**Electronic Supplementary Information**

# Kolbe Radical-Initiated Electrochemical Desulfurization of Thioamides under Aerobic Conditions

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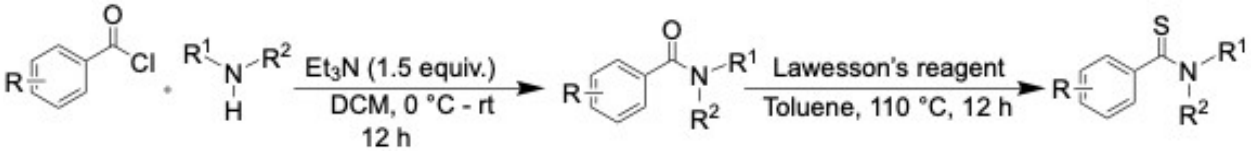
General information

Unless specified otherwise, all reactions and experimental operations were performed under ambient air. Electrochemical experiments were conducted in undivided 5 mL electrochemical cells using pre-dried glassware whenever required. Platinum electrodes (99.9%) were sourced from ChemPur, Karlsruhe, Germany, and reticulated vitreous carbon (RVC) electrodes were obtained from SGL Carbon, Wiesbaden, Germany. Electrocatalytic studies were carried out on a GAMRY Reference 600 potentiostat operated in constant current mode. Reported yields correspond to isolated and spectroscopically pure products, confirmed by $^{1}$H NMR analysis. All solvents were used without further purification. Silica gel (60 Å, Chemtronica) was employed for column chromatography. Separations were achieved using a petroleum ether/acetone gradient, guided by TLC analysis on Aldrich silica gel 254 nm fluorescent indicator aluminium sheets. Nuclear magnetic resonance (NMR) spectra were acquired using a Bruker 400 MHz spectrometer. $^{1}$H NMR chemical shifts are reported in parts per million (ppm) and referenced to the residual signal of chloroform-d (δ = 7.26 ppm), unless specified otherwise. $^{13}$C NMR spectra are likewise reported in ppm and referenced to the central peak of chloroform-d (δ = 77.23 ppm) with proton decoupling applied, unless noted differently.

General procedure for the synthesis of thioamides




Thioamide synthesis was carried out following the procedure reported by Michal Szostak.[1] The structures of the synthesized thioamides were confirmed by comparison with the previously reported spectral data.

General procedure for the desulfurization of thioamides

A 10 mL undivided electrochemical cell equipped with a reticulated vitreous carbon (RVC) anode (2 × 2 × 0.5 cm) and a platinum plate cathode (2 × 2 cm) was charged with thioamide **1** (0.050 g, 0.26 mmol), isobutyric acid (47 µL, 0.52 mmol, 2.0 equiv), triethylamine (18 µL, 0.13 mmol, 0.5 equiv), and $nBu_4NBF_4$ (0.17 g, 0.52 mmol, 2.0 equiv) in a mixture of 2,2,2-trifluoroethanol (TFE, 48 µL, 0.65 mmol) and 1,2-dichloroethane (1,2-DCE, 3.0 mL). The cell was subjected to constant current electrolysis (10 mA) under ambient air at room temperature for 20 h. Upon completion, the electrodes were removed and rinsed with acetone (3 × 10 mL). The combined organic layers were concentrated under reduced pressure, and the crude residue was purified by column chromatography on silica gel (eluent: Petroleum ether/Acetone) to afford the corresponding amide **2** as a pure product (0.045 g, 98% yield).

General procedure for the desulfurization of thioamides at gram scale

An undivided electrochemical cell (40 mL) equipped with a reticulated vitreous carbon (RVC) anode (4 × 4 × 0.5 cm) and a platinum plate cathode (4 × 4 cm) was charged with *N,N*-diisopropylbenzothioamide (0.600 g, 2.71 mmol), isobutyric acid (469 µL, 5.42 mmol, 2.0 equiv), triethylamine (721 µL, 5.42 mmol, 2.0 equiv), and $nBu_4NBF_4$ (1.70 g, 5.42 mmol, 2.0 equiv) in 10 mL of 1,2-dichloroethane (DCE) containing 2,2,2-trifluoroethanol (372 µL, 5.02 mmol). The cell was subjected to constant current electrolysis (40 mA) under ambient air at room temperature for 20 h. After completion, the electrodes were removed and washed with acetone (3 × 10 mL). The combined organic layers were concentrated under reduced pressure, and the residue was purified by column chromatography on silica gel (eluent: Petroleum ether/Acetone gradient) to afford the corresponding amide as a brown liquid (0.541 g, 97% yield).

Electrochemical cell set up

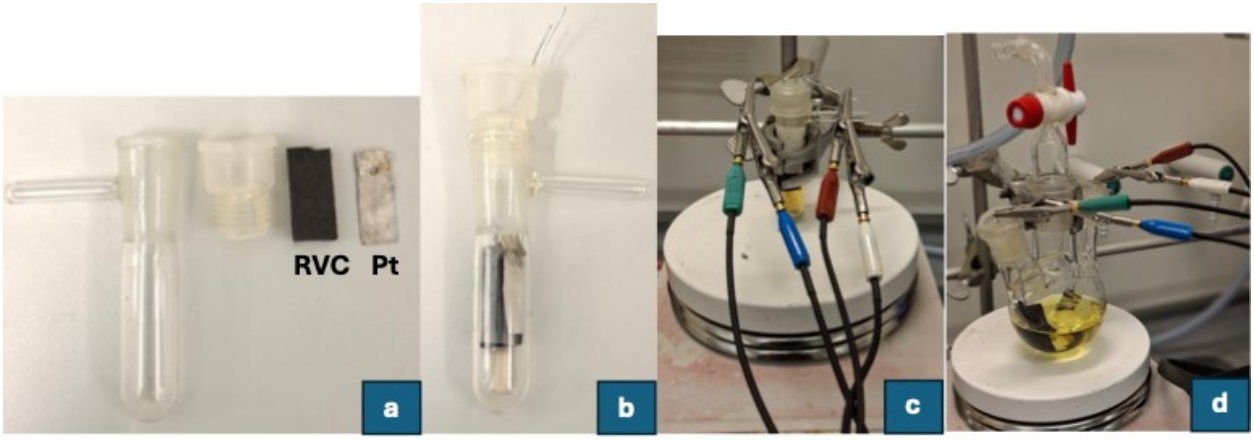



Figure S1: a) Electrode and glass tube with septa; b) Cell setup before adding reagents; c) Cell setup after reagents addition.; d) Gram scale reaction set up. ***Note:*** *The reaction required continuous exposure to air throughout its course. When we performed the reaction in a vessel with complete removal of air, no conversion was observed.*

## General procedure for CV studies

Cyclic voltammetry (CV) experiments were performed in a 10 mL single-compartment cell equipped with a RVC working electrode, a platinum wire counter electrode, and a silver wire reference electrode, using 0.10 M $nBu_4NBF_4$ in DCE/TFE as the supporting electrolyte (Fig S2). Solutions were prepared by pipetting the appropriate volumes of reagents into the cell and adjusting to a final volume of 10 mL with electrolyte. For blank experiments, the cell contained only supporting electrolyte, which was sparged with $N_2$ for 20 minutes before recording the voltammogram. Carboxylate solutions were prepared by mixing $iBuCO_2H$ and $Et_3N$ in a 1:1 ratio (10 mM final concentration) prior to dilution with electrolyte to give a clear Kolbe wave under $N_2$. Thioamide solutions (5 mM final) were prepared analogously, and mixed solutions were formulated to mimic the reaction stoichiometry (5 mM thioamide, 10 mM $iBuCO_2H$, 0.5 equiv $Et_3N$) or, in control experiments, to fully deprotonate the acid (1:1 acid/base ratio). After sparging with $N_2$, CV traces were recorded at constant scan rate.

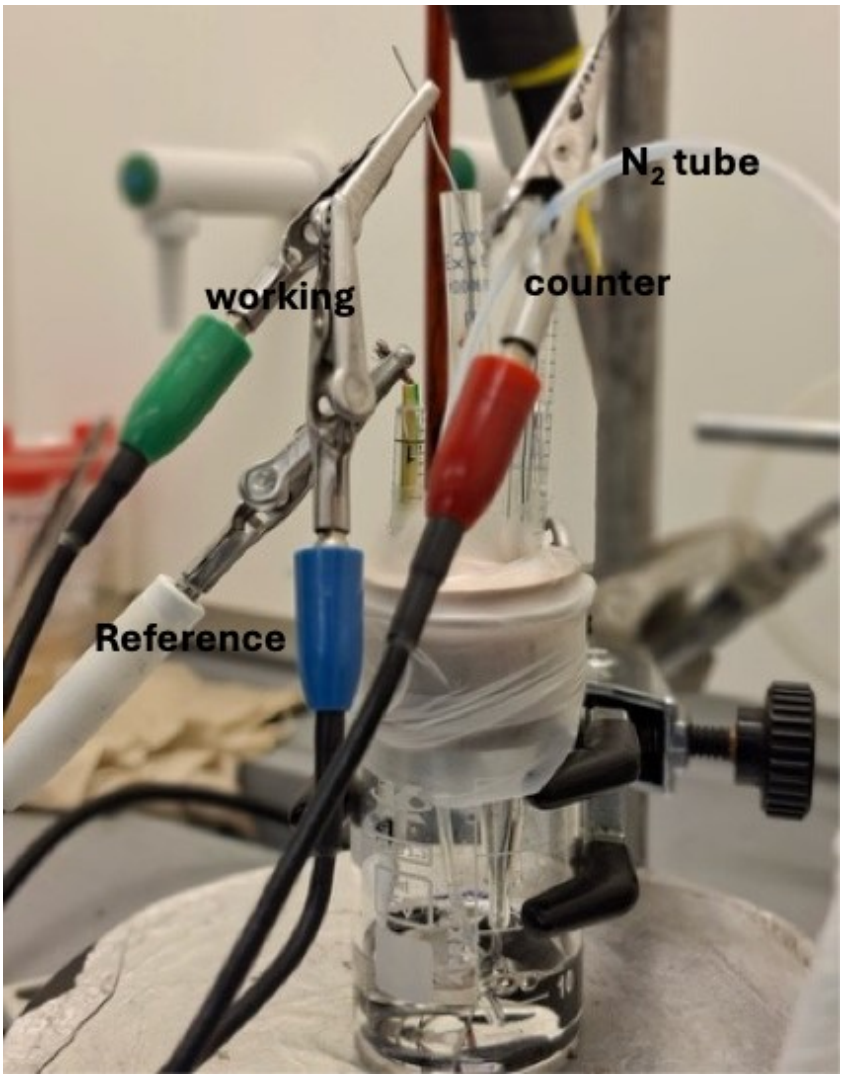



*Figure S2: Cyclic voltammetry (CV) experiment*

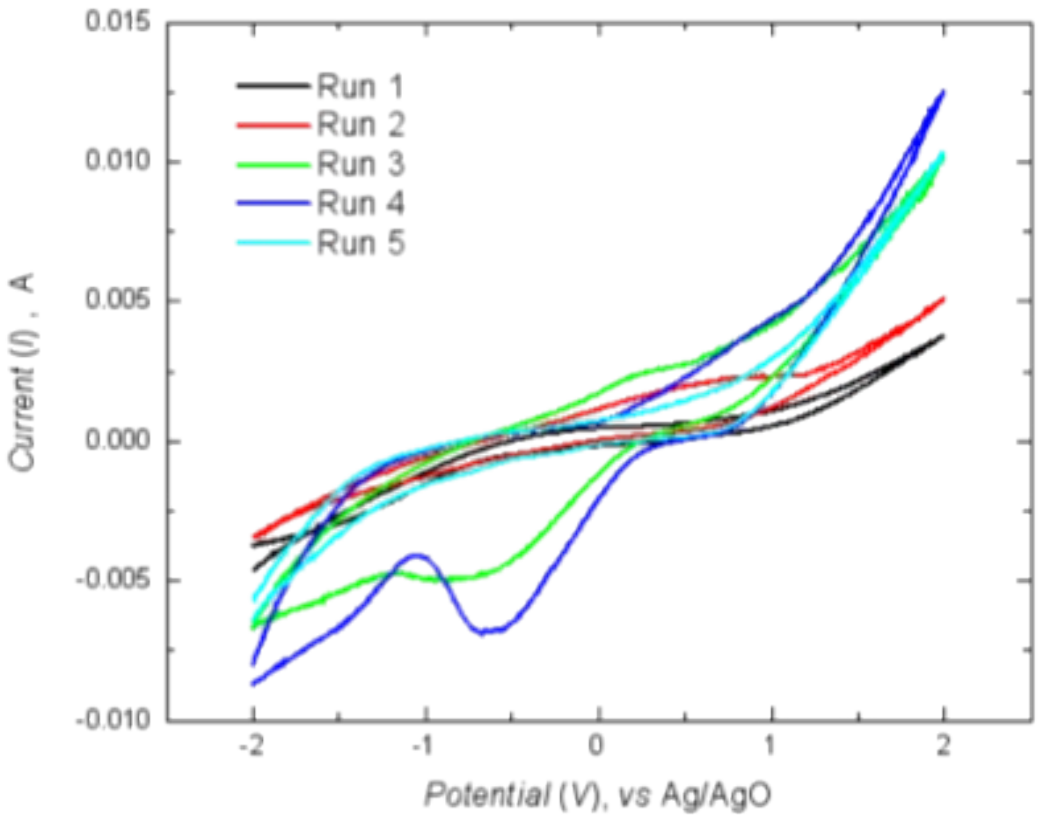
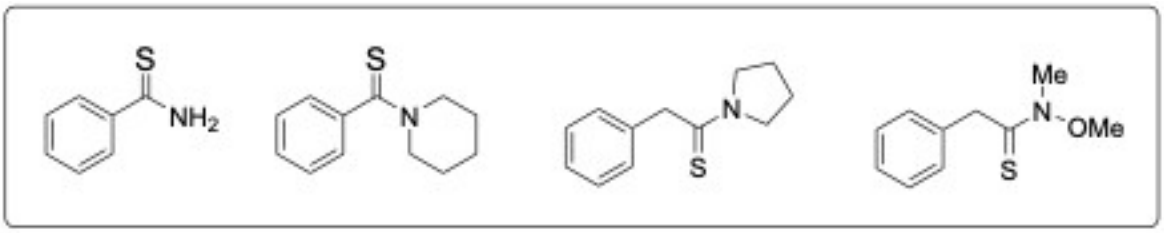


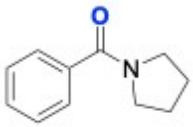

Fig. S3: Cyclic voltammograms recorded under identical conditions (10 mL electrolyte, RVC working electrode, Pt counter, Ag/AgO reference) comparing: (Run 1) blank electrolyte; (Run 2) 10 mM $iPrCO_2^-$; (Run 3) 5 mM thioamide; (Run 4 & 5) thioamide/carboxylate mixtures with partial (0.5 equiv.) or complete (1 equiv.) neutralization of $iPrCO_2H$.

The CV experiments (Fig. S3) support a Kolbe radical mediated mechanism, as the blank electrolyte showed no faradaic response (Run 1). $iPrCO_2^-$ alone displayed a weak anodic wave consistent with Kolbe oxidation (Run 2). Thioamide alone exhibited a modest anodic peak and a strong cathodic feature beyond –0.5 V (Run 3). W5hen thioamide and $iPrCO_2^-$ were combined (Run 4), the current response increased substantially, showing additional reductive features that point to coupling between the Kolbe radical and thioamide. Under fully basic conditions (Run 5), the disappearance of the intense downstream cathodic wave suggests that the extent of carboxylate formation and radical generation controls the overall electrochemical response, consistent with the pathway outlined in scheme 4.

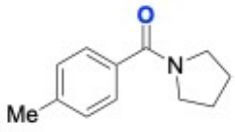
List of unsuccessful substrates



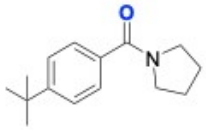

NMR data



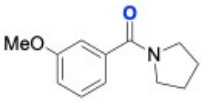
**Phenyl(pyrrolidin-1-yl)methanone (2a):** Colorless liquid, 45 mg. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.45 – 7.41 (m, 2H), 7.35 – 7.29 (m, 3H), 3.57 (t, *J* = 6.9 Hz, 2H), 3.35 (t, *J* = 6.6 Hz, 2H), 1.88 (p, *J* = 6.7 Hz, 2H), 1.79 (p, *J* = 6.7 Hz, 2H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 168.7, 136.1, 128.7, 127.2, 126.0, 48.6, 45.1, 25.3, 23.4. The spectral data were identical to those reported in the literature.[2]



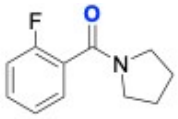
**Pyrrolidin-1-yl(*p*-tolyl)methanone (2b):** Brown liquid, 47 mg. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.35 (d, *J* = 8.1 Hz, 2H), 7.11 (d, *J* = 7.9 Hz, 2H), 3.46 (d, *J* = 73.5 Hz, 4H), 2.29 (s, 3H), 1.83 (d, *J* = 18.9 Hz, 4H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 169.9, 139.9, 134.1, 128.8, 127.2, 49.7, 46.2, 26.4, 24.4, 21.3. The spectral data were identical to those reported in the literature.[2]



**(4-(tert-butyl)phenyl)(pyrrolidin-1-yl)methanone (2c):** Brown liquid, 45 mg. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.39 (d, *J* = 8.5 Hz, 3H), 7.32 (d, *J* = 8.4 Hz, 3H), 3.64 – 3.34 (m, 4H), 1.85 (s, 4H), 1.24 (s, 9H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 170.0, 153.1, 133.9, 129.9, 127.0, 125.1, 49.7, 46.2, 34.8, 31.2, 26.3, 24.4. The spectral data were identical to those reported in the literature.[2]

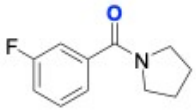


**(3-Methoxyphenyl)(pyrrolidin-1-yl)methanone (2d):** Brown liquid, 36 mg. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.27 – 7.18 (m, 1H), 7.03 – 6.95 (m, 4H), 6.87 (ddd, *J* = 8.3, 2.6, 1.0 Hz, 1H), 3.75 (s, 3H), 3.57 (t, *J* = 7.0 Hz, 2H), 3.35 (t, *J* = 6.6 Hz, 2H), 1.84 (dq, *J* = 29.4, 6.3 Hz, 3H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 169.48, 159.46, 138.57, 129.33, 119.24, 115.75, 112.38, 55.35, 49.59, 46.15, 26.37, 24.46. The spectral data were identical to those reported in the literature.[3]

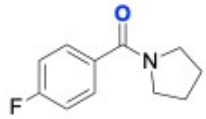


**(2-fluorophenyl)(pyrrolidin-1-yl)methanone (2e):** Brown liquid, 48 mg. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.37 – 7.27 (m, 2H), 7.12 (td, *J* = 7.5, 1.0 Hz, 1H), 7.05 – 6.98 (m, 1H), 3.58 (t, *J* = 7.0 Hz, 2H), 3.24 (t, *J* = 6.7 Hz, 2H), 1.94 – 1.86 (m, 2H), 1.86 – 1.77 (m, 2H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 165.1, 159.5, 157.0, 131.2 (d, *J* = 8 Hz), 131.1, 128.9 ((d, *J* = 4 Hz), 128.8, 125.8 (d, *J* = 8 Hz), 125.6, 124.5 ((d, *J* = 3 Hz),

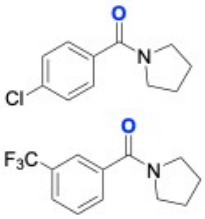
124.4, 115.9 (d, *J* = 21 Hz), 115.7, 47.8, 47.8, 45.8, 25.8, 24.5. **$^{19}$F NMR (376 MHz, $CDCl_3$)** δ -115.0, -115.1. The spectral data were identical to those reported in the literature.[4]



**(3-fluorophenyl)(pyrrolidin-1-yl)methanone (2f):** Light brown liquid, 53 mg. **$^{1}$H NMR (400 MHz, $CDCl_3$)** δ 7.30 (td, J = 7.9, 5.6 Hz, 1H), 7.22 (d, J = 6.9 Hz, 1H), 7.15 (dt, J = 9.1, 1.8 Hz, 1H), 7.07 – 7.00 (m, 1H), 3.56 (t, J = 6.9 Hz, 2H), 3.34 (t, J = 6.6 Hz, 2H), 1.90 (dq, J = 13.2, 6.2 Hz, 2H), 1.81 (p, J = 6.2 Hz, 2H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 168.2, 168.2, 163.6, 161.1, 139.2 (d, *J* = 7 Hz), 139.1, 130.0 (d, *J* = 8 Hz), 122.8 (d, *J* = 3 Hz), 122.7, 116.8 (d, *J* = 21 Hz), 116.6, 114.4 (d, *J* = 23 Hz), 114.2, 49.5, 46.2, 26.3, 24.4. **$^{19}$F NMR (376 MHz, $CDCl_3$)** δ -112.26, -112.27. The spectral data were identical to those reported in the literature.[3]

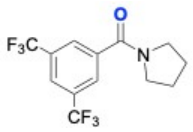


**(4-fluorophenyl)(pyrrolidin-1-yl)methanone (2g):** Brown liquid, 53 mg. **$^{1}$H NMR (400 MHz, $CDCl_3$)** δ 7.47 (dd, J = 8.7, 5.4 Hz, 2H), 7.01 (t, J = 8.7 Hz, 2H), 3.57 (t, J = 6.9 Hz, 2H), 3.36 (t, J = 6.6 Hz, 2H), 1.85 (dp, J = 32.5, 6.8 Hz, 4H). ). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 168.7, 164.7, 162.2, 133.3 ((d, *J* = 4 Hz), 133.2, 129.4 (d, *J* = 8 Hz), 115.3, 115.1 (d, *J* = 21 Hz), 49.7, 46.3, 26.4, 24.4. **$^{19}$F NMR (376 MHz, $CDCl_3$) δ -110.3.** The spectral data were identical to those reported in the literature. [5]

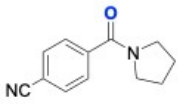


**(4-chlorophenyl)(pyrrolidin-1-yl)methanone (2h):** Pale yellow liquid, 33 mg. **$^{1}$H NMR (400 MHz, $CDCl_3$)** δ 7.39 (dd, *J* = 13.6, 8.5 Hz, 3H), 7.30 (d, *J* = 8.5 Hz, 2H), 3.57 (t, *J* = 6.9 Hz, 2H), 3.35 (t, *J* = 6.6 Hz, 2H), 1.86 (dp, *J* = 31.8, 6.7 Hz, 4H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 168.6, 135.8, 135.4, 131.5, 128.8, 128.6, 128.5, 77.3, 77.0, 76.7, 49.6, 46.3, 26.4, 24.4. The spectral data were identical to those reported in the literature.[3]

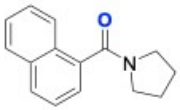
**Pyrrolidin-1-yl(3-(trifluoromethyl)phenyl)methanone (2i):** Brown liquid, 41 mg. **$^{1}$H NMR (400 MHz, $CDCl_3$)** δ 7.72 (s, 1H), 7.62 (dd, *J* = 13.7, 8.0 Hz, 2H), 7.46 (t, J = 7.7 Hz, 1H), 3.59 (t, *J* = 6.9 Hz, 2H), 3.34 (t, *J* = 6.6 Hz, 2H), 1.96 – 1.79 (m, 4H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 168.1, 137.9, 130.4, 130.4, 128.9, 126.5, 126.5, 124.1, 124.1, 49.5, 46.3, 26.4, 24.4. The spectral data were identical to those reported in the literature.[3]



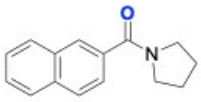
**(3,5-bis(trifluoromethyl)phenyl)(pyrrolidin-1-yl)methanone (2j): $^{1}$H NMR (400 MHz, $CDCl_3$)** δ 7.92 (d, *J* = 1.8 Hz, 2H), 7.86 (s, 1H), 3.60 (t, *J* = 6.9 Hz, 2H), 3.35 (t, *J* = 6.5 Hz, 2H), 1.99 – 1.83 (m, 4H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 166.4, 139.0, 132.3, 132.0, 131.7, 131.3, 127.5, 127.5, 124.3, 123.5, 123.5, 123.5, 121.6, 49.5, 46.5, 26.4, 24.3. **$^{19}$F NMR (376 MHz, $CDCl_3$)** δ -63.0. The spectral data were identical to those reported in the literature.[7]



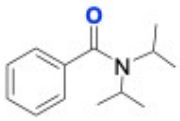
**4-(pyrrolidine-1-carbonyl)benzonitrile (2k):** Brown liquid, 39 mg. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.64 (d, J = 8.4 Hz, 2H), 7.55 (d, J = 8.3 Hz, 2H), 3.58 (t, J = 6.9 Hz, 2H), 3.30 (t, J = 6.6 Hz, 2H), 1.96 – 1.80 (m, 4H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 167.6, 141.3, 132.2, 127.8, 118.2, 113.5, 49.4, 46.3, 26.3, 24.3. The spectral data were identical to those reported in the literature.[6]



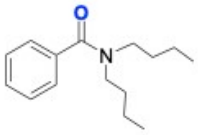
**Naphthalen-1-yl(pyrrolidin-1-yl)methanone (2m):** Brown liquid, 45 mg. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.81 – 7.79 (m, 1H), 7.78 (s, 1H), 7.45 – 7.41 (m, 1H), 7.41 – 7.36 (m, 1H), 3.72 (t, J = 7.1 Hz, 2H), 3.05 (t, J = 6.7 Hz, 2H), 1.92 (p, J = 6.9 Hz, 2H), 1.75 (p, J = 6.7 Hz, 2H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 169.3, 135.7, 133.5, 129.2, 129.1, 128.4, 126.9, 126.3, 125.2, 124.9, 123.7, 48.5, 45.6, 26.0, 24.6. The spectral data were identical to those reported in the literature.[2]



**Naphthalen-2-yl(pyrrolidin-1-yl)methanone (2n):** Brown liquid, 51 mg. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.92 (d, J = 1.6 Hz, 1H), 7.80 – 7.74 (m, 3H), 7.53 (dd, J = 8.5, 1.7 Hz, 1H), 7.47 – 7.40 (m, 2H), 3.62 (t, J = 7.0 Hz, 2H), 3.40 (t, J = 6.6 Hz, 3H), 1.90 (p, J = 6.6 Hz, 3H), 1.80 (q, J = 6.4 Hz, 3H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 168.7, 133.4, 132.7, 131.5, 127.4, 127.0, 126.7, 126.0, 125.9, 125.5, 123.3, 48.7, 45.3, 25.3, 23.4. The spectral data were identical to those reported in the literature.[2]

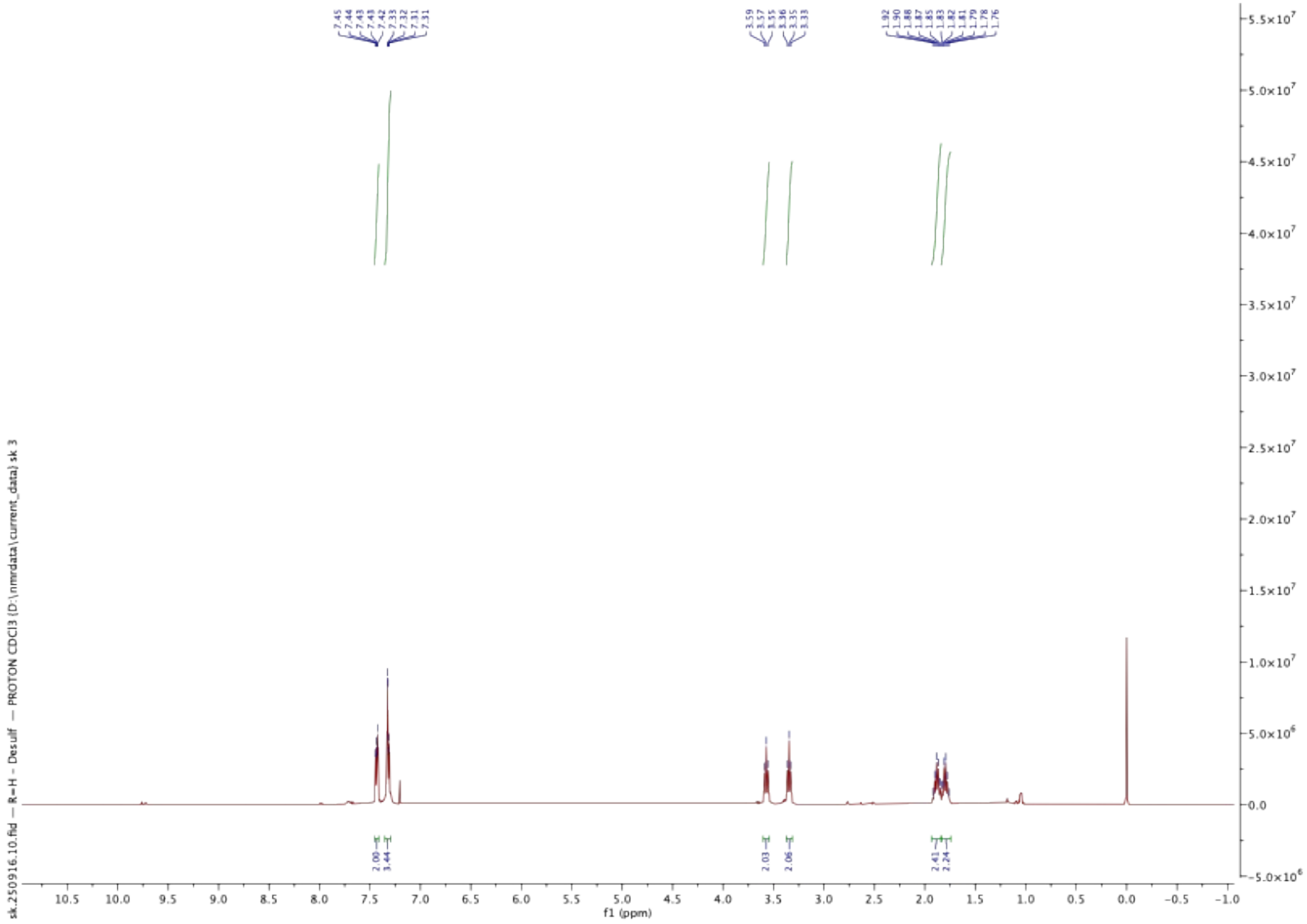


***N*,*N*-diisopropylbenzamide (2p):** Pale yellow solid, 52 mg. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.32 – 7.26 (m, 3H), 7.23 (dd, J = 7.1, 2.6 Hz, 2H), 3.60 (d, J = 72.5 Hz, 2H), 1.71 – 0.73 (m, 12H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 171.0, 138.9, 128.6, 128.4, 125.5, 50.7, 45.8, 29.7, 20.7. The spectral data were identical to those reported in the literature.[8]

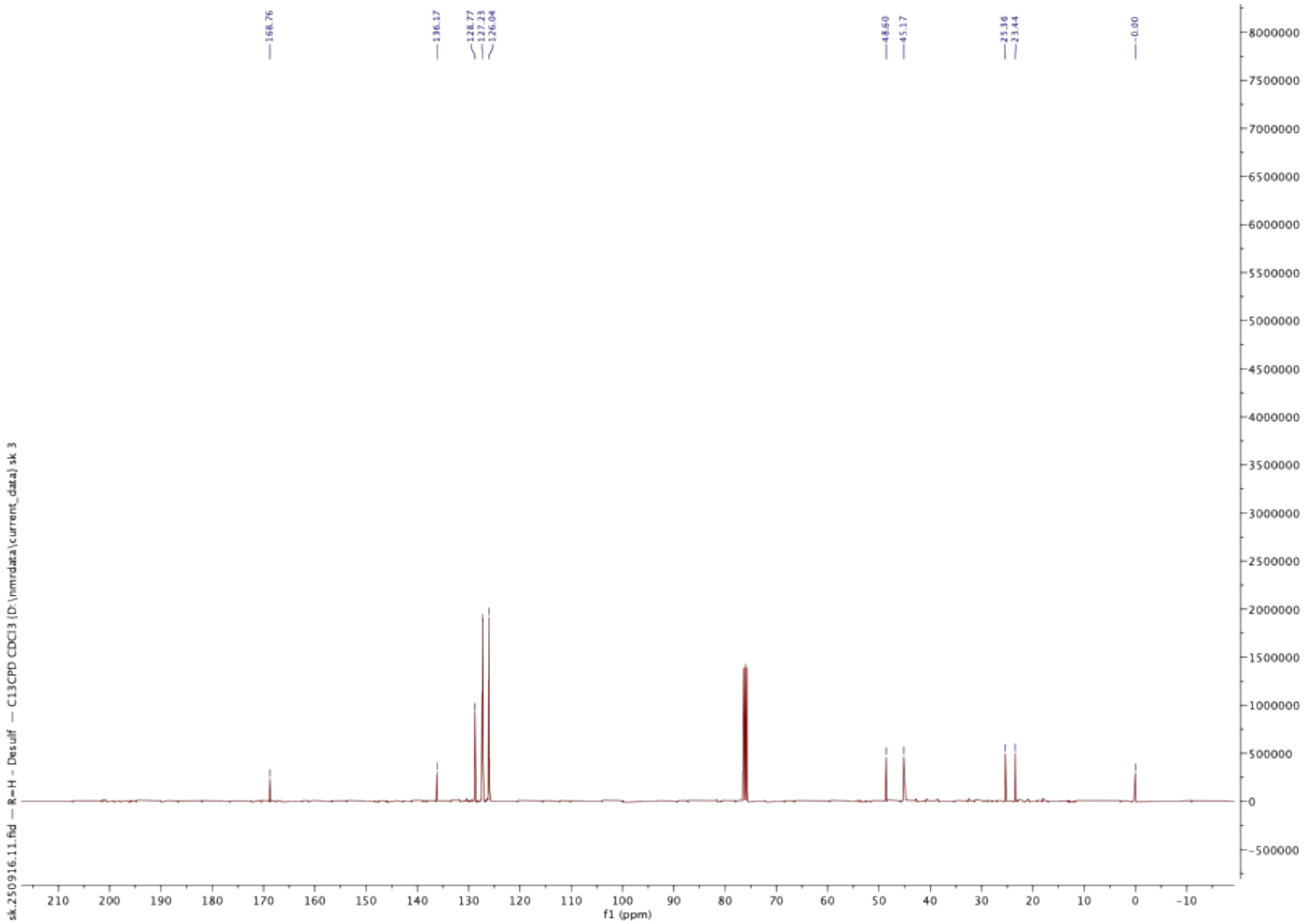


***N*,*N*-dibutylbenzamide (2q):** Brown liquid, 45 mg. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.33 – 7.24 (m, 5H), 3.41 (d, J = 5.4 Hz, 2H), 3.10 (d, J = 9.0 Hz, 2H), 1.56 (s, 2H), 1.47 – 1.25 (m, 4H), 1.11 – 0.99 (m, 2H), 0.90 (s, 3H), 0.77 – 0.62 (m, 3H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 171.6, 137.3, 128.9, 128.3, 126.4, 48.7, 44.4, 30.8, 29.7, 29.6, 20.3, 19.7, 13.9, 13.6. The spectral data were identical to those reported in the literature.[9]

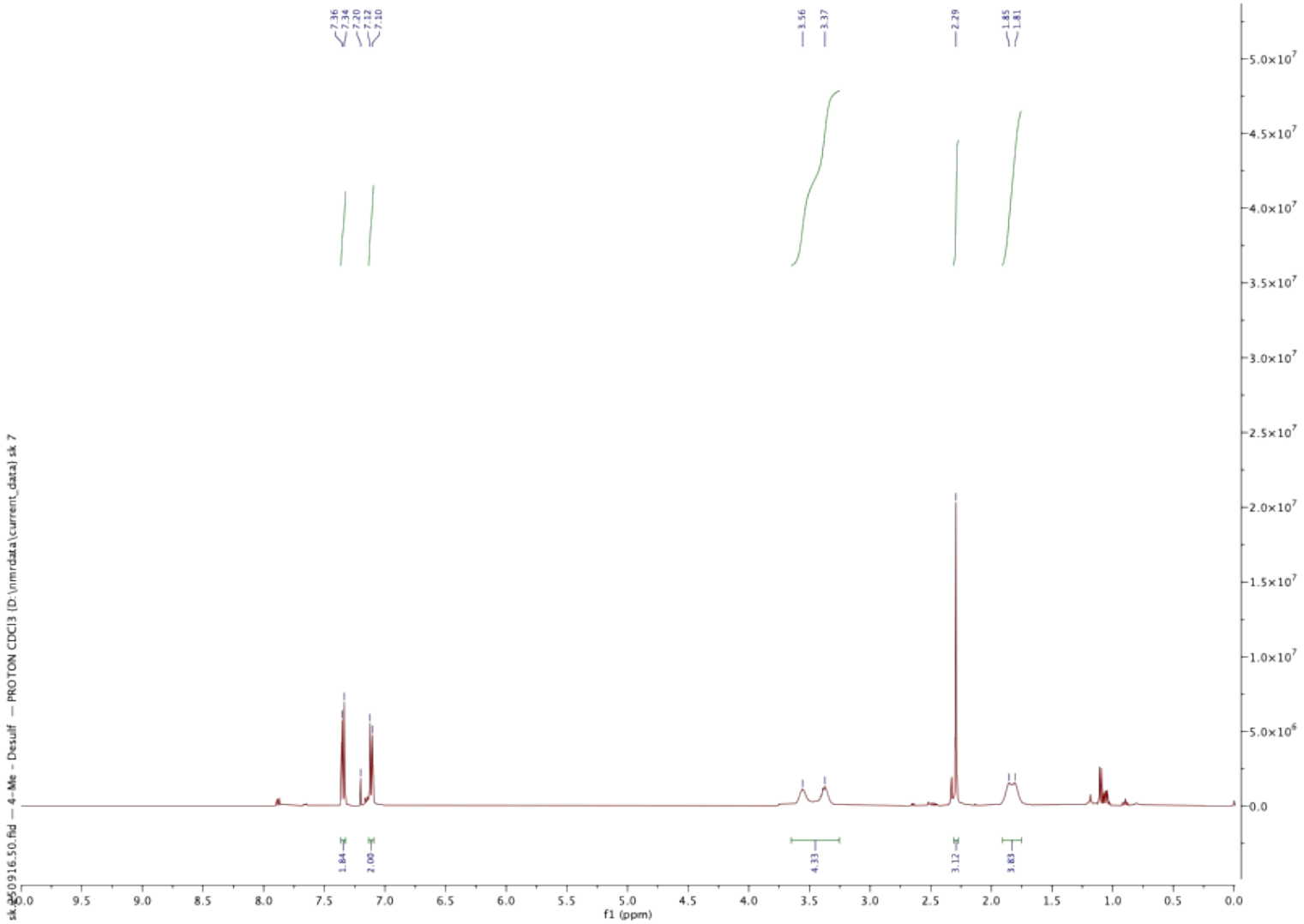
$^{1}H$ (400 MHz, $CDCl_3$) NMR spectrum of **2a**



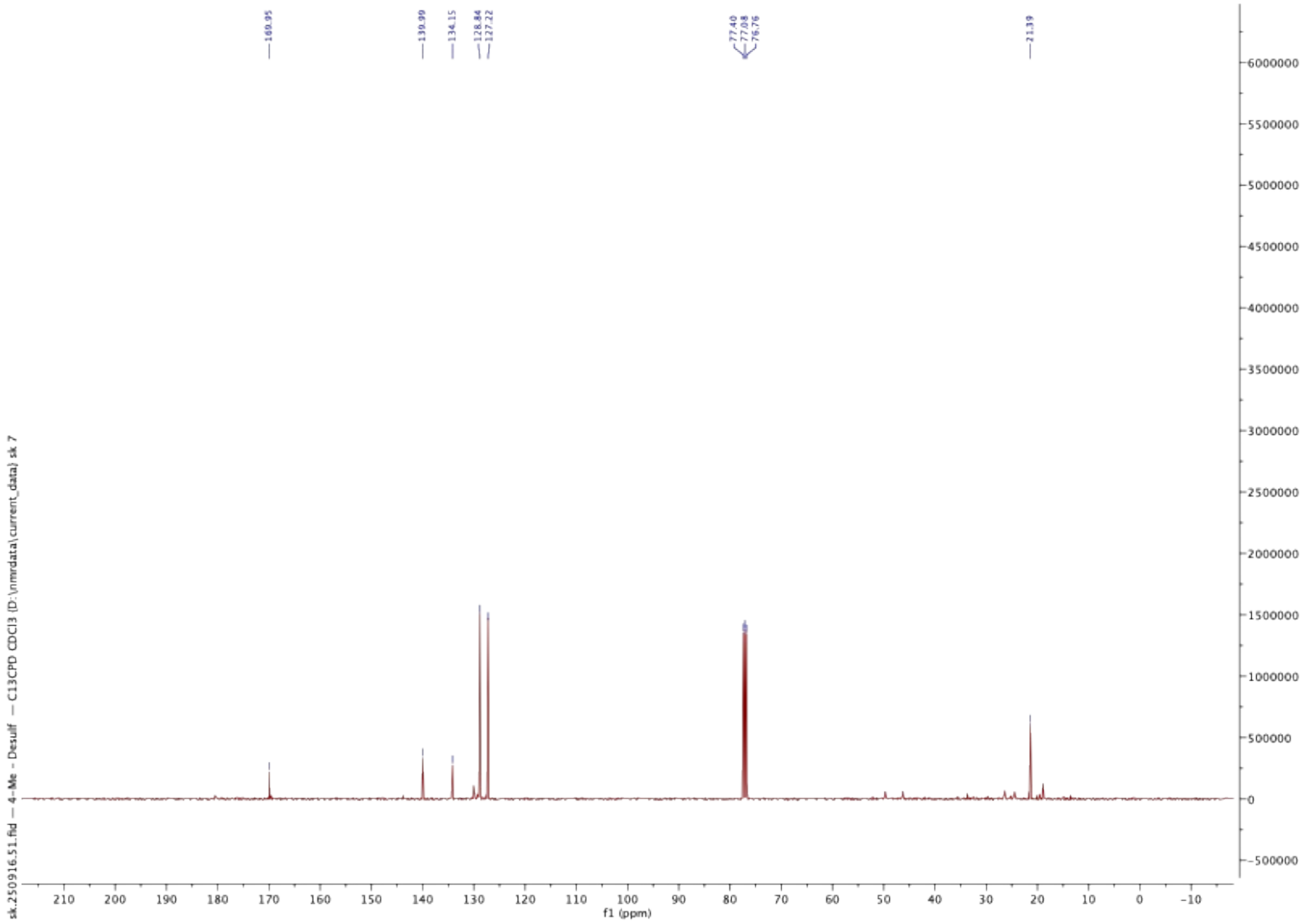
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectrum of **2a**



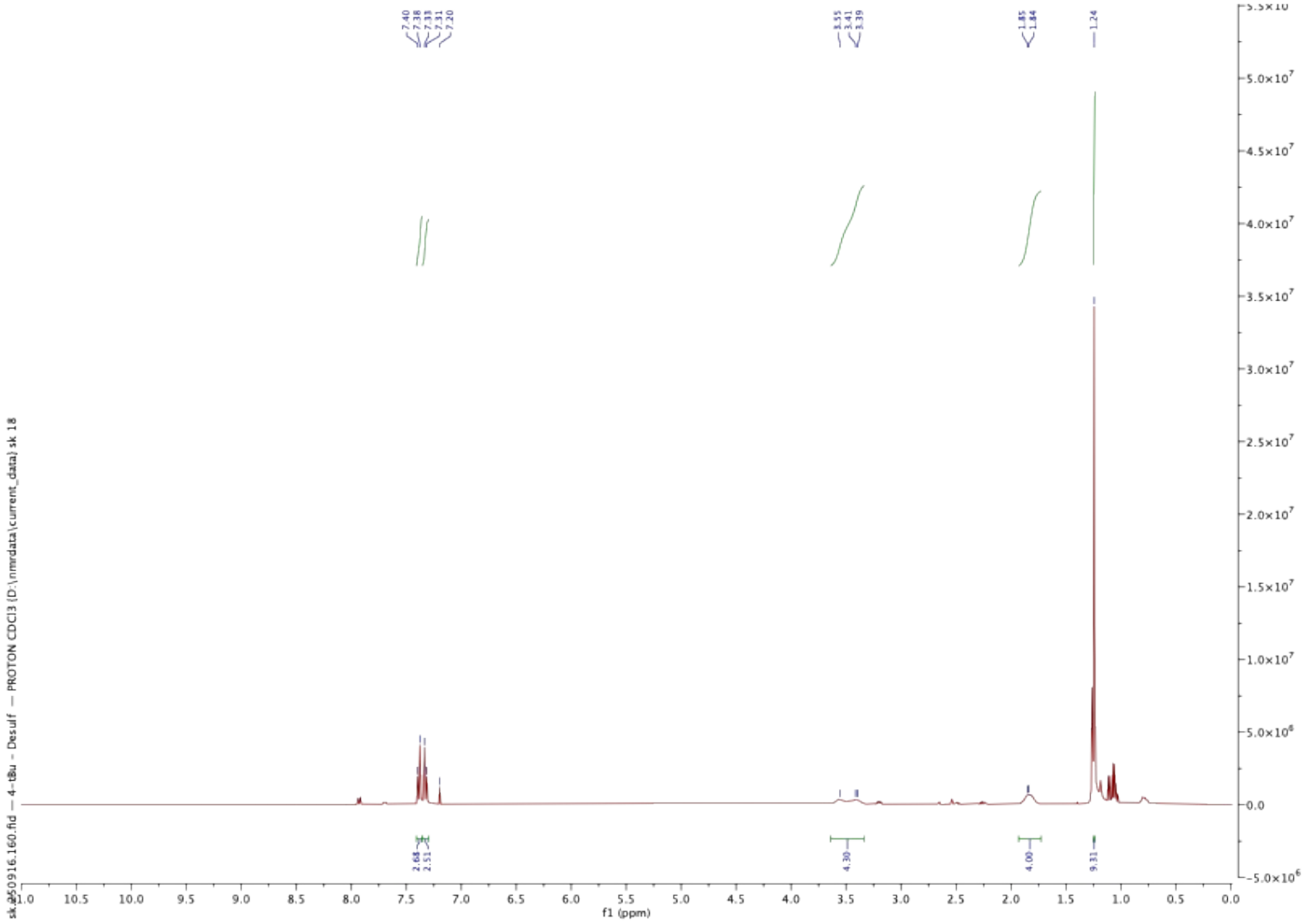
$^{1}$H (400 MHz, $CDCl_3$) NMR spectrum of **2b**



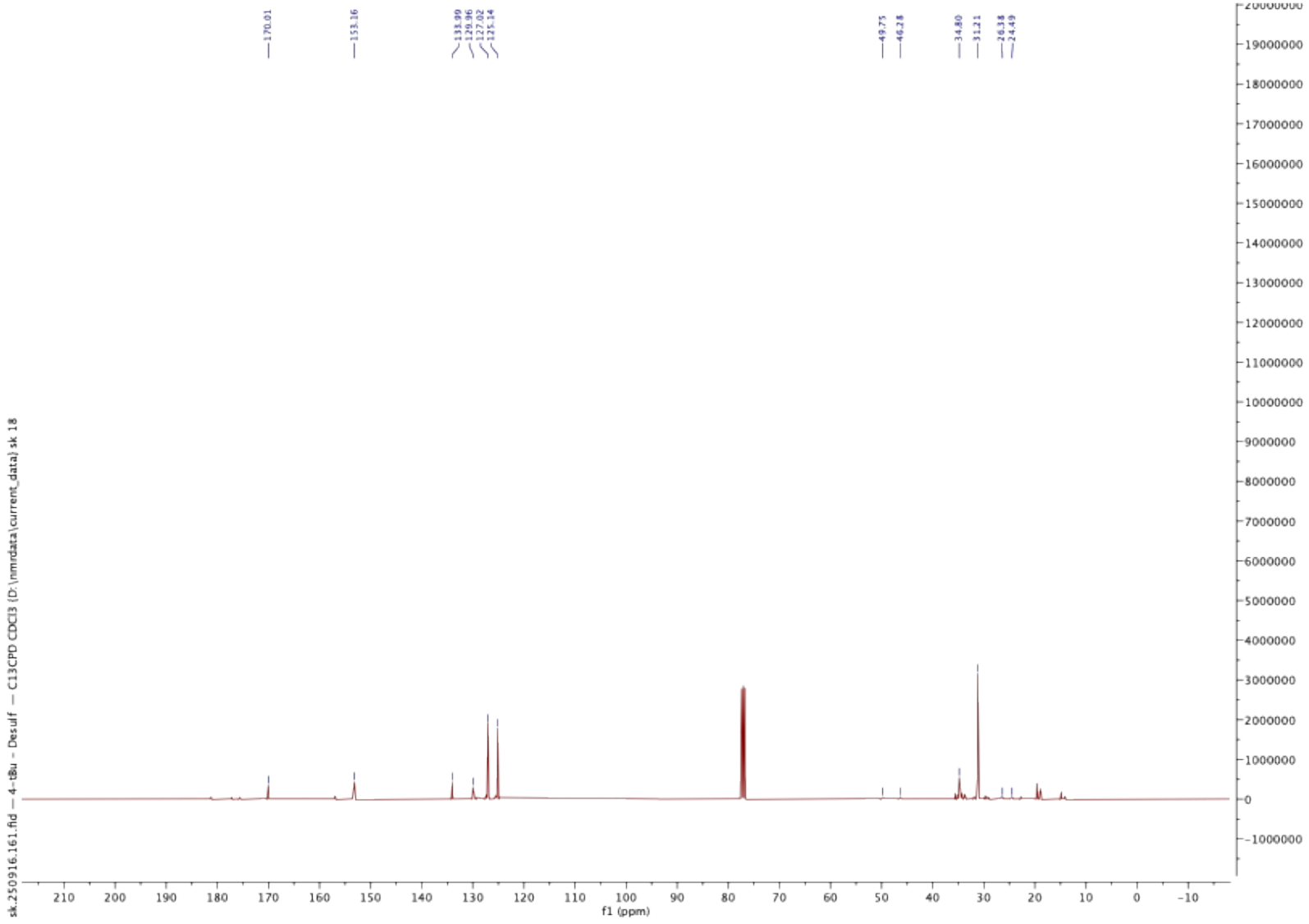
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectrum of **2b**



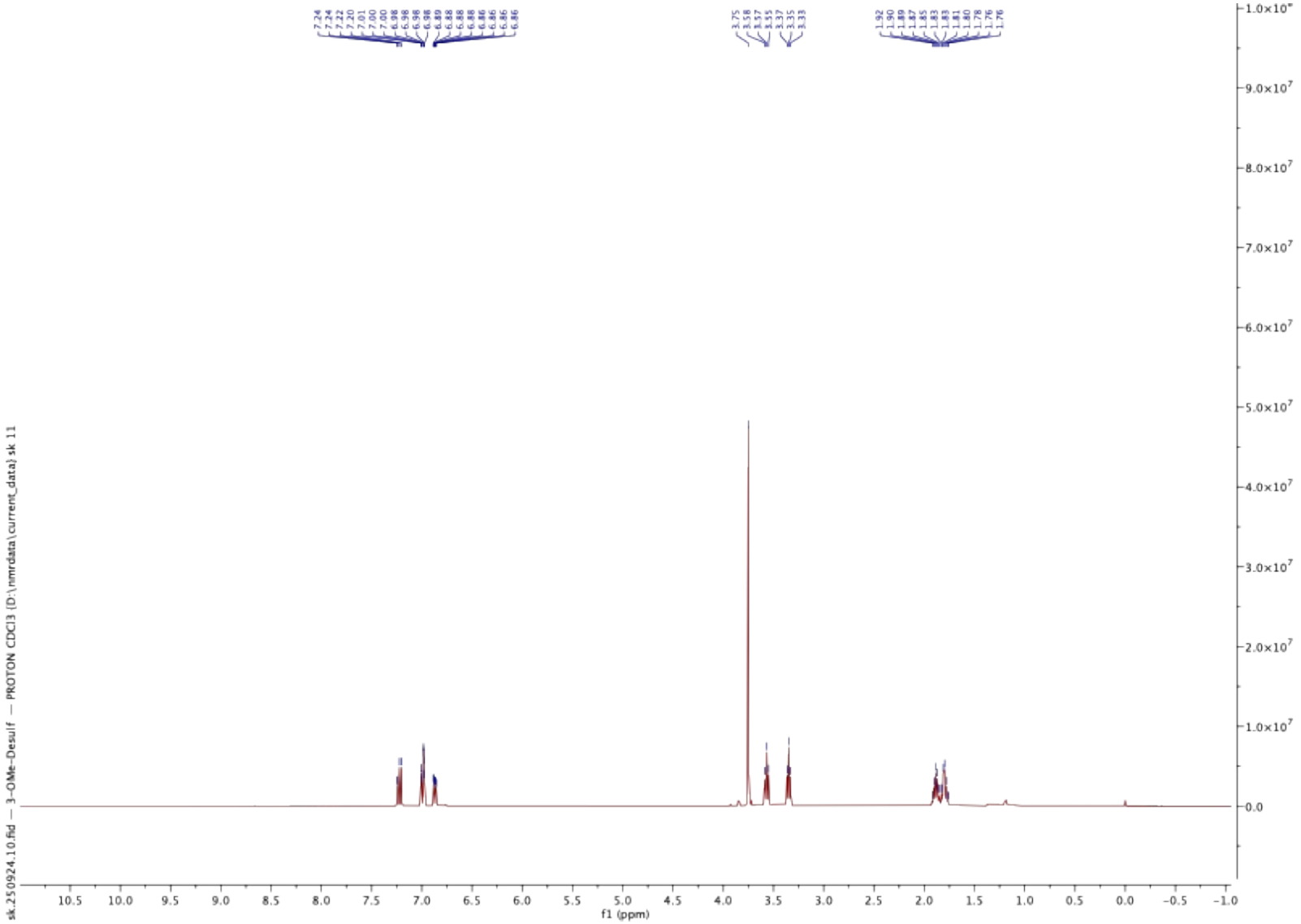
$^1$H (400 MHz, $CDCl_3$) NMR spectrum of **2c**



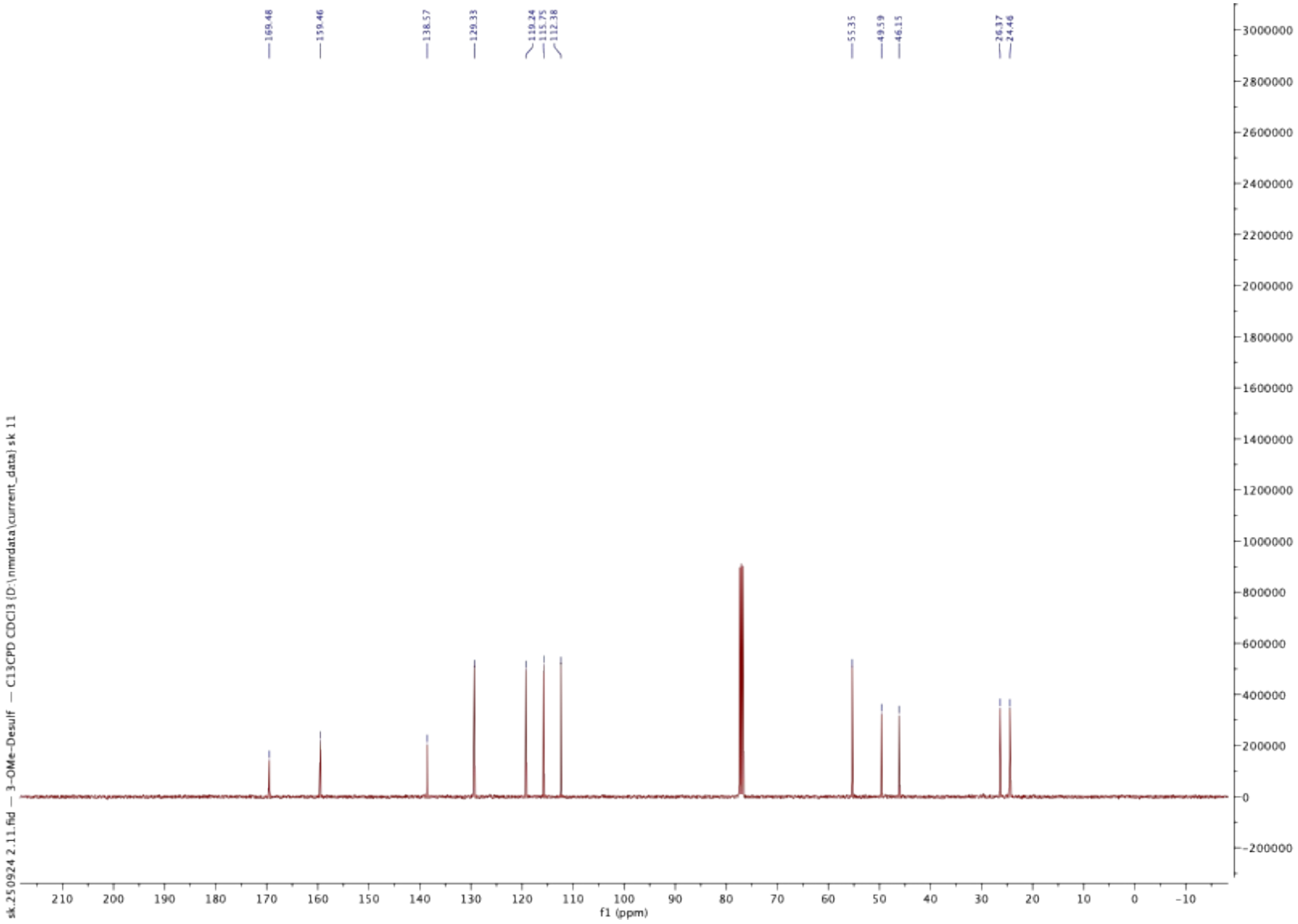
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectrum of **2c**



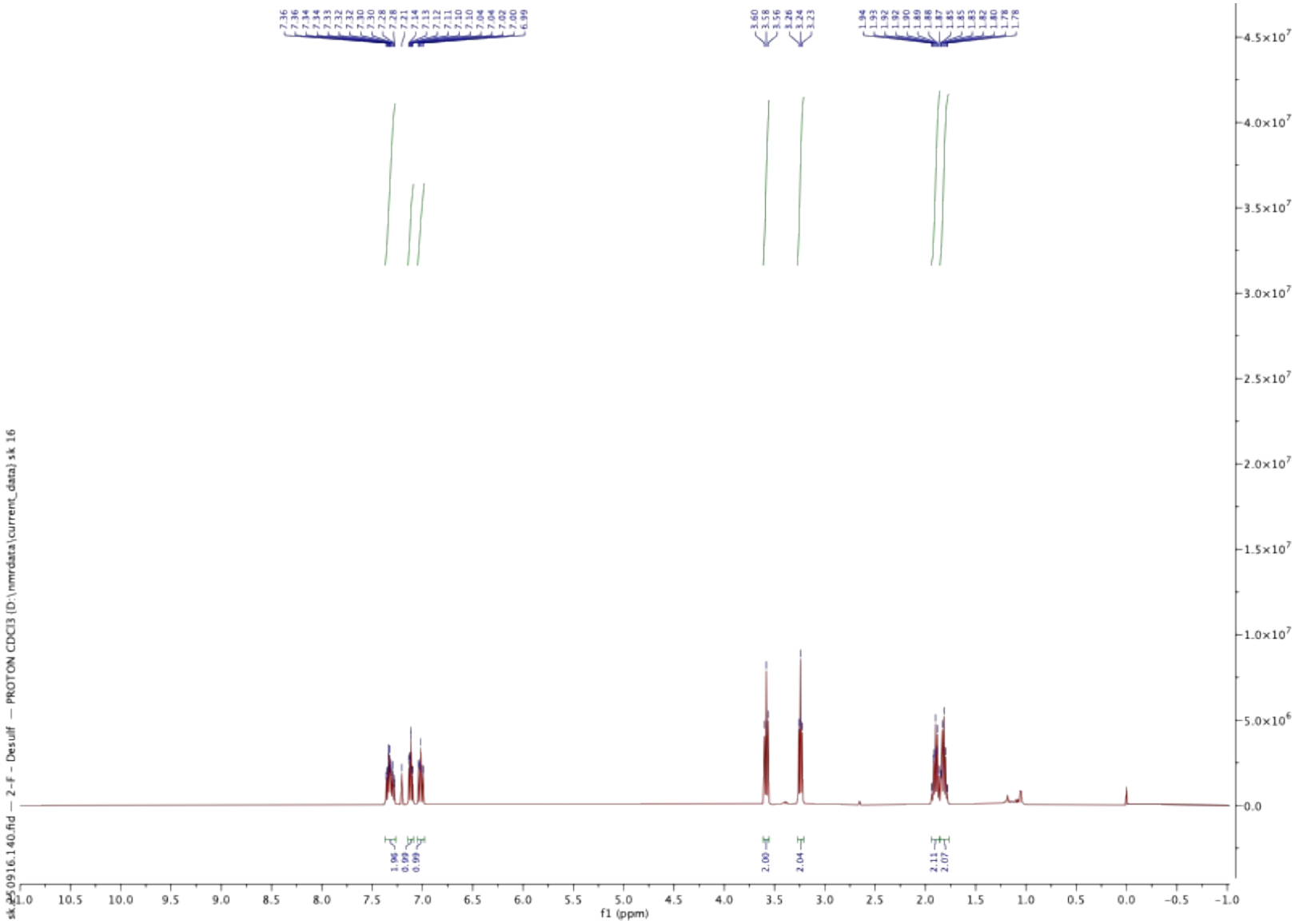
$^{1}H$ (400 MHz, $CDCl_3$) NMR spectrum of **2d**



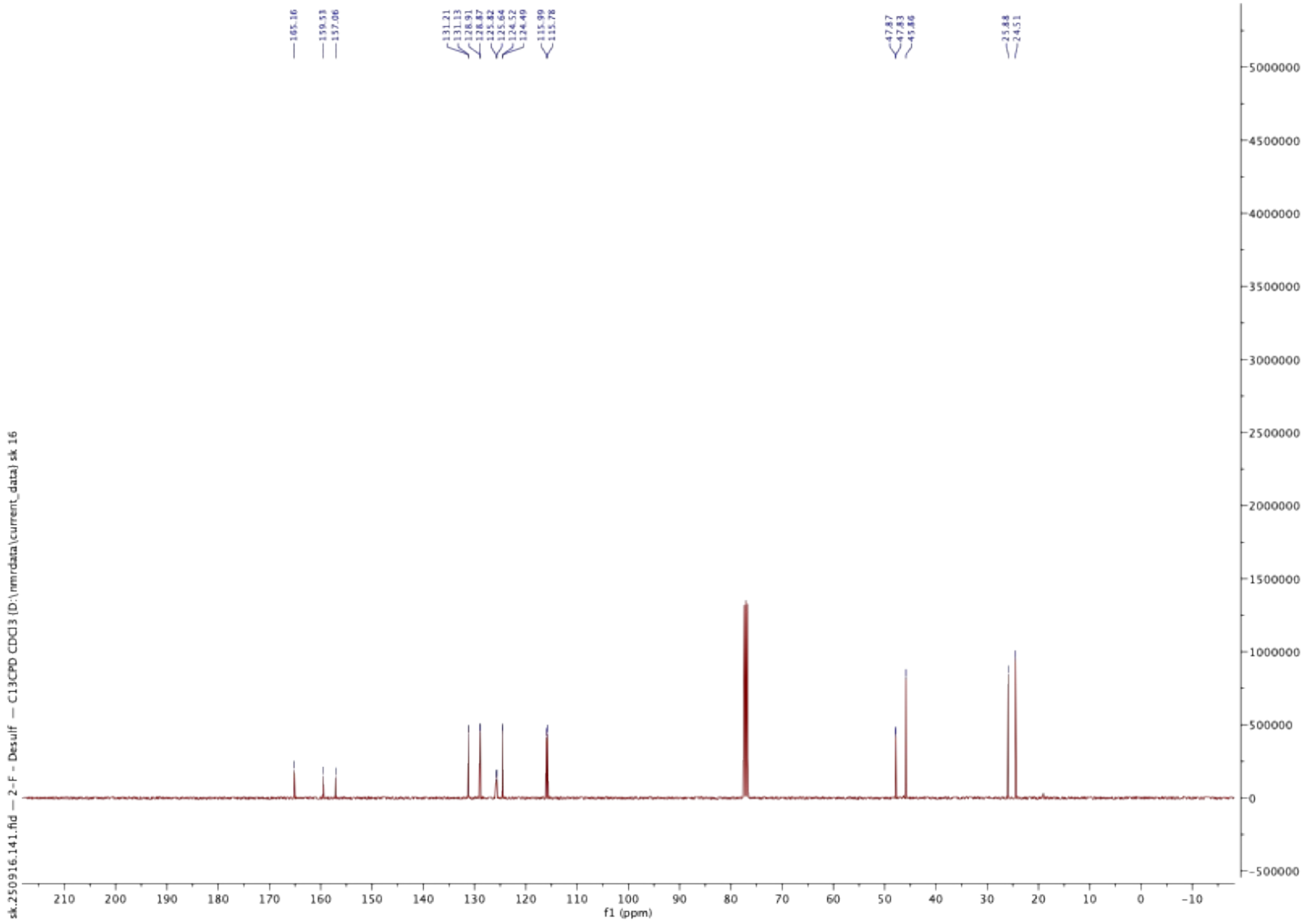
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectrum of **2d**



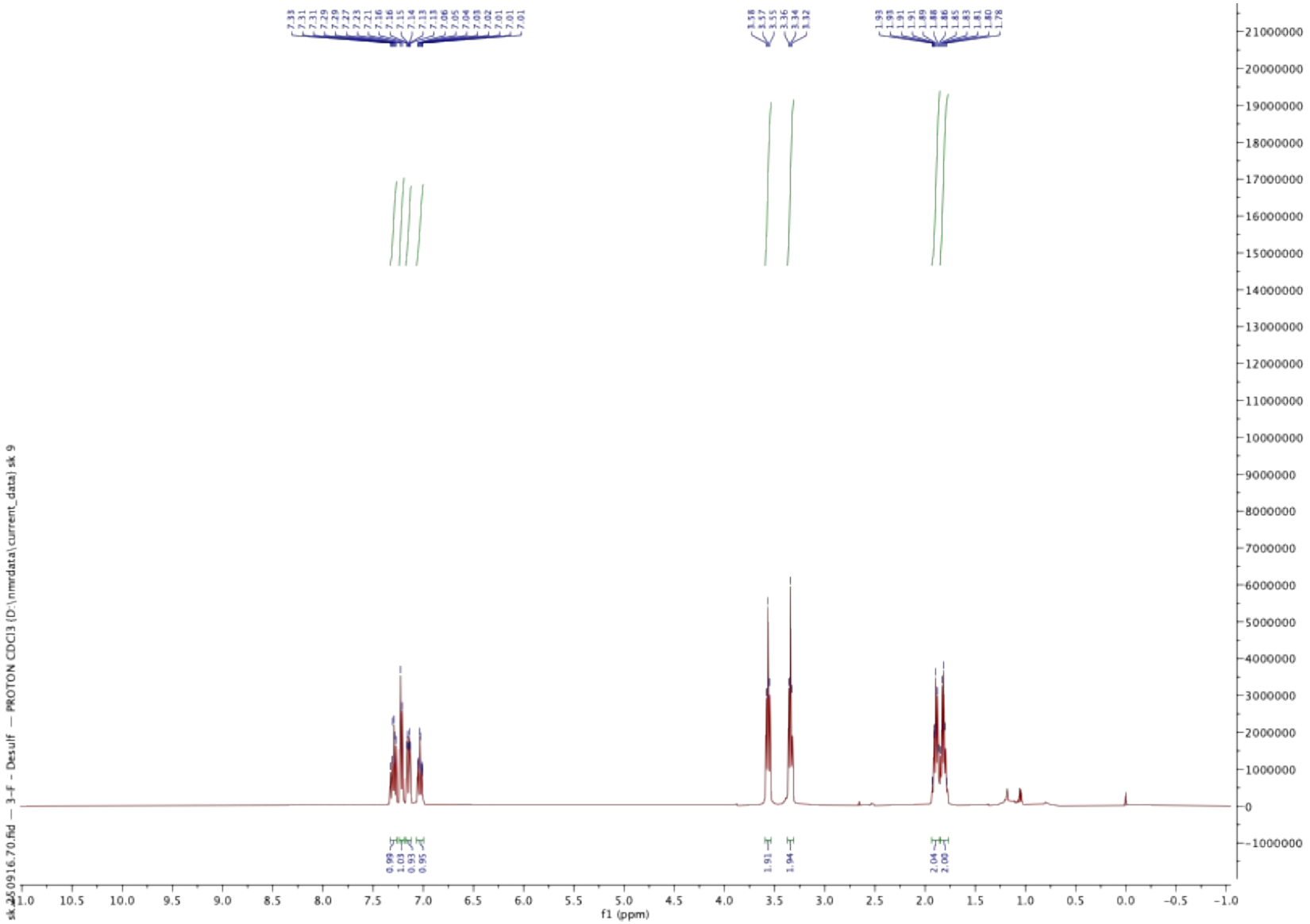
$^1$H (400 MHz, $CDCl_3$) NMR spectrum of **2e**



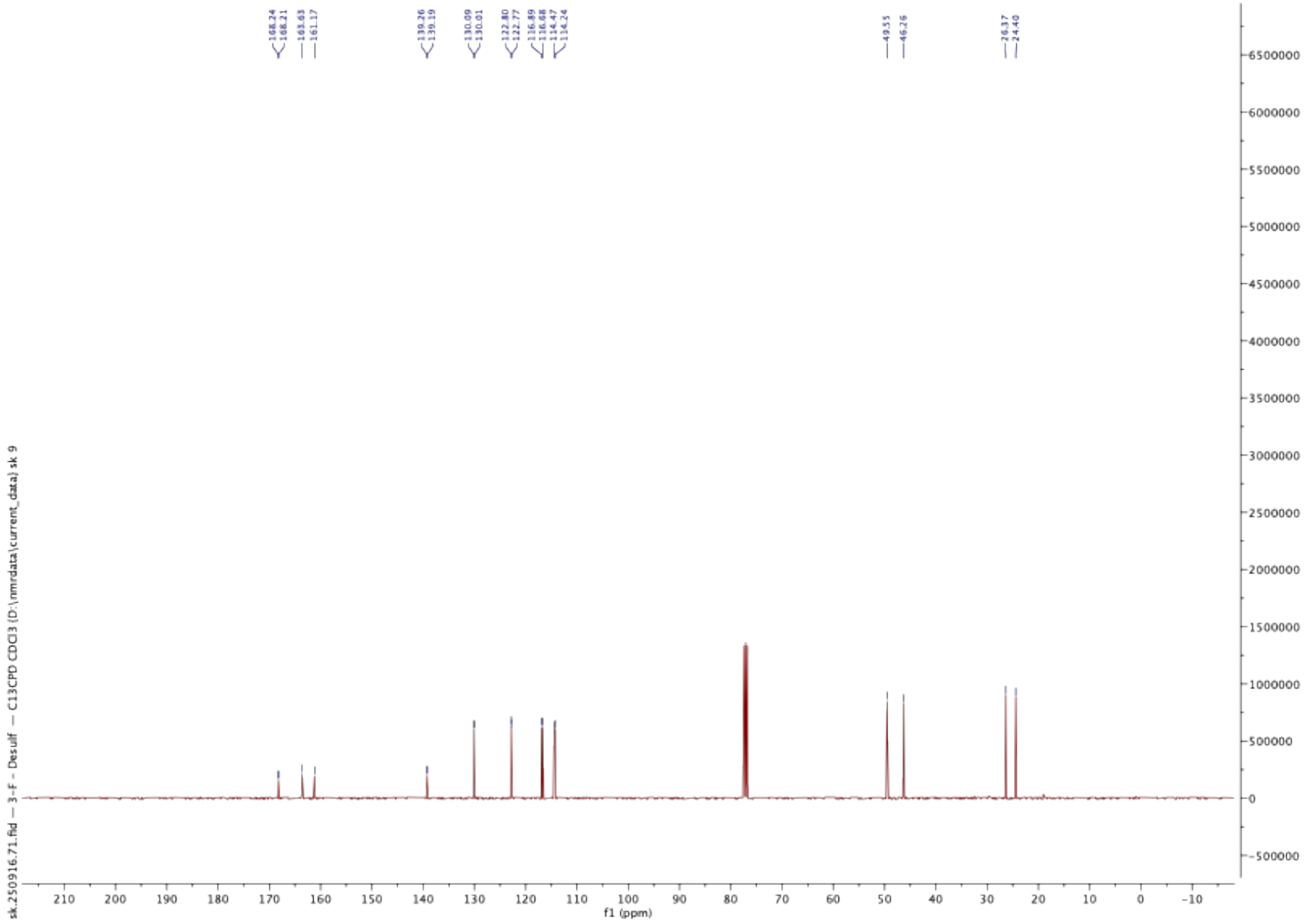
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectrum of **2e**



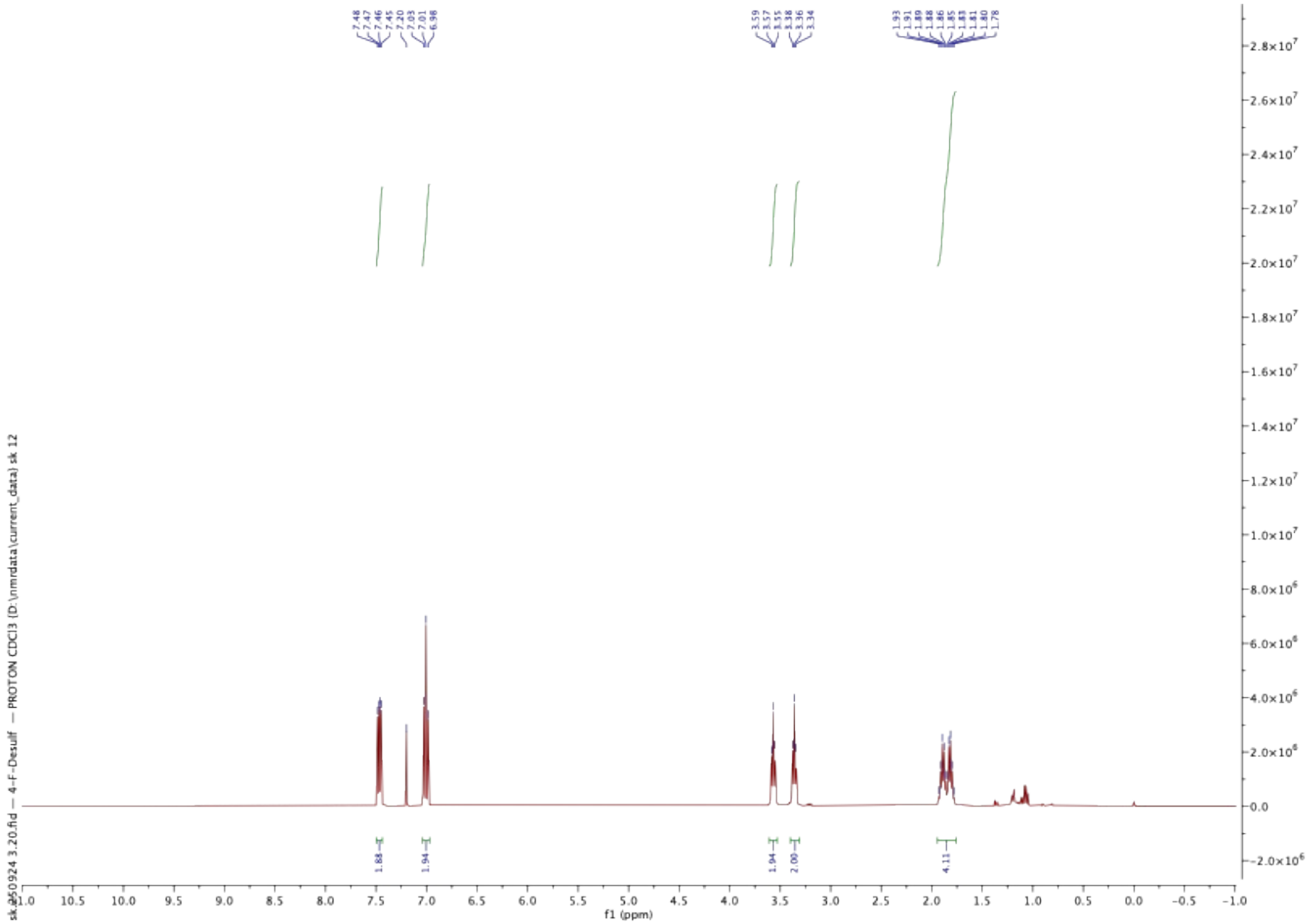
$^{1}H$ (400 MHz, $CDCl_3$) NMR spectrum of **2f**



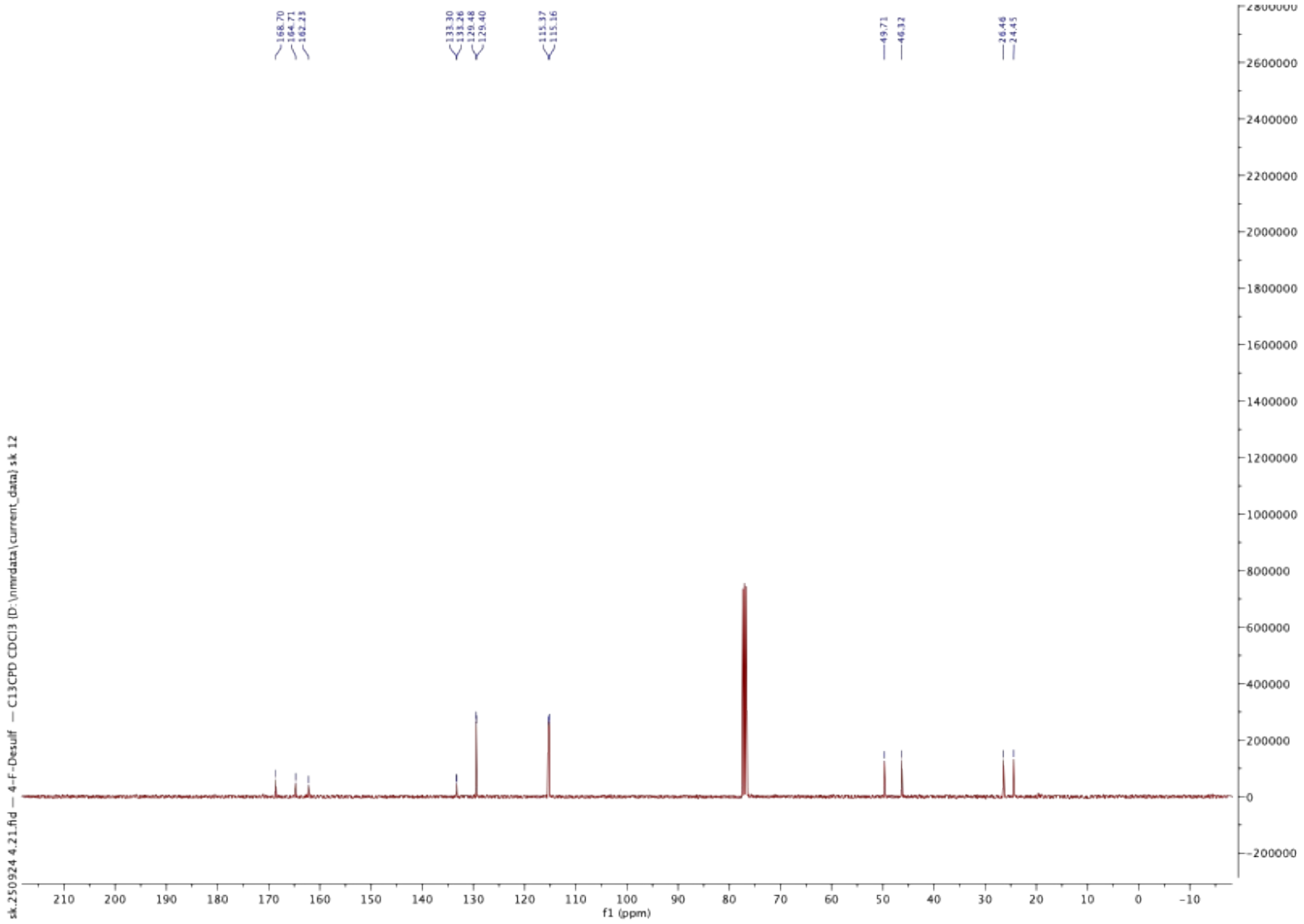
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectrum of **2f**



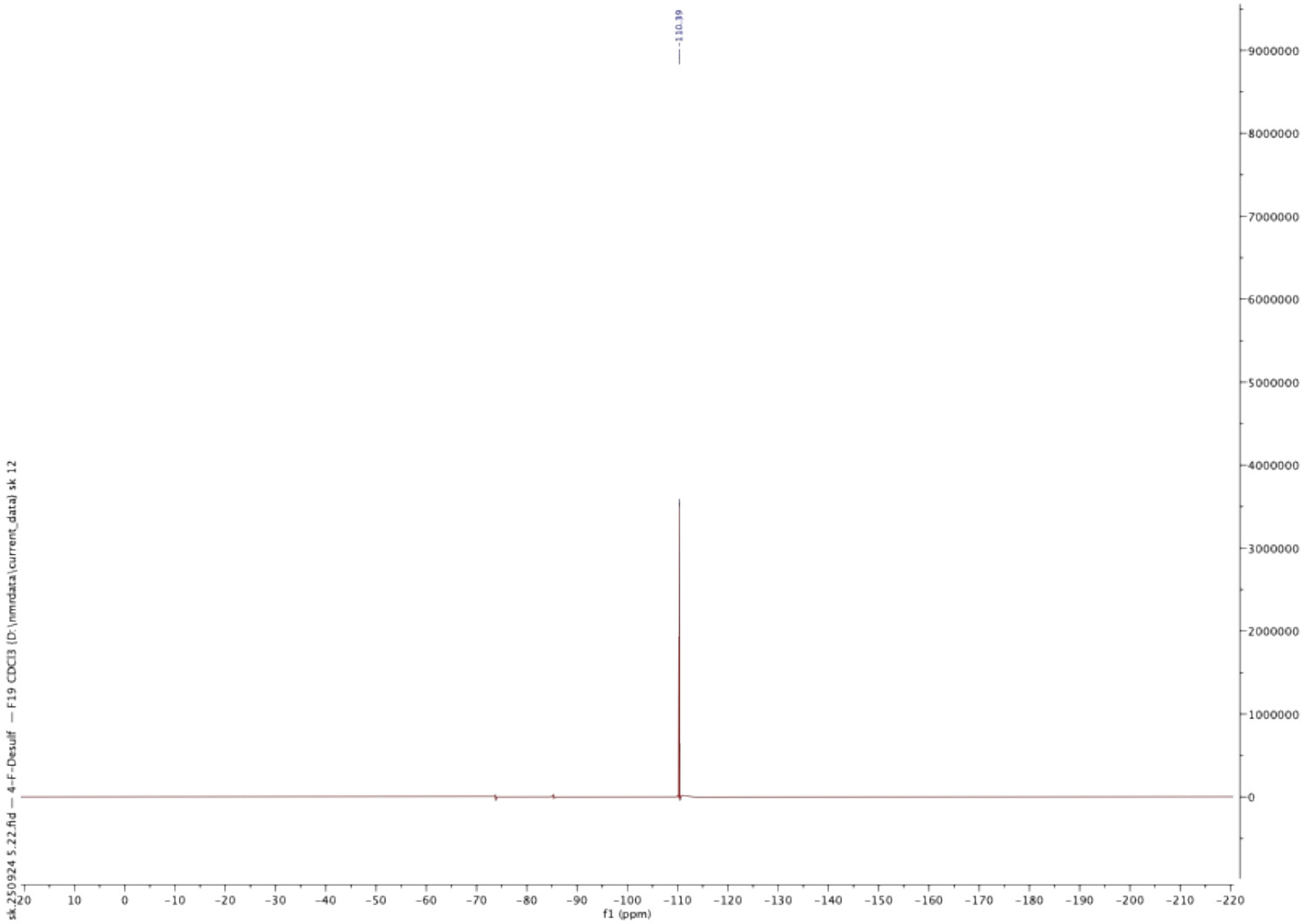
$^{1}$H (400 MHz, $CDCl_3$) NMR spectrum of **2g**



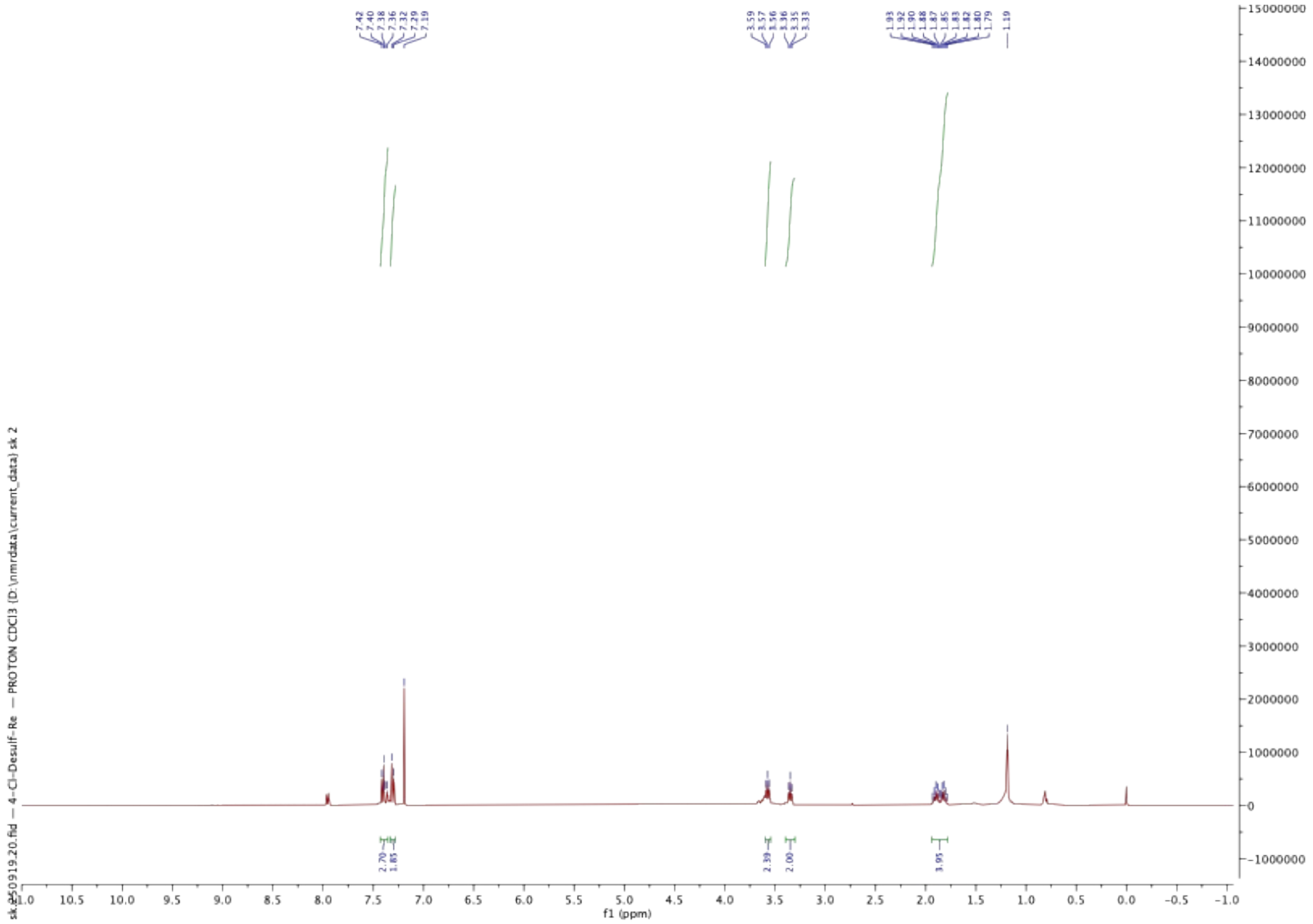
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectrum of **2g**



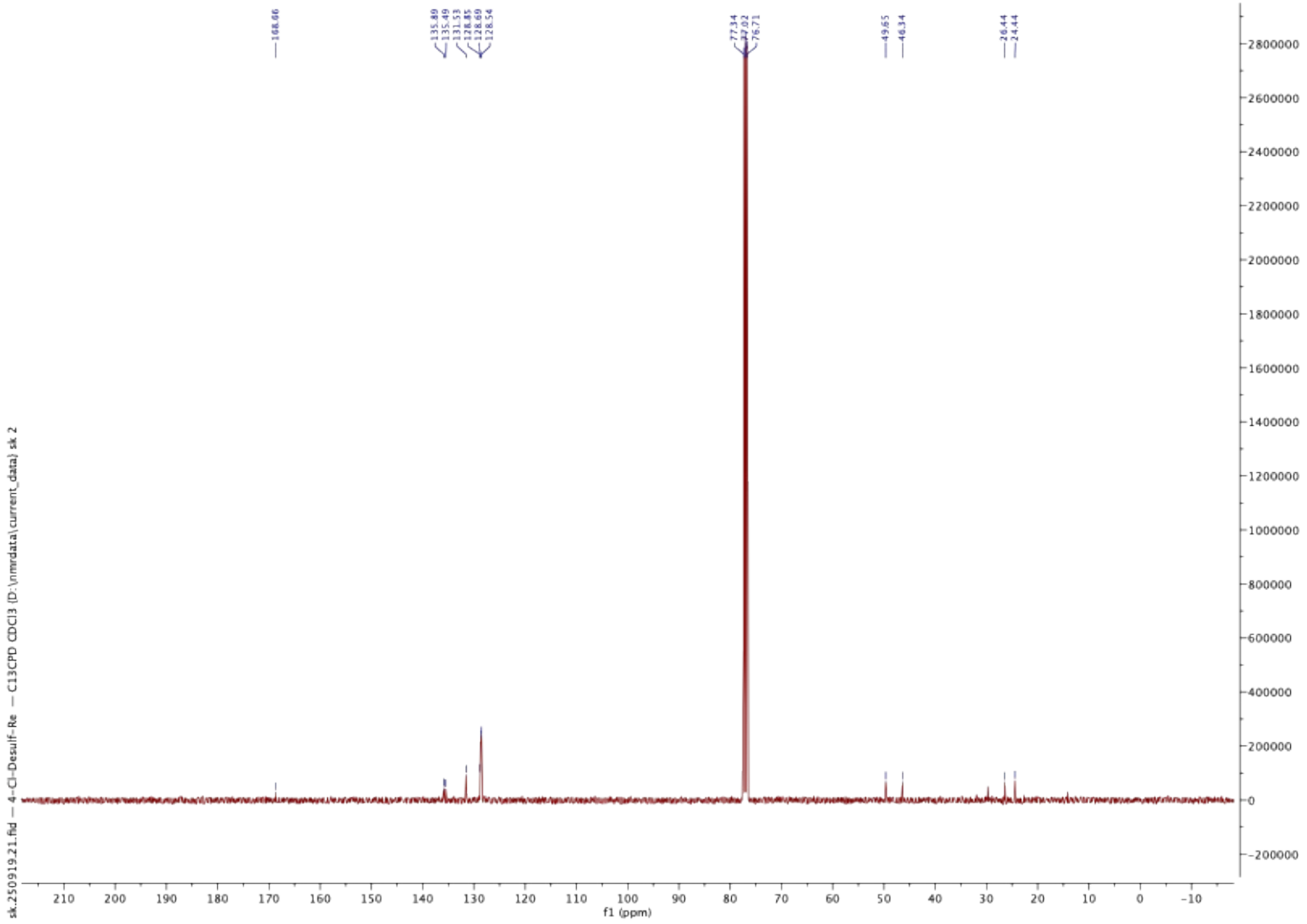
$^{19}F$ (375 MHz, $CDCl_3$) NMR spectrum of **2g**



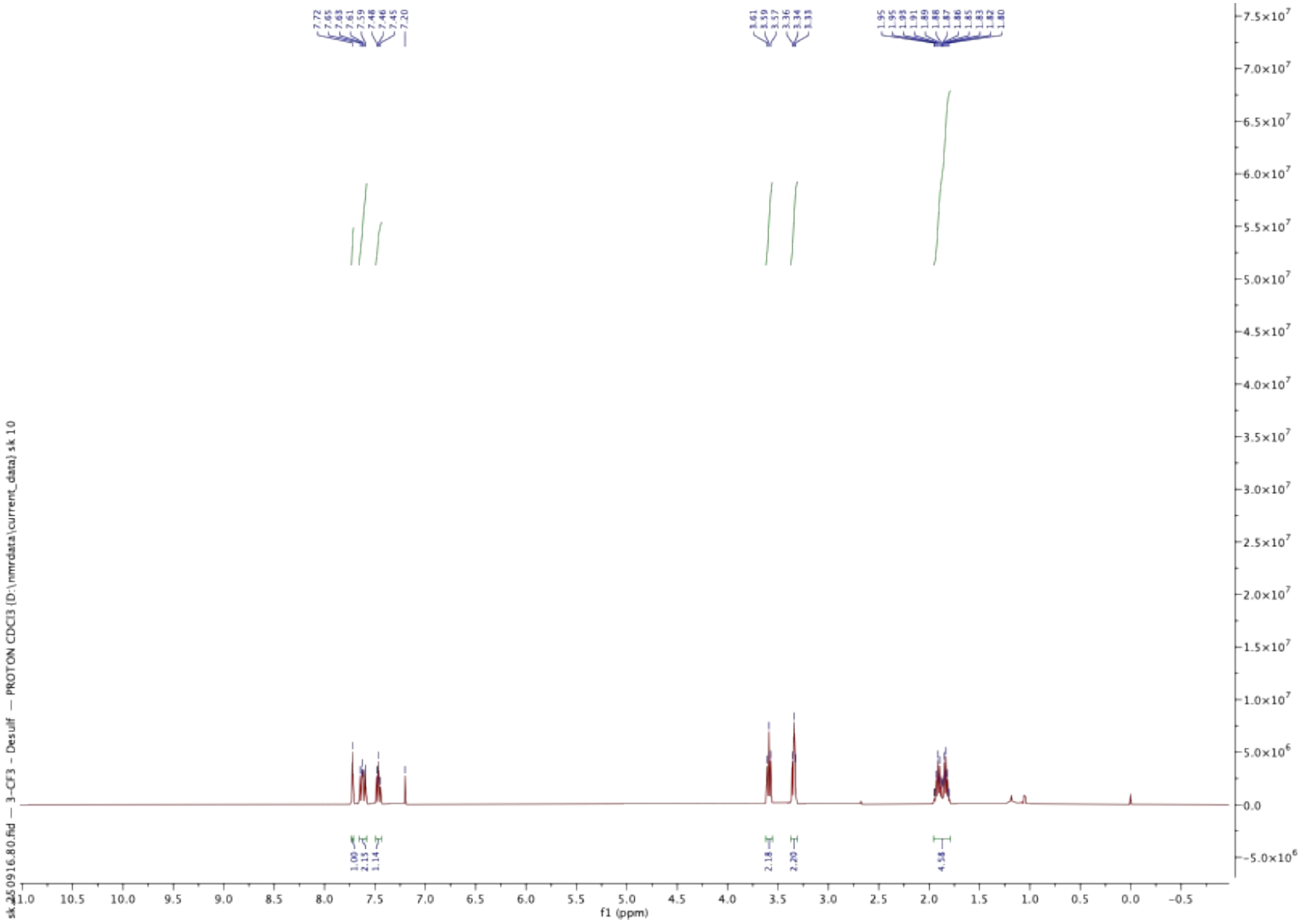
$^{1}H$ (400 MHz, $CDCl_3$) NMR spectrum of **2h**



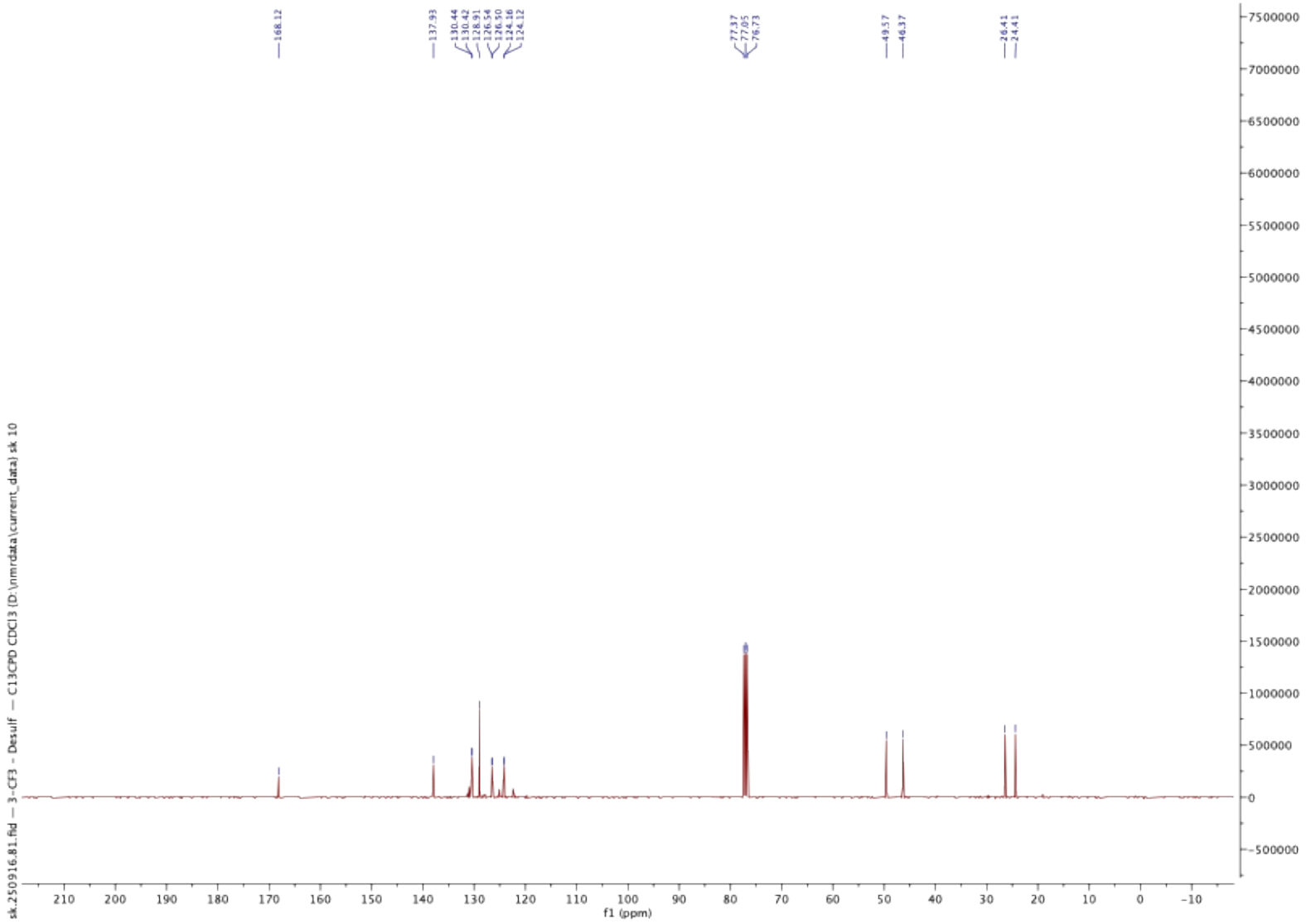
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectrum of **2h**



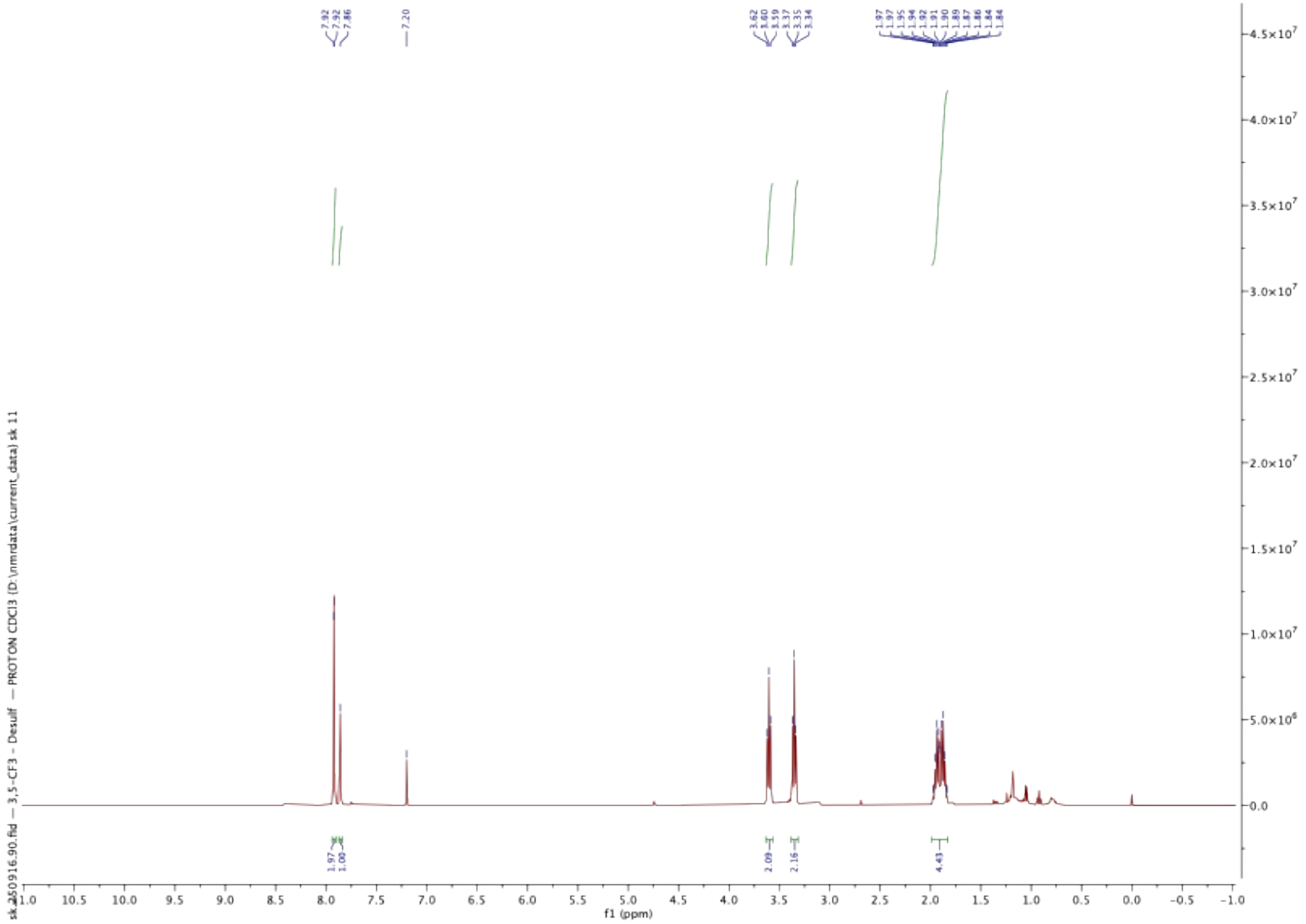
$^1$H (400 MHz, $CDCl_3$) NMR spectrum of **2i**



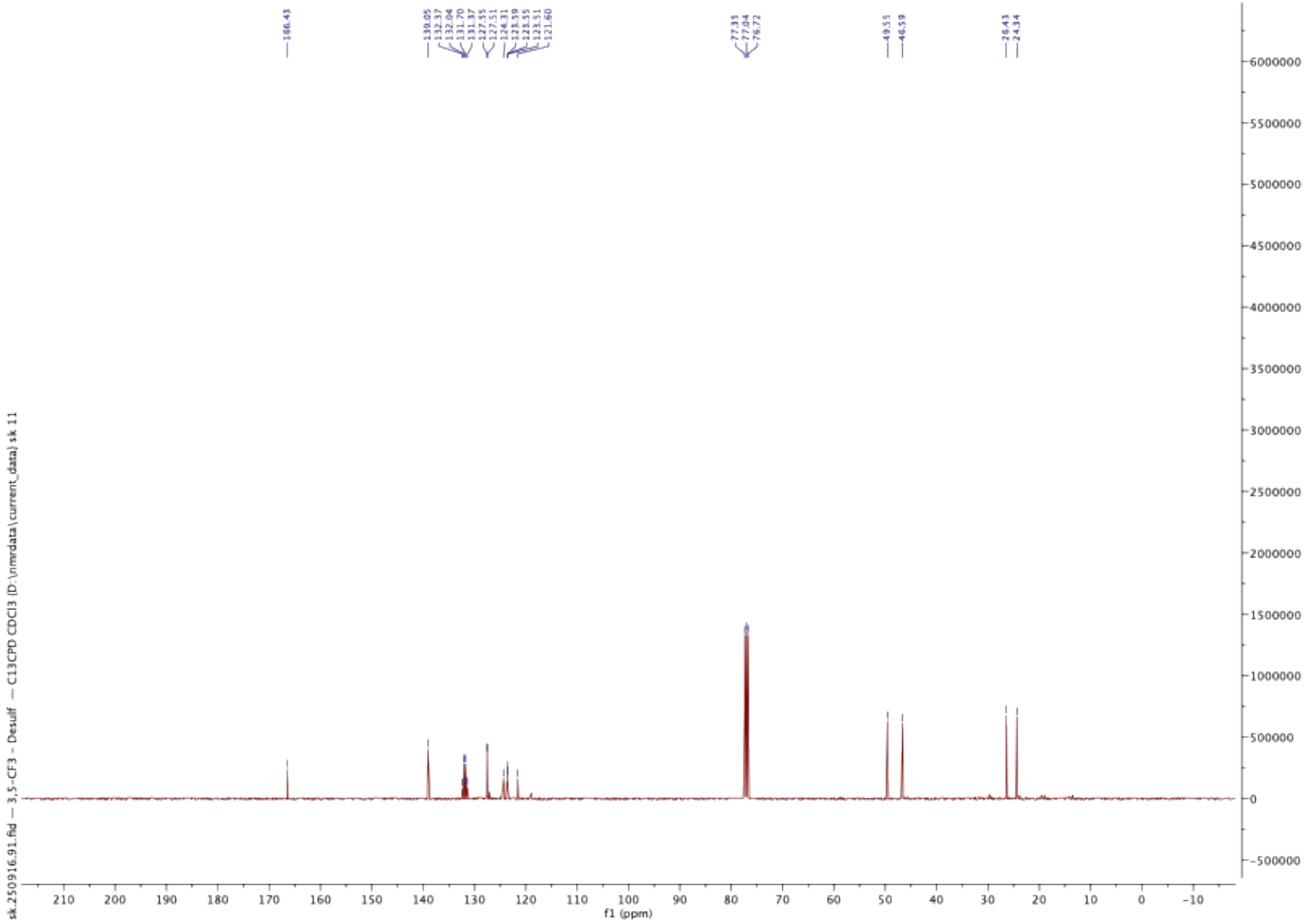
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectrum of **2i**



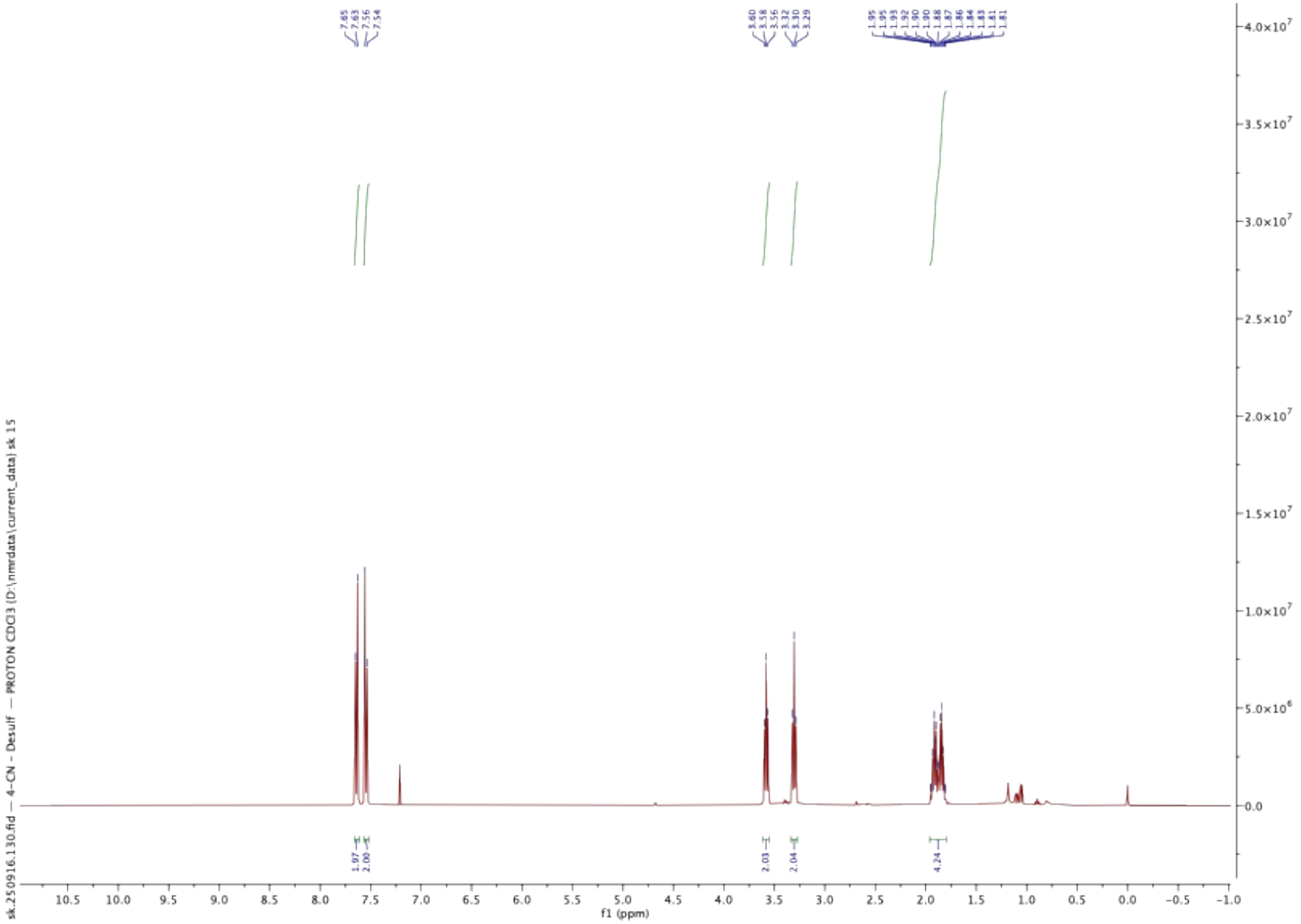
$^{1}H$ (400 MHz, $CDCl_3$) NMR spectrum of **2j**



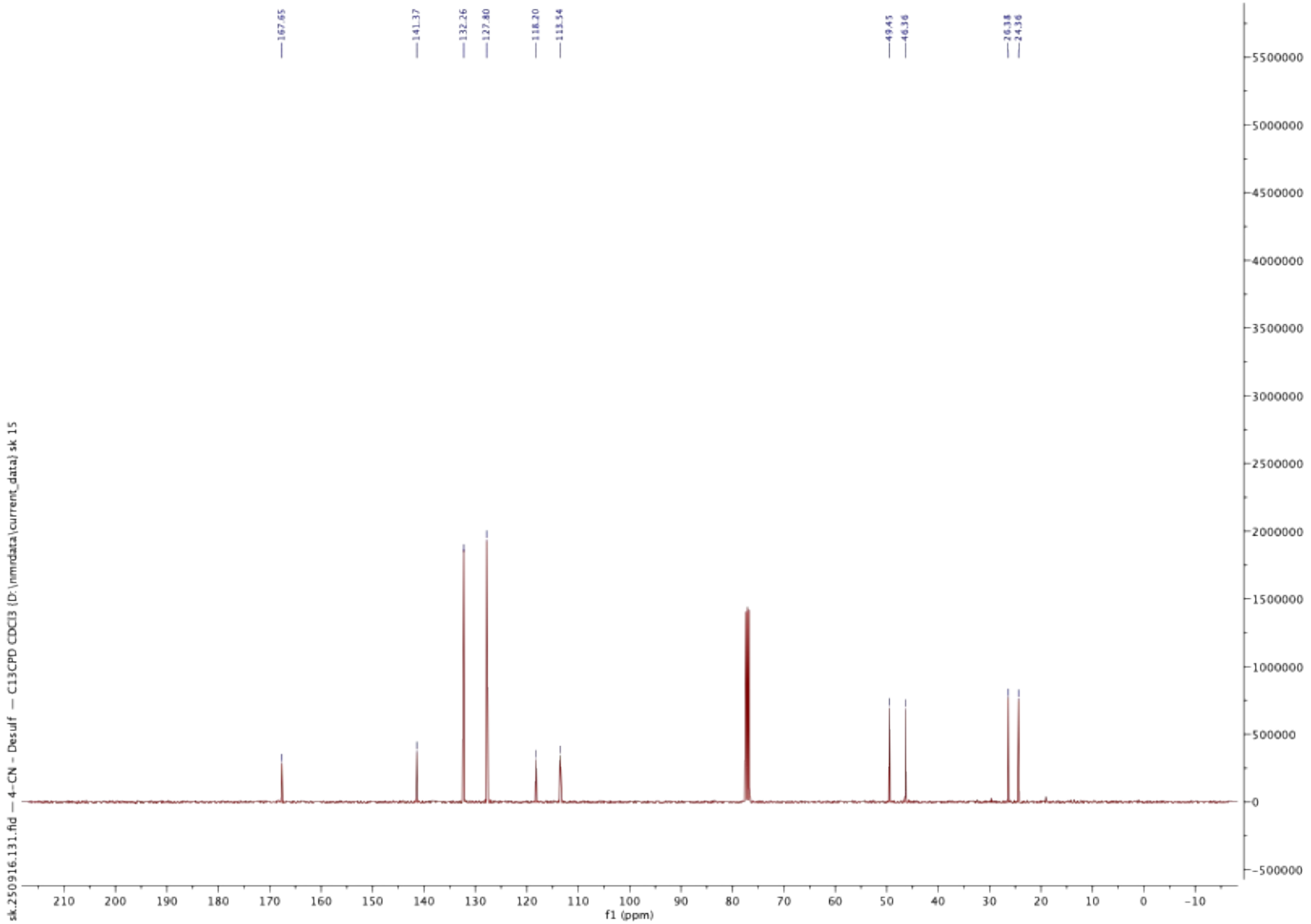
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) NMR spectrum of **2j**



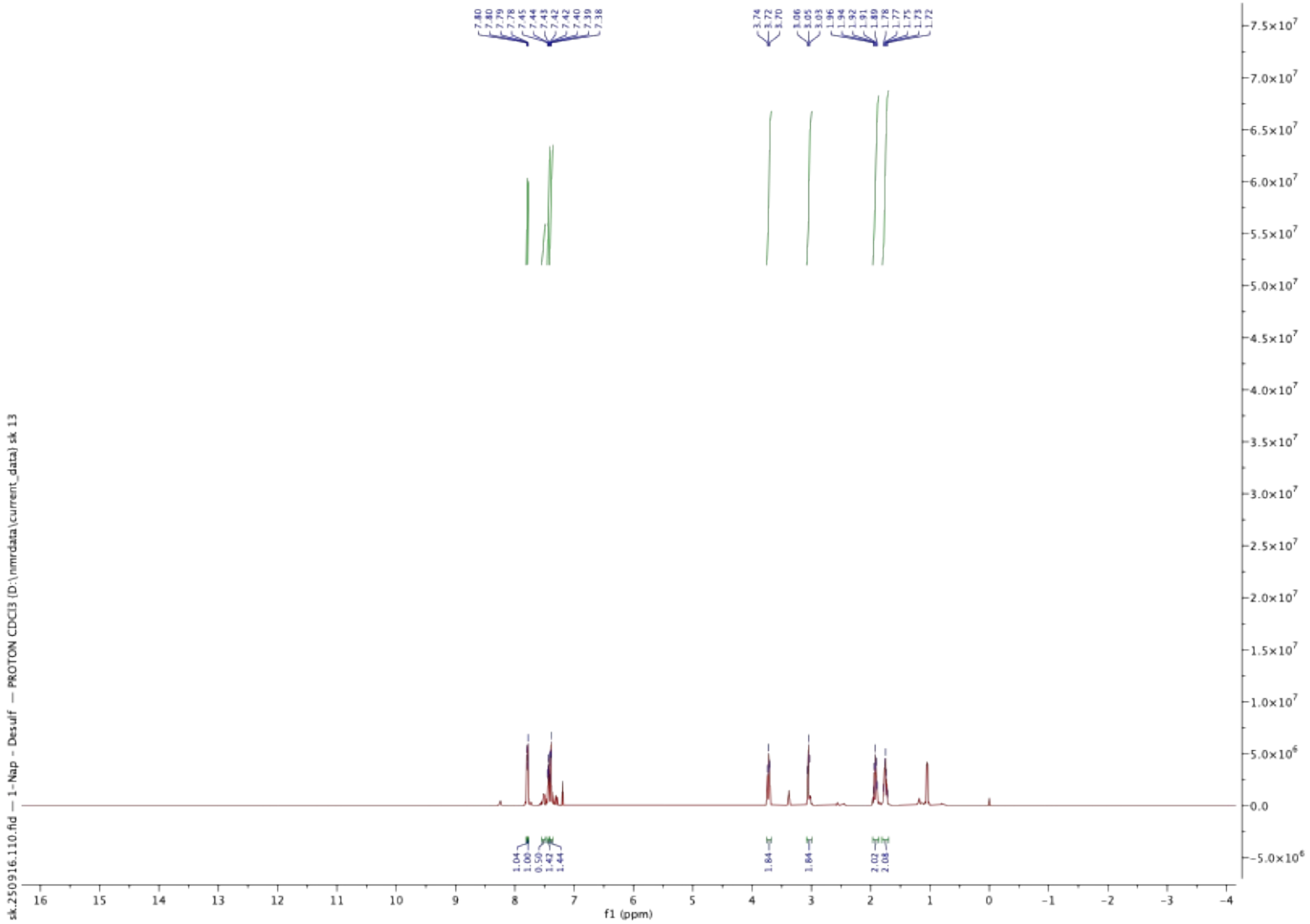
$^1$H (400 MHz, $CDCl_3$) NMR spectrum of **2k**



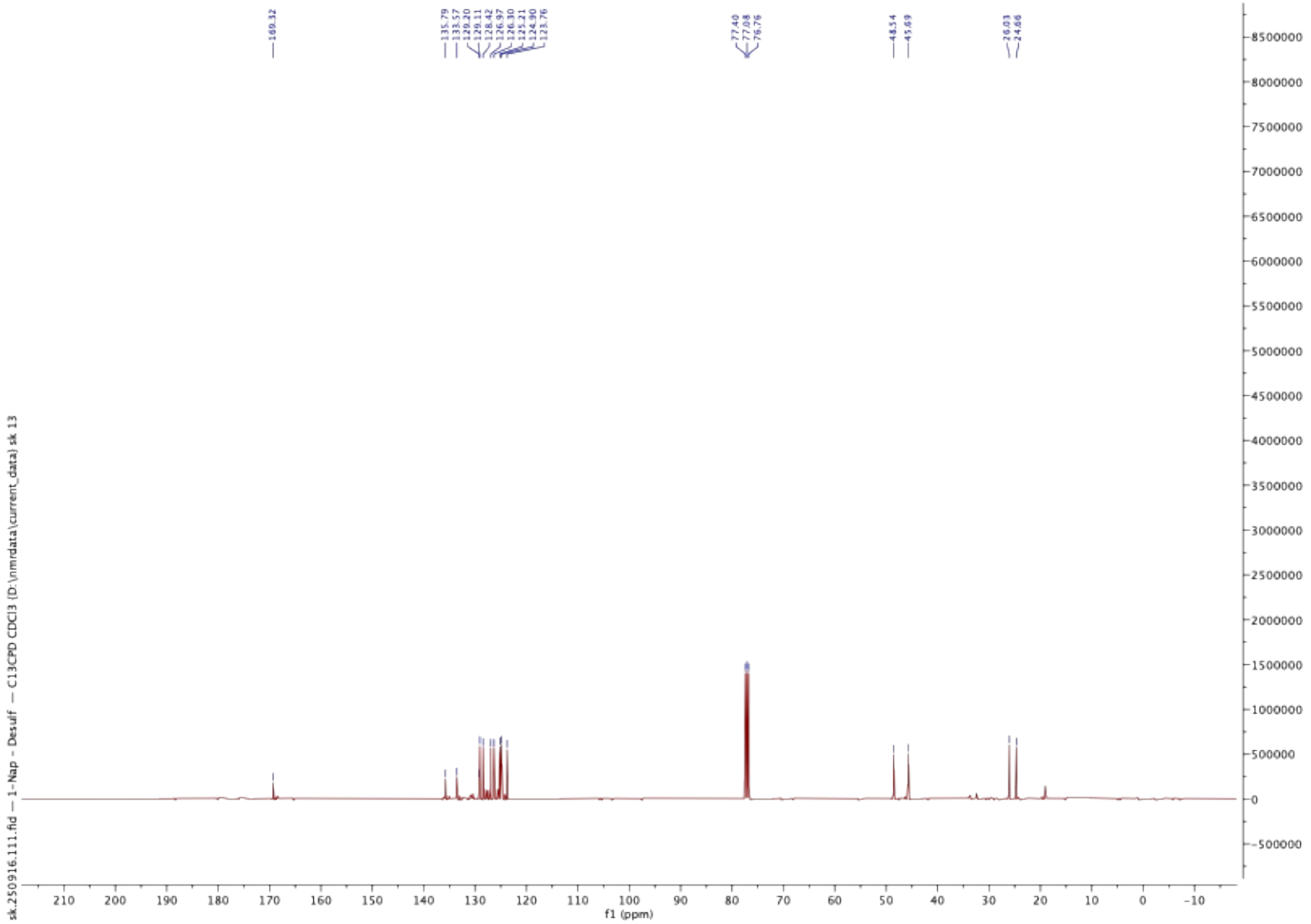
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectrum of **2k**



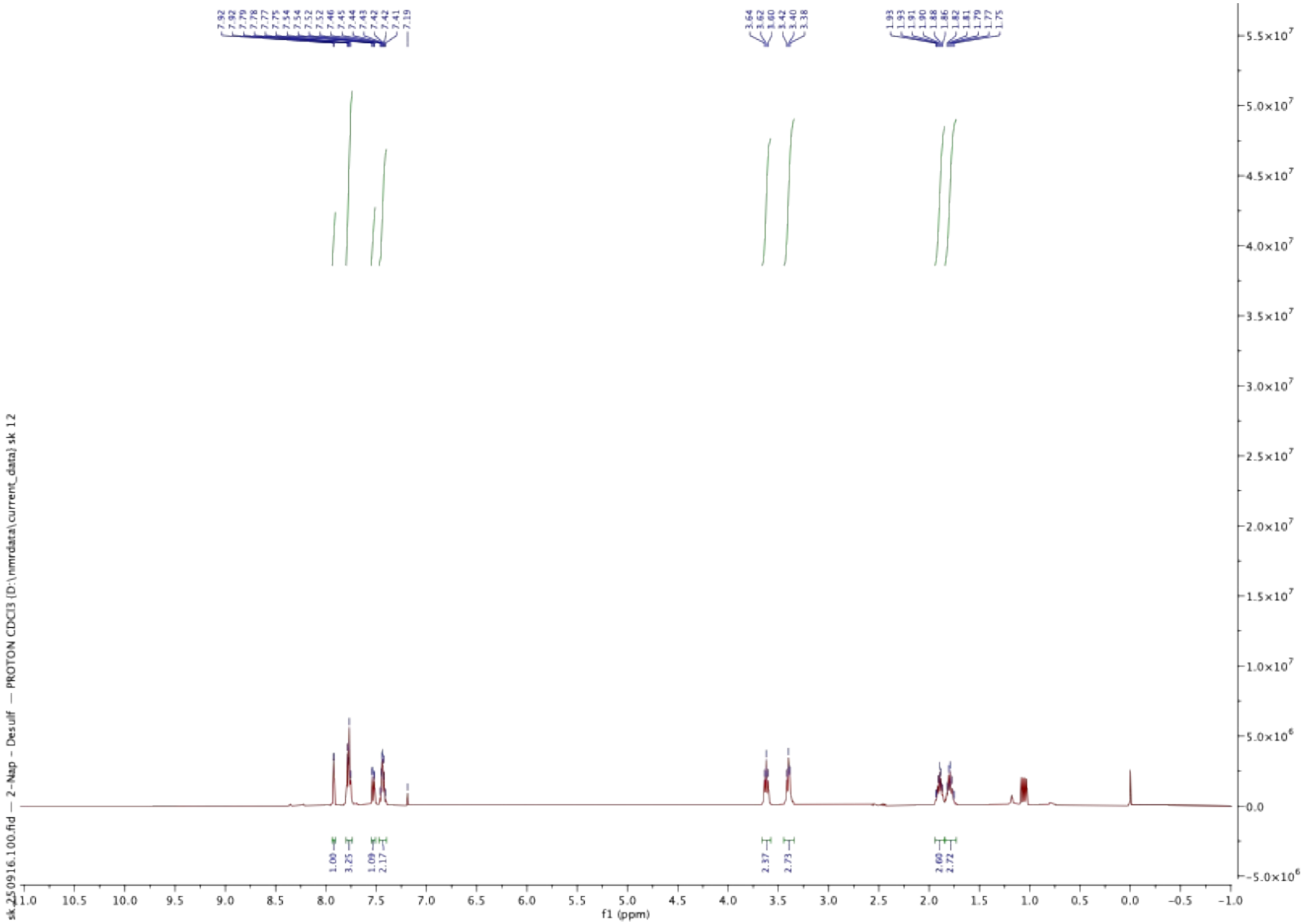
$^{1}H$ (400 MHz, $CDCl_3$) NMR spectrum of **2m**



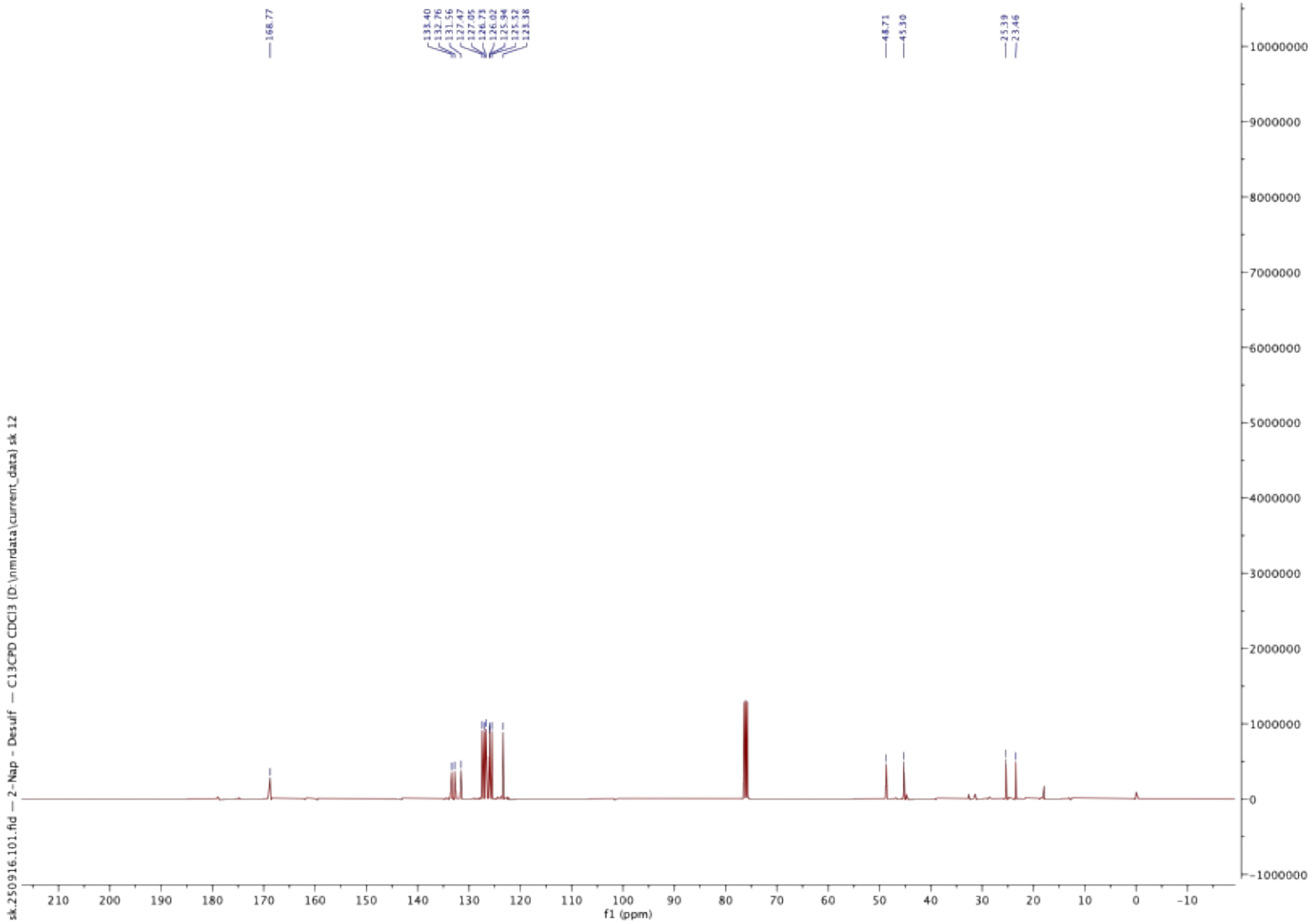
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectrum of **2m**



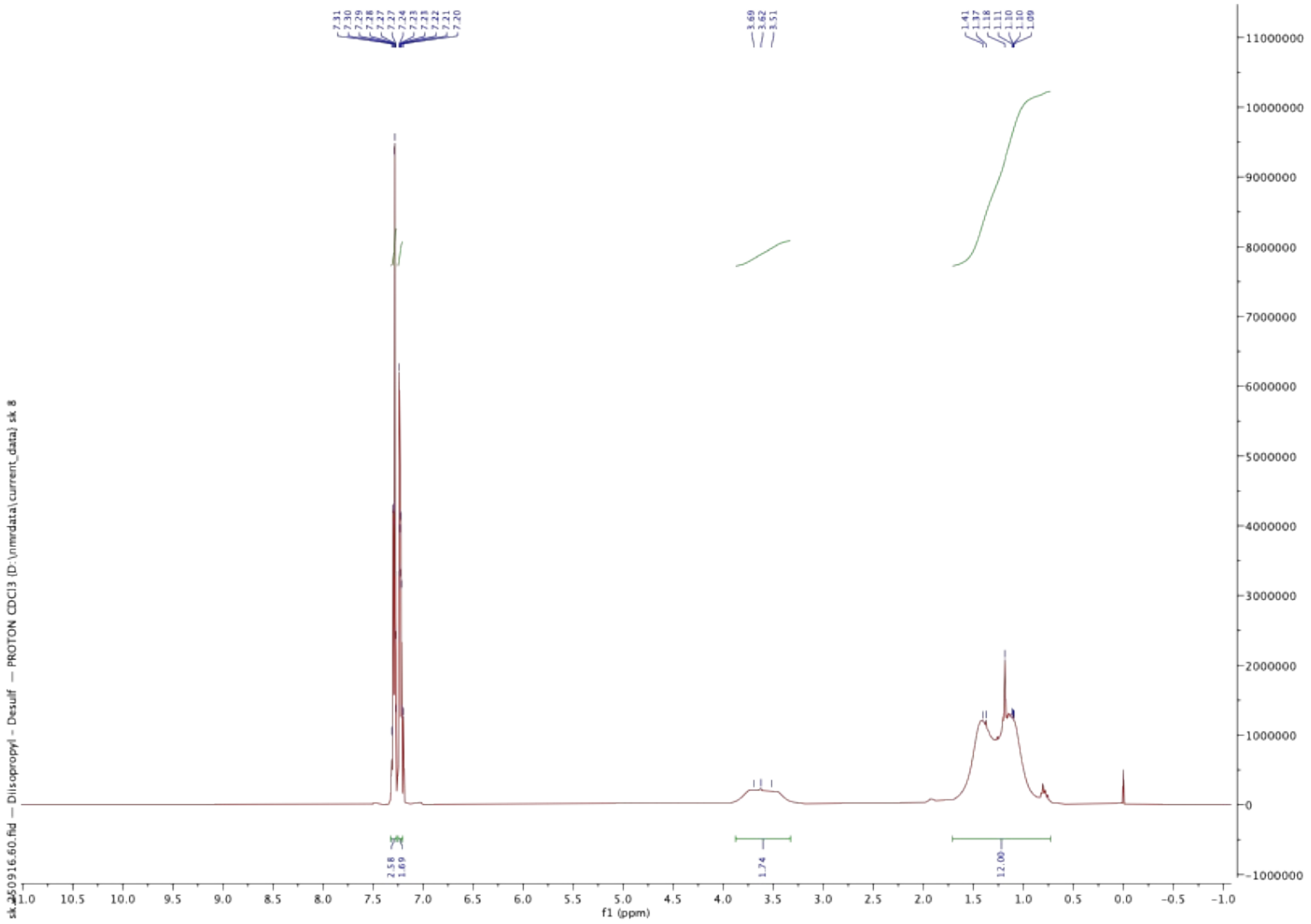
$^1$H (400 MHz, $CDCl_3$) NMR spectrum of **2n**



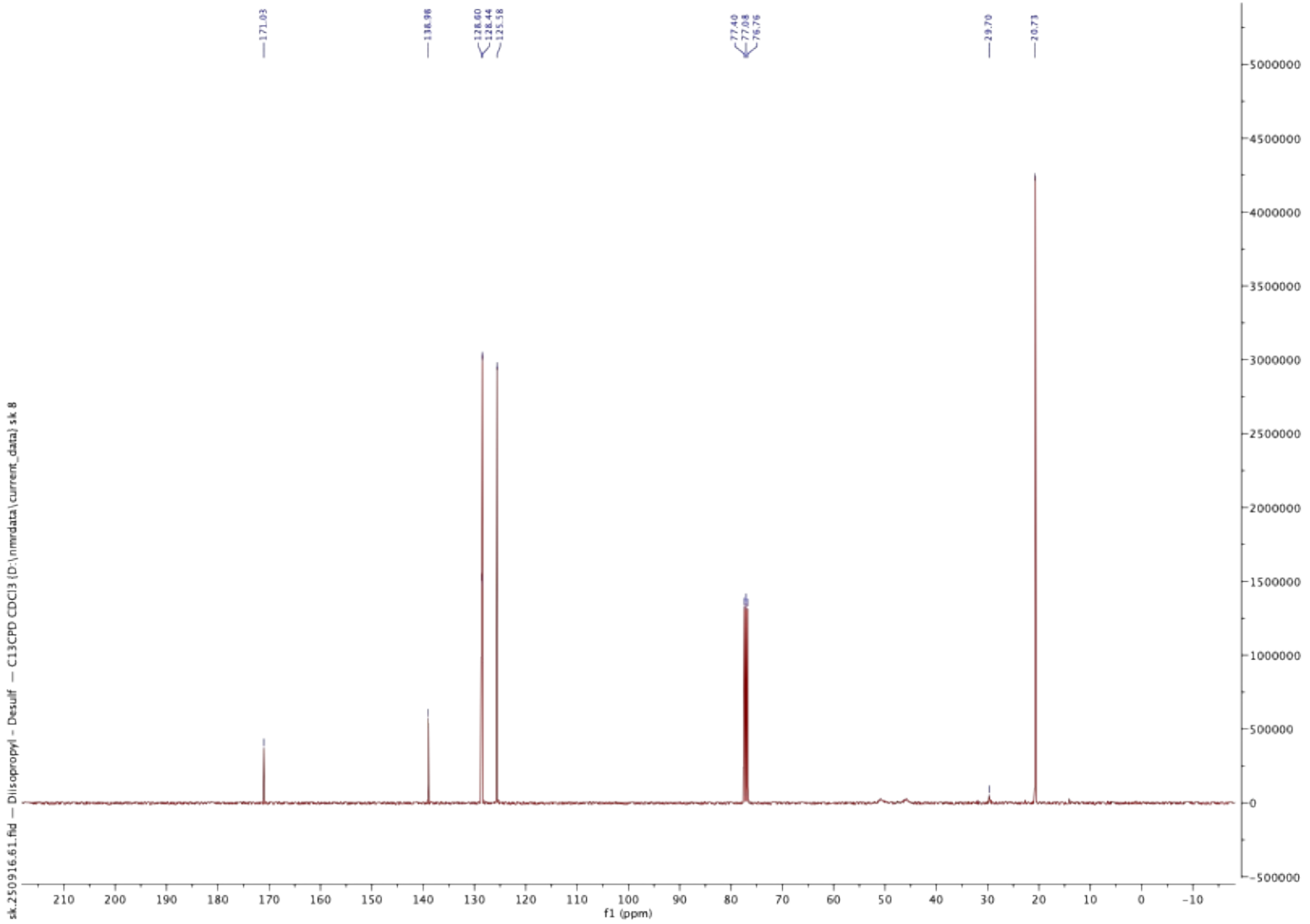
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectrum of **2n**



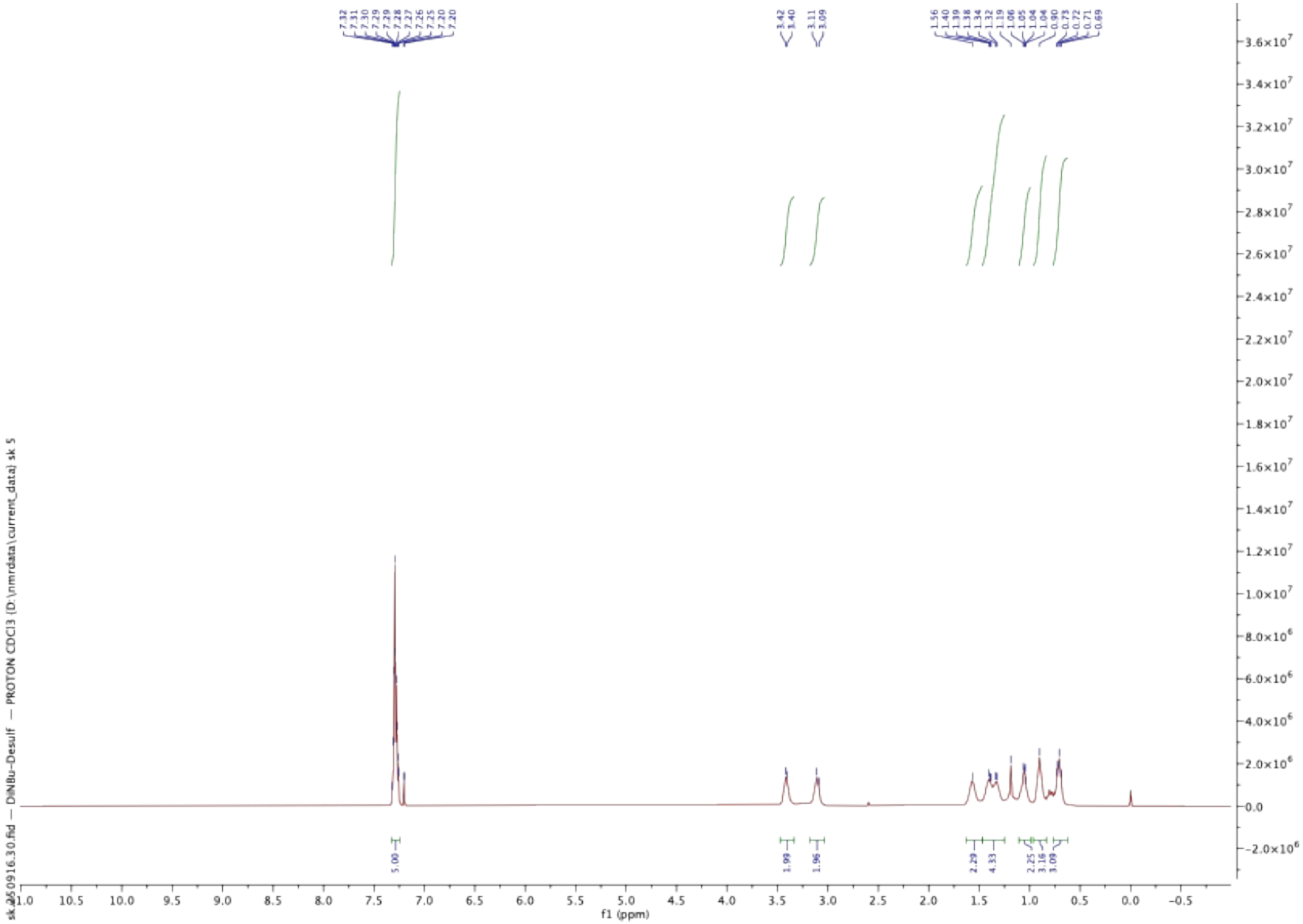
$^{1}H$ (400 MHz, $CDCl_3$) NMR spectrum of **2p**



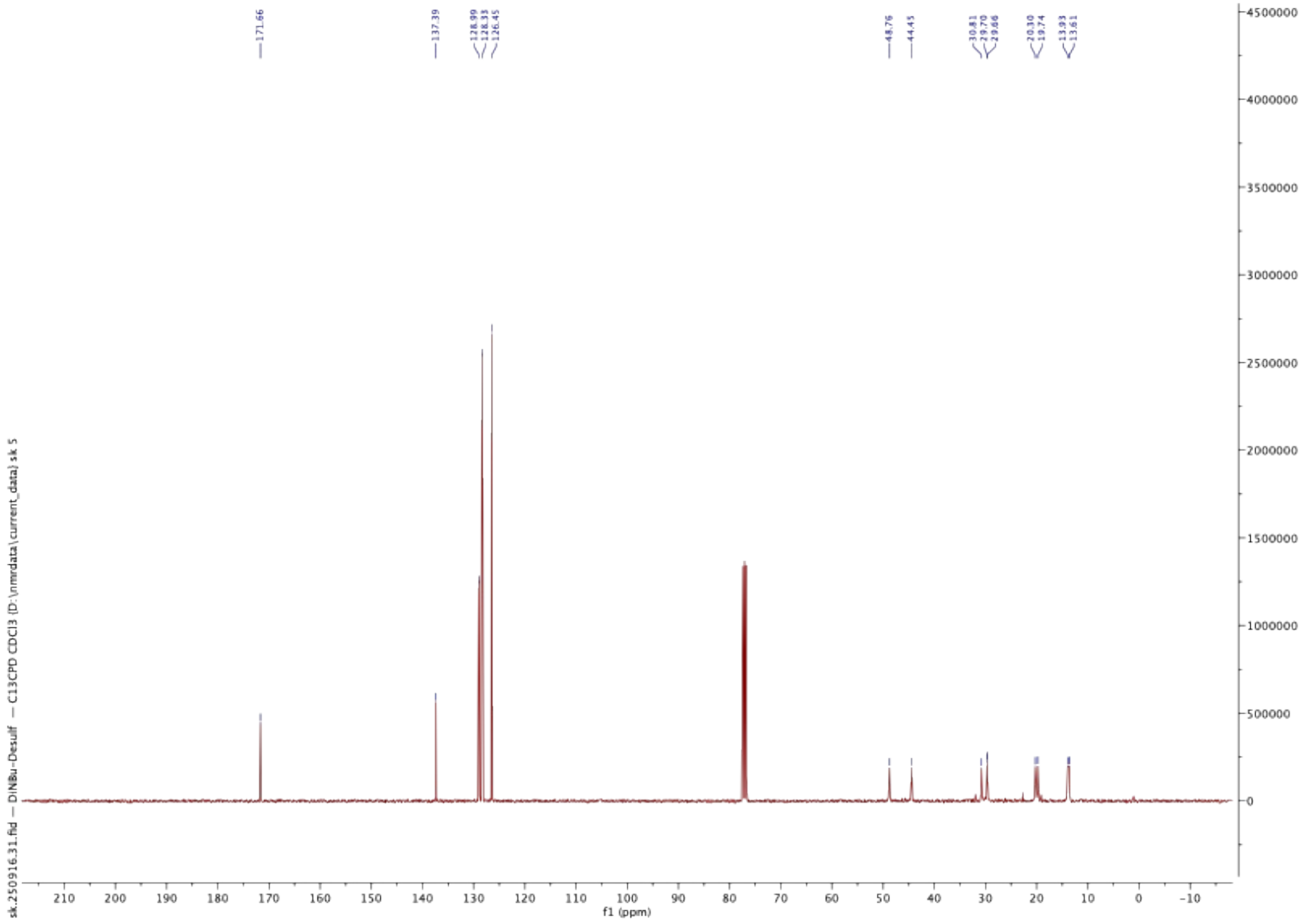
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectrum of **2p**



$^1$H (400 MHz, $CDCl_3$) NMR spectrum of **2q**



$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectrum of **2q**



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