

Supporting Information for:

C-H Addition Reactivity of 2-Phenylpyridine and 2,2'- Bipyridine with Pentaphenylborole

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Figure S-1: ^1H NMR spectrum of **1** in CDCl_3 .

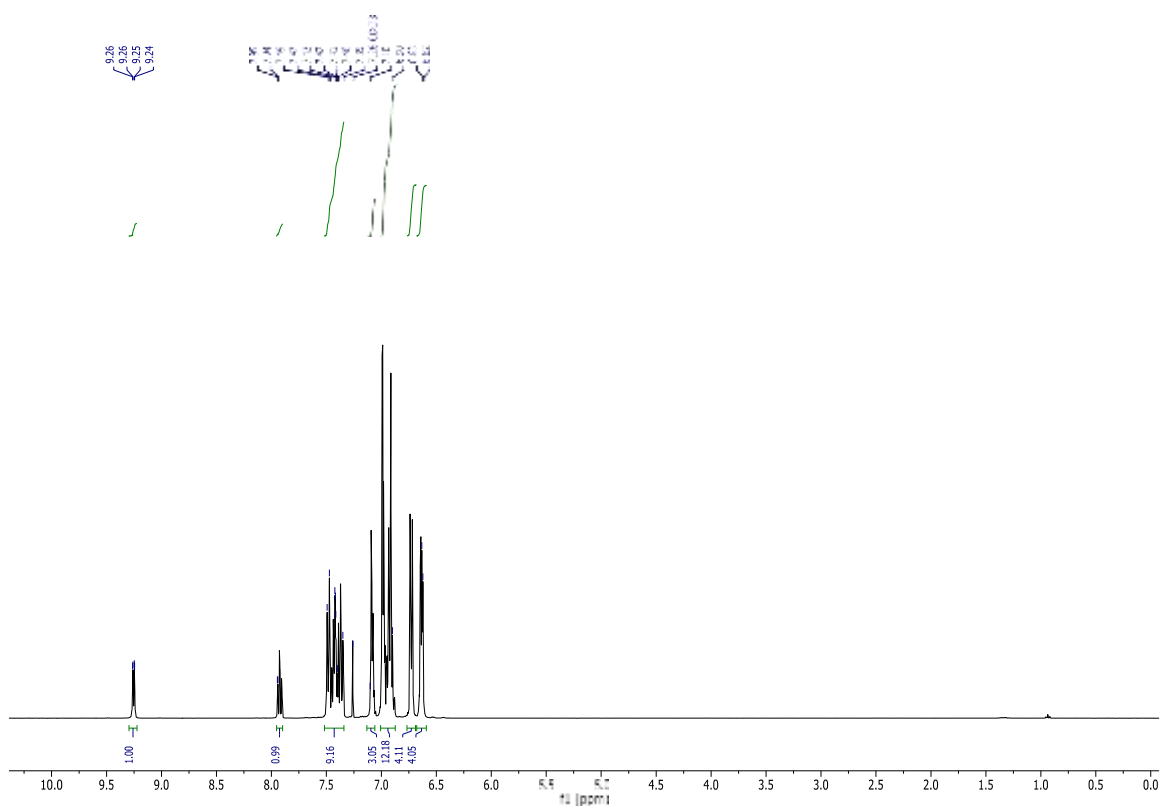


Figure S-2: Expansion of ^1H NMR spectrum of **1** in CDCl_3 (aryl region).

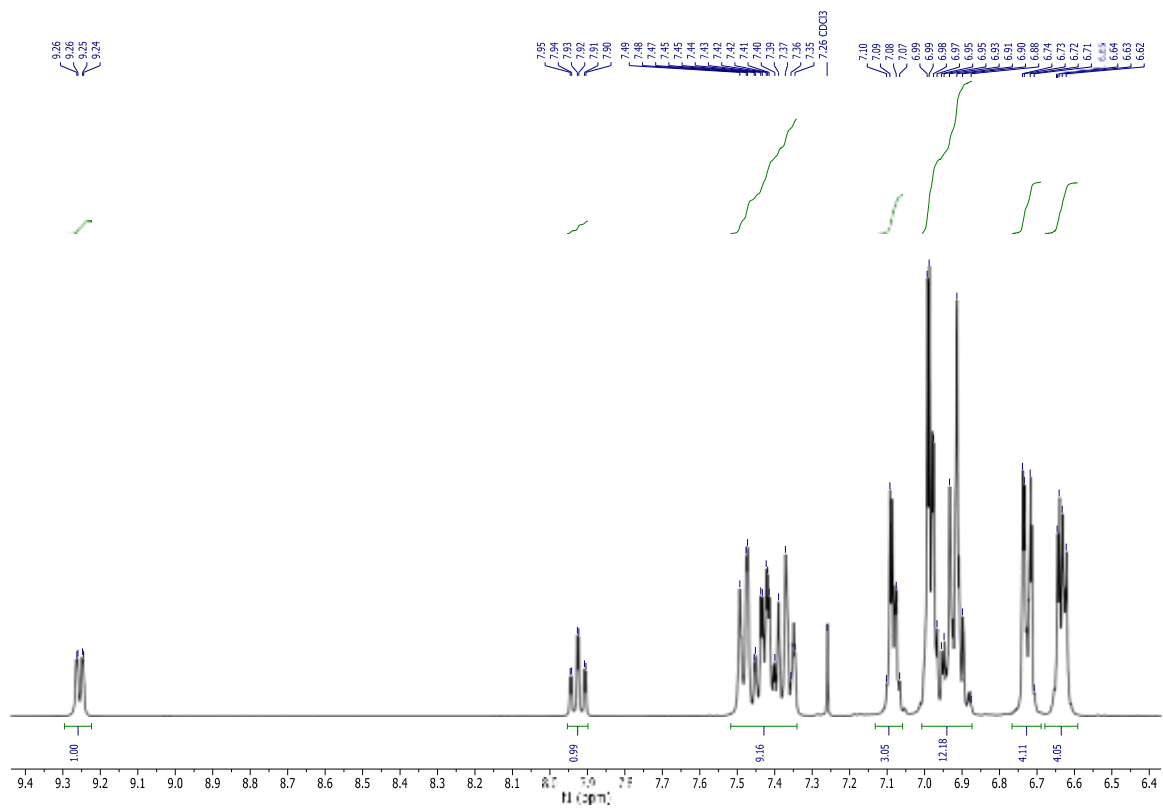


Figure S-3: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **1** in CDCl_3 .

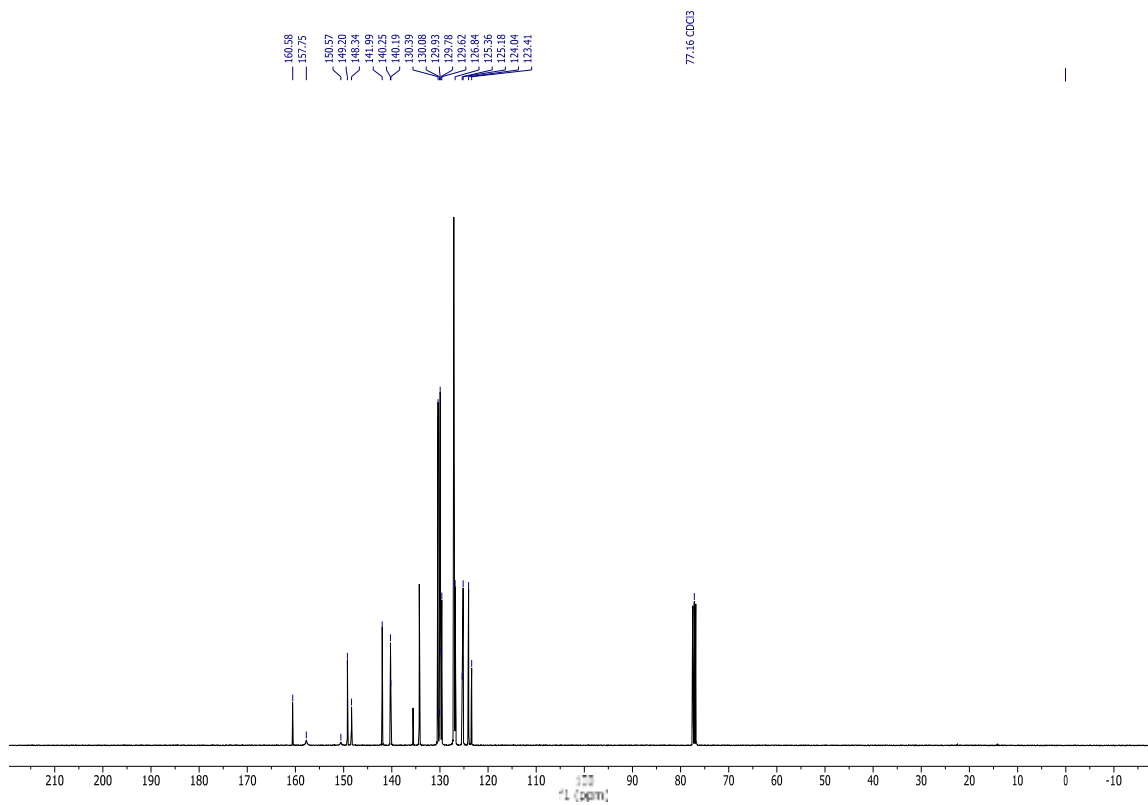


Figure S-4: Expansion of $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **1** in CDCl_3 (aryl region).

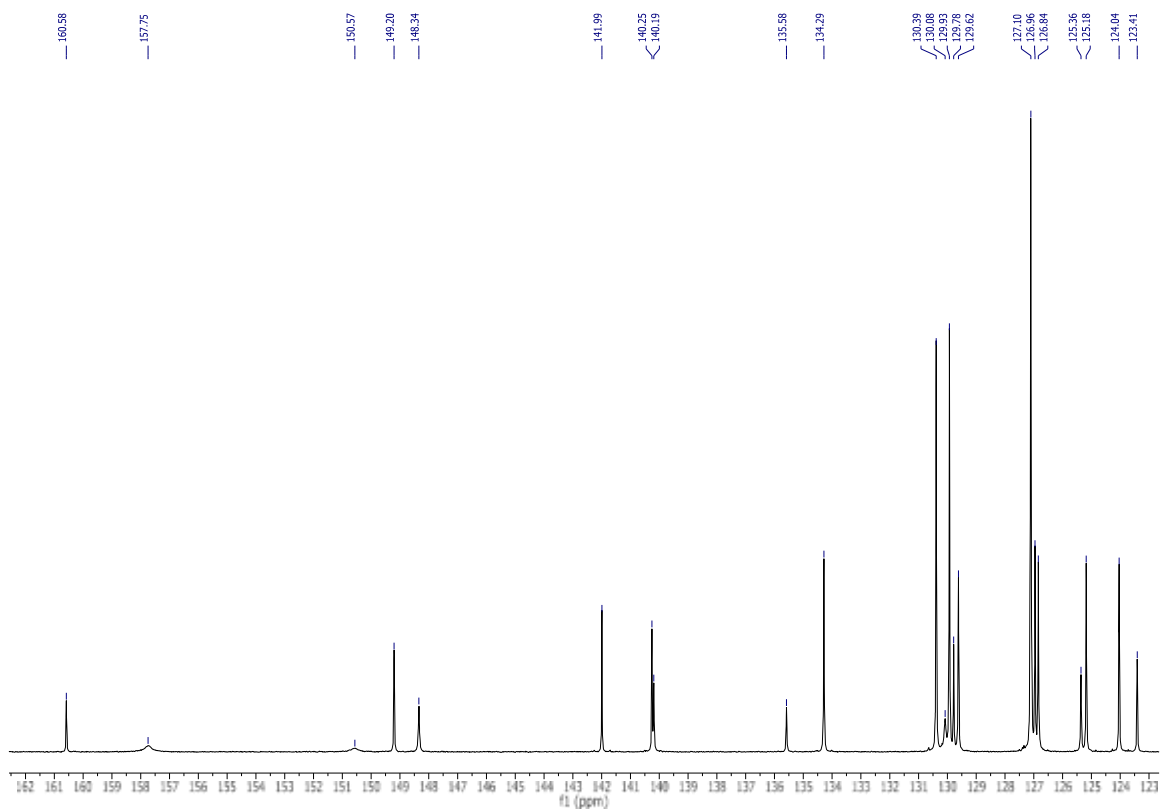


Figure S-5: $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **1** in CDCl_3 .

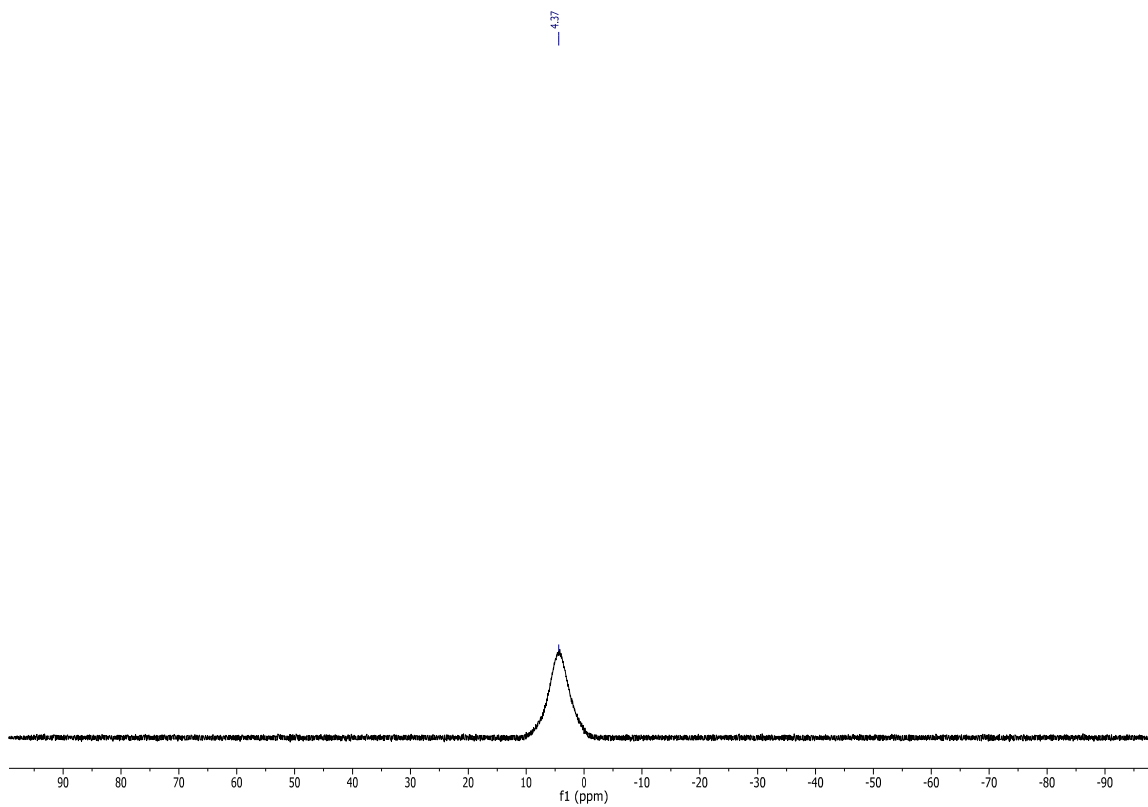


Figure S-6: FT-IR spectrum of **1**.

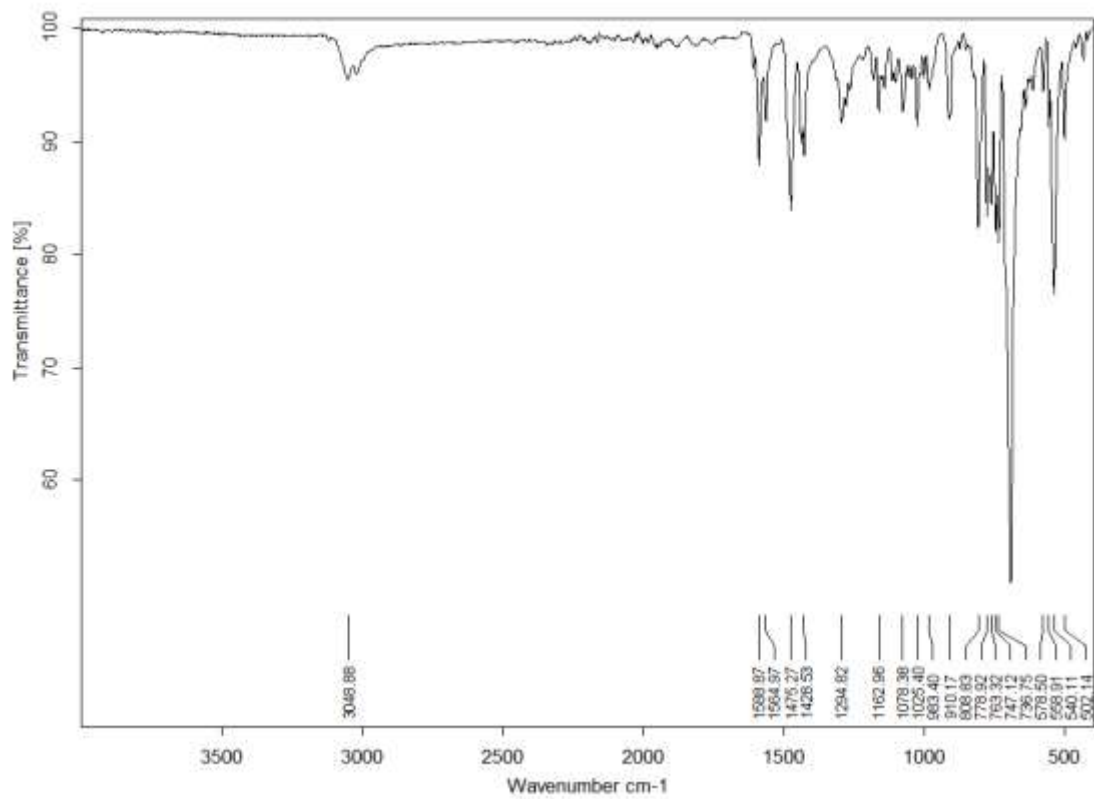


Figure S-7: Stacked plot of ^1H NMR spectrum of **2** at 25 °C and -80 °C in CD_2Cl_2 (\bullet CD_2Cl_2).

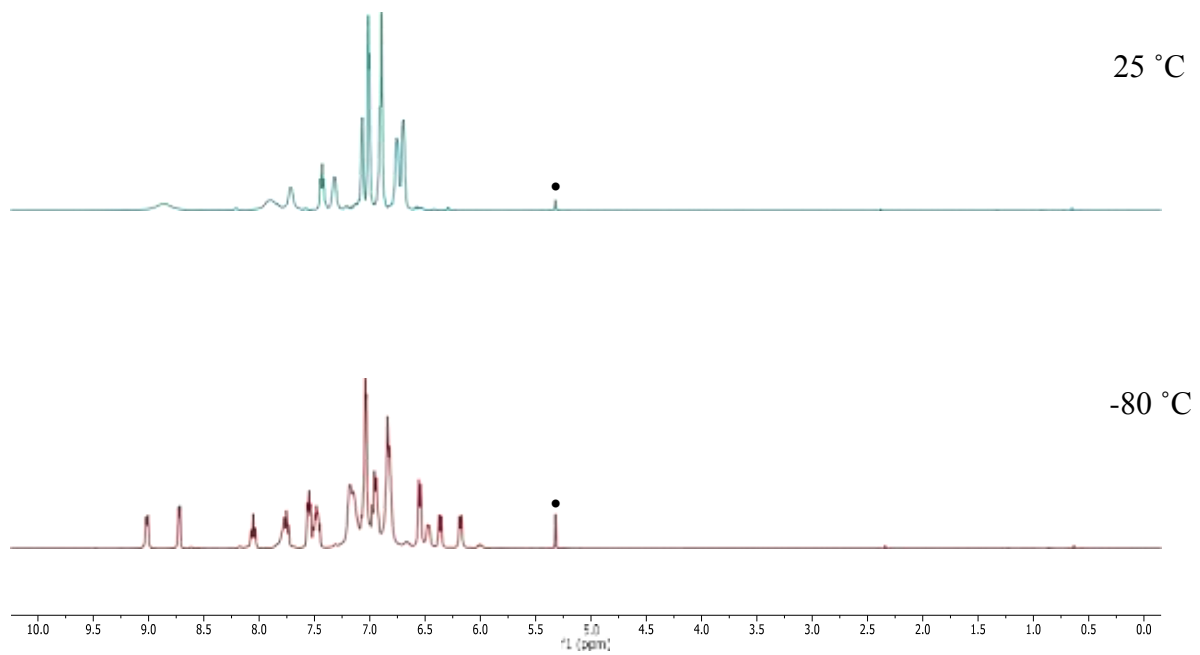


Figure S-8: ^1H NMR spectrum of **2** in CD_2Cl_2 at -80°C .

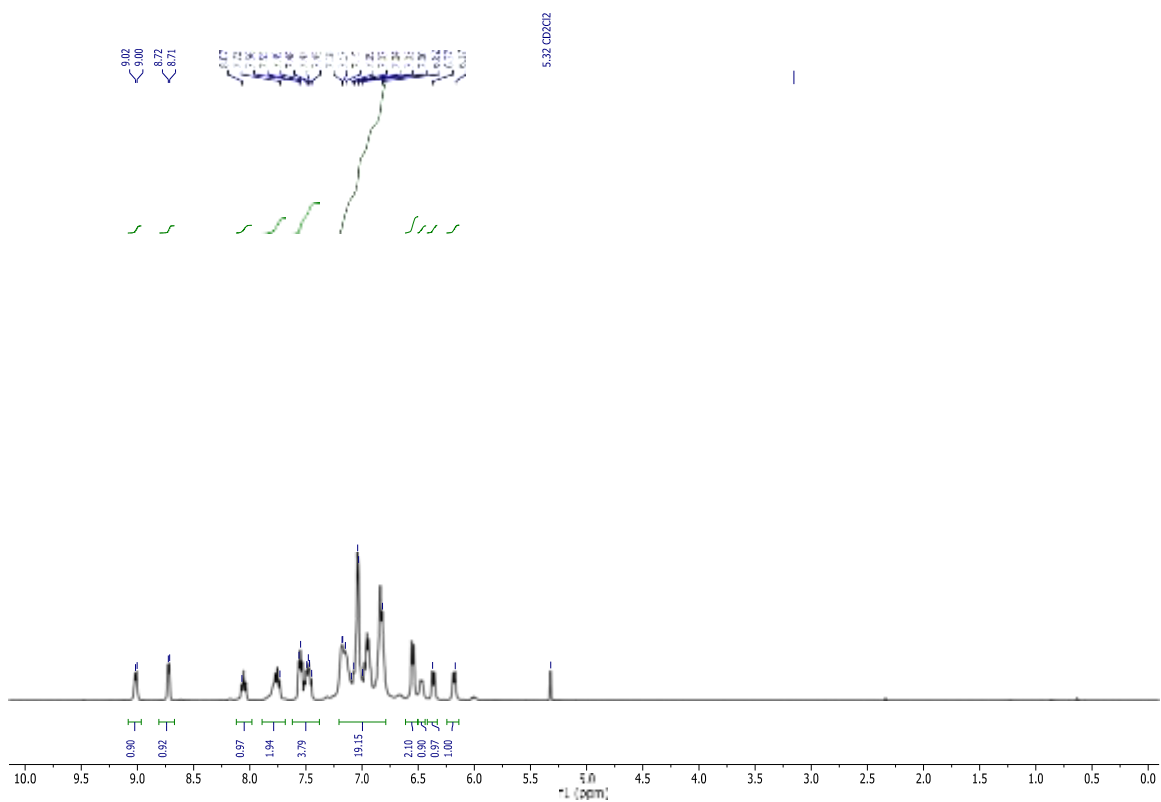


Figure S-9: Expansion of ^1H NMR spectrum of **2** in CD_2Cl_2 at $-80\text{ }^\circ\text{C}$ (aryl region).

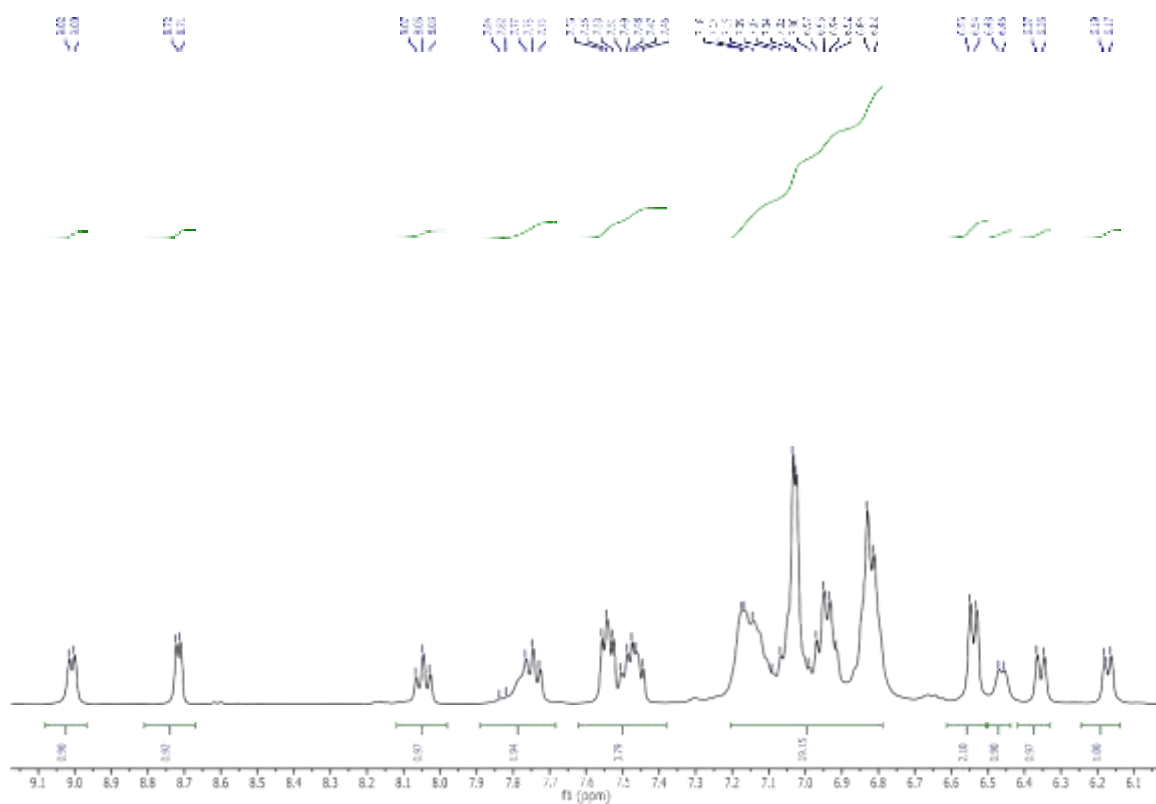


Figure S-10: ^1H NMR spectrum of **2** in CDCl_3 .

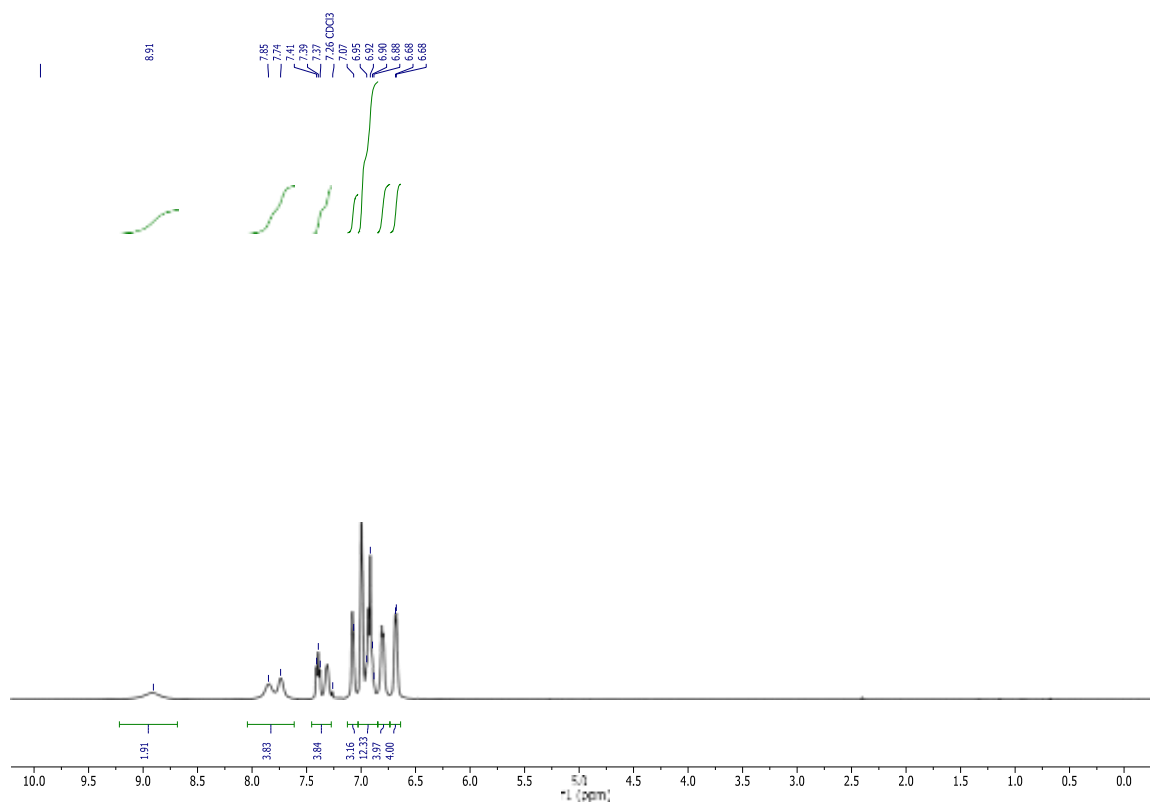


Figure S-11: Expansion of ^1H NMR spectrum of **2** in CDCl_3 (aryl region).

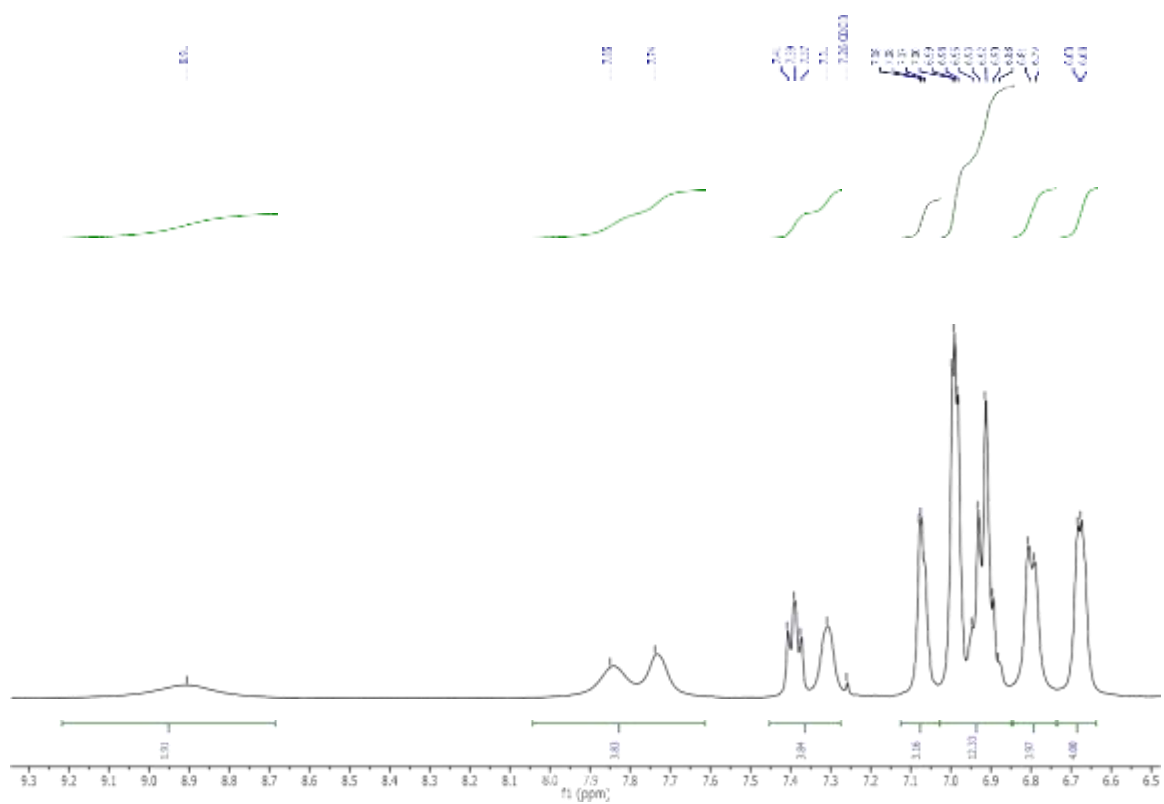


Figure S-12: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2** in CDCl_3 .

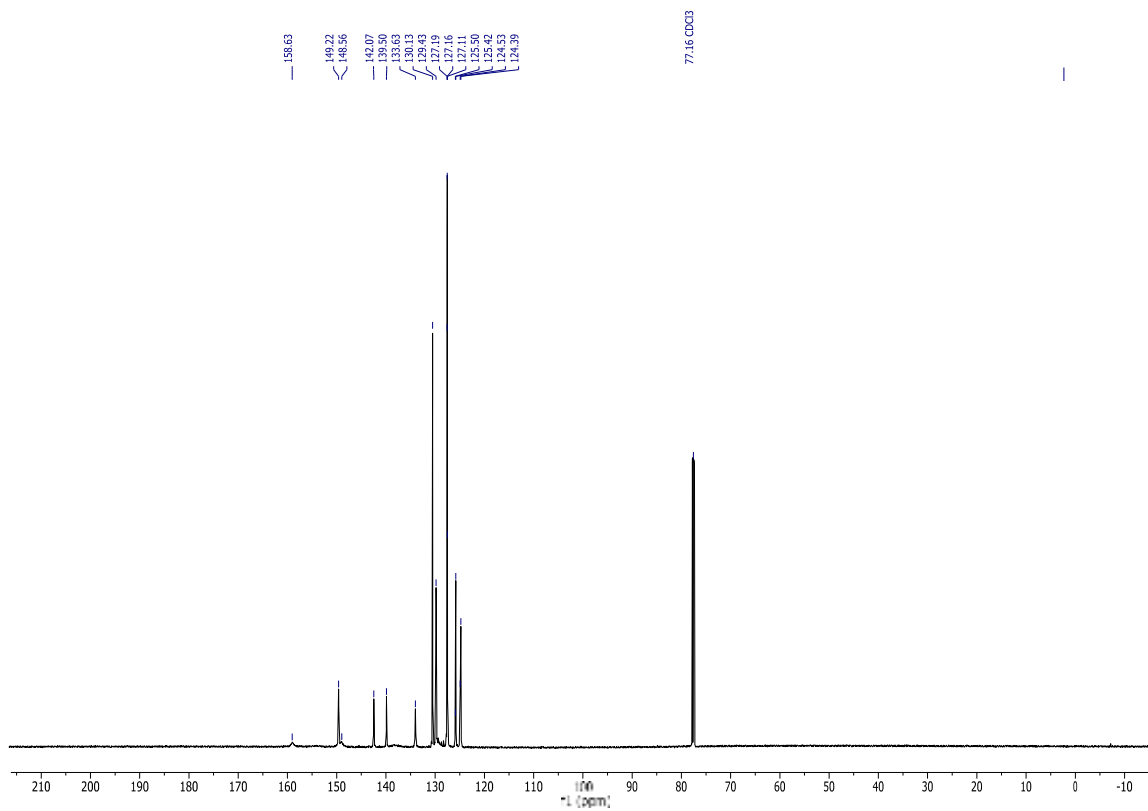


Figure S-13: Expansion of $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2** in CDCl_3 (aryl region).

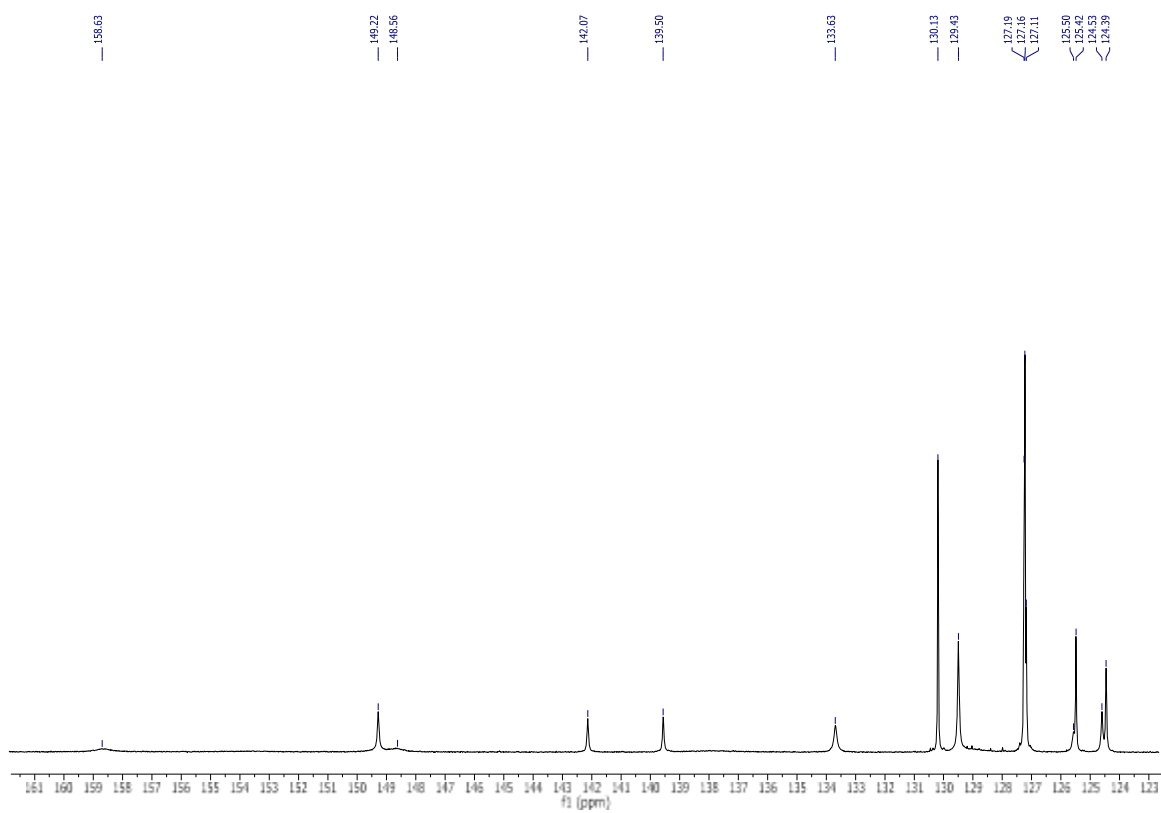


Figure S-14: $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **2** in CDCl_3 .

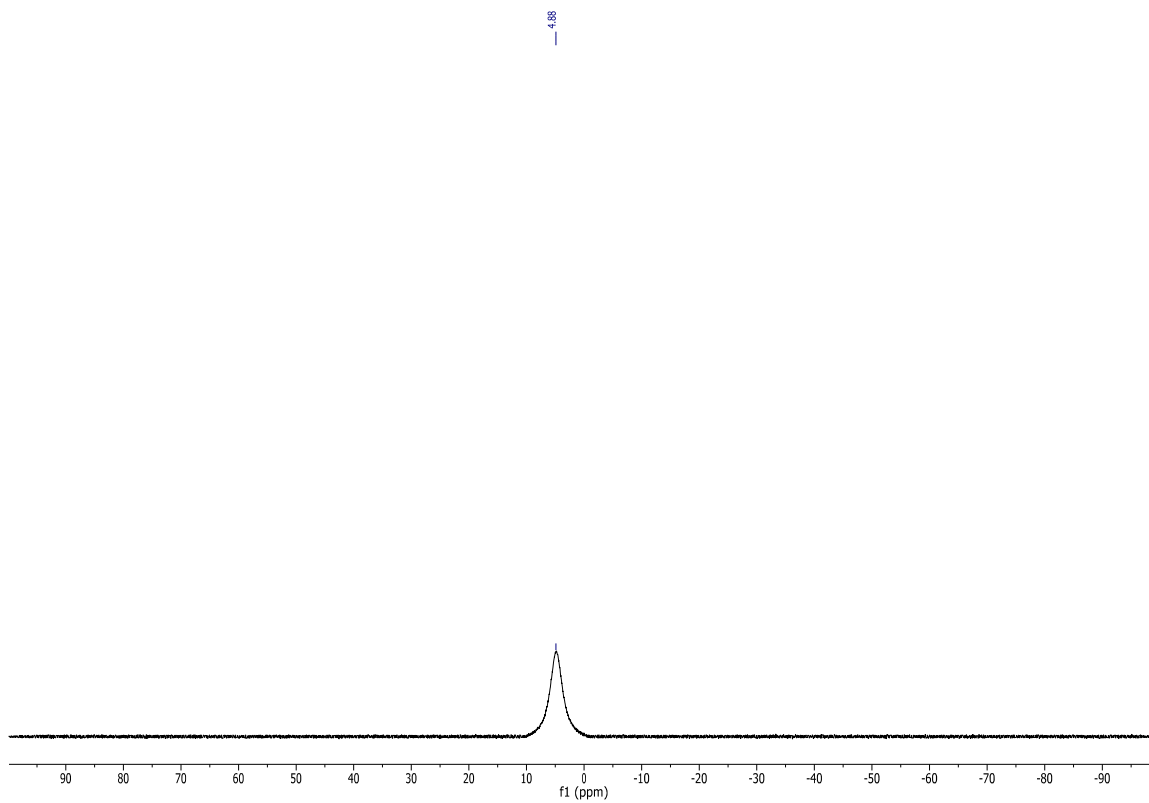


Figure S-15: FT-IR spectrum of **2**.

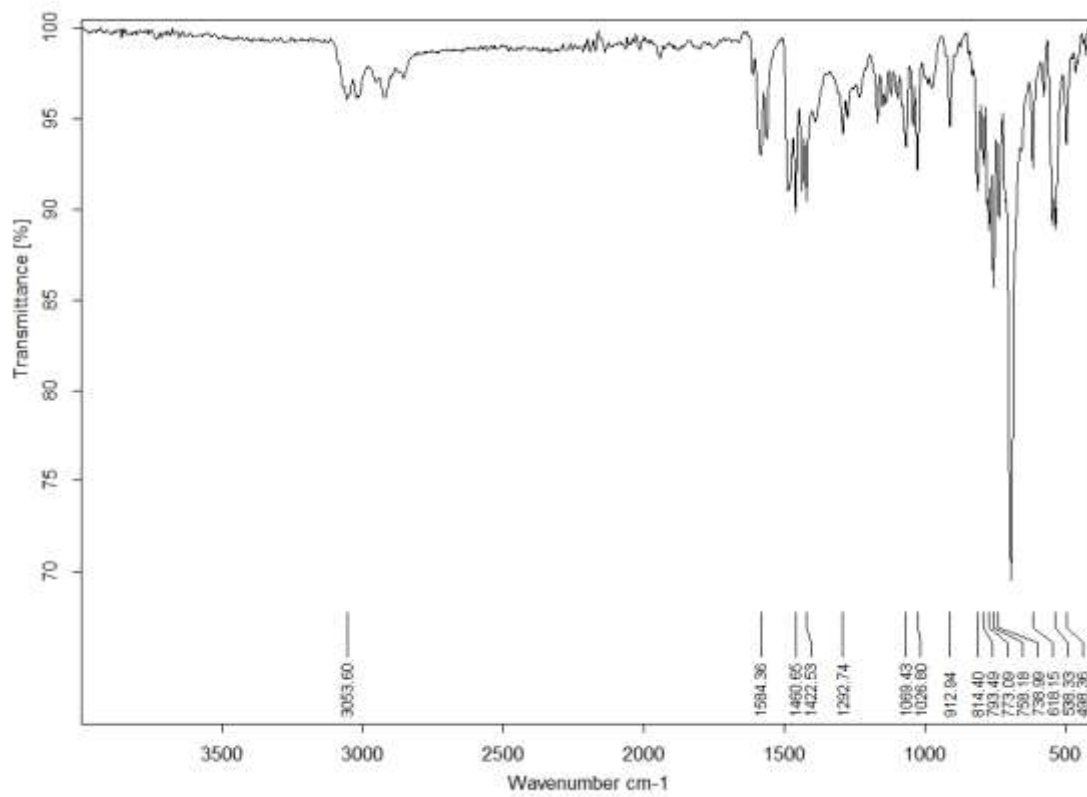


Figure S-16: Stacked plot of ^1H NMR spectrum of *syn-3* at 25 °C and -70 °C in toluene- d_8 (• toluene- d_8).

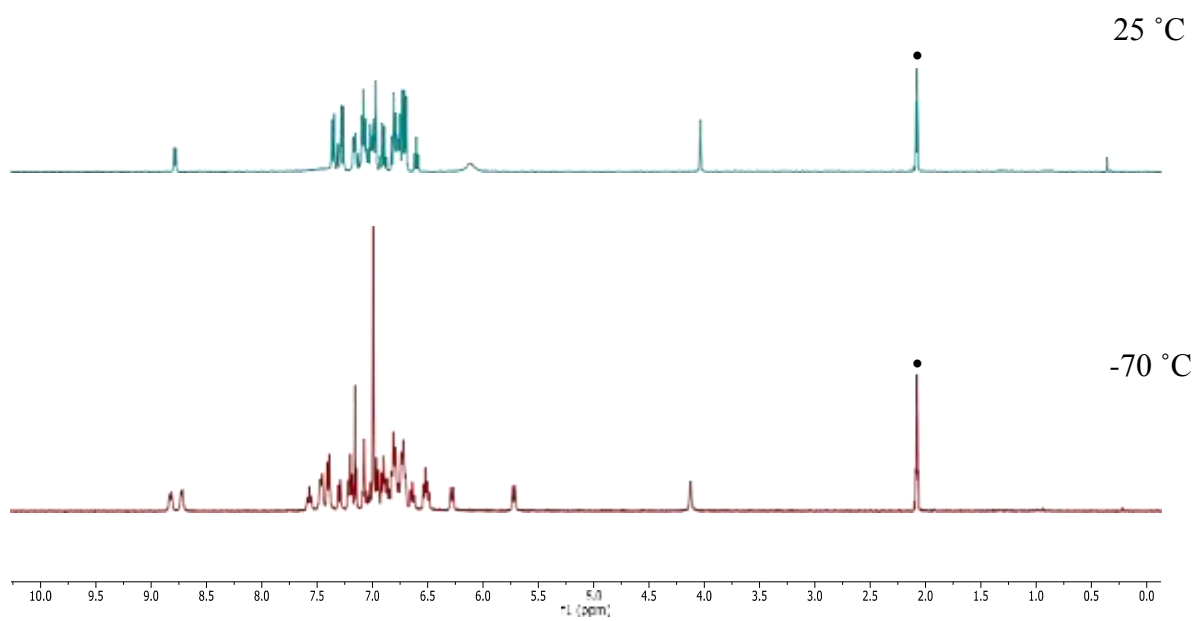


Figure S-17: ^1H NMR spectrum of *syn-3* in toluene- d_8 at $-70\text{ }^\circ\text{C}$.

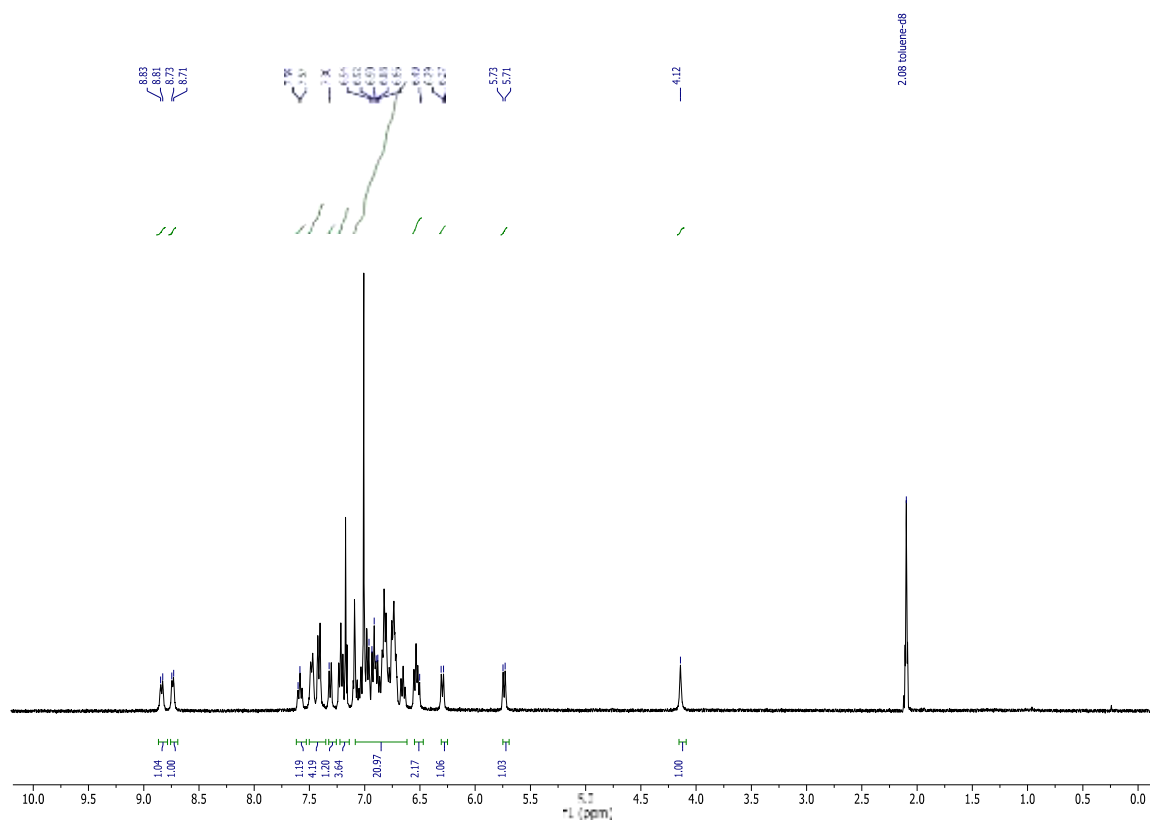


Figure S-18: Expansion of ^1H NMR spectrum of *syn-3* in toluene- d_8 (aryl region) at $-70\text{ }^\circ\text{C}$.

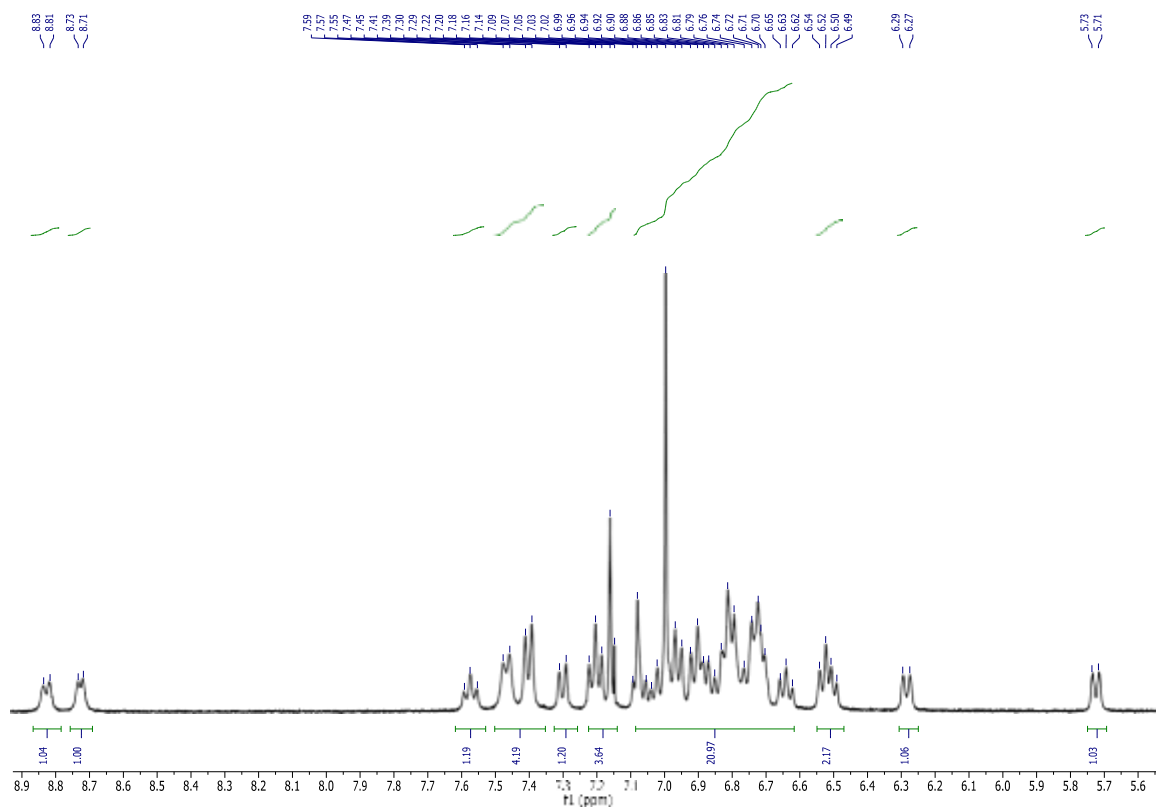


Figure S-19: ^1H NMR spectrum of *syn-3* in CDCl_3 .

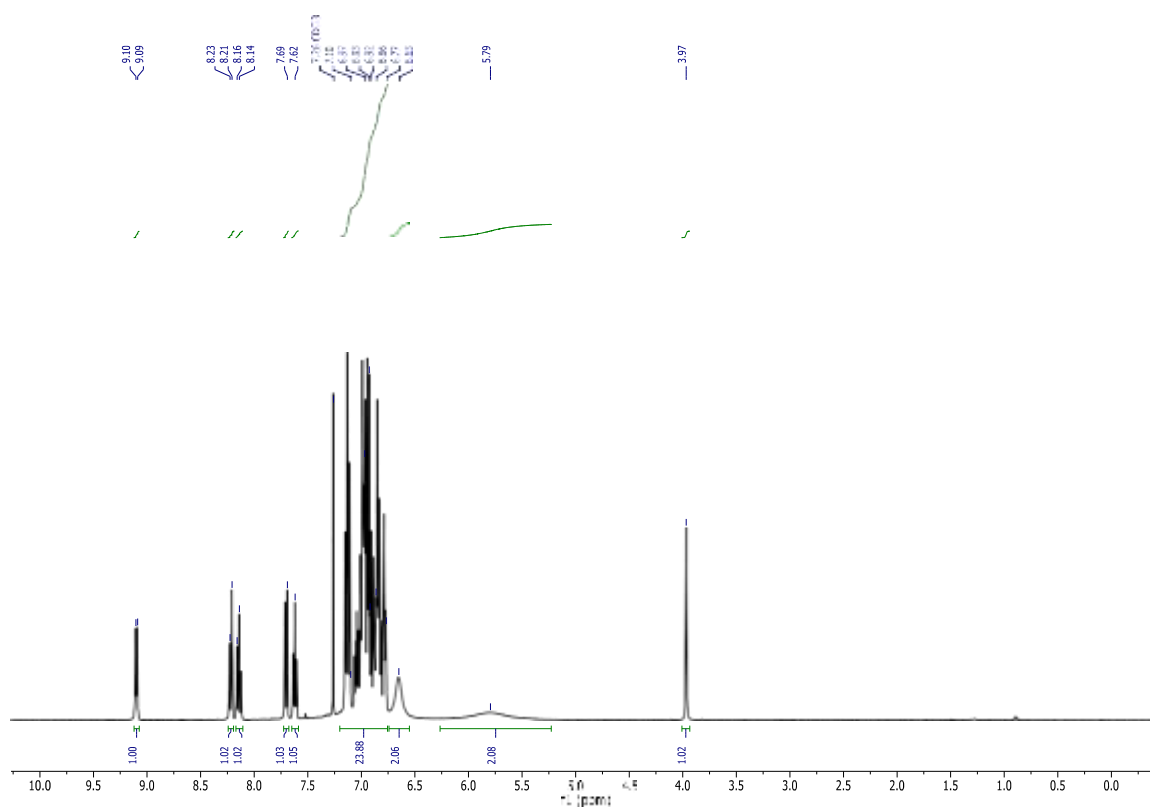


Figure S-20: Expansion of ^1H NMR spectrum of *syn-3* in CDCl_3 (aryl region).

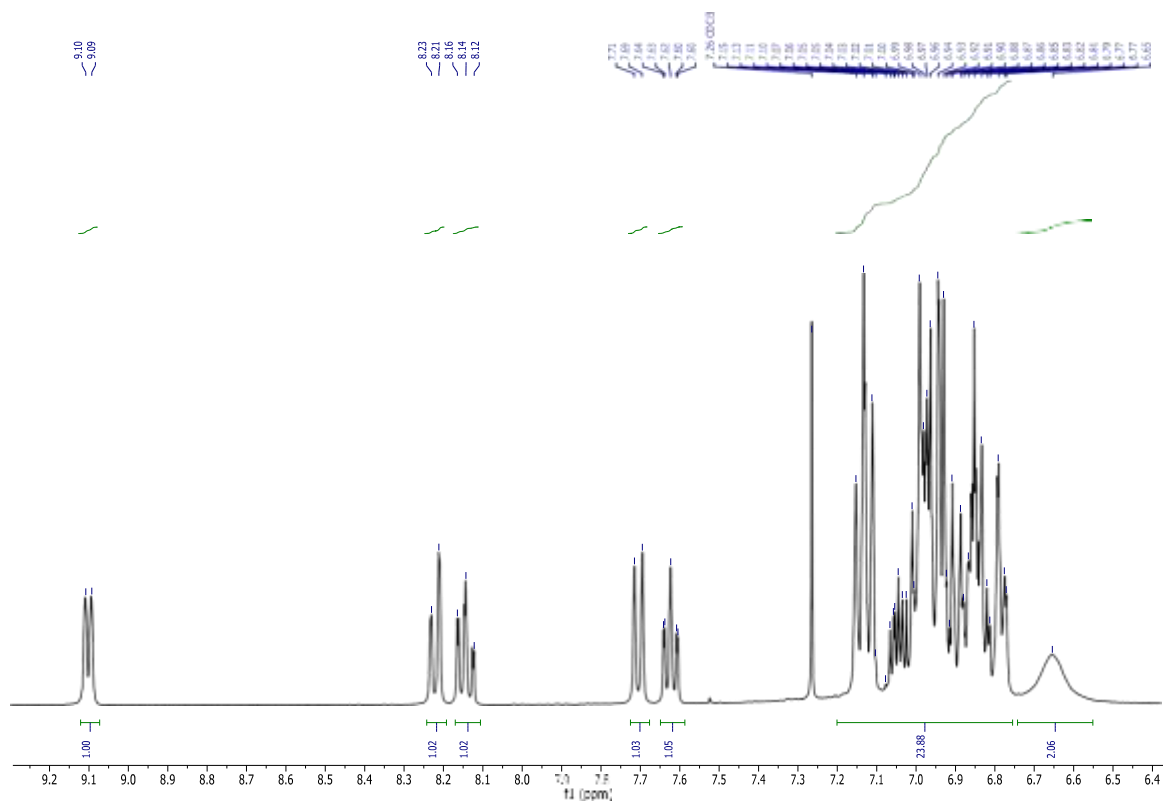


Figure S-21: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of *syn-3* in CDCl_3 .

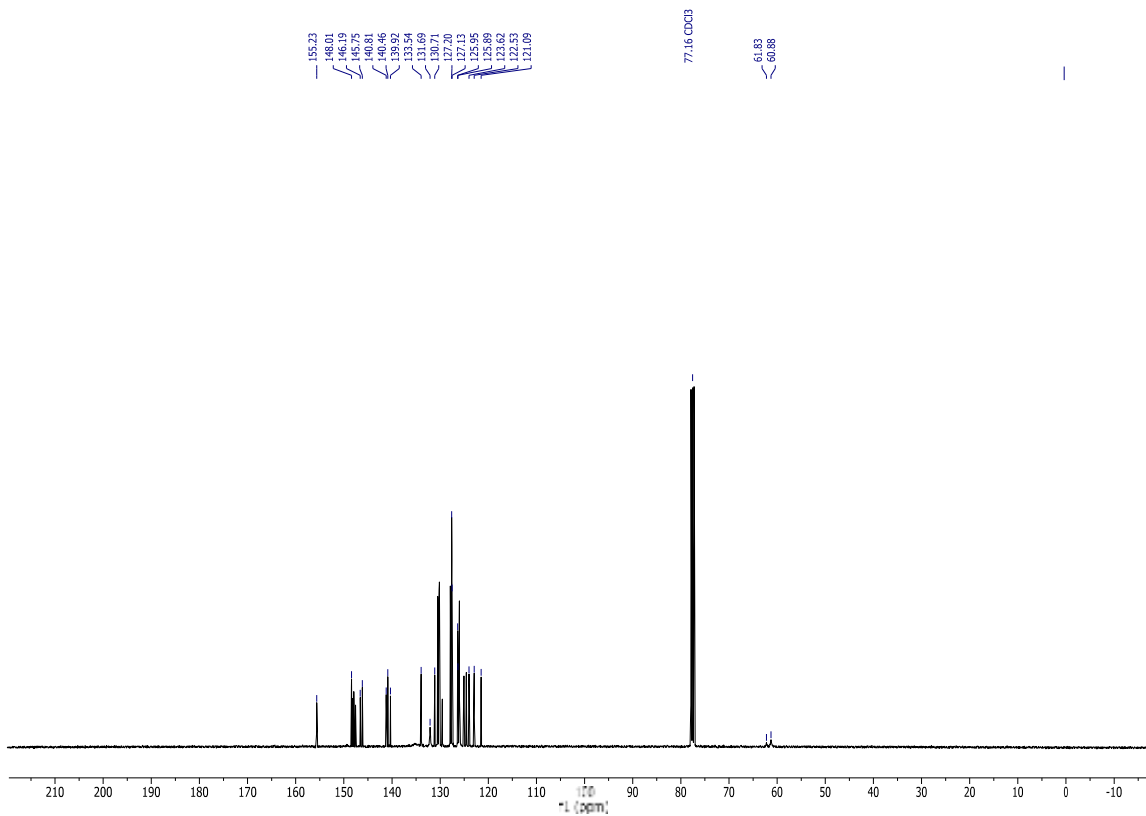


Figure S-22: Expansion of $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of *syn-3* (in CDCl_3 (aryl region)).

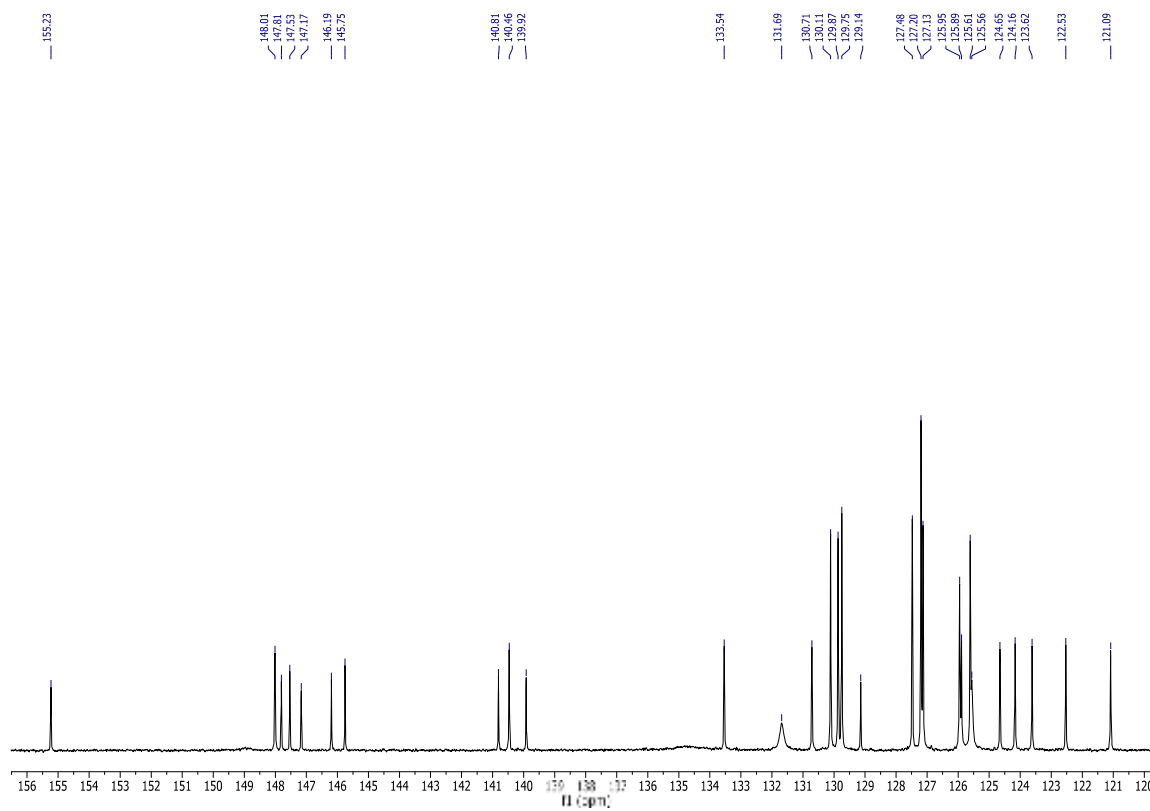


Figure S-23: $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of *syn-3* in CDCl_3 .

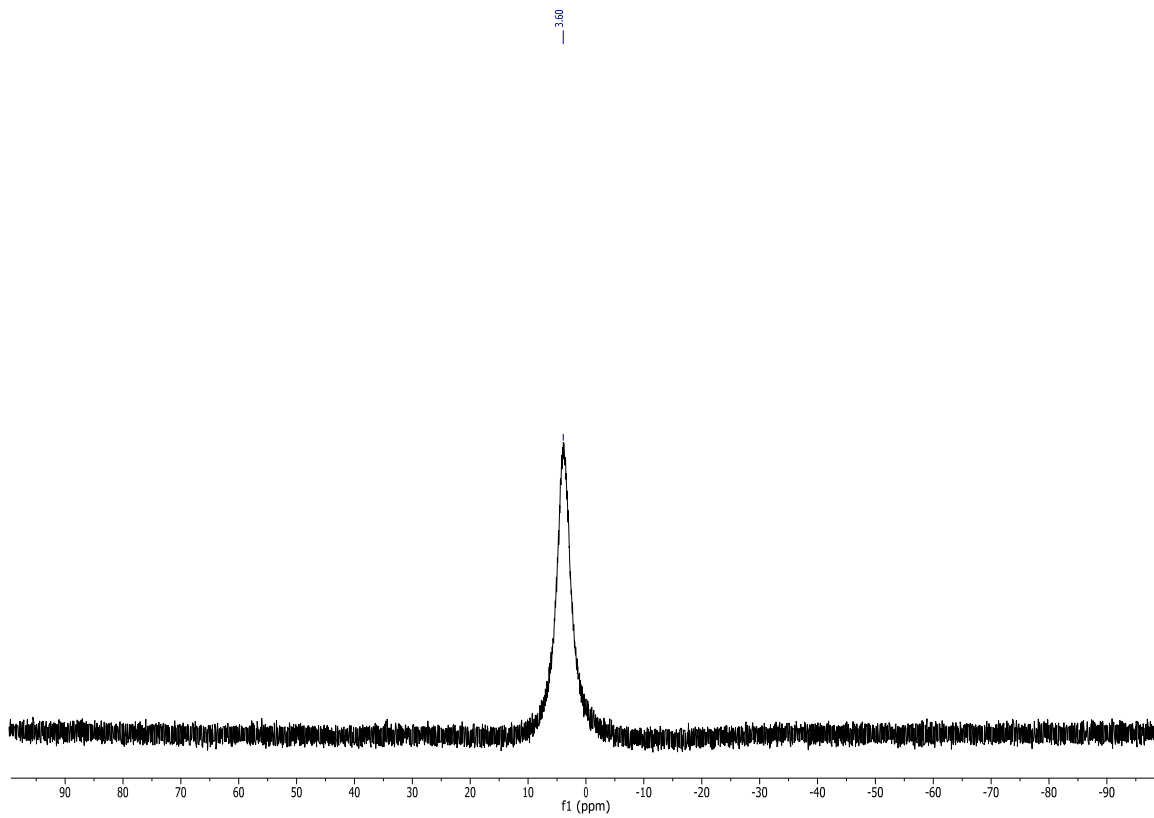


Figure S-24: FT-IR spectrum of *syn-3*.

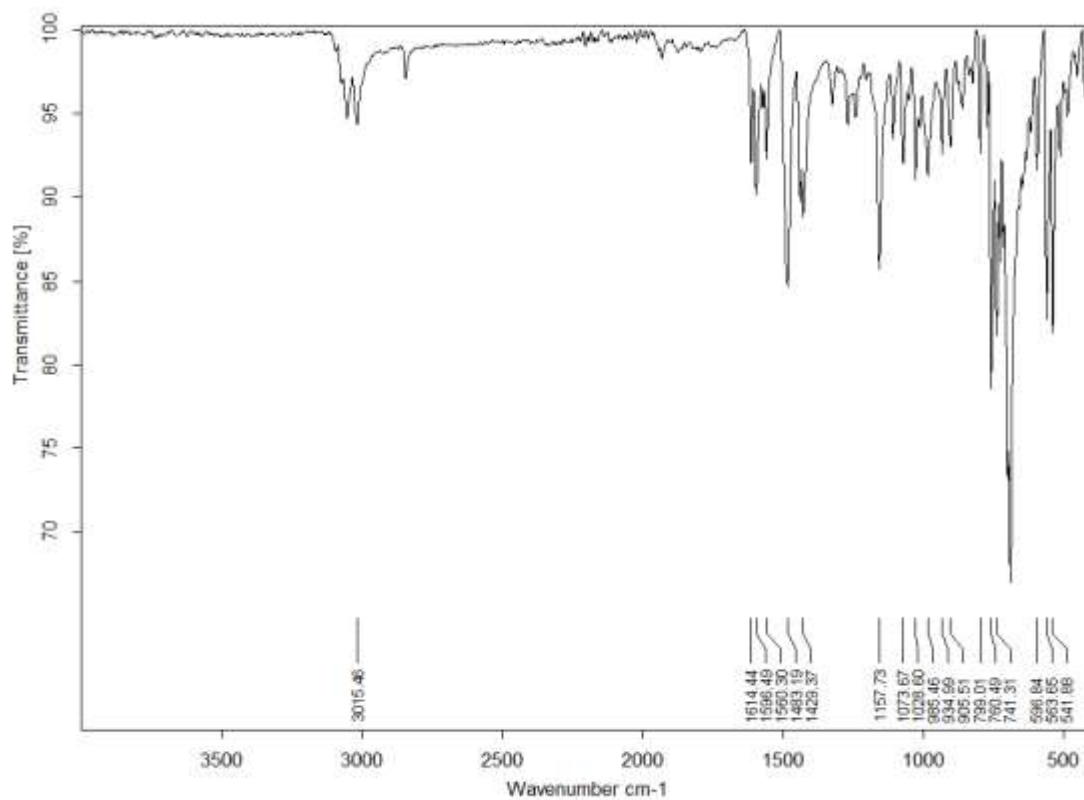


Figure S-25: ^1H NMR spectrum of *anti*-**3** in CDCl_3 (* *n*-pentane).

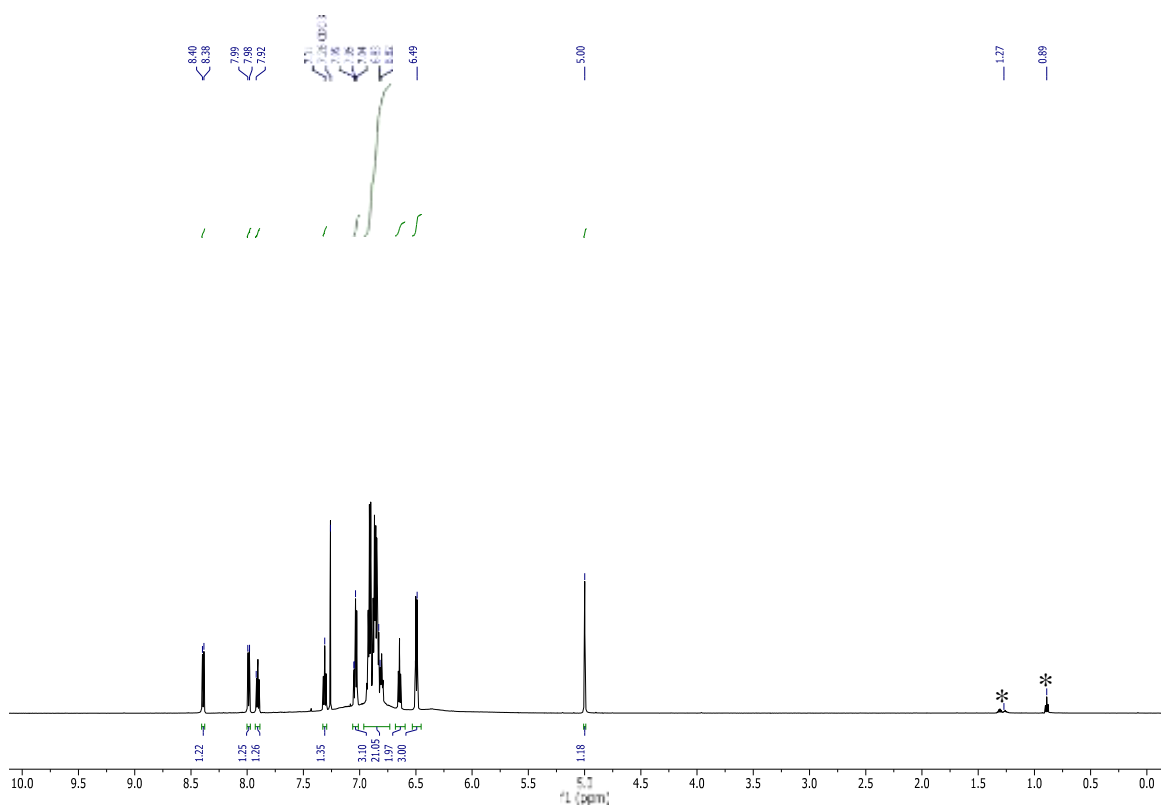


Figure S-26: Expansion of ^1H NMR spectrum of *anti-3* in CDCl_3 (aryl region).

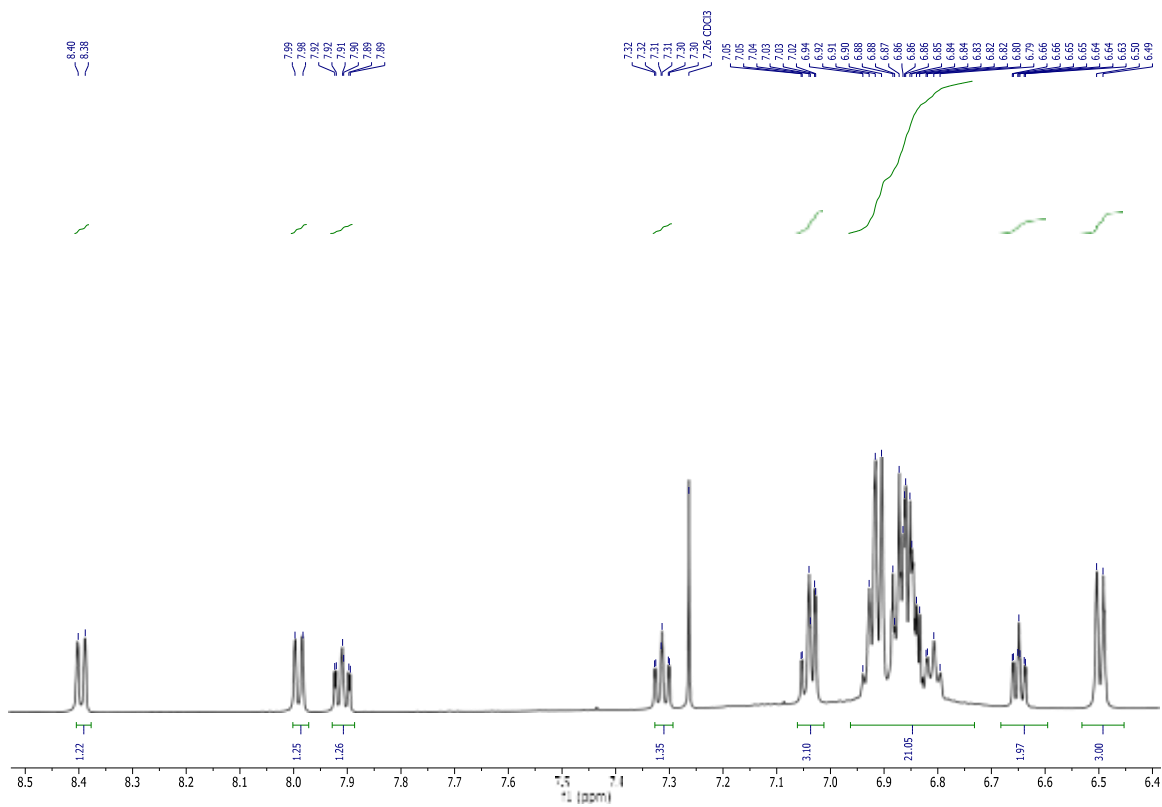


Figure S-27: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of *anti*-**3** in CDCl_3 (**n*-pentane).

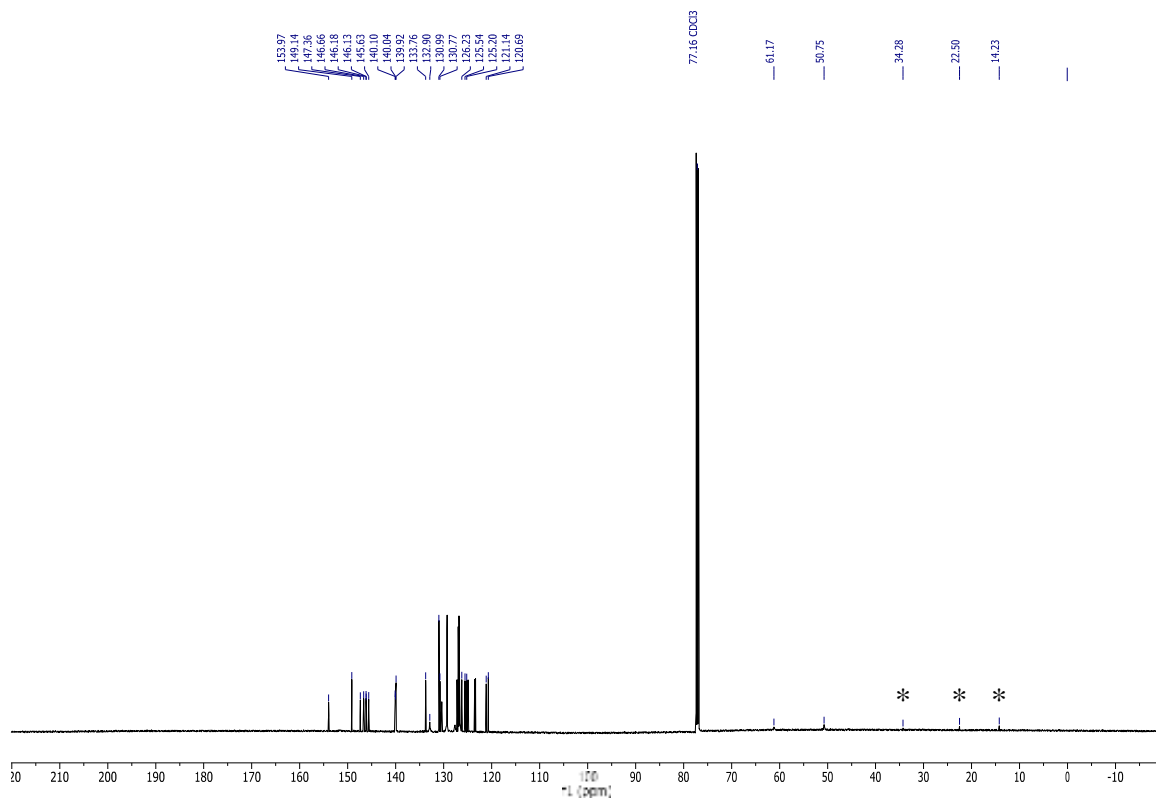


Figure S-28: Expansion of $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of *anti*-**3** in CDCl_3 (aryl region).

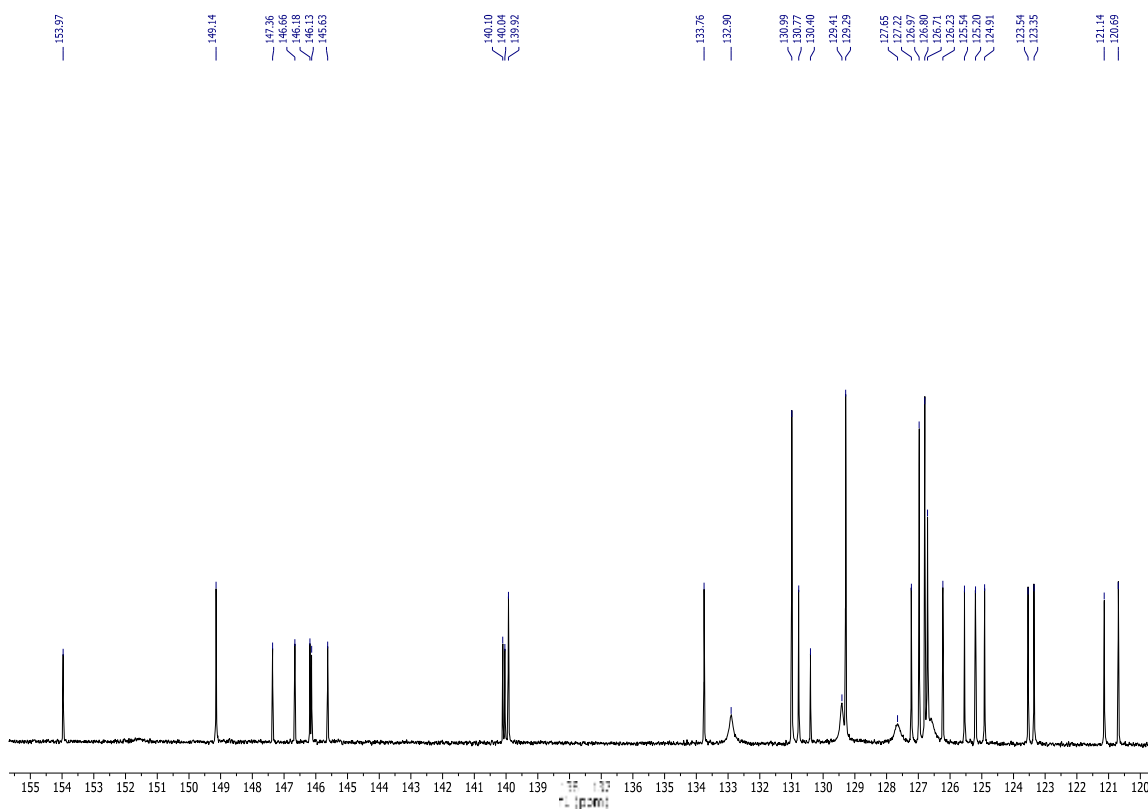


Figure S-29: $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of *anti*-**3** in CDCl_3 .

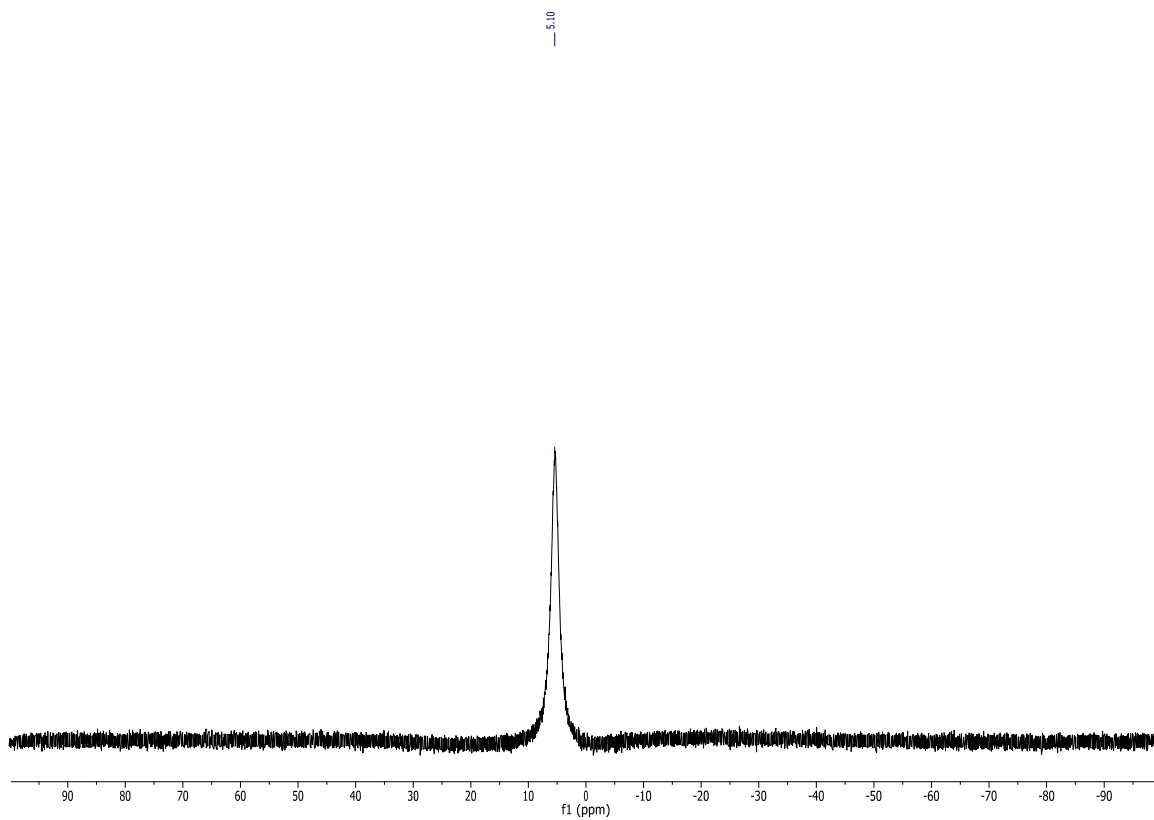


Figure S-30: FT-IR spectrum of *anti-3*.

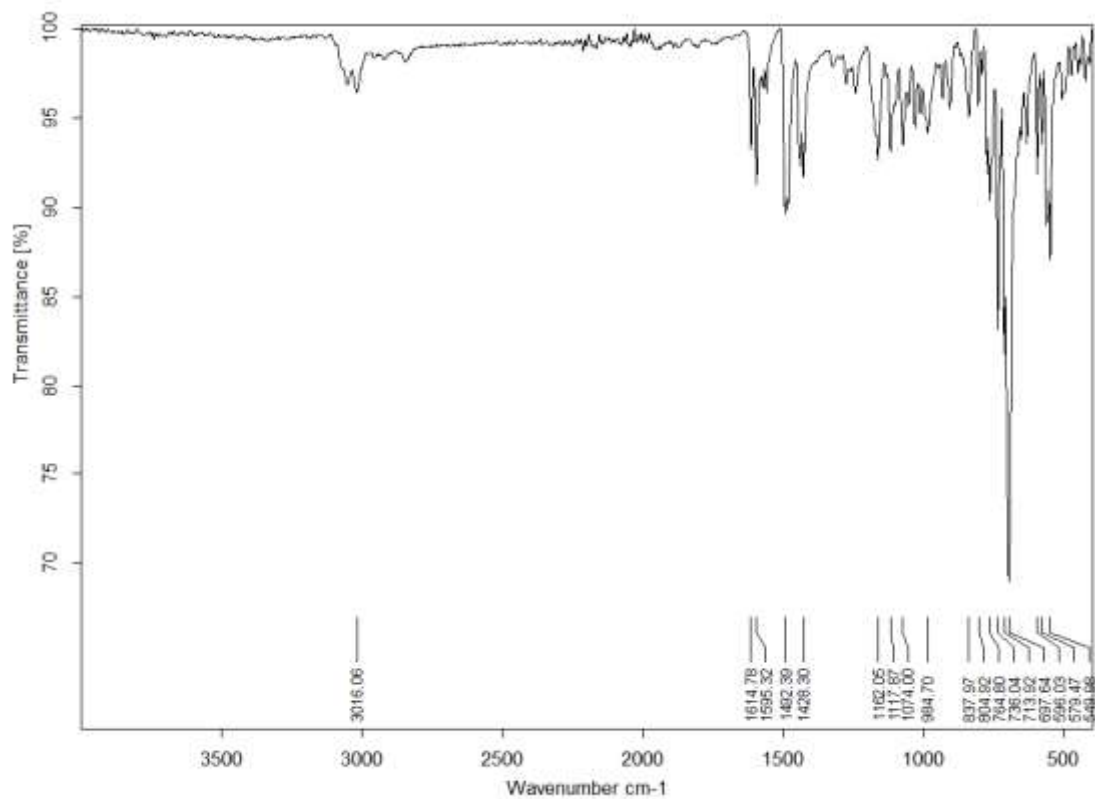


Figure S-31: Stacked plot of ^1H NMR spectrum of *syn-4* at 25 °C and -45 °C in CDCl_3 (
• CDCl_3).

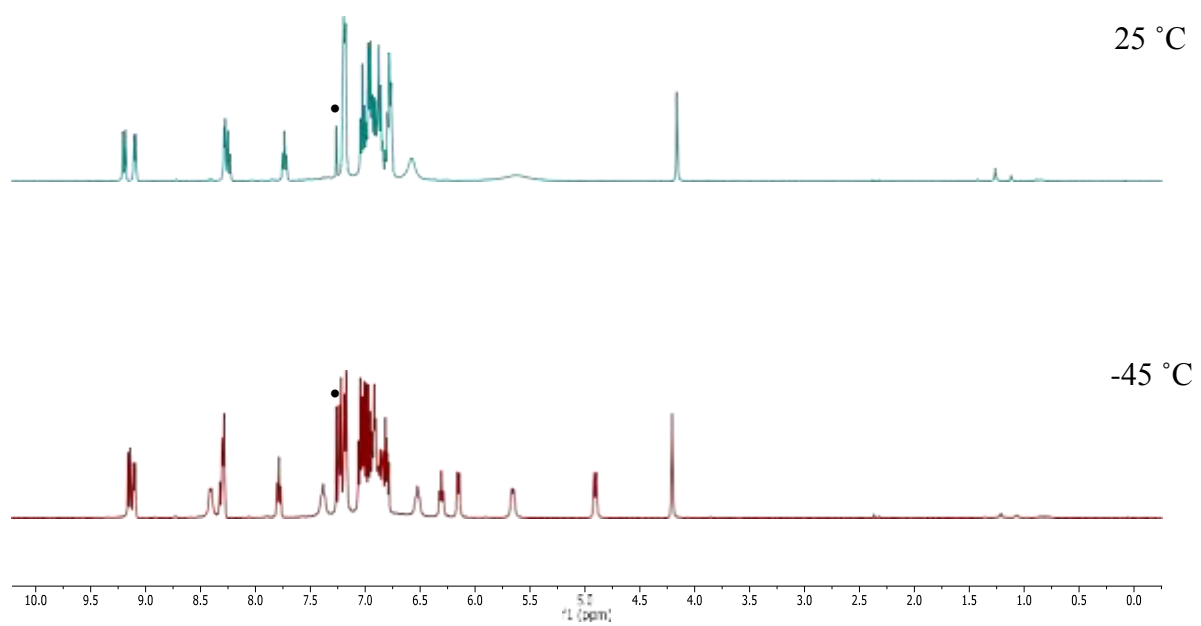


Figure S-32: ^1H NMR spectrum of *syn-4* in CDCl_3 at $-45\text{ }^\circ\text{C}$.

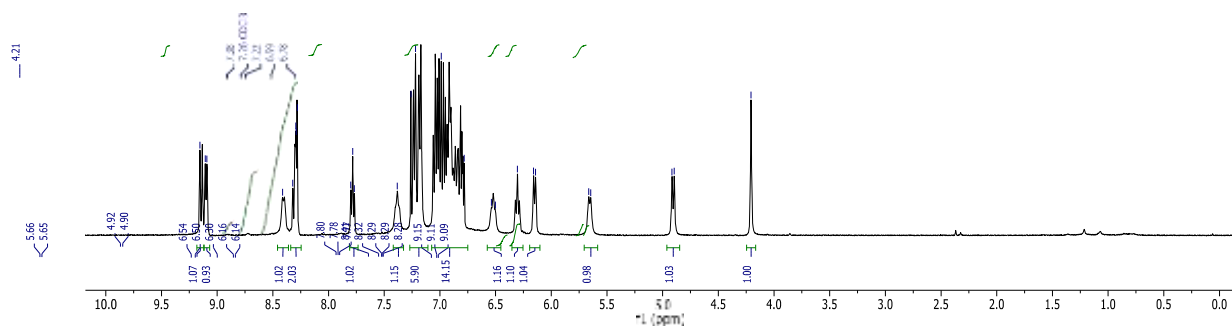


Figure S-33: Expansion of ^1H NMR spectrum of *syn-4* in CDCl_3 at -45°C (aryl region).

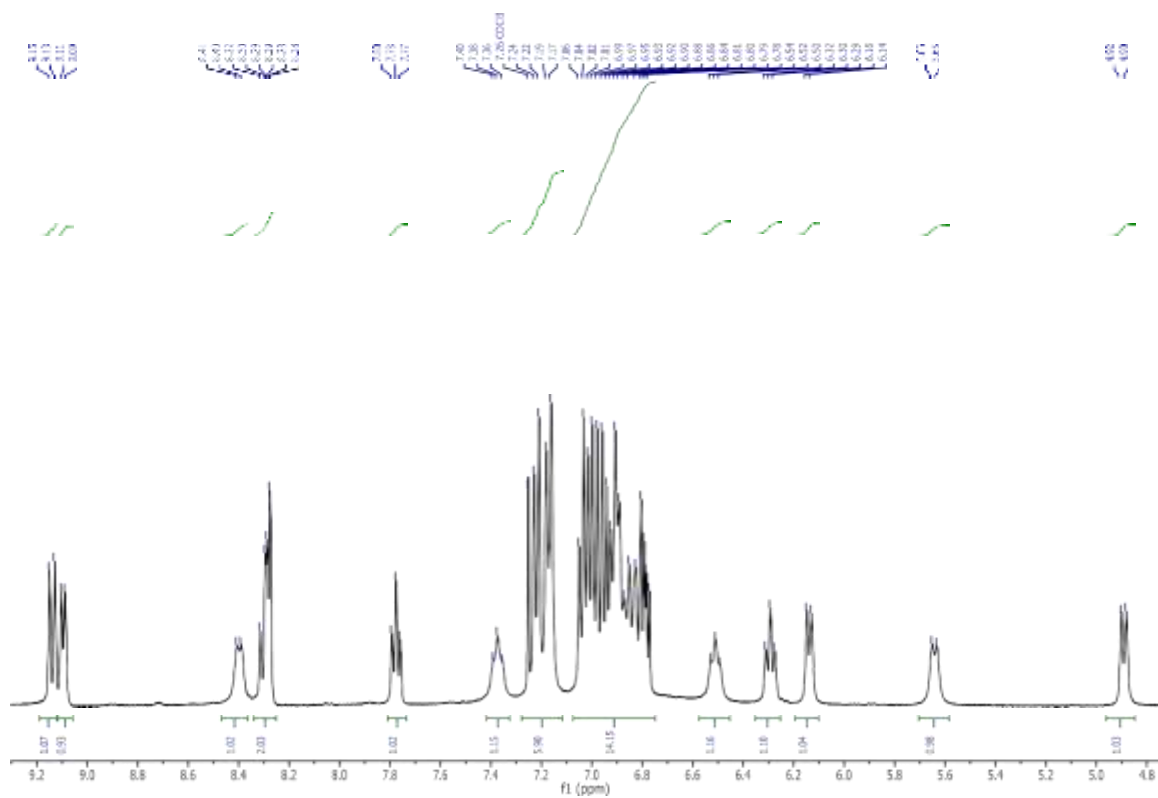


Figure S-34: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of *syn-4* in CDCl_3 .

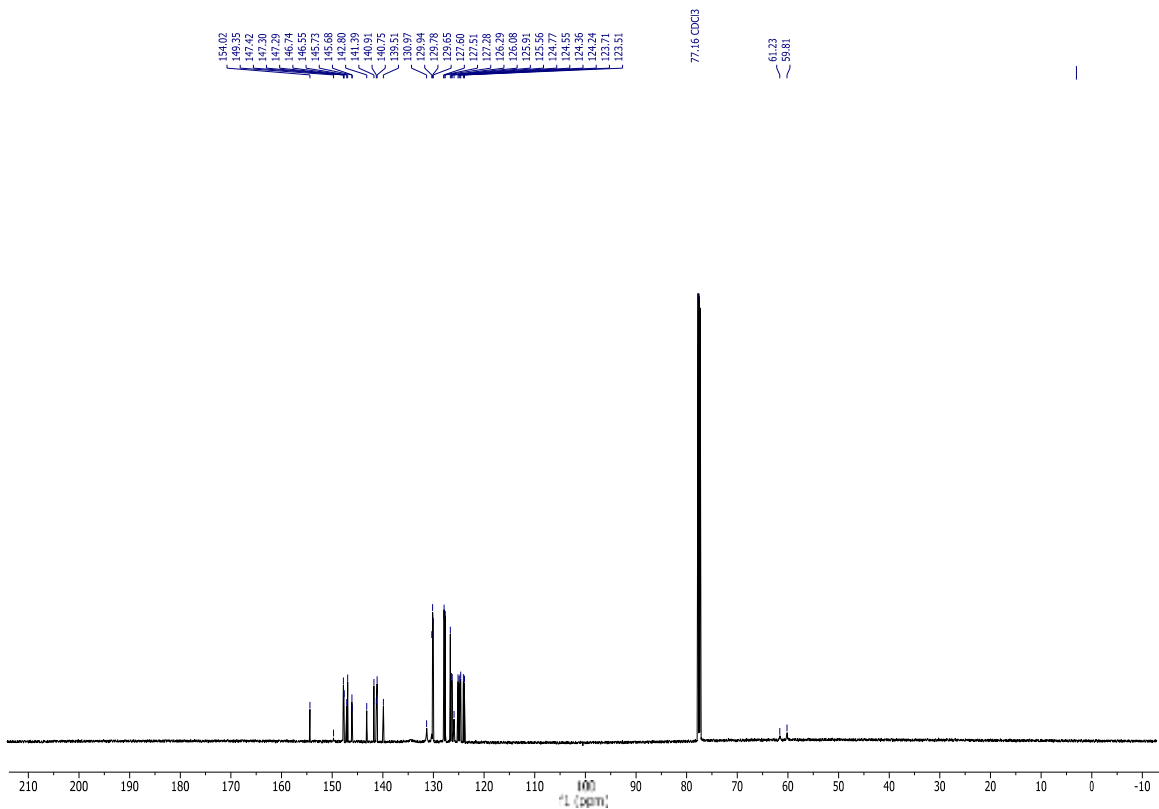


Figure S-35: Expansion of $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of *syn-4* in CDCl_3 (aryl region).

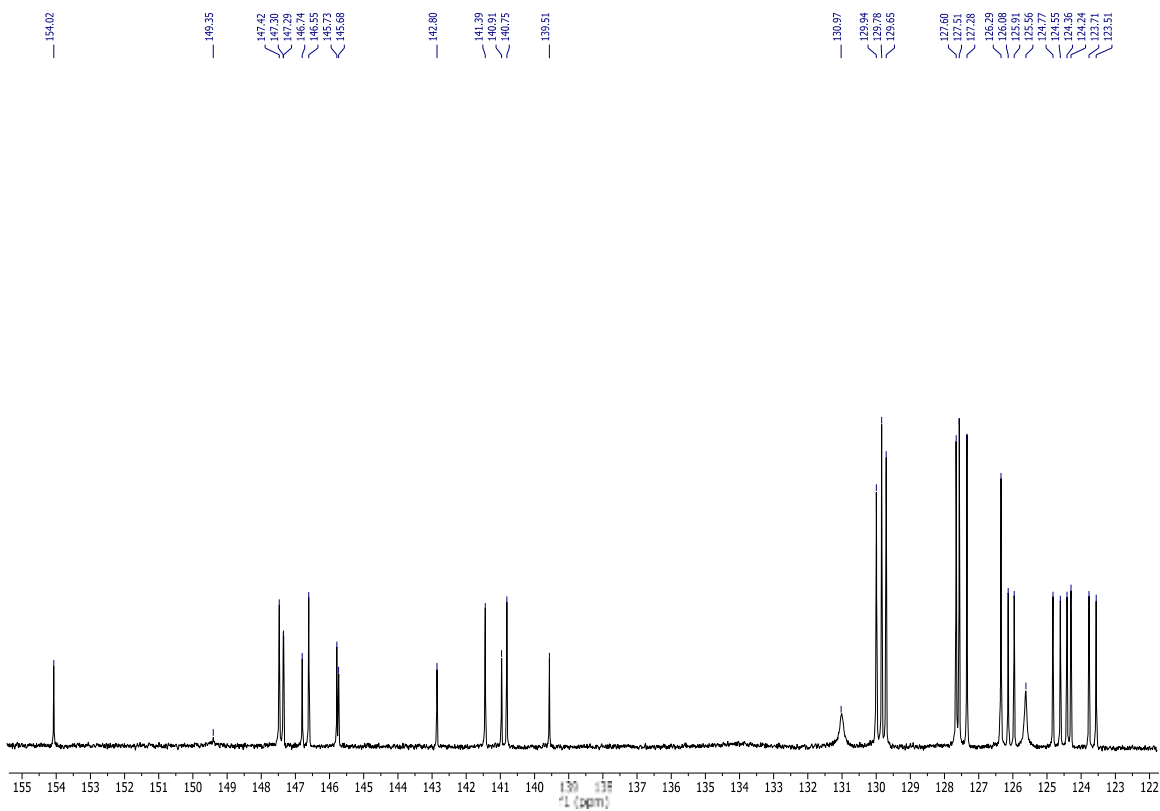


Figure S-36: $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of *syn-4* in CDCl_3 .

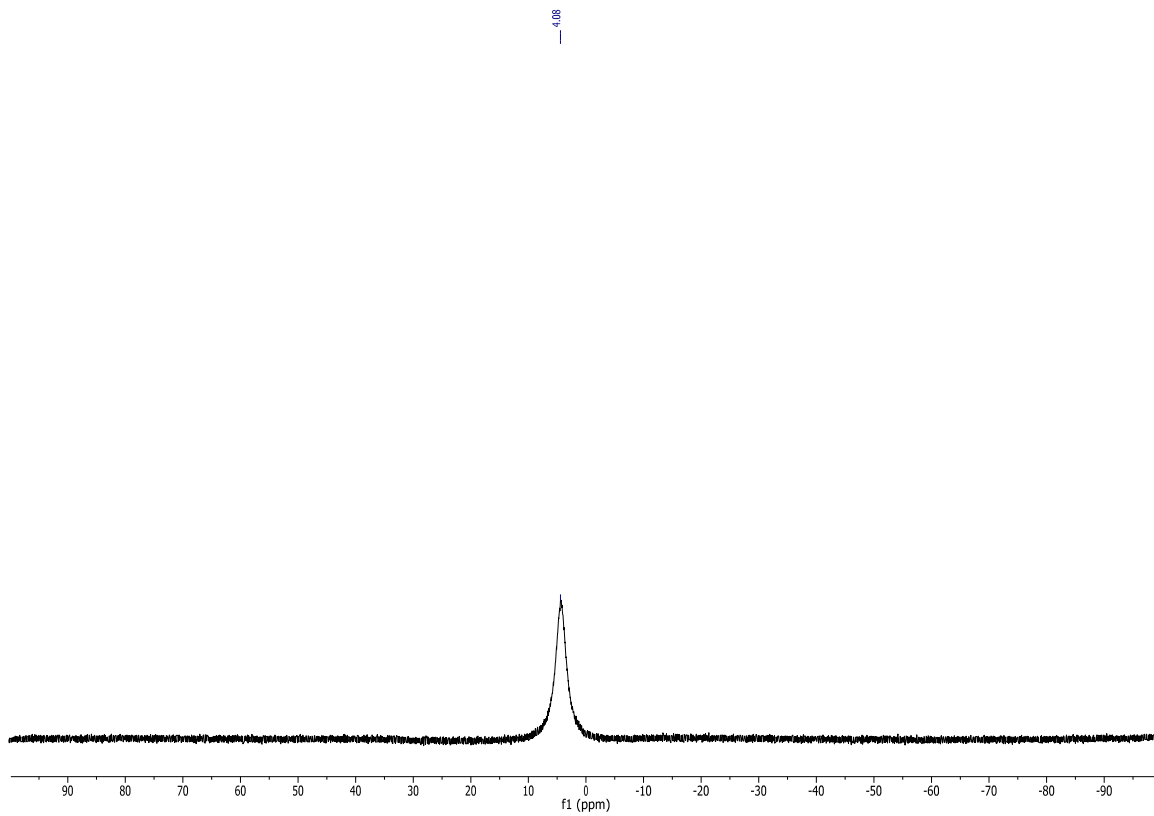


Figure S-37: FT-IR spectrum of *syn-4*.

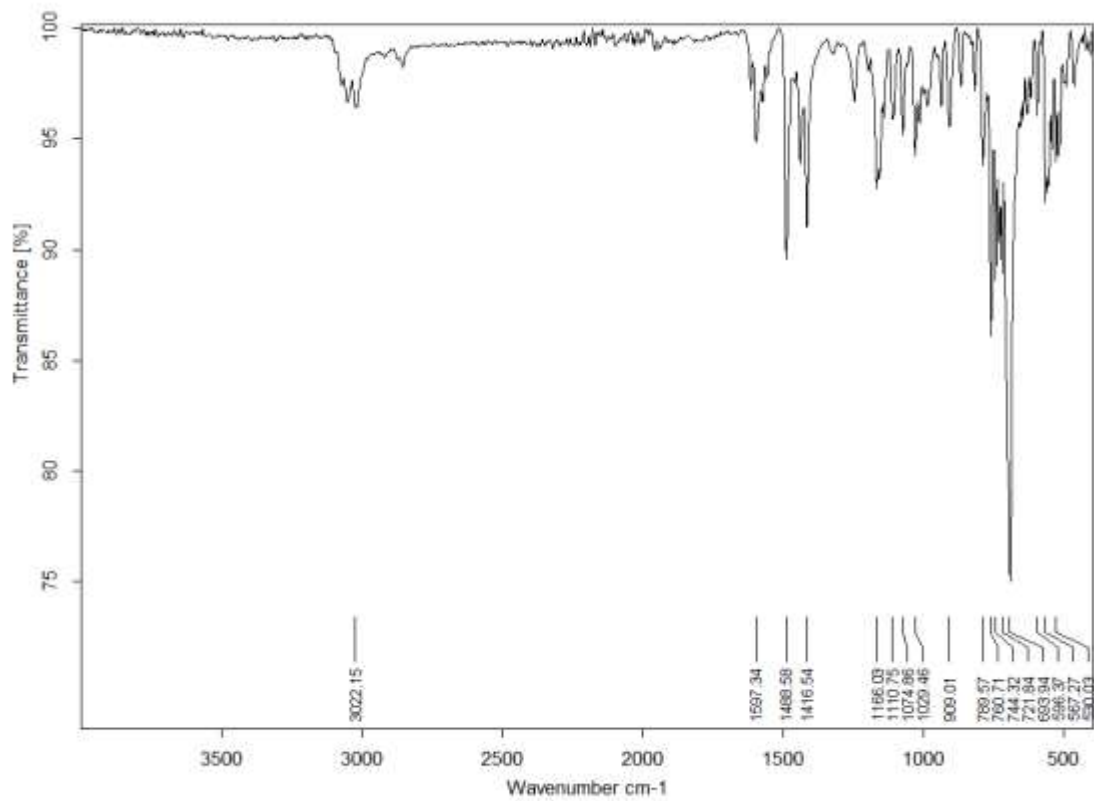


Table S-1: Crystallographic data for **1**, **2**, *syn-3*, *anti-3*, and *syn-4*.

	1	2	<i>syn-3</i>	<i>anti-3</i>	<i>syn-4</i>
CCDC	1558294	1558293	1558290	1558291	1558292
Empirical formula	C ₄₅ H ₃₄ BN	C ₄₄ H ₃₃ BN ₂	C ₄₅ H ₃₄ BN	C ₄₅ H ₃₄ BN	C ₄₄ H ₃₃ BN ₂
FW (g/mol)	599.54	600.53	599.54	599.54	600.53
Crystal system	Monoclinic	Monoclinic	Monoclinic	Triclinic	Monoclinic
Space group	<i>P2₁/n</i>	<i>P2₁/n</i>	<i>P2₁/c</i>	<i>P-1</i>	<i>P2₁/c</i>
<i>a</i> (Å)	9.8501(6)	10.0560(4)	11.3768(8)	9.6901(8))	11.3303(5)
<i>b</i> (Å)	17.9052(12)	18.4589(7)	17.7046(14)	9.8569(7)	17.6684(7)
<i>c</i> (Å)	18.5912(12)	17.5795(6)	17.1202(14)	17.5945(15)	17.0803(6)
α (deg)	90	90	90	87.561(4)	90
β (deg)	93.551(2)	96.5170(10)	108.047(2)	78.334(4)	107.431(2)
γ (deg)	90	90	90	83.695(3)	90
<i>V</i> (Å ³)	3272.6(4)	3242.1(2)	3278.7(4)	1635.5(2)	3262.3(2)
<i>Z</i>	4	4	4	2	4
<i>D_c</i> (mg m ⁻³)	1.217	1.232	1.215	1.217	1.223
radiation, λ (Å)	0.71073	0.71073	0.71073	0.71073	0.71073
temp (K)	150(2)	150(2)	150(2)	274(2)	150(2)
<i>R1</i> [<i>I</i> > 2 σ <i>I</i>] ^a	0.0576	0.0450	0.0522	0.0485	0.0456
<i>wR2</i> (<i>F</i> ²) ^a	0.1409	0.1404	0.1523	0.1347	0.1341
GOF (<i>S</i>) ^a	1.057	1.096	1.173	1.068	1.108

^a $R1(F[I > 2(I)]) = \sum \|F_o\| - |F_c| / \sum |F_o|$; $wR2(F^2 [\text{all data}]) = [w(F_o^2 - F_c^2)]^{1/2}$; $S(\text{all data}) = [w(F_o^2 - F_c^2)/(n - p)]^{1/2}$ (*n* = no. of data; *p* = no. of parameters varied; $w = 1/[^2(F_o^2) + (aP)^2 + bP]$ where $P = (F_o^2 + 2F_c^2)/3$ and *a* and *b* are constants suggested by the refinement program.¹

References:

1. Sheldrick, G. M. *Acta Crystallogr.* **2008**, *A64*, 112.