

Supporting Information for:

Insights Into Reactivity Trends for Electrochemical C-N Bond Formations

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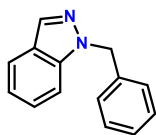
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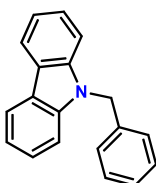
Figure S1: Picture of electrochemical setup in an H-Cell performed with a Tygon tube used to balance pressure. Reticulated Vitreous Carbon electrodes were employed as cathode and anode and connected to the potentiostat via stainless steel wires that are kept out of solution.

List of ^1H & ^{13}C NMR peaks of products

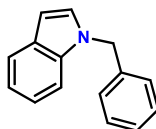
Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet).



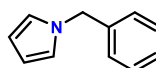
The isolated product (**1**) was a white solid, isolated using a Hexane/EtOAc system (4:1), ^1H NMR (DMSO, 400 MHz) δ 8.48 (s, 1H), 7.72-7.70 (d, 1H), 7.60-7.58 (dd, 1H), 7.38-7.29 (m, 5H), 7.25-7.21 (t, 1H), 7.05-7.01 (t, 1H), 5.64 (s, 2H). ^{13}C NMR (DMSO, 400 MHz) δ (ppm): 148.71, 137.45, 129.03, 128.38, 128.32, 125.90, 124.44, 122.03, 121.50, 121.10, 117.46, 56.74.



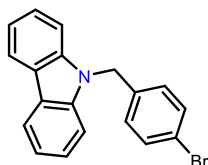
The isolated product (**2**) was a white solid, 3.2 mg, isolated using a Hexane/EtOAc system (4:1), ^1H NMR (DMSO, 300 MHz) δ (ppm): 8.219-8.17 (d, 2H), 7.64-7.61 (d, 2H), 7.45-7.40 (t, 2H), 7.26-7.15 (m, 7H), 5.66 (s, 2H). ^{13}C NMR (DMSO, 300 MHz) δ (ppm): 140.61, 138.31, 129.03, 127.71, 127.19, 126.30, 122.68, 120.80, 119.48, 109.99, 46.02.



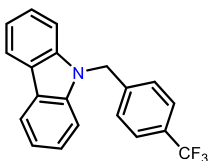
The isolated product (**3**) was a white solid, isolated using a Hexane/EtOAc system (4:1), ^1H NMR (DMSO, 300 MHz) δ (ppm): 7.60-7.57 (d, 1H), 7.51-7.50 (d, 1H), 7.46-7.44 (d, 1H), 7.32-7.19 (m, 5H), 7.13-7.01 (m, 2H), 6.52-6.51 (d, 1H), 5.43 (s, 2H). ^{13}C NMR (DMSO, 300 MHz) δ (ppm): 138.77, 136.23, 129.57, 128.98, 128.89, 128.80, 128.70, 127.78, 127.46, 121.63, 120.95, 119.56, 110.60, 101.45, 49.57.



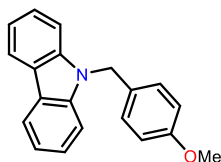
The isolated product (**4**) was a clear oil, isolated using Hexane/DCM (6:1), ^1H NMR (DMSO, 400 MHz) δ 7.35-7.27 (m, 3H), 7.19-7.17 (d, 2H), 6.82-6.81 (t, 2H), 6.03-6.02 (t, 2H) 5.10 (s, 2H). ^{13}C NMR (DMSO, 400 MHz) δ (ppm): 139.53, 128.94, 127.78, 127.58, 121.40, 108.43, 52.64.



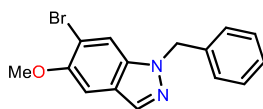
The isolated product (**5**) was a white solid, isolated using Hexane/DCM (6:1), ^1H NMR (DMSO, 400 MHz) δ (ppm): 8.25-8.23 (d, 2H), 7.69-7.66 (d, 2H), 7.54-7.47 (m, 4H), 7.30-7.26 (t, 2H), 7.18-7.16 (d, 2H), 5.71 (s, 2H). ^{13}C NMR (DMSO, 400 MHz) δ (ppm): 140.50, 137.79, 131.95, 129.41, 126.41, 122.73, 120.85, 119.61, 109.94, 45.40.



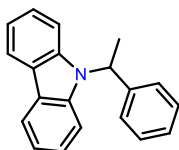
The isolated product (**6**) was a tan solid, isolated using Hexane/EtOAc (4:1), ^1H NMR (DMSO, 400 MHz) δ (ppm): 8.21-8.19 (d, 2H), 7.66-7.60 (m, 4H), 7.46-7.42 (t, 2H), 7.34-7.32 (d, 2H), 7.26-7.22 (t, 2H), 5.79 (s, 2H). ^{13}C NMR (DMSO, 400 MHz) δ (ppm): 140.52, 127.81, 126.45, 125.99, 122.75, 120.89, 119.73, 109.88, 45.59. ^{19}F NMR (DMSO, 400 MHz) δ (ppm): -60.94.



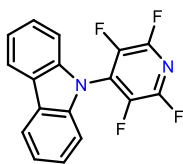
The isolated product (**7**) was a tan solid, isolated using Hexane/EtOAc (4:1), ^1H NMR (DMSO, 400 MHz) δ (ppm): 8.18-8.16 (d, 2H), 7.66-7.64 (d, 2H), 7.46-7.42 (t, 2H), 7.23-7.19 (t, 2H), 7.16-7.14 (d, 2H), 6.83-6.81 (d, 2H), 5.58 (s, 2H). ^{13}C NMR (DMSO, 400 MHz) δ (ppm): 158.94, 140.54, 130.18, 128.61, 126.24, 122.68, 120.77, 119.39, 114.42, 110.03, 55.47, 45.49.



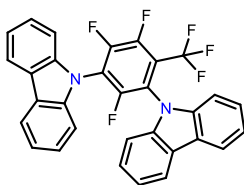
The isolated product (**8**) was a tan solid, isolated using Hexane/EtOAc (4:1), ^1H NMR (DMSO, 400 MHz) δ (ppm): 8.11 (s, 1H), 8.03 (s, 1H), 7.36 (s, 1H), 7.33-7.58 (m, 3H), 7.21-7.19 (d, 2H), 5.63 (s, 2H), 3.86 (s, 3H). ^{13}C NMR (DMSO, 400 MHz) δ (ppm): 150.48, 135.71, 133.10, 128.99, 127.97, 127.73, 114.64, 101.73, 56.90, 52.31.



The isolated product (**9**) was a white solid, isolated using Hexane/DCM (6:1), ^1H NMR (DMSO, 400 MHz) δ (ppm): 8.19-8.17 (d, 2H), 7.47-7.45 (d, 2H), 7.37-7.47 (m, 4H), 7.30-7.24 (m, 7H), 6.32-6.27 (dd, 1H), 1.97-1.95 (d, 3H). ^{13}C NMR (DMSO, 400 MHz) δ (ppm): 141.42, 139.90, 129.00, 127.65, 126.82, 126.02, 123.09, 120.76, 119.27, 110.88, 52.16, 17.74.



The isolated product (**10**) was a white solid, isolated using Hexane/DCM (6:1), ^1H NMR (DMSO, 400 MHz) δ (ppm): 8.30-8.28 (d, 2H), 7.56-7.50 (m, 4H), 7.43-7.39 (t, 2H). ^{13}C NMR (DMSO, 400 MHz) δ (ppm): 145.43, 143.04, 140.82, 140.47, 139.03, 138.23, 137.87, 128.09, 127.32, 124.10, 122.39, 121.20, 111.42. ^{19}F NMR (DMSO, 400 MHz) δ (ppm): -89.95, -144.08. High Resolution Mass Spec: Calculated: 316.0624 m/z; Found: 316.0629 m/z.



The isolated product (**11**) was a white solid, isolated using Hexane/DCM (6:1), ^1H NMR (DMSO, 400 MHz) δ (ppm): 8.27-8.23 (t, 2H), 7.57-7.48 (m, 4H), 7.39-7.32 (m, 2H). ^{13}C NMR (DMSO, 400 MHz) δ (ppm): 141.87, 139.69, 127.22, 127.17, 123.85, 123.47, 122.00, 121.36, 121.20, 121.10, 110.98, 110.31. ^{19}F NMR (DMSO, 400 MHz) δ (ppm): -56.75, -69.21, -71.10, -126.20, -133.67, -137.55. High Resolution Mass Spec: Calculated: 530.1218 m/z; Found: 530.1206 m/z.

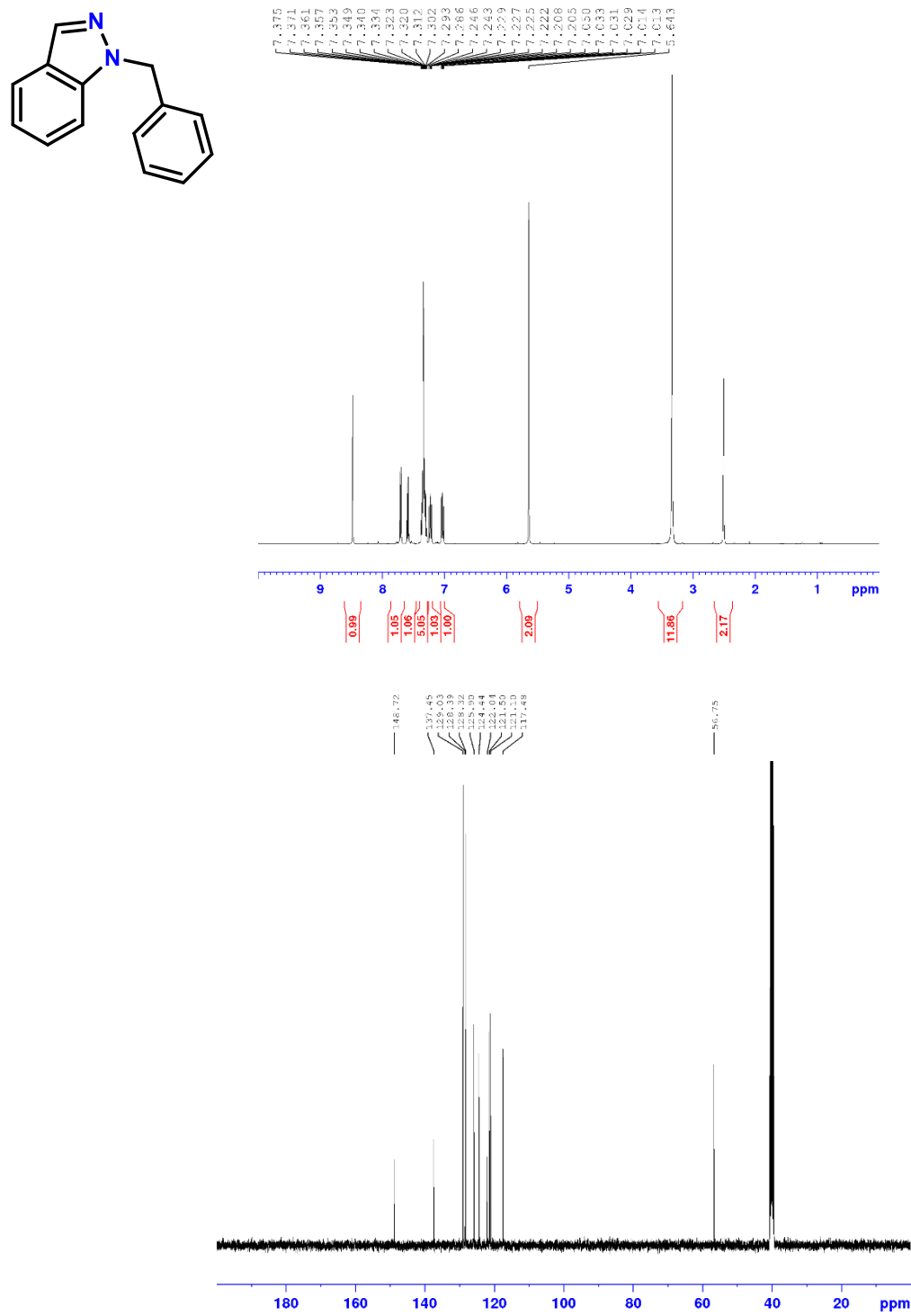


Figure S2: ¹H & ¹³C NMR spectra of product 1. The spectra matched reference.¹

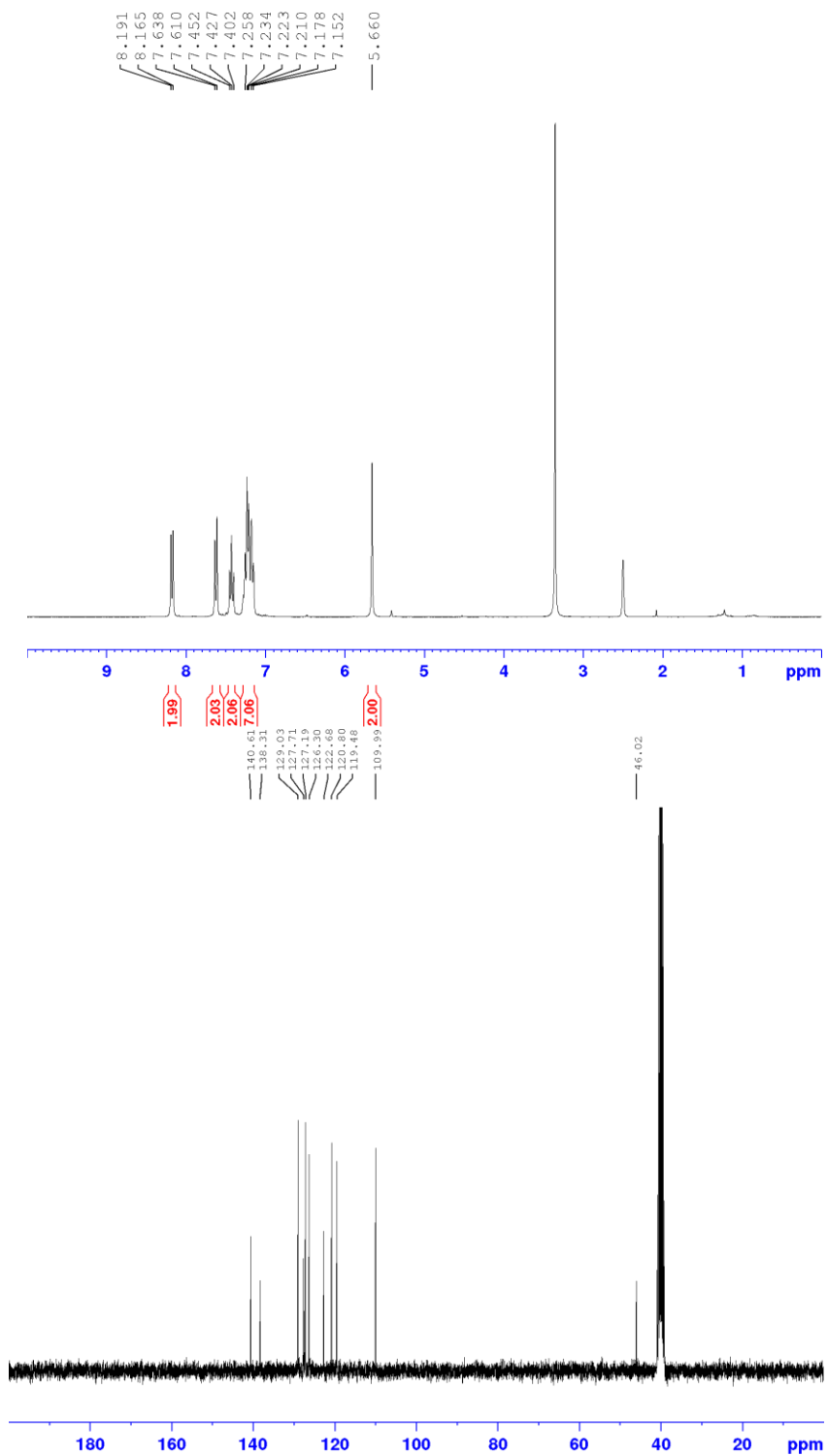
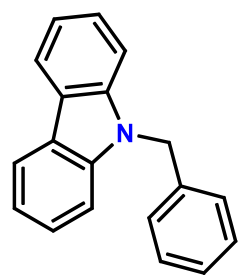


Figure S3: ^1H & ^{13}C NMR spectra of product 2. The spectra matched reference.²

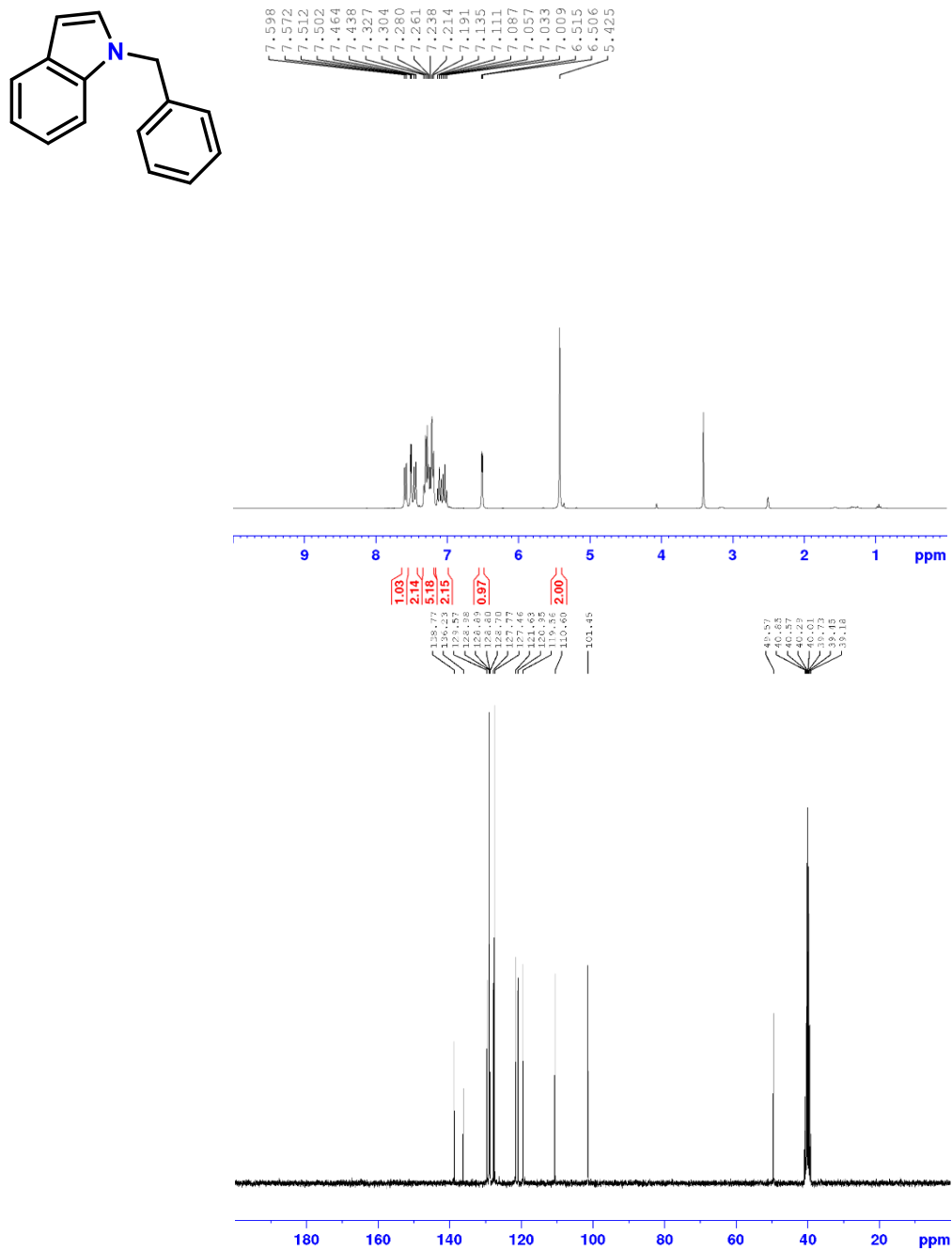


Figure S4: ¹H & ¹³C NMR spectra of product 3. The spectra matched reference.³

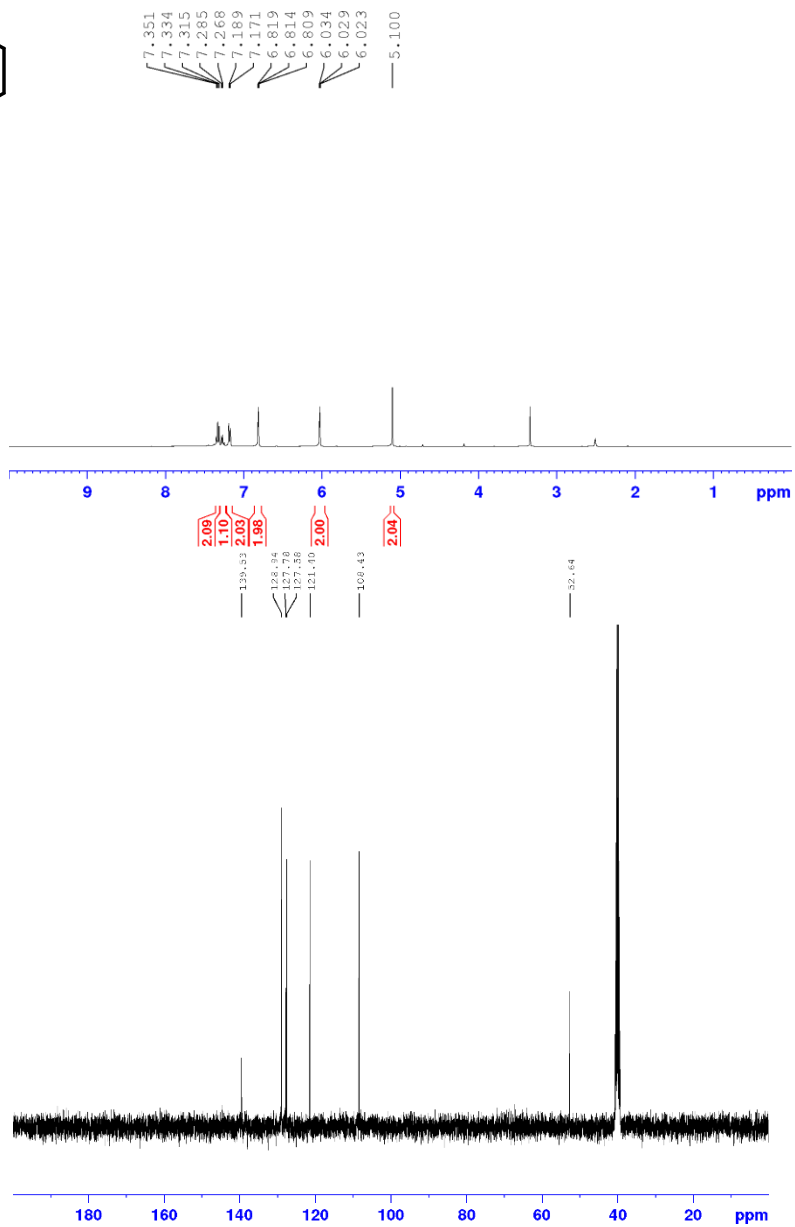
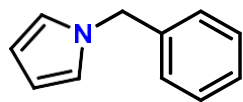


Figure S5: ¹H & ¹³C NMR spectra of product 4. The spectra matched reference.⁴

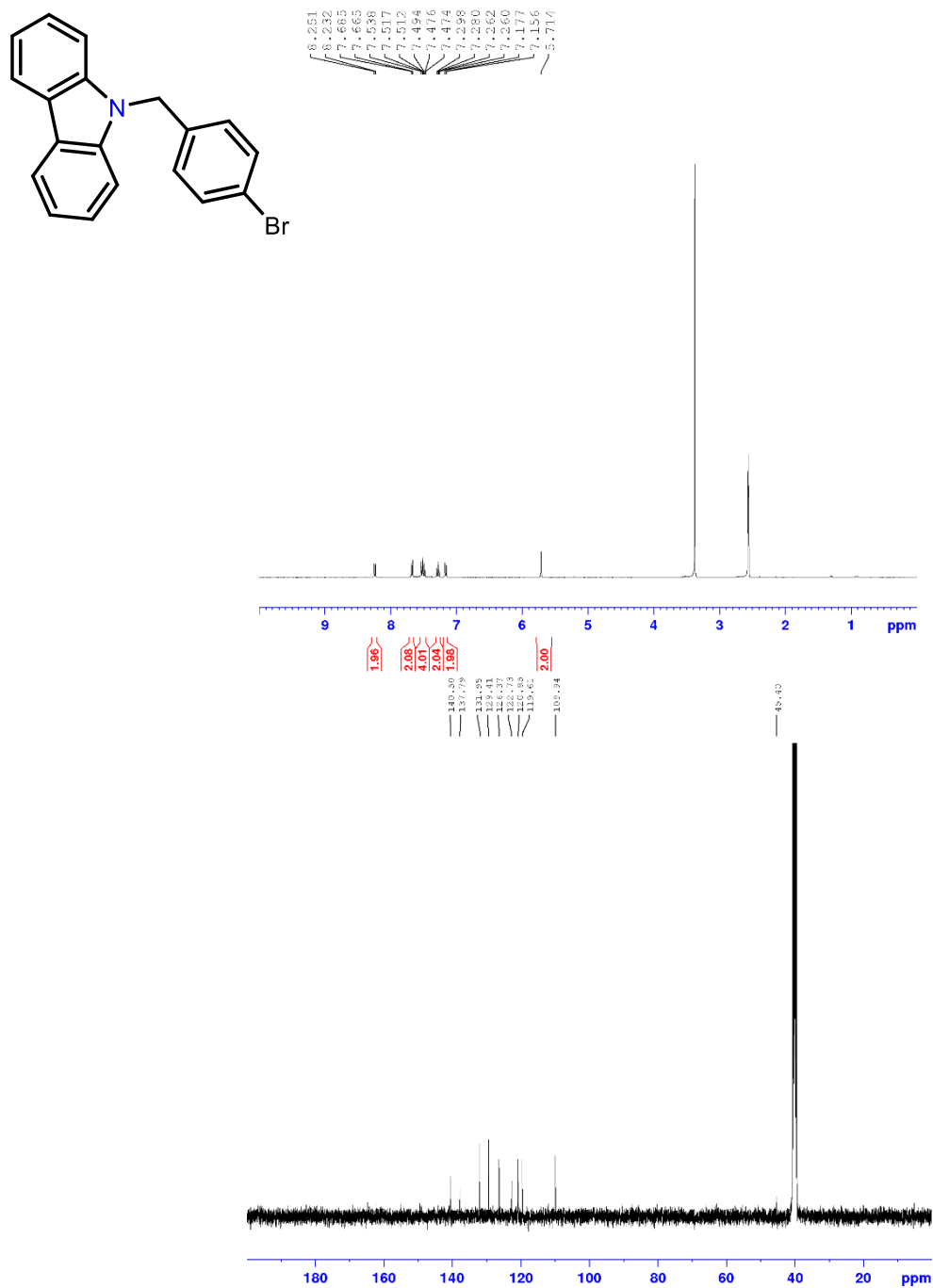
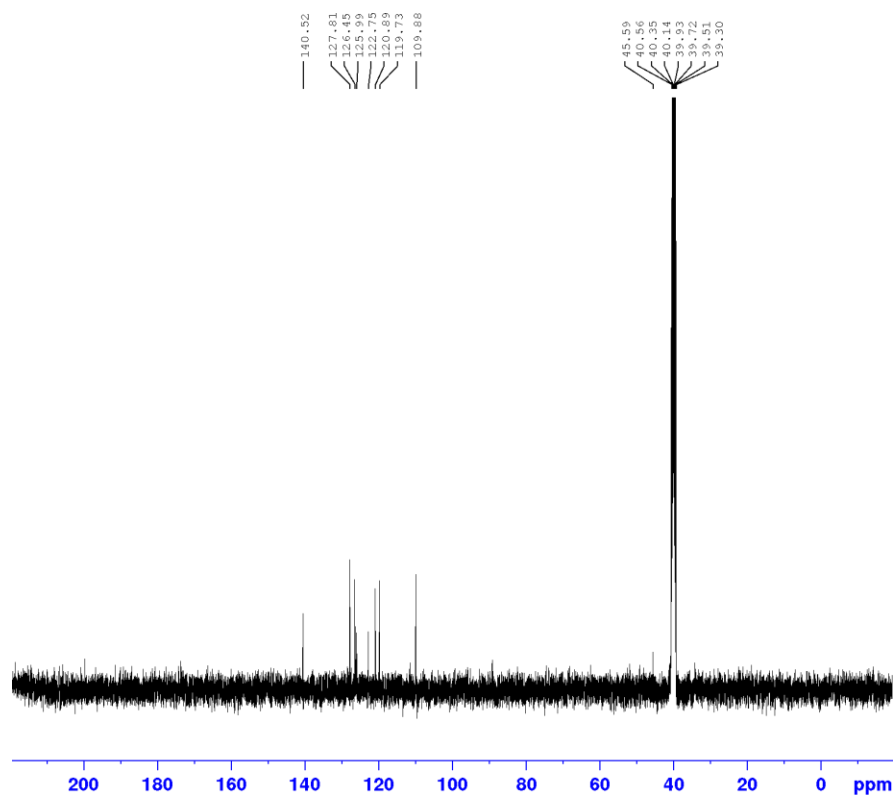
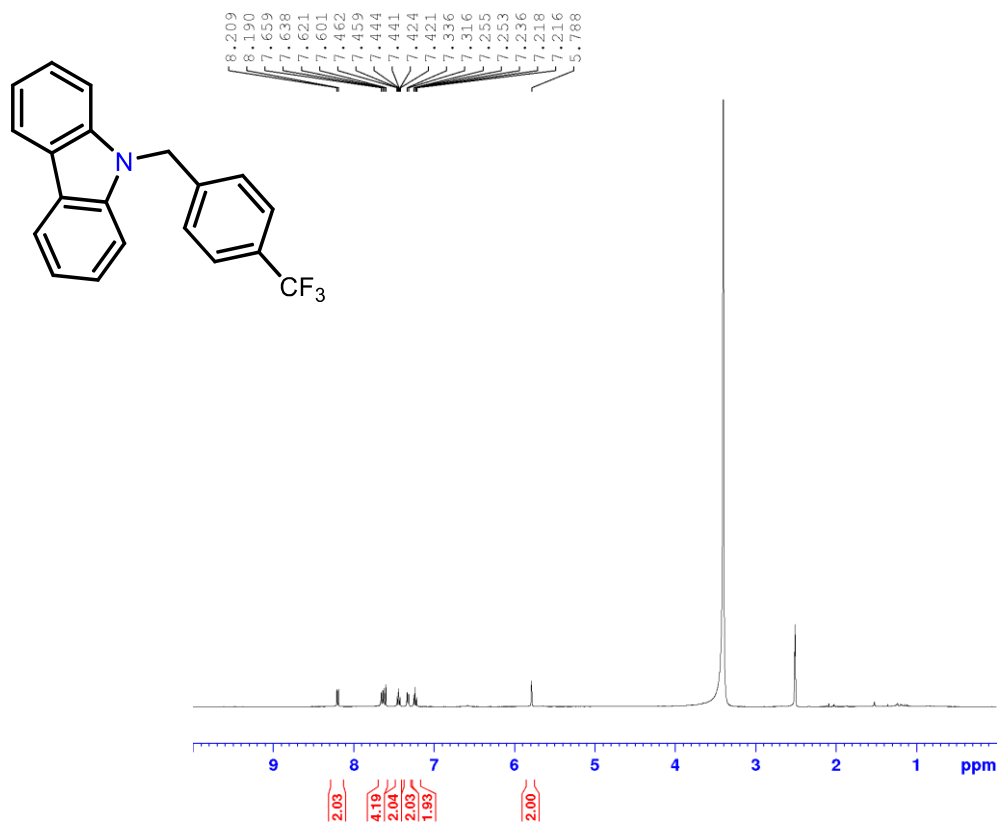


Figure S6: ¹H & ¹³C NMR spectra of product 5. The spectra matched reference.⁵



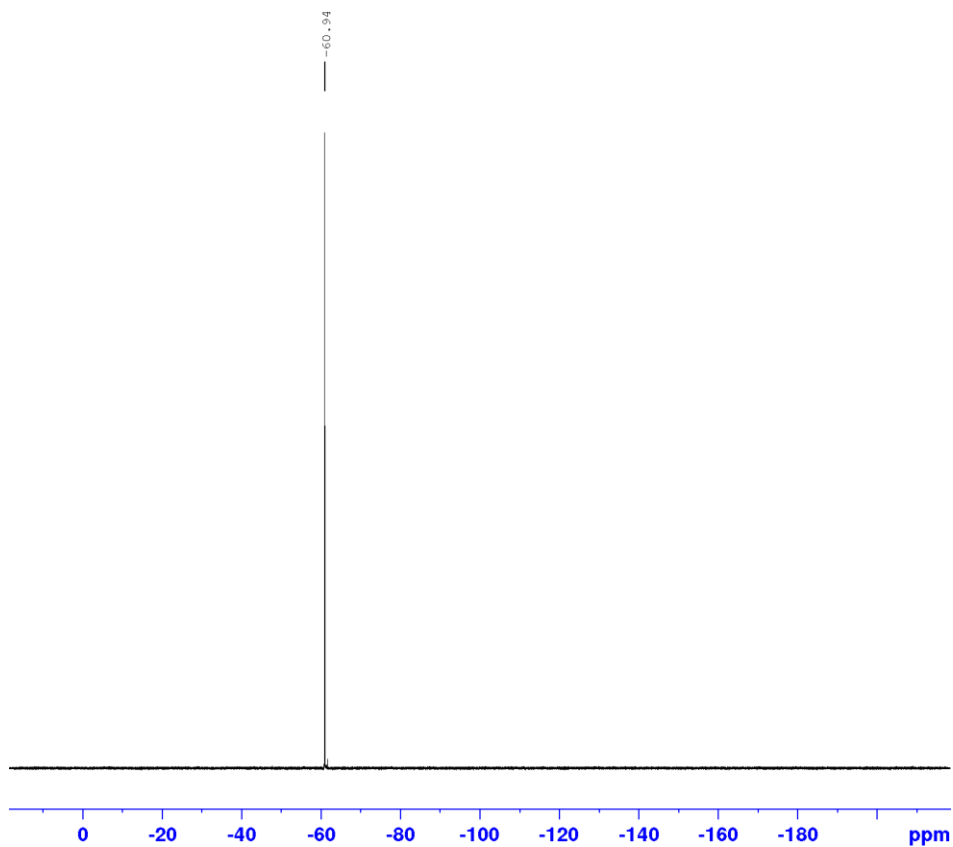


Figure S7: ^1H & ^{13}C & ^{19}F NMR spectra of product **6**. The spectra matched reference.⁶

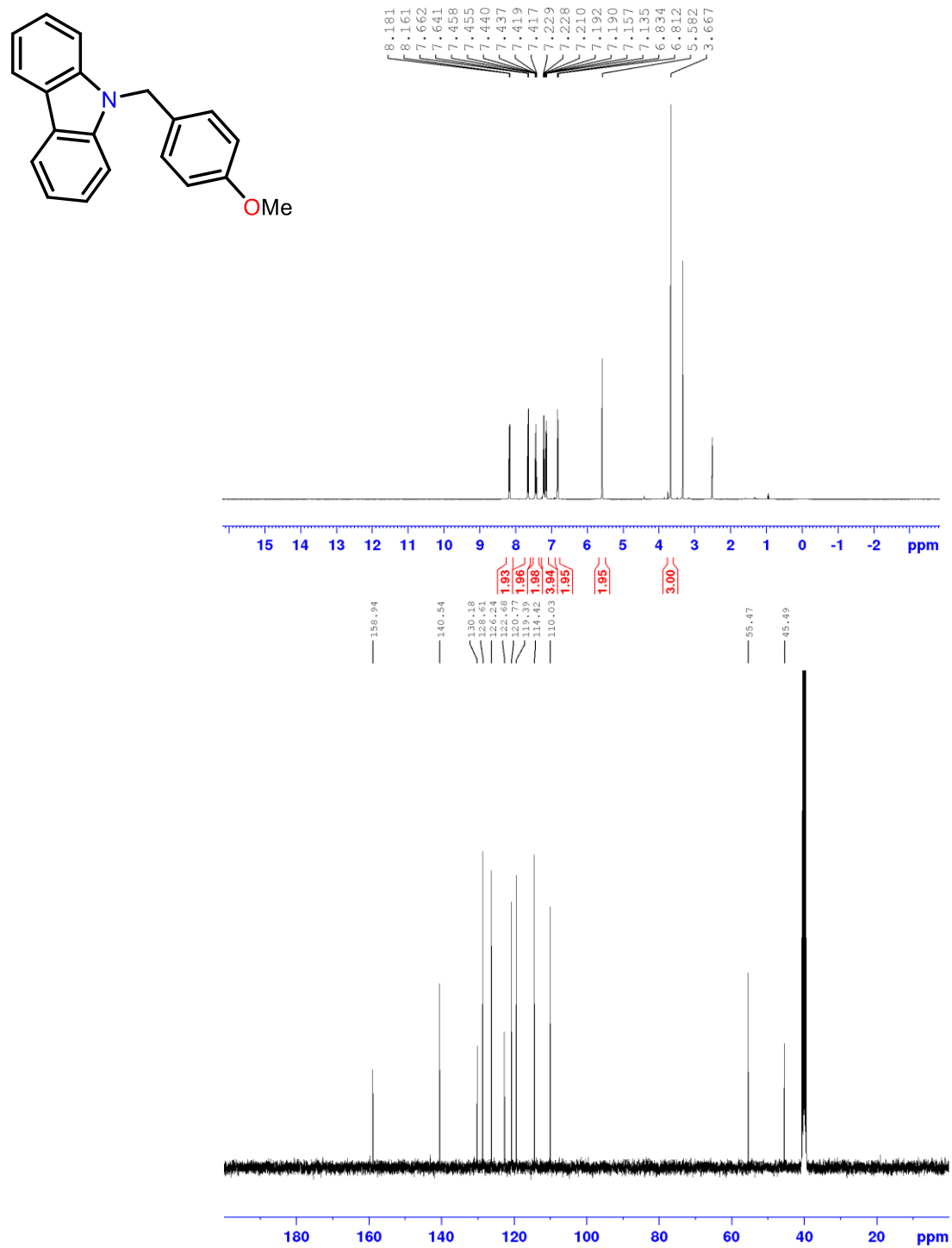


Figure S8: ¹H & ¹³C NMR spectra of product 7. The spectra matched reference.⁷

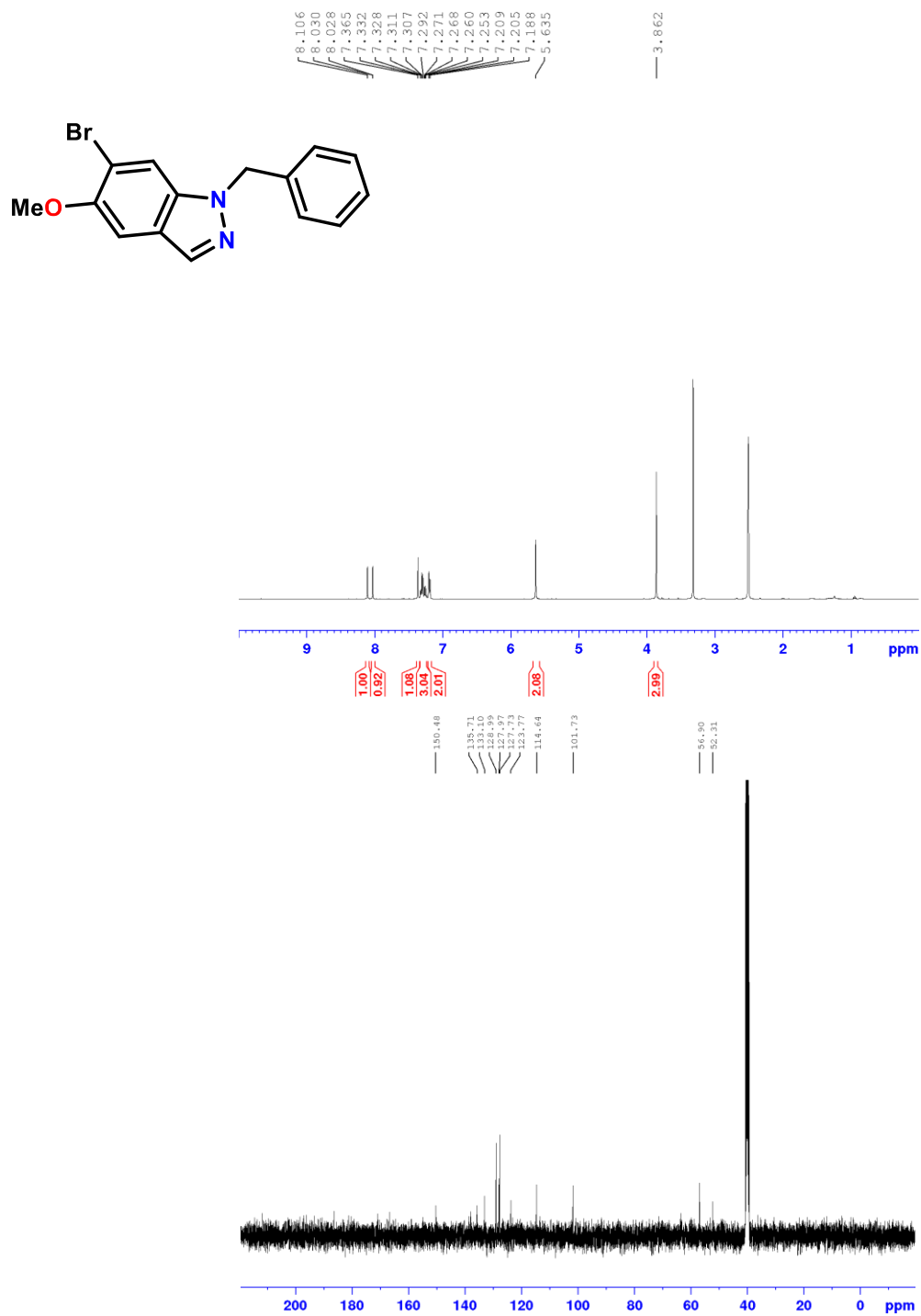


Figure S9: ^1H & ^{13}C NMR spectra of product **8**. The spectra matched reference.⁸

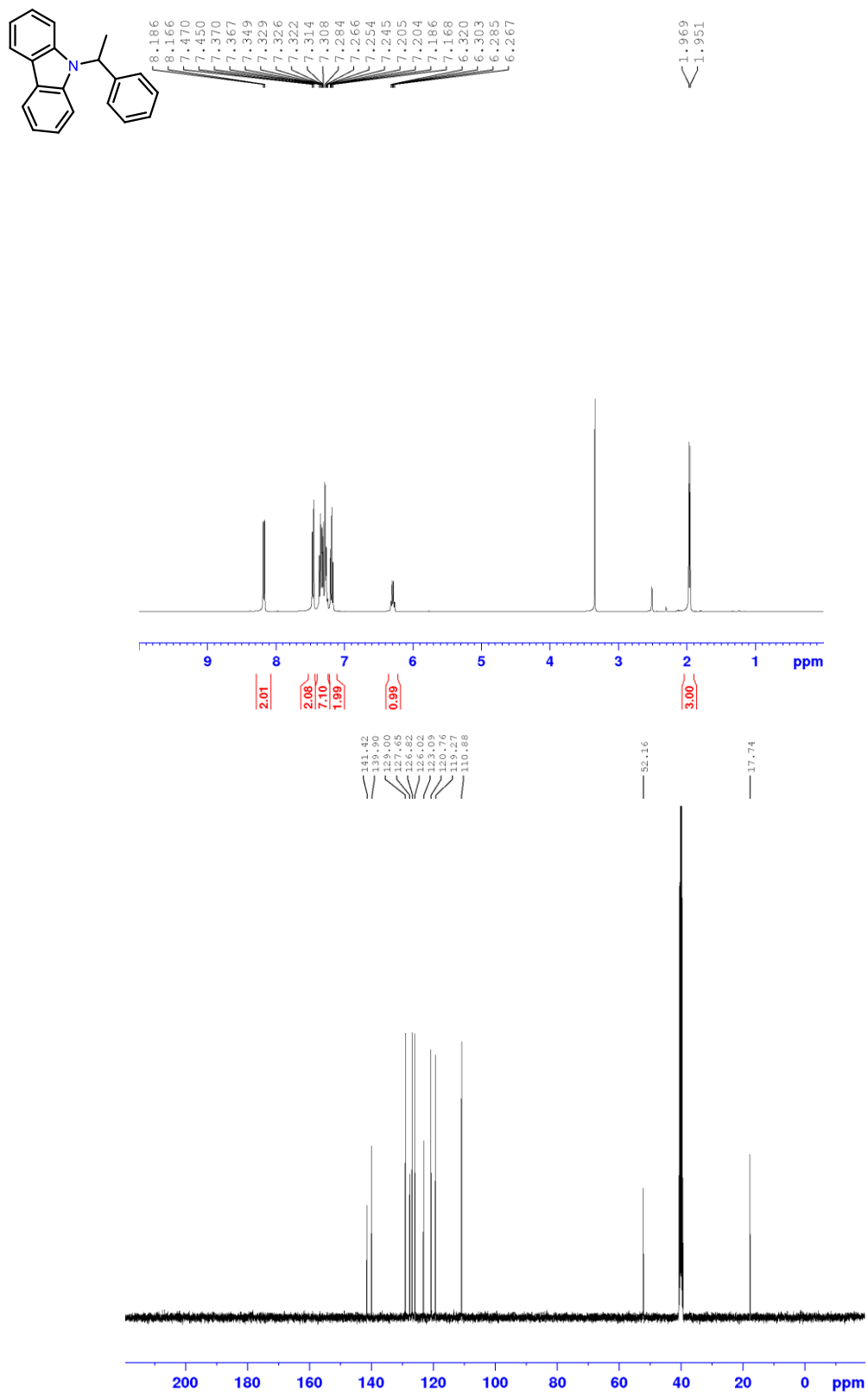
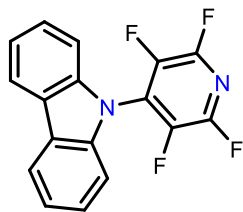
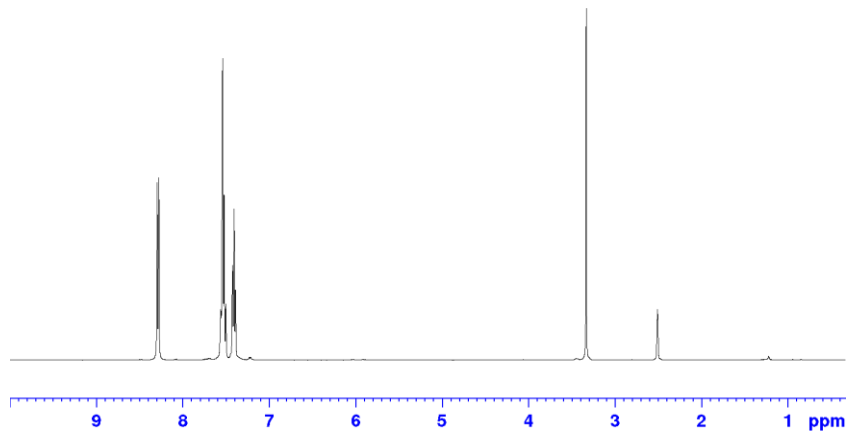


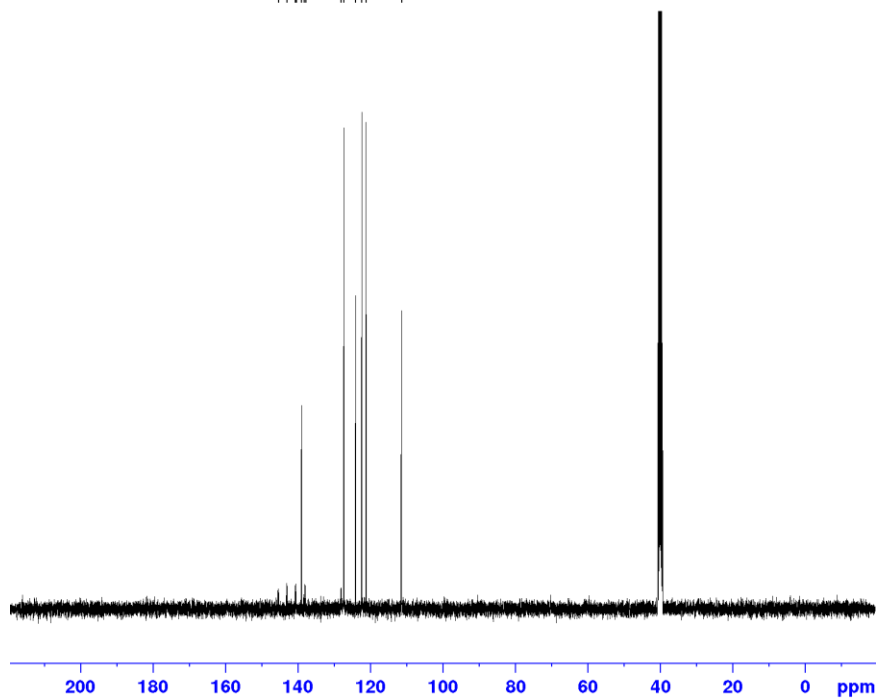
Figure S10: ¹H & ¹³C NMR spectra of product **9**. The spectra matched reference.⁸



8.298
8.278
7.561
7.540
7.523
7.503
7.428
7.424
7.408
7.392
7.388



145.41
143.08
140.82
140.47
138.03
137.87
137.82
128.09
127.32
124.10
122.39
111.42



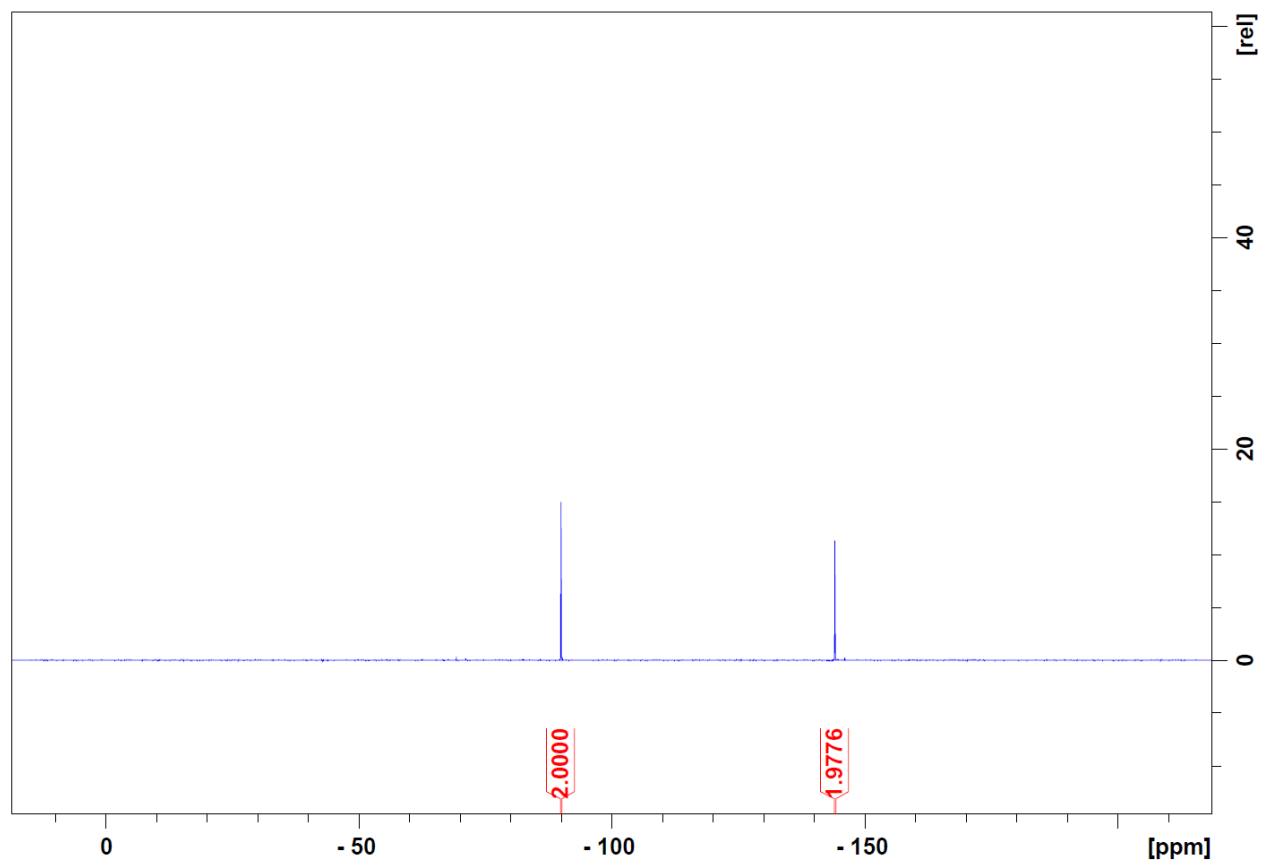
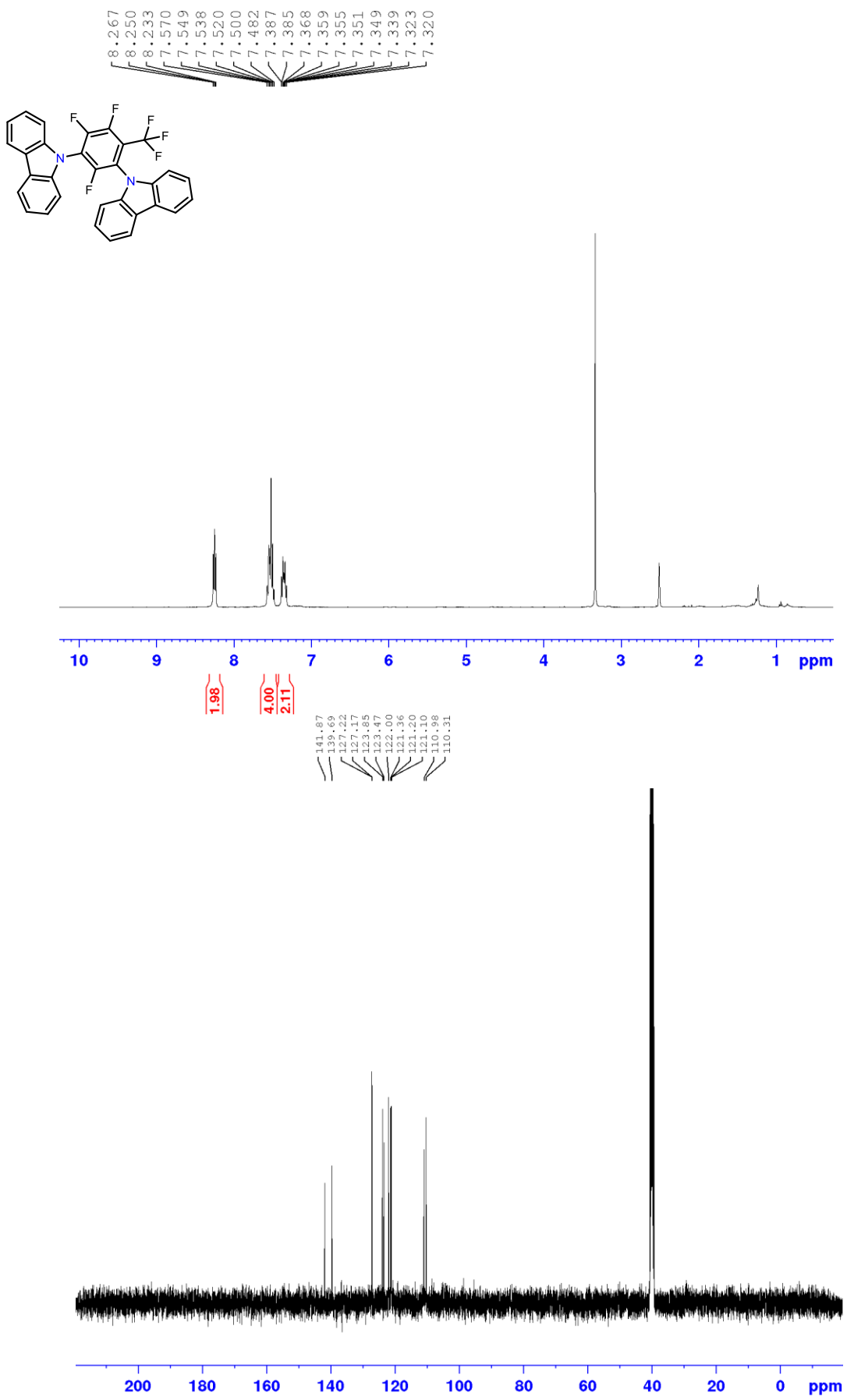


Figure S11: ^1H , ^{13}C & ^{19}F NMR spectra of product **10**.



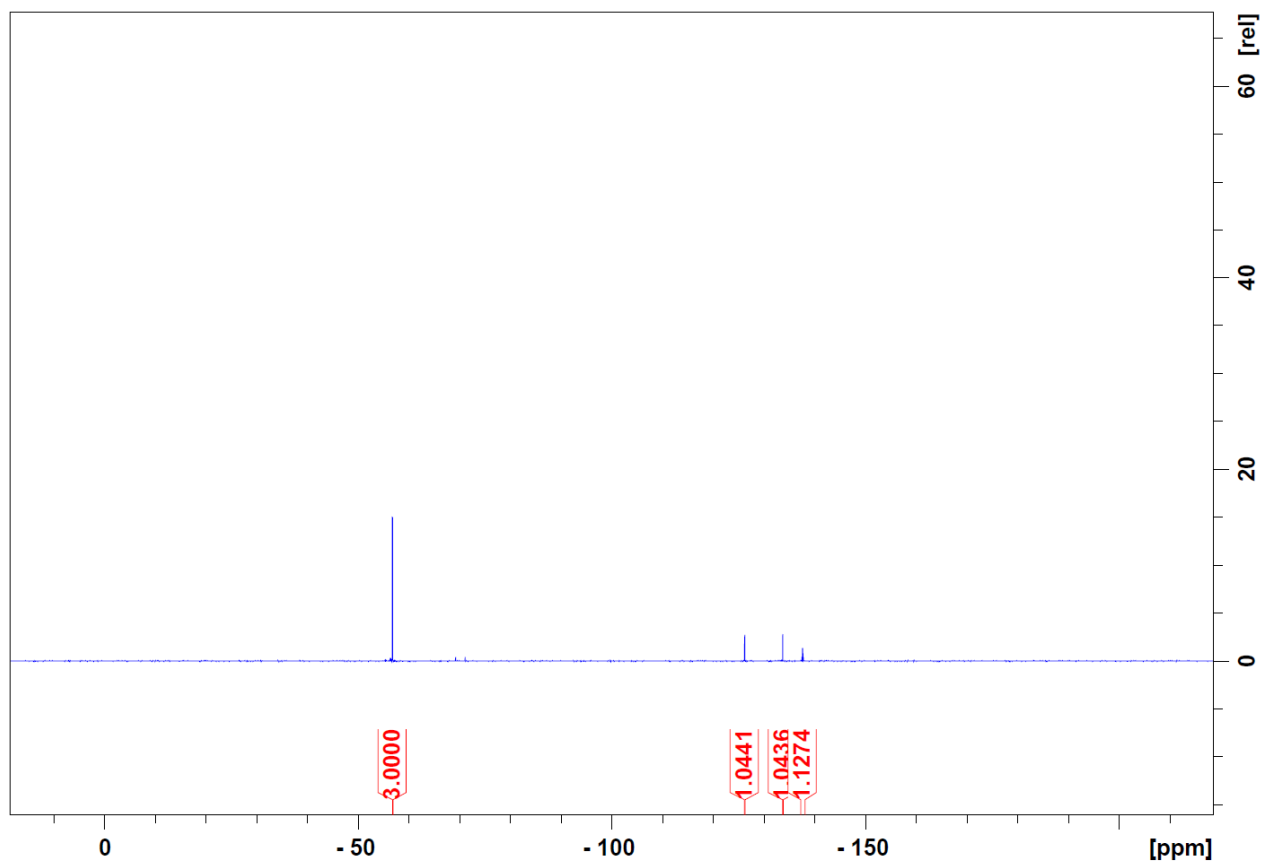


Figure S12: ^1H , ^{13}C & ^{19}F NMR spectra of product **11**.

References

1. Chen, Q., Mao, Z., Guo, F., Liu, X., *Tetrahedron Lett.*, 2016, **57**, 3735-3738.
2. Jordan-Hore, J., Johansson, C., Guillas Costa, M., Beck, E., Gaunt, M., *J. Am. Chem. Soc.*, 2008, **48**, 16184-16186.
3. Kwong, F., Lau, C., Choy, P., *J. Org. Chem.*, 2011, **76**, 80-84.
4. Taylor, J. E., Jones, M. D., Williams, J. M. J., Bull, S. D., *Org. Lett.*, 2010, **12**, 5740-5743.
5. Feng, Y., Shen, W., Huang, W., Tan, W., Lu, C., Cui, Y., Tian, H., *J. Phys. Org. Chem.*, 2007, **20**, 968-974.
6. Jiang, H., Zhang, W., Li, P., Want, J., Dong, C., Zhang, K., Chen, H., Du, Z., *Bioorg. Med. Chem. Lett.*, 2018, **28**, 1320-1323.
7. Kitawaki, T., Hayashi, Y., Ueno, A., Chida, N., *Tetrahedron*, 2006, **62**, 6792-6801.
8. Dissanayake, D. M. M. M.; Vannucci, A. K., *Org. Lett.*, 2019, **21**, 457-460.