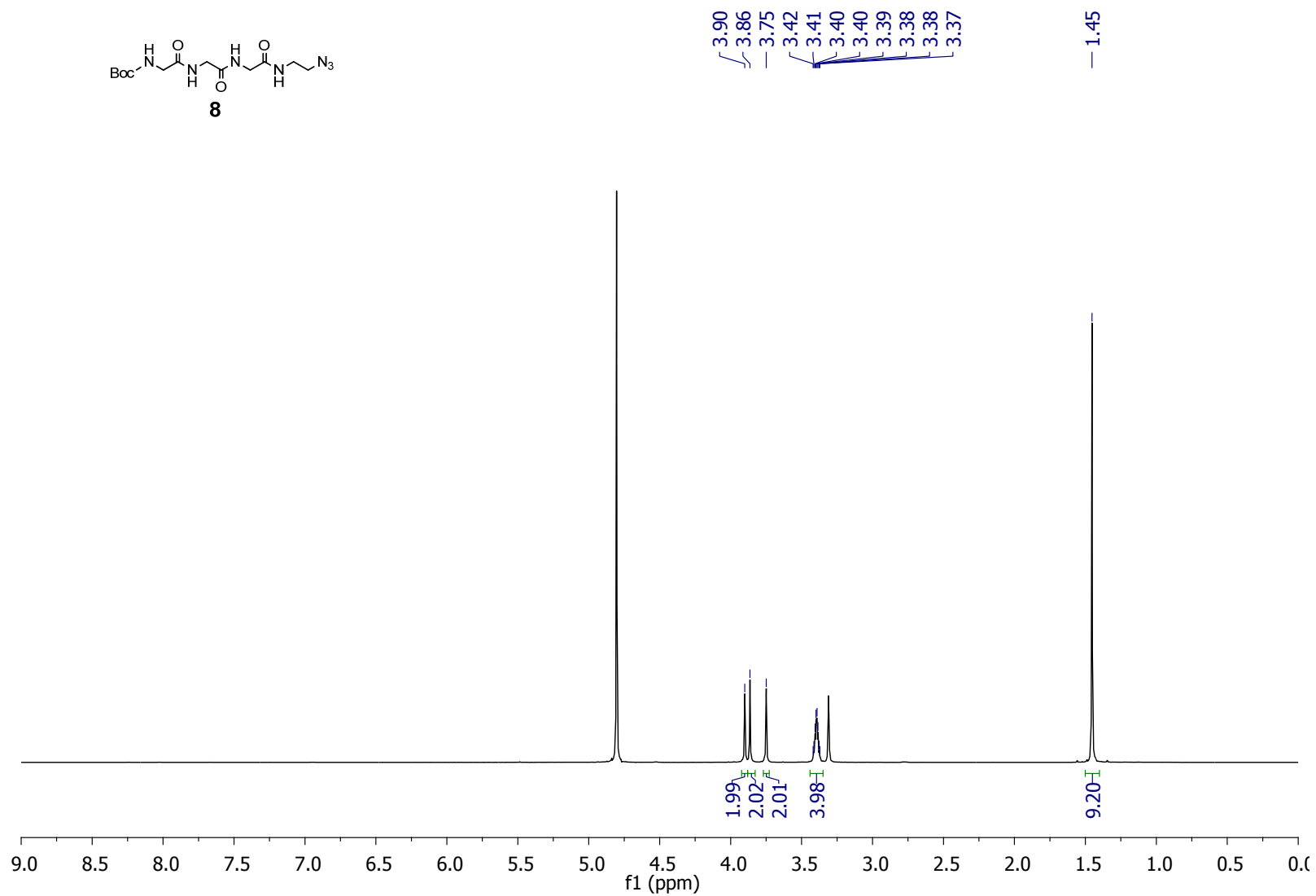
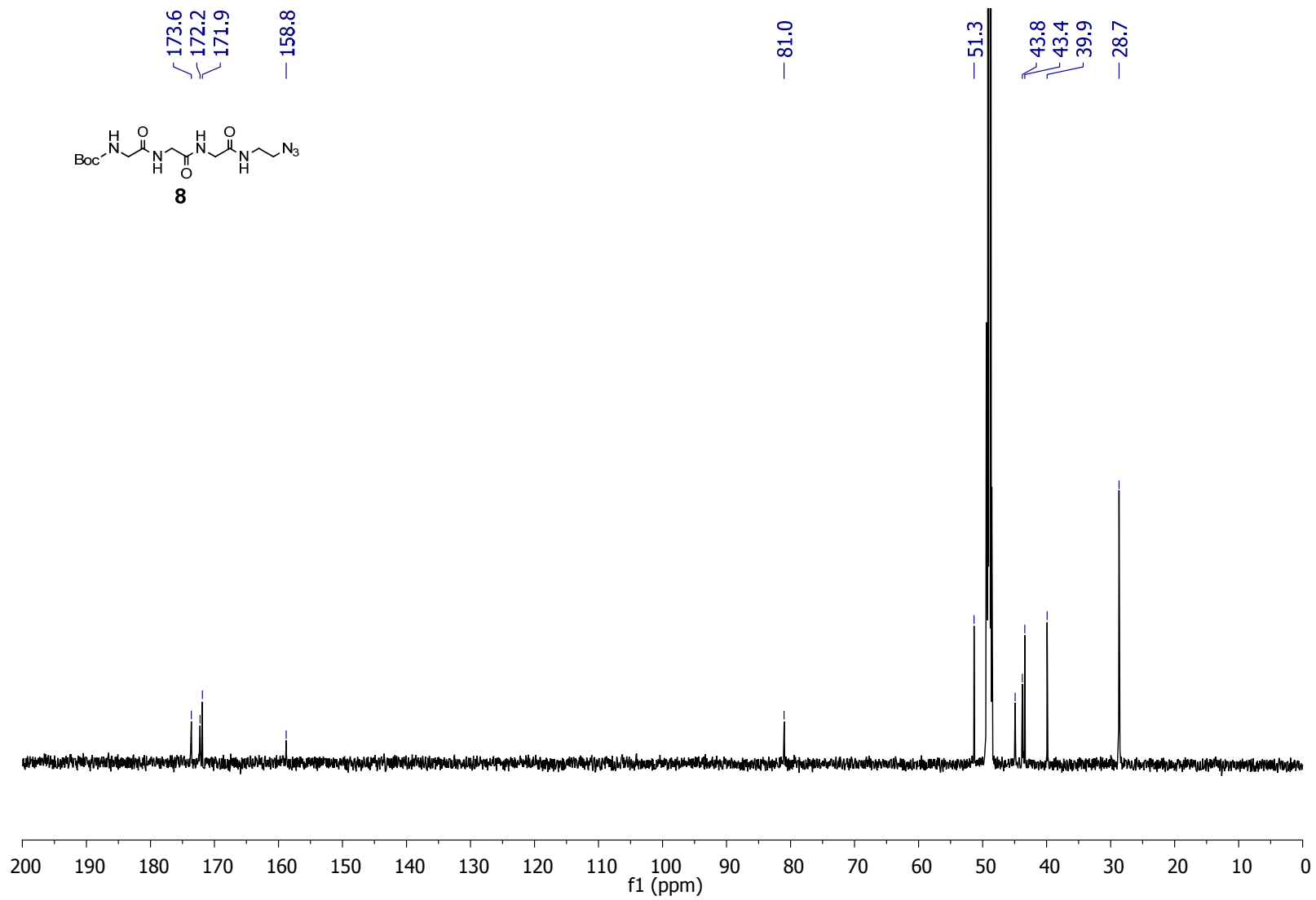


Figure S2. Compound 8: ^{13}C -NMR, CD_3OD , 298 K, 150 MHz.



9

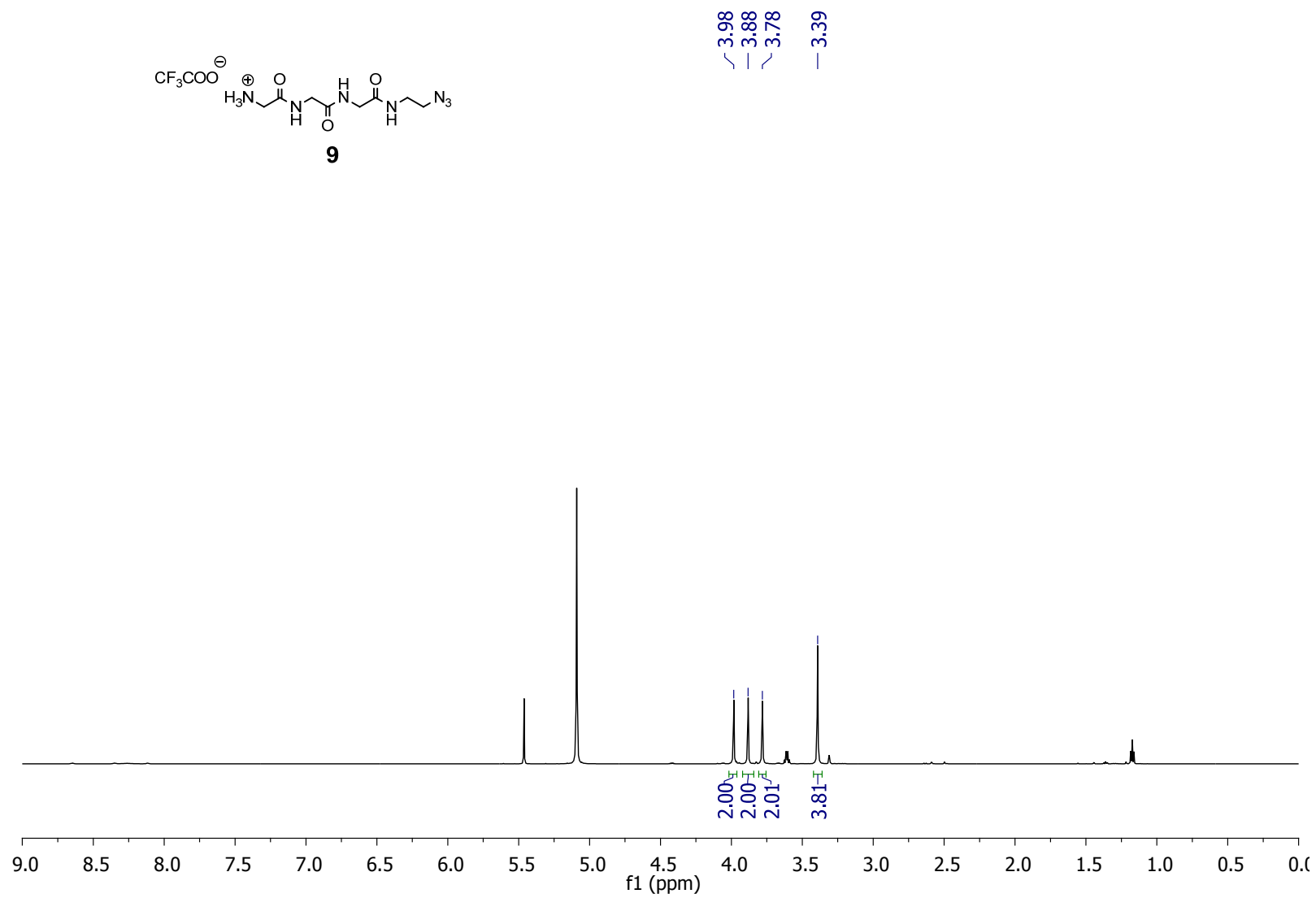


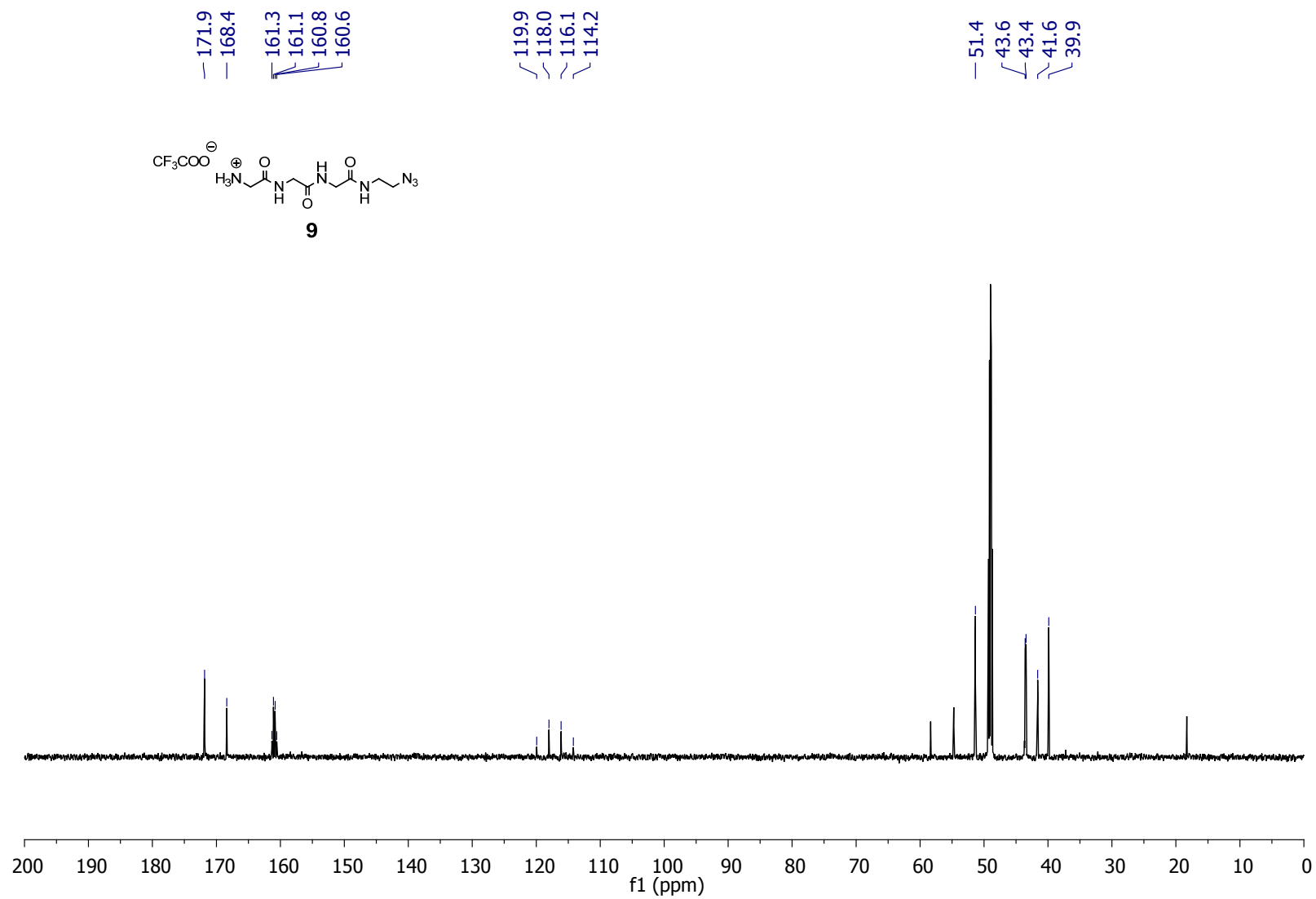
Figure S4. Compound **9**: ^{13}C -NMR, CD_3OD , 298 K, 150 MHz.

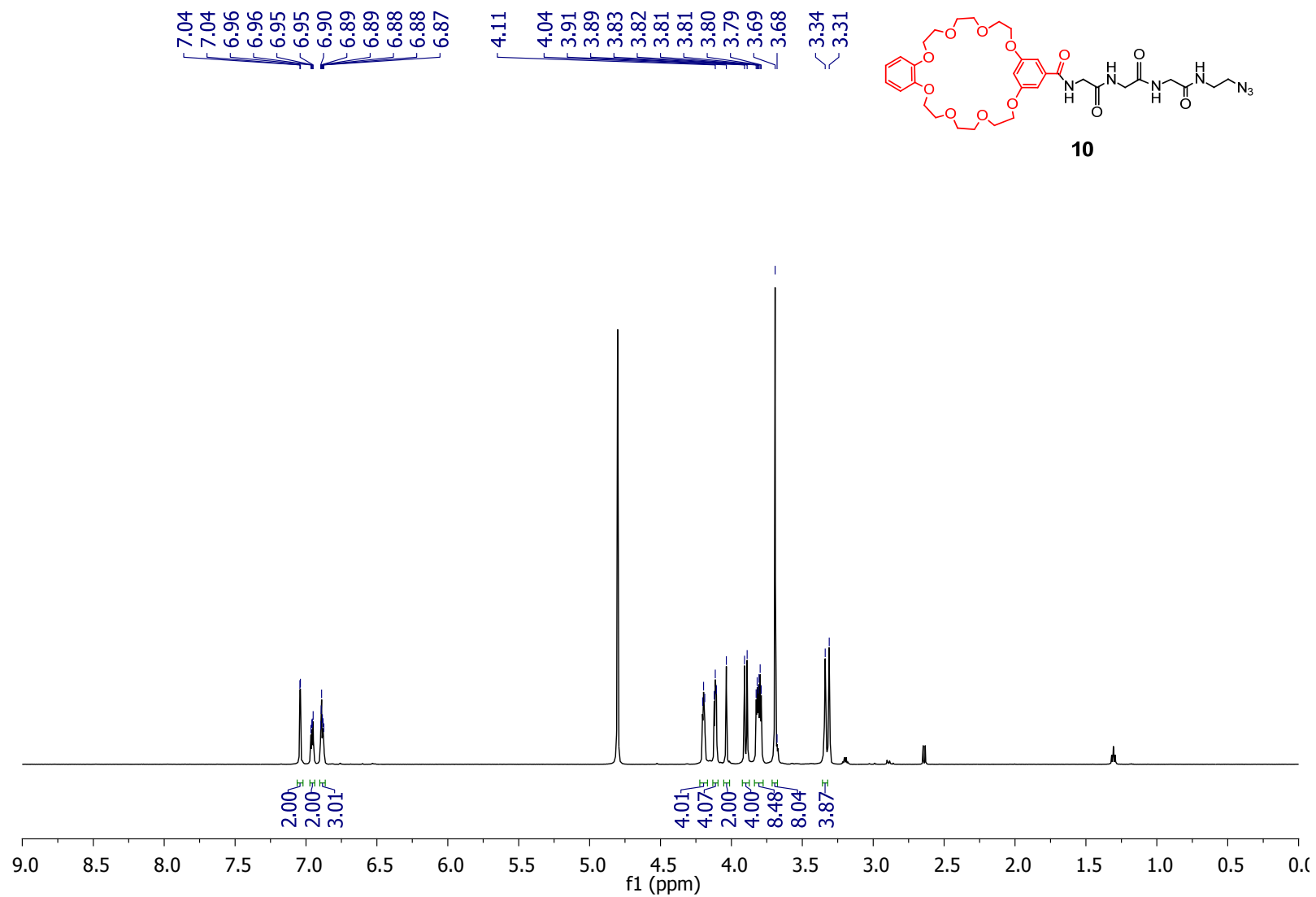
Figure S5. Compound **10**: ^1H -NMR, CD_3OD , 298 K, 600 MHz.

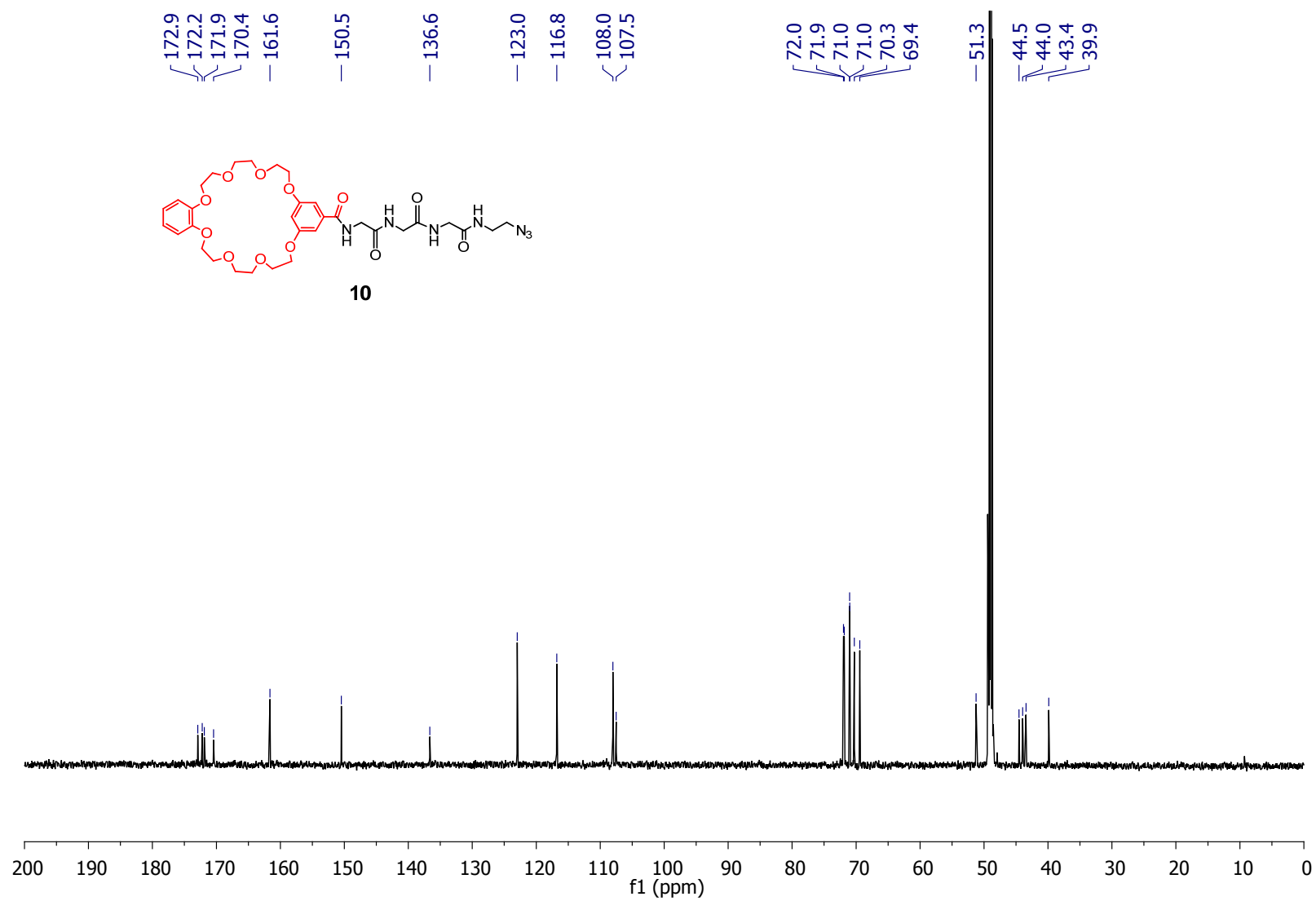
Figure S6. Compound **10**: ^{13}C -NMR, CD_3OD , 298 K, 150 MHz.

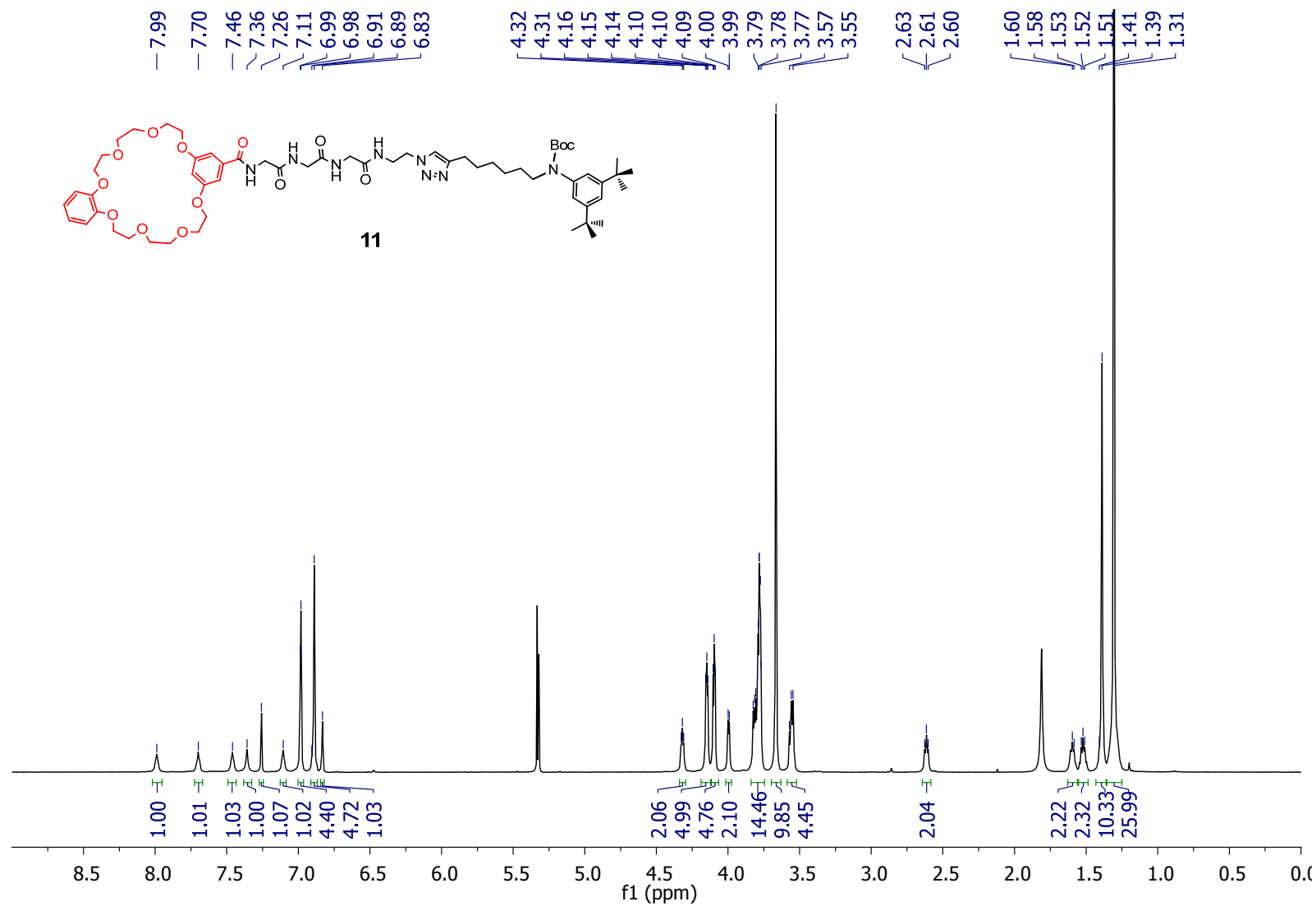
Figure S7. Compound 11: ^1H -NMR, CD_2Cl_2 , 298 K, 600 MHz.

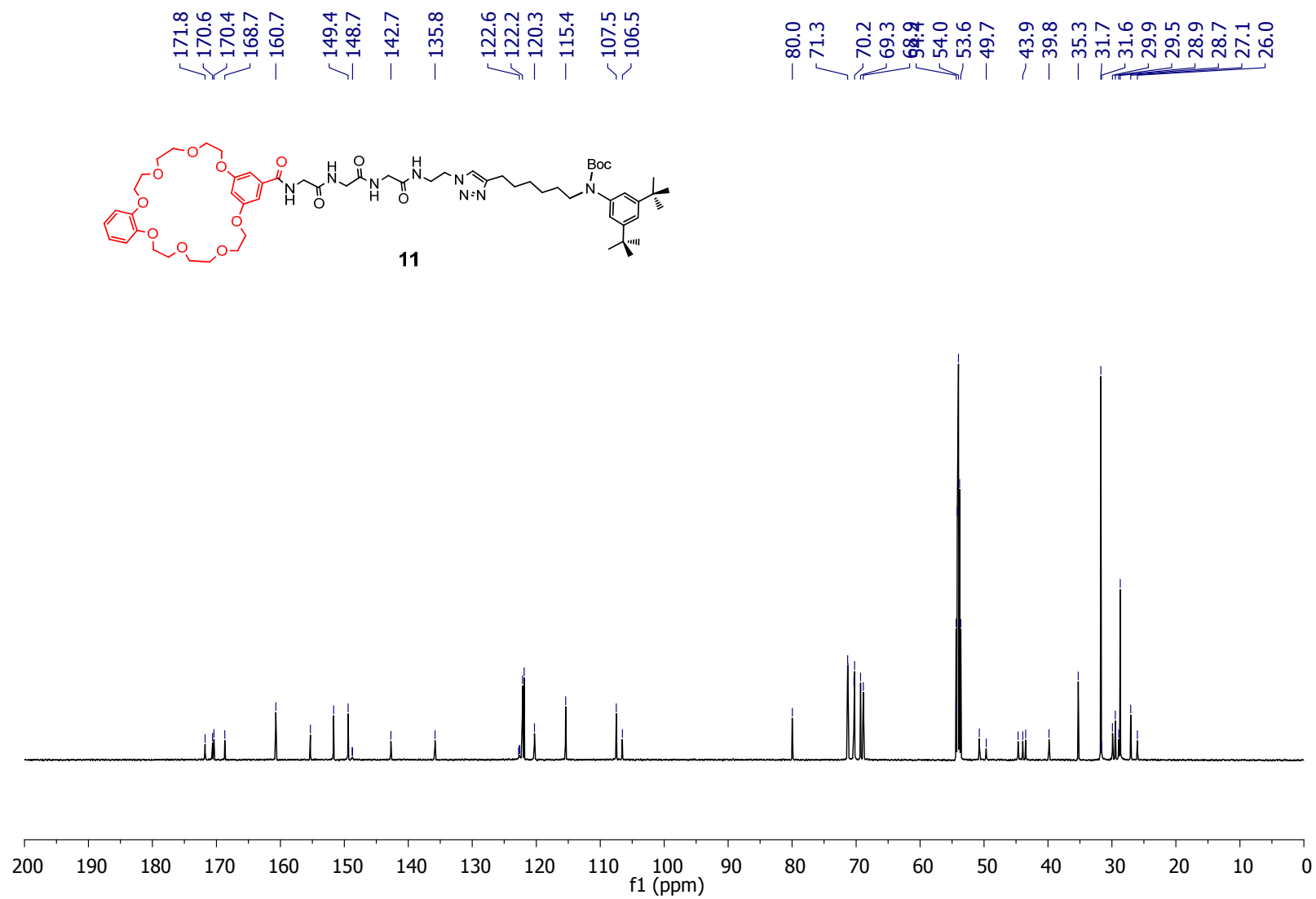
Figure S8. Compound **11**: ^{13}C -NMR, CD_2Cl_2 , 298 K, 150 MHz.

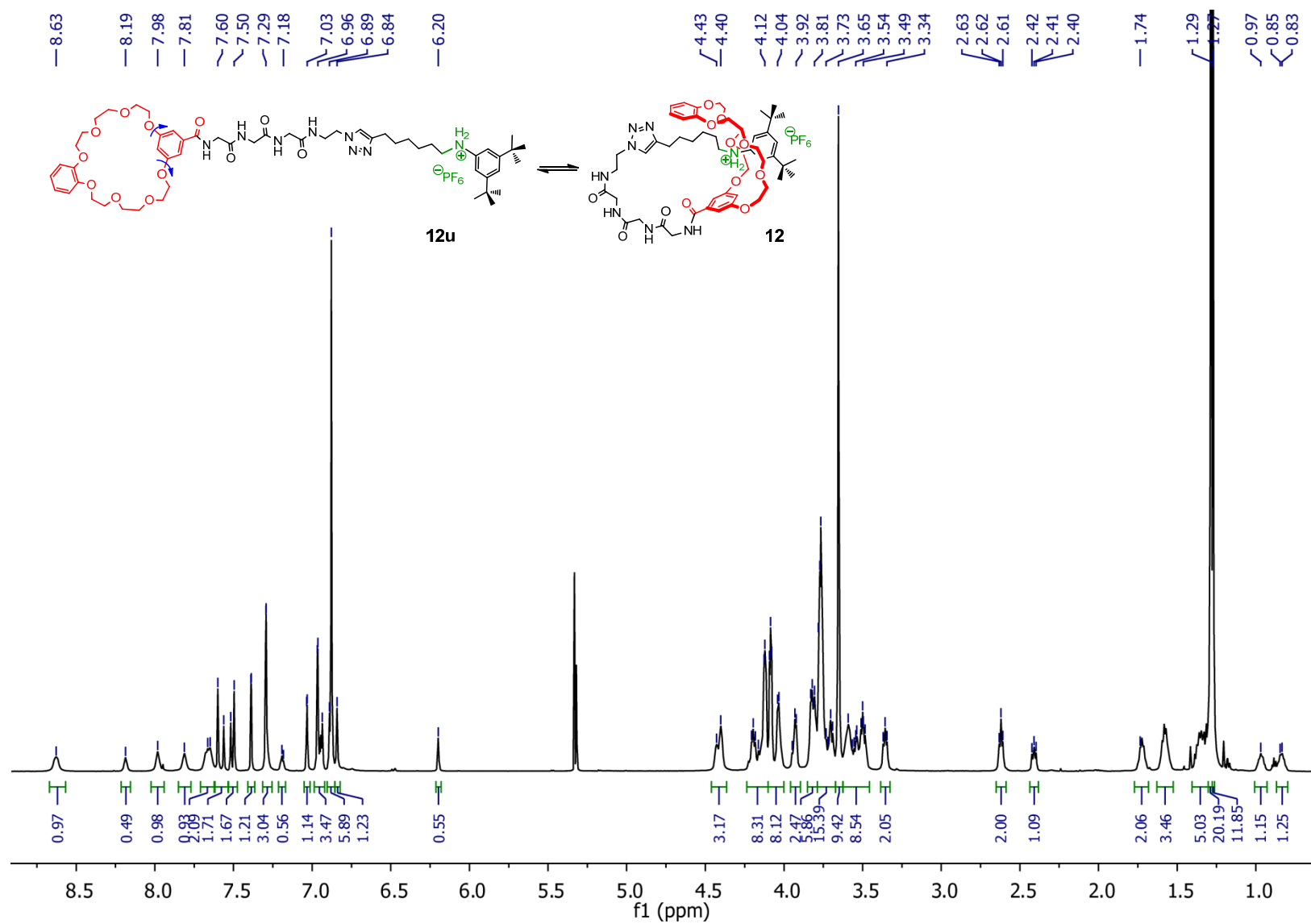
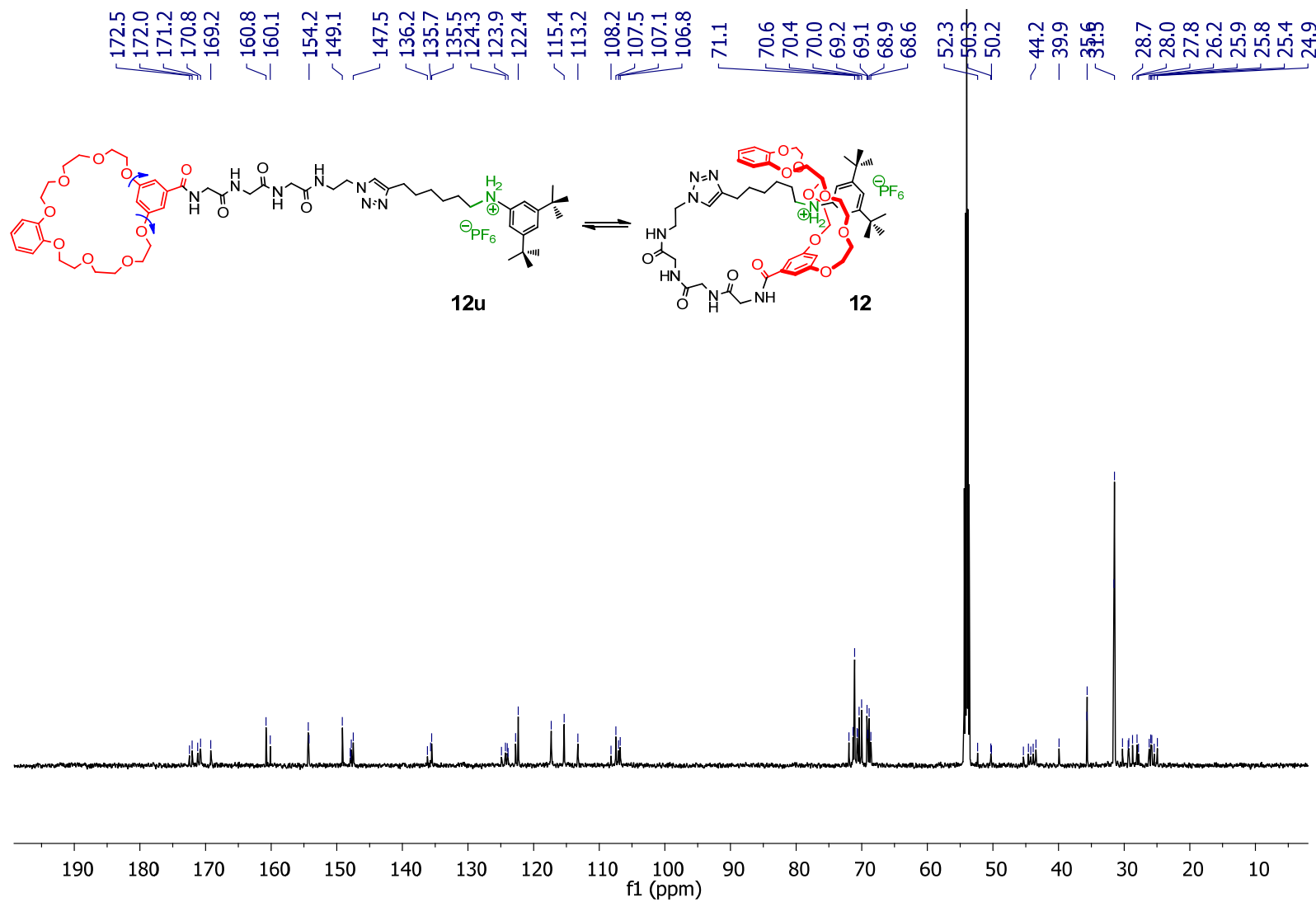
Figure S9. Compound **12**+**12u** ($c = 5.10 \times 10^{-2}$ M): ^1H -NMR, CD_2Cl_2 , 298 K, 600 MHz.

Figure S10. Compound **12+12u** ($c = 5.10^{-2}\text{M}$): ^{13}C -NMR, CD_2Cl_2 , 298 K, 150 MHz.



Chemical structure of 13: A complex macrocyclic molecule featuring a central benzimidazole core. The structure includes a phenyl group, a hexafluorophosphate (PF₆⁻) counterion, and a long chain containing a 1,3-dioxolane ring, a 1,3-dioxane ring, and a 1,3-dioxolane ring. The molecule is labeled **13**.

¹H NMR spectrum (CDCl₃):

Chemical Shift (ppm)	Integration
8.60	1.99
8.16	1.04
7.41	1.18
7.39	1.14
7.36	5.58
7.01	4.51
6.90	3.60
6.86	
6.85	
6.20	1.00
5.64	2.06
4.67	2.02
4.19	7.49
4.07	3.29
3.91	2.40
3.84	21.33
3.79	8.89
3.71	
3.65	
3.50	
3.44	
2.52	2.17
2.51	
2.49	
1.45	3.46
1.45	31.58
0.92	4.51
0.91	2.40
0.90	
0.89	
0.88	
0.87	
0.86	
0.71	

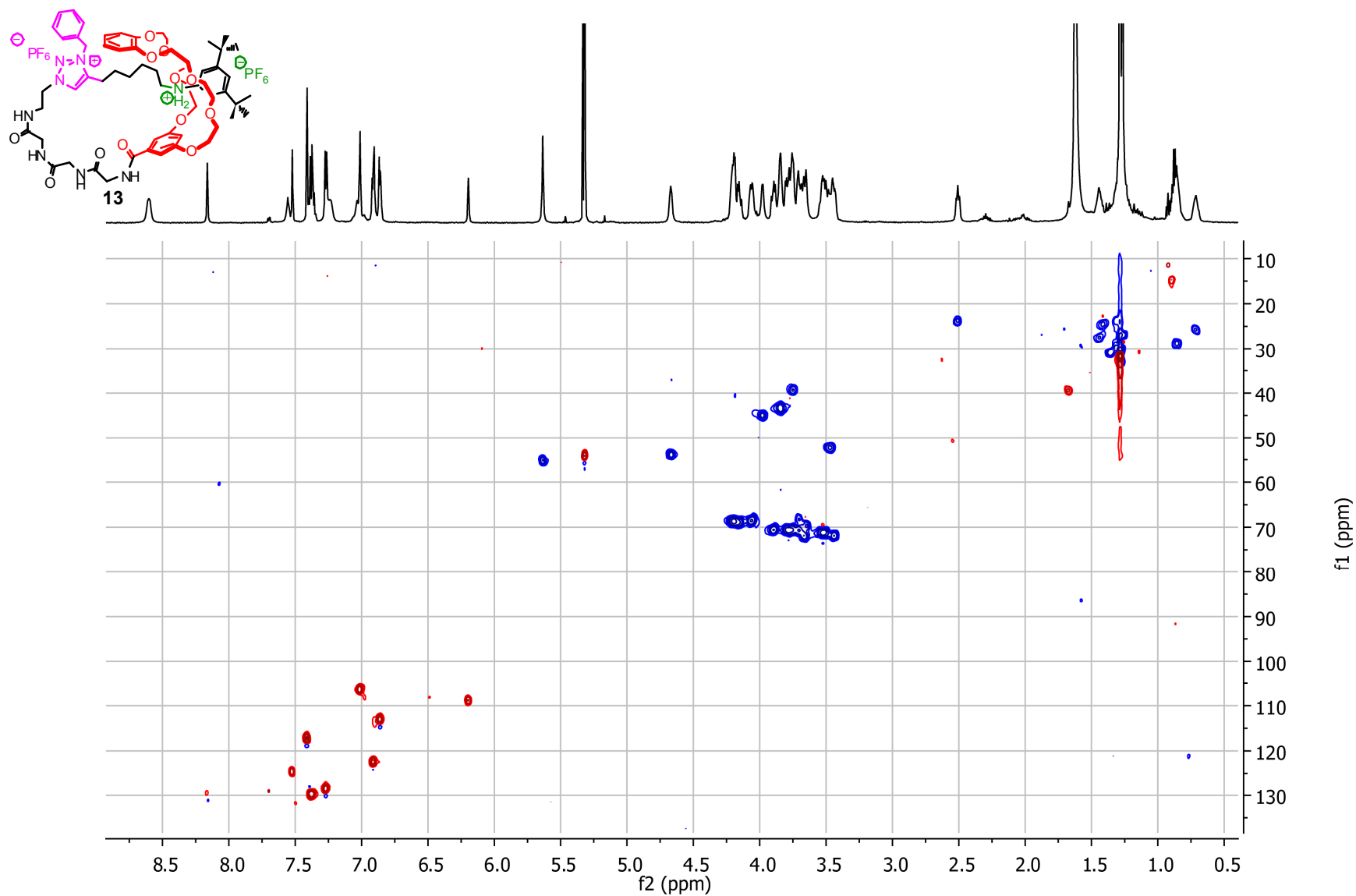
Figure S12. Compound **13**: HSQC NMR, CD₂Cl₂, 298 K, ¹H at 600MHz–¹³C at 150 MHz

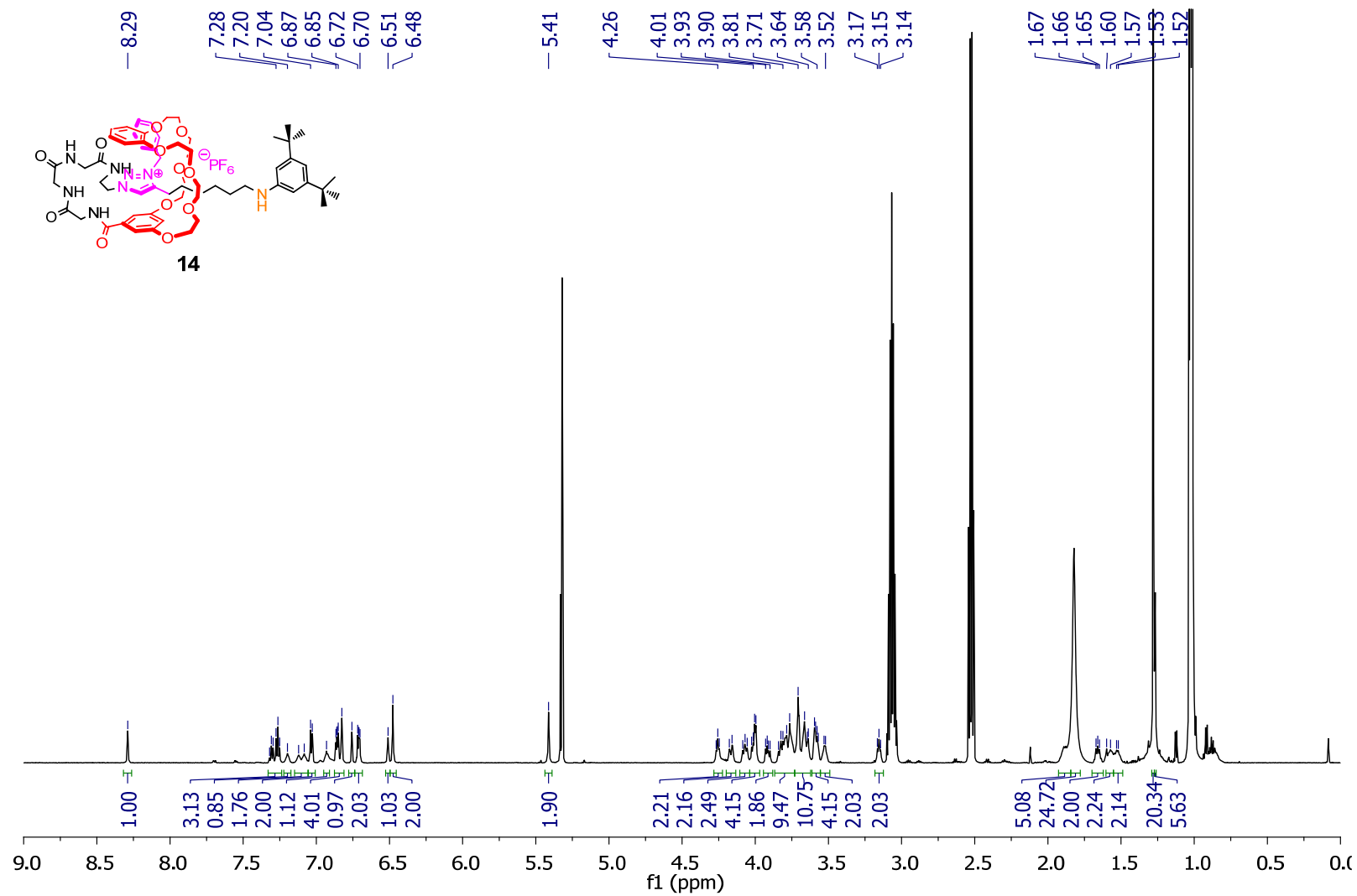
Figure S13. Compound **14**: ^1H -NMR, CD_2Cl_2 , 298 K, 600 MHz

Figure S14. Compound **14**: HSQC NMR, CD₂Cl₂, 298 K, ¹H at 600 MHz–¹³C at 150 MHz

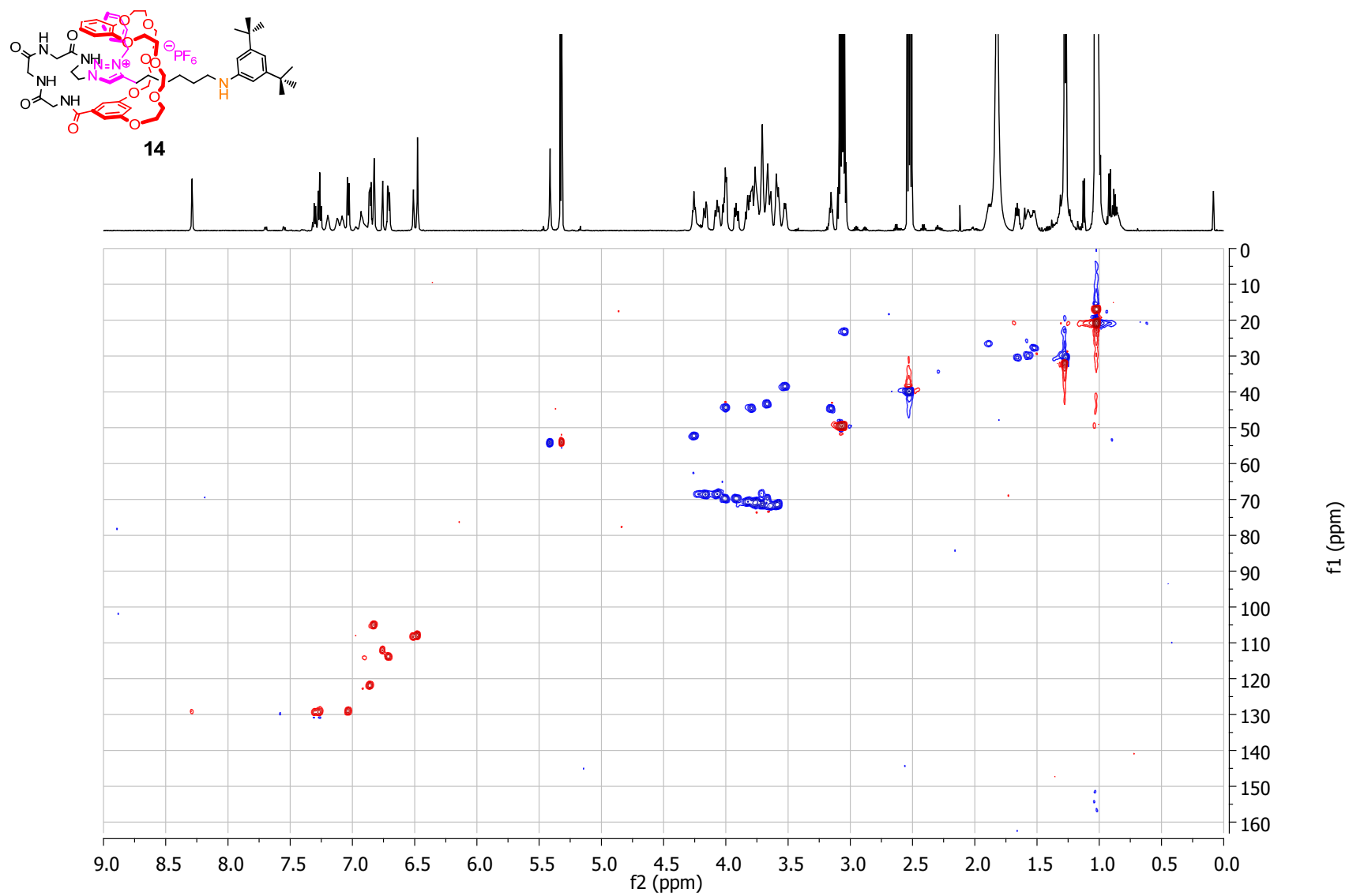


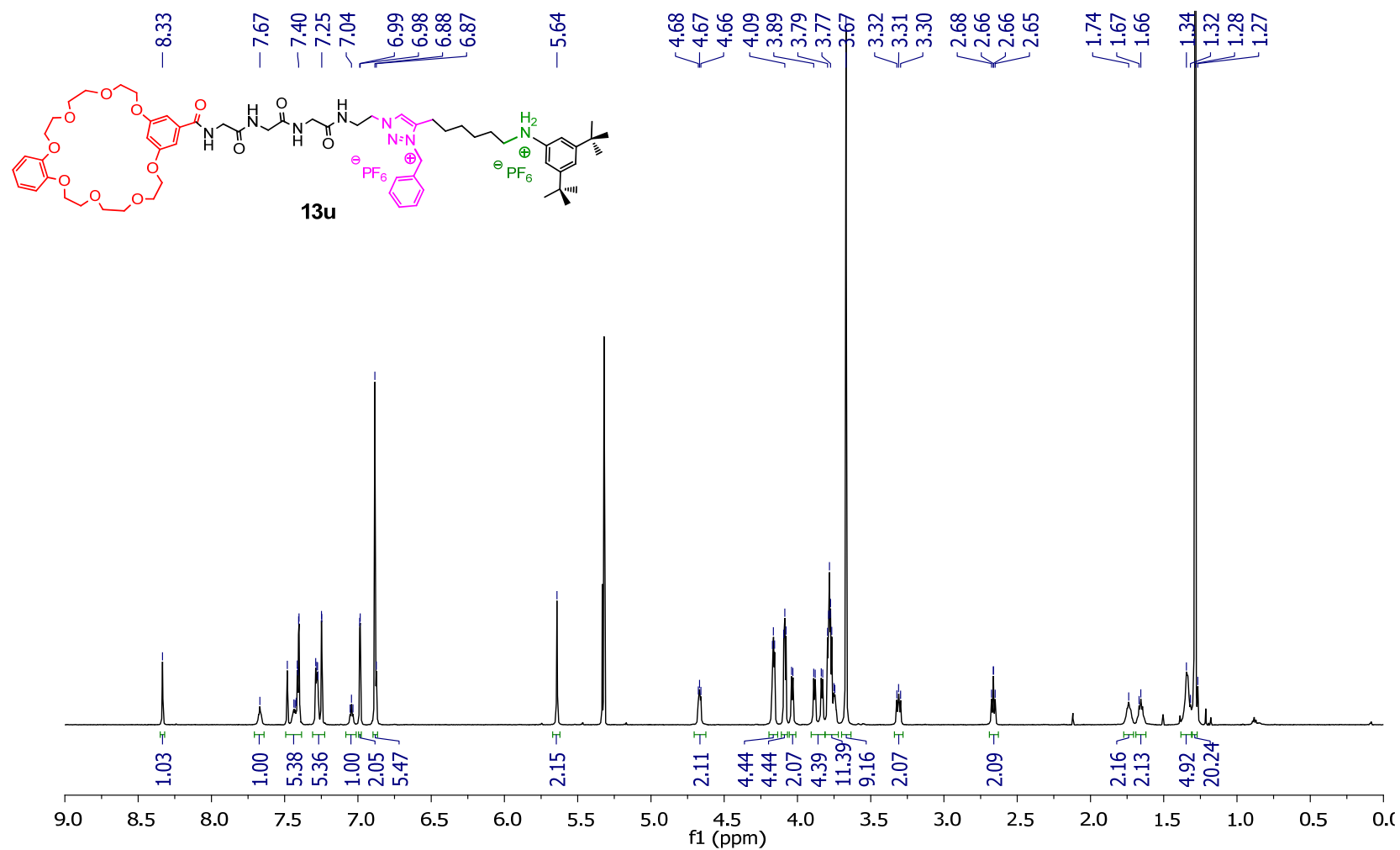
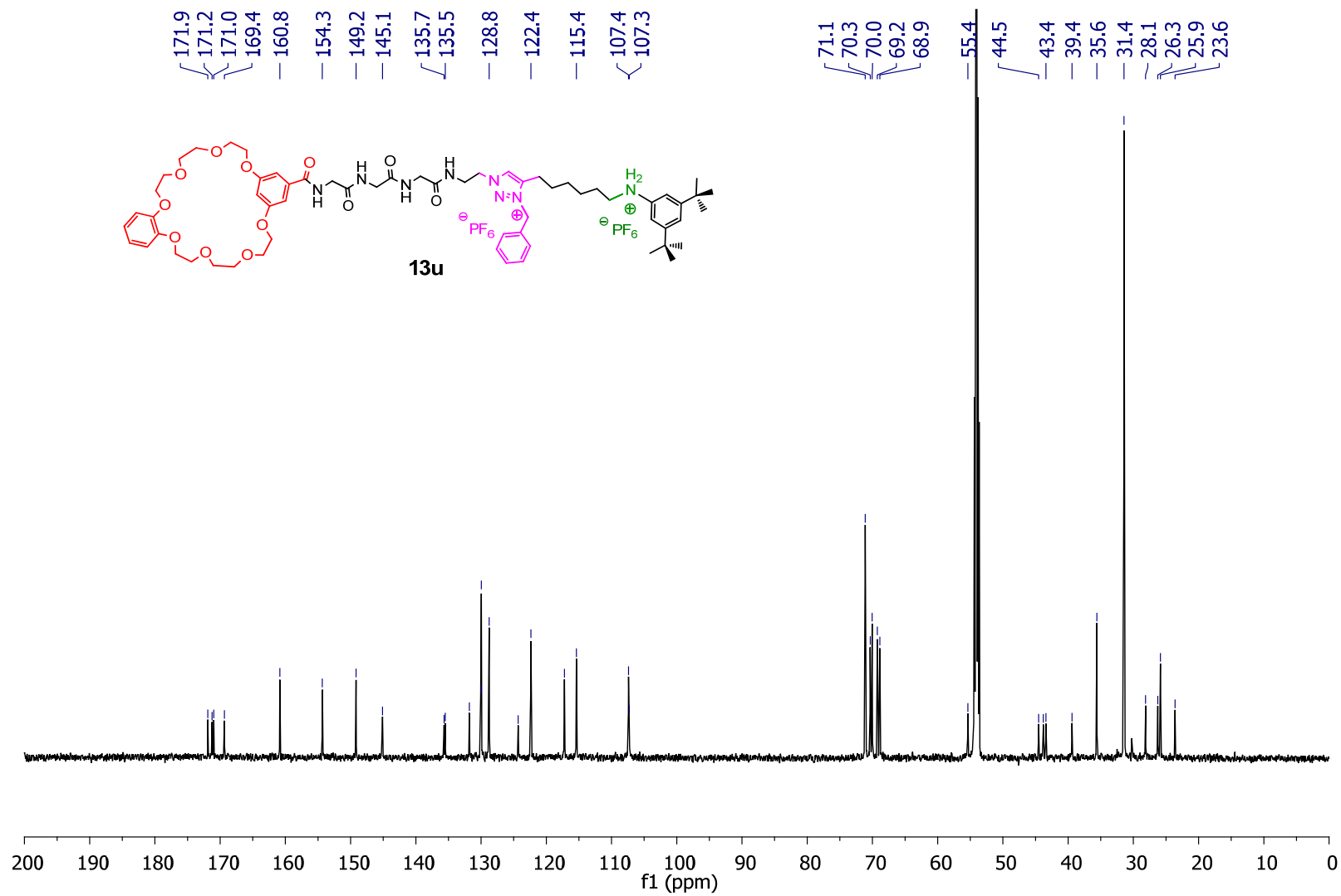
Figure S15. Compound **13u**: ^1H -NMR, CD_2Cl_2 , 298 K, 600 MHz.

Figure S16. Compound **13u**: ^{13}C -NMR, CD_2Cl_2 , 298 K, 150 MHz

14u

Chemical structure of **14u** is shown above the spectrum. The structure features a red macrocyclic ether, a blue amide chain, a green azo group, a yellow phenyl ring, and a blue 2,4,6-trimethylphenyl group.

¹H NMR spectrum (CDCl₃) of **14u**. The x-axis represents the chemical shift in ppm (f1), ranging from 9.0 to 0.0. The spectrum shows several peaks with corresponding integration values (bottom) and chemical shifts (top).

Chemical shifts (ppm) labeled above the peaks: 8.25, 7.42, 7.23, 7.21, 7.11, 6.99, 6.89, 6.75, 6.43, 5.64, 4.64, 4.63, 4.62, 4.08, 3.99, 3.79, 3.77, 3.67, 3.06, 3.05, 2.72, 2.71, 2.69, 1.58, 1.28.

Integration values labeled below the peaks: 1.01, 5.06, 2.90, 0.93, 7.43, 1.01, 2.05, 2.01, 1.99, 4.05, 4.10, 1.93, 12.73, 10.81, 2.04, 2.00, 4.92, 5.26, 4.97, 22.59.

Figure S18. Compound **14u**: ^{13}C -NMR, CD_2Cl_2 , 298 K, 150 MHz.