Supporting information

Title: Synthesis and anti-tubercular activity of fused thieno-/furo-quinoline compounds

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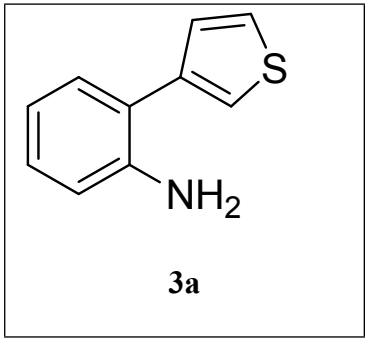
SUPPORTING INFORMATION

Contents:

2-(thiophen-3-yl)aniline (3a)

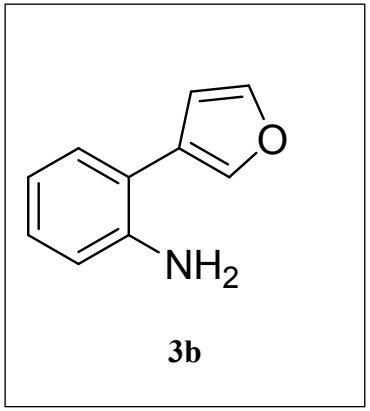
DMF (5ml) was taken in a round bottom flask and kept under nitrogen atmosphere. 2-iodoaniline (400 mg, 1.82 mmol), 3-thienyl boronic acid (280 mg, 2.2 mmol) and Cs_2CO_3 (1.2 gm, 3.6 mmol) were subsequently added to it and the reaction mixture was stirred for 5 minutes. Afterwards, $Pd(PPh_3)_4$ (422 mg, 20 mol%) was added and the reaction mixture was heated at 100 °C for 10hrs. After completion of reaction as indicated by TLC, reaction mixture was diluted with water and extracted with EtOAc (3 x 10 ml). The organic layer was washed with brine solution and dried over anhydrous Na_2SO_4. Crude product obtained was subjected to column chromatography (Silica gel, 10% EtOAc-Hexane) to afford the compound as yellow oil. Yield: 274 mg (86%). IR ν_{max}(KBr)/cm^{-1} 3445, 3365, 3098, 1615, 1489, 1259, 860, 758, 751; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.84 (s, 2H), 6.75 (dd, *J* = 8.0, 0.9 Hz, 1H), 6.80 (td, *J* = 7.5, 1.2 Hz, 1H), 7.13 (ddd, *J* = 7.9, 7.5, 1.6 Hz, 1H), 7.20 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.26 (dd, *J* = 4.9, 1.3 Hz, 1H), 7.36 (dd, *J* = 3.0, 1.3 Hz, 1H), 7.41 (dd, *J* = 4.9, 3.0 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 115.7, 118.5, 122.4, 122.5, 126.0, 128.4, 128.5, 130.2, 139.8, 143.9; ESI-MS: 176$[M+H]^+$.



3a

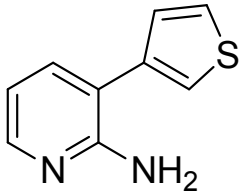
2-(furan-3-yl)aniline (3b)

DMF (5ml) was taken in a round bottom flask and kept under nitrogen atmosphere. 2-iodoaniline (200 mg, 0.91mmol), furan-3-boronic acid (122 mg, 1.09 mmol) and Cs_2CO_3 (600 mg, 1.8 mmol) were subsequently added to it and the reaction mixture was stirred for 5 minutes. Afterwards, $Pd(PPh_3)_4$ (210 mg, 20 mol%) was added and the reaction mixture was heated at 100 °C for 7hr. After completion of reaction as indicated by TLC, reaction mixture was diluted with water and extracted with EtOAc (3 x 10 ml).The organic layer was washed with brine solution and dried over anhydrous Na_2SO_4. Crude product obtained was subjected to column chromatography (Silica gel, 10% EtOAc-Hexane) to afford the compound as brown oil. Yield: 143 mg (98%). IR ν_{max}(KBr)/cm^{-1} 3465, 3365, 1615, 1489, 1157, 1080, 894, 862, 788; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.83 (s, 2H), 6.63 (dd, *J* = 1.8, 0.9 Hz, 1H), 6.73 – 6.82 (m, 2H), 7.09 – 7.14 (m, 1H), 7.20 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.51 (t, *J* = 1.7 Hz, 1H), 7.65 (dd, *J* = 1.4, 0.9 Hz, 1H); ESI-MS: 160$[M+H]^+$.



3b

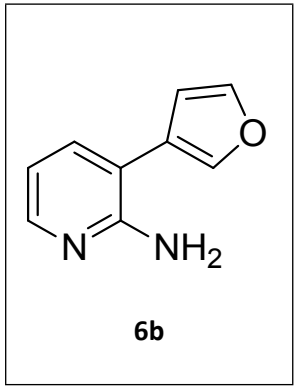
3-(thiophen-3-yl)pyridin-2-amine (6a)



6a

Prepared according to **3a** procedure except usage of 2-amino-3-bromopyridine, Yield: 82% (758 mg) brown solid, m.p. 112 °C; R_f 0.4 (hexane/EtOAc = 6 : 4); IR ν_{max}(KBr)/cm^{-1} 3476, 3295, 3136, 1629, 1574, 1448, 904, 787, 661; ^{1}H NMR (300 MHz, DMSO-d_6) δ 5.66 (s, 2H), 6.63 (dd, *J* = 7.3, 5.0 Hz, 1H), 7.32 (dd, *J* = 4.1, 2.0 Hz, 1H), 7.45 (d, *J* = 7.3 Hz, 1H), 7.74 – 7.56 (m, 2H), 8.03 – 7.85 (m, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ 113.4, 116.0, 123.4, 127.2, 128.2, 137.5, 138.7, 147.2, 156.9; ESI-MS: 177[M+H]$^+$.

3-(furan-3-yl)pyridin-2-amine (6b)

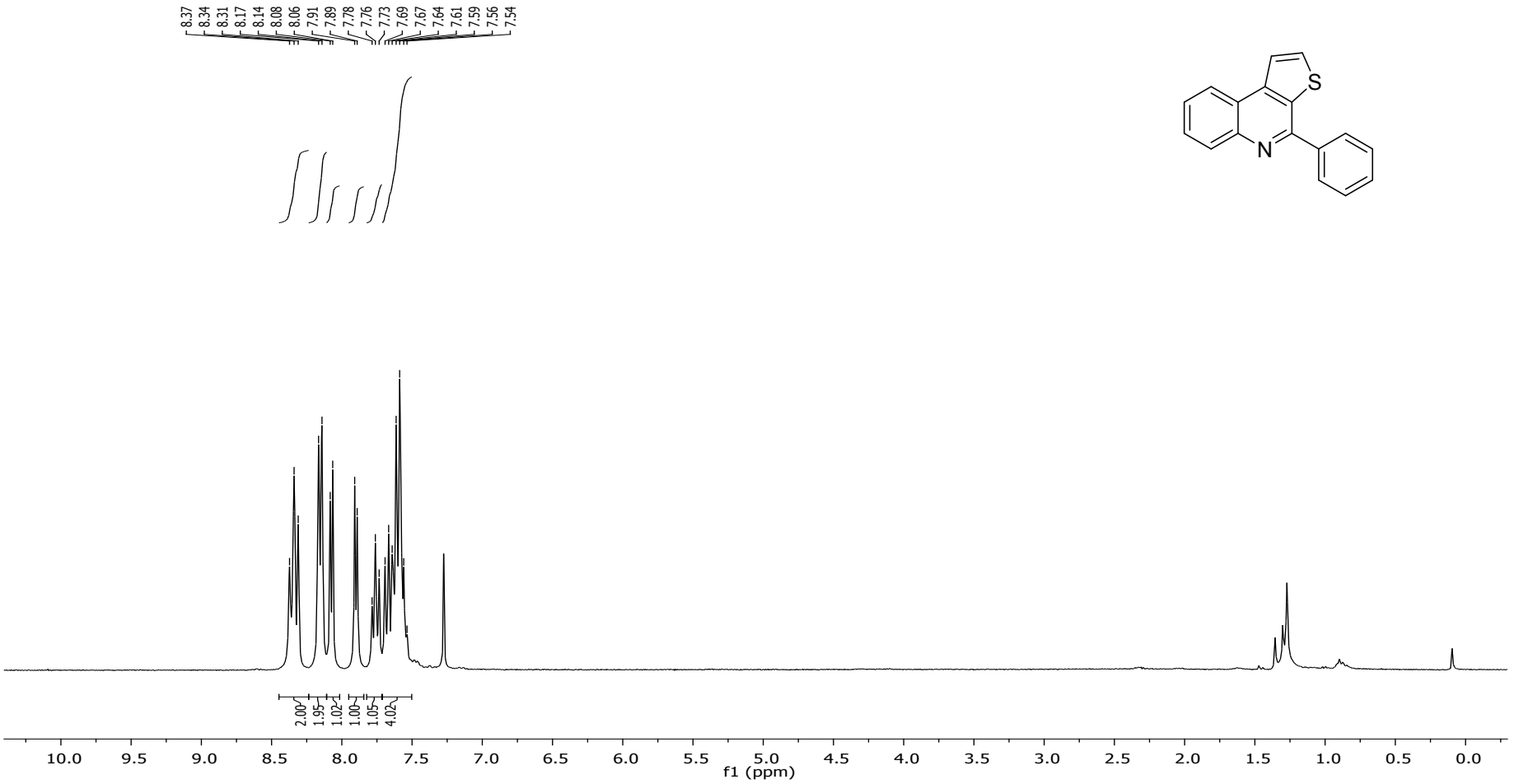


6b

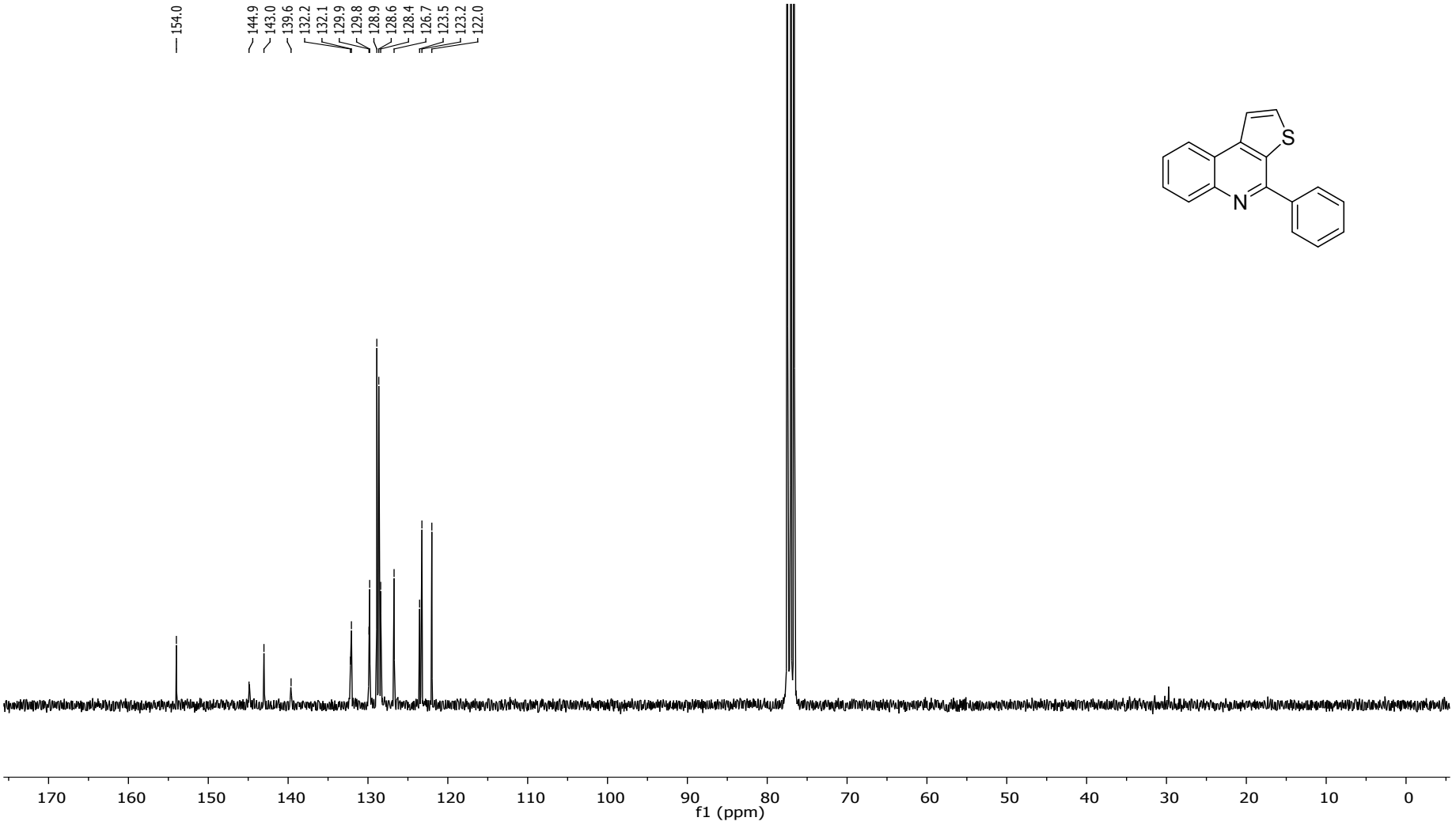
The title compound was isolated as a brown liquid (92.6%, 935mg), R_f 0.3 [hexane–EtOAc (6 : 4)]; IR ν_{max}(KBr)/cm^{-1} 3462, 3317, 1614, 1505, 1451, 1161, 926, 872, 774; ^{1}H NMR (300 MHz, DMSO-d_6): δ 5.69 (s, 2H), 6.64 (dd, *J* = 7.4, 4.9 Hz, 1H), 6.84 (d, *J* = 0.6 Hz, 1H), 7.52 (d, *J* =

6.4 Hz, 1H), 7.78 (s, 1H), 7.92 (d, J = 3.6 Hz, 1H), 8.02 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ 110.6, 112.0, 113.5, 122.4, 136.7, 140.4, 144.1, 146.9, 156.8; ESI-MS:161$[M+H]^+$.

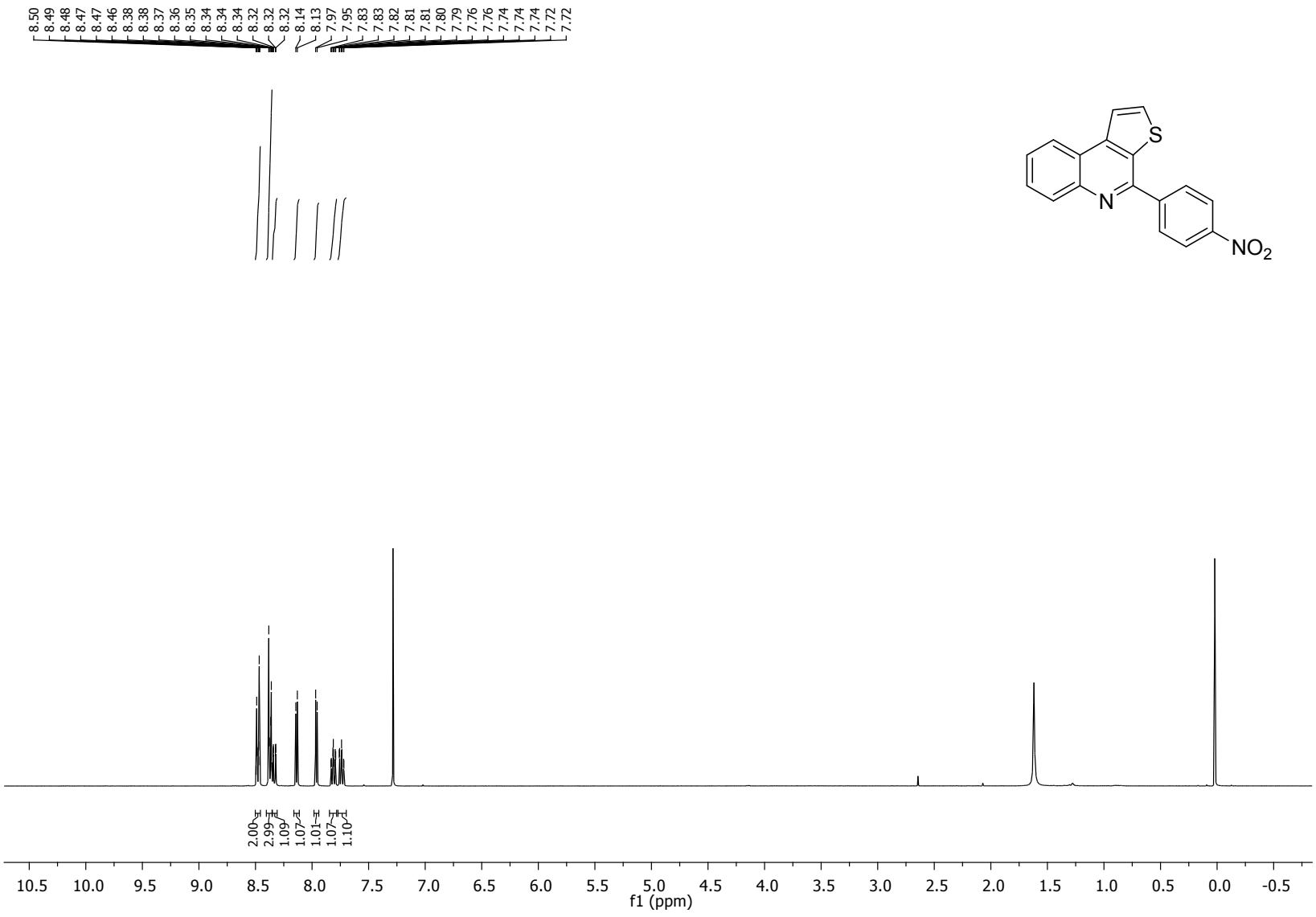
^{1}H NMR (300 MHz, $CDCl_3$): 4-phenylthieno[2,3-*c*]quinoline(4a)



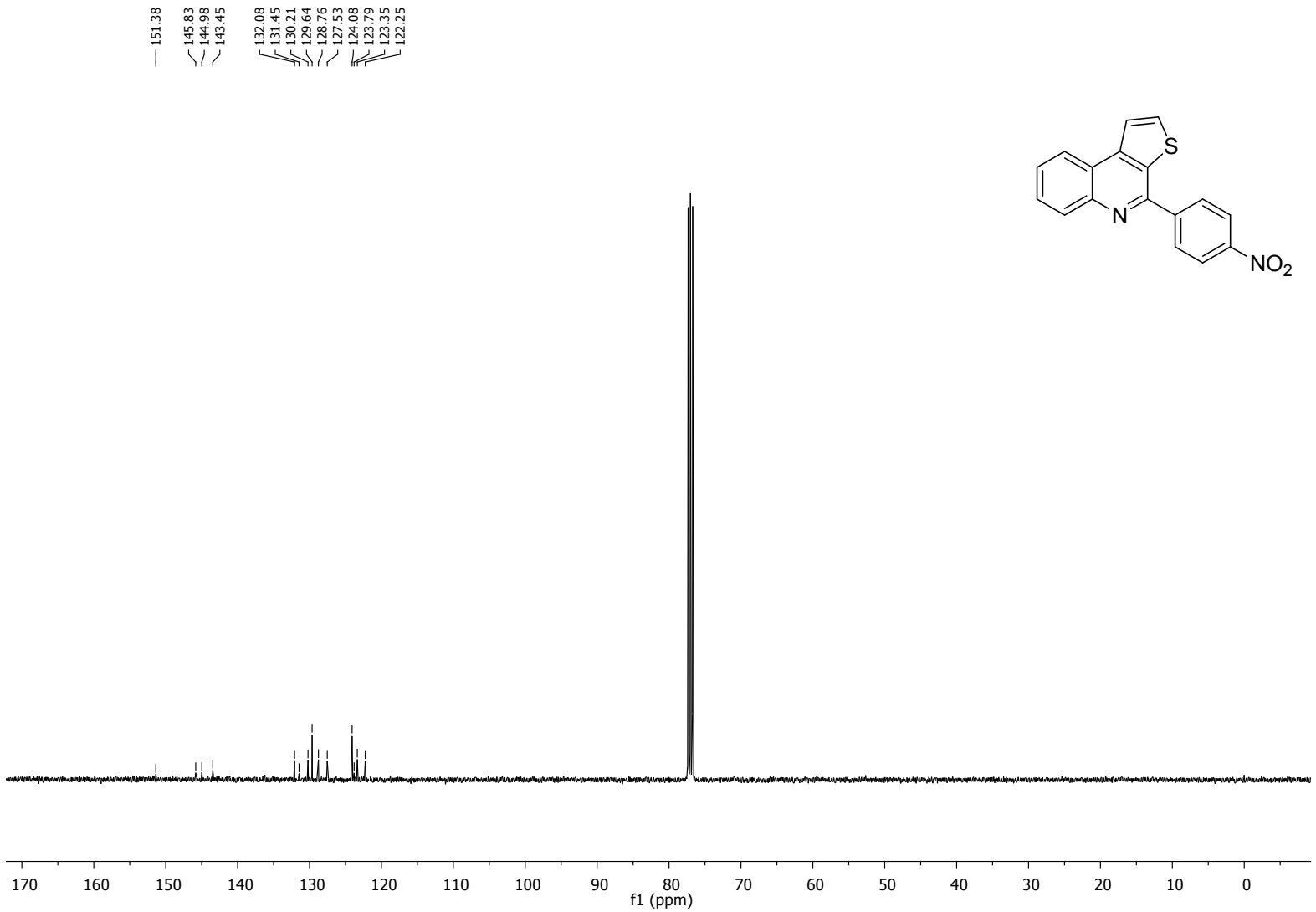
^{13}C NMR (75 MHz, $CDCl_3$):4-phenylthieno[2,3-*c*]quinoline(4a)



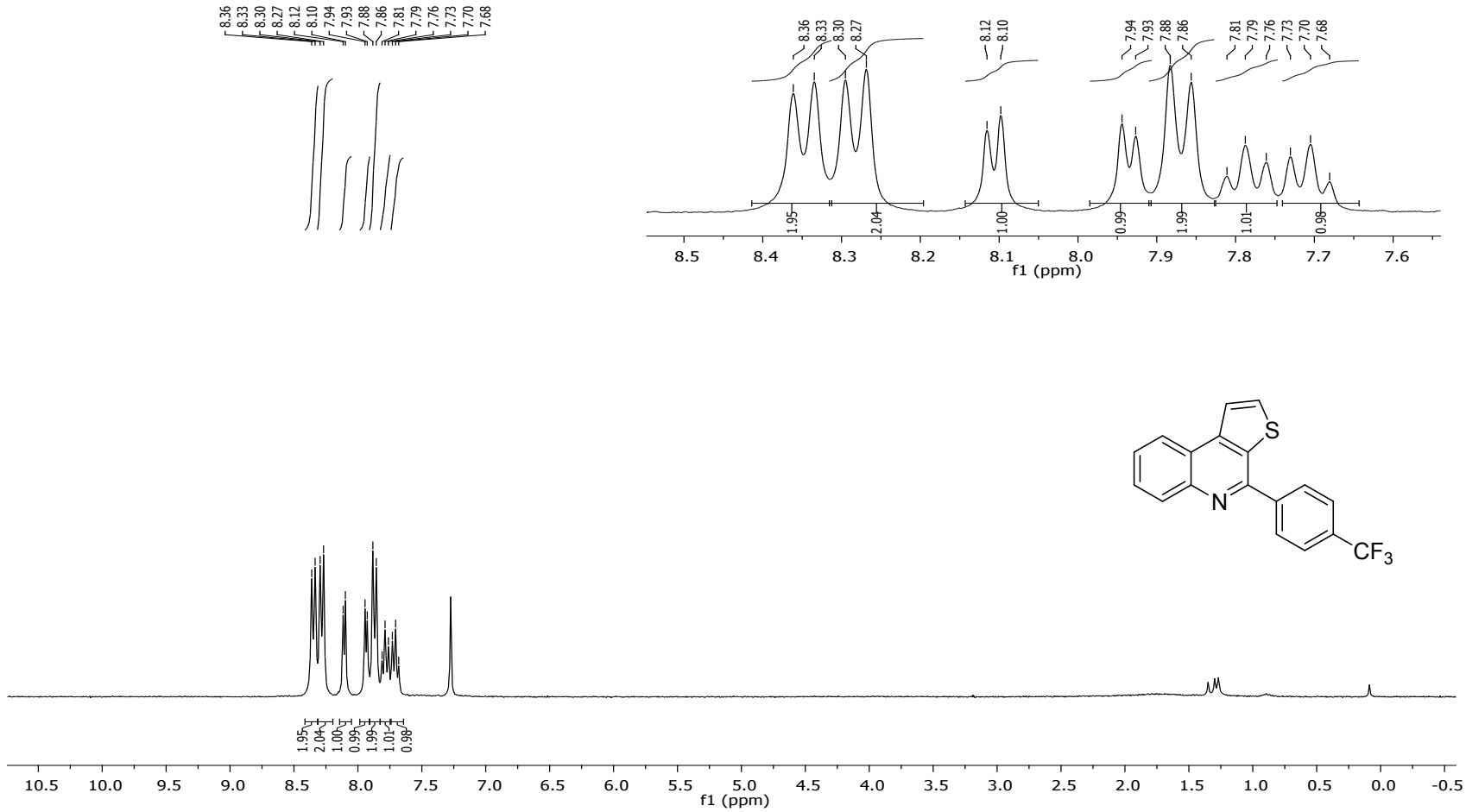
^{1}H NMR (400 MHz, $CDCl_3$):4-(4-nitrophenyl)thieno[2,3-*c*]quinoline(4b)



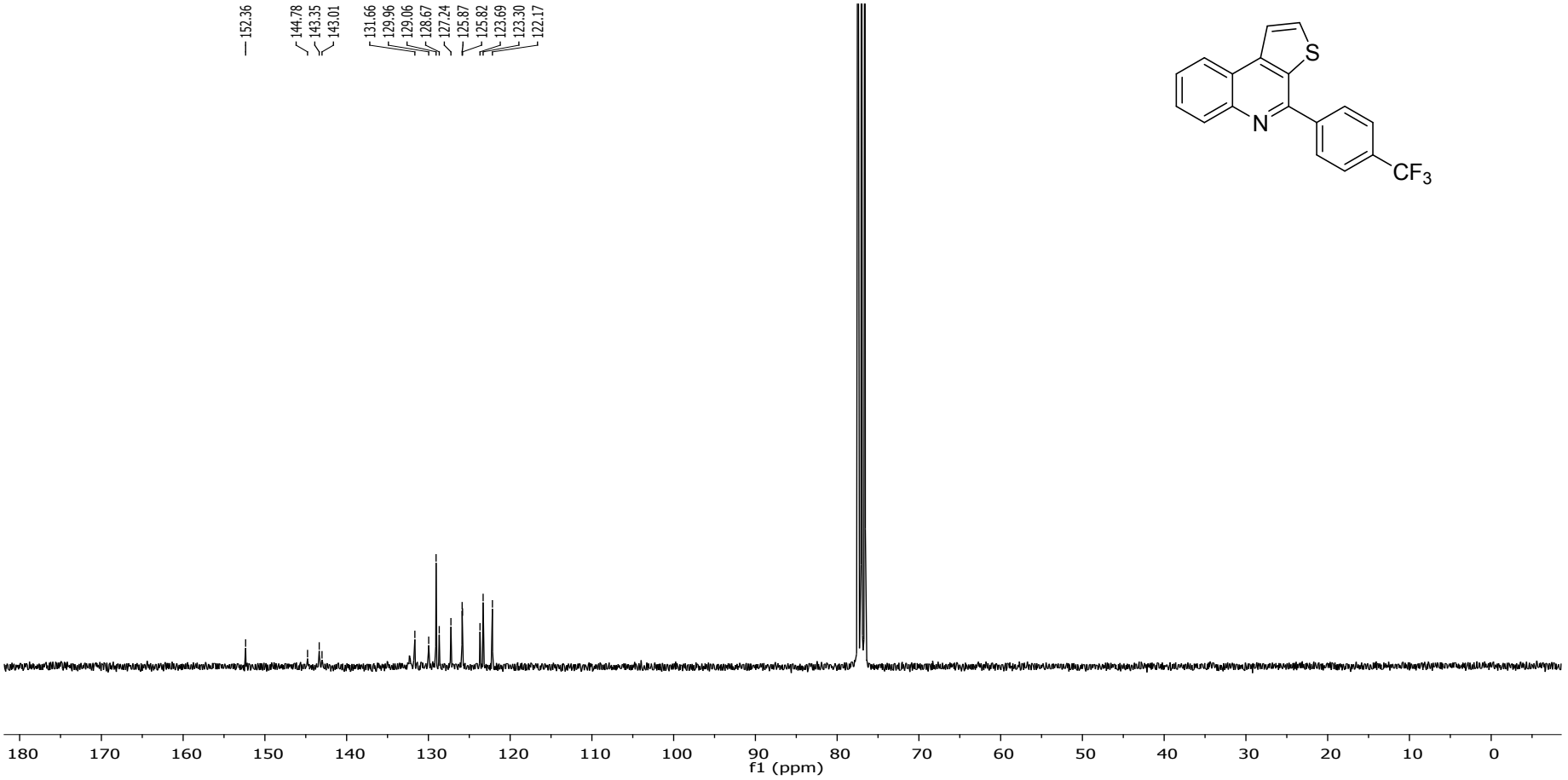
^{13}C NMR (101 MHz, $CDCl_3$): 4-(4-nitrophenyl)thieno[2,3-*c*]quinoline(4b)



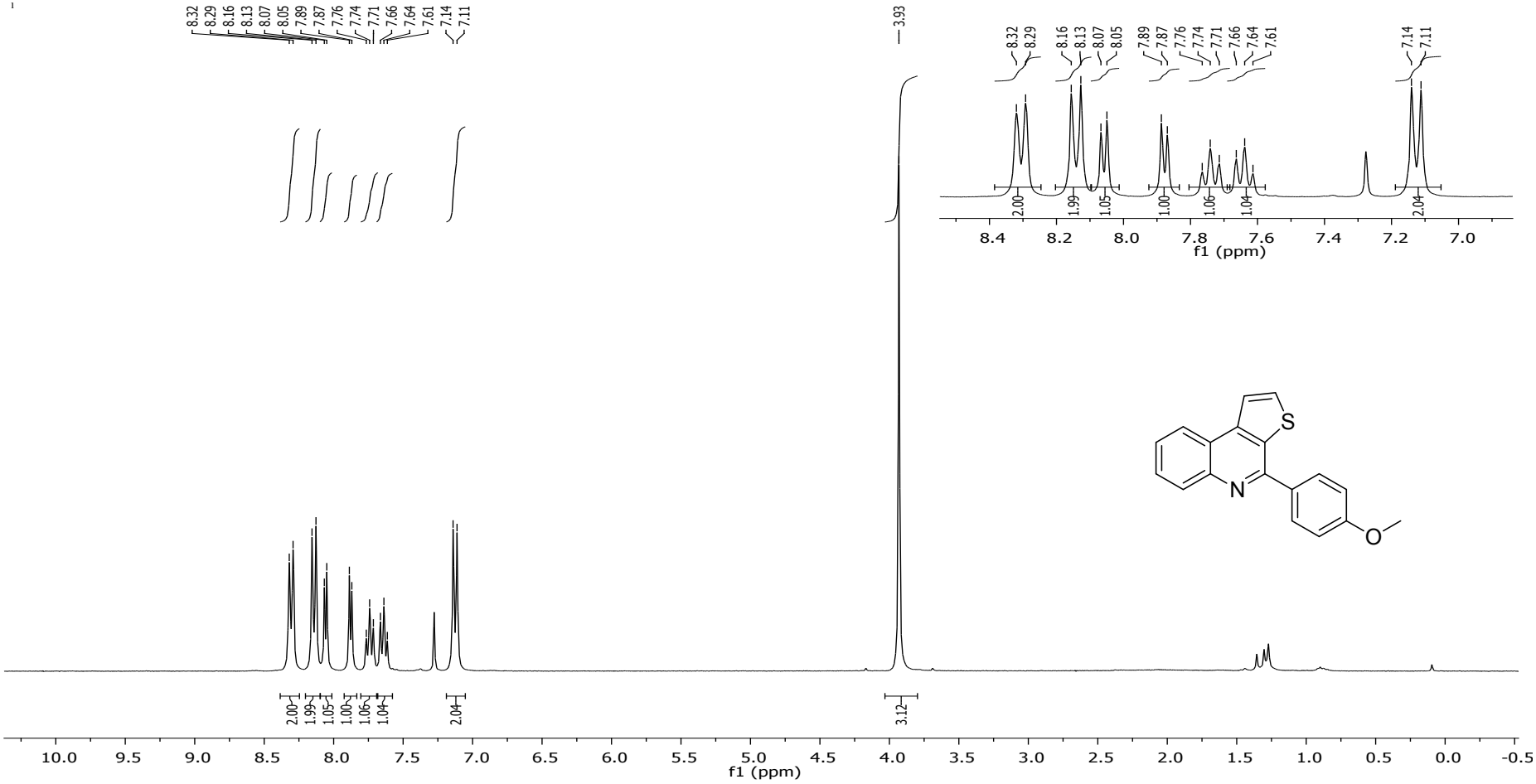
^{1}H NMR (300 MHz, $CDCl_3$): 4-(4-(trifluoromethyl)phenyl)thieno[2,3-*c*]quinoline(4c)



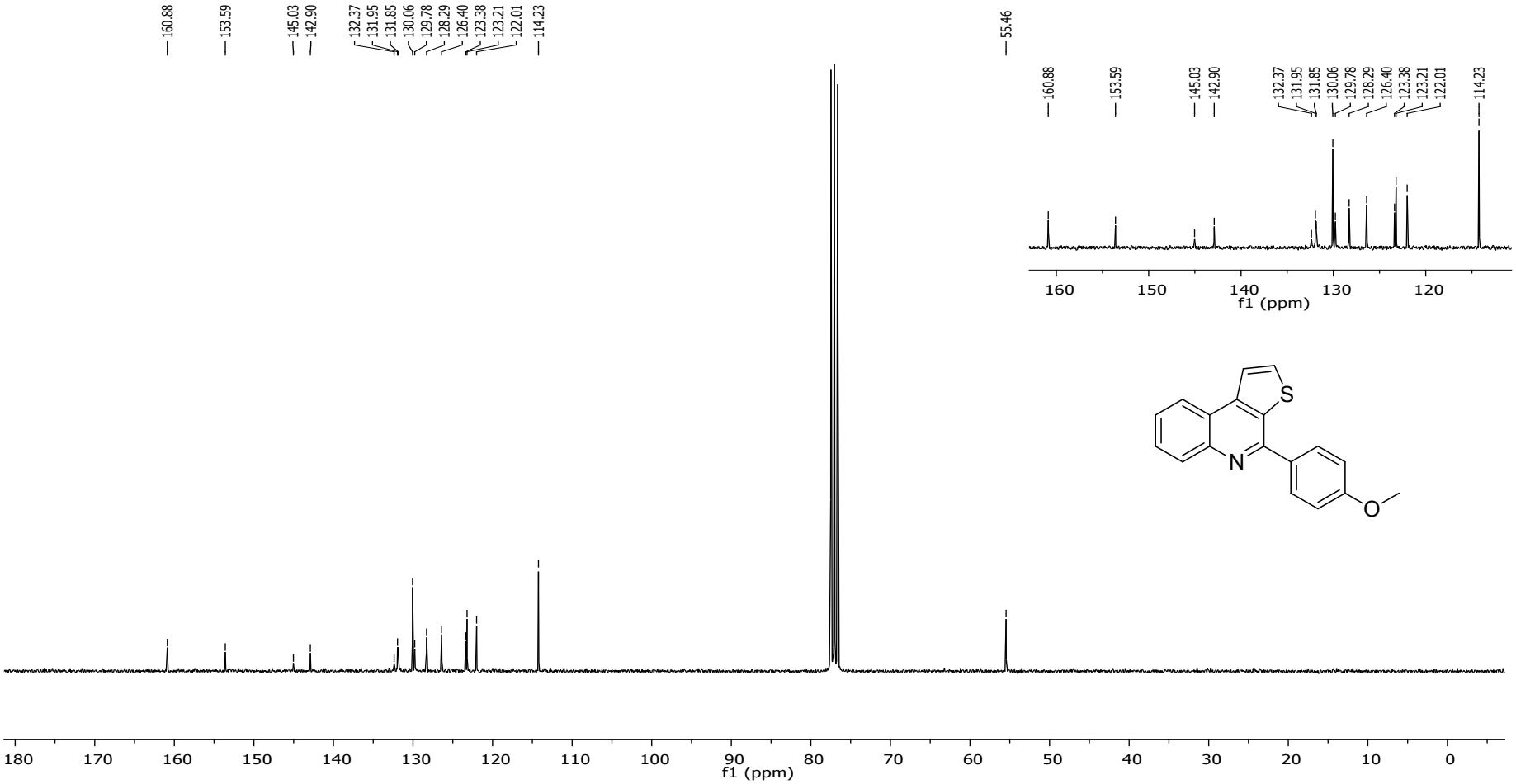
^{13}C NMR (75 MHz, $CDCl_3$): 4-(4-(trifluoromethyl)phenyl)thieno[2,3-*c*]quinoline(4c)



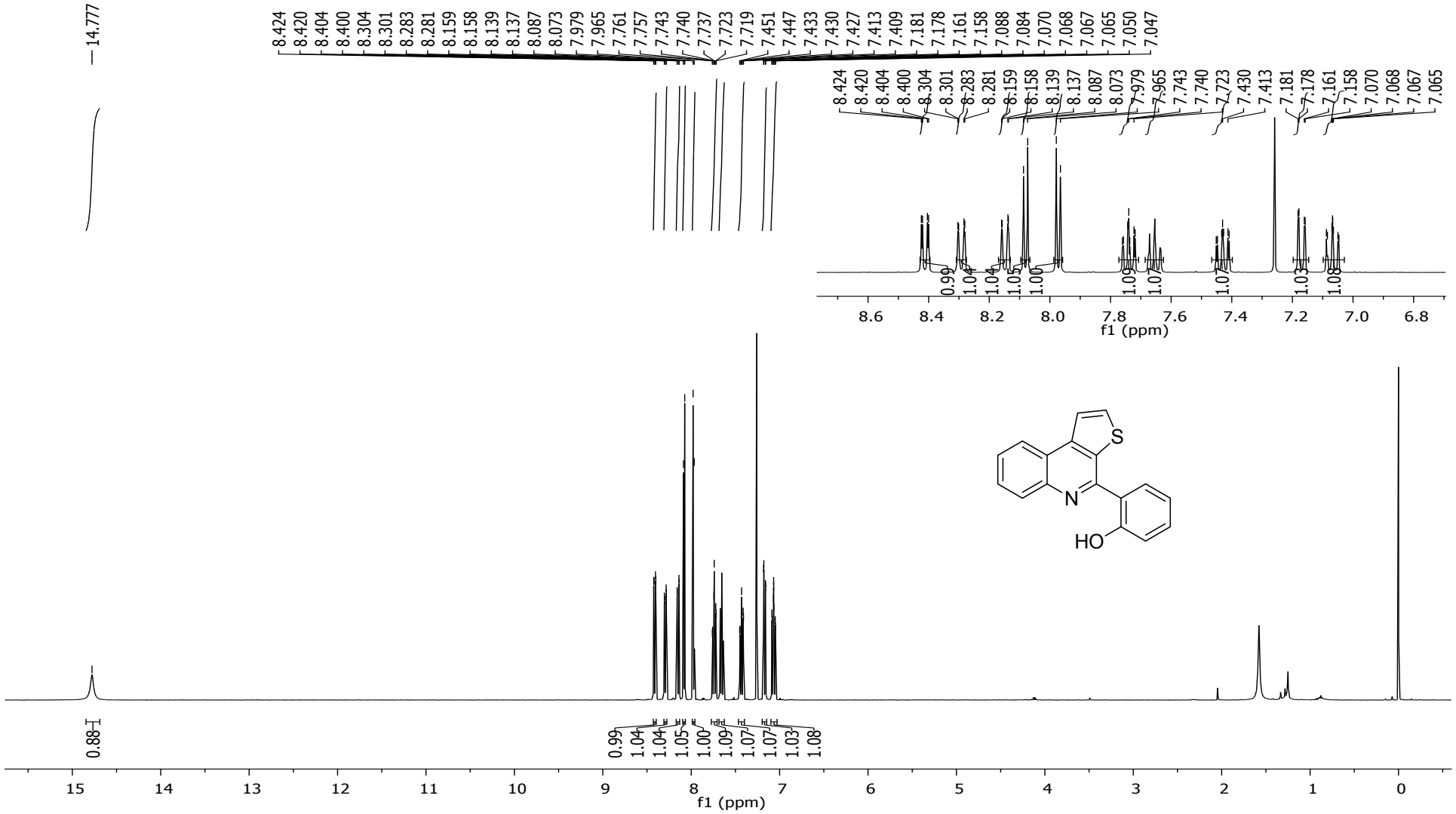
^{1}H NMR (300 MHz, $CDCl_3$): 4-(4-methoxyphenyl)thieno[2,3-*c*]quinoline(4d)



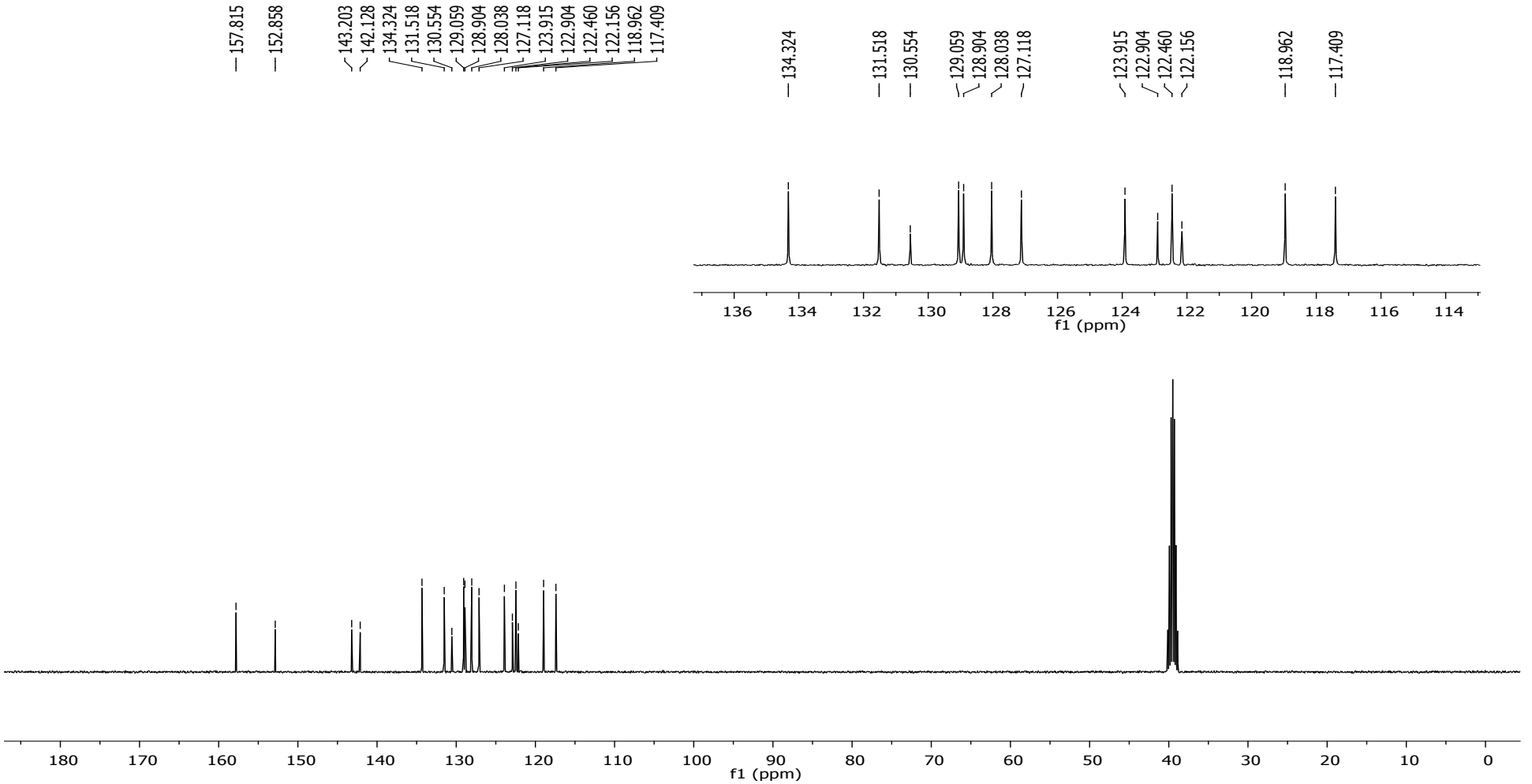
^{13}C NMR (75 MHz, $CDCl_3$): 4-(4-methoxyphenyl)thieno[2,3-*c*]quinoline(4d)



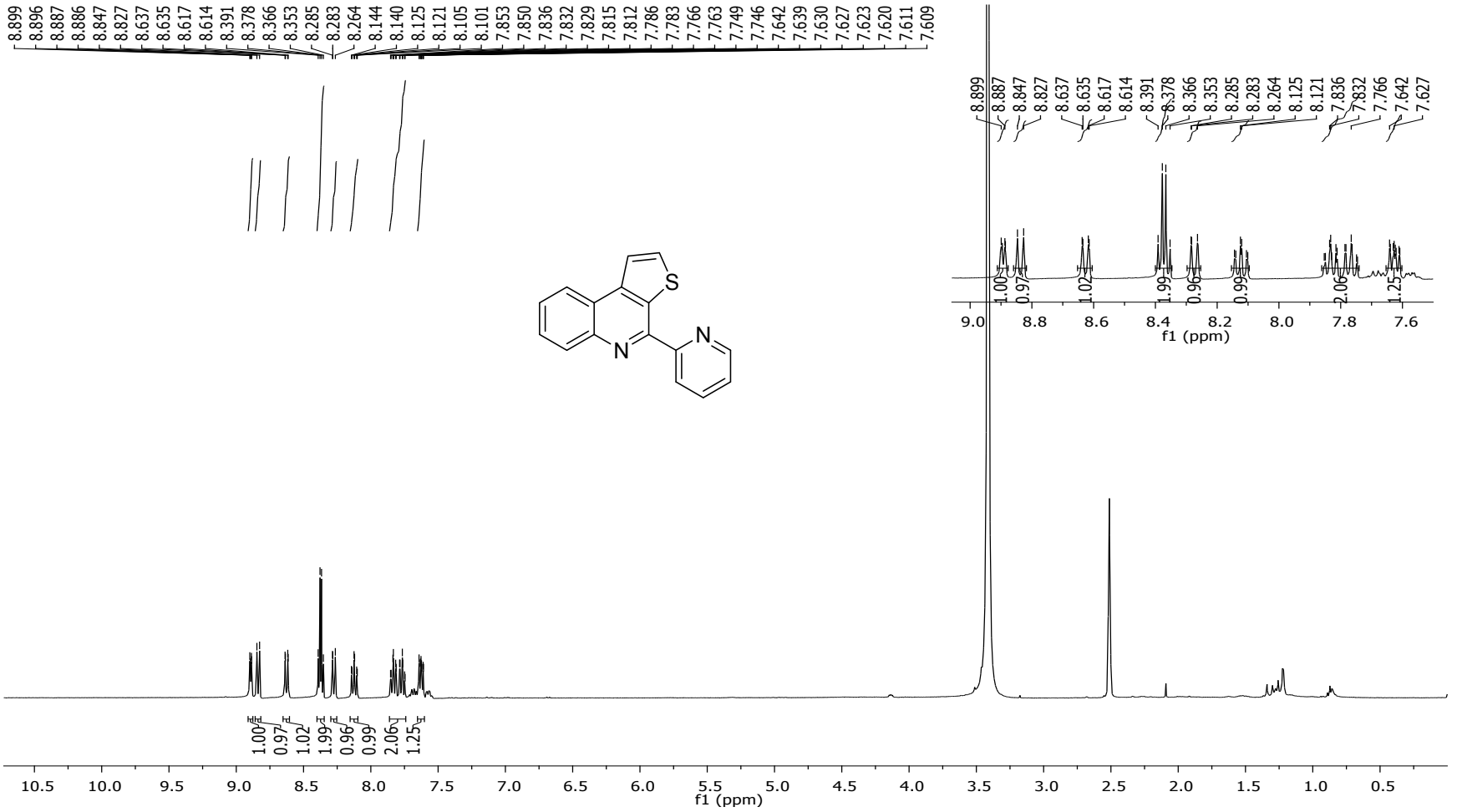
^{1}H NMR (400 MHz, $CDCl_3$): 2-(thieno[2,3-*c*]quinolin-4-yl)phenol(4e)



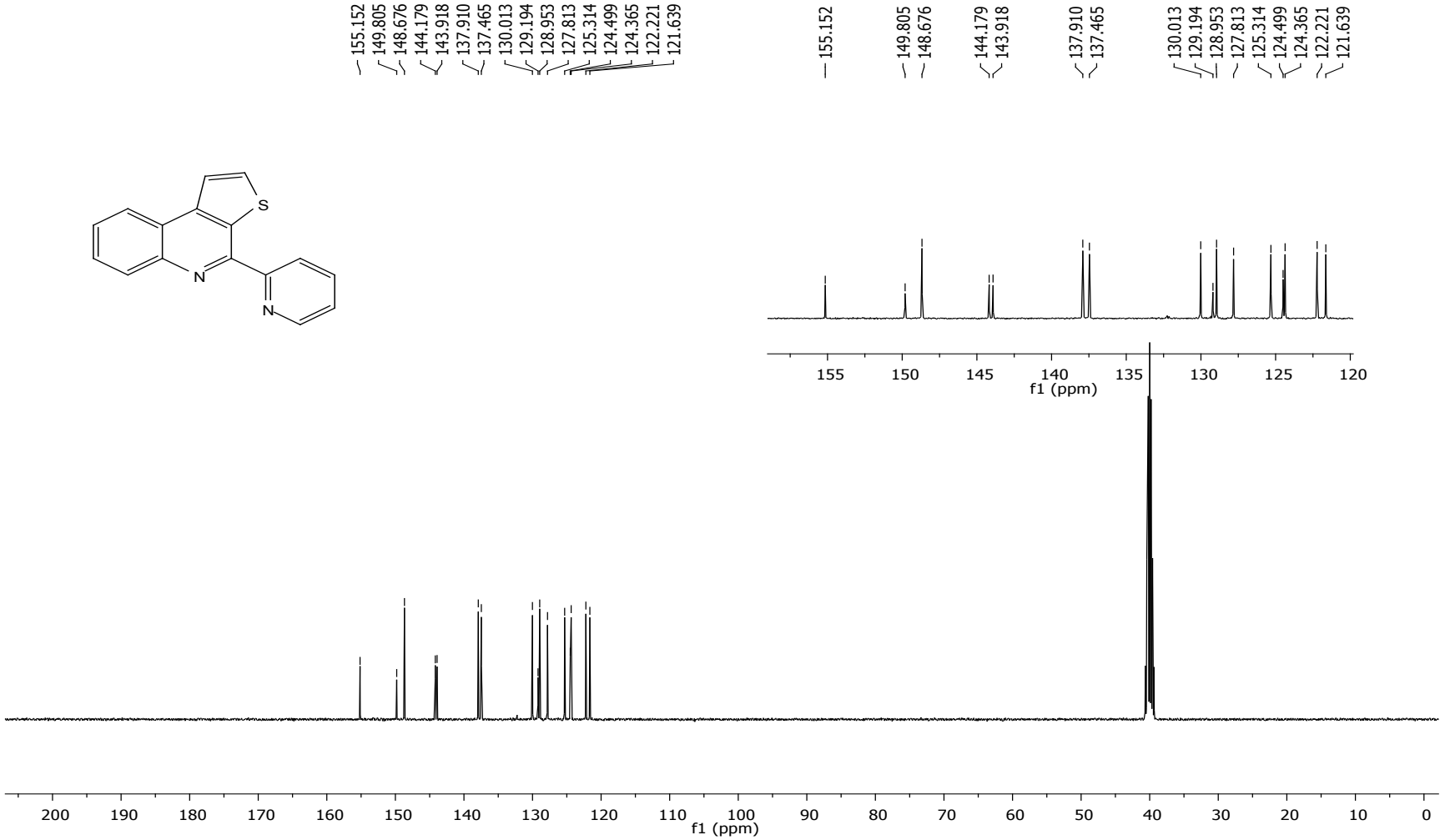
^{13}C NMR (101 MHz, $CDCl_3$): 2-(thieno[2,3-*c*]quinolin-4-yl)phenol(4e)



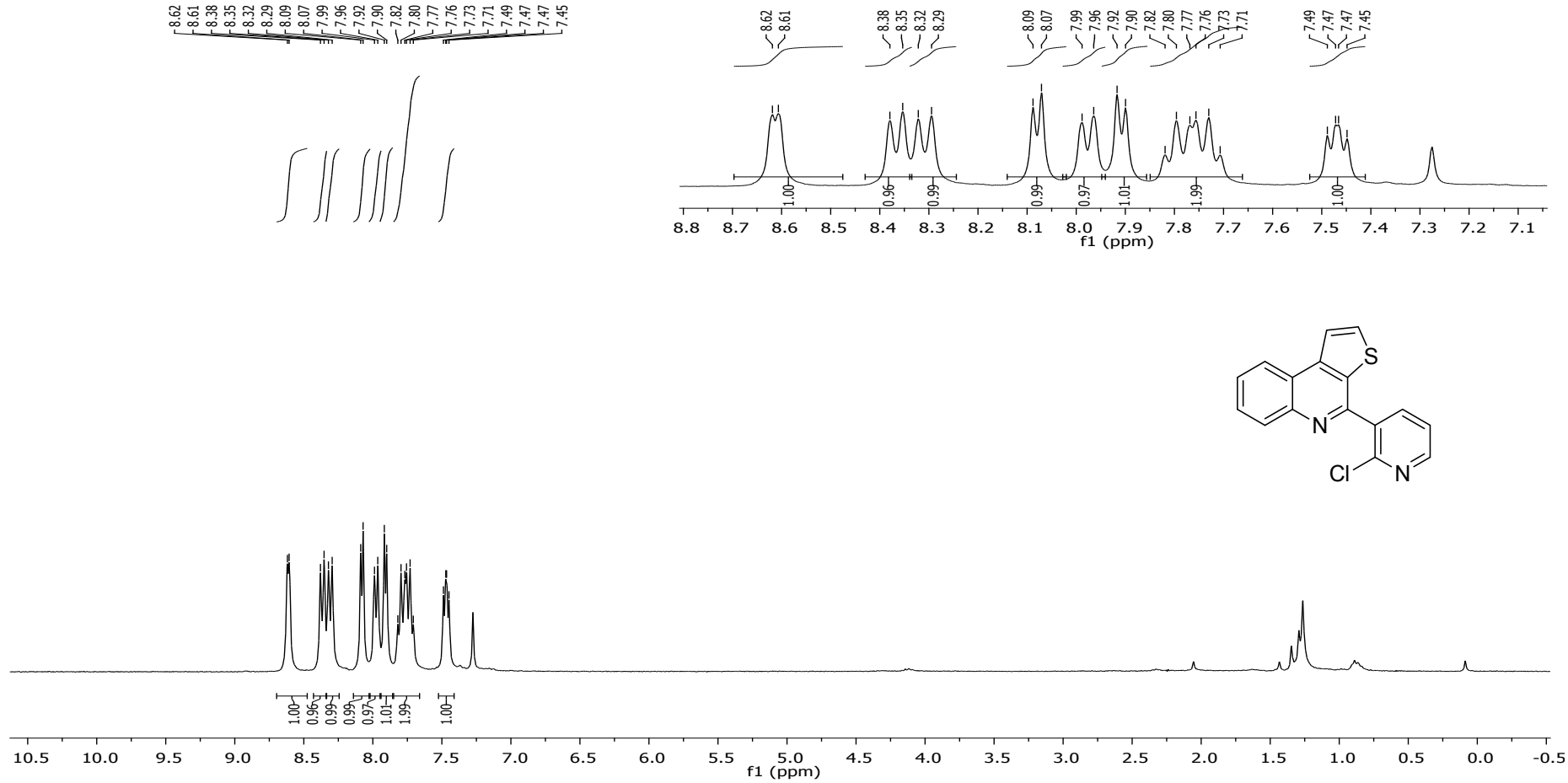
^{1}H NMR (400 MHz, DMSO-d_6): 4-(pyridin-2-yl)thieno[2,3-*c*]quinoline(4f)



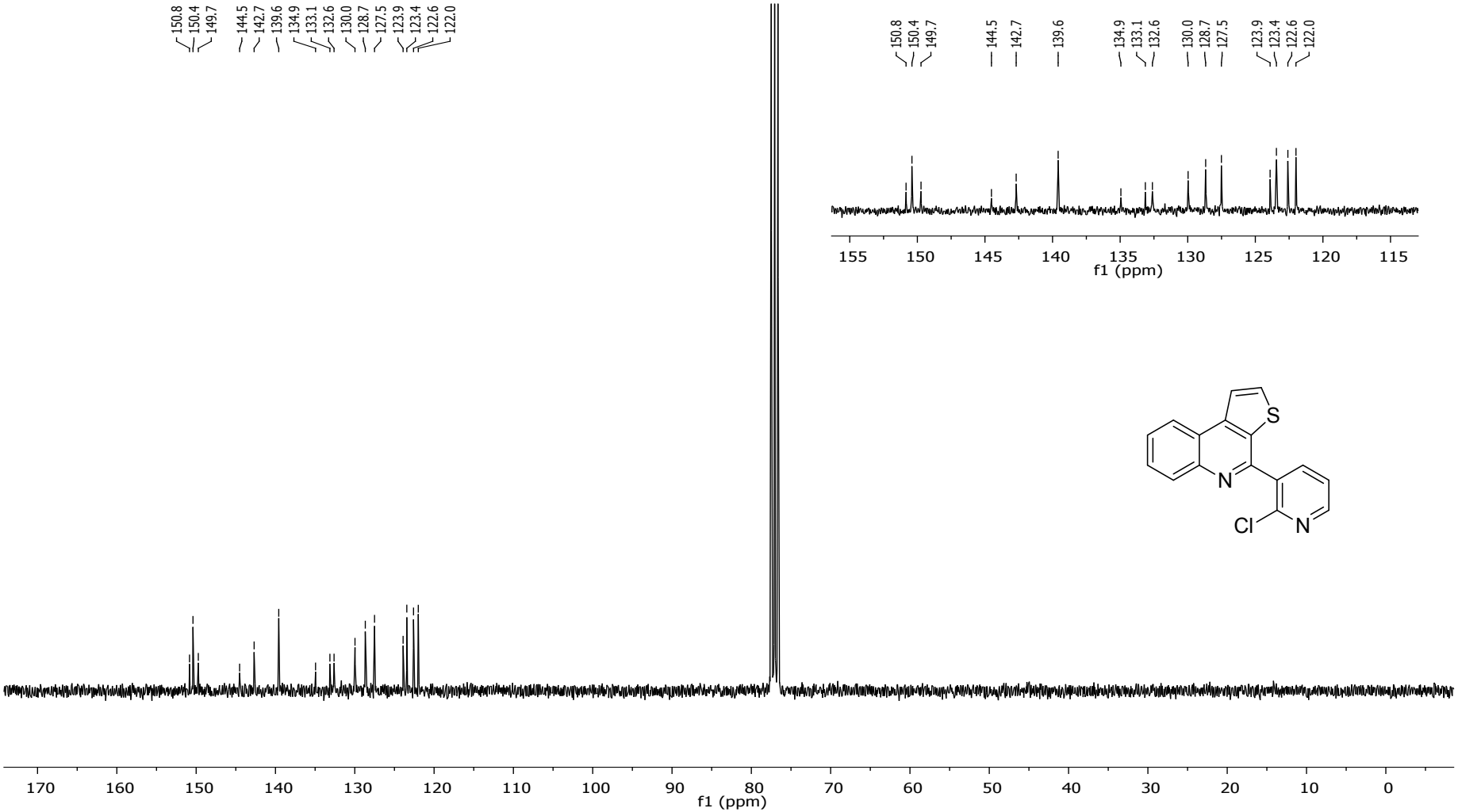
^{13}C NMR (101 MHz, DMSO-d_6): 4-(pyridin-2-yl)thieno[2,3-*c*]quinoline(4f)



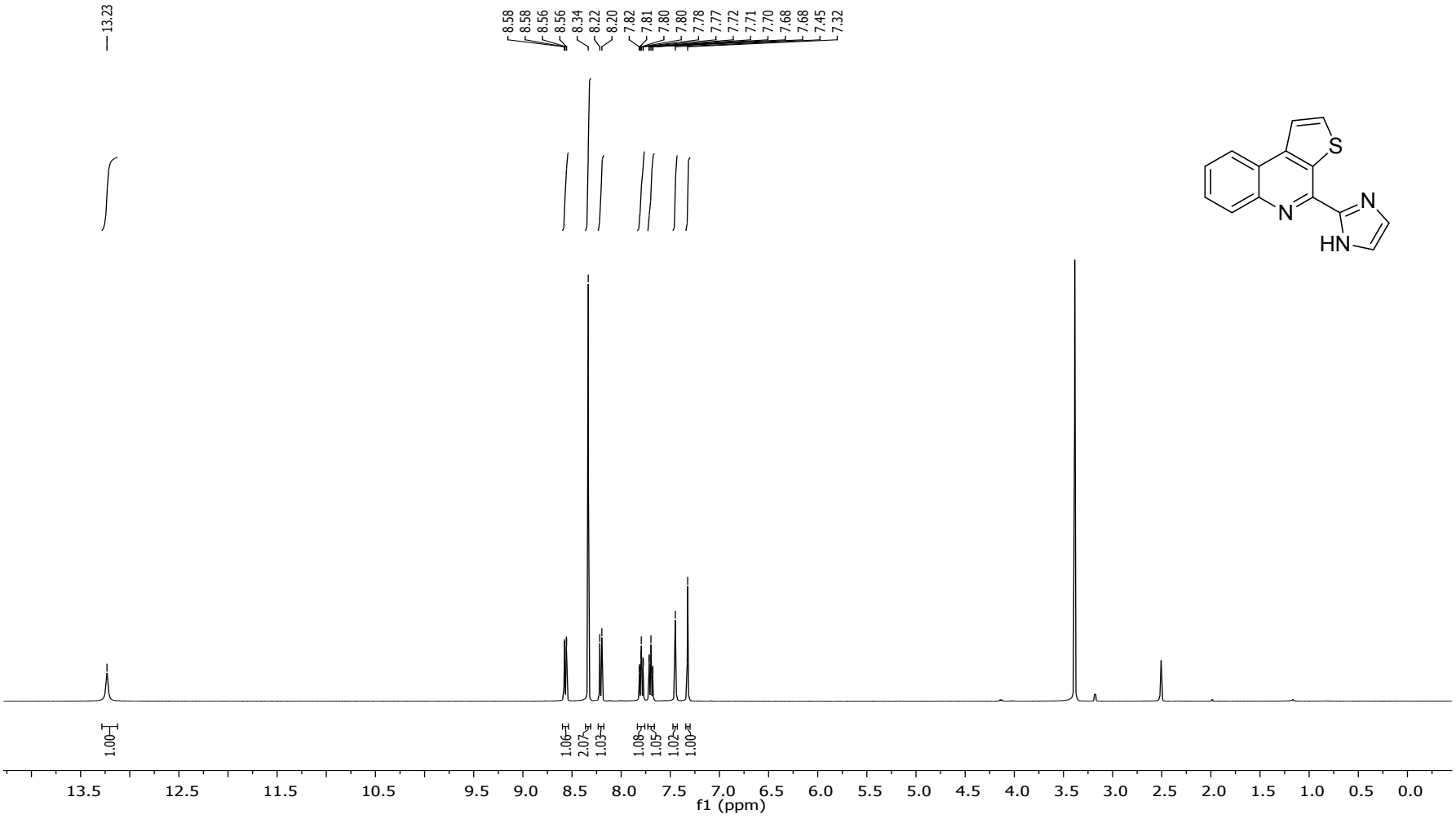
^{1}H NMR (300 MHz, $CDCl_3$): 4-(2-chloropyridin-3-yl)thieno[2,3-*c*]quinoline(4g)



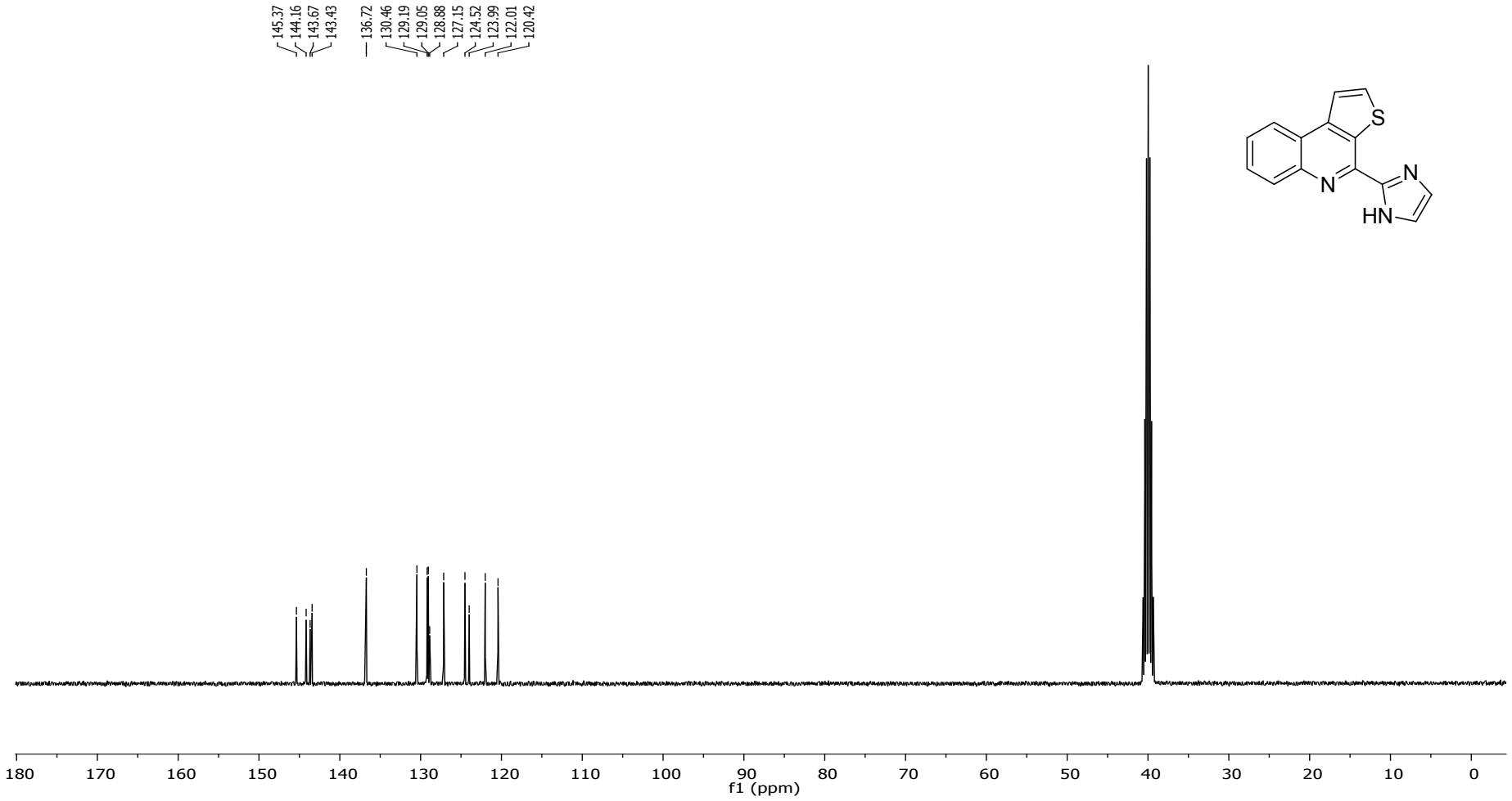
^{13}C NMR (75 MHz, $CDCl_3$): 4-(2-chloropyridin-3-yl)thieno[2,3-*c*]quinoline(4g)



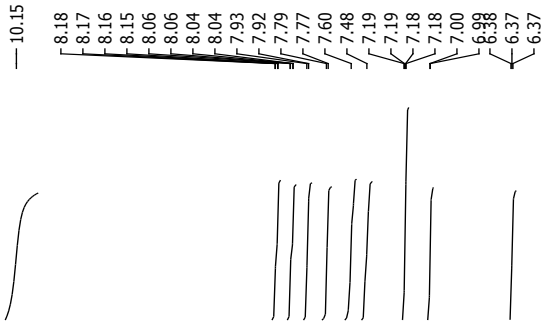
^{1}H NMR (400 MHz, DMSO-d$_6$): 4-(1*H*-imidazol-2-yl)thieno[2,3-*c*]quinoline(4h)

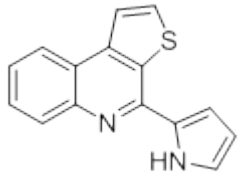


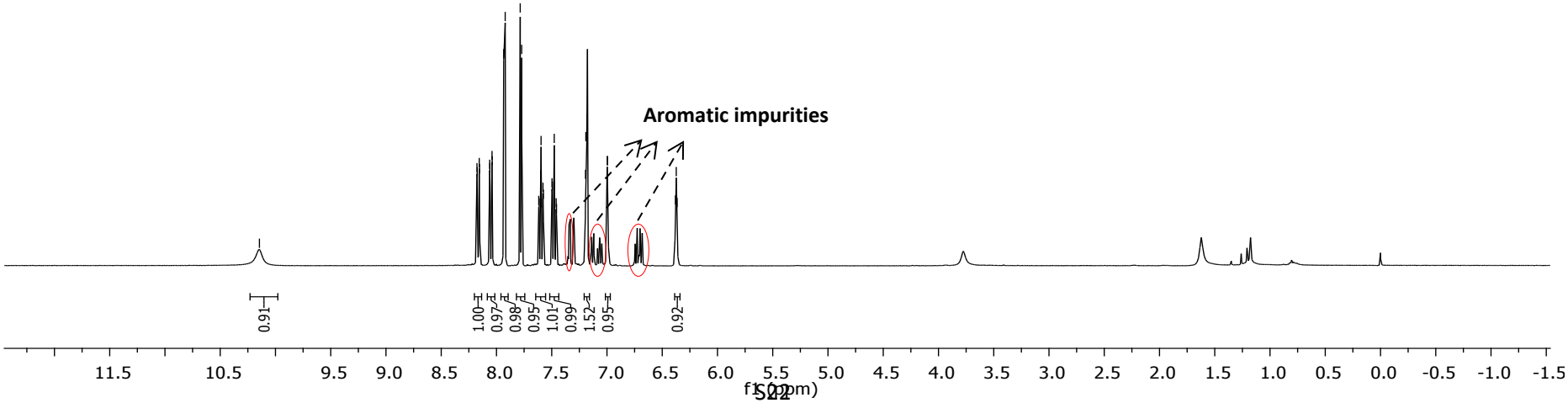
^{13}C NMR (101 MHz, DMSO-d_6): 4-(1*H*-imidazol-2-yl)thieno[2,3-*c*]quinoline(4h)



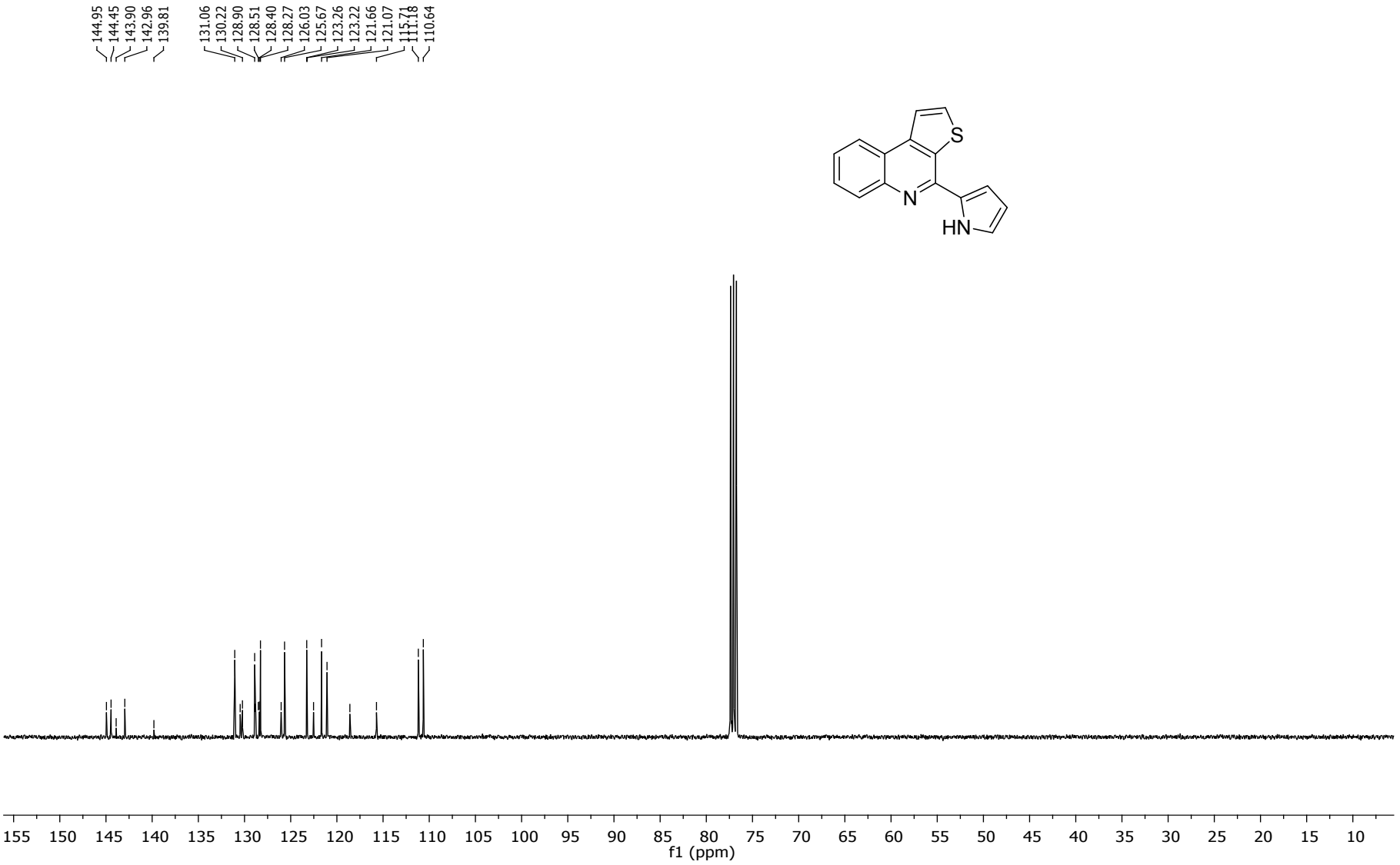
^{1}H NMR (300 MHz, $CDCl_3$): 4-(1*H*-pyrrol-2-yl)thieno[2,3-*c*]quinoline(4i)



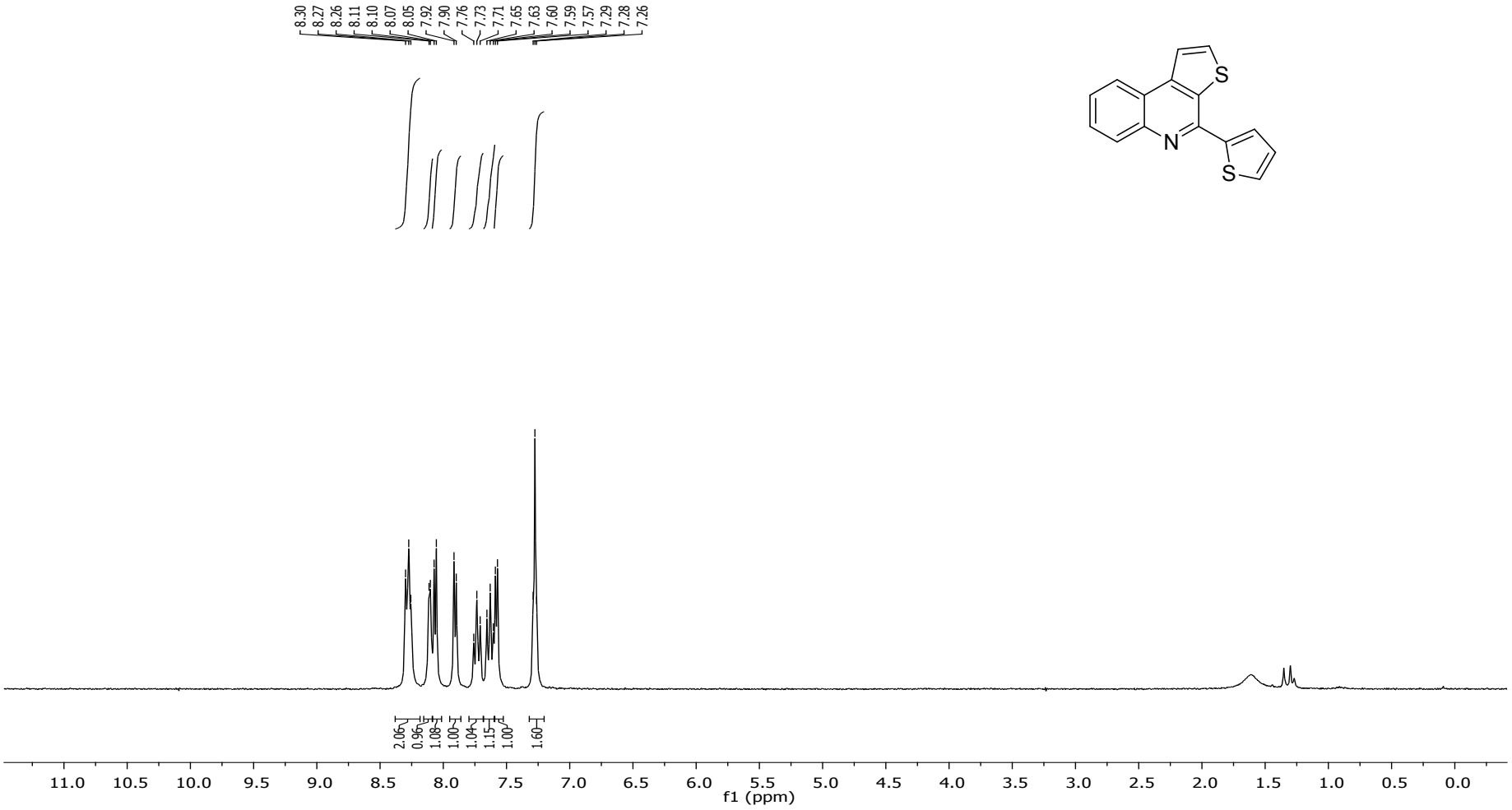




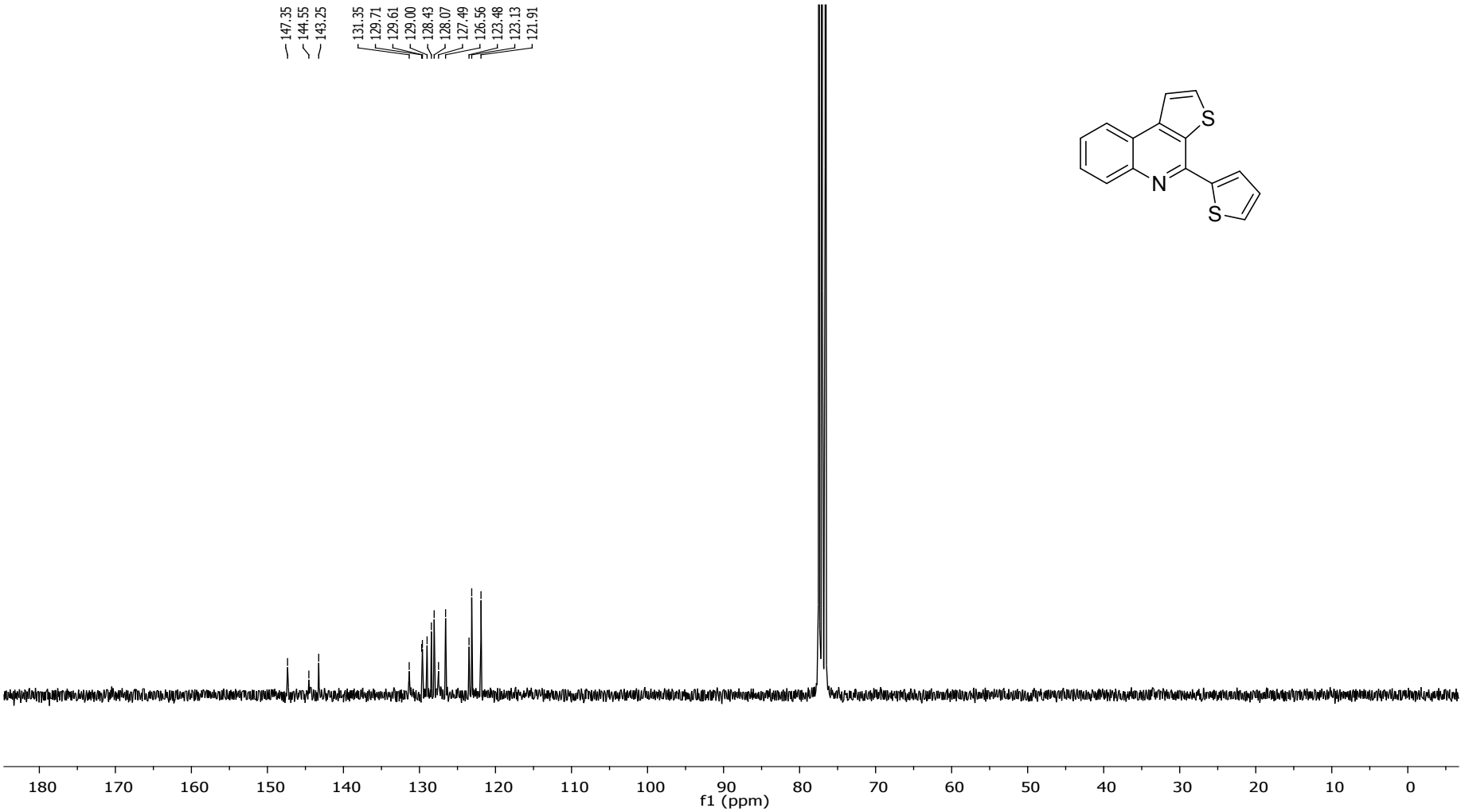
^{13}C NMR (75 MHz, $CDCl_3$): 4-(1*H*-pyrrol-2-yl)thieno[2,3-*c*]quinoline(4i)



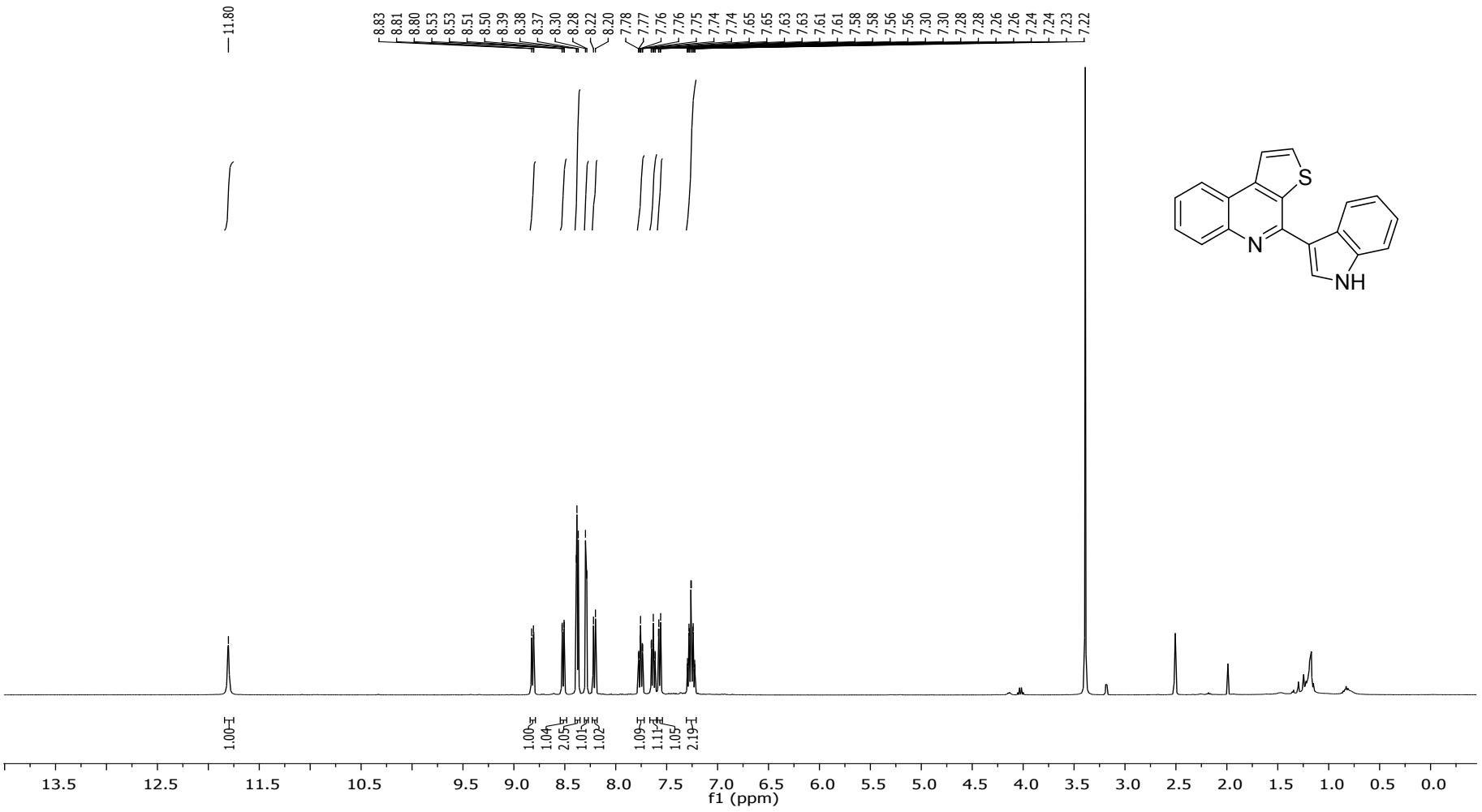
^{1}H NMR (300 MHz, $CDCl_3$): 4-(thiophen-2-yl)thieno[2,3-*c*]quinoline(4j)



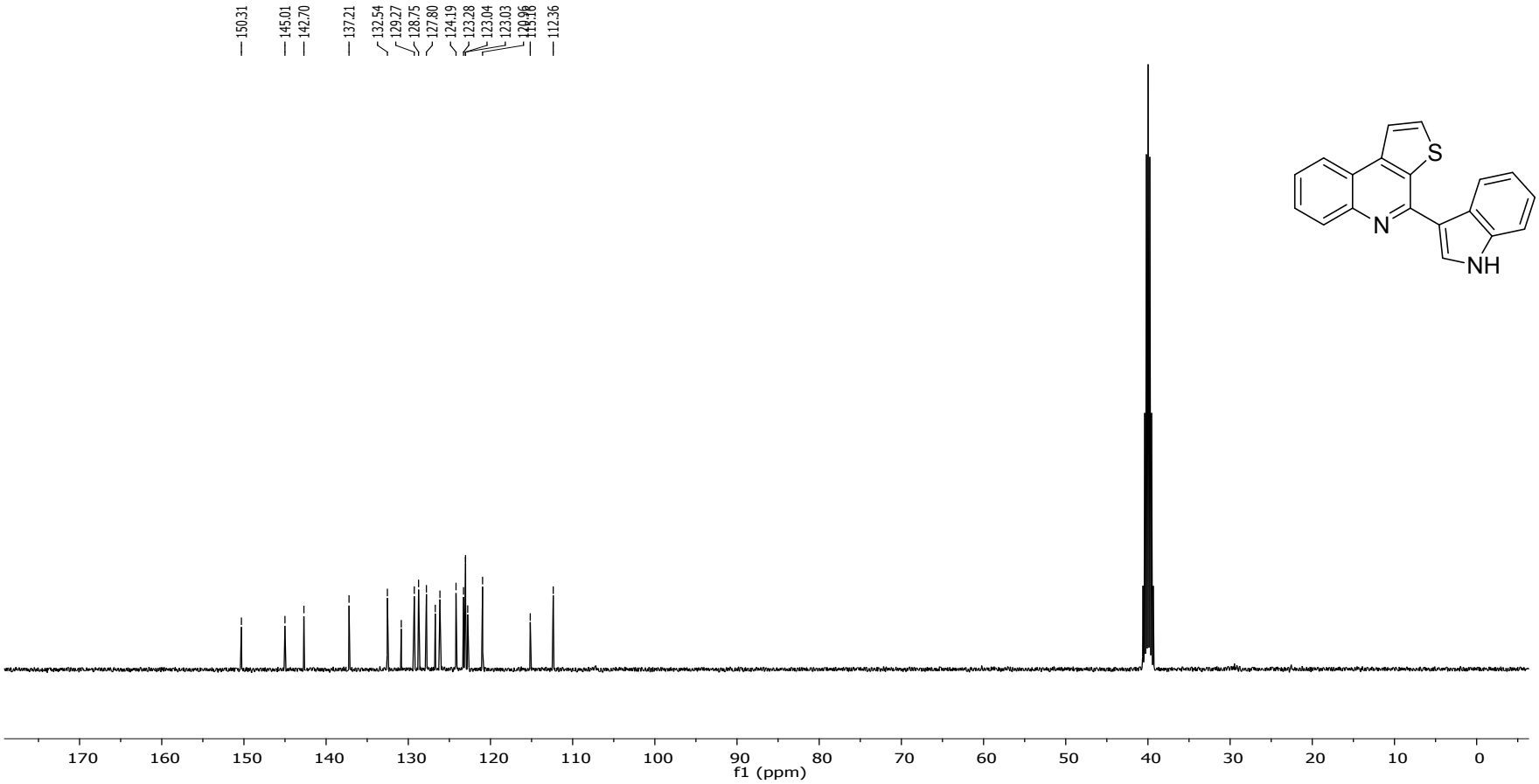
^{13}C NMR (75 MHz, $CDCl_3$): 4-(thiophen-2-yl)thieno[2,3-*c*]quinoline(4j)



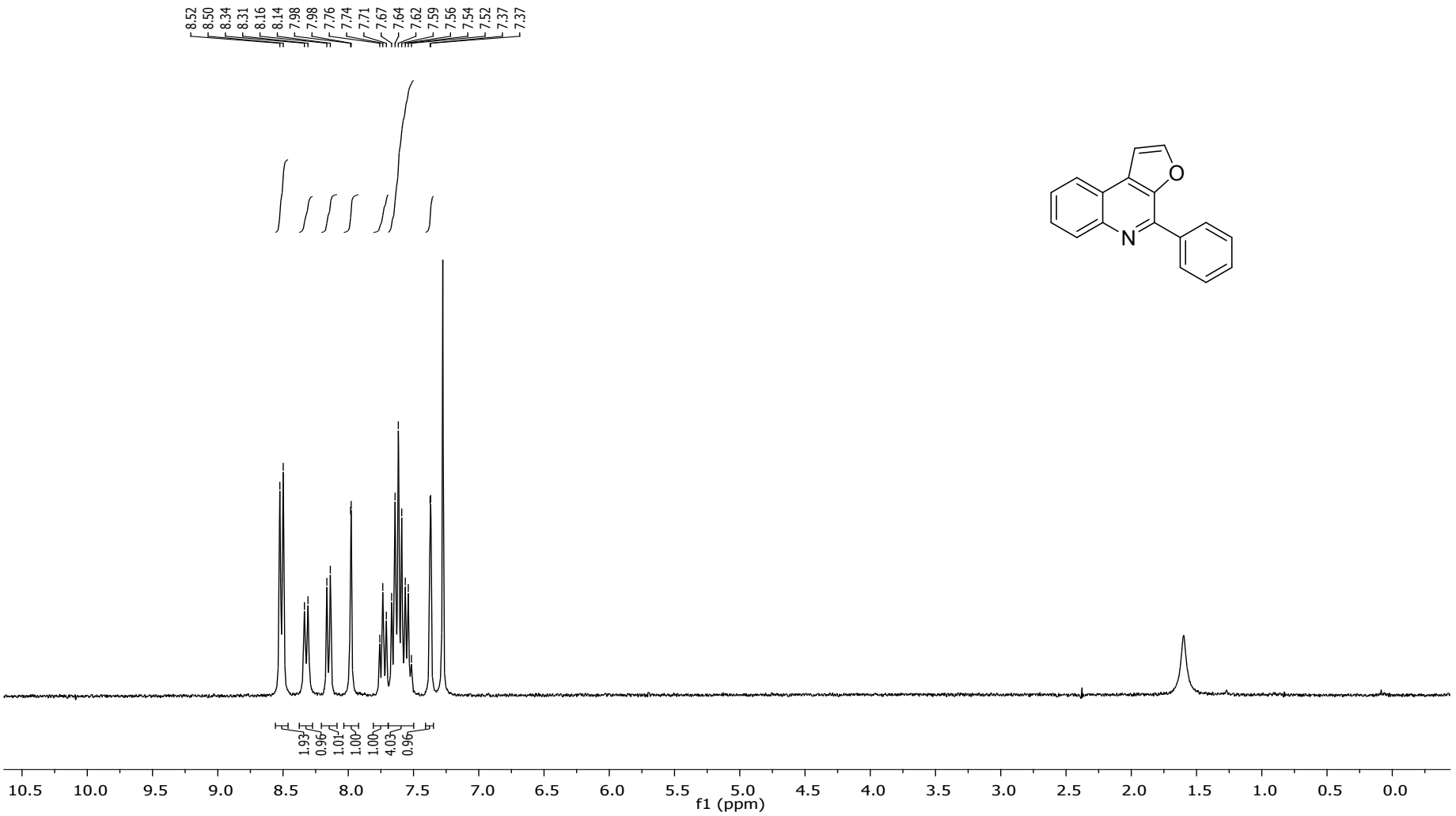
^{1}H NMR (400 MHz, DMSO-d$_6$): 4-(1*H*-indol-3-yl)thieno[2,3-*c*]quinoline(4k)



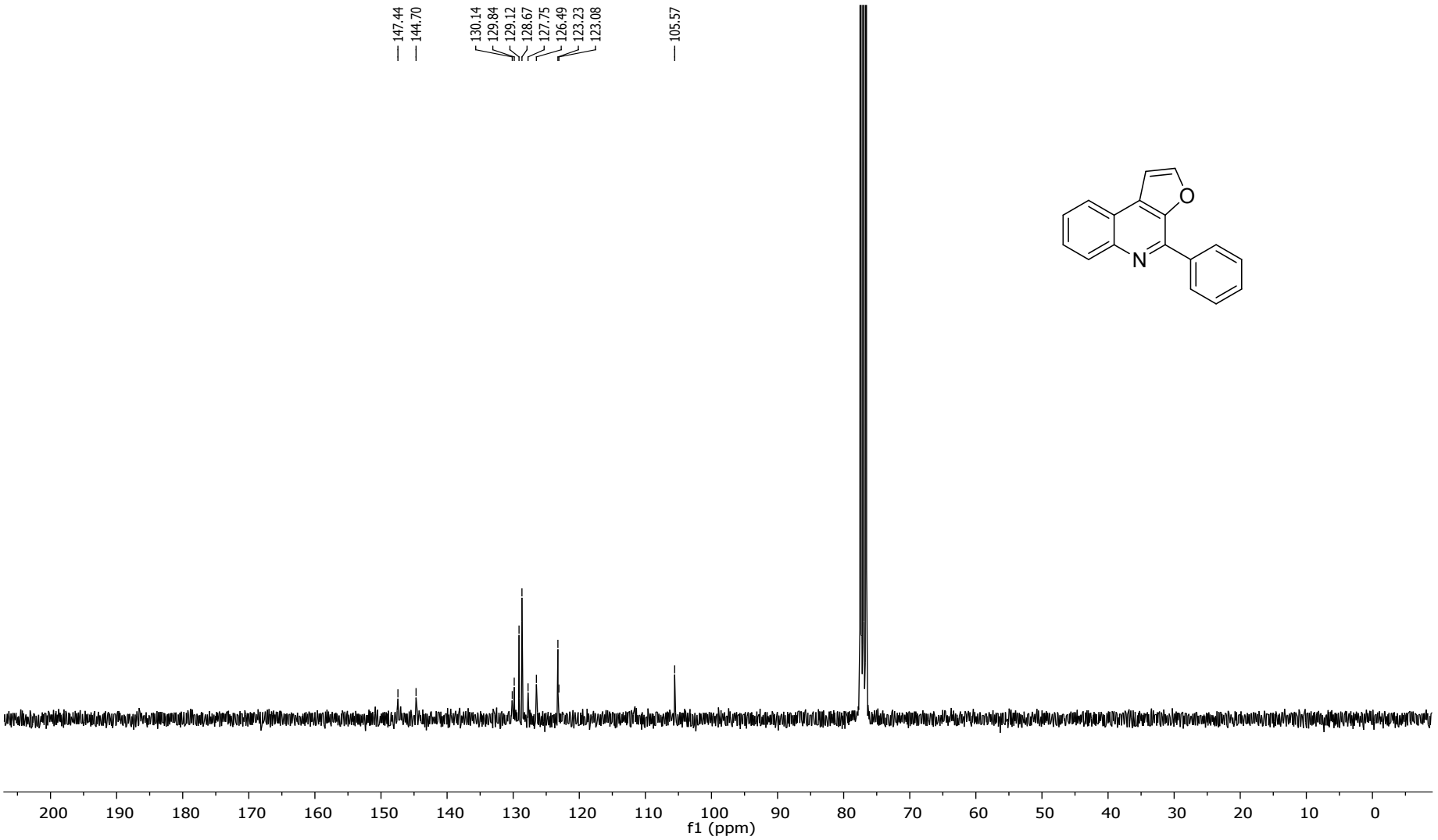
^{13}C NMR (101 MHz, DMSO-d$_6$): 4-(1*H*-indol-3-yl)thieno[2,3-*c*]quinoline(4k)



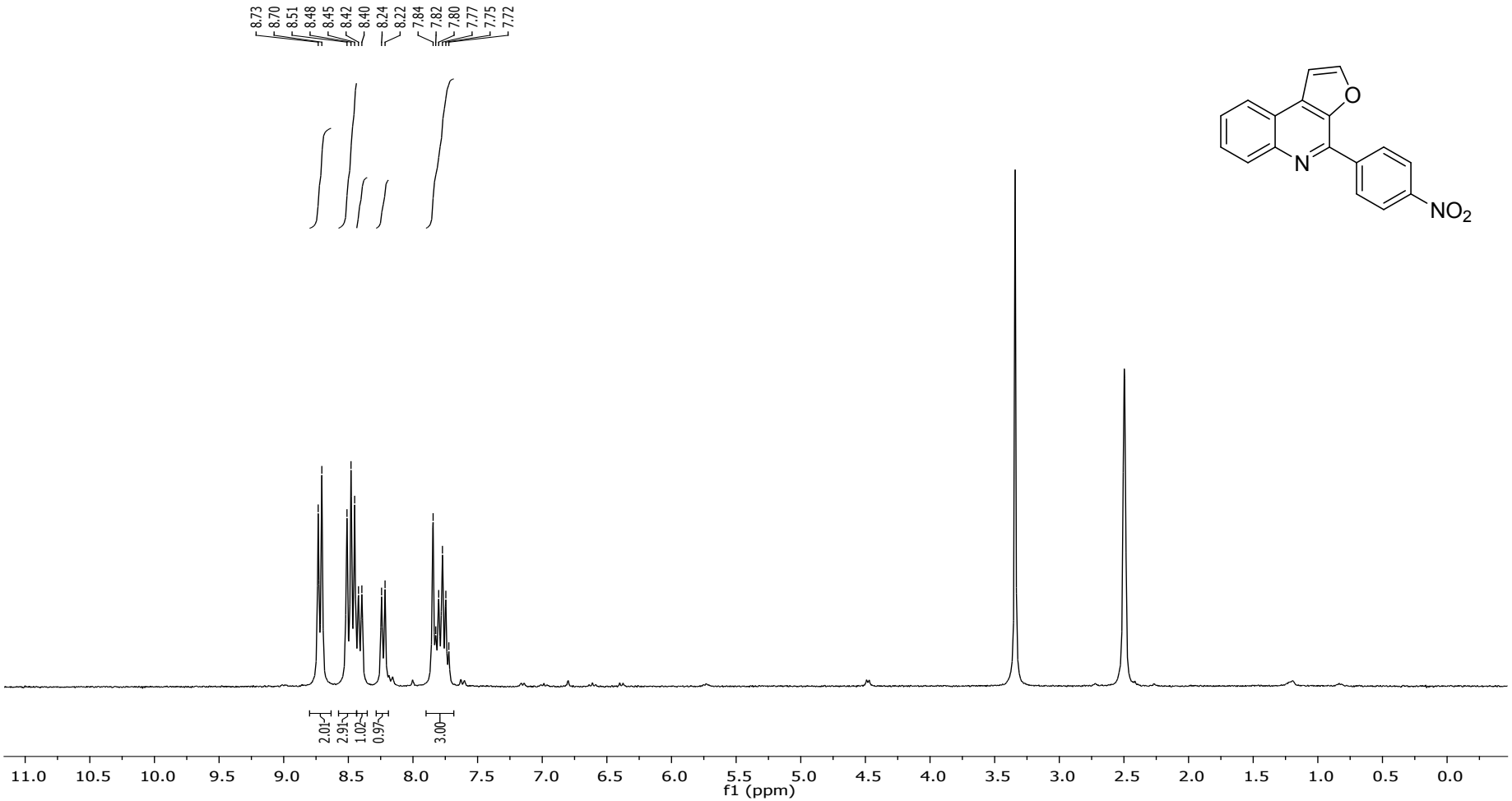
^{1}H NMR (300 MHz, $CDCl_3$):4-phenylfuro[2,3-*c*]quinoline(5a)



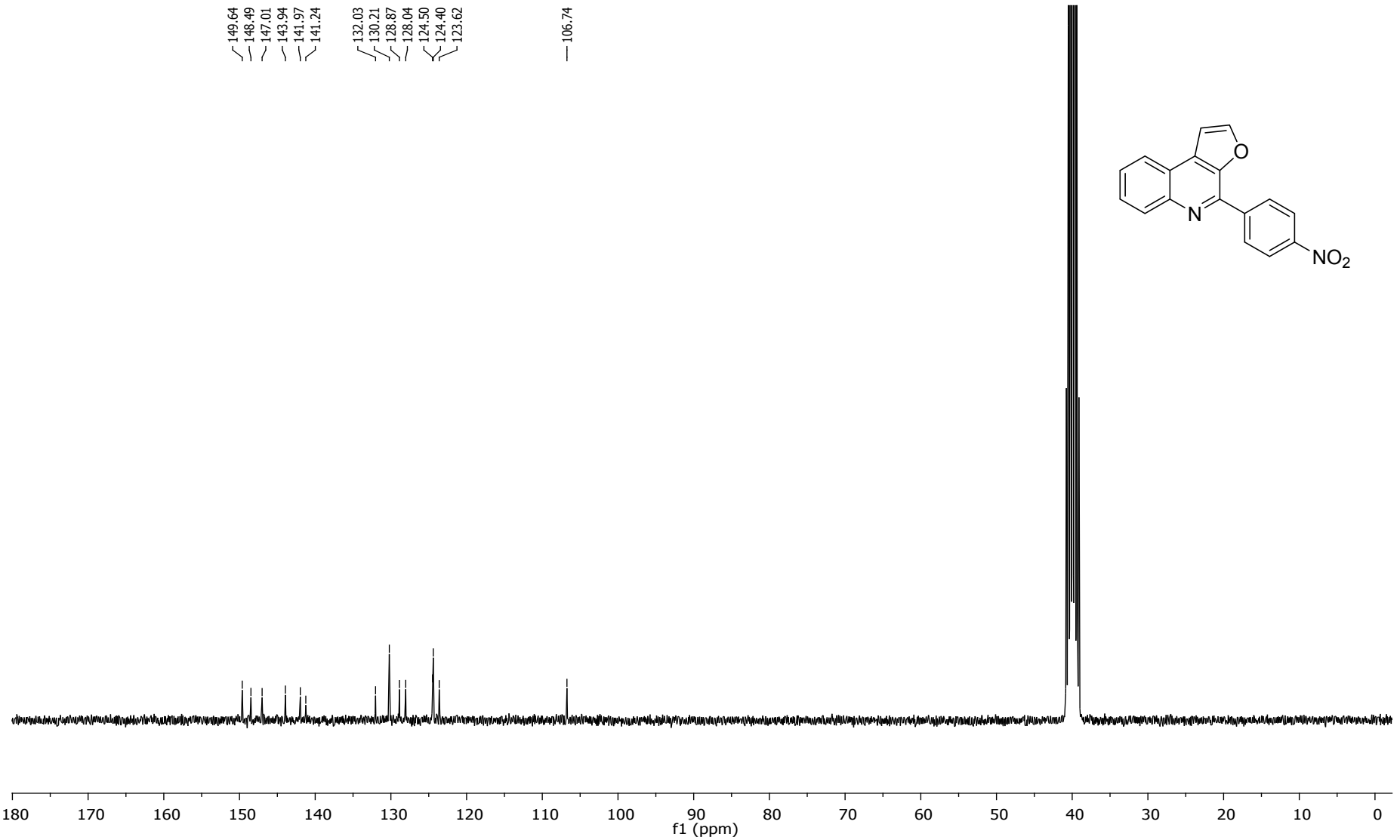
^{13}C NMR (75 MHz, $CDCl_3$): 4-phenylfuro[2,3-*c*]quinoline(5a)



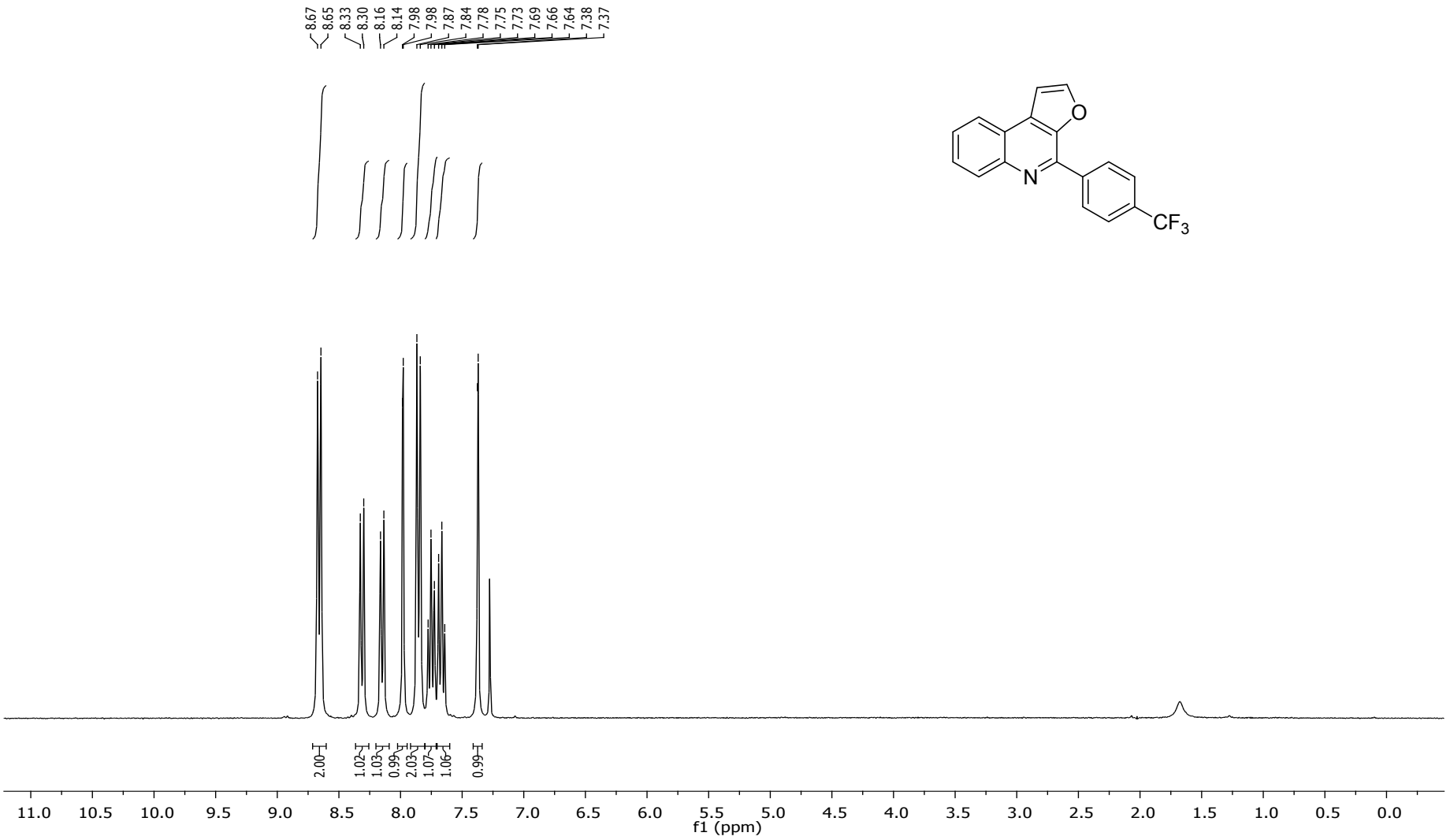
^{1}H NMR (300 MHz, DMSO-d$_6$): 4-(4-nitrophenyl)furo[2,3-*c*]quinoline(5b)



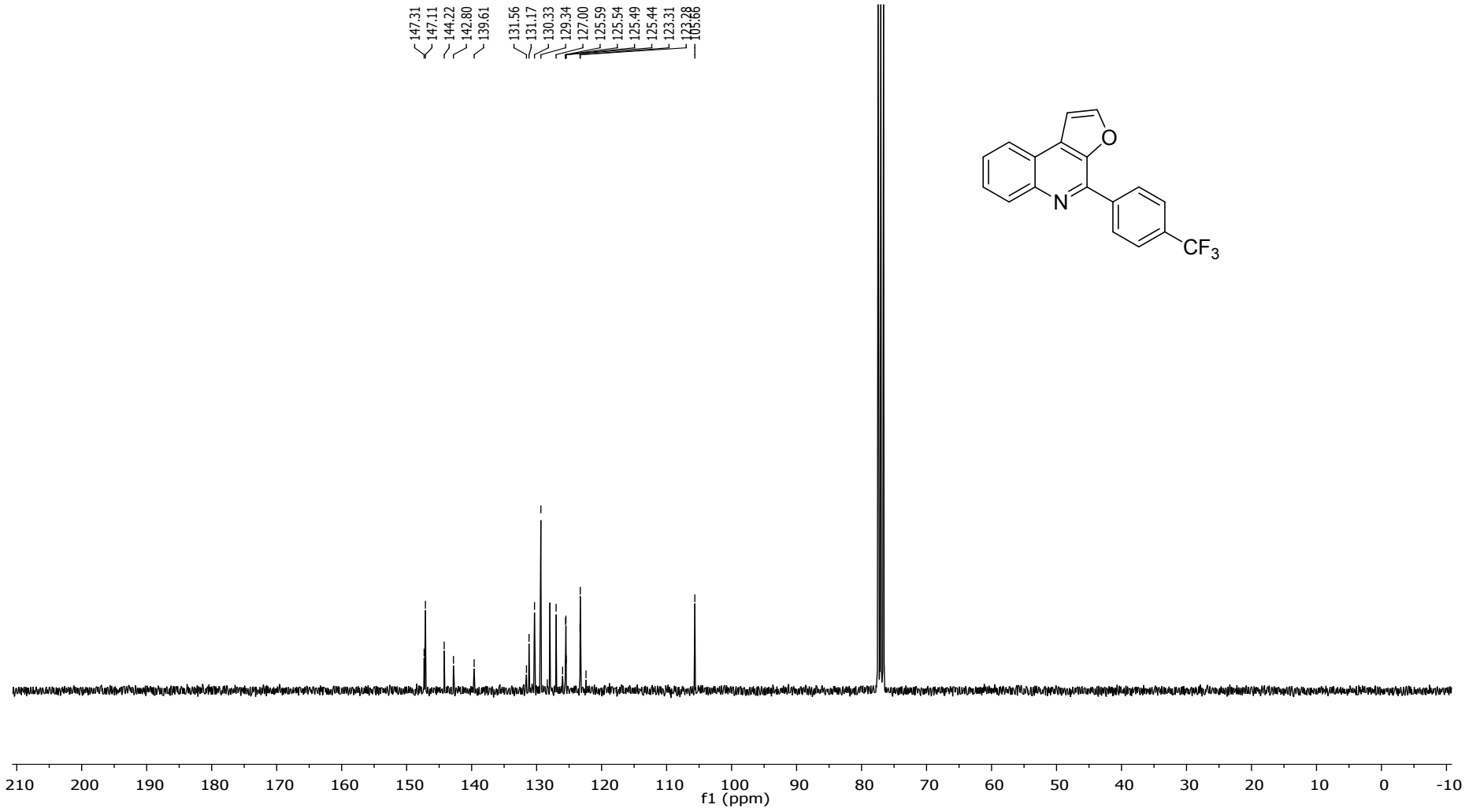
^{13}C NMR (75 MHz, DMSO-d$_6$): 4-(4-nitrophenyl)furo[2,3-*c*]quinoline(5b)



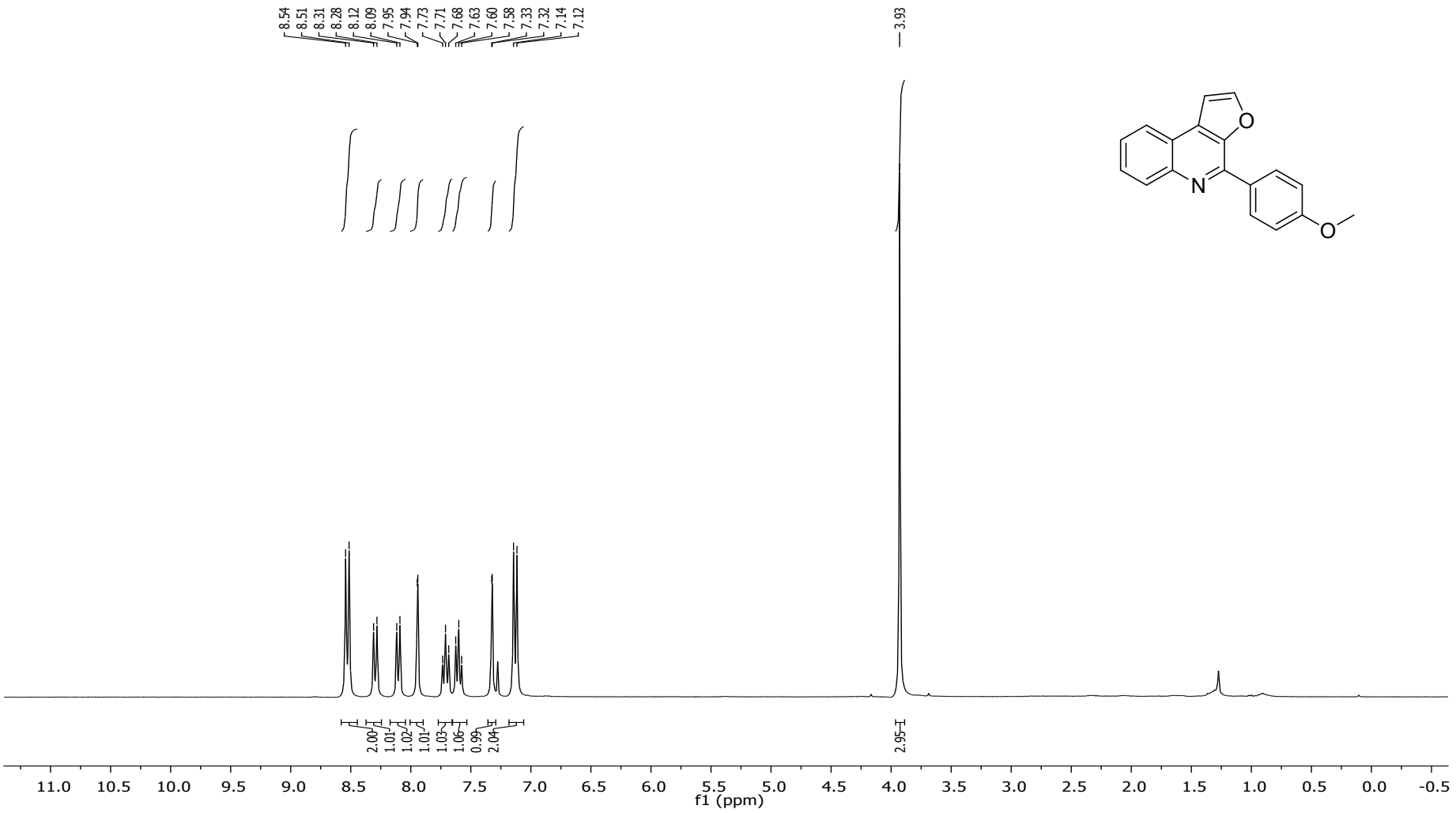
^{1}H NMR (300 MHz, $CDCl_3$): 4-(4-(trifluoromethyl)phenyl)furo[2,3-*c*]quinoline(5c)



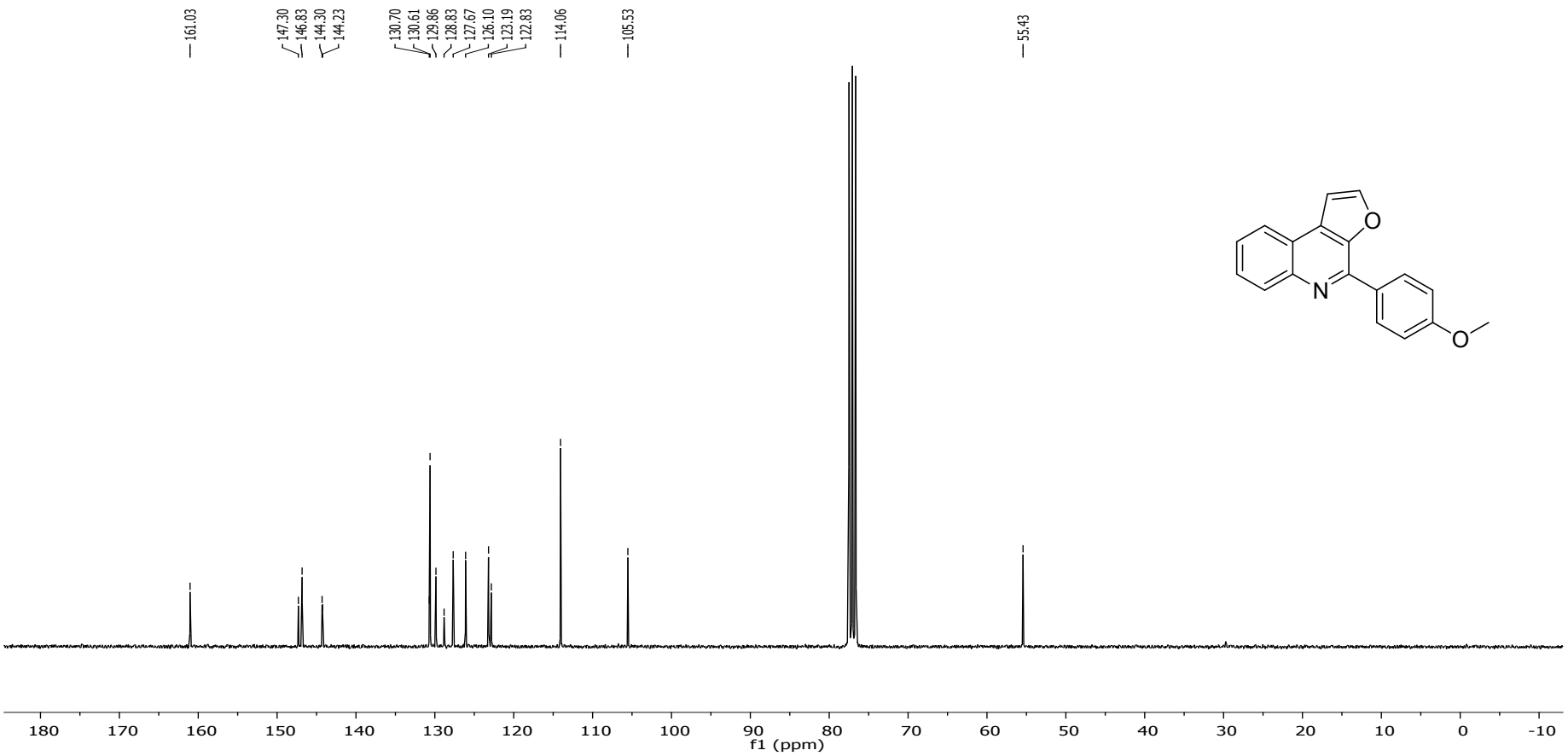
^{13}C NMR (75 MHz, $CDCl_3$): 4-(4-(trifluoromethyl)phenyl)furo[2,3-*c*]quinoline(5c)



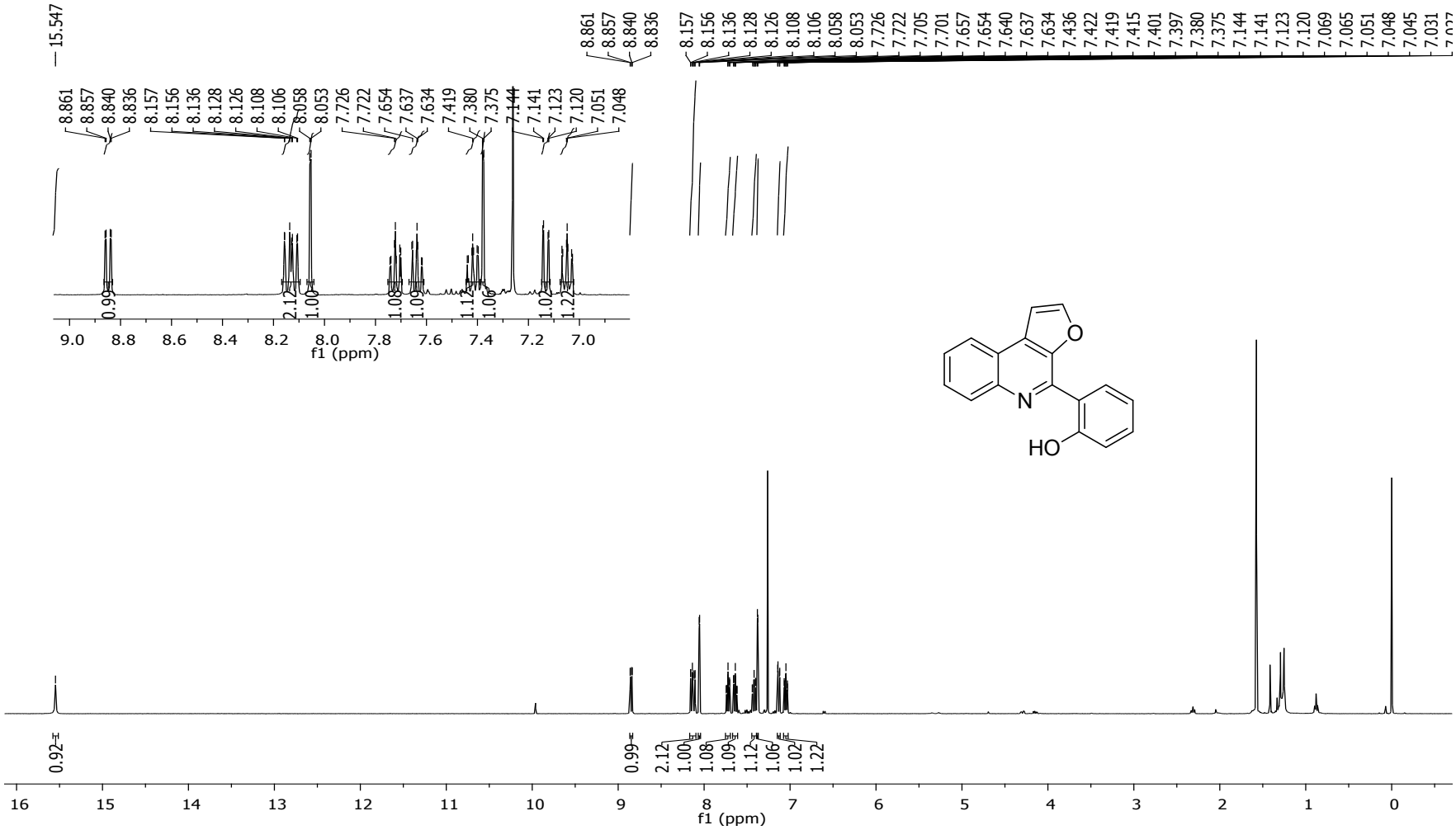
^{1}H NMR (300 MHz, $CDCl_3$): 4-(4-methoxyphenyl)furo[2,3-*c*]quinoline(5d)



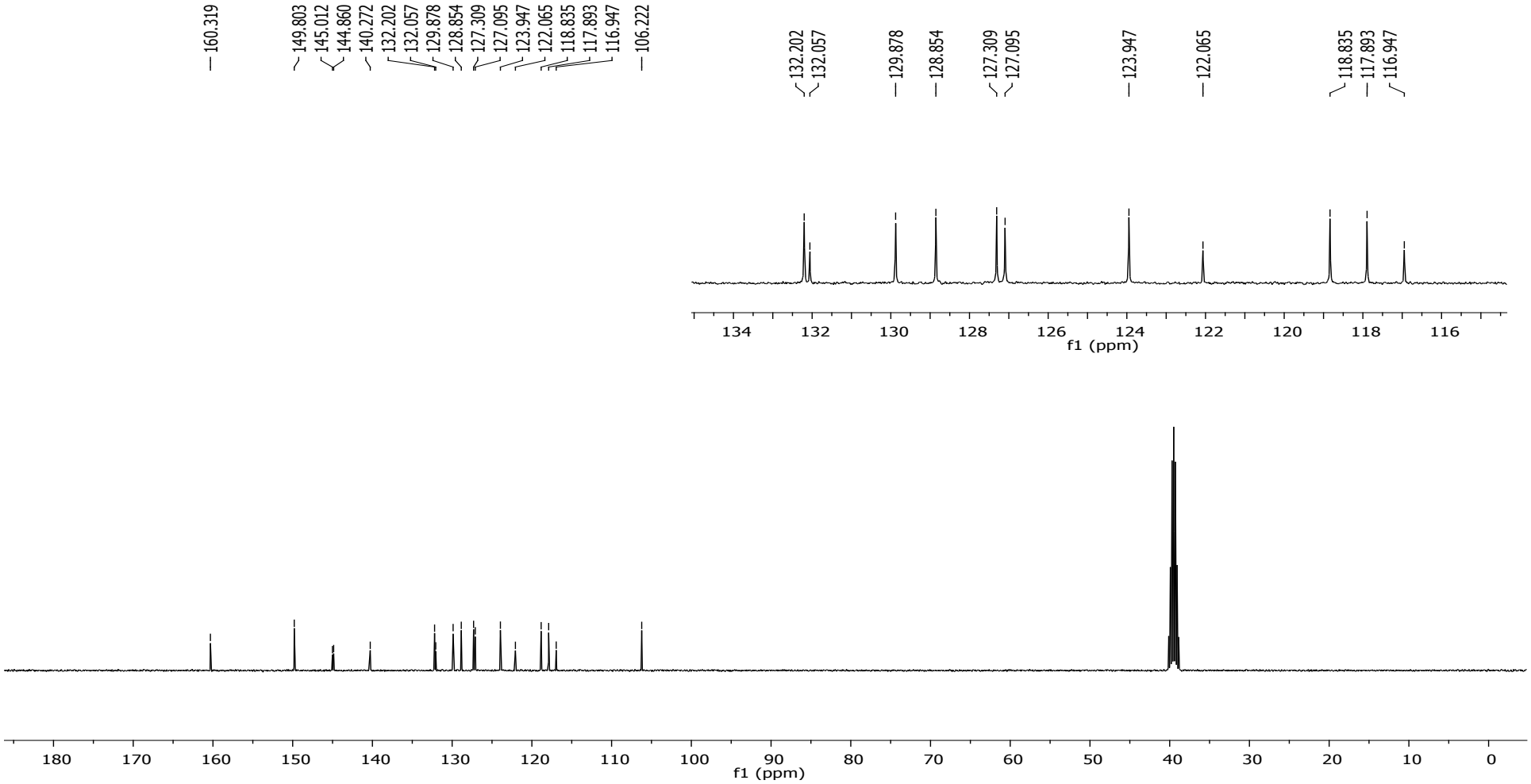
^{13}C NMR (75 MHz, $CDCl_3$): 4-(4-methoxyphenyl)furo[2,3-*c*]quinoline(5d)



^{1}H NMR (400 MHz, $CDCl_3$): 2-(furo[2,3-*c*]quinolin-4-yl)phenol(5e)



^{13}C NMR (101 MHz, $CDCl_3$): 2-(furo[2,3-*c*]quinolin-4-yl)phenol(5e)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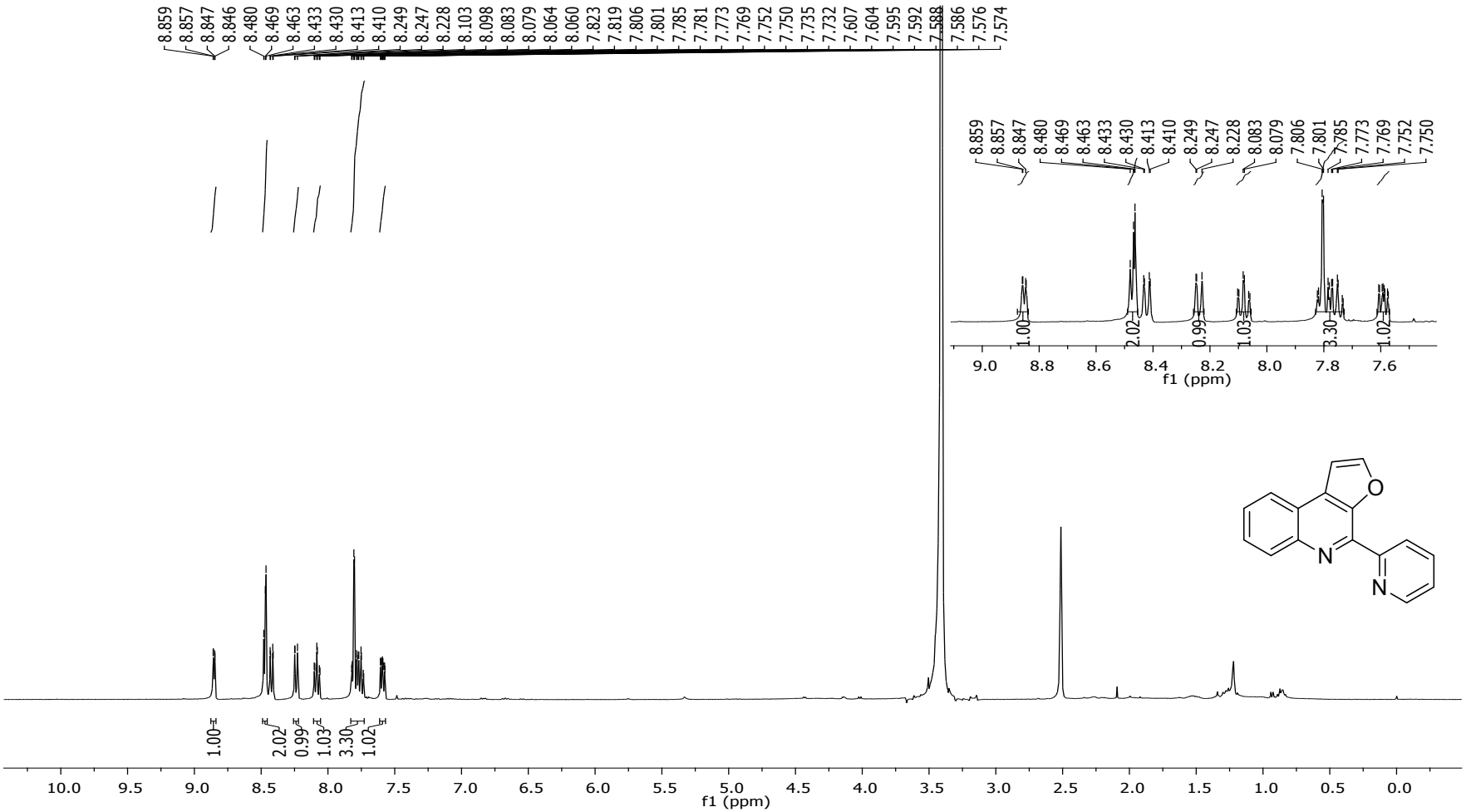
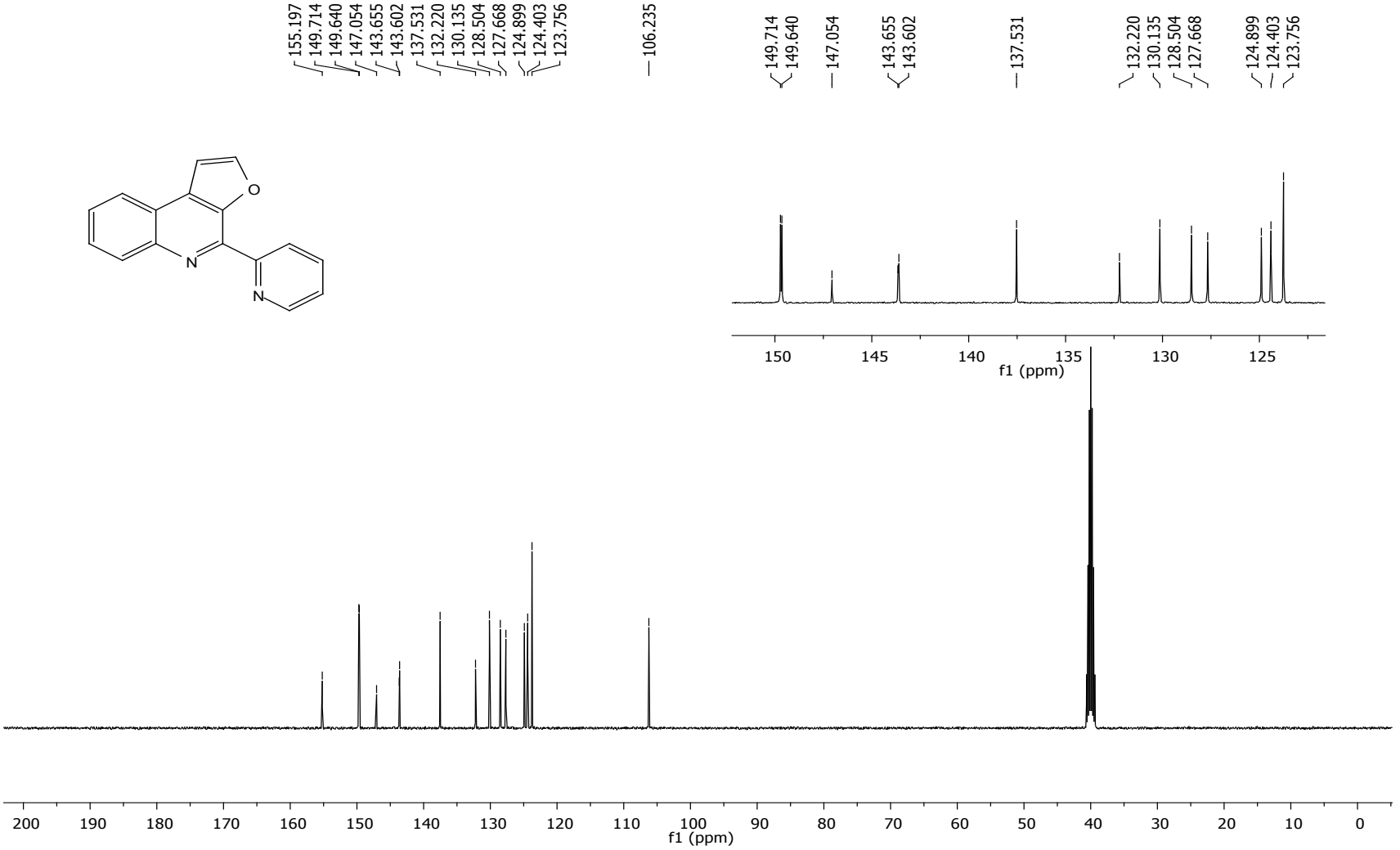
149.803
145.012
144.860
140.272
132.202
132.057
129.878
128.854
127.309
127.095
123.947
122.065
118.835
117.893
116.947
106.222
f1 (ppm)
f1 (ppm)

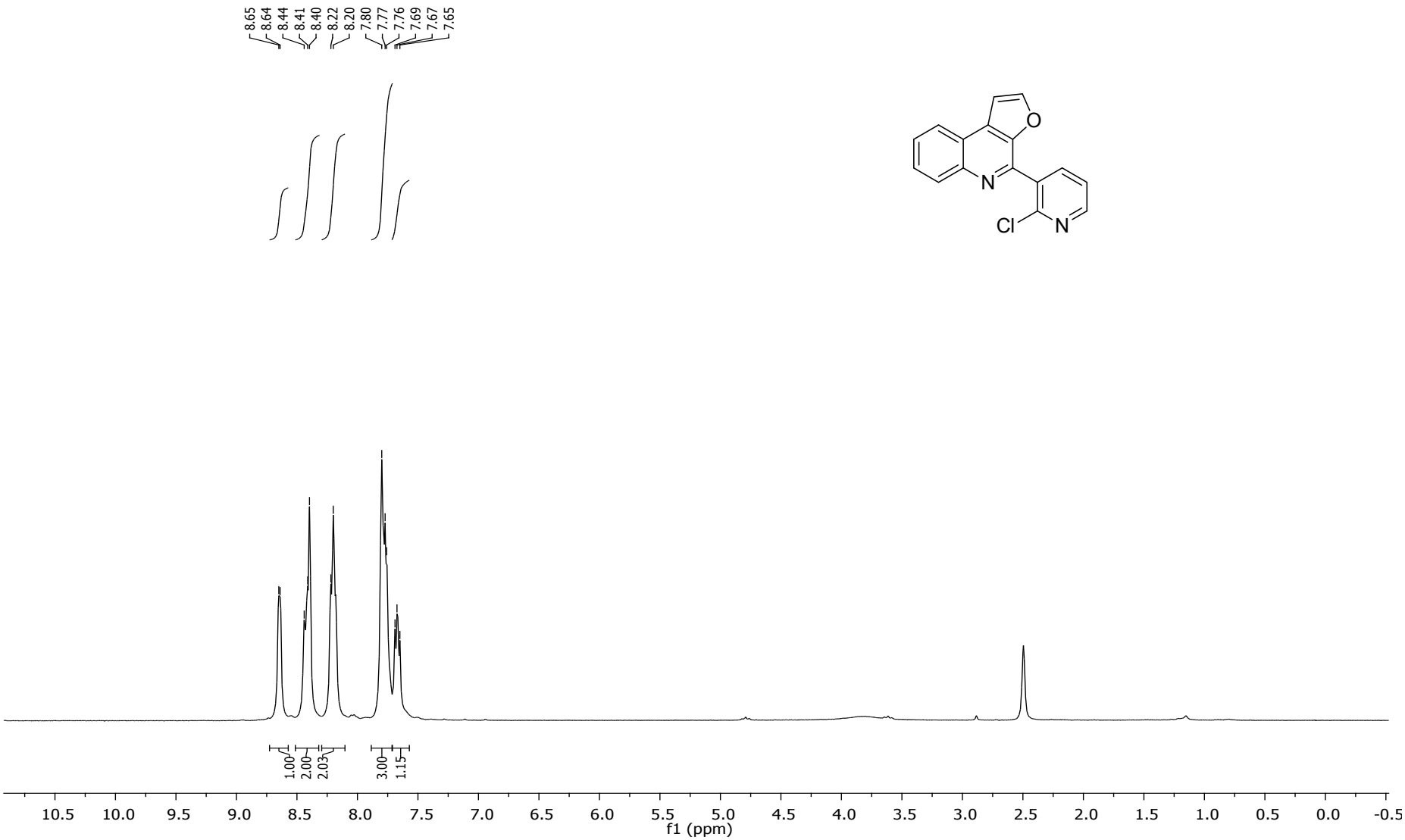
^{1}H NMR (400 MHz, DMSO-d_6): 4-(pyridin-2-yl)furo[2,3-*c*]quinoline(5f)



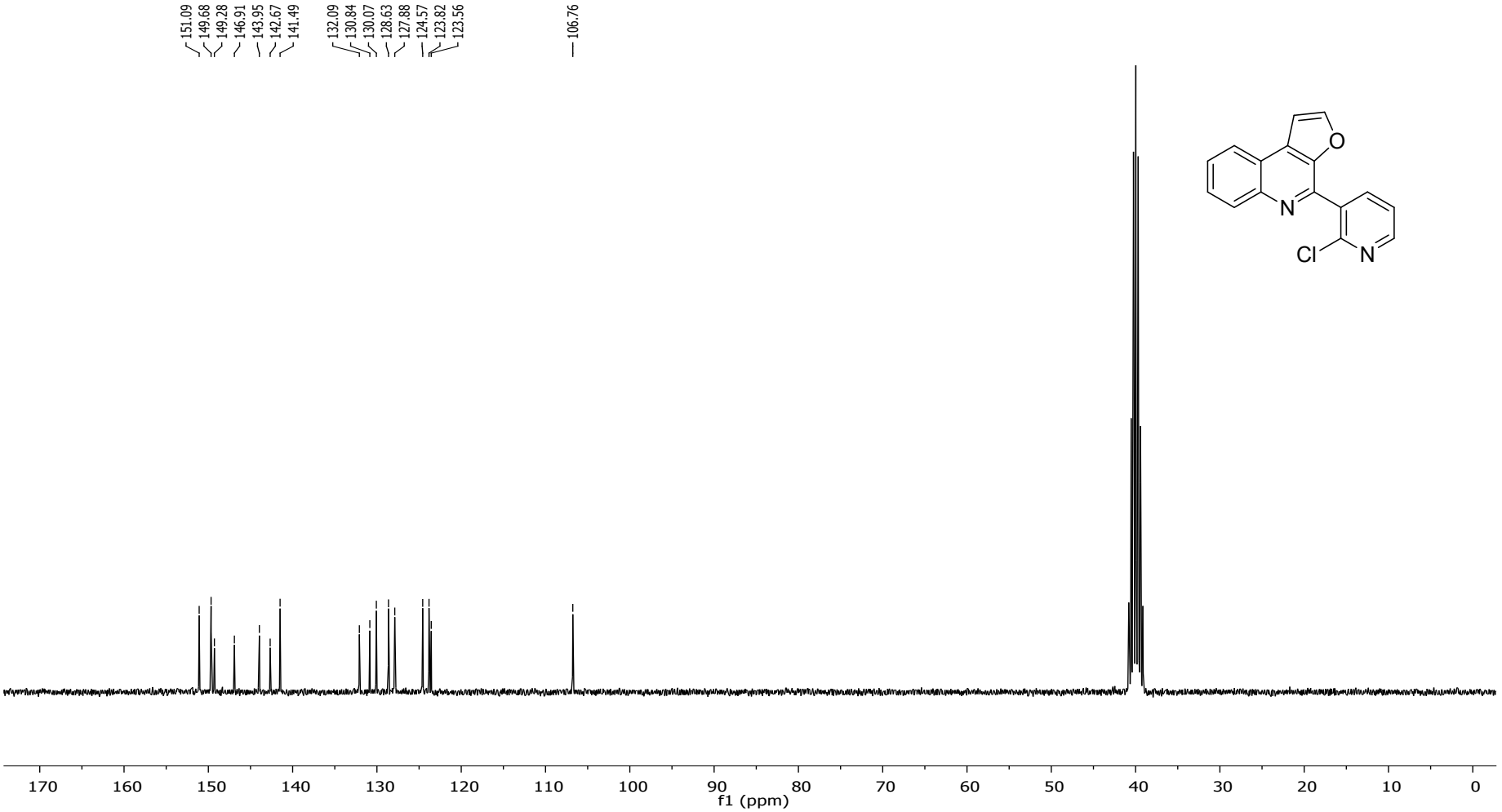
^{13}C NMR (101 MHz, DMSO-d_6): 4-(pyridin-2-yl)furo[2,3-*c*]quinoline(5f)



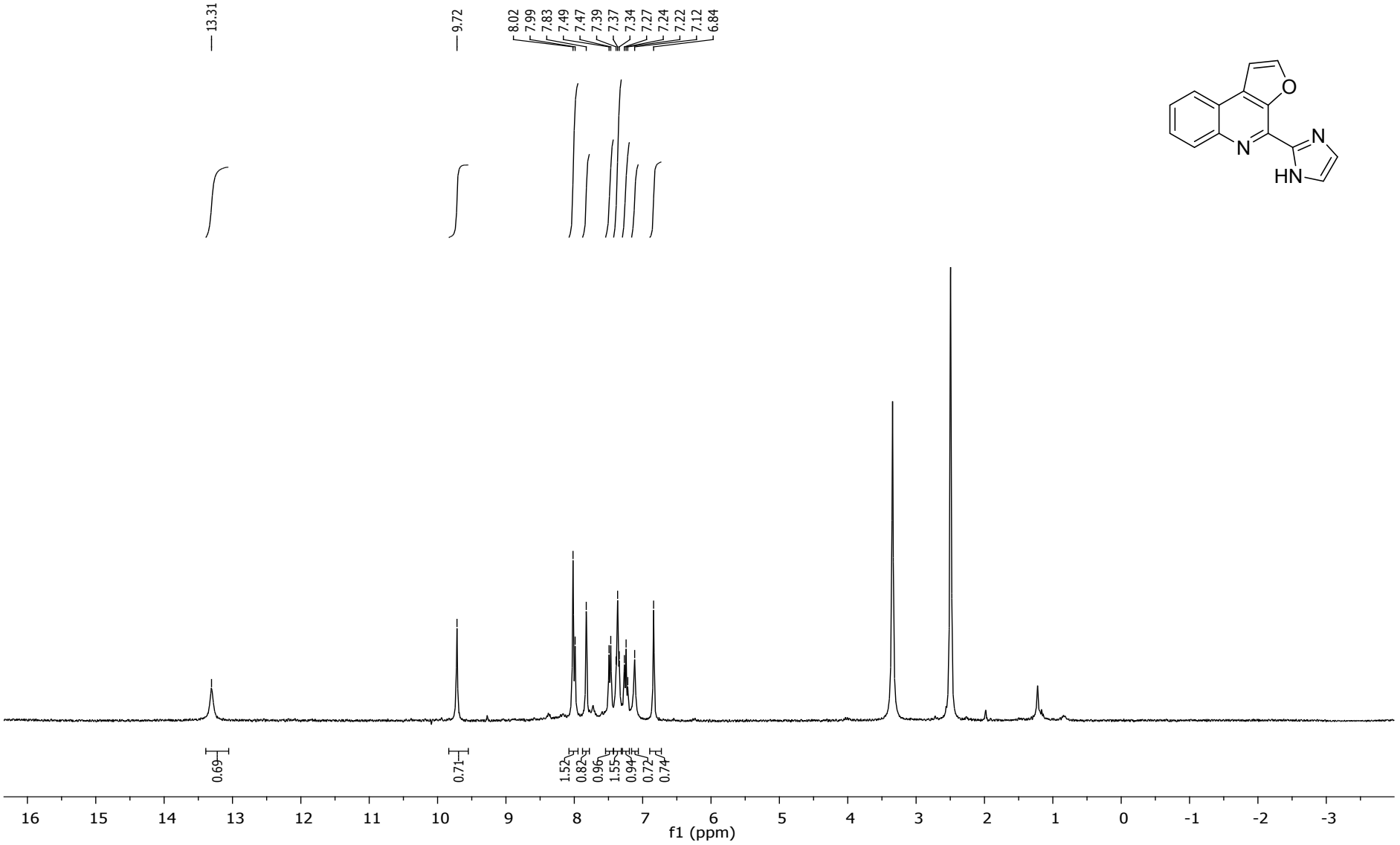
^{1}H NMR (300 MHz, DMSO-d_6): 4-(2-chloropyridin-3-yl)furo[2,3-*c*]quinoline(5g)



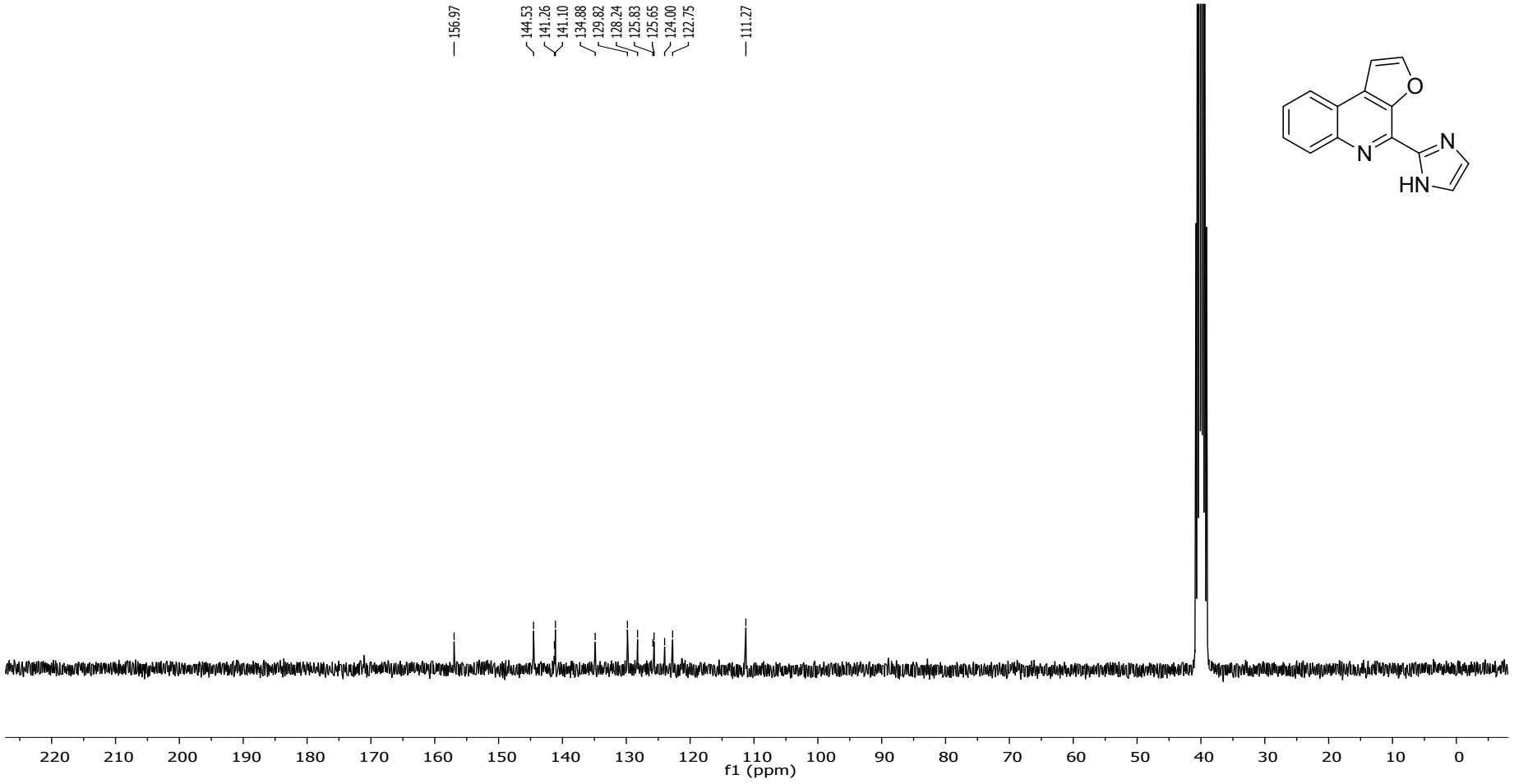
^{13}C NMR (75 MHz, DMSO-d_6): 4-(2-chloropyridin-3-yl)furo[2,3-*c*]quinoline(5g)



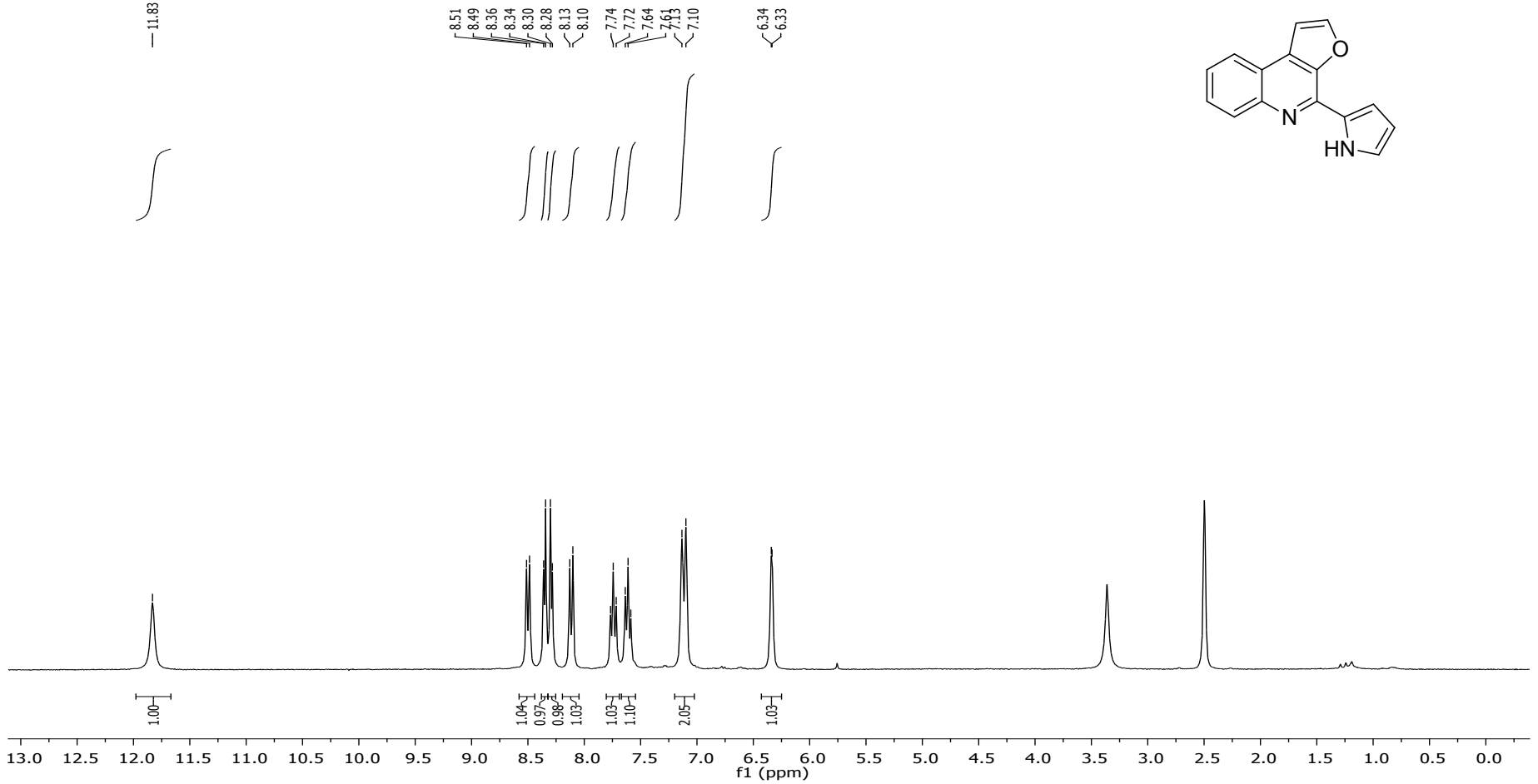
^{1}H NMR (300 MHz, DMSO-d_6): 4-(1*H*-imidazol-2-yl)furo[2,3-*c*]quinoline(5h)



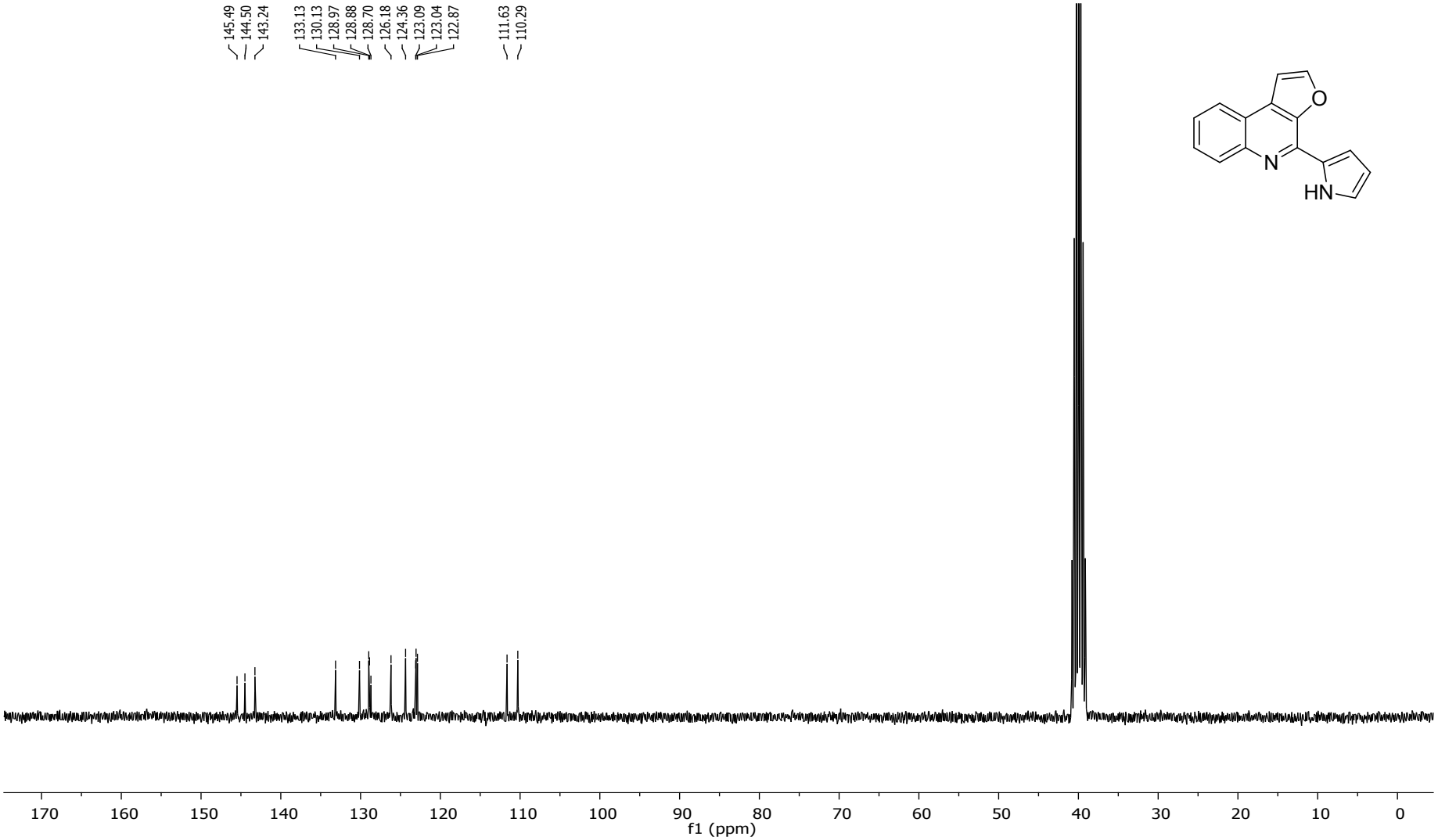
^{13}C NMR (75 MHz, DMSO-d_6): 4-(1*H*-imidazol-2-yl)furo[2,3-*c*]quinoline(5h)



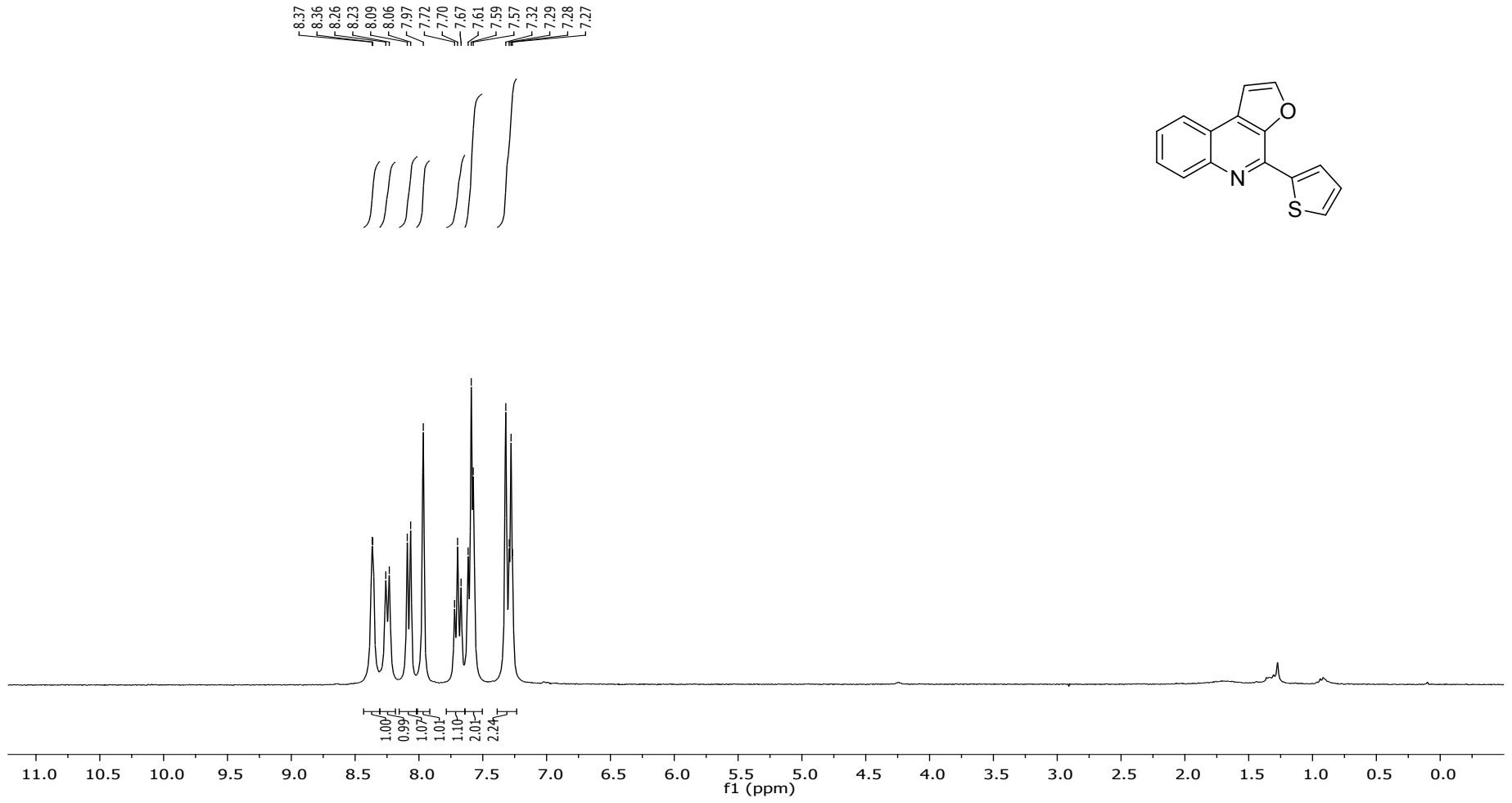
^{1}H NMR (300 MHz, DMSO-d$_6$): 4-(1*H*-pyrrol-2-yl)furo[2,3-*c*]quinoline(5i)



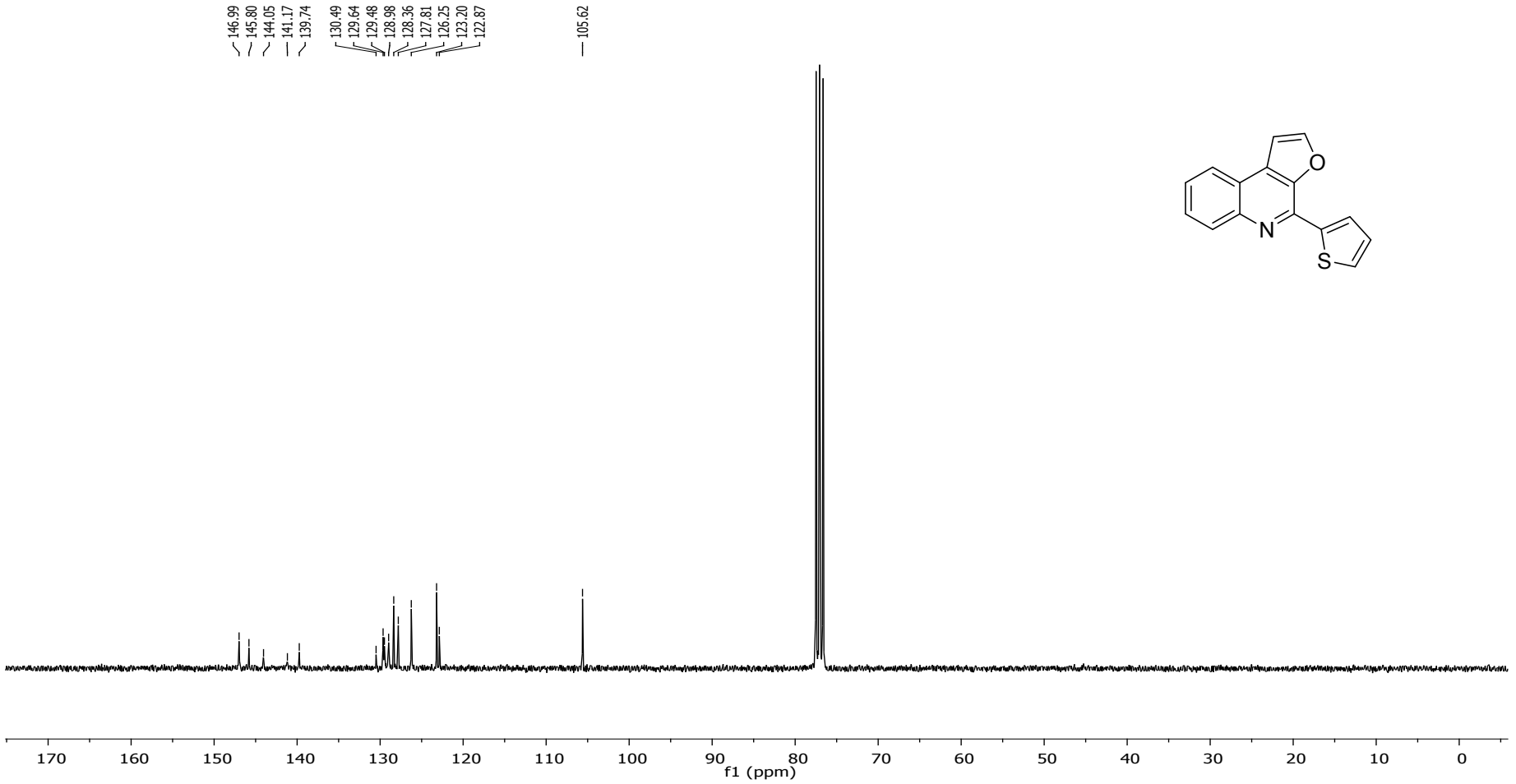
^{13}C NMR (75 MHz, DMSO-d_6): 4-(1*H*-pyrrol-2-yl)furo[2,3-*c*]quinoline(5i)



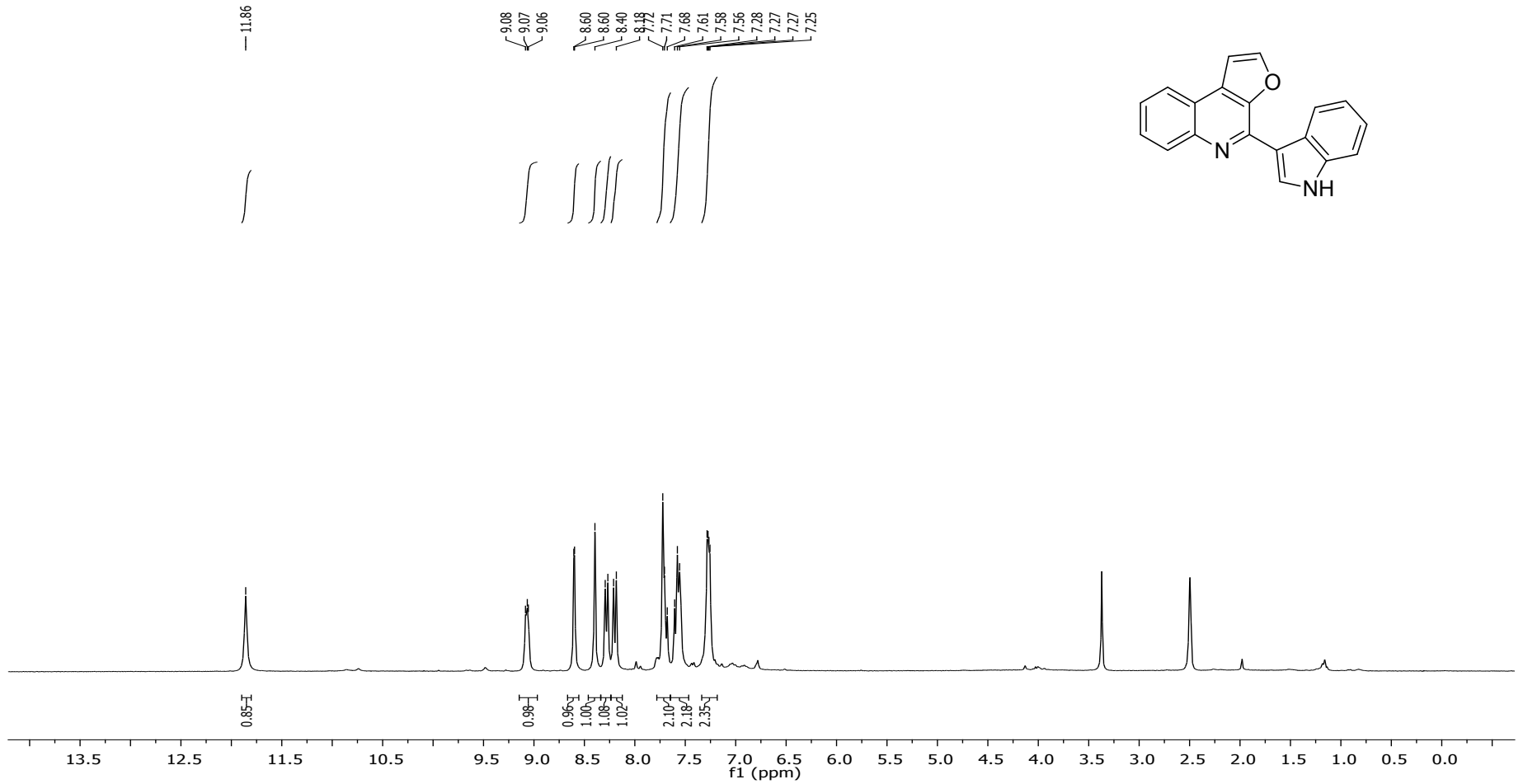
^{1}H NMR (300 MHz, $CDCl_3$): 4-(thiophen-2-yl)furo[2,3-*c*]quinoline(5j)



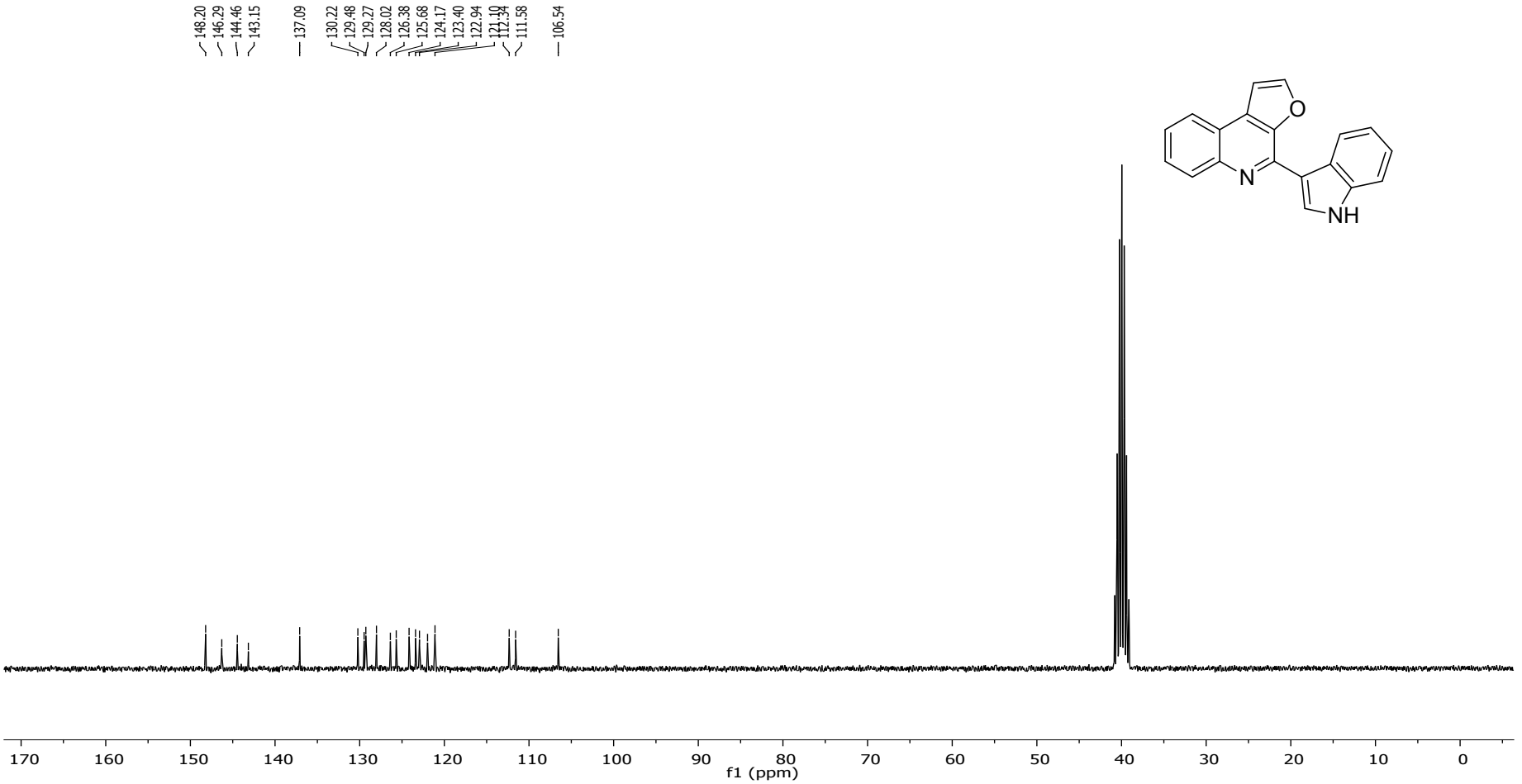
^{13}C NMR (75 MHz, $CDCl_3$): 4-(thiophen-2-yl)furo[2,3-*c*]quinoline(5j)



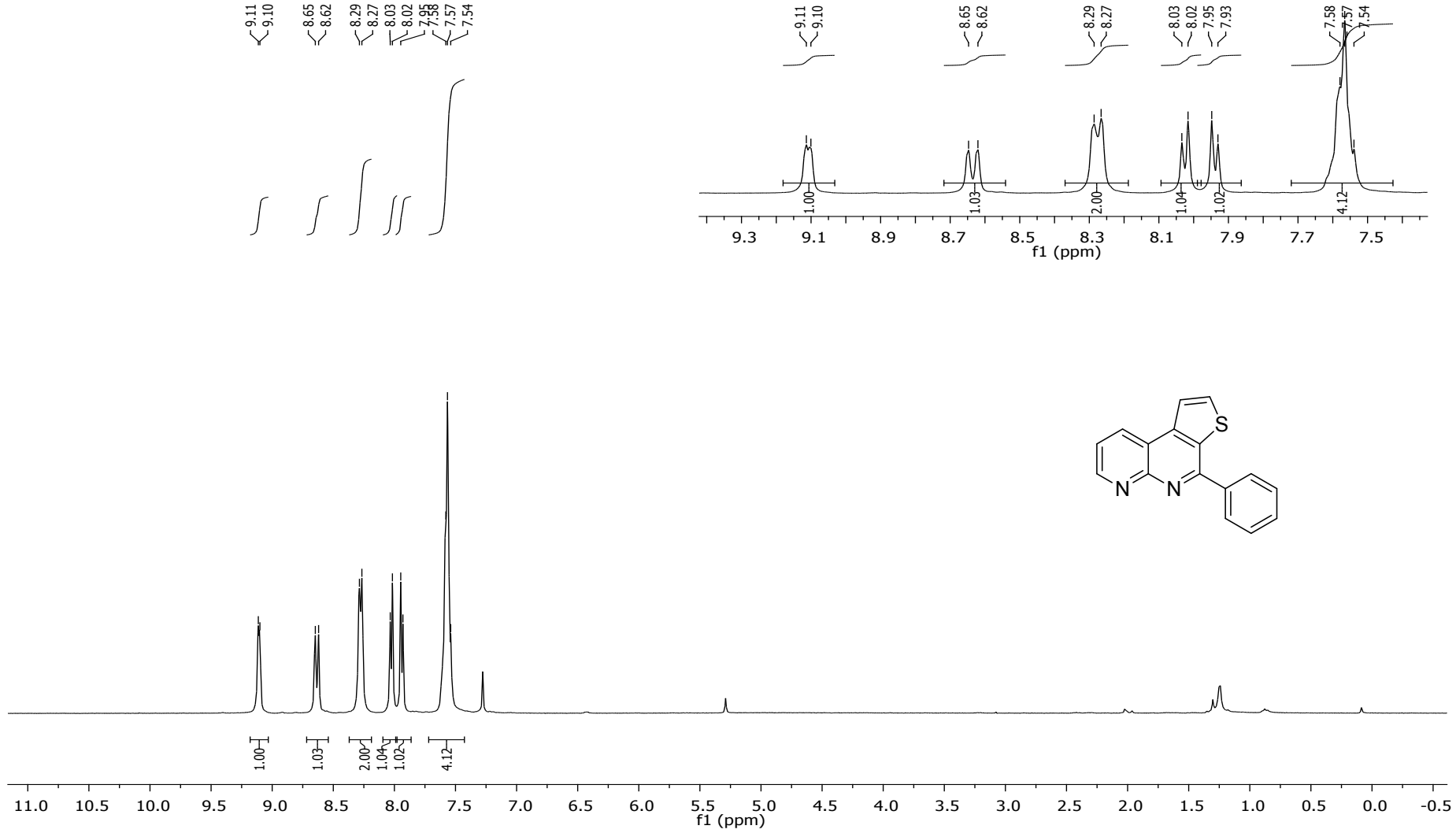
^{1}H NMR (300 MHz, DMSO-d$_6$): 4-(1*H*-indol-3-yl)furo[2,3-*c*]quinoline(5k)



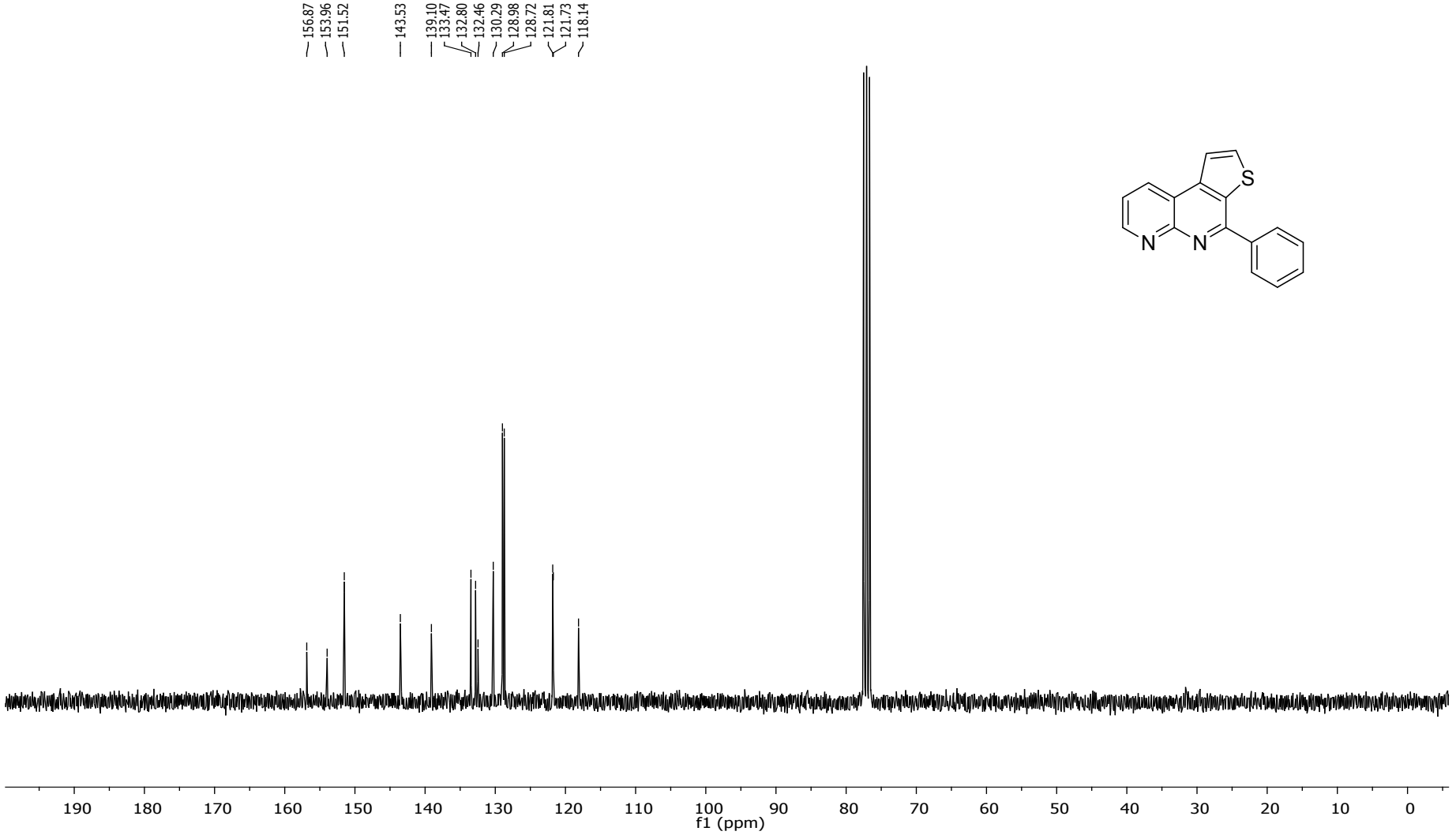
^{13}C NMR (75 MHz, DMSO-d_6): 4-(1*H*-indol-3-yl)furo[2,3-*c*]quinoline(5k)



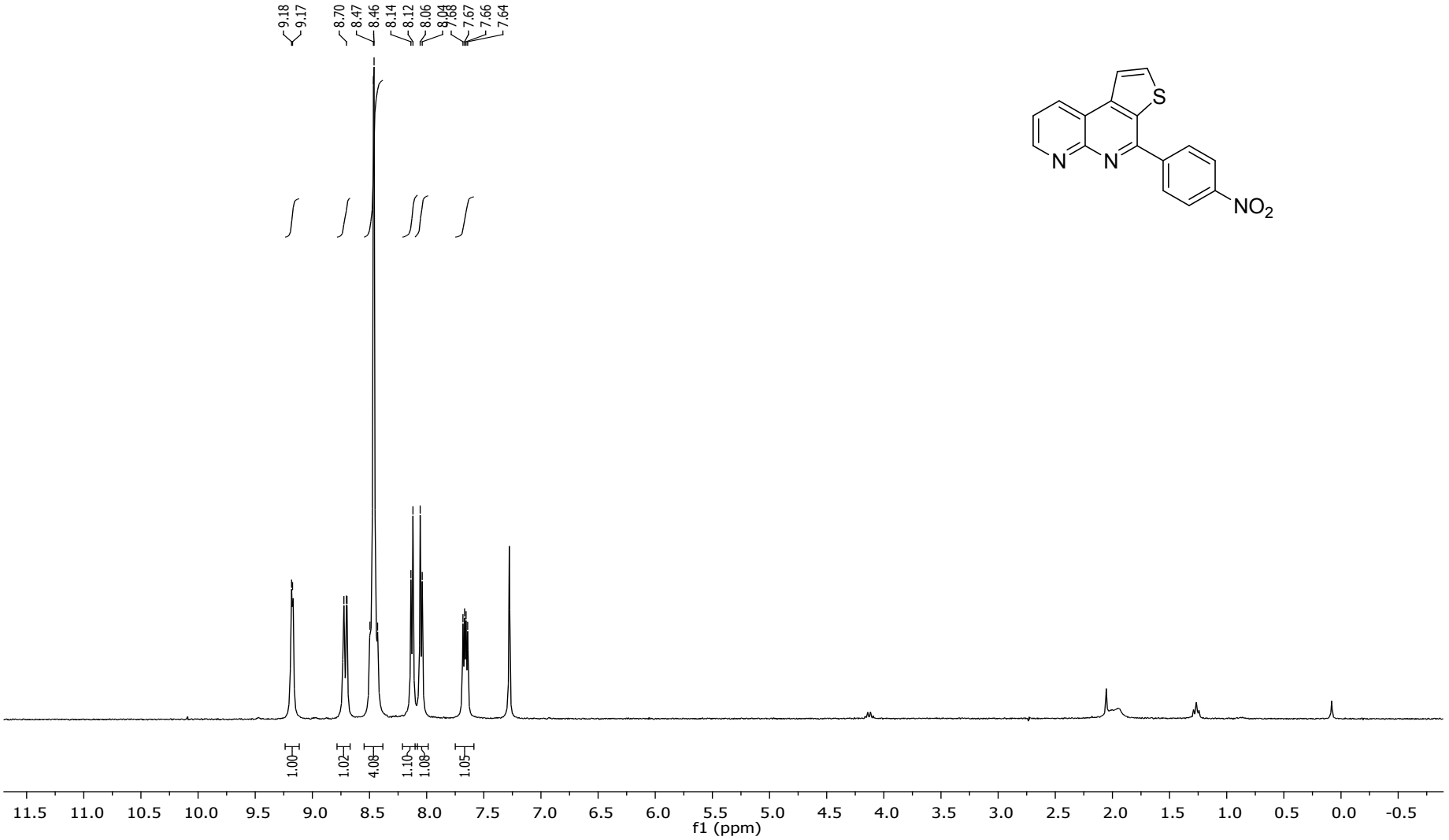
^{1}H NMR (300 MHz, $CDCl_3$): 4-phenylthieno[2,3-*c*][1,8]naphthyridine(7a)



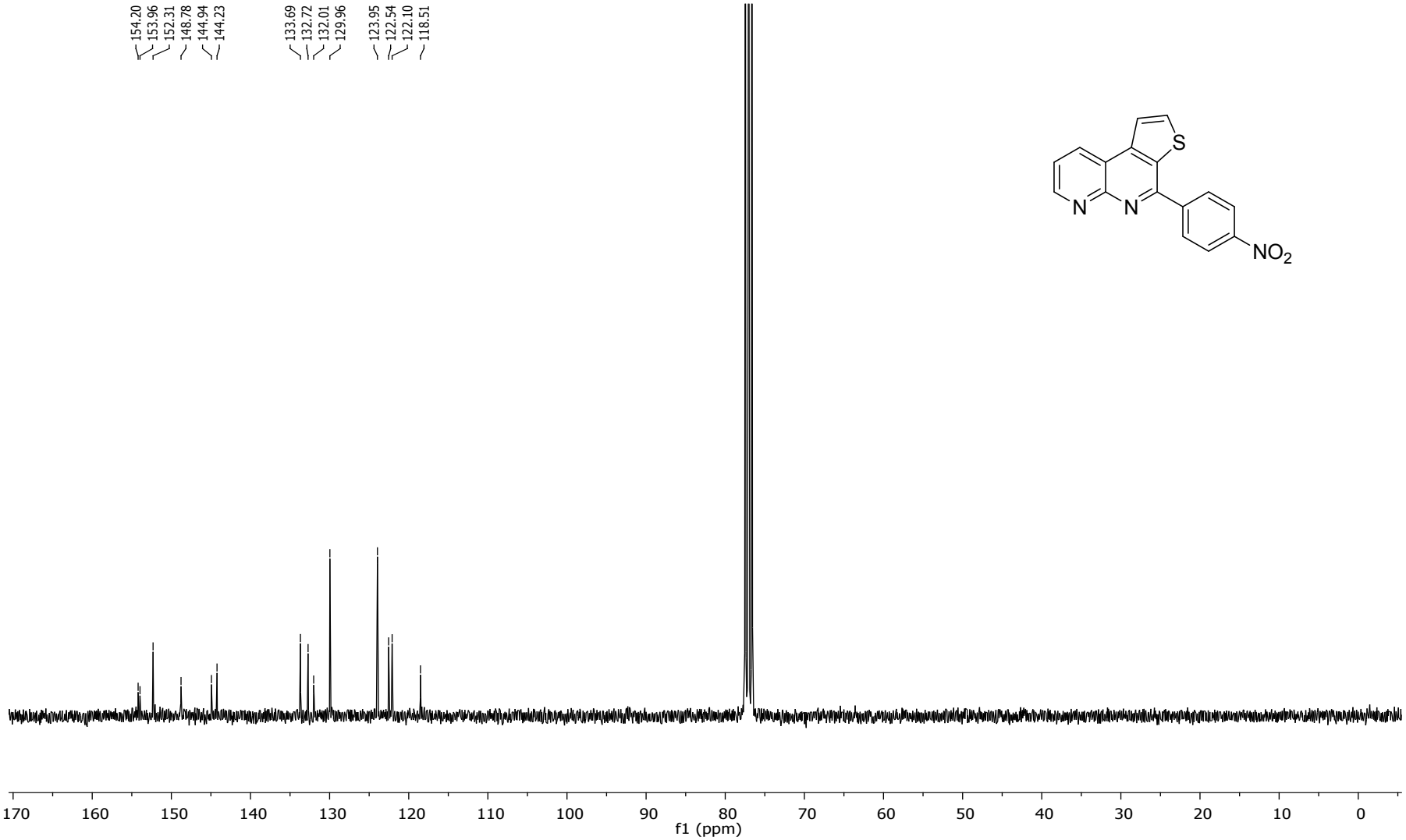
^{13}C NMR (75 MHz, $CDCl_3$): 4-phenylthieno[2,3-*c*][1,8]naphthyridine(7a)



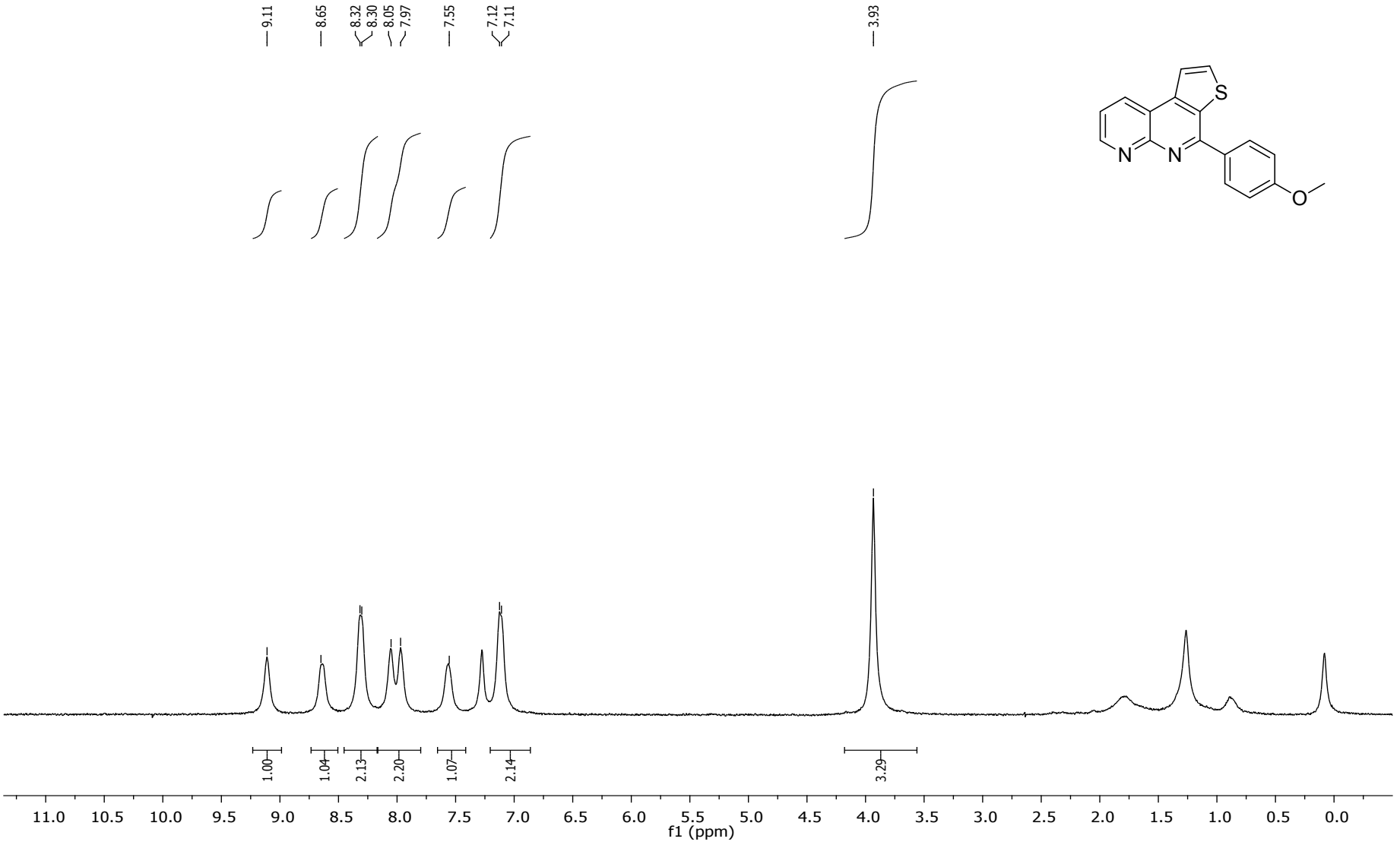
^{1}H NMR (300 MHz, $CDCl_3$): 4-(4-nitrophenyl)thieno[2,3-*c*][1,8]naphthyridine(7b)



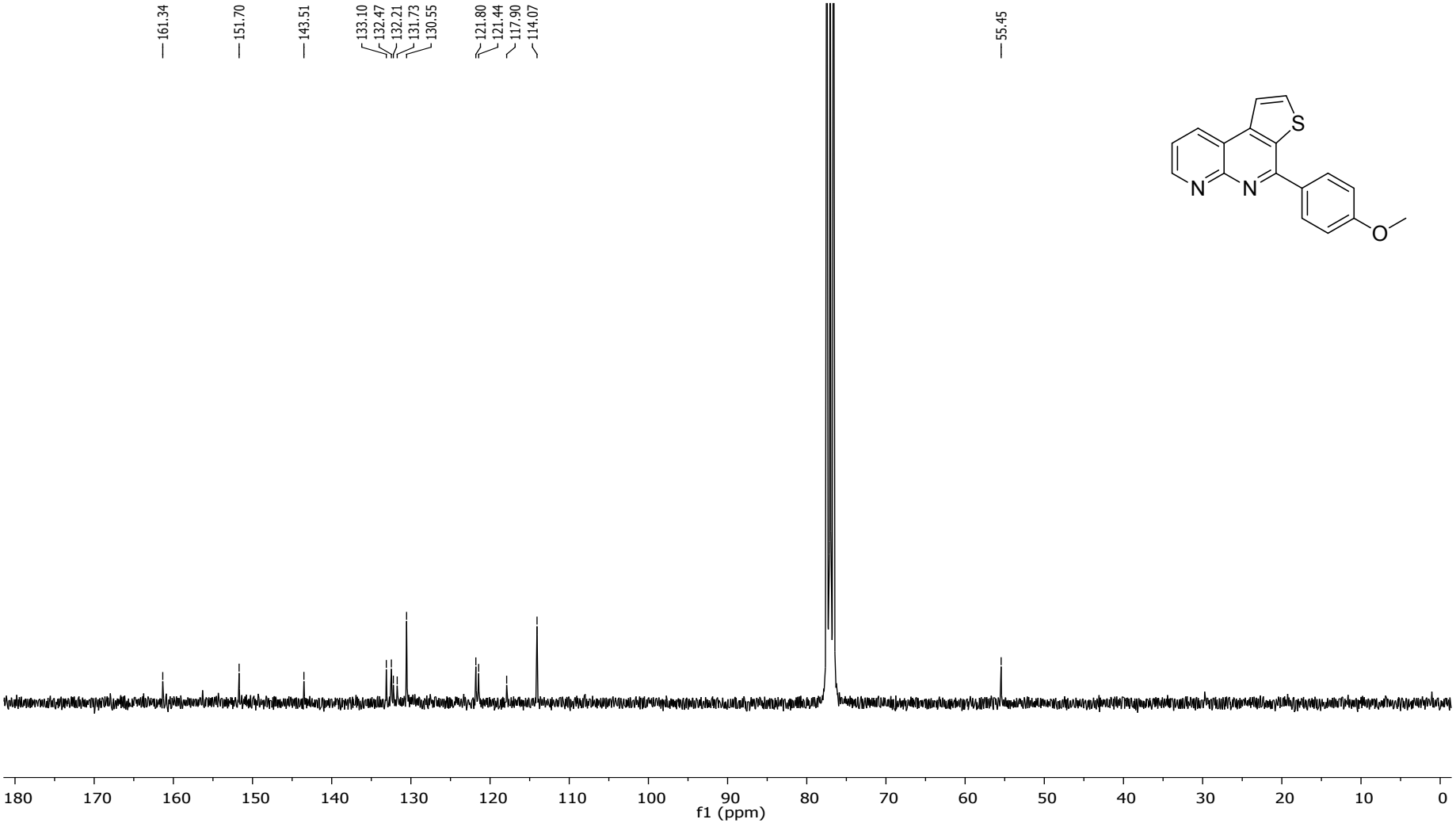
^{13}C NMR (75 MHz, $CDCl_3$): 4-(4-nitrophenyl)thieno[2,3-*c*][1,8]naphthyridine(7b)



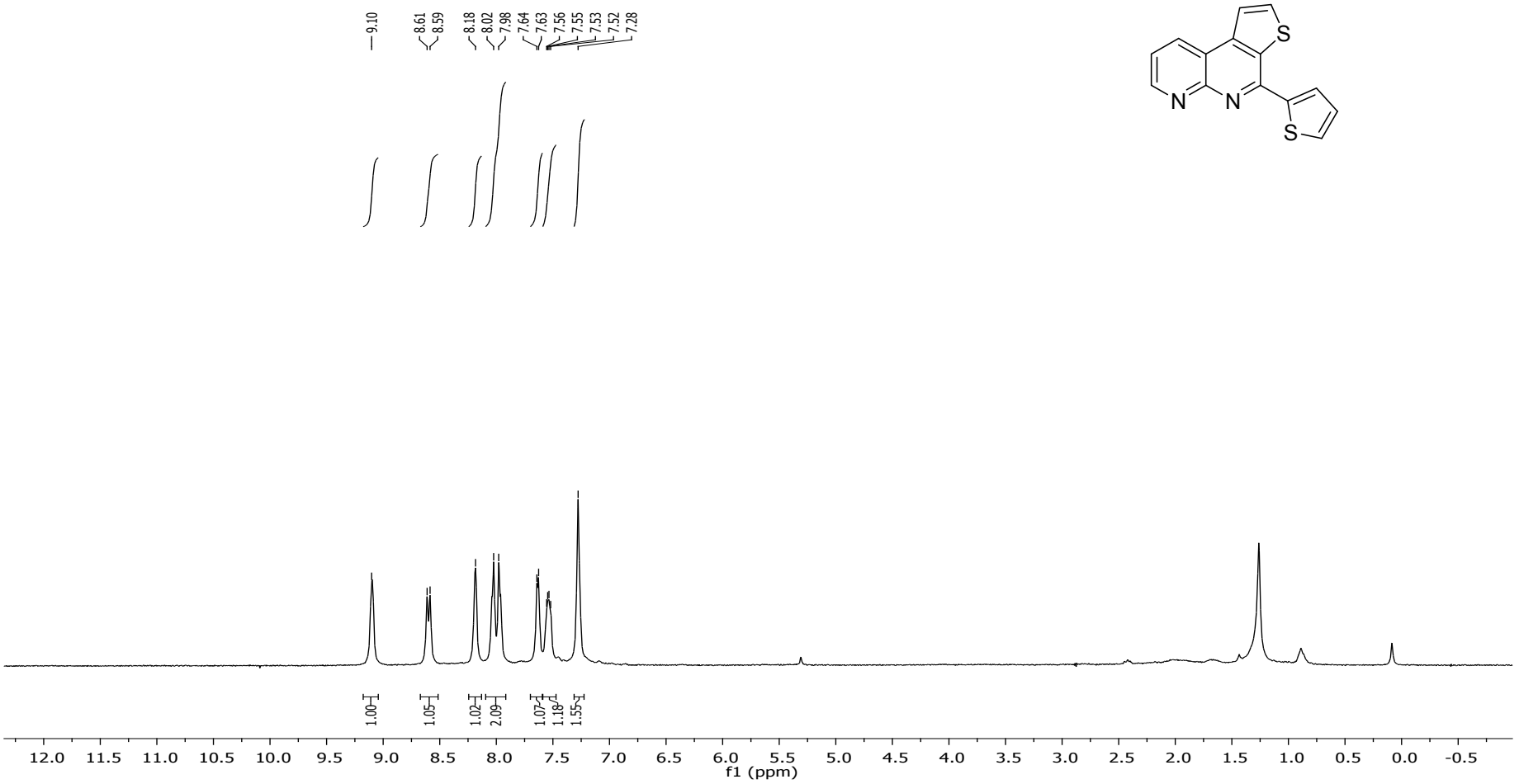
^{1}H NMR (300 MHz, $CDCl_3$): 4-(4-methoxyphenyl)thieno[2,3-*c*][1,8]naphthyridine(7c)



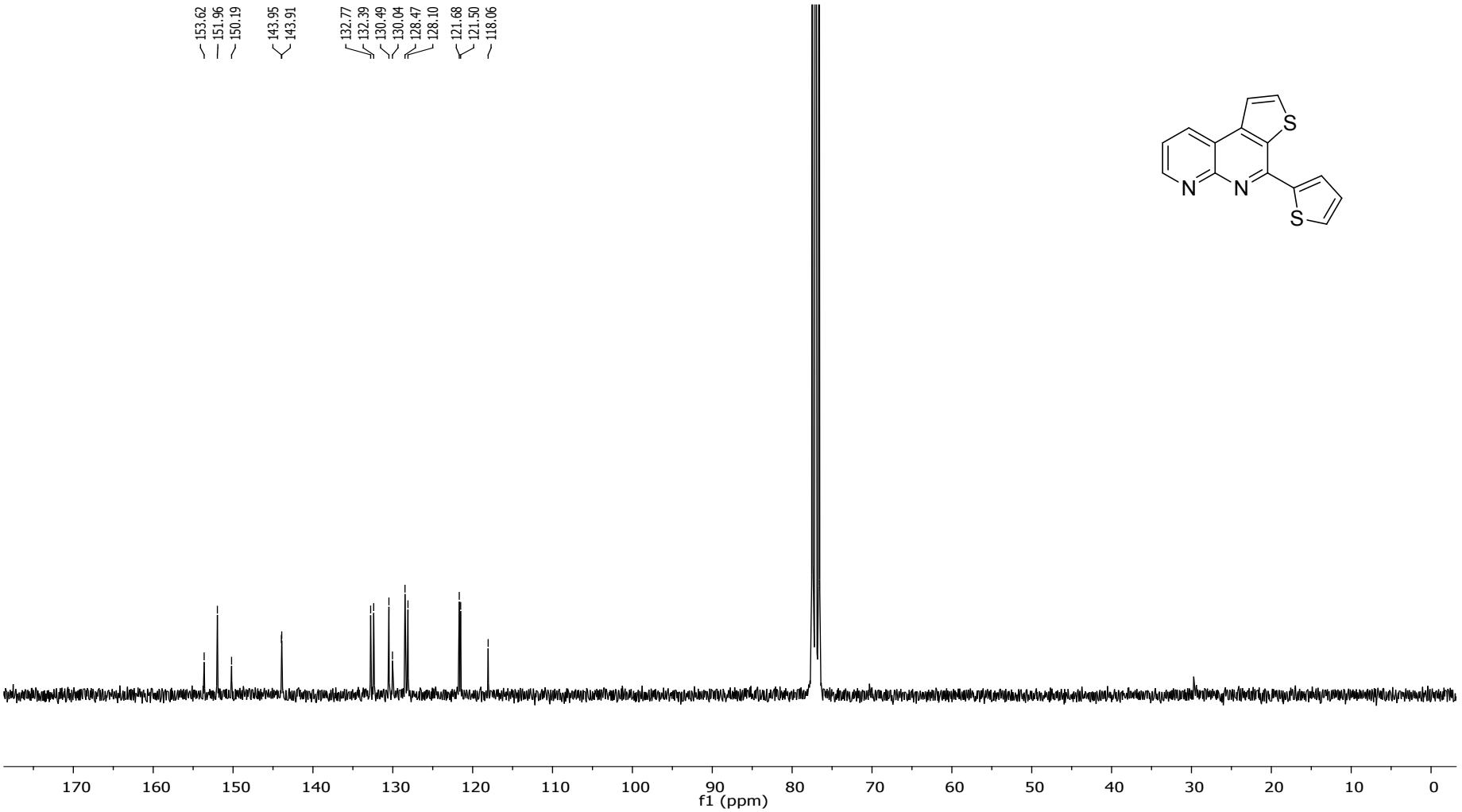
^{13}C NMR (75 MHz, $CDCl_3$): 4-(4-methoxyphenyl)thieno[2,3-*c*][1,8]naphthyridine(7c)



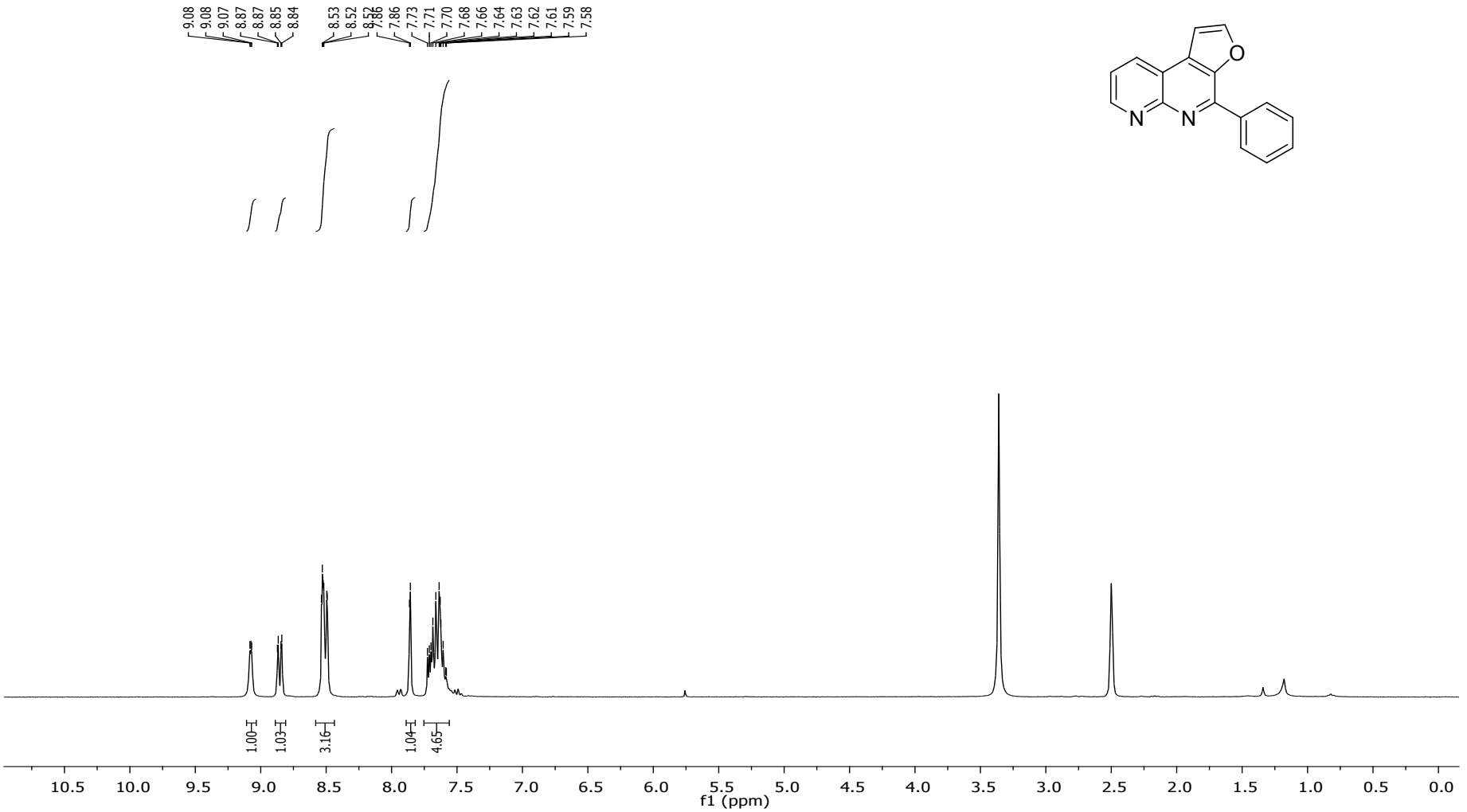
^{1}H NMR (300 MHz, $CDCl_3$): 4-(thiophen-2-yl)thieno[2,3-*c*][1,8]naphthyridine(7d)



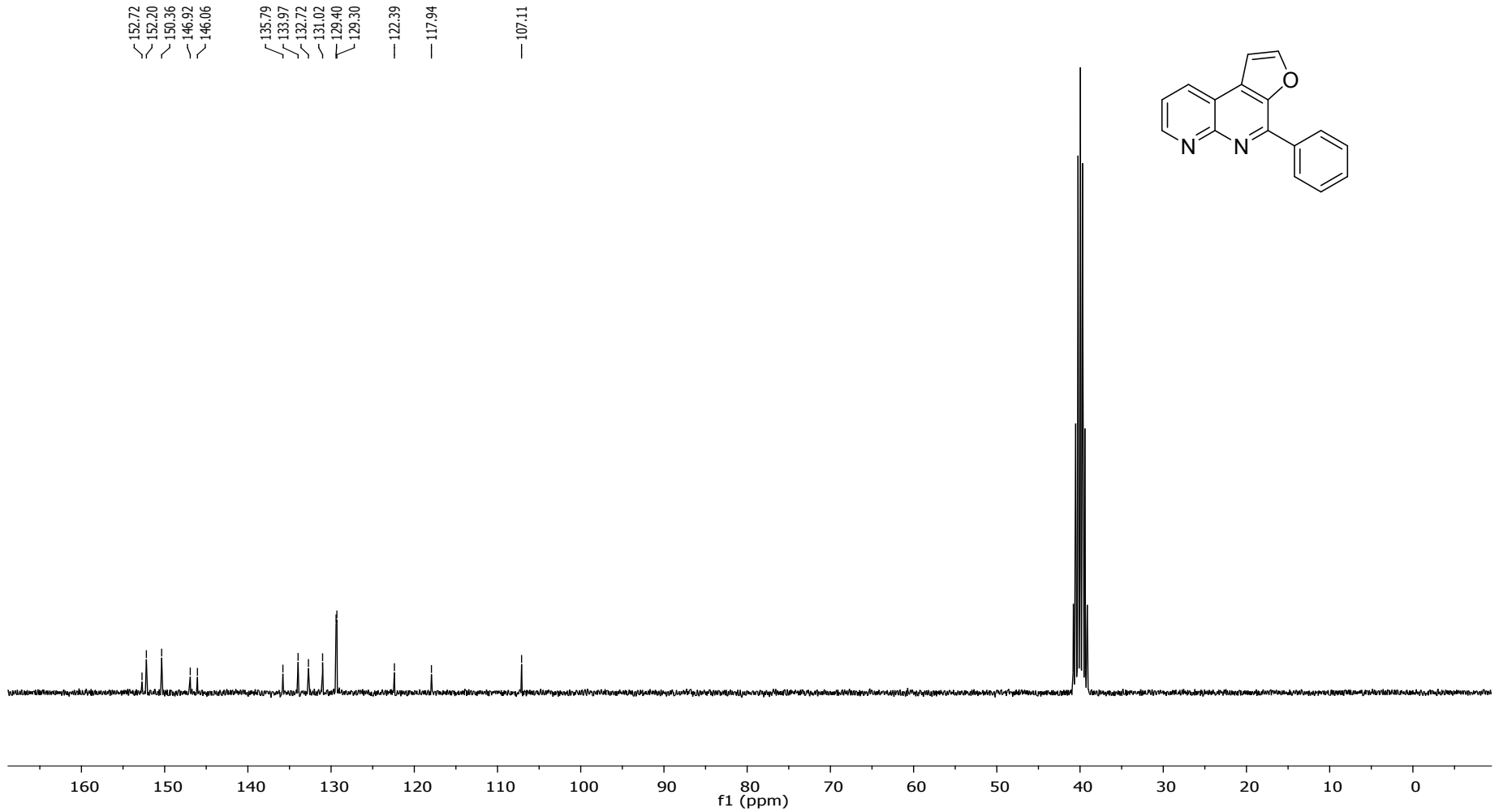
^{13}C NMR (75 MHz, $CDCl_3$): 4-(thiophen-2-yl)thieno[2,3-*c*][1,8]naphthyridine(7d)



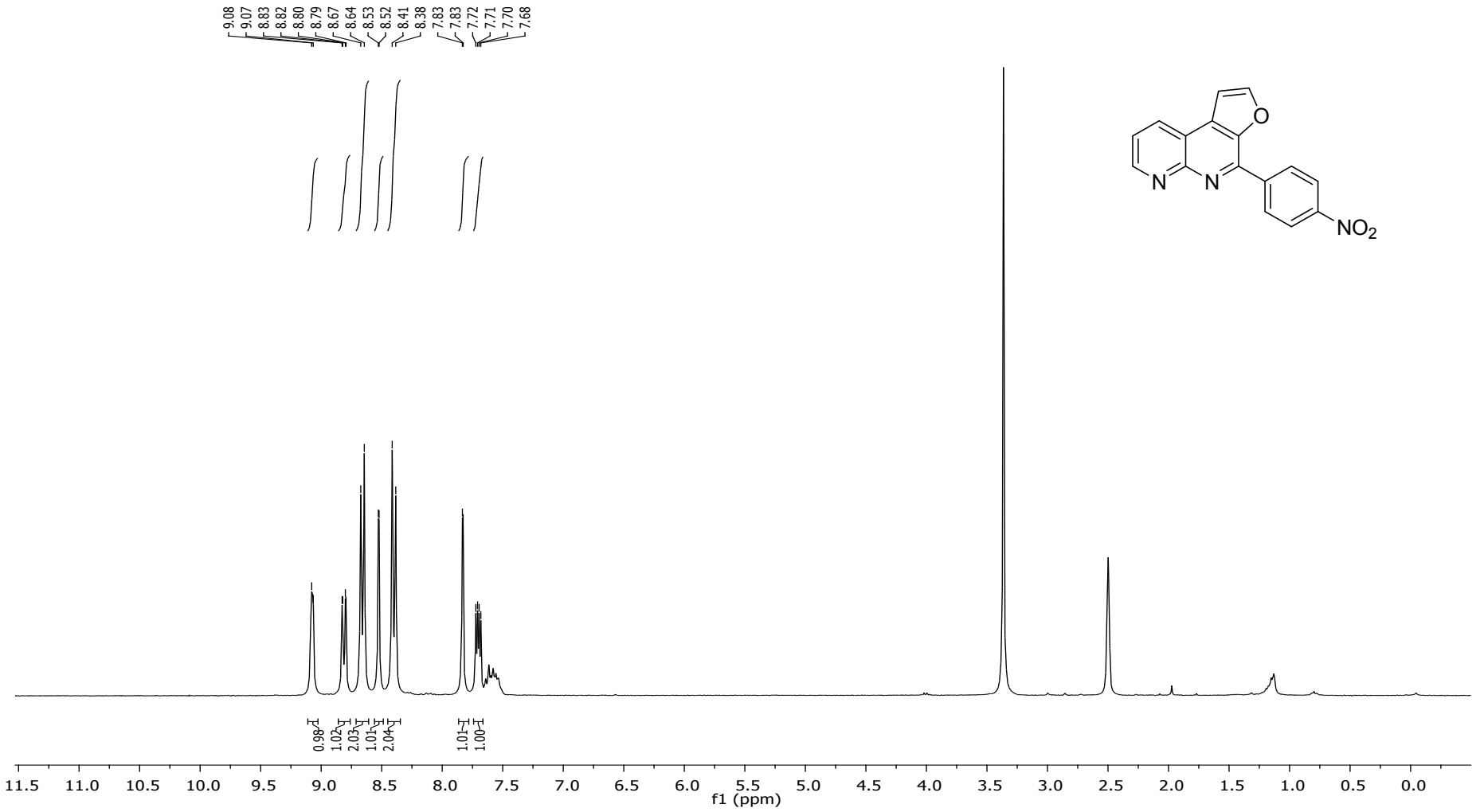
^{1}H NMR (300 MHz, $CDCl_3$): 4-phenylfuro[2,3-*c*][1,8]naphthyridine(8a)



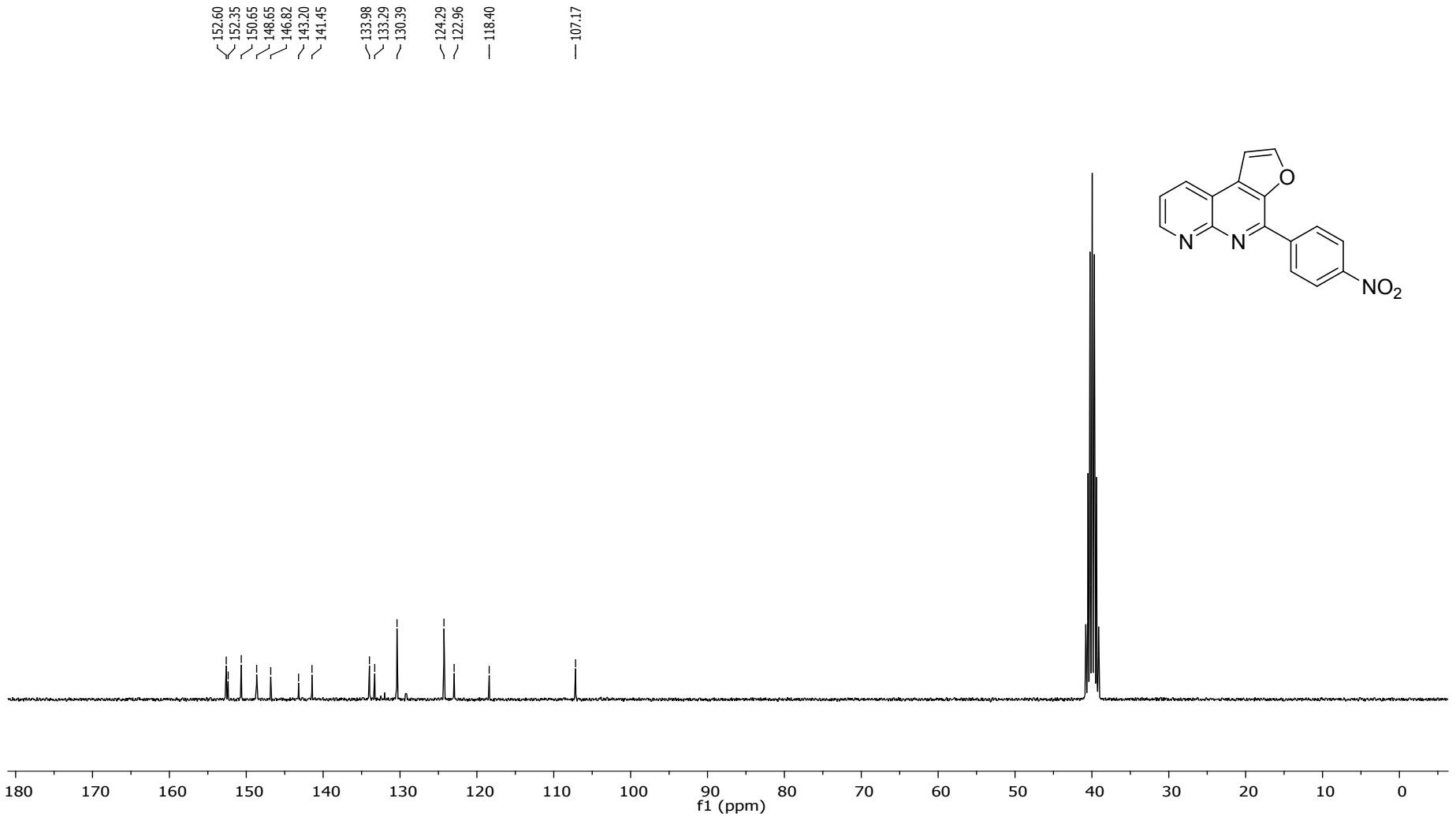
^{13}C NMR (75 MHz, $CDCl_3$): 4-phenylfuro[2,3-*c*][1,8]naphthyridine(8a)



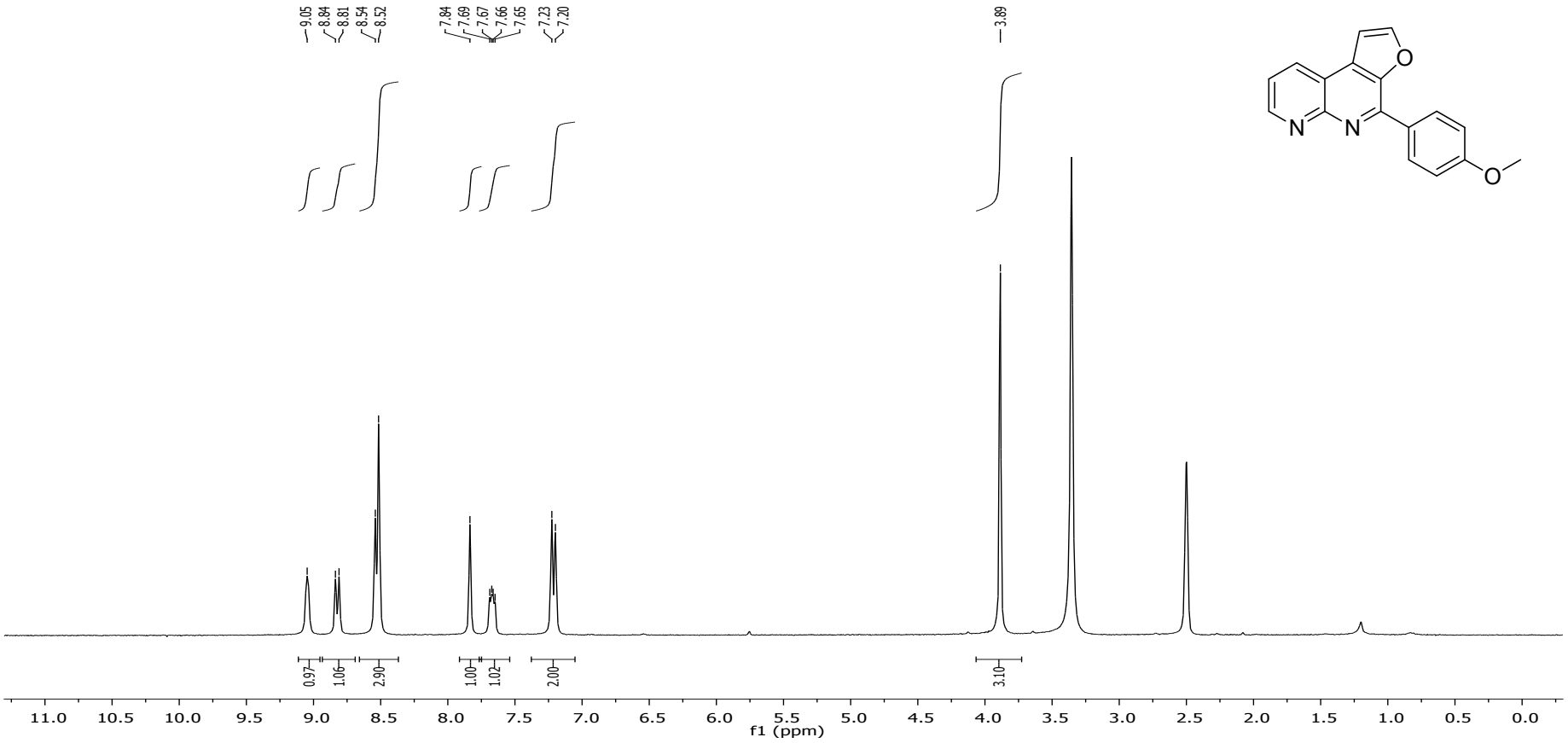
^{1}H NMR (300 MHz, DMSO-d$_6$): 4-(4-nitrophenyl)furo[2,3-*c*][1,8]naphthyridine(8b)



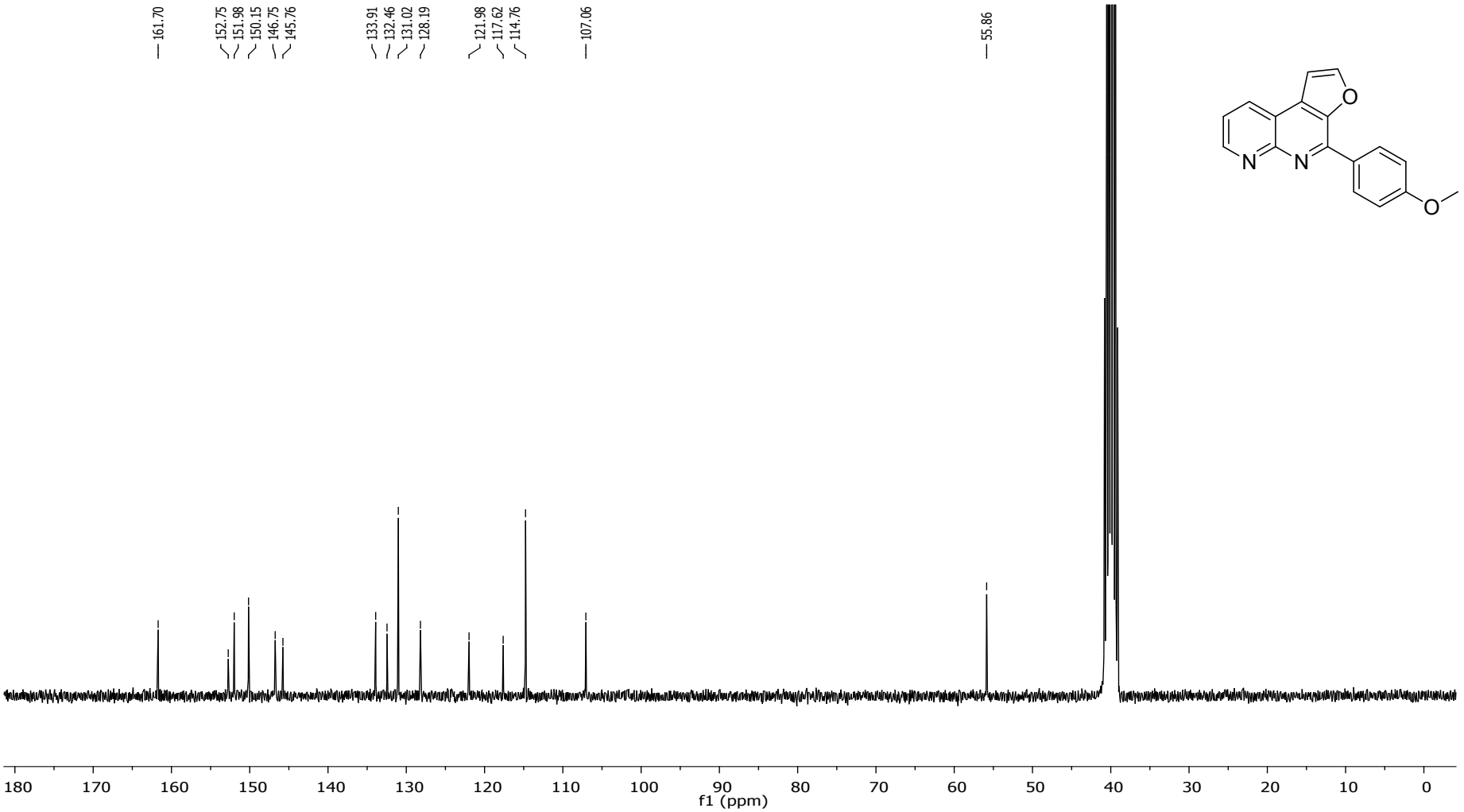
^{13}C NMR (75 MHz, DMSO-d_6): 4-(4-nitrophenyl)furo[2,3-*c*][1,8]naphthyridine(8b)



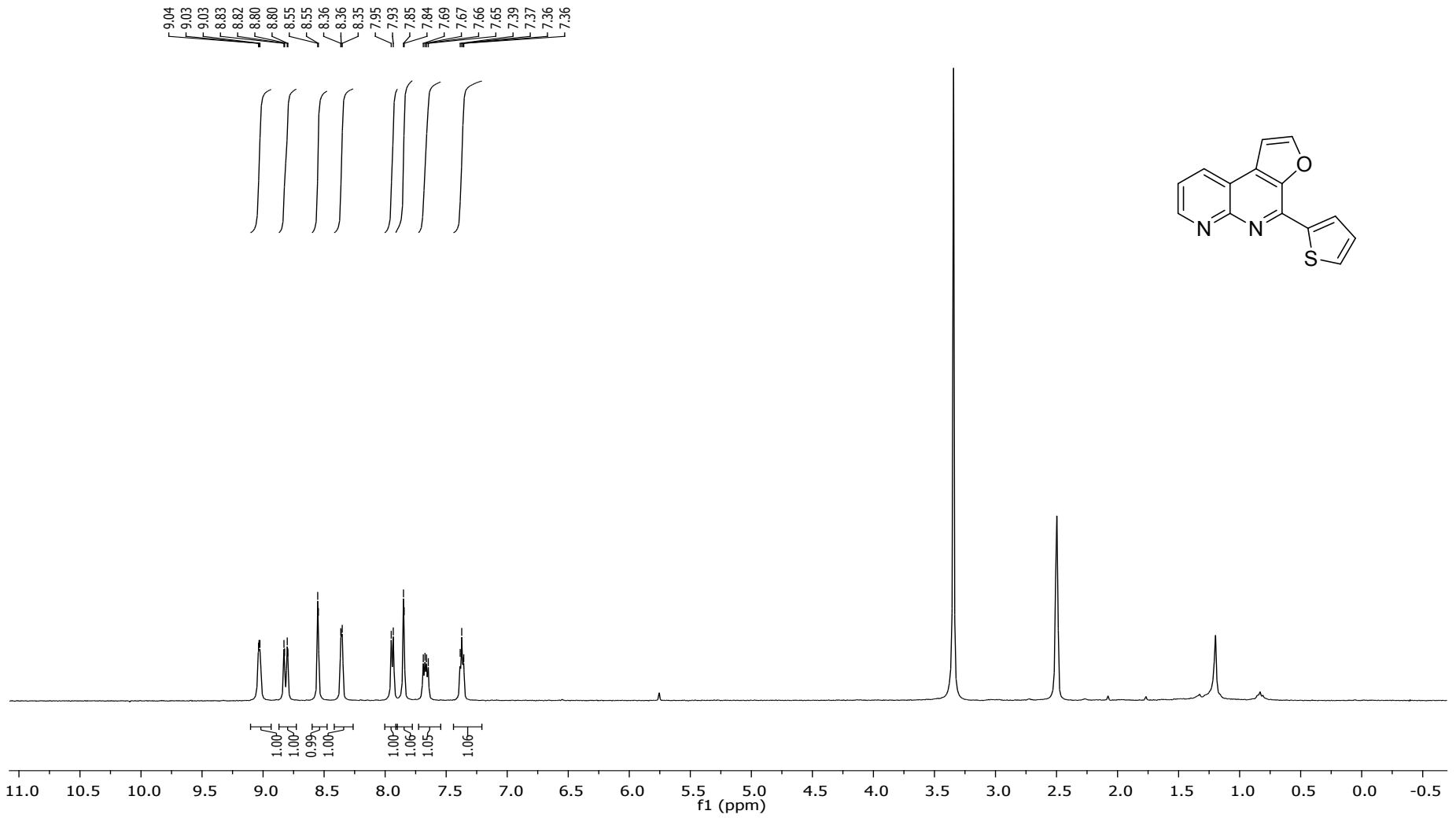
^{1}H NMR (300 MHz, DMSO-d_6): 4-(4-methoxyphenyl)furo[2,3-*c*][1,8]naphthyridine(8c)



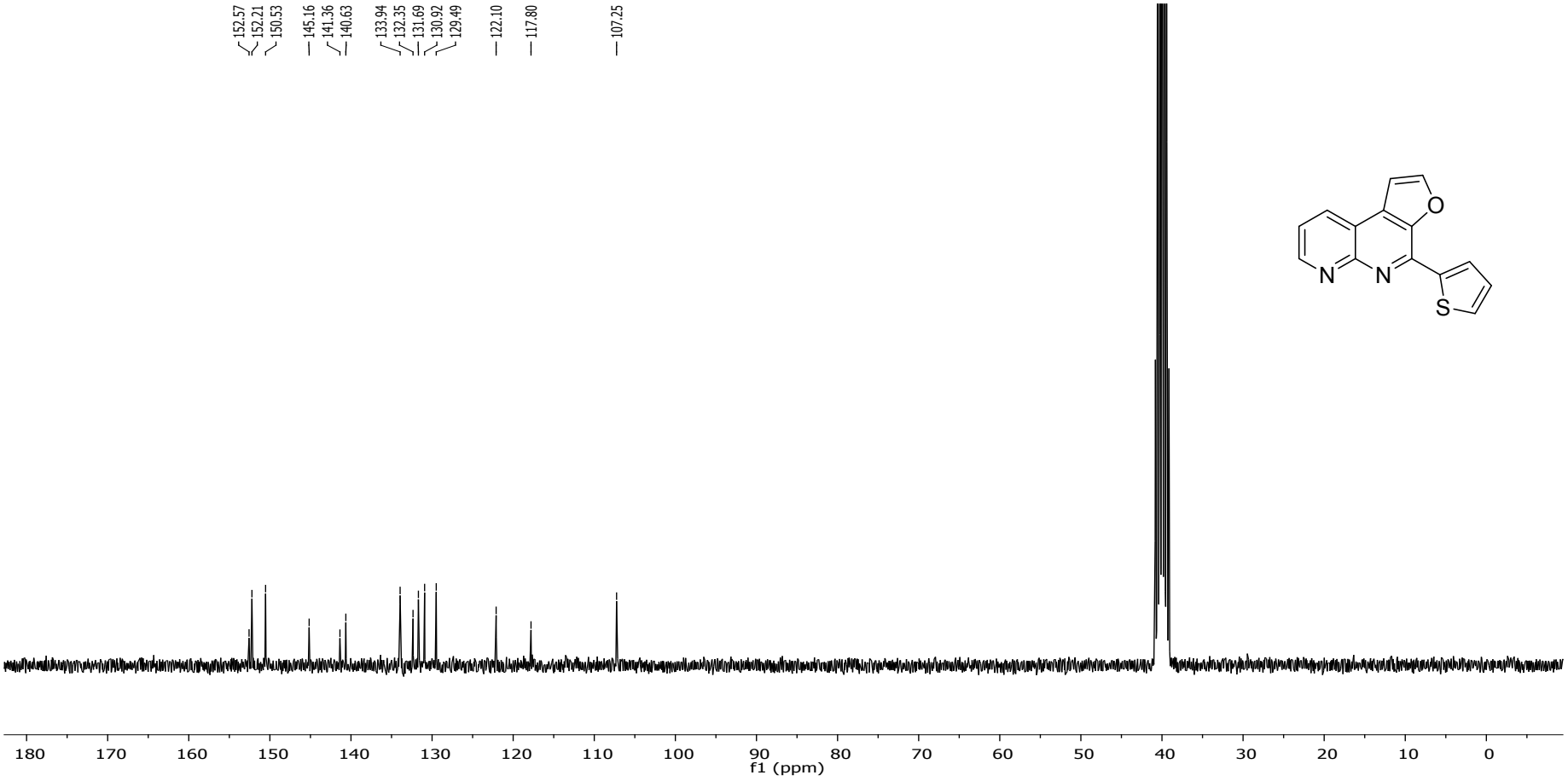
^{13}C NMR (75 MHz, DMSO-d_6): 4-(4-methoxyphenyl)furo[2,3-*c*][1,8]naphthyridine(8c)



^{1}H NMR (300 MHz, DMSO-d_6): 4-(thiophen-2-yl)furo[2,3-*c*][1,8]naphthyridine(8d)



^{13}C NMR (75 MHz, DMSO-d_6): 4-(thiophen-2-yl)furo[2,3-*c*][1,8]naphthyridine(8d)



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