

Supporting Information

Electrochemical sulfonylation of imidazoheterocycles in Batch and continuous Flow

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I. Materials and methods

All reagents were purchased from commercial suppliers (Fisher scientific, Sigma-Aldrich or Fluorochem) and were used without further purification unless otherwise indicated.

Substituted imidazoheterocycles were synthesized using procedure from the literature.¹ Sodium sulfonate salts **2a** and **2l** were purchased from commercial suppliers, all the other sodium sulfonate salts were prepared according to literature method.²

Electrode materials. The electrodes used in this work for were bought from IKA (<https://www.ika.com/en/Products-Lab-Eq/Electrochemistry-Kit-csp-516/ElectraSyn-20-Package-Accessories-cpacc-20008980/>). For experiments using an ElectraSyn vial (10 mL, for 0.1–0.2 mmol scale), the dimensions of the electrodes were approximately W8 × D2 × H40 mm (with the submerged exterior surface of the electrode approximately W8 × D2 × H35 mm), unless otherwise stated.

For experiments using an ElectraSyn flow cell (<https://www.ikaprocess.com/en/Products/Electro-Organic-Synthesis-Systems-cph-45/>) the dimensions of the electrodes were W20 × H60 mm (Figure 1).

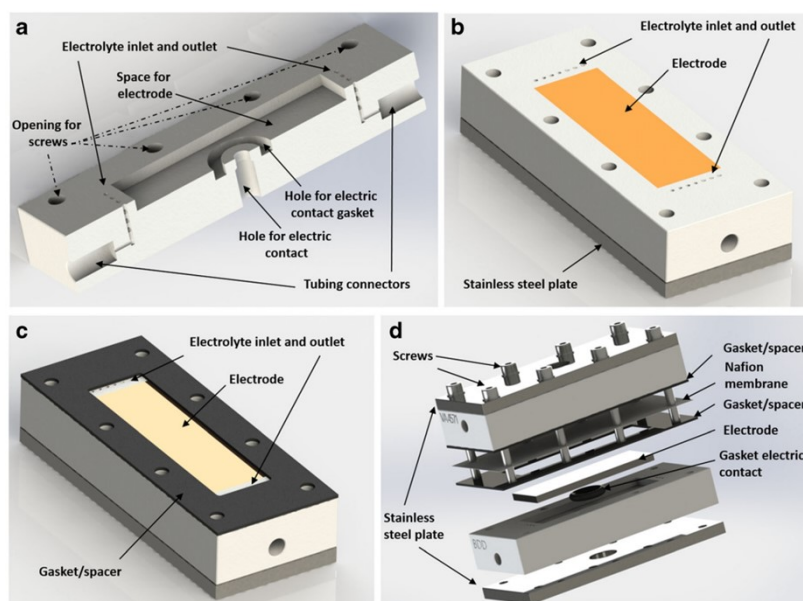


Figure 1. Technical drawings of 2 cm × 6 cm flow electrolysis cell: a Cross-section of the Teflon piece with connection for tubing, inlet, outlet and free space for electrode. b Complete half-cell containing Teflon piece, the electrode (yellow) and a stainless steel plate. c Half-cell with gasket/ spacer on top. d Exploded drawing of a complete divided flow electrolysis cell. For the undivided mode, the Nafion membrane and one gasket/spacer is omitted. Reprinted with permission from Börner *et al. Chem. Rev.* **2012**, 112, 5675–5732. Copyright [2020] American Chemical Society

Power supplies. Electrolysis in batch was conducted using a DC power supply (OrigaFlex OGFWR-OGF01A) in constant current mode. For the ElectraSyn flow cell a power supply Keysight E36104A was applied.

Pumps. Flow-rate was regulated as a function of internal volume and residence time of molecules in the reactor thanks to the formula $Q = V/t_r$, where Q is the flow rate ($\mu\text{L}\cdot\text{min}^{-1}$), V the internal volume of the reactor and t_r the residence time of molecules in the reactor. The

different flow rates of the reactions were regulated using a Chemyx Fusion 200-X syringe pump fitted with 20 mL plastic syringes from HENKE-JECT.

Analytical thin-layer chromatography (TLC) were performed on 0.25 mm E. Merck silica plates (60F-254), using short-wave UV light as the visualizing agent, and KMnO₄ and heat as developing agents. Column chromatography was performed using E. Merck silica gel (60, particle size 0.043–0.063 mm).

NMR spectra were recorded on a Bruker AVANCEIIIHD 300 spectrometer and are calibrated using residual undeuterated solvent (CDCl₃ at 7.26 ppm ¹H NMR, 77.16 ppm ¹³C NMR). Chemical shifts of ¹H NMR and ¹³C NMR were recorded in parts per million (ppm, δ) relative to solvent signal. The following abbreviations are used for the proton spectra multiplicities: s=singlet; d=doublet; t=triplet; q=quartet; m=multiplet. Coupling constant are reported in hertz (Hz).

High-resolution mass spectra (HRMS) were recorded with a Maxis Bruker 4G instrument and were performed in positive mode with an ESI source on a Q-TOF mass spectrometer with an accuracy tolerance of 2 ppm.

II. Cyclic voltammetry experiments

Cyclic voltammetry was measured under N_2 atmosphere with conventional three electrode system (Reference electrode: Ag/AgCl, working electrode: Glassy carbon, counter electrode: Pt wire, Supporting electrolyte: 0.1 M nBu_4NClO_4 in MeCN:H₂O v/v (5:2) on a Metrohm PGSTAT128N potentiostat/galvanostat. Conventional concentrations of 1 mM electroactive species were used. Acquisitions were performed with 100 mV/s scan rates.

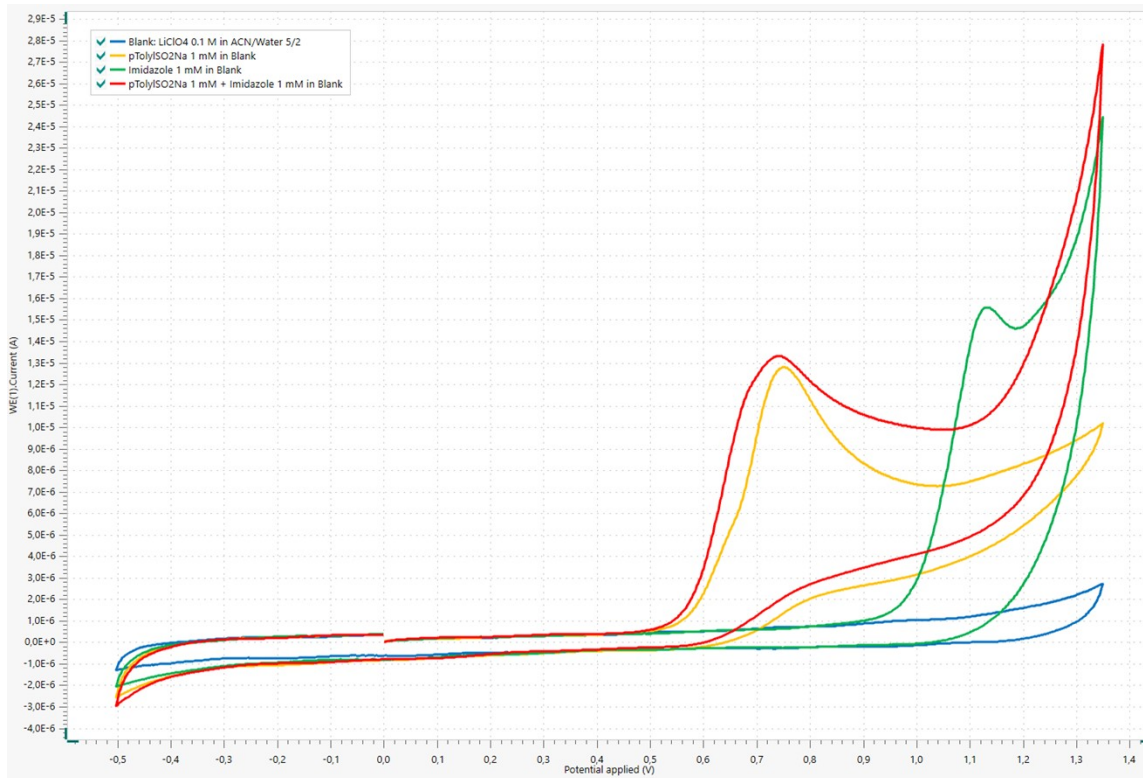


Figure 2 : Example of cyclic voltammetry studies.

III. Experimental procedures and characterization data

A. General Procedure A. Electrochemical sulfonylation under batch condition

Unless otherwise specified, the reaction was carried out on 0.257 mmol scale. To a 10 mL undivided ElectroSyn vial equipped with a stirrer bar was added imidazoheterocycles (0.257 mmol, 1 eq), sulfinate salts (0.514 mmol, 2 eq), $n\text{Bu}_4\text{NClO}_4$ (342 mg, 0.15 M) and then a mixture of MeCN:H₂O (5:2, 10.0 mL). The ElectroSyn vial cap equipped with anode (graphite) and cathode (platinum) was inserted into the mixture. The reaction mixture was electrolyzed at a constant current of 6 mA under air at room temperature for 5 hours. After electrolysis, the cap was removed, and the electrodes were taken out and rinsed with EtOAc into the reaction mixture. The solution was then concentrated under vacuum, water was added to the residue and the resulting mixture was extracted with EtOAc. The combined organics were washed with brine, dried over anhydrous Na₂SO₄ and concentrated under reduced pressure [Note: Yield may be determined by analysis of ¹H-NMR spectra of crude reaction product using an internal standard (1,3,5-trimethoxybenzene) at this point]. The crude material was purified by silica gel column chromatography to furnish the desired product.

B. General Procedure B. Electrochemical sulfonylation under flow condition

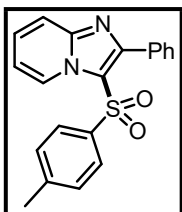
Unless otherwise specified, the reaction was carried out on 0.207 mmol scale. A solution of 2 imidazoheterocycles (0.207 mmol, 1 eq), sulfinate salts (0.358 mmol, 1.7 eq), $n\text{Bu}_4\text{NClO}_4$ (274 mg, 0.1 M) in a mixture of MeCN:H₂O (5:2, 8.0 mL) was pumped through the ElectraSyn flow cell (H = 500 μm , L = 60 cm, I = 20 cm, V = 0.6 mL) and was electrolyzed for 15 min ($Q = 0.04 \text{ mL} \cdot \text{min}^{-1}$) at a constant current of 6 mA at room temperature. The solution was then concentrated under vacuum, water was added to the residue and the resulting mixture was extracted with EtOAc. The solution was then concentrated under vacuum. Water was added, and the resulting mixture was extracted with EtOAc. The combined organics were washed with brine, dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The crude material was purified by silica gel column chromatography to furnish the desired product.

2-phenyl-3-tosylimidazo[1,2-*a*]pyridine **3aa**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 91% yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 74.3 mg (83%) of the title compound **3aa**.

Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 61.3 mg (85%) of the title compound **3aa**.

Spectral data are in agreement with the literature.³

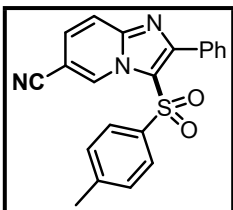


¹H NMR (300 MHz, CDCl₃): δ 9.10 (d, $J = 7.0$ Hz, 1H), 7.77 - 7.68 (m, 3H), 7.52 (d, $J = 8.2$ Hz, 2H), 7.48 - 7.40 (m, 4H), 7.13 (d, $J = 8.1$ Hz, 2H), 7.04 (t, $J = 6.7$ Hz, 1H), 2.31 (s, 3H). ¹³C NMR (75 MHz, CDCl₃): δ 152.9, 146.7, 144.5, 139.2, 132.8, 130.6 (2), 129.8 (2), 129.4, 128.5, 127.9 (2), 126.9, 126.5 (2), 118.1, 117.9, 114.7, 21.6. HRMS (ESI): m/z [M+H]⁺ calc. for C₂₀H₁₇N₂O₂S, 349.1005. found

349.1004.

2-phenyl-3-tosylimidazo[1,2-a]pyridine-6-carbonitrile **3ba**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 36% yield. Purification by silica gel column chromatography (100% cyclohexane to 2:1 cyclohexanes/EtOAc) afforded 28.8 mg (30%) of the title compound **3ba**.



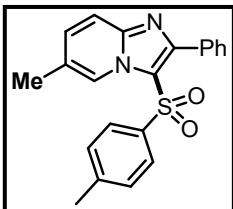
¹H NMR (300 MHz, CDCl₃): δ 9.62 (dd, *J* = 1.6, 1.0 Hz, 1H), 7.78 (dd, *J* = 9.3, 0.9 Hz, 1H), 7.75 – 7.69 (m, 2H), 7.54 (dd, *J* = 9.3, 1.6 Hz, 1H), 7.51 – 7.43 (m, 5H), 7.18 – 7.13 (m, 2H), 2.34 (s, 3H). ¹³C NMR (75 MHz, CDCl₃): δ 154.75, 146.13, 145.76, 138.75, 133.36, 132.07 (2), 131.01, 130.52, 130.45 (2), 128.72, 128.57 (2), 127.12 (2), 120.21, 119.54, 116.42, 101.52, 22.11. HRMS (ESI): *m/z* [M+H]⁺ calc. for C₂₁H₁₆N₃O₂S, 374.0957 found 374.0963.

6-methyl-2-phenyl-3-tosylimidazo[1,2-a]pyridine **3ca**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 79% yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 65.5 mg (70%) of the title compound **3ca**.

Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 51.1 mg (68%) of the title compound **3ca**.

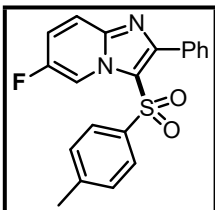
Spectral data are in agreement with the literature.³



¹H NMR (300 MHz, CDCl₃): δ 8.78 (d, *J* = 0.7 Hz, 1H), 7.62 (d, *J* = 2.2 Hz, 1H), 7.61 – 7.59 (m, 1H), 7.49 (d, *J* = 9.1 Hz, 1H), 7.41 (d, *J* = 8.4 Hz, 2H), 7.33 (dd, *J* = 5.0, 1.8 Hz, 3H), 7.20 – 7.16 (m, 1H), 7.02 (d, *J* = 8.0 Hz, 2H), 2.30 (s, 3H), 2.21 (s, 3H). ¹³C NMR (75 MHz, CDCl₃): δ 152.7, 145.7, 144.3, 139.3, 132.9, 132.9, 131.5, 130.6 (2), 129.7 (2), 129.2, 127.8 (2), 126.4 (2), 124.7, 124.6, 117.4, 117.2, 21.6, 18.7. HRMS (ESI): *m/z* [M+H]⁺ calc. for C₂₁H₁₉N₂O₂S, 363.1161 found 363.1160.

6-fluoro-2-phenyl-3-tosylimidazo[1,2-a]pyridine **3da**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 59% yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 47.0 mg (50%) of the title compound **3da**. Following Spectral data are in agreement with the literature.³



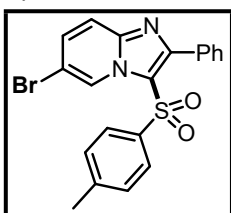
¹H NMR (300 MHz, CDCl₃): δ 9.13 (dd, *J* = 4.5, 2.1 Hz, 1H), 7.73 – 7.66 (m, 3H), 7.50 (d, *J* = 8.4 Hz, 2H), 7.46 (dd, *J* = 5.0, 2.3 Hz, 3H), 7.37 (ddd, *J* = 9.9, 7.6, 2.4 Hz, 1H), 7.14 (d, *J* = 8.1 Hz, 2H), 2.33 (s, 3H). ¹³C NMR (75 MHz, CDCl₃): δ 154.09 (d, *J* = 240.0 Hz), 153.5, 144.8, 144.2, 138.9, 132.5, 130.6 (2), 129.9 (2), 129.6, 128.0 (2), 126.6 (2), 120.4 (d, *J* = 25.2 Hz), 119.4, 118.5 (d, *J* = 8.8

Hz), 114.5 (d, $J = 43.7$ Hz), 21.7. **HRMS** (ESI): m/z $[M+H]^+$ calc. for $C_{20}H_{16}FN_2O_2S$, 367.0911 found 367.0911.

6-bromo-2-phenyl-3-tosylimidazo[1,2-*a*]pyridine **3ea**

Following General Procedure A on 0.257 mmol scale with 6 h of reaction time. Purification by silica gel column chromatography (100% cyclohexane to 2:1 cyclohexanes/EtOAc) afforded 60.2 mg (55%) of the title compound **3ea**.

Spectral data are in agreement with the literature.³



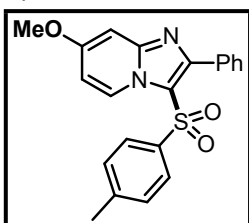
¹H NMR (300 MHz, $CDCl_3$): δ 9.31 (dd, $J = 1.8, 0.8$ Hz, 1H), 7.70 (dd, $J = 7.5, 2.1$ Hz, 2H), 7.60 (dd, $J = 9.4, 0.7$ Hz, 1H), 7.54 - 7.43 (m, 6H), 7.15 (d, $J = 8.1$ Hz, 2H), 2.34 (s, 3H). **¹³C NMR** (75 MHz, $CDCl_3$): δ 152.9, 145.0, 144.9, 138.8, 132.2 (2), 130.6 (2), 129.9 (2), 129.7, 128.0 (2), 127.1, 126.6 (2), 118.8, 118.6, 109.7, 21.7. **HRMS** (ESI): m/z $[M+H]^+$ calc. for $C_{20}H_{16}BrN_2O_2S$, 427.0110 found 427.0111.

7-methoxy-2-phenyl-3-tosylimidazo[1,2-*a*]pyridine **3fa**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 77% yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 68.0 mg (70%) of the title compound **3fa**.

Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 60.2 mg (77%) of the title compound **3fa**.

Spectral data are in agreement with the literature.³

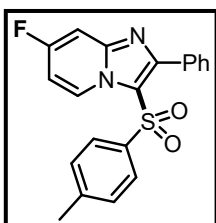


¹H NMR (300 MHz, $CDCl_3$): δ 8.89 (d, $J = 7.7$ Hz, 1H), 7.74 (dd, $J = 6.5, 3.0$ Hz, 2H), 7.53 - 7.42 (m, 5H), 7.12 (d, $J = 8.1$ Hz, 2H), 6.95 (d, $J = 2.3$ Hz, 1H), 6.71 (dd, $J = 7.7, 2.6$ Hz, 1H), 3.86 (s, 3H), 2.31 (s, 3H). **¹³C NMR** (75 MHz, $CDCl_3$): δ 160.5, 153.3, 148.8, 144.2, 139.5, 132.8, 130.6 (2), 129.7 (2), 129.4, 127.8 (2), 127.3, 126.3 (2), 116.6, 109.1, 95.6, 55.9, 21.6. **HRMS** (ESI): m/z $[M+H]^+$ calc. for $C_{21}H_{19}N_2O_3S$, 379.1110 found 379.1118.

7-fluoro-2-phenyl-3-tosylimidazo[1,2-*a*]pyridine **3ga**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 80% yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 65.9 mg (70%) of the title compound **3ga**.

Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 38.1 mg (50%) of the title compound **3ga**.



¹H NMR (300 MHz, $CDCl_3$): δ 9.12 (dd, $J = 7.7, 5.5$ Hz, 1H), 7.79 - 7.69 (m, 2H), 7.53 - 7.41 (m, 5H), 7.31 (dd, $J = 8.4, 2.4$ Hz, 1H), 7.12 (d, $J = 8.0$ Hz, 2H), 6.94 - 6.84 (m, 1H). **¹³C NMR** (75 MHz, $CDCl_3$): δ 162.2 (d, $J = 257.5$ Hz), 153.9,

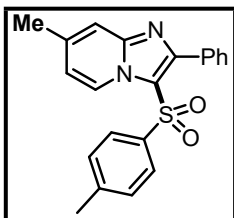
147.5 (d, J = 13.7 Hz), 144.6, 138.9, 132.4, 130.5 (2), 129.8 (2), 129.6, 128.71 (d, J = 10.7 Hz), 127.9 (2), 126.4 (2), 118.0, 106.7 (d, J = 28.3 Hz), 102.0 (d, J = 23.5 Hz), 21.6. **HRMS** (ESI): m/z $[M+H]^+$ calc. for $C_{20}H_{16}FN_2O_2S$, 367.0911 found 367.0911.

7-methyl-2-phenyl-3-tosylimidazo[1,2-*a*]pyridine **3ha**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of 1H -NMR spectra of crude reaction product: 81% yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 54.0 mg (58%) of the title compound **3ha**.

Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 47.1 mg (63%) of the title compound **3ha**.

Spectral data are in agreement with the literature.³

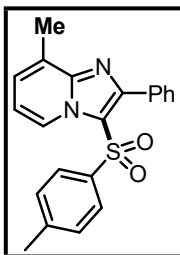


1H NMR (300 MHz, $CDCl_3$): δ 8.96 (d, J = 7.2 Hz, 1H), 7.76 - 7.70 (m, 2H), 7.50 (d, J = 8.3 Hz, 2H), 7.47 - 7.41 (m, 4H), 7.12 (d, J = 8.0 Hz, 2H), 6.87 (dd, J = 7.2, 1.7 Hz, 1H), 2.44 (s, 3H), 2.31 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$): δ 153.1, 147.2, 144.3, 140.1, 139.4, 132.9, 130.6 (2), 129.8 (2), 129.4, 127.9 (2), 126.4 (2), 126.0, 117.2, 116.6, 21.6, 21.5. **HRMS** (ESI): m/z $[M+H]^+$ calc. for $C_{21}H_{19}N_2O_2S$, 363.1161 found 363.1160.

8-methyl-2-phenyl-3-tosylimidazo[1,2-*a*]pyridine **3ia**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of 1H -NMR spectra of crude reaction product: 85 % yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 72.6 mg (78%) of the title compound **3ia**. Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 63.1 mg (84%) of the title compound **3ia**.

Spectral data are in agreement with the literature.³

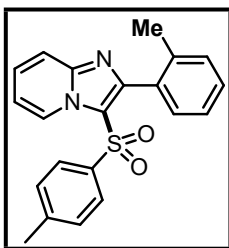


1H NMR (300 MHz, $CDCl_3$): δ 8.96 (d, J = 7.0 Hz, 1H), 7.74 - 7.68 (m, 2H), 7.52 (d, J = 8.4 Hz, 2H), 7.49 - 7.42 (m, 3H), 7.25 - 7.20 (m, 1H), 7.13 (d, J = 8.0 Hz, 2H), 6.95 (t, J = 7.0 Hz, 1H), 2.63 (s, 3H), 2.32 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$): δ 152.4, 147.0, 144.4, 139.3, 133.1, 130.7 (2), 129.7 (2), 129.3, 128.2, 127.9 (2), 127.4, 126.5 (2), 124.6, 118.2, 114.7, 21.6, 17.2. **HRMS** (ESI): m/z $[M+H]^+$ calc. for $C_{21}H_{19}N_2O_2S$, 363.1161 found 363.1161

2-(*o*-tolyl)-3-tosylimidazo[1,2-*a*]pyridine **3ka**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of 1H -NMR spectra of crude reaction product: 91 % yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 77.2 mg (83%) of the title compound **3ka**.

Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 63.8 mg (85%) of the title compound

3ka.

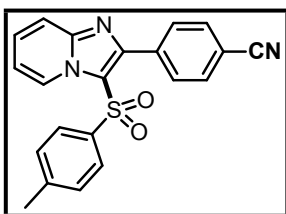
¹H NMR (300 MHz, CDCl₃): δ 9.14 (dt, *J* = 7.0, 1.1 Hz, 1H), 7.69 (dt, *J* = 9.0, 1.1 Hz, 1H), 7.54 - 7.47 (m, 2H), 7.46 - 7.41 (m, 1H), 7.40 - 7.31 (m, 1H), 7.27 - 7.22 (m, 3H), 7.15 (d, *J* = 8.0 Hz, 2H), 7.07 (td, *J* = 7.0, 1.3 Hz, 1H), 2.35 (s, 3H), 2.03 (s, 3H). **¹³C NMR** (75 MHz, CDCl₃): δ 152.3, 146.5, 144.6, 139.10, 137.7, 132.5, 130.7, 129.9, 129.7 (2), 129.3, 128.3, 126.8, 126.8 (2), 125.0, 119.0, 118.1, 114.6, 21.7, 20.1. **HRMS** (ESI): *m/z* [M+H]⁺ calc. for C₂₁H₁₉N₂O₂S, 363.1161 found 363.1162.

4-(3-tosylimidazo[1,2-*a*]pyridin-2-yl)benzonitrile 3la

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 83% yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 71.9 mg (75%) of the title compound **3ka**.

Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 59.4 mg (77%) of the title compound **3ka**.

Spectral data are in agreement with the literature.⁴



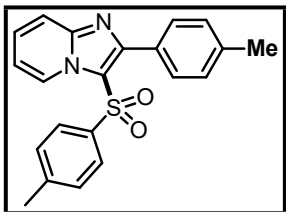
¹H NMR (300 MHz, CDCl₃): δ 9.06 (dt, *J* = 7.0, 1.0 Hz, 1H), 7.94 - 7.86 (m, 2H), 7.80 - 7.68 (m, 3H), 7.56 - 7.45 (m, 3H), 7.18 (d, *J* = 8.0 Hz, 2H), 7.10 (td, *J* = 7.0, 1.2 Hz, 1H), 2.34 (s, 3H). **¹³C NMR** (75 MHz, CDCl₃): δ 150.3, 146.7, 144.9, 138.6, 137.2, 131.5 (2), 131.3 (2), 129.9 (2), 128.9, 126.7, 126.3 (2), 118.6, 118.4, 118.2, 115.0, 112.9, 21.5. **HRMS** (ESI): *m/z* [M+H]⁺ calc. for C₂₁H₁₆N₃O₂S, 374.0957 found 374.0963.

2-(*p*-tolyl)-3-tosylimidazo[1,2-*a*]pyridine 3ma

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 79% yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 64.2 mg (69%) of the title compound **3ma**.

Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 52.6 mg (70%) of the title compound **3ma**.

Spectral data are in agreement with the literature.³



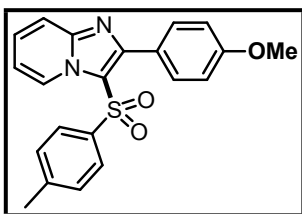
¹H NMR (300 MHz, CDCl₃): δ 9.08 (dt, *J* = 7.0, 1.1 Hz, 1H), 7.76 - 7.62 (m, 3H), 7.59 - 7.51 (m, 2H), 7.43 (ddd, *J* = 9.0, 6.9, 1.2 Hz, 1H), 7.26 (d, *J* = 7.9 Hz, 2H), 7.14 (d, *J* = 8.0 Hz, 2H), 7.02 (td, *J* = 7.0, 1.3 Hz, 1H), 2.43 (s, 3H), 2.32 (s, 3H). **¹³C NMR** (75 MHz, CDCl₃): δ 153.1, 146.7, 144.4, 139.4, 139.3, 130.6 (2), 129.8 (2), 128.6 (2), 128.5, 126.9, 126.5 (2), 118.0, 117.6, 114.6, 21.6, 21.6. **HRMS** (ESI): *m/z* [M+H]⁺ calc. for C₂₁H₁₉N₂O₂S, 363.1161 found 363.1162.

2-(4-methoxyphenyl)-3-tosylimidazo[1,2-a]pyridine **3na**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ^1H -NMR spectra of crude reaction product: 86% yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 73.9 mg (76%) of the title compound **3na**.

Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 56.4 mg (72%) of the title compound **3na**.

Spectral data are in agreement with the literature.³



^1H NMR (300 MHz, CDCl_3): δ 9.01 (d, J = 7.0 Hz, 1H), 7.71 - 7.64 (m, 2H), 7.61 (d, J = 9.0 Hz, 1H), 7.45 (d, J = 8.3 Hz, 2H), 7.39 - 7.31 (m, 1H), 7.06 (d, J = 8.1 Hz, 2H), 6.93 (ddd, J = 12.5, 6.9, 1.6 Hz, 3H), 3.81 (s, 3H), 2.24 (s, 3H). ^{13}C NMR (75 MHz, CDCl_3): δ 160.3, 152.5, 146.3, 144.0, 138.9, 131.7 (2), 129.4 (4), 128.1, 126.5, 126.0 (2), 124.7, 117.5, 116.9, 114.1, 113.0 (2), 55.0, 21.2. HRMS (ESI): m/z $[\text{M}+\text{H}]^+$

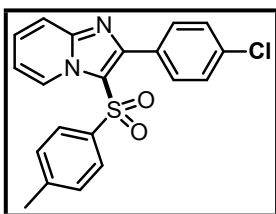
calc. for $\text{C}_{21}\text{H}_{19}\text{N}_2\text{O}_3\text{S}$, 379.1110 found 379.1118.

2-(4-chlorophenyl)-3-tosylimidazo[1,2-a]pyridine **3oa**

Following General Procedure A on 0.257 mmol scale with 6 h of reaction time. Yield determined by analysis of ^1H -NMR spectra of crude reaction product: 78 % yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 68.7 mg (70%) of the title compound **3oa**.

Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 27.6 mg (35%) of the title compound **3oa**.

Spectral data are in agreement with the literature.³



^1H NMR (300 MHz, CDCl_3): δ 9.08 (d, J = 7.0 Hz, 1H), 7.76 - 7.67 (m, 3H), 7.53 (d, J = 8.3 Hz, 2H), 7.46 (dd, J = 13.4, 4.7 Hz, 3H), 7.16 (d, J = 8.1 Hz, 2H), 7.06 (td, J = 7.0, 1.0 Hz, 1H), 2.33 (s, 3H). ^{13}C NMR (75 MHz, CDCl_3): δ 151.6, 146.7, 144.7, 139.0, 135.7, 132.1 (2), 131.3, 129.9 (2), 128.8, 128.2 (2), 126.9, 126.5 (2), 118.1, 118.0, 114.8, 21.7. HRMS (ESI): m/z $[\text{M}+\text{H}]^+$ calc. for $\text{C}_{20}\text{H}_{16}\text{ClN}_2\text{O}_2\text{S}$, 383.0615 found 383.0615.

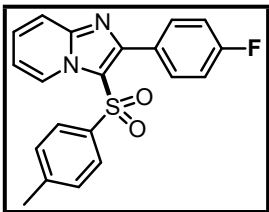
2-(4-fluorophenyl)-3-tosylimidazo[1,2-a]pyridine **3pa**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ^1H -NMR spectra of crude reaction product: 79% yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 65.0 mg (69%) of the title compound **3pa**.

Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 48.4 mg (64%) of the title compound

3pa.

Spectral data are in agreement with the literature.³



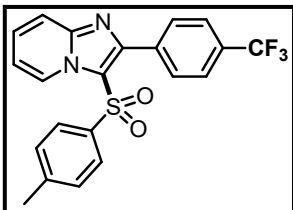
¹H NMR (300 MHz, CDCl₃): δ 9.02 (dt, *J* = 7.1, 1.1 Hz, 1H), 7.74 - 7.59 (m, 3H), 7.49 - 7.33 (m, 3H), 7.08 (ddd, *J* = 8.8, 4.8, 2.5 Hz, 4H), 6.98 (td, *J* = 7.0, 1.2 Hz, 1H), 2.26 (s, 3H). **¹³C NMR** (75 MHz, CDCl₃): δ 163.8 (d, *J* = 249.1 Hz), 151.9, 146.7, 144.7, 139.1, 132.7, 132.6, 129.9 (2), 128.9 (d, *J* = 3.2 Hz), 128.7, 127.0, 126.5 (2), 118.1, 117.9, 115.2, 114.9, 114.8, 21.7. **HRMS** (ESI): *m/z* [M+H]⁺ calc. for C₂₀H₁₆FN₂O₂S, 367.0911 found 367.0911.

3-tosyl-2-(4-(trifluoromethyl)phenyl)imidazo[1,2-a]pyridine 3qa

Following General Procedure A on 0.257 mmol scale with 6 h of reaction time. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 42% yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 37.4 mg (35%) of the title compound **3qa**.

Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (98:2 Dichloromethane/EtOAc) afforded 18.9 mg (22%) of the title compound **3qa**.

Spectral data are in agreement with the literature.³

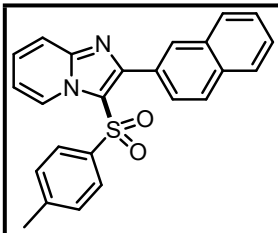


¹H NMR (300 MHz, CDCl₃): δ 9.08 (dt, *J* = 7.1, 1.0 Hz, 1H), 7.89 (d, *J* = 8.1 Hz, 2H), 7.79 - 7.68 (m, 3H), 7.53 (d, *J* = 8.4 Hz, 2H), 7.51 - 7.44 (m, 1H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.08 (td, *J* = 7.0, 1.2 Hz, 1H), 2.33 (s, 3H). **¹³C NMR** (75 MHz, CDCl₃): δ 151.0, 146.8, 144.9, 138.8, 136.4, 131.5, 131.1 (2), 130.0 (2), 128.9, 126.9, 126.5 (2), 124.9, 124.8, 124.2 (d, *J* = 270.6 Hz), 118.5, 118.3, 115.1, 21.6. **HRMS** (ESI): *m/z* [M+H]⁺ calc. for C₂₁H₁₆F₃N₂O₂S, 417.0879 found 417.0879.

2-(naphthalen-2-yl)-3-tosylimidazo[1,2-a]pyridine 3ra

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 70% yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 66.5 mg (65%) of the title compound **3ra**.

Spectral data are in agreement with the literature.⁴

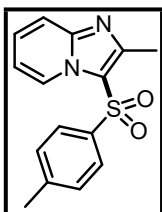


¹H NMR (300 MHz, CDCl₃): δ 9.14 (d, *J* = 7.0 Hz, 1H), 8.29 (s, 1H), 7.98 - 7.85 (m, 4H), 7.73 (d, *J* = 9.0 Hz, 1H), 7.53 (dd, *J* = 6.4, 1.9 Hz, 4H), 7.49 - 7.40 (m, 1H), 7.07 (d, *J* = 8.1 Hz, 2H), 7.03 (d, *J* = 7.0 Hz, 1H), 2.26 (s, 3H). **¹³C NMR** (75 MHz, CDCl₃): δ 152.7, 146.8, 144.5, 139.1, 133.7, 132.8, 130.6, 130.2, 129.7 (2), 128.8, 128.6, 127.9, 127.8, 127.4, 126.9, 126.9, 126.5 (2), 126.3, 118.1, 118.0, 114.7, 21.5. **HRMS** (ESI): *m/z* [M+H]⁺ calc. for C₂₄H₁₉N₂O₂S, 399.1161 found 399.1158.

2-methyl-3-tosylimidazo[1,2-*a*]pyridine 3sa

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 20% yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 9.6 mg (13%) of the title compound **3sa**.

Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 10.0 mg (17%) of the title compound **3sa**.



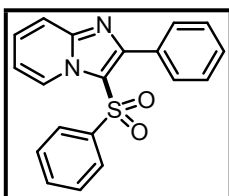
¹H NMR (300 MHz, CDCl₃): δ 8.82 (dt, *J* = 7.0, 1.1 Hz, 1H), 7.83 (d, *J* = 8.4 Hz, 2H), 7.59 (dt, *J* = 9.0, 1.0 Hz, 1H), 7.37 (ddd, *J* = 9.0, 6.9, 1.2 Hz, 1H), 7.29 (d, *J* = 8.3 Hz, 2H), 6.97 (td, *J* = 6.9, 1.2 Hz, 1H), 2.79 (s, 3H), 2.38 (s, 3H). **¹³C NMR** (75 MHz, CDCl₃): δ 151.2, 146.7, 144.7, 139.3, 130.1 (2), 129.6, 128.2, 126.3 (2), 126.0, 117.4, 114.2, 21.7, 15.4. **HRMS** (ESI): *m/z* [M+H]⁺ calc. for C₁₅H₁₅N₂O₂S, 287.0848. found 287.0846.

2-phenyl-3-(phenylsulfonyl)imidazo[1,2-*a*]pyridine 3ab

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 86 % yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 65.3 mg (76%) of the title compound **3ab**.

Following General Procedure B on 0.257 mmol scale. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 54.8 mg (79%) of the title compound **3ab**.

Spectral data are in agreement with the literature.³



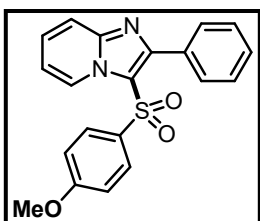
¹H NMR (300 MHz, CDCl₃): δ 9.13 (dt, *J* = 7.0, 1.0 Hz, 1H), 7.72 (ddd, *J* = 6.2, 3.4, 1.5 Hz, 3H), 7.62 (dd, *J* = 5.3, 3.3 Hz, 2H), 7.51 - 7.40 (m, 5H), 7.38 - 7.30 (m, 2H), 7.06 (td, *J* = 7.0, 1.2 Hz, 1H). **¹³C NMR** (75 MHz, CDCl₃): δ 153.3, 146.8, 142.1, 133.5, 132.7, 130.6 (2), 129.5, 129.2 (2), 128.7, 127.9 (2), 127.0, 126.4 (2), 118.2, 117.6, 114.8. **HRMS** (ESI): *m/z* [M+H]⁺ calc. for C₁₉H₁₅FN₂O₂S, 335.0848 found 335.0848.

3-((4-methoxyphenyl)sulfonyl)-2-phenylimidazo[1,2-*a*]pyridine 3ac

Following General Procedure A on 0.257 mmol scale on 1 h of reaction time. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 26 % yield.

Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 64.9 mg (86%) of the title compound **3ac**.

Spectral data are in agreement with the literature.³



¹H NMR (300 MHz, CDCl₃): δ 9.11 (d, *J* = 7.0 Hz, 1H), 7.72 (ddd, *J* = 9.1, 6.7, 4.6 Hz, 3H), 7.61 - 7.53 (m, 2H), 7.43 (ddd, *J* = 6.9, 5.7, 1.6 Hz, 4H),

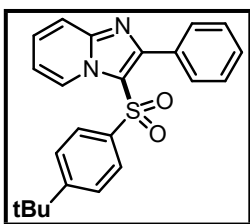
7.04 (td, $J = 7.0, 1.2$ Hz, 1H), 6.82 - 6.75 (m, 2H), 3.77 (s, 3H). ^{13}C NMR (75 MHz, CDCl_3): δ 163.6, 152.6, 146.6, 133.7, 132.9, 130.7 (2), 129.4, 128.8 (2), 128.4, 127.9 (2), 126.9, 118.4, 118.1, 114.6, 114.3 (2), 55.7. HRMS (ESI): m/z $[\text{M}+\text{H}]^+$ calc. for $\text{C}_{20}\text{H}_{17}\text{N}_2\text{O}_3\text{S}$, 365.0954 found 365.0953.

3-((4-(*tert*-butyl)phenyl)sulfonyl)-2-phenylimidazo[1,2-*a*]pyridine 3ad

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ^1H -NMR spectra of crude reaction product: 81% yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 80.2 mg (80%) of the title compound **3ad**.

Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (100% cyclohexane to 2:1 cyclohexanes/EtOAc) afforded 73.4 mg (91%) of the title compound **3ad**.

Spectral data are in agreement with the literature.³



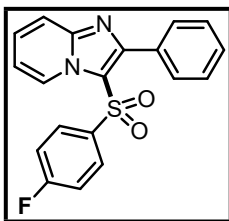
^1H NMR (300 MHz, CDCl_3): δ 9.13 (dt, $J = 7.0, 1.0$ Hz, 1H), 7.75 - 7.69 (m, 3H), 7.56 (d, $J = 8.8$ Hz, 2H), 7.48 - 7.42 (m, 4H), 7.35 (d, $J = 8.7$ Hz, 2H), 7.06 (td, $J = 7.0, 1.2$ Hz, 1H), 1.24 (s, 9H). ^{13}C NMR (75 MHz, CDCl_3): δ 157.5, 152.8, 146.6, 139.0, 132.7, 130.6 (2), 129.4, 128.6, 127.9 (2), 127.0, 126.4 (2), 126.2 (2), 118.2, 118.0, 114.7, 35.3, 31.1. HRMS (ESI): m/z $[\text{M}+\text{H}]^+$ calc. for $\text{C}_{23}\text{H}_{23}\text{N}_2\text{O}_2\text{S}$, 391.1474 found 391.1475.

3-((4-fluorophenyl)sulfonyl)-2-phenylimidazo[1,2-*a*]pyridine 3ae

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ^1H -NMR spectra of crude reaction product: 98% yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 81.4 mg (90%) of the title compound **3ae**.

Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 67.0 mg (92%) of the title compound **3ae**.

Spectral data are in agreement with the literature.³

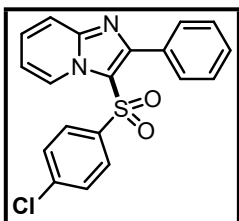


^1H NMR (300 MHz, CDCl_3): δ 9.13 (d, $J = 7.0$ Hz, 1H), 7.78 - 7.68 (m, 3H), 7.60 (dd, $J = 8.8, 5.0$ Hz, 2H), 7.46 (dd, $J = 8.6, 6.9$ Hz, 4H), 7.08 (d, $J = 7.0$ Hz, 1H), 6.98 (t, $J = 8.5$ Hz, 2H). ^{13}C NMR (75 MHz, CDCl_3): δ 165.4 (d, $J = 256.3$ Hz), 153.1, 146.7, 138.0 (d, $J = 2.9$ Hz), 132.5, 130.5 (2), 129.5, 129.3, 129.2, 128.7, 127.9 (2), 126.8, 118.1, 117.4, 116.4, 116.1, 114.8. HRMS (ESI): m/z $[\text{M}+\text{H}]^+$ calc. for $\text{C}_{19}\text{H}_{14}\text{FN}_2\text{O}_2\text{S}$, 353.0754 found 353.0757.

3-((4-chlorophenyl)sulfonyl)-2-phenylimidazo[1,2-*a*]pyridine 3af

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ^1H -NMR spectra of crude reaction product: 87% yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 75.6 mg (80%) of the title compound **3af**.

Spectral data are in agreement with the literature.³



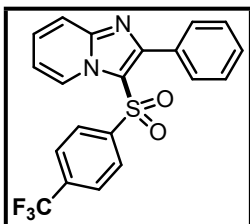
^1H NMR (300 MHz, CDCl_3): δ 9.05 (dt, $J = 7.1, 1.0$ Hz, 1H), 7.70 - 7.59 (m,

3H), 7.46 - 7.35 (m, 6H), 7.21 (d, $J = 8.7$ Hz, 2H), 7.00 (td, $J = 7.0, 1.2$ Hz, 1H). **^{13}C NMR** (75 MHz, CDCl_3): δ 153.4, 146.9, 140.5, 140.1, 132.5, 130.6 (2), 129.7, 129.4 (2), 128.9, 128.0 (2), 127.9 (2), 126.9, 118.2, 117.3, 115.0. **HRMS** (ESI): m/z $[\text{M}+\text{H}]^+$ calc. for $\text{C}_{19}\text{H}_{14}\text{ClN}_2\text{O}_2\text{S}$, 369.0459 found 369.0458.

2-phenyl-3-((4-(trifluoromethyl)phenyl)sulfonyl)imidazo[1,2-*a*]pyridine **3ag**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ^1H -NMR spectra of crude reaction product: 72 % yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 71.3 mg (69%) of the title compound **3ag**. Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 64.0 mg (77%) of the title compound **3ag**.

Spectral data are in agreement with the literature.³



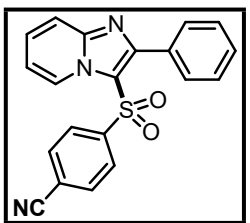
^1H NMR (300 MHz, CDCl_3): δ 9.16 (dt, $J = 7.0, 1.1$ Hz, 1H), 7.78 - 7.65 (m, 5H), 7.58 (d, $J = 8.4$ Hz, 2H), 7.53 - 7.42 (m, 4H), 7.10 (td, $J = 7.0, 1.3$ Hz, 1H). **^{13}C NMR** (75 MHz, CDCl_3): δ 154.0, 147.2, 145.5, 135.0 (td, $J = 33.2, 10.2$ Hz), 132.4, 130.6 (2), 129.8, 129.2, 128.1 (2), 127.0 (3), 126.31 (2C, dd, $J = 7.2, 3.5$ Hz), 123.1 (d, $J = 273.1$ Hz), 118.4, 116.7, 115.2. **HRMS** (ESI): m/z $[\text{M}+\text{H}]^+$ calc. for $\text{C}_{20}\text{H}_{14}\text{F}_3\text{N}_2\text{O}_2\text{S}$, 403.0722 found 403.0725.

4-((2-phenylimidazo[1,2-*a*]pyridin-3-yl)sulfonyl)benzonitrile **3ah**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ^1H -NMR spectra of crude reaction product: 69 % yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 52.6 mg (57%) of the title compound **3ah**.

Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 44.6 mg (60%) of the title compound **3ah**.

Spectral data are in agreement with the literature.⁴

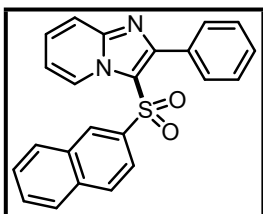


^1H NMR (300 MHz, CDCl_3): δ 9.17 (dt, $J = 7.1, 1.1$ Hz, 1H), 7.76 (dt, $J = 9.0, 1.0$ Hz, 1H), 7.70 - 7.66 (m, 2H), 7.62 (d, $J = 8.2$ Hz, 2H), 7.59 - 7.46 (m, 5H), 7.12 (td, $J = 7.0, 1.3$ Hz, 1H). **^{13}C NMR** (75 MHz, CDCl_3): δ (ppm) 153.3, 146.3, 145.0, 131.9 (2), 131.2, 129.6 (2), 129.0, 128.4, 127.2 (2), 126.0 (3), 117.5, 116.2, 116.1, 115.4, 114.3. **HRMS** (ESI): m/z $[\text{M}+\text{H}]^+$ calc. for $\text{C}_{20}\text{H}_{14}\text{N}_3\text{O}_2\text{S}$, 360.0801 found 360.0800.

3-(naphthalen-2-ylsulfonyl)-2-phenylimidazo[1,2-*a*]pyridine **3ai**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ^1H -NMR spectra of crude reaction product: 20 % yield. Purification by silica gel column chromatography (100 % cyclohexane to 2:1 cyclohexanes/EtOAc) afforded 16.8 mg (17%) of the title compound **3ai**.

Spectral data are in agreement with the literature.⁵



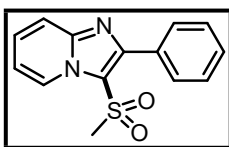
¹H NMR (300 MHz, CDCl₃): δ 9.21 (d, *J* = 7.0 Hz, 1H), 8.23 (d, *J* = 1.5 Hz, 1H), 7.85 – 7.68 (m, 6H), 7.58 (tt, *J* = 12.8, 3.5 Hz, 2H), 7.51 – 7.41 (m, 5H), 7.07 (td, *J* = 7.0, 1.2 Hz, 1H). **¹³C NMR** (75 MHz, CDCl₃): δ 153.36, 146.85, 138.77, 135.17, 132.73, 131.98, 130.73 (2), 129.67, 129.58, 129.52, 129.27, 128.72, 128.04, 127.97, 127.94 (2), 127.74, 127.05, 121.55, 118.19, 117.69, 114.82. **HRMS** (ESI): *m/z* [M+H]⁺ calc. for C₂₃H₁₇N₂O₂S, 385.1005 found 385.1007.

3-(methylsulfonyl)-2-phenylimidazo[1,2-*a*]pyridine **3aj**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 99% yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 62.9 mg (90%) of the title compound **3aj**.

Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 18.0 mg (32%) of the title compound **3aj**.

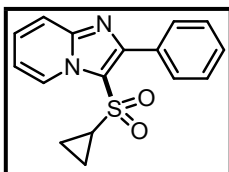
Spectral data are in agreement with the literature.³



¹H NMR (300 MHz, CDCl₃): δ 9.11 (d, *J* = 7.1 Hz, 1H), 7.87 (dd, *J* = 6.5, 3.2 Hz, 2H), 7.77 (d, *J* = 9.0 Hz, 1H), 7.53 - 7.45 (m, 4H), 7.07 (td, *J* = 7.0, 1.2 Hz, 1H), 3.02 (s, 3H). **¹³C NMR** (75 MHz, CDCl₃): δ 151.9, 146.5, 132.5, 130.4 (2), 129.8, 128.7, 128.4 (2), 127.2, 118.2, 117.2, 114.8, 45.1. **HRMS** (ESI): *m/z* [M+H]⁺ calc. for C₁₄H₁₃N₂O₂S, 273.0692 found 273.0690.

3-(cyclopropylsulfonyl)-2-phenylimidazo[1,2-*a*]pyridine **3ak**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 93 % yield. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 65.1 mg (85%) of the title compound **3ak**. Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (9:1 Dichloromethane/EtOAc) afforded 37.0 mg (60%) of the title compound **3ak**.

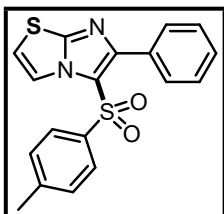


¹H NMR (300 MHz, CDCl₃): δ 9.10 (d, *J* = 7.1 Hz, 1H), 7.88 - 7.80 (m, 2H), 7.76 (d, *J* = 9.0 Hz, 1H), 7.46 (dt, *J* = 5.8, 2.8 Hz, 4H), 7.04 (td, *J* = 7.0, 1.2 Hz, 1H), 2.49 (tt, *J* = 8.0, 4.8 Hz, 1H), 1.18 - 1.11 (m, 2H), 0.84 (dd, *J* = 7.9, 2.0 Hz, 2H). **¹³C NMR** (75 MHz, CDCl₃): δ 152.3, 146.6, 132.9, 130.5 (2), 129.5, 128.5, 128.1 (2), 127.2, 118.1, 117.8, 114.6, 34.5, 5.5. **HRMS** (ESI): *m/z* [M+H]⁺ calc. for C₁₆H₁₅N₂O₂S, 299.0848 found 299.0846.

6-phenyl-5-tosylimidazo[2,1-*b*]thiazole **5a**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 59% yield. Purification by silica gel column chromatography

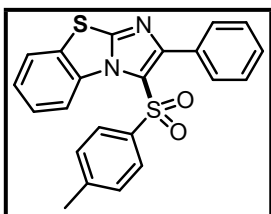
(100% cyclohexane to 2:1 cyclohexanes/EtOAc) afforded 51.0 mg (56%) of the title compound **5a**.



¹H NMR (300 MHz, CDCl₃): δ 8.20 (d, *J* = 4.5 Hz, 1H), 7.81 - 7.75 (m, 2H), 7.49 (d, *J* = 8.4 Hz, 2H), 7.45 - 7.40 (m, 3H), 7.12 (d, *J* = 8.0 Hz, 2H), 7.01 (d, *J* = 4.5 Hz, 1H), 2.31 (s, 3H). **¹³C NMR** (75 MHz, CDCl₃): δ 152.9, 152.6, 144.5, 139.2, 132.4, 130.0 (2), 129.8 (2), 129.4, 128.1 (2), 126.5 (2), 121.0, 120.7, 114.4, 21.6. **HRMS** (ESI): *m/z* [M+H]⁺ calc. for C₁₈H₁₅N₂O₂S₂, 349. found 349.

2-phenyl-3-tosylbenzo[d]imidazo[2,1-b]thiazole **5b**

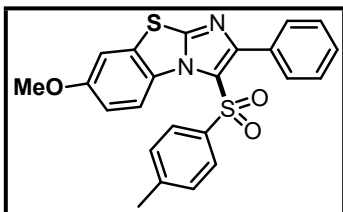
Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 59% yield. Purification by silica gel column chromatography (100% cyclohexane to 2:1 cyclohexanes/EtOAc) afforded 58.2 mg (56%) of the title compound **5b**.



¹H NMR (300 MHz, CDCl₃): δ 8.81 (dd, *J* = 8.4, 0.5 Hz, 1H), 7.71 (dd, *J* = 7.9, 1.0 Hz, 1H), 7.67 - 7.63 (m, 2H), 7.59 (d, *J* = 8.4 Hz, 2H), 7.49 (dd, *J* = 8.5, 1.3 Hz, 1H), 7.41 (dd, *J* = 5.5, 1.1 Hz, 4H), 7.15 (d, *J* = 8.0 Hz, 2H), 2.32 (s, 3H). **¹³C NMR** (75 MHz, CDCl₃): δ 155.6, 152.7, 144.5, 139.1, 133.3, 132.7, 130.6 (2), 130.3, 129.9 (2), 129.3, 127.8 (2), 127.1, 126.9 (2), 125.9, 124.1, 122.9, 117.4, 21.6. **HRMS** (ESI): *m/z* [M+H]⁺ calc. for C₂₂H₁₇N₂O₂S₂, 405.0725 found 405.0734.

7-methoxy-2-phenyl-3-tosylbenzo[d]imidazo[2,1-b]thiazole **5c**

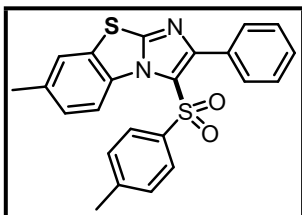
Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 68% yield. Purification by silica gel column chromatography (100% cyclohexane to 2:1 cyclohexanes/EtOAc) afforded 72.5 mg (65%) of the title compound **5c**.



¹H NMR (300 MHz, CDCl₃): δ 8.69 (d, *J* = 9.3 Hz, 1H), 7.67 - 7.61 (m, 2H), 7.57 (d, *J* = 8.4 Hz, 2H), 7.40 (dd, *J* = 5.1, 2.0 Hz, 3H), 7.17 (d, *J* = 2.6 Hz, 1H), 7.14 (d, *J* = 8.1 Hz, 2H), 7.03 (dd, *J* = 9.3, 2.6 Hz, 1H), 3.85 (s, 3H), 2.31 (s, 3H). **¹³C NMR** (75 MHz, CDCl₃): δ 157.7, 155.0, 152.0, 144.4, 139.2, 132.9, 131.4, 130.6 (2), 129.8 (2), 129.2, 127.7 (2), 127.4, 126.8 (2), 122.5, 118.1, 114.1, 108.1, 55.9, 21.6. **HRMS** (ESI): *m/z* [M+H]⁺ calc. for C₂₃H₁₉N₂O₃S₂, 435.0831 found 435.0831.

7-methyl-2-phenyl-3-tosylbenzo[d]imidazo[2,1-b]thiazole **5d**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 60% yield. Purification by silica gel column chromatography (100% cyclohexane to 2:1 cyclohexanes/EtOAc) afforded 59.1 mg (55%) of the title compound **5d**.

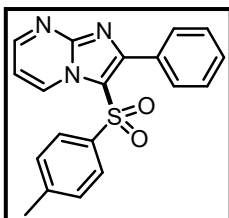


¹H NMR (300 MHz, CDCl₃): δ 8.66 (d, *J* = 8.7 Hz, 1H), 7.68 – 7.63 (m, 2H), 7.58 (d, *J* = 8.4 Hz, 2H), 7.49 (s, 1H), 7.44 – 7.38 (m, 3H), 7.29 (dd, *J* = 8.7, 1.6 Hz, 1H), 7.15 (d, *J* = 8.1 Hz, 2H), 2.46 (s, 3H), 2.32 (s, 3H). **¹³C NMR** (75 MHz, CDCl₃): δ 155.4, 152.5, 144.4, 139.2, 136.2, 132.9, 131.3, 130.6 (2), 130.1, 129.9 (2), 129.3, 128.1, 127.7 (2), 126.8 (2), 124.1, 122.6, 117.0, 21.6, 21.3. **HRMS** (ESI): *m/z* [M+H]⁺

calc. for C₂₃H₁₉N₂O₂S₂, 419.0882 found 419.0881.

2-phenyl-3-tosylimidazo[1,2-*a*]pyrimidine **5e**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 38% yield. Purification by silica gel column chromatography (98:2 Dichloromethane/EtOAc) afforded 31.4 mg (35%) of the title compound **5e**. Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (98:2 Dichloromethane/EtOAc) afforded 28.3 mg (39%) of the title compound **5e**.

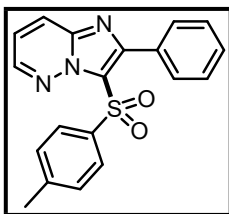


¹H NMR (300 MHz, CDCl₃): δ 9.48 (dd, *J* = 7.0, 2.0 Hz, 1H), 8.75 (dd, *J* = 4.2, 2.0 Hz, 1H), 7.83 - 7.78 (m, 2H), 7.45 (dd, *J* = 6.8, 1.5 Hz, 5H), 7.15 - 7.09 (m, 3H), 2.32 (s, 3H). **¹³C NMR** (75 MHz, CDCl₃): δ 154.2, 153.3, 149.0, 145.0, 138.8, 135.1, 132.0, 130.8 (2), 129.9, 129.9 (2), 128.0 (2), 126.6 (2), 116.9, 110.7, 21.7. **HRMS** (ESI): *m/z* [M+H]⁺ calc. for C₁₉H₁₆N₃O₂S, 350.0957 found 350.0953.

2-phenyl-3-tosylimidazo[1,2-*b*]pyridazine **5f**

Following General Procedure A on 0.257 mmol scale with 10 h of reaction time. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 55 % yield. Purification by silica gel column chromatography (98:2 Dichloromethane/EtOAc) afforded 44.9 mg (50%) of the title compound **5f**.

Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (98:2 Dichloromethane/EtOAc) afforded 10.8 mg (15%) of the title compound **5f**.

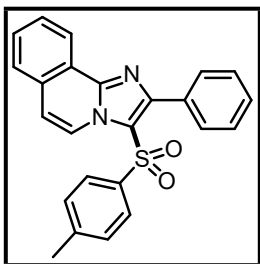


¹H NMR (300 MHz, CDCl₃): δ 8.57 (dd, *J* = 4.5, 1.6 Hz, 1H), 8.07 (dd, *J* = 9.0, 1.6 Hz, 3H), 7.89 (ddd, *J* = 6.0, 5.2, 3.4 Hz, 2H), 7.59 - 7.54 (m, 3H), 7.35 - 7.30 (m, 3H), 2.44 (s, 3H). **¹³C NMR** (75 MHz, CDCl₃): δ 150.9, 144.8, 143.9, 140.3, 138.0, 132.7, 130.7 (2), 129.6 (3), 128.5 (2), 128.0 (2), 125.9, 122.6, 120.3, 21.8. **HRMS** (ESI): *m/z* [M+H]⁺ calc. for C₁₉H₁₆N₃O₂S, 350.0957 found 350.0952.

2-phenyl-3-tosylimidazo[2,1-*a*]isoquinoline **5g**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 26% yield. Purification by silica gel column chromatography

(100% cyclohexane to 2:1 cyclohexanes/EtOAc) afforded 20.5 mg (20%) of the title compound **5g**.

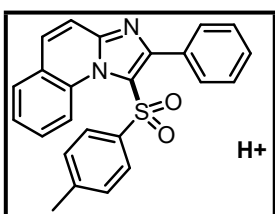


¹H NMR (300 MHz, CDCl₃): δ 8.90 (d, *J* = 7.5 Hz, 2H), 8.78 - 8.69 (m, 2H), 7.81 - 7.74 (m, 6H), 7.71 - 7.64 (m, 4H), 7.55 (d, *J* = 8.3 Hz, 4H), 7.51 - 7.44 (m, 6H), 7.27 (d, *J* = 8.5 Hz, 3H), 7.14 (d, *J* = 8.2 Hz, 4H), 2.31 (s, 6H). **¹³C NMR** (75 MHz, CDCl₃): δ 151.3, 144.9, 144.5, 139.3, 132.9, 130.8 (2), 130.5, 130.1, 129.8 (2), 129.3, 128.8, 127.9 (2), 127.0, 126.6 (2), 124.6, 123.1, 122.9, 119.9, 115.0, 21.6. **HRMS** (ESI): *m/z* [M+H]⁺ calc. for C₂₄H₁₉N₂O₂S, 399.1161 found 399.1162

2-phenyl-1-tosylimidazo[1,2-*a*]quinoline **5h**

Following General Procedure A on 0.257 mmol scale. Yield determined by analysis of ¹H-NMR spectra of crude reaction product: 76% yield. Purification by silica gel column chromatography (98:2 Dichloromethane/EtOAc) afforded 71.6 mg (70%) of the title compound **5h**.

Following General Procedure B on 0.207 mmol scale. Purification by silica gel column chromatography (98:2 Dichloromethane/EtOAc) afforded 51.8 mg (63%) of the title compound **5h**.



¹H NMR (300 MHz, CDCl₃): δ 8.94 (d, *J* = 8.7 Hz, 1H), 7.82 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.77 (d, *J* = 9.2 Hz, 1H), 7.68 (ddd, *J* = 8.2, 6.9, 1.3 Hz, 1H), 7.62 (d, *J* = 9.3 Hz, 1H), 7.59 - 7.54 (m, 2H), 7.51 (dd, *J* = 11.1, 4.0 Hz, 1H), 7.42 (d, *J* = 8.4 Hz, 2H), 7.36 - 7.27 (m, 3H), 6.96 (d, *J* = 8.0 Hz, 2H), 2.25 (s, 3H). **¹³C NMR** (75 MHz, CDCl₃): δ 155.0, 146.8, 144.2, 138.6, 134.2, 133.7, 131.4, 130.6 (2), 129.2 (2), 129.1 (2), 128.9, 127.8 (2), 127.7 (2), 125.9, 124.8, 122.4, 121.4, 116.6, 21.6. **HRMS** (ESI): *m/z* [M+H]⁺ calc. for C₂₄H₁₉N₂O₂S, 399.1161 found 399.1160

IV. Kinetics studies of 2-phenyl-3-tosylimidazo[1,2-*a*]pyridine **3aa**

Influence of the reactor volume :

Table 1: Influence of the thickness on the reaction efficiency at 15 min of residence time IKA ElectraSyn undivided Flow cell (x mL) with graphite plate anode (20 × 60 mm) and Pt plate cathode (20 × 60 mm), EPDM and Teflon gasket.

gasket thickness (mm)	Flow rate (μL.min ⁻¹)	Volume of the cell (mL)	Concentration 3aa (mol/L)
0,5	40	0.6	0,023
1	80	1.2	0,014
1,5	120	1.8	0,007
2	160	2.4	0,005
2,5	200	3	0,004
3	240	3.6	0,003

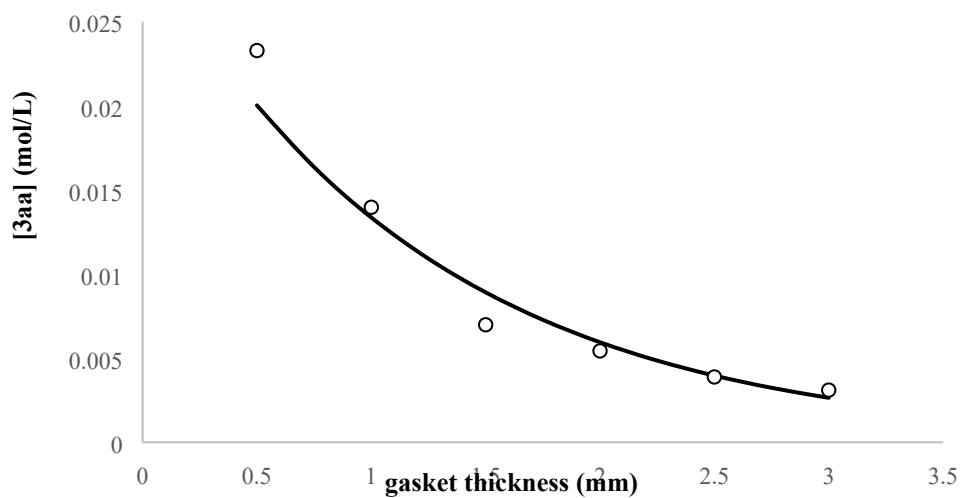


Figure 3: plot of [3aa] (mol.L⁻¹) vs gasket thickness (mm).

Scale-up studies

Table 2: Influence of the Surface to Volume ratio on the reaction efficiency at 15 min of residence time IKA ElectraSyn undivided Flow cell (x mL) with graphite plate anode (20 × 60 mm) and Pt plate cathode (20 × 60 mm), EPDM and Teflon gasket.

Yield 3aa (%)	Flow rate (μL.min ⁻¹)	Volume of the cell (mL)	Surface/Volume (cm ⁻¹)
92	20	0.3	40
90	40	0.6	20
54	80	1.2	10
27	120	1.8	6,67
21	160	2.4	5
15	200	3	4
12	240	3.6	3,33

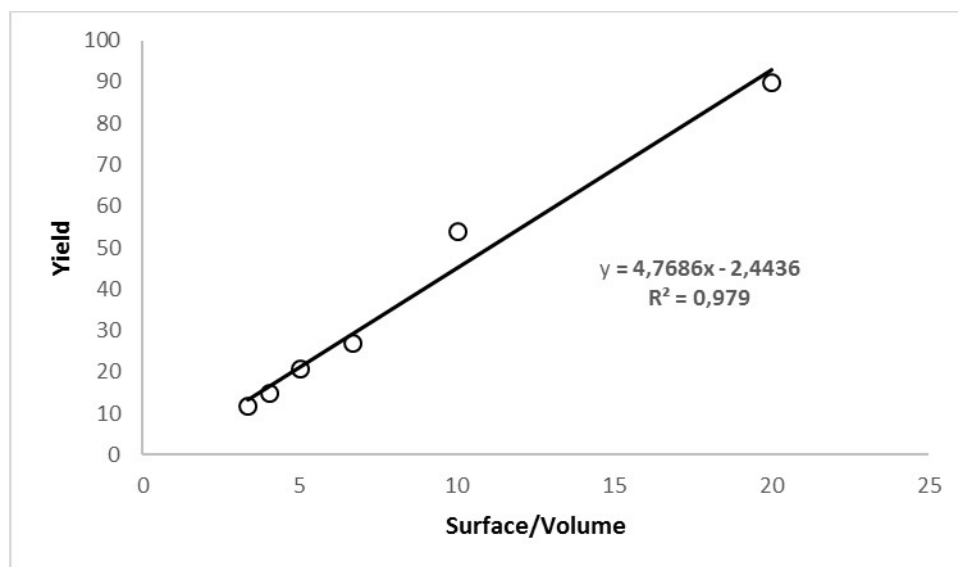


Figure 4 : Plot of yield of **3aa** vs surface/volume.

Kinetics studies:

Residence time (min)	Flow rate ($\mu\text{L}.\text{min}^{-1}$)	concentration (mol/L)
0	0	0
1	600	0,0016
2	300	0,0036
4	150	0,0072
6	100	0,0119
8	75	0,0153
10	60	0,0186
12	50	0,0204
15	40	0,0233
17	35	0,0091

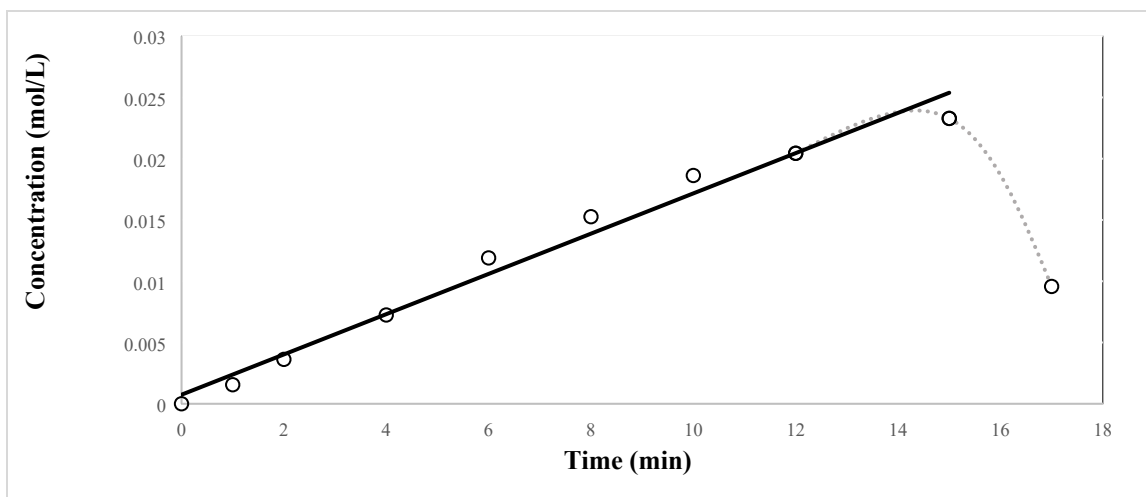
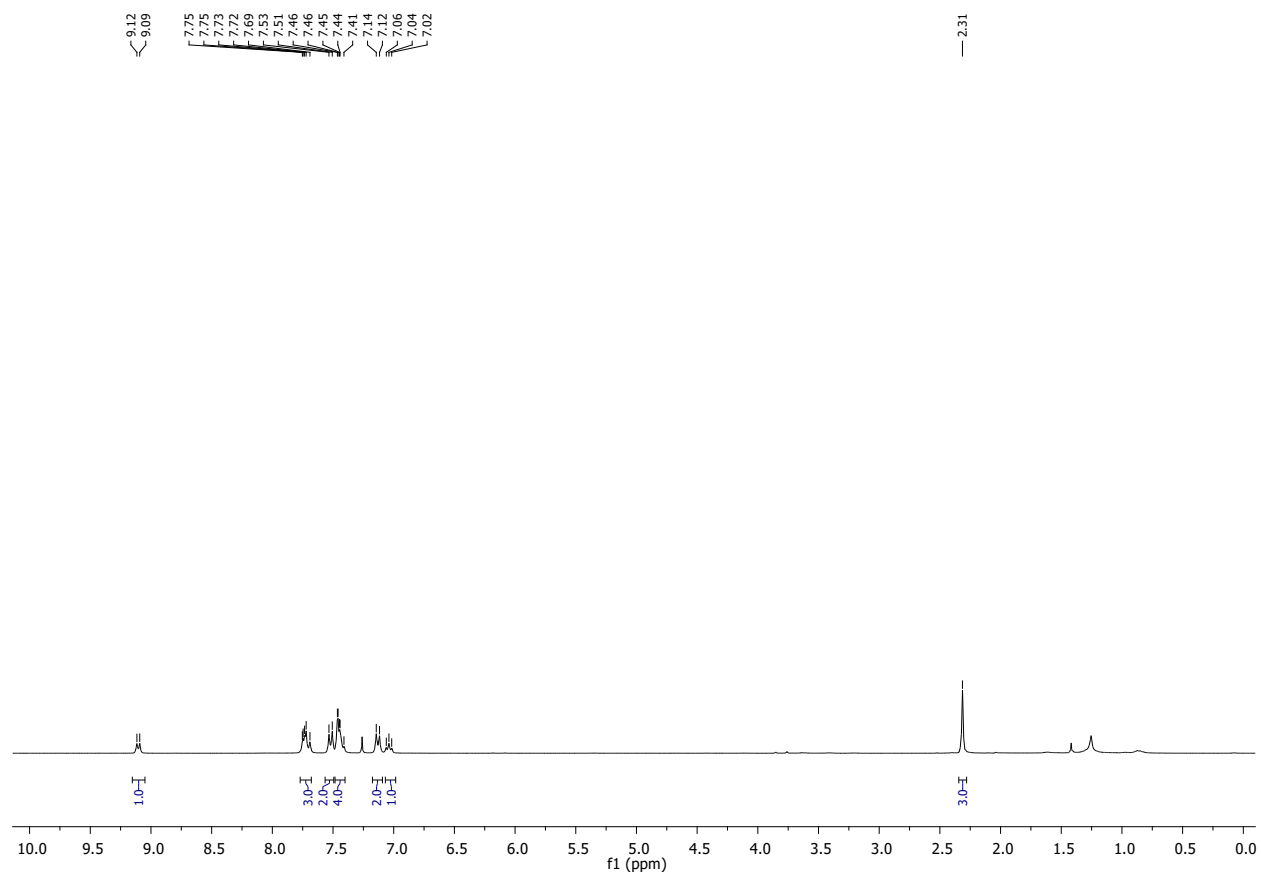


Figure 5 : Plot of [3aa] (mol.L⁻¹) vs time (min).

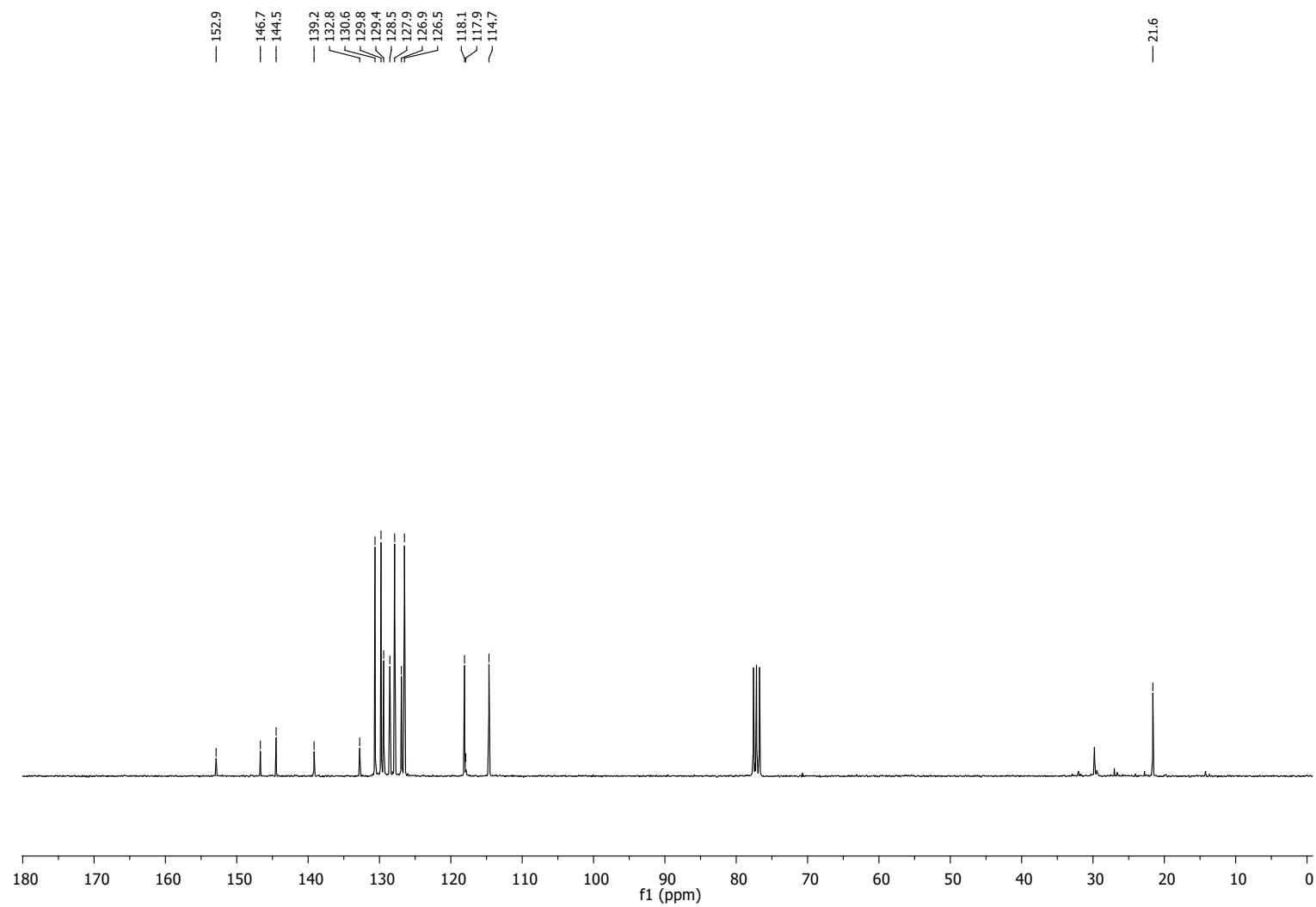
V. Copies of NMR spectra

2-phenyl-3-tosylimidazo[1,2-*a*]pyridine **3aa**

¹H NMR (CDCl₃, 300 MHz)



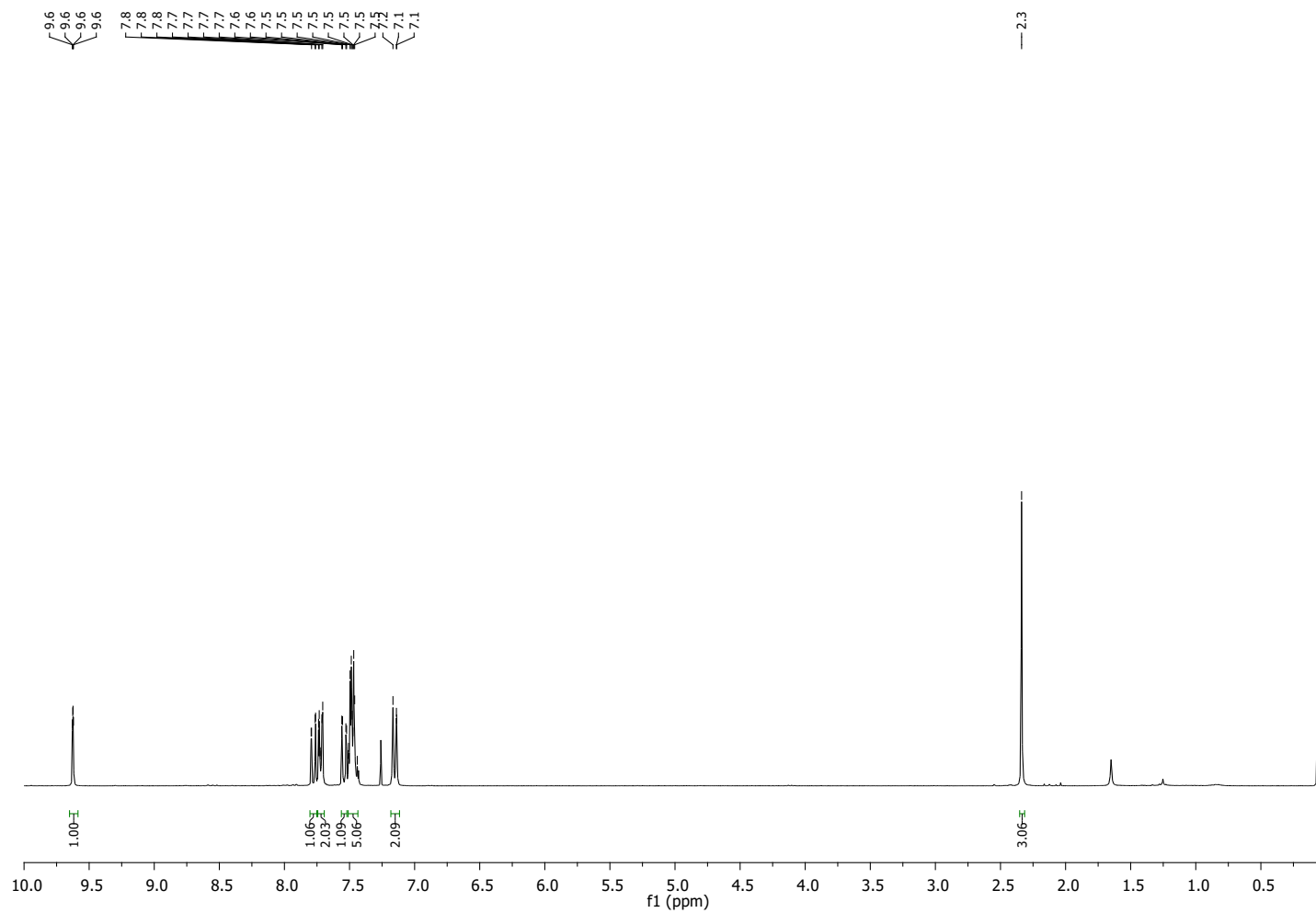
¹³C NMR (CDCl₃, 75 MHz)

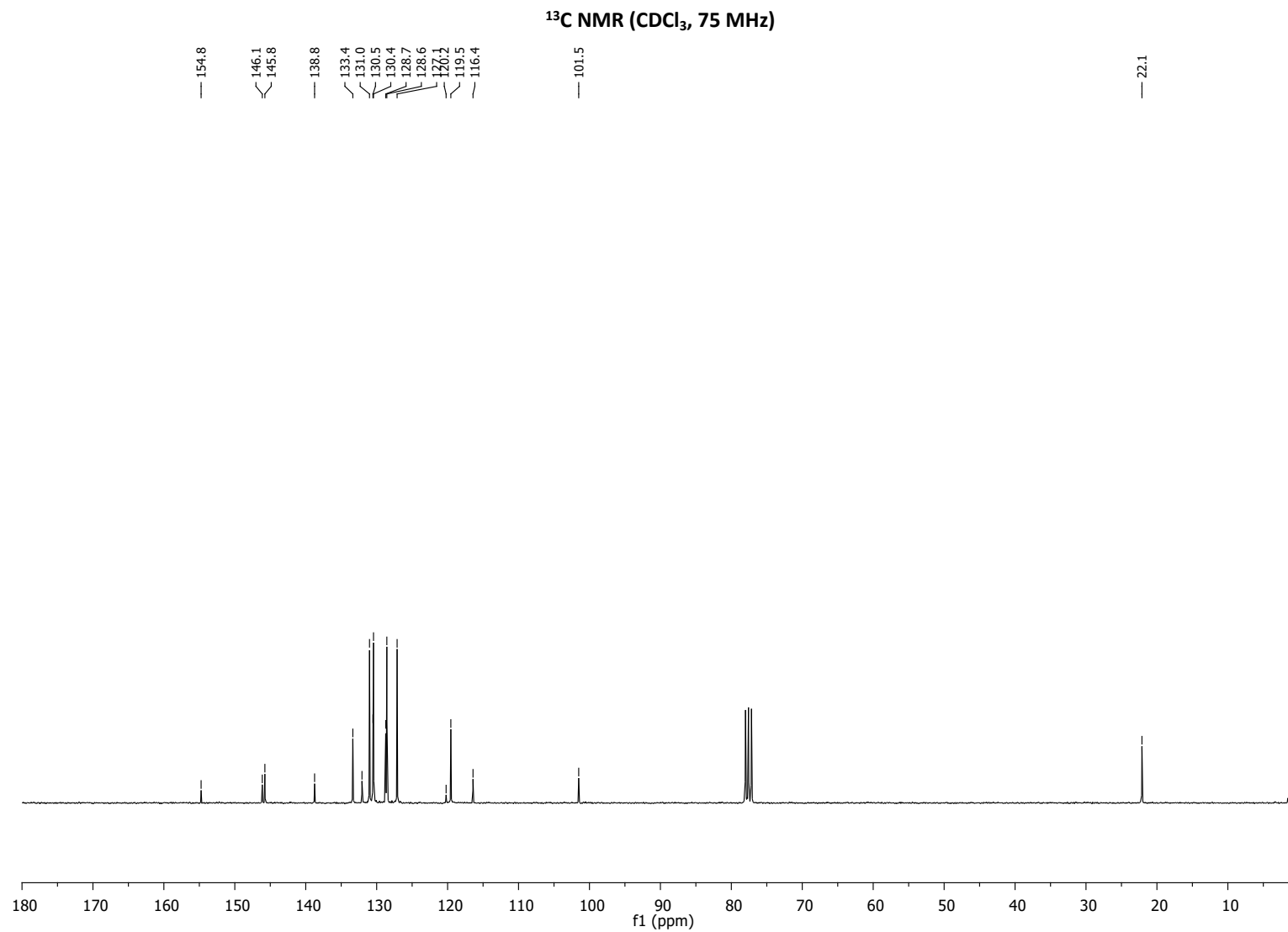


SI23

2-phenyl-3-tosylimidazo[1,2-*a*]pyridine-6-carbonitrile **3ba**

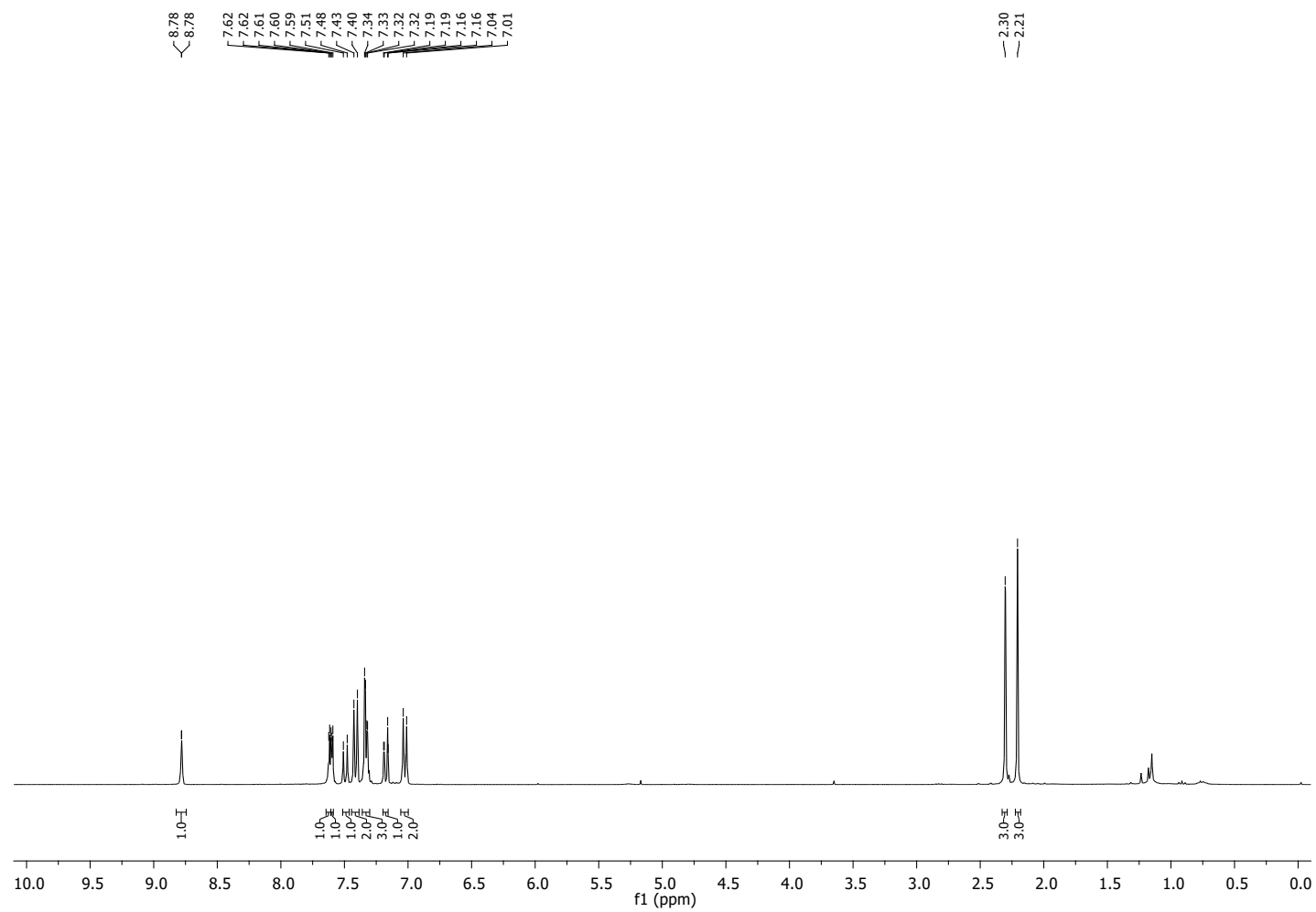
^1H NMR (CDCl_3 , 300 MHz)



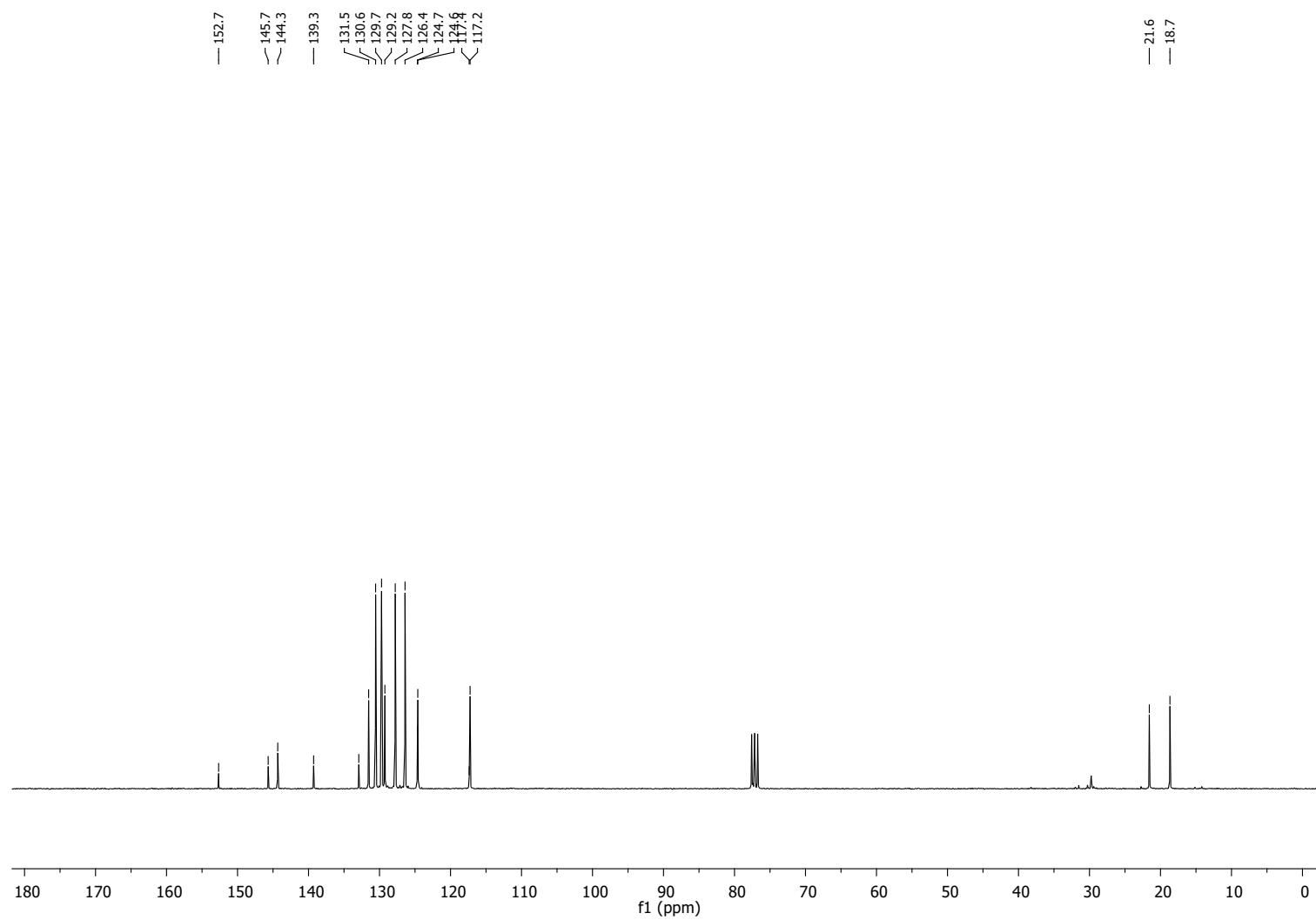


6-methyl-2-phenyl-3-tosylimidazo[1,2-a]pyridine **3ca**

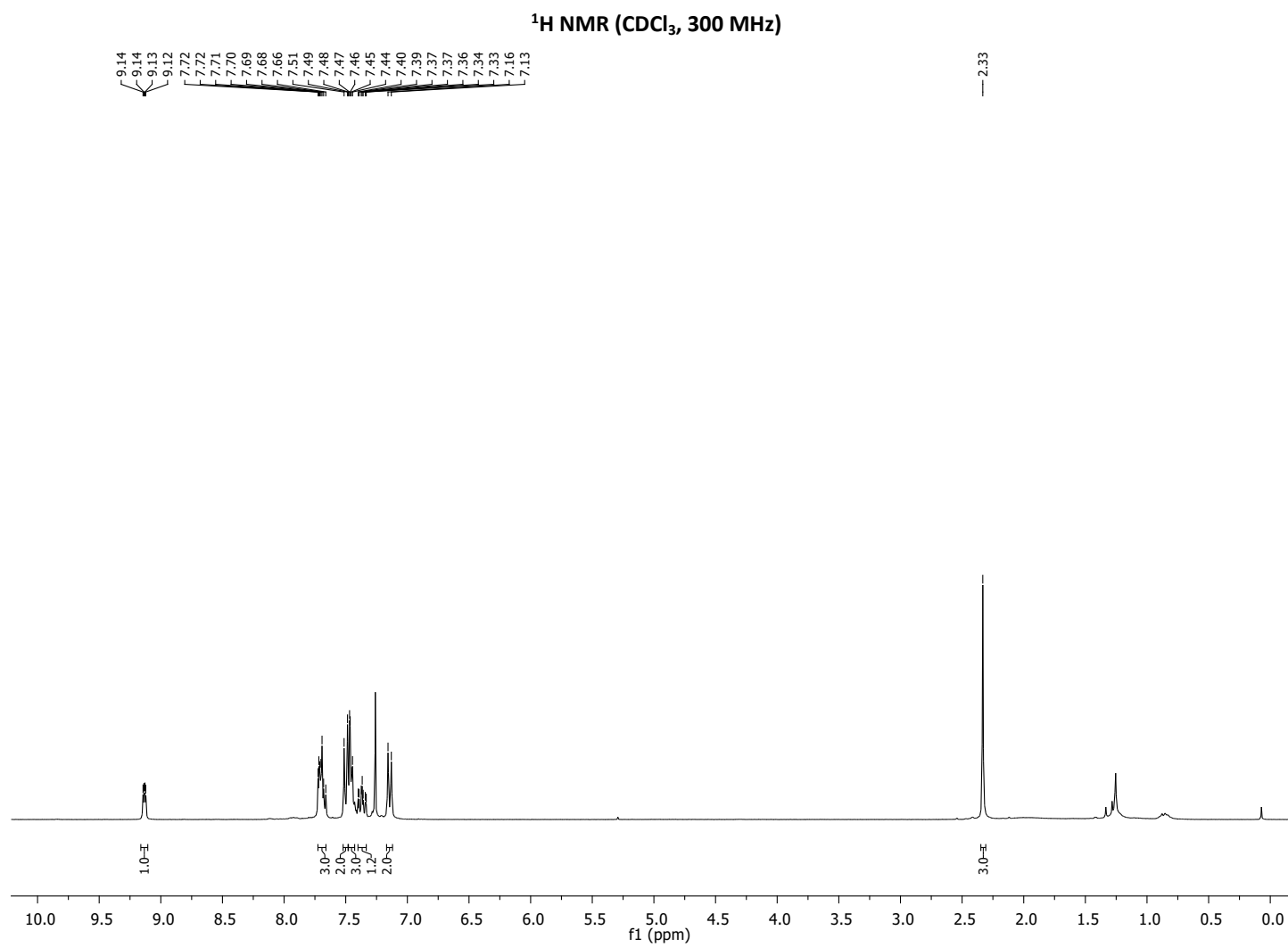
¹H NMR (CDCl₃, 300 MHz)



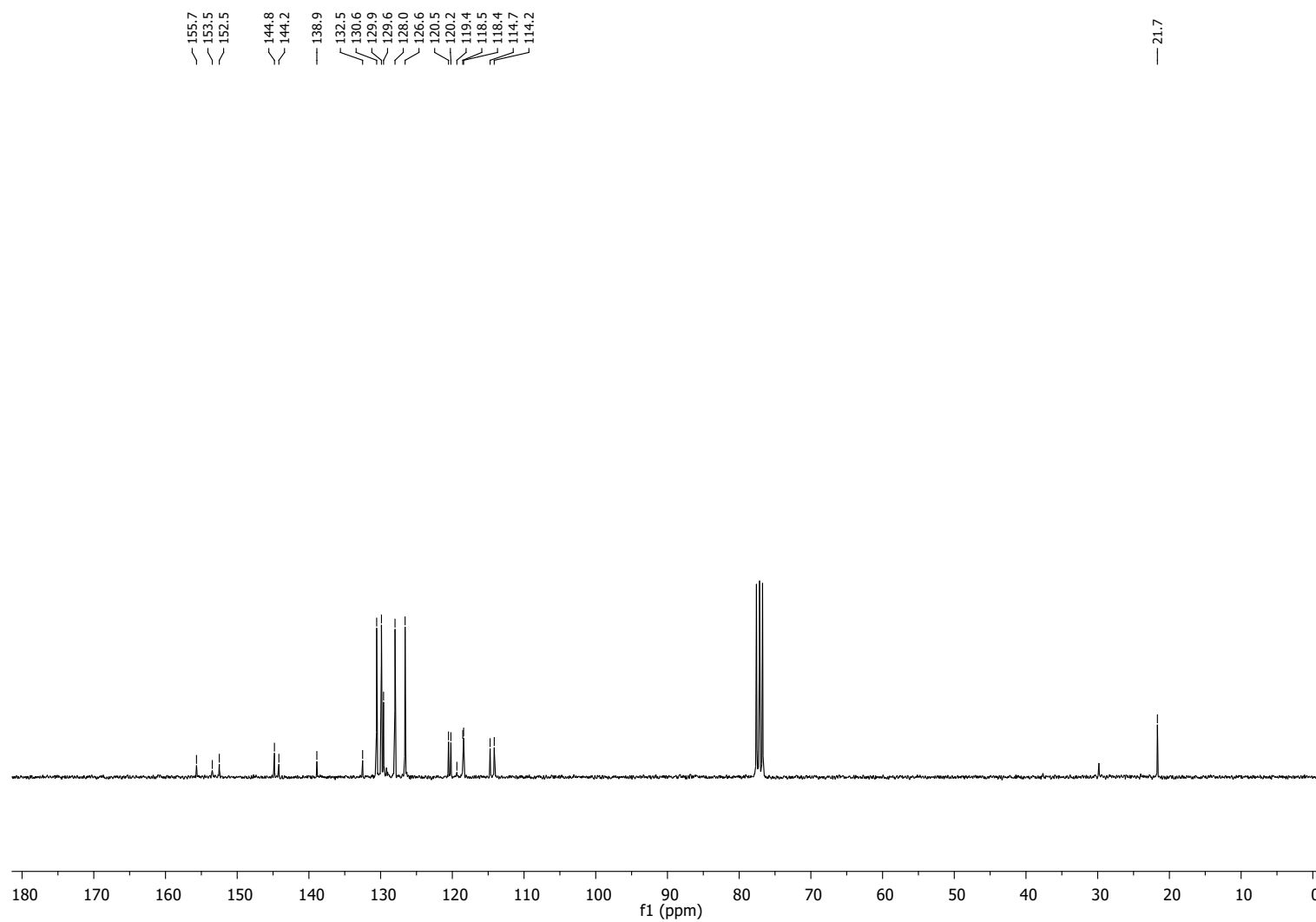
^{13}C NMR (CDCl₃, 75 MHz)



6-fluoro-2-phenyl-3-tosylimidazo[1,2-a]pyridine **3da**



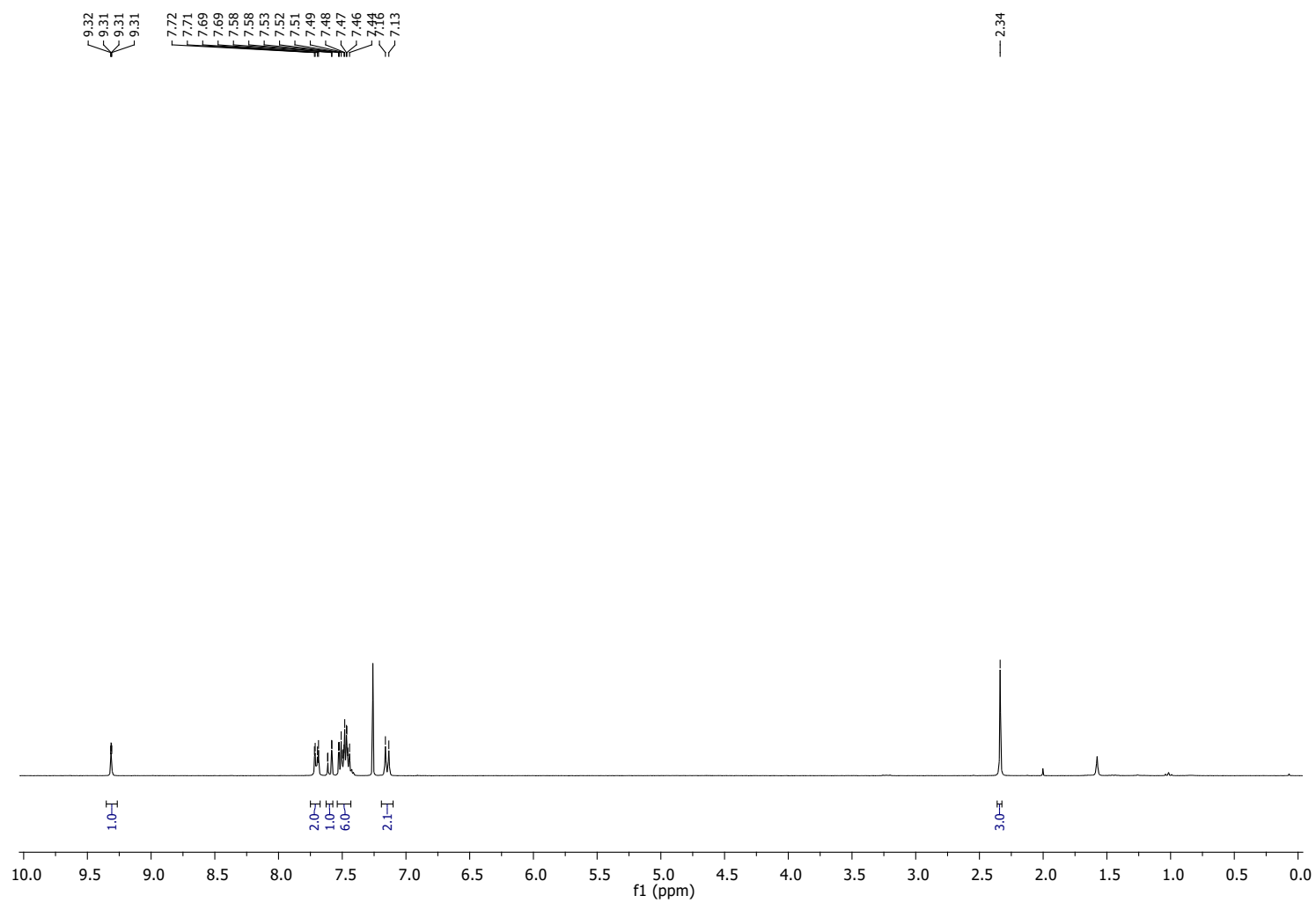
^{13}C NMR (CDCl₃, 75 MHz)



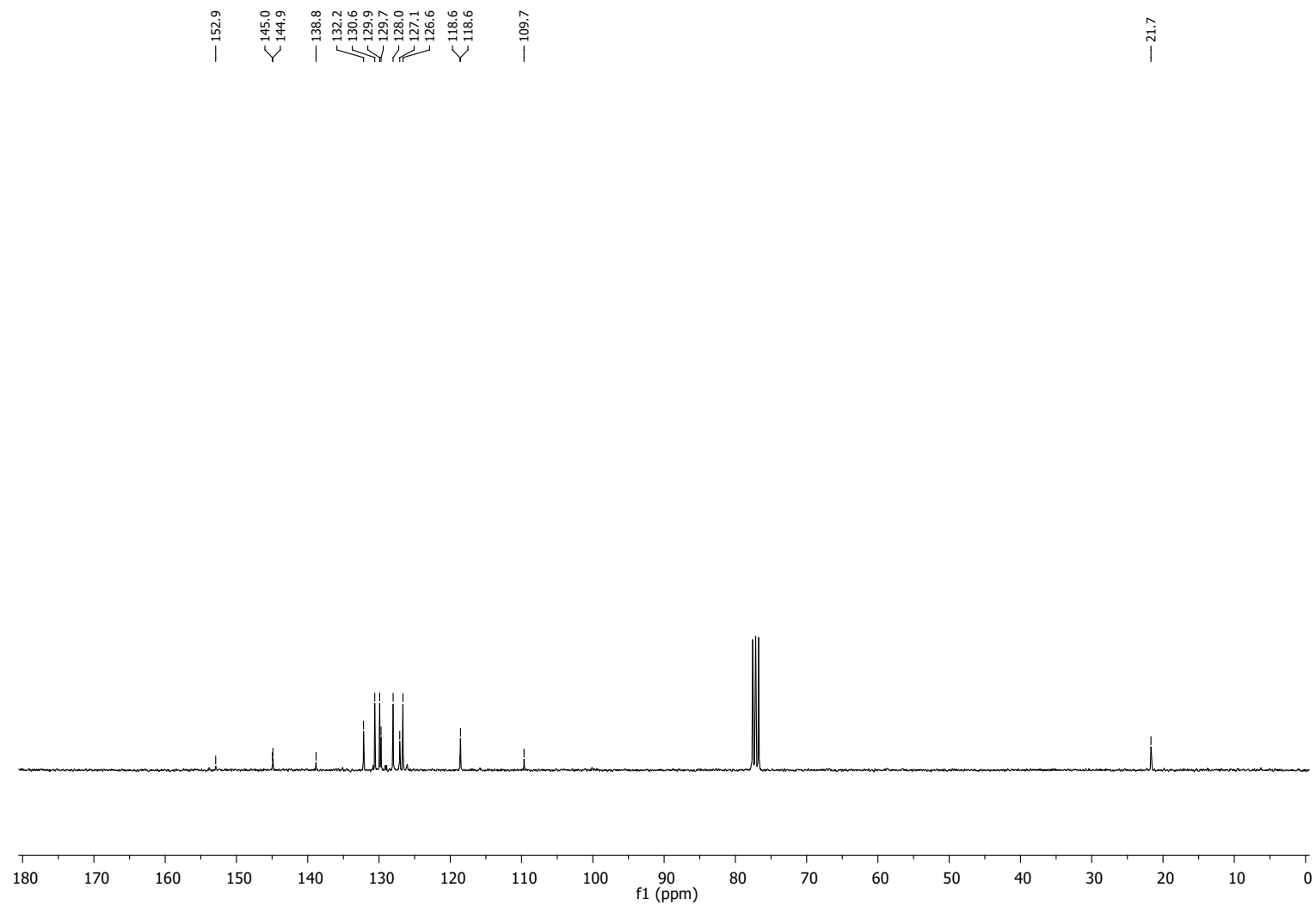
SI30

6-bromo-2-phenyl-3-tosylimidazo[1,2-*a*]pyridine **3ea**

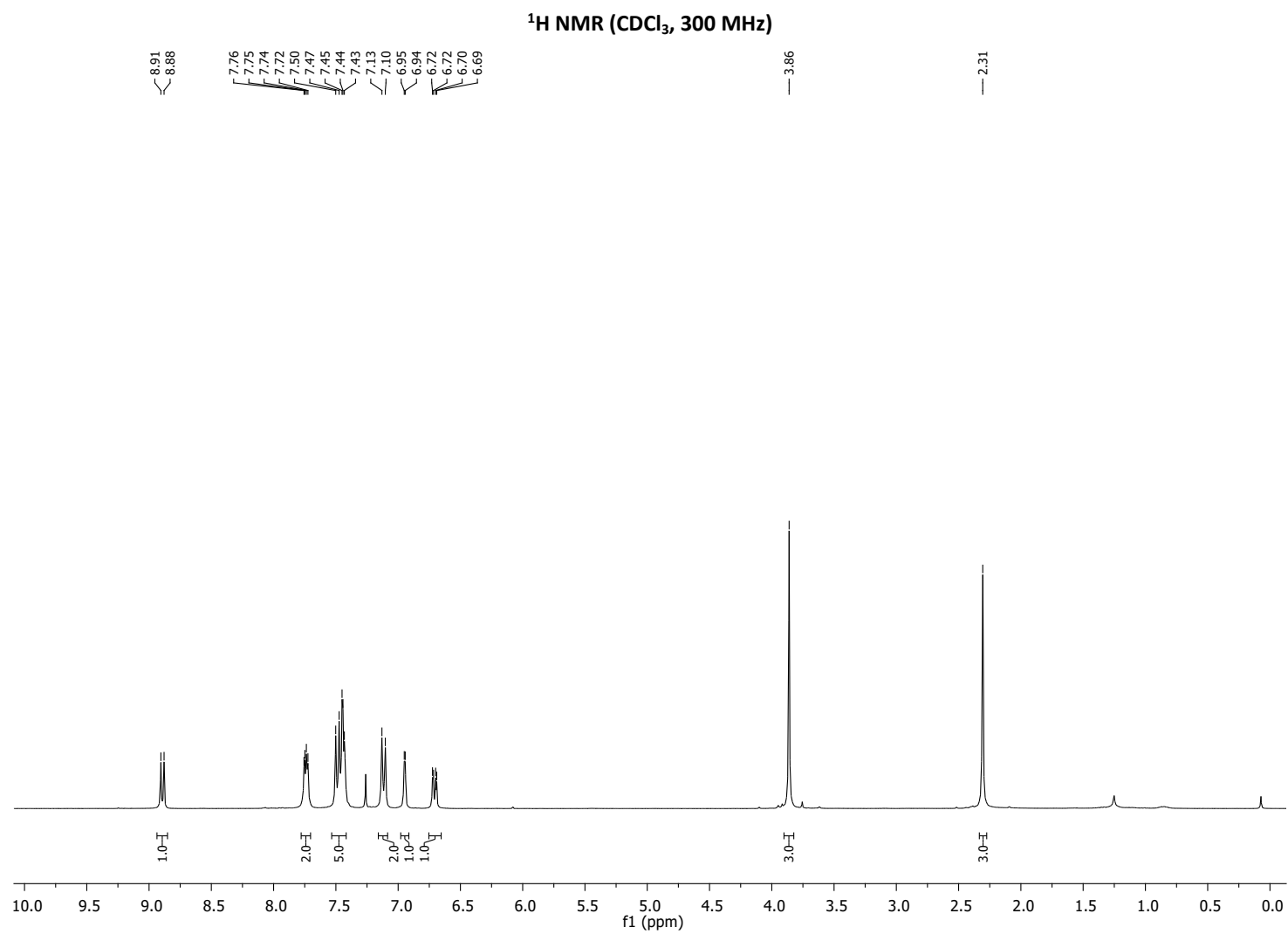
^1H NMR (CDCl_3 , 300 MHz)



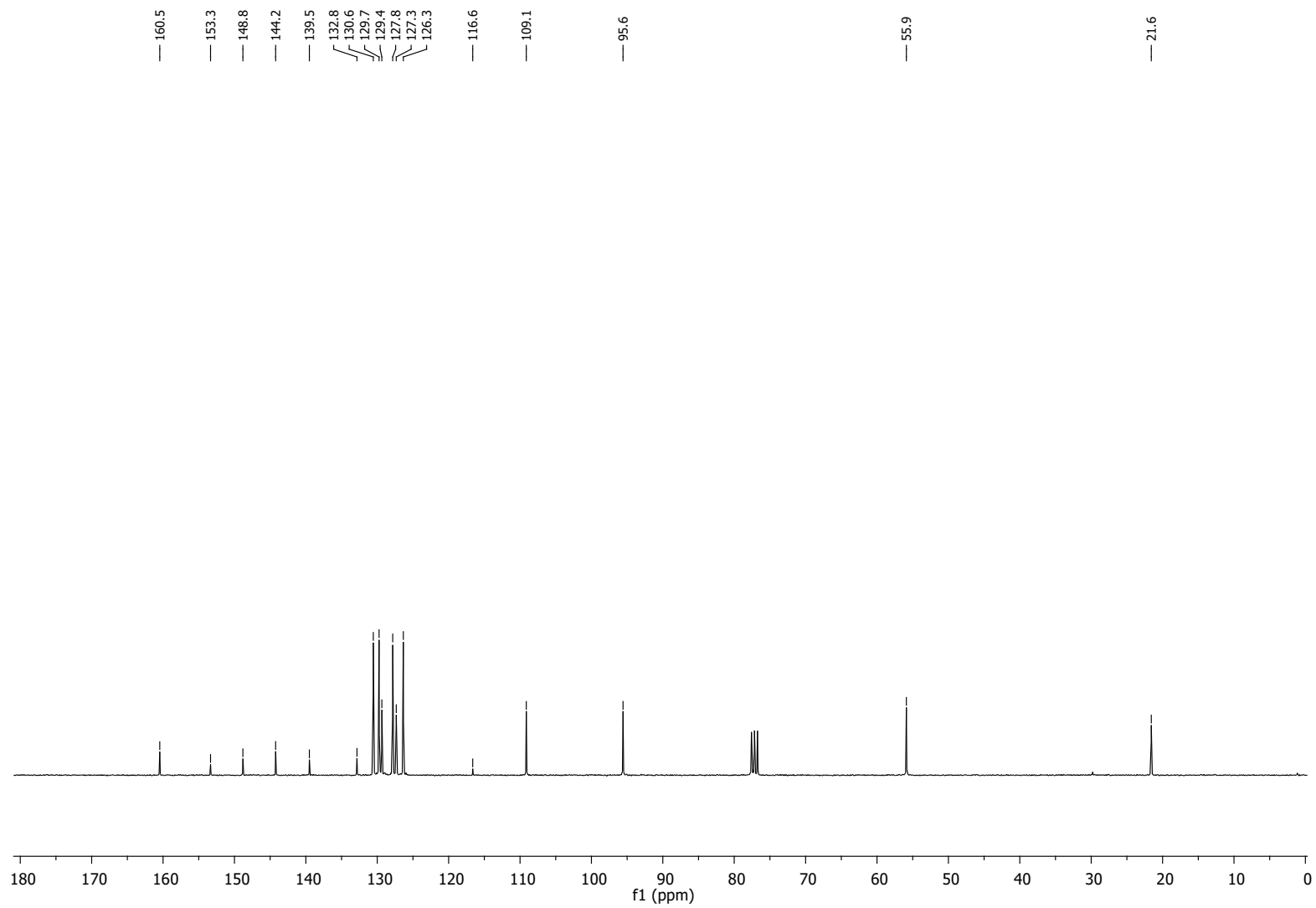
^{13}C NMR (CDCl₃, 75 MHz)



7-methoxy-2-phenyl-3-tosylimidazo[1,2-a]pyridine **3fa**

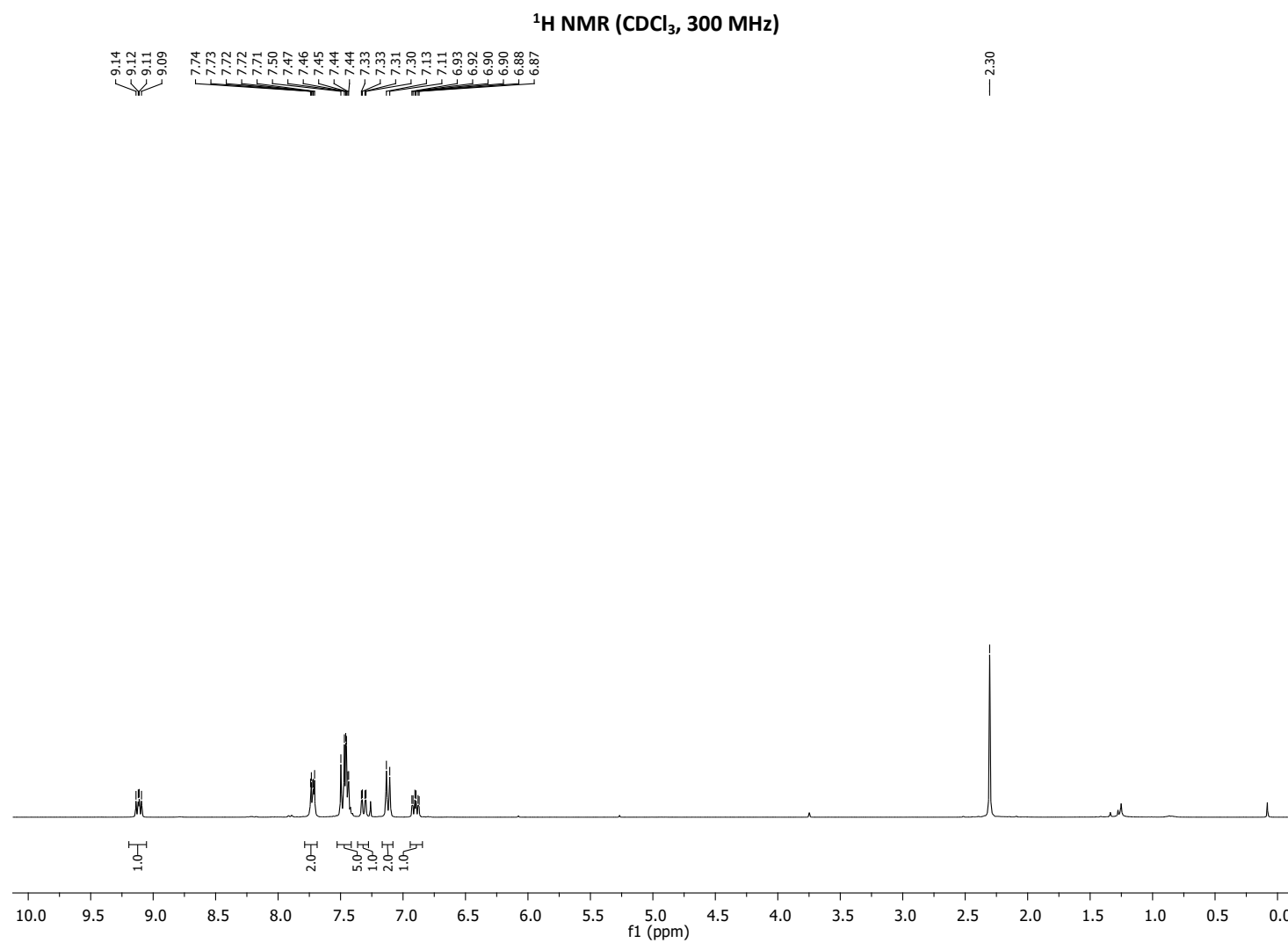


^{13}C NMR (CDCl₃, 75 MHz)

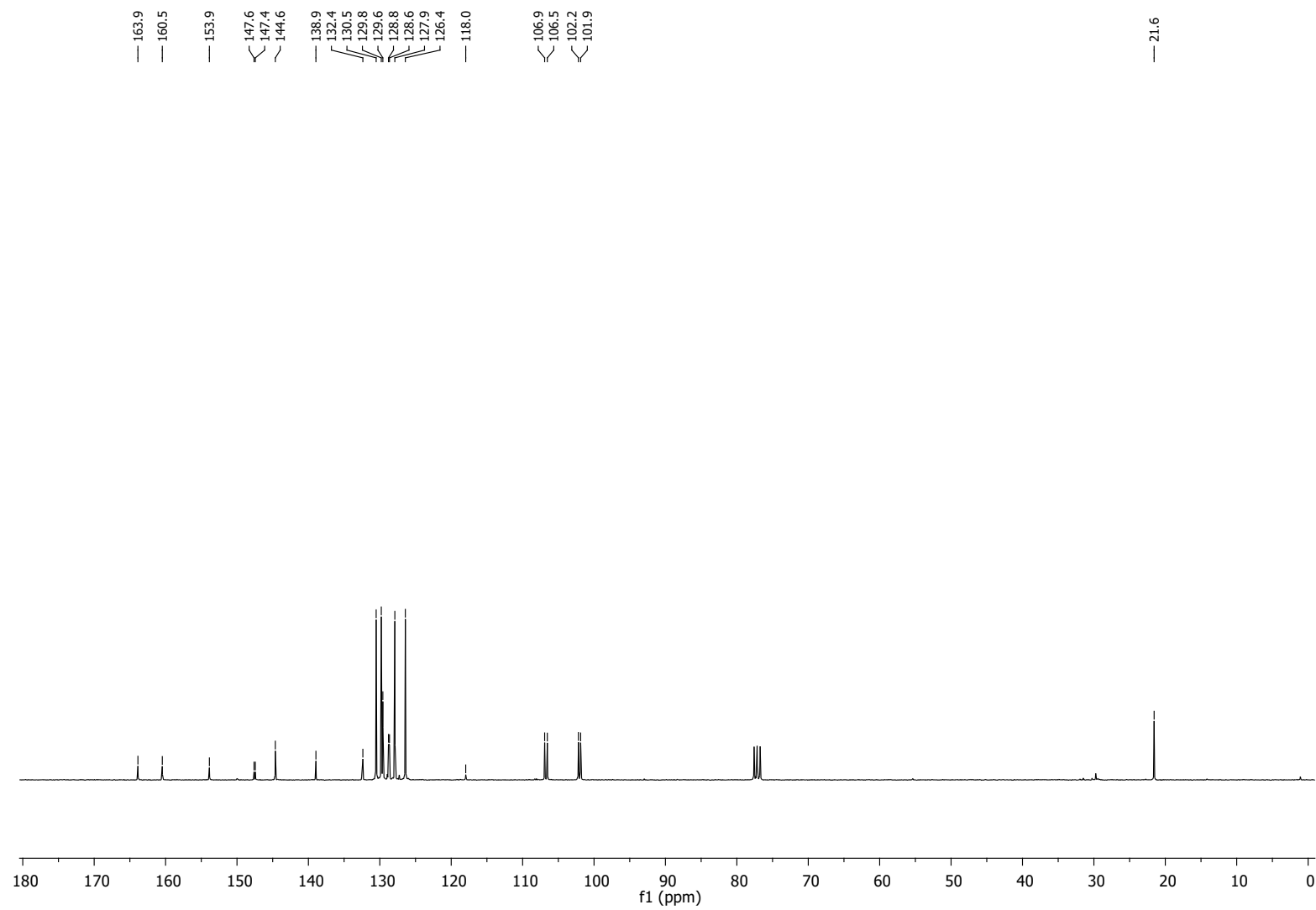


SI34

7-fluoro-2-phenyl-3-tosylimidazo[1,2-a]pyridine **3ga**

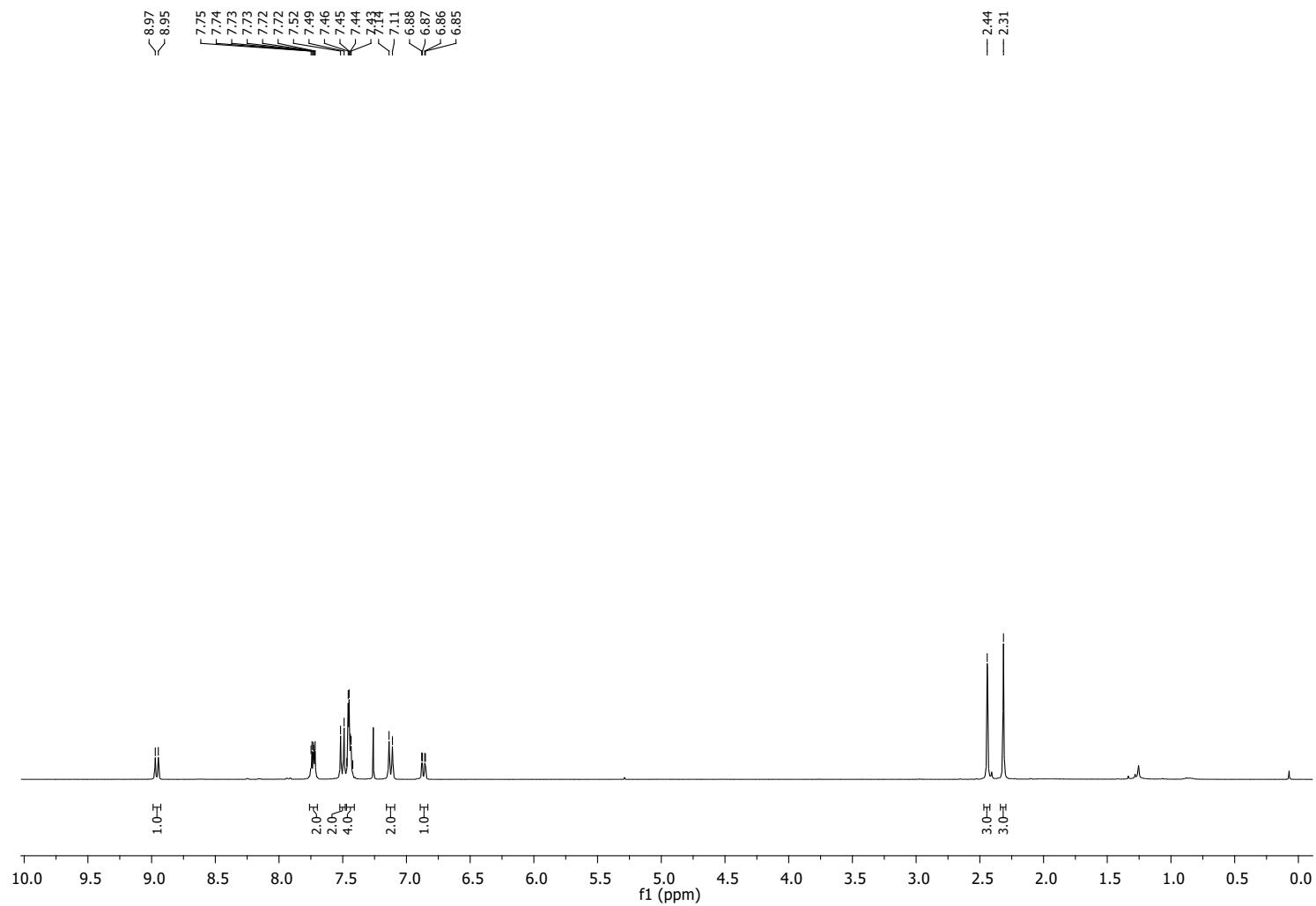


^{13}C NMR (CDCl₃, 75 MHz)

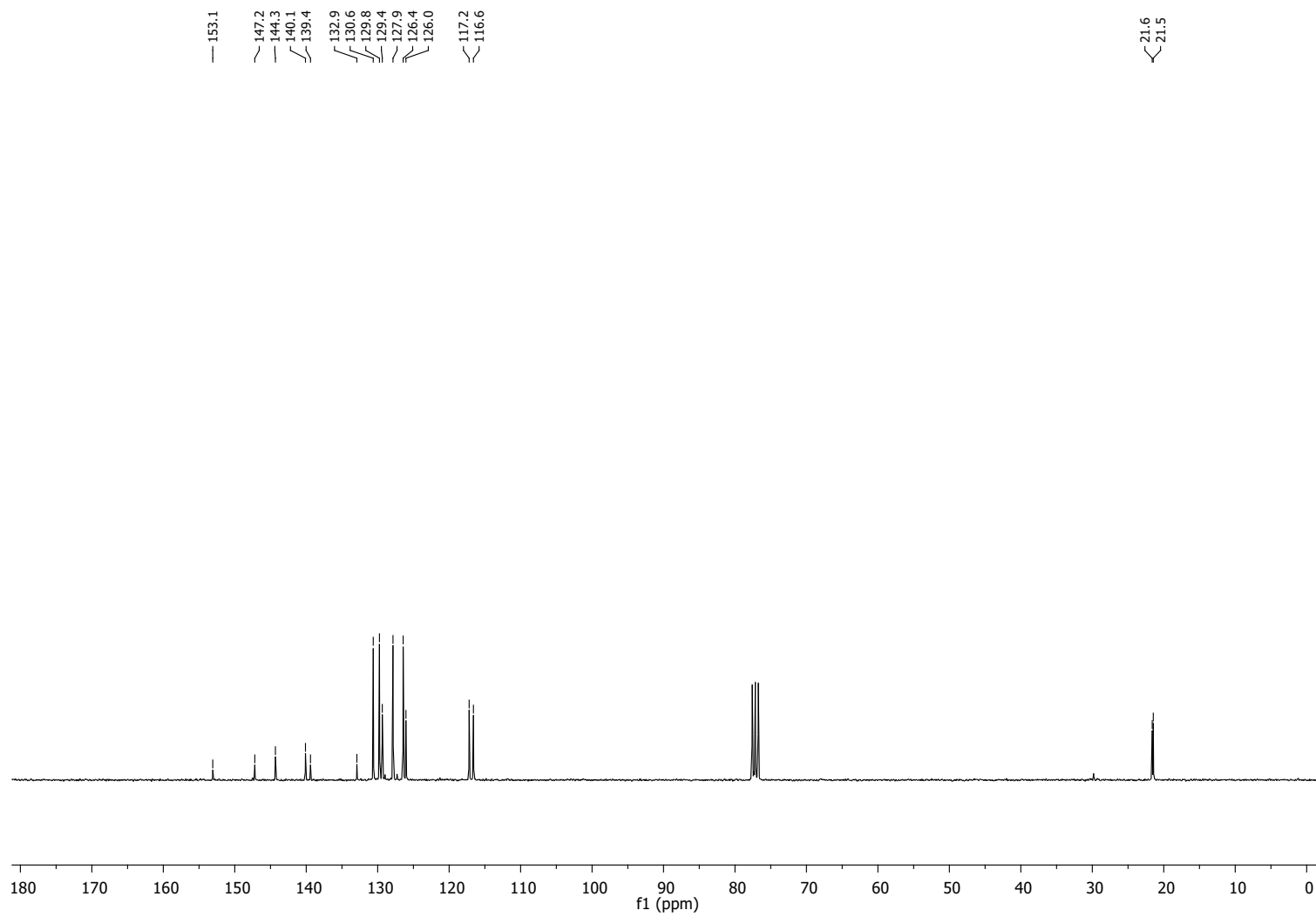


7-methyl-2-phenyl-3-tosylimidazo[1,2-a]pyridine **3ha**

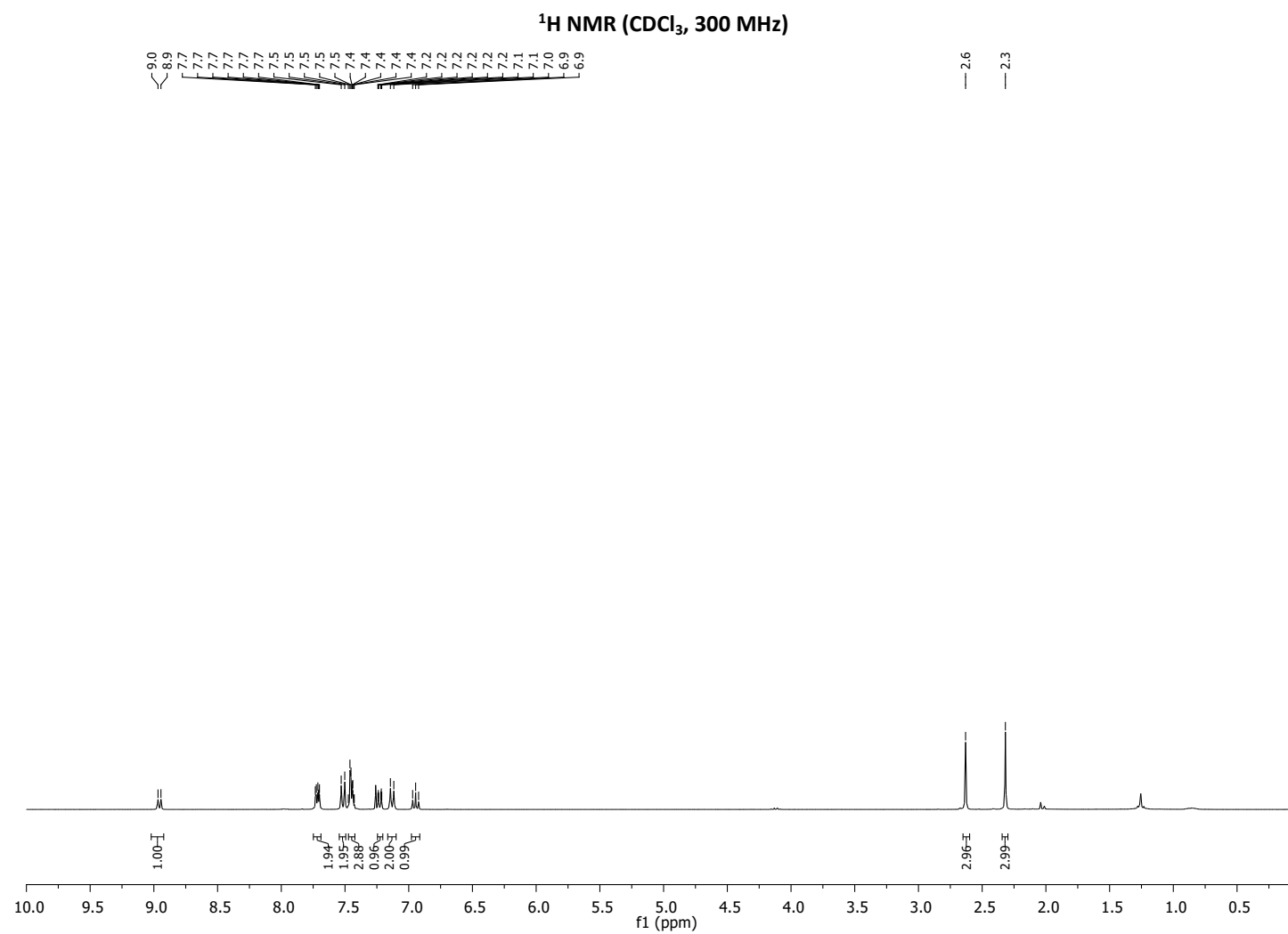
¹H NMR (CDCl₃, 300 MHz)



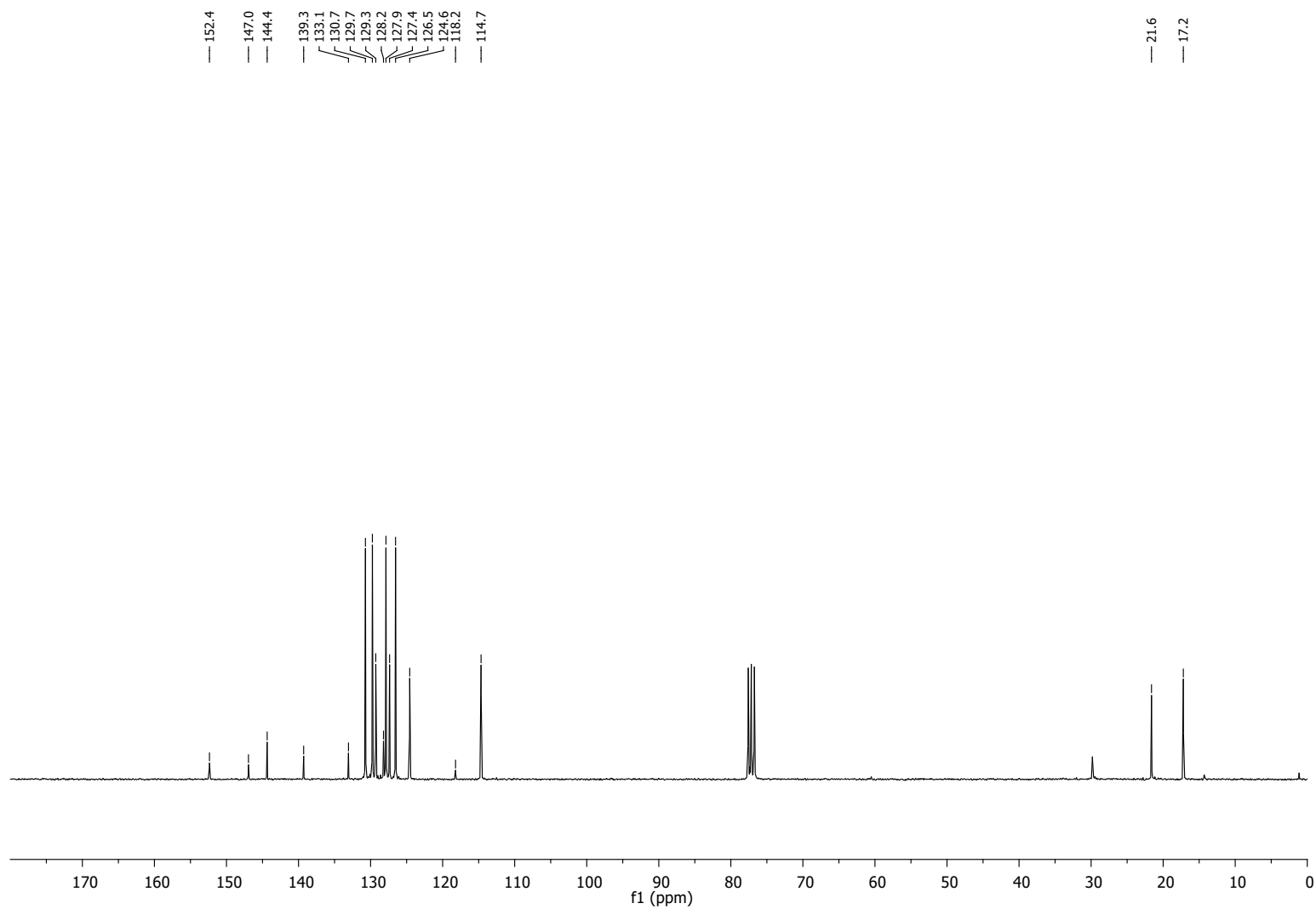
^{13}C NMR (CDCl₃, 75 MHz)



8-methyl-2-phenyl-3-tosylimidazo[1,2-a]pyridine **3ia**

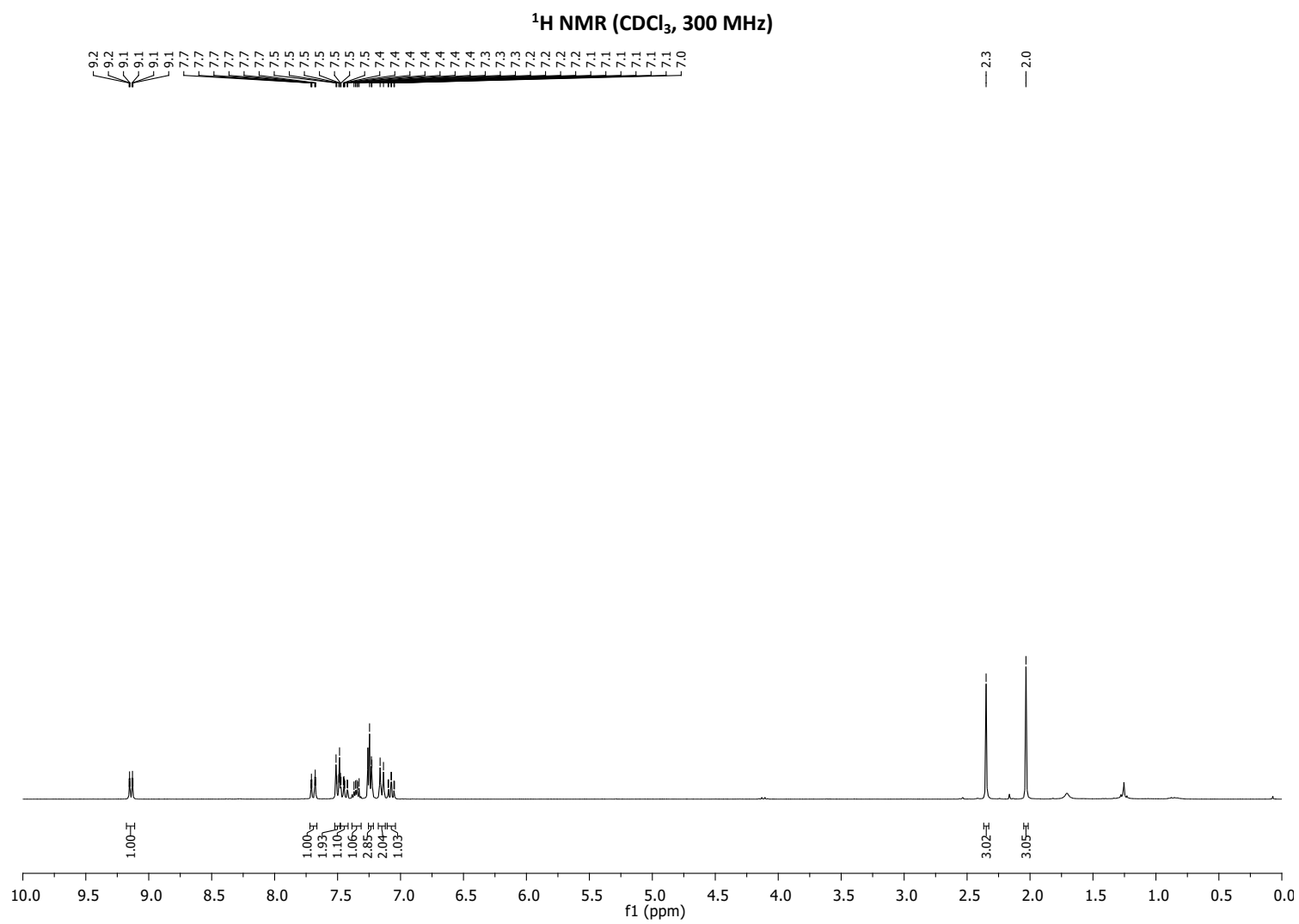


^{13}C NMR (CDCl₃, 75 MHz)

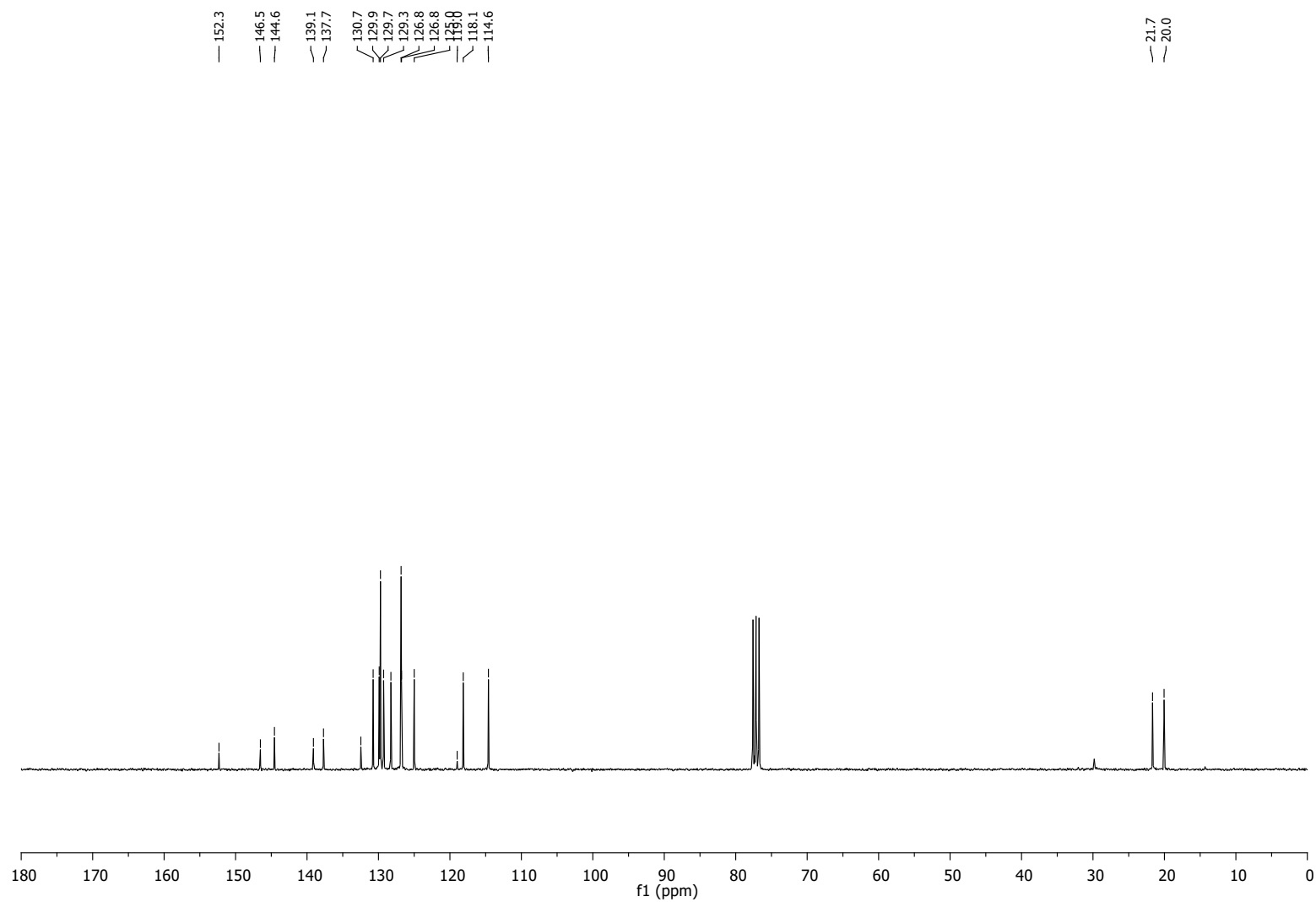


SI40

2-(*o*-tolyl)-3-tosylimidazo[1,2-*a*]pyridine **3ka**

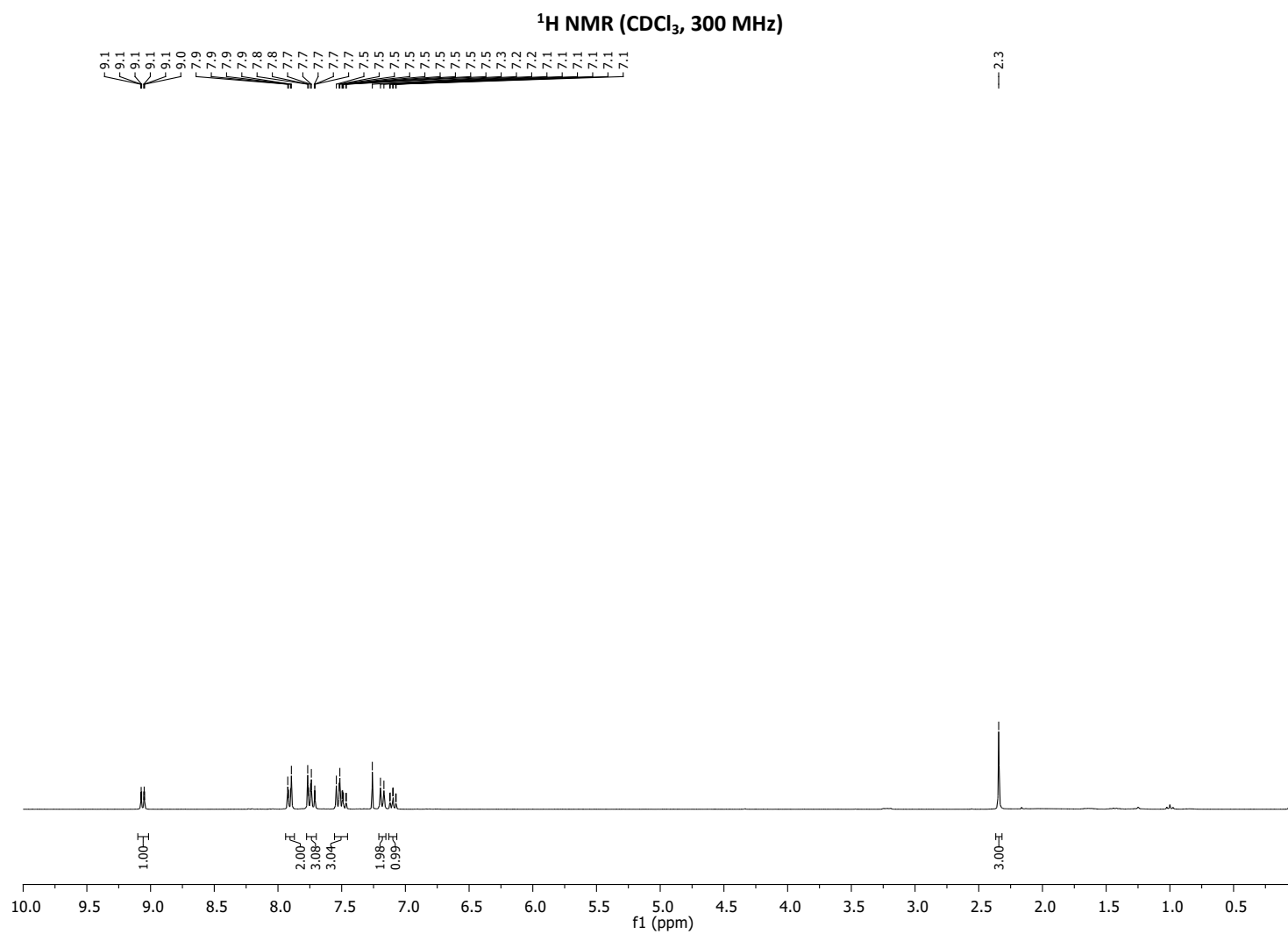


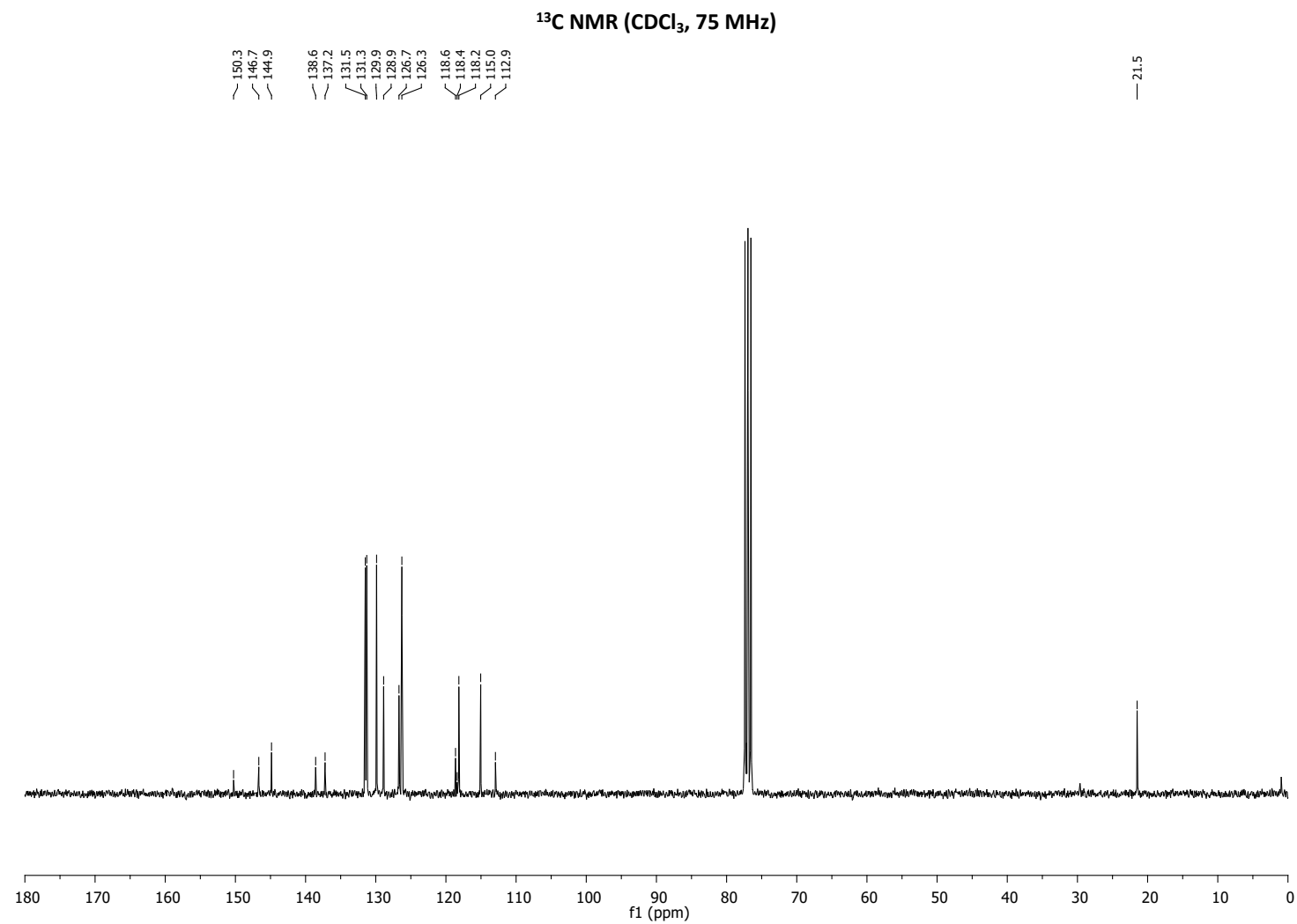
^{13}C NMR (CDCl₃, 75 MHz)



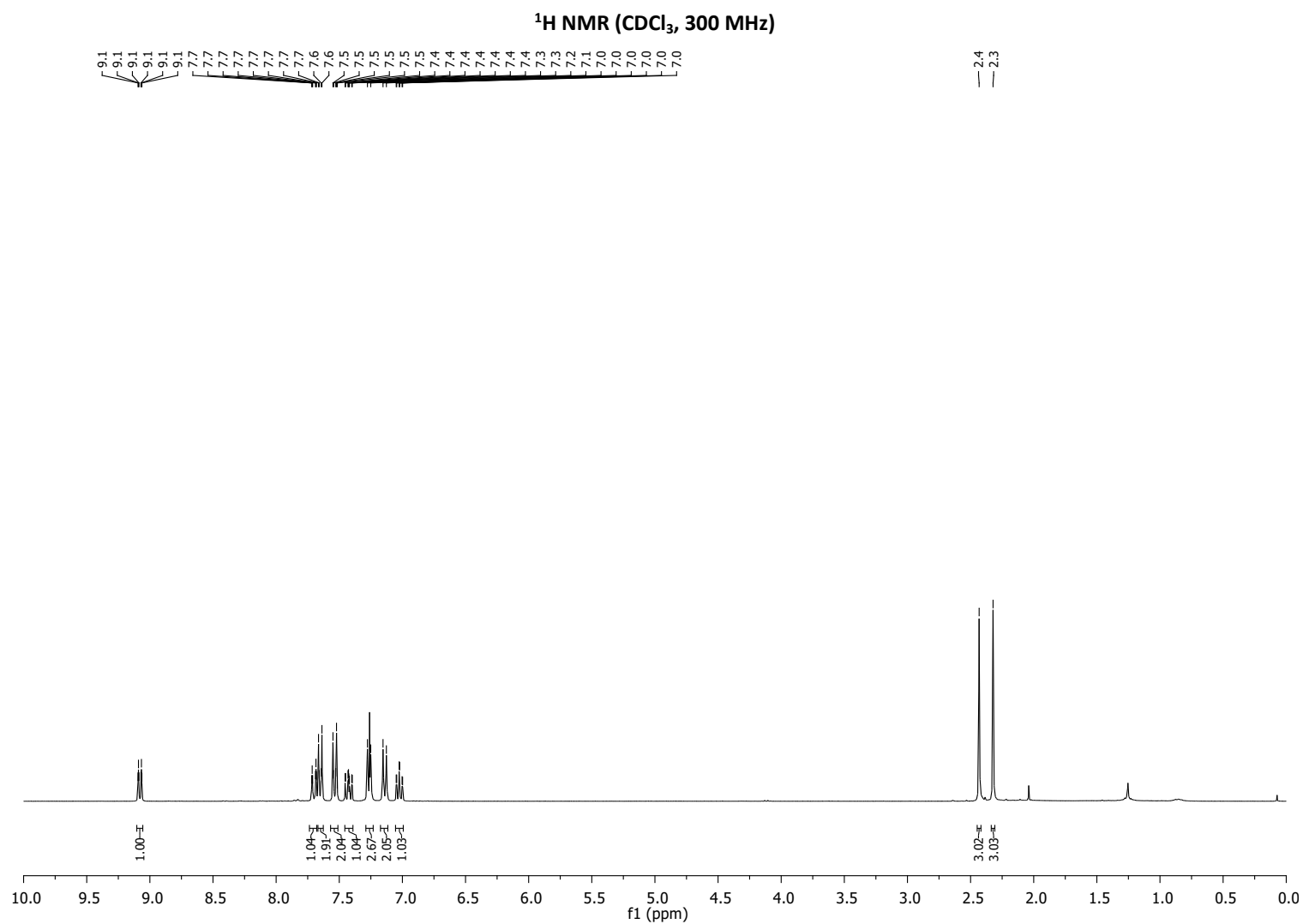
SI42

4-(3-tosylimidazo[1,2-*a*]pyridin-2-yl)benzonitrile **3la**

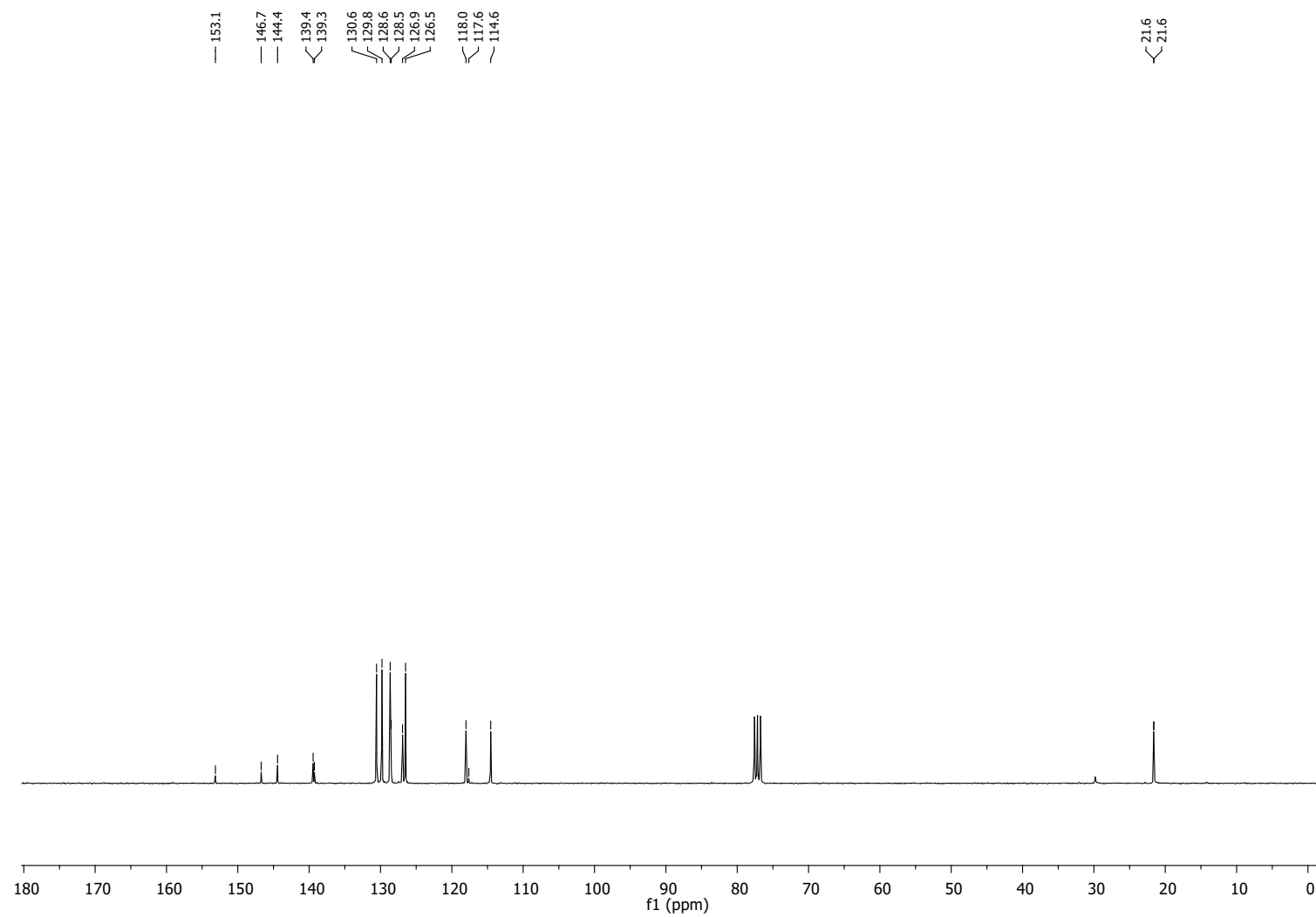




2-(p-tolyl)-3-tosylimidazo[1,2-a]pyridine **3ma**

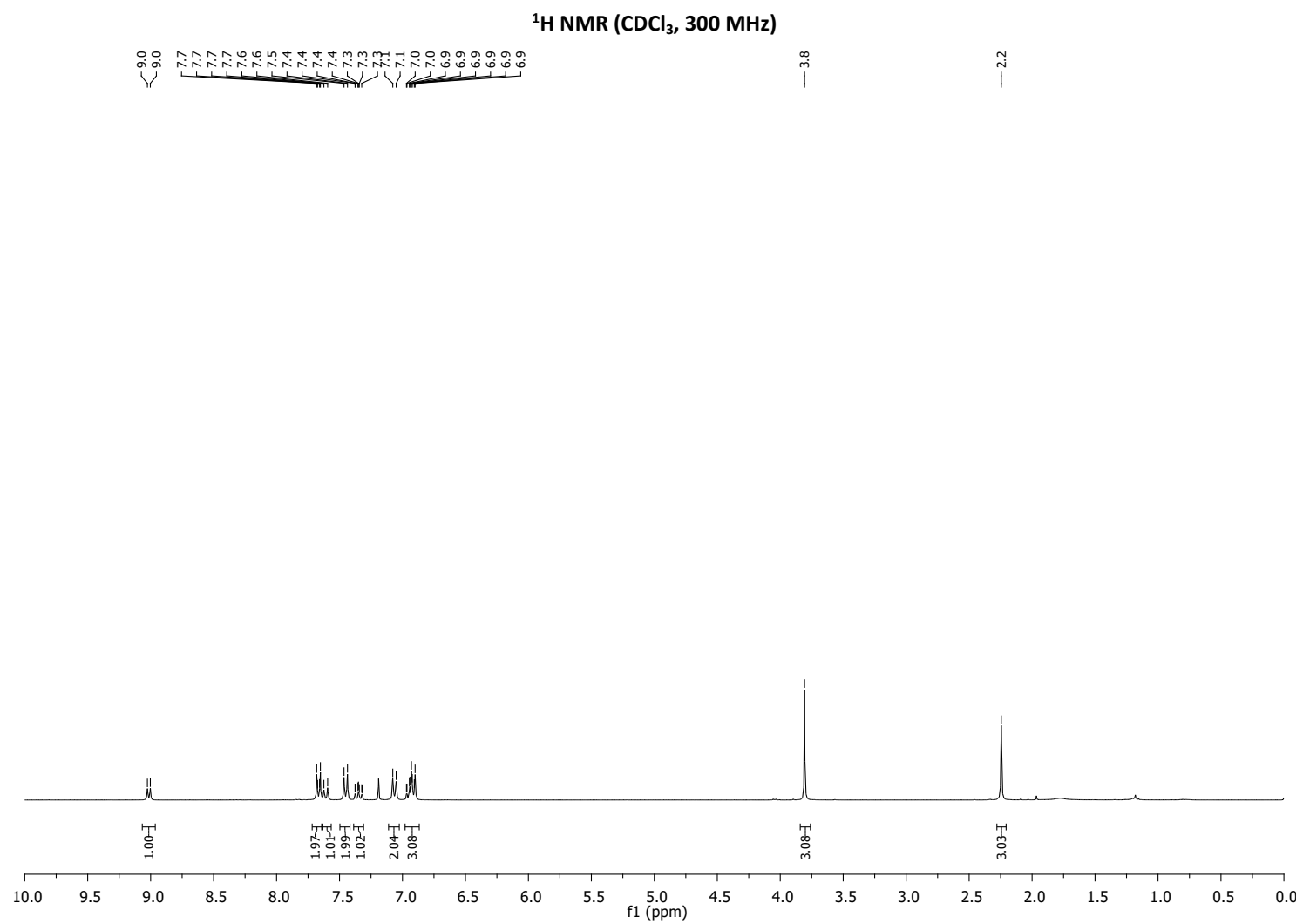


^{13}C NMR (CDCl₃, 75 MHz)

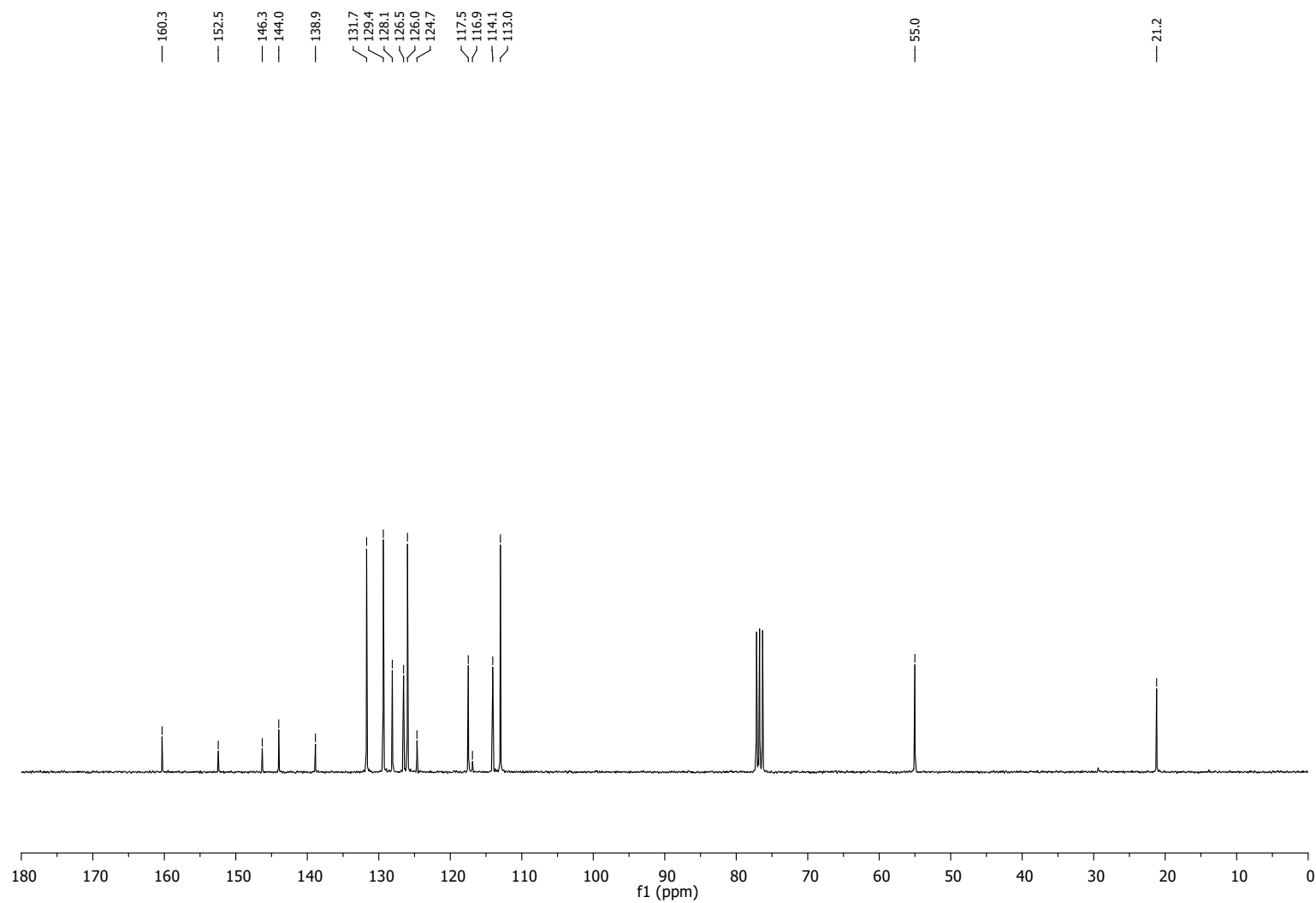


SI46

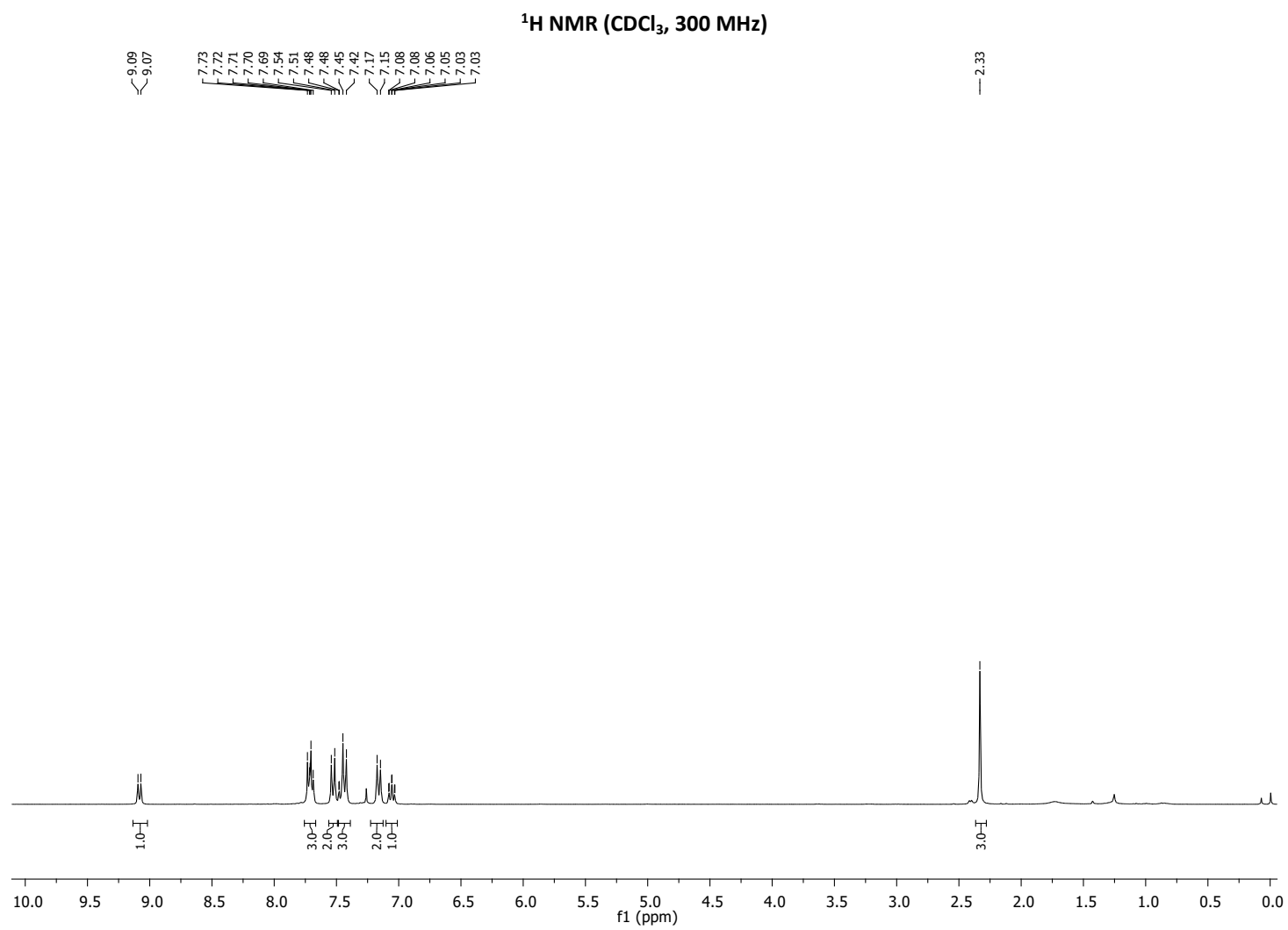
2-(4-methoxyphenyl)-3-tosylimidazo[1,2-a]pyridine **3na**



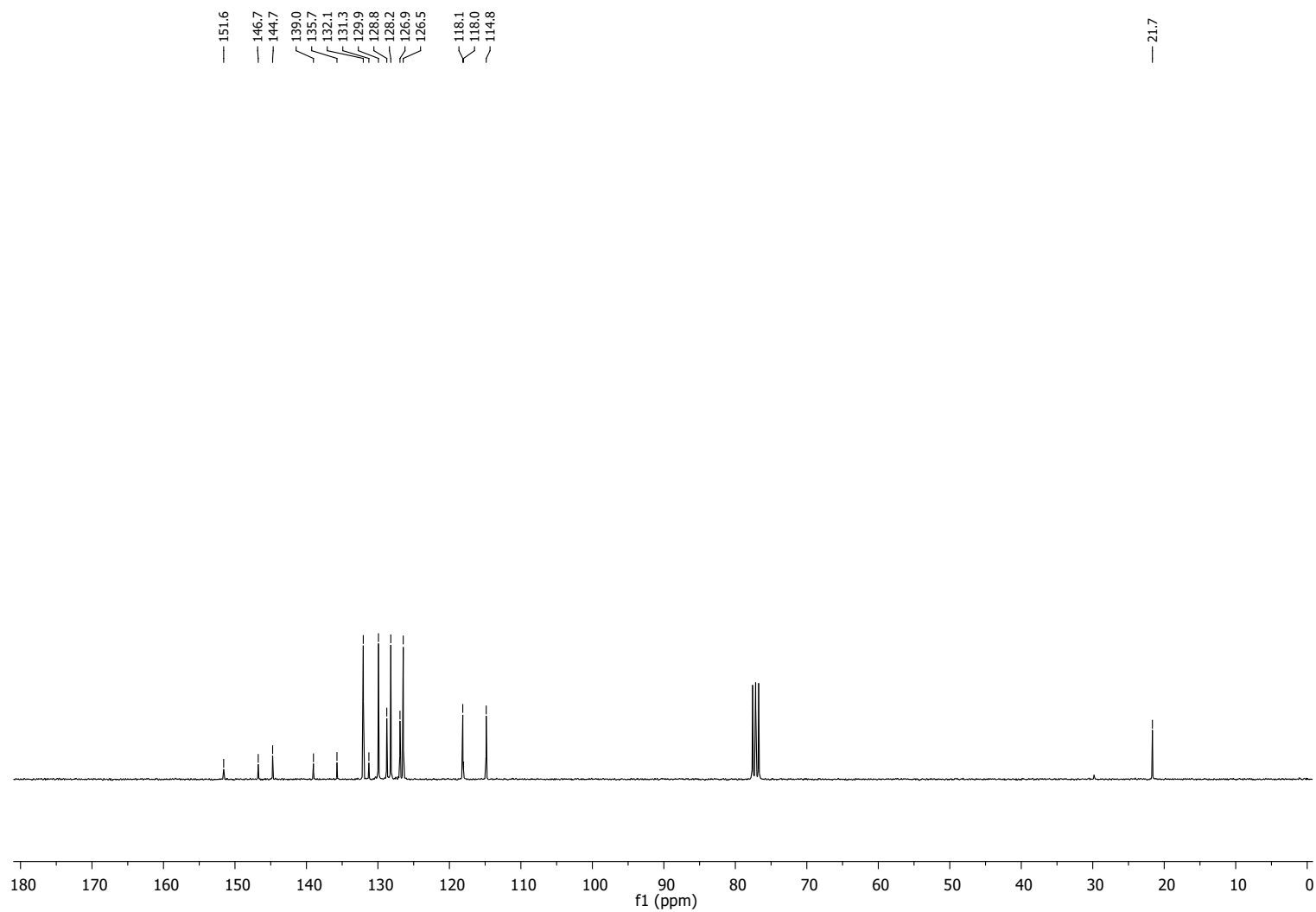
^{13}C NMR (CDCl₃, 75 MHz)



2-(4-chlorophenyl)-3-tosylimidazo[1,2-*a*]pyridine **3oa**

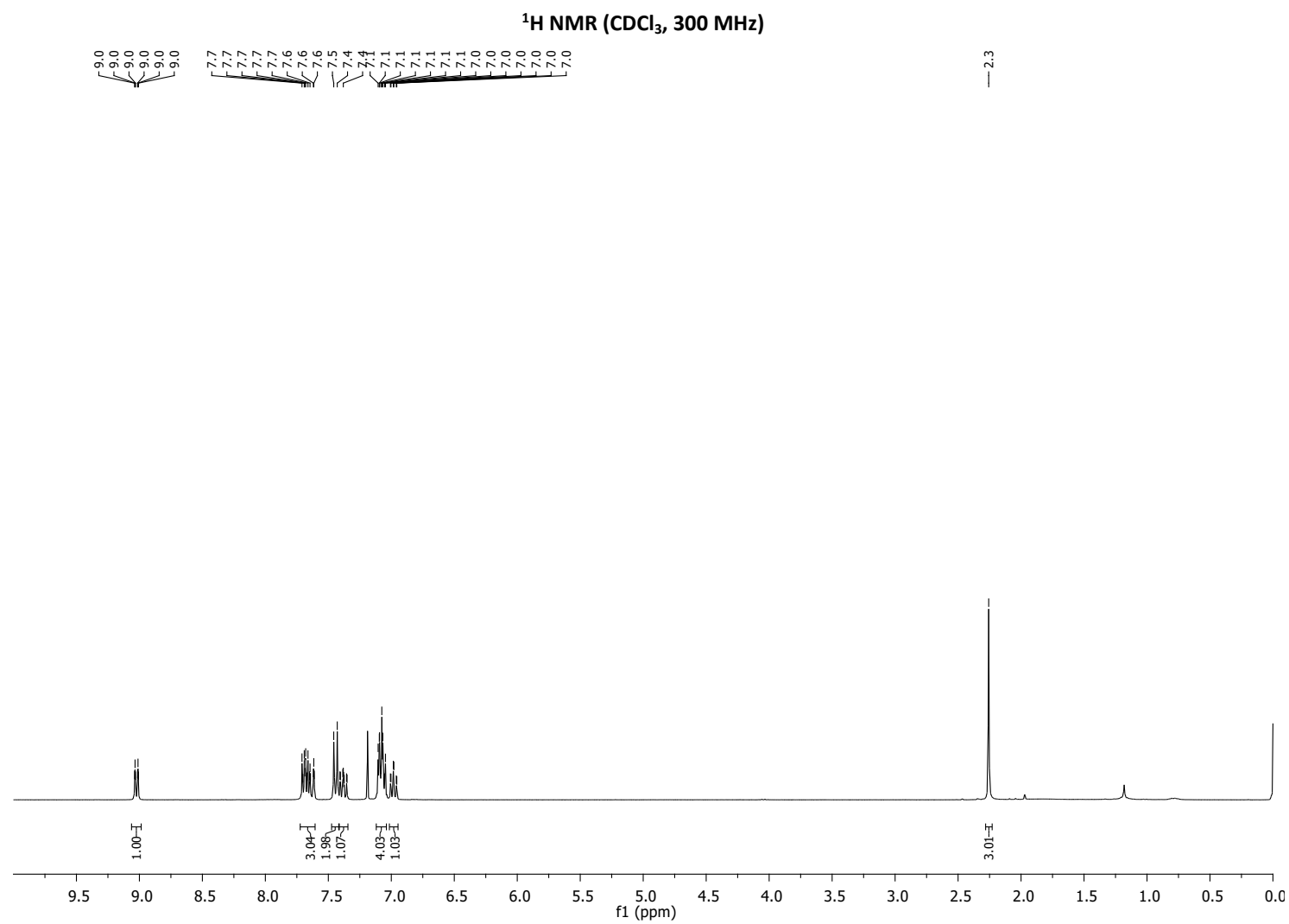


^{13}C NMR (CDCl₃, 75 MHz)

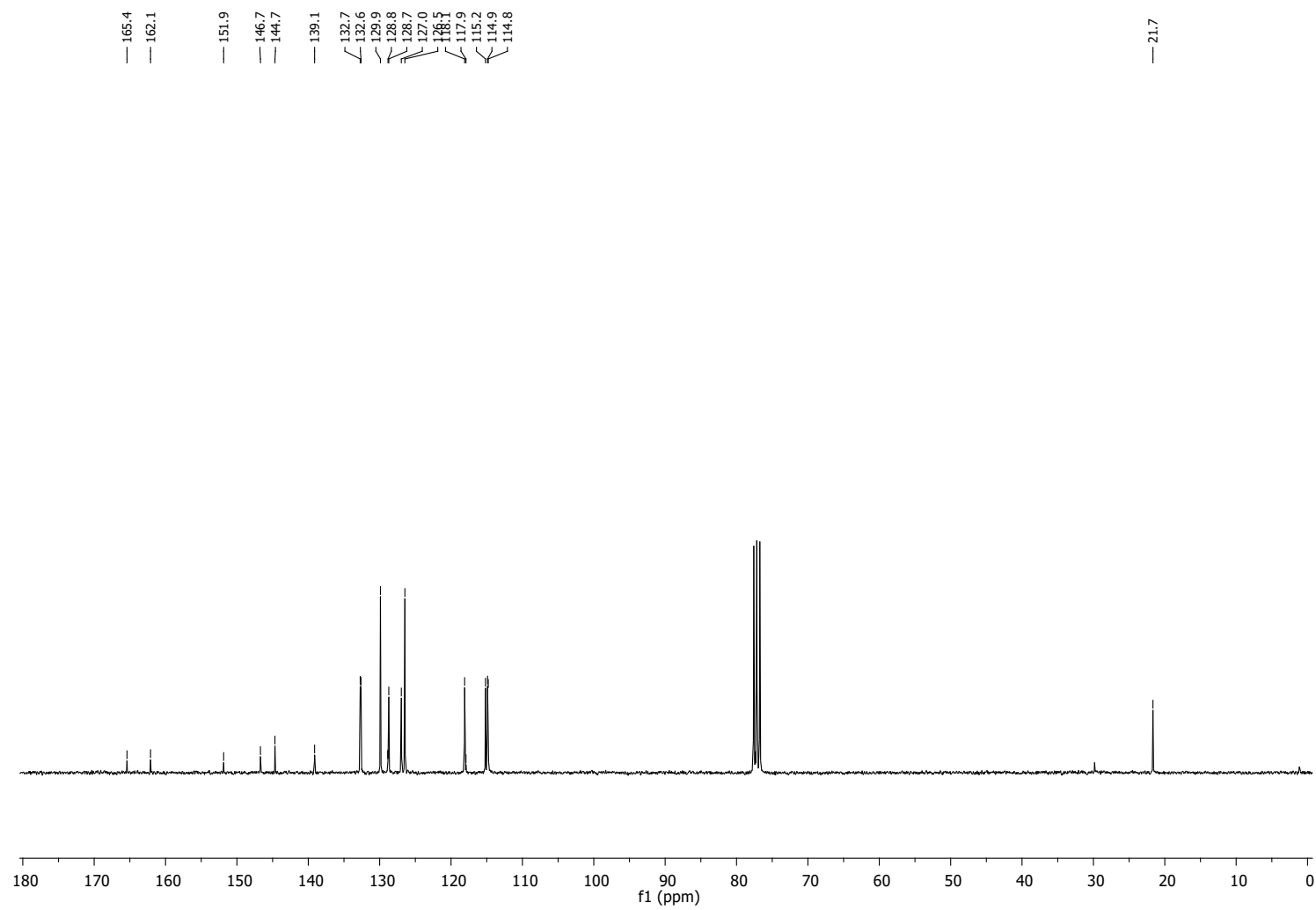


SI50

2-(4-fluorophenyl)-3-tosylimidazo[1,2-a]pyridine **3pa**



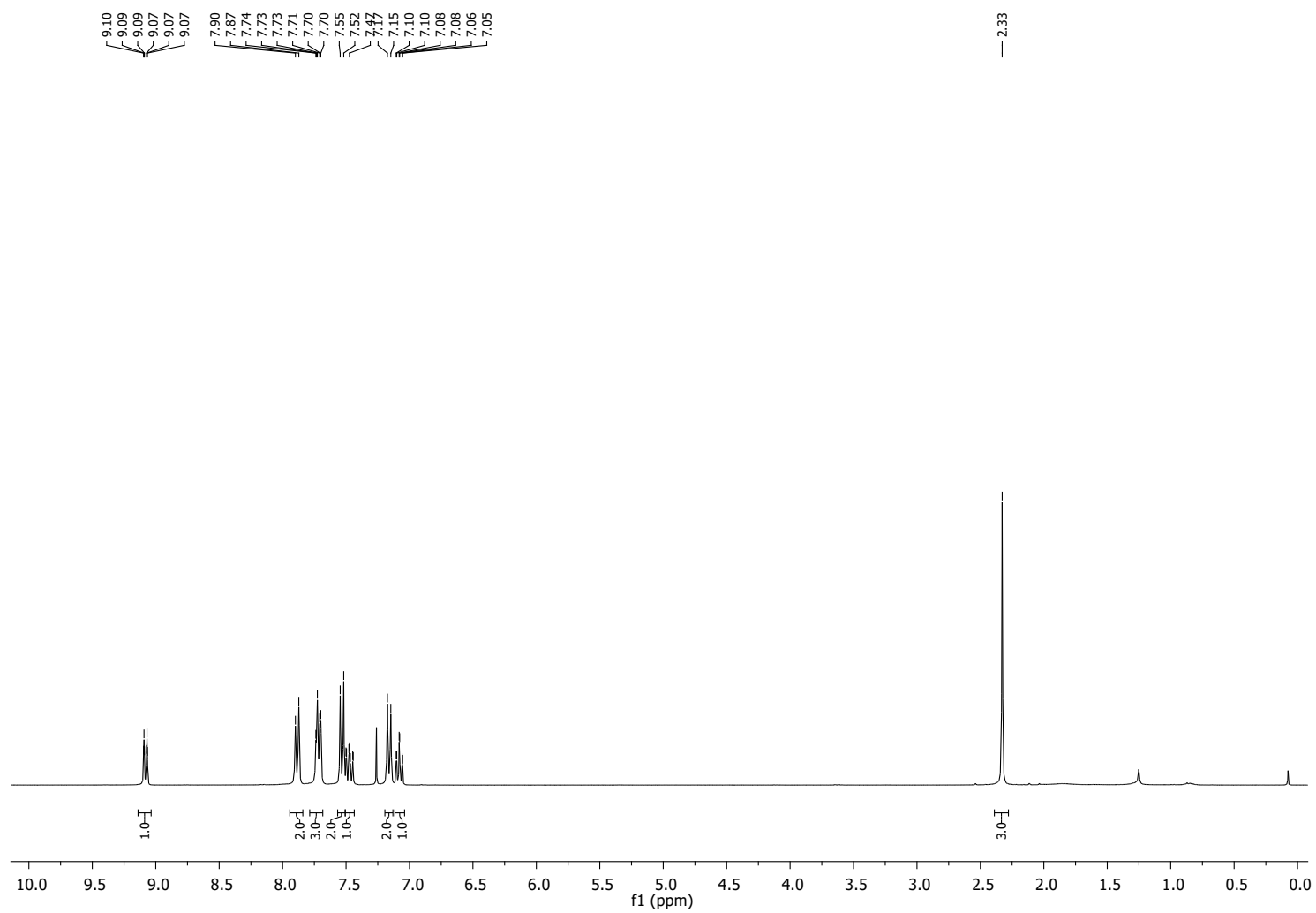
^{13}C NMR (CDCl₃, 75 MHz)



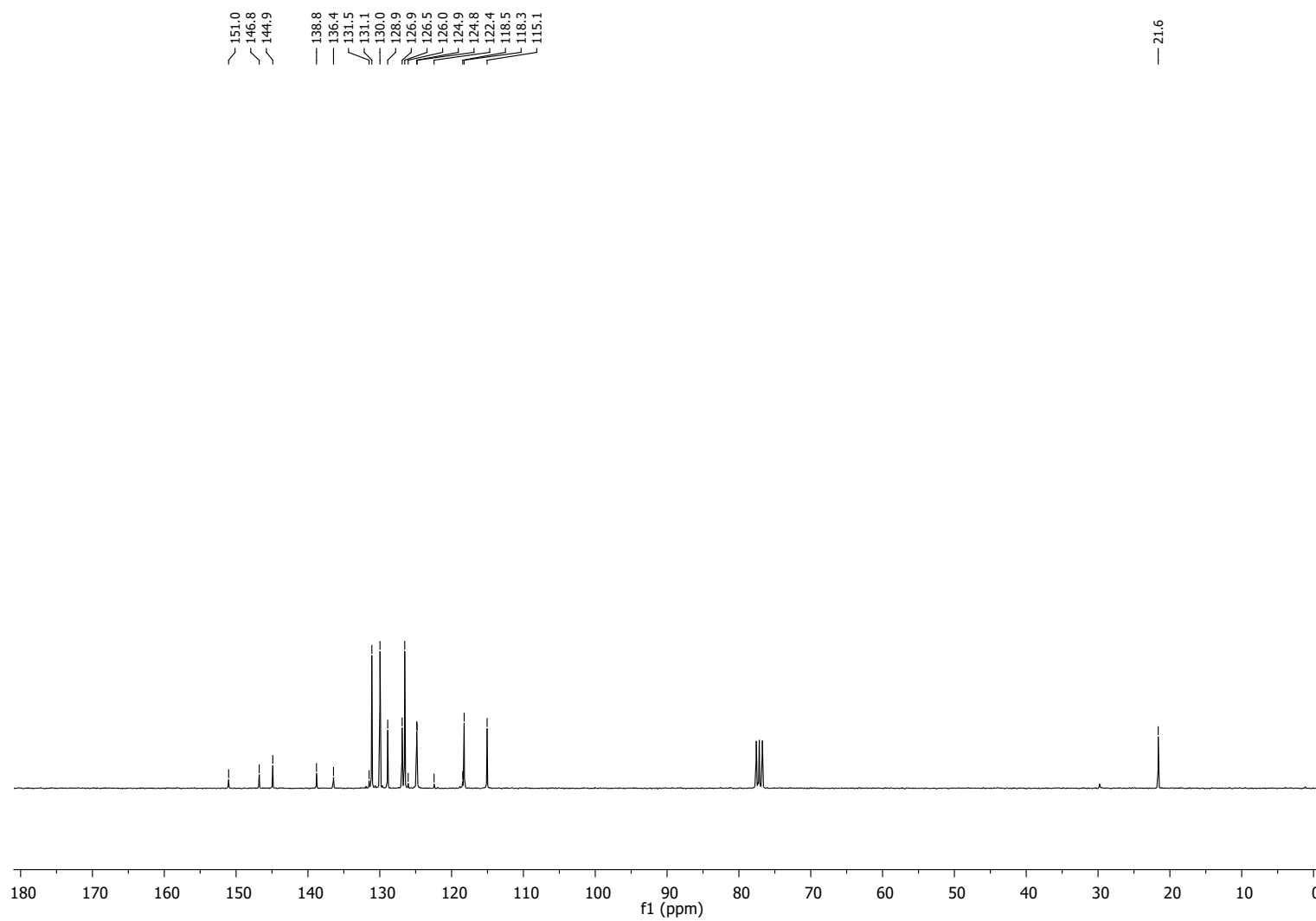
SI52

3-tosyl-2-(4-(trifluoromethyl)phenyl)imidazo[1,2-*a*]pyridine **3qa**

¹H NMR (CDCl₃, 300 MHz)



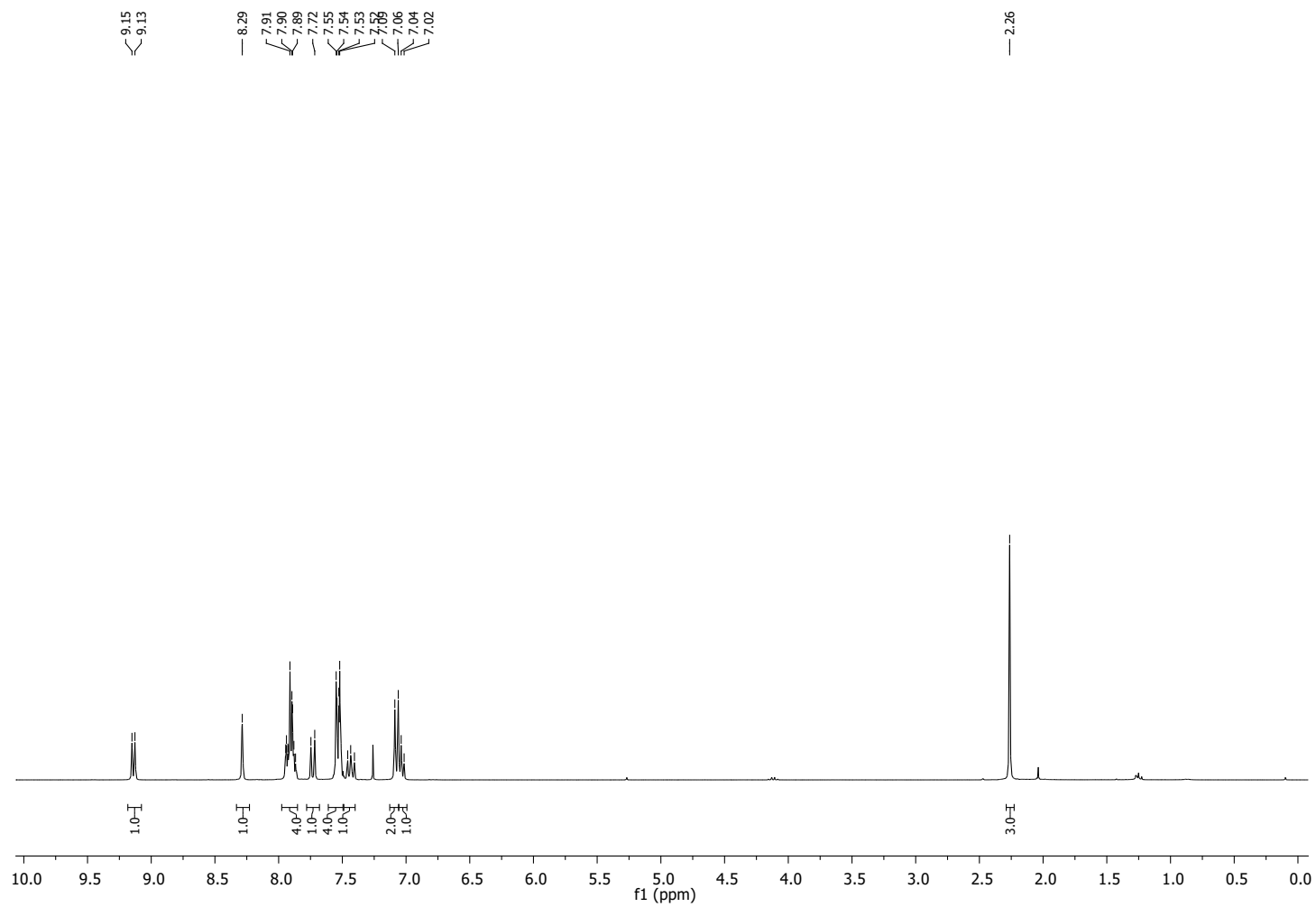
^{13}C NMR (CDCl₃, 75 MHz)



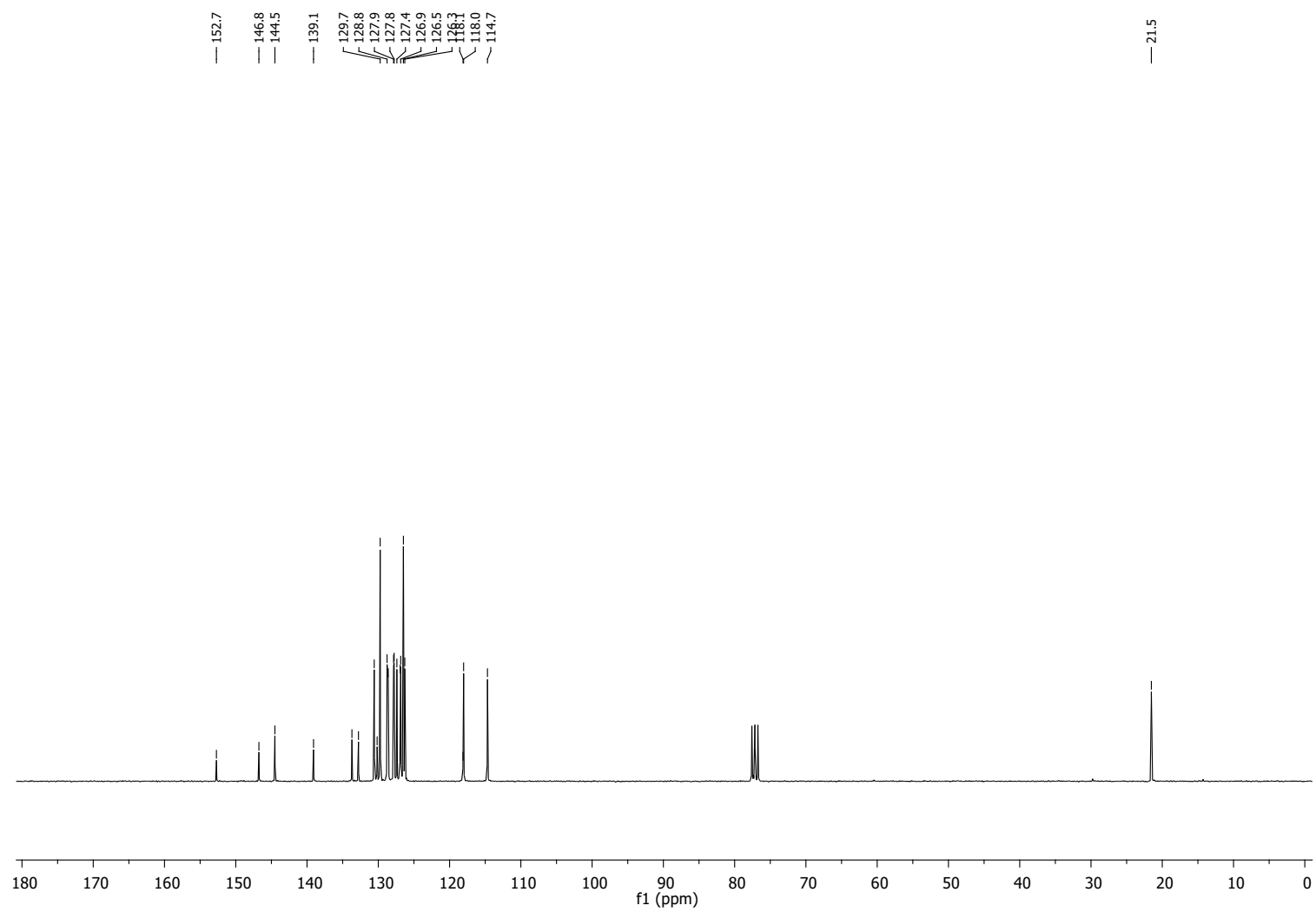
SI54

2-(naphthalen-2-yl)-3-tosylimidazo[1,2-*a*]pyridine **3ra**

¹H NMR (CDCl₃, 300 MHz)



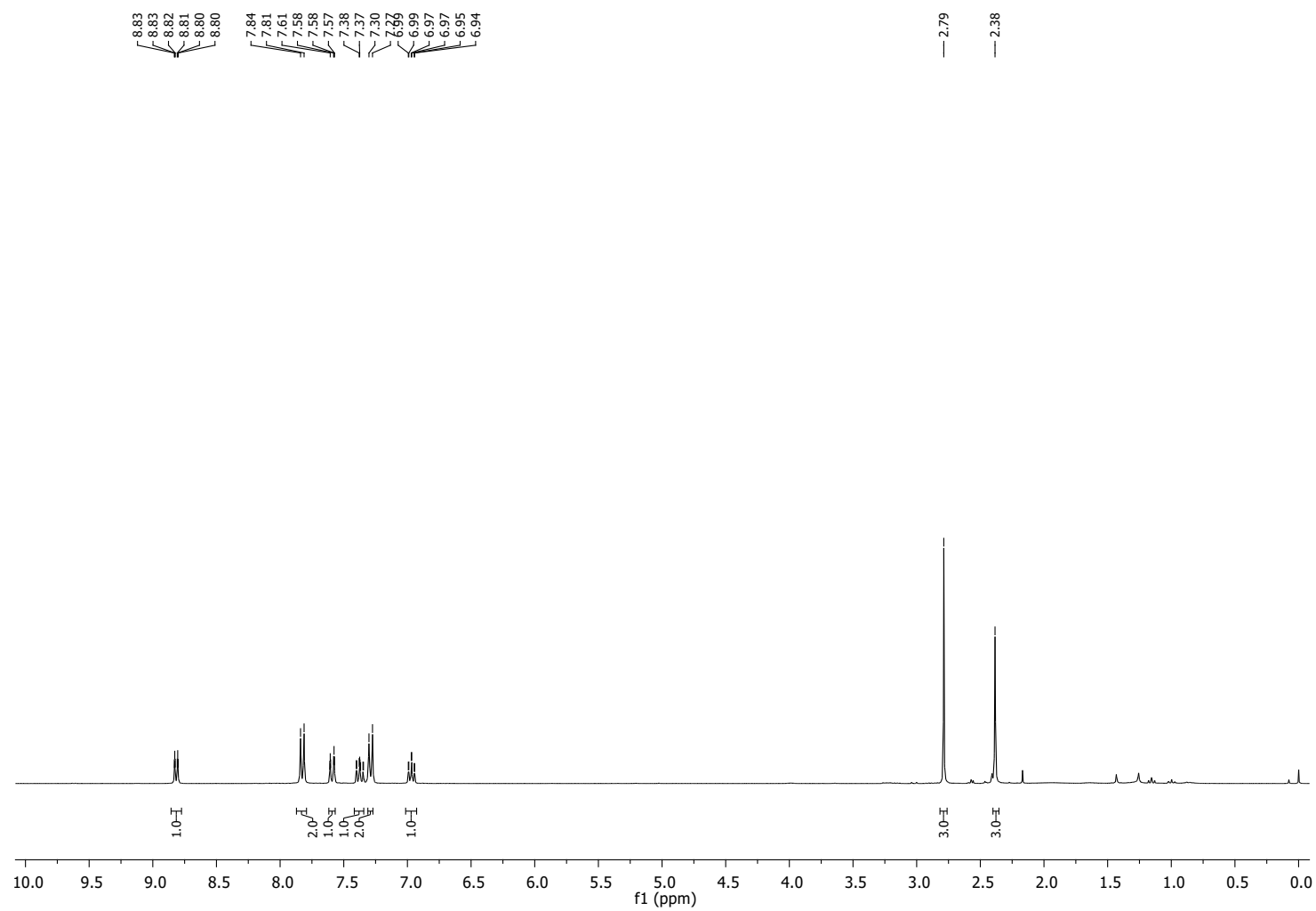
^{13}C NMR (CDCl₃, 75 MHz)



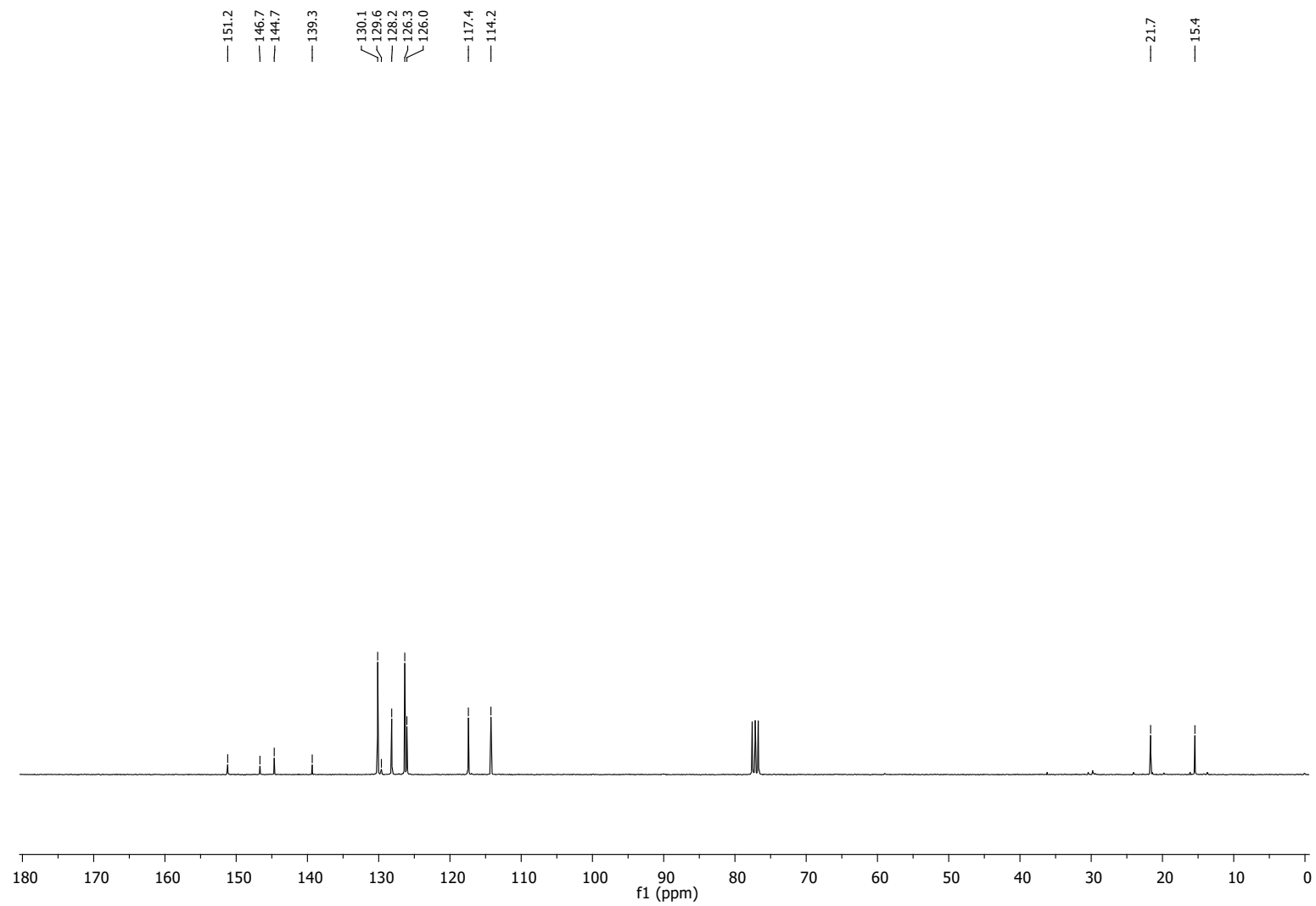
SI56

2-methyl-3-tosylimidazo[1,2-*a*]pyridine **3sa**

¹H NMR (CDCl₃, 300 MHz)

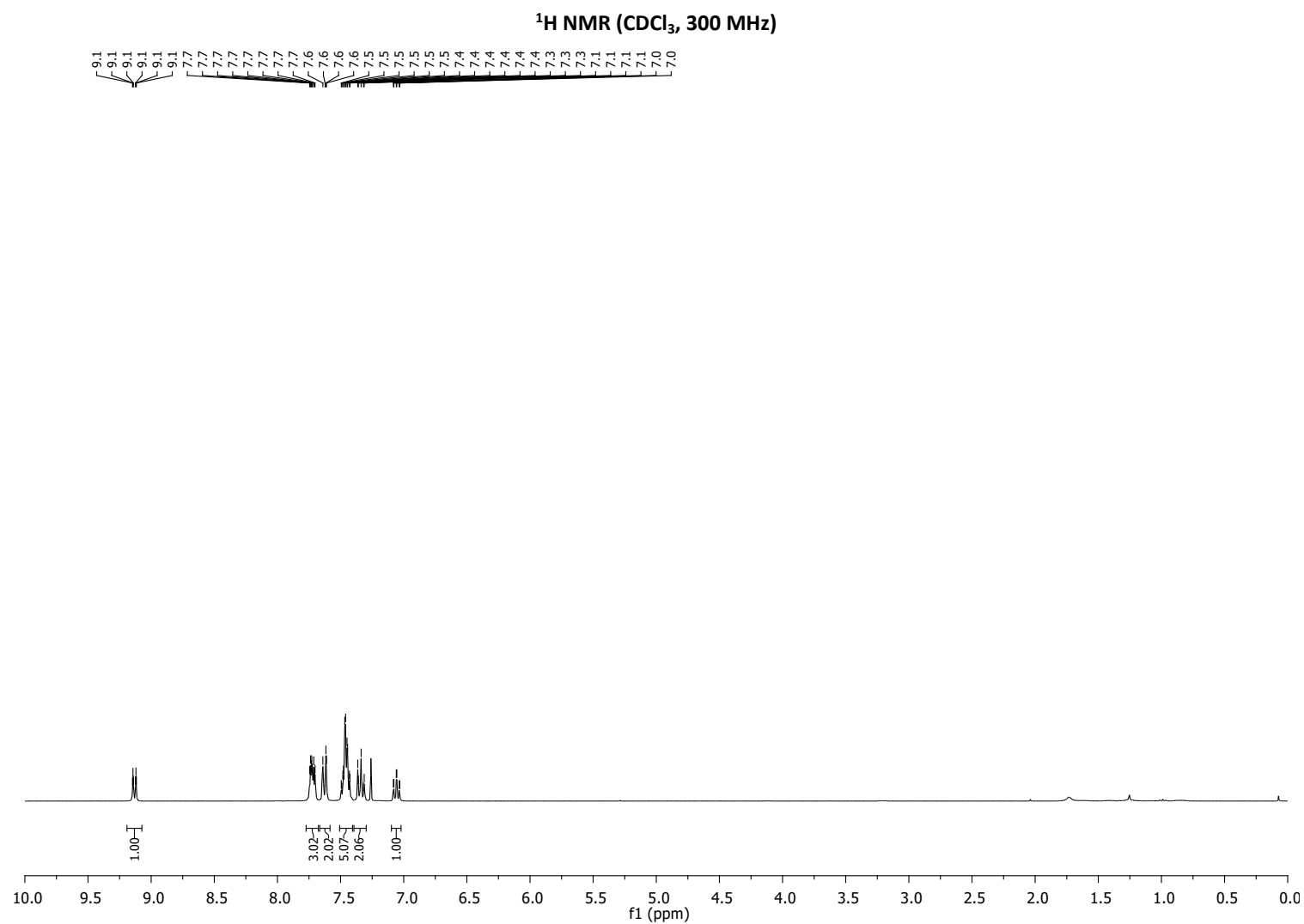


^{13}C NMR (CDCl₃, 75 MHz)

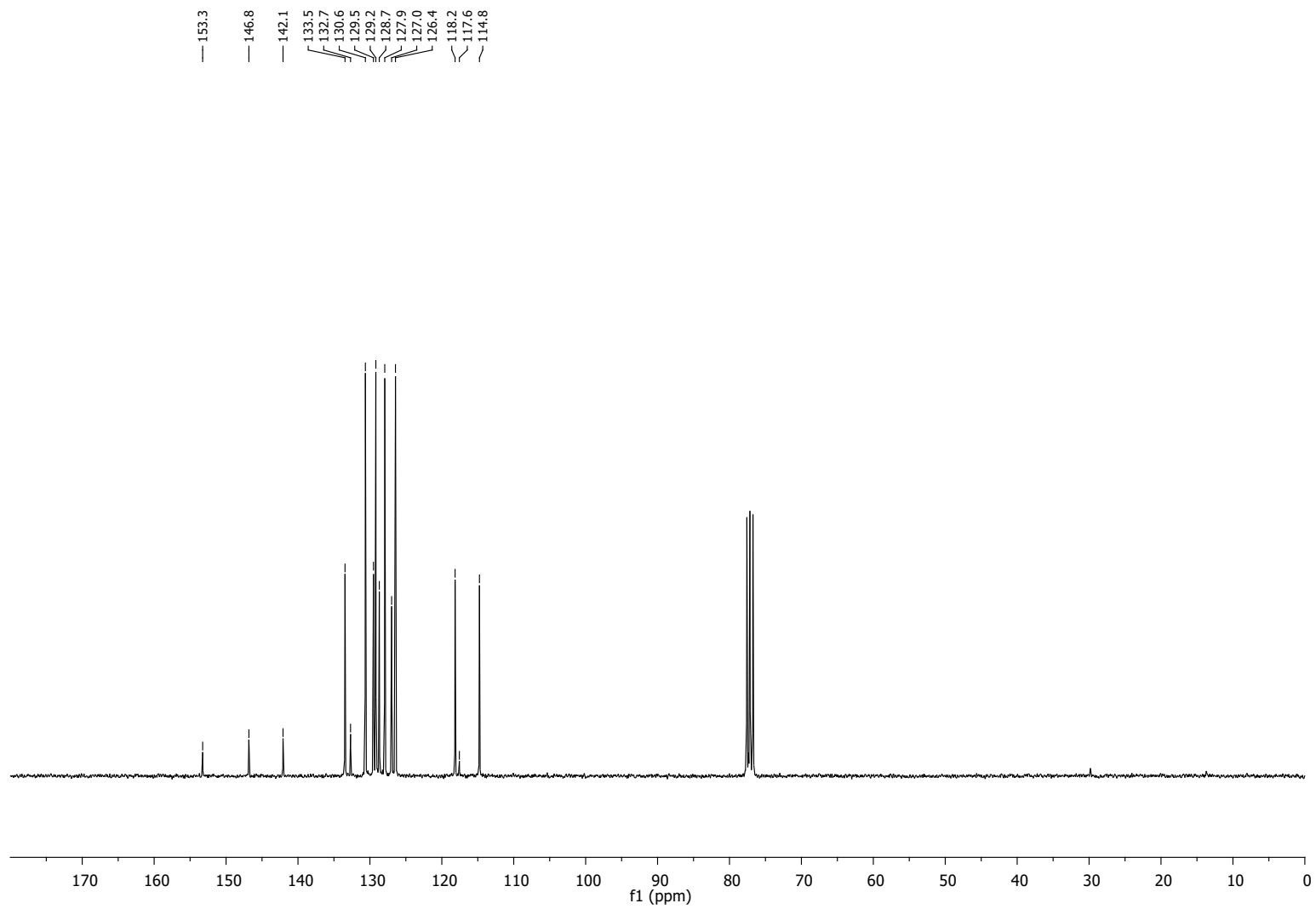


SI58

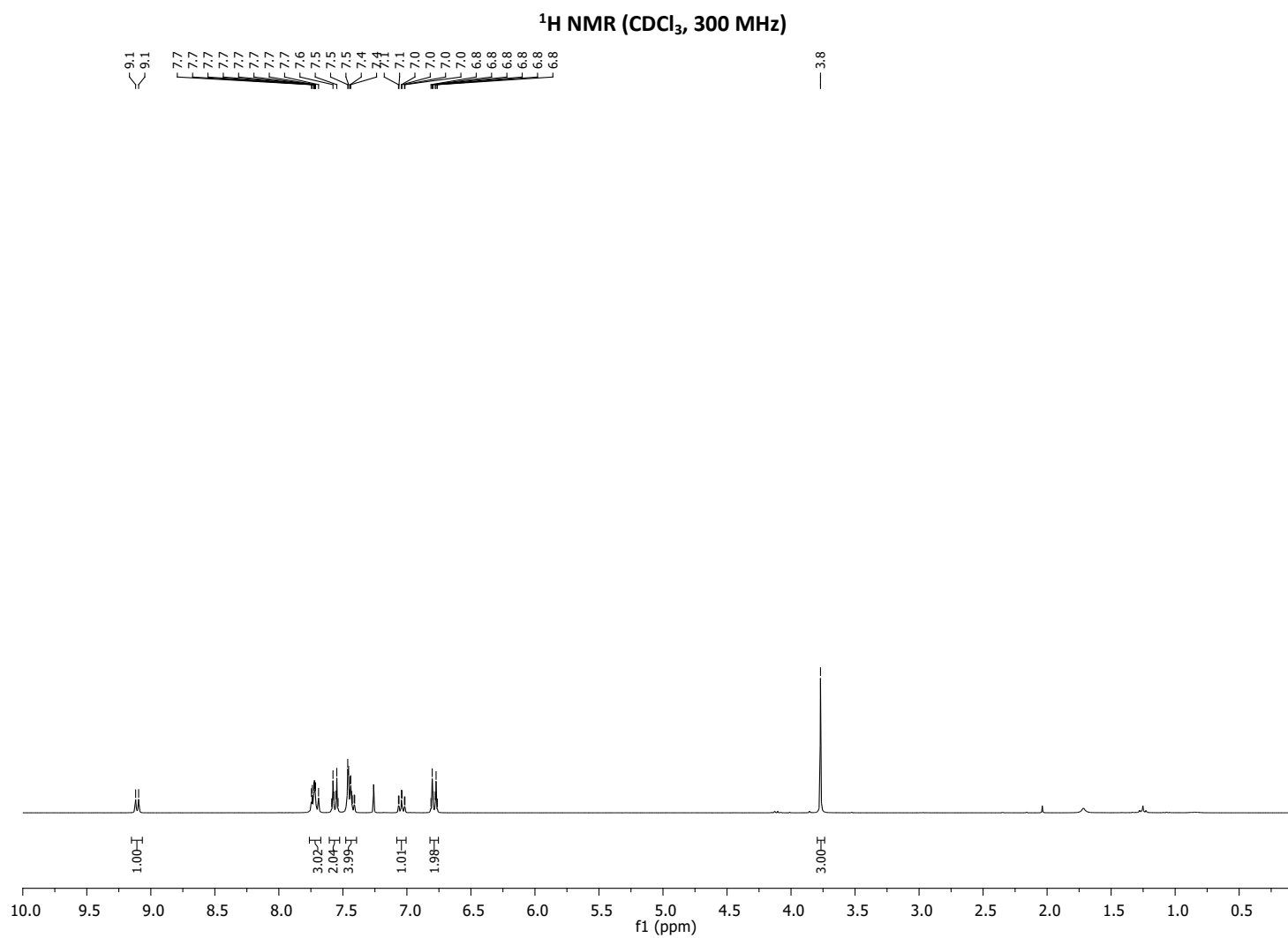
2-phenyl-3-(phenylsulfonyl)imidazo[1,2-a]pyridine **3ab**



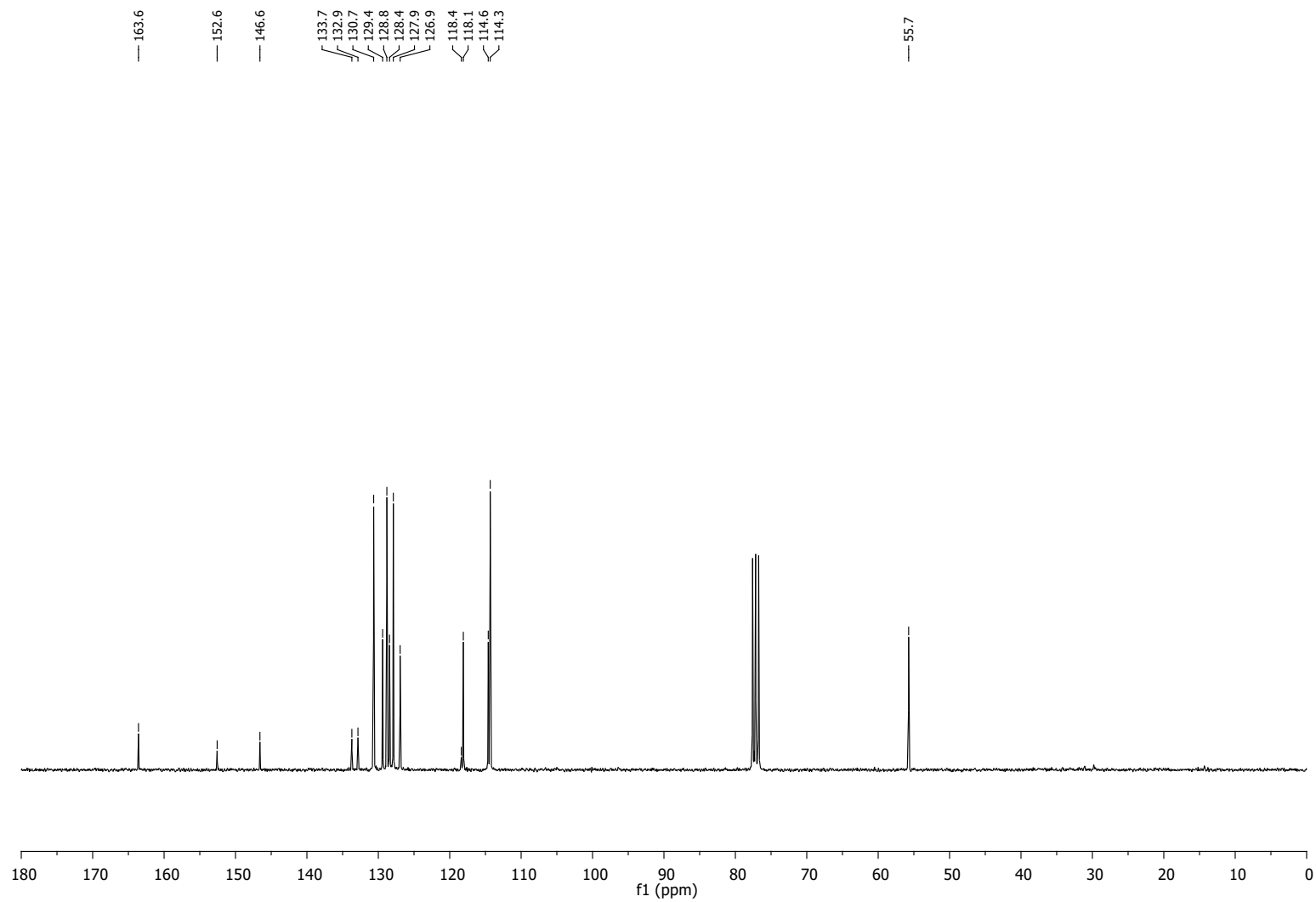
^{13}C NMR (CDCl₃, 75 MHz)



3-((4-methoxyphenyl)sulfonyl)-2-phenylimidazo[1,2-*a*]pyridine **3ac**

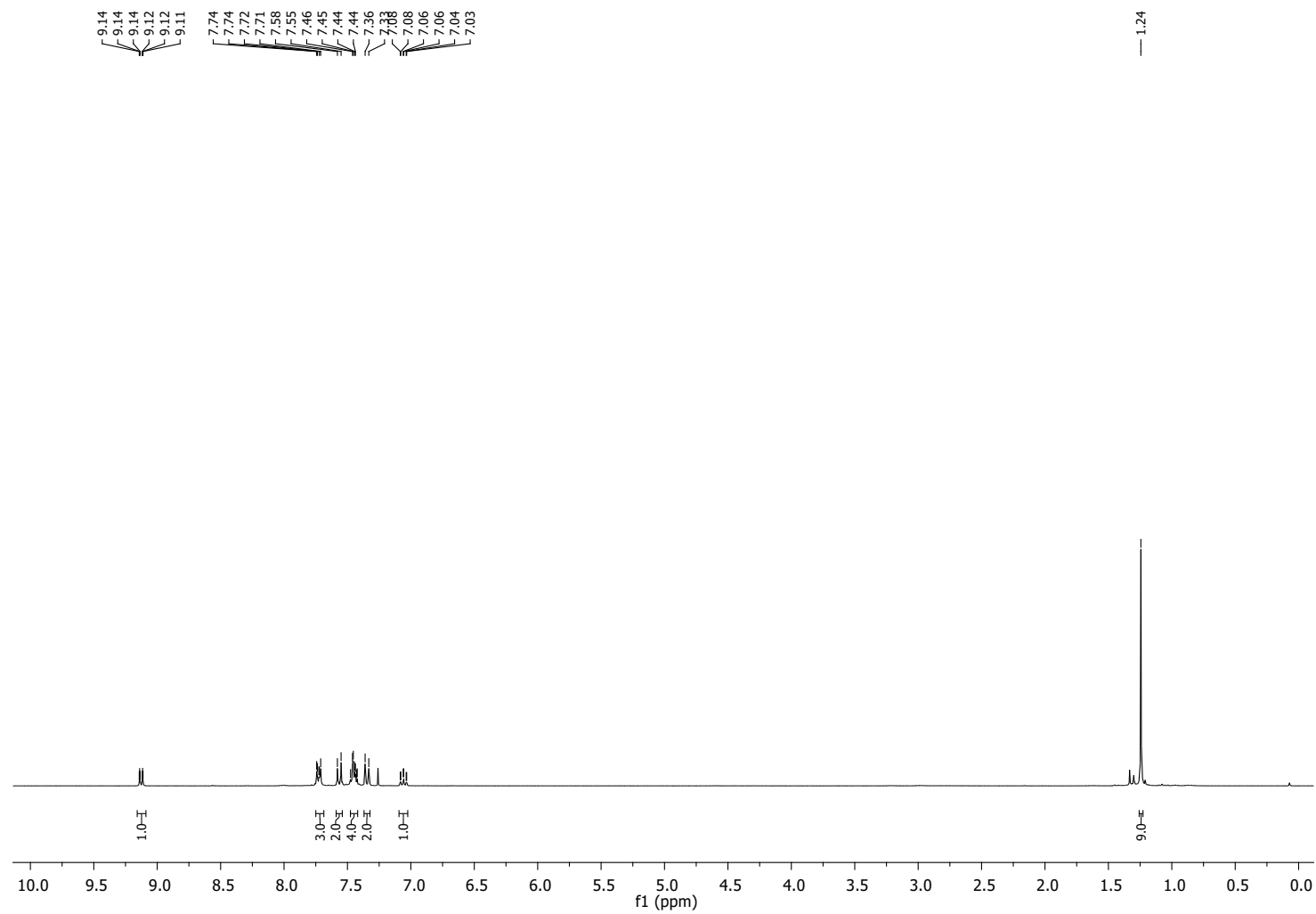


¹³C NMR (CDCl₃, 75 MHz)

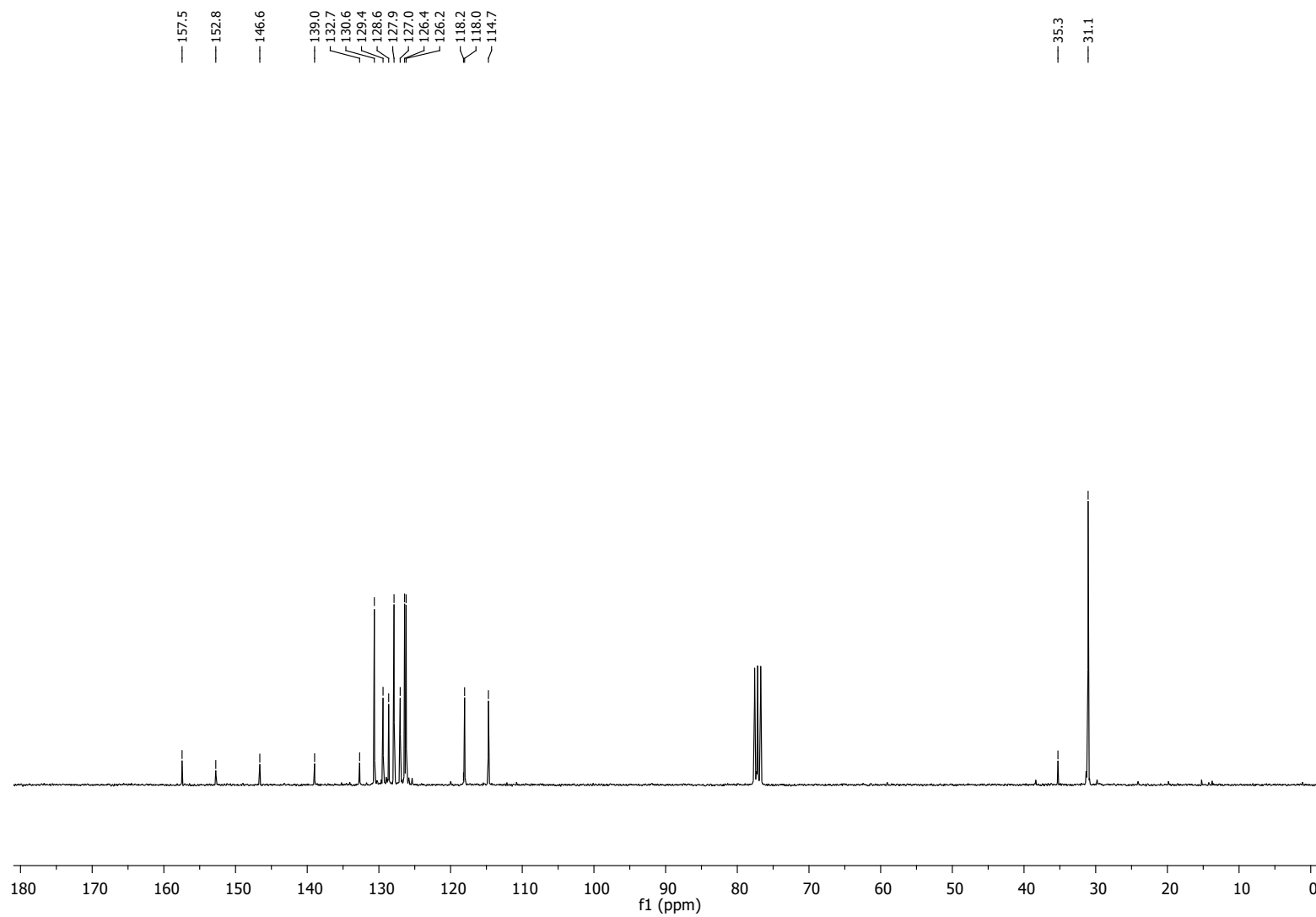


3-((4-(*tert*-butyl)phenyl)sulfonyl)-2-phenylimidazo[1,2-*a*]pyridine **3ad**

¹H NMR (CDCl₃, 300 MHz)

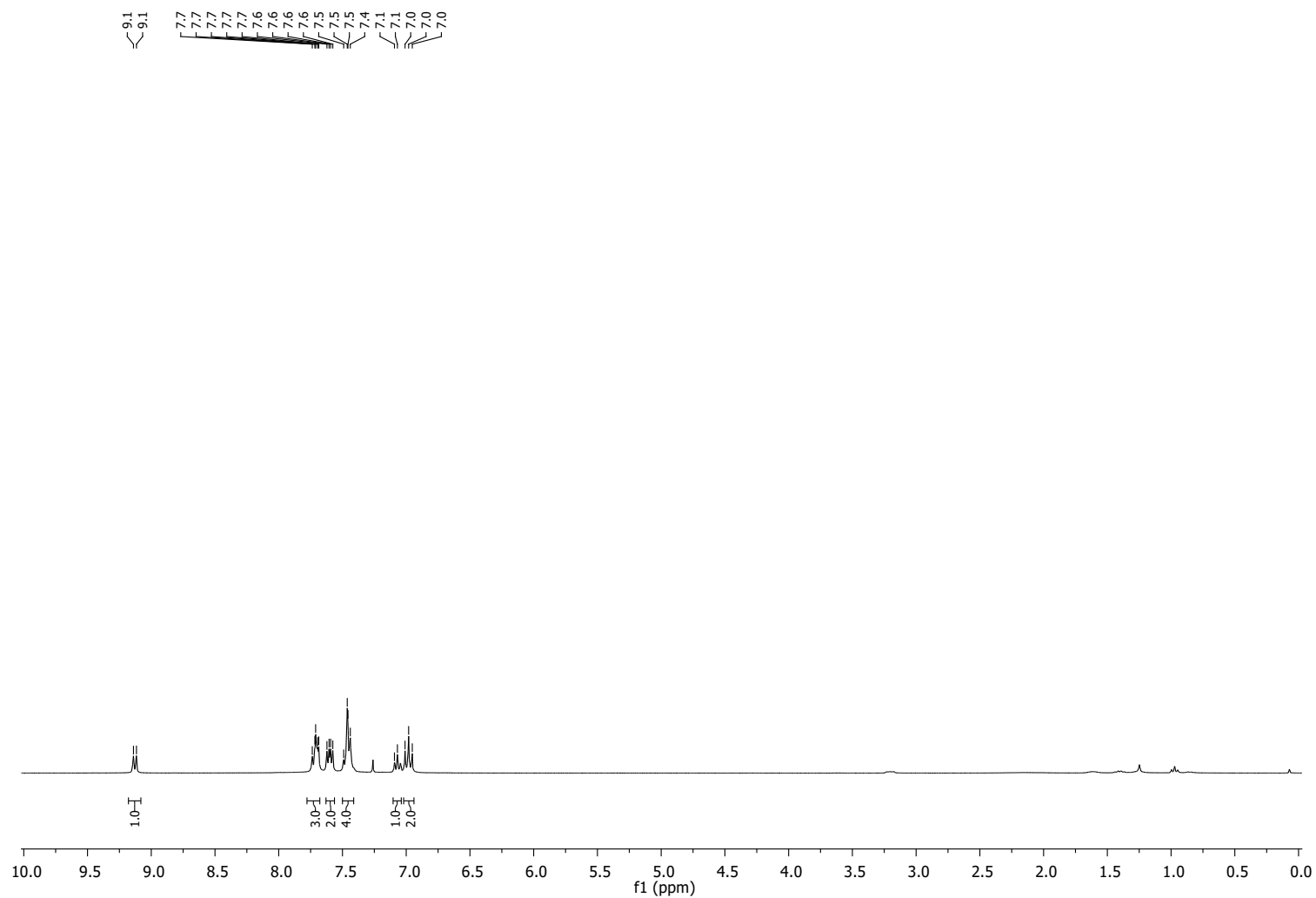


^{13}C NMR (CDCl₃, 75 MHz)

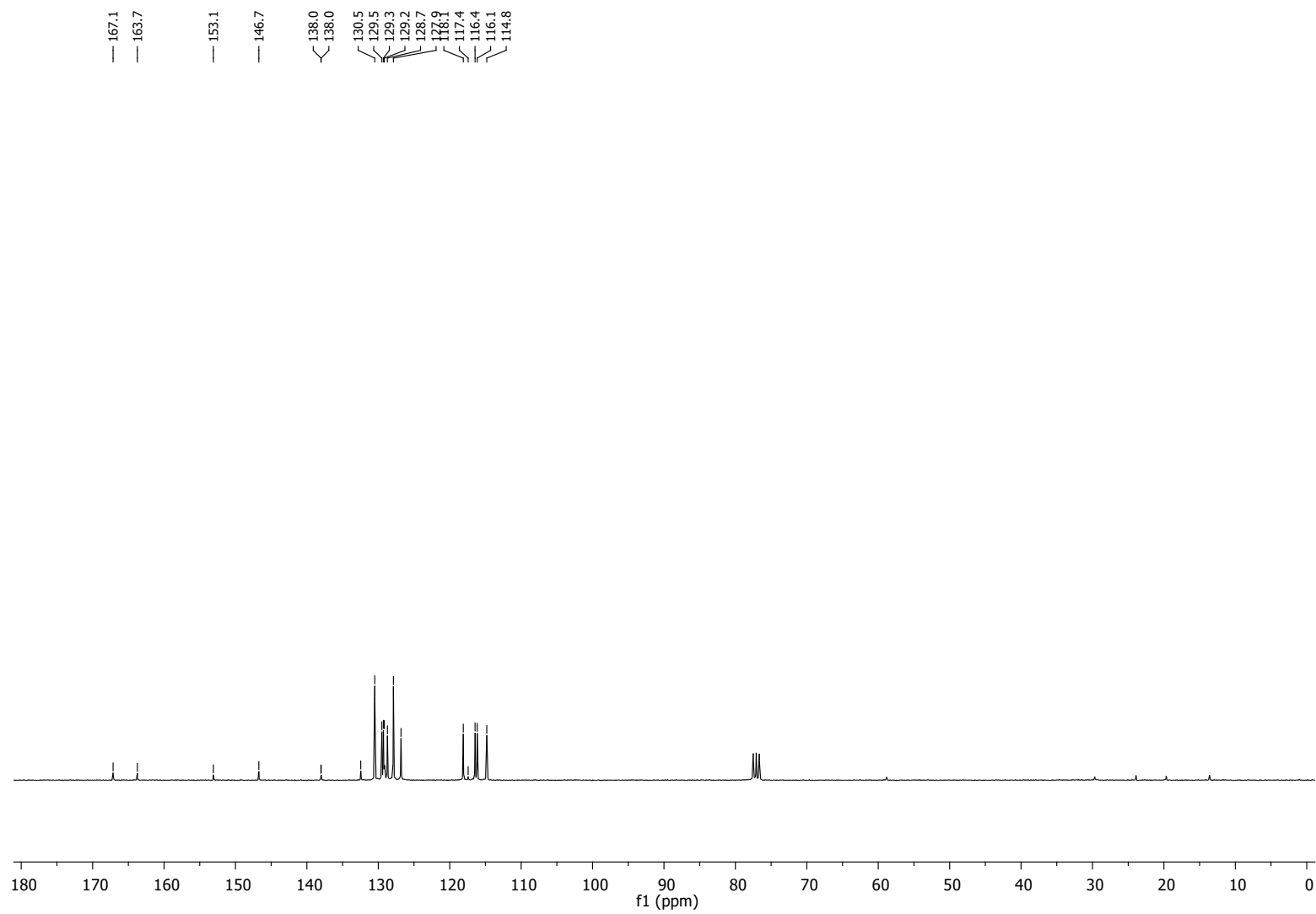


3-((4-fluorophenyl)sulfonyl)-2-phenylimidazo[1,2-*a*]pyridine **3ae**

¹H NMR (CDCl₃, 300 MHz)



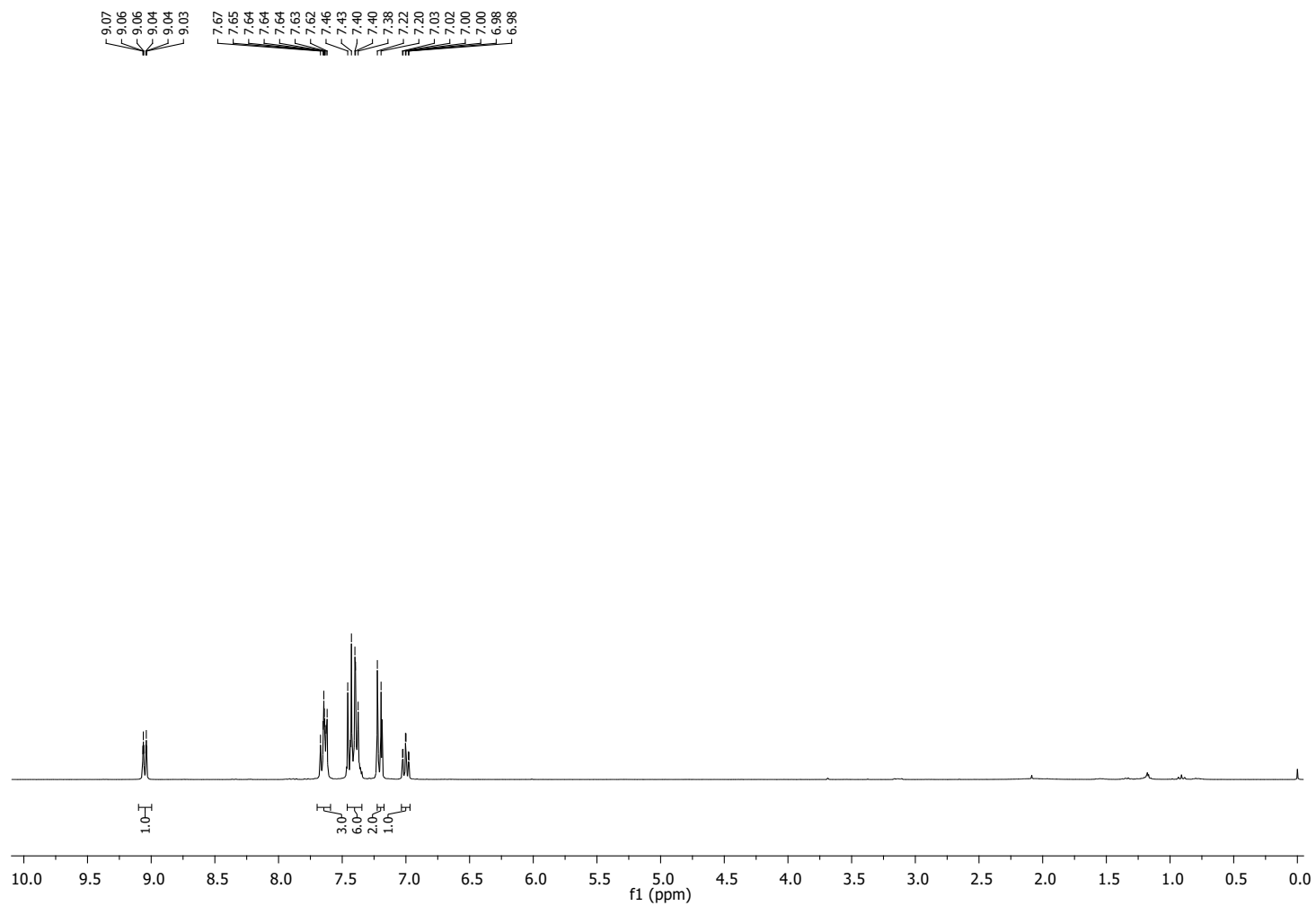
^{13}C NMR (CDCl₃, 75 MHz)



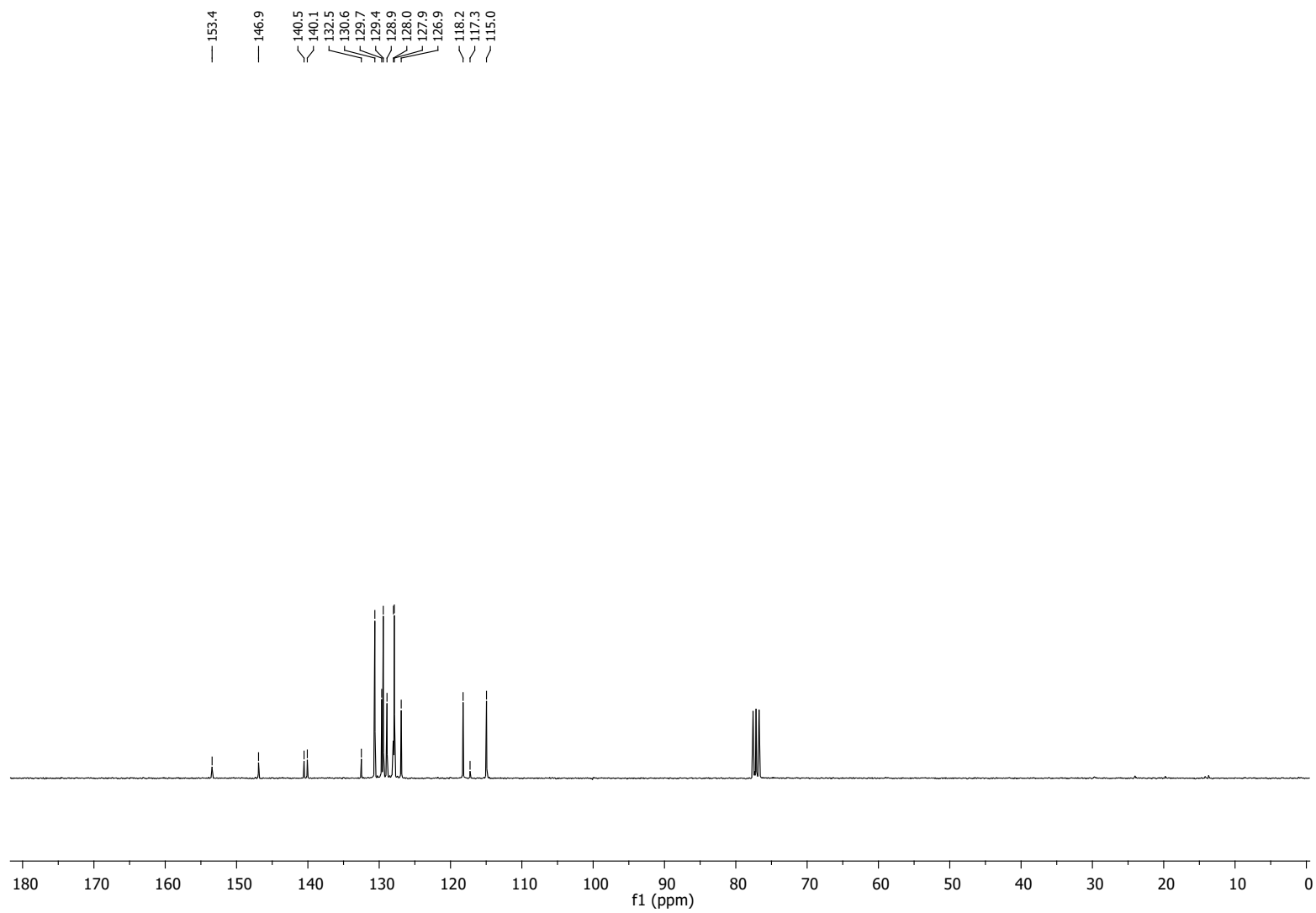
SI66

3-((4-chlorophenyl)sulfonyl)-2-phenylimidazo[1,2-*a*]pyridine **3af**

¹H NMR (CDCl₃, 300 MHz)

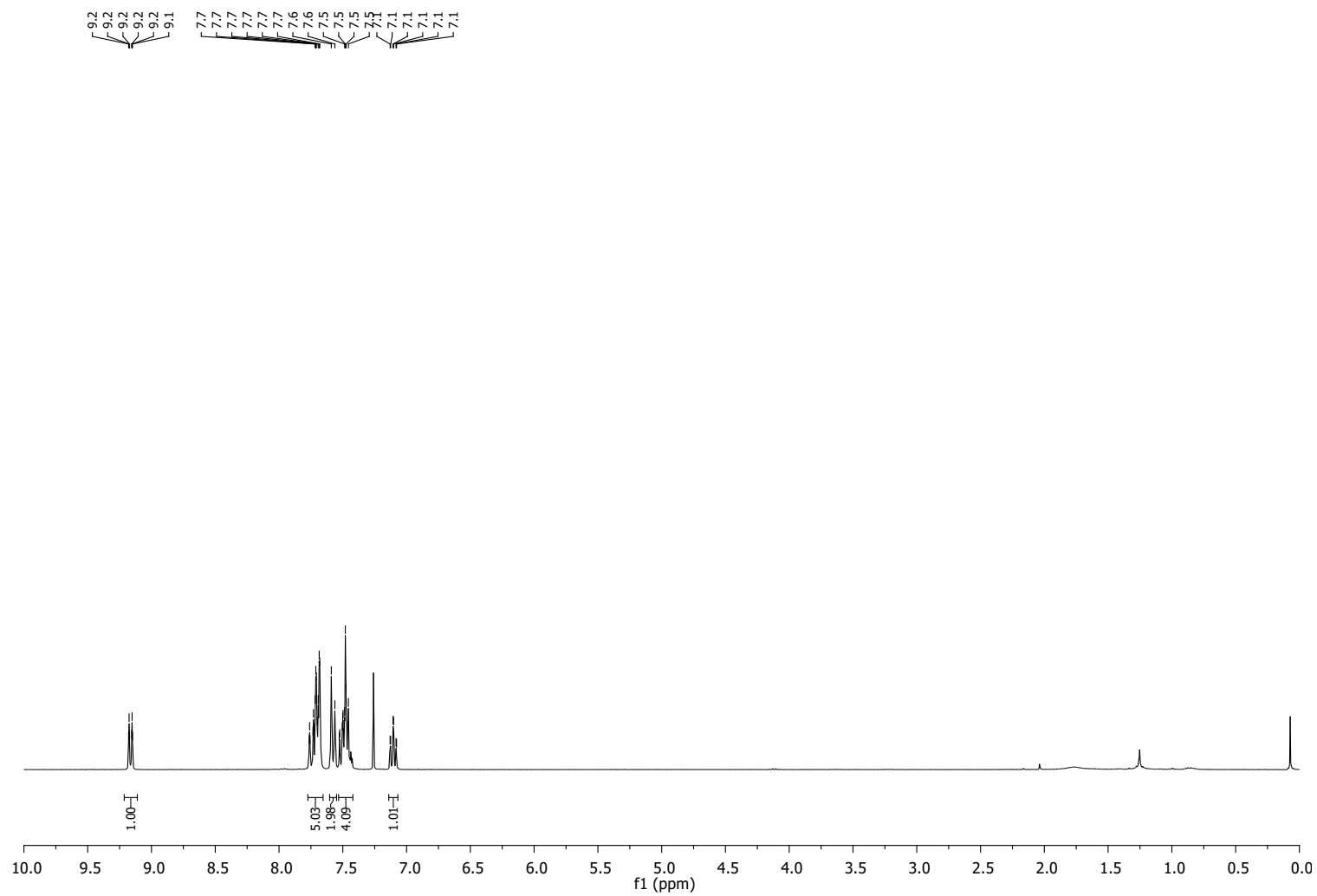


^{13}C NMR (CDCl₃, 75 MHz)

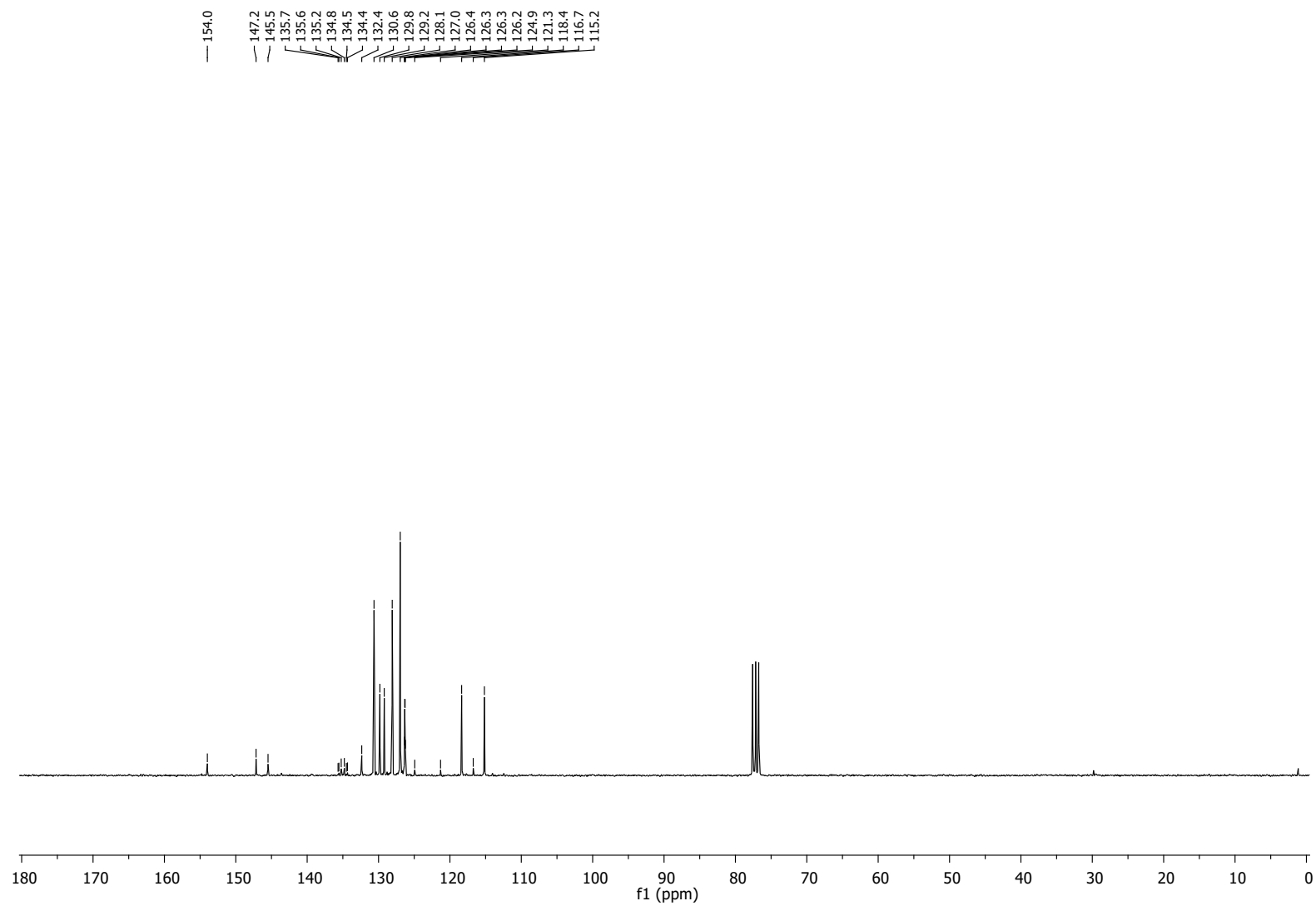


2-phenyl-3-((4-(trifluoromethyl)phenyl)sulfonyl)imidazo[1,2-*a*]pyridine **3ag**

¹H NMR (CDCl₃, 300 MHz)



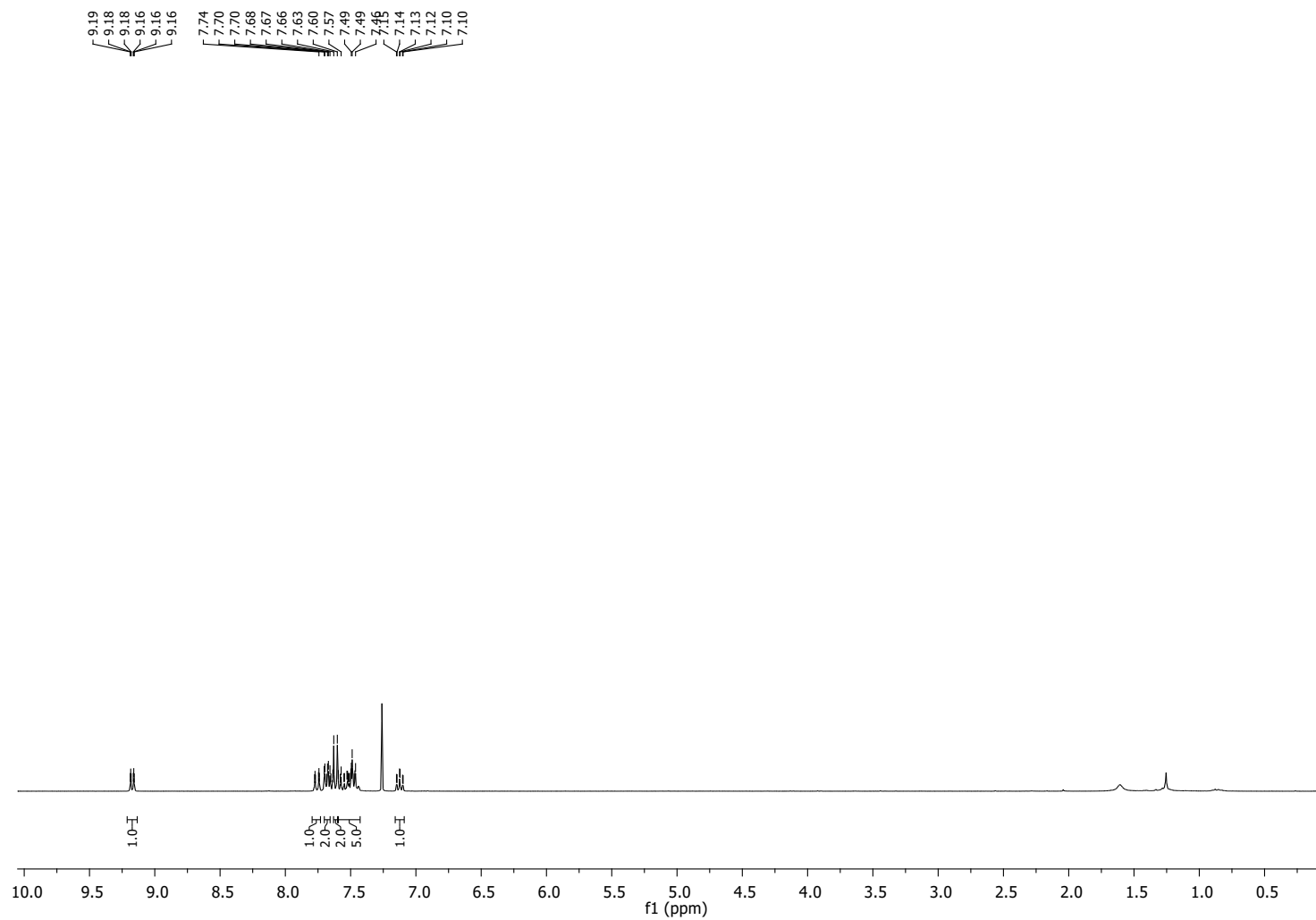
^{13}C NMR (CDCl₃, 75 MHz)



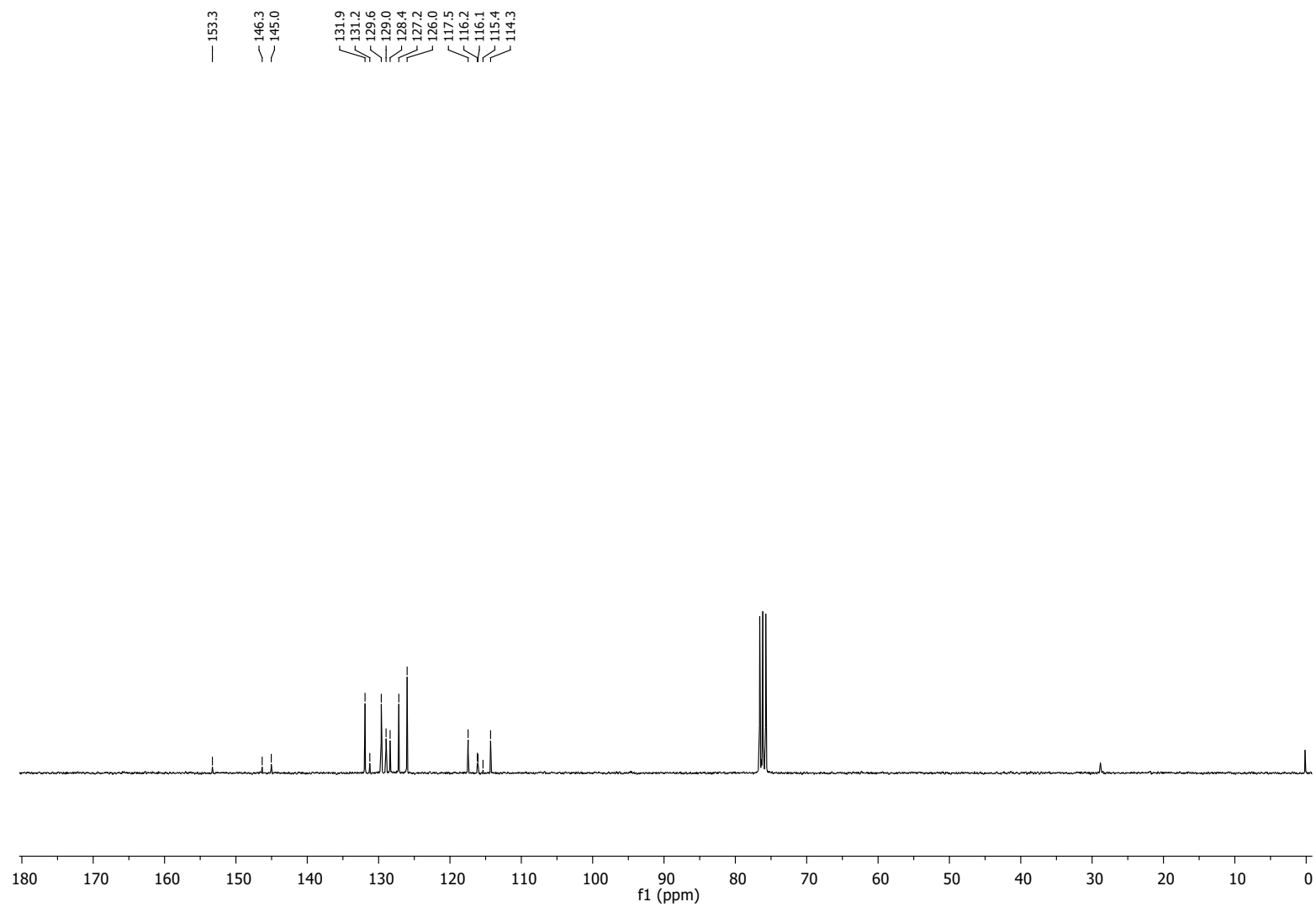
SI70

4-((2-phenylimidazo[1,2-*a*]pyridin-3-yl)sulfonyl)benzonitrile **3ah**

¹H NMR (CDCl₃, 300 MHz)



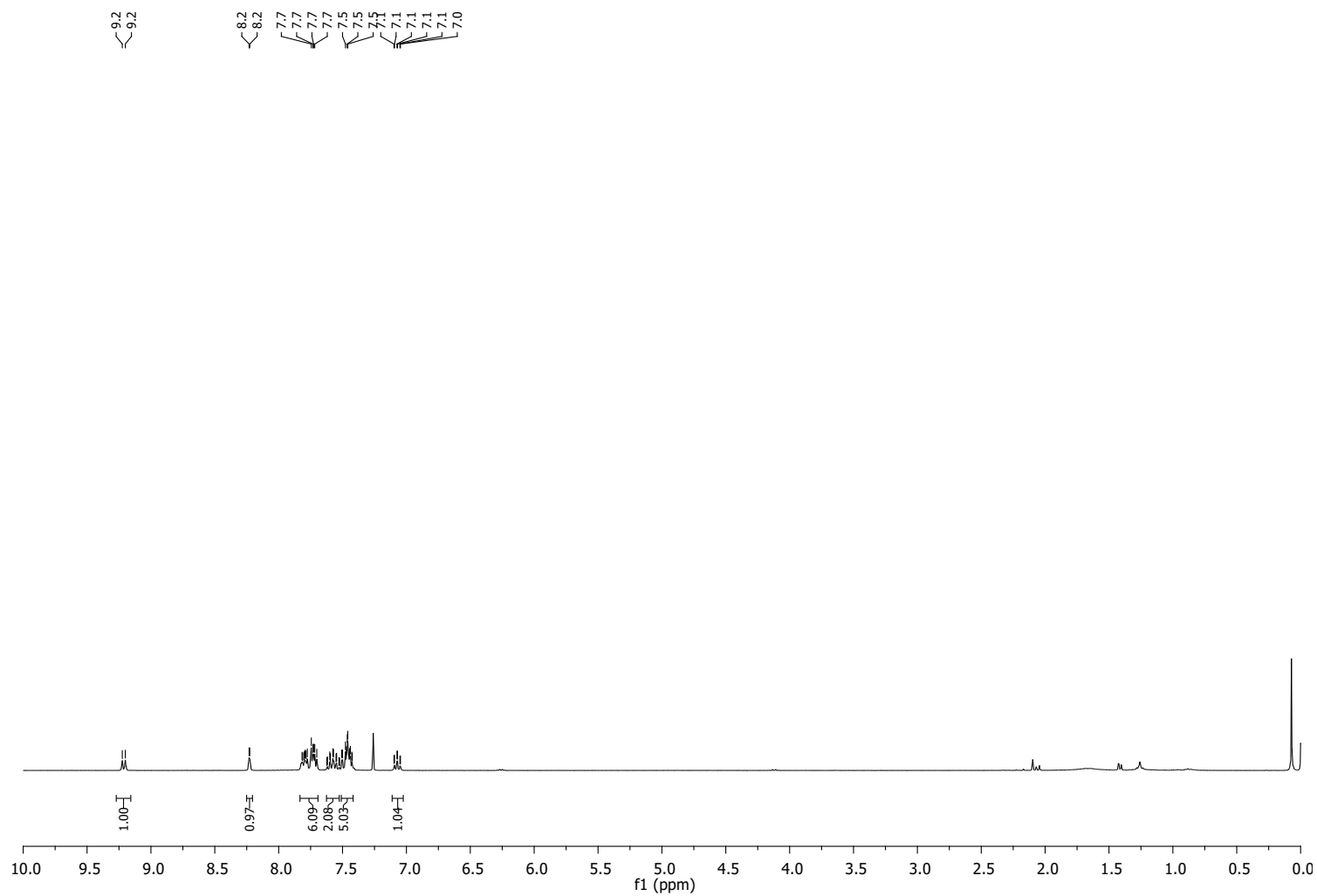
^{13}C NMR (CDCl₃, 75 MHz)



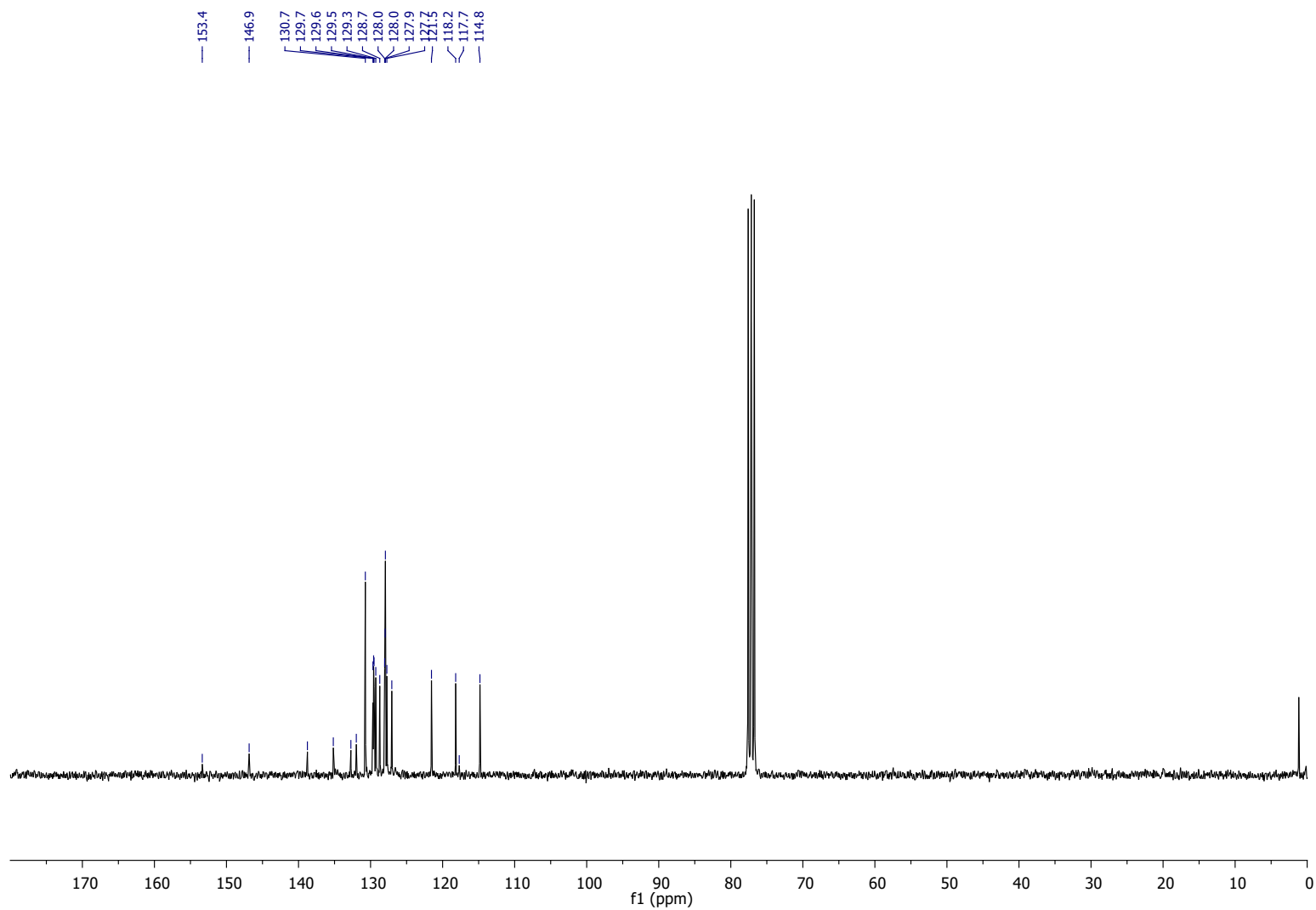
SI72

3-(naphthalen-2-ylsulfonyl)-2-phenylimidazo[1,2-*a*]pyridine **3ai**

¹H NMR (CDCl₃, 300 MHz)



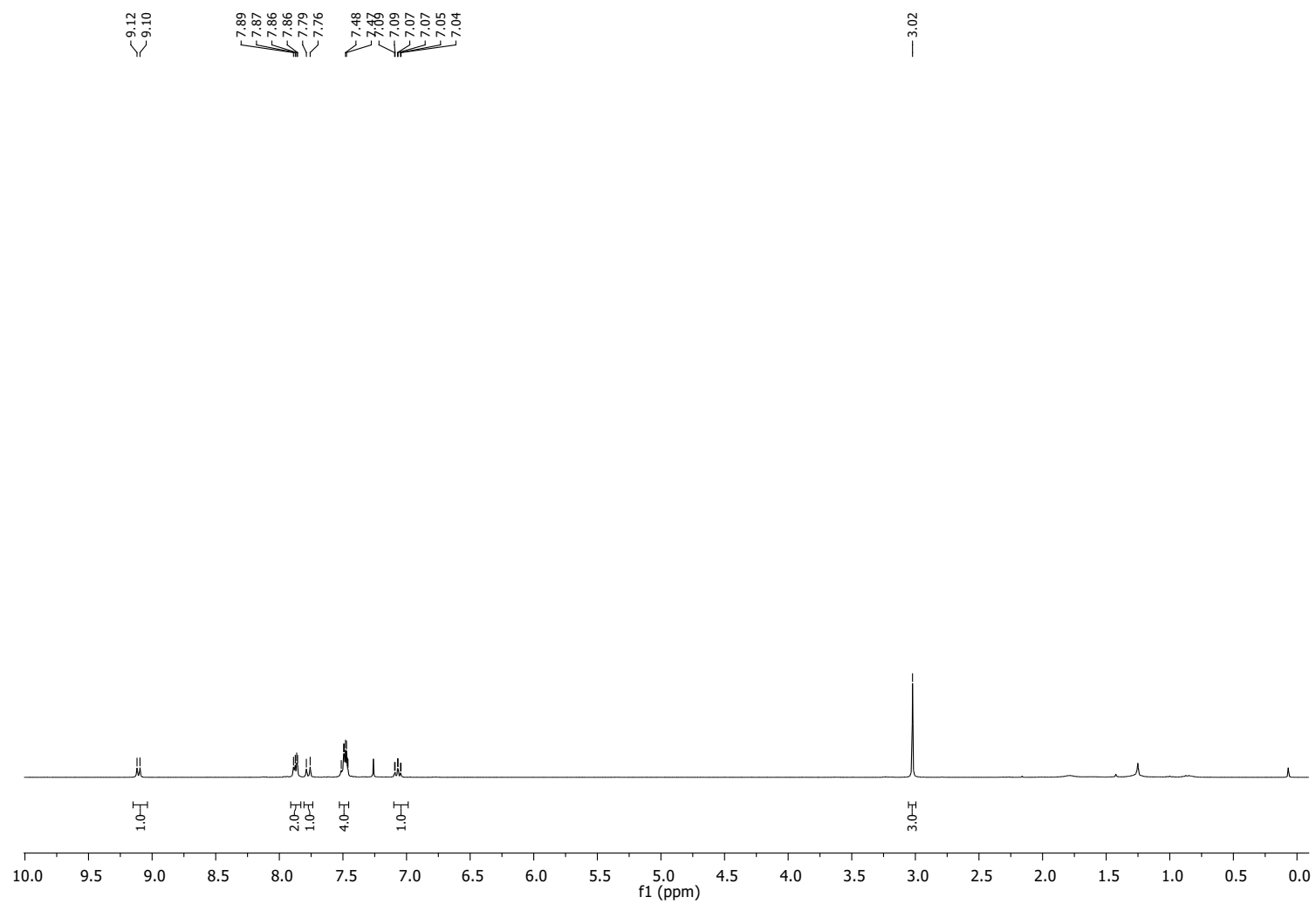
^{13}C NMR (CDCl₃, 75 MHz)



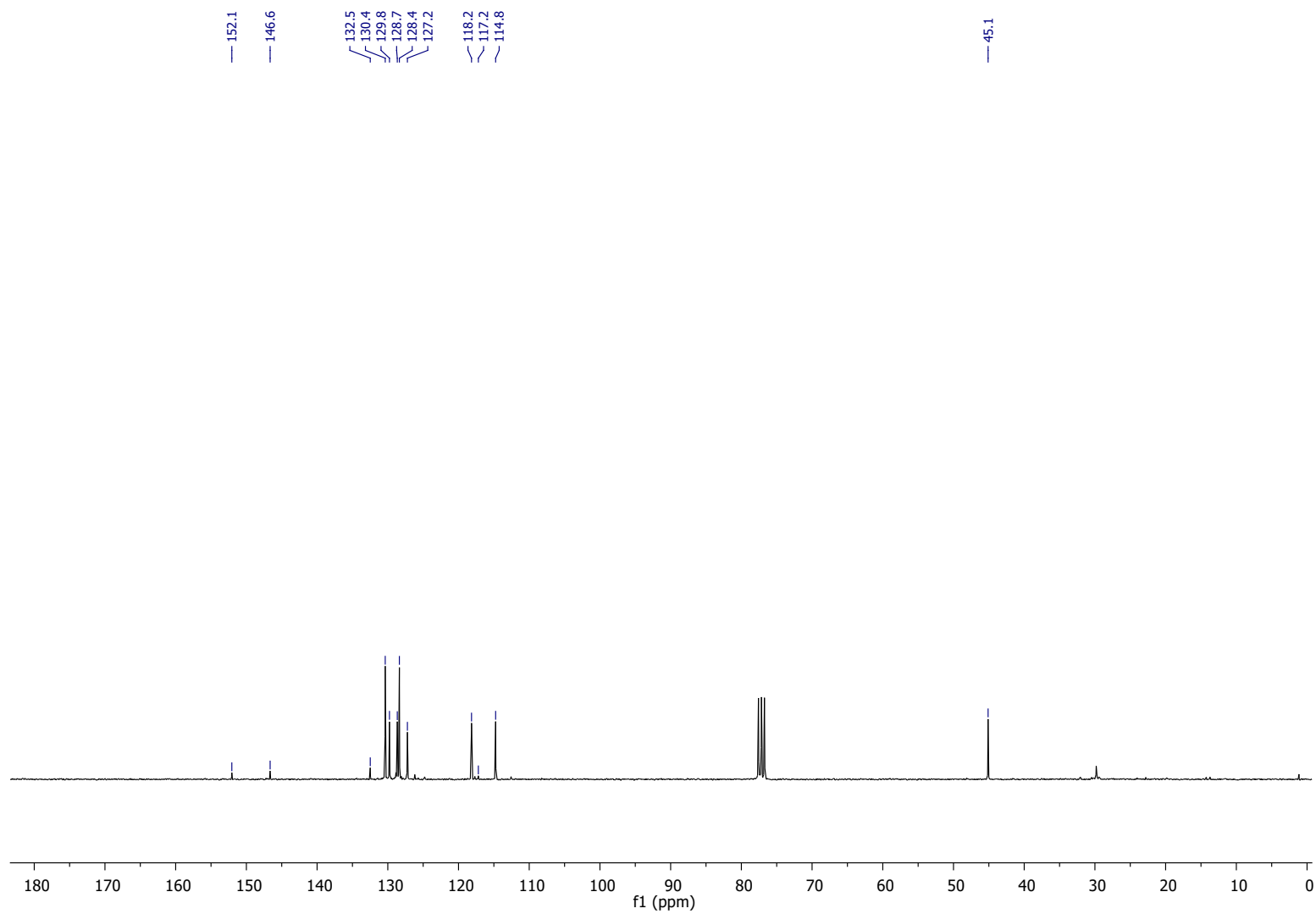
SI74

3-(methylsulfonyl)-2-phenylimidazo[1,2-*a*]pyridine **3aj**

¹H NMR (CDCl₃, 300 MHz)



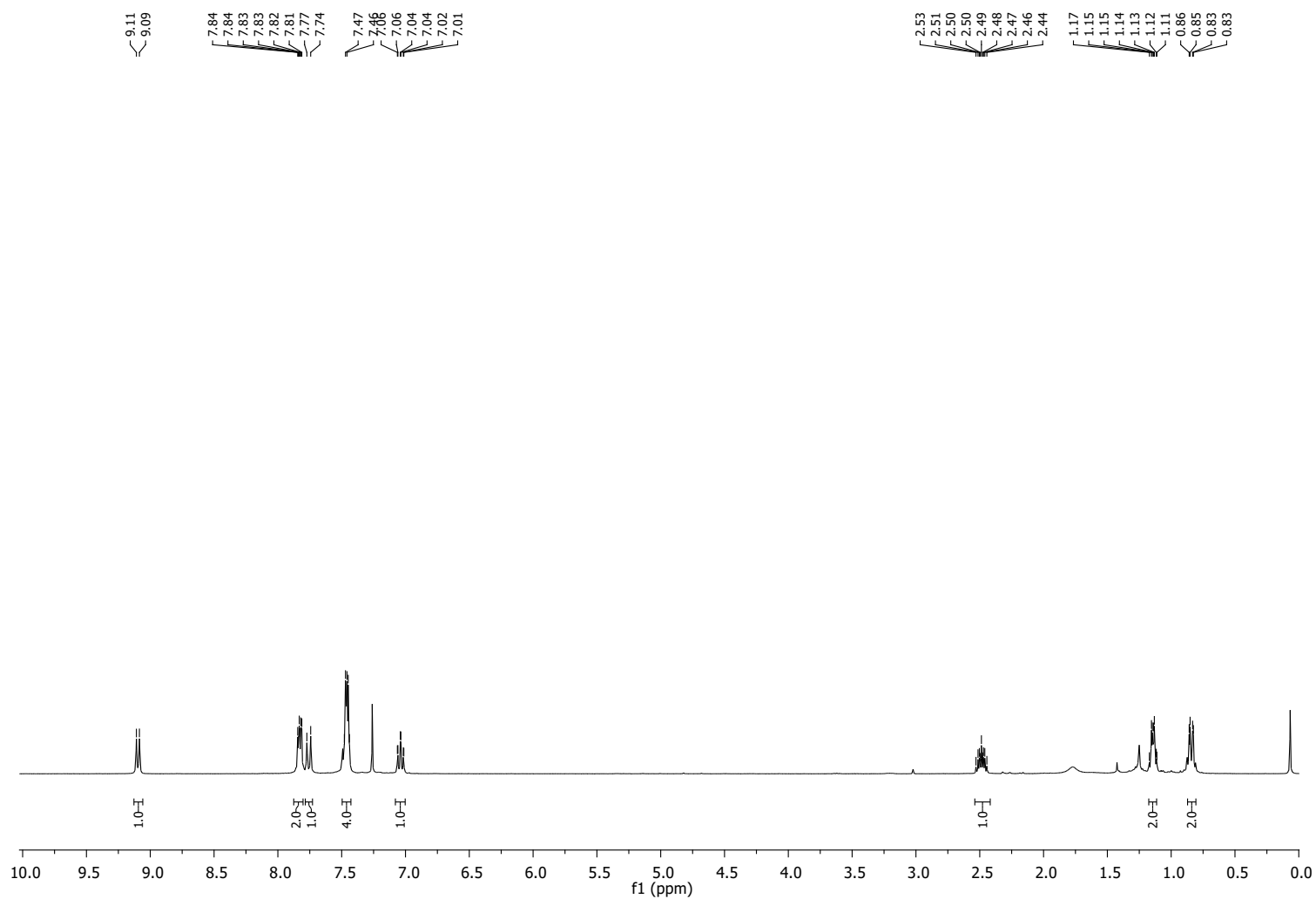
^{13}C NMR (CDCl₃, 75 MHz)



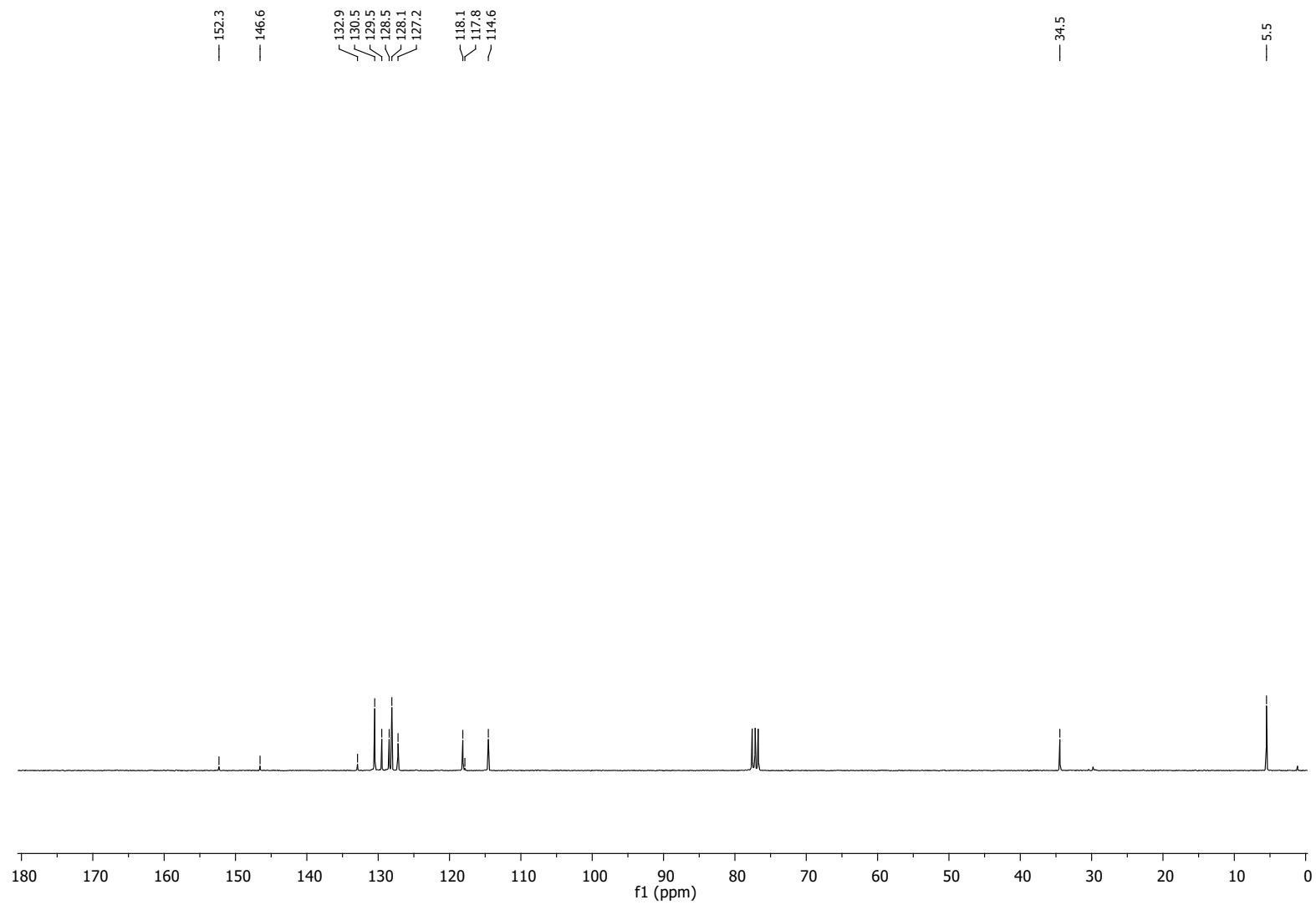
SI76

3-(cyclopropylsulfonyl)-2-phenylimidazo[1,2-*a*]pyridine **3ak**

¹H NMR (CDCl₃, 300 MHz)

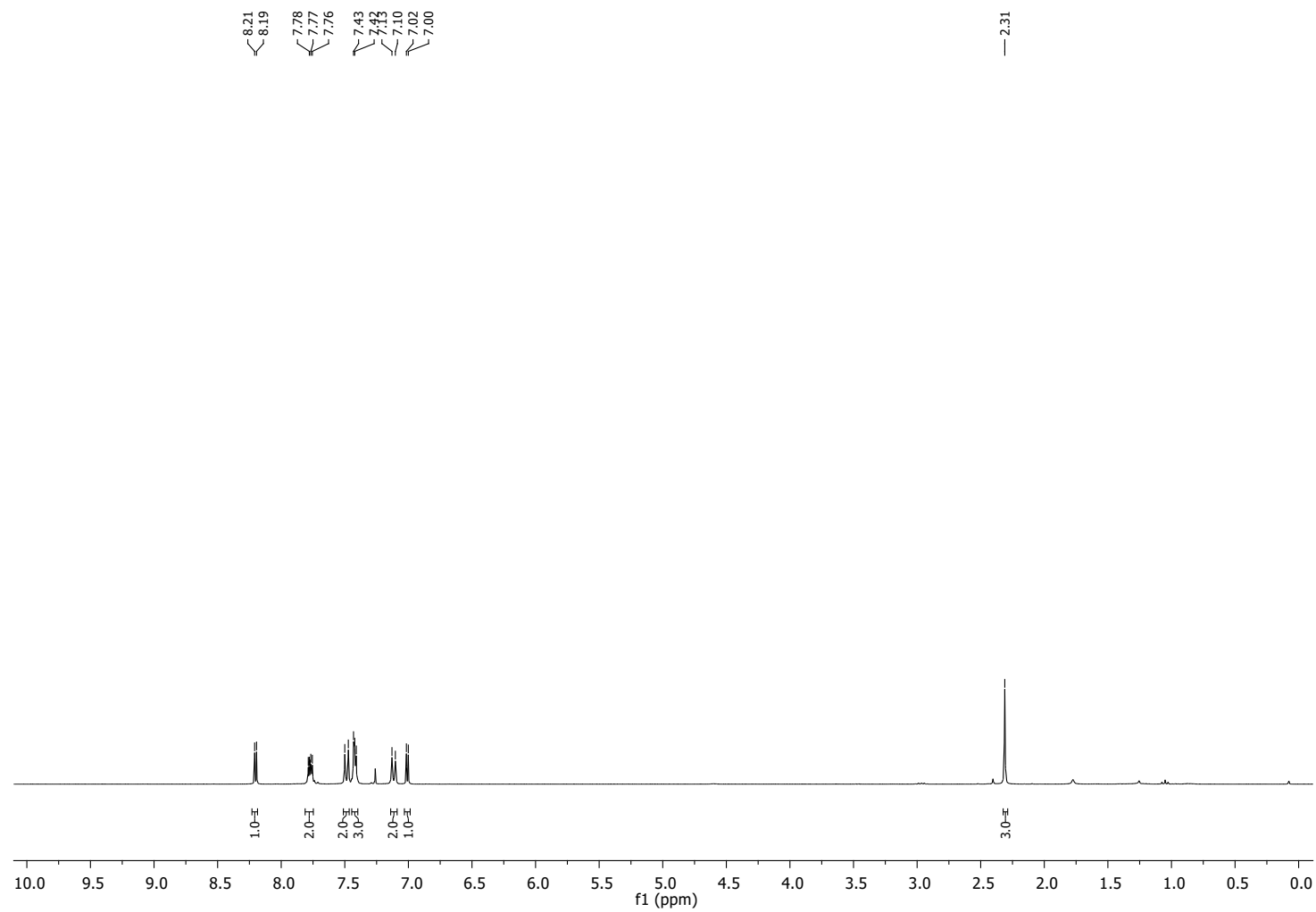


^{13}C NMR (CDCl₃, 75 MHz)

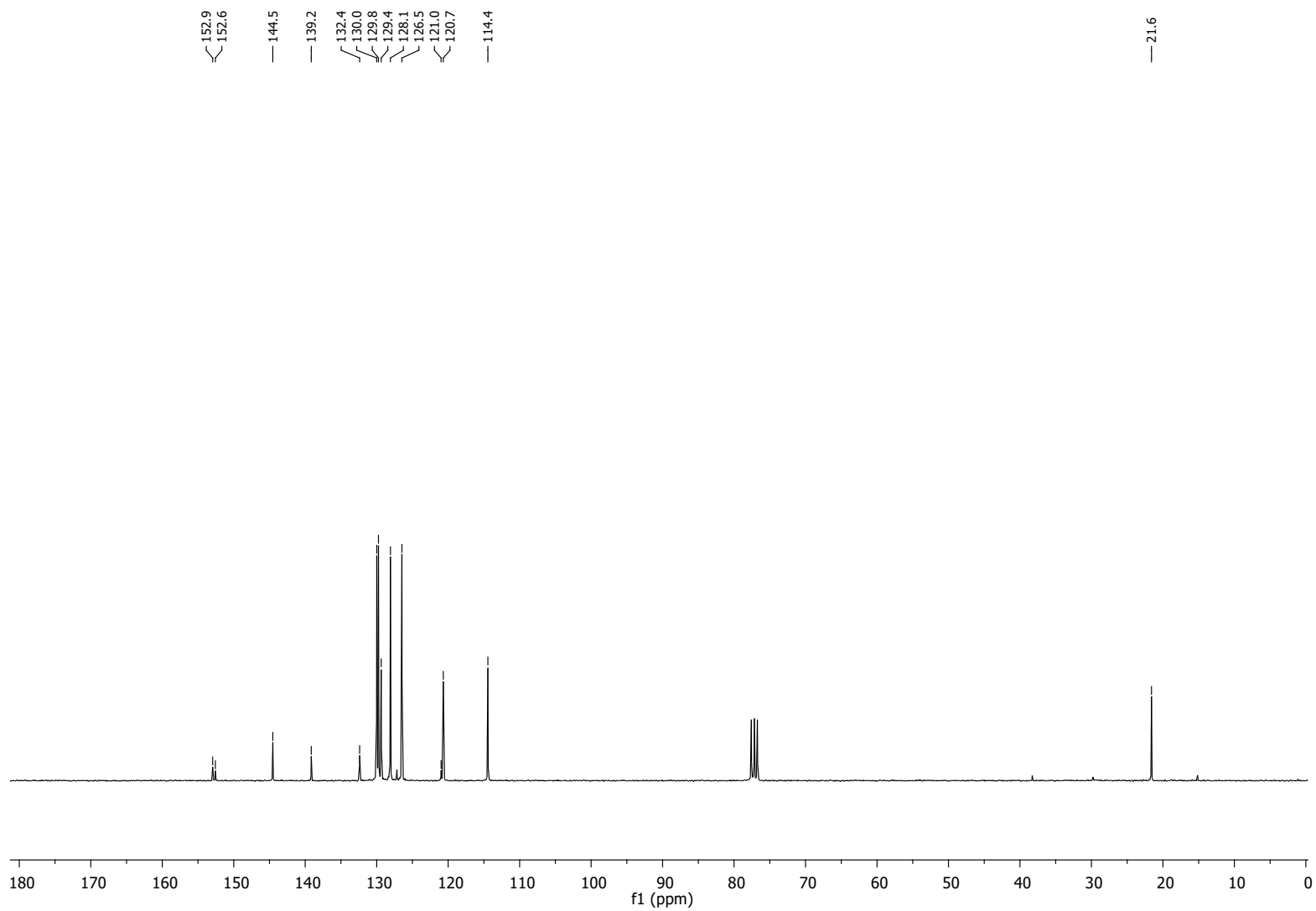


6-phenyl-5-tosylimidazo[2,1-*b*]thiazole **5a**

¹H NMR (CDCl₃, 300 MHz)

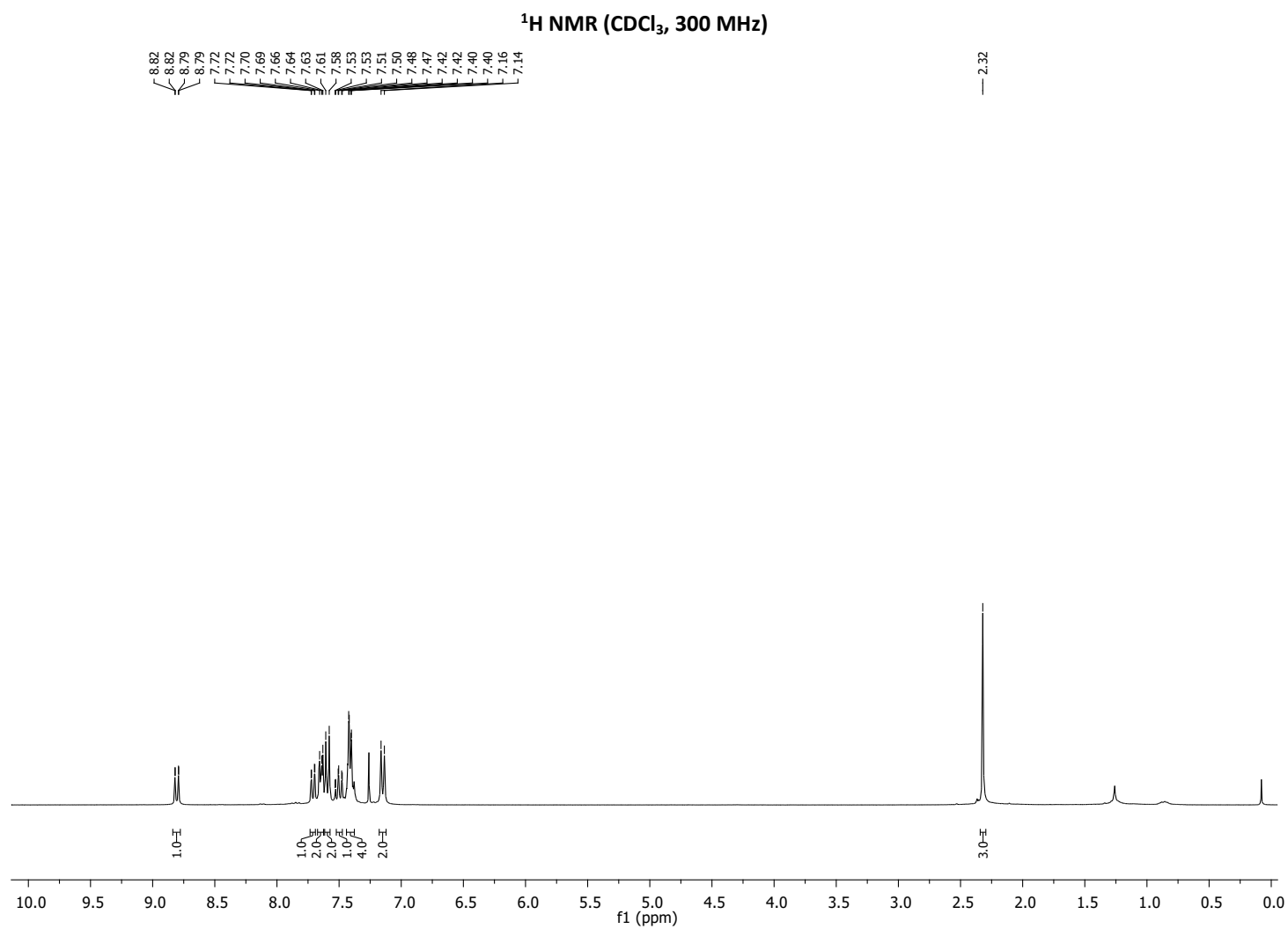


^{13}C NMR (CDCl₃, 75 MHz)

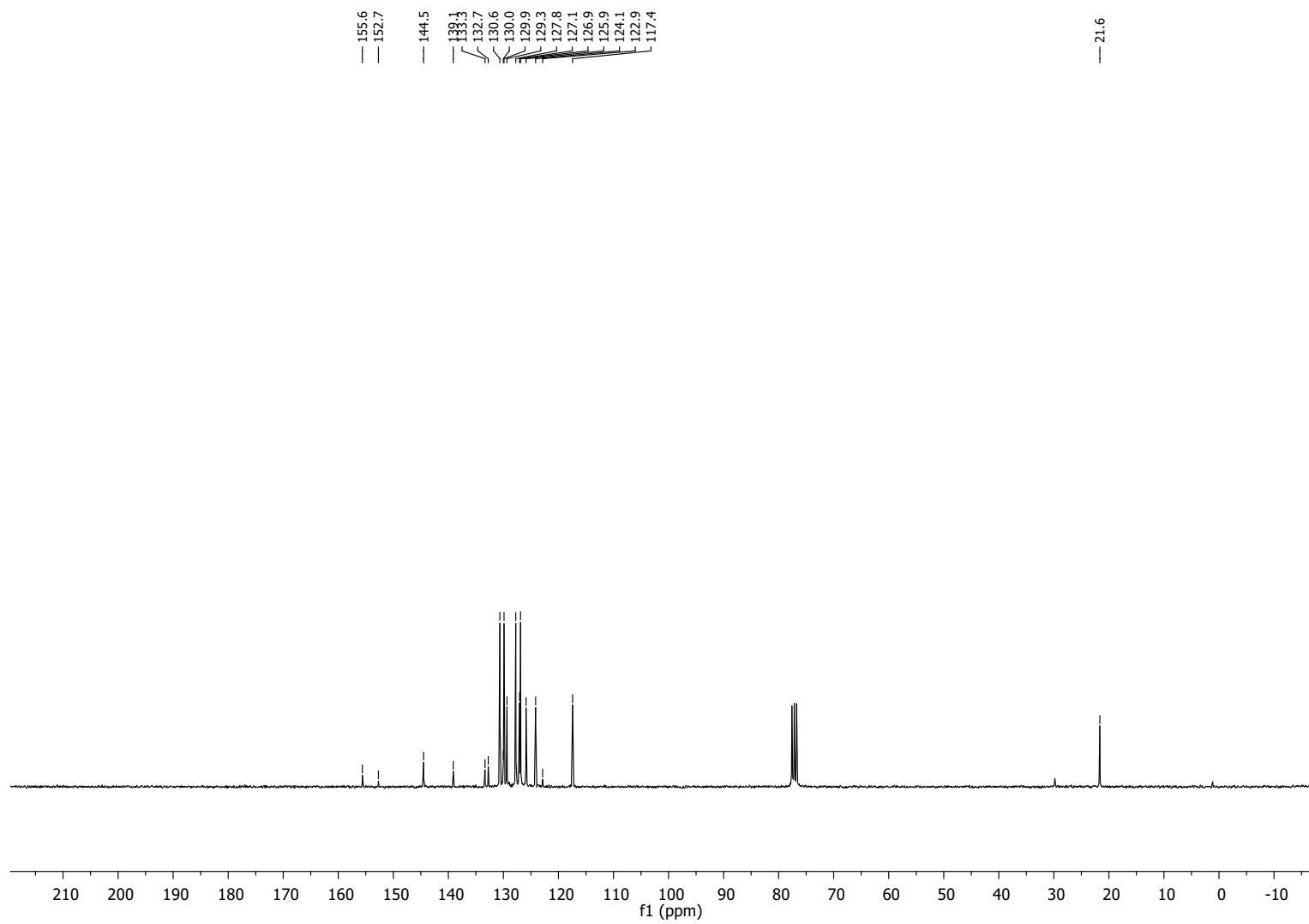


SI80

2-phenyl-3-tosylbenzo[d]imidazo[2,1-b]thiazole **5b**

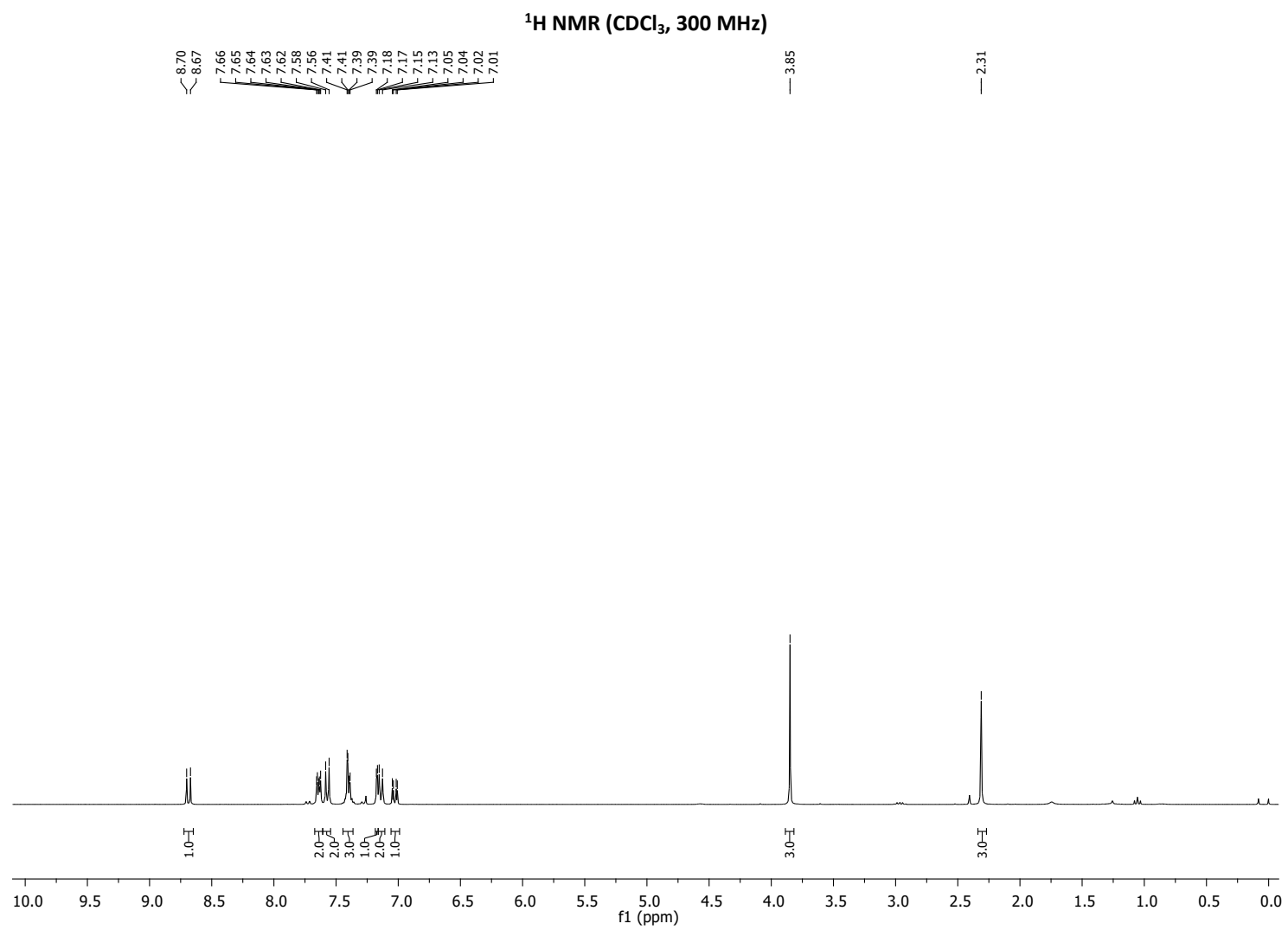


^{13}C NMR (CDCl₃, 75 MHz)

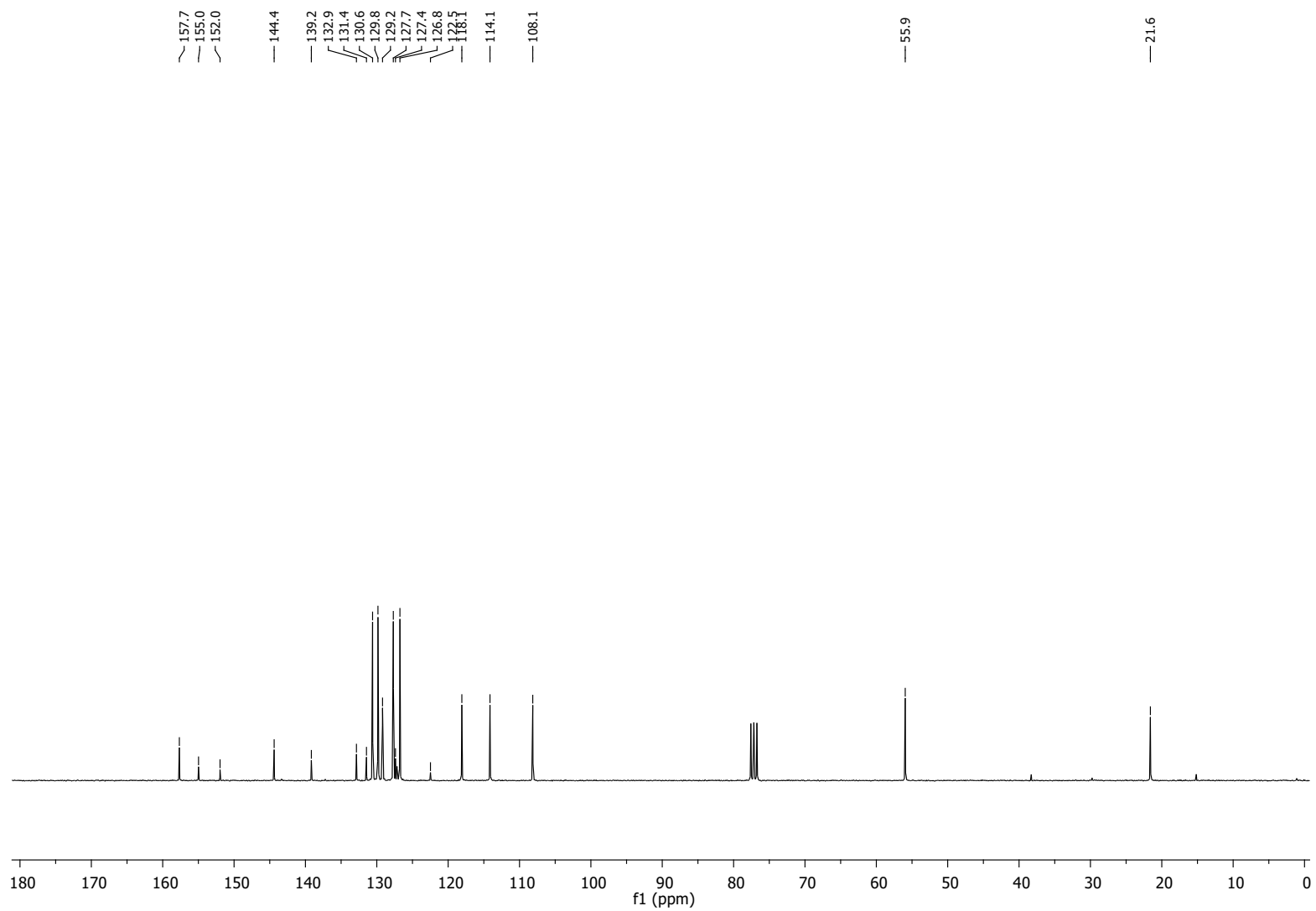


SI82

7-methoxy-2-phenyl-3-tosylbenzo[*d*]imidazo[2,1-*b*]thiazole **5c**

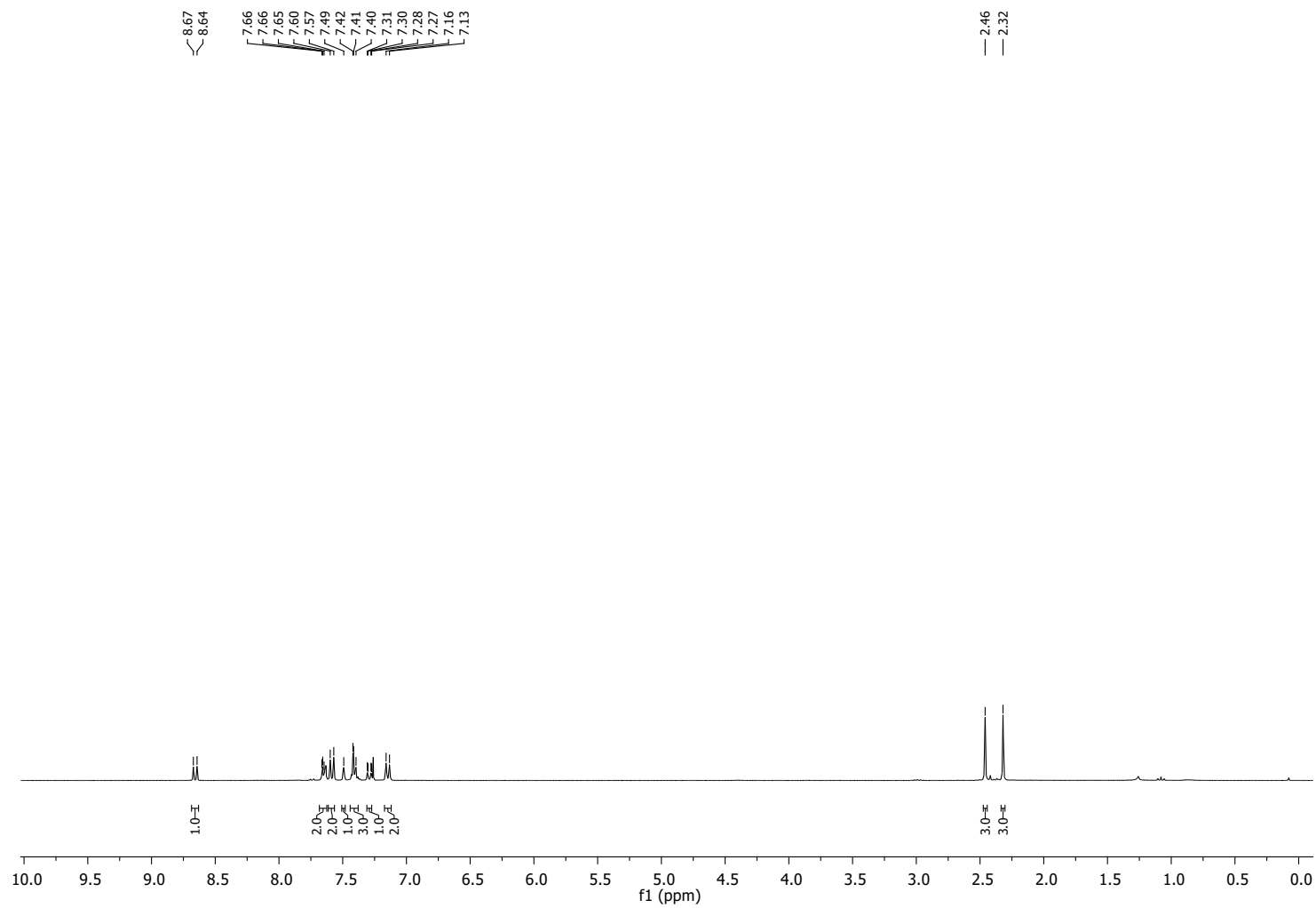


^{13}C NMR (CDCl₃, 75 MHz)

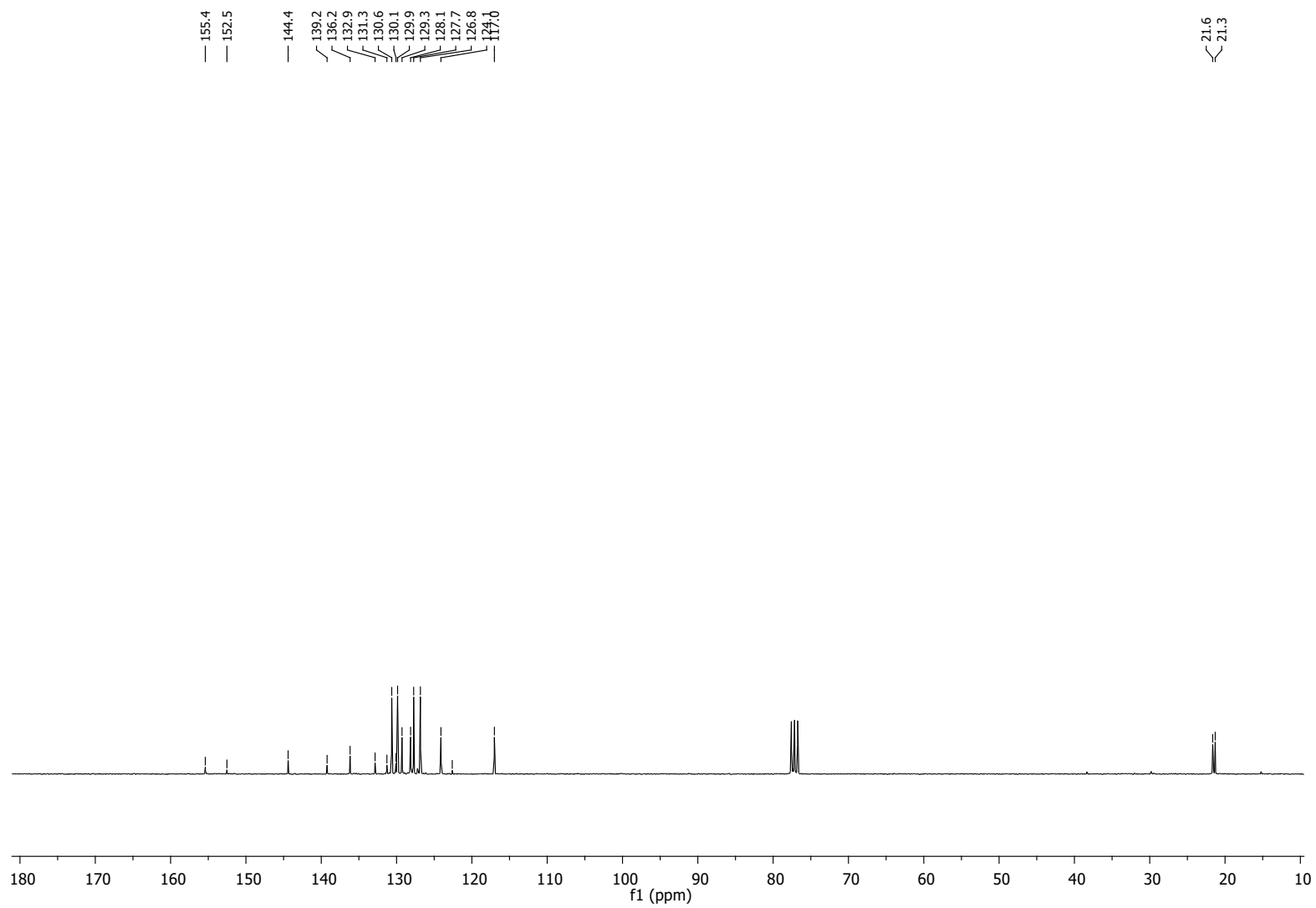


7-methyl-2-phenyl-3-(phenylsulfonyl)benzo[d]imidazo[2,1-b]thiazole **5d**

^1H NMR (CDCl_3 , 300 MHz)



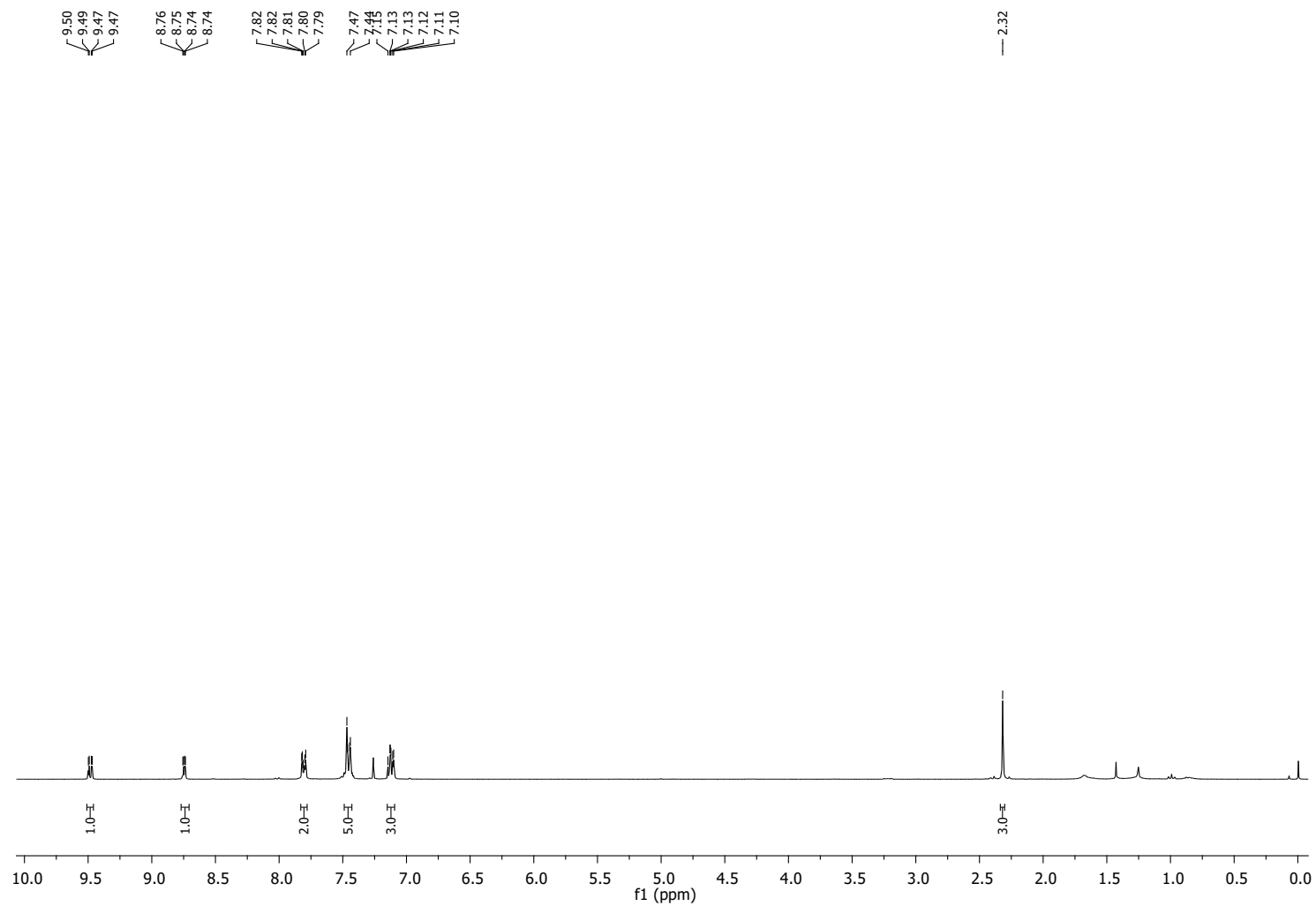
^{13}C NMR (CDCl₃, 75 MHz)



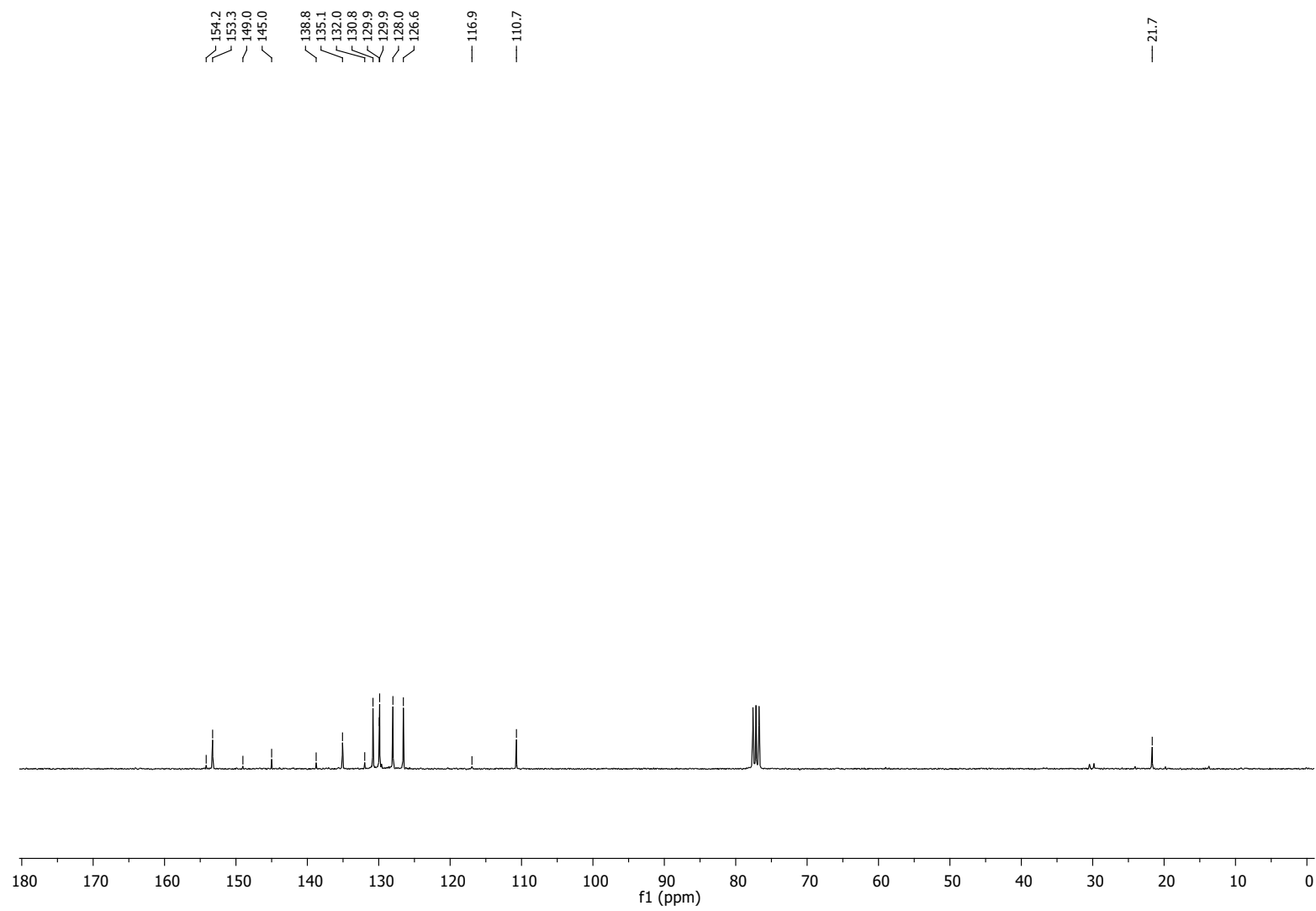
SI86

2-phenyl-3-tosylimidazo[1,2-*a*]pyrimidine **5e**

^1H NMR (CDCl_3 , 300 MHz)

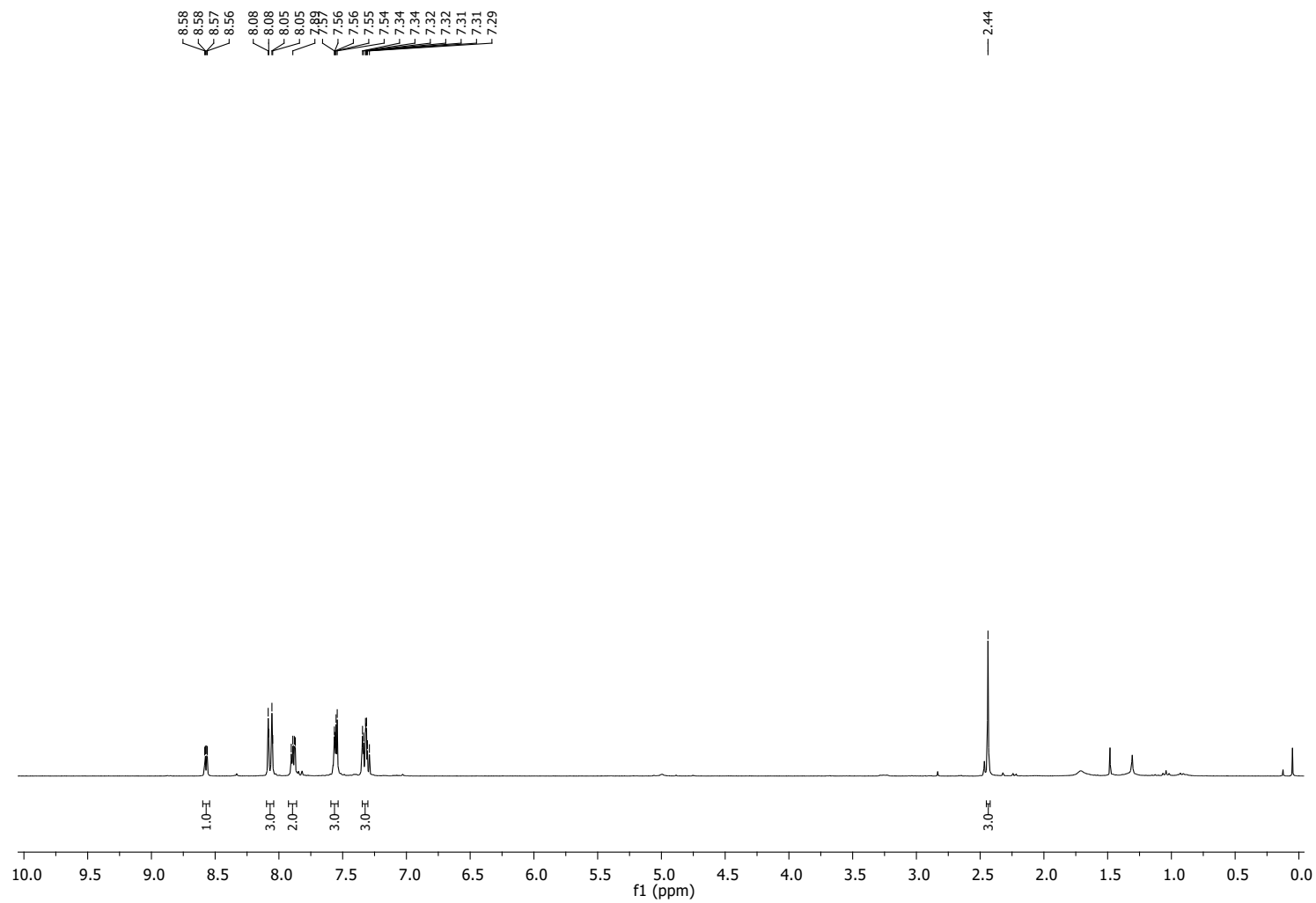


^{13}C NMR (CDCl₃, 75 MHz)

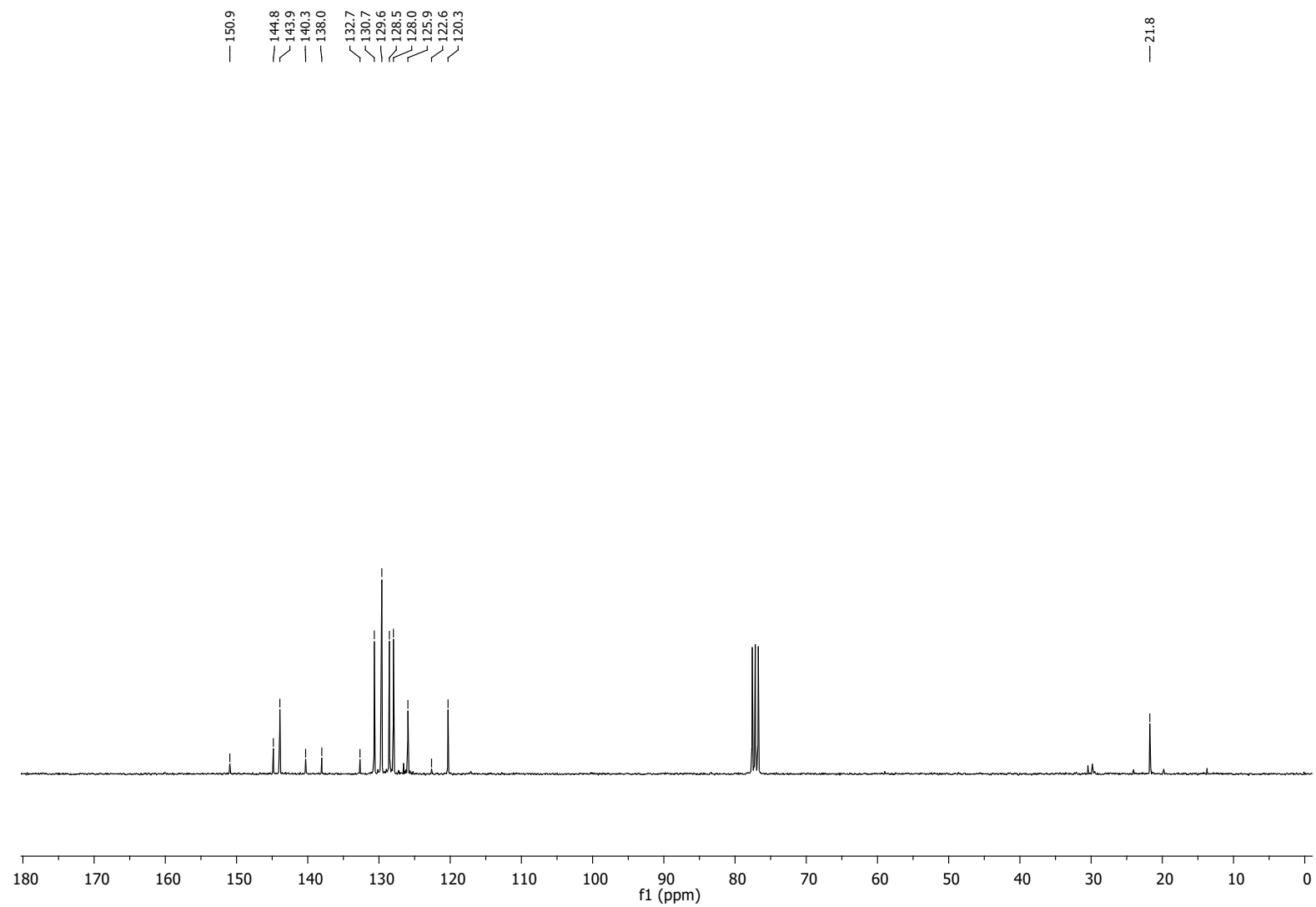


2-phenyl-3-tosylimidazo[1,2-*b*]pyridazine **5f**

¹H NMR (CDCl₃, 300 MHz)



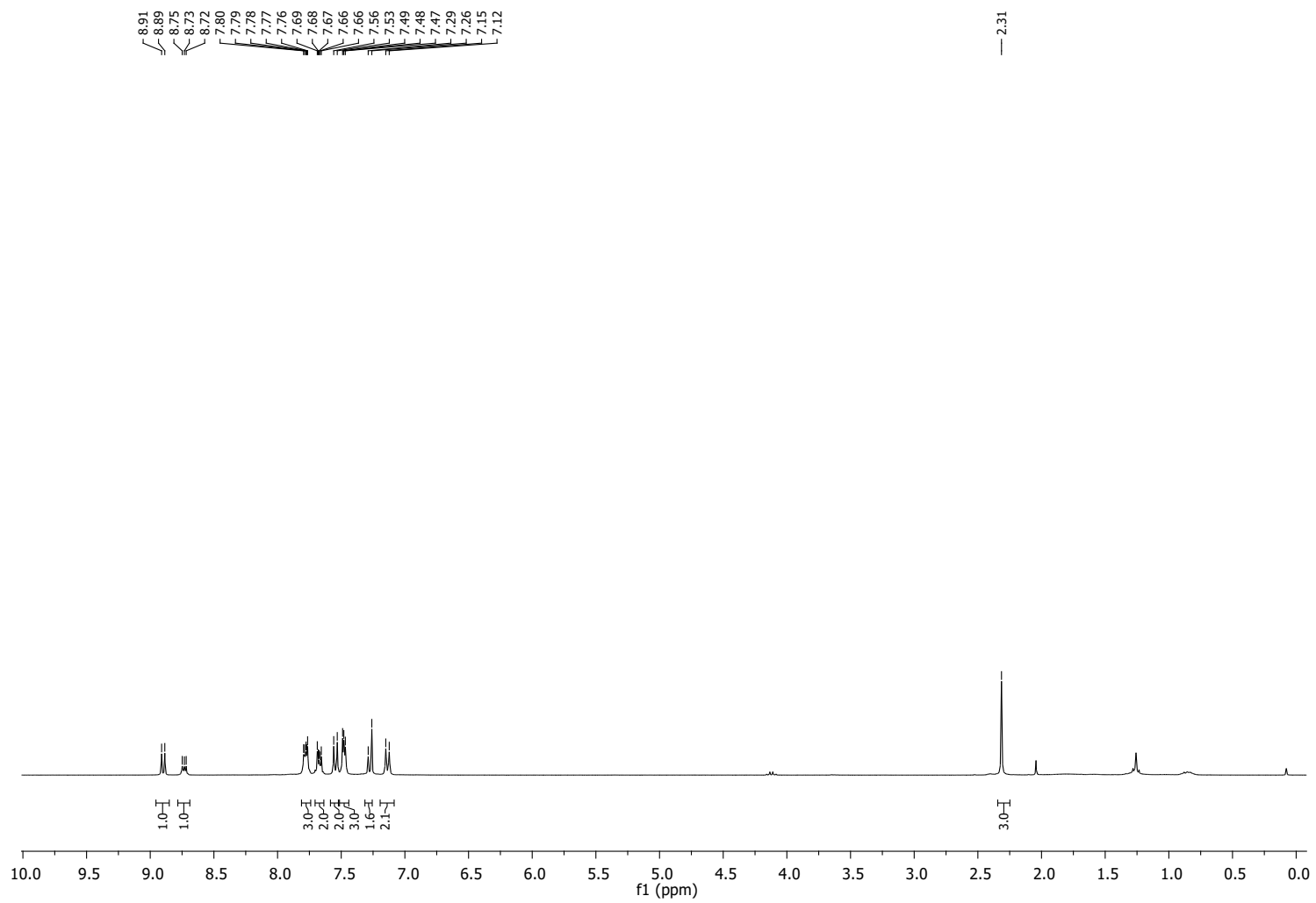
^{13}C NMR (CDCl₃, 75 MHz)



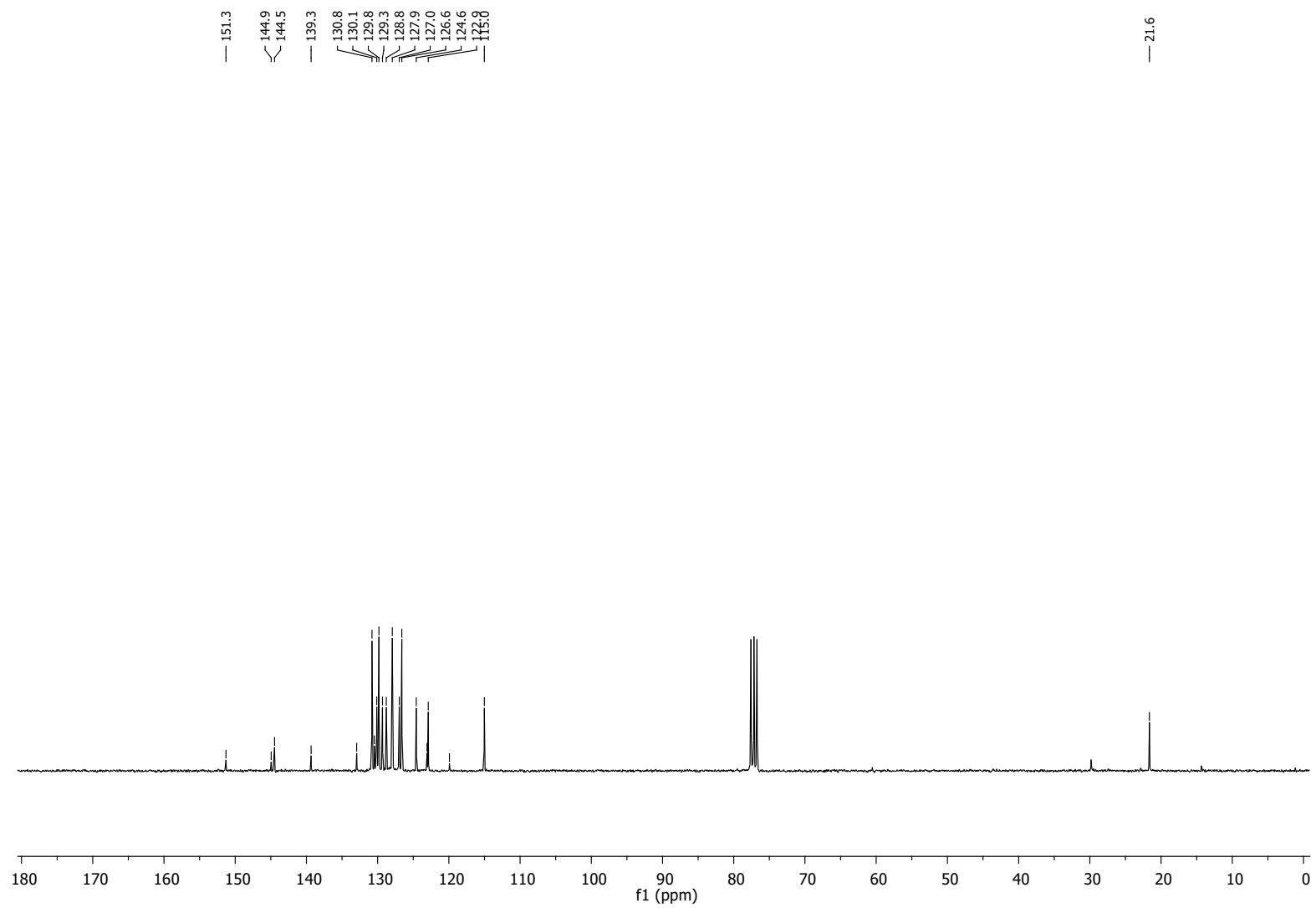
SI90

2-phenyl-3-tosylimidazo[2,1-*a*]isoquinoline **5g**

¹H NMR (CDCl₃, 300 MHz)

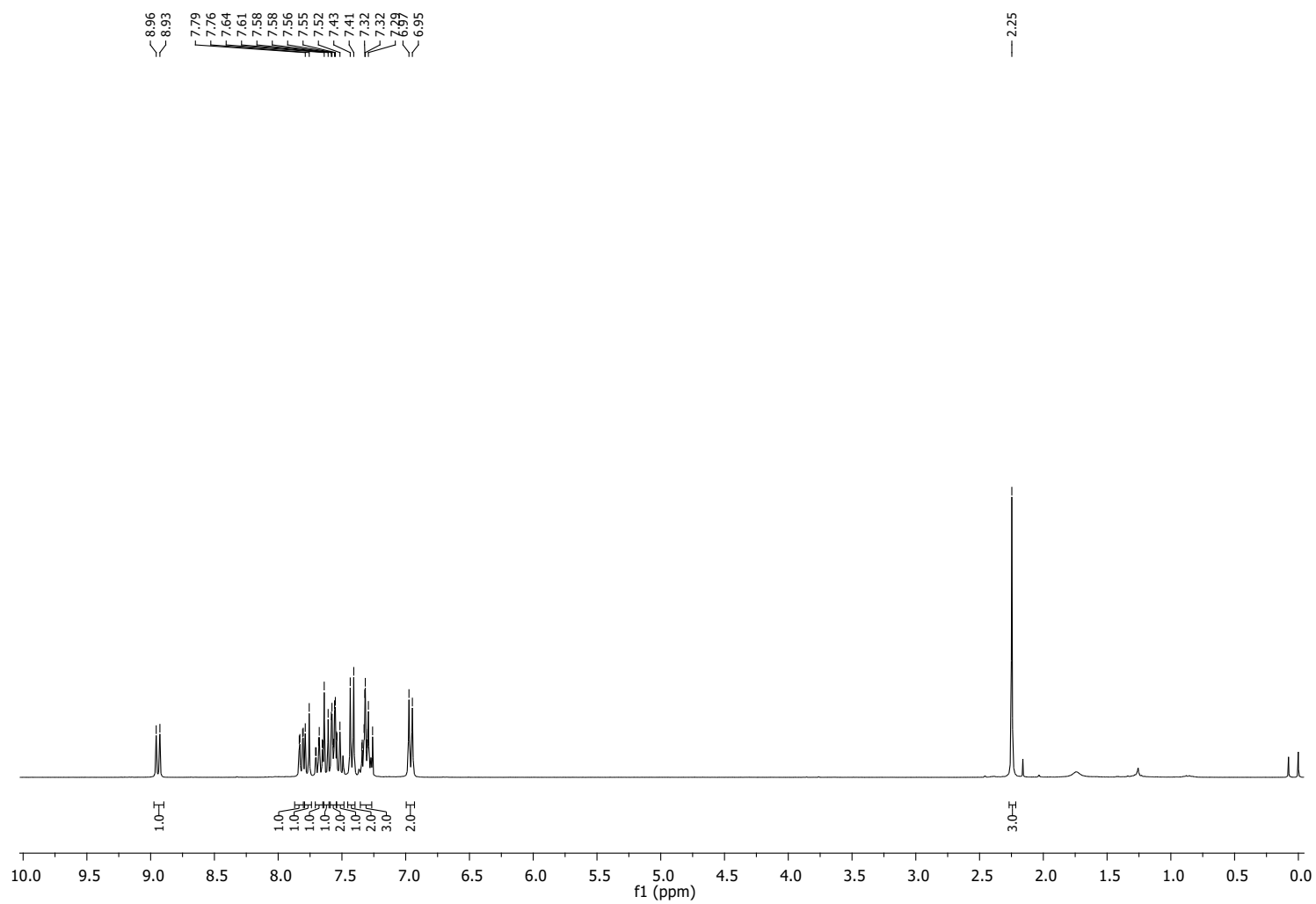


^{13}C NMR (CDCl₃, 75 MHz)

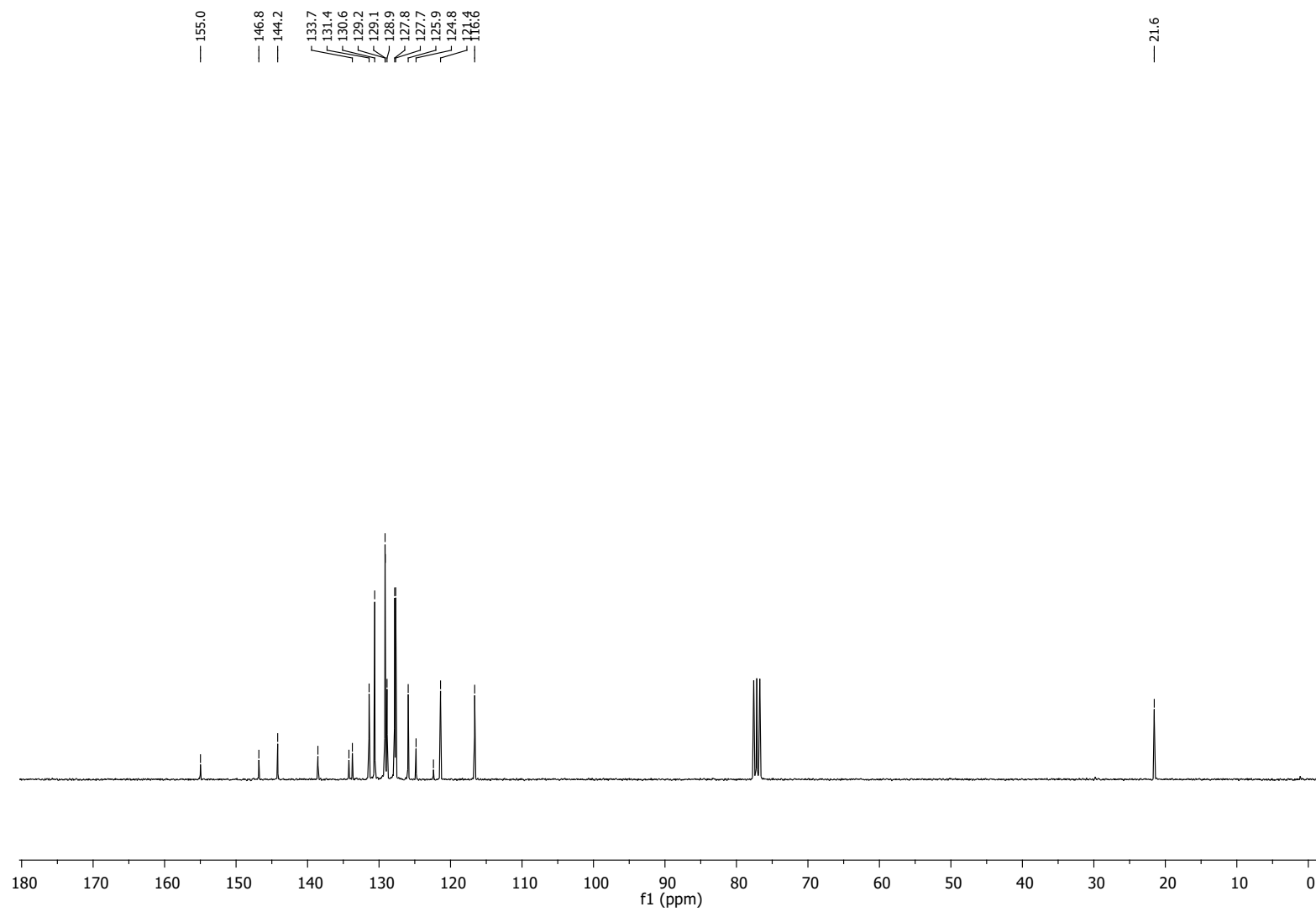


2-phenyl-1-tosylimidazo[1,2-*a*]quinoline **5h**

¹H NMR (CDCl₃, 300 MHz)



^{13}C NMR (CDCl₃, 75 MHz)



SI94

VI. References

1. (a) S.-Y. Takizawa, J.-I. Nishida, T. Tsuzuki, S. Tokito and Y. Yamashita, *Inorg. Chem.*, 2007, **46**, 4308-4319; (b) Y. Yu, Y. Yuan, H. Liu, M. He, M. Yang, P. Liu, B. Yu, X. Dong and A. Lei, *Chem. Commun.*, 2019, **55**, 1809-1812; (c) R. Aeluri, M. Alla, S. Polepalli and N. Jain, *Eur. J. Med. Chem.*, 2015, **100**, 18-23.
2. (a) M. Jiang, Y. Yuan, T. Wang, Y. Xiong, J. Li, H. Guo and A. Lei, *Chem. Commun.*, 2019, **55**, 13852-13855; (b) W. Kim, H. Y. Kim and K. Oh, *Org. Lett.*, 2020, **22**, 6319-6323.
3. L. Han, M. Huang, Y. Li, J. Zhang, Y. Zhu, J. K. Kim and Y. Wu, *Org. Chem. Front.*, 2021, **8**, 3110-3117.
4. J. Zhu, Z. Chen, M. He, D. Wang, L. Li, J. Qi, R. Shi and A. Lei, *Org. Chem. Front.*, 2021, **8**, 3815-3819.
5. Y.-J. Guo, S. Lu, L.-L. Tian, E.-L. Huang, X.-Q. Hao, X. Zhu, T. Shao and M.-P. Song, *J. Org. Chem.*, 2018, **83**, 338-349.