

One-pot ester and thioester formation mediated by pentafluoropyridine (PFP)

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Experimental

General

All starting materials and reagents were purchased from commercial sources and used as received. MeCN was dried over 4 Å molecular sieves which had been dried under vacuum at 150 °C for 3 h. All reactions were conducted under an atmosphere of air. Column chromatography was carried out on silica purchased from Fluorochem using hexane/ethyl acetate solvent systems or using a Combiflash Nextgen 100 equipped with a 4g redisepp column using hexane/ethyl acetate solvent systems.

¹H NMR spectra were recorded at 400 or 600 MHz using Bruker Avance III or Varian VNMRS-600 spectrometers respectively. ¹³C NMR spectra were recorded at 100 or 151 MHz using a Bruker Avance III or Varian VNMRS-600 respectively. ¹⁹F NMR spectra were recorded at 376 MHz using a Bruker Avance III spectrometer. All coupling constants are reported in Hertz (Hz). In cases where it was required 2D NMR techniques were used to confirm compound identity. Chemical shifts are reported in ppm and are referenced to residual solvent peaks; CHCl₃ (¹H 7.26 ppm, ¹³C 77.0 ppm), CH₃CN (¹H 1.94 ppm, ¹³C ppm) or DMSO (¹H 2.50 ppm, ¹³C 39.5 ppm).

Mass spectra were collected either using ESI-LC or GCMS. ESI-LC in MeCN were collected using a Waters TQD mass spectrometer with a Acquity UPLC BEH C18 1.7 μm (2.1 mm x 50 mm). ESI-LC was collected using water containing formic acid (0.1% v/v) and MeCN mixture in a 95:5 to 5:95 gradient over 5 min. GCMS experiments were carried out on a Shimadzu QP2010-Ultra with a Rxi-5Sil MS (0.15 μm x 10m x 0.15 mm). Helium was employed as the carrier gas (0.41 mL/min). EI is carried at 70eV and the working mass range is 35 – 650 u for all GCMS experiments.

ASAP samples were run isothermally at 350 °C vaporising the sample to enable atmospheric pressure chemical ionisation.

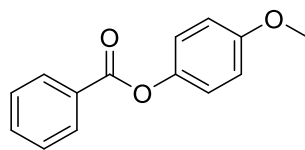
General procedure for the synthesis of esters

To an oven dried glass vial or Radley's carousel tube equipped with a stirrer bar was added carboxylic acid (1.0 equiv.), acetonitrile dried over 4 Å molecular sieves (3 mL), diisopropylethylamine (DIPEA) (2.0 equiv.) and pentafluoropyridine (PFP) (1.1 equiv.). This mixture was allowed to stir at 50 °C for 4 h, at which point the desired alcohol (1.0 equiv.) was added. The mixture was then stirred at 50 °C for 16 h. Following this time, the mixture was concentrated under reduced pressure, the resulting residue was dissolved in a minimum amount of DCM and the recovered crude material was purified directly by flash column chromatography which yielded the desired compounds.

General procedure for the synthesis of thioesters

To an oven dried glass vial or Radley's carousel tube equipped with a stirrer bar was added carboxylic acid (1.0 equiv.), acetonitrile dried over 4 Å molecular sieves (3 mL), diisopropylethylamine (DIPEA) (2.0 equiv.) and pentafluoropyridine (1.1 equiv.). This mixture was allowed to stir for a period of 4 h, after which thiol (1.0 equiv.) was added. The mixture was then allowed to stir at 50 °C for 16 h after this time, the mixture was allowed to cool, was concentrated under reduced pressure, and the resulting residue was dissolved in a minimum amount of dichloromethane (DCM) and purified directly by flash column chromatography which yielded the desired compounds.

Synthesis of 4-methoxyphenyl benzoate (5a)



Synthesised according to the general method for esterification from benzoic acid (0.102 g, 0.84 mmol) and 4-methoxyphenol (0.105 g, 0.86 mmol). The crude material was purified by flash column chromatography (100% hexane to 2.5% EtOAc 97.5% hexane) to give the desired product as a white solid in 71% yield (0.135 g).

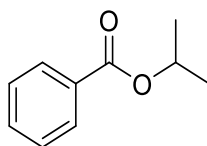
Characterisation data was consistent with previously reported literature values.¹

¹H NMR (400 MHz, CDCl₃) δ 8.25 – 8.22 (m, 2H), 7.64 (t, *J* = 7.5, 1H), 7.52 (t, *J* = 7.5, 2H), 7.17 (d, *J* = 9.1, 2H), 6.97 (d, *J* = 9.1, 2H), 3.83 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 165.5, 157.3, 144.4, 133.5, 130.1, 129.6, 128.6, 122.5, 114.5, 55.6.

LCMS (ESI⁺) *rt* = 2.7 mins, *m/z* = 229.2 [M+H]⁺

Synthesis of isopropyl benzoate (5b)



Synthesised according to the general method for esterification from benzoic acid (0.105 g, 0.86 mmol) and isopropanol (0.057 g, 0.095 mmol). The crude material was purified by automated flash column chromatography combiflash (eluted in 100% hexanes) to give the desired product as a colourless oil in 41% yield (0.055g).

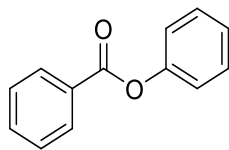
Characterisation data was consistent with previously reported literature values.²

¹H NMR (400 MHz, CDCl₃) δ 8.06 – 8.03 (m, 2H), 7.56 – 7.52 (m, 1H), 7.43 (ddt, *J* = 8.1, 6.8, 1.0, 2H), 5.26 (hept, *J* = 6.3, 1H), 1.37 (d, *J* = 6.3, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 166.2, 132.8, 131.0, 129.6, 128.4, 68.4, 22.0.

GCMS (EI⁺) *r.t* = 3.3 mins, *m/z* = 164.1 [M]⁺

Synthesis of phenyl benzoate (5d)



Synthesised according to the general method for esterification from benzoic acid (0.100 g, 0.82 mmol) and phenol (0.082 g, 0.87 mmol). The crude material was purified by flash column chromatography (100% hexane to 5% EtOAc 95% hexane) to give the desired product as a white solid 80% yield (0.130 g)

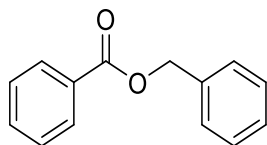
Characterisation data were consistent with previously reported literature values.³

¹H NMR (400 MHz, CDCl₃) δ 8.30 – 8.26 (m, 2H), 7.71 – 7.66 (m, 1H), 7.59-7.54 (m, 2H), 7.51 – 7.46 (m, 2H), 7.35 – 7.26 (m, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 165.3, 151.1, 133.7, 130.3, 130.2, 130.1, 129.7, 129.6, 128.7, 126.0, 121.82.

LCMS (ESI⁺) r.t. = 2.8 mins, *m/z* = 199.2 [M+H]⁺

Synthesis of benzyl benzoate (5e)



Synthesised according to the general method for esterification from benzoic acid (0.101 g, 0.83 mmol) and benzyl alcohol (0.090 g, 0.83 mmol). The crude material was purified by flash column chromatography (100% hexane to 2.5% EtOAc 97.5% hexane) to give the desired product as a colourless oil in 72% yield (0.122 g)

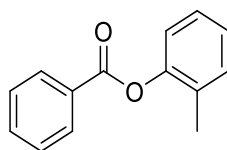
Characterisation data were consistent with previously reported literature values.³

¹H NMR (599 MHz, CDCl₃) δ 8.15-8.12z (m, 2H), 7.60-7.56 (m, 1H), 7.51 – 7.38 (m, 7H), 5.41 (s, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 166.7, 136.4, 133.3, 130.5, 130.0, 128.9, 128.7, 128.5, 128.5, 67.0.

GCMS (EI⁺) – r.t. = 4.8 mins, m/z = 212.1 [M]⁺

Synthesis of *o*-tolyl benzoate (5f)



Synthesised according to the general method for esterification from benzoic acid (0.101, 0.83 mmol) and *o*-cresol (0.092 g, 0.85 mmol). The crude material was purified using automated flash column chromatography combiflash (100% Hexane for 4 mins to 40% EtOAc 60% Hexane for 10 mins) to give the desired product as a clear oil in 67% yield (0.116 g).

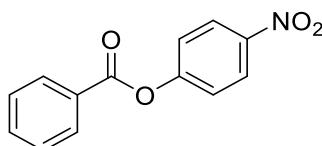
Characterisation data were consistent with previously reported literature values.⁴

¹H NMR (400 MHz, CDCl₃) δ 8.29 – 8.27 (m, 2H), 7.68 – 7.66 (m, 1H), 7.58 – 7.54 (m, 2H), 7.33 – 7.18 (m, 4H), 2.29 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 164.9, 149.6, 133.7, 131.3, 130.4, 130.2, 129.6, 128.7, 127.1, 126.2, 122.1, 16.3.

LCMS (ESI⁺) r.t. = 2.9 mins, m/z = 213.3 [M+H]⁺

Synthesis of 4-nitrophenyl benzoate (5g)



Synthesised according to the general method for esterification from benzoic acid (0.101 g, 0.83 mmol) and 4-nitrophenol (0.112 g, 0.81 mmol). The crude material was purified by flash column chromatography (100% hexane to 2.5% EtOAc 97.5% hexane) to give the desired product as a white solid in 58% yield (0.115 g)

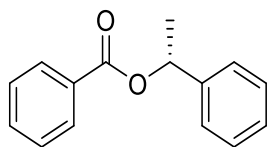
Characterisation data were consistent with previously reported literature values.⁵

¹H NMR (400 MHz, CDCl₃) δ 8.34 – 8.28 (m, 2H), 8.24 – 8.18 (m, 2H), 7.71-7.64 (m, 1H), 7.58 – 7.51 (m, 2H), 7.46 – 7.39 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 164.3, 155.8, 145.5, 134.4, 130.4, 128.9, 128.6, 125.4, 122.8.

LCMS (ESI⁻) - r.t. = 3.8 mins, m/z = 242.1 (M)⁻

Synthesis of (5h)



Synthesised according to the general method for esterification from benzoic acid (0.103 g, 0.84 mmol) and (*S*)-(-)-1-phenylethanol (0.102 g, 0.83 mmol). Purified using automated column chromatography combiflash (eluted in 100% hexanes) to give the desired product as a clear oil in 48% yield (0.091 g).

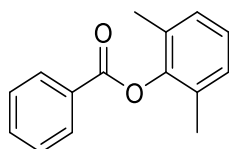
Characterisation data were consistent with previously reported literature values.⁶

¹H NMR (400 MHz, CDCl₃) δ 8.13 – 8.10 (m, 2H), 7.69 – 7.55 (m, 1H), 7.49 – 7.43 (m, 4H), 7.42 – 7.37 (m, 2H), 7.35 – 7.30 (m, 1H), 6.16 (q, *J* = 6.6, 1H), 1.70 (d, *J* = 6.6, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 165.8, 141.8, 133.0, 130.6, 129.7, 128.6, 128.4, 127.9, 126.1, 73.0, 22.5.

GCMS (EI⁺) r.t. = 4.8 mins, m/z = 226.2 [M]⁺

Synthesis of 2,6-dimethylphenyl benzoate (5i)



Synthesised according to the general method for esterification from benzoic acid (0.099 g, 0.81 mmol) and 2,6-dimethylphenol (0.103 g, 0.84 mmol). The crude material was purified using automated flash column chromatography combiflash (eluted in 100% hexanes) to give the desired product as a clear oil in 92% yield (0.172 g).

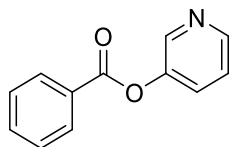
Characterisation data was consistent with previously reported literature values.⁵

¹H NMR (400 MHz, CDCl₃) δ 8.30 – 8.28 (m, 2H), 7.70 – 7.66 (m, 1H), 7.58 – 7.54 (m, 2H), 7.16 – 7.11 (m, 3H), 2.24 (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 164.5, 148.4, 133.7, 130.5, 130.3, 129.4, 128.8, 128.7, 126.0, 16.5.

LCMS (ESI⁺) r.t. = 3.1 mins, $m/z = 227.2$ [M+H]⁺

Synthesis of pyridine-3-yl-benzoate (5j)



Synthesised according to the general method for esterification from benzoic acid (0.100 g, 0.82 mmol) and 3-hydroxypyridine (0.081 g, 0.85 mmol). The crude material was purified by flash column chromatography (100% hexane to 5% EtOAc 95% hexane) to give the desired product as an orange solid in 80% yield (0.130 g).

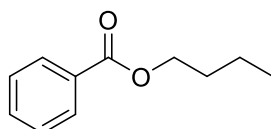
Characterisation data was consistent with previously reported literature.⁷

¹H NMR (400 MHz, CDCl₃) δ 8.56-8.55 (m, 1H), 8.54 (dd, $J = 4.8, 1.4$, 1H), 8.24 – 8.18 (m, 2H), 7.69 – 7.64 (m, 1H), 7.62 (ddd, $J = 8.3, 2.7, 1.4$, 1H), 7.53 – 7.48 (m, 2H), 7.36 (ddd, $J = 8.3, 4.8, 0.7$, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 164.7, 147.6, 147.0, 143.6, 134.0, 130.3, 129.4, 128.8, 128.7, 124.0

LCMS (ESI⁺) r.t. = 2.5 mins, $m/z = 200.1$ [M+H]⁺

Synthesis of butyl benzoate (5k)



Synthesised according to the general method for esterification from benzoic acid (0.100 g, 0.82 mmol) and butan-1-ol (0.067 g, 0.90 mmol). The crude material was purified by flash column chromatography (100% hexane to 5% EtOAc 95% hexane) to give the desired product as a clear oil in 64% yield (0.093 g).

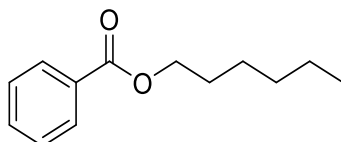
Characterisation data was consistent with previously reported literature values.⁸

¹H NMR (400 MHz, CDCl₃) δ 8.07 – 8.03 (m, 2H), 7.56 – 7.52 (m, 1H), 7.45 – 7.40 (m, 2H), 4.32 (t, $J = 6.6$, 2H), 1.79 – 1.71 (m, 2H), 1.53 – 1.43 (m, 2H), 0.98 (t, $J = 7.4$, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 166.68, 132.80, 130.54, 129.54, 128.32, 64.83, 30.80, 19.30, 13.79.

LCMS (ESI^+) r.t. = 2.9 mins, m/z = 179.2 $[\text{M}+\text{H}]^+$

Synthesis of hexyl benzoate (5l)



Synthesised according to the general method for esterification from benzoic acid (0.100g, 0.82 mmol) and hexan-1-ol (0.089 g, 0.87 mmol). The crude material was purified by automated flash column chromatography (100% hexane) to give the desired product as a clear oil in 78% yield (0.131 g).

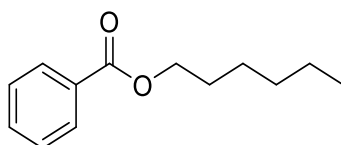
Characterisation data was consistent with reported literature.⁸

^1H NMR (400 MHz, CDCl_3) δ 8.11 – 8.01 (m, 2H), 7.58 – 7.49 (m, 1H), 7.48 – 7.36 (m, 2H), 4.31 (t, J = 6.7, 2H), 1.76 (dq, J = 8.0, 6.7, 2H), 1.53 – 1.37 (m, 2H), 1.40 – 1.29 (m, 4H), 0.97 – 0.85 (m, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 166.7, 132.8, 130.6, 129.6, 128.4, 77.5, 77.1, 76.8, 65.2, 31.5, 28.8, 25.8, 22.6, 14.1.

LCMS (ESI^+) r.t. = 3.4 mins, m/z = 207.3 $[\text{M}+\text{H}]^+$

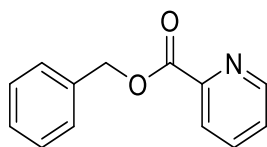
Gram Synthesis of hexyl benzoate (5l)



Following the general method but in a round bottomed flask, using 10 mL of MeCN. Synthesised from benzoic acid (1.001 g, 8.2 mmol) and hexan-1-ol (0.871 g, 8.5 mmol). Purified by automated column chromatography (100% hexane) to yield the desired product as a clear oil in 61% yield (1.030 g)

Characterisation data were consistent with those previously recorded for (5k)

Synthesis of Benzyl picolinate (5m)



Synthesised according to the general method for esterification from picolinic acid (0.105 g, 0.85 mmol) and benzyl alcohol (0.88 g, 0.81 mmol). Purified using automated column chromatography combiflash (100% Hexane to 60% Hexane 40% EtOAc gradient) to give the desired product as a red coloured oil in 68% yield (0.118 g)

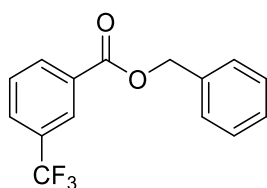
Characterisation data were consistent with previously reported literature values.⁶

¹H NMR (400 MHz, CDCl₃) δ 8.74 (ddd, *J* = 4.8, 1.8, 0.9, 1H), 8.10 (dt, *J* = 7.9, 1.1, 1H), 7.78 (td, *J* = 7.8, 1.8, 1H), 7.50 – 7.26 (m, 6H), 5.44 (s, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 164.9, 149.9, 147.9, 137.0, 135.6, 128.6, 128.5, 128.4, 126.9, 125.2, 67.4.

GCMS (EI⁺) r.t. = 4.9 mins, *m/z* 213.1 [M]⁺

Synthesis of benzyl 3-(trifluoromethyl)benzoate (5n)



Synthesised according to the general method for esterification from 3-(trifluoromethyl)benzoic acid (0.105 g, 0.55 mmol) and benzyl alcohol (0.064 g, 0.59 mmol). Purified using automated flash column chromatography combiflash (eluted in 100% hexane) to give the desired product as a colourless oil in 55% yield (0.085 g).

Characterisation data were consistent with previously reported literature values.⁹

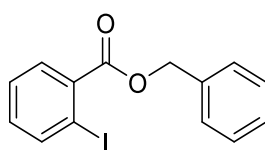
^1H NMR (400 MHz, CDCl_3) δ 8.36 (app s, 1H), 8.31 – 8.24 (m, 1H), 7.82 (m, 1H), 7.59 (tt, $J = 7.8, 0.8$, 1H), 7.54 – 7.33 (m, 5H), 5.42 (s, 2H).

^{19}F NMR (376 MHz, CDCl_3) δ -62.75 (s, 3F)

^{13}C NMR (101 MHz, CDCl_3) δ 165.2, 135.7, 133.0, 131.2 (q, $J = 33.0$), 129.7 (q, $J = 3.8$), 129.2, 128.8, 128.6, 128.5, 126.7 (q, $J = 3.8$), 125.1, 122.4, 67.4.

GCMS (EI^+) r.t = 4.6 mins, $m/z = 280.12$ $[\text{M}]^+$

Synthesis of benzyl 2-iodobenzoate (5o)



Synthesised according to the general method for esterification from 2-iodobenzoic acid (0.100 g, 0.40 mmol) and benzyl alcohol (0.052 g, 0.48 mmol). Purified using automated flash column chromatography combiflash (eluted in 100% hexane) to give the desired product as a colourless oil in 57% yield (0.077 g).

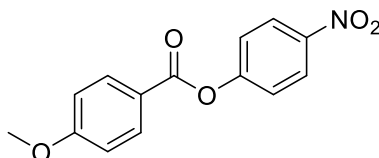
Characterisation data were consistent with previously reported literature values.¹⁰

^1H NMR (400 MHz, CDCl_3) δ 7.99 (dd, $J = 7.9, 1.2$, 1H), 7.82 (dt, $J = 7.9, 1.5$, 1H), 7.50 – 7.48 (m, 2H), 7.43 – 7.36 (m, 4H), 7.14 (td, $J = 7.7, 1.7$, 1H), 5.39 (s, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 166.3, 141.4, 135.5, 135.0, 132.8, 131.1, 128.7, 128.6, 128.5, 128.0, 94.3, 67.5.

GCMS (EI^+) r.t. = 5.5 mins, $m/z = 338.1$ $[\text{M}]^+$

Synthesis of 4-nitrophenyl 4-methoxybenzoate (5p)



Synthesised according to the general method for esterification from 4-methoxybenzoic acid (0.100 g, 0.66 mmol) and 4-nitrophenol (0.096 g, 0.66 mmol). Purified using automated flash

column chromatography combiflash (100% hexane for 6 mins to 50% hexane 50% EtOAc for 12 mins) to give the desired product as a cream solid in 49% yield (0.089 g).

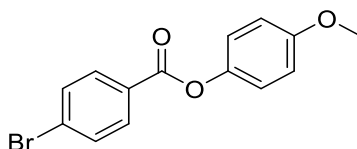
Characterisation data were consistent with previously reported literature values.¹¹

¹H NMR (400 MHz, CDCl₃) δ 8.31 (m, 2H), 8.19 – 8.11 (m, 2H), 7.44 – 7.36 (m, 2H), 7.04 – 6.96 (m, 2H), 3.91 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 164.5, 164.0, 156.1, 145.4, 132.7, 125.3, 122.8, 120.8, 114.2, 55.7.

GCMS (EI⁺) r.t. = 6.0 mins, *m/z* = 135.2 [M]⁺

Synthesis of 4-methoxyphenyl 4-bromobenzoate (5q)



Synthesised according to the general method for esterification from 4-bromobenzoic acid (0.100 g, 0.50 mmol) and 4-methoxyphenol (0.061 g, 0.50 mmol). The crude material was purified by flash column chromatography (eluted in 100% hexane) to give the desired product as a white solid in 50% yield (0.075 g).

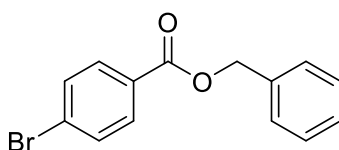
Characterisation data were consistent with previously reported literature values.¹²

¹H NMR (400 MHz, CDCl₃) δ 8.07 – 8.04 (m, 2H), 7.66 – 7.63 (m, 2H), 7.15 – 7.1 (m, 2H), 6.96 – 6.92 (m, 2H), 3.82 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 164.9, 157.5, 144.3, 132.0, 131.7, 128.8, 128.6, 122.4, 114.6, 55.7.

LCMS (ESI⁺) r.t. = 3.1 mins, *m/z* = 307.12 [M+H]⁺

Synthesis of benzyl 4-bromobenzoate (5r)



Synthesised according to the general method for esterification from 4-bromobenzoic acid (0.104 g, 0.52 mmol) and benzyl alcohol (0.061 g, 0.50 mmol). The crude material was purified by flash column chromatography (100% hexane to 5% EtOAc 95% hexane) to give the desired product as a clear oil in 69% yield (0.100 g).

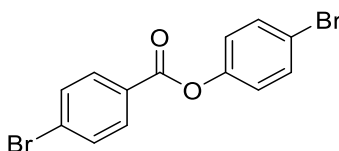
Characterisation data were consistent with previously reported literature values.¹³

¹H NMR (400 MHz, CDCl₃) δ 7.96 – 7.93 (m, 2H), 7.60 – 7.57 (m, 2H), 7.47 – 7.36 (m, 5H), 5.37 (s, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 165.8, 135.8, 131.8, 131.3, 129.1, 128.7, 128.5, 128.3, 128.3, 67.1.

GCMS (EI⁺) r.t. = 5.3 mins, *m/z* = 290.1 [M]⁺

Synthesis of 4-bromophenyl 4-bromobenzoate (5s)



Synthesised according to the general method for esterification from 4-bromobenzoic acid (0.099 g, 0.49 mmol) and 4-bromophenol (0.087 g, 0.50 mmol). Purified using automated flash column chromatography combiflash (eluted in 100% hexane) to give the product as a white solid in 18% yield (0.031 g).

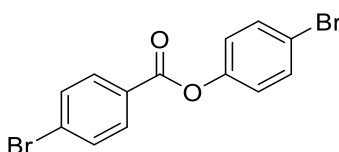
Characterisation data was consistent with previously reported literature values.¹⁴

¹H NMR (400 MHz, CDCl₃) δ 8.08 – 8.01 (m, 2H), 7.70 – 7.62 (m, 2H), 7.59 – 7.50 (m, 2H), 7.14 – 7.07 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 164.2, 149.8, 132.6, 132.1, 131.7, 129.1, 128.1, 123.5, 119.2.

GCMS (EI⁺) r.t. = 5.7 mins, *m/z* = 353.9, 355.9, 357.9 [M]⁺

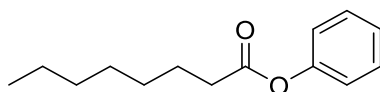
Synthesis of 4-bromophenyl 4-bromobenzoate (5s)



Alternatively synthesised from 4-bromobenzoic acid (0.113 g, 0.56 mmol) and 4-bromophenol (0.094, 0.54 mmol). Following the general method for esterification but heated in a sealed pressure tube at 100°C throughout. Purified using the combiflash equipped with a rediseq 12 g column (eluted in 100% hexanes) to yield a white solid in 77% yield (0.148 g).

Characterisation data

Synthesis of Phenyl Octanoate (5t)



Synthesised according to the general method for esterification from octanoic acid (0.099 g, 0.69 mmol) and phenol (0.065 g, 0.69 mmol). The crude material was purified by flash column chromatography (100% hexane to 10% EtOAc 90% hexane) to give the desired product as a clear oil in 79% yield (0.120 g).

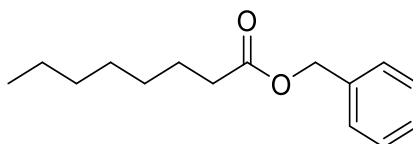
Characterisation data was consistent with previously reported literature values.¹⁵

¹H NMR (400 MHz, CDCl₃) δ 7.44 – 7.39 (m, 2H), 7.28 – 7.23 (m, 1H), 7.14 – 7.10 (m, 2H), 2.60 (t, *J* = 7.5, 2H), 1.80 (p, *J* = 7.5, 2H), 1.5 – 1.31 (m, 8H), 0.97 – 0.93 (m, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 172.4, 150.9, 129.5, 125.8, 121.7, 34.5, 31.8, 29.2, 29.0, 25.0, 22.7, 14.2.

LCMS (ESI⁺) r.t. = 3.6 mins, *m/z* = 221.3 [M+H]⁺

Synthesis of Benzyl Octanoate (5u)



Synthesised according to the general method for esterification from octanoic acid (0.099 g, 0.69 mmol) and benzyl alcohol (0.079 g, 0.73 mmol). The crude material was purified by flash

column chromatography (100% hexane to 10% EtOAc 90% hexane) to give the desired product as a clear oil in 75% yield (0.129 g).

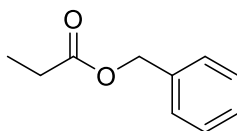
Characterisation data were consistent with previously reported literature values.¹⁶

¹H NMR (400 MHz, CDCl₃) δ 7.39 – 7.31 (m, 5H), 5.13 (s, 2H), 2.37 (t, *J* = 7.5, 2H), 1.72 – 1.62 (m, 2H), 1.37 – 1.23 (m, 8H), 0.91 – 0.87 (m, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 173.8, 136.2, 128.6, 128.3, 128.2, 66.1, 34.4, 31.8, 29.2, 29.0, 25.1, 22.7, 14.2.

GCMS (EI⁺) r.t. = 4.7, *m/z* = 234.2 [M]⁺

Synthesis of Benzyl Propionate (5v)



Synthesised according to the general method* of esterification from propionic acid (0.100 g, 1.35 mmol) and benzyl alcohol (0.152 g, 1.41 mmol). The crude material was purified using automated flash column chromatography combiflash (eluted in 100% hexane) to give the desired product as a colourless oil in 48% yield (0.107 g)

Characterisation data was consistent with previously reported literature values.¹⁷

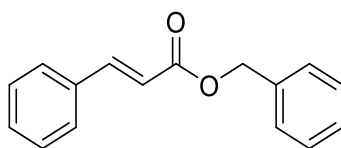
¹H NMR (400 MHz, CDCl₃) δ 7.40 – 7.32 (m, 5H), 5.13 (s, 2H), 2.40 (q, *J* = 7.6, 2H), 1.18 (t, *J* = 7.6, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 174.4, 136.2, 128.6, 128.3, 66.2, 27.7, 9.2.

GCMS (EI⁺) r.t. = 3.4 mins, *m/z* = 164.1 [M]⁺

*Performed at 35°C

Synthesis of benzyl cinnamate (5w)



Synthesised according to the general method for esterification from cinnamic acid (0.103 g, 0.070 mmol) and benzyl alcohol (0.084 g, 0.77 mmol). Purified using automated flash column

chromatography combiflash (eluted in step gradient hexane 100% to hexane 99.2% EtOAc 0.8%) to give the desired product as a colourless oil in 80% yield (0.132 g).

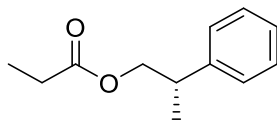
Characterisation data were consistent with previously reported literature values.¹⁸

¹H NMR (400 MHz, CDCl₃) δ 7.77 (d, *J* = 16.0, 1H), 7.55 – 7.53 (m, 2H), 7.46 – 7.38 (m, 8H), 6.52 (d, *J* = 16.0, 1H), 5.28 (s, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 166.9, 145.3, 136.2, 134.4, 130.4, 129.0, 128.7, 128.4, 128.3, 128.2, 118.0, 66.4.

LCMS (ESI⁺) r.t. = 3.1 mins, *m/z* = 239.2 [M+H]⁺

Synthesis of (*S*)-2-phenylpropyl propionate (**5x**)



Synthesised according to the general method* for esterification from propionic acid (0.101 g, 1.36 mmol) and 2-phenylpropanol (0.187 g, 1.37 mmol). The crude material was purified by flash column chromatography (hexane to 10% EtOAc 90% hexane) to give the desired product as a clear oil in 46% yield (0.120 g).

Characterisation data were consistent with previously reported literature values.¹⁹

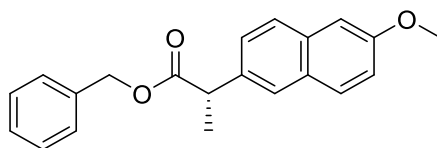
¹H NMR (400 MHz, CDCl₃) δ 7.35 (dd, *J* = 7.9, 6.9, 2H), 7.30 – 7.22 (m, 3H), 4.30 – 4.13 (m, 2H), 3.15 (h, *J* = 7.1, 1H), 2.33 (q, *J* = 7.6, 2H), 1.35 (d, *J* = 7.0, 3H), 1.14 (t, *J* = 7.6, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 174.4, 143.3, 128.5, 127.4, 126.7, 69.3, 39.0, 27.6, 18.1, 9.2.

GCMS (EI⁺) r.t. = 3.8 mins, *m/z* 193.1 [M]⁺

*Performed at 35°C

Synthesis of benzyl (S)-2-(6-methoxynaphthalen-2-yl)propanoate (5y)



Synthesised according to the general method for esterification from naproxen (0.101 g, 0.44 mmol) and benzyl alcohol (0.053 g, 0.49 mmol). Purified using automated flash column chromatography combiflash (step gradient; 100% hexane for 4 mins, 90% hexane to EtOAc for 4 mins) to yield the product as a colourless oil in 69% yield (0.095 g).

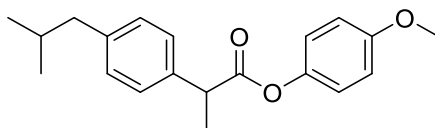
Characterisation data was consistent with previously reported literature values.²⁰

¹H NMR (400 MHz, CDCl₃) δ 7.76 – 7.71 (m, 3H), 7.46 (dd, *J* = 8.5, 1.8, 1H), 7.35 – 7.28 (m, 5H), 7.21 – 7.16 (m, 2H), 5.19 (m 2H), 3.95 (m, 4H), 1.65 (d, *J* = 7.1, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 174.6, 157.7, 136.1, 135.6, 133.8, 129.4, 129.0, 128.6, 128.2, 128.0, 127.2, 126.4, 126.1, 119.1, 105.6, 66.6, 55.4, 45.5, 18.7.

GCMS (EI⁺) r.t. = 6.4 mins, *m/z* = 320 [M+H]⁺

Synthesis of 4-methoxyphenyl 2-(4-isobutylphenyl)propanoate (5z)



Synthesised according to the general method for esterification from ibuprofen (0.100 g, 0.48 mmol) and 4-methoxyphenol (0.061 g, 0.49 mmol). Purified using automated flash column chromatography combiflash (eluted in 100% hexane) to give the desired product as a clear oil in 63% yield (0.094 g).

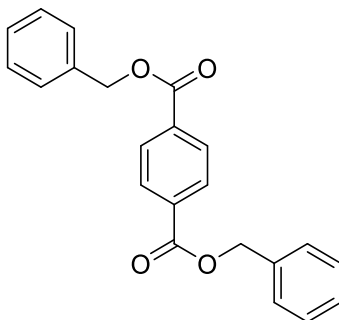
Characterisation data were consistent with previously reported literature values.²¹

¹H NMR (400 MHz, CDCl₃) δ 7.34 – 7.31 (m, 2H), 7.18 – 7.15 (m, 2H), 6.98 – 6.90 (m, 2H), 6.89 – 6.83 (m, 2H), 3.94 (q, *J* = 7.1, 1H), 3.78 (s, 3H), 2.50 (d, *J* = 7.2, 2H), 1.97 – 1.82 (m, 1H), 1.62 (d, *J* = 7.1, 3H), 0.94 (d, *J* = 6.6, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 173.7, 157.2, 144.5, 140.8, 137.5, 129.6, 127.3, 122.2, 114.4, 55.6, 45.3, 45.1, 30.3, 22.5, 18.7.

LCMS (ESI^+) r.t = 3.6 mins, m/z = 313.4 $[\text{M}+\text{H}]^+$

Synthesis of dibenzyl terephthalate (5aa)



Synthesised according to the general method for esterification from terephthalic acid (0.100 g, 0.60 mmol) and benzyl alcohol (0.130 g, 1.20 mmol). The crude material was purified by automated flash column chromatography combiflash (100% hexane 4 mins to 20% EtOAc 80% hexane using a step gradient 9 mins) to give the desired product as a white solid in 40% yield (0.082 g).

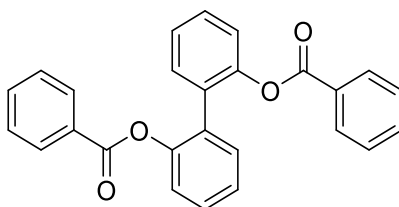
Characterisation data were consistent with previously reported literature value.²²

^1H NMR (400 MHz, CDCl_3) δ 8.16 (s, 4H), 7.49 – 7.37 (m, 10H), 5.40 (s, 4H).

^{13}C NMR (101 MHz, CDCl_3) δ 165.7, 135.7, 134.1, 129.8, 128.7, 128.5, 128.4, 67.2.

GCMS (EI^+): r.t. = 6.9 mins, m/z 346.5 $[\text{M}]^+$

Synthesis of [1,1'-biphenyl]-2,2'-diyl dibenzoate (7a)



Synthesised according to the general method for esterification from benzoic acid (0.106 g, 0.87 mmol) and 2,2'-biphenol (0.076 g, 0.41 mmol, 0.82 mmol OH equivalent). The crude material was purified by flash column chromatography (100% hexane to 7.5% EtOAc 92.5% hexane) to give the desired product as a viscous clear oil in 85% yield (0.138 g).

^1H NMR (599 MHz, cdCl_3) δ 8.00-7.98 (m, 2H), 7.57-7.54 (m, 1H), 7.43 – 7.36 (m, 4H), 7.30– 7.26 (m, 2H).

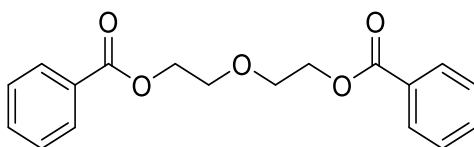
^{13}C NMR (151 MHz, cdCl_3) δ 165.0, 148.5, 133.5, 131.3, 130.7, 130.1, 129.5, 129.1, 128.5, 126.0, 122.6.

LCMS (ESI^+) r.t. = 3.3 mins, m/z = 395.3 $[\text{M}+\text{H}]^+$

HRMS – Calculated for $[\text{M}+\text{H}]^+$ $\text{C}_{26}\text{H}_{19}\text{O}_4$ 395.1283, found 395.1281

IR ν_{max} (ATR)/ cm^{-1} 3069, 1732, 1601, 1474, 1449, 1248, 1192, 1059, 700

Synthesis of oxybis(ethane-2,1-diyl) dibenzoate (7b)



Synthesised according to the general method for esterification from benzoic acid (0.097 g, 0.79 mmol) and diethylene glycol (0.044 g, 0.41 mmol, 0.82 mmol OH equivalent). The crude material was purified by flash column chromatography (100% hexane to 20% EtOAc 80% hexane) to give the desired product as a viscous clear oil in 64% yield (0.082 g).

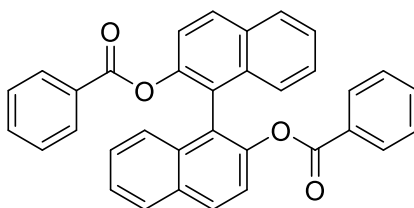
Characterisation data was consistent with previously reported literature values.²³

^1H NMR (400 MHz, CDCl_3) δ 8.05 – 8.02 (m, 4H), 7.56 – 7.51(m, 2H), 7.42 – 7.37 (m, 4H), 4.51 – 4.49 (m, 4H), 3.89 – 3.87 (m, 4H).

^{13}C NMR (101 MHz, CDCl_3) δ 166.6, 133.1, 130.1, 129.7, 128.4, 69.3, 64.1.

LCMS (ESI^+): r.t.= 2.7 mins, m/z = 315.2 $[\text{M}+\text{H}]^+$

Synthesis of [1,1'-binaphthalene]-2,2'-diyl dibenzoate (7c)



Synthesised according to the general method for esterification from benzoic acid (0.106 g, 0.87 mmol) and [1,1'-Binaphthalene]-2,2'-diol (0.120 g, 0.42 mmol, 0.84 OH equivalent). The crude

material was purified by flash column chromatography (100% hexane to 7.5% EtOAc 92.5% hexane) to give the desired product as a white solid in 72% yield (0.149 g).

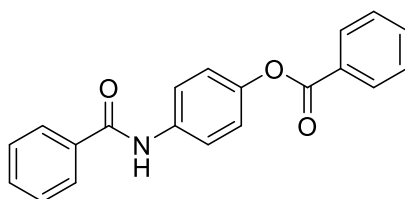
Characterisation data was consistent with previously reported literature values.²⁴

¹H NMR (400 MHz, CDCl₃) δ 8.02 – 7.98 (d, *J* = 8.4, 2H), 7.92 (dt, *J* = 8.2, 0.9, 2H), 7.75 – 7.67 (m, 4H), 7.61 (d, *J* = 8.9, 2H), 7.51 – 7.41 (m, 6H), 7.36 (ddd, *J* = 8.7, 6.6, 1.3, 2H), 7.32 – 7.22 (m, 4H).

¹³C NMR (101 MHz, CDCl₃) δ 164.8, 147.1, 133.5, 133.3, 131.6, 129.9, 129.7, 129.3, 128.3, 128.1, 126.9, 126.2, 125.8, 123.7, 121.9.

LCMS (ESI⁺) r.t.= 3.7 mins, *m/z* = 495.3 [M+H]⁺

Synthesis of 4-benzamidophenyl benzoate (7d)



Synthesised according to the general method for esterification from benzoic acid (0.100 g, 0.82 mmol) and 4-aminophenol (0.048 g, 0.44 mmol (0.88 mol equiv.)). During the course of the reaction a white solid precipitated from the reaction mixture. The solid was filtered and then triturated with MeCN (25 mL), hexane (25 mL), ethyl acetate (50 mL) and DCM (50 mL). The solid was allowed to dry and removed from the filter paper to yield a flaky white solid in 31% yield (0.040 g)

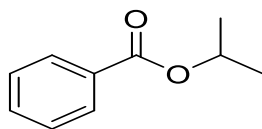
¹H NMR (599 MHz, dmsO) δ 10.38 (s, 1H), 8.16 – 8.14 (m, 2H), 7.99 – 7.97 (m, 2H), 7.88 – 7.86 (m, 2H), 7.76 – 7.74 (m, 1H), 7.63 – 7.59 (m, 3H), 7.54-7.51 (m, 2H), 7.29 – 7.28 (m, 2H).

¹³C NMR (101 MHz, DMSO) δ 165.7, 164.8, 146.3, 137.1, 134.9, 134.1, 131.7, 129.8, 129.1, 129.0, 128.5, 127.7, 122.1, 121.4.

ASAP HRMS (ESI⁺) Calculated for [M+H]⁺ C₂₀H₁₆NO₃ = 318.1130, Found = 318.1108

IR v_{max} (ATR)/cm⁻¹ 3333, 1726, 1653, 1510, 1410, 1314, 1272, 1194, 1065, 703

Synthesis of S-benzyl benzothioate (9a)



Synthesised according to the general method for thioesterification from benzoic acid (0.103 g, 0.84 mmol) and benzyl mercaptan (0.107 g, 0.86 mmol). The crude material was purified by flash column chromatography (100% hexane to 2.5% EtOAc 97.5% hexane) to give the desired product as a clear oil in 89% yield (0.166 g)

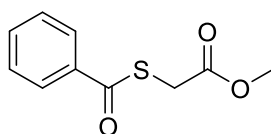
Characterisation data was consistent with previously reported literature values.²⁵

¹H NMR (400 MHz, CDCl₃) δ 8.06 – 7.98 (m, 2H), 7.58 (m, 1H), 7.51 – 7.40 (m, 4H), 7.39 – 7.24 (m, app 4H, 3H), 4.37 (s, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 191.3, 137.6, 136.8, 135.3, 129.1, 129.0, 128.9, 128.8, 128.7, 128.6, 128.3, 127.4, 33.4.

LCMS (ESI⁺) r.t. = 3.1 mins, *m/z* = 229.2 [M+H]⁺

Synthesis of methyl 2-(benzoylthio)acetate (9b)



Synthesised according to the general method for thioesterification from benzoic acid (0.107 g, 0.88 mmol) and methyl 2-mercaptoacetate (0.092 g, 0.86 mmol). The crude material was purified by flash column chromatography (100% hexane to 5% EtOAc 95% hexane) to give the desired product as a clear oil in 70% yield (0.126 g).

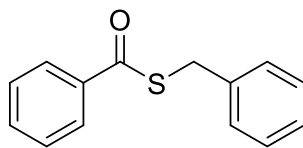
¹H NMR (400 MHz, CDCl₃) δ 7.96– 7.93 (m, 2H), 7.58-7.54 (m, 1H), 7.45 – 7.41 (m, 2H), 3.87 (s, 2H), 3.74 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 190.1, 169.3, 136.1, 133.9, 128.8, 127.4, 52.9, 31.1.

LCMS (ESI⁺) r.t. = 2.6 mins, *m/z* = 211.16 [M+H]⁺

HRMS – Calc [C₁₀H₁₁O₃S] 211.0429, Obtained – [C₁₀H₁₁O₃S] 211.0437 [M+H]⁺

Synthesis of *S*-(2-acetamidoethyl) benzothioate (**9c**)



Synthesised according to the general method for esterification from benzoic acid (0.098 g, 0.80 mmol) and *N*-(2-mercaptoethyl)acetamide (0.142 g, 1.20 mmol). The crude material was purified by flash column chromatography (100% hexane to 85% EtOAc 15% hexane) to give the desired product as white solid in 79% yield (0.141 g).

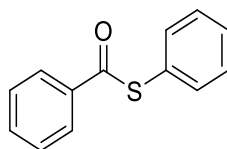
Characterisation data were consistent with previously reported literature values.²⁶

¹H NMR (400 MHz, CDCl₃) δ 7.95 – 7.90 (m, 2H), 7.58 – 7.52 (m, 1H), 7.45 – 7.40 (m, 2H), 6.36 (s, 1H), 3.49 (q, *J* = 6.3, 2H), 3.19 (t, *J* = 6.5, 2H), 1.94 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 192.2, 170.6, 136.7, 133.7, 128.7, 127.3, 39.6, 28.6, 23.2.

LCMS (ESI⁺) r.t. = 1.7 mins, *m/z* = 246.2 [M+Na]⁺

Synthesis of *S*-Phenyl benzothioate (**9d**)



Synthesised according to the general method for thioesterification from benzoic acid (0.100 g, 0.82 mmol) and thiophenol (0.090 g, 0.82 mmol). The crude material was purified using automated column chromatography combiflash (eluted in 100% hexane) to yield the desired product as cream solid in 52% yield (0.091 g).

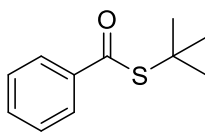
Characterisation data were consistent with previously reported literature.²⁷

¹H NMR (400 MHz, CDCl₃) δ 8.08 – 8.04 (m, 2H), 7.64 – 7.60 (m, 1H), 7.57 – 7.45 (m, 7H).

¹³C NMR (101 MHz, CDCl₃) δ 190.2, 136.7, 135.2, 133.8, 129.6, 129.4, 128.9, 127.6, 127.5.

LCMS (ESI⁺) r.t. = 3.2 mins, *m/z* = 215.4 [M+H]⁺

Synthesis of S-(tert-butyl) benzothioate (9e)



Synthesised according to the general method for thioesterification from benzoic acid (0.099 g, 0.81 mmol) and 2-methylpropane-2-thiol (0.074 g, 0.82 mmol). The crude material was purified using automated column chromatography (eluted in 100% hexane). The fractions were contaminated with ether side product so the fractions were re-purified using manual column chromatography (100% hexane to 90% hexane and 10% EtOAc) to yield the desired product as a colourless oil in 35% yield (0.052 g).

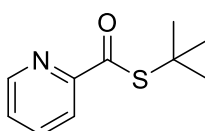
Characterisation data were consistent with previously reported literature values.²⁸

¹H NMR (400 MHz, CDCl₃) δ 7.95 – 7.89 (m, 2H), 7.56 – 7.48 (m, 1H), 7.45 – 7.37 (m, 2H), 1.58 (s, 9H).

¹³C NMR (101 MHz, CDCl₃) δ 192.9, 138.3, 132.9, 128.5, 127.0, 48.2, 30.0.

LCMS (ESI⁺) r.t = 3.2 mins, *m/z* 195.2 [M+H]⁺

Synthesis of S-(tert-butyl) pyridine-2-carbothioate (9f)



Synthesised according to the general method for thioesterification from 2-picolinic acid (0.100 g, 0.81 mmol) and 2-methylpropane-2-thiol (0.074g, 0.82 mmol). The crude material was purified using automated column chromatography (product eluted in 100% hexane) to yield the desired product as a white solid in 59% yield (0.093 g)

¹H NMR (400 MHz, CDCl₃) δ 8.64 (ddd, *J* = 4.8, 1.7, 0.9 Hz, 1H), 7.94 – 7.88 (m, 1H), 7.83 (td, *J* = 7.7, 1.7 Hz, 1H), 7.47 (ddd, *J* = 7.5, 4.8, 1.3 Hz, 1H), 1.57 (s, 9H).

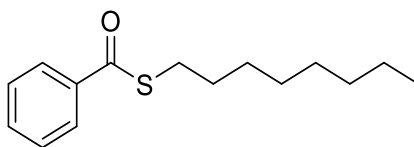
¹³C NMR (151 MHz, cdcl₃) δ 193.8, 153.3, 149.0, 137.4, 127.6, 120.1, 47.1, 47.1, 29.9.

LCMS (ESI⁺) r.t = 2.5 mins, *m/z* 196.2 [M+H]⁺

HRMS – Calculated [C₁₀H₁₄NOS] 196.0796, Found [C₁₀H₁₄NOS] 196.0794

IR

Synthesis of Octyl Benzothioate (9g)



Synthesised according to the general method for thioesterification from (0.100 g, 0.82 mmol) and octanthiol (0.140g, 0.96 mmol). The crude material was purified by flash column chromatography (100% hexane to 5% EtOAc 95% hexane) to give the desired product as a colourless oil in 40% yield (0.081g).

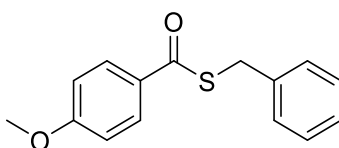
Characterisation data was consistent with previously reported literature.²⁷

¹H NMR (400 MHz, CDCl₃) δ 7.99 – 7.96 (m, 2H), 7.55 – 7.53 (m, 1H), 7.45 – 7.41 (m, 2H), 3.09 – 3.05 (m, 2H), 1.69 – 1.63 (m, 2H), 1.44 – 1.27 (m, 9H), 0.90 – 0.87 (m, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 192.2, 137.4, 133.3, 128.6, 127.3, 31.9, 29.7, 29.3, 29.2, 29.2, 29.1, 22.8, 14.2.

LCMS (ESI⁺) r.t. = 4.2 mins, *m/z* = 251.3 [M+H]⁺

Synthesis of S-benzyl 4-methoxybenzothioate (9h)



Synthesised according to the general method for thioesterification from 4-methoxybenzoic acid (0.099 g, 0.65 mmol) and benzyl mercaptan (0.089 g, 0.72 mmol). The crude material was purified using automated flash column chromatography combiflash (product eluted in 100% hexane) to yield the desired product as a white solid in 49% (0.083 g).

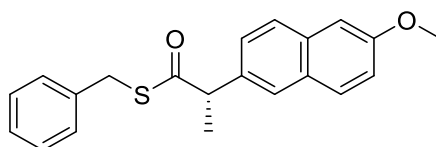
Characterisation data were consistent with previously reported literature values.²⁹

^1H NMR (400 MHz, CDCl_3) δ 8.01 – 7.97 (m, 2H), 7.43 – 7.40 (m, 2H), 7.37 – 7.33 (m, 2H), 7.31 – 7.27 (m, 1H), 6.97 – 6.93 (m, 2H), 4.34 (s, 2H), 3.88 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 189.8, 163.9, 137.8, 129.7, 129.6, 129.1, 128.7, 127.3, 113.9, 55.6, 33.3.

GCMS (EI^+) r.t = 5.8 mins, m/z = 258.2 [M] $^+$

Synthesis of S-benzyl 2-(6-methoxynaphthalen-2-yl)propanethioate (9i)



Synthesised according to the general method for thioesterification from Naproxen (0.100 g, 0.43 mmol) and benzyl mercaptan (0.054 g, 0.43 mmol). The crude material was purified using automated column chromatography (100% hexanes 5.5 mins, gradient to 10% EtOAc 90% hexane 0.5 mins, 10% EtOAc 90% hexane 2 mins followed by gradient to 50/50 EtOAc/hexane) to yield the desired product as a colourless oil in 68% yield (0.100 g)

^1H NMR (599 MHz, CDCl_3) δ 7.77 – 7.72 (m, 3H), 7.46 (dd, J = 8.5, 1.9, 1H), 7.32 – 7.25 (m, 5H), 7.21-7.16 (m, 2H), 4.19 – 4.05 (m, 3H), 3.95 (s, 3H), 1.68 (d, J = 7.1, 3H).

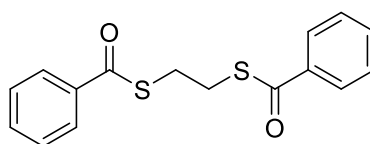
^{13}C NMR (151 MHz, CDCl_3) δ 200.8, 157.9, 137.5, 134.9, 134.0, 129.5, 129.0, 128.9, 128.7, 127.3, 127.3, 126.8, 126.5, 119.2, 105.7, 55.4, 54.1, 33.6, 18.5.

LCMS (ESI^+) r.t = 3.5 mins, m/z = 337.3 [$\text{M}+\text{H}$] $^+$

HRMS – Calculated [$\text{C}_{21}\text{H}_{21}\text{O}_2\text{S}$] 337.1262, Found [$\text{C}_{21}\text{H}_{21}\text{O}_2\text{S}$] 337.1272 [$\text{M}+\text{H}$] $^+$

IR ν_{max} (ATR)/ cm^{-1} 3029, 2938, 1677, 1601, 1268, 1229, 1029, 944, 854, 818, 750, 698

Synthesis of S,S'-(ethane-1,2-diyl) dibenzothioate (9j)



Synthesised according to the general method for thioesterification from benzoic acid (0.100 g, 0.82 mmol) and ethandithiol (0.35 mL, 0.41 mmol, 0.82 SH equivalent). The crude material

was purified using automated column chromatography (product eluted in 100% hexane) to give the desired product as a cream solid in 37% yield (0.046 g).

Characterisation data were consistent with previously reported literature values.³⁰

¹H NMR (400 MHz, CDCl₃) δ 8.01 – 7.95 (m, 4H), 7.62 – 7.55 (m, 2H), 7.50 – 7.41 (m, 4H), 3.36 (s, 4H).

¹³C NMR (101 MHz, CDCl₃) δ 191.4, 136.8, 133.7, 128.8, 127.4, 29.0.

LCMS (ESI⁺) r.t = 3.6 mins, *m/z* = 303.2 [M+H]⁺

Acyl Fluoride Reaction Monitoring

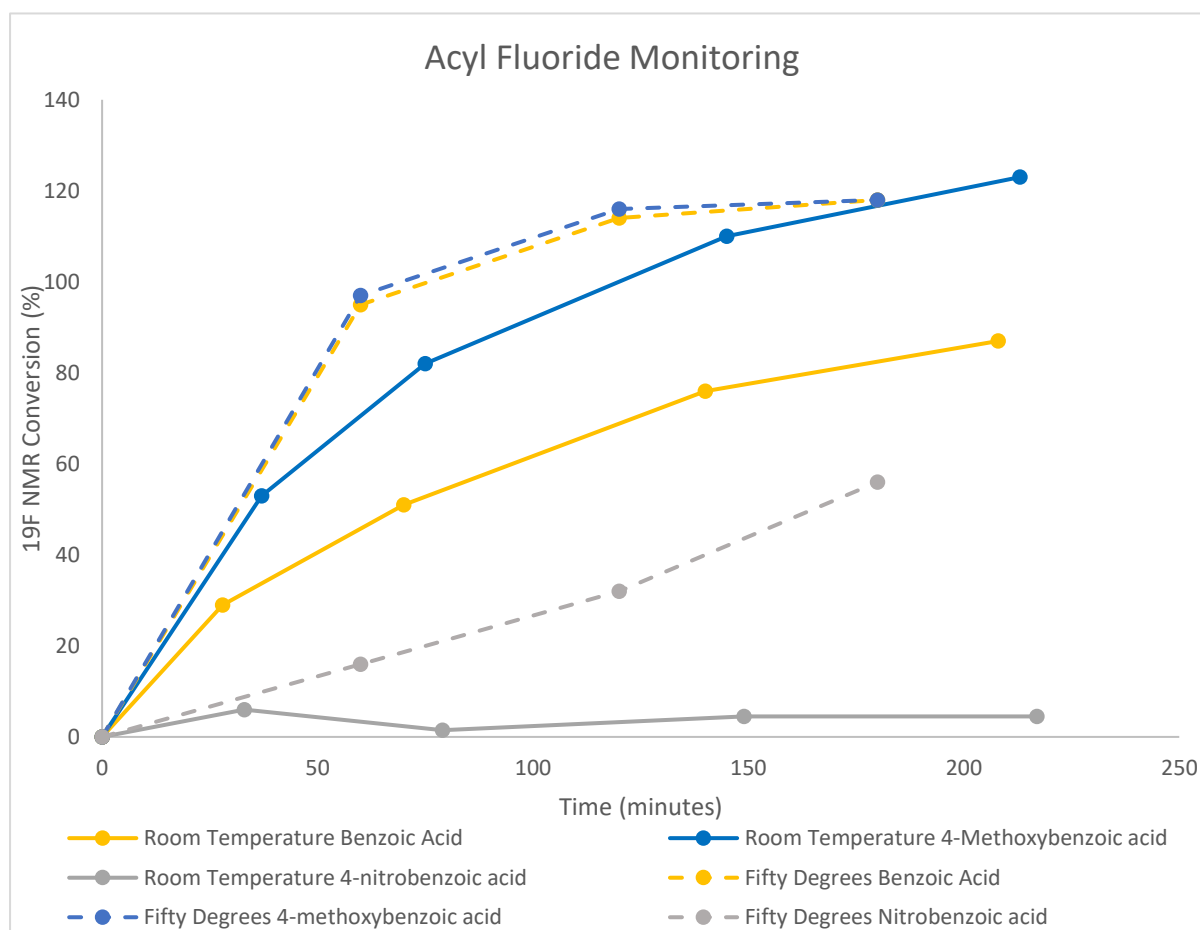
To an oven dried vial was added carboxylic acid (1 equiv.), DIPEA (2 equiv.), PFP (1.1 equiv.), fluorobenzene (1 equiv.) and CD₃CN (0.6 mL). The contents were then transferred from the vial to a clean, dry NMR tube stoppered with a lid.

For rt analysis the NMR tubes were placed into the NMR instrument autosampler and left there for the duration of the experiment.

For reactions conducted above rt the NMR tubes were transferred to a thermostatically controlled water bath set at 50 °C, the tubes were removed after 1 h and an NMR spectrum recorded. When the acquisition was complete the tube was retrieved and placed back into the water bath.

The time points recorded equates to the amount of time the sample was held in the water bath.

NMR spectra were analysed by direct integration to the resonance corresponding to the acyl fluoride compared to the integration value of the internal standard.



Raw data for acyl fluoride monitoring at room temperature

Benzoic Acid		4-methoxybenzoic acid		4-nitrobenzoic acid*	
mins	Area AF	mins	Area AF	mins	Area AF
0	0	0	0	0	0
28	0.29	37	0.53	33	0.06
70	0.51	75	0.82	79	0.015
140	0.76	145	1.1	149	0.045
208	0.87	213	1.23	217	0.045

*A 1.5 equiv of fluorobenzene was added, areas of acyl fluorides were multiplied by 1.5 to accommodate.

Raw data for acyl fluoride monitoring 50 °C

Benzoic Acid		4-methoxybenzoic acid		4-nitrobenzoic acid	
Mins**	Area AF	Mins**	Area AF	Mins**	Area AF
0	0	0	0	0	0
60	0.95	60	0.97	60	0.16
120	1.14	120	1.16	120	0.32
180	1.18	180	1.18	180	0.56

**Minutes spent in the water bath

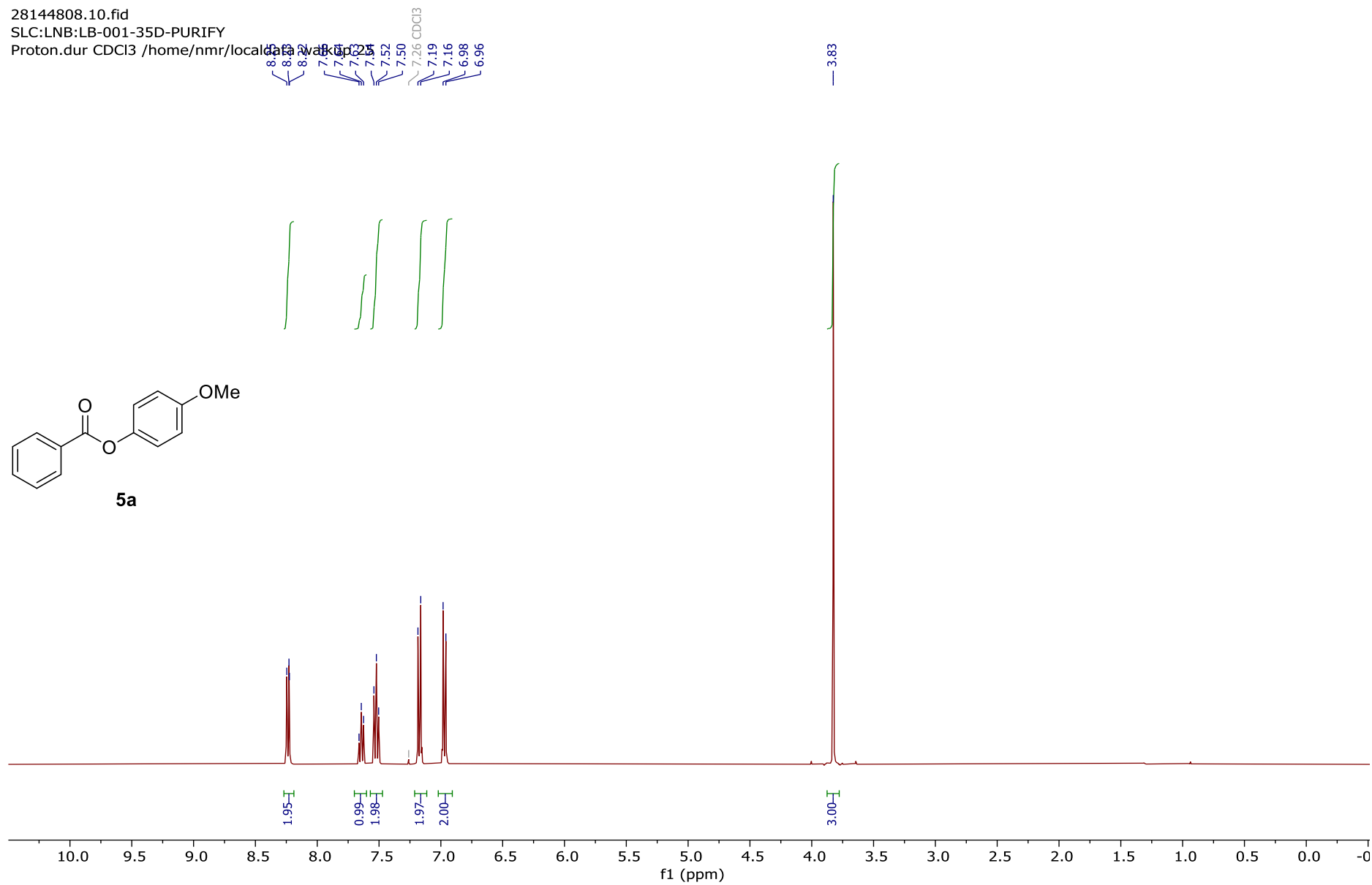
The areas in excess of the expected 1:1 ratio is attributed to different relaxation times. It was not feasible to employ a longer relaxation time on the NMR spectrometers used for this analysis. The relaxation time used was 2.77 seconds.

NMR Data for Synthesised Compounds

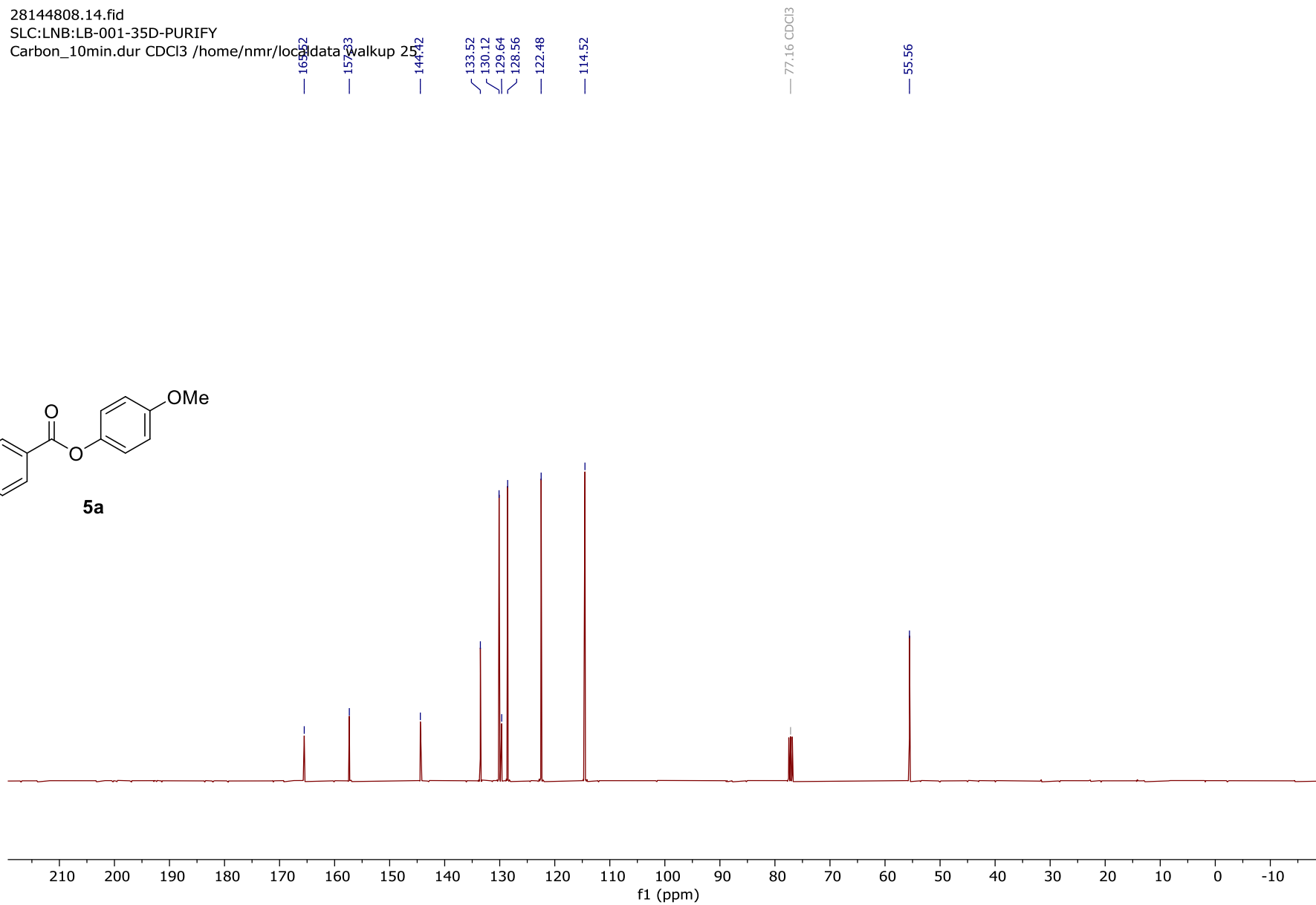
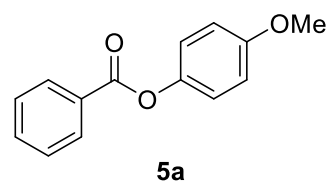
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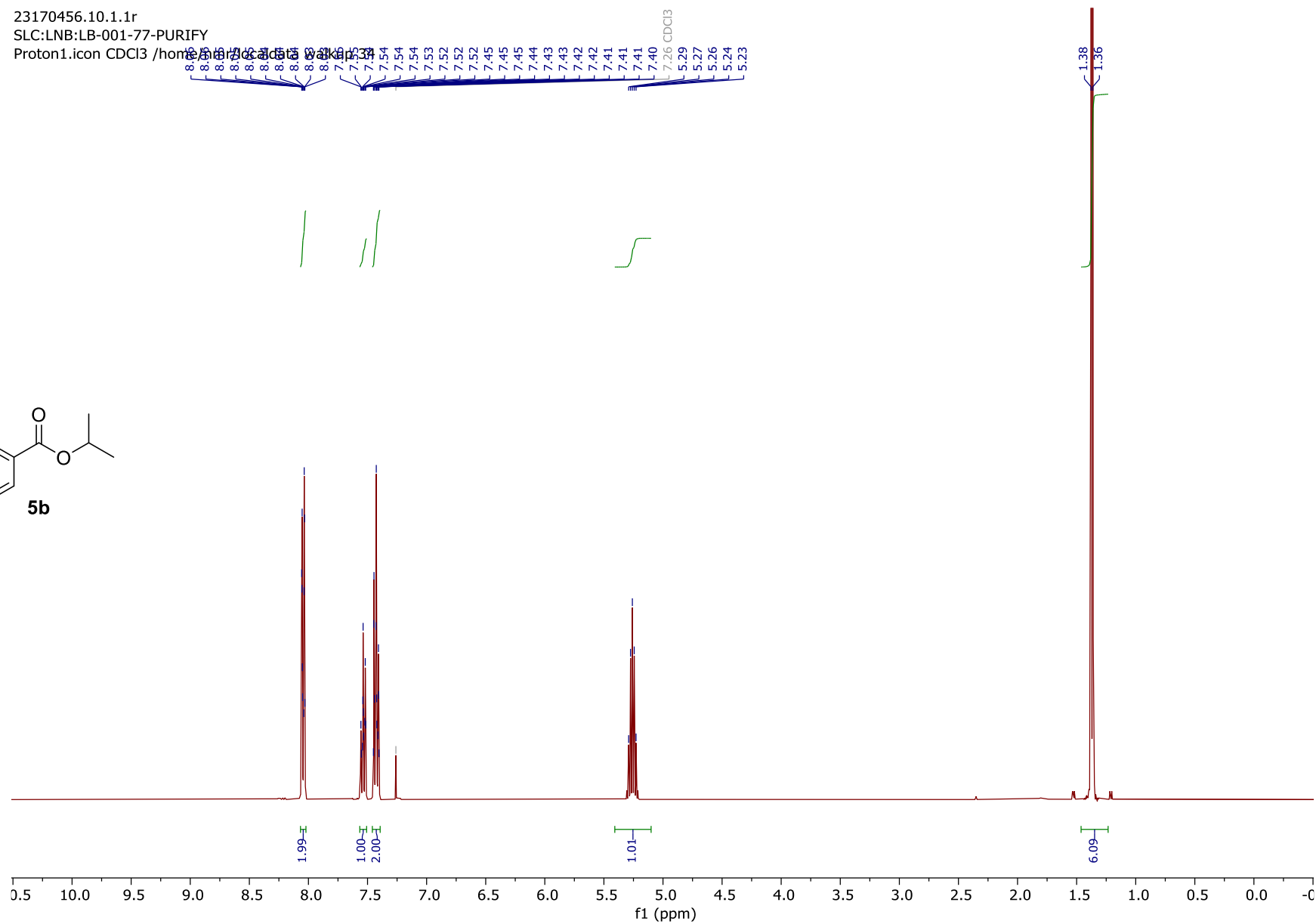
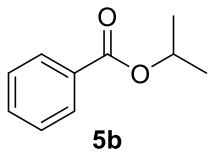


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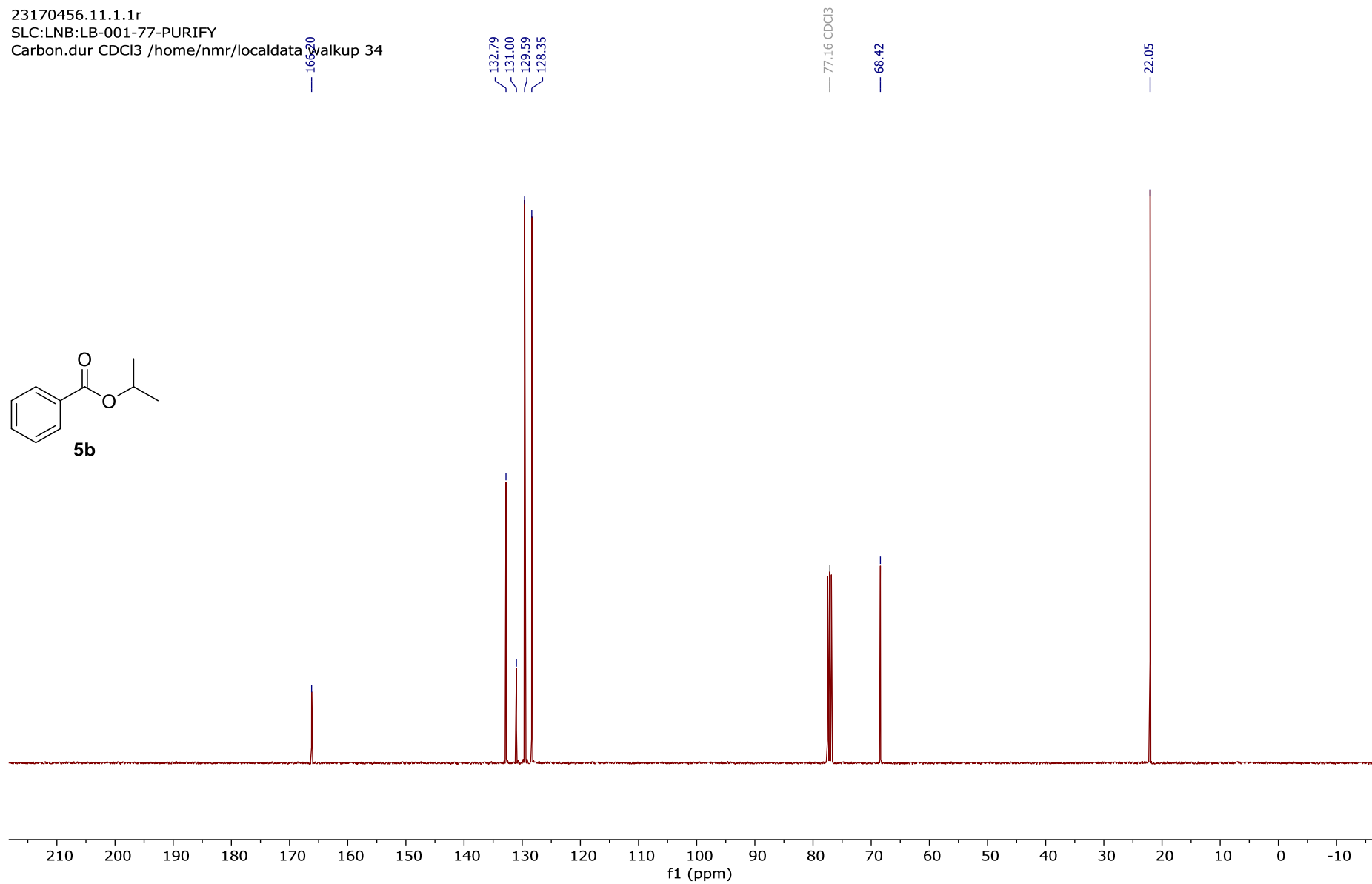


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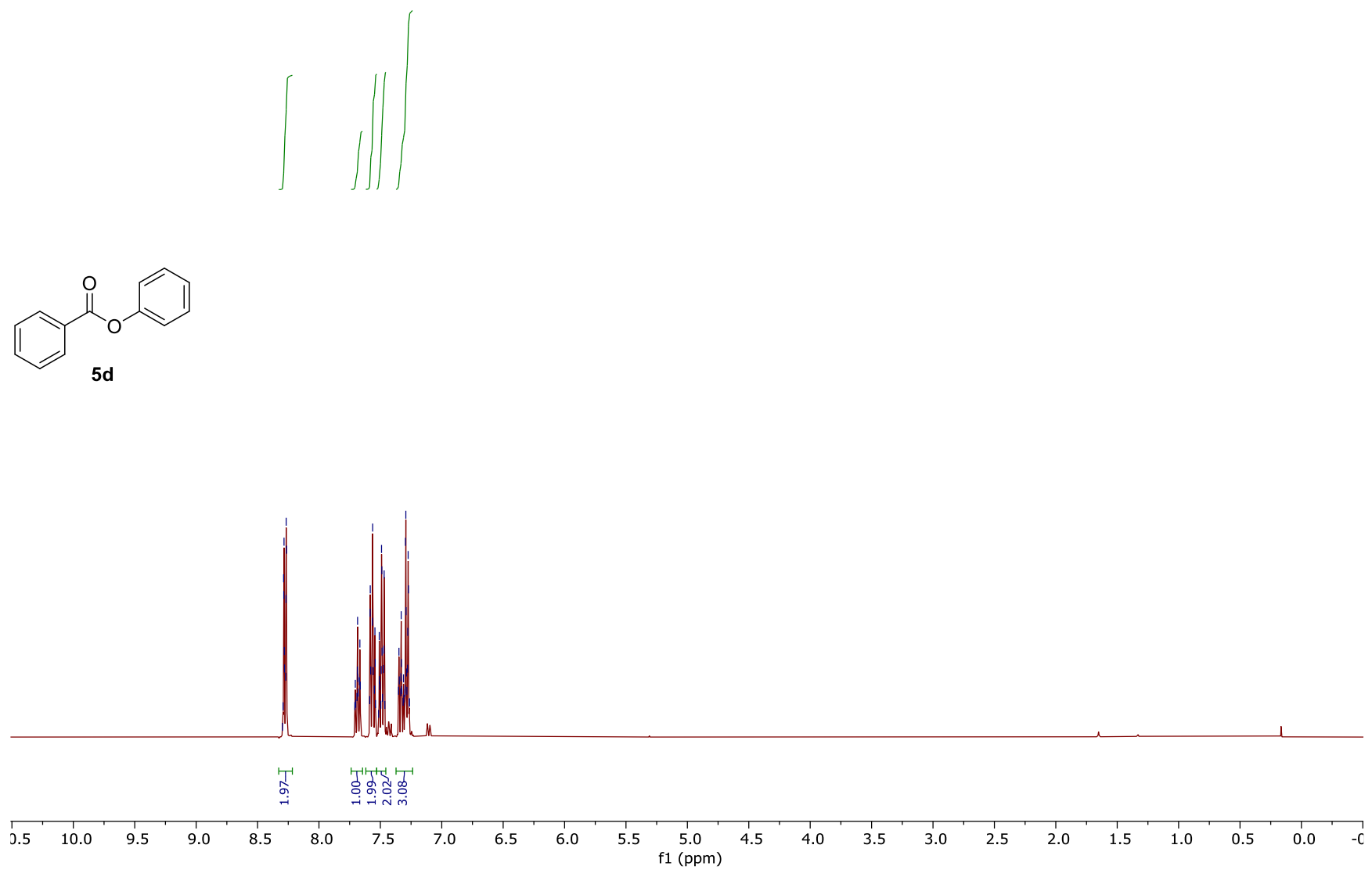
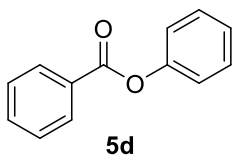
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5.24
5.23



23170456.11.1.1r
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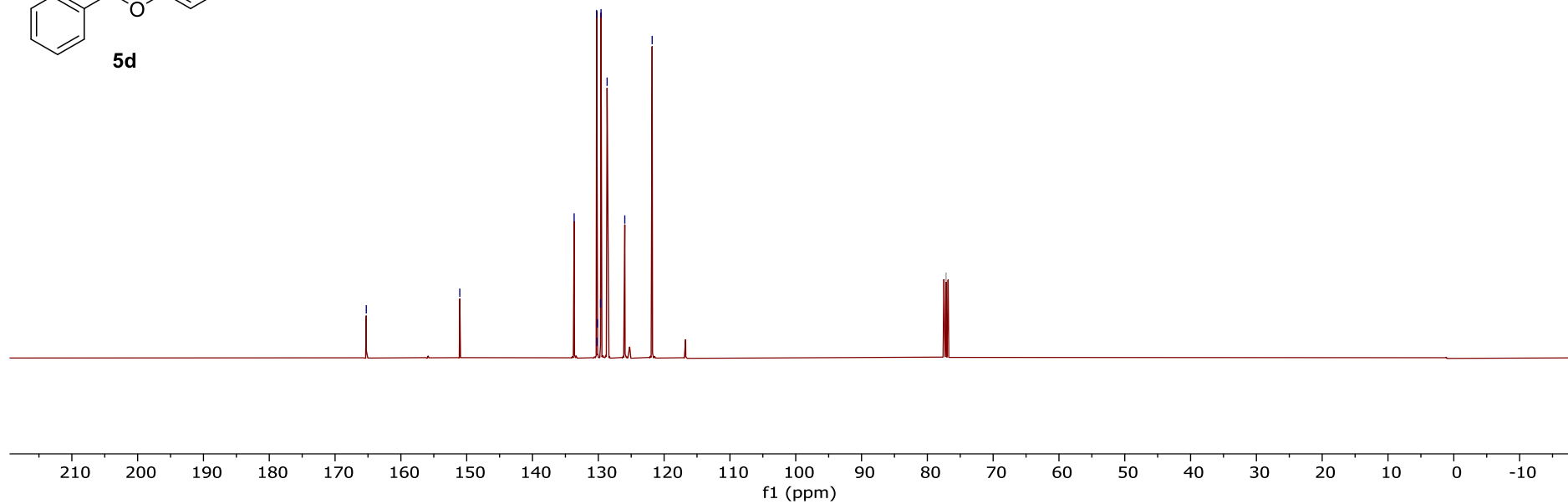
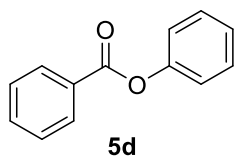
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Carbon.dur CDCl3 /home/nmr/localdata/walkup 15

166.56

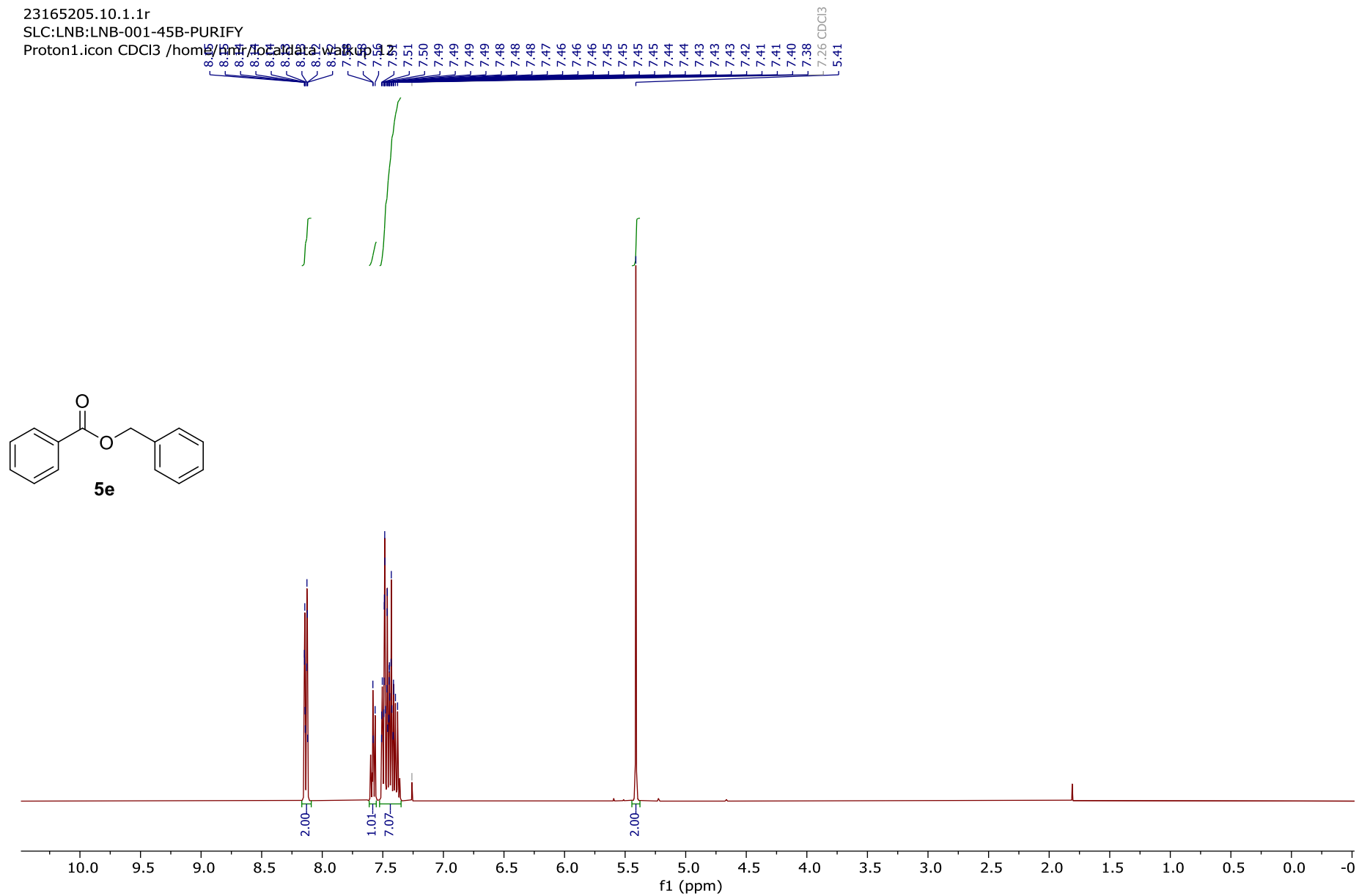
151.05

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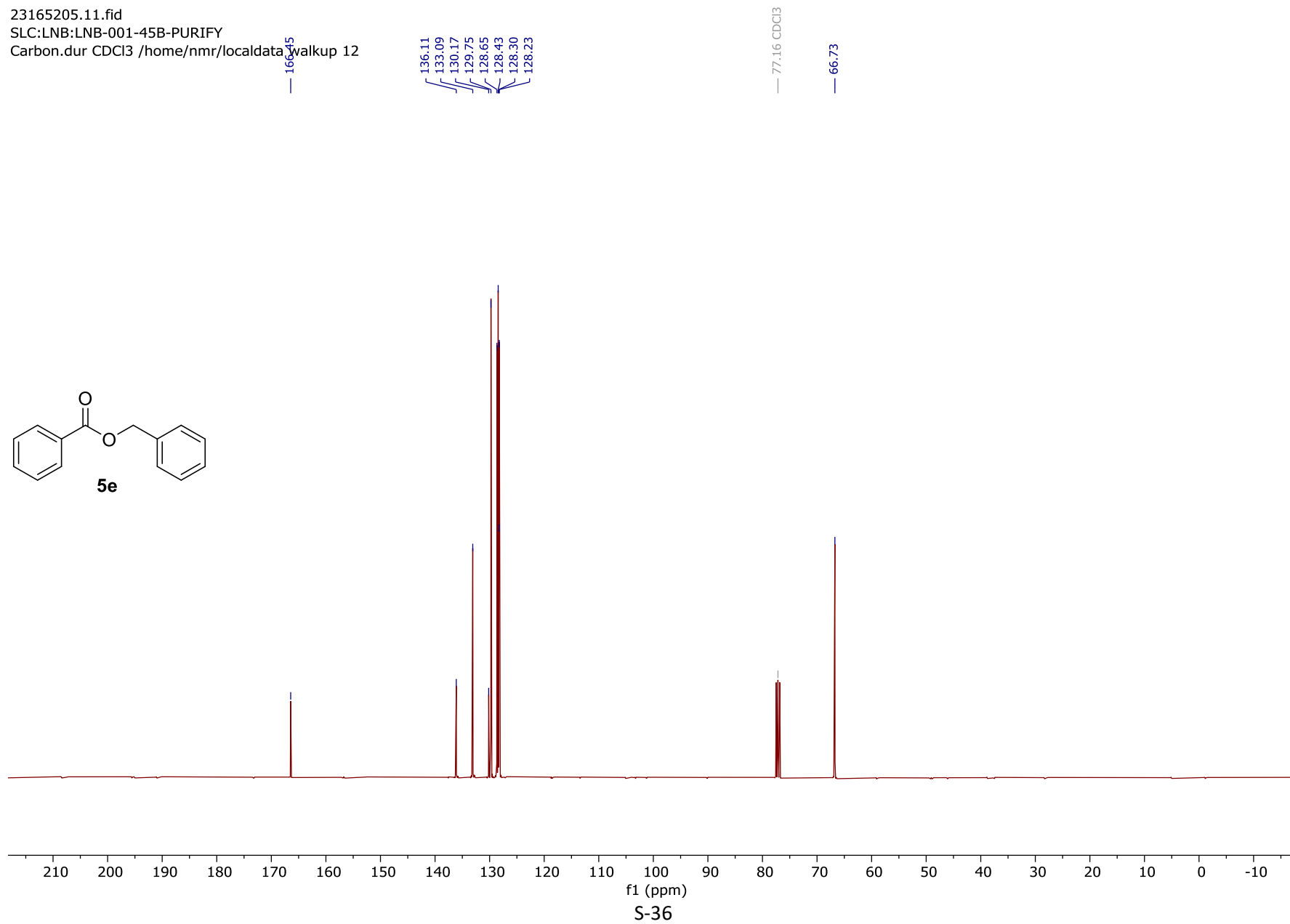
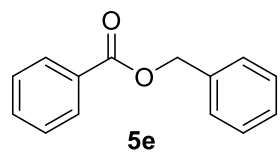
77.16 CDCl3



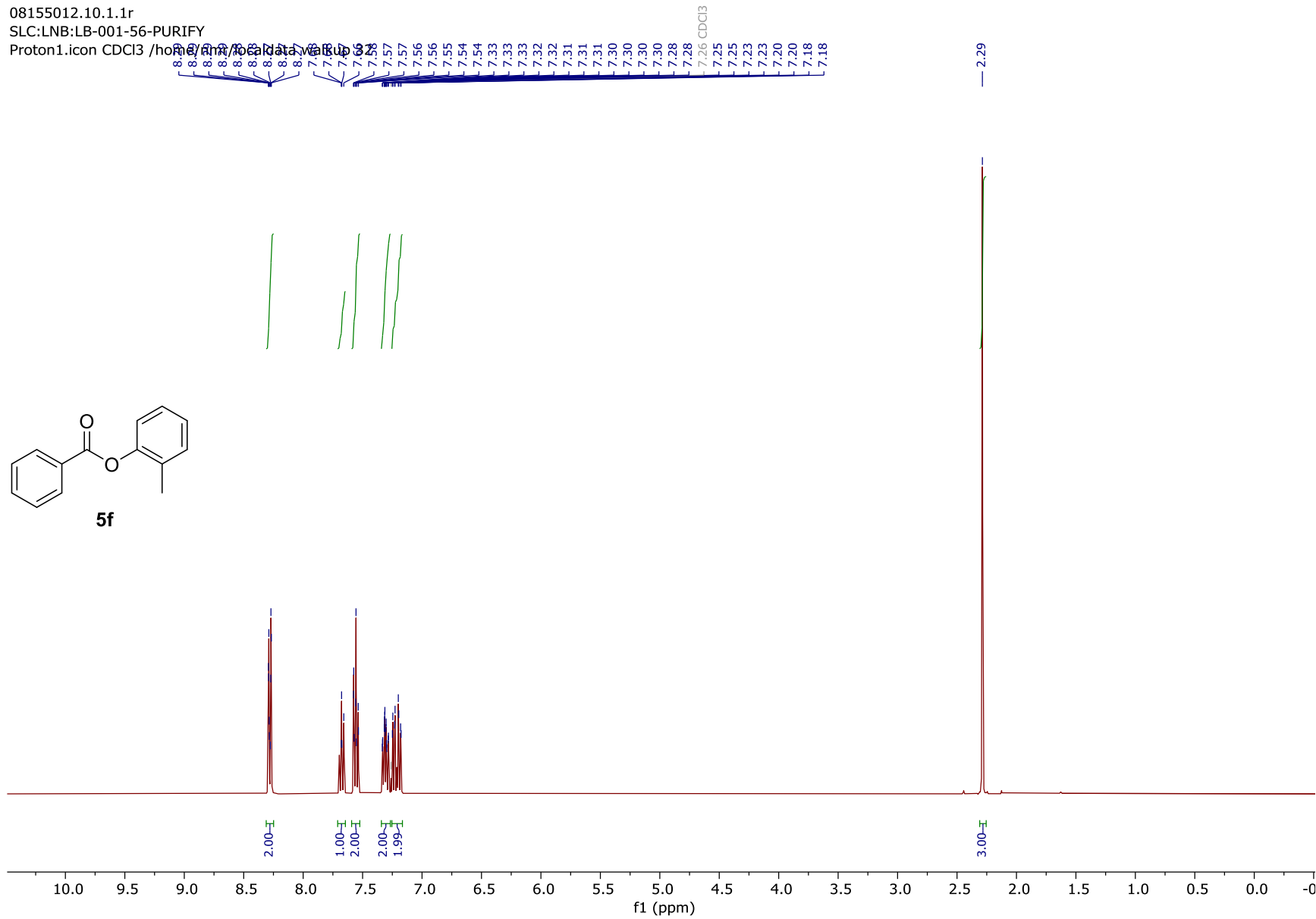
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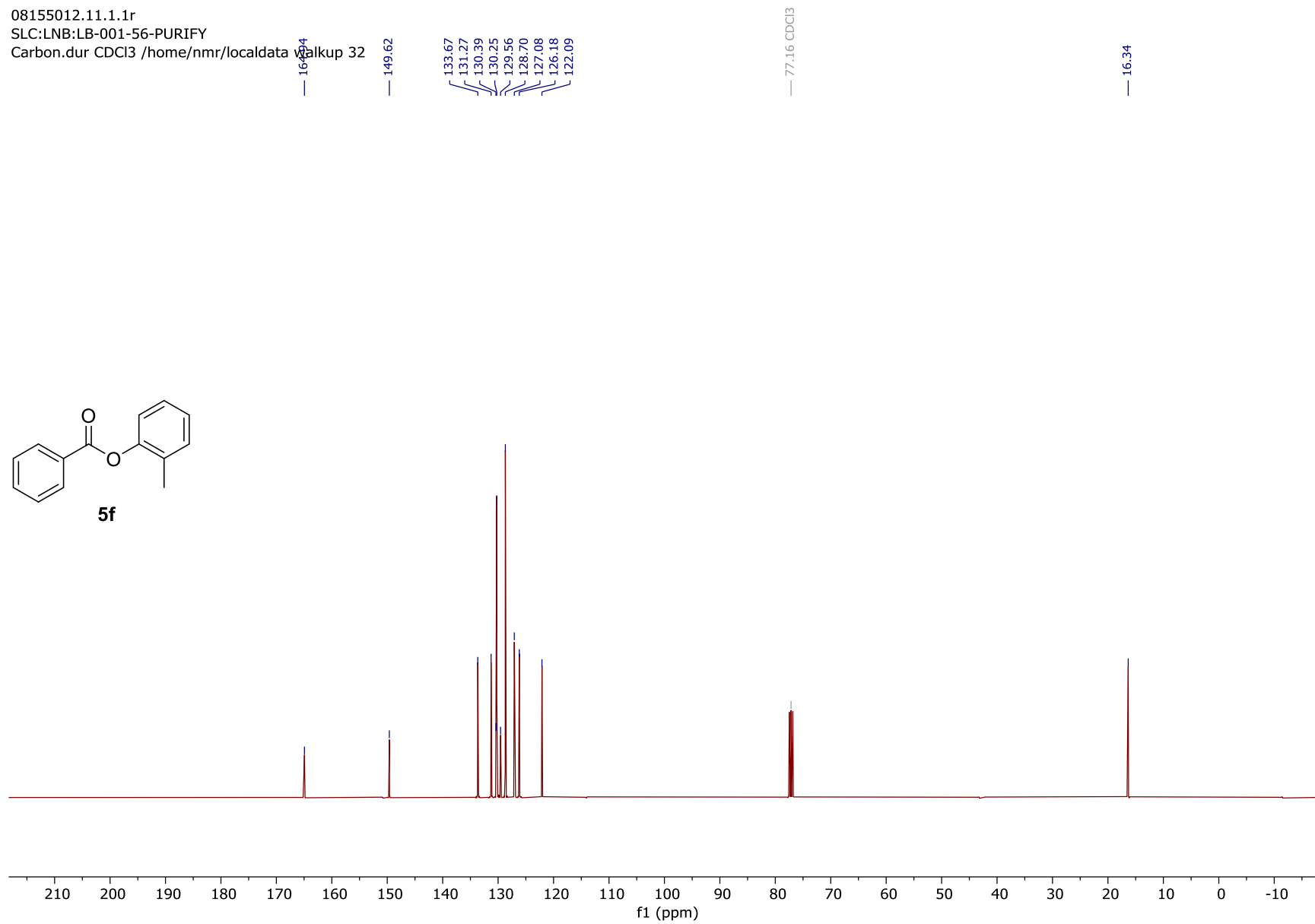
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Carbon.dur CDCl3 /home/nmr/localdata/walkup 12



08155012.10.1.1r
SLC:LNB:LB-001-56-PURIFY
Proton1.icon CDCl3 /home/ana/08155012



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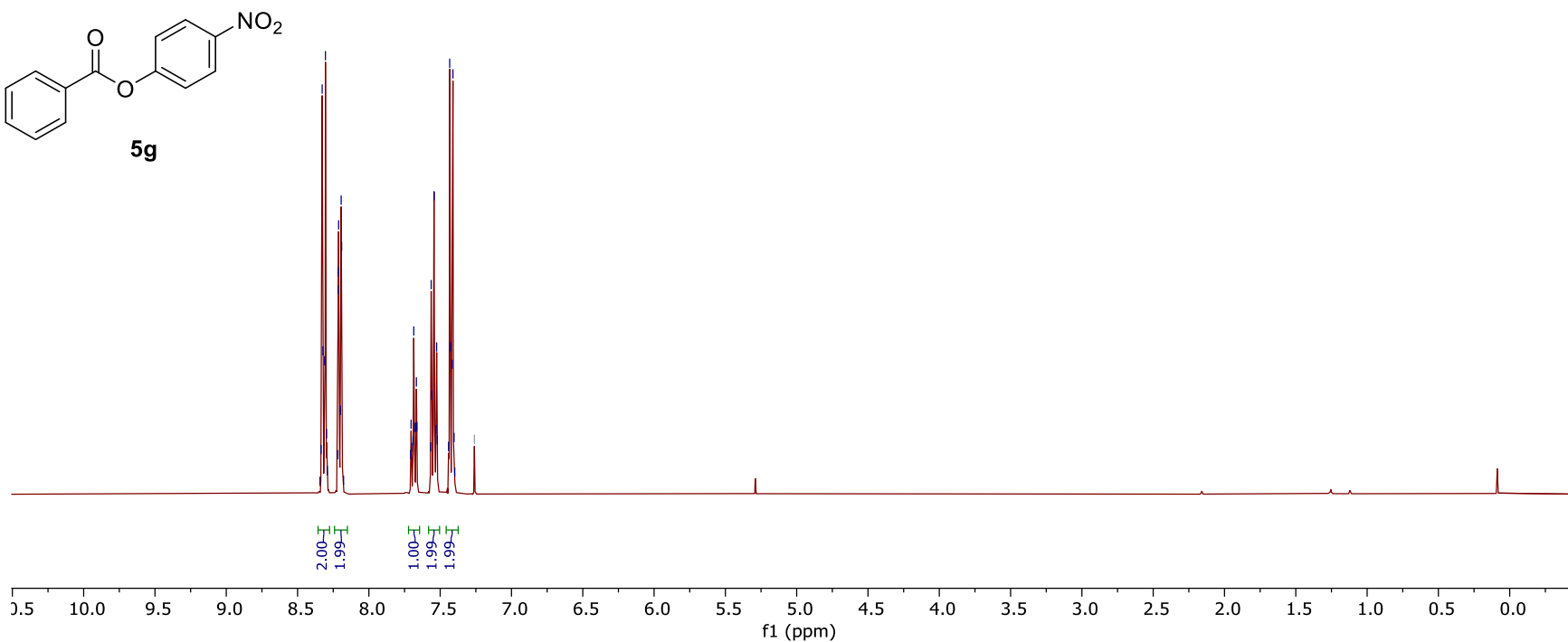
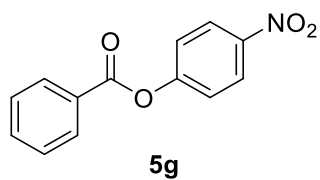
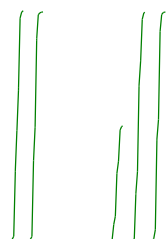
18162932.10.1.1r

SLC:LNB:LB-001-45A-PURIFY

Proton1 in CDCl3 Phenyl Nitro Data

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- 7.26

CDCl3



S-39

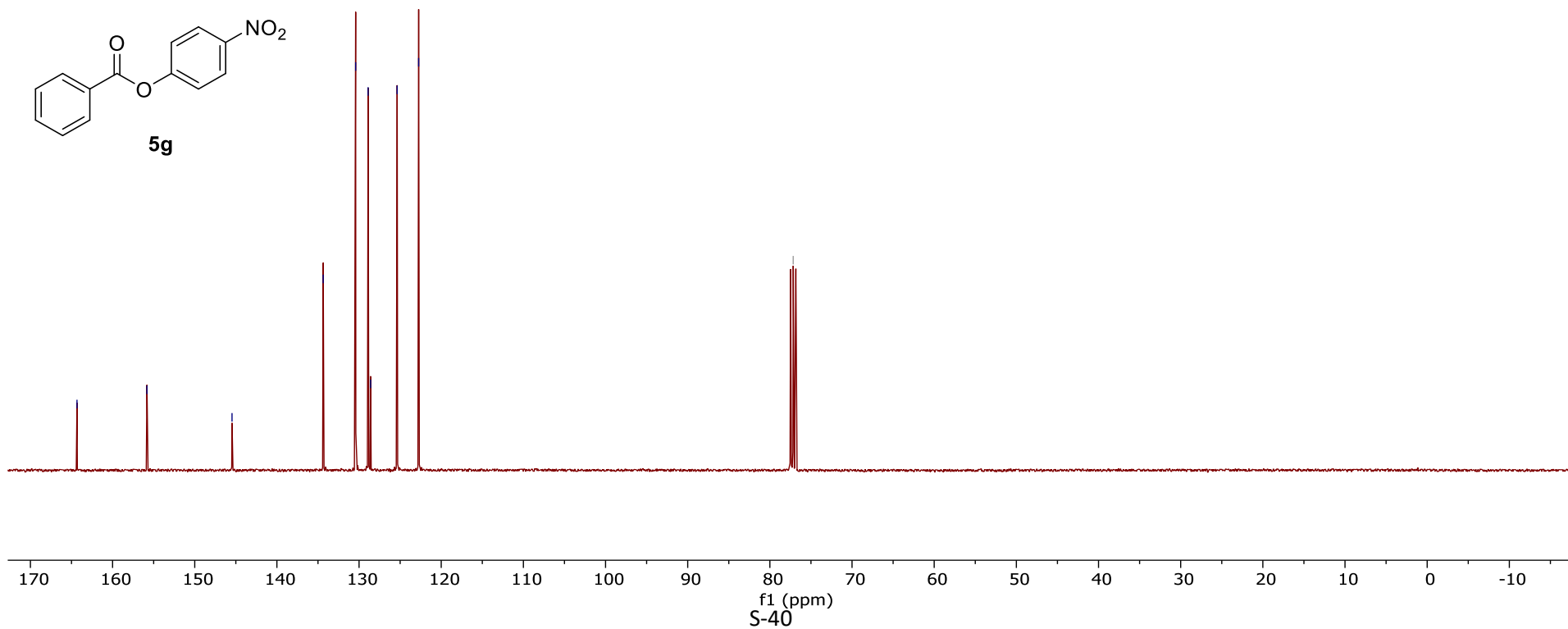
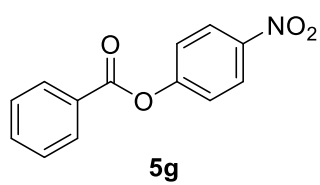
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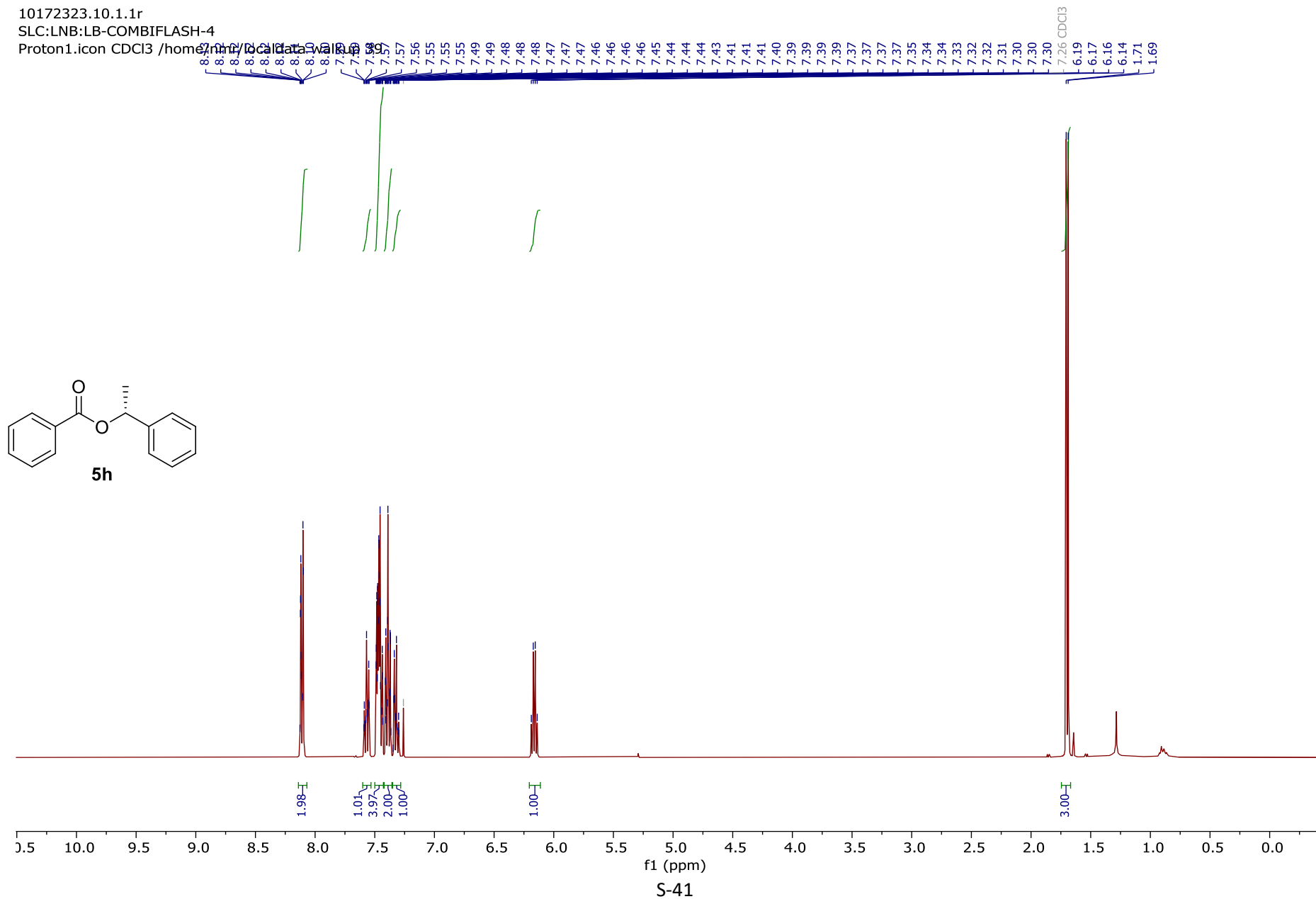
Carbon sur CDC13 /home/jamr/localdata

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130.42
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128.80
122.67
122.56

77.16 CDC13



10172323.10.1.1r
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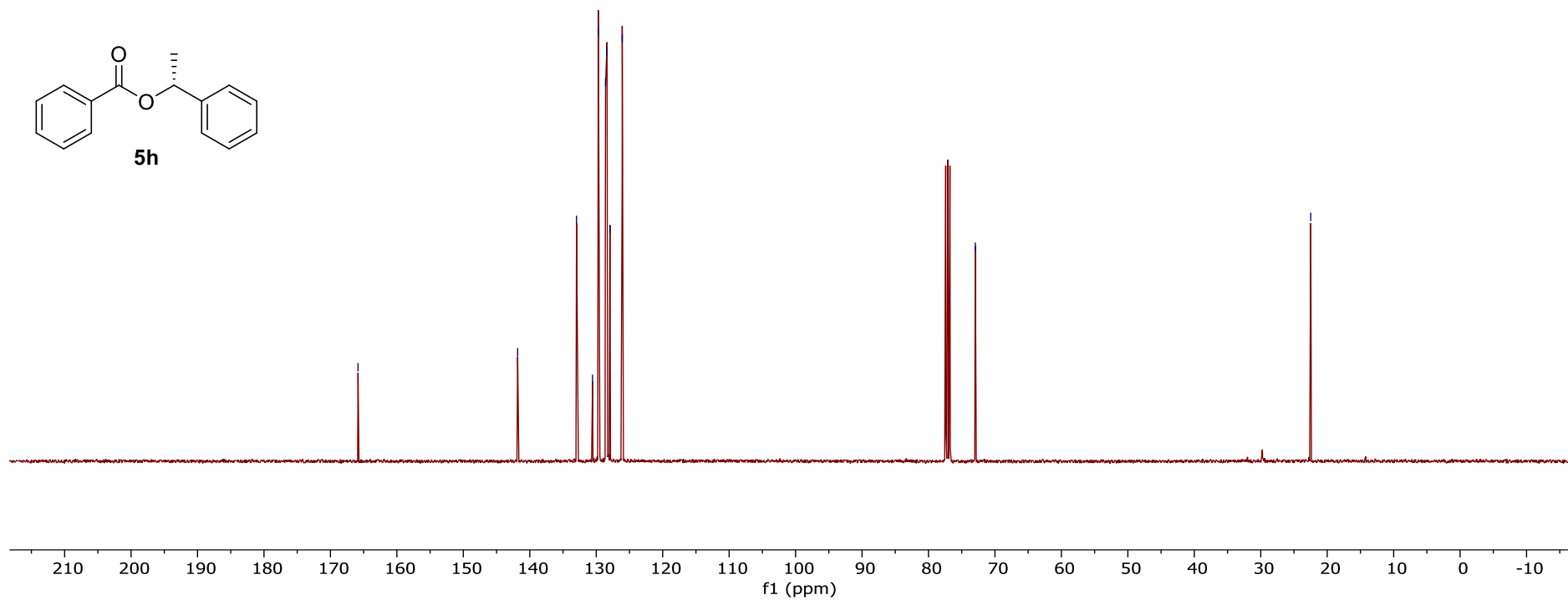
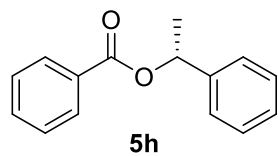


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SLC:LNB:LB-COMBIFLASH-4
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128.38
127.94
126.09

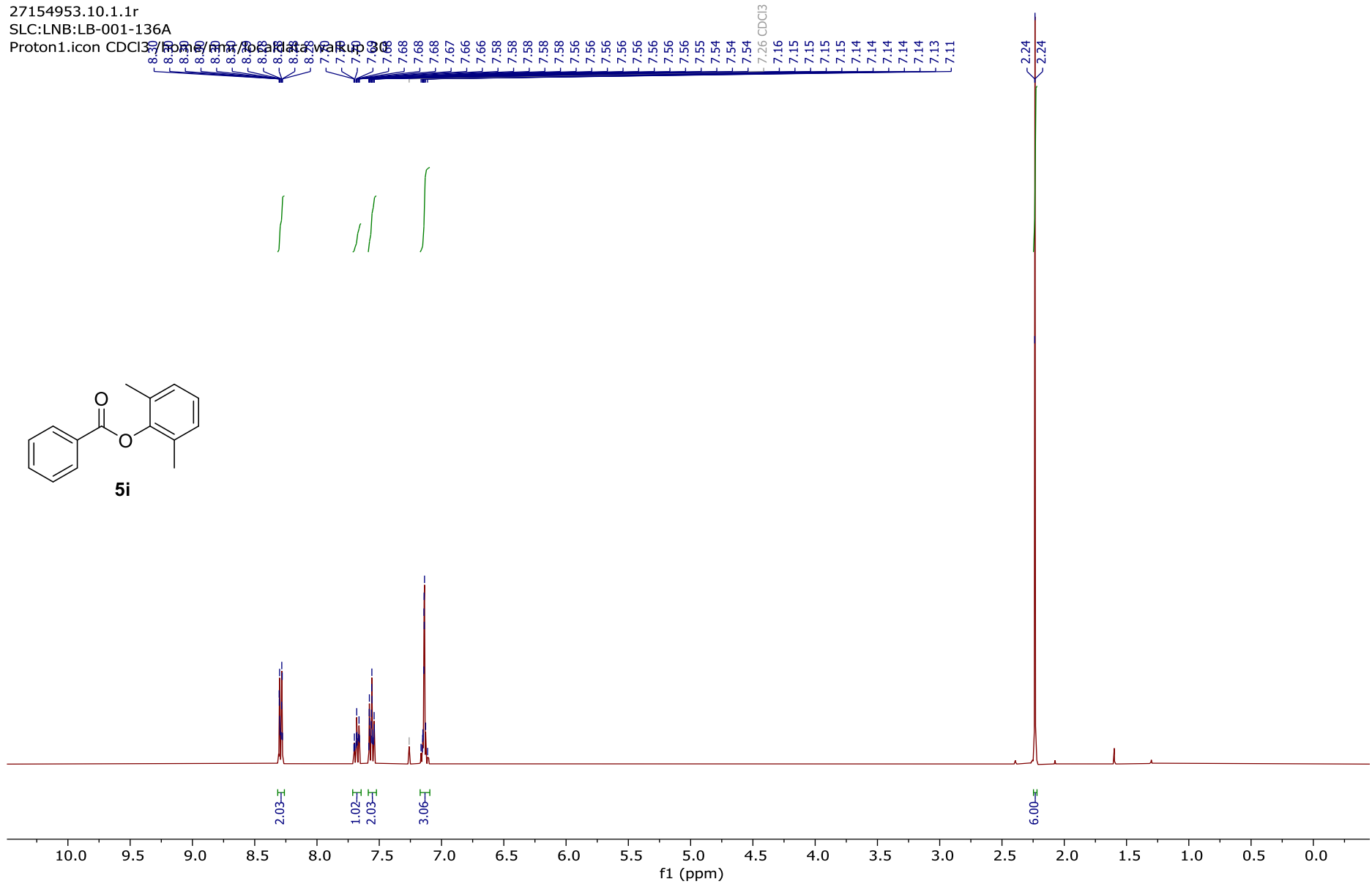
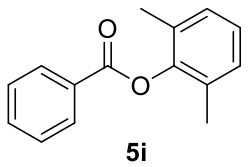
72.96

22.48



27154953.10.1.1r
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Proton1.icon CDCl3

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27154953.11.1.1r

SLC:LNB:LB-001-136A

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133.69

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130.28

129.39

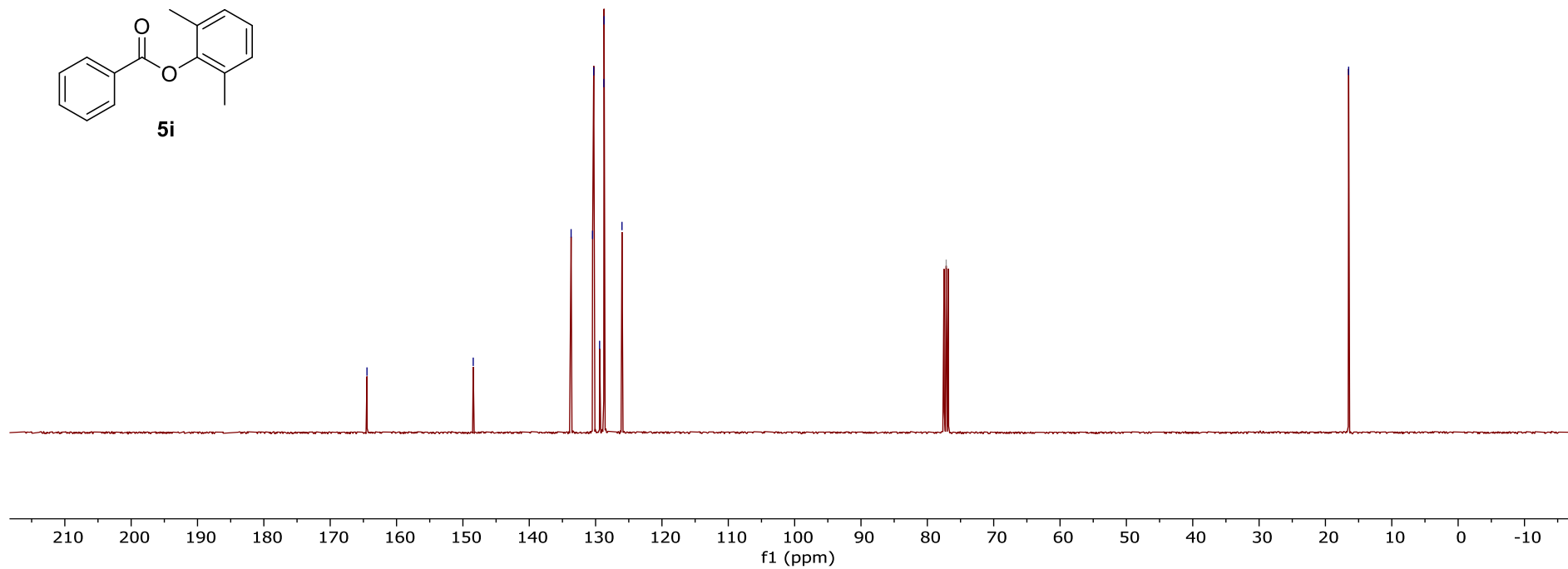
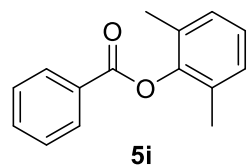
128.75

128.72

126.02

77.16 CDCl3

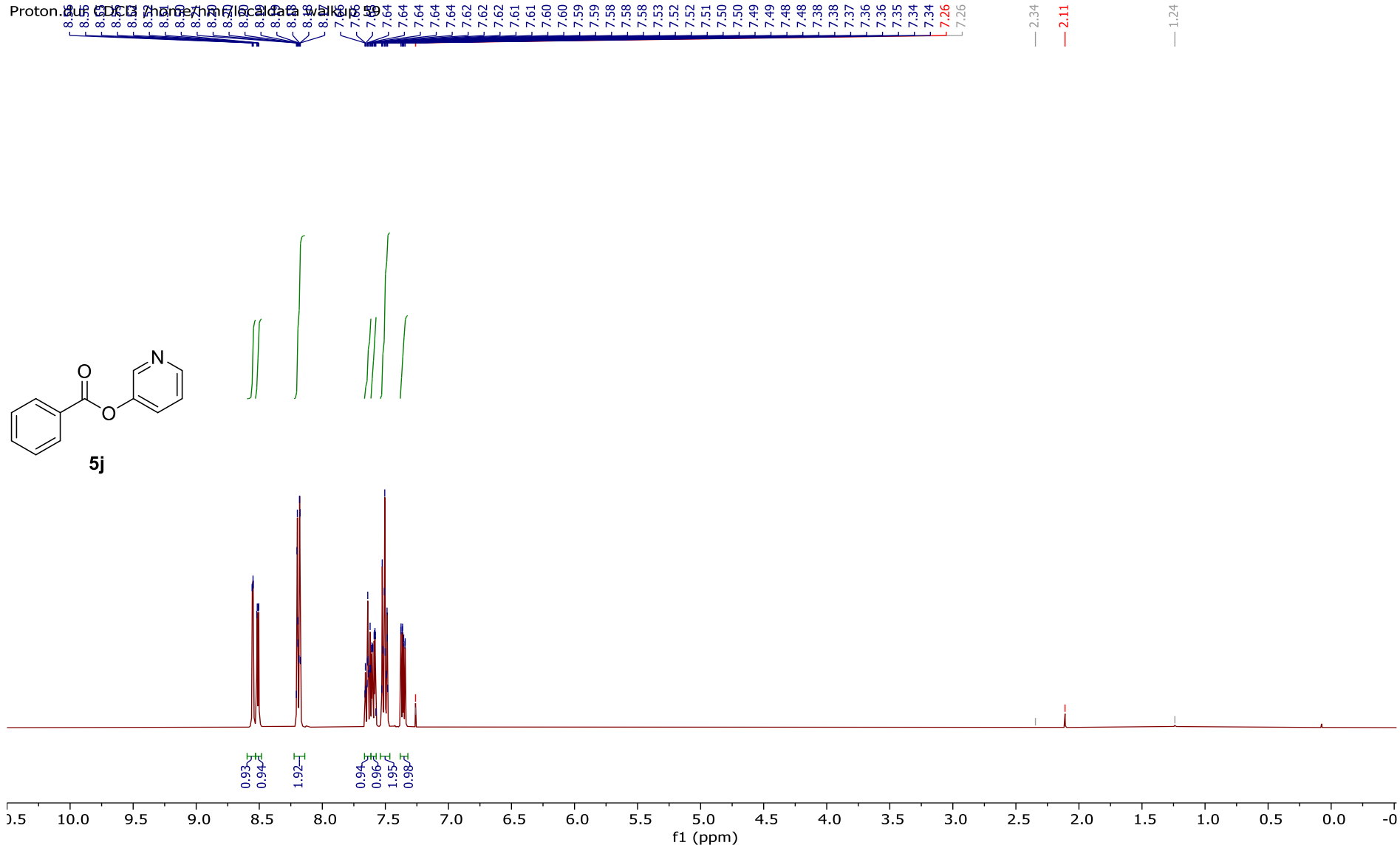
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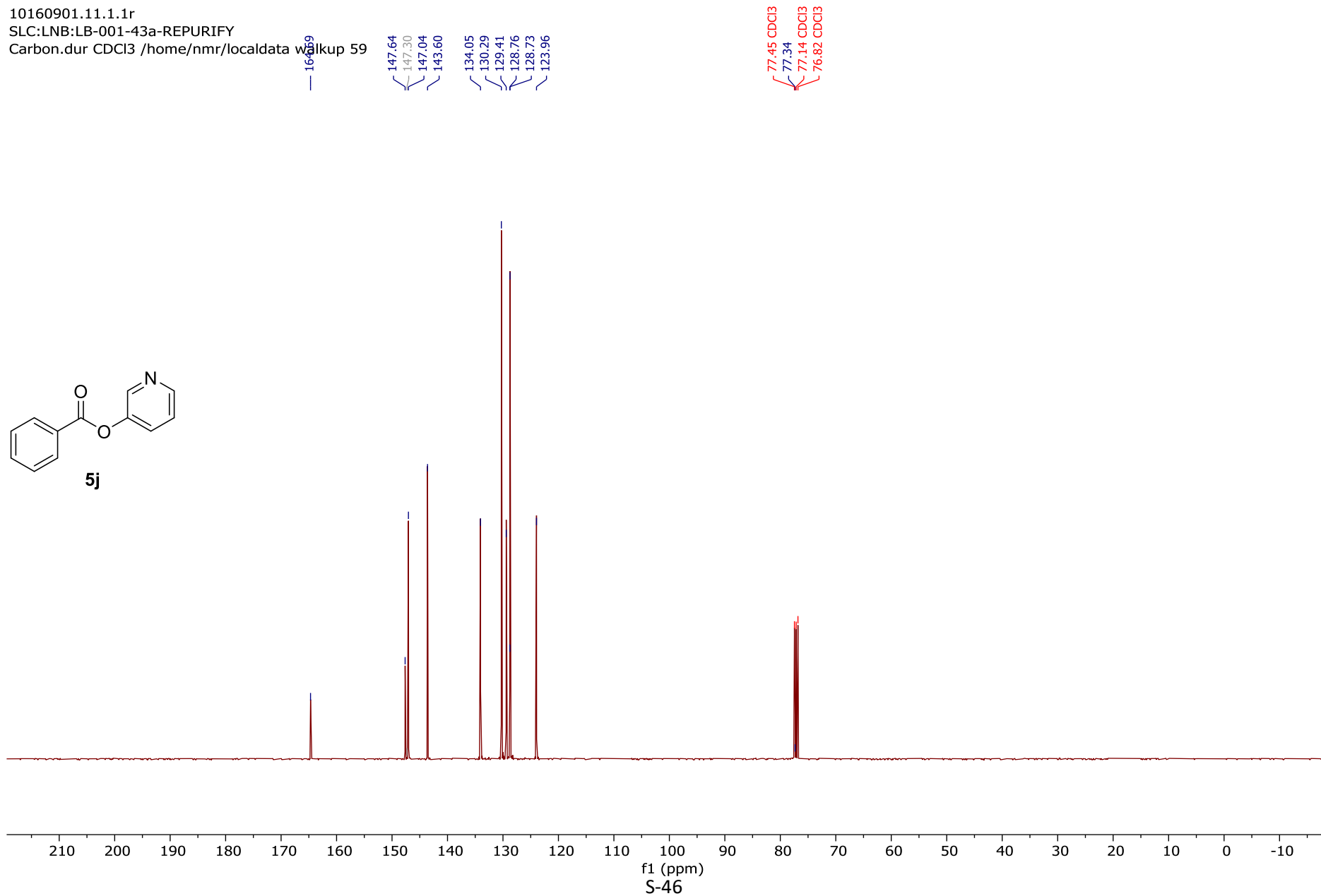
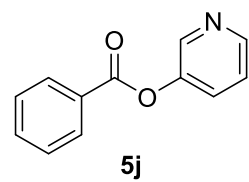
Proton. 2.34 2.11 H2O 1.24



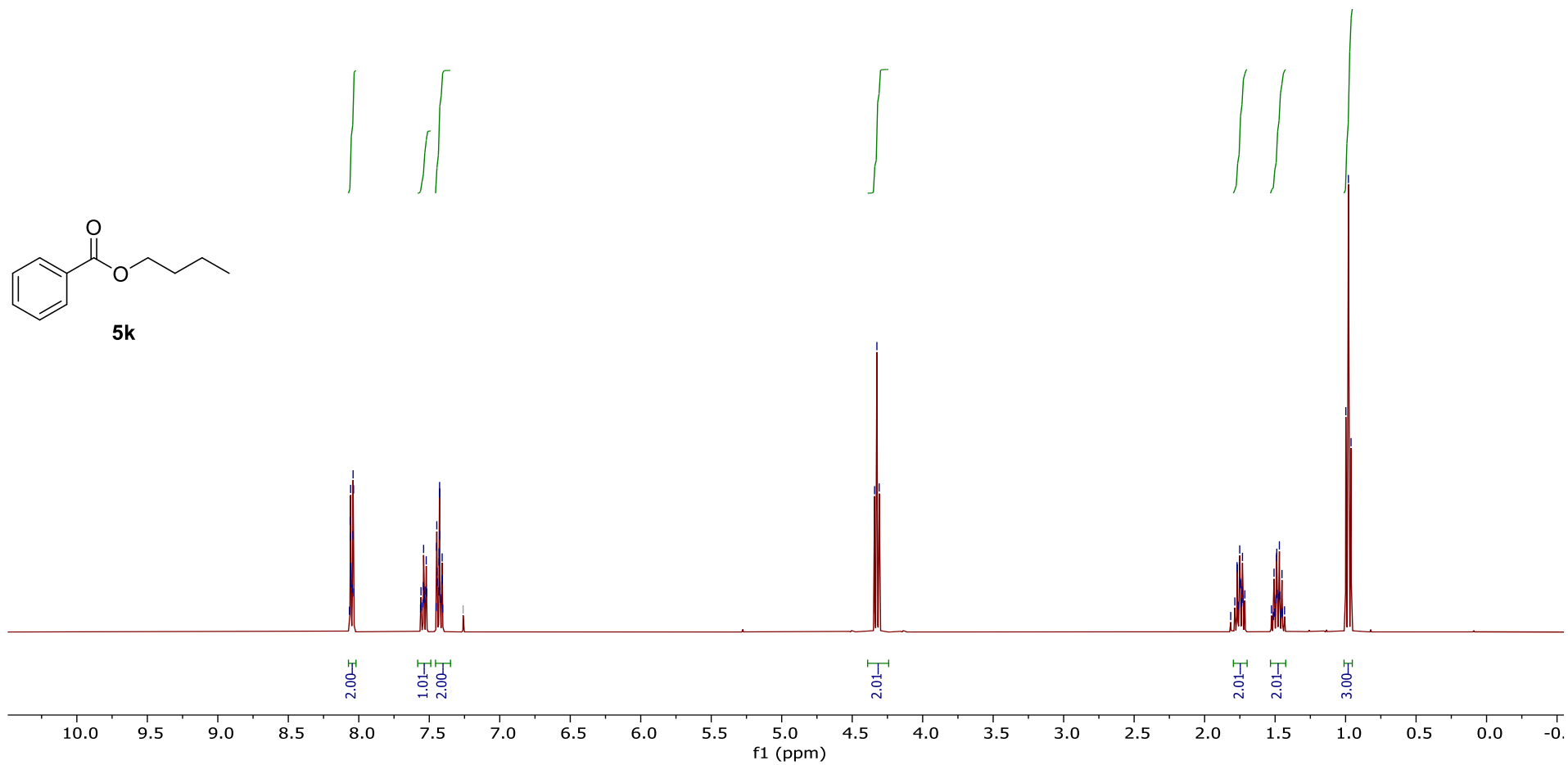
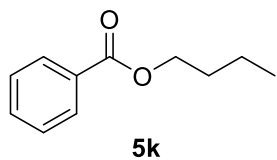
10160901.11.1.1r

SLC:LNB:LB-001-43a-REPURIFY

Carbon.dur CDCl3 /home/nmr/localdata/walkup 59



16170600.10.1.1r
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16170600.11.1.1r
SLC:LNB:LB-001-44C-PURIFY
Carbon.dur CDCl3 /home/nmr/localdata/walkup 25

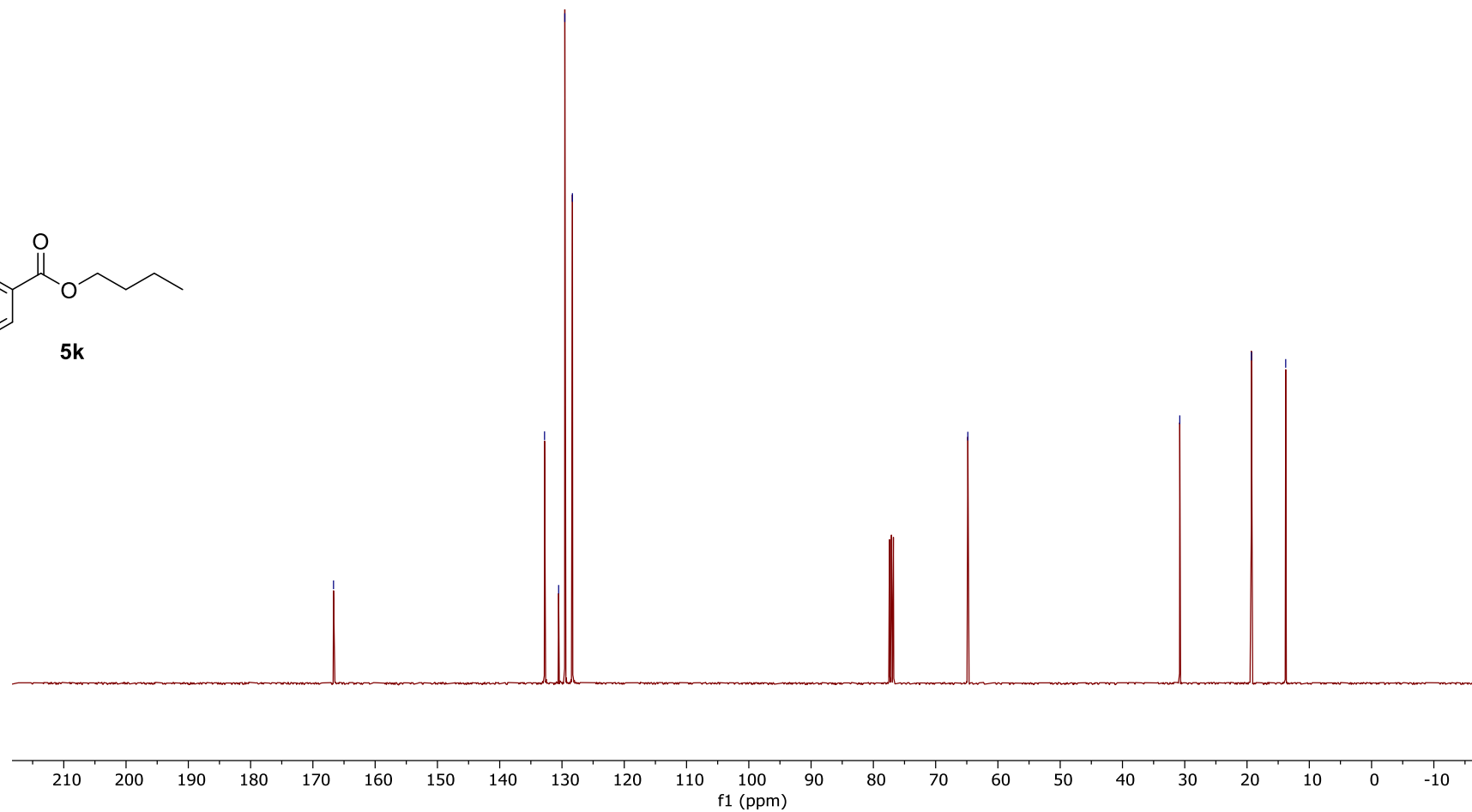
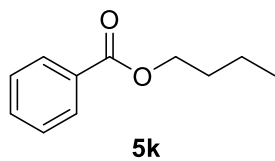
132.80
130.54
129.54
128.32

64.83

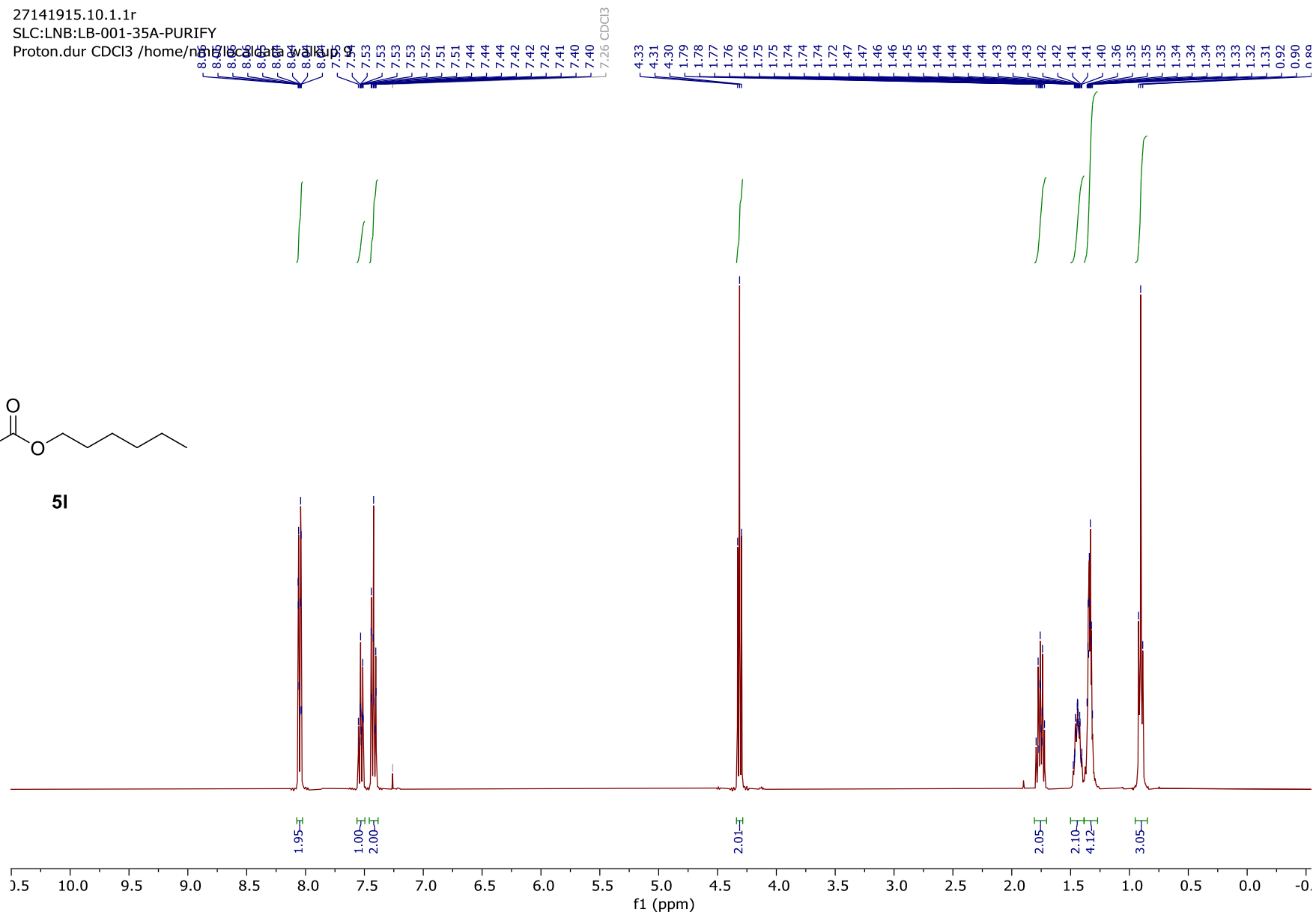
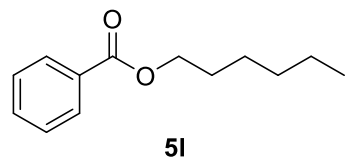
30.80

19.30

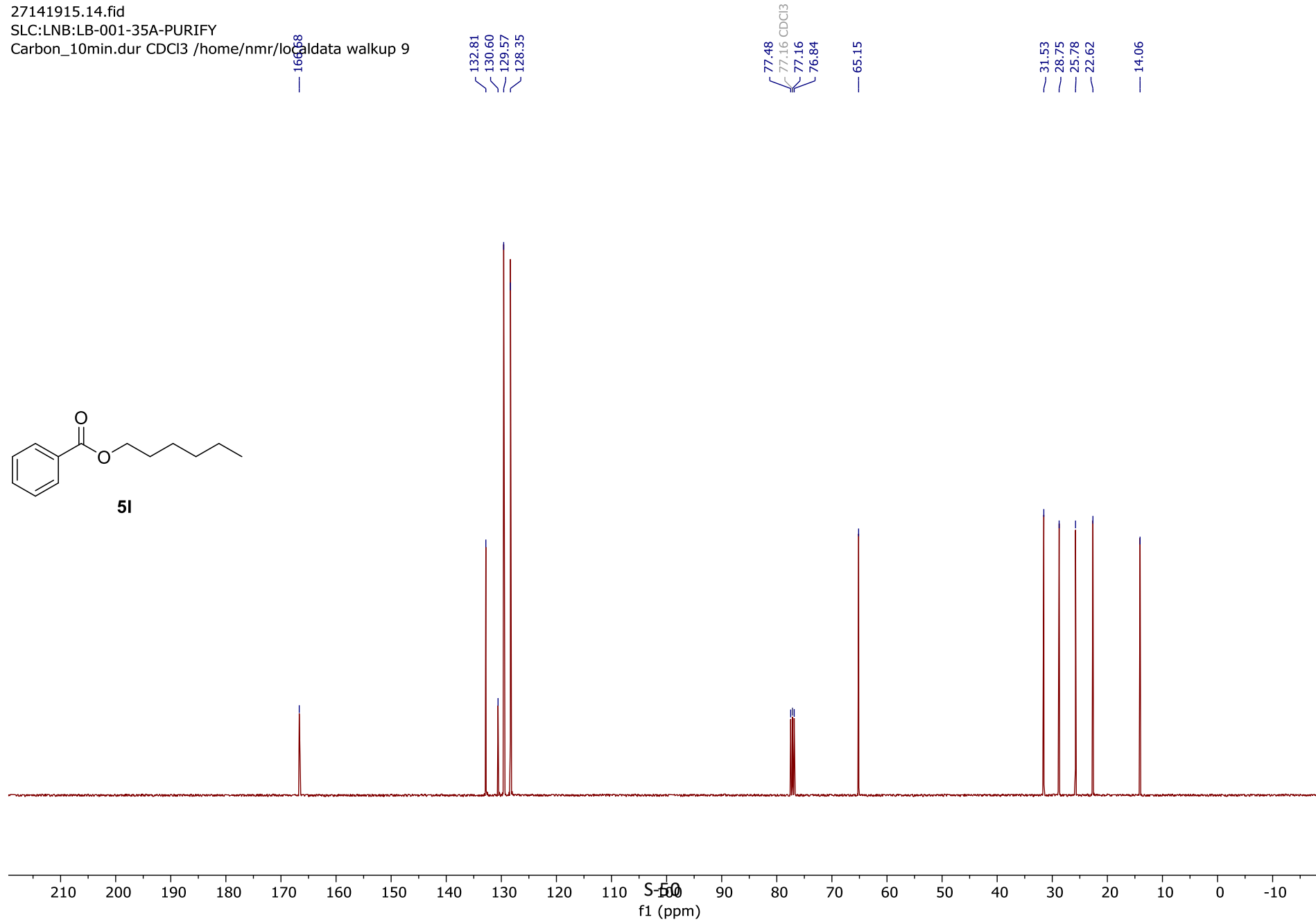
13.79



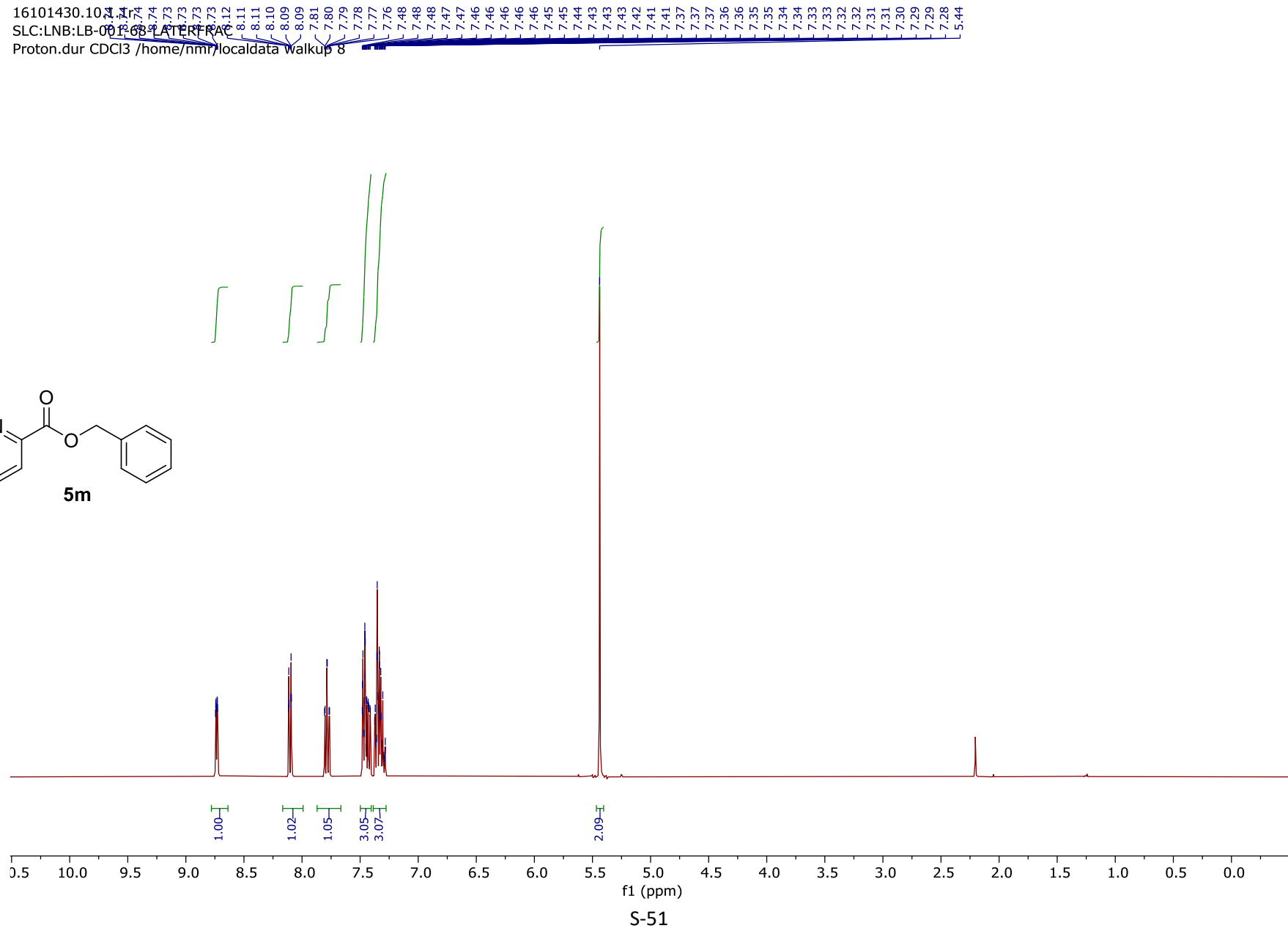
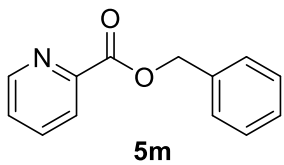
27141915.10.1.1r
SLC:LNB:LB-001-35A-PURIFY
Proton.dur CDCl3 /home/nb/1915/10/1/1r



27141915.14.fid
SLC:LNB:LB-001-35A-PURIFY
Carbon_10min.dur CDCl3 /home/nmr/loc data walkup 9



16101430.10314
SLC:LNB:LB-001-08-LATERFRAC
Proton.dur CDCl3 /home/nmi/localdata/walkup 8



16101430.11.1.1r

SLC:LNB:LB-001-68-LATERFRAC

Carbon_10min.dur CDCl3 /home/nmr/local/data/walk

166.85

146.89

147.92

136.96

135.57

128.56

128.53

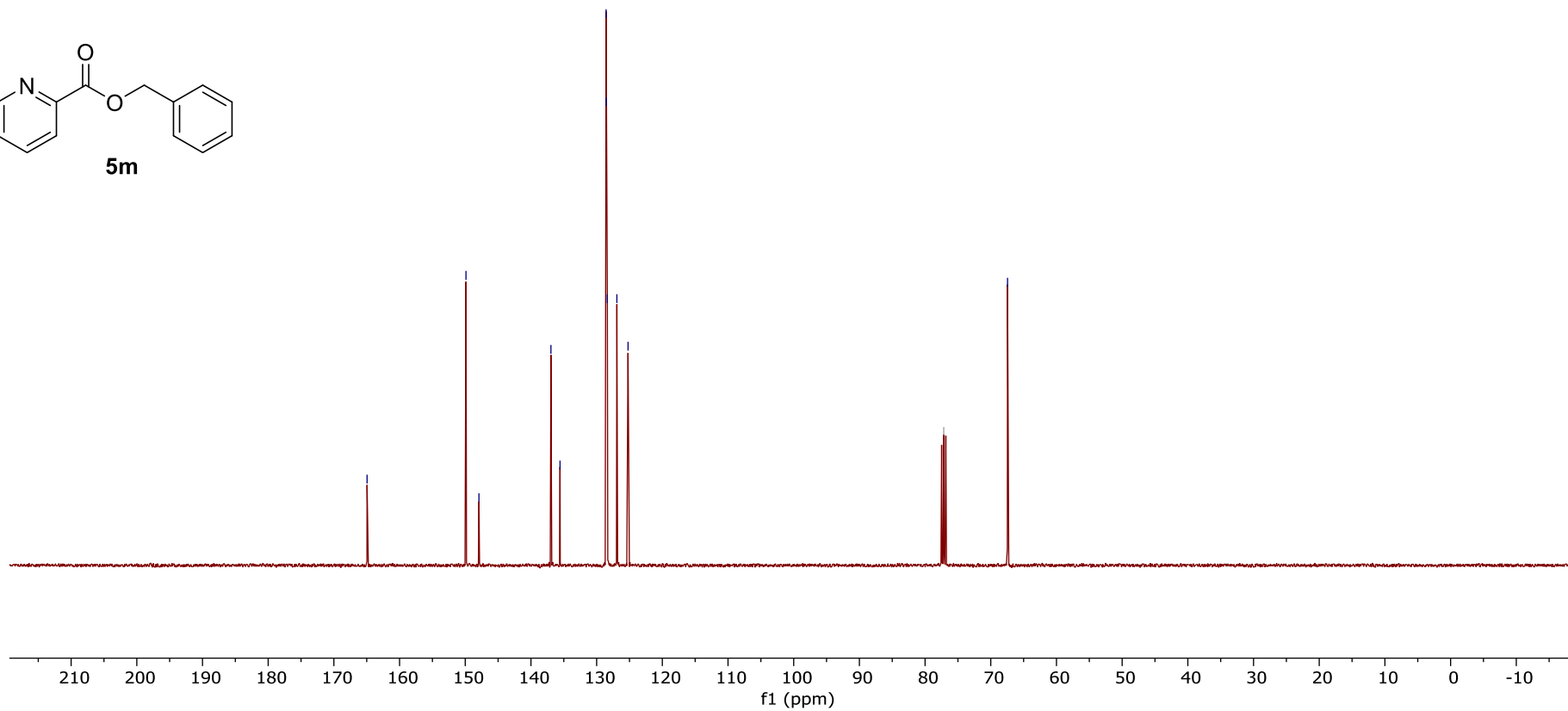
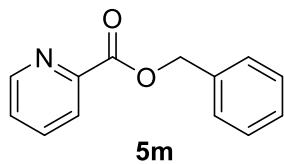
128.37

126.92

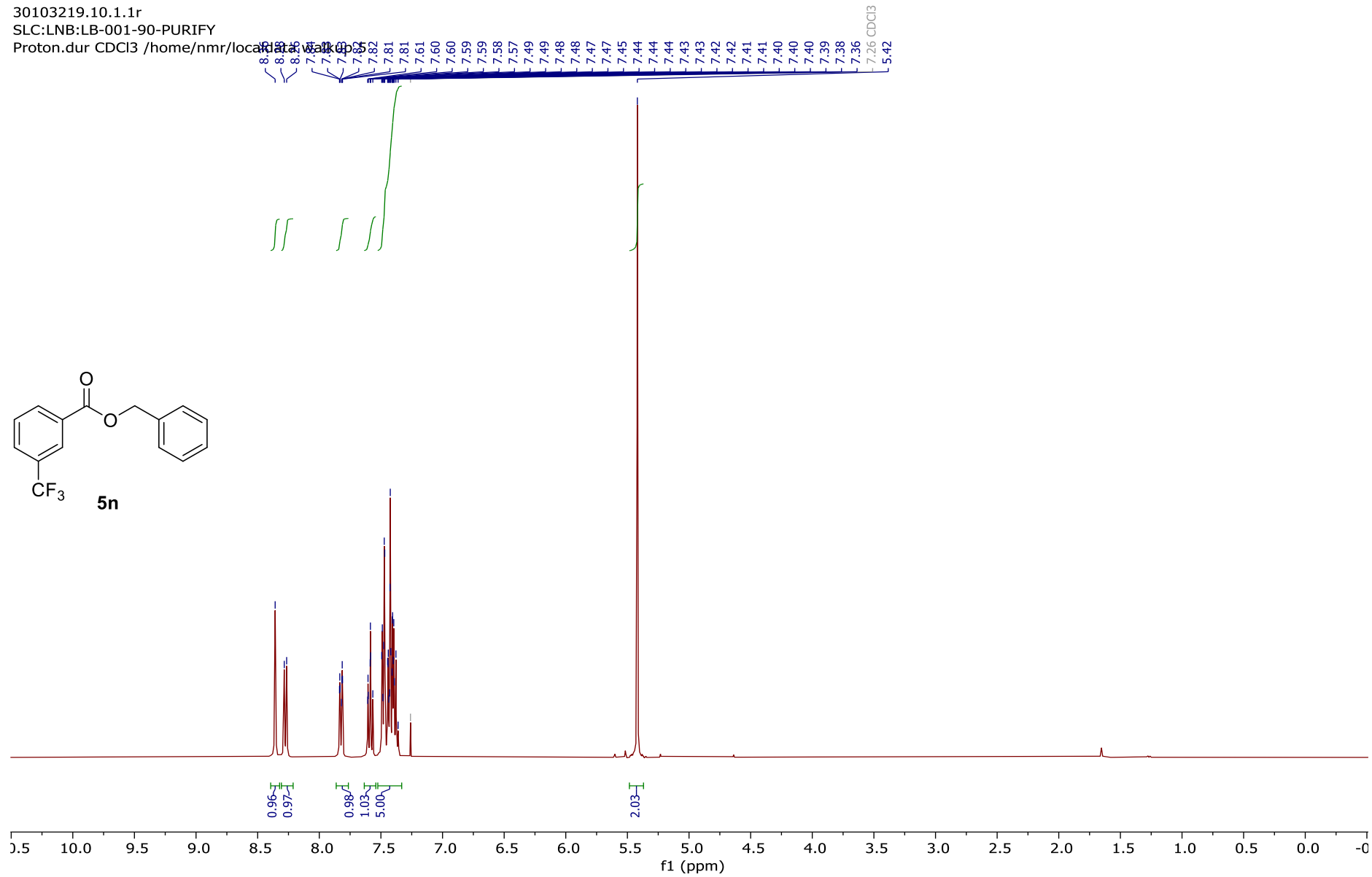
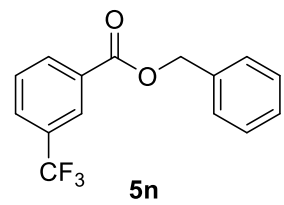
125.22

77.16 CDCl3

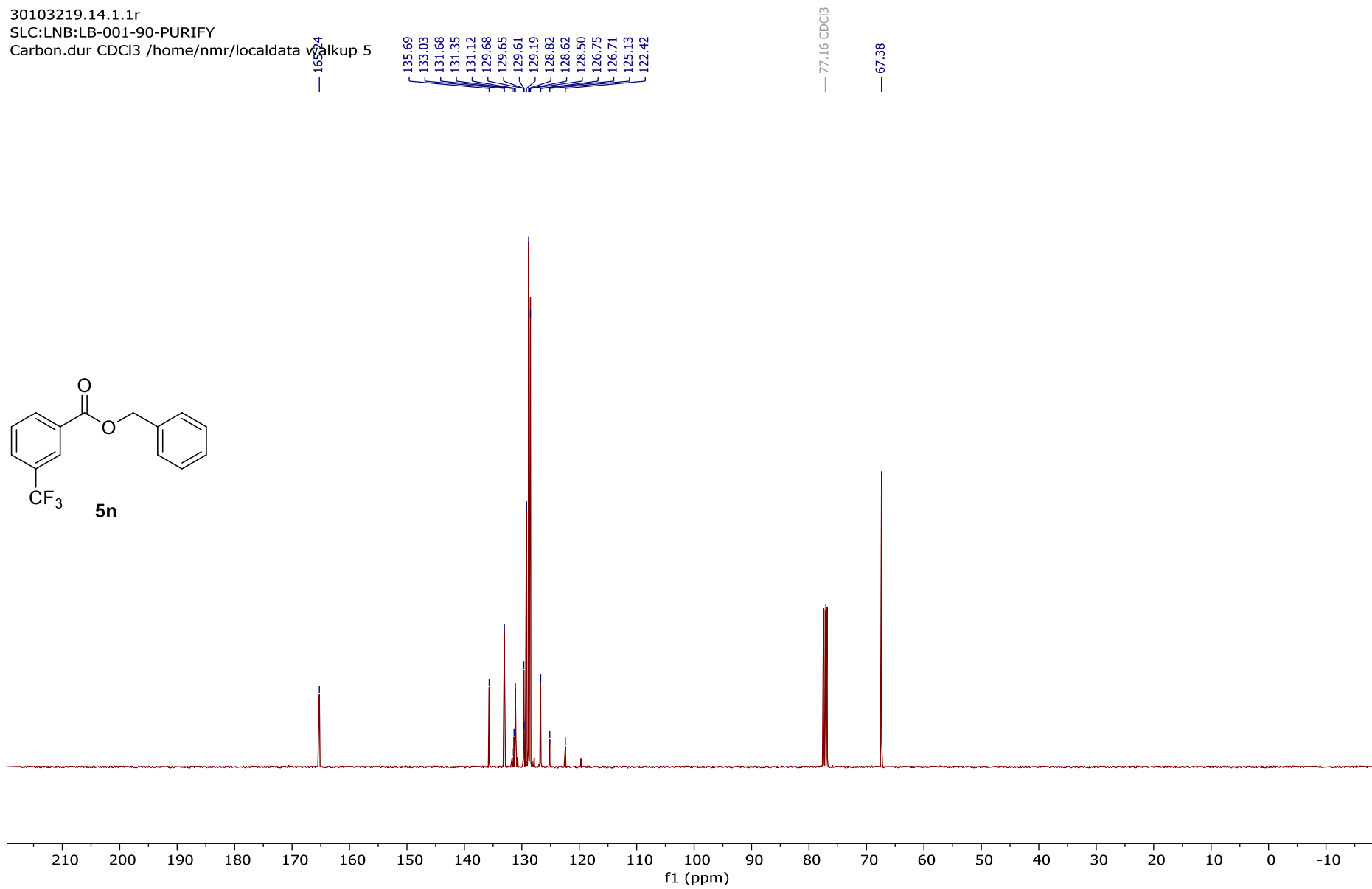
67.45



30103219.10.1.1r
SLC:LNB:LB-001-90-PURIFY
Proton.dur CDCl3 /home/nmr/local

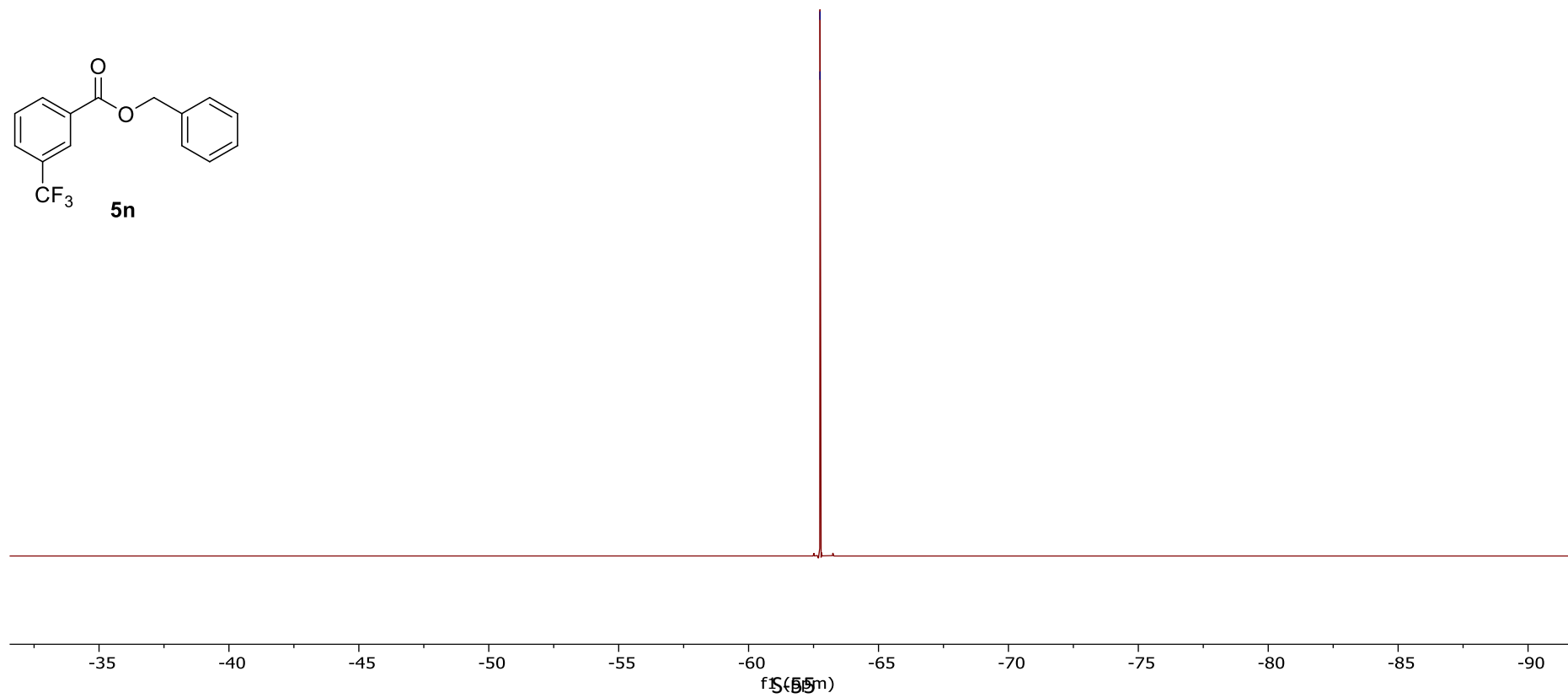
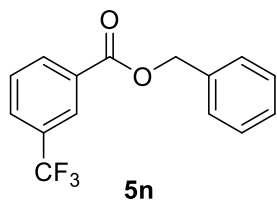


30103219.14.1.1r
SLC:LNB:LB-001-90-PURIFY
Carbon.dur CDCl3 /home/nmr/localdata/walkup 5



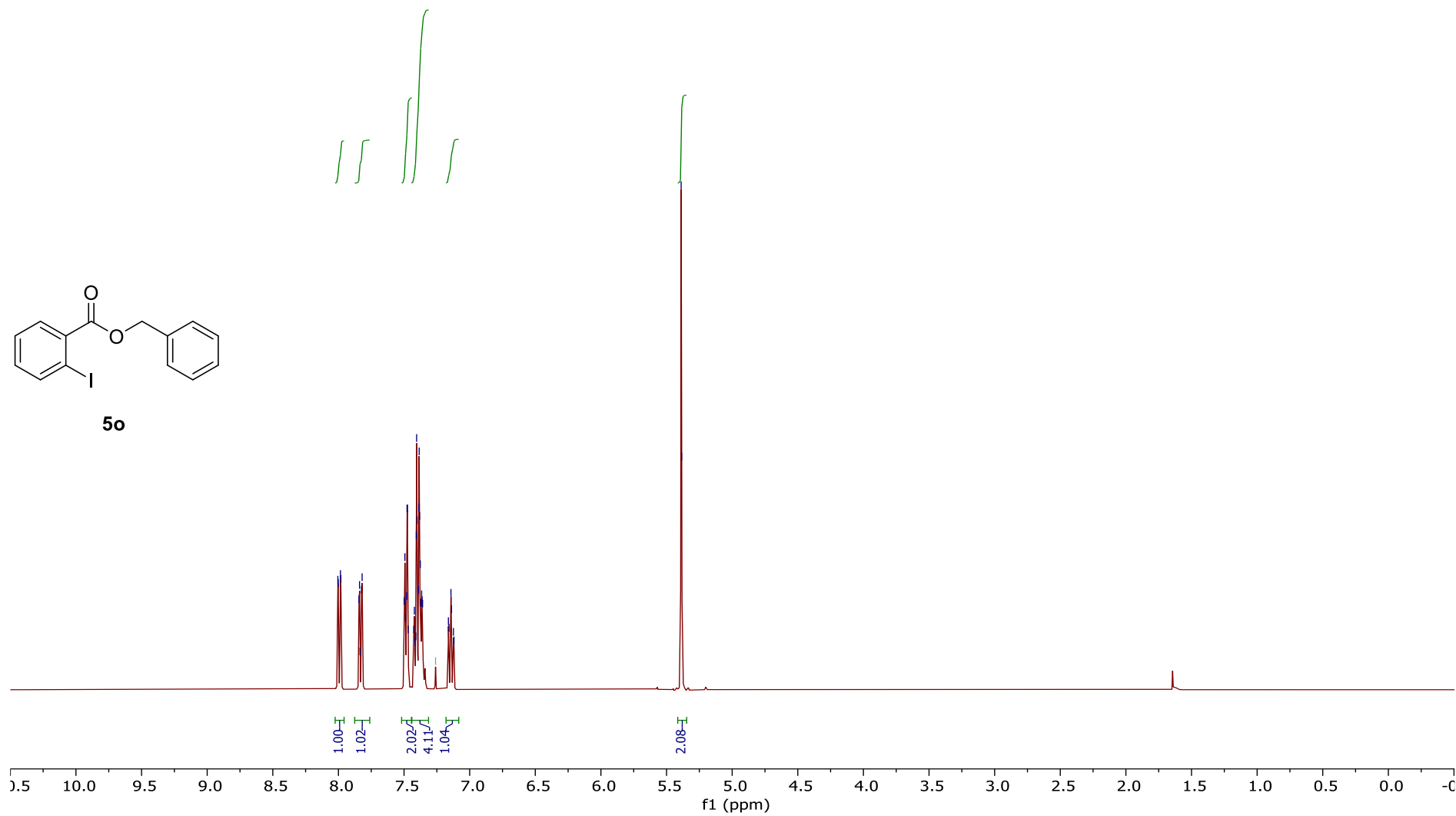
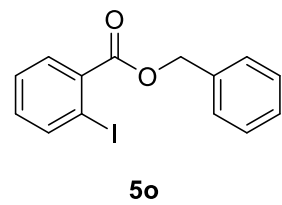
30103219.13.1.1r
SLC:LNB:LB-001-90-PURIFY
F19_limits_dec.dur CDCl3 /home/nmr/localdata walkup 5

-62.75
-62.75

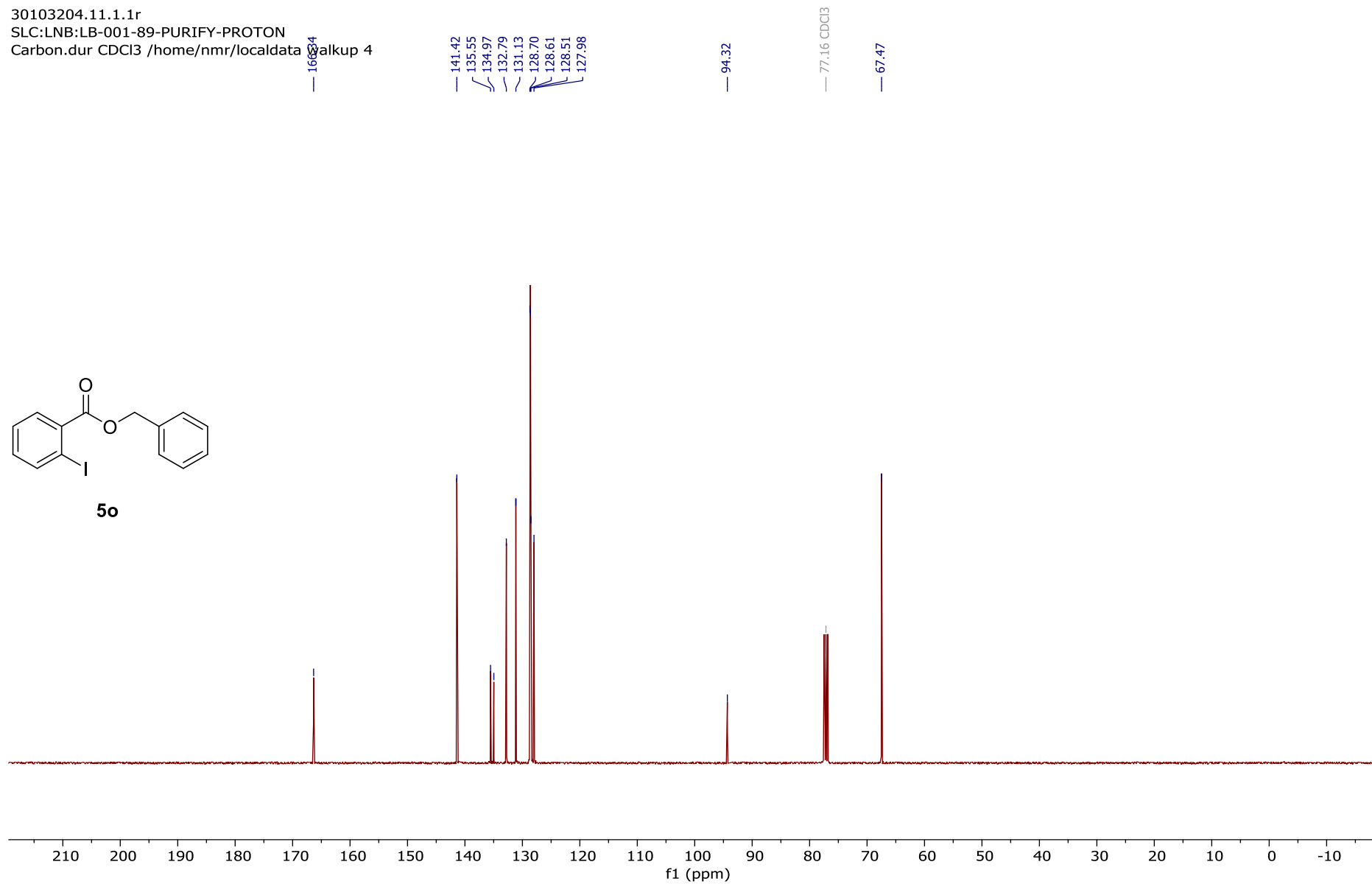
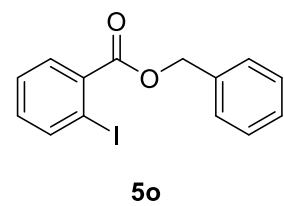


30103204.10.1.1r
SLC:LNB:LB-001-89-PURIFY-PROTON

8.81, 8.79, 8.77, 8.76, 8.75, 8.74, 8.73, 8.72, 8.71, 8.70, 8.69, 8.68, 8.67, 8.66, 8.65, 8.64, 8.63, 8.62, 8.61, 8.60, 8.59, 8.58, 8.57, 8.56, 8.55, 8.54, 8.53, 8.52, 8.51, 8.50, 8.49, 8.48, 8.47, 8.46, 8.45, 8.44, 8.43, 8.42, 8.41, 8.40, 8.39, 8.38, 8.37, 8.36, 8.35, 8.34, 8.33, 8.32, 8.31, 8.30, 8.29, 8.28, 8.27, 8.26, 8.25, 8.24, 8.23, 8.22, 8.21, 8.20, 8.19, 8.18, 8.17, 8.16, 8.15, 8.14, 8.13, 8.12, 8.11, 8.10, 8.09, 8.08, 8.07, 8.06, 8.05, 8.04, 8.03, 8.02, 8.01, 8.00, 7.99, 7.98, 7.97, 7.96, 7.95, 7.94, 7.93, 7.92, 7.91, 7.90, 7.89, 7.88, 7.87, 7.86, 7.85, 7.84, 7.83, 7.82, 7.81, 7.80, 7.79, 7.78, 7.77, 7.76, 7.75, 7.74, 7.73, 7.72, 7.71, 7.70, 7.69, 7.68, 7.67, 7.66, 7.65, 7.64, 7.63, 7.62, 7.61, 7.60, 7.59, 7.58, 7.57, 7.56, 7.55, 7.54, 7.53, 7.52, 7.51, 7.50, 7.49, 7.48, 7.47, 7.46, 7.45, 7.44, 7.43, 7.42, 7.41, 7.40, 7.39, 7.38, 7.37, 7.36, 7.35, 7.34, 7.33, 7.32, 7.31, 7.30, 7.29, 7.28, 7.27, 7.26, 7.25, 7.24, 7.23, 7.22, 7.21, 7.20, 7.19, 7.18, 7.17, 7.16, 7.15, 7.14, 7.13, 7.12, 7.11, 7.10, 7.09, 7.08, 7.07, 7.06, 7.05, 7.04, 7.03, 7.02, 7.01, 7.00, 6.99, 6.98, 6.97, 6.96, 6.95, 6.94, 6.93, 6.92, 6.91, 6.90, 6.89, 6.88, 6.87, 6.86, 6.85, 6.84, 6.83, 6.82, 6.81, 6.80, 6.79, 6.78, 6.77, 6.76, 6.75, 6.74, 6.73, 6.72, 6.71, 6.70, 6.69, 6.68, 6.67, 6.66, 6.65, 6.64, 6.63, 6.62, 6.61, 6.60, 6.59, 6.58, 6.57, 6.56, 6.55, 6.54, 6.53, 6.52, 6.51, 6.50, 6.49, 6.48, 6.47, 6.46, 6.45, 6.44, 6.43, 6.42, 6.41, 6.40, 6.39, 6.38, 6.37, 6.36, 6.35, 6.34, 6.33, 6.32, 6.31, 6.30, 6.29, 6.28, 6.27, 6.26, 6.25, 6.24, 6.23, 6.22, 6.21, 6.20, 6.19, 6.18, 6.17, 6.16, 6.15, 6.14, 6.13, 6.12, 6.11, 6.10, 6.09, 6.08, 6.07, 6.06, 6.05, 6.04, 6.03, 6.02, 6.01, 6.00, 5.99, 5.98, 5.97, 5.96, 5.95, 5.94, 5.93, 5.92, 5.91, 5.90, 5.89, 5.88, 5.87, 5.86, 5.85, 5.84, 5.83, 5.82, 5.81, 5.80, 5.79, 5.78, 5.77, 5.76, 5.75, 5.74, 5.73, 5.72, 5.71, 5.70, 5.69, 5.68, 5.67, 5.66, 5.65, 5.64, 5.63, 5.62, 5.61, 5.60, 5.59, 5.58, 5.57, 5.56, 5.55, 5.54, 5.53, 5.52, 5.51, 5.50, 5.49, 5.48, 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3.81, 3.80, 3.79, 3.78, 3.77, 3.76, 3.75, 3.74, 3.73, 3.72, 3.71, 3.70, 3.69, 3.68, 3.67, 3.66, 3.65, 3.64, 3.63, 3.62, 3.61, 3.60, 3.59, 3.58, 3.57, 3.56, 3.55, 3.54, 3.53, 3.52, 3.51, 3.50, 3.49, 3.48, 3.47, 3.46, 3.45, 3.44, 3.43, 3.42, 3.41, 3.40, 3.39, 3.38, 3.37, 3.36, 3.35, 3.34, 3.33, 3.32, 3.31, 3.30, 3.29, 3.28, 3.27, 3.26, 3.25, 3.24, 3.23, 3.22, 3.21, 3.20, 3.19, 3.18, 3.17, 3.16, 3.15, 3.14, 3.13, 3.12, 3.11, 3.10, 3.09, 3.08, 3.07, 3.06, 3.05, 3.04, 3.03, 3.02, 3.01, 3.00, 2.99, 2.98, 2.97, 2.96, 2.95, 2.94, 2.93, 2.92, 2.91, 2.90, 2.89, 2.88, 2.87, 2.86, 2.85, 2.84, 2.83, 2.82, 2.81, 2.80, 2.79, 2.78, 2.77, 2.76, 2.75, 2.74, 2.73, 2.72, 2.71, 2.70, 2.69, 2.68, 2.67, 2.66, 2.65, 2.64, 2.63, 2.62, 2.61, 2.60, 2.59, 2.58, 2.57, 2.56, 2.55, 2.54, 2.53, 2.52, 2.51, 2.50, 2.49, 2.48, 2.47, 2.46, 2.45, 2.44, 2.43, 2.42, 2.41, 2.40, 2.39, 2.38, 2.37, 2.36, 2.35, 2.34, 2.33, 2.32, 2.31, 2.30, 2.29, 2.28, 2.27, 2.26, 2.25, 2.24, 2.23, 2.22, 2.21, 2.20, 2.19, 2.18, 2.17, 2.16, 2.15, 2.14, 2.13, 2.12, 2.11, 2.10, 2.09, 2.08, 2.07, 2.06, 2.05, 2.04, 2.03, 2.02, 2.01, 2.00, 1.99, 1.98, 1.97, 1.96, 1.95, 1.94, 1.93, 1.92, 1.91, 1.90, 1.89, 1.88, 1.87, 1.86, 1.85, 1.84, 1.83, 1.82, 1.81, 1.80, 1.79, 1.78, 1.77, 1.76, 1.75, 1.74, 1.73, 1.72, 1.71, 1.70, 1.69, 1.68, 1.67, 1.66, 1.65, 1.64, 1.63, 1.62, 1.61, 1.60, 1.59, 1.58, 1.57, 1.56, 1.55, 1.54, 1.53, 1.52, 1.51, 1.50, 1.49, 1.48, 1.47, 1.46, 1.45, 1.44, 1.43, 1.42, 1.41, 1.40, 1.39, 1.38, 1.37, 1.36, 1.35, 1.34, 1.33, 1.32, 1.31, 1.30, 1.29, 1.28, 1.27, 1.26, 1.25, 1.24, 1.23, 1.22, 1.21, 1.20, 1.19, 1.18, 1.17, 1.16, 1.15, 1.14, 1.13, 1.12, 1.11, 1.10, 1.09, 1.08, 1.07, 1.06, 1.05, 1.04, 1.03, 1.02, 1.01, 1.00, 0.99, 0.98, 0.97, 0.96, 0.95, 0.94, 0.93, 0.92, 0.91, 0.90, 0.89, 0.88, 0.87, 0.86, 0.85, 0.84, 0.83, 0.82, 0.81, 0.80, 0.79, 0.78, 0.77, 0.76, 0.75, 0.74, 0.73, 0.72, 0.71, 0.70, 0.69, 0.68, 0.67, 0.66, 0.65, 0.64, 0.63, 0.62, 0.61, 0.60, 0.59, 0.58, 0.57, 0.56, 0.55, 0.54, 0.53, 0.52, 0.51, 0.50, 0.49, 0.48, 0.47, 0.46, 0.45, 0.44, 0.43, 0.42, 0.41, 0.40, 0.39, 0.38, 0.37, 0.36, 0.35, 0.34, 0.33, 0.32, 0.31, 0.30, 0.29, 0.28, 0.27, 0.26, 0.25, 0.24, 0.23, 0.22, 0.21, 0.20, 0.19, 0.18, 0.17, 0.16, 0.15, 0.14, 0.13, 0.12, 0.11, 0.10, 0.09, 0.08, 0.07, 0.06, 0.05, 0.04, 0.03, 0.02, 0.01, 0.00, -0.01, -0.02, -0.03, -0.04, -0.05, -0.06, -0.07, -0.08, -0.09, -0.10



30103204.11.1.1r
SLC:LNB:LB-001-89-PURIFY-PROTON
Carbon.dur CDCl3 /home/nmr/localdata/alkup 4

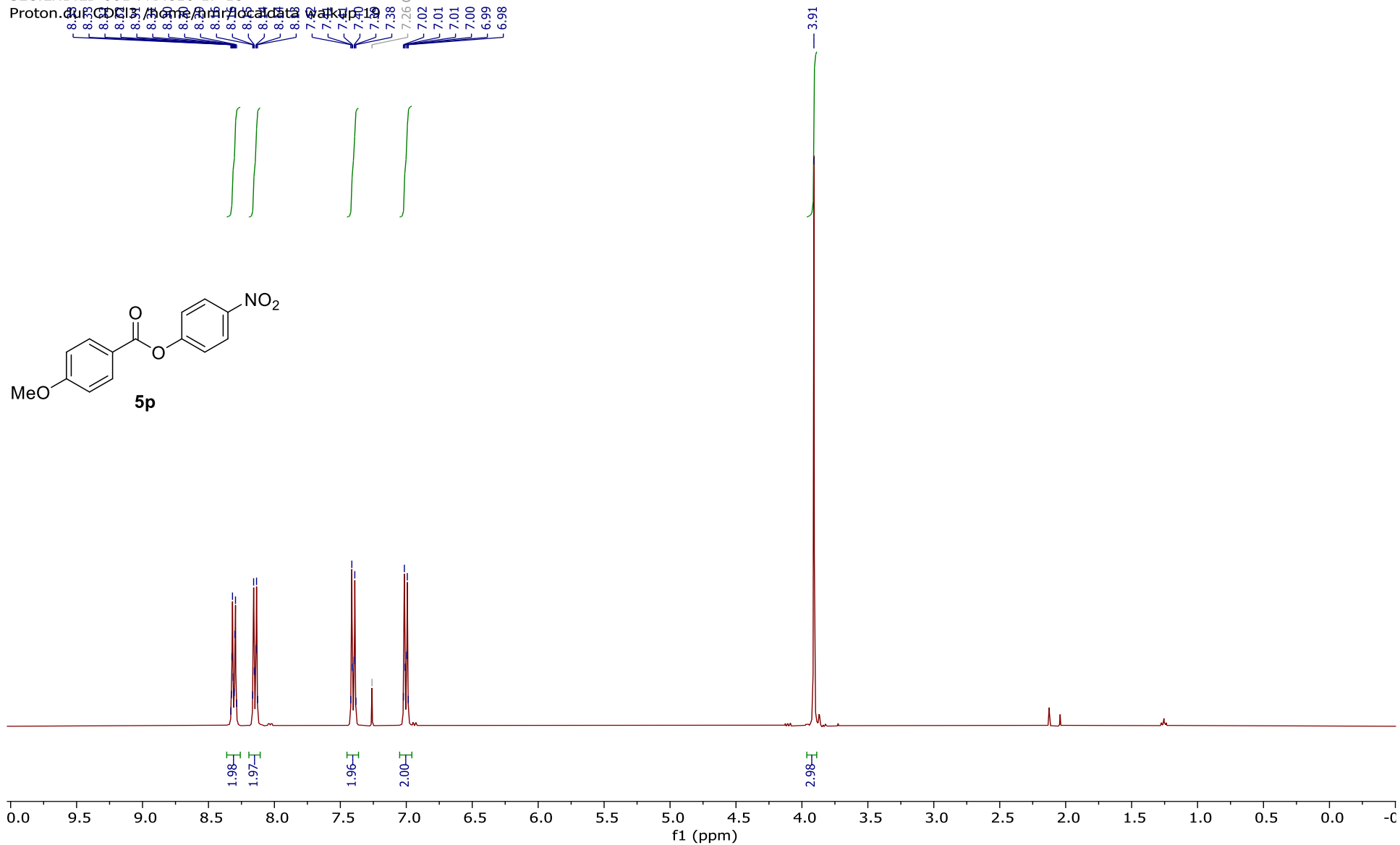
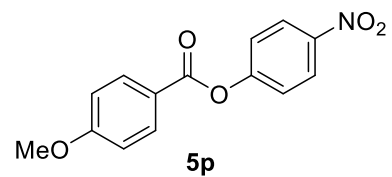


09163850.10.1.1r

SLC:LNB:LB-001-FRAC16-17-18

Proton. 1H NMR (400 MHz, CDCl3) of 5p

8.28, 8.27, 8.26, 8.25, 8.24, 8.23, 8.22, 8.21, 8.20, 8.19, 8.18, 8.17, 8.16, 8.15, 8.14, 8.13, 8.12, 8.11, 8.10, 8.09, 8.08, 8.07, 8.06, 8.05, 8.04, 8.03, 8.02, 8.01, 8.00, 7.99, 7.98, 7.97, 7.96, 7.95, 7.94, 7.93, 7.92, 7.91, 7.90, 7.89, 7.88, 7.87, 7.86, 7.85, 7.84, 7.83, 7.82, 7.81, 7.80, 7.79, 7.78, 7.77, 7.76, 7.75, 7.74, 7.73, 7.72, 7.71, 7.70, 7.69, 7.68, 7.67, 7.66, 7.65, 7.64, 7.63, 7.62, 7.61, 7.60, 7.59, 7.58, 7.57, 7.56, 7.55, 7.54, 7.53, 7.52, 7.51, 7.50, 7.49, 7.48, 7.47, 7.46, 7.45, 7.44, 7.43, 7.42, 7.41, 7.40, 7.39, 7.38, 7.37, 7.36, 7.35, 7.34, 7.33, 7.32, 7.31, 7.30, 7.29, 7.28, 7.27, 7.26, 7.25, 7.24, 7.23, 7.22, 7.21, 7.20, 7.19, 7.18, 7.17, 7.16, 7.15, 7.14, 7.13, 7.12, 7.11, 7.10, 7.09, 7.08, 7.07, 7.06, 7.05, 7.04, 7.03, 7.02, 7.01, 7.00, 6.99, 6.98



S-58

09163850.14.1.1r
SLC:LNB:LB-001-FRAC16-17-18
Carbon.dur CDCl3 /home/nmr/localdata

166.33
166.34
156.15

145.37

132.65

125.34

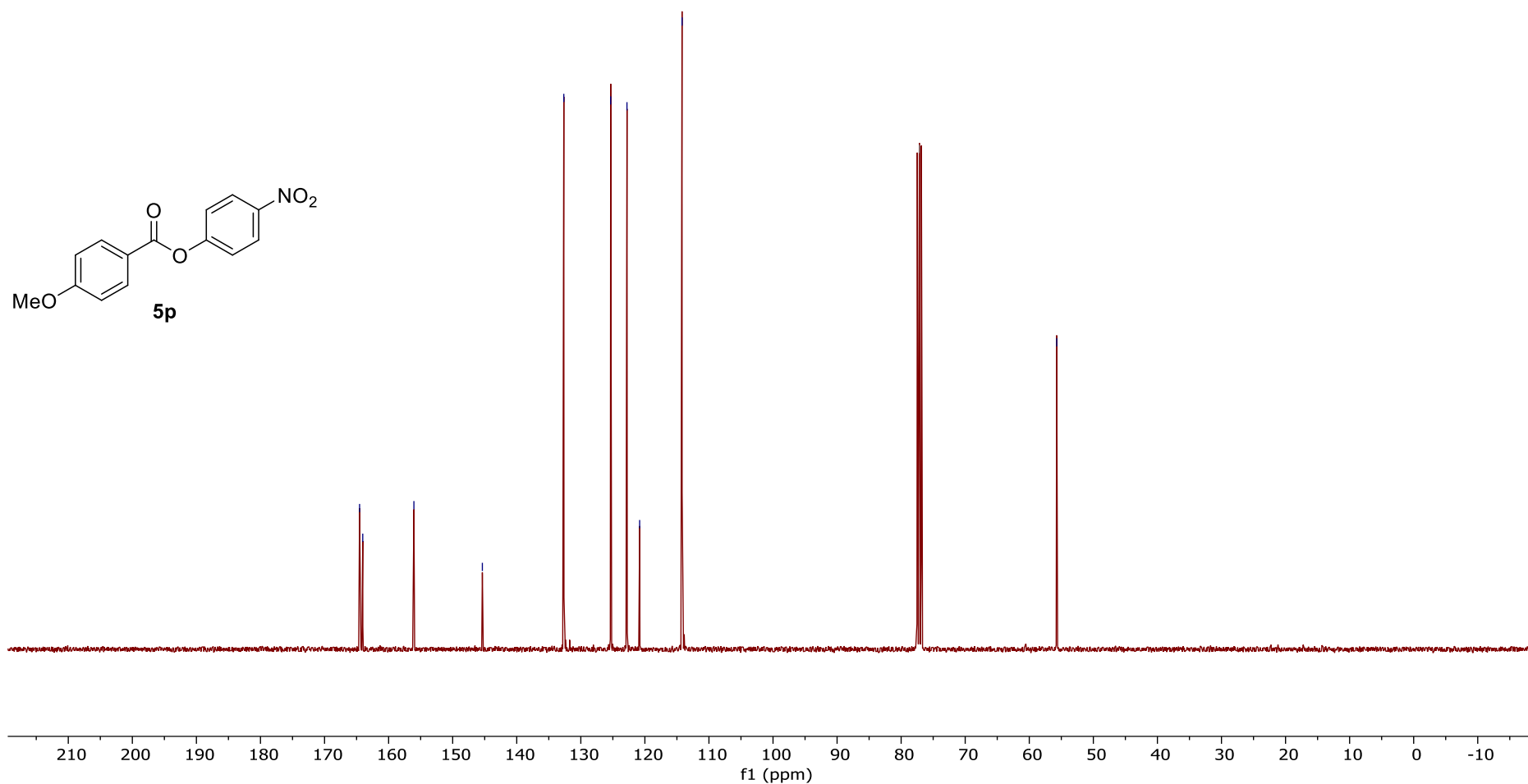
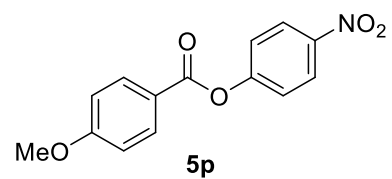
122.79

120.82

114.20

77.16 CDCl3

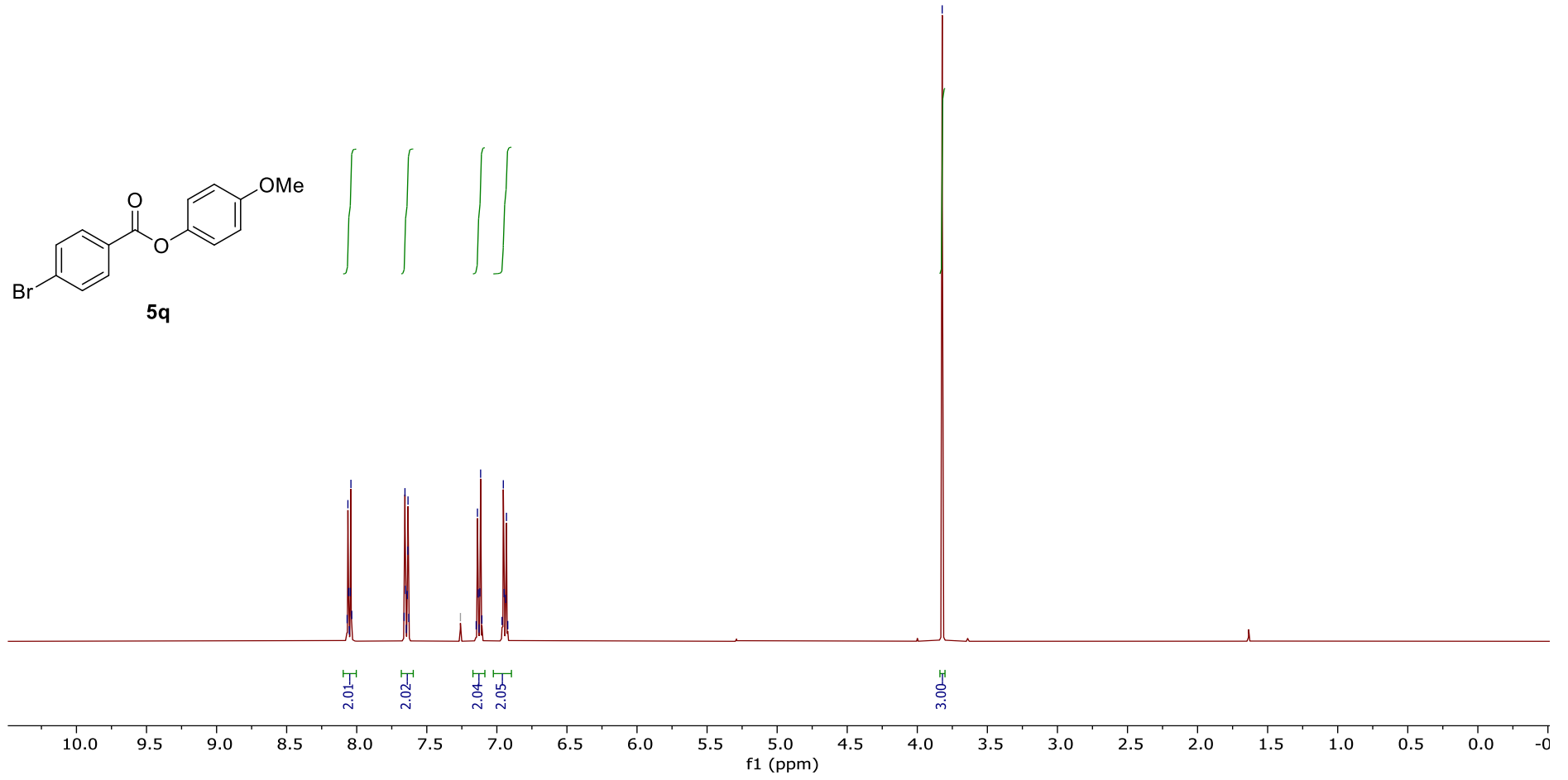
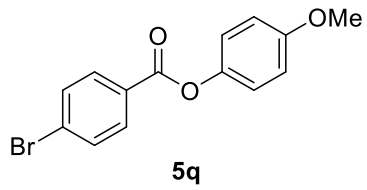
55.72



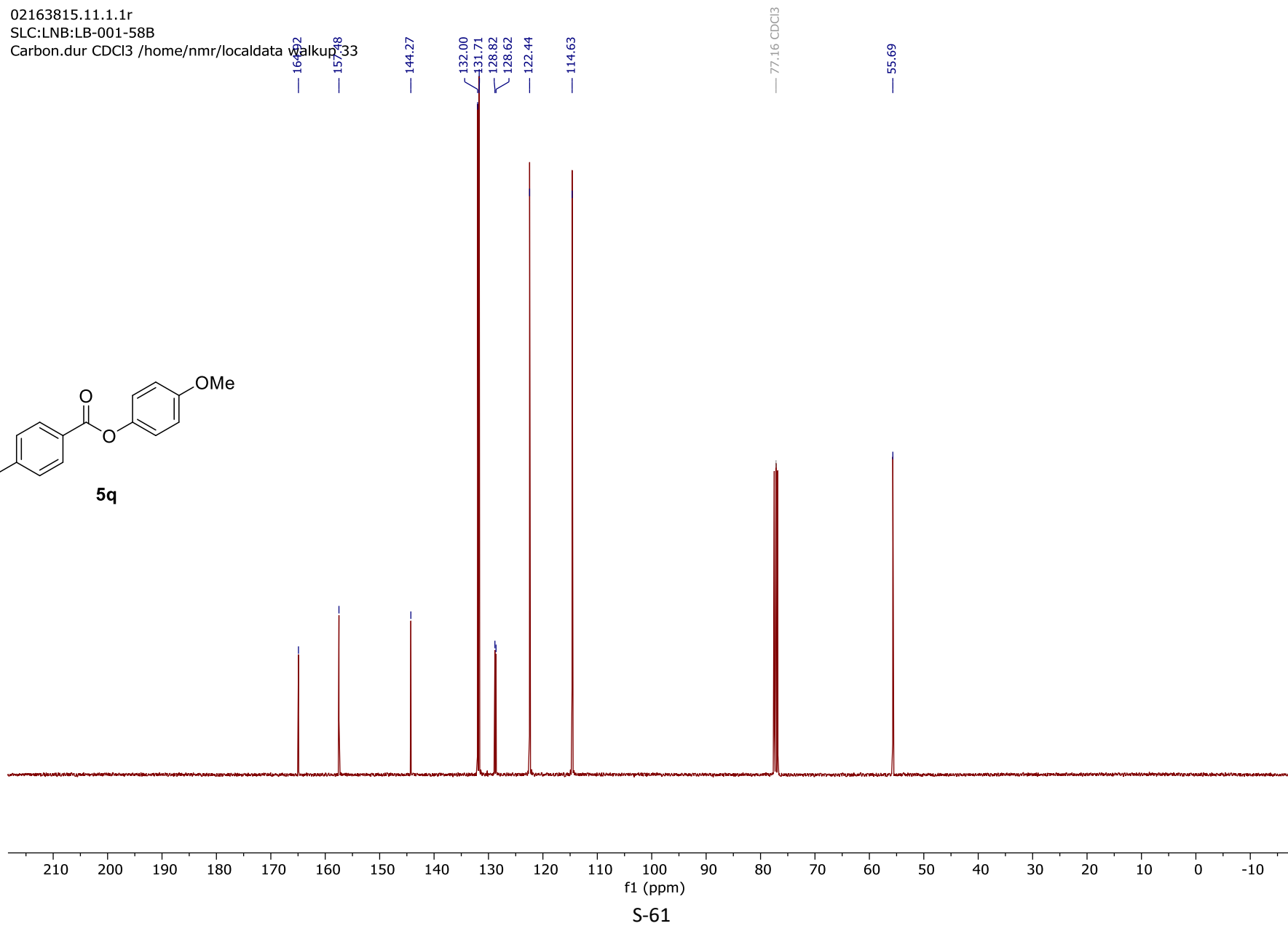
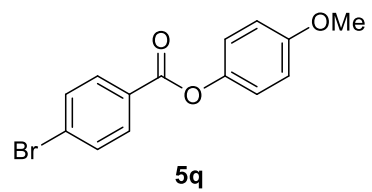
02163815.10.1.1r
SLC:LNB:LB-001-58B
Proton1.icon CDCl3

Typo- Should read 48B

8.07, 8.06, 8.05, 8.04, 8.03, 8.02, 8.01, 7.76, 7.75, 7.74, 7.73, 7.72, 7.71, 7.70, 7.69, 7.68, 7.67, 7.66, 7.65, 7.64, 7.63, 7.62, 7.61, 7.60, 7.59, 7.58, 7.57, 7.56, 7.55, 7.54, 7.53, 7.52, 7.51, 7.50, 7.49, 7.48, 7.47, 7.46, 7.45, 7.44, 7.43, 7.42, 7.41, 7.40, 7.39, 7.38, 7.37, 7.36, 7.35, 7.34, 7.33, 7.32, 7.31, 7.30, 7.29, 7.28, 7.27, 7.26, 7.25, 7.24, 7.23, 7.22, 7.21, 7.20, 7.19, 7.18, 7.17, 7.16, 7.15, 7.14, 7.13, 7.12, 7.11, 7.10, 7.09, 7.08, 7.07, 7.06, 7.05, 7.04, 7.03, 7.02, 7.01, 7.00, 6.99, 6.98, 6.97, 6.96, 6.95, 6.94, 6.93, 6.92, 3.82

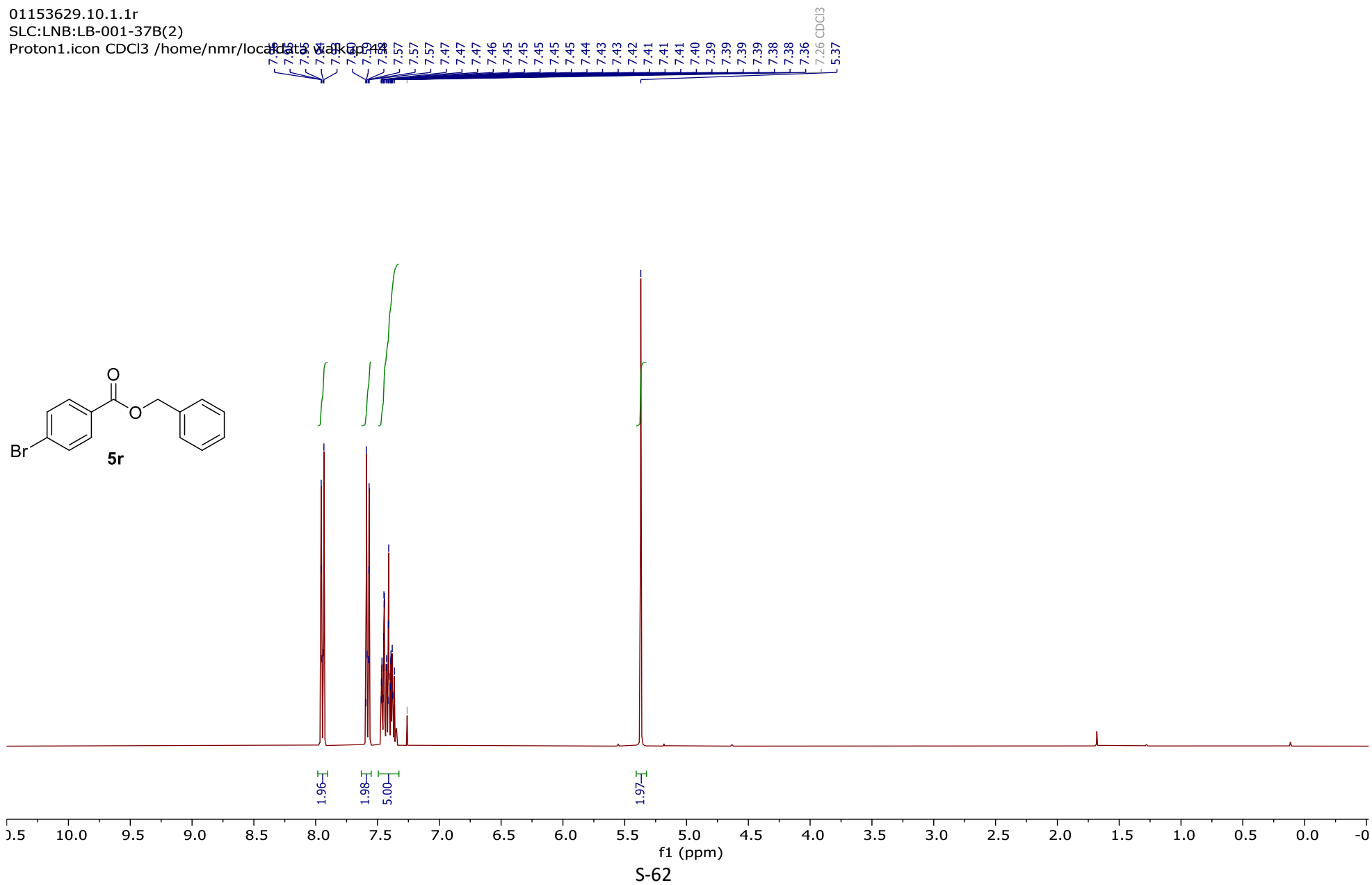


02163815.11.1.1r
SLC:LNB:LB-001-58B
Carbon.dur CDCI3 /home/nmr/localdata/backup

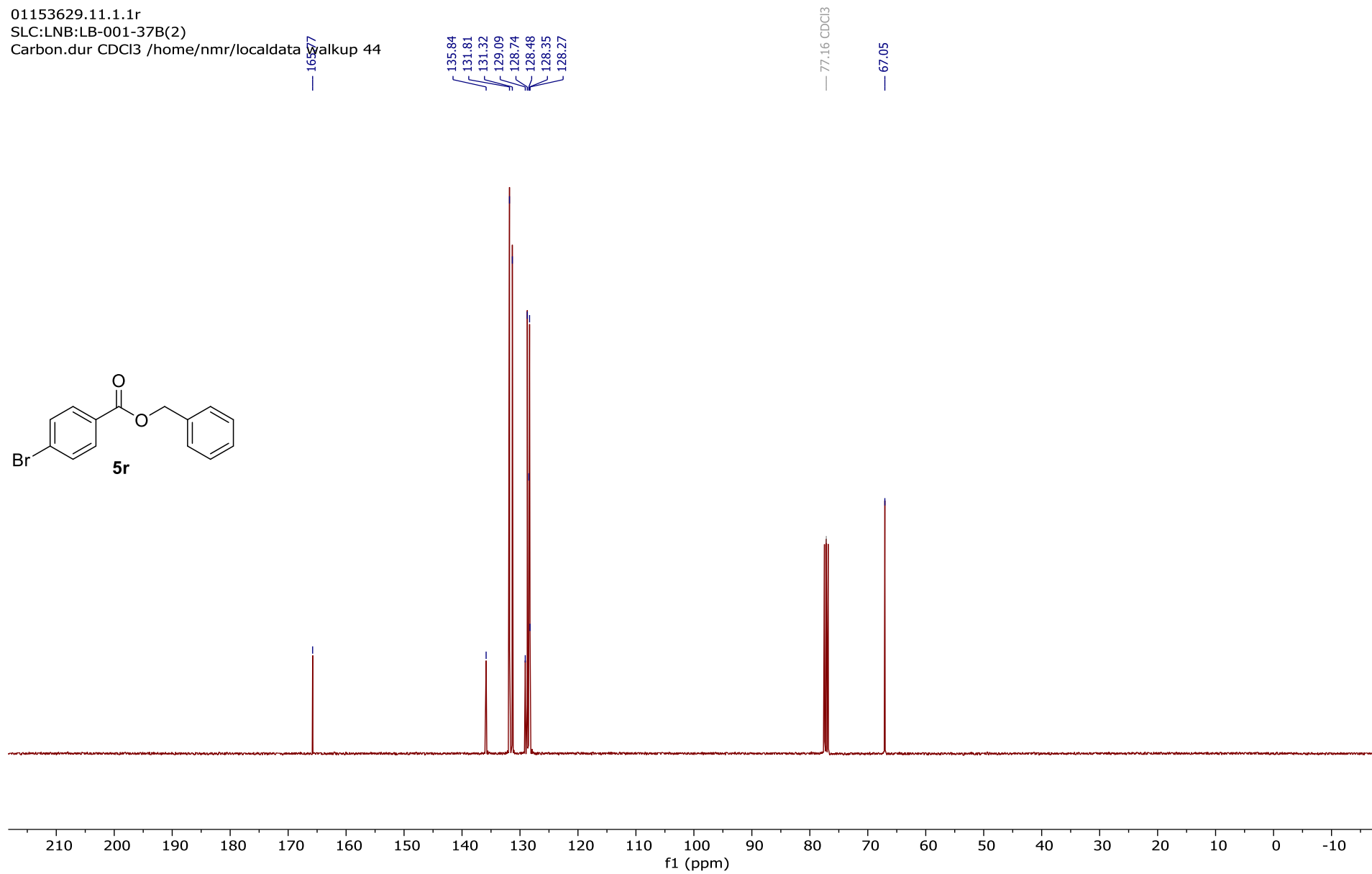
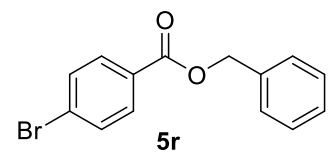


01153629.10.1.1r
SLC:LNB:LB-001-37B(2)

Proton1.icon CDCl3 /home/nmr/local/data/

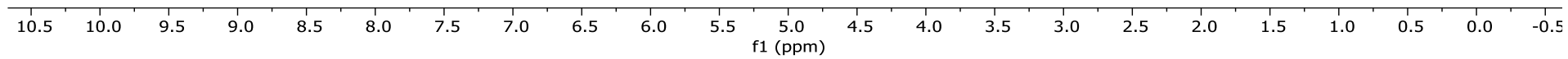
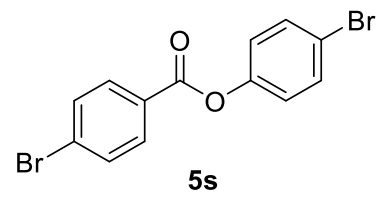


01153629.11.1.1r
SLC:LNB:LB-001-37B(2)
Carbon.dur CDCl3 /home/nmr/localdata/alkup 44



09163920.10.1.1r
SLC:LNB:LB-001-FRAC32
Proton.dur CDCl3

8.18, 8.17, 8.16, 8.15, 8.14, 8.13, 8.12, 8.11, 8.10, 8.09, 8.08, 8.07, 8.06, 8.05, 8.04, 8.03, 8.02, 8.01, 7.99, 7.98, 7.97, 7.96, 7.95, 7.94, 7.93, 7.92, 7.91, 7.90, 7.89, 7.88, 7.87, 7.86, 7.85, 7.84, 7.83, 7.82, 7.81, 7.80, 7.79, 7.78, 7.77, 7.76, 7.75, 7.74, 7.73, 7.72, 7.71, 7.70, 7.69, 7.68, 7.67, 7.66, 7.65, 7.64, 7.63, 7.62, 7.61, 7.60, 7.59, 7.58, 7.57, 7.56, 7.55, 7.54, 7.53, 7.52, 7.51, 7.50, 7.49, 7.48, 7.47, 7.46, 7.45, 7.44, 7.43, 7.42, 7.41, 7.40, 7.39, 7.38, 7.37, 7.36, 7.35, 7.34, 7.33, 7.32, 7.31, 7.30, 7.29, 7.28, 7.27, 7.26, 7.25, 7.24, 7.23, 7.22, 7.21, 7.20, 7.19, 7.18, 7.17, 7.16, 7.15, 7.14, 7.13, 7.12, 7.11, 7.10, 7.09, 7.08, 7.07, 7.06, 7.05, 7.04, 7.03, 7.02, 7.01, 7.00, 6.99, 6.98, 6.97, 6.96, 6.95, 6.94, 6.93, 6.92, 6.91, 6.90, 6.89, 6.88, 6.87, 6.86, 6.85, 6.84, 6.83, 6.82, 6.81, 6.80, 6.79, 6.78, 6.77, 6.76, 6.75, 6.74, 6.73, 6.72, 6.71, 6.70, 6.69, 6.68, 6.67, 6.66, 6.65, 6.64, 6.63, 6.62, 6.61, 6.60, 6.59, 6.58, 6.57, 6.56, 6.55, 6.54, 6.53, 6.52, 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-0.12, -0.13, -0.14, -0.15, -0.16, -0.17, -0.18, -0.19, -0.20, -0.21, -0.22, -0.23, -0.24, -0.25, -0.26, -0.27, -0.28, -0.29, -0.30, -0.31, -0.32, -0.33, -0.34, -0.35, -0.36, -0.37, -0.38, -0.39, -0.40, -0.41, -0.42, -0.43, -0.44, -0.45, -0.46, -0.47, -0.48, -0.49, -0.50



09163920.11.1.1r
SLC:LNB:LB-001-FRAC32
Carbon.dur CDCl3 /home/nmr/localdata/walk

166.81

148.89

132.73

132.17

131.80

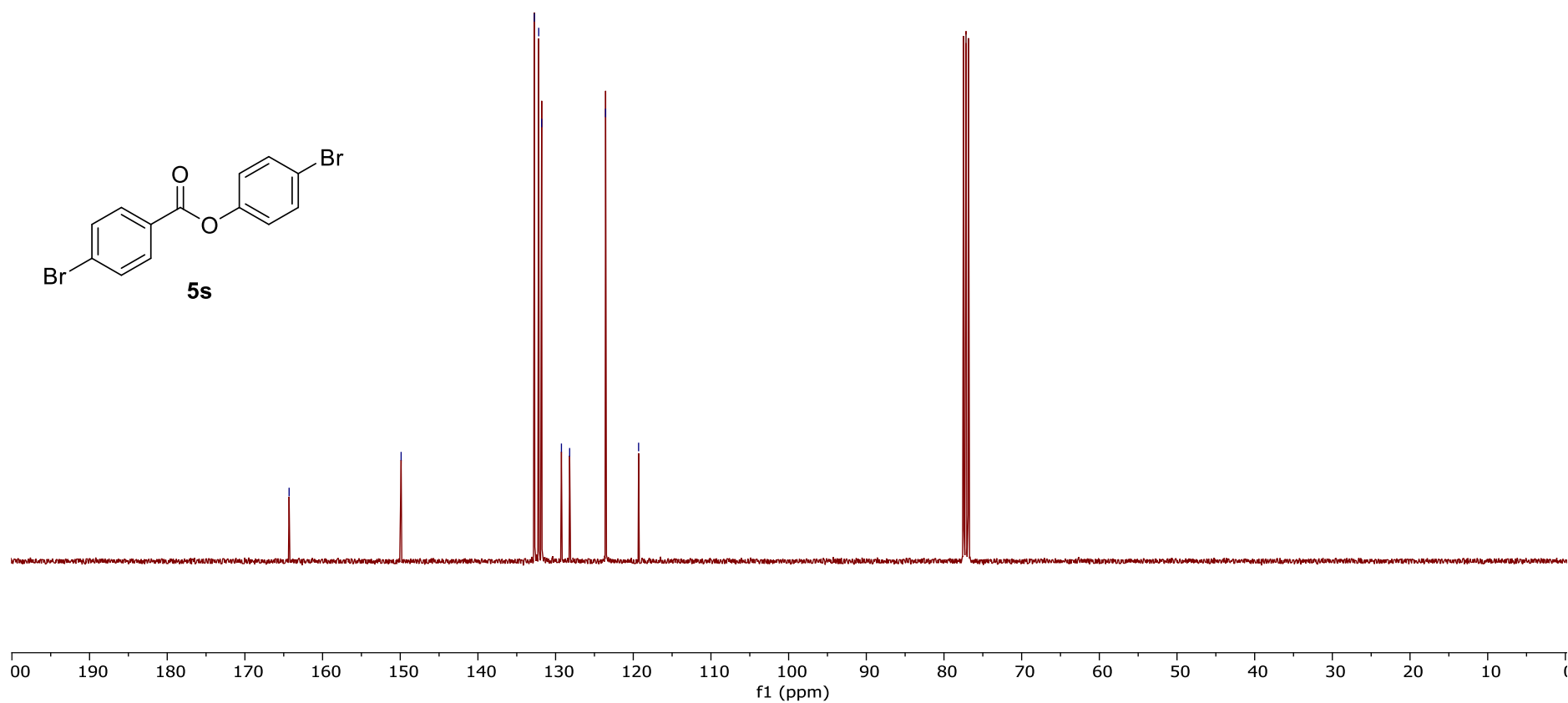
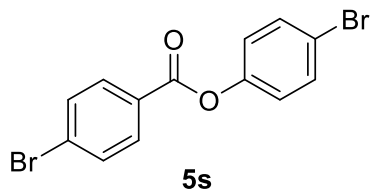
129.25

128.19

123.56

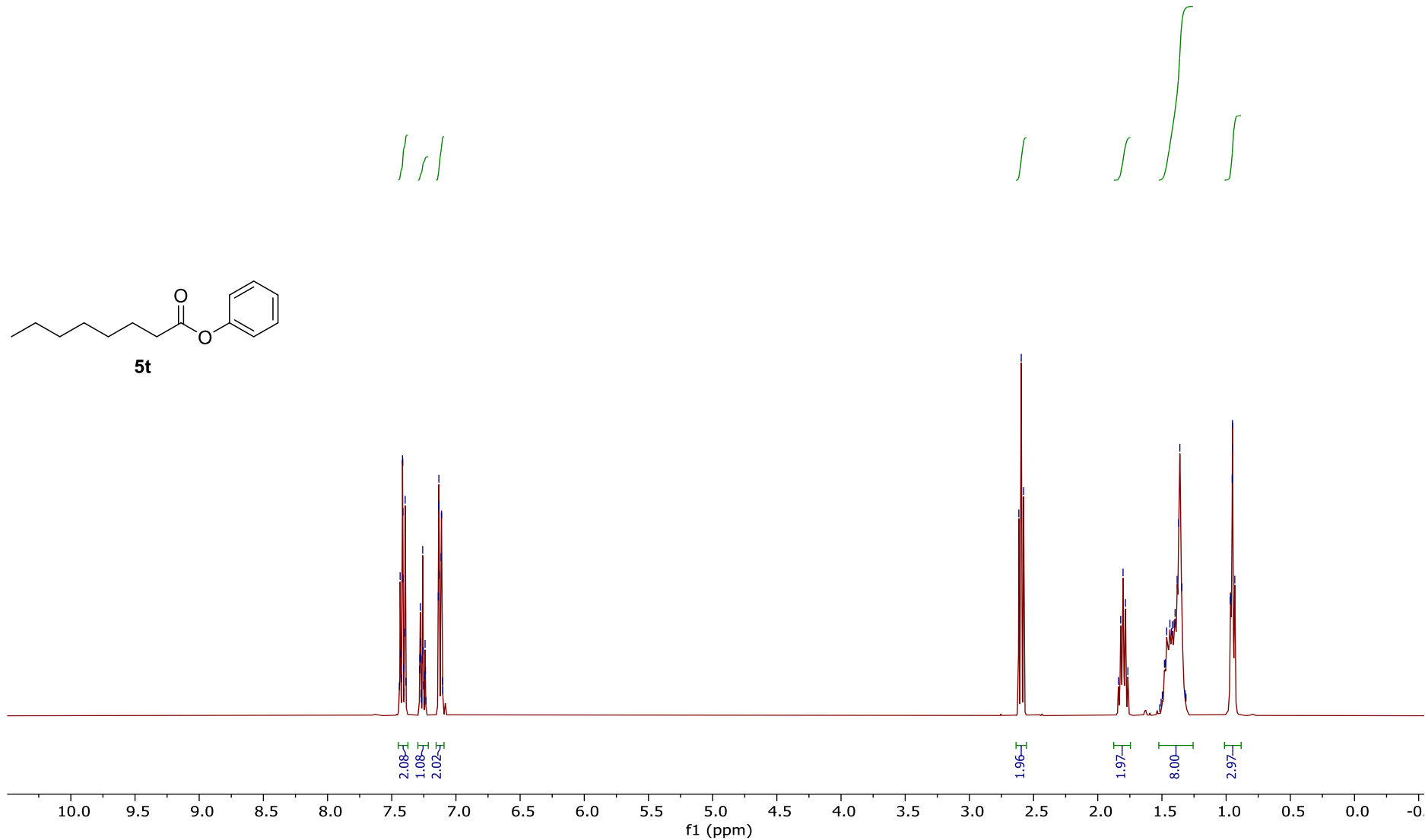
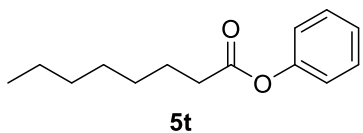
119.32

77.16 CDCl3

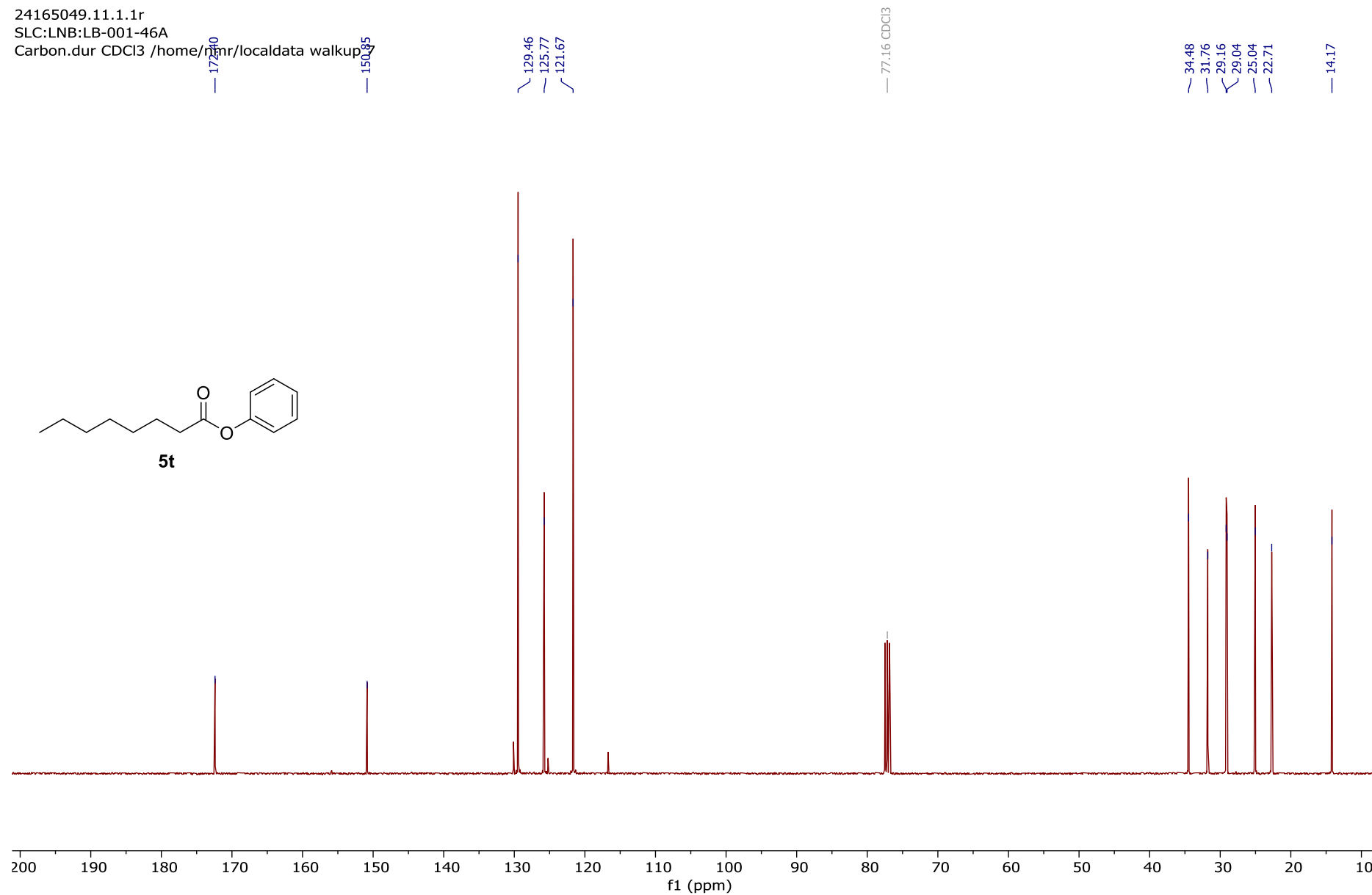
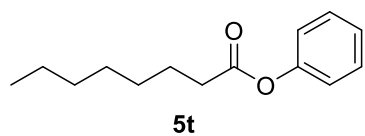


24165049.10.11
SLC:LNB:LB-001-46A
Proton1.icon CDCl3 /home/nmr/localdata/walkup/

2.61
2.60
2.58
1.84
1.82
1.80
1.78
1.77
1.52
1.51
1.50
1.49
1.48
1.47
1.46
1.44
1.43
1.42
1.41
1.40
1.38
1.37
1.36
1.35
1.32
1.31
0.97
0.96
0.95
0.95
0.93



24165049.11.1.1r
SLC:LNB:LB-001-46A
Carbon.dur CDCl3 /home/nmr/localdata walkup

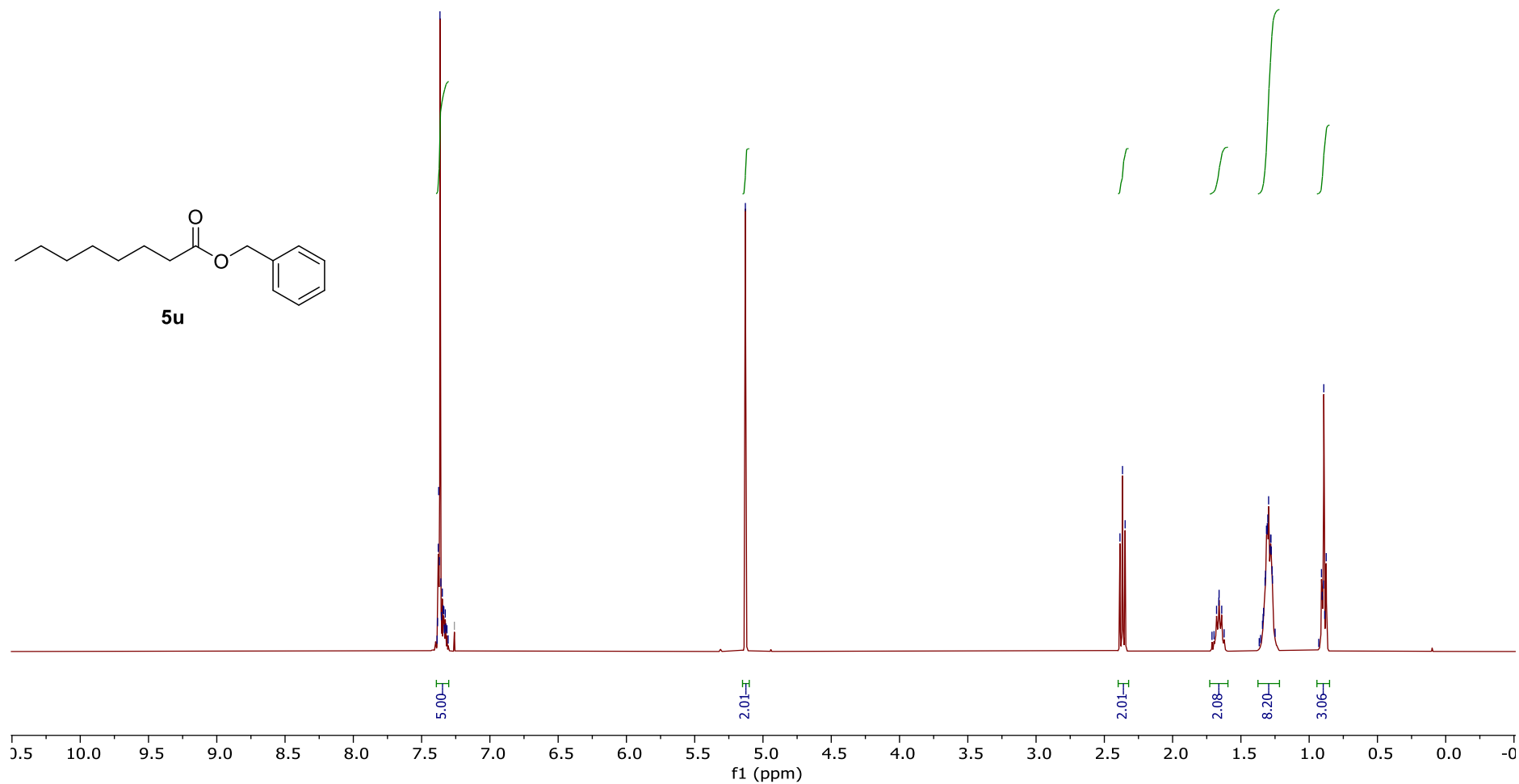
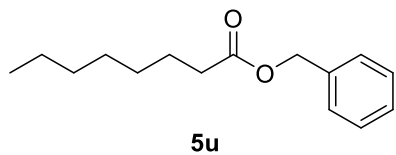


24172807.10.1.1r
SLC:LNB:LB-001-46B-PURIFY
Proton1.icon CDCl3 /home/nmr/local/

7.737
7.736
7.735
7.734
7.733
7.733
7.732
7.731
7.726 CDCl3

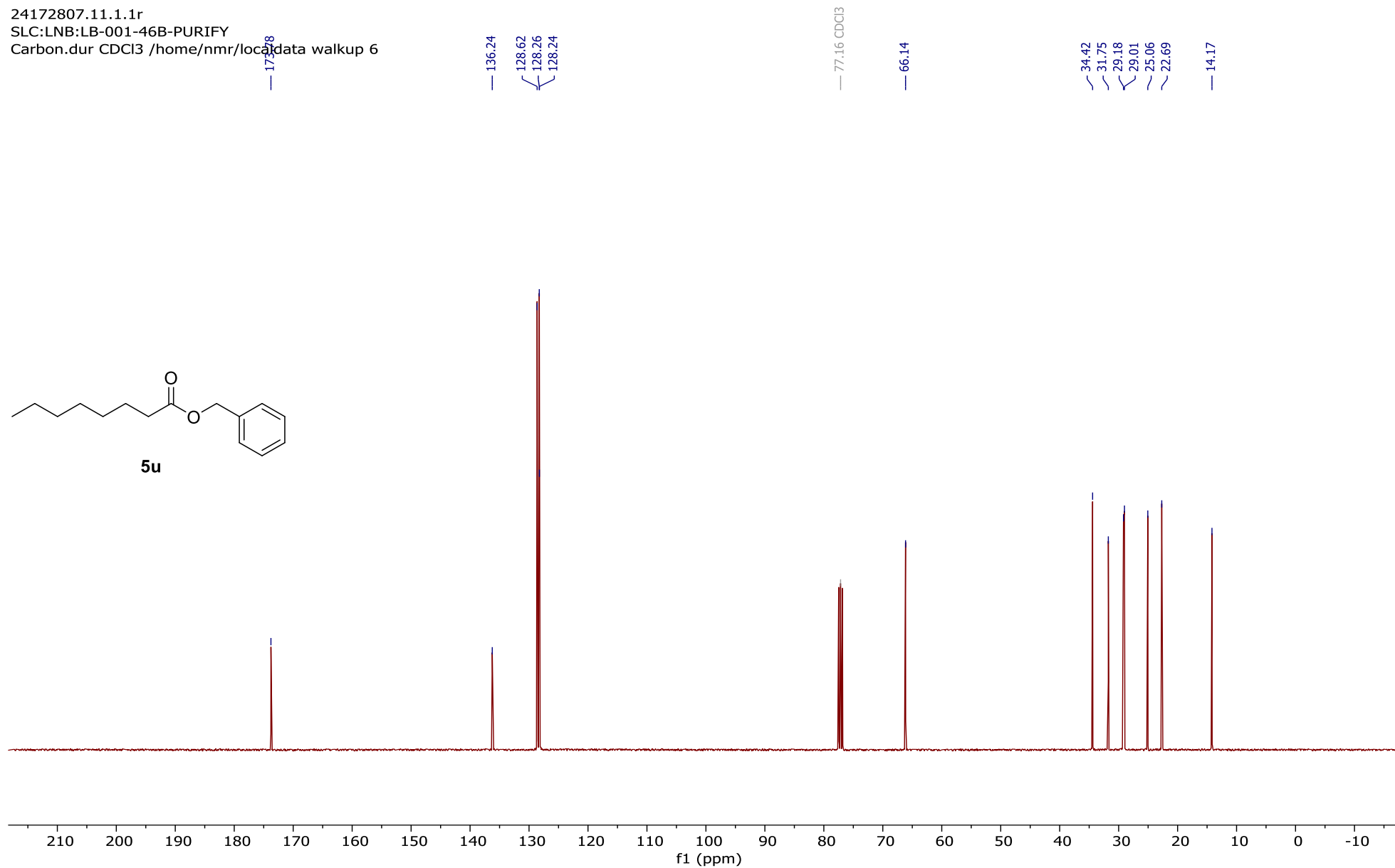
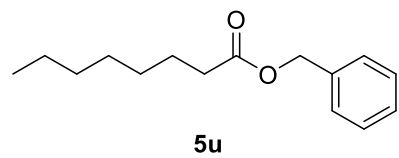
5.13

2.39
2.37
2.35
1.71
1.70
1.68
1.66
1.66
1.64
1.62
1.37
1.36
1.34
1.34
1.33
1.32
1.32
1.31
1.31
1.31
1.30
1.30
1.29
1.28
1.28
1.28
1.27
1.27
1.25
0.93
0.91
0.90
0.89
0.89
0.88

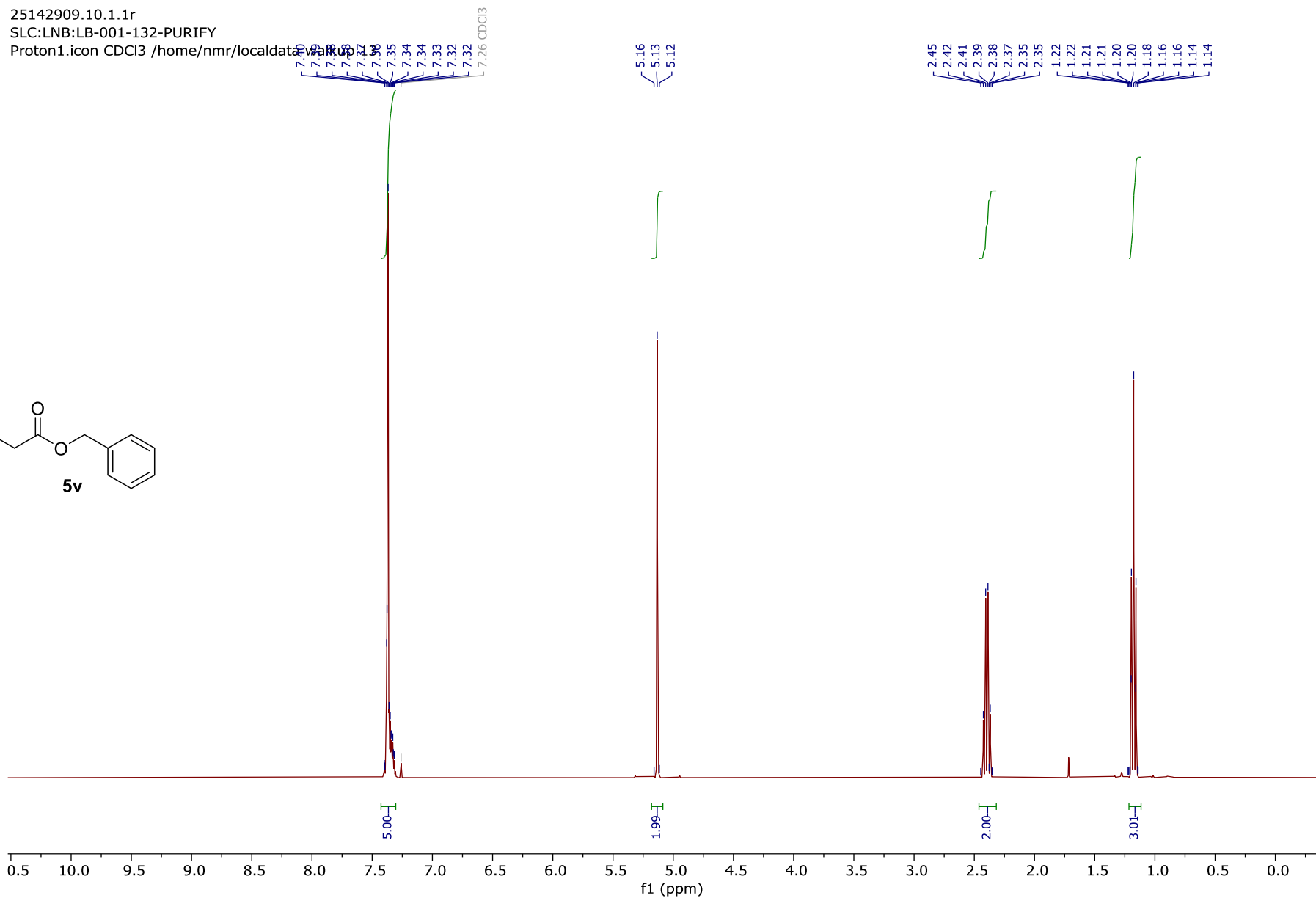
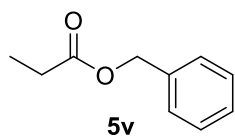


S-68

24172807.11.1.1r
SLC:LNB:LB-001-46B-PURIFY
Carbon.dur CDCl3 /home/nmr/local/data/walkup 6

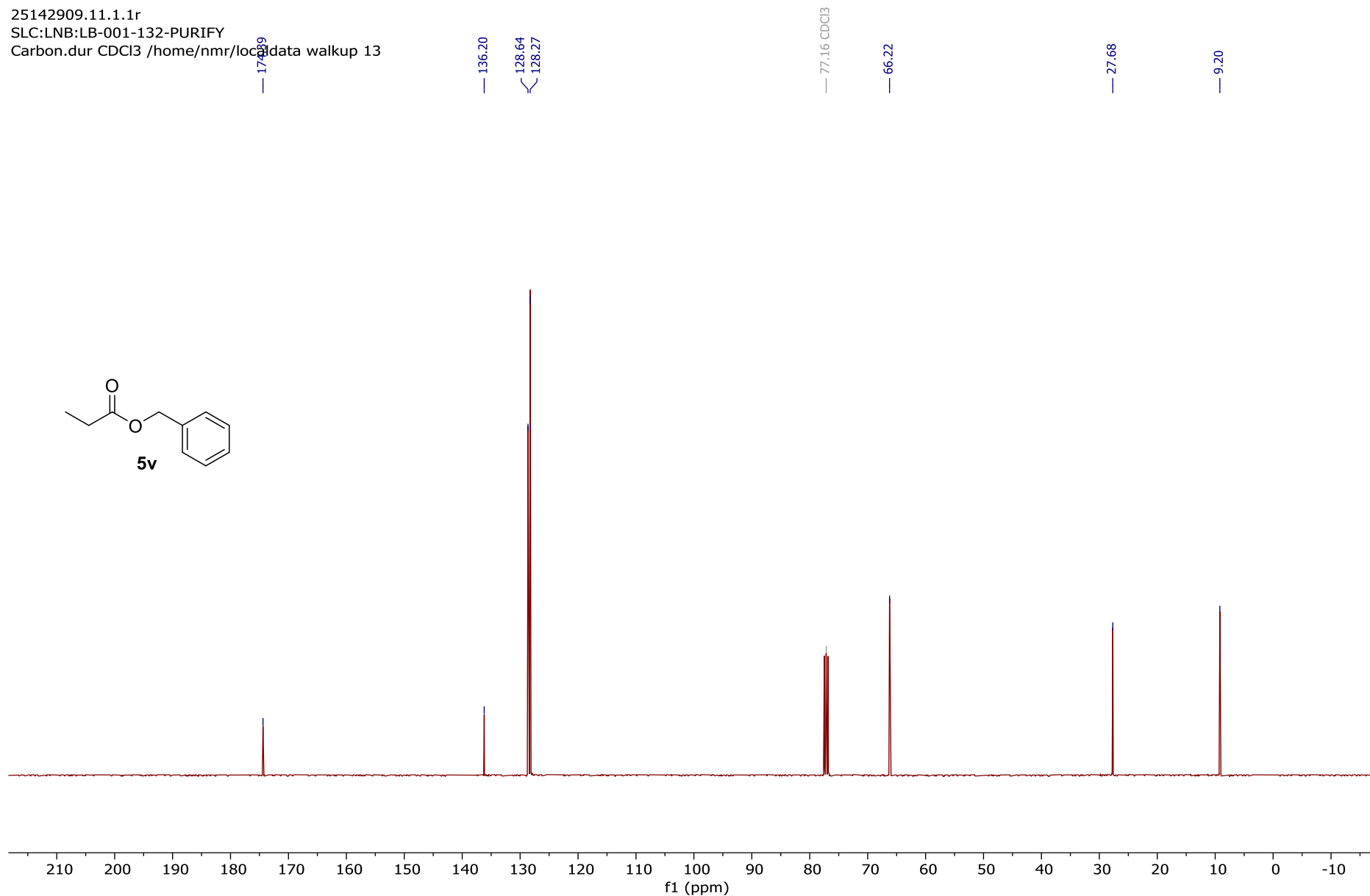
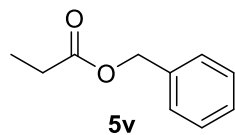


25142909.10.1.1r
SLC:LNB:LB-001-132-PURIFY
Proton1.icon CDCl3 /home/nmr/localdata/



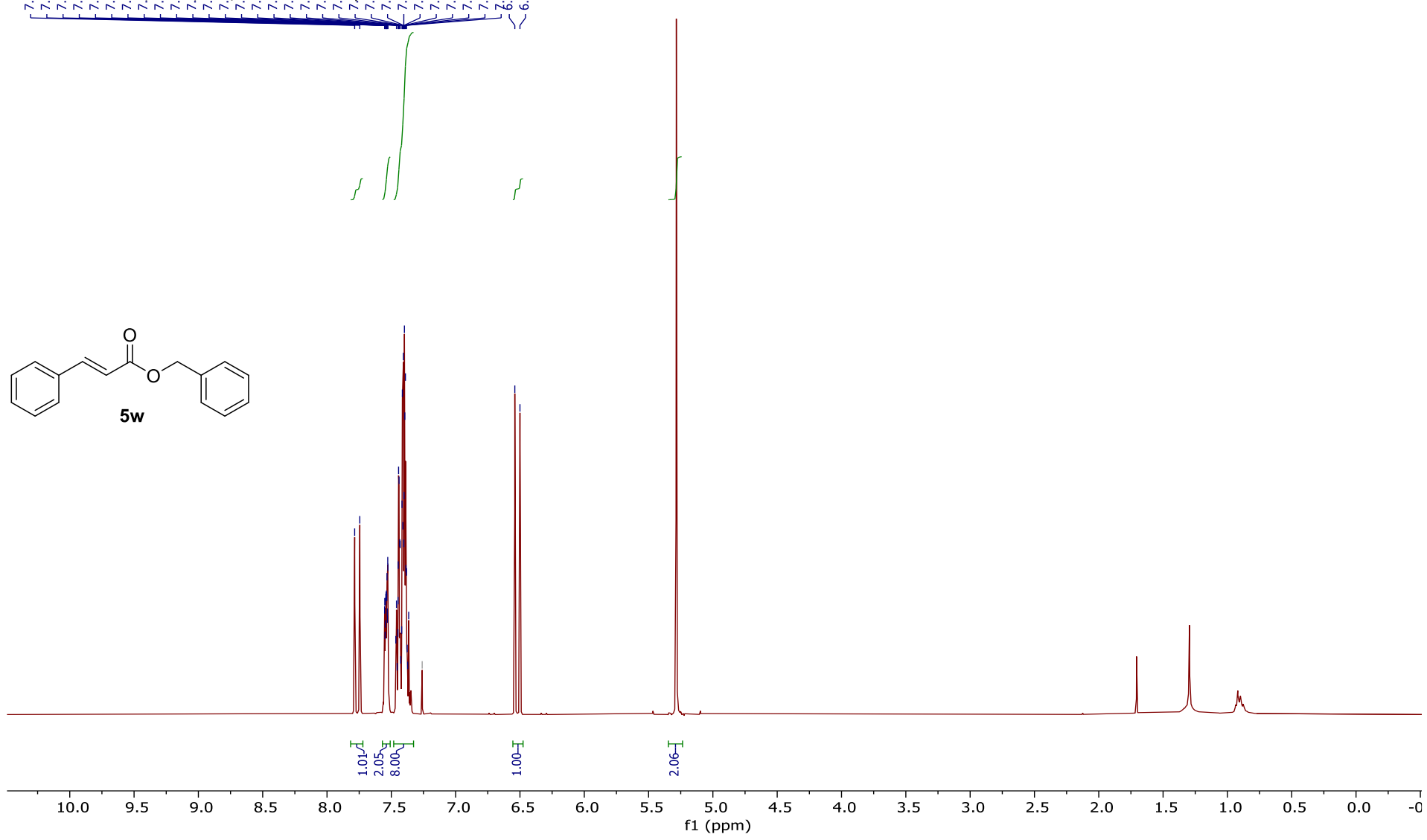
S-70

25142909.11.1.1r
SLC:LNB:LB-001-132-PURIFY
Carbon.dur CDCl3 /home/nmr/locdata walkup 13



13154617.10.1.1r
SLC:LNB:LB-001-100-PURIFY

Proton NMR (CDCl₃) of compound 5w



13154617.11.1.1r
SLC:LNB:LB-001-100-PURIFY

Carbon_10min.dur CDCl3 /home/nmr/localdata/walkup 2022

166.86

145.27

136.16

134.43

130.44

128.98

128.69

128.38

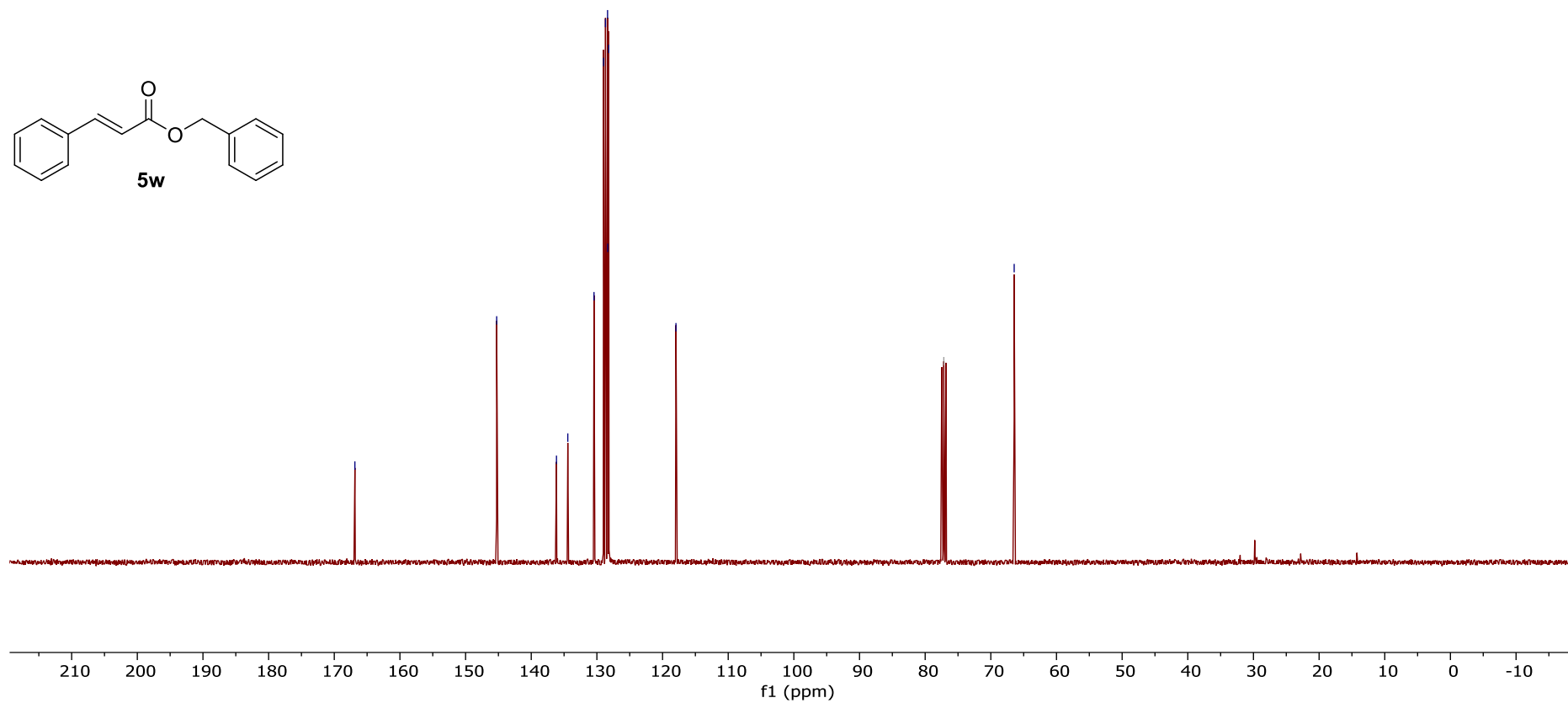
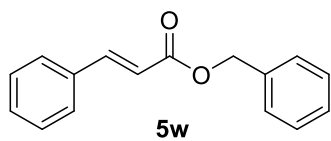
128.35

128.20

117.96

77.16 CDCl3

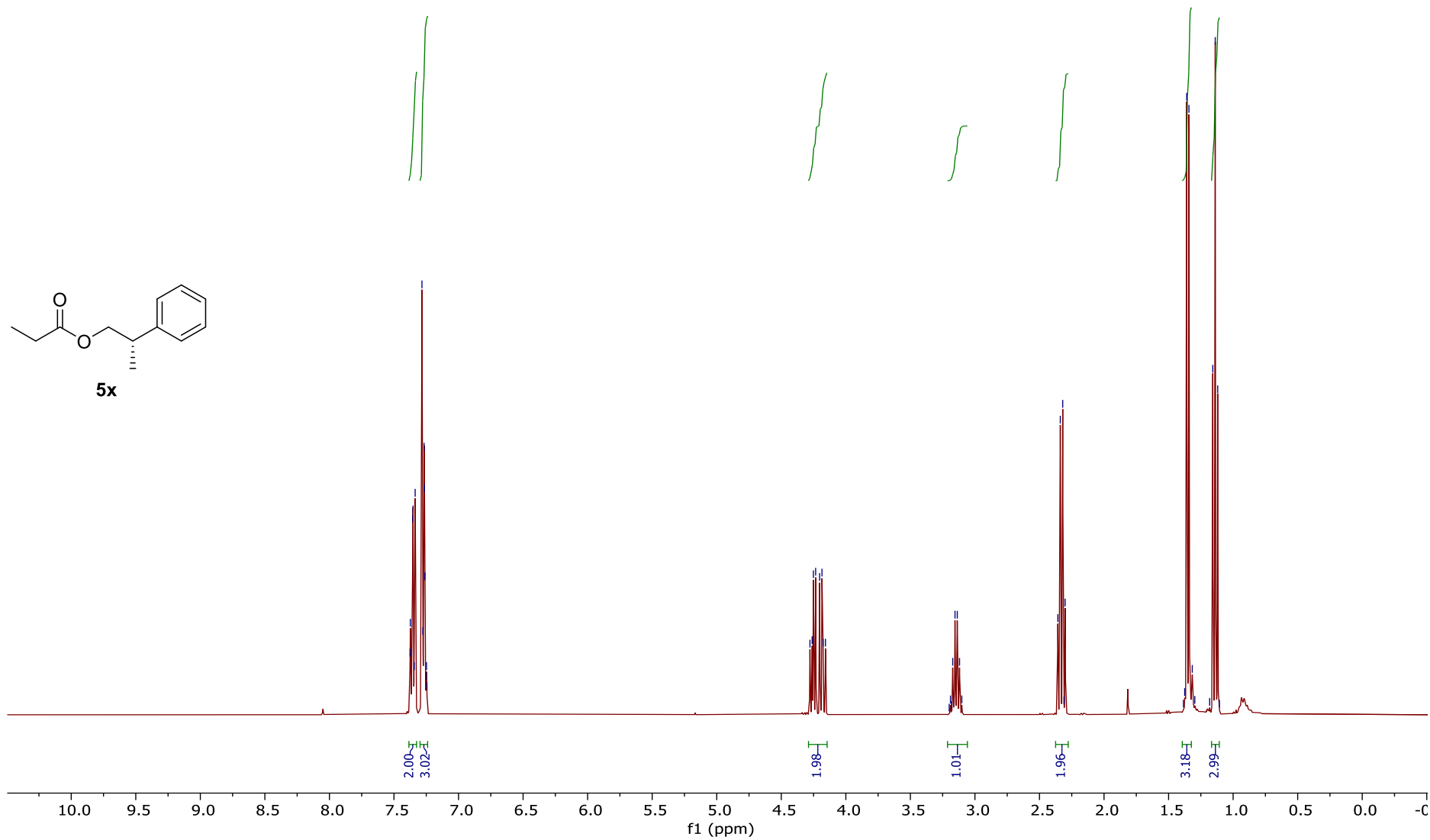
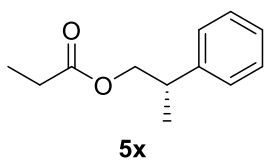
66.44



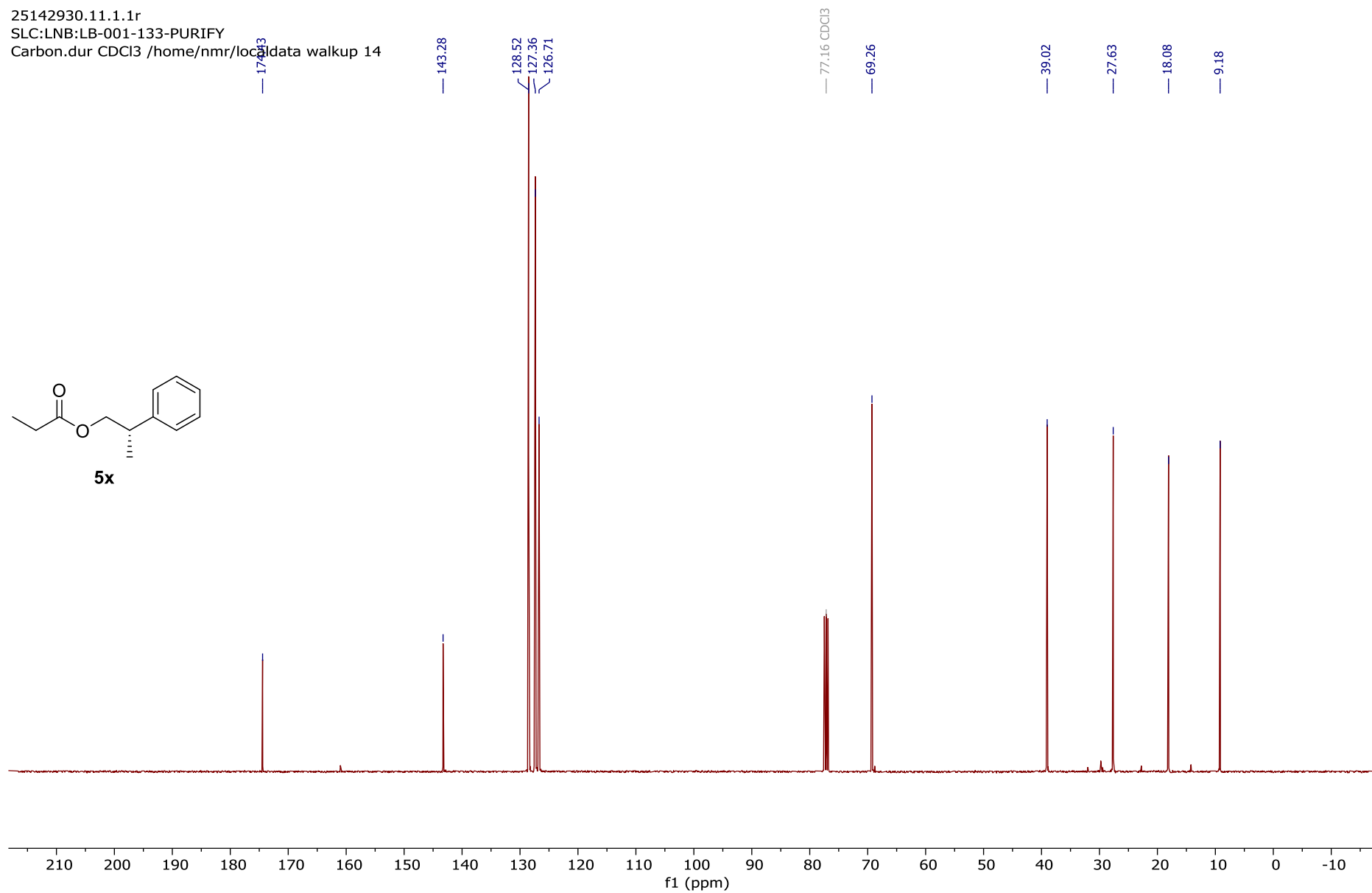
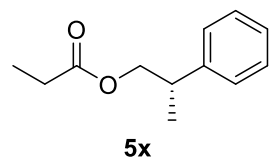
25142930.10.1.1r
SLC:LNB:LB-001-133-PURIFY
Proton1.icon CDCl3 /home/nmr/localdata walkup 14

7.38
7.37
7.35
7.35
7.34
7.34
7.28
7.28
7.27
7.26
7.26
7.25
7.25
7.24

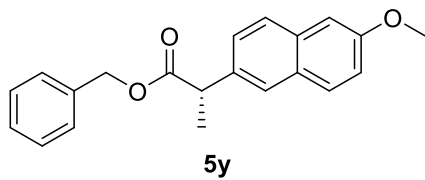
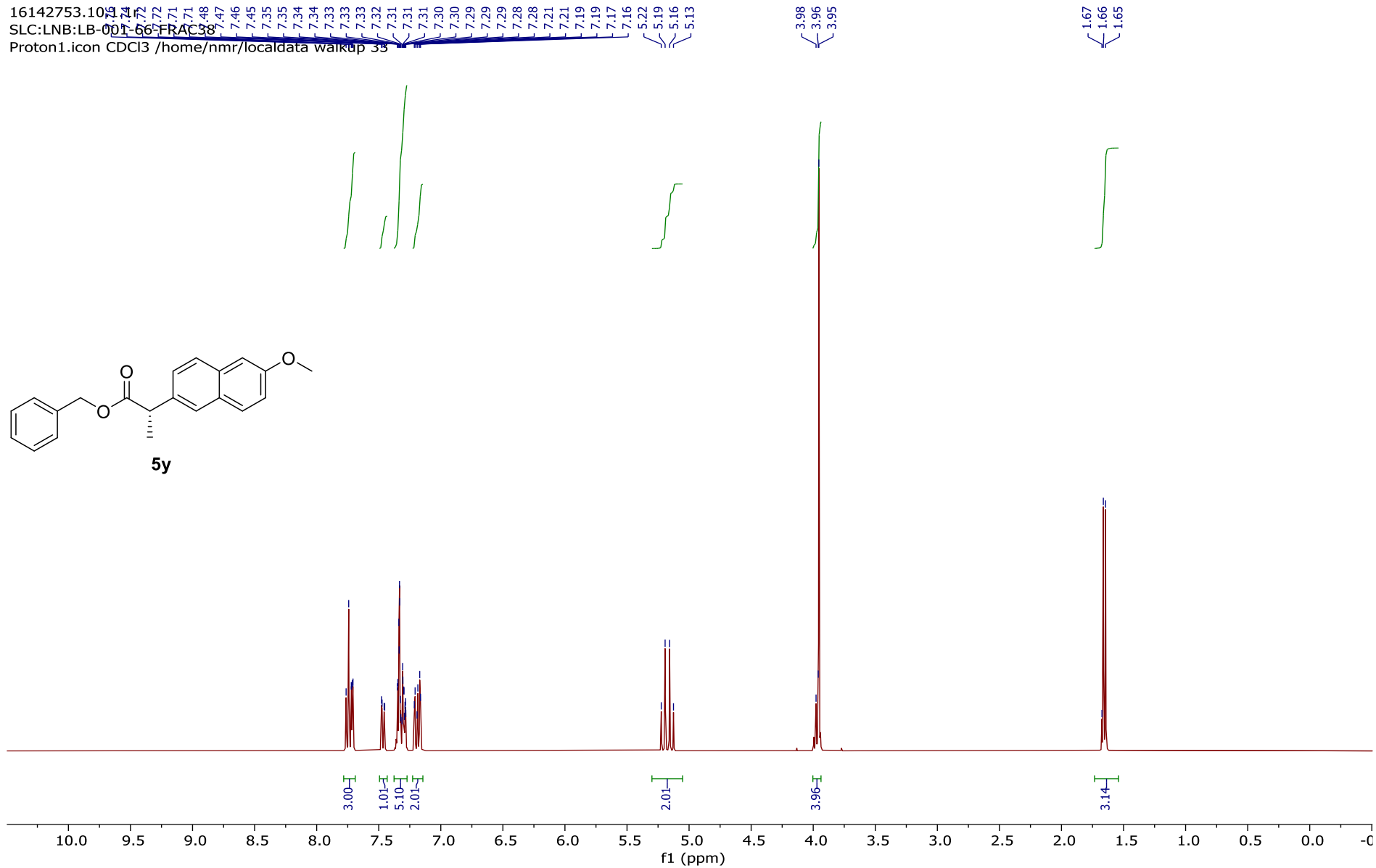
4.28
4.26
4.25
4.23
4.20
4.18
4.18
4.16
3.20
3.19
3.18
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3.15
3.14
3.12
3.10
2.36
2.34
2.32
2.31
2.30
1.38
1.38
1.36
1.34
1.32
1.30
1.18
1.16
1.14
1.12
1.11



25142930.11.1.1r
SLC:LNB:LB-001-133-PURIFY
Carbon.dur CDCl3 /home/nmr/local/data walkup 14



16142753.10
SLC:LNB:LB-001-66-FRAC38
Proton1.icon CDCl3 /home/nmr/localdata/waipup 33



16142753.11.1.1r

SLC:LNB:LB-001-66-FRAC38

Carbon.dur CDCl3 /home/nmr/localdata/walkup/

177.656

155.71

136.08

135.64

133.79

129.39

129.00

128.56

128.17

128.03

127.24

126.39

119.07

105.63

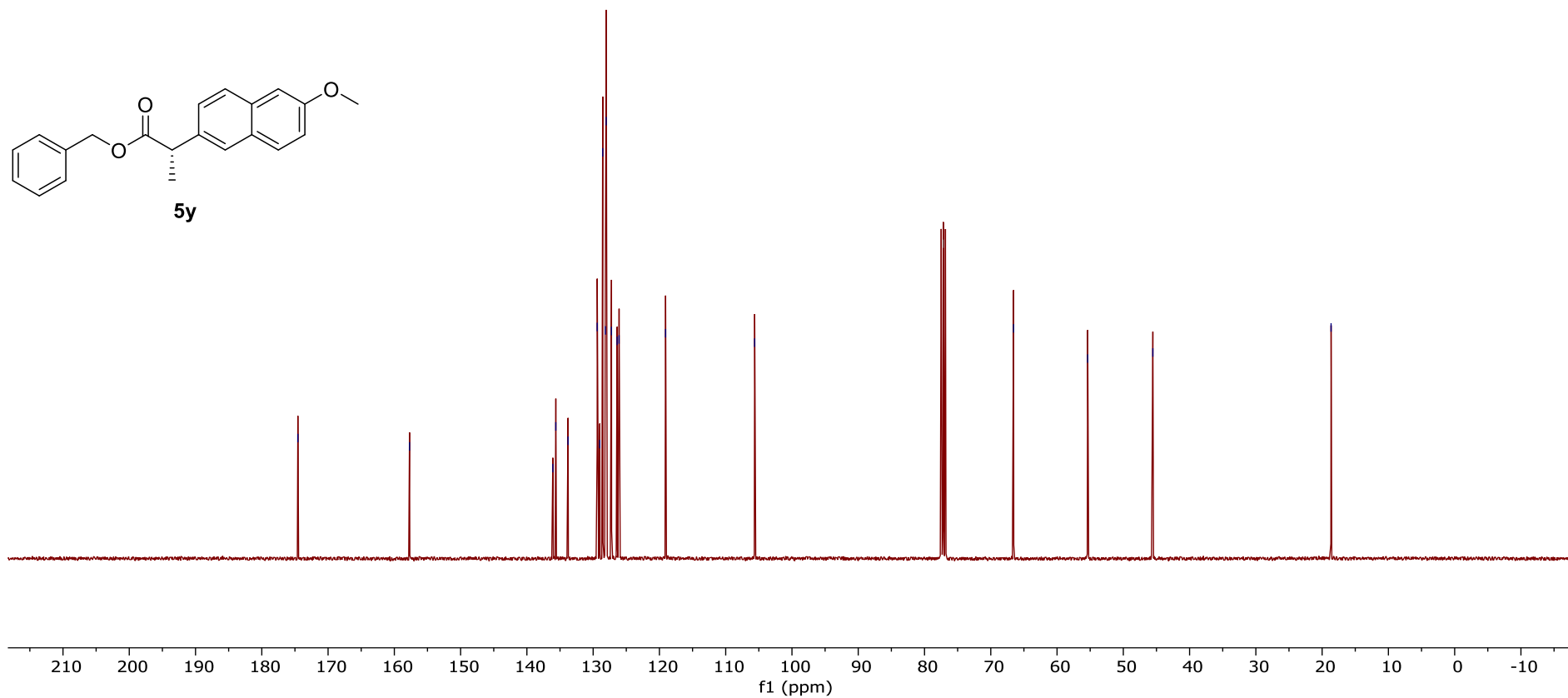
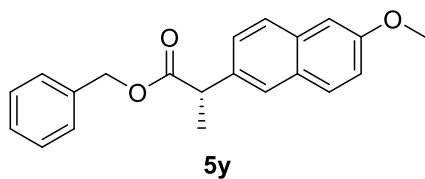
77.16 CDCl3

66.56

55.38

45.54

18.65

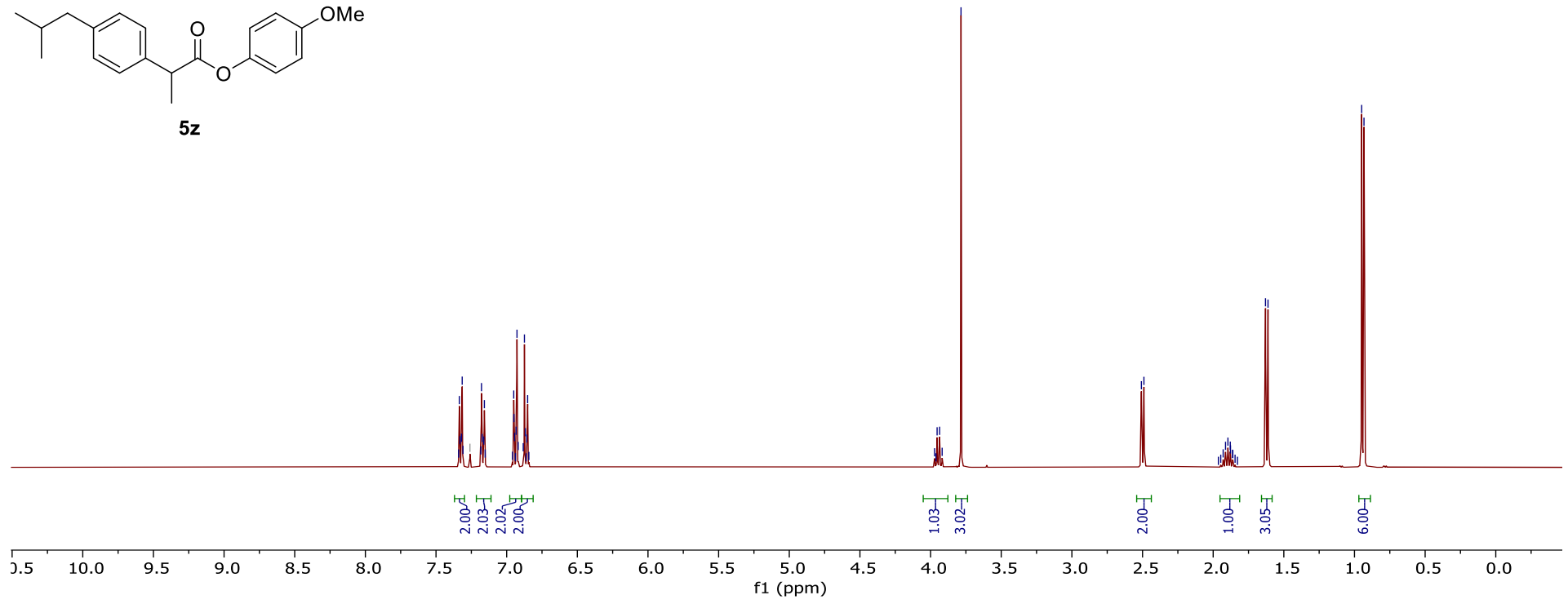
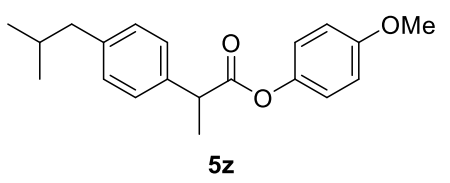
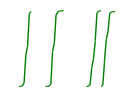


17145200.10.1.1r
SLC:LNB:LB-001-65-PURIFY
Proton1.icon CDCl3 /home/mf/16-01-18

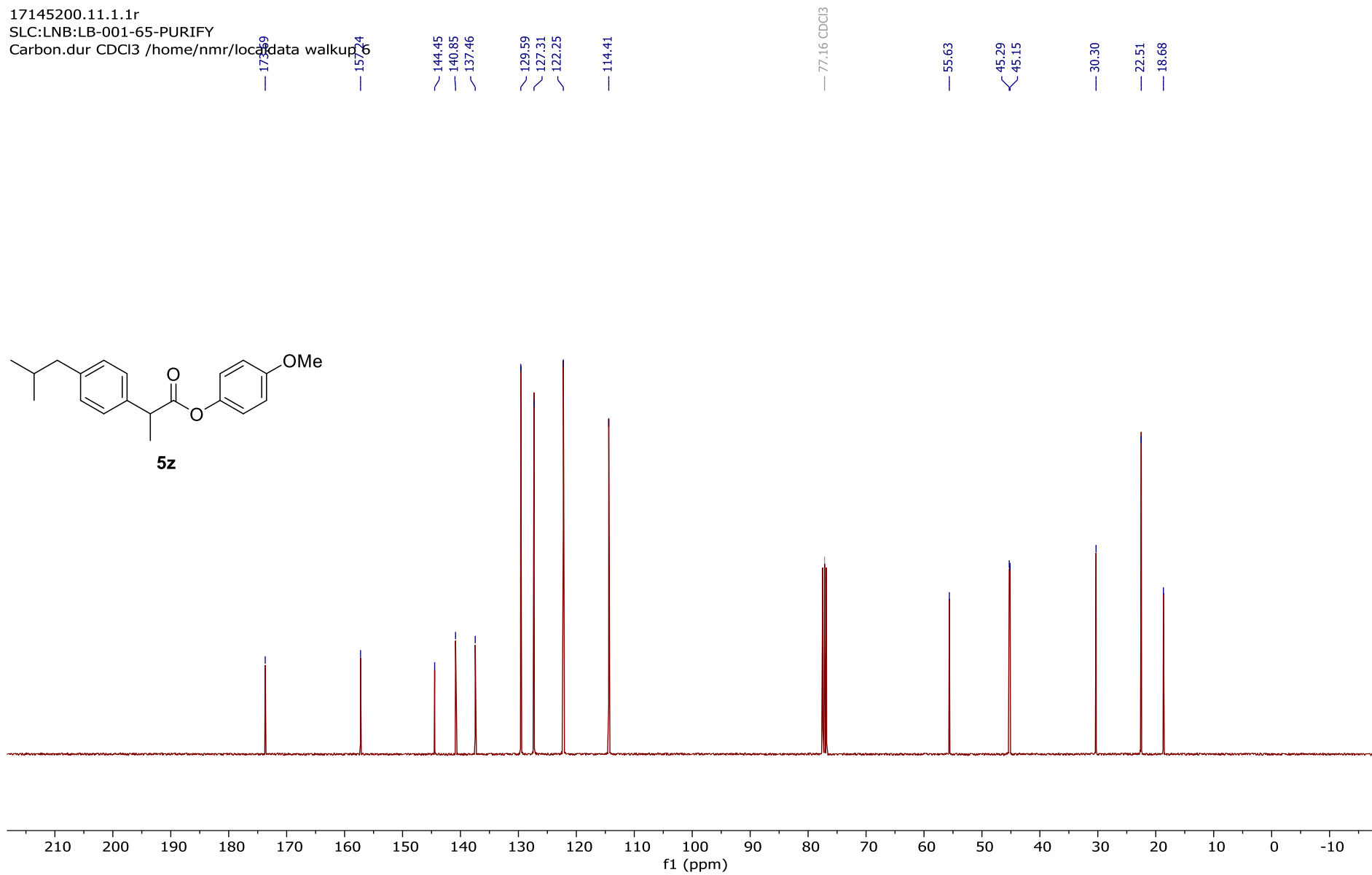
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7.693
7.692
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7.684

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3.96
3.95
3.94
3.92
3.78

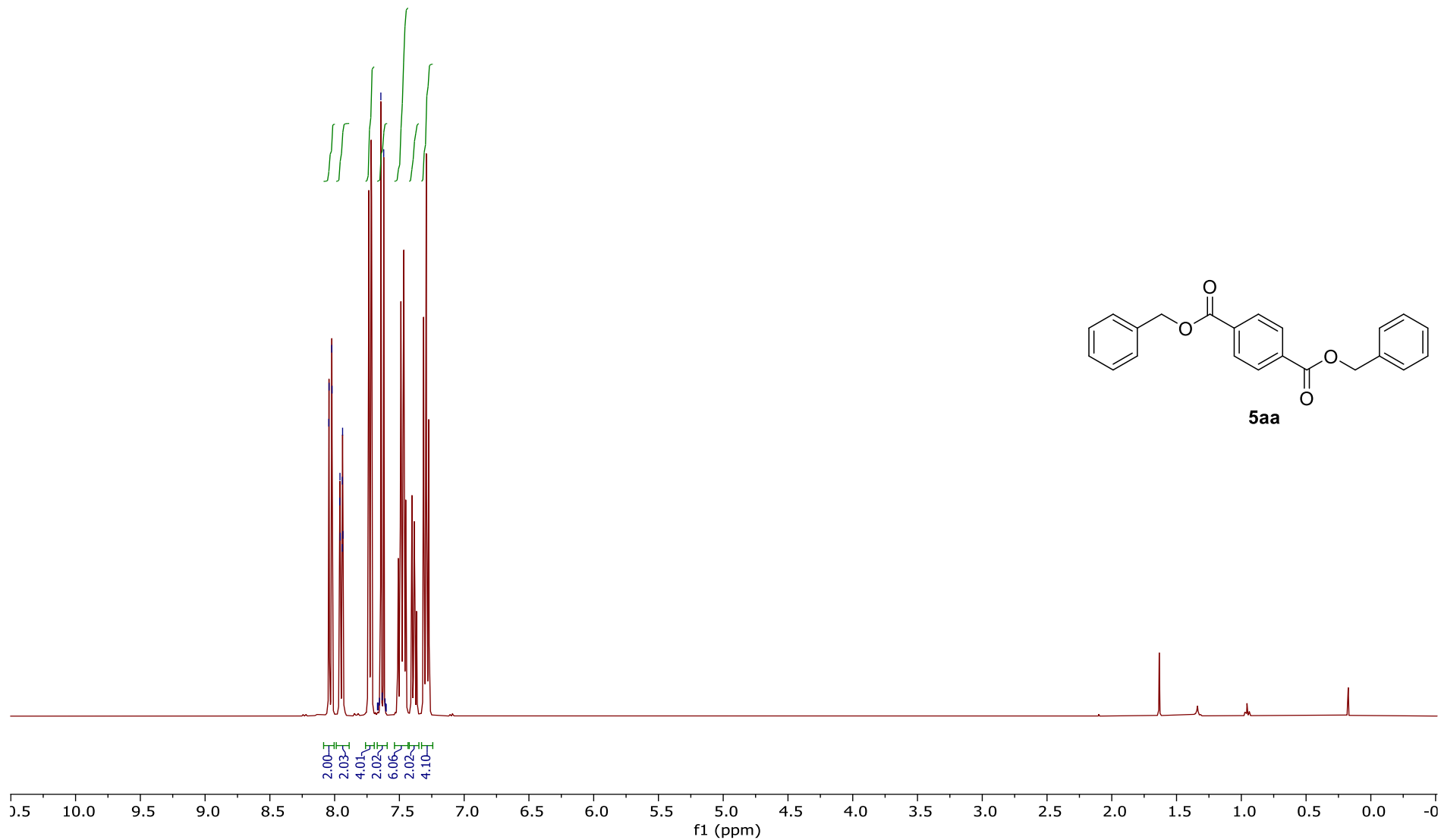
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1.84
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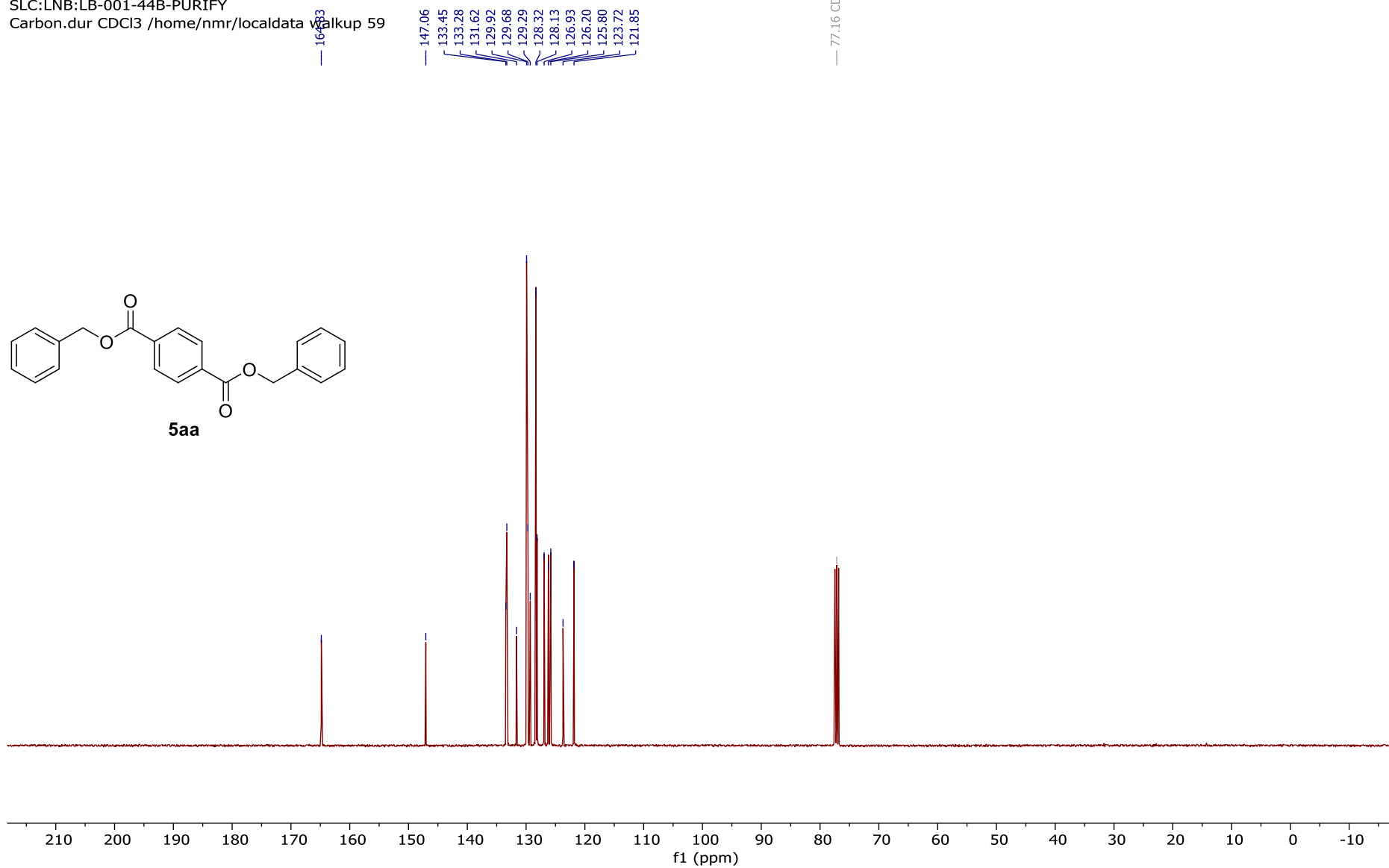


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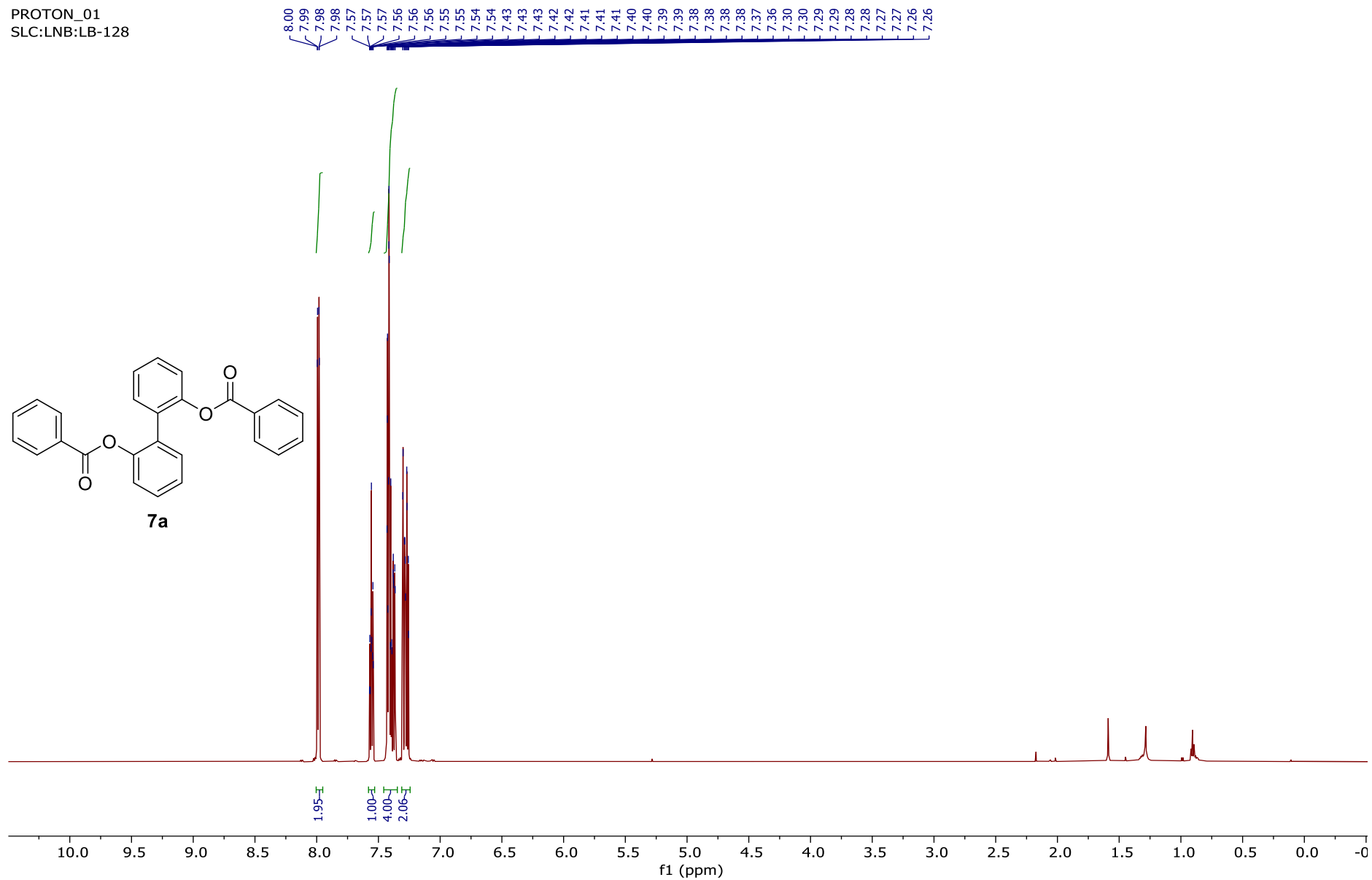


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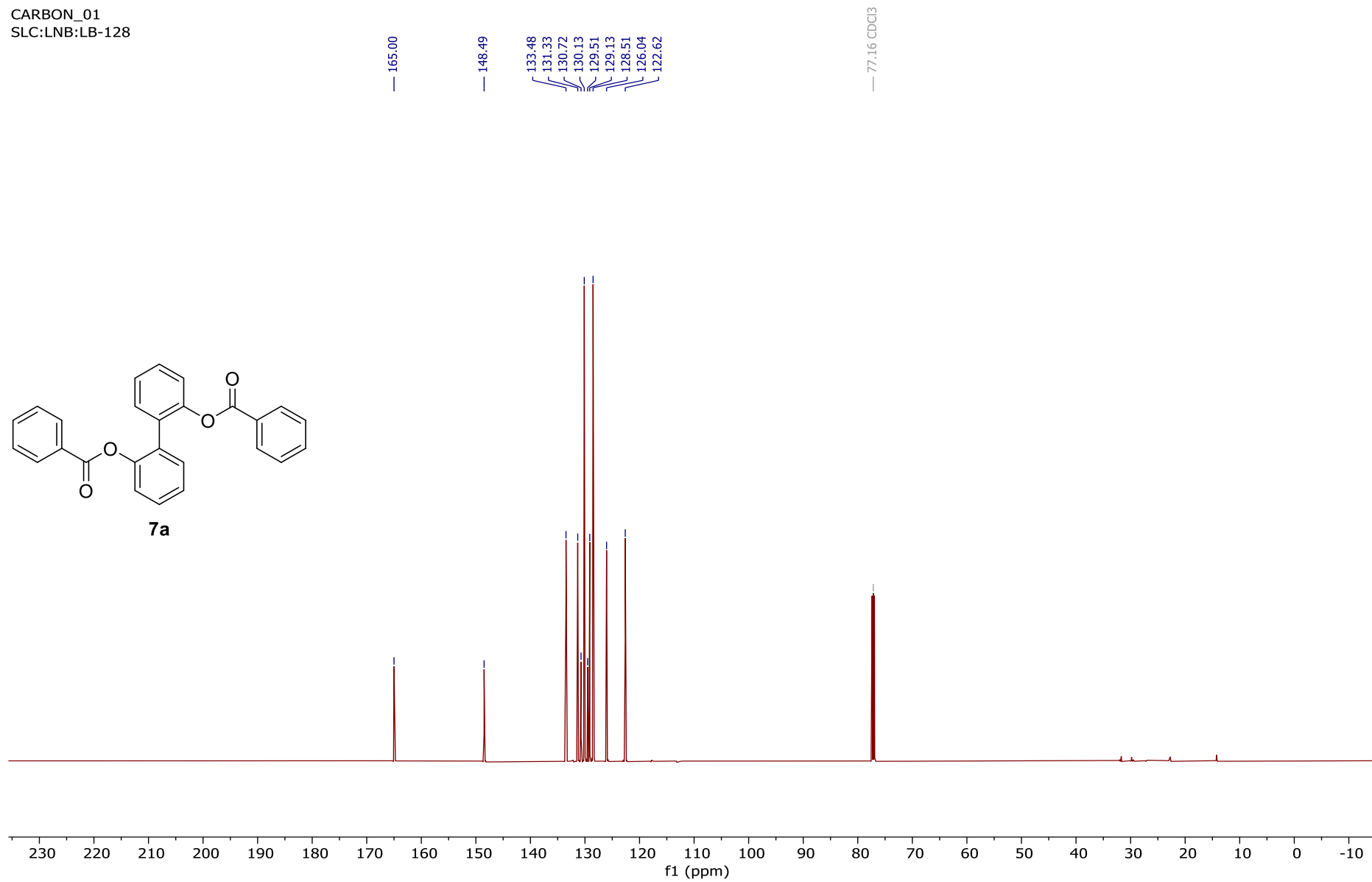
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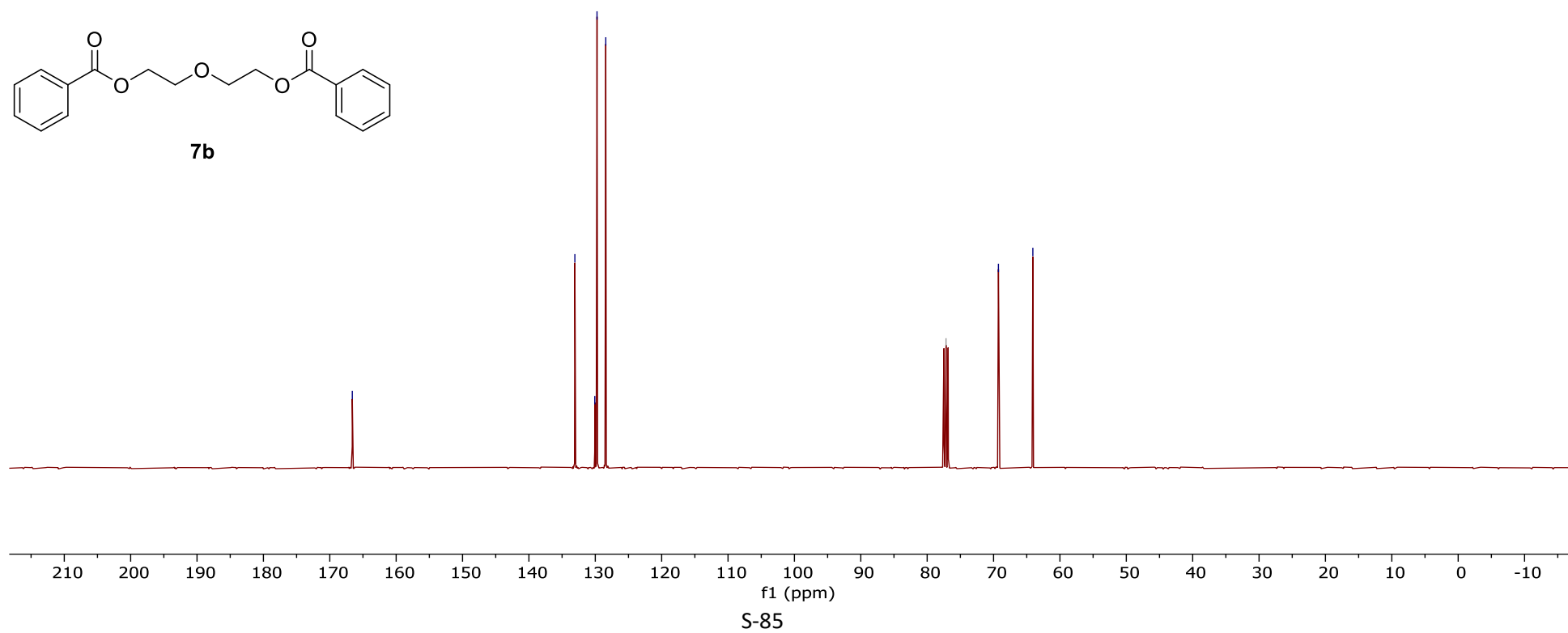
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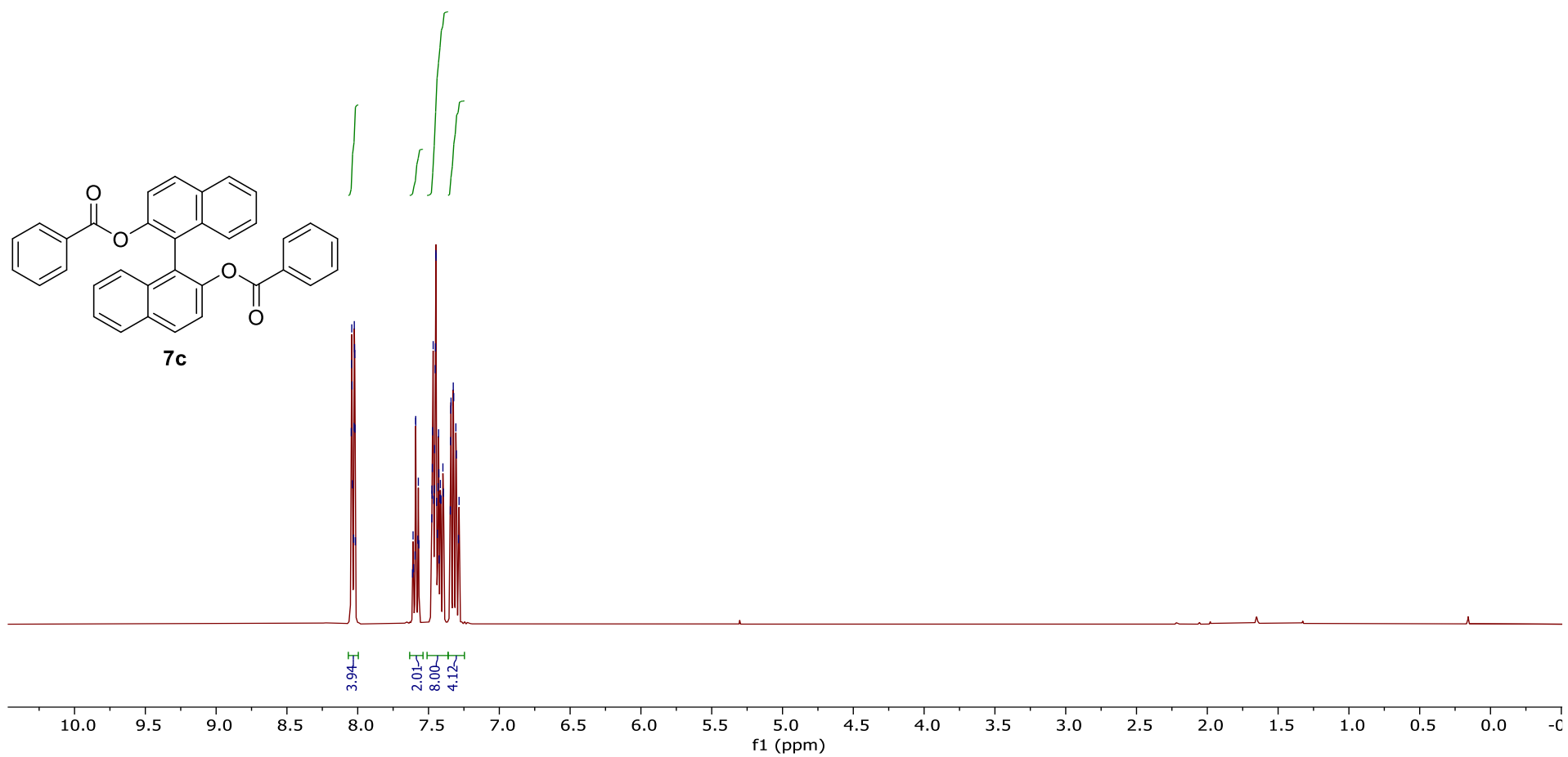
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-41.03
-41.

17151058.11.1.1r
SLC:LNB:LB-001-44D
Carbon.dur CDCl3 /home/nmr/localdata/backup 49



11114603.10.1.1r
SLC:LNB:LB-001-44A-DRY
Proton1.icon CDCl3 /home/nmr/localdata/walkup/45



11114603.11.1.1r
SLC:LNB:LB-001-44A-DRY
Carbon.dur CDCl3 /home/nmr/localdata

166.99

148.43

133.48

131.30

130.67

130.10

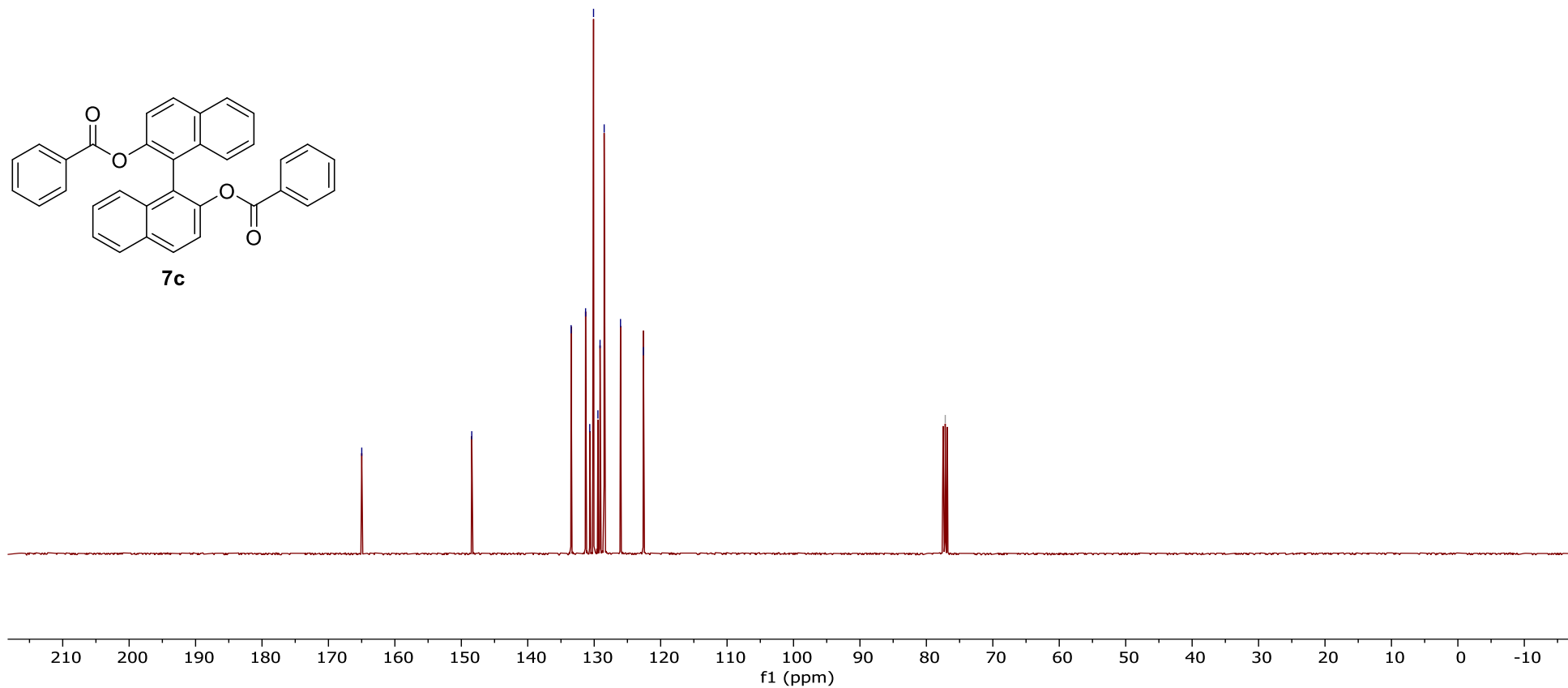
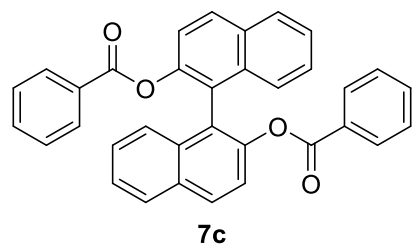
129.44

129.12

128.50

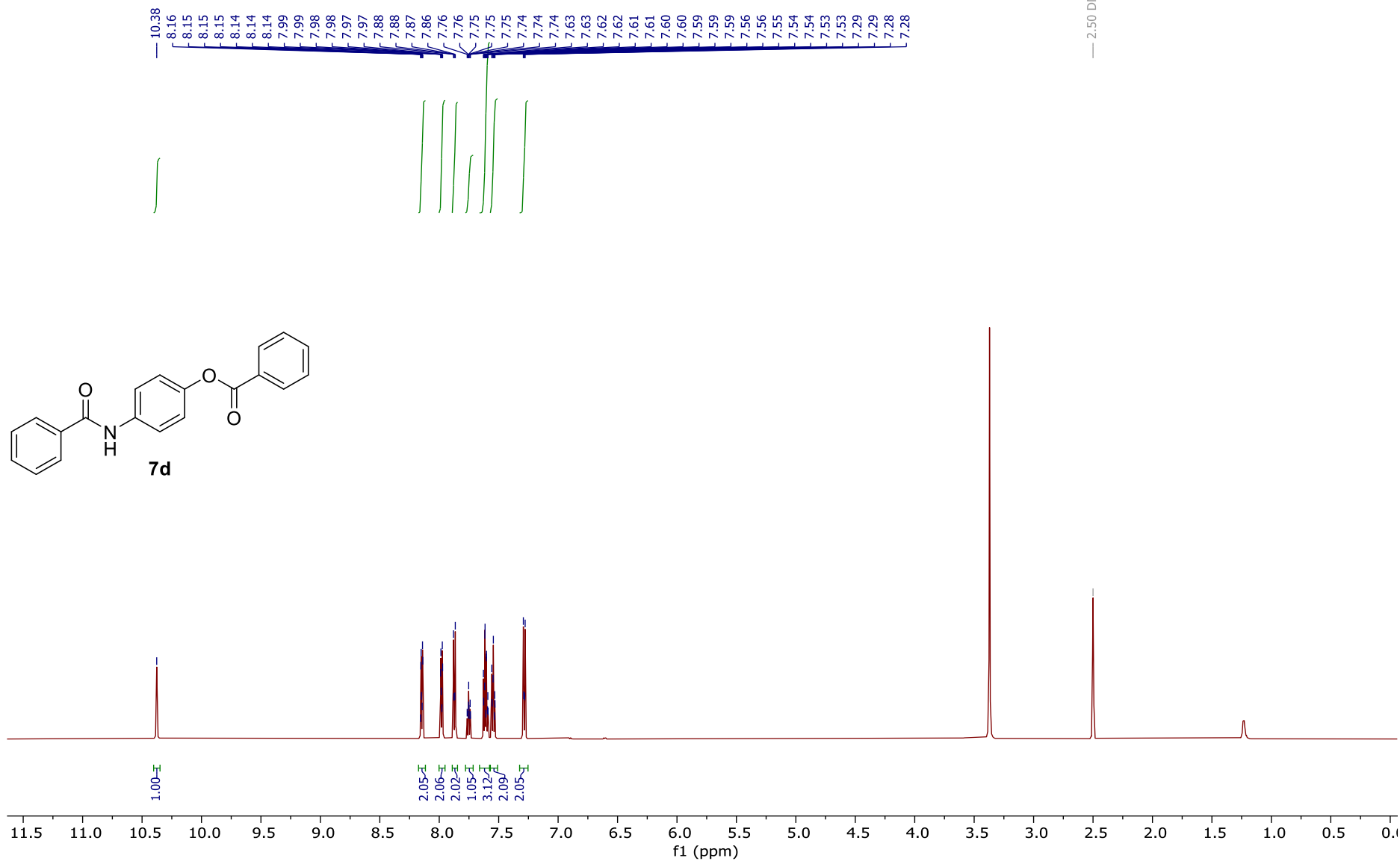
126.04

77.16 CDCl3



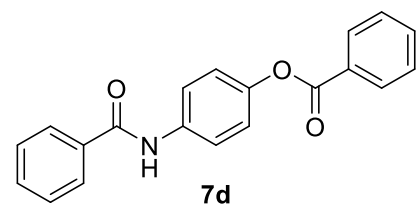
PROTON_02
SLC:LNB:LB-127

2.50 DMSO-d6



S-88

CARBON_01
SLC:LNB:LB-127

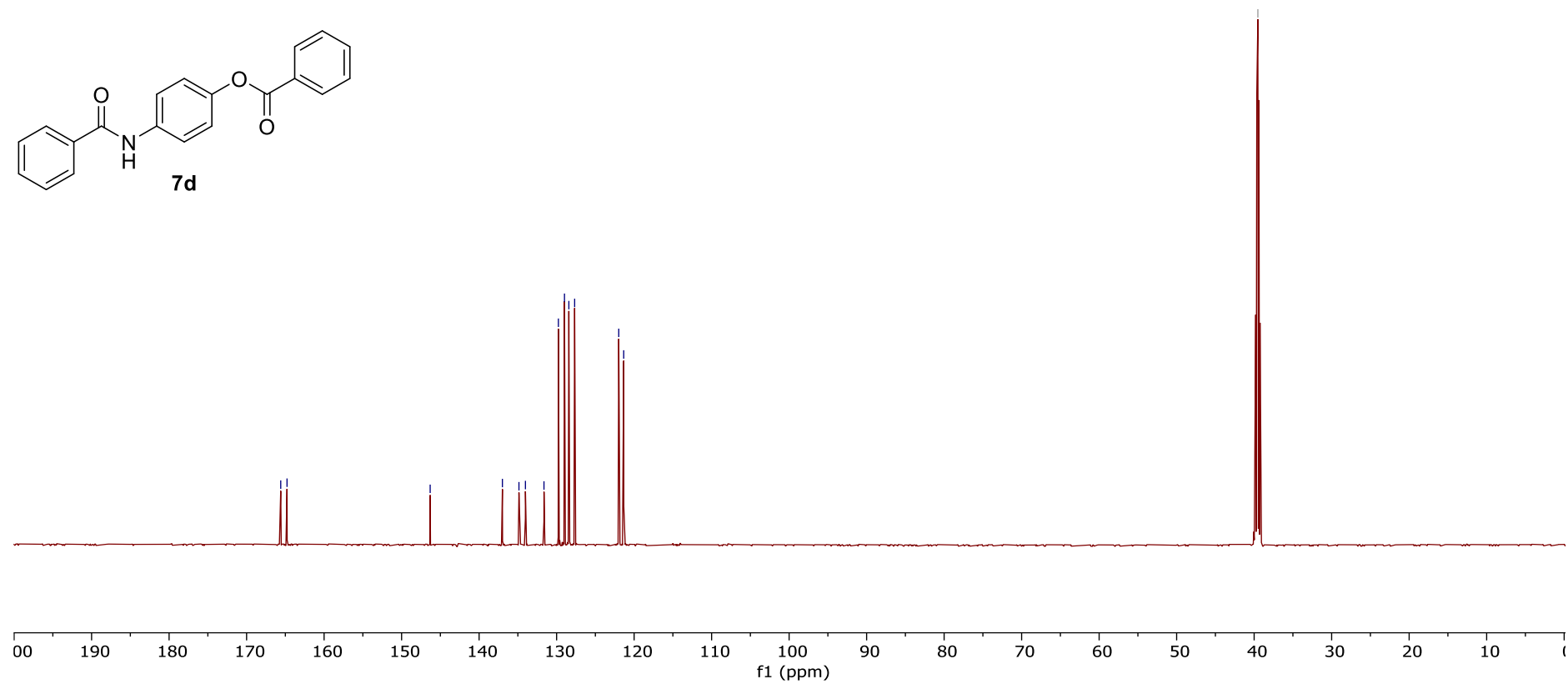


165.60
164.78

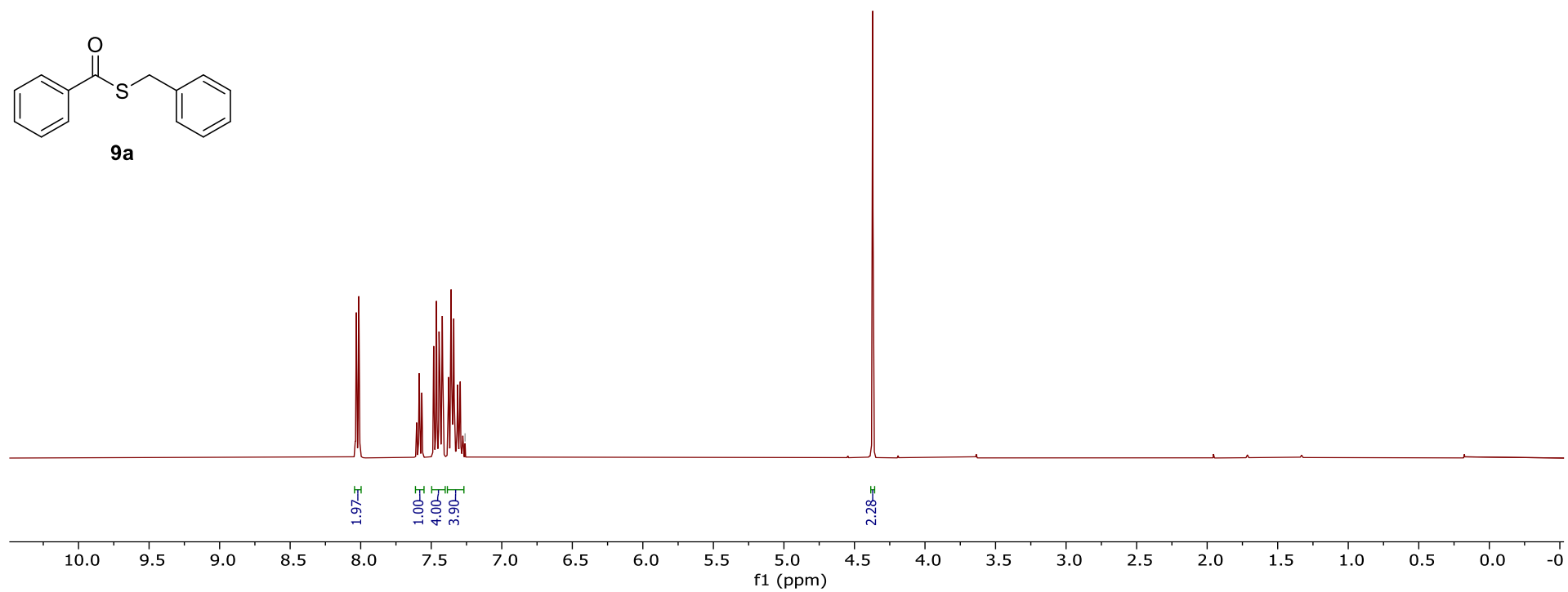
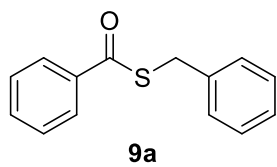
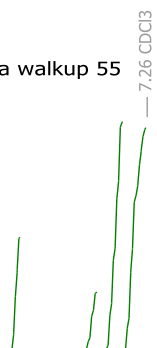
146.31

136.99
134.86
134.02
131.63
129.77
128.98
128.42
127.67
121.98
121.34

39.52 DMSO-d6

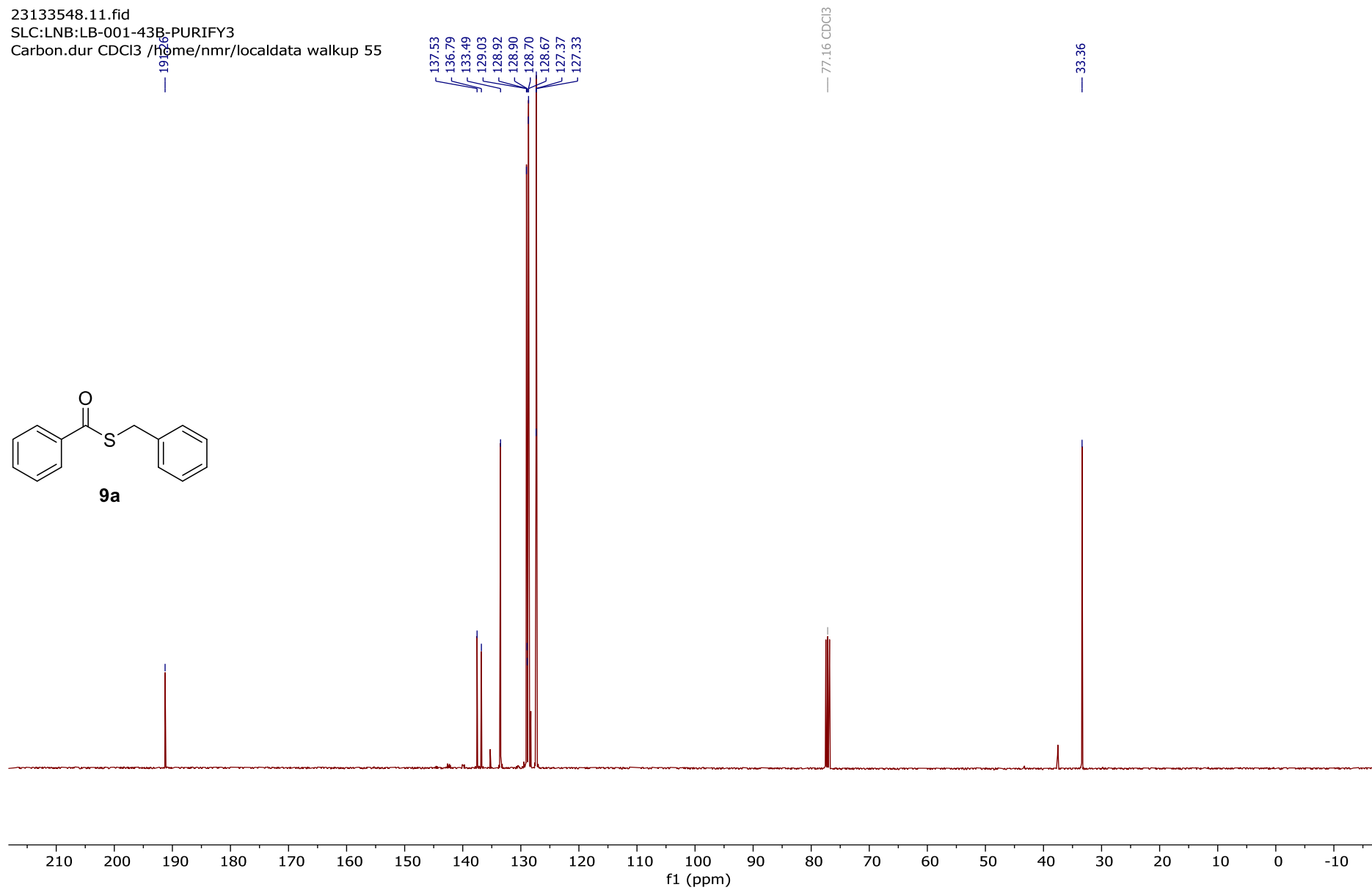
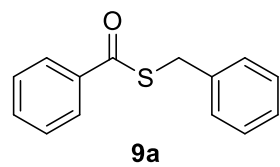


23133548.10.fid
SLC:LNB:LB-001-43B-PURIFY3
Proton1.icon CDCl3 /home/nmr/localdata walkup 55



S-90

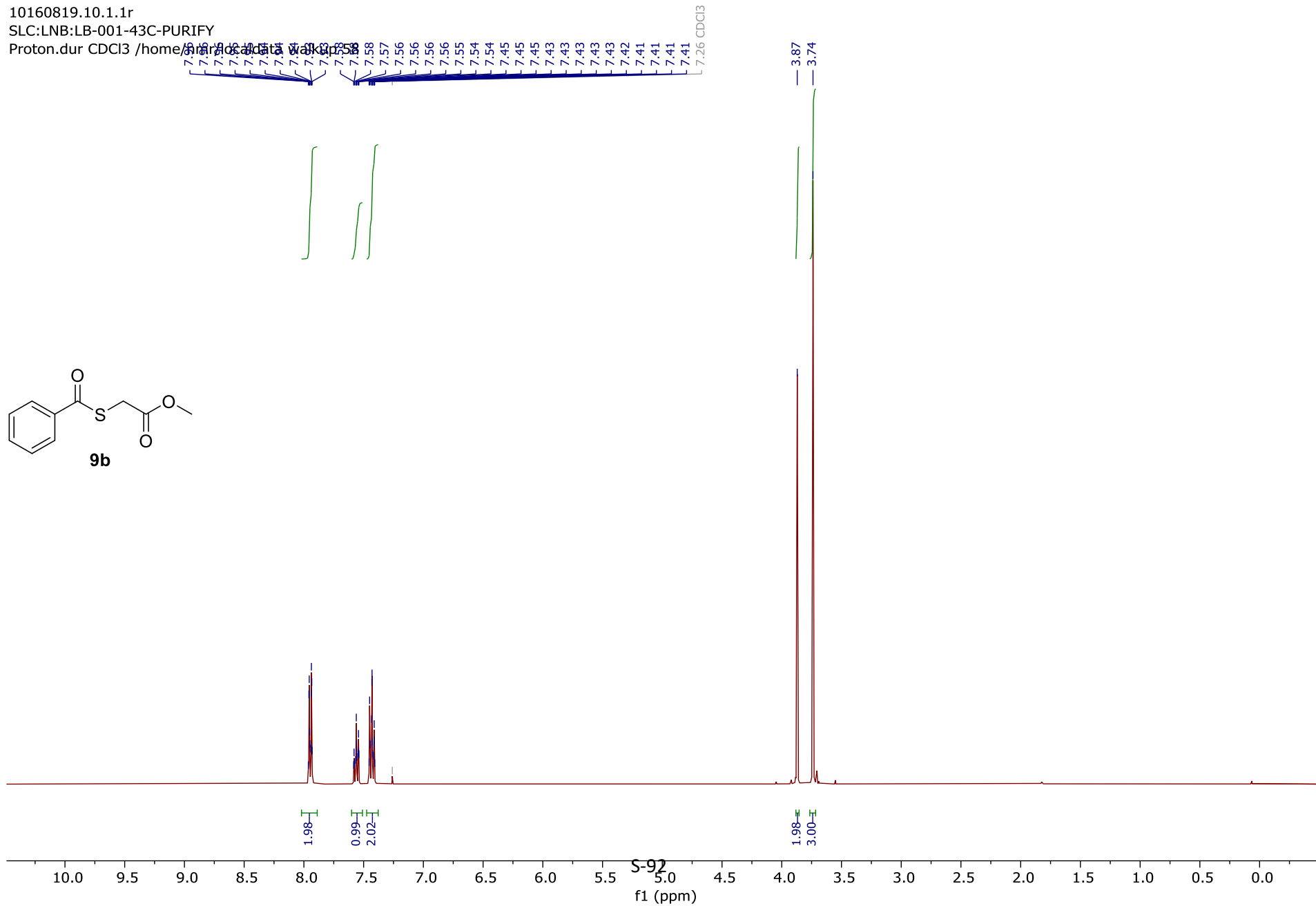
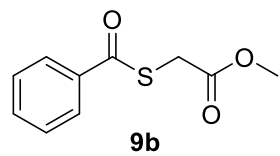
23133548.11.fid
SLC:LNB:LB-001-43B_PURIFY3
Carbon.dur CDCl3 / Home/nmr/localdata walkup 55



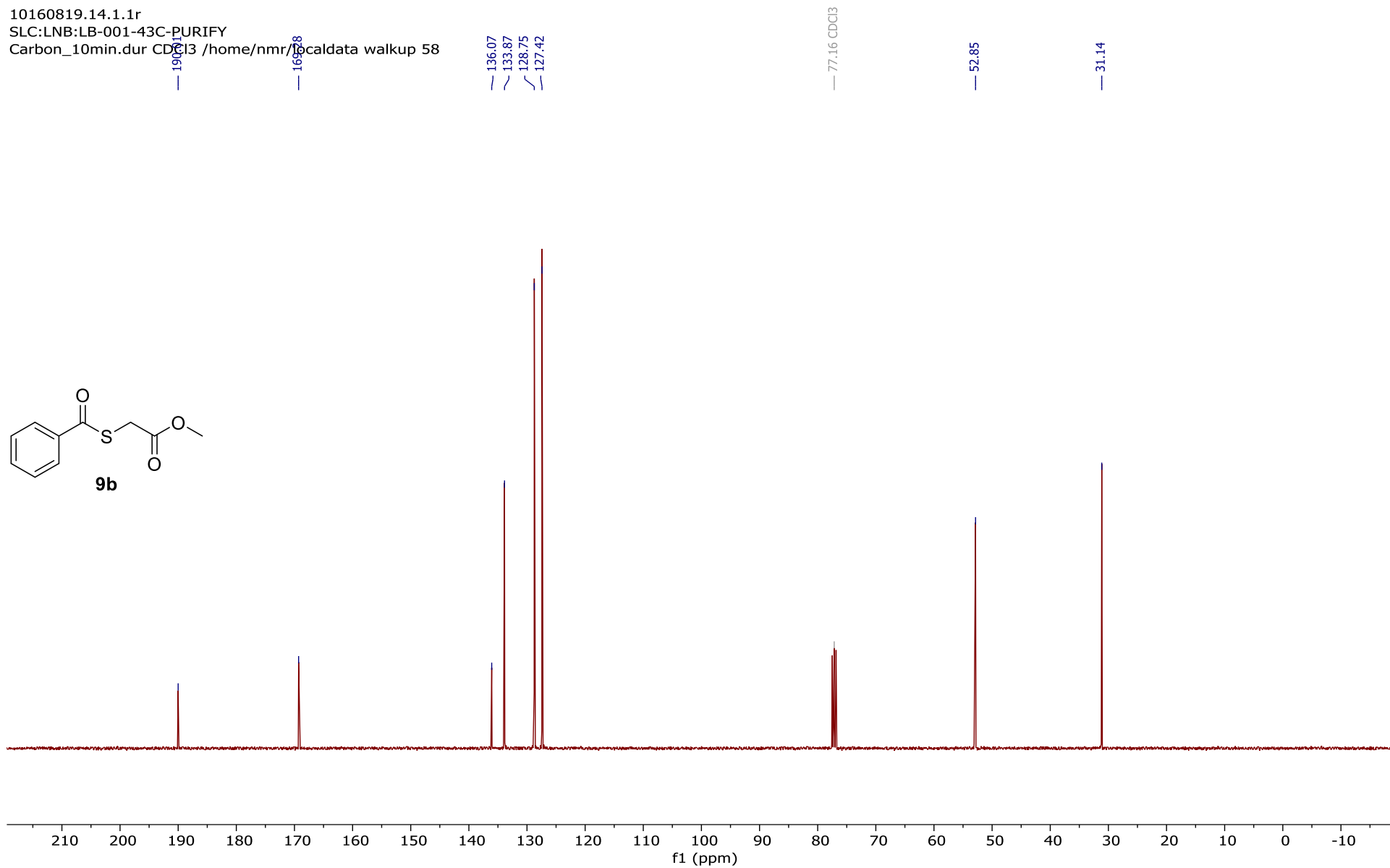
10160819.10.1.1r

SLC:LNB:LB-001-43C-PURIFY

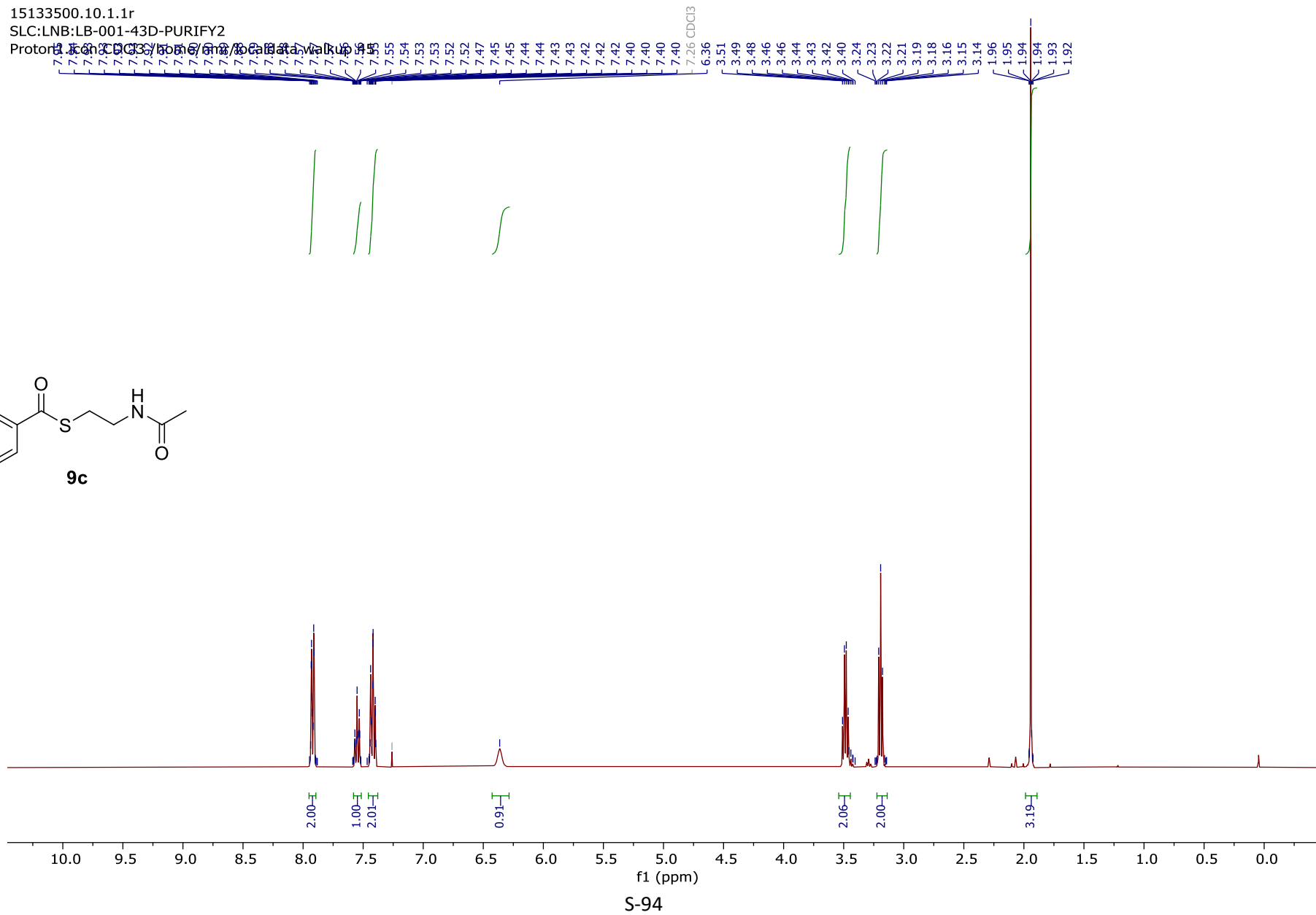
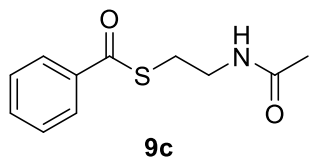
Proton.dur CDCl3 /home/.../.../...



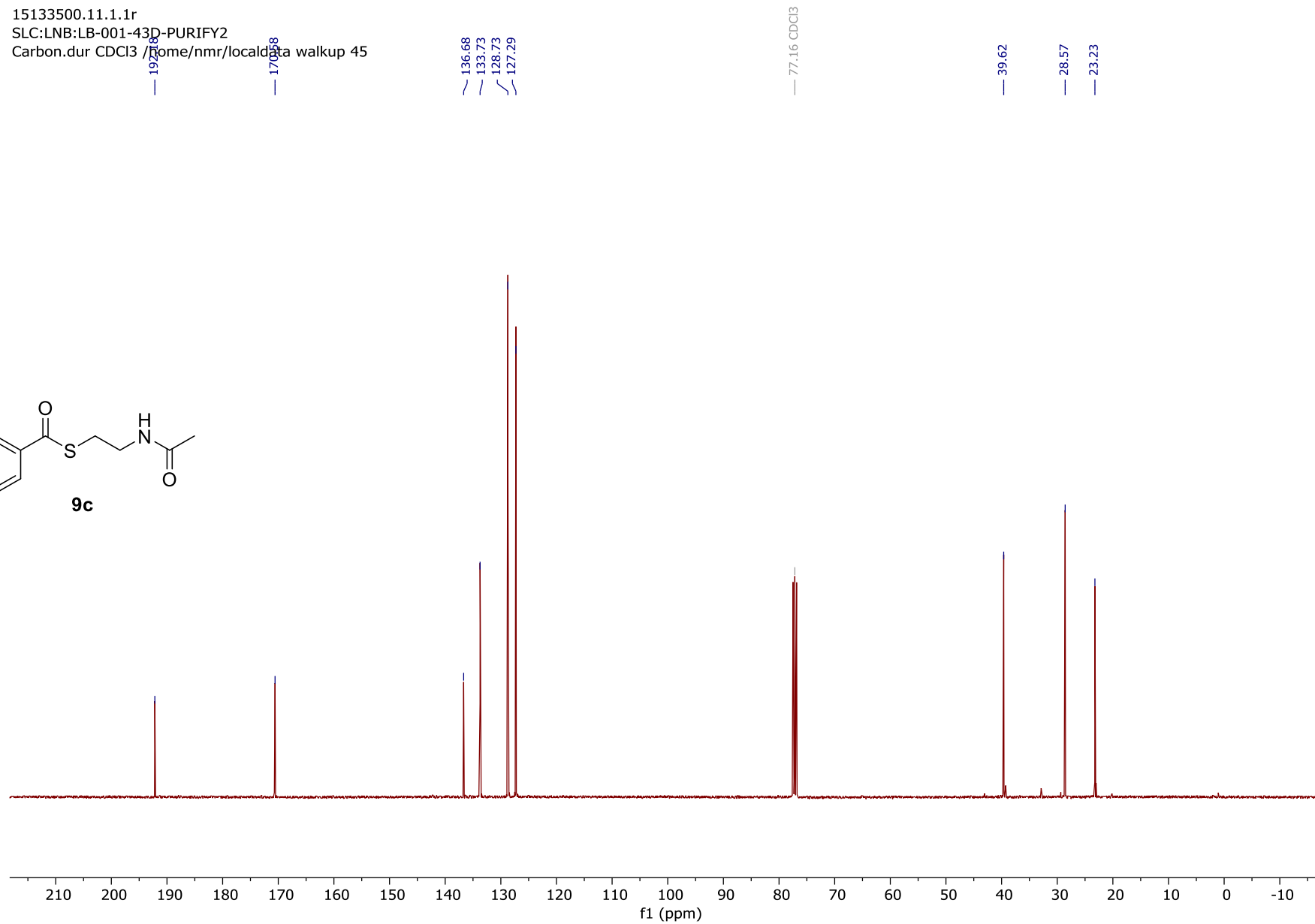
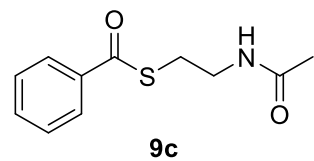
10160819.14.1.1r
SLC:LNB:LB-001-43C-PURIFY
Carbon_10min.dur CDCl3 /home/nmr/localdata walkup 58



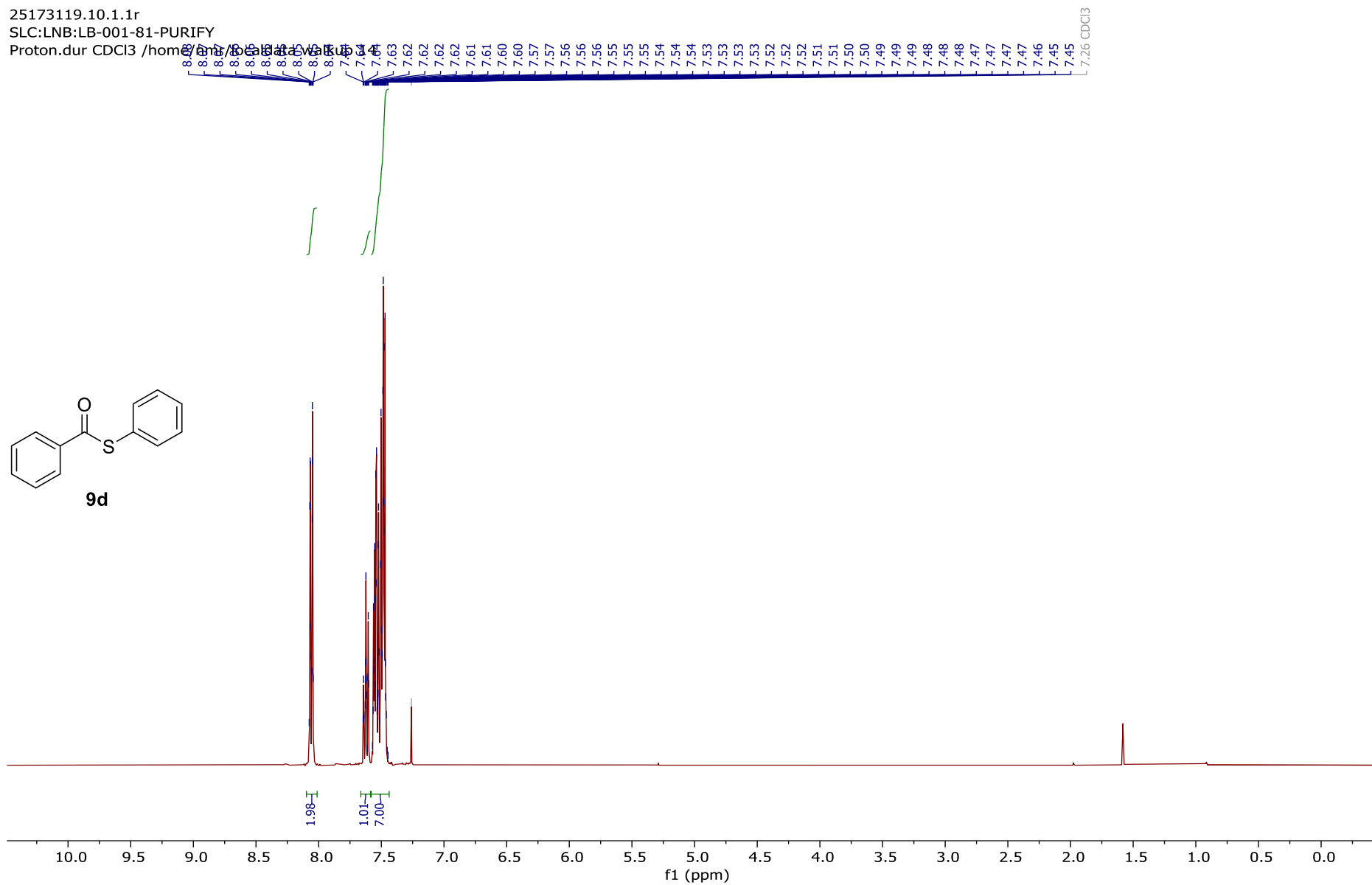
15133500.10.1.1r
SLC:LNB:LB-001-43D-PURIFY2
Proton 1 in CDCl3



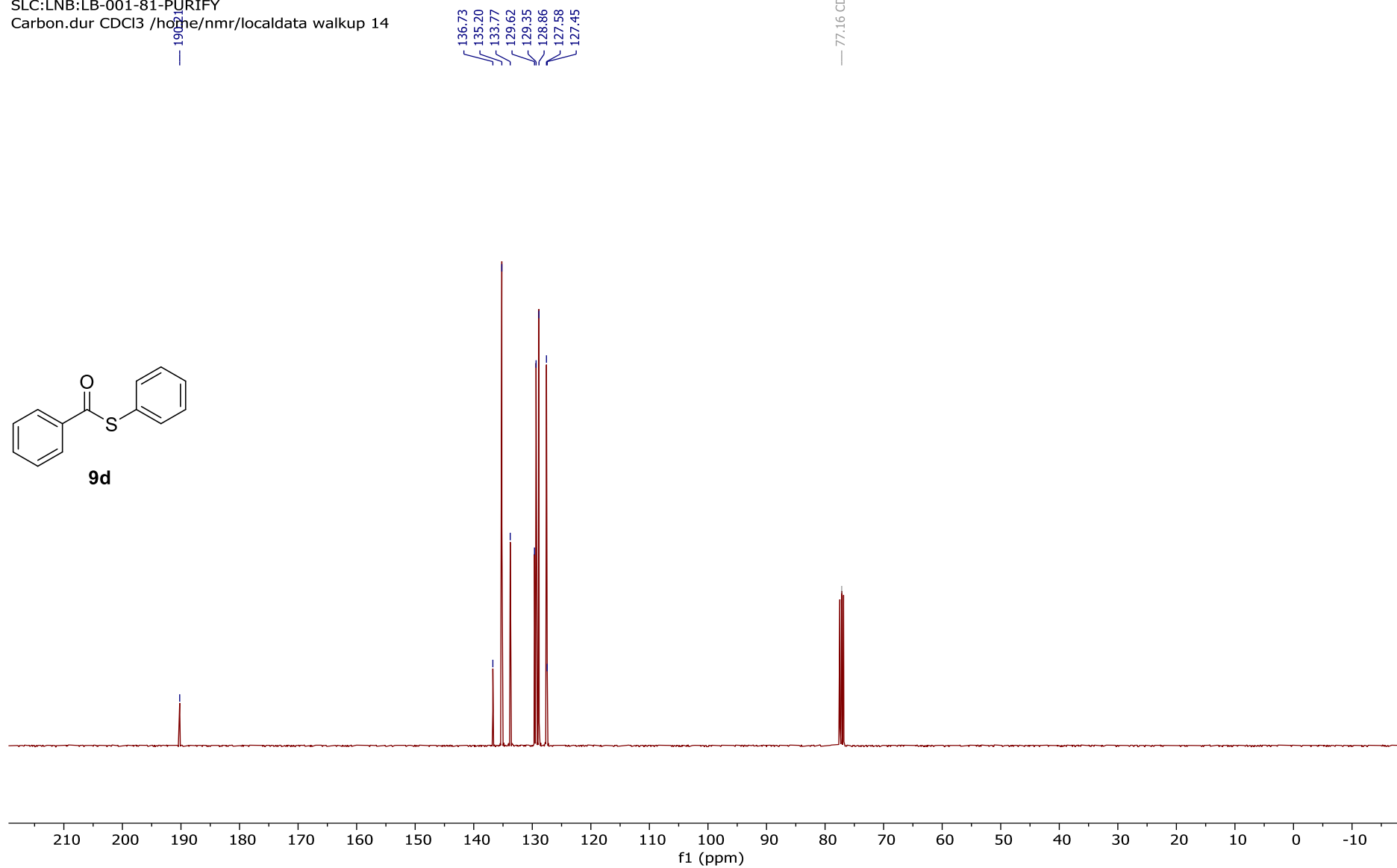
15133500.11.1.1r
SLC:LNB:LB-001-43D-PURIFY2
Carbon.dur CDCl3 // Home/nmr/localdata walkup 45



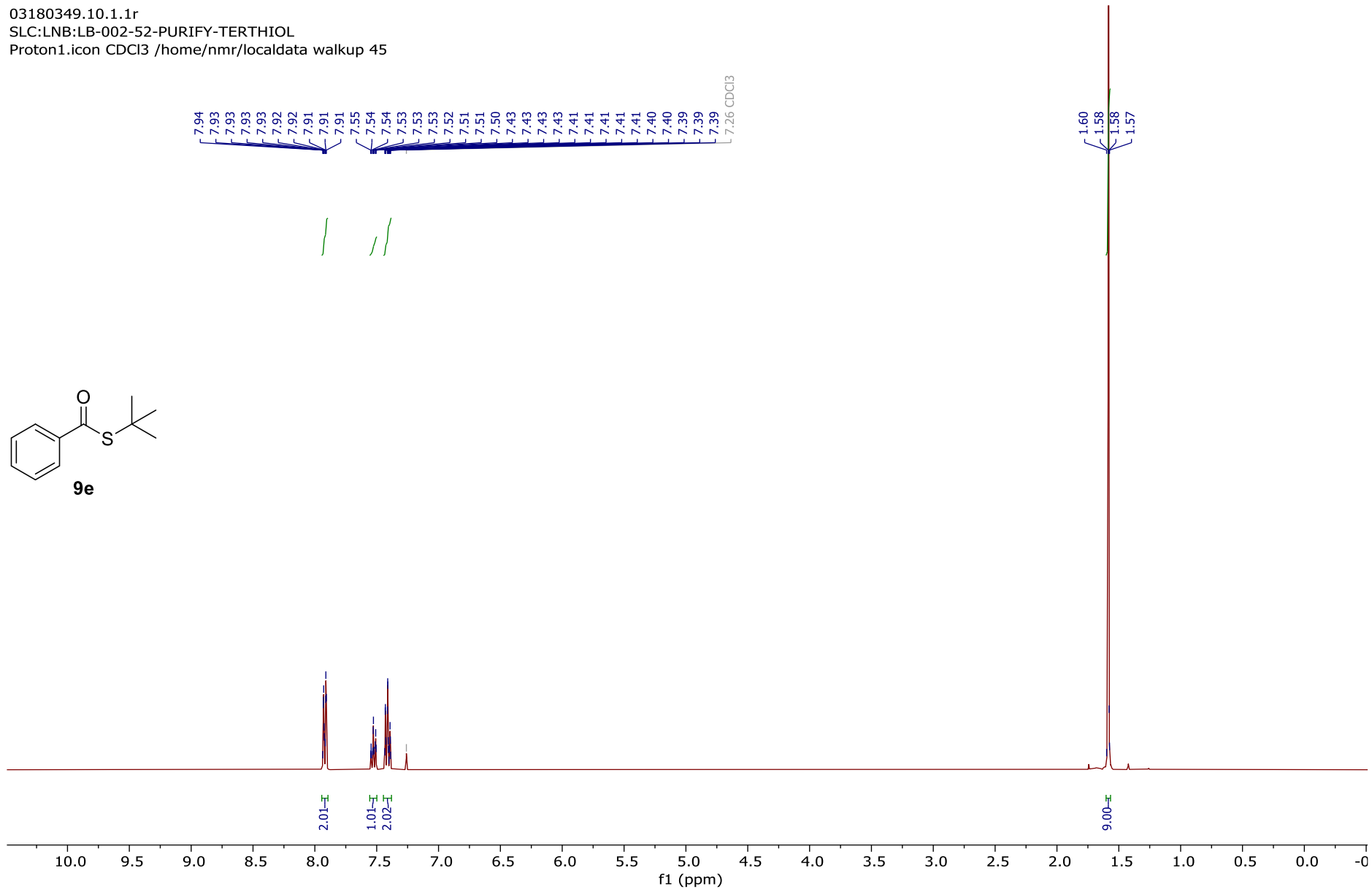
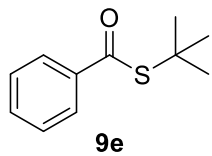
25173119.10.1.1r
SLC:LNB:LB-001-81-PURIFY
Proton.dur CDCl3 /hom



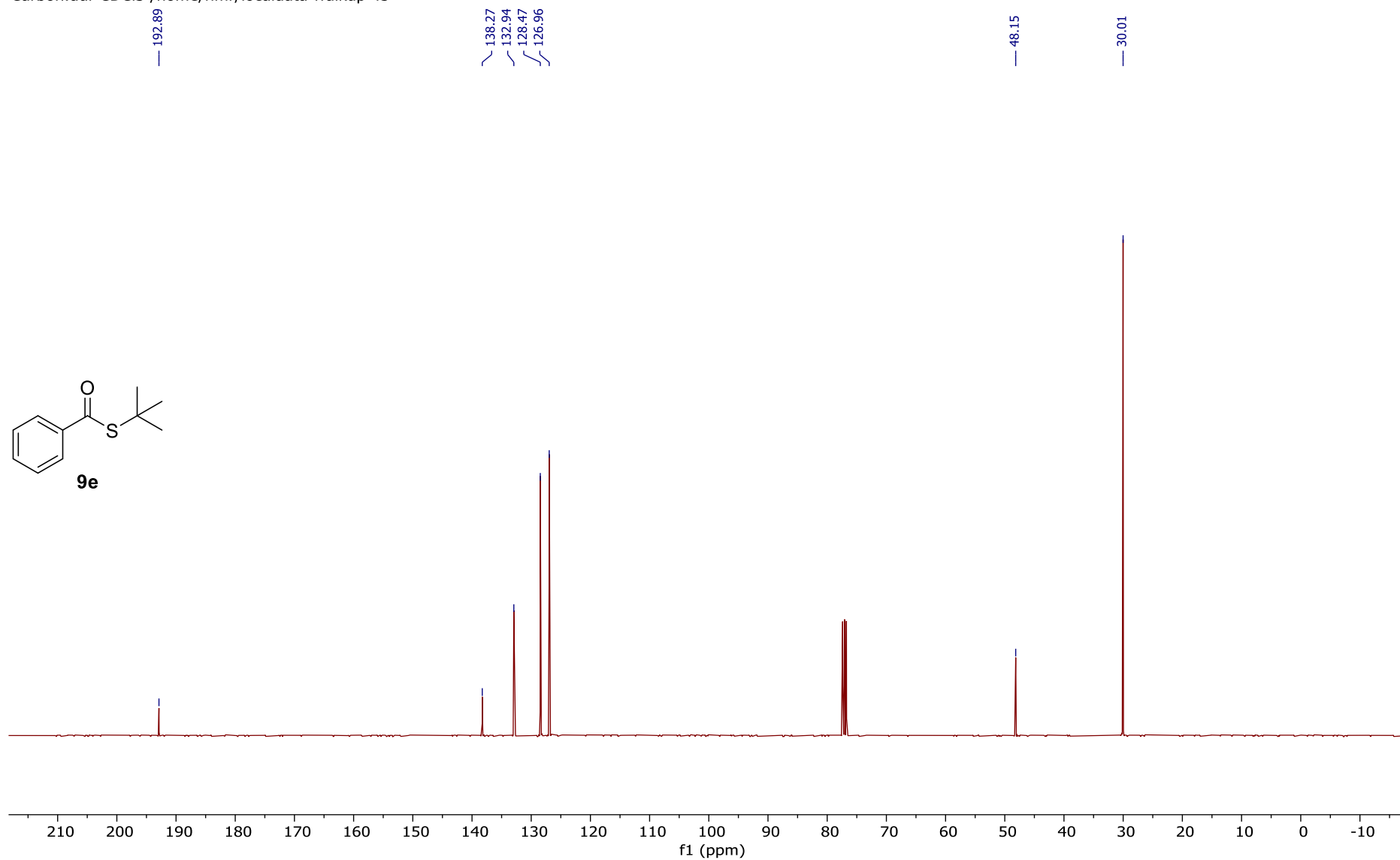
25173119.11.1.1r
SLC:LNB:LB-001-81-PURIFY
Carbon.dur CDCl3 /home/nmr/localdata walkup 14



03180349.10.1.1r
SLC:LNB:LB-002-52-PURIFY-TERTHIOL
Proton1.icon CDCl3 /home/nmr/localdata walkup 45

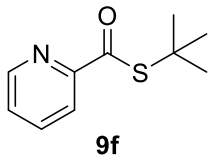
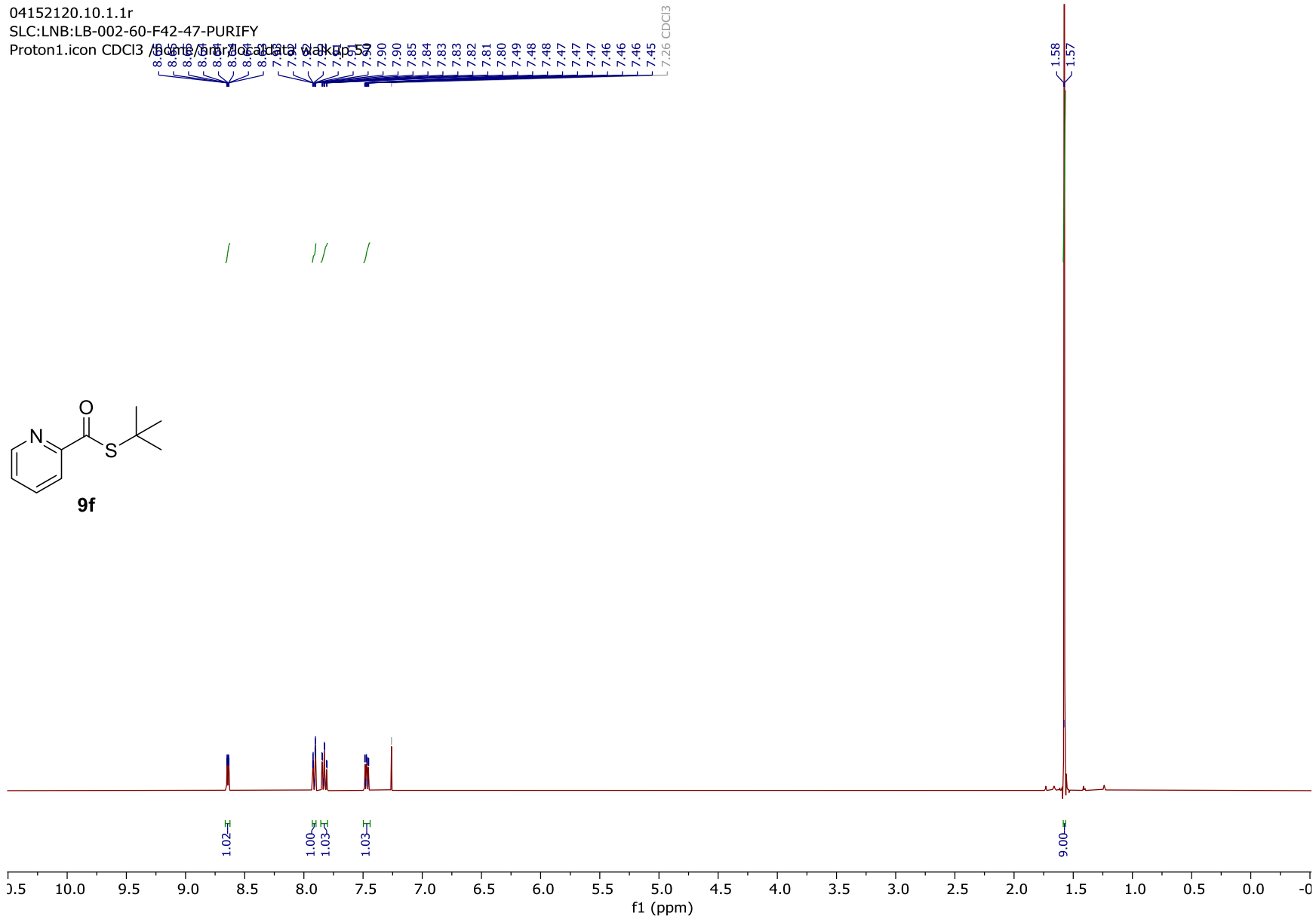


03180349.11.1.1r
SLC:LNB:LB-002-52-PURIFY-TERTHIOL
Carbon.dur CDCl3 /home/nmr/localdata walkup 45



S-99

04152120.10.1.1r
SLC:LNB:LB-002-60-F42-47-PURIFY
Proton1.icon CDCl3



CARBON_01
SLC:LNB:LB-002-60

— 193.79

— 153.27

— 148.98

— 137.35

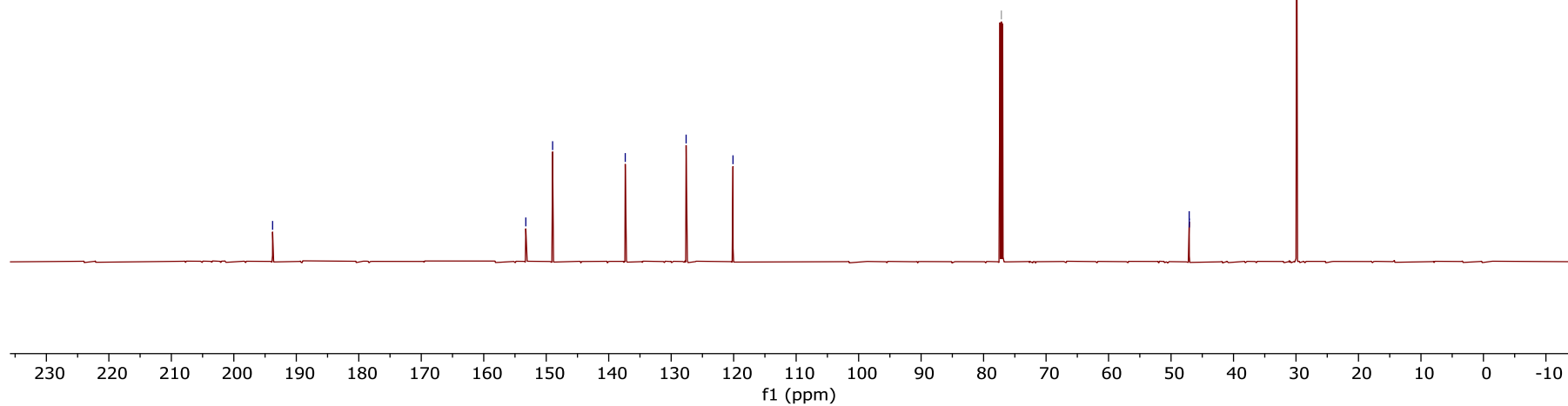
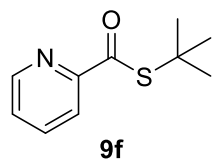
— 127.61

— 120.11

— 77.16 CDCl₃

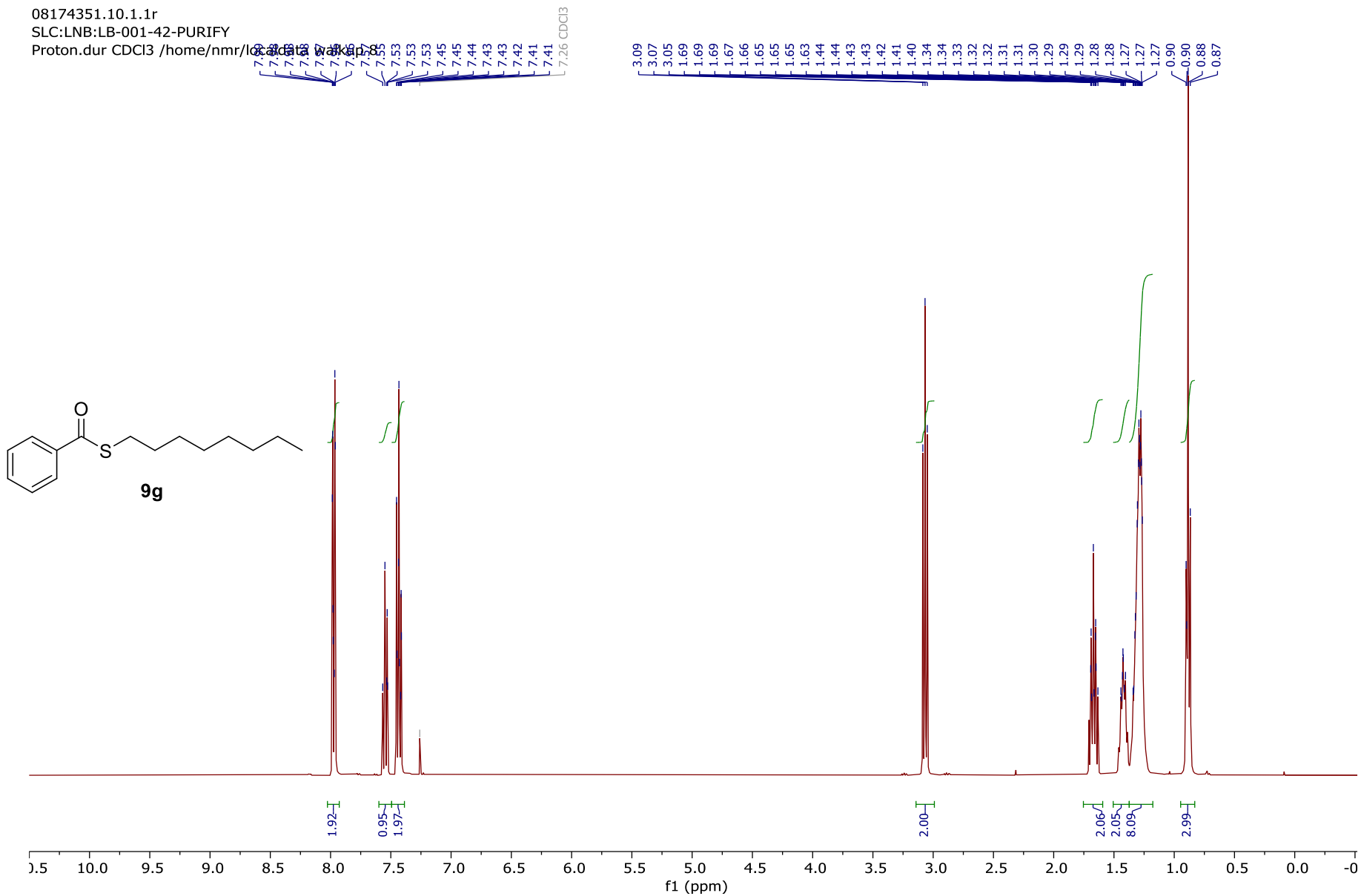
— 47.09
— 47.09

— 29.89

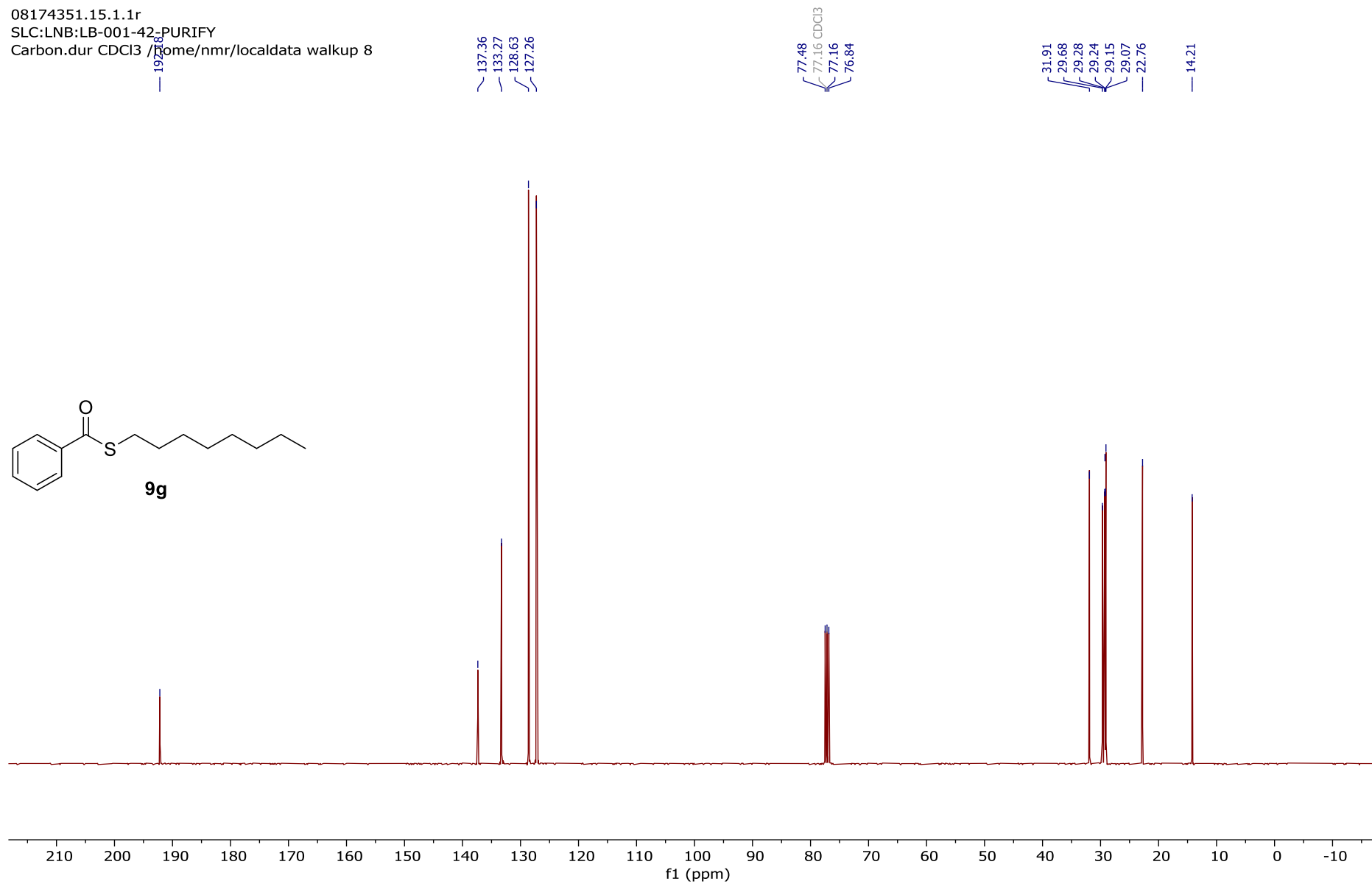
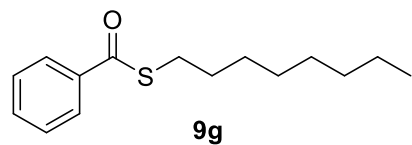


S-101

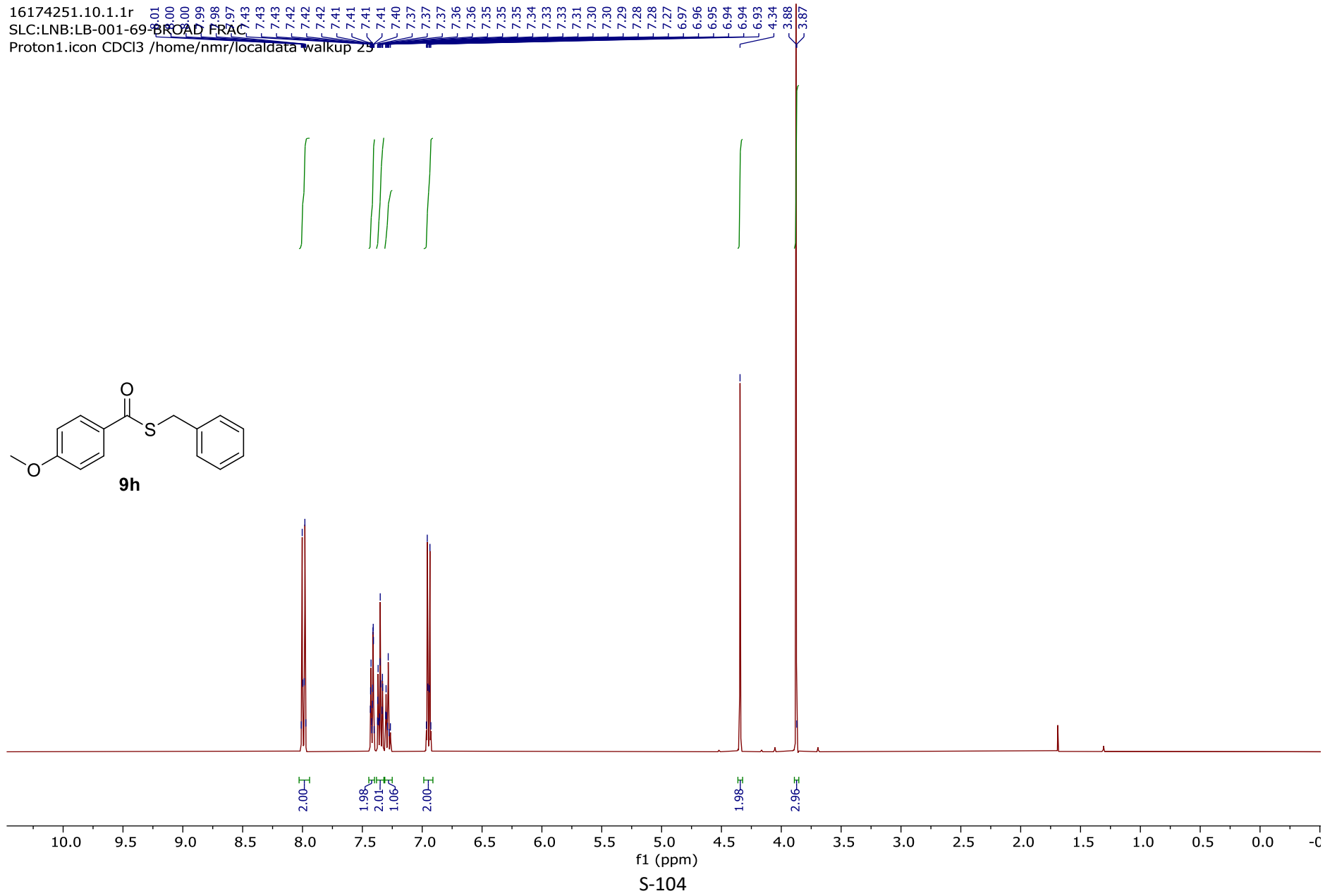
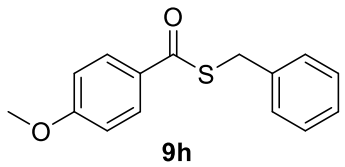
08174351.10.1.1r
SLC:LNB:LB-001-42-PURIFY
Proton.dur CDCl3 /home/nmr/



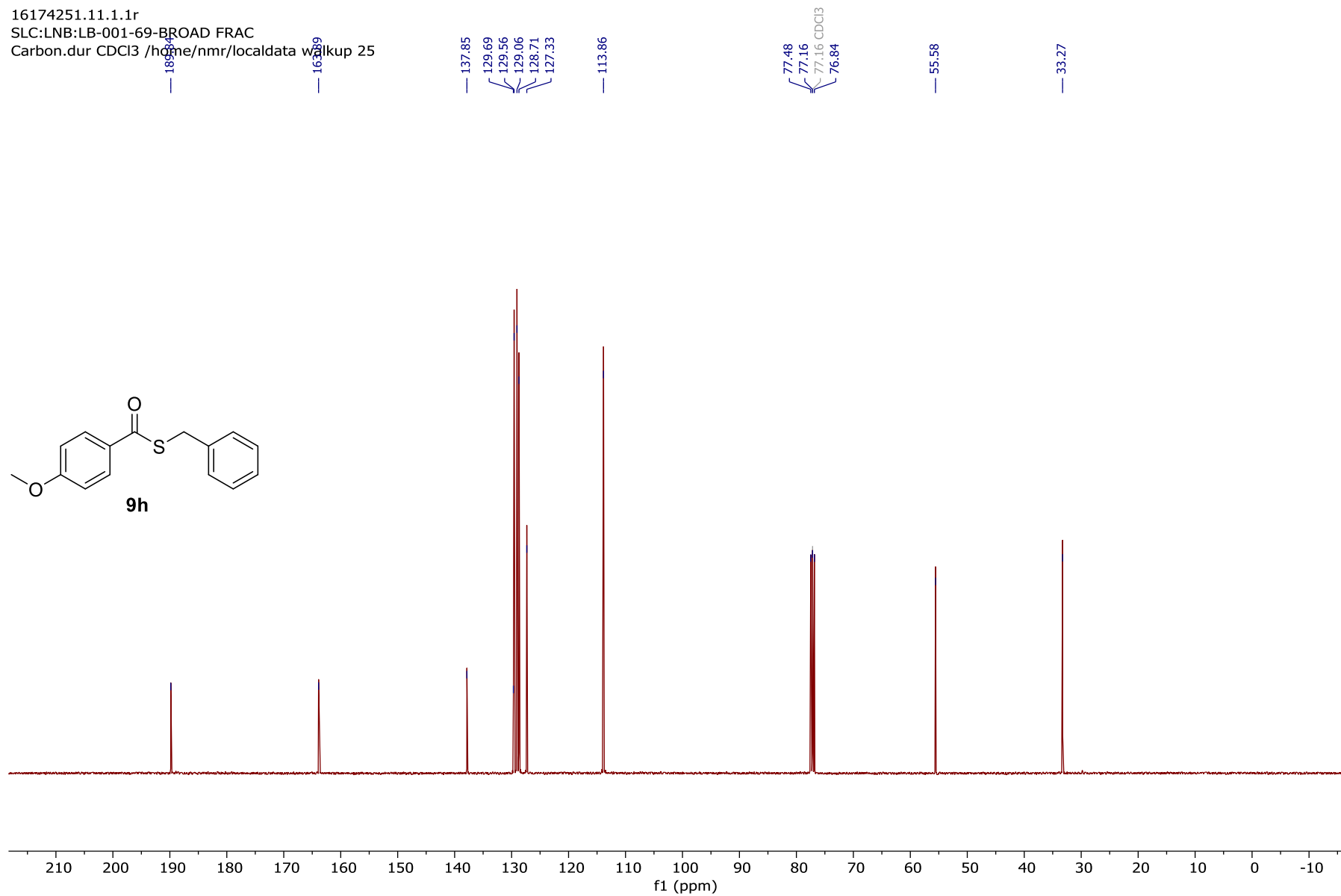
08174351.15.1.1r
SLC:LNB:LB-001-42-PURIFY
Carbon.dur CDCl3 / Home/nmr/localdata walkup 8



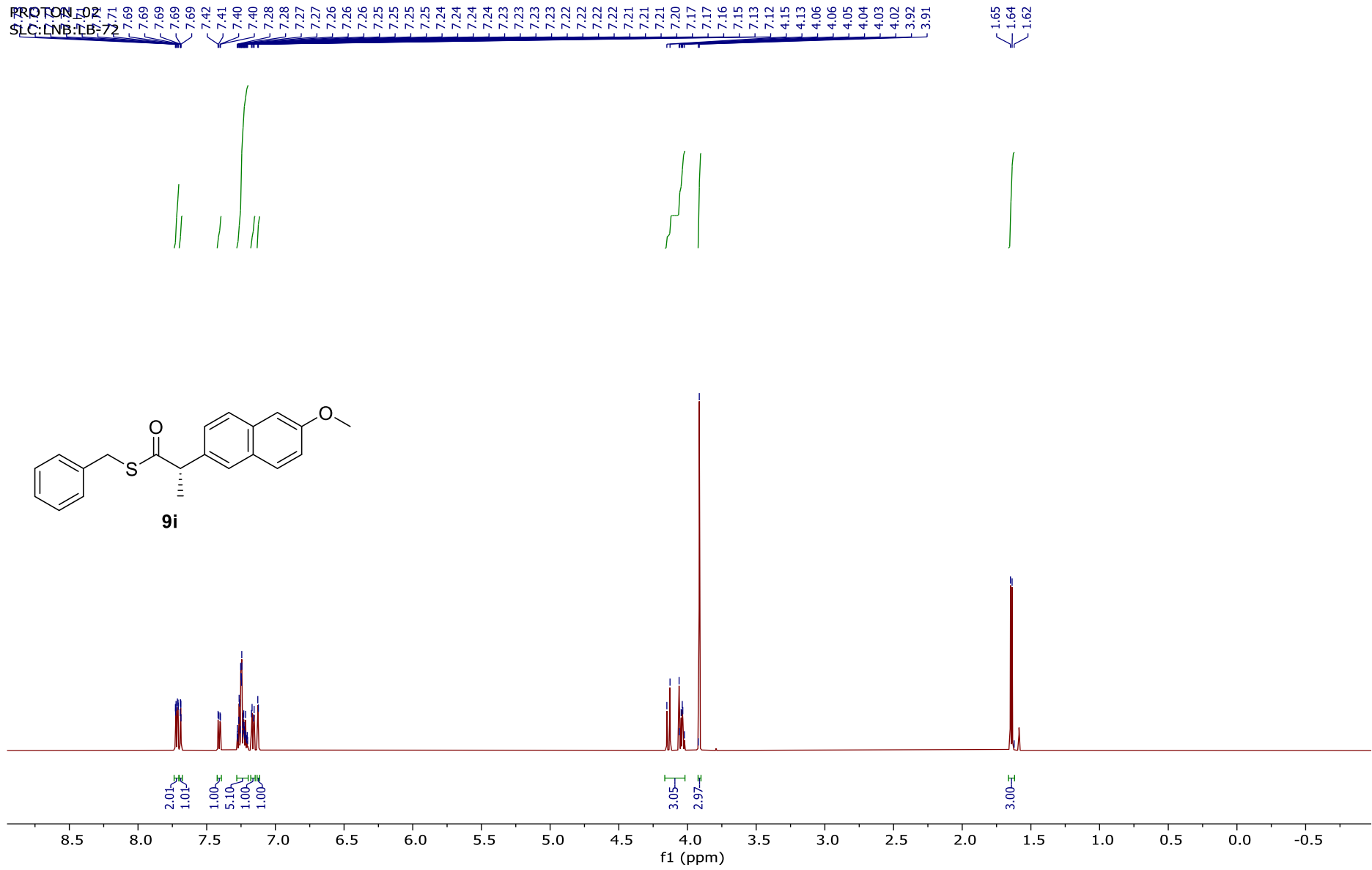
16174251.10.1.1r
SLC:LNB:LB-001-69-BROAD FRAC
Proton1.icon CDCl3 /home/nmr/localdata/walkup 25



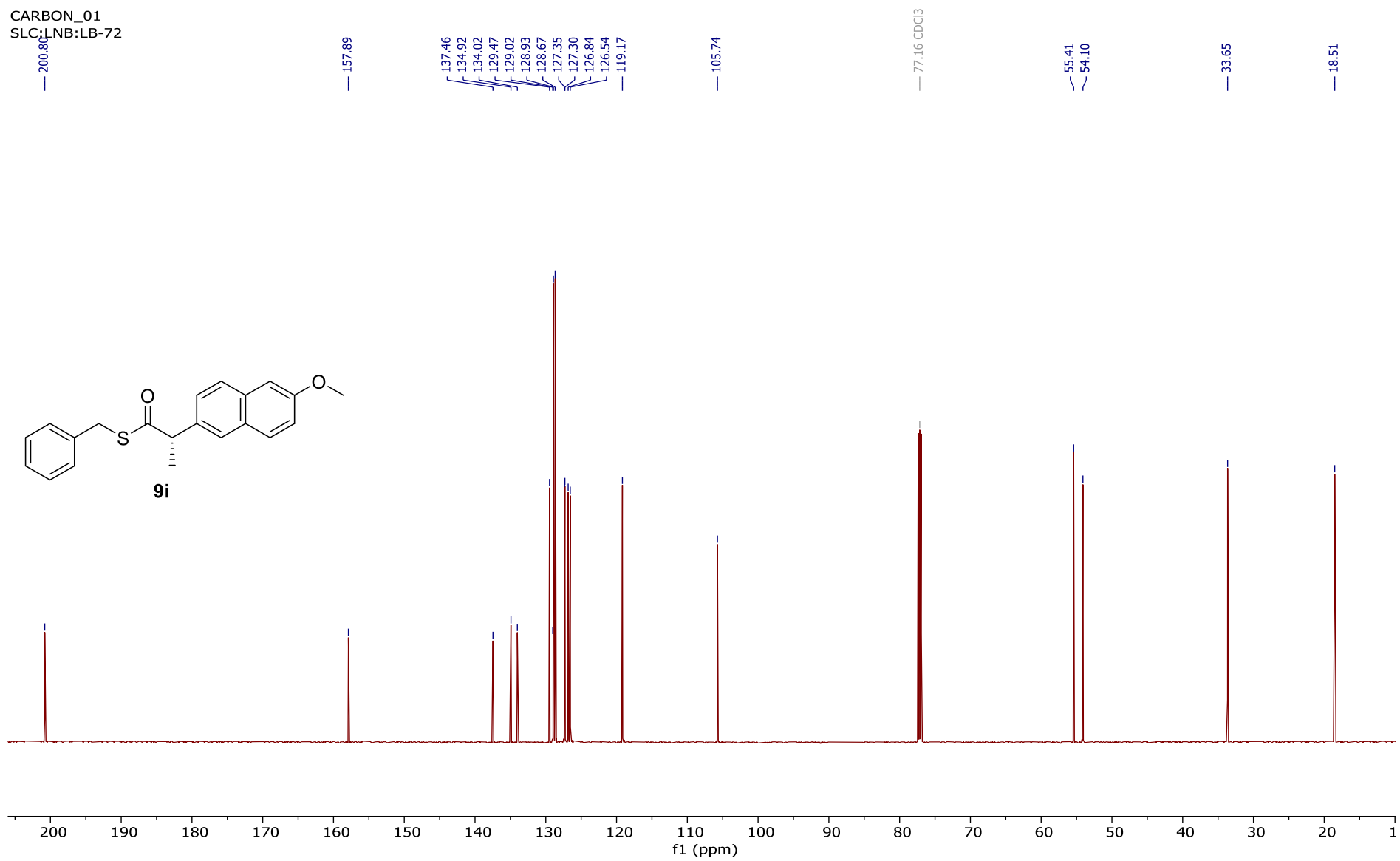
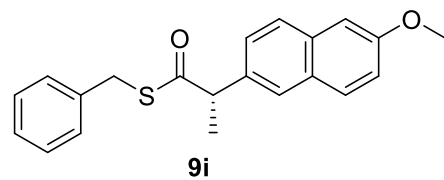
16174251.11.1.1r
SLC:LNB:LB-001-69-BROAD FRAC
Carbon.dur CDCl3 /home/nmr/localdata/walkup 25

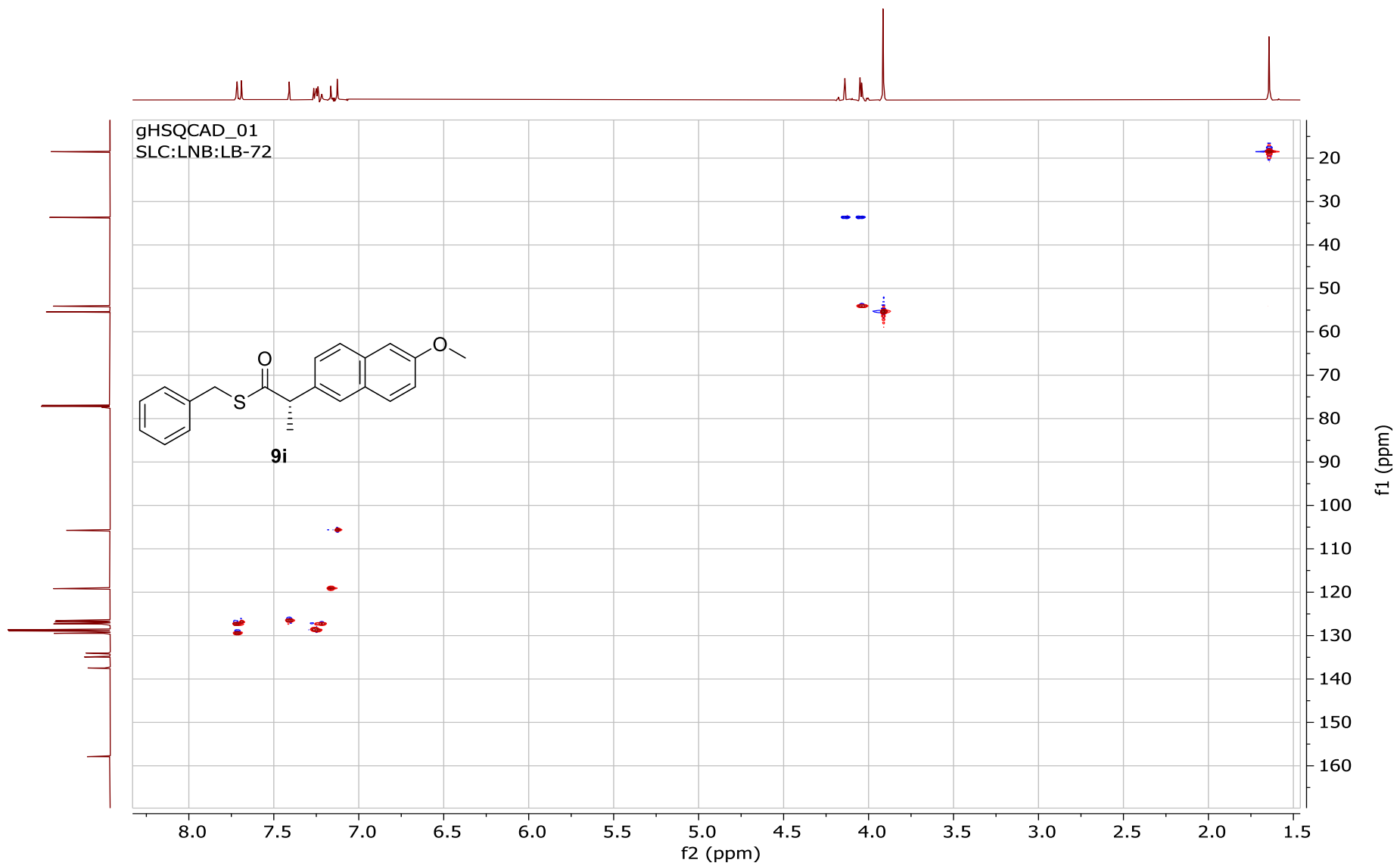


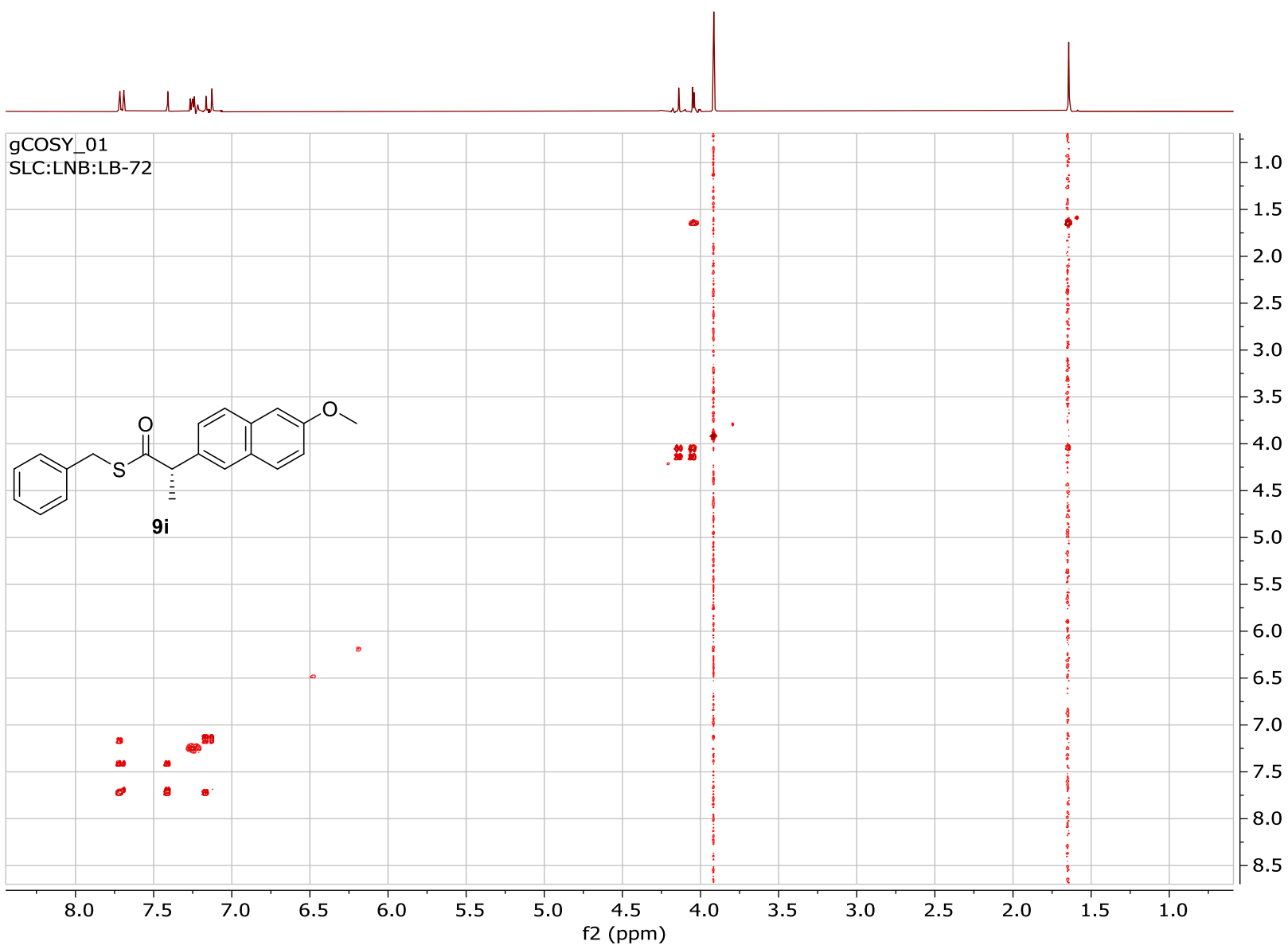
PROTON 10
SLC: LNB: LB: 72



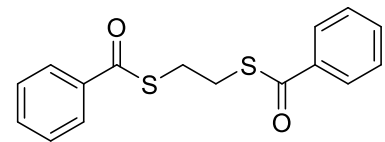
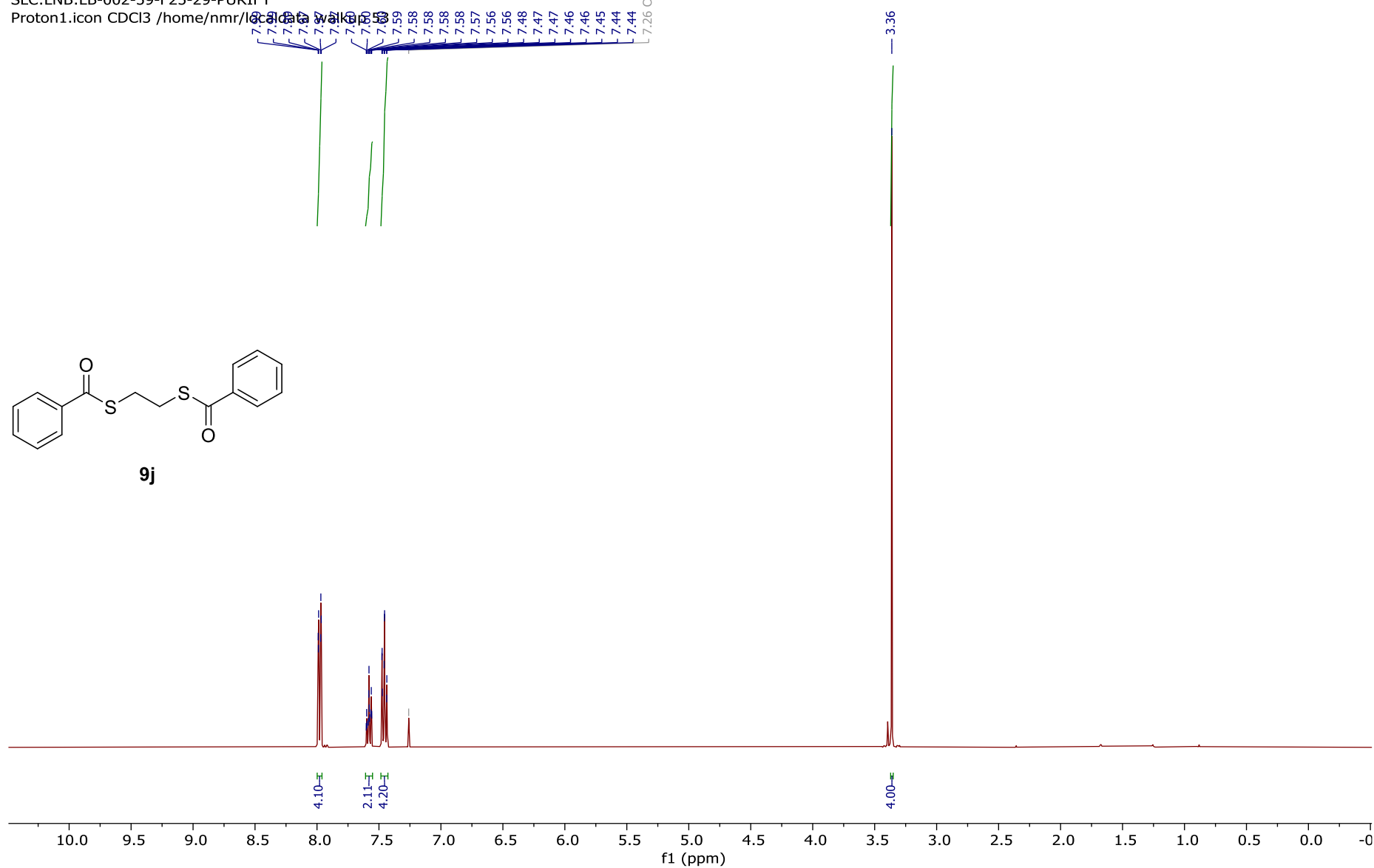
CARBON_01
SLC_LNB:LB-72







04151100.10.1.1r
SLC:LNB:LB-002-59-F25-29-PURIFY
Proton1.icon CDCl3 /home/nmr/10251100.10.1.1r



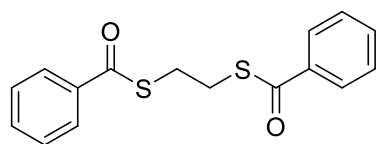
9j

04151100.11.1.1r
SLC:LNB:LB-002-59-F25-29-PURIFY
Carbon.dur CDCl3 /home/nmr/localdata walkup 53

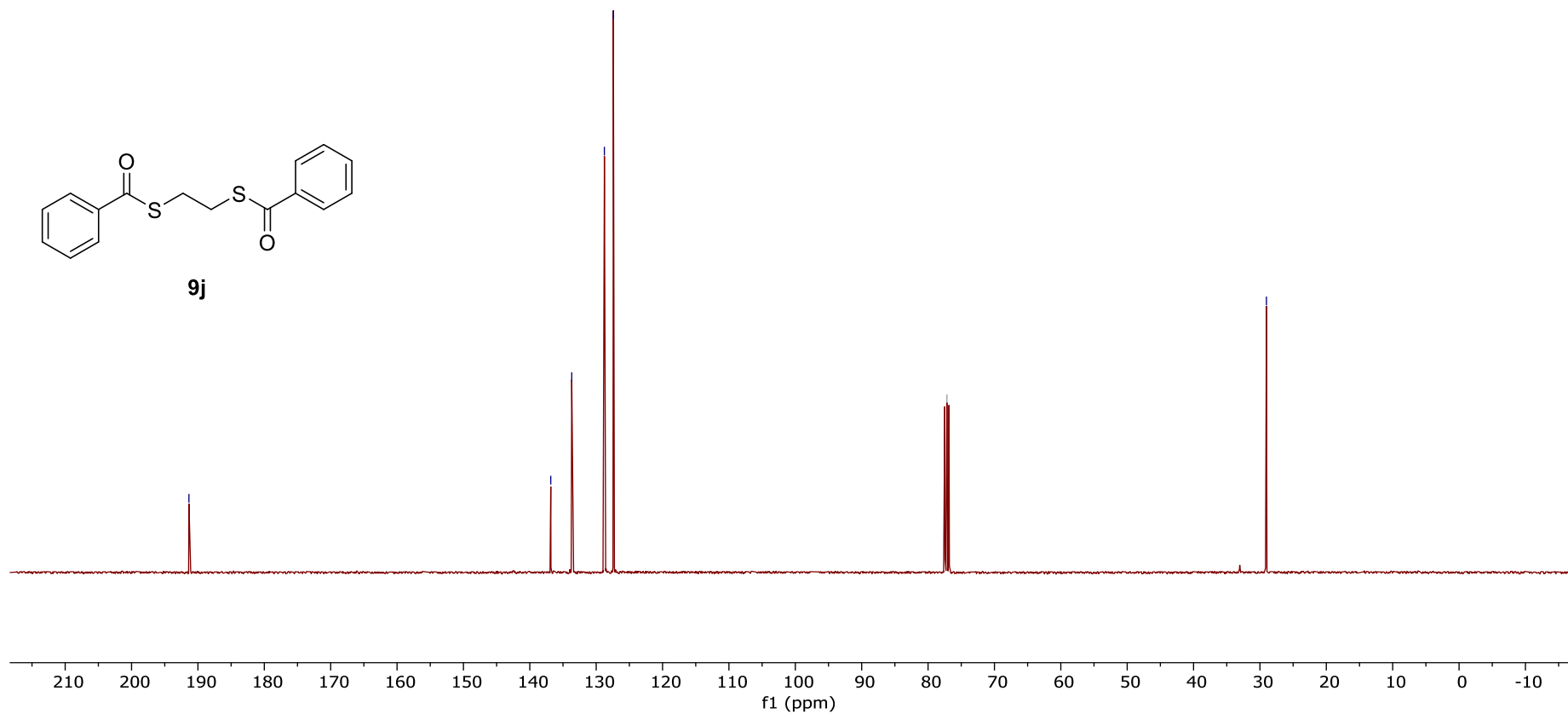
136.84
133.68
128.76
127.41

77.16 CDCl3

29.02



9j



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