

Supporting Information

Phthalide: a direct building-block towards P,O and P,N hemilabile ligands: Application in the palladium-catalyzed Suzuki-Miyaura cross-coupling of aryl chlorides.

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General Information

All fine chemicals were obtained from Sigma-Aldrich and used as obtained. Dichloromethane was distilled over calcium hydride. Toluene was distilled over sodium metal in the presence of benzophenone indicator. HBr-AcOH (33% by wt) was used as obtained from Sigma Aldrich. ^1H and ^{13}C and ^{31}P NMR spectra were obtained on a 600 MHz Bruker NMR spectrometer. Chemical shifts are reported in units of δ (ppm) and coupling constants (J) are expressed in Hz. Mass spectra were run on a Micromass Quattro Ultima spectrometer fitted with a direct injection probe (DIP) with ionization energy set at 70 eV and HRMS (EI) were performed with a Micromass Q-TOF Ultima spectrometer. Thin layer chromatography (TLC) was run using Macherey-Nagel aluminum-backed plates. Melting points were obtained on an Electronic Research Associates Inc. melting point apparatus corrected against an external calibrant.

Synthesis of 2-((di-tert-butylphosphino)methyl)benzoic acid hydrobromide 2:

Into a flame-dried flask with a stirring bar was added HBr-AcOH (10.0 mL, 33% by wt). A mixture of phthalide (0.500 g) in glacial acetic acid was slowly added to it and the reaction mixture allowed to stir at room temperature for 2 h and 1.5 h at 70 °C. The reaction mixture was stirred overnight. The reaction mixture was poured on ice-water (100.0 mL) to obtain precipitate. The precipitate was filtered and dried under high vacuum to obtain α -bromo-*o*-toluic acid as white solid in 70% yield having spectroscopic properties in accord with the literature (^1H NMR (600 MHz, DMSO) δ 7.89 (m, 1H), 7.59 – 7.56 (m, 2H), 7.45 (m, 2H), 5.08 (s, 2H)).¹ To a flame-dried Schlenk flask with a stirring bar was added the α -bromo-*o*-toluic acid (0.570 g, 2.65 mmol) followed by degassed acetone (25.0 mL). Under an argon atmosphere, di-*t*-butylphosphine (0.490 mL, 2.65 mmol) was added to the flask. The Schlenk flask was then flushed with two times with argon atmosphere and closed under argon atmosphere. The reaction mixture was then heated to reflux overnight. Upon cooling the reaction flask, the acetone was removed under vacuum and diethyl ether (20.0 mL) was added to the flask to obtain 2-((di-tert-butylphosphino)methyl)benzoic acid hydrobromide **2** as a colourless amorphous solid in quantitative yield. Melting point: 205-208 °C. ^1H NMR (600 MHz, CD_2Cl_2) δ 8.22 (d, J = 7.7 Hz, 1H), 7.67 (d, J = 7.7 Hz, 1H), 7.64 (t, J = 7.7 Hz, 1H), 7.53 (d, J = 7.7 Hz, 1H), 7.10 (d, J = 484.71 Hz, 1H), 4.33 (dd, J = 14.9, 6.6 Hz, 2H), 1.52 (s, 9H), 1.50 (s, 9H). ^{31}P NMR (243 MHz, CD_2Cl_2) δ 38.25 (d, J = 484.4 Hz). ^{31}P NMR ^1H decoupled (243 MHz, CD_2Cl_2) δ 38.37. ^{13}C NMR (151 MHz,

CD_2Cl_2) δ 168.4, 133.8, 133.6 (d, J = 6.5 Hz), 133.3, 132.2, 129.7, 129.7, 34.1 (d, J = 33.6 Hz), 28.3, 23.4 (d, J = 39.5 Hz). HRMS: calcd. For $\text{C}_{16}\text{H}_{25}\text{O}_2\text{P}$ $[\text{M}]^+$ 281.1682; found 281.1670.

Synthesis of 2-((di-tert-butylphosphino)methyl)-N,N-diethylbenzamide hydrobromide 3a:

Into a flame-dried flask with a stirring bar was added AlCl_3 (1.3 g). Dichloroethane (5.0 mL) was added to the flask. A mixture of diethylamine (1.95 mL, 2.5 equiv) in dichloroethane (3.0 mL) was added to the flask maintaining the temperature below 25 °C. The reaction mixture was stirred for 30 min at rt. Phthalide (1.0 g, 1.0 equiv) was added to the flask slowly in portions. The reaction mixture was stirred for 50 min at rt. Afterwards ice and water was added to the flask and reaction mixture was stirred for additional 30 min. Reaction mixture was filtered through celite pad and aqueous phase was extracted with dichloromethane, washed with brine and dried to give crude product which was purified using silica-gel flash chromatography (EtOAc : hexanes, gradient elution) to gave *N,N*-diethyl-2-(hydroxymethyl)benzamide as an orange oil.² Yield 88%. ^1H NMR (600 MHz, CDCl_3) δ 7.46 (dd, J = 7.6, 0.7 Hz, 1H), 7.40 (td, J = 7.5, 1.3 Hz, 1H), 7.32 (td, J = 7.5, 1.3 Hz, 1H), 7.24 (dd, J = 7.5, 1.1 Hz, 1H), 4.53 (s, 2H), 3.59 (q, J = 7.1 Hz, 2H), 3.45 (brs, 1H), 3.24 (q, J = 7.1 Hz, 2H), 1.29 (t, J = 7.1 Hz, 3H), 1.10 (t, J = 7.1 Hz, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 171.3, 138.6, 136.0, 129.7, 129.5, 127.4, 125.7, 63.7, 43.4, 39.3, 14.0, 12.7.

Into a flame-dried flask with a stirring bar was added *N,N*-diethyl-2-(hydroxymethyl)benzamide (0.110 g, 0.530 mmol, 1.0 equiv). Freshly distilled dichloromethane (3.0 mL) was added to the flask. The reaction mixture was cooled to 0 °C and PBr_3 (0.250 mL, 2.65 mmol, 5.0 equiv) was added to it. The reaction mixture was stirred at 0 °C for 3 h. Dichloromethane was removed under vacuum and the water (2.0 mL) was added to the flask. The reaction mixture was extracted with ethyl acetate, washed with brine, dried over sodium sulphate. The solvent was evaporated under high vacuum and the crude sample, was obtained as an oil. A sample of the 2-(bromomethyl)-*N,N*-diethylbenzamide so obtained was allowed to dry under high vacuum for 30 min and then dissolved in 1.0 mL degassed acetone whereupon it was transferred to a flame-dried, argon flushed Schlenk flask containing a stirring bar. Additional degassed acetone (2.0 mL) was added to the flask. Under an argon atmosphere, di-*t*-butylphosphine (0.093 mL, 0.504 mmol, 0.95 equiv) was added to the flask. The flask was then vented under vacuum and flushed with argon twice and closed under argon. The reaction mixture was then heated to reflux overnight. Upon cooling the reaction flask, acetone was removed under vacuum and diethyl ether (20.0 mL) was added to the flask. Resultant

colourless crystalline solid was filtered to obtain 2-((di-tert-butylphosphino)methyl)-*N,N*-diethylbenzamidehydrobromide **3a** in quantitative yield. Yield 99%. Melting point: 163-165 °C. ¹H NMR (600 MHz, CDCl₃) δ 8.38 (d, *J* = 484.9 Hz, 1H), 8.24 (d, *J* = 7.8 Hz, 1H), 7.52 (t, *J* = 7.6 Hz, 1H), 7.37 (t, *J* = 7.6 Hz, 1H), 7.24 (d, *J* = 7.6 Hz, 1H), 4.01 (s, 1H), 3.60 (q, *J* = 7.1 Hz, 2H), 3.19 (q, *J* = 7.1 Hz, 2H), 2.62 (s, 1H), 1.56 (s, 9H), 1.54 (s, 9H), 1.29 (t, *J* = 7.1 Hz, 3H), 1.12 (t, *J* = 7.1 Hz, 3H). ¹H NMR (600 MHz, CD₂Cl₂) δ 8.52 (d, *J* = 485.7 Hz, 1H), 8.17 (d, *J* = 7.9 Hz, 1H), 7.54 (td, *J* = 7.7, 1.2 Hz, 1H), 7.43 (t, *J* = 7.7 Hz, 1H), 7.29 (d, *J* = 7.7 Hz, 1H), 4.23-3.93 (brs, 2H), 3.59 (q, *J* = 7.1 Hz, 2H), 3.20 (q, *J* = 7.1 Hz, 2H), 1.56 (s, 9H), 1.53 (s, 9H), 1.29 (t, *J* = 7.1 Hz, 3H), 1.13 (t, *J* = 7.1 Hz, 3H). ³¹P NMR (243 MHz, CDCl₃) δ 36.47 (d, *J* = 483.1 Hz). ³¹P NMR ¹H decoupled (243 MHz, CDCl₃) δ 36.58. ¹³C NMR (151 MHz, CDCl₃) δ 169.9, 136.1 (d, *J* = 5.9 Hz), 132.55 (d, *J* = 5.2 Hz), 130.4, 128.2, 127.2 (d, *J* = 7.6 Hz), 126.5, 43.7, 39.5, 33.8 (d, *J* = 32.9 Hz), 28.1, 19.7 (d, *J* = 37.5 Hz), 14.2, 13.1. HRMS: calcd. For C₁₈H₂₉NOP [M]⁺ 306.2001; found 306.1987 (Loss of one C₂H₅ group).

An identical procedure was followed to obtain ligand **3c** and **3d**.

(2-(hydroxymethyl)phenyl)(piperidin-1-yl)methanone:² Yield 90%. ¹H NMR (600 MHz, CDCl₃) δ 7.47 (d, *J* = 7.5 Hz, 1H), 7.41 (t, *J* = 7.5 Hz, 1H), 7.34 (t, *J* = 7.4 Hz, 1H), 7.25 (d, *J* = 7.5 Hz, 1H), 4.56 (m, 2H), 3.77 (s, 2H), 3.50 (s, 1H), 3.33 (s, 2H), 1.71 (s, 4H), 1.52 (s, 2H). ¹³C NMR (151 MHz, CDCl₃) δ 170.3, 139.1, 135.6, 129.9, 129.6, 127.4, 126.3, 64.1, 48.7, 43.0, 26.6, 25.7, 24.5.

3c: Yield 99%. Melting point: 178-180 °C. ¹H NMR (600 MHz, CDCl₃) δ 8.29 (d, *J* = 483.2 Hz, 1H), 8.18 (d, *J* = 7.8 Hz, 1H), 7.52 (t, *J* = 7.7 Hz, 1H), 7.37 (t, *J* = 7.7 Hz, 1H), 7.25 (d, *J* = 7.7 Hz, 1H), 4.54 (brs, 1H), 3.98 (brs, 1H), 3.62 (brs, 2H), 3.32 (t, *J* = 5.5 Hz, 2H), 1.85-1.64 (m, 6H), 1.58 (s, 9H), 1.56 (s, 9H). ³¹P NMR (243 MHz, CDCl₃) δ 36.62 (d, *J* = 483.0 Hz). ³¹P NMR ¹H decoupled (243 MHz, CDCl₃) δ 36.73. ¹³C NMR (151 MHz, CDCl₃) δ 168.6, 135.5, 132.8 (d, *J* = 5.2 Hz), 130.5, 128.2, 127.9 (d, *J* = 7.9 Hz), 127.0, 48.6, 42.9, 28.1, 26.7, 25.8, 24.3, 19.9 (d, *J* = 37.3 Hz). HRMS: calcd. For C₁₇H₂₅NOP [M]⁺ 290.1678; found 290.1674 (Loss of one ^tBu group).

N-benzyl-2-(hydroxymethyl)-*N*-methylbenzamide:² Yield 94%. Rotamer mixture. Major: ¹H NMR (600 MHz, CDCl₃) δ 7.48 (dd, *J* = 7.5, 1.8 Hz, 1H), 7.45 - 7.40 (m, 3H), 7.39-7.32 (m, 3H), 7.31 (t, *J* = 5.7 Hz, 1H), 7.15 (d, *J* = 7.3 Hz, 1H), 4.81 (s, 2H), 4.60 (s, 2H), 3.67 (brs, 1H), 2.86 (s, 3H). Minor: ¹H NMR (600 MHz, CDCl₃) δ 7.48 (dd, *J* = 7.5, 1.8 Hz, 1H), 7.45 - 7.40 (m, 3H), 7.39-7.32 (m, 3H), 7.31 (t, *J* = 5.7 Hz, 1H), 7.15 (d, *J* = 7.3 Hz, 1H), 4.60

(s, 2H), 4.51 (s, 2H), 3.67 (s, 1H), 3.10 (s, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 172.4, 171.8, 139.2, 139.0, 136.7, 136.1, 135.4, 135.2, 130.1, 130.0, 129.8, 128.9, 128.8, 128.2, 127.8, 127.7, 127.5, 127.5, 126.9, 126.6, 126.3, 64.1, 55.3, 50.6, 36.8, 33.1.

3d: Yield 99%. Melting point: 168-171 °C. Rotamer mixture. Major: ^1H NMR (600 MHz, CDCl_3) δ 8.58 (d, J = 485.9 Hz, 1H), 8.17 (d, J = 7.7 Hz, 1H), 7.58 – 7.49 (m, 1H), 7.43 – 7.27 (m, 6H), 7.09 (d, J = 7.3 Hz, 1H), 4.83 (s, 2H), 3.96 (s, 2H), 2.91 (s, 3H), 1.55 (s, 9H), 1.53 (s, 9H). Minor: ^1H NMR (600 MHz, CDCl_3) δ 8.63 (d, J = 485.9 Hz, 1H), 8.11 (d, J = 7.9 Hz, 1H), 7.58 – 7.49 (m, 1H), 7.43 – 7.27 (m, 6H), 7.09 (d, J = 7.3 Hz, 1H), 4.53 (s, 2H), 3.74 (s, 2H), 3.23 (s, 3H), 1.57 (s, 9H), 1.54 (s, 9H). ^{31}P NMR ^1H decoupled (243 MHz, CDCl_3) δ 34.83, 34.26. ^{31}P NMR (243 MHz, CDCl_3) δ 34.70 (d, J = 485.38 Hz), 34.13 (d, J = 485.38 Hz). ^{13}C NMR (151 MHz, CDCl_3) Major: ^{13}C NMR (151 MHz, CDCl_3) δ 170.0, 136.6, 136.3, 135.3 (d, J = 5.20 Hz), 132.5, 130.7, 128.9, 128.8, 128.3, 128.2, 127.8, 127.3, 126.9, 50.9, 37.4, 33.8 (d, J = 32.71 Hz), 28.0, 19.3 (d, J = 38.68 Hz). HRMS: calcd. For $\text{C}_{23}\text{H}_{31}\text{NOP}$ $[\text{M}]^+$ 368.2161; found 368.2143 (Loss of one methyl group).

Synthesis of 2-((di-tert-butylphosphino)methyl)-*N,N*-diisopropylbenzamidehydrobromide **3b**:

Into a flame-dried flask with a stirring bar was added *N,N*-diisopropylbenzamide (**I**) (1.0 g, 4.87 mmol, 1.0 equiv). Freshly distilled THF (4.0 mL) was added to the flask. *s*-BuLi (3.82 mL, 1.1 equiv, 1.4 M solution in cyclohexane) was added to the flask at -78 °C. After 60 min, DMF (1.80 mL, 19.4 mmol, 4.0 equiv) was added to the flask and stirred for 30 min at rt. To the reaction mixture 15.0 mL water was added and reaction mixture was extracted with diethyl ether, washed with brine and solvent was dried over sodium sulphate. The solvent was removed under vacuum to give off white solid which was recrystallized from pentane to give light yellow solid. The crude aldehyde was dissolved in 15.0 mL methanol at 0 °C and NaBH_4 (0.185 g, 4.87 mmol, 1.0 equiv) was added by portions. The reaction mixture was stirred at 0 °C for 30 min, followed by overnight at rt. The reaction mixture was quenched and solvent was evaporated under vacuum. The crude reaction mixture was extracted with ethyl acetate, washed with brine and dried over sodium sulphate. The solvent evaporation and a flash column afforded 2-(hydroxymethyl)-*N,N*-diisopropylbenzamide.³ Yield 70%. ^1H NMR (600 MHz, CDCl_3) δ 7.45 (d, J = 7.6 Hz, 1H), 7.45 (d, J = 7.6 Hz, 1H), 7.39 (t, J = 7.5 Hz, 1H), 7.39 (t, J = 7.5 Hz, 1H), 7.33 (t, J = 7.5 Hz, 1H), 7.19 (d, J = 7.5 Hz, 1H), 7.19 (d, J = 7.5 Hz, 1H), 4.67 (d, J = 11.7 Hz, 1H), 4.45 (d, J = 12.0 Hz, 1H), 3.92 – 3.80 (m, 1H), 3.63 (d, J = 29.0 Hz, 1H), 3.61 –

3.53 (m, 1H), 3.61 – 3.50 (m, 1H), 1.59 (m, 6H), 1.16 (m, 6H). ^{13}C NMR (151 MHz, CDCl_3) δ 171.3, 138.3, 137.6, 129.9, 129.1, 127.5, 125.0, 64.0, 51.3, 46.1, 20.9, 20.6, 20.4, 20.3.

Into a flame-dried flask with a stirring bar was added *N,N*-diisopropyl-2-(hydroxymethyl)benzamide (0.103 g, 0.437 mmol, 1.0 equiv). Freshly distilled dichloromethane (3.0 mL) was added to the flask. The reaction mixture was cooled to 0 °C and PBr_3 (0.206 mL, 2.12 mmol, 5.0 equiv) was added to it. The reaction mixture was stirred at 0 °C for 3 h. Dichloromethane was removed under vacuum and the water (2.0 mL) was added to the flask. The reaction mixture was extracted with ethyl acetate, washed with brine, dried over sodium sulphate. The solvent was evaporated under high vacuum and the crude sample, was obtained as an oil. Crude sample of 2-(bromomethyl)-*N,N*-diisopropylbenzamide allowed to dry under high vacuum for 30 min and then dissolved in 1.0 mL degassed acetone where upon it was added to a flame-dried, argon flushed Schlenk flask with a stirring bar. Additional degassed acetone (2.0 mL) was added to the flask. Under argon atmosphere, di-*t*-butylphosphine (0.076 mL, 0.415 mmol, 0.95 equiv) was added to the flask. The Schlenk flask was then flushed with two times with argon atmosphere and closed under argon atmosphere. The reaction mixture was then heated to reflux overnight. Upon cooling the reaction flask, acetone was removed under vacuum and diethyl ether (20.0 mL) was added to the flask to obtain 2-((di-*tert*-butylphosphino)methyl)-*N,N*-diisopropylbenzamidehydrobromide **3b**. Yield 99%. Melting point: 172-175 °C. ^1H NMR (600 MHz, CDCl_3) δ 8.39 (d, J = 480.54 Hz, 1H), 8.28 (d, J = 7.6 Hz, 1H), 7.42 (t, J = 7.5 Hz, 1H), 7.28 (t, J = 7.5 Hz, 1H), 7.08 (d, J = 7.5 Hz, 1H), 4.50-4.33 (m, 1H, P-CH), 3.51 (sept, J = 6.0 Hz, 2H, isopropyl CH's), 3.45 – 3.28 (m, 1H, P-CH), 1.63-1.31 (m, 24H), 1.11-0.95 (m, 6H). ^{31}P NMR ^1H decoupled (243 MHz, CDCl_3) δ 36.18. ^{31}P NMR (243 MHz, CDCl_3) δ 36.08 (d, J = 483.1 Hz). ^{13}C NMR (151 MHz, CDCl_3) δ 169.8, 137.6, 132.4, 130.0, 128.5, 126.1, 125.5, 51.1, 46.1, 34.1 (d, J = 32.3 Hz), 33.5 (d, J = 33.2 Hz), 21.4, 20.9, 20.4, 20.2, 19.8 (d, J = 37.2 Hz). HRMS: calcd. For $\text{C}_{22}\text{H}_{38}\text{NOP} [\text{M}]^+$ 363.2703; found 363.2691

General procedure for Suzuki-Miyaura coupling reaction:

Into an oven dried Schlenk flask equipped with a magnetic stirring stir bar were added under argon the aryl halide (0.040 g, 0.355 mmol), boronic acid (0.065 g, 0.426 mmol), $\text{Pd}_2(\text{dba})_3$ (0.0065 g, 2 mol%), ligand **3c** (0.0045 g, 3 mol%), and K_3PO_4 (0.226 g, 1.065 mmol, Aldrich, ReagentPlus, 99%). The flask was capped, evacuated, and flushed with argon three times. Toluene (2.5 ml) was introduced and the reaction mixture was immersed in a pre-heated oil bath at the indicated temperature for 110 °C for 12 h. (Table 3, entry 2). The reaction mixture was then

diluted with ethyl acetate, filtered through silica and the solvent was removed at reduced pressure. The crude product was then purified by column chromatography on silica gel to get pure biphenyl (**6b**).

Biphenyl 6a: ^1H NMR (600 MHz, CDCl_3) δ 7.64 (dd, J = 8.3, 1.2 Hz, 4H), 7.52 – 7.46 (m, 4H), 7.42 – 7.36 (m, 2H). ^{13}C NMR (151 MHz, CDCl_3) δ 141.2, 128.7, 127.2, 127.1.⁴

4-Methoxybiphenyl 6b: ^1H NMR (600 MHz, CDCl_3) δ 7.47 (dd, J = 7.5, 0.9 Hz, 2H), 7.45 (d, J = 8.7 Hz, 2H), 7.33 (t, J = 7.5 Hz, 2H), 7.22 (t, J = 7.5 Hz, 1H), 6.90 (d, J = 8.7 Hz, 2H), 3.76 (s, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 158.1, 139.8, 132.7, 127.6, 127.1, 125.7, 125.6, 113.1, 54.3.⁴

4-Methylbiphenyl 6c: ^1H NMR (600 MHz, CDCl_3) δ 7.63 (dd, J = 8.3, 1.2 Hz, 2H), 7.55 (d, J = 8.1 Hz, 2H), 7.48 (t, J = 7.7 Hz, 2H), 7.37 (t, J = 7.7 Hz, 1H), 7.30 (d, J = 8.1 Hz, 2H), 2.45 (s, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 141.2, 138.4, 137.0, 129.5, 128.7, 127.0, 127.0, 21.1.⁴

1-(biphenyl-4-yl)ethanone 6d: ^1H NMR (600 MHz, CDCl_3) δ 7.94 (d, J = 8.4 Hz, 2H), 7.59 (d, J = 8.4 Hz, 2H), 7.4 (dd, J = 7.0, 1.5 Hz, 2H), 7.38 (dd, J = 7.34 Hz, 2H), 7.31 (dd, J = 7.4, 1.5 Hz, 1H), 2.54 (s, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 197.7, 145.7, 139.8, 135.8, 128.9, 128.9, 128.2, 127.2, 127.2, 26.6.⁴

4-nitrobiphenyl 6e: ^1H NMR (600 MHz, CDCl_3) δ 8.33 (d, J = 8.8 Hz, 2H), 7.77 (d, J = 8.8 Hz, 2H), 7.66 (dd, J = 7.2, 1.3 Hz, 2H), 7.56 – 7.50 (m, 2H), 7.48 (dd, J = 7.2, 1.2 Hz, 1H). ^{13}C NMR (151 MHz, CDCl_3) δ 146.6, 146.0, 137.7, 128.1, 127.8, 126.7, 126.3, 123.0.⁵

2-methylbiphenyl 6f: ^1H NMR (600 MHz, CDCl_3) δ 7.46 (m, 2H), 7.39 (m, 3H), 7.35 – 7.26 (m, 4H), 2.33 (s, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 142.0, 141.9, 135.3, 130.3, 129.8, 129.2, 128.0, 127.2, 126.7, 125.7, 20.4.⁶

2-methoxybiphenyl 6g: ^1H NMR (600 MHz, CDCl_3) δ 7.45 (dd, J = 8.2, 1.2 Hz, 2H), 7.35 – 7.30 (m, 2H), 7.26 – 7.22 (m, 3H), 6.95 (td, J = 7.5, 1.1 Hz, 1H), 6.91 (dd, J = 8.5, 0.8 Hz, 1H), 3.73 (s, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 155.4, 137.5, 129.8, 128.5, 127.5, 126.9, 125.8, 119.7, 110.2, 54.5.⁶

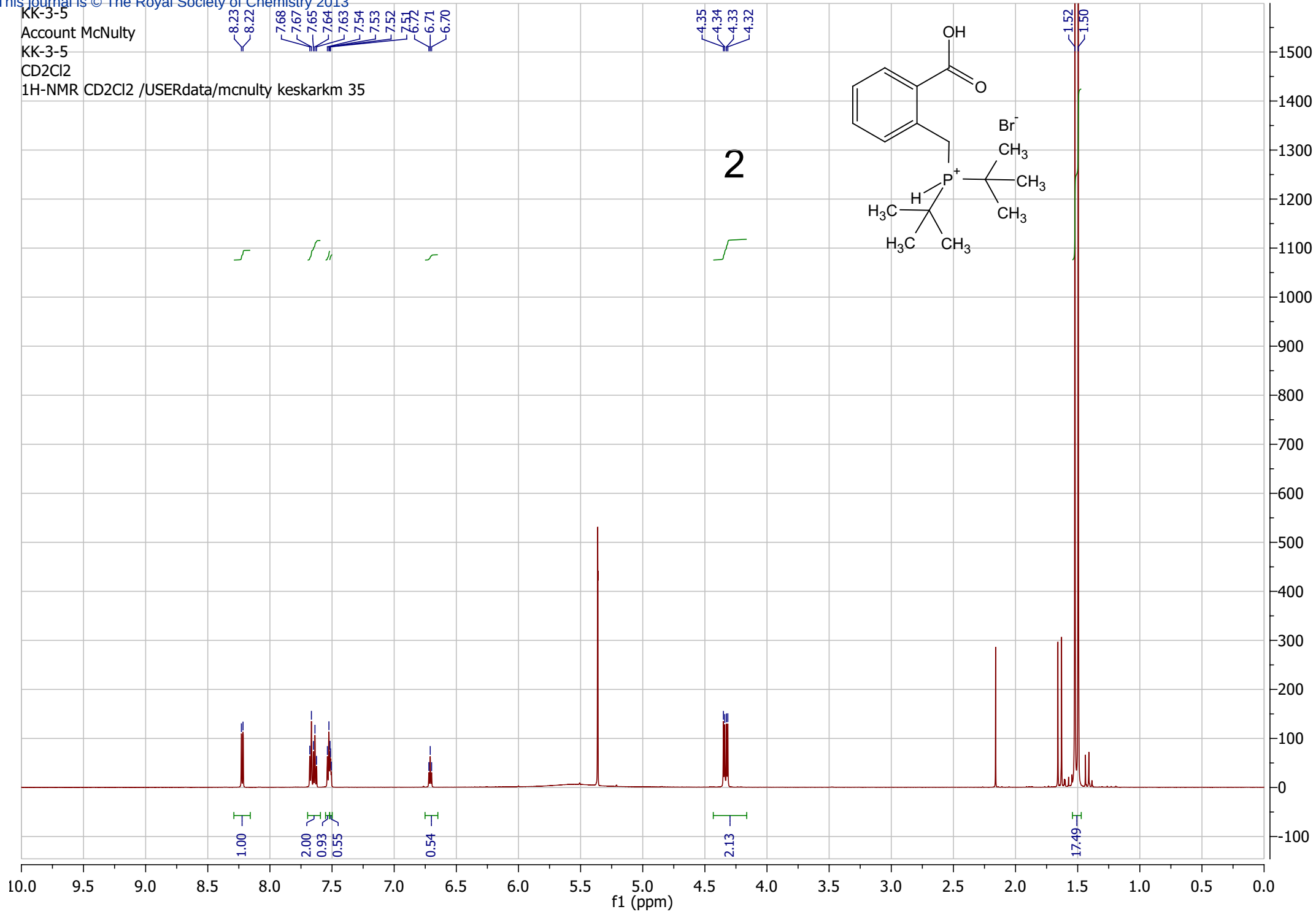
4,4'-dimethoxybiphenyl 6h: ^1H NMR (600 MHz, CDCl_3) δ 7.40 (d, J = 8.8 Hz, 4H), 6.88 (d, J = 8.8 Hz, 4H), 3.76 (s, 6H). ^{13}C NMR (151 MHz, CDCl_3) δ 157.6, 126.7, 113.1, 54.3.⁷

4-methoxy-4'-methylbiphenyl **6i**: ^1H NMR (600 MHz, CDCl_3) δ 7.43 (d, J = 8.8 Hz, 2H), 7.37 (d, J = 7.8 Hz, 2H), 7.14 (d, J = 7.8 Hz, 2H), 6.88 (d, J = 8.8 Hz, 2H), 3.76 (s, 3H), 2.30 (s, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 157.9, 136.9, 135.3, 132.7, 128.4, 126.9, 125.5, 113.1, 54.2, 20.0.⁷

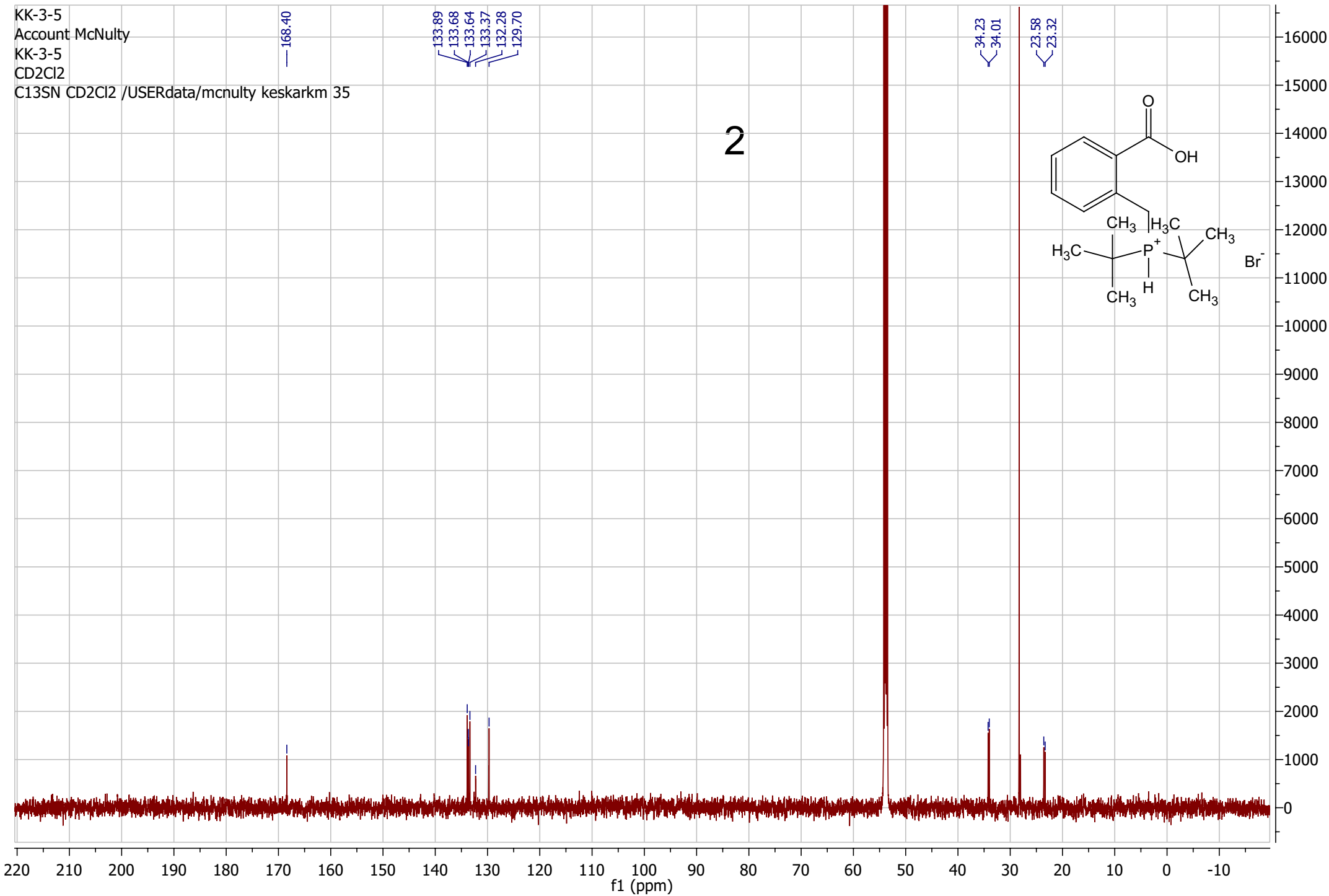
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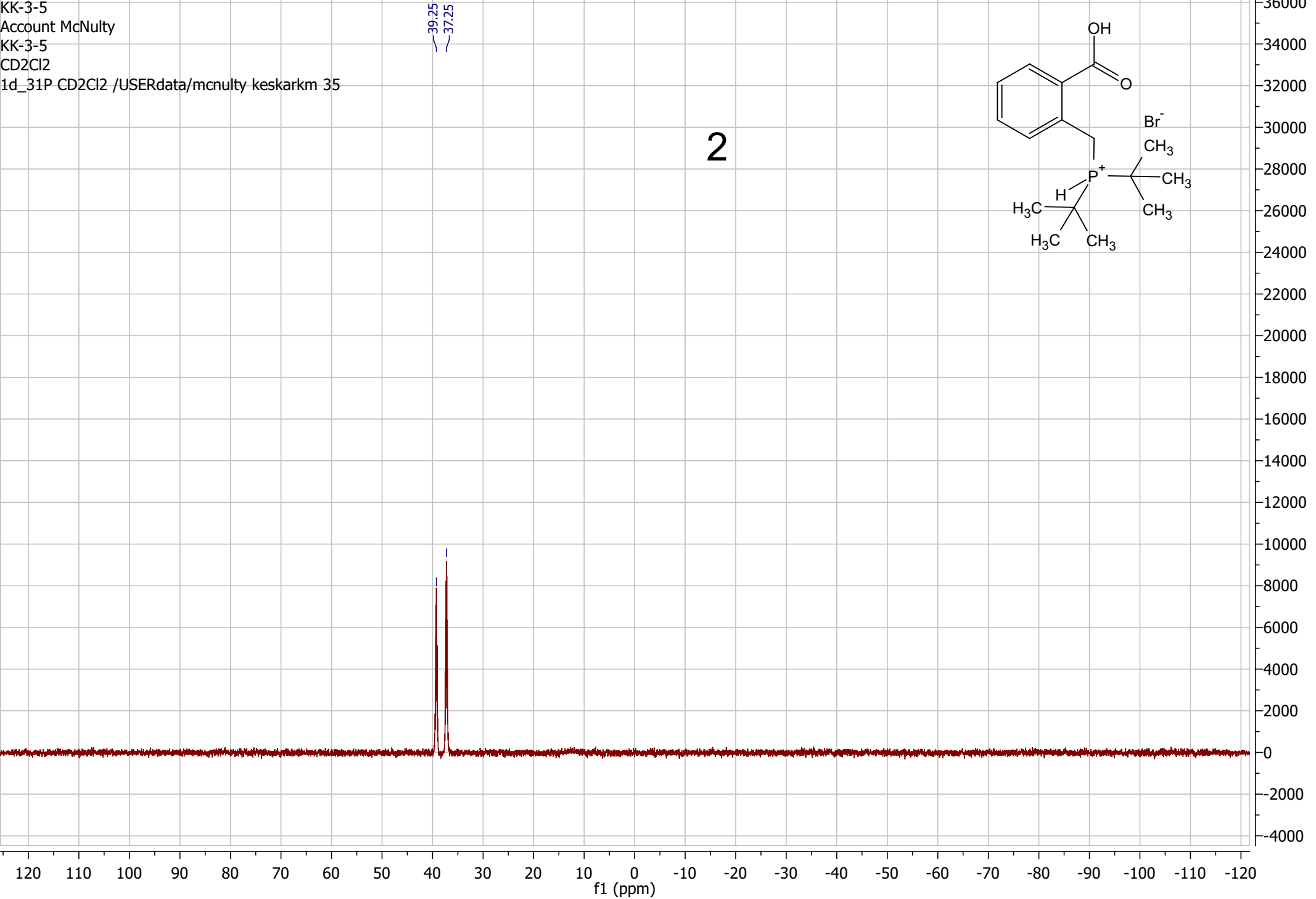
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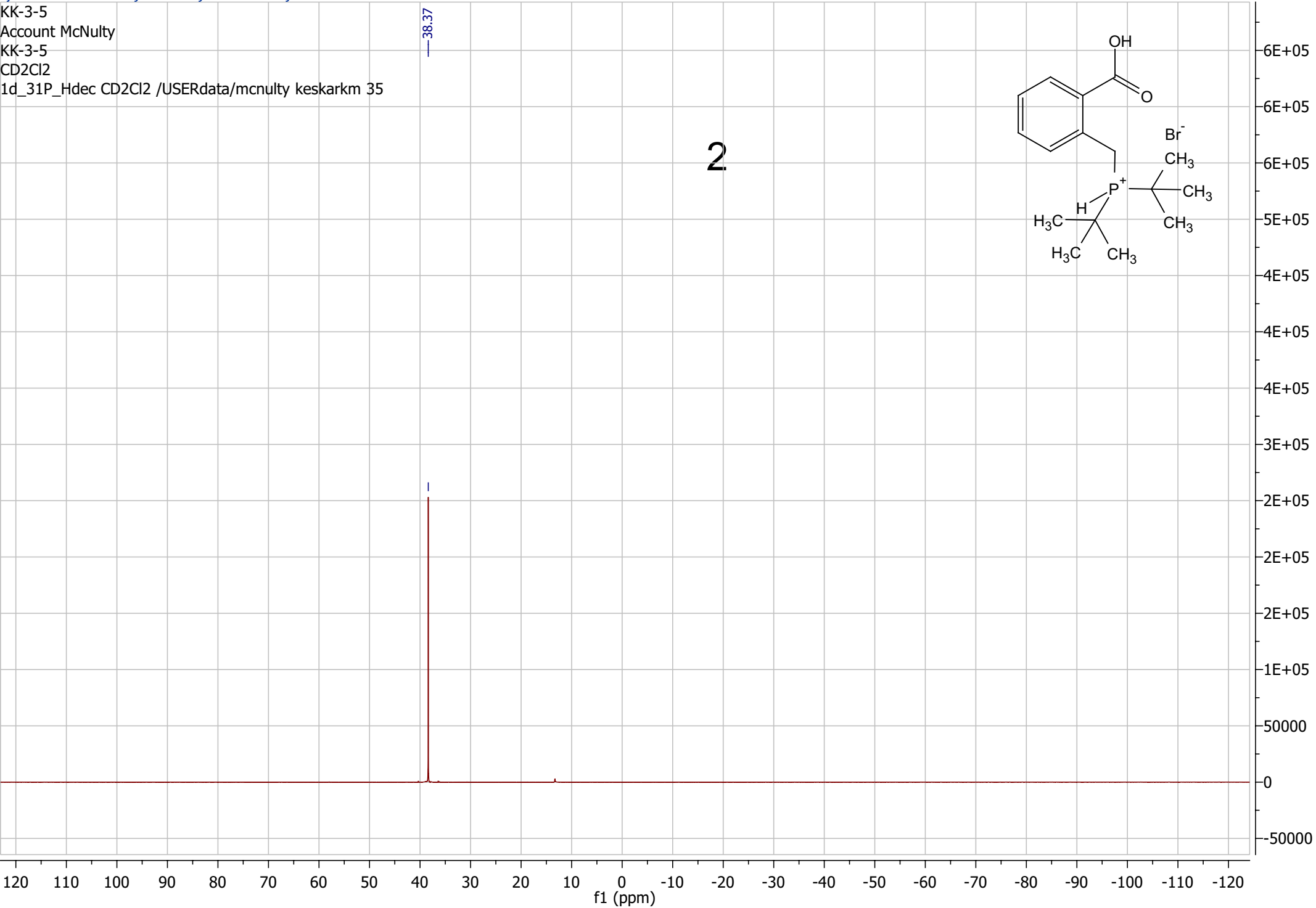
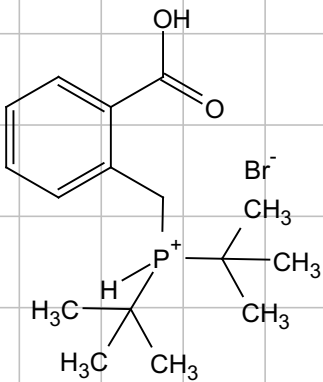
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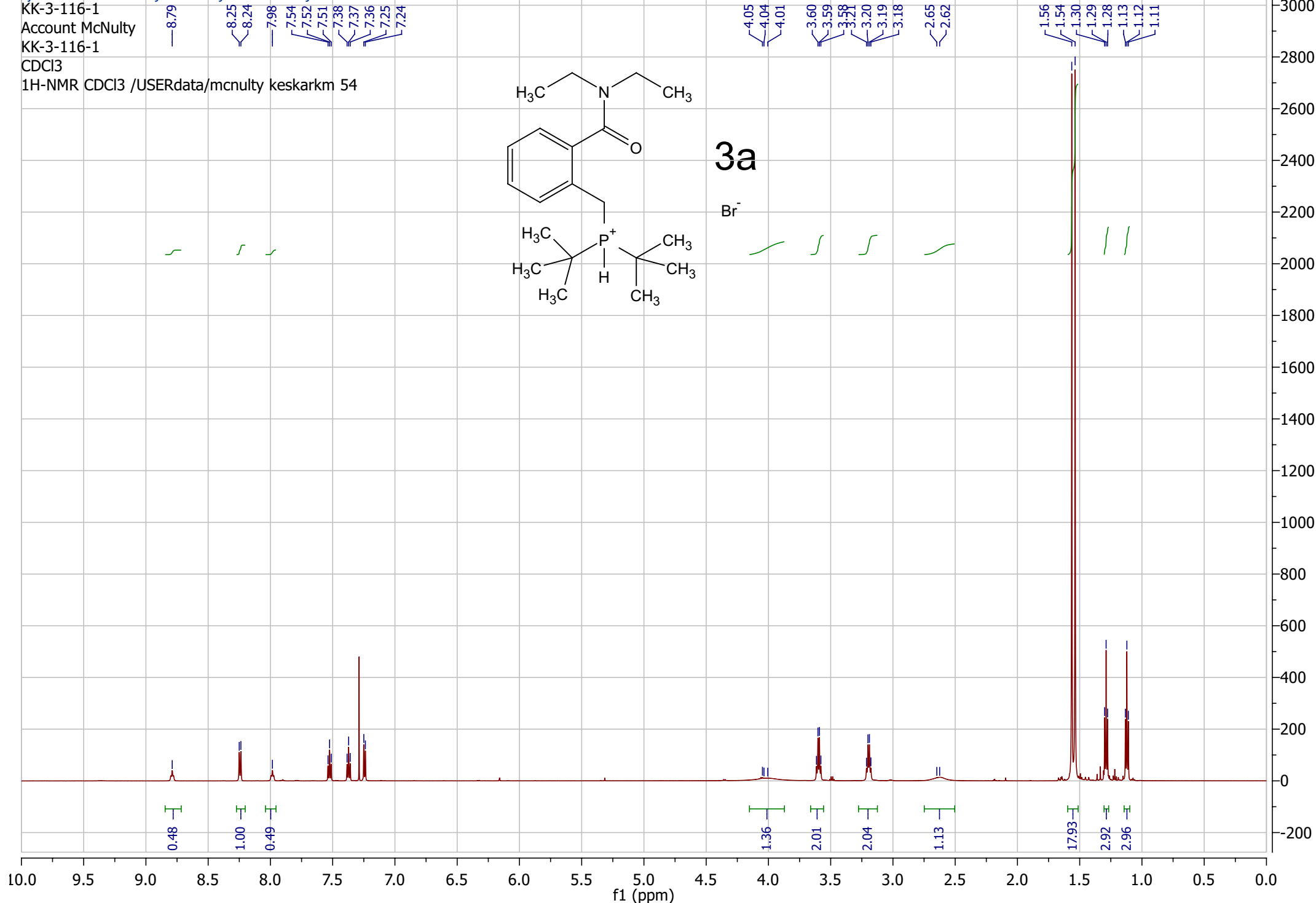
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38.37

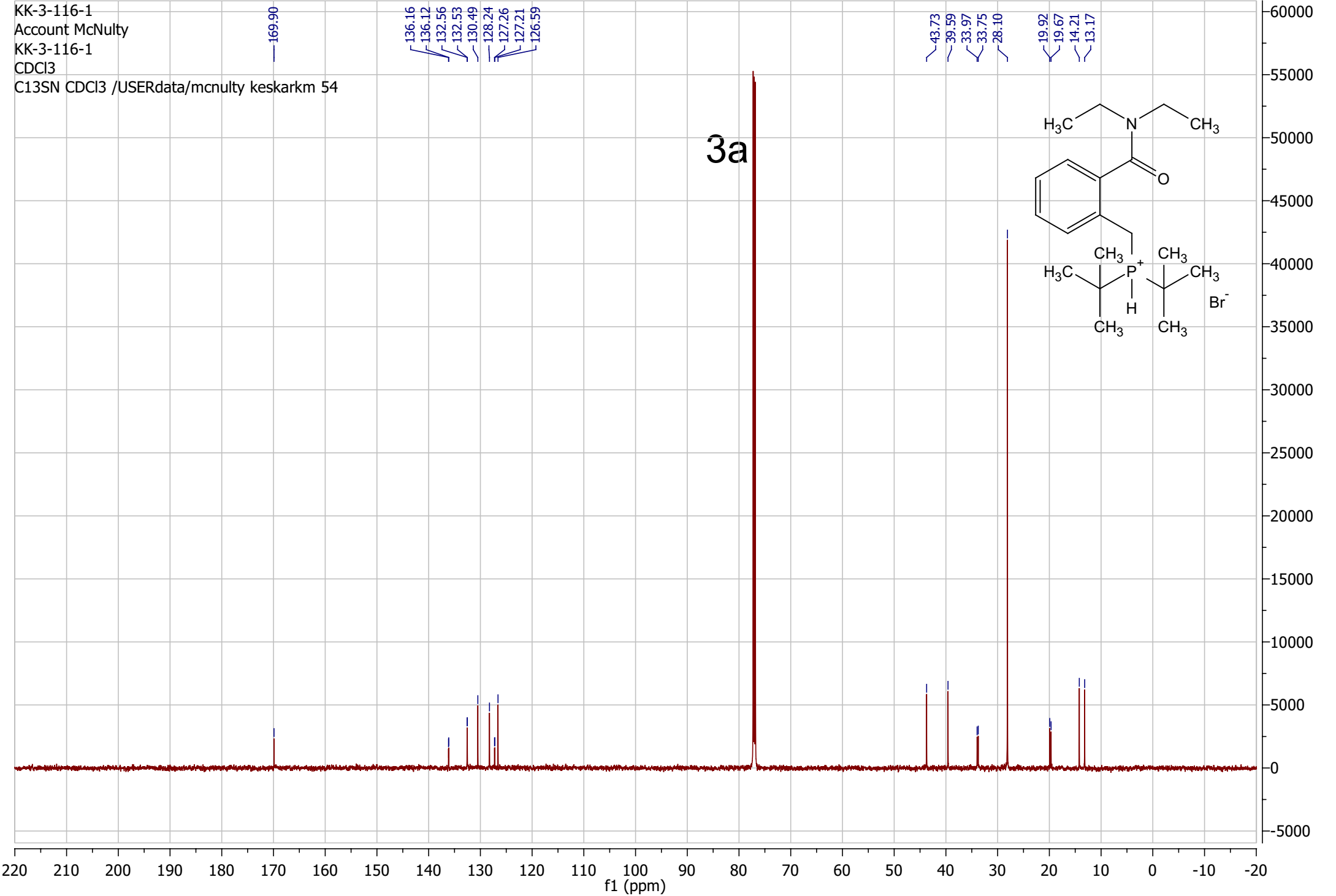
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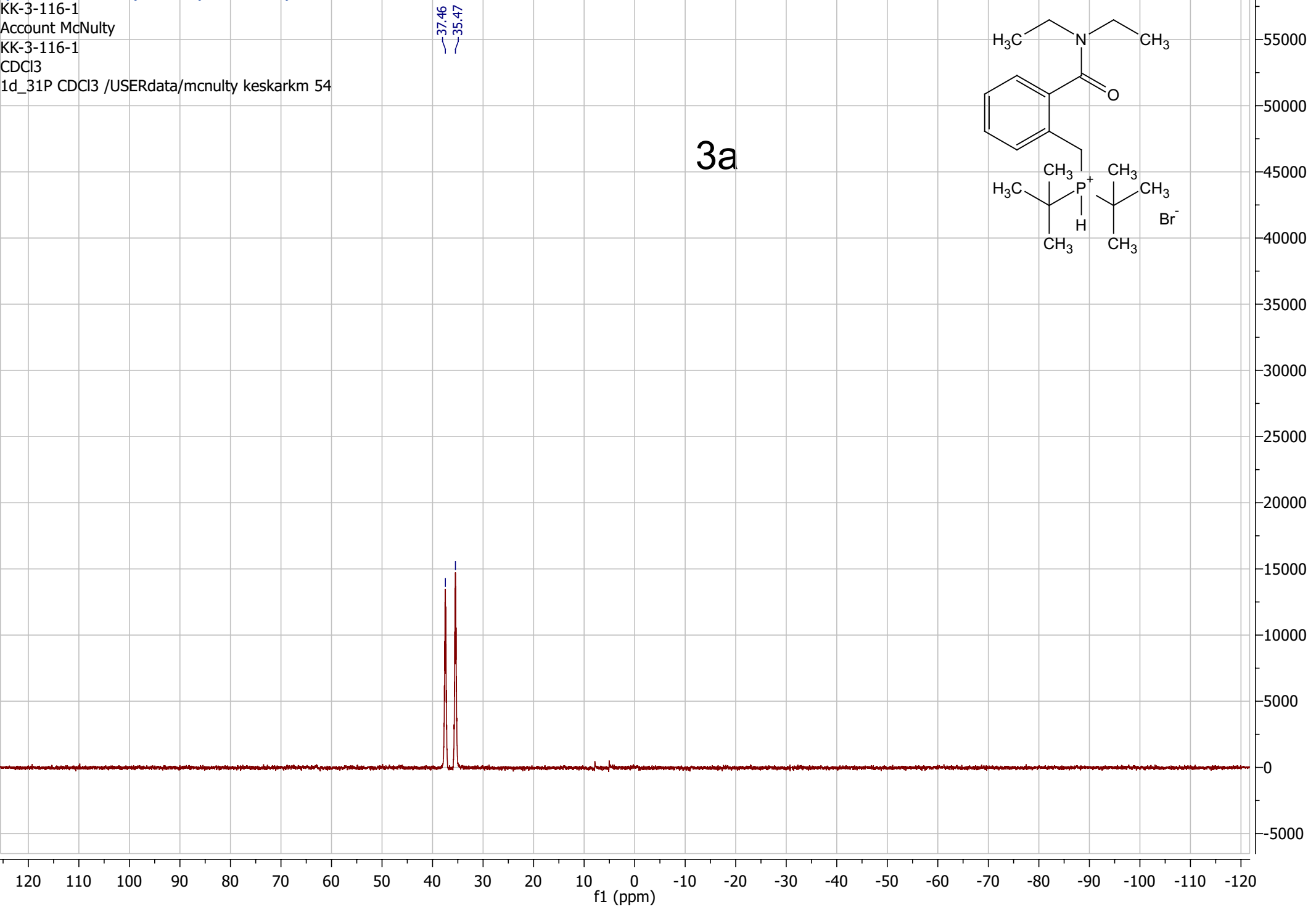
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CDCl3
1H-NMR CDCl3 /USERdata/mcnulty keskarkm 54



KK-3-116-1
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KK-3-116-1
CDCl3
C13SN CDCl3 /USERdata/mcnulty keskarkm 54



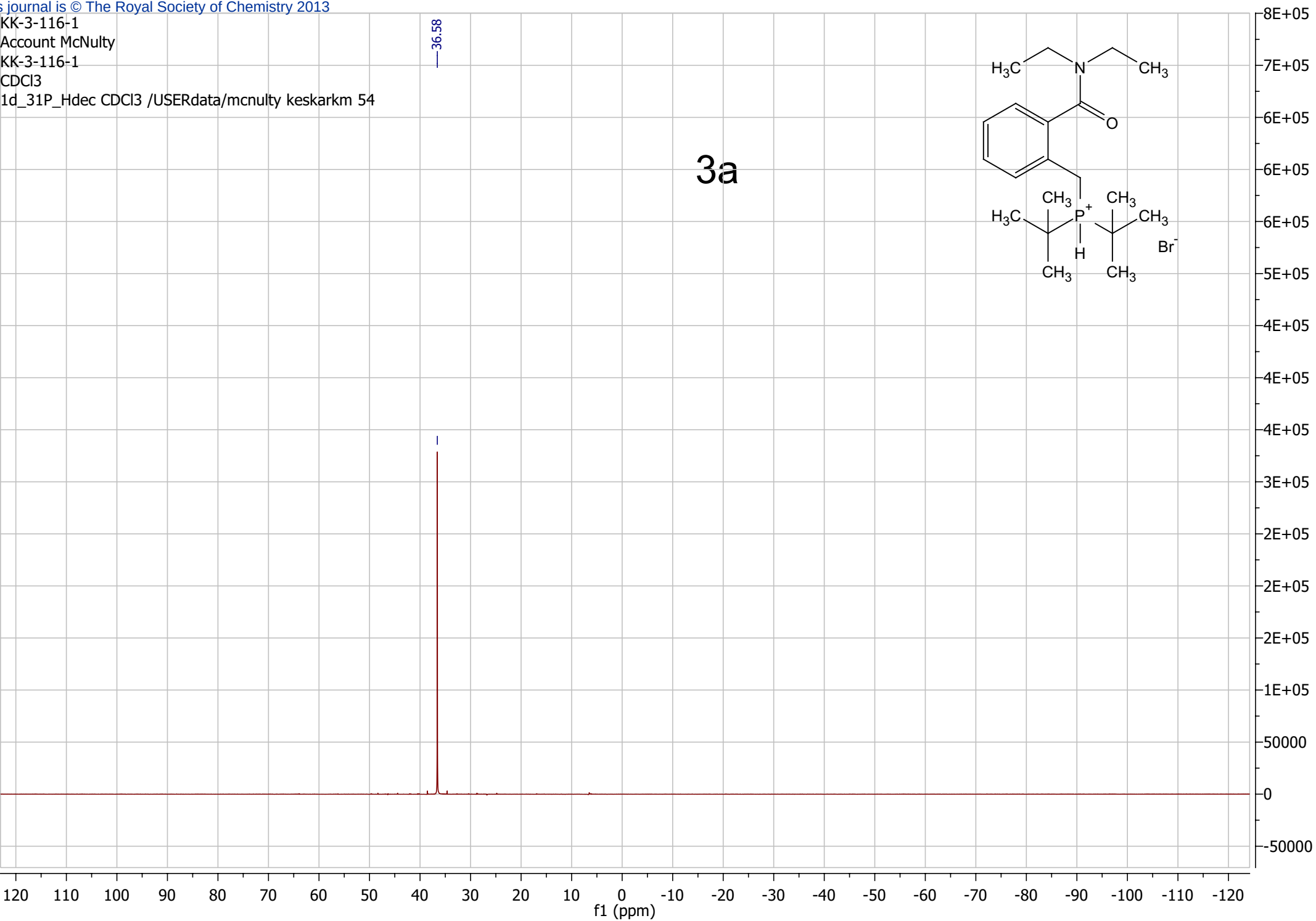
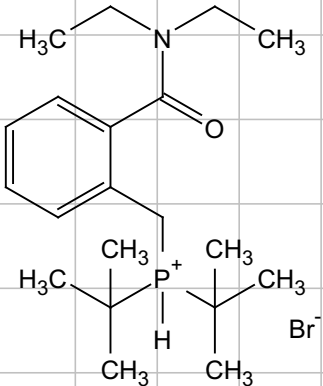
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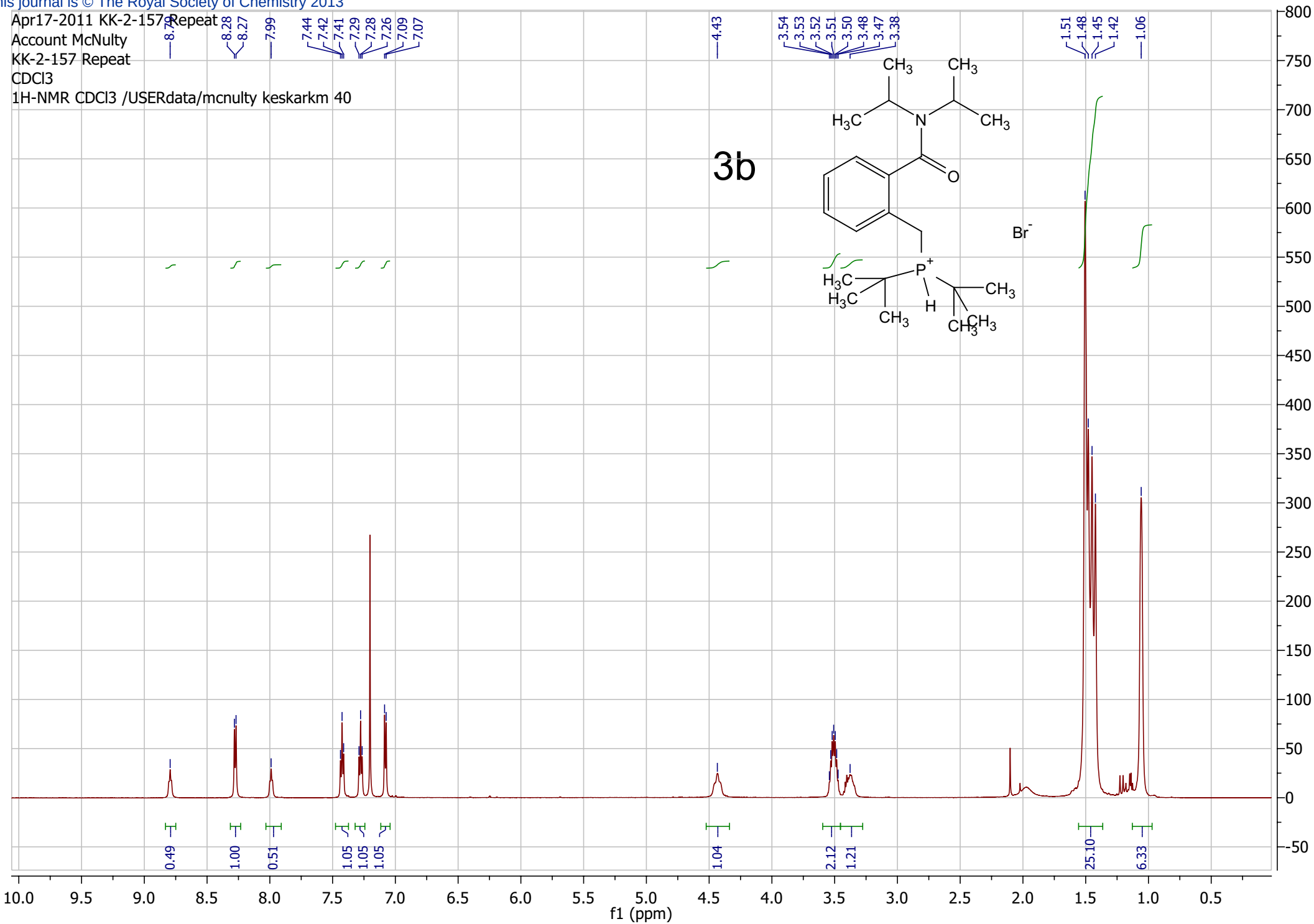
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36.58

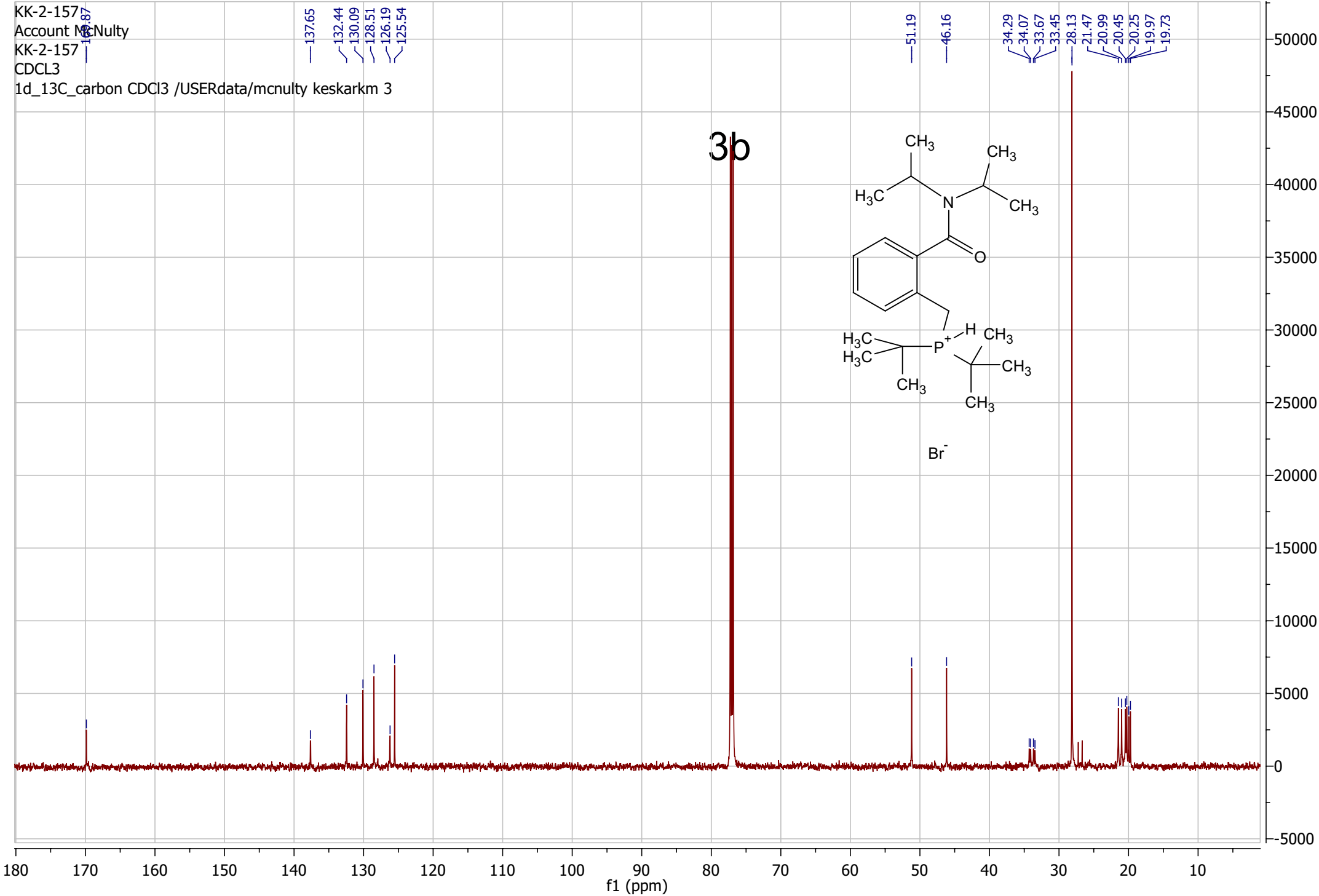
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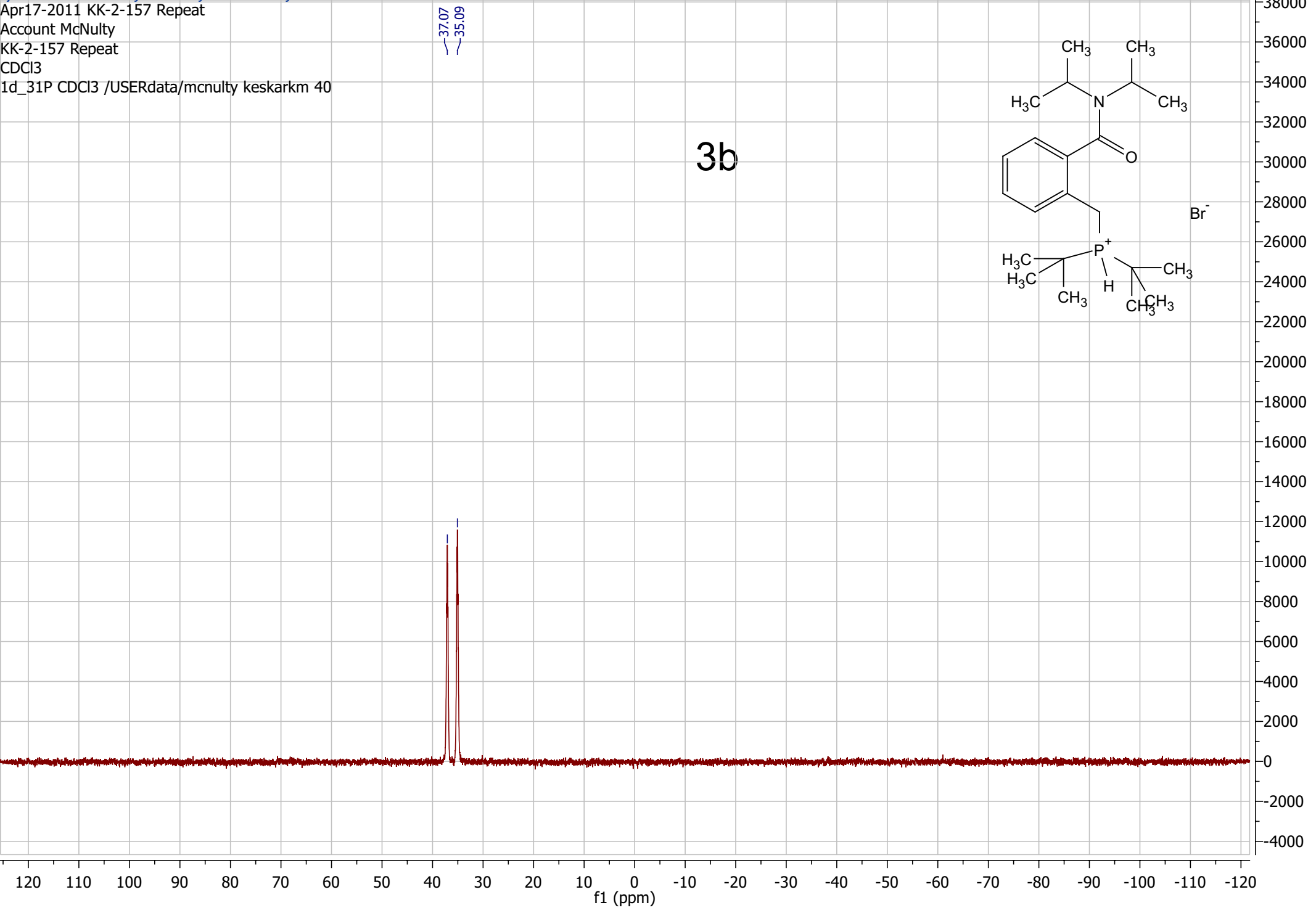
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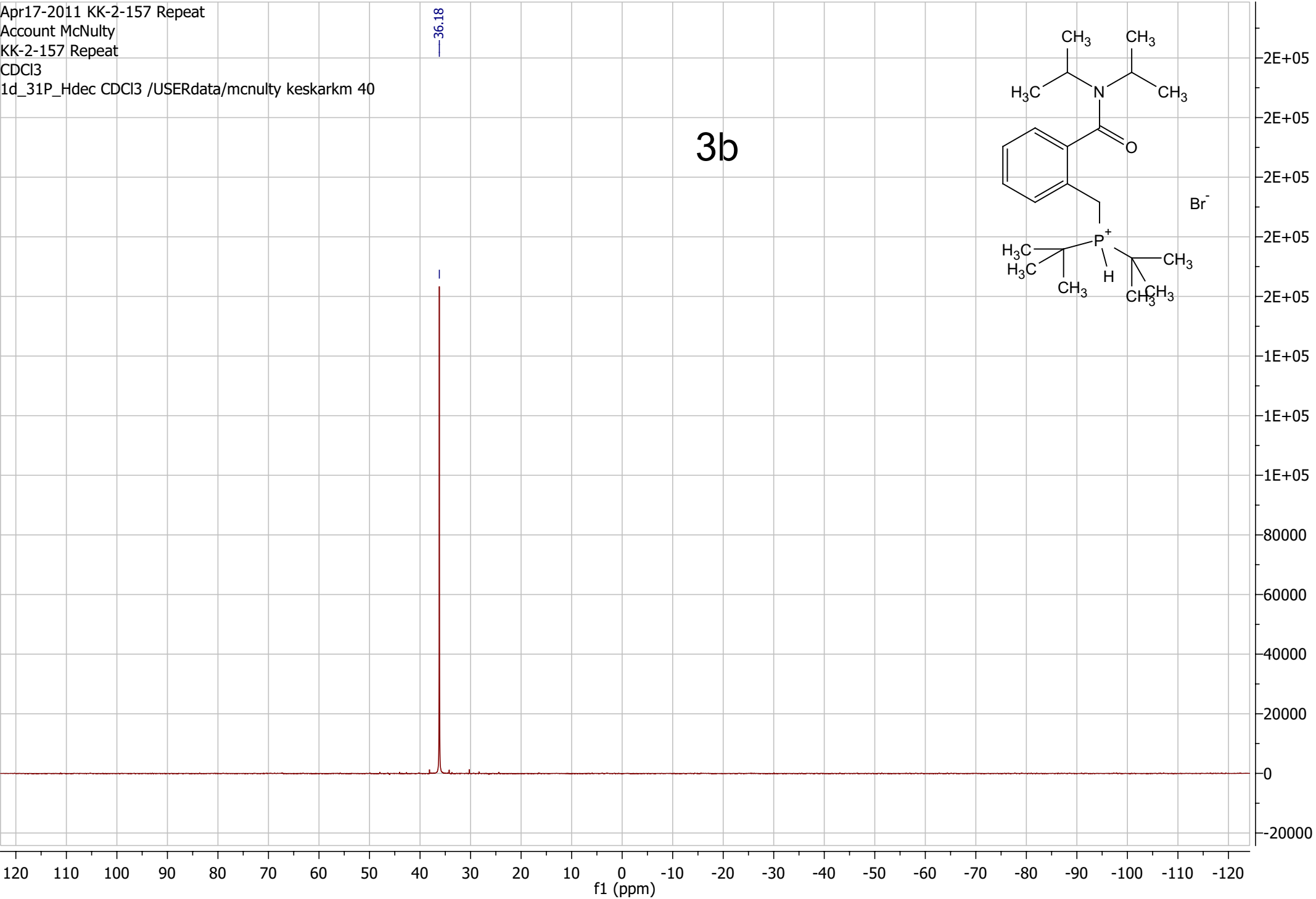
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1d_13C_carbon CDCl3 /USERdata/mcnulty keskarkm 3



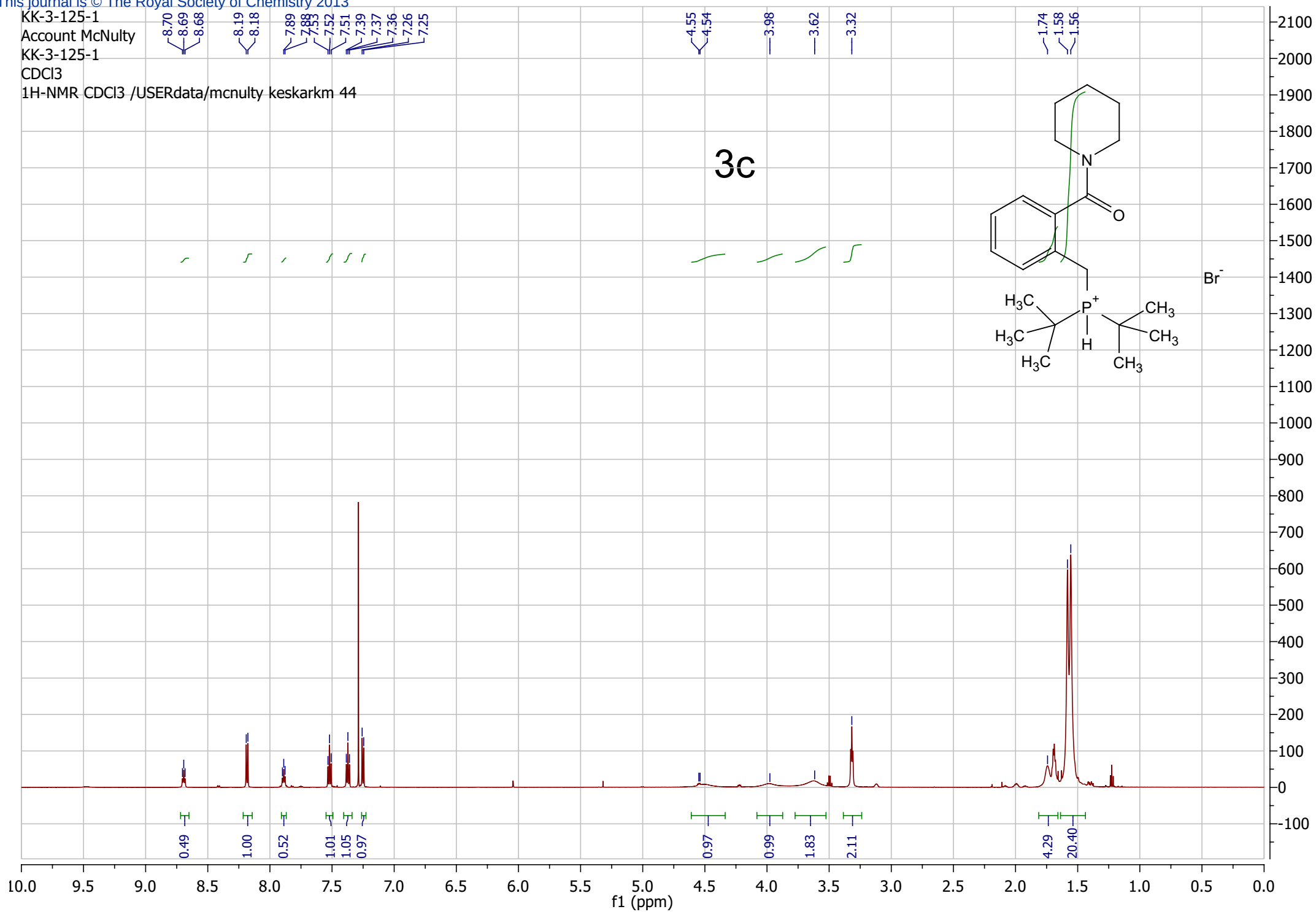
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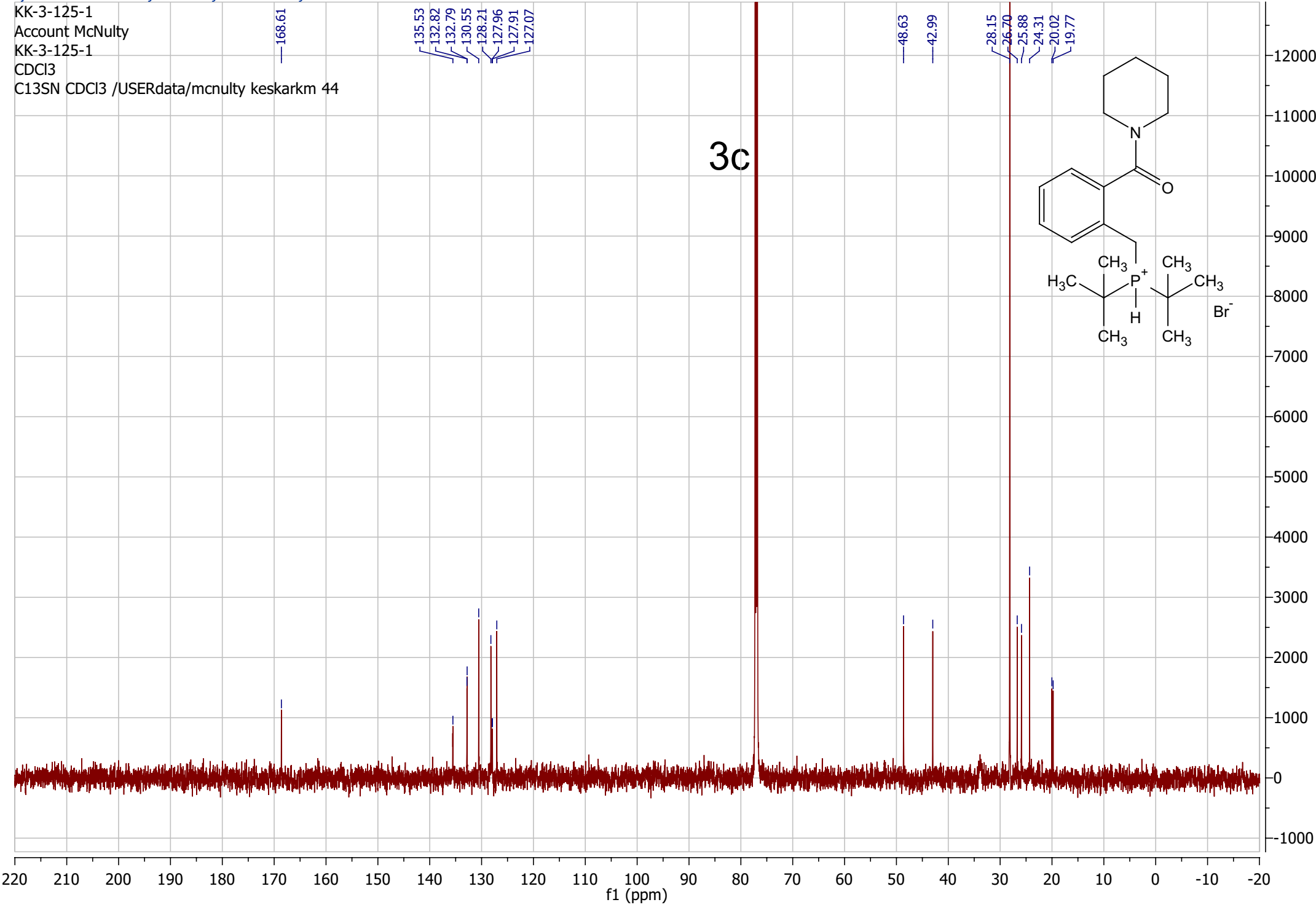
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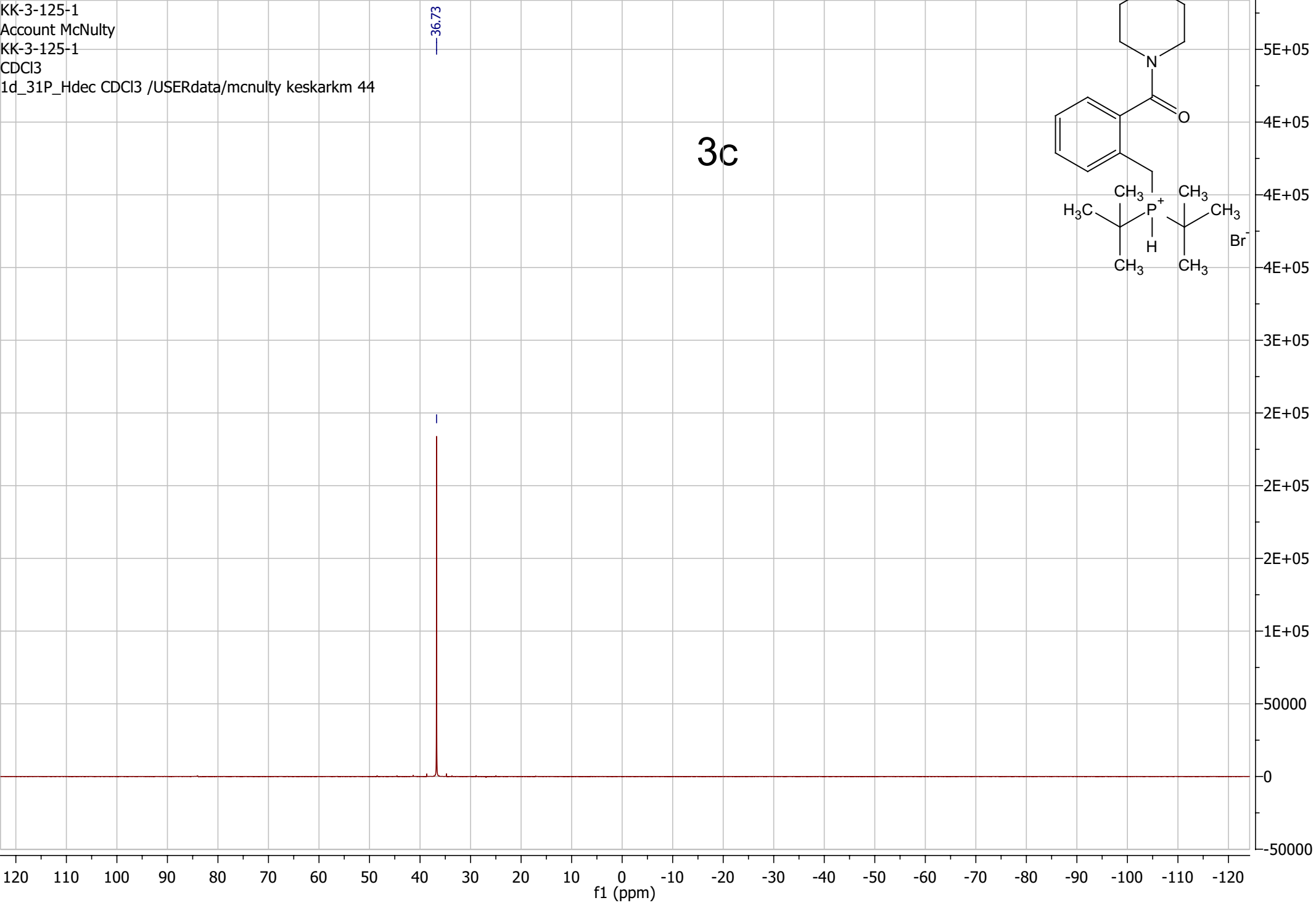
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KK-3-125-1
CDCl3
1H-NMR CDCl3 /USERdata/mcnulty keskarkm 44



KK-3-125-1
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KK-3-125-1
CDCl3
C13SN CDCl3 /USERdata/mcnulty keskarkm 44

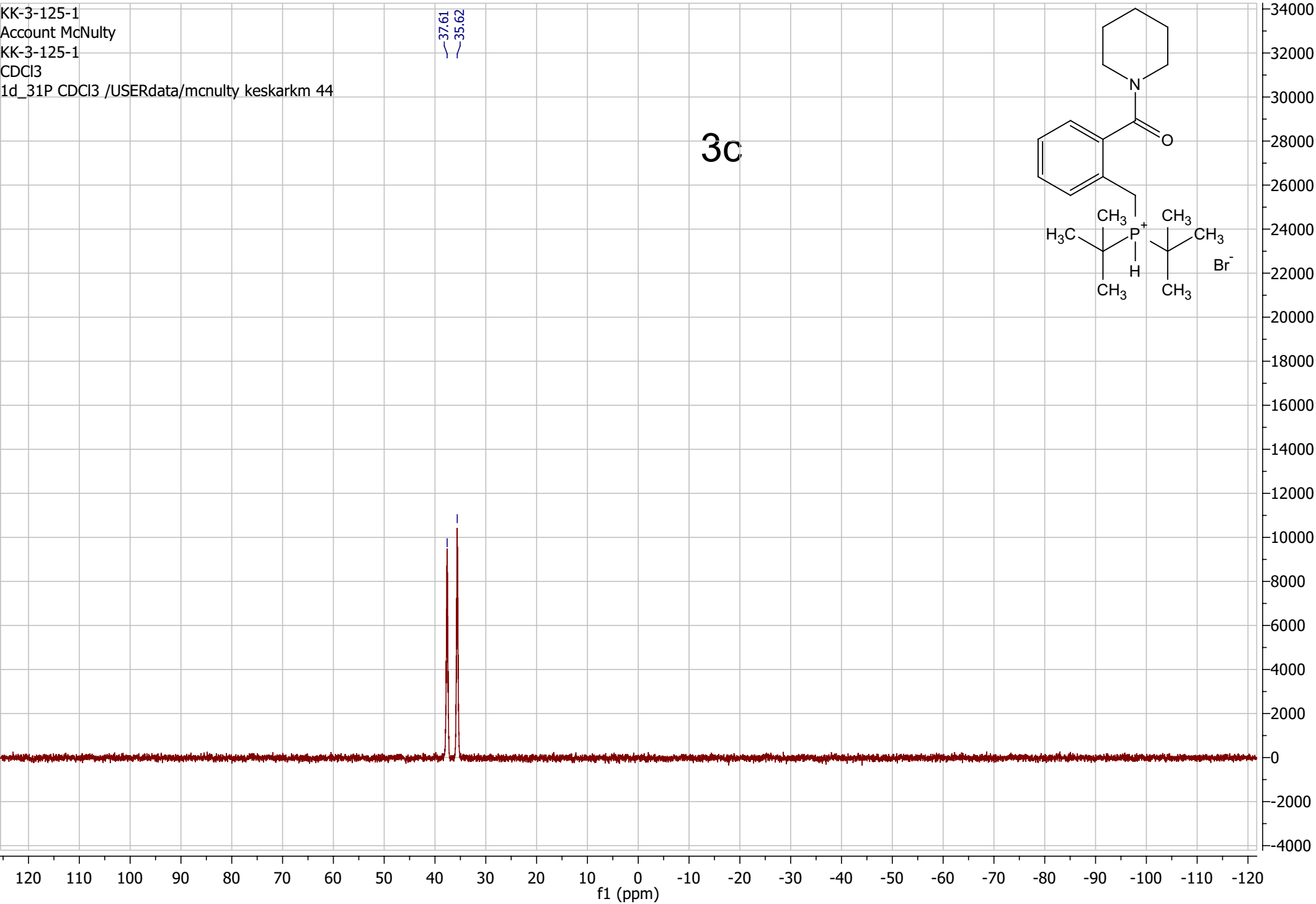
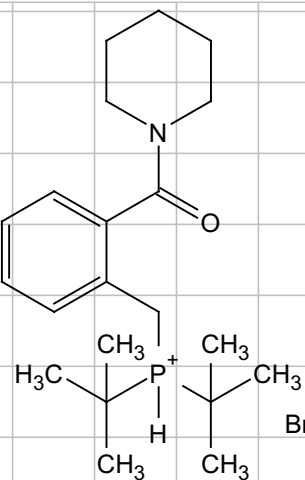


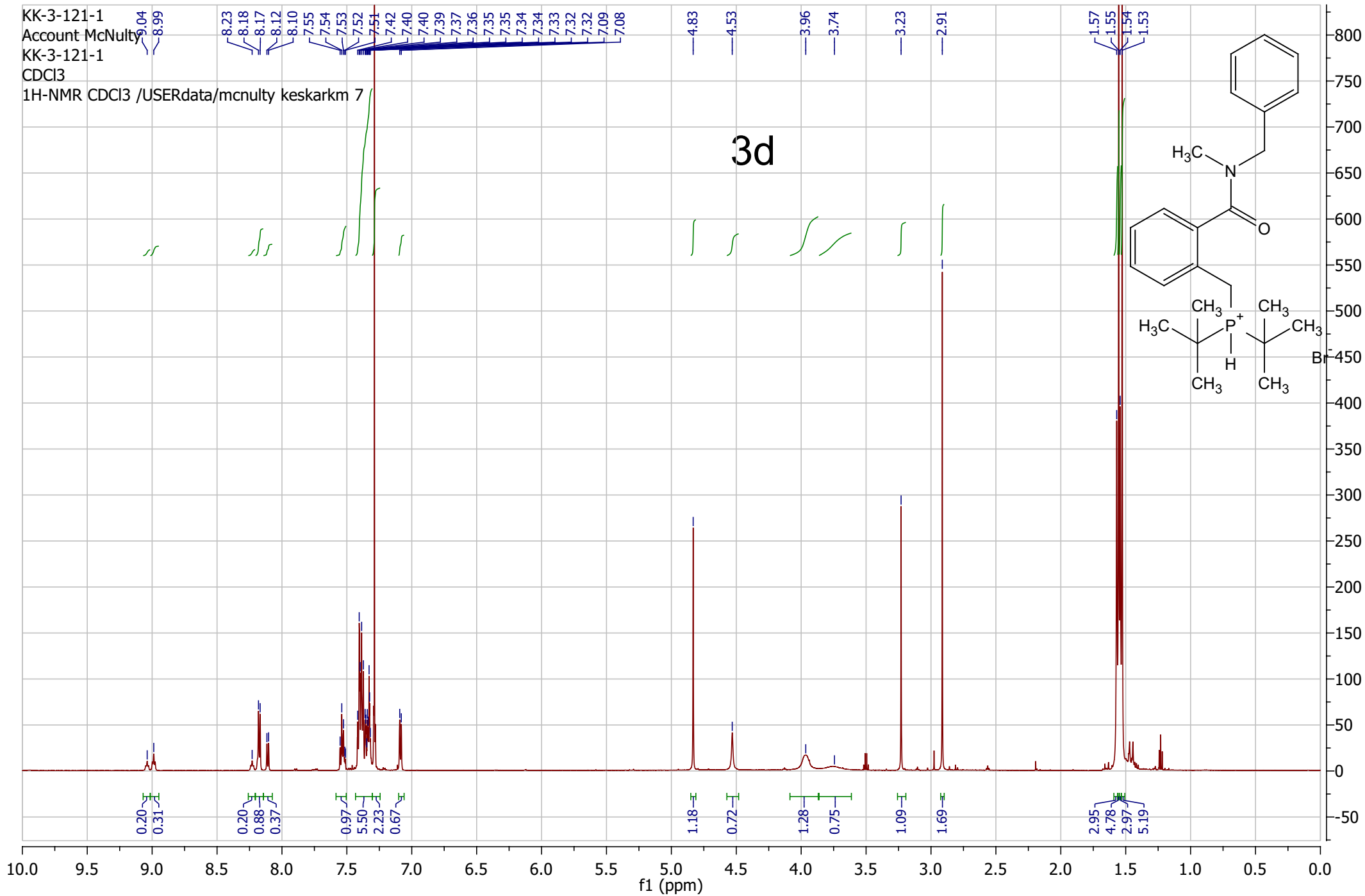
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CDCl3
1d_31P_Hdec CDCl3 /USERdata/mcnulty keskarkm 44



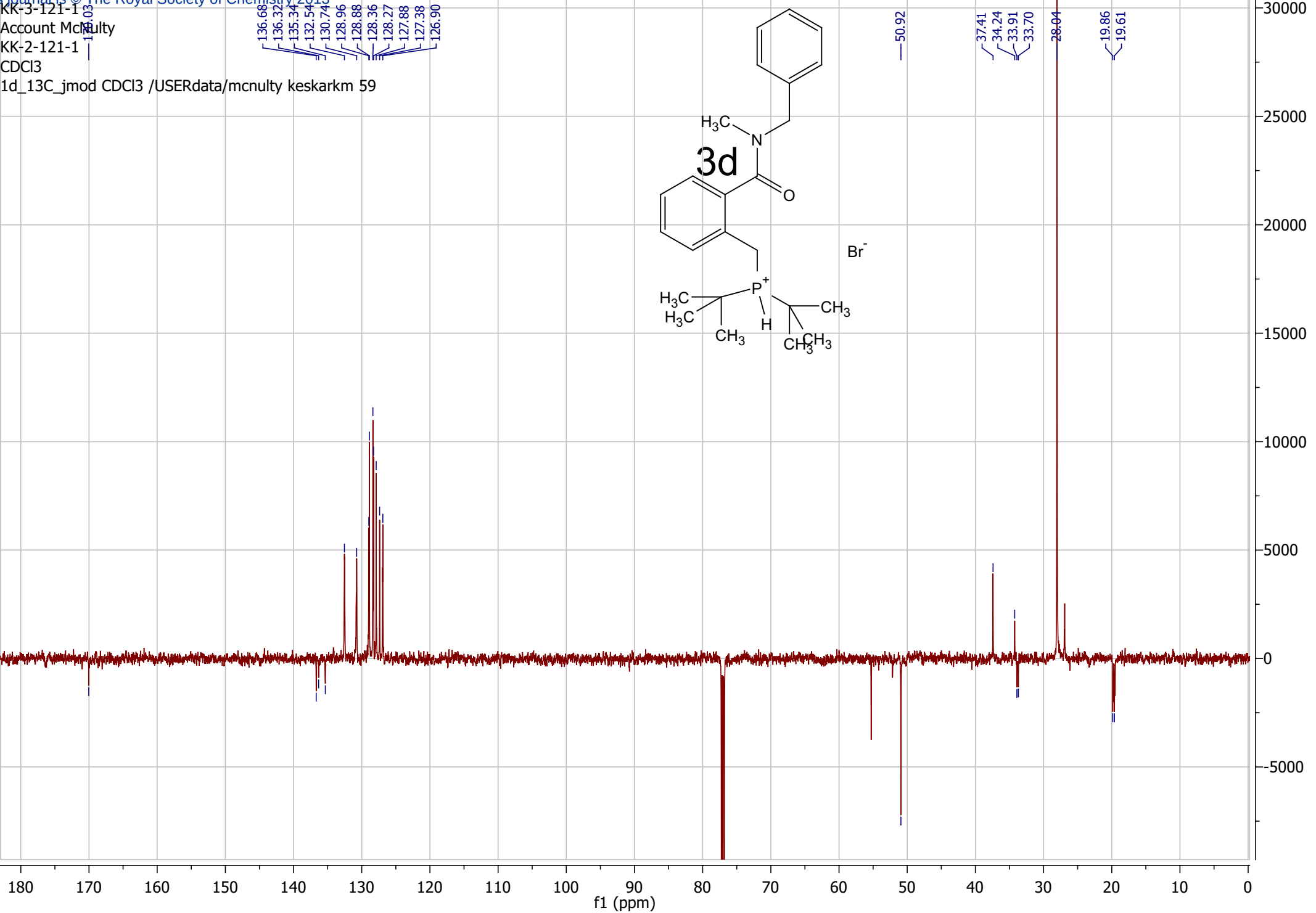
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3c

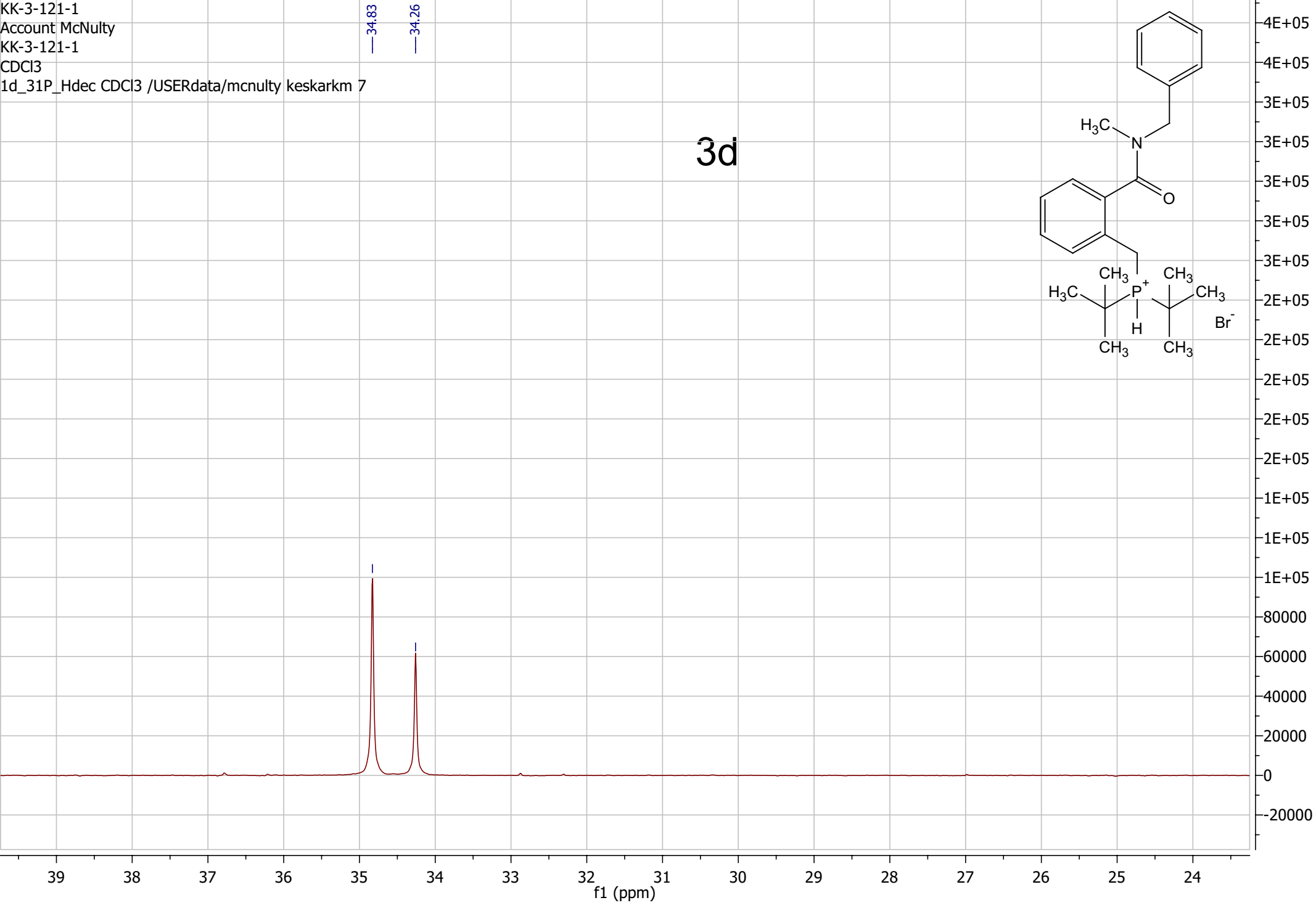




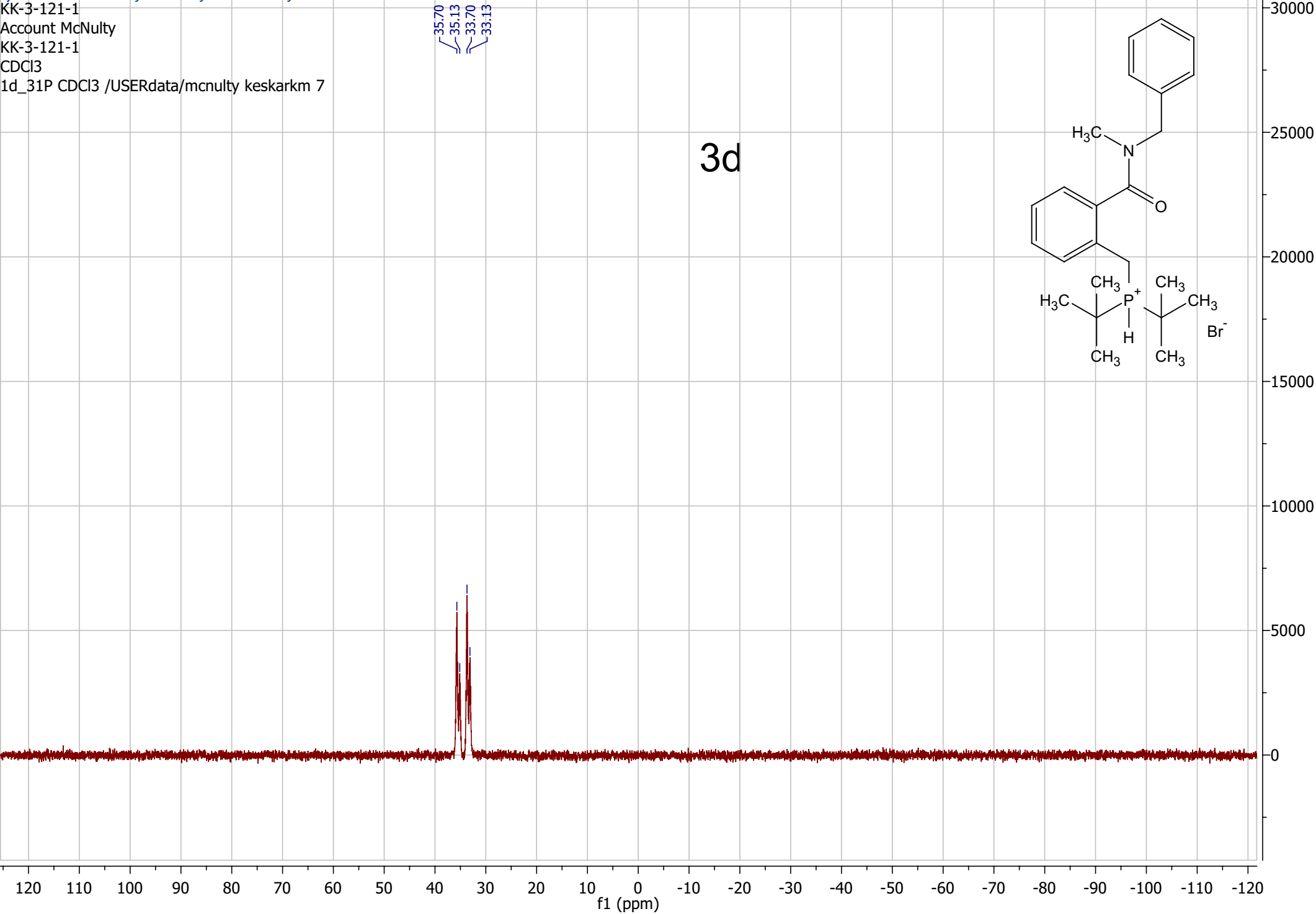
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KK-2-121-1
CDCl3
1d_13C_jmod CDCl3 /USERdata/mcnulty keskarkm 59



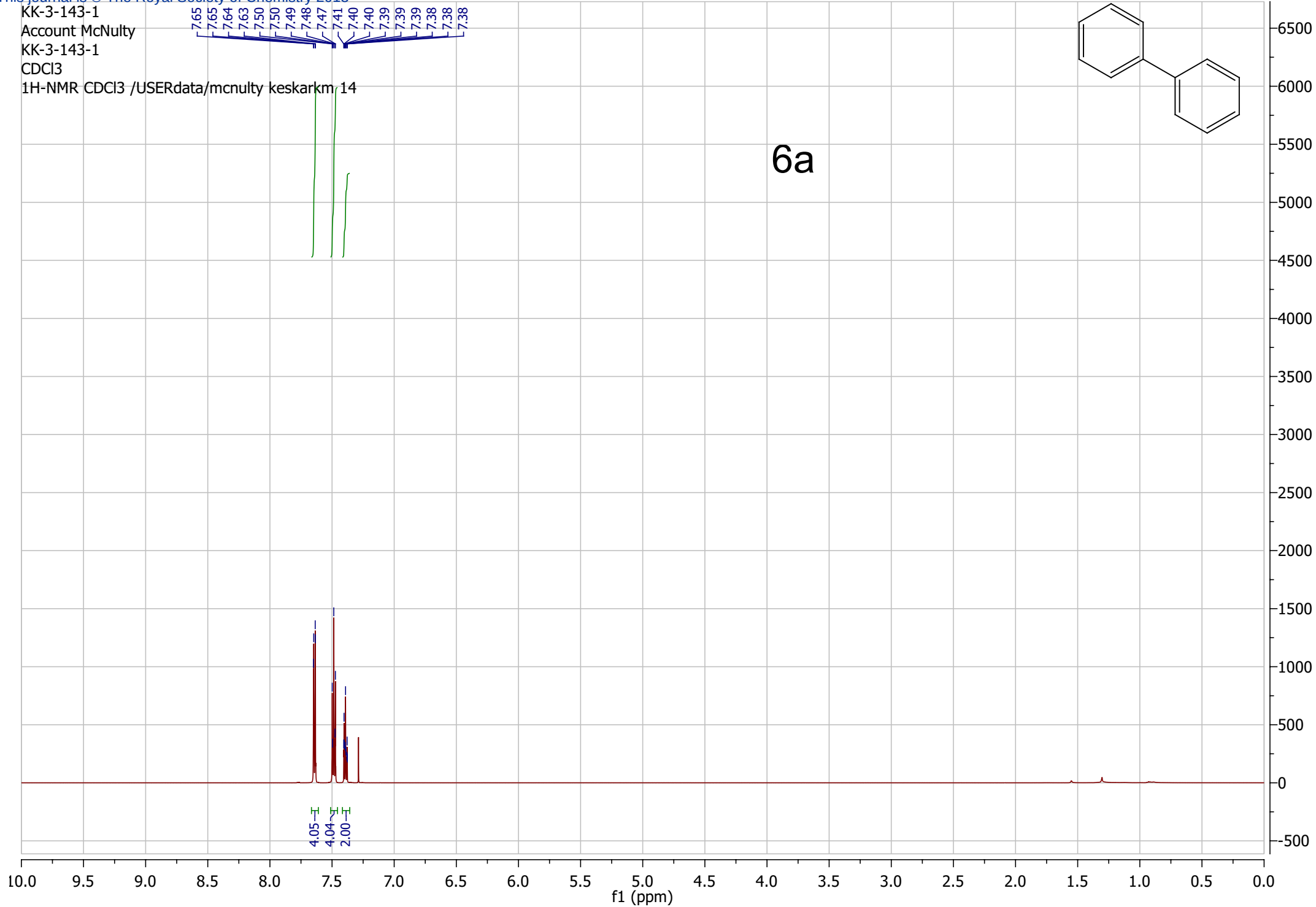
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Account McNulty
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CDCl3
1d_31P_Hdec CDCl3 /USERdata/mcnulty keskarkm 7



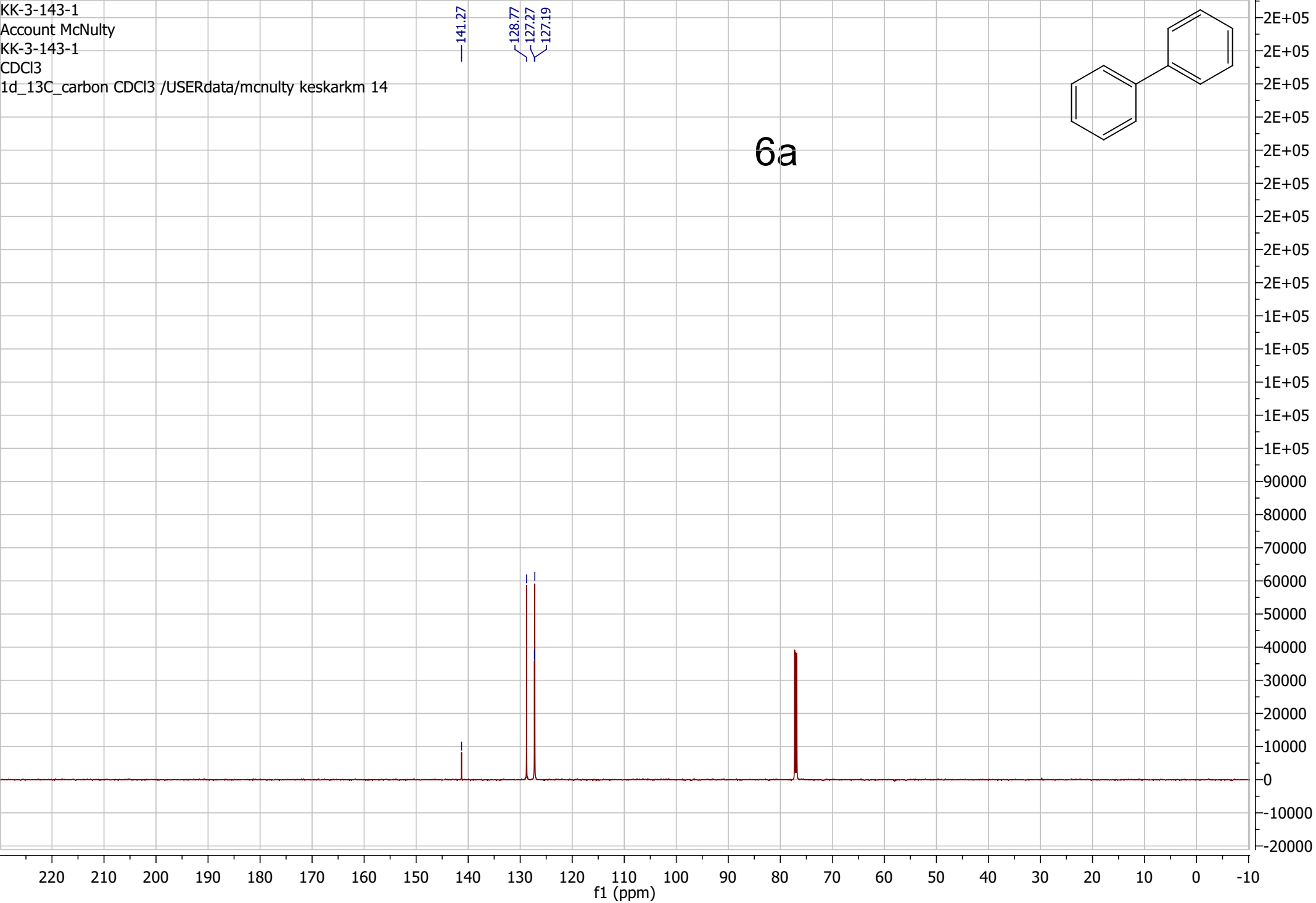
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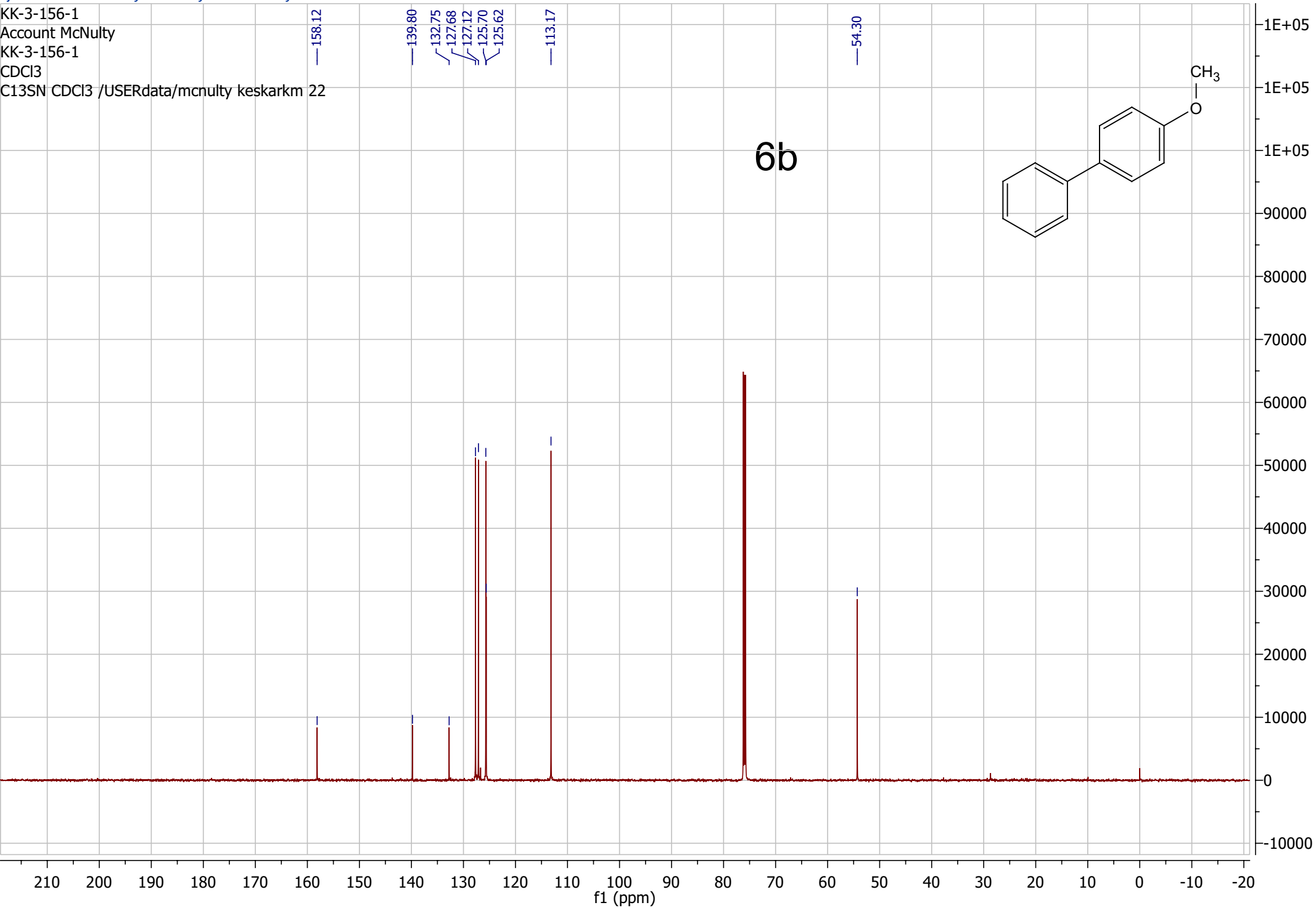
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CDCl3
1H-NMR CDCl3 /USERdata/mcnulty keskarkm 14



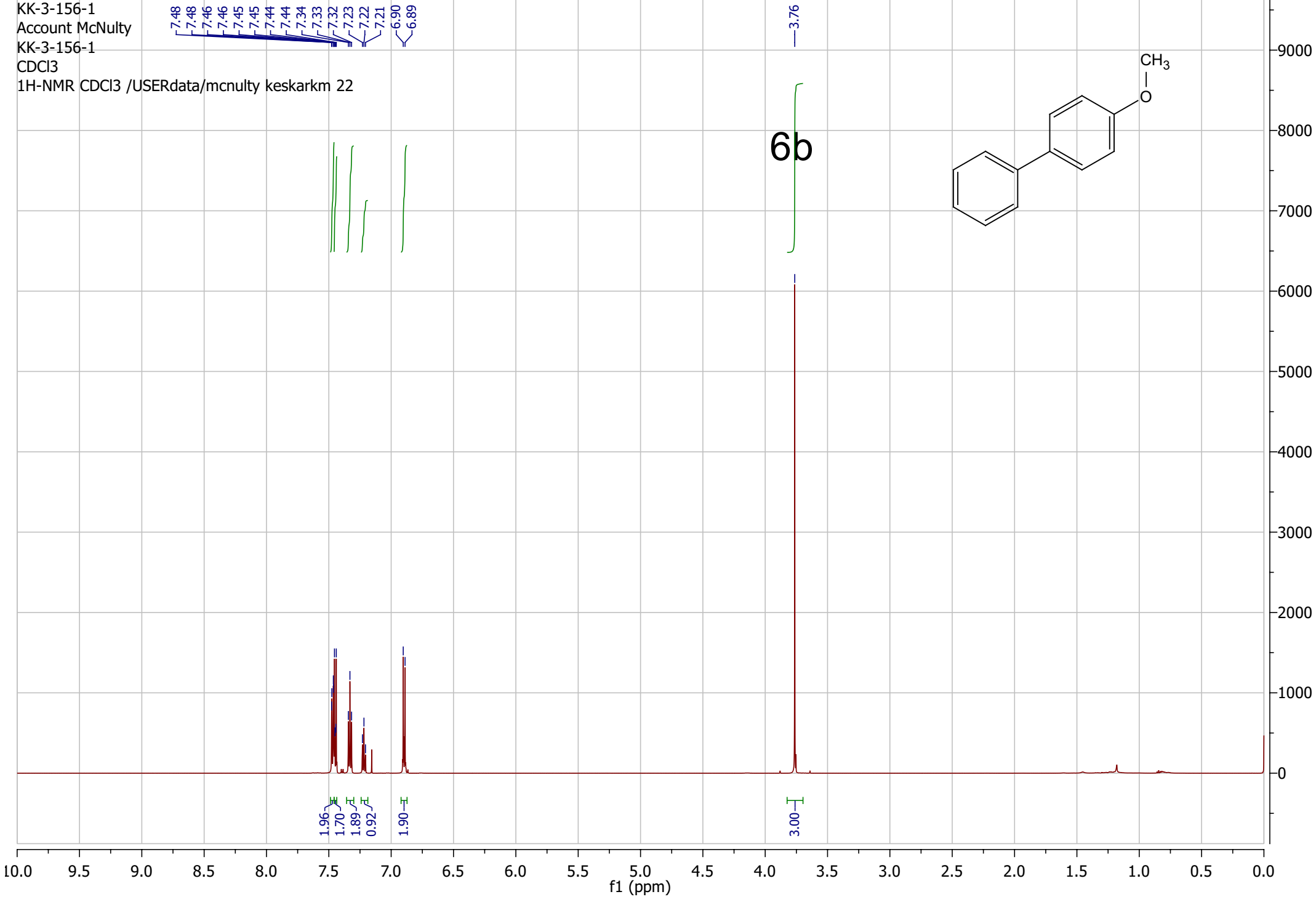
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Account McNulty
KK-3-143-1
CDCl3
1d_13C_carbon CDCl3 /USERdata/mcnulty keskarkm 14



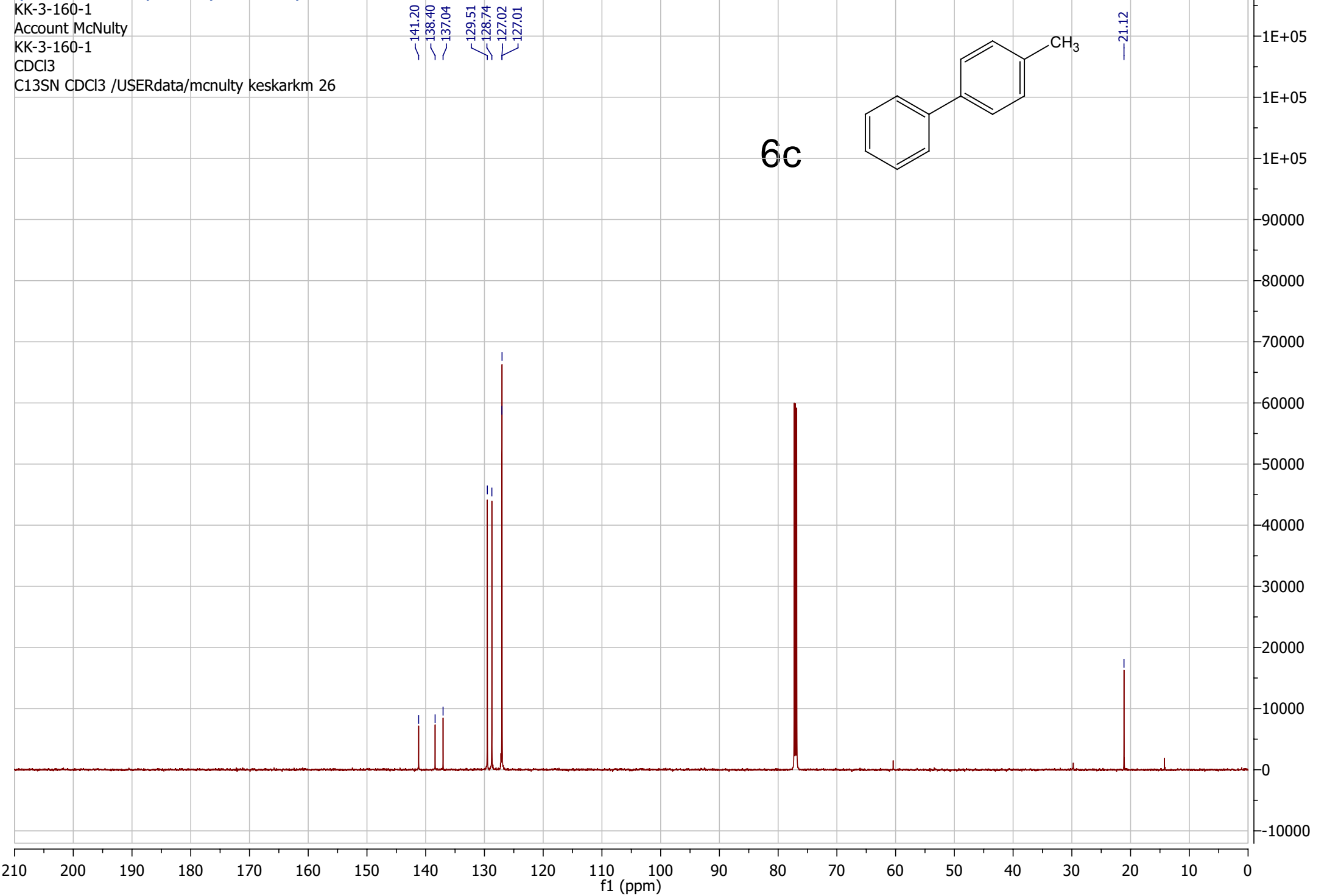
KK-3-156-1
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CDCl3
C13SN CDCl3 /USERdata/mcnulty keskarkm 22

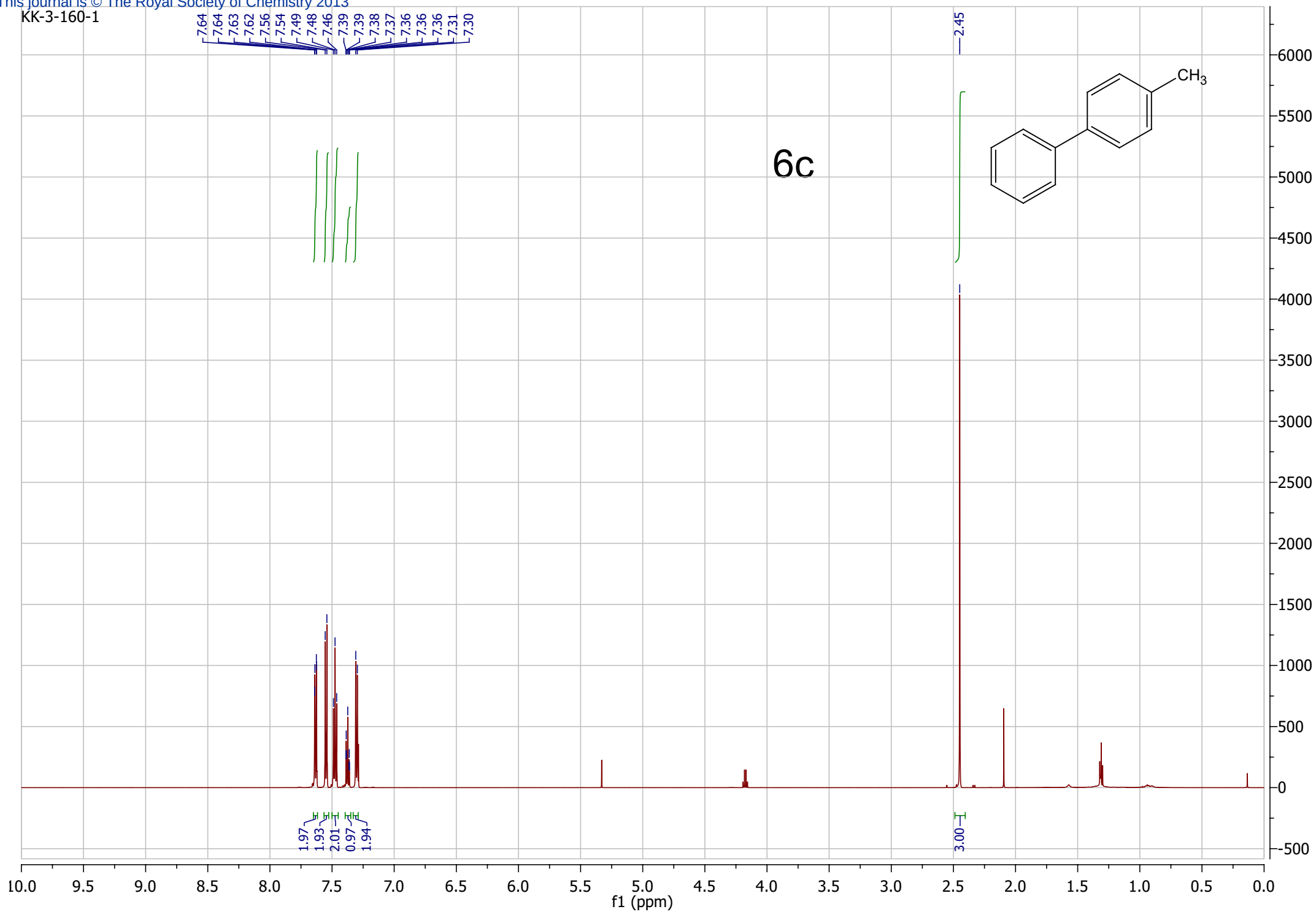


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1H-NMR CDCl3 /USERdata/mcnulty keskarkm 22

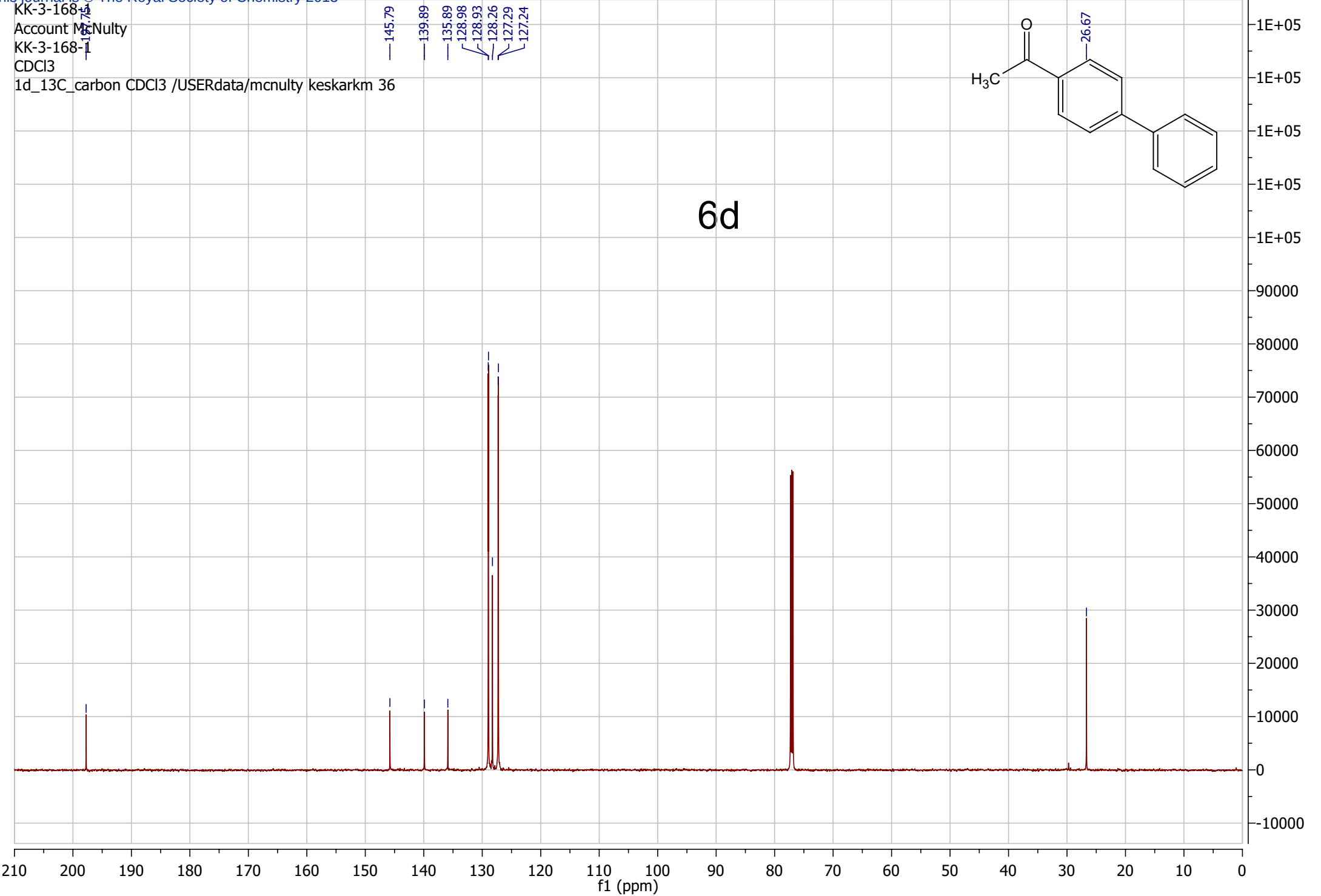


KK-3-160-1
Account McNulty
KK-3-160-1
CDCl3
C13SN CDCl3 /USERdata/mcnulty keskarkm 26

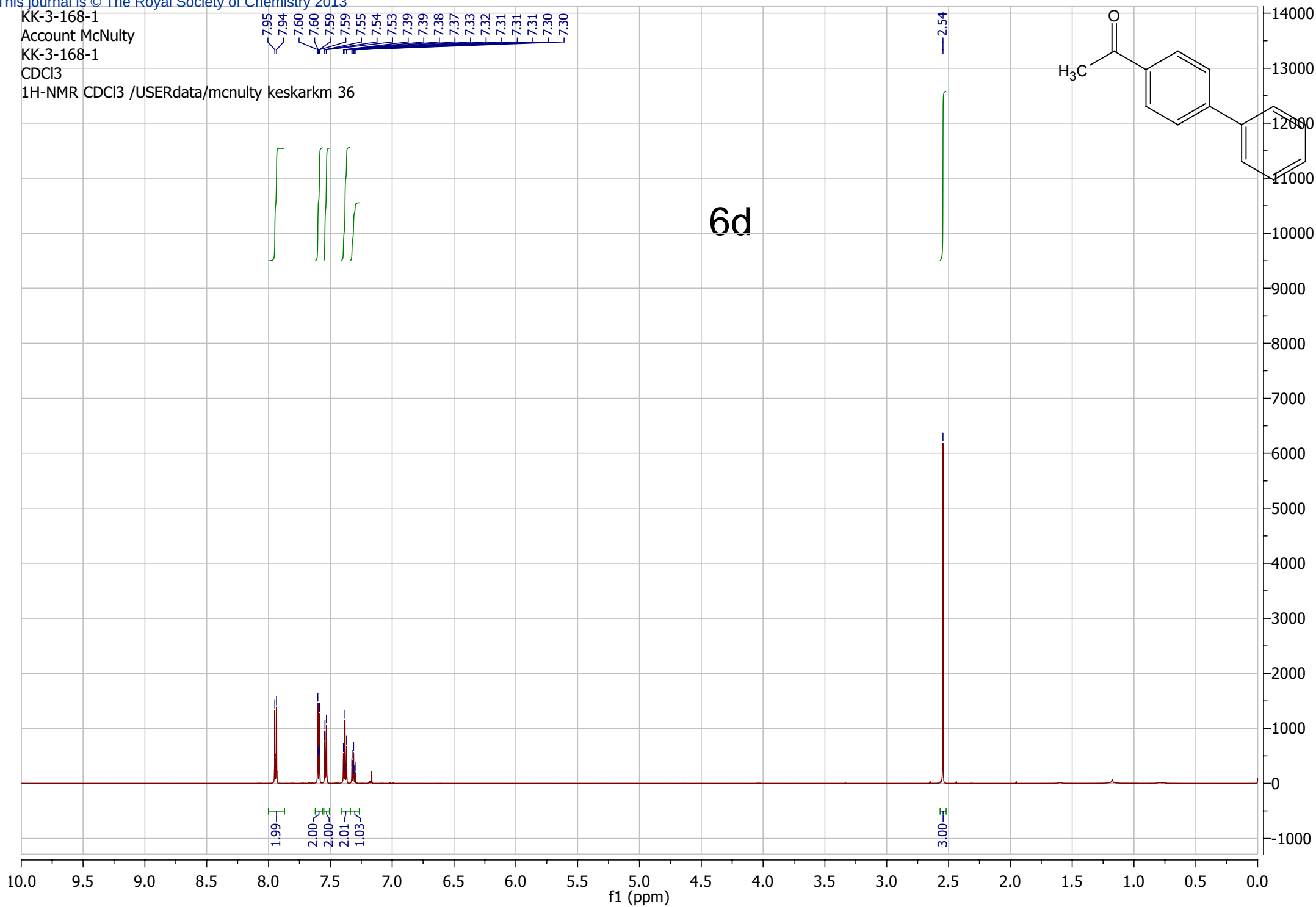




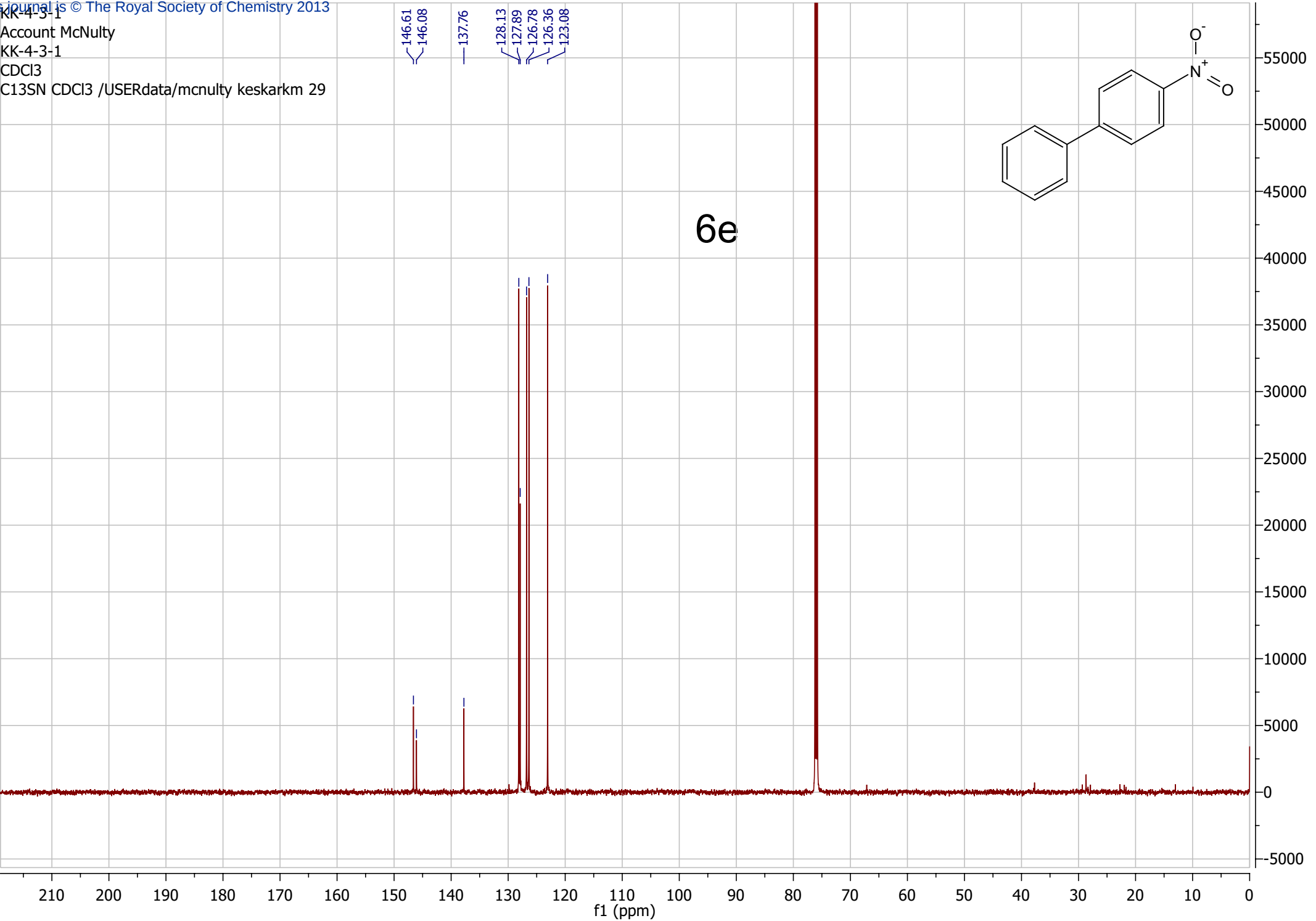
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KK-3-168-1
CDCl3
1d_13C_carbon CDCl3 /USERdata/mcnulty keskarkm 36



KK-3-168-1
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CDCl3
1H-NMR CDCl3 /USERdata/mcnulty keskarkm 36



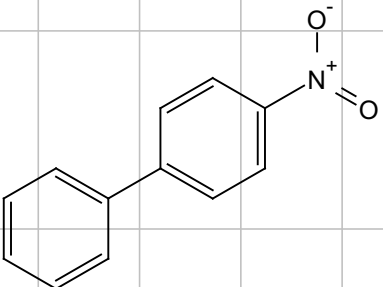
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KK-4-3-1
CDCI3
C13SN CDCI3 /USERdata/mcnulty keskarkm 29



KK-4-3-1



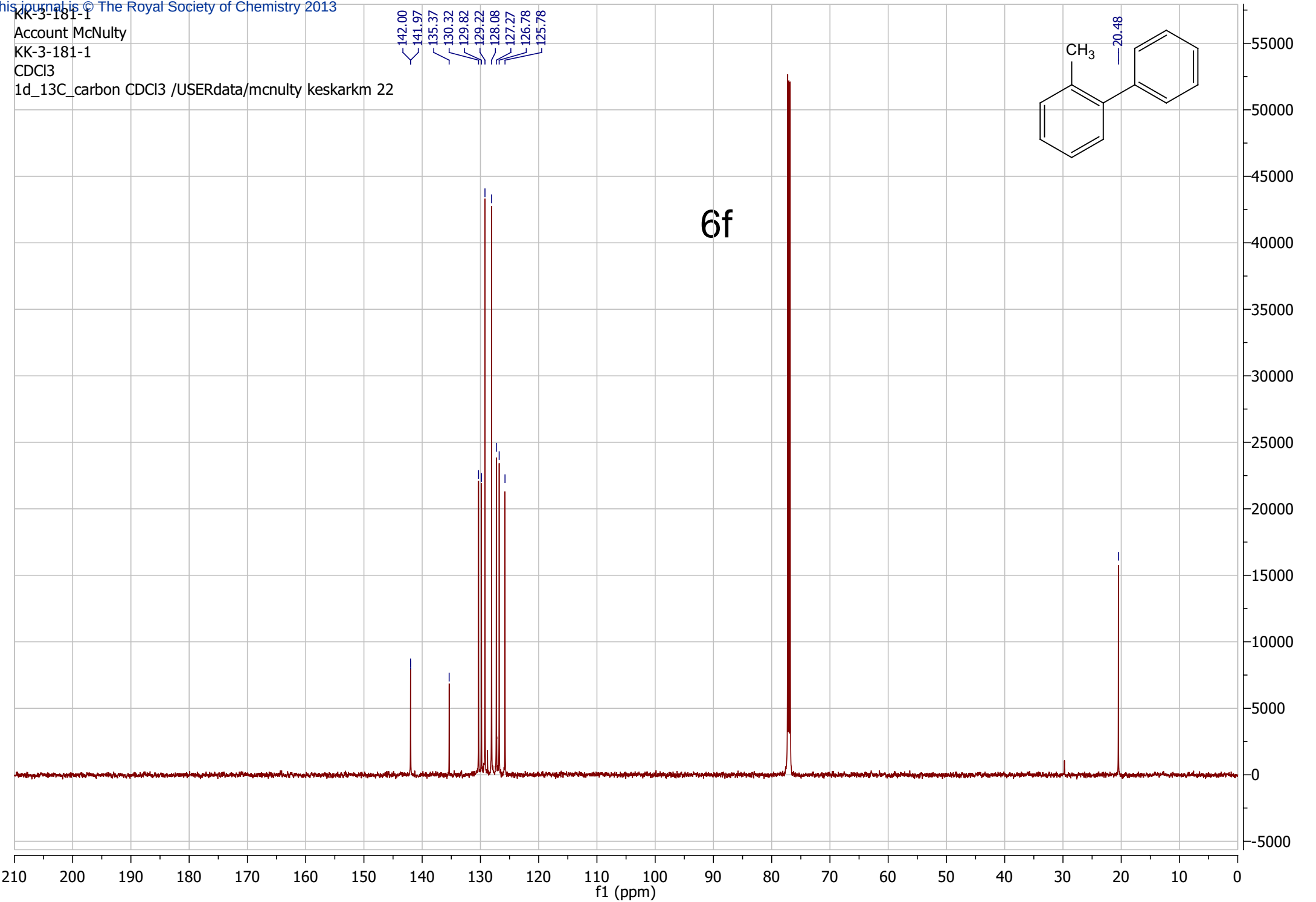
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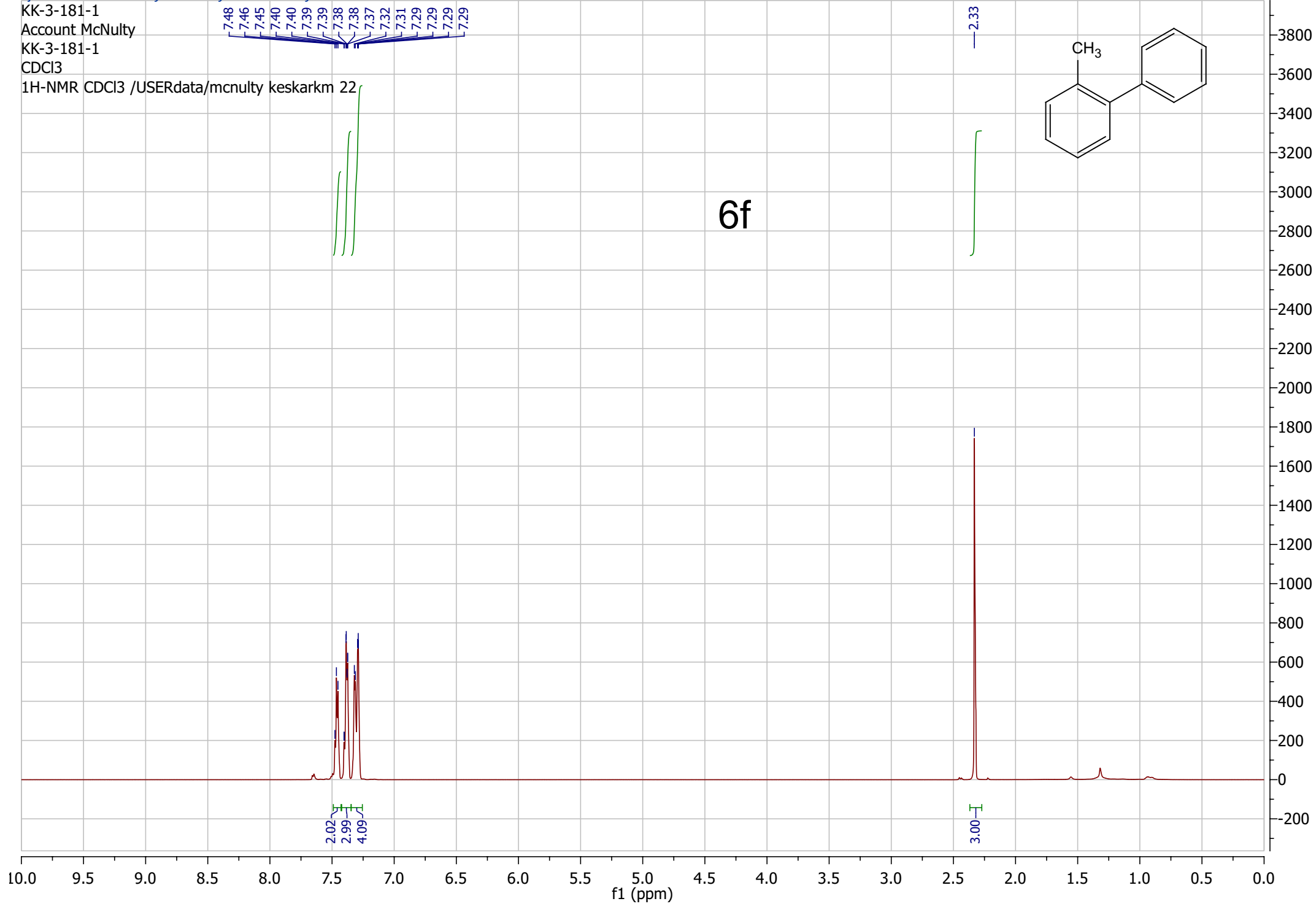
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f1 (ppm)

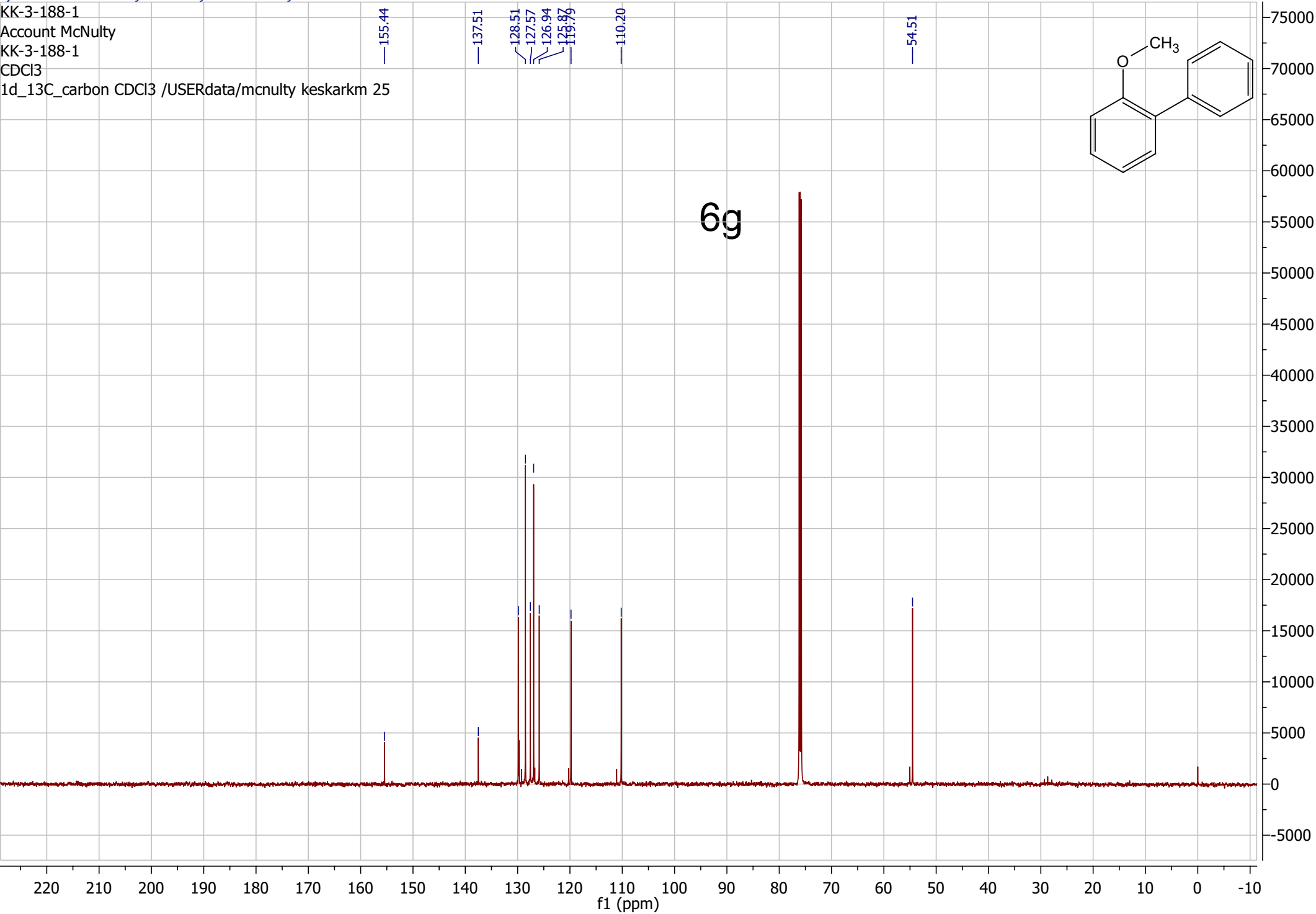
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Account McNulty
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CDCl3
1d_13C_carbon CDCl3 /USERdata/mcnulty keskarkm 22

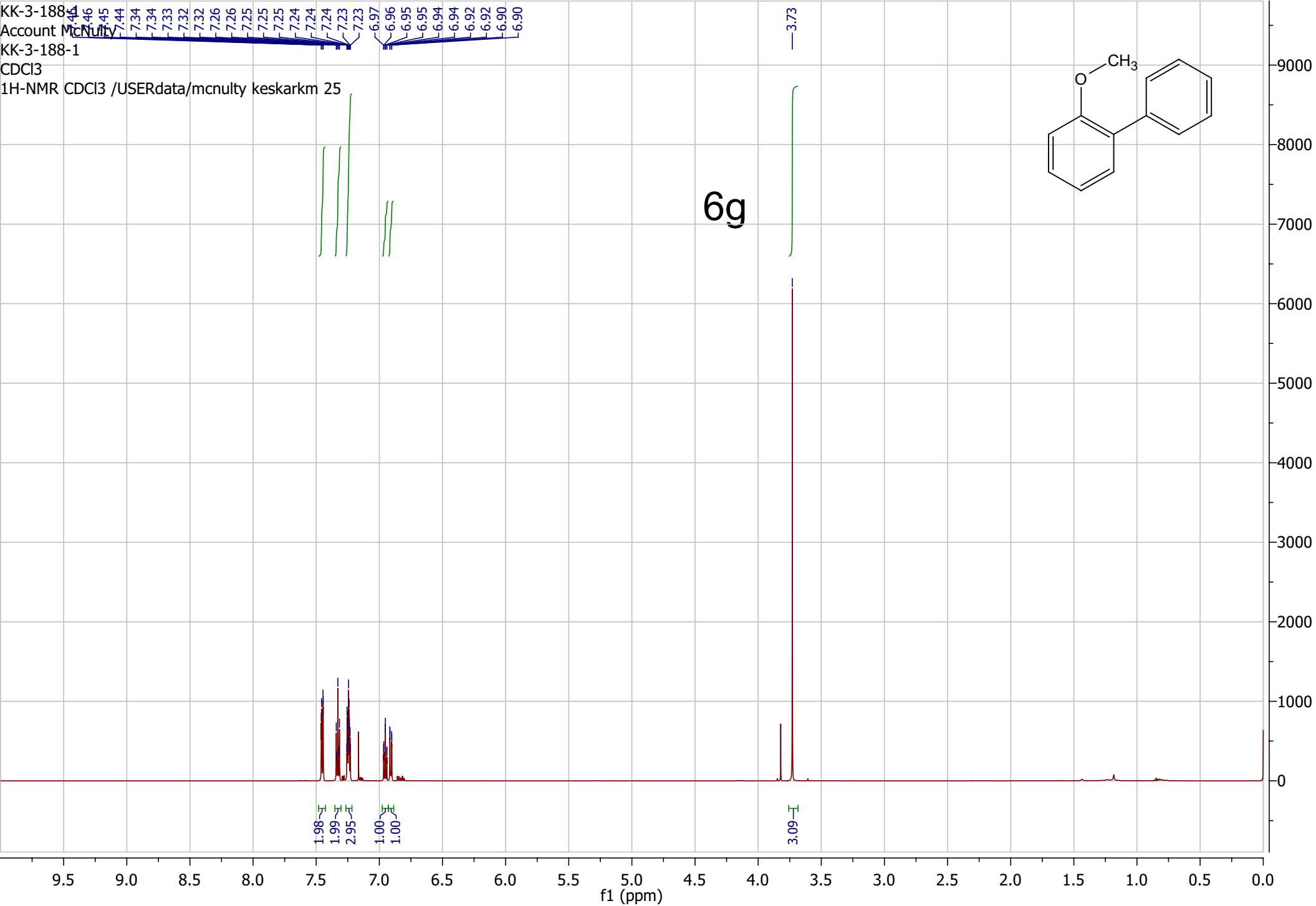


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CDCl3
1H-NMR CDCl3 /USERdata/mcnulty keskarkm 22

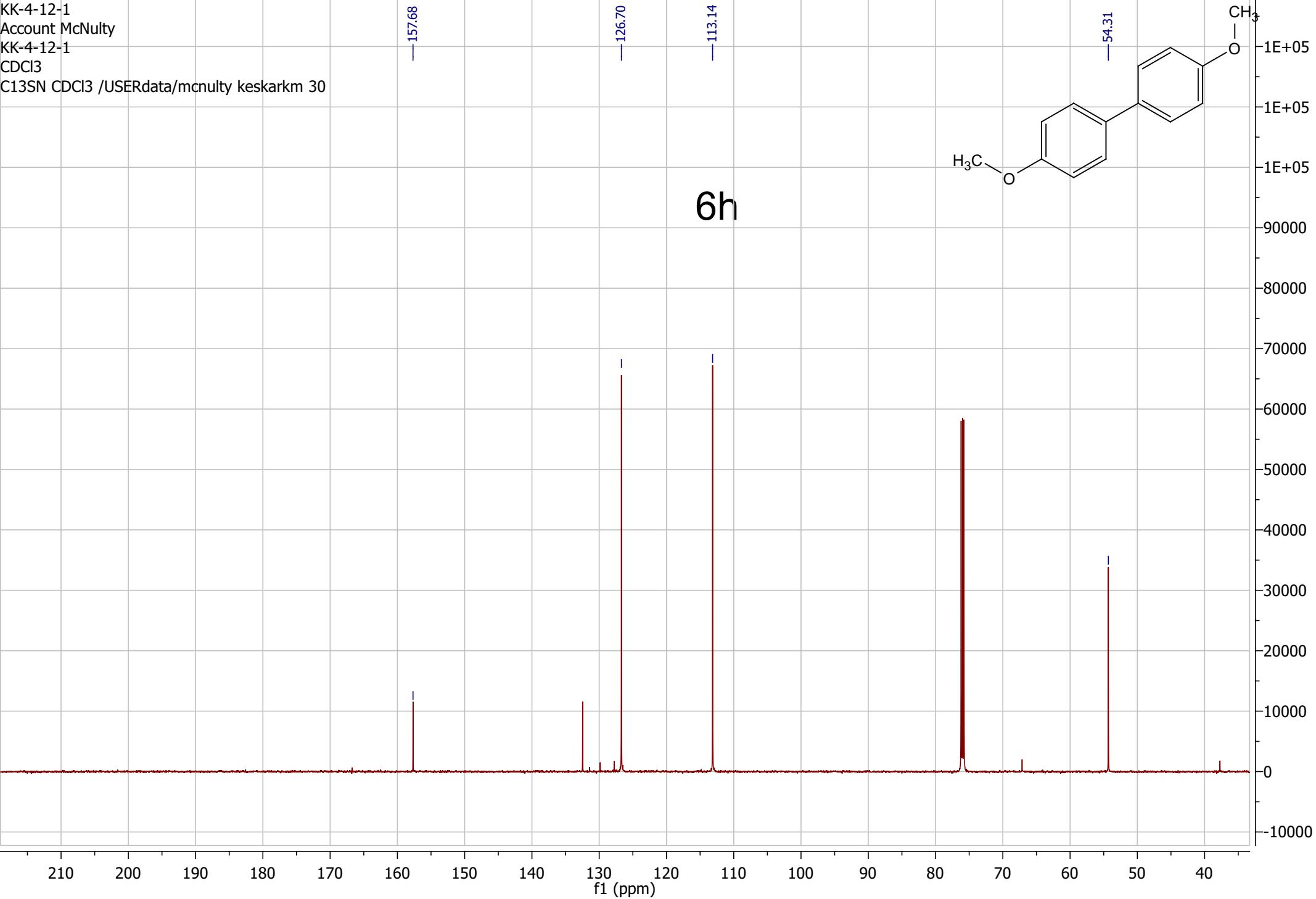


KK-3-188-1
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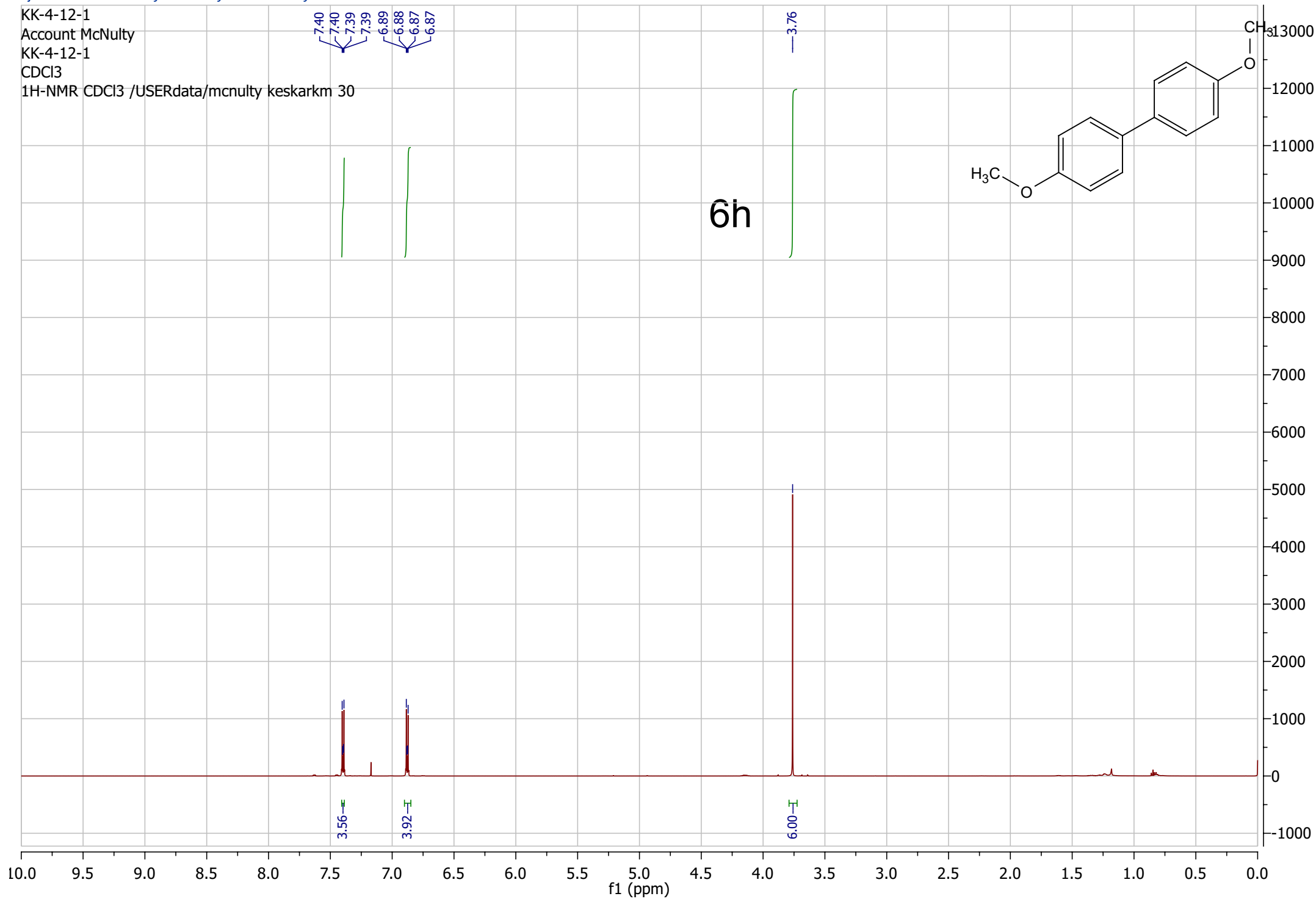




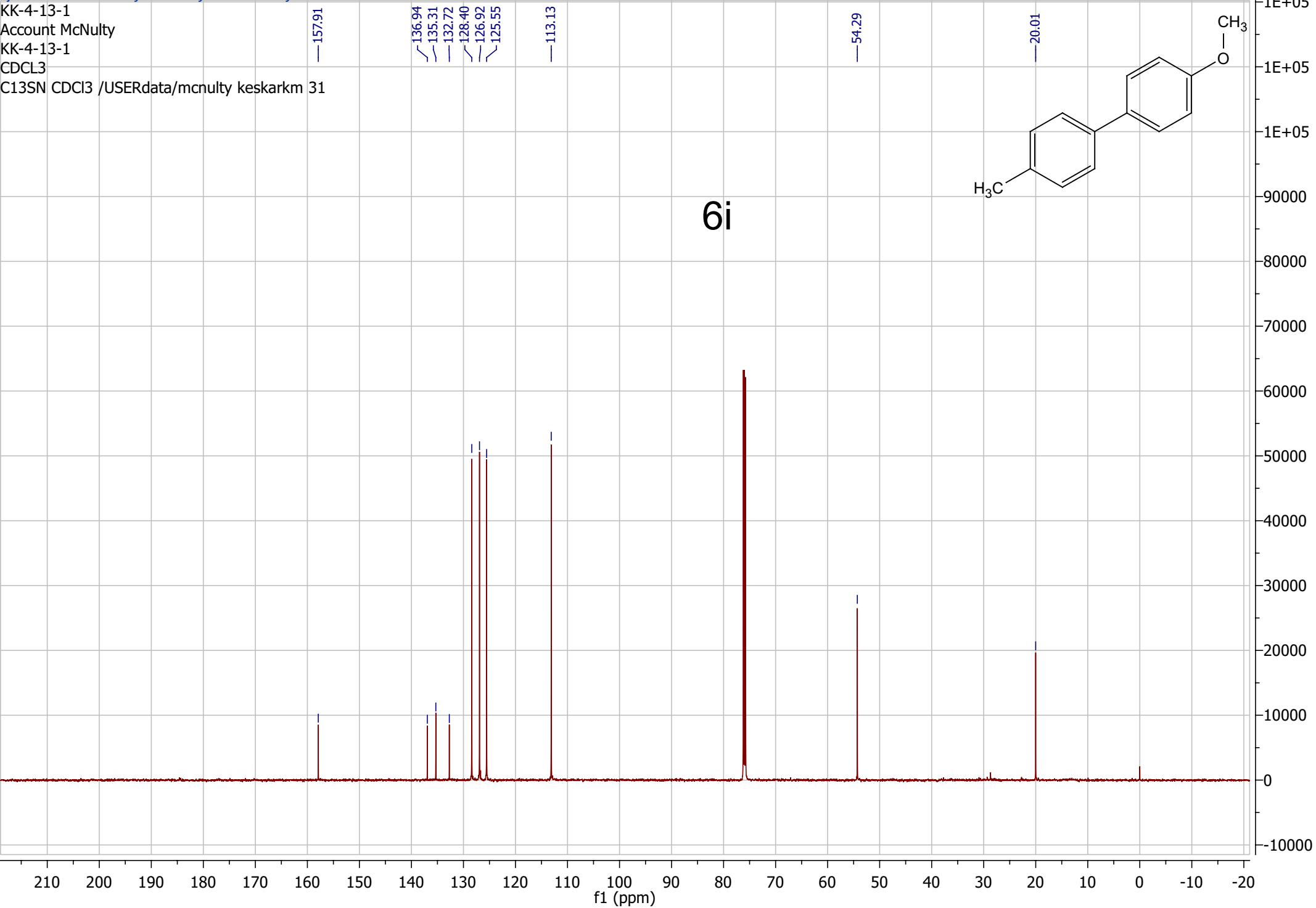
KK-4-12-1
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KK-4-12-1
CDCl3
C13SN CDCl3 /USERdata/mcnulty keskarkm 30



KK-4-12-1
Account McNulty
KK-4-12-1
CDCl3
1H-NMR CDCl3 /USERdata/mcnulty keskarkm 30



KK-4-13-1
Account McNulty
KK-4-13-1
CDCL3
C13SN CDCl3 /USERdata/mcnulty keskarkm 31



KK-4-13-1
Account McNulty
KK-4-13-1
CDCL3
1H-NMR CDCl3 /USERdata/mcnulty keskarkm 31

