

## Supporting Information

# Carboxylic Acid Deoxyfluorination and One-pot Amide Bond Formation using Pentafluoropyridine (PFP)

William D. G. Brittain\*<sup>a</sup> and Steven L. Cobb\*<sup>a</sup>

<sup>a</sup>*Department of Chemistry, Durham University, South Road, Durham DH1 3LE, United Kingdom*

Email: [william.d.brittain@durham.ac.uk](mailto:william.d.brittain@durham.ac.uk), [s.l.cobb@durham.ac.uk](mailto:s.l.cobb@durham.ac.uk)

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### General

All starting materials and reagents were bought from commercial sources and used as received. All reagents were purchased from Sigma Aldrich or Fluorochem and used as received. All reactions apart from where noted were carried out in air. MeCN was dried over activated 4 Å molecular sieves which had been dried under vacuum at 150 °C for 3 h. All flash column chromatography was carried out using silica purchased from Fluorochem using the solvent system noted. <sup>1</sup>H NMR spectra were recorded at 400 MHz using a Bruker Avance III spectrometer. <sup>13</sup>C NMR spectra were recorded at 101 MHz using a Bruker Avance III spectrometer. <sup>19</sup>F 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; CHCl<sub>3</sub> (<sup>1</sup>H 7.26 ppm, <sup>13</sup>C 77.0 ppm) and CH<sub>3</sub>CN (<sup>1</sup>H 1.96 ppm,

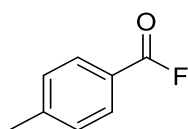
<sup>13</sup>C 118.3). Mass spectra were collected using ESI-LC in MeCN using a Waters TQD mass spectrometer with a Acquity UPLC BEH C18 1.7 μm (2.1 mm x 50 mm). ESI-LC was collected using water containing formic acid (0.1% v/v) and MeCN mixture in a 95:5 to 5:95 gradient over 5 min.

## General Method for the Synthesis of Acyl Fluorides

To a solution of carboxylic acid (1 equiv.) in MeCN was added pentafluoropyridine (1 equiv.) and DIPEA (1 equiv.) and the reaction mixture stirred at room temperature for 16 h. Following this time the reaction mixture was concentrated under reduced pressure and the residue purified directly via either flash column chromatography or by passing through a silica plug (3 cm wide by 3 cm deep). The solution was then concentrated under reduced pressure to yield the desired acyl fluorides.

*NOTE: Acyl fluorides are prone to hydrolysis over time thus we suggest that purification of the reaction mixtures takes place as quickly as possible following the end of the reaction period. Any hydrolysed materials may be repurified by passing through a second silica plug. Also, some acyl fluorides are known to be unstable on silica and thus we suggest using the in-situ generation presented below if there are stability problems with substrates.*

## 4-methylbenzoyl fluoride 2a



Synthesised according to the general method for the synthesis of acyl fluorides from 4-methylbenzoic acid (0.25 g, 1.84 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% toluene) to give the desired product as a clear oil in 64% yield (0.162 g).

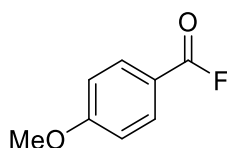
Characterisation data were consistent with the previously reported literature values.<sup>[1]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.96 (d, *J* = 8.2, 2H), 7.35 (d, *J* = 8.2, 2H), 2.48 (s, 4H).

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  17.44 (s, 1F).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  157.6 (d,  $J = 342.8$ ), 146.6, 131.5 (d,  $J = 4.0$ ), 129.8 (d,  $J = 1.3$ ), 122.1 (d,  $J = 60.9$ ), 21.9.

### 4-methoxybenzoyl fluoride 2b



Synthesised according to the general method for the synthesis of acyl fluorides from 4-methoxybenzoic acid (0.25 g, 1.64 mmol). The crude material was purified by flash column chromatography (100% hexane to 90% hexane 10% toluene) to give the desired product as a clear oil in 73% yield (0.185 g).

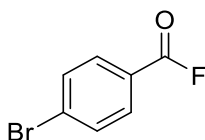
Characterisation data were consistent with the previously reported literature values.<sup>[2]</sup>

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.02 (d,  $J = 8.7$ , 2H), 7.01 (dd,  $J = 8.7, 1.2$ , 2H), 3.92 (s, 3H).

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  15.95 (s, 1F).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  165.2, 157.3 (d,  $J = 339.9$ ), 133.8 (d,  $J = 4.2$ ), 116.8 (d,  $J = 61.6$ ), 114.4 (d,  $J = 1.4$ ), 55.7.

### 4-bromobenzoyl fluoride 2c



Synthesised according to the general method for the synthesis of acyl fluorides from 4-bromobenzoic acid (0.25 g, 1.24 mmol). The crude material was purified by passing the crude material through a silica

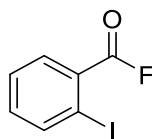
plug (2 cm × 3 cm) and washing with hexane (25 mL) to give the desired product as a white solid in 52% yield (0.128 g).

Characterisation data were consistent with the previously reported literature values.<sup>[3]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.93 (d, *J* = 8.5, 2H), 7.71 (dd, *J* = 8.5, 1.2, 3H).

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ 18.44 (s, 1F).

## 2-iodobenzoyl fluoride 2d



Synthesised according to the general method for the synthesis of acyl fluorides from 2-iodobenzoic acid (0.25 g, 1.04 mmol). The crude material was purified by passing the crude material through a silica plug (2 cm × 3 cm) and washing with hexane (25 mL) to give the desired product as a white solid in 49% yield (0.127 g).

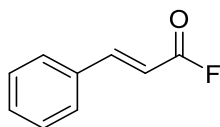
Characterisation data were consistent with the previously reported literature values.<sup>[1]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.16 (dt, *J* = 7.9, 1.2, 1H), 8.06 (dd, *J* = 7.9, 1.7, 1H), 7.53 (td, *J* = 7.7, 1.2, 1H), 7.33 (td, *J* = 7.7, 1.7, 1H).

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ 28.63 (s, 1F).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 155.3 (d, *J* = 345.0), 142.7 (d, *J* = 3.9), 135.2, 133.5 (d, *J* = 2.0), 128.4.

## cinnamoyl fluoride 2e



Synthesised according to the general method for the synthesis of acyl fluorides from *trans*-cinnamic acid (0.25 g, 1.68 mmol). The crude material was purified by passing the crude material through a silica plug (2 cm × 3 cm) and washing with hexane (25 mL) to give the desired product as a low melting point solid in 29% yield (0.074 g).

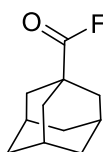
Characterisation data were consistent with the previously reported literature values.<sup>[1]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.87 (d, *J* = 16.0, 1H), 7.62 – 7.57 (m, 2H), 7.51 – 7.43 (m, 3H), 6.40 (dd, *J* = 16.0, 7.4, 1H).

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ 25.59 (d, *J* = 7.4, 1F).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 157.1 (d, *J* = 338.8), 151.5 (d, *J* = 6.3), 133.2, 131.9, 129.2, 128.8, 112.1 (d, *J* = 67.4).

### **(3r,5r,7r)-adamantane-1-carbonyl fluoride 2f**



Synthesised according to the general method for the synthesis of acyl fluorides from 1-adamantanecarboxylic acid (0.25 g, 1.39 mmol). The crude material was purified by passing the crude material through a silica plug (2 cm × 3 cm) and washing with hexane (25 mL) to give the desired product as a white solid in 85% yield (0.215 g).

*NOTE: A small amount of hydrolysis was observed following purification, leading to minor impurities in obtained spectra.*

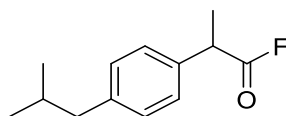
Characterisation data were consistent with the previously reported literature values.<sup>[1]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 2.11 – 2.04 (m, 3H), 2.00 – 1.93 (m, 6H), 1.82 – 1.69 (m, 6H).

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ 23.77 (s, 1F).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 167.0 (d, *J* = 371.6), 40.5 (d, *J* = 44.6), 37.9, 36.1, 27.4.

## 2-(4-isobutylphenyl)propanoyl fluoride 2g



Synthesised according to the general method for the synthesis of acyl fluorides from (±)-2-(4-Isobutylphenyl)propanoic acid (0.25 g, 1.21 mmol). The crude material was purified by passing the crude material through a silica plug (2 cm × 3 cm) and washing with a 1:1 mixture of hexane:DCM (20 mL) to give the desired product as a yellow oil in 93% yield (0.232 g).

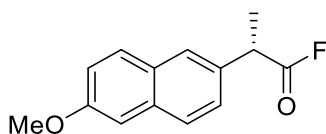
Characterisation data were consistent with the previously reported literature values.<sup>[3]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.27 – 7.07 (m, 4H), 3.87 (q, *J* = 7.1), 2.49 (d, *J* = 7.2, 2H), 1.97 – 1.78 (m, 1H), 1.61 (dd, *J* = 7.2, 0.9, 3H), 0.93 (d, *J* = 6.6, 6H).

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ 39.28 (s, 1F).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 164.5 (d, *J* = 367.1), 134.6, 129.8, 127.2, 45.0, 43.9 (d, *J* = 49.3), 30.2, 22.4, 18.1 (d, *J* = 1.4).

## (S)-2-(6-methoxynaphthalen-2-yl)propanoyl fluoride 2h



Synthesised according to the general method for the synthesis of acyl fluorides from (S)-(+)-2-(6-methoxy-2-naphthyl)propionic acid (0.25 g, 1.08 mmol). The crude material was purified by passing the crude material through a silica plug (2 cm × 3 cm) and washing with a 1:1 mixture of hexane:DCM (20 mL) to give the desired product as a white solid in 94% yield (0.236 g).

Characterisation data were consistent with the previously reported literature values.<sup>[3]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.81 – 7.70 (m, 3H), 7.40 (dd, *J* = 8.5, 1.9, 1H), 7.22 (dd, *J* = 8.9, 2.6, 1H), 7.17 (d, *J* = 2.6, 1H), 4.03 (q, *J* = 7.2, 1H), 3.95 (s, 3H), 1.70 (dd, *J* = 7.2, 0.9, 3H).

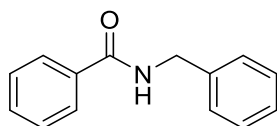
<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ 39.70 (s, 1F).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 164.6 (d, *J* = 367.3), 158.0, 134.1, 132.4, 129.4, 128.9, 127.8, 126.1 (d, *J* = 73.3), 119.5, 105.7, 55.4, 44.2 (d, *J* = 49.3), 18.1.

## General Method for the Synthesis of Amides and Esters *via in situ* Acyl Fluoride Generation

To a solution of carboxylic acid (1 equiv.) in MeCN was added pentafluoropyridine (1.1 equiv.) and DIPEA (2 equiv.) and the mixture was stirred for 30 min at rt. Following this the desired amine or alcohol (1 equiv.) was added and the reaction stirred for 16 h at rt. The reaction mixture was then concentrated under reduced pressure and the residue was directly purified using flash column chromatography to yield the respective amides or esters.

## *N*-benzylbenzamide 4a



Synthesised according to the general method for the synthesis of amides from benzoic acid (0.100 g, 0.82 mmol) and benzylamine (0.088 g, 0.82 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a white solid in 94% yield (0.155 g).

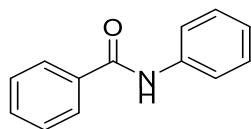
Characterisation data were consistent with the previously reported literature values.<sup>[4]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.84 – 7.80 (m, 2H), 7.55 – 7.49 (m, 1H), 7.47 – 7.41 (m, 2H), 7.39 – 7.36 (m, 4H), 7.35 – 7.30 (m, 1H), 6.59 (brs, 1H), 4.66 (d, *J* = 5.7, 2H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 167.5, 138.2, 134.4, 131.6, 128.8, 128.6, 128.0, 127.7, 127.0, 44.2.

LCMS (ESI<sup>+</sup>) *rt* = 2.0 min, *m/z* = 212.1 [M+H]<sup>+</sup>

## ***N*-phenylbenzamide 4b**



Synthesised according to the general method for the synthesis of amides from benzoic acid (0.100 g, 0.82 mmol) and aniline (0.076 g, 0.82 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a white solid in 83% yield (0.134 g).

Characterisation data were consistent with the previously reported literature values.<sup>[5]</sup>

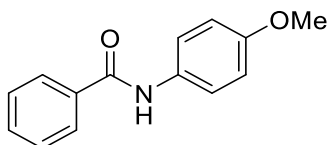
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.94 – 7.83 (m, 3H), 7.71 – 7.64 (m, 2H), 7.61 – 7.56 (m, 1H), 7.54 – 7.49 (m, 2H), 7.44 – 7.36 (m, 2H), 7.22 – 7.15 (m, 1H).



$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  165.7, 137.9, 135.0, 131.9, 129.1, 128.8, 127.0, 124.6, 120.2.

LCMS (ESI<sup>+</sup>)  $rt = 2.1$  min,  $m/z = 198.2$   $[\text{M}+\text{H}]^+$

### ***N*-(4-methoxyphenyl)benzamide 4c**



Synthesised according to the general method for the synthesis of amides from benzoic acid (0.100 g, 0.82 mmol) and 4-methoxyaniline (0.101 g, 0.82 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a colourless crystalline solid in 64% yield (0.118 g).

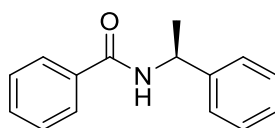
Characterisation data were consistent with the previously reported literature values.<sup>[6]</sup>

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.92 – 7.84 (m, 2H), 7.78 (s, 1H), 7.61 – 7.54 (m, 3H), 7.54 – 7.48 (m, 2H), 6.94 (appd,  $J = 9.0$ , 2H), 3.84 (s, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  156.7, 135.0, 131.8, 131.0, 128.8, 127.0, 122.1, 114.3, 55.5.

LCMS (ESI<sup>+</sup>)  $rt = 2.0$  min,  $m/z = 228.1$   $[\text{M}+\text{H}]^+$

### ***(S)*-*N*-(1-phenylethyl)benzamide 4d**



Synthesised according to the general method for the synthesis of amides from benzoic acid (0.100 g, 0.82 mmol) and (*R*)-(+)- $\alpha$ -methylbenzylamine (0.099 g, 0.82 mmol). The crude material was purified

by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a white solid in 51% yield (0.094 g).

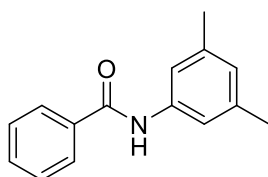
Characterisation data were consistent with the previously reported literature values.<sup>[7]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.86 – 7.76 (m, 2H), 7.54 – 7.47 (m, 1H), 7.45 – 7.34 (m, 6H), 7.34 – 7.26 (m, 1H), 6.59 (appd, *J* = 7.9, 1H), 5.37 (p, *J* = 7.1, 1H), 1.62 (d, *J* = 6.9, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 166.8, 143.2, 134.6, 131.5, 128.8, 128.6, 127.5, 127.0, 126.3, 49.3, 21.8.

LCMS (ESI<sup>+</sup>) *rt* = 2.2 min, *m/z* = 226.1 [M+H]<sup>+</sup>

### ***N*-(3,5-dimethylphenyl)benzamide 4e**



Synthesised according to the general method for the synthesis of amides from benzoic acid (0.100 g, 0.82 mmol) and 3,5-dimethylaniline (0.099 g, 0.82 mmol) The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a white solid in 67% yield (0.123 g).

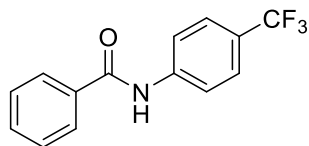
Characterisation data were consistent with the previously reported literature values.<sup>[8]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.98 (brs, 1H), 7.91 – 7.83 (m, 2H), 7.59 – 7.52 (m, 1H), 7.52 – 7.43 (m, 2H), 7.31 (s, 2H), 6.81 (s, 1H), 2.32 (s, 6H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 165.8, 138.8, 137.8, 135.1, 131.7, 128.7, 127.1, 126.3, 118.1, 21.4.

LCMS (ESI<sup>+</sup>) *rt* = 2.6 min, *m/z* = 226.1 [M+H]<sup>+</sup>

## **N-(4-(trifluoromethyl)phenyl)benzamide 4f**



Synthesised according to the general method for the synthesis of amides from benzoic acid (0.100 g, 0.82 mmol) and 4-(trifluoromethyl)aniline (0.132 g, 0.82 mmol) with the following modification, after addition of the amine the reaction mixture was heated to 100 °C in a sealed tube using a hotplate. The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a yellow solid in 87% yield (0.190 g).

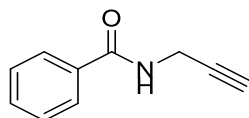
Characterisation data were consistent with the previously reported literature values.<sup>[9]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.98 (brs, 1H), 7.94 – 7.88 (m, 2H), 7.84 – 7.78 (m, 2H), 7.69 – 7.59 (m, 3H), 7.58 – 7.51 (m, 2H).

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -62.09 (s, 3F).

LCMS (ESI<sup>+</sup>) rt = 2.8 min, m/z = 266.1 [M+H]<sup>+</sup>

## **N-(prop-2-yn-1-yl)benzamide 4g**



Synthesised according to the general method for the synthesis of amides from benzoic acid (0.100 g, 0.82 mmol) and propargyl amine (0.045 g, 0.82 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a white solid in 51% yield (0.094 g).

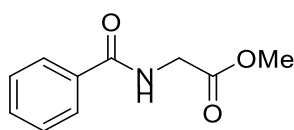
Characterisation data were consistent with the previously reported literature values.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.85 – 7.77 (m, 2H), 7.56 – 7.50 (m, 1H), 7.49 – 7.40 (m, 2H), 6.56 (brs, 1H), 4.27 (dd,  $J = 5.2, 2.6$ , 2H), 2.30 (t,  $J = 2.6$ , 1H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  167.2, 133.7, 131.8, 128.6, 127.1, 79.5, 71.9, 29.8.

LCMS (ESI<sup>+</sup>)  $rt = 1.3$  min,  $m/z = 160.1$   $[\text{M}+\text{H}]^+$

### **methyl benzoylglycinate 4h**



Synthesised according to the general method for the synthesis of amides from benzoic acid (0.100 g, 0.82 mmol) and glycine methyl ester (0.103 g, 0.82 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a yellow oil in 45% yield (0.072 g).

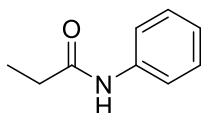
Characterisation data were consistent with the previously reported literature values.<sup>[10]</sup>

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.85 – 7.79 (m, 2H), 7.55 – 7.49 (m, 1H), 7.47 – 7.39 (m, 2H), 6.92 (brs, 1H), 4.24 (d,  $J = 5.2$ , 2H), 3.79 (s, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  170.6, 167.6, 133.6, 131.8, 128.6, 127.1, 52.5, 41.7.

LCMS (ESI<sup>+</sup>)  $rt = 1.3$  min,  $m/z = 294.1$   $[\text{M}+\text{H}]^+$

### **N-phenylpropionamide 4i**



Synthesised according to the general method for the synthesis of amides from propanoic acid (0.100 g, 1.35 mmol) and aniline (0.126 g, 1.35 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a cream coloured solid in 82% yield (0.164 g).

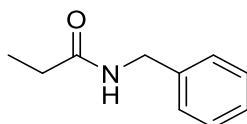
Characterisation data were consistent with the previously reported literature values.<sup>[11]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.71 (brs, 1H), 7.55 (appd, *J* = 7.9, 2H), 7.31 (appt, *J* = 7.9, 2H), 7.11 (appt, *J* = 7.4, 1H), 2.40 (q, *J* = 7.6, 2H), 1.25 (t, *J* = 7.6, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 172.4, 138.1, 128.9, 124.2, 120.0, 30.7, 9.8.

LCMS (ESI<sup>+</sup>) rt = 1.5 min, m/z = 150.1 [M+H]<sup>+</sup>

### ***N*-benzylpropionamide 4j**



Synthesised according to the general method for the synthesis of amides from propanoic acid (0.100 g, 1.35 mmol) and benzylamine (0.144 g, 1.35 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a white solid in 54% yield (0.119 g).

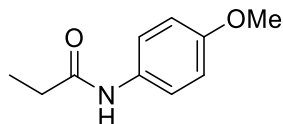
Characterisation data were consistent with the previously reported literature values.<sup>[12]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.37 – 7.31 (m, 2H), 7.31 – 7.25 (m, 3H), 6.15 (s, 1H), 4.42 (d, *J* = 5.7, 2H), 2.24 (q, *J* = 7.6, 2H), 1.17 (t, *J* = 7.6, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 173.8, 138.5, 128.7, 127.8, 127.4, 43.5, 29.7, 9.9.

LCMS (ESI<sup>+</sup>) rt = 1.5 min, m/z = 164.1 [M+H]<sup>+</sup>

### ***N*-(4-methoxyphenyl)propionamide 4k**



Synthesised according to the general method for the synthesis of amides from propanoic acid (0.100 g, 1.35 mmol) and 4-methoxyaniline (0.166 g, 1.35 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a grey solid in 80% yield (0.193 g).

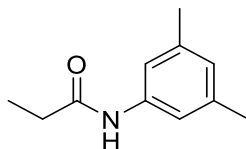
Characterisation data were consistent with the previously reported literature values.<sup>[13]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.94 (s, 1H), 7.41 (d, *J* = 9.1, 2H), 6.81 (d, *J* = 9.1, 2H), 3.77 (s, 3H), 2.34 (q, *J* = 7.6, 2H), 1.21 (t, *J* = 7.6, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 172.5, 156.2, 131.3, 122.0, 114.0, 55.5, 30.4, 9.9.

LCMS (ESI<sup>+</sup>) *rt* = 1.5 min, *m/z* = 180.1 [M+H]<sup>+</sup>

### ***N*-(3,5-dimethylphenyl)propionamide 4l**



Synthesised according to the general method for the synthesis of amides from propanoic acid (0.100 g, 1.35 mmol) and 3,5-dimethylaniline (0.163 g, 1.35 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a brown solid in 91% yield (0.217 g).

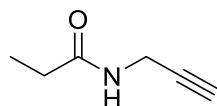
Characterisation data were consistent with the previously reported literature values.<sup>[14]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.62 (s, 1H), 7.19 (s, 2H), 6.75 (s, 1H), 2.39 (q, *J* = 7.6, 2H), 2.28 (s, 6H), 1.24 (t, *J* = 7.6, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 172.3, 138.6, 137.9, 125.9, 117.7, 30.7, 21.4, 9.8.

LCMS (ESI<sup>+</sup>) *rt* = 2.1 min, *m/z* = 178.1 [M+H]<sup>+</sup>

### ***N*-(prop-2-yn-1-yl)propionamide 4m**



Synthesised according to the general method for the synthesis of amides from propanoic acid (0.100 g, 1.35 mmol) and propargylamine (0.074 g, 1.35 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a clear oil in 27% yield (0.041 g).

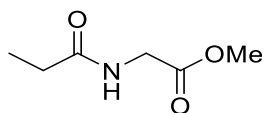
Characterisation data were consistent with the previously reported literature values.<sup>[15]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 6.08 (s, 1H), 4.05 (dd, *J* = 5.3, 2.6, 2H), 2.29 – 2.19 (m, 3H), 1.16 (t, *J* = 7.6, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 173.6, 79.7, 71.4, 29.4, 29.1, 9.6.

LCMS (ESI<sup>+</sup>) *rt* = 0.6 min, *m/z* = 112.1 [M+H]<sup>+</sup>

### **methyl propionylglycinate 4n**



Synthesised according to the general method for the synthesis of amides from propanoic acid (0.100 g, 1.35 mmol) and glycine methyl ester (0.169 g, 1.35 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a yellow oil in 53% yield (0.103 g).

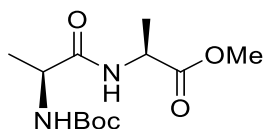
Characterisation data were consistent with the previously reported literature values.<sup>[16]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 6.36 (brs, 1H), 4.02 (d, *J* = 5.3, 2H), 3.73 (s, 3H), 2.27 (q, *J* = 7.6, 2H), 1.14 (t, *J* = 7.6, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 174.2, 170.7, 52.3, 41.2, 29.3, 9.6.

LCMS (ESI<sup>+</sup>) *rt* = 0.6 min, *m/z* = 146.1 [M+H]<sup>+</sup>

### **methyl (*tert*-butoxycarbonyl)-L-alanyl-L-alaninate 4o**



Synthesised according to the general method for the synthesis of amides from Boc-Ala-OH (0.100 g, 0.53 mmol) and alanine methyl ester (0.188 g, 1.35 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a white solid in 82% yield (0.119 g).

Characterisation data were consistent with the previously reported literature values.<sup>[17]</sup>

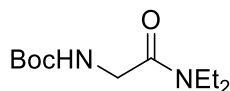
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 6.79 – 6.61 (brm, 1H), 5.14 – 4.99 (brm, 1H), 4.59 (p, *J* = 7.3, 1H), 4.26 – 4.14 (brm, 1H), 3.76 (s, 3H), 1.46 (s, 9H), 1.42 (d, *J* = 7.1, 3H), 1.38 (d, *J* = 7.1, 3H).



$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  173.2, 172.2, 155.5, 80.2, 52.5, 50.0, 48.0, 28.3, 18.4.

LCMS (ESI<sup>+</sup>)  $rt = 1.6$  min,  $m/z = 275.2$   $[\text{M}+\text{H}]^+$

### ***tert*-butyl (2-(diethylamino)-2-oxoethyl)carbamate 4p**



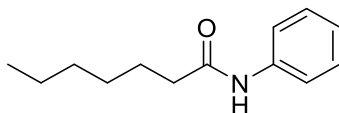
Synthesised according to the general method for the synthesis of amides from Boc-Gly-OH (0.172 g, 0.98 mmol) and diethylamine (0.072 g, 0.98 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a clear oil in 36% yield (0.081 g).

Characterisation data were consistent with the previously reported literature values.<sup>[18]</sup>

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.58 (brs, 1H), 3.99 (s, 2H), 3.42 (q,  $J = 7.2$ , 2H), 3.28 (q,  $J = 7.2$ , 2H), 1.46 (s, 9H), 1.21 (t,  $J = 7.2$ , 3H), 1.15 (t,  $J = 7.2$ , 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  167.6, 156.0, 79.9, 42.1, 41.1, 40.6, 28.3, 14.0, 12.9.

### ***N*-phenylheptanamide 4q**



Synthesised according to the general method for the synthesis of amides from heptanoic acid (0.111 g, 0.85 mmol) and aniline (0.079 g, 0.85 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a brown solid in 71% yield (0.124 g).

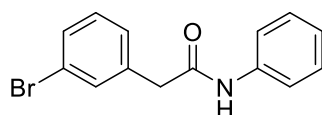
Characterisation data were consistent with the previously reported literature values.<sup>[19]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.61 – 7.49 (m, 3H), 7.32 (appt, *J* = 7.9, 2H), 7.11 (appt, *J* = 7.4, 1H), 2.42 – 2.31 (m, 2H), 1.73 (p, *J* = 7.5 Hz, 3H), 1.44 – 1.24 (m, 6H), 0.95 – 0.84 (m, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 171.7, 138.0, 129.0, 124.2, 119.9, 37.8, 31.6, 29.0, 25.7, 22.5, 14.1.

LCMS (ESI<sup>+</sup>) *rt* = 2.7 min, *m/z* = 206.7 [M+H]<sup>+</sup>

### ***N*-phenyl-2-(3-(bromo)phenyl)acetamide 4r**



Synthesised according to the general method for the synthesis of amides from 3-bromophenylacetic acid (0.183 g, 0.85 mmol) and aniline (0.079 g, 0.85 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a white solid in 61% yield (0.151 g).

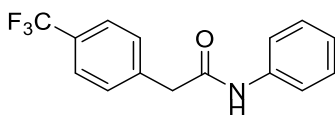
Characterisation data were consistent with the previously reported literature values.<sup>[20]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.53 – 7.51 (m, 1H), 7.50 – 7.44 (m, 3H), 7.35 – 7.27 (m, 5H), 7.16 – 7.10 (m, 1H), 3.70 (s, 2H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 168.3, 137.5, 136.7, 132.5, 130.8, 130.6, 129.0, 128.1, 124.7, 123.1, 120.0, 44.2.

LCMS (ESI<sup>+</sup>) *rt* = 2.6 min, *m/z* = 290.1 [M+H]<sup>+</sup>

### ***N*-phenyl-2-(4-(trifluoromethyl)phenyl)acetamide 4s**



Synthesised according to the general method for the synthesis of amides from 4-(trifluoromethyl)phenylacetic acid (0.173 g, 0.85 mmol) and aniline (0.079 g, 0.85 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a white solid in 66% yield (0.157 g).

Characterisation data were consistent with the previously reported literature values.<sup>[21]</sup>

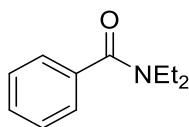
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.68 (appd, *J* = 8.0, 2H), 7.54 – 7.43 (m, 3H), 7.36 – 7.30 (m, 2H), 7.19 – 7.10 (m, 1H), 3.82 (s, 2H).

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -62.56 (s, 3F).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 168.1, 137.8 (d, *J* = 103.5), 129.8, 129.1, 128.9, 126.0 (q, *J* = 3.7), 124.9, 120.0, 44.4.

LCMS (ESI<sup>+</sup>) *rt* = 2.6 min, *m/z* = 280.2 [M+H]<sup>+</sup>

### ***N,N*-diethylbenzamide 4t**



Synthesised according to the general method for the synthesis of amides from benzoic acid (0.120 g, 0.98 mmol) and diethylamine (0.072 g, 0.98 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a clear oil in 91% yield (0.159 g).

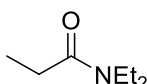
Characterisation data were consistent with the previously reported literature values.<sup>[22]</sup>

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.41 – 7.33 (m, 5H), 3.55 (q,  $J = 7.2$ , 2H), 3.24 (q,  $J = 7.2$ , 2H), 1.25 (t,  $J = 6.5$ , 3H), 1.10 (t,  $J = 6.5$ , 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  171.4, 137.2, 129.1, 128.4, 126.2, 43.3, 39.3, 14.2, 12.9.

LCMS (ESI<sup>+</sup>)  $r_t = 2.1$  min,  $m/z = 178.1$   $[\text{M}+\text{H}]^+$

### ***N,N*-diethylpropionamide 4u**



Synthesised according to the general method for the synthesis of amides from propanoic acid (0.073 g, 0.98 mmol) and diethylamine (0.072 g, 0.98 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a clear oil in 68% yield (0.085 g).

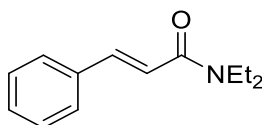
Characterisation data were consistent with the previously reported literature values.<sup>[23]</sup>

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  3.37 (q,  $J = 7.1$ , 2H), 3.30 (q,  $J = 7.1$ , 2H), 2.33 (q,  $J = 7.5$ , 2H), 1.19 – 1.08 (m, 9H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  173.2, 41.9, 40.2, 26.2, 14.3, 13.0, 9.6.

LCMS (ESI<sup>+</sup>)  $r_t = 1.3$  min,  $m/z = 130.1$   $[\text{M}+\text{H}]^+$

### ***N,N*-diethylcinnamamide 4v**



Synthesised according to the general method for the synthesis of amides from *trans*-cinnamic acid (0.145 g, 0.98 mmol) and diethylamine (0.072 g, 0.98 mmol). The crude material was purified by flash

column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a white solid in 52% yield (0.104 g).

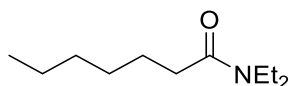
Characterisation data were consistent with the previously reported literature values.<sup>[24]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.72 (d, *J* = 15.4, 1H), 7.54 (dd, *J* = 7.9, 1.7, 2H), 7.42 – 7.34 (m, 3H), 6.84 (d, *J* = 15.4, 1H), 3.55 – 3.45 (m, 4H), 1.27 (t, *J* = 7.1, 3H), 1.20 (t, *J* = 7.1, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 165.8, 142.4, 135.5, 129.5, 128.8, 127.8, 117.7, 42.4, 41.2, 15.1, 13.2.

LCMS (ESI<sup>+</sup>) *rt* = 2.2 min, *m/z* = 204.2 [M+H]<sup>+</sup>

### ***N,N*-diethylheptanamide 4w**



Synthesised according to the general method for the synthesis of amides from heptanoic acid (0.128 g, 0.98 mmol) and diethylamine (0.072 g, 0.98 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a clear oil in 87% yield (0.157 g).

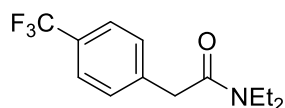
Characterisation data were consistent with the previously reported literature values.<sup>[25]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 3.36 (q, *J* = 7.1, 2H), 3.30 (q, *J* = 7.1, 2H), 2.32 – 2.24 (m, 2H), 1.68 – 1.56 (m, 2H), 1.34 – 1.27 (m, 6H), 1.17 (t, *J* = 7.1, 3H), 1.10 (t, *J* = 7.1, 3H), 0.92 – 0.83 (m, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 172.4, 42.0, 40.0, 33.2, 31.7, 29.2, 25.5, 22.5, 14.4, 14.0, 13.1.

LCMS (ESI<sup>+</sup>) *rt* = 2.5 min, *m/z* = 186.2 [M+H]<sup>+</sup>

### ***N,N*-diethyl-2-(4-(trifluoromethyl)phenyl)acetamide 4x**



Synthesised according to the general method for the synthesis of amides from 4-(trifluoromethyl)phenylacetic acid (0.200 g, 0.98 mmol) and diethylamine (0.072 g, 0.98 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a clear oil in 67% yield (0.167 g).

Characterisation data were consistent with the previously reported literature values.<sup>[26]</sup>

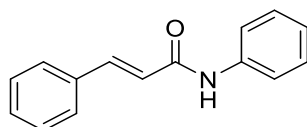
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.59 (d, *J* = 7.9, 2H), 7.39 (d, *J* = 7.9, 2H), 3.77 (s, 2H), 3.42 (q, *J* = 7.1, 2H), 3.33 (q, *J* = 7.1, 2H), 1.20 – 1.11 (m, 6H).

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -62.45 (s, 3F).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 169.5, 139.5 (q, *J* < 2.0), 129.2, 128.4 (q, *J* = 35.7), 125.5 (q, *J* = 3.7), 121.5 (q, *J* = 271.9), 42.5, 40.4, 40.3, 14.3, 12.9.

LCMS (ESI<sup>+</sup>) *rt* = 2.5 min, *m/z* = 260.2 [M+H]<sup>+</sup>

## ***N*-phenylcinnamamide 4y**



Synthesised according to the general method for the synthesis of amides from *trans*-cinnamic acid (0.126 g, 0.85 mmol) and aniline (0.079, 0.85 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a white solid in 68% yield (0.128 g).

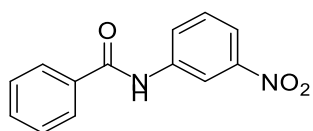
Characterisation data were consistent with the previously reported literature values.<sup>[27]</sup>

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.82 – 7.73 (m, 2H), 7.68 (d,  $J = 8.0$ , 2H), 7.55 – 7.45 (m, 2H), 7.41 – 7.32 (m, 5H), 7.15 (t,  $J = 7.4$ , 1H), 6.63 (d,  $J = 15.5$ , 1H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  164.1, 142.4, 138.1, 134.6, 130.0, 129.1, 128.9, 128.0, 124.5, 120.9, 120.1.

LCMS (ESI<sup>+</sup>)  $r_t = 2.4$  min,  $m/z = 224.1$   $[\text{M}+\text{H}]^+$

### ***N*-(3-nitrophenyl)benzamide 4z**



Synthesised according to the general method for the synthesis of amides from benzoic acid (0.100 g, 0.82 mmol) and 3-nitroaniline (0.113 g, 0.82 mmol) with the following modification, after addition of the amine the reaction mixture was heated to 100 °C in a sealed tube using a hotplate. The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a yellow solid in 73% yield (0.144 g).

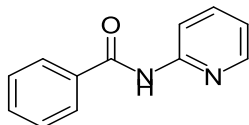
Characterisation data were consistent with the previously reported literature values.<sup>[28]</sup>

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.53 (t,  $J = 2.2$ , 1H), 8.13 (ddd,  $J = 8.2$ , 2.2, 1.0, 1H), 8.09 – 8.02 (m, 2H), 7.96 – 7.89 (m, 2H), 7.66 – 7.59 (m, 1H), 7.59 – 7.53 (m, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  165.9, 148.7, 139.0, 134.0, 132.5, 130.0, 129.0, 127.1, 125.8, 119.2, 114.9.

LCMS (ESI<sup>+</sup>)  $r_t = 2.3$  min,  $m/z = 243.1$   $[\text{M}+\text{H}]^+$

## ***N*-(pyridin-2-yl)benzamide 4aa**



Synthesised according to the general method for the synthesis of amides from benzoic acid (0.100 g, 0.82 mmol) and 2-aminopyridine (0.077 g, 0.82 mmol) with the following modification, after addition of the amine the reaction mixture was heated to 100 °C in a sealed tube using a hotplate. The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a cream coloured solid in 86% yield (0.140 g).

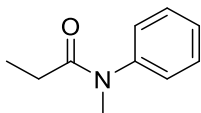
Characterisation data were consistent with the previously reported literature values.<sup>[29]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 9.38 (brs, 1H), 8.43 (d, *J* = 8.4, 1H), 8.10 (ddd, *J* = 5.0, 1.9, 0.9, 1H), 7.97 – 7.90 (m, 2H), 7.76 (ddd, *J* = 8.4, 7.4, 1.9, 1H), 7.60 – 7.53 (m, 1H), 7.51 – 7.44 (m, 2H), 7.03 (ddd, *J* = 7.4, 5.0, 1.1, 1H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 166.1, 151.8, 147.8, 138.5, 134.4, 132.2, 128.8, 127.4, 119.9, 114.4.

LCMS (ESI<sup>+</sup>) *rt* = 1.4 min, *m/z* = 199.1 [M+H]<sup>+</sup>

## ***N*-methyl-*N*-phenylpropionamide 4ab**



Synthesised according to the general method for the synthesis of amides from propanoic acid (0.100 g, 1.35 mmol) and *N*-methylaniline (0.144 g, 1.35 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a white solid in 57% yield (0.126 g).



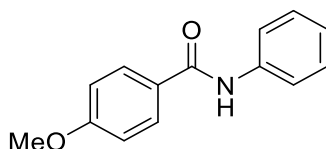
Characterisation data were consistent with the previously reported literature values.<sup>[10]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.42 (appt, *J* = 7.5, 2H), 7.34 (appt, *J* = 7.3, 1H), 7.19 (appd, *J* = 7.4, 2H), 3.27 (s, 3H), 2.08 (q, *J* = 7.6, 2H), 1.05 (t, *J* = 7.6, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 174.0, 144.2, 129.7, 127.7, 127.3, 37.3, 27.5, 9.7.

LCMS (ESI<sup>+</sup>) *rt* = 1.8 min, *m/z* = 164.1 [M+H]<sup>+</sup>

#### 4-methoxy-*N*-phenylbenzamide 4ac



Synthesised according to the general method for the synthesis of amides from 4-methoxy benzoic acid (0.100 g, 0.66 mmol) and aniline (0.061 g, 0.66 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a cream coloured solid in 86% yield (0.129 g).

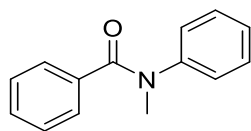
Characterisation data were consistent with the previously reported literature values.<sup>[30]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.90 – 7.84 (m, 2H), 7.77 (brs, 1H), 7.68 – 7.63 (m, 2H), 7.43 – 7.36 (m, 2H), 7.21 – 7.13 (m, 1H), 7.04 – 6.97 (m, 2H), 3.90 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 165.2, 162.5, 138.1, 129.1, 128.9, 127.1, 124.4, 120.1, 114.0, 55.5.

LCMS (ESI<sup>+</sup>) *rt* = 2.2 min, *m/z* = 228.1 [M+H]<sup>+</sup>

#### *N*-methyl-*N*-phenylbenzamide 4ad



Synthesised according to the general method for the synthesis of amides from benzoic acid (0.100 g, 0.82 mmol) and *N*-methylaniline (0.088 g, 0.82 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a white solid in 81% yield (0.141 g).

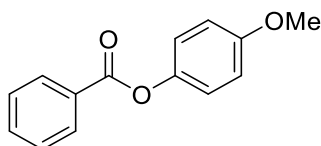
Characterisation data were consistent with the previously reported literature values.<sup>[31]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.34 – 7.29 (m, 2H), 7.27 – 7.21 (m, 3H), 7.21 – 7.15 (m, 3H), 7.08 – 7.03 (m, 2H), 3.53 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 170.9, 144.8, 135.8, 129.7, 129.2, 128.7, 127.8, 126.9, 126.6, 38.5.

LCMS (ESI<sup>+</sup>) rt = 2.1 min, m/z = 212.1 [M+H]<sup>+</sup>

### 4-methoxyphenyl benzoate 6a



Synthesised according to the general method for the synthesis of esters from benzoic acid (0.100 g, 0.82 mmol) and 4-methoxyphenol (0.102 g, 0.82 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a white solid in 68% yield (0.128 g).

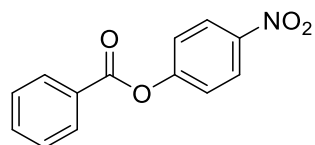
Characterisation data were consistent with the previously reported literature values.<sup>[32]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.26 – 8.20 (m, 2H), 7.70 – 7.64 (m, 2H), 7.58 – 7.50 (m, 2H), 7.17 (d, *J* = 9.1, 2H), 6.98 (d, *J* = 9.1, 2H), 3.86 (s, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  165.6, 157.4, 144.4, 133.5, 130.2, 129.7, 128.6, 122.5, 114.6, 55.6.

LCMS (ESI<sup>+</sup>)  $rt = 2.8$  min,  $m/z = 229.1$   $[\text{M}+\text{H}]^+$

### 4-nitrophenyl benzoate **6b**



Synthesised according to the general method for the synthesis of esters from benzoic acid (0.100 g, 0.82 mmol) and 4-nitrophenol (0.114 g, 0.82 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a yellow solid in 50% yield (0.099 g).

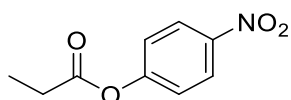
Characterisation data were consistent with the previously reported literature values.<sup>[33]</sup>

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.36 (d,  $J = 9.1$ , 2H), 8.26 – 8.20 (m, 2H), 7.74 – 7.68 (m, 1H), 7.57 (appt,  $J = 7.7$ , 2H), 7.45 (d,  $J = 9.1$ , 2H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  164.3, 155.8, 145.4, 134.3, 130.4, 128.8, 128.6, 125.3, 122.7.

LCMS (ESI<sup>-</sup>)  $rt = 2.6$  min,  $m/z = 278.1$   $[\text{M}+\text{K}]^-$

### 4-nitrophenyl propionate **6c**



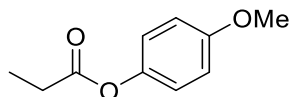
Synthesised according to the general method for the synthesis of esters from propanoic acid (0.100 g, 1.35 mmol) and 4-nitrophenol (0.188 g, 1.35 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a white solid in 23% yield (0.060 g).

Characterisation data were consistent with the previously reported literature values.<sup>[34]</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.30 (d, *J* = 9.1, 2H), 7.31 (d, *J* = 9.1, 2H), 2.67 (q, *J* = 7.5, 2H), 1.31 (t, *J* = 7.5, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 172.0, 155.5, 125.2, 122.4, 27.8, 8.9.

### 4-methoxyphenyl propionate **6d**



Synthesised according to the general method for the synthesis of esters from propanoic acid (0.100 g, 1.35 mmol) and 4-methoxyphenol (0.167 g, 1.35 mmol). The crude material was purified by flash column chromatography (100% hexane to 50% hexane 50% ethyl acetate) to give the desired product as a clear oil in 24% yield (0.061 g).

Characterisation data were consistent with the previously reported literature values.<sup>[35]</sup>

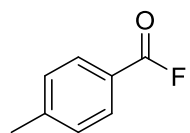
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.03 (d, *J* = 9.1, 2H), 6.91 (d, *J* = 9.1, 2H), 3.82 (s, 3H), 2.60 (q, *J* = 7.6, 2H), 1.28 (t, *J* = 7.6, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 173.4, 157.2, 144.3, 122.3, 114.4, 55.6, 27.7, 9.1.

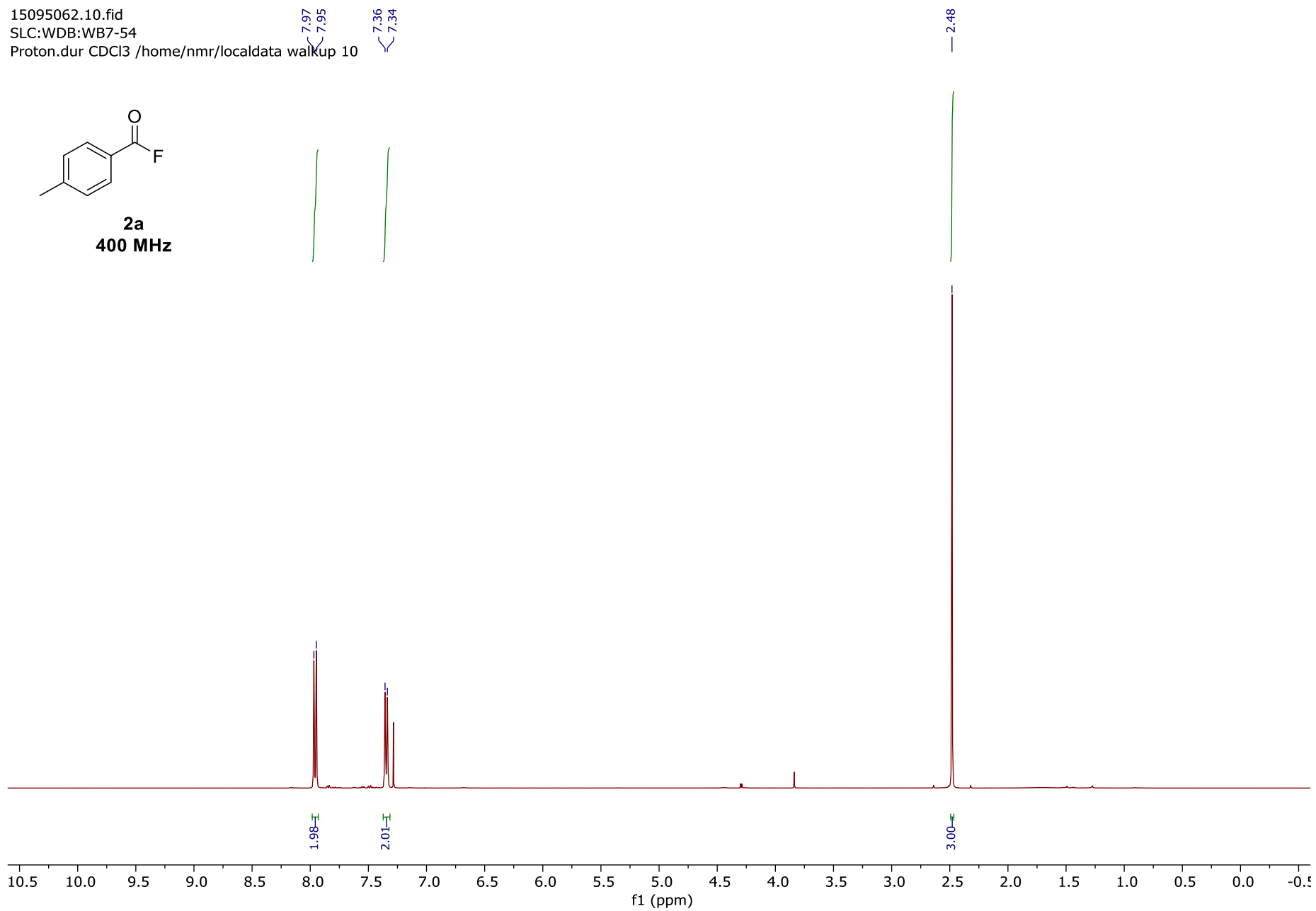
LCMS (ESI<sup>+</sup>) *rt* = 2.2 min, *m/z* = 181.1 [M+H]<sup>+</sup>

## **NMR Data for Synthesised Compounds**

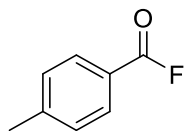
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Proton.dur CDCl3 /home/nmr/localdata walkup 10



**2a**  
400 MHz

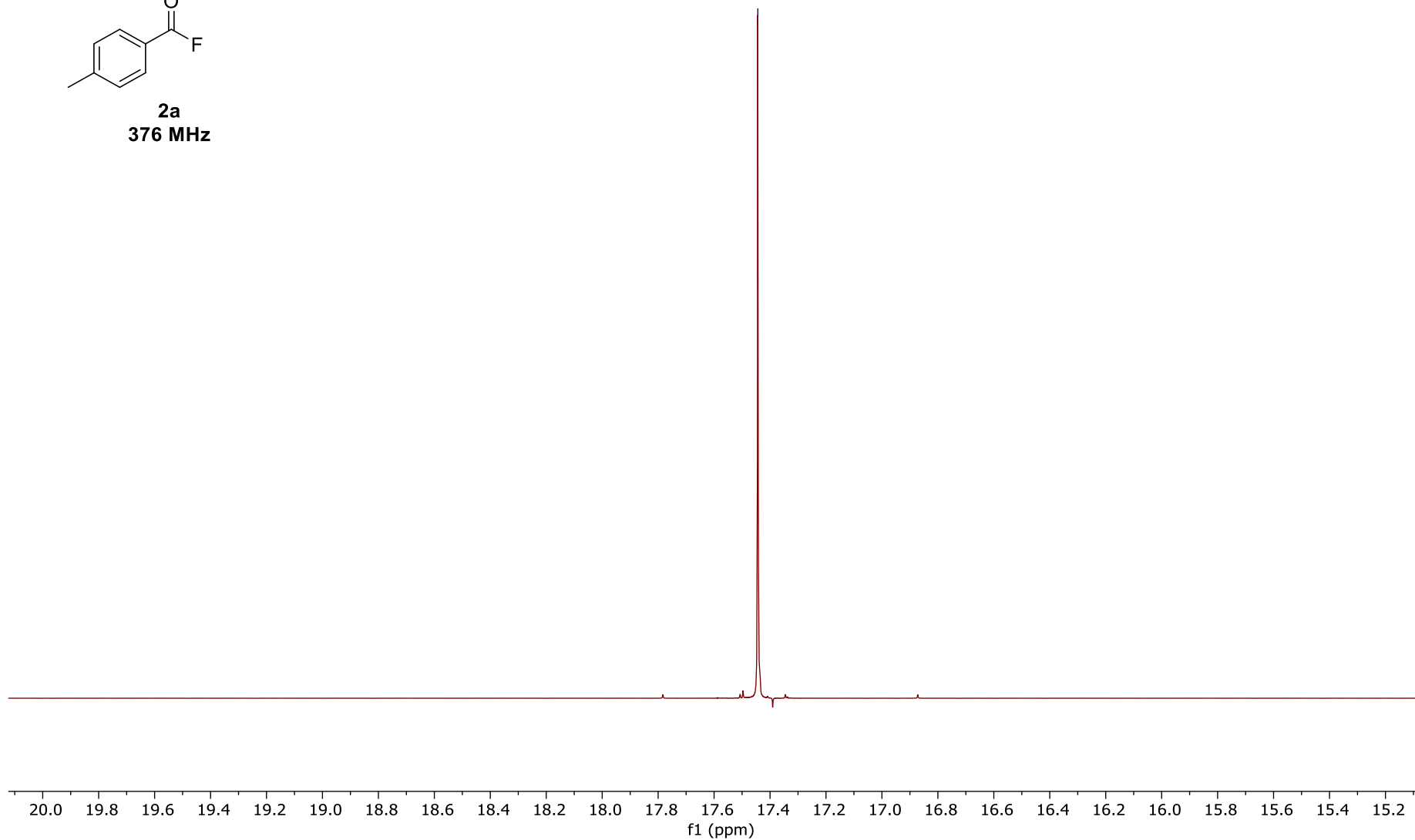


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F19\_limits\_dec.dur CDCl3 /home/nmr/localdata walkup 10



**2a**  
**376 MHz**

17.44



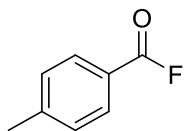
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155.86

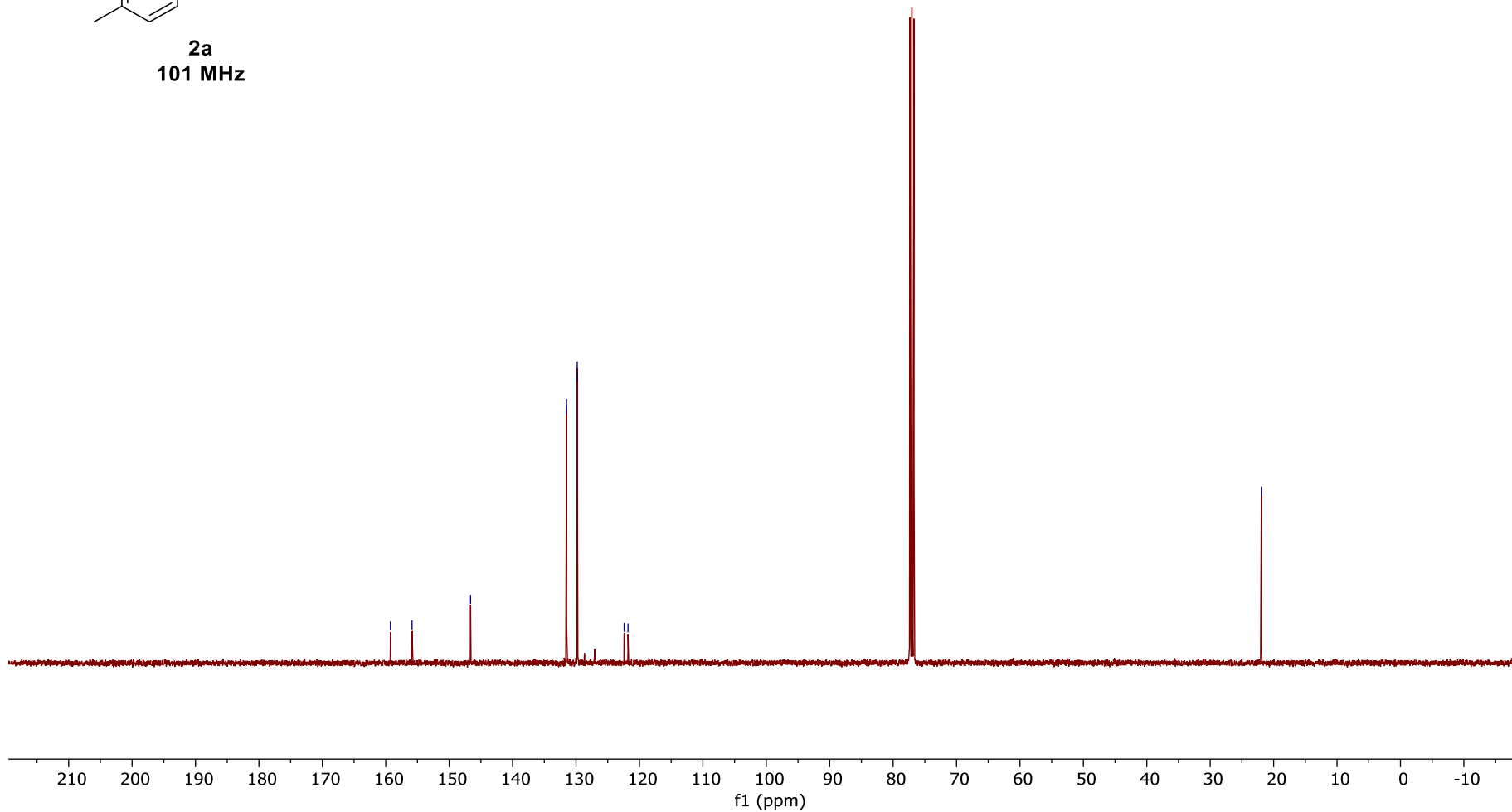
146.63

131.53  
131.49  
129.81  
129.80  
122.41  
121.80

21.94

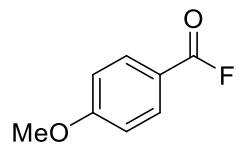


**2a**  
101 MHz

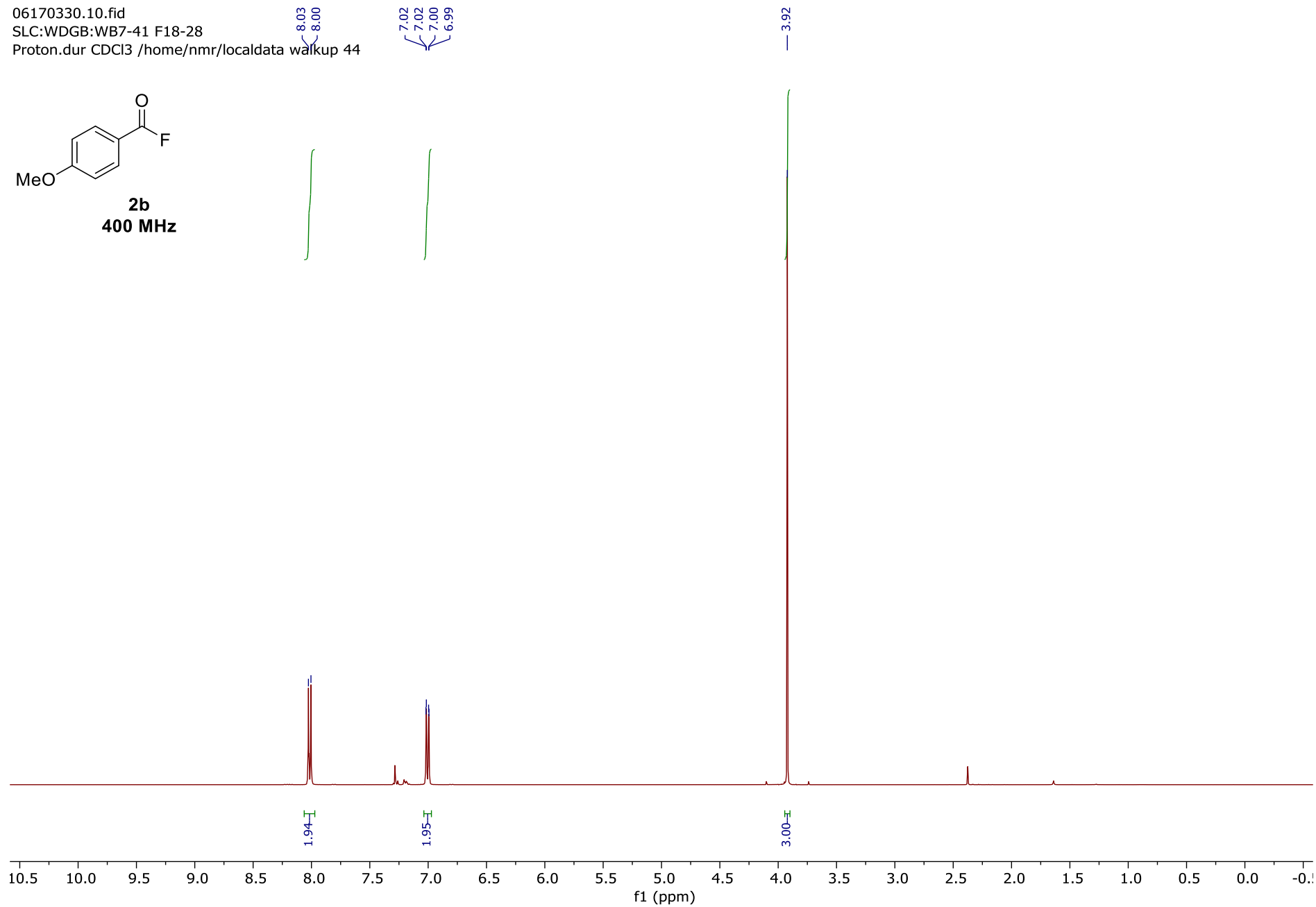




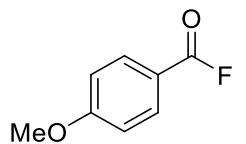
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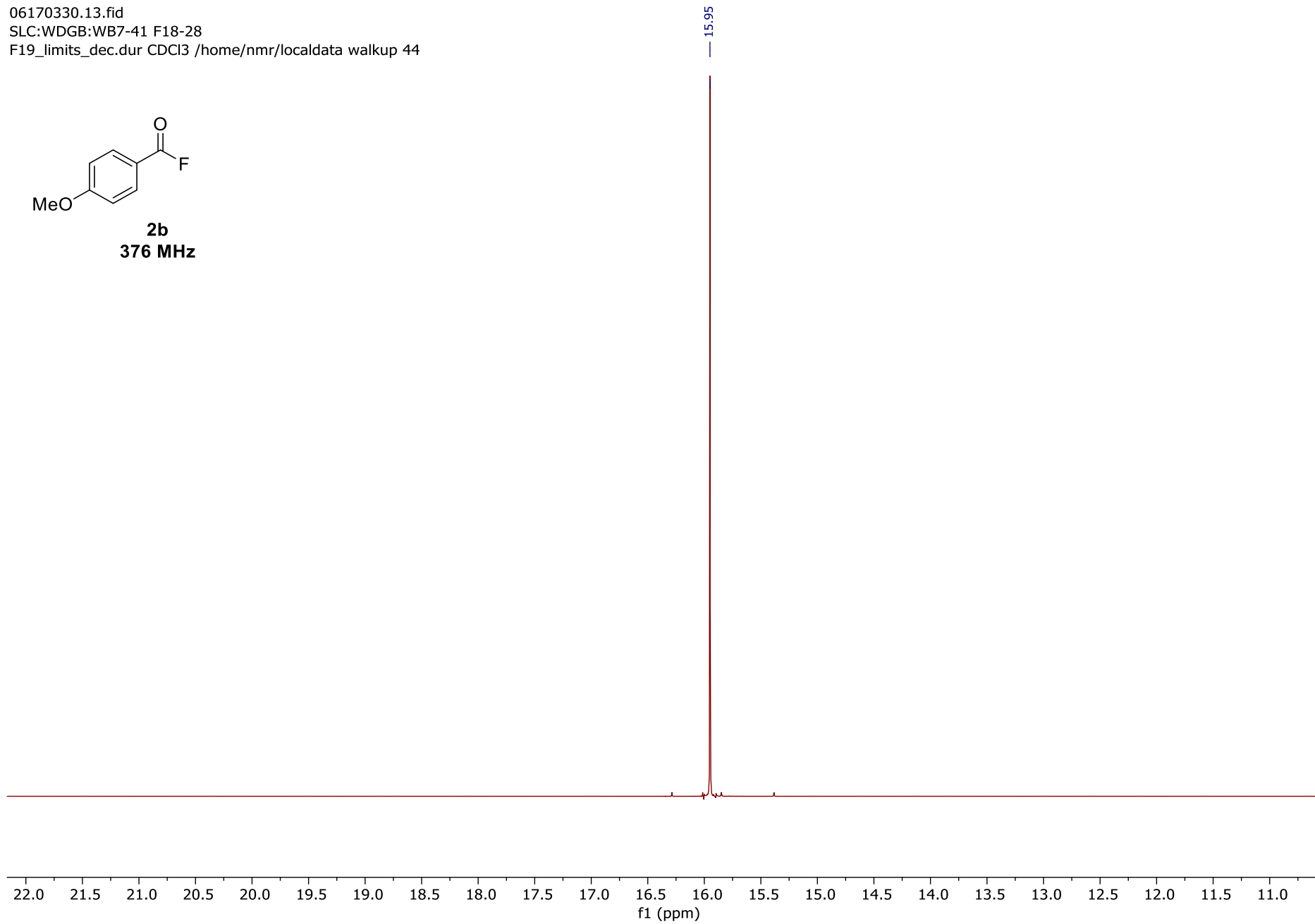
**2b**  
400 MHz



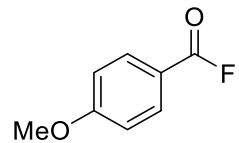
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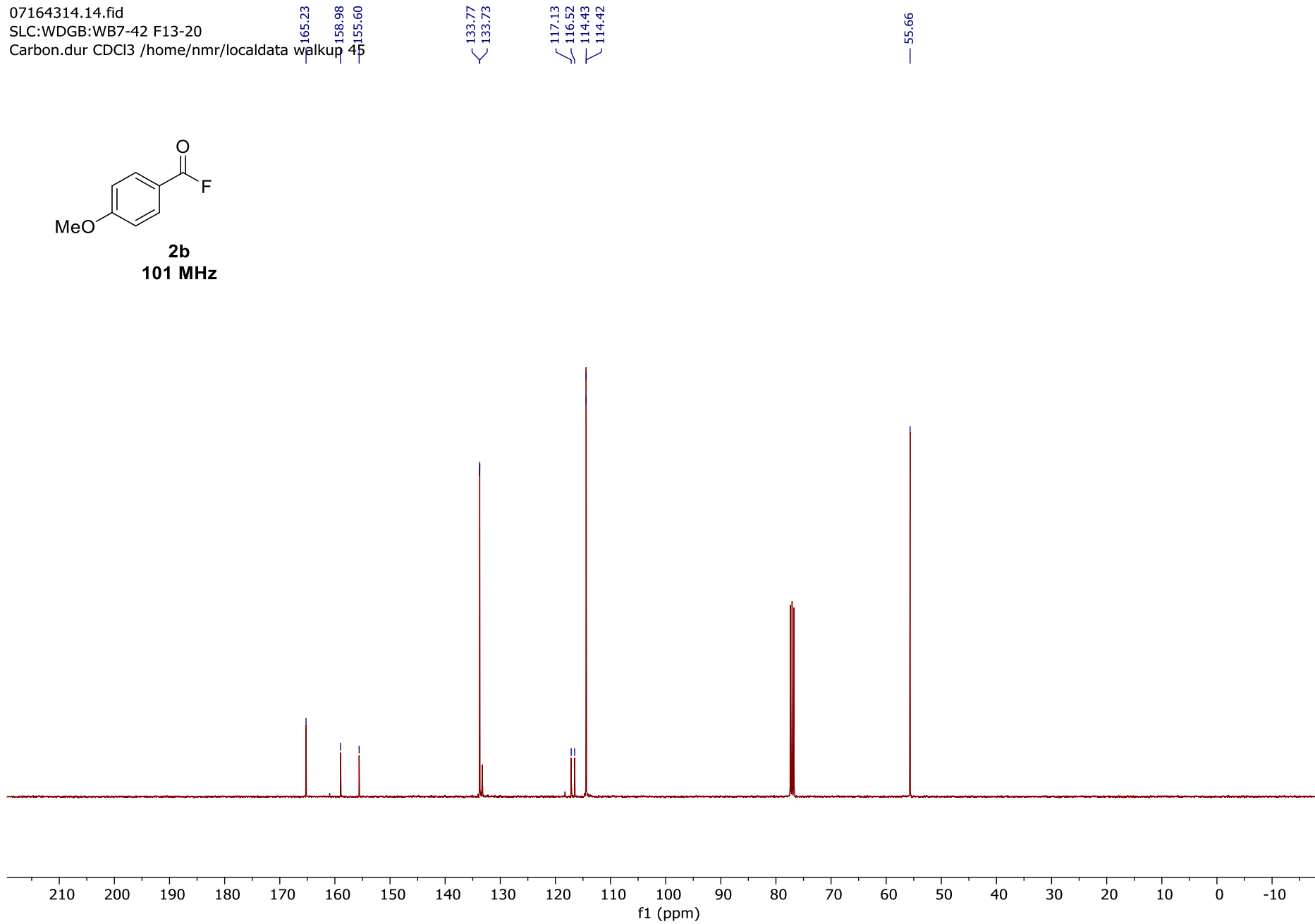
**2b**  
**376 MHz**



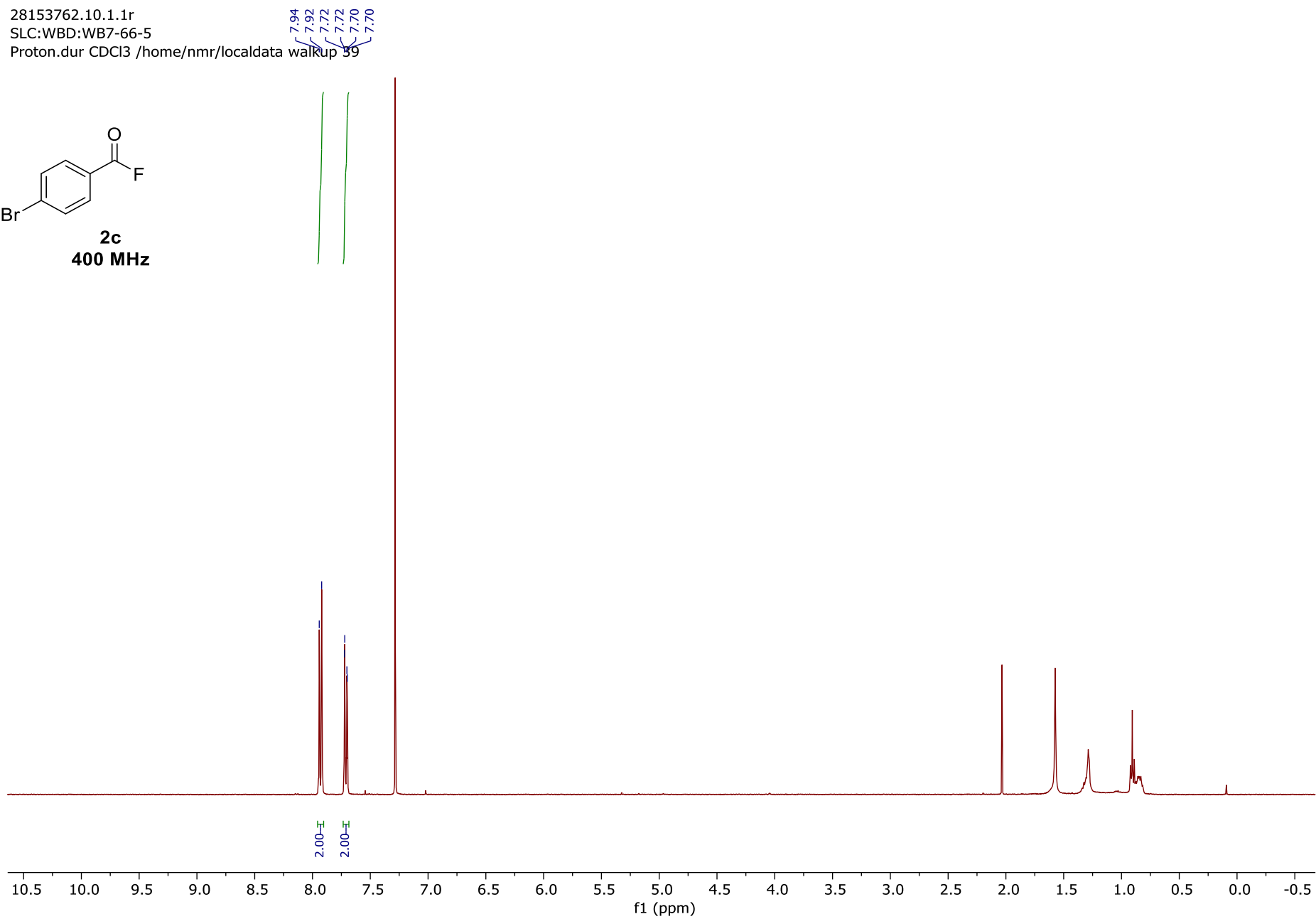
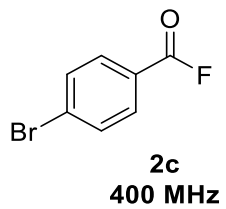
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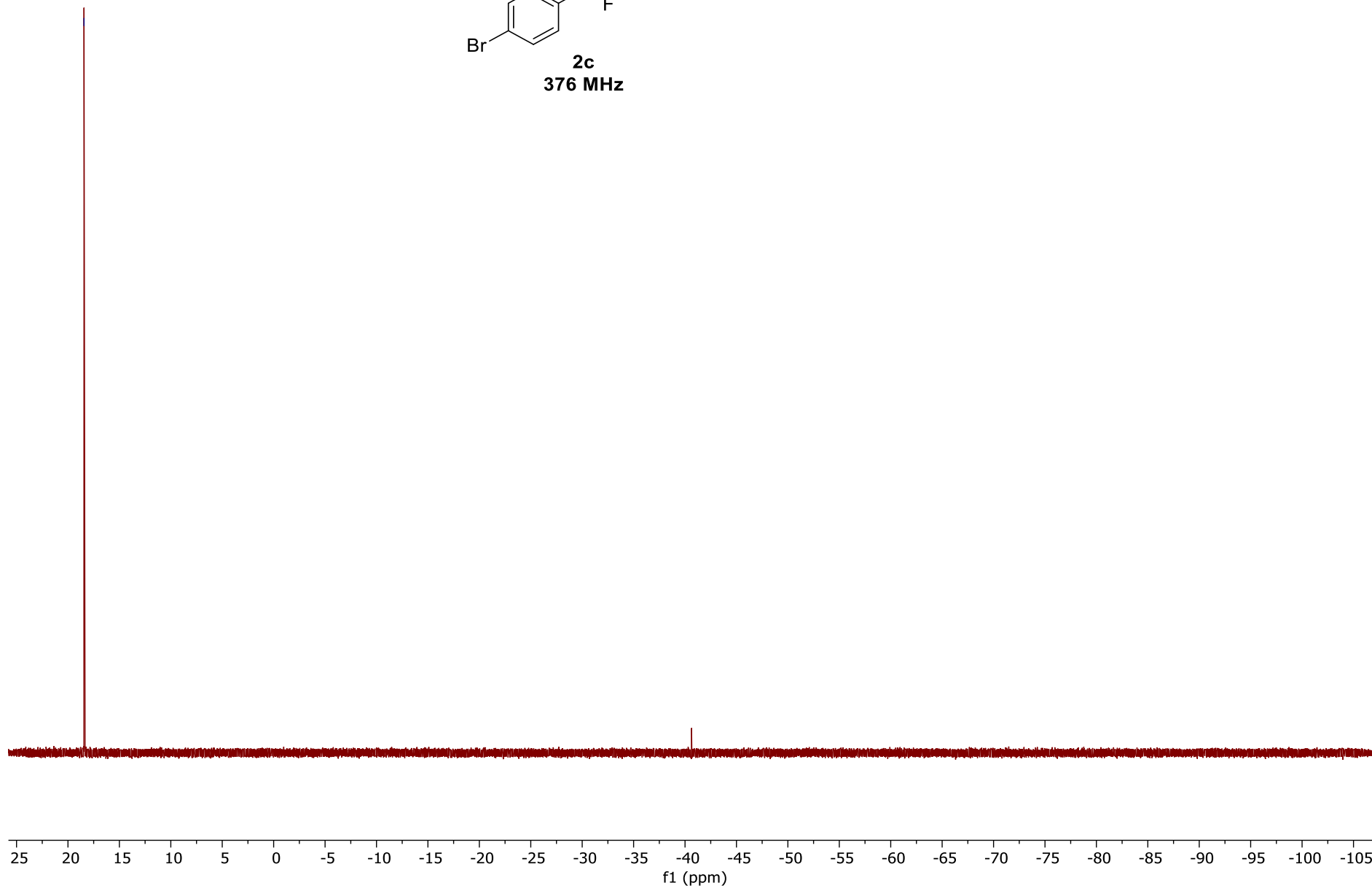
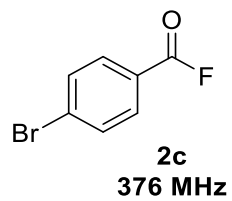
**2b**  
101 MHz



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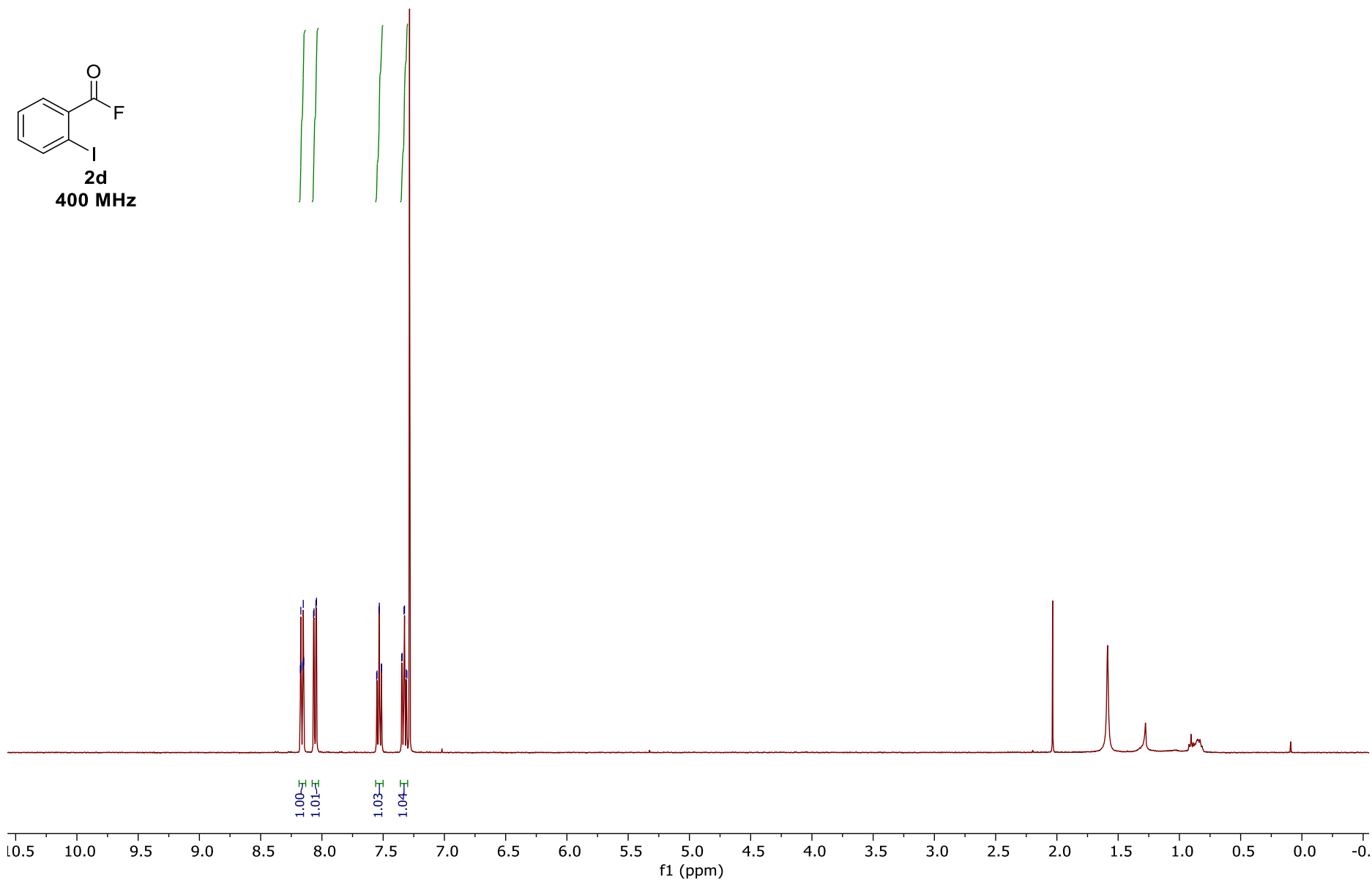
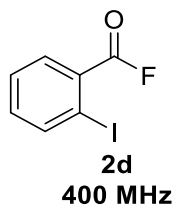


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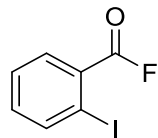


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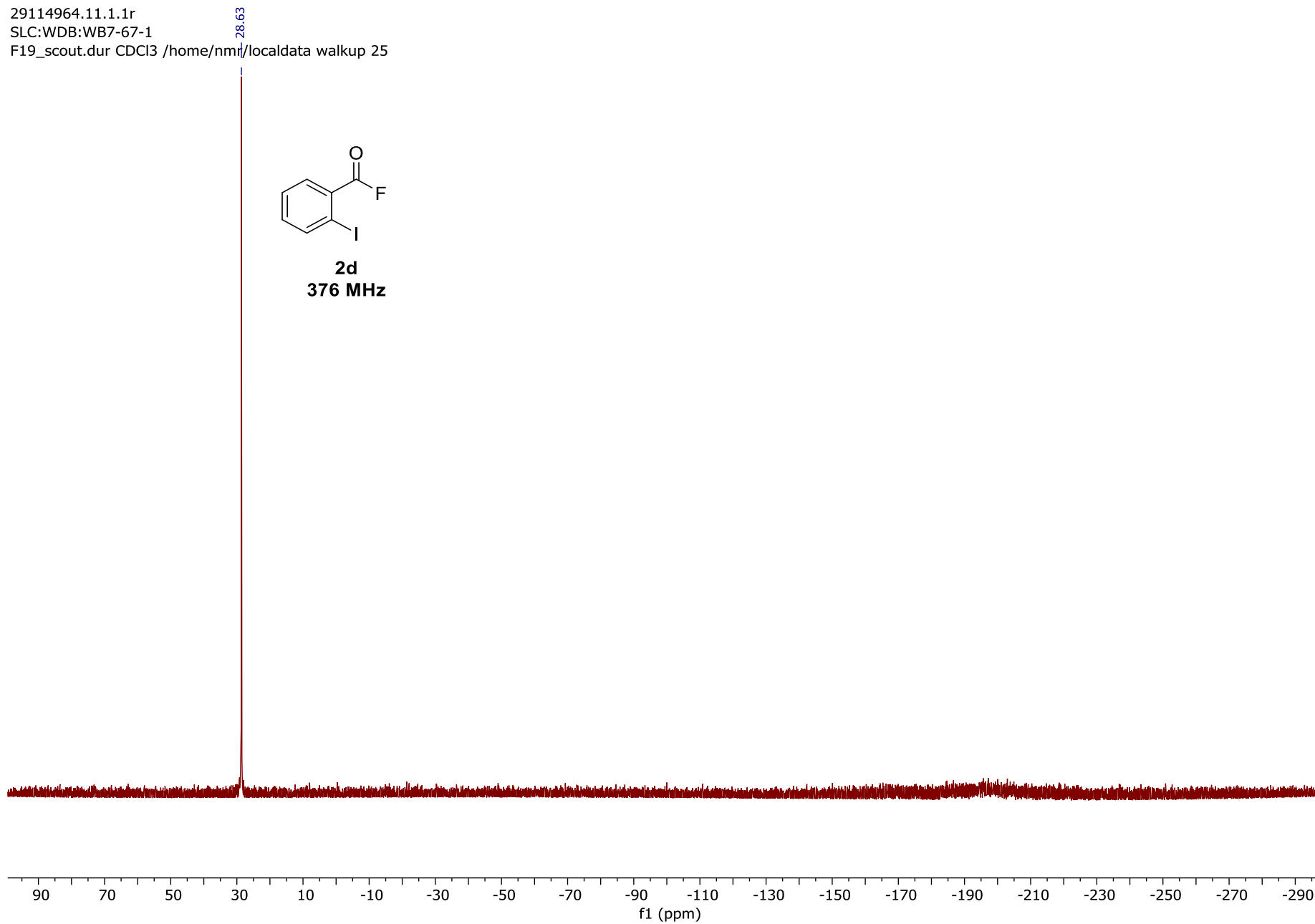
8.18  
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8.15  
8.15  
8.07  
8.06  
8.05  
8.04  
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7.55  
7.53  
7.53  
7.51  
7.35  
7.34  
7.33  
7.33  
7.31  
7.31



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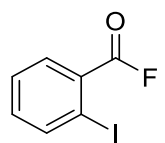


**2d**  
**376 MHz**

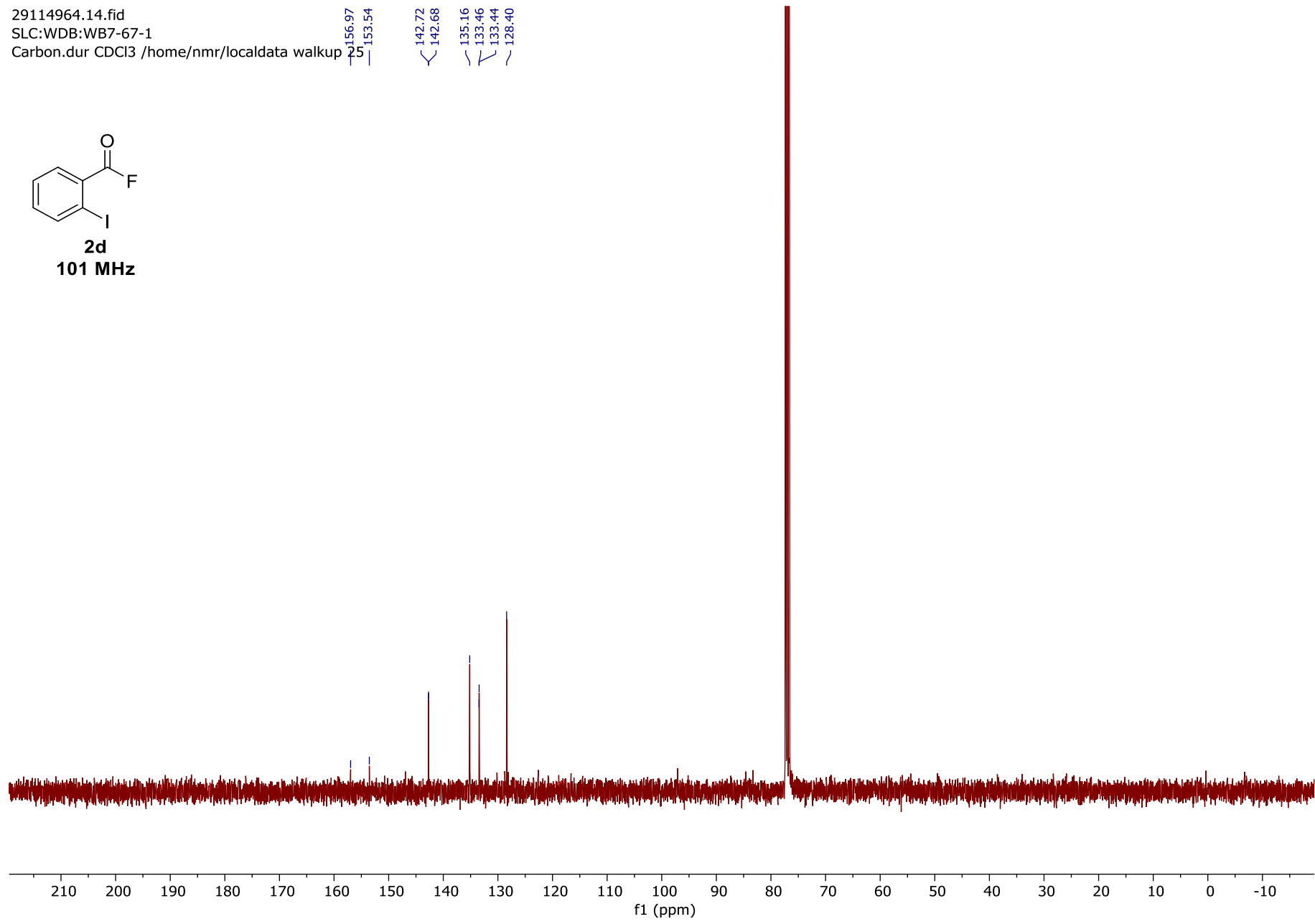


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Carbon.dur CDCl3 /home/nmr/localdata walkup

156.97  
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142.68  
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133.44  
128.40

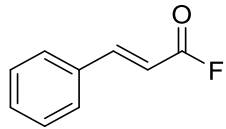


**2d**  
**101 MHz**

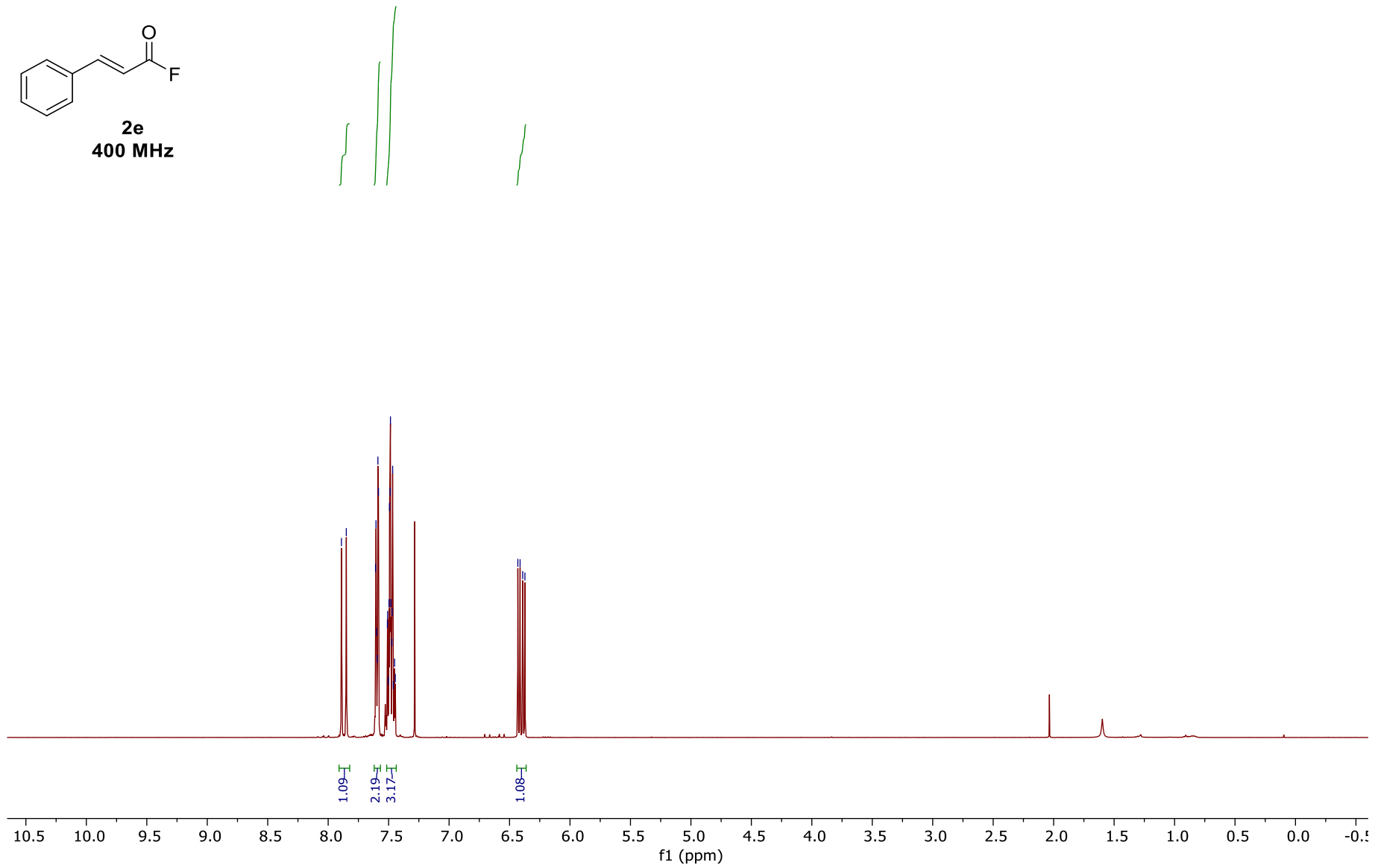




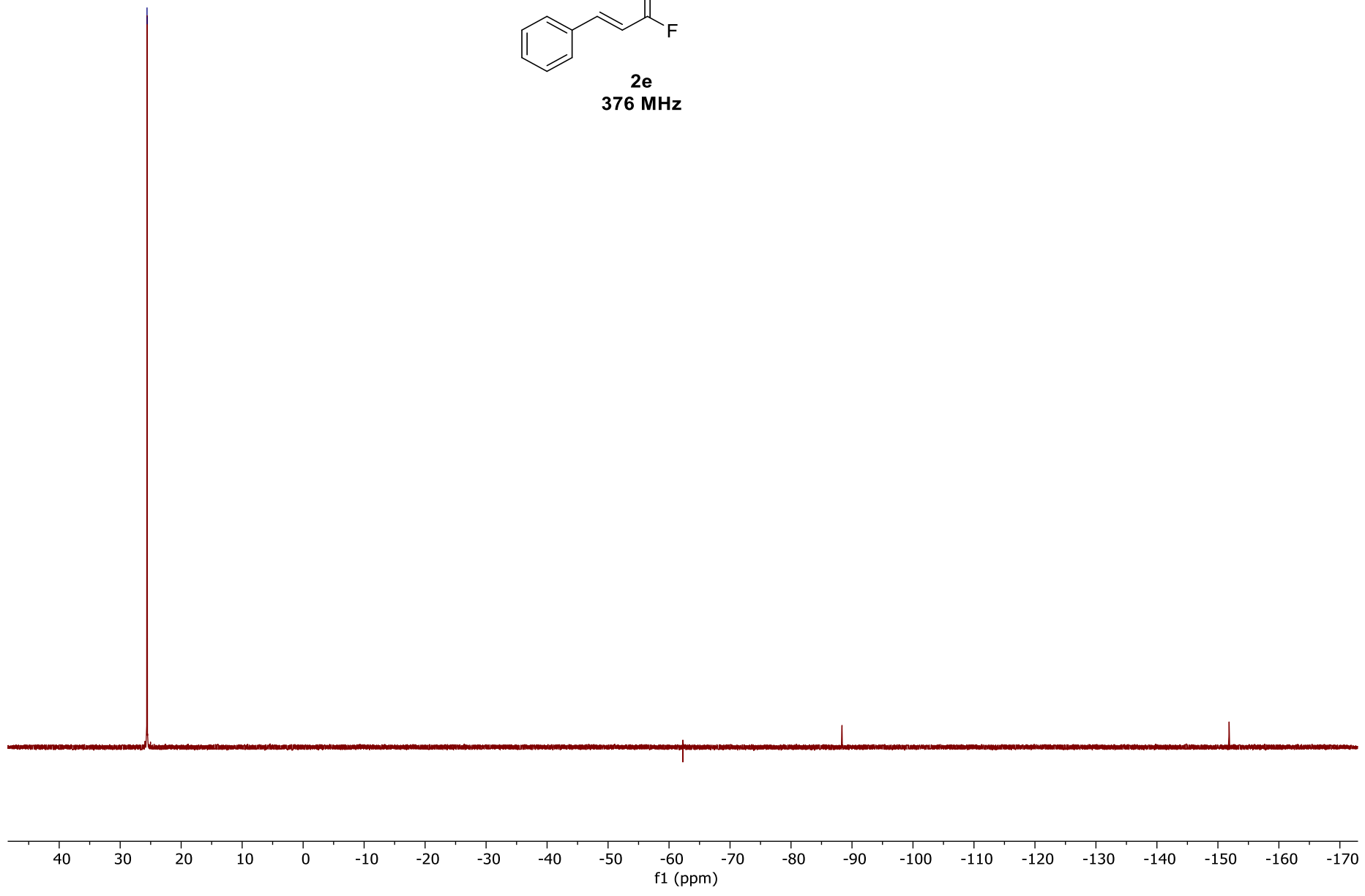
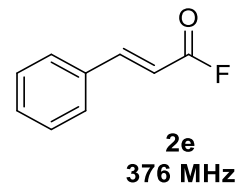
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2e  
400 MHz



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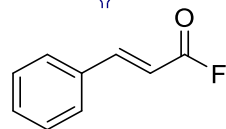


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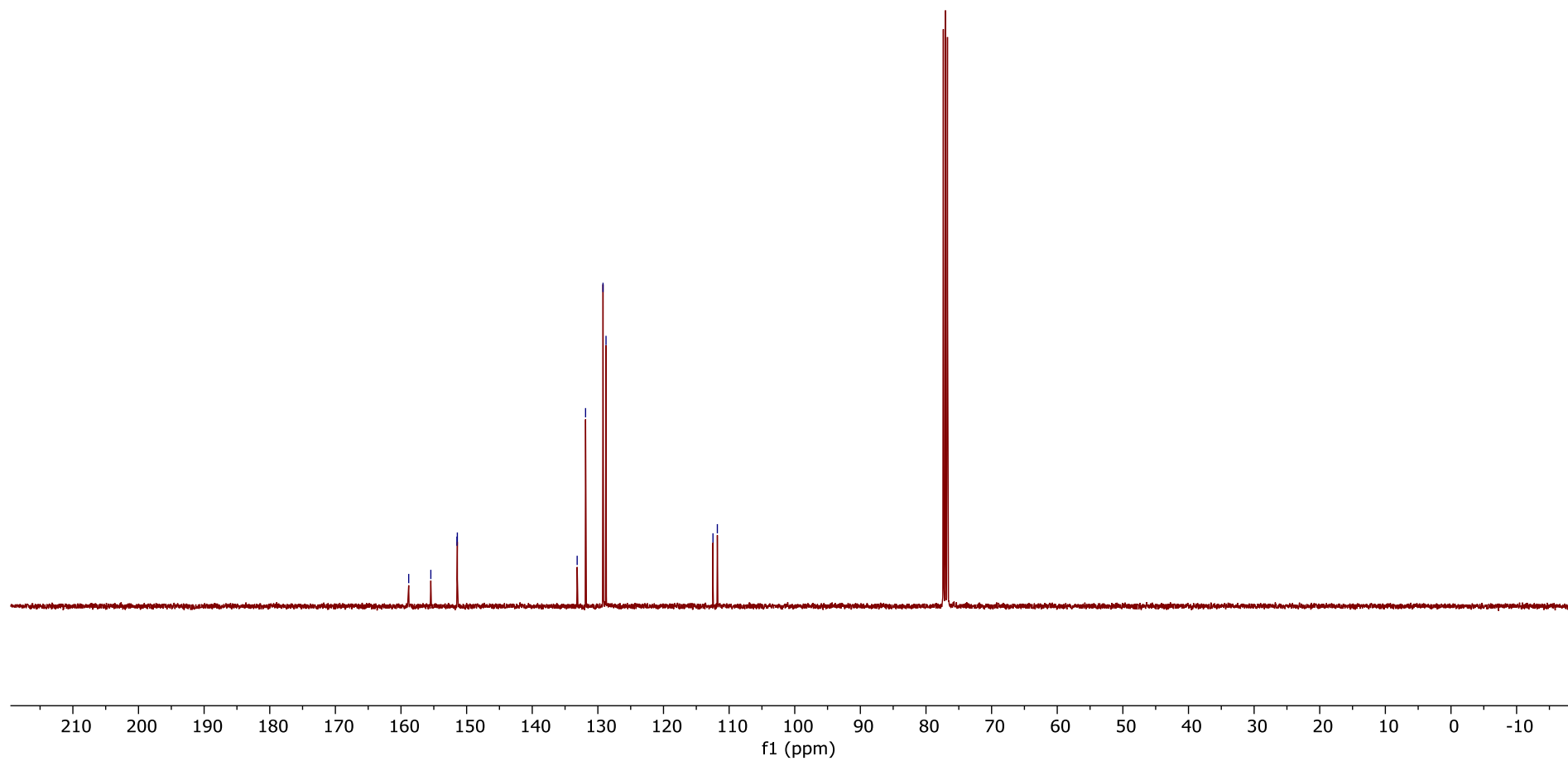
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151.41

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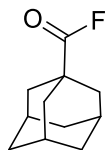
112.46  
111.79



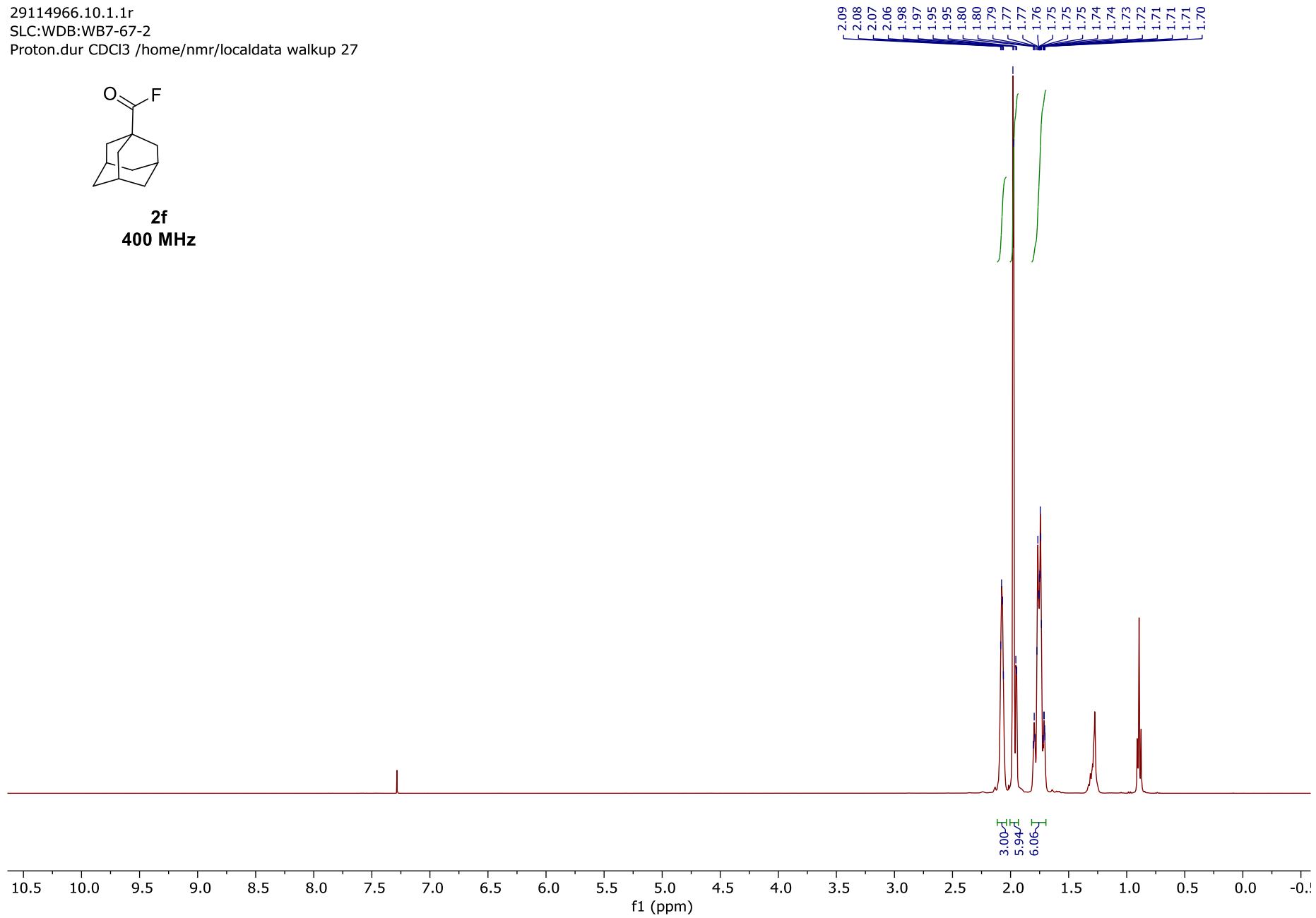
**2e**  
**101 MHz**



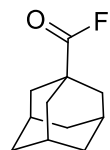
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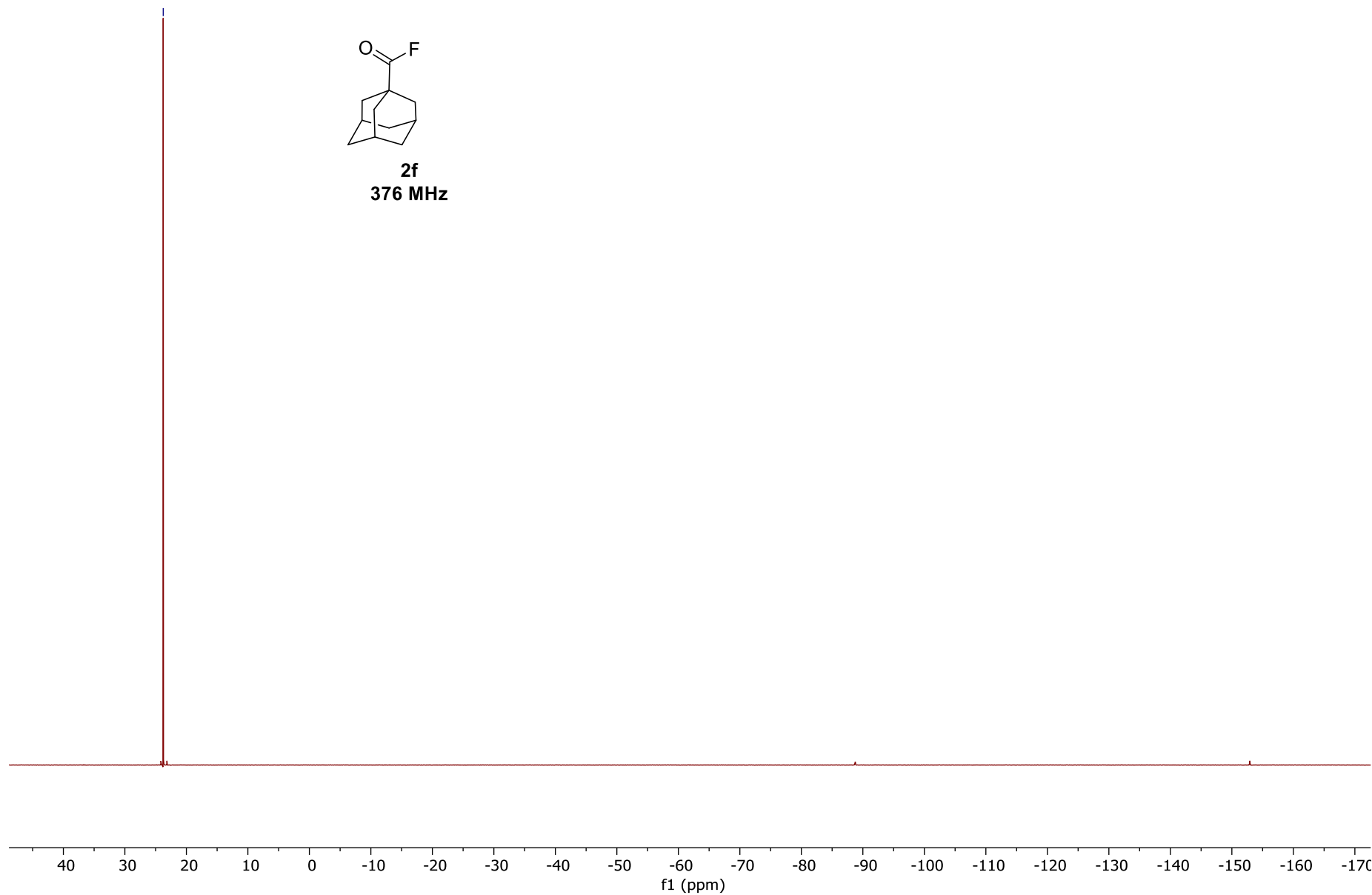
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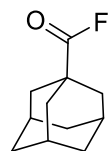


**2f**  
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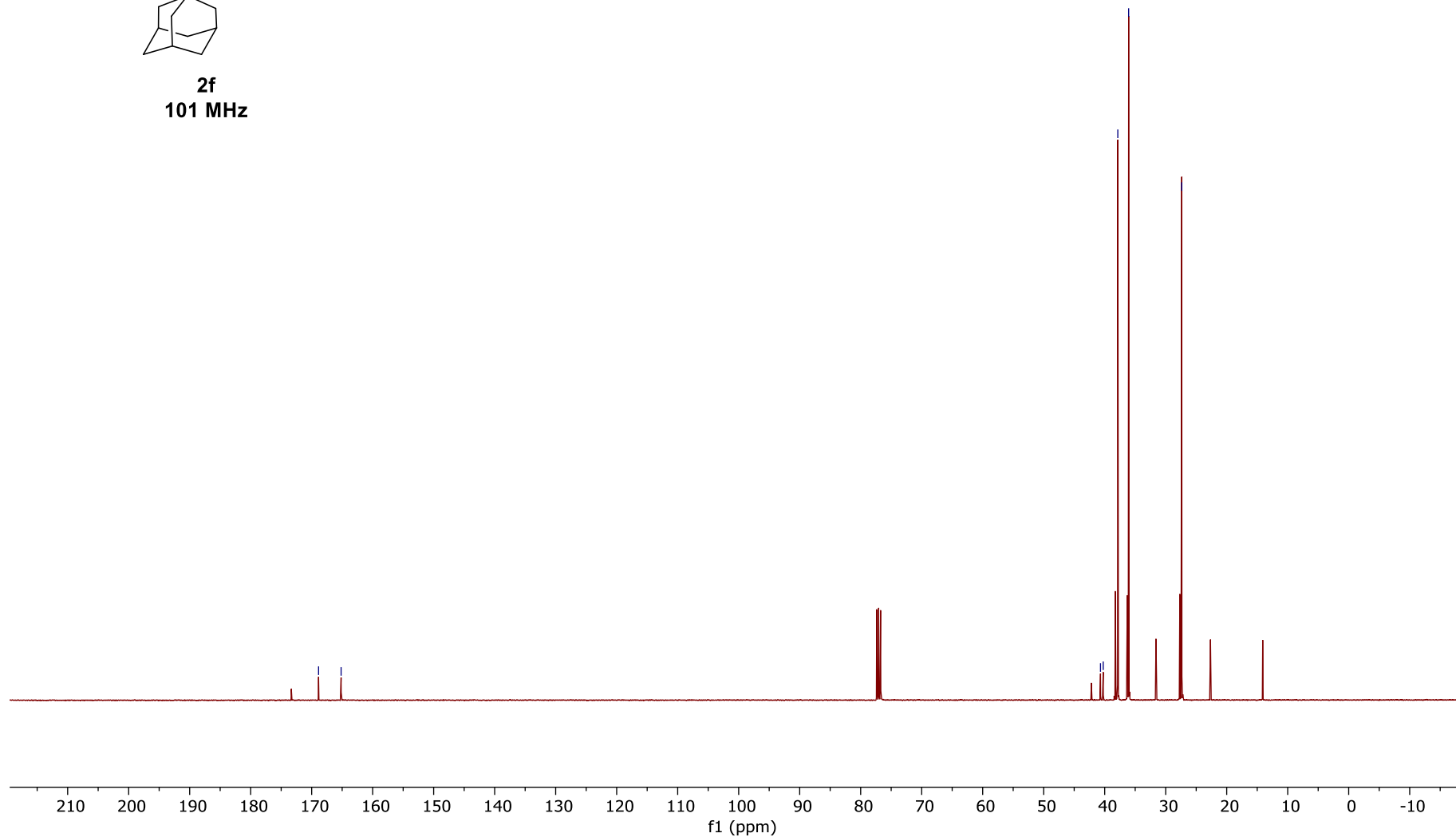


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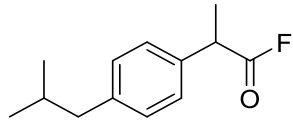
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27.37



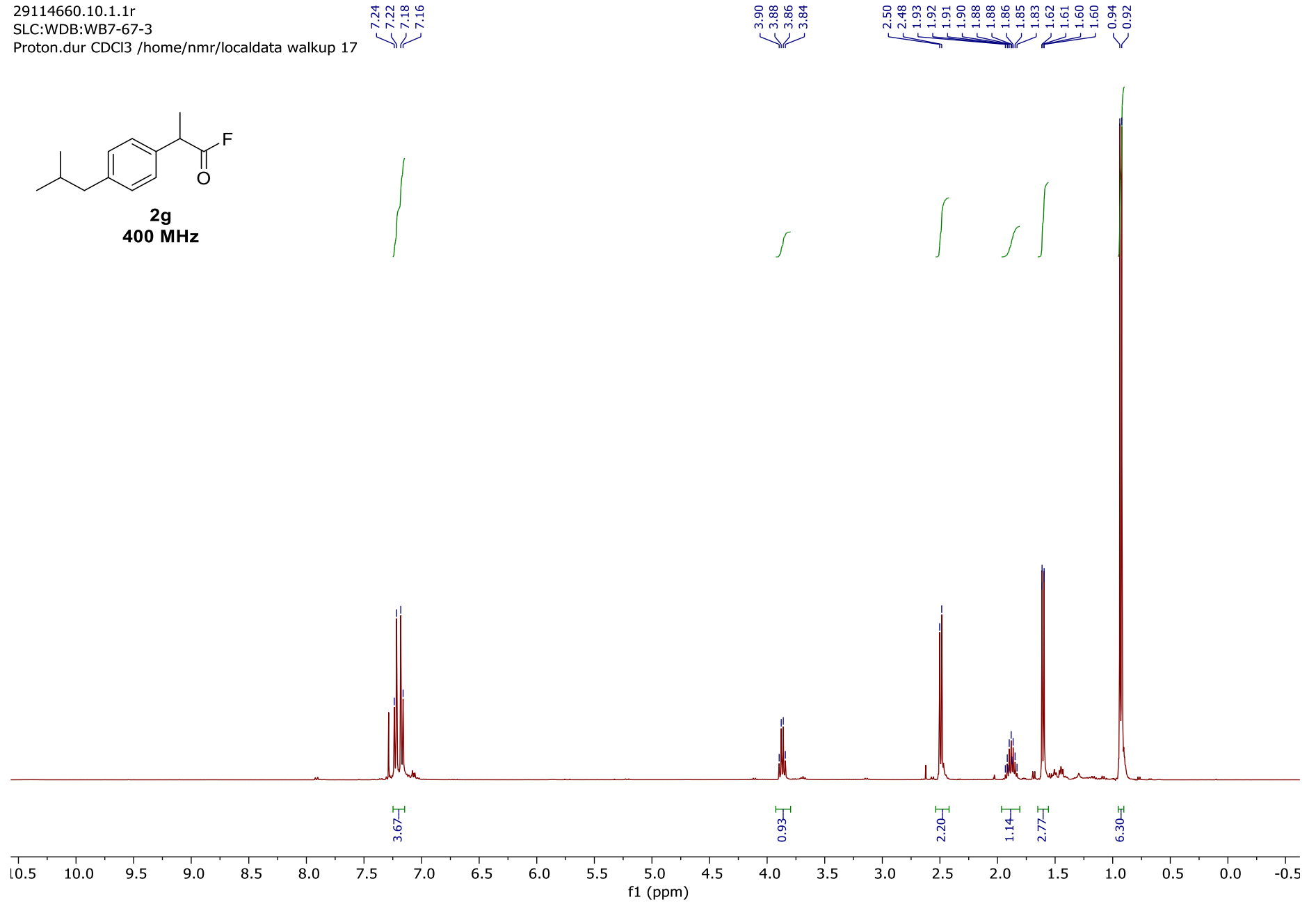
**2f**  
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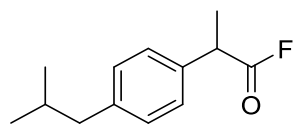
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Proton.dur CDCl3 /home/nmr/localdata walkup 17



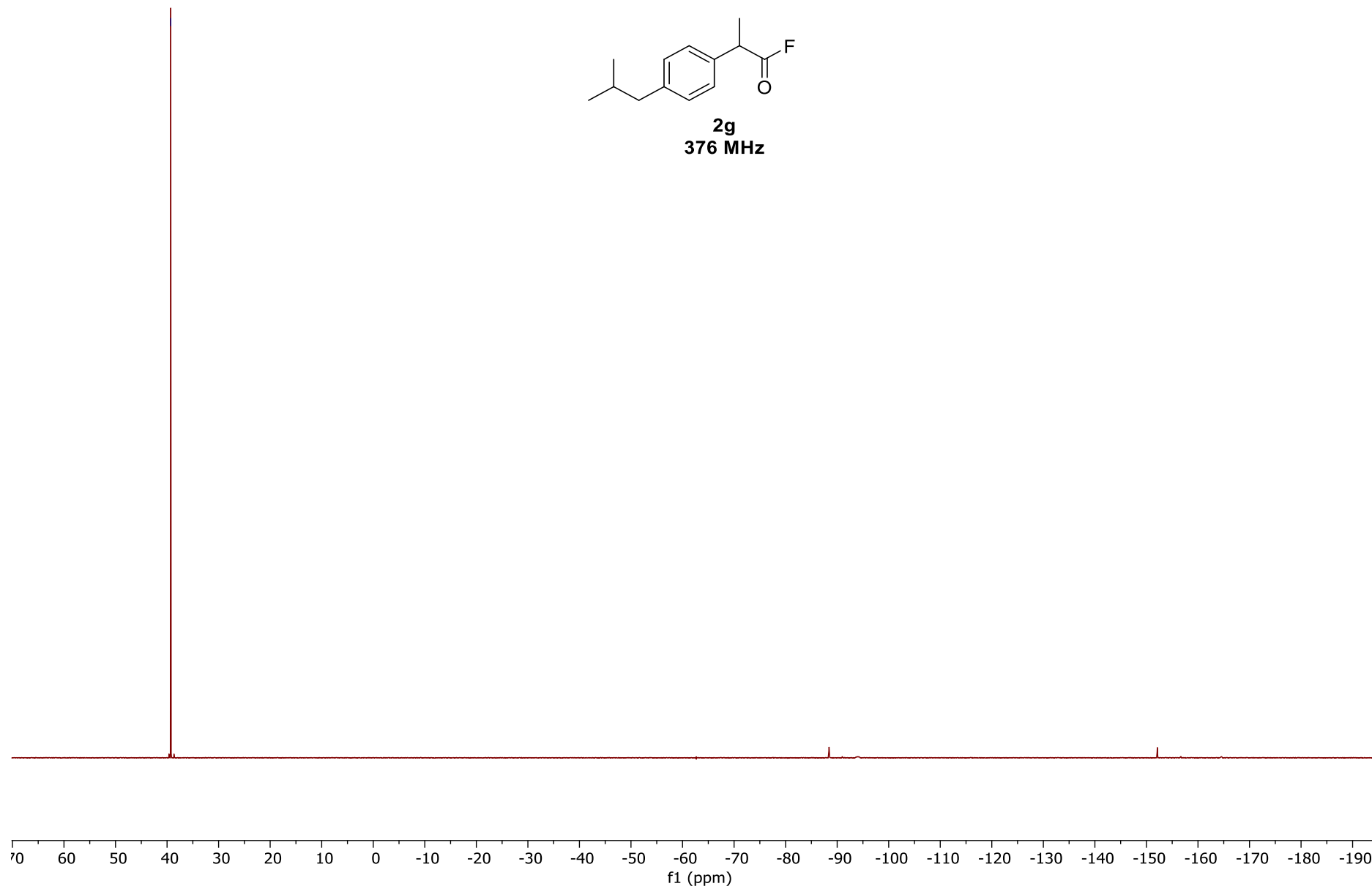
**2g**  
400 MHz



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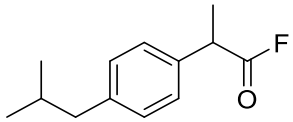


**2g**  
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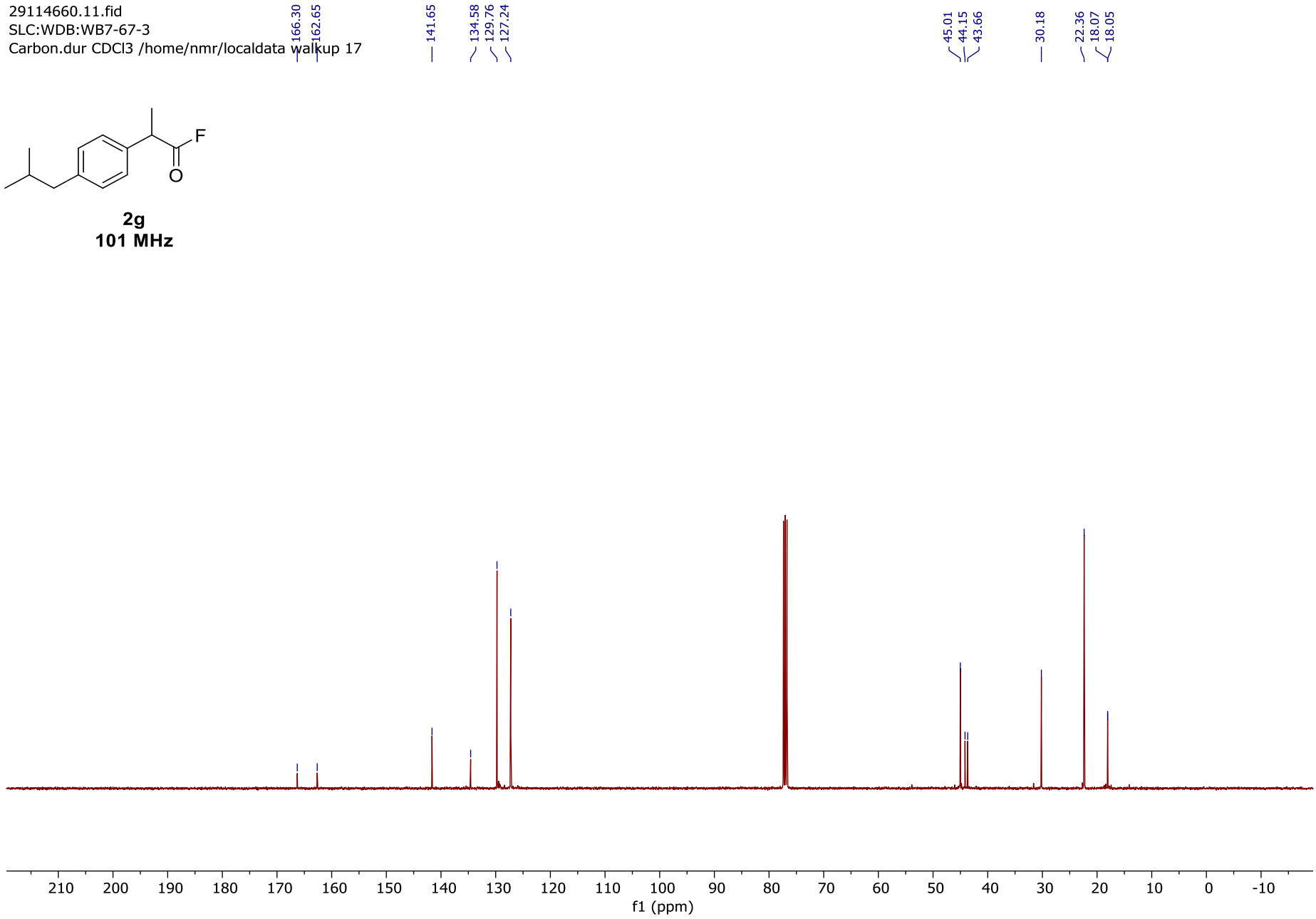




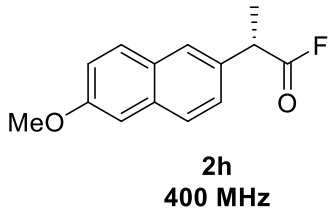
29114660.11.fid  
SLC:WDB:WB7-67-3  
Carbon.dur CDCl3 /home/nmr/localdata walkup 17



**2g**  
**101 MHz**



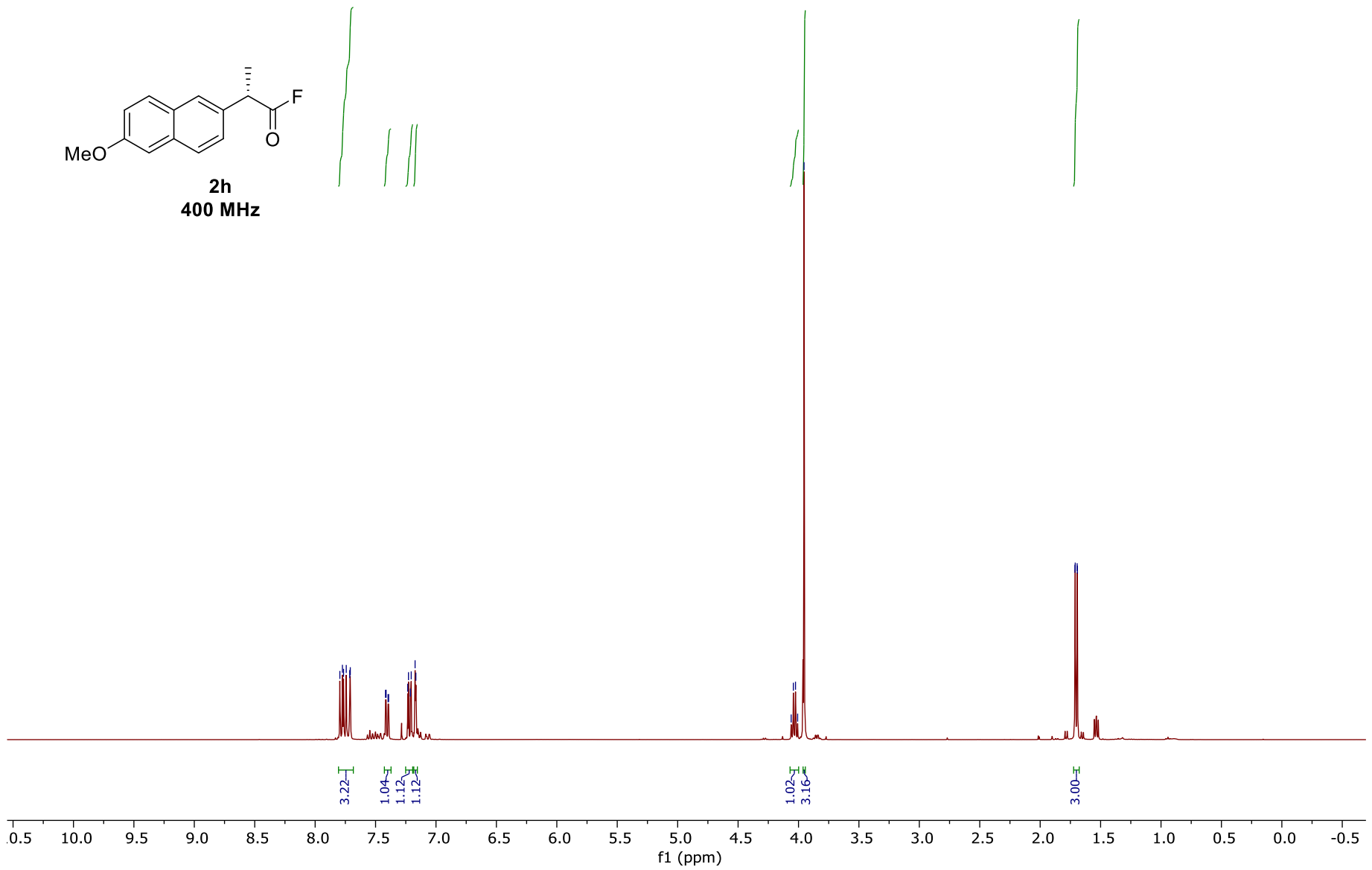
29114663.10.1.1r  
SLC:WDB:WB7-67-4  
Proton.dur CDCl3 /home/nmr/localdata walkup 20



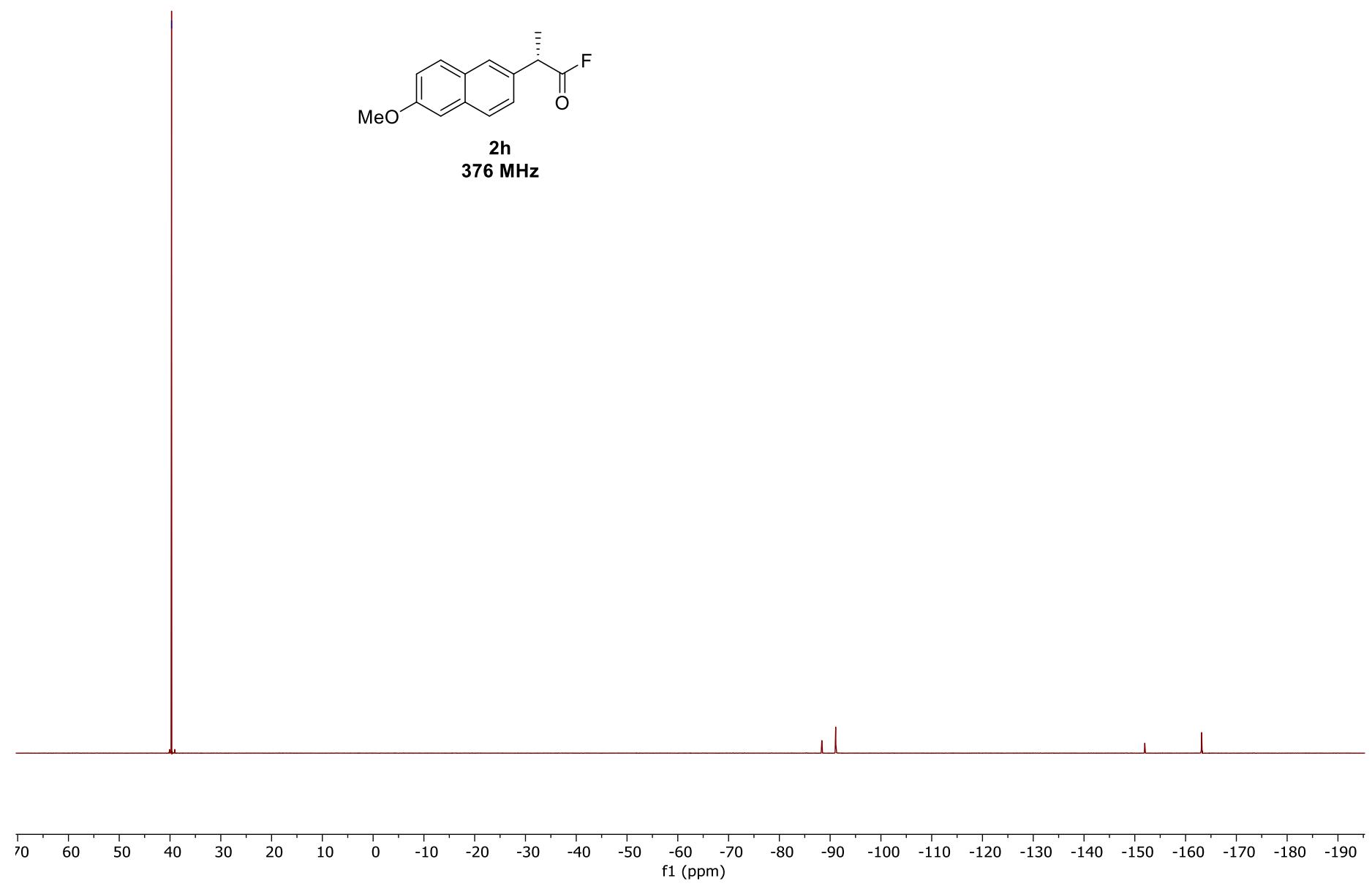
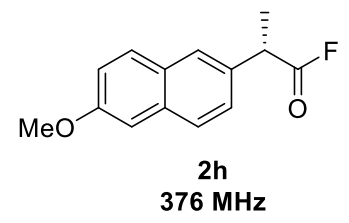
7.80  
7.77  
7.76  
7.74  
7.71  
7.71  
7.42  
7.41  
7.40  
7.39  
7.23  
7.23  
7.21  
7.21  
7.17  
7.17

4.06  
4.04  
4.02  
4.01  
3.95

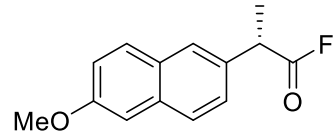
1.71  
1.71  
1.69  
1.69



29114663.14.fid  
SLC:WDB:WB7-67-49.70  
F19\_limits\_dec.dur CDCl3 /home/nmr/localdata walkup 20



29114663.11.fid  
SLC:WDB:WB7-67-4  
Carbon.dur CDCl3 /home/nmr/localdata/walkup 20



2h  
101 MHz

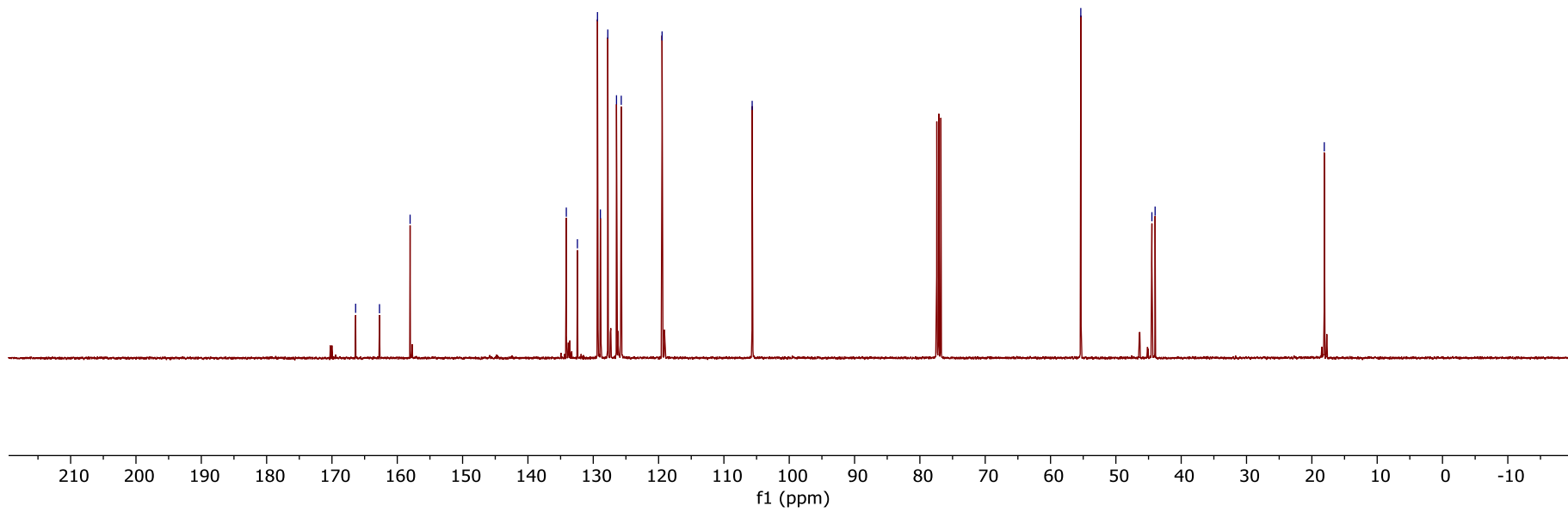
134.12  
132.42  
129.35  
128.91  
127.78  
126.44  
125.71  
119.46

105.67

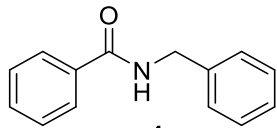
55.37

44.48  
43.99

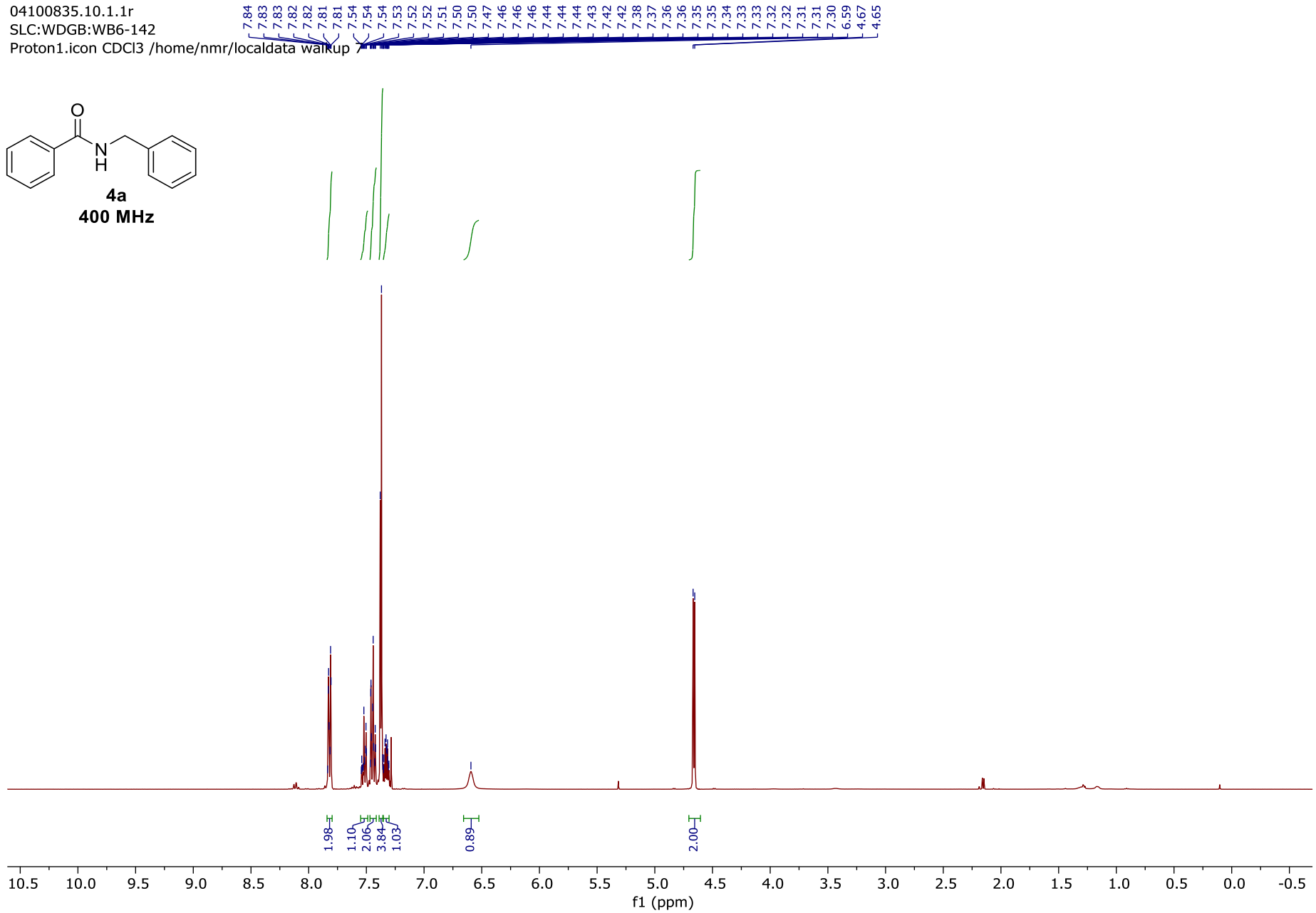
18.10



04100835.10.1.1r  
SLC:WDGB:WB6-142  
Proton1.icon CDCl3 /home/nmr/localdata walkup 7



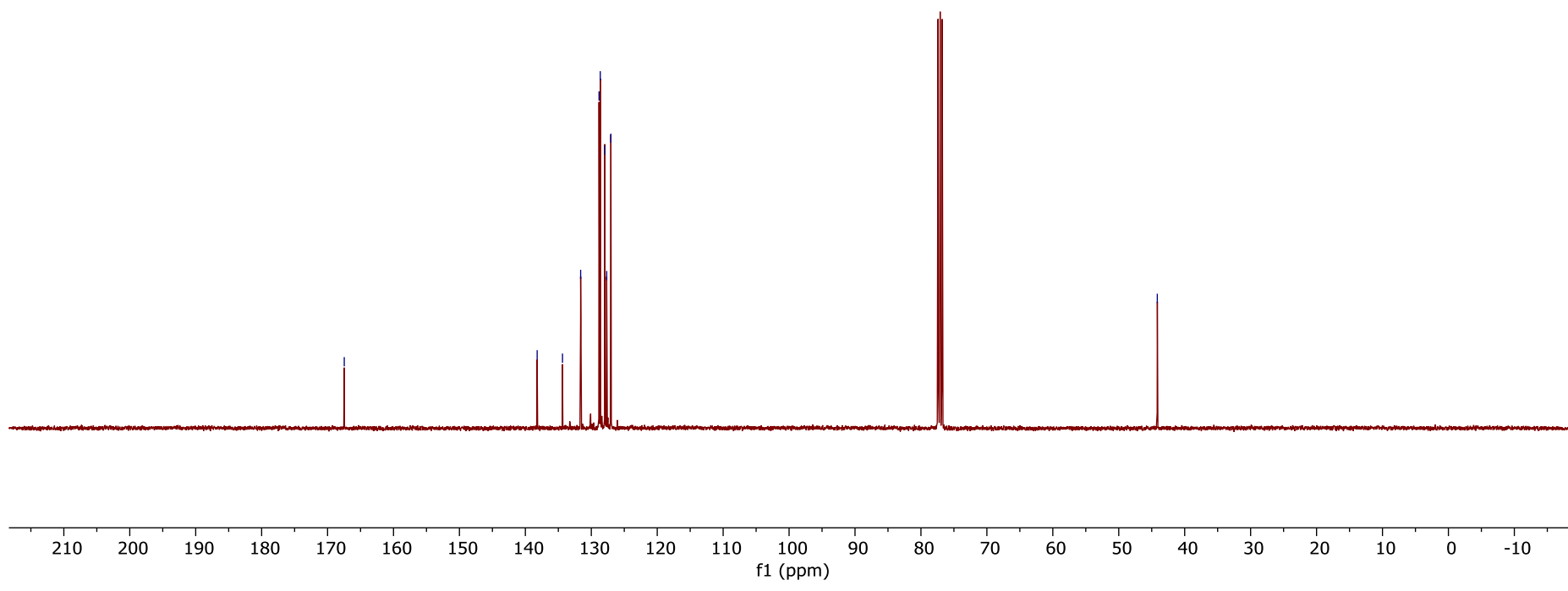
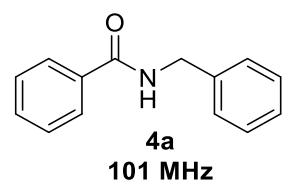
400 MHz



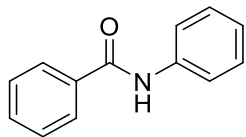
04100835.11.1.1r  
SLC:WDGB:WB6-142  
Carbon.dur CDCl3 /home/nmr/localdata/walkup 7

138.21  
134.37  
131.61  
128.83  
128.64  
127.96  
127.66  
127.03

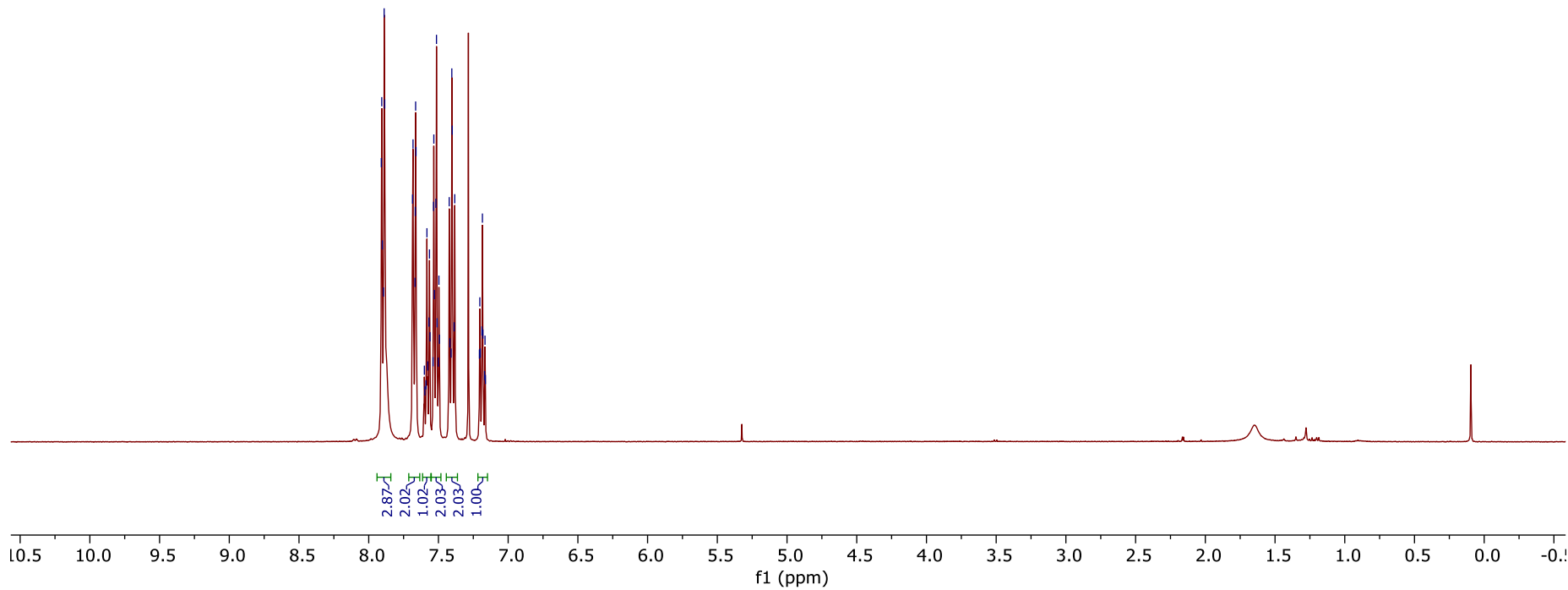
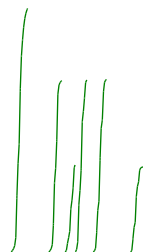
44.15



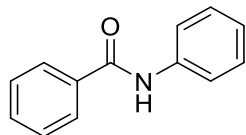
111233907  
SLC:WDGB:WB6-151  
Proton.dur CDCl3 /home/nmr/localdata/wakup 23



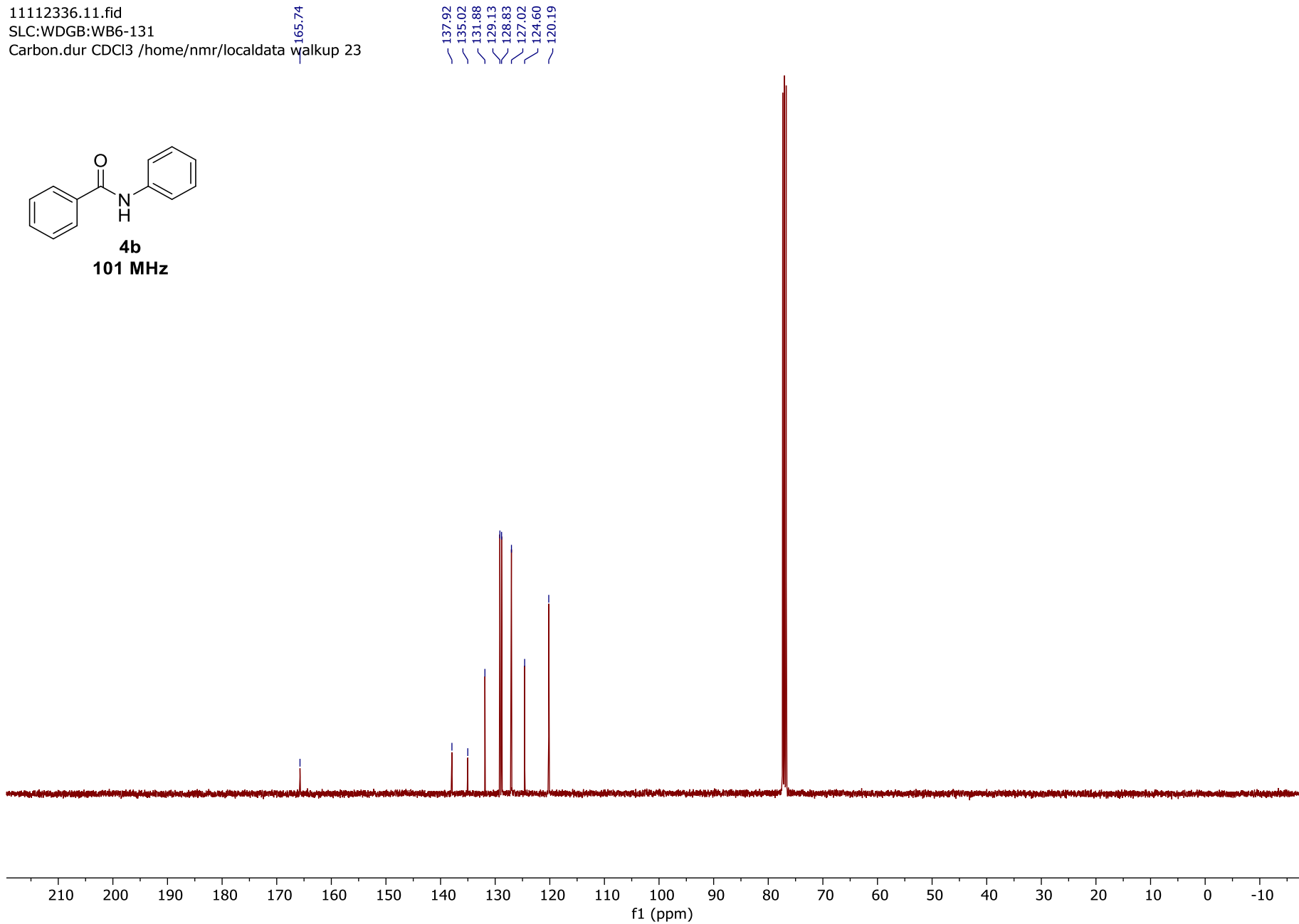
**4b**  
400 MHz



11112336.11.fid  
SLC:WDGB:WB6-131  
Carbon.dur CDCl3 /home/nmr/localdata walkup 23

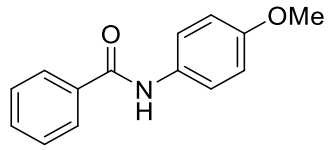


**4b**  
**101 MHz**

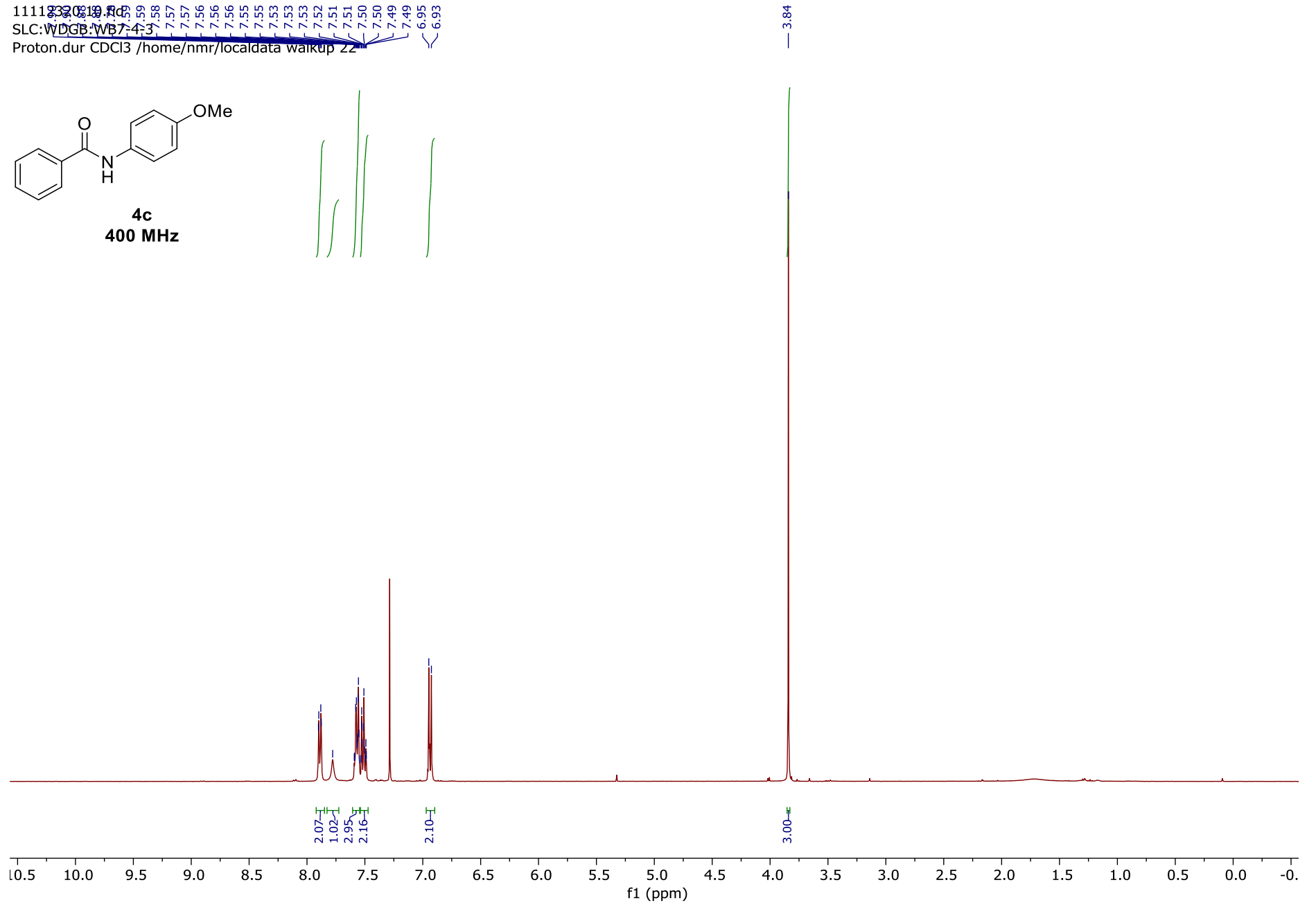




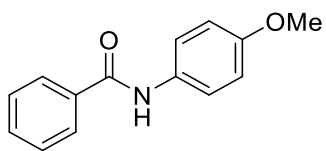
11118320818.F  
SLC:WDGB:WB7-4-3  
Proton.dur CDCl3 /home/nmr/localdata/wakup 22



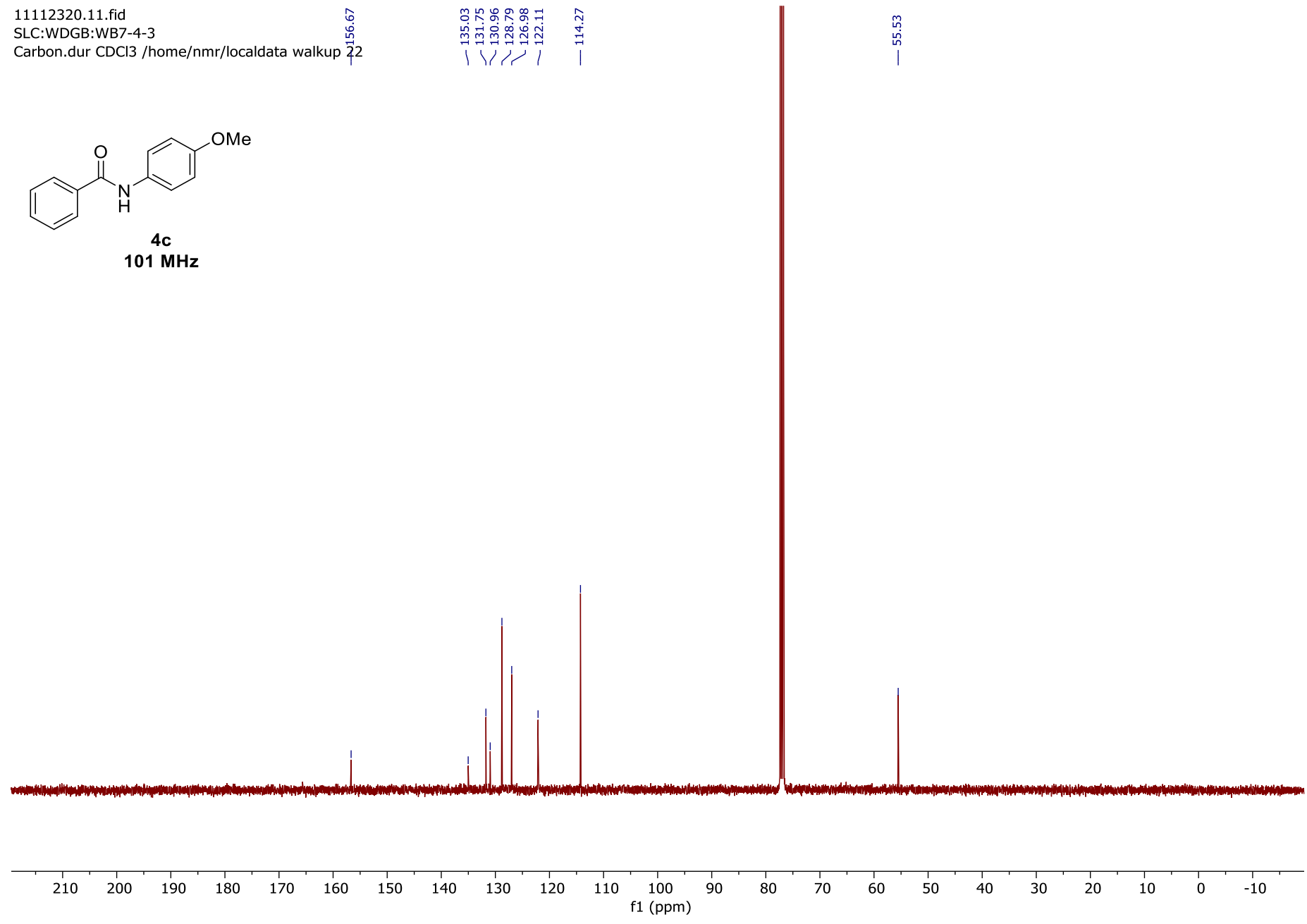
**4c**  
400 MHz



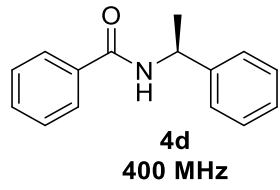
11112320.11.fid  
SLC:WDGB:WB7-4-3  
Carbon.dur CDCl3 /home/nmr/localdata walkup 22



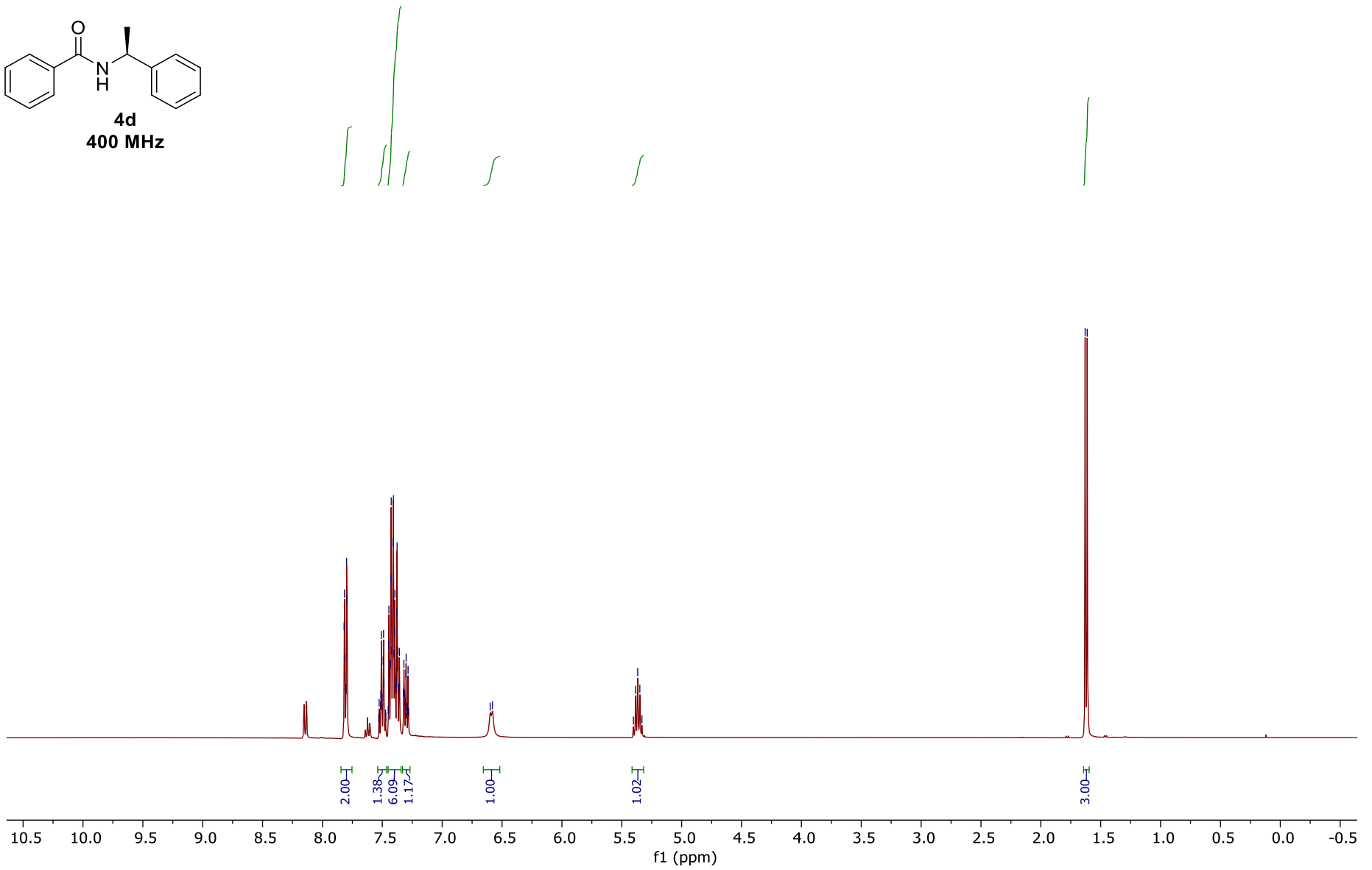
**4c**  
101 MHz



11112353.10.fid  
SLC:WDGB:WB7-4-4  
Proton.dur CDCl3 /home/nmr/localdata walkup 24



7.82 7.82 7.81 7.80 7.79 7.53 7.53 7.52 7.51 7.51 7.51 7.50 7.49 7.49 7.47 7.47 7.45 7.45 7.44 7.44 7.43 7.43 7.43 7.42 7.41 7.41 7.40 7.40 7.39 7.39 7.38 7.38 7.38 7.37 7.37 7.36 7.36 7.32 7.32 7.31 7.31 7.30 7.30 7.30 7.29 7.28 7.28 6.60 6.58 5.40 5.38 5.37 5.35 5.33 1.63 1.61

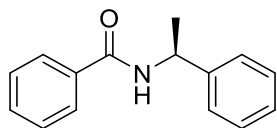


11112353.11.fid  
SLC:WDGB:WB7-4-4  
Carbon.dur CDCl3 /home/nmr/localdata walkup 24

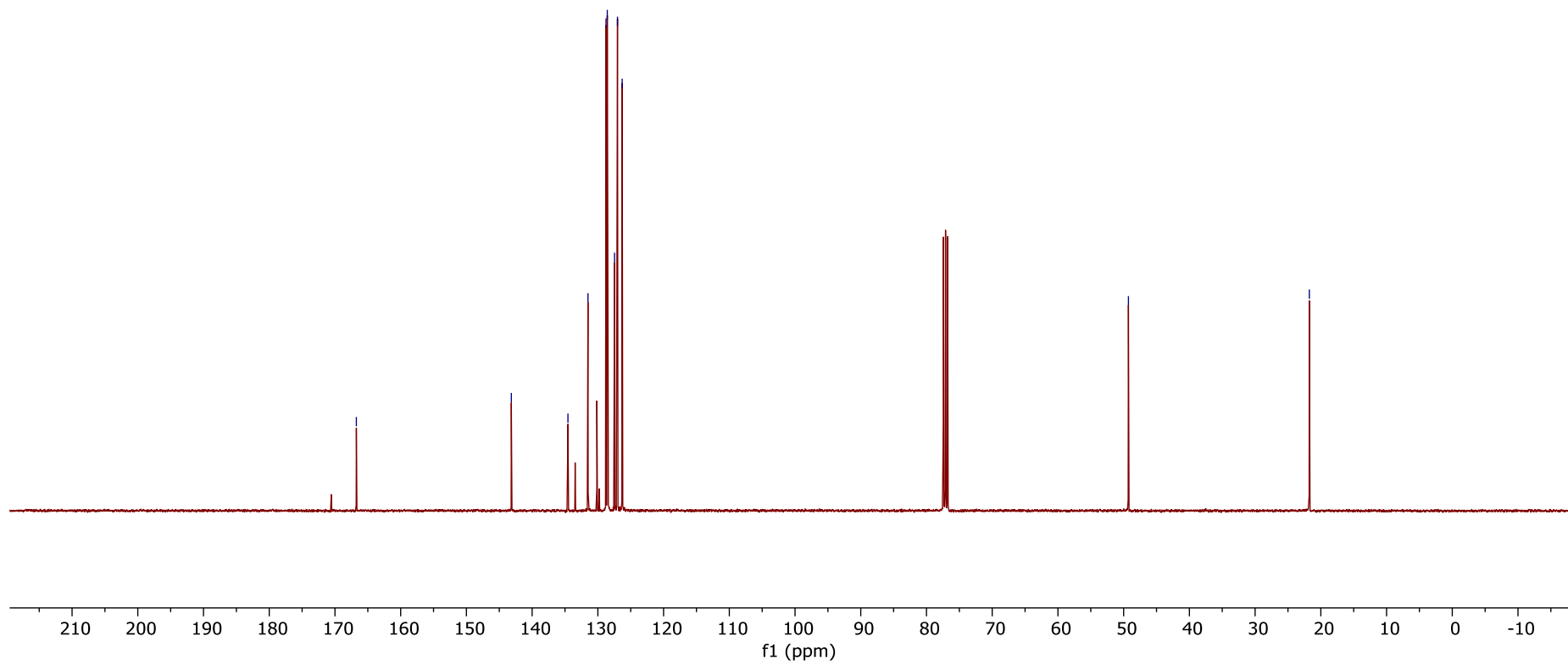
143.17  
134.55  
131.50  
128.76  
128.57  
127.46  
127.01  
126.30

49.27

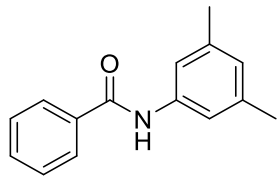
21.75



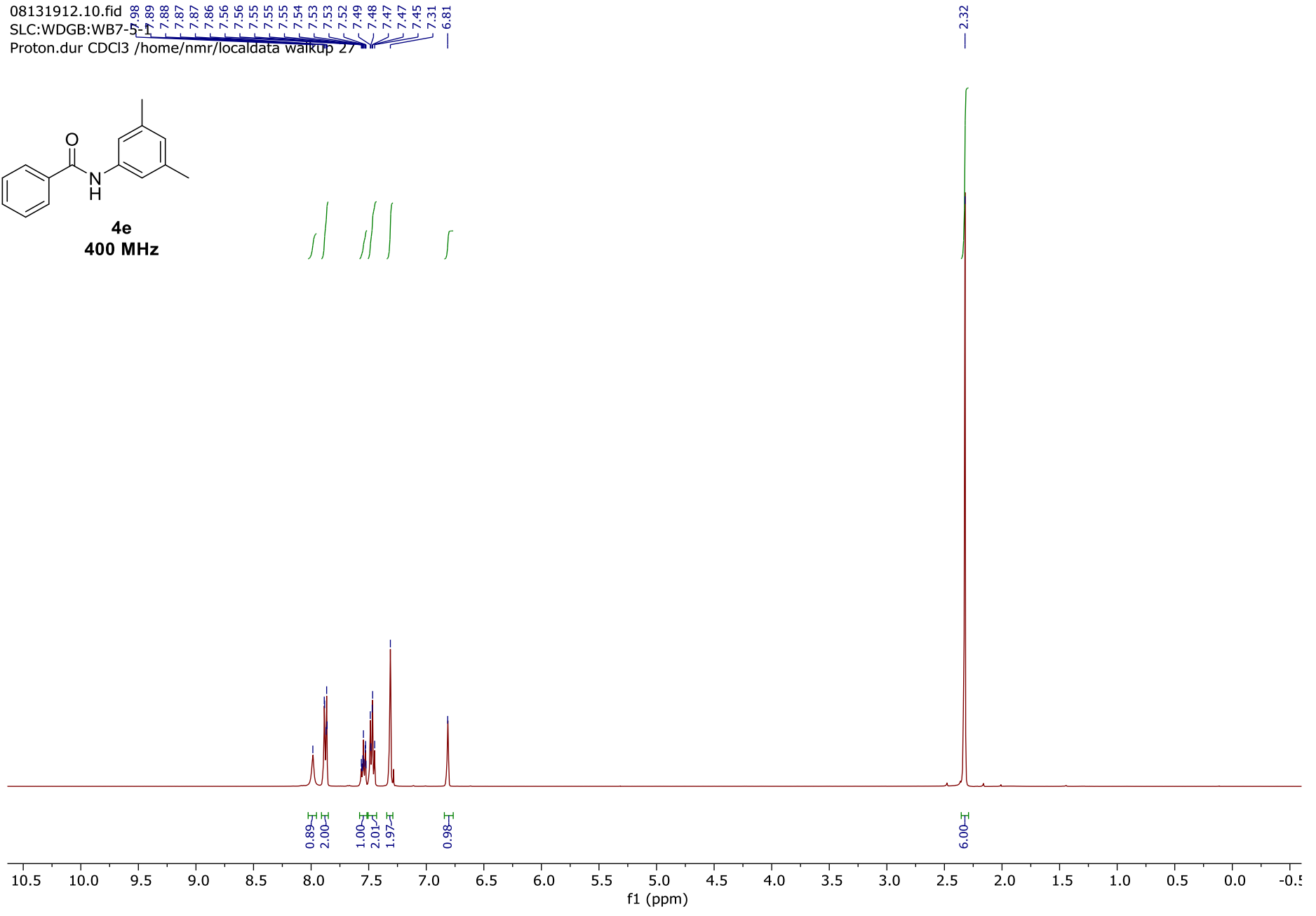
**4d**  
101 MHz



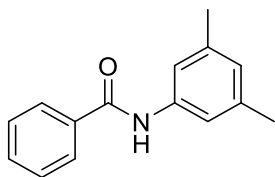
08131912.10.fid  
SLC:WDGB:WB7-5  
Proton.dur CDCl3 /home/nmr/localdata/waikup 27



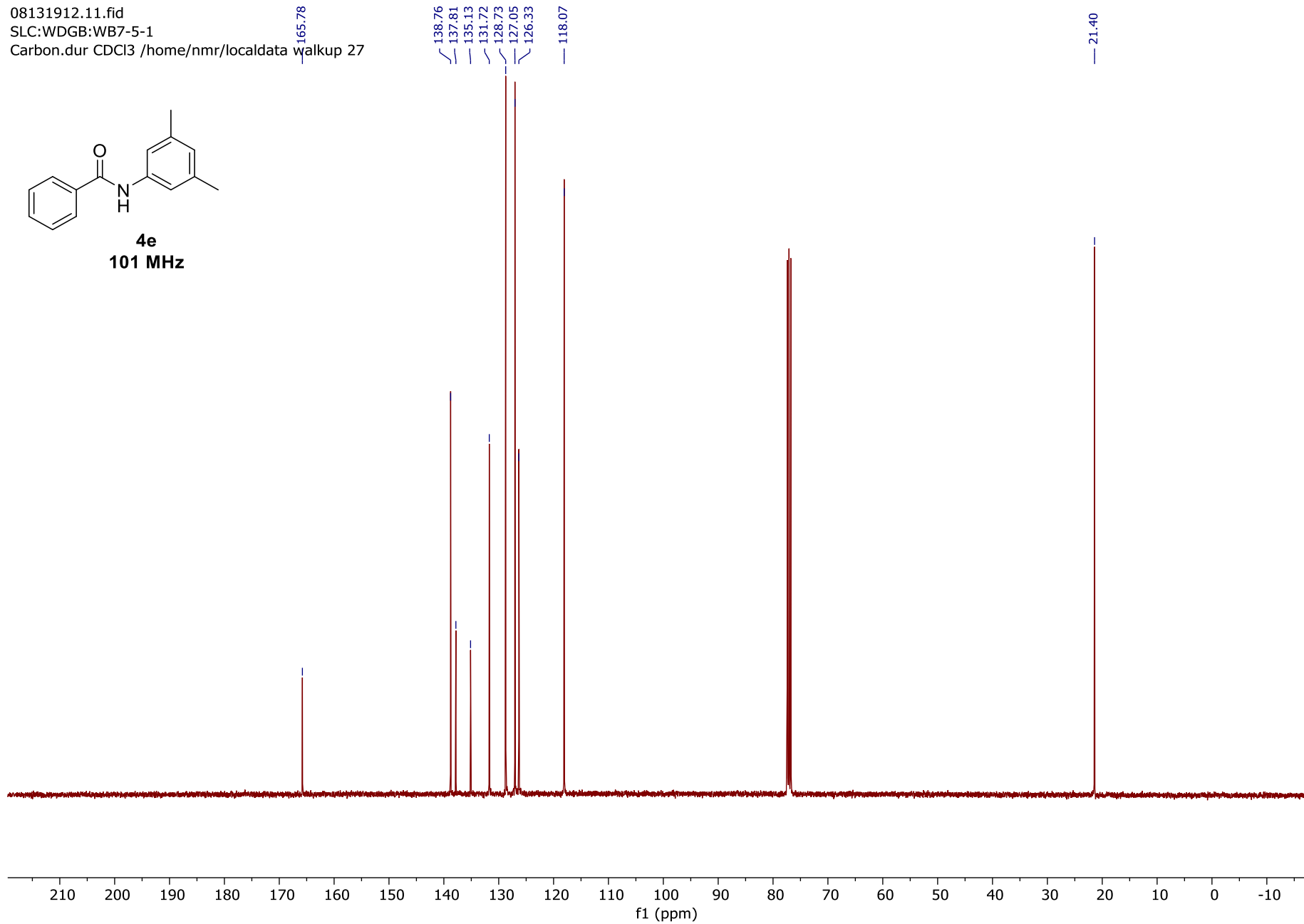
**4e**  
**400 MHz**



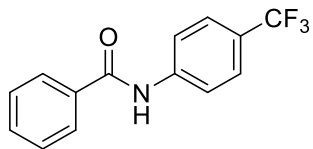
08131912.11.fid  
SLC:WDGB:WB7-5-1  
Carbon.dur CDCl3 /home/nmr/localdata walkup 27



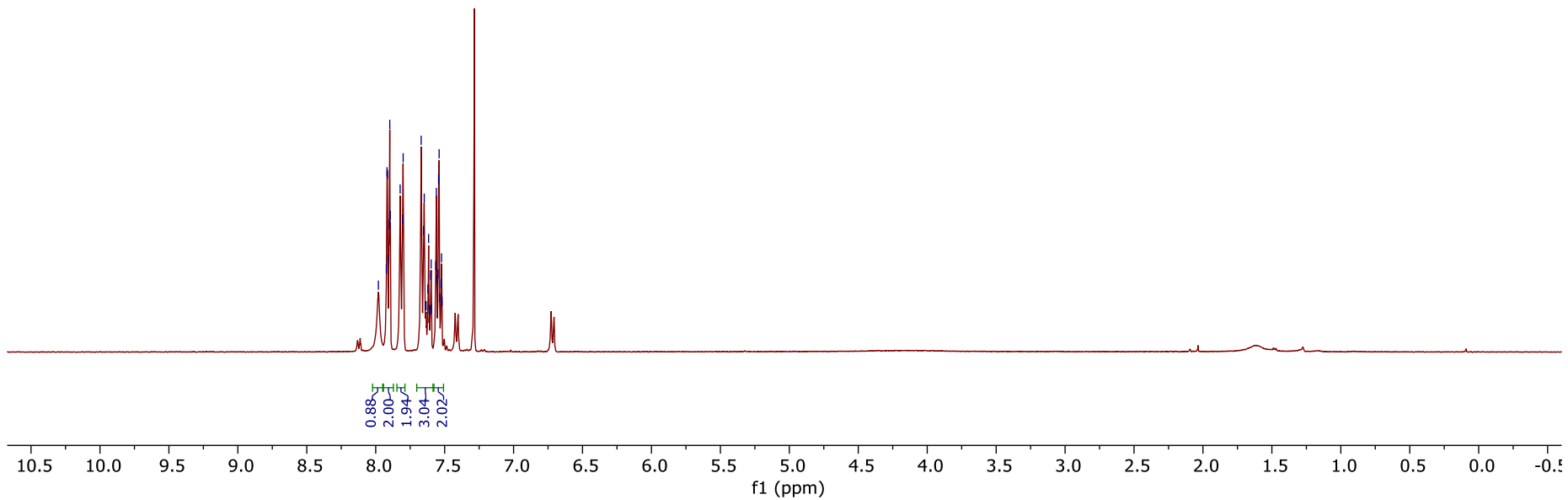
**4e**  
**101 MHz**



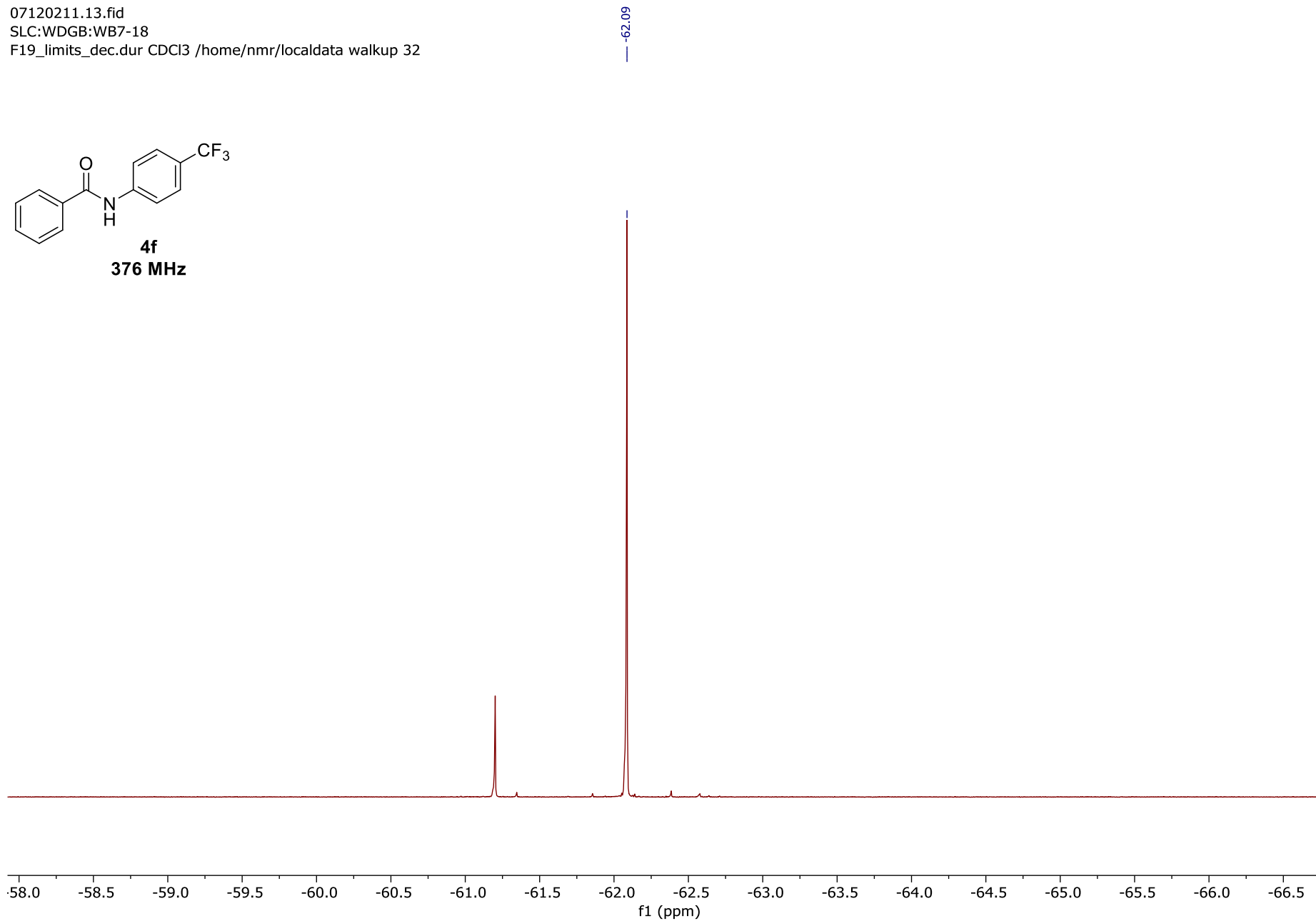
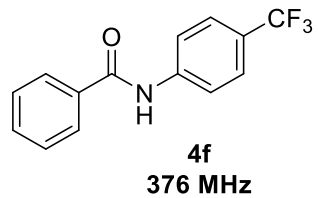
07120211.10816  
H NMR (400 MHz, CDCl<sub>3</sub>)  
SLC.WDGB:WB7-18  
Proton.dup CDCl<sub>3</sub> /home/nmr/localdata/waikup/32  
7.51 (m, 1H), 7.50 (m, 2H), 7.58



4f  
400 MHz

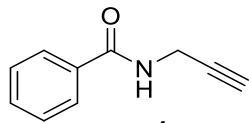


07120211.13.fid  
SLC:WDGB:WB7-18  
F19\_limits\_dec.dur CDCl3 /home/nmr/localdata walkup 32

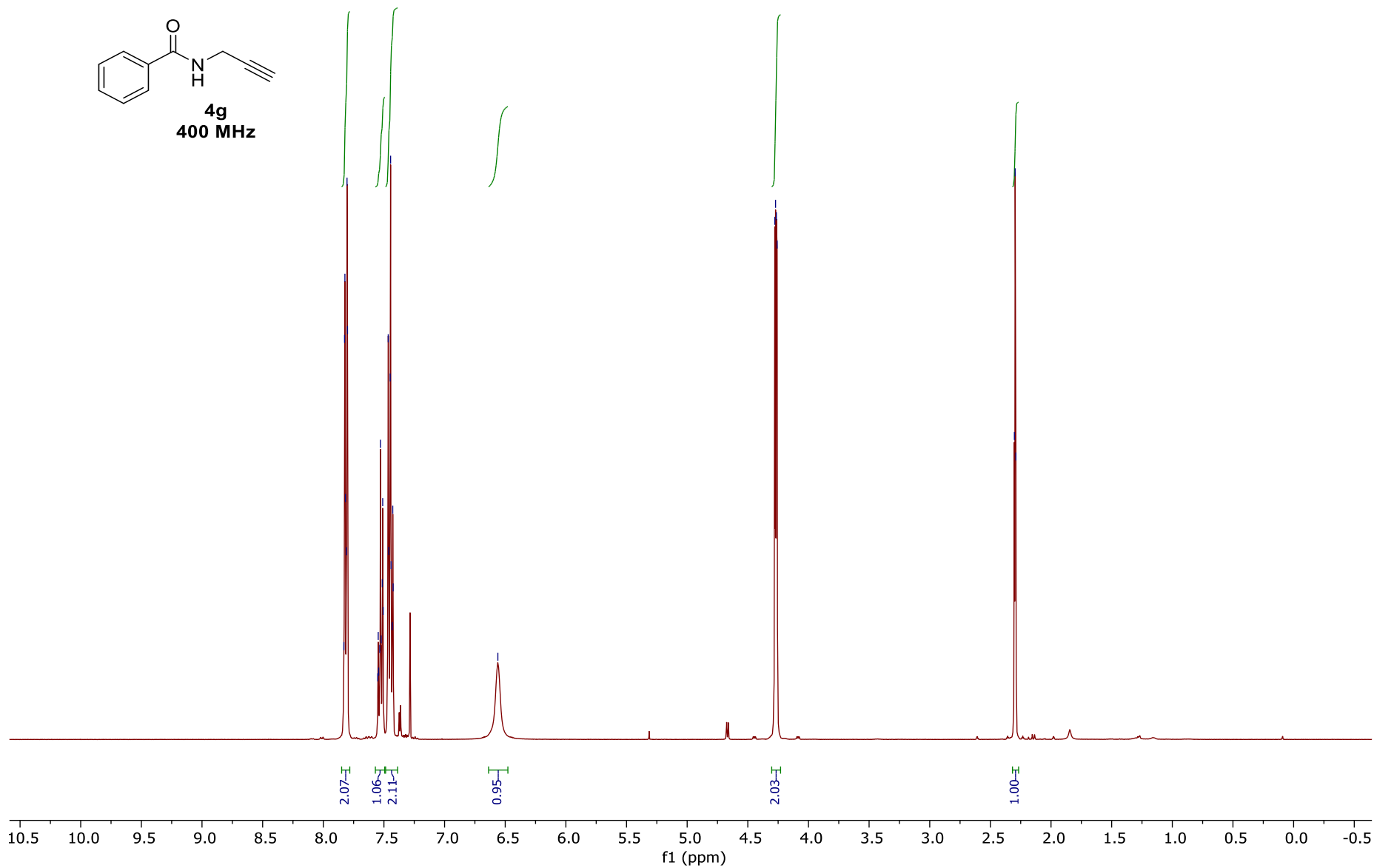




08123621.10.02  
H NMR (400 MHz, CDCl<sub>3</sub>)  
SLC.WDGB.WB776  
Proton 1: icon CDCl<sub>3</sub> /home/nmr/localdata/wakup 9  
(s, 1H), 4.27 (d, 2H), 2.30 (s, 3H), 2.29 (s, 3H), 7.49 – 7.40 (m, 2H), 6.56 (s, 1H).



**4g**  
400 MHz

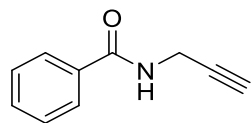


08123621.11.fid  
SLC:WDGB:WB7-76  
Carbon.dur CDCl3 /home/nmr/localdata/walkup 9

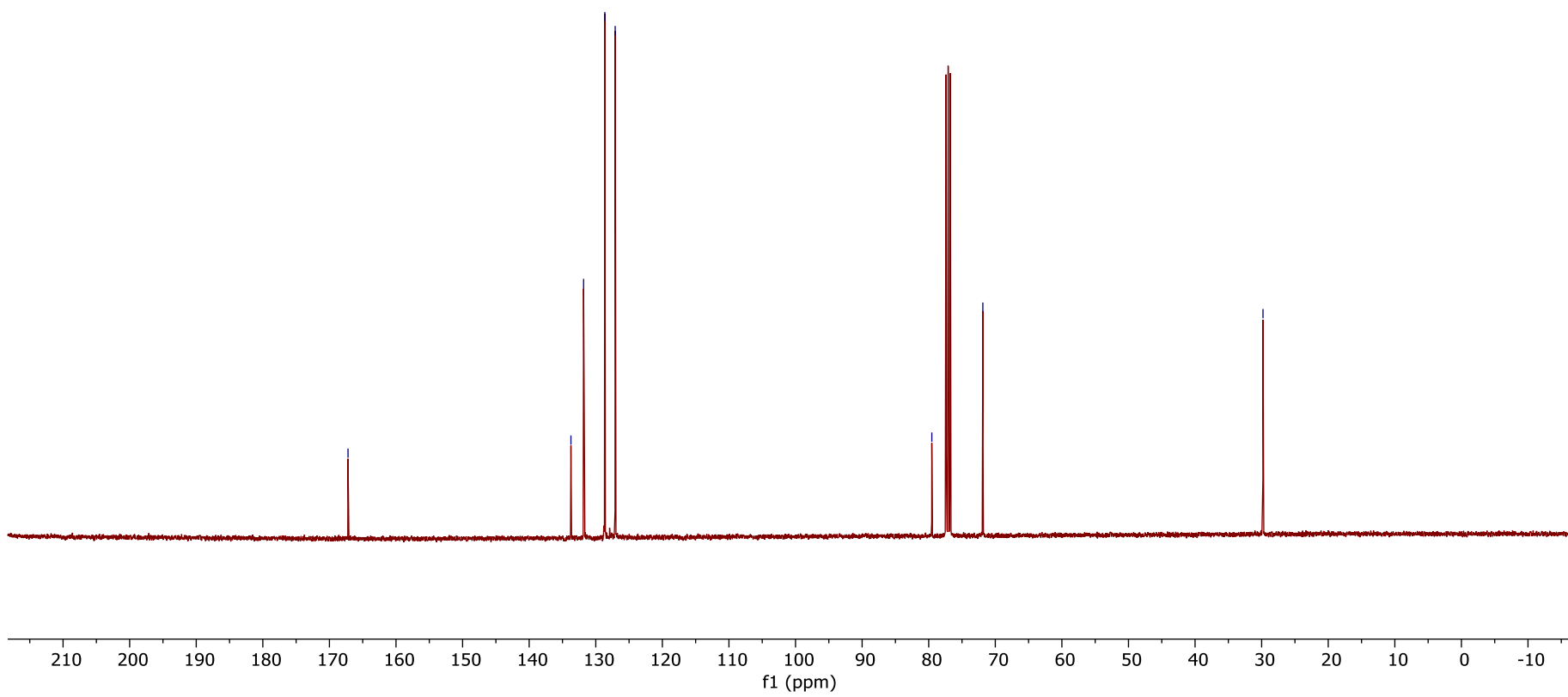
133.73  
131.83  
128.64  
127.08

79.53  
71.87

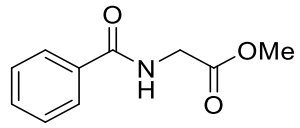
29.80



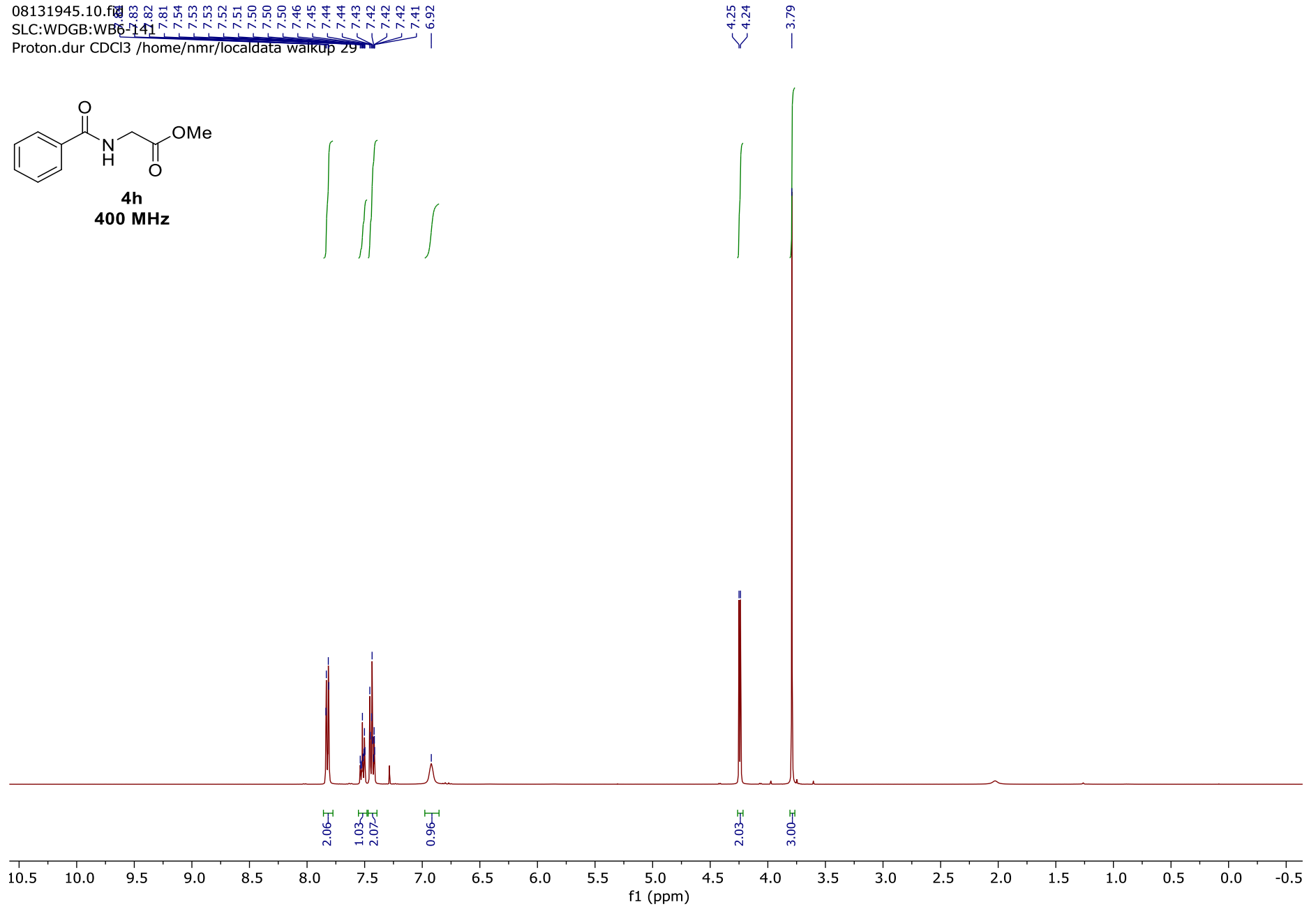
**4g**  
**101 MHz**



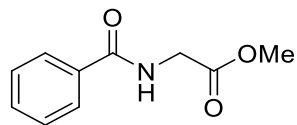
08131945.10.f1  
SLC:WDGB:WB6-141  
Proton.dur CDCl3 /home/nmr/localdata/wakup\_29



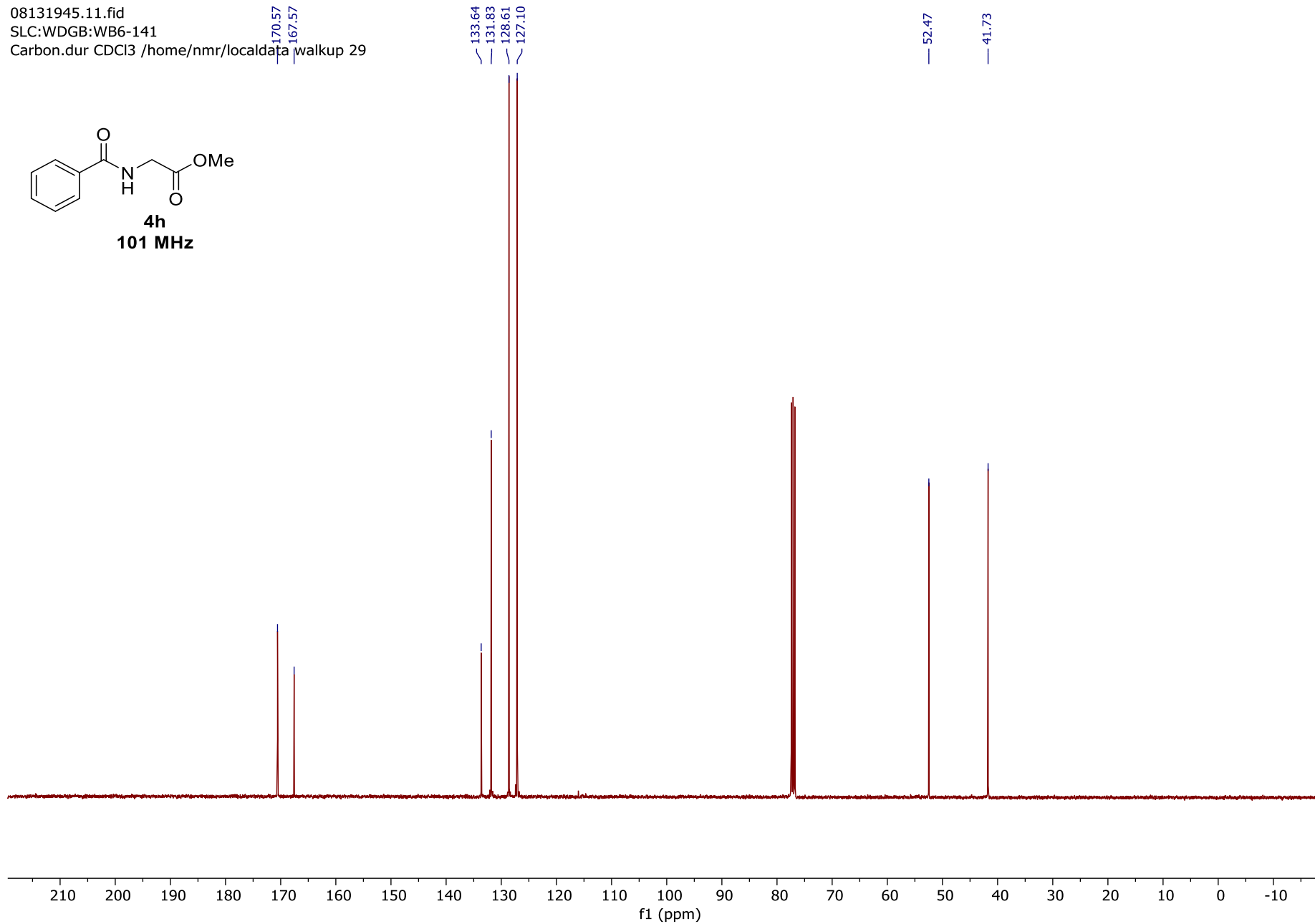
4h  
400 MHz



08131945.11.fid  
SLC:WDGB:WB6-141  
Carbon.dur CDCl3 /home/nmr/localdata/walkup 29



4h  
101 MHz

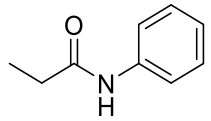


07173716.10.fid  
SLC:WDGB:WB7-10-1  
Proton.dur CDCl3 /home/nmr/localdata walkup 25

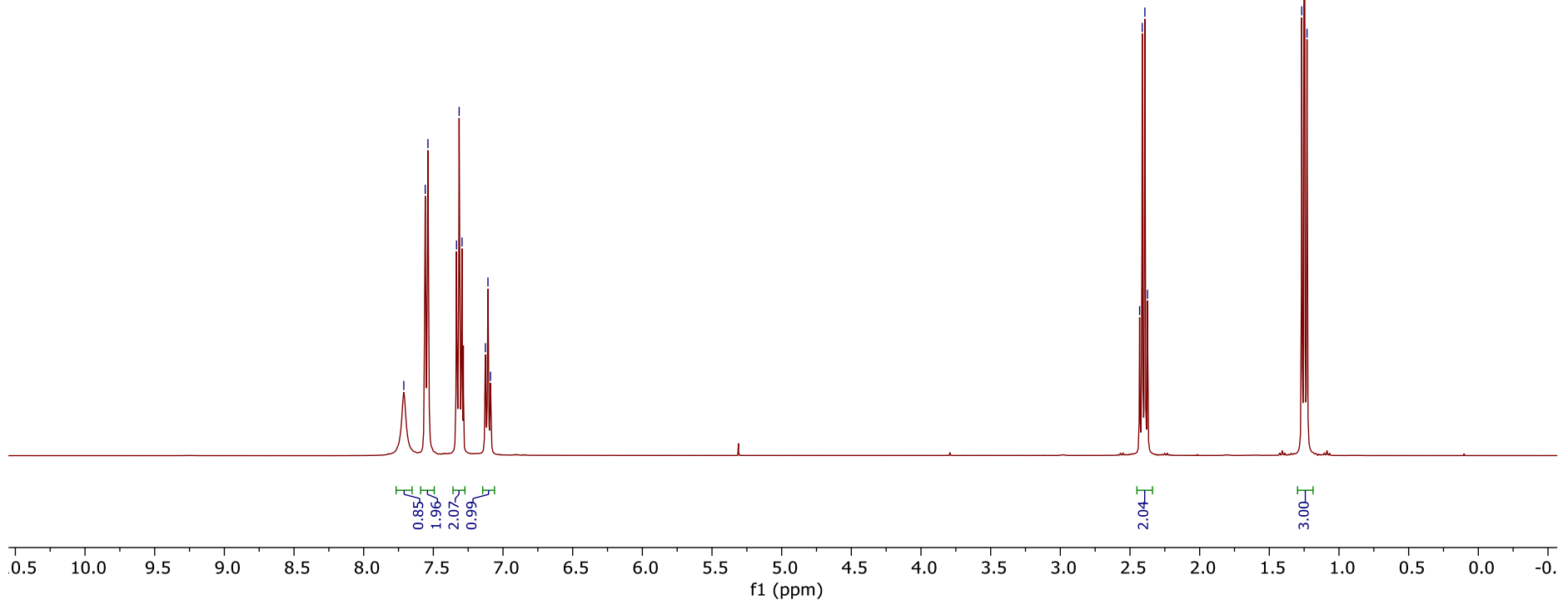
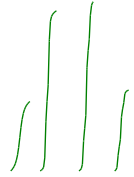
7.71  
7.56  
7.54  
7.33  
7.31  
7.29  
7.13  
7.11  
7.09

2.43  
2.41  
2.39  
2.37

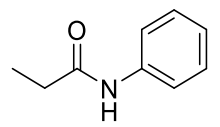
1.27  
1.25  
1.23



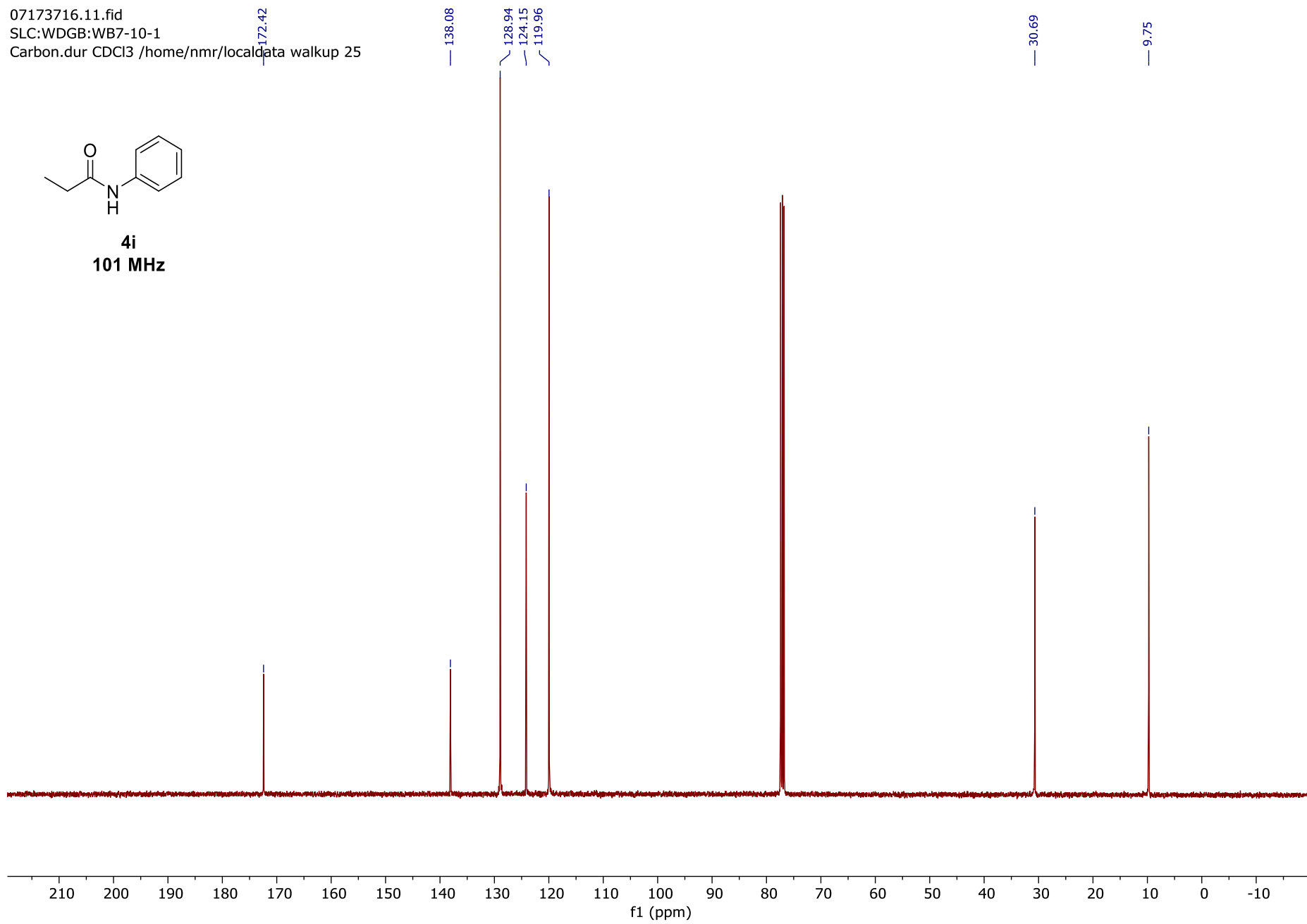
4i  
400 MHz



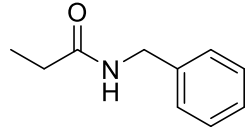
07173716.11.fid  
SLC:WDGB:WB7-10-1  
Carbon.dur CDCl3 /home/nmr/localdata walkup 25



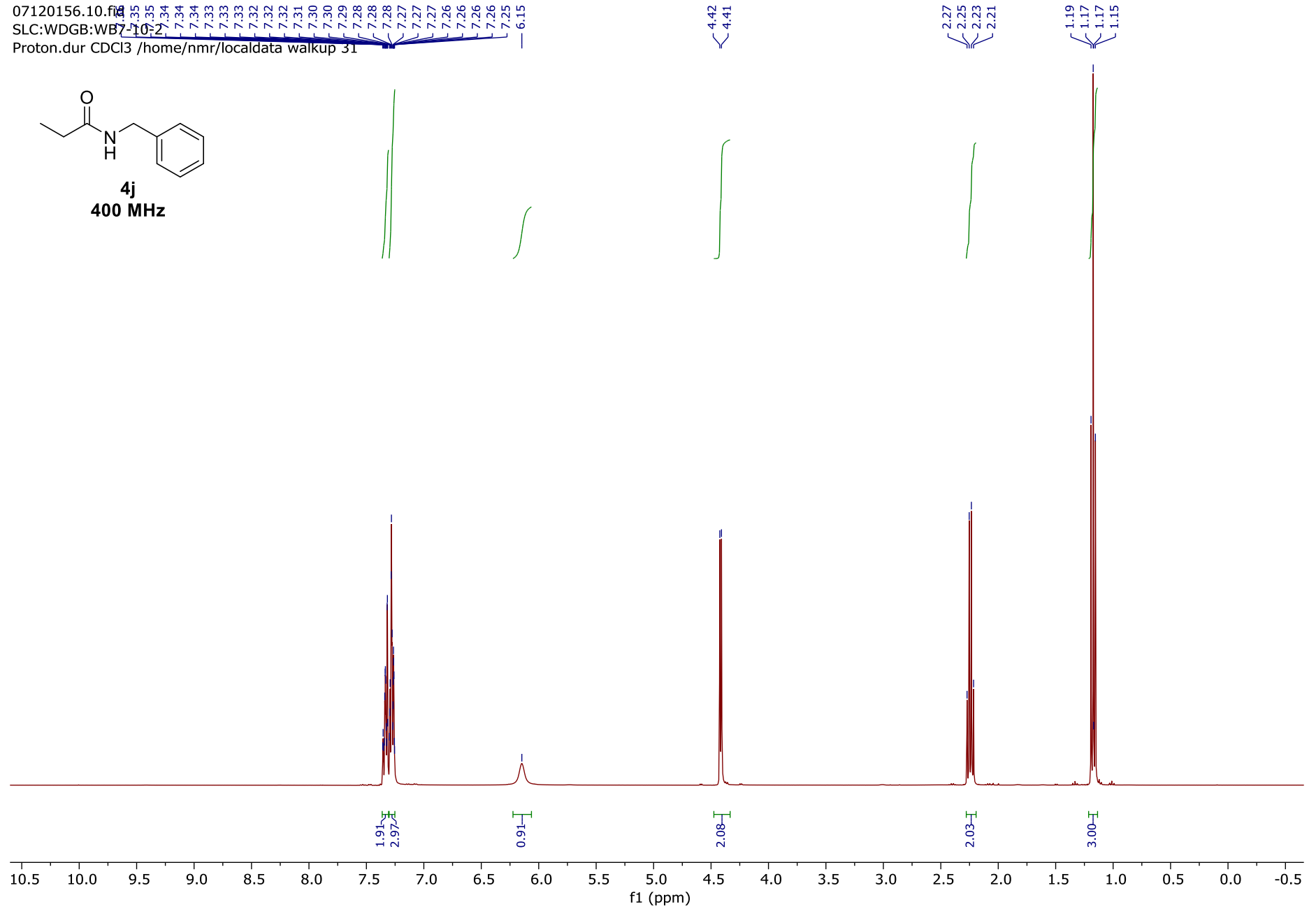
4i  
101 MHz



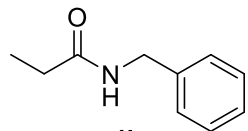
07120156.10.fid  
SLC:WDGB:WB7-10-2  
Proton.dur CDCl3 /home/nmr/localdata walkup 31



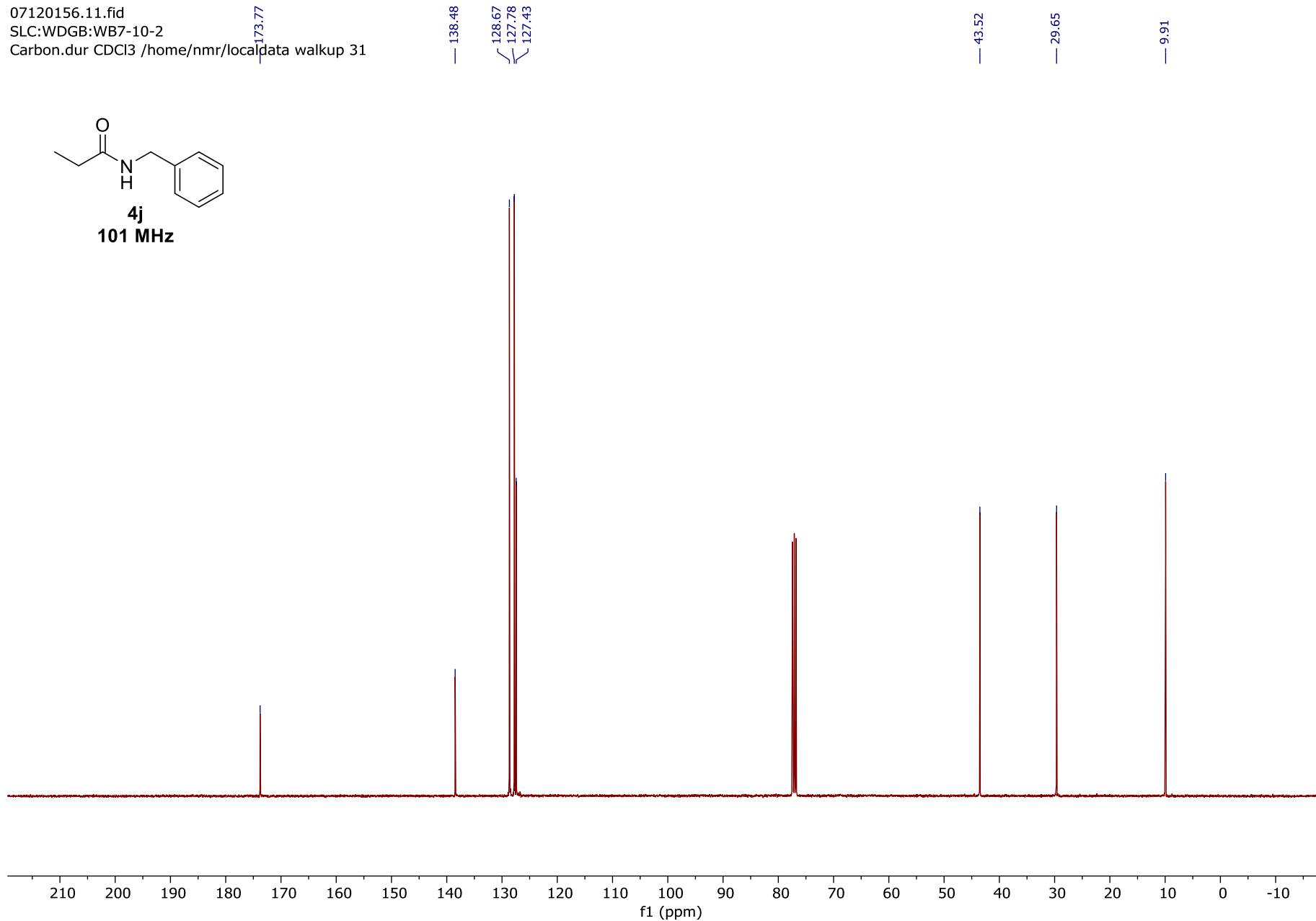
**4j**  
400 MHz



07120156.11.fid  
SLC:WDGB:WB7-10-2  
Carbon.dur CDCl3 /home/nmr/localdata walkup 31

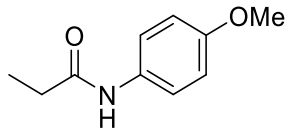


**4j**  
**101 MHz**

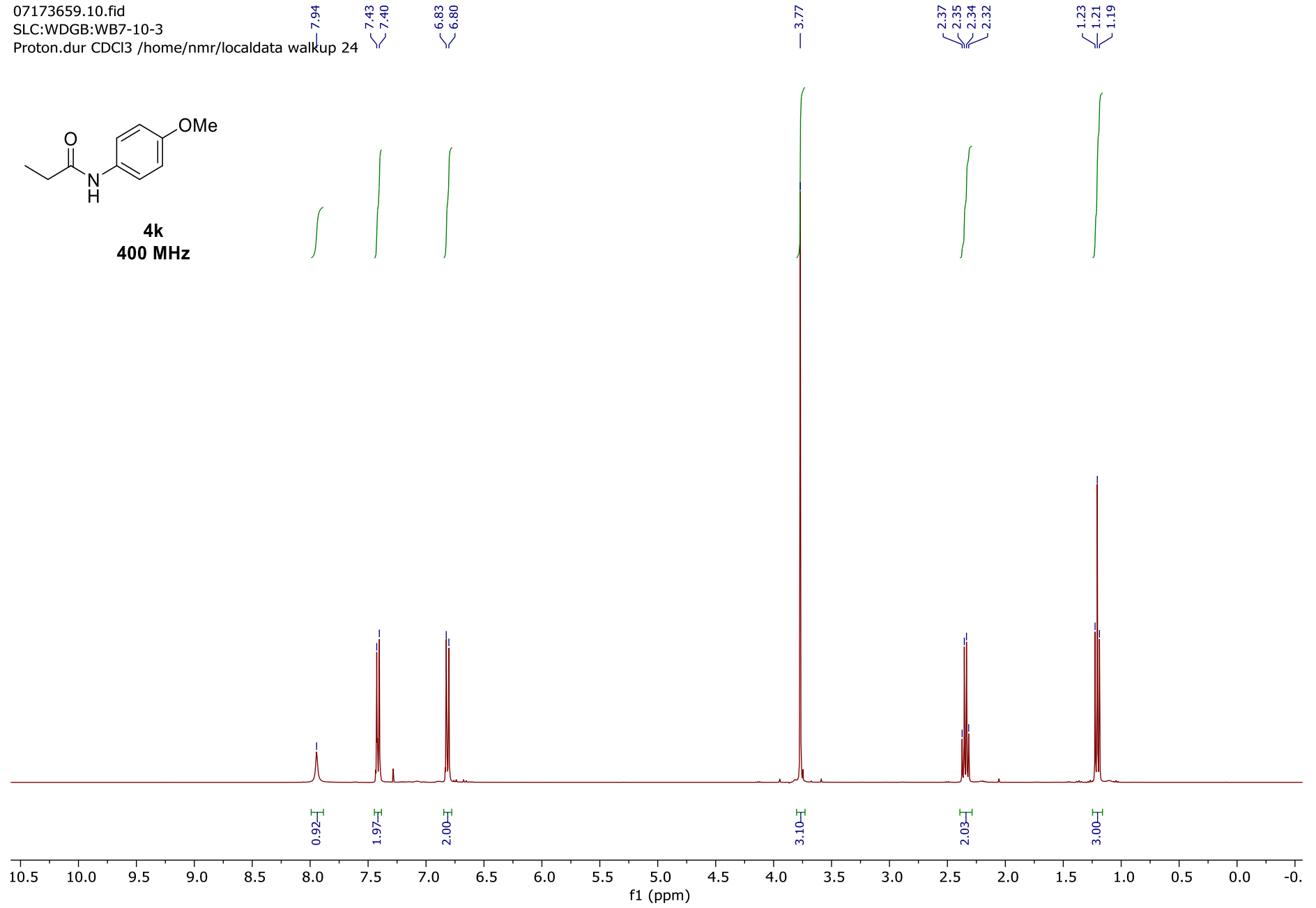




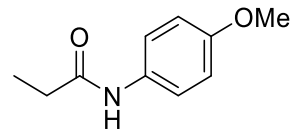
07173659.10.fid  
SLC:WDGB:WB7-10-3  
Proton.dur CDCl3 /home/nmr/localdata walkup 24



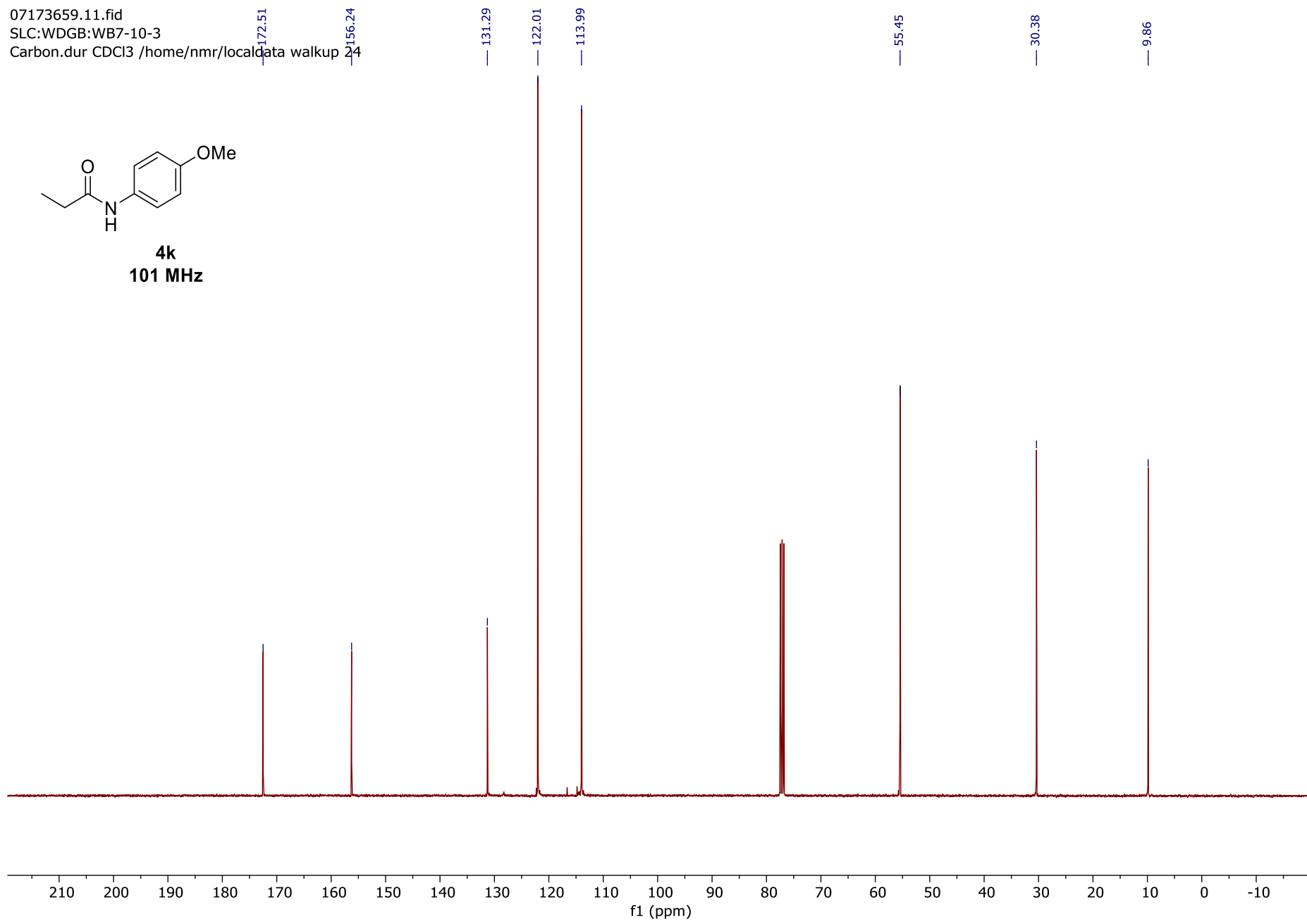
4k  
400 MHz



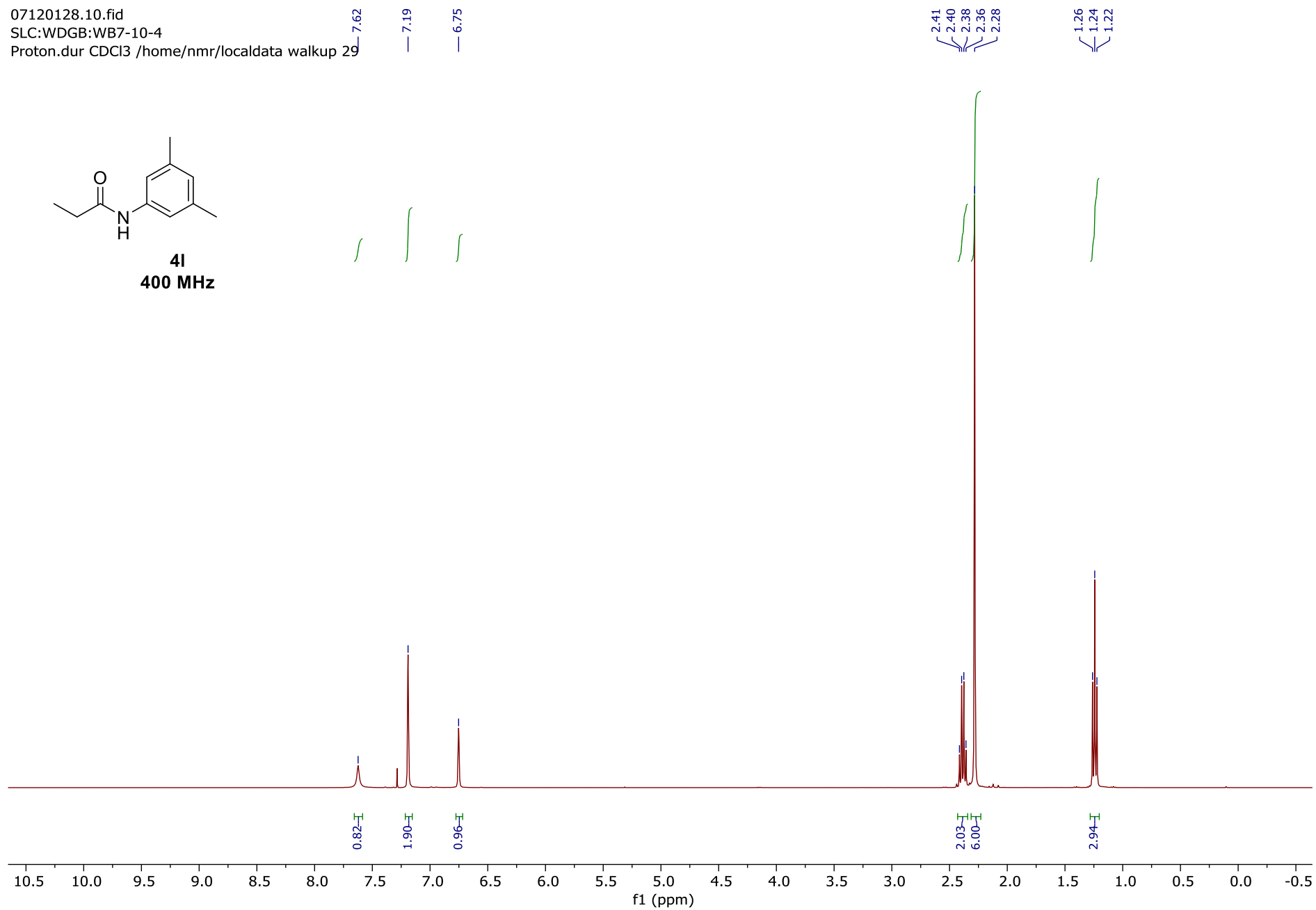
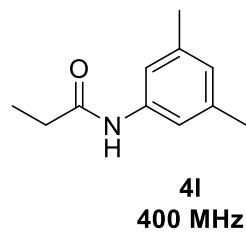
07173659.11.fid  
SLC:WDGB:WB7-10-3  
Carbon.dur CDCl3 /home/nmr/localdata walkup 24



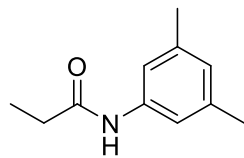
**4k**  
**101 MHz**



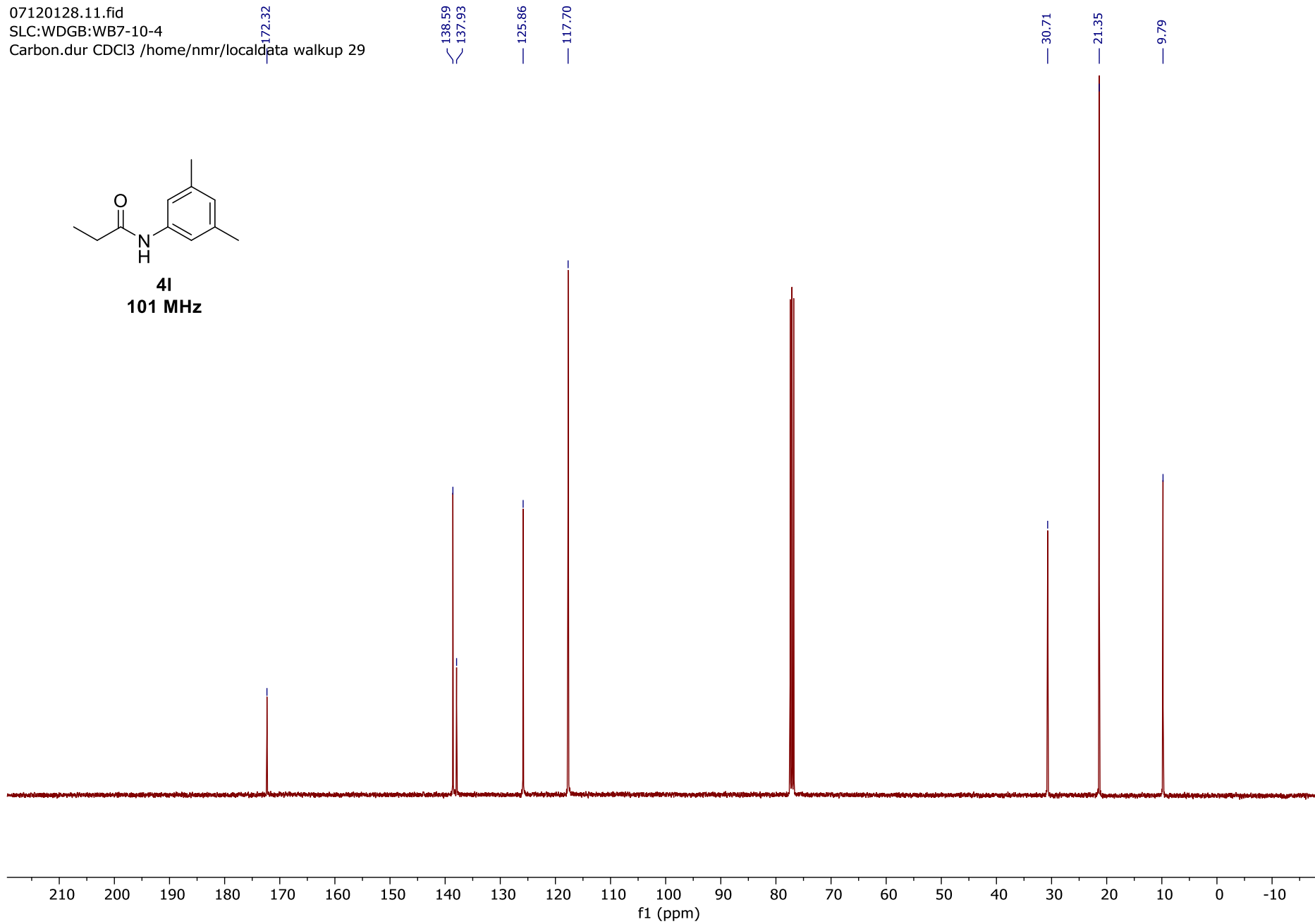
07120128.10.fid  
SLC:WDGB:WB7-10-4  
Proton.dur CDCl3 /home/nmr/localdata walkup 29



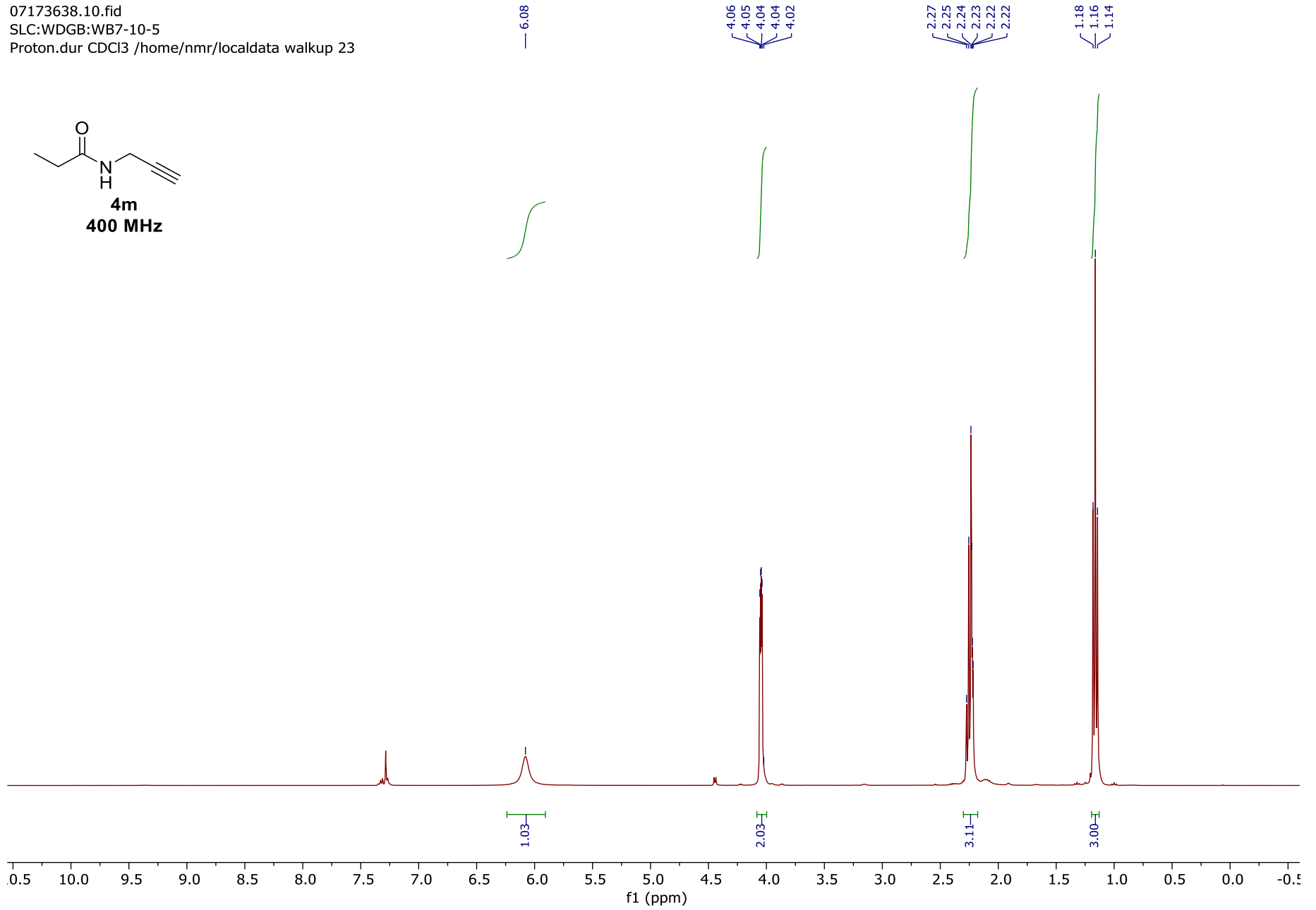
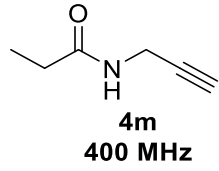
07120128.11.fid  
SLC:WDGB:WB7-10-4  
Carbon.dur CDCl3 /home/nmr/localdata walkup 29



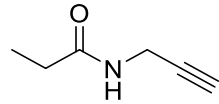
**4I**  
**101 MHz**



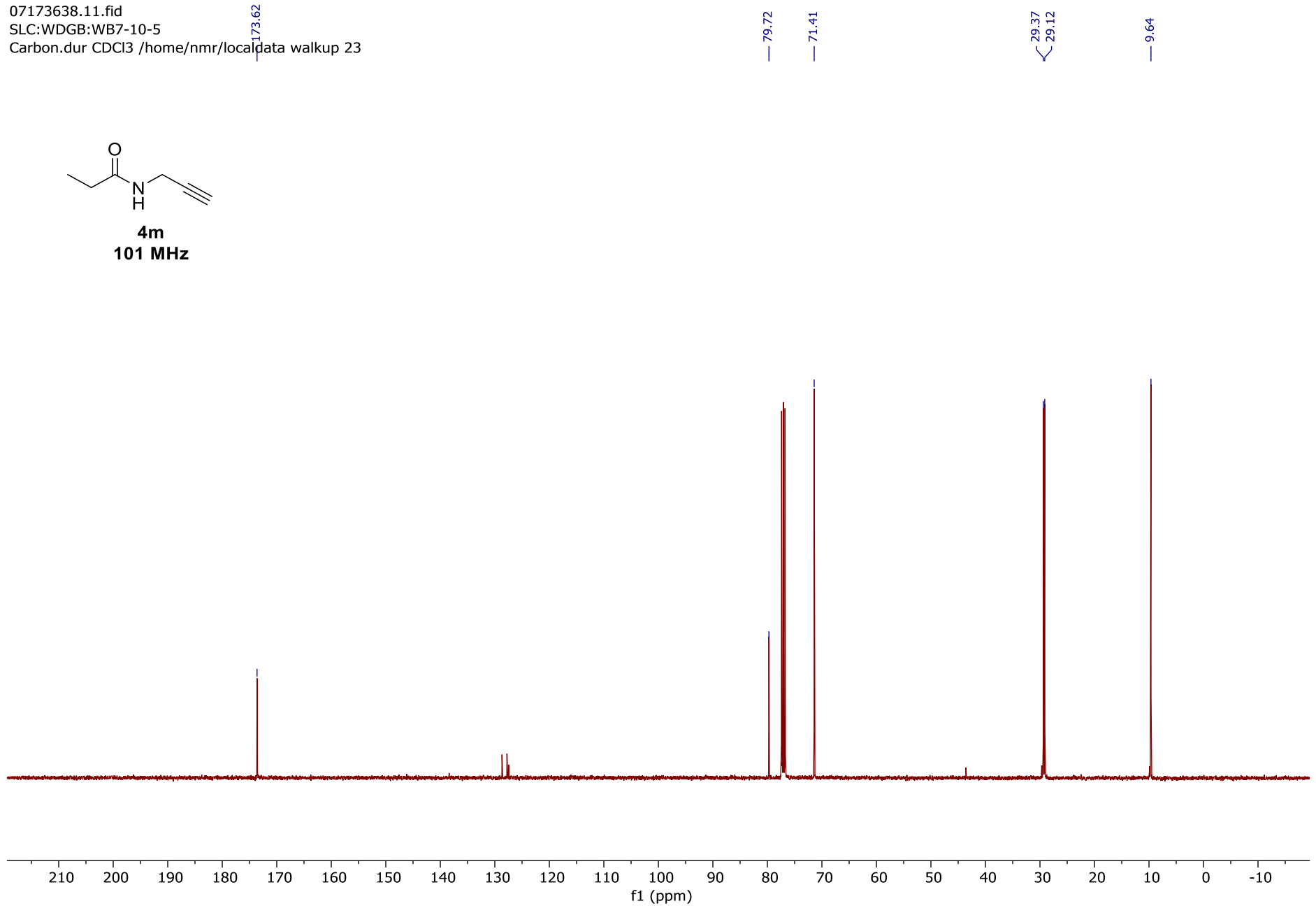
07173638.10.fid  
SLC:WDGB:WB7-10-5  
Proton.dur CDCl3 /home/nmr/localdata walkup 23



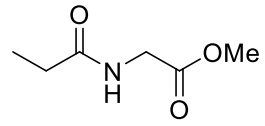
07173638.11.fid  
SLC:WDGB:WB7-10-5  
Carbon.dur CDCl3 /home/nmr/localdata walkup 23



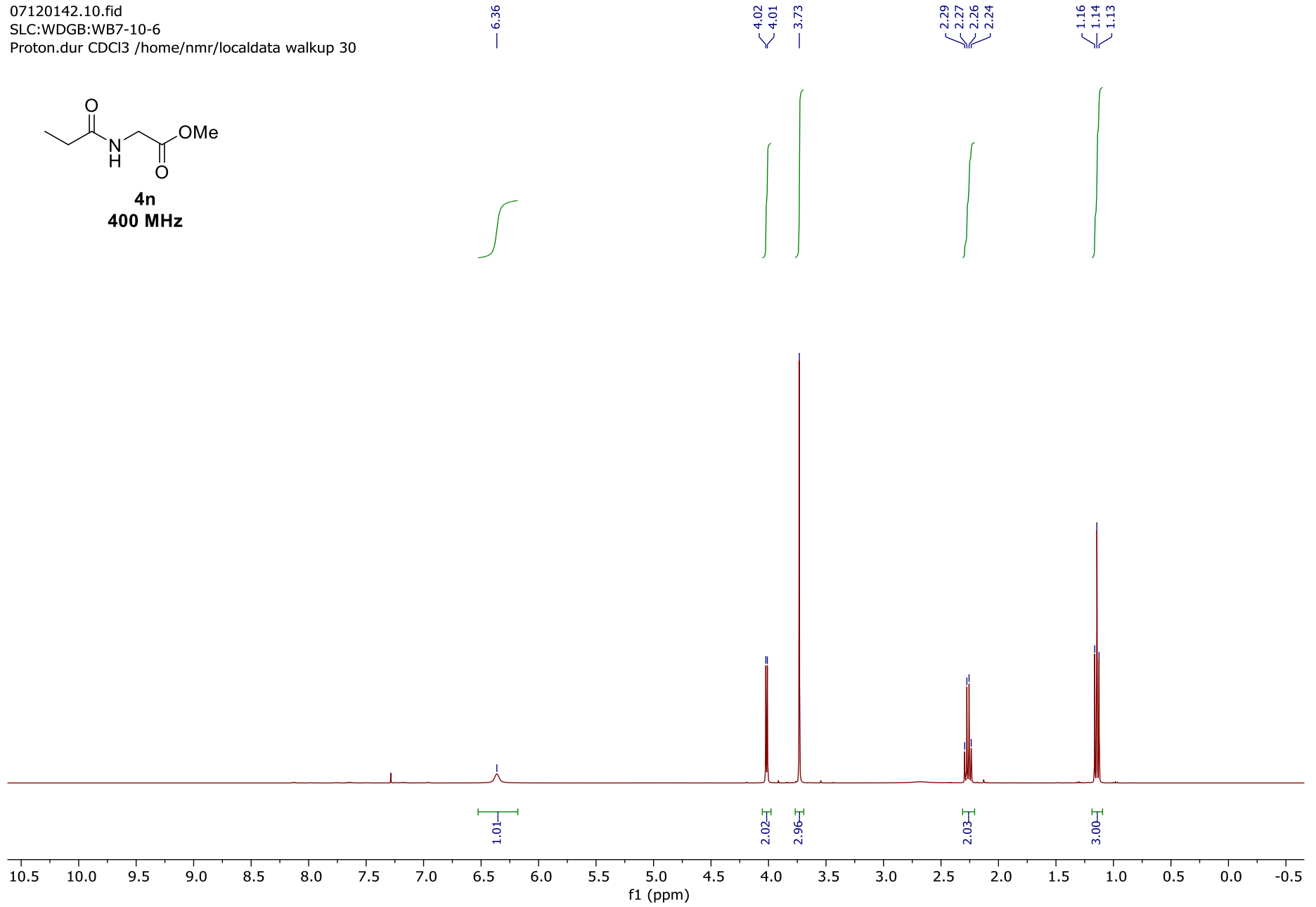
4m  
101 MHz



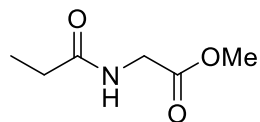
07120142.10.fid  
SLC:WDGB:WB7-10-6  
Proton.dur CDCl3 /home/nmr/localdata walkup 30



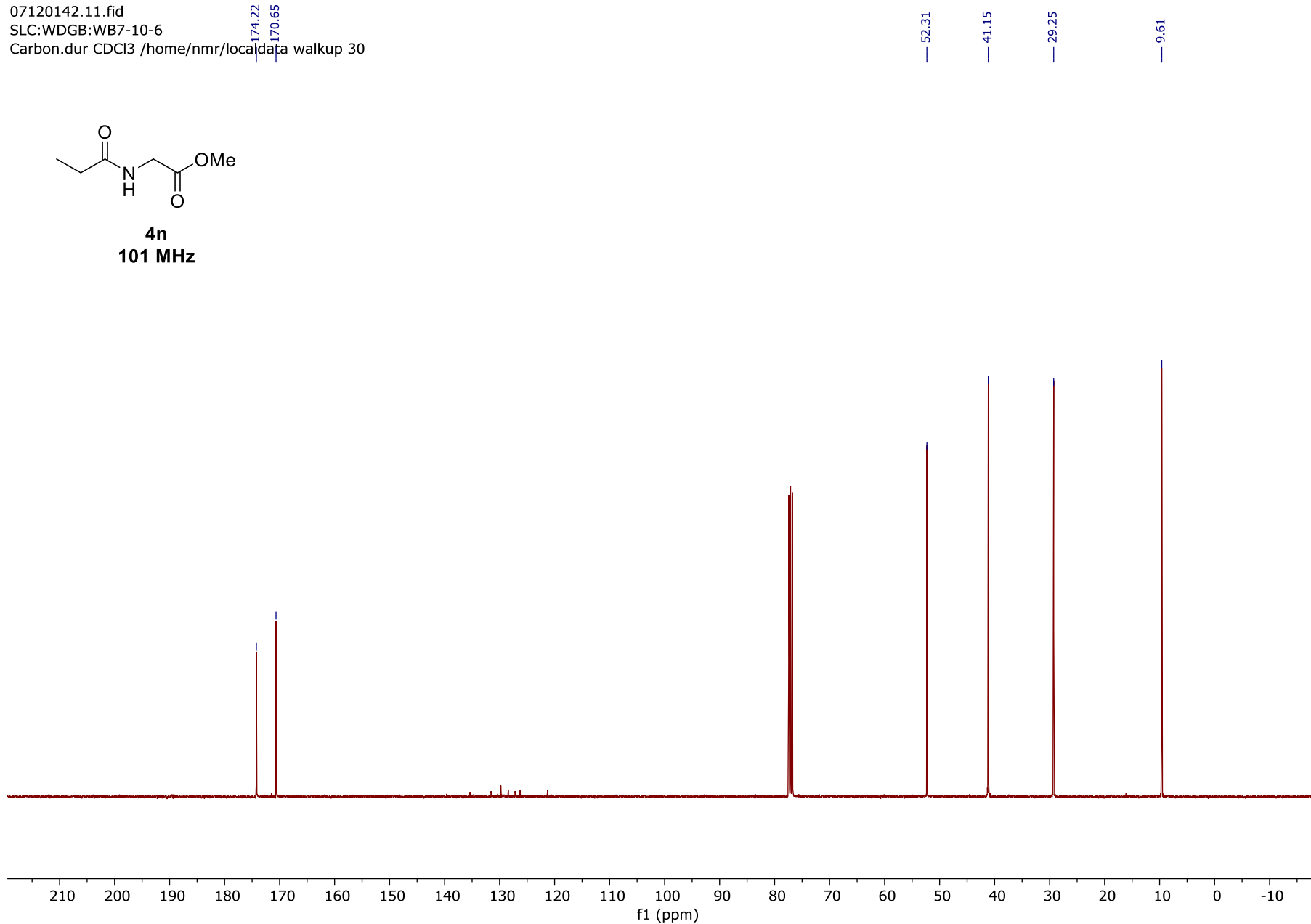
4n  
400 MHz



07120142.11.fid  
SLC:WDGB:WB7-10-6  
Carbon.dur CDCl3 /home/nmr/localdata walkup 30

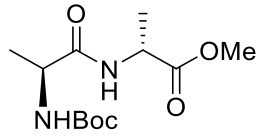


**4n**  
**101 MHz**

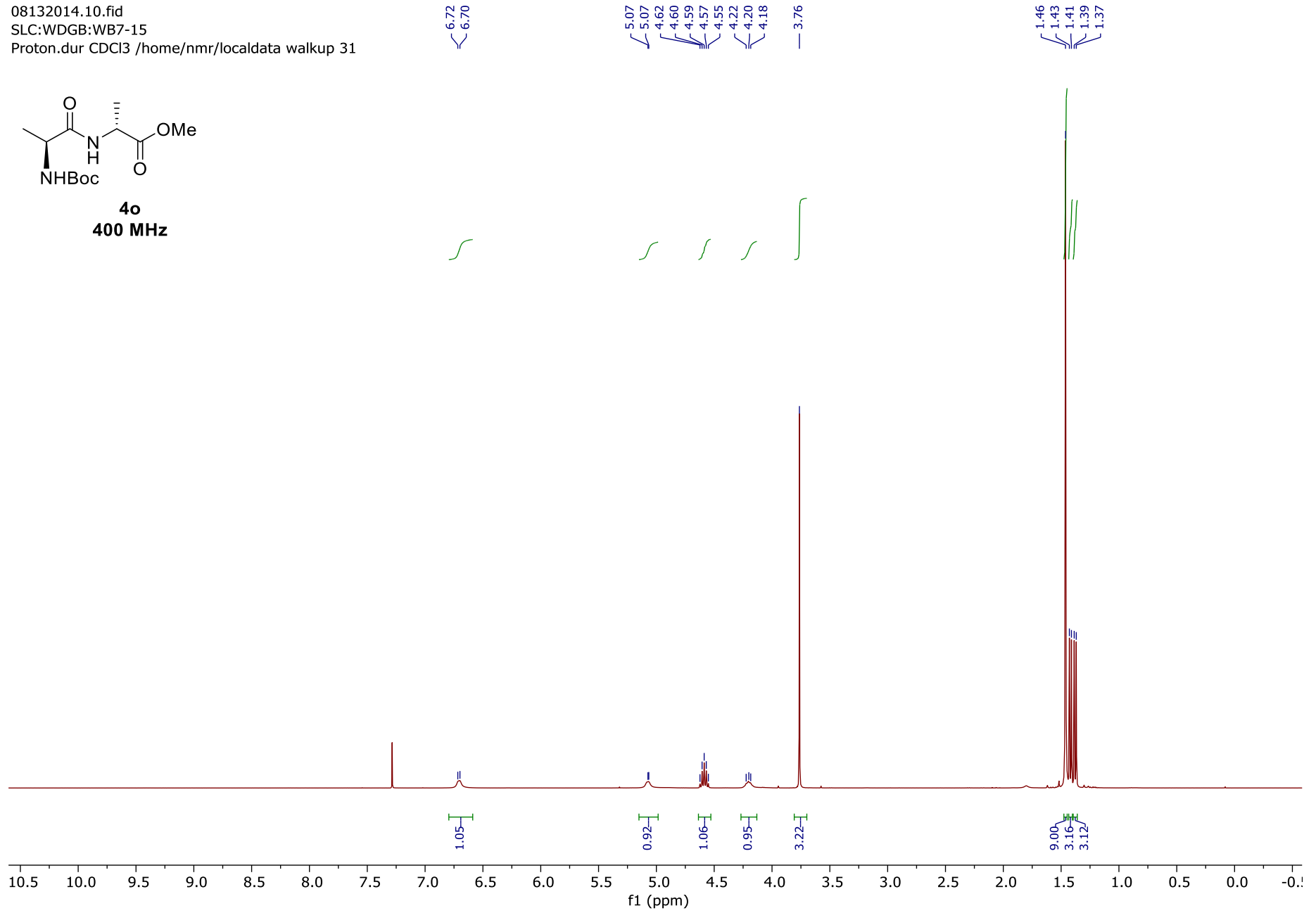




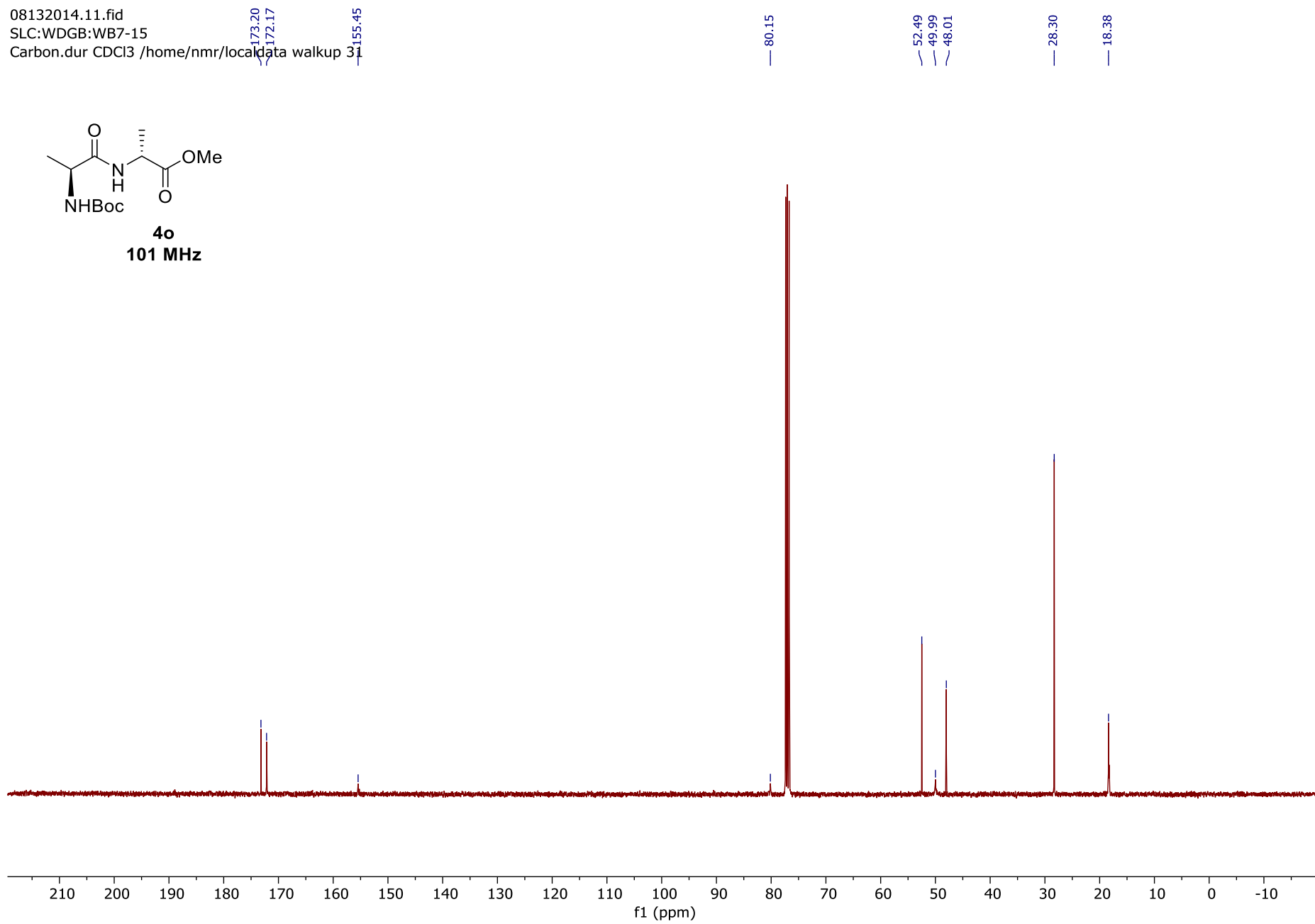
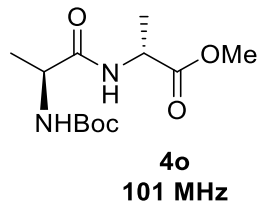
08132014.10.fid  
SLC:WDGB:WB7-15  
Proton.dur CDCl3 /home/nmr/localdata walkup 31



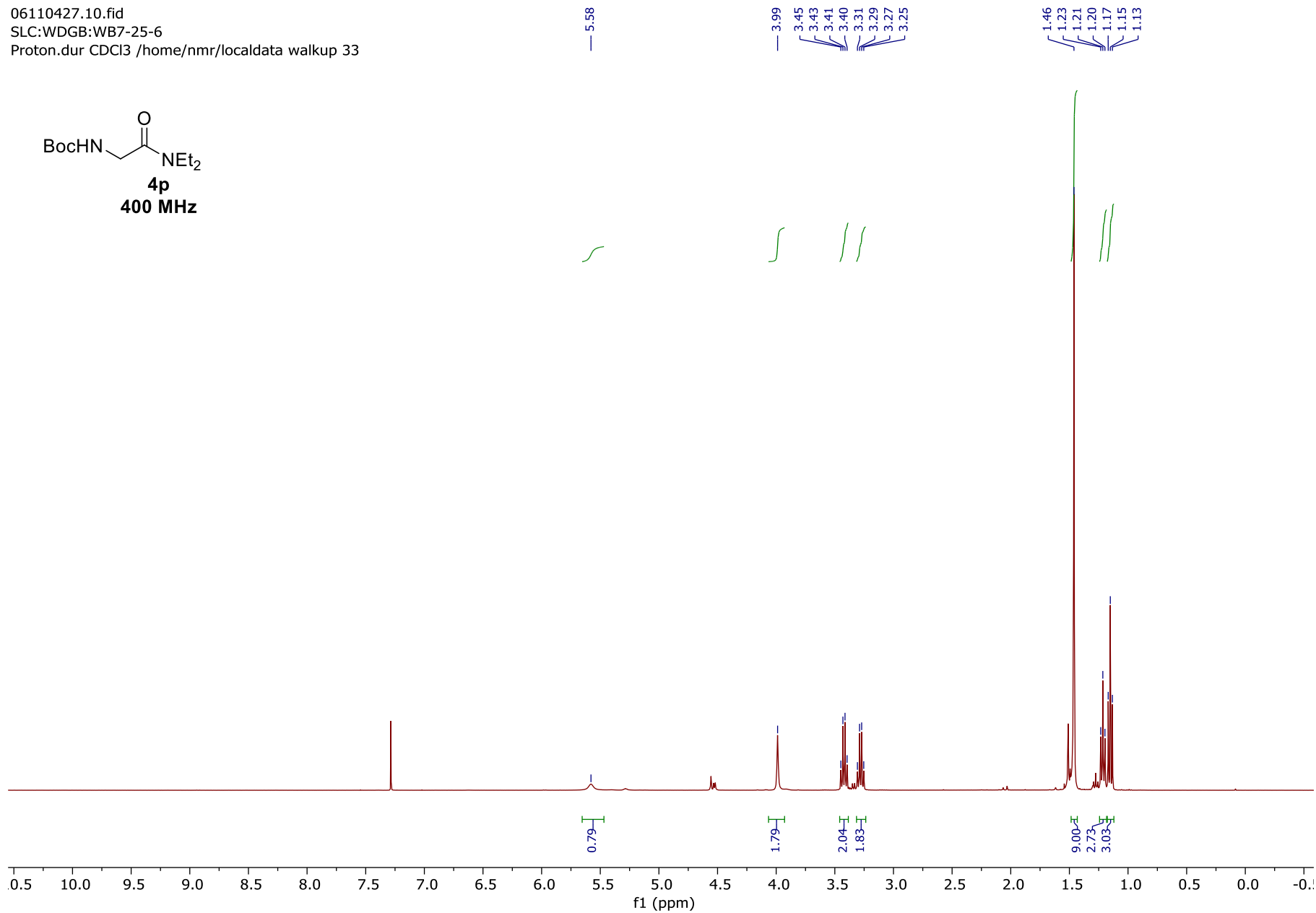
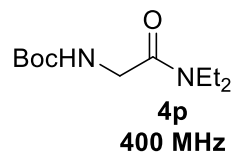
**4o**  
**400 MHz**



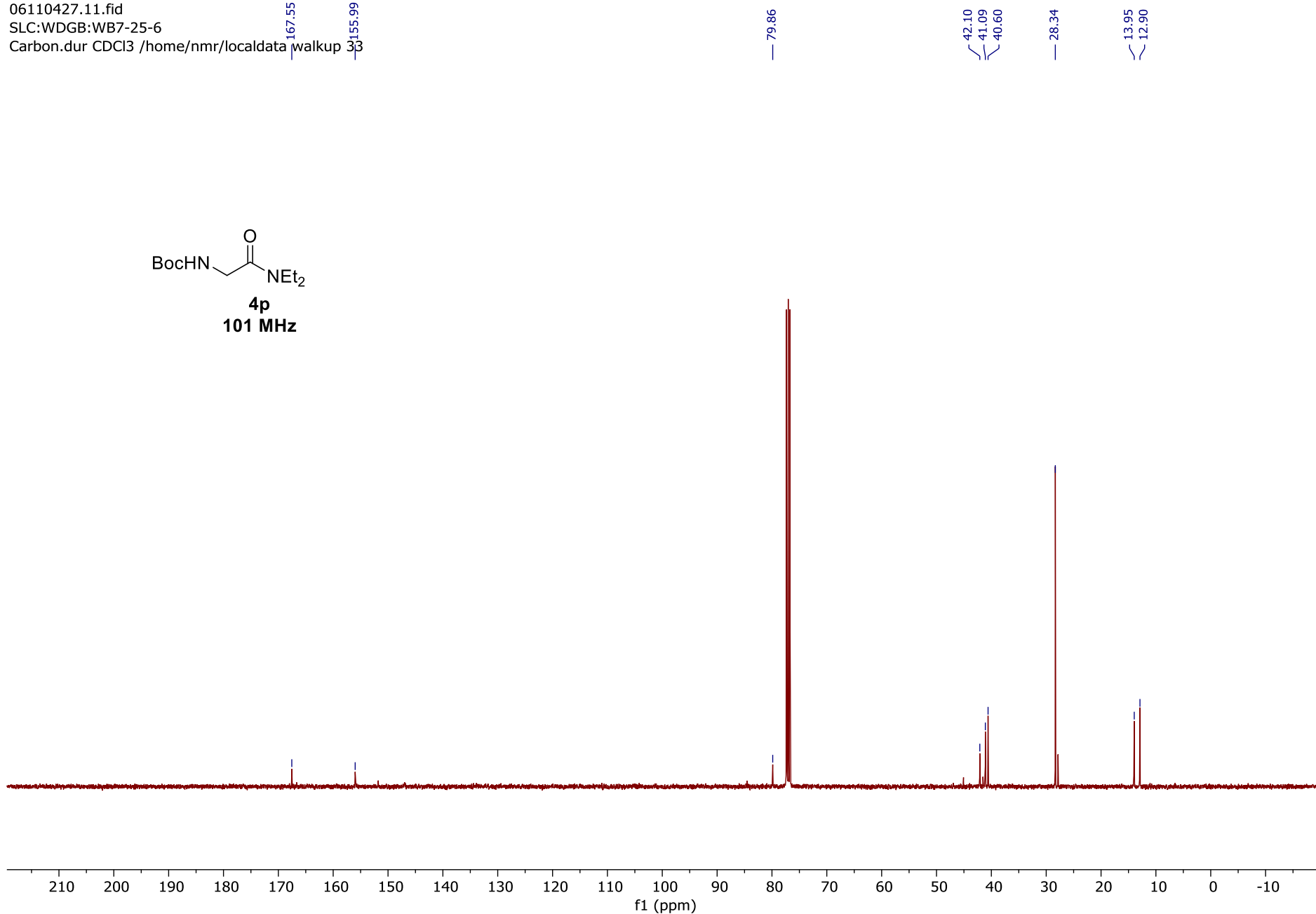
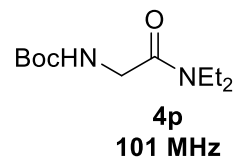
08132014.11.fid  
SLC:WDGB:WB7-15  
Carbon.dur CDCl3 /home/nmr/localdata walkup 31



06110427.10.fid  
SLC:WDGB:WB7-25-6  
Proton.dur CDCl3 /home/nmr/localdata walkup 33



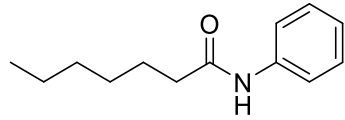
06110427.11.fid  
SLC:WDGB:WB7-25-6  
Carbon.dur CDCl3 /home/nmr/localdata/walkup 33



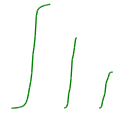
07173742.10.fid  
SLC:WDGB:WB7-17-2  
Proton.dur CDCl3 /home/nmr/localdata walkup 26

7.58  
7.56  
7.54  
7.34  
7.32  
7.30  
7.13  
7.11  
7.09

2.39  
2.37  
2.35  
1.77  
1.75  
1.73  
1.71  
1.69  
1.40  
1.39  
1.38  
1.37  
1.36  
1.35  
1.34  
1.33  
1.32  
1.30  
1.30  
0.92  
0.90  
0.89



4q  
400 MHz

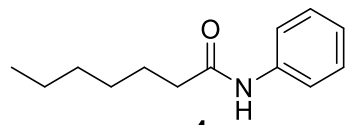


2.59  
1.76  
0.90

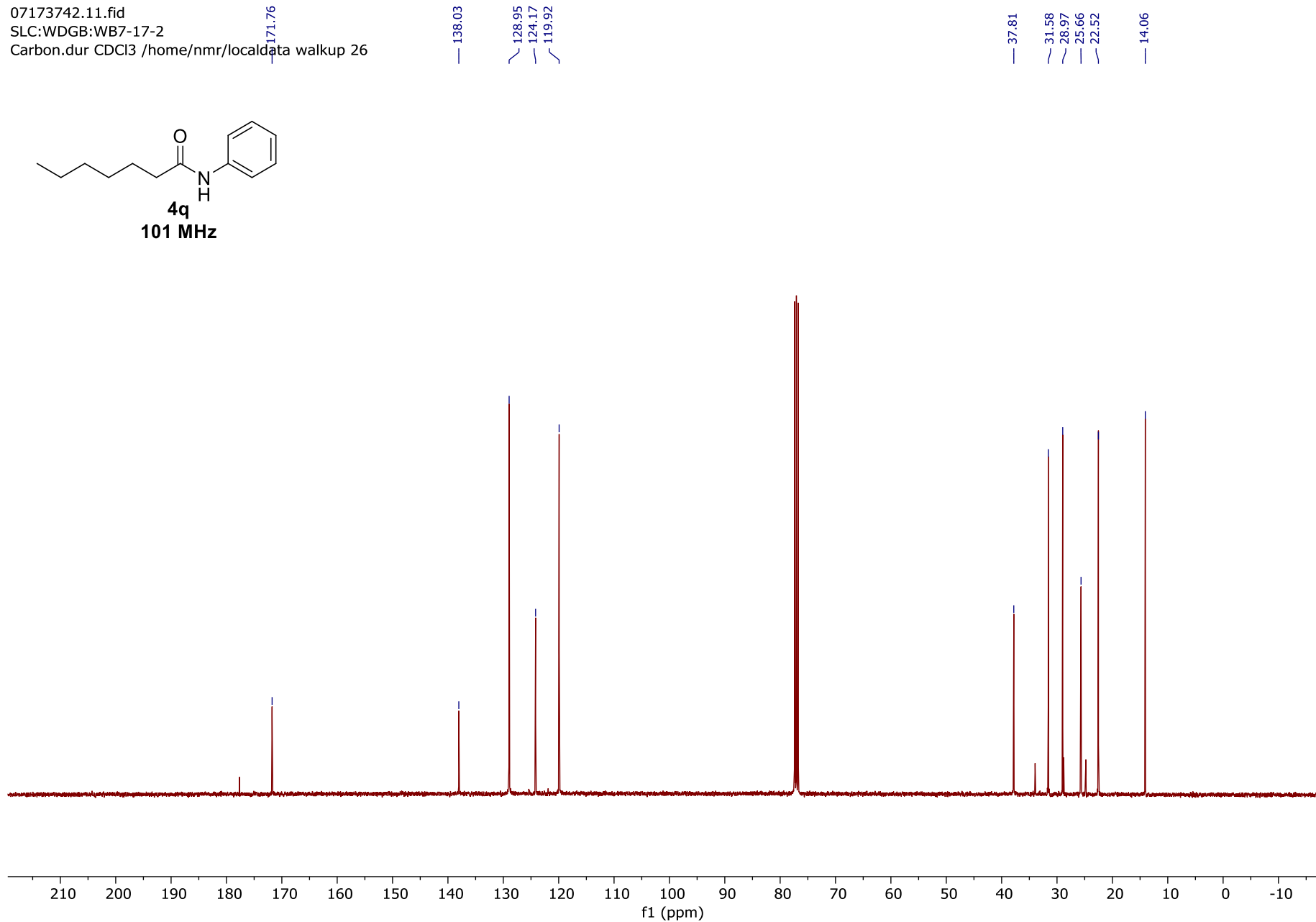
1.97  
1.79  
6.08  
3.00

10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5  
f1 (ppm)

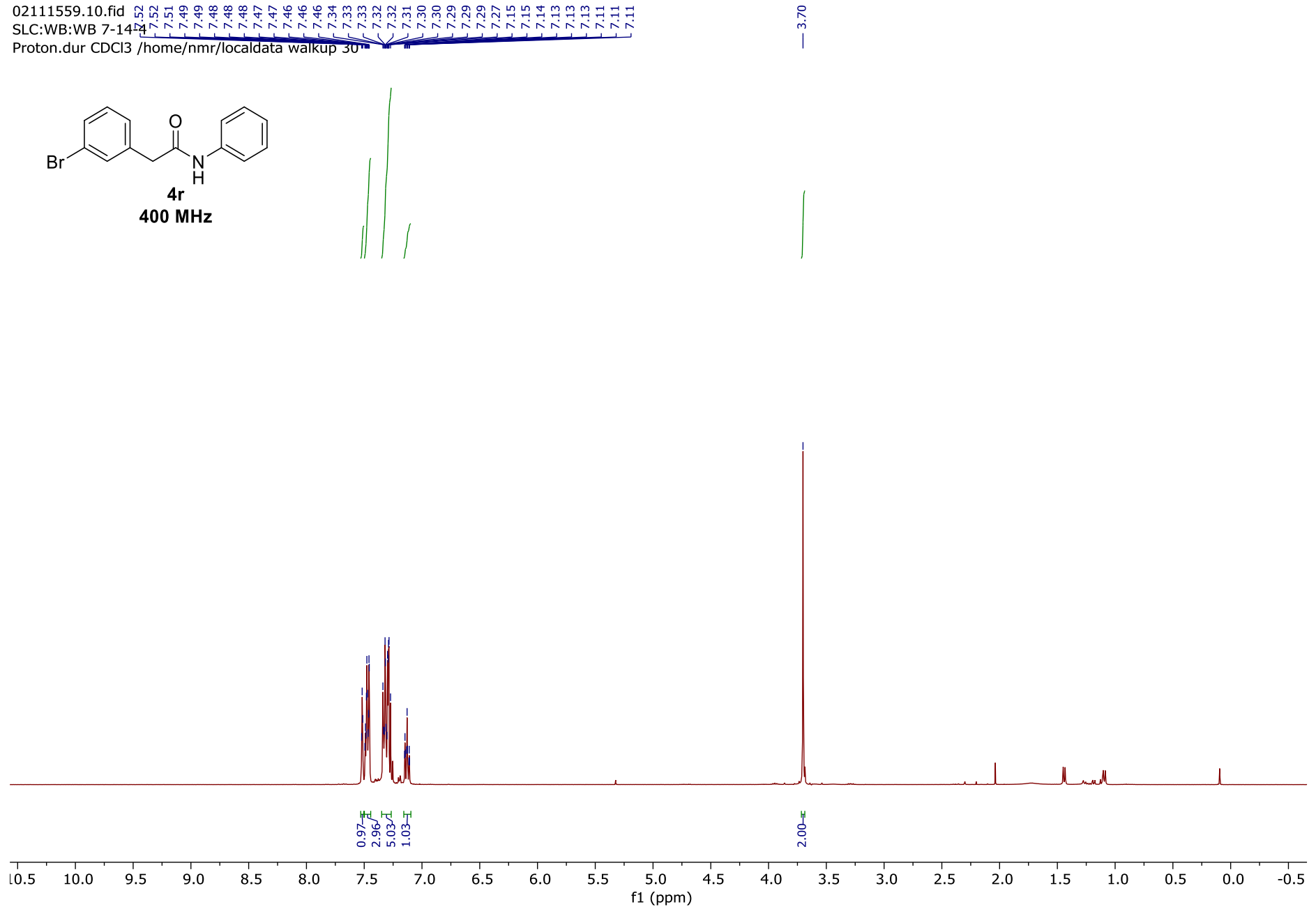
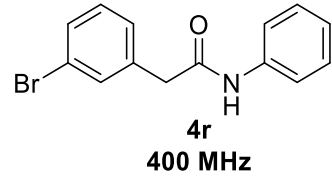
07173742.11.fid  
SLC:WDGB:WB7-17-2  
Carbon.dur CDCl3 /home/nmr/localdata walkup 26



4q  
101 MHz



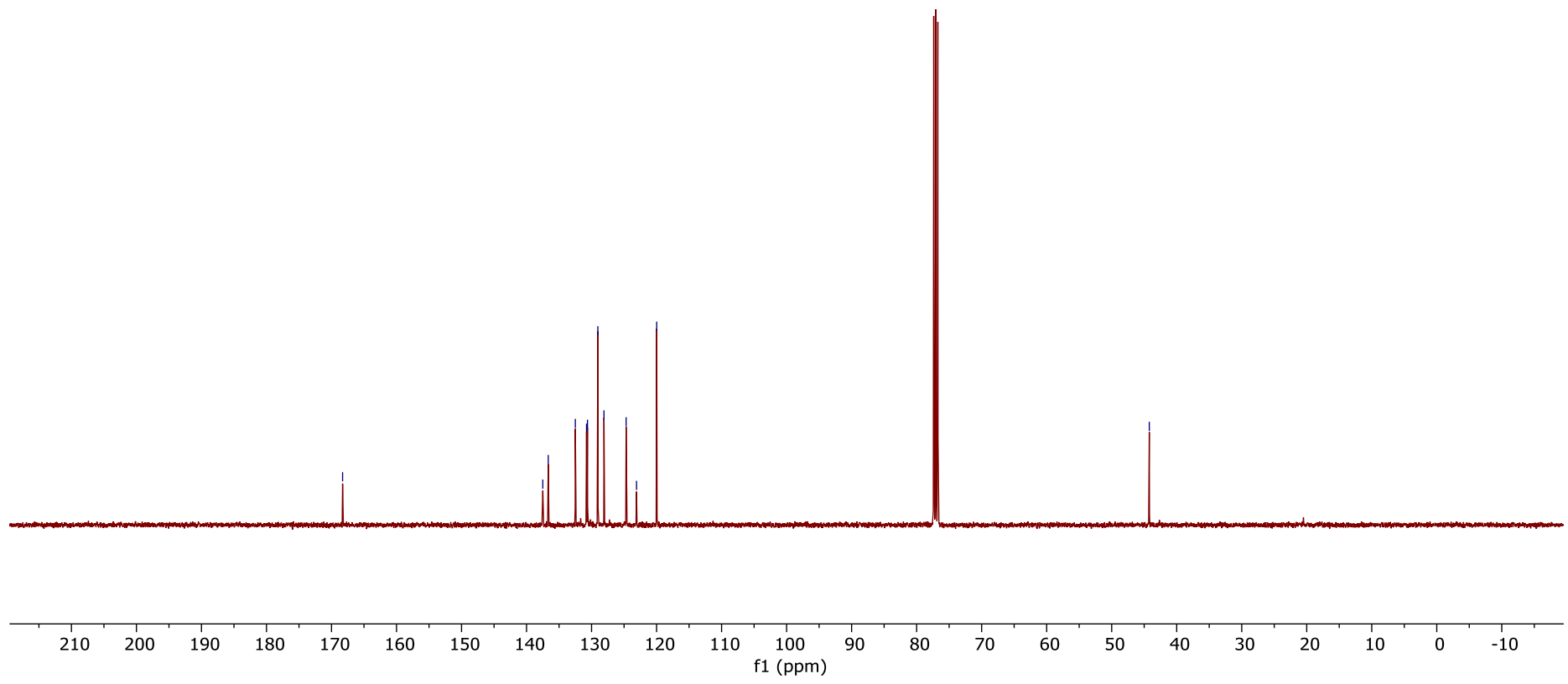
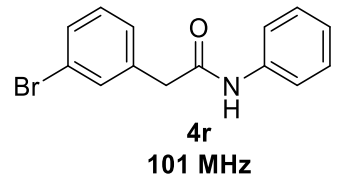
02111559.10.fid  
SLC:WB:WB 7-14  
Proton.dur CDCl3 /home/nmr/localdata walkup 30



02111559.11.1.1r  
SLC:WB:WB 7-14-4  
Carbon.dur CDCl3 /home/nmr/localdata/walkup 30

137.50  
136.66  
132.50  
130.77  
130.63  
129.03  
128.09  
124.68  
123.08  
119.98

44.22

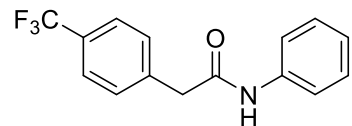




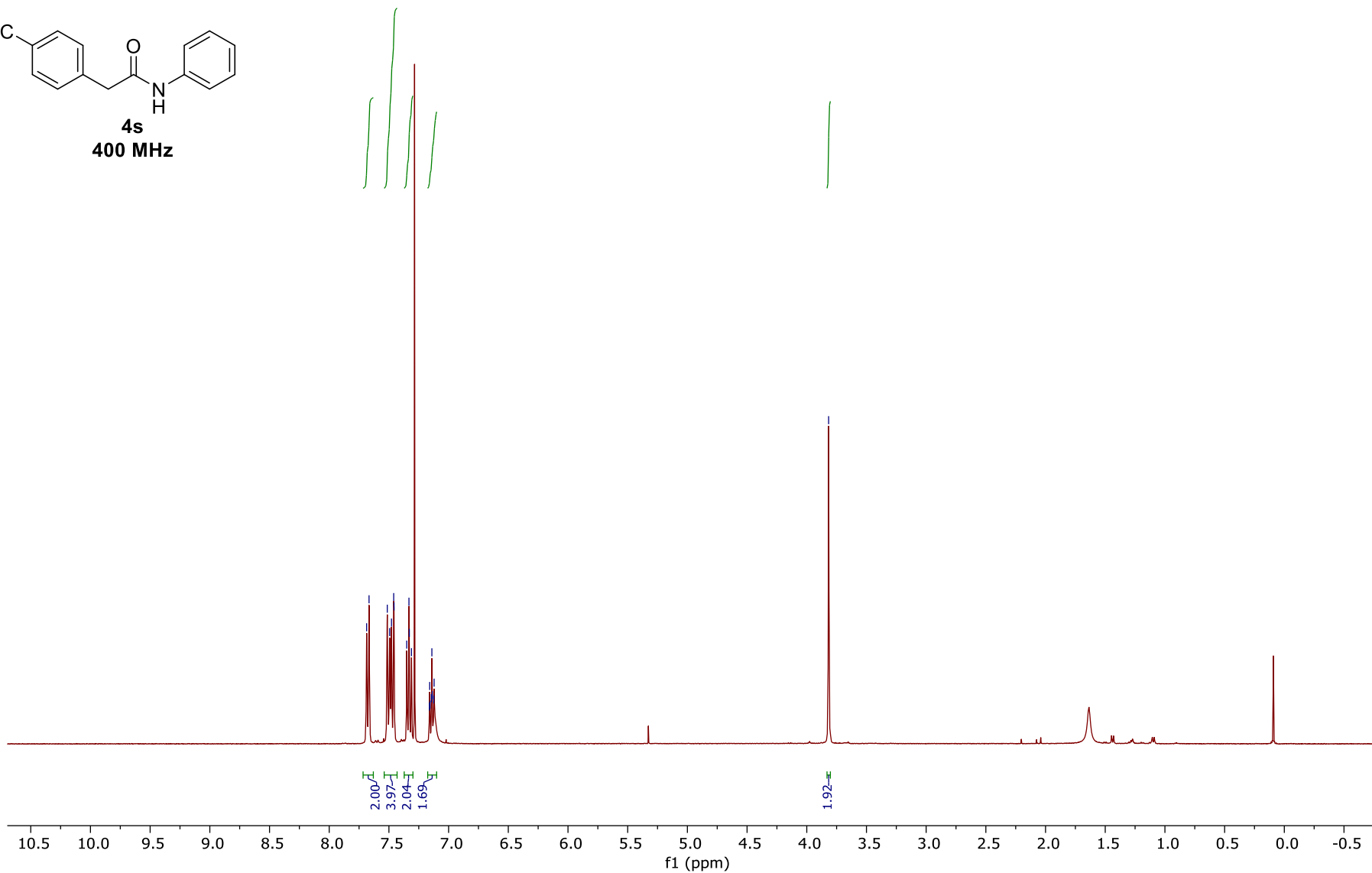
02111733.10.fid  
SLC:WB:WB 7-14-5  
Proton.dur CDCl3 /home/nmr/localdata walkup 32

7.69  
7.67  
7.51  
7.49  
7.48  
7.46  
7.46  
7.35  
7.33  
7.33  
7.31  
7.16  
7.16  
7.16  
7.14  
7.14  
7.14  
7.12  
7.12

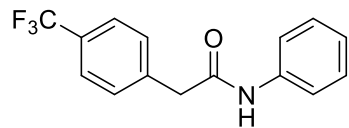
3.82



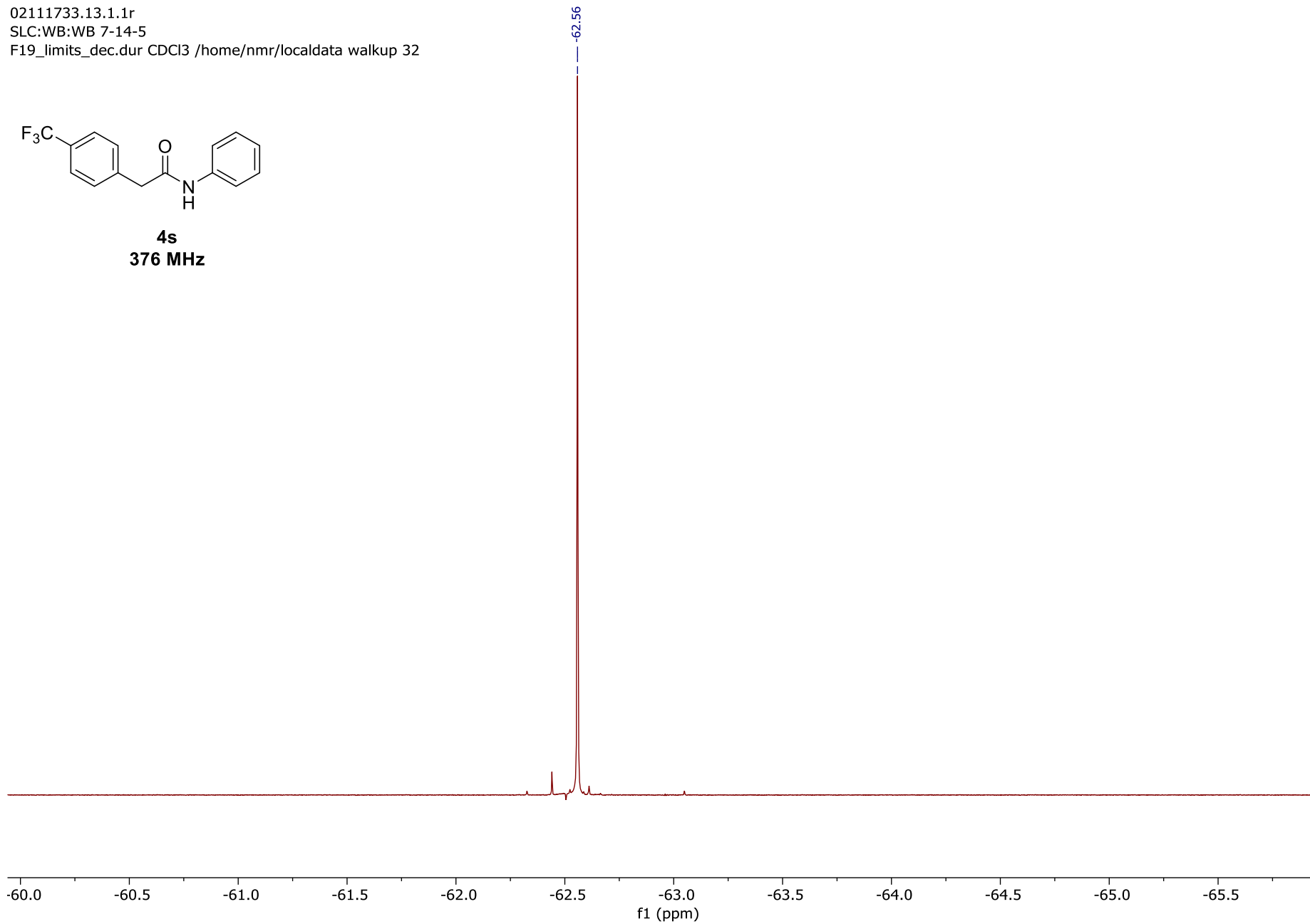
4s  
400 MHz



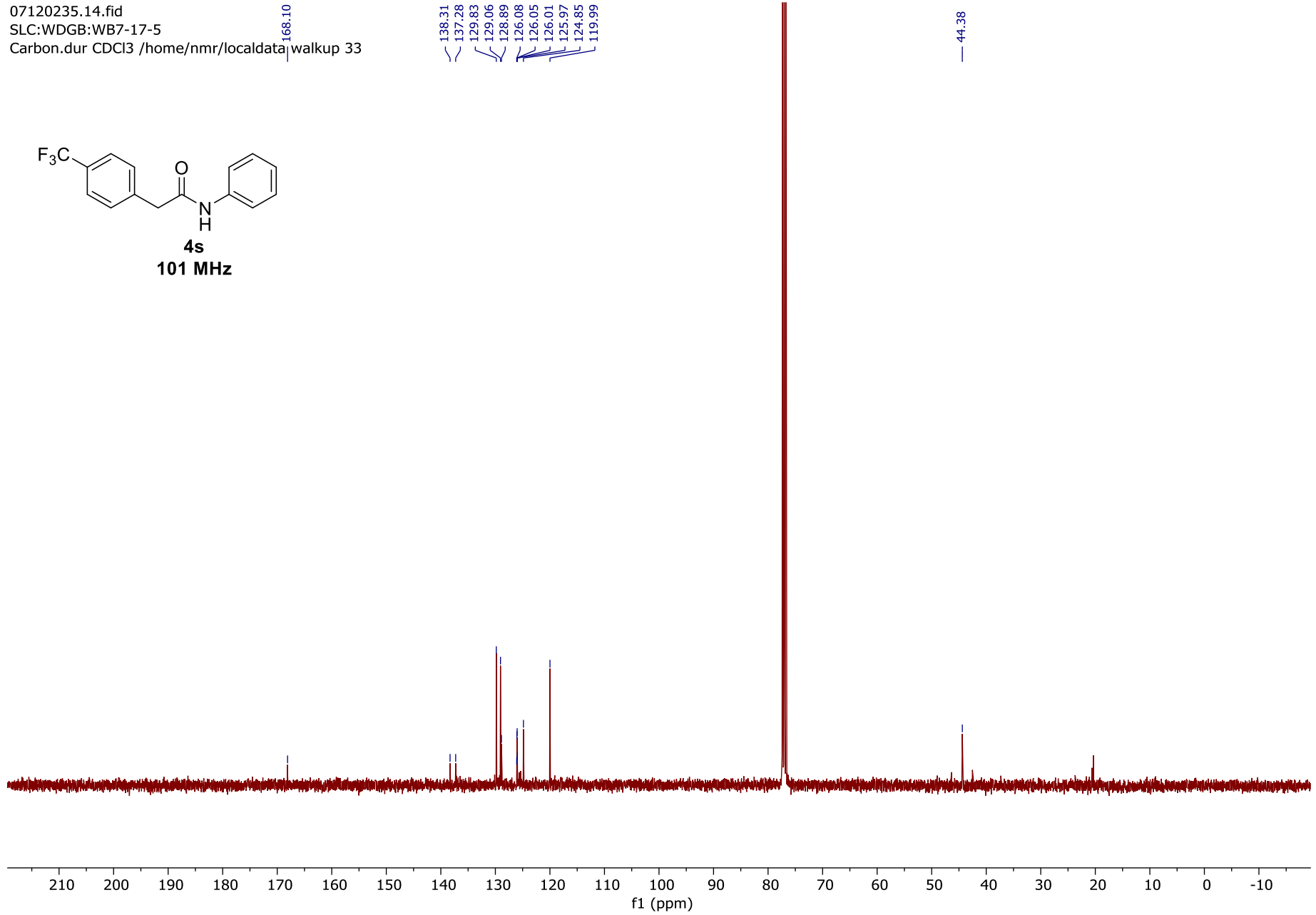
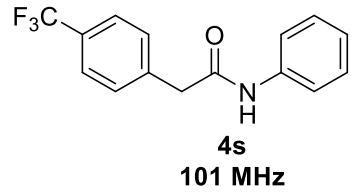
02111733.13.1.1r  
SLC:WB:WB 7-14-5  
F19\_limits\_dec.dur CDCl3 /home/nmr/localdata walkup 32



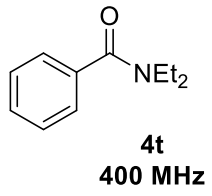
**4s**  
**376 MHz**



07120235.14.fid  
SLC:WDGB:WB7-17-5  
Carbon.dur CDCl3 /home/nmr/localdata/walkup 33



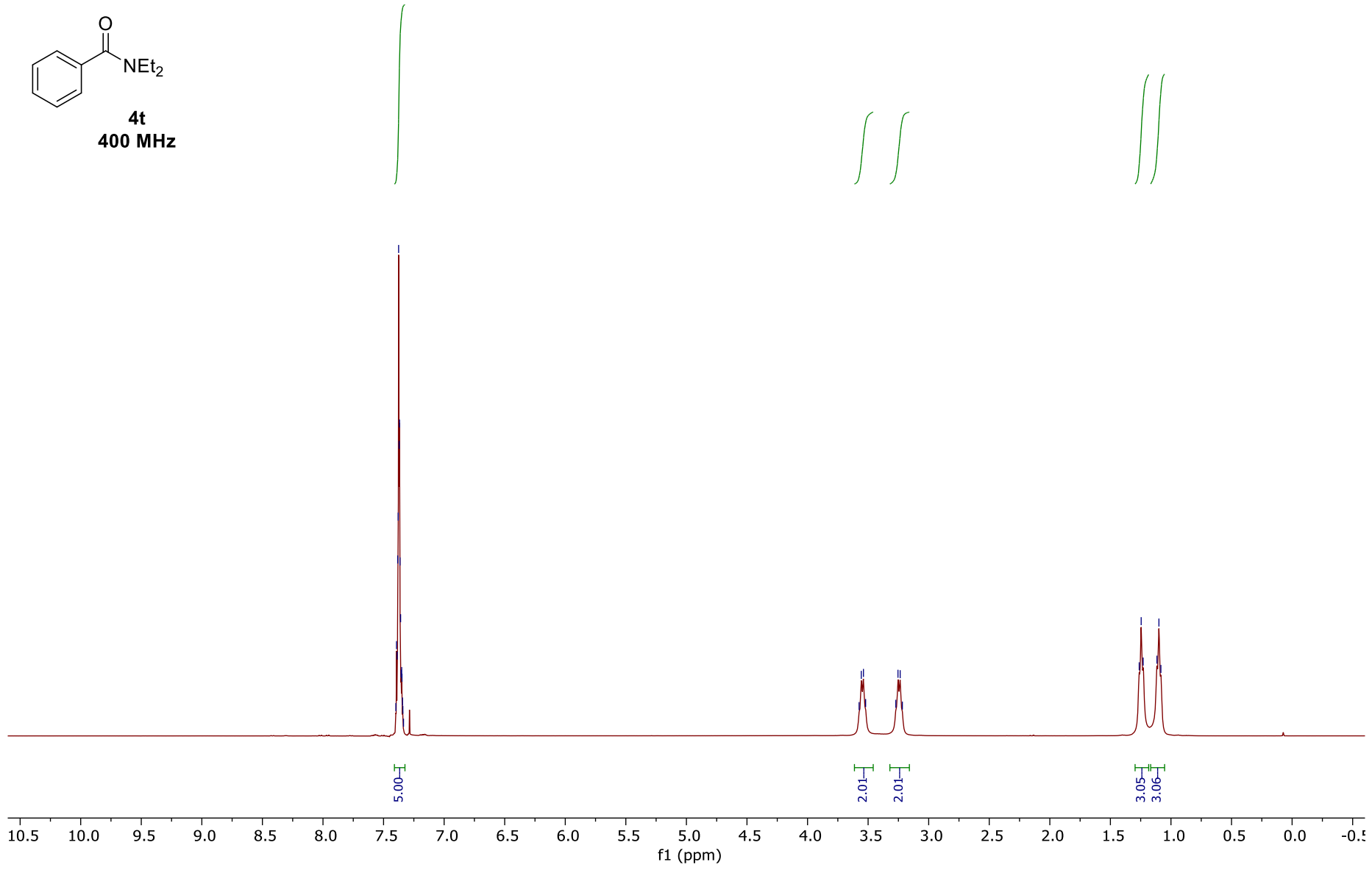
06132011.10.fid  
SLC:WDGB:WB7-25-1  
Proton.dur CDCl3 /home/nmr/localdata walkup 21



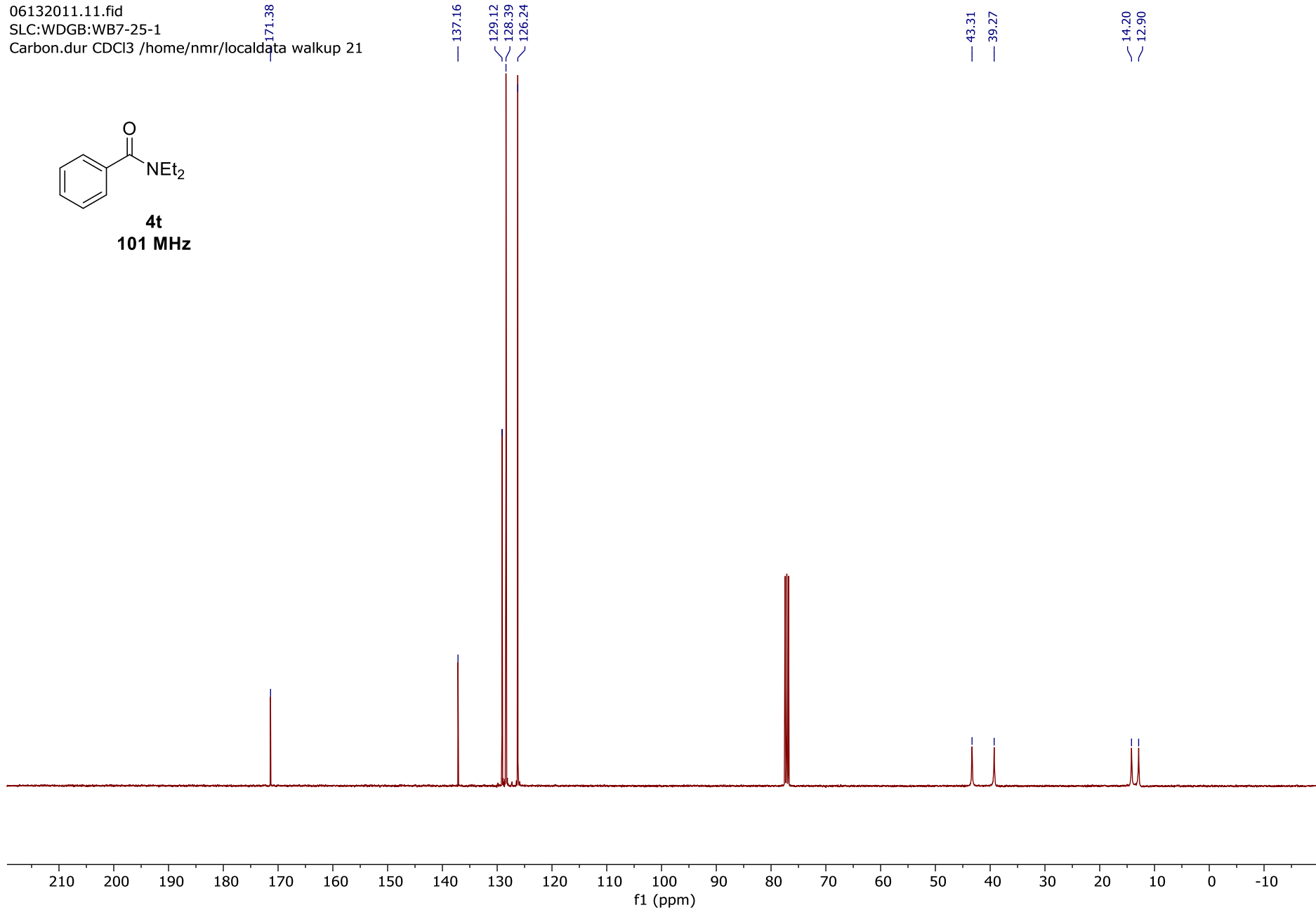
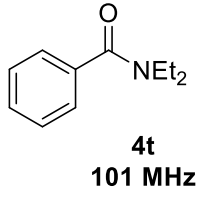
7.40  
7.39  
7.39  
7.38  
7.38  
7.37  
7.37  
7.36  
7.36  
7.35  
7.35  
7.34  
7.34

3.57  
3.56  
3.54  
3.52  
3.27  
3.25  
3.24  
3.22

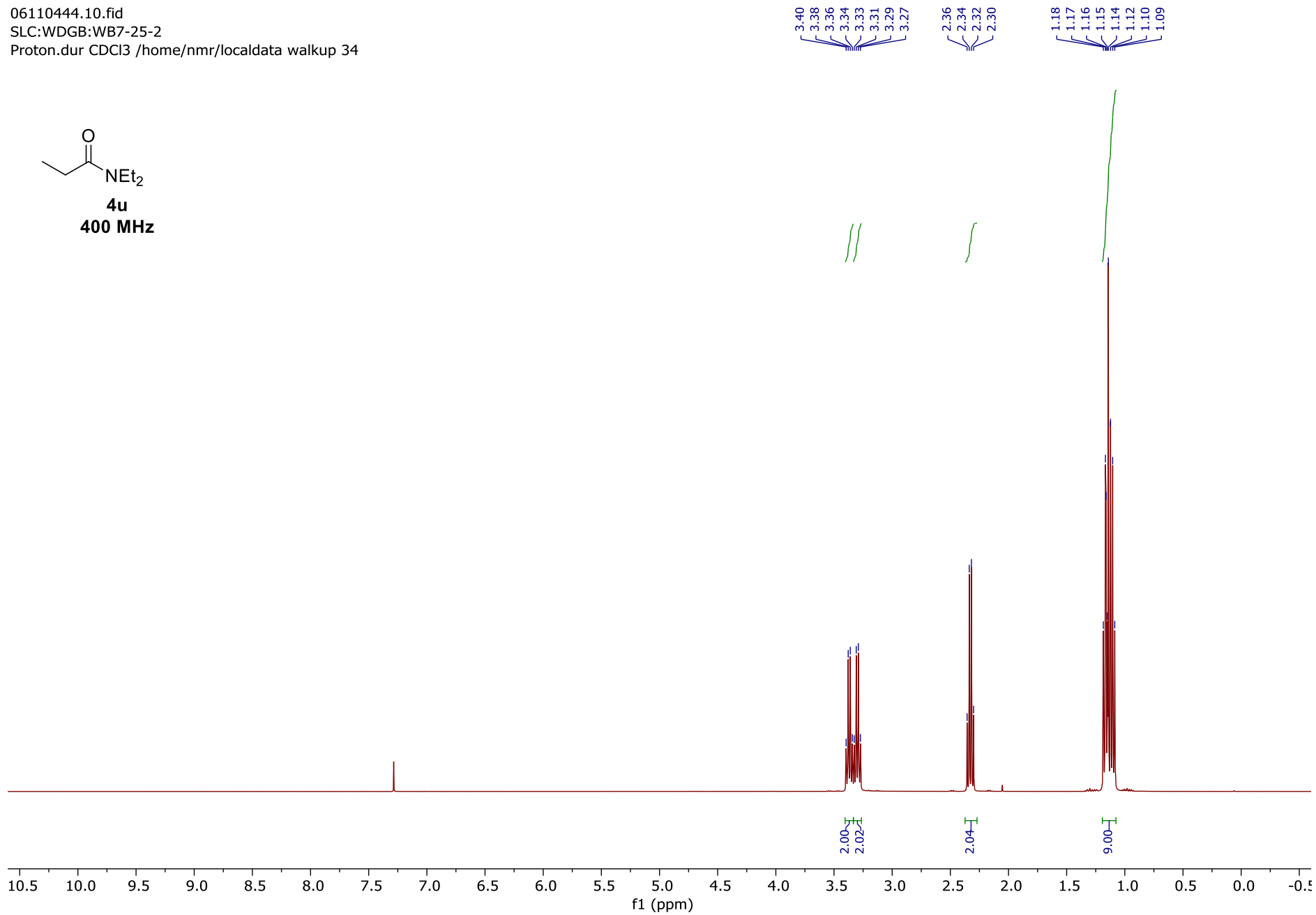
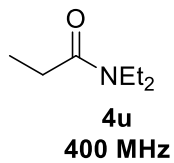
1.26  
1.25  
1.23  
1.12  
1.10  
1.08



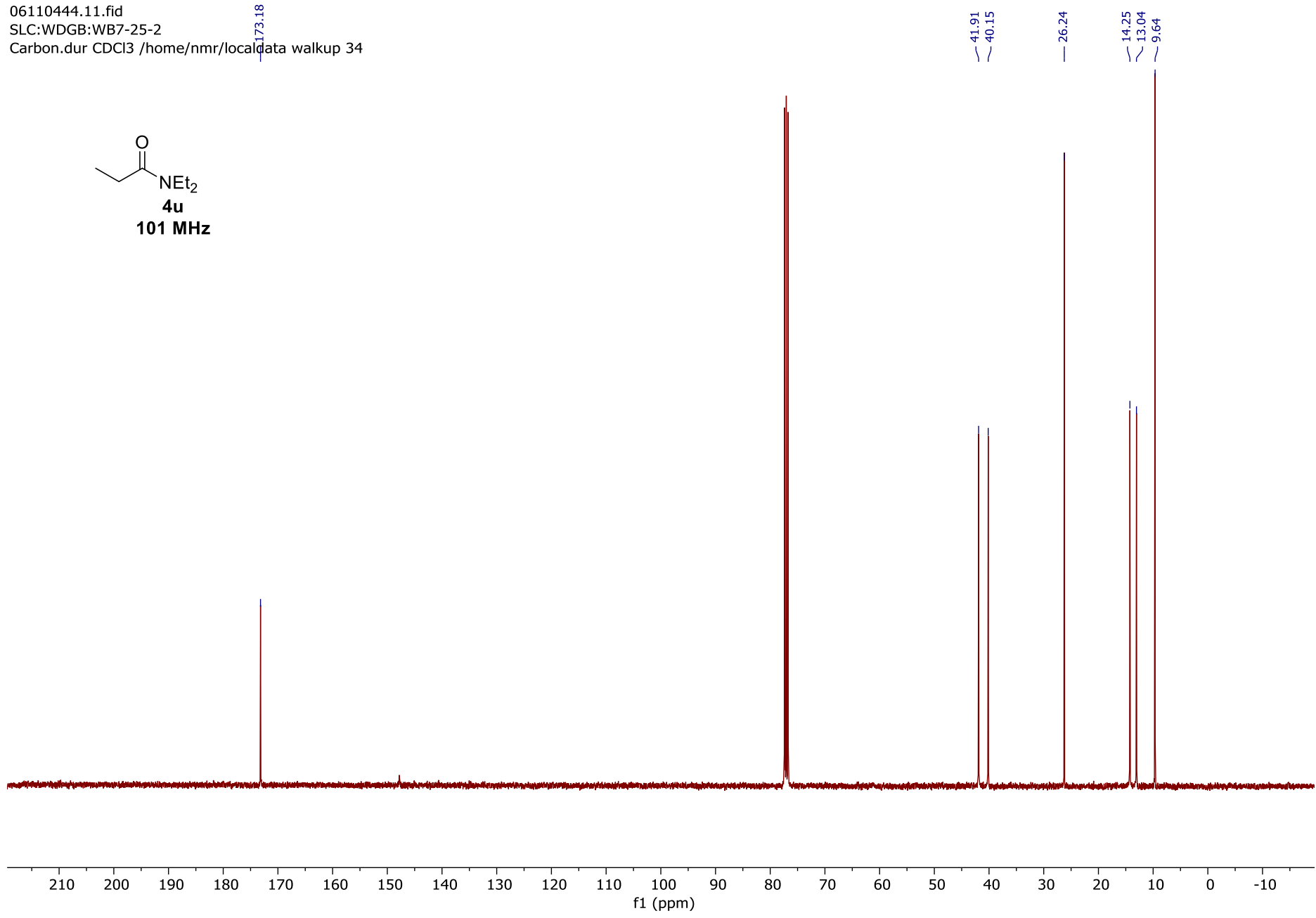
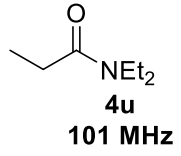
06132011.11.fid  
SLC:WDGB:WB7-25-1  
Carbon.dur CDCl3 /home/nmr/localdata walkup 21



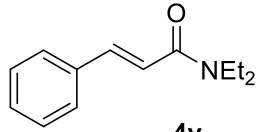
06110444.10.fid  
SLC:WDGB:WB7-25-2  
Proton.dur CDCl3 /home/nmr/localdata walkup 34



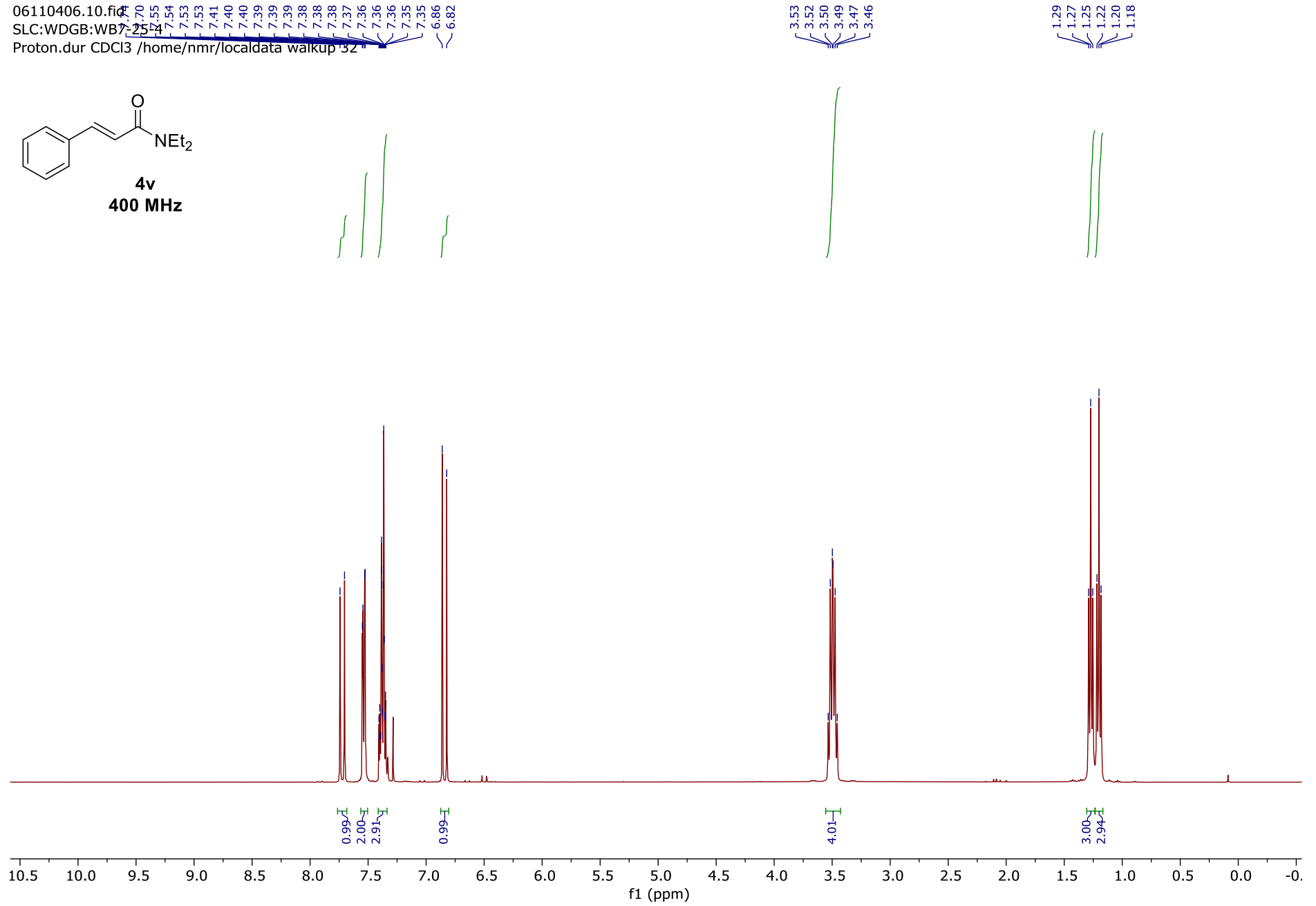
06110444.11.fid  
SLC:WDGB:WB7-25-2  
Carbon.dur CDCl3 /home/nmr/localdata walkup 34



06110406.10.fid  
SLC:WDGB:WB7-254  
Proton.dur CDCl3 /home/nmr/localdata/walkup-32

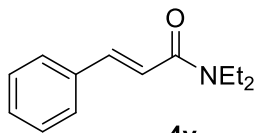


4v  
400 MHz

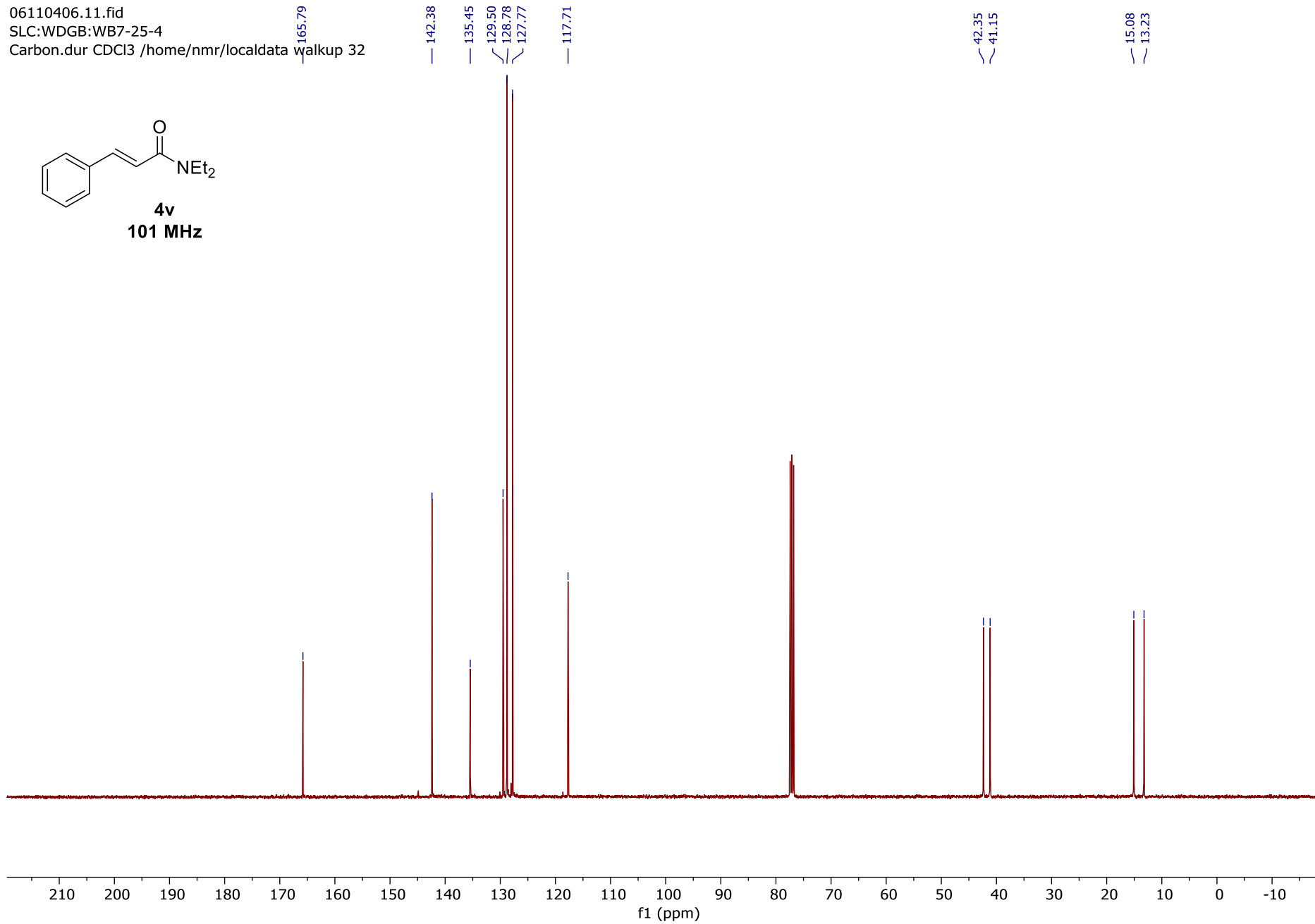




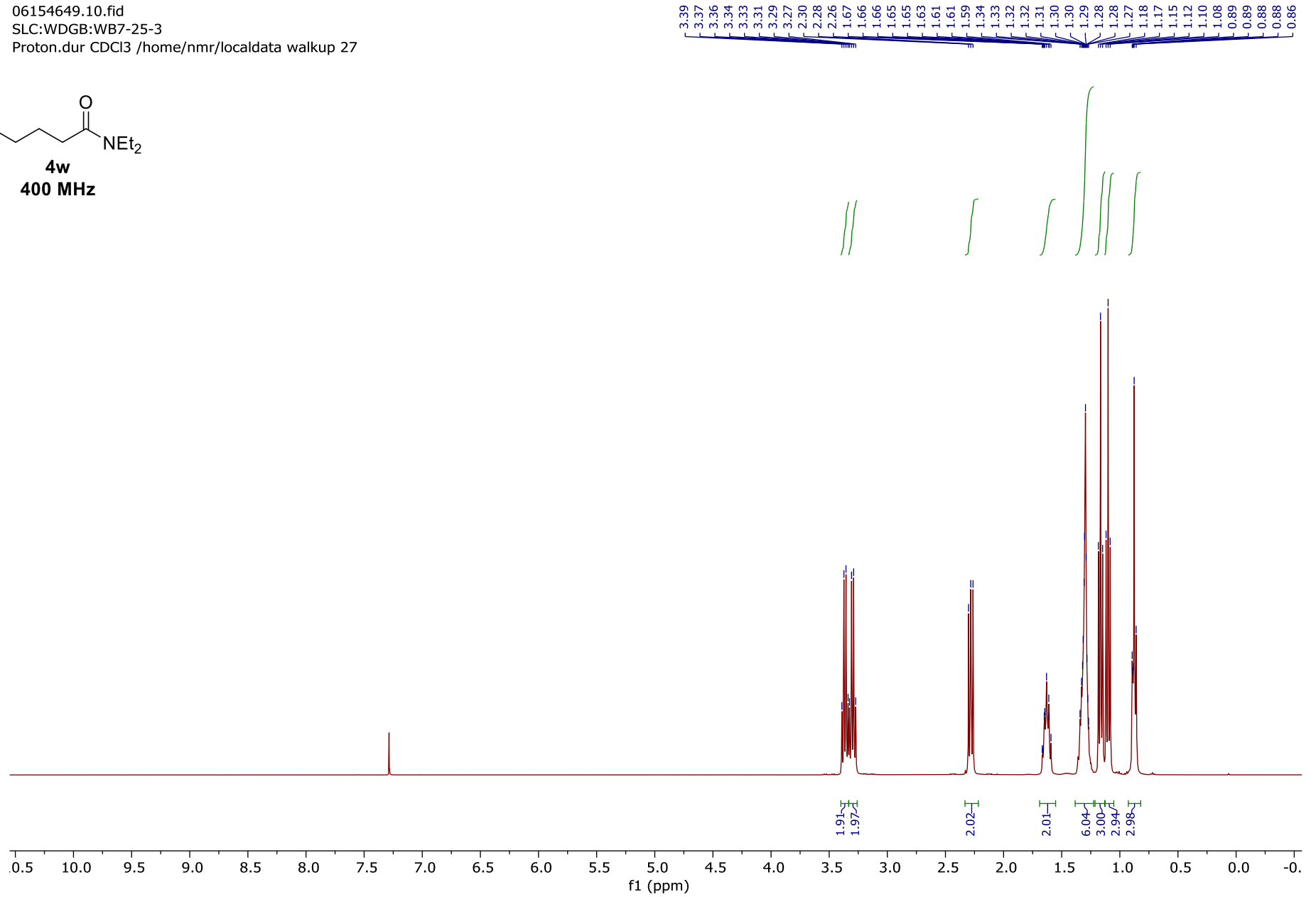
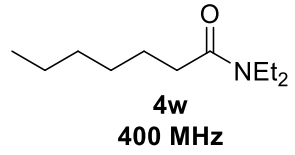
06110406.11.fid  
SLC:WDGB:WB7-25-4  
Carbon.dur CDCl3 /home/nmr/localdata walkup 32



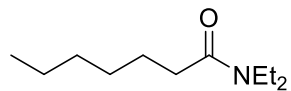
4v  
101 MHz



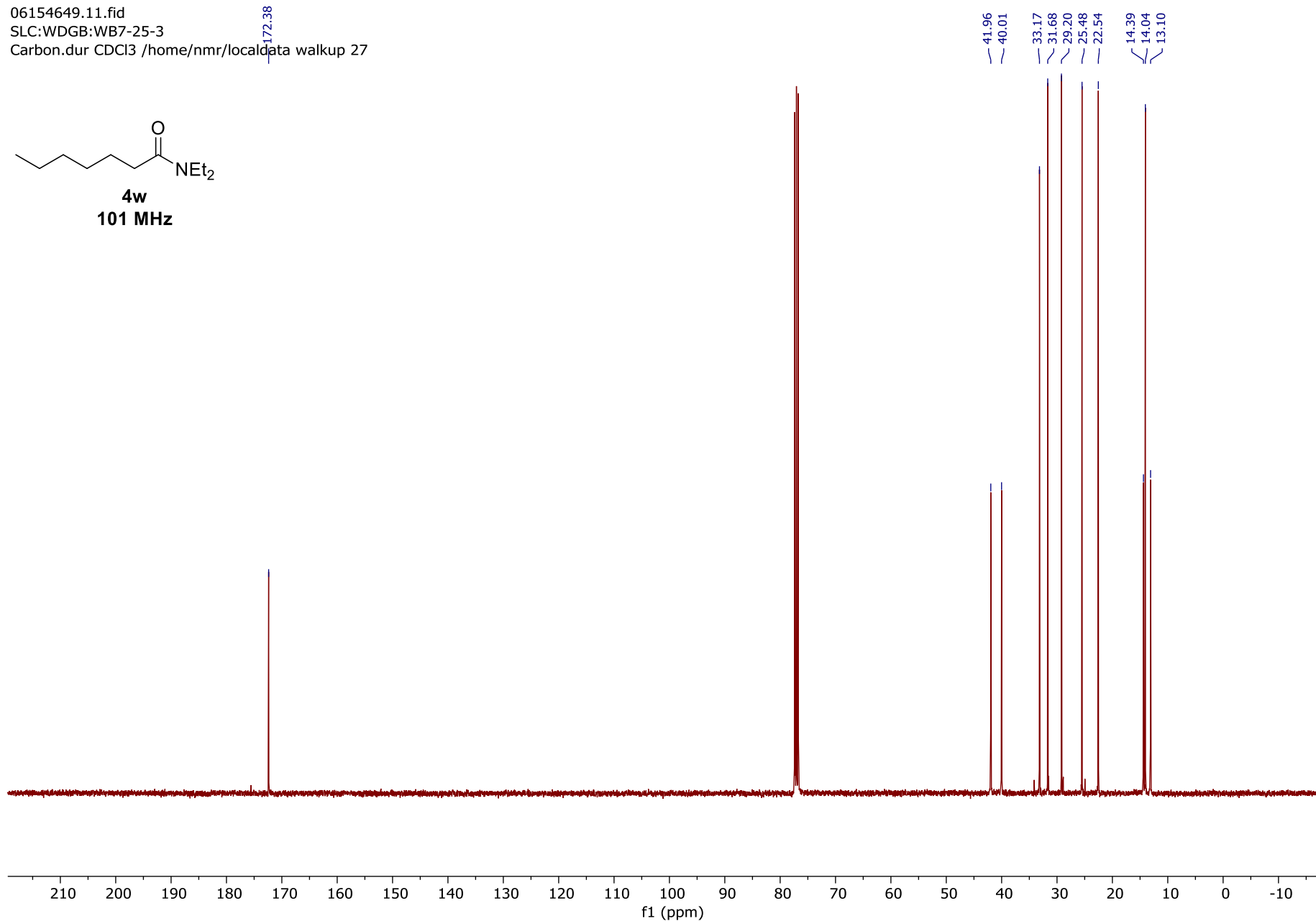
06154649.10.fid  
SLC:WDGB:WB7-25-3  
Proton.dur CDCl3 /home/nmr/localdata walkup 27



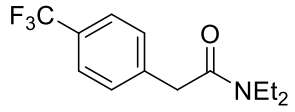
06154649.11.fid  
SLC:WDGB:WB7-25-3  
Carbon.dur CDCl3 /home/nmr/localdata walkup 27



4w  
101 MHz



06154746.10.fid  
SLC:WDGB:WB7-25-5  
Proton.dur CDCl3 /home/nmr/localdata walkup 30

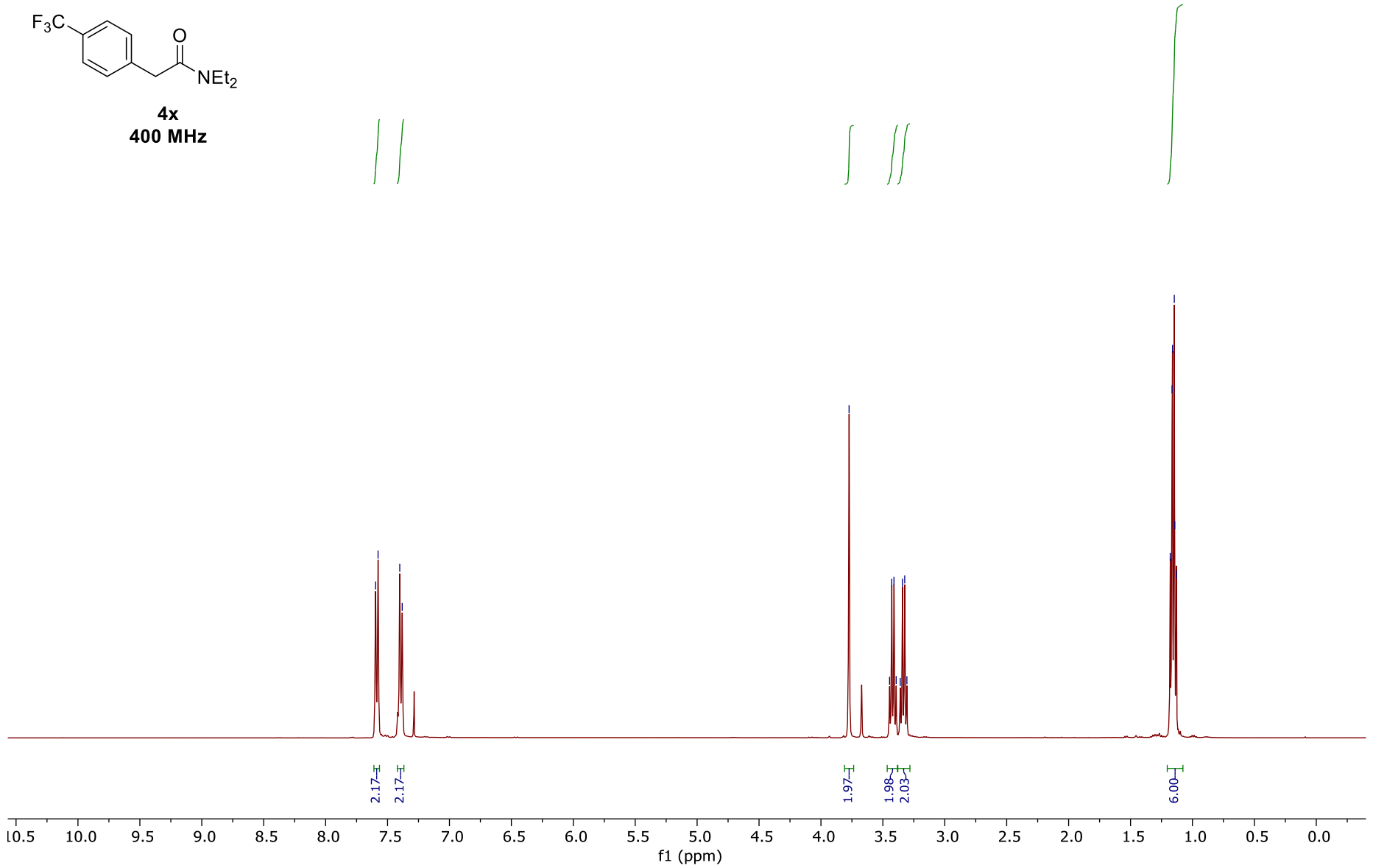


4x  
400 MHz

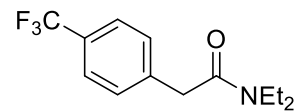
7.60  
7.58  
7.40  
7.38

3.77  
3.45  
3.43  
3.41  
3.39  
3.36  
3.34  
3.32  
3.31

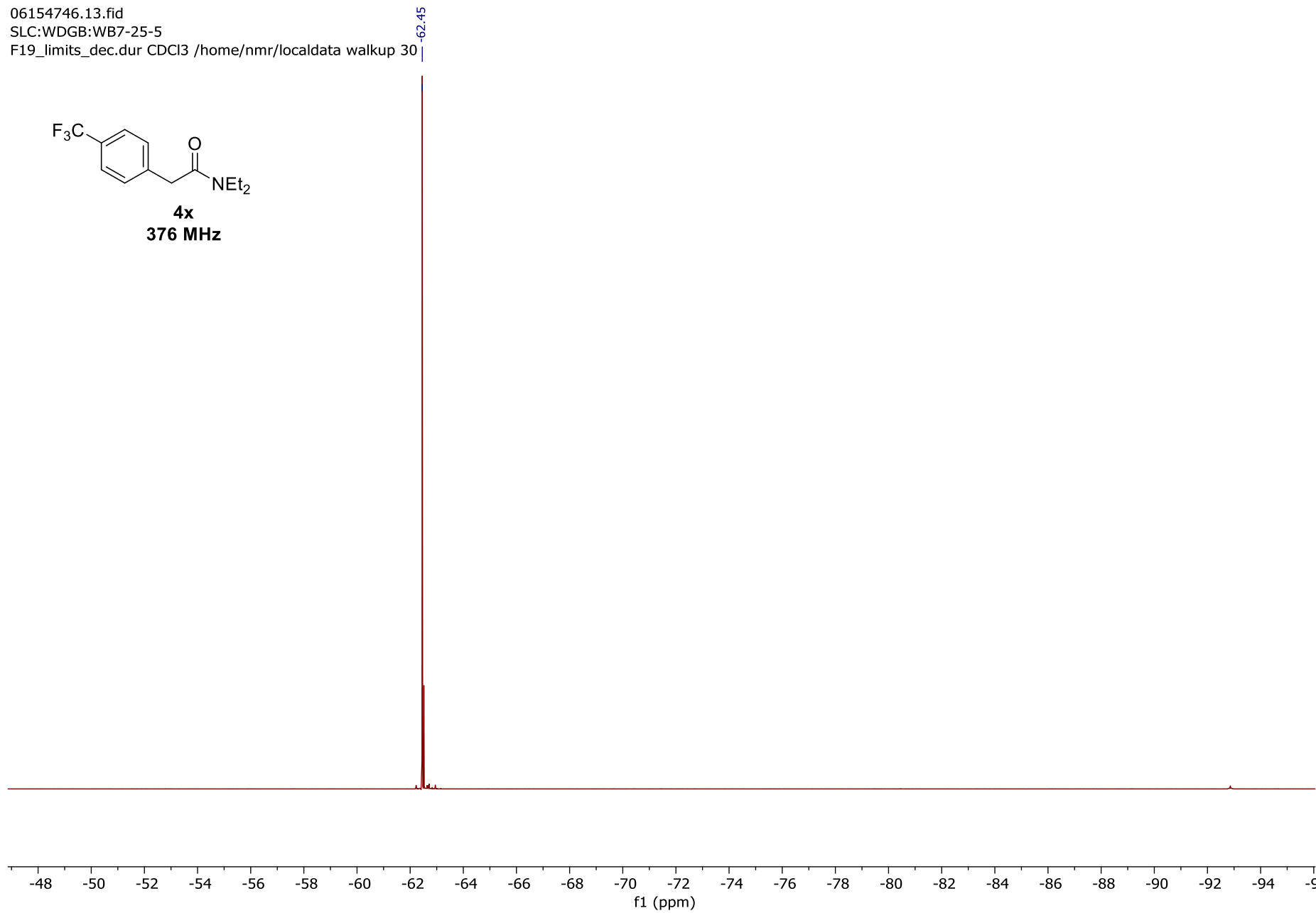
1.18  
1.16  
1.15  
1.14  
1.13



06154746.13.fid  
SLC:WDGB:WB7-25-5  
F19\_limits\_dec.dur CDCl3 /home/nmr/localdata walkup 30



**4x**  
**376 MHz**

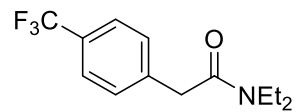


06154746.14.fid  
SLC:WDGB:WB7-25-5  
Carbon.dur CDCl3 /home/nmr/localdata/walkup 30

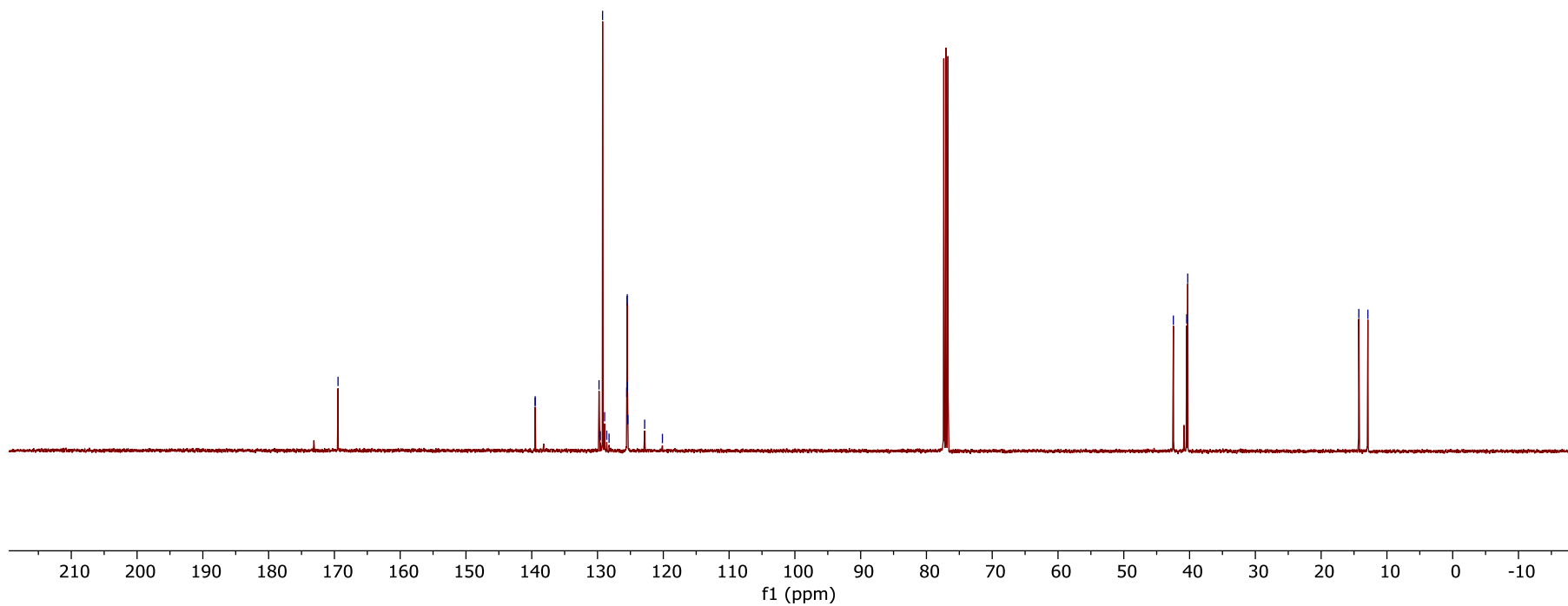
139.49  
139.47  
129.77  
129.57  
129.23  
128.92  
128.60  
128.25  
125.56  
125.52  
125.48  
125.44  
125.39  
122.84  
120.13

42.46  
40.44  
40.28

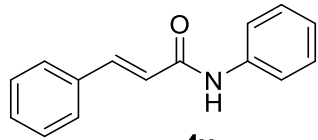
14.28  
12.89



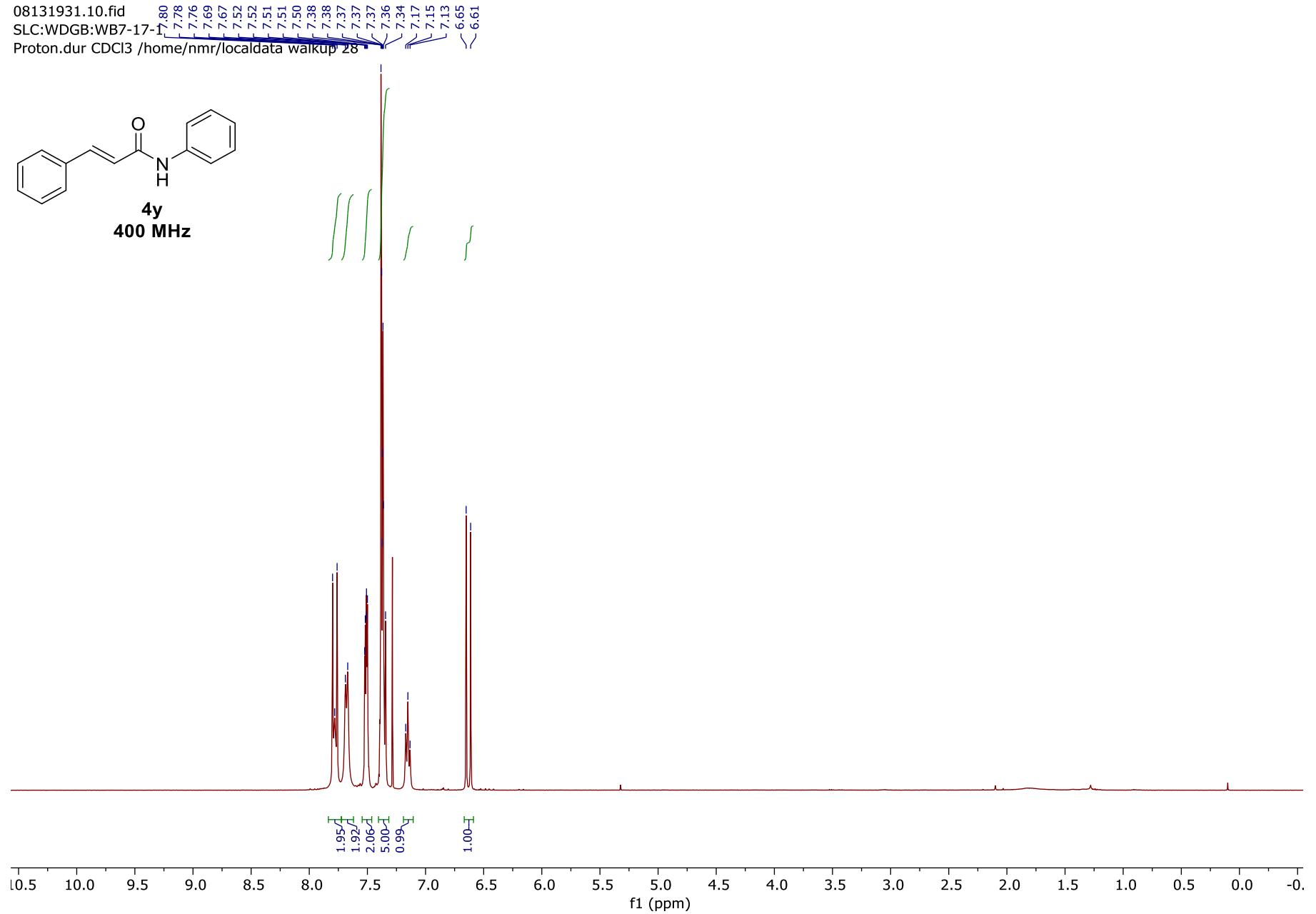
4x  
101 MHz



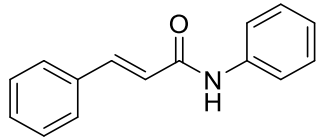
08131931.10.fid  
SLC:WDGB:WB7-17-1  
Proton.dur CDCl3 /home/nmr/localdata walkup 28



4y  
400 MHz

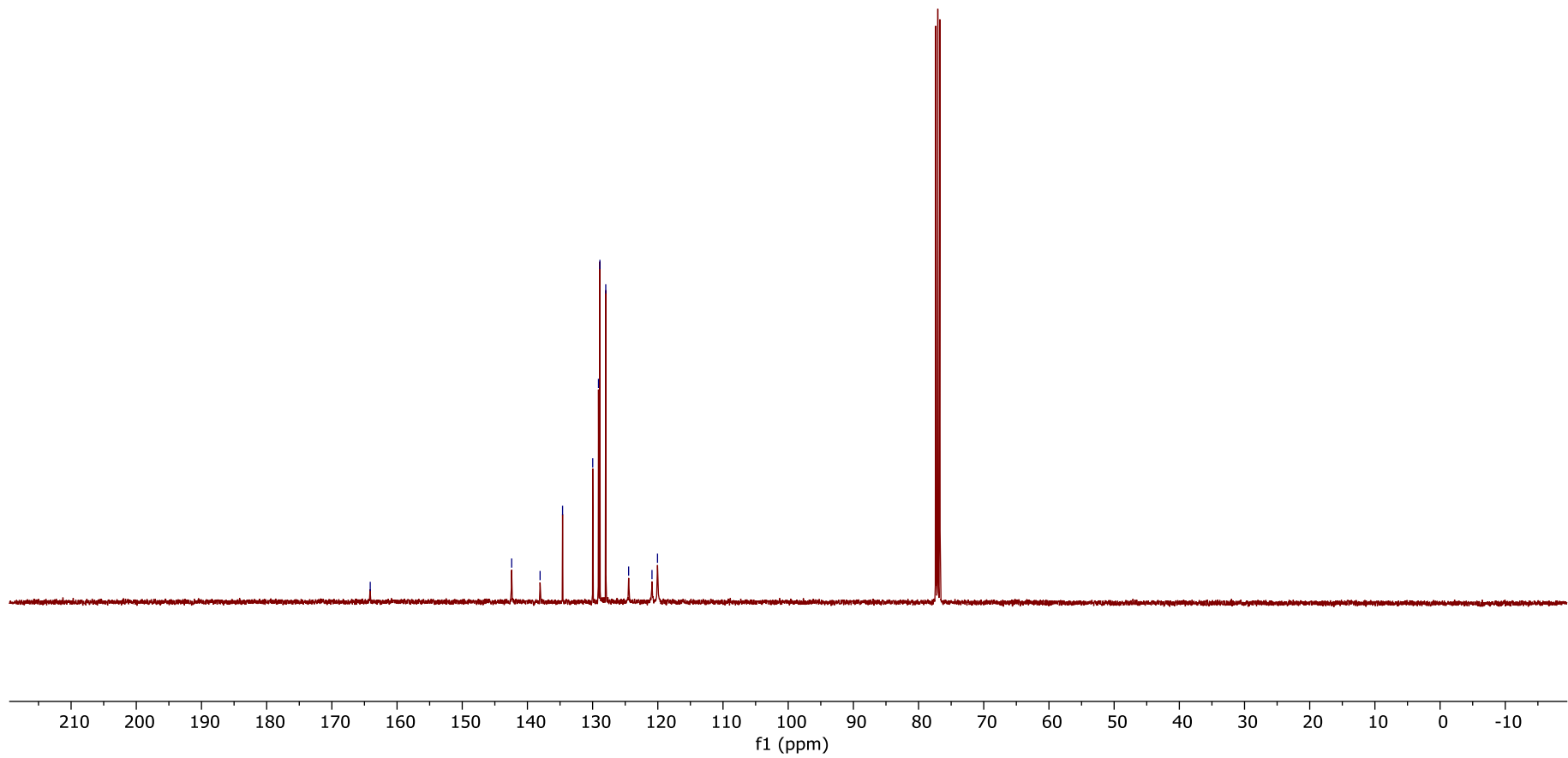


08131931.11.fid  
SLC:WDGB:WB7-17-1  
Carbon.dur CDCl3 /home/nmr/localdata walkup 28



**4y**  
**101 MHz**

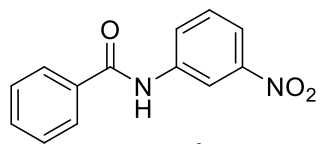
164.11  
142.42  
138.06  
134.61  
129.98  
129.10  
128.88  
127.99  
124.47  
120.89  
120.05



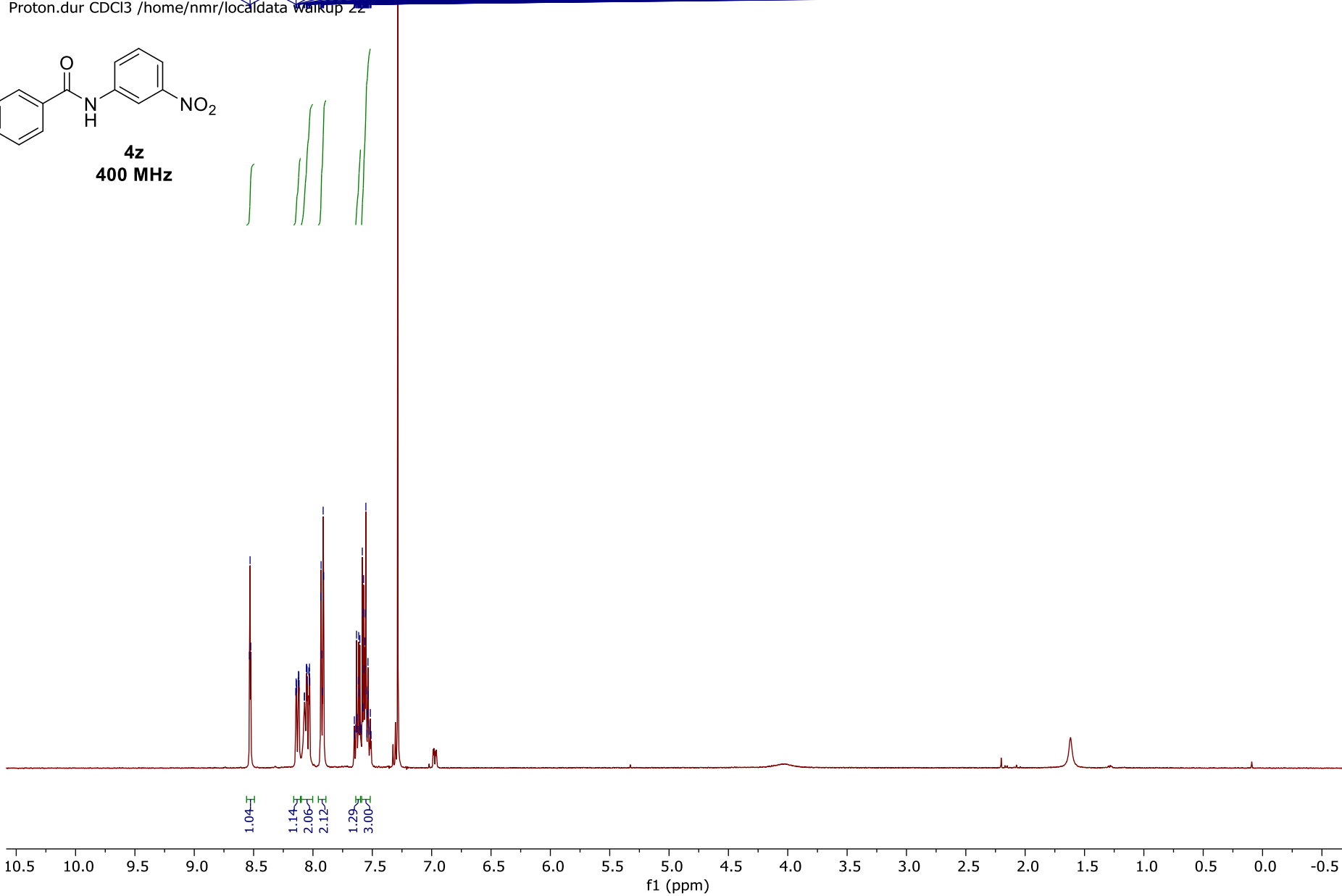
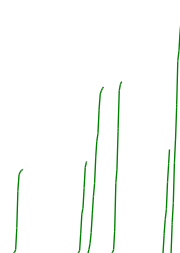


06132030.10.fid  
SLC:WDGB:WB7-21  
Proton.dur CDCl3 /home/nmr/localdata/waiku

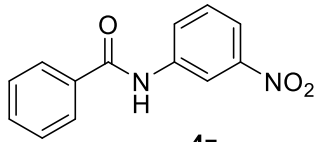
8.54  
8.53  
8.52  
8.14  
8.14  
8.14  
8.12  
8.12  
8.12  
8.12  
8.07  
8.07  
8.06  
8.05  
8.05  
8.05  
8.03  
8.03  
8.03  
7.93  
7.93  
7.92  
7.91  
7.91  
7.65  
7.65  
7.64  
7.63  
7.63  
7.62  
7.62  
7.61  
7.61  
7.60  
7.60  
7.60  
7.59  
7.58  
7.58  
7.57  
7.57  
7.56  
7.56  
7.55  
7.55  
7.54  
7.54  
7.53  
7.53  
7.52  
7.52  
7.51



4z  
400 MHz

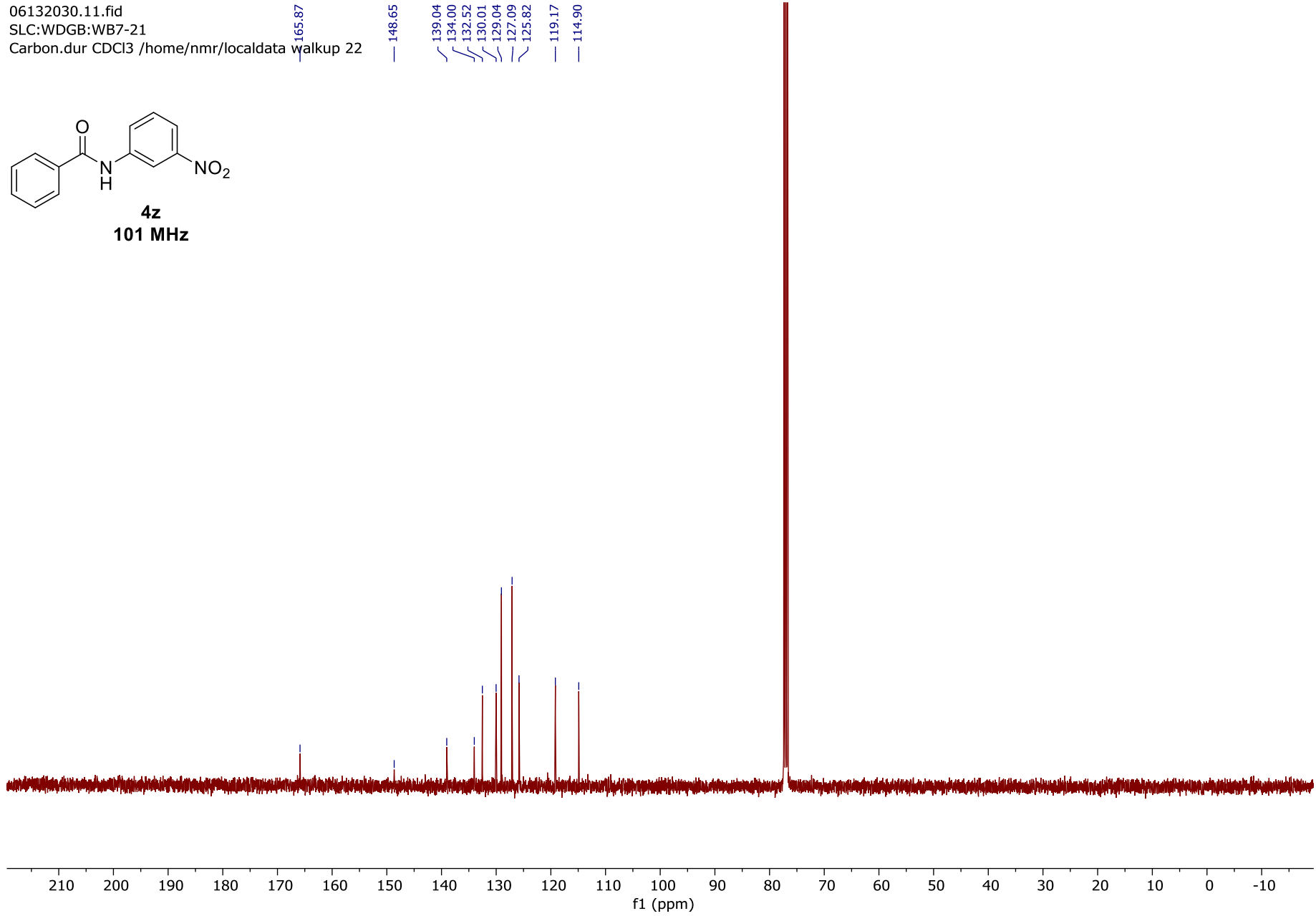


06132030.11.fid  
SLC:WDGB:WB7-21  
Carbon.dur CDCl3 /home/nmr/localdata walkup 22

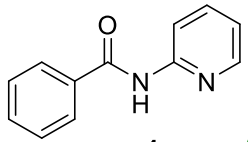


**4z**  
**101 MHz**

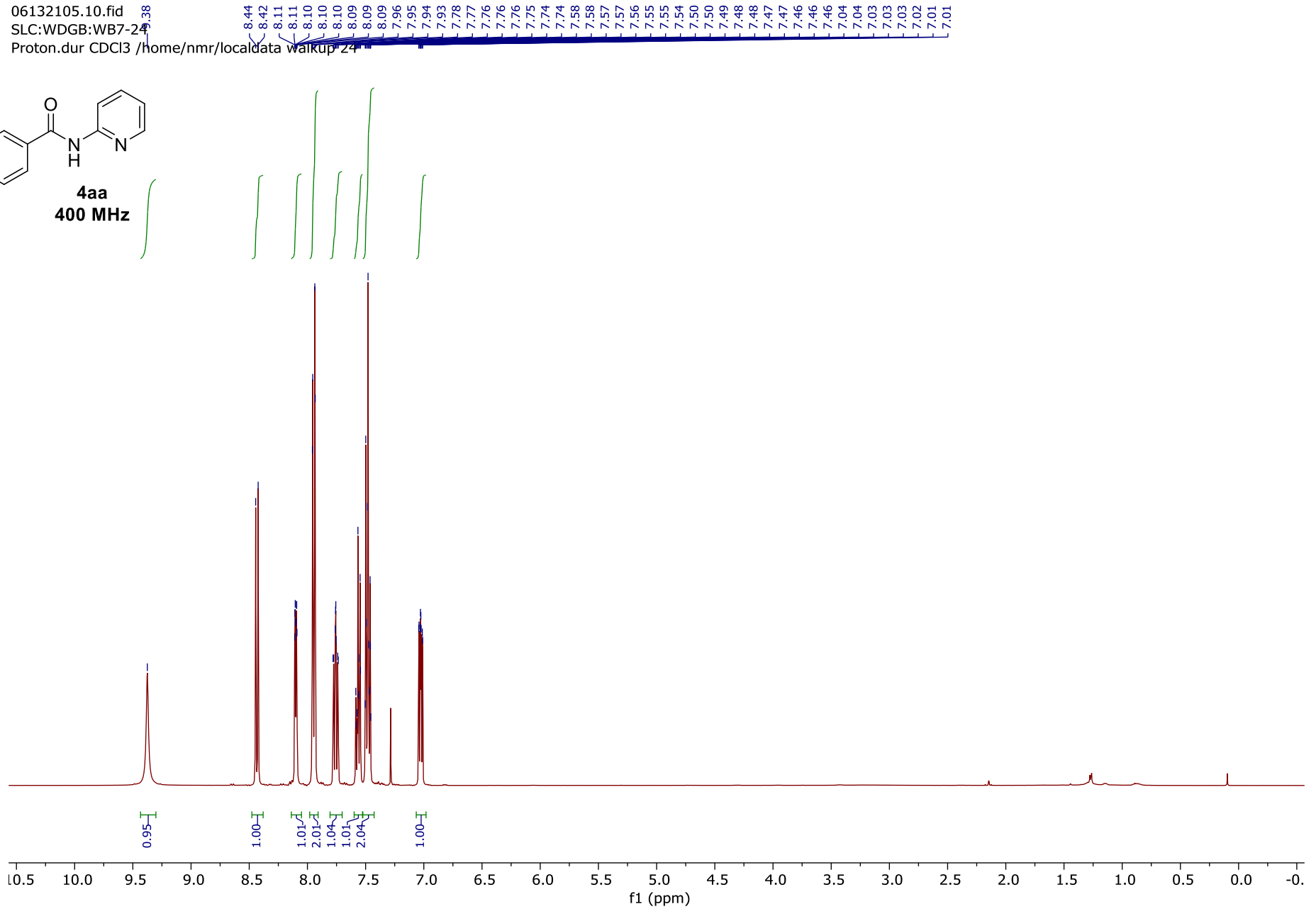
165.87  
148.65  
139.04  
134.00  
132.52  
130.01  
129.04  
127.09  
125.82  
119.17  
114.90



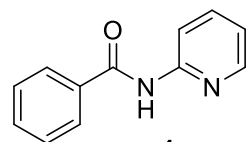
06132105.10.fid  
SLC:WDGB:WB7-24  
Proton.dur CDCl3 /home/nmr/localdata/walkup/24



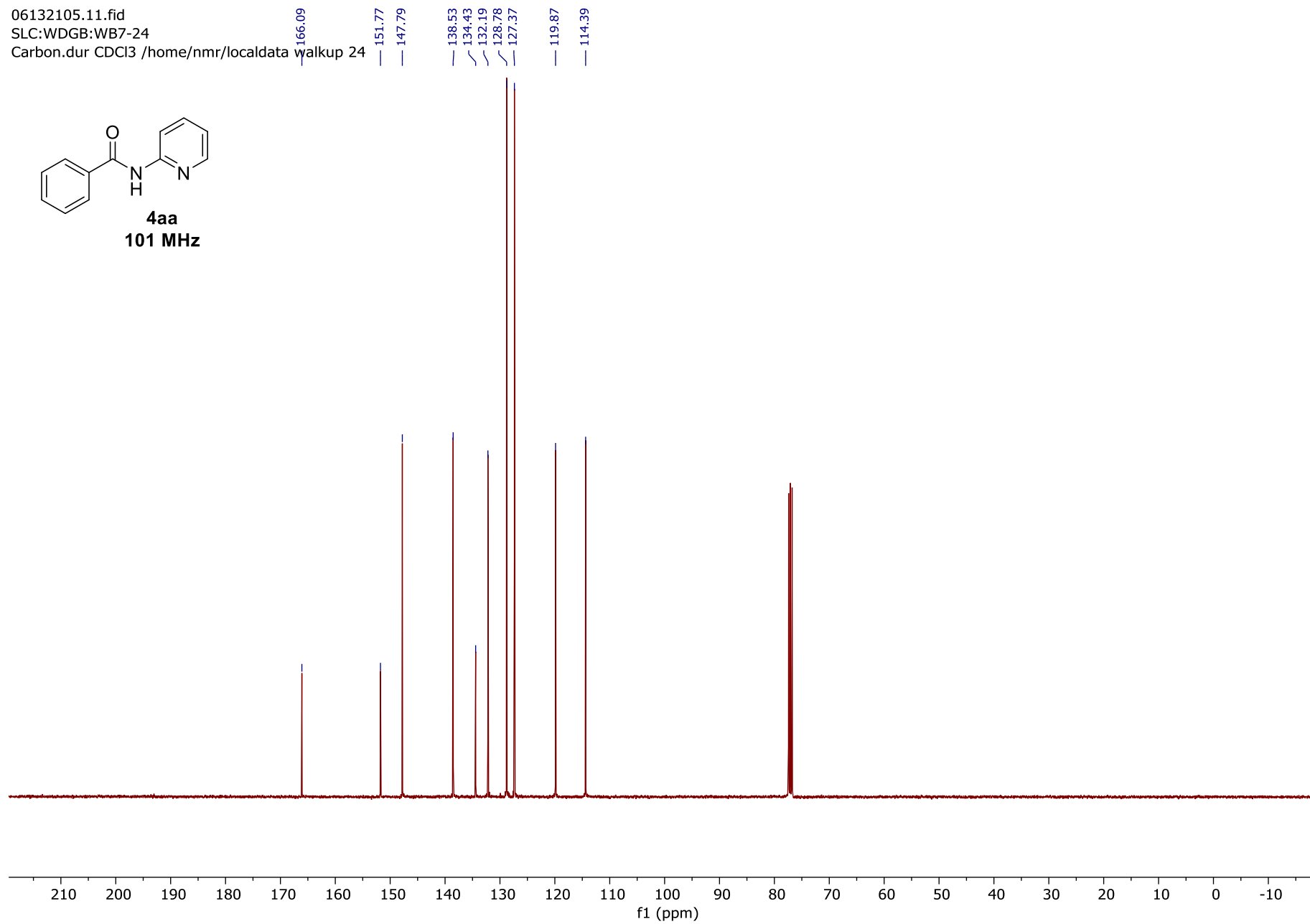
**4aa**  
400 MHz



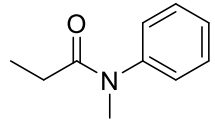
06132105.11.fid  
SLC:WDGB:WB7-24  
Carbon.dur CDCl3 /home/nmr/localdata walkup 24



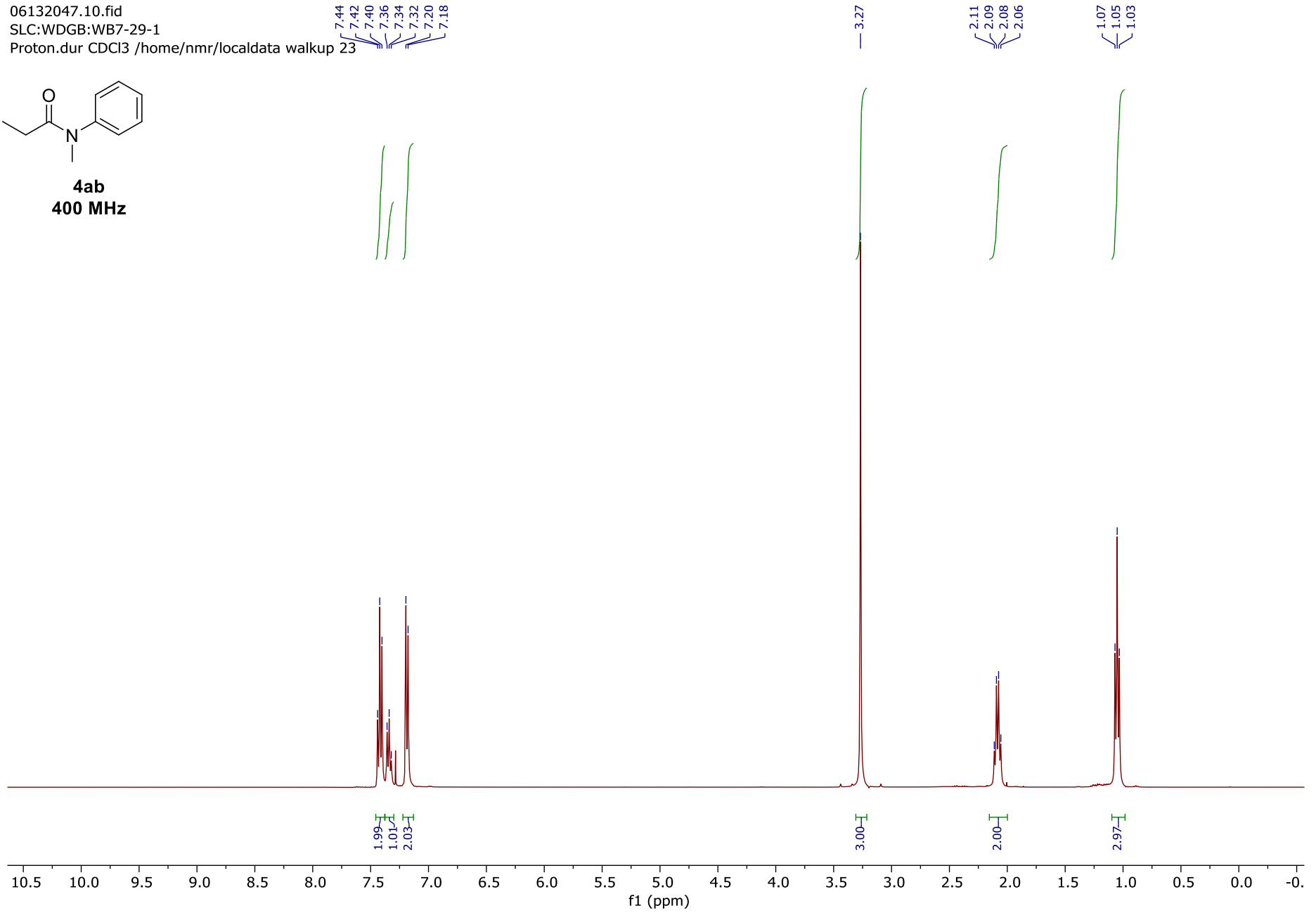
**4aa**  
101 MHz



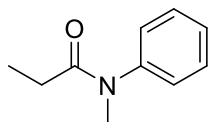
06132047.10.fid  
SLC:WDGB:WB7-29-1  
Proton.dur CDCl3 /home/nmr/localdata walkup 23



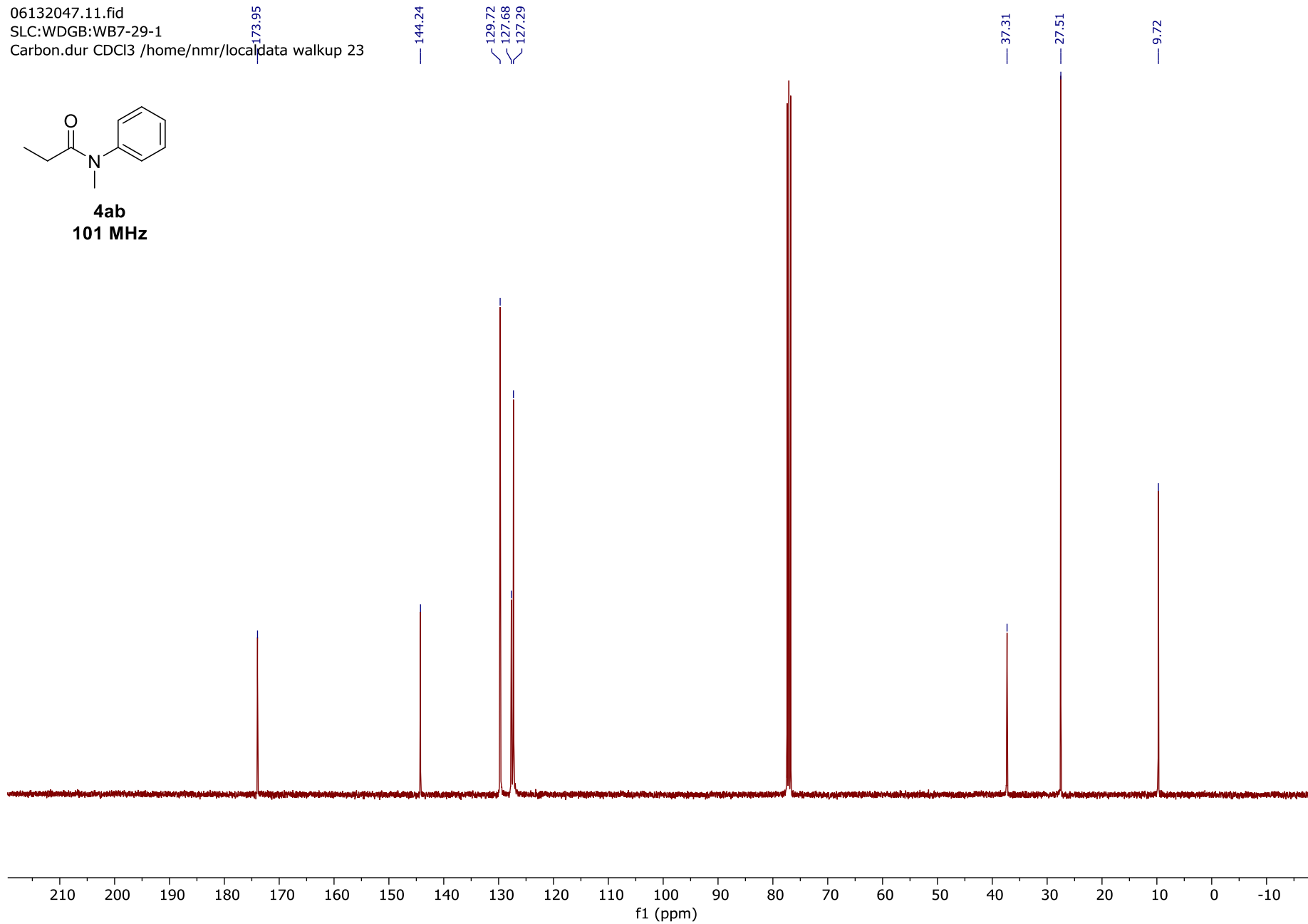
**4ab**  
**400 MHz**



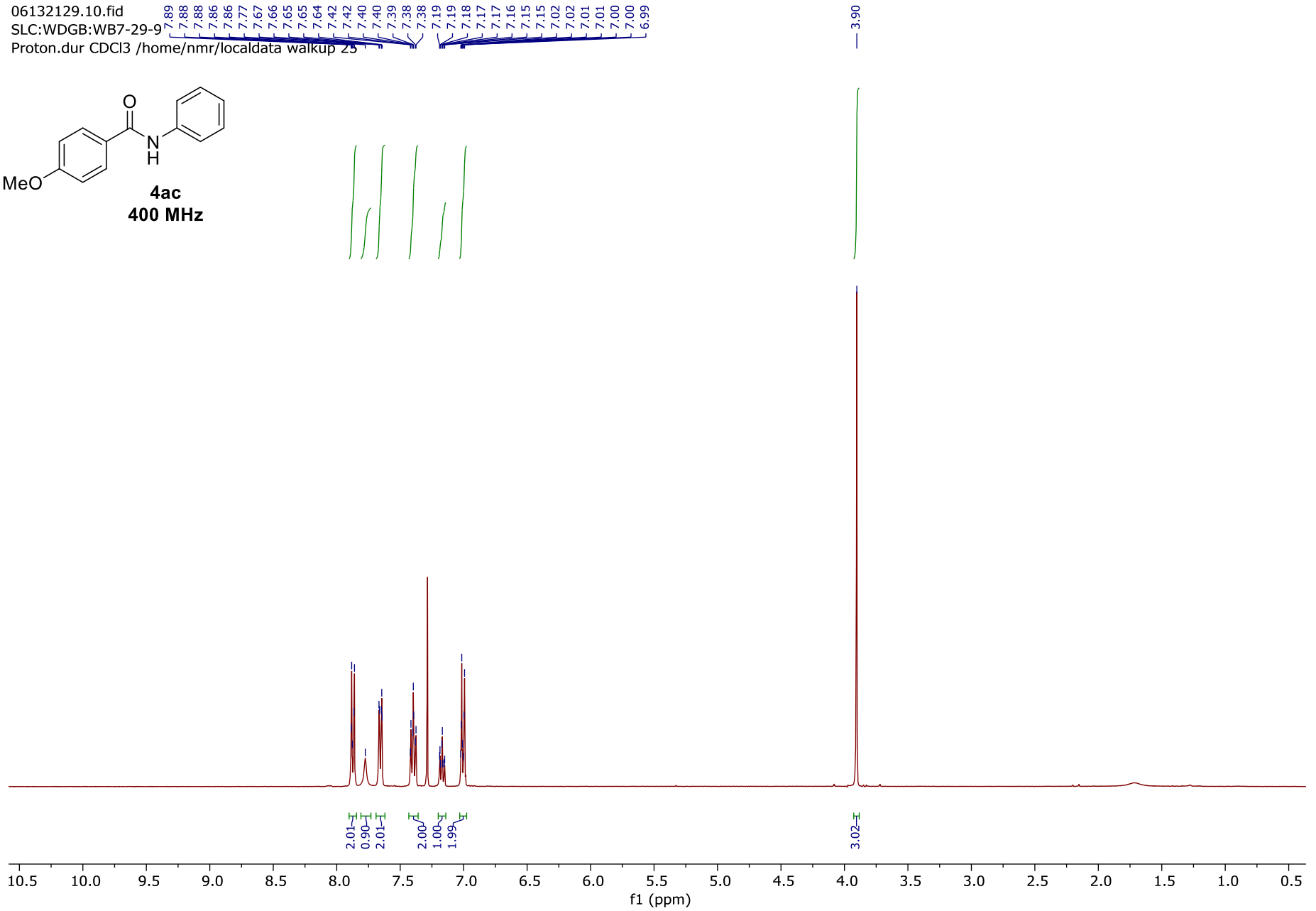
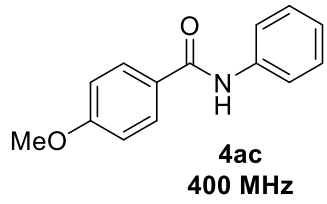
06132047.11.fid  
SLC:WDGB:WB7-29-1  
Carbon.dur CDCl3 /home/nmr/local/data/walkup 23



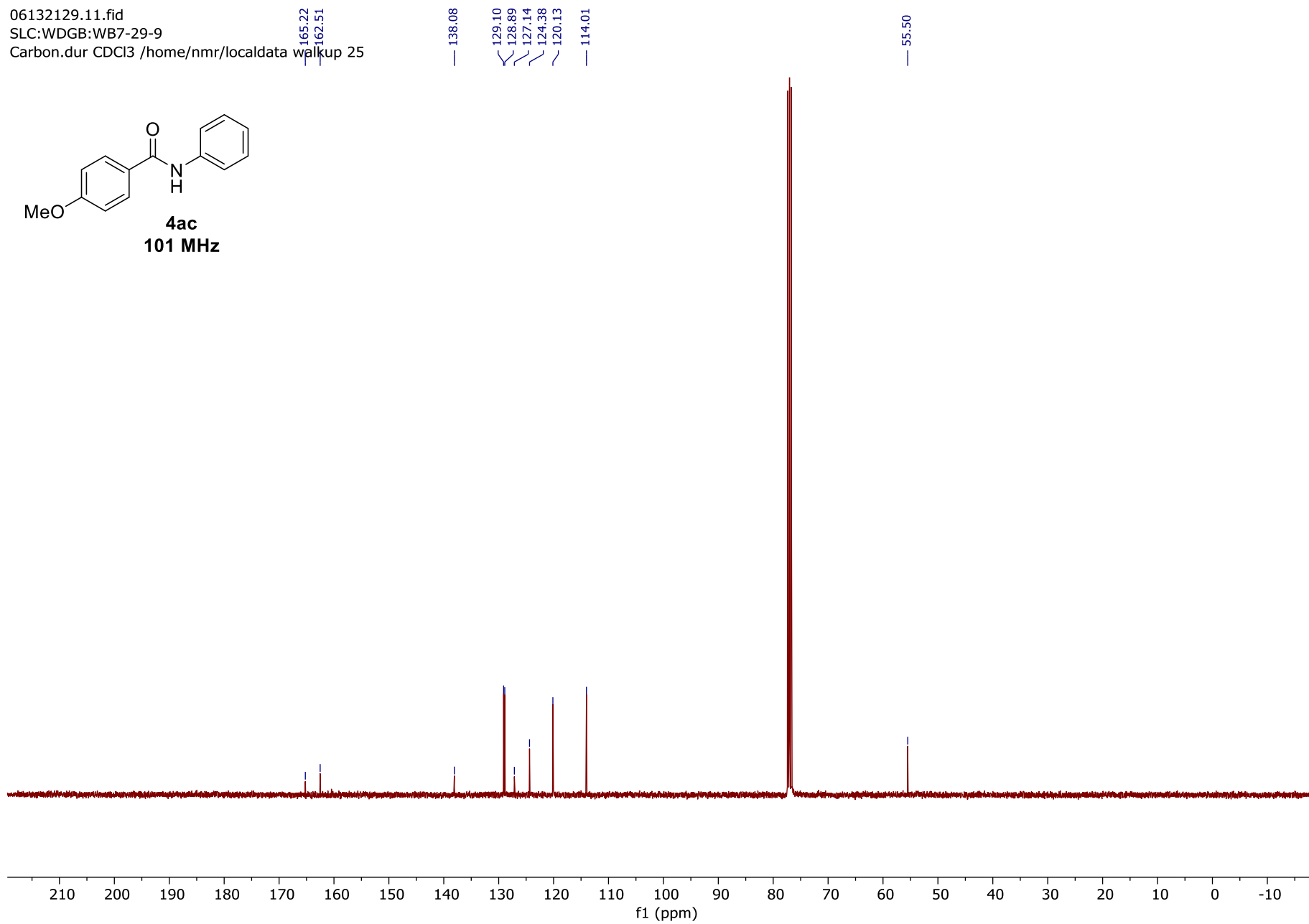
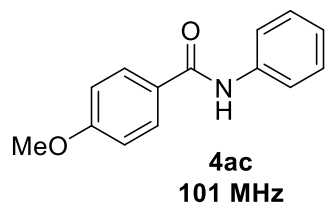
**4ab**  
101 MHz



06132129.10.fid  
SLC:WDGB:WB7-29-9  
Proton.dur CDCl3 /home/nmr/localdata walkup 25

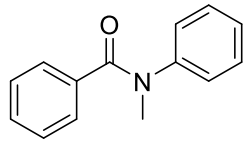


06132129.11.fid  
SLC:WDGB:WB7-29-9  
Carbon.dur CDCl3 /home/nmr/localdata walkup 25

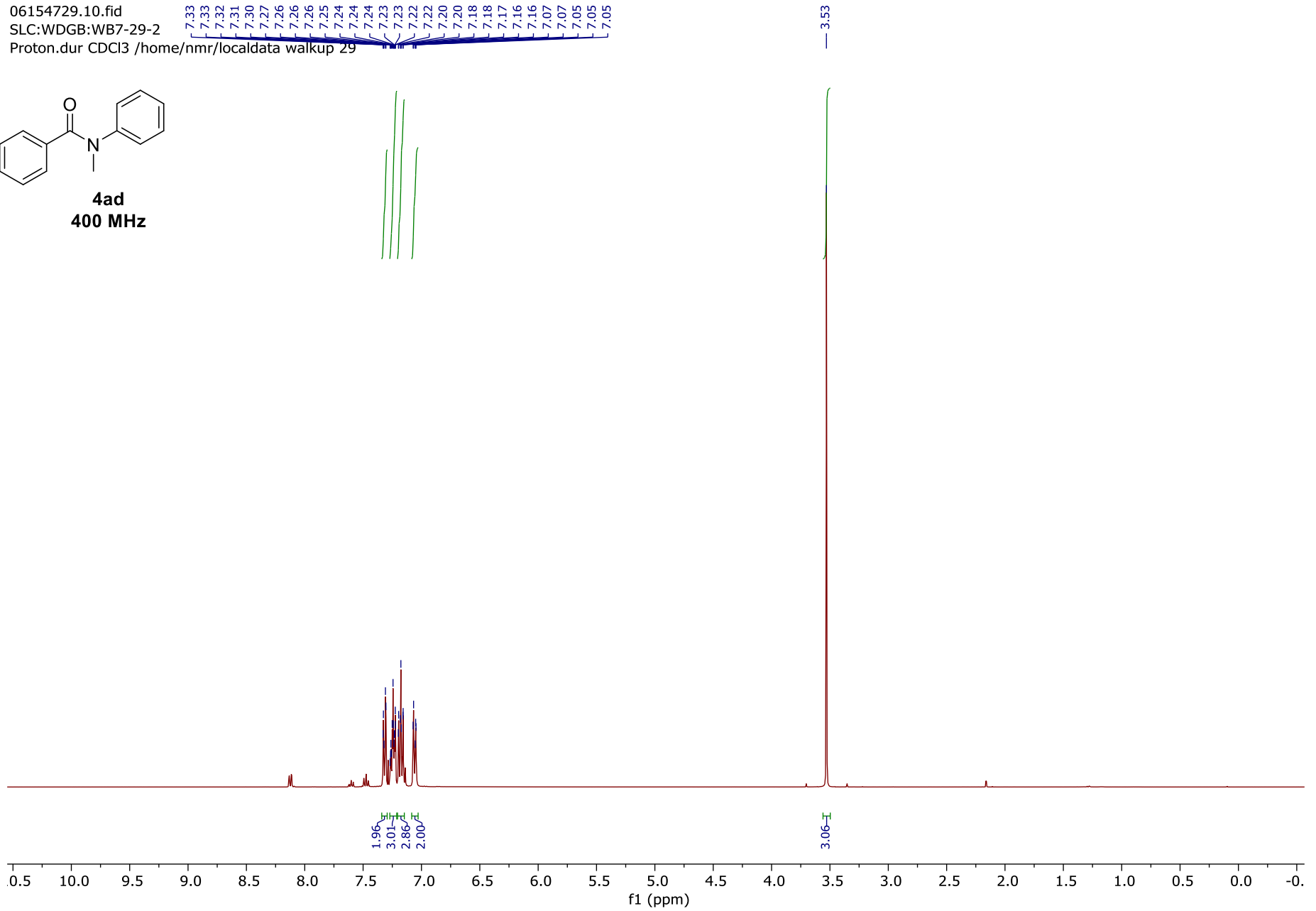




06154729.10.fid  
SLC:WDGB:WB7-29-2  
Proton.dur CDCl3 /home/nmr/localdata walkup 29



**4ad**  
**400 MHz**



06154729.11.fid  
SLC:WDGB:WB7-29-2  
Carbon.dur CDCl3 /home/nmr/localdata walkup 29

170.89

144.81

135.78

129.67

129.18

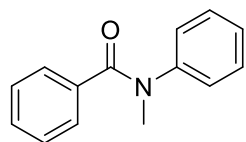
128.71

127.75

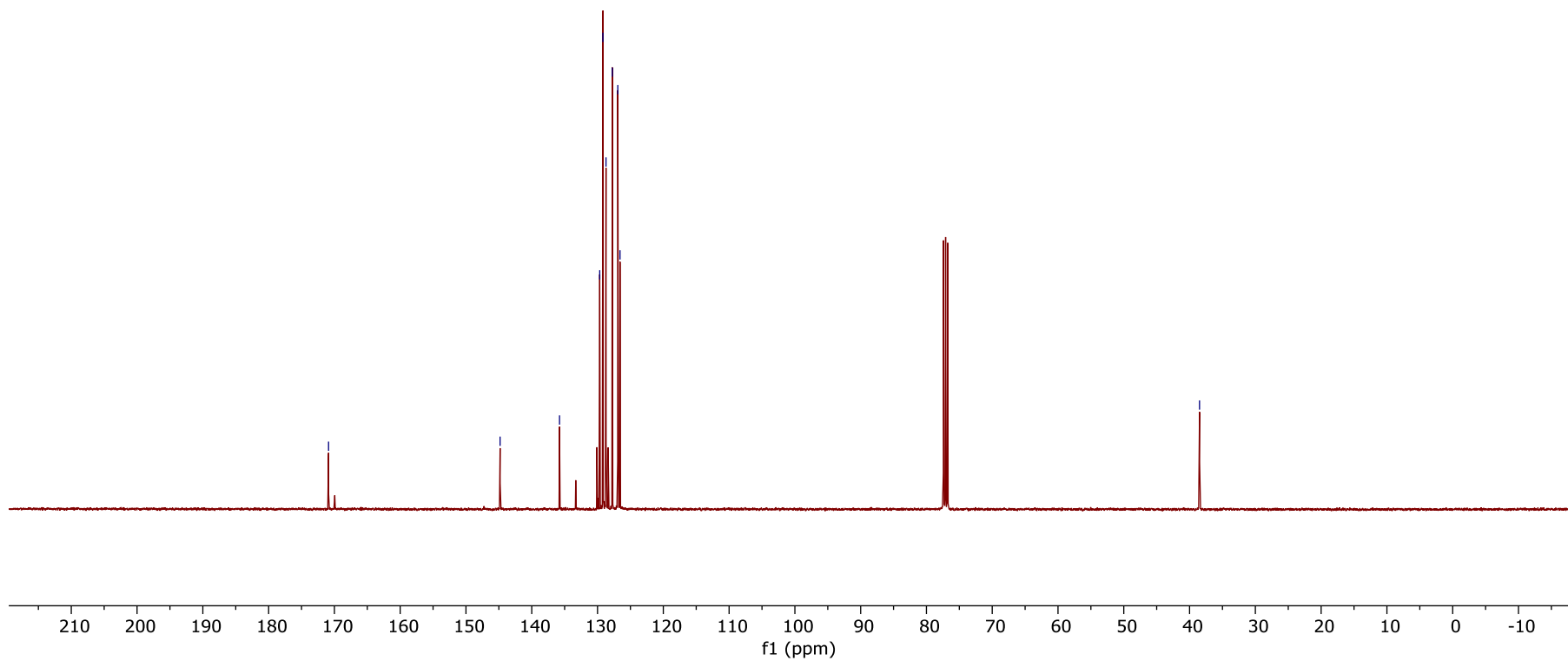
126.92

126.58

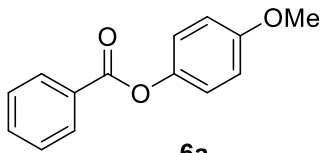
38.48



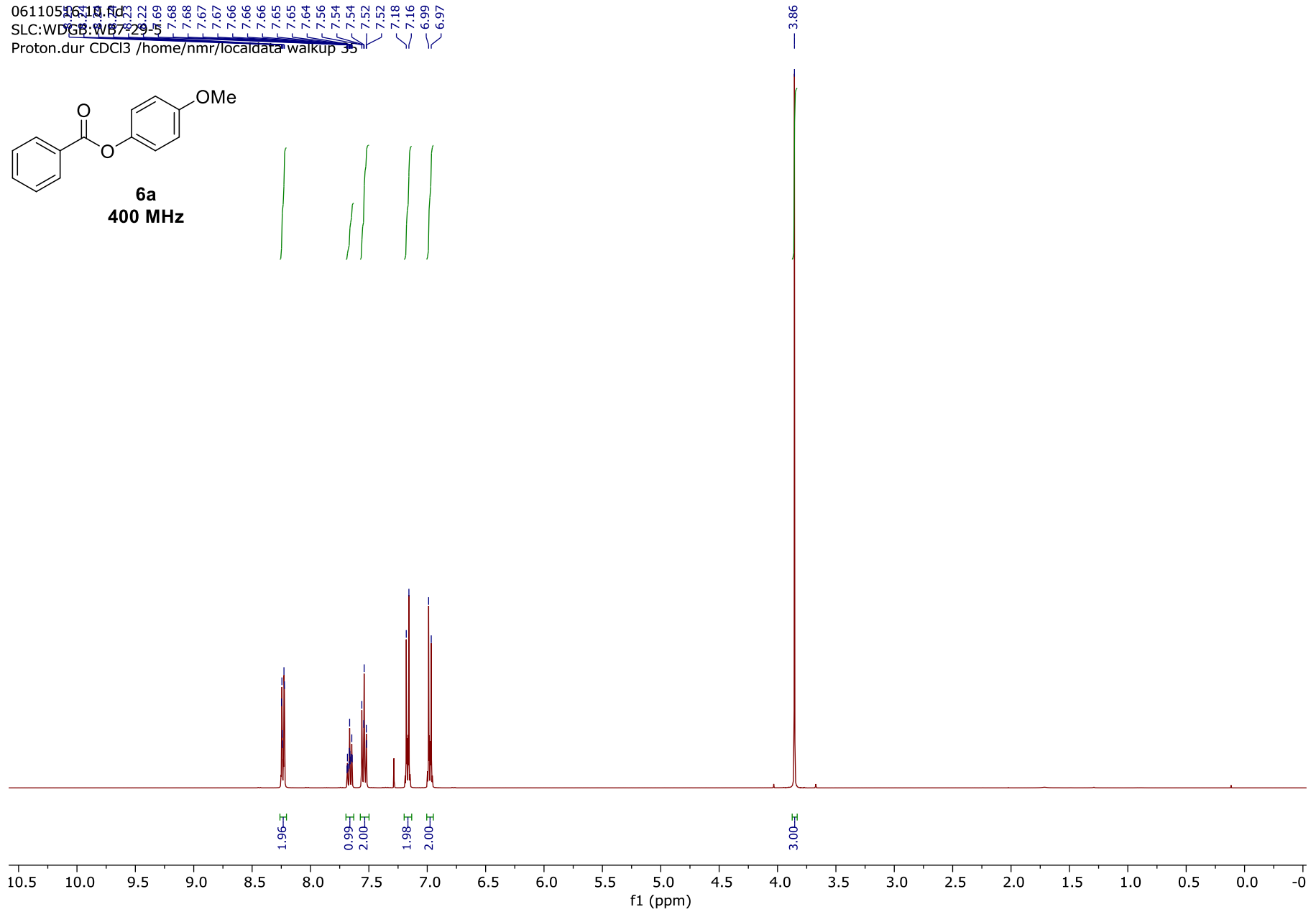
**4ad**  
**101 MHz**



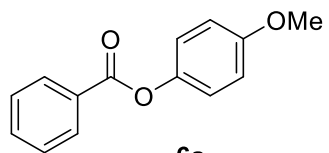
06110516310.00  
SLC:WDSB-WB729-5  
Proton.dur CDCl3 /home/nmr/localdata/walkup 35



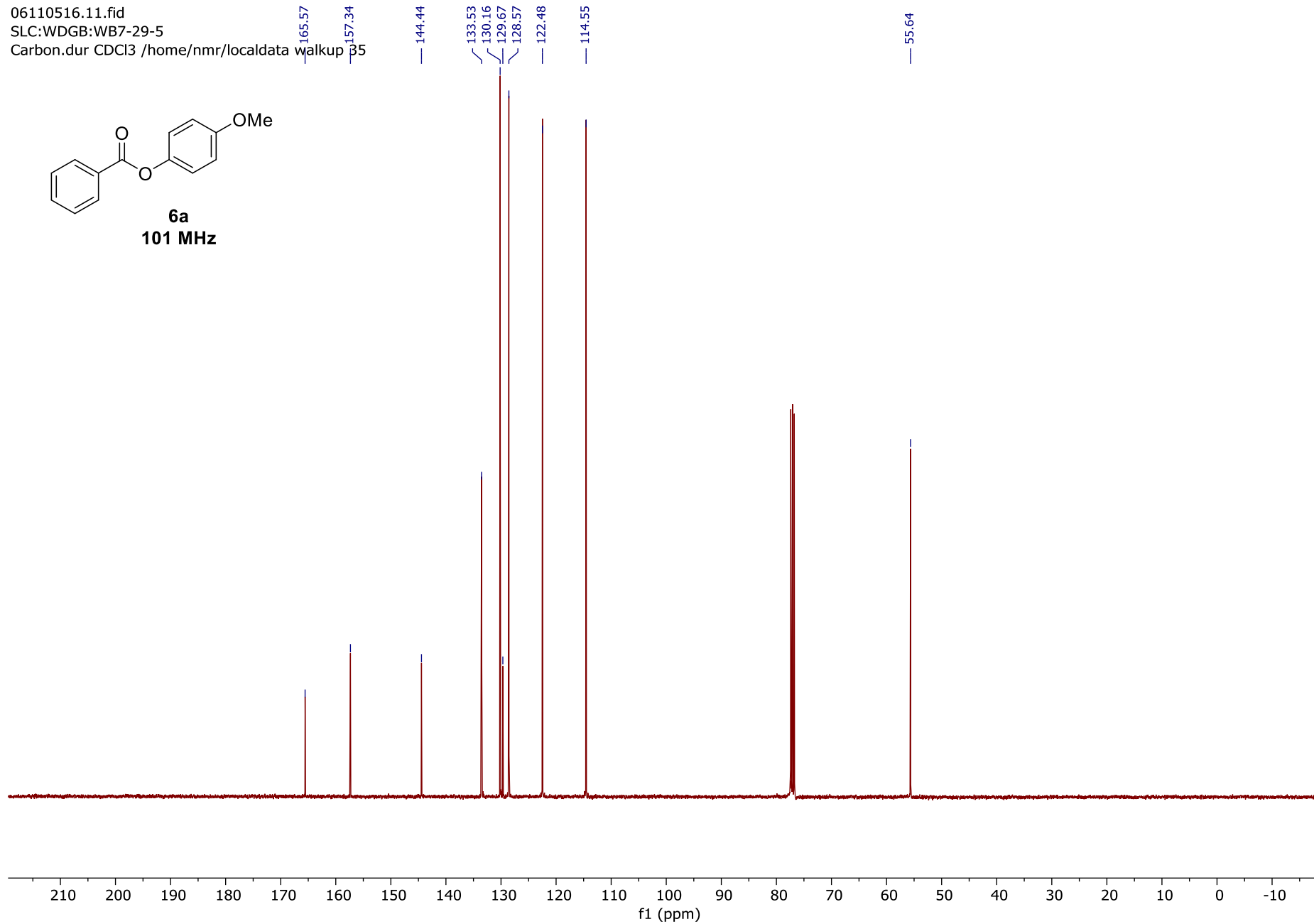
400 MHz



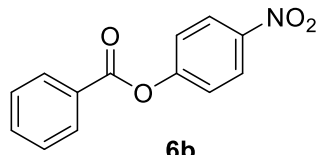
06110516.11.fid  
SLC:WDGB:WB7-29-5  
Carbon.dur CDCl3 /home/nmr/localdata walkup 35



**6a**  
101 MHz

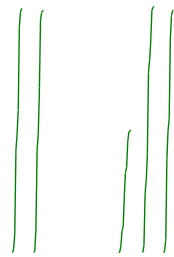


06110543.10.fid  
SLC:WDGB:WB7-29-8  
Proton.dur CDCl3 /home/nmr/localdata/walkup/36



**6b**  
400 MHz

8.37  
8.35  
8.24  
8.24  
8.24  
8.24  
8.23  
8.22  
8.22  
7.74  
7.73  
7.73  
7.72  
7.72  
7.71  
7.71  
7.71  
7.70  
7.70  
7.69  
7.59  
7.57  
7.55  
7.46  
7.44

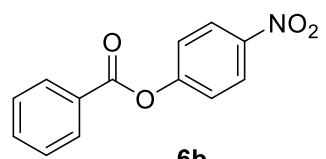


2.00  
1.98  
1.00  
2.01  
1.98

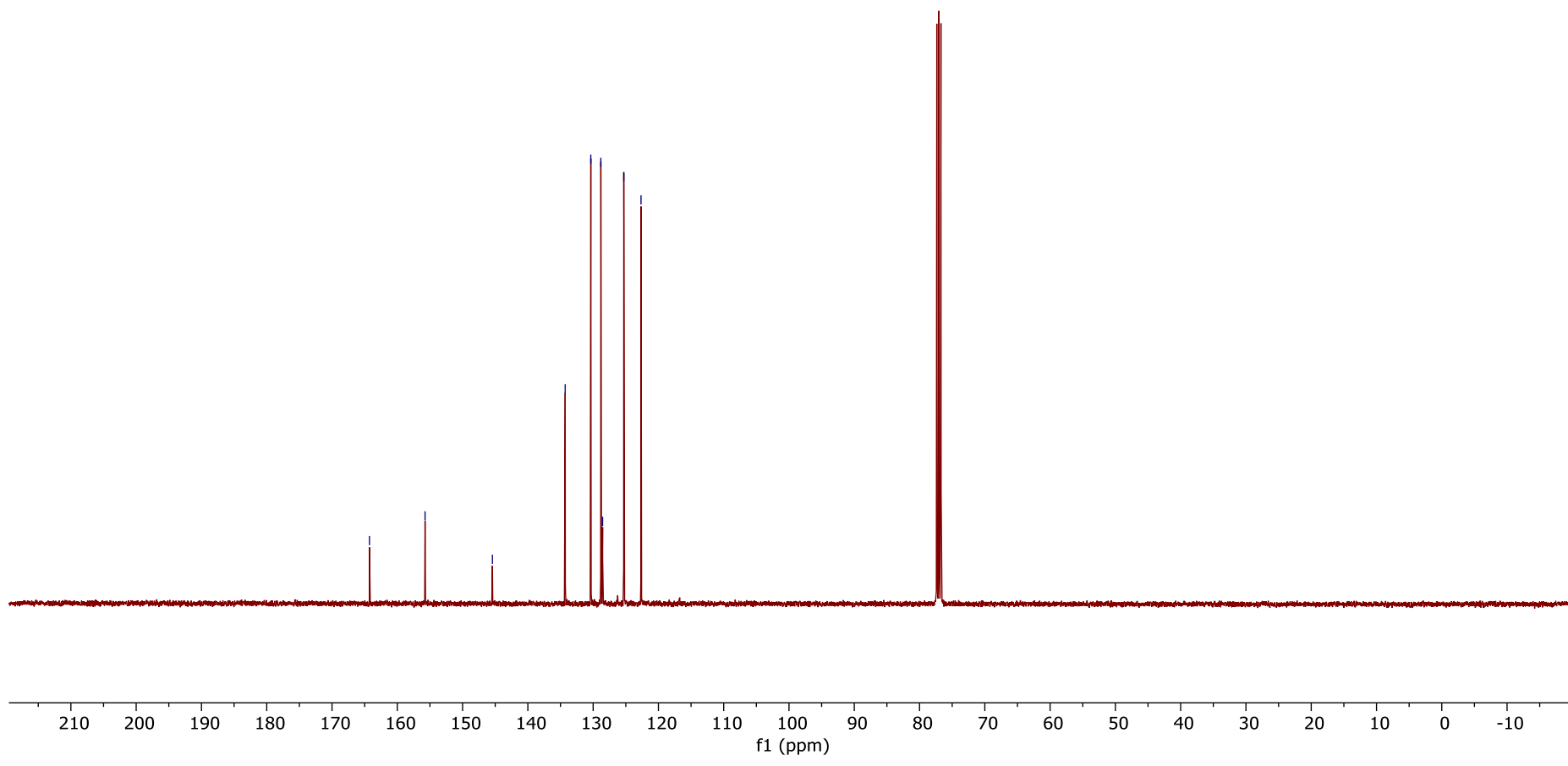
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5  
f1 (ppm)

06110543.11.fid  
SLC:WDGB:WB7-29-8  
Carbon.dur CDCl3 /home/nmr/localdata

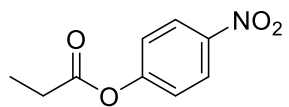
164.26  
155.75  
145.43  
134.28  
130.36  
128.82  
128.55  
125.31  
122.67



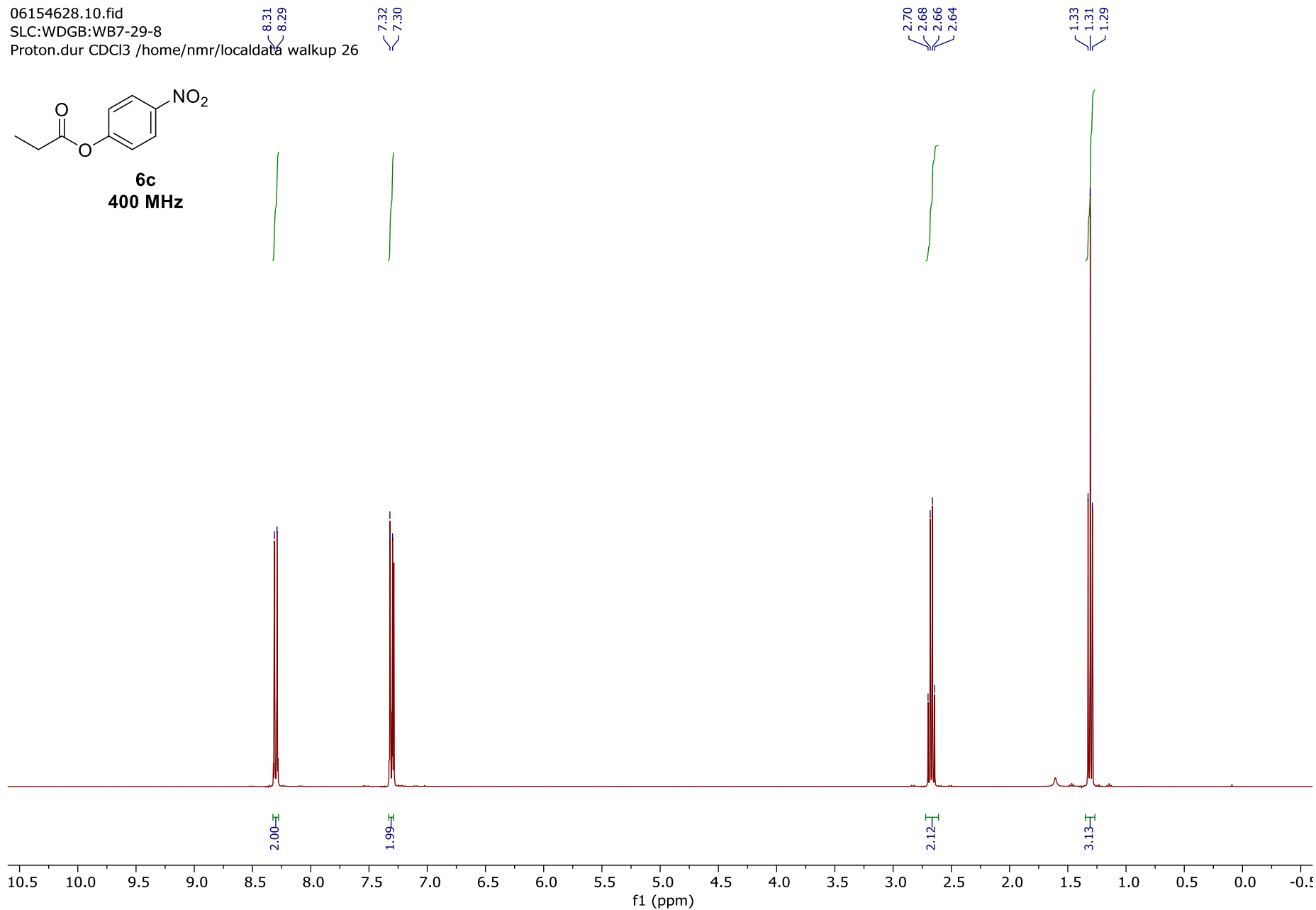
**6b**  
101 MHz



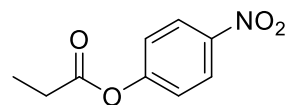
06154628.10.fid  
SLC:WDGB:WB7-29-8  
Proton.dur CDCl3 /home/nmr/localdata walkup 26



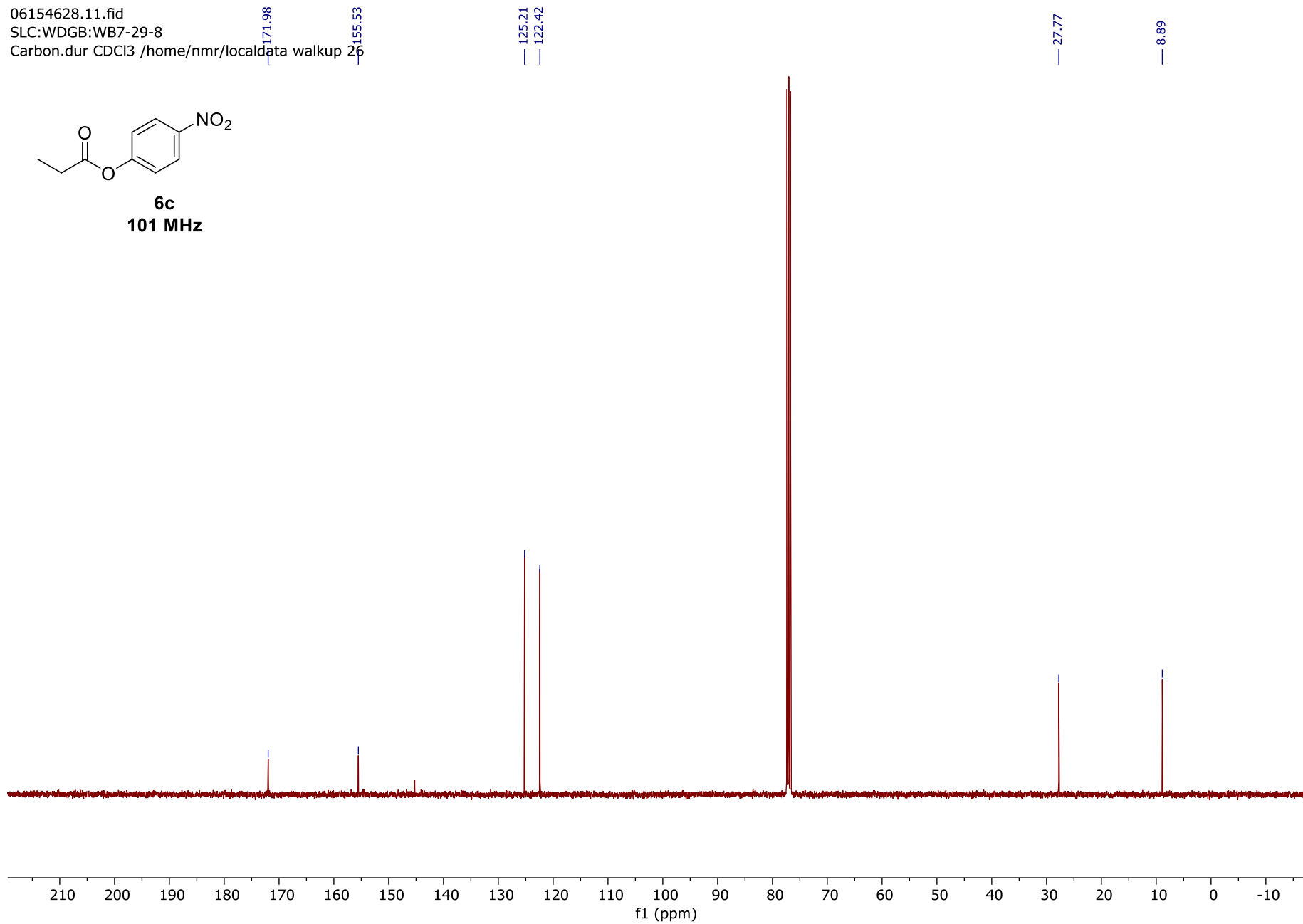
**6c**  
400 MHz



06154628.11.fid  
SLC:WDGB:WB7-29-8  
Carbon.dur CDCl3 /home/nmr/localdata walkup 26

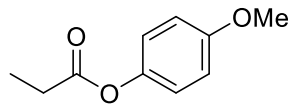


**6c**  
101 MHz

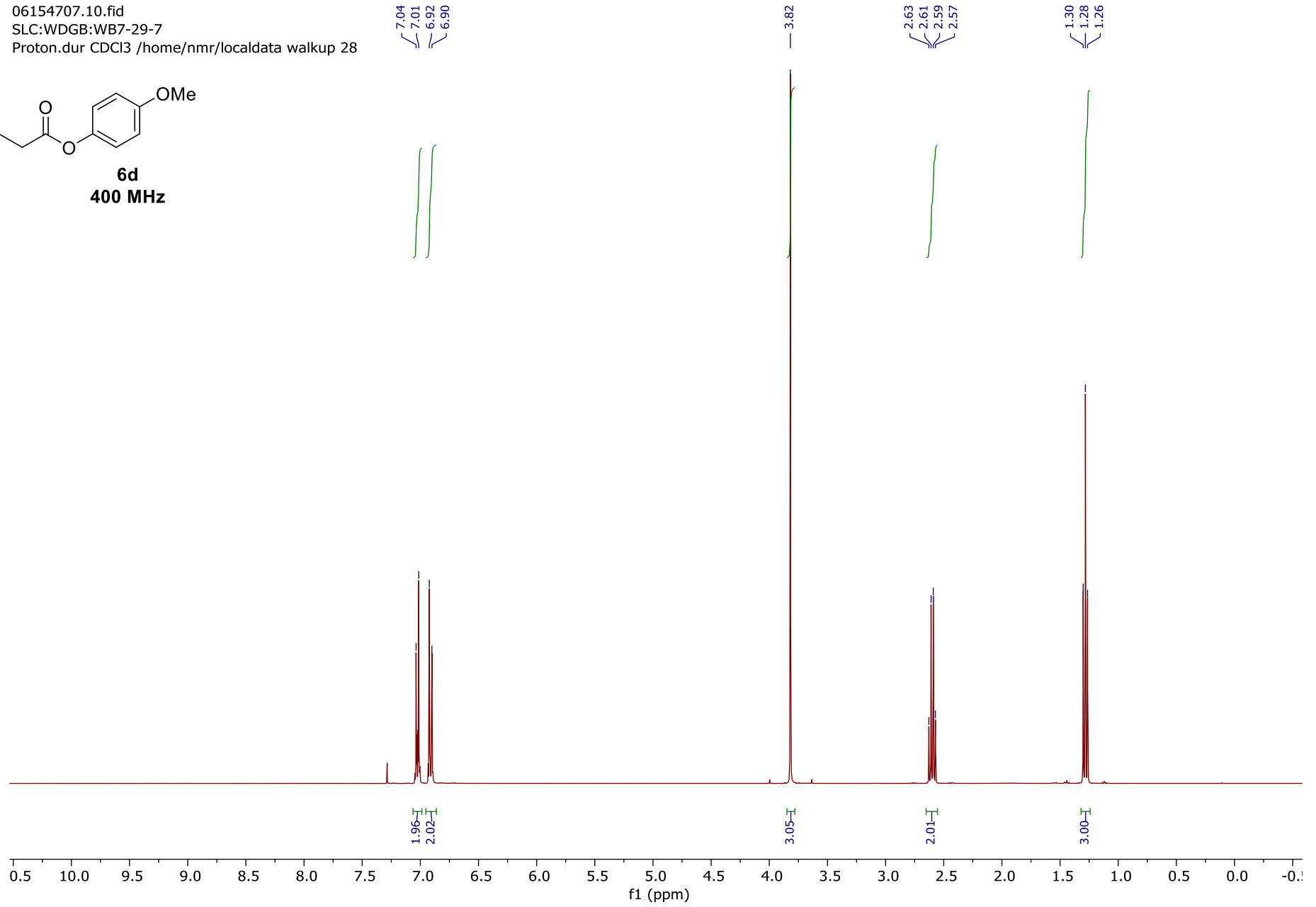




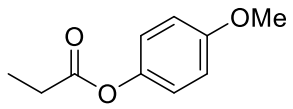
06154707.10.fid  
SLC:WDGB:WB7-29-7  
Proton.dur CDCl3 /home/nmr/localdata walkup 28



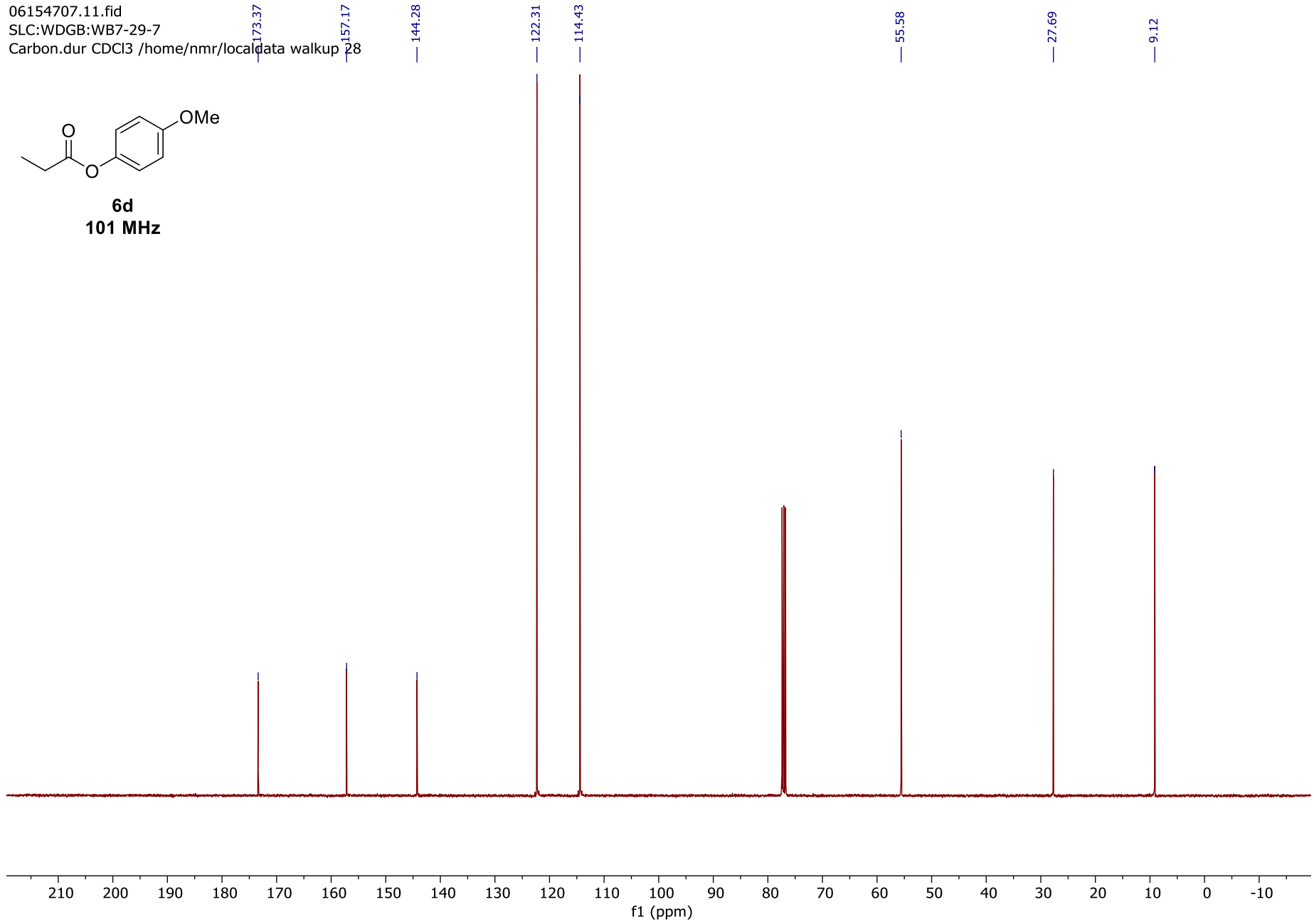
**6d**  
**400 MHz**



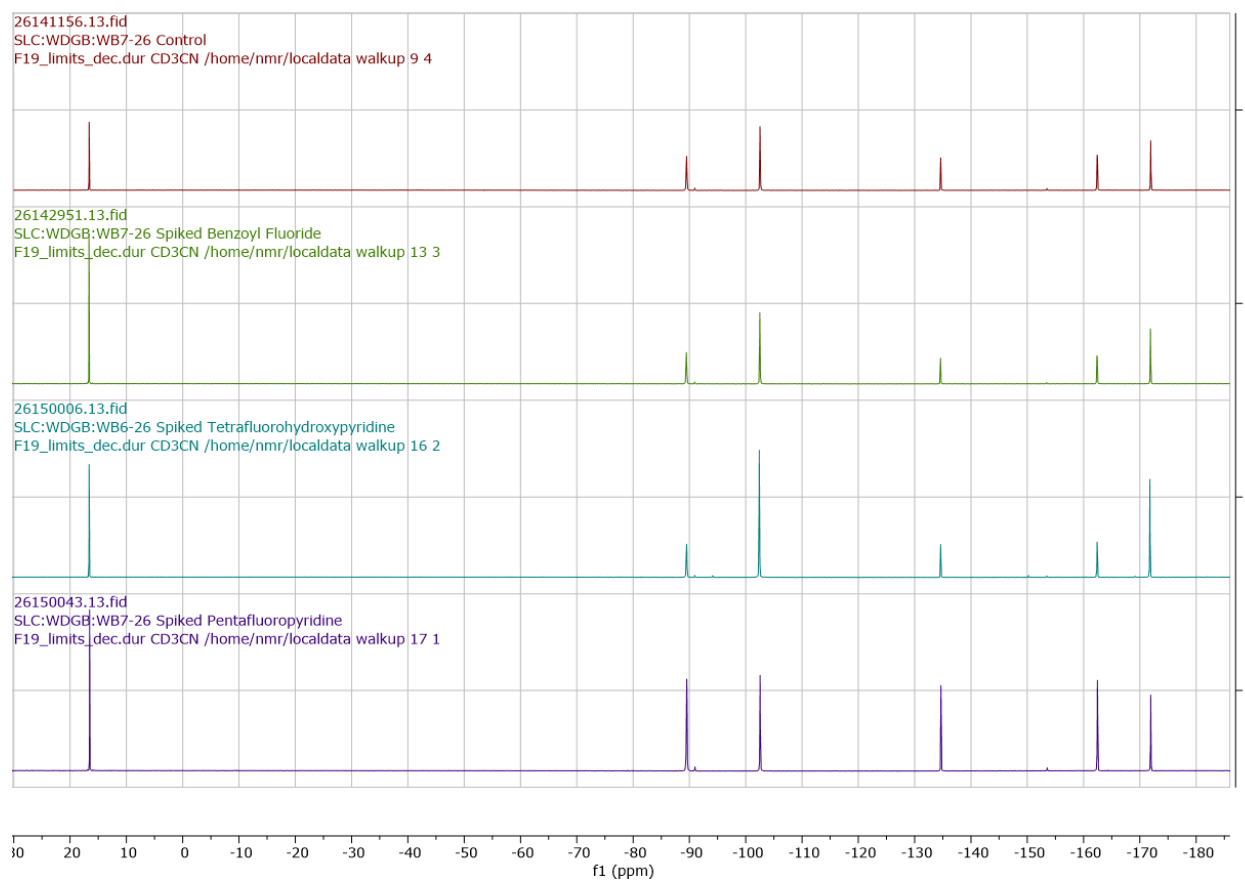
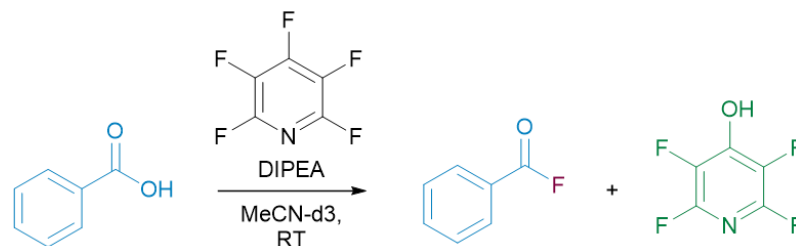
06154707.11.fid  
SLC:WDGB:WB7-29-7  
Carbon.dur CDCl3 /home/nmr/local/data/walkup/28



**6d**  
**101 MHz**



## NMR Study of Acyl Fluoride Formation



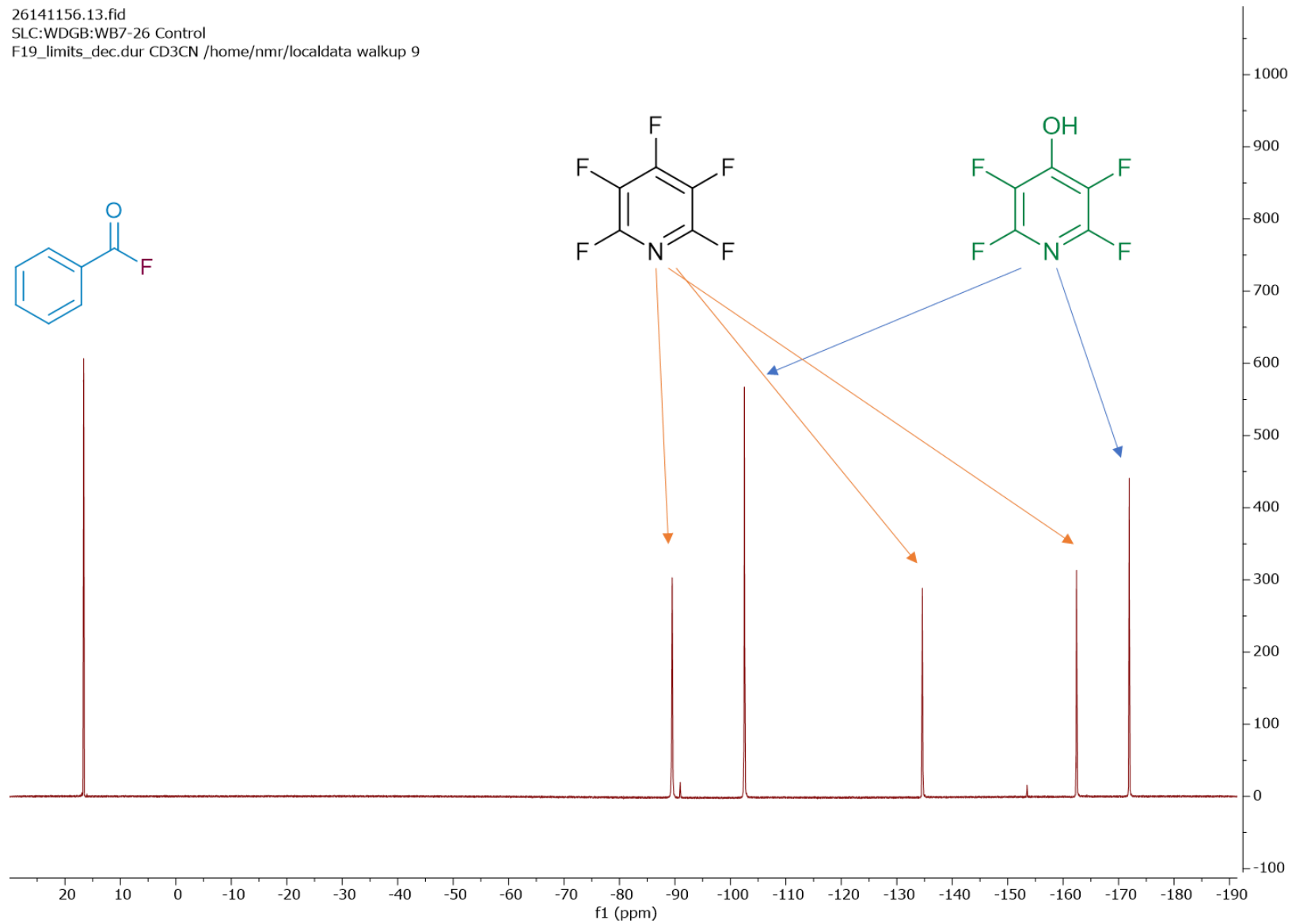
After activation period  
– no amine

After activation period –  
spiked with benzoyl fluoride

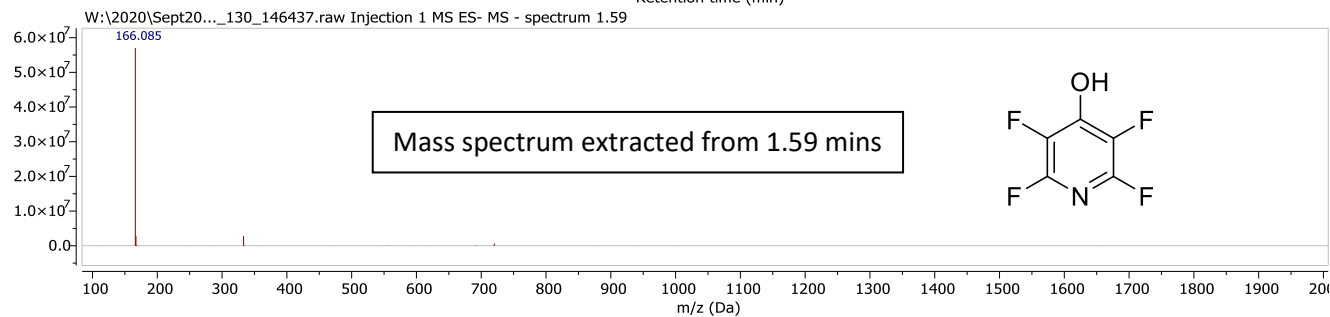
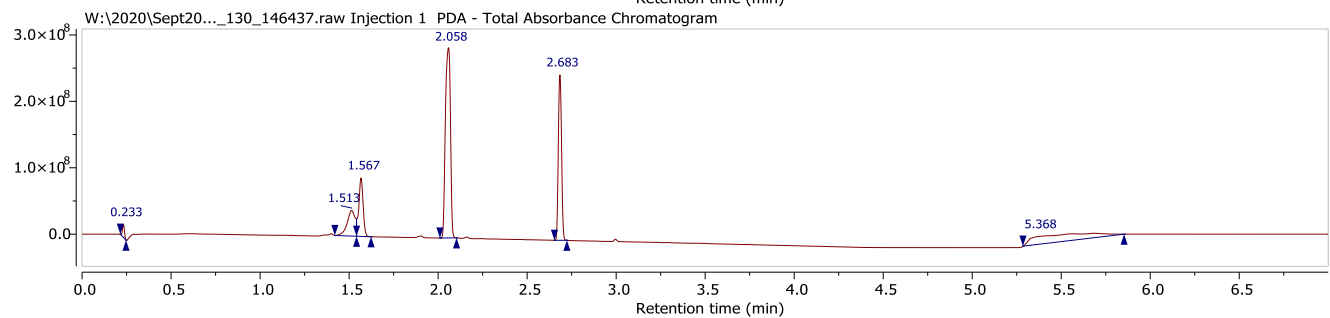
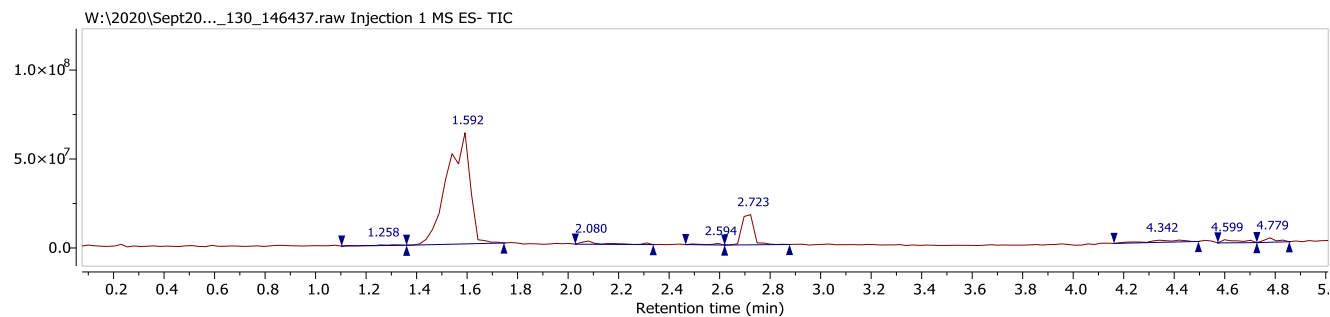
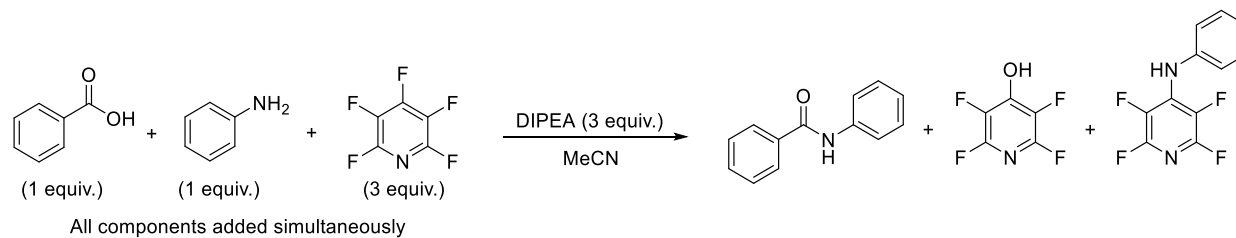
After activation period –  
spiked with  
tetrafluorohydroxypyridine

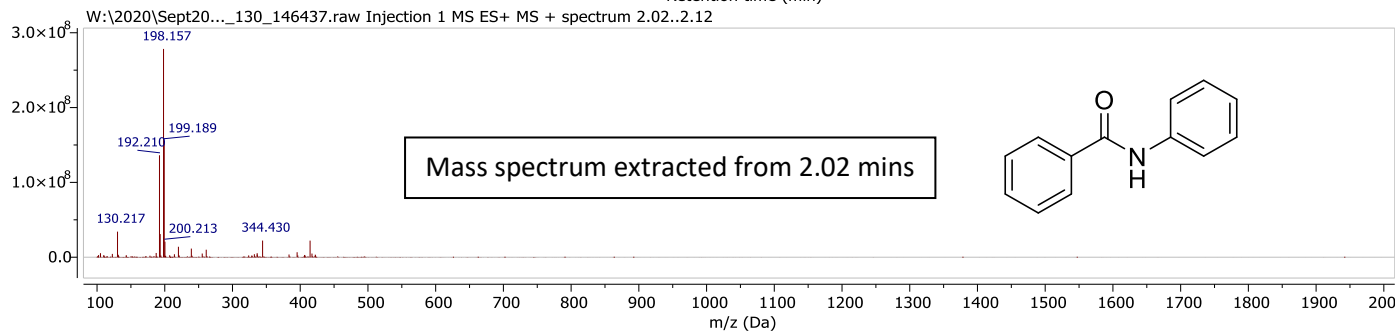
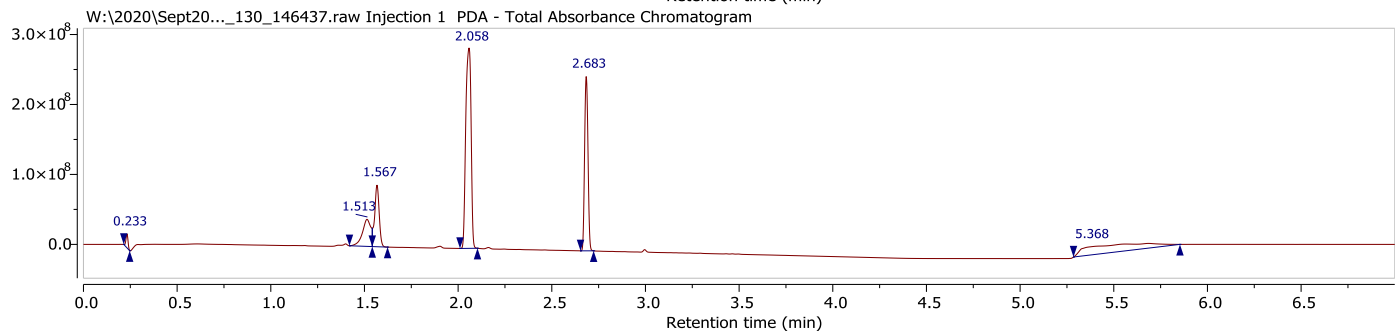
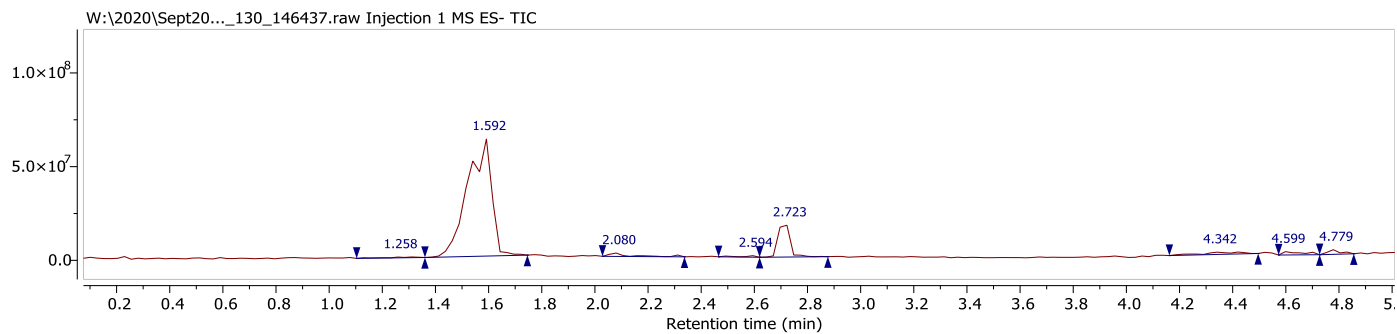
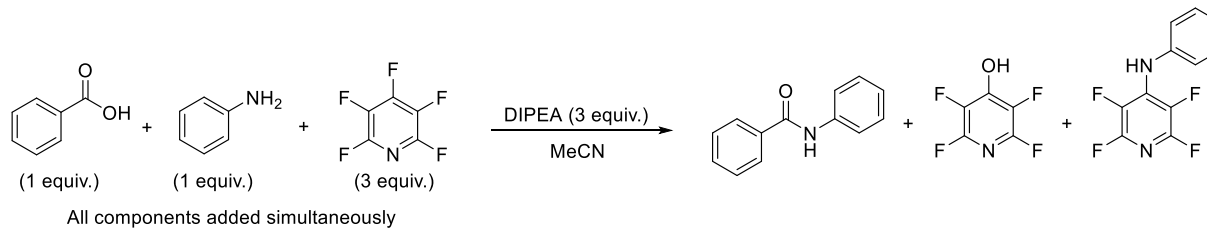
After activation period –  
spiked with  
pentafluoropyridine

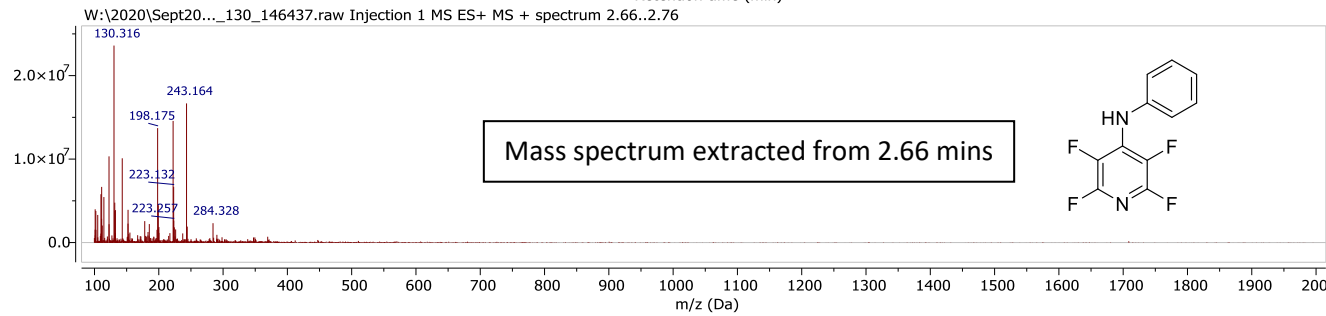
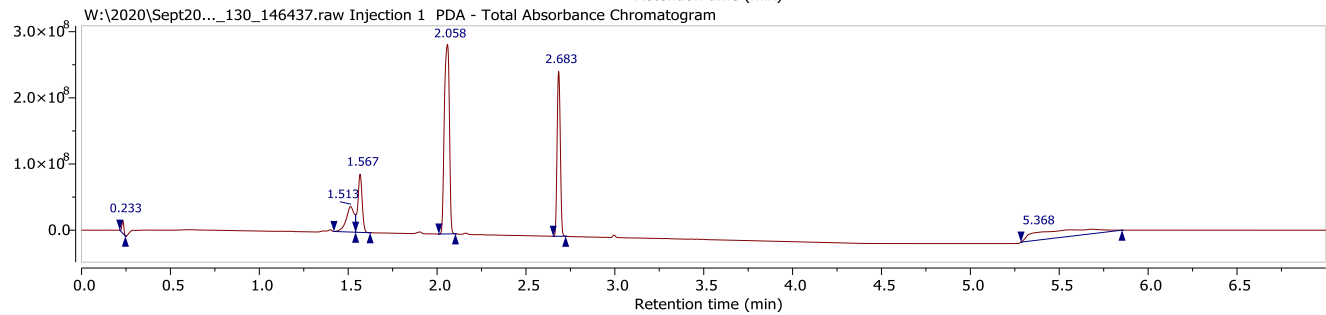
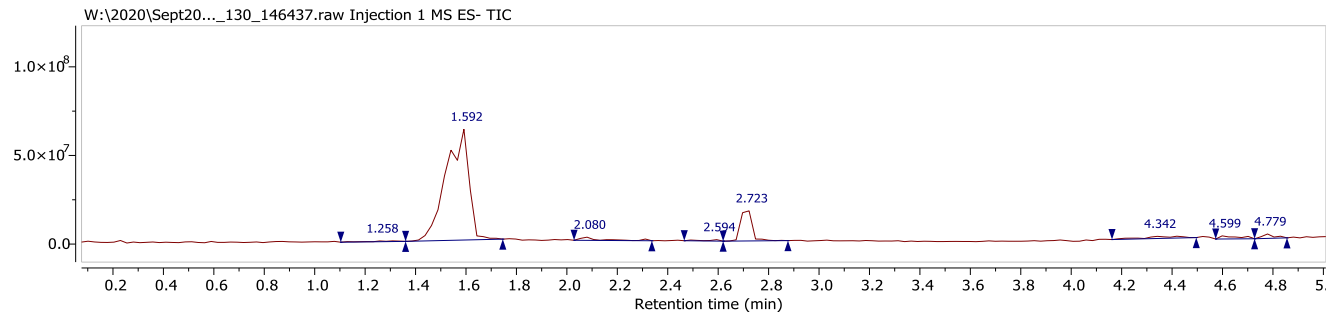
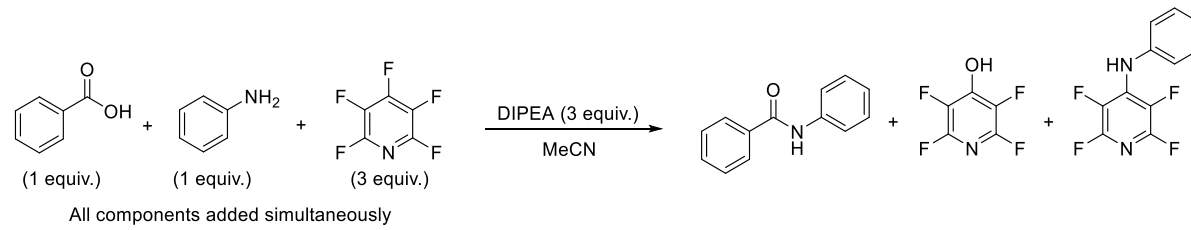
26141156.13.fid  
SLC:WDGB:WB7-26 Control  
F19\_limits\_dec.dur CD3CN /home/nmr/localdata walkup 9



# LCMS of Crude Reaction Mixture

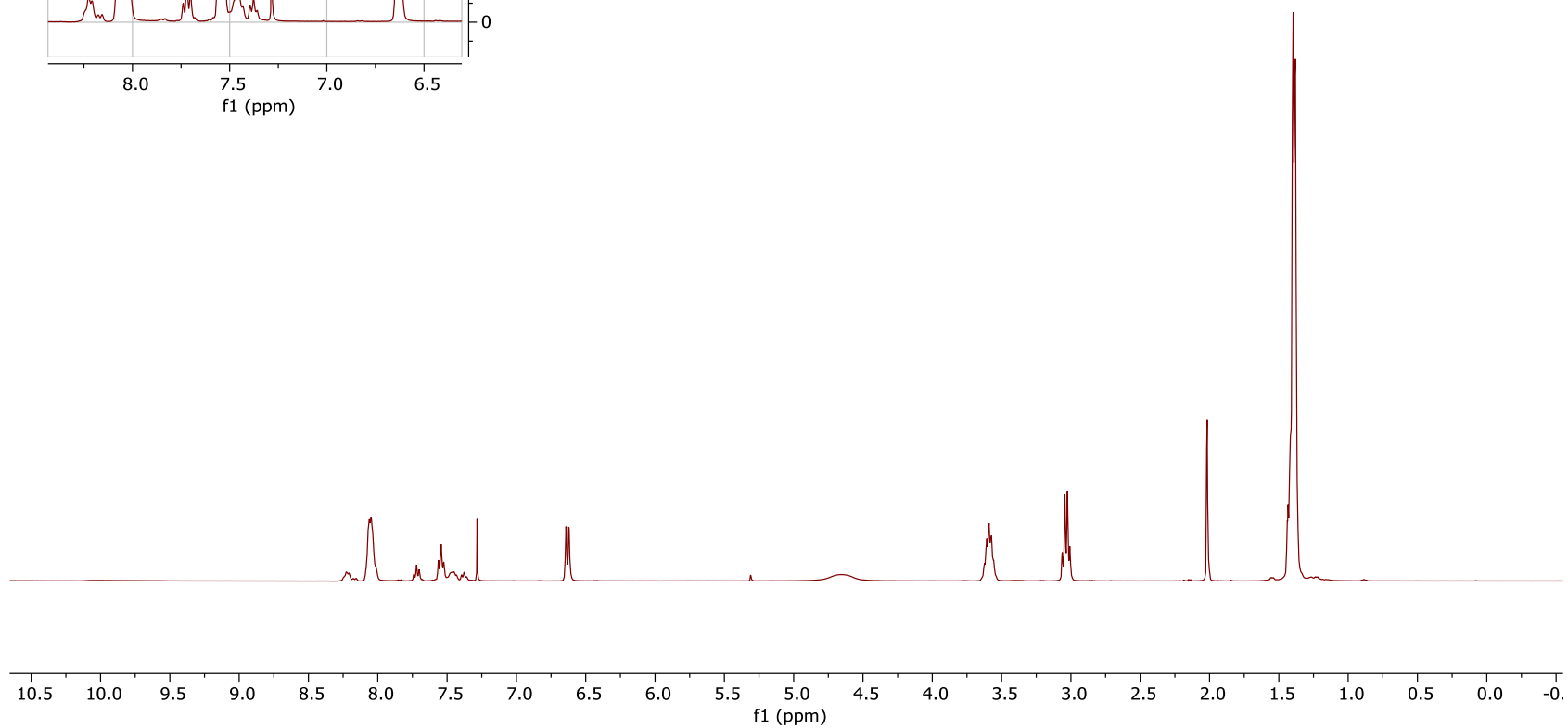
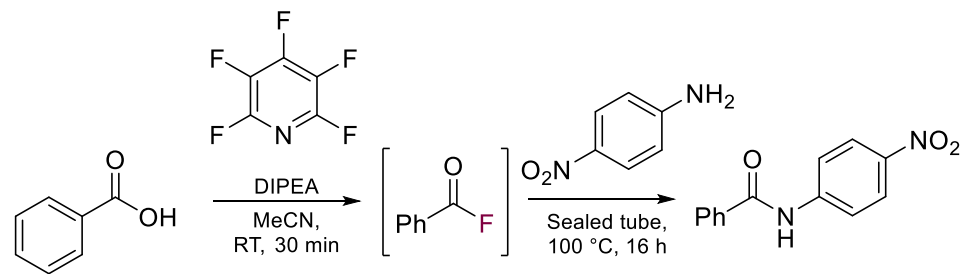
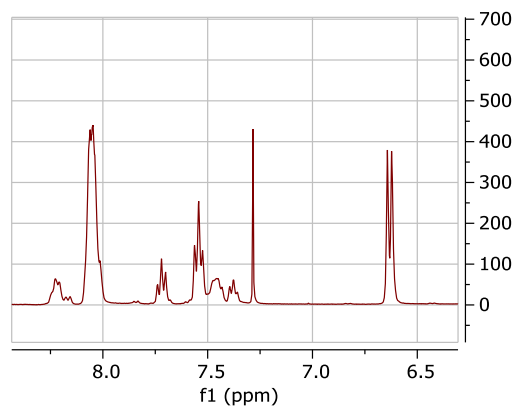






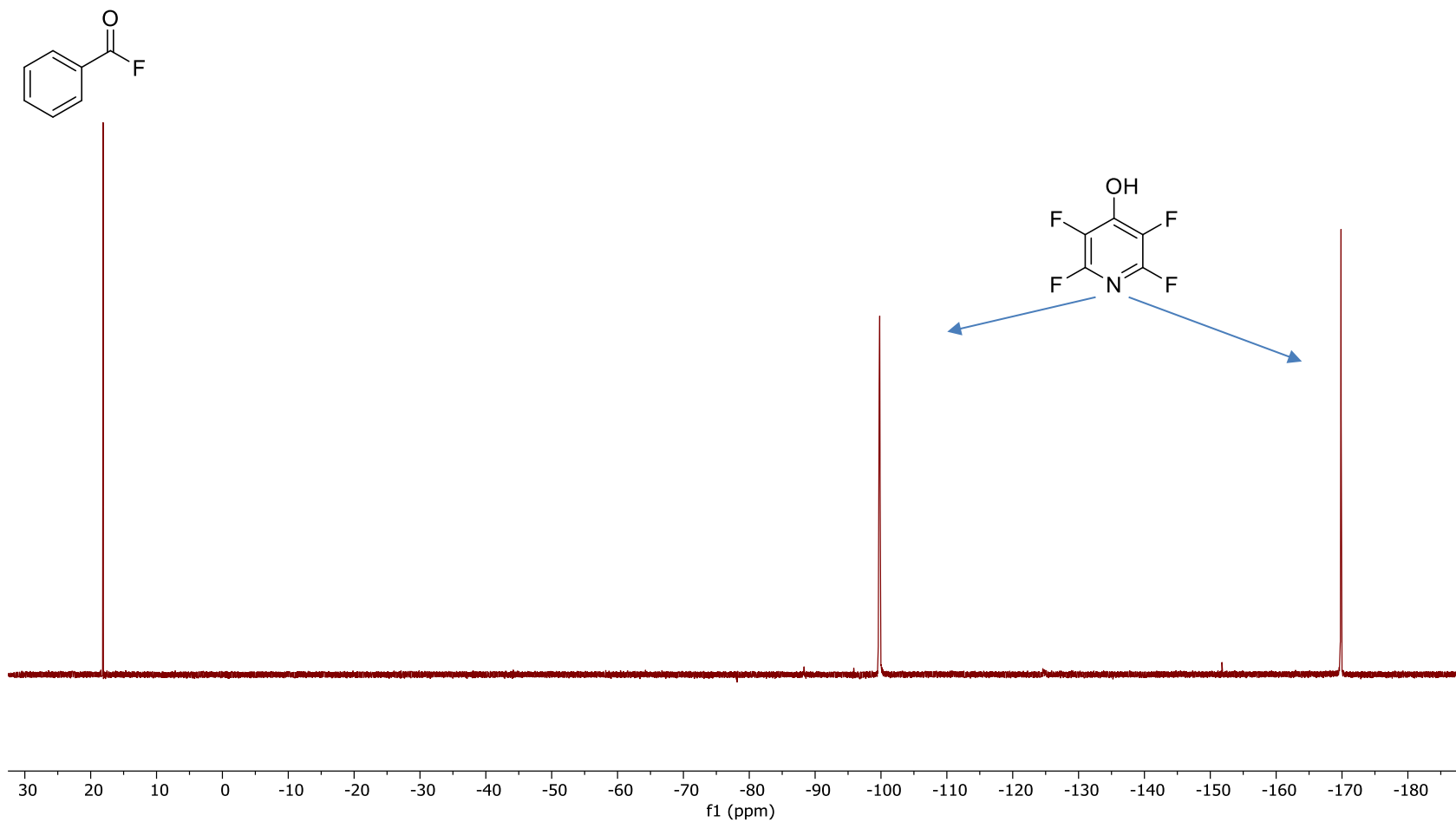
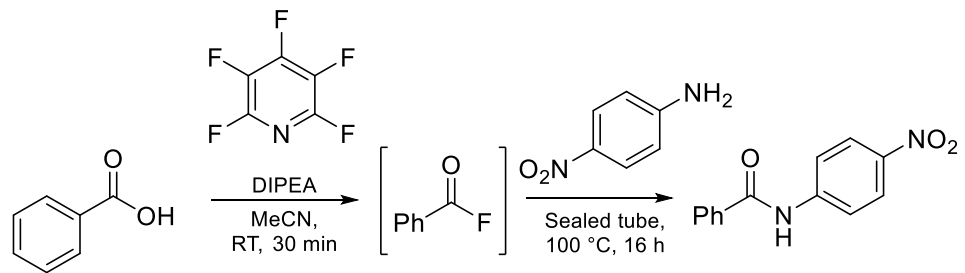
# 4-Nitroaniline Sealed Tube Reaction Crude NMRs

13110345.10.fid  
SLC:WDB:WB7-11  
Proton.dur CDCI3 /home/nmr/localdata walkup 7





13110345.13.fid  
SLC:WDB:WB7-11  
F19\_limits\_dec.dur CDCl3 /home/nmr/localdata walkup 7



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