Supplimentary Materials

Quinazolin-4(3*H*)-ones: a Tangible Synthesis Protocol *via* an Oxidative Olefin Bond Cleavage Using Metal-catalyst Free Conditions

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Synthesis of Quinazolinones

General Preparation

All chemicals and the reagents used for the execution of the current report were commercially available and procured from Sigma Aldrich, Alfa Aeser, TCI Europe, Acros and used without further purifications unless otherwise reported. All reactions were performed under air in pressure tubes and monitored by thin layer chromatography (TLC) analysis. The synthesized products were characterized by different spectroscopic techniques, which includes ^{1}H- and ^{13}C-NMR, GCMS, HRMS as well as IR spectroscopy. Nuclear magnetic resonance (NMR) spectroscopic analysis were performed on Bruker AV-300 and AV-400 spectrometers using deuterated solvents $CDCl_3$ as well as DMSO-d_6. Gas chromatography (GC) and GC-MS were performed on an Agilent 6890 Agilent 5973 chromatograph. IR spectroscopic characterizations were performed on FTIR ALPHA with Platinum ATR (Bruker).

2. -(4-(tert-butyl)phenyl)quinazolin-4(3H)-one (3b)[66]

yield: (180 mg, 65%); ^{1}HNMR (300 MHz, DMSO-d_6): δ = 1.35 (s, 9H), 7.51-7.62 (m, 3H), 7.73-7.89 (m, 2H), 8.14-8.20 (m, 2H), 8.47 (s, 1H, NH); ^{13}CNMR (DMSO-d_6): δ = 31.8 ($3CH_3$), 35.5 (C), 121.7 (C), 126.2 (2CH), 126.7 (CH), 127.3 (CH), 128.2 (CH), 128.5 (2CH), 130.8 (C), 135.4 (CH), 149.5 (C), 153.1 (C), 155.1 (C), 163.2 (CO); GCMS (EI, 70 eV): *m*/*z* (%) [M^+] 278 (84), 263 (100).

2. -(4-nitrophenyl)quinazolin-4(3H)-one (3c)[66]

yield: (172 mg, 64%); ^{1}HNMR (300 MHz, DMSO-d_6): δ = 6.74 (t, *J* = 7.65 Hz, 1H), 6.82 (d, *J* = 8.55 Hz, 1H), 7.26-7.33 (m, 1H), 7.37 (s, 1H), 7.64-7.65 (m, 1H), 7.79 (dt, *J* = 8.60 Hz, 1H), 8.30 (dt, *J* = 8.60 Hz, 2H), 8.57 (s, 1H, NH). ^{13}CNMR (DMSO-d_6): δ = 115.5, 115.9, 118.4, 124.6, 128.4, 129.0, 134.5, 148.2, 148.4, 150.3, 164.2 (CO); GC-MS (EI, 70 eV): *m*/*z* (%) [M^+] 267 (100), 221 (34), 192 (11), 119 (30), 92 (12), 90 (13).

2-(4-methoxyphenyl)quinazolin-4(3H)-one (3d) [66]

yield: (126 mg, 50%); ^{1}HNMR (300 MHz, DMSO-d_6): δ = 3.39 (s, OCH_3), 7.14 (d, *J* = 8.75 Hz, 2H);7.52 (t, *J* = 8.14 Hz, 1H), 7.73-7.75 (m, 1H), 7.82-7.88 (m, 1H), 8.16 (t, *J* = 8.14 Hz, 1H), 8.23 (d, *J* = 8.75 Hz, 2H), 12.5 (s, 1H, NH); GCMS (EI, 70 eV): *m*/*z* (%) [M^+] 252 (100), 119 (75), 92 (14), 90 (14). HRMS (ESI): Calc. for $C_{15}H_{12}N_2O_2$: 252.08933; found: 252.08948.

2. -(4-fluorophenyl)quinazolin-4(3H)-one (3e) [66]

yield: (130 mg, 54%); ^{1}HNMR (300 MHz, DMSO-d_6): δ = 7.04-7.91 (m, 6H), 8.12-8.40 (m, 2H), 12.6 (s, 1H, NH); ^{13}CNMR (DMSO-d_6): δ = 115.8, 116.3, 116.7, 121.8, 126.8, 127.5, 128.4, 131.2, 135.5, 163.3 (CO); GCMS (EI, 70 eV): *m*/*z* (%) [M$^+$] 240 (100), 122 (11), 120 (14), 119 (90), 95 (16), 92 (14), 90 (11).

2. -(4-chlorophenyl)quinazolin-4(3H)-one (3f) [66]

yield: (143 mg, 56%); ^{1}HNMR (300 MHz, DMSO-d_6): δ = 7.35-7.42 (m, 1H), 7.55-7.64 (m, 1H), 7.67-7.79 (m, 2H), 8.17-8.28 (m, 2H), 12.7 (s, 1H, NH); ^{13}CNMR (DMSO-d_6): δ = 115.2 (C), 116.2 (CH), 123.4 (CH), 128.5 (CH), 129.7 (2CH), 132.0 (2CH), 133.6 (C), 135.5 (C), 136.2 (CH), 140.7 (C), 150.9 (C), 163.0 (CO); GCMS (EI, 70 eV): *m*/*z* (%) [M$^+$] 256 (71), 119 (100), 111 (12), 92 (11), 90 (15), 75 (12); HRMS (ESI): Calc. for $C_{15}H_9N_2OCl$: 256.03979; found: 256.03928.

2. -(4-(trifluoromethyl)phenyl)quinazolin-4(3H)-one (3g) [66]

yield: (160 mg, 55%); ^{1}HNMR (300 MHz, DMSO-d_6): δ = 7.57-7.64 (m, 1H), 7.80-7.99 (m, 4H), 8.20-8.23 (m, 1H), 8.40-8.43 (m, 2H), 12.8 (s, 1H, NH); ^{13}CNMR (DMSO-d_6): δ = 122.2 (C), 123.1 (C), 126.4 (2CH), 126.8 (CH), 128.1 (CH), 128.6 (CH), 129.7 (2CH), 131.9, 132.2 (C), 135.8 (CH), 137.6 (C), 149.4 (C), 152.2 (C), 163.0 (CO); GCMS (EI, 70 eV): *m*/*z* (%) [M$^+$] 290 (100), 145 (21), 119 (98), 92 (15), 90 (14); HRMS (ESI): Calc. for $C_{15}H_9N_2OF_3$: 290.06615; found: 290.06587.

2. -(naphthalen-2-yl)quinazolin-4(3H)-one (3h) [66]

yield: (190 mg, 70%); ^{1}HNMR (300 MHz, DMSO-d_6): δ = 7.59-7.76 (m, 4H), 7.82-7.95 (m, 3H), 8.15-8.28 (m, 4H), 12.7 (s, 1H, NH); ^{13}CNMR (DMSO-d$_6$): δ = 122.1 (C), 126.0 (CH), 126.1 (CH), 126.7 (CH), 127.3 (CH), 127.7 (CH), 127.9 (CH), 128.4 (CH), 128.6 (CH), 129.2 (CH), 131.2 (CH), 132.6 (CH), 134.01 (C), 135.4 (C), 149.6 (C), 162.8 (CO); GCMS (EI, 70 eV): *m*/*z* (%) [M$^+$] 272 (55), 272 (100).

2. -(pyridin-3-yl)quinazolin-4(3H)-one (3i) [66]

yield: (138 mg, 62%); ^{1}HNMR (300 MHz, DMSO-d_6): δ = 7.59-7.73 (m, 2H), 7.83-8.09 (m, 2H), 8.12-8.22 (m, 1H), 8.19-8.24 (m, 1H), 8.49-8.79 (m, 1H), 8.81 (d, *J* = 4.95 Hz, 1H), 10.7 (s, 1H, NH); ^{13}CNMR (DMSO-d_6): δ = 122.9 (C), 123.0 (CH), 127.0 (CH), 127.5 (CH), 128.1 (C), 128.4 (C), 135.6 (CH), 138.9 (CH), 149.6 (CH), 150.1 (CH), 152.5 (C), 161.9 (CO); GCMS (EI, 70 eV): *m*/*z* (%) [M$^+$] 223 (91), 119 (100), 92 (13), 90 (15), 78 (10); HRMS (ESI): Calc. for $C_{13}H_9N_3O$: 223.07401; found: 223.07411.

3. -(m-tolyl)-2H-benzo[e][1,2,4]thiadiazine 1,1-dioxide (5b) [66]

yield: (160 mg, 59%); ^{1}HNMR (300 MHz, DMSO-d_6): δ = 2.45 (s, 3H, CH_3), 7.47-7.56 (m, 3H), 7.60-7.67 (m, 1H), 7.72-7.80 (m, 1H), 7.82-7.89 (m, 3H), 12.2 (s, 1H, NH); ^{13}CNMR (DMSO-d$_6$): δ = 21.8 (CH_3), 119.3, 122.3, 124.2, 126.3, 127.5, 129.5, 129.7, 132.7, 134.0, 134.4, 136.4, 139.3, 155.8; GCMS (EI, 70 eV): *m*/*z* (%) [M$^+$] 262 (60), 208 (13), 155 (100), 91 (55), 64 (15); HRMS (ESI): Calc. for $C_{14}H_{12}N_2O_2S$: 272.06195; found: 272.06189.

NMR spectra for synthesized quinazolinones'

Figure S 1. ^{1}HNMR of (3).

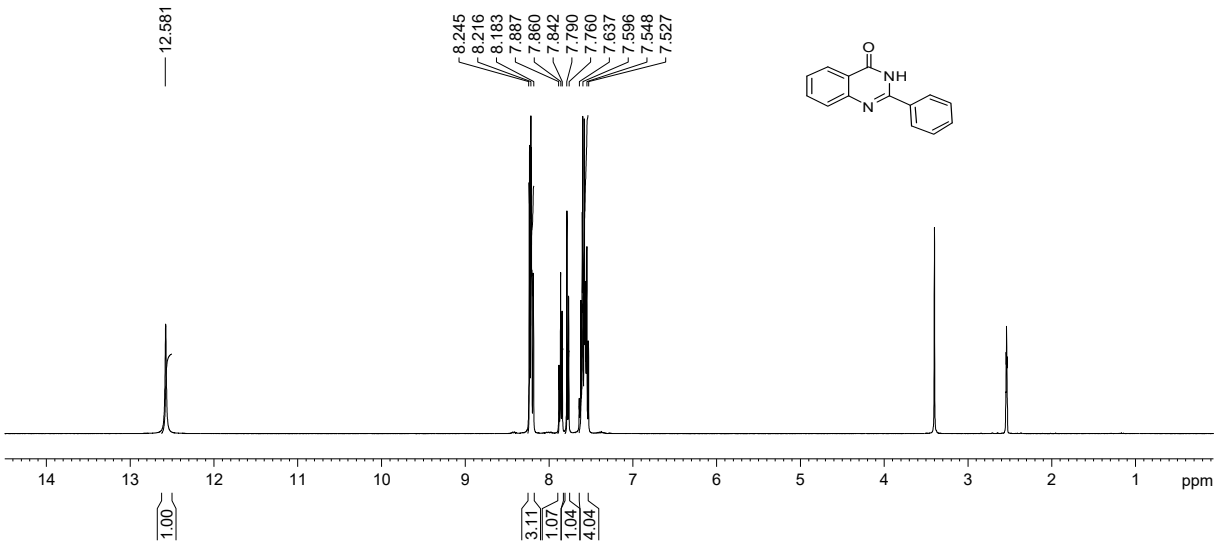

Figure S 2. ^{13}CNMR of (3).

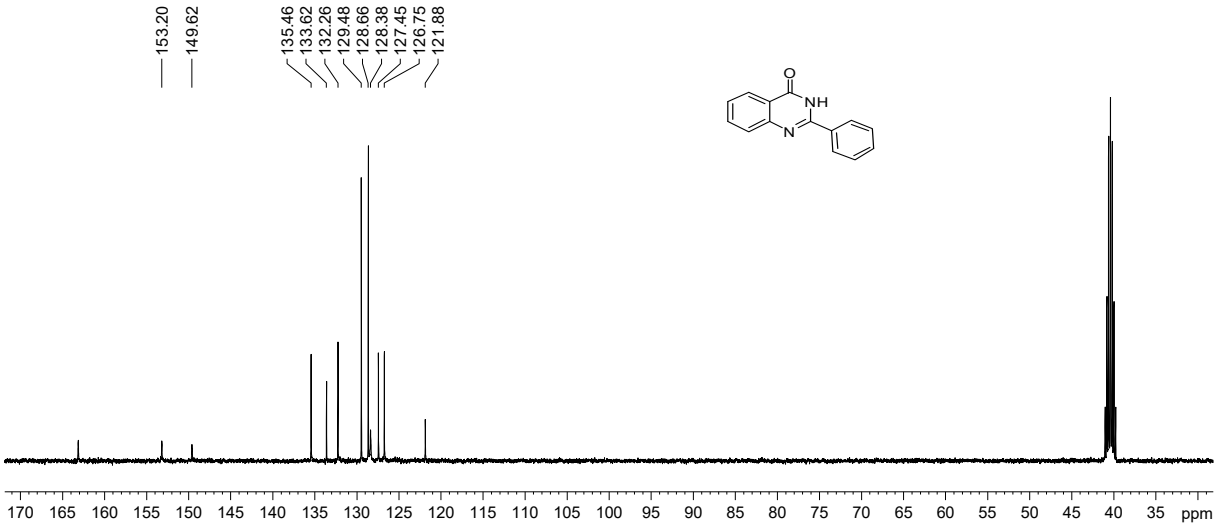

Figure S 3. [1]HNMR of (3b).

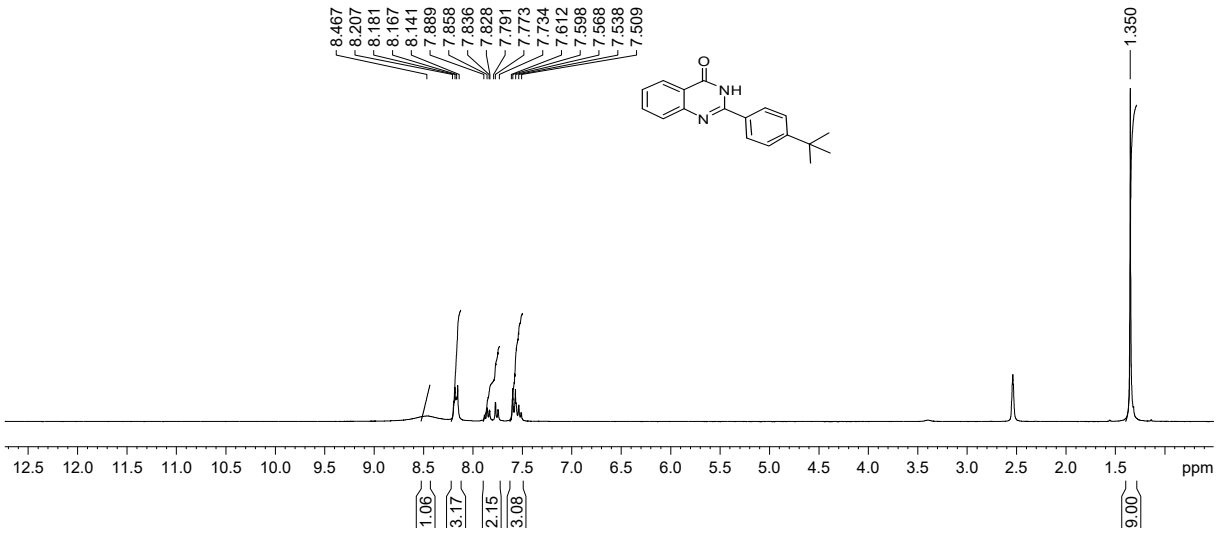

Figure S 4. [13]CNMR of (3b).

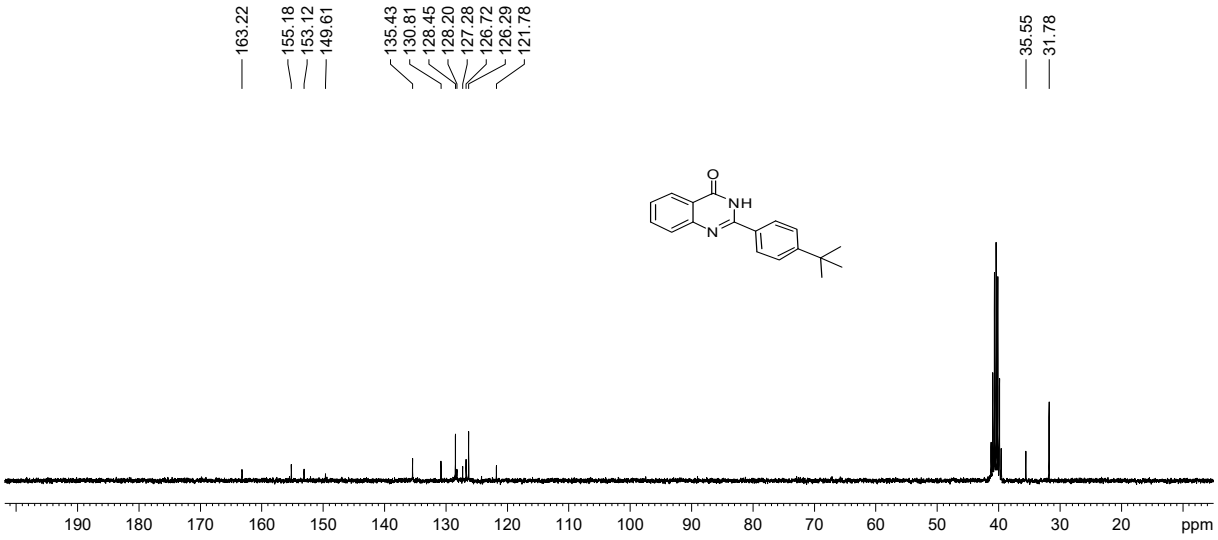

Figure S 5. [1]HNMR of (3c).

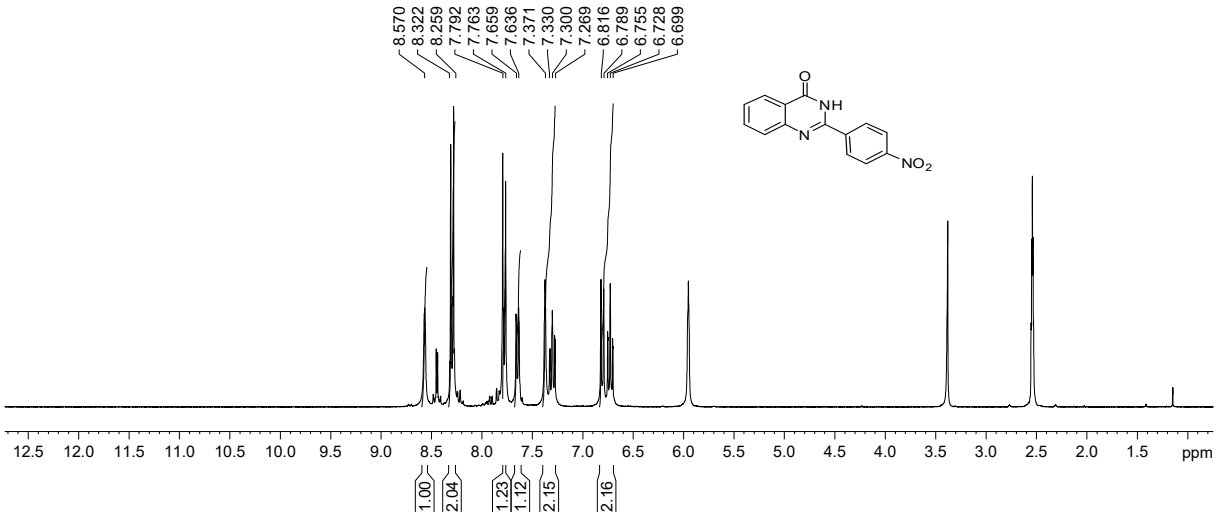

Figure S 6. ^{13}CNMR of (3c).

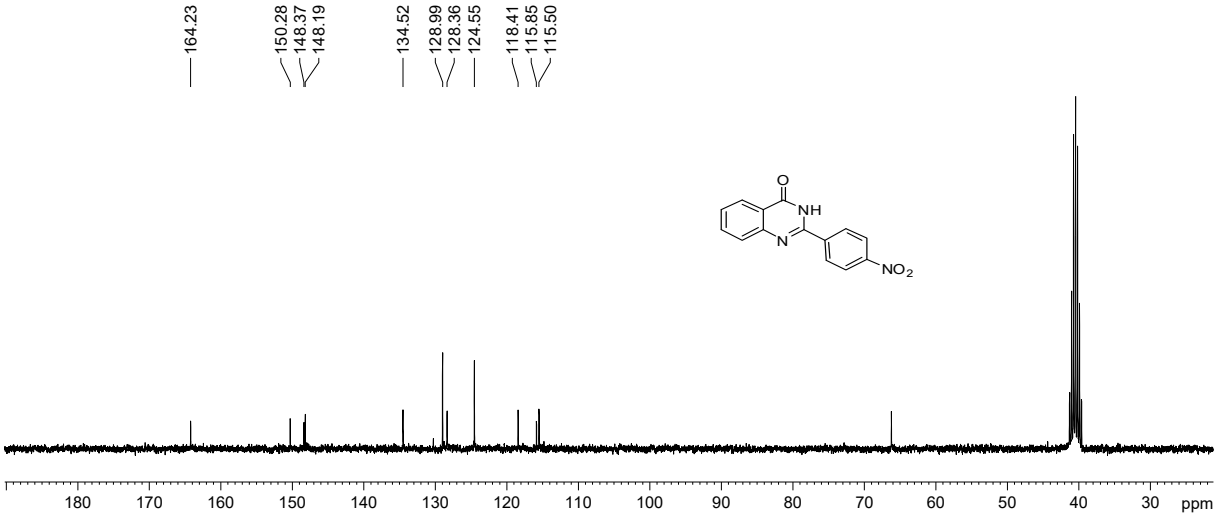

Figure S 7. ^{1}HNMR of (3d).

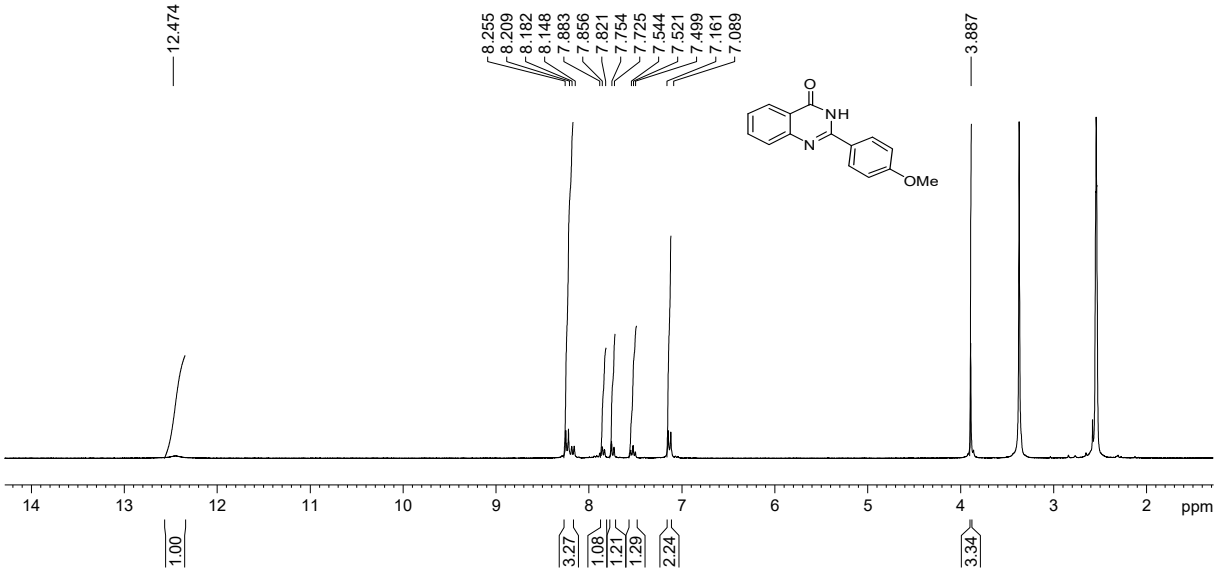

Figure S 8. ^{1}HNMR of (3e).

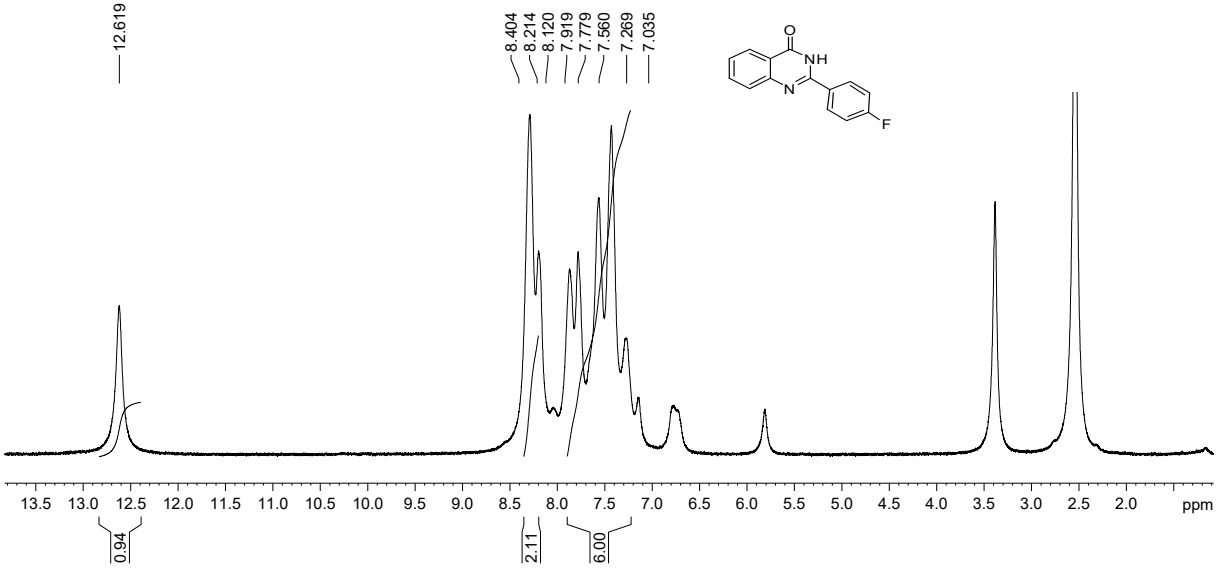

Figure S 9. ^{13}CNMR of (3e).

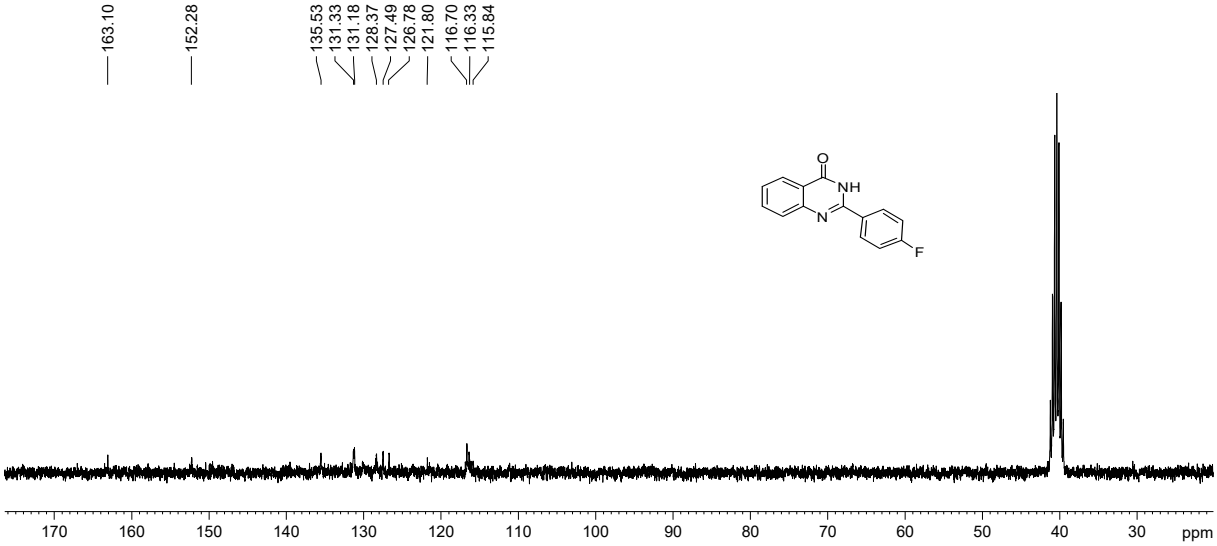

Figure S 10. ^{1}HNMR of (3f).

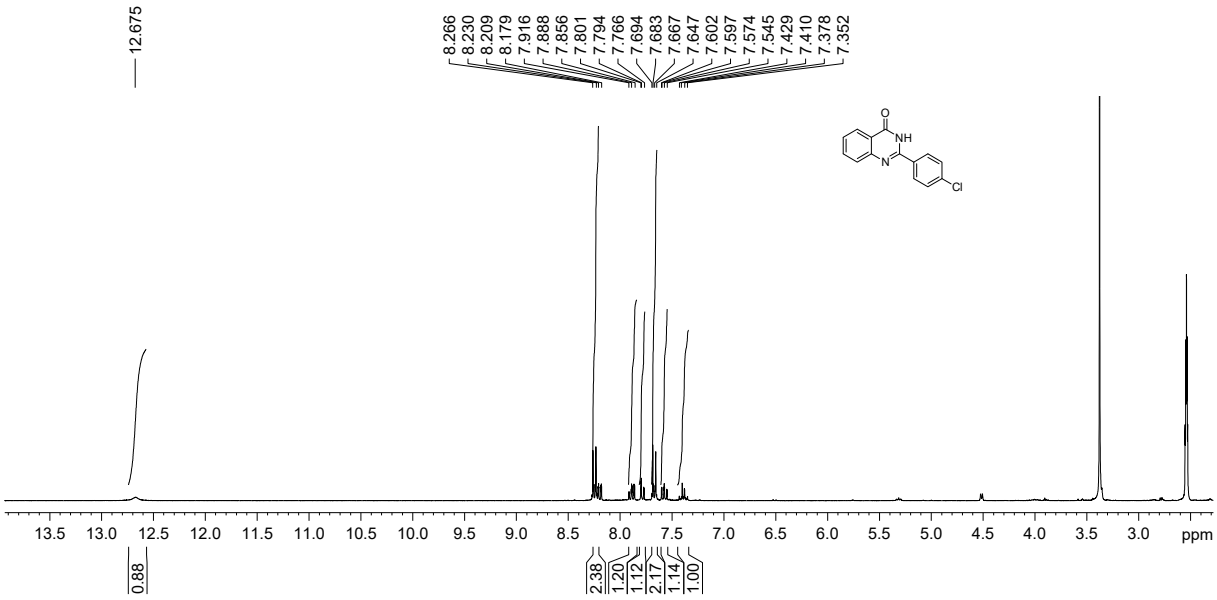

Figure S 11. ^{13}CNMR of (3f).

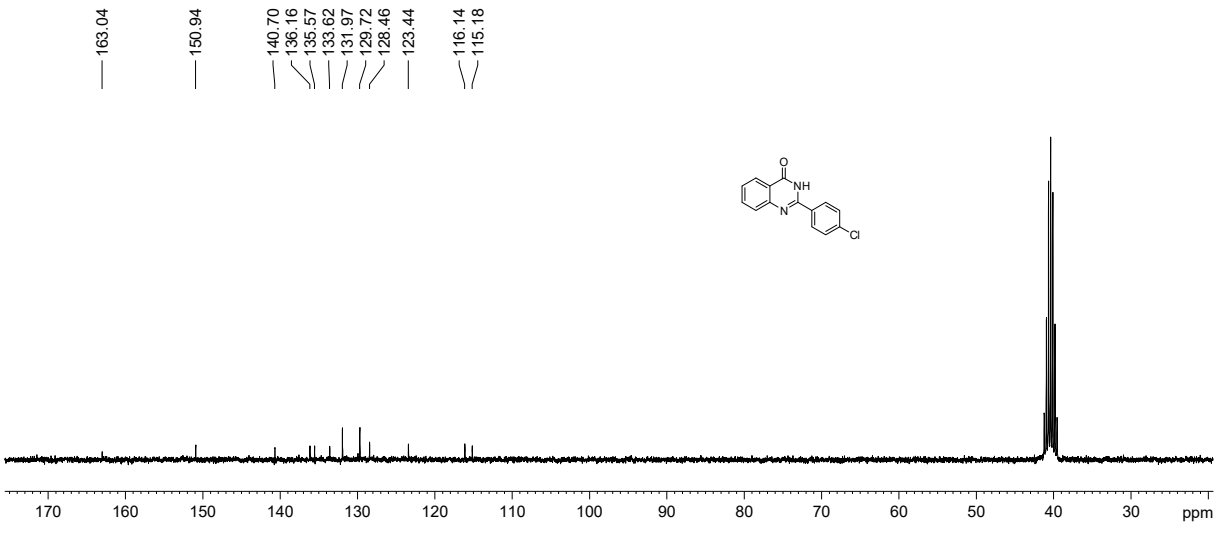

Figure S 12. [1]HNMR (3g).

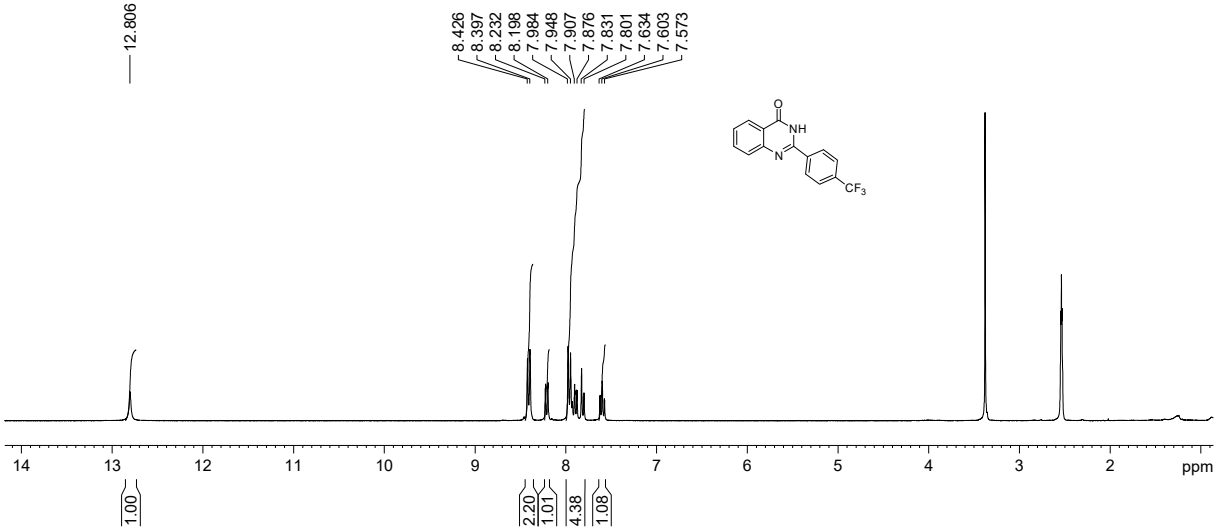

Figure S 13. [13]CNMR of (3g).

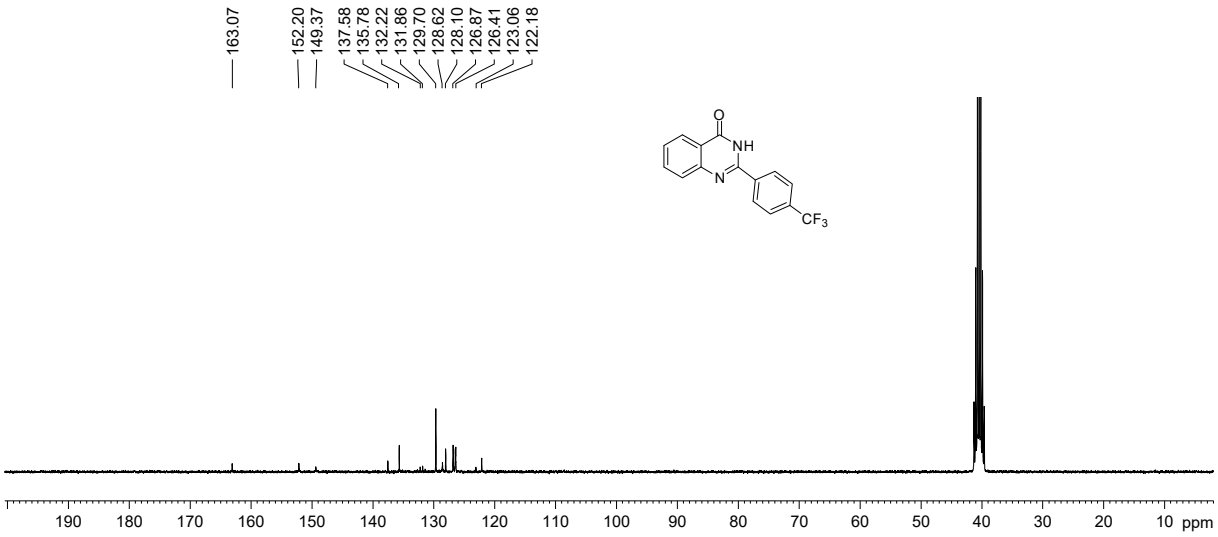

Figure S 14. [1]HNMR of (3h).

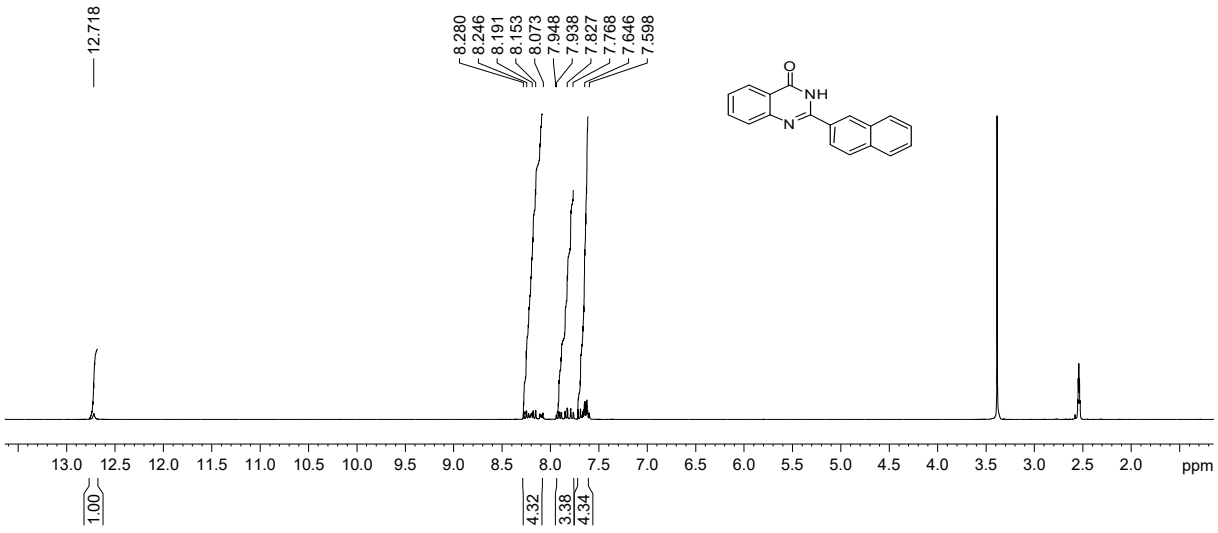

Figure S 15. ^{13}CNMR of (3h).

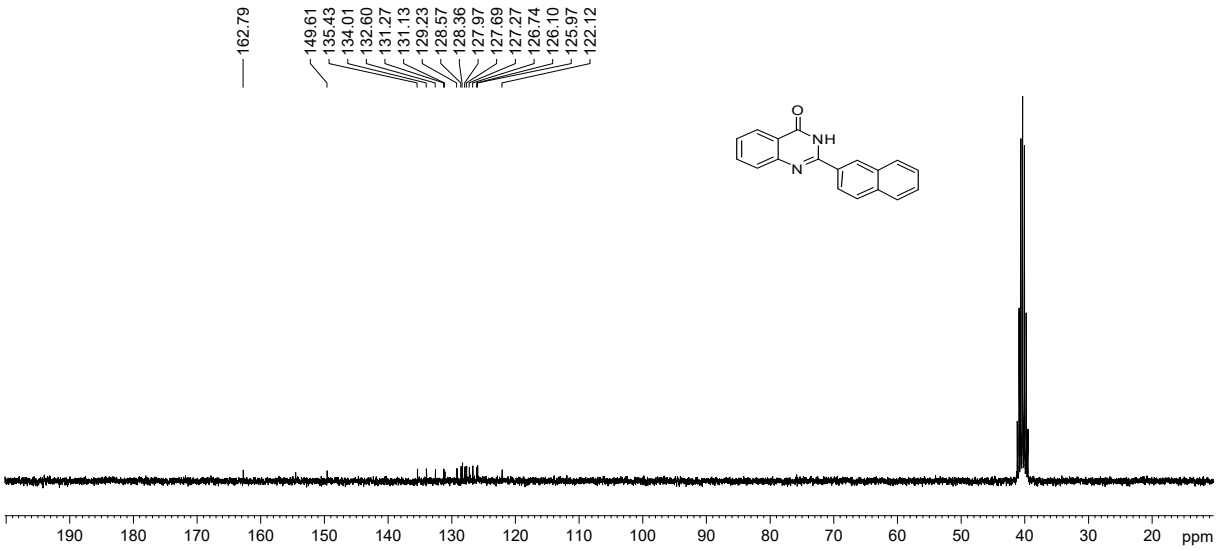

Figure S 16. ^{1}HNMR of (3i).

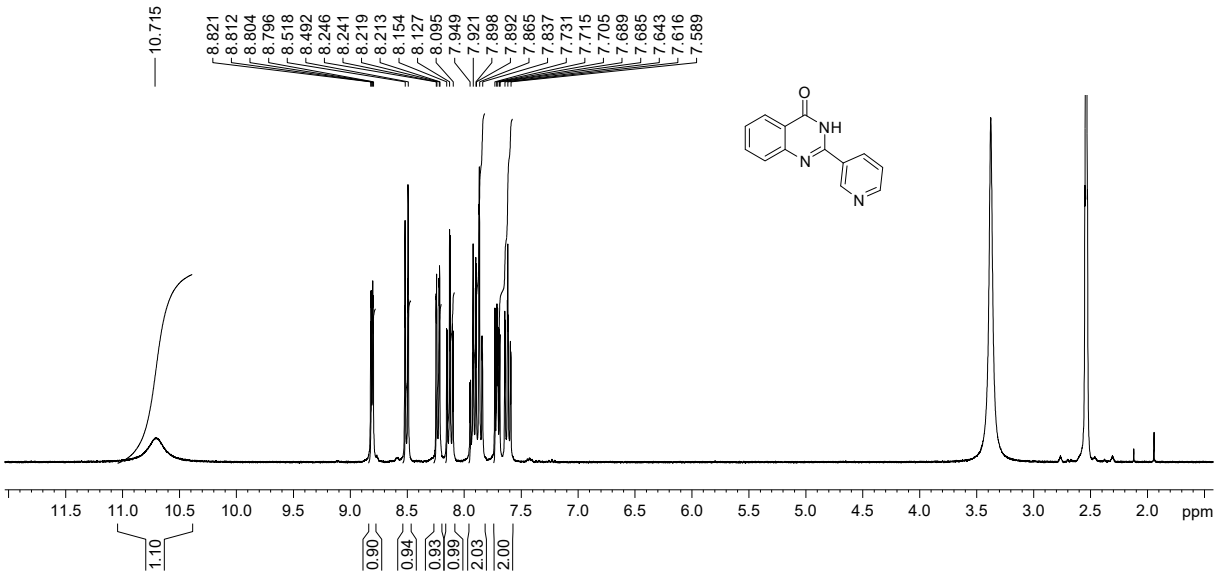

Figure S 17. ^{13}CNMR of (3i).

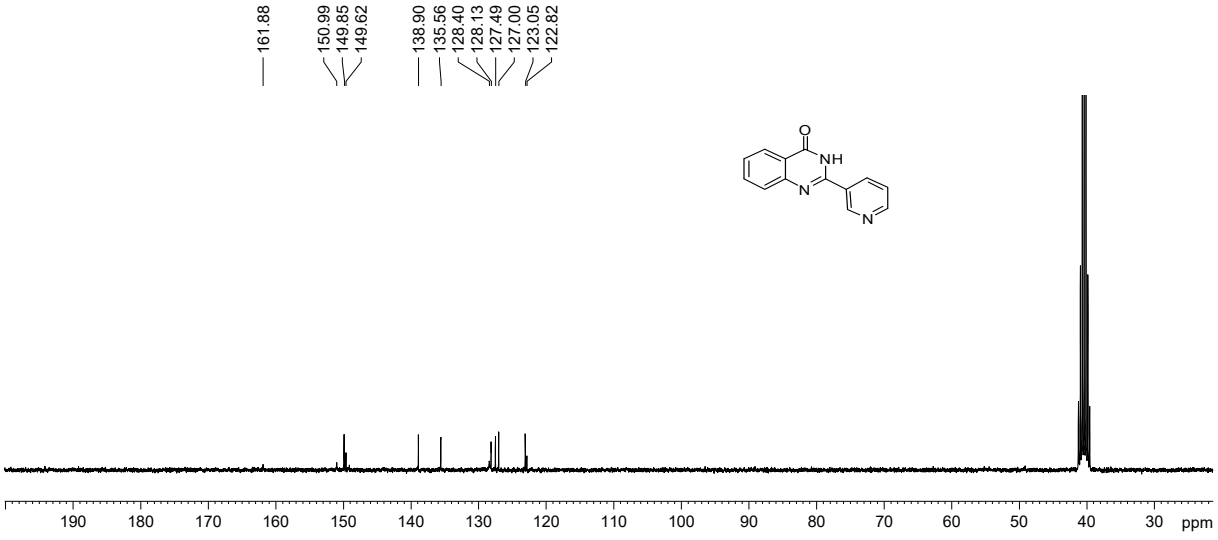

Figure S 18. [1]HNMR of (5).

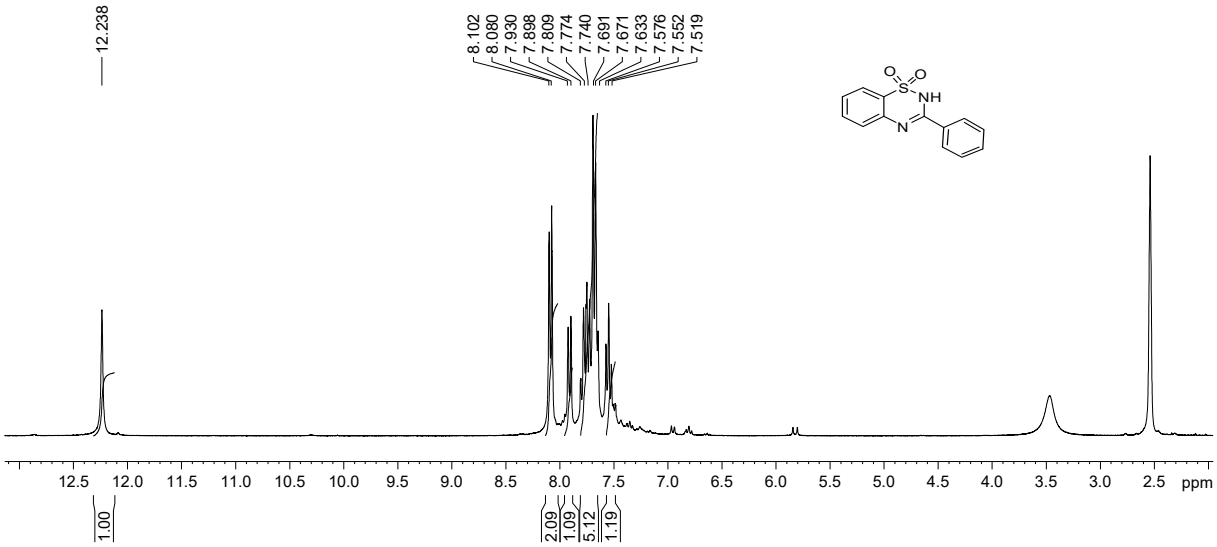

Figure S 19. [13]CNMR (5).

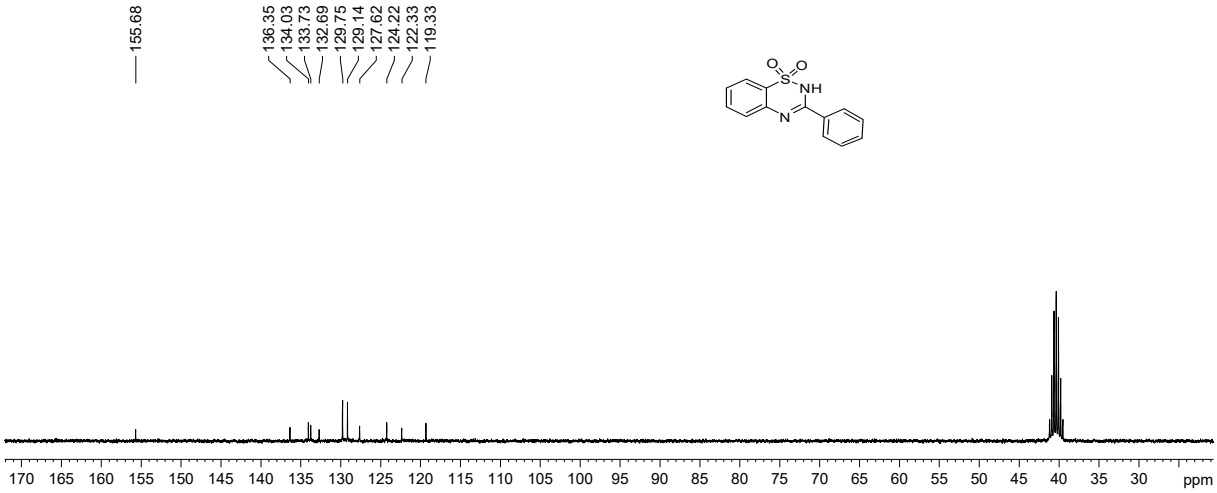

Figure S 20. [1]HNMR of (5a).

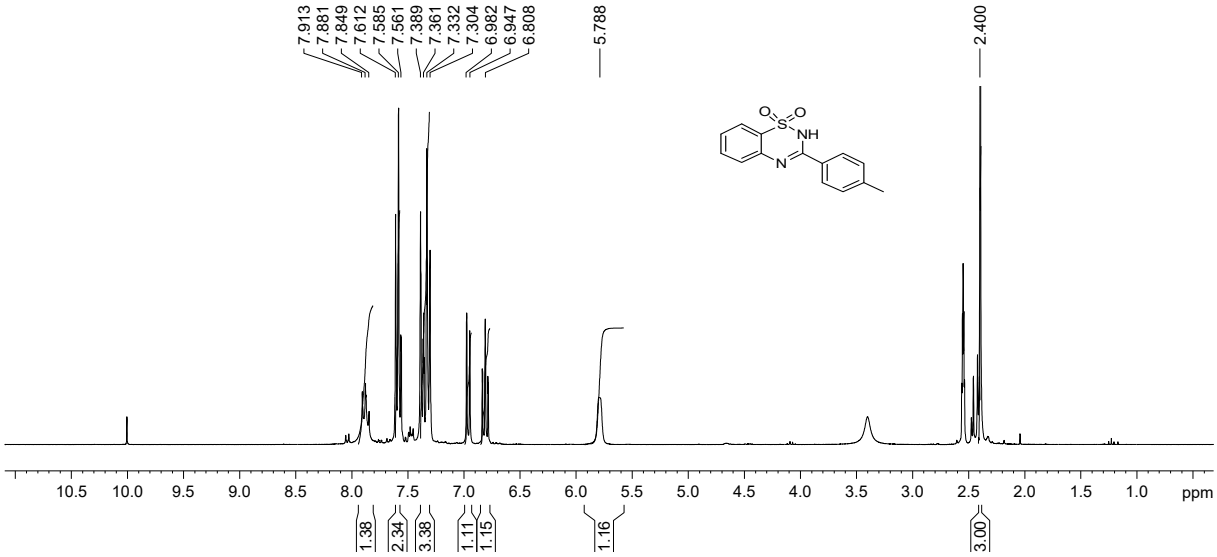

Figure S 21. ^{13}CNMR of (5b).

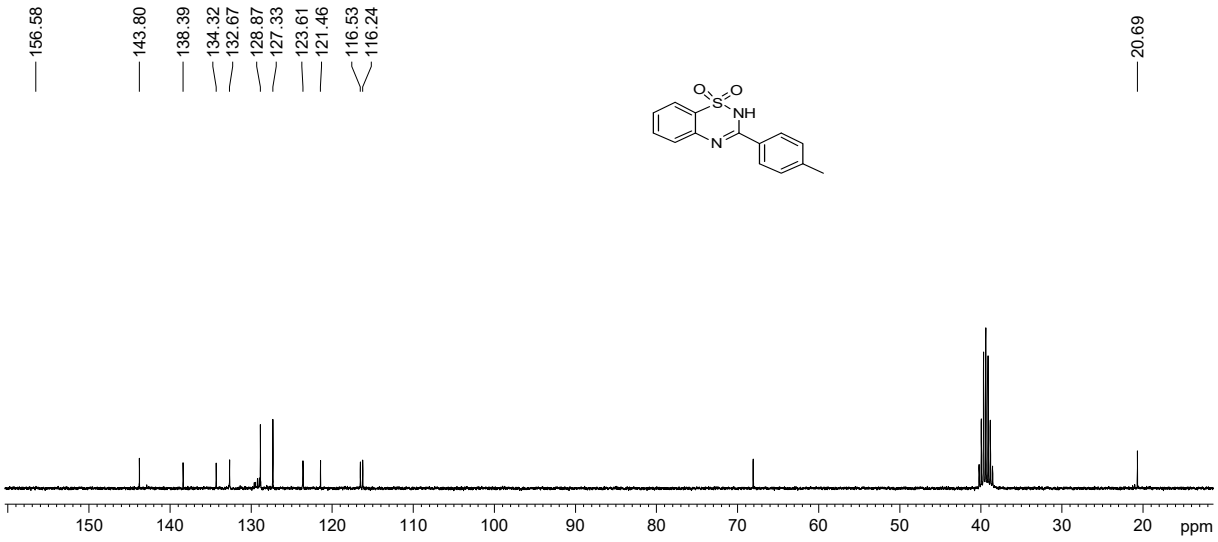

Figure S 22. ^{1}HNMR of (5b).

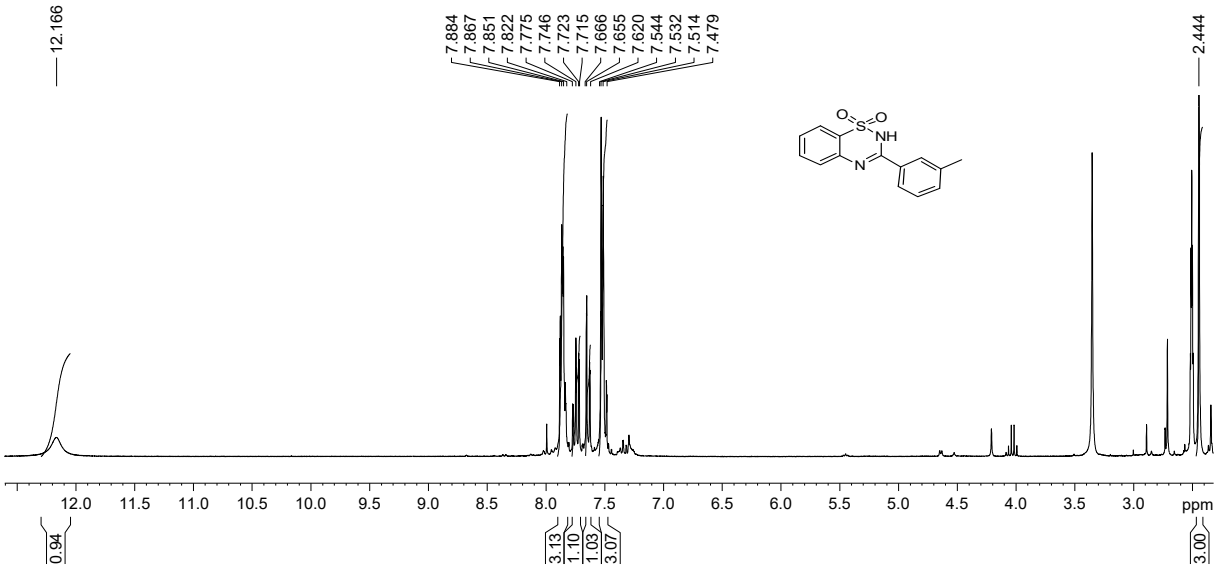

Figure S 23. ^{13}CNMR (5b).