

Ionic hydrogenation of naphthyl tetrafluoropyridin-4-yl ethers as a new route to 5,6,7,8-tetrahydronaphthols

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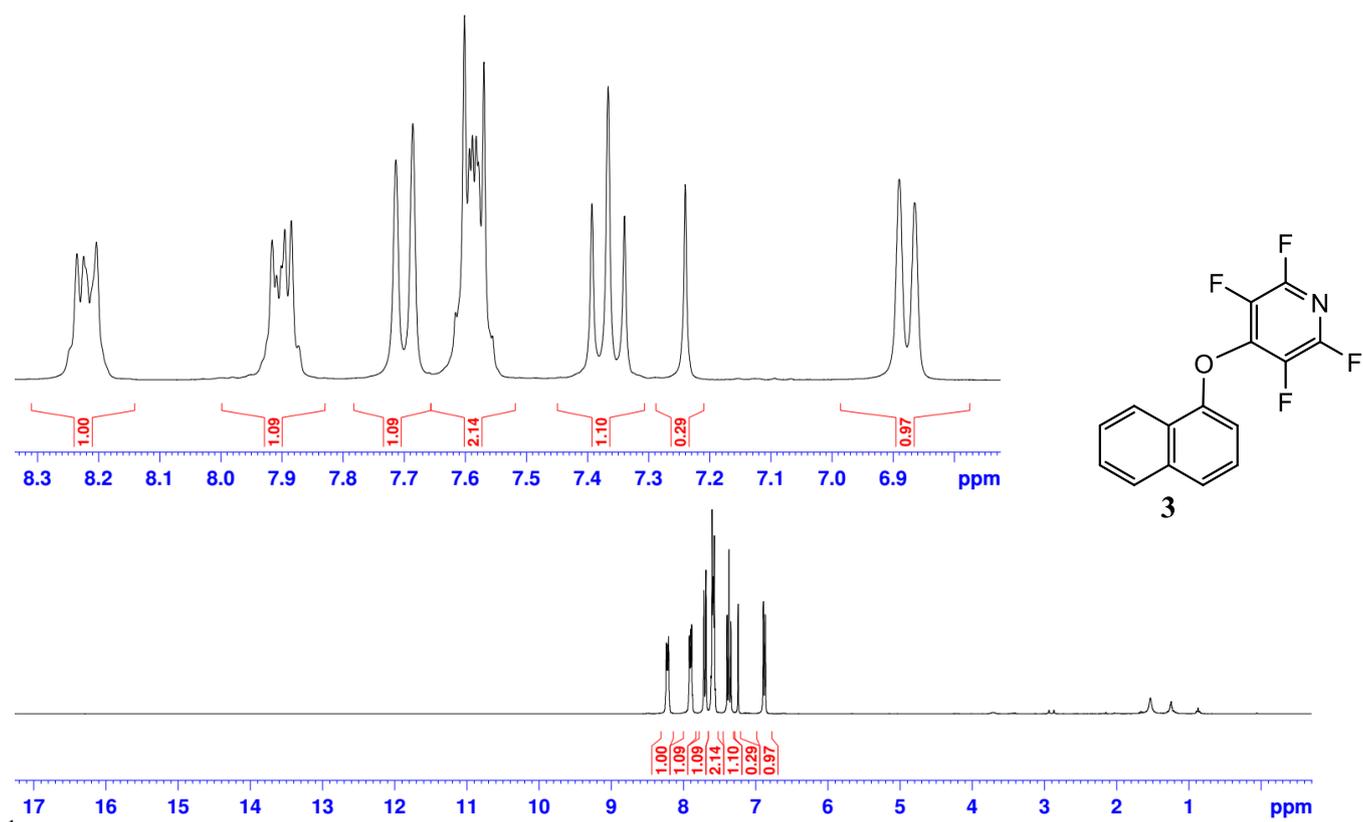
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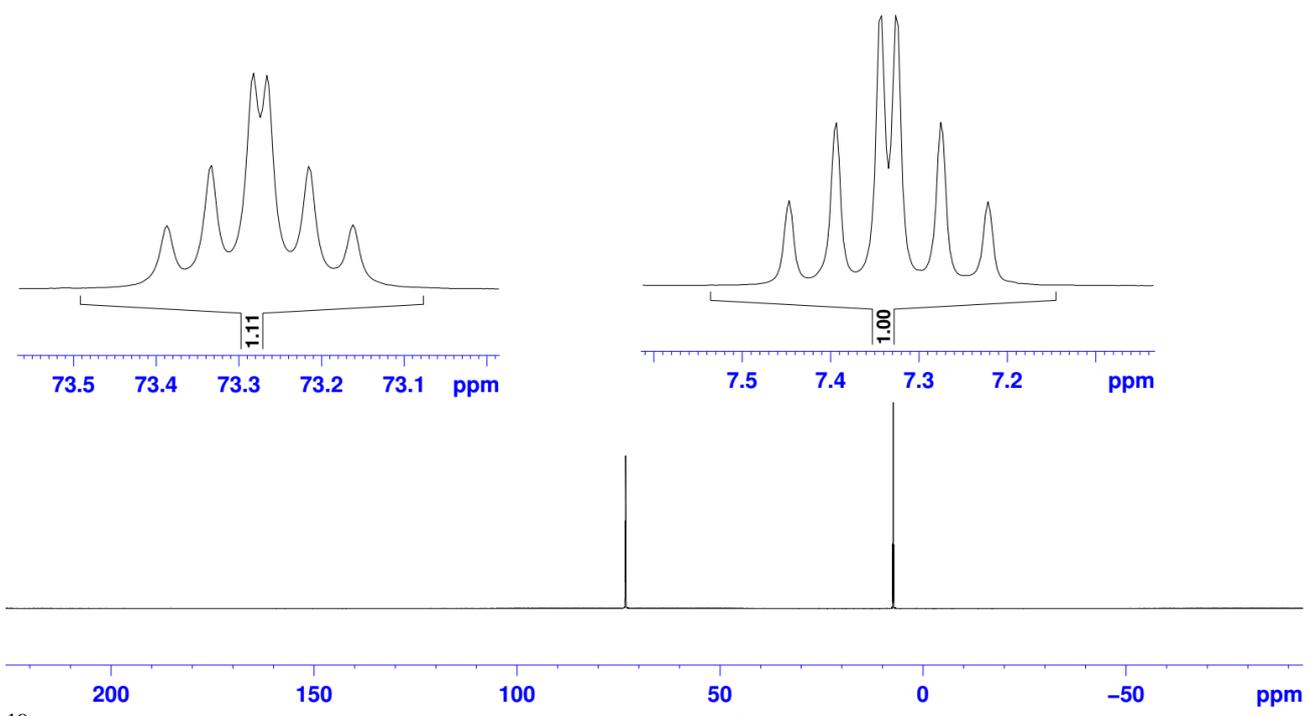
General. The ¹H, ¹³C and ¹⁹F NMR spectra were recorded on a Bruker AVANCE III 500 spectrometer at 500.13, 125.77 and 470.59 MHz, respectively. The ¹H and ¹³C NMR spectra were referenced on the solvent signals (CDCl₃ δ_H 7.26 ppm and δ_C 77 ppm). The ¹⁹F NMR spectra were referenced on external C₆F₆. High-resolution mass spectra (HRMS) were acquired on a double-focusing high-resolution quadrupole time-of-flight mass spectrometer equipped with a high-performance toroidal ESA.

2,3,5,6-Tetrafluoro-4-(naphthalen-1-yloxy)pyridine 3 (typical procedure). Pentafluoropyridine (1.21 g, 7.2 mmol) was added dropwise to a stirred mixture of 1-naphthol (1 g, 7 mmol) and powdered K₂CO₃ (1.11 g, 8 mmol) in DMF (15 ml). The resulting mixture was stirred at 20 °C for 1 h (TLC control), and then poured into water (100 ml). The precipitated white crystals were filtered off, washed with water and dried to obtain pure product **3** (2.04 g, 99%), mp 78–79 °C (subl.). HRMS, *m/z*: 293.0460 (calc. for C₁₅H₇F₄NO, *m/z*: 293.0458). ¹H NMR δ: 6.87 (d, 1H, *J* 7.5 Hz), 7.37 (dd, 1H, *J* 7.5, 2.4 Hz), 7.5–7.6 (m, 2H), 7.70 (d, 1H, *J* 7.5), 7.8–7.9 (m, 1H), 8.2–8.3 (m, 1H). ¹⁹F NMR δ: 7.33 (m, 2F), 73.28 (m, 2F).

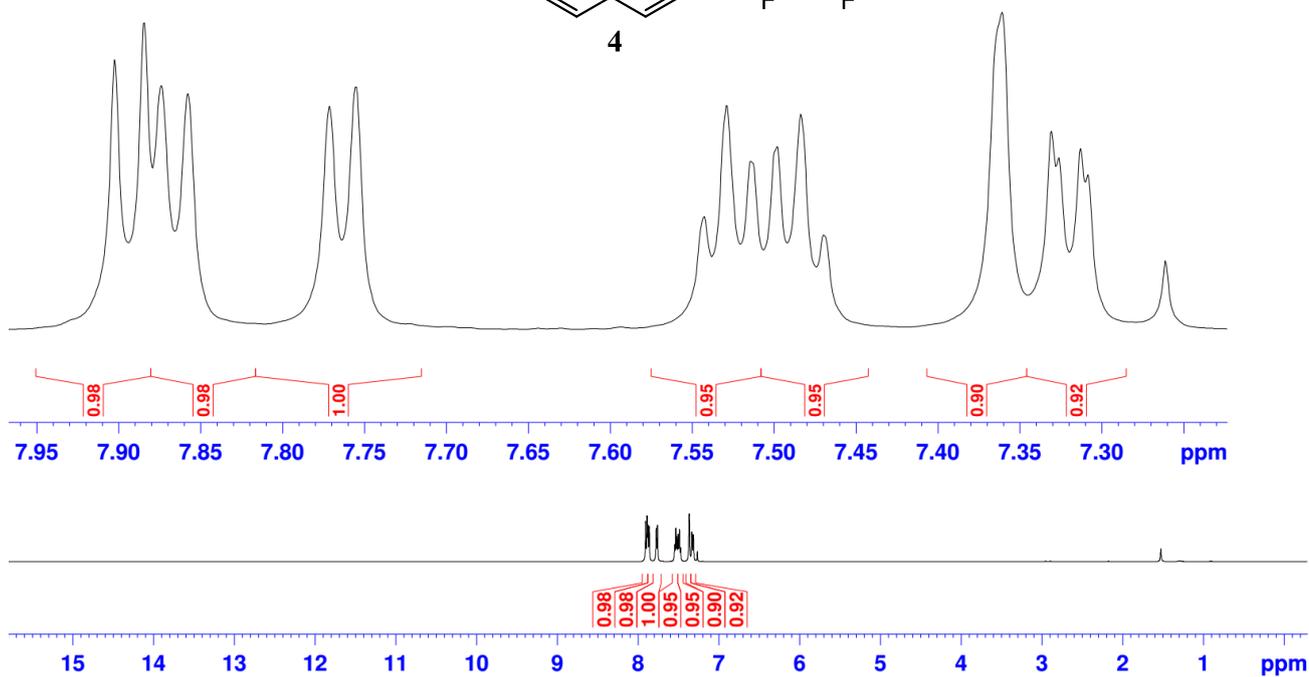
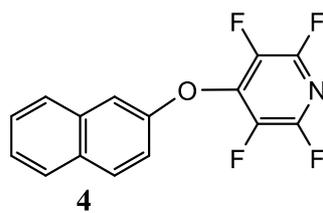
2,3,5,6-Tetrafluoro-4-(naphthalen-2-yloxy)pyridine 4. Mp 89–90 °C (subl.). HRMS, *m/z*: 293.0461 (calc. for C₁₅H₇F₄NO, *m/z*: 293.0458). ¹H NMR δ: 7.32 (dd, 1H, *J* 8, 2.5 Hz), 7.36 (d, 1H, *J* 2.5 Hz), 7.48 (t, 1H, *J* 7.7 Hz), 7.53 (t, 1H, *J* 7.7 Hz), 7.76 (d, 1H, *J* 8.2), 7.87 (d, 1H, *J* 7.7), 7.89 (d, 1H, *J* 7.7). ¹⁹F NMR δ: 10.37 (m, 2F), 76.09 (m, 2F).



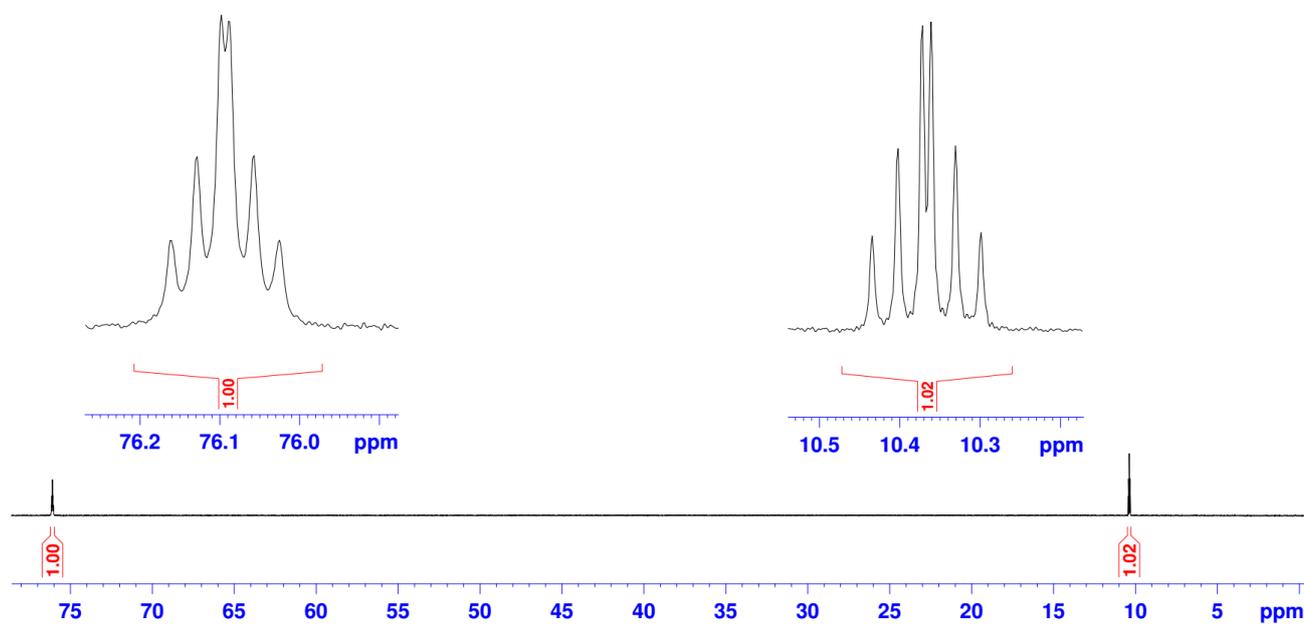
¹H NMR spectrum of compound **3** (500.13 MHz, CDCl₃)



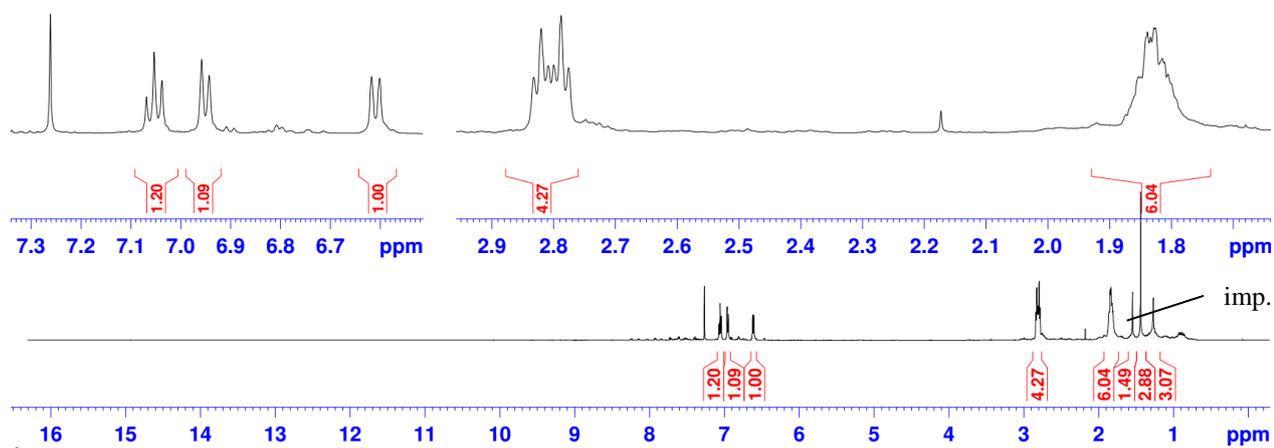
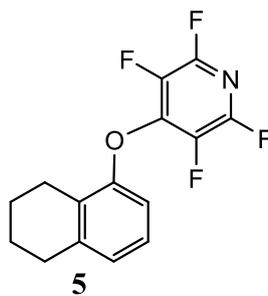
¹⁹F NMR spectrum of compound **3** (470.59 MHz, CDCl₃)



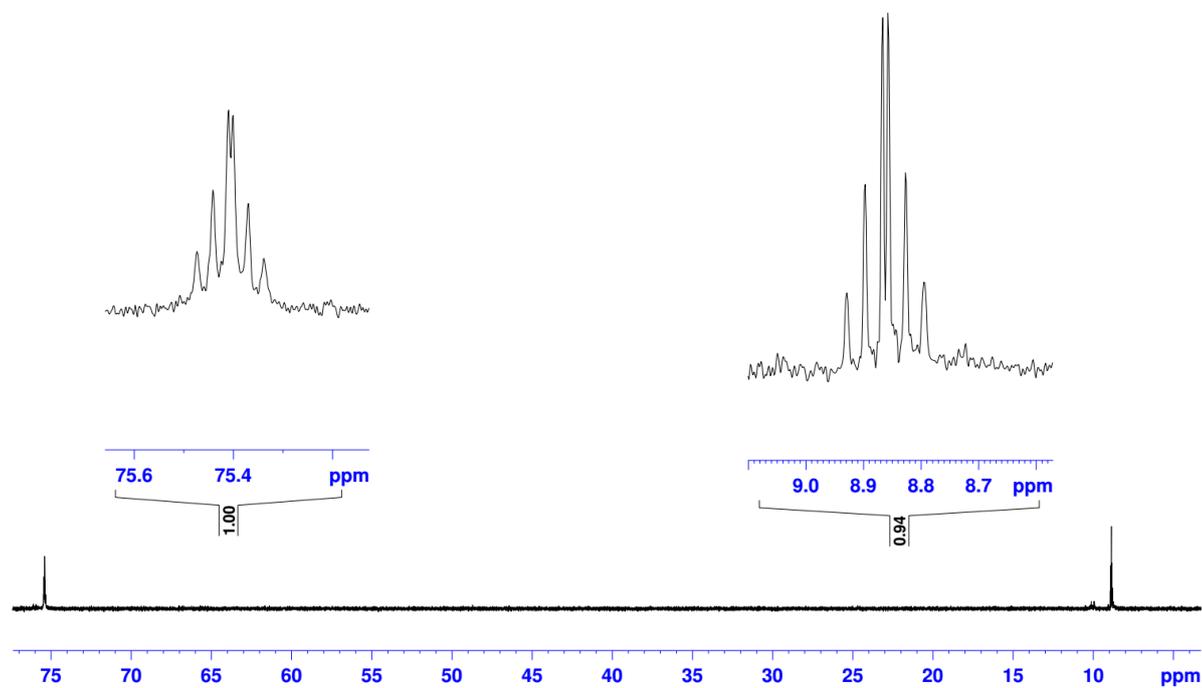
¹H NMR spectrum of compound **4** (500.13 MHz, CDCl₃)



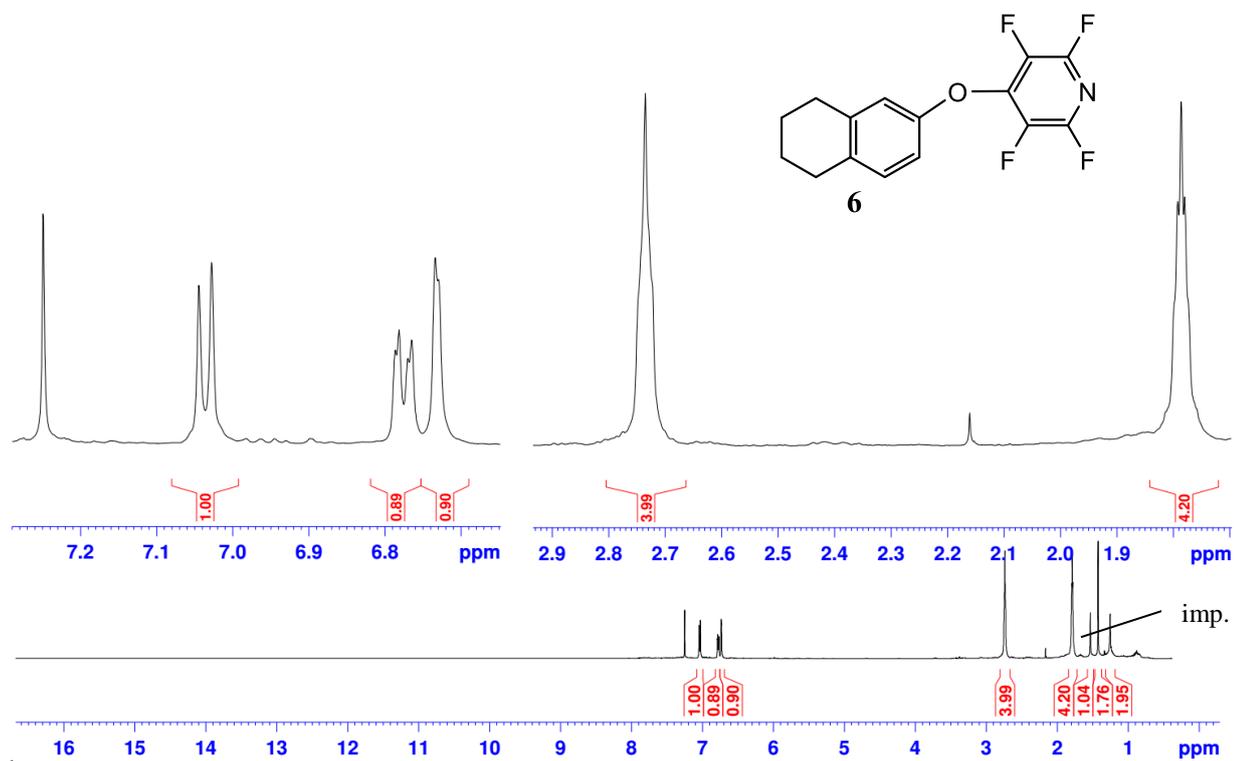
¹⁹F NMR spectrum of compound **4** (470.59 MHz, CDCl₃)



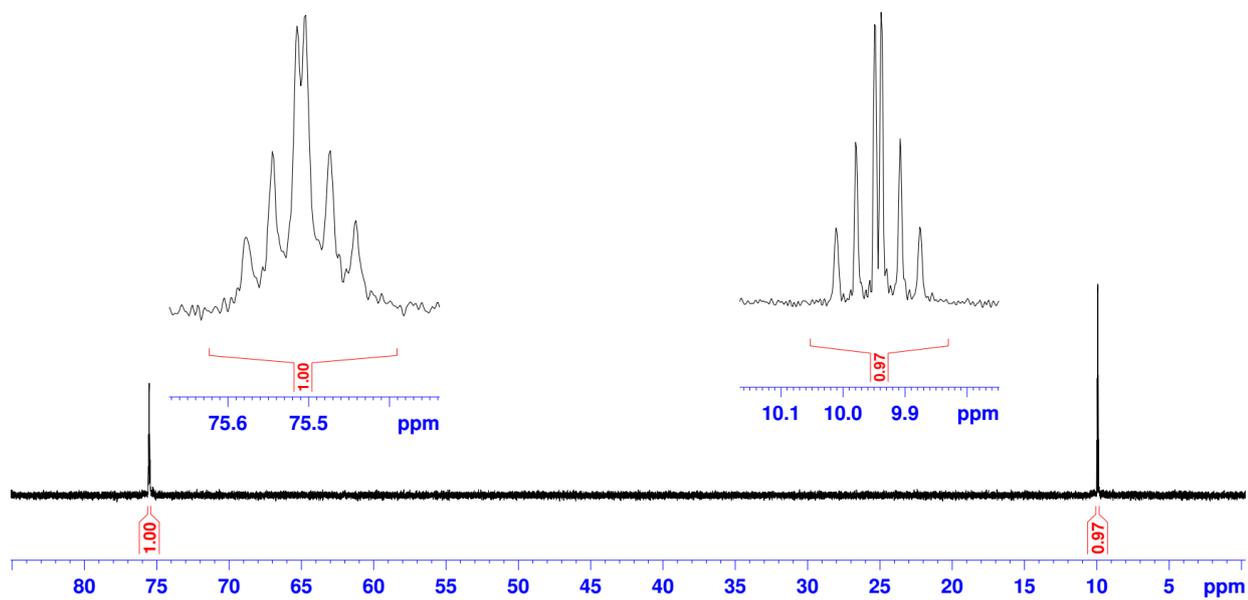
¹H NMR spectrum of compound **5** (500.13 MHz, CDCl₃)



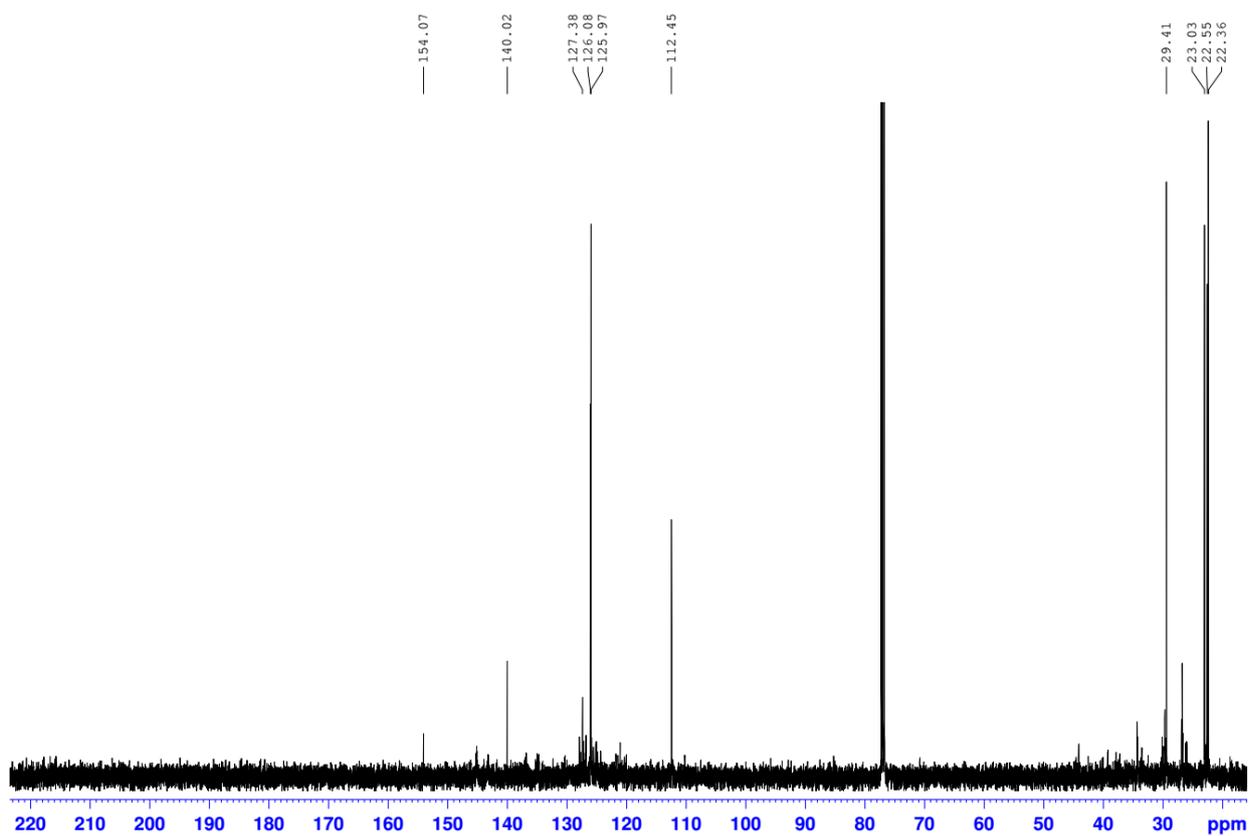
¹⁹F NMR spectrum of compound **5** (470.59 MHz, CDCl₃)



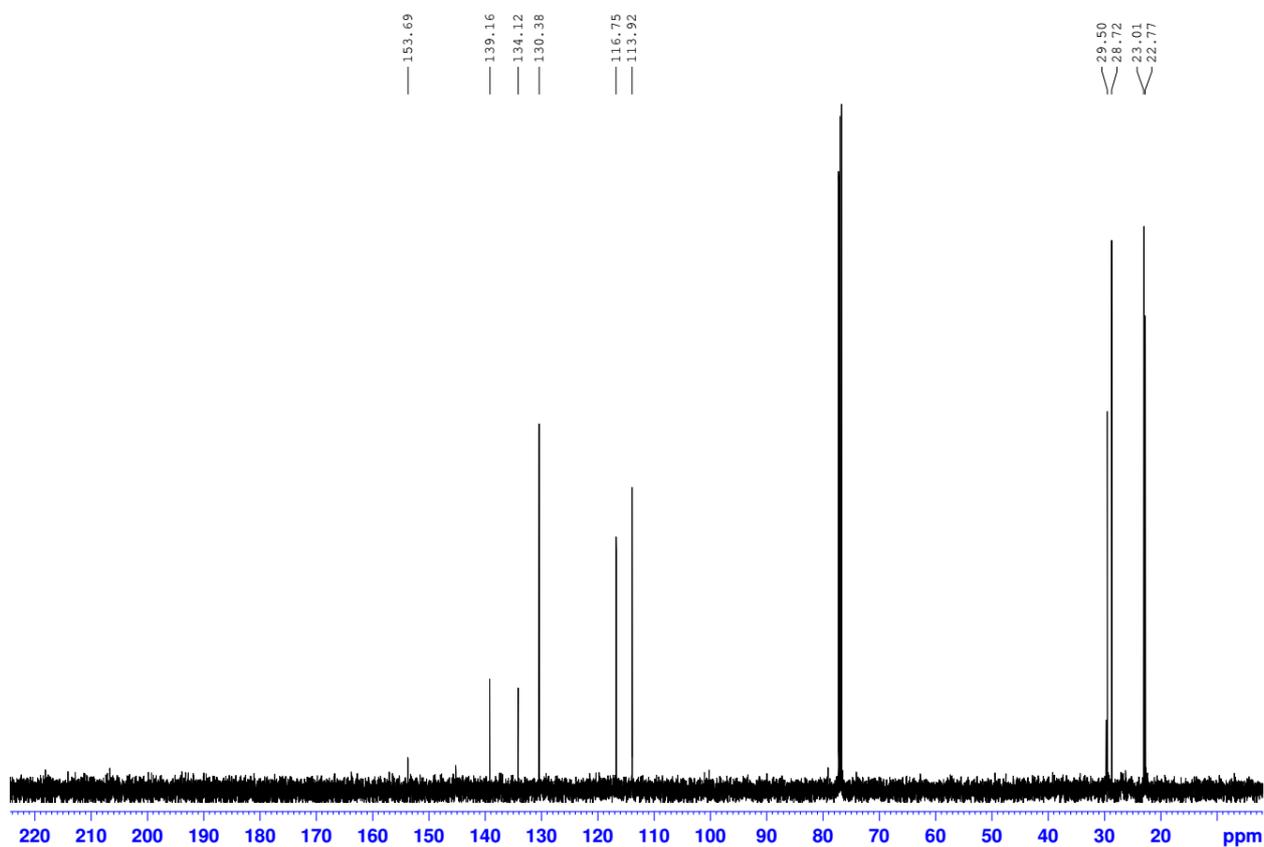
¹H NMR spectrum of compound **6** (500.13 MHz, CDCl₃)



¹⁹F NMR spectrum of compound **6** (470.59 MHz, CDCl₃)



^{13}C NMR spectrum of compound **5** (125.77 MHz, CDCl_3)



^{13}C NMR spectrum of compound **6** (125.77 MHz, CDCl_3)