

Facile “on water” domino reactions for the expedient synthesis of *2H*-thiopyrano[2,3-*b*]quinolines

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Supporting Information

1. Experimental Section

1.1. General methods

The melting points were measured in open capillary tubes and are uncorrected. The ¹H NMR, ¹³C NMR, DEPT, H,H-COSY, C,H-COSY and HMBC spectra were recorded on a Bruker (Avance) 300 MHz NMR instrument using TMS as internal standard and CDCl₃ as solvent. Standard Bruker software was used throughout. Chemical shifts are given in parts per million (δ-scale) and the coupling constants are given in Hertz. Silica gel-G plates (Merck) were used for TLC analysis with a mixture of petroleum ether (60–80°C) and ethyl acetate as eluent. Mass spectra were recorded in LCQ Fleet mass spectrometer, Thermo Fisher Instruments Limited, US. Electrospray ionisation mass spectrometry (ESI-MS) analysis was performed in the positive ion and negative ion mode on a liquid chromatography ion trap.

General procedure for the synthesis of 3-nitro-2-aryl-*2H*-thiopyrano[2,3-*b*]quinolines **3a-o**:

A mixture of 2-mercaptoquinoline-3-carbaldehyde (1.0 mmol) and substituted β-nitrostyrenes (1.0 mmol) in the presence of triethylamine (TEA) (0.25 mmol) in water (10 ml) was heated under reflux at 100°C for 3-4 h. After completion of the reaction (TLC), the resulting crude product was purified by washing with cold ethanol which afforded a library of 3-nitro-2-aryl-*2H*-thiopyrano[2,3-*b*]quinolines **3** in excellent yields.

2-(4-fluorophenyl)-3-nitro-2*H*-thiopyrano[2,3-*b*]quinoline 3a: Yellow solid. Yield: 87%; mp. 167–168 °C; ¹H NMR (300 MHz, CDCl₃) δ_H: 5.77 (s, 1H), 6.89–6.96 (m, 2H), 7.20–7.26 (m, 2H), 7.52–7.57 (m, 1H), 7.77 (td, *J* = 8.4, 1.2 Hz, 1H), 7.84 (d, *J* = 8.1 Hz, 1H), 7.95 (d, *J* = 8.4 Hz, 1H), 8.20 (s, 1H), 8.31 (s, 1H); ¹³C NMR (75 MHz, CDCl₃) δ_C: 41.5, 116.0 (²*J*_{C,F} = 21.7 Hz), 121.9, 126.1, 127.0, 128.1 (³*J*_{C,F} = 8.4 Hz), 128.4, 128.5, 130.9, 132.5, 136.0 (⁴*J*_{C,F} = 3.3 Hz), 139.4, 145.2, 149.4, 155.1, 162.6 (¹*J*_{C,F} = 246.6 Hz); ESI-MS: *m/z*. Calcd: 338.05; Found: 339.14 (M⁺).

2-(4-chlorophenyl)-3-nitro-2*H*-thiopyrano[2,3-*b*]quinoline 3b: Pale Yellow solid. Yield: 89%; mp. 163–164 °C; ¹H NMR (300 MHz, CDCl₃) δ_H: 5.75 (s, 1H), 7.17–7.23 (m, 4H), 7.53–7.58 (m, 1H), 7.77 (td, *J* = 8.4, 1.2 Hz, 1H), 7.84 (d, *J* = 8.1 Hz, 1H), 7.94 (d, *J* = 8.7 Hz, 1H), 8.21 (s, 1H), 8.32 (s, 1H); ¹³C NMR (75 MHz, CDCl₃) δ_C: 41.6, 121.9, 126.1, 127.1, 127.3, 127.7, 128.5, 129.3, 131.1, 132.6, 134.5, 138.6, 139.5, 145.0, 149.4, 155.0; ESI-MS: *m/z*. Calcd: 354.02; Found: 355.19 (M⁺).

2-(4-bromophenyl)-3-nitro-2*H*-thiopyrano[2,3-*b*]quinoline 3c: Pale Yellow solid. Yield: 92%; mp. 198–199 °C; ¹H NMR (300 MHz, CDCl₃) δ_H: 5.74 (s, 1H), 7.13 (d, *J* = 8.4 Hz, 2H), 7.35–7.38 (m, 2H), 7.53–7.58 (m, 1H), 7.76 (td, *J* = 7.8, 1.3 Hz, 1H), 7.85 (d, *J* = 8.4 Hz, 1H), 7.95 (d, *J* = 8.4 Hz, 1H), 8.21 (s, 1H), 8.32 (s, 1H); ¹³C NMR (75 MHz, CDCl₃) δ_C: 41.7, 121.9, 122.7, 126.1, 127.1, 128.0, 128.5, 128.5, 131.1, 132.3, 132.6, 139.1, 139.5, 144.9, 149.4, 154.9; ESI-MS: *m/z*. Calcd: 397.97; Found: 399.06 (M⁺), 401.12 (M+3).

3-nitro-2-(4-(trifluoromethyl)phenyl)-2*H*-thiopyrano[2,3-*b*]quinoline 3d: Yellow solid. Yield: 85%; mp. 160–161 °C; ¹H NMR (300 MHz, CDCl₃) δ_H: 5.82 (s, 1H), 7.38 (d, *J* = 8.1 Hz, 2H), 7.50–7.57 (m, 3H), 7.77 (t, *J* = 7.6 Hz, 1H), 7.84 (d, *J* = 7.8 Hz, 1H), 7.94 (d, *J* = 8.4 Hz, 1H), 8.29 (s, 1H), 8.35 (s, 1H); ¹³C NMR (75 MHz, CDCl₃) δ_C: 41.8, 121.8, 126.1, 126.2, 126.7, 127.2, 128.5, 128.6, 130.7 (²*J*_{C,F} = 32.9 Hz), 131.5, 132.7, 139.7, 143.8, 144.5, 149.4, 154.7; ESI-MS: *m/z*. Calcd: 388.05; Found: 389.20 (M⁺).

3-nitro-2-phenyl-2*H*-thiopyrano[2,3-*b*]quinoline 3e: Pale Yellow solid. Yield: 94%; mp. 206–207 °C; ¹H NMR (300 MHz, CDCl₃) δ_H: 5.78 (s, 1H), 7.21–7.24 (m, 5H), 7.50–7.56 (m, 1H), 7.75 (td, *J* = 8.4, 1.2 Hz, 1H), 7.83 (d, *J* = 8.1 Hz, 1H), 7.93 (d, *J* = 8.4 Hz, 1H), 8.19 (s, 1H), 8.31 (s, 1H); ¹³C NMR (75 MHz, CDCl₃) δ_C: 42.2, 122.2, 126.4, 126.3, 126.9, 128.4, 128.5, 128.6, 129.1,

130.9, 132.4, 139.3, 140.0, 145.3, 149.3, 155.5; ESI-MS: *m/z*. Calcd: 320.06; Found: 321.21 (M^+).

3-nitro-2-(*p*-tolyl)-2*H*-thiopyrano[2,3-*b*]quinoline 3f: Yellow solid. Yield: 95%; mp. 214–215 °C; ^1H NMR (300 MHz, CDCl_3) δ_{H} : 2.26 (s, 3H), 5.76 (s, 1H), 7.03 (d, $J = 8.4$ Hz, 2H), 7.13 (d, $J = 8.1$ Hz, 2H), 7.53 (t, $J = 7.5$ Hz, 1H), 7.75 (td, $J = 7.8, 1.5$ Hz, 1H), 7.83 (d, $J = 8.4$ Hz, 1H), 7.93 (d, $J = 8.7$ Hz, 1H), 8.18 (s, 1H), 8.30 (s, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ_{C} : 21.0, 42.1, 122.2, 126.2, 126.3, 126.8, 128.4, 128.5, 129.8, 130.5, 132.2, 137.2, 138.5, 139.0, 145.7, 149.4, 155.7; ESI-MS: *m/z*. Calcd: 334.08; Found: 335.18 (M^+).

2-(4-ethylphenyl)-3-nitro-2*H*-thiopyrano[2,3-*b*]quinoline 3g: Pale Yellow solid. Yield: 93%; mp. 217–218 °C; ^1H NMR (300 MHz, CDCl_3) δ_{H} : 1.14 (t, $J = 7.5$ Hz, 3H), 2.55 (q, $J = 7.6$ Hz, 2H), 5.76 (s, 1H), 7.05 (d, $J = 8.1$ Hz, 2H), 7.15 (d, $J = 8.4$ Hz, 2H), 7.50–7.55 (m, 1H), 7.75 (td, $J = 7.7, 1.4$ Hz, 1H), 7.83 (d, $J = 7.8$ Hz, 1H), 7.93 (d, $J = 8.4$ Hz, 1H), 8.18 (s, 1H), 8.30 (s, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ_{C} : 15.3, 28.4, 42.0, 122.2, 126.1, 126.2, 126.9, 128.4, 128.5, 128.6, 130.6, 132.3, 137.3, 139.2, 144.8, 145.5, 149.3, 155.6; ESI-MS: *m/z*. Calcd: 348.09; Found: 349.22 (M^+).

2-(4-(*tert*-butyl)phenyl)-3-nitro-2*H*-thiopyrano[2,3-*b*]quinoline 3h: Pale Yellow solid. Yield: 92%; mp. 252–253 °C; ^1H NMR (300 MHz, CDCl_3) δ_{H} : 1.22 (s, 9H), 5.77 (s, 1H), 7.16 (d, $J = 8.4$ Hz, 2H), 7.24 (d, $J = 8.4$ Hz, 2H), 7.53 (t, $J = 7.5$ Hz, 1H), 7.73–7.78 (m, 1H), 7.84 (d, $J = 8.1$ Hz, 1H), 7.94 (d, $J = 8.4$ Hz, 1H), 8.19 (s, 1H), 8.31 (s, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ_{C} : 31.1, 34.5, 41.9, 122.3, 125.9, 126.0, 126.1, 126.9, 128.4, 128.5, 130.6, 132.3, 136.9, 139.1, 145.6, 149.3, 151.6, 155.6; ESI-MS: *m/z*. Calcd: 376.12; Found: 377.21 (M^+).

2-(4-methoxyphenyl)-3-nitro-2*H*-thiopyrano[2,3-*b*]quinoline 3i: Yellow solid. Yield: 96%; mp. 208–209 °C; ^1H NMR (300 MHz, CDCl_3) δ_{H} : 3.72 (s, 3H), 5.75 (s, 1H), 6.73–6.76 (m, 2H), 7.15–7.18 (m, 2H), 7.51–7.56 (m, 1H), 7.76 (td, $J = 7.7, 1.4$ Hz, 1H), 7.83 (d, $J = 8.1$ Hz, 1H), 7.94 (d, $J = 8.4$ Hz, 1H), 8.19 (s, 1H), 8.28 (s, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ_{C} : 41.8, 55.2, 114.4, 122.2, 126.1, 126.9, 127.6, 128.4, 128.5, 130.4, 132.2, 132.3, 145.6, 149.3, 155.6, 159.7; ESI-MS: *m/z*. Calcd: 350.07; Found: 351.21 (M^+).

2-(2-fluorophenyl)-3-nitro-2*H*-thiopyrano[2,3-*b*]quinoline 3j: Pale Yellow solid. Yield: 90%; mp. 216–217 °C; ^1H NMR (300 MHz, CDCl_3) δ_{H} : 6.10 (s, 1H), 6.94 (t, $J = 7.5$ Hz, 1H),

7.01–7.11 (m, 2H), 7.19–7.24 (m, 1H) 7.54 (t, $J = 7.5$ Hz, 1H), 7.73–7.78 (m, 1H), 7.84 (d, $J = 7.8$ Hz, 1H), 7.93 (d, $J = 8.4$ Hz, 1H), 8.21 (s, 1H), 8.40 (s, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ_{C} : 35.9 ($^3J_{\text{C},\text{F}} = 3.7$ Hz), 116.3 ($^2J_{\text{C},\text{F}} = 21.2$ Hz), 122.0, 124.5 ($^3J_{\text{C},\text{F}} = 3.5$ Hz), 126.2, 127.0, 127.1 ($^4J_{\text{C},\text{F}} = 2.7$ Hz), 127.3, 128.5, 130.3 ($^3J_{\text{C},\text{F}} = 8.3$ Hz), 132.0, 132.4, 139.4, 143.8, 149.4, 155.4, 159.2 ($^1J_{\text{C},\text{F}} = 248.2$ Hz); ESI-MS: m/z . Calcd: 338.05; Found: 339.18 (M^+).

2-(2-methoxyphenyl)-3-nitro-2*H*-thiopyrano[2,3-*b*]quinoline 3k: Yellow solid. Yield: 94%; mp. 199–200 °C; ^1H NMR (300 MHz, CDCl_3) δ_{H} : 3.86, (s, 3H), 6.19 (s, 1H), 6.72 (t, $J = 7.5$ Hz, 1H), 6.89 (d, $J = 8.4$ Hz, 1H), 6.94 (dd, $J = 7.5, 1.5$ Hz, 1H), 7.17–7.23 (m, 1H), 7.51 (t, $J = 7.5$ Hz, 1H), 7.72 (td, $J = 7.7, 1.3$ Hz, 1H), 7.82 (d, $J = 8.1$ Hz, 1H), 7.89 (d, $J = 8.4$ Hz, 1H), 8.18 (s, 1H), 8.40 (s, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ_{C} : 36.4, 55.6, 111.3, 120.5, 122.5, 126.0, 126.1, 126.7, 127.4, 128.3, 128.4, 129.7, 132.0, 132.1, 139.0, 144.4, 149.2, 155.7, 156.7; ESI-MS: m/z . Calcd: 350.07; Found: 351.18 (M^+).

2-(3-fluorophenyl)-3-nitro-2*H*-thiopyrano[2,3-*b*]quinoline 3l: Yellow solid. Yield: 92%; mp. 210–211 °C; ^1H NMR (300 MHz, CDCl_3) δ_{H} : 5.77 (s, 1H), 6.94 (d, $J = 9.3$ Hz, 2H), 7.05 (d, $J = 7.8$ Hz, 1H), 7.19–7.23 (m, 1H), 7.55 (t, $J = 7.5$ Hz, 1H), 7.75 (m, 1H), 7.85 (d, $J = 8.1$ Hz, 1H), 7.95 (d, $J = 8.1$ Hz, 1H), 8.22 (s, 1H), 8.34 (s, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ_{C} : 39.3, 111.5 ($^2J_{\text{C},\text{F}} = 22.3$ Hz), 113.4 ($^2J_{\text{C},\text{F}} = 20.8$ Hz), 120.3 ($^4J_{\text{C},\text{F}} = 2.8$ Hz), 120.4, 124.4, 125.1, 126.1, 127.3, 129.2 ($^3J_{\text{C},\text{F}} = 8.1$ Hz), 130.6, 130.7, 138.9, 141.6 ($^3J_{\text{C},\text{F}} = 6.7$ Hz), 142.4, 147.1, 153.0, 160.7 ($^1J_{\text{C},\text{F}} = 241.0$ Hz); ESI-MS: m/z . Calcd: 338.05; Found: 339.2 (M^+).

2-(3-methoxyphenyl)-3-nitro-2*H*-thiopyrano[2,3-*b*]quinoline 3m: Pale Yellow solid. Yield: 95%; mp. 152–153 °C; ^1H NMR (300 MHz, CDCl_3) δ_{H} : 3.71 (s, 3H), 5.75 (s, 1H), 6.74–6.83 (m, 3H), 7.15 (t, $J = 7.6$ Hz, 1H), 7.53 (t, $J = 7.5$ Hz, 1H), 7.72–7.78 (m, 1H), 7.82 (d, $J = 8.1$ Hz, 1H), 7.93 (d, $J = 8.7$ Hz, 1H), 8.18 (s, 1H), 8.32 (s, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ_{C} : 42.1, 55.2, 112.5, 113.5, 118.5, 122.1, 126.1, 126.9, 128.4, 128.5, 130.2, 131.0, 132.3, 139.3, 141.4, 145.1, 149.3, 155.4, 159.9; ESI-MS: m/z . Calcd: 350.07 Found: 351.20 (M^+).

2-(naphthalen-1-yl)-3-nitro-2*H*-thiopyrano[2,3-*b*]quinoline 3n: Yellow solid. Yield: 91%; mp. 287–288 °C; ^1H NMR (300 MHz, CDCl_3) δ_{H} : 6.62 (s, 1H), 7.14–7.22 (m, 2H), 7.49–7.59 (m, 2H), 7.66–7.75 (m, 3H), 7.86 (t, $J = 7.9$ Hz, 3H), 8.20 (d, $J = 8.7$ Hz, 1H), 8.26 (s, 1H), 8.55 (s, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ_{C} : 37.9, 122.2, 122.7, 123.2, 125.0, 126.1, 126.2, 127.0,

127.1, 128.5, 129.1, 129.2, 129.4, 132.3, 132.4, 134.2, 134.5, 139.5, 144.7, 149.3, 155.5; ESI-MS: m/z. Calcd: 370.08; Found: 371.19 (M⁺).

3-nitro-2-(thiophen-2-yl)-2H-thiopyrano[2,3-b]quinoline 3o: Green solid. Yield: 94%; mp. 201–202 °C; ¹H NMR (300 MHz, CDCl₃) δ_H: 6.06 (s, 1H), 6.82 (dd, *J* = 5.1, 3.6 Hz, 1H), 6.95 (dt, *J* = 3.6, 1.0 Hz, 1H), 7.11 (dd, *J* = 5.1, 1.2 Hz, 1H), 7.55 (ddd, *J* = 8.1, 6.9, 1.2 Hz, 1H), 7.78 (ddd, *J* = 8.4, 6.9, 1.5 Hz, 1H), 7.84 (d, *J* = 8.1 Hz, 1H), 7.98 (d, *J* = 8.1 Hz, 1H), 8.22 (s, 1H), 8.25 (s, 1H); ¹³C NMR (75 MHz, CDCl₃) δ_C: 37.6, 122.1, 125.5, 125.9, 126.2, 127.0, 127.1, 128.5, 130.4, 132.5, 139.5, 143.0, 145.5, 149.4, 155.0, 166.1; ESI-MS: m/z. Calcd: 326.02; Found: 327.14 (M⁺).

2-(4-chlorophenyl)-7-methyl-3-nitro-2H-thiopyrano[2,3-b]quinoline 3p: Yellow solid. Yield: 90% mp. 251–252; ¹H NMR (300 MHz, CDCl₃) δ_H: 2.54 (s, 3H), 5.73 (s, 1H), 7.16–7.22 (m, 4H), 7.59–7.61 (m, 2H), 7.84 (d, *J* = 9.3 Hz, 1H), 8.11 (s, 1H), 8.31 (s, 1H); ¹³C NMR (75 MHz, CDCl₃) δ_C: 21.4, 41.5, 121.8, 126.2, 127.3, 127.7, 128.1, 129.2, 131.3, 134.4, 134.8, 137.2, 138.6, 138.9, 144.8, 148.0, 153.7; ESI-MS: m/z. Calcd: 368.04; Found: 369.06 (M⁺).

7-methyl-3-nitro-2-(p-tolyl)-2H-thiopyrano[2,3-b]quinoline 3q: Light yellow solid. Yield: 93% mp. 238–239; ¹H NMR (300 MHz, CDCl₃) δ_H: 2.25 (s, 3H), 2.53 (s, 3H), 5.74 (s, 1H), 7.02 (d, *J* = 8.1 Hz, 2H), 7.12 (d, *J* = 7.8 Hz, 2H), 7.56–7.58 (m, 2H), 7.83 (d, *J* = 9.0 Hz, 1H), 8.09 (s, 1H), 8.28 (s, 1H); ¹³C NMR (75 MHz, CDCl₃) δ_C: 21.0, 21.4, 41.9, 122.1, 126.2, 127.3, 128.1, 129.7, 130.9, 134.6, 137.0, 137.1, 138.4, 138.6, 145.3, 148.0, 154.3; ESI-MS: m/z. Calcd: 348.09; Found: 349.12 (M⁺).

7-methoxy-3-nitro-2-(p-tolyl)-2H-thiopyrano[2,3-b]quinoline 3r: orange solid. Yield: 91% mp. 197–198; ¹H NMR (300 MHz, CDCl₃) δ_H: 2.25 (s, 3H), 3.94 (s, 3H), 5.72 (s, 1H), 7.00–7.13 (m, 5H), 7.39 (dd, *J* = 9.1, 2.8 Hz, 1H), 7.83 (d, *J* = 9.3 Hz, 1H), 8.01 (s, 1H), 8.27 (s, 1H); ¹³C NMR (75 MHz, CDCl₃) δ_C: 21.0, 41.8, 55.6, 105.8, 122.4, 124.8, 126.1, 127.1, 129.7, 129.8, 130.8, 137.0, 137.9, 138.4, 145.5, 152.2, 157.9; ESI-MS: m/z. Calcd: 364.09; Found: 365.08 (M⁺).

Structure determination using NMR spectroscopic data of **3f**:

The structures of 3-nitro-2-(*p*-tolyl)-2*H*-thiopyrano[2,3-*b*]quinoline **3f** were deduced from one- and two-dimensional NMR spectroscopic data. The structural elucidation using NMR spectroscopy is discussed below.

In the ^1H NMR spectrum of **3f**, the H-2 appears as a singlet at 5.76 ppm and shows HMBCs with C-3, C-4, C-10a and C-2' at 145.7, 126.3, 155.7 and 138.5 ppm respectively. The H-4 gives a singlet at 8.18 ppm which shows HMBCs with C-3, C-5, and C-10a appearing at 145.7, 139.0, and 155.7 respectively. The H-5 appears as a singlet at 8.30 ppm and shows HMBCs with C-10a at 155.7 ppm. The H-6 gives a doublet at 7.83 ppm ($J = 8.4$ Hz) which shows HMBCs with C-5 and C-9a at 139.0 and 149.4 ppm respectively. The H-8 appearing as a triplet of doublets at 7.75 ppm ($J = 7.8, 1.5$ Hz) shows HMBC with C-9a at 149.4 ppm. Likewise, the methyl protons appearing as a singlet at 2.26 ppm shows HMBC with C-4' at 129.8 ppm.

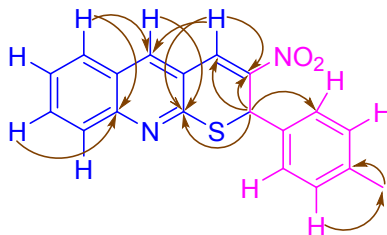
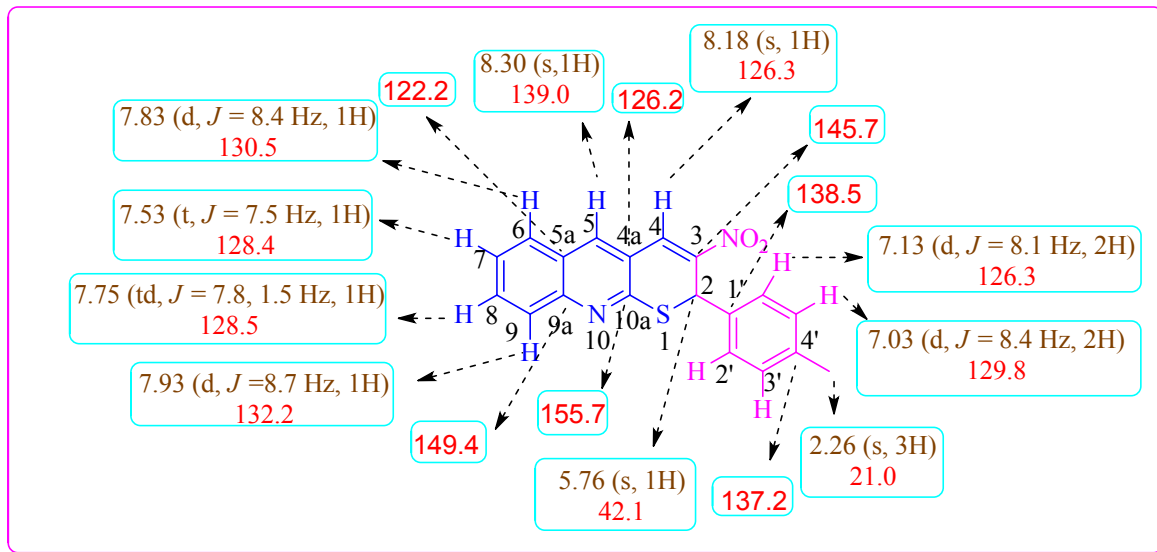
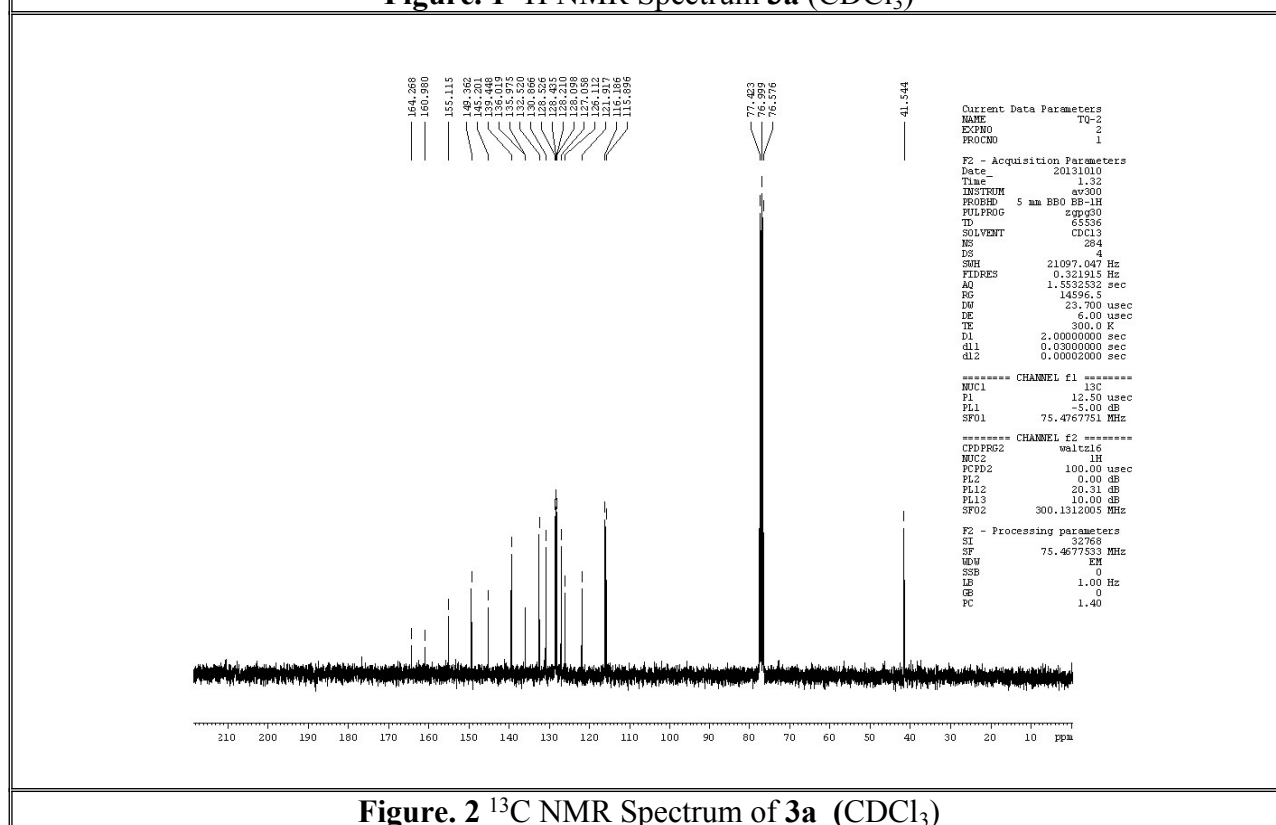
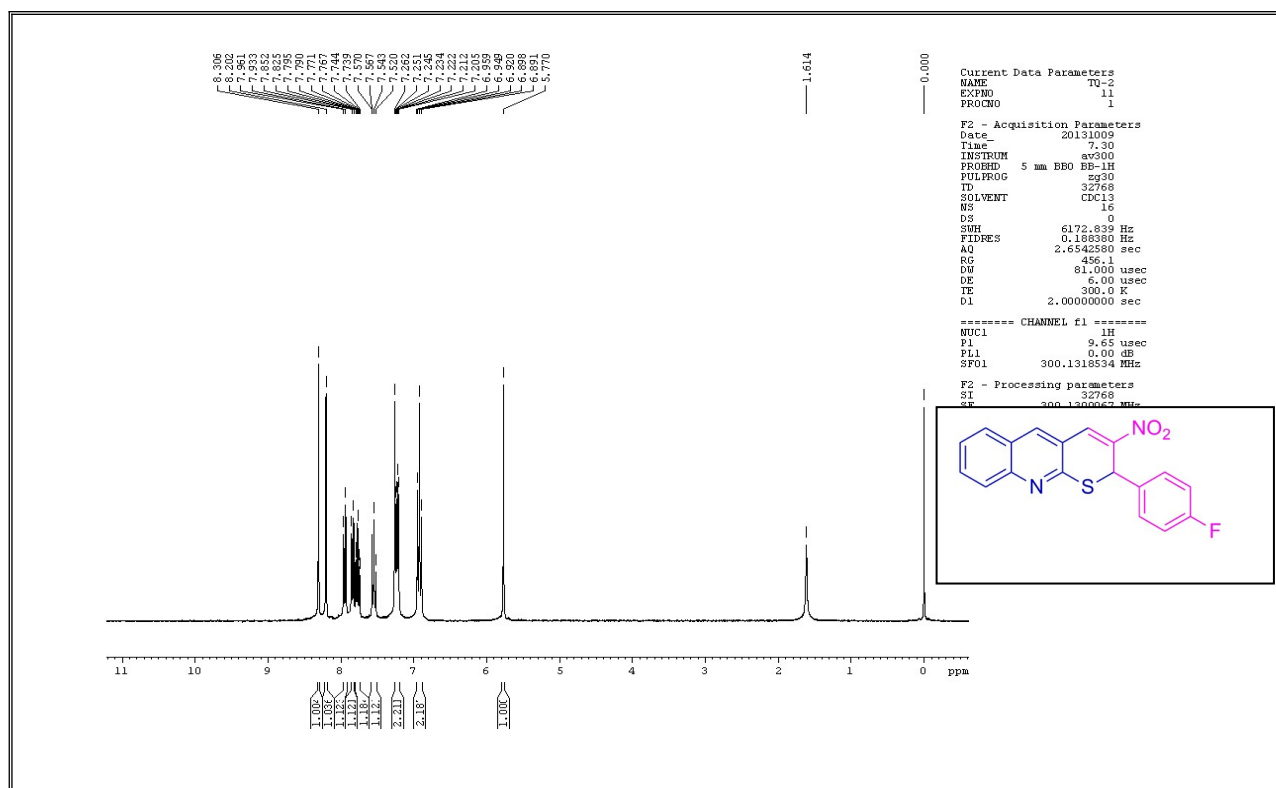


Figure 1. Selected ^1H and ^{13}C Chemical shifts and HMB correlations of **3f**.

No.	List of Figures	Page
1	¹ H NMR Spectrum 3a	10
2	¹³ C NMR Spectrum of 3a	10
3	ESI mass spectrum of Spectrum of 3a	11
4	¹ H NMR Spectrum 3b	11
5	¹³ C NMR Spectrum of 3b	12
6	ESI mass spectrum of Spectrum of 3b	12
7	¹ H NMR Spectrum 3c	13
8	¹³ C NMR Spectrum of 3c	13
9	ESI mass spectrum of Spectrum of 3c	14
10	¹ H NMR Spectrum 3d	14
11	¹³ C NMR Spectrum of 3d	15
12	ESI mass spectrum of Spectrum of 3d	15
13	¹ H NMR Spectrum 3e	16
14	¹³ C NMR Spectrum of 3e	16
15	ESI mass spectrum of Spectrum of 3e	17
16	¹ H NMR Spectrum 3f	17
17	¹³ C NMR Spectrum of 3f	18
18	DEPT Spectrum of 3f	18
19	H, H-COSY Spectrum of 3f	19
20	HMBC Spectrum of 3f	19
21	C, H-COSY Spectrum of 3f	20
22	ESI mass spectrum of Spectrum of 3f	20
23	¹ H NMR Spectrum 3g	21
24	¹³ C NMR Spectrum of 3g	21
25	ESI mass spectrum of Spectrum of 3g	22
26	¹ H NMR Spectrum 3h	22
27	¹³ C NMR Spectrum of 3h	23

28	ESI mass spectrum of Spectrum of 3h	23
29	¹ H NMR Spectrum 3i	24
30	¹³ C NMR Spectrum of 3i	24
31	ESI mass spectrum of Spectrum of 3i	25
32	¹ H NMR Spectrum 3j	25
33	¹³ C NMR Spectrum of 3j	26
34	ESI mass spectrum of Spectrum of 3j	26
35	¹ H NMR Spectrum 3k	27
36	¹³ C NMR Spectrum of 3k	27
37	ESI mass spectrum of Spectrum of 3k	28
38	¹ H NMR Spectrum 3l	28
39	¹³ C NMR Spectrum of 3l	29
40	ESI mass spectrum of Spectrum of 3l	29
41	¹ H NMR Spectrum 3m	30
42	¹³ C NMR Spectrum of 3m	30
43	ESI mass spectrum of Spectrum of 3m	31
44	¹ H NMR Spectrum 3n	31
45	¹³ C NMR Spectrum of 3n	32
46	ESI mass spectrum of Spectrum of 3n	32
47	¹ H NMR Spectrum 3o	33
48	¹³ C NMR Spectrum of 3o	33
49	ESI mass spectrum of Spectrum of 3o	34
50	¹ H NMR Spectrum 3p	34
51	¹³ C NMR Spectrum of 3p	35
52	ESI mass spectrum of Spectrum of 3p	35

53	¹ H NMR Spectrum 3q	36
54	¹³ C NMR Spectrum of 3q	36
55	ESI mass spectrum of Spectrum of 3q	37
56	¹ H NMR Spectrum 3r	37
57	¹³ C NMR Spectrum of 3r	38
58	ESI mass spectrum of Spectrum of 3r	38



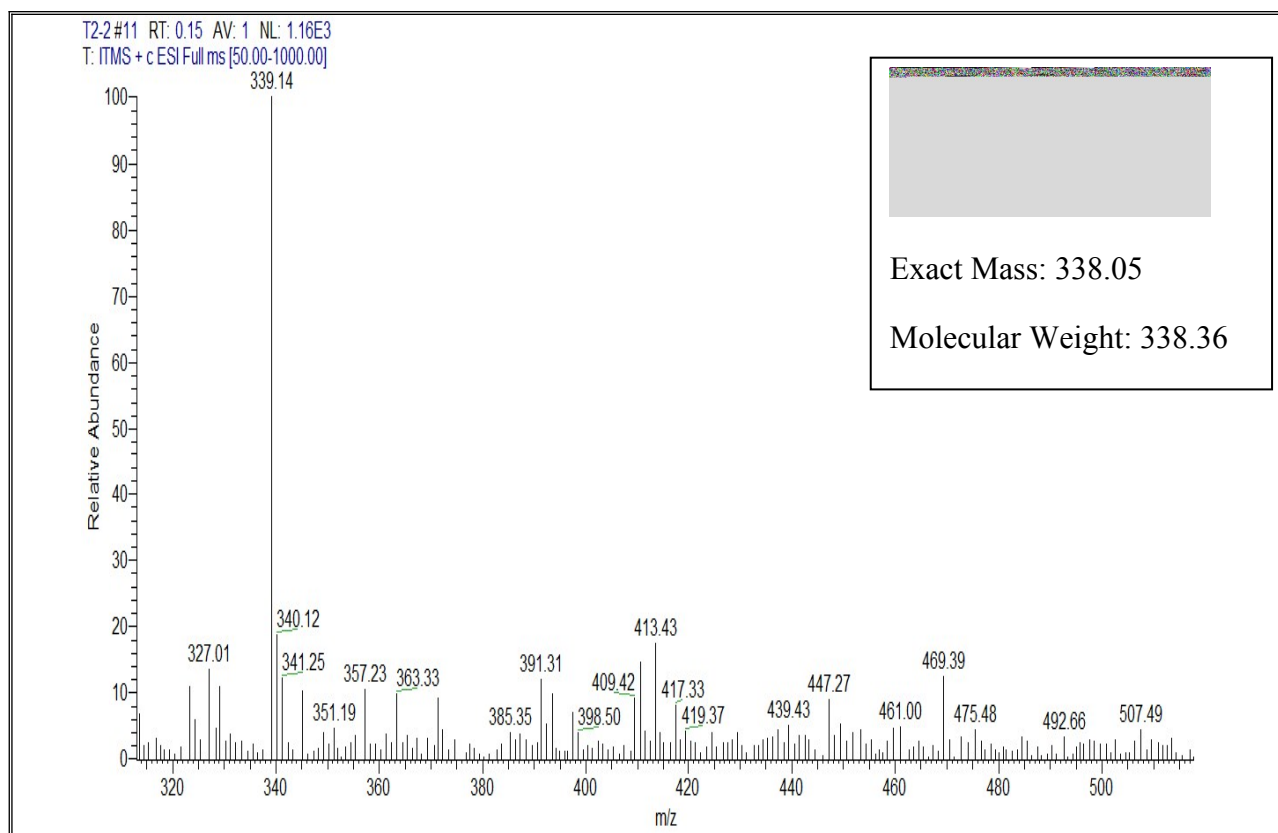


Figure. 3 ESI mass spectrum of Spectrum of **3a**

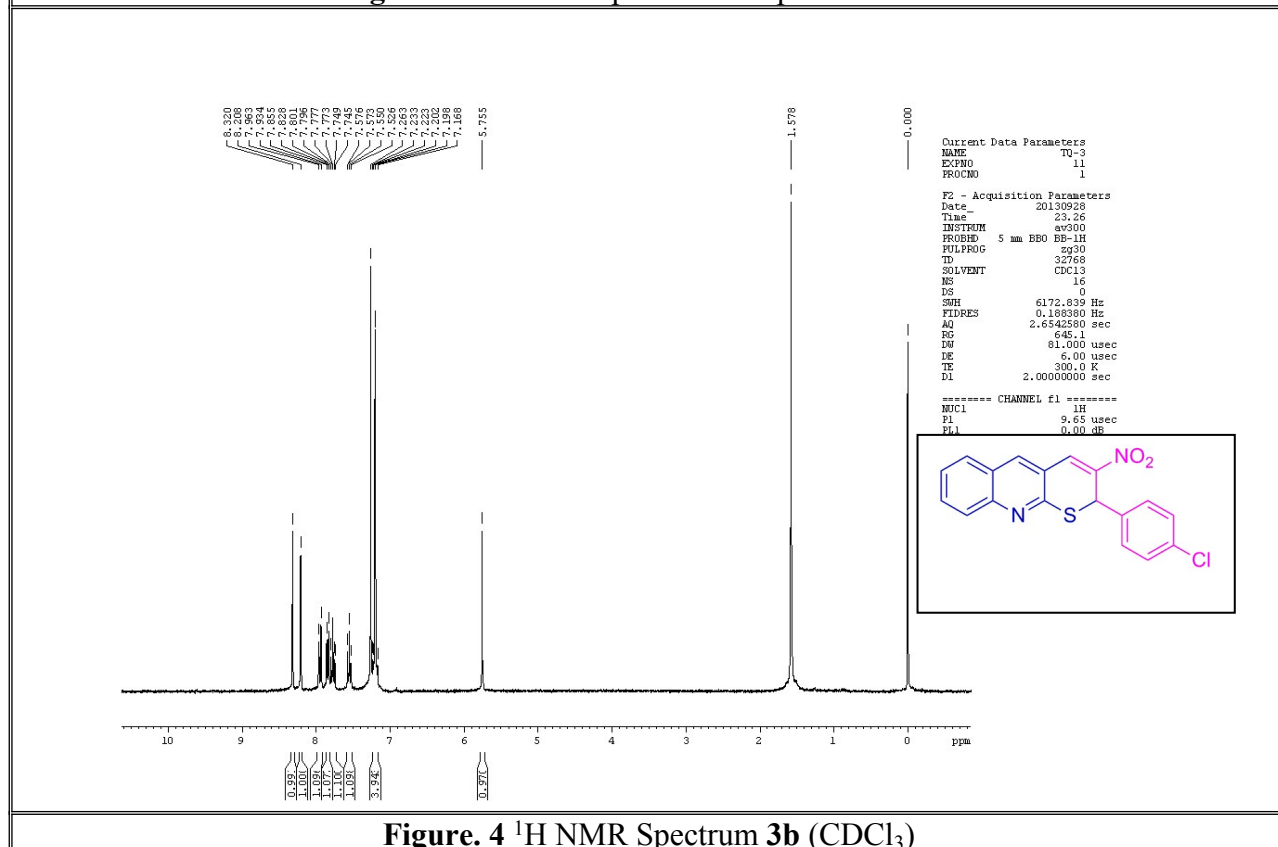


Figure. 4 ^1H NMR Spectrum **3b** (CDCl_3)

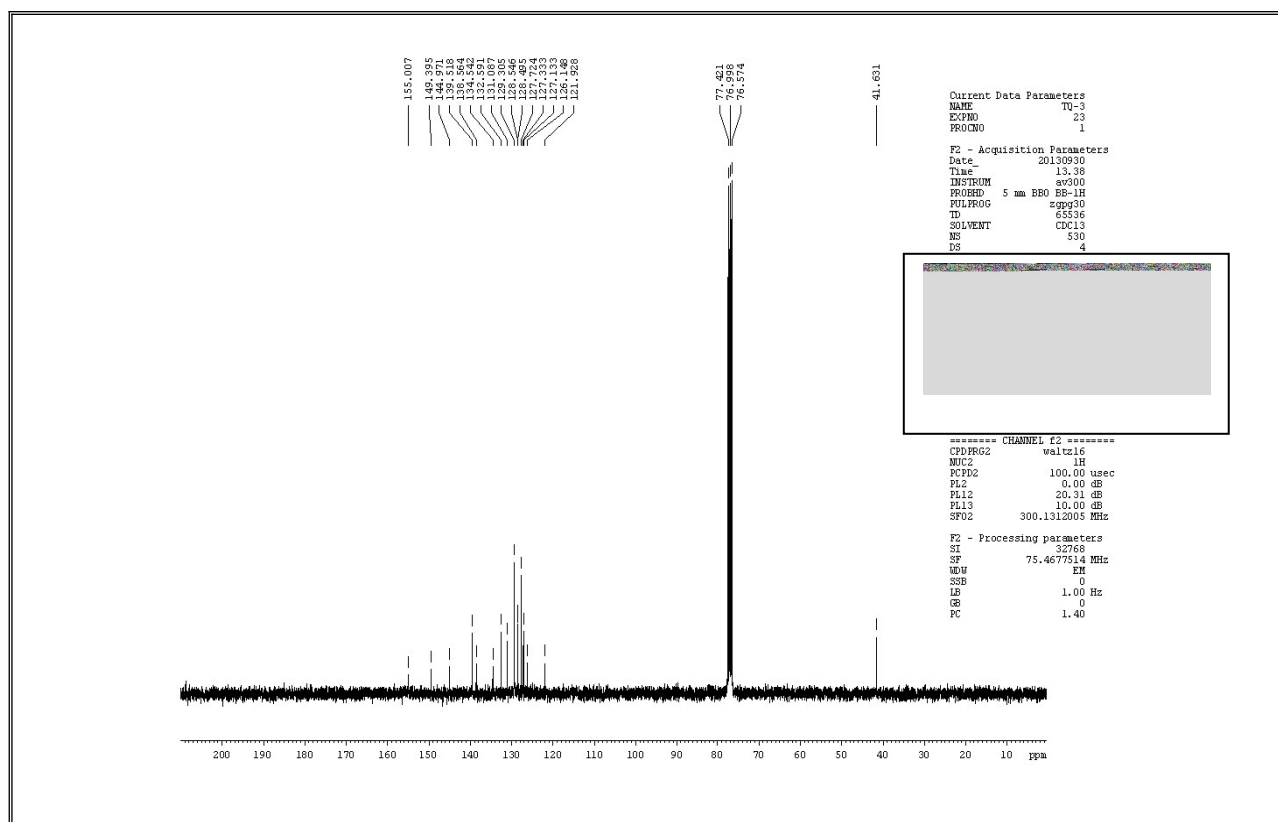


Figure. 5 ^{13}C NMR Spectrum of **3b** (CDCl_3)

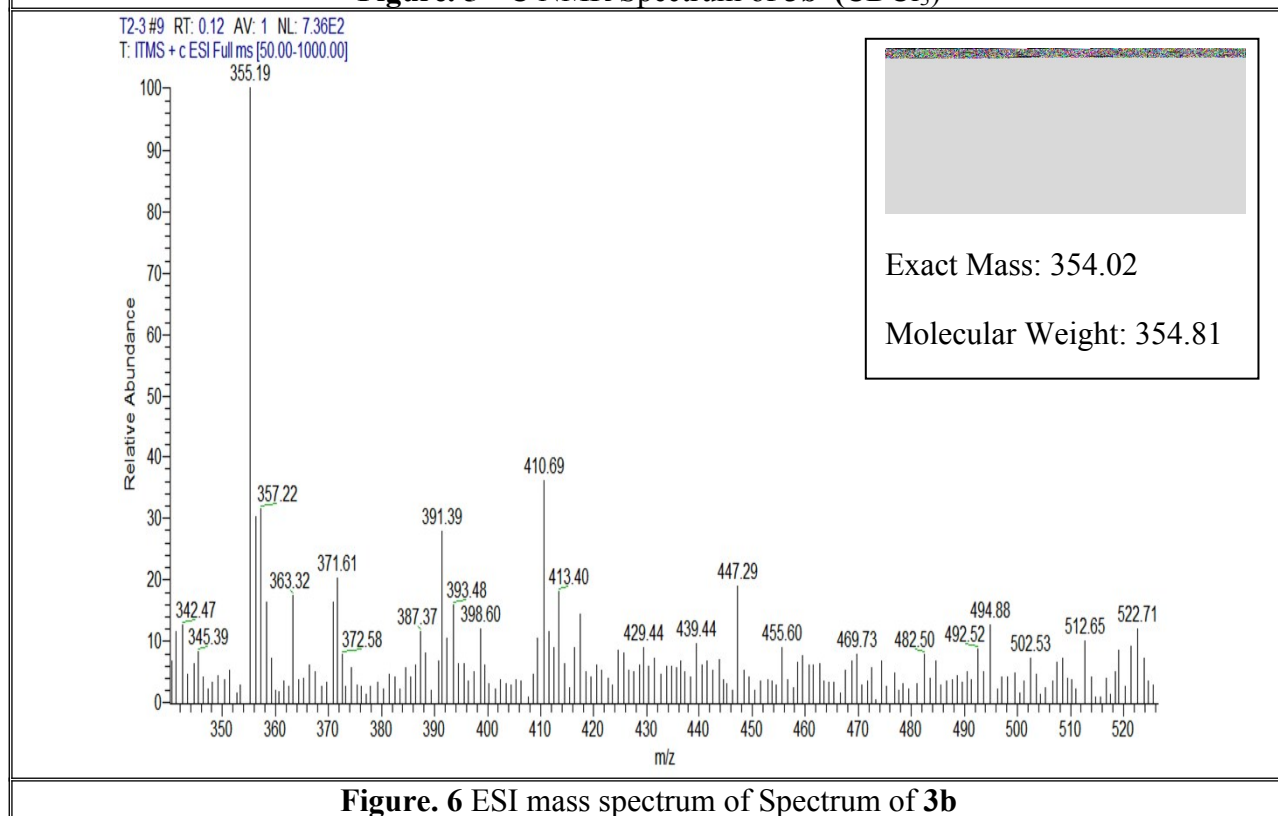


Figure. 6 ESI mass spectrum of Spectrum of **3b**

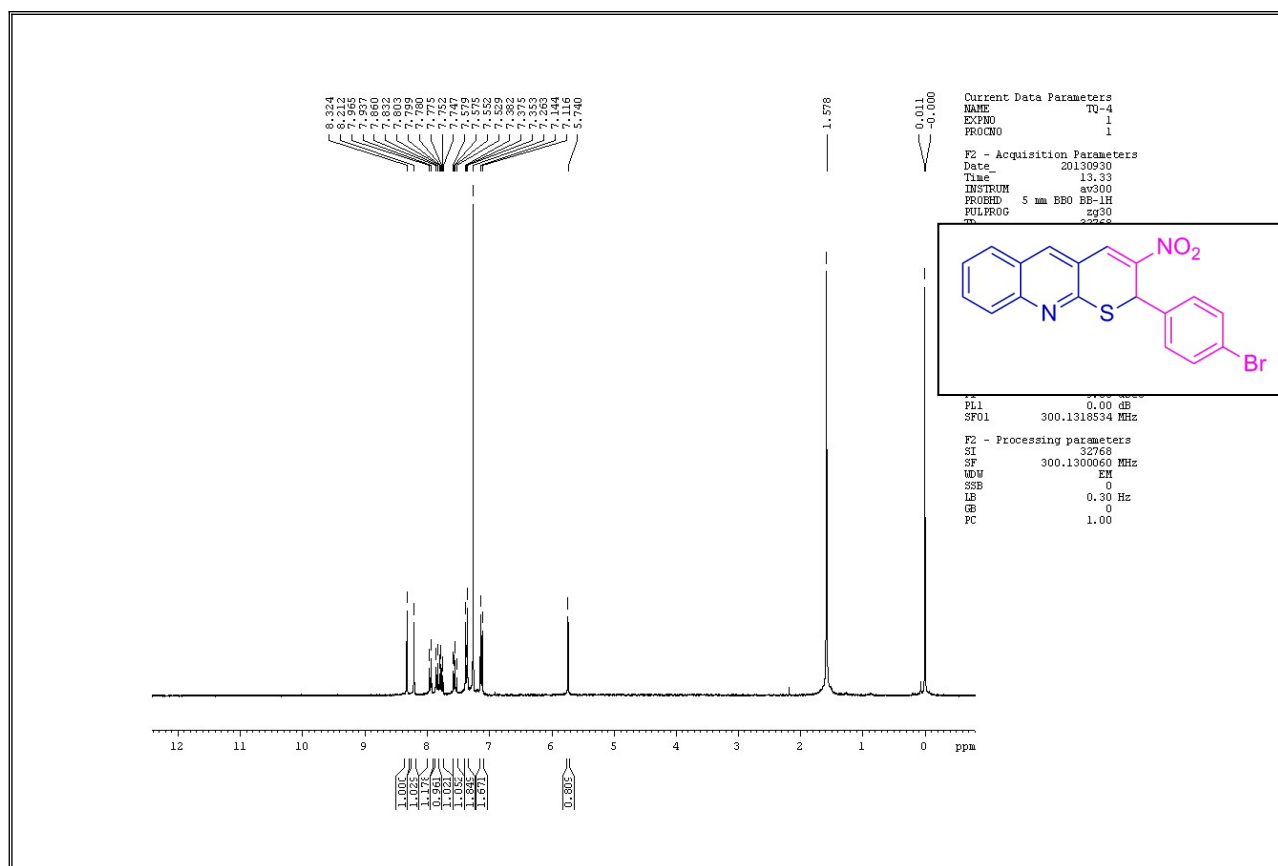


Figure. 7 ^1H NMR Spectrum **3c** (CDCl_3)

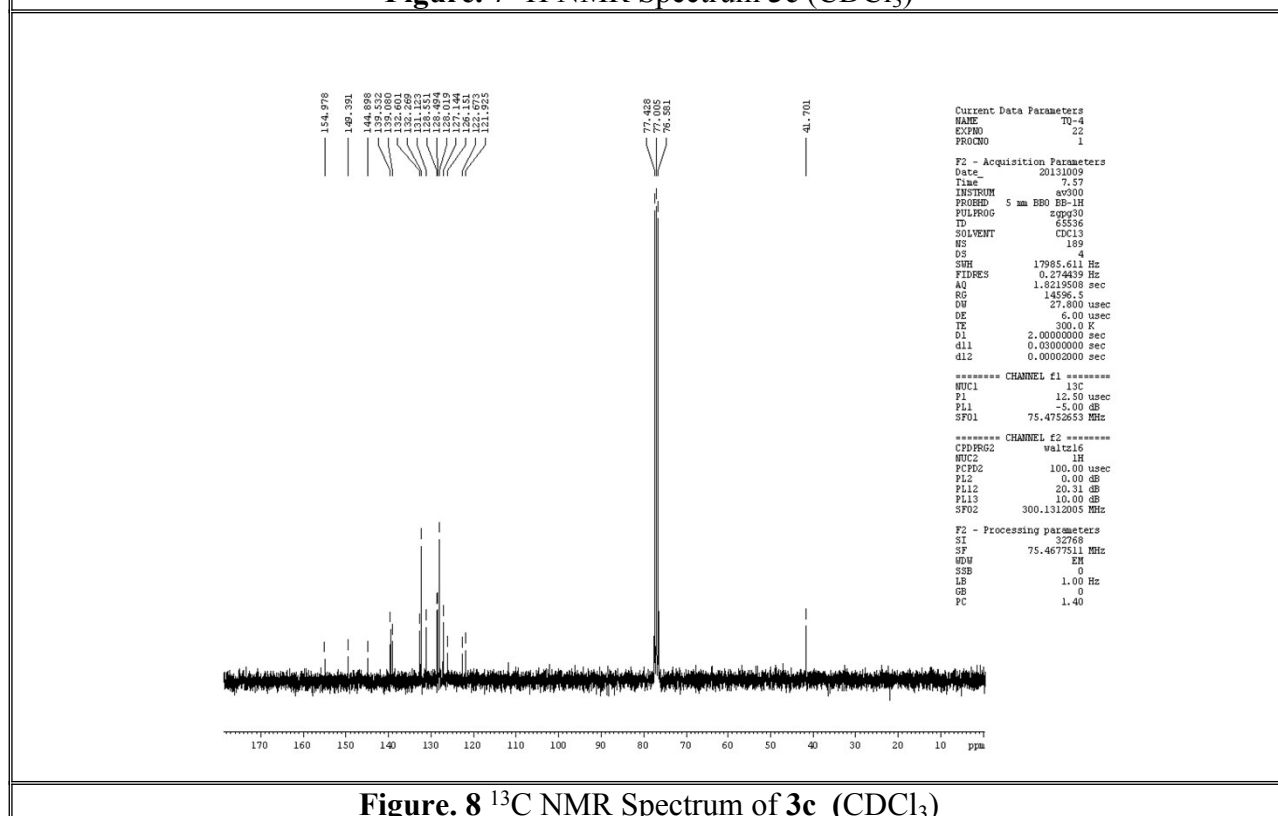


Figure. 8 ^{13}C NMR Spectrum of **3c** (CDCl_3)

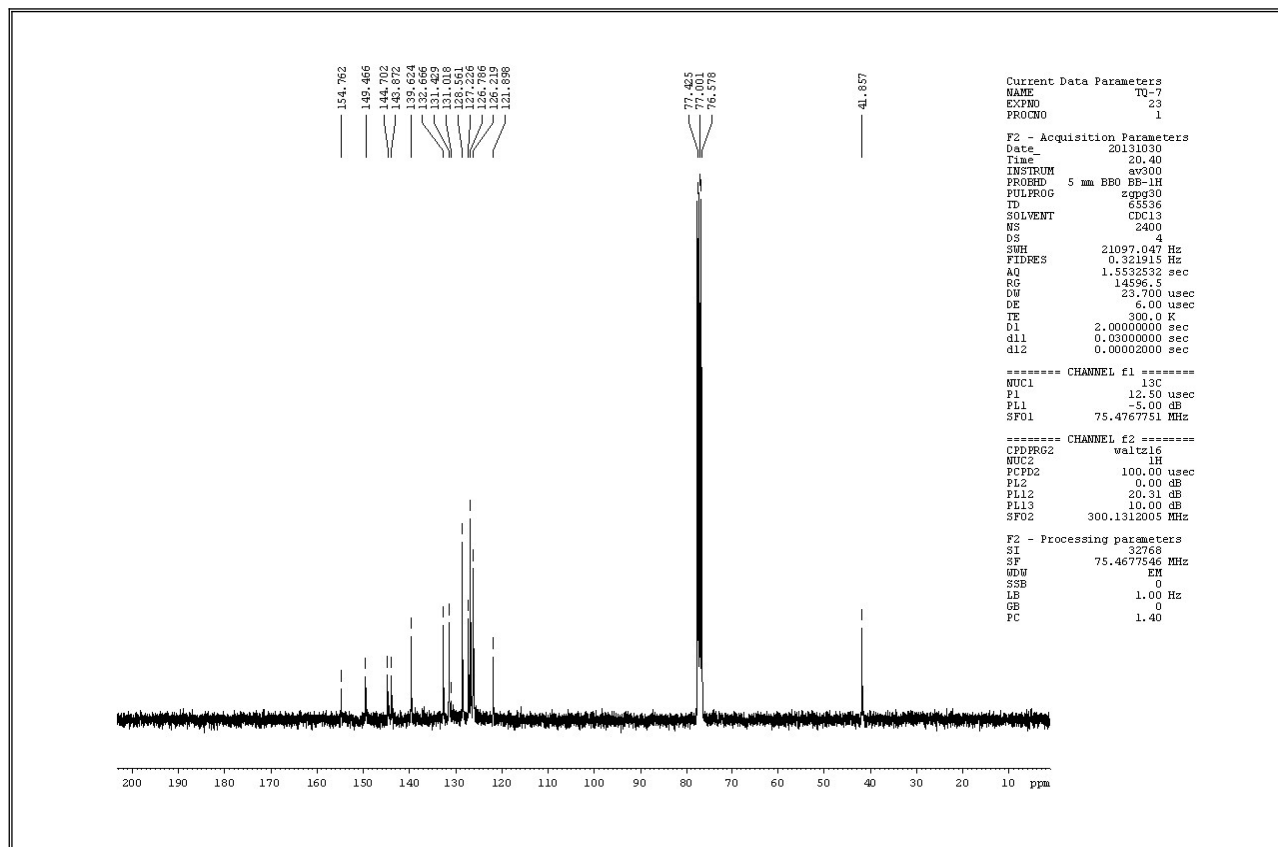


Figure. 11 ^{13}C NMR Spectrum of **3d** (CDCl_3)

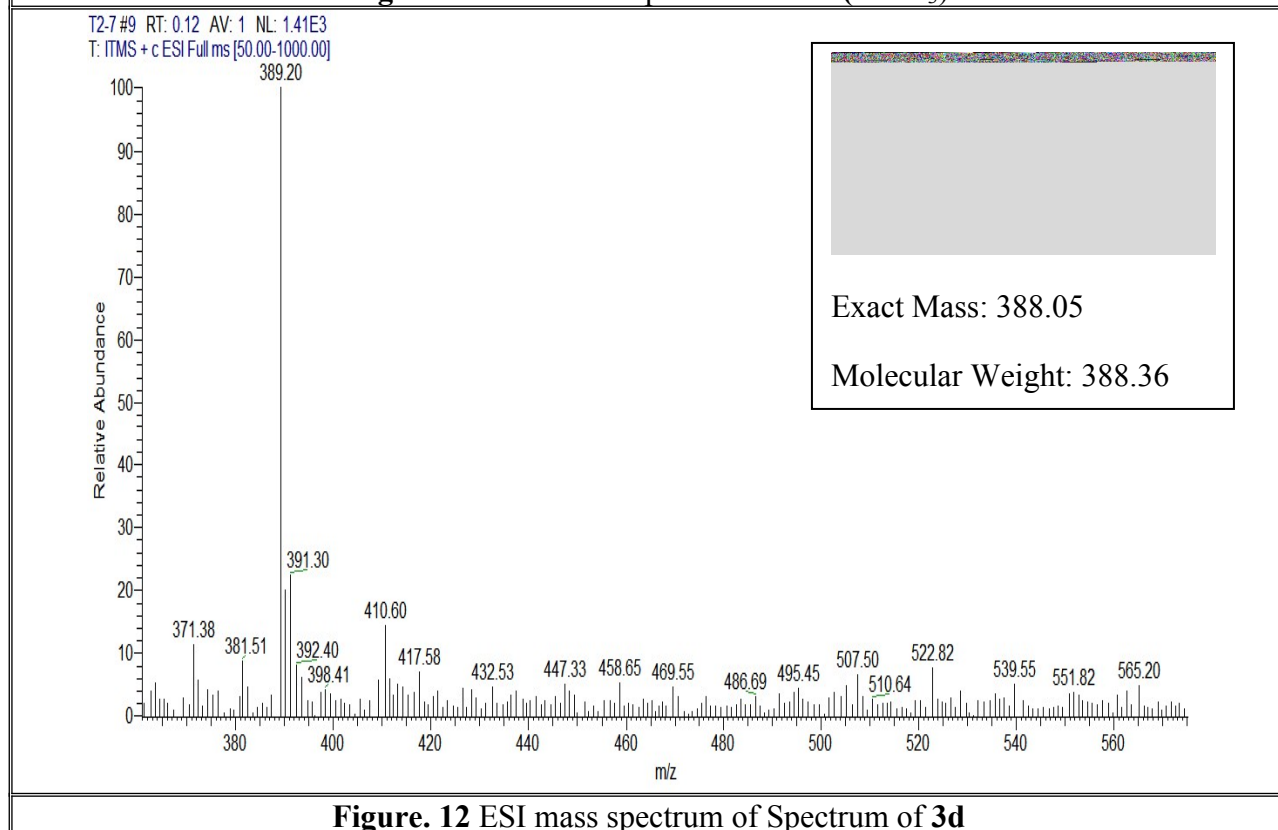


Figure. 12 ESI mass spectrum of Spectrum of **3d**

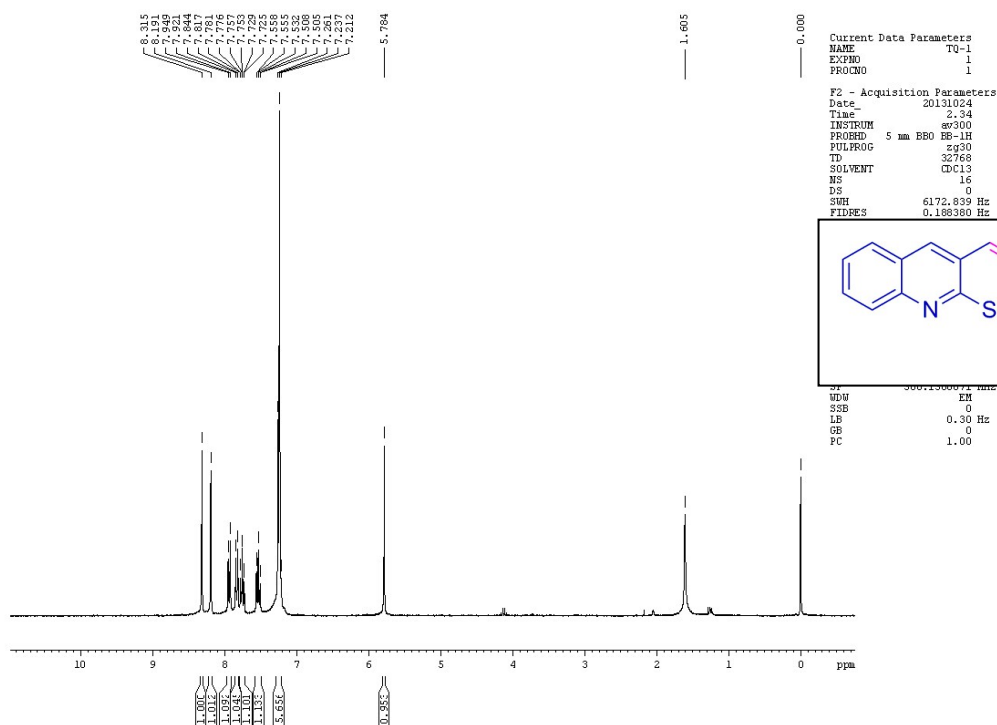


Figure. 13 ^1H NMR Spectrum 3e (CDCl_3)

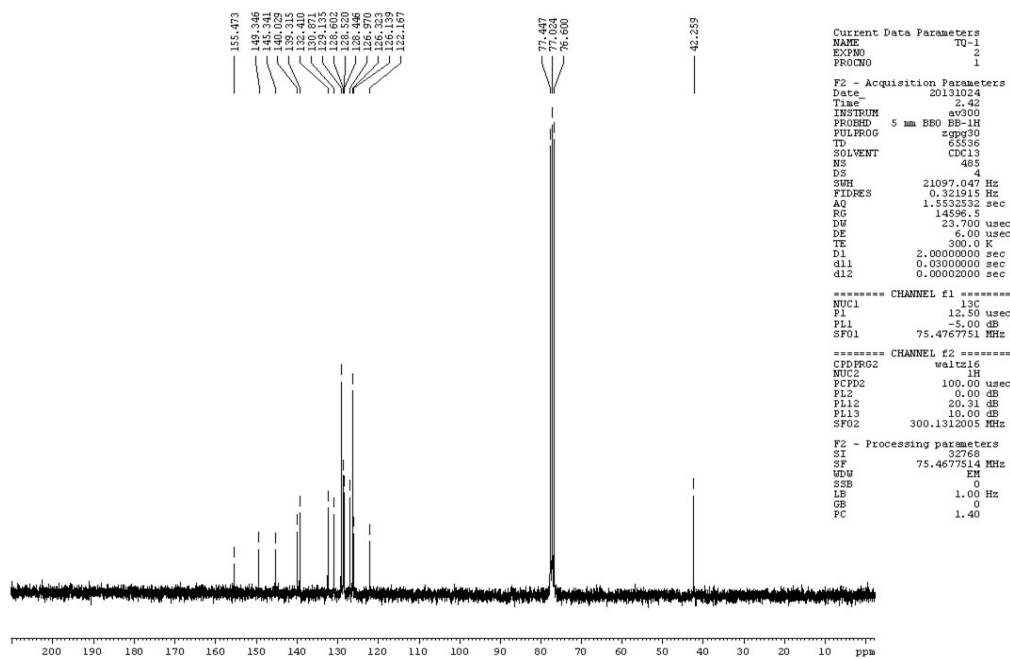


Figure. 14 ^{13}C NMR Spectrum of 3e (CDCl_3)

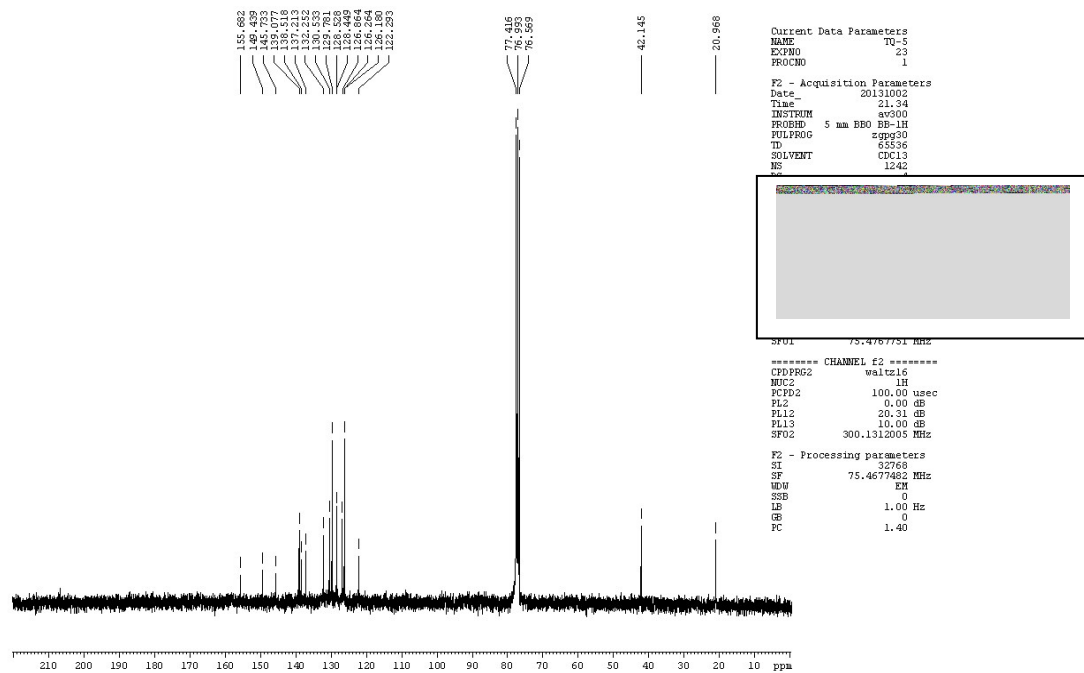


Figure. 17 ^{13}C NMR Spectrum of **3f** (CDCl_3)

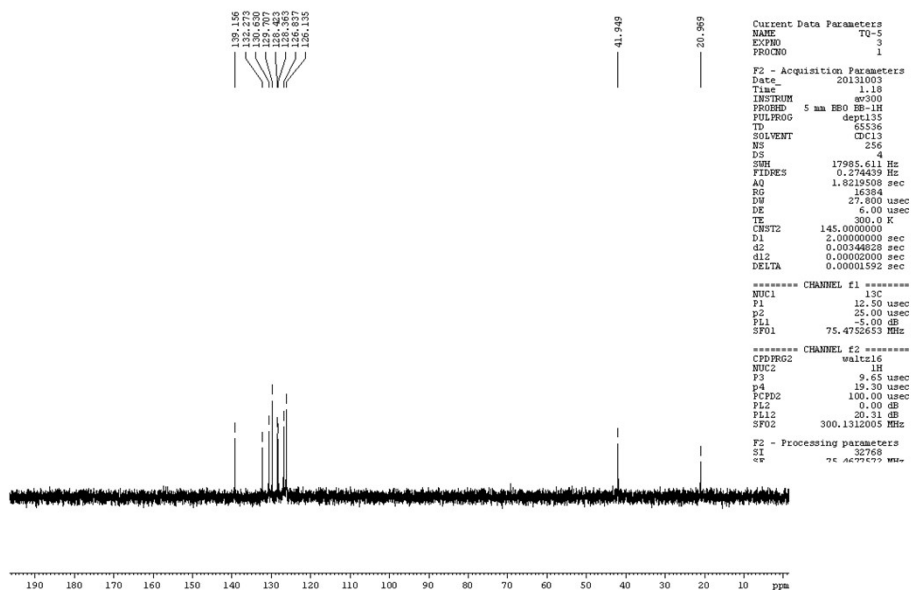


Figure. 18 DEPT-135 Spectrum of **3f** (CDCl_3)

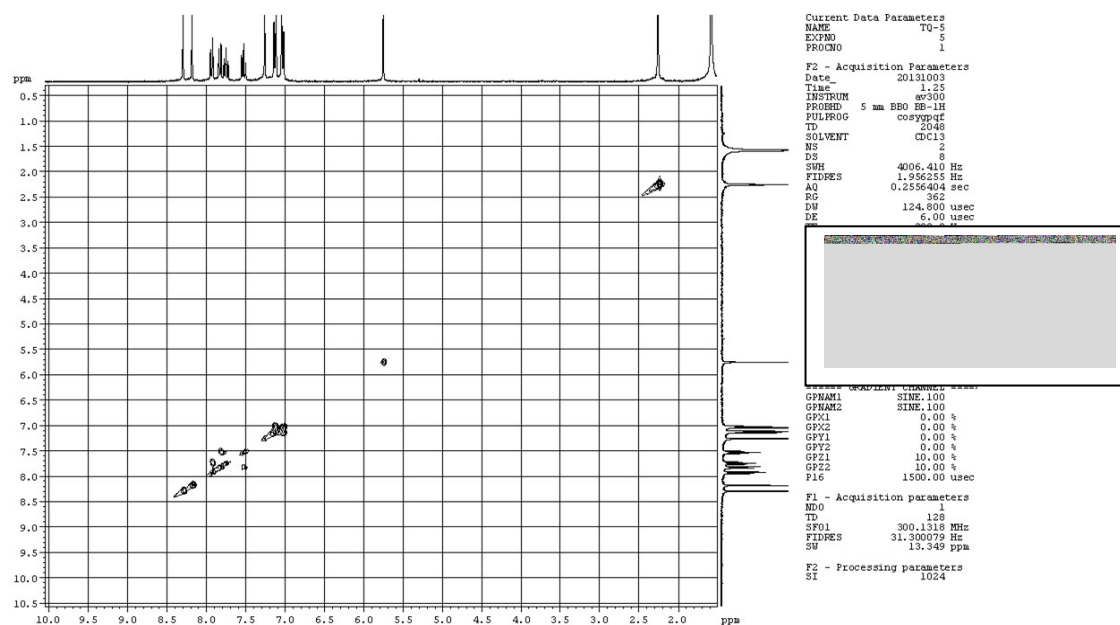


Figure. 19 H,H-COSY Spectrum of **3f** (CDCl₃)

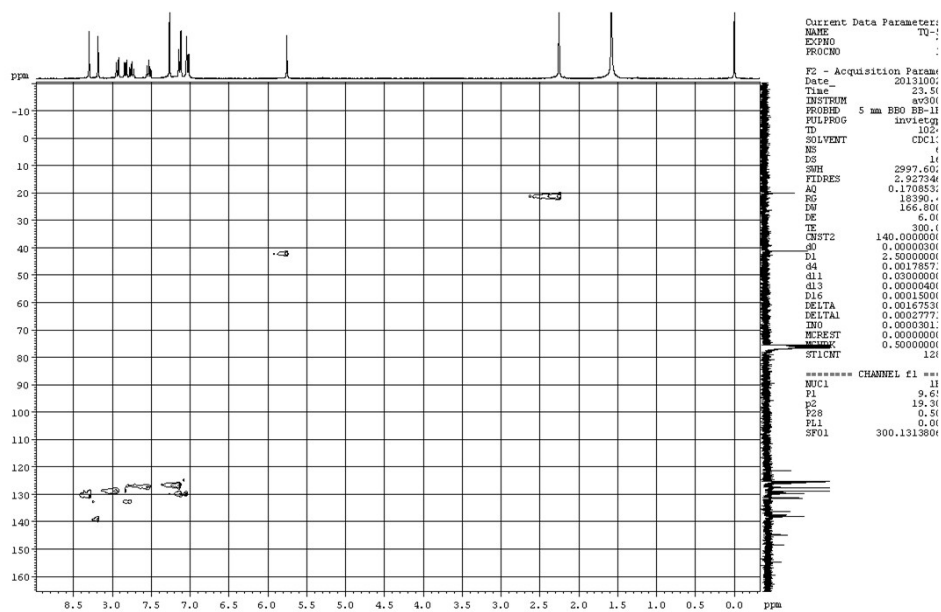


Figure. 20 C,H-COSY Spectrum of **3f** (CDCl₃)

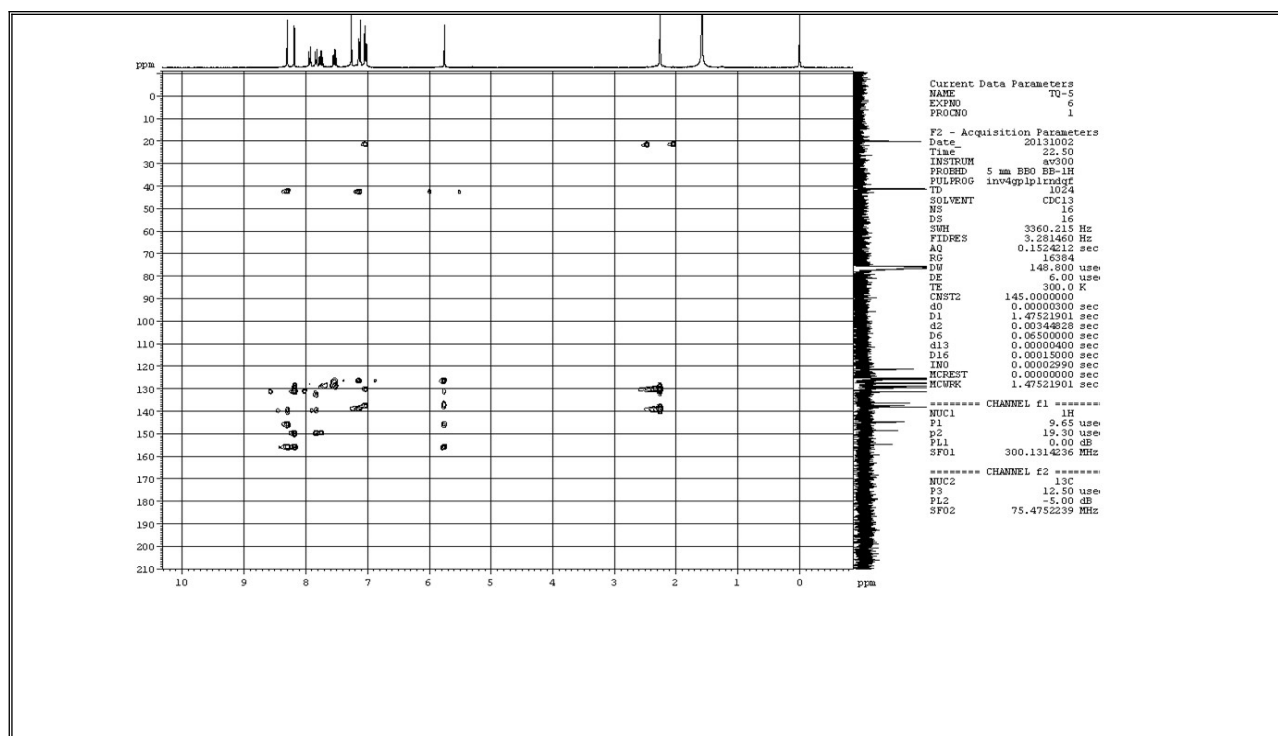


Figure. 21 HMBC Spectrum of **3f** (CDCl₃)

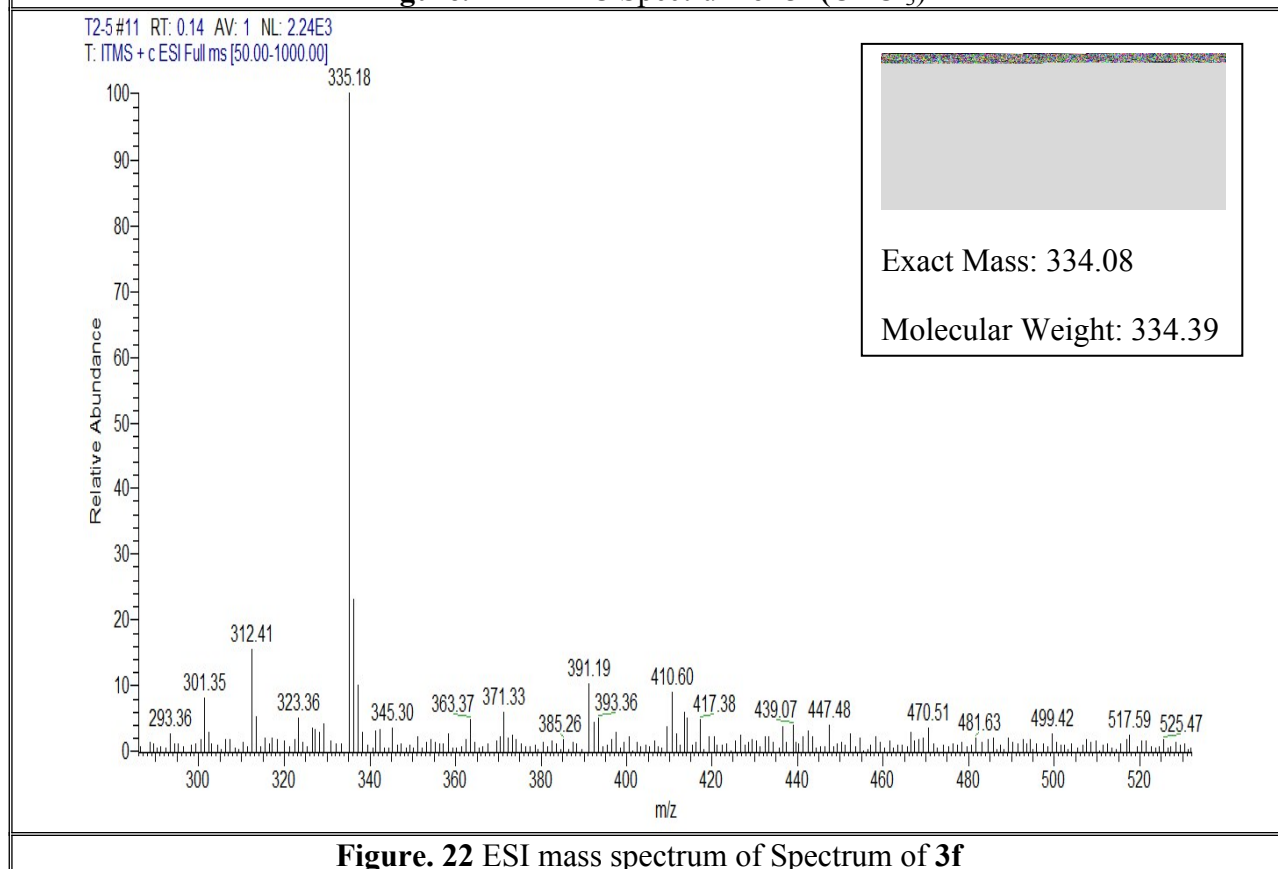


Figure. 22 ESI mass spectrum of Spectrum of **3f**

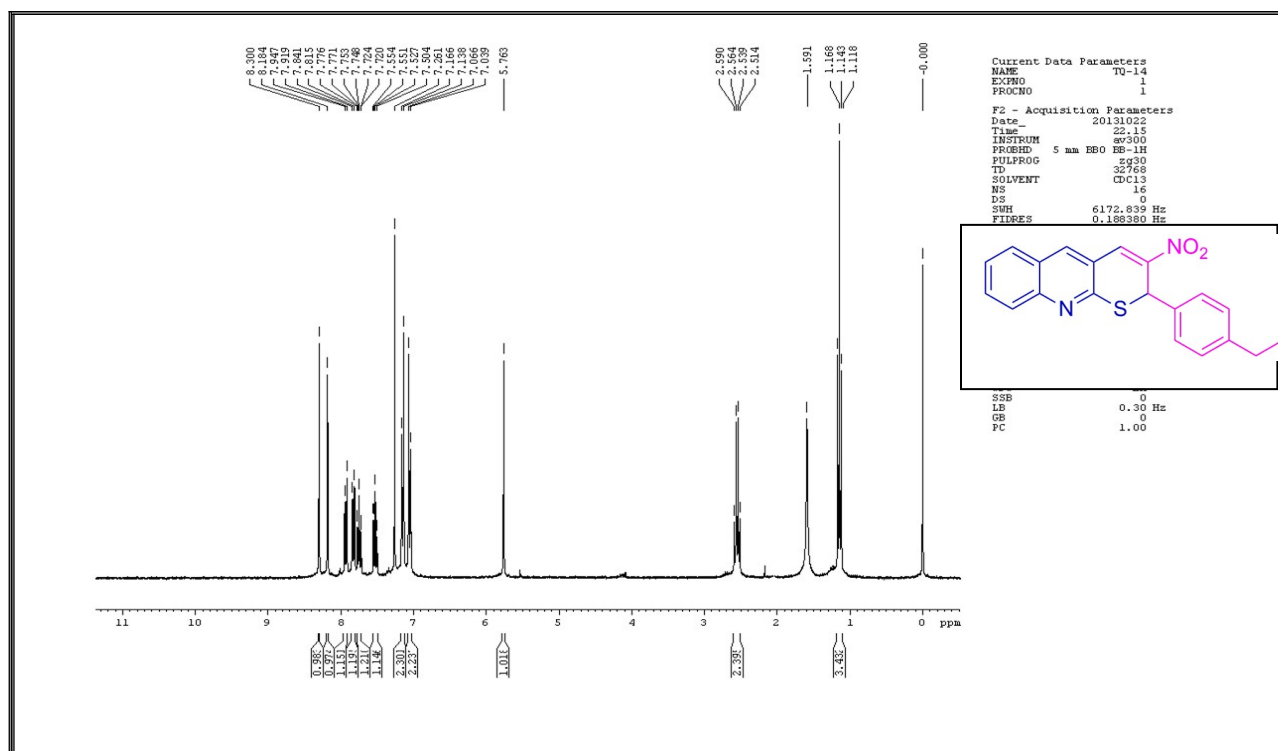


Figure. 23 ^1H NMR Spectrum **3g** (CDCl_3)

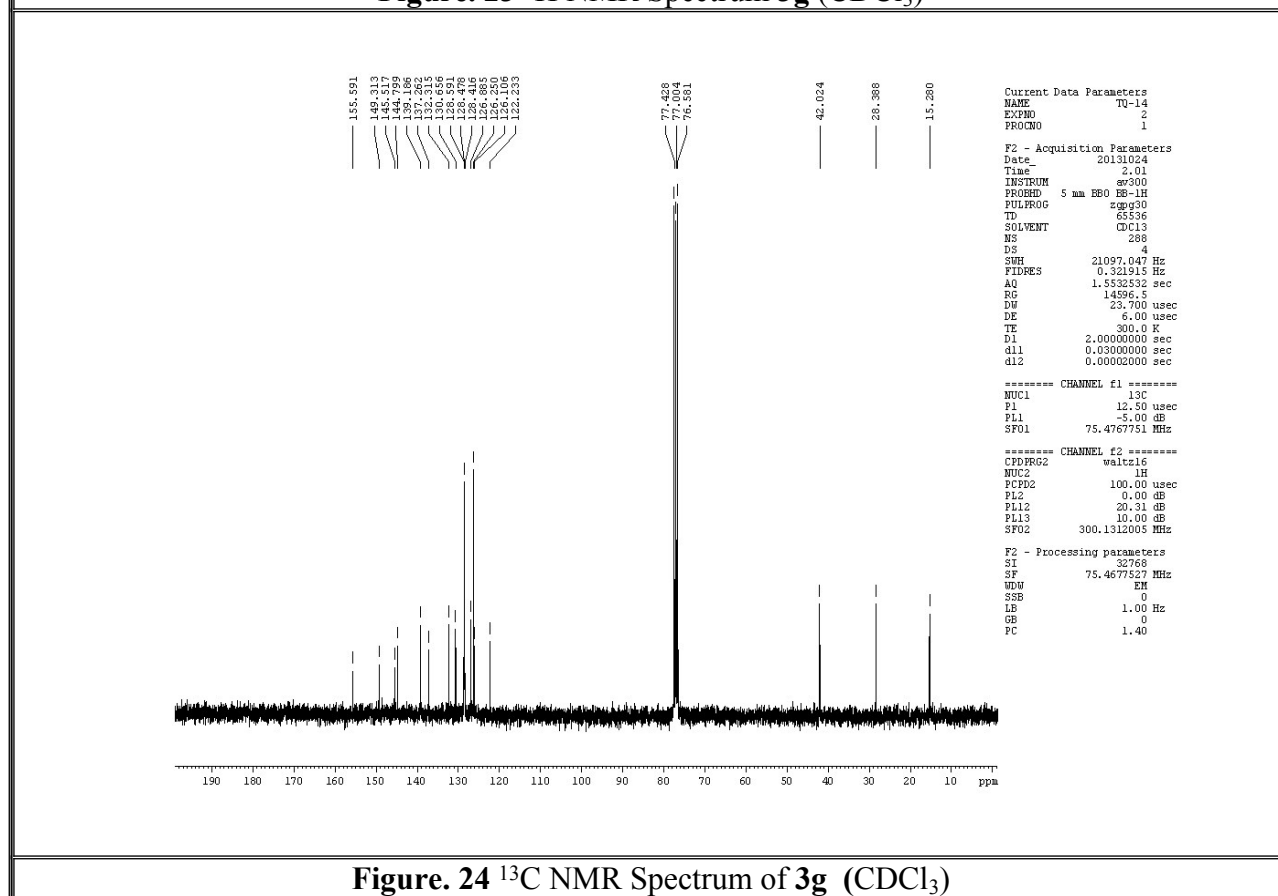


Figure. 24 ^{13}C NMR Spectrum of **3g** (CDCl_3)

T2-14 #11 RT: 0.15 AV: 1 NL: 1.51E3
T: ITMS + c ESI Full ms [50.00-1000.00]

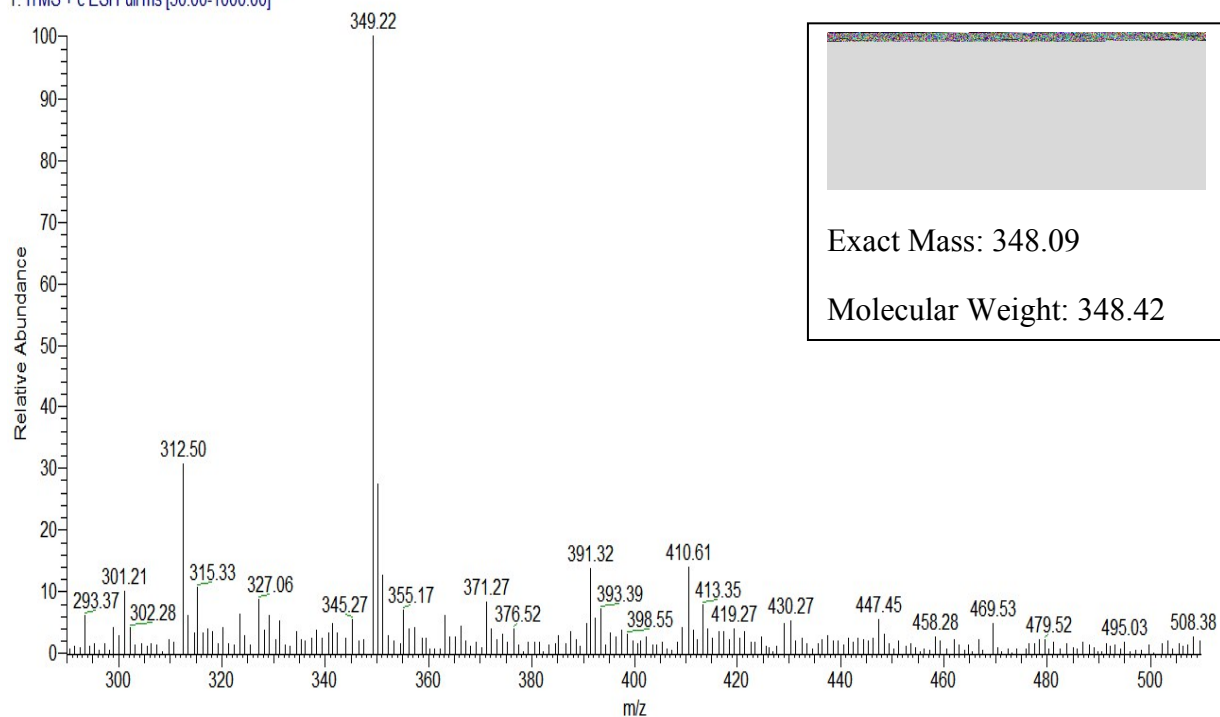


Figure. 25 ESI mass spectrum of Spectrum of **3g**

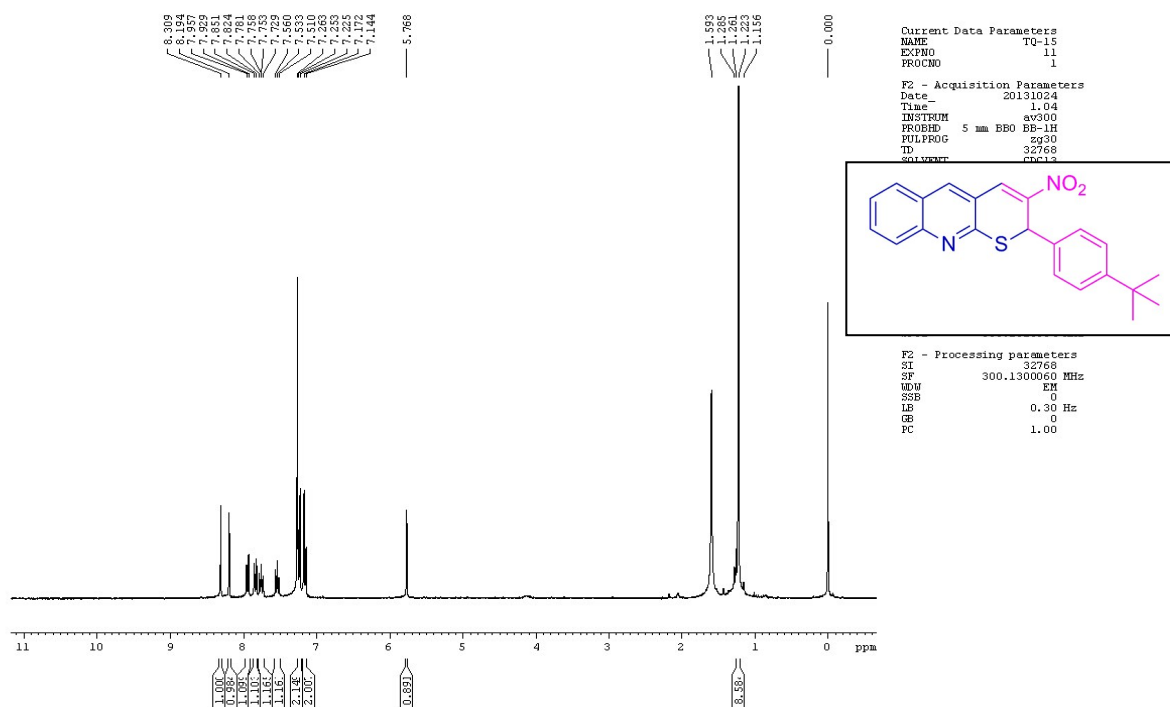


Figure. 26 ^1H NMR Spectrum **3h** (CDCl_3)

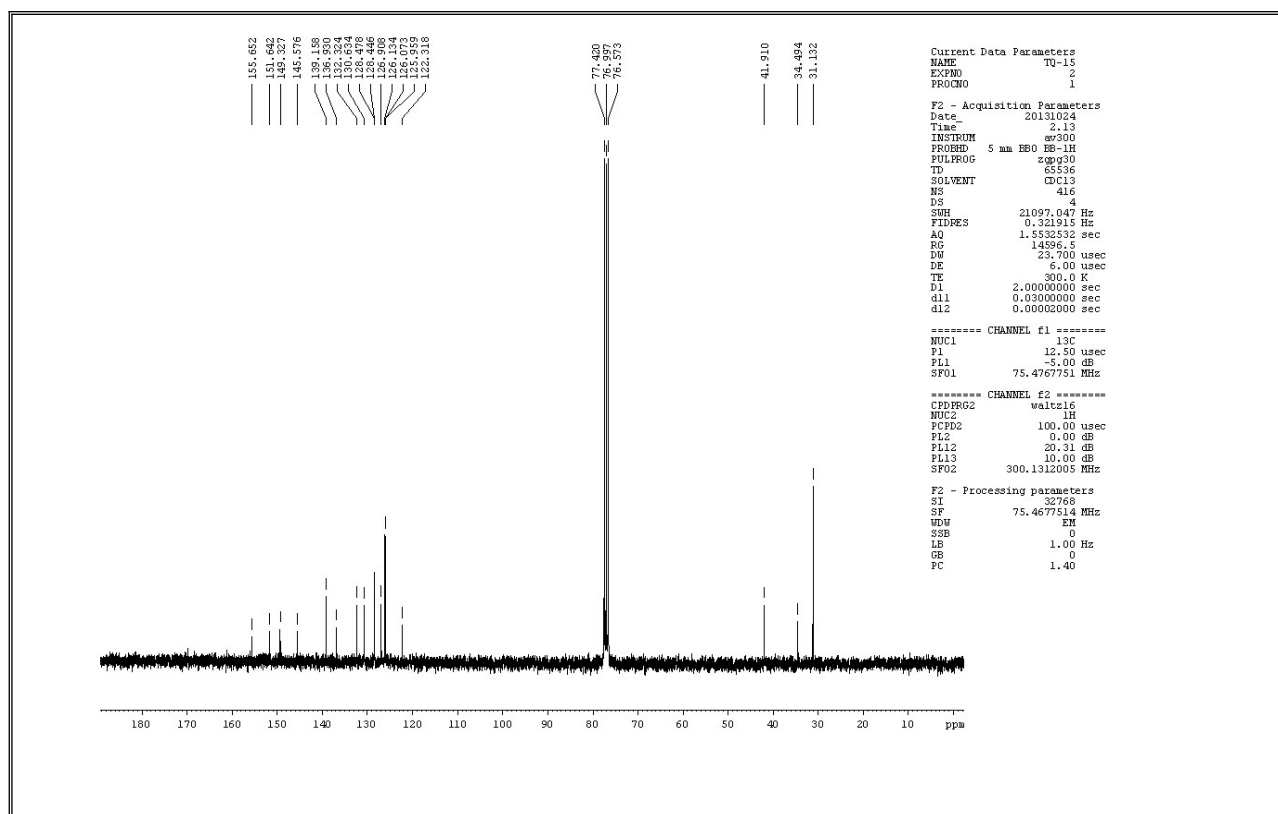


Figure. 27 ^{13}C NMR Spectrum of **3h** (CDCl_3)

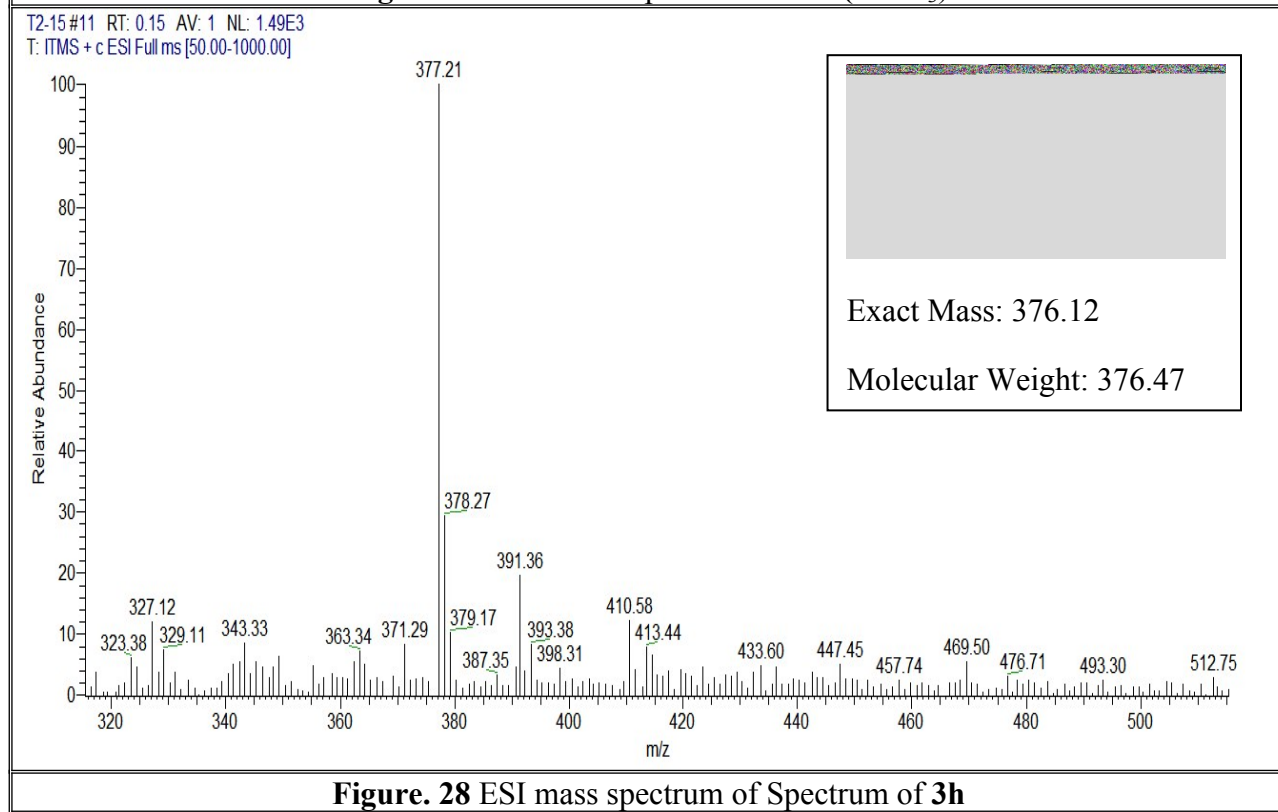


Figure. 28 ESI mass spectrum of Spectrum of **3h**

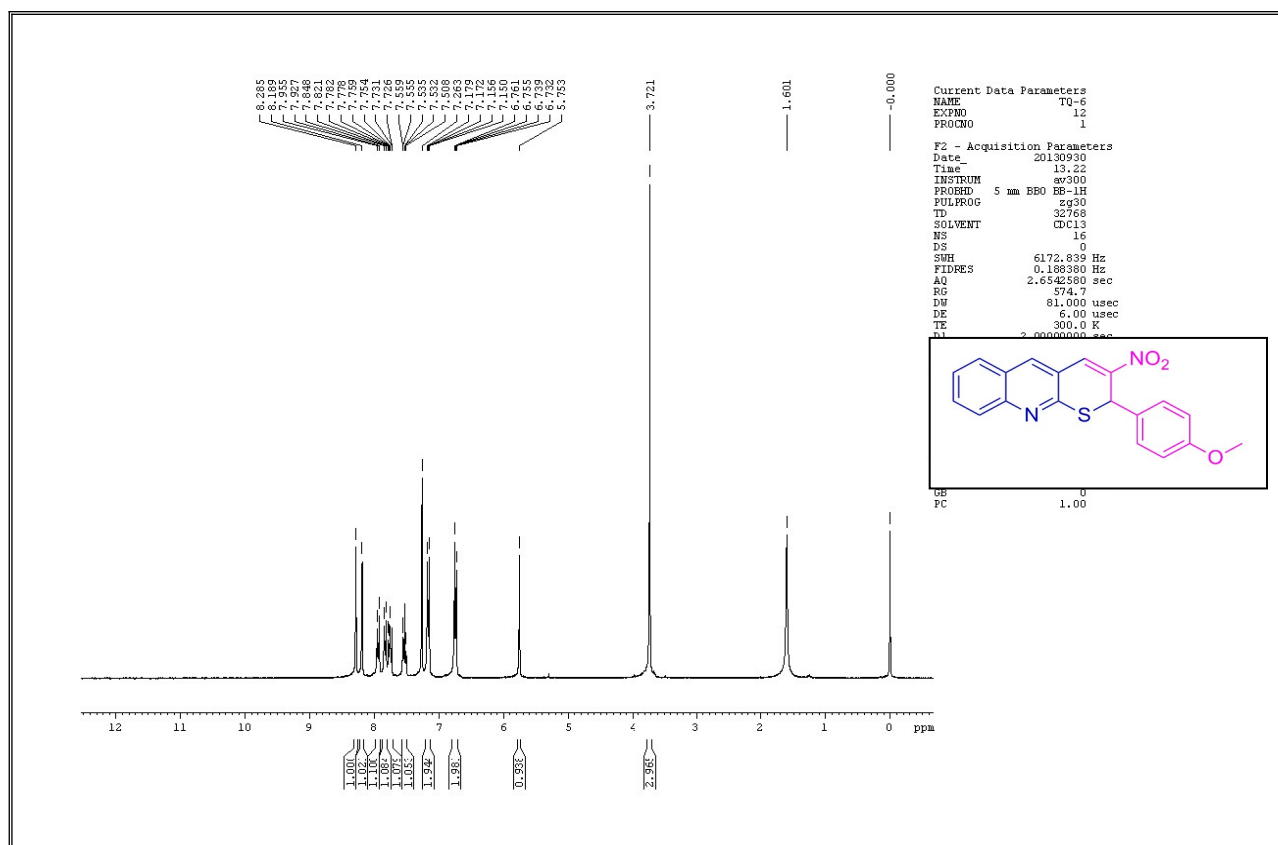


Figure. 29 ^1H NMR Spectrum 3i (CDCl_3)

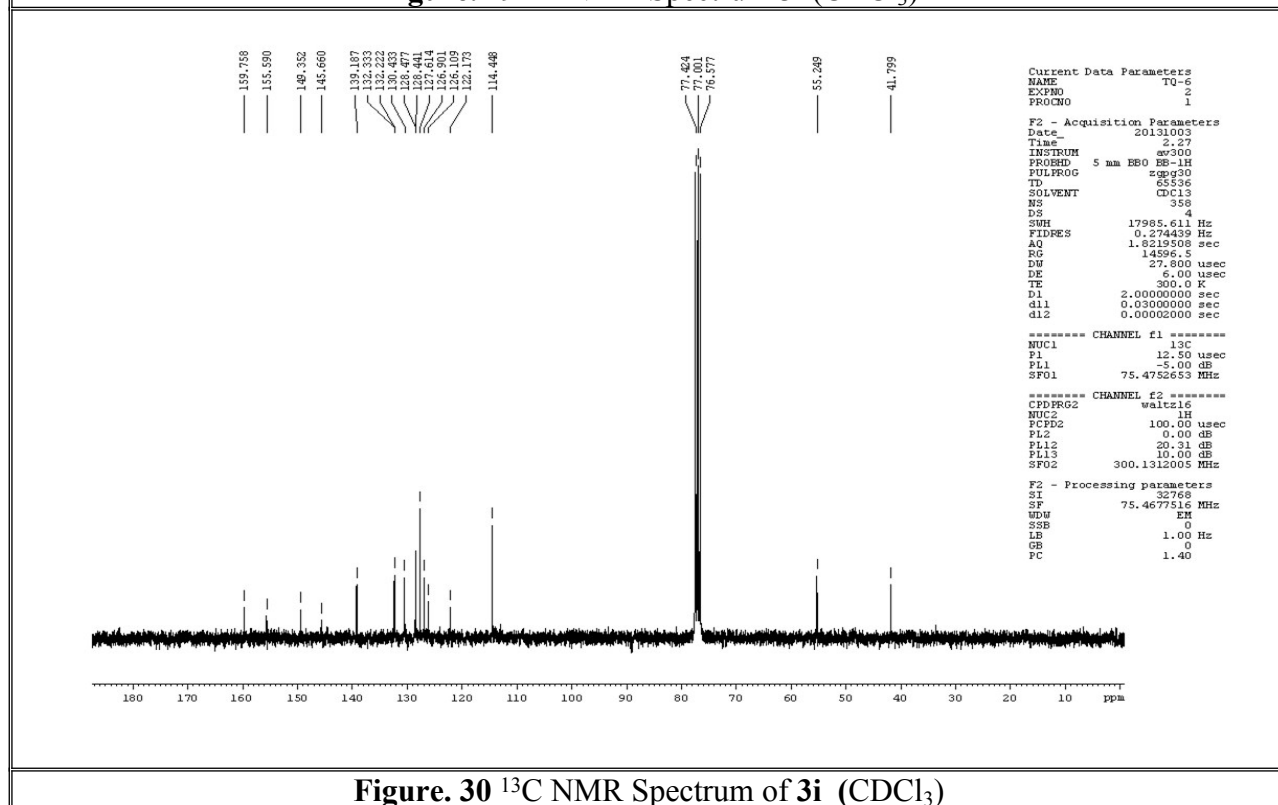


Figure. 30 ^{13}C NMR Spectrum of 3i (CDCl_3)

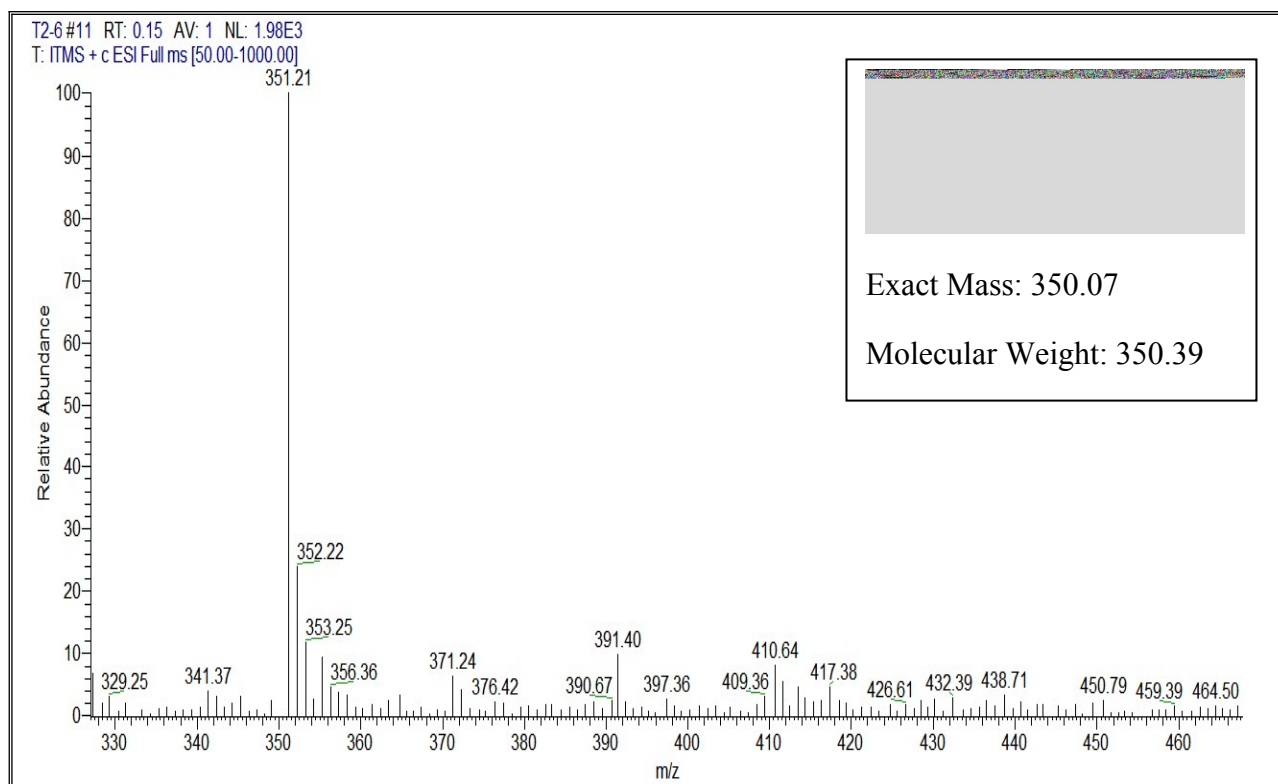
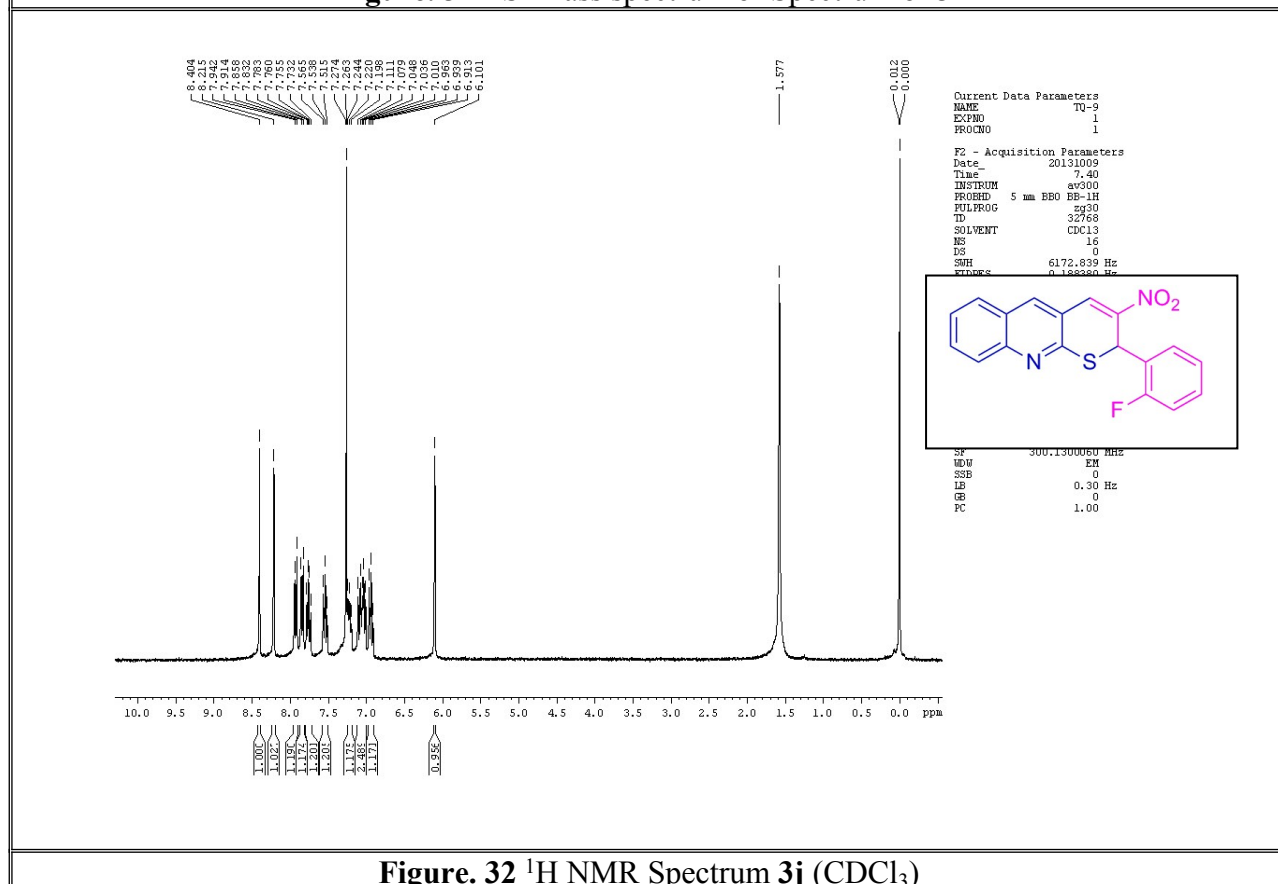


Figure. 31 ESI mass spectrum of Spectrum of **3i**



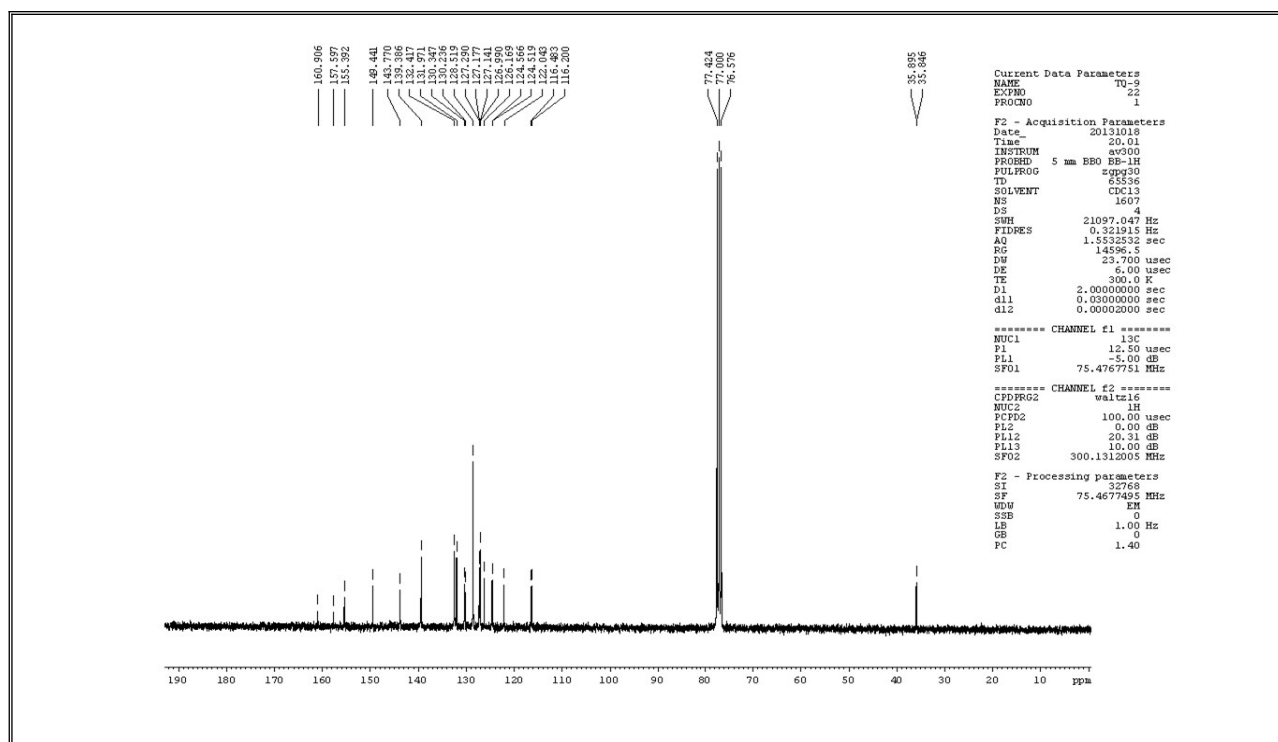


Figure. 33 ^{13}C NMR Spectrum of **3j** (CDCl_3)

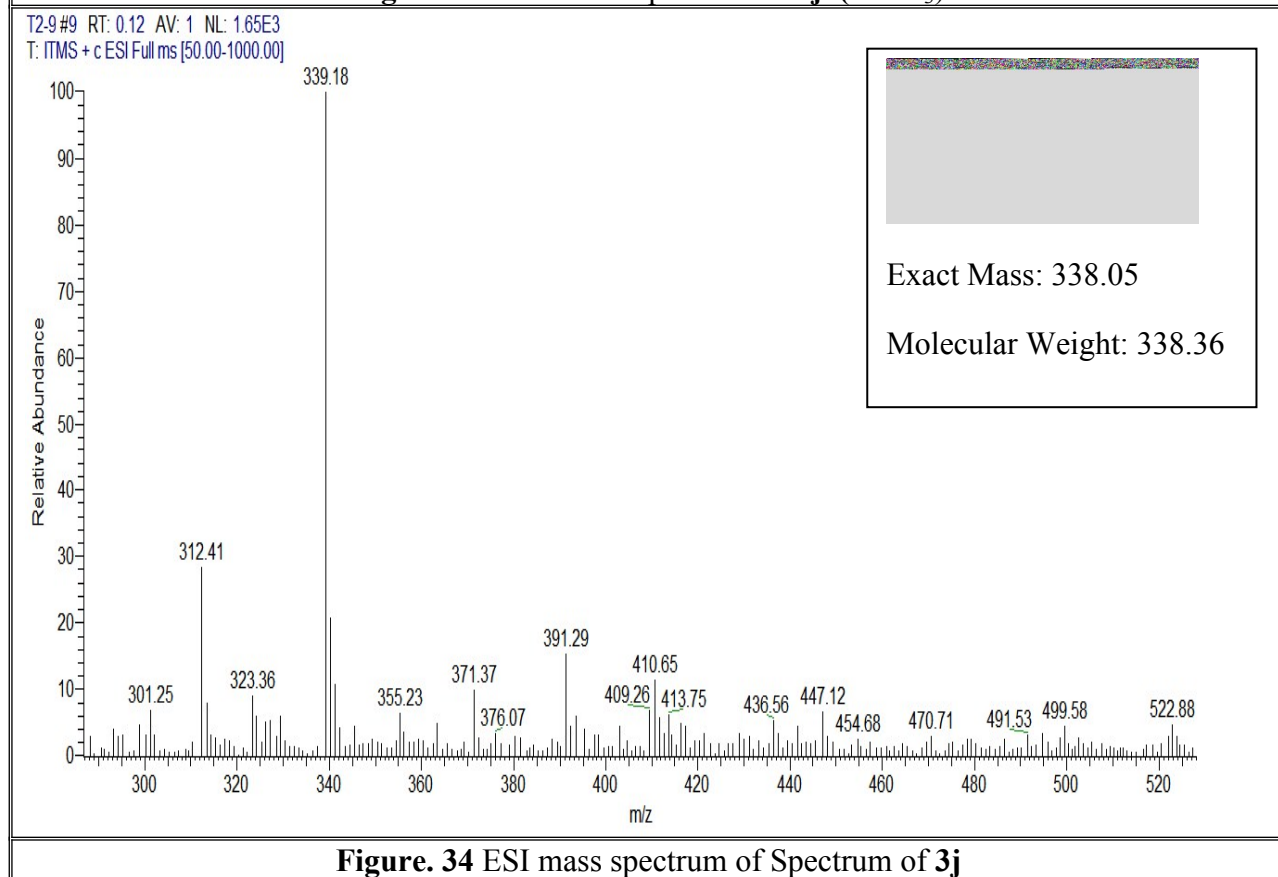


Figure. 34 ESI mass spectrum of Spectrum of **3j**

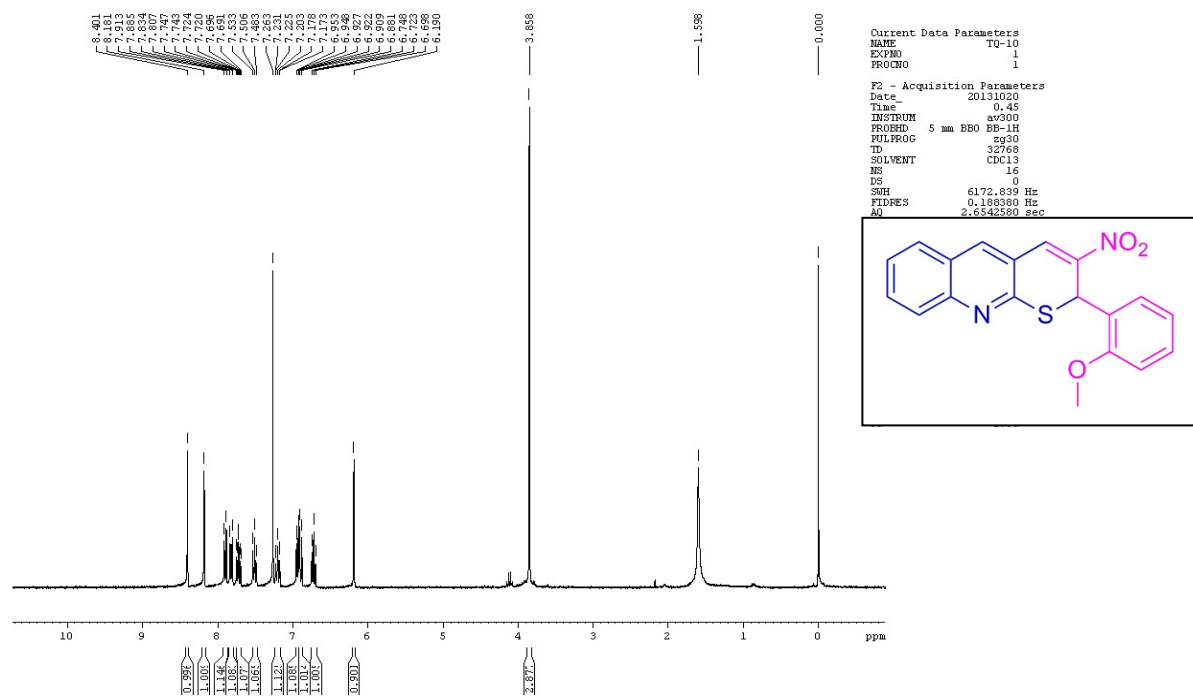


Figure. 35 ^1H NMR Spectrum **3k** (CDCl_3)

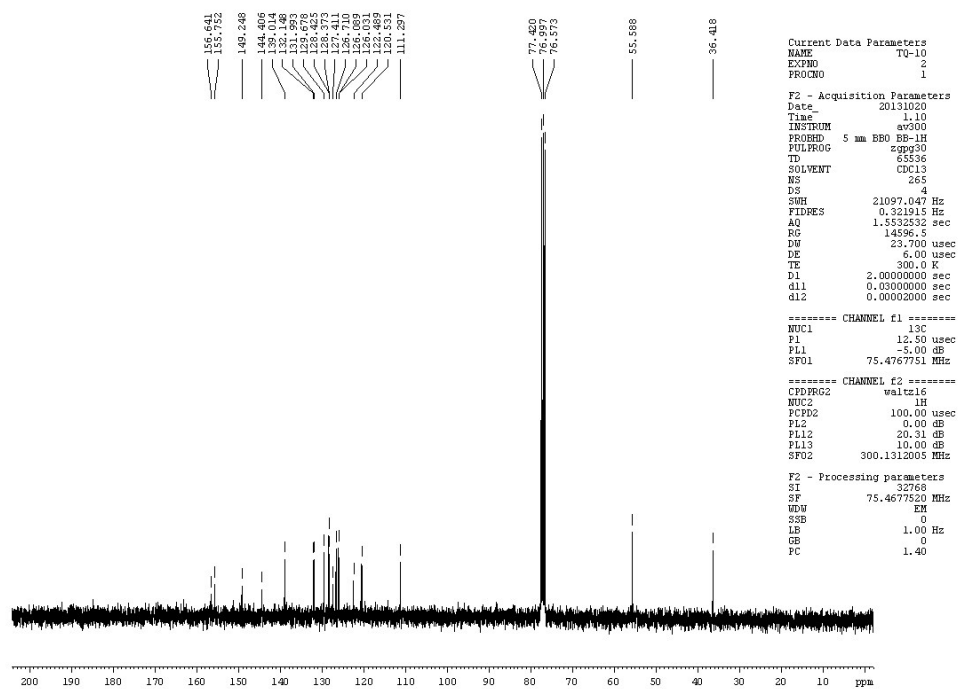


Figure. 36 ^{13}C NMR Spectrum of **3k** (CDCl_3)

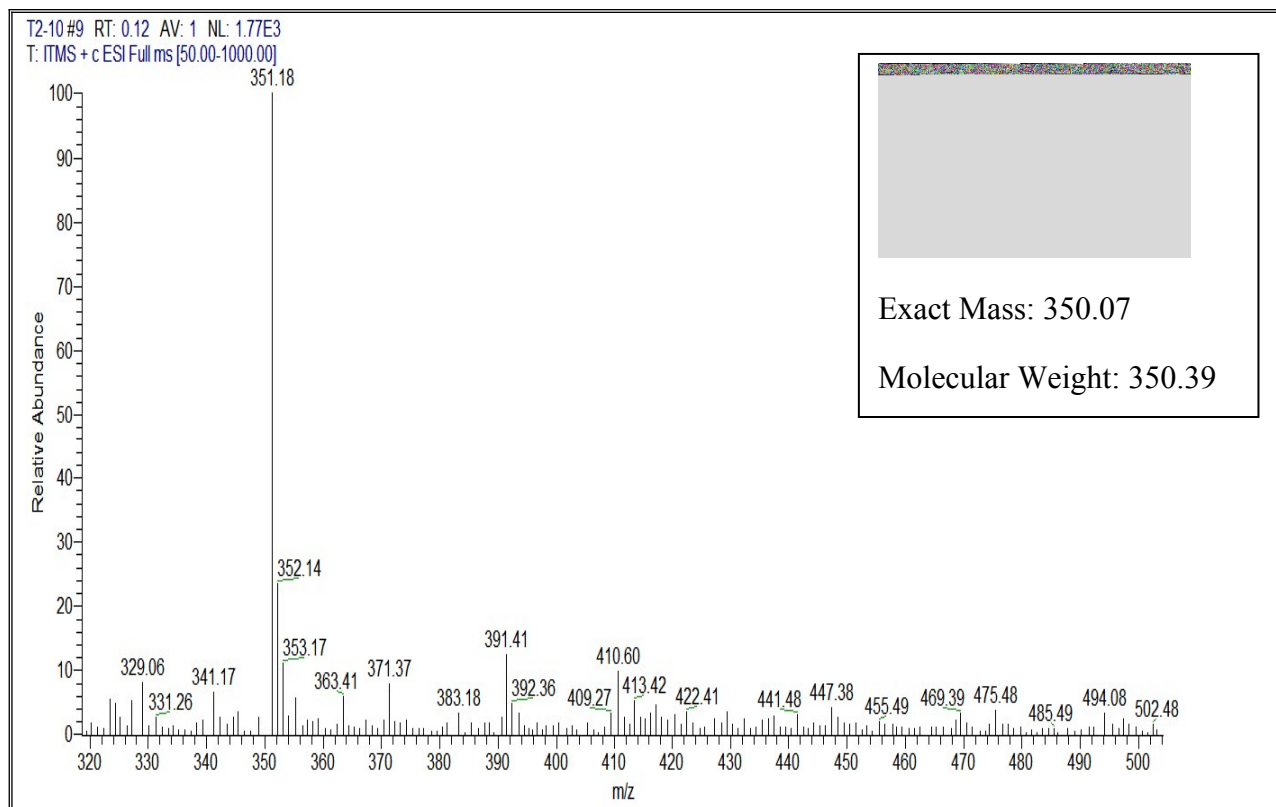


Figure. 37 ESI mass spectrum of Spectrum of **3k**

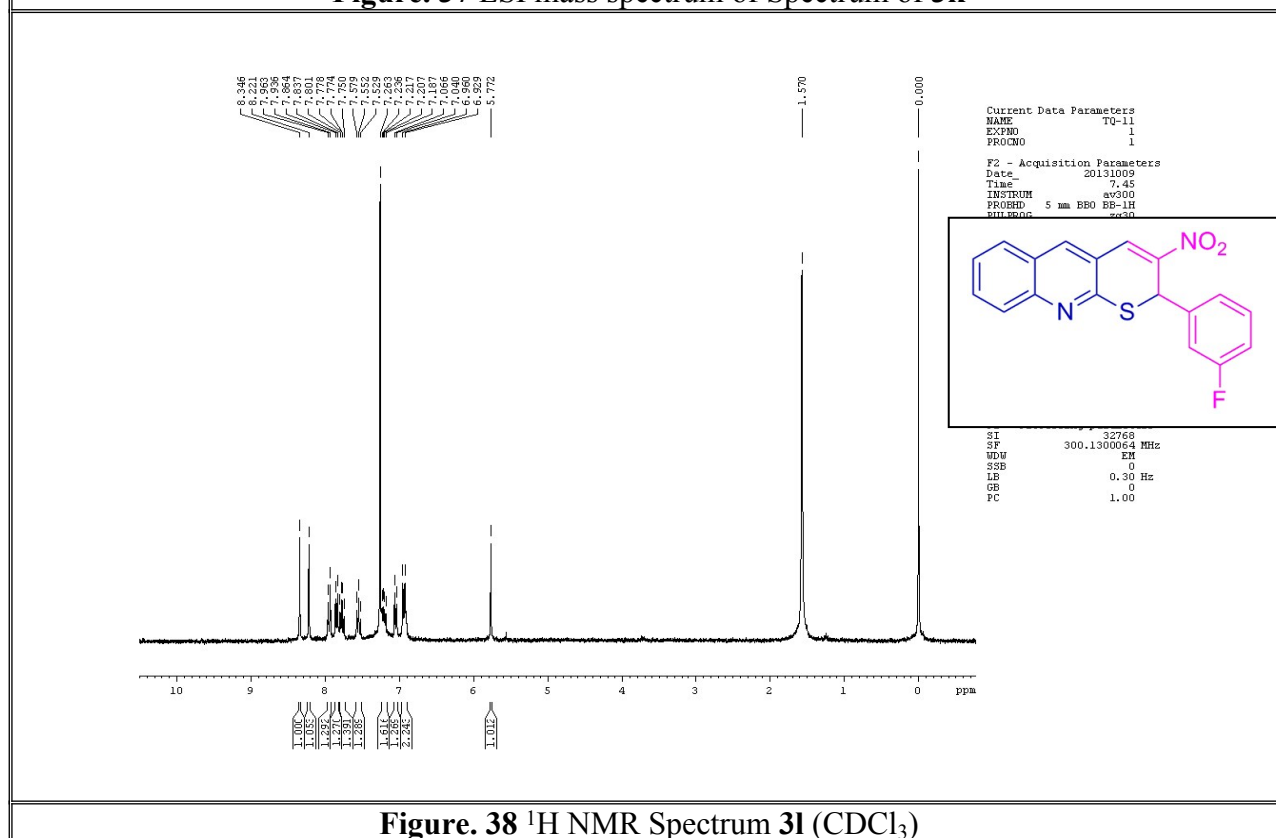


Figure. 38 ^1H NMR Spectrum **3l** (CDCl_3)

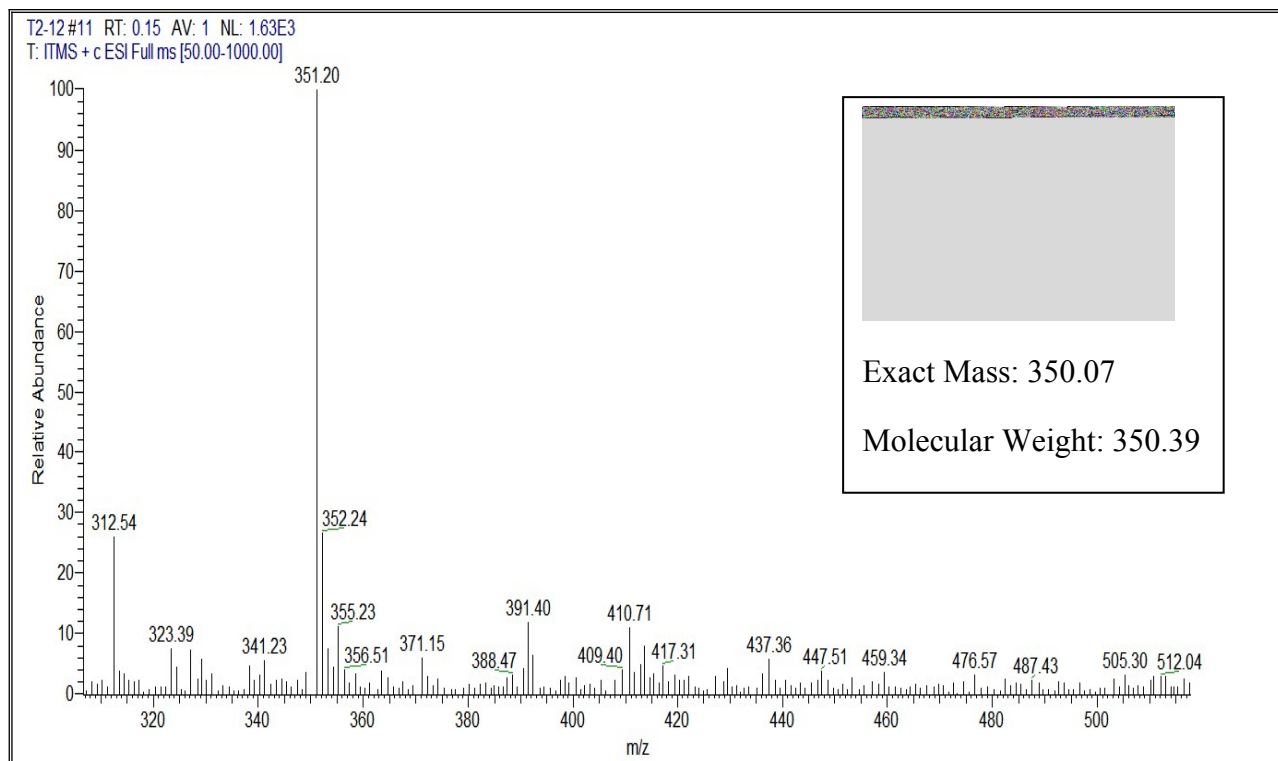


Figure. 43 ESI mass spectrum of Spectrum of **3m**

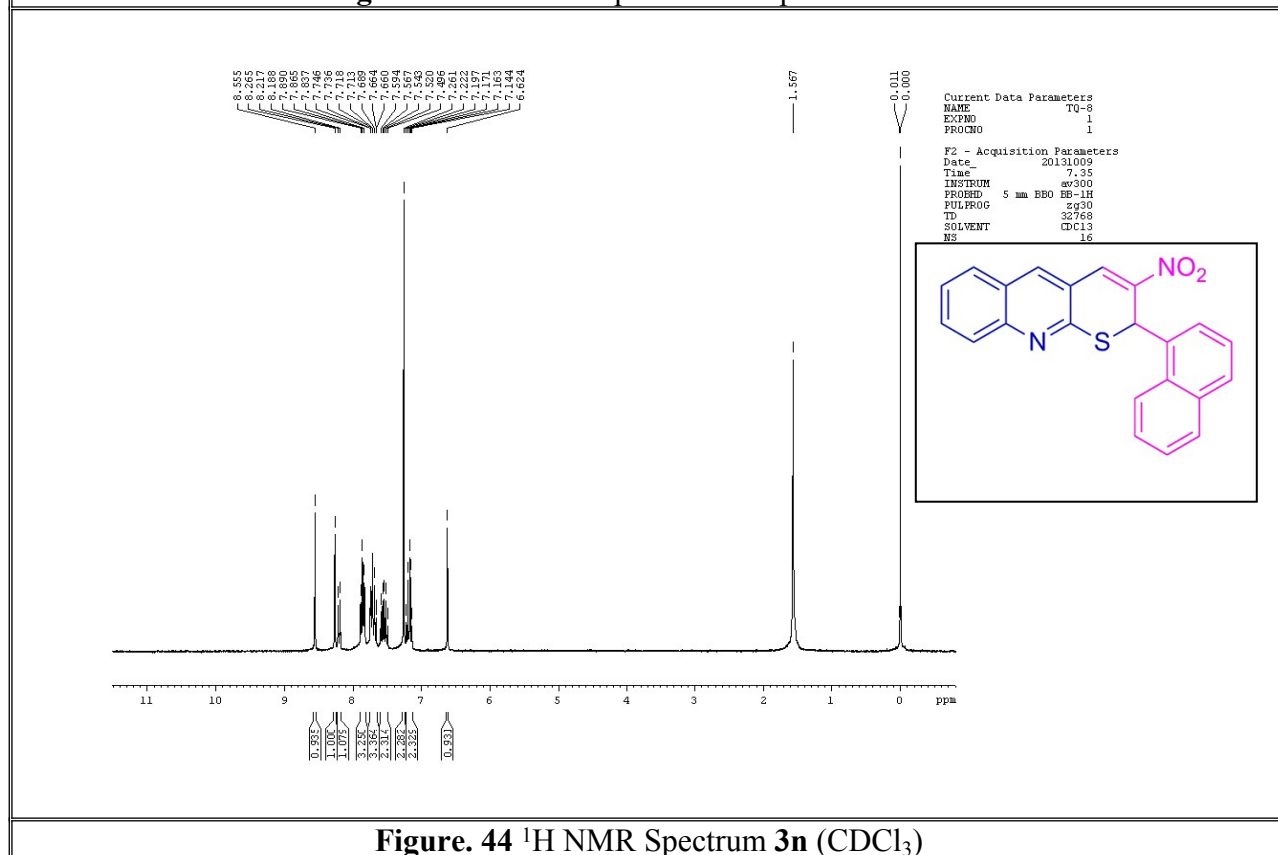


Figure. 44 ¹H NMR Spectrum **3n** (CDCl₃)

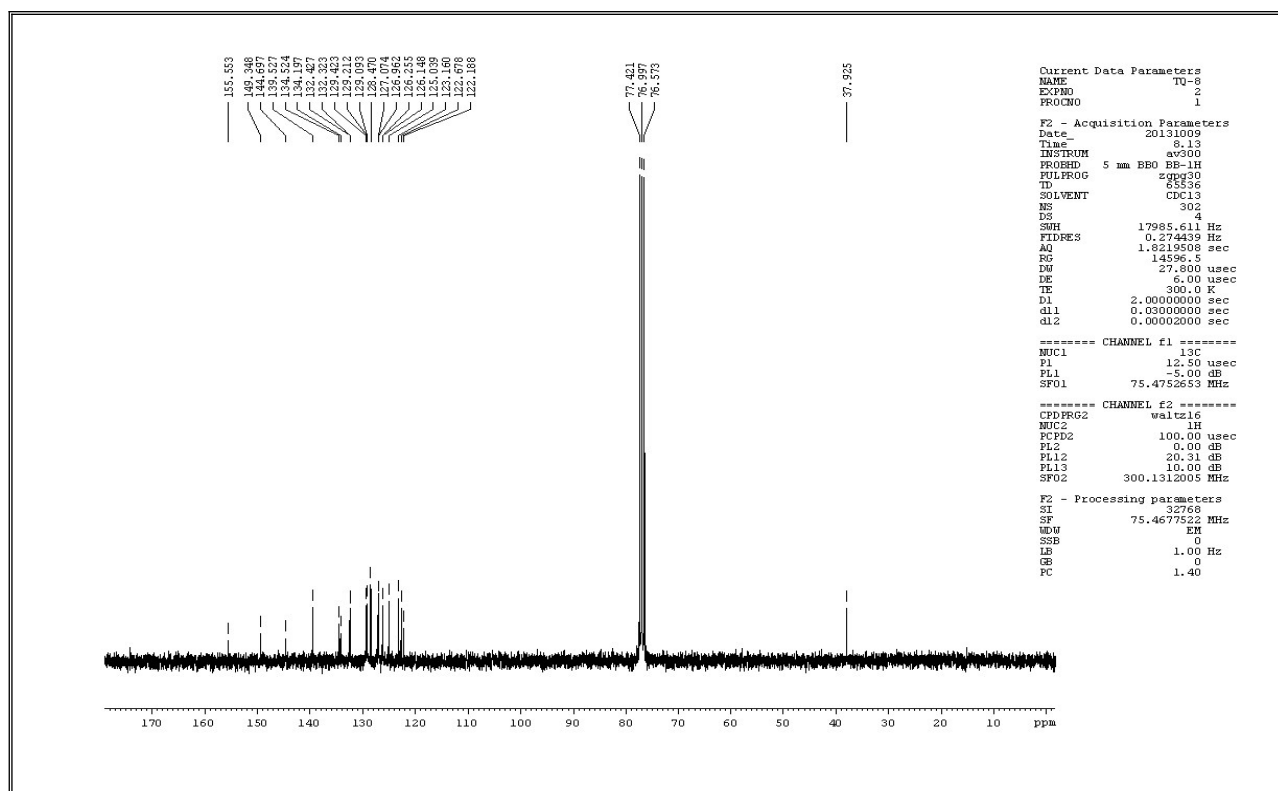


Figure. 45 ^{13}C NMR Spectrum of **3n** (CDCl_3)

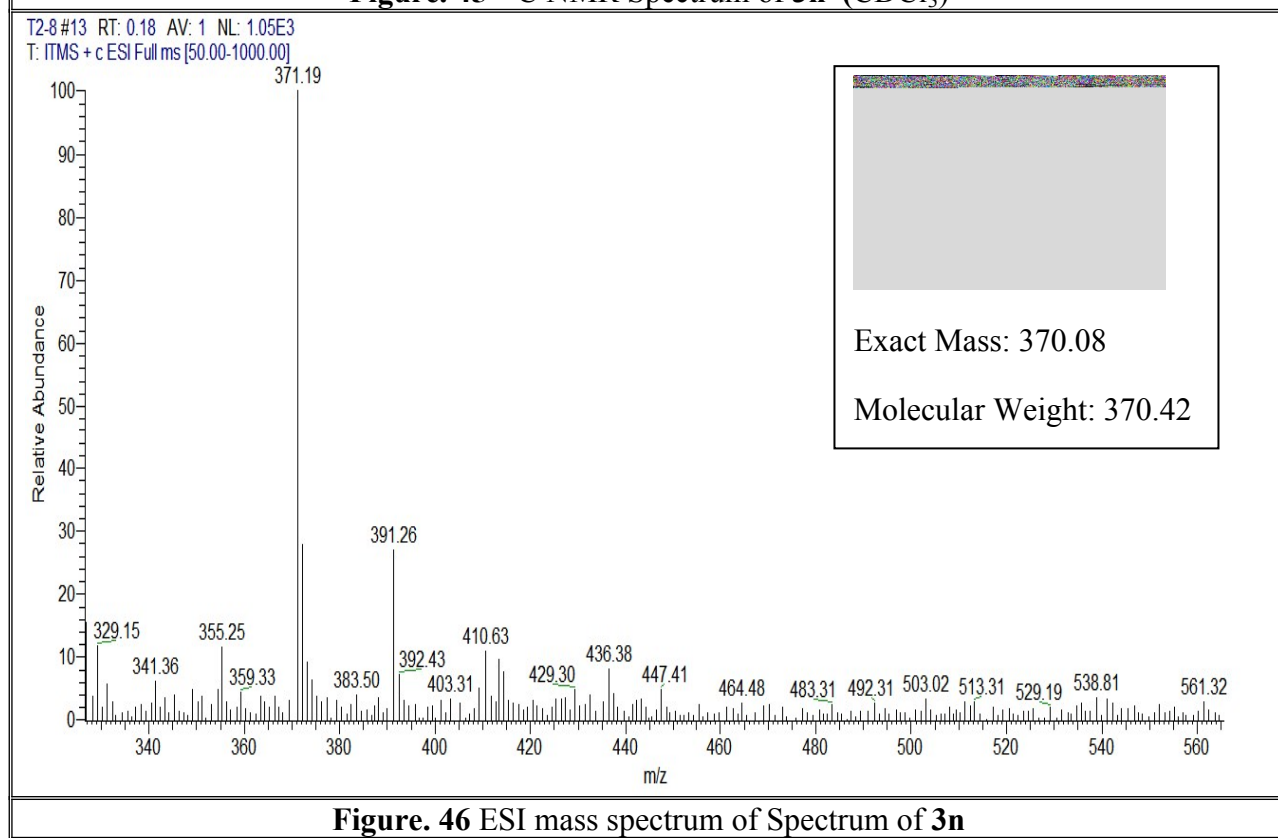


Figure. 46 ESI mass spectrum of Spectrum of **3n**

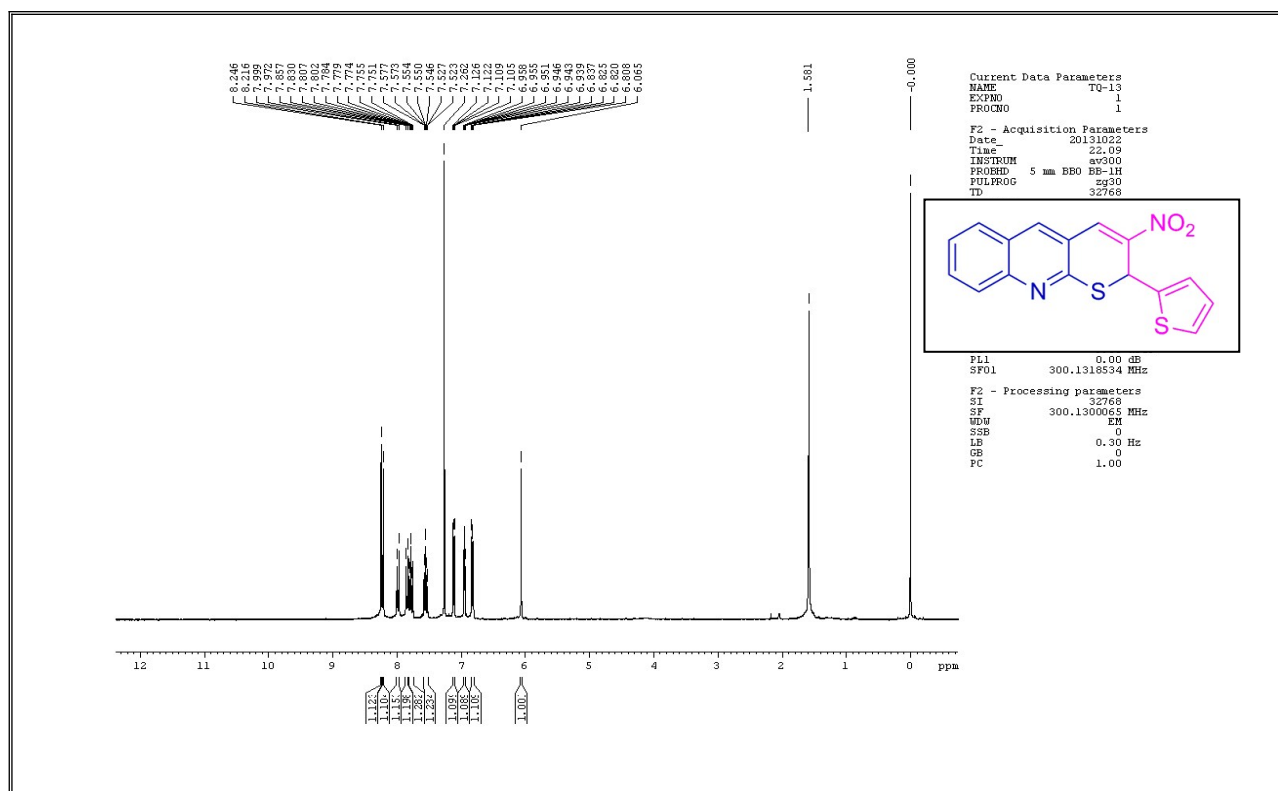


Figure. 47 ^1H NMR Spectrum **3o** (CDCl_3)

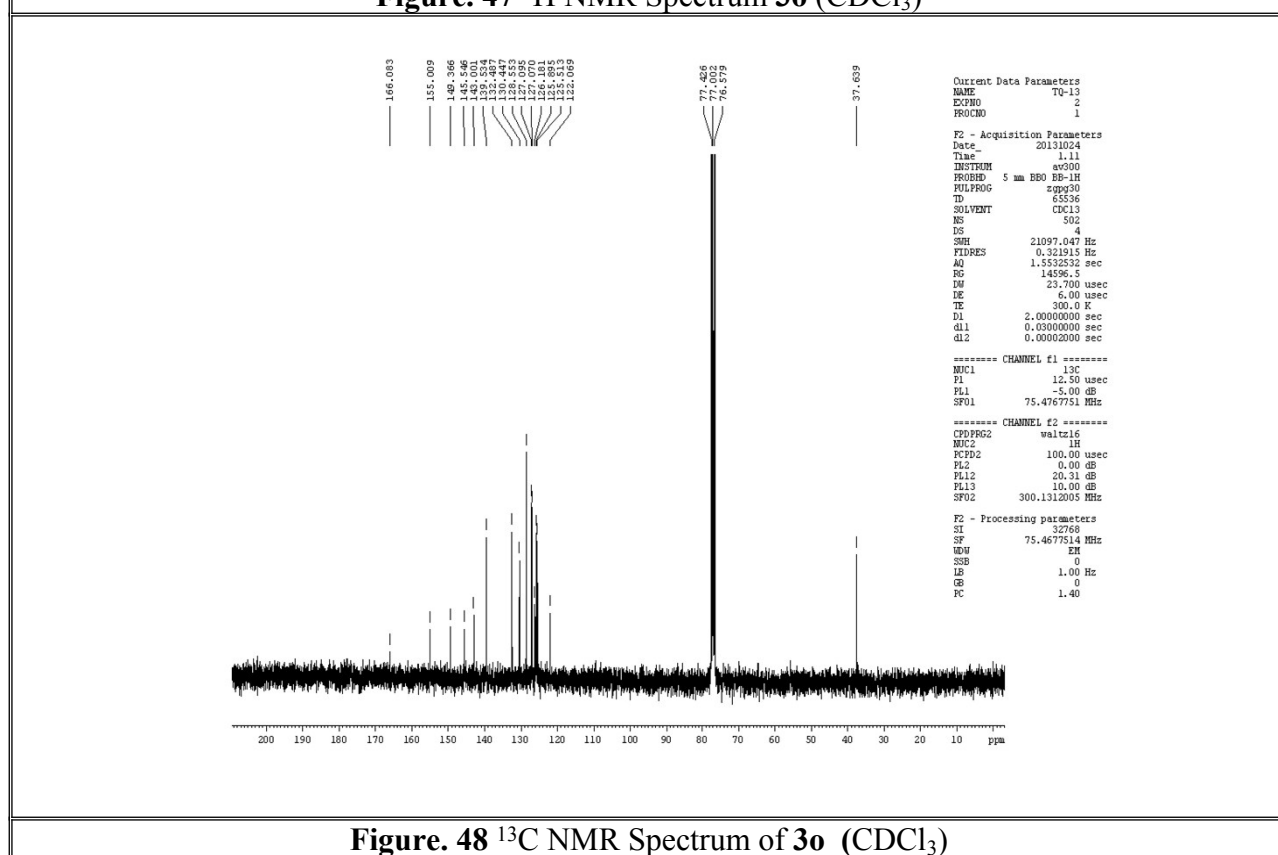


Figure. 48 ^{13}C NMR Spectrum of **3o** (CDCl_3)

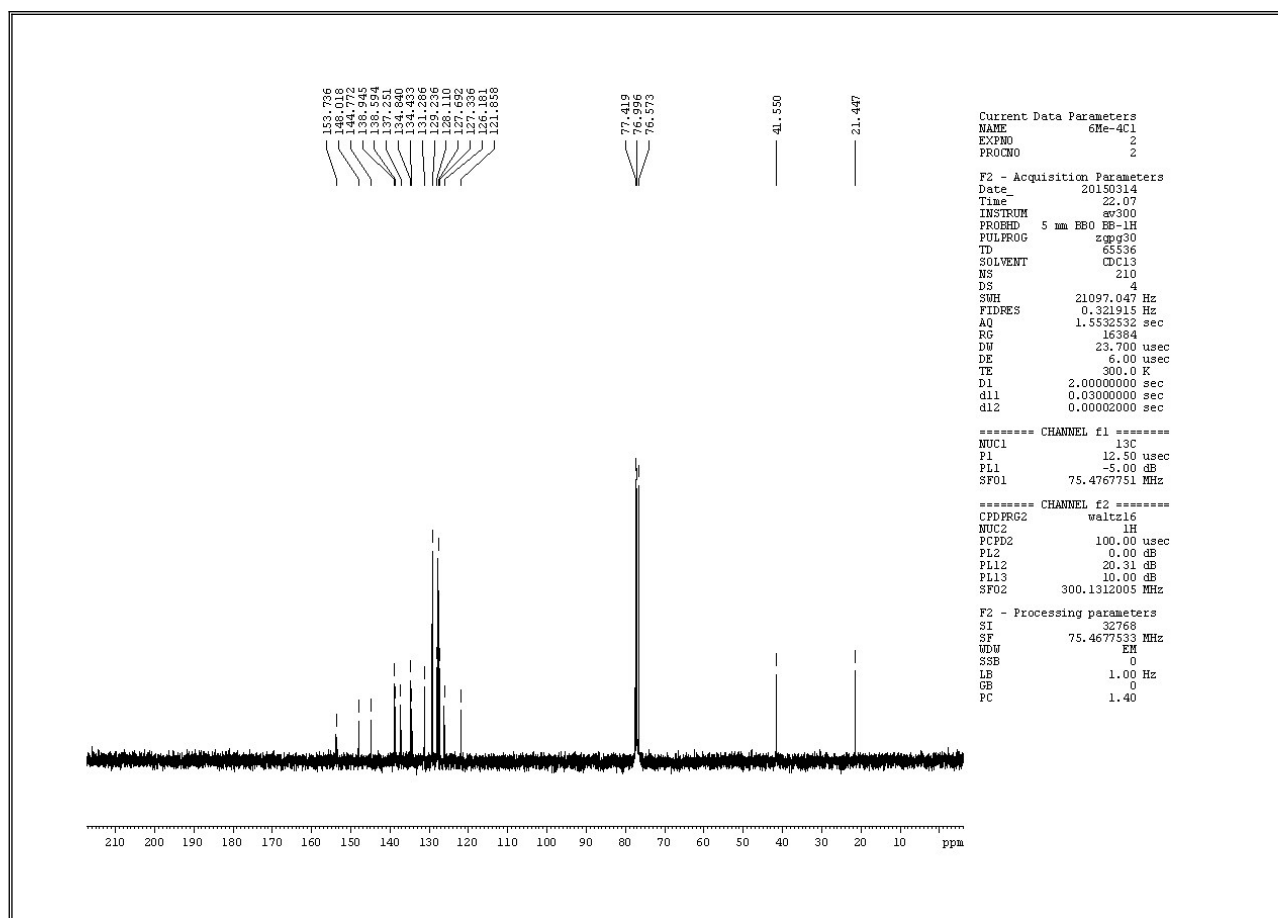


Figure. 51 ^{13}C NMR Spectrum of **3p** (CDCl_3)

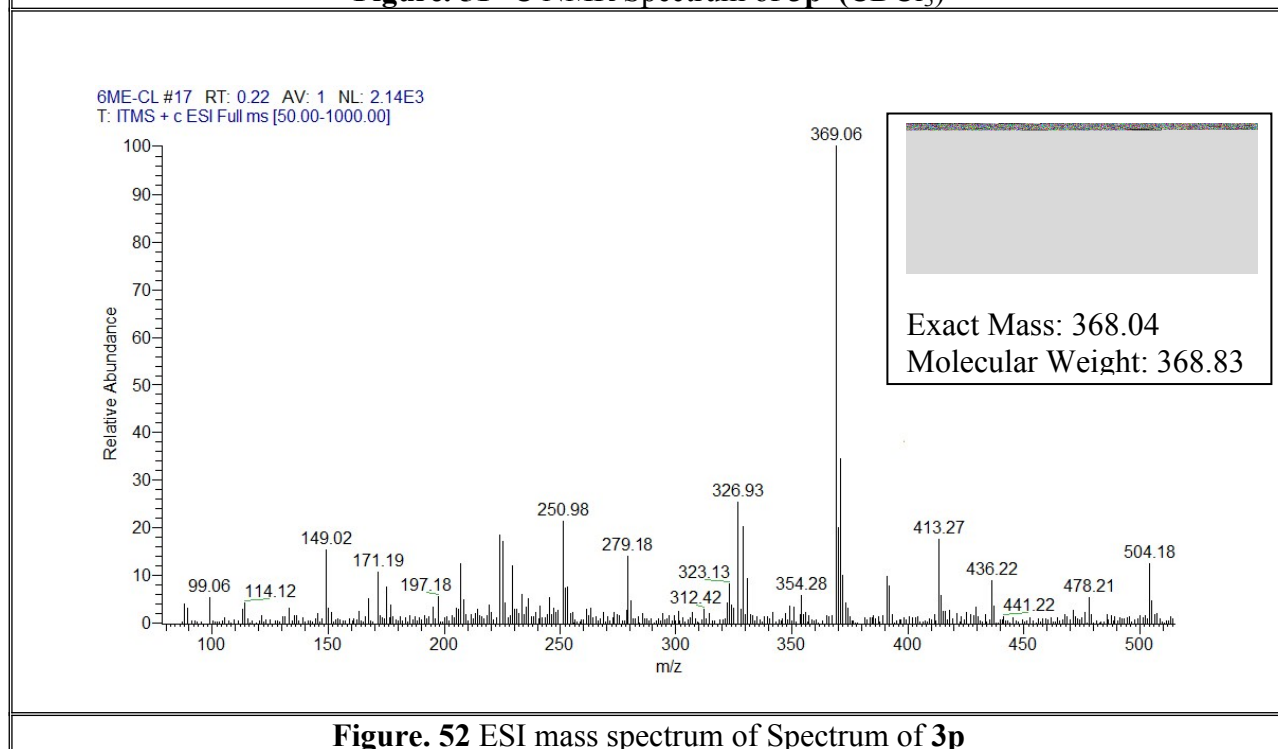


Figure. 52 ESI mass spectrum of Spectrum of **3p**

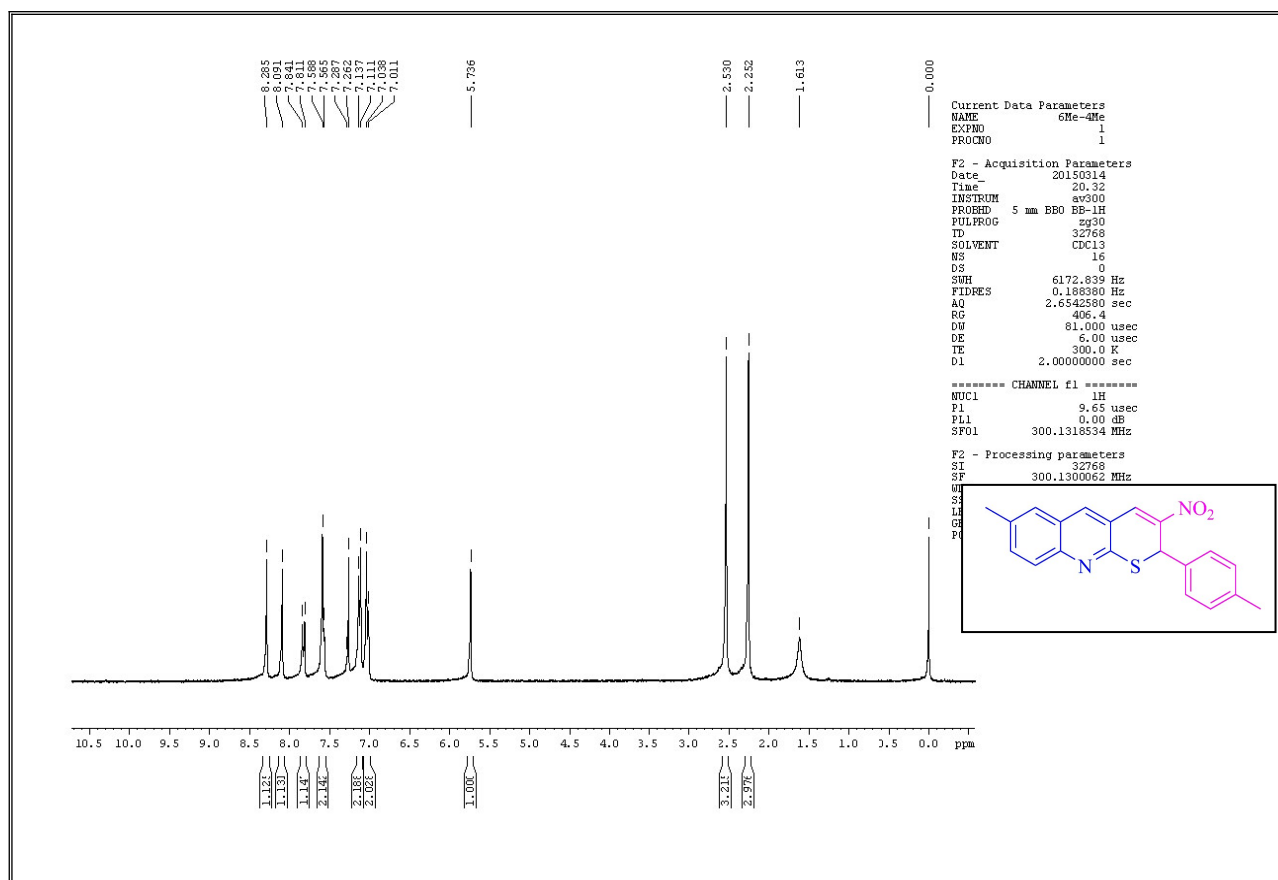


Figure. 53 ¹H NMR Spectrum **3q** (CDCl₃)

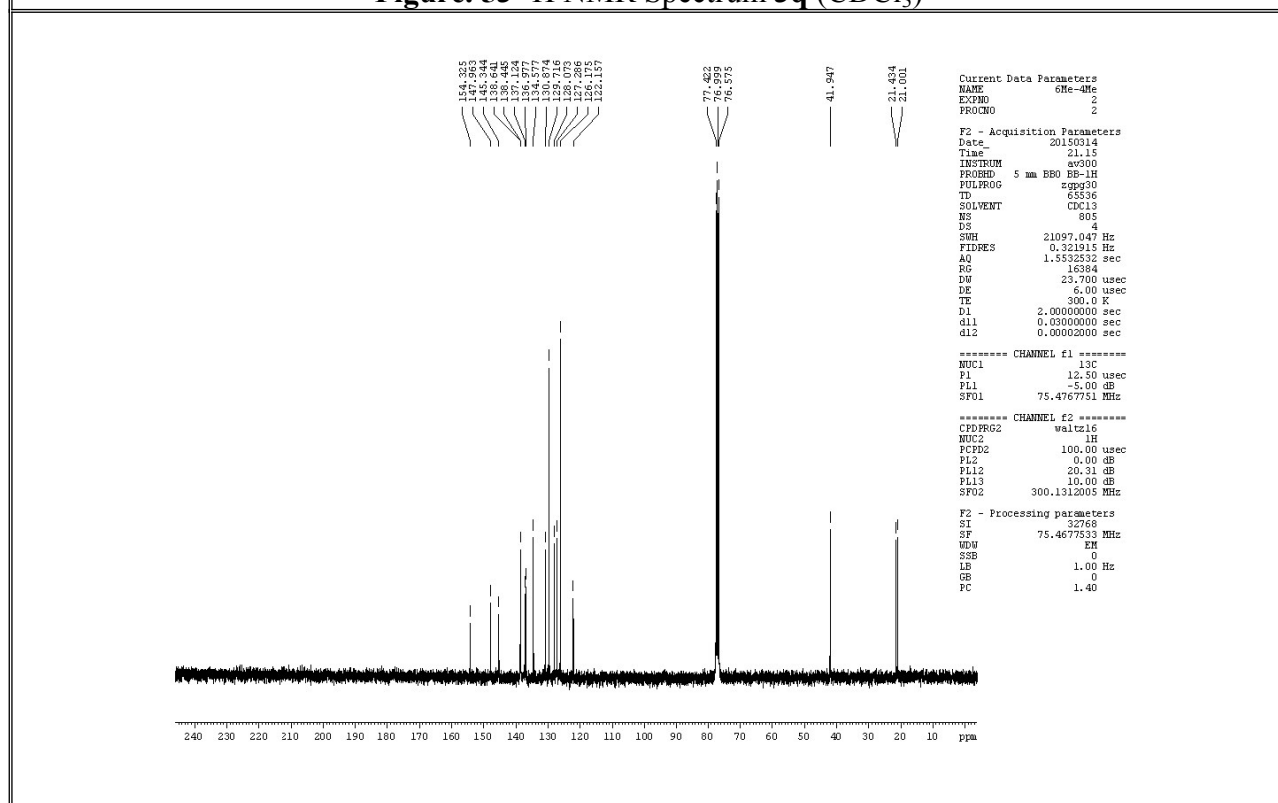


Figure. 54 ^{13}C NMR Spectrum of **3q** (CDCl_3)

6ME-ME #13 RT: 0.17 AV: 1 NL: 5.09E3
T: ITMS + c ESI Full ms [50.00-1000.00]

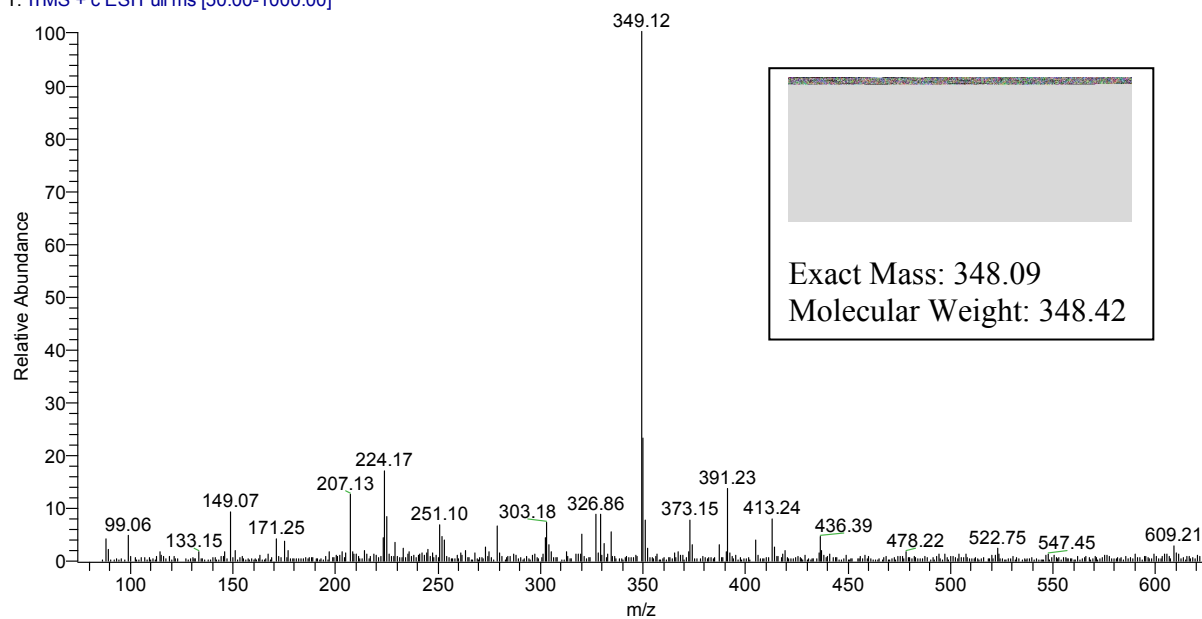


Figure. 55 ESI mass spectrum of Spectrum of **3q**

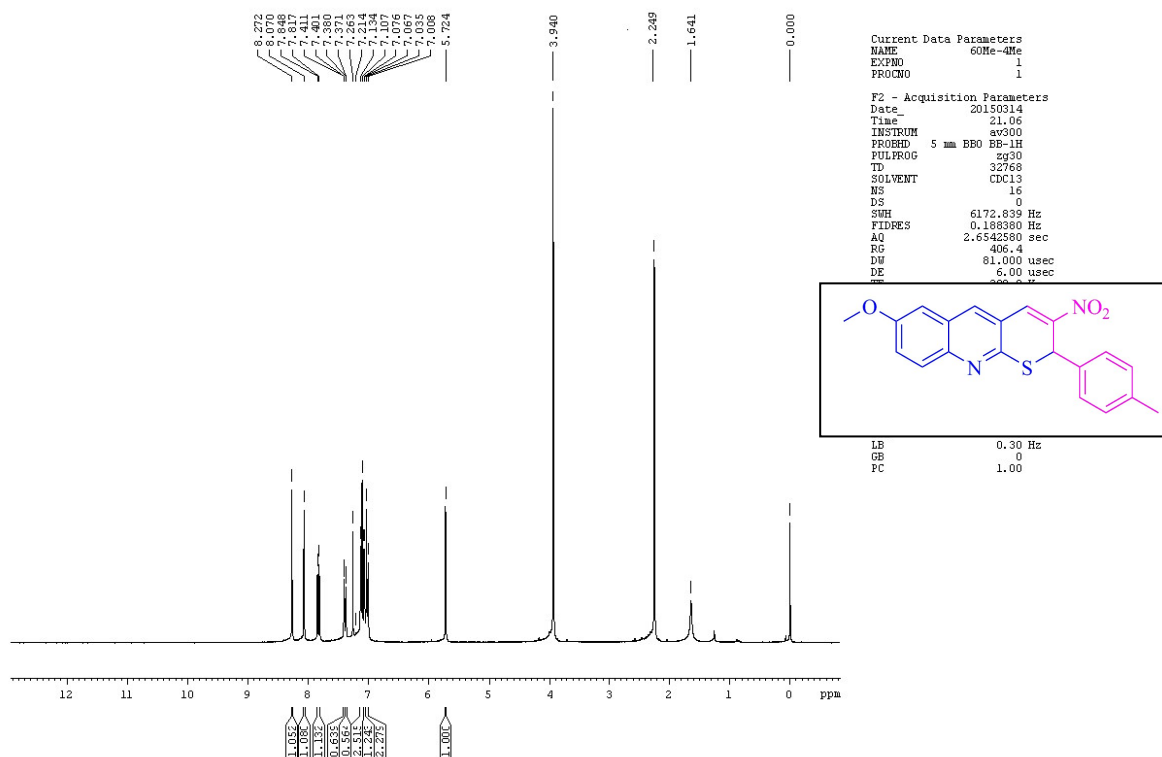


Figure. 56 ^1H NMR Spectrum **3r** (CDCl_3)

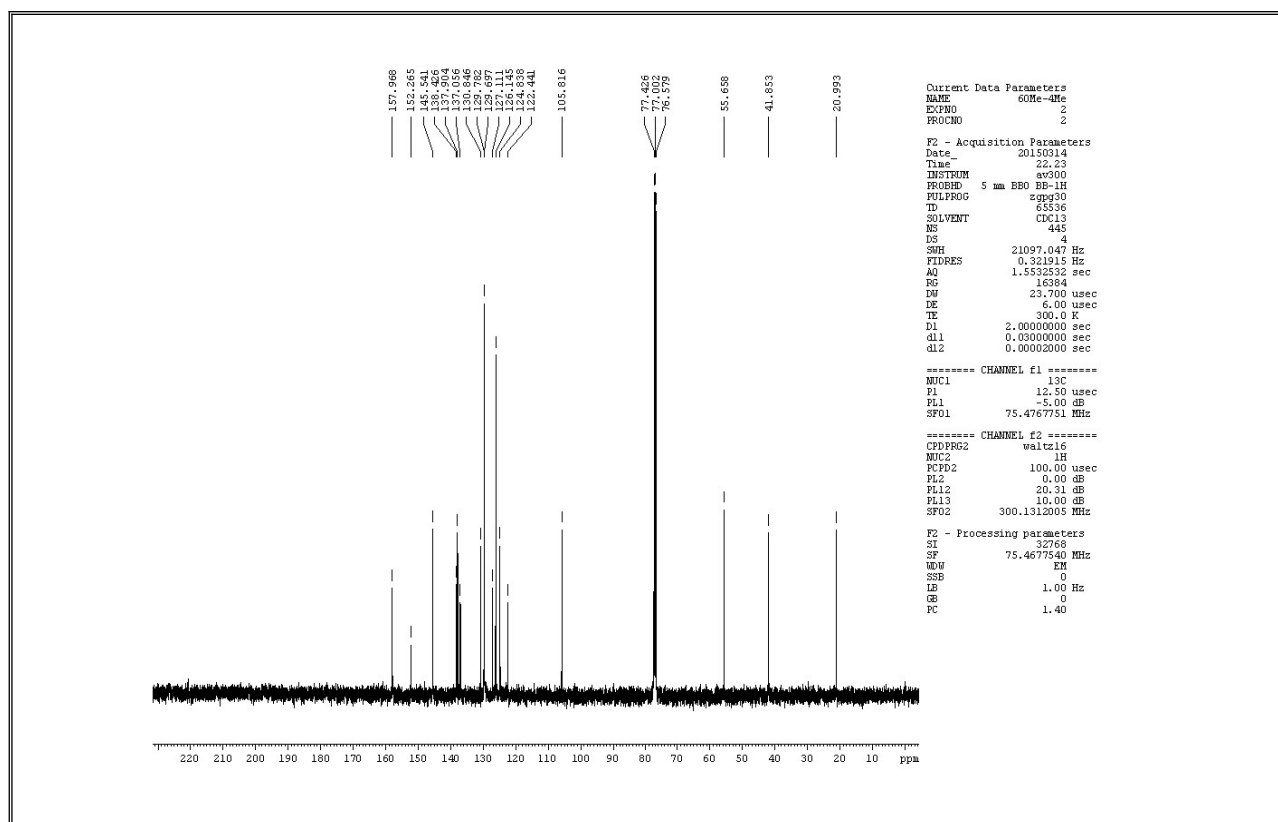


Figure. 57 ^{13}C NMR Spectrum of **3r** (CDCl_3)

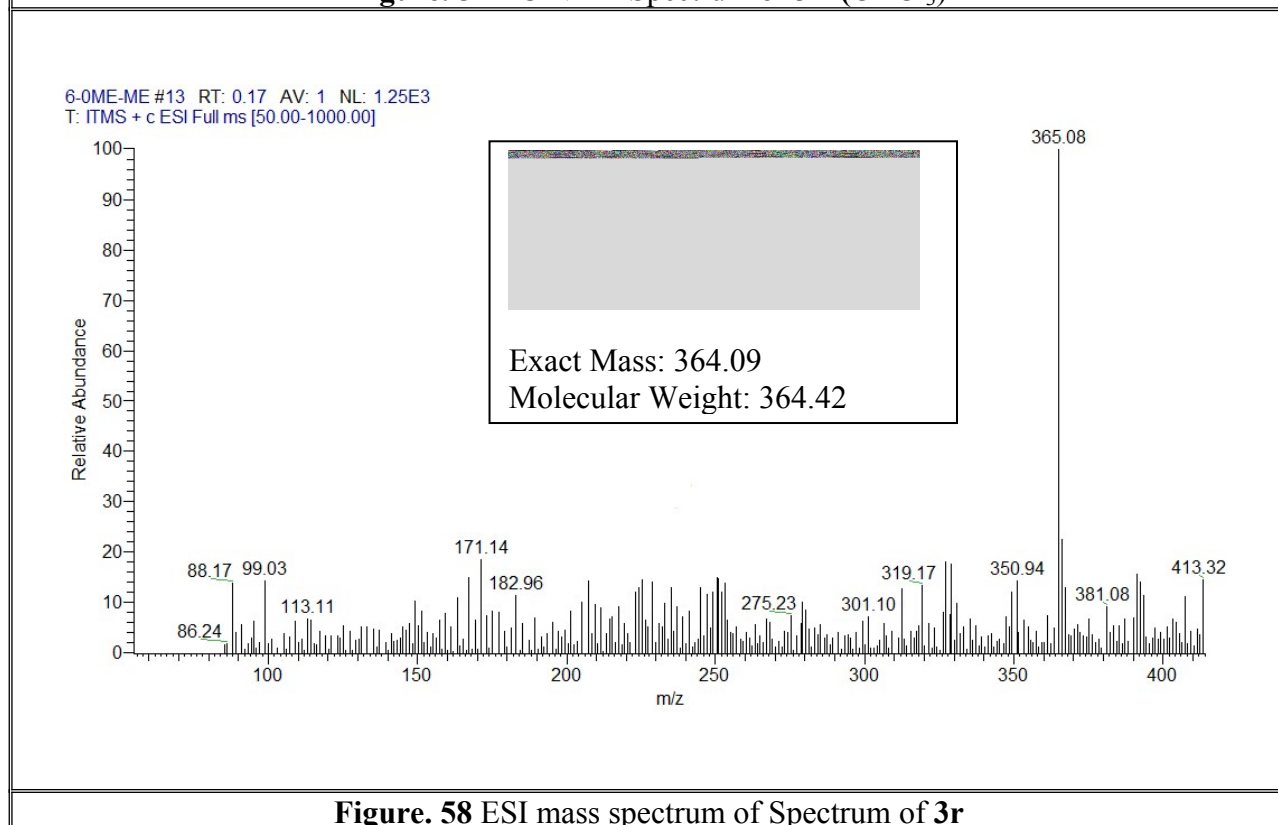


Figure. 58 ESI mass spectrum of Spectrum of **3r**