

# Oxidative Tandem Nitrosation/Cyclization of N-Aryl Enamines with Nitromethane toward 3-(Trifluoromethyl)quinoxalines

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## Supporting Information

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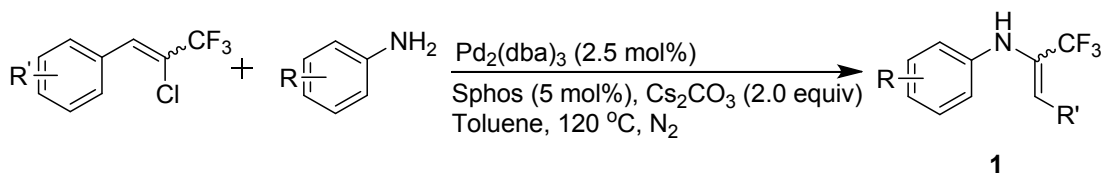
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## (A) Typical Experimental Procedure

### (a) General Information:

Chemicals were either purchased or purified by standard techniques.  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra were measured on a 500 MHz spectrometer ( $^1\text{H}$  at 500 MHz,  $^{13}\text{C}$  at 125 MHz), using  $\text{CDCl}_3$  as the solvent with tetramethylsilane (TMS) as an internal standard at room temperature. Chemical shifts are given in  $\delta$  relative to TMS, and the coupling constants  $J$  are given in hertz.  $^{19}\text{F}$  NMR spectra were recorded on a 500MHz spectrometer ( $^{19}\text{F}$  at 470 MHz) and are reported relative to the  $\text{CDCl}_3$  as the internal standard. High-resolution mass spectra were recorded on an ESI-Q-TOF mass spectrometer. All reactions under nitrogen atmosphere were conducted using standard Schlenk techniques. Melting points were measured on X4 melting point apparatus (Beijing Tech. Instrument) and uncorrected. Column chromatography was performed using EM silica gel 60 (300–400 mesh).

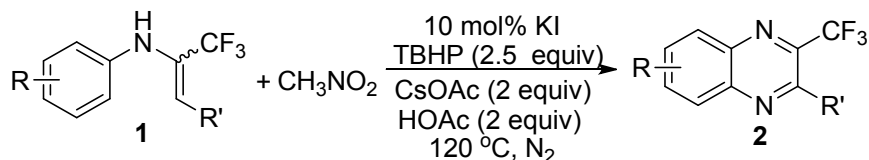
### (b) Typical Experimental Procedure for the Synthesis of Substrates 1:



#### N-(3,3,3-trifluoro-1-phenylprop-1-en-2-yl)anilines (**1**):<sup>[1]</sup>

(2-chloro-3,3,3-trifluoroprop-1-en-1-yl)benzenes (2 mmol), anilines (2.4 mmol),  $\text{Pd}_2\text{dba}_3$  (45.8 mg, 2.5 mol%), Sphos (41.0 mg, 5 mol%),  $\text{Cs}_2\text{CO}_3$  (1.304 g, 2.0 equiv) and toluene (8 mL) were added subsequently in a 25 mL two-neck flask under  $\text{N}_2$  atmosphere. The reaction mixture was stirred at 120 °C for 12h, upon completion of the reaction, the resulting mixture cooled to room temperature and filtered through a short path of silica gel, eluting with EtOAc, the volatile compounds were evaporated under vacuum and the residue was purified by flash column chromatography (hexane/ethyl acetate) to afford the desired products **1**.

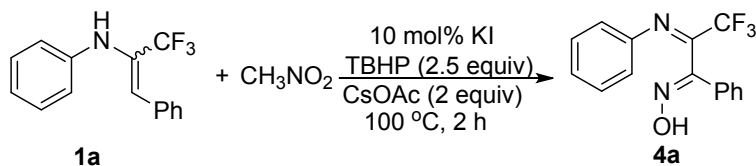
**(c) Typical Experimental Procedure for the Synthesis of 3-(Trifluoromethyl)-quinoxalines derivatives (2a–2v):**



To a 10 mL flame-dried Schlenk tube with a magnetic stirring bar were charged with N-(3,3,3-trifluoro-1-phenylprop-1-en-2-yl)anilines (0.2 mmol), KI (3.3 mg, 0.02 mmol), TBHP (0.5 mmol, 0.09 mL, 5.5 mol/L in decane), CsOAc (76.8 mg, 0.4 mmol),  $\text{CH}_3\text{COOH}$  (Anhydrous, 24.0 mg, 0.4 mmol), and  $\text{CH}_3\text{NO}_2$  (Anhydrous solvent 2 mL) under  $\text{N}_2$  atmosphere. The reaction mixture was stirred at  $120\text{ }^\circ\text{C}$  for 12 hours. After the reaction equilibrium, then cooled to room temperature, the mixture was poured into ethyl acetate and evaporated under vacuum. After removal  $\text{CH}_3\text{NO}_2$ , 0.2 equiv of Pd/C and 10 equiv of  $\text{HCOONH}_4$  in 2 mL MeOH were added, and then stirred at  $100\text{ }^\circ\text{C}$  for 12 h. It was then quenched with saturated  $\text{Na}_2\text{SO}_3$  solution and extracted with ethyl acetate. After the aqueous layer was extracted with ethyl acetate, the combined organic layers were dried over anhydrous  $\text{MgSO}_4$  and evaporated under vacuum. The residue was purified by flash column chromatography (hexane/ethyl acetate) to afford the desired products **2a–2v**.

**(d) Control Experiments**

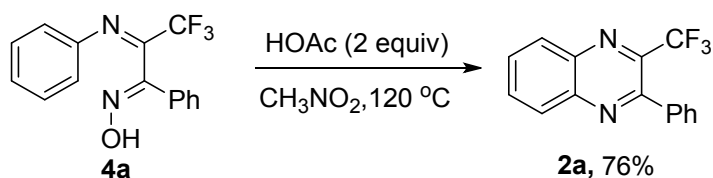
**(1) Preparation of (1E,2E)-3,3,3-trifluoro-1-phenyl-2-(phenylimino)propan-1-one oxime (4a):**



To a 10 mL flame-dried Schlenk tube with a magnetic stirring bar were charged with N-(3,3,3-trifluoro-1-phenylprop-1-en-2-yl)aniline (0.2 mmol), KI (3.3 mg, 0.02 mmol),

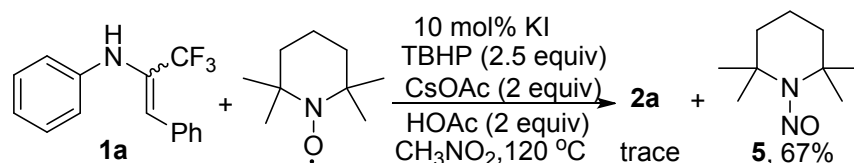
TBHP (0.5 mmol, 0.09 mL, 5.5 mol/L in decane ), CsOAc (76.8 mg, 0.4mmol ), and CH<sub>3</sub>NO<sub>2</sub> (Anhydrous solvent 2 mL) in the absence of acetic acid under N<sub>2</sub> atmosphere. The reaction mixture was stirred at 100 °C for 2 hours, and then cooled to room temperature, it was then quenched with saturated Na<sub>2</sub>SO<sub>3</sub> solution and extracted with ethyl acetate. After the aqueous layer was extracted with ethyl acetate, the combined organic layers were dried over anhydrous MgSO<sub>4</sub>. The residue was purified by flash column chromatography (hexane/ethyl acetate = 5:1) to afford the desired product **4a**. Its structure was confirmed by <sup>1</sup>H, <sup>13</sup>C NMR and HRMS analysis.

## (2) The transformation of intermidate **4a** to **2a**



To a 10 mL flame-dried Schlenk tube with a magnetic stirring bar were charged with the isolated intermidate **4a** (58.4mg, 0.2 mmol), CH<sub>3</sub>COOH (24 mg, 2 equiv) and CH<sub>3</sub>NO<sub>2</sub> (Anhydrous solvent 2 mL) under N<sub>2</sub> atmosphere at 120 °C for 12 h, and then cooled to room temperature, and extracted with ethyl acetate. The residue was purified by flash column chromatography (hexane/ethyl acetate = 10:1) to afford the desired product **2a** (41.6 mg) in 76% yield.

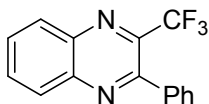
## (3) Preparation of 2,2,6,6-tetramethyl-1-nitrosopiperidine (**5**):



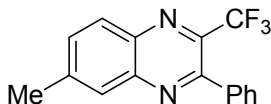
To a 10 mL flame-dried Schlenk tube with a magnetic stirring bar were charged with N-(3,3,3-trifluoro-1-phenylprop-1-en-2-yl)aniline (0.2 mmol), KI (3.3 mg, 0.02 mmol), TBHP (0.5 mmol, 0.09 mL, 5.5 mol/L in decane ), CsOAc (76.8 mg, 0.4mmol ), and

CH<sub>3</sub>NO<sub>2</sub> (Anhydrous solvent 2 mL), meanwhile, 2 equiv of TEMPO (62.4 mg), a radical inhibitor, was added to the current reaction under the standard conditions. The reaction mixture was stirred at 120 °C for 12 hours, then cooled to room temperature, it was then quenched with saturated Na<sub>2</sub>SO<sub>3</sub> solution and extracted with ethyl acetate. After the aqueous layer was extracted with ethyl acetate, the combined organic layers were dried over anhydrous MgSO<sub>4</sub> and evaporated under low temperature vacuum. The residue was purified by flash column chromatography (hexane/ethyl acetate=10:1) to afford the product **5**. Its structure was confirmed by <sup>1</sup>H, <sup>13</sup>C NMR and HRMS analysis.

**(B) Analytical data of compounds 2a-2v, 3a, 4a, 5**

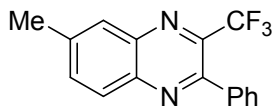


*2-Phenyl-3-(trifluoromethyl)quinoxaline (2a)*: yellow solid (48.2 mg, 88% yield), mp 115–116 °C<sup>[2]</sup>; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.28–8.26 (m, 1H), 8.23–8.21 (m, 1H), 7.94–7.87 (m, 2H), 7.65–7.63 (m, 2H); 7.55–7.52 (m, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 152.6, 142.5, 141.2 (q, *J*<sub>C-F</sub> = 33.8 Hz), 139.3, 137.3, 132.6, 131.1, 129.7, 129.5, 129.3, 128.8, 128.3, 121.4 (q, *J*<sub>C-F</sub> = 273.8 Hz); <sup>19</sup>F NMR (470 MHz, CDCl<sub>3</sub>) δ -61.66 (3F); LRMS (EI 70 ev), *m/z* (%): 274 (100), 205 (99), 179 (23), 77 (30), 76 (18); HRMS (ESI) calcd for C<sub>15</sub>H<sub>10</sub>F<sub>3</sub>N<sub>2</sub><sup>+</sup> ([M + H]<sup>+</sup>) 275.0791, found 275.0785.

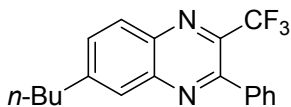


*6-Methyl-3-phenyl-2-(trifluoromethyl)quinoxaline (2b)*: yellow solid (47.8 mg, 83% yield), mp 120–121 °C; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.15 (d, *J* = 8.5 Hz, 1H), 7.98 (s, 1H), 7.23–7.71 (m, 1H), 7.63–7.61 (m, 2H), 7.53–7.52 (m, 3H), 2.65 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 152.6, 143.7, 142.7, 140.3 (q, *J*<sub>C-F</sub> = 33.8 Hz), 137.9, 137.5, 133.6,

129.4, 129.2, 128.8, 128.3, 128.0, 121.5 (q,  $J_{C-F} = 273.8$  Hz), 22.1;  $^{19}\text{F}$  NMR (470 MHz,  $\text{CDCl}_3$ )  $\delta$  -61.51 (3F); LRMS (EI 70 ev),  $m/z$  (%): 288 (100), 219 (87), 218 (22), 192 (29), 91 (36); HRMS (ESI) calcd for  $\text{C}_{16}\text{H}_{12}\text{F}_3\text{N}_2^+$  ( $[\text{M} + \text{H}]^+$ ) 289.0947, found 289.0939.

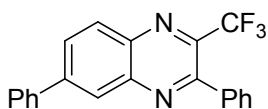


*6-Methyl-2-phenyl-3-(trifluoromethyl)quinoxaline (2c)*: yellow solid (44.4 mg, 77% yield), mp 109–110 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.10 (d,  $J = 8.5$  Hz, 1H), 8.04 (s, 1H), 7.77–7.75 (m, 1H), 7.63–7.61 (m, 2H), 7.54–7.51 (m, 3H), 2.65 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  151.7, 142.0, 141.1, 141.0 (q,  $J_{C-F} = 33.8$  Hz), 139.5, 137.5, 135.1, 129.3, 128.84, 128.78, 128.4, 128.3, 121.5 (q,  $J_{C-F} = 273.8$  Hz), 21.9;  $^{19}\text{F}$  NMR (470 MHz,  $\text{CDCl}_3$ )  $\delta$  -61.65 (3F); LRMS (EI 70 ev),  $m/z$  (%): 289 (18), 288 (100), 219 (86), 89 (31), 77 (23); HRMS (ESI) calcd for  $\text{C}_{16}\text{H}_{12}\text{F}_3\text{N}_2^+$  ( $[\text{M} + \text{H}]^+$ ) 289.0947, found 289.0944.

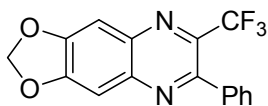


*6-Butyl-3-phenyl-2-(trifluoromethyl)quinoxaline (2d)*: yellow solid (51.5 mg, 78% yield), mp 87–88 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.17 (d,  $J = 8.5$  Hz, 1H), 7.99 (s, 1H), 7.74 (d,  $J = 8.5$  Hz, 1H), 7.63–7.62 (m, 2H), 7.53–7.52 (m, 3H), 2.90 (t,  $J = 7.5$  Hz, 2H), 1.78–1.72 (m, 2H), 1.45–1.38 (m, 2H), 0.96 (t,  $J = 7.5$  Hz, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  152.5, 148.5, 142.7, 140.3 (q,  $J_{C-F} = 33.8$  Hz), 138.1, 137.6, 133.0, 129.3, 129.2, 128.8, 128.2, 127.4, 121.5 (q,  $J_{C-F} = 273.8$  Hz), 35.9, 32.8, 22.2, 13.8;  $^{19}\text{F}$  NMR (470 MHz,  $\text{CDCl}_3$ )  $\delta$  -61.49 (3F); LRMS (EI 70 ev),  $m/z$  (%): 330 (38), 288 (100), 287 (24), 219

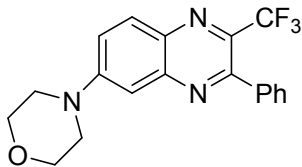
(20), 115 (18); HRMS (ESI) calcd for  $C_{19}H_{18}F_3N_2^+$  ( $[M + H]^+$ ) 331.1417, found 334.1414.



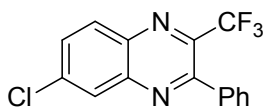
*3,6-Diphenyl-2-(trifluoromethyl)quinoxaline (2e)*: yellow solid (56.0 mg, 80% yield), mp 104–106 °C;  $^1H$  NMR (500 MHz,  $CDCl_3$ )  $\delta$  8.42 (s, 1H), 8.32 (d,  $J = 7.5$  Hz, 1H), 8.17–8.15 (m, 1H), 7.78–7.77 (m, 2H), 7.66–7.65 (m, 2H), 7.55–7.51 (m, 5H), 7.47–7.44 (m, 1H);  $^{13}C$  NMR (125 MHz,  $CDCl_3$ )  $\delta$  152.9, 145.3, 142.7, 140.7 (q,  $J_{C-F} = 33.8$  Hz), 138.9, 138.6, 137.3, 130.8, 129.9, 129.4, 129.1, 128.69, 128.66, 128.2, 127.4, 126.3, 121.3 (q,  $J_{C-F} = 275.0$  Hz);  $^{19}F$  NMR (470 MHz,  $CDCl_3$ )  $\delta$  -61.54 (3F); LRMS (EI 70 ev),  $m/z$  (%): 350 (100), 281 (77), 152 (26), 151 (38), 102 (34); HRMS (ESI) calcd for  $C_{21}H_{14}F_3N_2^+$  ( $[M + H]^+$ ) 351.1104, found 351.1099.



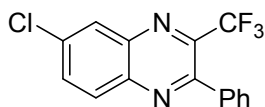
*6-Phenyl-7-(trifluoromethyl)-[1,3]dioxolo[4,5-g]quinoxaline (2f)*: yellow liquid (45.8 mg, 72% yield);  $^1H$  NMR (500 MHz,  $CDCl_3$ )  $\delta$  7.60–7.59 (m, 2H), 7.51–7.50 (m, 3H), 7.46 (s, 1H), 7.41 (s, 1H), 6.24 (s, 2H);  $^{13}C$  NMR (125 MHz,  $CDCl_3$ )  $\delta$  153.2, 152.1, 150.3, 141.9, 138.5 (q,  $J_{C-F} = 35.0$  Hz), 138.3, 137.6, 129.2, 128.9, 128.2, 121.8 (q,  $J_{C-F} = 273.8$  Hz), 104.6, 104.4, 102.9;  $^{19}F$  NMR (470 MHz,  $CDCl_3$ )  $\delta$  -61.23 (3F); LRMS (EI 70 ev),  $m/z$  (%): 318 (100), 249 (65), 120 (28), 88 (17), 77 (35); HRMS (ESI) calcd for  $C_{16}H_{10}F_3N_2O_2^+$  ( $[M + H]^+$ ) 319.0689, found 319.0682.



*4-(3-Phenyl-2-(trifluoromethyl)quinoxalin-6-yl)morpholine (2g)*: yellow solid (33.0 mg, 46% yield), mp 137–138 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.08 (d,  $J = 9.5$  Hz, 1H), 7.62–7.59 (m, 3H), 7.51–7.50 (m, 3H), 7.33 (s, 1H), 3.91 (t,  $J = 5.0$  Hz, 4H), 3.43 (t,  $J = 5.0$  Hz, 4H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  153.6, 153.0, 144.5, 137.8, 137.6 (q,  $J_{\text{C-F}} = 33.8$  Hz), 134.7, 130.2, 129.2, 128.7, 128.2, 122.5, 121.8 (q,  $J_{\text{C-F}} = 273.8$  Hz) 108.2, 66.5, 48.0;  $^{19}\text{F}$  NMR (470 MHz,  $\text{CDCl}_3$ )  $\delta$  -61.00 (3F); LRMS (EI 70 eV),  $m/z$  (%): 359 (78), 301 (100), 232 (60), 103 (24), 102 (30); HRMS (ESI) calcd for  $\text{C}_{19}\text{H}_{17}\text{F}_3\text{N}_3\text{O}^+$  ( $[\text{M} + \text{H}]^+$ ) 360.1318, found 360.1315.

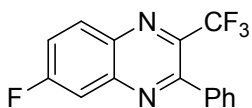


*6-Chloro-3-phenyl-2-(trifluoromethyl)quinoxaline (2h)*: yellow solid (32.0 mg, 52% yield), mp 123–124 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.22–8.20 (m, 2H), 7.85–7.83 (m, 1H), 7.63–7.62 (m, 2H), 7.55–7.54 (m, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  153.6, 142.8, 141.3 (q,  $J_{\text{C-F}} = 35.0$  Hz), 138.9, 137.9, 136.9, 132.4, 130.9, 129.7, 128.8, 128.4, 128.2, 121.2 (q,  $J_{\text{C-F}} = 273.8$  Hz);  $^{19}\text{F}$  NMR (470 MHz,  $\text{CDCl}_3$ )  $\delta$  -61.75 (3F); LRMS (EI 70 eV),  $m/z$  (%): 310 (32), 308 (100), 239 (93), 241 (29), 77 (58); HRMS (ESI) calcd for  $\text{C}_{15}\text{H}_9\text{ClF}_3\text{N}_2^+$  ( $[\text{M} + \text{H}]^+$ ) 309.0401, found 309.0390.

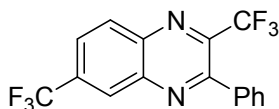




*6-Chloro-2-phenyl-3-(trifluoromethyl)quinoxaline (2i)*: yellow liquid (25.3 mg, 41% yield);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.54 (d,  $J = 9.0$  Hz, 1H), 8.28 (s, 1H), 7.81 (d,  $J = 9.0$  Hz, 1H), 7.58–7.56 (m, 3H), 7.42–7.40 (m, 2H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  145.5 (q,  $J_{\text{C-F}} = 33.8$  Hz), 143.2, 140.4, 139.0, 136.7, 133.2, 130.4, 129.7, 129.6, 128.8, 127.8, 121.0, 120.4 (q,  $J_{\text{C-F}} = 275.0$  Hz);  $^{19}\text{F}$  NMR (470 MHz,  $\text{CDCl}_3$ )  $\delta$  -62.98 (3F); LRMS (EI 70 ev),  $m/z$  (%): 310 (35), 308 (100), 239 (85), 77 (73), 75 (28); HRMS (ESI) calcd for  $\text{C}_{15}\text{H}_9\text{ClF}_3\text{N}_2^+$  ( $[\text{M} + \text{H}]^+$ ) 309.0401, found 309.0397.

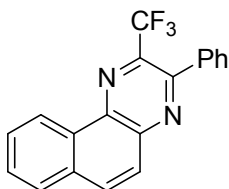


*6-Fluoro-3-phenyl-2-(trifluoromethyl)quinoxaline (2j)*: yellow solid (39.7 mg, 68% yield), mp 95–96 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.30–8.27 (m, 1H), 7.85–7.82 (m, 1H), 7.70–7.62 (m, 3H), 7.56–7.52 (m, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  164.5 (d,  $J_{\text{C-F}} = 255.0$  Hz), 153.5, 143.6, 140.7 (q,  $J_{\text{C-F}} = 37.5$  Hz), 137.0, 136.6, 132.1, 129.7, 128.8, 128.4, 121.9 (d,  $J_{\text{C-F}} = 26.3$  Hz), 121.3 (q,  $J_{\text{C-F}} = 275.0$  Hz), 112.9 (d,  $J_{\text{C-F}} = 21.3$  Hz);  $^{19}\text{F}$  NMR (470 MHz,  $\text{CDCl}_3$ )  $\delta$  -61.67 (3F), -103.06 (1F); LRMS (EI 70 ev),  $m/z$  (%): 293 (17), 292 (100), 223 (91), 197 (23), 77 (39); HRMS (ESI) calcd for  $\text{C}_{15}\text{H}_9\text{F}_4\text{N}_2^+$  ( $[\text{M} + \text{H}]^+$ ) 293.0696, found 293.0691.

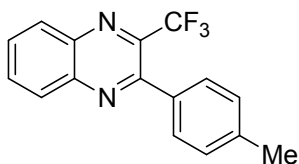


*3-Phenyl-2,6-bis(trifluoromethyl)quinoxaline (2k)*: yellow solid (22.6 mg, 33% yield), mp 84–85 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.55 (s, 1H), 8.41 (d,  $J = 9.0$  Hz, 1H), 8.08–8.06 (m, 1H), 7.66–7.64 (m, 2H), 7.58–7.55 (m, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$

154.1, 143.1 (q,  $J_{C-F} = 33.8$  Hz), 141.6, 140.3, 136.7, 134.2 (q,  $J_{C-F} = 33.8$  Hz), 131.1, 129.9, 128.8, 128.4, 127.4, 126.7, 123.2 (q,  $J_{C-F} = 271.3$  Hz), 121.0 (q,  $J_{C-F} = 275.0$  Hz);  $^{19}\text{F}$  NMR (470 MHz,  $\text{CDCl}_3$ )  $\delta$  -62.00 (3F), -63.08 (3F); LRMS (EI 70 ev), m/z (%): 342 (82), 274 (16), 273 (100), 247 (20), 77 (54); HRMS (ESI) calcd for  $\text{C}_{16}\text{H}_9\text{F}_6\text{N}_2^+$  ( $[\text{M} + \text{H}]^+$ ) 343.0664, found 343.0649.

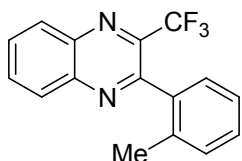


*3-Phenyl-2-(trifluoromethyl)benzo[f]quinoxaline (21)*: yellow solid (39.5 mg, 61% yield), mp 178–179 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  9.28–9.26 (m, 1H), 8.16 (d,  $J = 9.0$  Hz, 1H), 8.01 (d,  $J = 9.0$  Hz, 1H), 7.98–7.96 (m, 1H), 7.85–7.79 (m, 2H), 7.70–7.68 (m, 2H), 7.55–7.54 (m, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  152.3, 142.8, 139.2 (q,  $J_{C-F} = 33.8$  Hz), 138.3, 137.5, 134.6, 133.5, 130.3, 129.7, 129.4, 129.0, 128.4, 128.3, 128.1, 126.1, 125.0, 121.8 (q,  $J_{C-F} = 273.8$  Hz);  $^{19}\text{F}$  NMR (470 MHz,  $\text{CDCl}_3$ )  $\delta$  -61.05 (3F); LRMS (EI 70 ev), m/z (%): 325 (21), 324 (100), 255 (58), 152 (47), 126 (29); HRMS (ESI) calcd for  $\text{C}_{19}\text{H}_{12}\text{F}_3\text{N}_2^+$  ( $[\text{M} + \text{H}]^+$ ) 325.0947, found 325.0945.

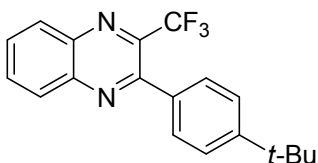


*2-(p-tolyl)-3-(trifluoromethyl)quinoxaline (2m)*: yellow solid (48.4 mg, 84% yield), mp 127–128 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.27–8.25 (m, 1H), 8.22–8.20 (m, 1H), 7.93–7.86 (m, 2H), 7.54 (d,  $J = 8.0$  Hz, 2H), 7.34 (d,  $J = 8.0$  Hz, 2H), 2.46 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  152.7, 142.6, 141.2 (q,  $J_{C-F} = 33.8$  Hz), 139.6, 139.2, 134.5, 132.5,

131.0, 129.7, 129.3, 129.0, 128.8, 121.4 (q,  $J_{C-F} = 273.8$  Hz), 21.4;  $^{19}\text{F}$  NMR (470 MHz,  $\text{CDCl}_3$ )  $\delta$  -61.71 (3F); LRMS (EI 70 ev),  $m/z$  (%): 289 (18), 288 (100), 219 (85), 91 (45), 65 (19); HRMS (ESI) calcd for  $\text{C}_{16}\text{H}_{12}\text{F}_3\text{N}_2^+$  ( $[\text{M} + \text{H}]^+$ ) 289.0947, found 289.0943.

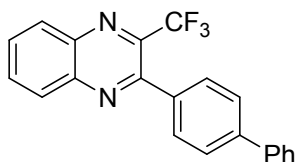


*2-(O-tolyl)-3-(trifluoromethyl)quinoxaline (2n)*: yellow solid (35.7 mg, 62% yield), mp 94–95 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.31–8.29 (m, 1H), 8.23–8.21 (m, 1H), 7.96–7.90 (m, 2H), 7.43–7.40 (m, 1H), 7.35–7.27 (m, 3H), 2.12 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  153.0, 142.6, 141.7 (q,  $J_{C-F} = 33.8$  Hz), 139.5, 136.30, 136.26, 132.6, 131.2, 130.3, 129.8, 129.34, 129.30, 128.7, 125.4, 121.2 (q,  $J_{C-F} = 273.8$  Hz), 19.7;  $^{19}\text{F}$  NMR (470 MHz,  $\text{CDCl}_3$ )  $\delta$  -63.75 (3F); LRMS (EI 70 ev),  $m/z$  (%): 288 (100), 219 (86), 116(28), 89 (49), 77 (23); HRMS (ESI) calcd for  $\text{C}_{16}\text{H}_{12}\text{F}_3\text{N}_2^+$  ( $[\text{M} + \text{H}]^+$ ) 289.0947, found 289.0943.

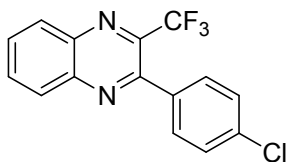


*2-(4-(Tert-butyl)phenyl)-3-(trifluoromethyl)quinoxaline (2o)*: yellow solid (50.2 mg, 76% yield), mp 134–135 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.30–8.29 (m, 1H), 8.25–8.24 (m, 1H), 7.97–7.89 (m, 2H), 7.63–7.57 (m, 4H), 1.43 (s, 9H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  152.8, 152.6, 142.6, 141.2 (q,  $J_{C-F} = 33.8$  Hz), 139.3, 134.5, 132.5, 130.9, 129.7, 129.3, 128.6, 125.3, 121.4 (q,  $J_{C-F} = 275.0$  Hz), 34.8, 31.3;  $^{19}\text{F}$  NMR (470 MHz,  $\text{CDCl}_3$ )  $\delta$  -

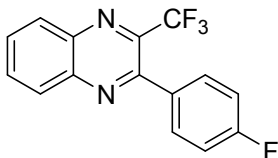
61.70 (3F); LRMS (EI 70 ev), m/z (%): 330 (24), 316 (21), 315 (100), 300 (11), 287 (18); HRMS (ESI) calcd for  $C_{19}H_{18}F_3N_2^+$  ( $[M + H]^+$ ) 331.1417, found 331.1412.



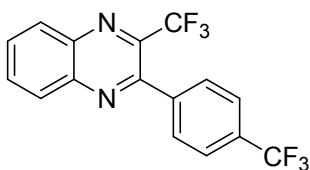
*2-([1,1'-Biphenyl]-4-yl)-3-(trifluoromethyl)quinoxaline (2p)*: white solid (42.7 mg, 61% yield), mp 158–159 °C;  $^1H$  NMR (500 MHz,  $CDCl_3$ )  $\delta$  8.29–8.27 (m, 1H), 8.24–8.22 (m, 1H), 7.96–7.88 (m, 2H), 7.77–7.72 (m, 4H), 7.69–7.67 (m, 2H), 7.50–7.47 (m, 2H), 7.41–7.38 (m, 1H);  $^{13}C$  NMR (125 MHz,  $CDCl_3$ )  $\delta$  152.3, 142.6, 142.4, 141.2 (q,  $J_{C-F} = 33.8$  Hz), 140.4, 139.3, 136.2, 132.7, 131.1, 129.7, 129.36, 129.35, 128.9, 127.7, 127.2, 127.1, 121.4 (q,  $J_{C-F} = 275.0$  Hz);  $^{19}F$  NMR (470 MHz,  $CDCl_3$ )  $\delta$  –61.58 (3F); LRMS (EI 70 ev), m/z (%): 351 (23), 350 (100), 281(60), 152 (46), 151 (19); HRMS (ESI) calcd for  $C_{21}H_{14}F_3N_2^+$  ( $[M + H]^+$ ) 351.1104, found 351.1099.



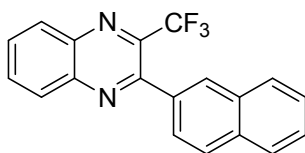
*2-(4-Chlorophenyl)-3-(trifluoromethyl)quinoxaline (2q)*: white solid (41.9 mg, 68% yield), mp 148–149 °C;  $^1H$  NMR (500 MHz,  $CDCl_3$ )  $\delta$  8.28–8.26 (m, 1H), 8.22–8.20 (m, 1H), 7.97–7.89 (m, 2H), 7.60 (d,  $J = 8.0$  Hz, 2H), 7.52–7.50 (m, 2H);  $^{13}C$  NMR (125 MHz,  $CDCl_3$ )  $\delta$  151.4, 142.5, 141.0 (q,  $J_{C-F} = 33.8$  Hz), 139.4, 135.9, 135.8, 132.8, 131.4, 130.3, 129.8, 129.3, 128.6, 121.3 (q,  $J_{C-F} = 273.8$  Hz);  $^{19}F$  NMR (470 MHz,  $CDCl_3$ )  $\delta$  –61.65 (3F); LRMS (EI 70 ev), m/z (%): 310 (32), 308 (100), 241(26), 239 (73), 102 (33); HRMS (ESI) calcd for  $C_{15}H_9ClF_3N_2^+$  ( $[M + H]^+$ ) 309.0401, found 309.0399.



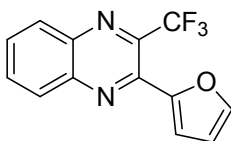
*2-(4-Fluorophenyl)-3-(trifluoromethyl)quinoxaline (2r)*: white solid (36.2 mg, 62% yield), mp 143–144 °C; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.28–8.26 (m, 1H), 8.22–8.20 (m, 1H), 7.96–7.89 (m, 2H), 7.65–7.63 (m, 2H), 7.26–7.21 (m, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 163.6 (d, *J*<sub>C-F</sub> = 248.8 Hz), 151.6, 142.5, 141.1 (q, *J*<sub>C-F</sub> = 33.8 Hz), 139.4, 133.5, 132.8, 131.3, 131.0, 129.8, 129.3, 121.4 (q, *J*<sub>C-F</sub> = 275.0 Hz), 115.5 (q, *J*<sub>C-F</sub> = 21.3 Hz); <sup>19</sup>F NMR (470 MHz, CDCl<sub>3</sub>) δ -61.74 (3F), -111.46 (1F); LRMS (EI 70 ev), *m/z* (%): 292 (94), 223 (100), 102 (35), 95 (26), 76 (26); HRMS (ESI) calcd for C<sub>15</sub>H<sub>9</sub>F<sub>4</sub>N<sub>2</sub><sup>+</sup> ([M + H]<sup>+</sup>) 293.0696, found 293.0700.



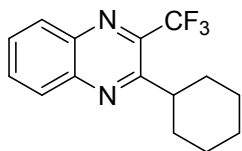
*2-(Trifluoromethyl)-3-(4-(trifluoromethyl)phenyl)quinoxaline (2s)*: yellow solid (41.0 mg, 60% yield), mp 134–135 °C; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.31–8.29 (m, 1H), 8.23–8.21 (m, 1H), 7.99–7.92 (m, 2H), 7.81–7.76 (m, 4H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 151.1, 142.5, 141.0 (q, *J*<sub>C-F</sub> = 35.0 Hz), 140.9, 139.6, 133.0, 131.7, 131.6 (q, *J*<sub>C-F</sub> = 32.5 Hz), 129.8, 129.4, 125.4, 125.3, 123.9 (q, *J*<sub>C-F</sub> = 271.3 Hz), 121.3 (q, *J*<sub>C-F</sub> = 273.8 Hz); <sup>19</sup>F NMR (470 MHz, CDCl<sub>3</sub>) δ -61.63 (3F), -61.82 (3F); LRMS (EI 70 ev), *m/z* (%): 342 (77), 273 (100), 102 (48), 76 (33), 75 (22); HRMS (ESI) calcd for C<sub>16</sub>H<sub>9</sub>F<sub>6</sub>N<sub>2</sub><sup>+</sup> ([M + H]<sup>+</sup>) 343.0664, found 343.0654.



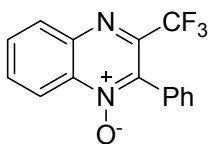
*2-(Naphthalen-2-yl)-3-(trifluoromethyl)quinoxaline (2t)*: white solid (53.1 mg, 82% yield), mp 115–116 °C; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.31–8.29 (m, 1H), 8.25–8.23 (m, 1H), 8.13 (s, 1H), 8.00 (d, *J* = 8.5 Hz, 1H), 7.96–7.89 (m, 4H), 7.76–7.74 (m, 1H), 7.60–7.55 (m, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 152.6, 142.6, 141.3 (q, *J*<sub>C-F</sub> = 35.0 Hz), 139.4, 134.7, 133.6, 132.8, 132.7, 131.2, 129.8, 129.4, 128.9, 128.6, 128.1, 127.8, 127.1, 126.7, 126.0, 121.4 (q, *J*<sub>C-F</sub> = 275.0 Hz); <sup>19</sup>F NMR (470 MHz, CDCl<sub>3</sub>) δ -61.60 (3F); LRMS (EI 70 ev), *m/z* (%): 325 (21), 324 (100), 255 (62), 153 (36), 127 (65); HRMS (ESI) calcd for C<sub>19</sub>H<sub>12</sub>F<sub>3</sub>N<sub>2</sub><sup>+</sup> ([M + H]<sup>+</sup>) 325.0947, found 325.0937.



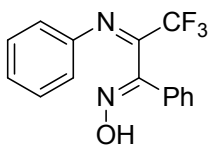
*2-(Furan-2-yl)-3-(trifluoromethyl)quinoxaline (2u)*: yellow solid (27.5 mg, 52% yield), mp 84–85 °C; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.20–8.18 (m, 1H), 7.92–7.89 (m, 1H), 7.85–7.82 (m, 1H), 7.74 (d, *J* = 2.0 Hz, 1H), 7.34 (d, *J* = 3.5 Hz, 1H), 6.65 (dd, *J*<sub>1</sub> = 2.0 Hz, *J*<sub>2</sub> = 3.5 Hz, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 149.3, 145.6, 142.5, 141.5, 138.70 (q, *J*<sub>C-F</sub> = 35.0 Hz), 138.65, 132.8, 130.9, 129.8, 129.1, 121.3 (q, *J*<sub>C-F</sub> = 273.8 Hz), 114.8, 112.3; <sup>19</sup>F NMR (470 MHz, CDCl<sub>3</sub>) δ -64.97 (3F); LRMS (EI 70 ev), *m/z* (%): 264 (100), 195 (41), 140 (22), 101 (23), 76 (17); HRMS (ESI) calcd for C<sub>13</sub>H<sub>8</sub>F<sub>3</sub>N<sub>2</sub>O<sup>+</sup> ([M + H]<sup>+</sup>) 265.0583, found 265.0575.



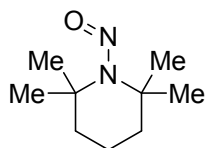
*2-Cyclohexyl-3-(trifluoromethyl)quinoxaline (2v)*: yellow solid (29.7 mg, 53% yield), mp 76–78 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.57–8.55 (m, 1H), 8.18–8.16 (m, 1H), 7.86–7.82 (m, 2H), 3.23 (s, 1H), 2.72 (d,  $J = 11.0$  Hz, 2H), 1.92–1.90 (m, 2H), 1.78–1.77 (m, 1H), 1.62–1.61 (m, 1H), 1.45–1.39 (m, 4H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  144.8, 144.3 (q,  $J_{\text{C-F}} = 32.5$  Hz), 141.6, 138.7, 132.1, 131.6, 130.7, 121.3 (q,  $J_{\text{C-F}} = 273.8$  Hz), 118.9, 40.0, 26.3, 25.5, 24.4;  $^{19}\text{F}$  NMR (470 MHz,  $\text{CDCl}_3$ )  $\delta$  -64.09 (3F); LRMS (EI 70 ev),  $m/z$  (%): 288 (100), 219 (86), 116 (28), 89 (49), 77 (23); HRMS (ESI) calcd for  $\text{C}_{15}\text{H}_{16}\text{F}_3\text{N}_2^+$  ( $[\text{M} + \text{H}]^+$ ) 281.1260, found 281.1274.



*2-Phenyl-3-(trifluoromethyl)quinoxaline 1-oxide (3a)*: yellow solid,  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.61–8.59 (m, 1H), 8.19–8.27 (m, 1H), 7.94–7.86 (m, 2H), 7.57–7.56 (m, 3H), 7.43–7.42 (m, 2H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  144.4 (q,  $J_{\text{C-F}} = 33.8$  Hz), 142.7, 140.1, 138.0, 132.39, 132.36, 130.9, 130.2, 129.6, 128.7, 128.2, 120.6 (q,  $J_{\text{C-F}} = 273.8$  Hz), 119.4;  $^{19}\text{F}$  NMR (470 MHz,  $\text{CDCl}_3$ )  $\delta$  -62.79 (3F); LRMS (EI 70 ev),  $m/z$  (%): 290 (40), 289 (71), 274 (96), 205 (100), 77 (35); HRMS (ESI) calcd for  $\text{C}_{15}\text{H}_{10}\text{F}_3\text{N}_2\text{O}^+$  ( $[\text{M} + \text{H}]^+$ ) 291.0740, found 291.0741.



(1*E*,2*E*)-3,3,3-Trifluoro-1-phenyl-2-(phenylimino)propan-1-one oxime (**4a**): yellow solid, <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.33 (s, 1H), 7.38 (d, *J* = 7.0 Hz, 2H), 7.34–7.31 (m, 1H), 7.29–7.26 (m, 2H), 7.23–7.20 (m, 2H), 7.11–7.08 (m, 1H), 7.01 (d, *J* = 7.5 Hz, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 153.4 (q, *J*<sub>C-F</sub> = 36.3 Hz), 151.3, 146.4, 130.6, 130.1, 128.82, 128.78, 127.0, 126.2, 119.4, 118.5 (q, *J*<sub>C-F</sub> = 277.5 Hz); LRMS (EI 70 eV), *m/z* (%): 292 (17), 275 (47), 193 (56), 104 (87), 77 (100); HRMS (ESI) calcd for C<sub>15</sub>H<sub>12</sub>F<sub>3</sub>N<sub>2</sub>O<sup>+</sup> ([M + H]<sup>+</sup>) 293.0896, found 293.0901.



2,2,6,6-Tetramethyl-1-nitrosopiperidine (**5**)<sup>[3]</sup>: yellow liquid, <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 1.82 (t, *J* = 6.0 Hz, 2H), 1.70–1.65 (m, 2H), 1.63–1.61 (m, 8H), 1.41 (s, 6H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 62.0, 60.1, 41.5, 38.8, 31.8, 26.0, 16.1; LRMS (EI 70 eV), *m/z* (%): 170 (45), 140 (58), 69 (100), 56 (46), 55 (55); HRMS (ESI) calcd for C<sub>9</sub>H<sub>19</sub>N<sub>2</sub>O<sup>+</sup> ([M + H]<sup>+</sup>) 171.1492, found 171.1487.

## (C). Referances

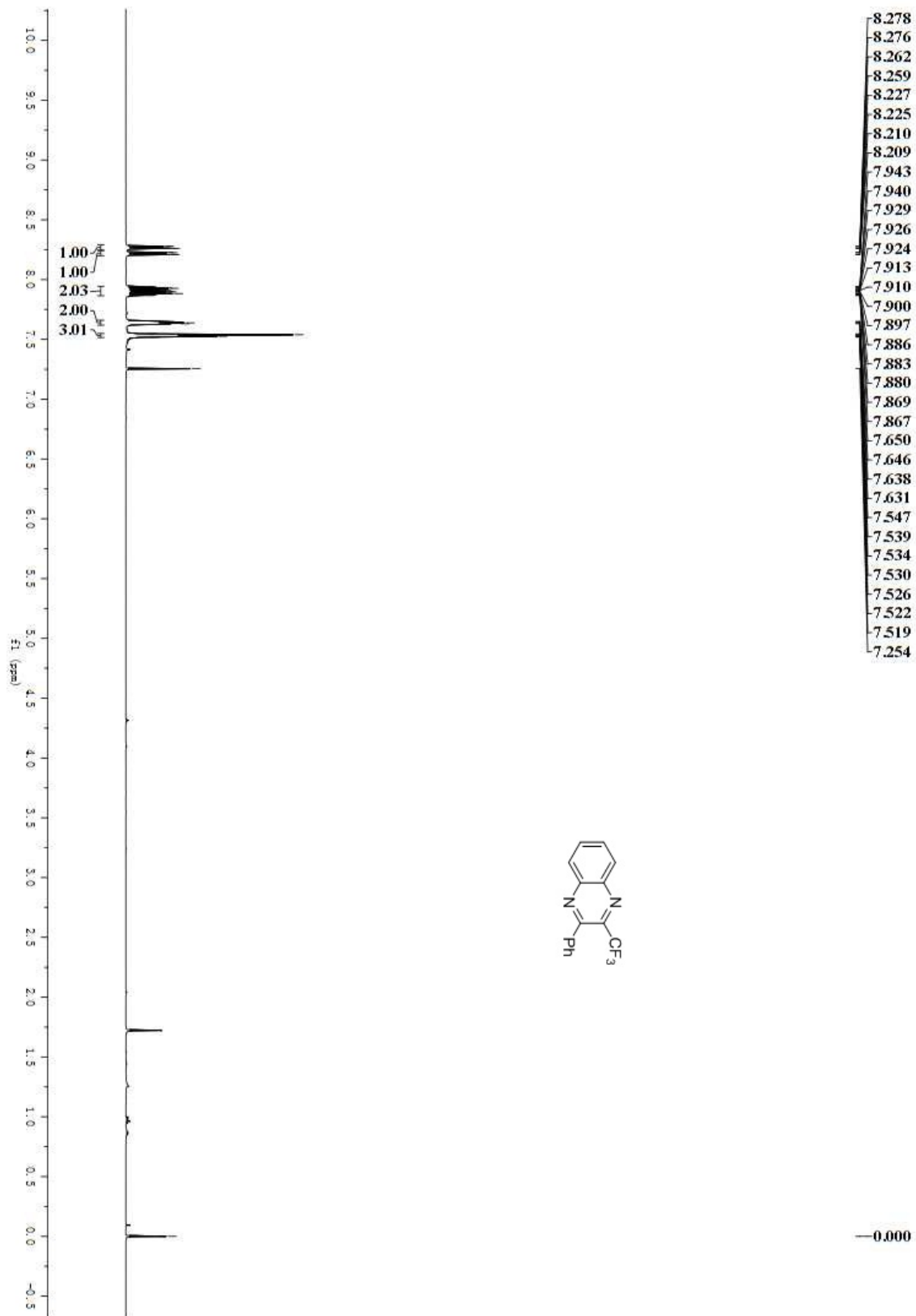
- [1] (a) A. C. Tadd, A. Matsuno, M. R. Fielding and M. C. Willis, *Org. Lett.*, 2009, **11**,583; (b) M. C. Harris, X.-H. Huang and S. L. Buchwald, *Org. Lett.*, 2002, **4**, 2885; (c) S. M. Raders, J. N. Moore, J. K. Parks, A. D. Miller, T. M. Leißing, S. P. Kelley, R. D. Rogers and K. H. Shaughness, *J. Org. Chem.*, 2013, **78**, 4649.
- [2] H. Watanabe, F. Yan, T. Sakai and K. Uneyama, *J. Org. Chem.*, 1994, **59**, 758.



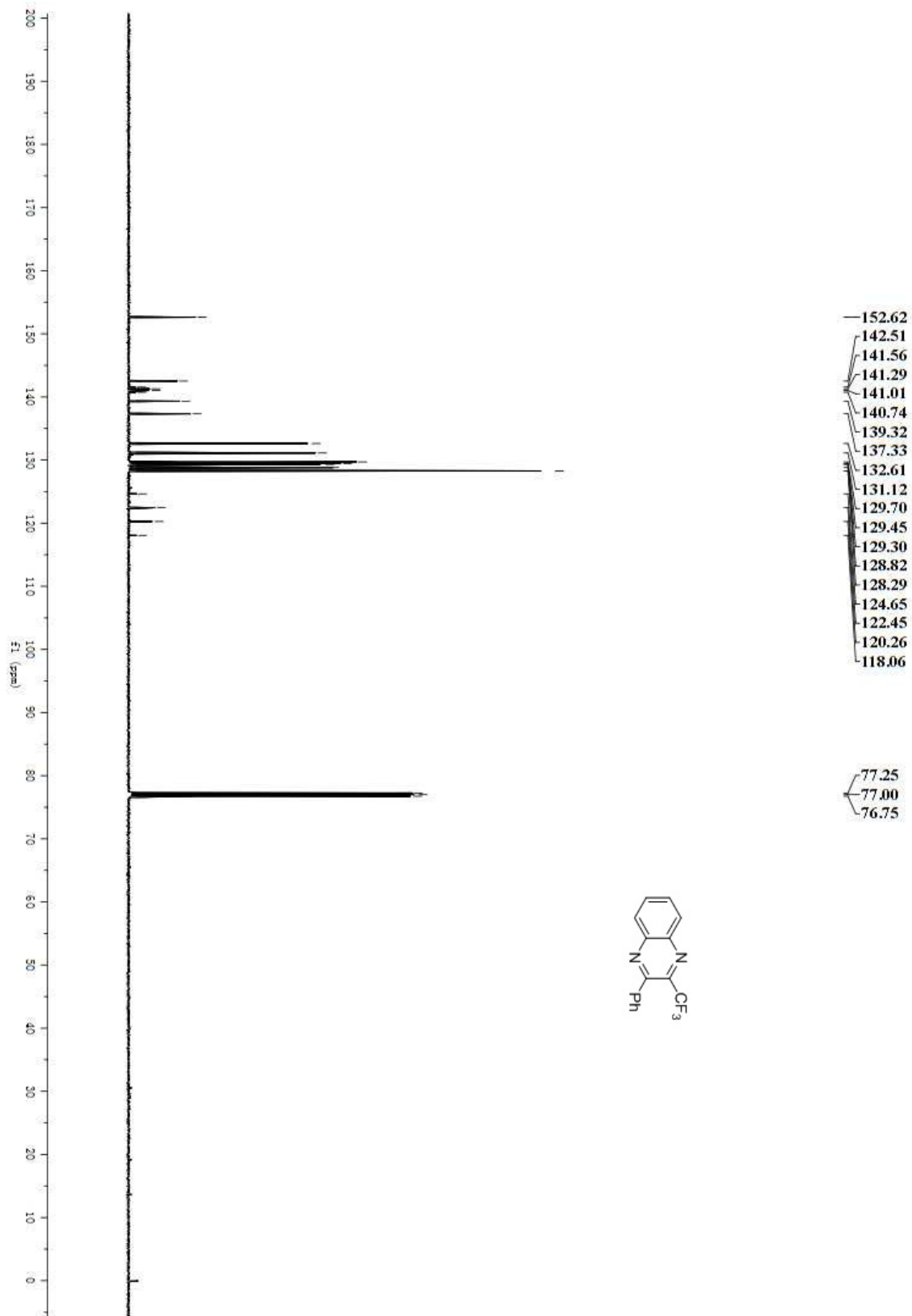
[3] G. V. Shustov, N. B. Tavakalyan, L. L. Shustova, I. I. Chervin and R. G. Kostyanovskii, *Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya*, 1980, **5**, 1058.

**(D).  $^1\text{H}$  and  $^{13}\text{C}$  NMR Spectra of compounds 2a-2v, 3a, 4a, 5**

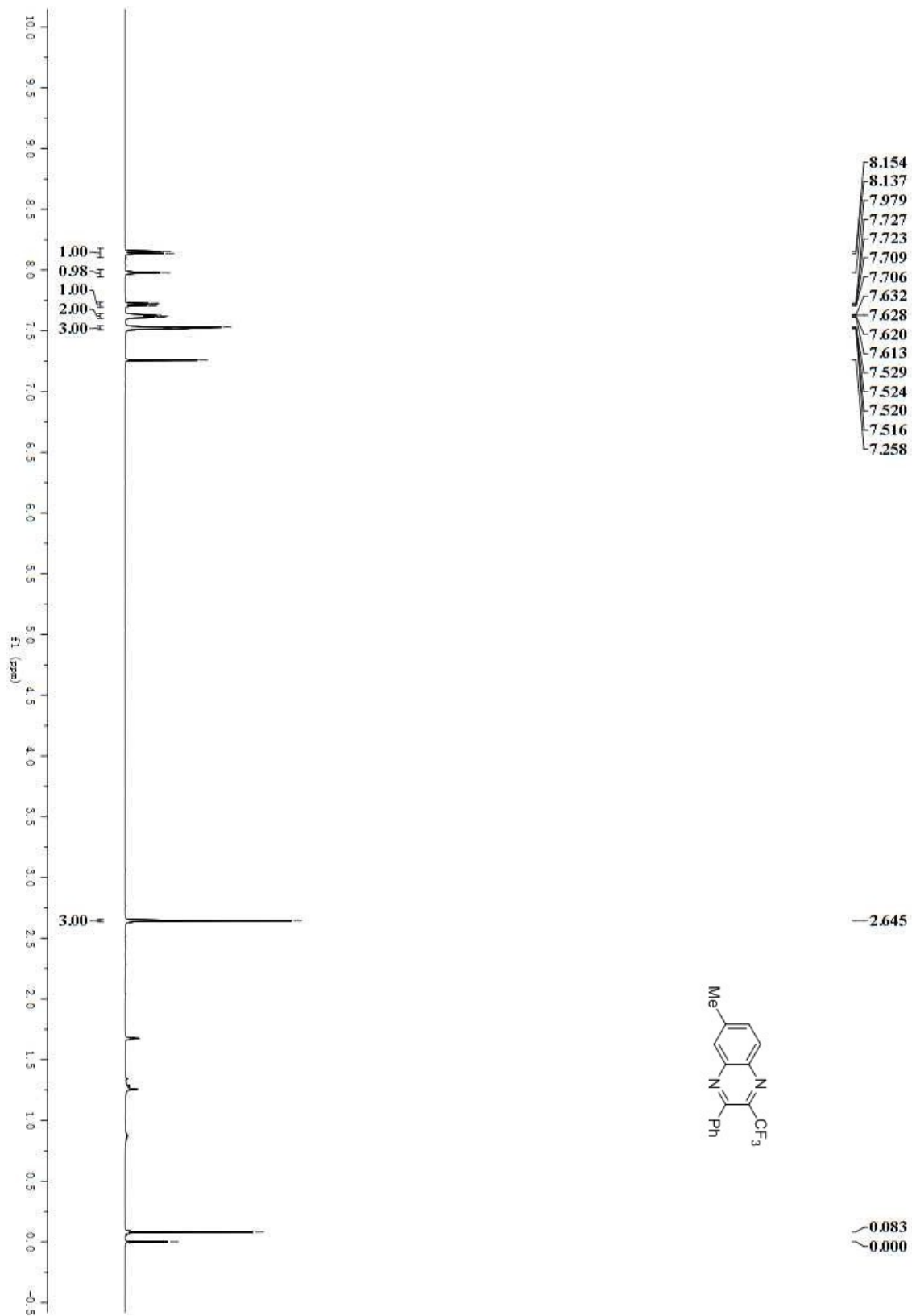
**$^1\text{H}$ -NMR: 2-Phenyl-3-(trifluoromethyl)quinoxaline (2a)**

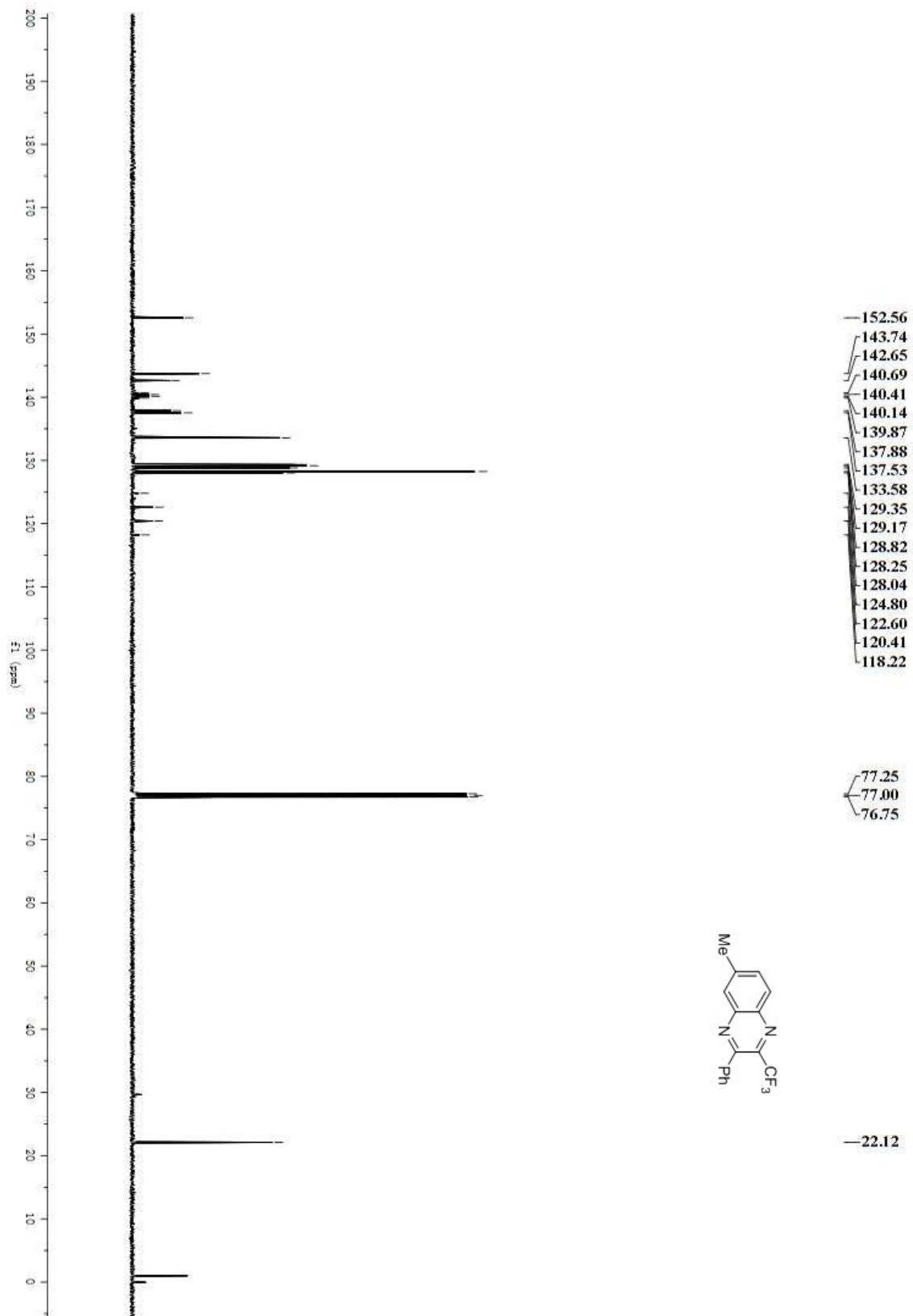


<sup>13</sup>C-NMR: 2-Phenyl-3-(trifluoromethyl)quinoxaline(2a)

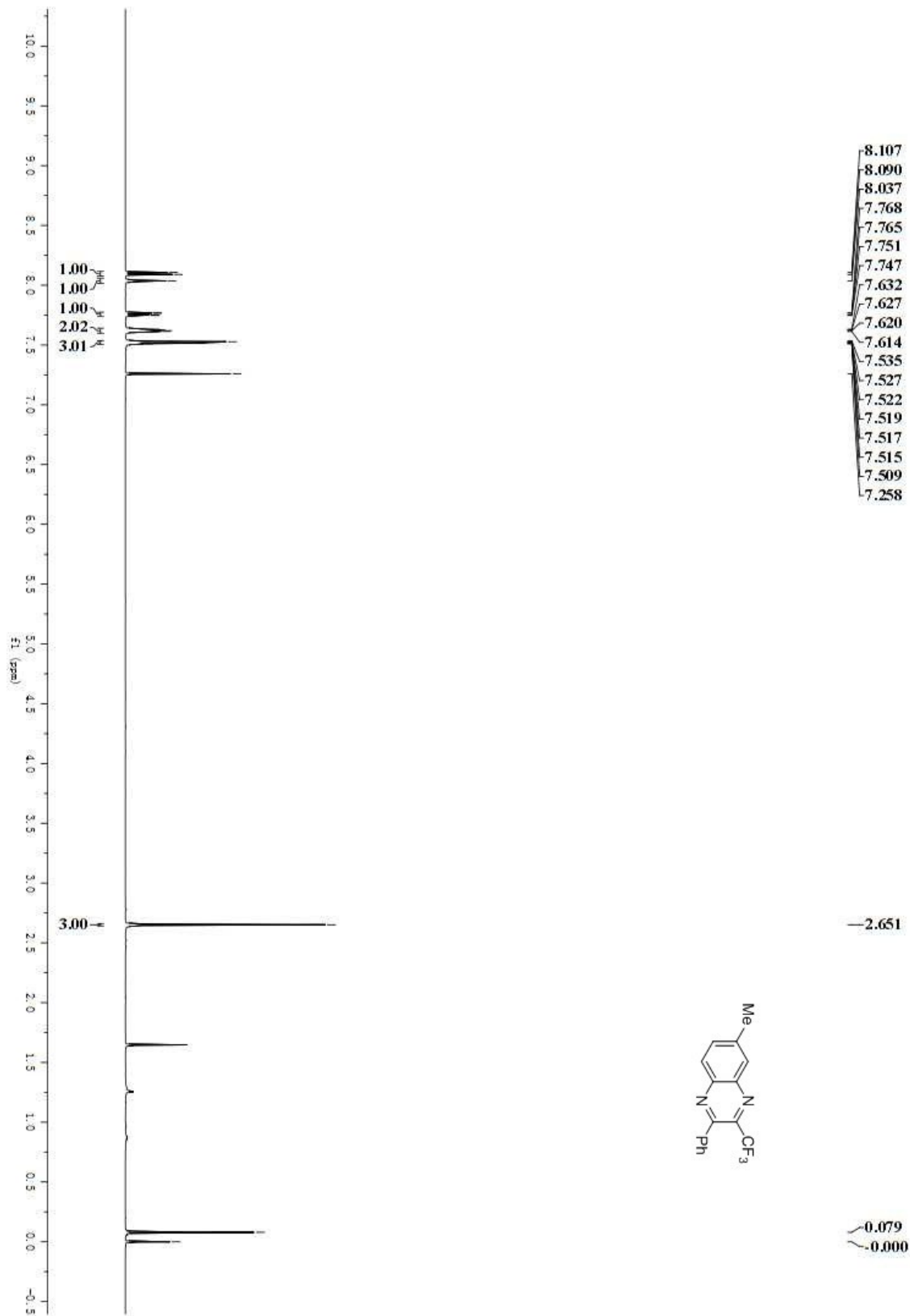


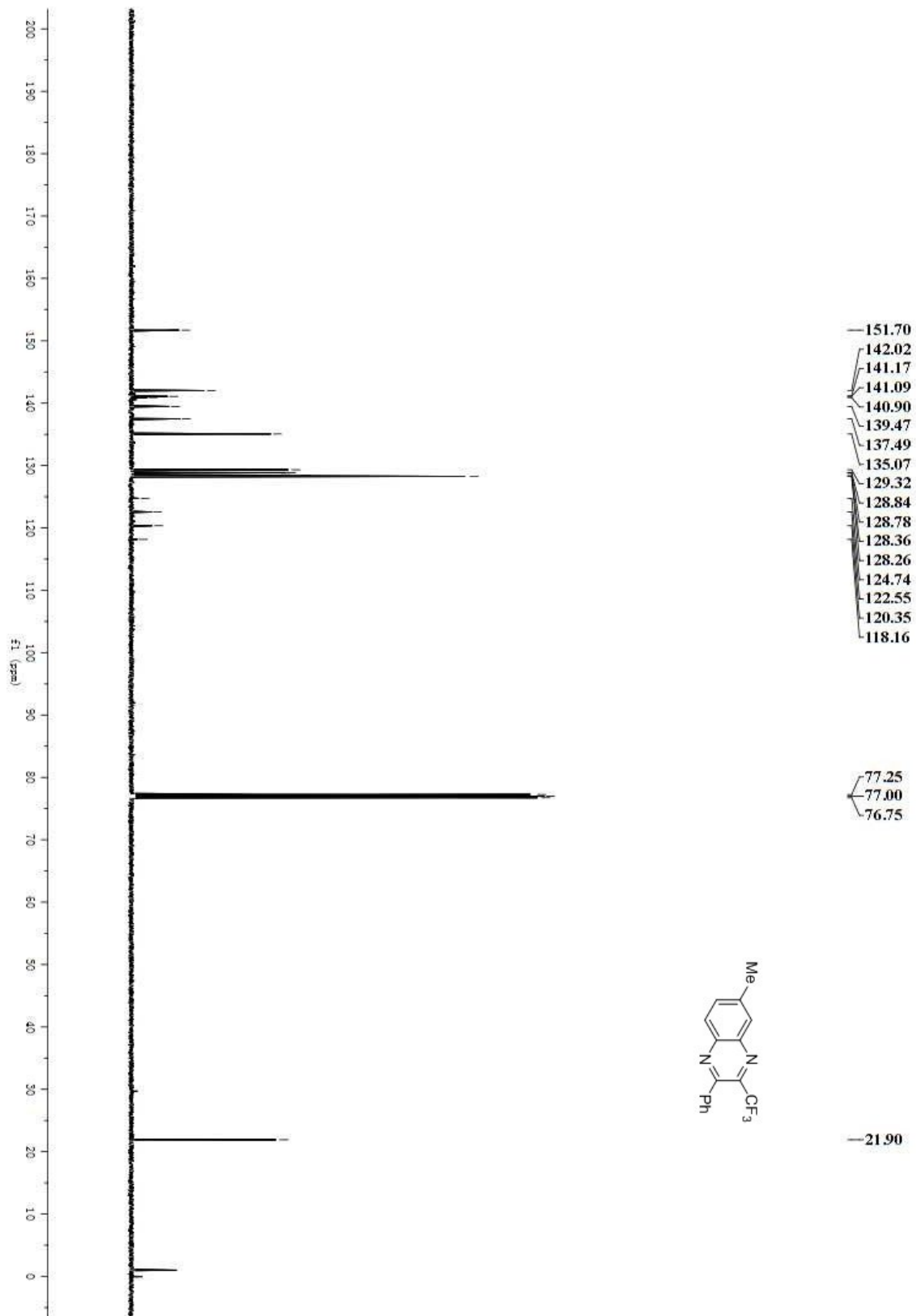
<sup>13</sup>C-NMR: 6-Methyl-3-phenyl-2-(trifluoromethyl)quinoxaline(2b)



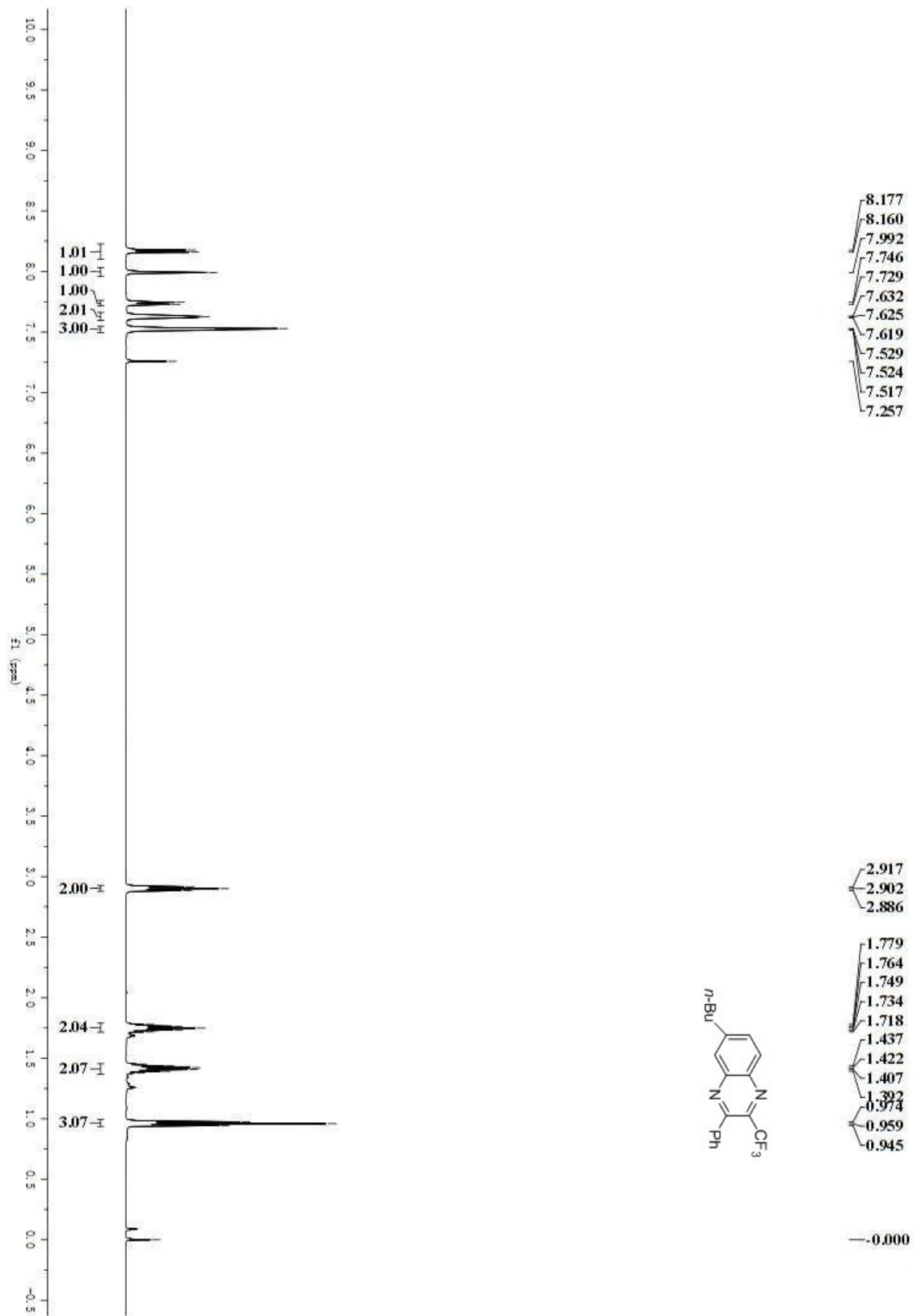


<sup>13</sup>C-NMR: 6-Methyl-2-phenyl-3-(trifluoromethyl)quinoxaline (2c)

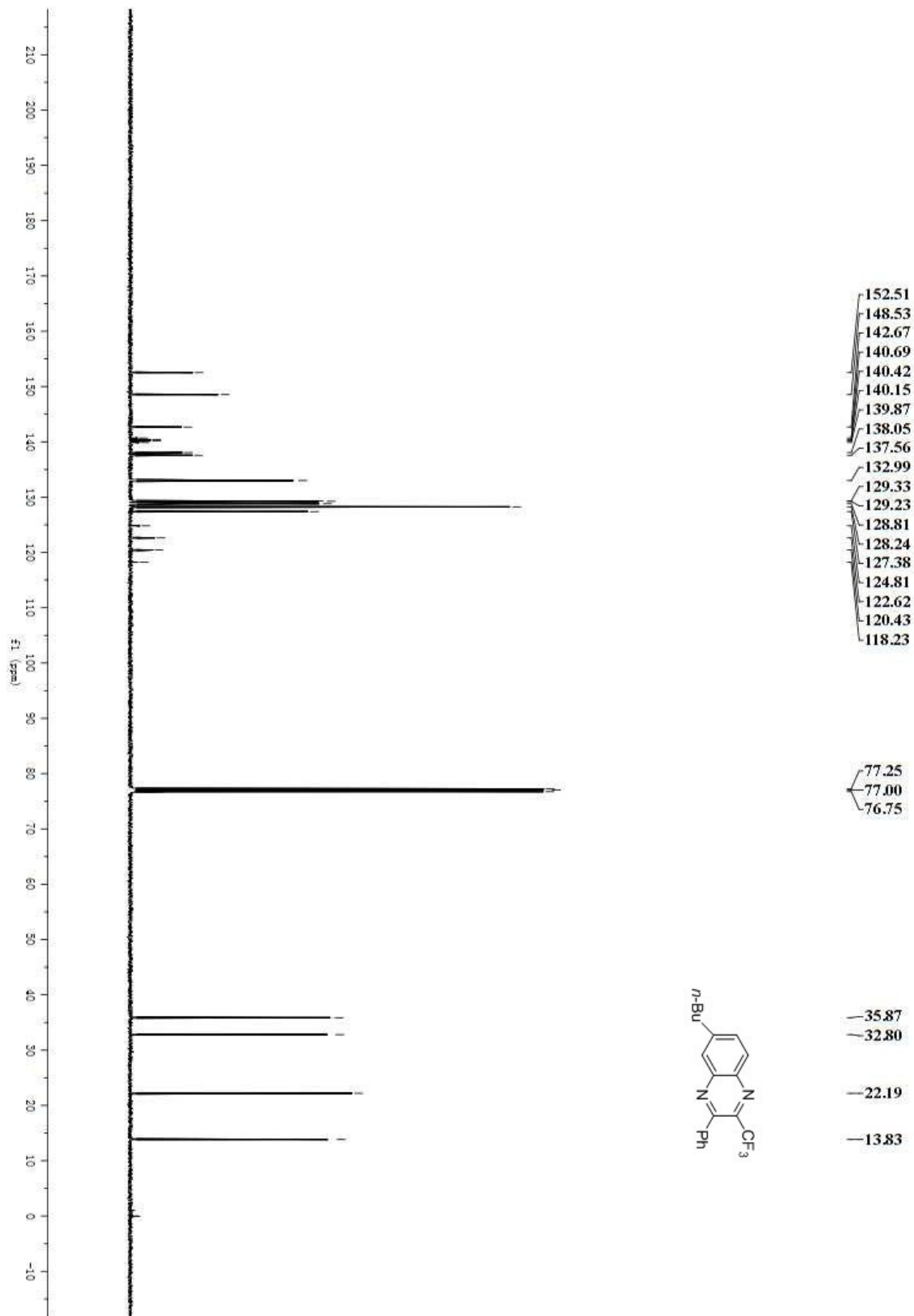




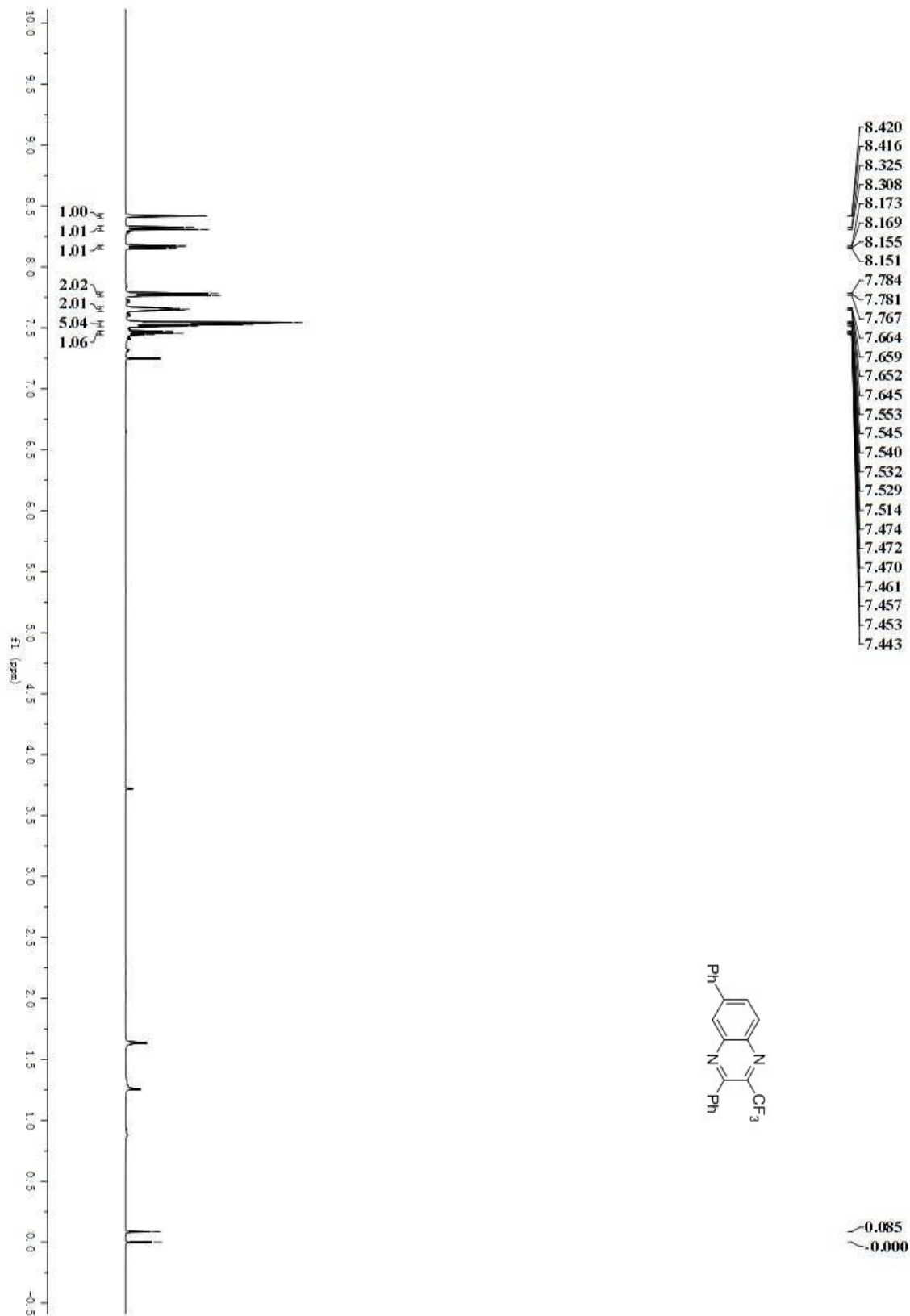
<sup>1</sup>H-NMR: 6-Butyl-3-phenyl-2-(trifluoromethyl)quinoxaline (2d)



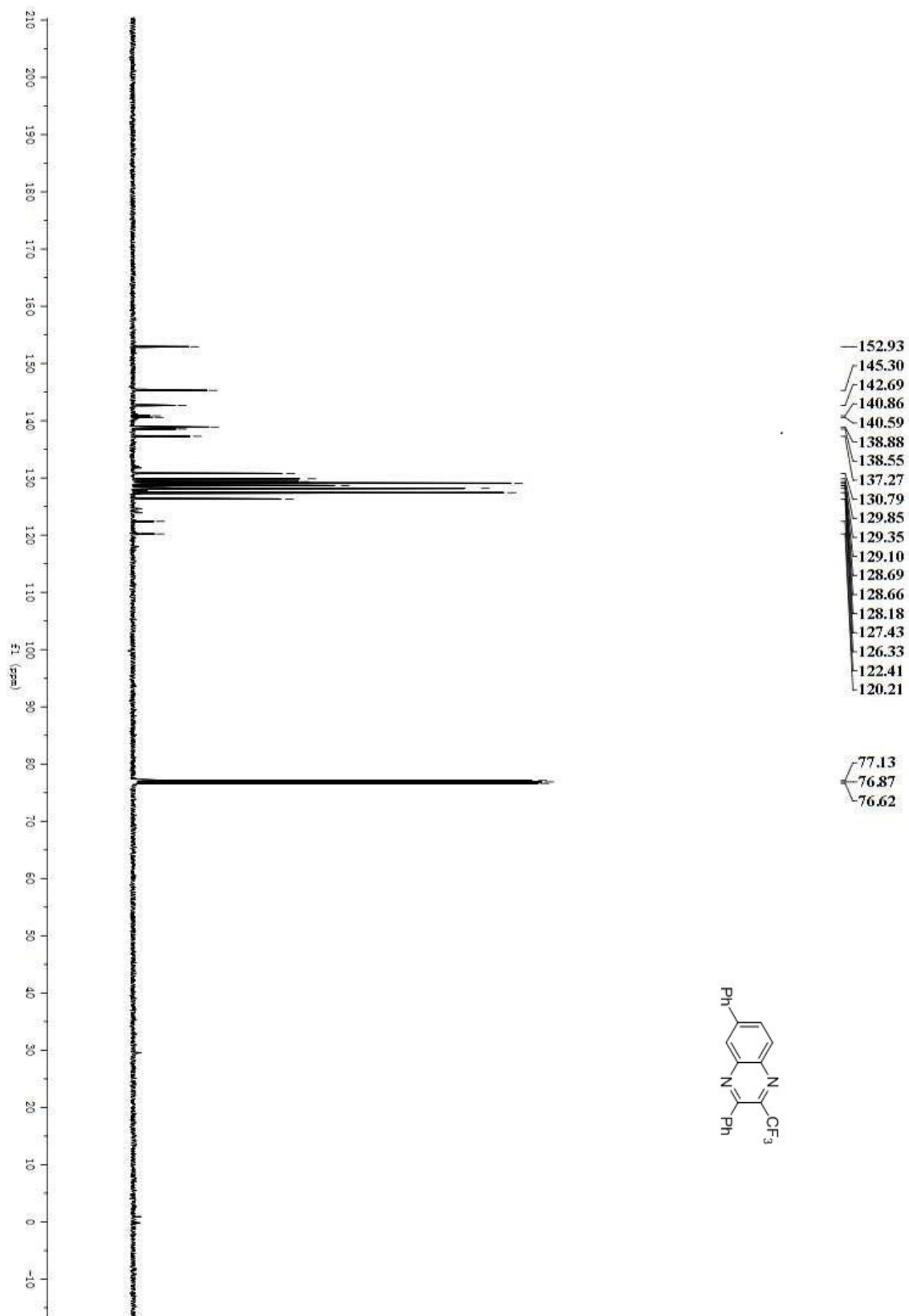




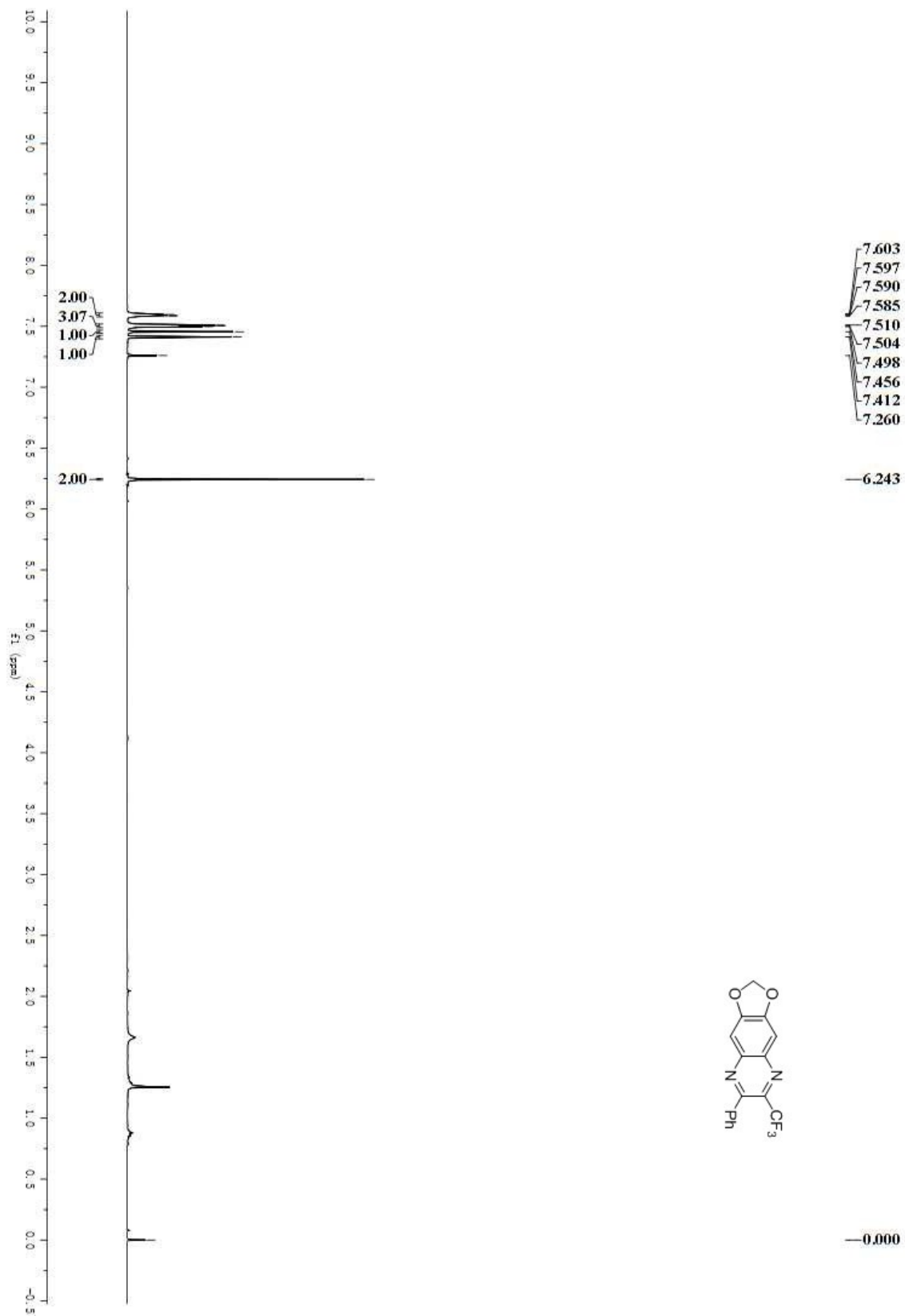
**<sup>1</sup>H-NMR: 3,6-Diphenyl-2-(trifluoromethyl)quinoxaline (2e)**



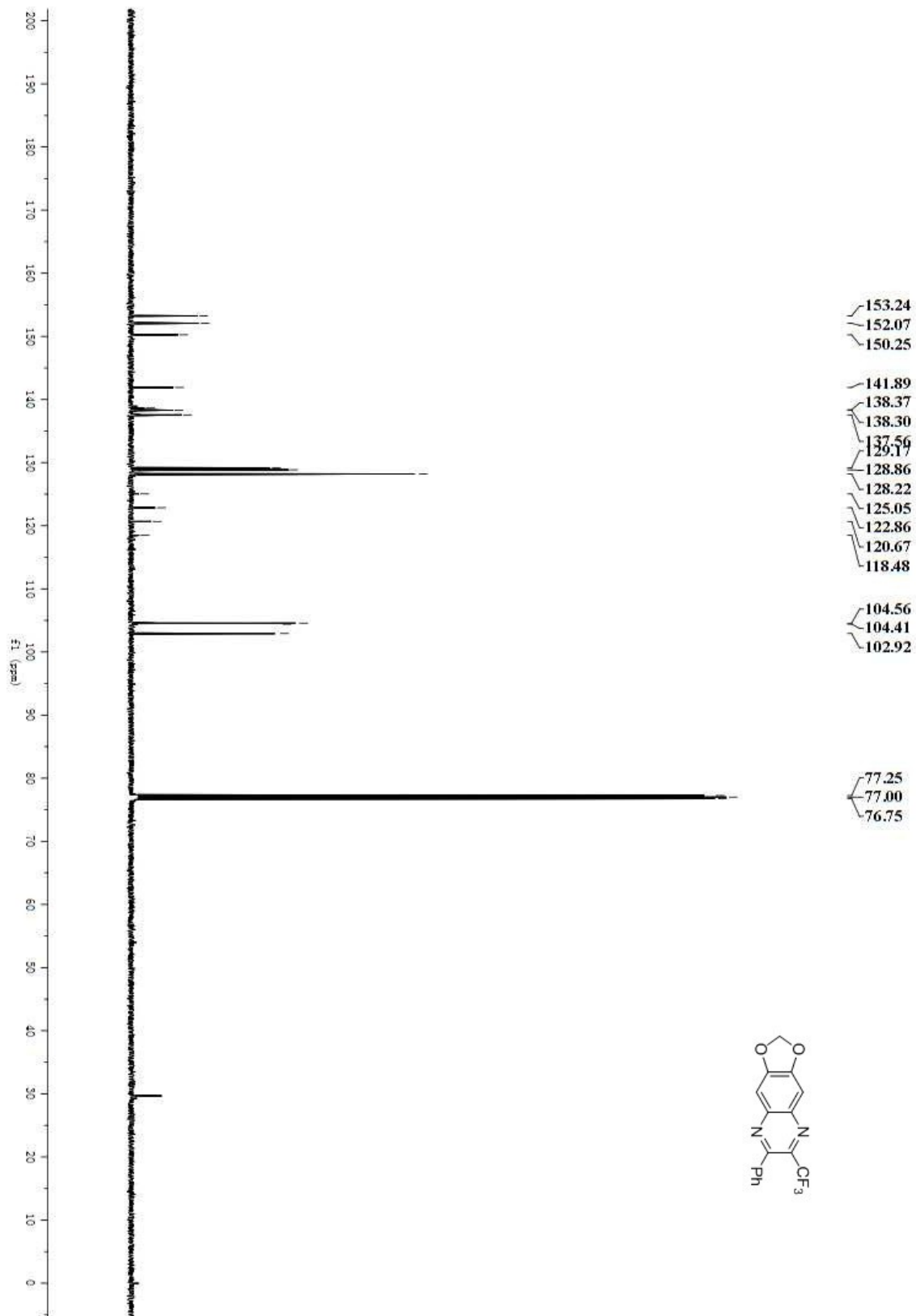
<sup>13</sup>C-NMR: 3,6-Diphenyl-2-(trifluoromethyl)quinoxaline (2e)



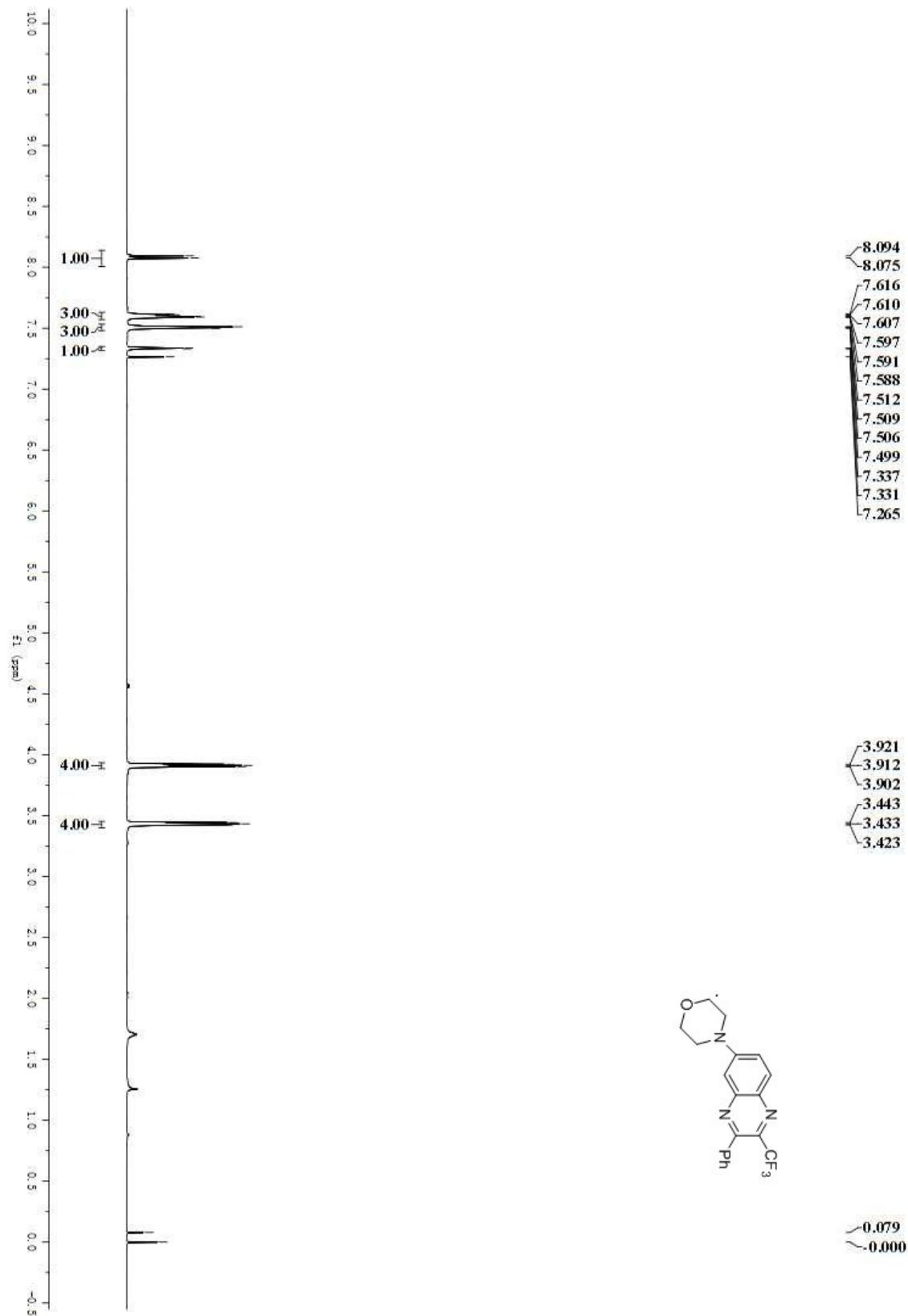
<sup>13</sup>C-NMR: 6-Phenyl-7-(trifluoromethyl)-[1,3]dioxolo[4,5-g]quinoxaline (2f)

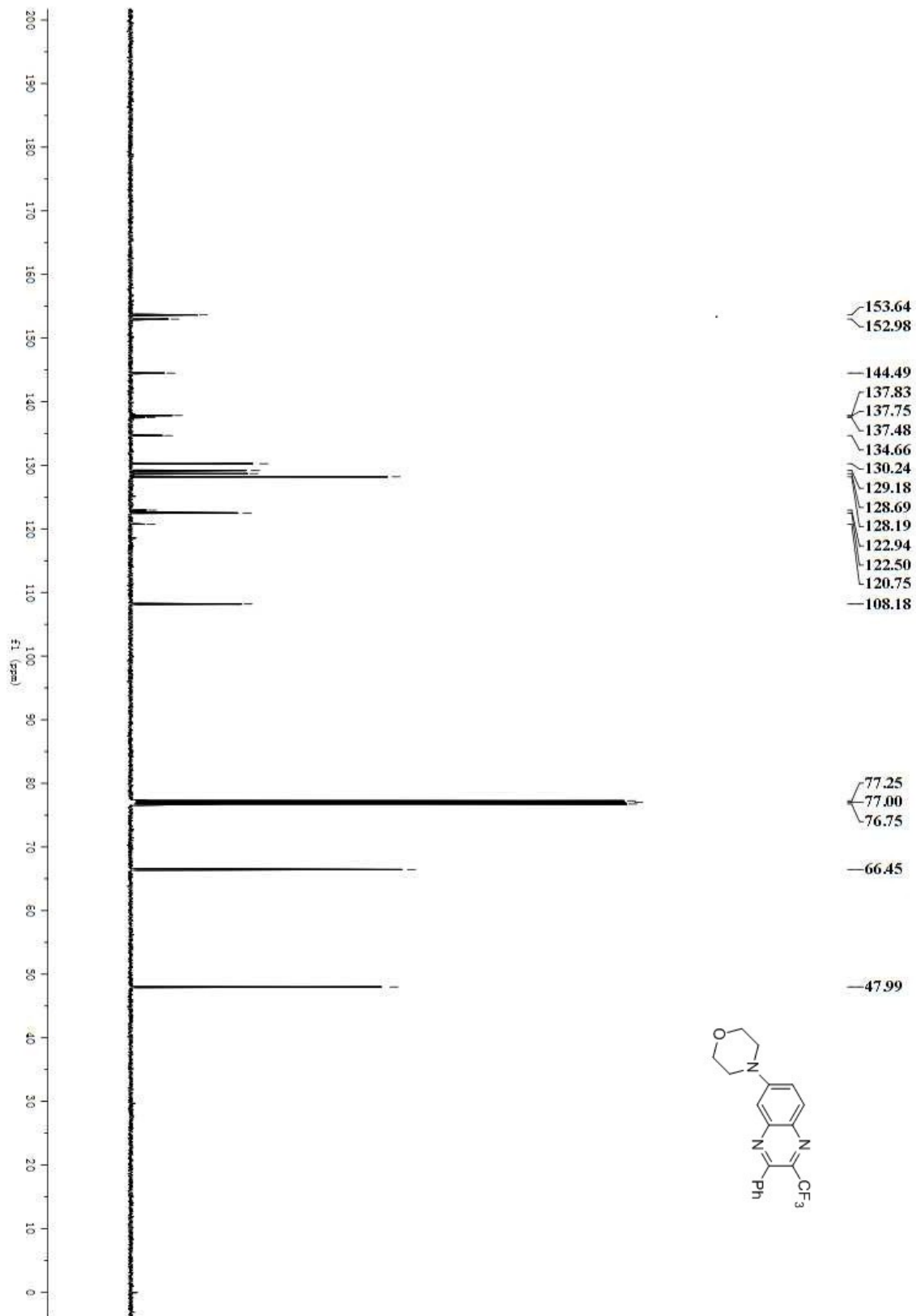


<sup>13</sup>C-NMR: 6-Phenyl-7-(trifluoromethyl)-[1,3]dioxolo[4,5-g]quinoxaline (2f)

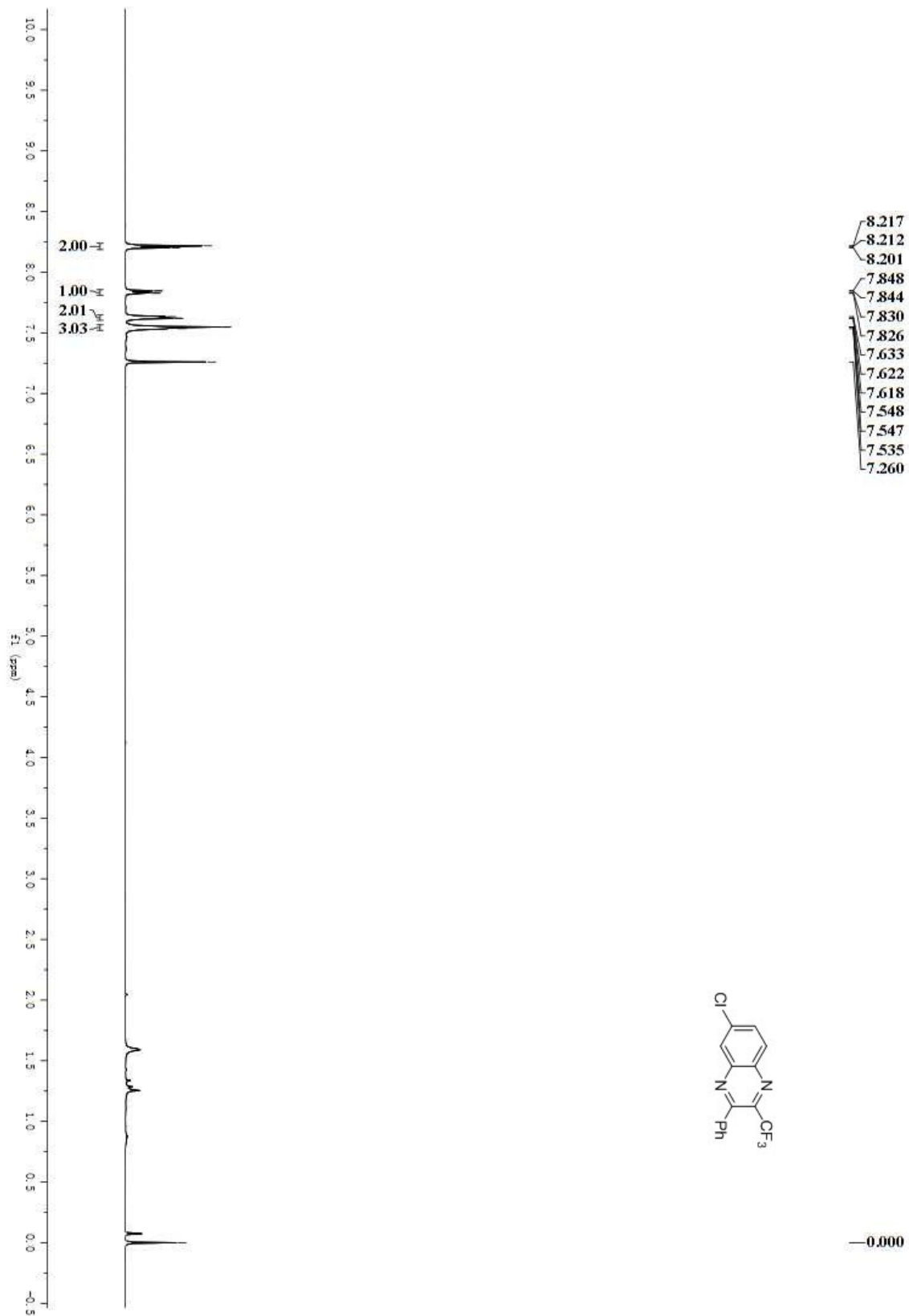


<sup>1</sup>H-NMR: 4-(3-Phenyl-2-(trifluoromethyl)quinoxalin-6-yl)morpholine (2g)



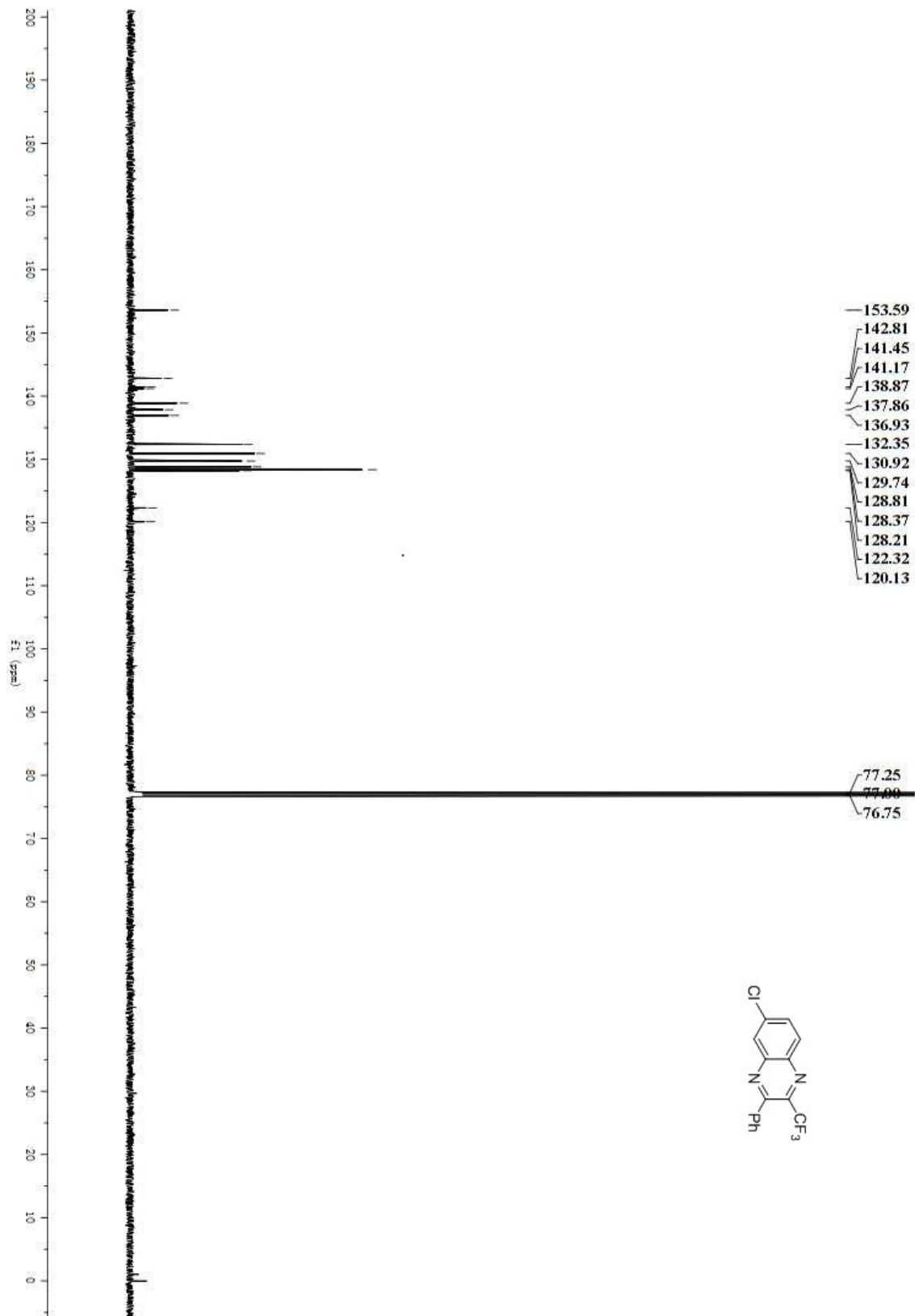


**<sup>13</sup>C-NMR: 6-Chloro-3-phenyl-2-(trifluoromethyl)quinoxaline (2h)**

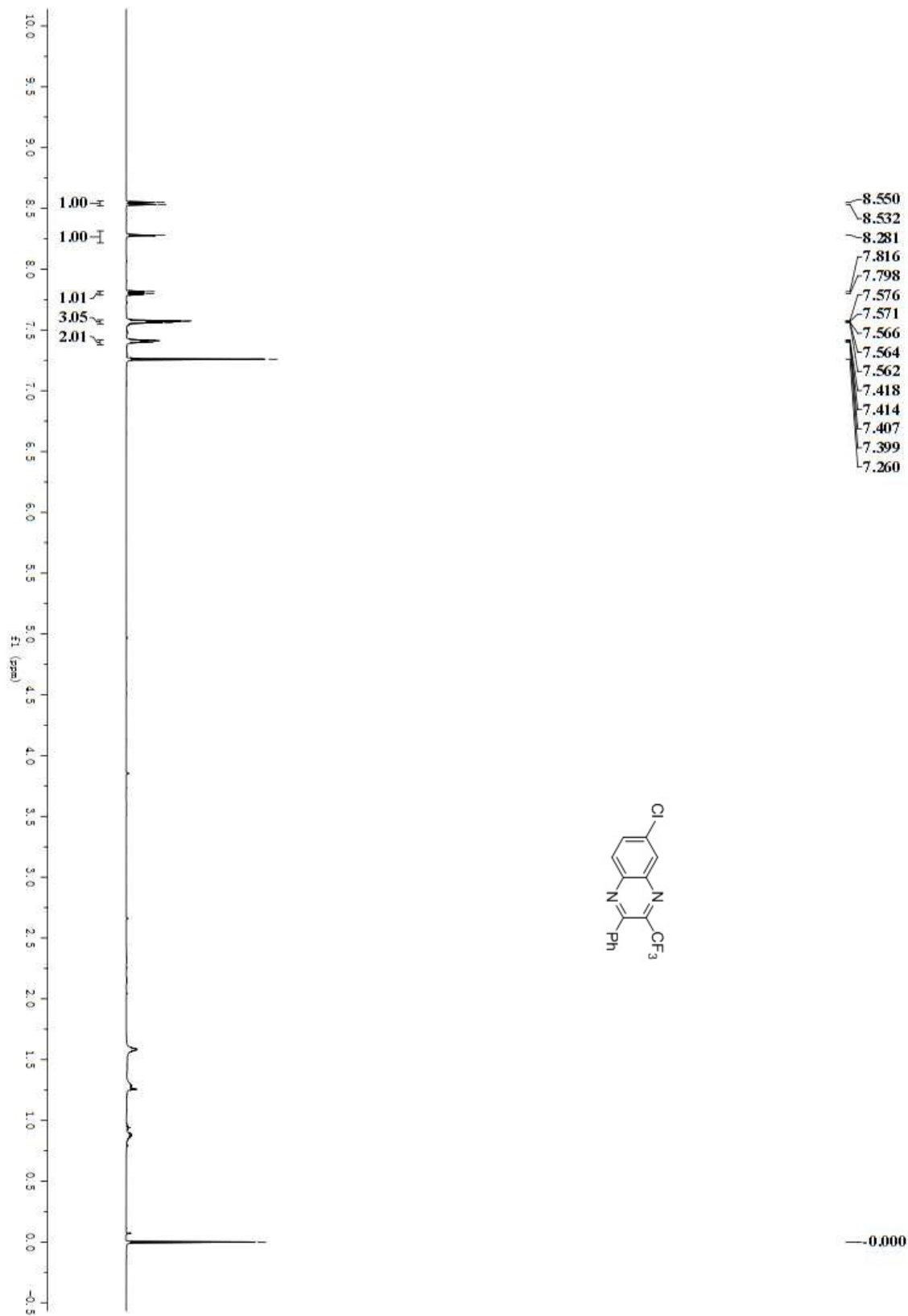


<sup>13</sup>C-NMR: 6-Chloro-3-phenyl-2-(trifluoromethyl)quinoxaline (2h)

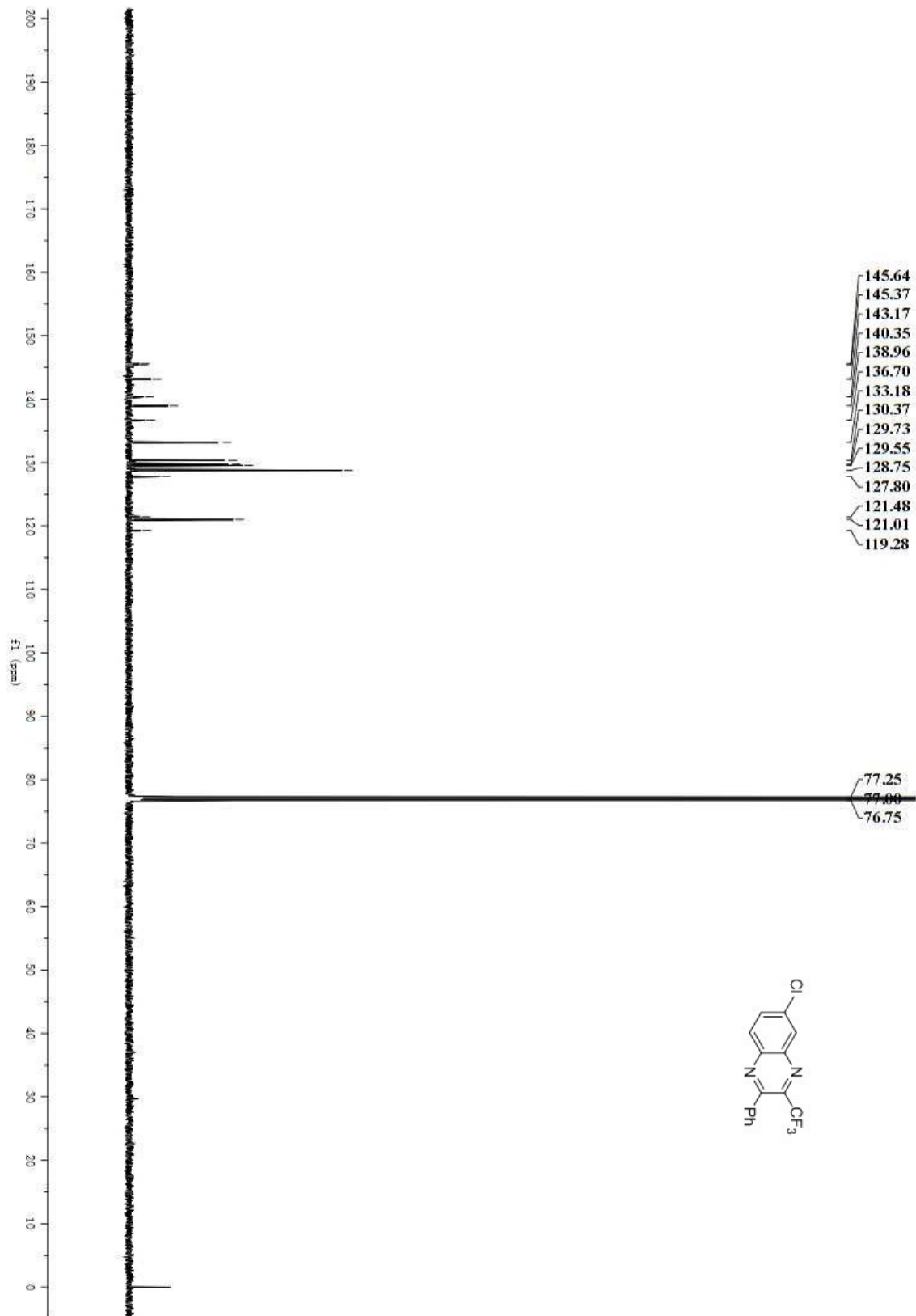




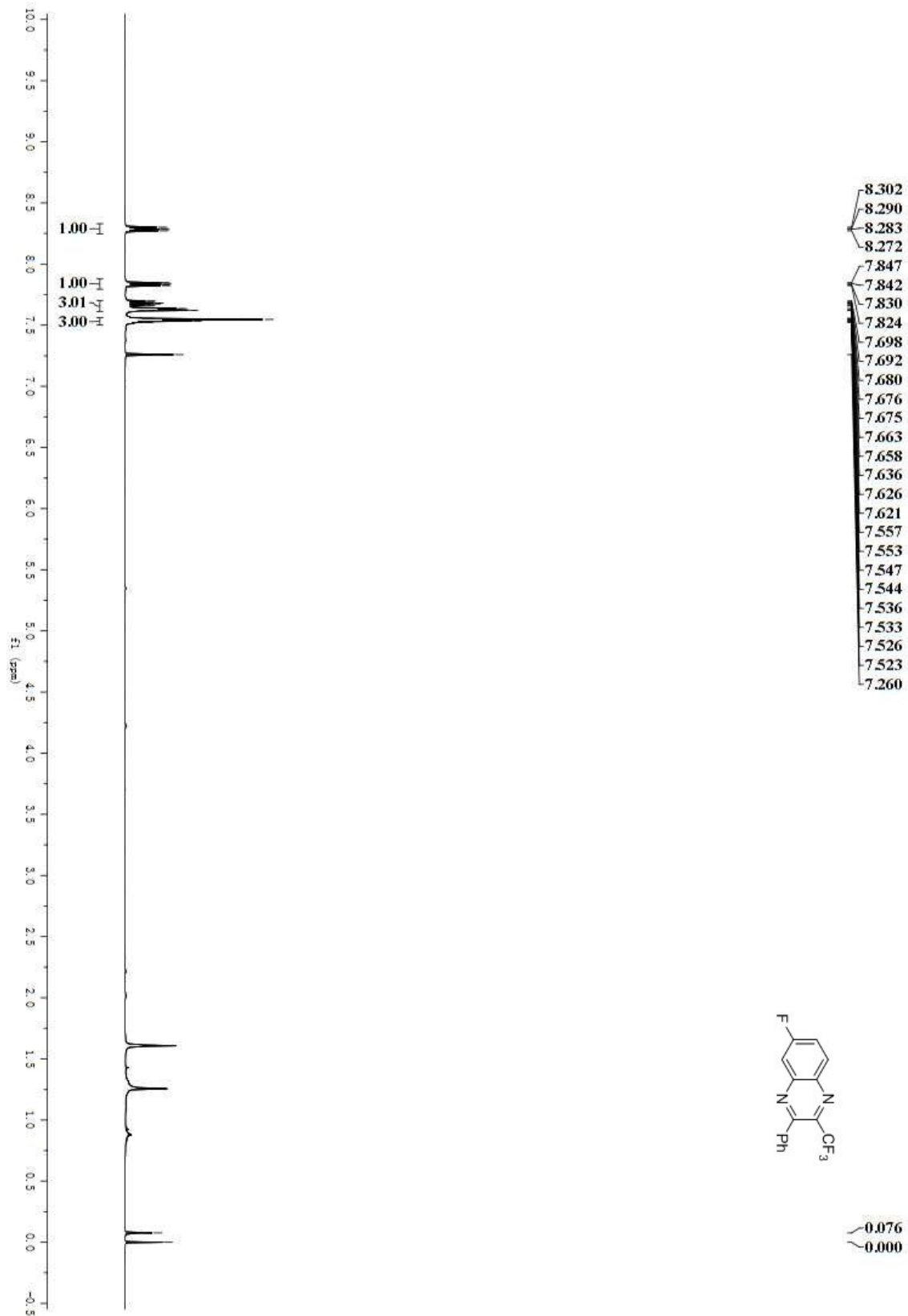
<sup>1</sup>H-NMR: 6-Chloro-2-phenyl-3-(trifluoromethyl)quinoxaline (2i)

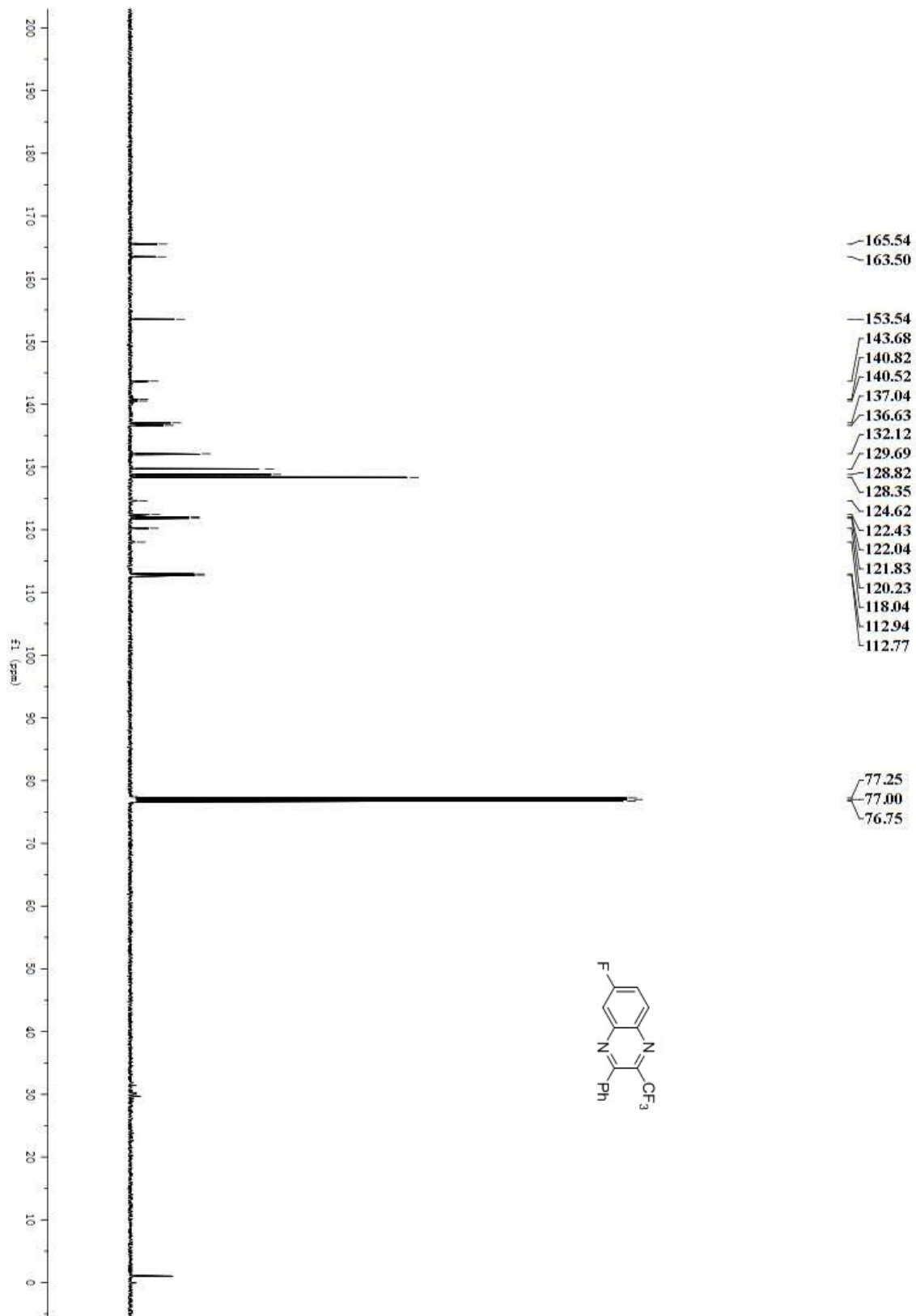


<sup>13</sup>C-NMR: 6-Chloro-2-phenyl-3-(trifluoromethyl)quinoxaline (2i)

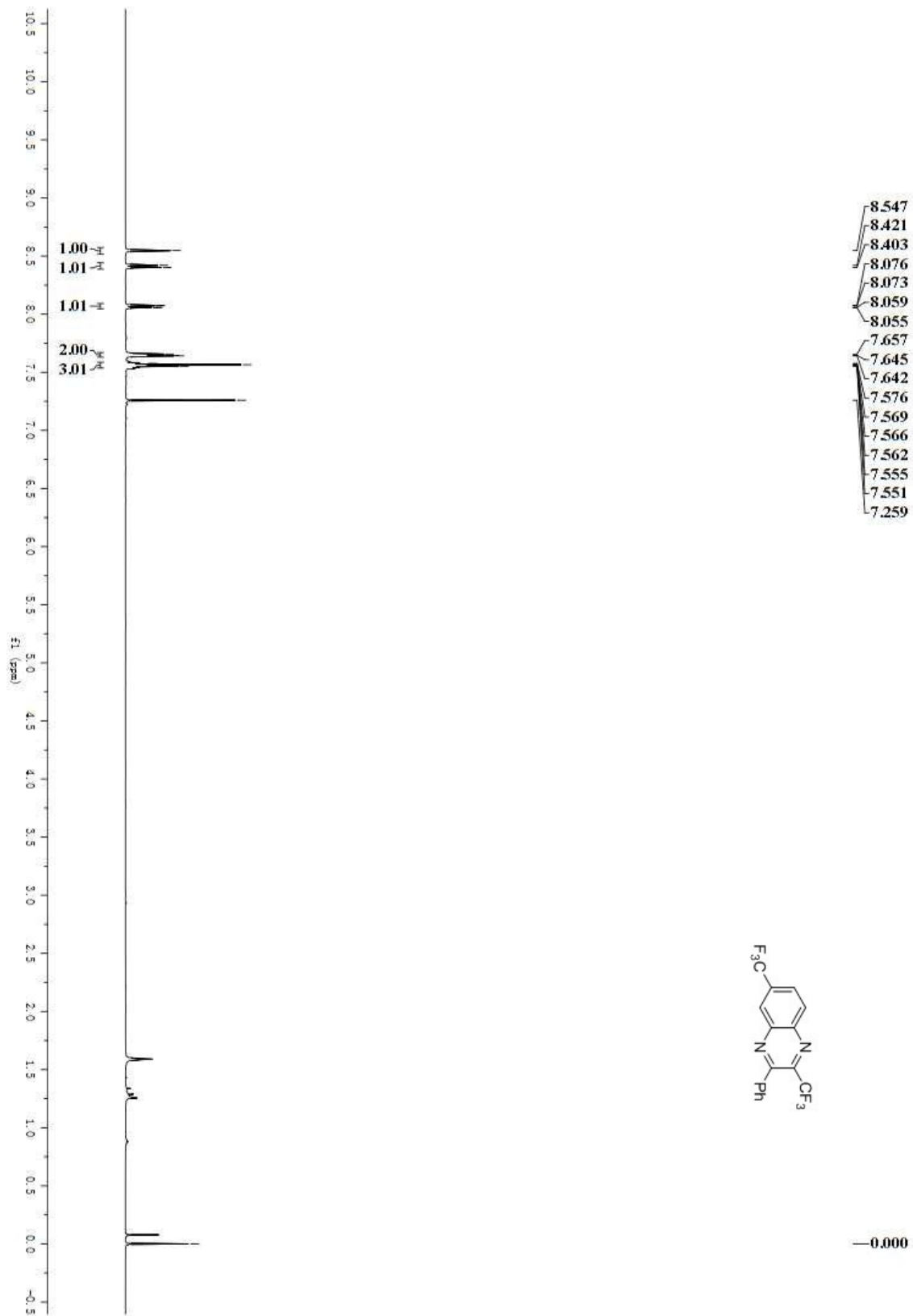


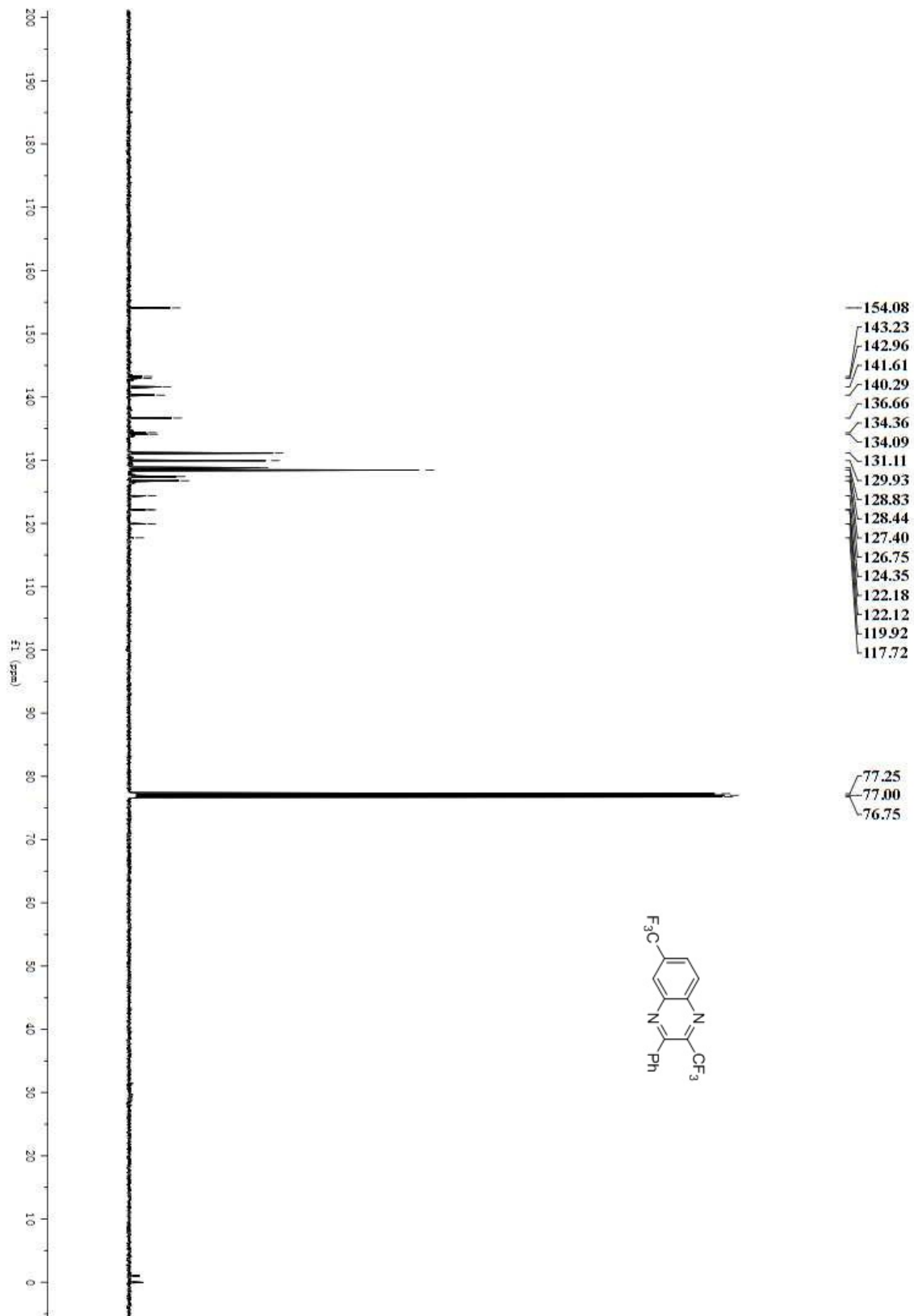
<sup>13</sup>C-NMR: 6-Fluoro-3-phenyl-2-(trifluoromethyl)quinoxaline (2j)



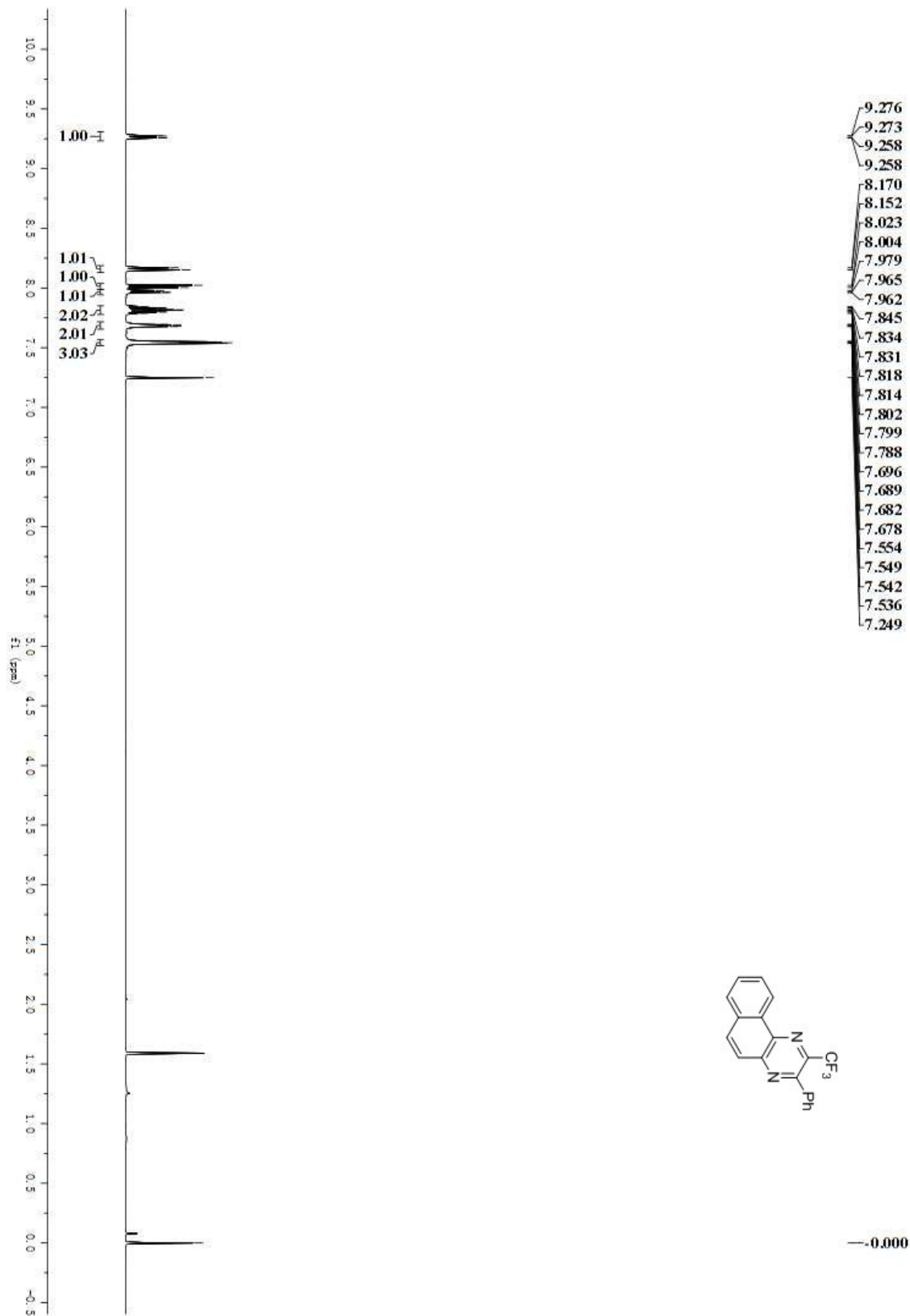


<sup>13</sup>C-NMR: 3-Phenyl-2,6-bis(trifluoromethyl)quinoxaline (2k)



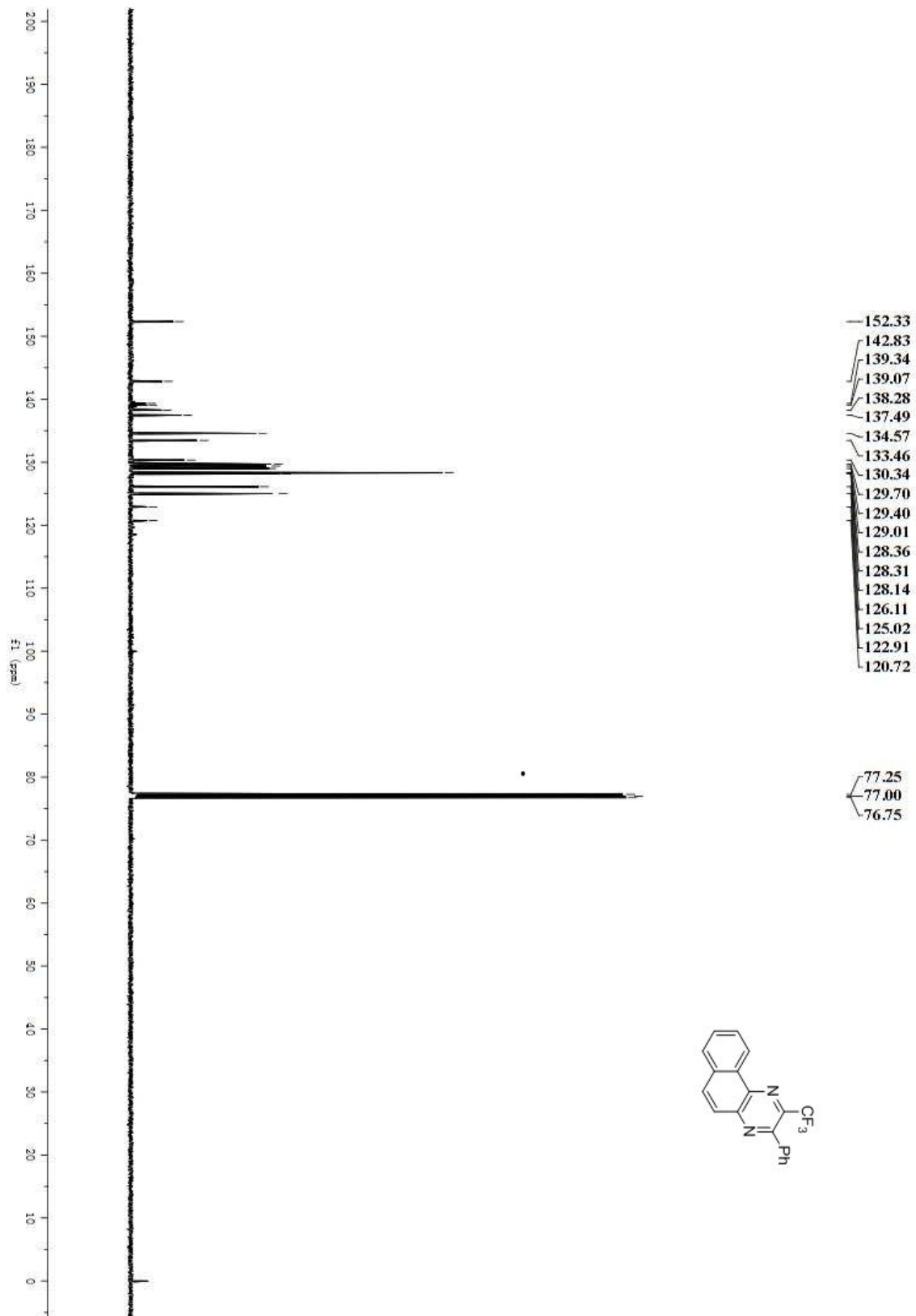


<sup>13</sup>C-NMR: 3-Phenyl-2-(trifluoromethyl)benzo[f]quinoxaline (2l)

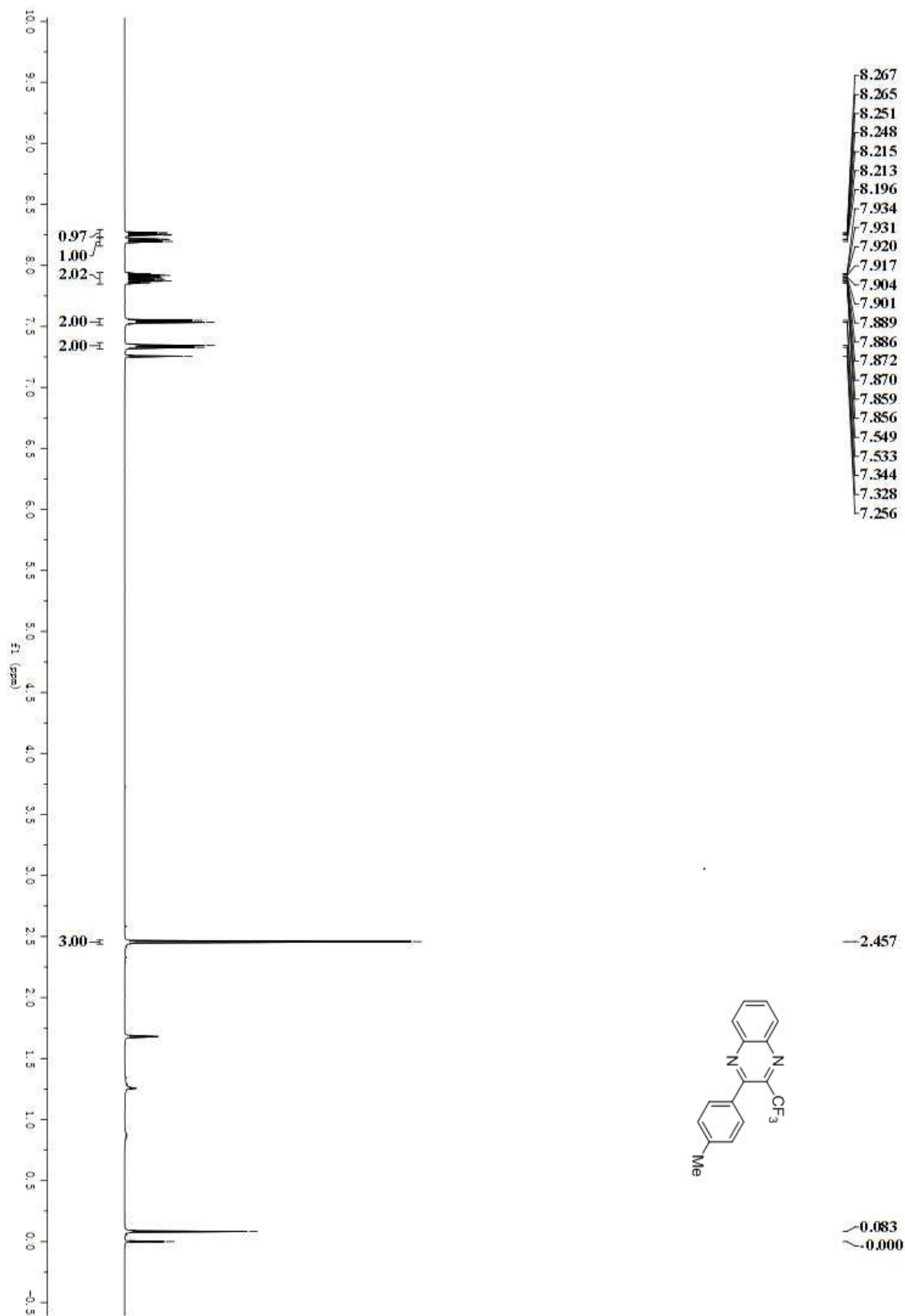


<sup>13</sup>C-NMR: 3-Phenyl-2-(trifluoromethyl)benzo[f]quinoxaline (21)

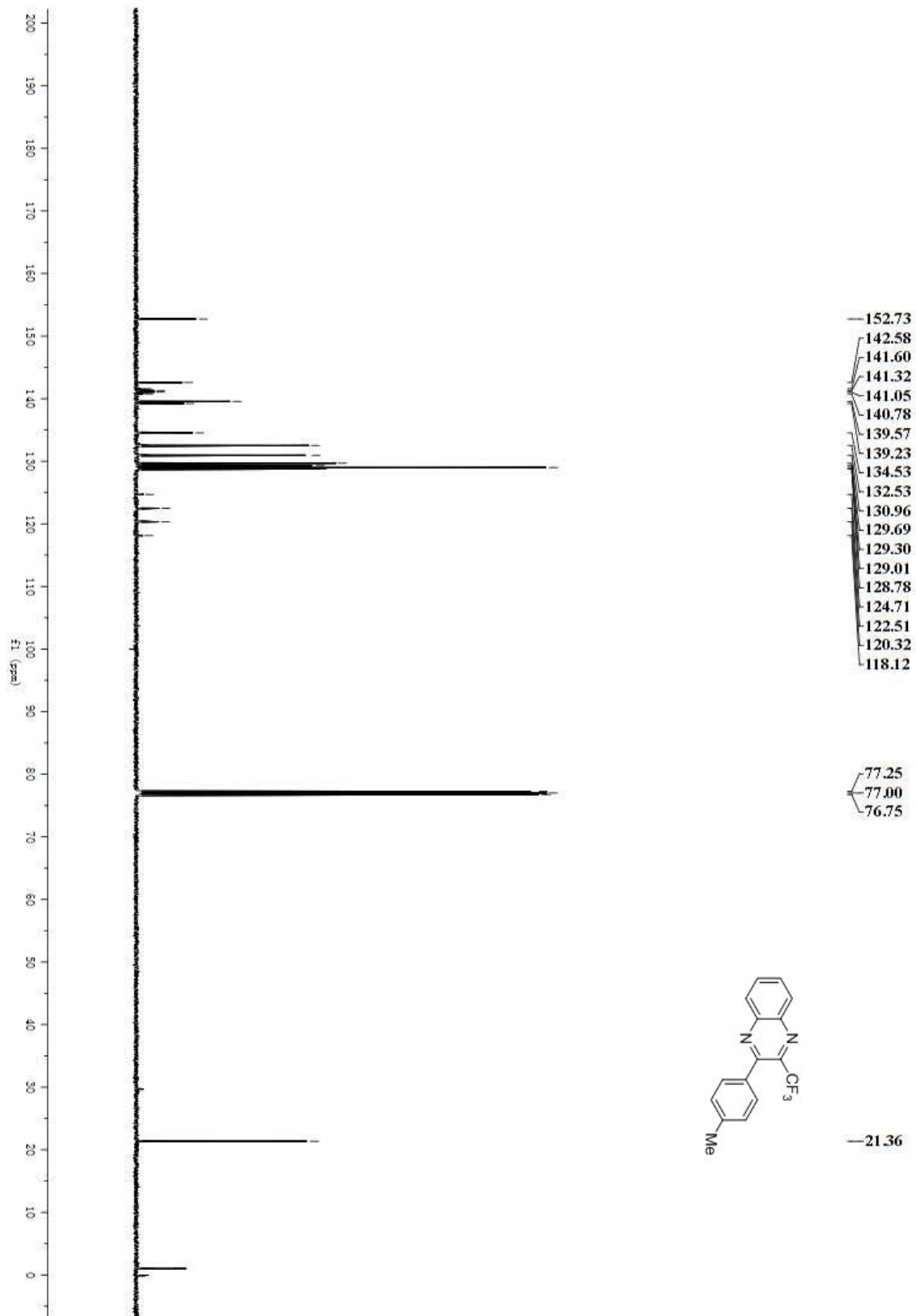




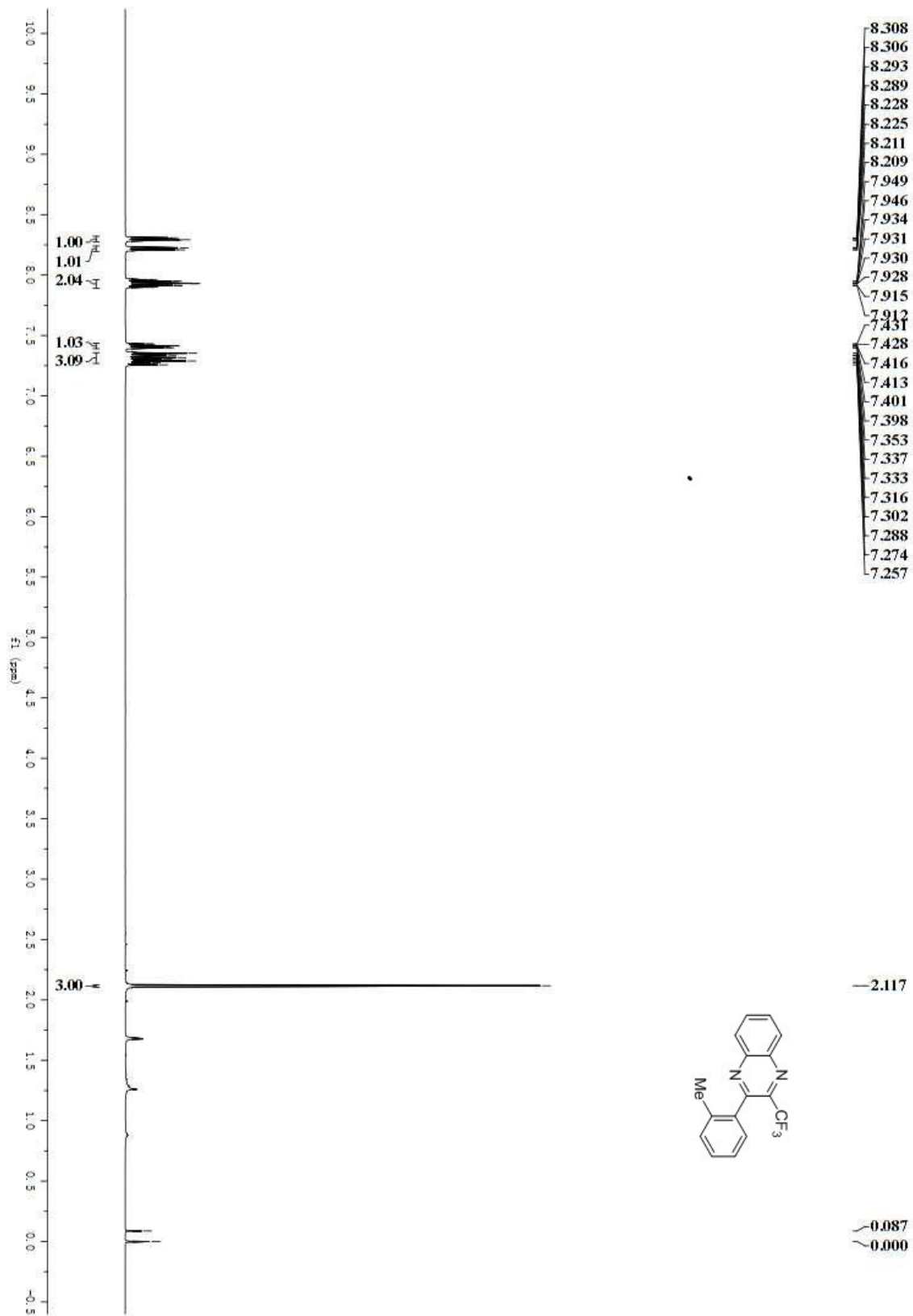
<sup>13</sup>C-NMR: 2-(p-tolyl)-3-(trifluoromethyl)quinoxaline (2m)



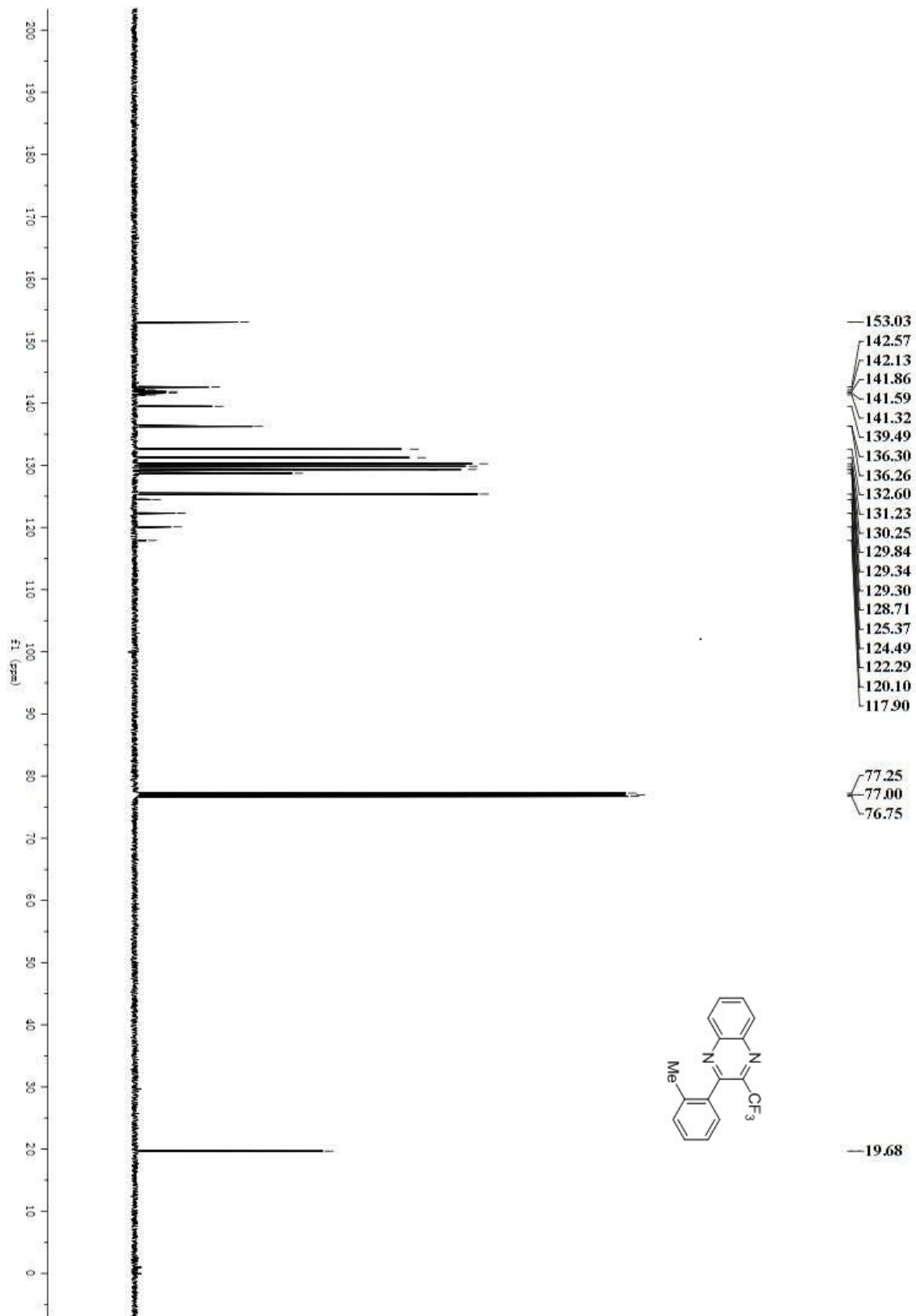
<sup>13</sup>C-NMR: 2-(p-tolyl)-3-(trifluoromethyl)quinoxaline (2m)



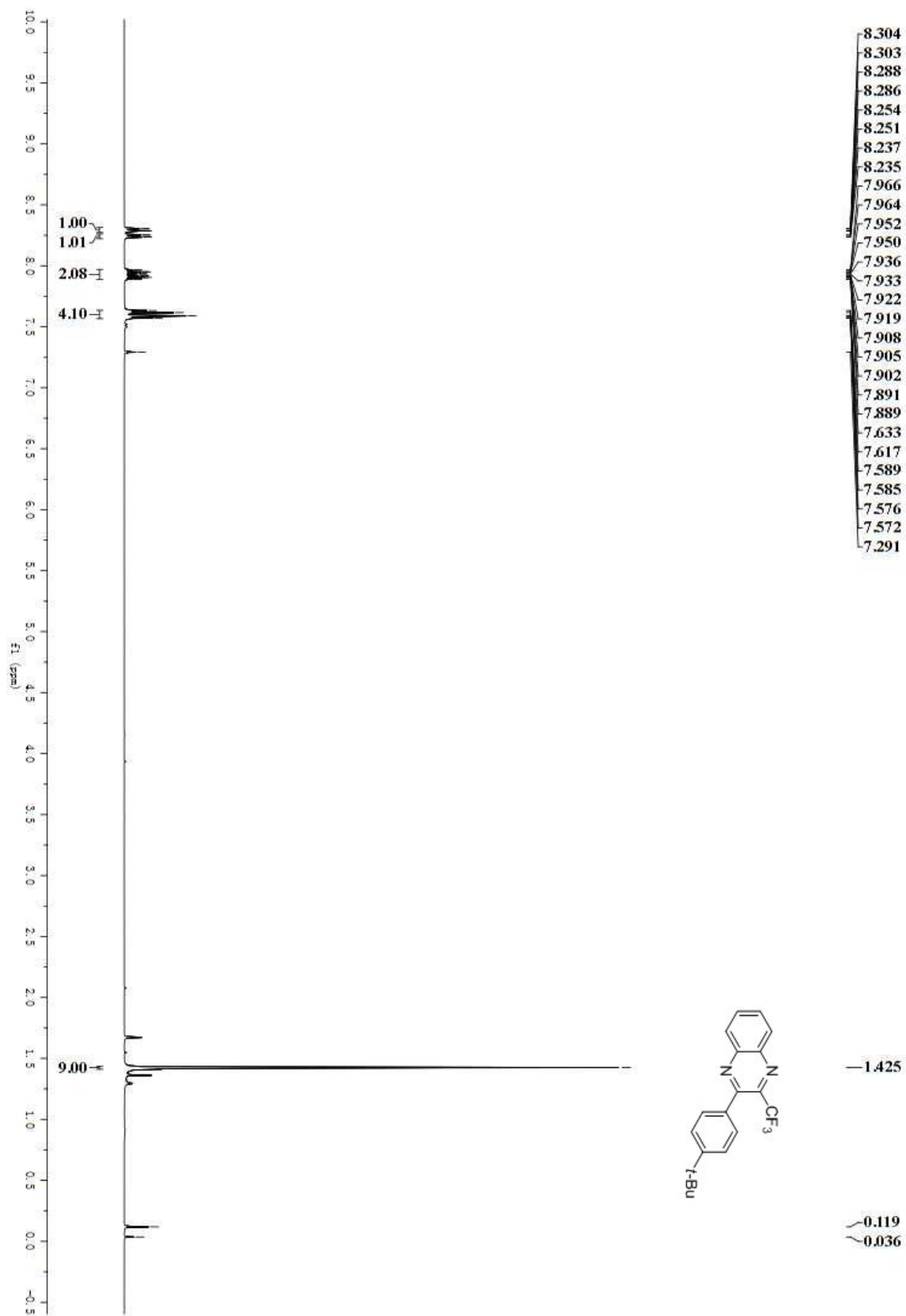
**<sup>1</sup>H-NMR: 2-(O-tolyl)-3-(trifluoromethyl)quinoxaline (2n)**



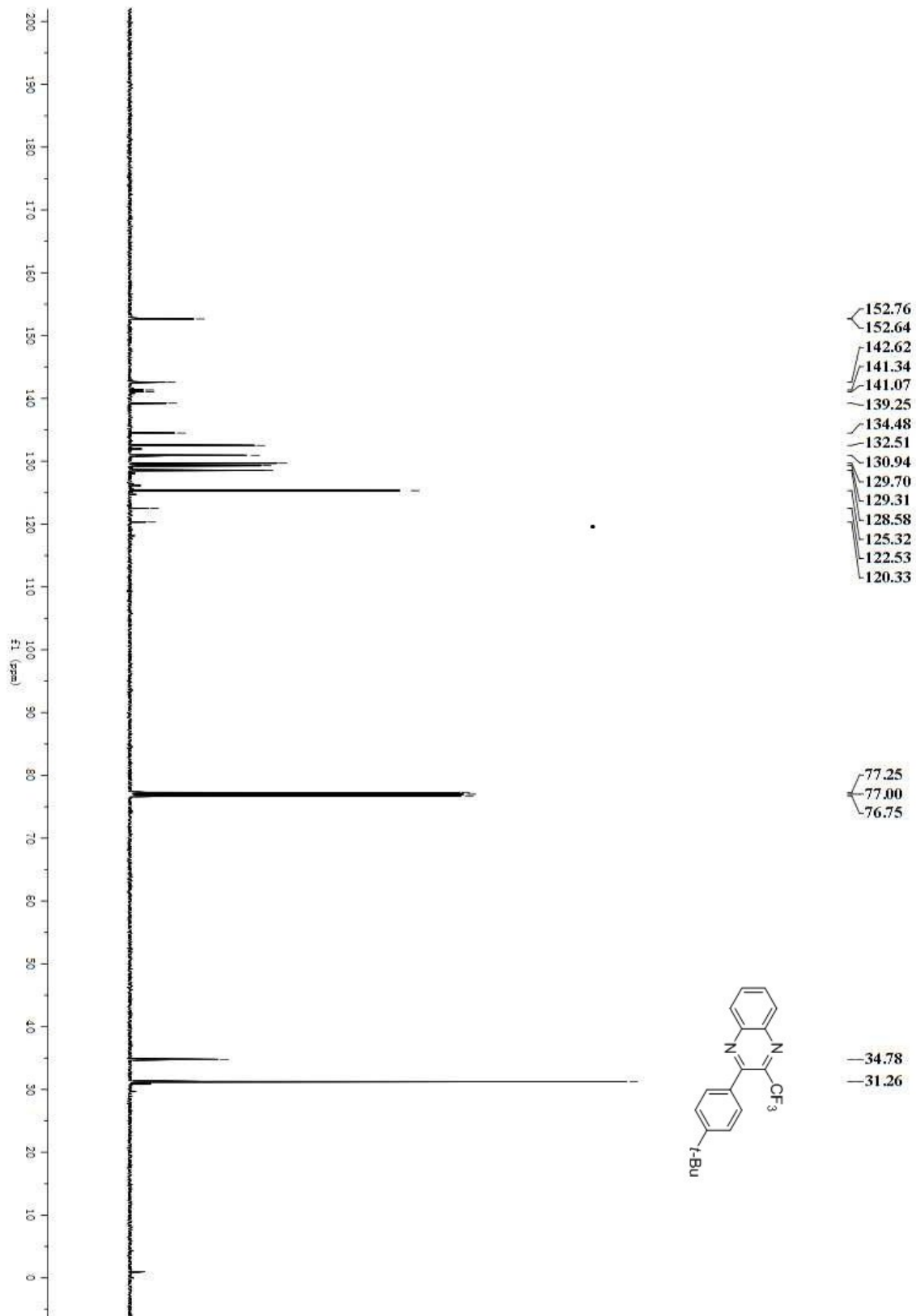
<sup>13</sup>C-NMR: 2-(O-tolyl)-3-(trifluoromethyl)quinoxaline (2n)



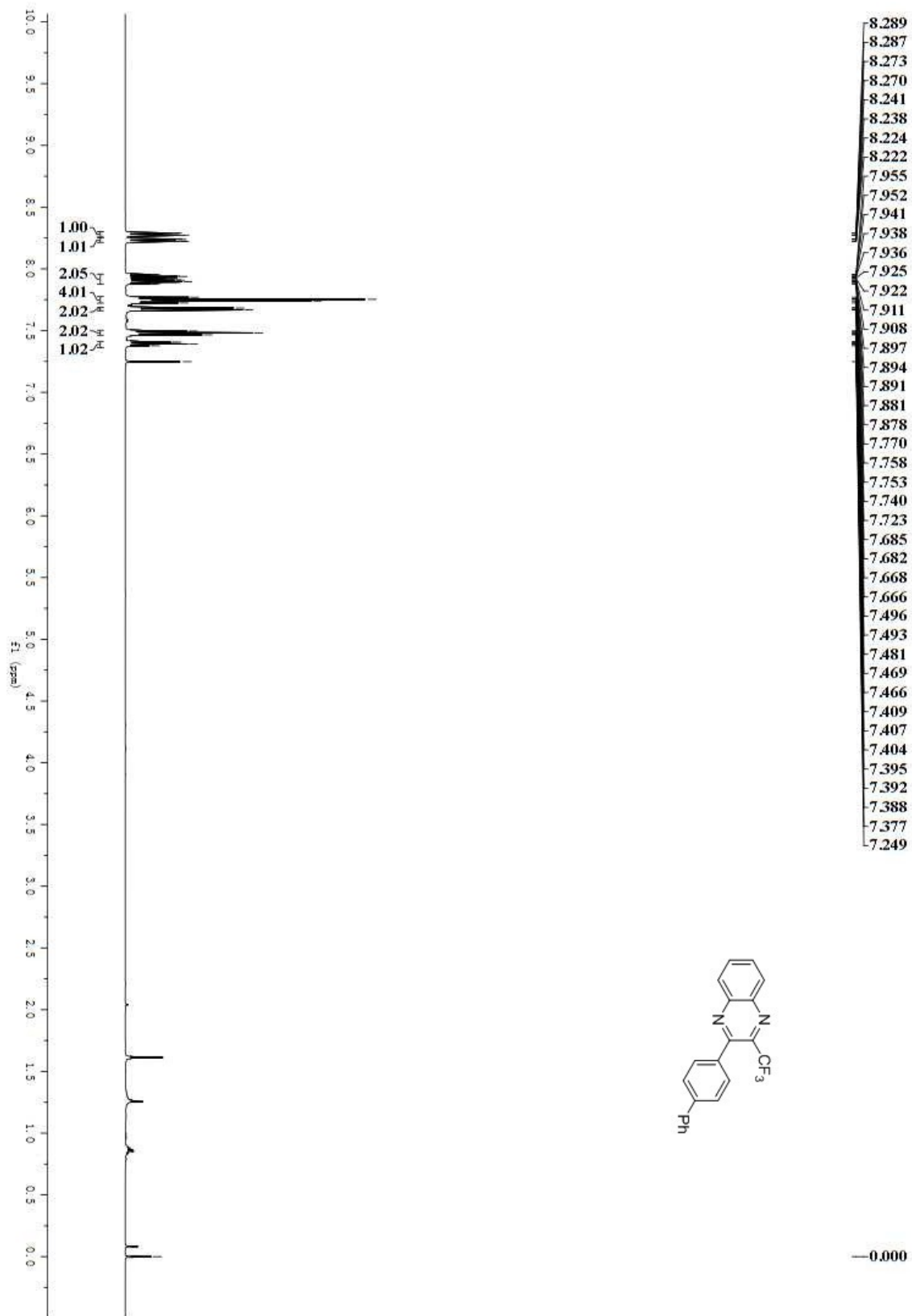
<sup>13</sup>C-NMR: 2-(4-(Tert-butyl)phenyl)-3-(trifluoromethyl)quinoxaline (2o)



<sup>13</sup>C-NMR: 2-(4-(Tert-butyl)phenyl)-3-(trifluoromethyl)quinoxaline (2o)

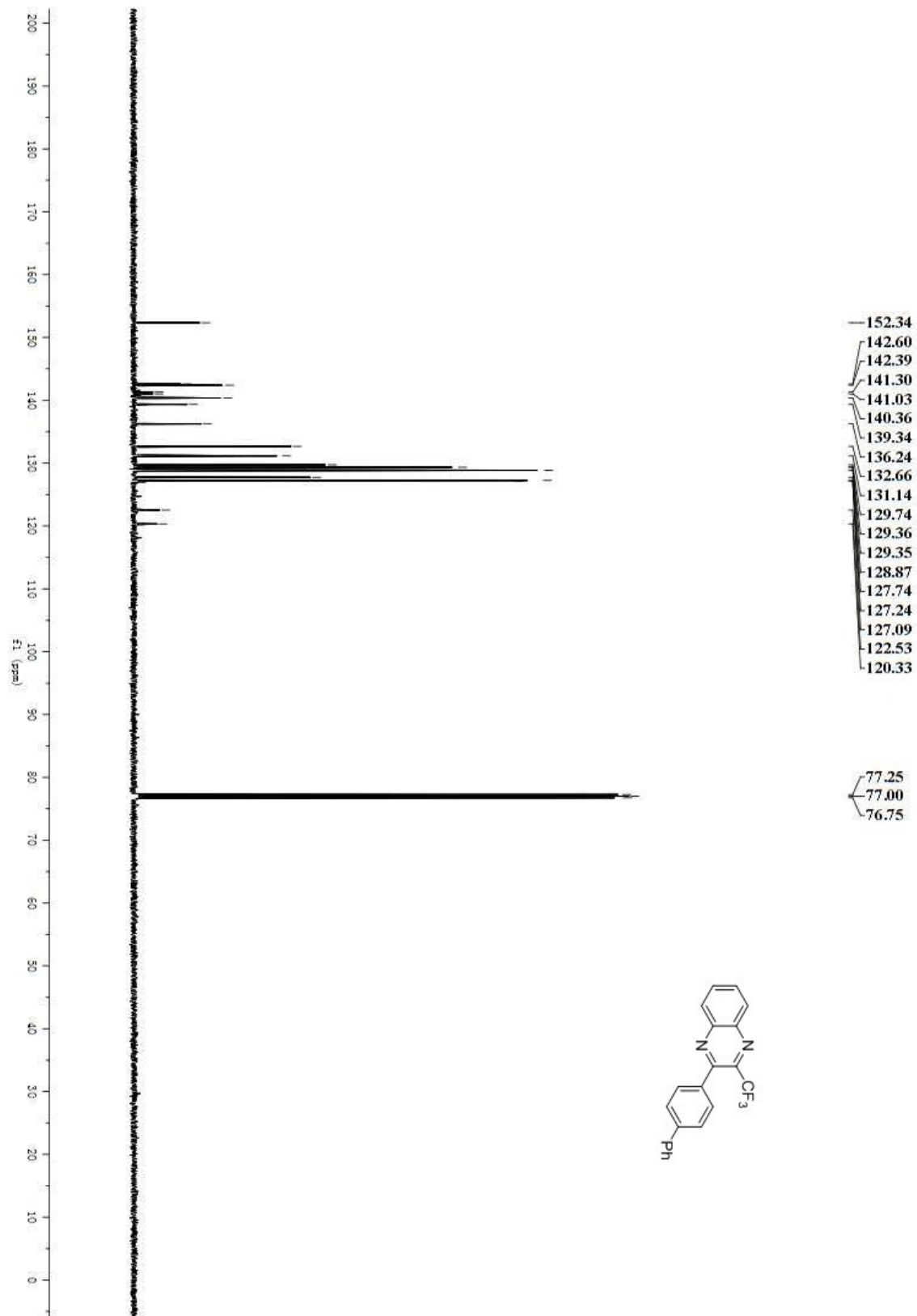


<sup>1</sup>H-NMR: 2-([1,1'-Biphenyl]-4-yl)-3-(trifluoromethyl)quinoxaline (2p)

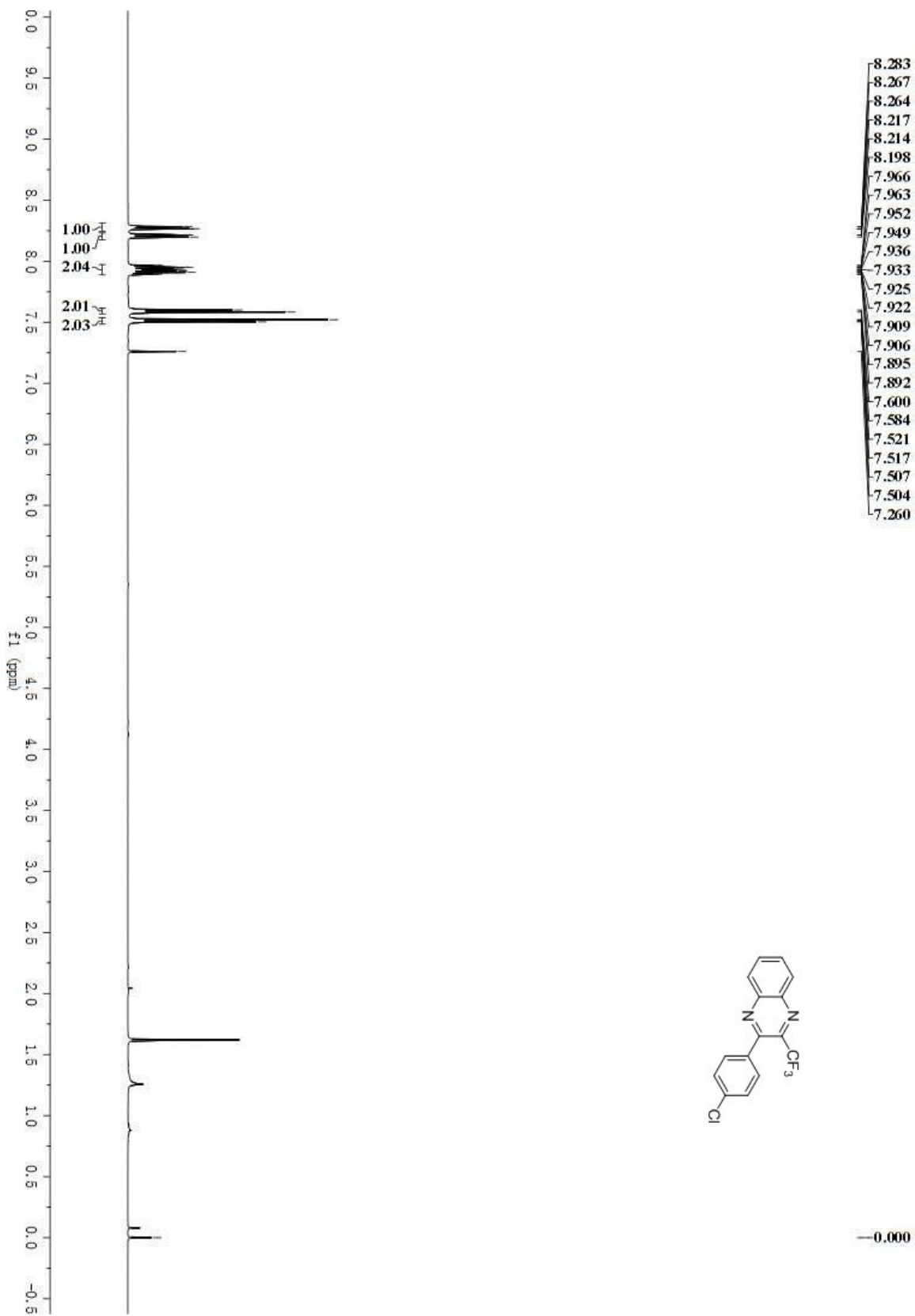


**<sup>13</sup>C-NMR: 2-([1,1'-Biphenyl]-4-yl)-3-(trifluoromethyl)quinoxaline (2p)**

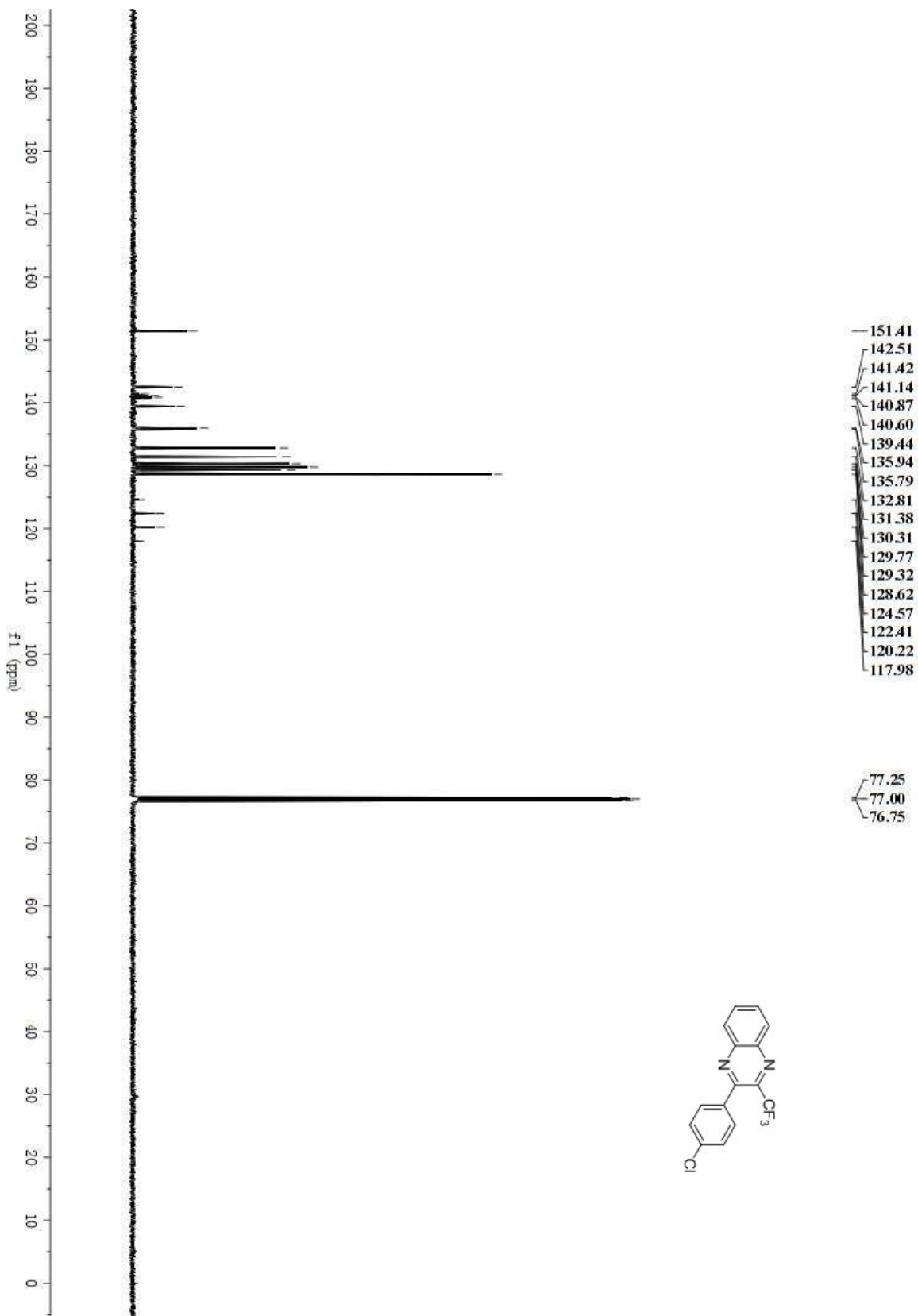




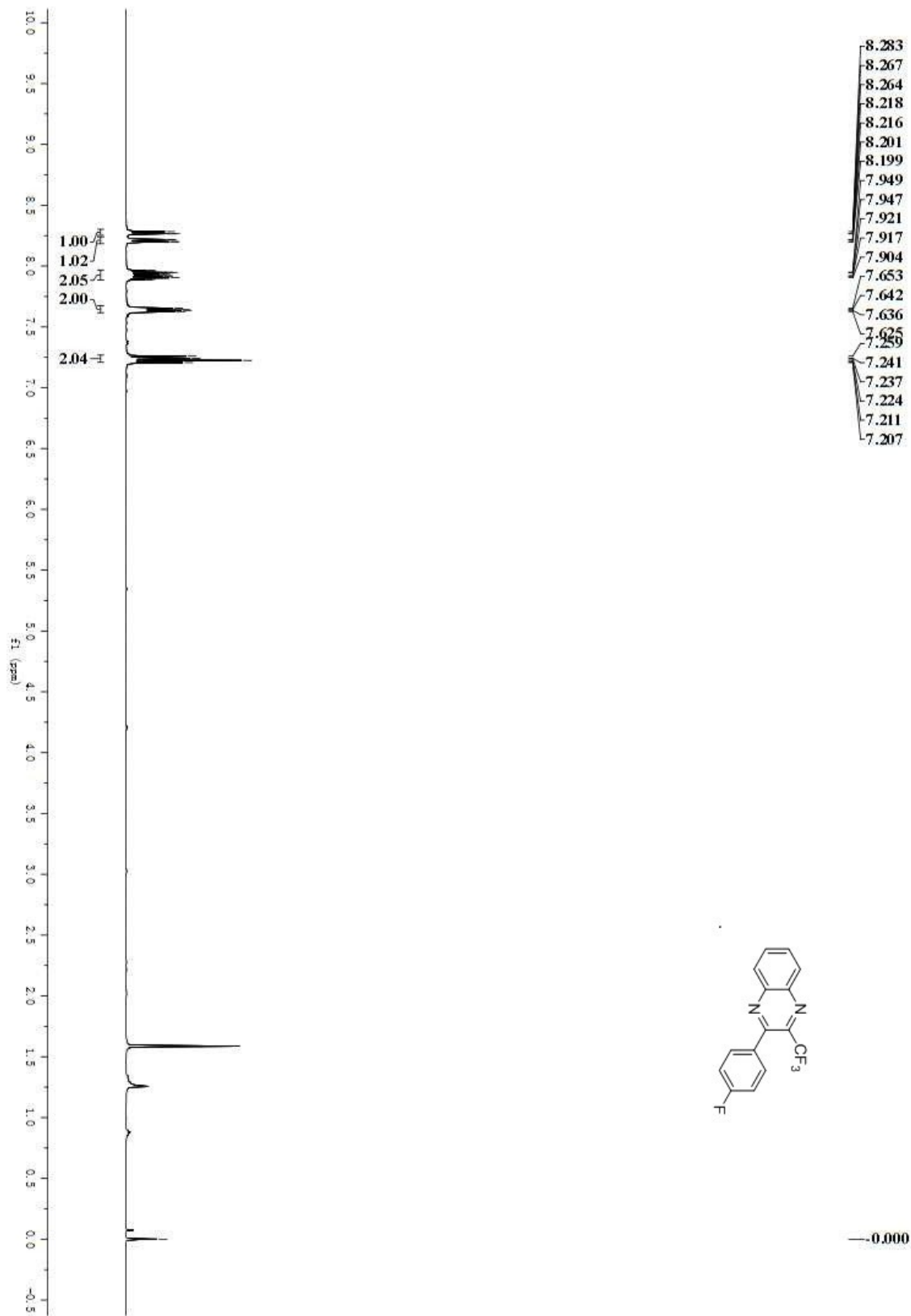
<sup>13</sup>C-NMR: 2-(4-Chlorophenyl)-3-(trifluoromethyl)quinoxaline (2q)



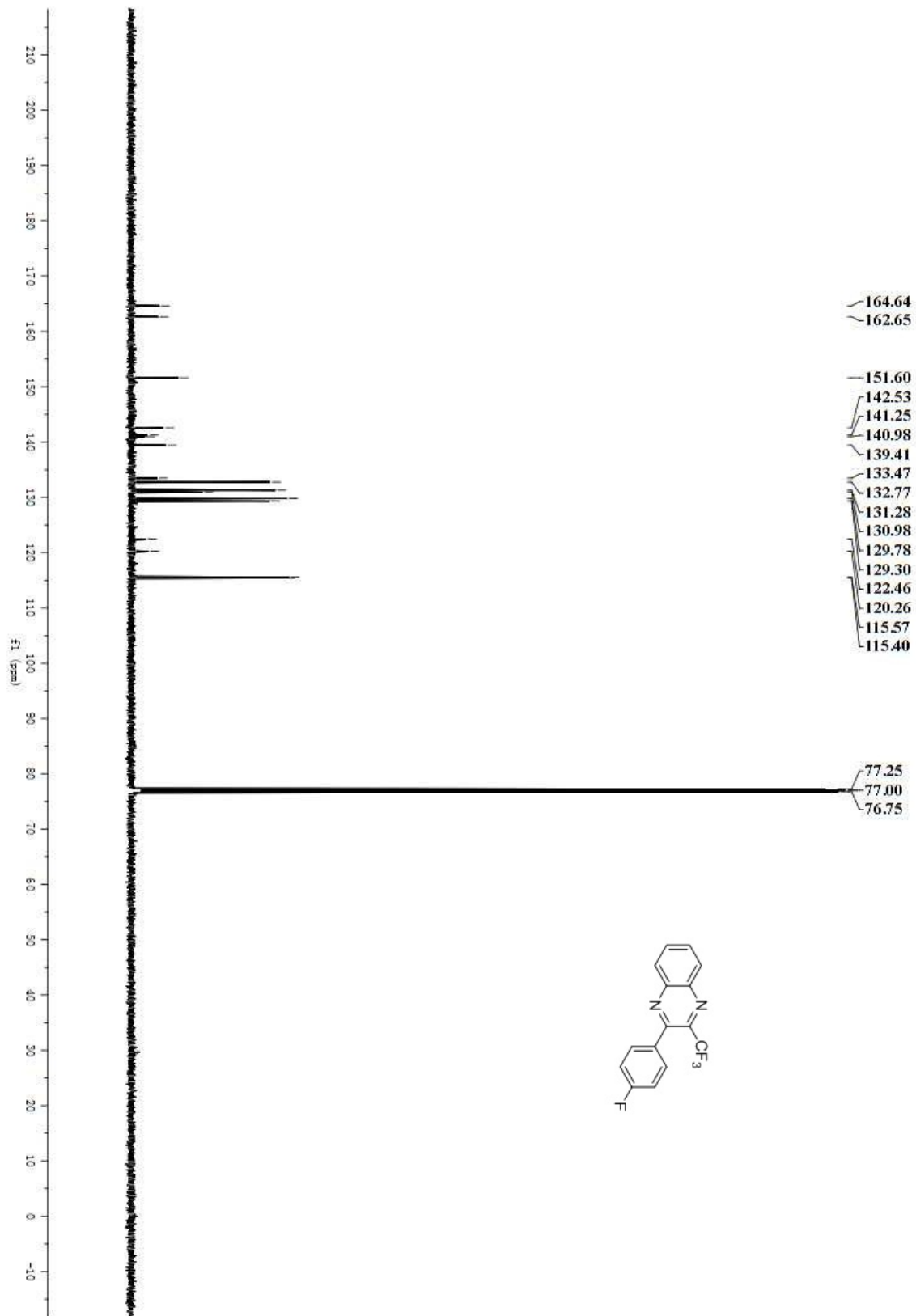
<sup>13</sup>C-NMR: 2-(4-Chlorophenyl)-3-(trifluoromethyl)quinoxaline (2q)



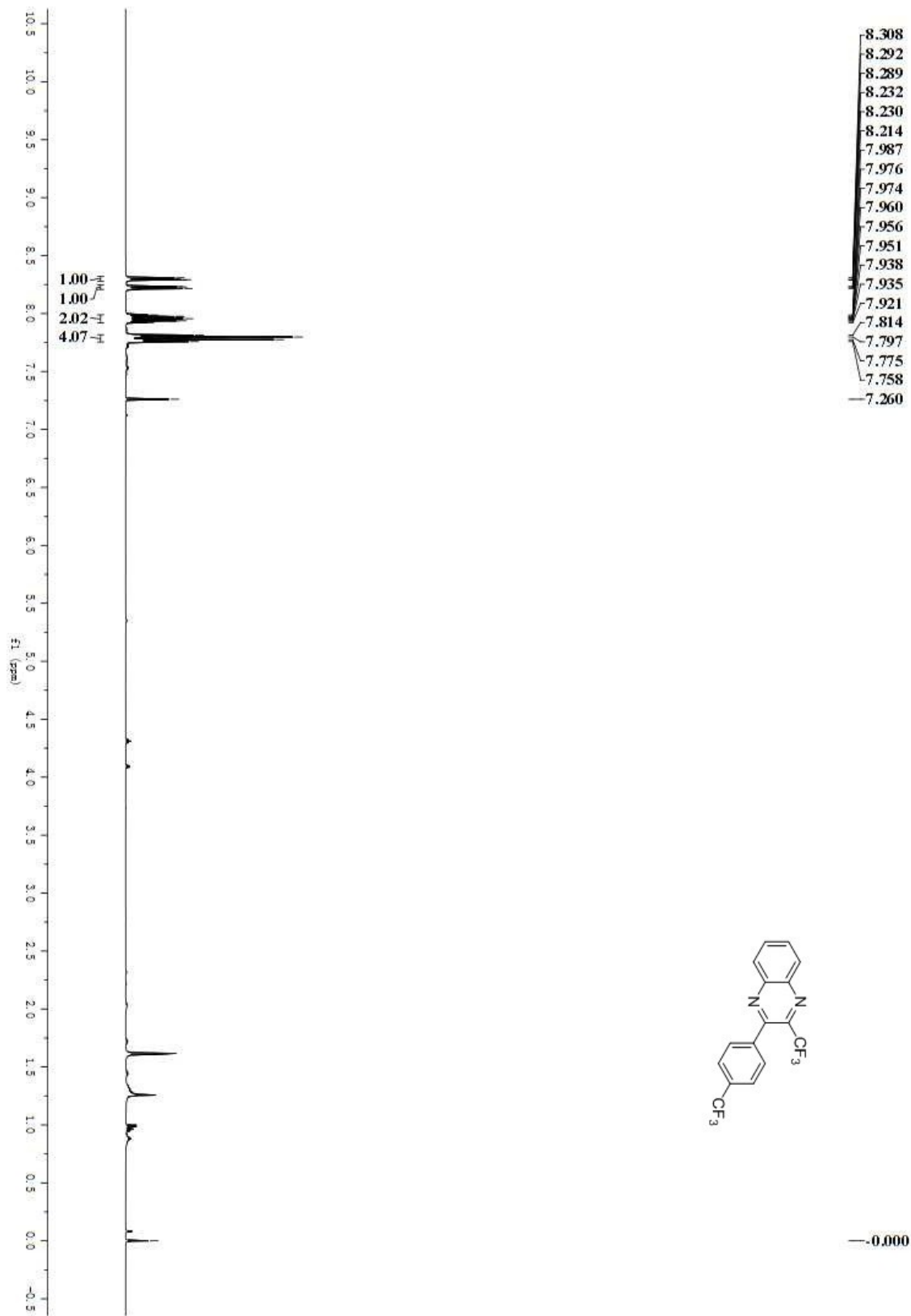
<sup>1</sup>H-NMR: 2-(4-Fluorophenyl)-3-(trifluoromethyl)quinoxaline (2r)



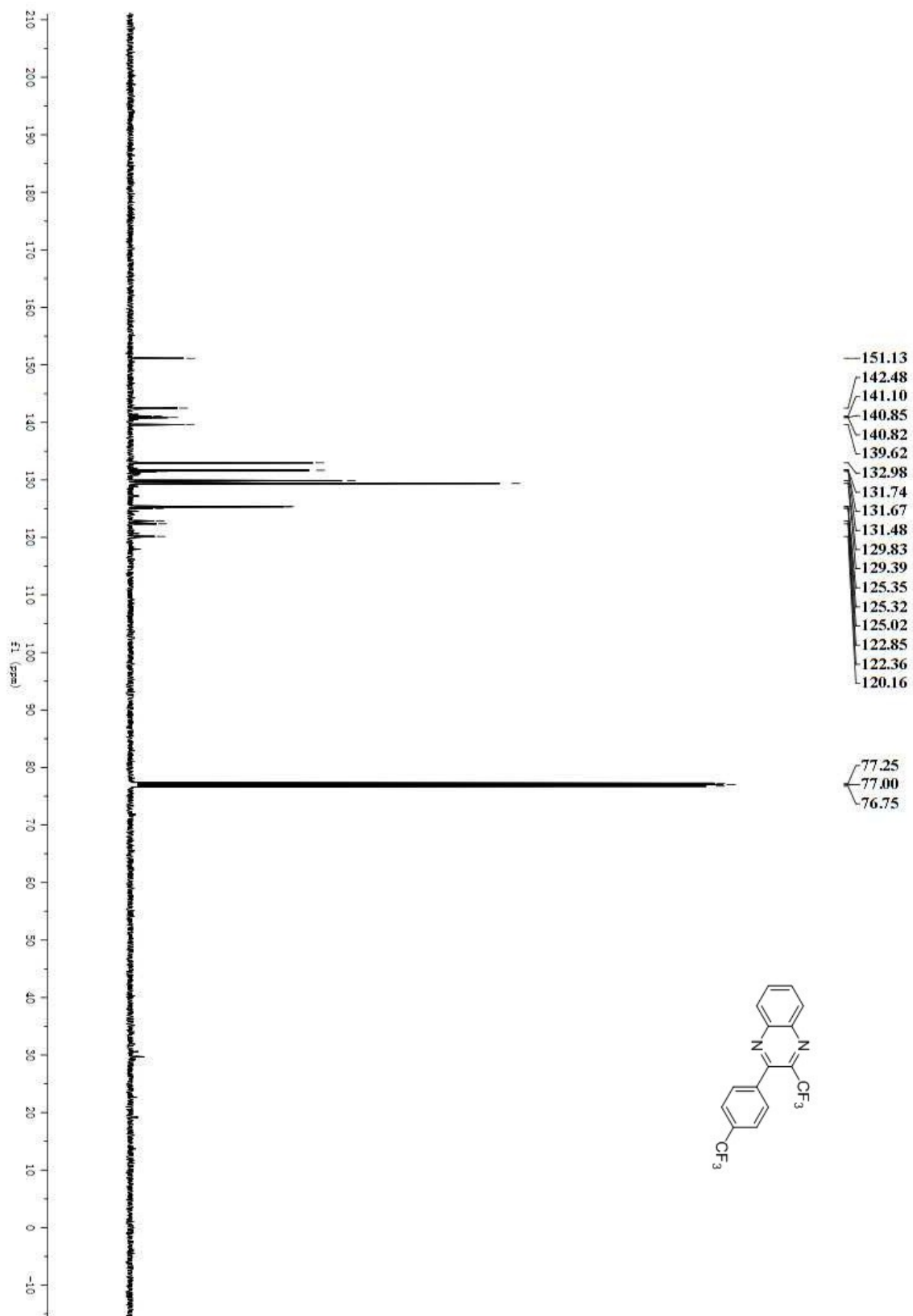
<sup>13</sup>C-NMR: 2-(4-Fluorophenyl)-3-(trifluoromethyl)quinoxaline (2r)



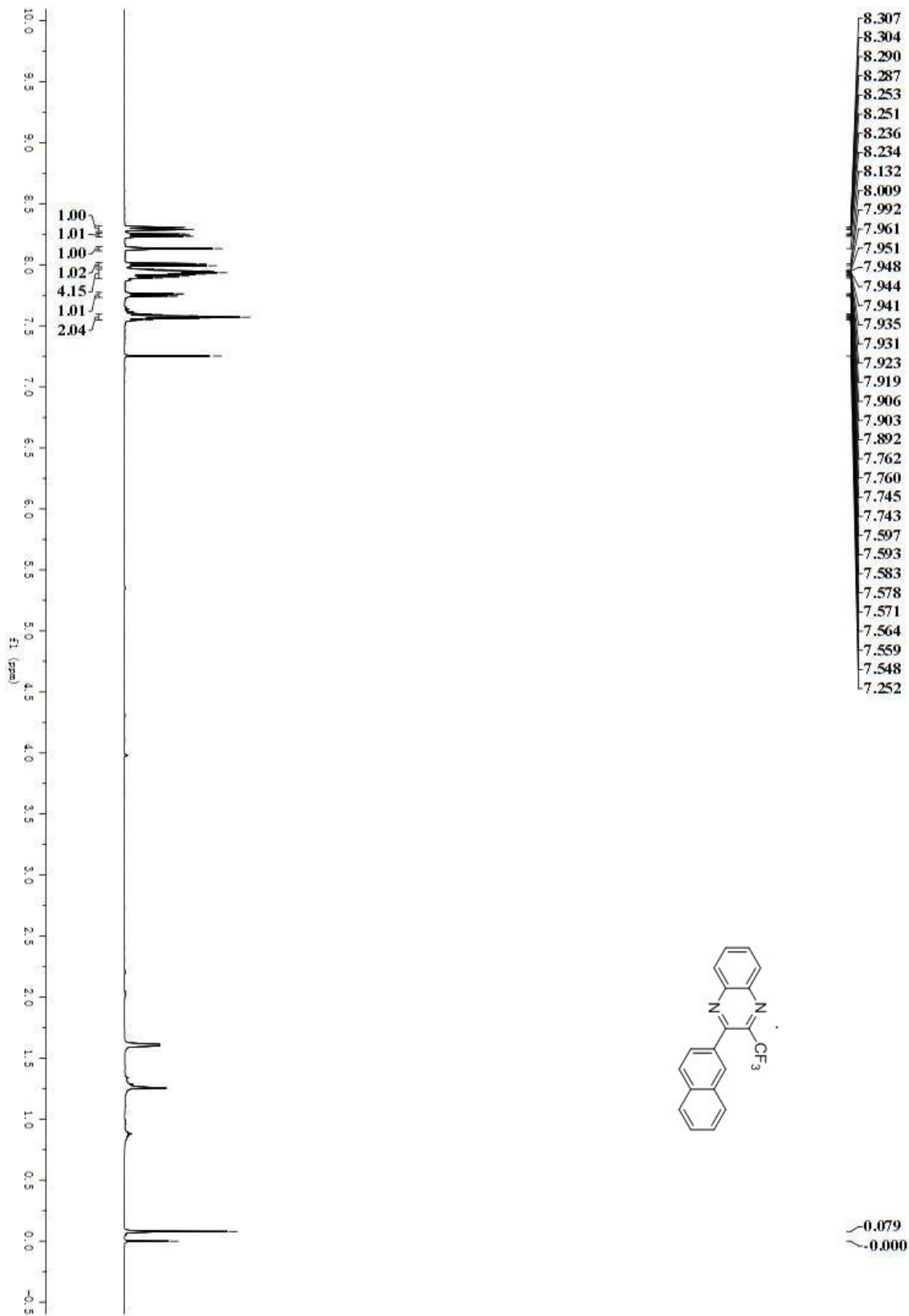
<sup>13</sup>C-NMR: 2-(Trifluoromethyl)-3-(4-(trifluoromethyl)phenyl)quinoxaline (2s)



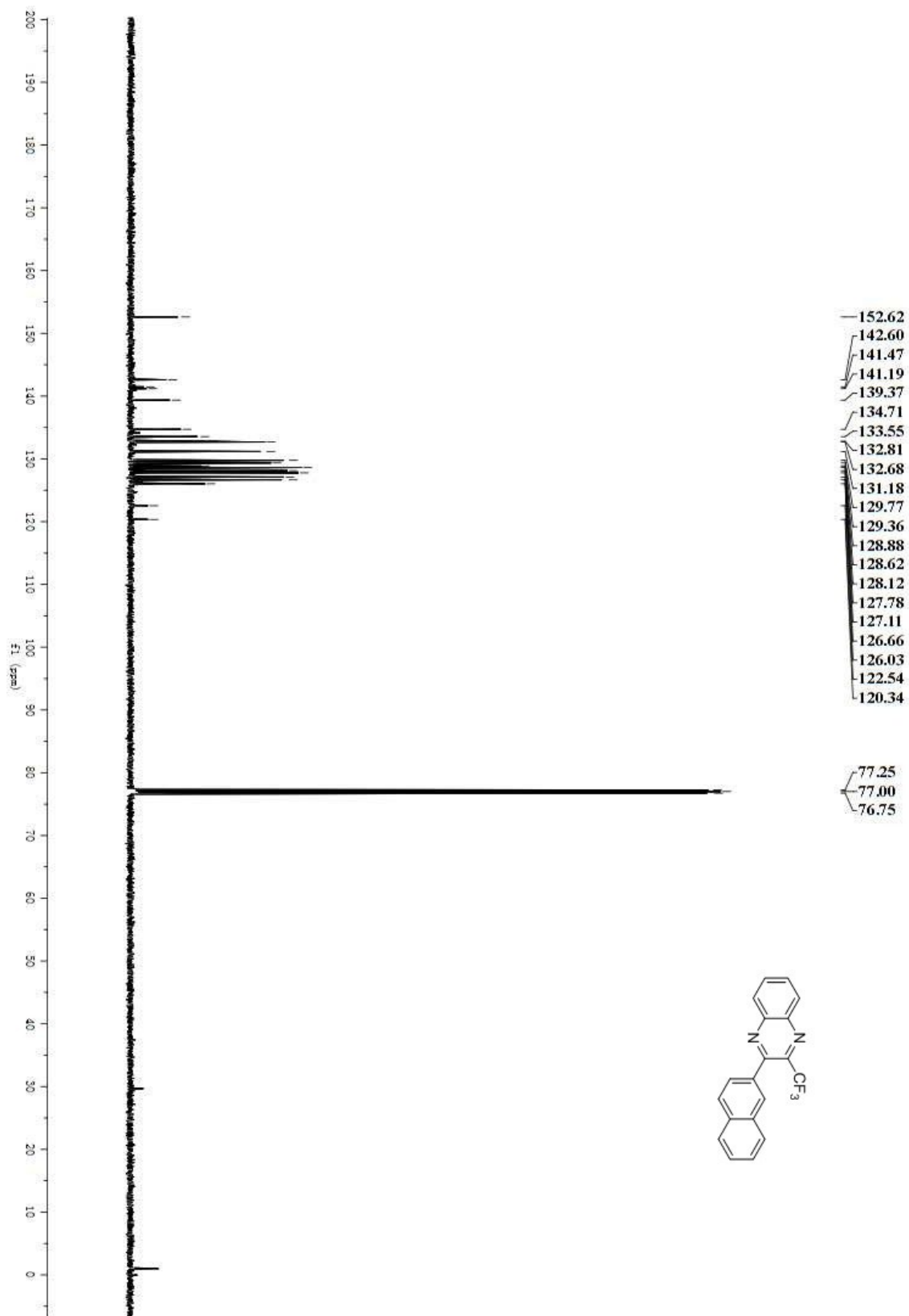
$^{13}\text{C-NMR}$ : 2-(Trifluoromethyl)-3-(4-(trifluoromethyl)phenyl)quinoxaline (2s)



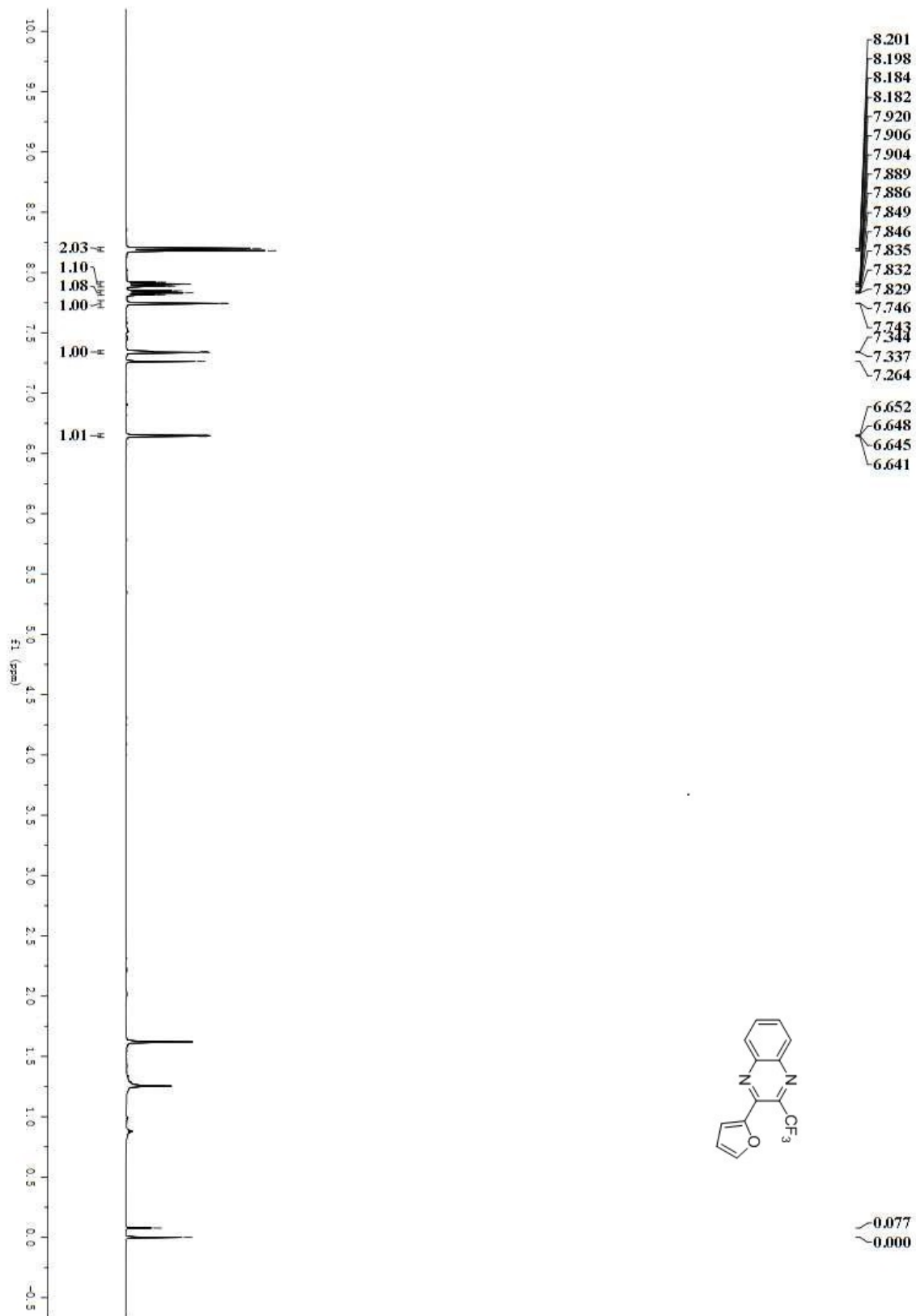
<sup>1</sup>H-NMR: 2-(Naphthalen-2-yl)-3-(trifluoromethyl)quinoxaline (2t)



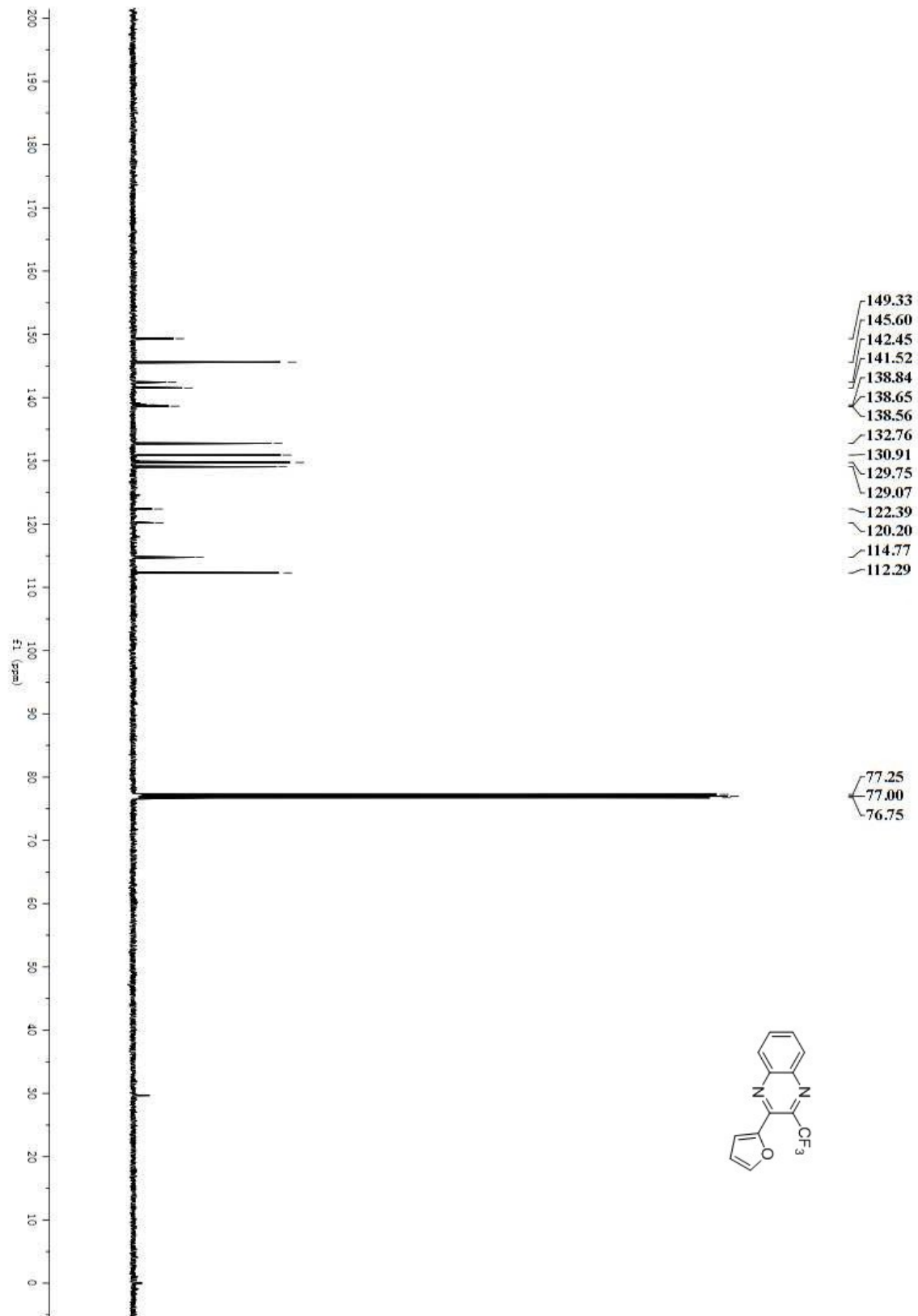




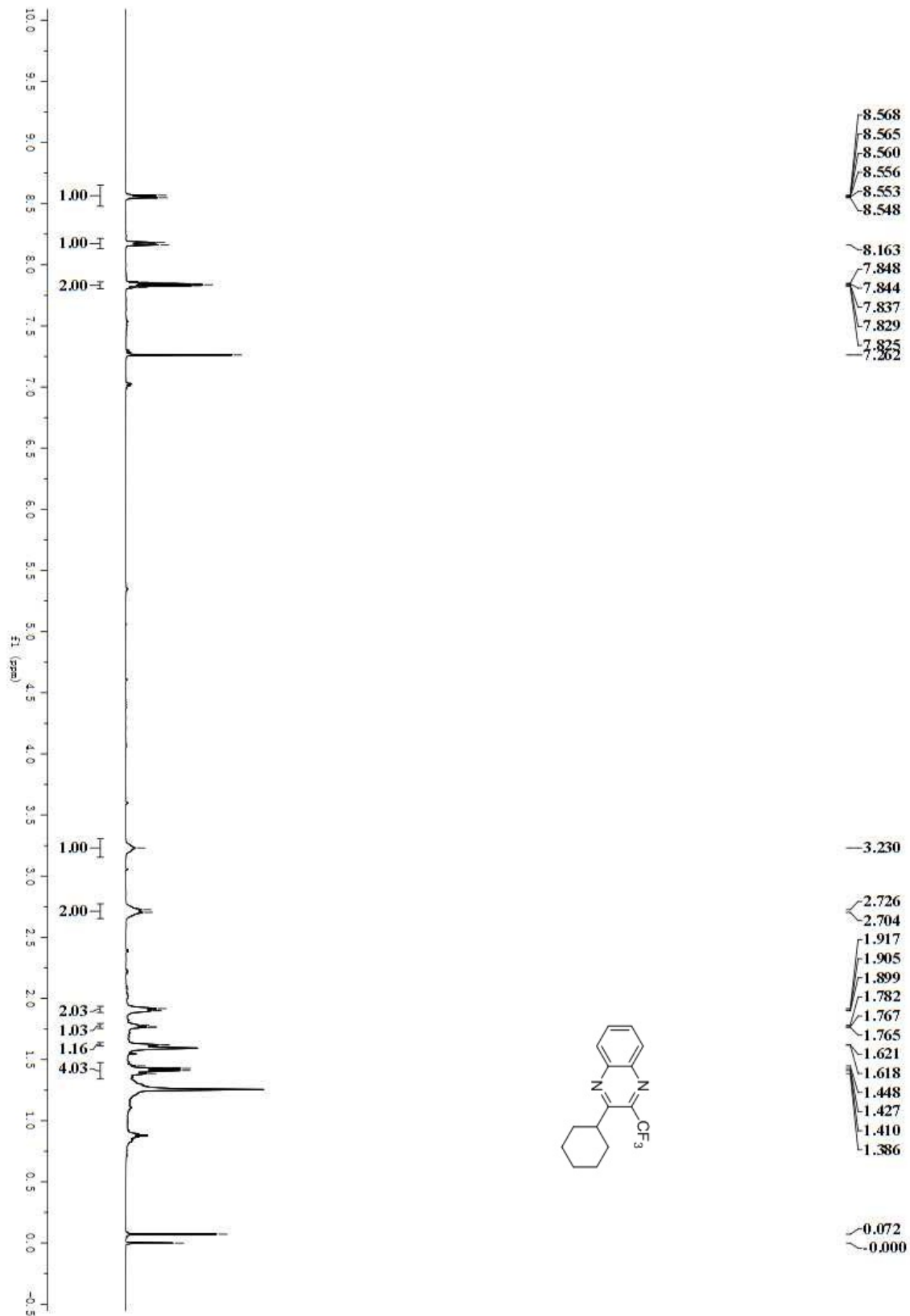
**<sup>13</sup>C-NMR: 2-(Furan-2-yl)-3-(trifluoromethyl)quinoxaline (2u)**



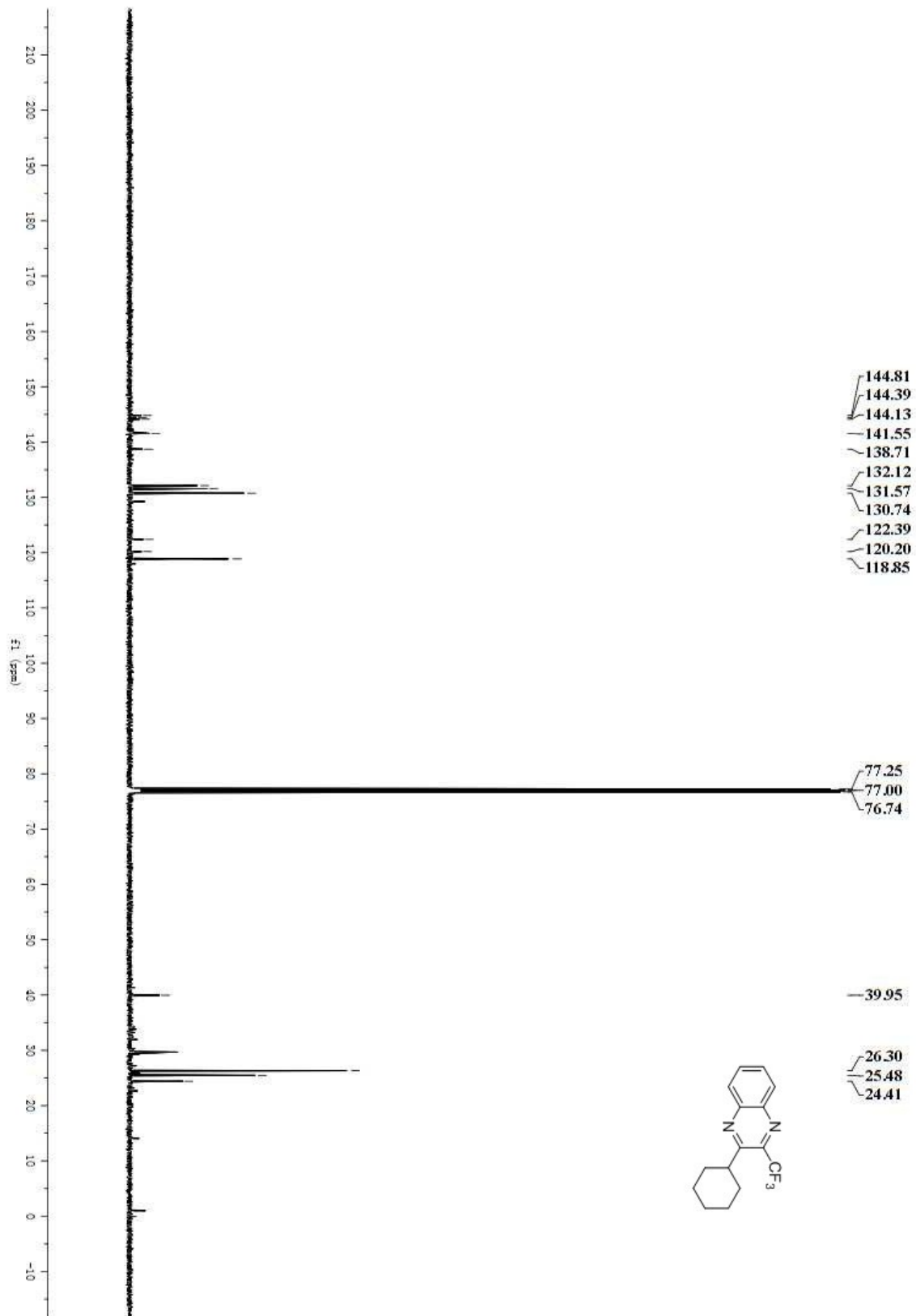
<sup>13</sup>C-NMR: 2-(Furan-2-yl)-3-(trifluoromethyl)quinoxaline (2u)



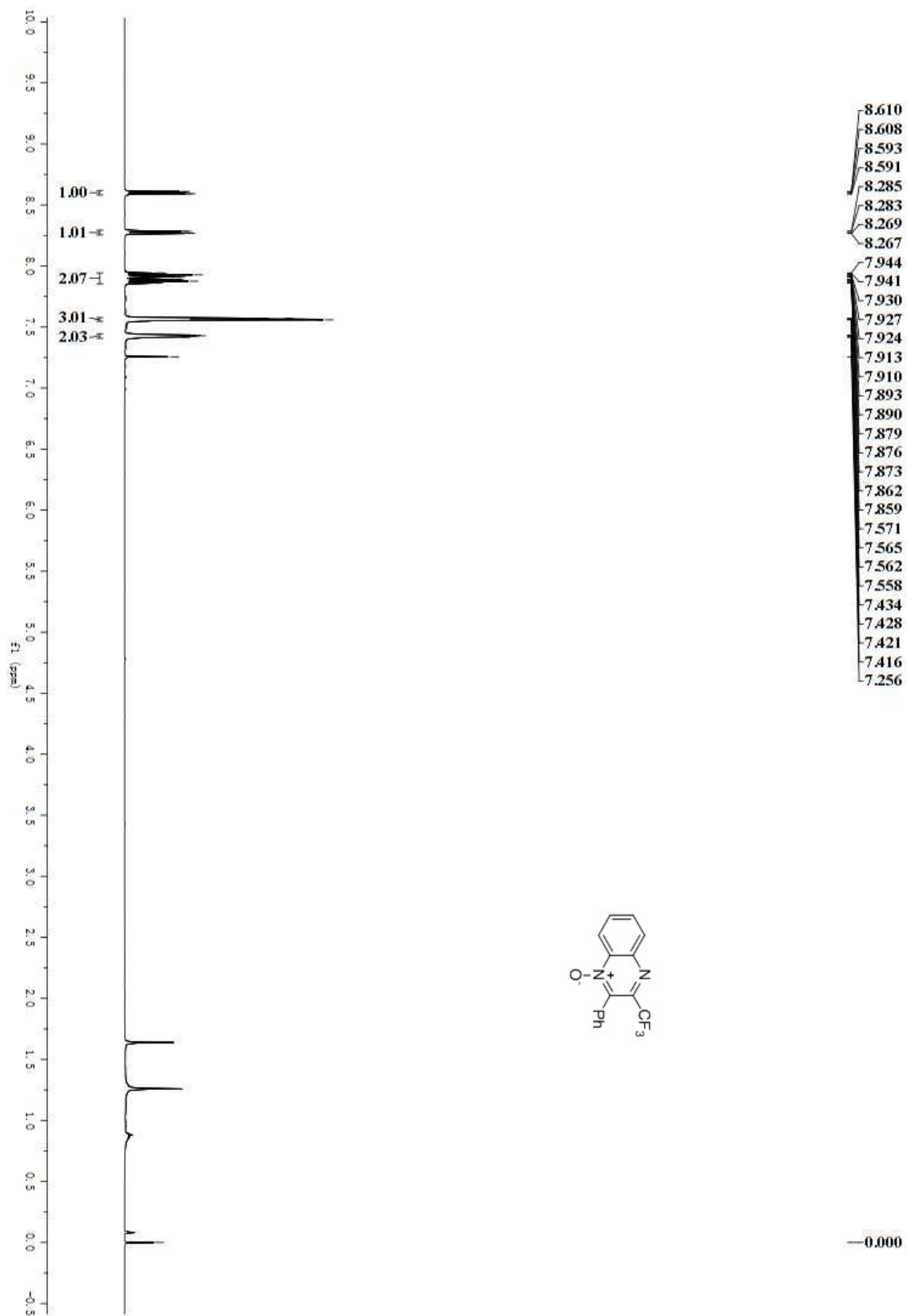
<sup>1</sup>H-NMR: 2-Cyclohexyl-3-(trifluoromethyl)quinoxaline (2v)



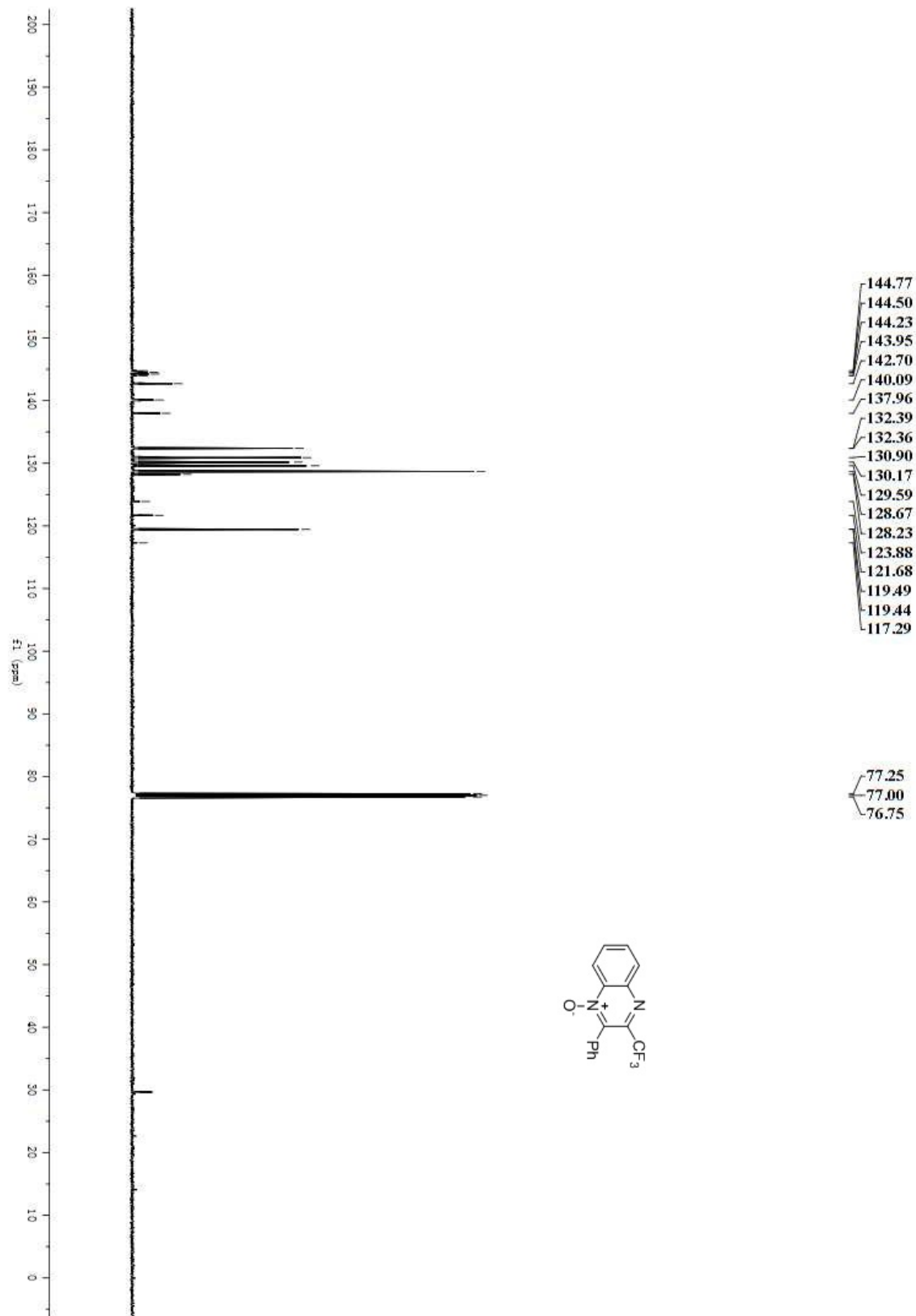
**<sup>13</sup>C-NMR: 2-Cyclohexyl-3-(trifluoromethyl)quinoxaline (2v)**



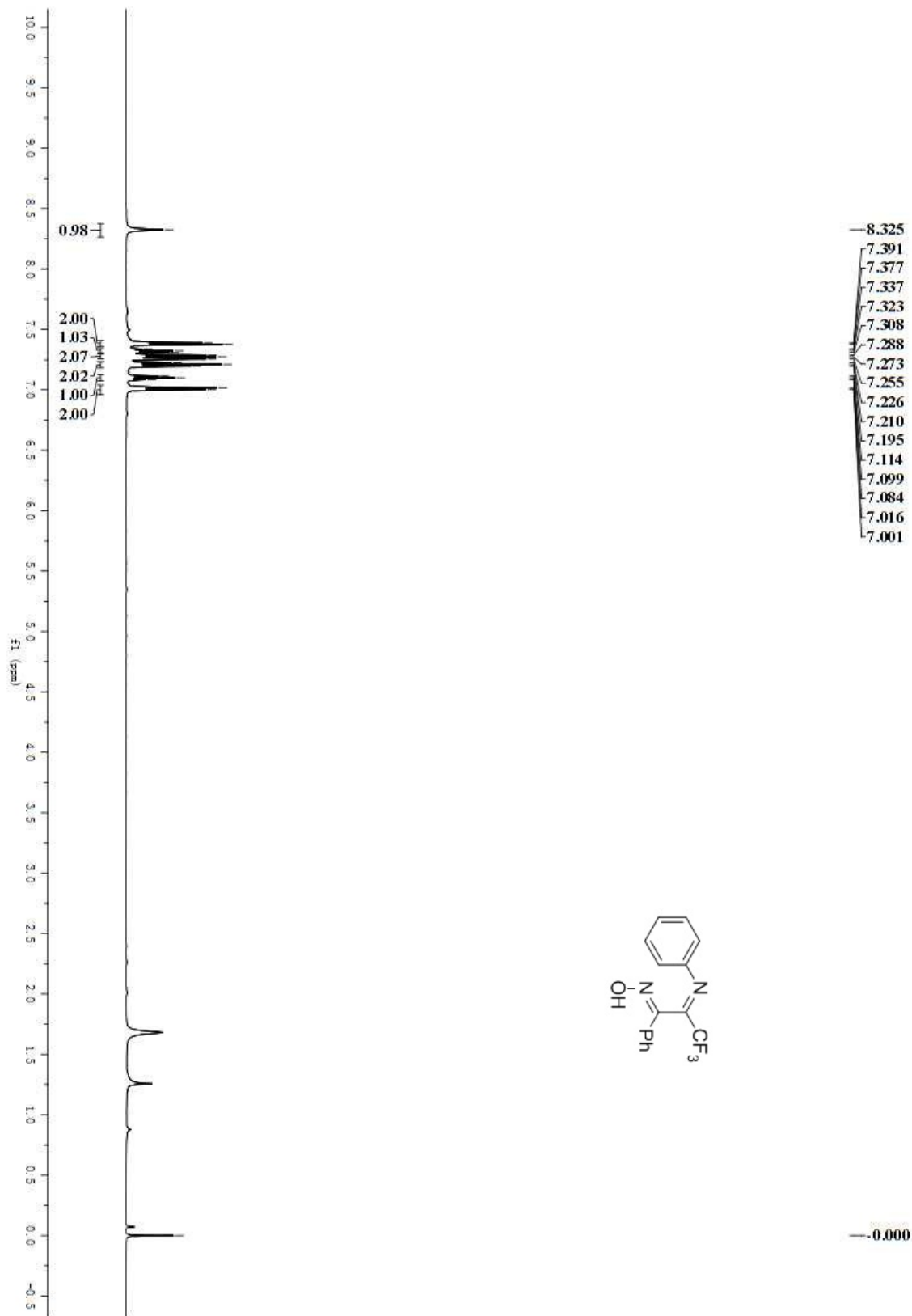
<sup>1</sup>H-NMR: 2-Phenyl-3-(trifluoromethyl)quinoxaline 1-oxide (3a)



<sup>13</sup>C-NMR: 2-Phenyl-3-(trifluoromethyl)quinoxaline 1-oxide (3a)

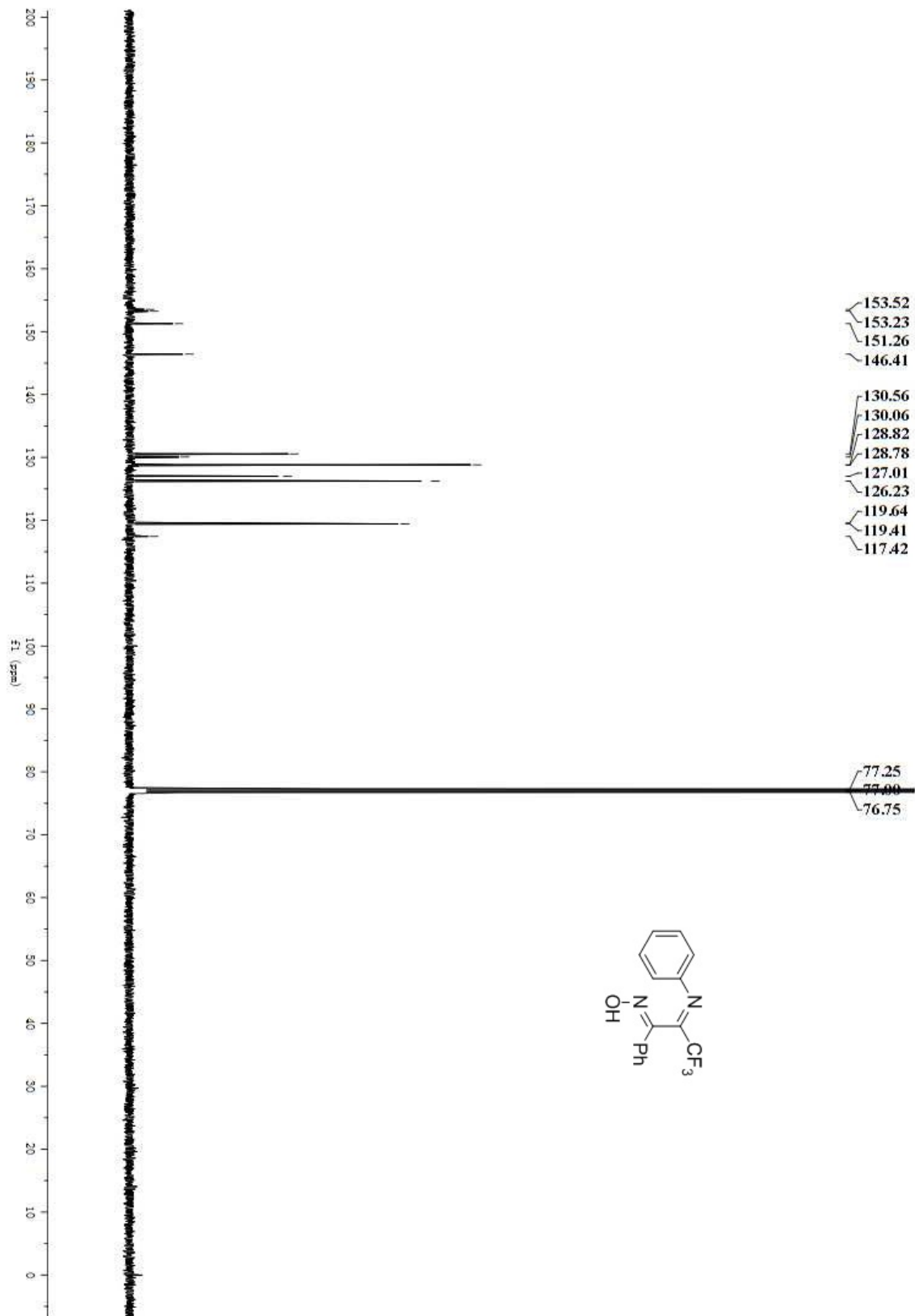


**<sup>1</sup>H-NMR: (1E, 2E)-3,3,3-trifluoro-1-phenyl-2-(phenylimino)propan-1-one oxime (4a)**

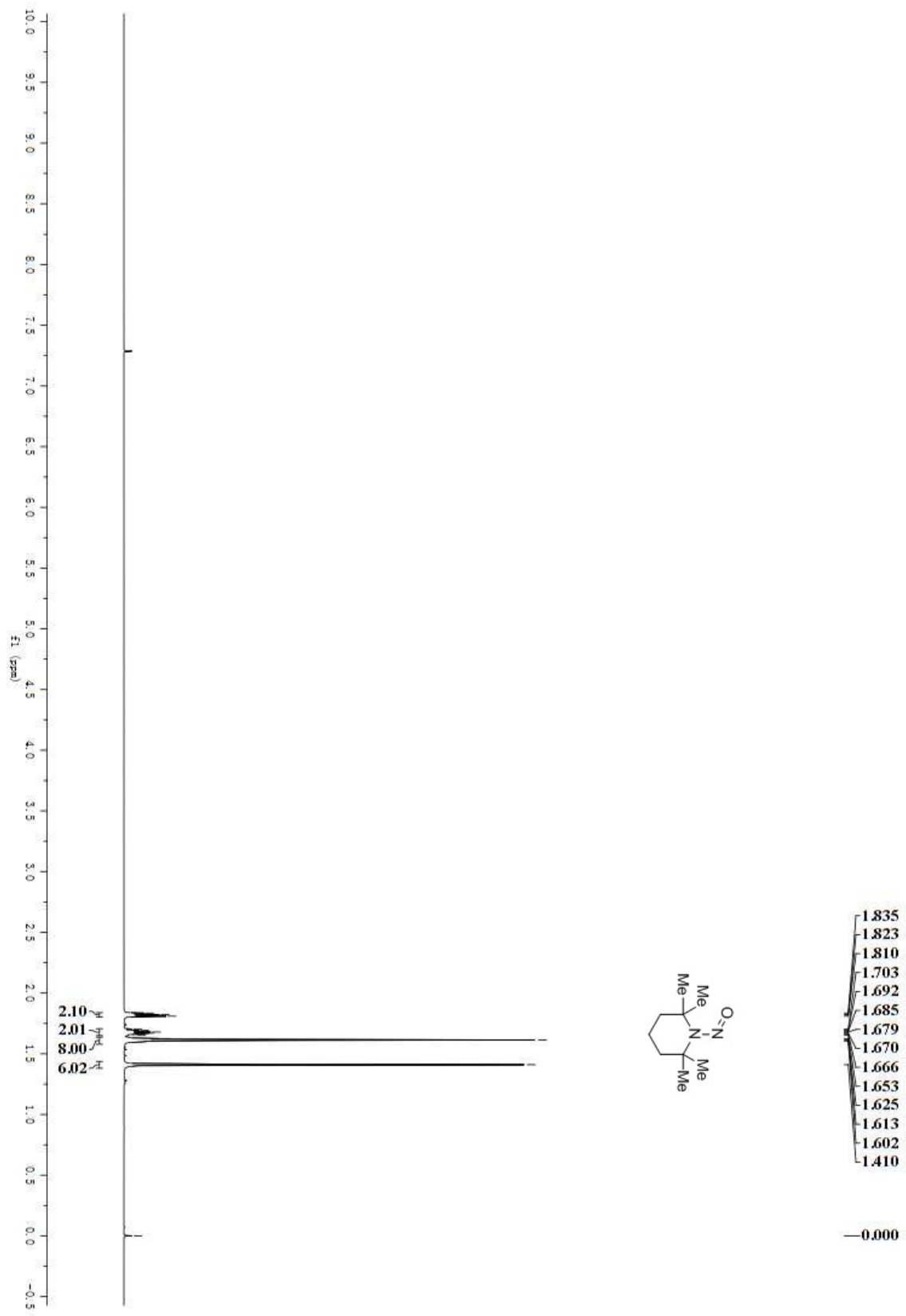


<sup>13</sup>C-NMR: (1E, 2E)-3,3,3-trifluoro-1-phenyl-2-(phenylimino)propan-1-one oxime(4a)

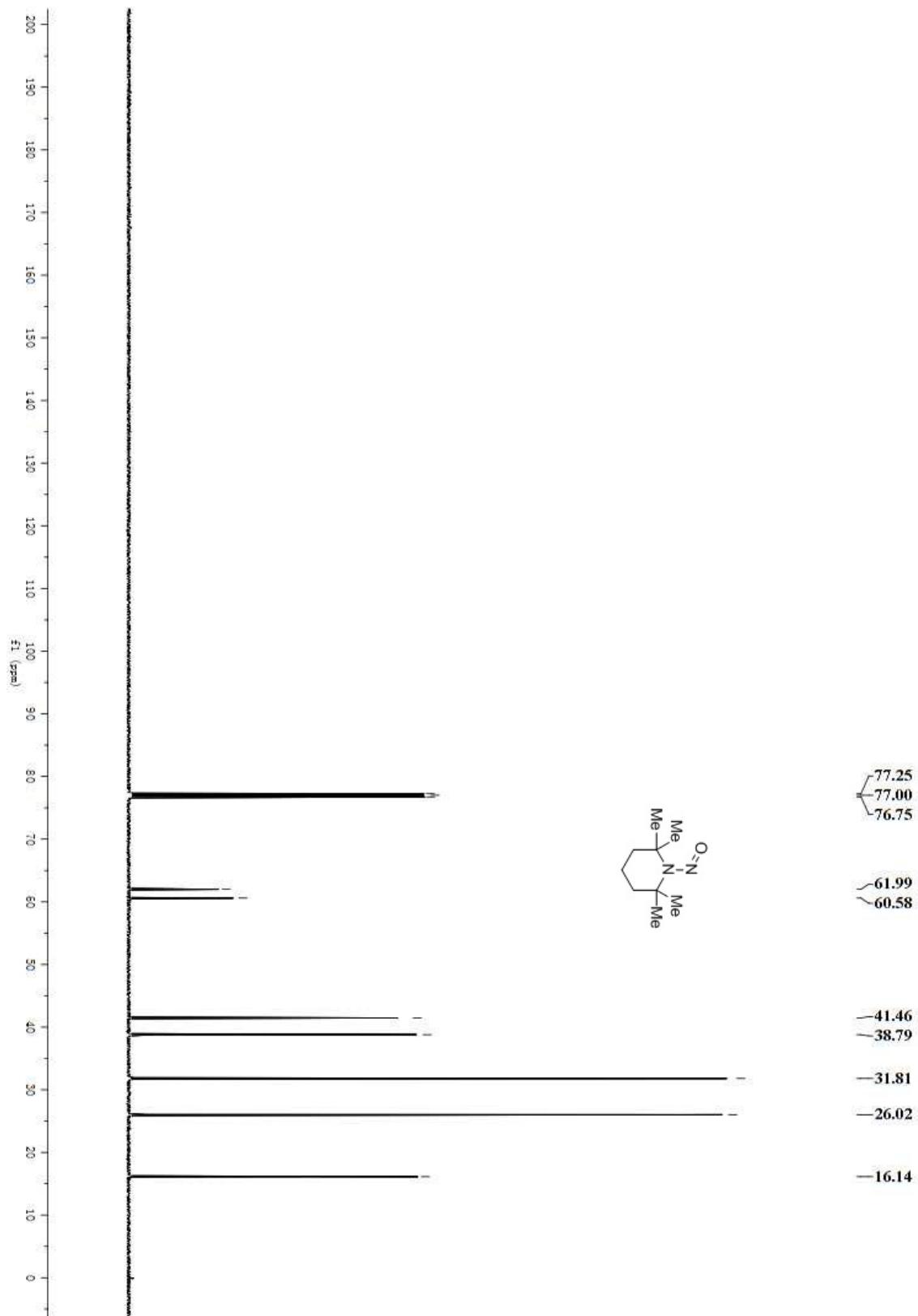




<sup>1</sup>H-NMR: 2,2,6,6-Tetramethyl-1-nitrosopiperidine (5)

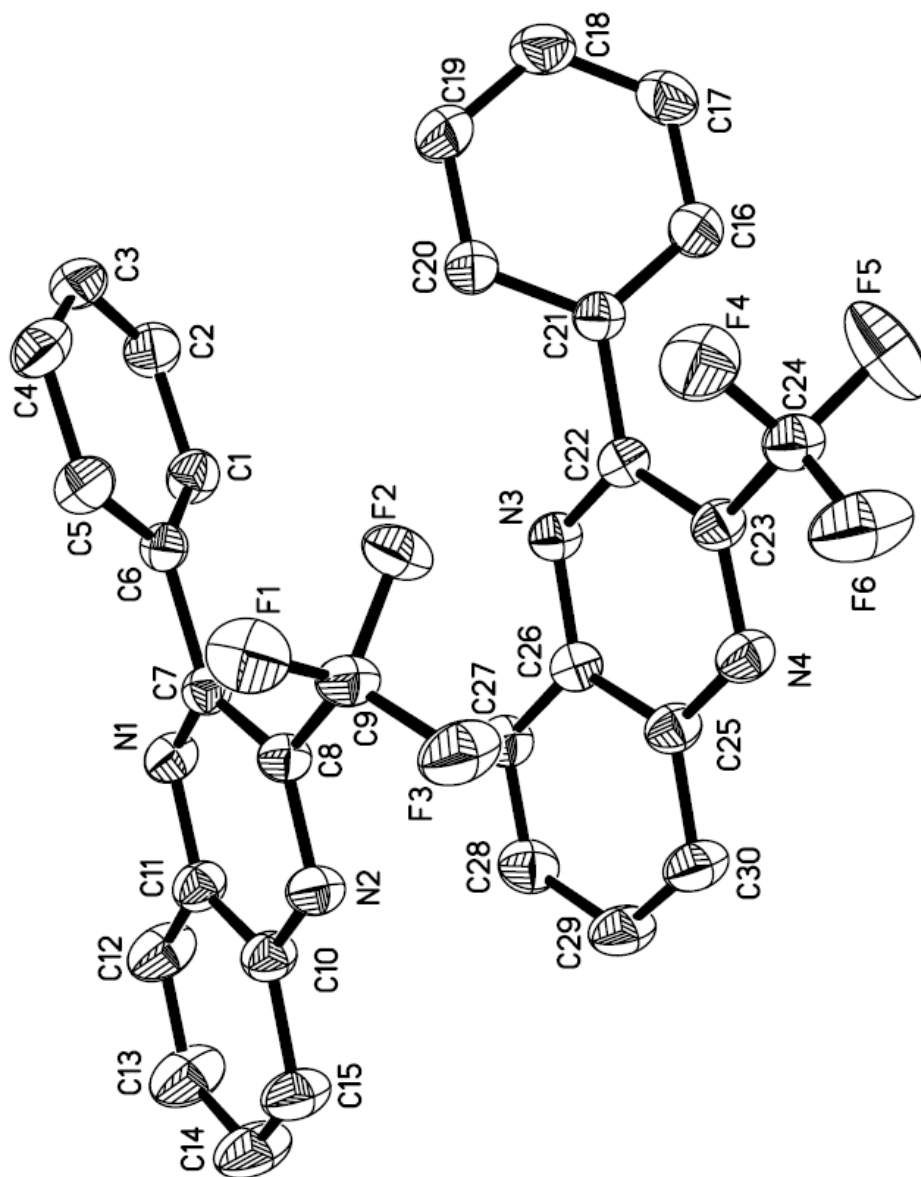


<sup>13</sup>C-NMR: 2,2,6,6-Tetramethyl-1-nitrosopiperidine (5)



**(E). X-ray crystal structure of compound 2a, 3a**

**(a) X-ray crystal structure of compound 2a**



(b) X-ray crystal structure of compound **3a**

