# Synthesis and Characterization of Super Bulky β-Diketiminato Group 1 Metal Complexes

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**Supplementary Information (9 pages)**

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## S1. Summary of crystallographic data for 2-7

**Table S1.** Summary of crystallographic data for **2-7**.

| | **2** | **3** | **4**·(hexane)$_{0.5}$(OEt$_2$)$_{0.5}$ | **5** | **6** | **7** |
|---|---|---|---|---|---|---|
| empirical formula | $C_{41}H_{60}N_2$ | $C_{29}H_{43}NO$ | $C_{62}H_{99}LiN_2O_{1.5}$ | $C_{45}H_{69}LiN_2O$ | $C_{106}H_{154}K_2N_4$ | $C_{41}H_{59}KN_2$ |
| formula weight | 580.91 | 421.64 | 903.37 | 660.96 | 1562.52 | 619.00 |
| crystal system | monoclinic | triclinic | monoclinic | triclinic | monoclinic | orthorhombic |
| space group | $P2_1/c$ | $P$-1 | $P2_1/n$ | $P$-1 | $P2_1/n$ | $Pnma$ |
| $a$ (Å) | 15.0415(2) | 10.75380(10) | 10.31590(10) | 15.8715(2) | 14.0249(5) | 11.8341(10) |
| $b$ (Å) | 10.7081(2) | 15.3298(2) | 19.3423(2) | 17.0248(3) | 17.4283(4) | 16.6685(19) |
| $c$ (Å) | 22.1277(2) | 15.9068(2) | 28.3898(2) | 18.3141(4) | 19.1994(7) | 18.3950(15) |
| $\alpha$ (deg.) | 90 | 85.3680(10) | 90 | 117.557(2) | 90 | 90 |
| $\beta$ (deg) | 93.373(1) | 74.0270(10) | 99.0910(10) | 104.449(2) | 103.306(4) | 90 |
| $\gamma$ (deg.) | 90 | 84.4420(10) | 90 | 98.068(2) | 90 | 90 |
| vol (Å$^3$) | 3557.84(9) | 2505.18(5) | 5593.55(9) | 4055.50(15) | 4566.9(3) | 3628.5(6) |
| $Z$ | 4 | 4 | 4 | 4 | 2 | 4 |
| $\rho$ (calcd) (g.cm$^{-3}$) | 1.084 | 1.118 | 1.073 | 1.083 | 1.136 | 1.133 |
| $\mu$ (mm$^{-1}$) | 0.458 | 0.497 | 0.461 | 0.468 | 1.275 | 1.485 |
| $F$(000) | 1280 | 928 | 2000 | 1456 | 1712 | 1352 |
| reflections collected | 26373 | 48933 | 40419 | 76675 | 38794 | 16307 |
| unique reflections | 6598 | 9104 | 10265 | 15037 | 8295 | 3489 |
| $R_{int}$ | 0.0661 | 0.0313 | 0.0795 | 0.0614 | 0.1989 | 0.0704 |
| R1 indices [$I>2\sigma(I)$] | 0.0559 | 0.0745 | 0.0775 | 0.0618 | 0.0991 | 0.0638 |
| $w$R2 indices (all data) | 0.1563 | 0.2185 | 0.2296 | 0.1668 | 0.2918 | 0.1731 |
| largest peak/hole (e/Å$^3$) | 0.387, -0.307 | 0.739, -0.350 | 0.913, -0.536 | 0.349, -0.278 | 0.629, -1.197 | 0.249, -0.370 |
| CCDC No. | 2105059 | 2105062 | 2105061 | 2105064 | 2105060 | 2105063 |

## S2. NMR spectra of 2-7

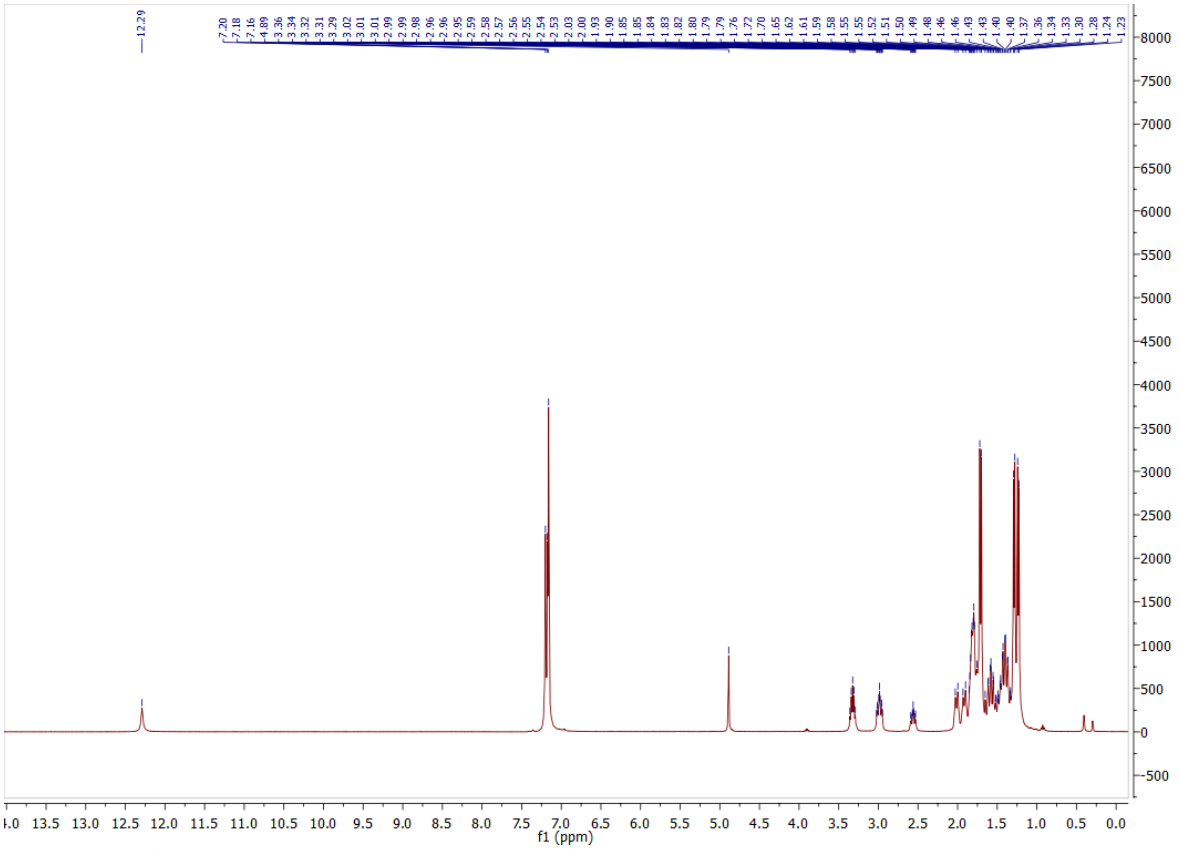


**Figure S1**. $^{1}$H NMR spectrum (400 MHz, 298 K, $C_6D_6$) of **2**.

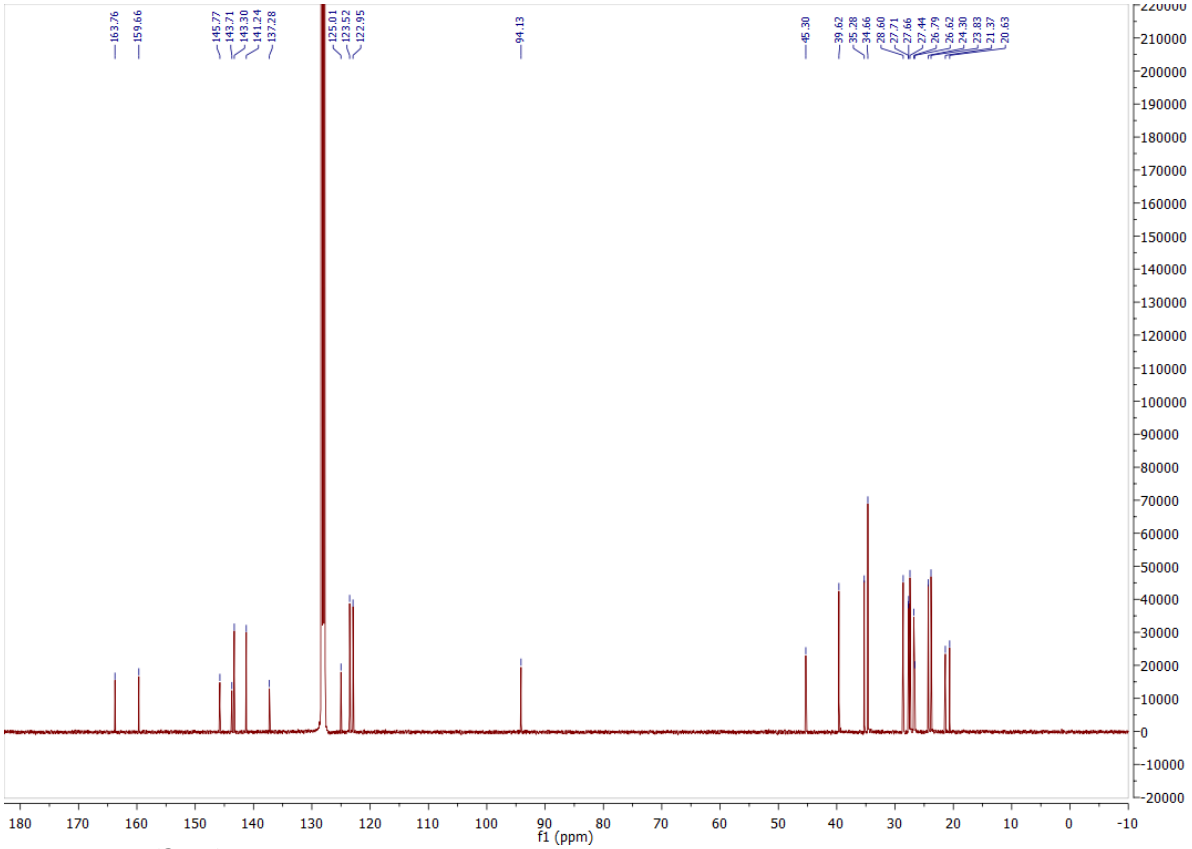


**Figure S2**. $^{13}$C{$^{1}$H} NMR spectrum (101 MHz, 298 K, $C_6D_6$) of **2**.

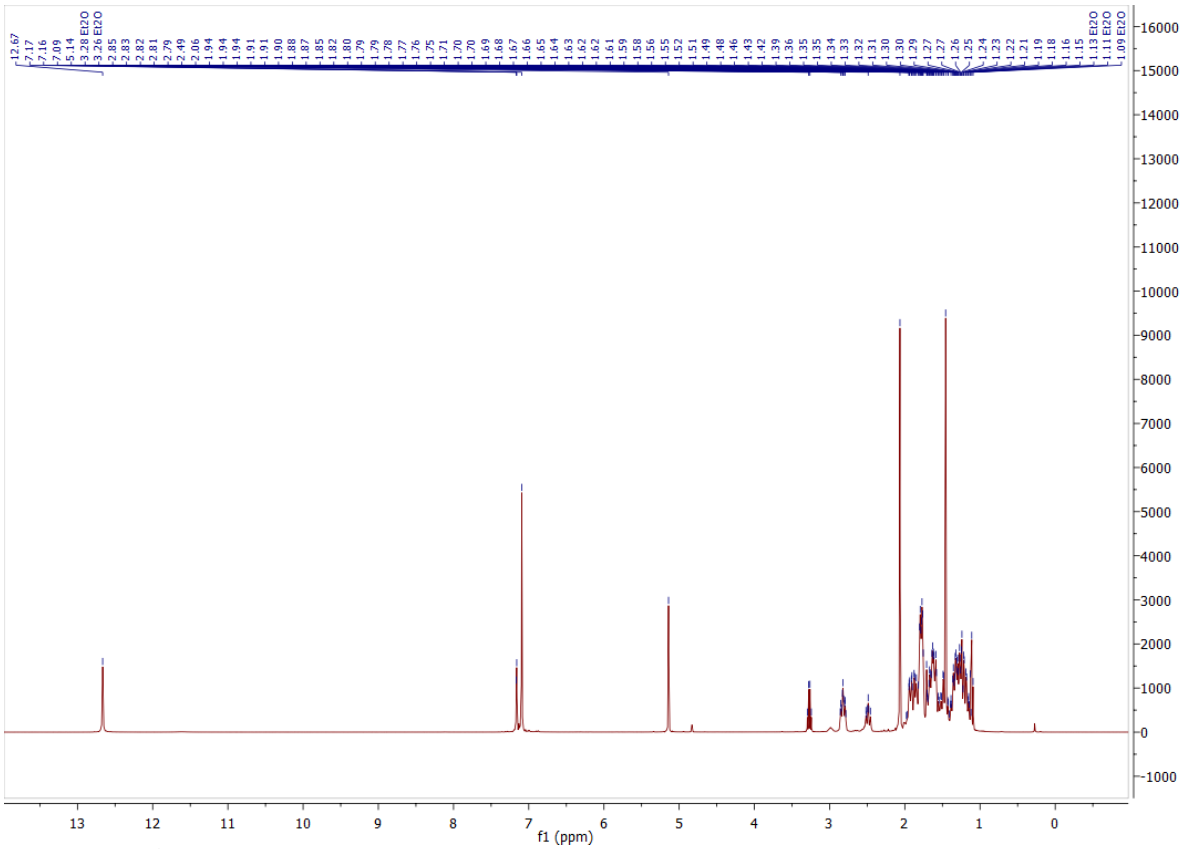


**Figure S3**. $^{1}$H NMR spectrum (400 MHz, 298 K, $C_6D_6$) of **3**.

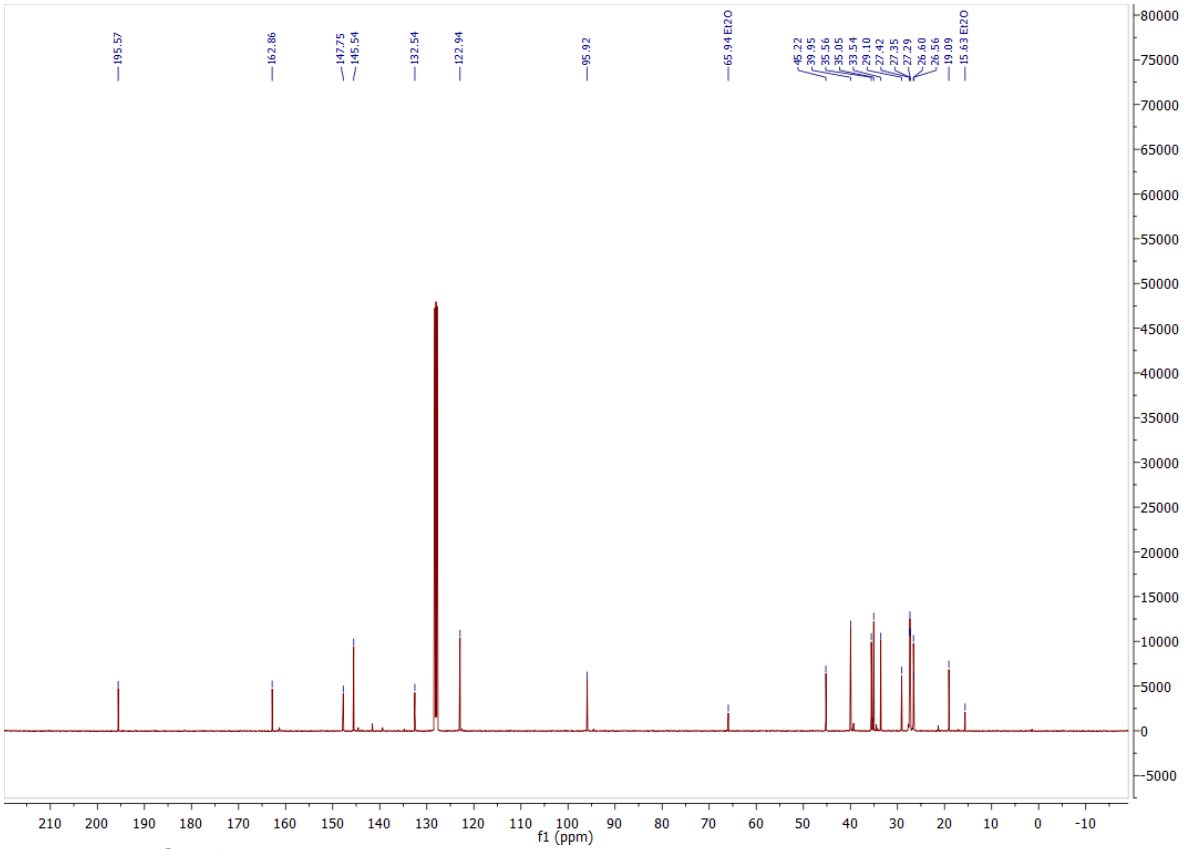


**Figure S4**. $^{13}$C{$^{1}$H} NMR spectrum (101 MHz, 298 K, $C_6D_6$) of **3**.

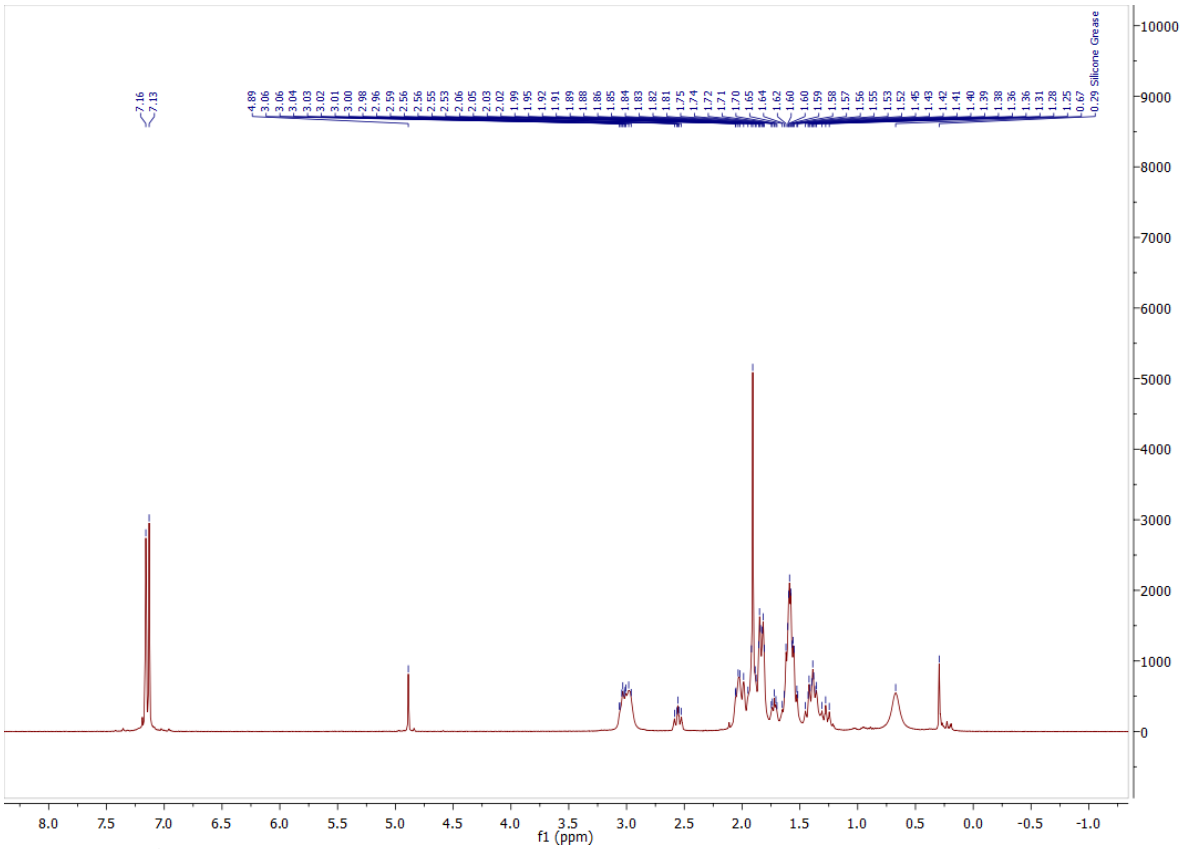


**Figure S5**. $^{1}$H NMR spectrum (400 MHz, 298 K, $C_6D_6$) of **4**.

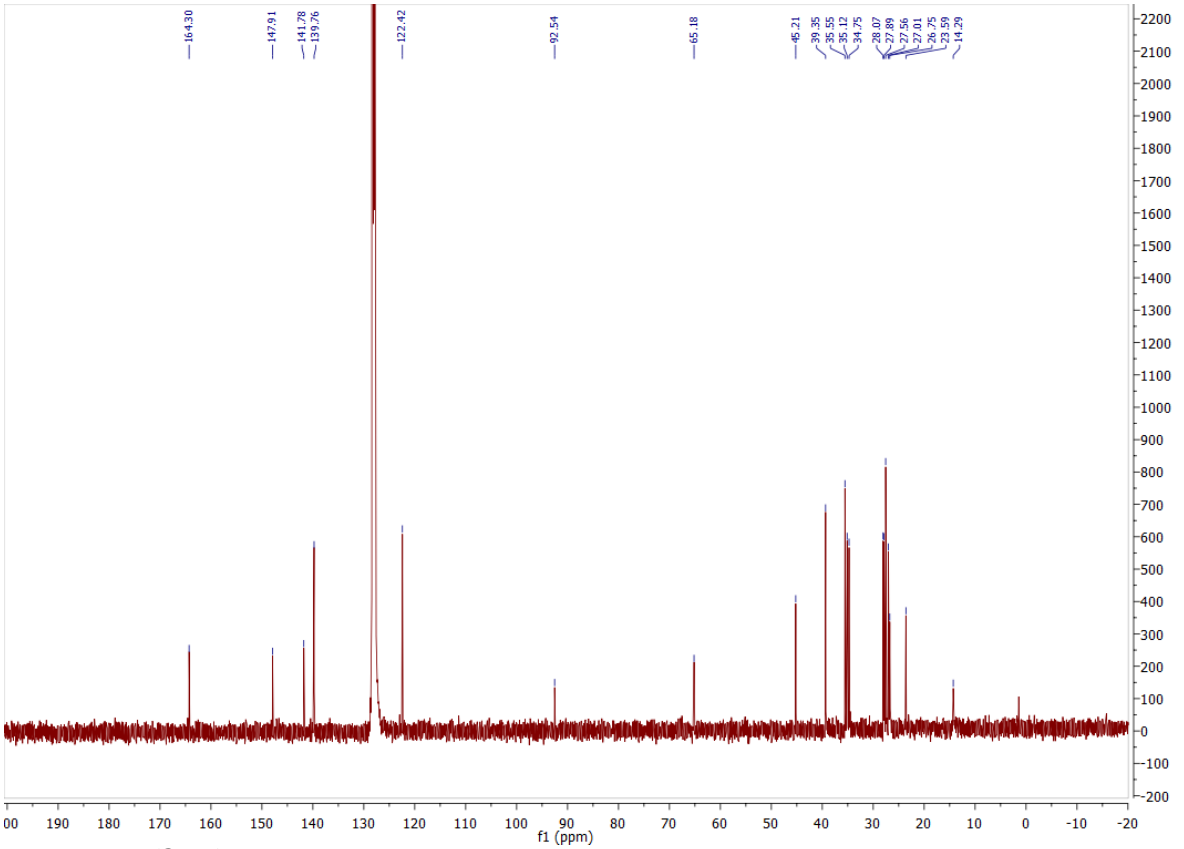


**Figure S6**. $^{13}$C{$^{1}$H} NMR spectrum (101 MHz, 298 K, $C_6D_6$) of **4**.

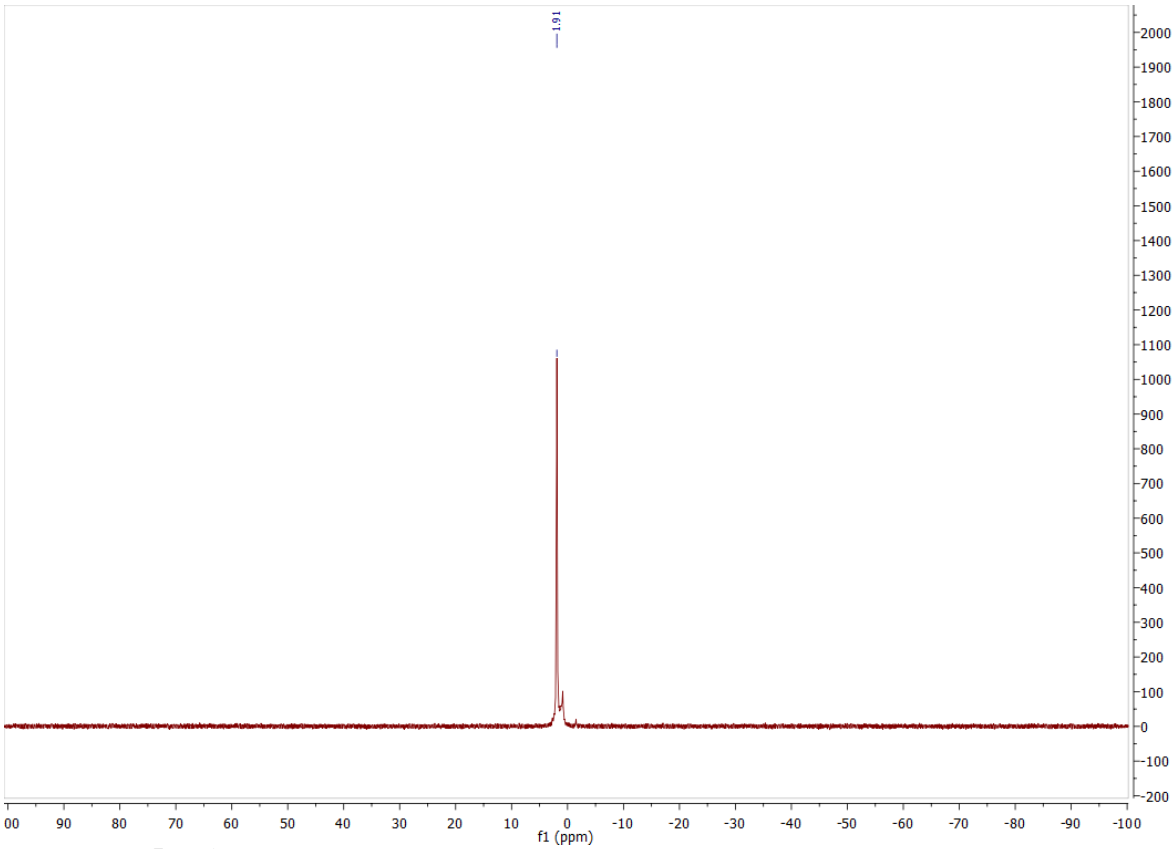


**Figure S7**. $^{7}Li\{^{1}H\}$ NMR spectrum (155 MHz, 298 K, $C_6D_6$) of **4**.

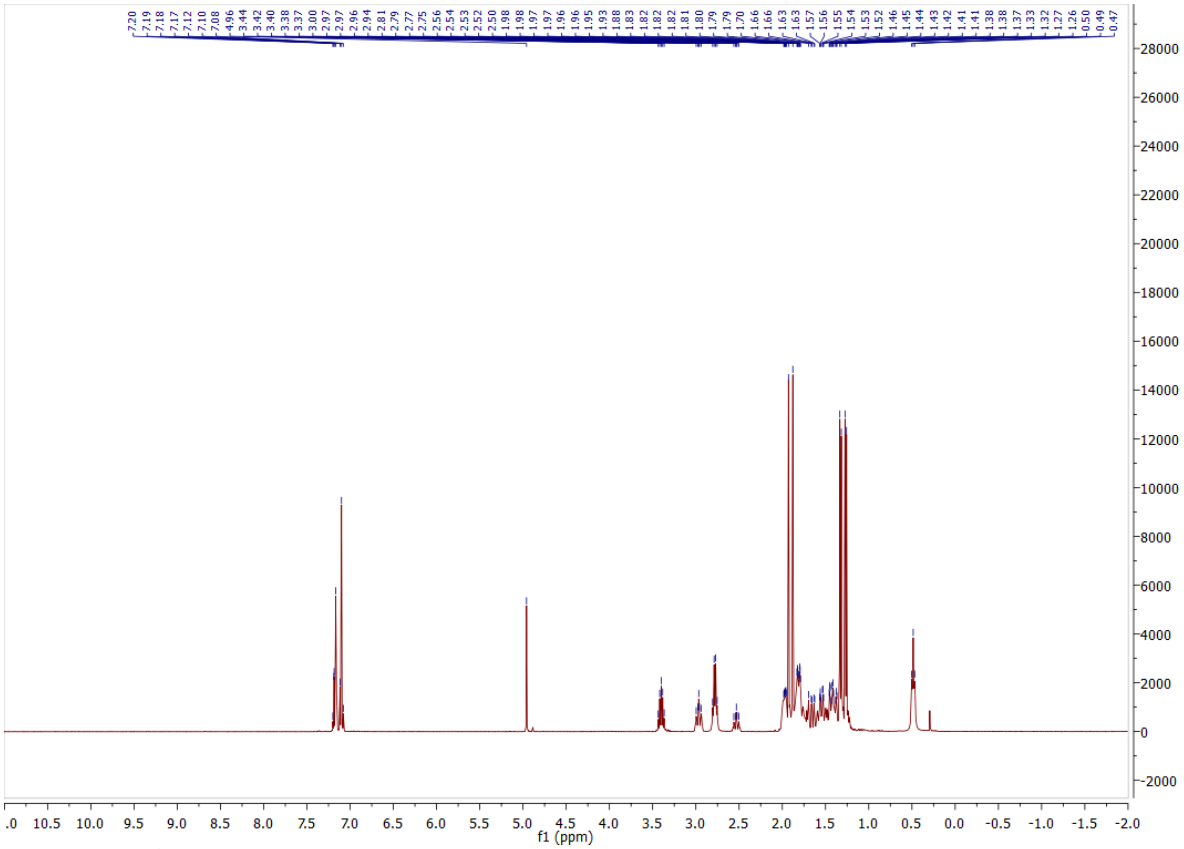


**Figure S8**. $^{1}H$ NMR spectrum (400 MHz, 298 K, $C_6D_6$) of **5**.

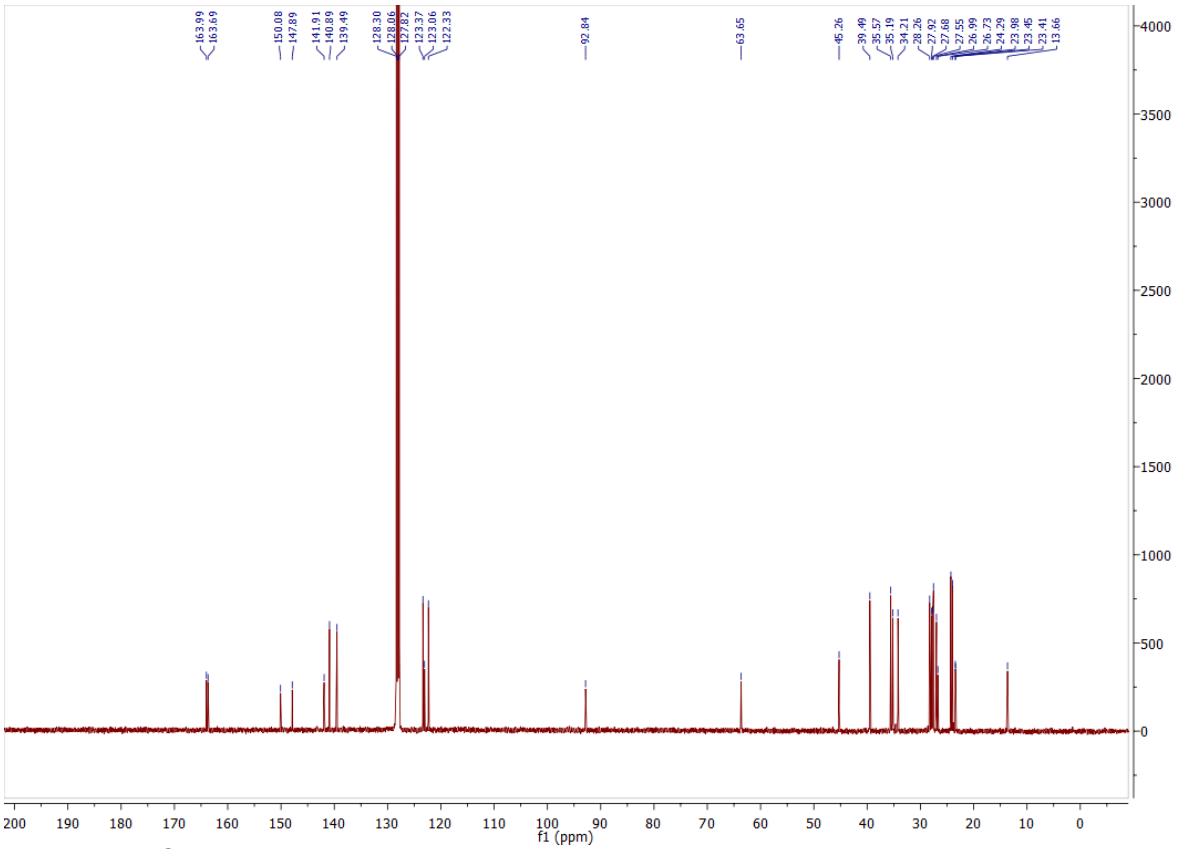


**Figure S9**. $^{13}C\{^{1}H\}$ NMR spectrum (101 MHz, 298 K, $C_6D_6$) of **5**.

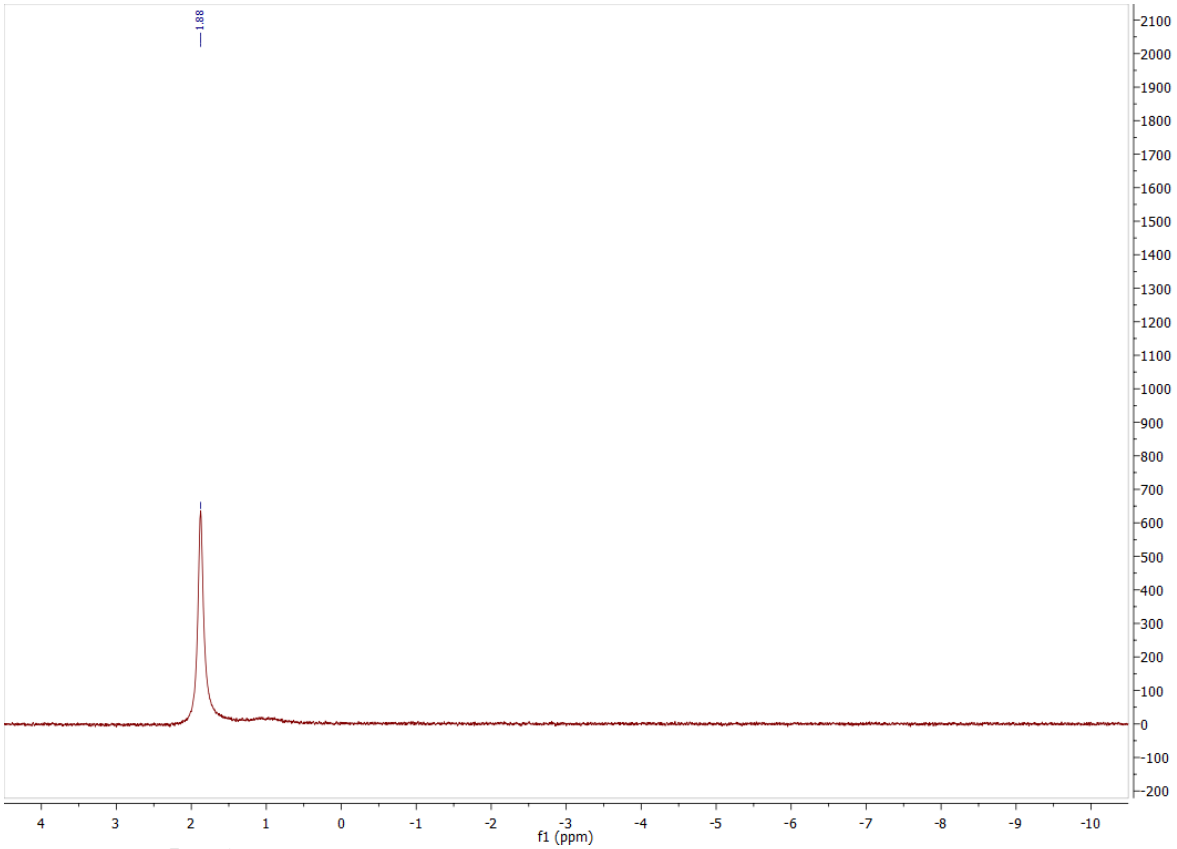


**Figure S10**. $^{7}Li\{^{1}H\}$ NMR spectrum (155 MHz, 298 K, $C_6D_6$) of **5**.

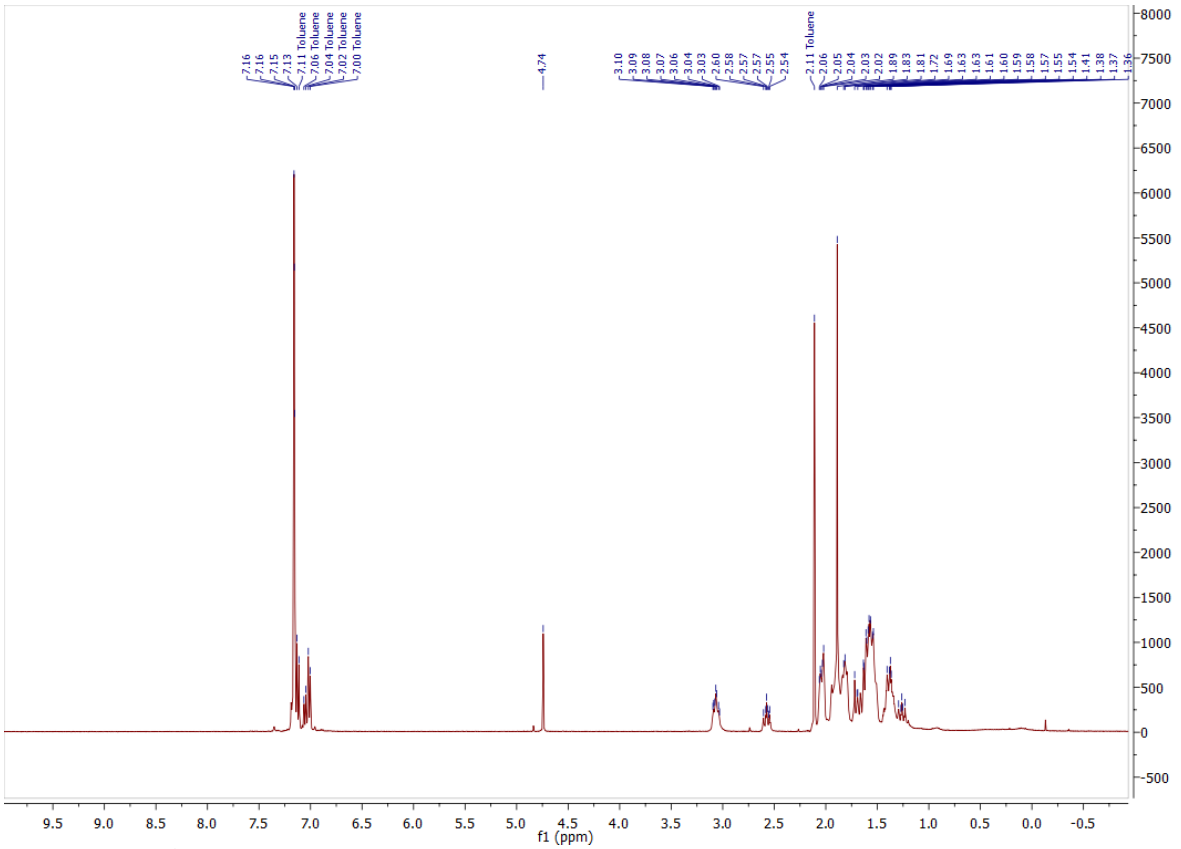


**Figure S11**. $^{1}$H NMR spectrum (400 MHz, 298 K, $C_6D_6$) of **6**.

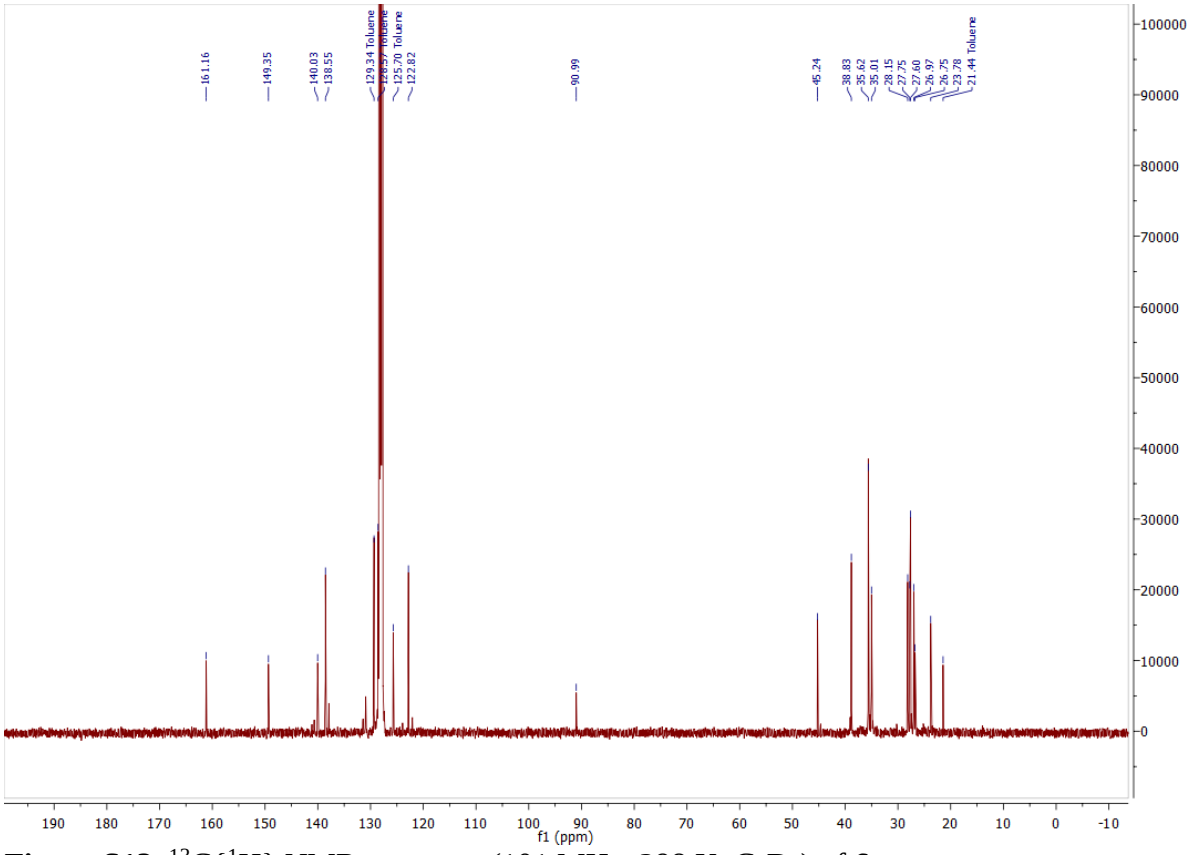


**Figure S12**. $^{13}$C{$^{1}$H} NMR spectrum (101 MHz, 298 K, $C_6D_6$) of **6**.

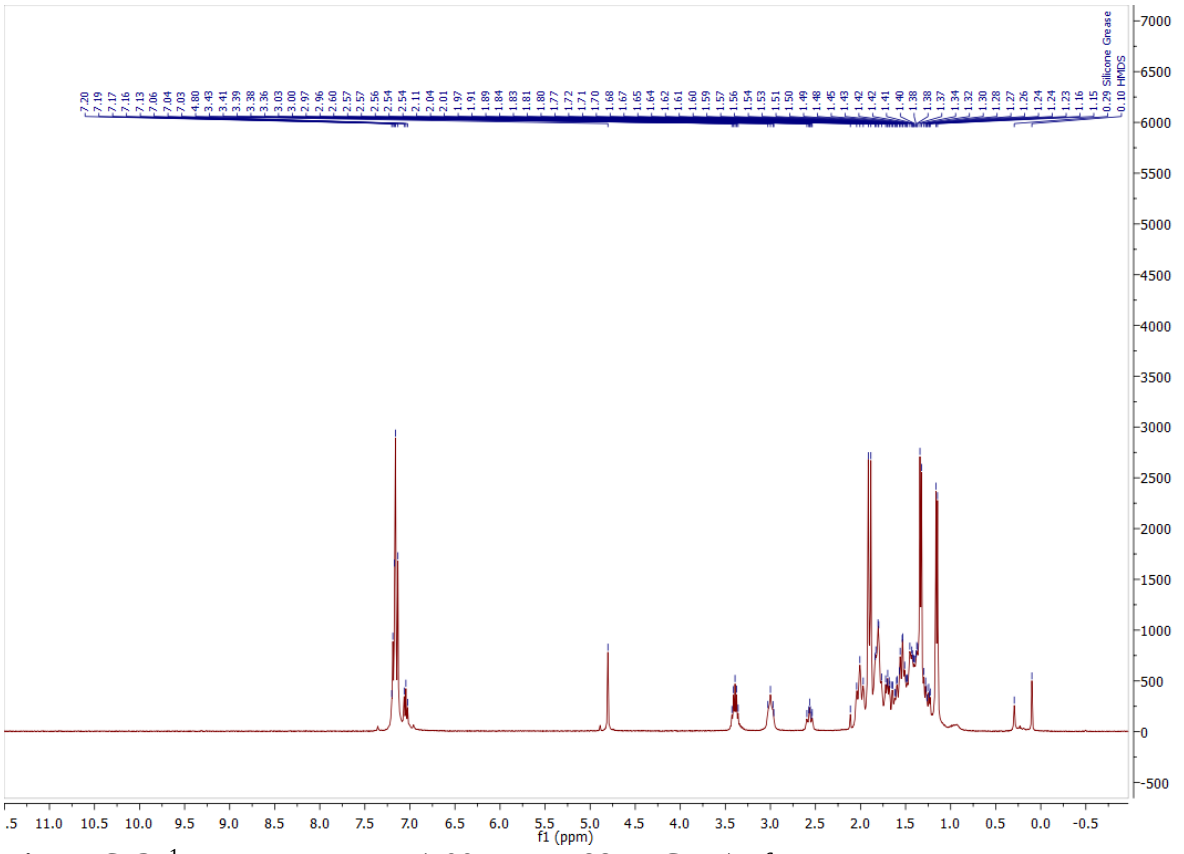


**Figure S13**. $^{1}$H NMR spectrum (400 MHz, 298 K, $C_6D_6$) of **7**.

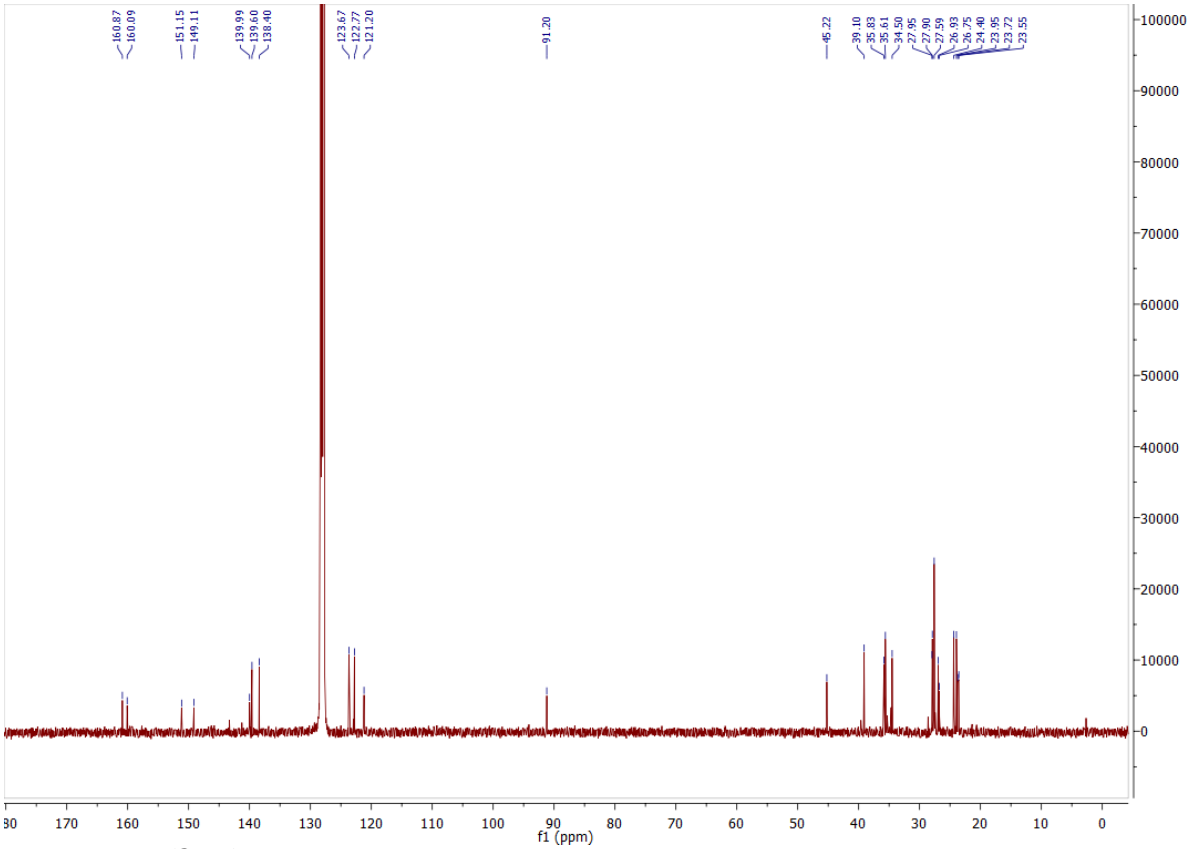


**Figure S14**. $^{13}C\{^{1}H\}$ NMR spectrum (101 MHz, 298 K, $C_6D_6$) of **7**.