The synthesis of non-racemic β-alkyl-β-aryl-disubstituted allyl alcohols and their transformation into allylamines and amino acids bearing a quaternary stereocenter

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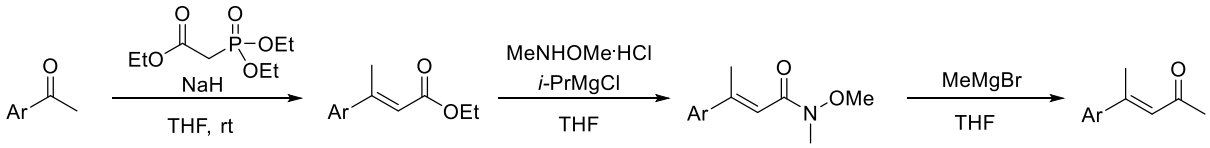
SUPPLEMENTARY MATERIALS

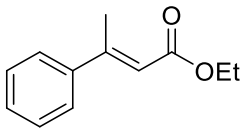
The synthesis of enones 1. Step 1 - olefination:[1]



To a suspension of sodium hydride (60% dispersion in oil, 1.3 equiv) in dry THF (c 0.2-0.3 M), triethyl phosphonoacetate (1.3 equiv) was added dropwise at room temperature under argon atmosphere. The mixture was stirred for 0.5 h and then cooled to 0 °C. Ketone (1 equiv) was added dropwise and the resulting mixture

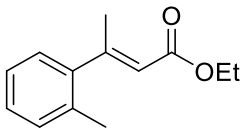
was warmed slowly to room temperature. The progress of the reaction was followed by TLC. When the reaction was completed, a saturated aq. $NaHCO_3$ solution was added to the mixture. The aqueous phase was extracted with AcOEt and the combined organic phase was washed with brine, and dried over Na_2SO_4. After the removal of solvents, the residue was purified by column chromatography on silica gel. *E*-Isomer was a dominant. The minor *Z*-isomer was removed by column chromatography of the corresponding Weinreb amides.

Ethyl (*E*)-3-phenylbut-2-enoate:



The reaction mixture was stirred at room temperature for 24 h. Column chromatography: silica gel, 2-5% AcOEt in hexanes. Yield: 3.06 g (overall 80%; *E/Z* ratio 33:1, ^{1}H NMR) starting from 5.83 g of acetophenone; yellowish oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51–7.44 (m, 2H), 7.40 – 7.33 (m, 3H), 6.14 (q, *J* = 1.4 Hz, 1H), 4.22 (q, *J* = 7.1 Hz, 2H), 2.59 (d, *J* = 1.4 Hz, 3H), 1.32 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.8, 155.4, 142.3, 128.9, 128.5, 126.3, 117.2, 59.8, 17.9, 14.3.

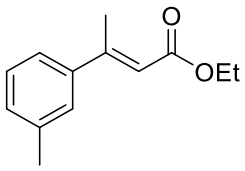
Ethyl (*E*)-3-(*o*-tolyl)but-2-enoate:



The reaction mixture was stirred at room temperature for 24 h. Column chromatography: silica gel, 3% AcOEt in hexanes. Yield: 3.71 g (overall 91%; *E/Z*

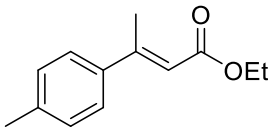
ratio 3.7:1, ^{1}H NMR) starting from 2.68 g of methyl *o*-tolyl ketone; yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23–7.14 (m, 3H), 7.10 – 7.04 (m, 1H), 5.77 (q, *J* = 1.5 Hz, 1H), 4.22 (q, *J* = 7.1 Hz, 2H), 2.46 (d, *J* = 1.5 Hz, 3H), 2.30 (s, 4H), 1.32 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.6, 158.2, 143.9, 133.8, 130.4, 127.6, 127.1, 125.7, 119.4, 59.8, 20.8, 19.7, 14.3.

Ethyl (*E*)-3-(m-tolyl)but-2-enoate:



The reaction mixture was stirred at room temperature for 24 h. Column chromatography: silica gel, 3% AcOEt in hexanes. Yield: 3.06 g (overall 75%; *E/Z* ratio 10:1, ^{1}H NMR) starting from 2.68 g of methyl *m*-tolyl ketone; yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31–7.23 (m, 3H), 7.19 – 7.15 (m, 1H), 6.14 (q, *J* = 1.0 Hz, 1H), 4.23 (q, *J* = 7.1 Hz, 2H), 2.58 (d, *J* = 1.0 Hz, 3H), 2.38 (s, 3H), 1.33 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.8, 155.7, 142.3, 138.0, 129.7, 128.4, 127.0, 123.4, 117.0, 59.7, 21.4, 17.9, 14.3.

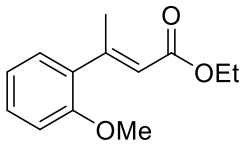
Ethyl (*E*)-3-(*p*-tolyl)but-2-enoate:



The reaction mixture was stirred at room temperature for 48 h. Column chromatography: silica gel, 3% AcOEt in hexanes. Yield: 4.06 g (overall 99%; *E/Z* ratio 6.7:1, NMR) starting from 2.68 g methyl *p*-tolyl ketone; yellow liquid; ^{1}H NMR

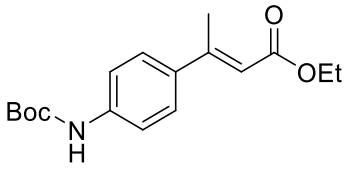
(400 MHz, $CDCl_3$) δ 7.42–7.36 (m, 2H), 7.21–7.14 (m, 2H), 6.15 (q, *J* = 1.2 Hz, 1H), 4.23 (q, *J* = 7.1 Hz, 2H), 2.59 (d, *J* = 1.2 Hz, 3H), 2.37 (s, 3H), 1.33 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.9, 155.3, 139.3, 139.0, 129.2, 126.2, 116.3, 59.7, 21.1, 17.7, 14.4.

Ethyl (*E*)-3-(2-methoxyphenyl)but-2-enoate:



The reaction mixture was stirred at room temperature for 24 h. Column chromatography: silica gel, 3% AcOEt in hexanes. Yield: 4.12 g (overall 94%; *E/Z* ratio 25:1, ^1H NMR) starting from 3.00 g of *o*-methoxyacetophenone; yellowish oil; ^1H NMR (400 MHz, $CDCl_3$) δ 7.32–7.24 (m, 1H), 7.14 (d, *J* = 7.5 Hz, 1H), 6.95 – 6.92 (m, 1H), 6.91 – 6.87 (m, 1H), 5.90 (q, *J* = 1.4 Hz, 1H), 4.20 (q, *J* = 7.1 Hz, 2H), 3.82 (s, 3H), 2.50 (d, *J* = 1.4 Hz, 3H), 1.30 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.7, 156.5, 156.3, 133.1, 129.4, 128.8, 120.5, 119.3, 111.0, 59.7, 55.4, 19.8, 14.3.

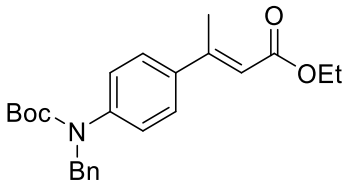
Ethyl (*E*)-3-(4-((tert-butoxycarbonyl)amino)phenyl)but-2-enoate:



The reaction mixture was stirred for 4 days at rt, and additionally for 3 days at 50 °C. Column chromatography: silica gel, 5% AcOEt in hexanes. Yield: 1.02 g (overall 96%; *E/Z* ratio 2.9:1, ^1H NMR) starting from 826 mg of *t*-butyl (4-acetylphenyl)carbamate; yellowish oil; ^1H NMR (400 MHz, $CDCl_3$) δ 7.47–7.30 (m,

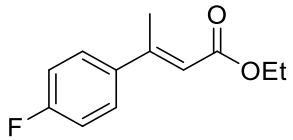
4H), 6.69 (br s, 1H), 6.14–6.07 (m, 1H), 4.20 (q, *J* = 7.1 Hz, 2H), 2.54 (t, *J* = 1.0 Hz, 3H), 1.51 (s, 9H), 1.30 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.0, 154.7, 152.5, 139.3, 136.4, 127.0, 118.1, 115.8, 80.8, 59.7, 28.3, 17.6, 14.3.

Ethyl (*E*)-3-(4-(benzyl(*tert*-butoxycarbonyl)amino)phenyl)but-2-enoate:



The reaction mixture was stirred at room temperature for 48 h. Column chromatography: silica gel, 5-10% AcOEt in hexanes. Yield: 310 mg (overall 76%; *E/Z* ratio 4:1, ^{1}H NMR) starting from 335 mg of *t*-butyl (4-acetylphenyl)(benzyl)carbamate; yellowish oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43–7.35 (m, 2H), 7.34–7.27 (m, 2H), 7.27–7.21 (m, 3H), 7.21–7.15 (m, 2H), 6.10 (q, *J* = 1.3 Hz, 1H), 4.85 (s, 2H), 4.20 (q, *J* = 7.1 Hz, 2H), 2.53 (d, *J* = 1.3 Hz, 3H), 1.43 (s, 9H), 1.30 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.8, 154.6, 154.5, 143.6, 139.2, 138.5, 128.4, 127.2, 127.1, 126.6, 126.0, 116.8, 80.9, 59.8, 53.7, 28.2, 17.7, 14.3.

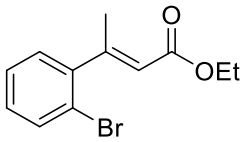
Ethyl (*E*)-3-(4-fluorophenyl)but-2-enoate:



The reaction mixture was stirred at room temperature for 24 h. Column chromatography: silica gel, 3% AcOEt in hexanes. Yield: 3.40 g (overall 82%; *E/Z* ratio 11:1, ^{1}H NMR) starting from 2.76 g of *p*-fluoroacetophenone; yellowish oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47–7.39 (m, 2H), 7.07–6.99 (m, 2H), 6.07 (q, *J* = 1.4 Hz,

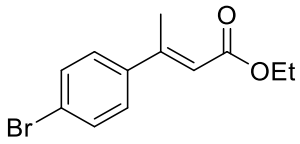
1H), 4.20 (q, *J* = 7.1 Hz, 2H), 2.54 (d, *J* = 1.4 Hz, 3H), 1.30 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.6, 163.2 (d, *J* = 248.9 Hz), 154.1, 138.2 (d, $J_{C\text{-}F}$ = 3.0 Hz), 128.1 (d, $J_{C\text{-}F}$ = 8.2 Hz), 117.1 (d, $J_{C\text{-}F}$ = 1.0 Hz), 115.4 (d, $J_{C\text{-}F}$ = 21.6 Hz), 59.8, 17.8, 14.3; ^{19}F NMR (376 MHz, $CDCl_3$) δ −112.6.

Ethyl (*E*)-3-(2-bromophenyl)but-2-enoate:



The reaction mixture was stirred at room temperature for 24 h. Column chromatography: silica gel, 3-10% AcOEt in hexanes. Yield: 4.84 g (overall 90%, *E/Z* ratio 2.3:1, ^{1}H NMR) starting from 3.98 g of *o*-bromoacetophenone; yellowish oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57–7.53 (m, 1H), 7.30–7.24 (m, 1H), 7.17–7.11 (m, 2H), 5.80 (q, *J* = 1.5 Hz, 1H), 4.22 (q, *J* = 7.1 Hz, 2H), 2.48 (d, *J* = 1.5 Hz, 3H), 1.30 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.3, 156.9, 144.7, 133.0, 129.1, 128.8, 127.3, 120.7, 120.5, 59.9, 20.3, 14.3.

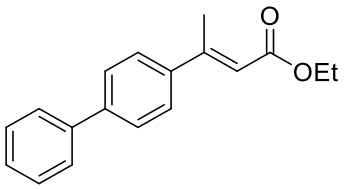
Ethyl (*E*)-3-(4-bromophenyl)but-2-enoate:



The reaction mixture was stirred at room temperature for 24 h. Column chromatography: silica gel, 3-10% AcOEt in hexanes. Yield: 4.07 g (overall 76%; *E/Z* ratio 6.3:1, ^{1}H NMR) starting from 3.98 g *p*-bromoacetophenone; colourless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51–7.45 (m, 2H), 7.36–7.29 (m, 2H), 6.12–6.07 (m, 1H),

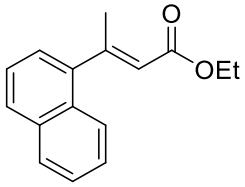
4.21 (q, *J* = 7.2 Hz, 2H), 2.53 (s, 3H), 1.30 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 154.0, 141.0, 131.6, 127.9, 123.2, 117.6, 59.9, 17.7, 14.3.

Ethyl (*E*)-3-([1,1'-biphenyl]-4-yl)but-2-enoate:



The reaction mixture was stirred at room temperature for 48 h. Column chromatography: silica gel, 3% AcOEt in hexanes. Yield: 4.53 g (overall 85%, *E/Z* ratio 3.5:1, ^{1}H NMR) starting from 3.93 g of *p*-phenylacetophenone; waxy solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66–7.54 (m, 6H), 7.51–7.43 (m, 2H), 7.41–7.34 (m, 1H), 6.23 (q, *J* = 1.3 Hz, 1H), 4.25 (q, *J* = 7.1 Hz, 2H), 2.64 (d, *J* = 1.3 Hz, 3H), 1.35 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.9, 154.8, 141.8, 141.0, 140.3, 128.9, 127.6, 127.1, 127.0, 126.8, 117.0, 59.9, 17.8, 14.4.

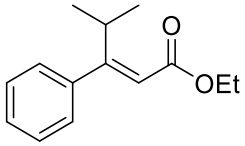
Ethyl (*E*)-3-(naphthalen-1-yl)but-2-enoate:



The reaction mixture was stirred at room temperature for 24 h. Column chromatography: silica gel, 2-7% AcOEt in hexanes. Yield: 4.26 g (overall 89%; *E/Z* ratio 4.5:1, ^{1}H NMR) starting from 3.40 g of 1-acetonaphtone; yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96–7.85 (m, 2H), 7.81 (d, *J* = 8.3 Hz, 1H), 7.54–7.47 (m, 2H), 7.47–7.41 (m, 1H), 7.30 (d, *J* = 7.1 Hz, 1H), 6.02 (q, *J* = 1.4 Hz, 1H), 4.29 (q, *J* = 7.1 Hz,

2H), 2.65 (d, *J* = 1.4 Hz, 3H), 1.36 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.6, 157.0, 142.1, 133.7, 130.1, 128.5, 128.1, 126.3, 126.0, 125.3, 125.2, 124.2, 120.6, 59.9, 21.7, 14.4.

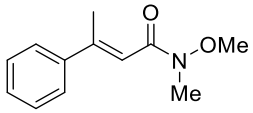
Ethyl (*E*)-4-methyl-3-phenylpent-2-enoate:



To a 1M solution of LiHMDS in THF (6.5 mL, 6.5 mmol), triethyl phosphonoacetate (6.5 mmol) was added dropwise at room temperature under argon atmosphere. The mixture was stirred for 0.5 h and then cooled to 0 °C, and phenyl *i*-propyl ketone (5 mmol) was added dropwise. The resulting mixture was warmed slowly to room temperature. After stirring for 24 h, an additional portion of HWE reagent (6.5 mmol) was added. The progress of the reaction was followed by TLC. The starting material was consumed after 4 days. Next, sat. NH_4Cl solution was added and the aqueous phase was extracted with AcOEt. The combined organic layers were washed with brine, and dried over Na_2SO_4. After the removal of the solvent, the crude residue was purified by column chromatography on silica gel (2% AcOEt in hexanes) to obtain 1.03 g of product as an inseparable mixture of *E/Z* isomers in 1.5:1 ratio (overall yield 94%). Colourless oil; ^{1}H NMR (400 MHz, $CDCl_3$, selected signals for major *E*- isomer) δ 7.34–7.29 (m, 3H), 7.22–7.16 (m, 2H), 5.70 (s, 1H), 4.20 (q, *J* = 7.1 Hz, 2H), 4.11 (sept, *J* = 7.0 Hz, 1H), 1.30 (t, *J* = 7.1 Hz, 3H), 1.09 (d, *J* = 7.0 Hz, 6H);

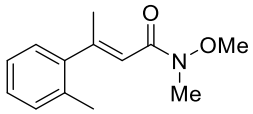
Step 2 - synthesis of Weinreb Amides:[1] Suspension of α,β-unsaturated ester (1 equiv) and *N*,*O*-dimethylhydroxylamine hydrochloride (2 equiv) in dry THF (c 0.2-0.3 M) was cooled to −5 °C under argon atmosphere, and 2M soln. of *i*-PrMgCl in THF (4 equiv) was added dropwise. The progress of the reaction was followed by TLC. After stirring at −5 °C for 0.5-1 h, the reaction was quenched by the addition of sat. NH_4Cl. The aqueous phase was extracted with ethyl acetate and the combined organic phase was washed with brine, and dried over sodium sulfate. After the removal of the solvent, the crude residue was purified by column chromatography on silica gel to provide pure *E* isomer.

(*E*)-*N*-Methoxy-*N*-methyl-3-phenylbut-2-enamide:



Column chromatography: silica gel, 10-20% AcOEt in hexanes. Yield: 799 mg (86%) starting from 866 mg of ethyl (*E*)-3-phenylbut-2-enoate; yellowish oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49–7.44 (m, 2H), 7.39–7.30 (m, 3H), 6.56 (s, 1H), 3.68 (s, 3H), 3.25 (s, 3H), 2.52 (d, *J* = 1.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.9, 152.2, 143.0, 128.5, 128.4, 126.3, 116.0, 61.5, 32.4, 18.0.

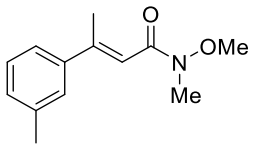
(*E*)-*N*-Methoxy-*N*-methyl-3-(*o*-tolyl)but-2-enamide:



Column chromatography: silica gel, 10-20% AcOEt in hexanes. Yield: 2.60 g (85%) starting from 2.85 g of ethyl (*E*)-3-(*o*-tolyl)but-2-enoate; yellowish oil; ^{1}H NMR (400

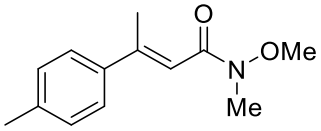
MHz, $CDCl_3$) δ 7.24–7.14 (m, 3H), 7.14–7.06 (m, 1H), 6.21 (s, 1H), 3.66 (s, 3H), 3.25 (s, 3H), 2.42 (d, *J* = 1.6 Hz, 3H), 2.31 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.7, 154.9, 144.6, 134.0, 130.3, 127.4, 127.3, 125.7, 117.8, 61.5, 32.2, 20.6, 19.7.

(*E*)-*N*-Methoxy-*N*-methyl-3-(*m*-tolyl)but-2-enamide:



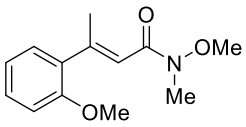
Column chromatography: silica gel, 10-20% AcOEt in hexanes. Yield: 2.97 g (89%) starting from 2.80 g of ethyl (*E*)-3-(*m*-tolyl)but-2-enoate; yellowish oil; ^1H NMR (400 MHz, $CDCl_3$) δ 7.31–7.22 (m, 3H), 7.18–7.11 (m, 1H), 6.55 (s, 1H), 3.70 (s, 3H), 3.26 (s, 3H), 2.52 (d, *J* = 1.4 Hz, 3H), 2.37 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.0, 152.4, 143.0, 138.0, 129.3, 128.3, 127.0, 123.4, 115.8, 61.5, 32.4, 21.5, 18.1.

(*E*)-*N*-Methoxy-*N*-methyl-3-(*p*-tolyl)but-2-enamide:



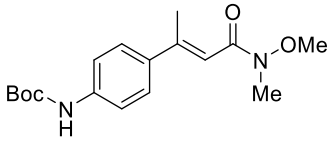
Column chromatography: silica gel, 10-20% AcOEt in hexanes. Yield: 2.93 g (82%) starting from 3.33 g of ethyl (*E*)-3-(*p*-tolyl)but-2-enoate; yellowish oil; ^1H NMR (400 MHz, $CDCl_3$) δ 7.40–7.33 (m, 2H), 7.14 (d, *J* = 8.0 Hz, 2H), 6.56 (s, 1H), 3.66 (s, 3H), 3.23 (s, 3H), 2.51 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.0, 152.1, 140.0, 138.5, 129.1, 126.1, 115.2, 61.5, 32.3, 21.1, 17.8.

(*E*)-*N*-Methoxy-3-(2-methoxyphenyl)-*N*-methylbut-2-enamide:



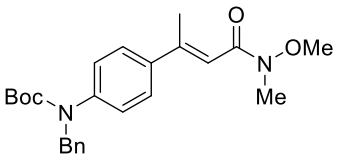
Column chromatography: silica gel, 30% AcOEt in hexanes. Yield: 3.65 g (95%) starting from 3.59 g of ethyl (*E*)-3-(2-methoxyphenyl)but-2-enoate; yellowish oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33–7.20 (m, 1H), 7.15 (d, *J* = 7.5 Hz, 1H), 6.99–6.80 (m, 2H), 6.32 (s, 1H), 3.79 (s, 3H), 3.66 (s, 3H), 3.23 (s, 3H), 2.44 (d, *J* = 1.5 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.9, 156.5, 152.8, 133.7, 129.1, 128.9, 120.5, 117.9, 111.1, 61.4, 55.4, 32.3, 19.7.

***tert*-Butyl (*E*)-(4-(4-(methoxy(methyl)amino)-4-oxobut-2-en-2-yl)phenyl)carbamate:**



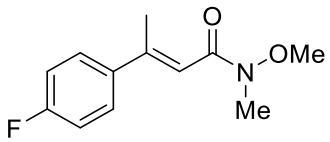
Column chromatography: silica gel, 40% AcOEt in hexanes. Yield: 335 g (86%) starting from 371 mg of ethyl (*E*)-3-(4-((*tert*-butoxycarbonyl)amino)phenyl)but-2-enoate; yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44–7.32 (m, 4H), 7.03 (br s, 1H), 6.52 (s, 1H), 3.66 (s, 3H), 3.22 (s, 3H), 2.47 (d, *J* = 1.4 Hz, 3H), 1.48 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.1, 152.7, 151.6, 139.2, 137.0, 126.9, 118.3, 114.6, 80.5, 61.5, 32.4, 28.3, 17.7.

***tert*-Butyl (*E*)-benzyl(4-(4-(methoxy(methyl)amino)-4-oxobut-2-en-2-yl)phenyl)carbamate:**



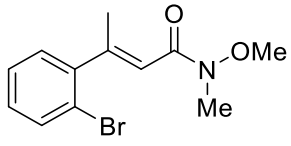
Column chromatography: silica gel, 20-40% AcOEt in hexanes. Yield: 86 mg (70%) starting from 120 mg of ethyl (*E*)-3-(4-(benzyl(*tert*-butoxycarbonyl)-amino)phenyl)but-2-enoate; yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43–7.34 (m, 2H), 7.34–7.14 (m, 7H), 6.53 (s, 1H), 4.85 (s, 2H), 3.68 (s, 3H), 3.24 (s, 3H), 2.48 (d, *J* = 1.3 Hz, 3H), 1.43 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.9, 154.6, 151.4, 143.2, 140.0, 138.6, 128.4, 127.1, 127.1, 126.5, 125.9, 115.7, 80.8, 61.5, 53.8, 32.4, 28.3, 17.8.

(*E*)-3-(4-Fluorophenyl)-*N*-methoxy-*N*-methylbut-2-enamide:



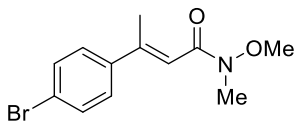
Column chromatography: silica gel, 30% AcOEt in hexanes. Yield: 1.35 g (81%) starting from 1.56 g of (*E*)-3-(4-fluorophenyl)but-2-enoate; yellowish oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46–7.39 (m, 2H), 7.07–6.99 (m, 2H), 6.51 (s, 1H), 3.69 (s, 3H), 3.24 (s, 3H), 2.48 (d, *J* = 1.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.8, 162.9 (d, $J_{C\text{-}F}$ = 248.1 Hz), 151.0, 138.9 (d, $J_{C\text{-}F}$ = 2.6 Hz), 128.0 (d, $J_{C\text{-}F}$ = 8.1 Hz), 115.9, 115.3 (d, $J_{C\text{-}F}$ = 21.5 Hz), 61.5, 32.3, 18.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ −113.29.

(*E*)-3-(2-Bromophenyl)-*N*-methoxy-*N*-methylbut-2-enamide:



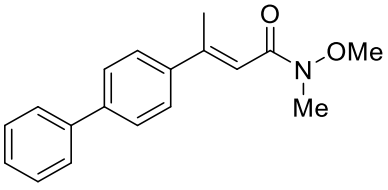
Column chromatography: silica gel, 30% AcOEt in hexanes. Yield: 1.22 g (98%) starting from 1.19 g of (*E*)-3-(2-bromophenyl)but-2-enoate; yellowish oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58–7.54 (m, 1H), 7.31–7.25 (m, 1H), 7.20–7.11 (m, 2H), 6.23 (s, 1H), 3.71–3.65 (m, 3H), 3.28–3.22 (m, 3H), 2.42 (d, *J* = 1.6 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.2, 153.4, 145.2, 132.9, 129.0, 128.9, 127.4, 120.9, 119.1, 61.8, 32.2, 20.1.

(*E*)-3-(4-Bromophenyl)-*N*-methoxy-*N*-methylbut-2-enamide:



Column chromatography: silica gel, 15% AcOEt in hexanes. Yield: 2.81 g (76%) starting from 3.50 g of (*E*)-3-(4-bromophenyl)but-2-enoate; yellowish solid; m.p. 38-40 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50–7.45 (m, 2H), 7.35–7.30 (m, 2H), 6.53 (s, 1H), 3.69 (s, 3H), 3.25 (s, 3H), 2.48 (d, *J* = 1.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.6, 150.8, 141.8, 131.6, 127.9, 122.7, 116.5, 61.6, 32.3, 17.8.

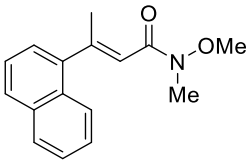
(*E*)-3-([1,1'-Biphenyl]-4-yl)-*N*-methoxy-*N*-methylbut-2-enamide:



Column chromatography: silica gel, 15-25% AcOEt in hexanes. Yield: 3.14 g (77%) starting from 3.86 g of (*E*)-3-(4-phenylphenyl)but-2-enoate; white solid; m.p. 123−124 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64–7.55 (m, 6H), 7.48–7.43 (m, 2H), 7.39–7.34 (m, 1H), 6.65 (s, 1H), 3.73 (s, 3H), 3.29 (s, 3H), 2.58 (d, *J* = 1.3 Hz, 3H); ^{13}C NMR

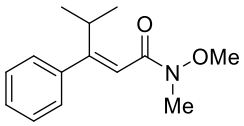
(101 MHz, $CDCl_3$) δ 168.0, 151.7, 141.8, 141.5, 140.4, 128.8, 127.5, 127.1, 127.0, 126.7, 115.9, 61.6, 32.4, 17.9.

(*E*)-*N*-Methoxy-*N*-methyl-3-(naphthalen-1-yl)but-2-enamide:



Column chromatography: silica gel, 20% AcOEt in hexanes. Yield: 719 mg (85%) starting from 800 mg of (*E*)-3-(1-naphtyl)but-2-enoate; yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99–7.94 (m, 1H), 7.90–7.84 (m, 1H), 7.81 (d, *J* = 8.3 Hz, 1H), 7.52–7.43 (m, 3H), 7.34–7.30 (m, 1H), 6.42 (s, 1H), 3.67 (s, 3H), 3.30 (s, 3H), 2.61 (d, *J* = 1.5 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.7, 153.7, 142.8, 133.8, 130.4, 128.5, 127.9, 126.2, 125.9, 125.4, 125.3, 124.3, 119.2, 61.6, 32.3, 21.5.

(*E*)-*N*-Methoxy-*N*,4-dimethyl-3-phenylpent-2-enamide:

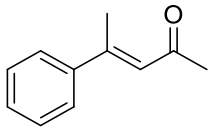


Column chromatography: silica gel, 15% AcOEt in hexanes. Yield: 941 mg (77%) starting from 725 mg of ethyl (*E*)-4-methyl-3-phenylpent-2-enoate; yellowish oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35–7.24 (m, 3H), 7.24–7.15 (m, 2H), 6.08 (s, 1H), 4.02–3.81 (m, 1H), 3.64 (s, 3H), 3.22 (s, 3H), 1.07 (d, *J* = 7.1 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.5, 162.8, 141.4, 127.9, 127.7, 127.3, 117.6, 61.4, 32.3, 30.0, 21.5.

Step 3 - synthesis of enones 1:[1] A 3 M soln. of MeMgBr in THF (1.3 equiv) was slowly added to a solution of Weinreb amide (1 equiv) in THF (c 0.2-0.3 M) cooled

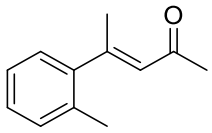
to −30 °C. After addition, the mixture was slowly warmed to −5°C and stirred for 0.5 h. The progress of the reaction was followed by TLC. Next, sat. NH_4Cl was added and the resulting mixture was extracted with AcOEt. The combined organic layers were washed with brine, and dried over Na_2SO_4. After the removal of the solvents, the residue was purified by column chromatography on silica gel.

(*E*)-4-Phenylpent-3-en-2-one:



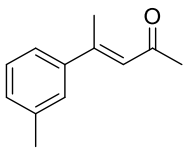
Column chromatography: silica gel, 5% AcOEt in hexanes. Yield: 595 mg (96%) starting from 793 mg of (*E*)-*N*-methoxy-*N*-methyl-3-phenylbut-2-enamide; yellowish oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50–7.45 (m, 2H), 7.40–7.34 (m, 3H), 6.50 (q, *J* = 1.3 Hz, 1H), 2.53 (d, *J* = 1.3 Hz, 3H), 2.28 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.8, 153.8, 142.5, 129.1, 128.5, 126.4, 124.5, 32.2, 18.3.

(*E*)-4-(*o*-Tolyl)pent-3-en-2-one:



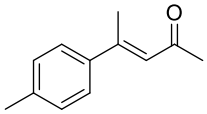
Column chromatography: silica gel, 5-10% AcOEt in hexanes. Yield: 1.45 g (96%) starting from 1.90 g of (*E*)-*N*-methoxy-*N*-methyl-3-(*o*-tolyl)but-2-enamide; yellowish oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23–7.14 (m, 3H), 7.09–7.04 (m, 1H), 6.15 (q, *J* = 1.5 Hz, 1H), 2.42 (d, *J* = 1.5 Hz, 3H), 2.29 (s, 3H), 2.25 (s, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.7, 156.5, 144.1, 133.9, 130.4, 127.7, 127.1, 126.6, 125.7, 32.0, 21.2, 19.7.

(*E*)-4-(*m*-Tolyl)pent-3-en-2-one:



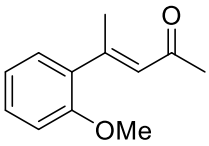
Column chromatography: silica gel, 5-10% AcOEt in hexanes. Yield: 2.10 g (89%) starting from 2.97 g of (*E*)-*N*-methoxy-*N*-methyl-3-(*m*-tolyl)but-2-enamide; yellowish oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30–7.23 (m, 3H), 7.20–7.15 (m, 1H), 6.52–6.47 (m, 1H), 2.53 (d, *J* = 1.4 Hz, 3H), 2.38 (s, 3H), 2.28 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.8, 154.0, 142.5, 138.1, 129.8, 128.4, 127.1, 124.3, 123.6, 32.2, 21.4, 18.3.

(*E*)-4-(*p*-Tolyl)pent-3-en-2-one:



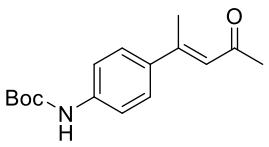
Column chromatography: silica gel, 5-10% AcOEt in hexanes. Yield: 2.15 g (92%) starting from 2.93 g of (*E*)-*N*-methoxy-*N*-methyl-3-(*p*-tolyl)but-2-enamide; yellowish oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40–7.36 (m, 2H), 7.19–7.14 (m, 2H), 6.50 (q, *J* = 1.4 Hz, 1H), 2.52 (d, *J* = 1.4 Hz, 3H), 2.35 (s, 3H), 2.26 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.7, 153.7, 139.5, 139.2, 129.2, 126.4, 123.7, 32.1, 21.2, 18.1.

(*E*)-4-(2-Methoxyphenyl)pent-3-en-2-one:



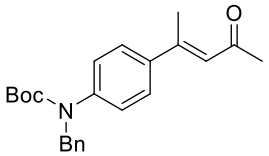
Column chromatography: silica gel, 5-10% AcOEt in hexanes. Yield: 2.92 g (96%) starting from 3.65 g of (*E*)-*N*-methoxy-*N*-methyl-3-(*o*-methoxyphenyl)but-2-enamide; yellowish oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31–7.25 (m, 1H), 7.14–7.11 (m, 1H), 6.95–6.92 (m, 1H), 6.92–6.87 (m, 1H), 6.27 (q, *J* = 1.5 Hz, 1H), 3.81 (s, 3H), 2.45 (d, *J* = 1.5 Hz, 3H), 2.24 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.8, 156.5, 154.9, 133.2, 129.5, 128.7, 126.6, 120.5, 111.1, 55.4, 32.0, 20.3.

***tert*-Butyl (*E*)-(4-(4-oxopent-2-en-2-yl)phenyl)carbamate:**



Column chromatography: silica gel, 25% AcOEt in hexanes. Yield: 263 mg (91%) starting from 331 mg of *tert*-butyl (*E*)-(4-(4-(methoxy(methyl)amino)-4-oxobut-2-en-2-yl)phenyl)carbamate; yellowish oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45–7.36 (m, 4H), 6.94 (br s, 1H), 6.48 (d, *J* = 1.4 Hz, 1H), 2.49 (d, *J* = 1.4 Hz, 3H), 2.26 (s, 3H), 1.50 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.9, 153.2, 152.6, 139.7, 136.4, 127.2, 123.1, 118.2, 80.7, 32.2, 28.3, 18.0.

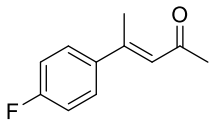
***tert*-Butyl (*E*)-benzyl(4-(4-oxopent-2-en-2-yl)phenyl)carbamate:**



Column chromatography: silica gel, 5-10% AcOEt in hexanes. Yield: 70 mg (91%) starting from 86 mg of *tert*-butyl (*E)*-benzyl(4-(4-(methoxy(methyl)amino)-4-oxobut-2-en-2-yl)phenyl)carbamate; yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45–

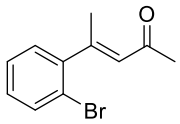
7.35 (m, 2H), 7.34–7.15 (m, 7H), 6.47 (s, 1H), 4.85 (s, 2H), 2.49 (d, *J* = 1.3 Hz, 3H), 2.26 (s, 3H), 1.43 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.7, 154.5, 152.9, 143.7, 139.4, 138.5, 128.4, 127.1, 127.1, 126.8, 126.0, 124.1, 80.9, 53.7, 32.2, 28.3, 18.1.

(*E*)-4-(4-Fluorophenyl)pent-3-en-2-one:



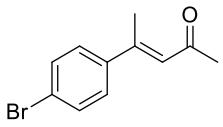
Column chromatography: silica gel, 5% AcOEt in hexanes. Yield: 961 mg (89%) starting from 1.35 g of (*E*)-*N*-methoxy-*N*-methyl-3-(*p*-fluorophenyl)but-2-enamide; yellowish oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48–7.42 (m, 2H), 7.08–7.01 (m, 2H), 6.45 (q, *J* = 1.3 Hz, 1H), 2.49 (d, *J* = 1.3 Hz, 3H), 2.27 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.6 , 163.3 (d, *J* = 249.1 Hz), 152.5 , 138.5 (d, *J* = 3.3 Hz), 128.3 (d, *J* = 8.4 Hz), 124.4 (d, *J* = 1.1 Hz), 115.5 (d, *J* = 21.6 Hz), 32.2 , 18.3; ^{19}F NMR (376 MHz, $CDCl_3$) δ −112.3.

(*E*)-4-(2-Bromophenyl)pent-3-en-2-one:



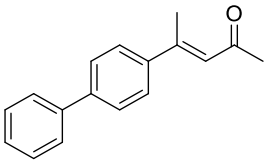
Column chromatography: silica gel, 2-5% AcOEt in hexanes. Yield: 852 mg (90%) starting from 1.13 g of (*E*)-*N*-methoxy-*N*-methyl-3-(*o*-bromophenyl)but-2-enamide; yellowish oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58–7.54 (m, 1H), 7.31–7.25 (m, 1H), 7.18–7.11 (m, 2H), 6.16 (q, *J* = 1.5 Hz, 1H), 2.42 (d, *J* = 1.5 Hz, 3H), 2.26 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.7, 154.9, 144.8, 133.0, 129.1, 128.8, 127.5, 127.3, 120.7, 32.0, 20.7.

(*E*)-4-(4-Bromophenyl)pent-3-en-2-one:



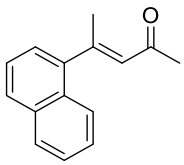
Column chromatography: silica gel, 5-20% AcOEt in hexanes. Yield: 2.21 g (93%) starting from 2.81 g of (*E*)-*N*-methoxy-*N*-methyl-3-(*p*-bromophenyl)but-2-enamide; yellowish solid; ; m.p. 54-56 °C [lit.[2] 49-51 °C]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51–7.46 (m, 2H), 7.35–7.31 (m, 2H), 6.47 (q, *J* = 1.3 Hz, 1H), 2.48 (d, *J* = 1.3 Hz, 3H), 2.27 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.6, 152.3, 141.3, 131.7, 128.0, 124.7, 123.3, 32.2, 18.1.

(*E*)-4-([1,1'-Biphenyl]-4-yl)pent-3-en-2-one:



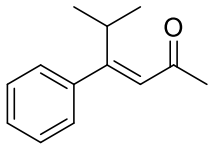
Column chromatography: silica gel, 30-50% AcOEt in hexanes. Yield: 2.10 g (97%) starting from 2.58 g of (*E*)-*N*-methoxy-*N*-methyl-3-(*p*-phenylphenyl)but-2-enamide; yellowish solid; m.p. 136-138 °C [Lit.[3] 120-121 °C]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65–7.56 (m, 6H), 7.49–7.43 (m, 2H), 7.41–7.36 (m, 1H), 6.59 (q, *J* = 1.4 Hz, 1H), 2.60 (d, *J* = 1.4 Hz, 3H), 2.32 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.7, 153.2, 142.0, 141.2, 140.2, 128.9, 127.7, 127.2, 127.0, 127.0, 124.3, 32.3, 18.2.

(*E*)-4-(Naphthalen-1-yl)pent-3-en-2-one:



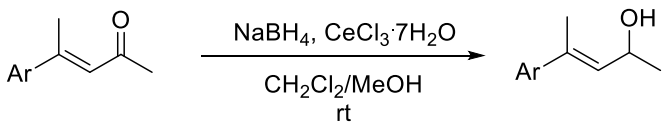
Column chromatography: silica gel, 5-20% AcOEt in hexanes. Yield: 2.08 g (96%) starting from 2.64 g of (*E*)-*N*-methoxy-*N*-methyl-3-(naphthalen-1-yl)but-2-enamide; yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93–7.85 (m, 2H), 7.81 (d, *J* = 8.3 Hz, 1H), 7.53–7.48 (m, 2H), 7.48–7.42 (m, 1H), 7.29 (d, *J* = 7.1 Hz, 1H), 6.38 (q, *J* = 1.4 Hz, 1H), 2.63 (d, *J* = 1.4 Hz, 3H), 2.30 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.8, 155.3, 142.3, 133.8, 130.1, 128.5, 128.2, 127.8, 126.3, 126.0, 125.3, 125.2, 124.2, 32.1, 22.1.

(*E*)-5-Methyl-4-phenylhex-3-en-2-one:



Column chromatography: silica gel, 5% AcOEt in hexanes. Yield: 562 mg (87%) starting from 802 mg of (*E*)-*N*-methoxy-*N*,4-dimethyl-3-phenylpent-2-enamide; yellowish oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36–7.27 (m, 3H), 7.24–7.15 (m, 2H), 6.06 (s, 1H), 4.03 (sept, *J* = 7.0 Hz, 1H), 2.23 (s, 3H), 1.06 (d, *J* = 7.0 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.7, 165.4, 140.9, 127.8, 127.6, 127.6, 126.0, 32.2, 29.5, 21.3.

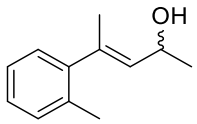
Synthesis of racemic allylic alcohols 3:



To a suspension of enone **1** (3 mmol) and $CeCl_3 \cdot 7H_2O$ (3.6 mmol) in CH_2Cl_2/MeOH (4:1 v/v, 50 mL), $NaBH_4$ (3.6 mmol) was added portionwise. After stirring at ambient temperature for 2 h, the reaction mixture was partitioned between CH_2Cl_2 and

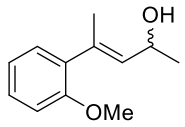
H_2O. The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2. The combined organic solutions were dried over anhydr. Na_2SO_4, and the solvent was removed under reduced pressure. The residue was chromatographed on silica gel (20% AcOEt in hexanes) to afford mixture of alcohols **3**.

***E*)-4-(*o*-Tolyl)pent-3-en-2-ol (3b):**



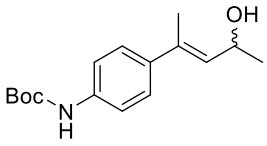
Yield: 246 mg (98%) starting from 248 mg enone **1b**; yellowish oil; spectral data in agreement with **2b**.

(*E*)-4-(2-Methoxyphenyl)pent-3-en-2-ol (3e):



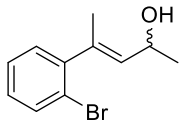
Yield: 168 mg (83%) starting from 200 mg enone **1e**; yellowish oil; spectral data in agreement with **2e**.

***t*-Butyl (*E*)-(4-(4-hydroxypent-2-en-2-yl)phenyl)carbamate (3f):**



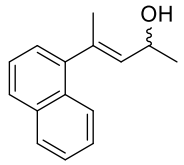
Yield: 221 mg (93%) starting from 235 mg enone **1f**; yellow oil; spectral data in agreement with **2f**.

(*E*)-4-(2-Bromophenyl)pent-3-en-2-ol (3i):



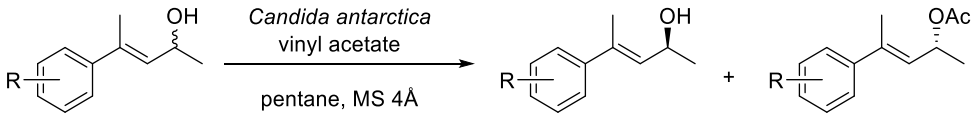
Yield: 167 mg (83%) starting from 200 mg enone **1i**; yellowish oil; spectral data in agreement with **2i**.

(*E*)-4-(Naphthalen-1-yl)pent-3-en-2-ol (3l):



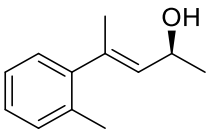
Yield: 188 mg (93%) starting from 200 mg enone **1l**; yellowish oil; spectral data in agreement with **2l**.

Enzymatic kinetic resolution of racemic β,β-disubstituted allyl alcohols 3:[4]



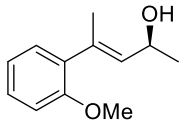
A suspension of racemic alcohols **3** (2.5 mmol), Novozyme 435 (18 mg), 4 Å molecular sieves (100 mg), and vinyl acetate (25 mmol, 2.2 g, 2.3 mL) in pentane (7 mL) was stirred in room temperature. The progress of the reaction was followed by ^{1}H NMR. The reaction mixture was filtered through Celite, and the solvent was removed under reduced pressure. The residue was chromatographed on silica gel (15% AcOEt in hexanes).

(*S*,*E*)-4-(*o*-Tolyl)pent-3-en-2-ol (2b):



The reaction mixture was stirred for 48 h. Yield: 109 mg (44%) starting from 247 mg racemic alcohol **3b**; yellowish oil; $[\alpha]_D^{23}$ –14.3 (*c* 1.06, $CHCl_3$), *e.e*. 93%; (HPLC: R_t 9.9 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21–7.12 (m, 3H), 7.12–7.06 (m, 1H), 5.38 (dq, *J* = 8.5, 1.6 Hz, 1H), 4.75 (dq, *J* = 8.5, 6.3 Hz, 1H), 2.30 (s, 3H), 2.00 (d, *J* = 1.6 Hz, 3H), 1.36 (d, *J* = 6.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.6, 137.6, 134.5, 133.6, 130.1, 128.0, 126.8, 125.6, 64.9, 23.5, 19.7, 18.3; FTIR (film) *v*: 3347, 2969, 2925, 1487, 1450, 1374, 1271, 1120, 1060, 759, 727 cm^{-1}; HRMS (EI) *m/z* calcd for $C_{12}H_{16}O$ [M] 176.1201; found 176.1198; HPLC (racemate) column Chiralcel OD-H, 10% *i*-PrOH in hexanes, flow 1 mL/min, det. 254 nm; 7.3 min (*R*-enantiomer) and R_t 9.7 min (*S*-enantiomer).

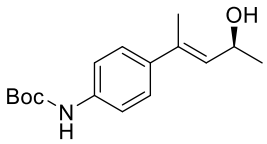
(*S*,*E*)-4-(2-Methoxyphenyl)pent-3-en-2-ol (2e):



The reaction mixture was stirred for 4 days. Yield: 107 mg (46%) starting from 233 mg racemic alcohol **3e**; yellowish oil; $[\alpha]_D^{22}$ –21.7 (*c* 1.32, $CHCl_3$), *e.e*. 96%; (HPLC: R_t 13.8 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26–7.20 (m, 1H), 7.14–7.10 (m, 1H), 6.94–6.88 (m, 1H), 6.88–6.84 (m, 1H), 5.50 (dq, *J* = 8.4, 1.5 Hz, 1H), 4.73 (dq, *J* = 8.4, 6.3 Hz, 1H), 3.81 (s, 3H), 2.04 (d, *J* = 1.5 Hz, 4H), 1.86 (br s, 1H), 1.33 (d, *J* = 6.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.6, 136.6, 133.9, 133.7, 129.5, 128.3, 120.6, 110.8, 64.8, 55.4, 23.3, 17.3; FTIR (film) *v*: 3367, 2967, 2927, 1489, 1463, 1435, 1248, 1227, 1121,

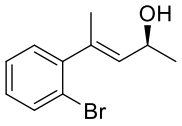
1054, 1028, 753 cm^{-1}; HRMS (ESI-TOF) *m/z* calcd for $C_{12}H_{16}O_2Na$ [$(M+Na)^+$] 215.1048; found 215.1043; HPLC (racemate) column Chiralcel OD-H, 10% *i*-PrOH in hexanes, flow 1 mL/min, det. 254 nm; 9.1 min (*R*-enantiomer) and R_t 13.8 min (*S*-enantiomer).

***tert*-Butyl (*S*,*E*)-(4-(4-hydroxypent-2-en-2-yl)phenyl)carbamate (2f):**



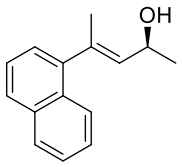
The reaction mixture was stirred for 24 h. Column chromatography: silica gel was deactivated by addition of TEA. Yield: 89 mg (46%) starting from 191 mg racemic alcohol **3f**; white solid; m.p. 102-103 °C $[\alpha]_D^{22}$ –36.5 (*c* 1.08, $CHCl_3$), *e.e*. 89%; (HPLC: R_t 23.9 min); ^1H NMR (400 MHz, $CDCl_3$) δ 7.30 (s, 4H), 6.69 (br s, 1H), 5.75 (dq, *J* = 8.3, 1.4 Hz, 1H), 4.72 (dq, *J* = 8.3, 6.3 Hz, 1H), 2.04 (d, *J* = 1.4 Hz, 3H), 1.90 (br s, 1H), 1.51 (s, 9H), 1.32 (d, *J* = 6.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 152.8, 137.5, 137.5, 135.4, 130.9, 126.3, 118.3, 80.5, 65.2, 28.3, 23.6, 15.9; FTIR (film) *v*: 3324, 2976, 2928, 1702, 1592, 1524, 1409, 1367, 1318, 1240, 1162, 1058, 830 cm^{-1}; HRMS (ESI-TOF) *m/z* calcd for $C_{16}H_{23}NO_3Na$ [$(M+Na)^+$] 300.1576; found 300.1571; HPLC (racemate) column Chiralcel OD-H, 10% *i*-PrOH in hexanes, flow 1 mL/min, det. 254 nm; 7.4 min (*R*-enantiomer) and R_t 9.9 min (*S*-enantiomer).

(*S*,*E*)-4-(2-Bromophenyl)pent-3-en-2-ol (2i):



The reaction mixture was stirred for 48 h. Yield: 69 mg (50%) starting from 138 mg racemic alcohol **3i**; yellowish oil; $[\alpha]_D^{22}$ –10.7 (*c* 2.31, $CHCl_3$), *e.e*. 91%; (HPLC: R_t 10.1 min); spectral data in agreement with literature:[2] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55–7.51 (m, 1H), 7.28–7.22 (m, 1H), 7.18–7.13 (m, 1H), 7.13–7.07 (m, 1H), 5.41 (dq, *J* = 8.5, 1.5 Hz, 1H), 4.73 (dq, *J* = 8.5, 6.3 Hz, 1H), 2.02 (d, *J* = 1.5 Hz, 3H), 1.61 (br s, 1H), 1.34 (d, *J* = 6.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 145.3, 137.6, 134.6, 132.7, 129.7, 128.3, 127.3, 122.0, 64.9, 23.2, 17.9; LRMS [EI] *m/z* 240.0; HPLC (racemate) column Chiralcel OD-H, 10% *i*-PrOH in hexanes, flow 1 mL/min, det. 254 nm; 7.4 min (*R*-enantiomer) and R_t 9.9 min (*S*-enantiomer).

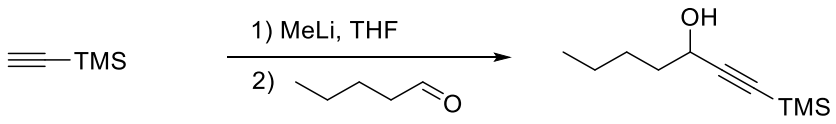
(*S*,*E*)-4-(Naphthalen-1-yl)pent-3-en-2-ol (2l):



The reaction mixture was stirred for 24 h. Yield: 101 mg (50%) starting from 201 mg racemic alcohol **3l**; yellowish oil; $[\alpha]_D^{23}$ –15.1 (*c* 1.37, $CHCl_3$), *e.e*. 99%; (HPLC: R_t 19.7 min); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05–7.94 (m, 1H), 7.93–7.84 (m, 1H), 7.78 (d, *J* = 8.2 Hz, 1H), 7.55–7.40 (m, 3H), 7.30 (d, *J* = 7.1 Hz, 1H), 5.62 (dq, *J* = 8.4, 1.6 Hz, 1H), 4.89 (dq, *J* = 8.5, 6.3 Hz, 1H), 2.20 (d, *J* = 1.6 Hz, 3H), 1.45 (d, *J* = 6.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 142.8, 136.7, 134.8, 133.8, 130.9, 128.4, 127.2, 125.8, 125.6, 125.5, 125.4, 124.8, 65.1, 23.7, 19.3; FTIR (film) *v*: 3341, 2967, 1444, 1395, 1373, 1120, 1056, 800, 777 cm^{-1}; HRMS (EI) *m/z* calcd for $C_{15}H_{16}O$ [M] 212.1201; found 212.1205; HPLC (racemate) column Chiralcel OD-H, 10% *i*-PrOH in hexanes,

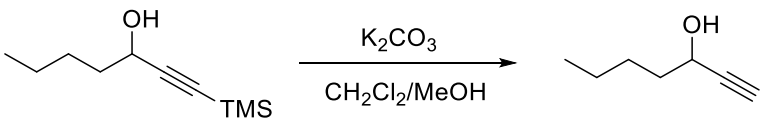
flow 1 mL/min, det. 254 nm; 16.2 min (*R*-enantiomer) and R_t 19.4 min (*S*-enantiomer).

1-(Trimethylsilyl)hept-1-yn-3-ol:



Ethynyltrimethylsilane (74.8 mmol, 7.4 g, 10.7 mL) was dissolved in THF (300 mL) and cooled to −78 °C under argon atmosphere. Then, a solution of MeLi (1.6 M in Et_2O, 81.6 mmol, 51 mL) was added dropwise. The mixture was stirred for 1.5 h at −78 °C. Next, pentanal (68.0 mmol, 5.8 g, 7.2 mL) was added dropwise. The mixture was slowly warmed to room temperature and stirred for another 1.5 h. The progress of the reaction was followed by NMR. When the reaction was complete, a saturated aqueous ammonium chloride solution was added to the mixture. The aqueous phase was extracted with Et_2O and the combined organic phase was dried over sodium sulfate. After the removal of the solvent, the crude residue was purified by column chromatography on silica gel (5% AcOEt in hexanes). Yield: 11.23 g (90%); colourless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.33 (td, *J* = 6.6, 5.4 Hz, 1H), 1.97 (br s, 1H), 1.76–1.59 (m, 2H), 1.53–1.21 (m, 4H), 0.90 (t, *J* = 7.1 Hz, 3H), 0.15 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 107.0, 89.2, 62.8, 37.4, 27.3, 22.3, 13.9, −0.2.

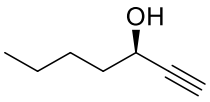
Hept-1-yn-3-ol:



A suspension of 1-(trimethylsilyl)hept-1-yn-3-ol (60.7 mmol, 11.2 g), K_2CO_3 (121.4 mmol, 16 g), MeOH (30 mL) and CH_2Cl_2 (100 mL) was stirred overnight at room temperature. When the reaction was complete, water was added to the mixture. The aqueous phase was extracted with CH_2Cl_2 and the combined organic phase was dried over sodium sulfate. After the removal of the solvent, the crude residue was purified by column chromatography on silica gel (10-50% Et_2O in pentanes). Yield: 6.21 g (91%); colourless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.32 (tdd, *J* = 6.7, 5.3, 2.1 Hz, 1H), 2.59 (br s, 1H), 2.42 (d, *J* = 2.1 Hz, 1H), 1.76–1.59 (m, 2H), 1.48–1.25 (m, 4H), 0.88 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 85.1, 72.7, 62.1, 37.3, 27.1, 22.3, 13.9.

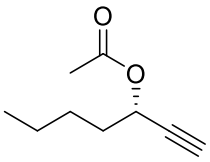
Kinetic resolution of hept-1-yn-3-ol: A suspension of racemic hept-1-yn-3-ol (52.7 mmol), Novozyme 435 (392 mg), 4 Å molecular sieves (1.8 g), and vinyl acetate (263.6 mmol) in pentane (73 mL) was stirred at room temperature. The progress of the reaction was followed by ^{1}H NMR. The reaction mixture was stirred for 5 h, and filtered through Celite. The solvent was carefully removed under reduced pressure (500-600 mBar). The residue was chromatographed on silica gel (0-50% Et_2O in pentanes). The purified product was analyzed by chiral GC for the determination of *e.e*. Yield: 3.96 g (49%) of (*S*)-hept-1-yn-3-yl acetate and 2.87 g (48%; *e.e*. 90%) of (*R*)-hept-1-yn-3-ol. In order to achieve enriched enantiopurity of the (*R*)-allyl alcohol, enzymatic resolution was performed again. Yield: 2.00 g (70%) of (*R*)-hept-1-yn-3-ol.

(*R*)-Hept-1-yn-3-ol:



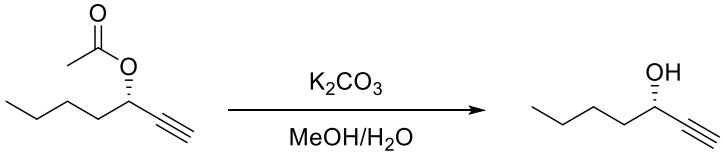
Yield: 2.00 g (34%, after 2 steps); colourless liquid; $[\alpha]_D^{20}$ +11.3 (*c* 1.07, $CHCl_3$), *e.e*. 99%; (GC: R_t 15.4 min) [lit.[5] $[\alpha]_D^{20}$ +9.3 (*c* 1.05, $CHCl_3$) for *e.e*. 97%]; spectral data in agreement with literature:[5] ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.32 (tdd, *J* = 6.7, 5.3, 2.1 Hz, 1H), 2.59 (br s, 1H), 2.42 (d, *J* = 2.1 Hz, 1H), 1.76–1.59 (m, 2H), 1.48 – 1.25 (m, 4H), 0.88 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 85.1, 72.7, 62.1, 37.3, 27.1, 22.3, 13.9; LRMS [EI] m/z 112.1; GC (racemate) column Agilent Cyclosil-B (30m x 0.25 mm x 0.25 µm), Injection temp.: 150 °C, Detection temp.: 270 °C, Oven temp: 90 °C (30 min), 5°C/min to 150°, hold for 5.00 min; 14.2 min (*S*-enantiomer) and R_t 15.5 min (*R*-enantiomer).

(*S*)-Hept-1-yn-3-yl acetate:



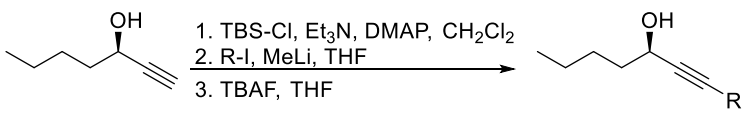
Yield: 3.96 g (49%); yellowish liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.34 (td, *J* = 6.7, 2.2 Hz, 1H), 2.43 (d, *J* = 2.2 Hz, 1H), 2.08 (s, 3H), 1.86–1.70 (m, 2H), 1.49–1.27 (m, 4H), 0.91 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.8, 81.3, 73.3, 63.8, 34.3, 27.0, 22.2, 20.9, 13.8.

(*S*)-Hept-1-yn-3-ol:



A suspension of (*S*)-hept-1-yn-3-yl acetate (25.4 mmol, 3.9 g), K_2CO_3 (50.8 mmol, 7.0 g), MeOH (50 mL) and H_2O (20 mL) was stirred for 15 minutes at room temperature. When the reaction was complete, water was added to the mixture. The aqueous phase was extracted with CH_2Cl_2 and the combined organic phase was dried over sodium sulfate. After the removal of the solvent, the crude product (NMR spectrum was clean) was used in the next step without further purification. Yield: 2.82 g (99%, *e.e*. 86%) of (*S*)-hept-1-yn-3-ol. Because of the low enantiomeric excess of (*S*)-hept-1-yn-3-ol (25.4 mmol, *e.e*. 86%), enzymatic resolution was performed again to enrich enantiopurity as in case of (*R*)-enantiomer (yield: 3.05 g, 78%). Hydrolysis was performed again, according to the procedure described above. Yield: 2.20 g (37%, after 4 steps) of (*S*)-hept-1-yn-3-ol; colorless oil; $[\alpha]_D^{19}$ –5.2 (*c* 1.07, $CHCl_3$), *e.e*. 97%; (GC: R_t 13.8 min) [lit.[29] $[\alpha]_D^{20}$ –9.7 (*c* 1.03, $CHCl_3$) for *e.e*. >99.9%]; spectral data were identical with those for *R*-enantiomer; LRMS [EI] *m/z* 112.1; GC (racemate) column Agilent Cyclosil-B (30m x 0.25 mm x 0.25 µm), Injection temp.: 150 °C, Detection temp.: 270 °C, Oven temp: 90 °C (30 min), 5°C/min to 150°, hold for 5.00 min; 14.2 min (*S*-enantiomer) and R_t 15.5 min (*R*-enantiomer).

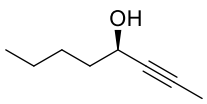
Synthesis of alkynes 13:



A solution of TBSCl (3.7 mmol, 558 mg) dissolved in CH_2Cl_2 (4 mL) was added dropwise to a solution of (*R*)-hept-1-yn-3-ol (3.7 mmol, 415 mg), Et_3N (7.4 mmol,

749 mg, 1 mL), DMAP (0.37 mmol, 45 mg) and CH_2Cl_2 (8 mL) at 0 °C. After stirring overnight, sat. NH_4Cl was added and the aqueous phase was extracted with CH_2Cl_2. The combined organic phase was dried over Na_2SO_4. After the removal of the solvent, the crude residue was filtered through a short pad of silica gel and rinsed with pentanes. O-Silylated alcohol was dissolved in THF (18 mL), cooled to −78 °C, and a 1.6 M soln. of MeLi in Et_2O (4.1 mmol, 2.8 mL) was added dropwise. After stirring for 1 h at −78 °C, alkyl iodide (4.1 mmol) was added, and the mixture was allowed to warm slowly to rt and left to stand overnight. Next, aq. NH_4Cl was added, the aqueous phase was extracted with CH_2Cl_2, and the combined organic layers was dried over Na_2SO_4. After the removal of the solvent, the crude residue was dissolved in THF (10 mL), cooled to 0 °C, and TBAF (1M in THF, 5.6 mmol, 5.6 mL) was added dropwise. The mixture was allowed to warm slowly to rt and left overnight. Next, water was added, the aqueous layer was extracted with CH_2Cl_2. The combined organic phase was dried over Na_2SO_4. After removal of solvents, the crude product was purified by column chromatography on silica gel. If necessary, the silyl by-products were removed by bulb-to-bulb distillation prior to chromatography.

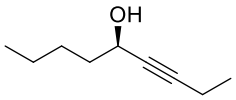
(*R*)-Oct-2-yn-4-ol (13a):



Column chromatography: silica gel, 10-50% Et_2O in pentanes. Bulb-to-bulb distillation (Kugelrohr) at 50-60 °C under 5 Torr to remove silyl by-products. Yield: 348 mg (75%, after 3

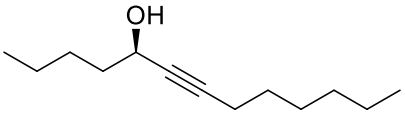
steps) starting from 412 mg (*R*)-hept-1-yn-3-ol; yellowish liquid; $[\alpha]_D^{17}$ +3.6 (*c* 0.98, $CHCl_3$), *e.e*. 99%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.41 – 4.22 (m, 1H), 1.84 (d, *J* = 2.2 Hz, 3H), 1.71 (d, *J* = 5.5 Hz, 1H), 1.70 – 1.60 (m, 2H), 1.47 – 1.27 (m, 4H), 0.91 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 80.7, 80.6, 62.7, 37.8, 27.3, 22.4, 13.9, 3.4; LRMS [EI] m/z 126.1.

(*R*)-Non-3-yn-5-ol (13b):



Column chromatography: silica gel, 5-10% AcOEt in hexanes. Yield: 85 mg (83%, after 3 steps) starting from 82 mg (*R*)-hept-1-yn-3-ol; yellowish liquid; $[\alpha]_D^{24}$ +2.9 (*c* 1.00, $CHCl_3$), *e.e*. 99%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.46–4.17 (m, 1H), 2.21 (qd, *J* = 7.5, 2.0 Hz, 2H), 1.76–1.56 (m, 3H), 1.49–1.23 (m, 4H), 1.13 (t, *J* = 7.5 Hz, 3H), 0.91 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 86.6, 80.8, 62.6, 37.8, 27.3, 22.3, 13.9, 13.8, 12.3; LRMS [EI] m/z 140.1.

(*R*)-Tridec-6-yn-5-ol (13c):



Column chromatography: silica gel, 5-10% AcOEt in hexanes. Yield: 95 mg (70%, after 3 steps) starting from 77 mg (*R*)-hept-1-yn-3-ol; yellowish liquid; $[\alpha]_D^{17}$ +6.7 (*c* 0.99, $CHCl_3$), *e.e*. 99%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.31 (tt, *J* = 6.6, 2.0 Hz, 1H), 2.17 (td, *J* = 7.1, 2.0 Hz, 2H), 2.03 (br s, 1H), 1.73–1.55 (m, 2H), 1.54–1.17 (m, 12H), 0.99–0.76 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 85.3, 81.4, 62.6, 37.9, 31.3, 28.6, 28.5, 27.4, 22.5, 22.4, 18.6, 13.9; LRMS [EI] m/z 196.2.

References:

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2. Voigtritter, K. R.; Isley, N. A.; Moser, R.; Aue, D. H.; Lipshutz, B. H., *Tetrahedron* **2012**, *68*, 3410-3416.
3. Bethi, V.; Fernandes, R. A., *J. Org. Chem.* **2016**, *81*, 8577-8584.
4. Szcześniak, P.; Październiok-Holewa, A.; Klimczak, U.; Stecko, S., *J. Org. Chem.* **2014**, *79*, 11700-11713.
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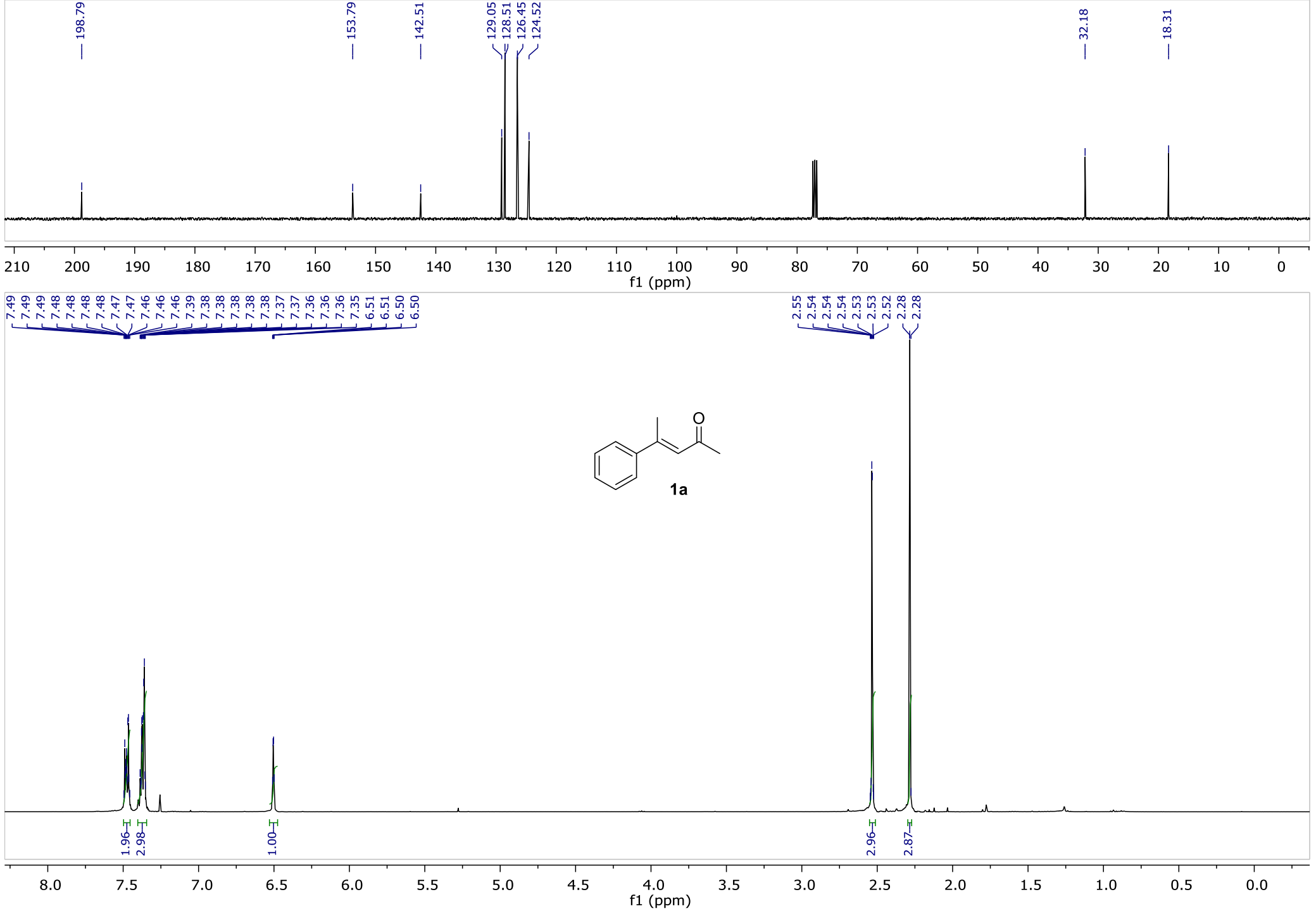
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126.45
124.52
32.18
18.31
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2.55 2.54 2.54 2.54 2.53 2.53 2.52 2.28 2.28
O
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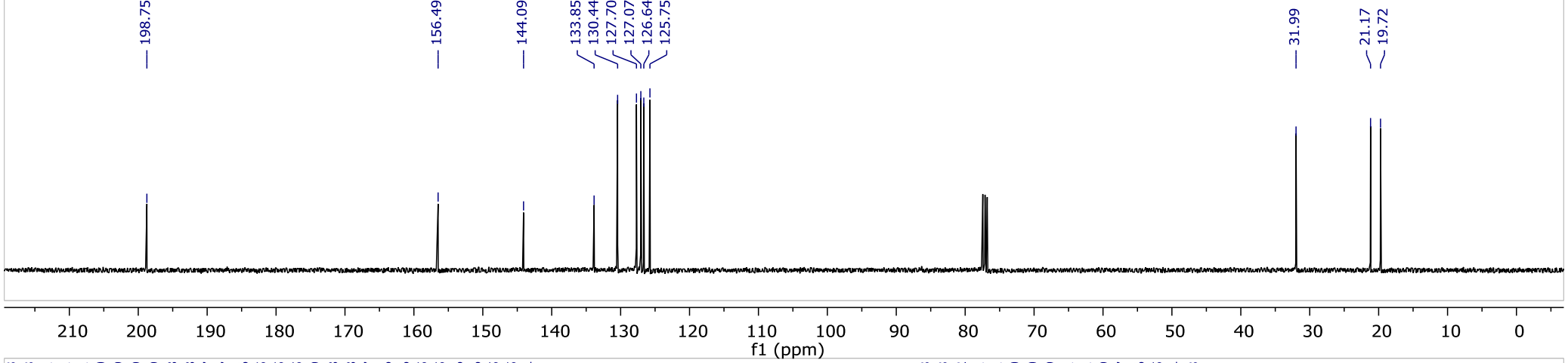
1.96
2.98
1.00
2.96
2.87
f1 (ppm)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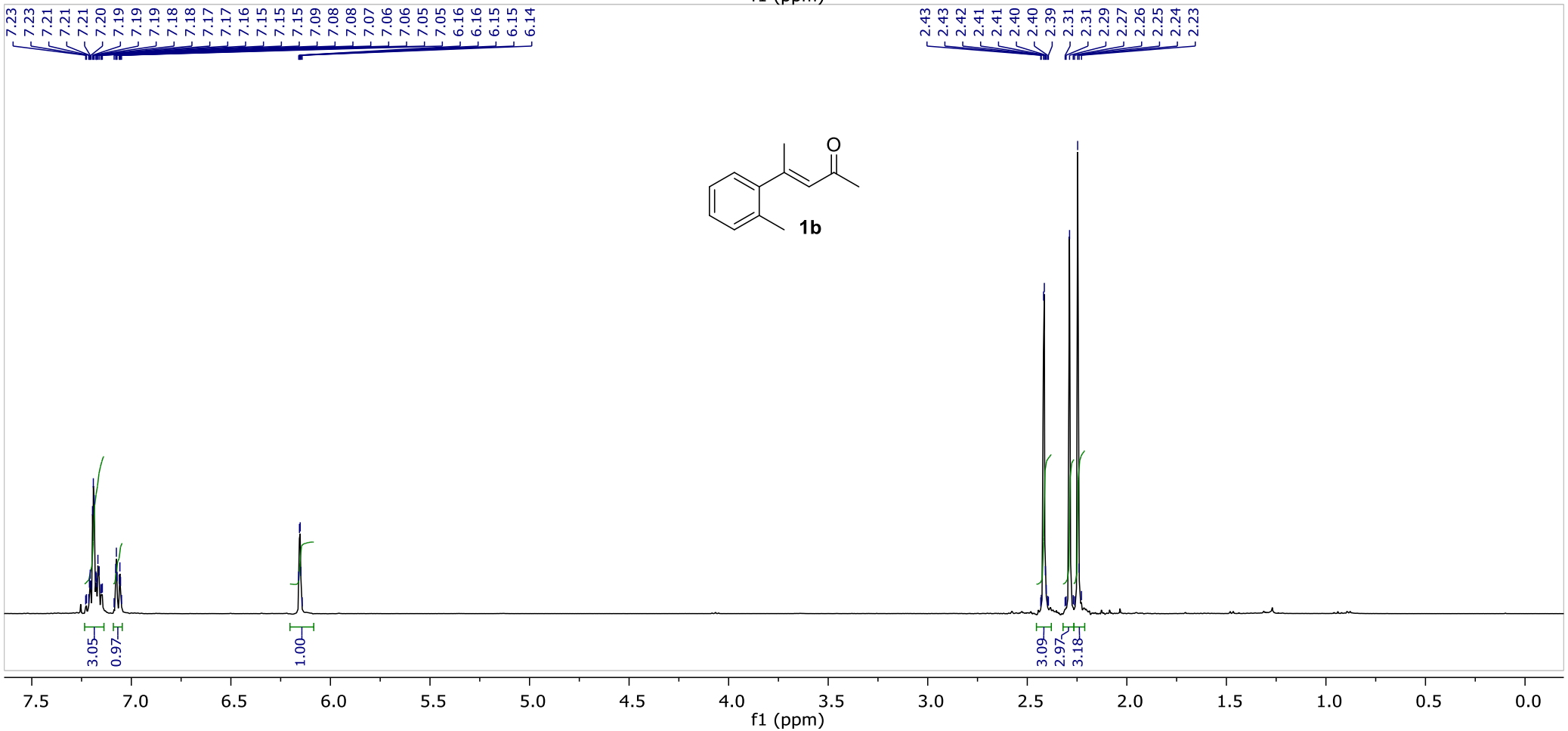
f1 (ppm)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2.43 2.43 2.42 2.41 2.41 2.40 2.40 2.39 2.31 2.31 2.29 2.27 2.26 2.25 2.24 2.23
O
1b
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0.97
1.00
3.09
2.97
3.18
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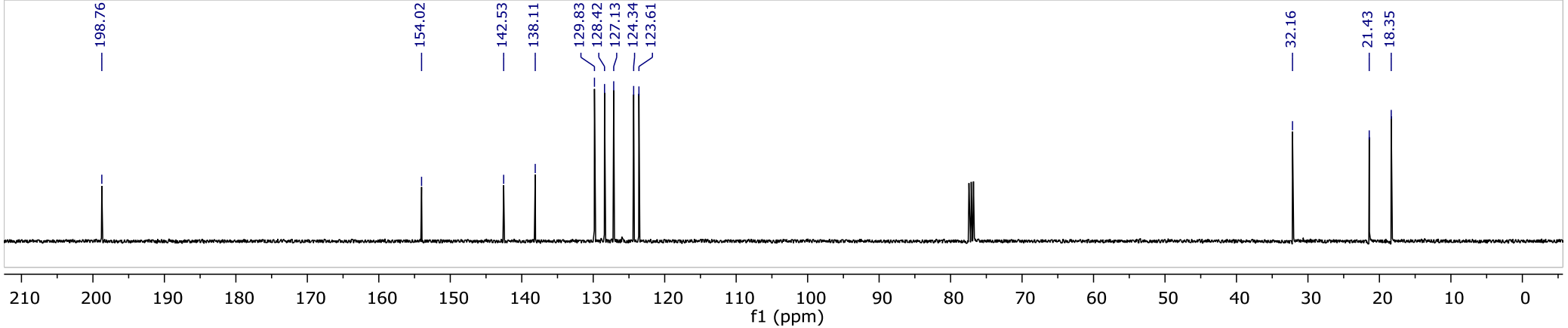
f1 (ppm)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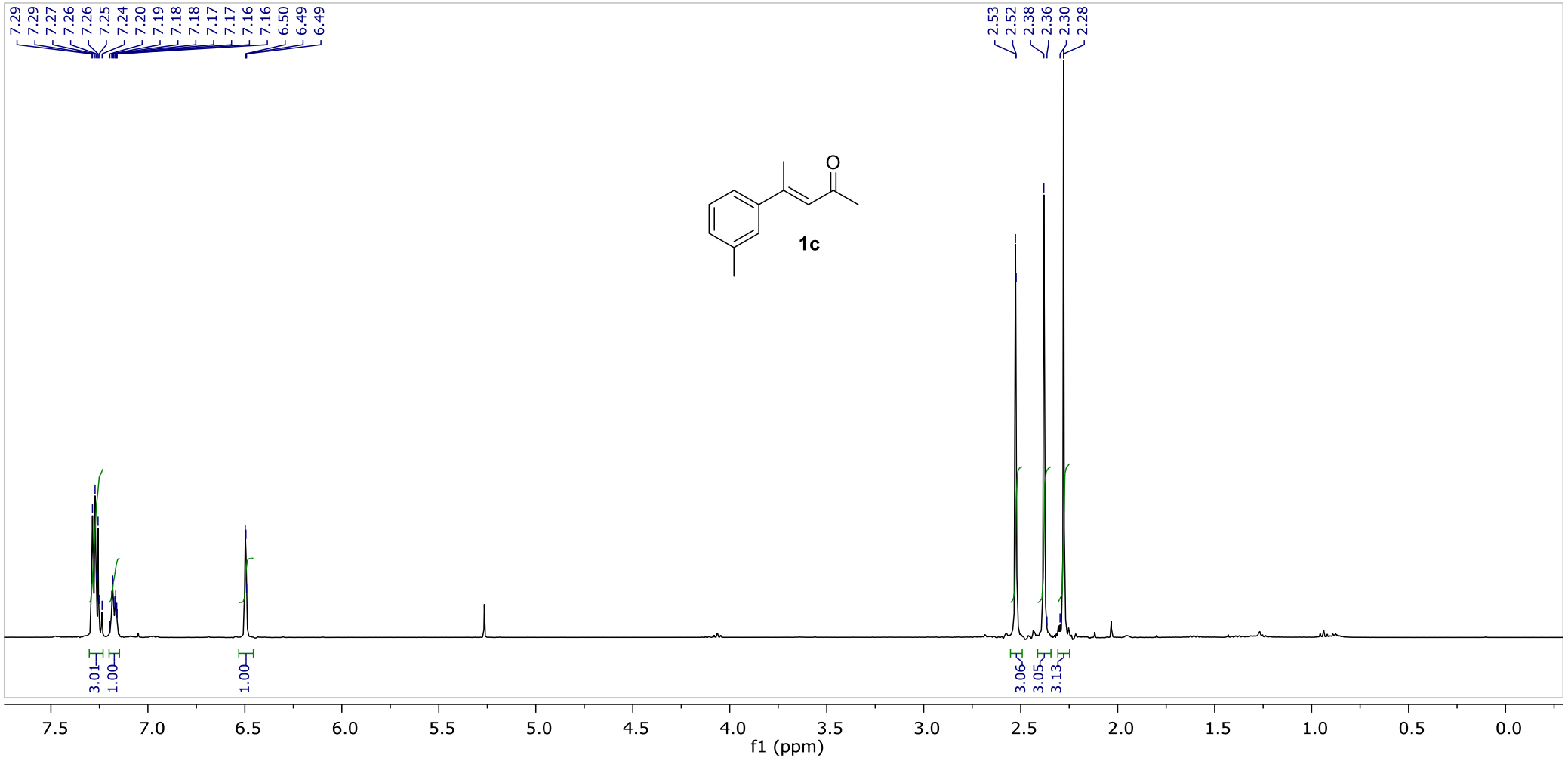
f1 (ppm)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O
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3.01 1.00 1.00 3.06 3.05 3.13
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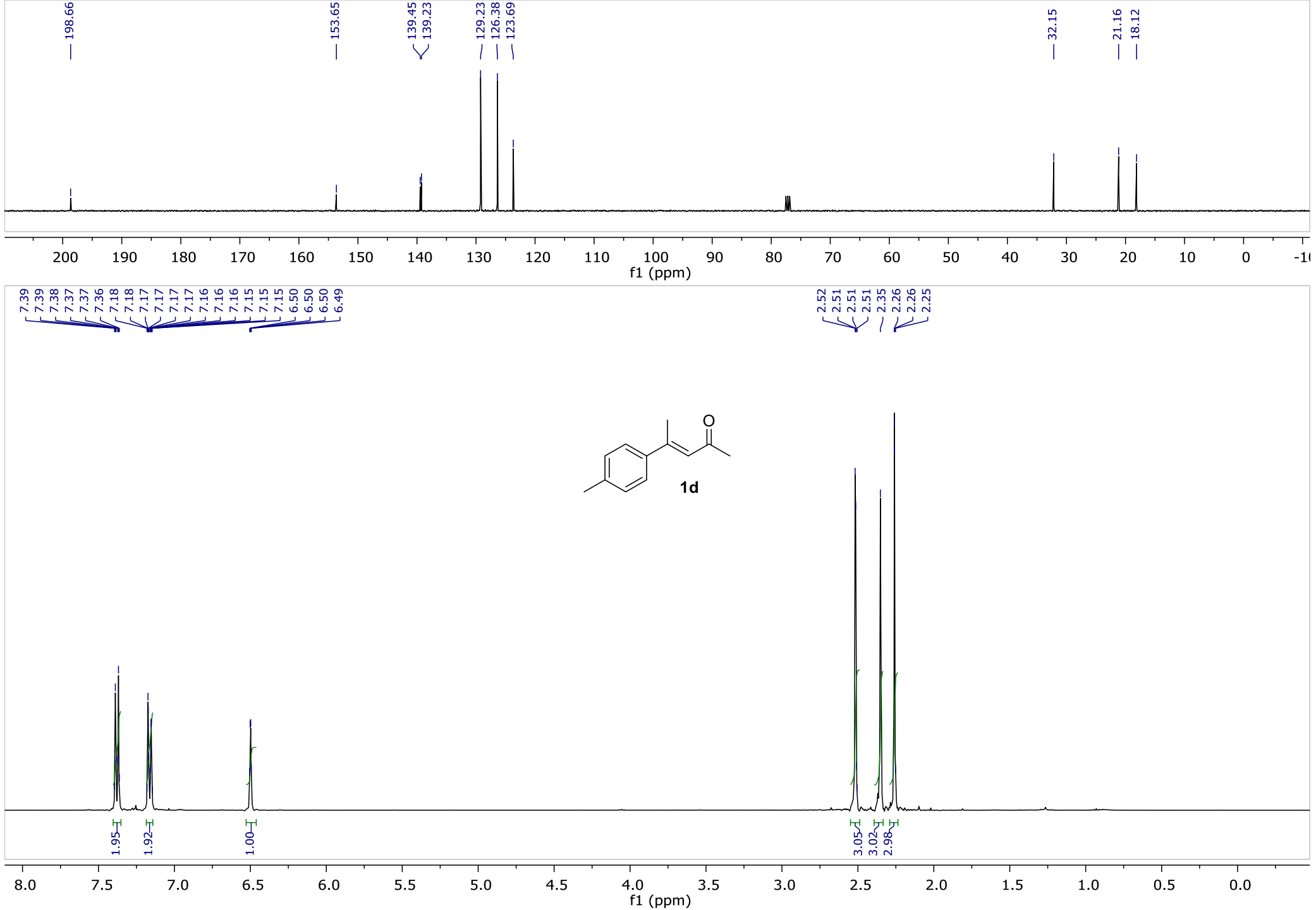
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6.49
2.52
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2.51
2.35
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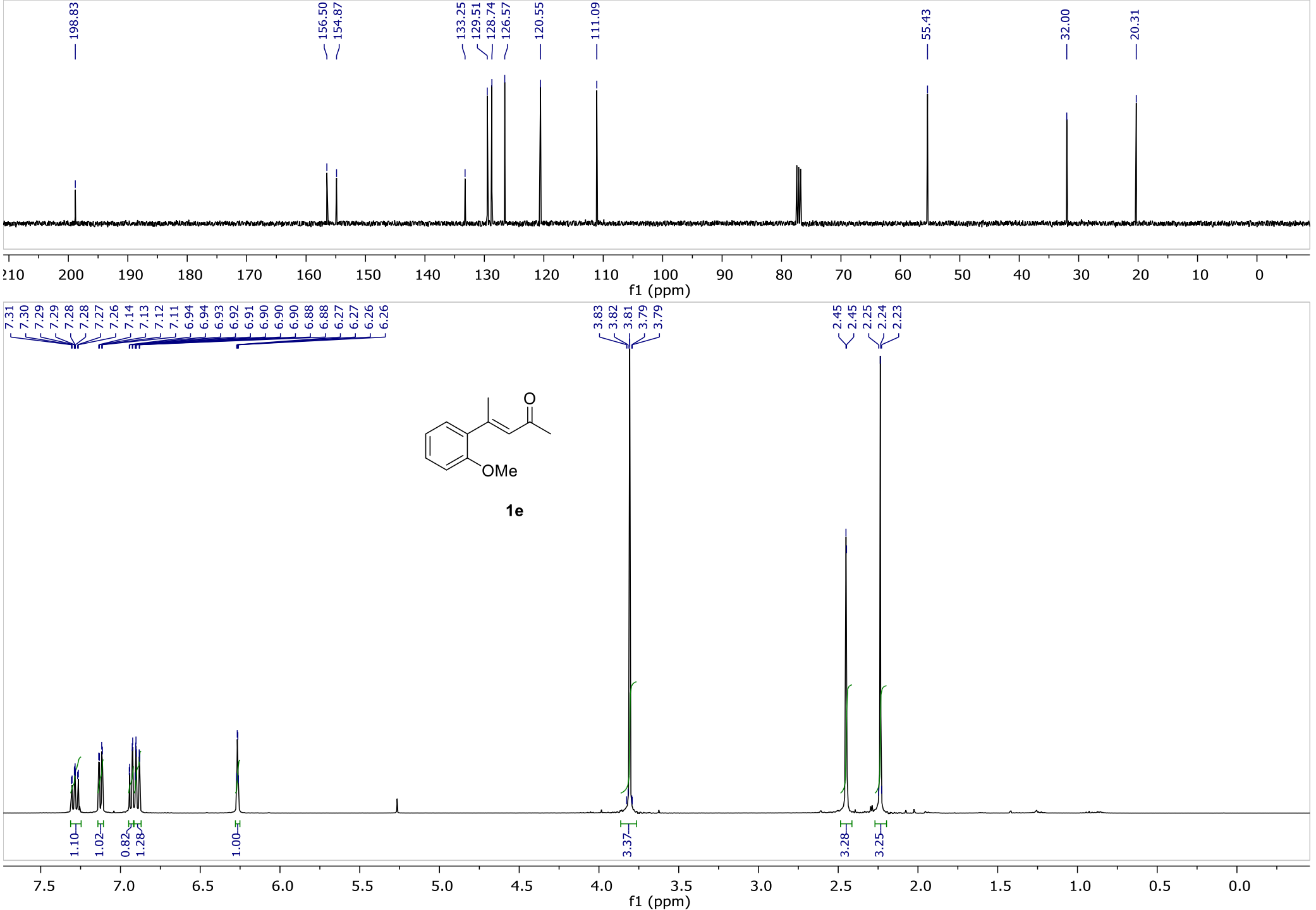
f1 (ppm)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7.28
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7.26
7.14
7.13
7.12
7.11
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6.94
6.93
6.92
6.91
6.90
6.90
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3.81
3.79
3.79
2.45
2.45
2.25
2.24
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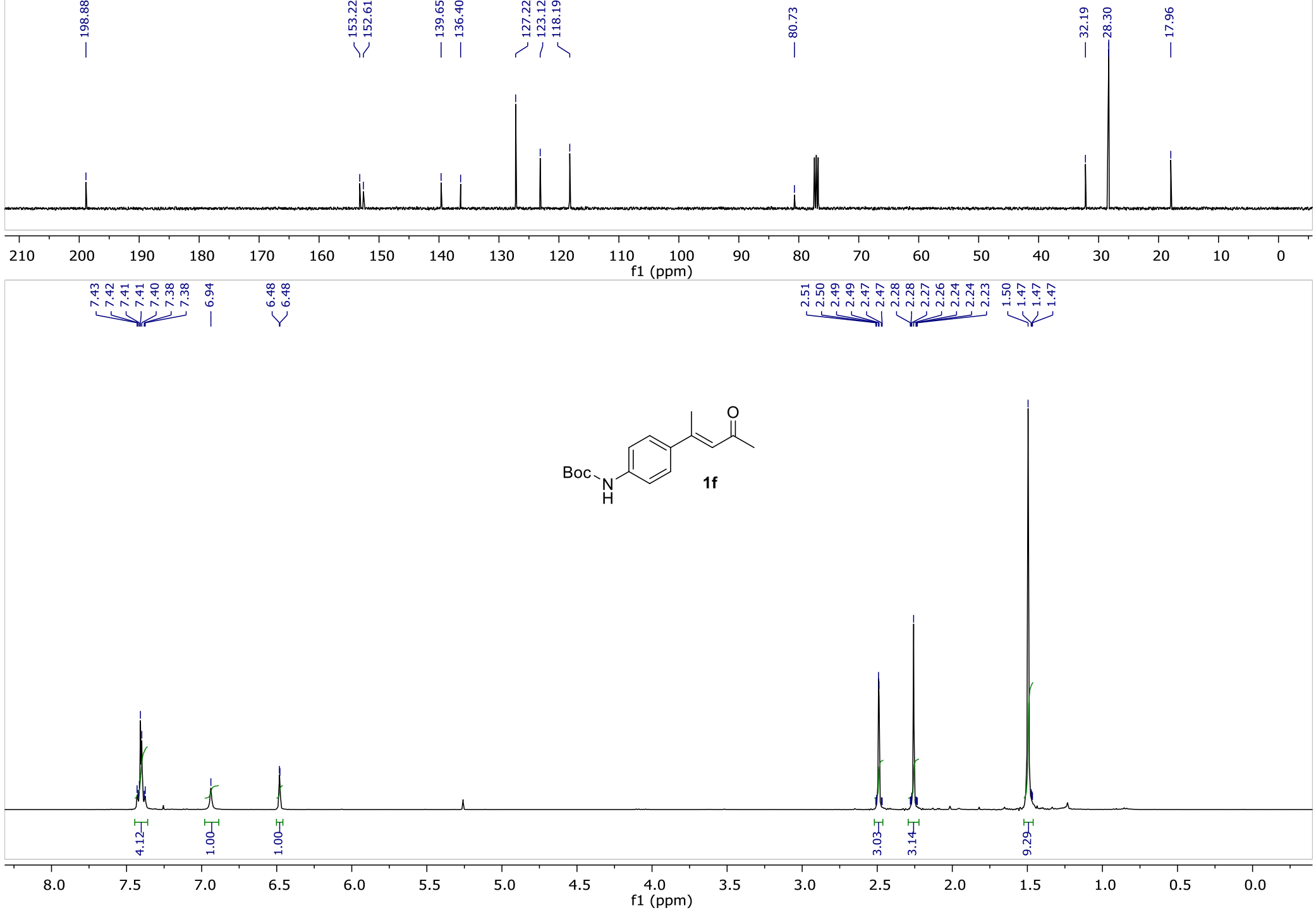
f1 (ppm)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6.94
6.48 6.48
2.51 2.50 2.49 2.49 2.47 2.47 2.28 2.28 2.27 2.26 2.24 2.24 2.23
1.50 1.47 1.47 1.47
Boc
N
H
O
1f
4.12
1.00
1.00
3.03
3.14
9.29
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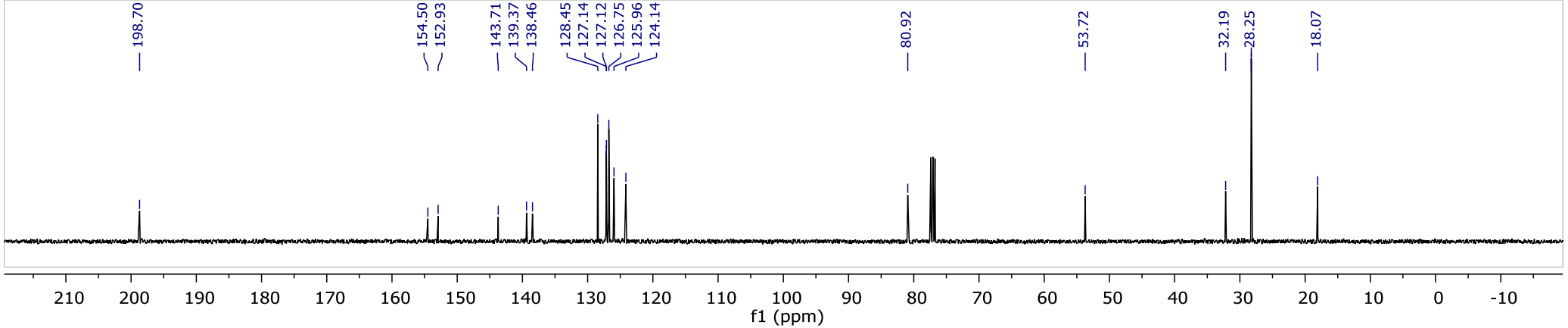
f1 (ppm)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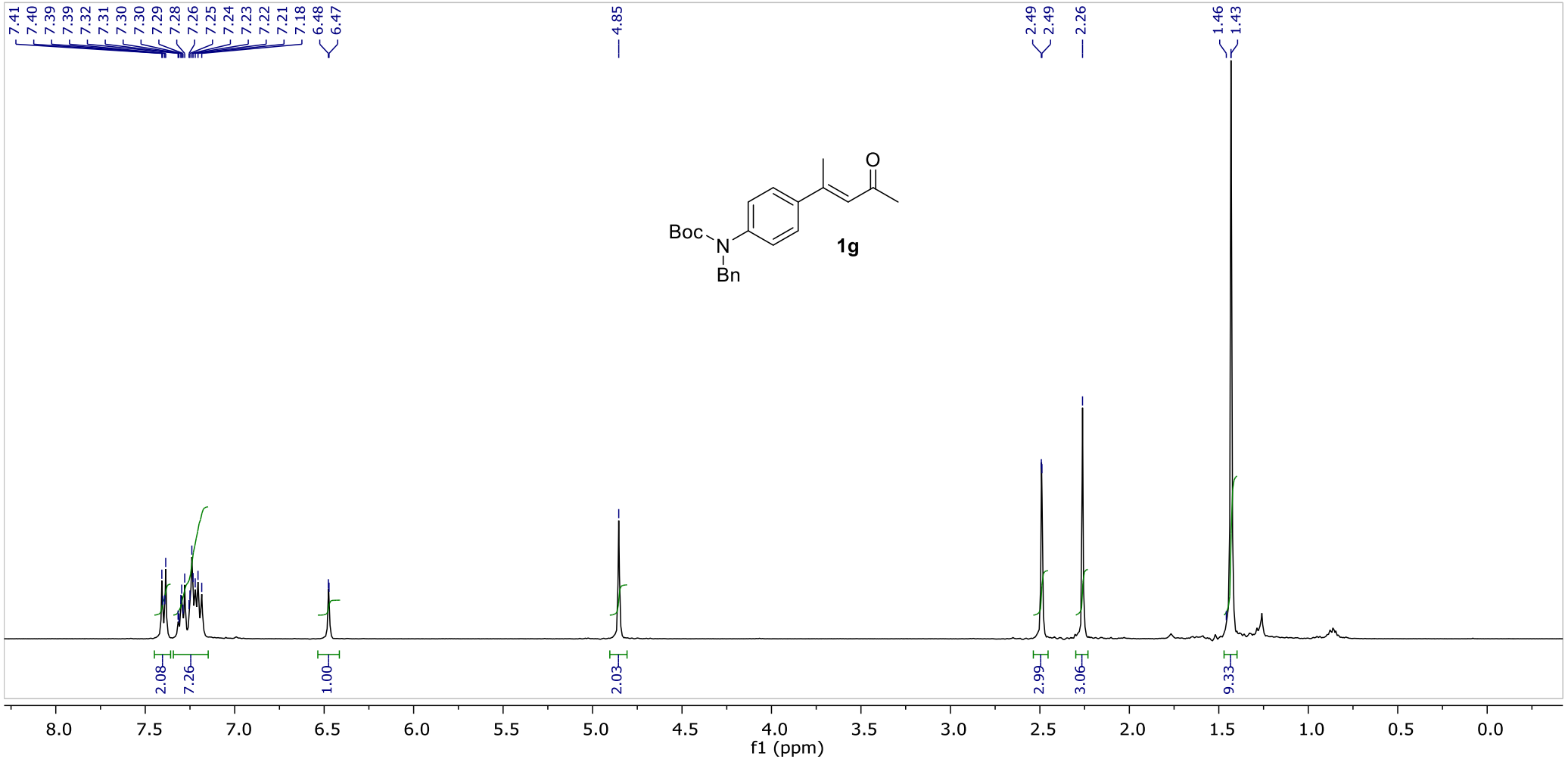
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7.23
7.22
7.21
7.18
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Boc
N
Bn
O
1g
2.08
7.26
1.00
2.03
2.99
3.06
9.33
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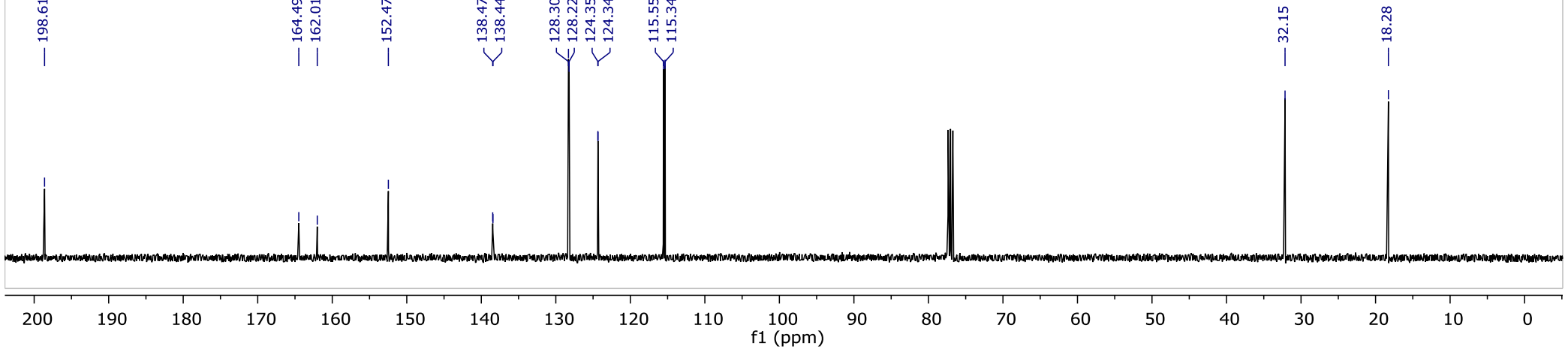
f1 (ppm)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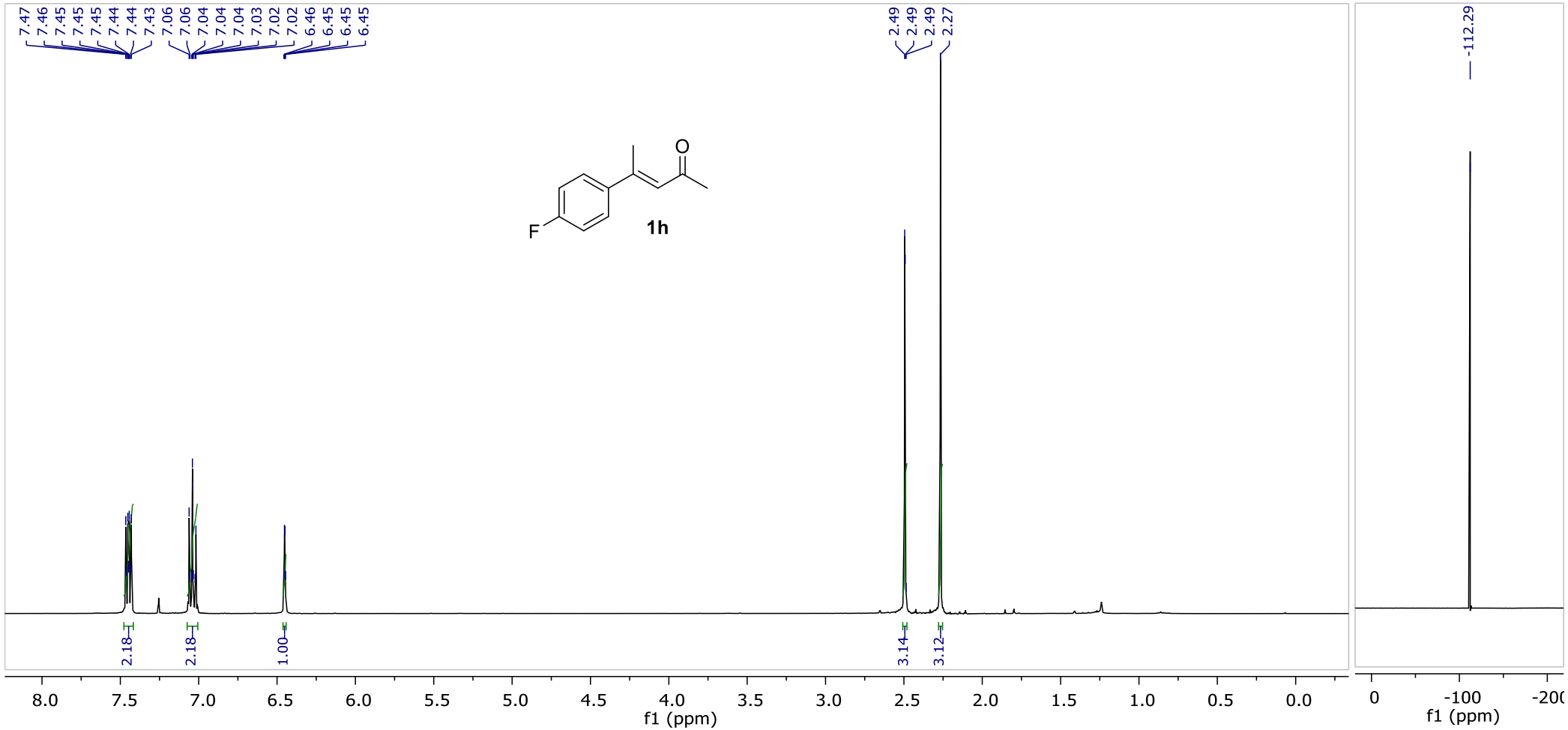
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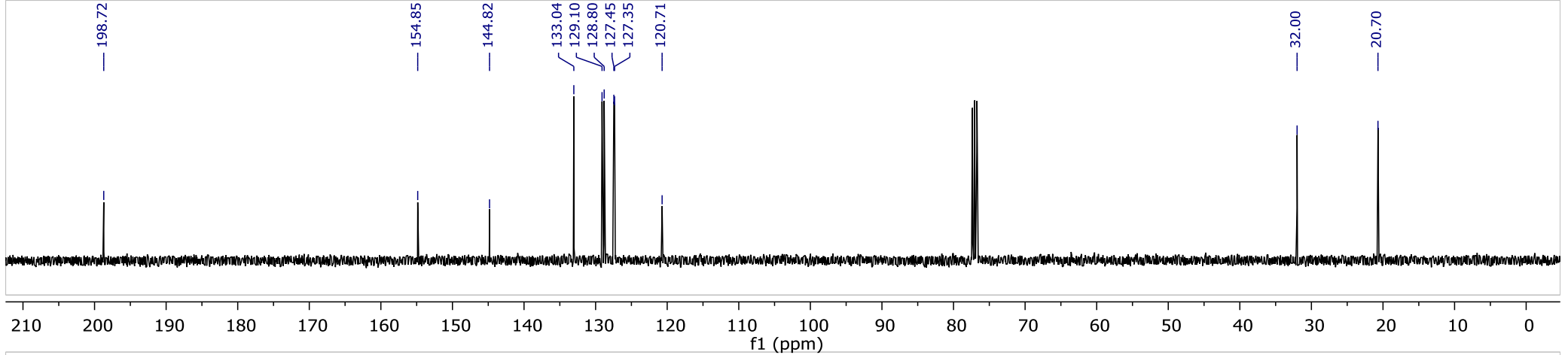
f1 (ppm)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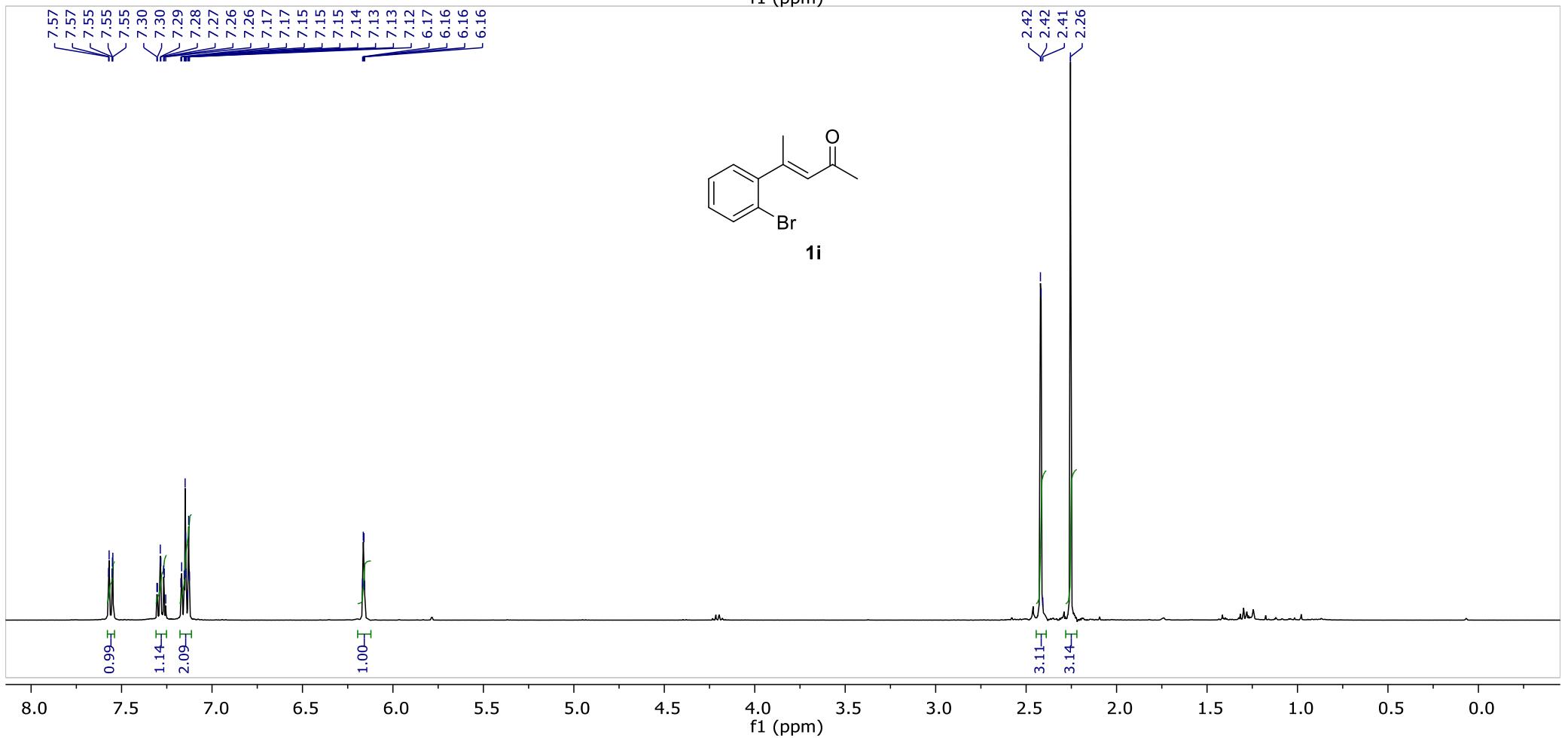
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O
Br
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1.00
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3.14
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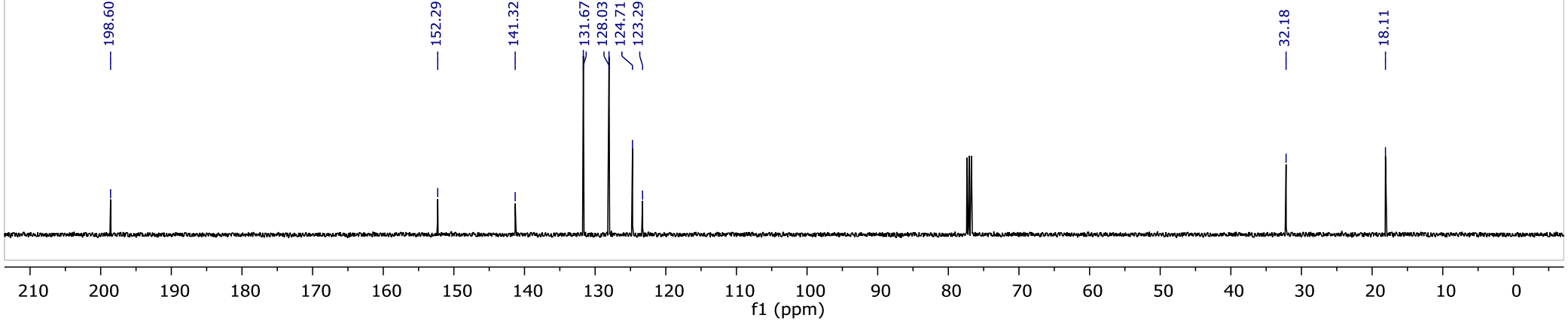
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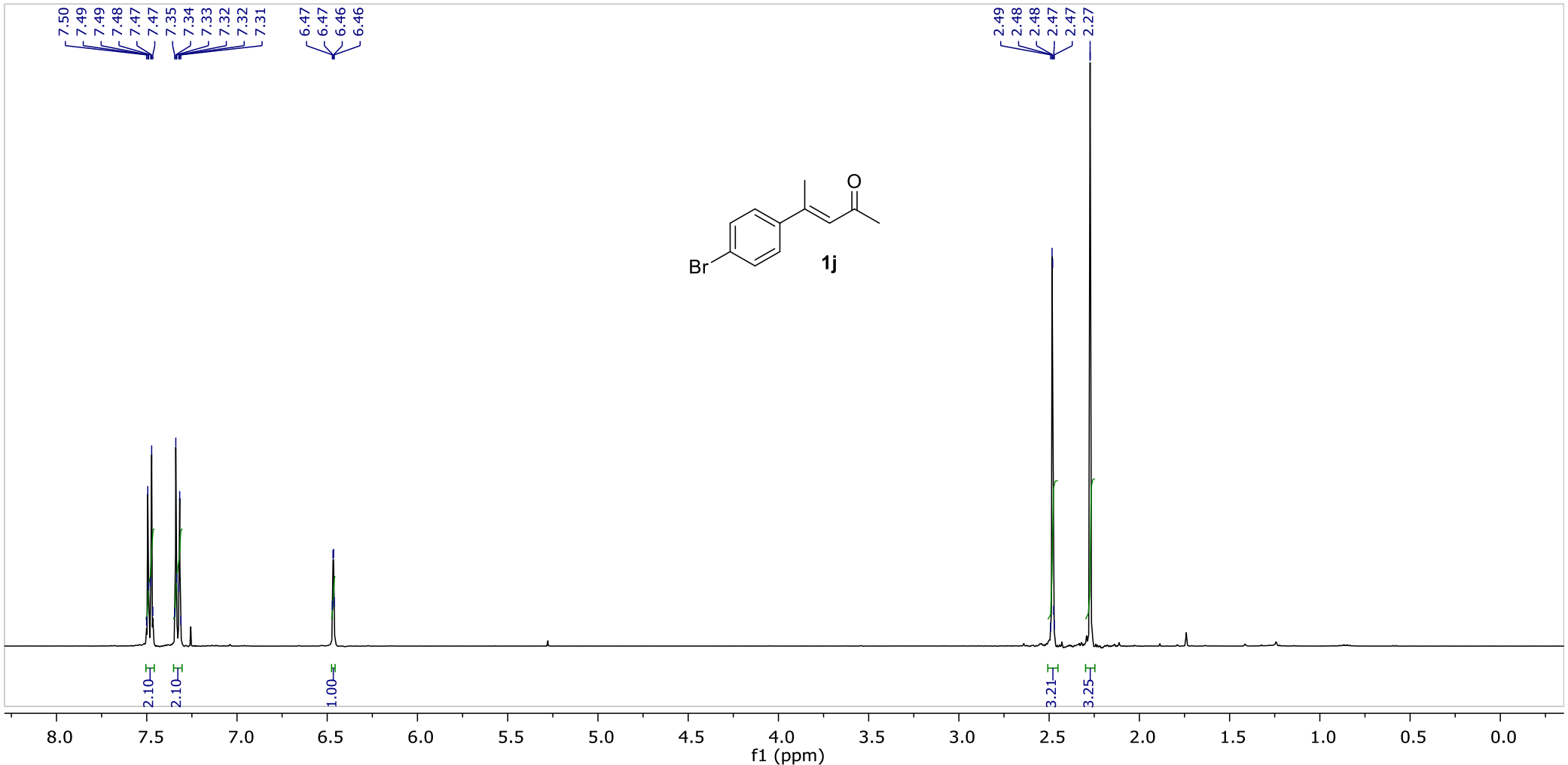
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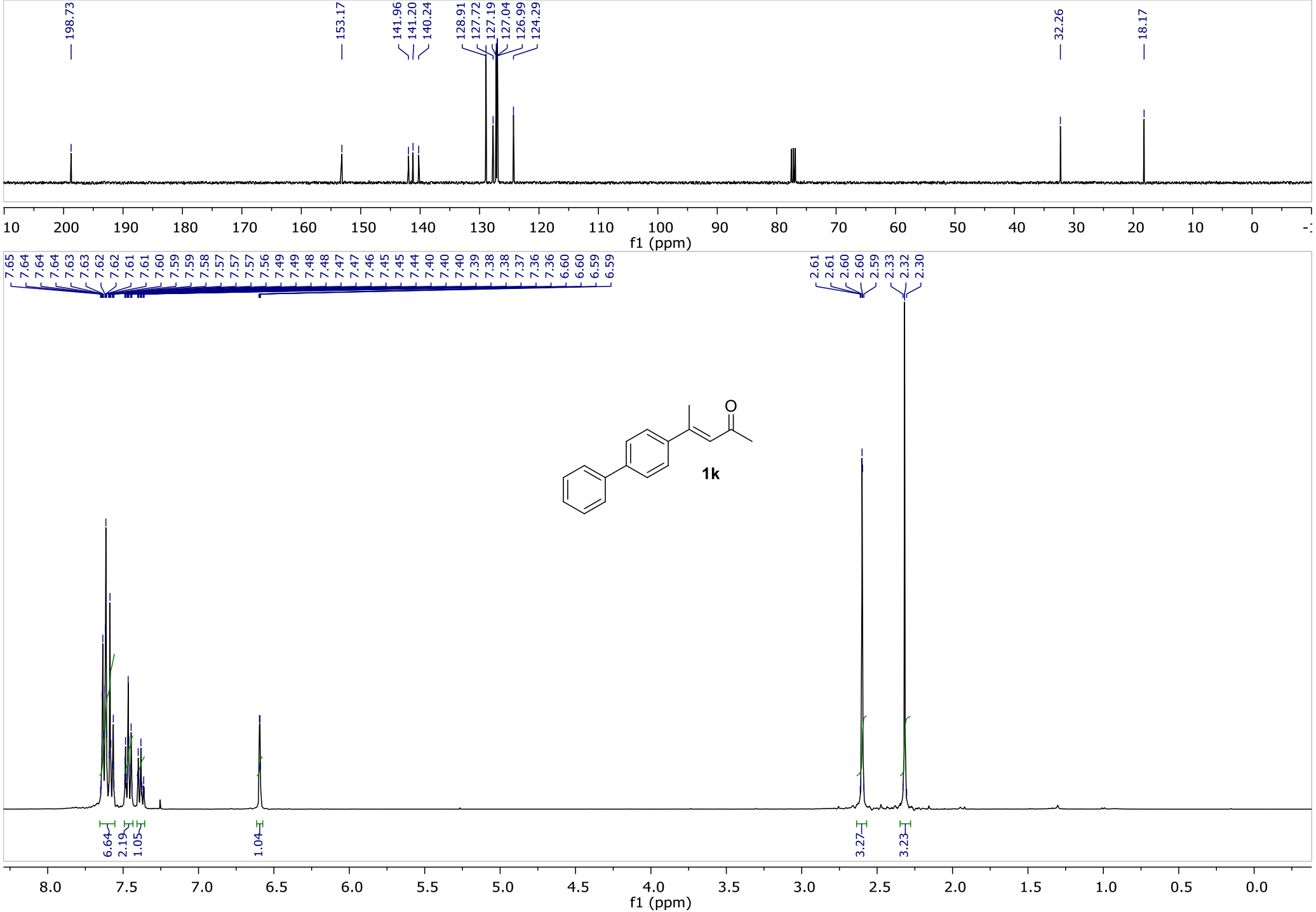
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2.61 2.61 2.60 2.60 2.59 2.33 2.32 2.30
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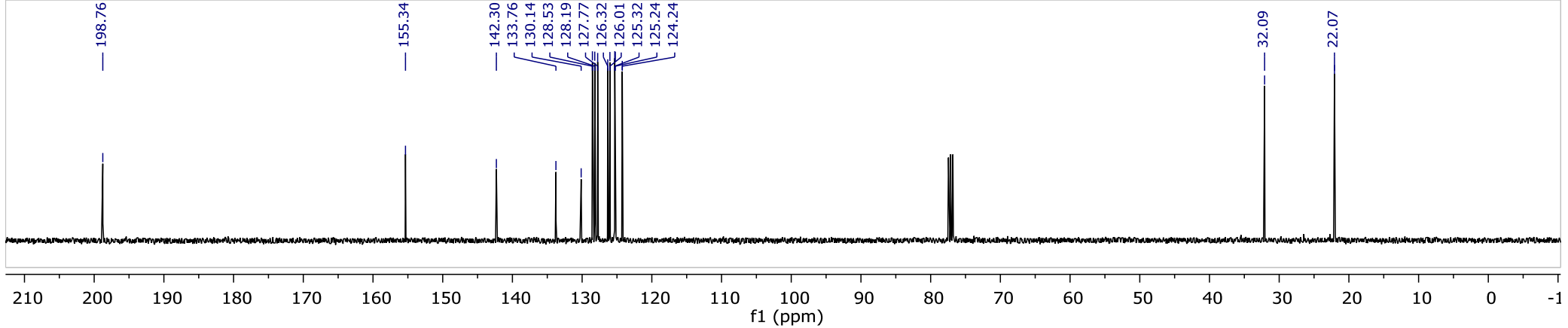
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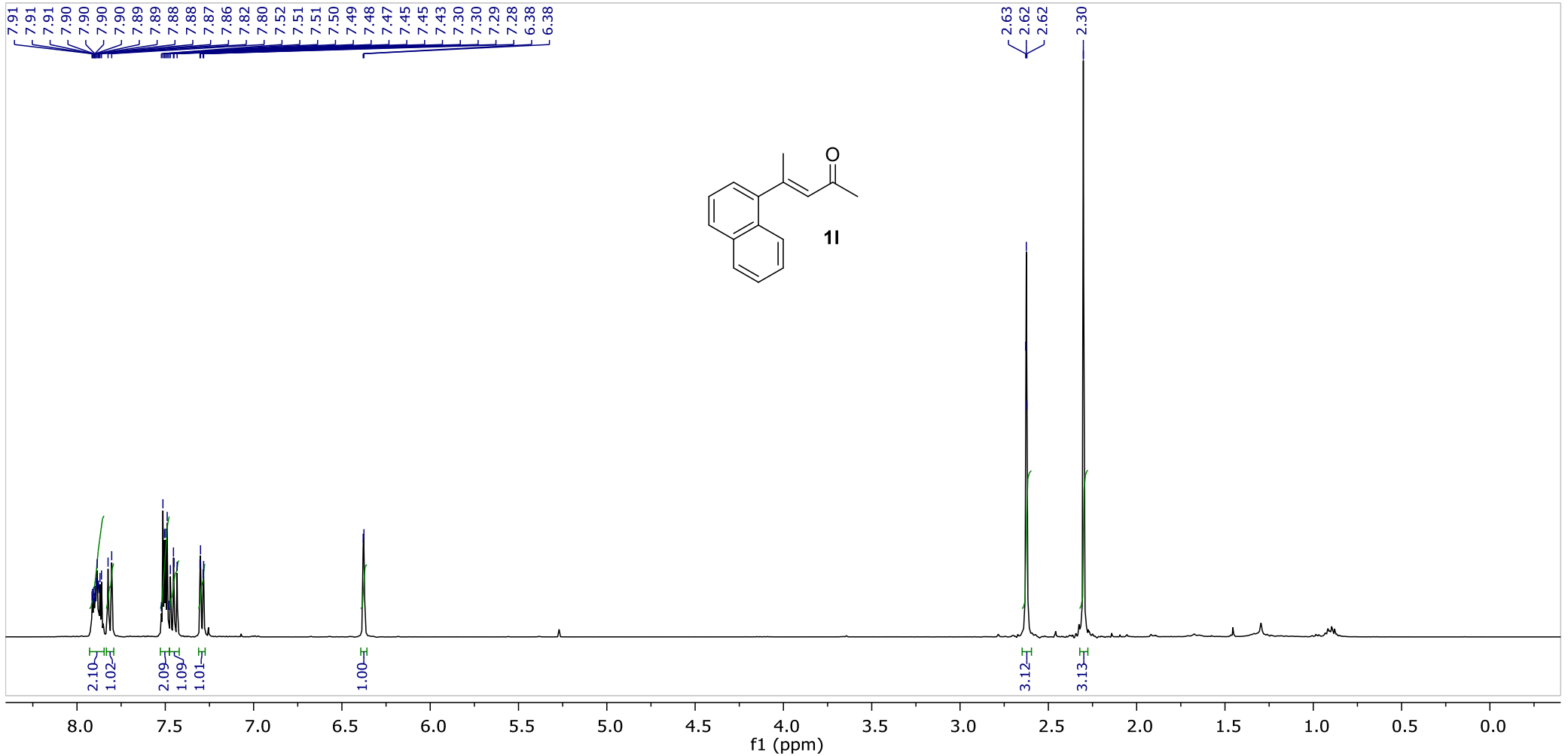
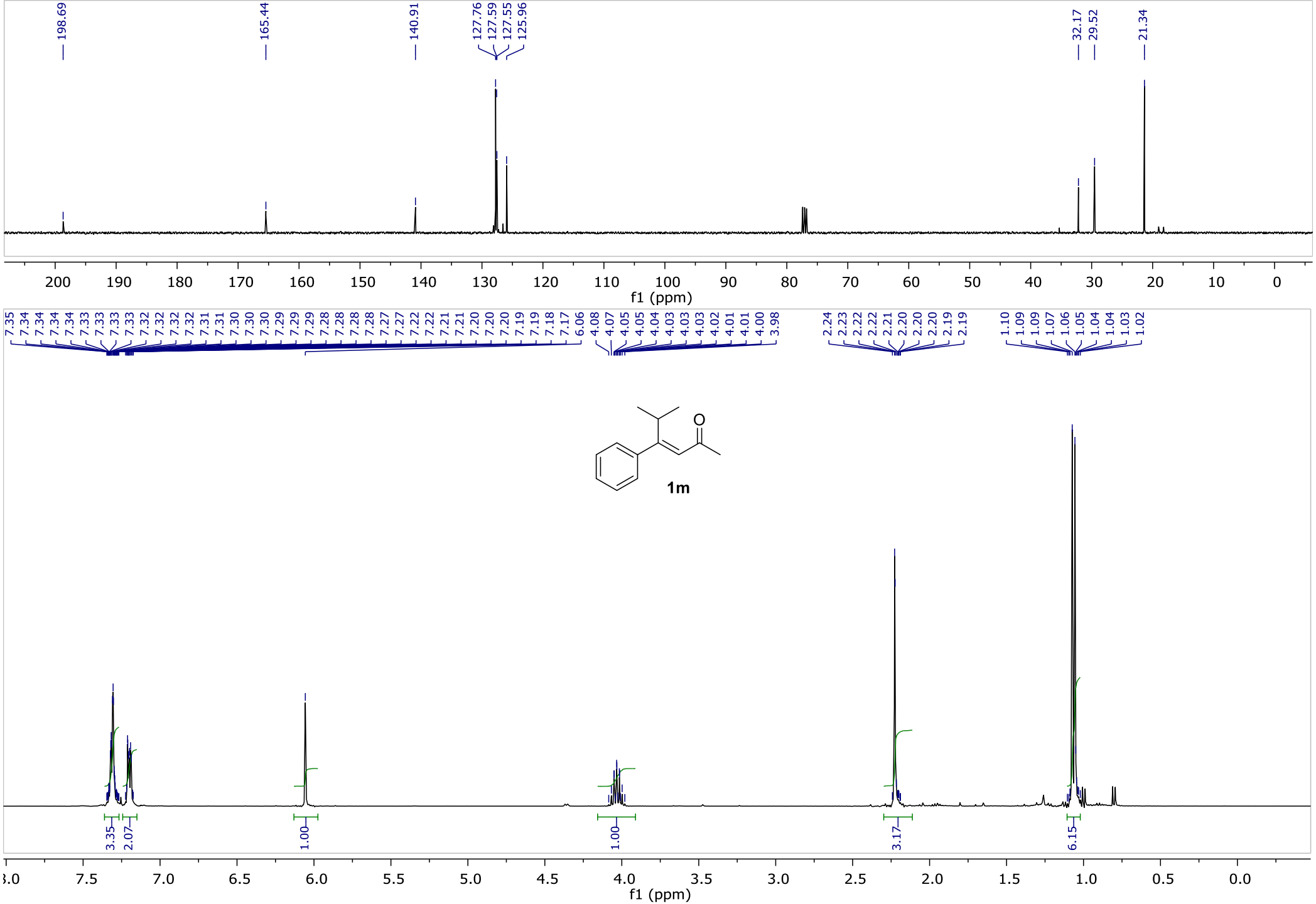
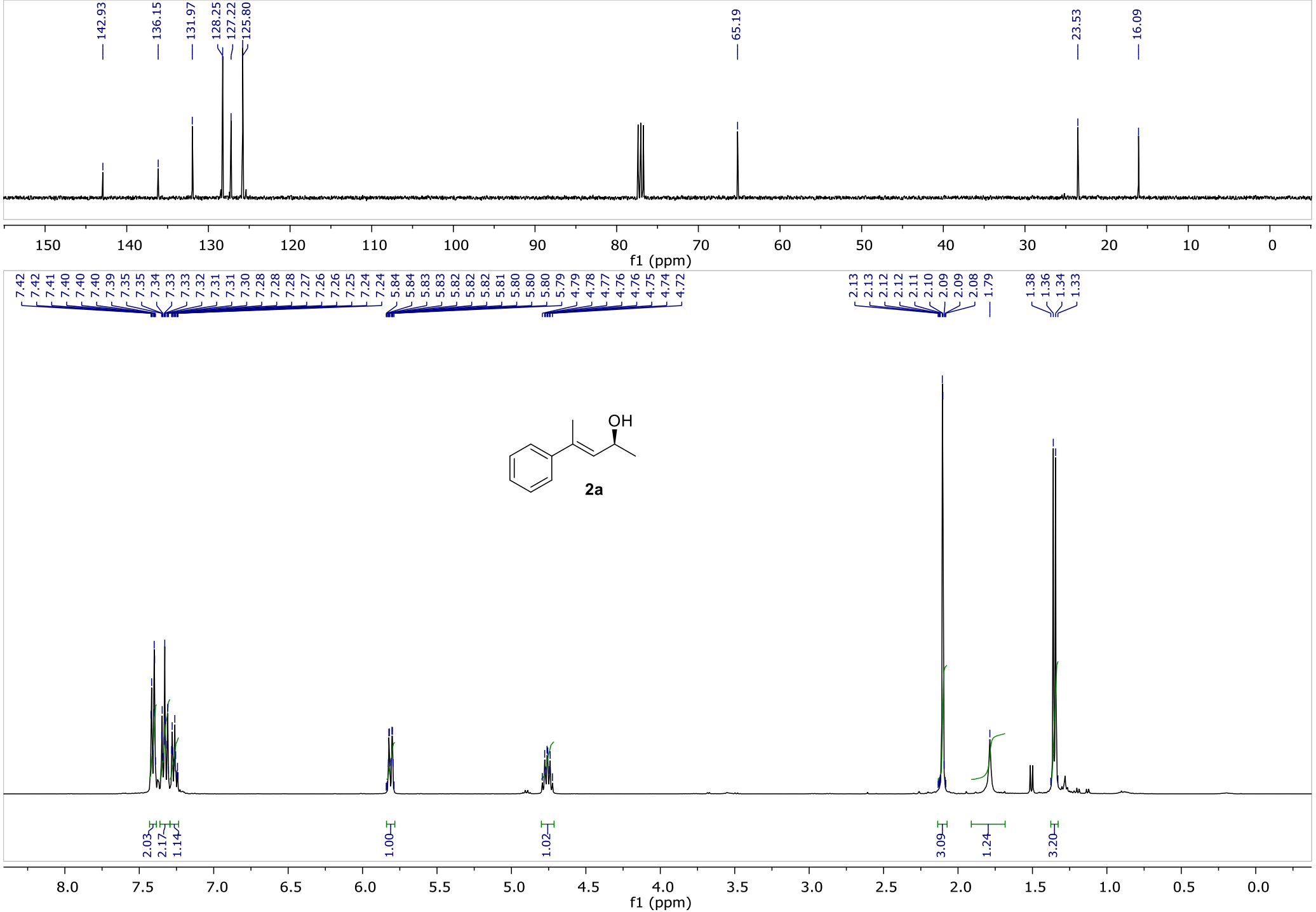
128.53
128.19
127.77
126.32
126.01
125.32
125.24
124.24
32.09
22.07
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

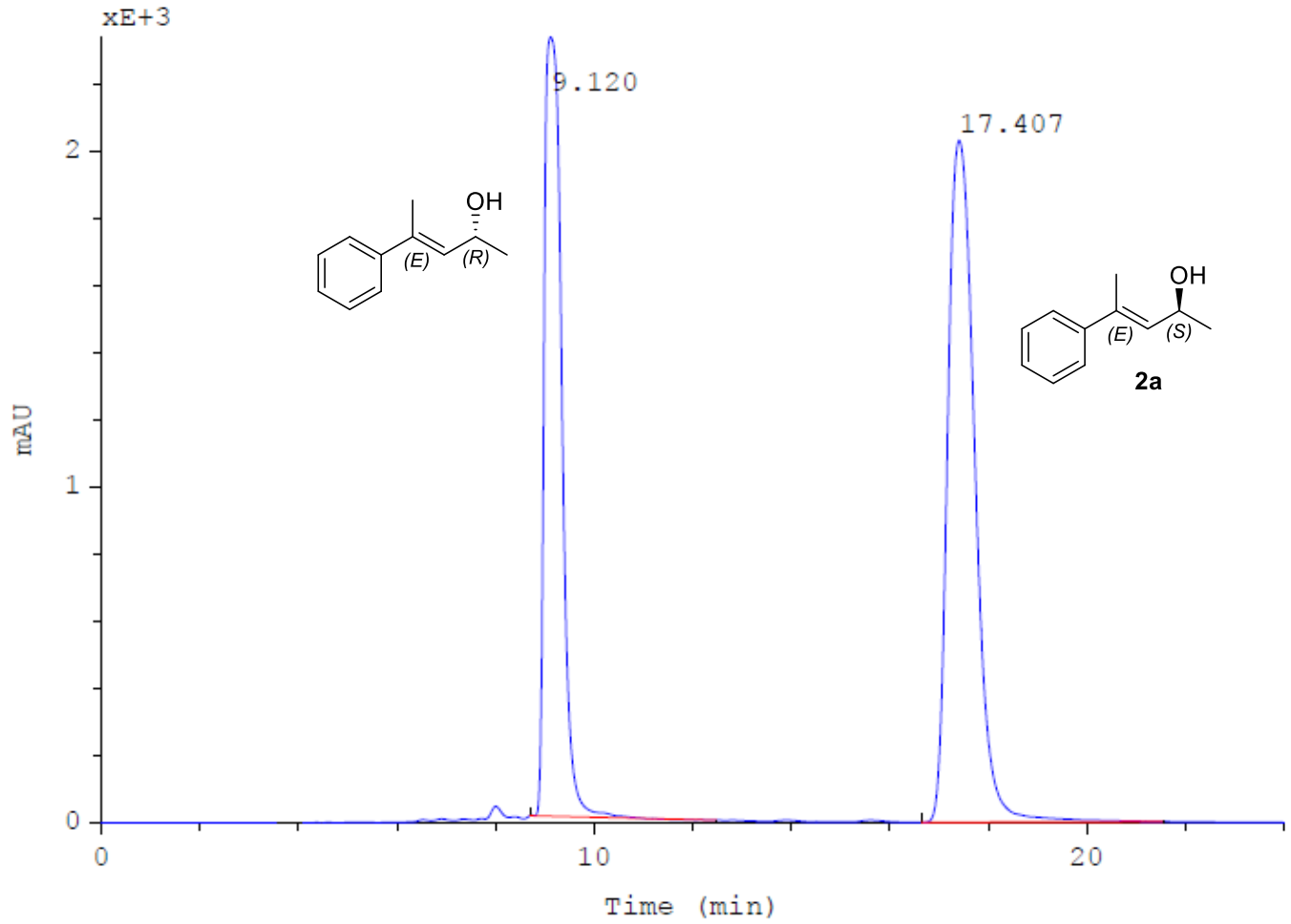

7.91 7.91 7.91 7.90 7.90 7.90 7.90 7.89 7.89 7.88 7.88 7.87 7.86 7.82 7.80 7.52 7.51 7.51 7.50 7.49 7.48 7.47 7.45 7.45 7.43 7.30 7.30 7.29 7.28 6.38 6.38
2.63 2.62 2.62
2.30
O
1l
2.10 1.02 2.09 1.09 1.01 1.00 3.12 3.13
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


198.69
165.44
140.91
127.76
127.59
127.55
125.96
32.17
29.52
21.34
f1 (ppm)
O
1m
3.35
2.07
1.00
1.00
3.17
6.15
f1 (ppm)


142.93
136.15
131.97
128.25
127.22
125.80
65.19
23.53
16.09
f1 (ppm)
OH
2a
2.03
2.17
1.14
1.00
1.02
3.09
1.24
3.20
f1 (ppm)

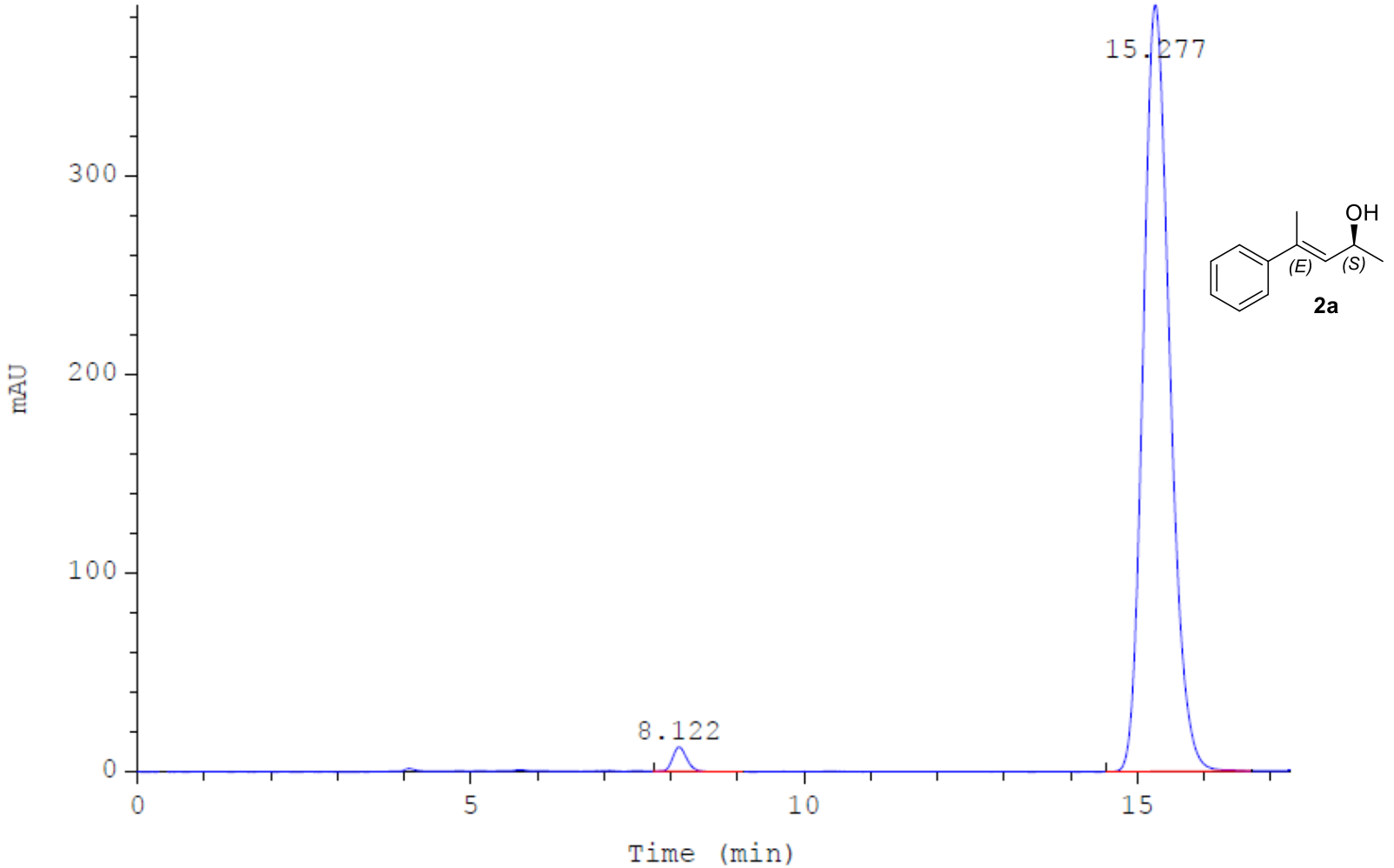
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	9.120	8.70	12.49	952.691	2320.28	42.3654	0.384
2	17.407	16.66	21.56	1296.06	2031.35	57.6347	0.589

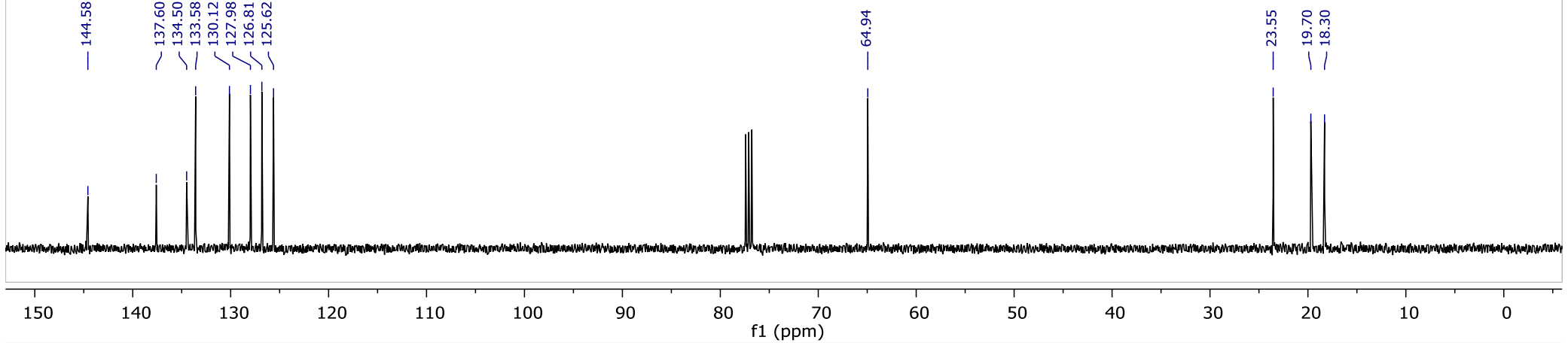


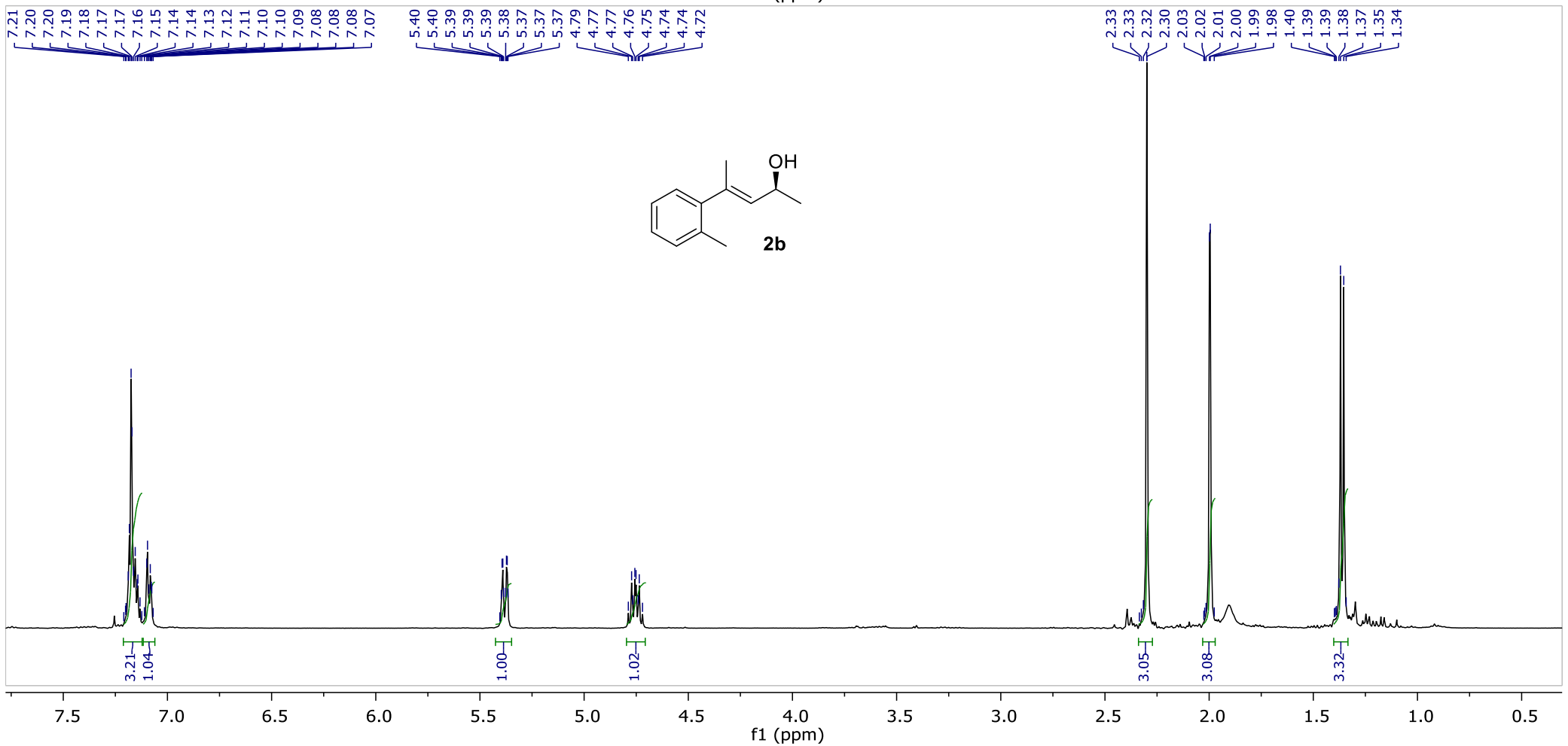
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	8.122	7.74	9.08	2.92562	12.2291	1.6073	0.223
2	15.277	14.54	16.71	179.098	385.911	98.3927	0.428



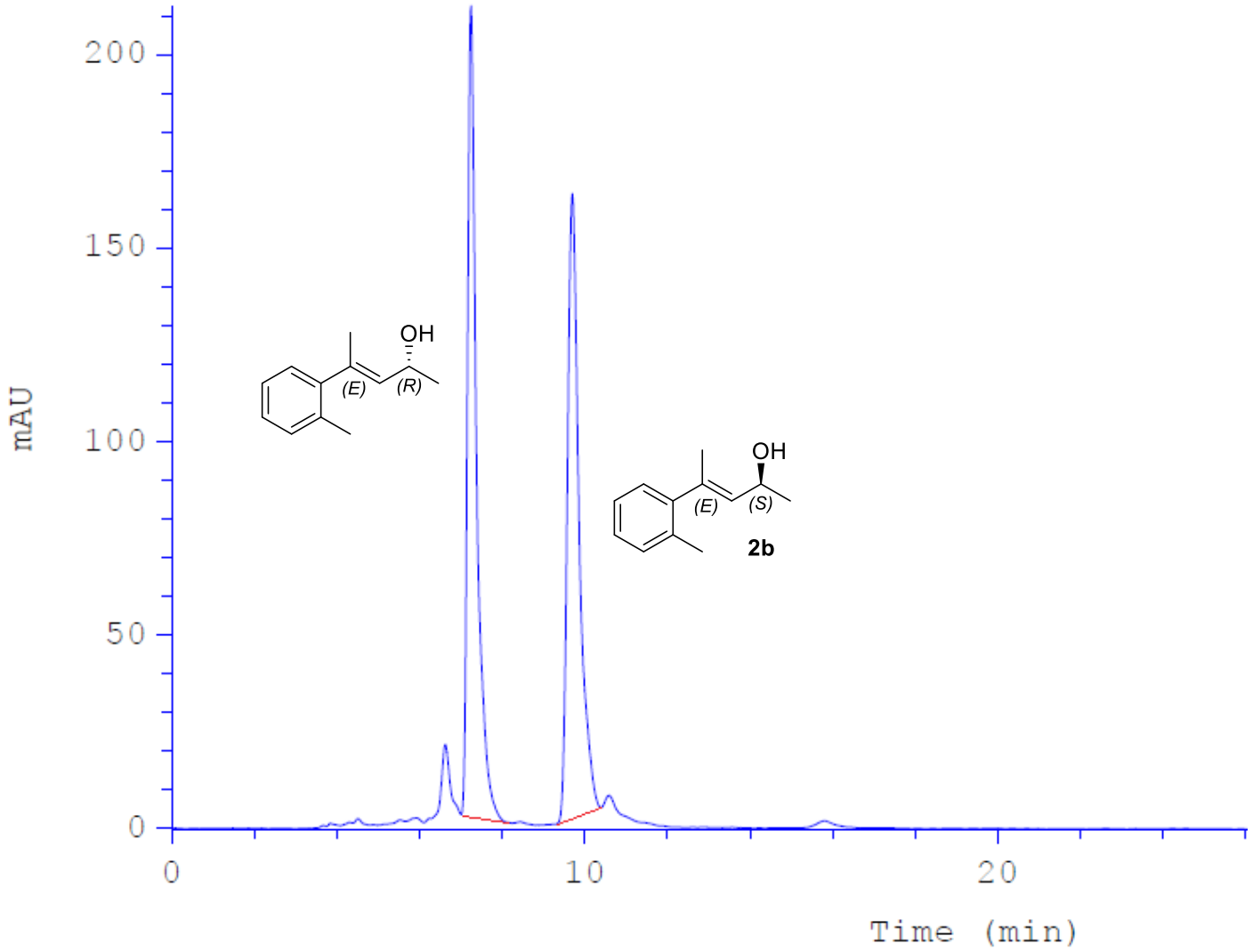
2a


144.58
137.60
134.50
133.58
130.12
127.98
126.81
125.62
64.94
23.55
19.70
18.30
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.21
7.20
7.20
7.19
7.18
7.17
7.17
7.16
7.15
7.14
7.14
7.13
7.12
7.11
7.10
7.10
7.09
7.08
7.08
7.08
7.07
5.40
5.40
5.39
5.39
5.39
5.38
5.37
5.37
5.37
4.79
4.77
4.77
4.76
4.75
4.74
4.74
4.72
2.33
2.33
2.32
2.30
2.03
2.02
2.01
2.00
1.99
1.98
1.40
1.39
1.39
1.38
1.37
1.35
1.34
OH
2b
3.21
1.04
1.00
1.02
3.05
3.08
3.32
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
f1 (ppm)

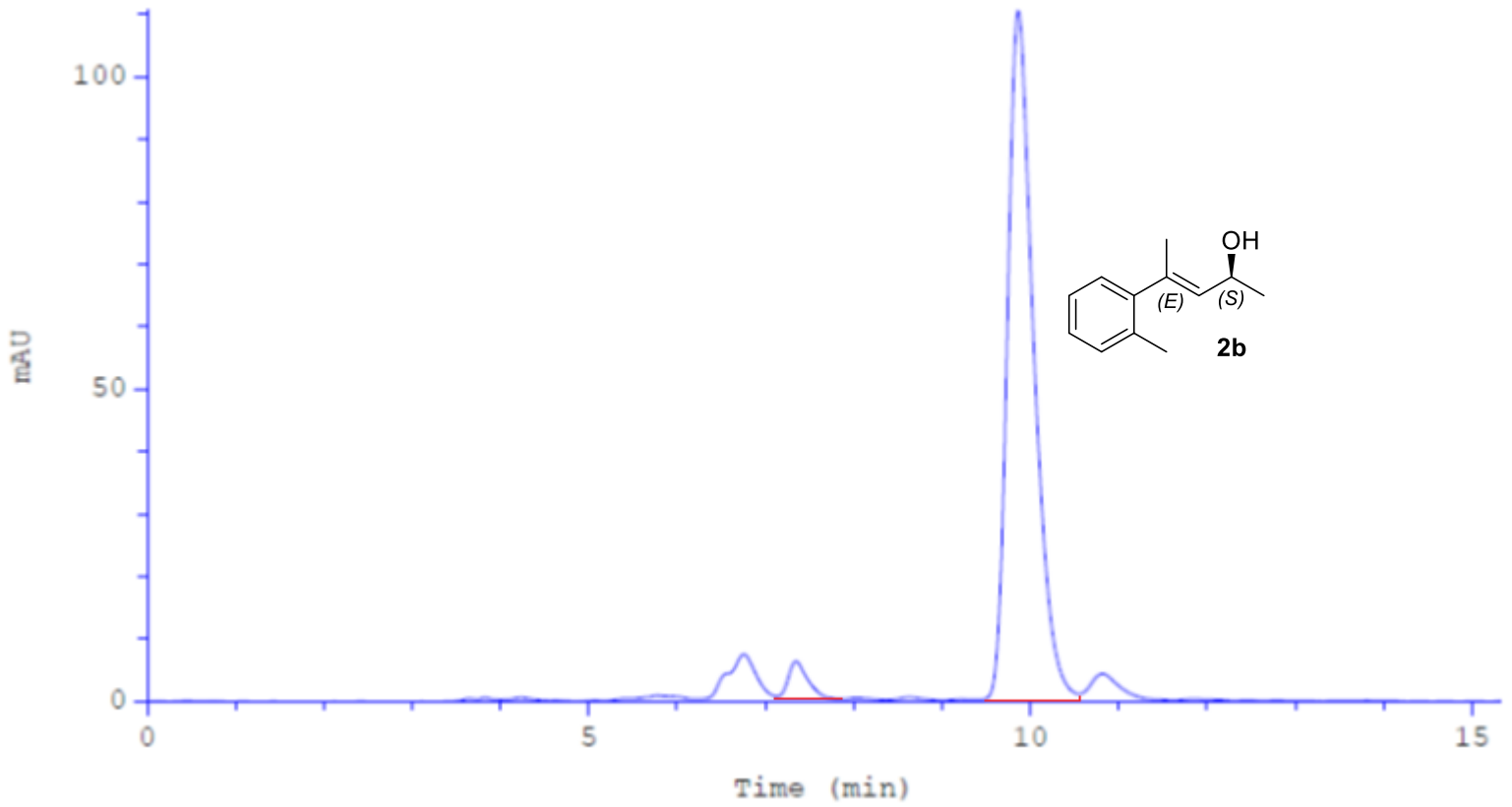
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

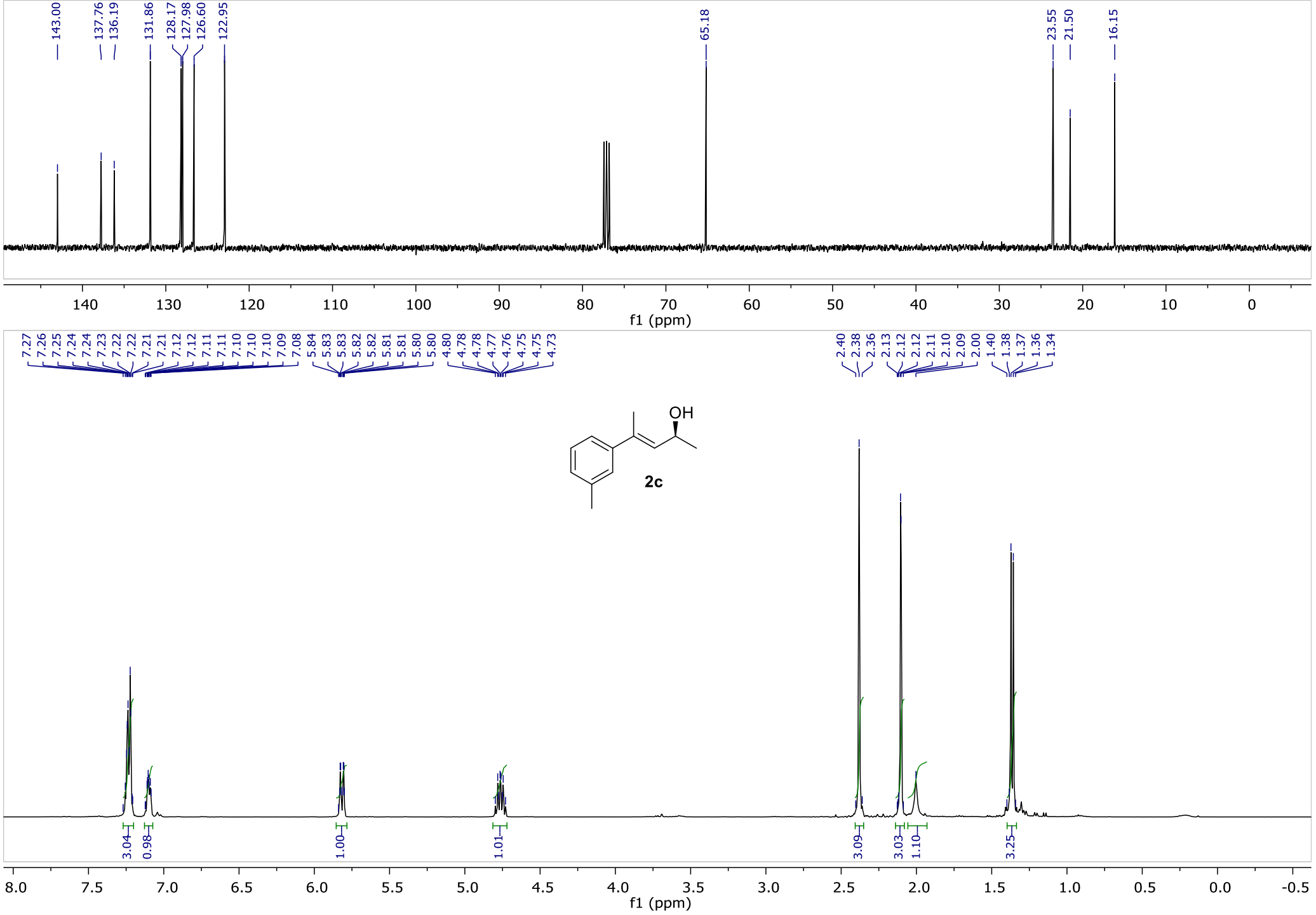
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	7.251	7.02	8.58	53.3967	209.441	50.3355	0.212
2	9.701	9.30	10.38	52.6849	161.482	49.6645	0.280



Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

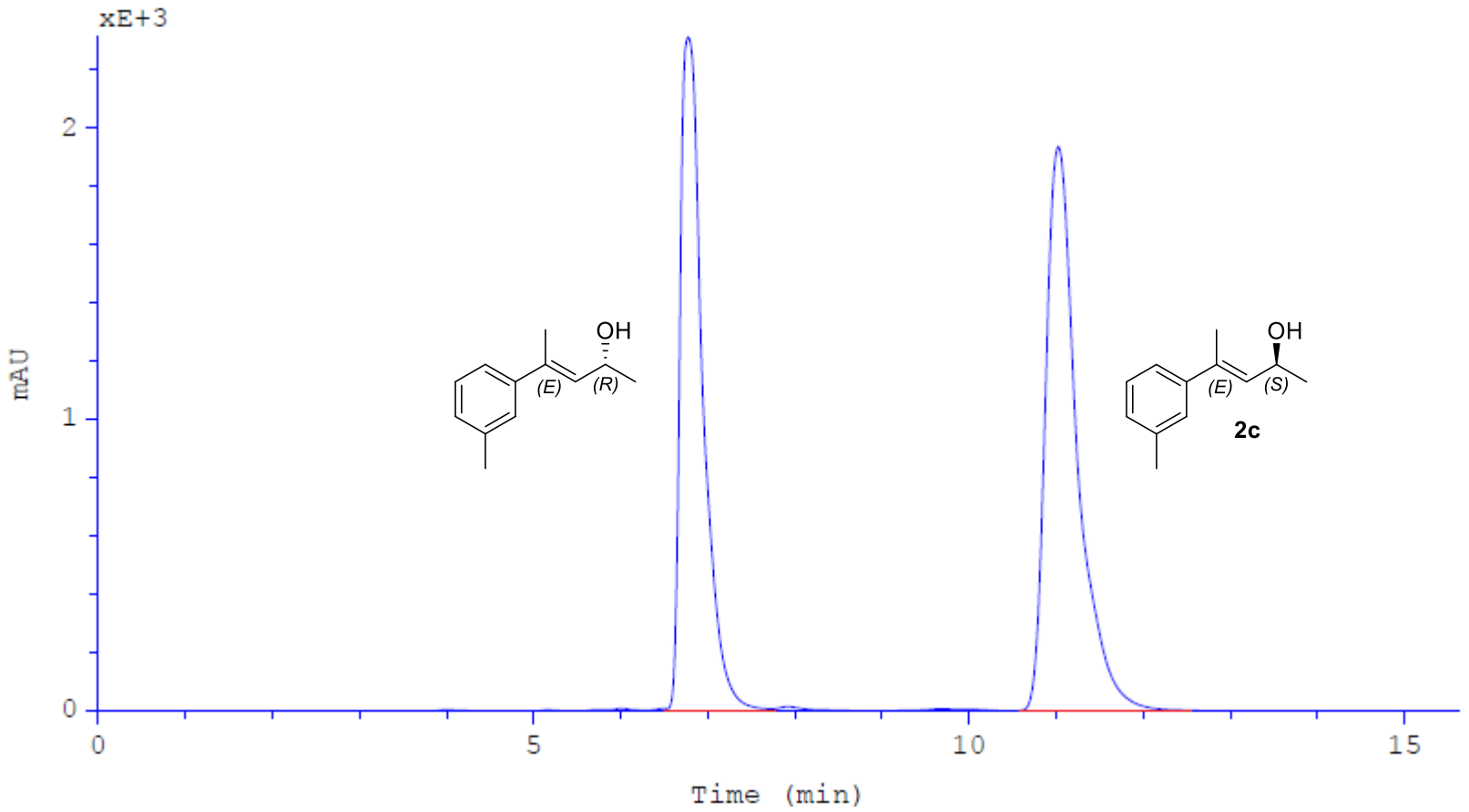
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	7.356	7.12	7.69	1.34611	5.65232	3.4950	0.221
2	9.858	9.50	10.61	37.169	109.737	96.5050	0.311




143.00
137.76
136.19
131.86
128.17
127.98
126.60
122.95
65.18
23.55
21.50
16.15
f1 (ppm)
OH
2c
3.04
0.98
1.00
1.01
3.09
3.03
1.10
3.25
f1 (ppm)

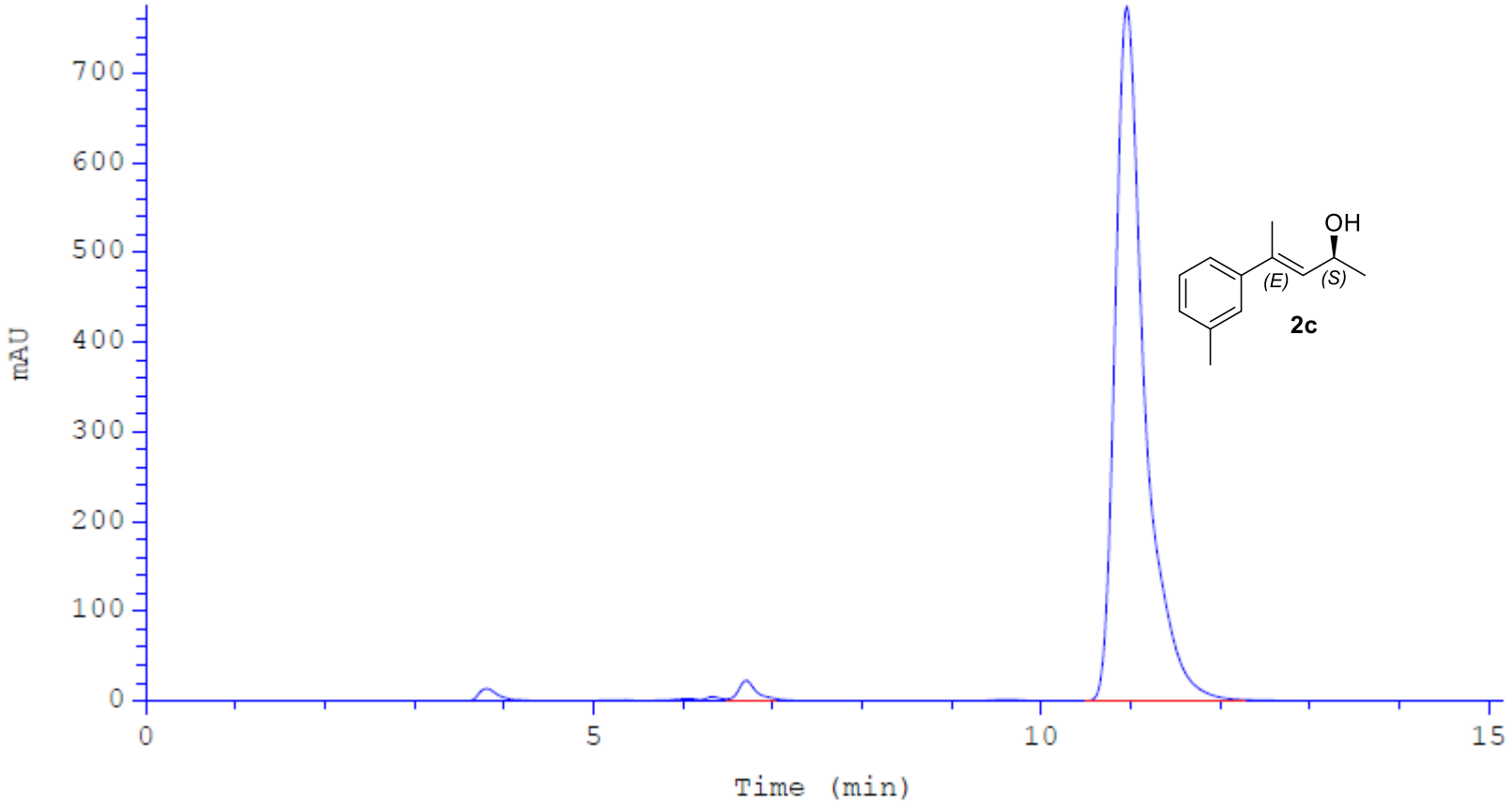
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

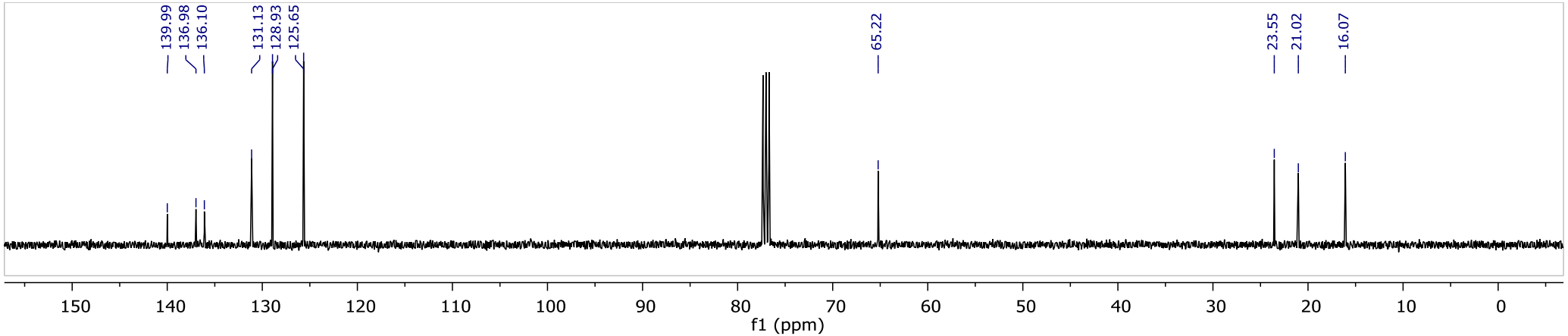
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	6.772	6.52	7.58	688.581	2304.35	45.8604	0.259
2	11.025	10.62	12.47	812.891	1935.07	54.1396	0.356

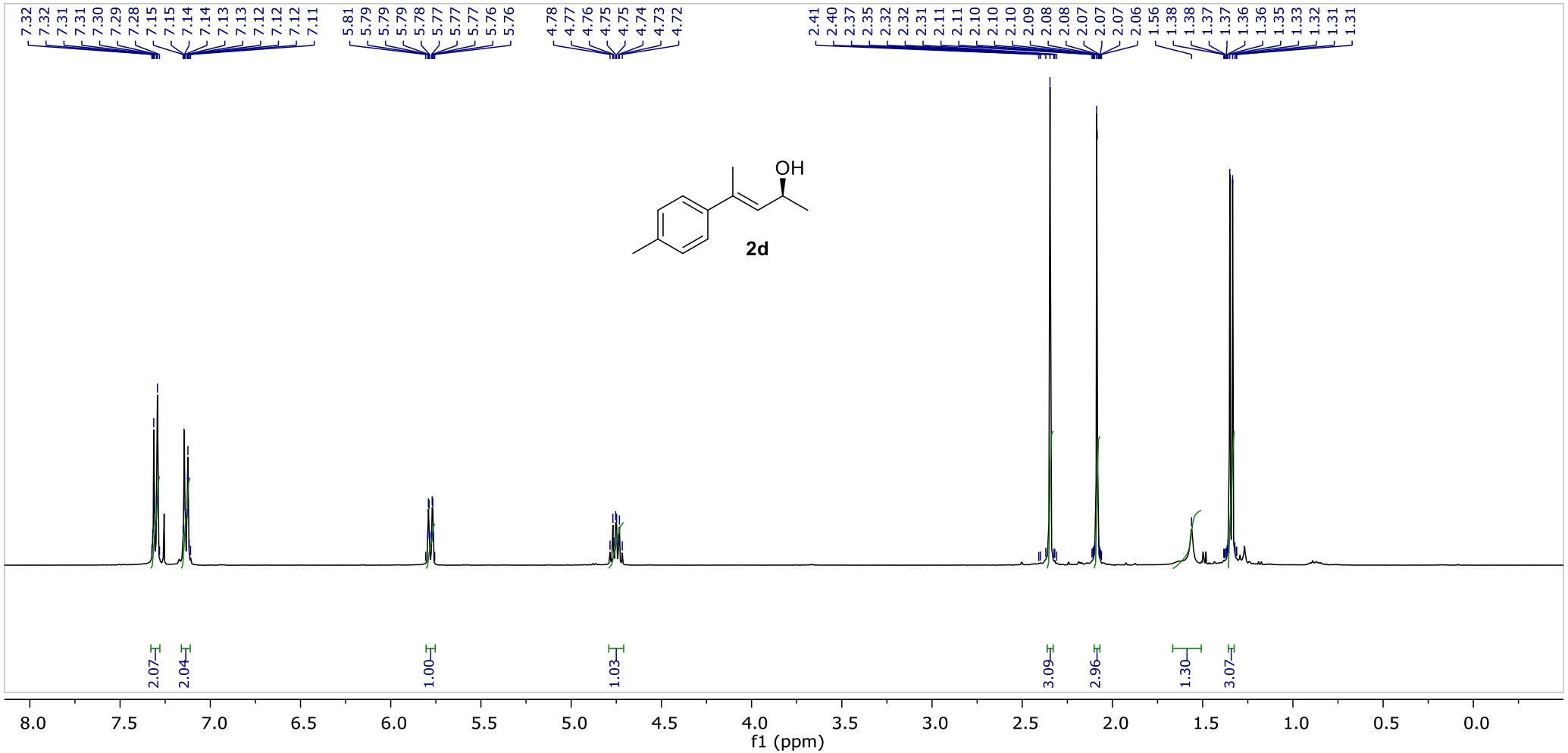


Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	6.706	6.56	7.08	3.84529	19.5029	1.2935	0.174
2	10.959	10.55	12.20	293.425	773.467	98.7065	0.316

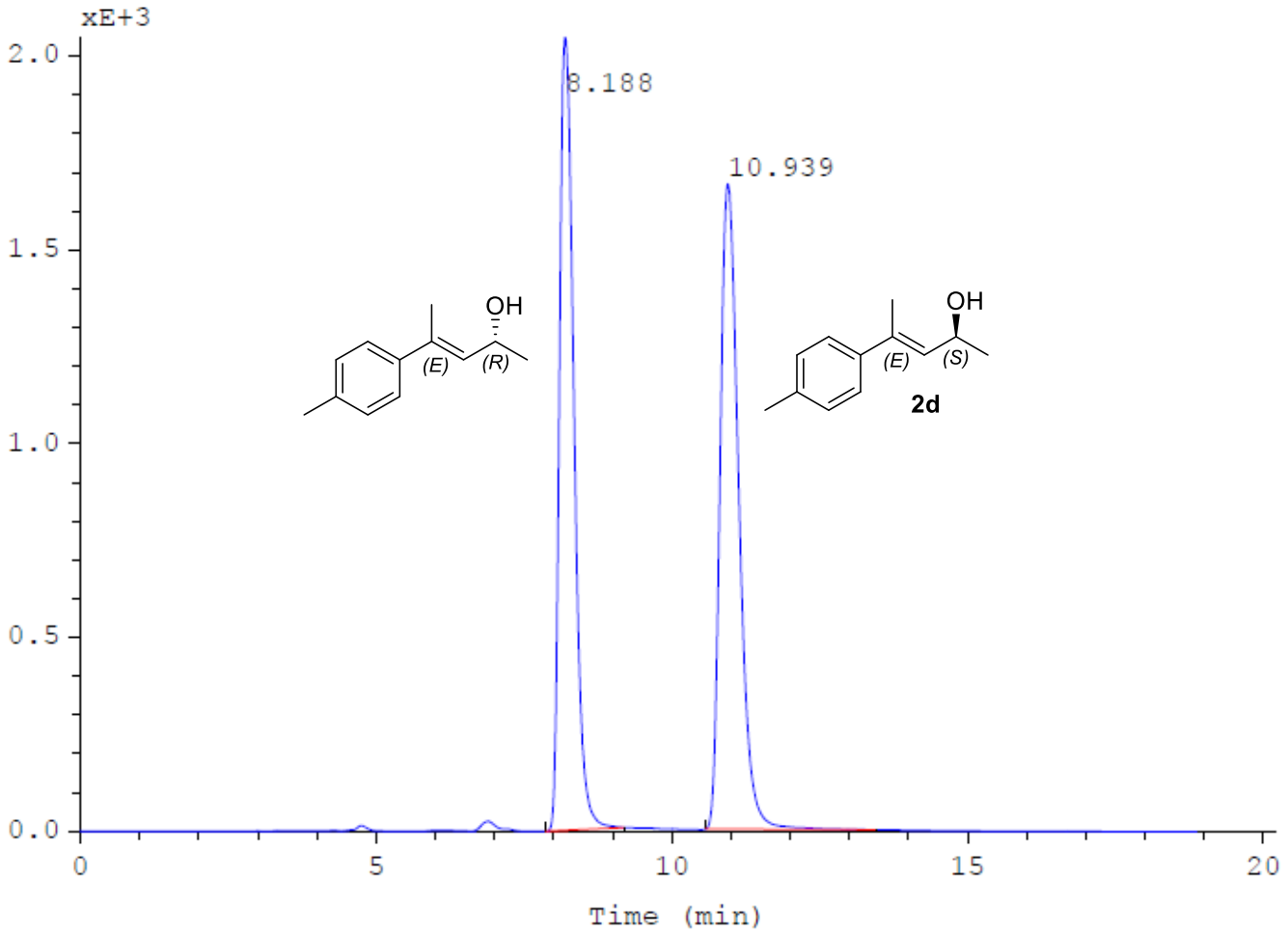



139.99
136.98
136.10
131.13
128.93
125.65
65.22
23.55
21.02
16.07
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.32
7.32
7.31
7.31
7.30
7.29
7.28
7.15
7.15
7.14
7.14
7.13
7.13
7.12
7.12
7.12
7.11
5.81
5.79
5.79
5.79
5.78
5.77
5.77
5.77
5.76
5.76
4.78
4.77
4.76
4.75
4.75
4.74
4.73
4.72
2.41
2.40
2.37
2.35
2.32
2.32
2.31
2.11
2.11
2.10
2.10
2.10
2.09
2.08
2.08
2.07
2.07
2.07
2.06
1.56
1.38
1.38
1.37
1.37
1.36
1.36
1.35
1.33
1.32
1.31
1.31
OH
2d
2.07
2.04
1.00
1.03
3.09
2.96
1.30
3.07
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
f1 (ppm)

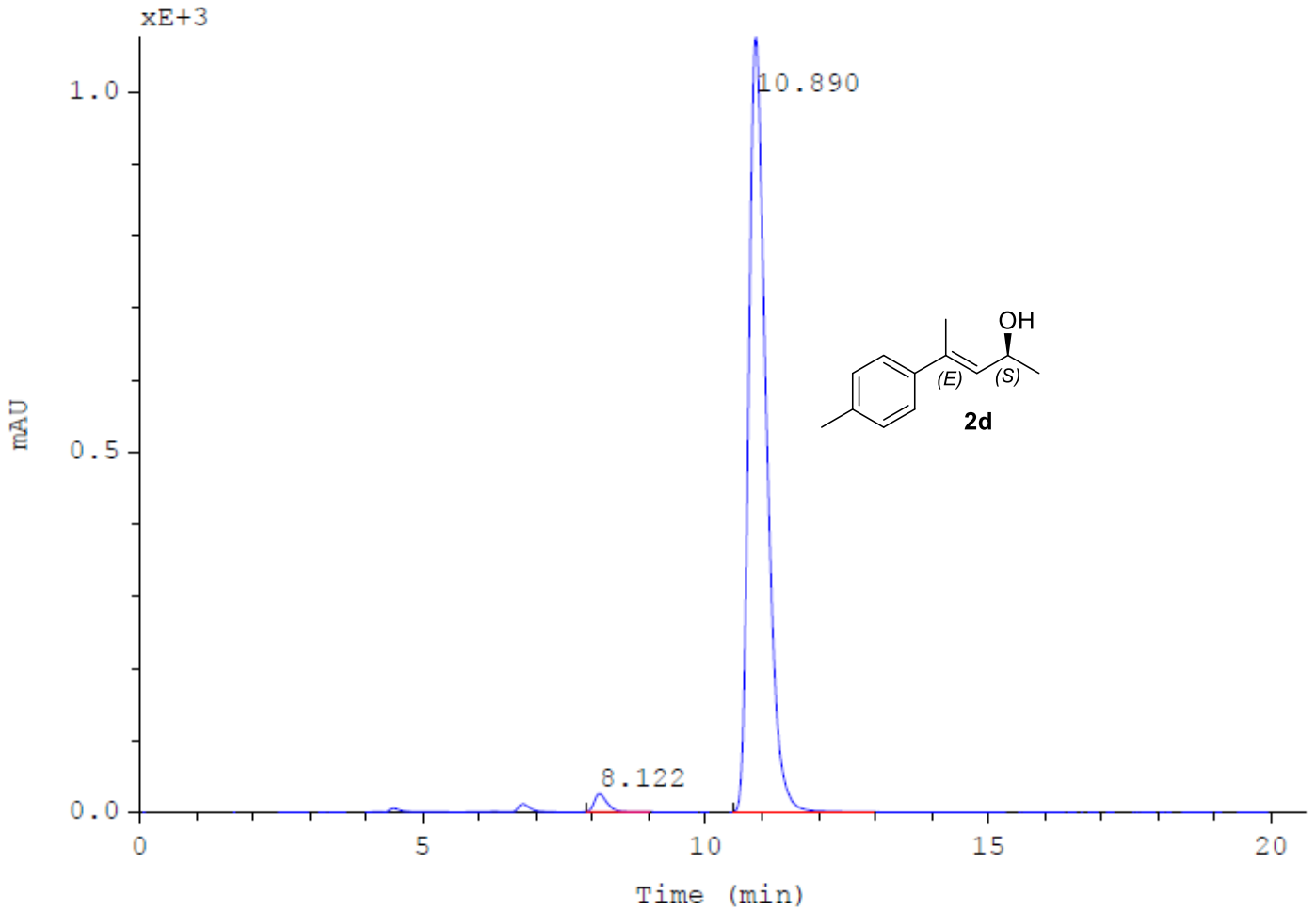
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

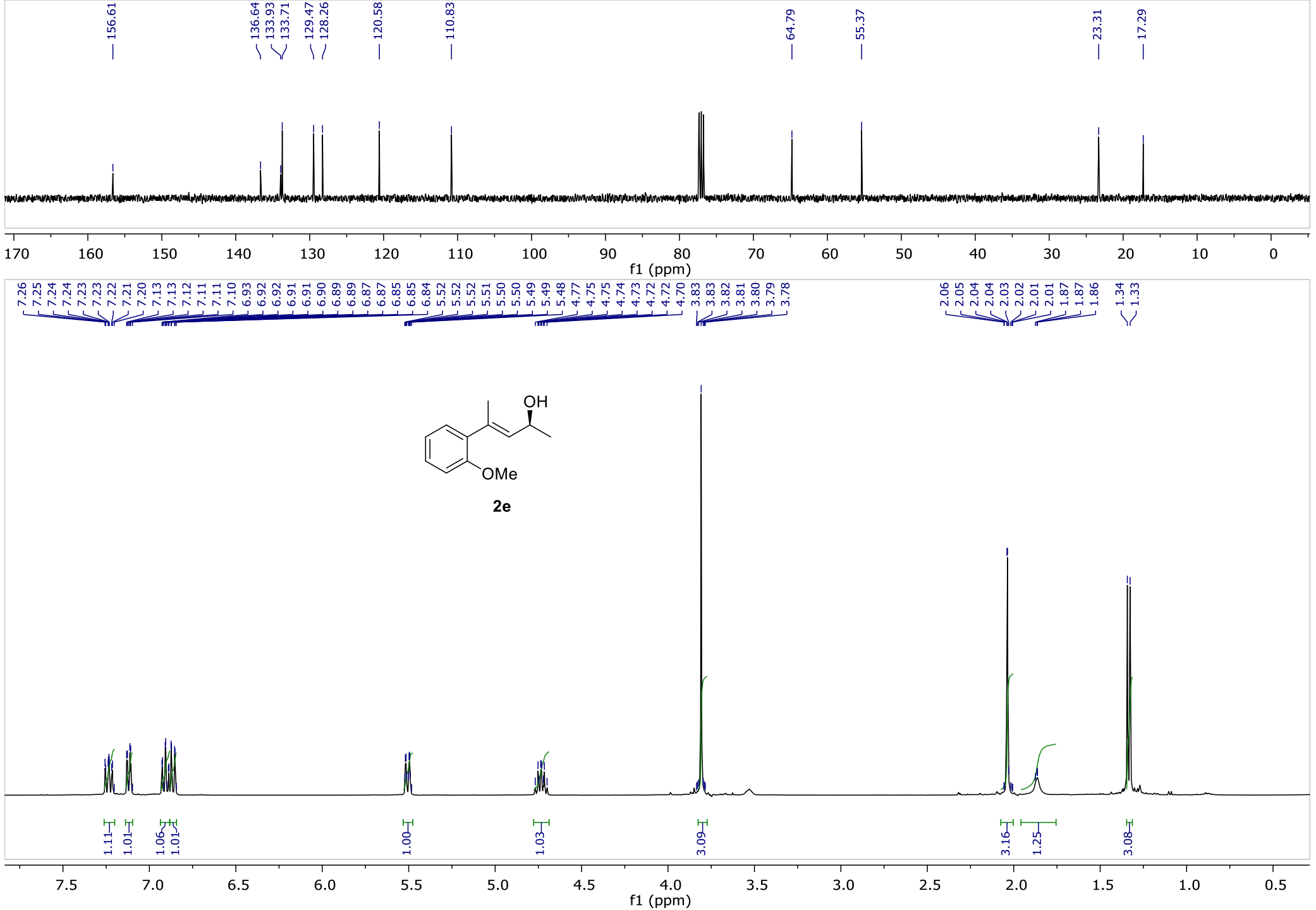
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	8.188	7.88	9.19	580.425	2046.28	48.7404	0.262
2	10.939	10.58	13.46	610.425	1665.72	51.2596	0.336



Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

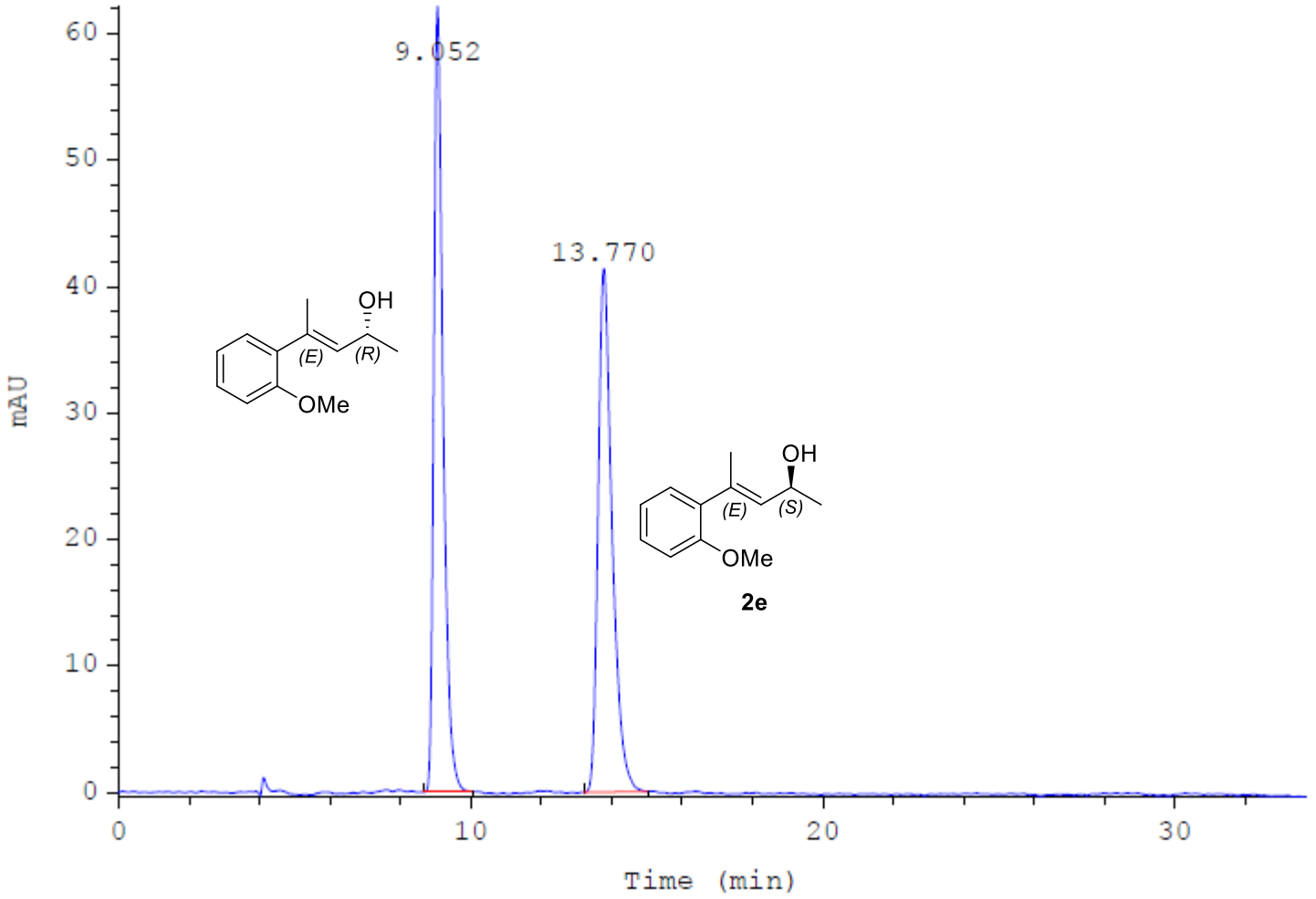
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	8.122	7.90	9.03	6.5009	24.9986	1.6570	0.238
2	10.890	10.49	12.99	385.818	1076.24	98.3430	0.326




156.61
136.64
133.93
133.71
129.47
128.26
120.58
110.83
64.79
55.37
23.31
17.29
f1 (ppm)
OH
OMe
2e
f1 (ppm)

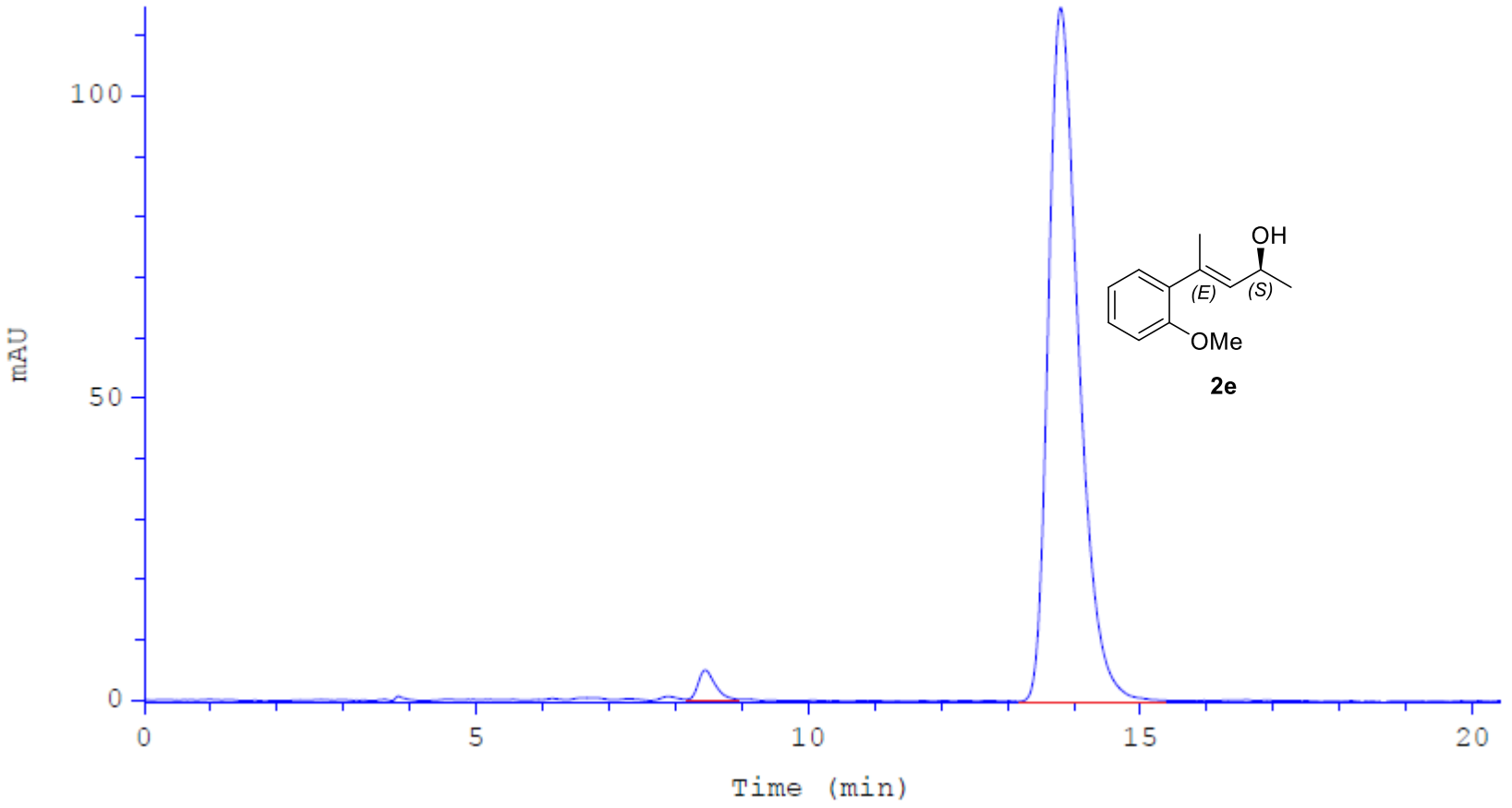
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

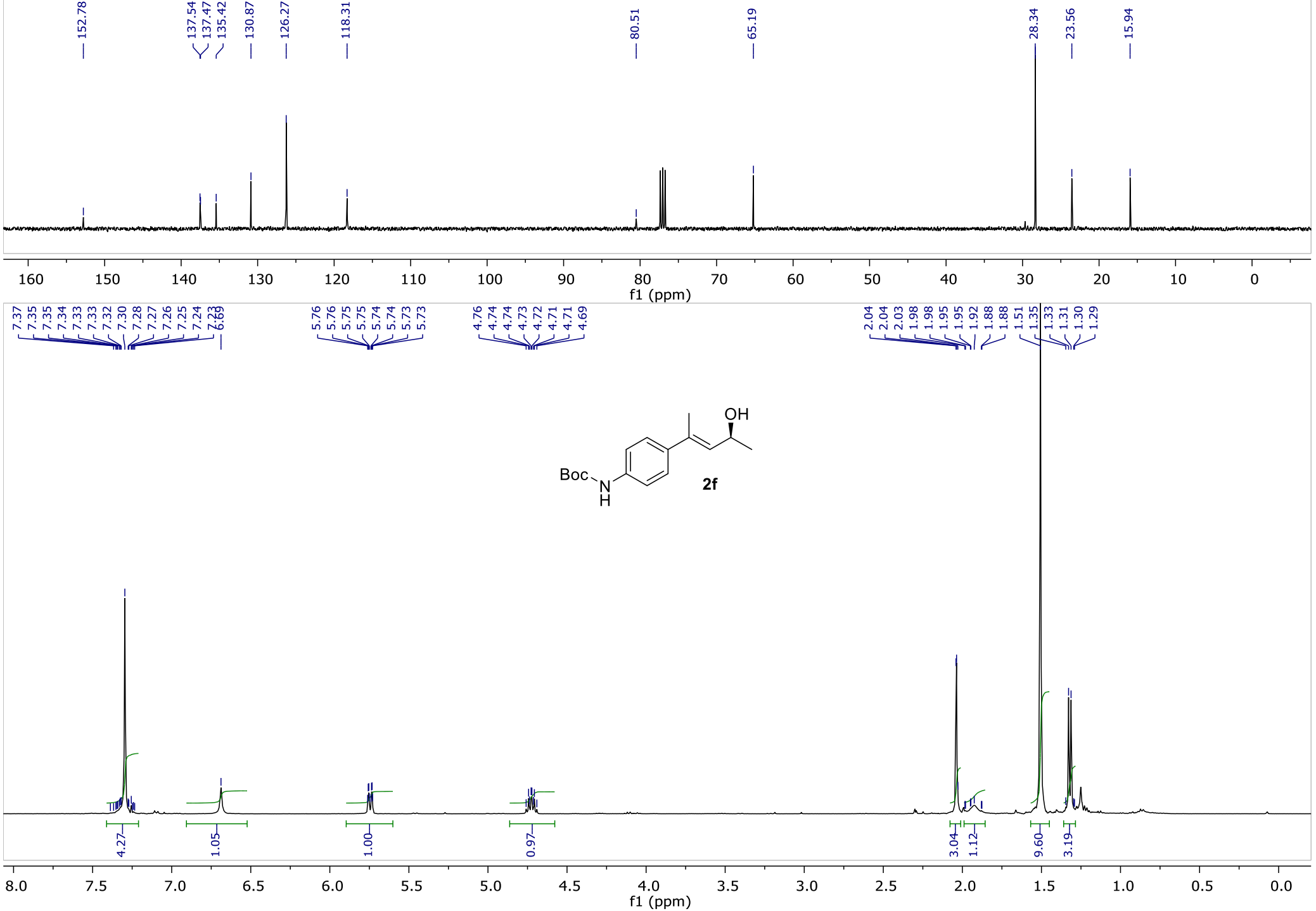
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	9.052	8.67	10.07	19.0154	62.1575	49.8933	0.270
2	13.770	13.22	15.02	19.0967	41.4038	50.1067	0.410



Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	8.455	8.21	8.80	1.24973	4.65458	2.0256	0.253
2	13.791	13.23	15.32	60.4457	114.758	97.9744	0.478




152.78
137.54
137.47
135.42
130.87
126.27
118.31
80.51
65.19
28.34
23.56
15.94
f1 (ppm)
7.37
7.35
7.35
7.34
7.33
7.33
7.32
7.30
7.28
7.27
7.26
7.25
7.24
7.23
6.69
5.76
5.76
5.75
5.75
5.74
5.74
5.73
5.73
4.76
4.74
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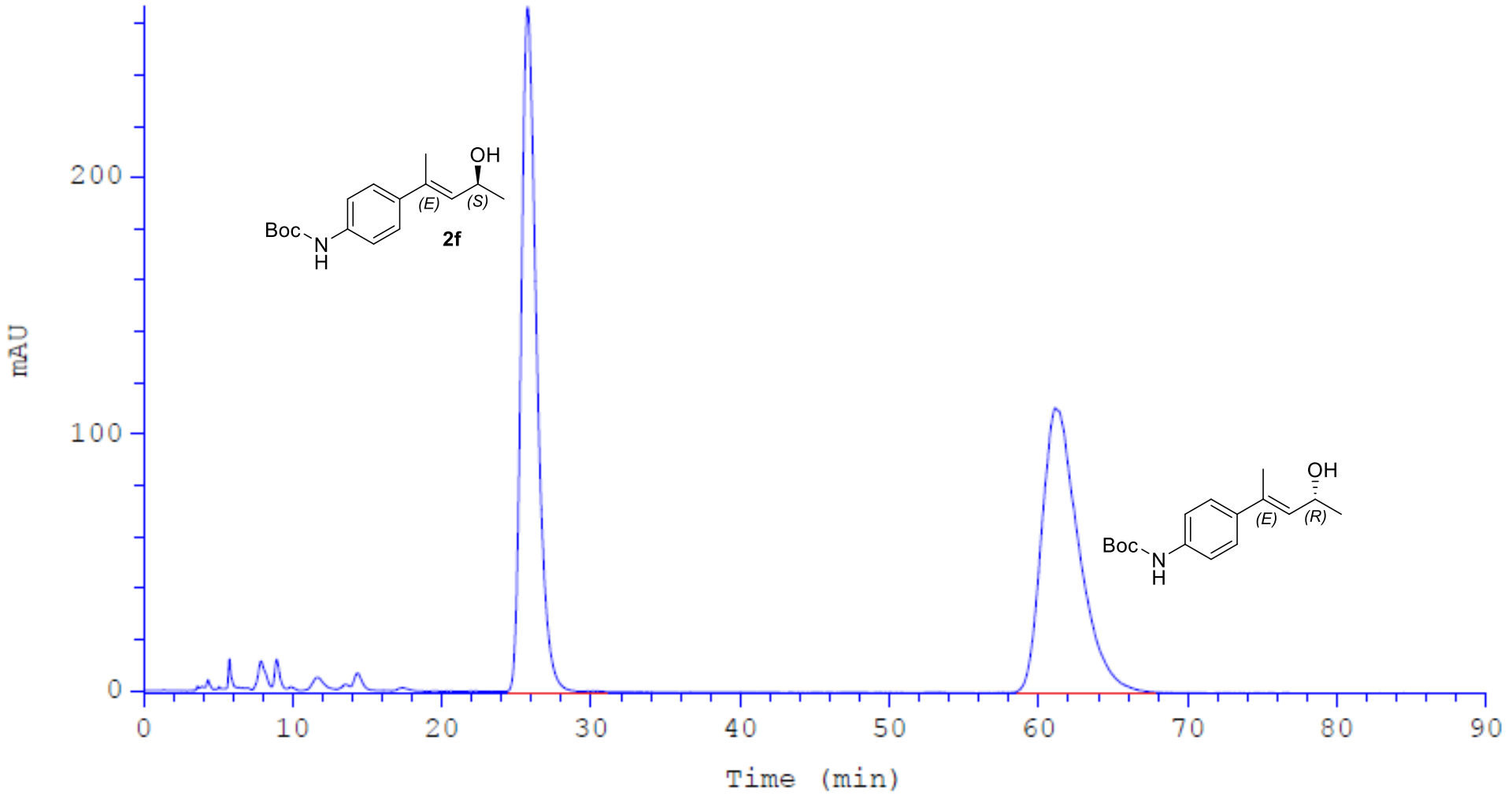
4.73
4.72
4.71
4.71
4.69
2.04
2.04
2.03
1.98
1.98
1.95
1.95
1.92
1.88
1.88
1.51
1.35
1.33
1.31
1.30
1.29
OH
Boc
N
H
2f
4.27
1.05
1.00
0.97
3.04
1.12
9.60
3.19
f1 (ppm)

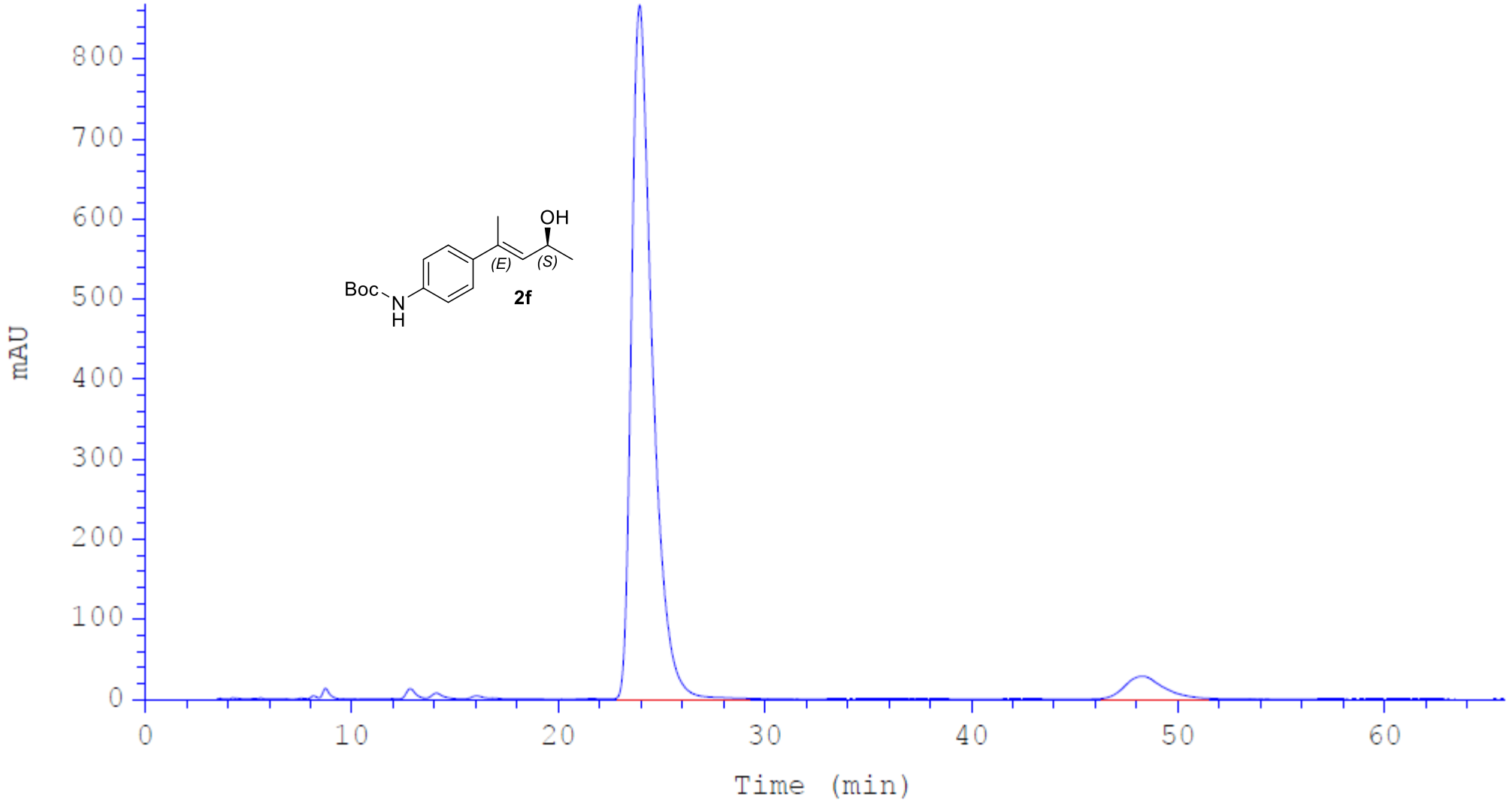
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

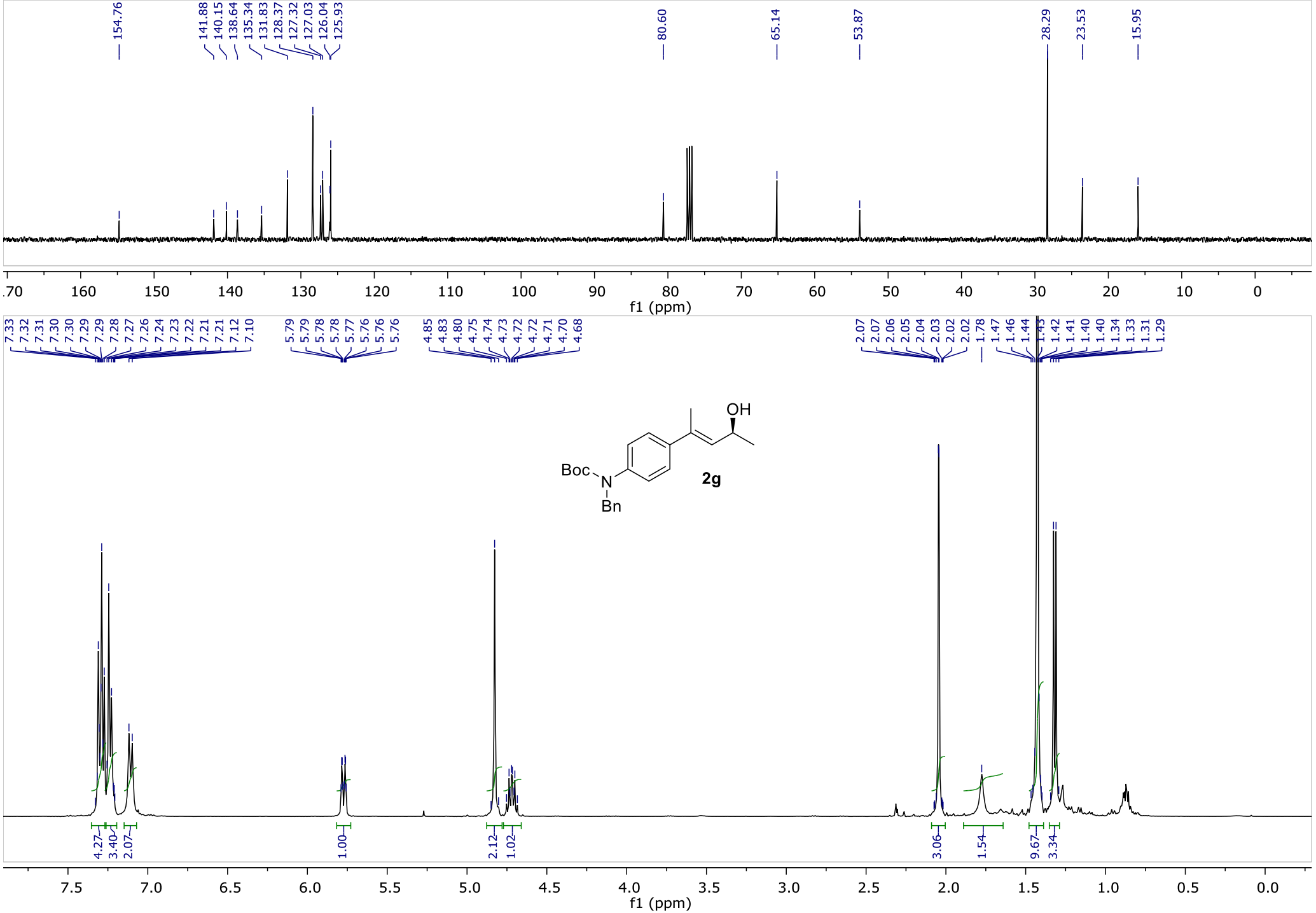
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	25.750	24.30	30.95	319.273	266.812	50.0495	1.082
2	61.100	58.40	67.56	318.641	110.728	49.9505	2.638



Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

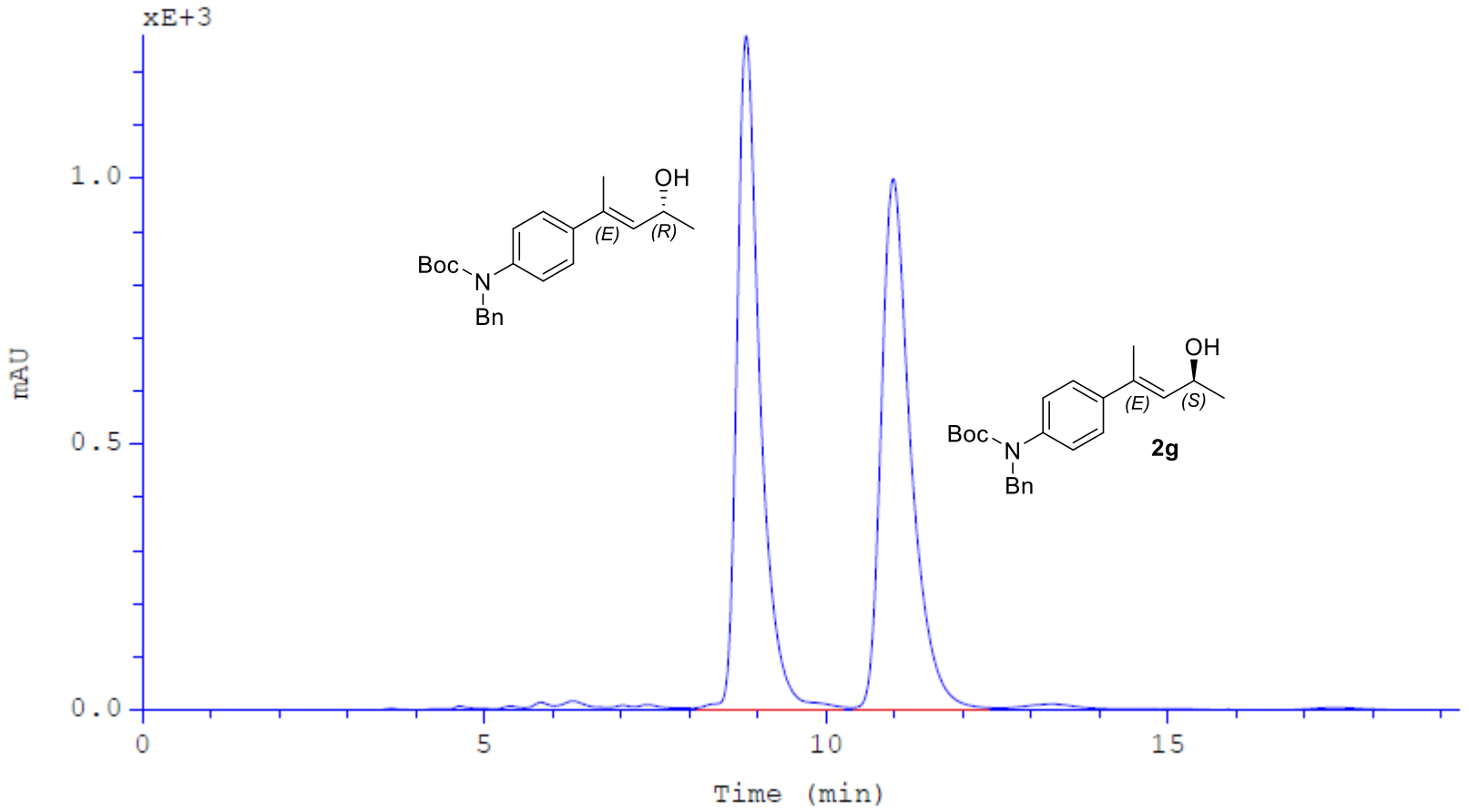
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	23.935	22.71	29.23	1012.84	866.758	94.5190	1.035
2	48.337	46.34	51.60	58.7327	27.7086	5.4810	1.938




154.76
141.88
140.15
138.64
135.34
131.83
128.37
127.32
127.03
126.04
125.93
80.60
65.14
53.87
28.29
23.53
15.95
f1 (ppm)
OH
Boc
N
Bn
2g
4.27
3.40
2.07
1.00
2.12
1.02
3.06
1.54
9.67
3.34
f1 (ppm)

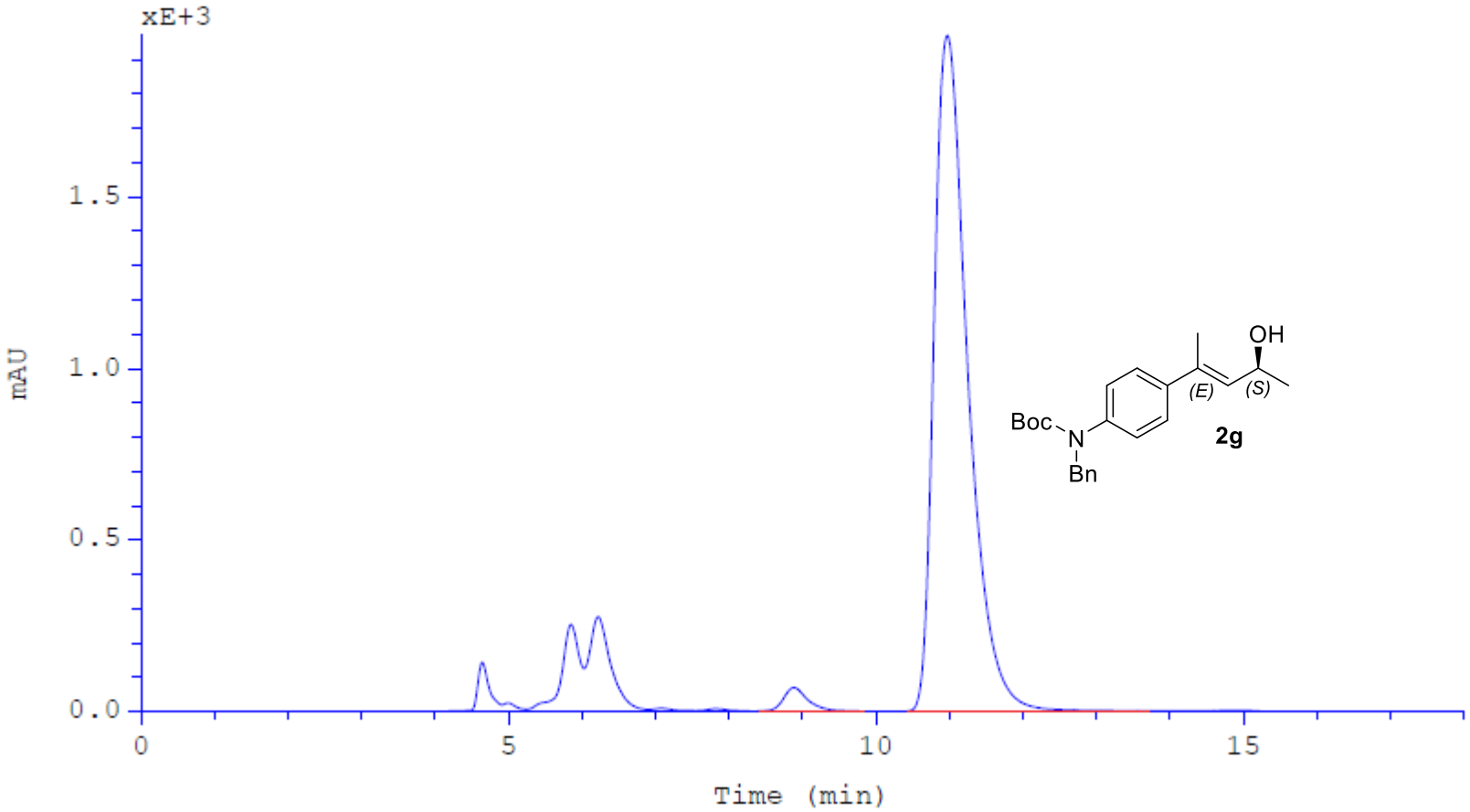
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

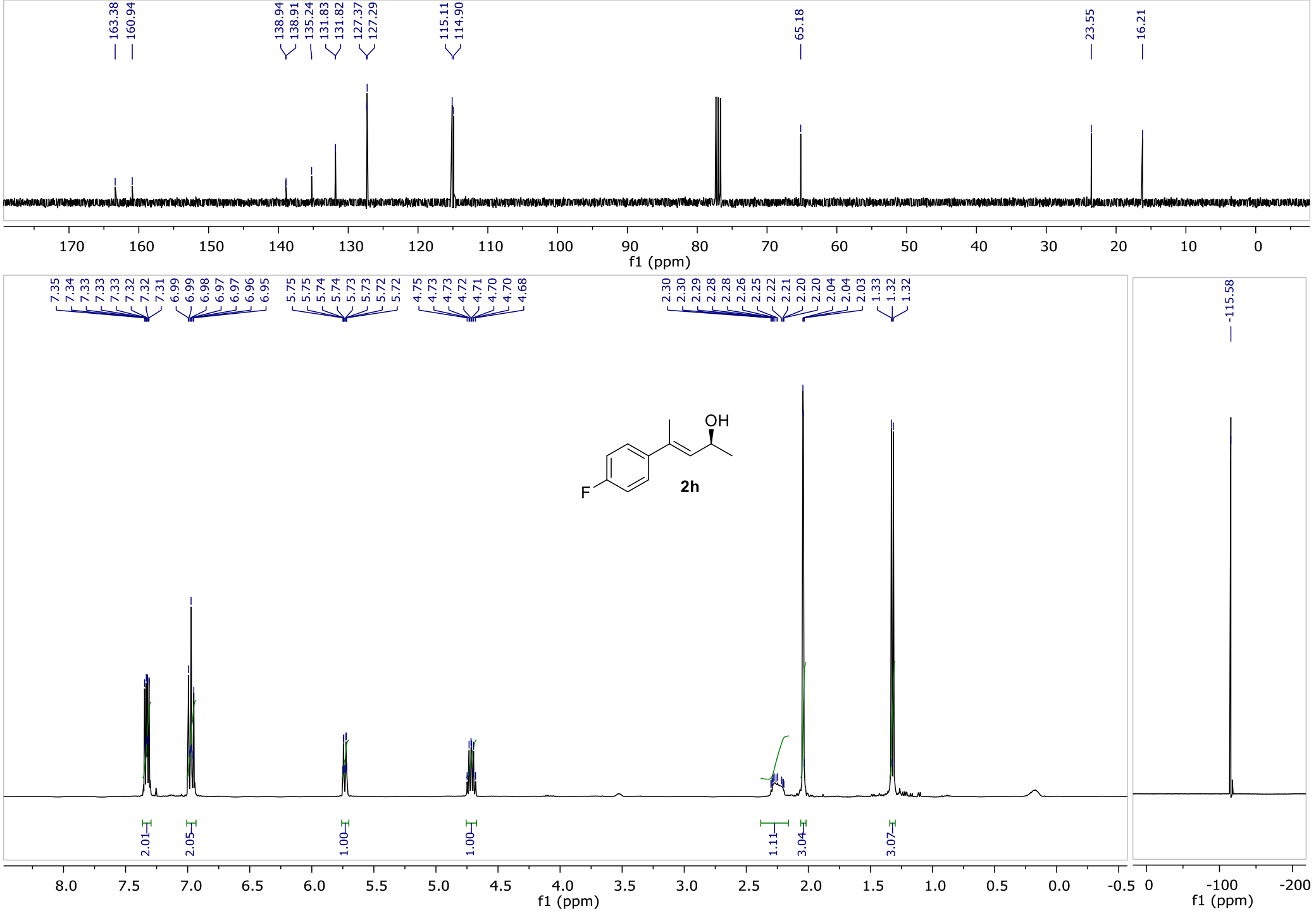
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	8.823	8.17	10.19	488.435	1261.15	49.8067	0.336
2	10.974	10.42	12.22	492.226	993.055	50.1933	0.440



Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	8.890	8.46	9.71	25.6102	68.1521	2.3848	0.331
2	10.975	10.42	13.89	1048.3	1969.63	97.6152	0.476




163.38
160.94
138.94
138.91
135.24
131.83
131.82
127.37
127.29
115.11
114.90
65.18
23.55
16.21
f1 (ppm)
7.35
7.34
7.33
7.33
7.33
7.32
7.32
7.31
6.99
6.99
6.98
6.97
6.97
6.96
6.95
5.75
5.75
5.74
5.74
5.73
5.73
5.72
5.72
4.75
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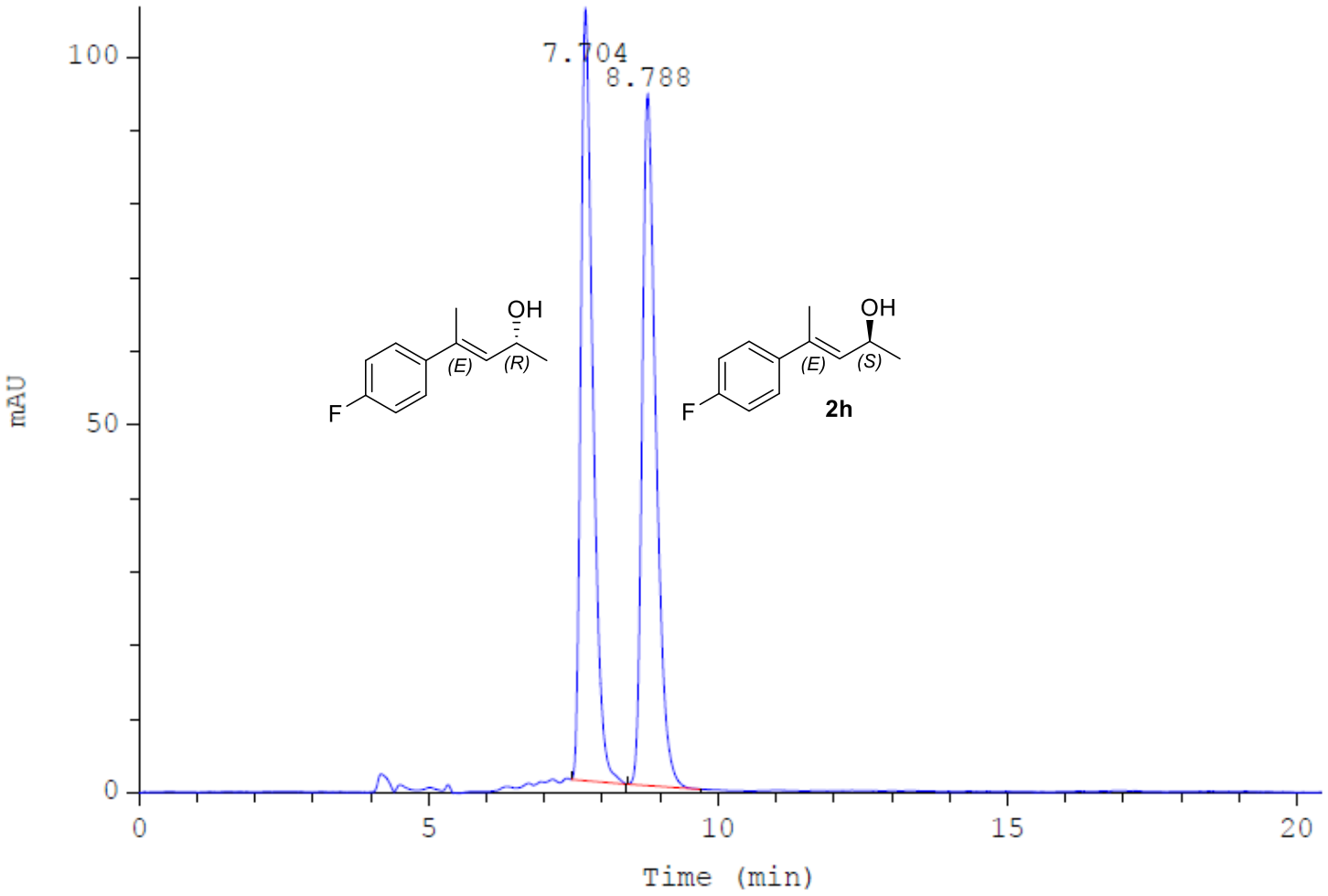
2.28
2.28
2.26
2.25
2.22
2.21
2.20
2.20
2.04
2.04
2.03
1.33
1.32
1.32
OH
F
2h
-115.58
2.01
2.05
1.00
1.00
1.11
3.04
3.07
f1 (ppm)
f1 (ppm)

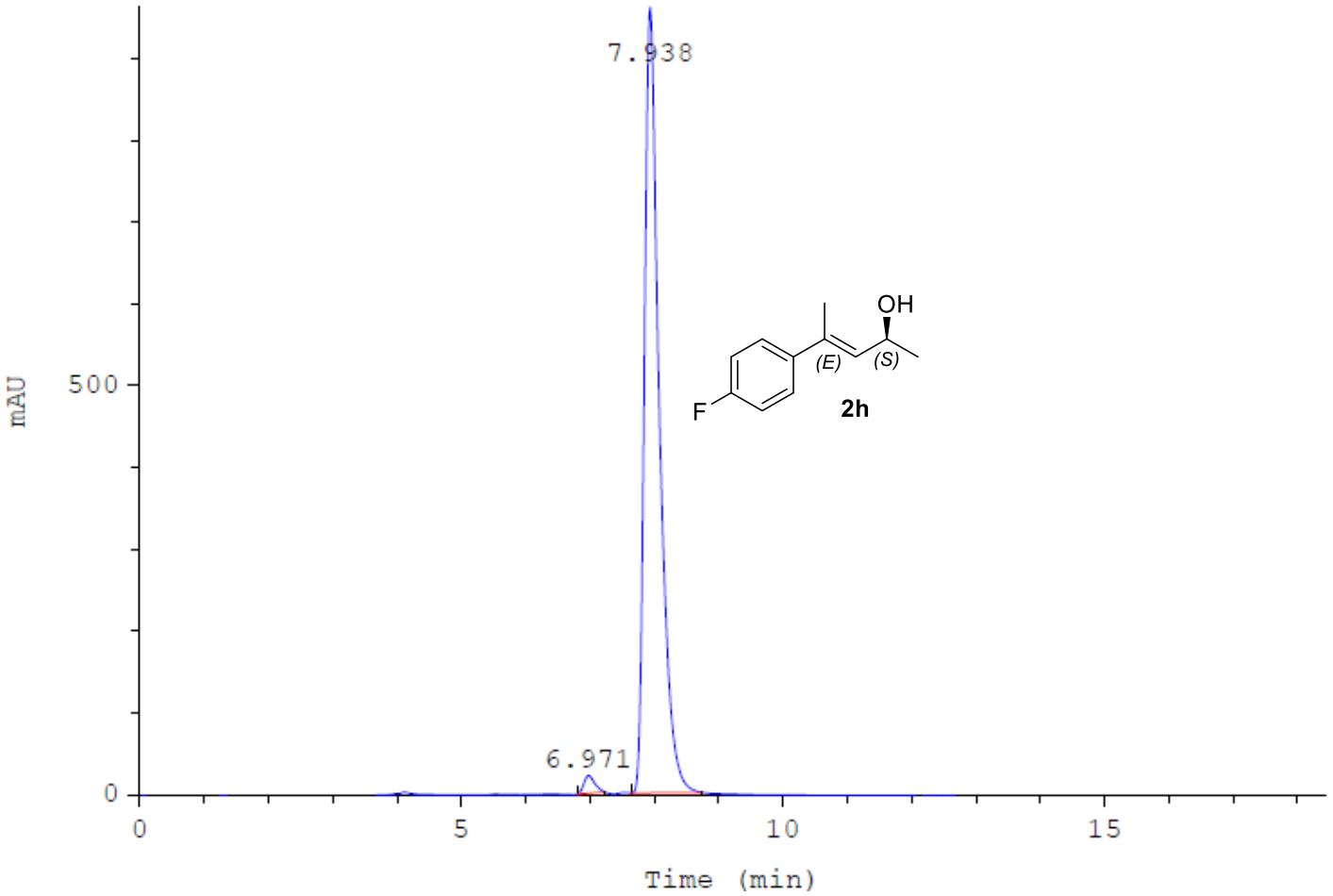
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

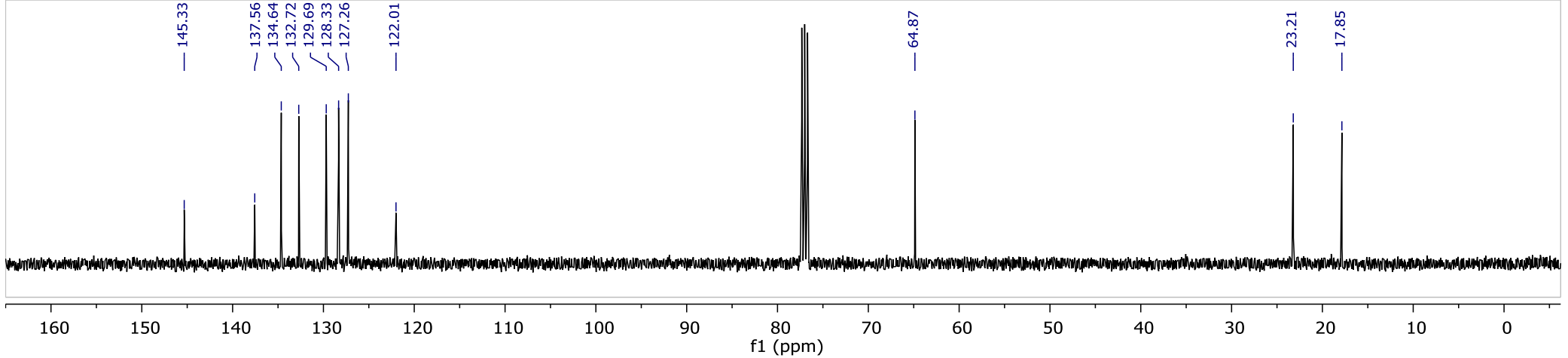
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	7.704	7.47	8.42	25.4682	105.096	49.4413	0.220
2	8.788	8.44	9.71	26.0438	94.106	50.5587	0.251

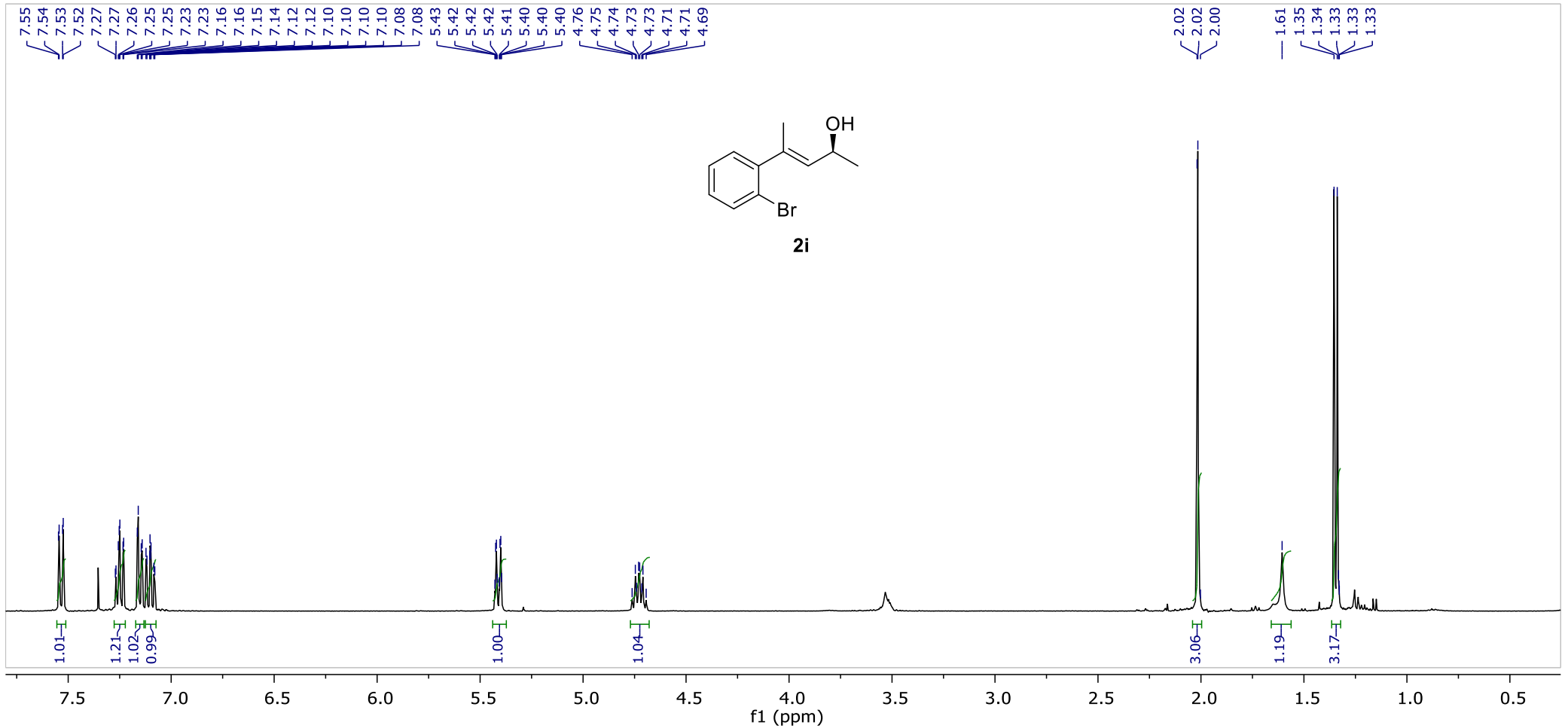


Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	6.971	6.81	7.23	4.1632	21.7962	1.5959	0.177
2	7.938	7.64	8.75	256.71	960.368	98.4041	0.233

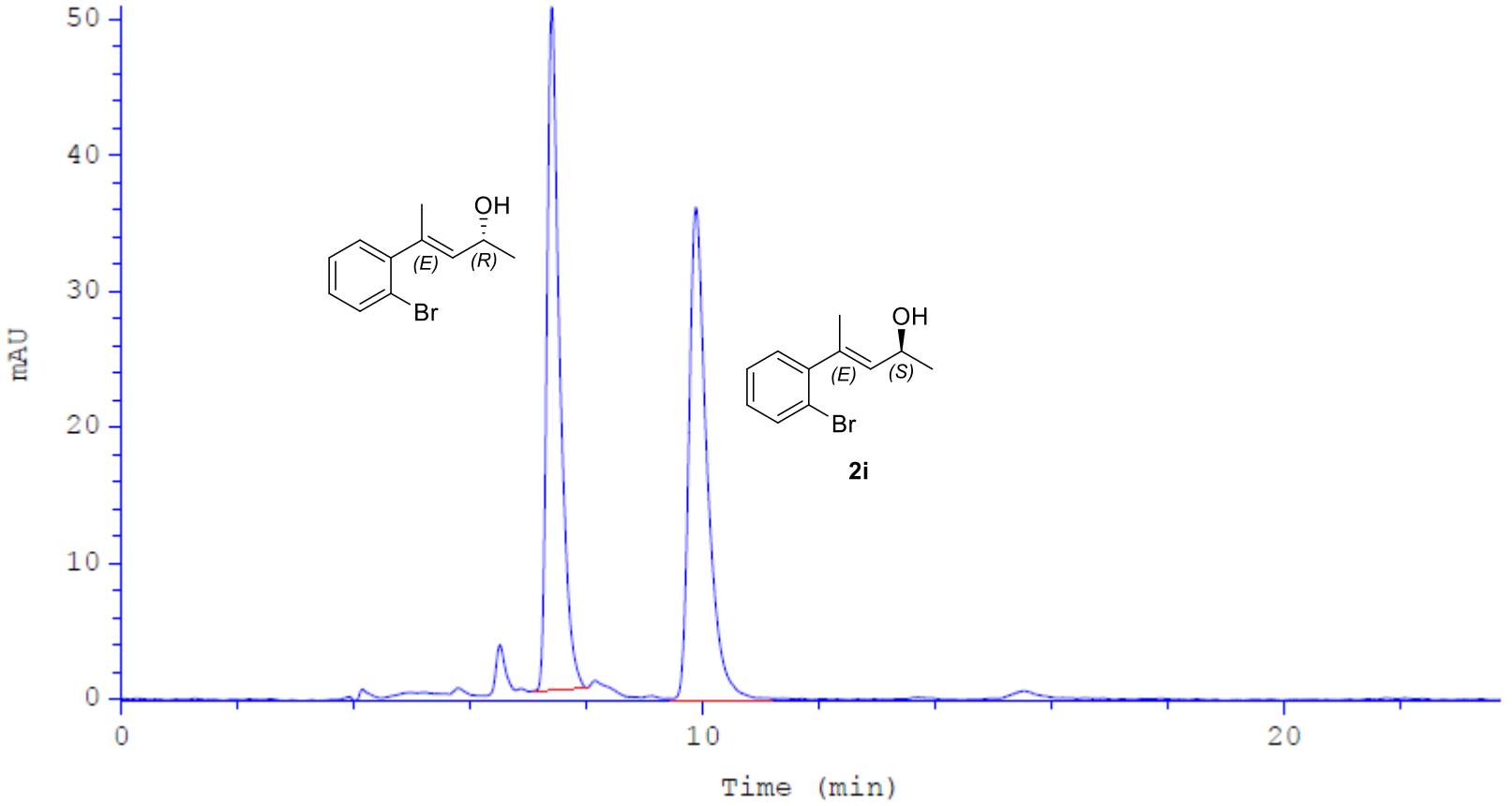



145.33
137.56
134.64
132.72
129.69
128.33
127.26
122.01
64.87
23.21
17.85
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.55
7.54
7.53
7.52
7.27
7.27
7.26
7.25
7.25
7.23
7.23
7.16
7.16
7.15
7.14
7.12
7.12
7.10
7.10
7.10
7.10
7.08
7.08
5.43
5.42
5.42
5.42
5.41
5.40
5.40
5.40
4.76
4.75
4.74
4.73
4.73
4.71
4.71
4.69
2.02
2.02
2.00
1.61
1.35
1.34
1.33
1.33
1.33
OH
Br
2i
1.01
1.21
1.02
0.99
1.00
1.04
3.06
1.19
3.17
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
f1 (ppm)

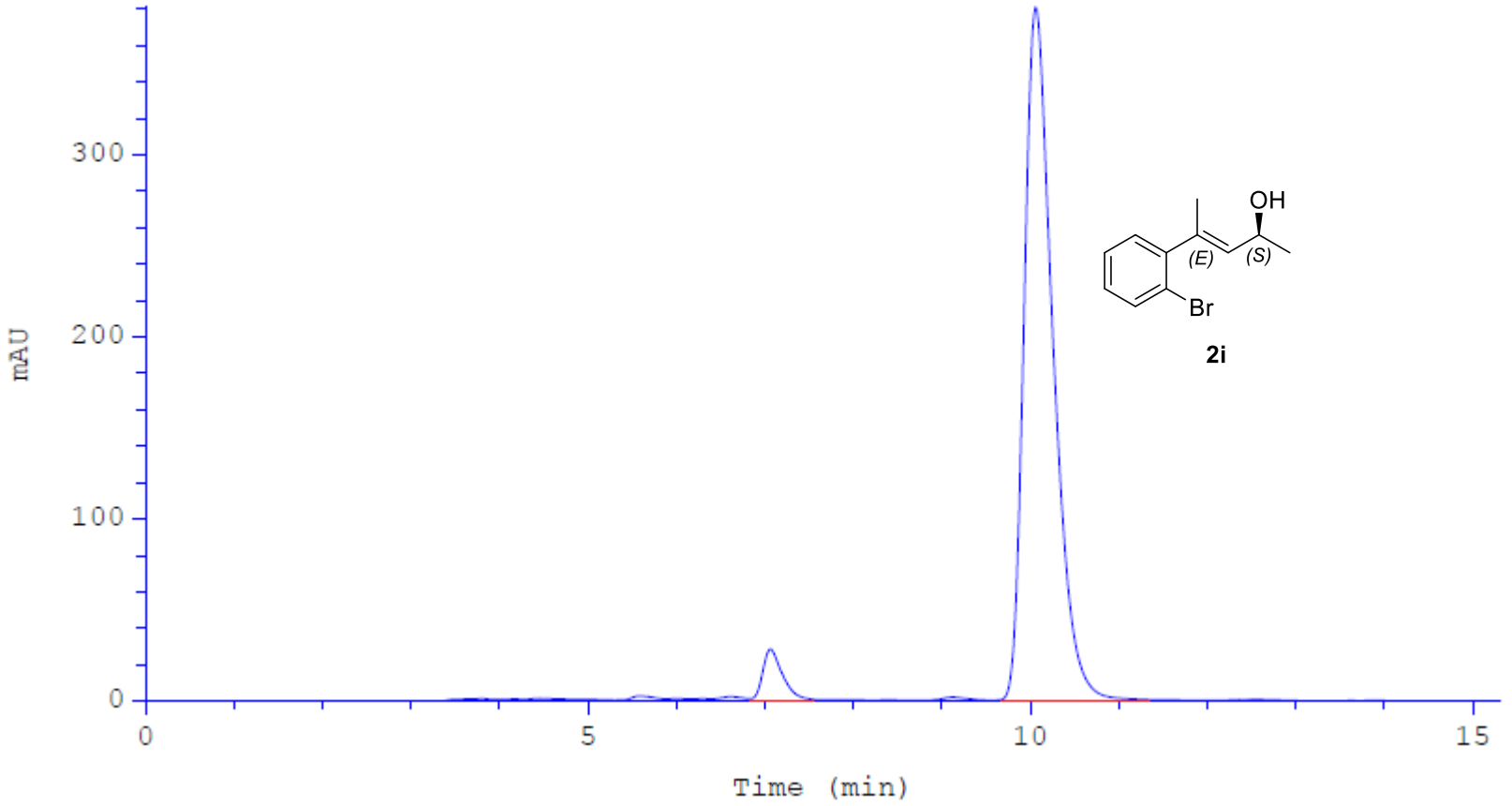
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	7.386	7.12	7.98	12.7896	50.2308	50.0287	0.223
2	9.871	9.50	10.88	12.7749	36.1299	49.9713	0.310

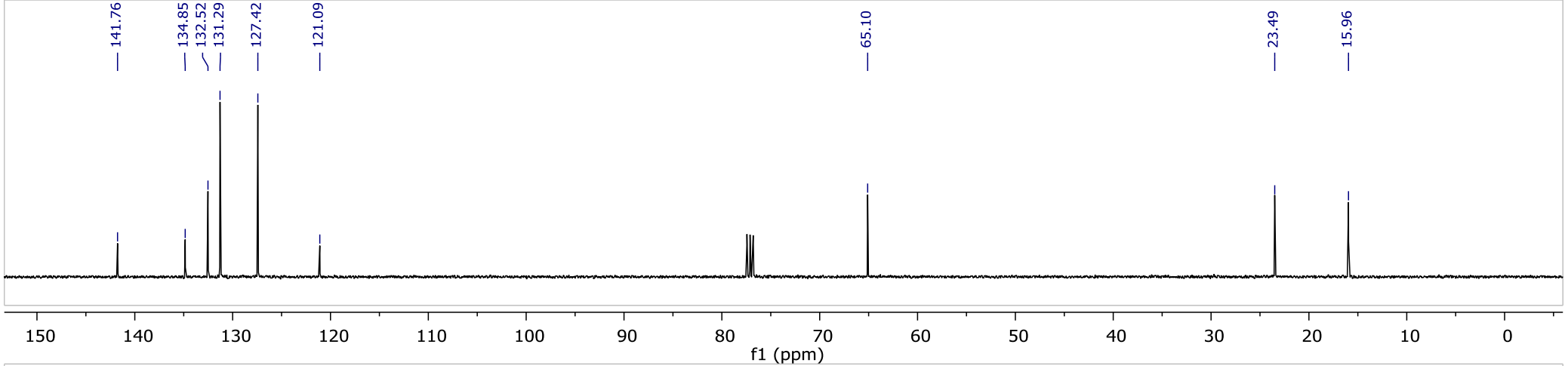


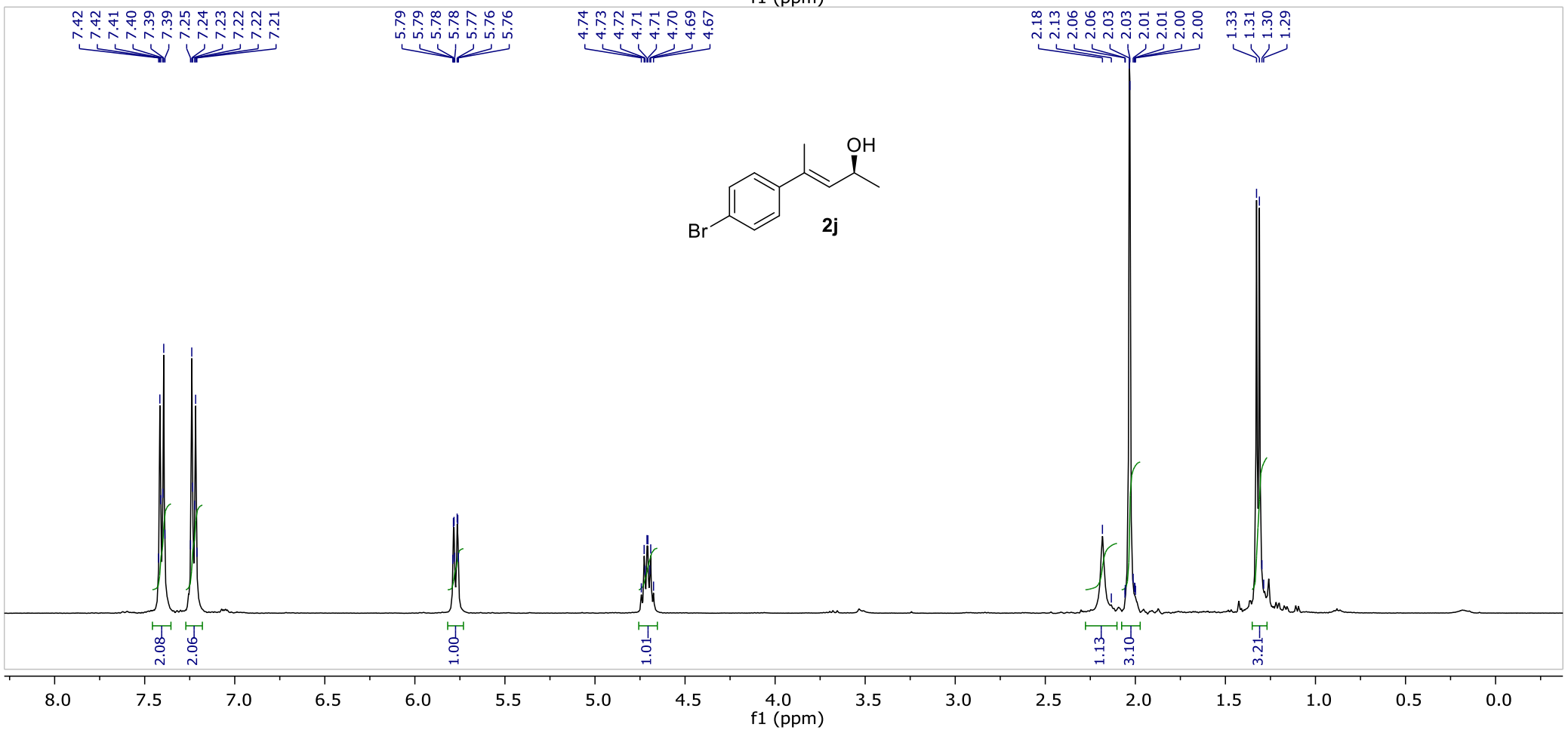
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	7.056	6.87	7.50	6.54871	26.8452	4.3799	0.225
2	10.058	9.69	11.18	142.967	379.783	95.6201	0.345



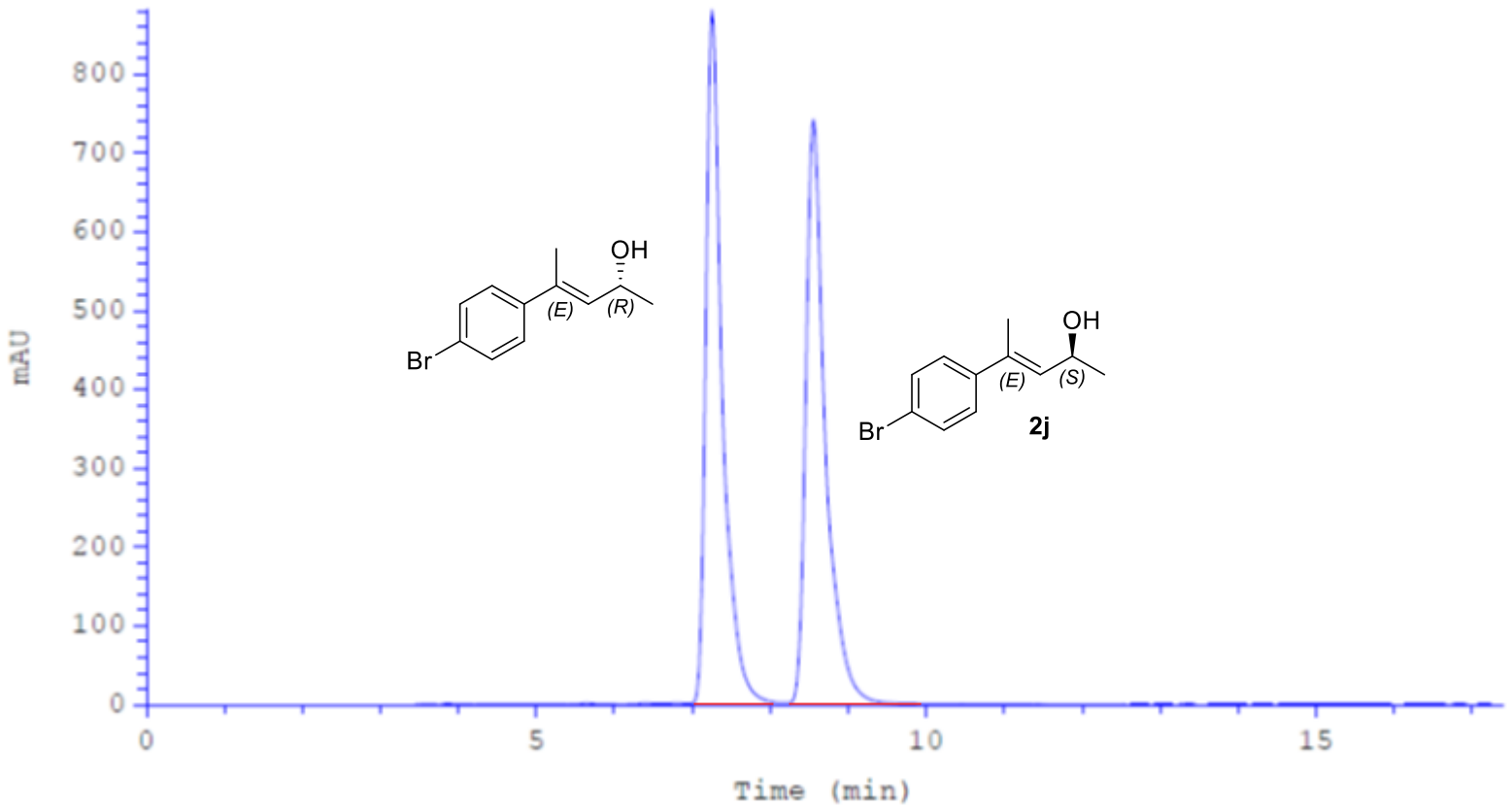
2i


141.76
134.85
132.52
131.29
127.42
121.09
65.10
23.49
15.96
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.42
7.42
7.41
7.40
7.39
7.39
7.25
7.24
7.23
7.22
7.22
7.21
5.79
5.79
5.78
5.78
5.77
5.76
5.76
4.74
4.73
4.72
4.71
4.71
4.70
4.69
4.67
2.18
2.13
2.06
2.06
2.03
2.03
2.01
2.01
2.00
2.00
1.33
1.31
1.30
1.29
OH
Br
2j
2.08
2.06
1.00
1.01
1.13
3.10
3.21
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
f1 (ppm)

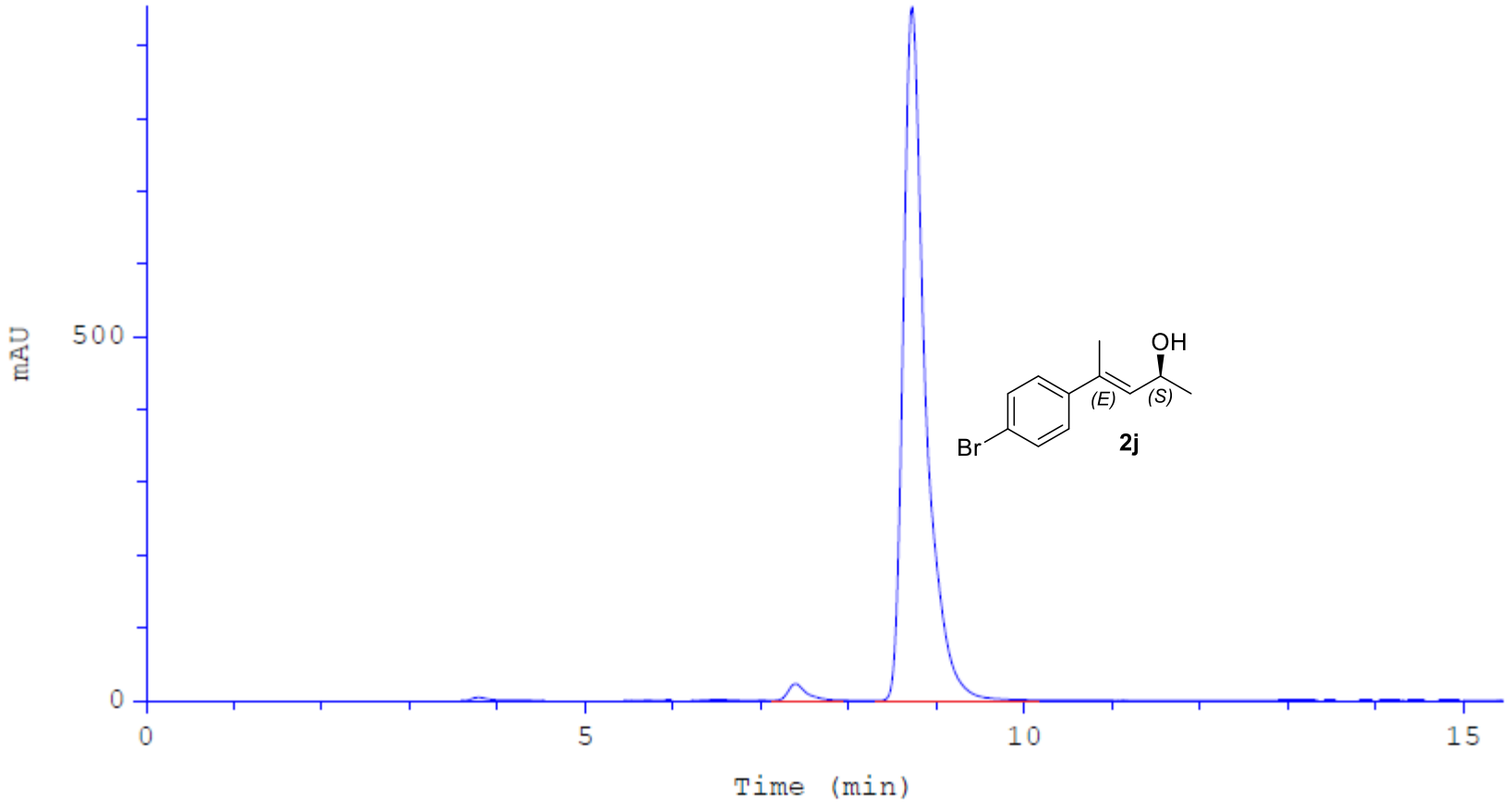
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

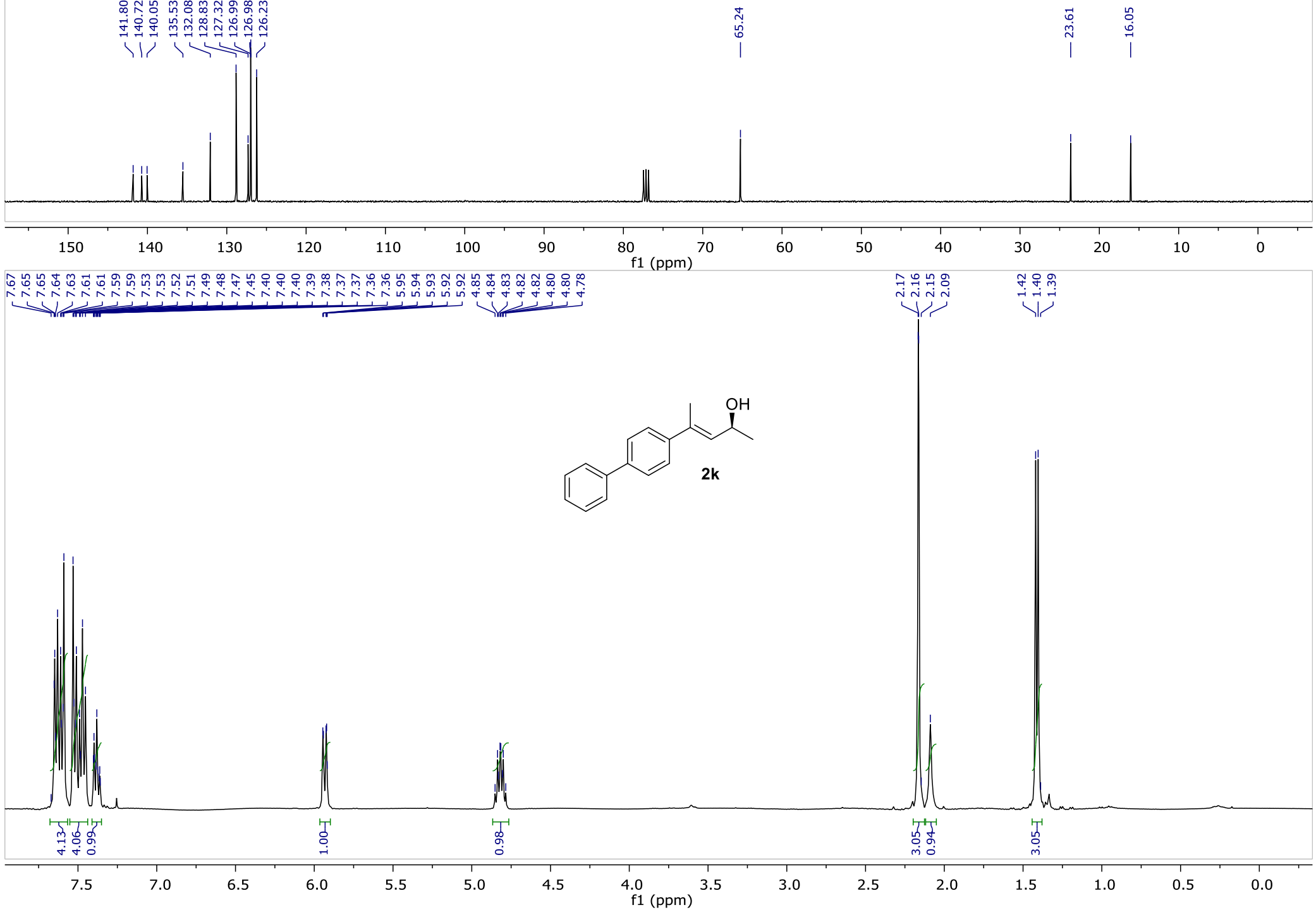
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	7.255	7.00	8.04	213.109	879.447	49.9770	0.201
2	8.556	8.27	9.96	213.306	739.344	50.0230	0.241



Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

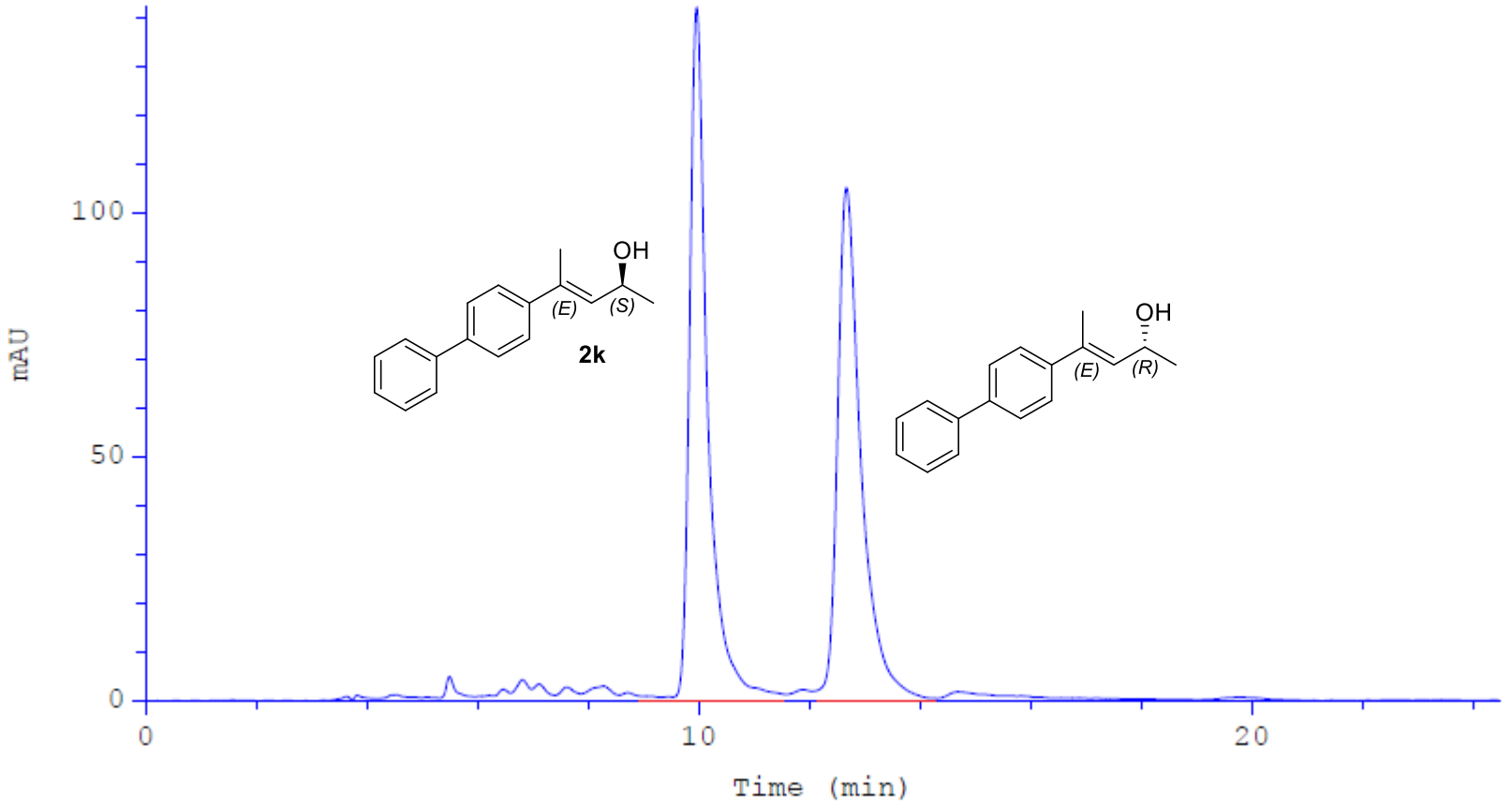
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	7.389	7.16	7.85	5.34146	22.7371	1.8779	0.200
2	8.724	8.38	10.15	279.098	953.007	98.1221	0.244




141.80
140.72
140.05
135.53
132.08
128.83
127.32
126.99
126.98
126.23
65.24
23.61
16.05
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)
7.67
7.65
7.65
7.64
7.63
7.61
7.61
7.59
7.59
7.53
7.53
7.52
7.51
7.49
7.48
7.47
7.45
7.40
7.40
7.40
7.39
7.38
7.37
7.37
7.36
7.36
5.95
5.94
5.93
5.92
5.92
4.85
4.84
4.83
4.82
4.82
4.80
4.80
4.78
2.17
2.16
2.15
2.09
1.42
1.40
1.39
OH
2k
4.13
4.06
0.99
1.00
0.98
3.05
0.94
3.05
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
f1 (ppm)

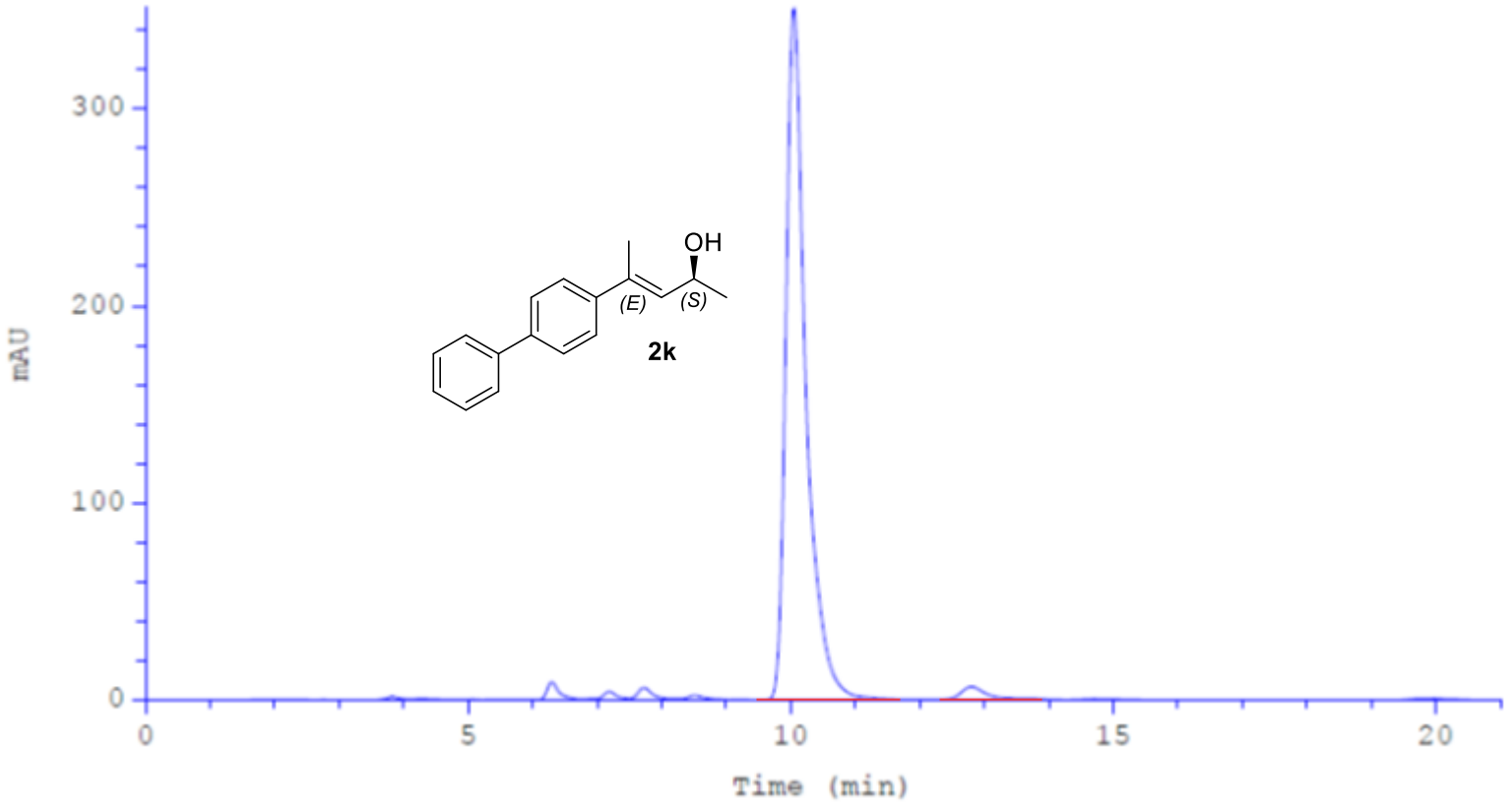
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

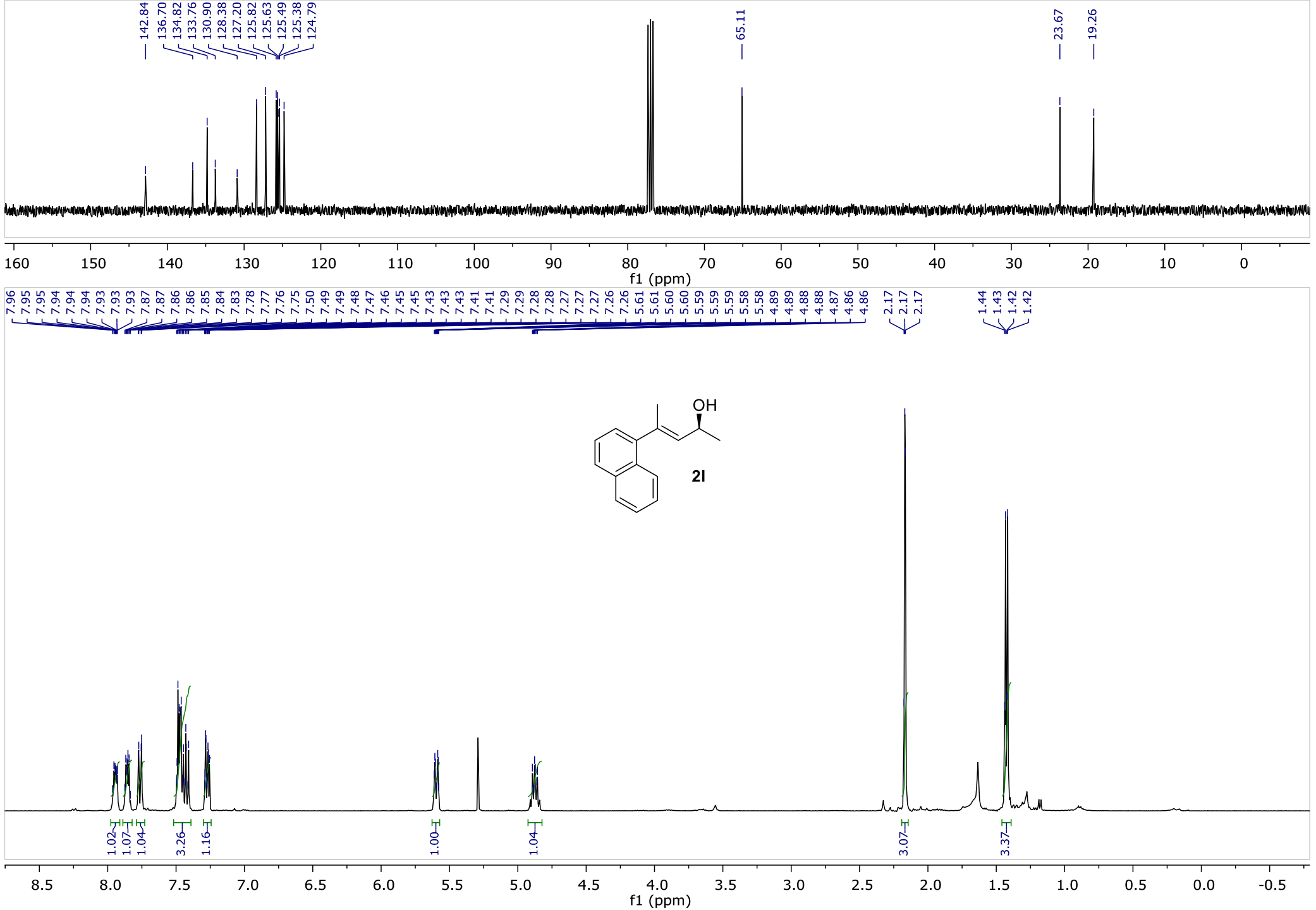
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	9.971	9.54	11.60	52.4511	141.21	51.2552	0.304
2	12.672	12.07	14.21	49.8821	103.646	48.7448	0.410



Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

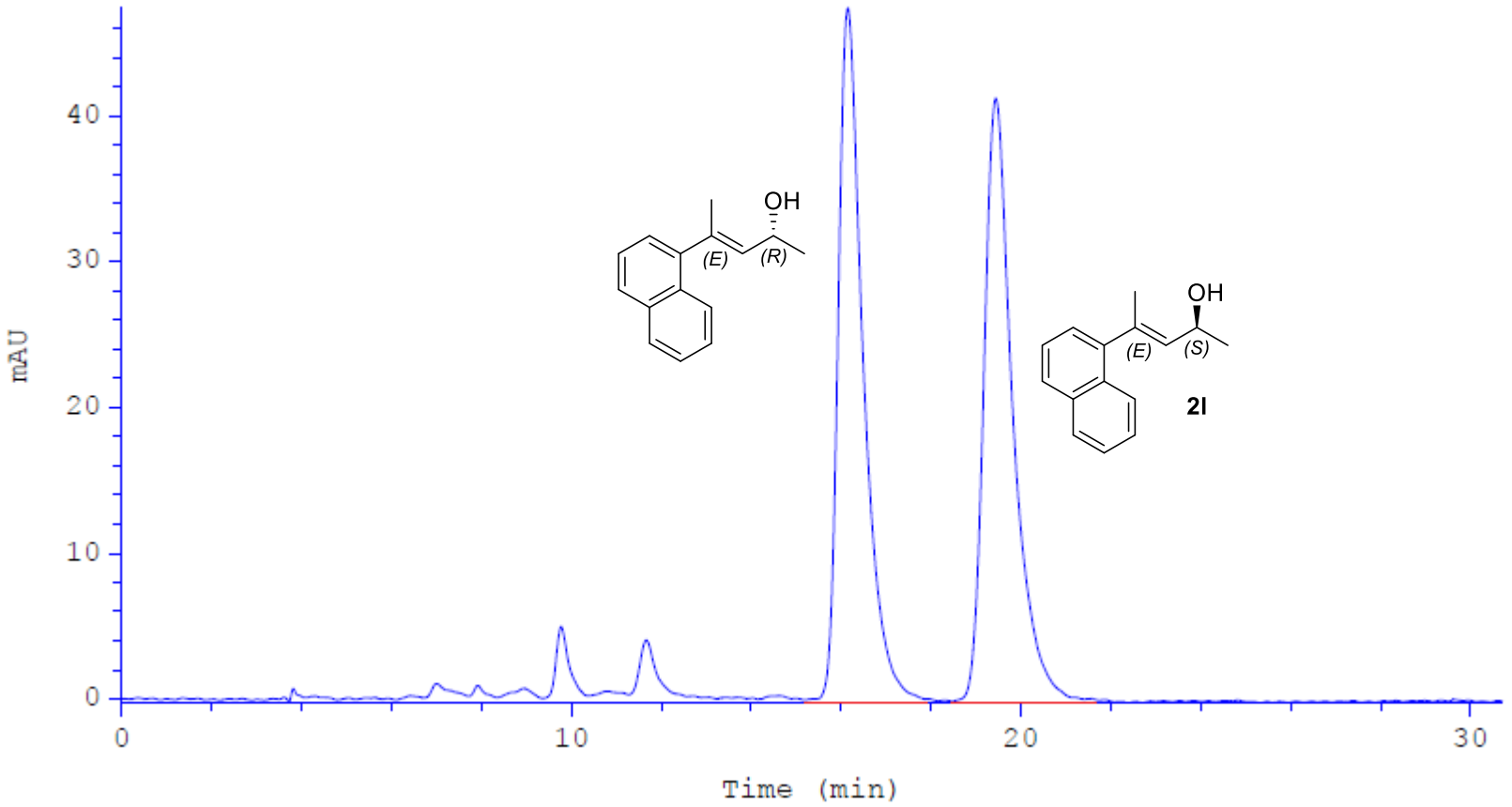
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	10.055	9.64	11.61	125.813	350.911	97.8268	0.300
2	12.807	12.42	13.90	2.79492	6.30792	2.1732	0.377




142.84
136.70
134.82
133.76
130.90
128.38
127.20
125.82
125.63
125.49
125.38
124.79
65.11
23.67
19.26
f1 (ppm)
OH
2l
1.02
1.07
1.04
3.26
1.16
1.00
1.04
3.07
3.37
f1 (ppm)

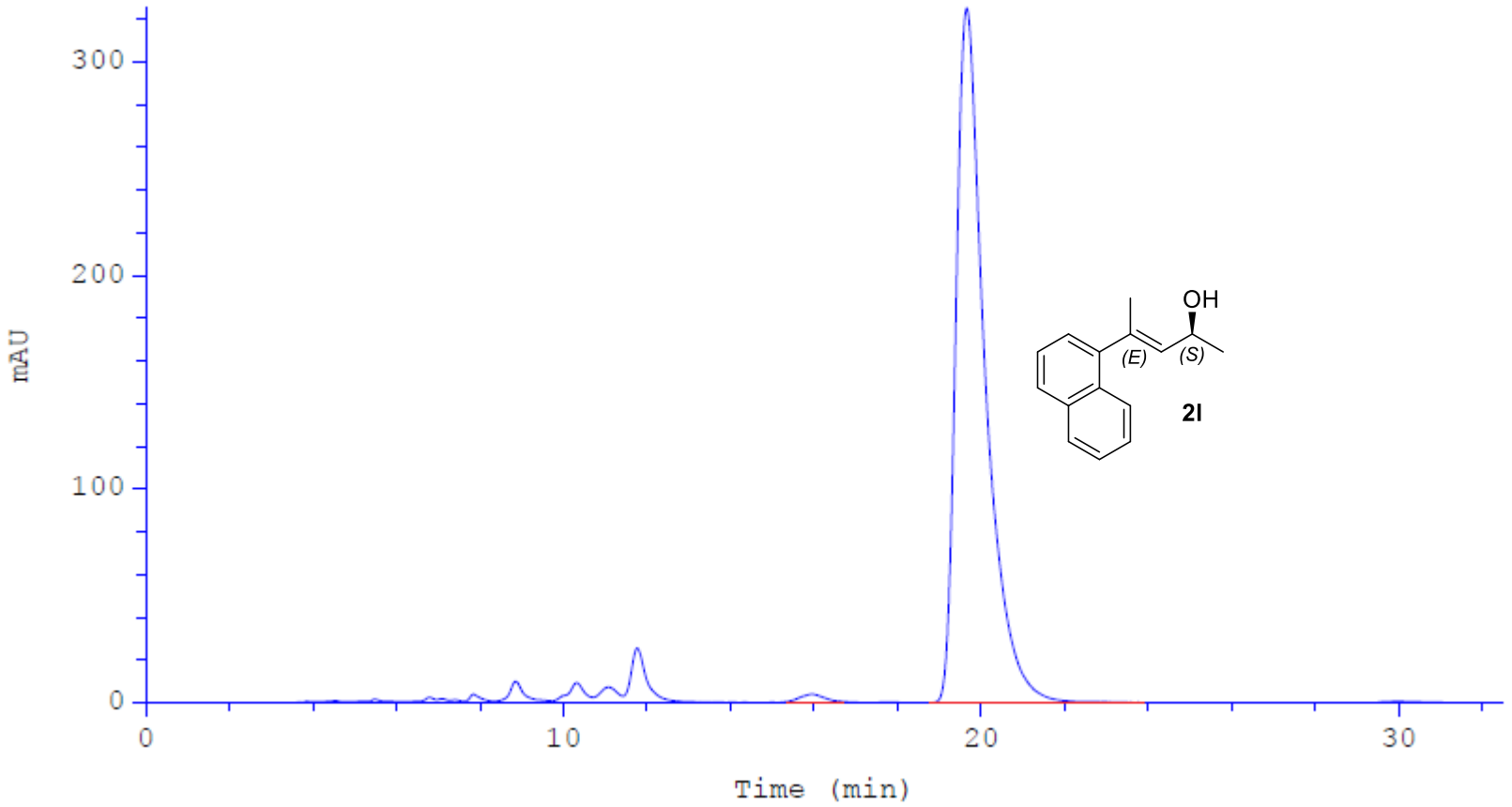
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

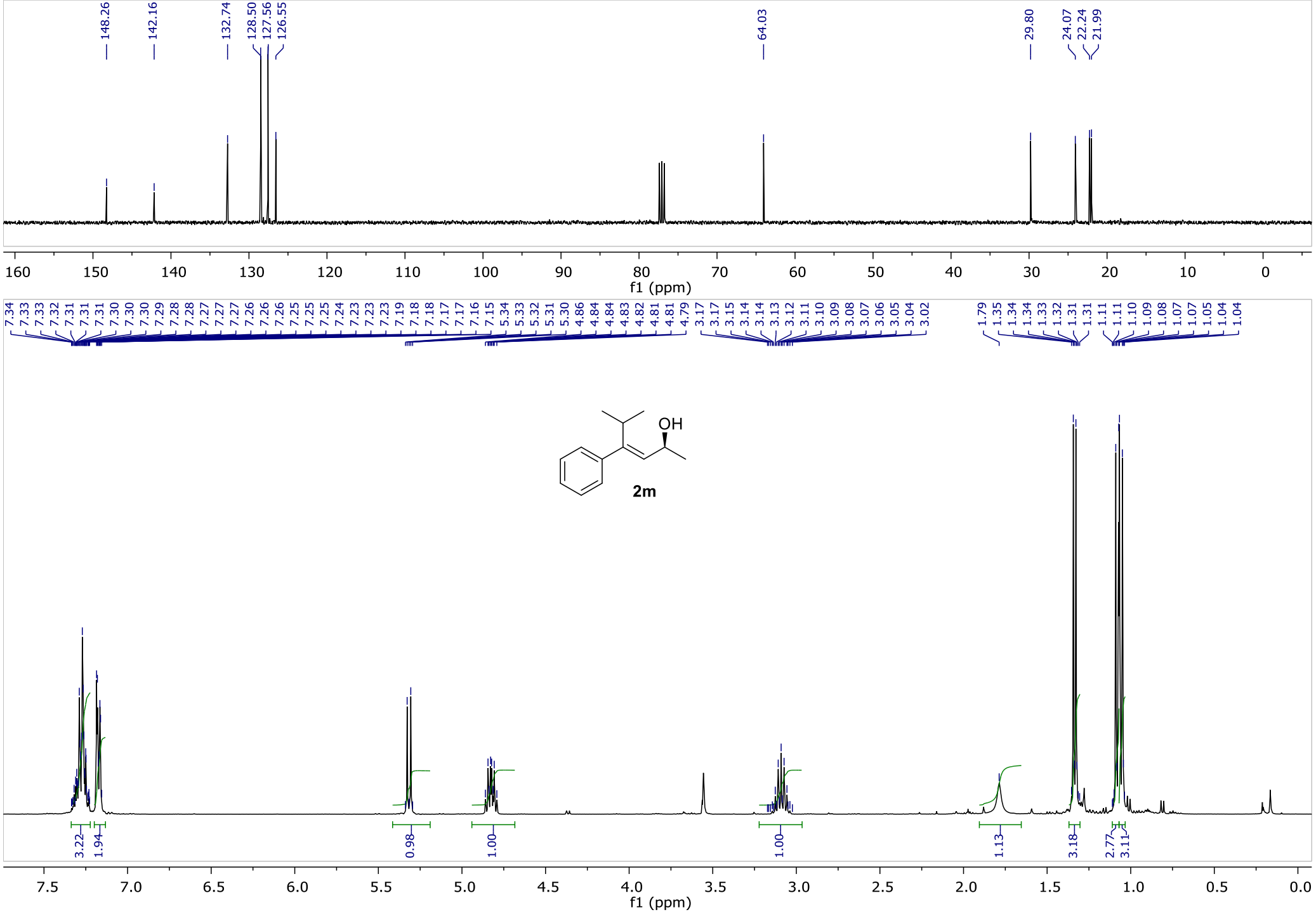
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	16.154	15.37	17.96	30.3979	47.4034	49.9883	0.565
2	19.438	18.67	21.43	30.4121	41.2347	50.0117	0.641



Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

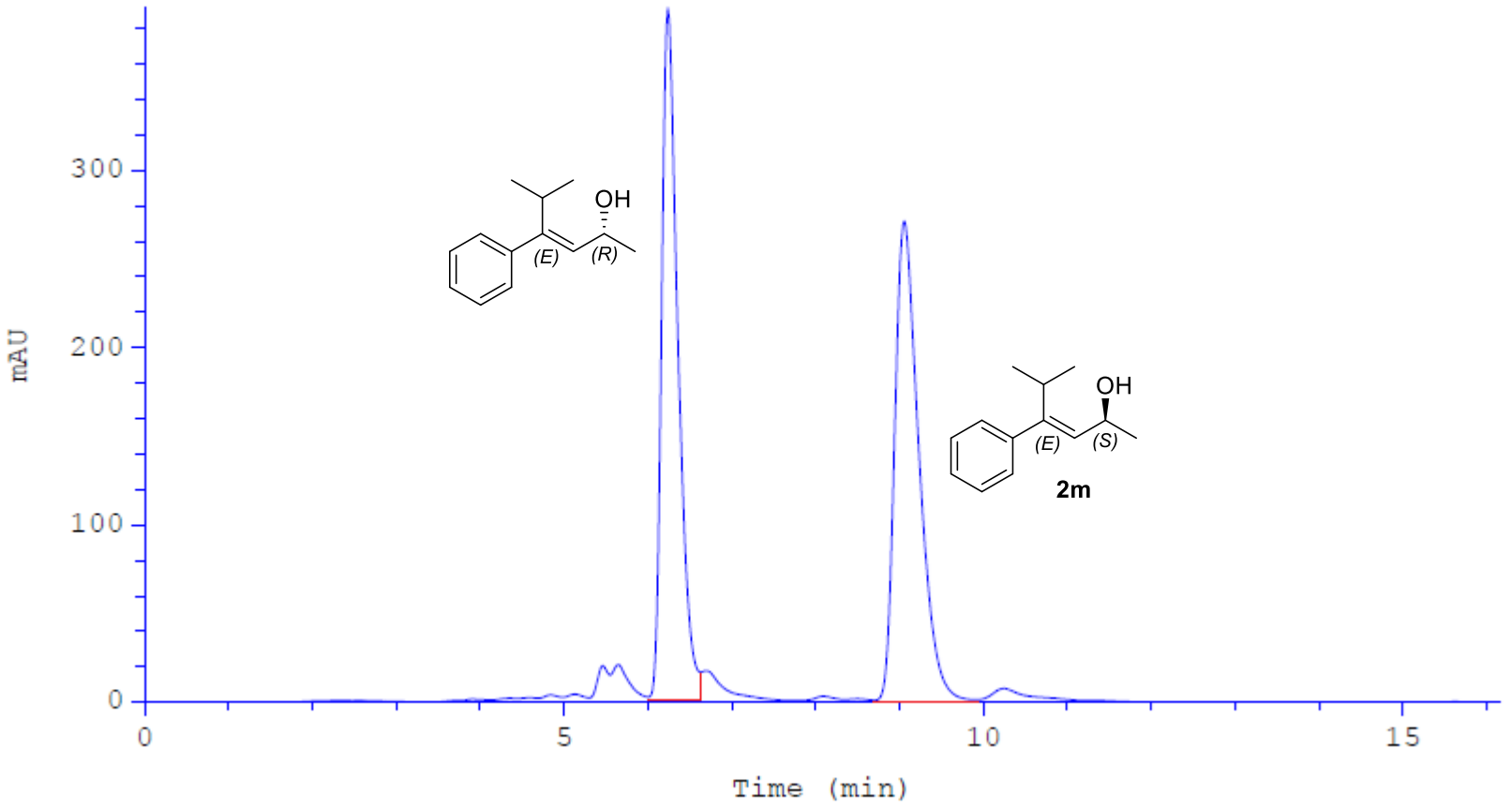
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	15.970	15.46	16.43	1.62042	2.93634	0.6170	0.552
2	19.655	18.80	21.94	260.994	324.84	99.3830	0.697




148.26
142.16
132.74
128.50
127.56
126.55
64.03
29.80
24.07
22.24
21.99
f1 (ppm)
OH
2m
3.22
1.94
0.98
1.00
1.00
1.13
3.18
2.77
3.11
f1 (ppm)

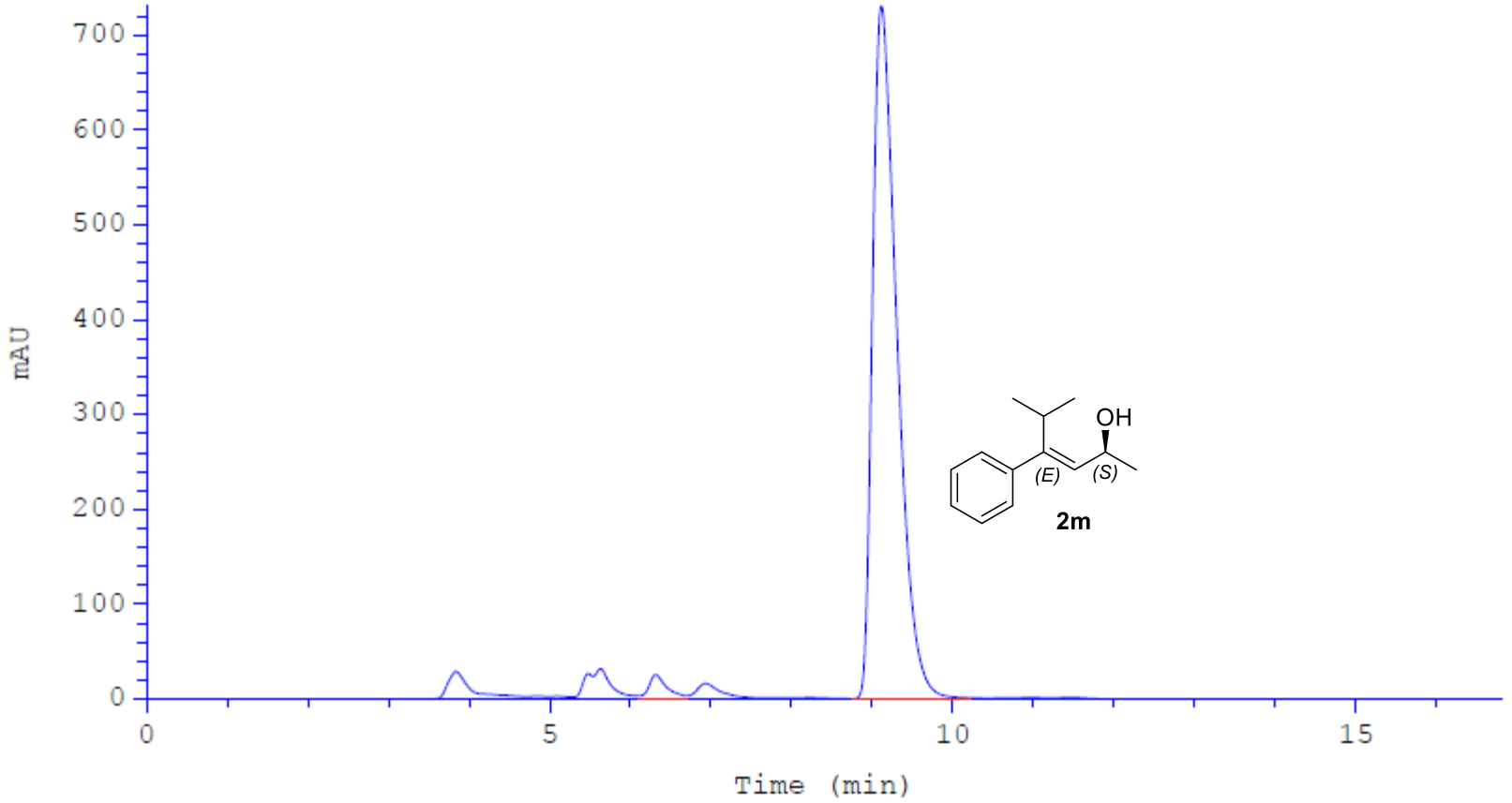
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

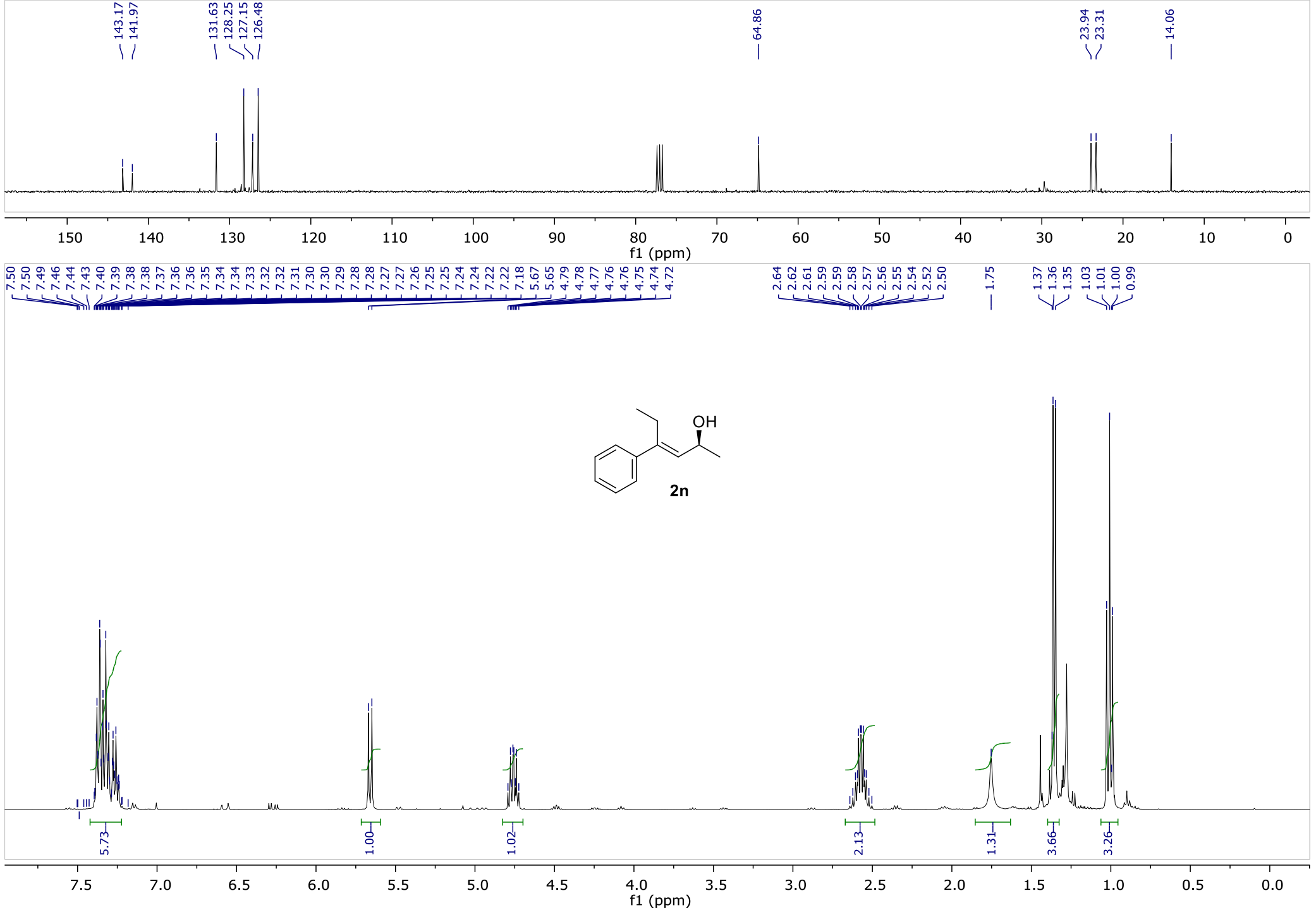
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	6.238	5.97	6.64	83.0466	382.912	48.3411	0.202
2	9.057	8.75	9.78	88.7463	269.615	51.6589	0.303

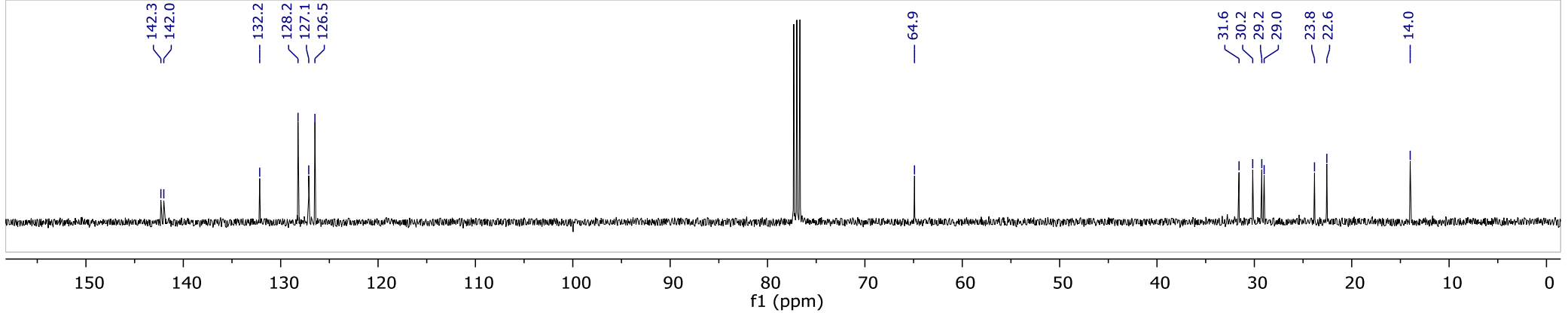


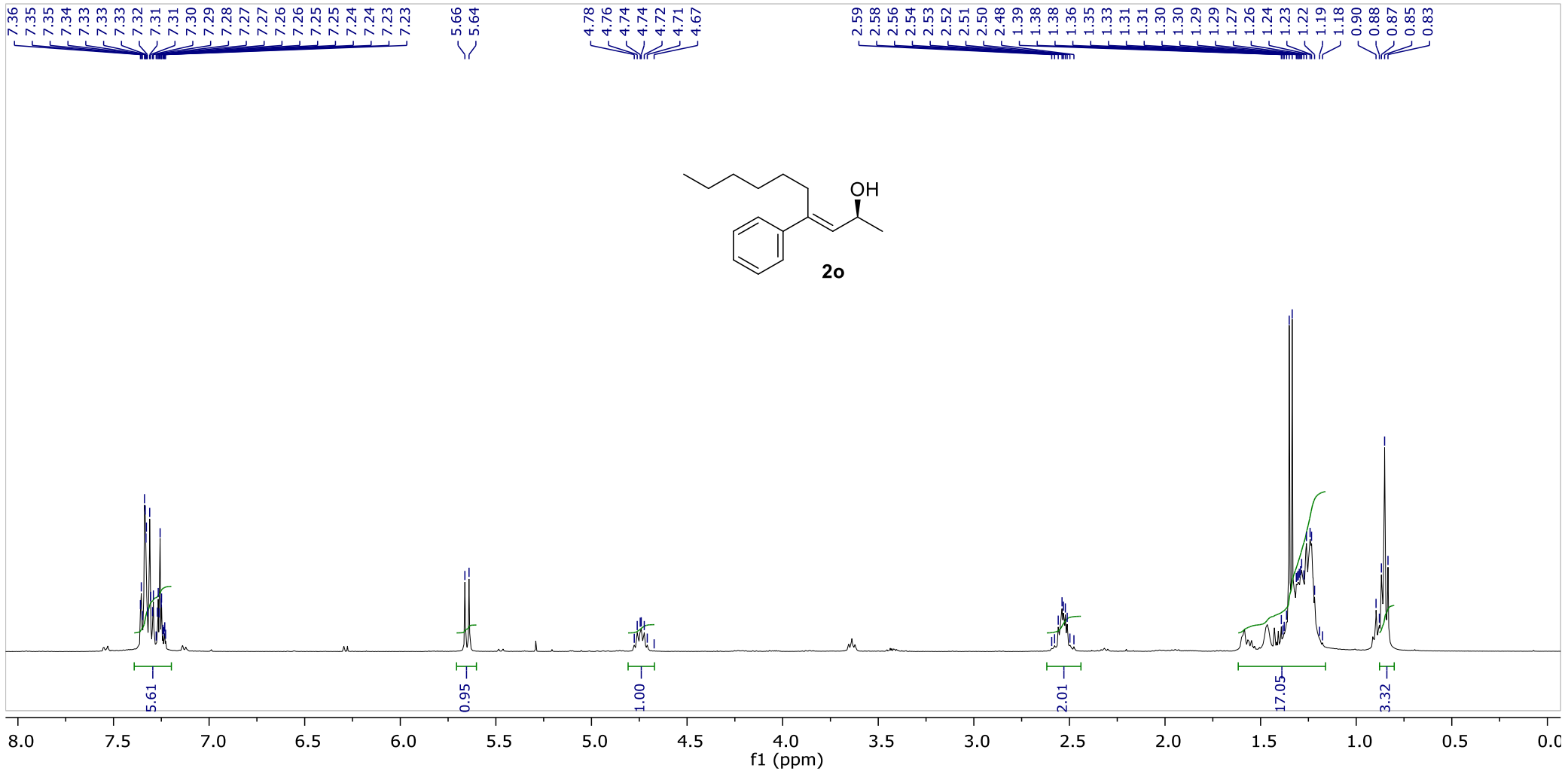
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

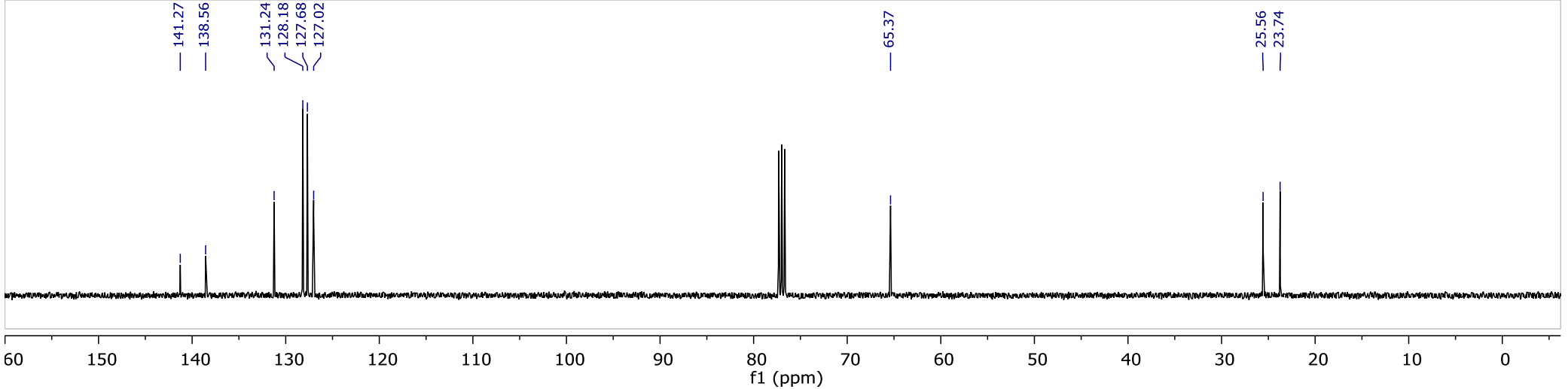
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	6.321	6.12	6.68	4.82593	22.0849	1.8503	0.195
2	9.106	8.79	10.13	255.998	729.49	98.1497	0.326

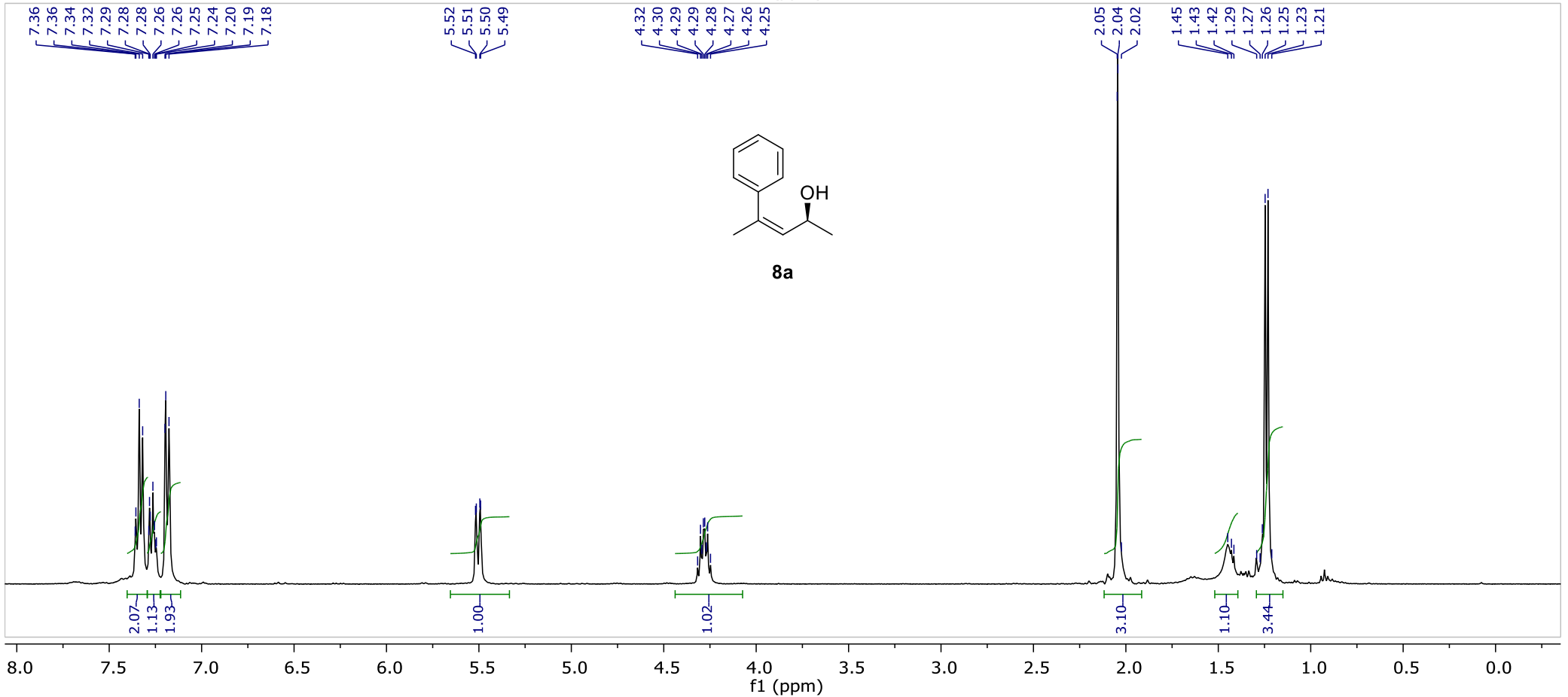


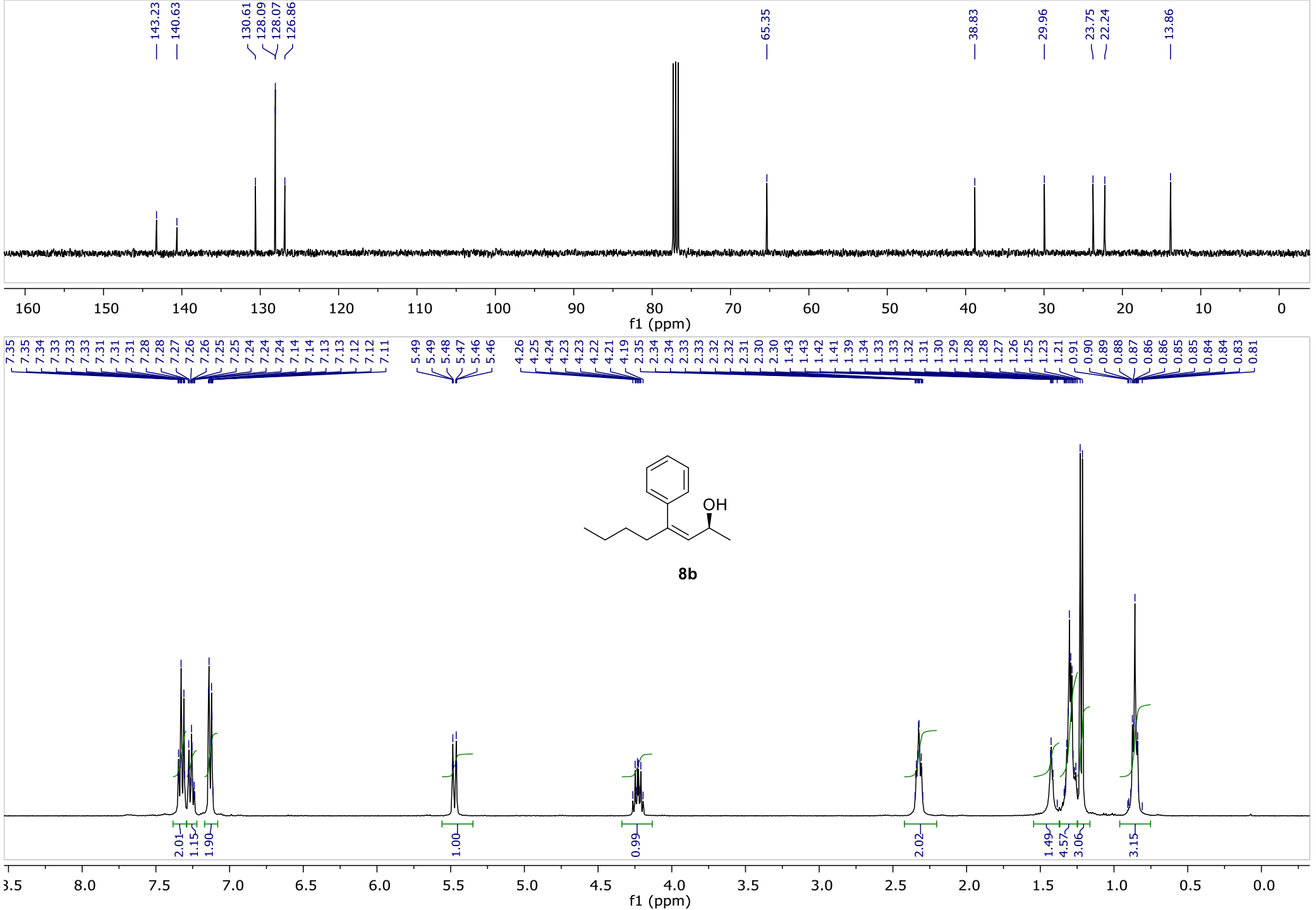

143.17
141.97
131.63
128.25
127.15
126.48
64.86
23.94
23.31
14.06
f1 (ppm)
OH
2n
5.73
1.00
1.02
2.13
1.31
3.66
3.26
f1 (ppm)

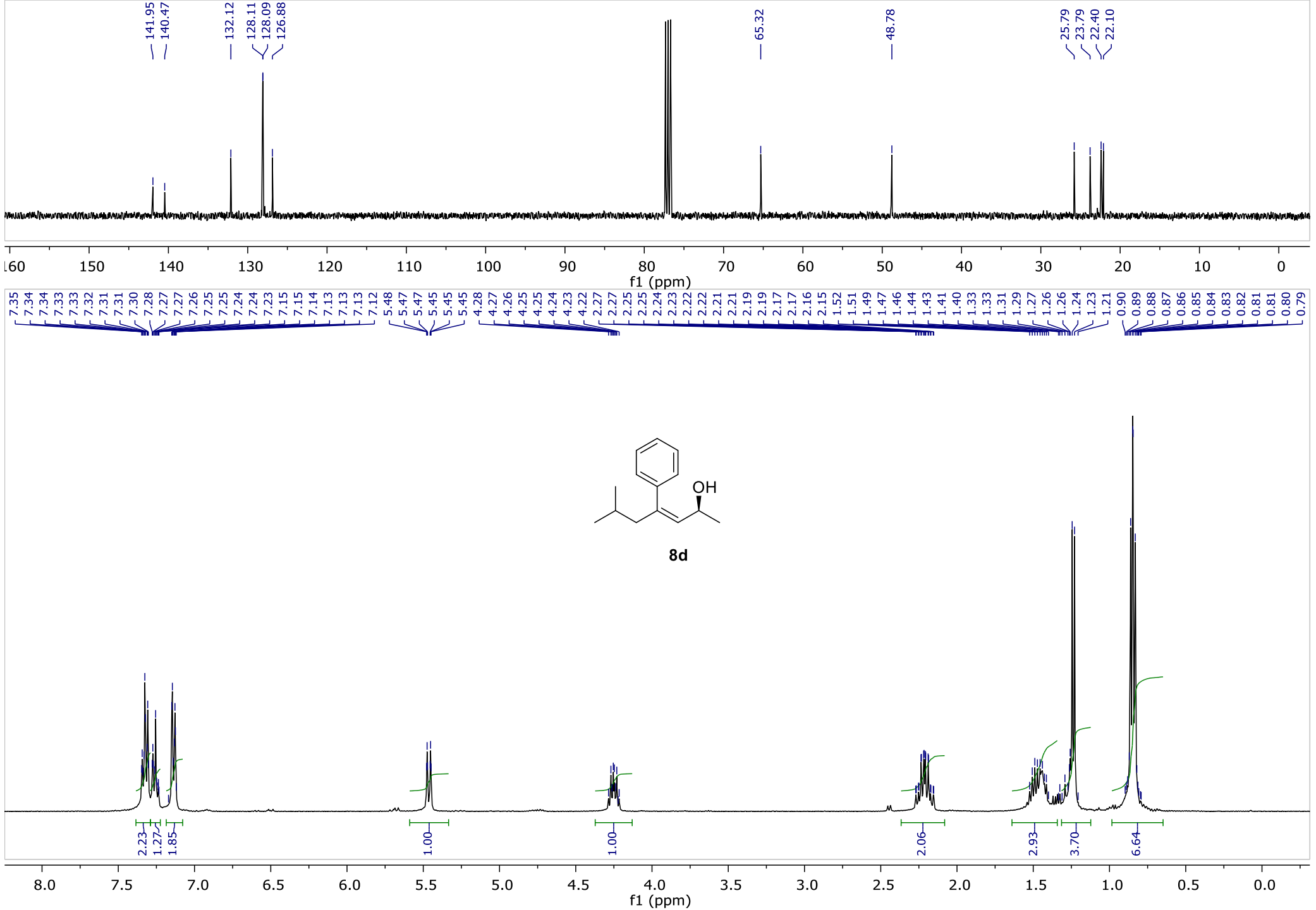

142.3
142.0
132.2
128.2
127.1
126.5
64.9
31.6
30.2
29.2
29.0
23.8
22.6
14.0
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

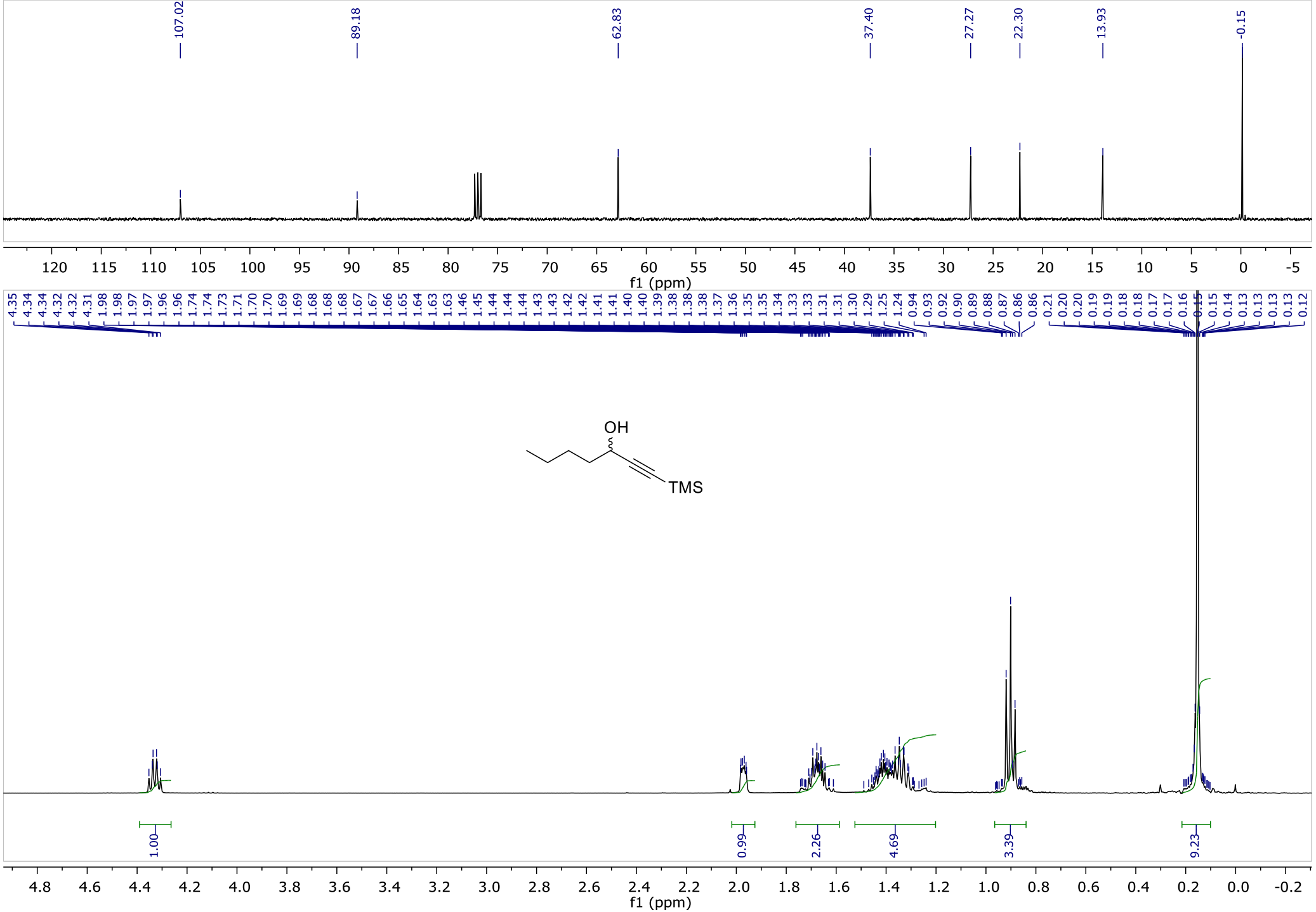

7.36
7.35
7.35
7.34
7.33
7.33
7.33
7.32
7.31
7.31
7.30
7.29
7.28
7.27
7.27
7.26
7.26
7.25
7.25
7.24
7.24
7.23
7.23
5.66
5.64
4.78
4.76
4.74
4.74
4.72
4.71
4.67
2.59
2.58
2.56
2.54
2.53
2.52
2.51
2.50
2.48
1.39
1.38
1.38
1.36
1.35
1.33
1.31
1.31
1.30
1.30
1.29
1.29
1.27
1.26
1.24
1.23
1.22
1.19
1.18
0.90
0.88
0.87
0.85
0.83
OH
2o
5.61
0.95
1.00
2.01
17.05
3.32
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
f1 (ppm)

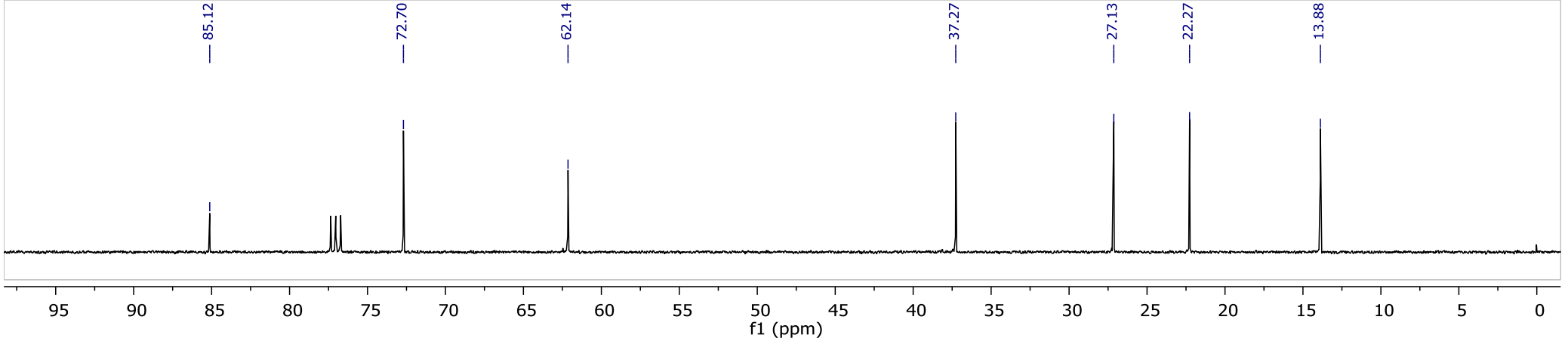

141.27
138.56
131.24
128.18
127.68
127.02
65.37
25.56
23.74
f1 (ppm)

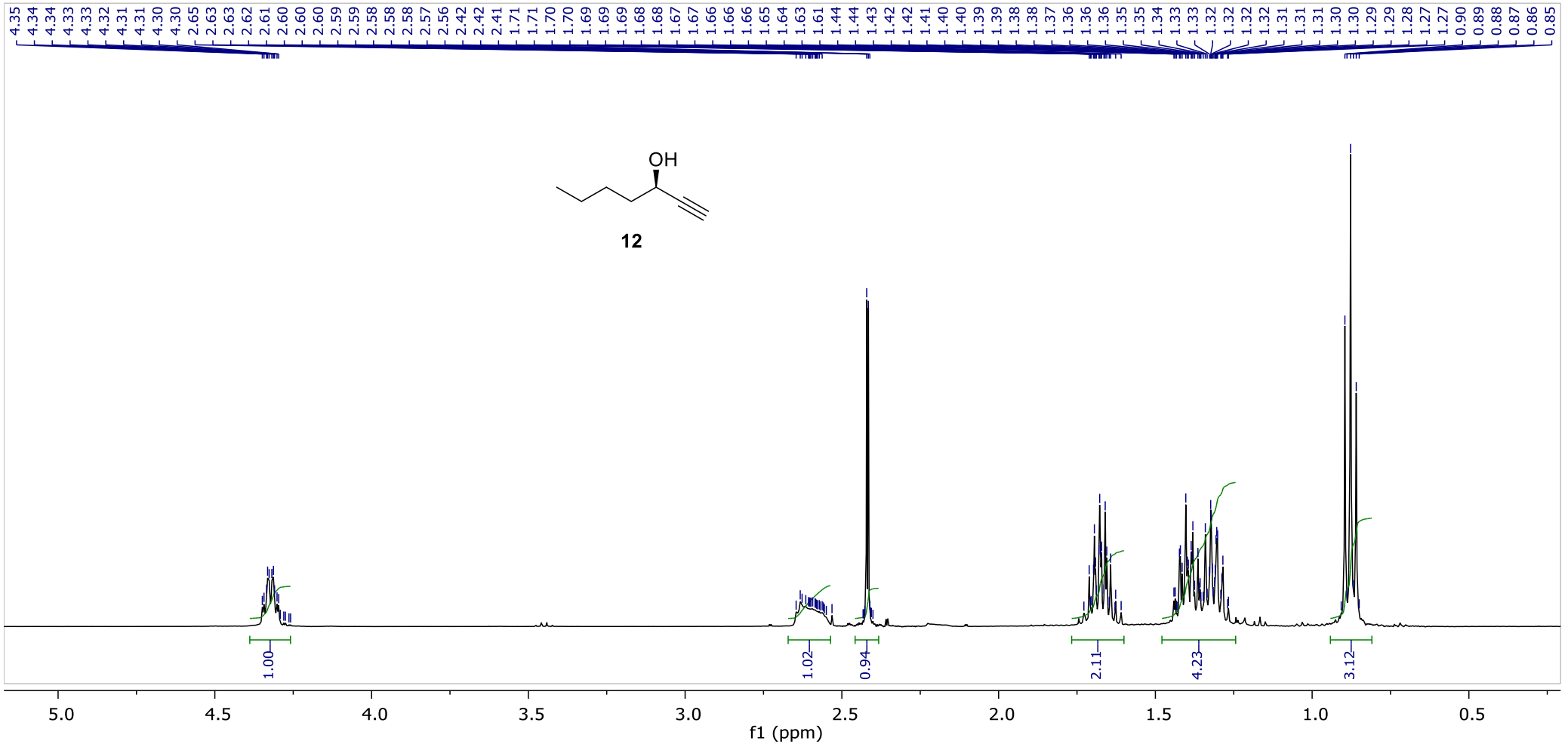

7.36
7.36
7.34
7.32
7.29
7.28
7.28
7.26
7.26
7.25
7.24
7.20
7.19
7.18
5.52
5.51
5.50
5.49
4.32
4.30
4.29
4.29
4.28
4.27
4.26
4.25
2.05
2.04
2.02
1.45
1.43
1.42
1.29
1.27
1.26
1.25
1.23
1.21
OH
8a
2.07
1.13
1.93
1.00
1.02
3.10
1.10
3.44
f1 (ppm)

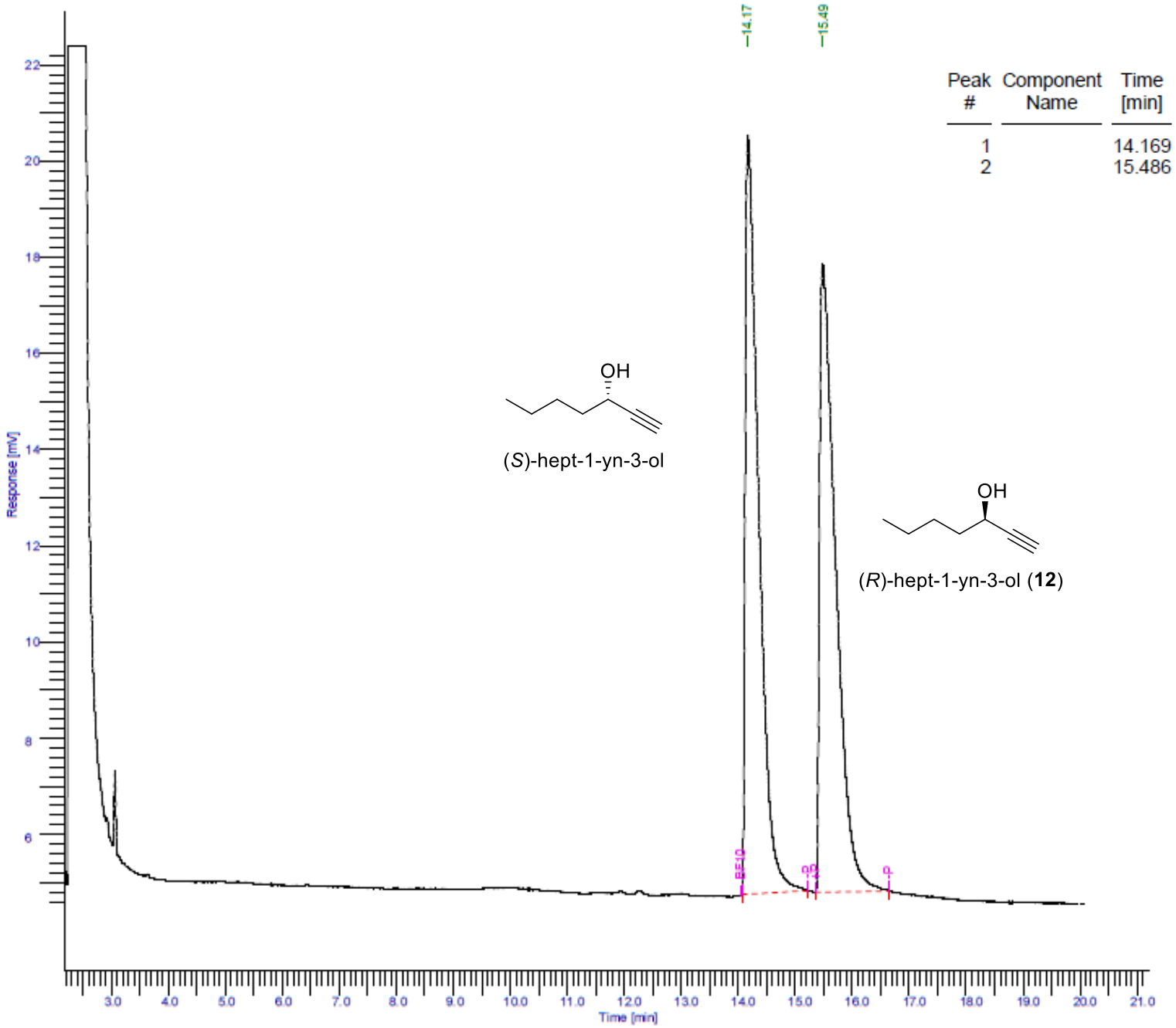

143.23
140.63
130.61
128.09
128.07
126.86
65.35
38.83
29.96
23.75
22.24
13.86
f1 (ppm)
OH
8b
2.01
1.15
1.90
1.00
0.99
2.02
1.49
4.57
3.06
3.15
f1 (ppm)


141.95
140.47
132.12
128.11
128.09
126.88
65.32
48.78
25.79
23.79
22.40
22.10
f1 (ppm)
OH
8d
2.23
1.27
1.85
1.00
1.00
2.06
2.93
3.70
6.64
f1 (ppm)


107.02
89.18
62.83
37.40
27.27
22.30
13.93
-0.15
f1 (ppm)
OH
TMS
1.00
0.99
2.26
4.69
3.39
9.23
f1 (ppm)


85.12
72.70
62.14
37.27
27.13
22.27
13.88
95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)


OH
12
1.00
1.02
0.94
2.11
4.23
3.12
5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)



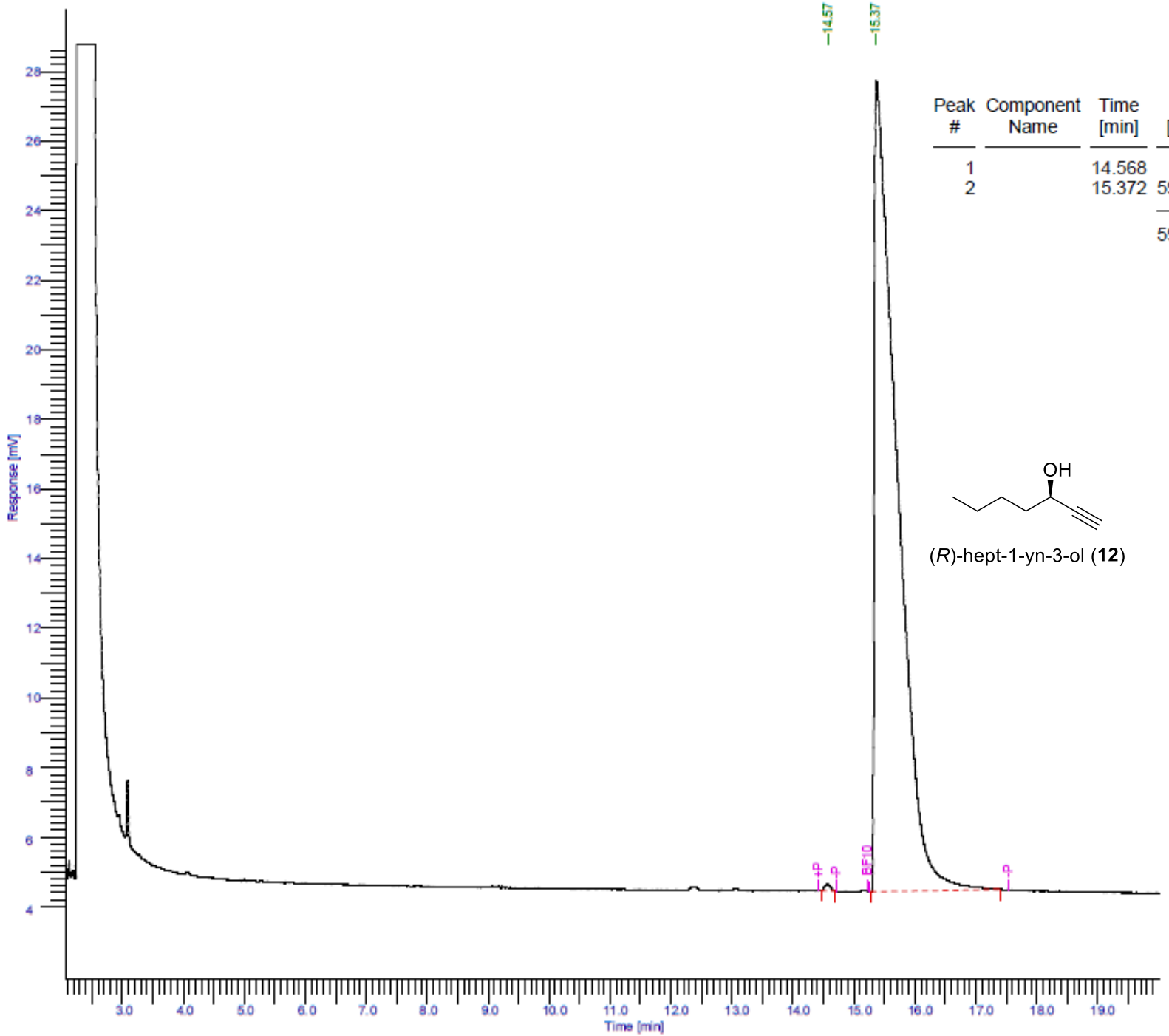
(*S*)-hept-1-yn-3-ol

(*R*)-hept-1-yn-3-ol (**12**)

DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		14.169	258124.99	15779.42	50.06	50.06		+	*BB	----------	----------
2		15.486	257557.62	13051.53	49.94	49.94			*BB	0.2576	0.2576
			515682.61	28830.95	100.00	100.00				0.2576	0.2576

Column: Agilent Cyclosil-B
(30m x 0.25 mm x 0.25 µm)
Injection temp.: 150 °C
Detection temp.: 270 °C
Oven temp: 90 °C (30 min),
5°C/min to 150°, hold for 5.00 min

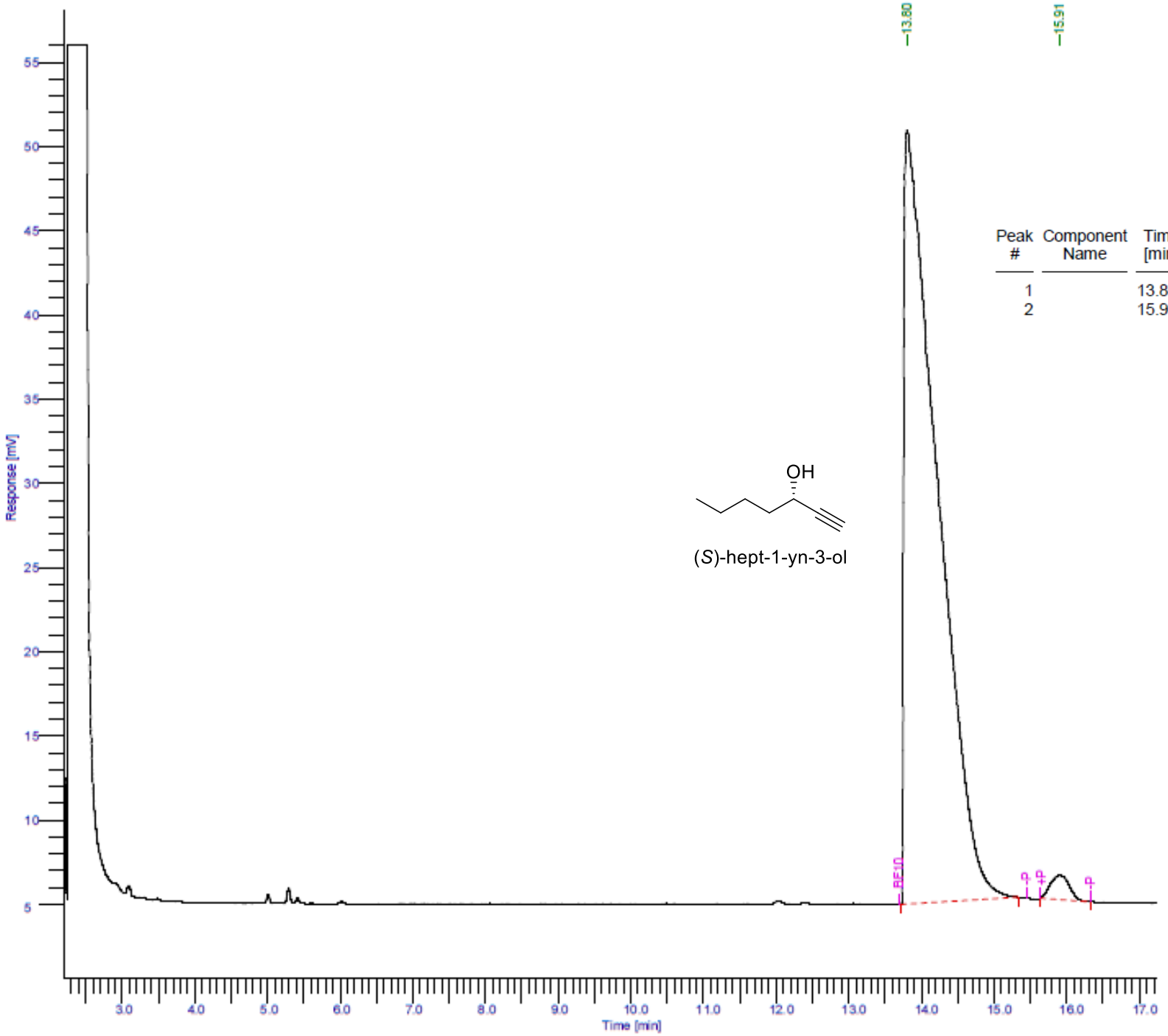


DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		14.568	1201.46	186.97	0.20	0.20			BB	0.0012	0.0012
2		15.372	596664.39	23303.66	99.80	99.80			*BB	0.5967	0.5967
			597865.85	23490.62	100.00	100.00				0.5979	0.5979

(*R*)-hept-1-yn-3-ol (**12**)

Column: Agilent Cyclosil-B
(30m x 0.25 mm x 0.25 µm)
Injection temp.: 150 °C
Detection temp.: 270 °C
Oven temp: 90 °C (30 min),
5°C/min to 150°, hold for 5.00 min

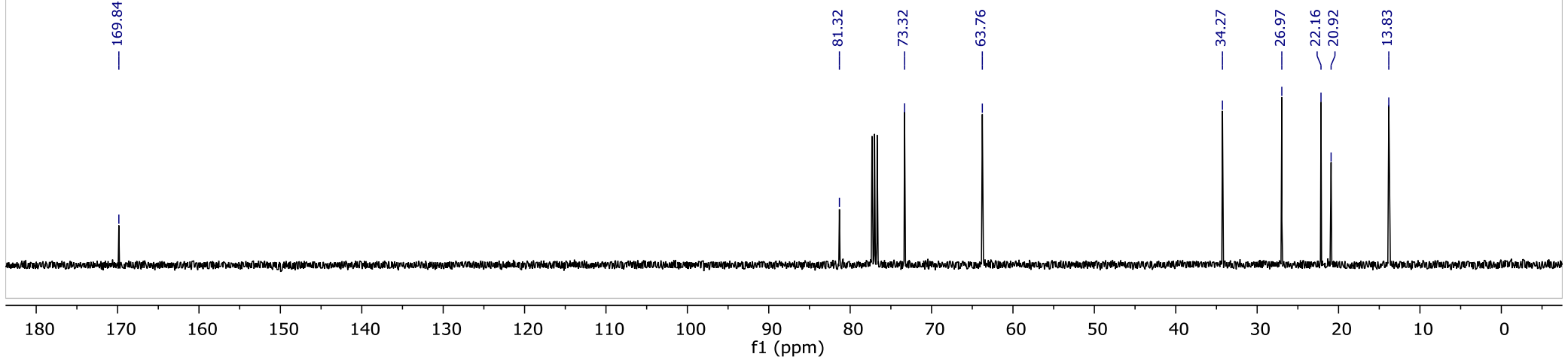


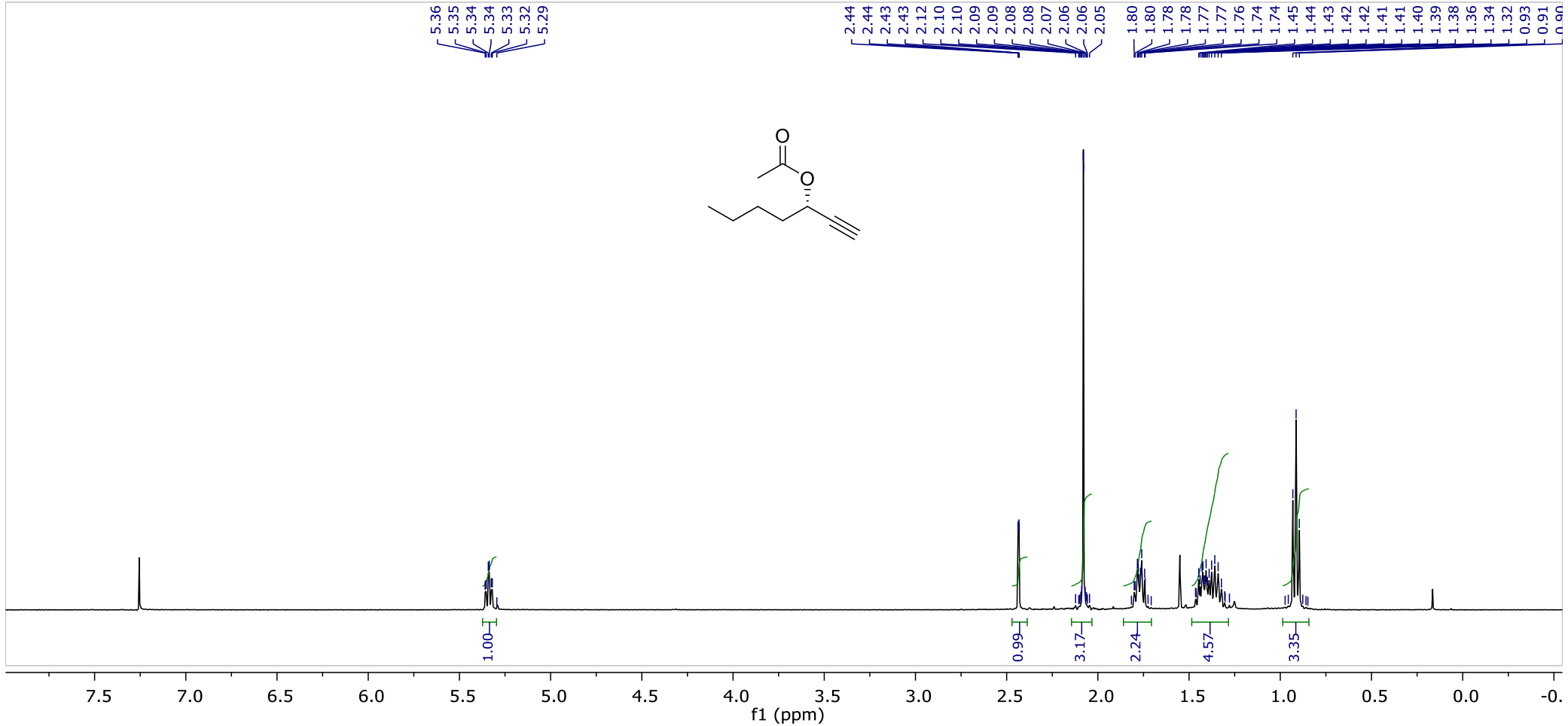
(*S*)-hept-1-yn-3-ol

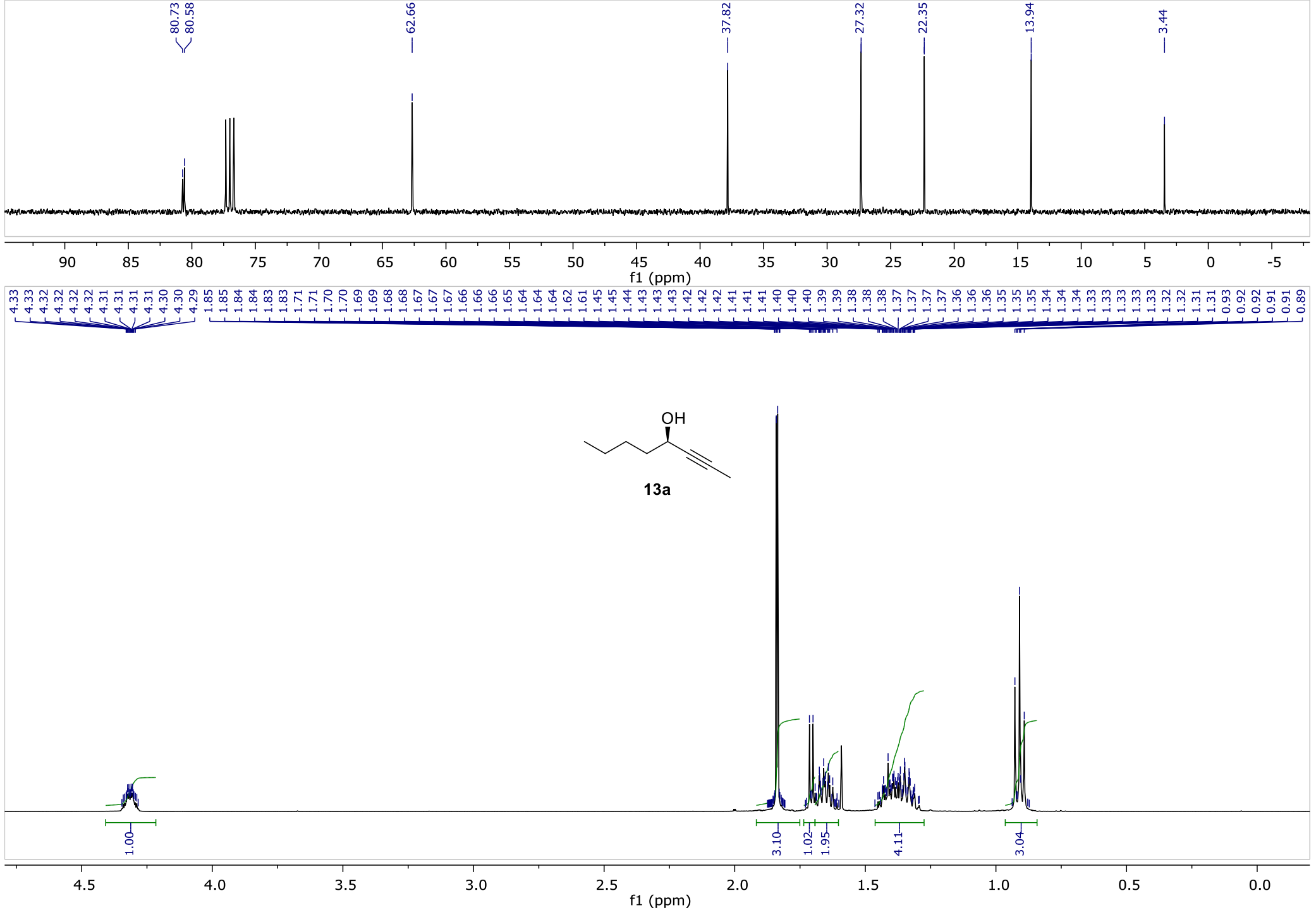
DEFAULT REPORT

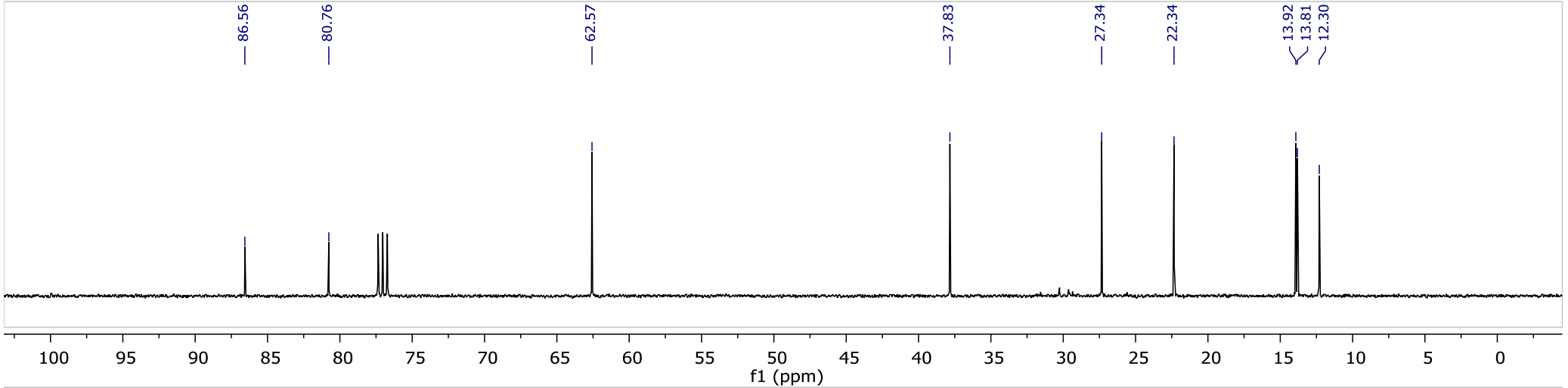
Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		13.802	1465400.63	45968.97	98.25	98.25			*BB	1.4654	1.4654
2		15.908	26135.29	1449.19	1.75	1.75			*BB	0.0261	0.0261
			1491535.92	47418.16	100.00	100.00				1.4915	1.4915

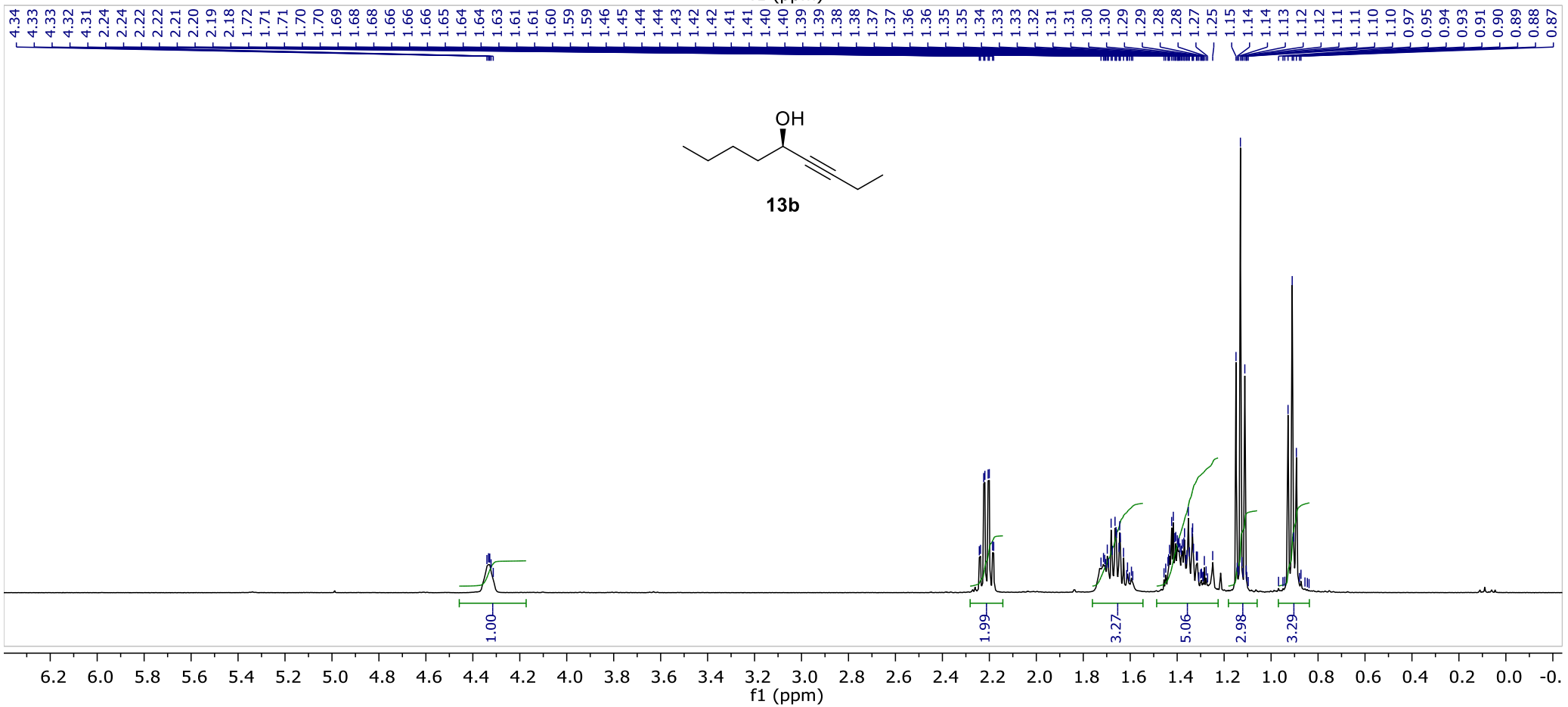
Column: Agilent Cyclosil-B
(30m x 0.25 mm x 0.25 µm)
Injection temp.: 150 °C
Detection temp.: 270 °C
Oven temp: 90 °C (30 min),
5°C/min to 150°, hold for 5.00 min

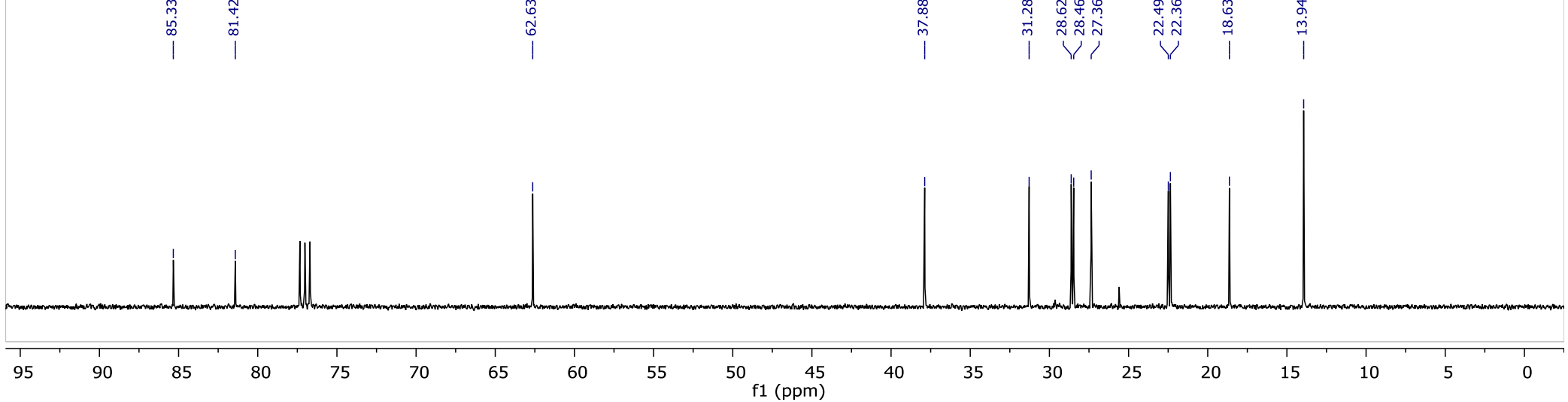

169.84
81.32
73.32
63.76
34.27
26.97
22.16
20.92
13.83
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

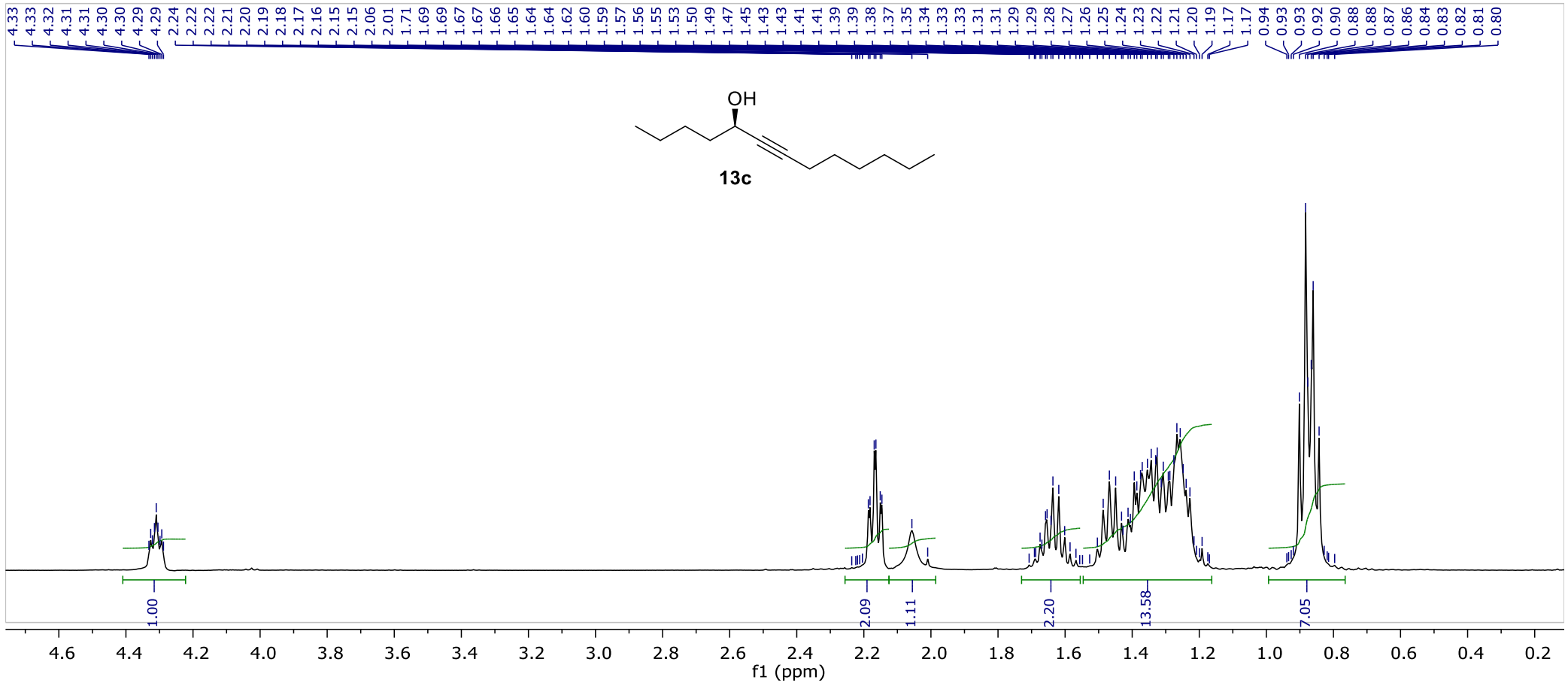

5.36
5.35
5.34
5.34
5.33
5.32
5.29
2.44
2.44
2.43
2.43
2.12
2.10
2.10
2.09
2.09
2.08
2.08
2.07
2.06
2.06
2.05
1.80
1.80
1.78
1.78
1.77
1.77
1.76
1.74
1.74
1.45
1.44
1.43
1.42
1.42
1.41
1.41
1.40
1.39
1.38
1.36
1.34
1.32
0.93
0.91
O
O
1.00
0.99
3.17
2.24
4.57
3.35
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)

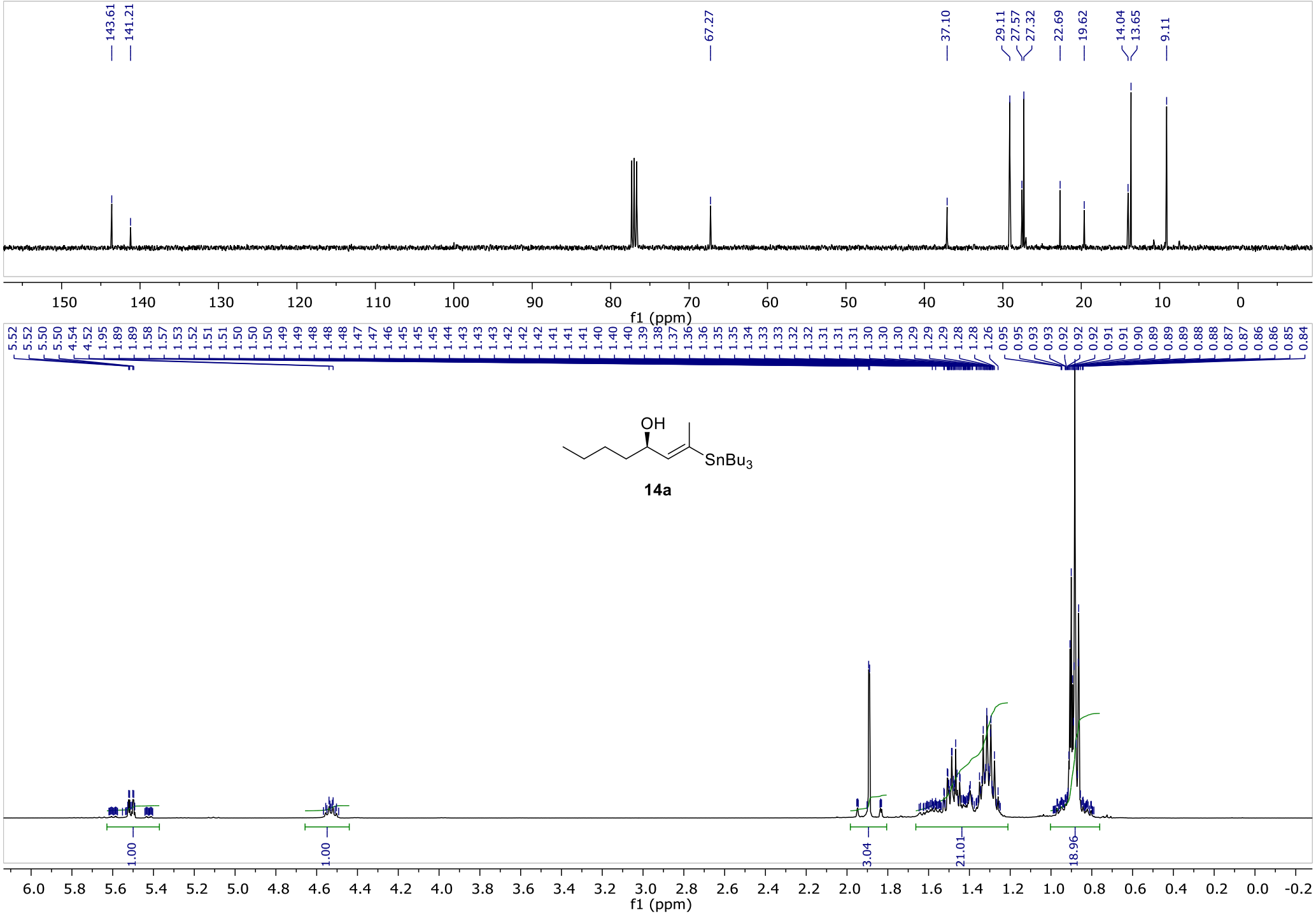

80.73
80.58
62.66
37.82
27.32
22.35
13.94
3.44
f1 (ppm)
OH
13a
1.00
3.10
1.02
1.95
4.11
3.04
f1 (ppm)

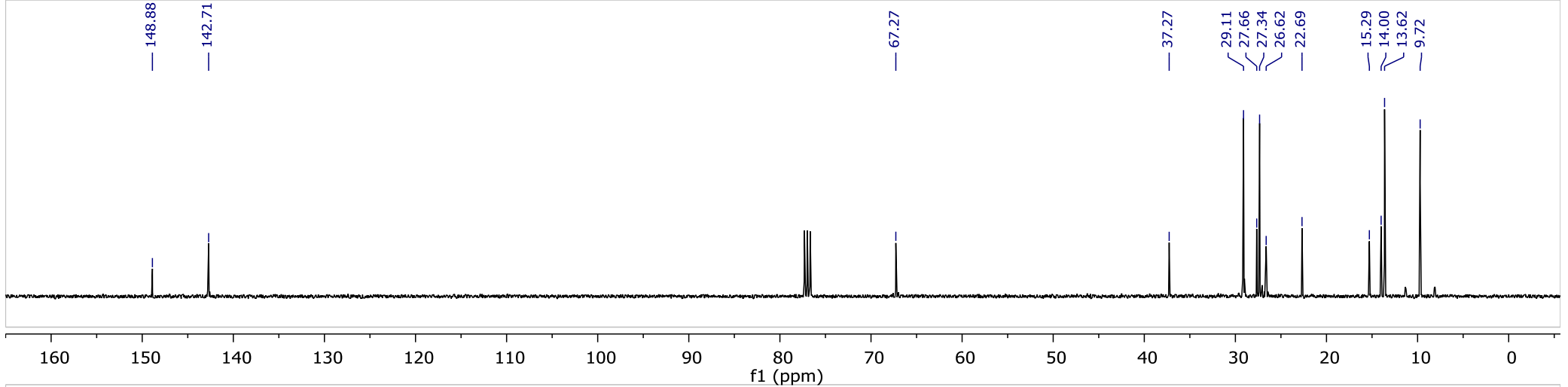

86.56
80.76
62.57
37.83
27.34
22.34
13.92
13.81
12.30
f1 (ppm)

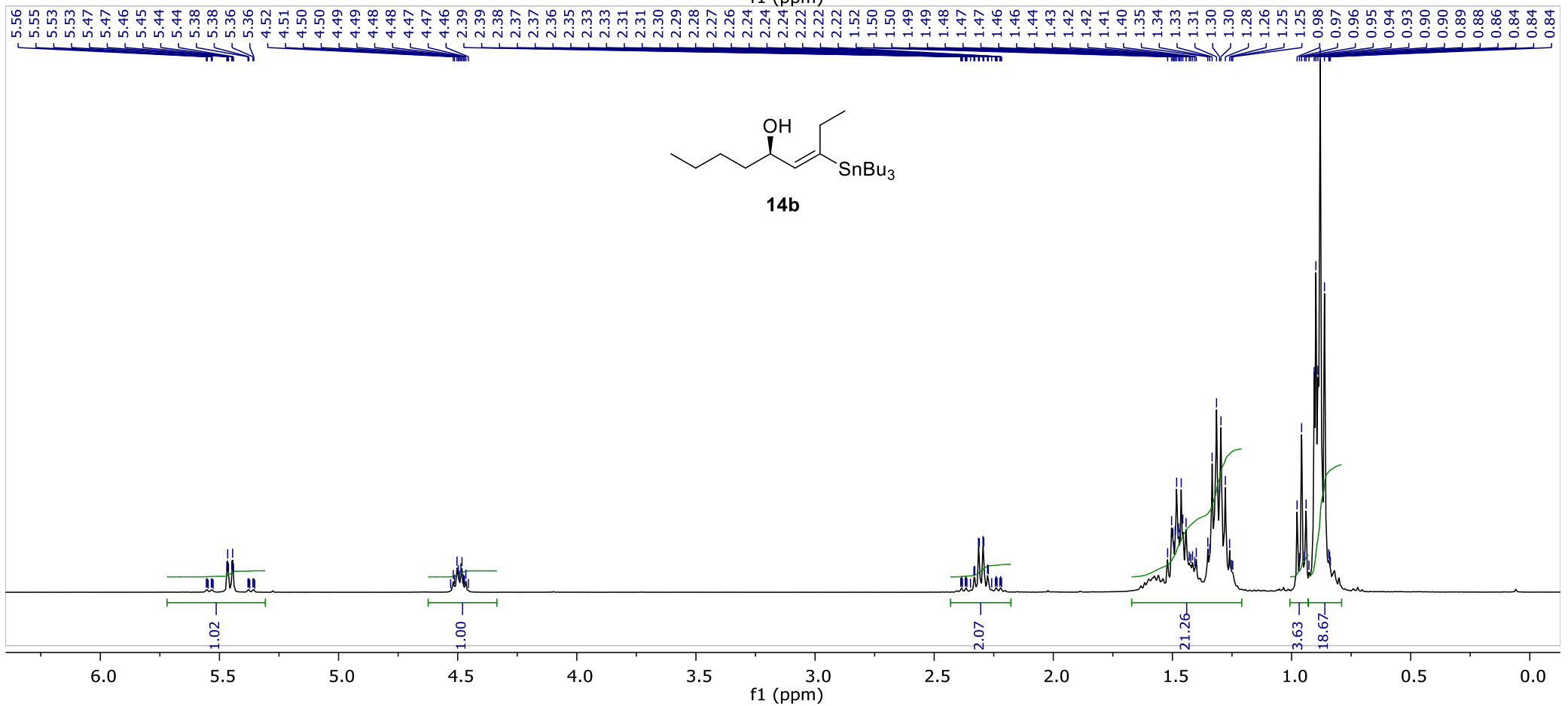

OH
13b
1.00
1.99
3.27
5.06
2.98
3.29
f1 (ppm)

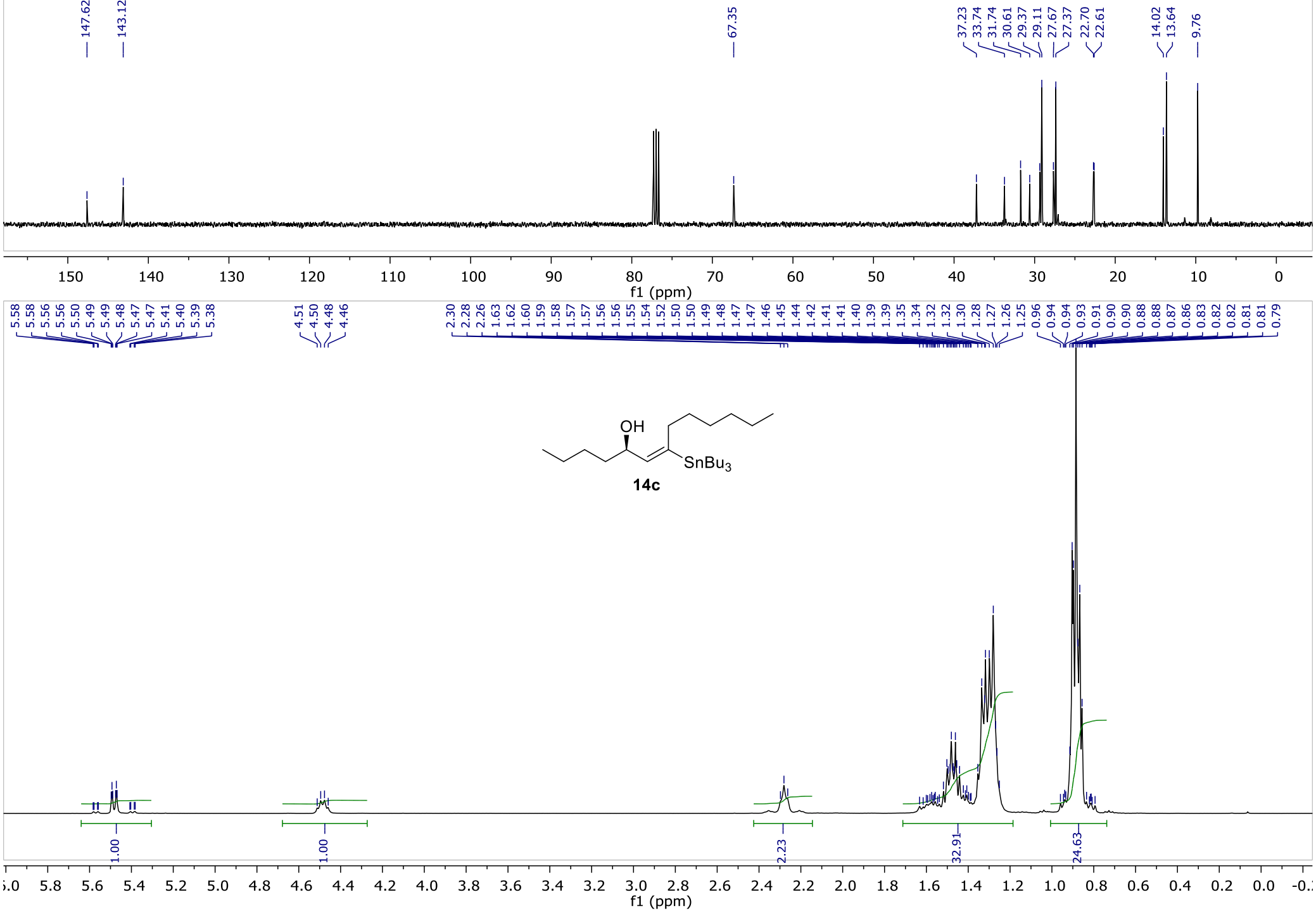

85.33
81.42
62.63
37.88
31.28
28.62
28.46
27.36
22.49
22.36
18.63
13.94
95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

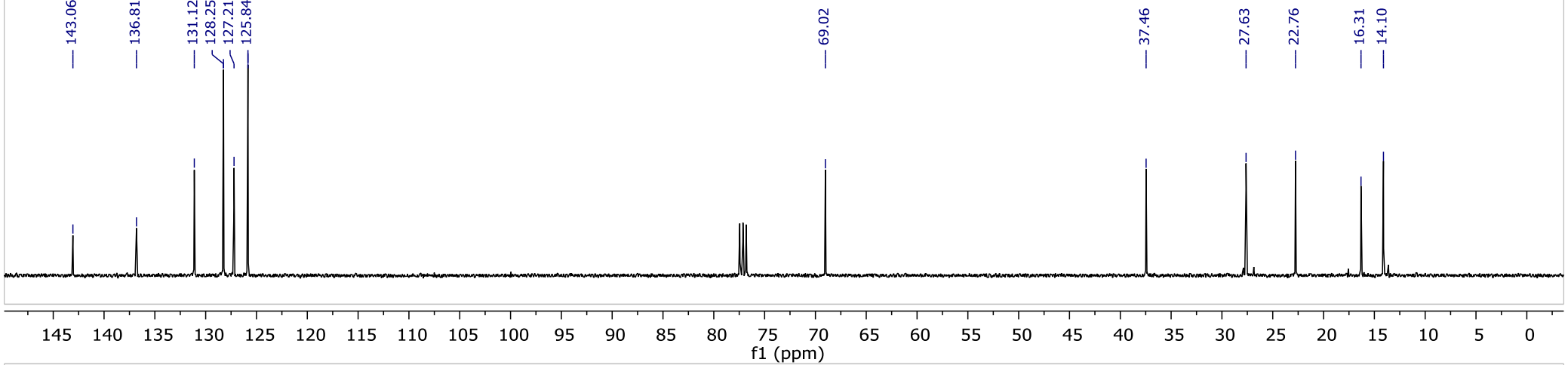

OH
13c
1.00
2.09
1.11
2.20
13.58
7.05
4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 1.6 1.4 1.2 1.0 0.8 0.6 0.4 0.2
f1 (ppm)

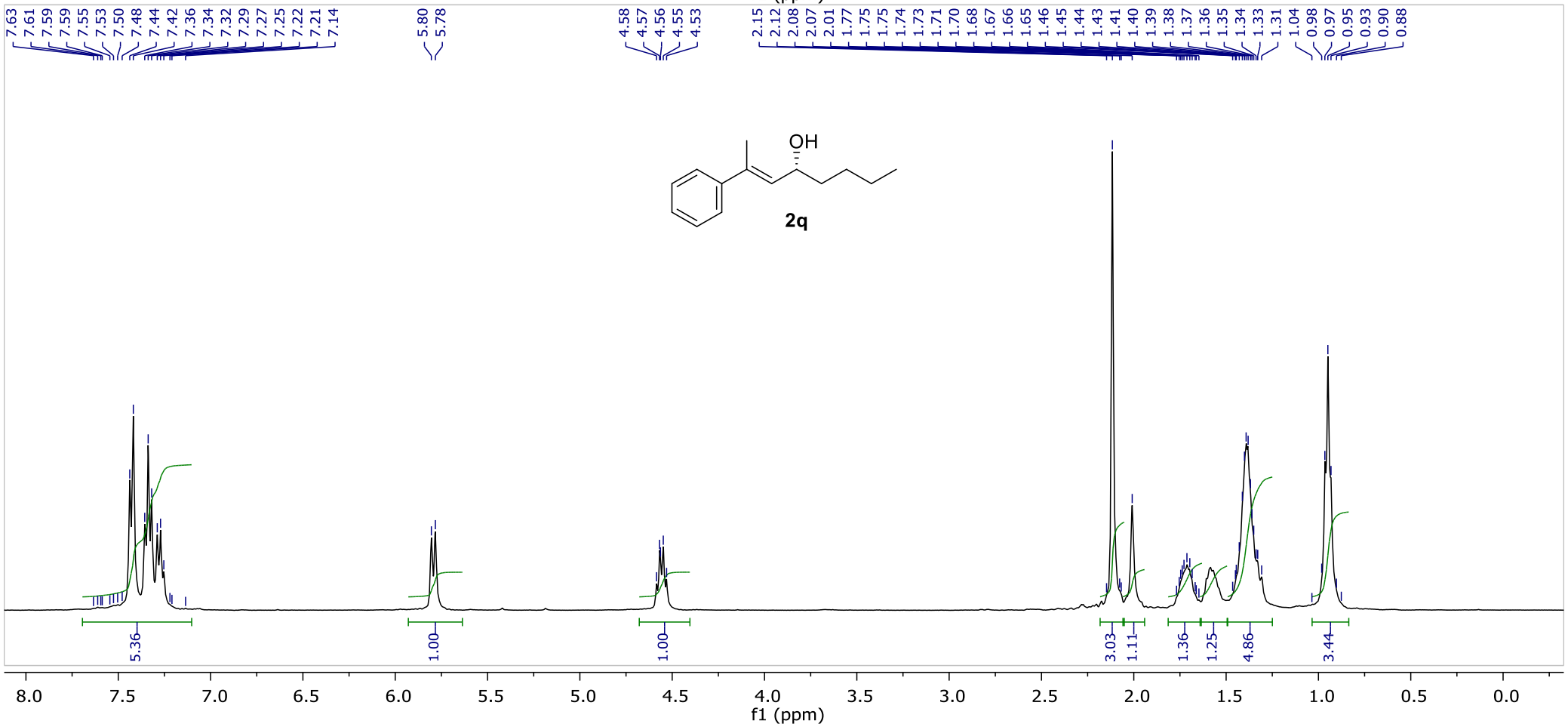

143.61
141.21
67.27
37.10
29.11
27.57
27.32
22.69
19.62
14.04
13.65
9.11
f1 (ppm)
OH
SnBu3
14a
1.00
1.00
3.04
21.01
18.96
f1 (ppm)


148.88
142.71
67.27
37.27
29.11
27.66
27.34
26.62
22.69
15.29
14.00
13.62
9.72
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


OH
SnBu3
14b
1.02
1.00
2.07
21.26
3.63
18.67
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
f1 (ppm)

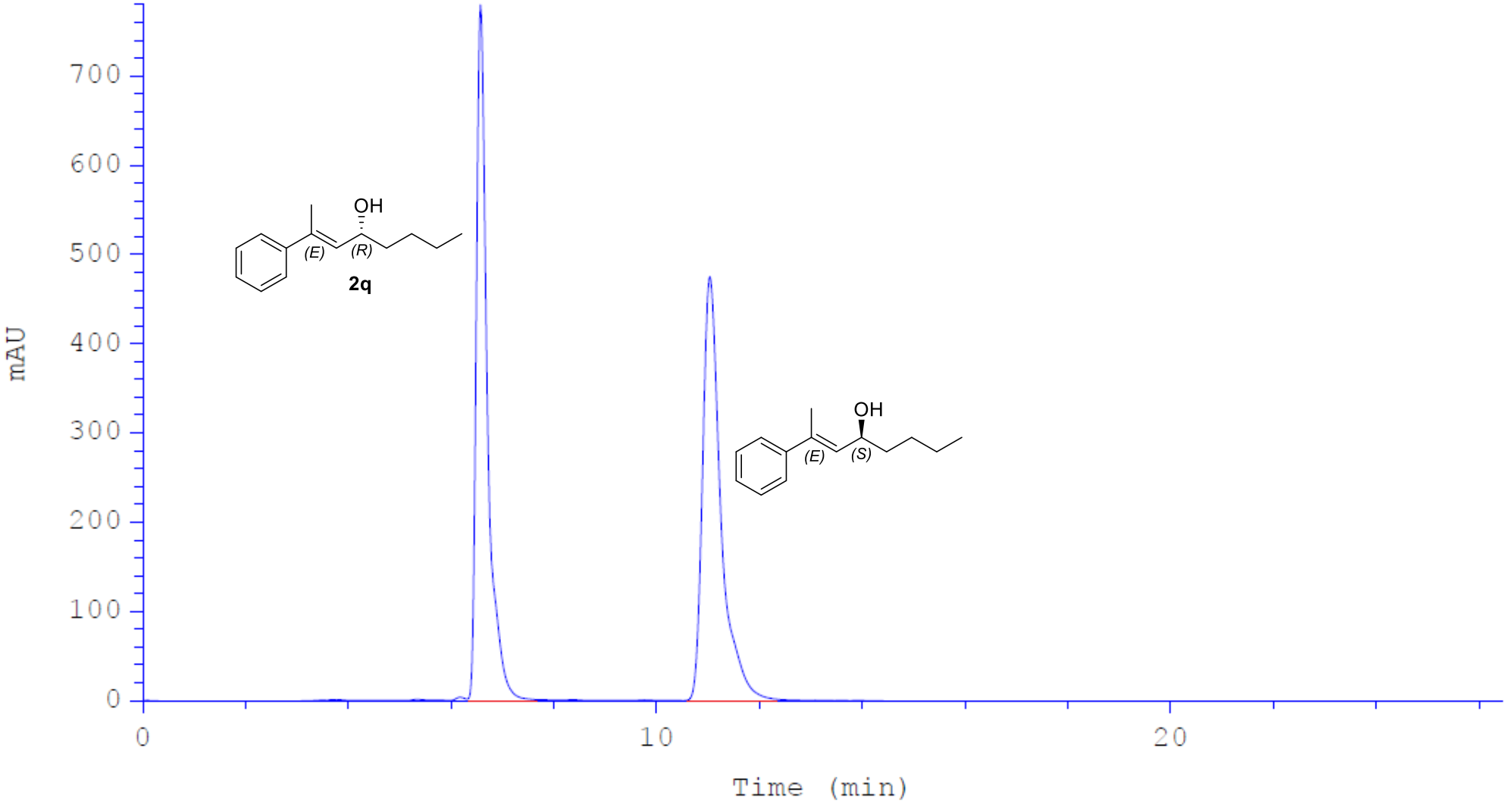

147.62
143.12
67.35
37.23
33.74
31.74
30.61
29.37
29.11
27.67
27.37
22.70
22.61
14.02
13.64
9.76
f1 (ppm)
OH
SnBu3
14c
1.00
1.00
2.23
32.91
24.63
f1 (ppm)


143.06
136.81
131.12
128.25
127.21
125.84
69.02
37.46
27.63
22.76
16.31
14.10
145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)


7.63 7.61 7.59 7.59 7.55 7.53 7.50 7.48 7.44 7.42 7.36 7.34 7.32 7.29 7.27 7.25 7.22 7.21 7.14
5.80 5.78
4.58 4.57 4.56 4.55 4.53
2.15 2.12 2.08 2.07 2.01 1.77 1.75 1.75 1.74 1.73 1.71 1.70 1.68 1.67 1.66 1.65 1.46 1.45 1.44 1.43 1.41 1.40 1.39 1.38 1.37 1.36 1.35 1.34 1.33 1.31 1.04 0.98 0.97 0.95 0.93 0.90 0.88
OH
2q
5.36
1.00
1.00
3.03
1.11
1.36
1.25
4.86
3.44
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
f1 (ppm)

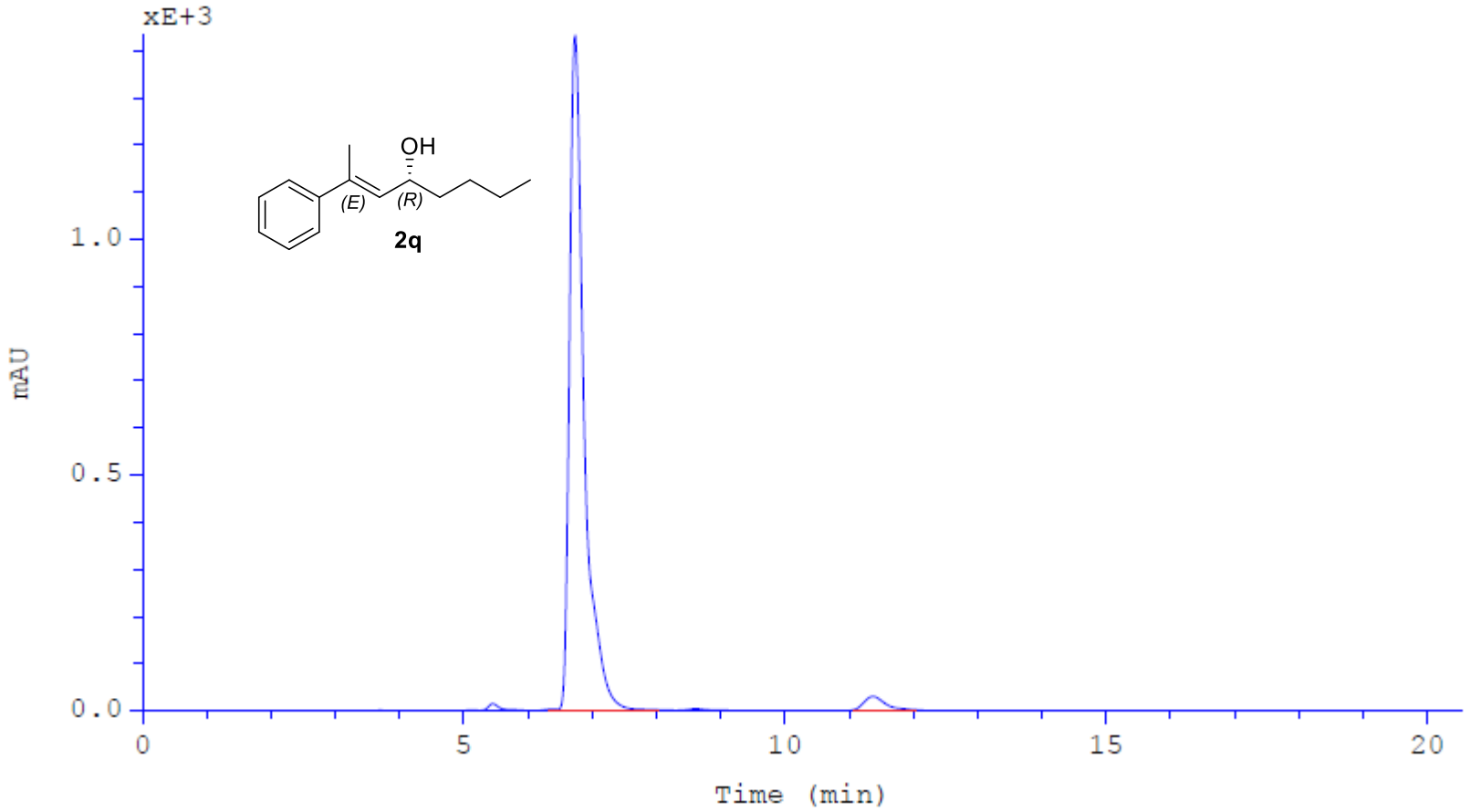
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

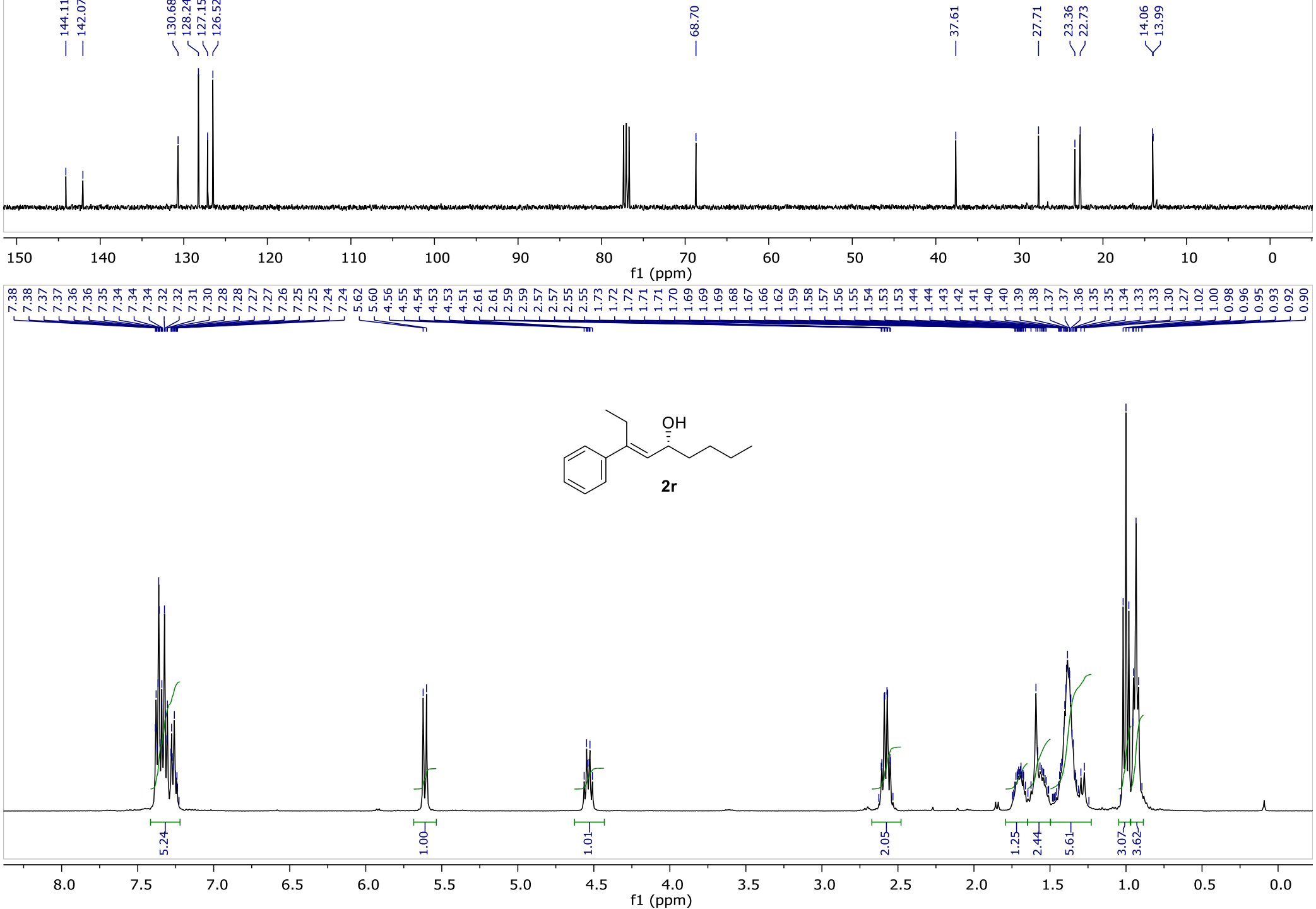
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	6.569	6.32	7.52	191.925	777.776	49.6627	0.205
2	11.037	10.58	12.28	194.532	474.402	50.3373	0.343



Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

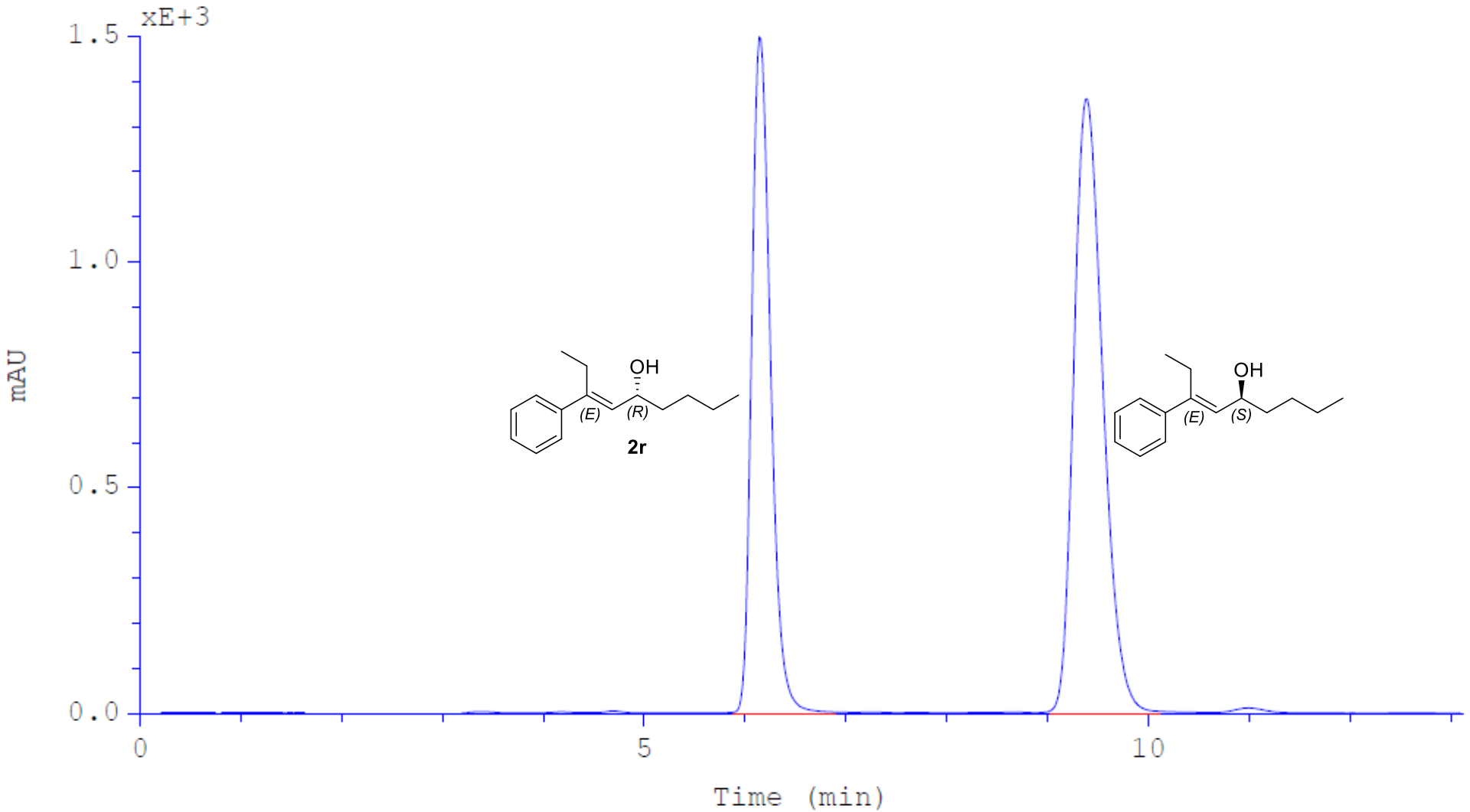
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	6.737	6.24	8.08	376.616	1431.04	97.0847	0.217
2	11.373	11.03	12.09	11.309	29.3863	2.9153	0.337




144.11
142.07
130.68
128.24
127.15
126.52
68.70
37.61
27.71
23.36
22.73
14.06
13.99
f1 (ppm)
OH
2r
5.24
1.00
1.01
2.05
1.25
2.44
5.61
3.07
3.62
f1 (ppm)

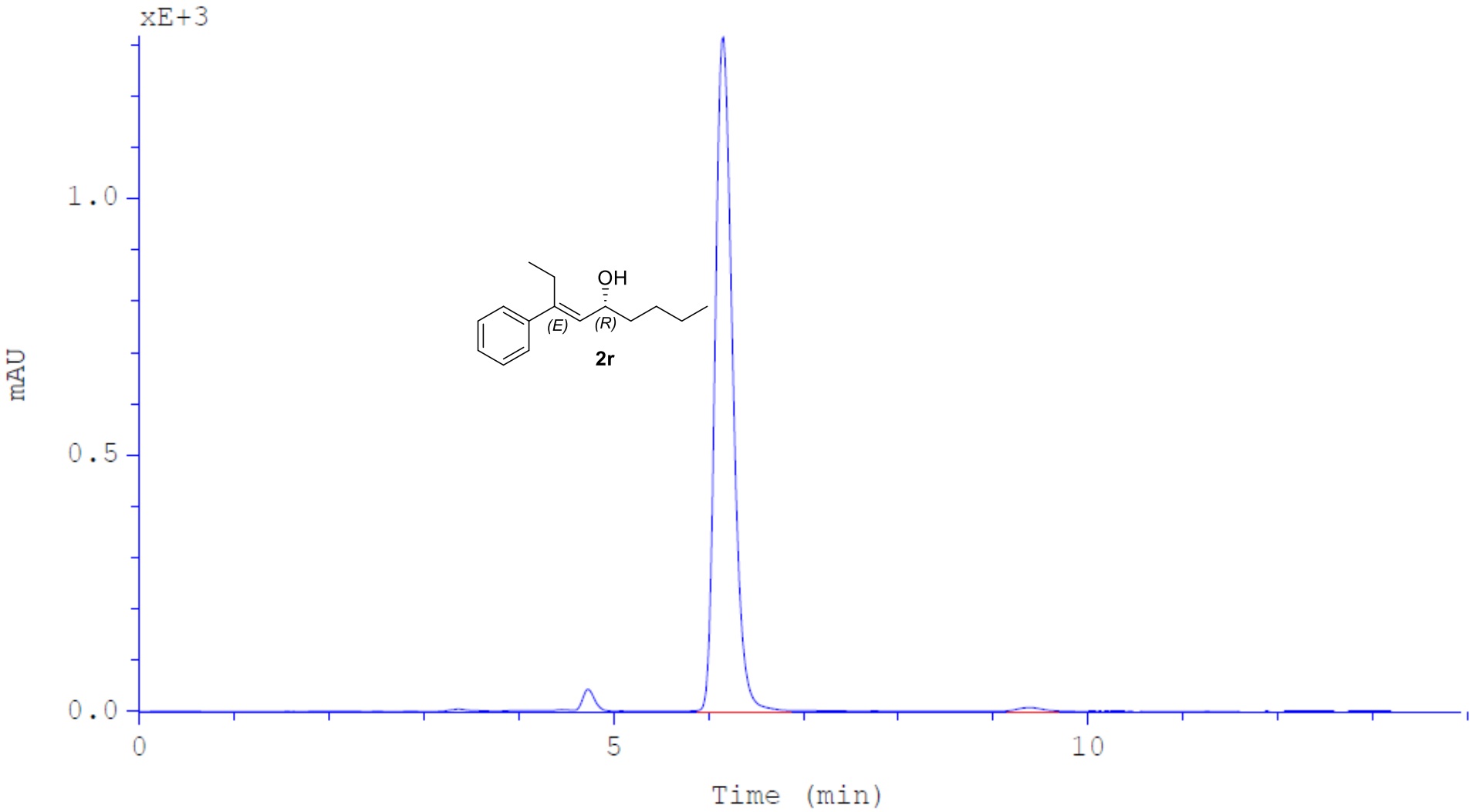
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

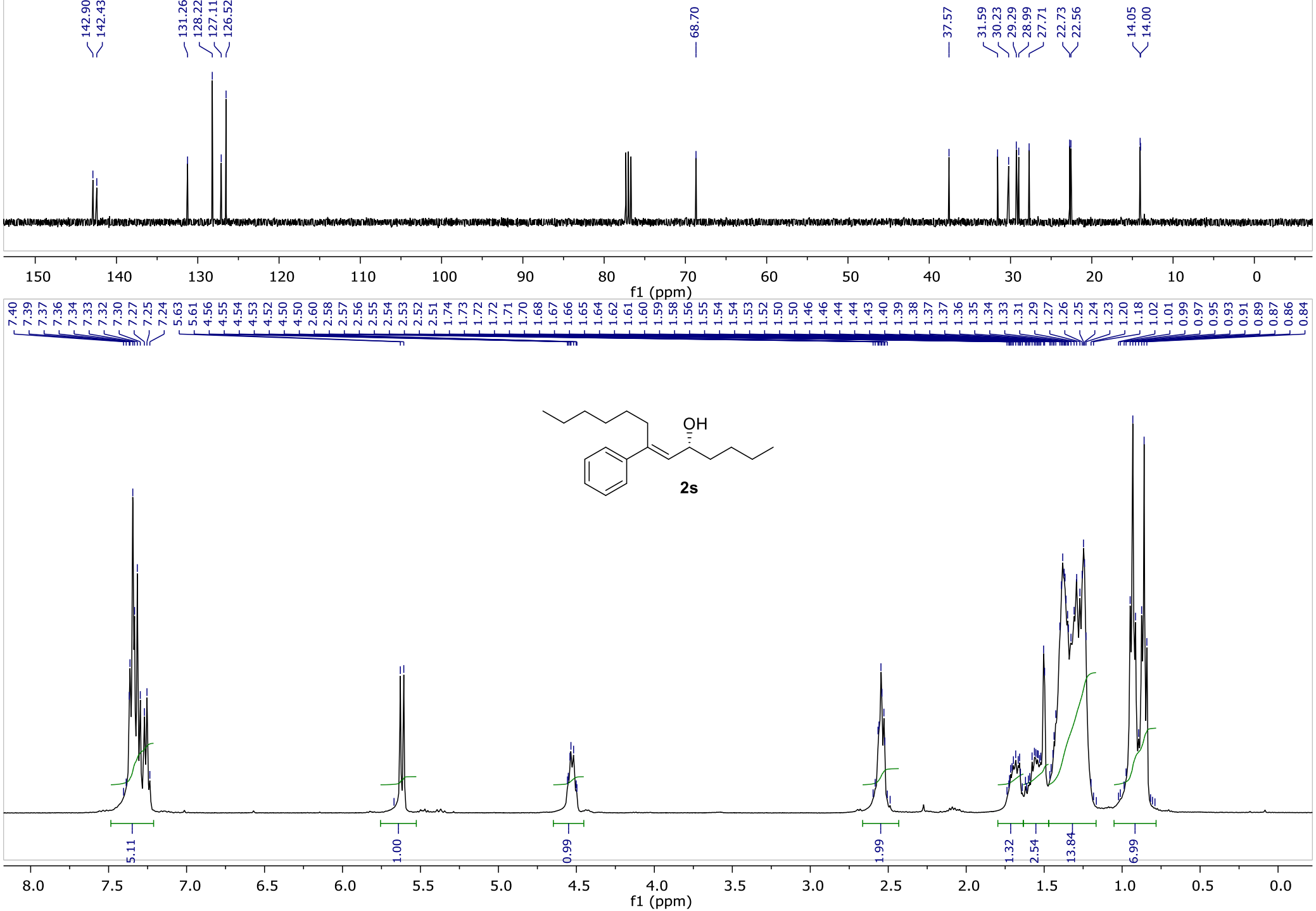
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	6.140	5.90	6.72	321.278	1494.72	41.6499	0.199
2	9.394	9.02	10.07	450.1	1357.15	58.3501	0.309



Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

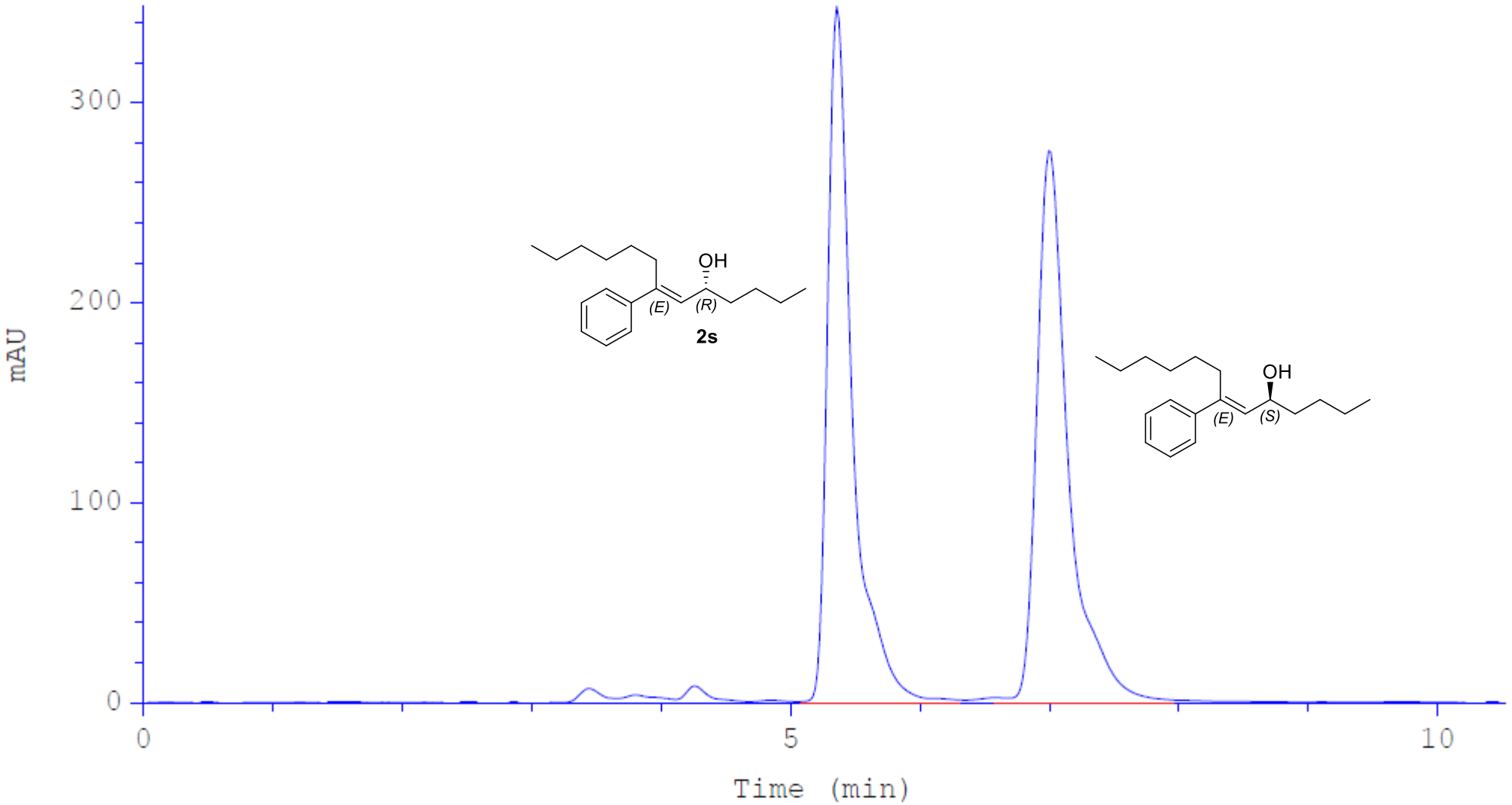
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	6.156	5.88	6.75	276.973	1313.17	99.6549	0.195
2	9.376	9.23	9.58	0.959164	4.65585	0.3451	0.200




142.90
142.43
131.26
128.22
127.11
126.52
68.70
37.57
31.59
30.23
29.29
28.99
27.71
22.73
22.56
14.05
14.00
f1 (ppm)
OH
2s
5.11
1.00
0.99
1.99
1.32
2.54
13.84
6.99
f1 (ppm)

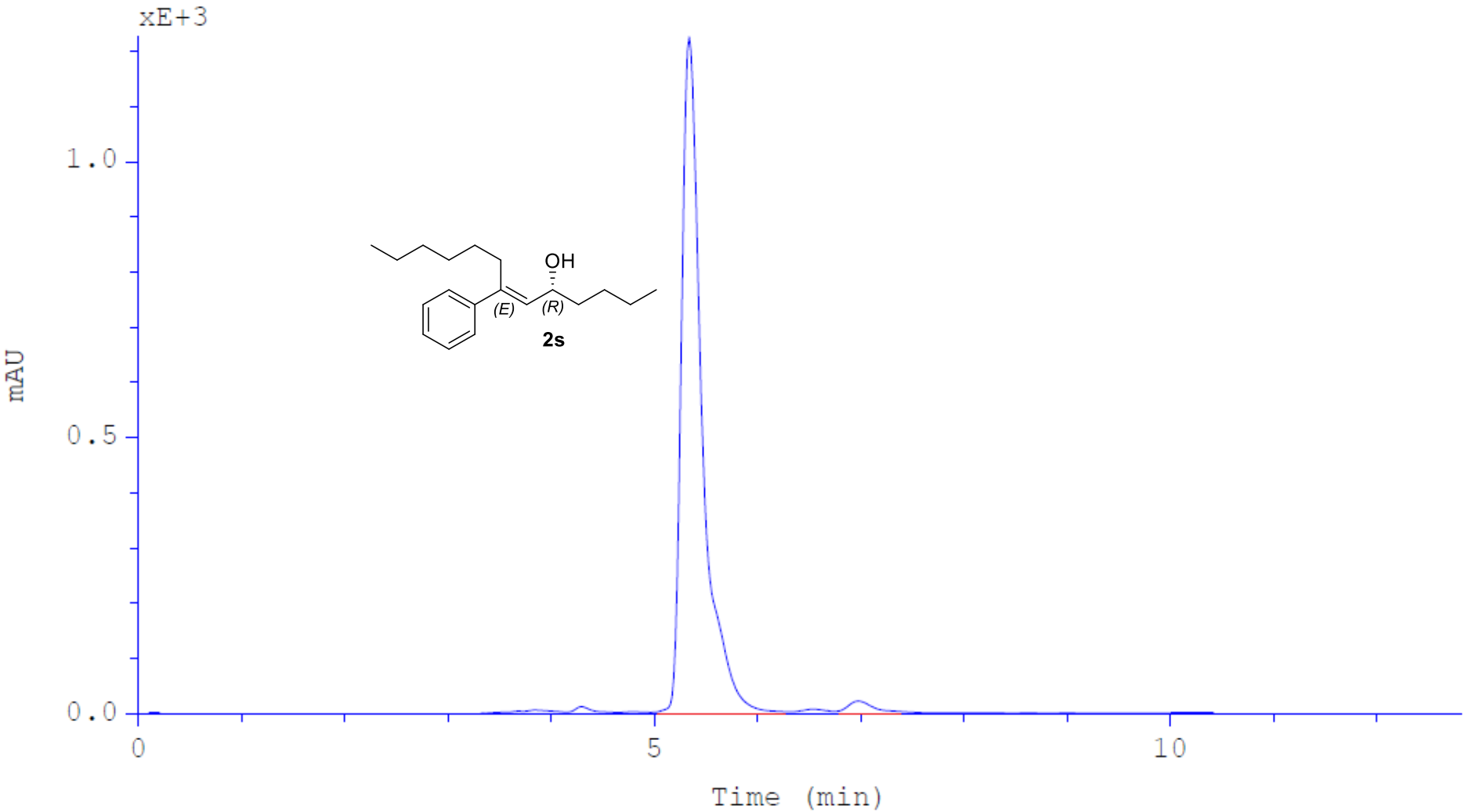
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

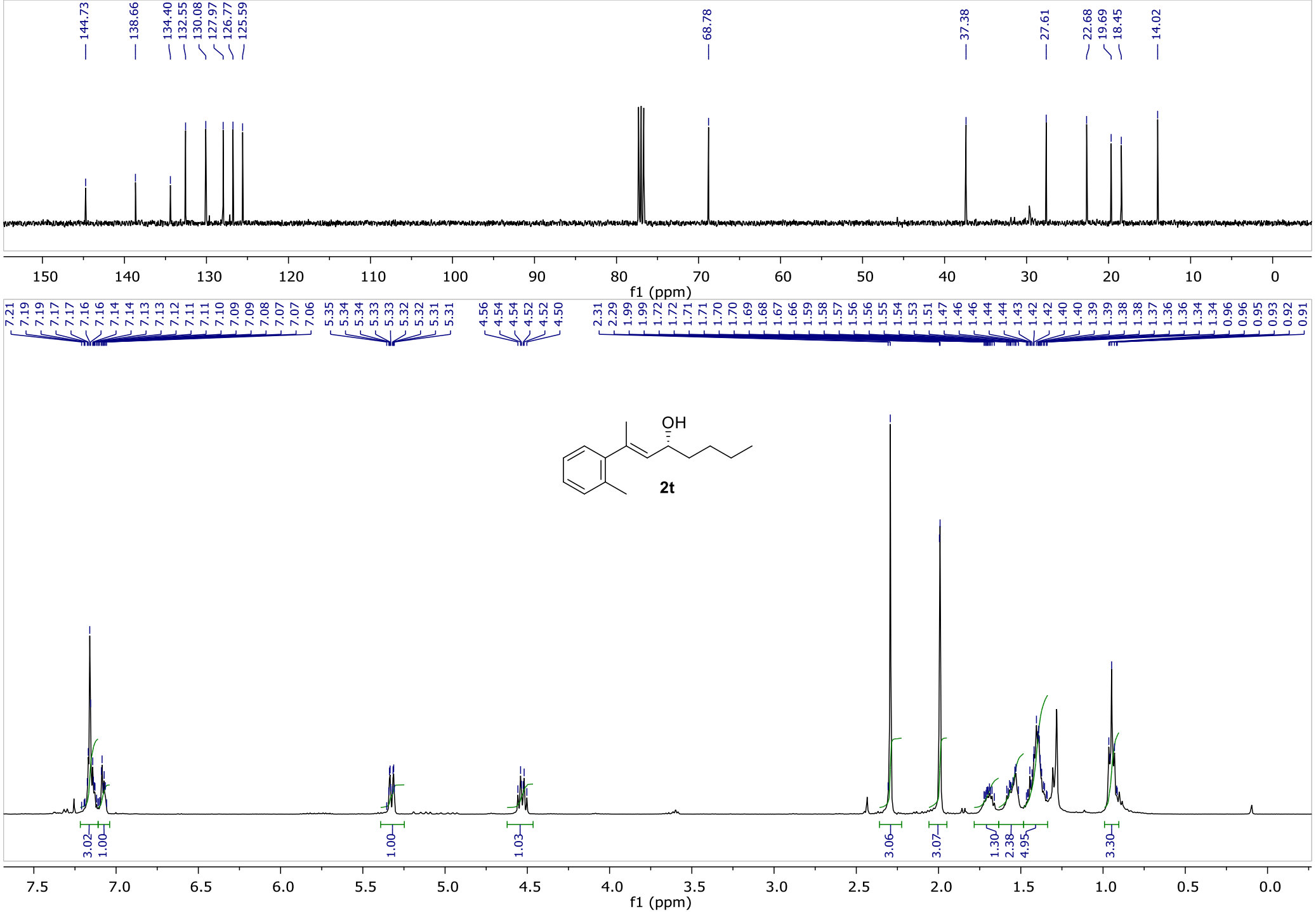
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	5.357	5.12	6.28	76.3848	347.354	49.9300	0.180
2	6.993	6.42	7.94	76.599	274.639	50.0700	0.232



Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

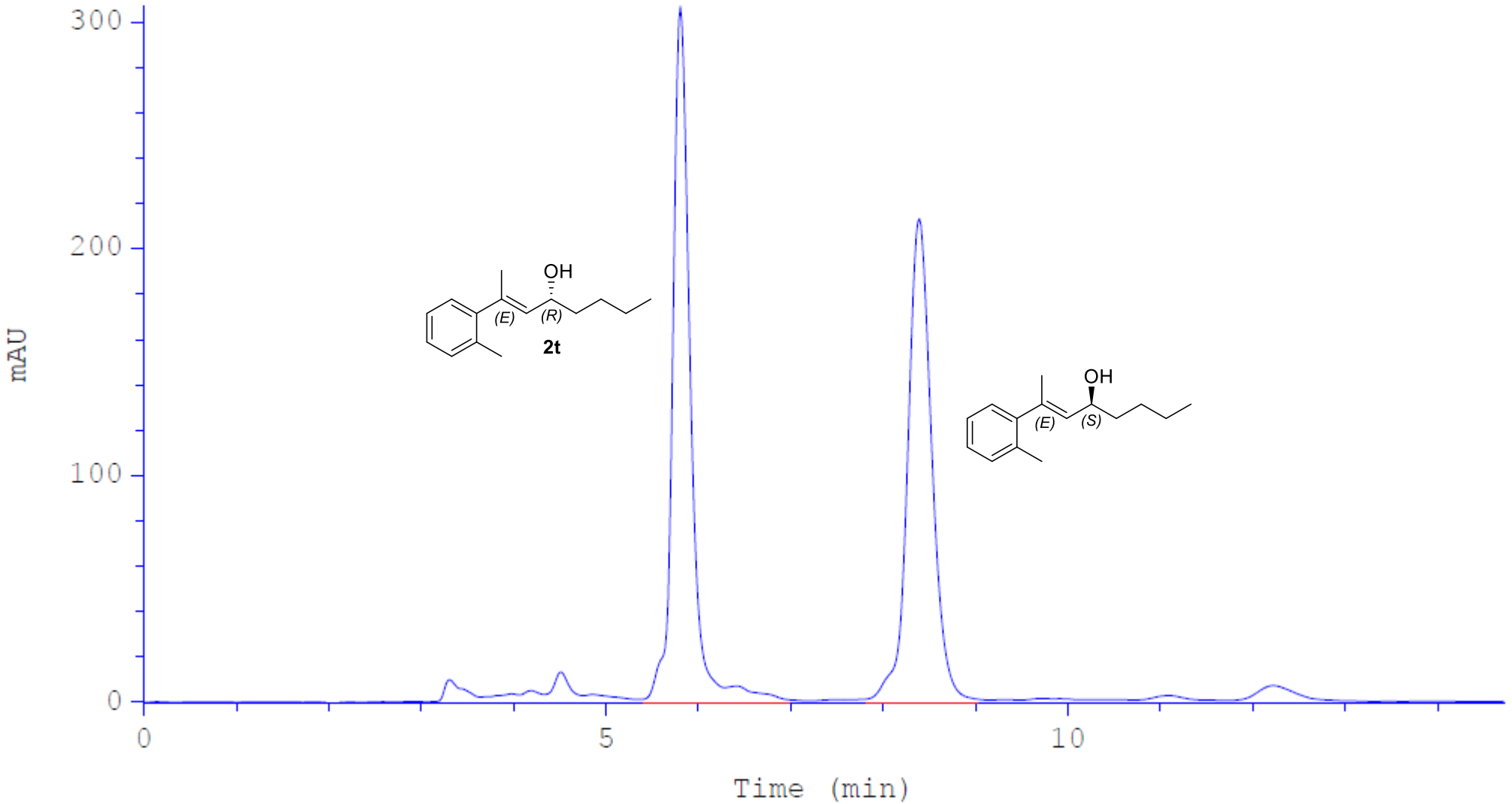
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	5.339	5.05	6.22	281.65	1222.52	98.5749	0.192
2	6.974	6.80	7.37	4.07188	17.7057	1.4251	0.220




144.73
138.66
134.40
132.55
130.08
127.97
126.77
125.59
68.78
37.38
27.61
22.68
19.69
18.45
14.02
f1 (ppm)
OH
2t
3.02
1.00
1.00
1.03
3.06
3.07
1.30
2.38
4.95
3.30
f1 (ppm)

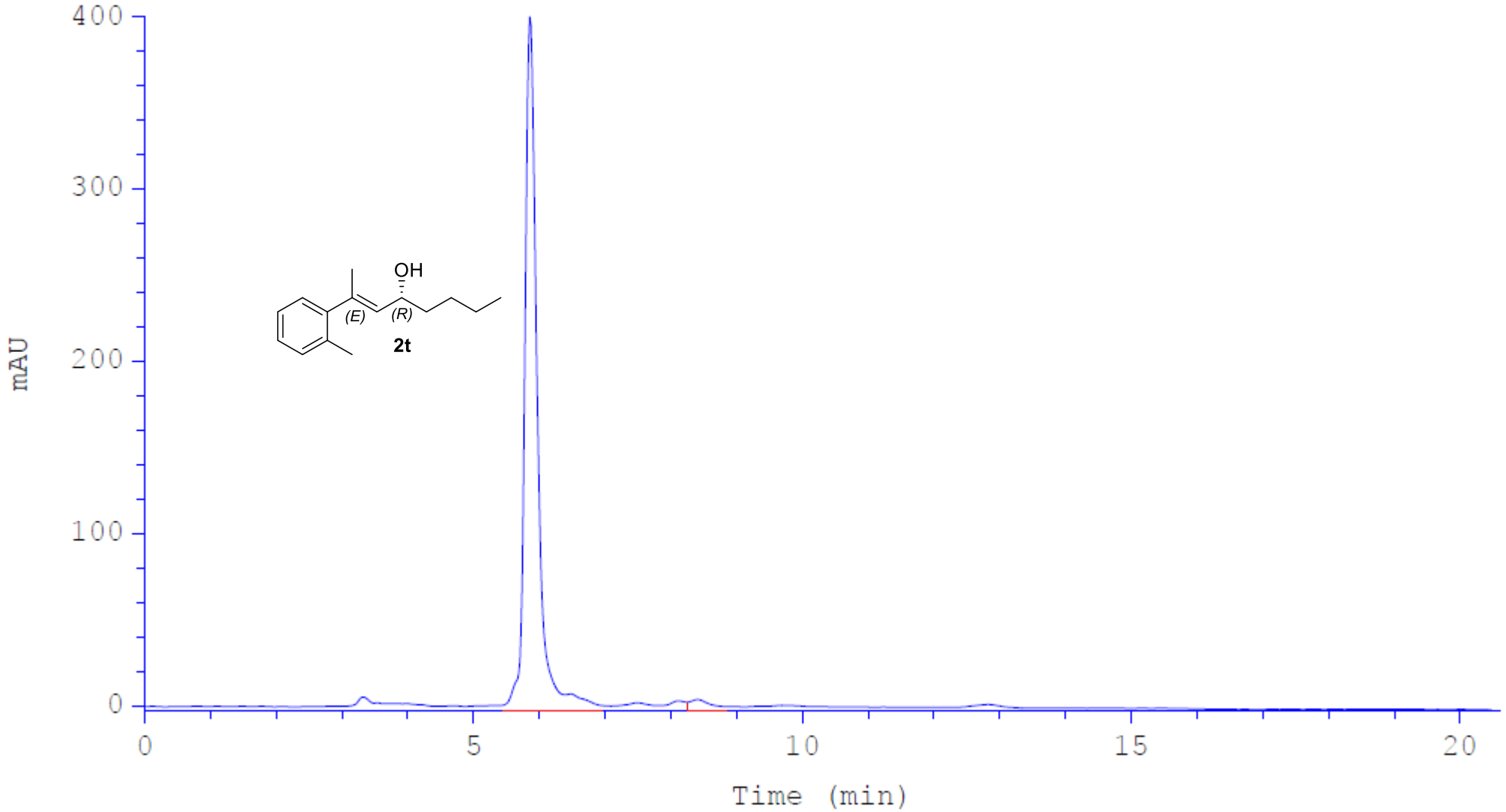
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

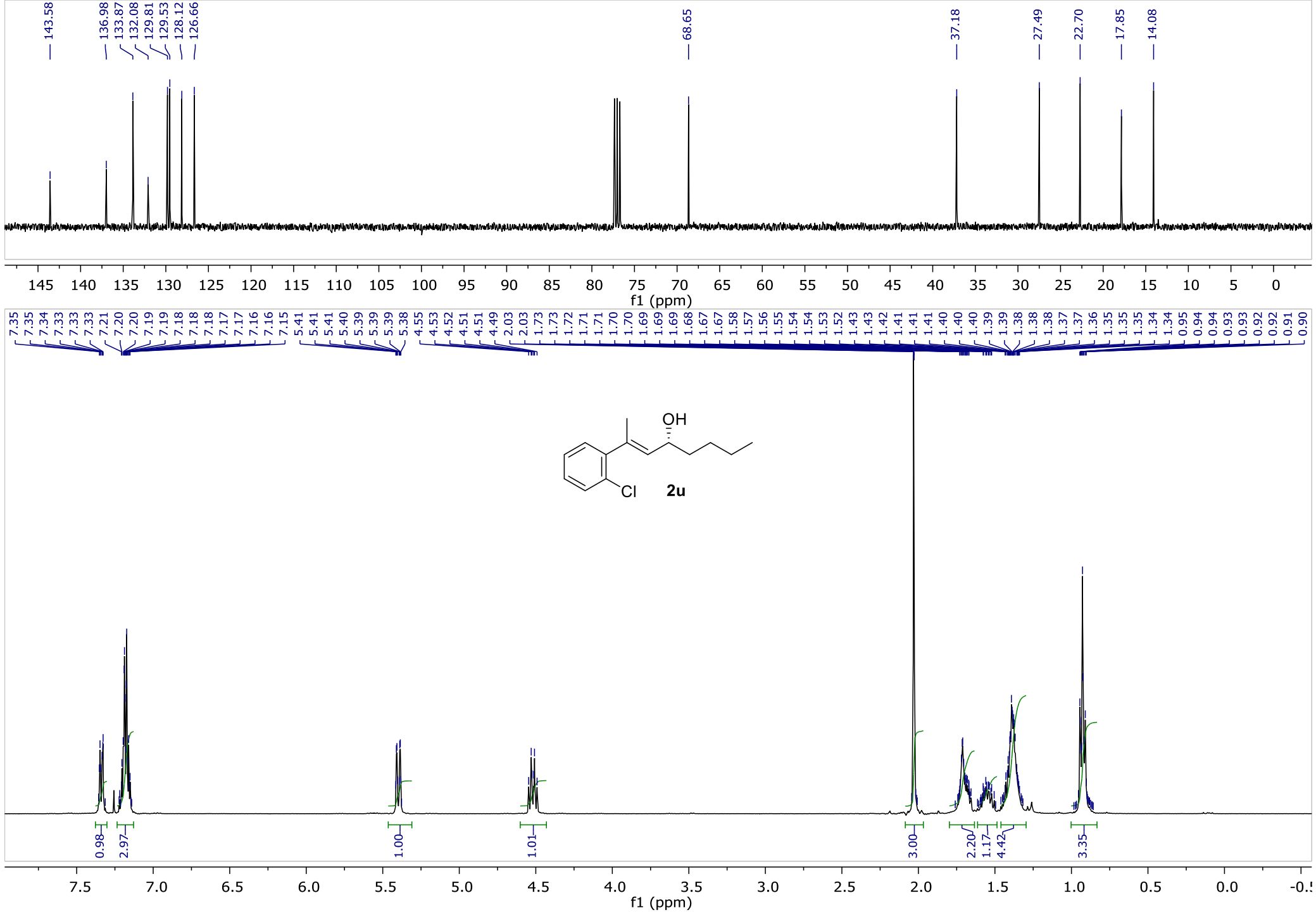
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	5.806	5.43	6.93	66.6667	305.35	51.2678	0.185
2	8.391	7.80	9.02	63.3695	212.105	48.7322	0.267



Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

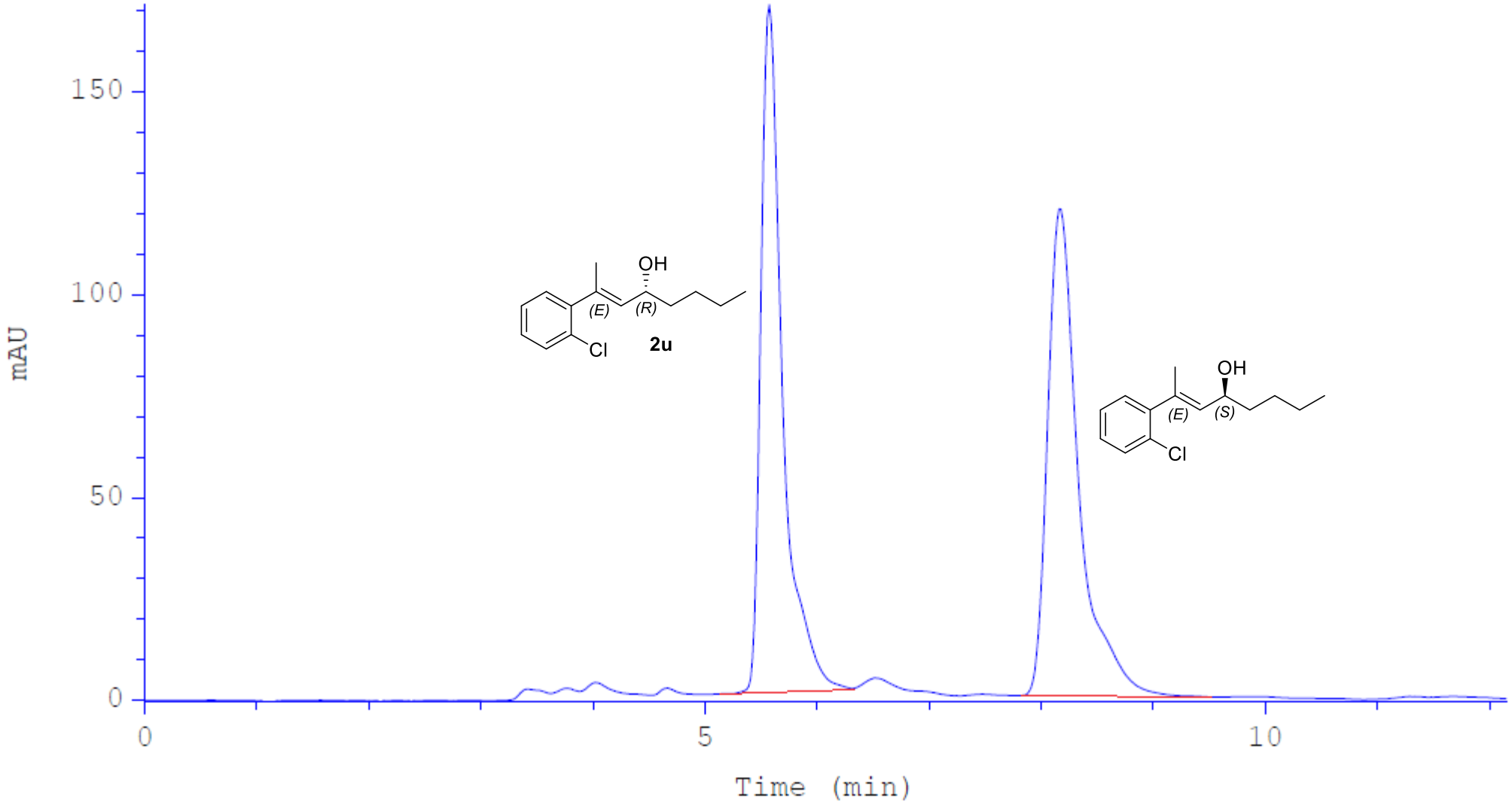
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	5.854	5.48	6.88	84.8736	398.87	99.4449	0.182
2	8.405	8.22	8.82	0.473728	2.27438	0.5551	0.227




143.58
136.98
133.87
132.08
129.81
129.53
128.12
126.66
68.65
37.18
27.49
22.70
17.85
14.08
f1 (ppm)
OH
Cl
2u
0.98
2.97
1.00
1.01
3.00
2.20
1.17
4.42
3.35
f1 (ppm)

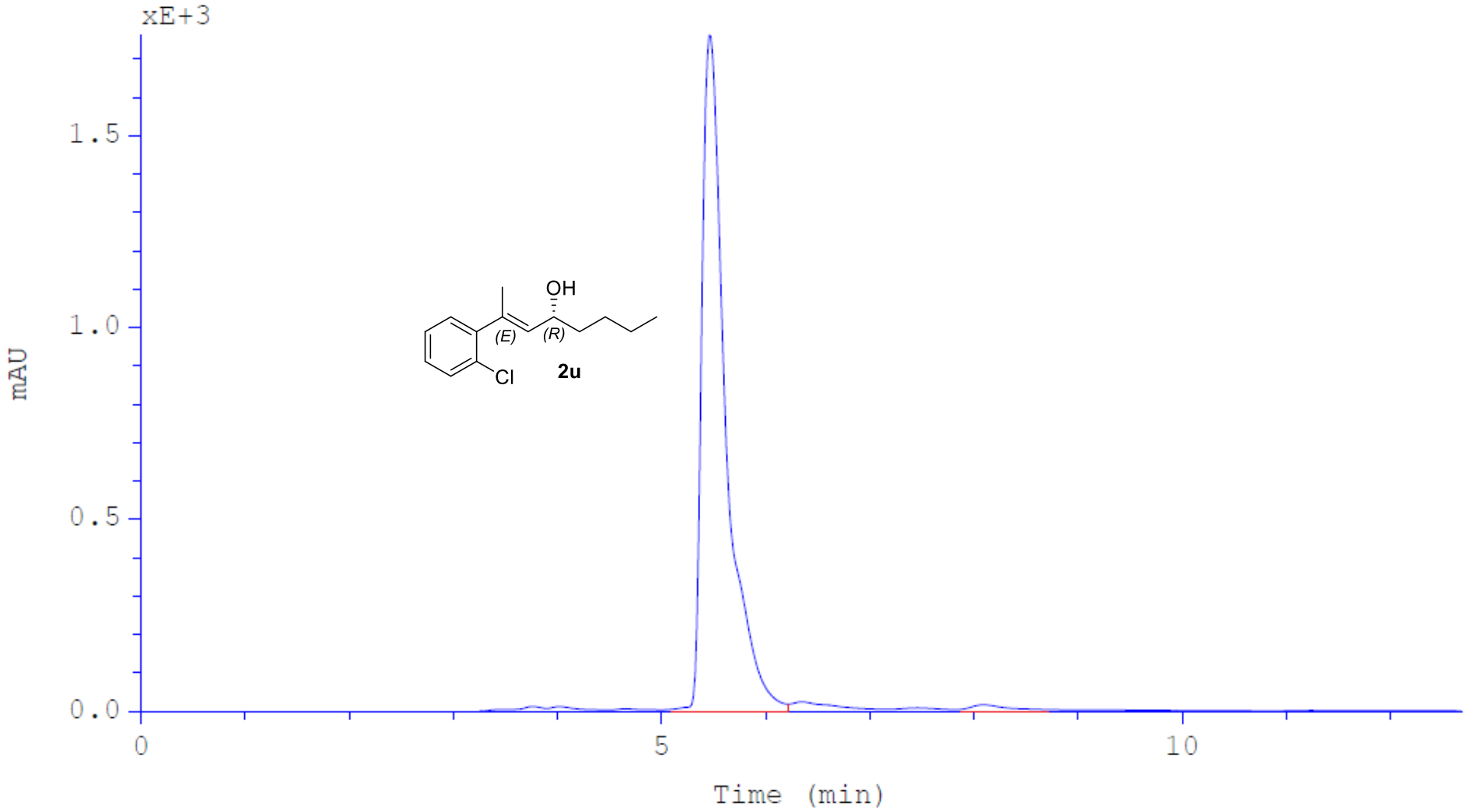
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

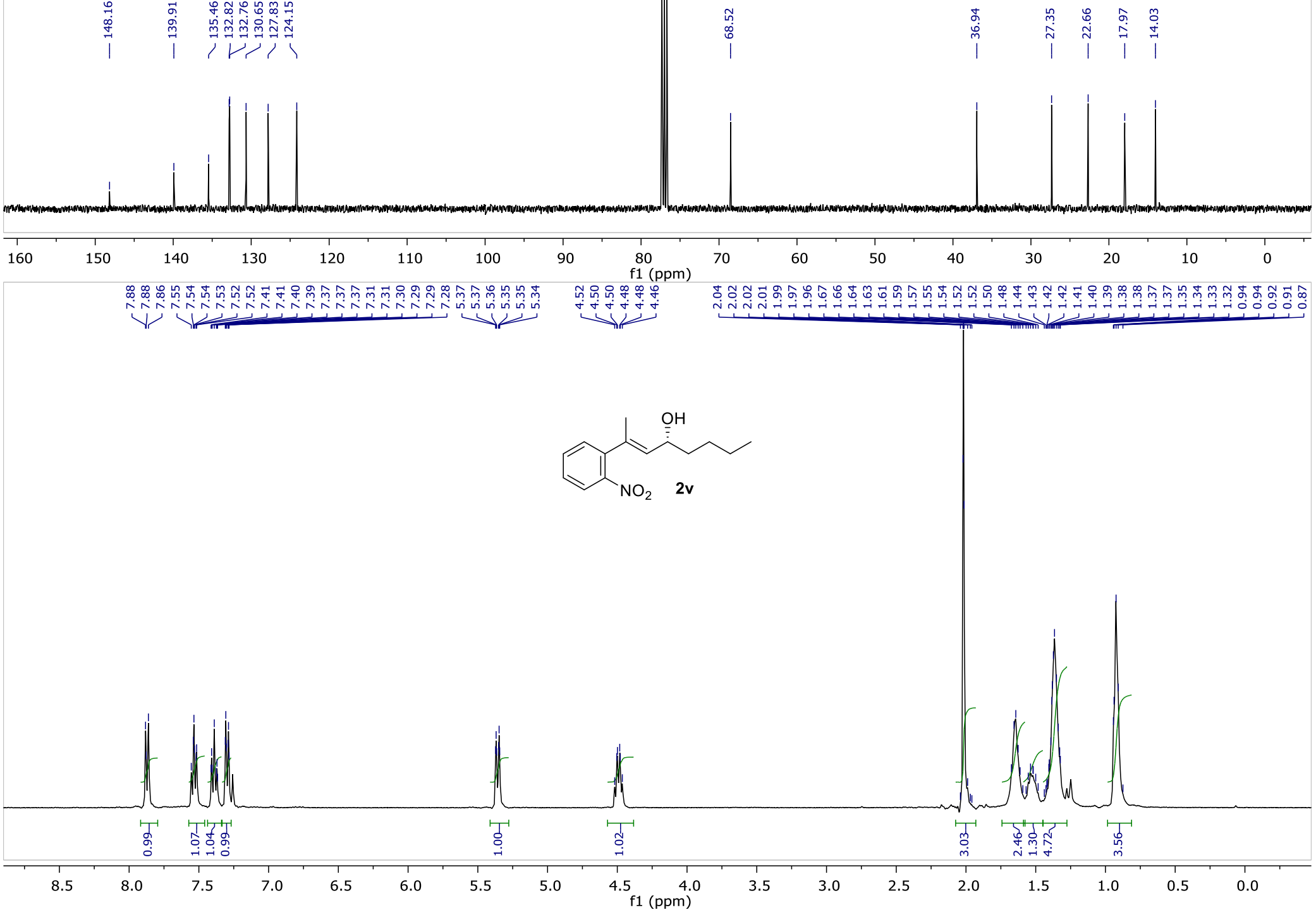
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	5.573	5.11	6.33	37.9536	169.363	49.5321	0.187
2	8.160	7.84	9.44	38.6707	120.028	50.4679	0.269



Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

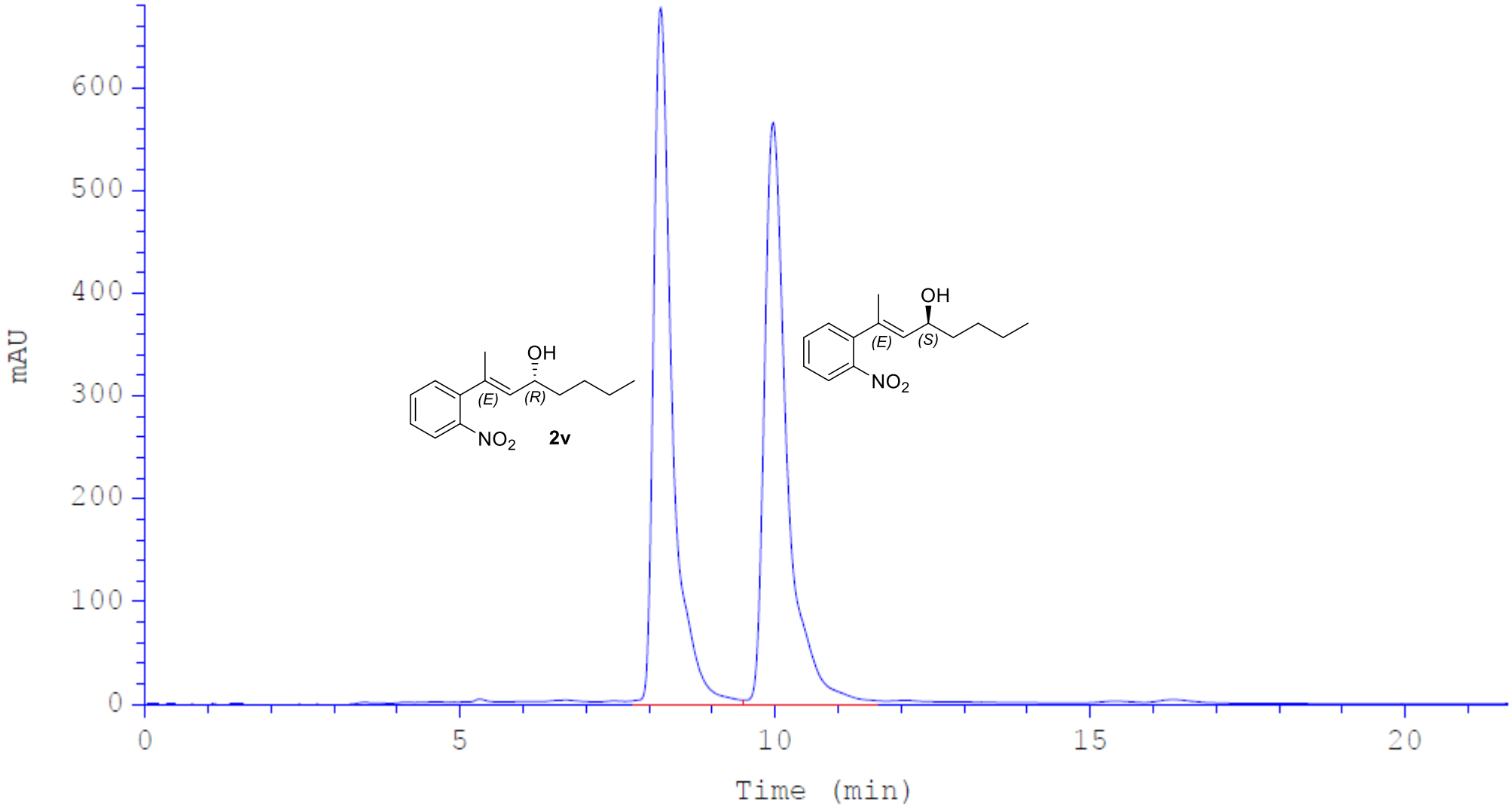
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	5.456	5.05	6.23	456.631	1751.89	99.4594	0.217
2	8.092	7.94	8.72	2.48212	9.54315	0.5406	0.259




148.16
139.91
135.46
132.82
132.76
130.65
127.83
124.15
68.52
36.94
27.35
22.66
17.97
14.03
f1 (ppm)
OH
NO2
2v
f1 (ppm)

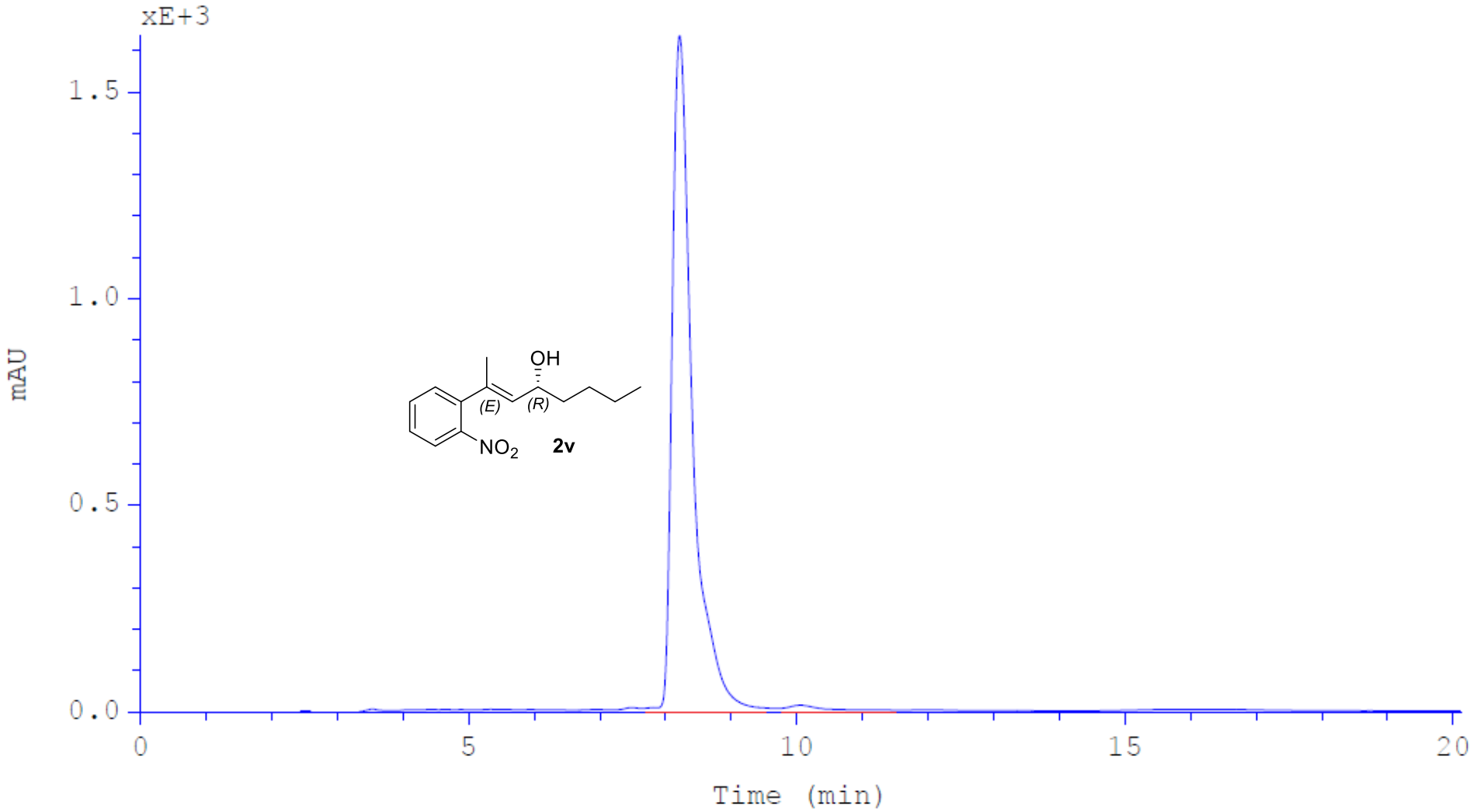
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

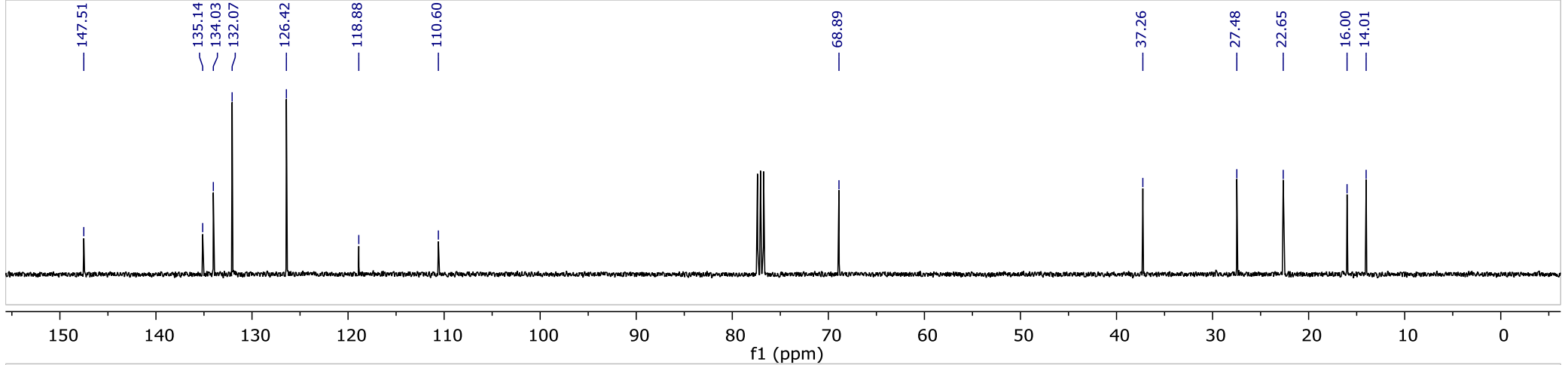
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	8.188	7.80	9.50	226.488	674.353	49.7288	0.281
2	9.973	9.52	11.53	228.958	562.191	50.2712	0.340

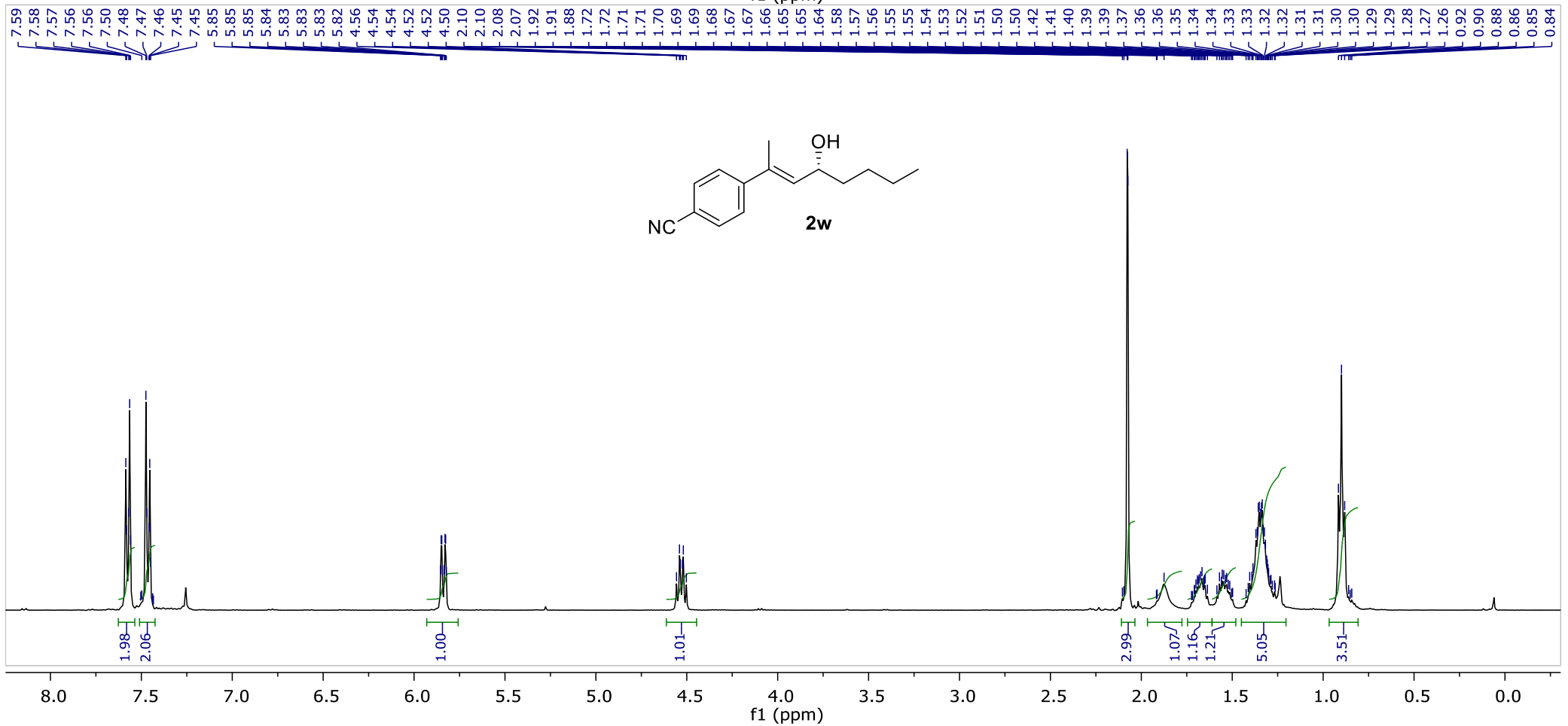


Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	8.222	7.71	9.52	572.109	1626.55	99.6857	0.296
2	10.056	9.80	11.45	1.80382	7.71316	0.3143	0.368

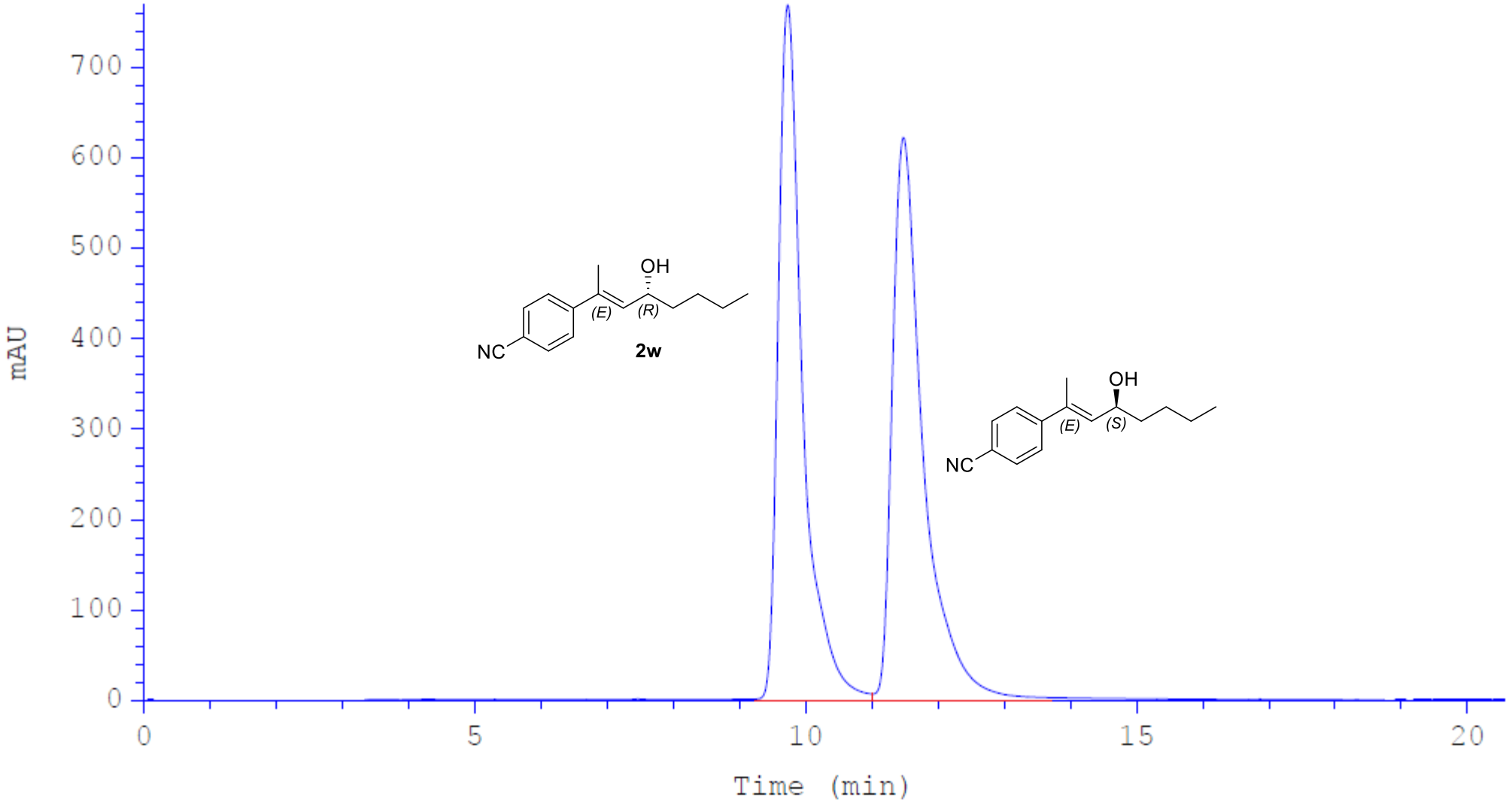



147.51
135.14
134.03
132.07
126.42
118.88
110.60
68.89
37.26
27.48
22.65
16.00
14.01
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


OH
NC
2w
1.98
2.06
1.00
1.01
2.99
1.07
1.16
1.21
5.05
3.51
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
f1 (ppm)

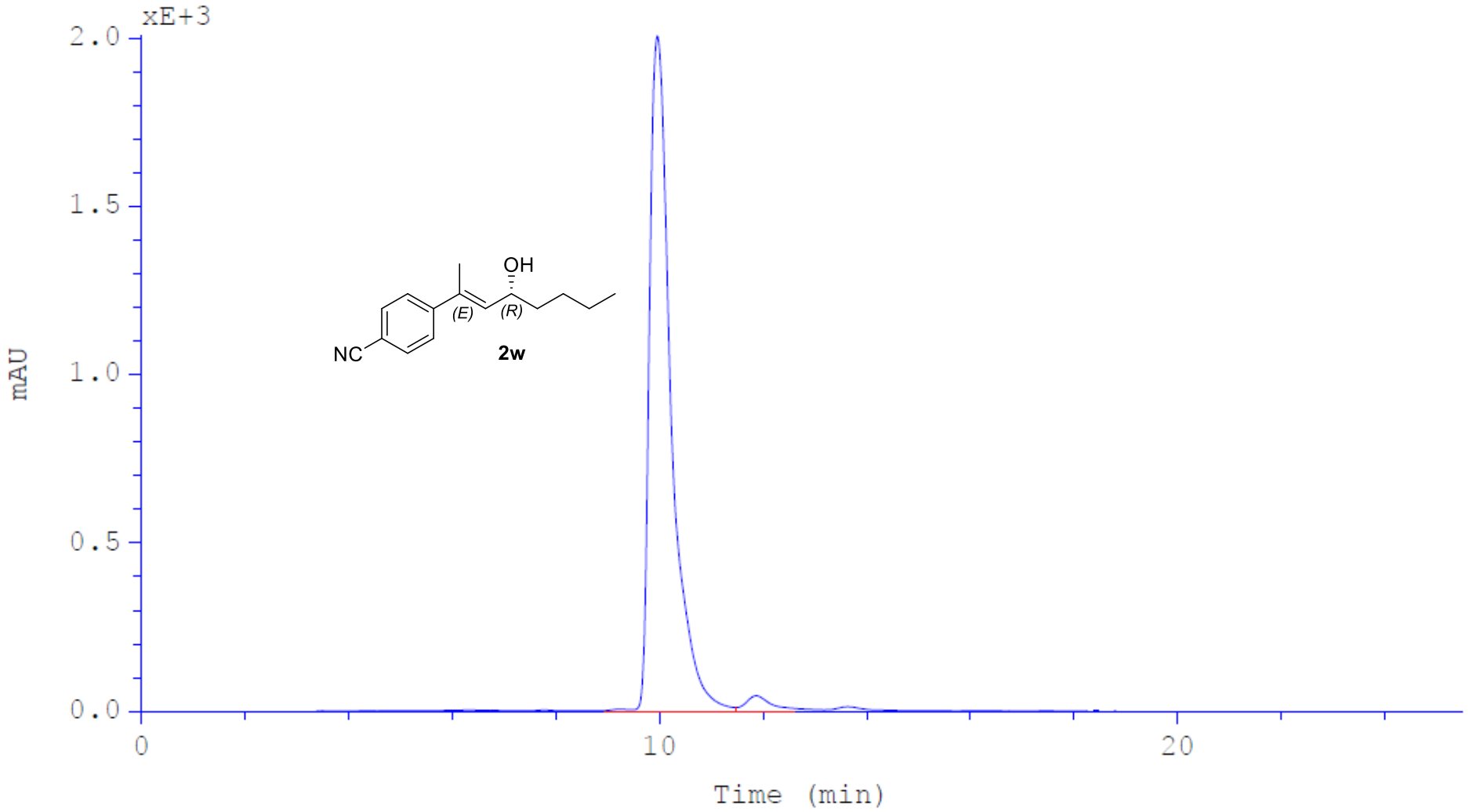
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

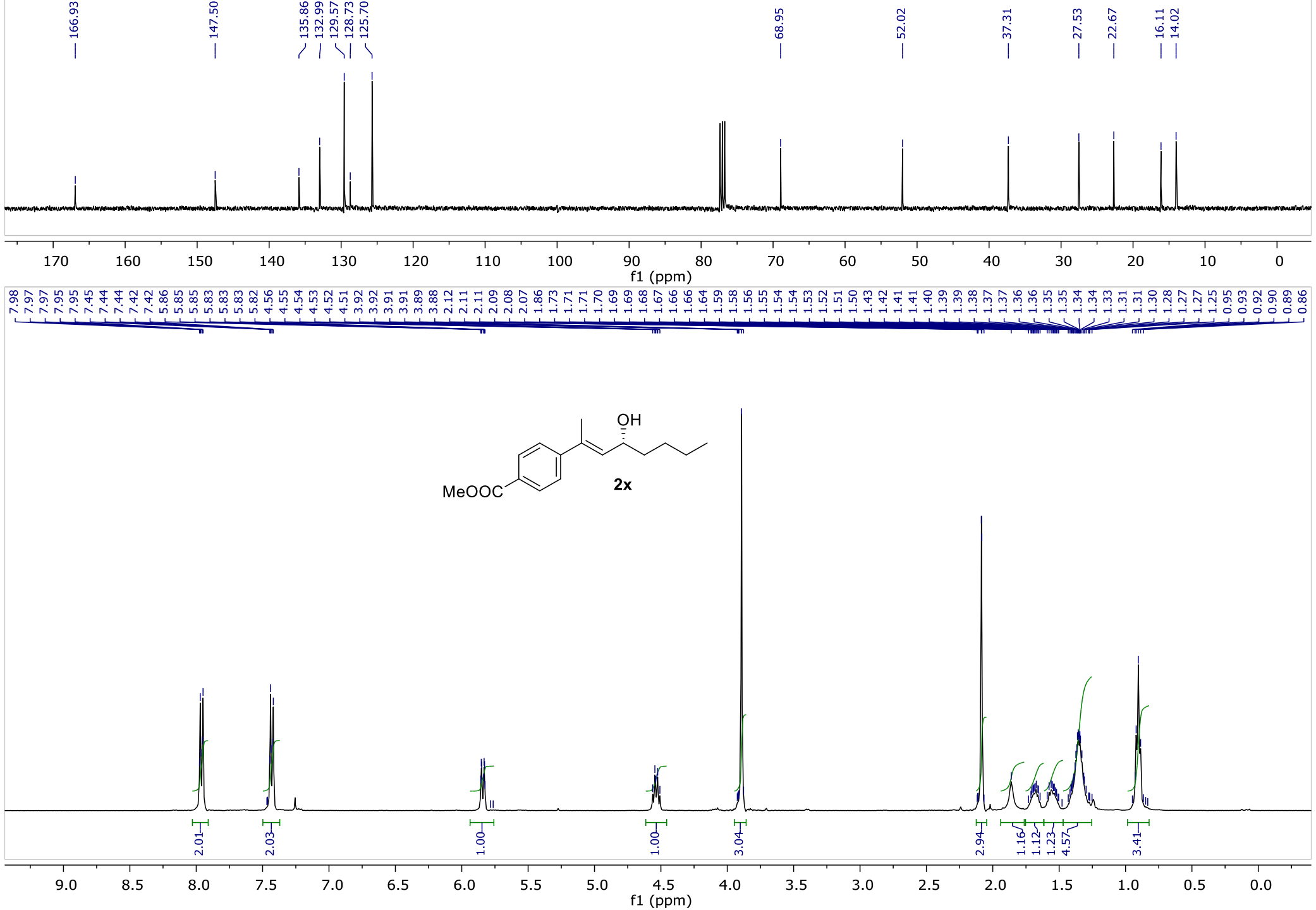
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	9.723	9.28	10.87	321.075	766.611	49.7003	0.355
2	11.474	11.01	13.57	324.947	617.235	50.2997	0.444



Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

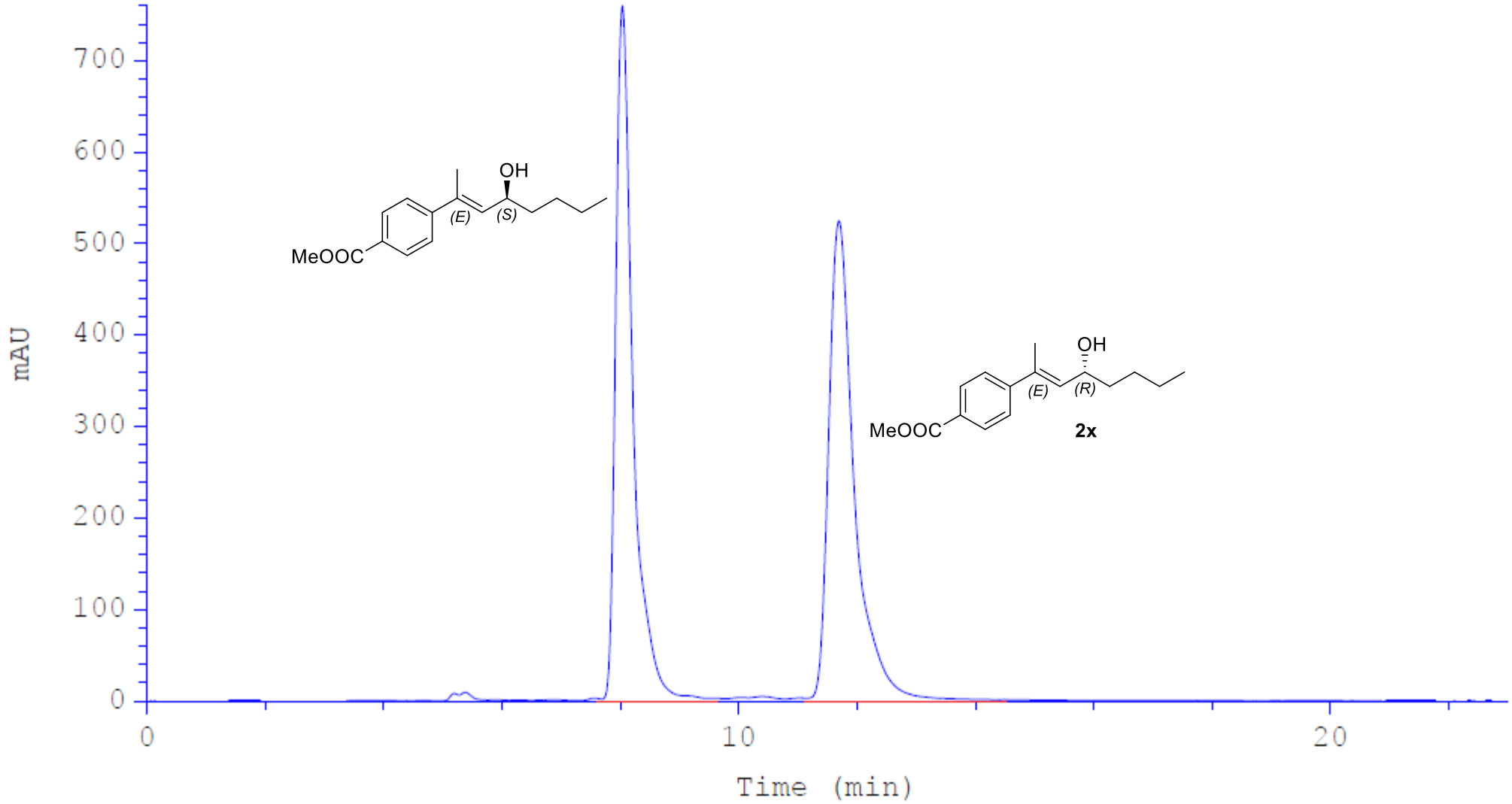
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	9.955	9.53	11.27	949.379	1998.86	98.6300	0.406
2	11.872	11.57	12.50	13.1876	33.1776	1.3700	0.378




166.93
147.50
135.86
132.99
129.57
128.73
125.70
68.95
52.02
37.31
27.53
22.67
16.11
14.02
f1 (ppm)
OH
MeOOC
2x
2.01
2.03
1.00
1.00
3.04
2.94
1.16
1.12
1.23
4.57
3.41
f1 (ppm)

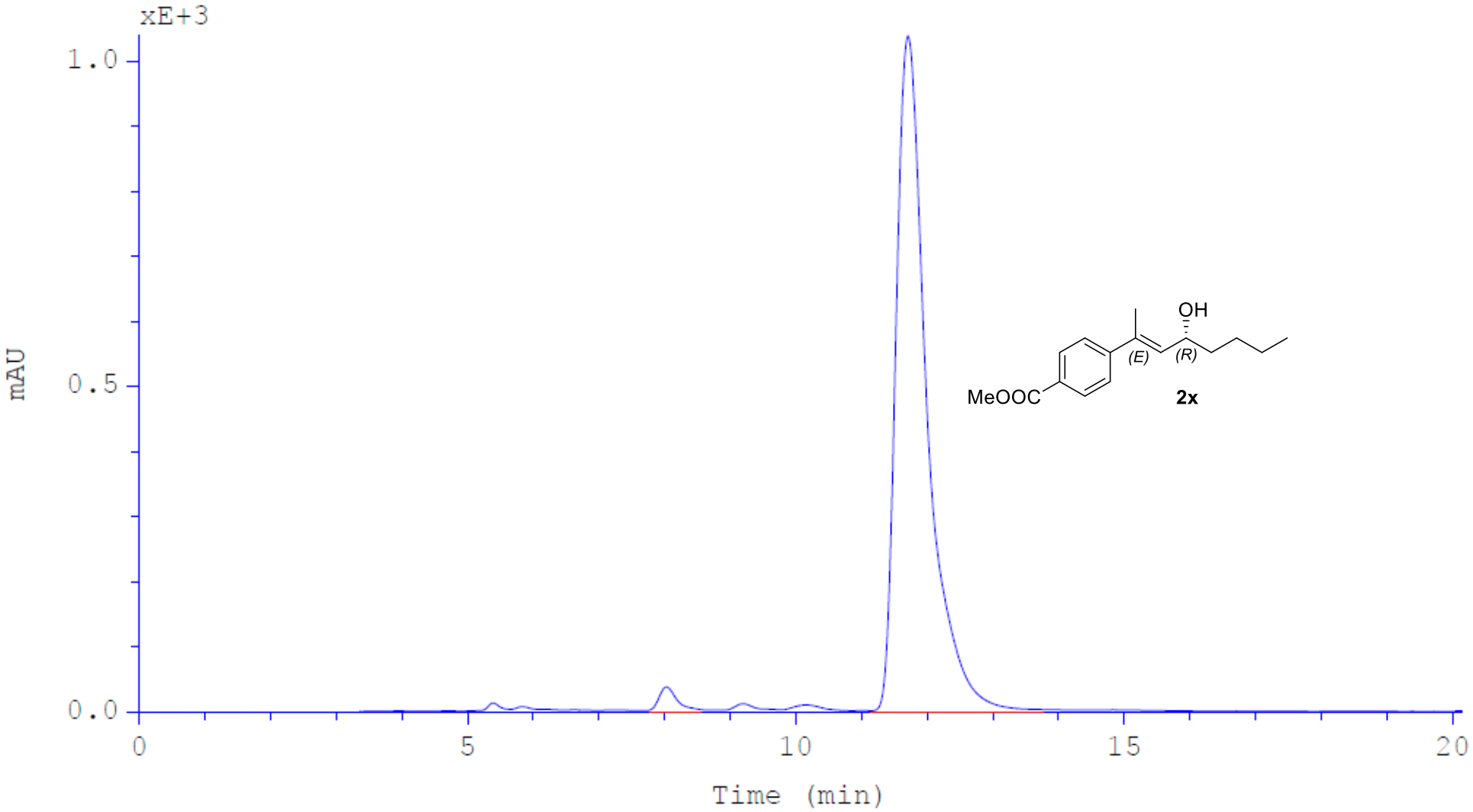
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

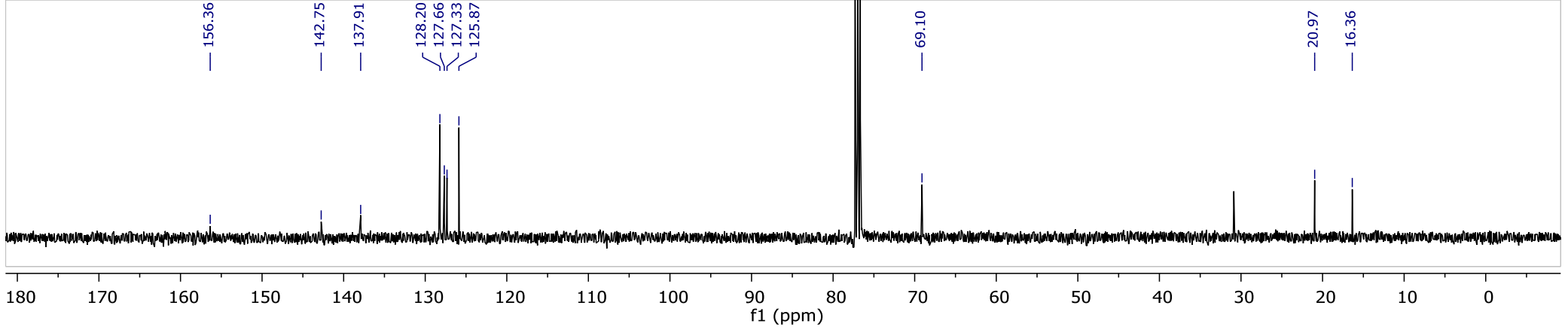
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	8.038	7.69	9.47	254.114	757.81	49.6862	0.281
2	11.690	11.20	13.41	257.324	521.955	50.3138	0.417

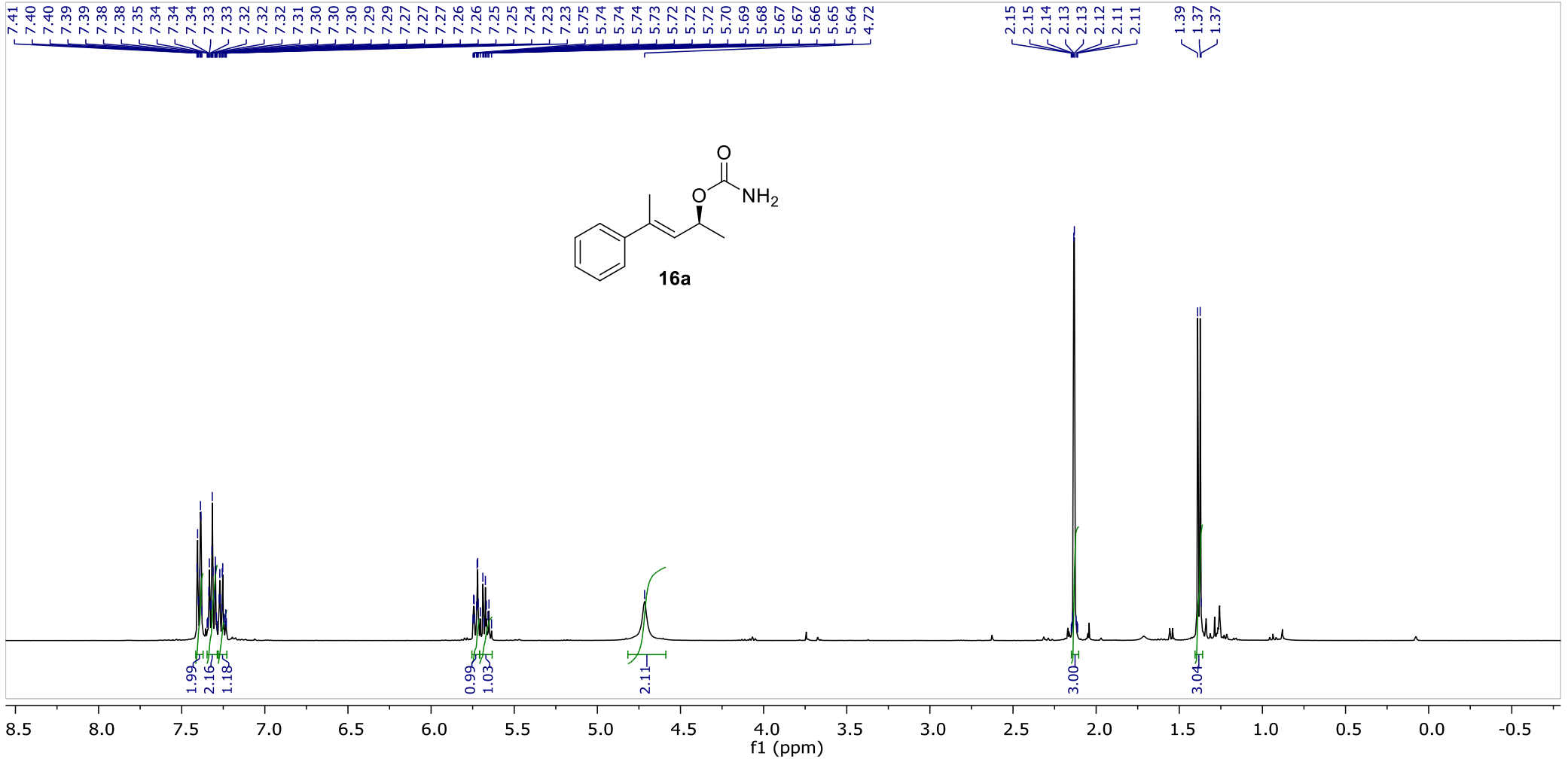


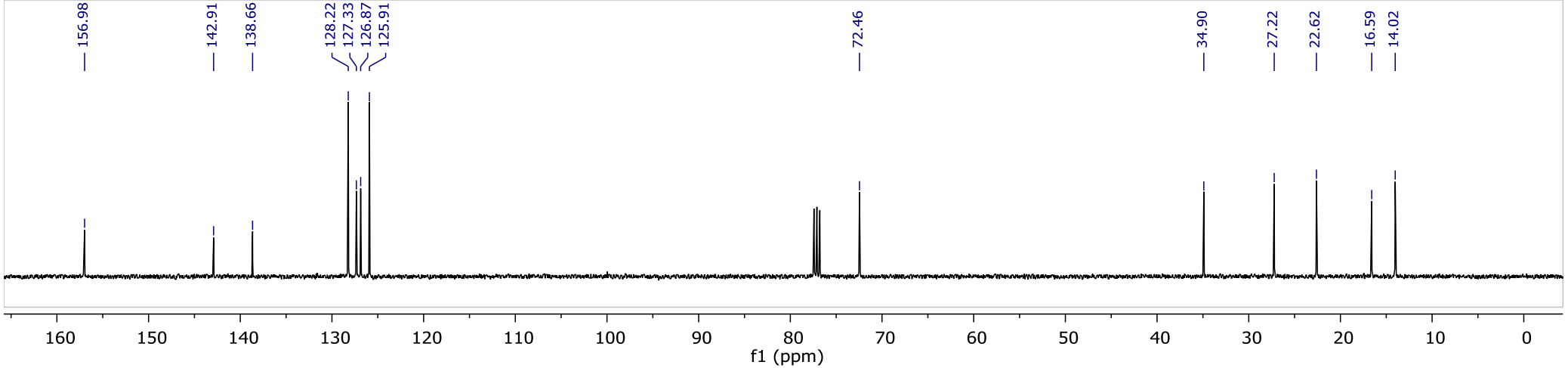
Column: Chiralcel OD-H
Eluent: 10% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 254 nm

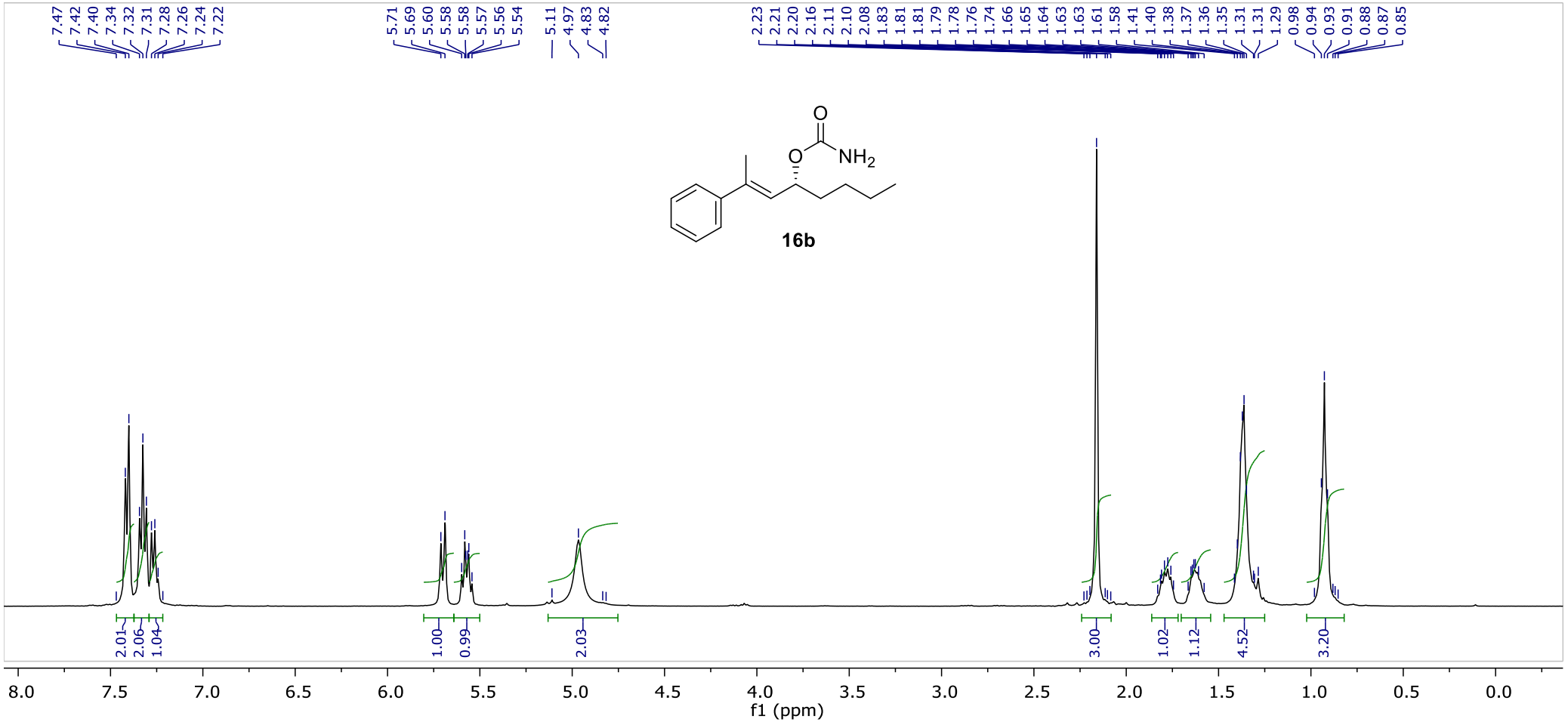
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	8.022	7.79	8.55	10.2689	35.0787	1.7944	0.262
2	11.708	11.20	13.68	562.019	1037.73	98.2056	0.462

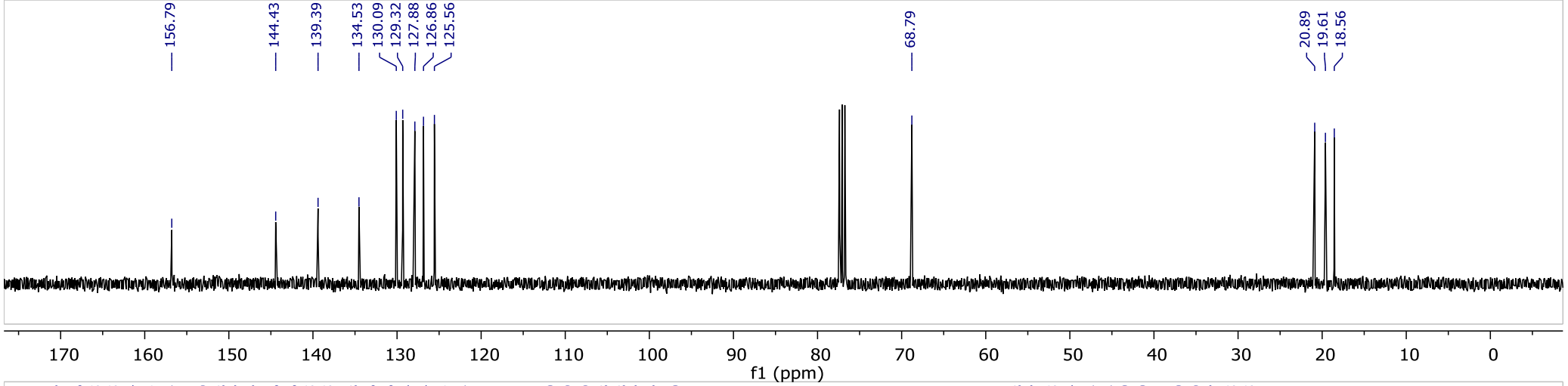


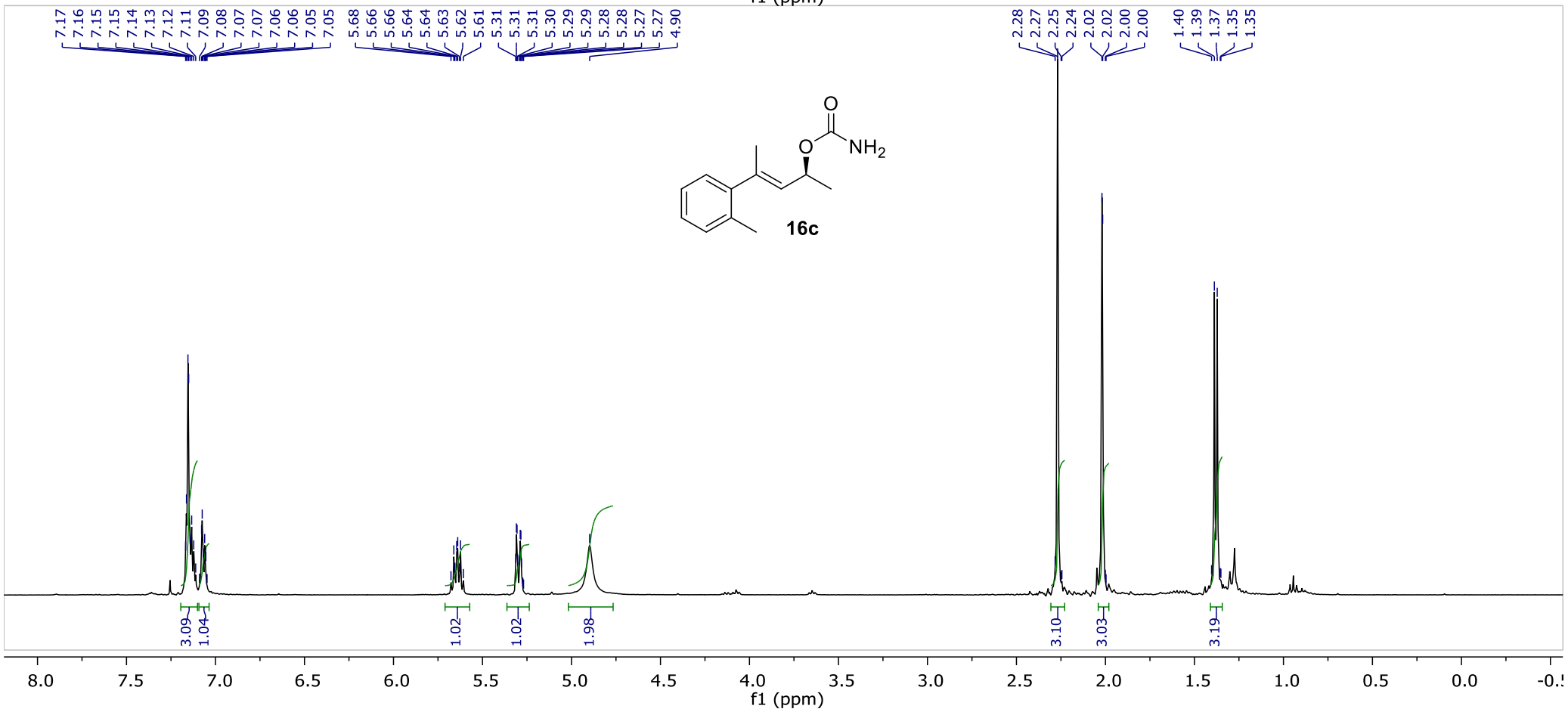

156.36
142.75
137.91
128.20
127.66
127.33
125.87
69.10
20.97
16.36
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

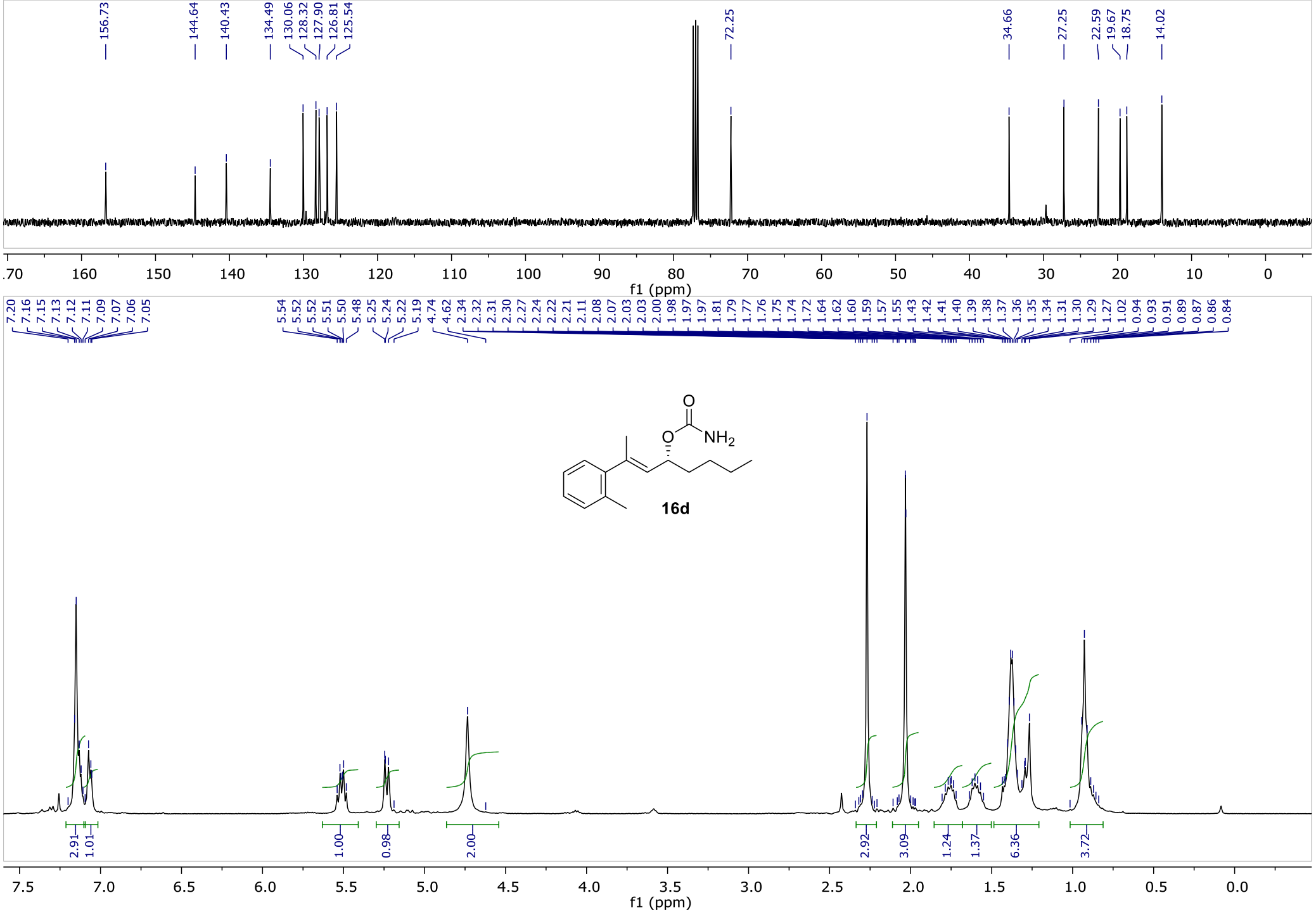

7.41 7.40 7.40 7.39 7.39 7.38 7.38 7.35 7.34 7.34 7.34 7.33 7.33 7.32 7.32 7.31 7.30 7.30 7.30 7.29 7.29 7.27 7.27 7.27 7.26 7.26 7.25 7.25 7.24 7.23 7.23 5.75 5.74 5.74 5.74 5.73 5.72 5.72 5.72 5.70 5.69 5.68 5.67 5.67 5.66 5.65 5.64 4.72
2.15 2.15 2.14 2.13 2.13 2.12 2.11 2.11
1.39 1.37 1.37
O
O
NH2
16a
1.99
2.16
1.18
0.99
1.03
2.11
3.00
3.04
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)

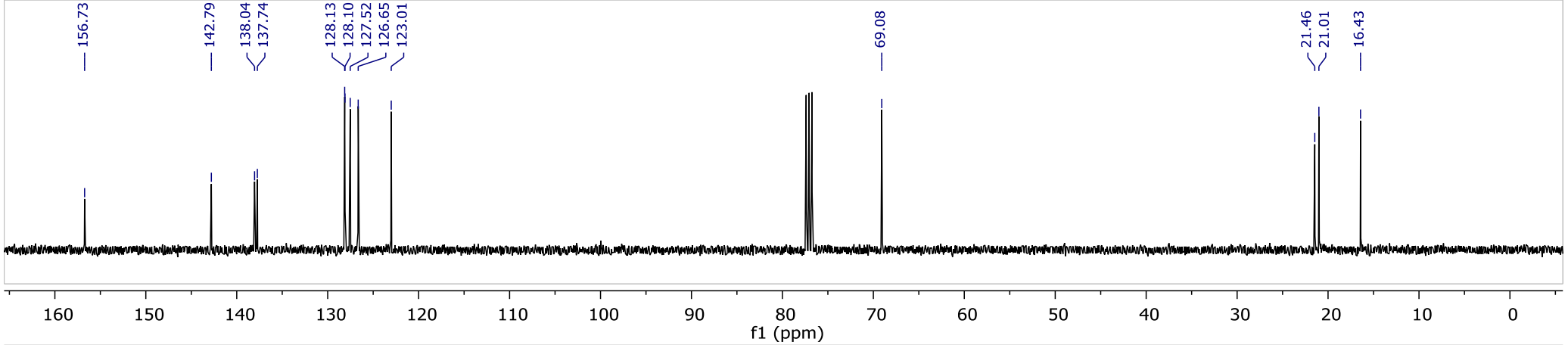

156.98
142.91
138.66
128.22
127.33
126.87
125.91
72.46
34.90
27.22
22.62
16.59
14.02
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

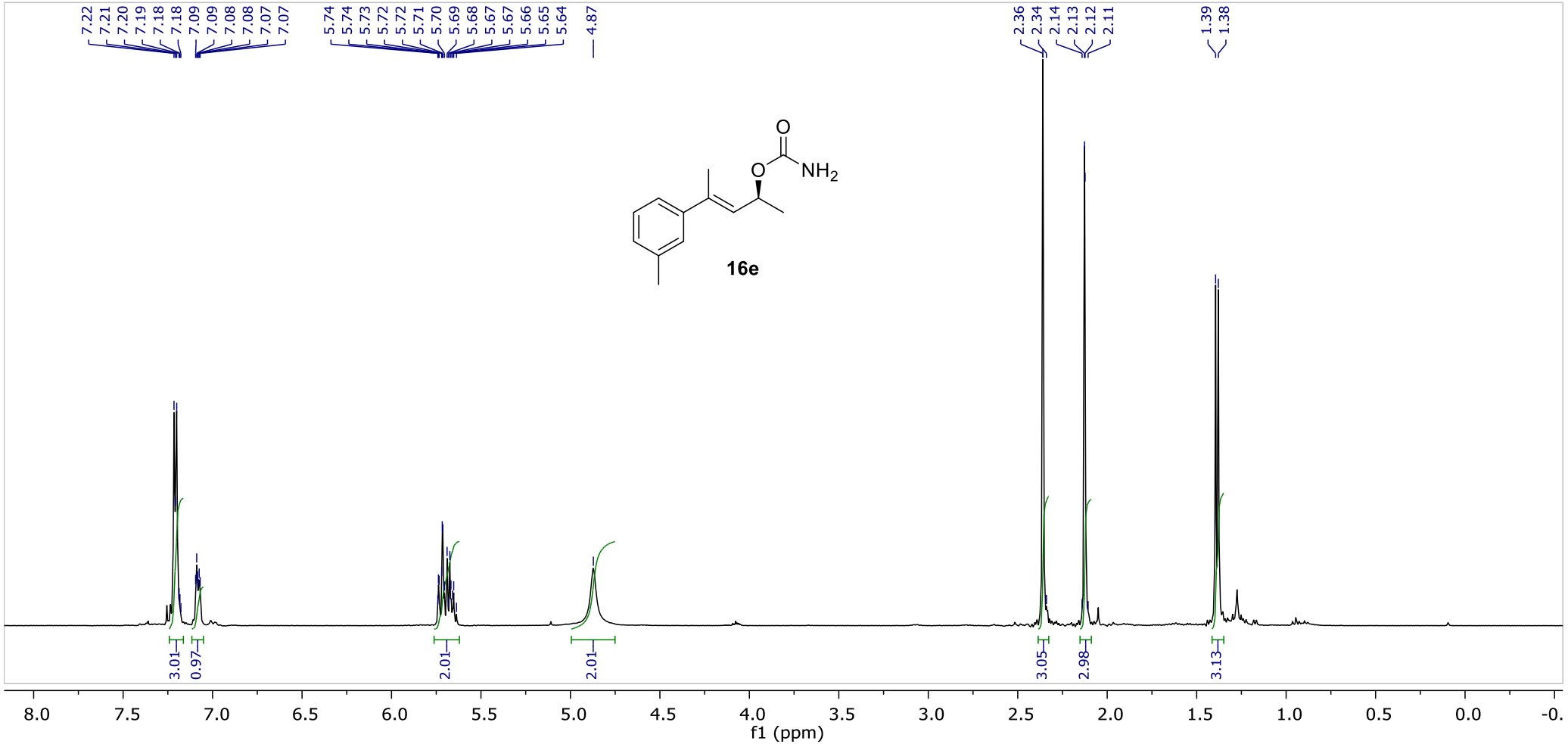

7.47
7.42
7.40
7.34
7.32
7.31
7.28
7.26
7.24
7.22
5.71
5.69
5.60
5.58
5.58
5.57
5.56
5.54
5.11
4.97
4.83
4.82
2.23
2.21
2.20
2.16
2.11
2.10
2.08
1.83
1.81
1.81
1.79
1.78
1.76
1.74
1.66
1.65
1.64
1.63
1.63
1.61
1.58
1.41
1.40
1.38
1.37
1.36
1.35
1.31
1.31
1.29
0.98
0.94
0.93
0.91
0.88
0.87
0.85
O
O
NH2
16b
2.01
2.06
1.04
1.00
0.99
2.03
3.00
1.02
1.12
4.52
3.20
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
f1 (ppm)

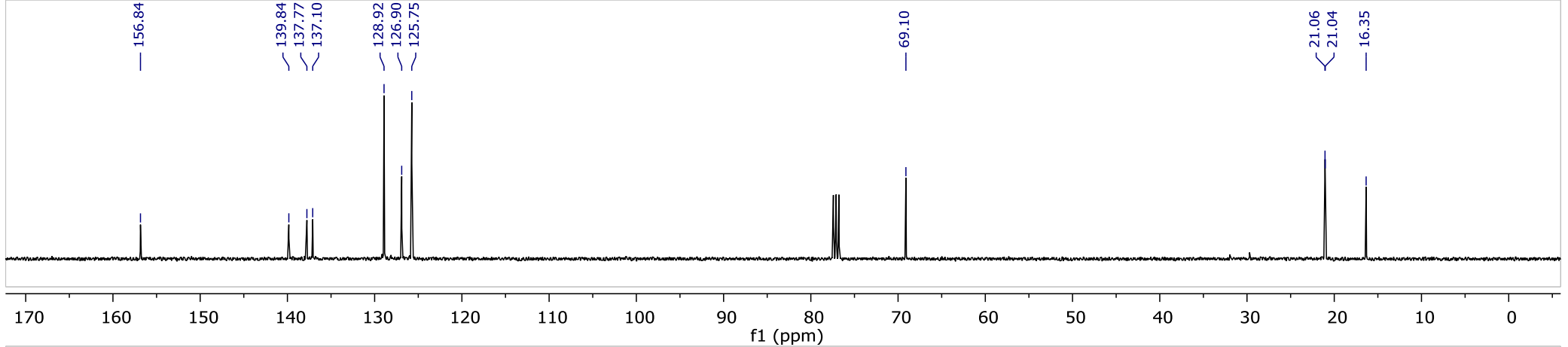

156.79
144.43
139.39
134.53
130.09
129.32
127.88
126.86
125.56
68.79
20.89
19.61
18.56
f1 (ppm)

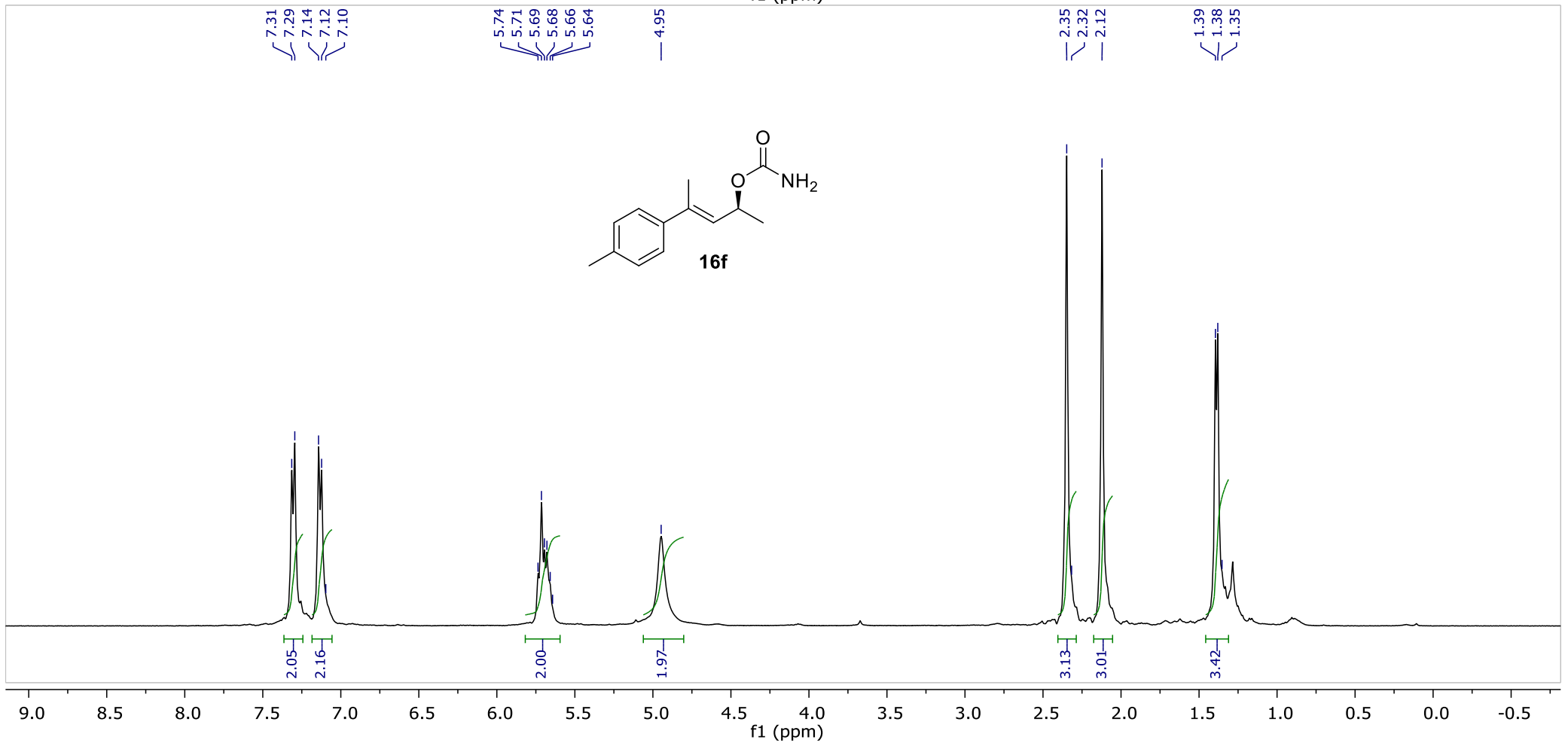

7.17
7.16
7.15
7.15
7.14
7.13
7.12
7.11
7.09
7.08
7.07
7.07
7.06
7.06
7.05
7.05
5.68
5.66
5.66
5.64
5.64
5.63
5.62
5.61
5.31
5.31
5.30
5.29
5.29
5.28
5.28
5.27
5.27
4.90
2.28
2.27
2.25
2.24
2.02
2.02
2.00
2.00
1.40
1.39
1.37
1.35
1.35
O
O
NH2
16c
3.09
1.04
1.02
1.02
1.98
3.10
3.03
3.19
f1 (ppm)

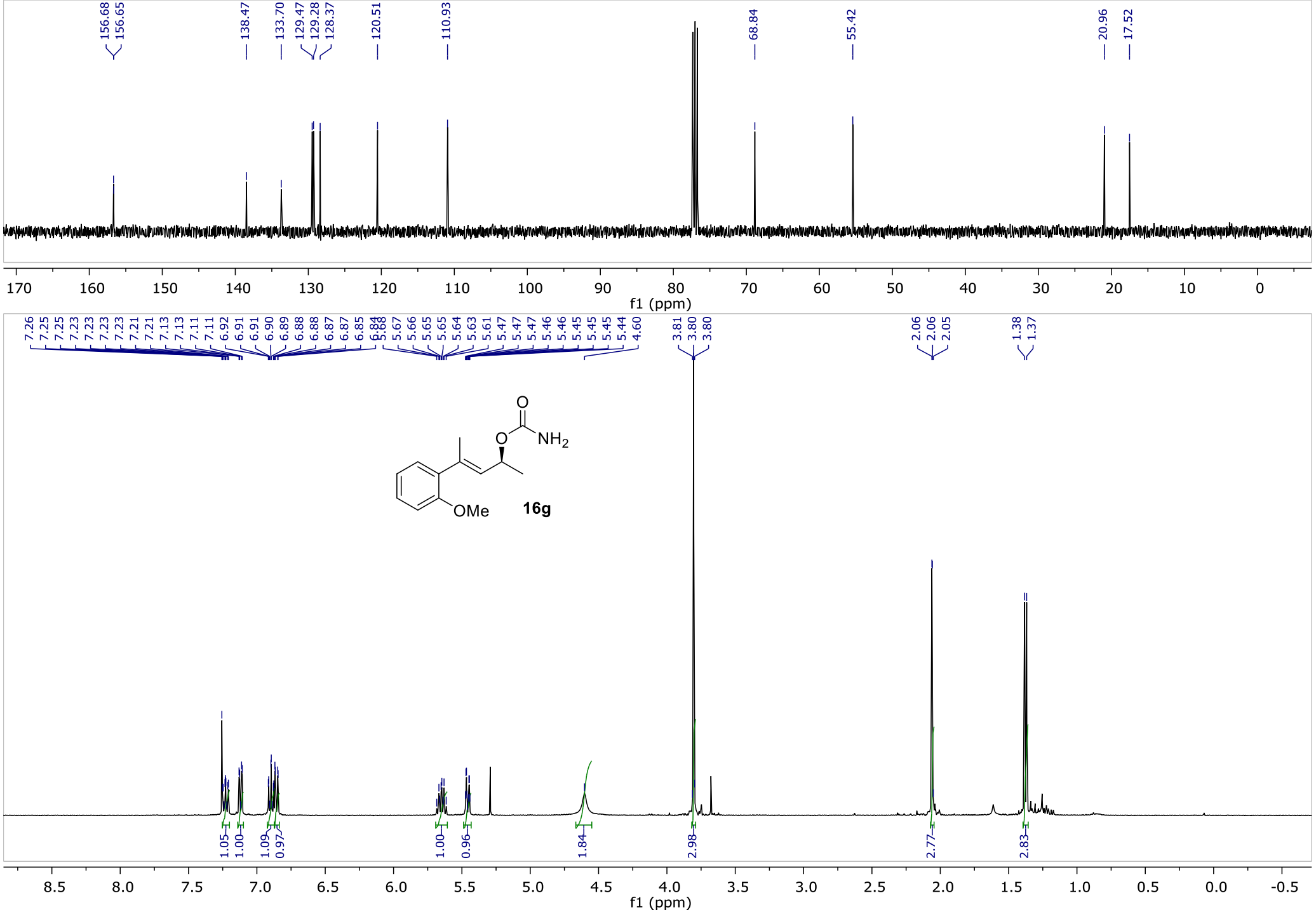

156.73
144.64
140.43
134.49
130.06
128.32
127.90
126.81
125.54
72.25
34.66
27.25
22.59
19.67
18.75
14.02
f1 (ppm)
7.20
7.16
7.15
7.13
7.12
7.11
7.09
7.07
7.06
7.05
5.54
5.52
5.52
5.51
5.50
5.48
5.25
5.24
5.22
5.19
4.74
4.62
2.34
2.32
2.31
2.30
2.27
2.24
2.22
2.21
2.11
2.08
2.07
2.03
2.03
2.00
1.98
1.97
1.97
1.81
1.79
1.77
1.76
1.75
1.74
1.72
1.64
1.62
1.60
1.59
1.57
1.55
1.43
1.42
1.41
1.40
1.39
1.38
1.37
1.36
1.35
1.34
1.31
1.30
1.29
1.27
1.02
0.94
0.93
0.91
0.89
0.87
0.86
0.84
O
O
NH2
16d
2.91
1.01
1.00
0.98
2.00
2.92
3.09
1.24
1.37
6.36
3.72
f1 (ppm)


156.73
142.79
138.04
137.74
128.13
128.10
127.52
126.65
123.01
69.08
21.46
21.01
16.43
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.22
7.21
7.20
7.19
7.18
7.18
7.09
7.09
7.08
7.08
7.07
7.07
5.74
5.74
5.73
5.72
5.72
5.71
5.70
5.69
5.68
5.67
5.67
5.66
5.65
5.64
4.87
2.36
2.34
2.14
2.13
2.12
2.11
1.39
1.38
O
O
NH2
16e
3.01
0.97
2.01
2.01
3.05
2.98
3.13
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.
f1 (ppm)


156.84
139.84
137.77
137.10
128.92
126.90
125.75
69.10
21.06
21.04
16.35
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.31
7.29
7.14
7.12
7.10
5.74
5.71
5.69
5.68
5.66
5.64
4.95
2.35
2.32
2.12
1.39
1.38
1.35
O
O
NH2
16f
2.05
2.16
2.00
1.97
3.13
3.01
3.42
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)


156.68
156.65
138.47
133.70
129.47
129.28
128.37
120.51
110.93
68.84
55.42
20.96
17.52
f1 (ppm)
7.26
7.25
7.25
7.23
7.23
7.23
7.23
7.21
7.21
7.13
7.13
7.11
7.11
6.92
6.91
6.91
6.90
6.89
6.88
6.88
6.87
6.87
6.85
6.84
5.88
5.67
5.66
5.65
5.65
5.64
5.63
5.61
5.47
5.47
5.47
5.46
5.46
5.45
5.45
5.45
5.44
4.60
3.81
3.80
3.80
2.06
2.06
2.05
1.38
1.37
O
O
NH2
OMe
16g
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1.09
0.97
1.00
0.96
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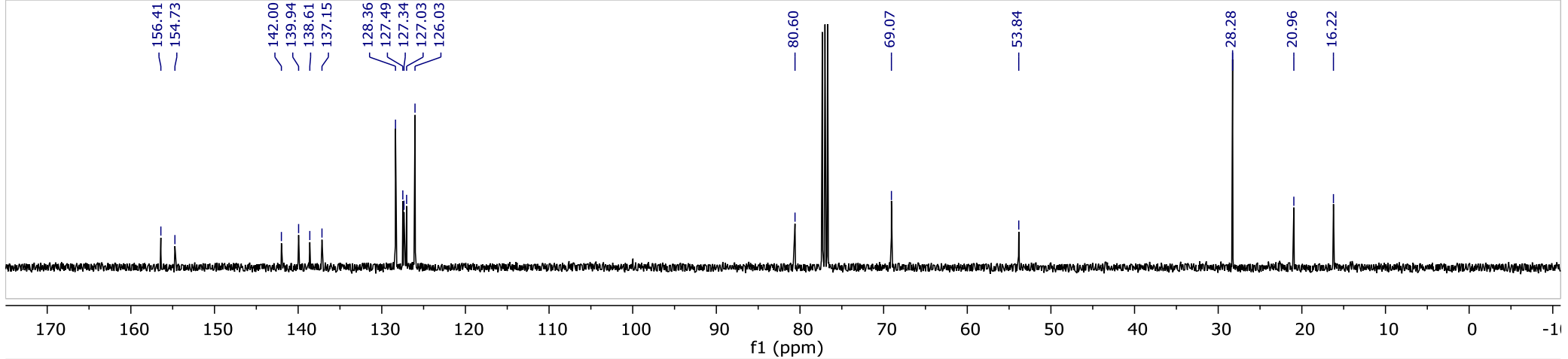
2.98
2.77
2.83
f1 (ppm)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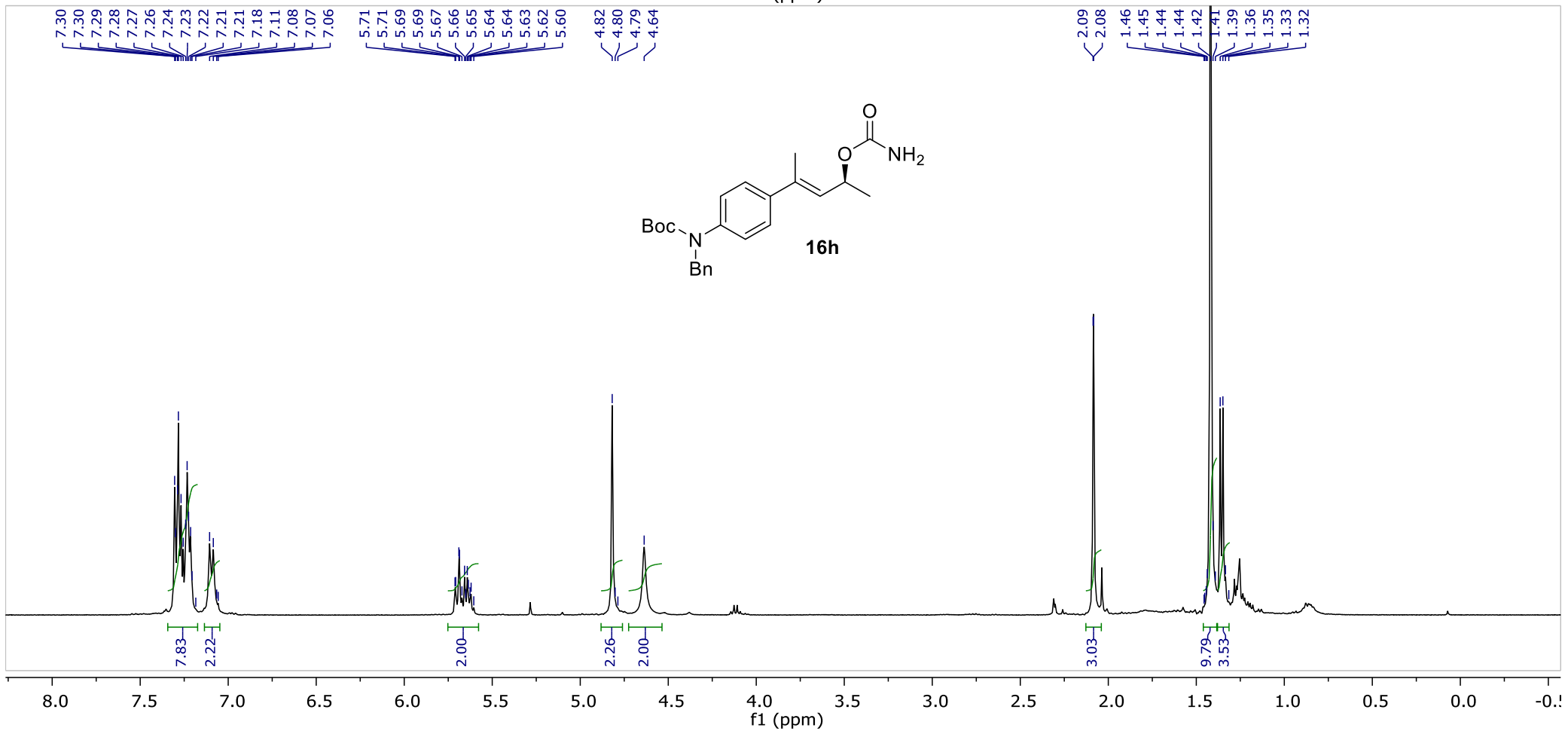
126.03
80.60
69.07
53.84
28.28
20.96
16.22
f1 (ppm)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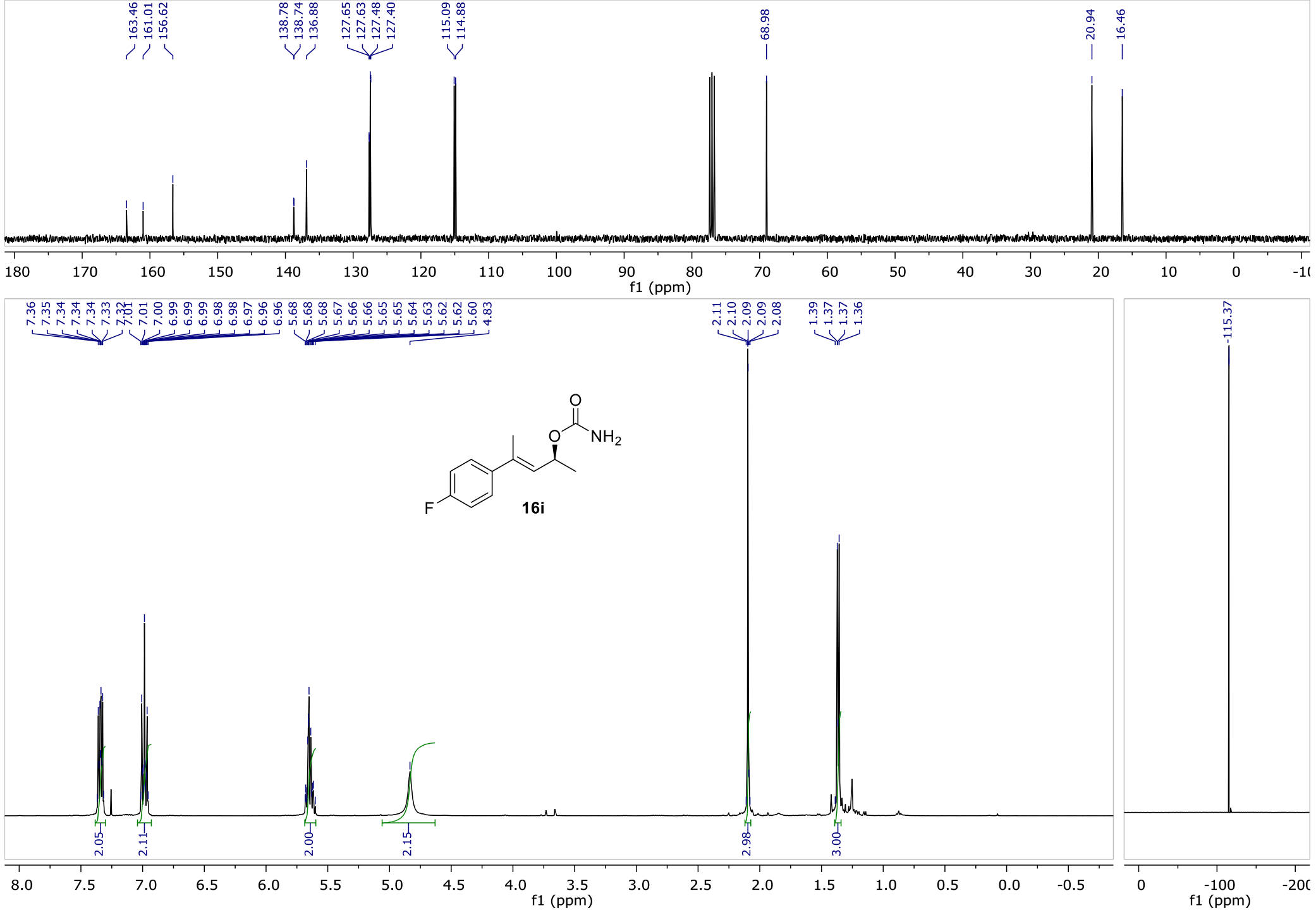
7.11
7.08
7.07
7.06
5.71
5.71
5.69
5.69
5.67
5.66
5.65
5.64
5.64
5.63
5.62
5.60
4.82
4.80
4.79
4.64
2.09
2.08
1.46
1.45
1.44
1.44
1.42
1.41
1.39
1.36
1.35
1.33
1.32
O
O
NH2
Boc
N
Bn
16h
7.83
2.22
2.00
2.26
2.00
3.03
9.79
3.53
f1 (ppm)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127.65
127.63
127.48
127.40
115.09
114.88
68.98
20.94
16.46
f1 (ppm)
7.36
7.35
7.34
7.34
7.34
7.33
7.32
7.01
7.01
7.00
6.99
6.99
6.99
6.98
6.98
6.97
6.96
6.96
5.68
5.68
5.68
5.67
5.66
5.66
5.65
5.65
5.64
5.63
5.62
5.62
5.60
4.83
2.11
2.10
2.09
2.09
2.08
1.39
1.37
1.37
1.36
O
O
NH2
F
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2.11
2.00
2.15
2.98
3.00
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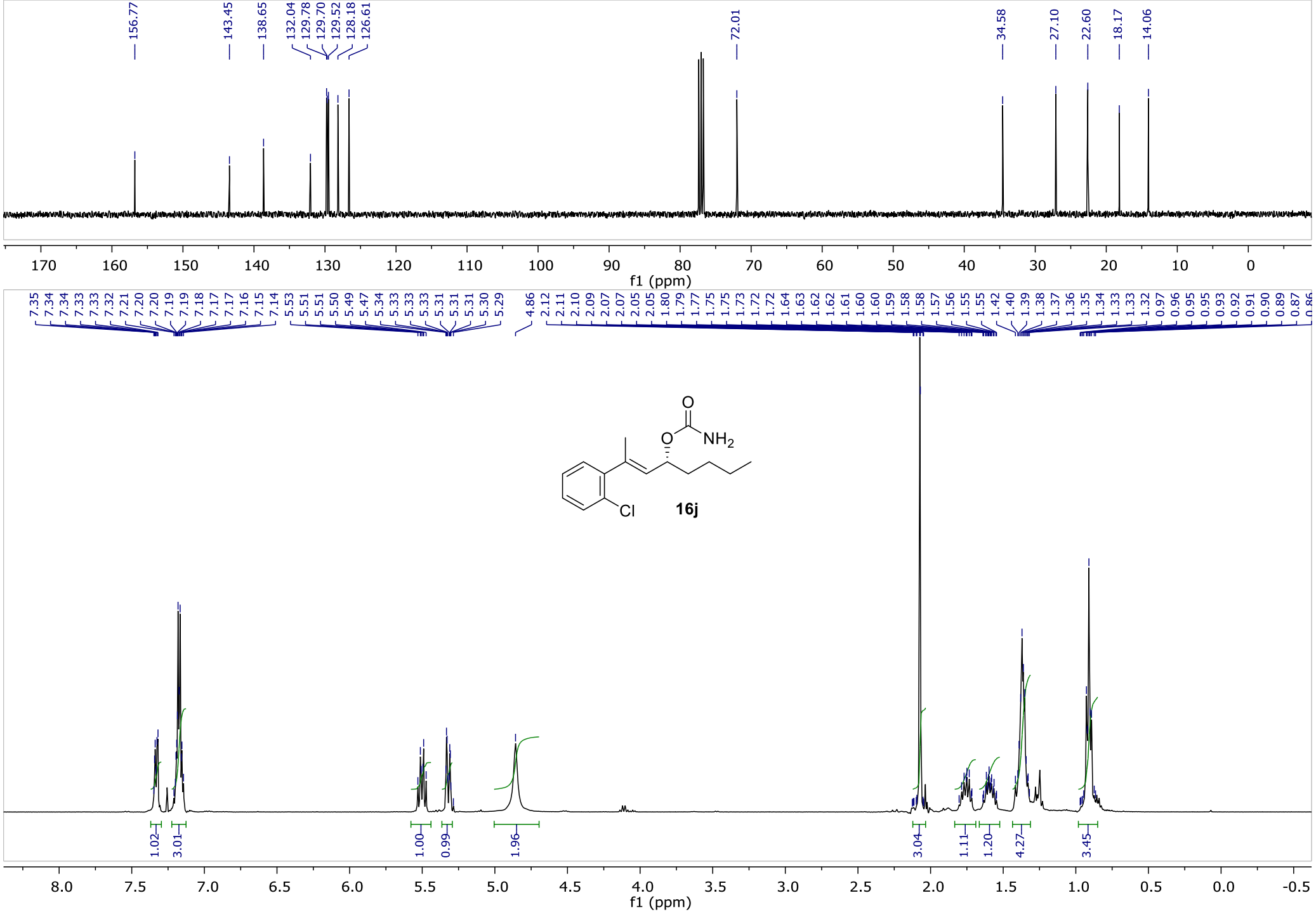
f1 (ppm)
f1 (ppm)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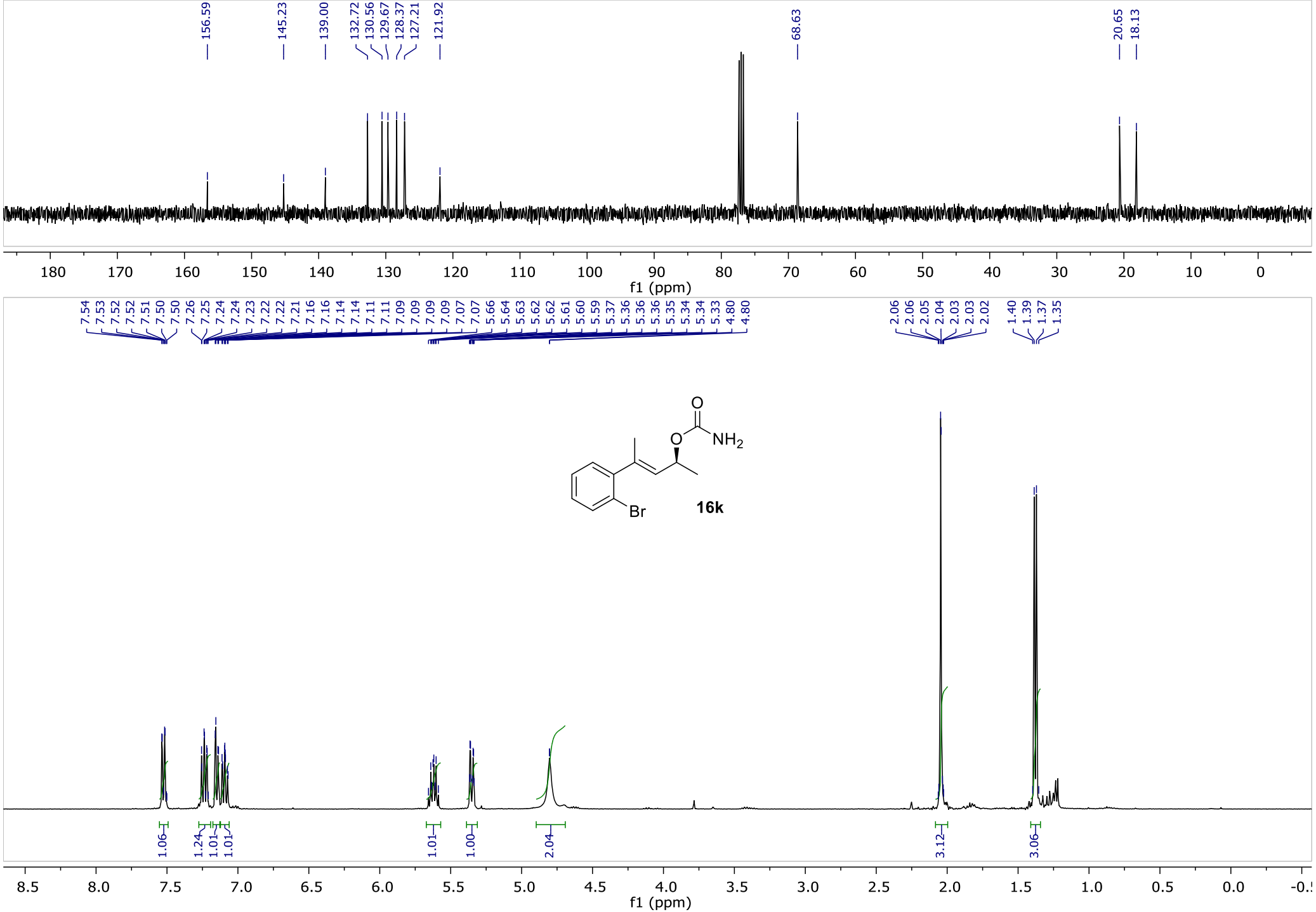
129.52
128.18
126.61
72.01
34.58
27.10
22.60
18.17
14.06
f1 (ppm)
O
O
NH2
Cl
16j
1.02
3.01
1.00
0.99
1.96
3.04
1.11
1.20
4.27
3.45
f1 (ppm)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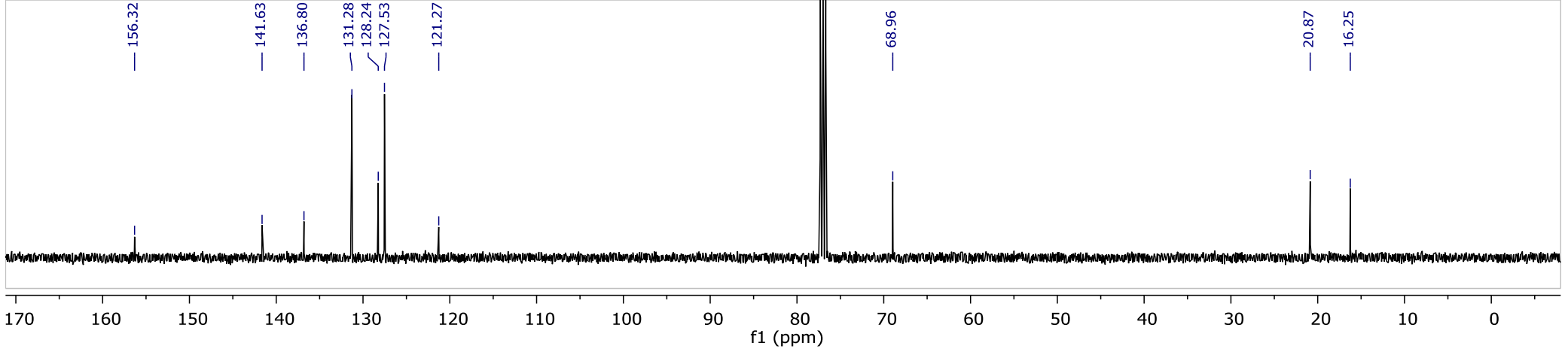
145.23
139.00
132.72
130.56
129.67
128.37
127.21
121.92
68.63
20.65
18.13
f1 (ppm)
7.54 7.53 7.52 7.52 7.51 7.50 7.50 7.26 7.25 7.24 7.24 7.23 7.22 7.22 7.21 7.16 7.16 7.14 7.14 7.11 7.11 7.09 7.09 7.09 7.09 7.07 7.07 5.66 5.64 5.63 5.62 5.62 5.61 5.60 5.59 5.37 5.36 5.36 5.36 5.35 5.34 5.34 5.33 4.80 4.80
2.06 2.06 2.05 2.04 2.03 2.03 2.02
1.40 1.39 1.37 1.35
O
O
NH2
Br
16k
1.06
1.24
1.01
1.01
1.01
1.00
2.04
3.12
3.06
f1 (ppm)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136.80
131.28
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127.53
121.27
68.96
20.87
16.25
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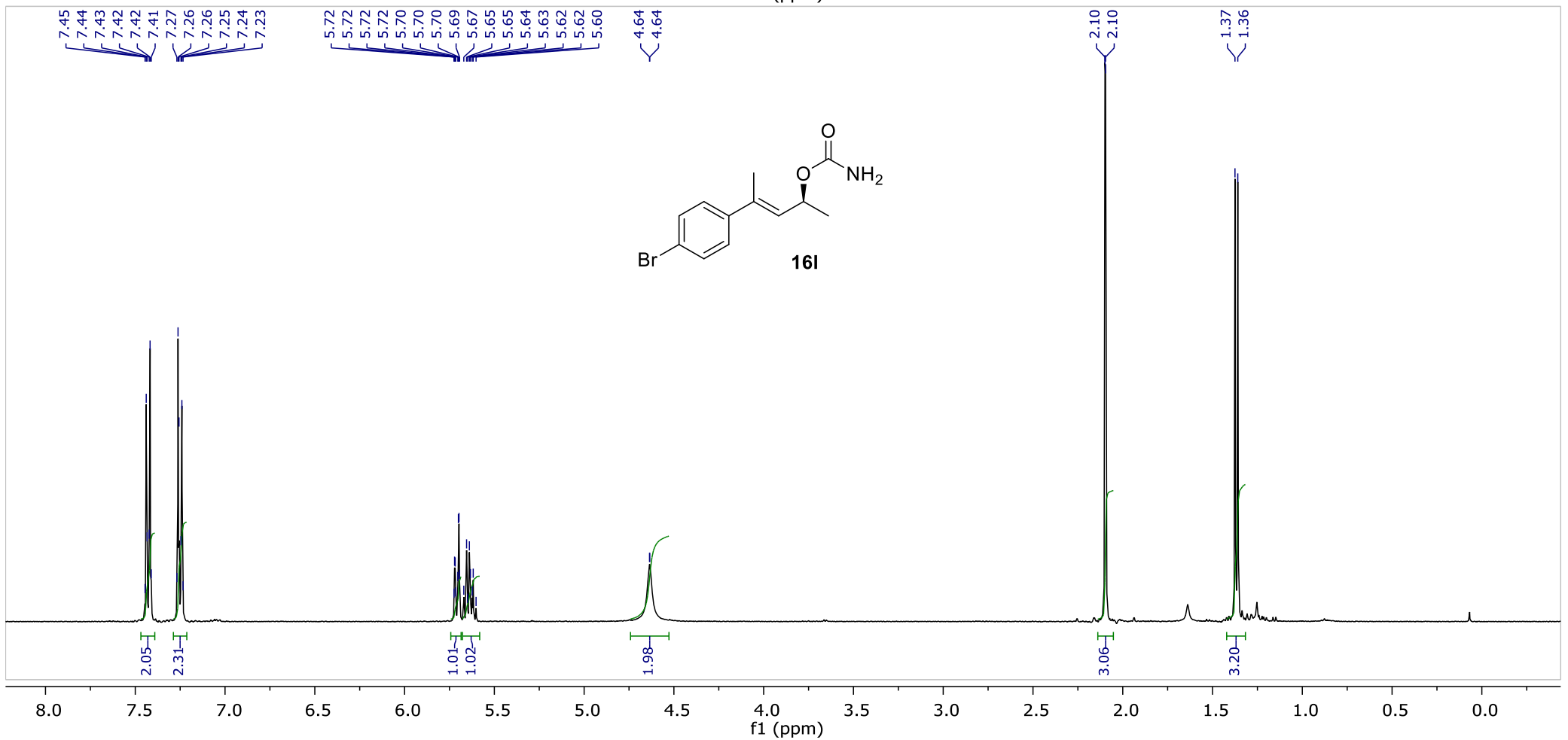
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.44
7.43
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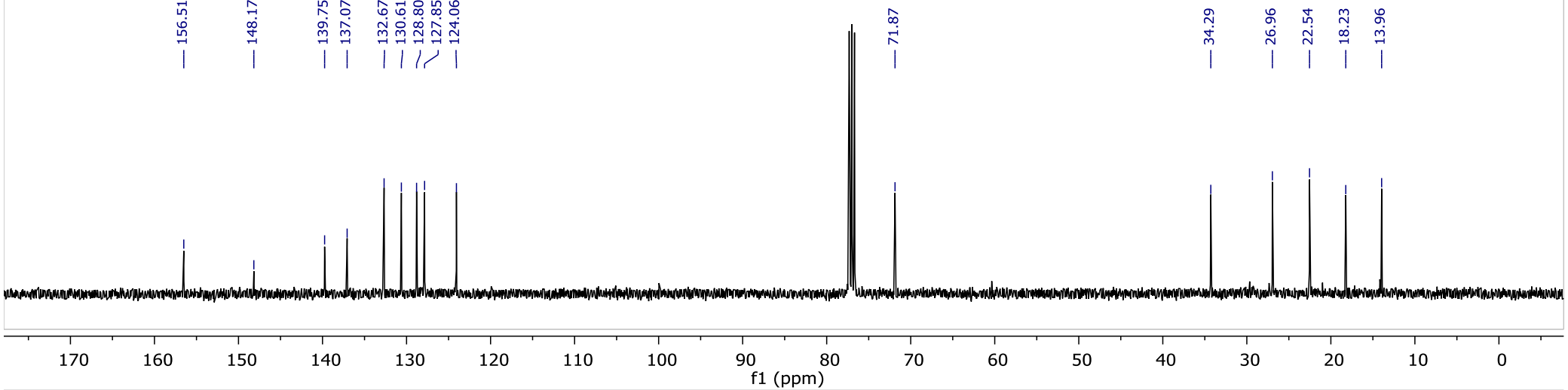
7.42
7.41
7.27
7.26
7.26
7.25
7.24
7.23
5.72
5.72
5.72
5.72
5.70
5.70
5.70
5.69
5.67
5.65
5.65
5.64
5.63
5.62
5.62
5.60
4.64
4.64
2.10
2.10
1.37
1.36
O
O
NH2
Br
16l
2.05
2.31
1.01
1.02
1.98
3.06
3.20
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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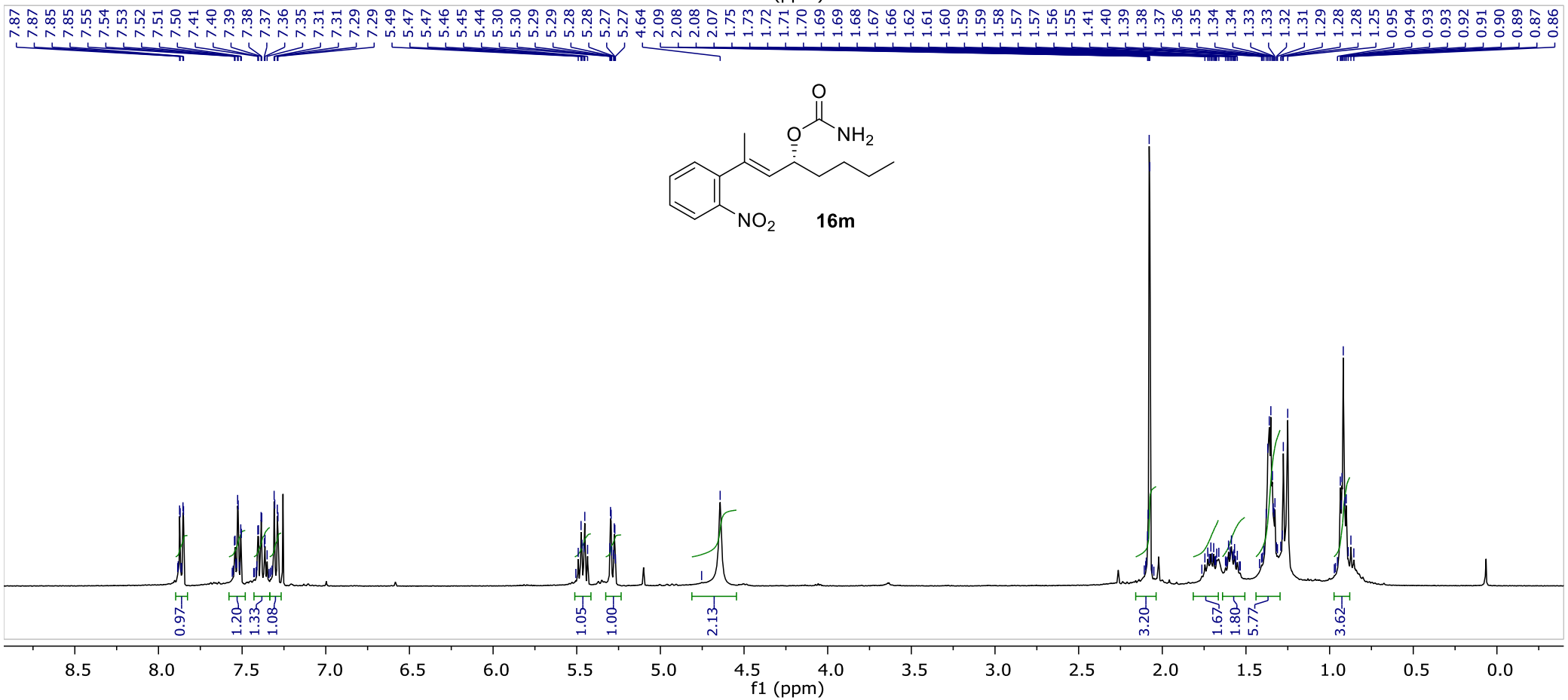
148.17
139.75
137.07
132.67
130.61
128.80
127.85
124.06
71.87
34.29
26.96
22.54
18.23
13.96
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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NO2
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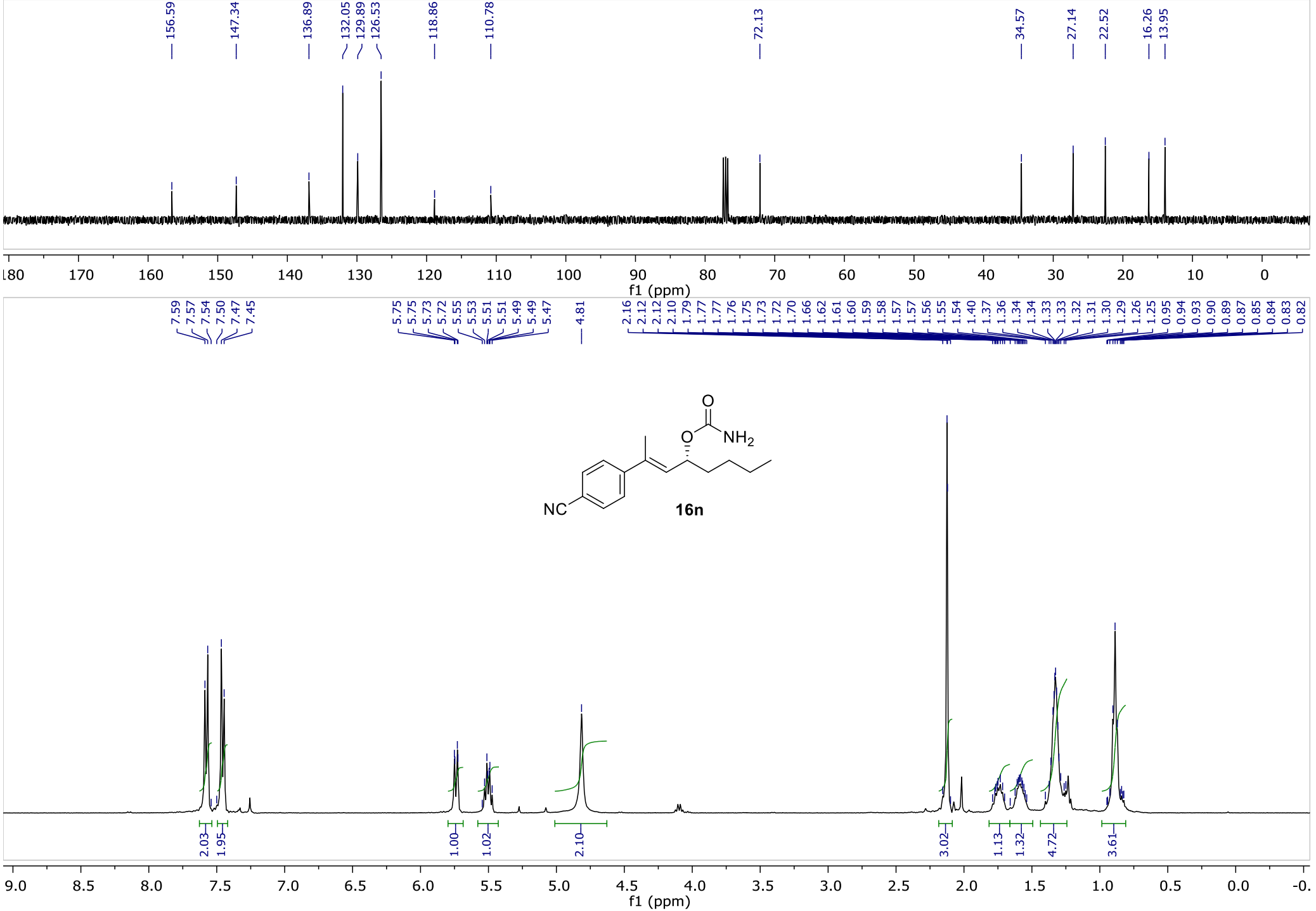
1.05
1.00
2.13
3.20
1.67
1.80
5.77
3.62
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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129.89
126.53
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110.78
72.13
34.57
27.14
22.52
16.26
13.95
f1 (ppm)
7.59
7.57
7.54
7.50
7.47
7.45
5.75
5.75
5.73
5.72
5.55
5.53
5.51
5.51
5.49
5.49
5.47
4.81
2.16
2.12
2.12
2.10
1.79
1.77
1.77
1.76
1.75
1.73
1.72
1.70
1.66
1.62
1.61
1.60
1.59
1.58
1.57
1.57
1.56
1.55
1.54
1.40
1.37
1.36
1.34
1.34
1.33
1.33
1.32
1.31
1.30
1.29
1.26
1.25
0.95
0.94
0.93
0.90
0.89
0.87
0.85
0.84
0.83
0.82
O
O
NH2
NC
16n
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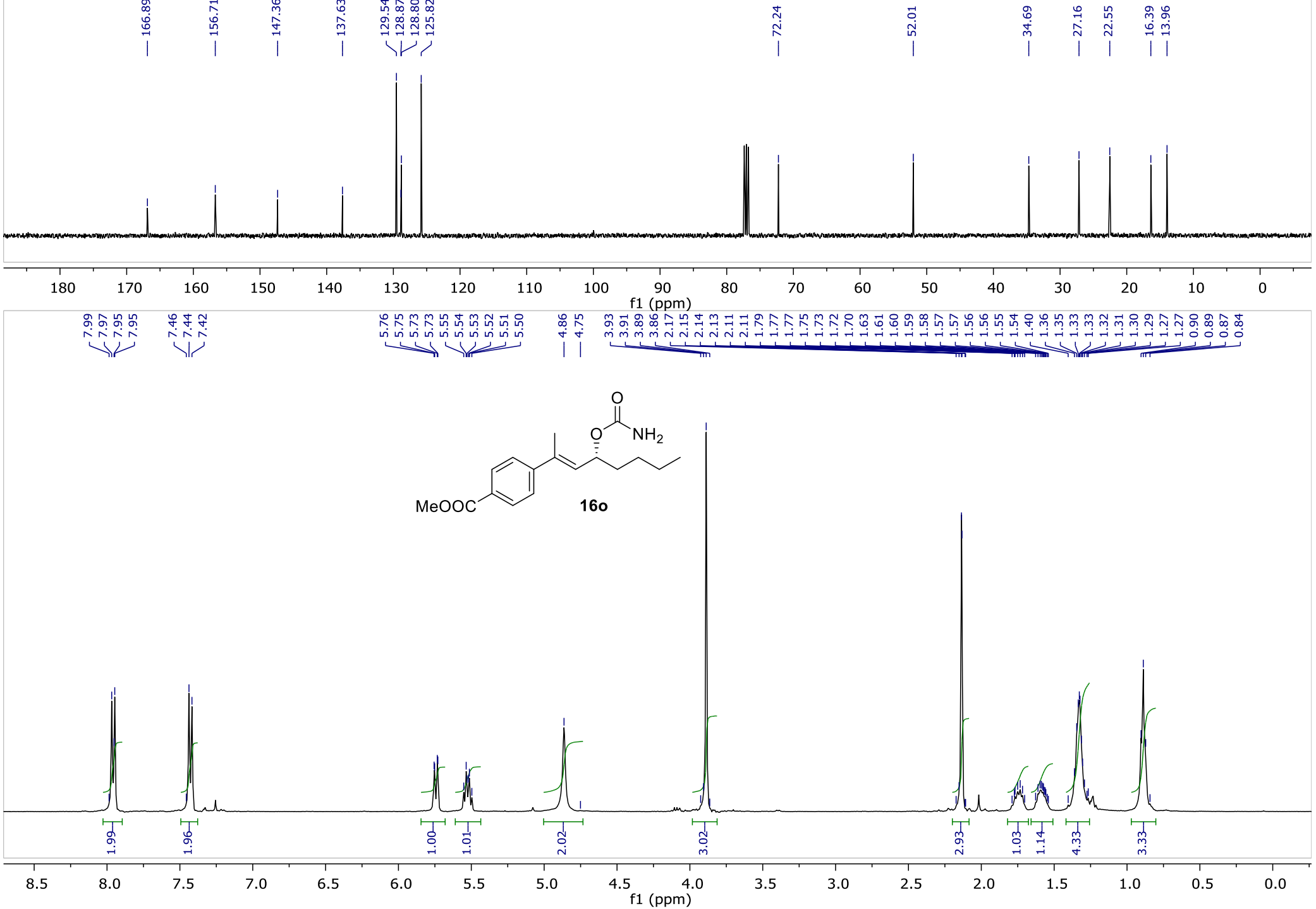
1.00
1.02
2.10
3.02
1.13
1.32
4.72
3.61
f1 (ppm)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128.80
125.82
72.24
52.01
34.69
27.16
22.55
16.39
13.96
f1 (ppm)
7.99
7.97
7.95
7.95
7.46
7.44
7.42
5.76
5.75
5.73
5.73
5.55
5.54
5.53
5.52
5.51
5.50
4.86
4.75
3.93
3.91
3.89
3.86
2.17
2.15
2.14
2.13
2.11
2.11
1.79
1.77
1.77
1.75
1.73
1.72
1.70
1.63
1.61
1.60
1.59
1.58
1.57
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1.56
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1.54
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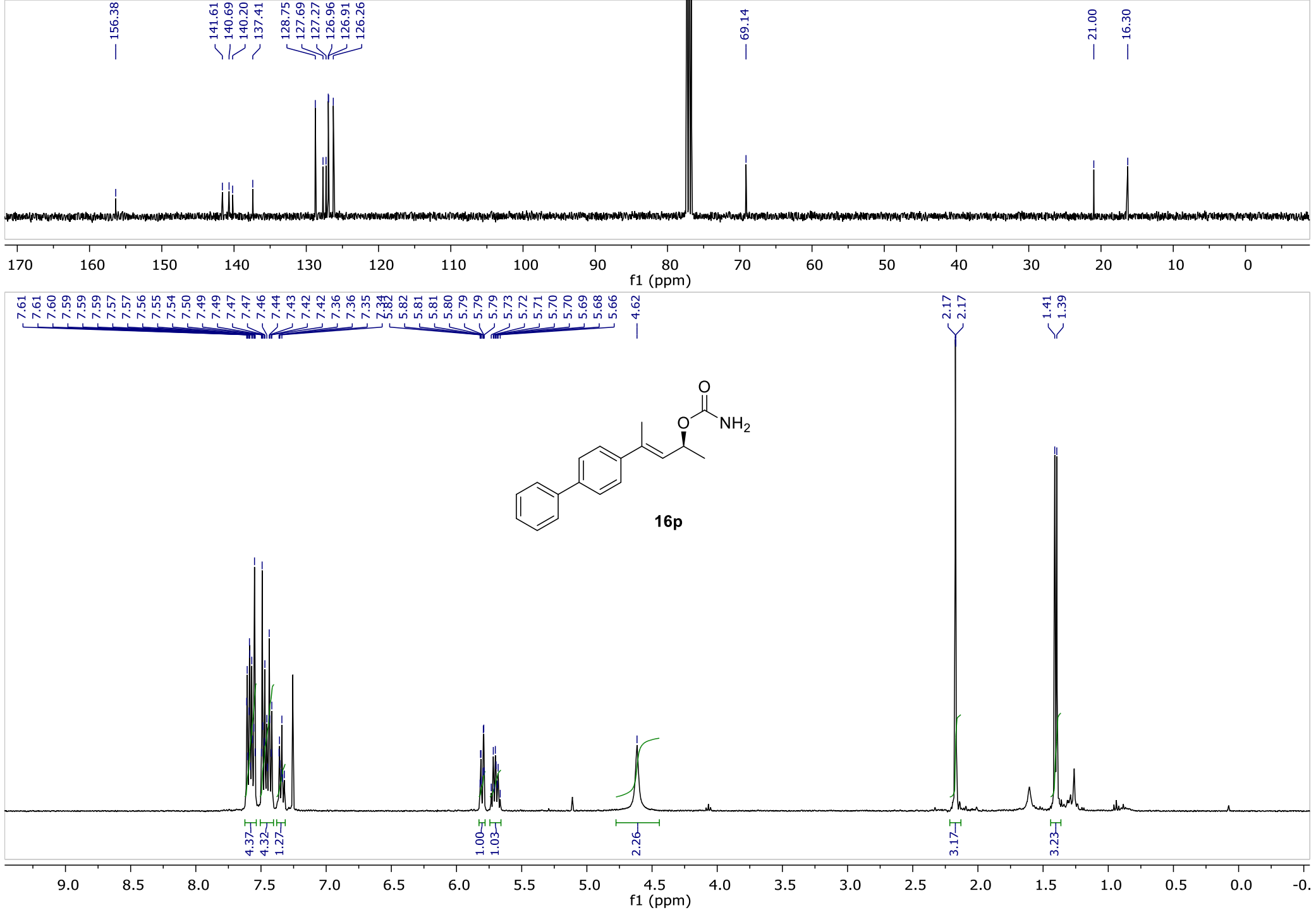
1.36
1.35
1.33
1.33
1.32
1.31
1.30
1.29
1.27
1.27
0.90
0.89
0.87
0.84
O
O
NH2
MeOOC
16o
1.99
1.96
1.00
1.01
2.02
3.02
2.93
1.03
1.14
4.33
3.33
f1 (ppm)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127.69
127.27
126.96
126.91
126.26
69.14
21.00
16.30
f1 (ppm)
7.61
7.61
7.60
7.59
7.59
7.59
7.57
7.57
7.56
7.55
7.54
7.50
7.49
7.49
7.47
7.47
7.46
7.44
7.43
7.42
7.42
7.36
7.36
7.35
7.34
5.82
5.82
5.81
5.81
5.80
5.79
5.79
5.79
5.73
5.72
5.71
5.70
5.70
5.69
5.68
5.66
4.62
2.17
2.17
1.41
1.39
O
O
NH2
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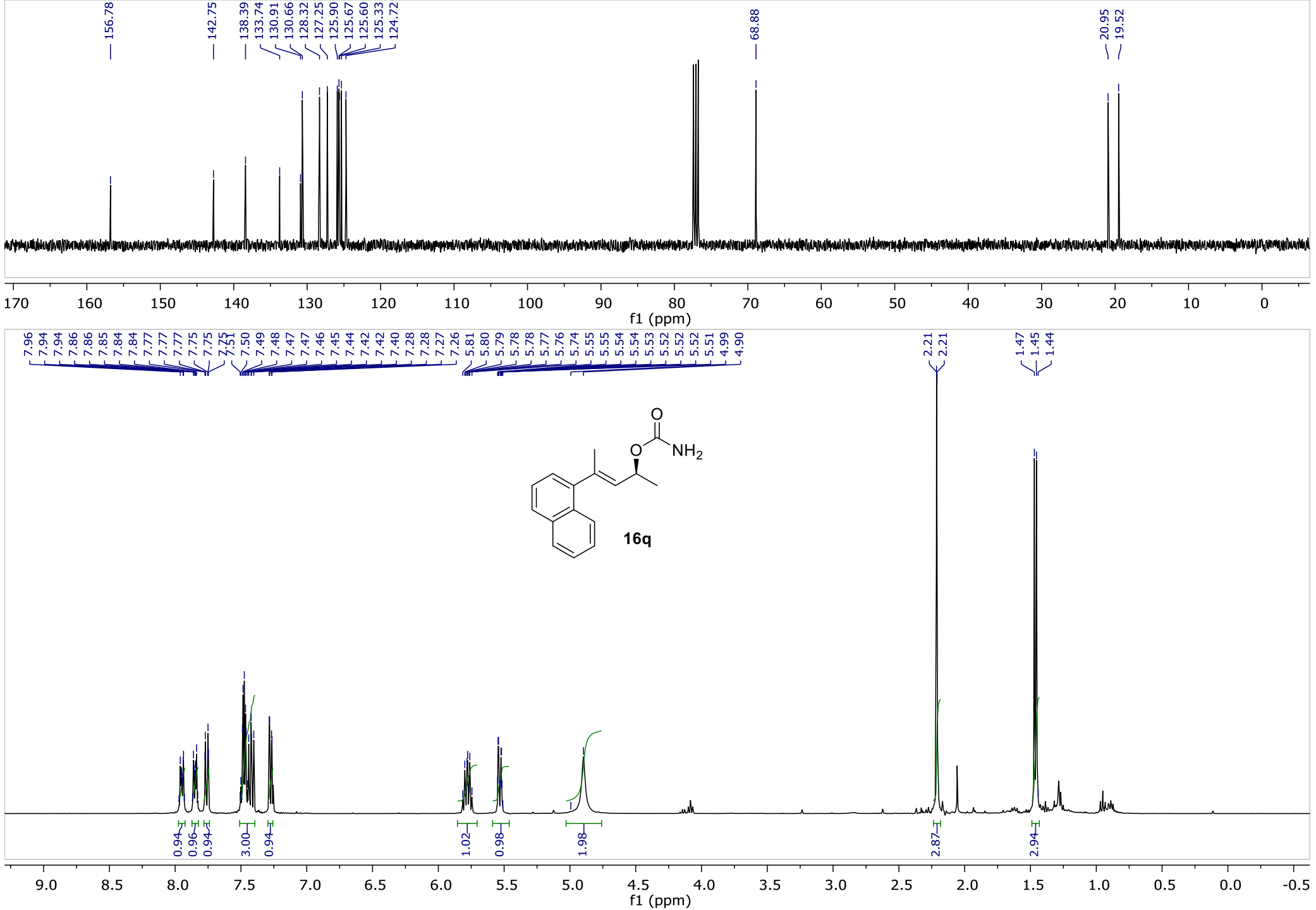
4.37
4.32
1.27
1.00
1.03
2.26
3.17
3.23
f1 (ppm)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127.25
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125.67
125.60
125.33
124.72
68.88
20.95
19.52
f1 (ppm)
7.96
7.94
7.94
7.86
7.86
7.85
7.84
7.84
7.77
7.77
7.77
7.75
7.75
7.51
7.50
7.49
7.48
7.47
7.47
7.46
7.45
7.44
7.42
7.42
7.40
7.28
7.28
7.27
7.26
5.81
5.80
5.79
5.78
5.78
5.77
5.76
5.74
5.55
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5.54
5.53
5.52
5.52
5.52
5.51
4.99
4.90
2.21
2.21
1.47
1.45
1.44
O
O
NH2
16q
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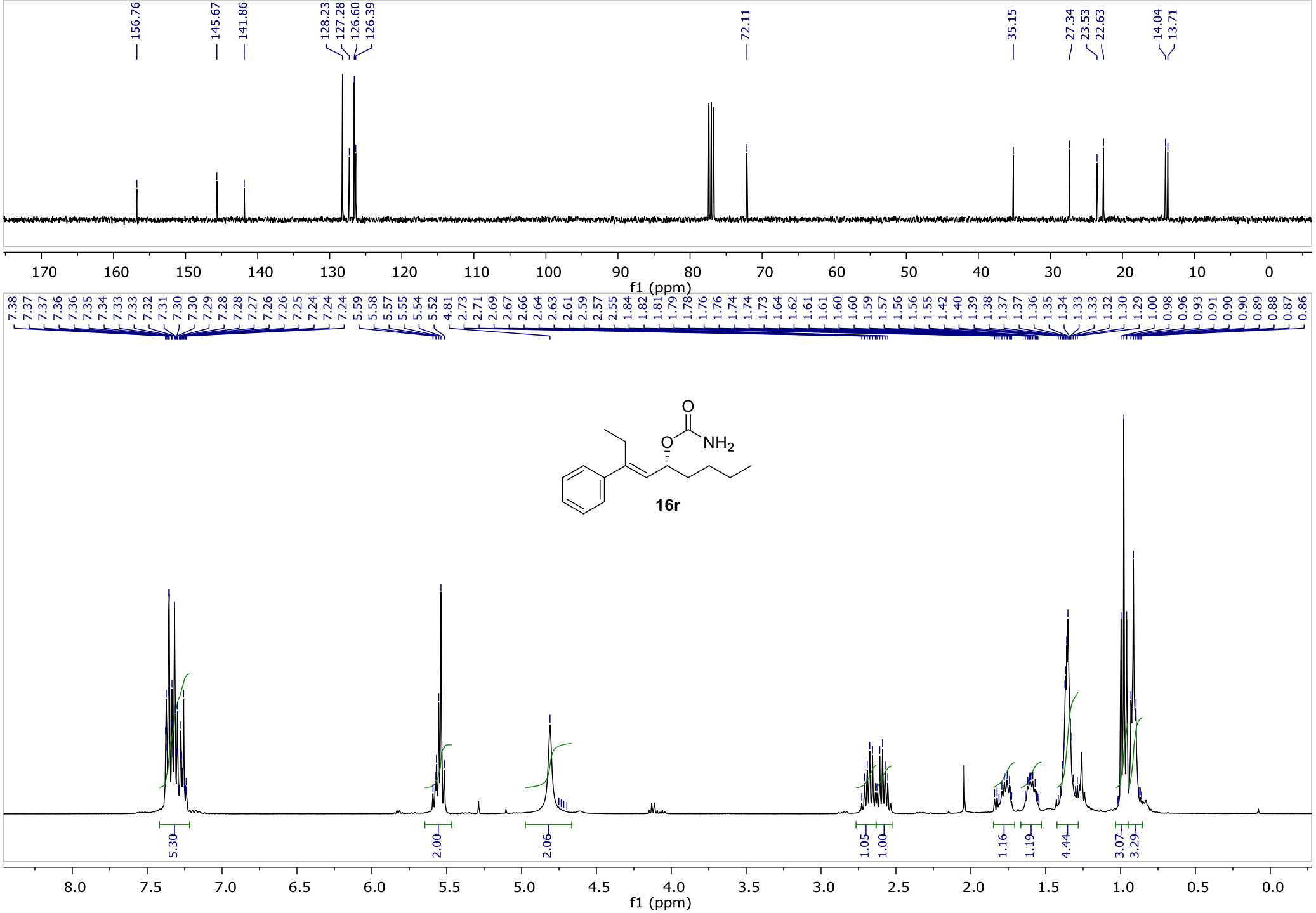
0.94
3.00
0.94
1.02
0.98
1.98
2.87
2.94
f1 (ppm)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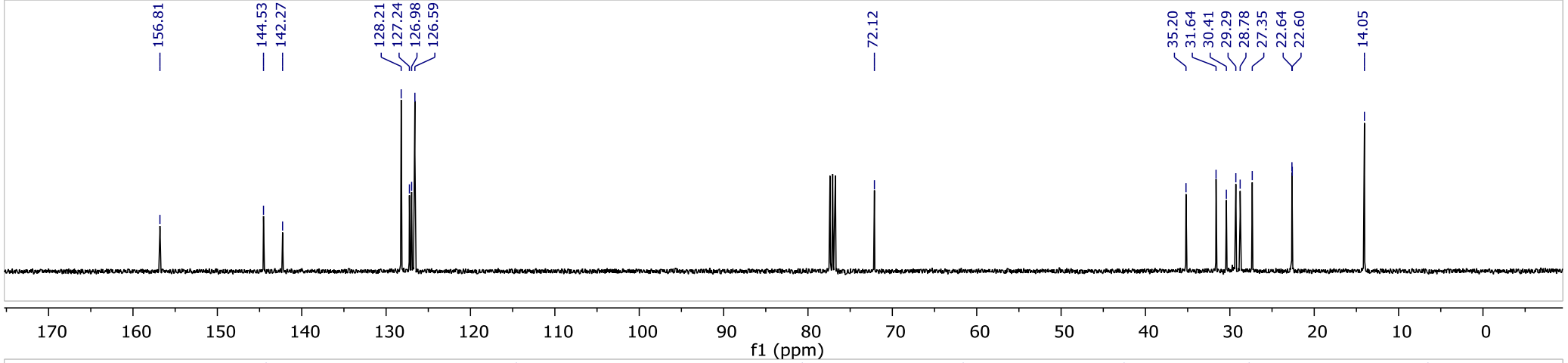
126.60
126.39
72.11
35.15
27.34
23.53
22.63
14.04
13.71
f1 (ppm)
O
O
NH2
16r
5.30
2.00
2.06
1.05
1.00
1.16
1.19
4.44
3.07
3.29
f1 (ppm)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127.24
126.98
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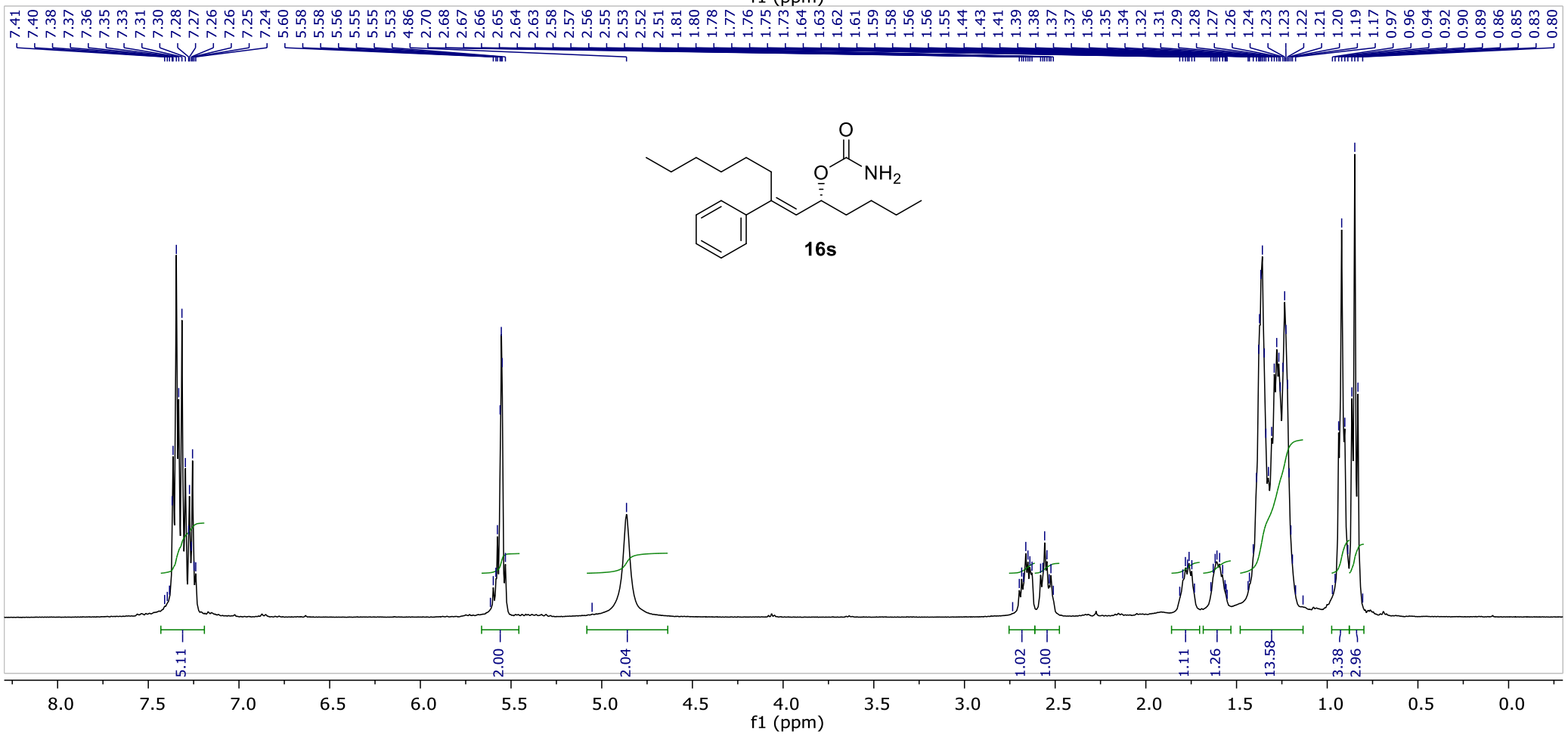
72.12
35.20
31.64
30.41
29.29
28.78
27.35
22.64
22.60
14.05
f1 (ppm)


O
O
NH2
16s
5.11
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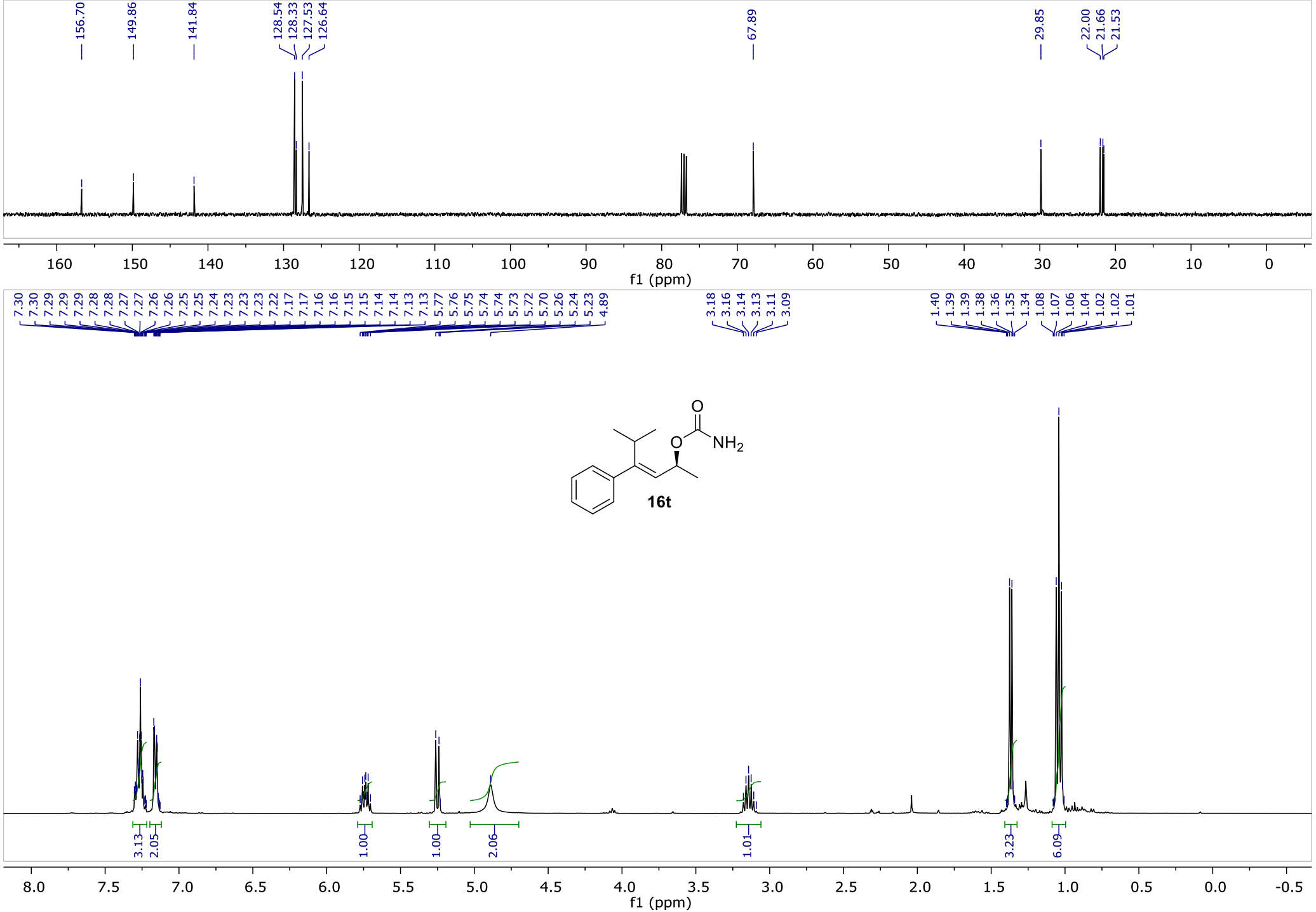
2.04
1.02
1.00
1.11
1.26
13.58
3.38
2.96
f1 (ppm)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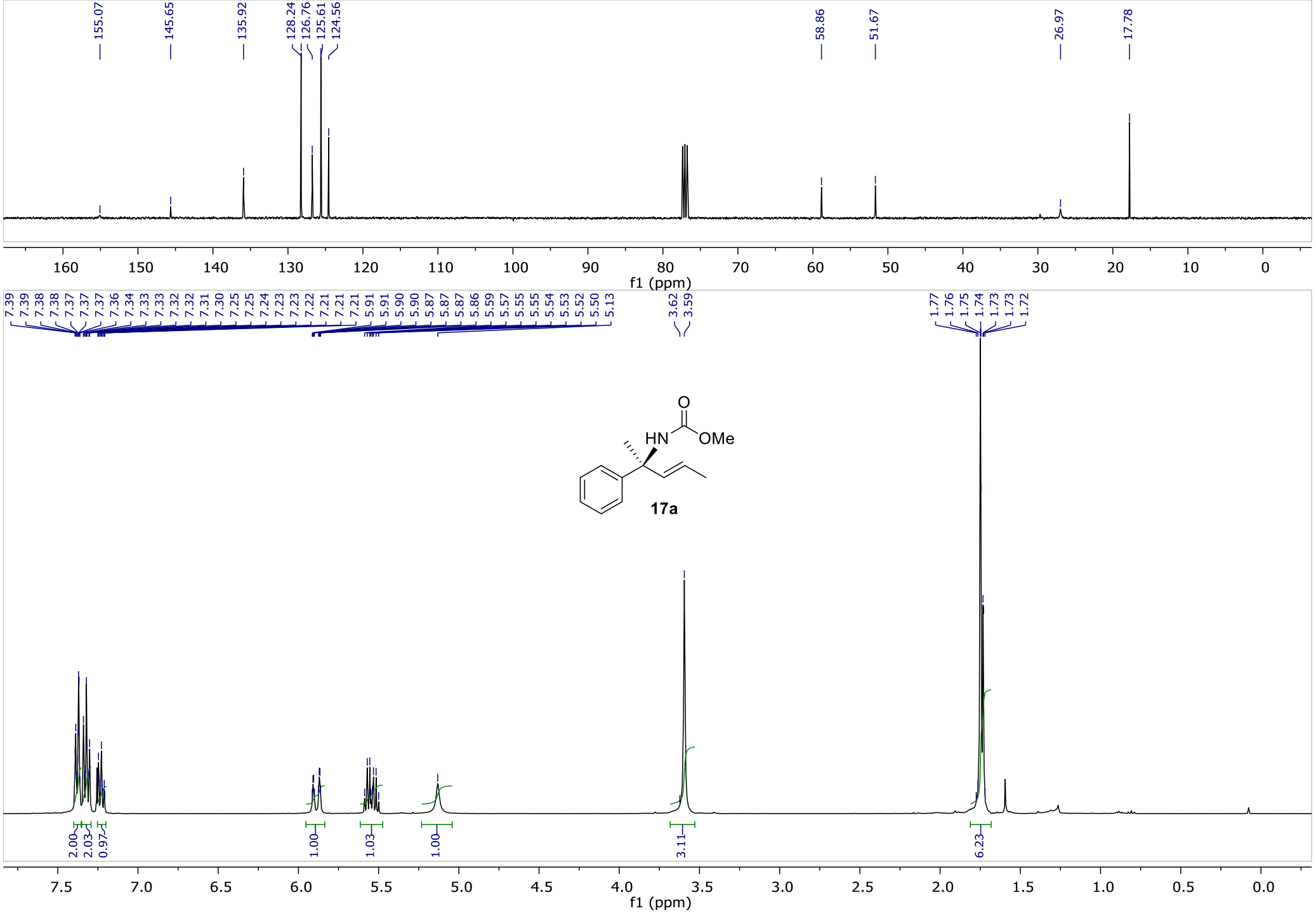
127.53
126.64
67.89
29.85
22.00
21.66
21.53
f1 (ppm)
7.30
7.30
7.29
7.29
7.29
7.28
7.28
7.27
7.27
7.26
7.26
7.25
7.25
7.24
7.23
7.23
7.23
7.22
7.17
7.17
7.16
7.16
7.15
7.15
7.14
7.14
7.13
7.13
5.77
5.76
5.75
5.74
5.74
5.73
5.72
5.70
5.26
5.24
5.23
4.89
3.18
3.16
3.14
3.13
3.11
3.09
1.40
1.39
1.39
1.38
1.36
1.35
1.34
1.08
1.07
1.06
1.04
1.02
1.02
1.01
O
O
NH2
16t
3.13
2.05
1.00
1.00
2.06
1.01
3.23
6.09
f1 (ppm)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125.61
124.56
58.86
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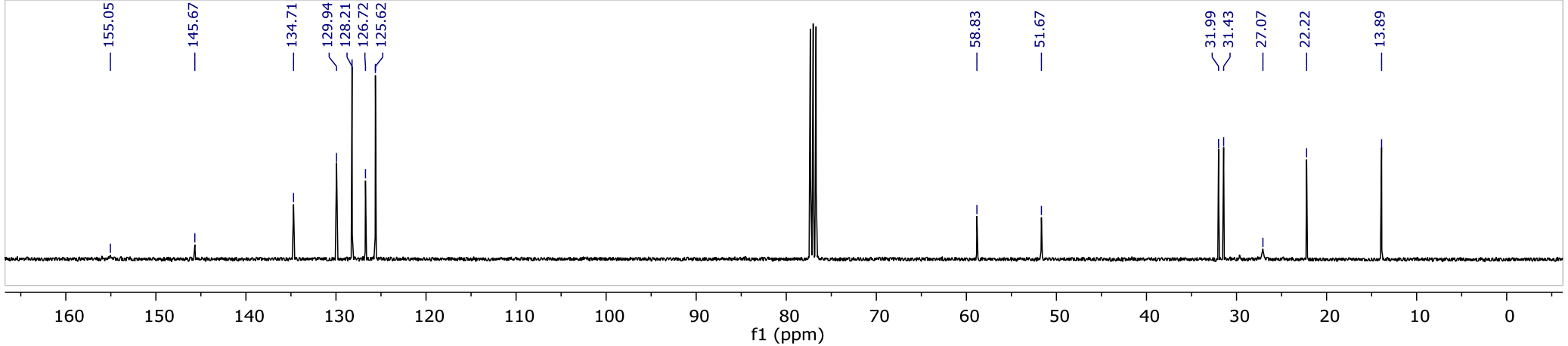
26.97
17.78
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)
7.39
7.39
7.38
7.38
7.37
7.37
7.37
7.36
7.34
7.33
7.33
7.32
7.32
7.31
7.30
7.25
7.25
7.24
7.23
7.23
7.22
7.21
7.21
7.21
5.91
5.91
5.90
5.90
5.87
5.87
5.87
5.86
5.59
5.57
5.55
5.55
5.54
5.53
5.52
5.50
5.13
3.62
3.59
1.77
1.76
1.75
1.74
1.73
1.73
1.72
O
HN
OMe
17a
2.00
2.03
0.97
1.00
1.03
1.00
3.11
6.23
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
f1 (ppm)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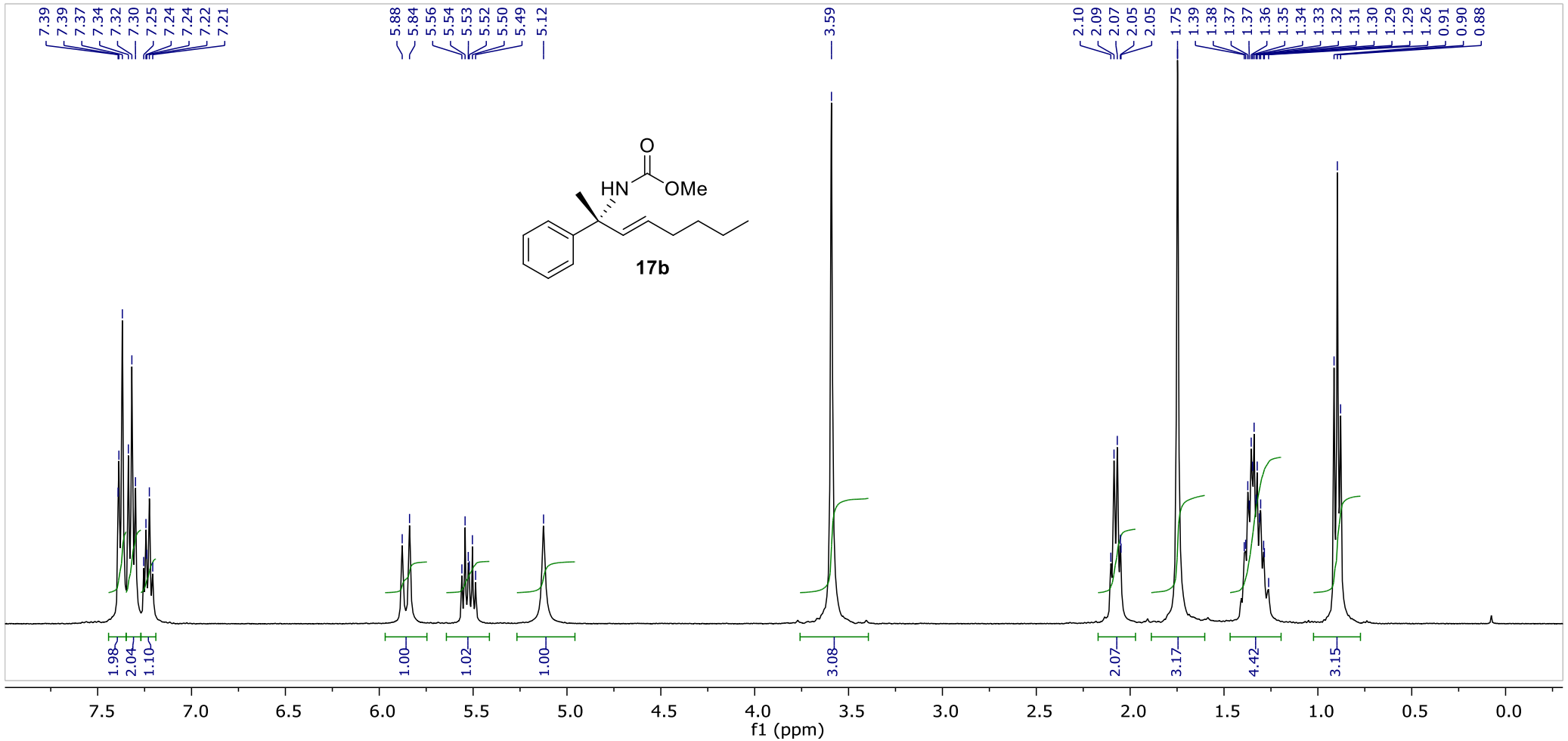
126.72
125.62
58.83
51.67
31.99
31.43
27.07
22.22
13.89
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.32
7.30
7.25
7.24
7.24
7.22
7.21
5.88
5.84
5.56
5.54
5.53
5.52
5.50
5.49
5.12
3.59
2.10
2.09
2.07
2.05
2.05
1.75
1.39
1.38
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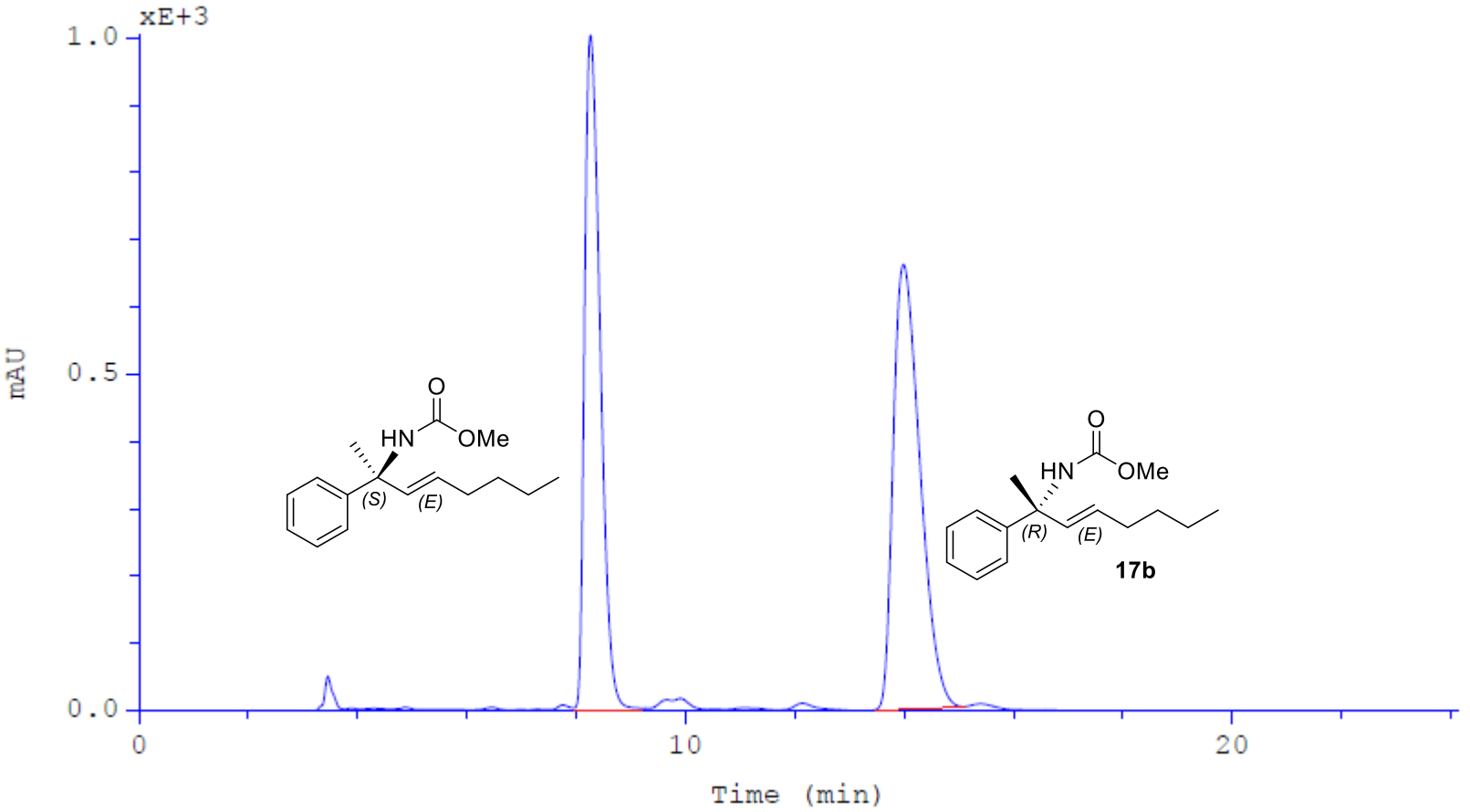
1.37
1.36
1.35
1.34
1.33
1.32
1.31
1.30
1.29
1.29
1.26
0.91
0.90
0.88
O
HN
OMe
17b
1.98
2.04
1.10
1.00
1.02
1.00
3.08
2.07
3.17
4.42
3.15
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

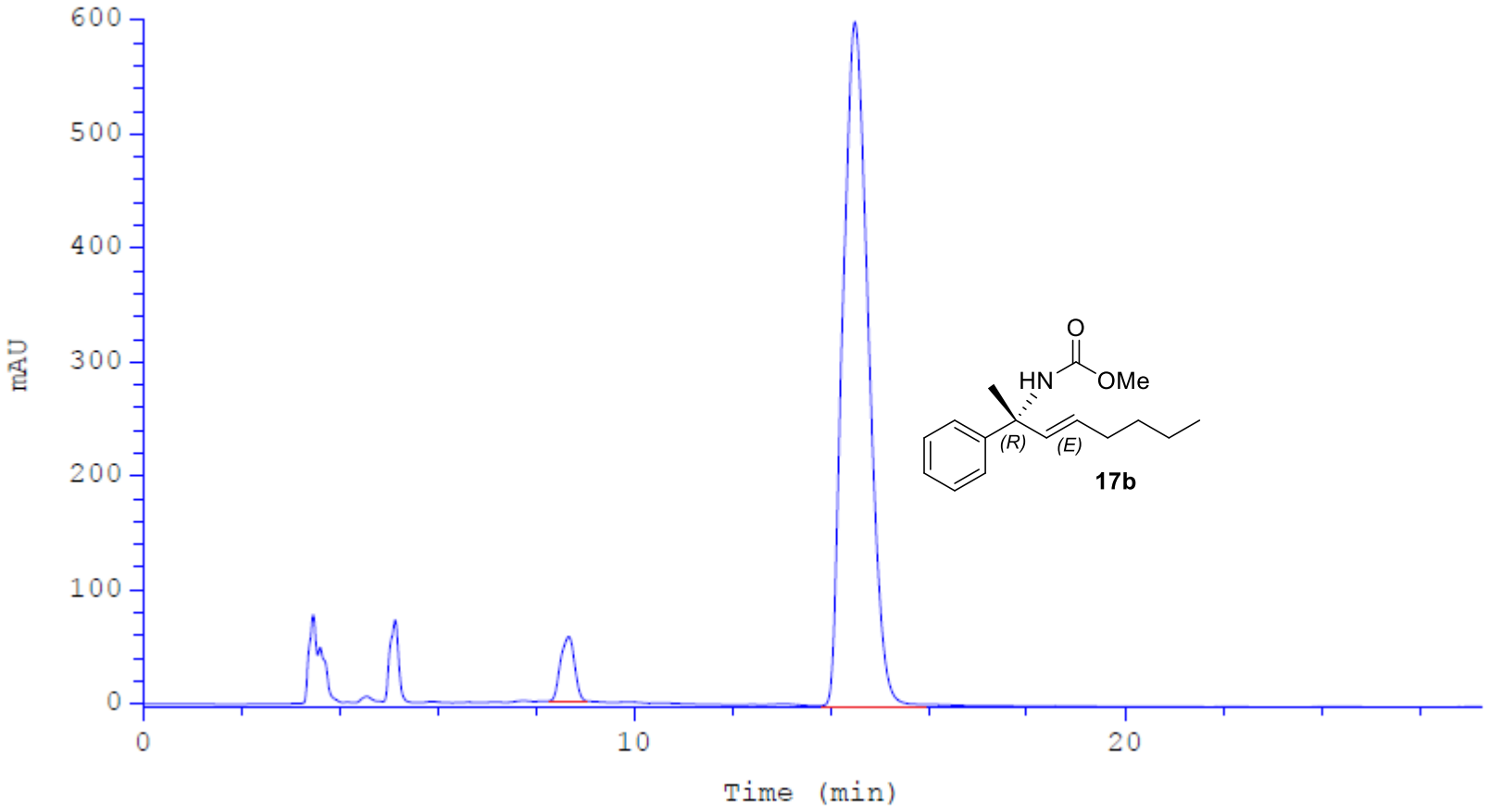
Column: Chiralcel OD-H
Eluent: 5% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 215 nm

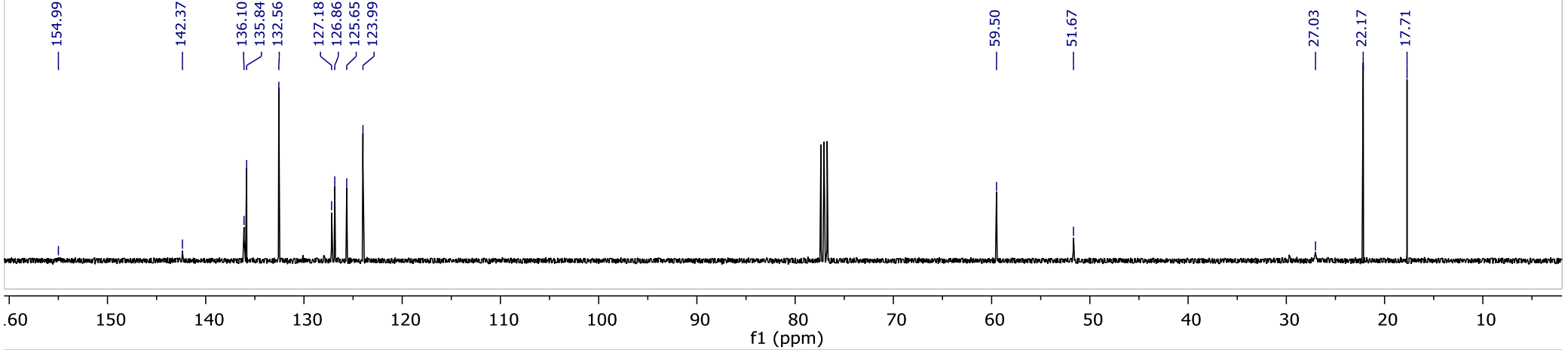
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	8.253	7.96	9.11	328.771	999.66	46.9562	0.312
2	13.989	13.48	14.93	371.394	659.341	53.0438	0.536

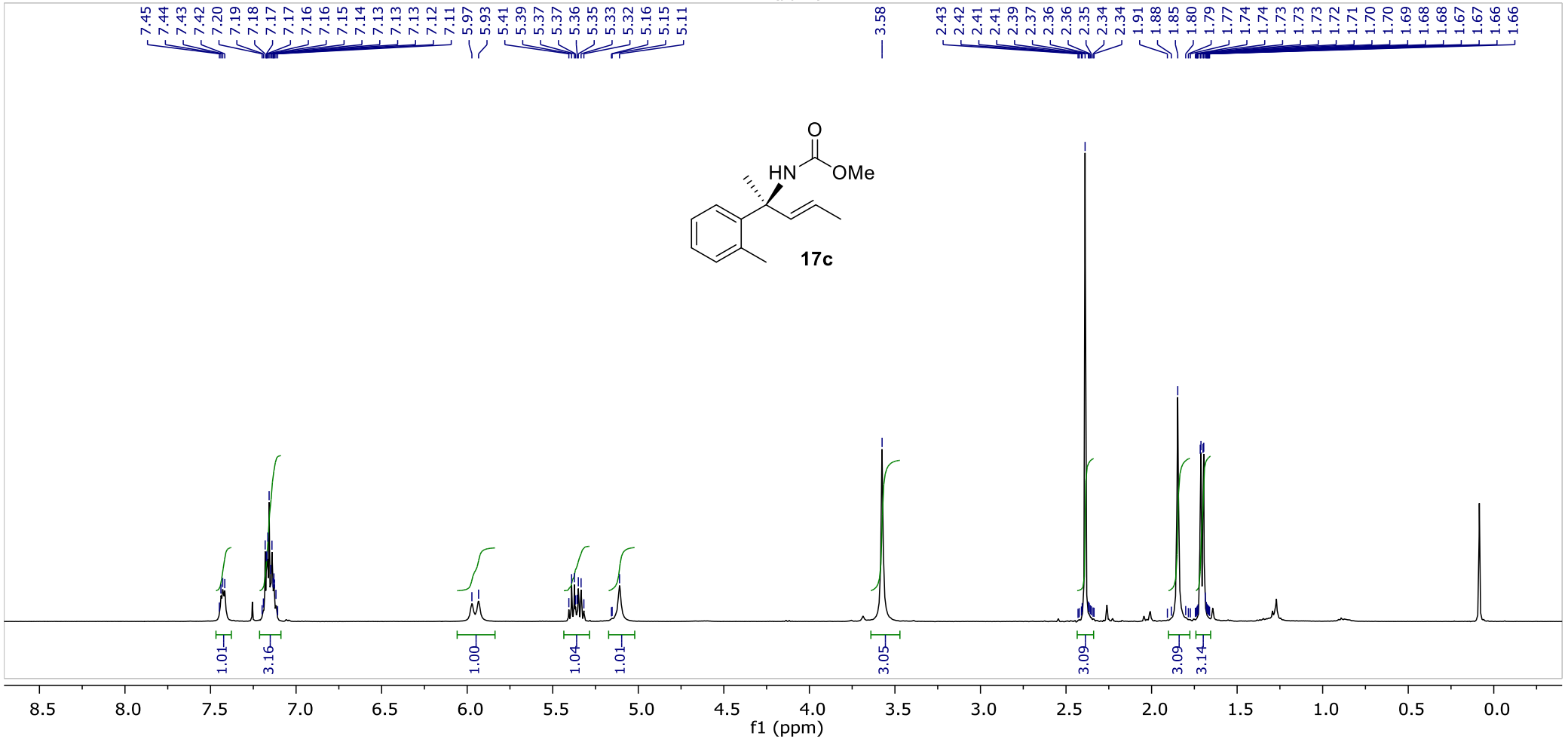


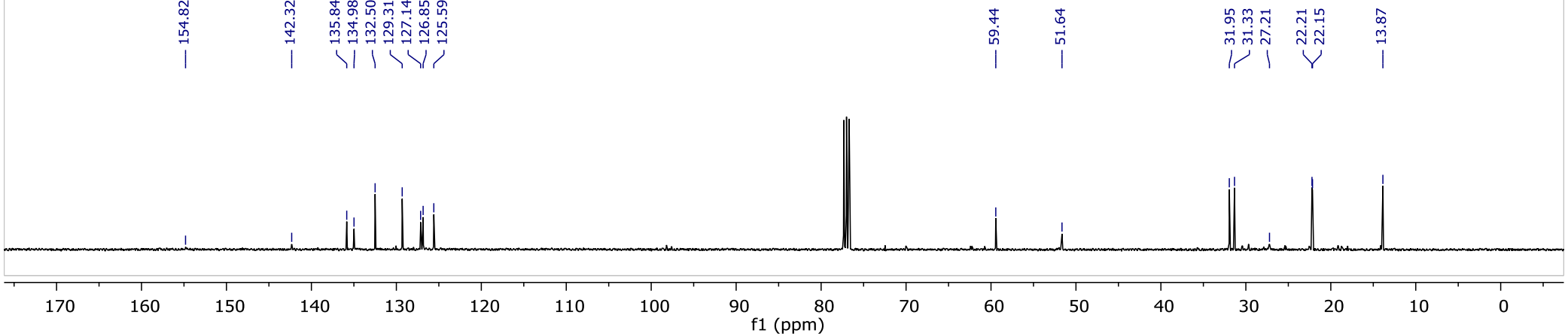
Column: Chiralcel OD-H
Eluent: 5% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 215 nm

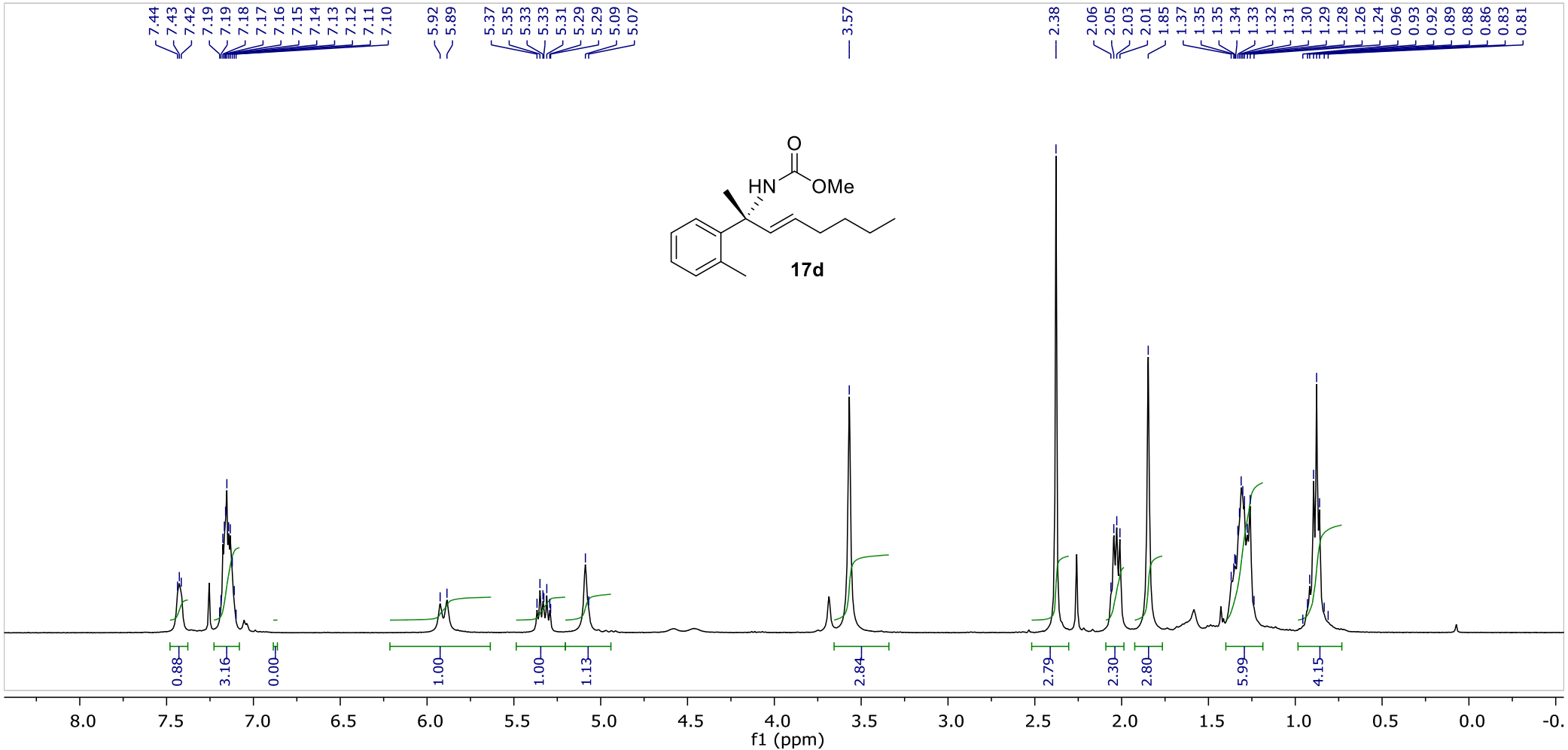
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	8.670	8.38	8.99	16.4914	53.2211	4.4119	0.316
2	14.505	13.84	15.93	357.3	599.355	95.5881	0.582




154.99
142.37
136.10
135.84
132.56
127.18
126.86
125.65
123.99
59.50
51.67
27.03
22.17
17.71
f1 (ppm)

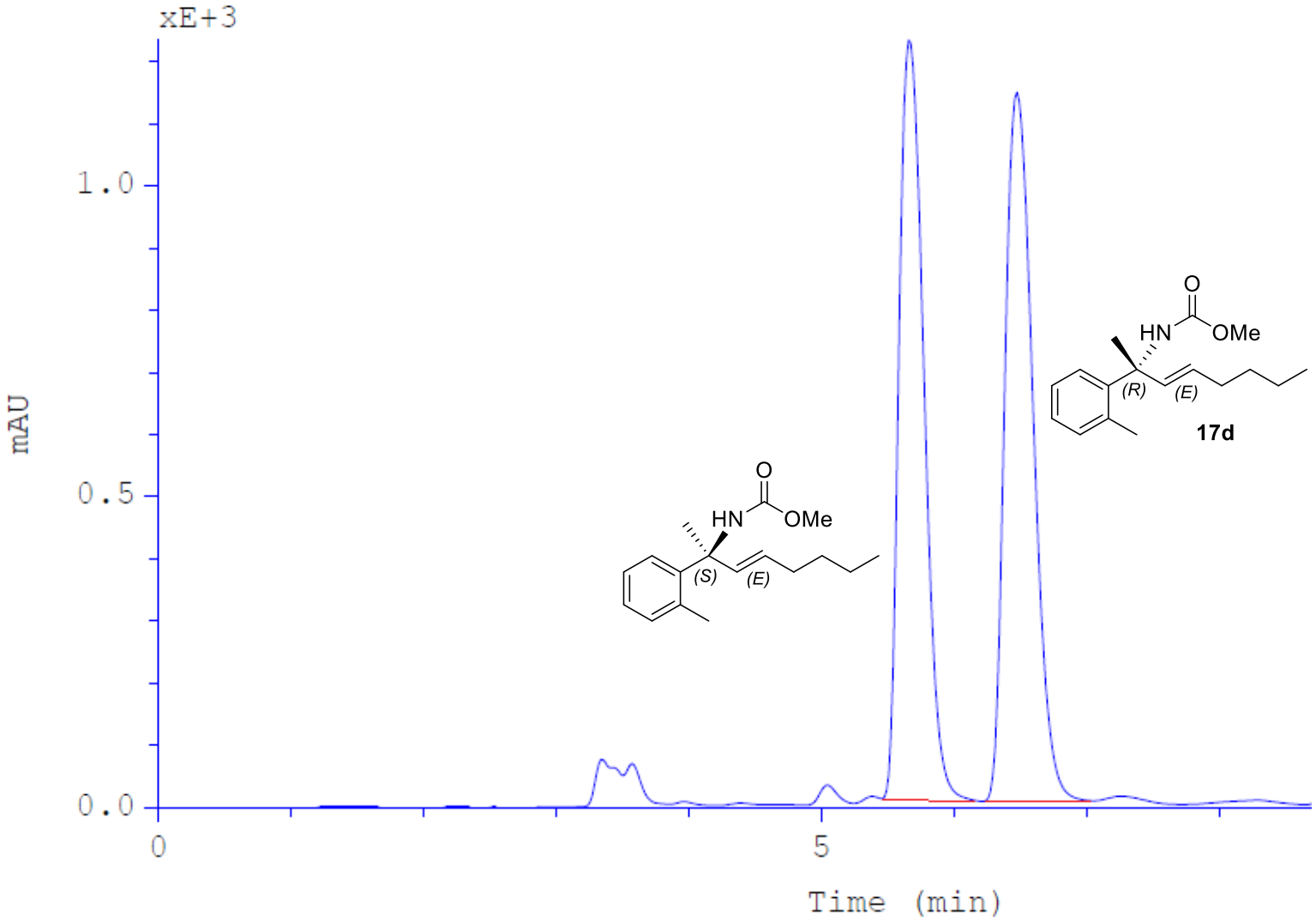

7.45
7.44
7.43
7.42
7.20
7.19
7.18
7.17
7.17
7.16
7.16
7.15
7.14
7.13
7.13
7.13
7.12
7.11
5.97
5.93
5.41
5.39
5.37
5.37
5.36
5.35
5.33
5.32
5.16
5.15
5.11
3.58
2.43
2.42
2.41
2.41
2.39
2.37
2.36
2.36
2.35
2.34
2.34
1.91
1.88
1.85
1.80
1.79
1.77
1.74
1.74
1.73
1.73
1.72
1.71
1.70
1.70
1.69
1.68
1.68
1.67
1.67
1.66
1.66
O
HN
OMe
17c
1.01
3.16
1.00
1.04
1.01
3.05
3.09
3.09
3.14
f1 (ppm)


154.82
142.32
135.84
134.98
132.50
129.31
127.14
126.85
125.59
59.44
51.64
31.95
31.33
27.21
22.21
22.15
13.87
f1 (ppm)


7.44
7.43
7.42
7.19
7.19
7.18
7.17
7.16
7.15
7.14
7.13
7.12
7.11
7.10
5.92
5.89
5.37
5.35
5.33
5.33
5.31
5.29
5.29
5.09
5.07
3.57
2.38
2.06
2.05
2.03
2.01
1.85
1.37
1.35
1.35
1.34
1.33
1.32
1.31
1.30
1.29
1.28
1.26
1.24
0.96
0.93
0.92
0.89
0.88
0.86
0.83
0.81
HN
O
OMe
17d
0.88
3.16
0.00
1.00
1.00
1.13
2.84
2.79
2.30
2.80
5.99
4.15
f1 (ppm)

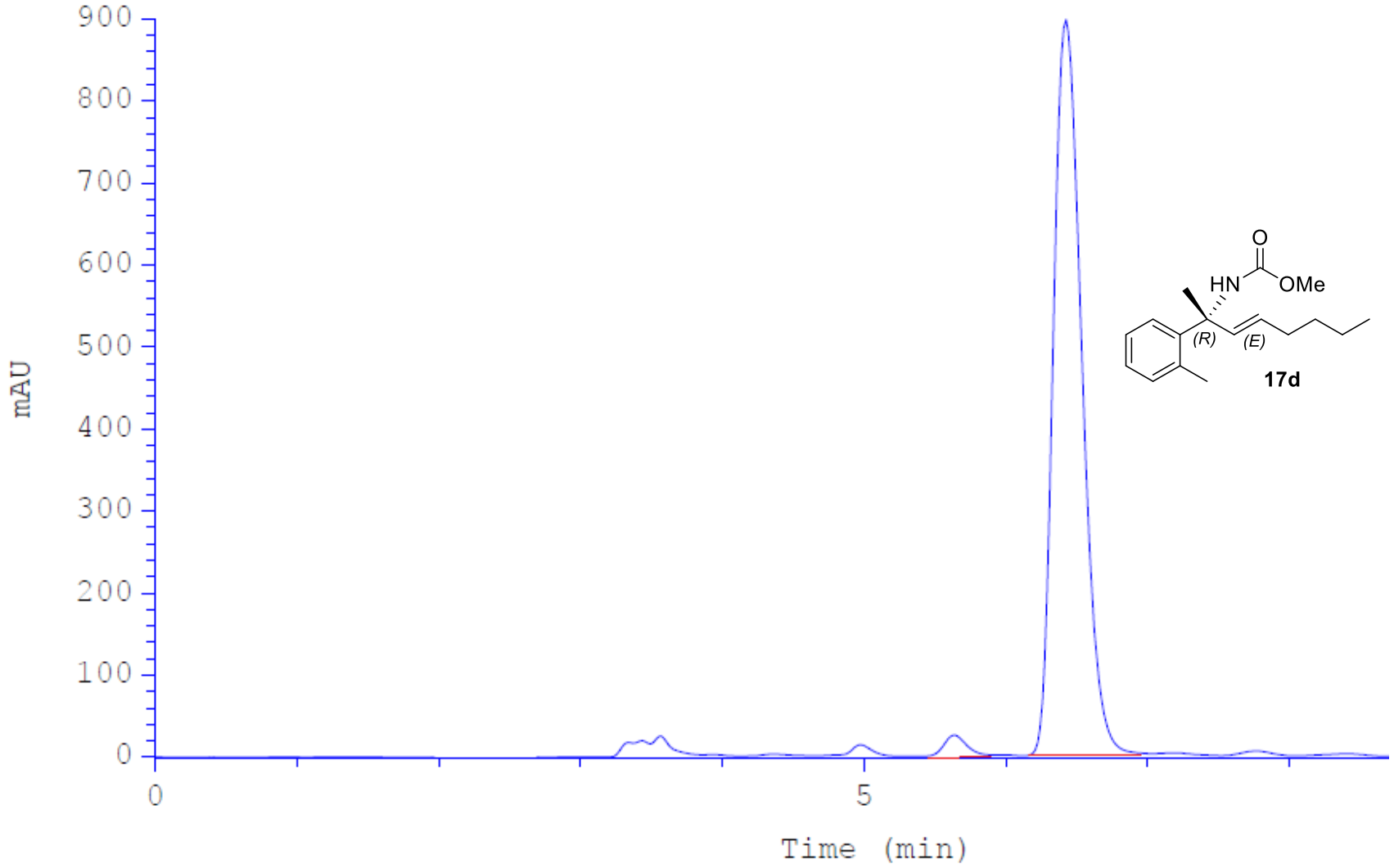
Column: Chiralcel OD-H
Eluent: 5% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 215 nm

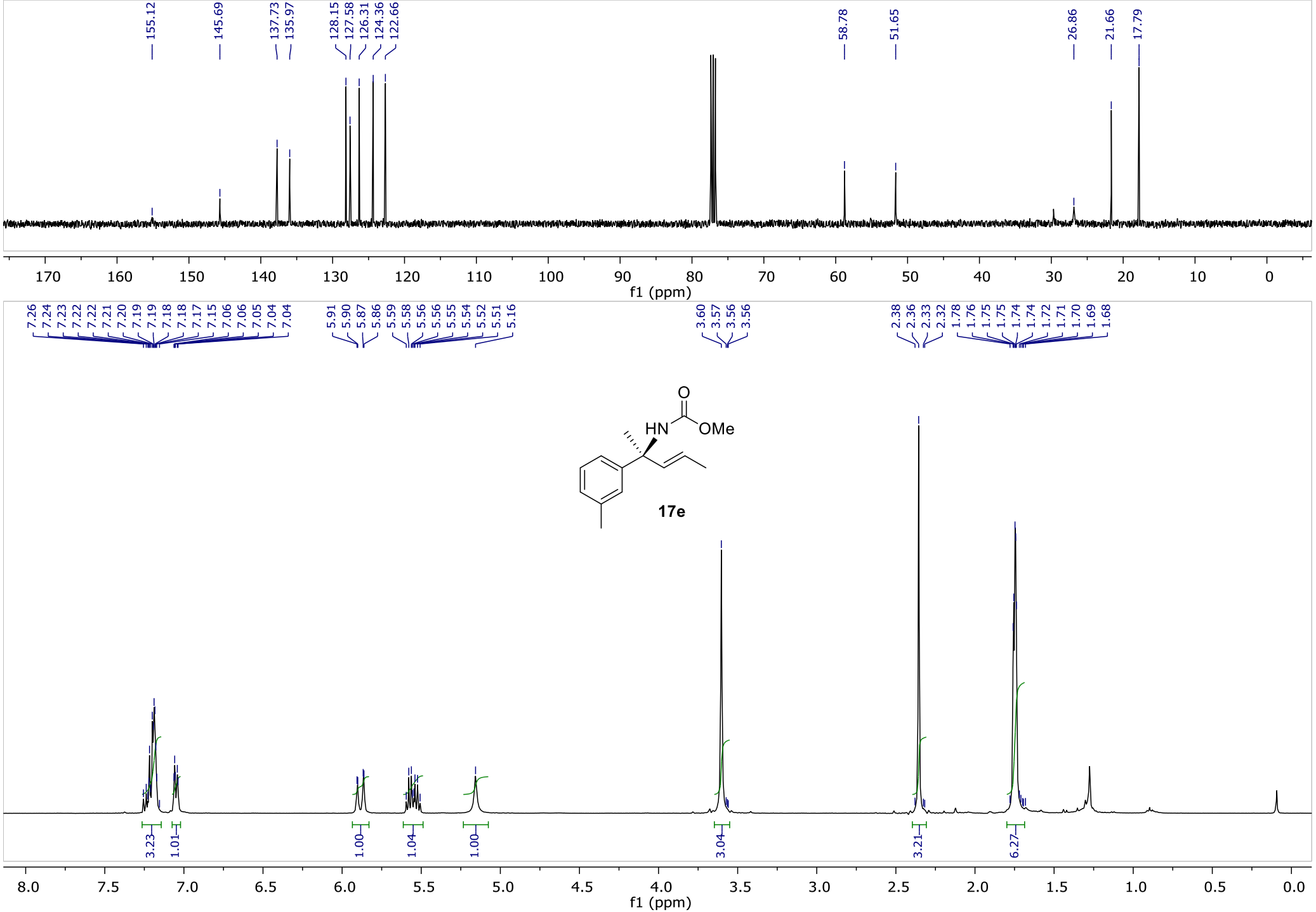
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	5.657	5.46	6.16	265.642	1221.88	48.6358	0.208
2	6.474	6.24	7.01	280.544	1140.44	51.3642	0.234



Column: Chiralcel OD-H
Eluent: 5% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 215 nm

	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	5.641	5.49	5.86	3.97294	23.7872	1.8836	0.159
2	6.425	6.16	6.88	206.949	895.261	98.1164	0.218

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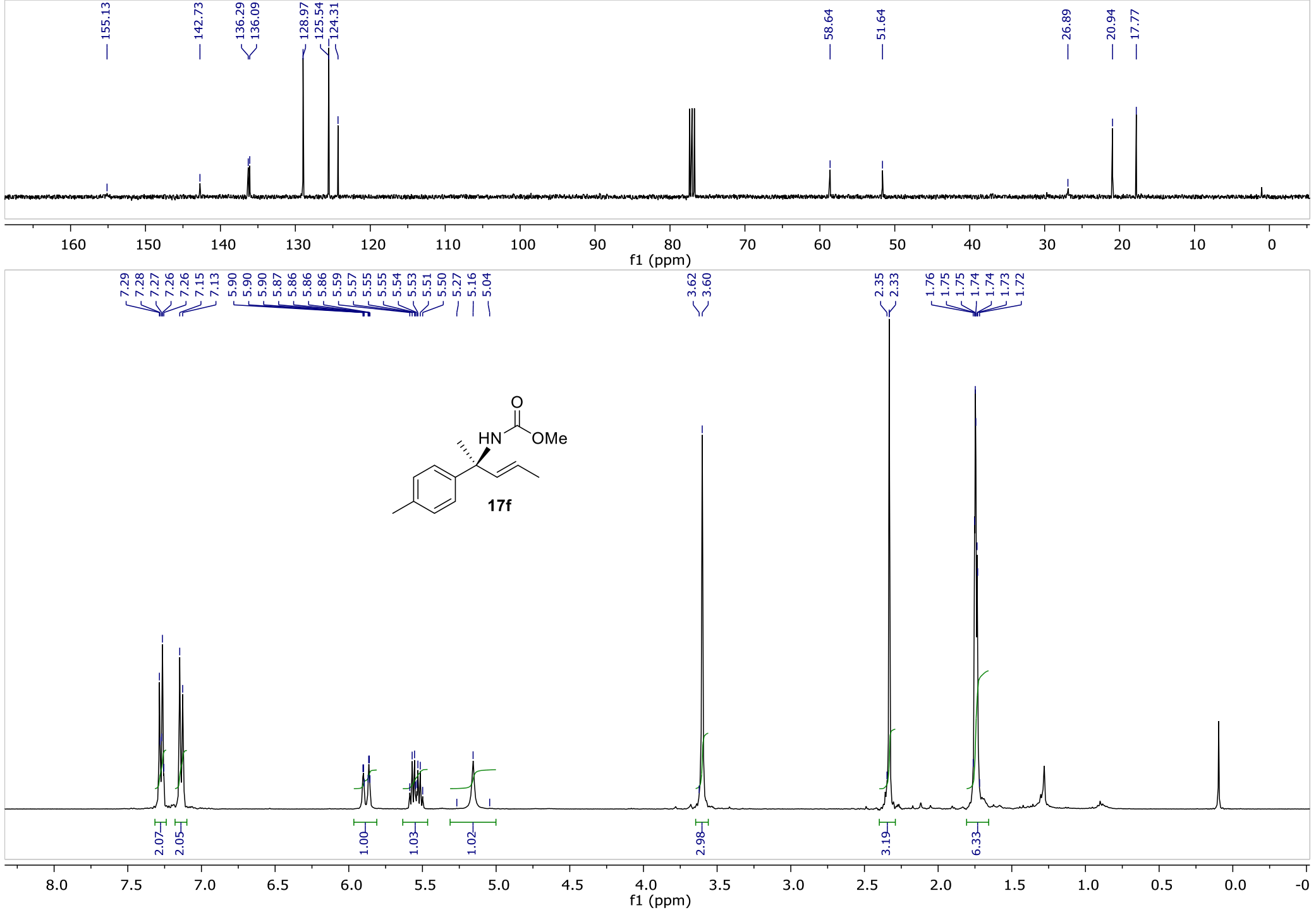
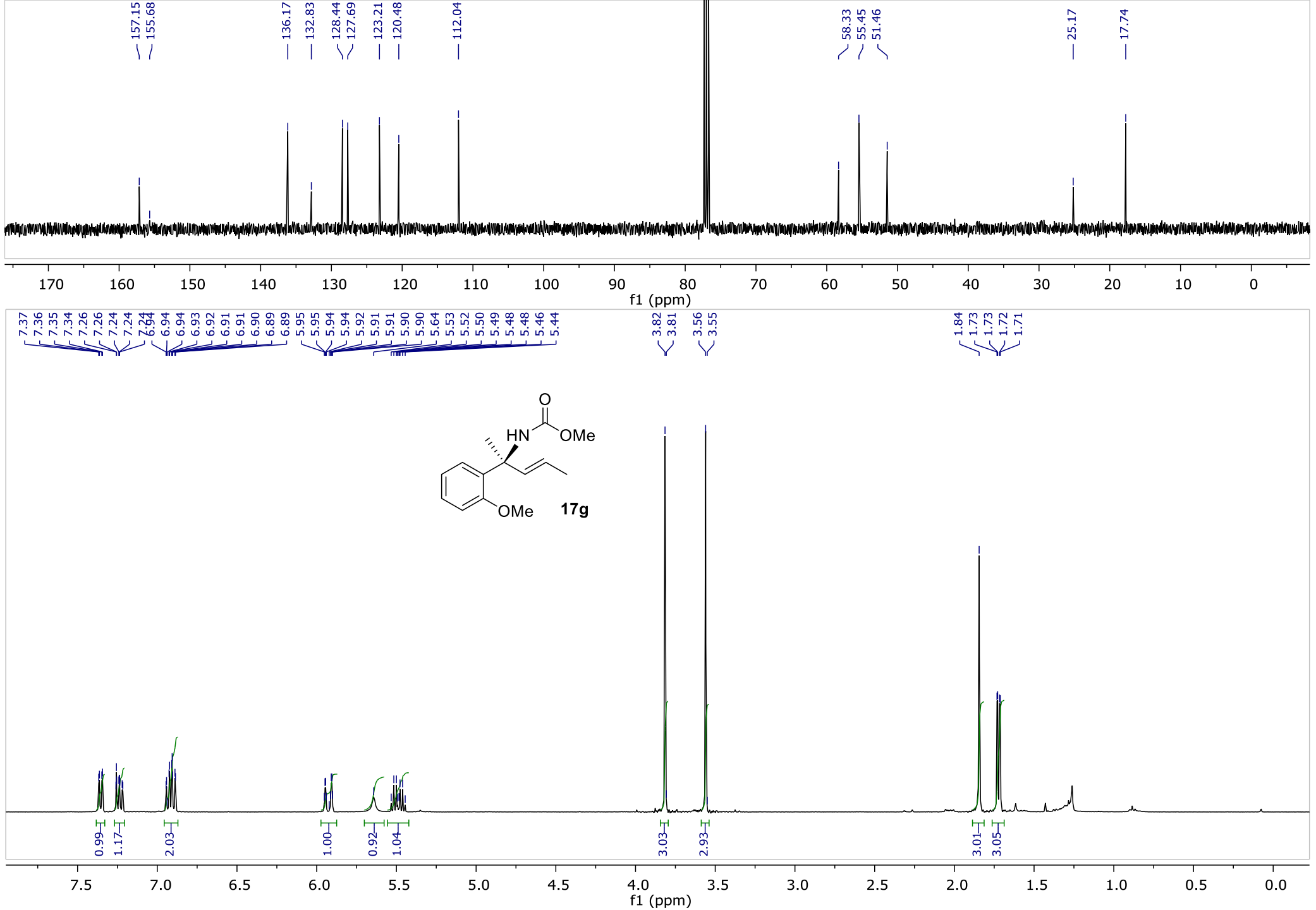
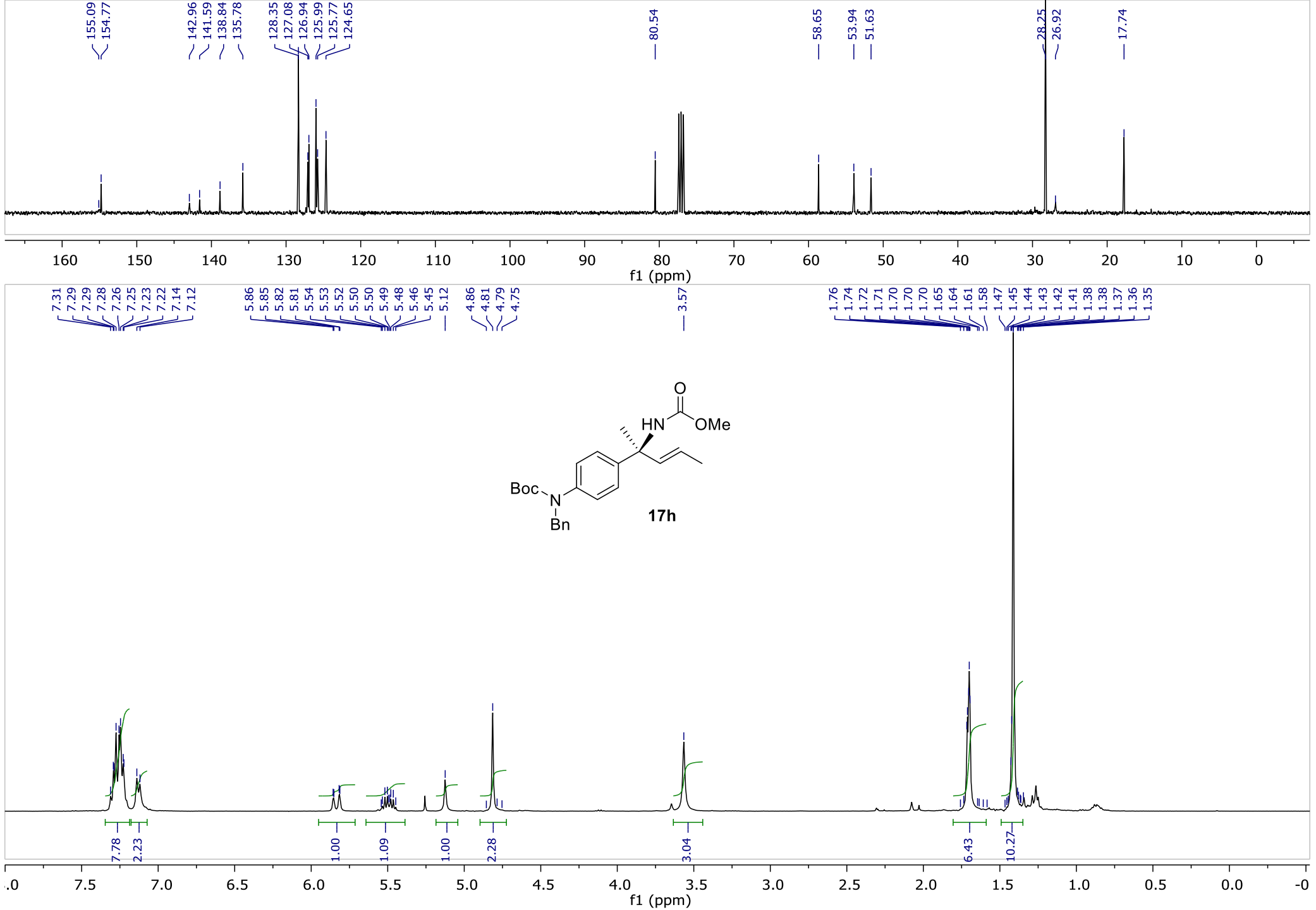
145.69
137.73
135.97
128.15
127.58
126.31
124.36
122.66
58.78
51.65
26.86
21.66
17.79
f1 (ppm)
7.26
7.24
7.23
7.22
7.22
7.21
7.20
7.19
7.19
7.18
7.18
7.17
7.15
7.06
7.06
7.05
7.04
7.04
5.91
5.90
5.87
5.86
5.59
5.58
5.56
5.56
5.55
5.54
5.52
5.51
5.16
3.60
3.57
3.56
3.56
2.38
2.36
2.33
2.32
1.78
1.76
1.75
1.75
1.74
1.74
1.72
1.71
1.70
1.69
1.68
O
HN
OMe
17e
3.23
1.01
1.00
1.04
1.00
3.04
3.21
6.27
f1 (ppm)


155.13
142.73
136.29
136.09
128.97
125.54
124.31
58.64
51.64
26.89
20.94
17.77
f1 (ppm)
7.29
7.28
7.27
7.26
7.26
7.15
7.13
5.90
5.90
5.90
5.87
5.86
5.86
5.86
5.59
5.57
5.55
5.55
5.54
5.53
5.51
5.50
5.27
5.16
5.04
3.62
3.60
2.35
2.33
1.76
1.75
1.75
1.74
1.74
1.73
1.72
O
HN
OMe
17f
2.07
2.05
1.00
1.03
1.02
2.98
3.19
6.33
f1 (ppm)


157.15
155.68
136.17
132.83
128.44
127.69
123.21
120.48
112.04
58.33
55.45
51.46
25.17
17.74
f1 (ppm)
7.37
7.36
7.35
7.34
7.26
7.24
6.94
6.93
6.92
6.91
6.90
6.89
5.95
5.94
5.92
5.91
5.90
5.64
5.53
5.52
5.50
5.49
5.48
5.46
5.44
3.82
3.81
3.56
3.55
1.84
1.73
1.72
1.71
HN
OMe
OMe
17g
0.99
1.17
2.03
1.00
0.92
1.04
3.03
2.93
3.01
3.05
f1 (ppm)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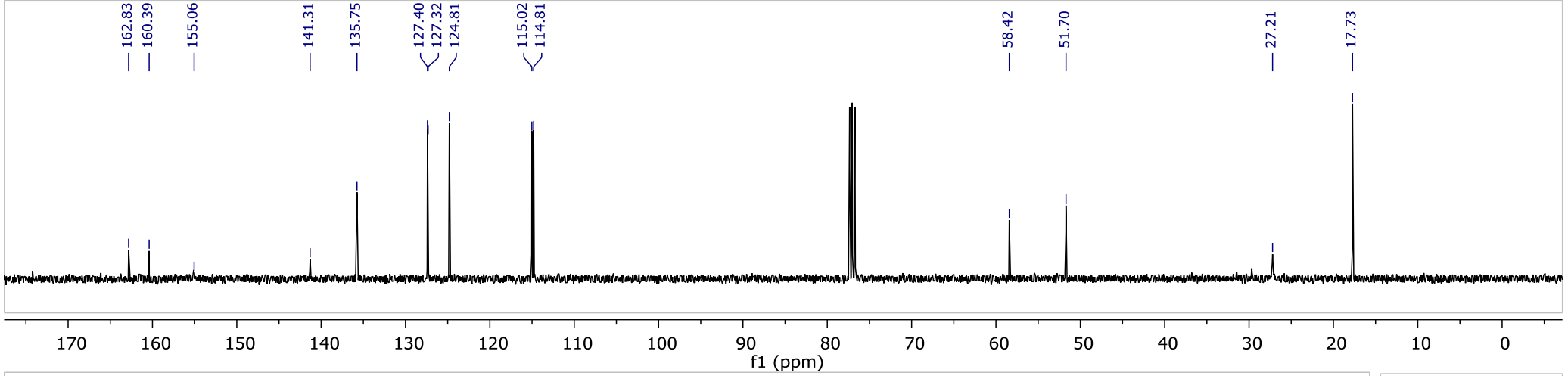
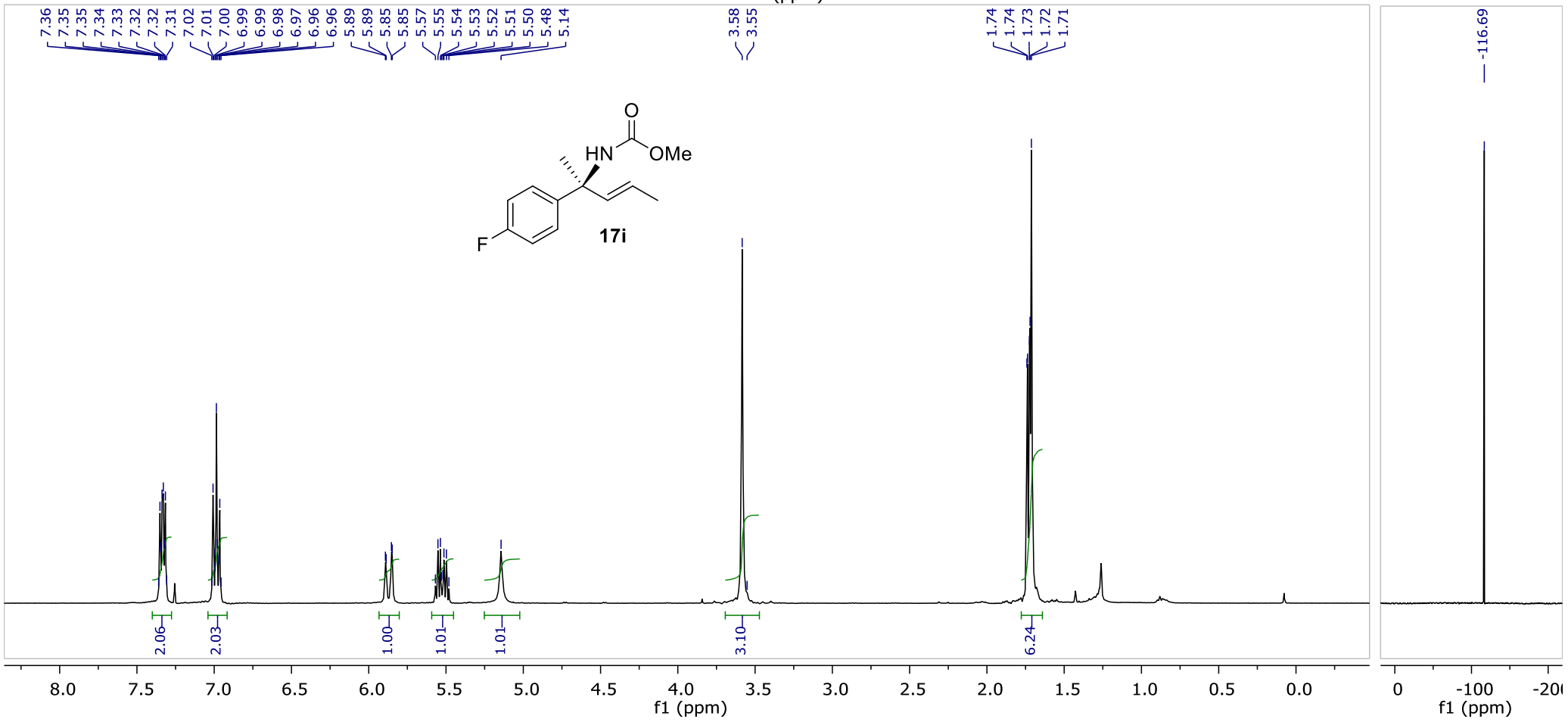
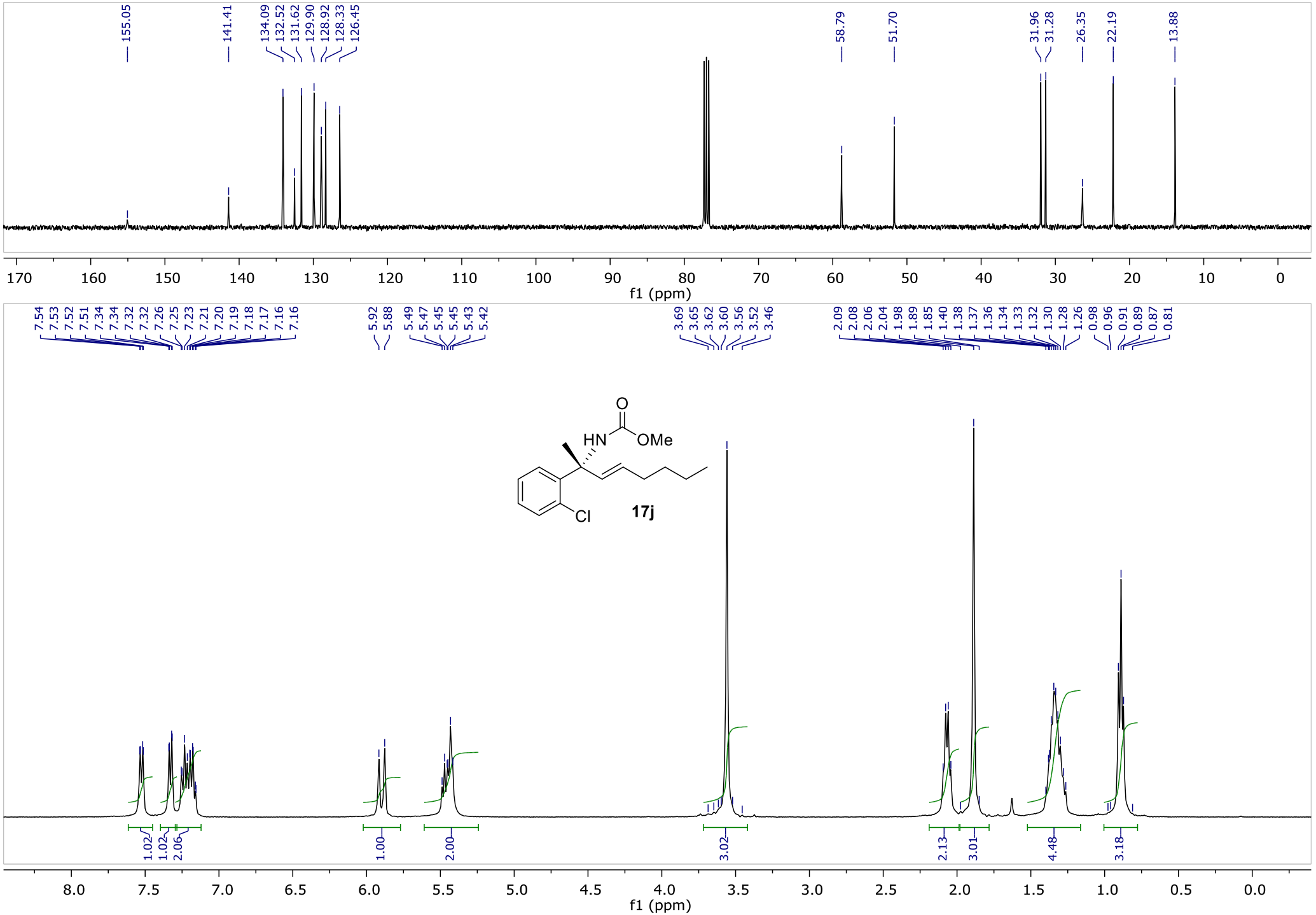
141.59
138.84
135.78
128.35
127.08
126.94
125.99
125.77
124.65
80.54
58.65
53.94
51.63
28.25
26.92
17.74
f1 (ppm)
7.31
7.29
7.29
7.28
7.26
7.25
7.23
7.22
7.14
7.12
5.86
5.85
5.82
5.81
5.54
5.53
5.52
5.50
5.50
5.49
5.48
5.46
5.45
5.12
4.86
4.81
4.79
4.75
3.57
1.76
1.74
1.72
1.71
1.70
1.70
1.70
1.65
1.64
1.61
1.58
1.47
1.45
1.44
1.43
1.42
1.41
1.38
1.38
1.37
1.36
1.35
HN
O
OMe
Boc
N
Bn
17h
7.78
2.23
1.00
1.09
1.00
2.28
3.04
6.43
10.27
f1 (ppm)

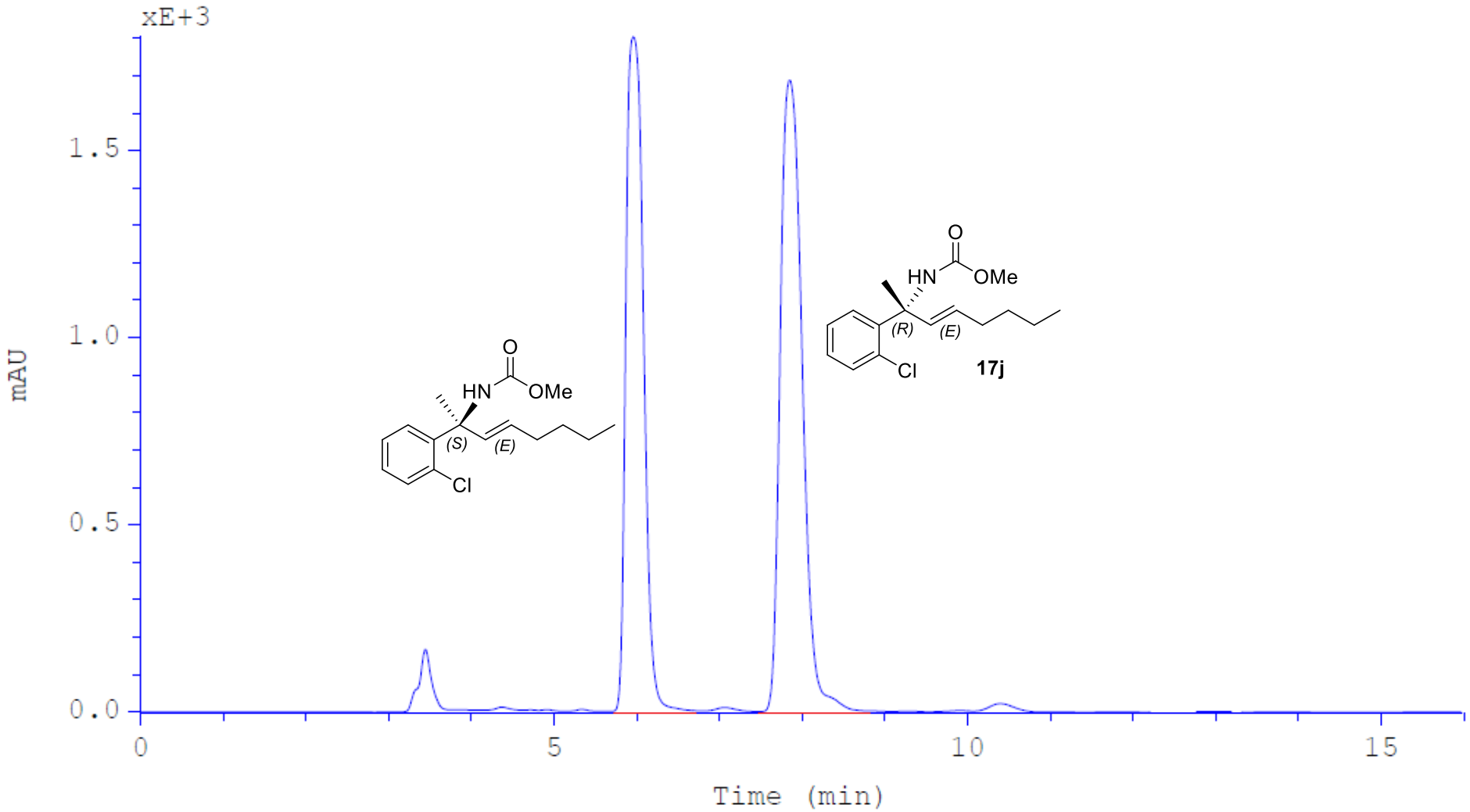

162.83
160.39
155.06
141.31
135.75
127.40
127.32
124.81
115.02
114.81
58.42
51.70
27.21
17.73
f1 (ppm)


7.36
7.35
7.35
7.34
7.33
7.32
7.32
7.31
7.02
7.01
7.00
6.99
6.99
6.98
6.97
6.96
6.96
6.96
5.89
5.89
5.85
5.85
5.57
5.55
5.54
5.53
5.52
5.51
5.50
5.48
5.14
3.58
3.55
1.74
1.74
1.73
1.72
1.71
-116.69
O
HN
OMe
F
17i
2.06
2.03
1.00
1.01
1.01
3.10
6.24
f1 (ppm)
f1 (ppm)


155.05
141.41
134.09
132.52
131.62
129.90
128.92
128.33
126.45
58.79
51.70
31.96
31.28
26.35
22.19
13.88
f1 (ppm)
7.54
7.53
7.52
7.51
7.34
7.34
7.32
7.32
7.26
7.25
7.23
7.21
7.20
7.19
7.18
7.17
7.16
7.16
5.92
5.88
5.49
5.47
5.45
5.45
5.43
5.42
3.69
3.65
3.62
3.60
3.56
3.52
3.46
2.09
2.08
2.06
2.04
1.98
1.89
1.85
1.40
1.38
1.37
1.36
1.34
1.33
1.32
1.30
1.28
1.26
0.98
0.96
0.91
0.89
0.87
0.81
O
HN
OMe
Cl
17j
1.02
1.02
2.06
1.00
2.00
3.02
2.13
3.01
4.48
3.18
f1 (ppm)

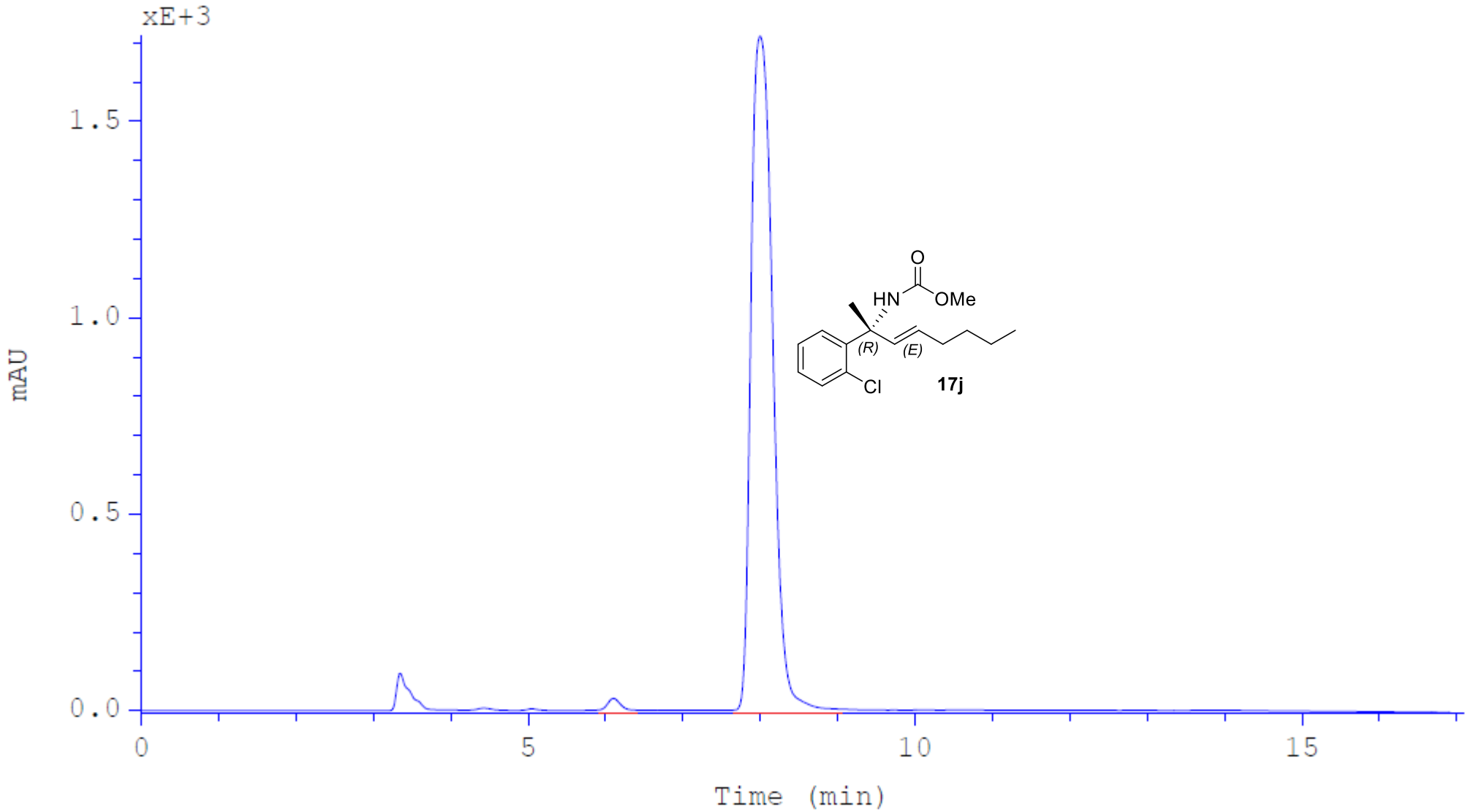
Column: Chiralcel OD-H
Eluent: 5% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 215 nm

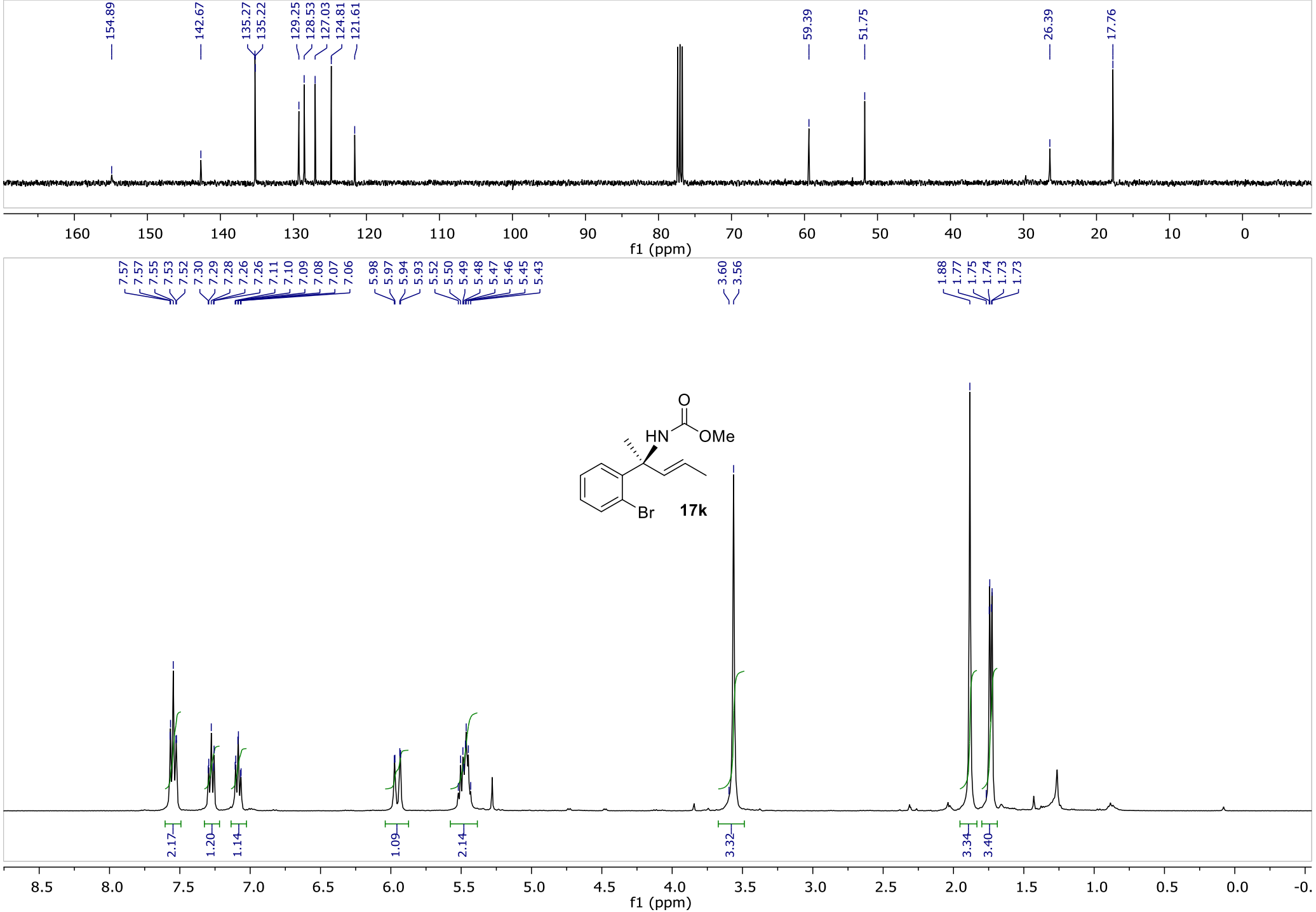
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	5.955	5.72	6.66	443.727	1801.01	46.1824	0.235
2	7.839	7.54	8.71	517.088	1684.65	53.8176	0.287

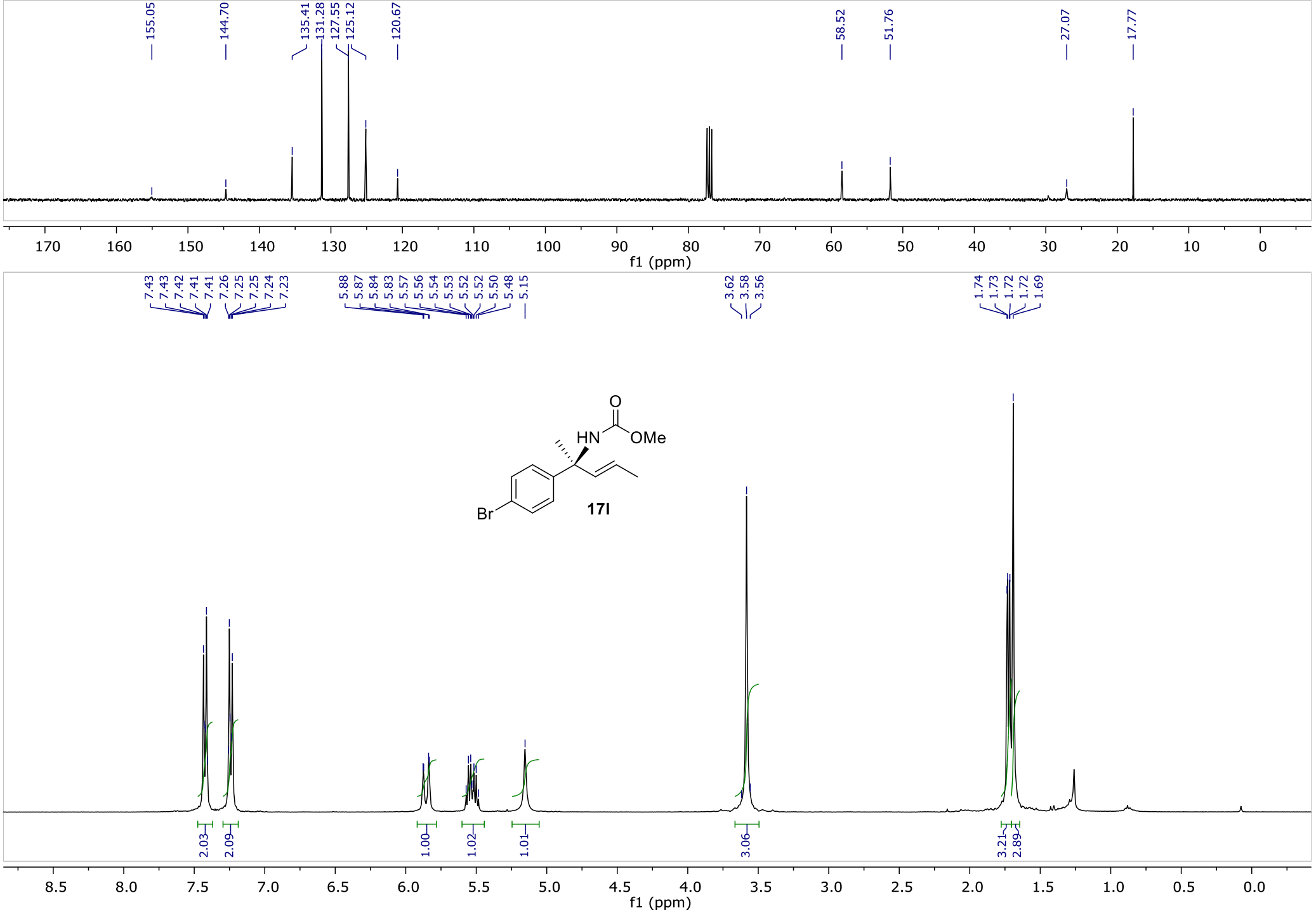


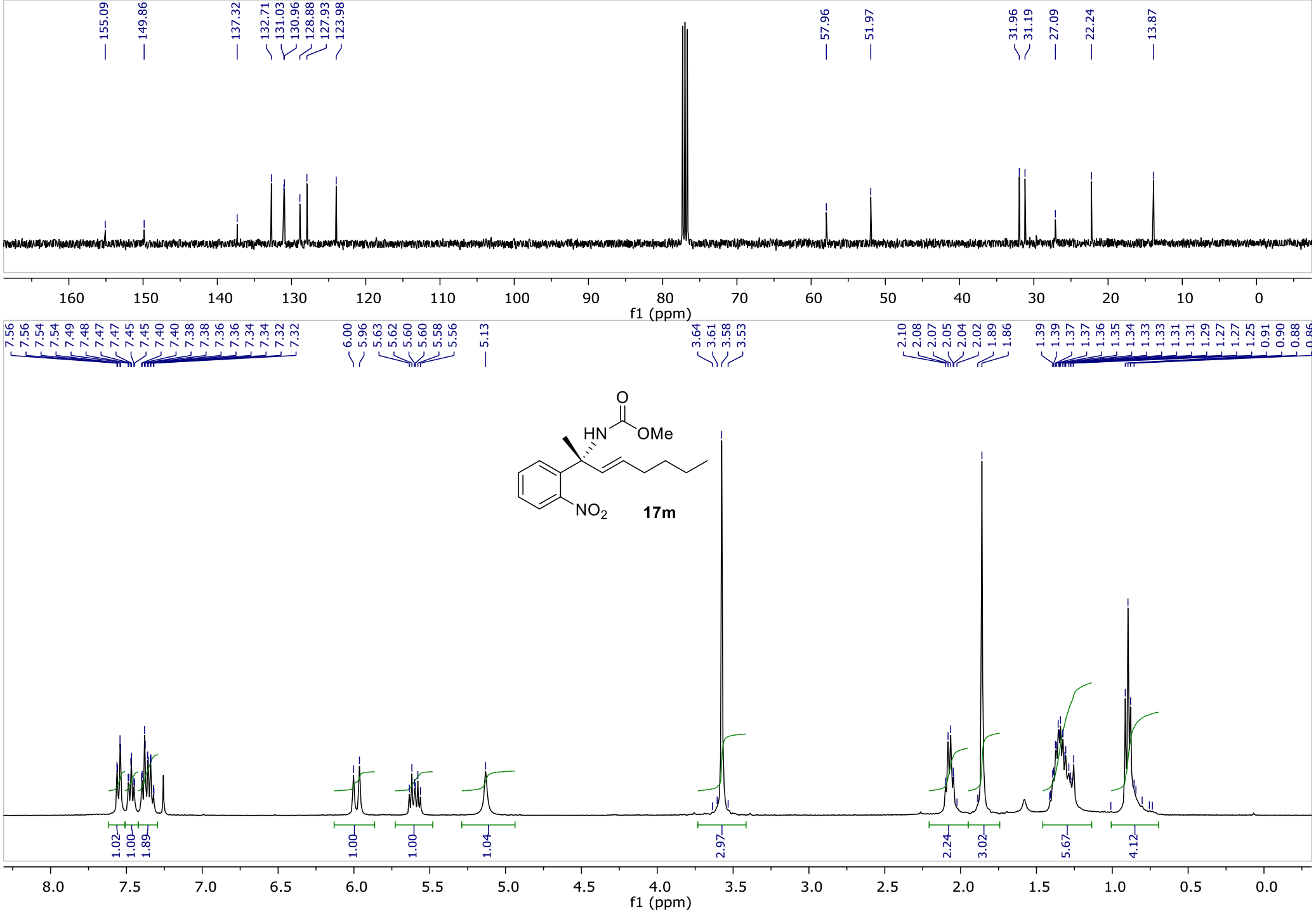
Column: Chiralcel OD-H
Eluent: 5% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 215 nm

	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	6.104	5.95	6.31	4.68586	28.0389	0.8392	0.162
2	7.989	7.69	9.07	553.678	1713.9	99.1608	0.306



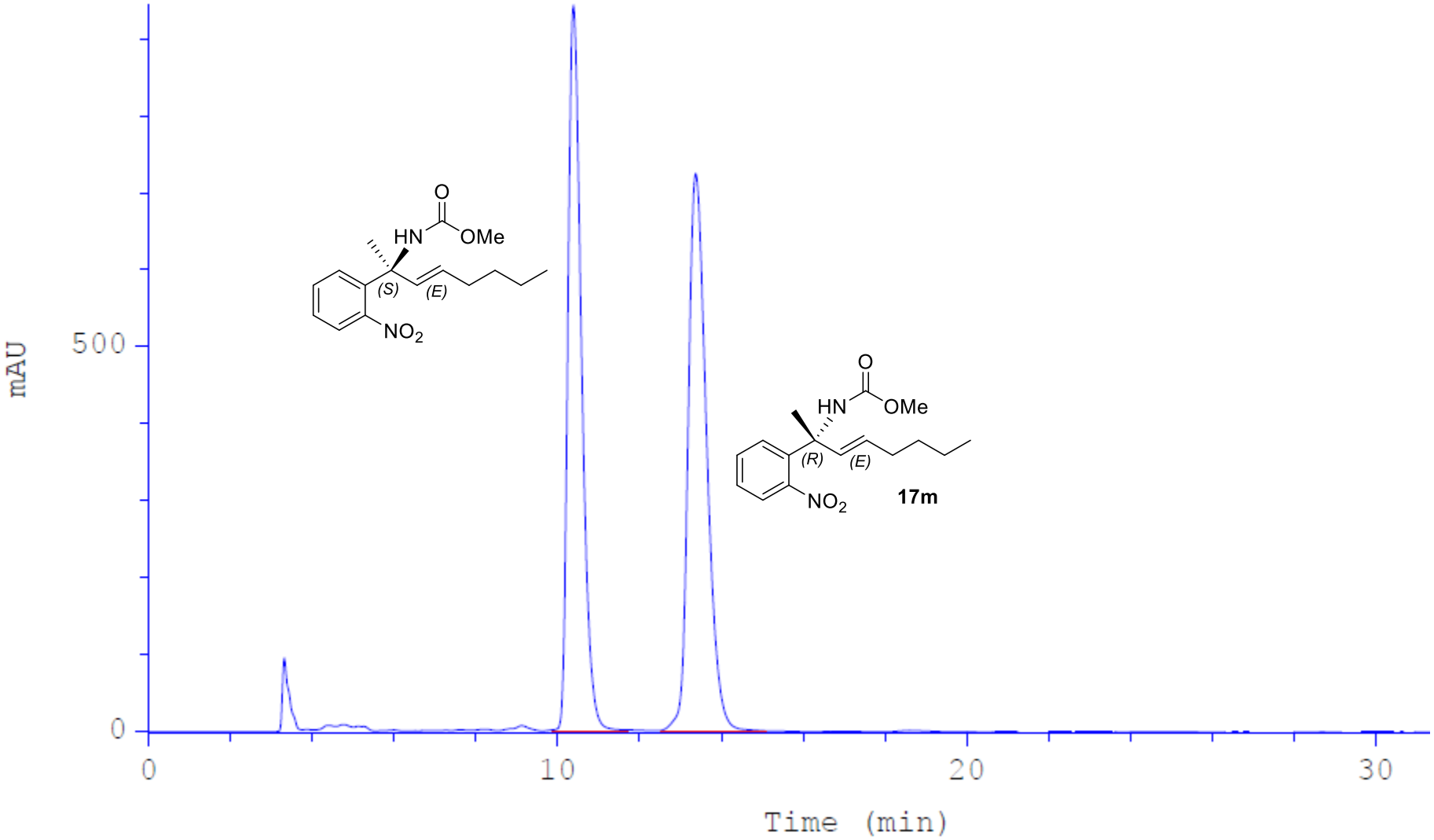

154.89
142.67
135.27
135.22
129.25
128.53
127.03
124.81
121.61
59.39
51.75
26.39
17.76
f1 (ppm)
7.57
7.57
7.55
7.53
7.52
7.30
7.29
7.28
7.26
7.26
7.11
7.10
7.09
7.08
7.07
7.06
5.98
5.97
5.94
5.93
5.52
5.50
5.49
5.48
5.47
5.46
5.45
5.43
3.60
3.56
1.88
1.77
1.75
1.74
1.73
1.73
HN
O
OMe
Br
17k
2.17
1.20
1.14
1.09
2.14
3.32
3.34
3.40
f1 (ppm)


155.05
144.70
135.41
131.28
127.55
125.12
120.67
58.52
51.76
27.07
17.77
f1 (ppm)
7.43
7.43
7.42
7.41
7.41
7.26
7.25
7.25
7.24
7.23
5.88
5.87
5.84
5.83
5.57
5.56
5.54
5.53
5.52
5.52
5.50
5.48
5.15
3.62
3.58
3.56
1.74
1.73
1.72
1.72
1.69
O
HN
OMe
Br
17l
2.03
2.09
1.00
1.02
1.01
3.06
3.21
2.89
f1 (ppm)


155.09
149.86
137.32
132.71
131.03
130.96
128.88
127.93
123.98
57.96
51.97
31.96
31.19
27.09
22.24
13.87
f1 (ppm)
7.56
7.56
7.54
7.54
7.49
7.48
7.47
7.47
7.45
7.45
7.40
7.40
7.38
7.38
7.36
7.36
7.34
7.34
7.32
7.32
6.00
5.96
5.63
5.62
5.60
5.60
5.58
5.56
5.13
3.64
3.61
3.58
3.53
2.10
2.08
2.07
2.05
2.04
2.02
1.89
1.86
1.39
1.39
1.37
1.37
1.36
1.35
1.34
1.33
1.33
1.31
1.31
1.29
1.27
1.27
1.25
0.91
0.90
0.88
HN
O
OMe
NO2
17m
1.02
1.00
1.89
1.00
1.00
1.04
2.97
2.24
3.02
5.67
4.12
f1 (ppm)

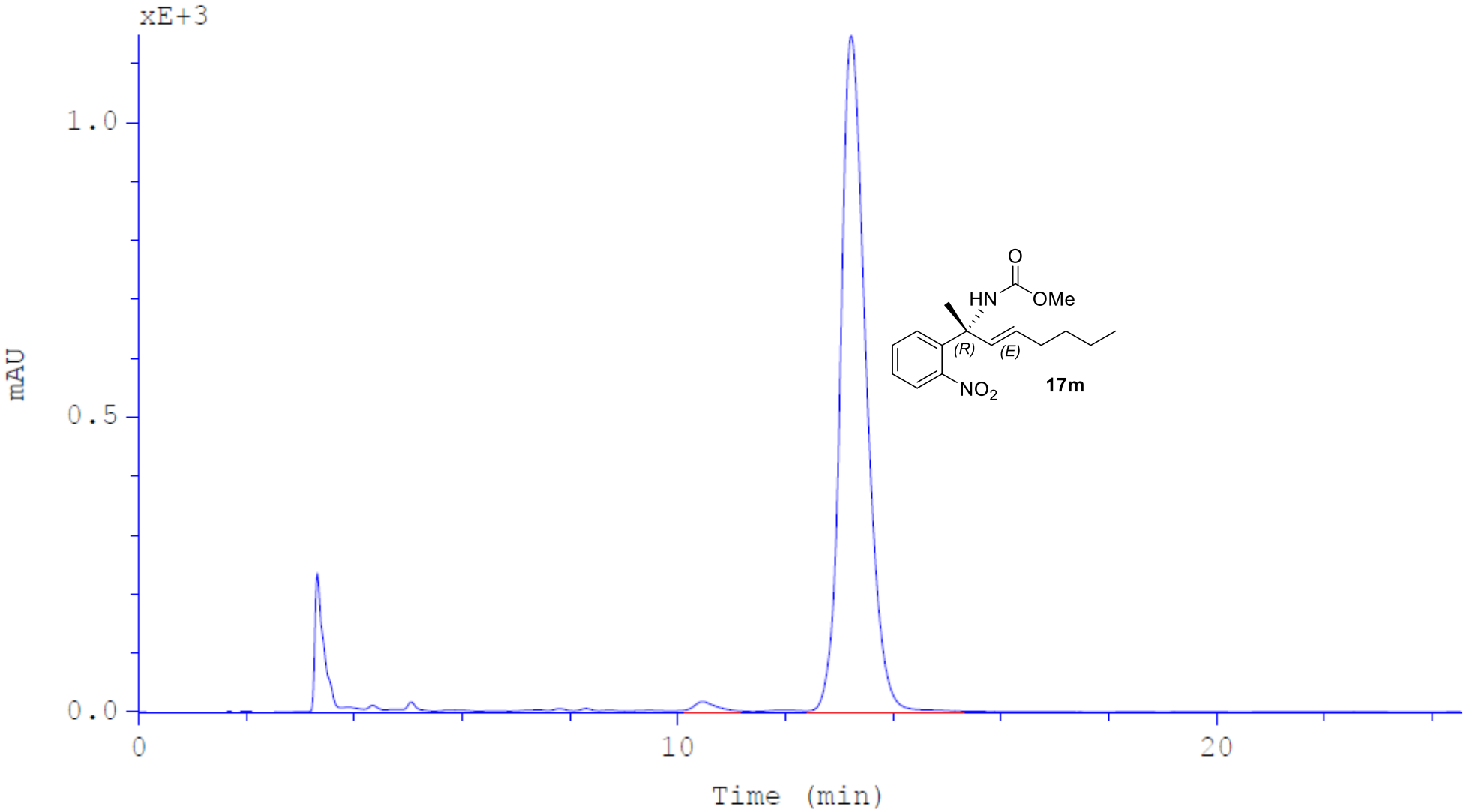
Column: Chiralcel OD-H
Eluent: 5% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 215 nm

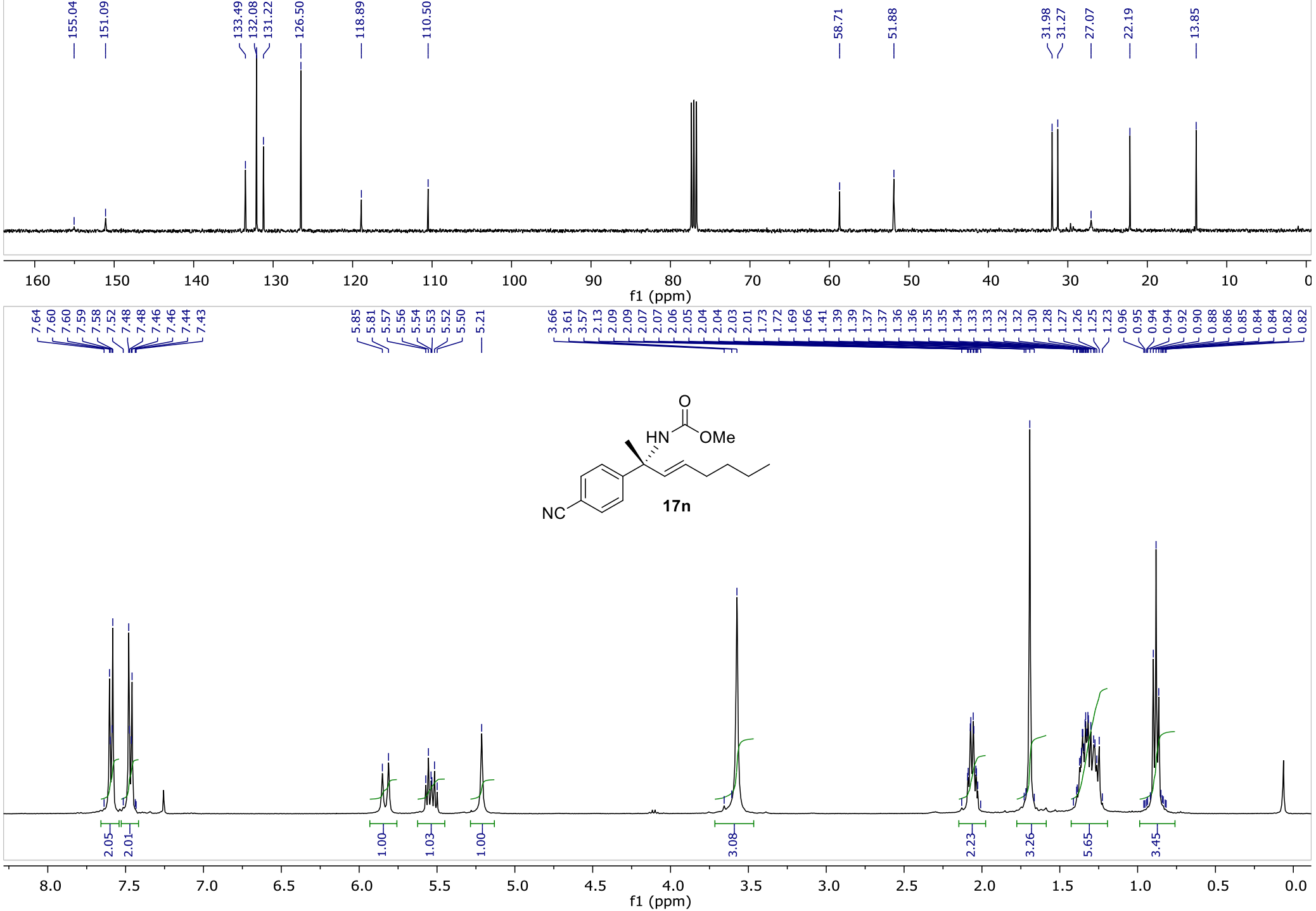
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	10.385	9.97	11.42	359.999	940.537	49.0925	0.354
2	13.369	12.59	14.54	373.307	722.36	50.9075	0.475



Column: Chiralcel OD-H
Eluent: 5% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 215 nm

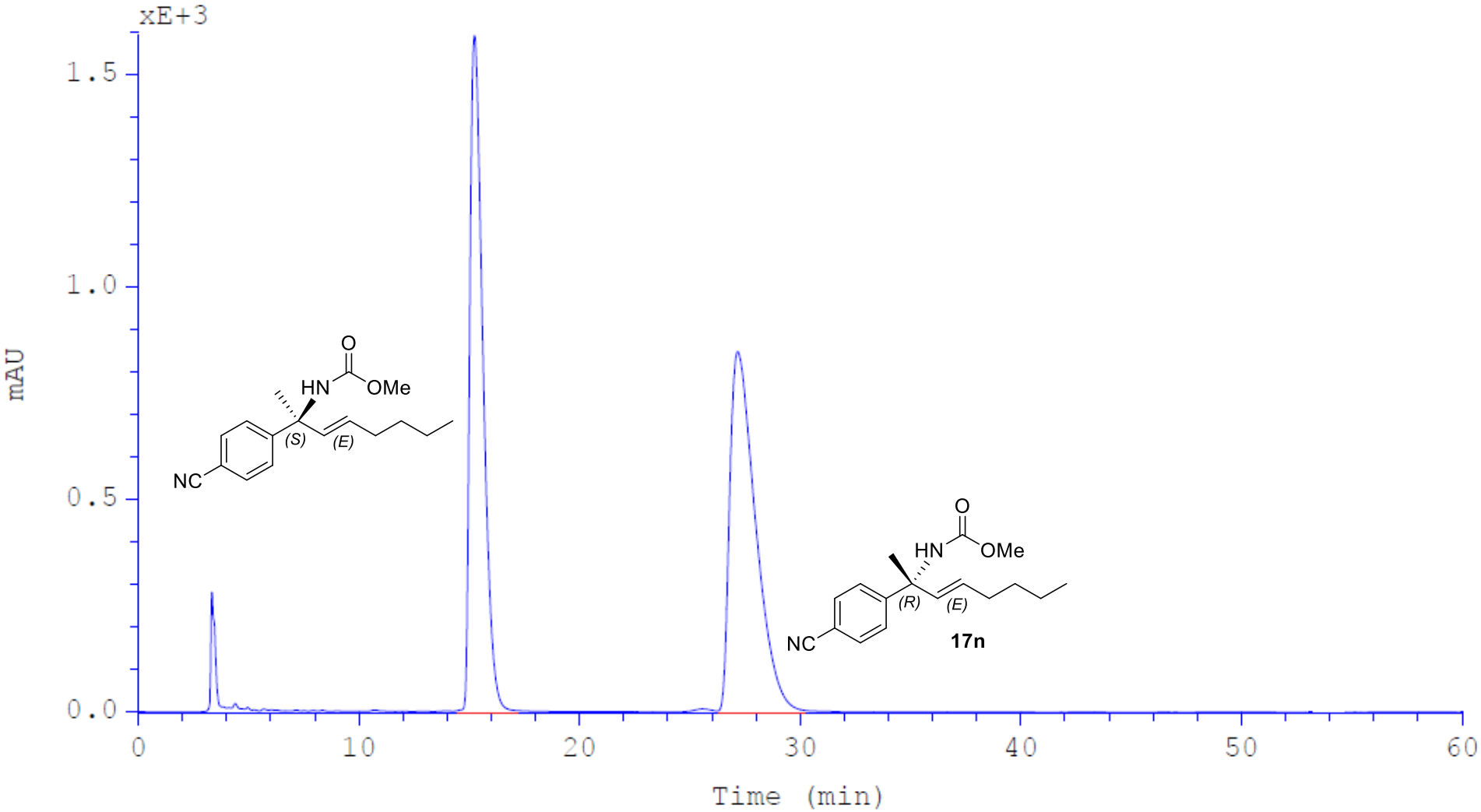
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	10.454	10.14	11.00	5.7683	14.4982	0.9207	0.371
2	13.222	12.45	14.82	620.728	1144.54	99.0793	0.491




155.04
151.09
133.49
132.08
131.22
126.50
118.89
110.50
58.71
51.88
31.98
31.27
27.07
22.19
13.85
f1 (ppm)
7.64 7.60 7.60 7.59 7.58 7.52 7.48 7.48 7.46 7.46 7.44 7.43
5.85 5.81 5.57 5.56 5.54 5.53 5.52 5.50 5.21
3.66 3.61 3.57 2.13 2.09 2.09 2.07 2.07 2.06 2.05 2.04 2.04 2.03 2.01 1.73 1.72 1.69 1.66 1.41 1.39 1.39 1.37 1.37 1.36 1.36 1.35 1.35 1.34 1.33 1.33 1.32 1.32 1.30 1.28 1.27 1.26 1.25 1.23 0.96 0.95 0.94 0.94 0.92 0.90 0.88 0.86 0.85 0.84 0.84 0.82 0.82
O
HN
OMe
NC
17n
2.05
2.01
1.00
1.03
1.00
3.08
2.23
3.26
5.65
3.45
f1 (ppm)

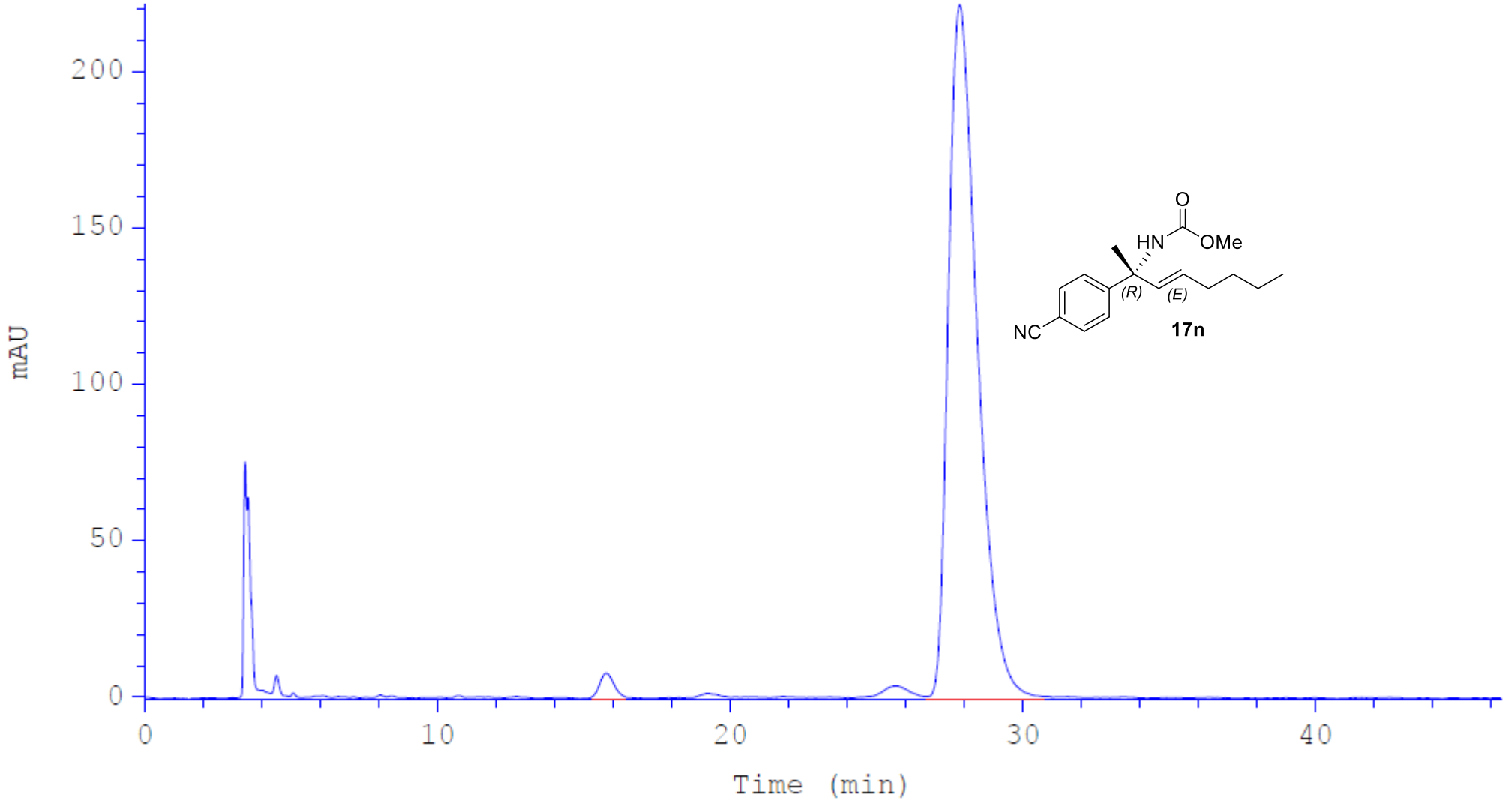
Column: Chiralcel OD-H
Eluent: 5% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 215 nm

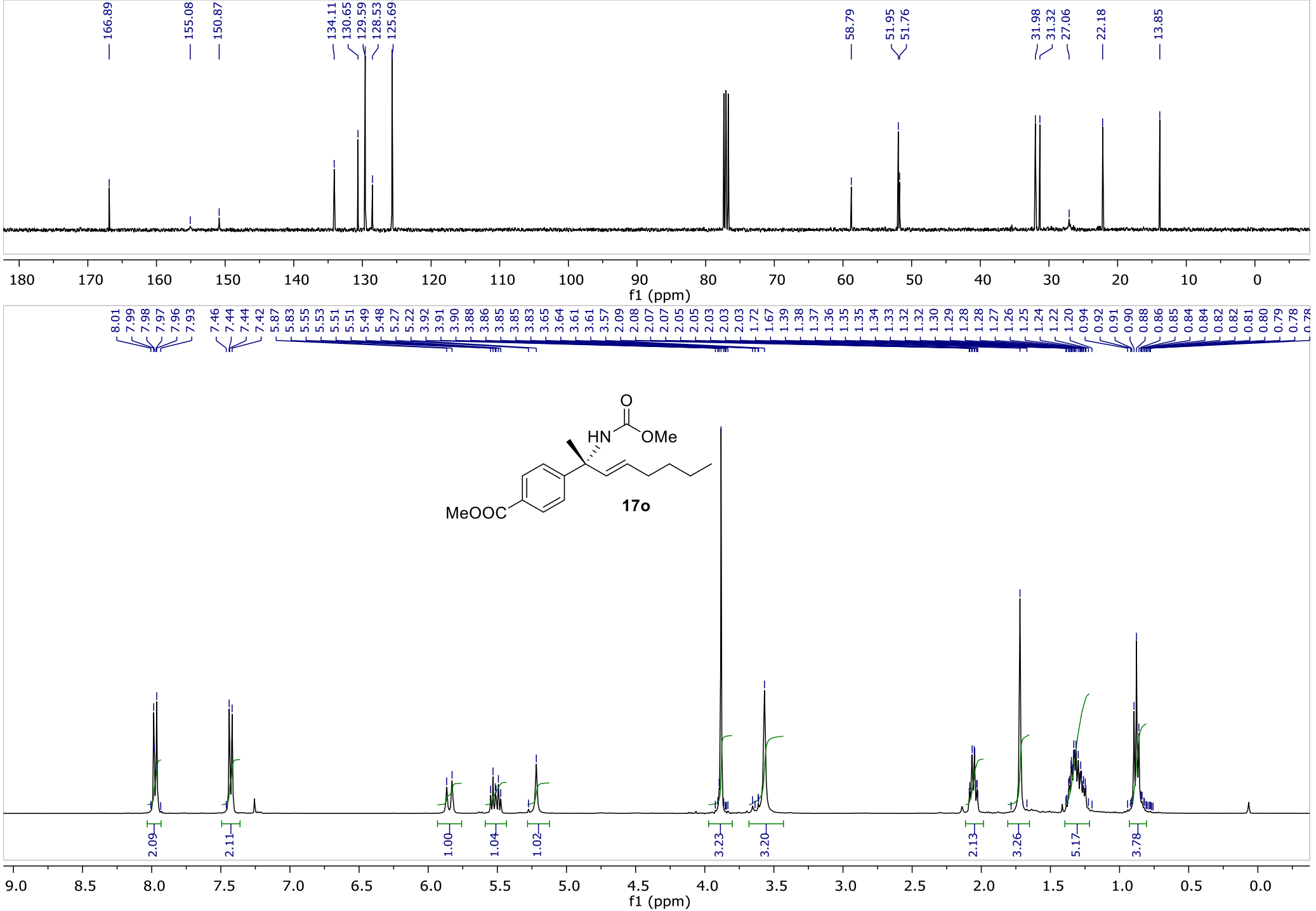
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	15.233	14.53	16.99	1082.47	1587.74	48.7383	0.638
2	27.167	26.18	30.14	1138.51	845.353	51.2617	1.256



Column: Chiralcel OD-H
Eluent: 5% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 215 nm

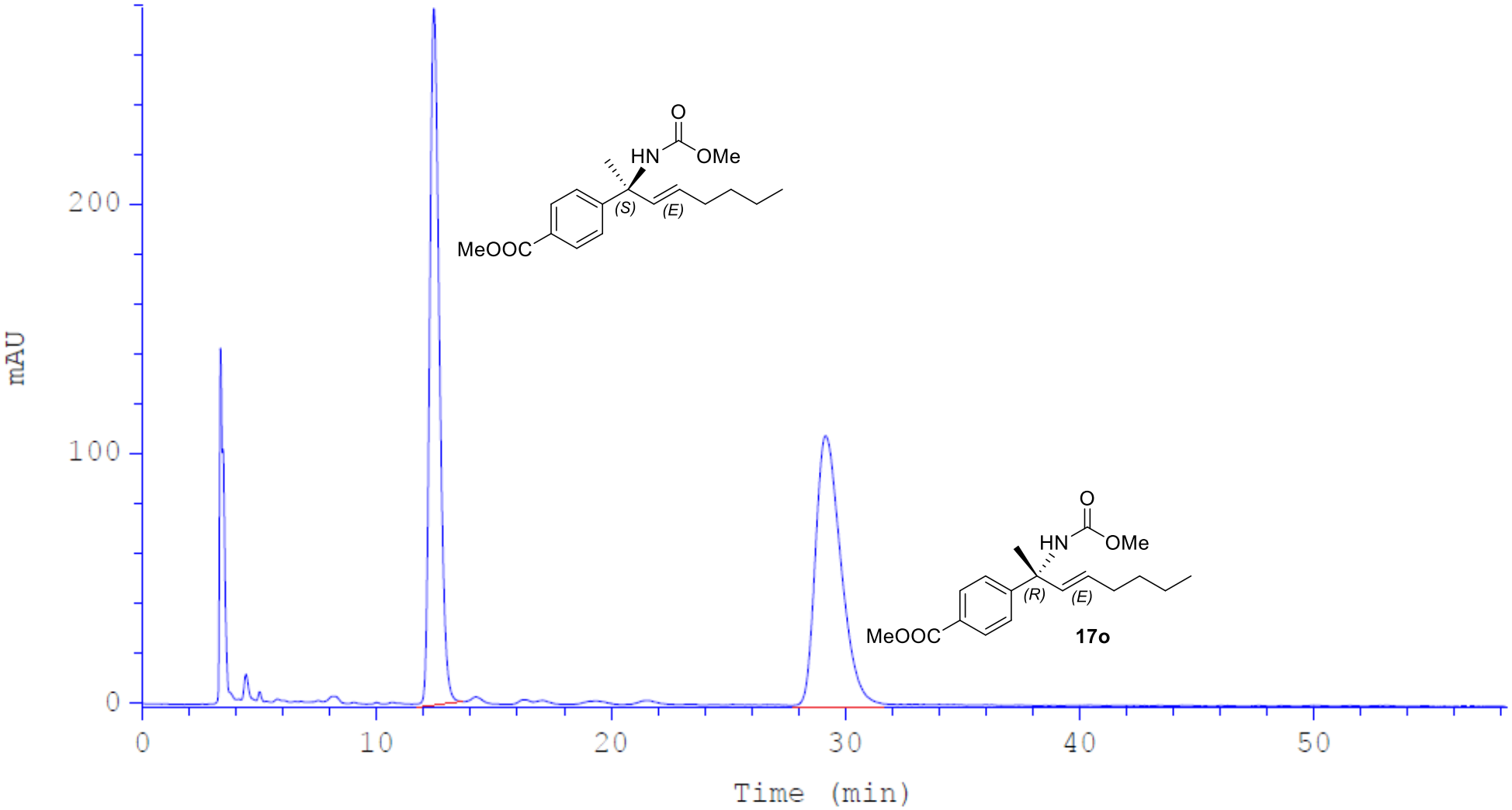
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	15.768	15.09	16.37	4.23921	7.70122	1.6528	0.524
2	27.836	26.74	30.53	252.241	221.07	98.3472	1.054




166.89
155.08
150.87
134.11
130.65
129.59
128.53
125.69
58.79
51.95
51.76
31.98
31.32
27.06
22.18
13.85
f1 (ppm)
HN
OMe
O
MeOOC
17o
2.09
2.11
1.00
1.04
1.02
3.23
3.20
2.13
3.26
5.17
3.78
f1 (ppm)

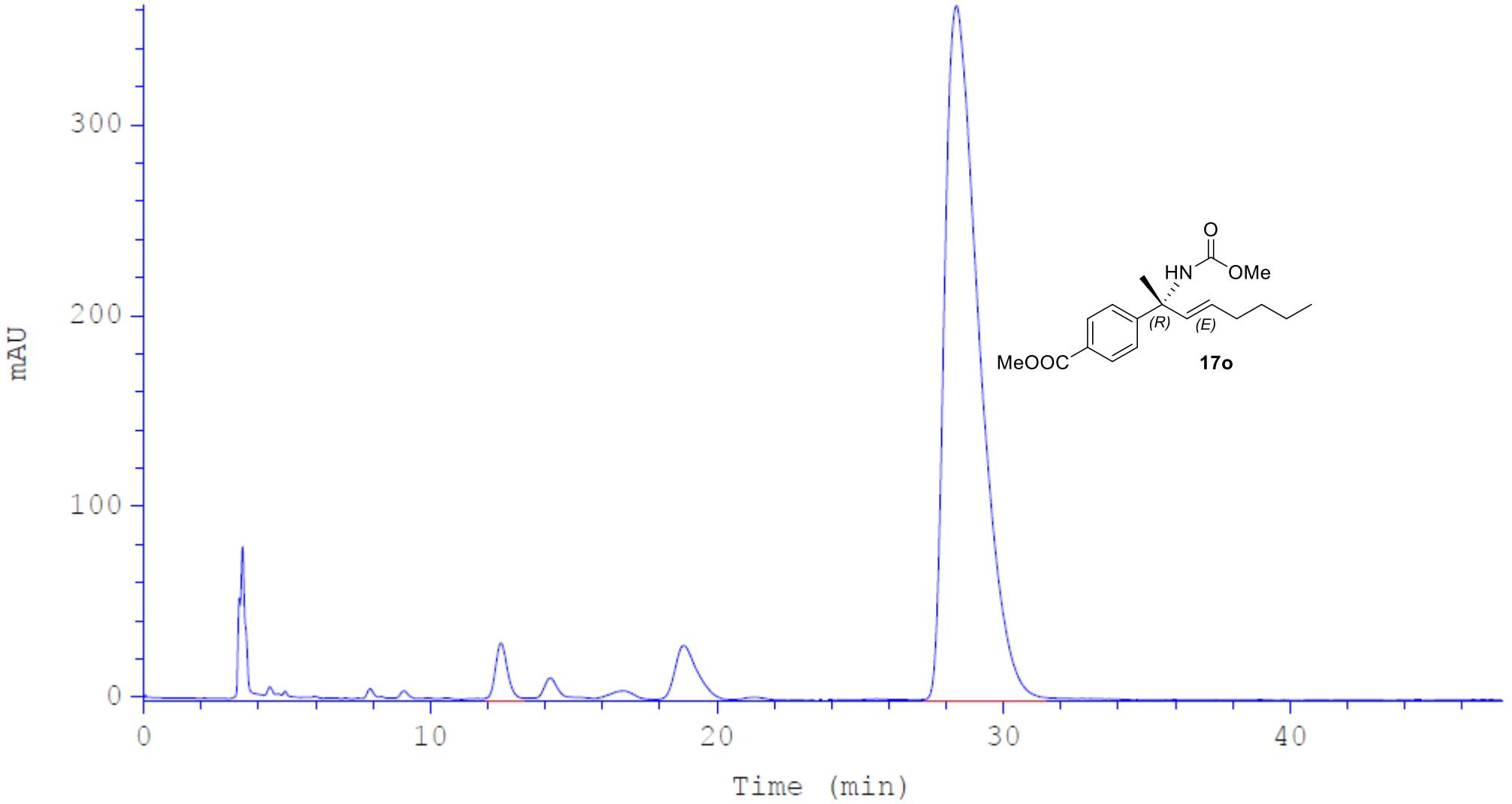
Column: Chiralcel OD-H
Eluent: 5% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 215 nm

	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	12.433	11.86	13.53	132.055	278.176	49.7354	0.440
2	29.150	27.74	31.42	133.46	107.694	50.2646	1.157

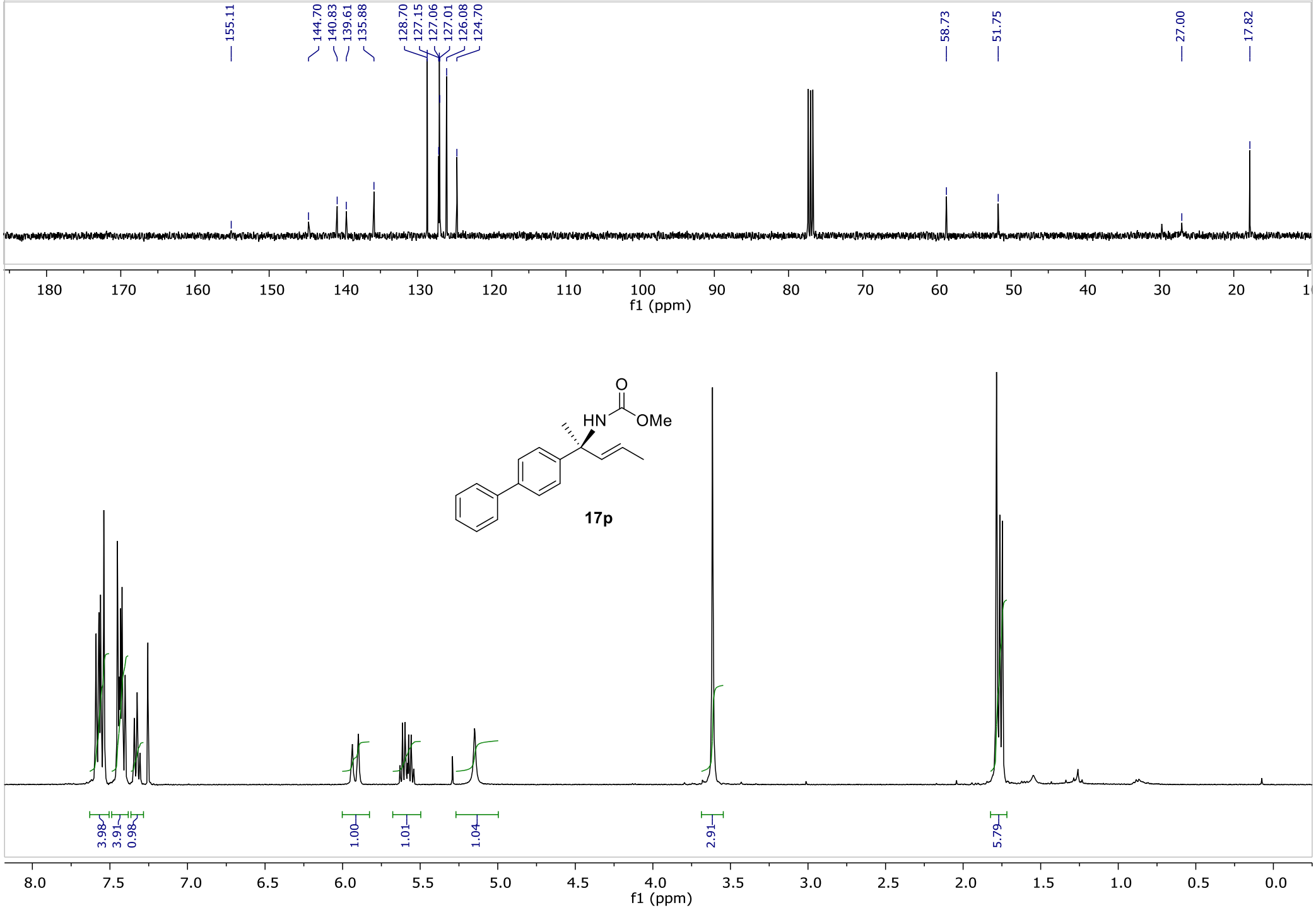


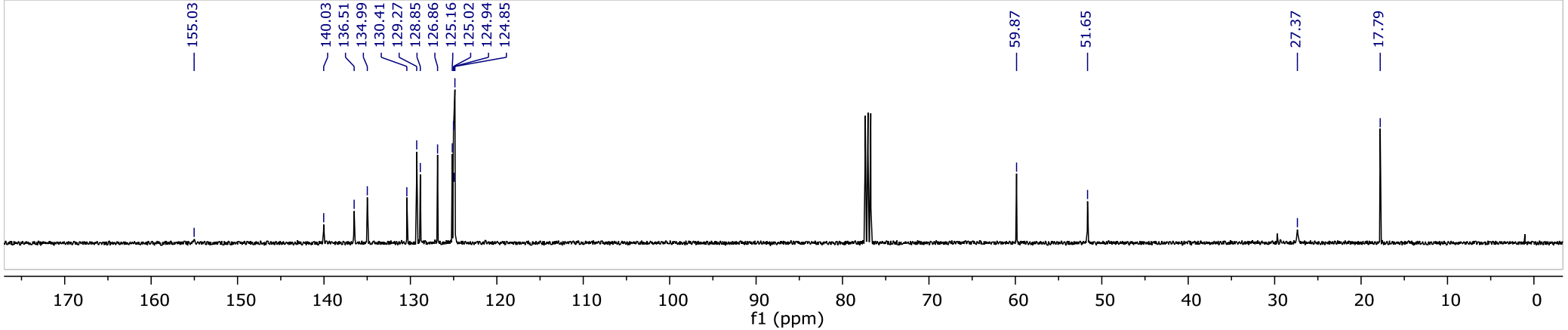
Column: Chiralcel OD-H
Eluent: 5% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 215 nm

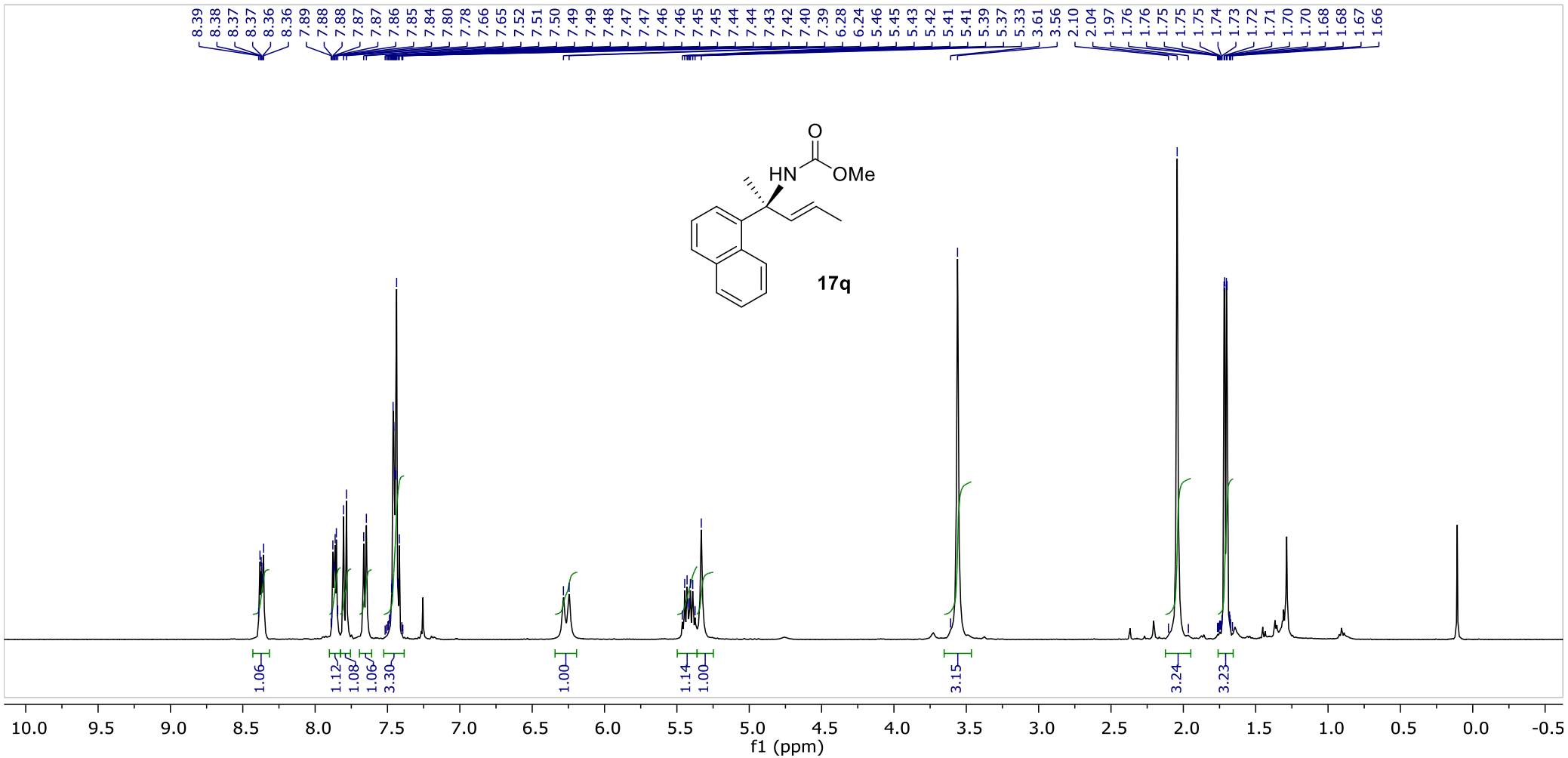
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	12.468	11.97	13.14	13.2338	28.6651	2.5545	0.433
2	28.352	27.24	31.59	504.819	363.456	97.4455	1.288

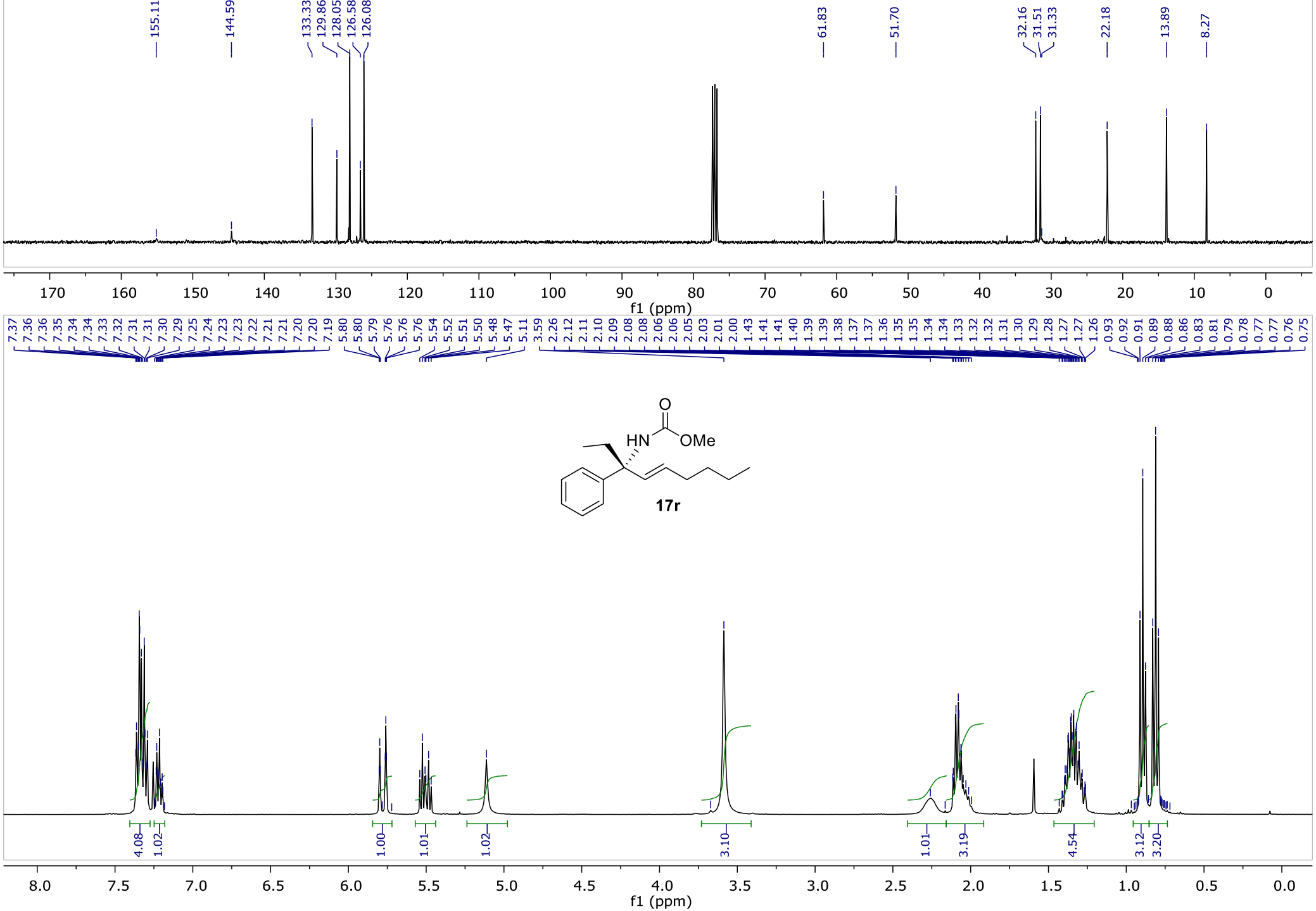


17o


155.11
144.70
140.83
139.61
135.88
128.70
127.15
127.06
127.01
126.08
124.70
58.73
51.75
27.00
17.82
f1 (ppm)
O
HN
OMe
17p
3.98
3.91
0.98
1.00
1.01
1.04
2.91
5.79
f1 (ppm)

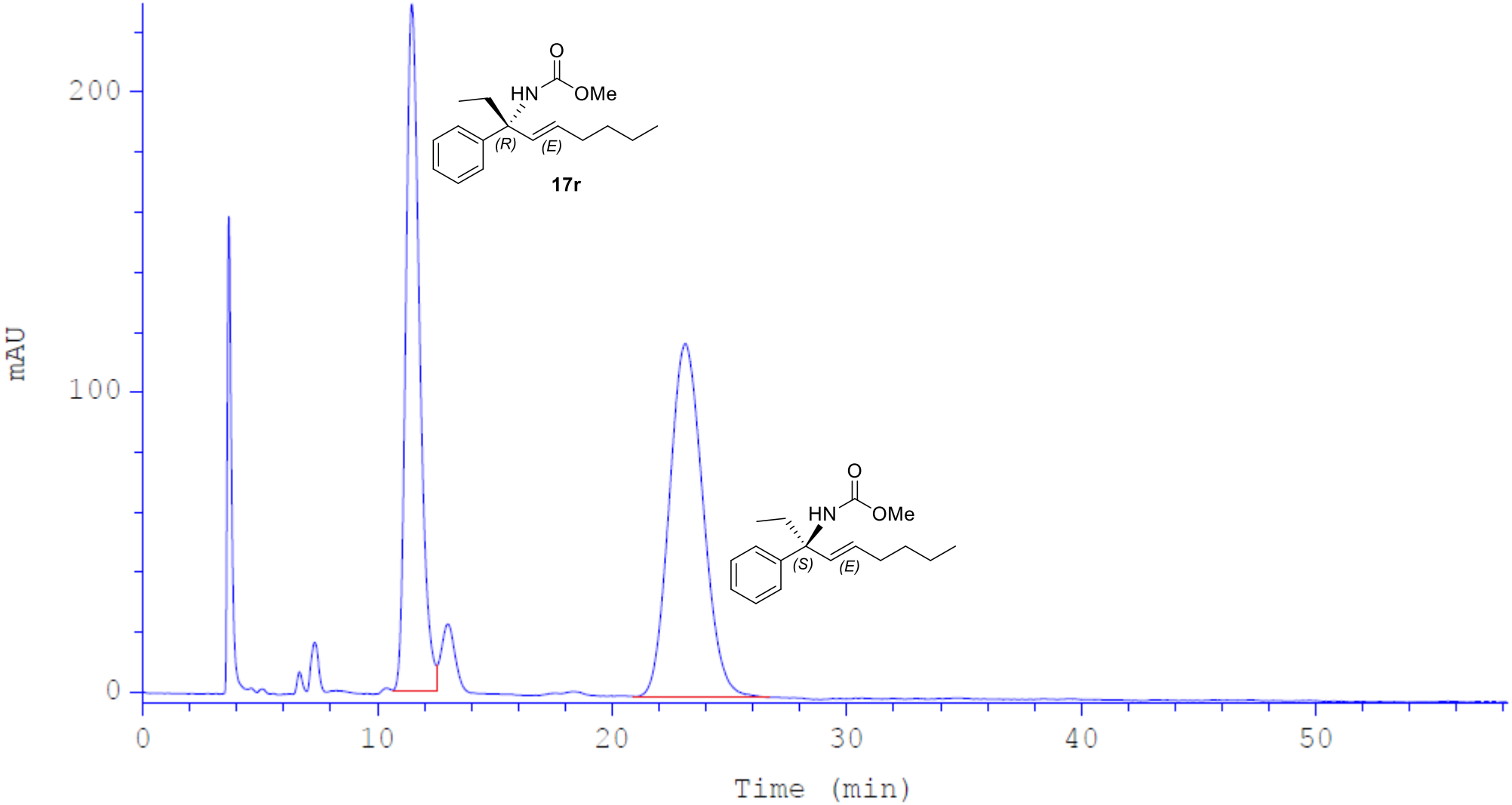

155.03
140.03
136.51
134.99
130.41
129.27
128.85
126.86
125.16
125.02
124.94
124.85
59.87
51.65
27.37
17.79
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.39 8.38 8.37 8.37 8.36 8.36 7.89 7.88 7.88 7.87 7.87 7.86 7.85 7.84 7.80 7.78 7.66 7.65 7.52 7.51 7.50 7.49 7.49 7.48 7.47 7.47 7.46 7.46 7.45 7.45 7.44 7.44 7.43 7.42 7.40 7.39 6.28 6.24 5.46 5.45 5.43 5.42 5.41 5.41 5.39 5.37 5.33 3.61 3.56 2.10 2.04 1.97 1.76 1.76 1.75 1.75 1.75 1.74 1.73 1.72 1.71 1.70 1.70 1.68 1.68 1.67 1.66
HN
O
OMe
17q
1.06
1.12
1.08
1.06
3.30
1.00
1.14
1.00
3.15
3.24
3.23
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)


155.11
144.59
133.33
129.86
128.05
126.58
126.08
61.83
51.70
32.16
31.51
31.33
22.18
13.89
8.27
f1 (ppm)
O
HN
OMe
17r
4.08
1.02
1.00
1.01
1.02
3.10
1.01
3.19
4.54
3.12
3.20
f1 (ppm)

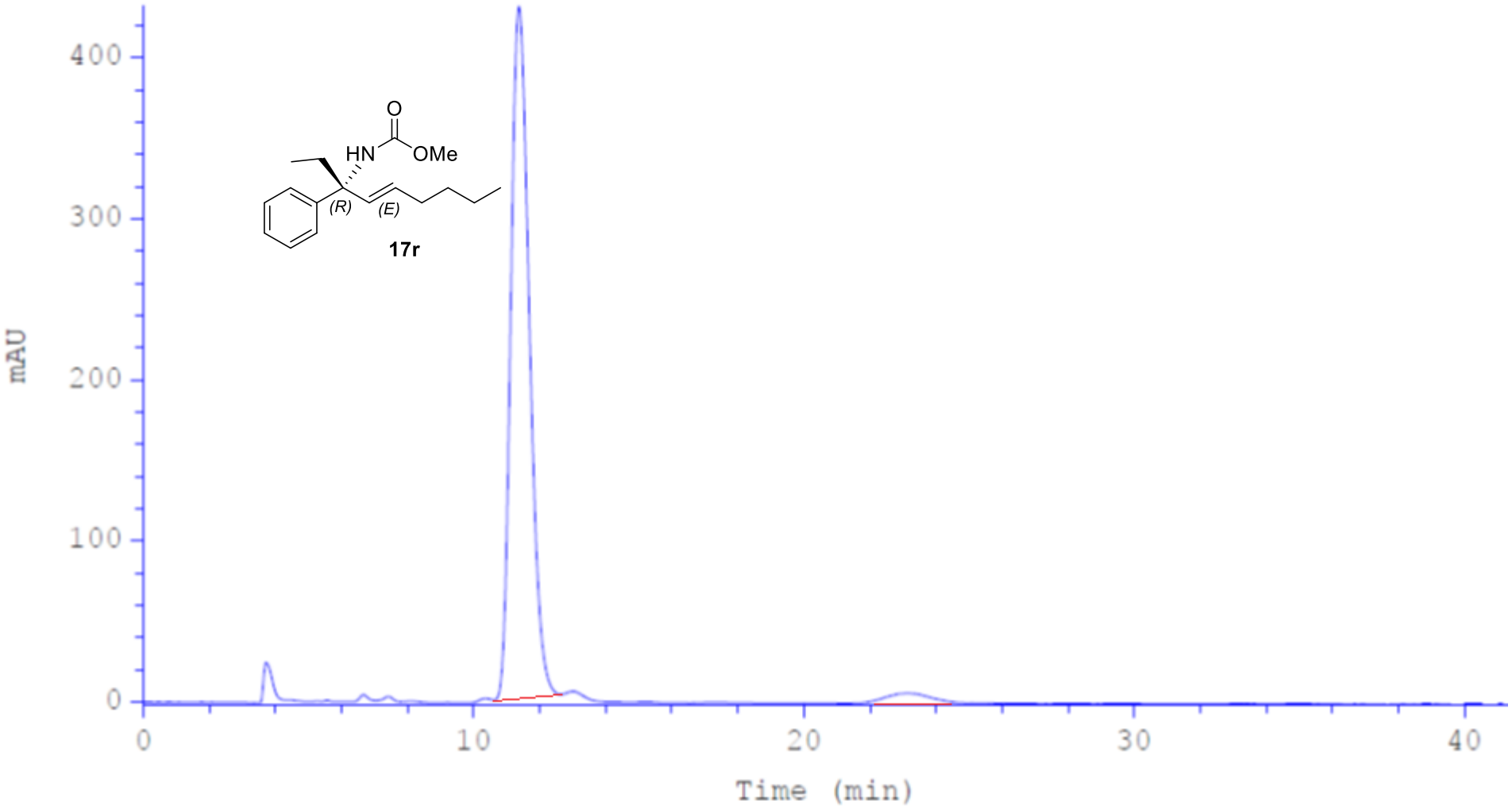
Column: Chiralcel OD-H
Eluent: 5% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 215 nm

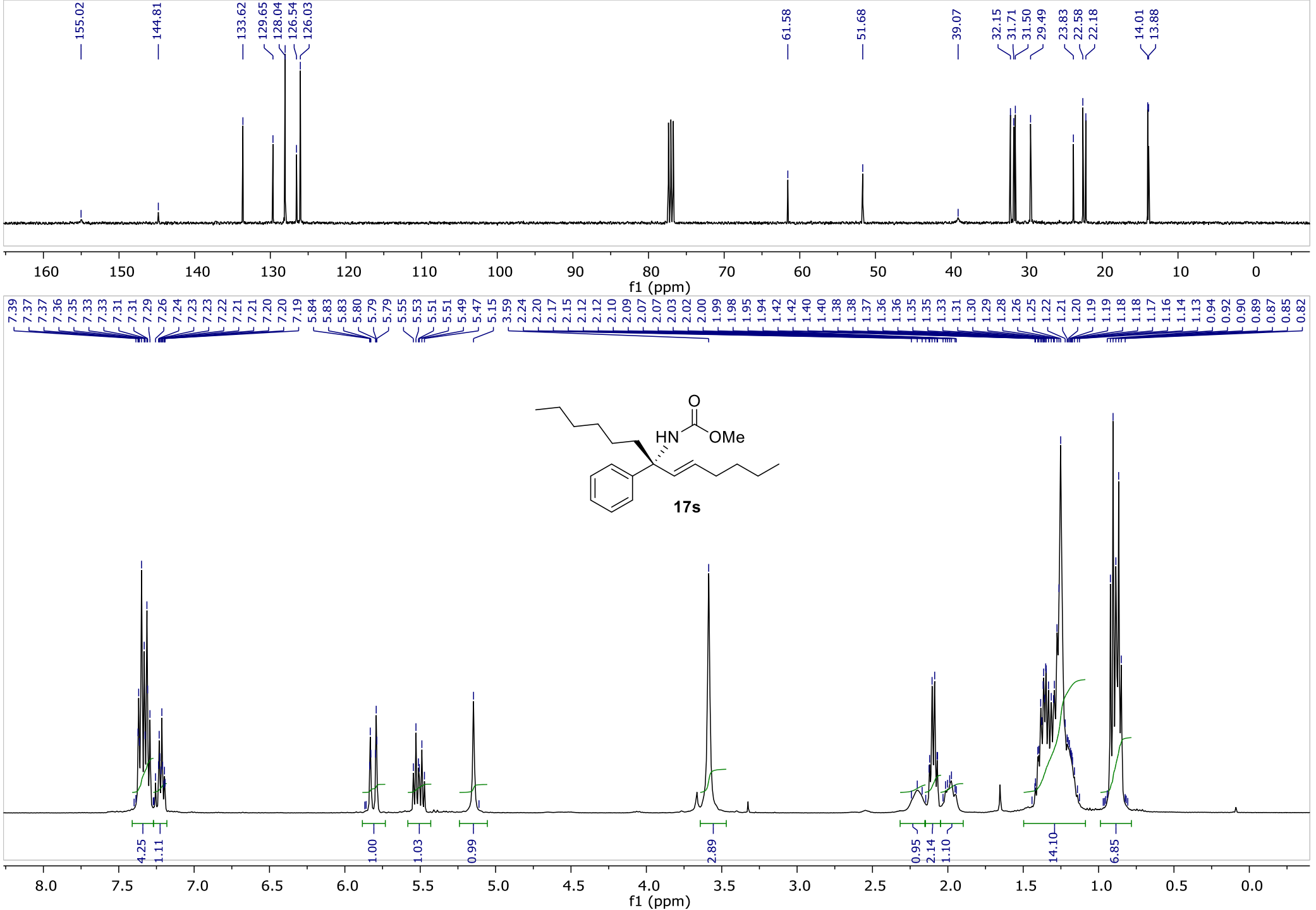
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	11.467	10.15	12.57	146.813	224.144	42.8863	0.615
2	23.117	21.14	26.45	195.518	117.534	57.1137	1.560



Column: Chiralcel OD-H
Eluent: 5% *i*-PrOH in hexanes
Flow: 1mL/min
Detection: UV, 215 nm

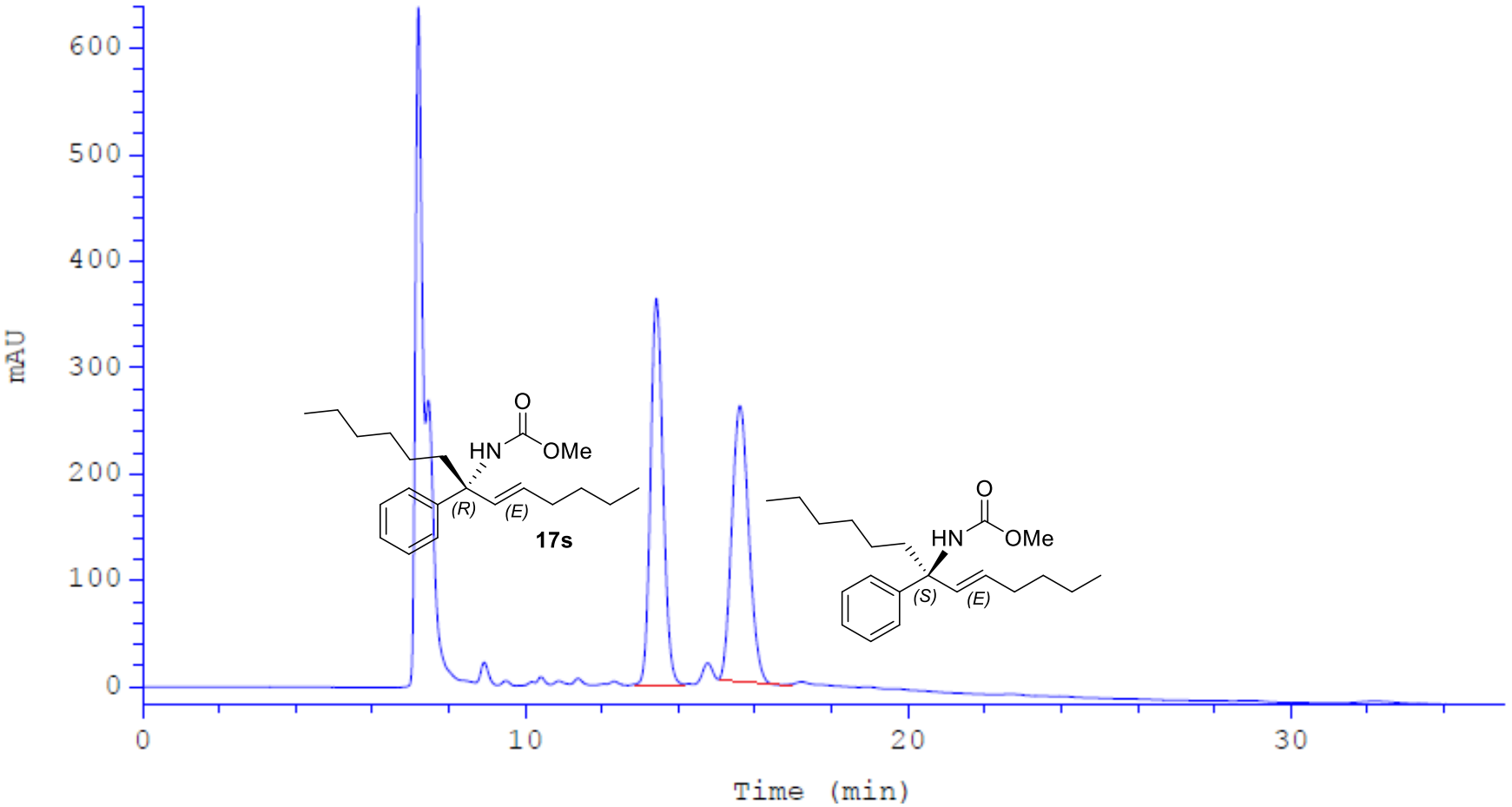
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	11.368	10.57	12.79	278.879	429.014	97.6656	0.610
2	23.153	22.04	24.36	6.66588	5.05244	2.3344	1.338




155.02
144.81
133.62
129.65
128.04
126.54
126.03
61.58
51.68
39.07
32.15
31.71
31.50
29.49
23.83
22.58
22.18
14.01
13.88
f1 (ppm)
HN
O
OMe
17s
4.25
1.11
1.00
1.03
0.99
2.89
0.95
2.14
1.10
14.10
6.85
f1 (ppm)

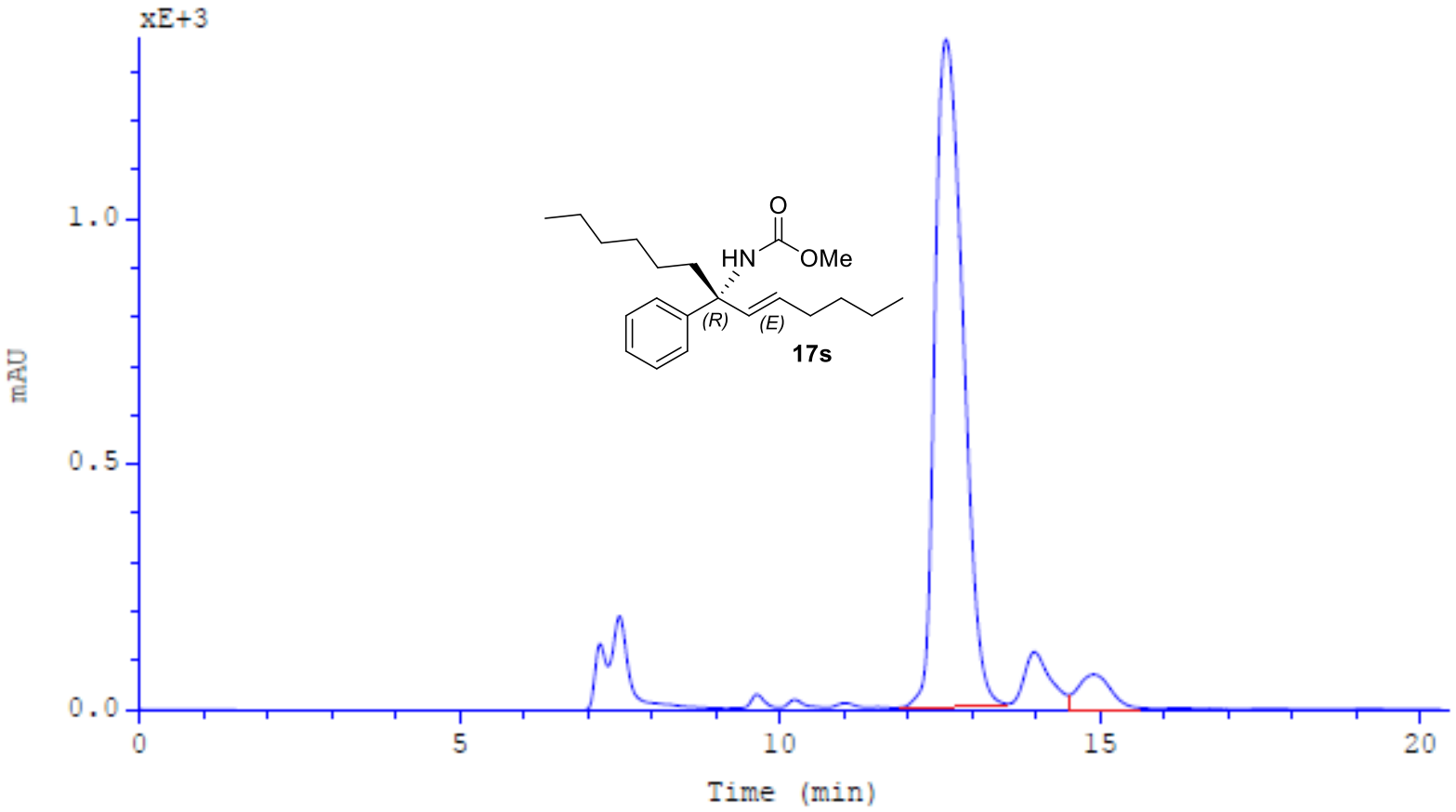
Column: Chiralpak AS-H
Eluent: 1% *i*-PrOH in hexanes
Flow: 0.5mL/min
Detection: UV, 215 nm

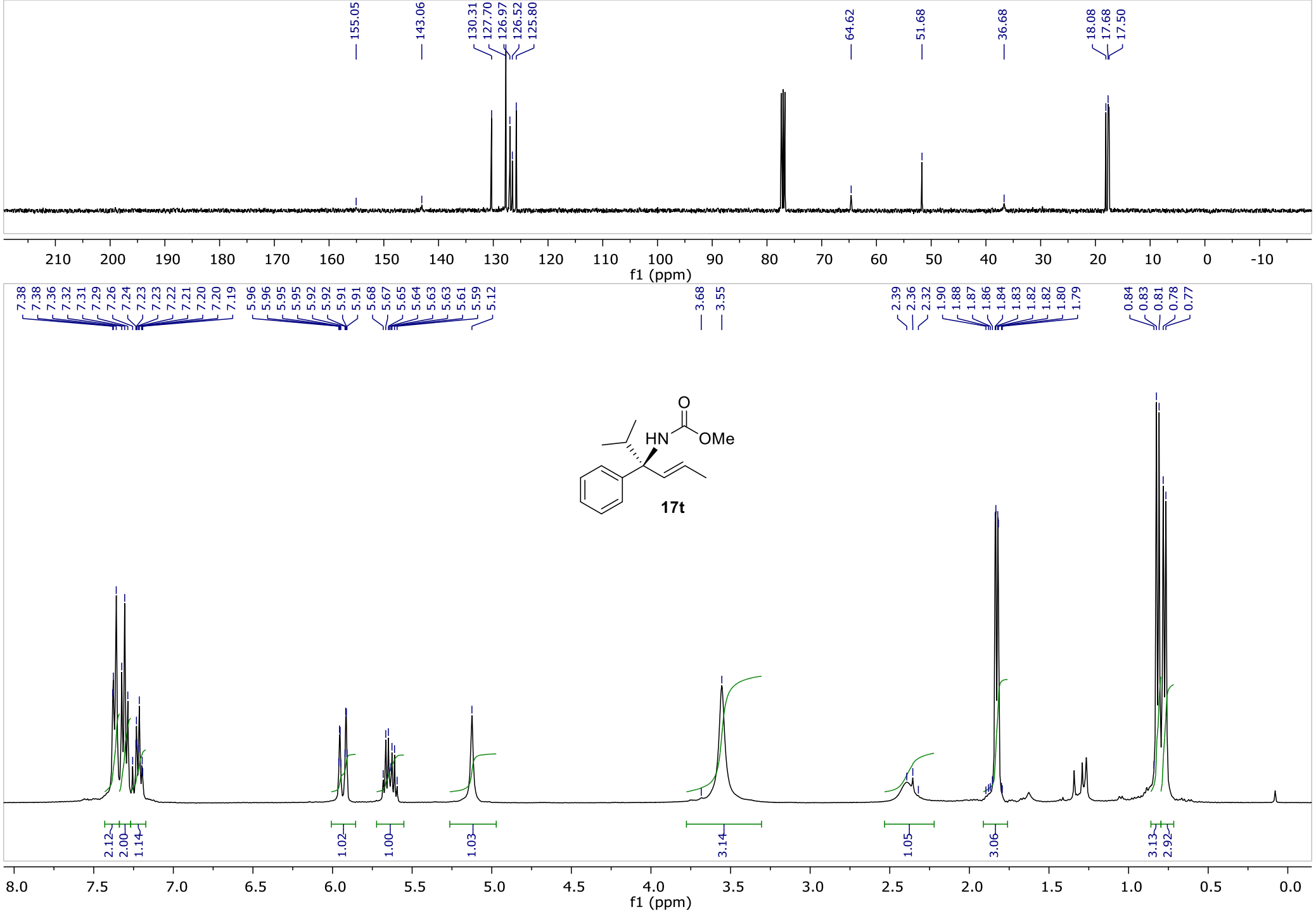
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	13.419	12.86	14.13	136.465	362.278	50.5122	0.351
2	15.603	15.03	16.44	133.697	258.986	49.4878	0.487

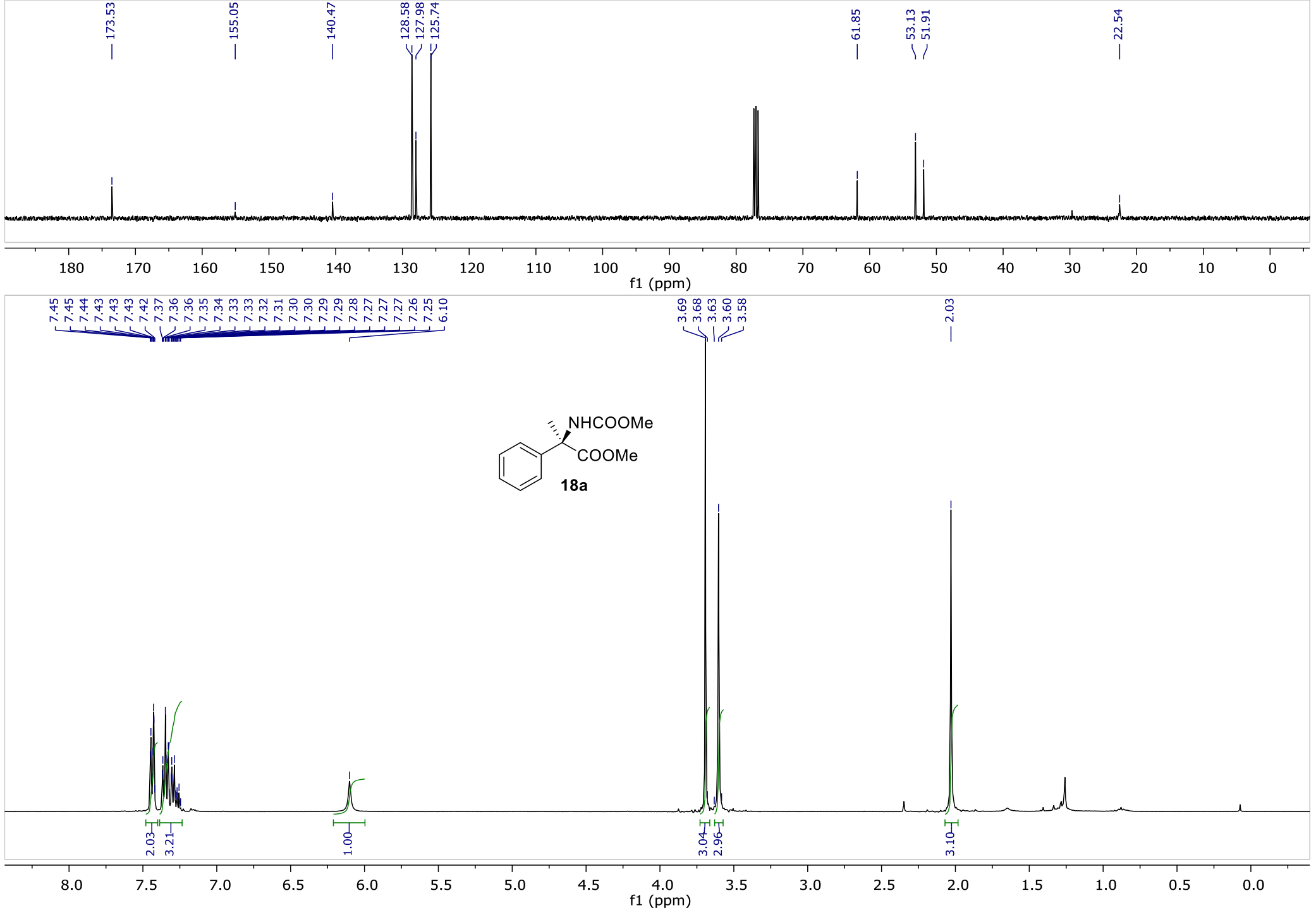


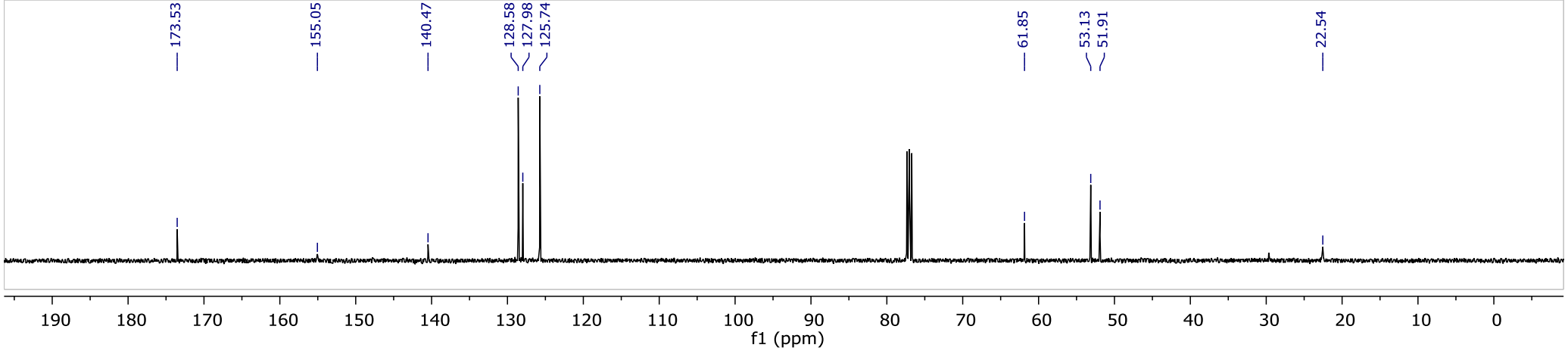
Column: Chiralpak AS-H
Eluent: 1% *i*-PrOH in hexanes
Flow: 0.5mL/min
Detection: UV, 215 nm

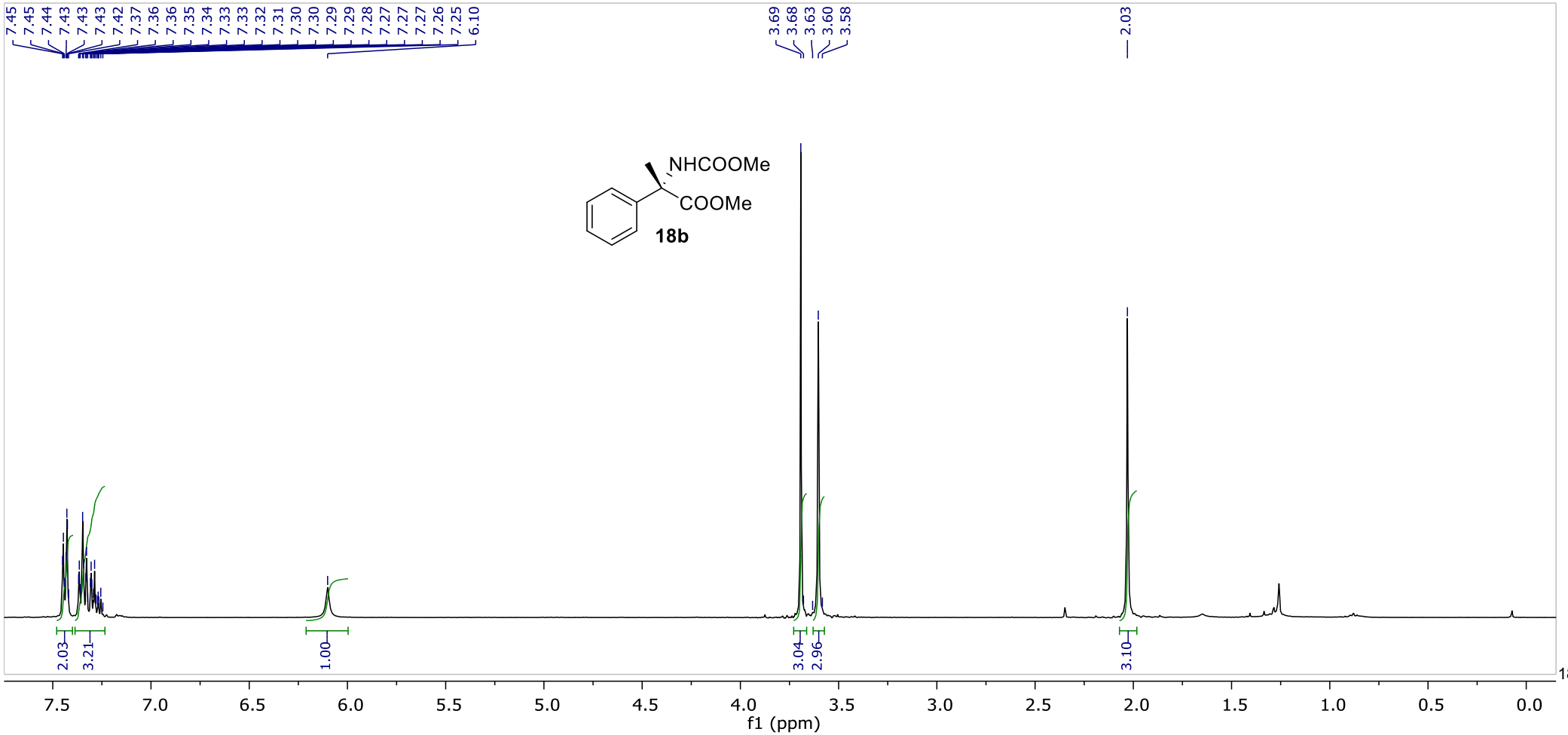
	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	12.590	11.86	13.48	687.027	1357.58	96.4789	0.488
2	14.908	14.51	15.63	25.074	51.1382	3.5211	0.503

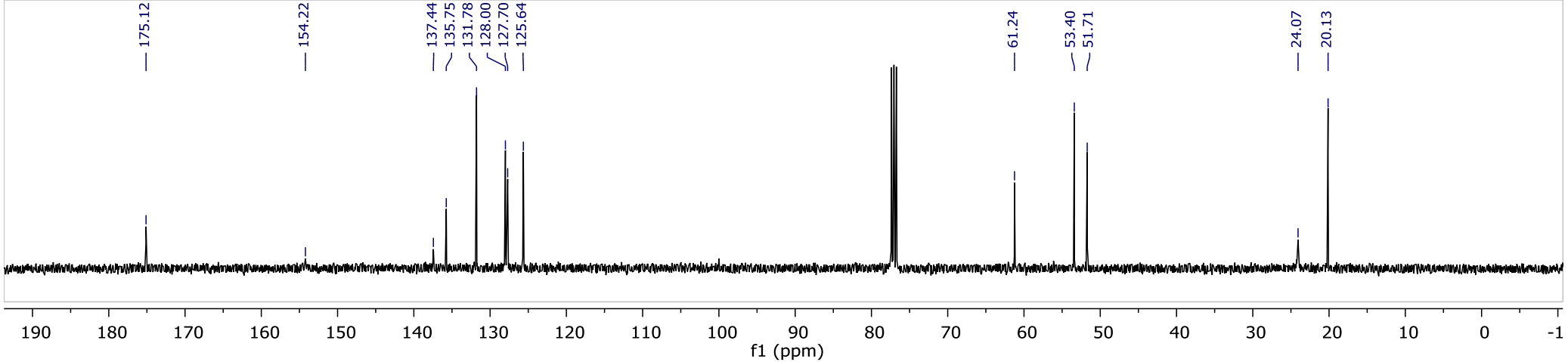


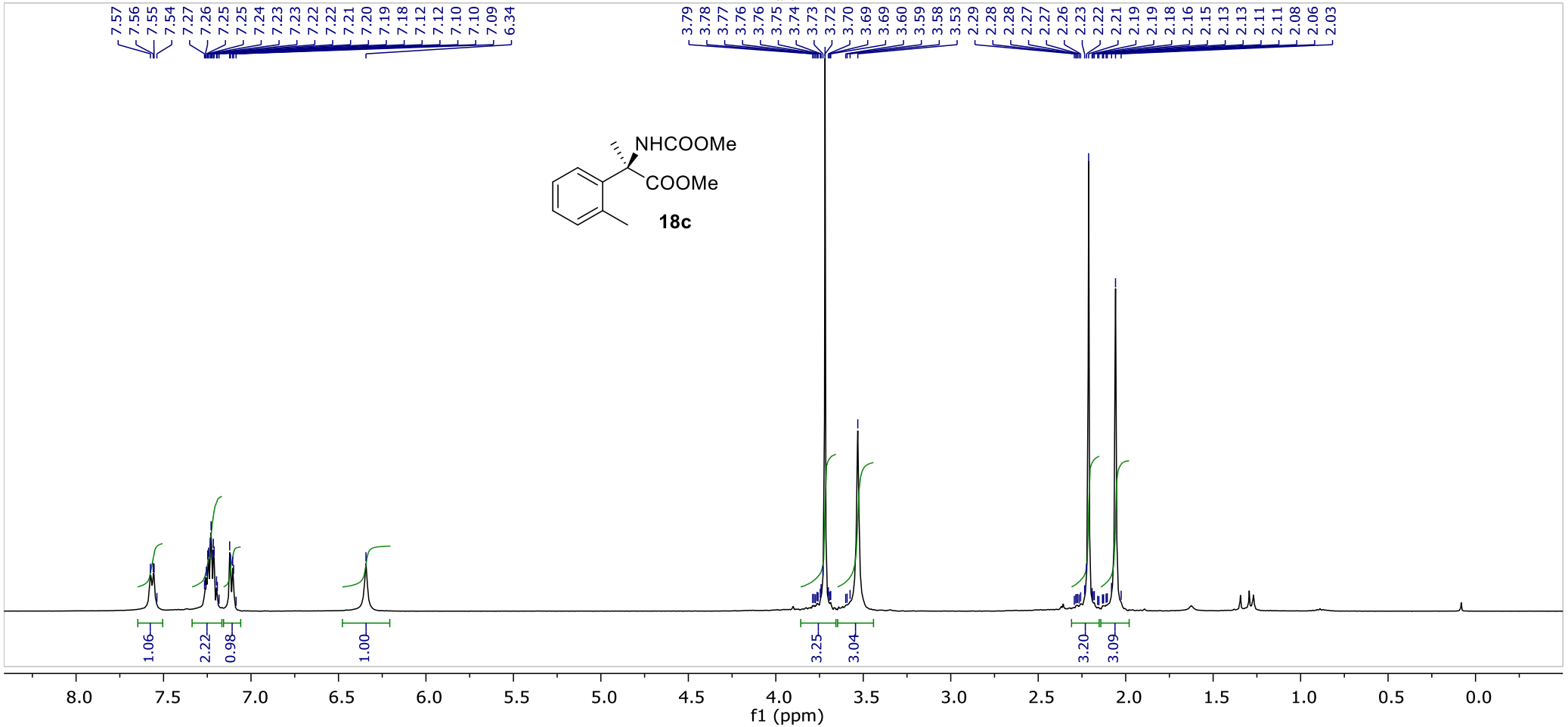

155.05
143.06
130.31
127.70
126.97
126.52
125.80
64.62
51.68
36.68
18.08
17.68
17.50
f1 (ppm)
7.38
7.38
7.36
7.32
7.31
7.29
7.26
7.24
7.23
7.23
7.22
7.21
7.20
7.20
7.19
5.96
5.96
5.95
5.95
5.92
5.92
5.91
5.91
5.68
5.67
5.65
5.64
5.63
5.63
5.61
5.59
5.12
3.68
3.55
2.39
2.36
2.32
1.90
1.88
1.87
1.86
1.84
1.83
1.82
1.82
1.80
1.79
0.84
0.83
0.81
0.78
0.77
O
HN
OMe
17t
2.12
2.00
1.14
1.02
1.00
1.03
3.14
1.05
3.06
3.13
2.92
f1 (ppm)

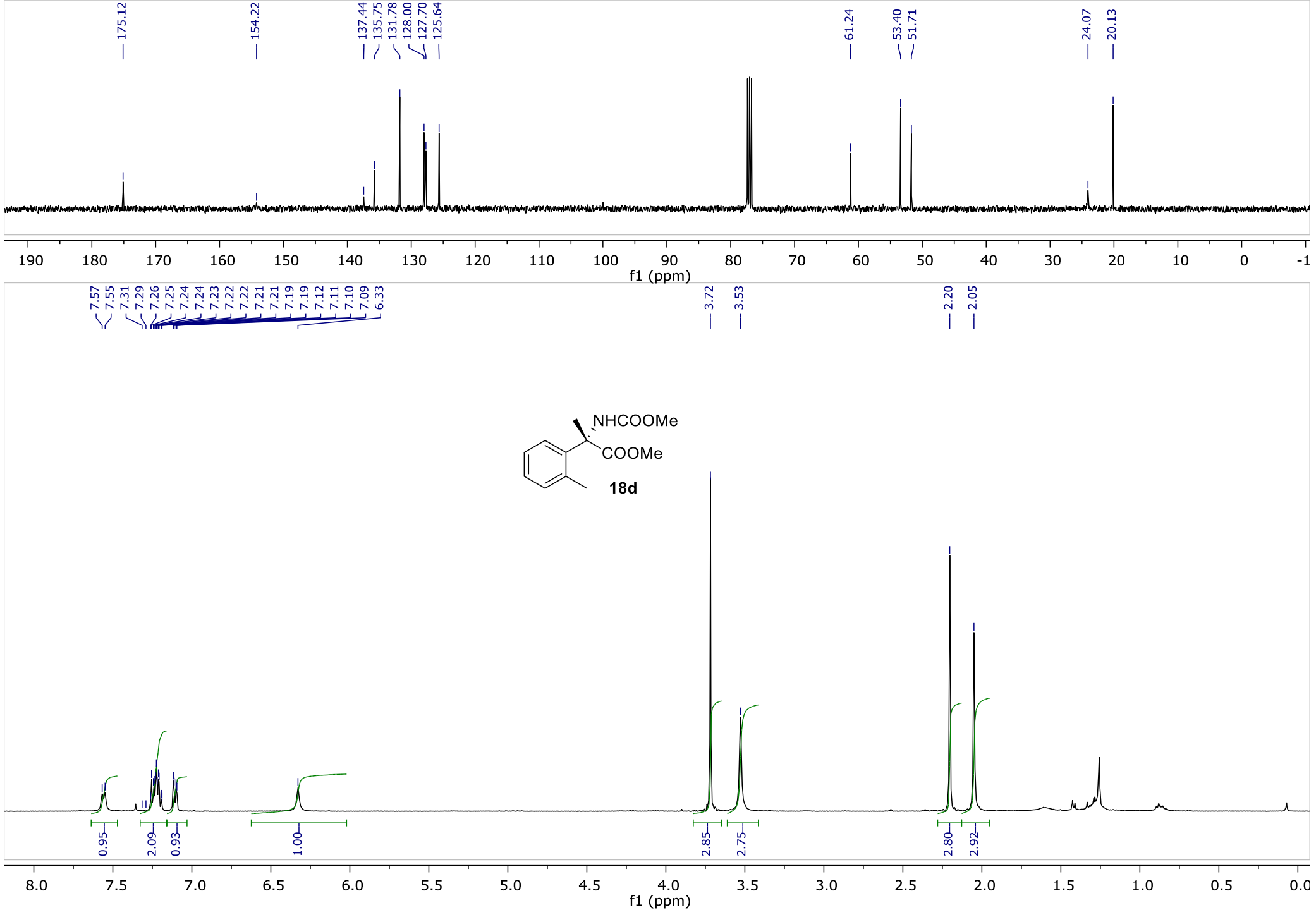

173.53
155.05
140.47
128.58
127.98
125.74
61.85
53.13
51.91
22.54
f1 (ppm)
7.45
7.45
7.44
7.43
7.43
7.43
7.42
7.37
7.36
7.36
7.35
7.34
7.33
7.33
7.32
7.31
7.30
7.30
7.29
7.29
7.28
7.27
7.27
7.27
7.26
7.25
6.10
3.69
3.68
3.63
3.60
3.58
2.03
NHCOOMe
COOMe
18a
2.03
3.21
1.00
3.04
2.96
3.10
f1 (ppm)

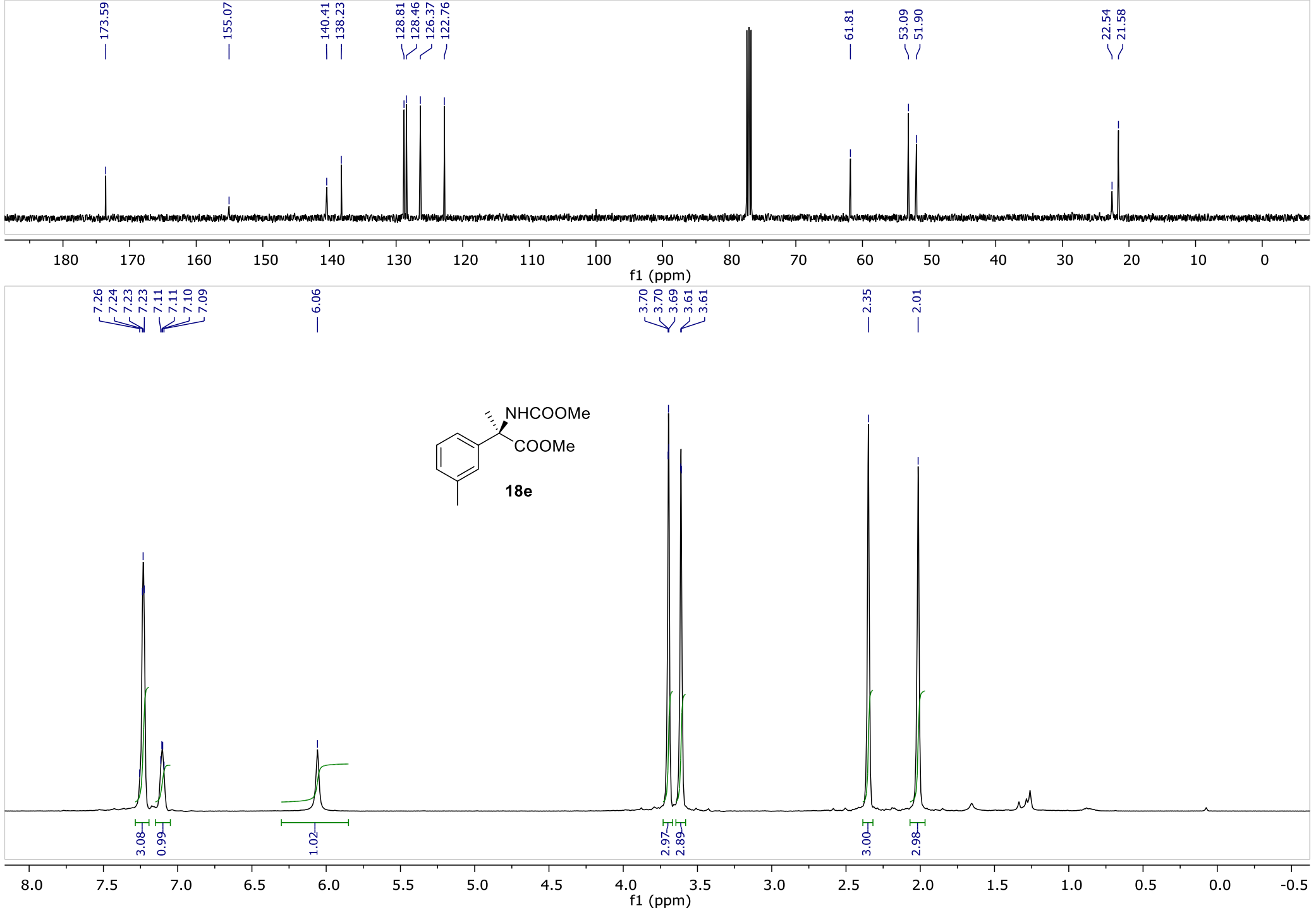

173.53
155.05
140.47
128.58
127.98
125.74
61.85
53.13
51.91
22.54
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

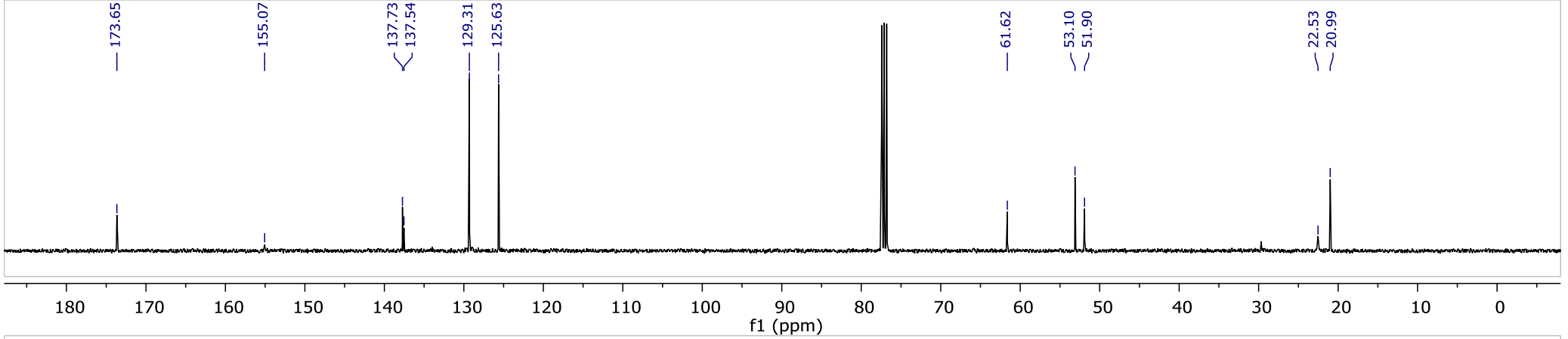

7.45 7.45 7.44 7.43 7.43 7.43 7.42 7.37 7.36 7.36 7.35 7.34 7.33 7.33 7.32 7.31 7.30 7.30 7.29 7.29 7.28 7.27 7.27 7.27 7.26 7.25 6.10
3.69 3.68 3.63 3.60 3.58
2.03
NHCOOMe
COOMe
18b
2.03 3.21 1.00 3.04 2.96 3.10
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

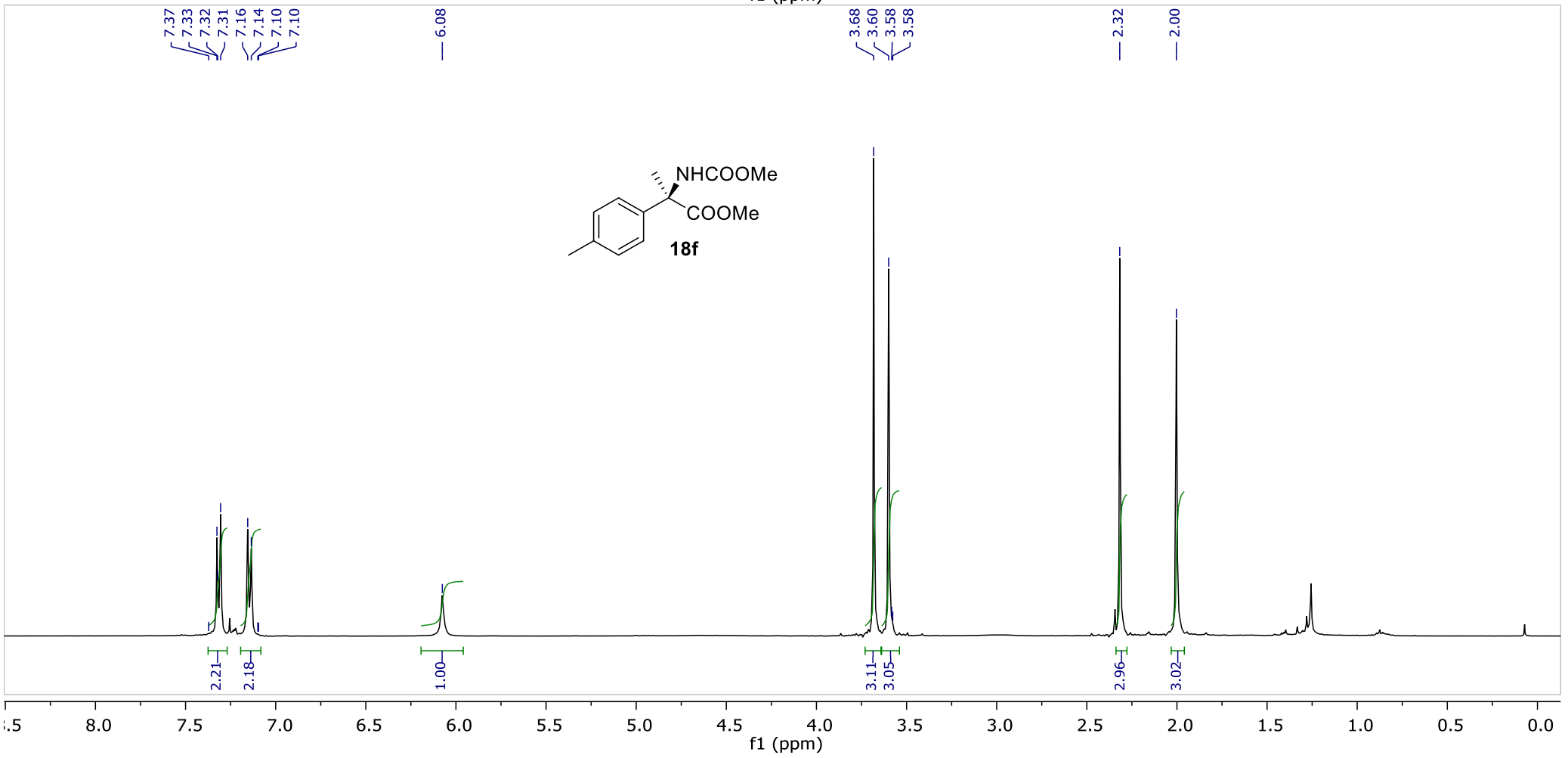

175.12
154.22
137.44
135.75
131.78
128.00
127.70
125.64
61.24
53.40
51.71
24.07
20.13
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)


7.57 7.56 7.55 7.54 7.27 7.26 7.25 7.25 7.24 7.23 7.23 7.22 7.22 7.21 7.20 7.19 7.18 7.12 7.12 7.10 7.10 7.09 6.34
3.79 3.78 3.77 3.76 3.76 3.75 3.74 3.73 3.72 3.70 3.69 3.69 3.60 3.59 3.58 3.53
2.29 2.28 2.28 2.27 2.27 2.26 2.23 2.22 2.21 2.19 2.19 2.18 2.16 2.15 2.13 2.13 2.11 2.11 2.08 2.06 2.03
NHCOOMe
COOMe
18c
1.06 2.22 0.98 1.00 3.25 3.04 3.20 3.09
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
f1 (ppm)


175.12
154.22
137.44
135.75
131.78
128.00
127.70
125.64
61.24
53.40
51.71
24.07
20.13
f1 (ppm)
7.57
7.55
7.31
7.29
7.26
7.25
7.24
7.24
7.23
7.22
7.22
7.21
7.21
7.19
7.19
7.12
7.11
7.10
7.09
6.33
3.72
3.53
2.20
2.05
NHCOOMe
COOMe
18d
0.95
2.09
0.93
1.00
2.85
2.75
2.80
2.92
f1 (ppm)


173.59
155.07
140.41
138.23
128.81
128.46
126.37
122.76
61.81
53.09
51.90
22.54
21.58
f1 (ppm)
7.26
7.24
7.23
7.23
7.11
7.11
7.10
7.09
6.06
3.70
3.70
3.69
3.61
3.61
2.35
2.01
NHCOOMe
COOMe
18e
3.08
0.99
1.02
2.97
2.89
3.00
2.98
f1 (ppm)


173.65
155.07
137.73
137.54
129.31
125.63
61.62
53.10
51.90
22.53
20.99
f1 (ppm)


7.37
7.33
7.32
7.31
7.16
7.14
7.10
7.10
6.08
3.68
3.60
3.58
3.58
2.32
2.00
NHCOOMe
COOMe
18f
2.21
2.18
1.00
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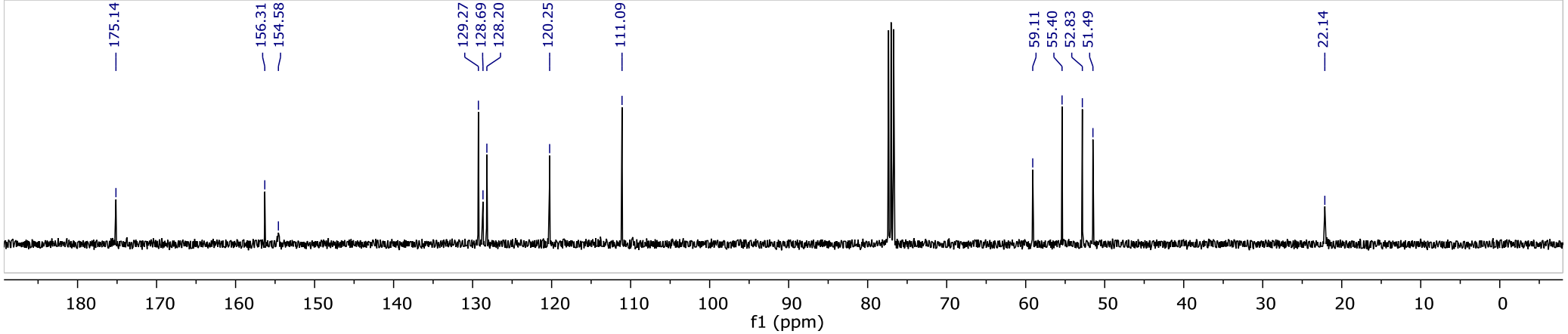
2.96
3.02
f1 (ppm)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111.09
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55.40
52.83
51.49
22.14
180
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140
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120
110
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90
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50
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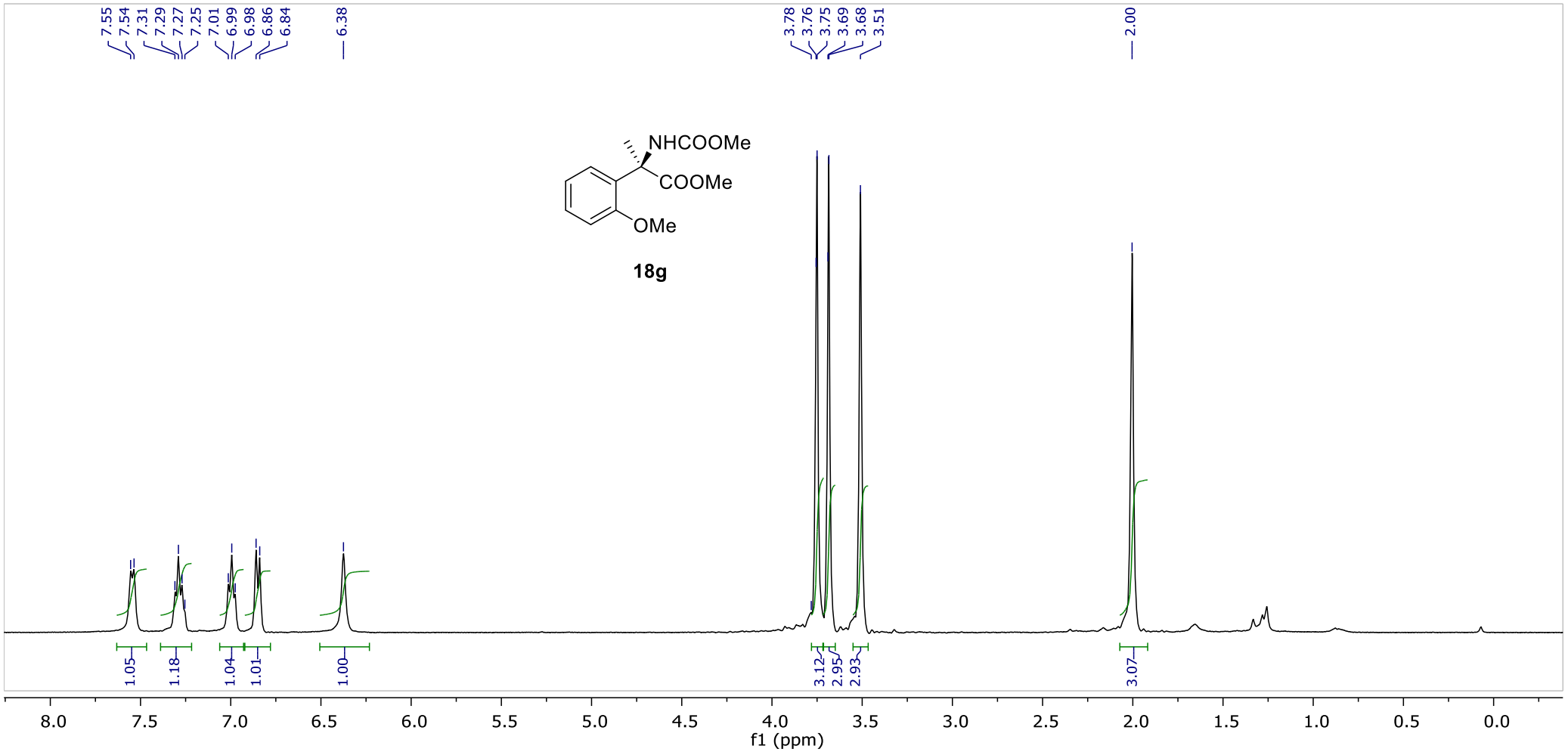
10
0
f1 (ppm)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6.84
6.38
3.78
3.76
3.75
3.69
3.68
3.51
2.00
NHCOOMe
COOMe
OMe
18g
1.05
1.18
1.04
1.01
1.00
3.12
2.95
2.93
3.07
8.0
7.5
7.0
6.5
6.0
5.5
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4.5
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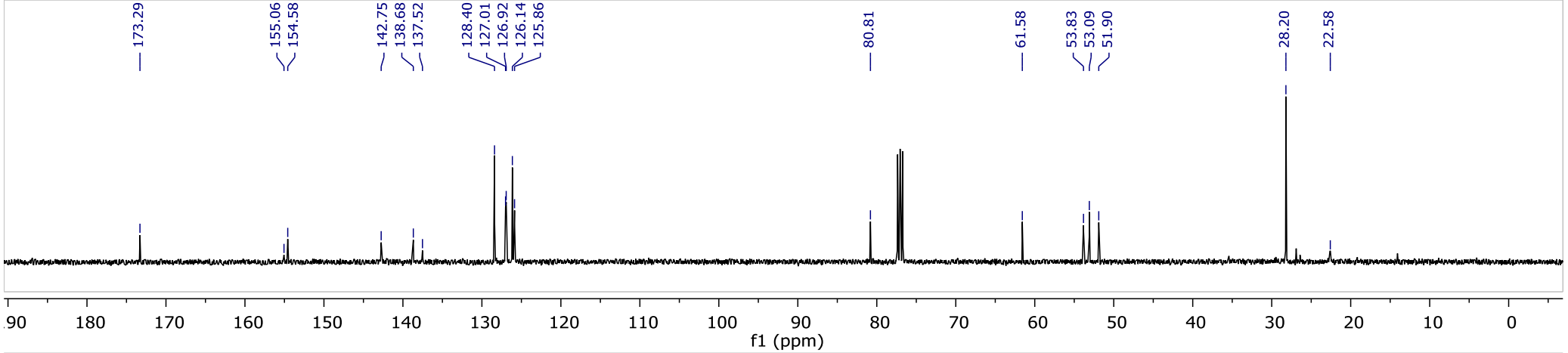
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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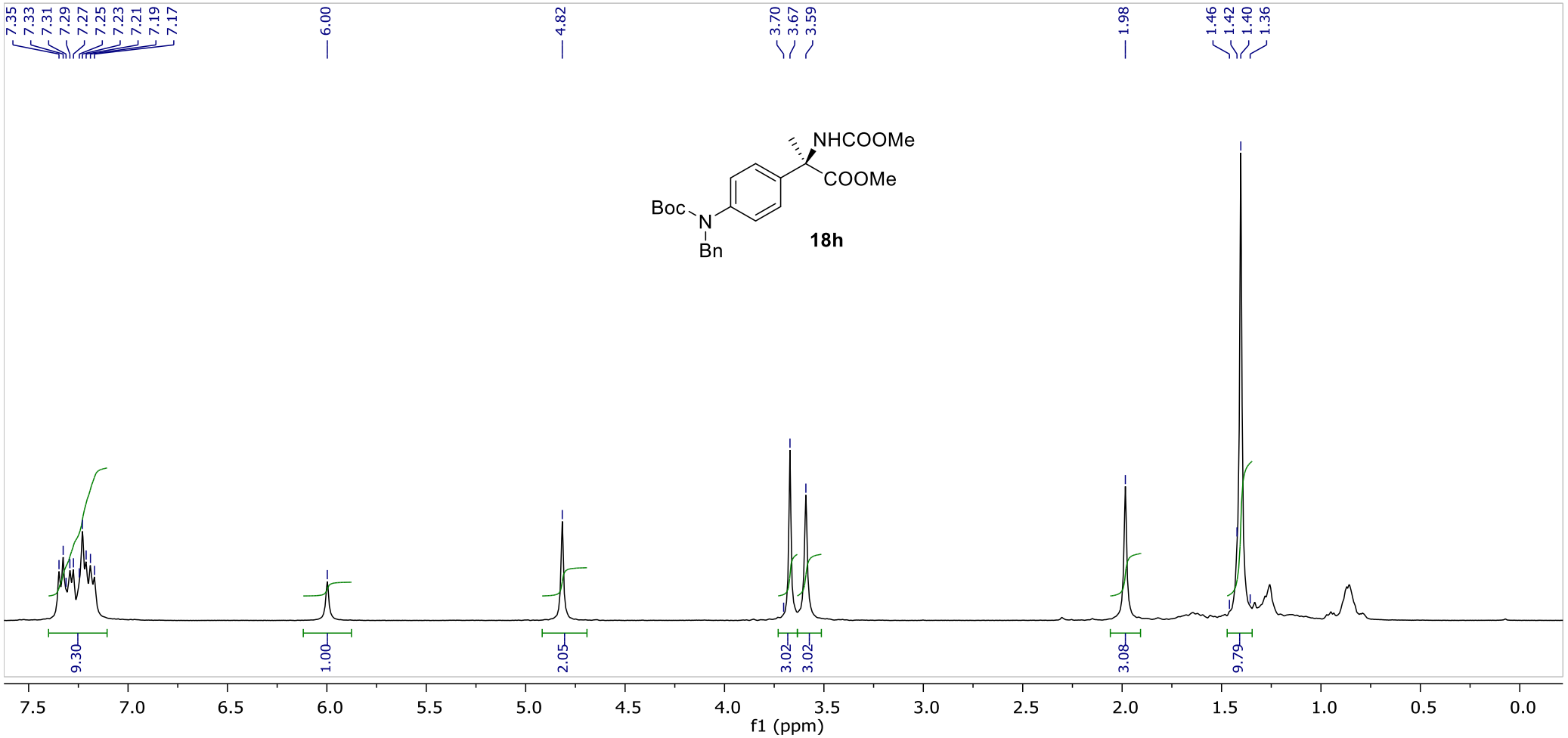
53.09
51.90
28.20
22.58
f1 (ppm)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7.21
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7.17
6.00
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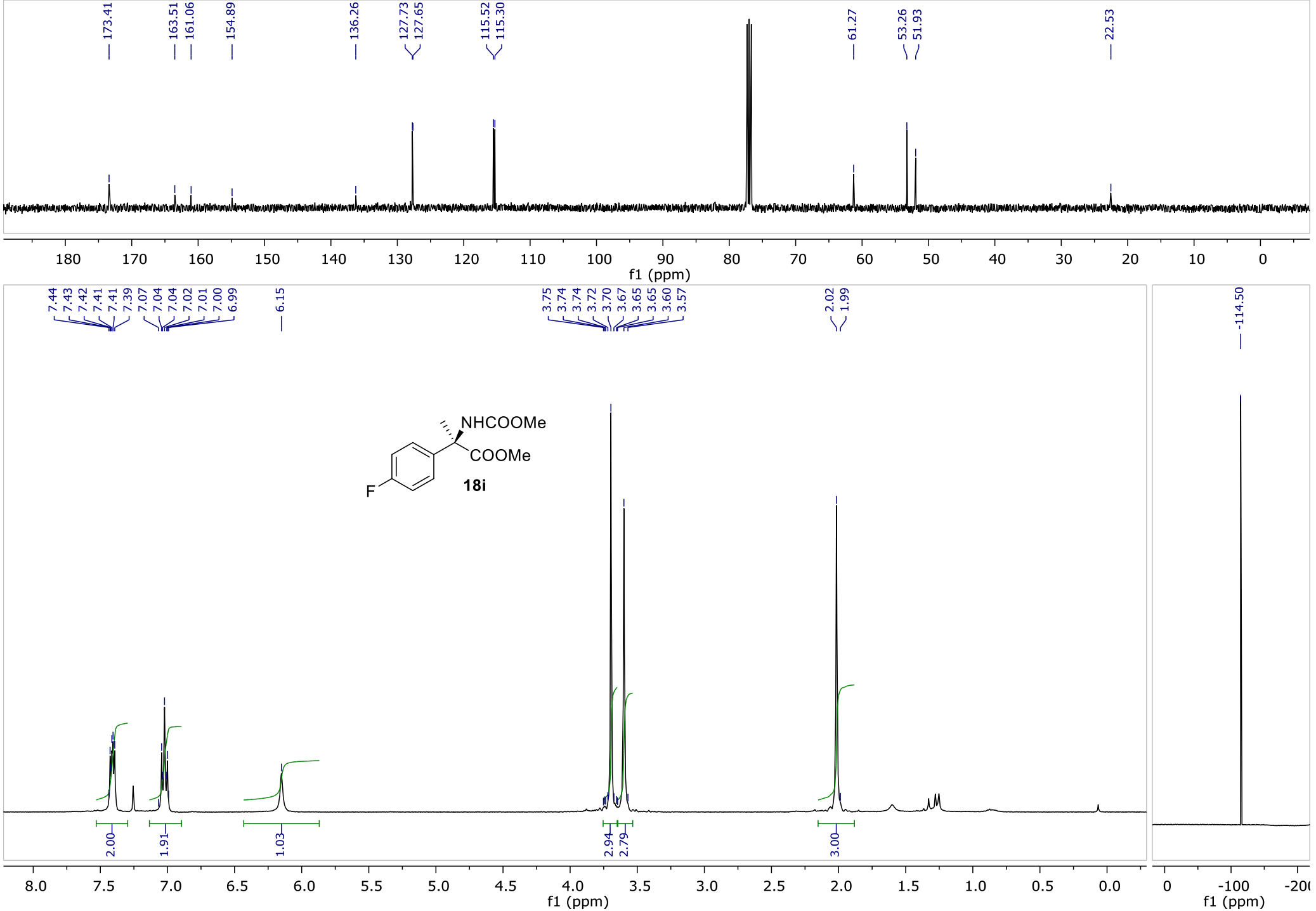
3.70
3.67
3.59
1.98
1.46
1.42
1.40
1.36
NHCOOMe
COOMe
Boc
N
Bn
18h
9.30
1.00
2.05
3.02
3.02
3.08
9.79
f1 (ppm)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F
18i
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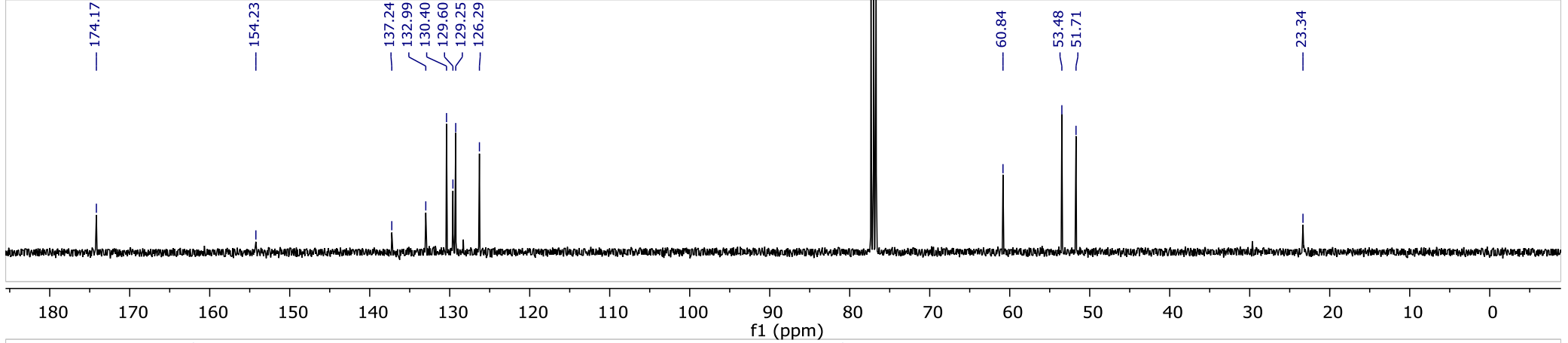
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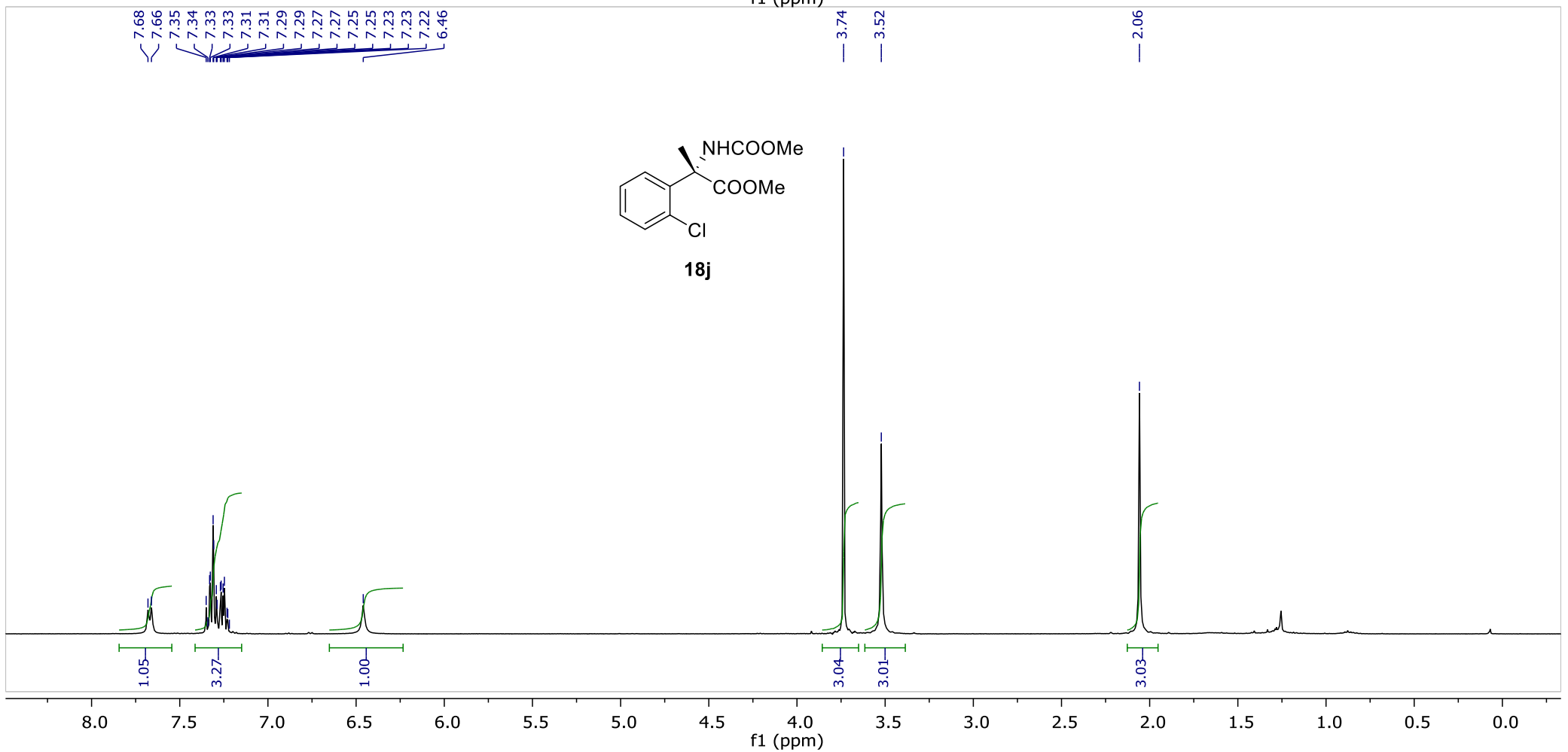
129.25
126.29
60.84
53.48
51.71
23.34
f1 (ppm)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7.29
7.29
7.27
7.27
7.25
7.25
7.23
7.23
7.22
6.46
3.74
3.52
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COOMe
Cl
18j
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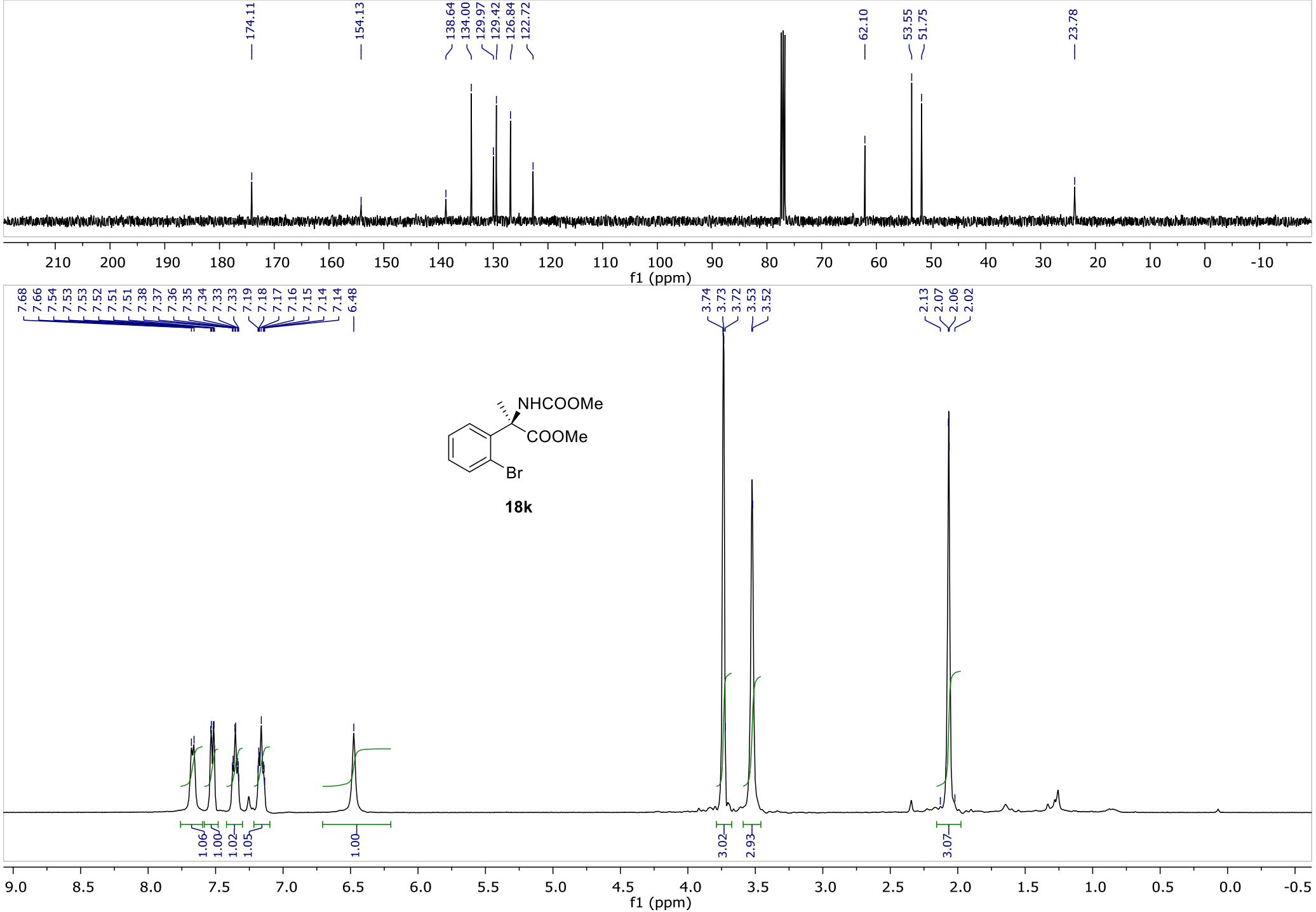
3.27
1.00
3.04
3.01
3.03
f1 (ppm)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126.84
122.72
62.10
53.55
51.75
23.78
f1 (ppm)
7.68
7.66
7.54
7.53
7.53
7.52
7.51
7.51
7.38
7.37
7.36
7.35
7.34
7.33
7.33
7.19
7.18
7.17
7.16
7.15
7.14
7.14
6.48
3.74
3.73
3.72
3.53
3.52
2.13
2.07
2.06
2.02
NHCOOMe
COOMe
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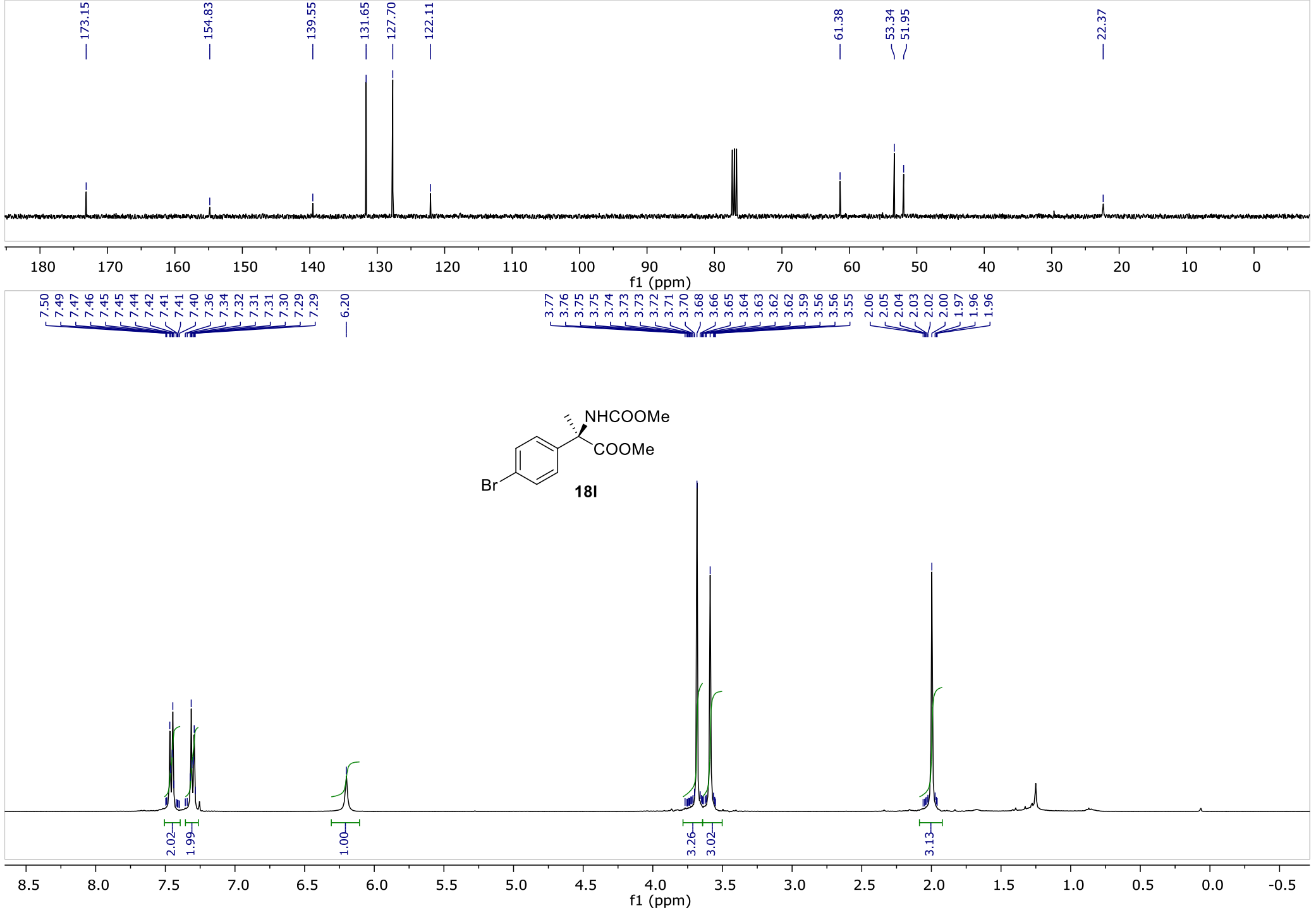
1.00
1.02
1.05
1.00
3.02
2.93
3.07
f1 (ppm)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154.83
139.55
131.65
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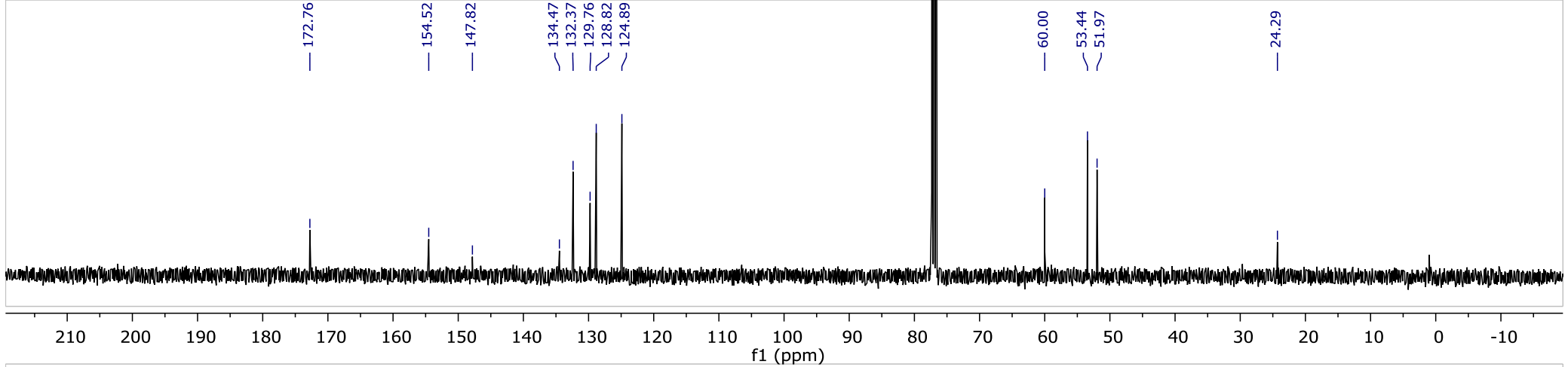
22.37
f1 (ppm)
NHCOOMe
COOMe
Br
18l
f1 (ppm)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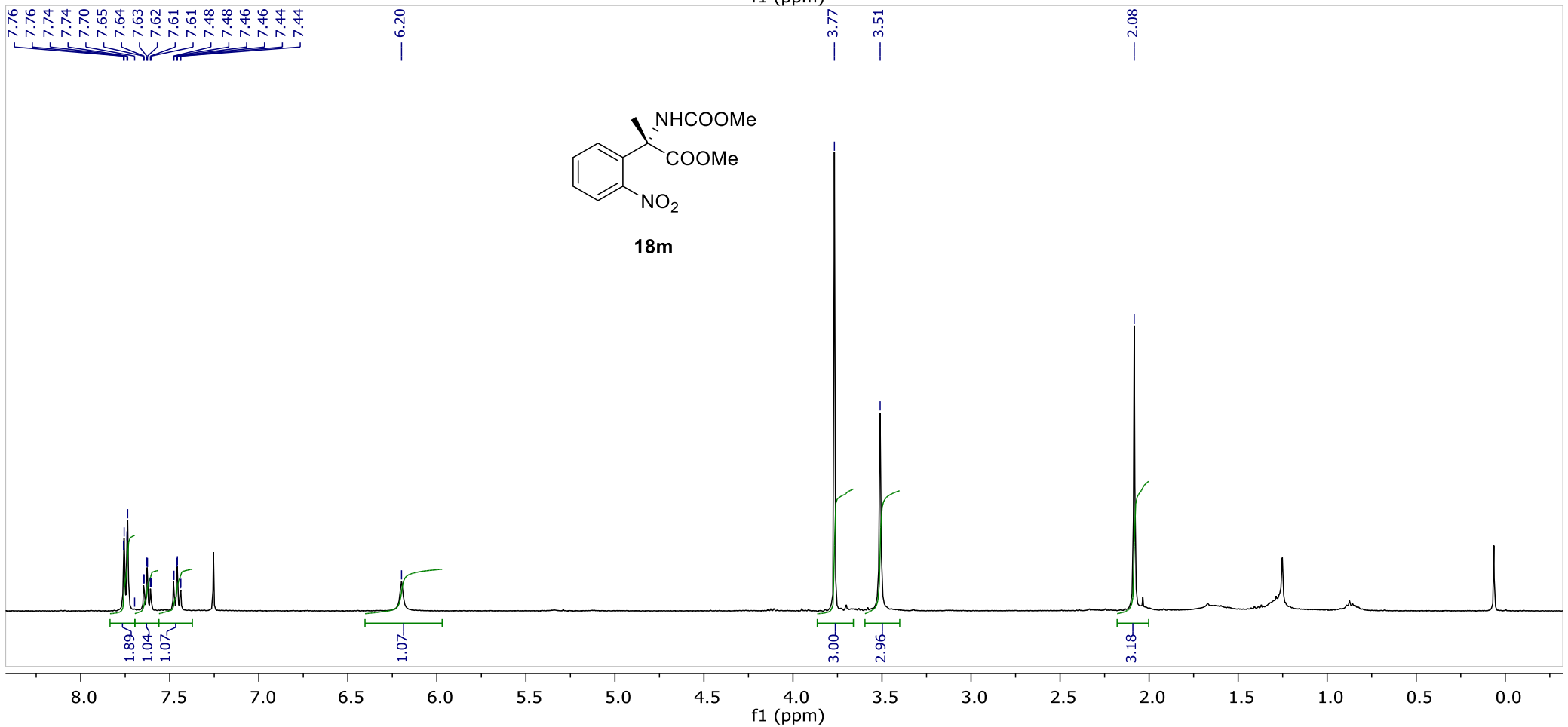
60.00
53.44
51.97
24.29
f1 (ppm)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7.64
7.63
7.62
7.61
7.61
7.48
7.48
7.46
7.46
7.44
7.44
6.20
3.77
3.51
2.08
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COOMe
NO2
18m
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1.04
1.07
1.07
3.00
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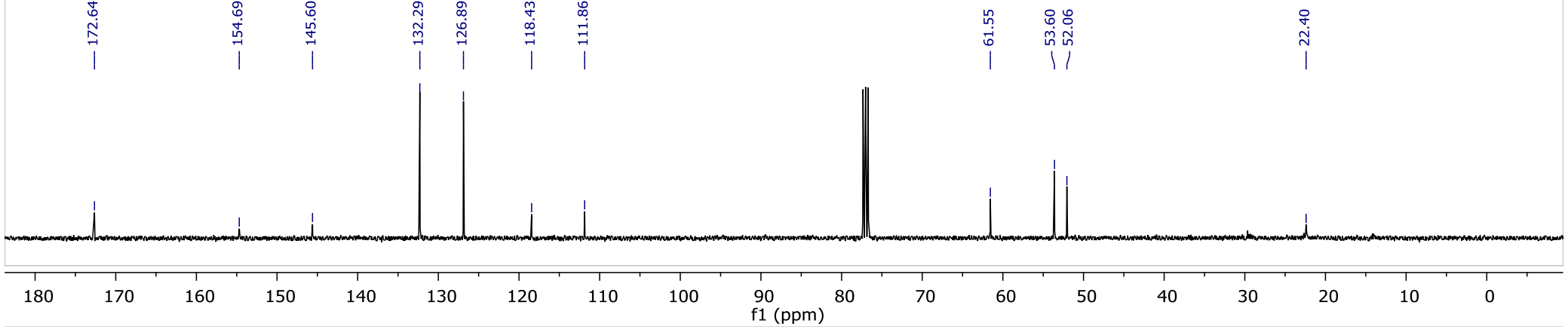
3.18
f1 (ppm)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111.86
61.55
53.60
52.06
22.40
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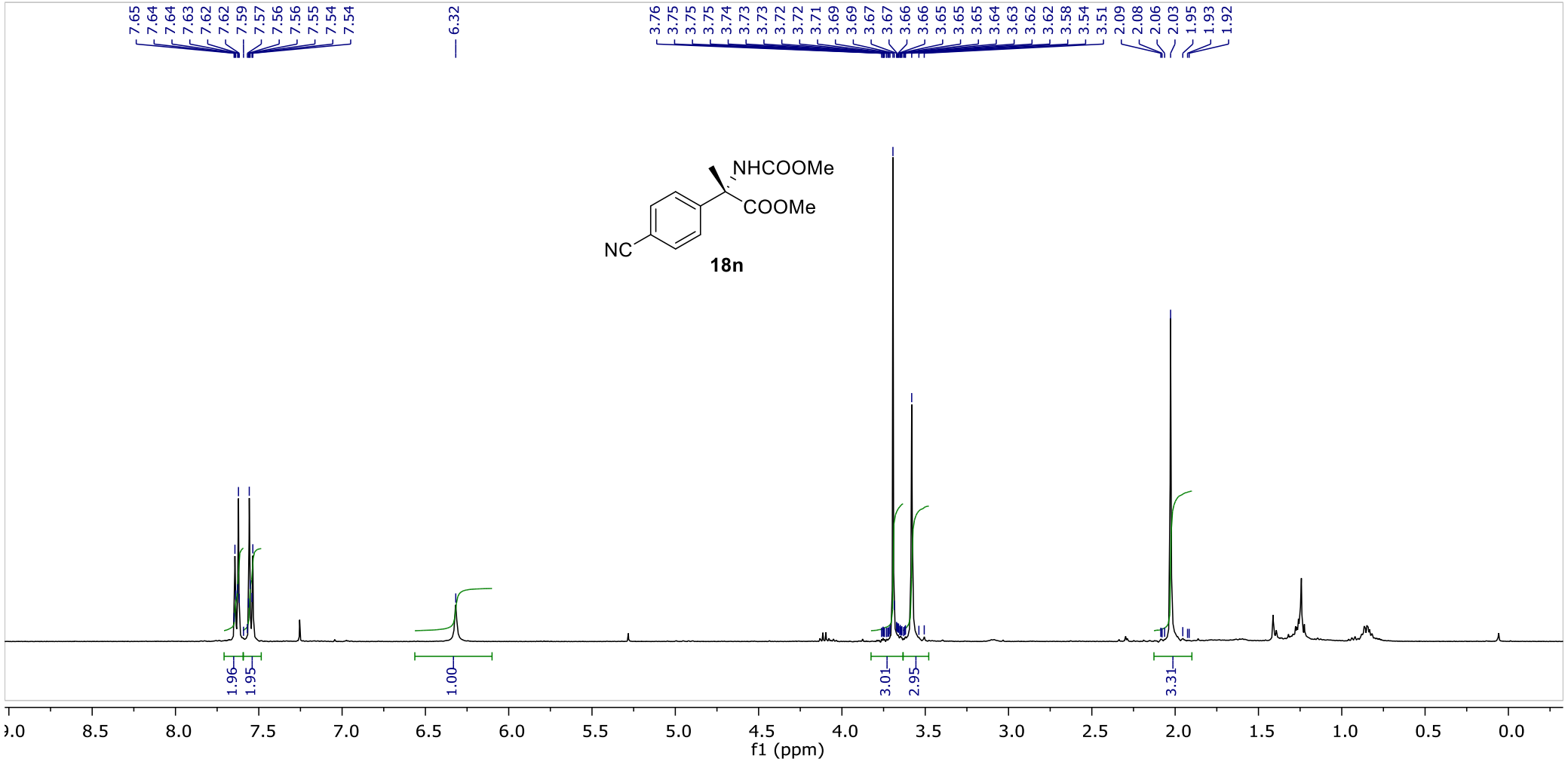
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COOMe
NC
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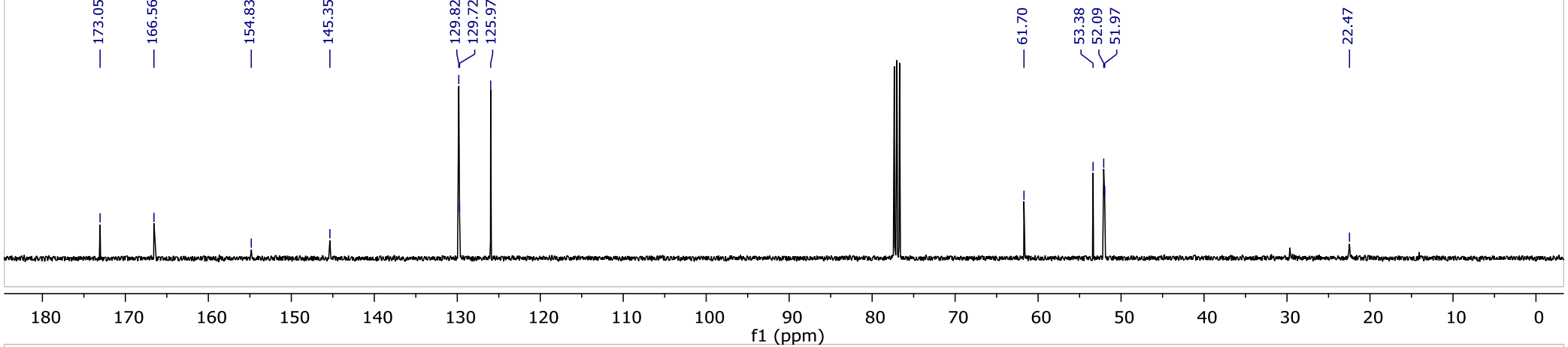
3.31
f1 (ppm)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52.09
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22.47
180
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130
120
110
100
90
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50
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20
10
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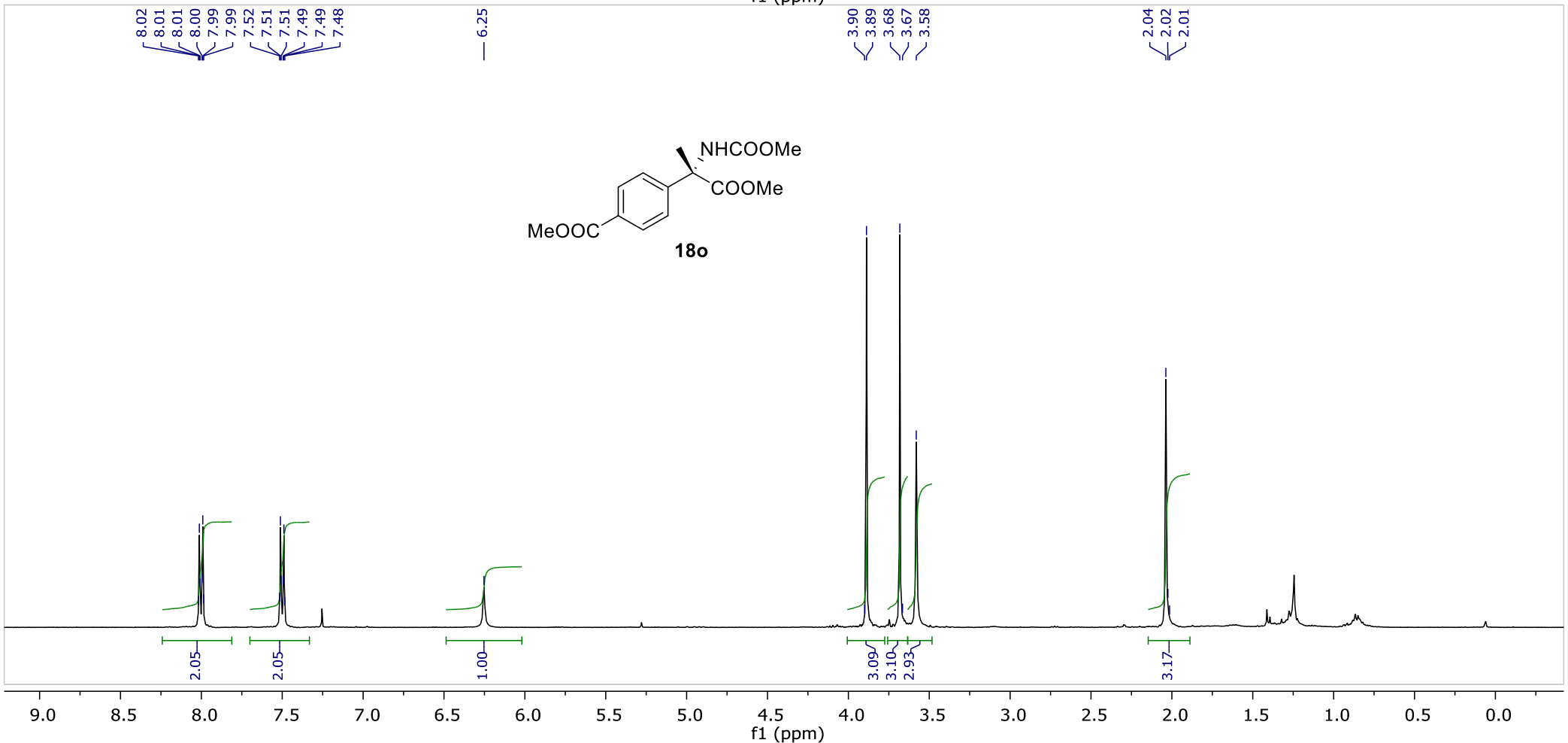
f1 (ppm)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7.51
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7.49
7.48
6.25
3.90
3.89
3.68
3.67
3.58
2.04
2.02
2.01
NHCOOMe
COOMe
MeOOC
18o
2.05
2.05
1.00
3.09
3.10
2.93
3.17
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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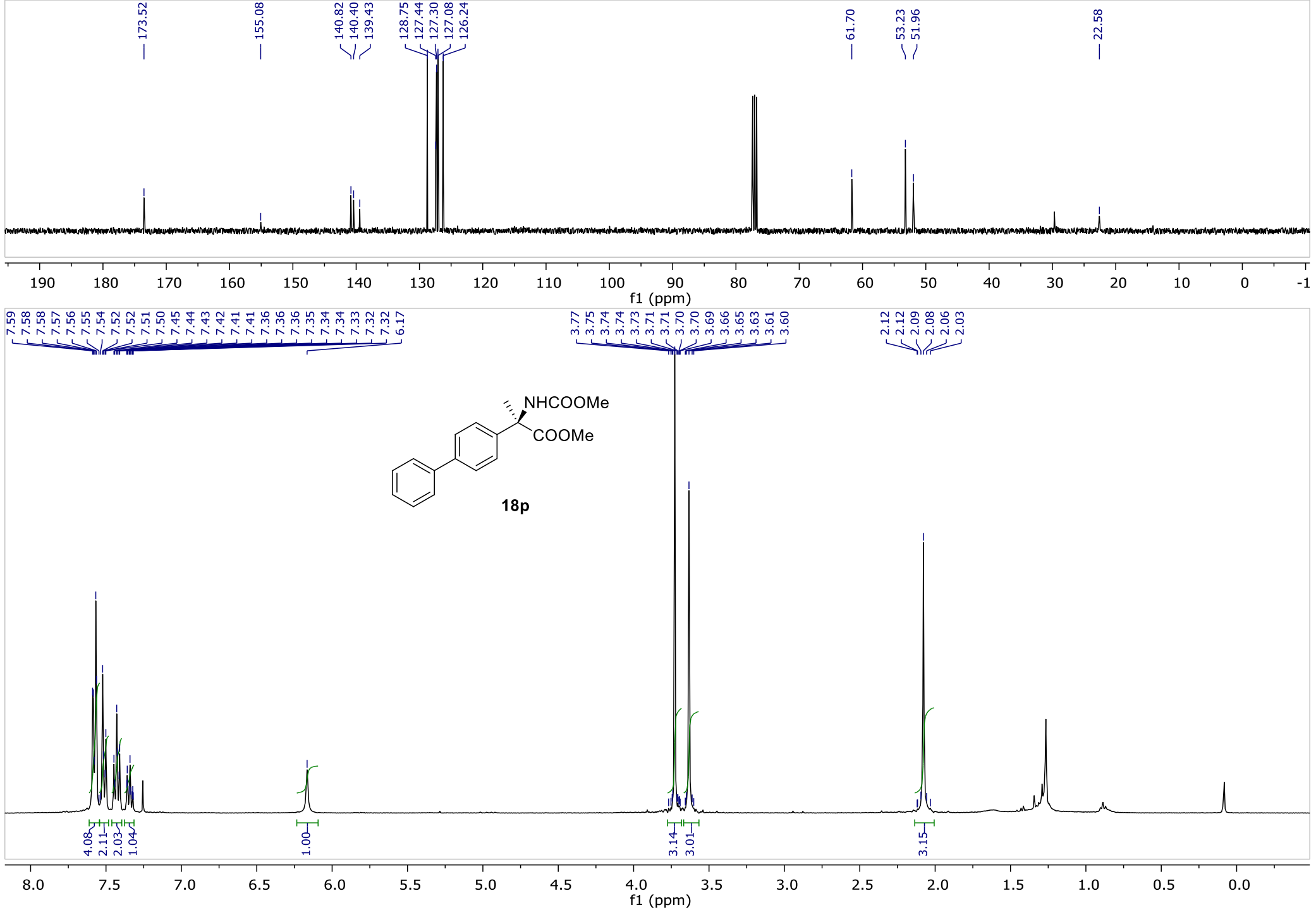
0.0
f1 (ppm)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127.30
127.08
126.24
61.70
53.23
51.96
22.58
f1 (ppm)
7.59 7.58 7.58 7.57 7.56 7.55 7.54 7.52 7.52 7.51 7.50 7.45 7.44 7.43 7.42 7.41 7.41 7.36 7.36 7.36 7.35 7.34 7.34 7.33 7.32 7.32 6.17
3.77 3.75 3.74 3.74 3.73 3.71 3.71 3.70 3.70 3.69 3.66 3.65 3.63 3.61 3.60
2.12 2.12 2.09 2.08 2.06 2.03
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COOMe
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4.08
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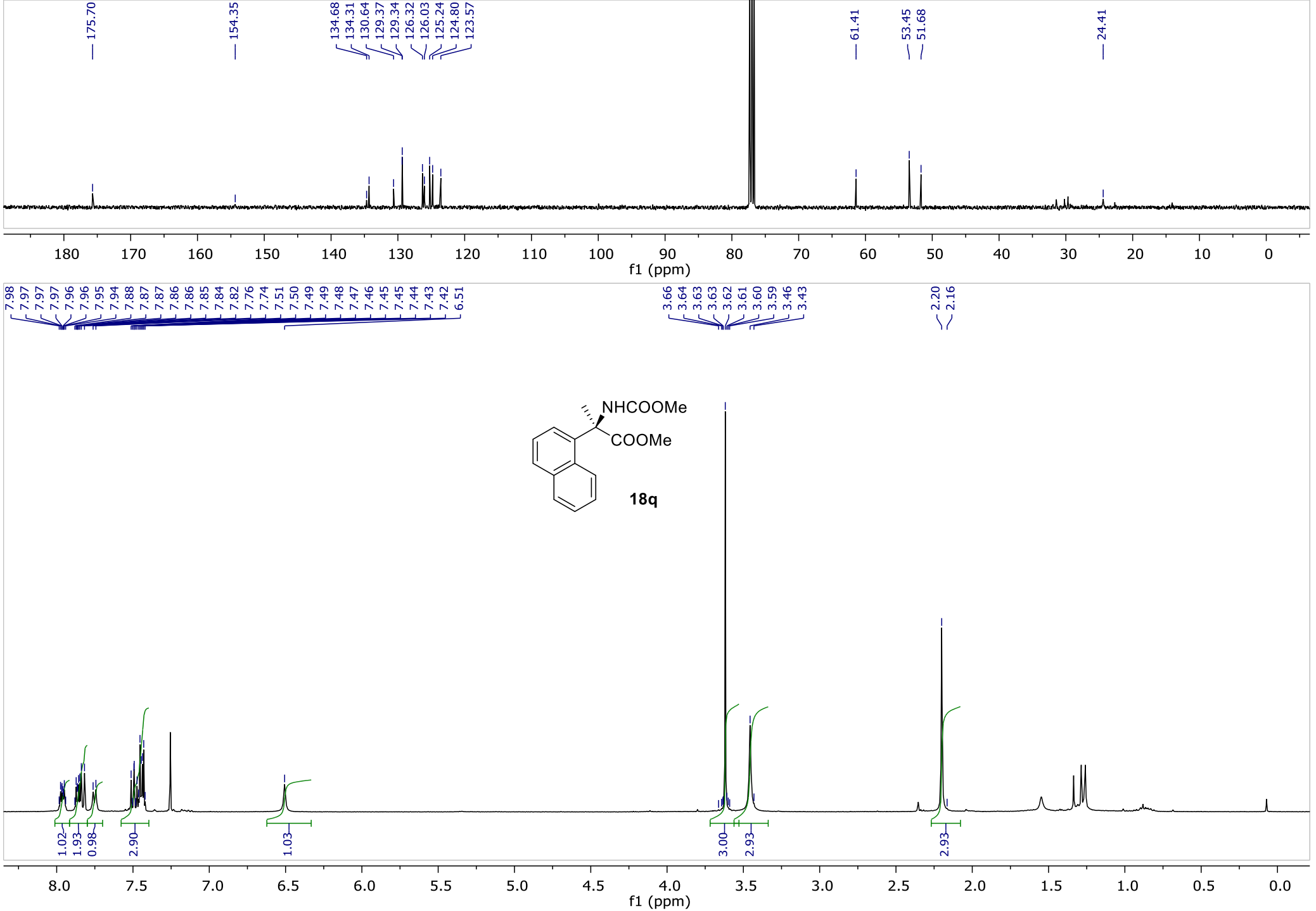
1.04
1.00
3.14
3.01
3.15
f1 (ppm)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53.45
51.68
24.41
f1 (ppm)
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7.97
7.97
7.96
7.96
7.95
7.94
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7.87
7.87
7.86
7.86
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3.62
3.61
3.60
3.59
3.46
3.43
2.20
2.16
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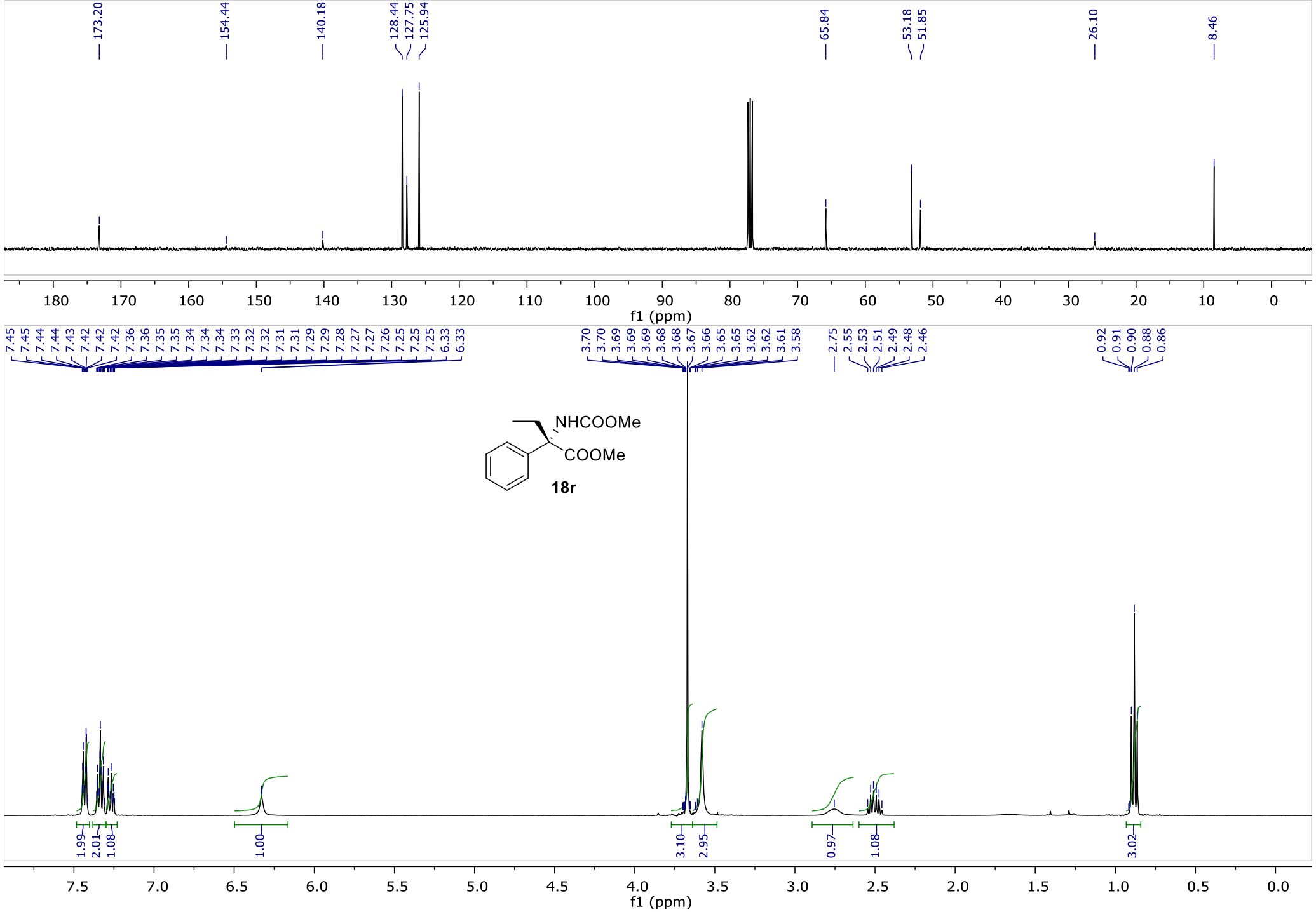
2.93
f1 (ppm)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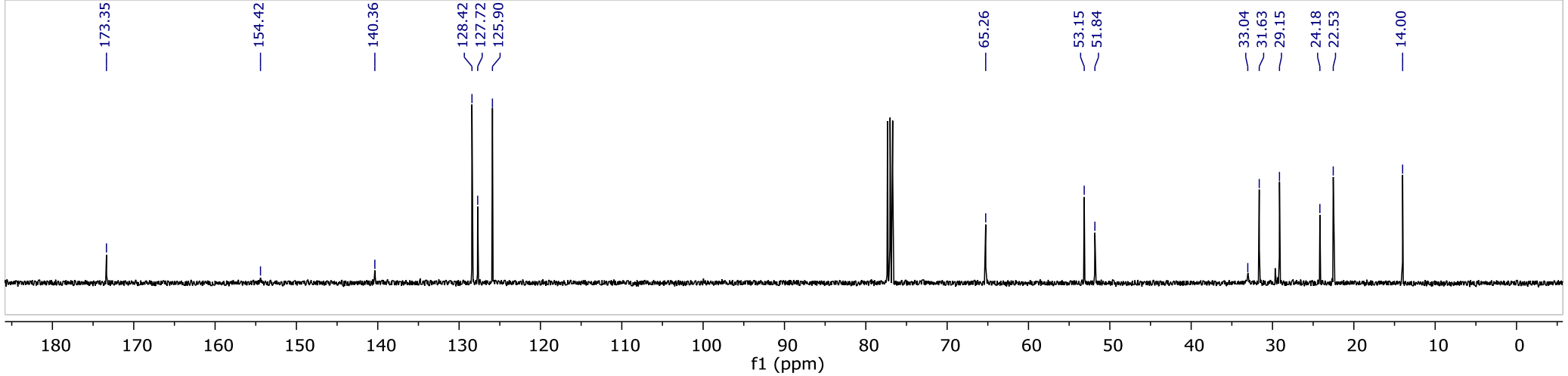
f1 (ppm)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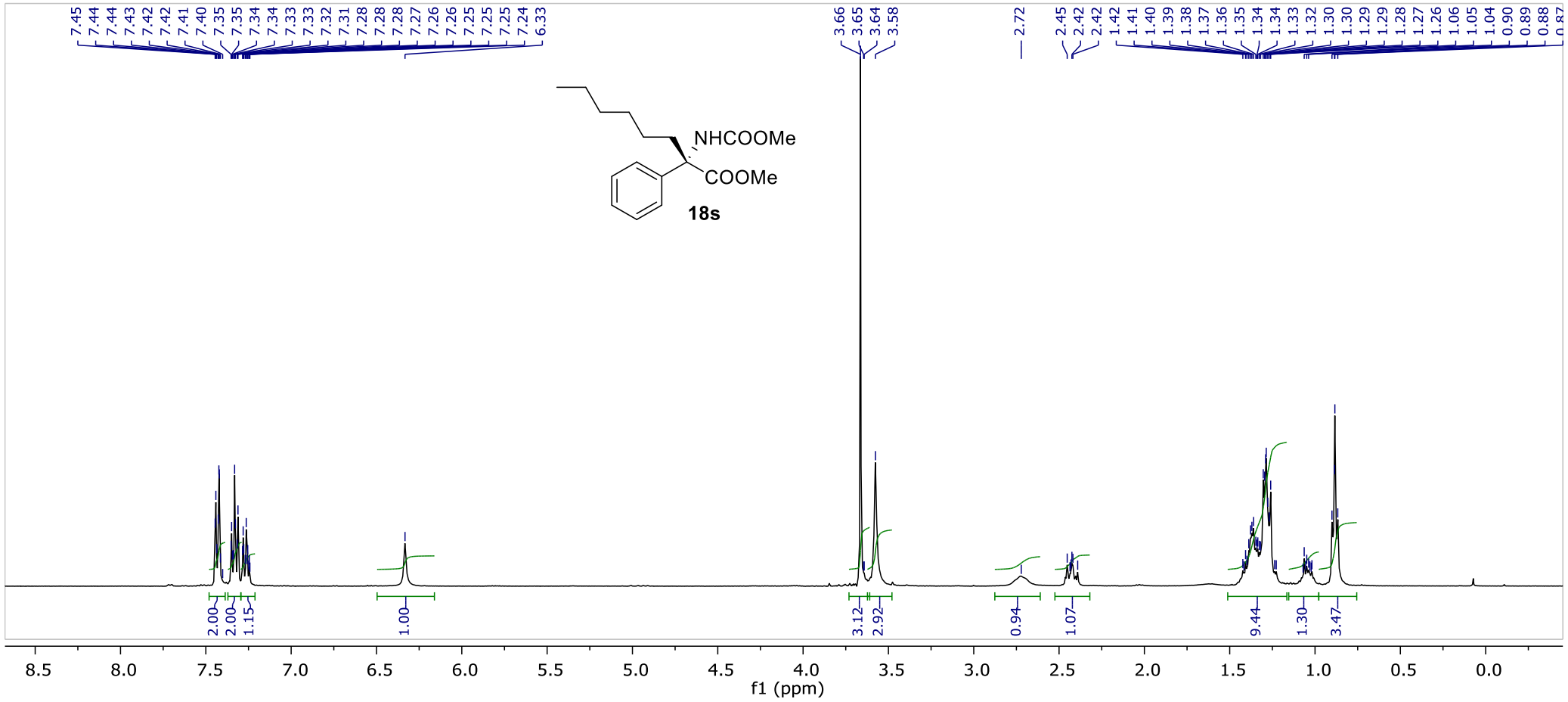
14.00
f1 (ppm)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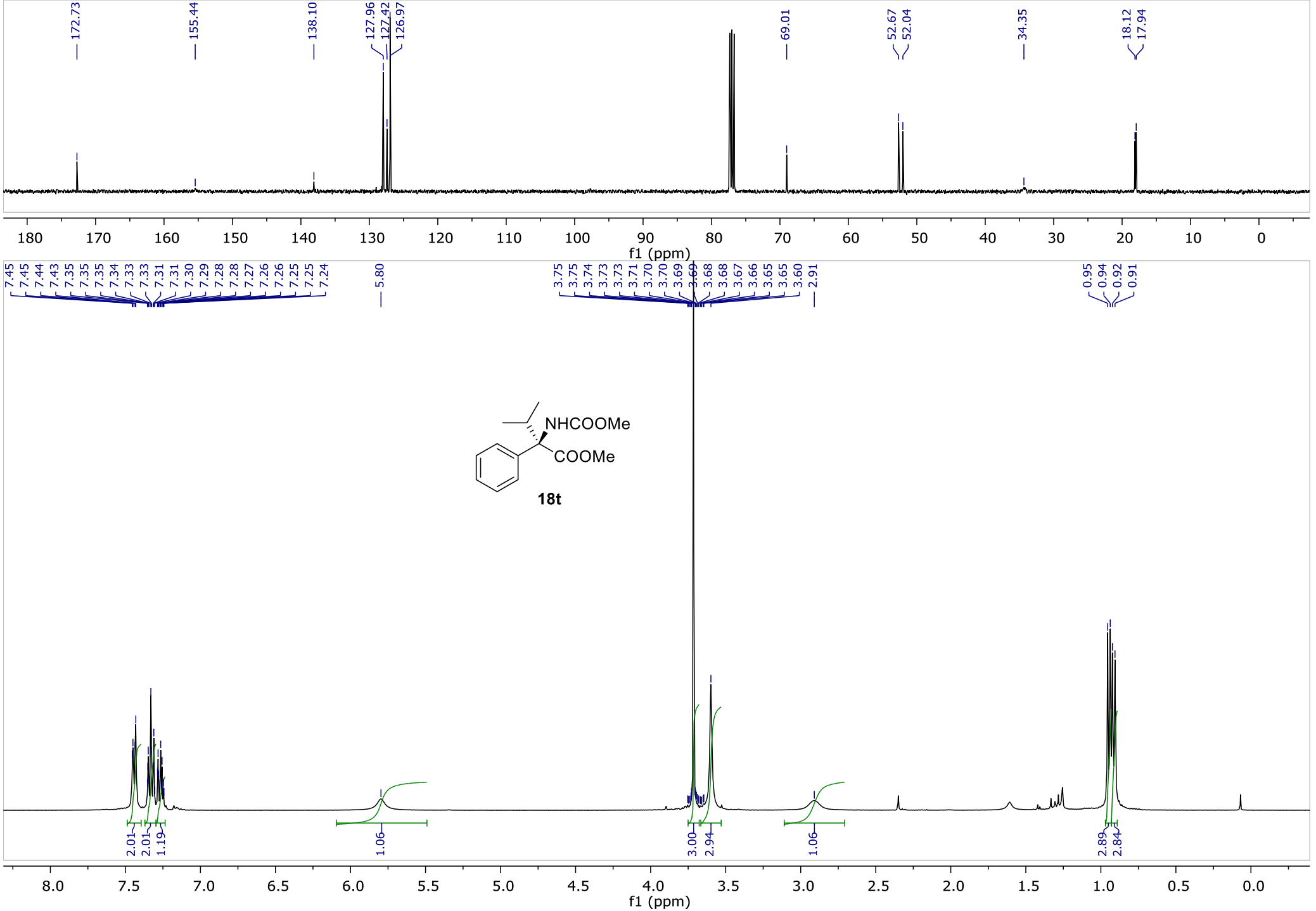
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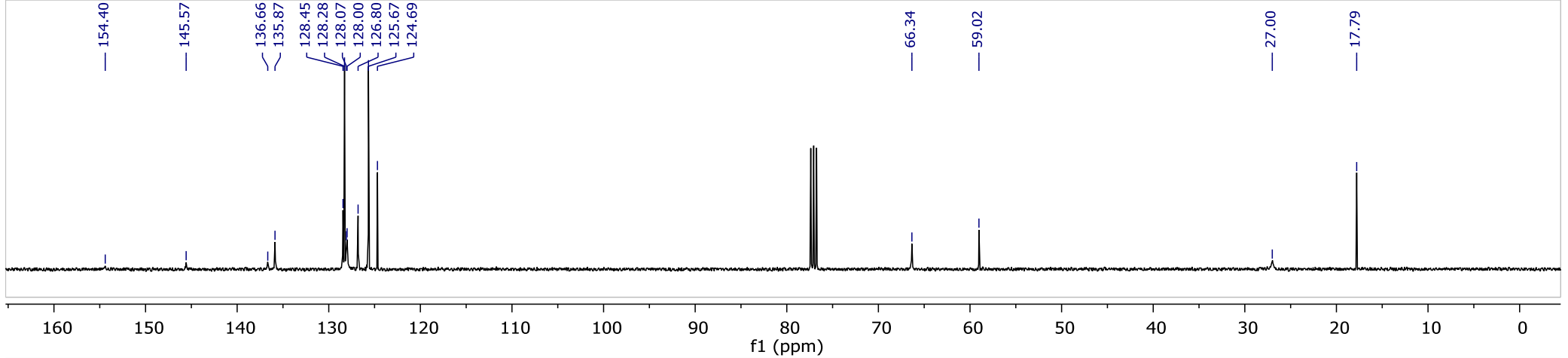
18t
f1 (ppm)
f1 (ppm)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124.69
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27.00
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70
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50
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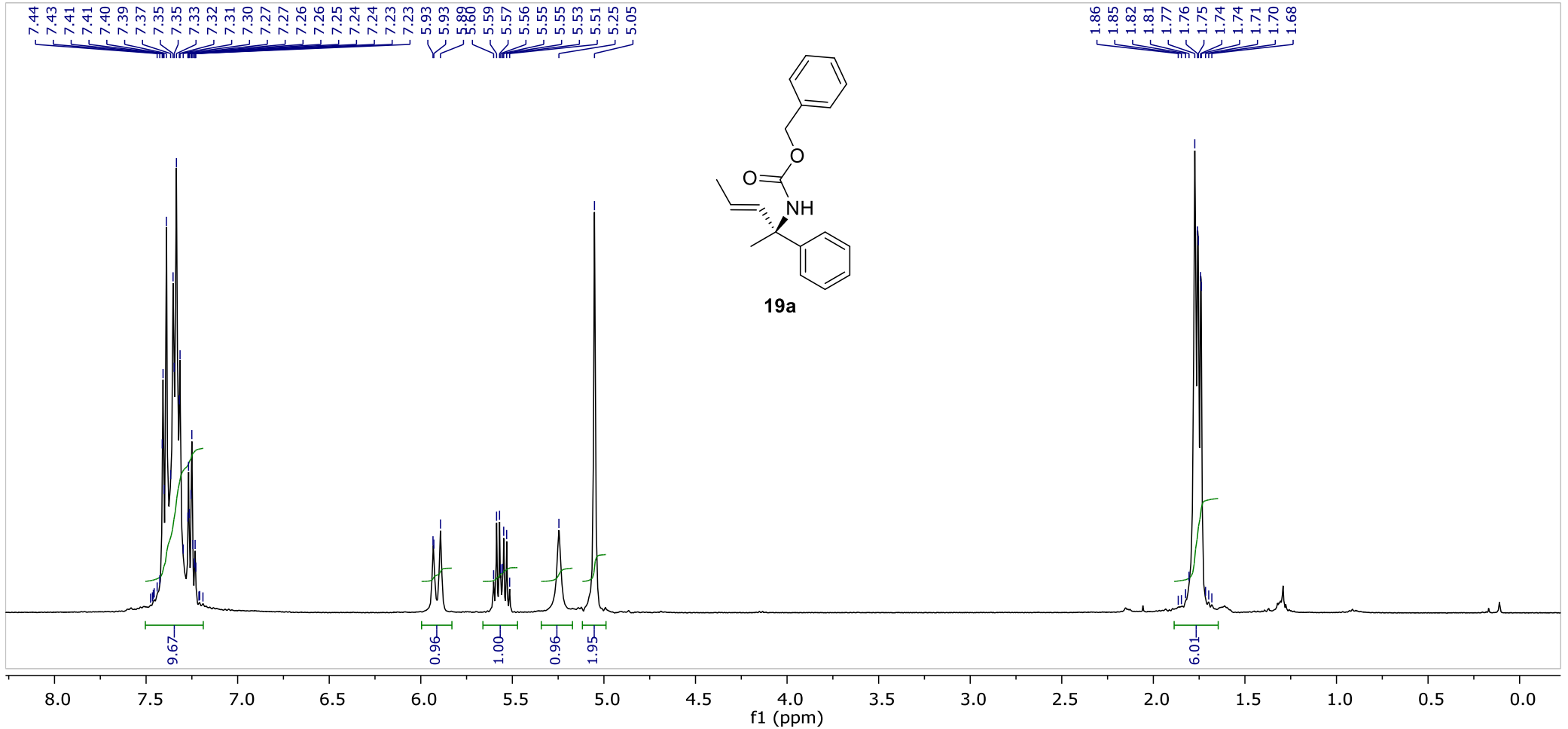
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5.25
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1.81
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1.76
1.75
1.74
1.74
1.71
1.70
1.68
O
O
NH
19a
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1.95
6.01
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7.0
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6.0
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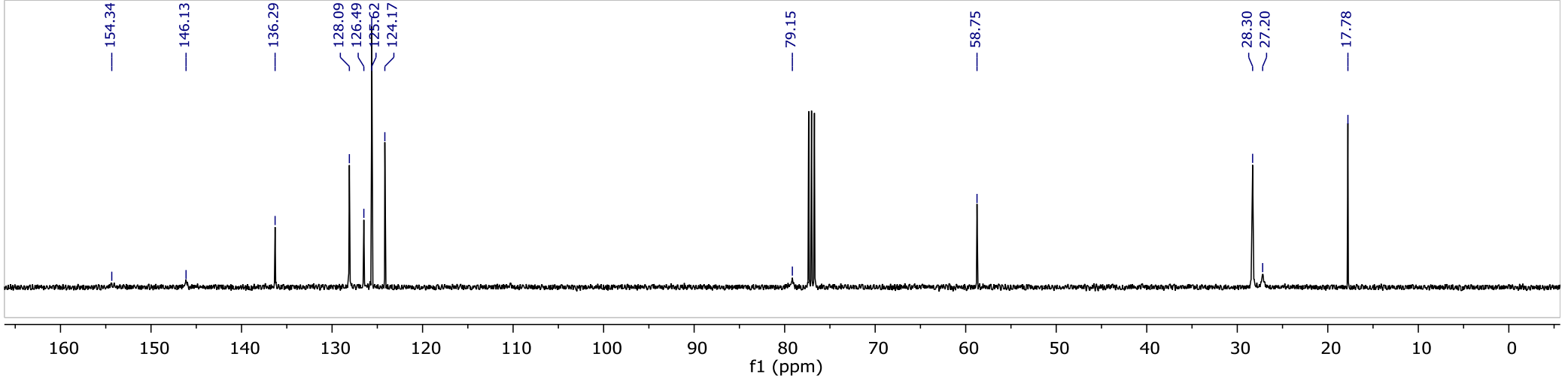
f1 (ppm)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110
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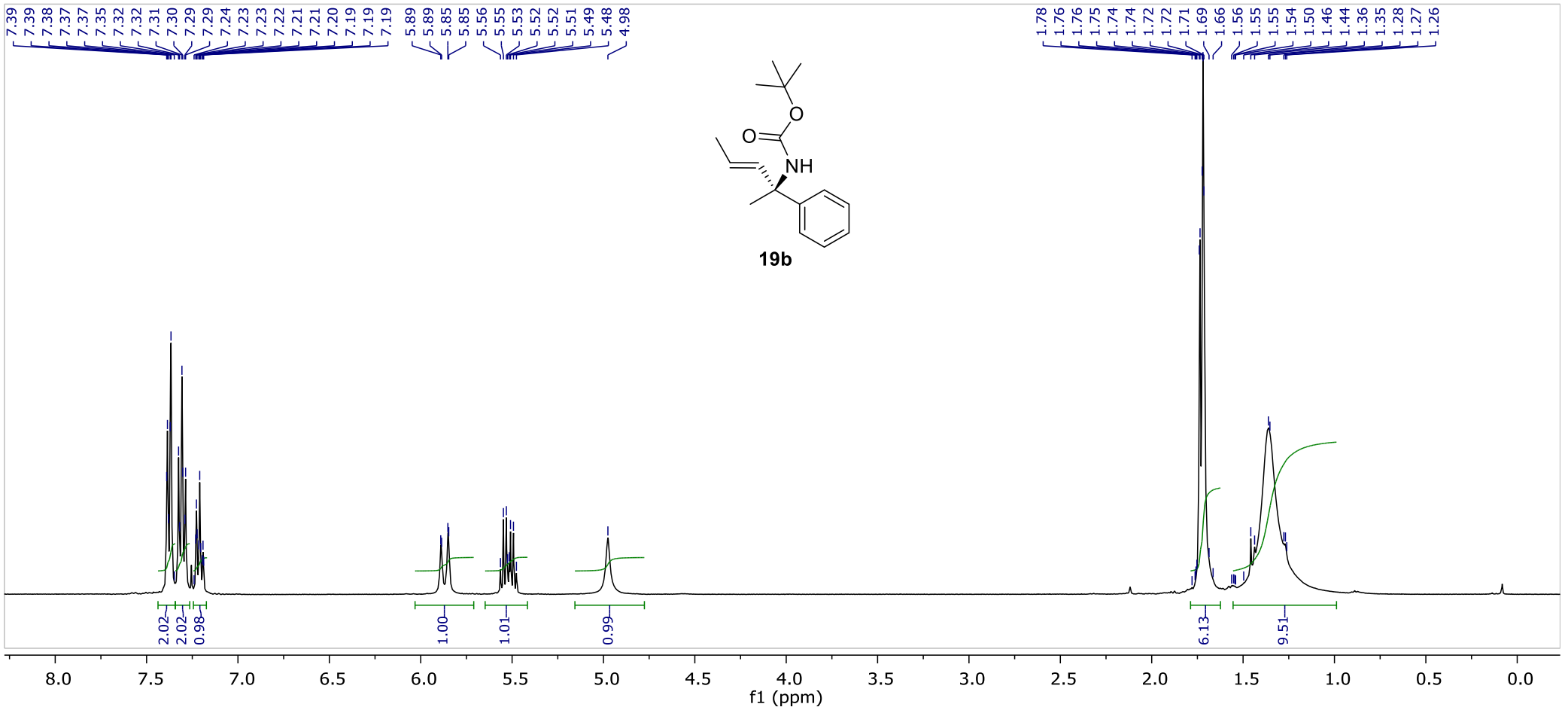
f1 (ppm)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1.72
1.72
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1.69
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1.50
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1.35
1.28
1.27
1.26
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O
NH
19b
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2.02
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1.01
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9.51
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7.0
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6.0
5.5
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4.5
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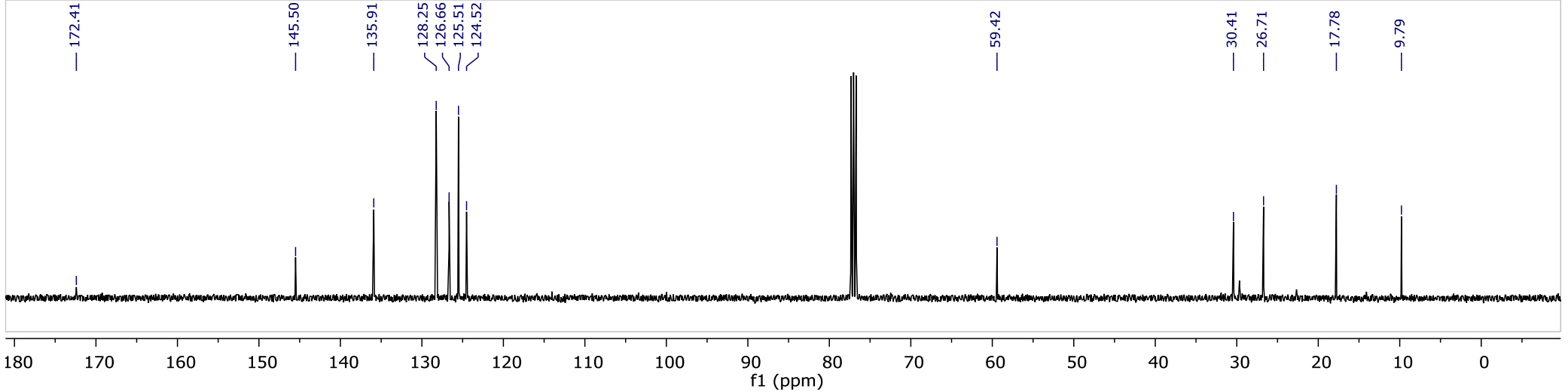
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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26.71
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9.79
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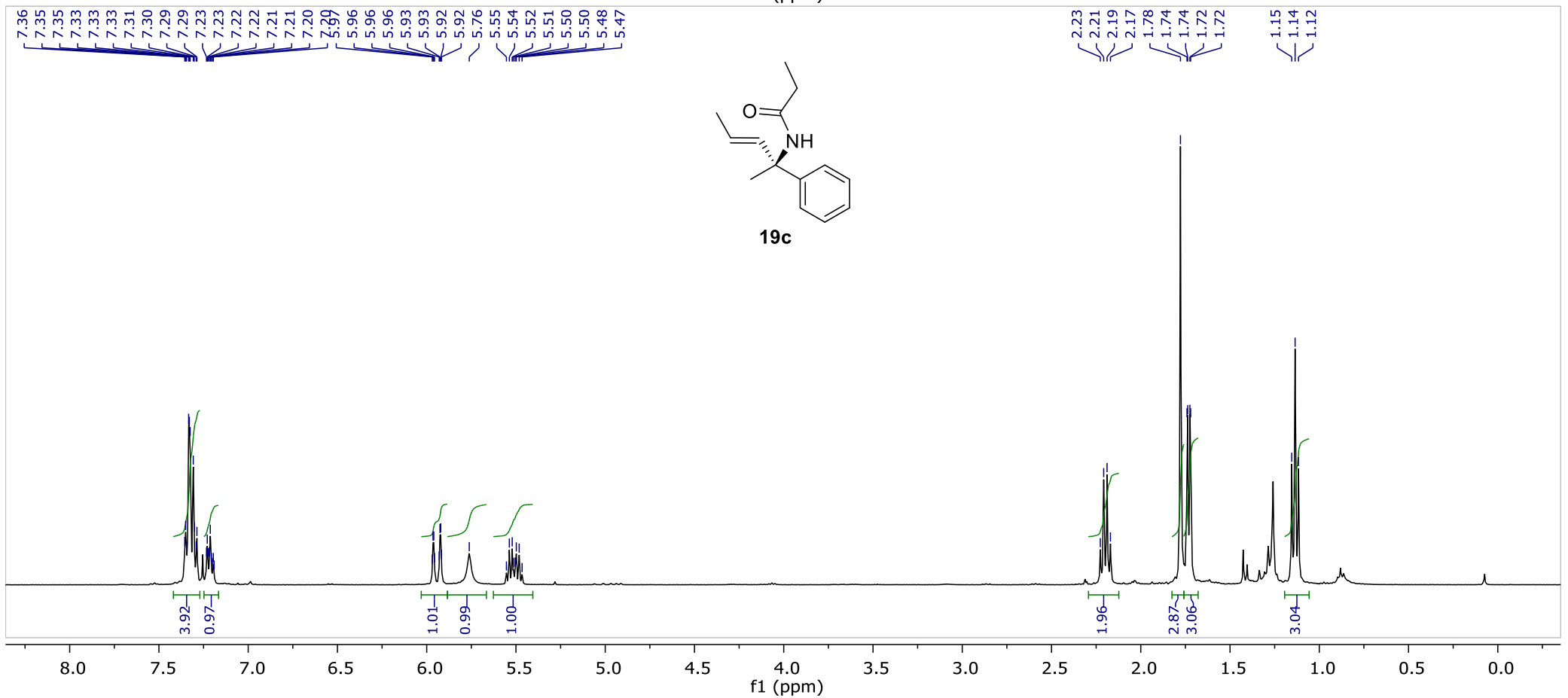
f1 (ppm)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5.97 5.96 5.96 5.96 5.93 5.93 5.92 5.92 5.76 5.55 5.54 5.52 5.51 5.50 5.50 5.48 5.47
2.23 2.21 2.19 2.17 1.78 1.74 1.74 1.72 1.72 1.15 1.14 1.12
O
NH
19c
3.92 0.97 1.01 0.99 1.00 1.96 2.87 3.06 3.04
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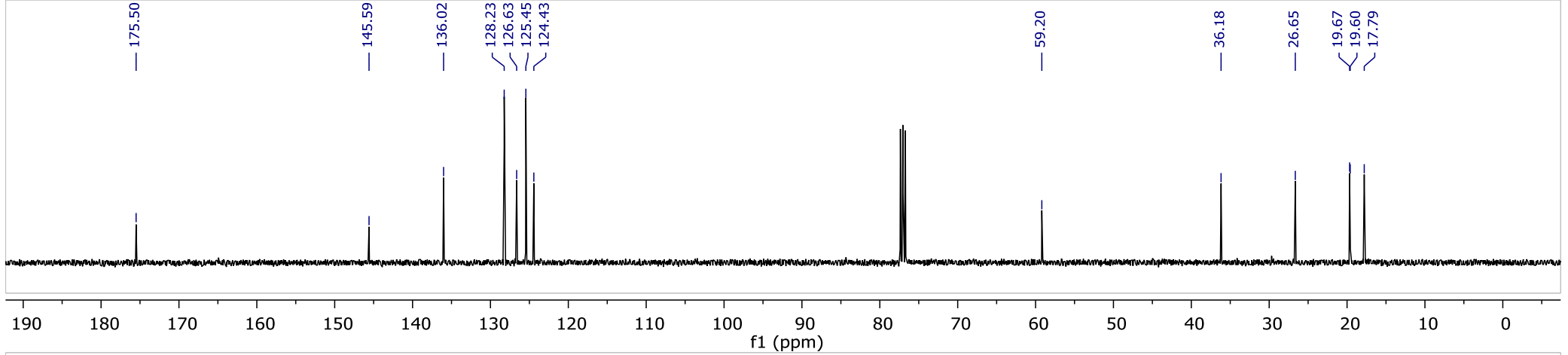
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125.45
124.43
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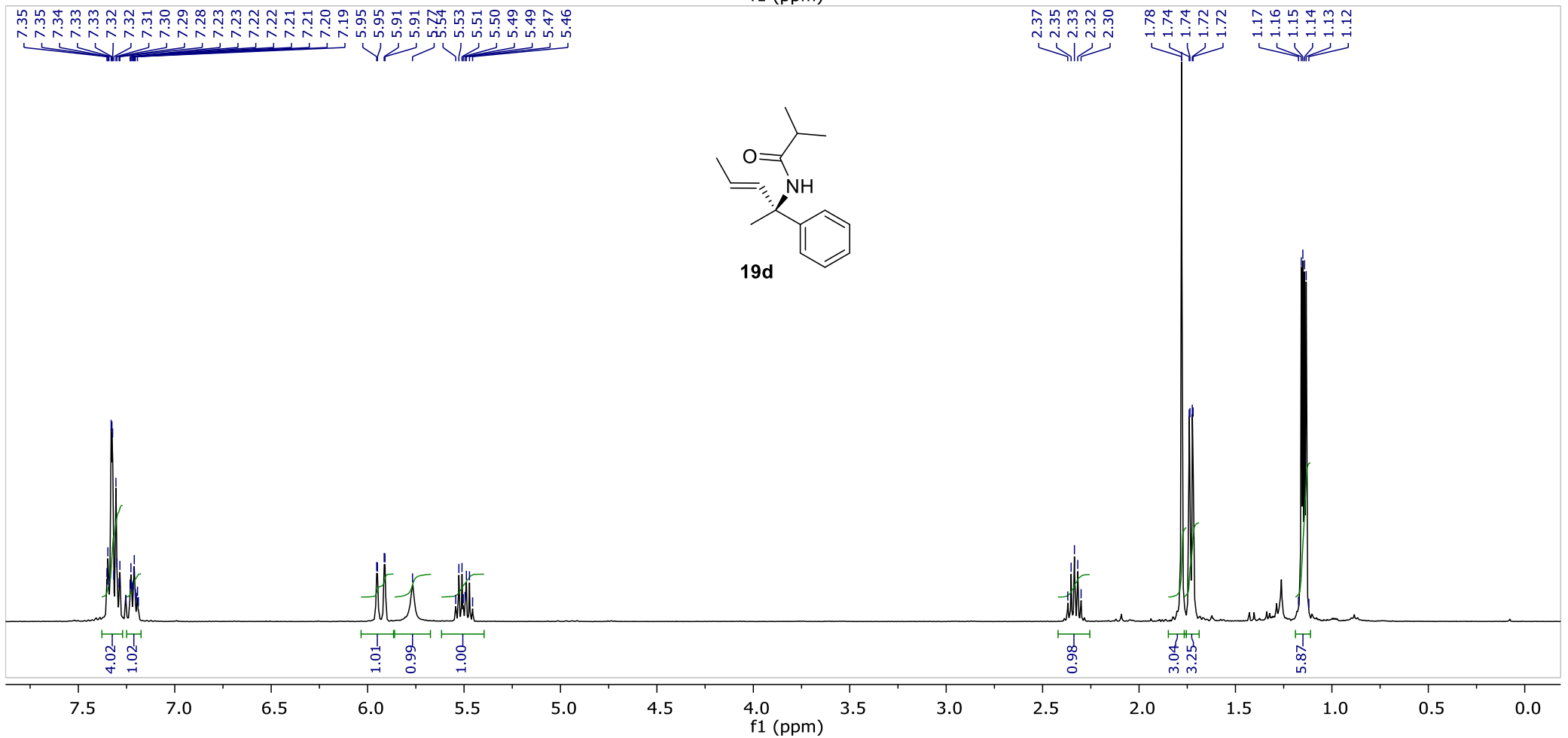
26.65
19.67
19.60
17.79
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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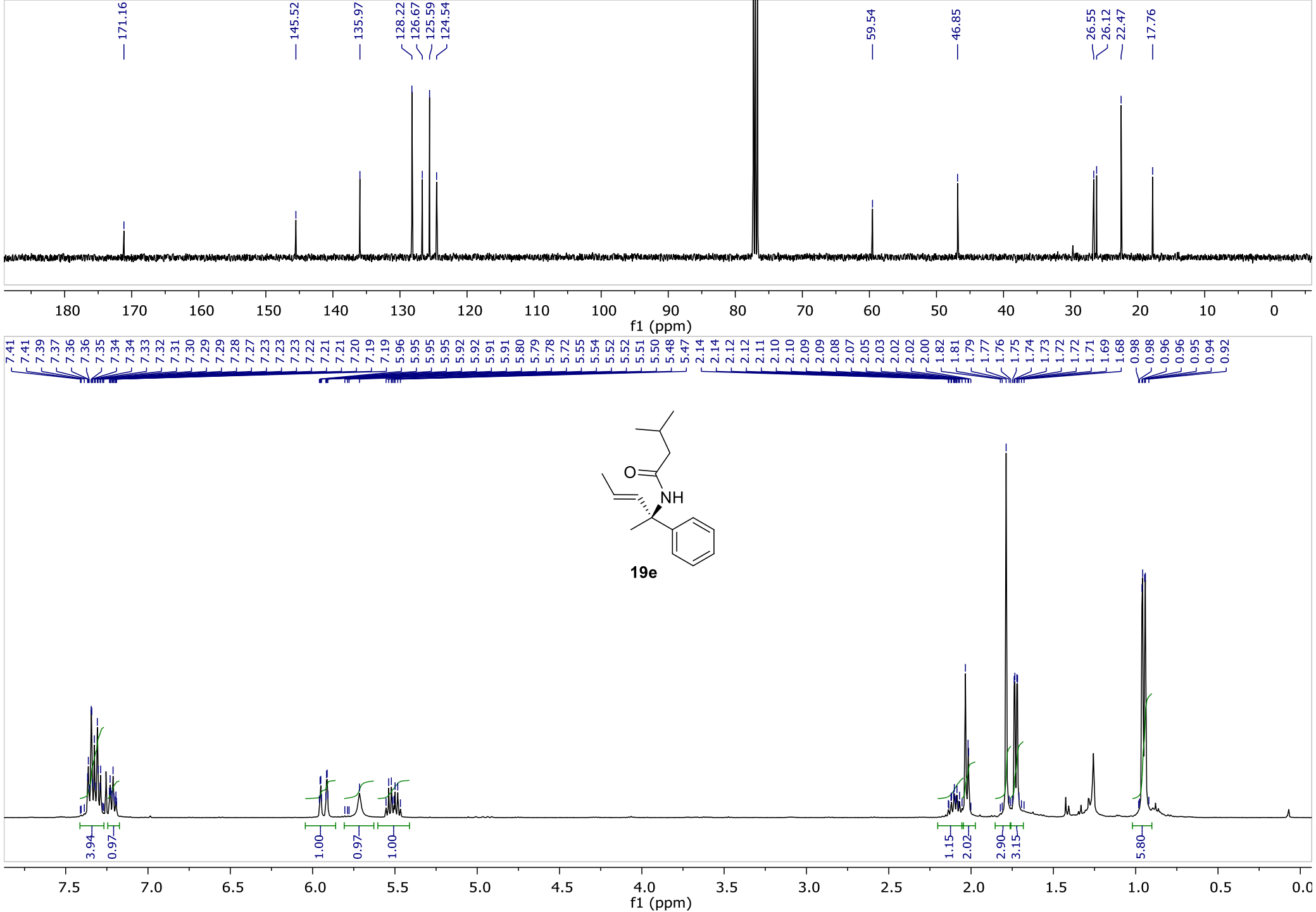
5.95 5.95 5.91 5.91 5.77 5.54 5.53 5.51 5.50 5.49 5.49 5.47 5.46
2.37 2.35 2.33 2.32 2.30
1.78 1.74 1.74 1.72 1.72
1.17 1.16 1.15 1.14 1.13 1.12
O
NH
19d
4.02 1.02 1.01 0.99 1.00 0.98 3.04 3.25 5.87
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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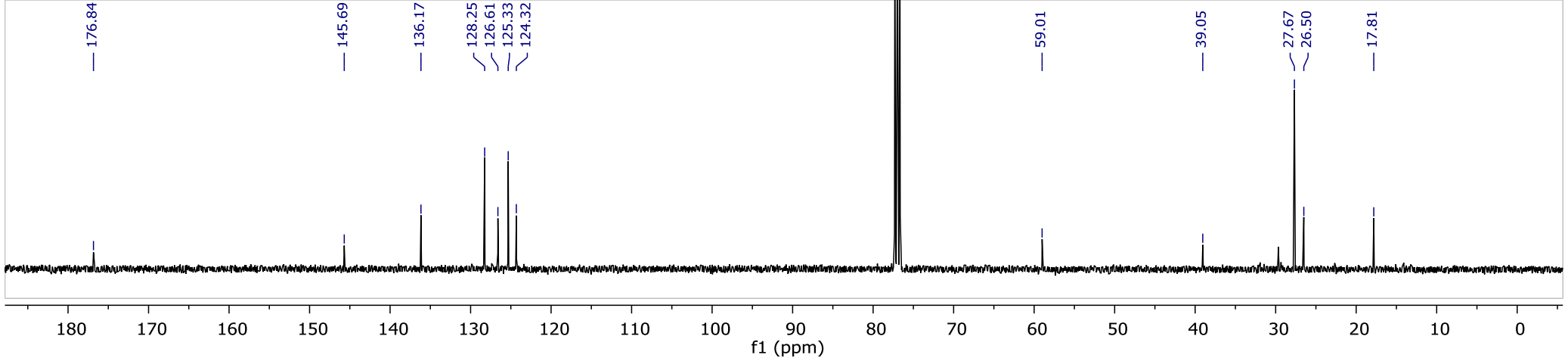
145.52
135.97
128.22
126.67
125.59
124.54
59.54
46.85
26.55
26.12
22.47
17.76
f1 (ppm)
7.41 7.41 7.39 7.37 7.36 7.36 7.35 7.34 7.34 7.33 7.32 7.31 7.30 7.29 7.29 7.28 7.27 7.23 7.23 7.23 7.22 7.21 7.21 7.20 7.19 7.19 5.96 5.95 5.95 5.95 5.92 5.92 5.91 5.91 5.80 5.79 5.78 5.72 5.55 5.54 5.52 5.52 5.51 5.50 5.48 5.47
2.14 2.14 2.12 2.12 2.11 2.10 2.10 2.09 2.09 2.08 2.07 2.05 2.03 2.02 2.02 2.00 1.82 1.81 1.79 1.77 1.76 1.75 1.74 1.73 1.72 1.72 1.71 1.69 1.68 0.98 0.98 0.96 0.96 0.95 0.94 0.92
O
NH
19e
3.94
0.97
1.00
0.97
1.00
1.15
2.02
2.90
3.15
5.80
f1 (ppm)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136.17
128.25
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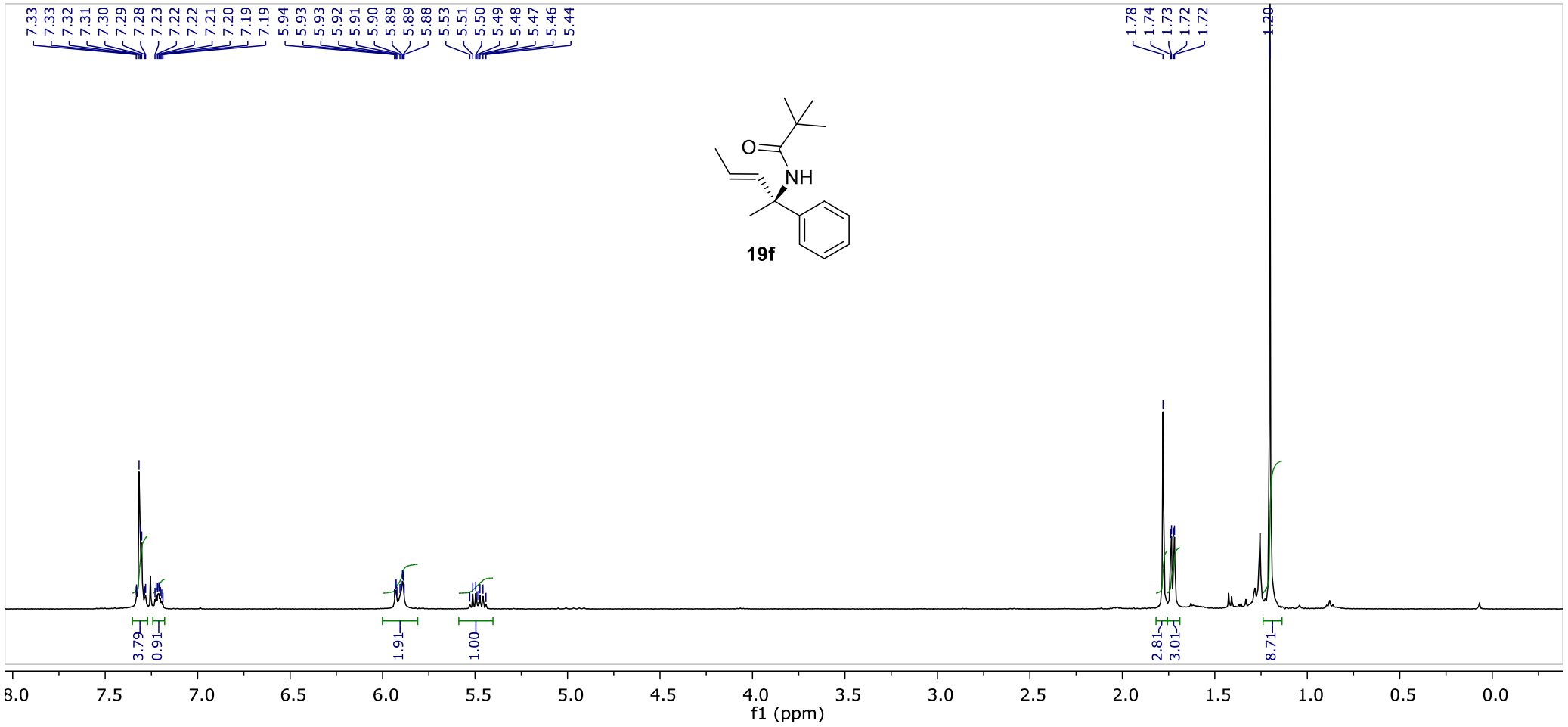
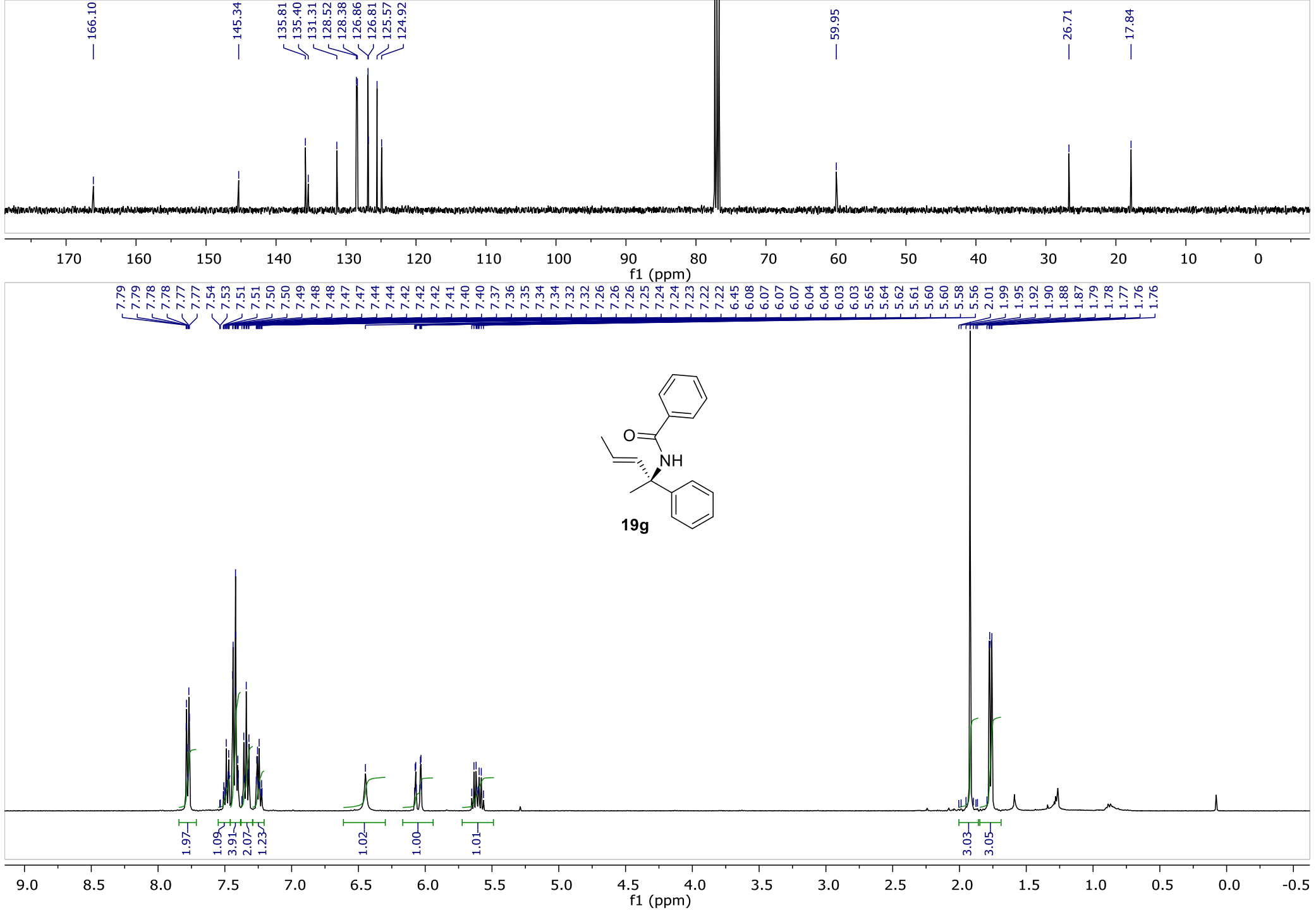
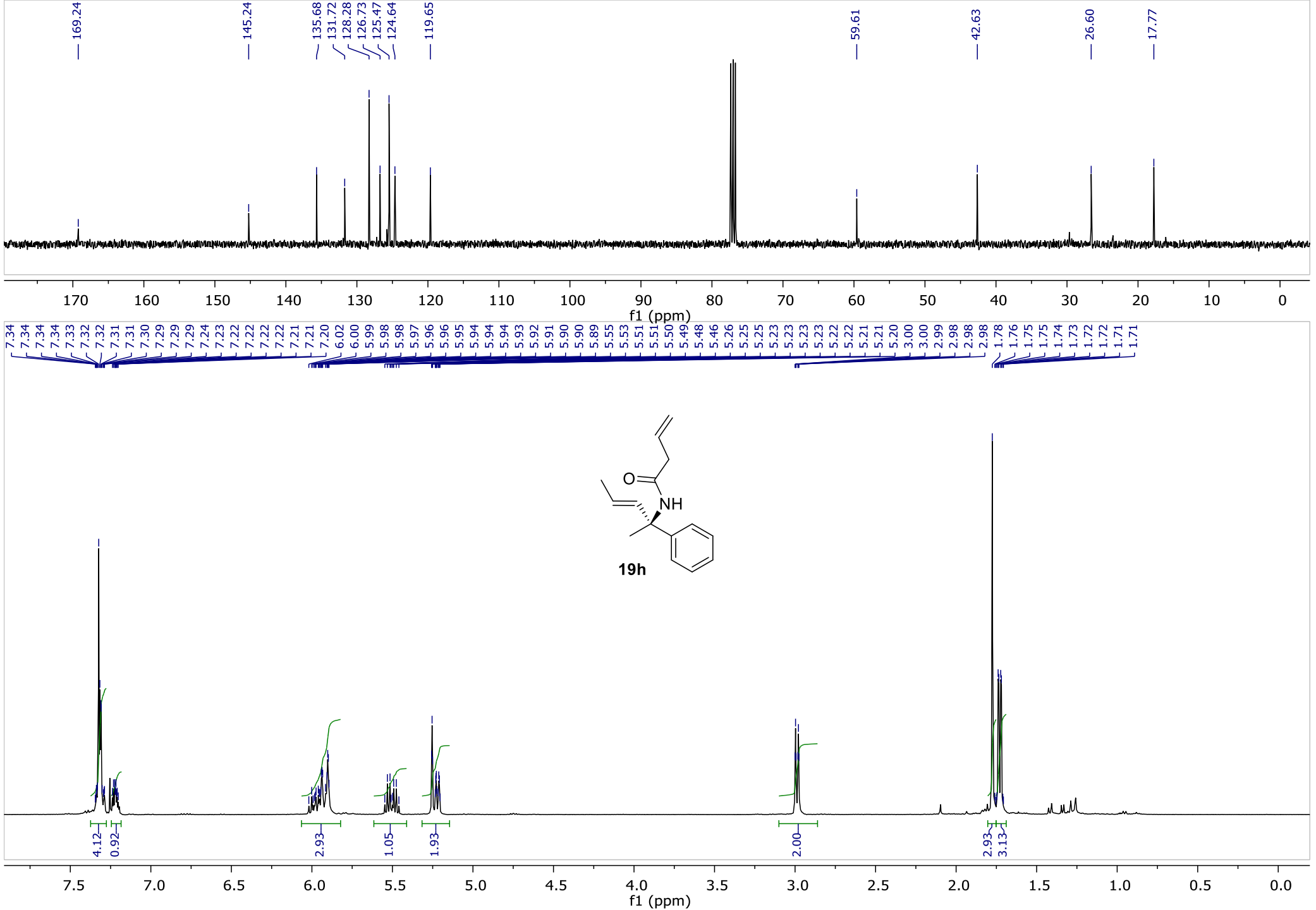
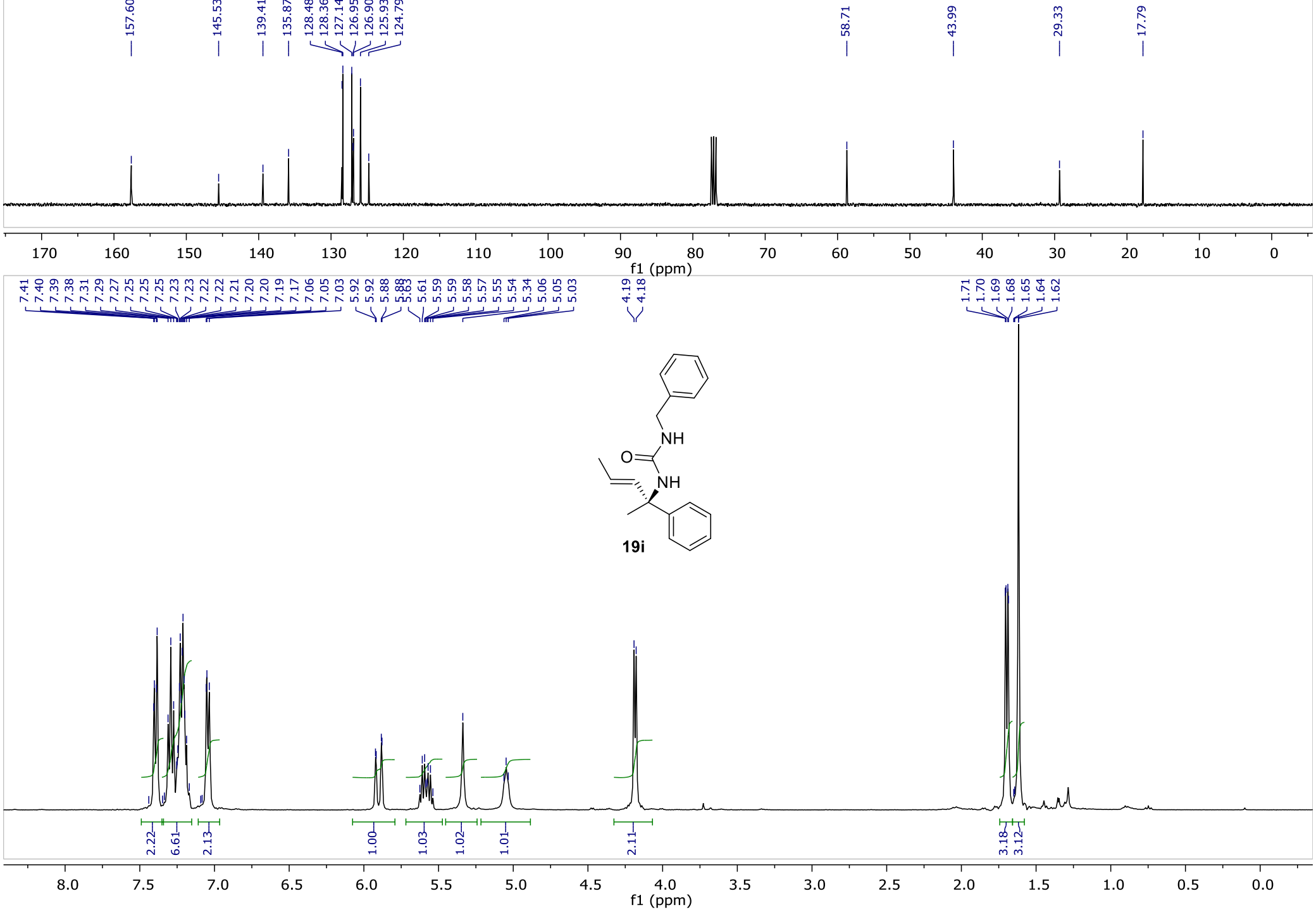
125.33
124.32
59.01
39.05
27.67
26.50
17.81
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.33
7.33
7.32
7.31
7.30
7.29
7.28
7.23
7.22
7.22
7.21
7.20
7.19
7.19
5.94
5.93
5.93
5.92
5.91
5.90
5.89
5.89
5.88
5.53
5.51
5.50
5.49
5.48
5.47
5.46
5.44
1.78
1.74
1.73
1.72
1.72
1.20
O
NH
19f
3.79
0.91
1.91
1.00
2.81
3.01
8.71
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
f1 (ppm)


166.10
145.34
135.81
135.40
131.31
128.52
128.38
126.86
126.81
125.57
124.92
59.95
26.71
17.84
f1 (ppm)
O
NH
19g
1.97
1.09
3.91
2.07
1.23
1.02
1.00
1.01
3.03
3.05
f1 (ppm)


169.24
145.24
135.68
131.72
128.28
126.73
125.47
124.64
119.65
59.61
42.63
26.60
17.77
f1 (ppm)
O
NH
19h
4.12
0.92
2.93
1.05
1.93
2.00
2.93
3.13
f1 (ppm)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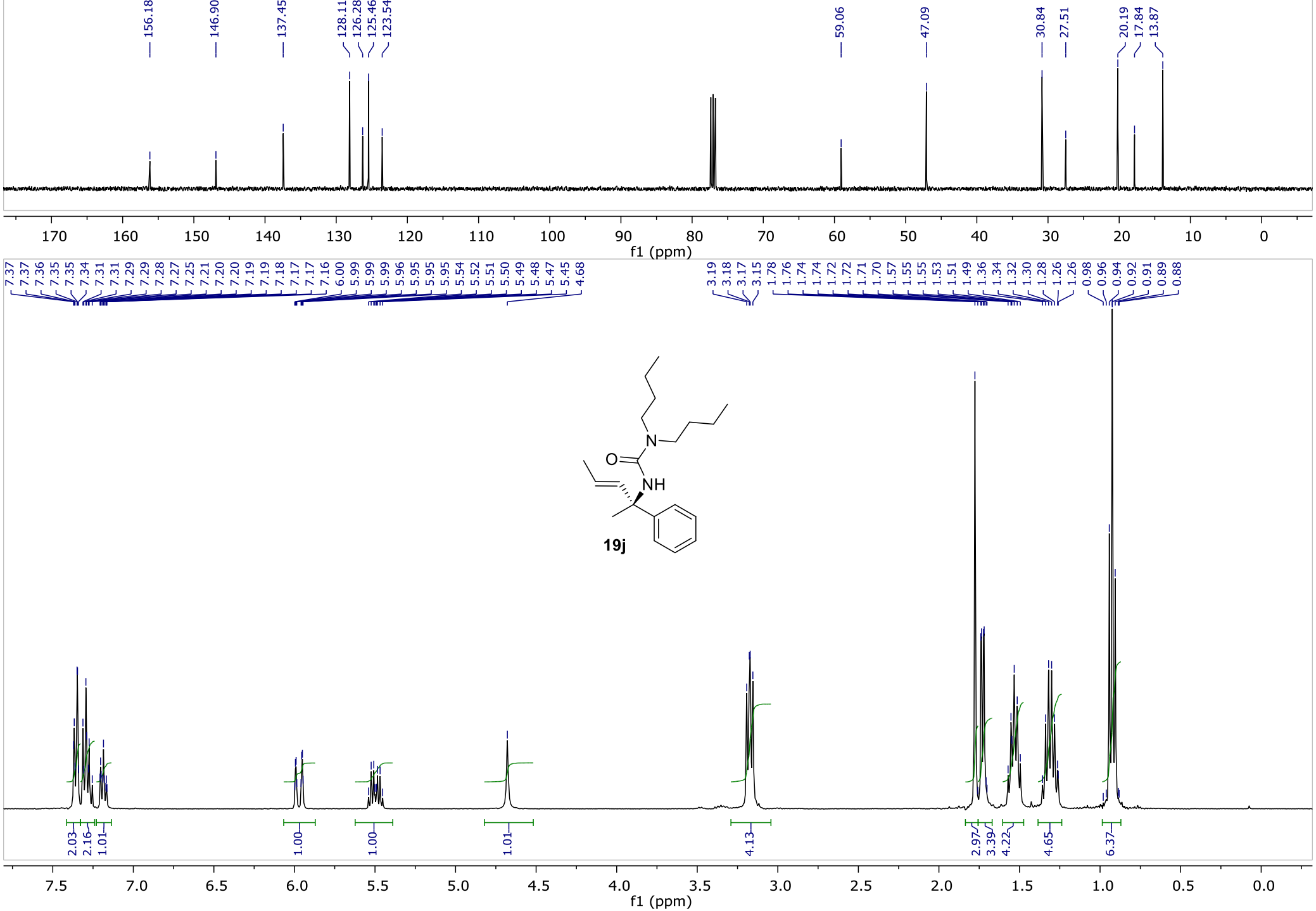
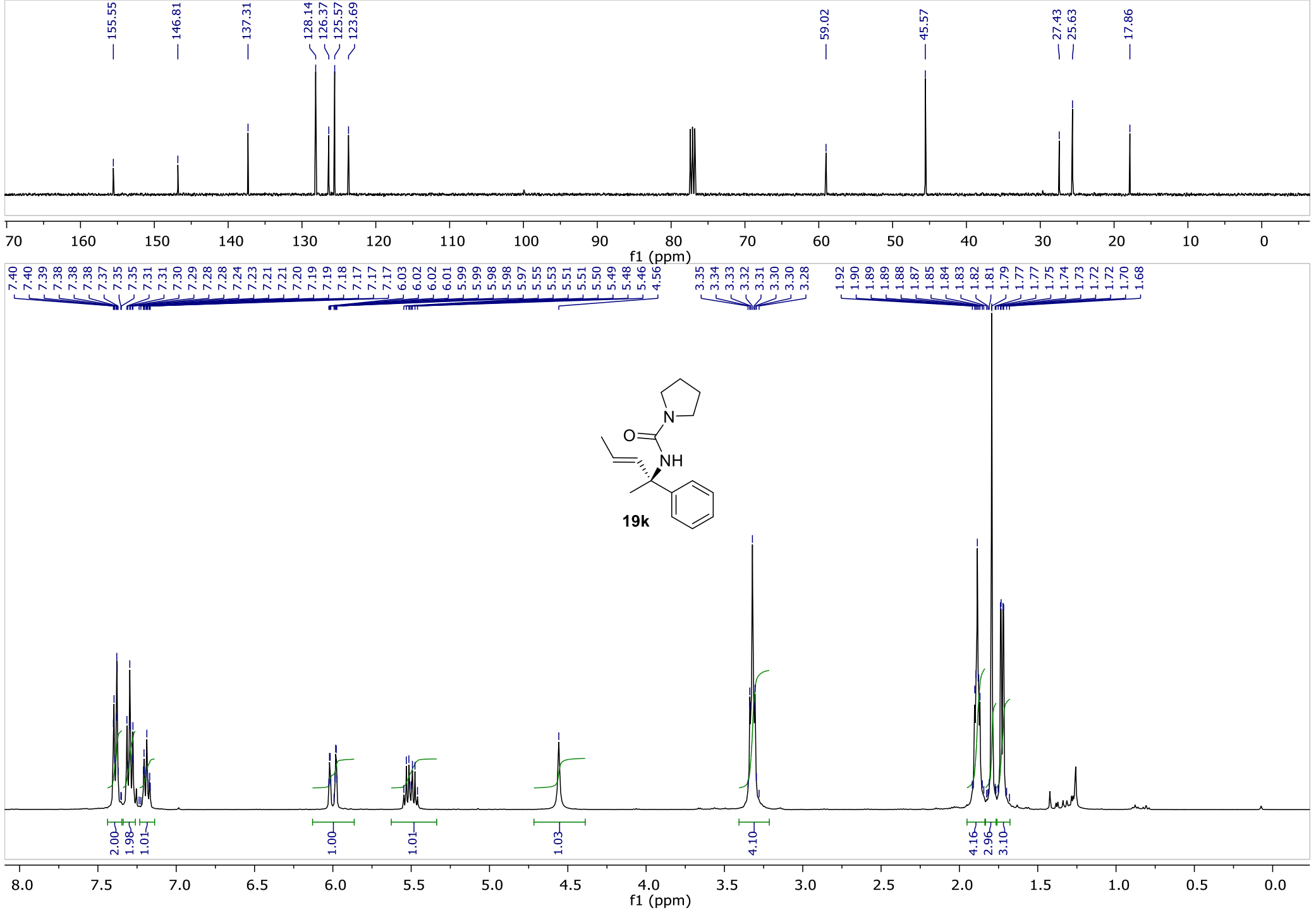
145.53
139.41
135.87
128.48
128.36
127.14
126.95
126.90
125.93
124.79
58.71
43.99
29.33
17.79
f1 (ppm)
7.41 7.40 7.39 7.38 7.31 7.29 7.27 7.25 7.25 7.25 7.23 7.23 7.22 7.22 7.21 7.20 7.20 7.19 7.17 7.06 7.05 7.03
5.92 5.92 5.88 5.61 5.59 5.59 5.58 5.57 5.55 5.54 5.34 5.06 5.05 5.03
4.19 4.18
1.71 1.70 1.69 1.68 1.65 1.64 1.62
NH
O
NH
19i
2.22 6.61 2.13 1.00 1.03 1.02 1.01 2.11 3.18 3.12
f1 (ppm)


156.18
146.90
137.45
128.11
126.28
125.46
123.54
59.06
47.09
30.84
27.51
20.19
17.84
13.87
f1 (ppm)
19j
f1 (ppm)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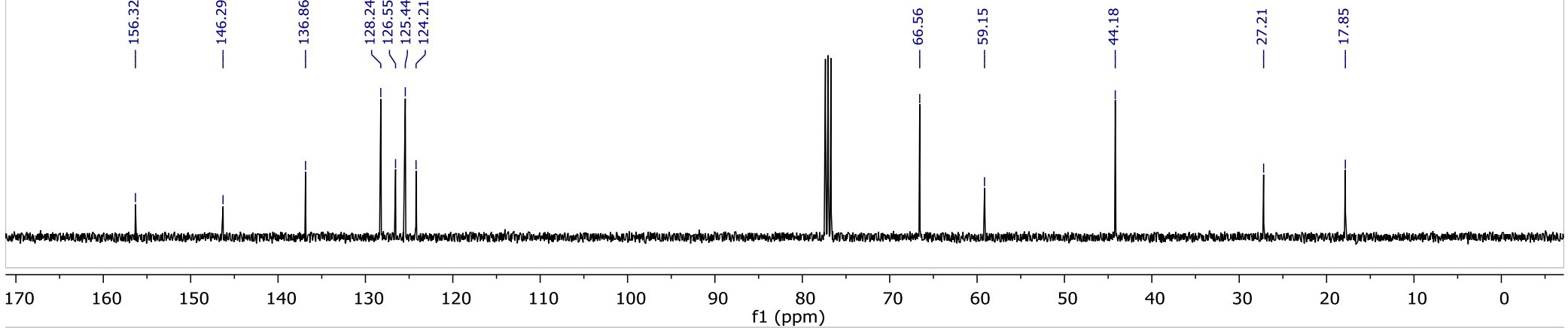
137.31
128.14
126.37
125.57
123.69
59.02
45.57
27.43
25.63
17.86
f1 (ppm)
7.40 7.40 7.39 7.38 7.38 7.38 7.37 7.35 7.35 7.31 7.31 7.30 7.29 7.28 7.28 7.24 7.23 7.21 7.21 7.20 7.19 7.19 7.18 7.17 7.17 7.17 6.03 6.02 6.02 6.01 5.99 5.99 5.98 5.98 5.97 5.55 5.53 5.51 5.51 5.50 5.49 5.48 5.46 4.56
3.35 3.34 3.33 3.32 3.31 3.30 3.30 3.28
1.92 1.90 1.89 1.89 1.88 1.87 1.85 1.84 1.83 1.82 1.81 1.79 1.77 1.77 1.75 1.74 1.73 1.72 1.70 1.68
N
O
NH
19k
2.00
1.98
1.01
1.00
1.01
1.03
4.10
4.16
2.96
3.10
f1 (ppm)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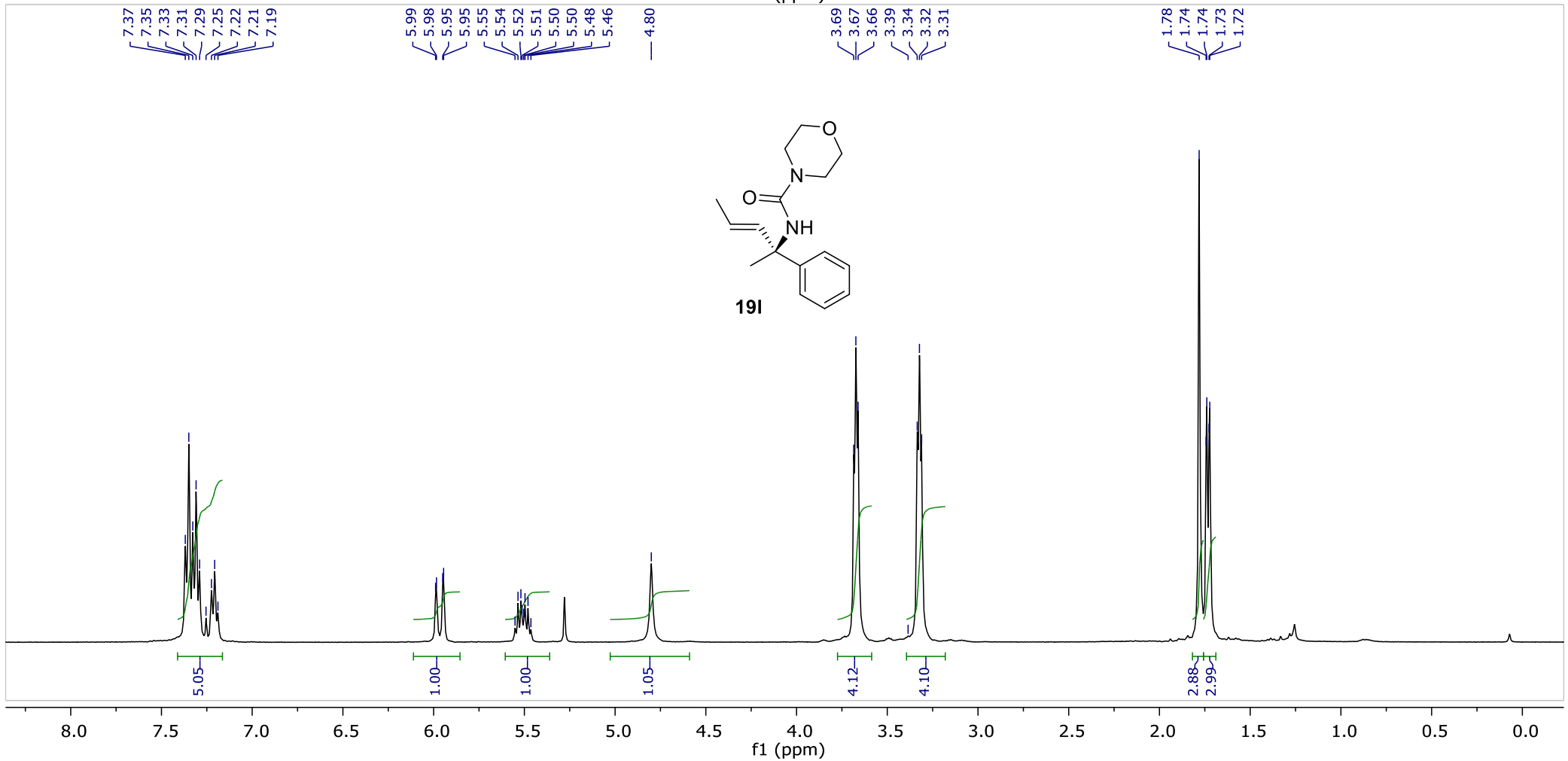
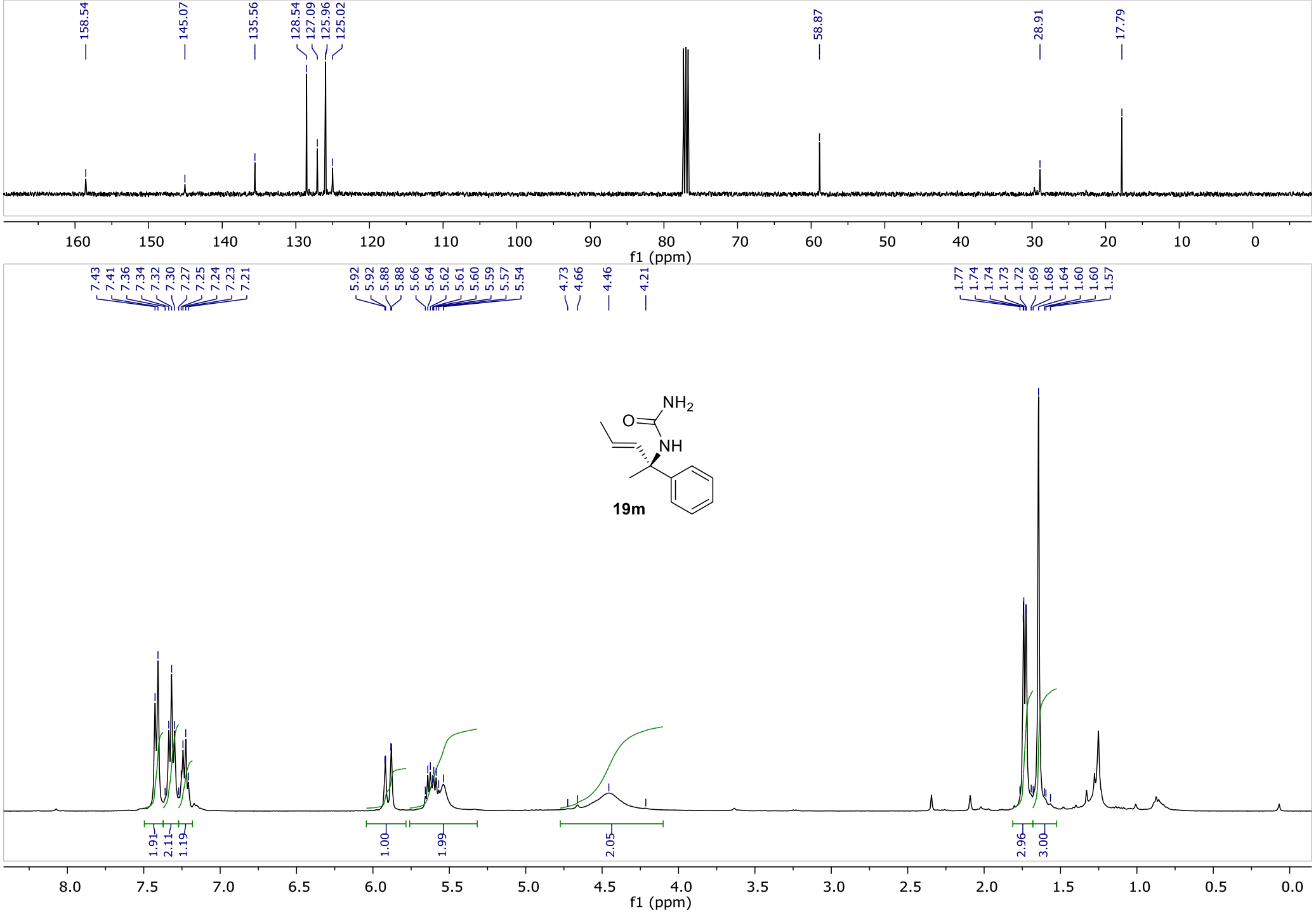
136.86
128.24
126.55
125.44
124.21
66.56
59.15
44.18
27.21
17.85
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.37
7.35
7.33
7.31
7.29
7.25
7.22
7.21
7.19
5.99
5.98
5.95
5.95
5.55
5.54
5.52
5.51
5.50
5.50
5.48
5.46
4.80
3.69
3.67
3.66
3.39
3.34
3.32
3.31
1.78
1.74
1.74
1.73
1.72
O
N
O
NH
19l
5.05
1.00
1.00
1.05
4.12
4.10
2.88
2.99
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
f1 (ppm)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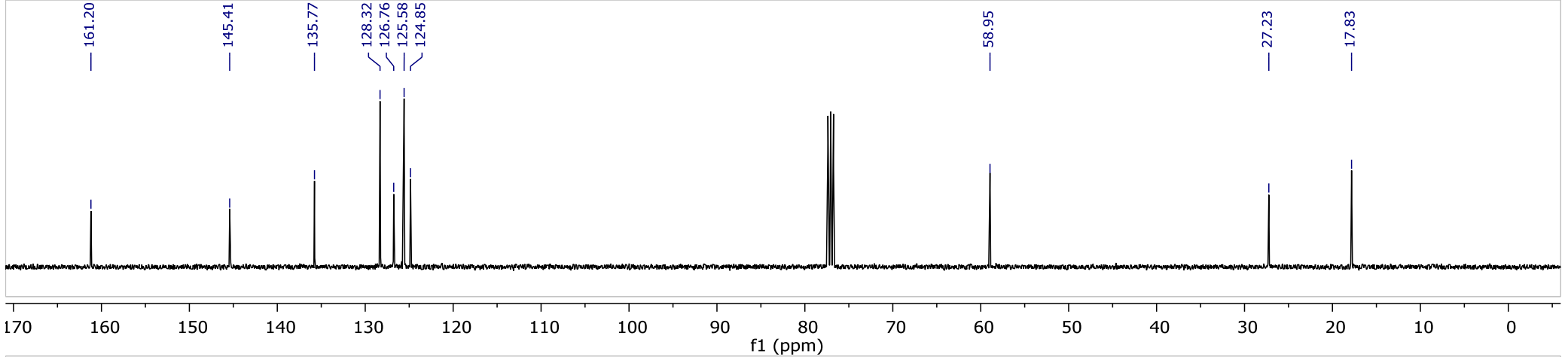
135.56
128.54
127.09
125.96
125.02
58.87
28.91
17.79
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)
7.43
7.41
7.36
7.34
7.32
7.30
7.27
7.25
7.24
7.23
7.21
5.92
5.92
5.88
5.88
5.66
5.64
5.62
5.61
5.60
5.59
5.57
5.54
4.73
4.66
4.46
4.21
1.77
1.74
1.74
1.73
1.72
1.69
1.68
1.64
1.60
1.60
1.57
NH2
O
NH
19m
1.91
2.11
1.19
1.00
1.99
2.05
2.96
3.00
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
f1 (ppm)


161.20
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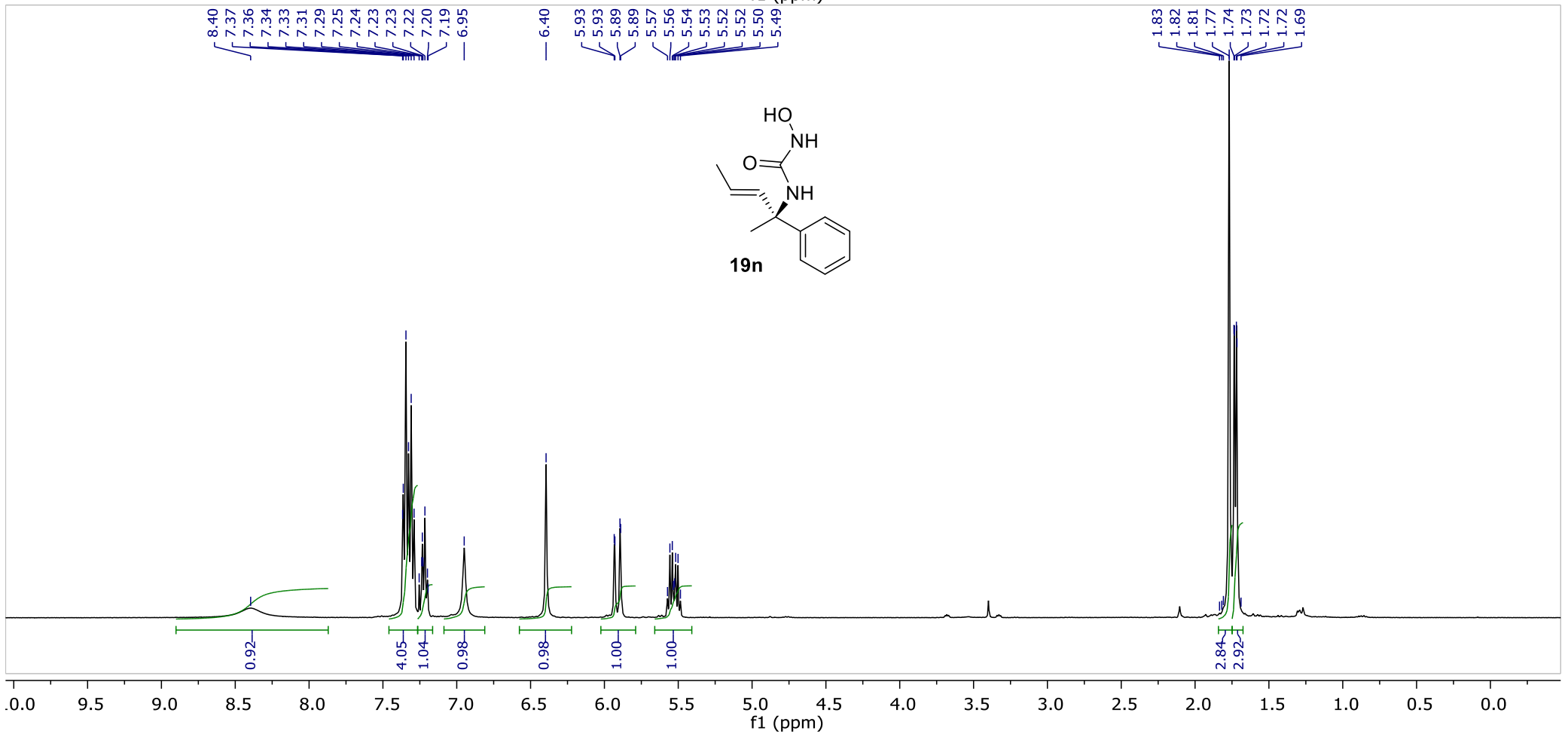
126.76
125.58
124.85
58.95
27.23
17.83
f1 (ppm)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7.37
7.36
7.34
7.33
7.31
7.29
7.25
7.24
7.23
7.23
7.22
7.20
7.19
6.95
6.40
5.93
5.93
5.89
5.89
5.57
5.56
5.54
5.53
5.52
5.52
5.50
5.49
1.83
1.82
1.81
1.77
1.74
1.73
1.72
1.72
1.69
HO
NH
O
NH
19n
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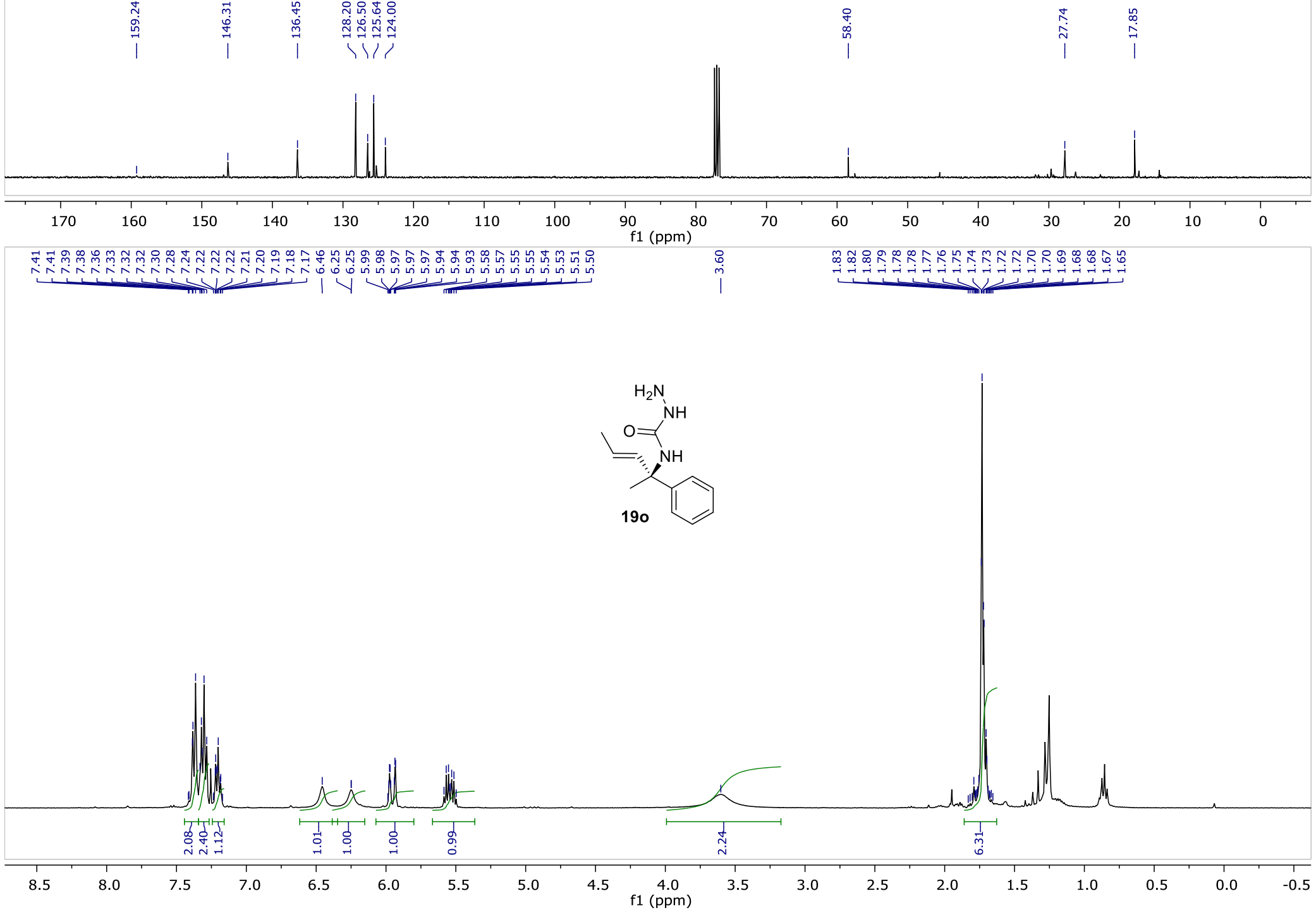
4.05
1.04
0.98
0.98
1.00
1.00
2.84
2.92
f1 (ppm)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126.50
125.64
124.00
58.40
27.74
17.85
f1 (ppm)
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7.41
7.39
7.38
7.36
7.33
7.32
7.32
7.30
7.28
7.24
7.22
7.22
7.22
7.21
7.20
7.19
7.18
7.17
6.46
6.25
6.25
5.99
5.98
5.97
5.97
5.97
5.94
5.94
5.93
5.58
5.57
5.55
5.55
5.54
5.53
5.51
5.50
3.60
1.83
1.82
1.80
1.79
1.78
1.78
1.77
1.76
1.75
1.74
1.73
1.72
1.72
1.70
1.70
1.69
1.68
1.68
1.67
1.65
H2N
NH
O
NH
19o
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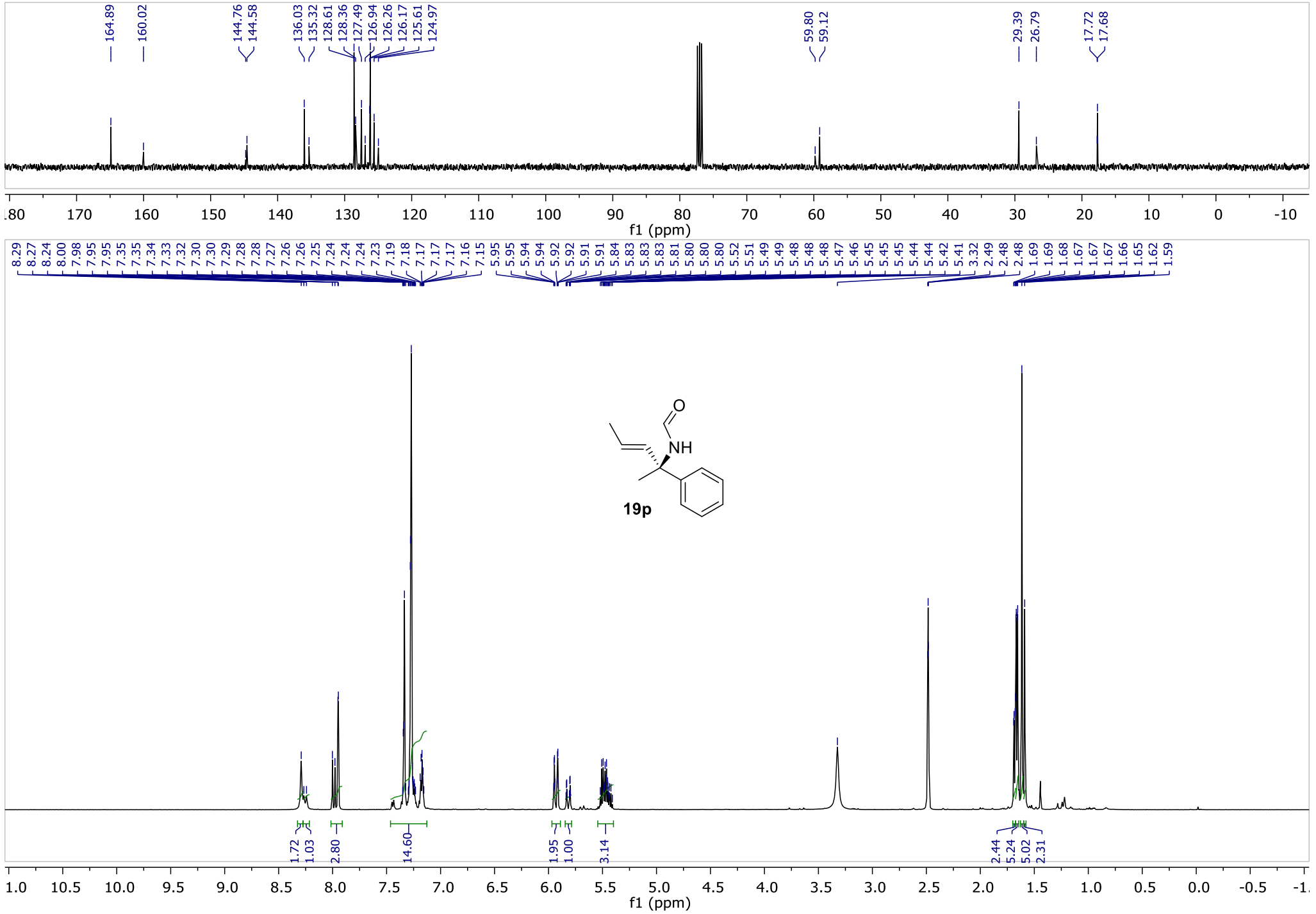
2.40
1.12
1.01
1.00
1.00
0.99
2.24
6.31
f1 (ppm)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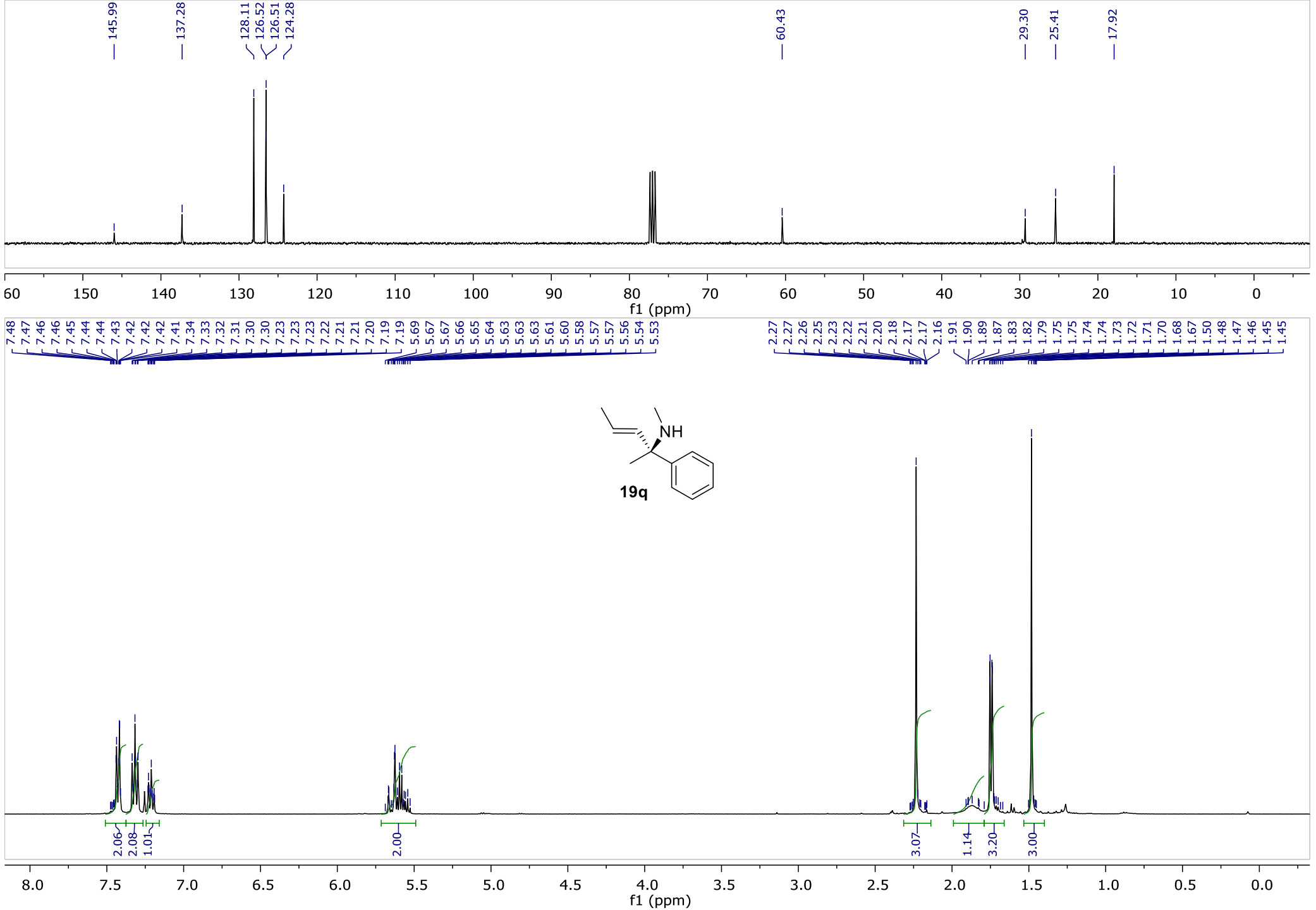
144.58
136.03
135.32
128.61
128.36
127.49
126.94
126.26
126.17
125.61
124.97
59.80
59.12
29.39
26.79
17.72
17.68
f1 (ppm)
O
NH
19p
1.72
1.03
2.80
14.60
1.95
1.00
3.14
2.44
5.24
5.02
2.31
f1 (ppm)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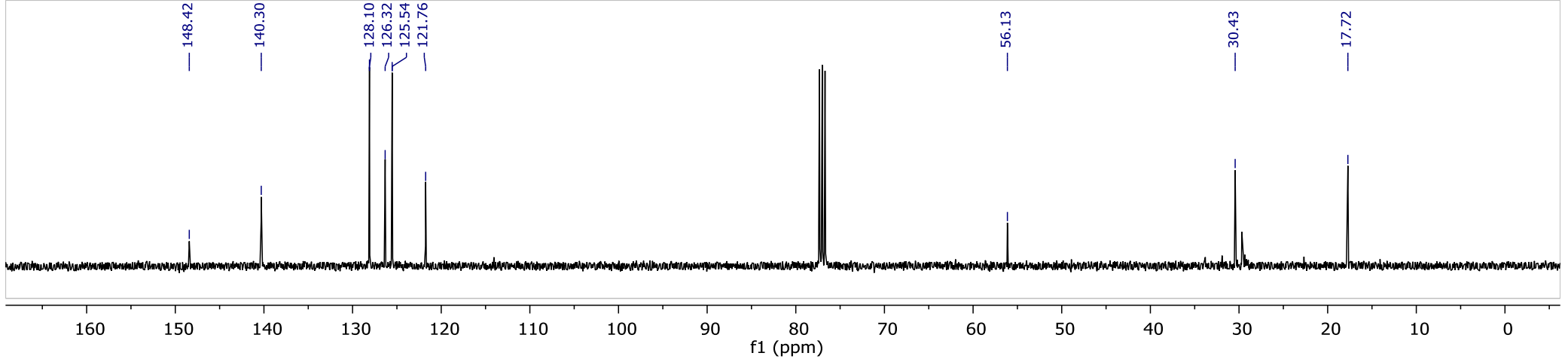
126.52
126.51
124.28
60.43
29.30
25.41
17.92
f1 (ppm)
NH
19q
2.06
2.08
1.01
2.00
3.07
1.14
3.20
3.00
f1 (ppm)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121.76
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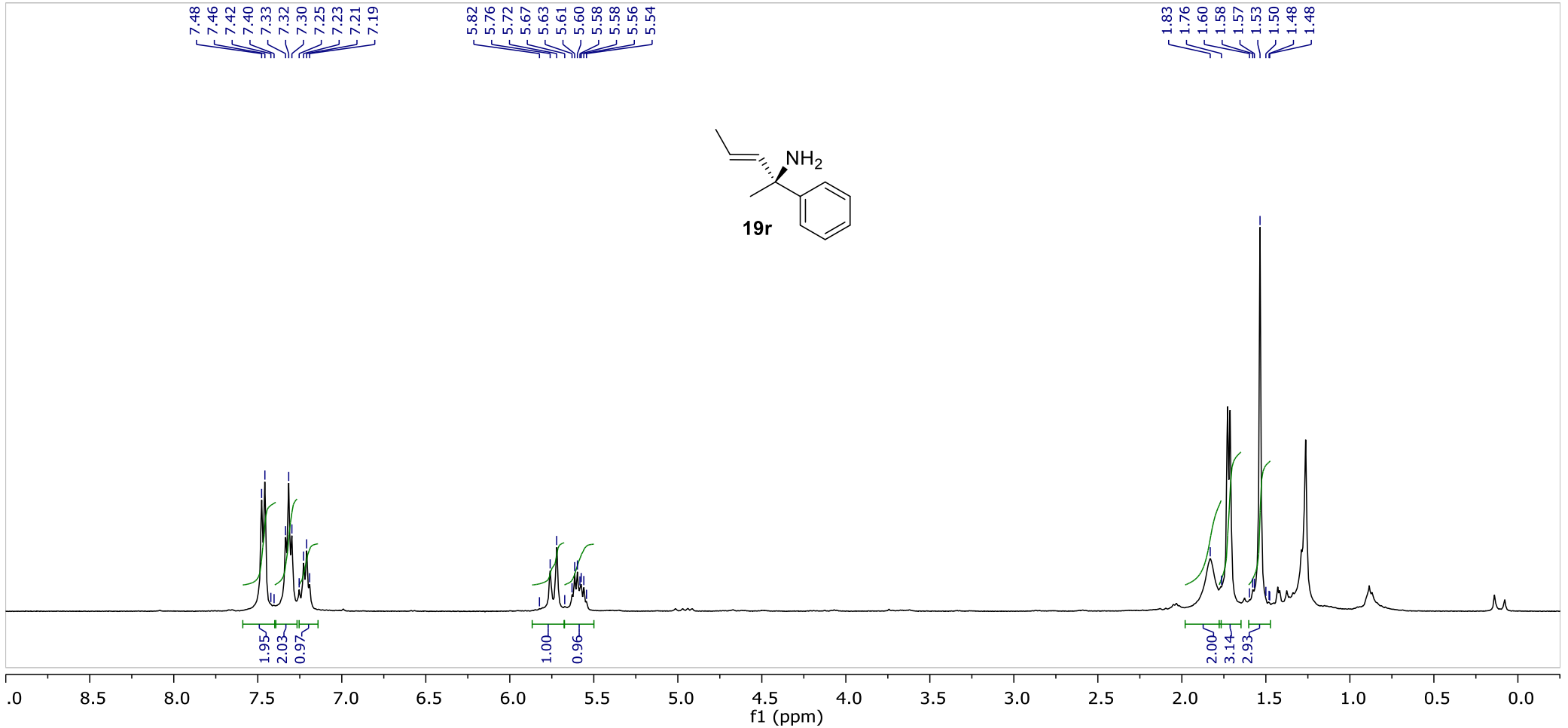
30.43
17.72
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.23
7.21
7.19
5.82
5.76
5.72
5.67
5.63
5.61
5.60
5.58
5.58
5.56
5.54
1.83
1.76
1.60
1.58
1.57
1.53
1.50
1.48
1.48
NH2
19r
1.95
2.03
0.97
1.00
0.96
2.00
3.14
2.93
.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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3.5
3.0
2.5
2.0
1.5
1.0
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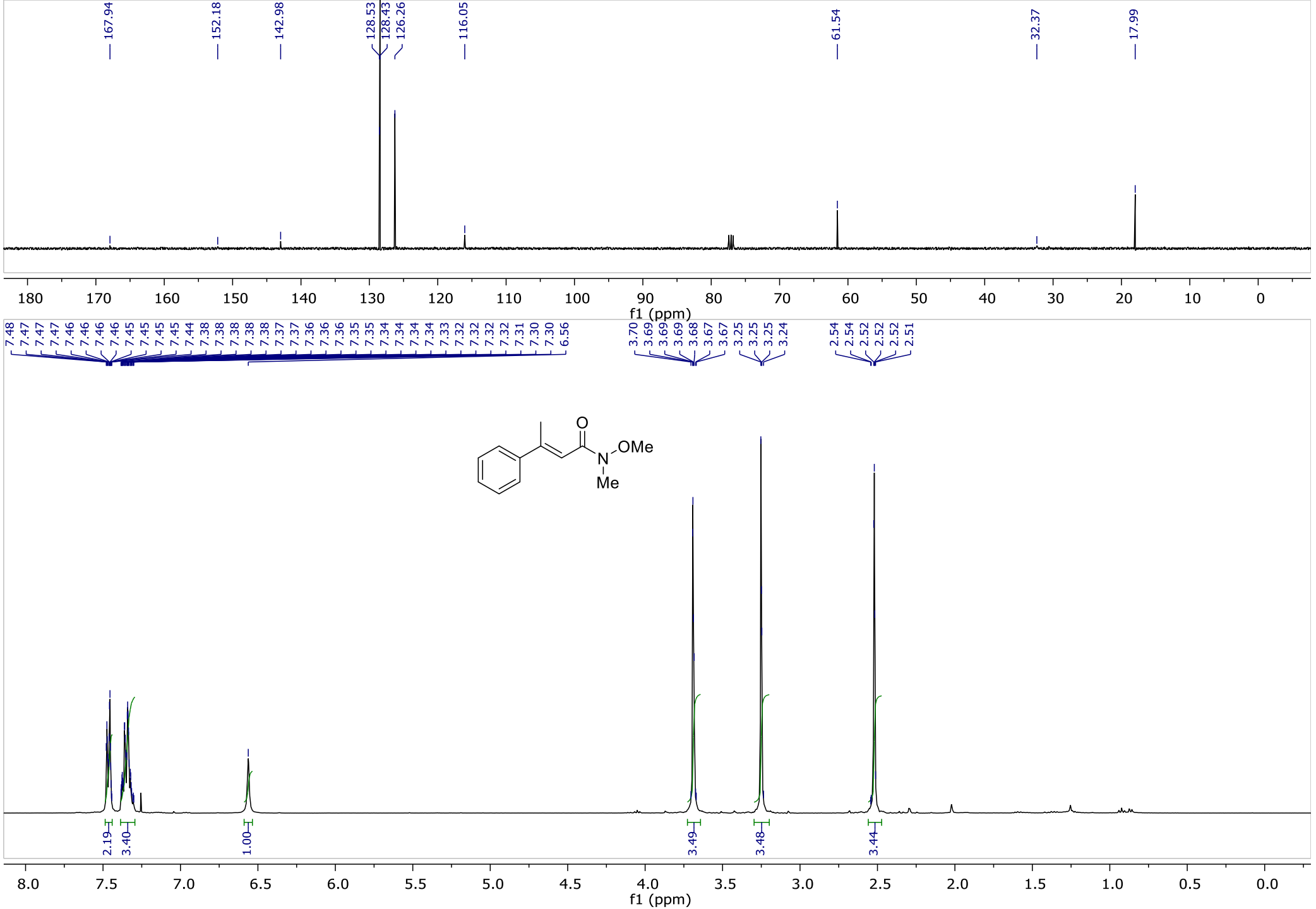
0.0
f1 (ppm)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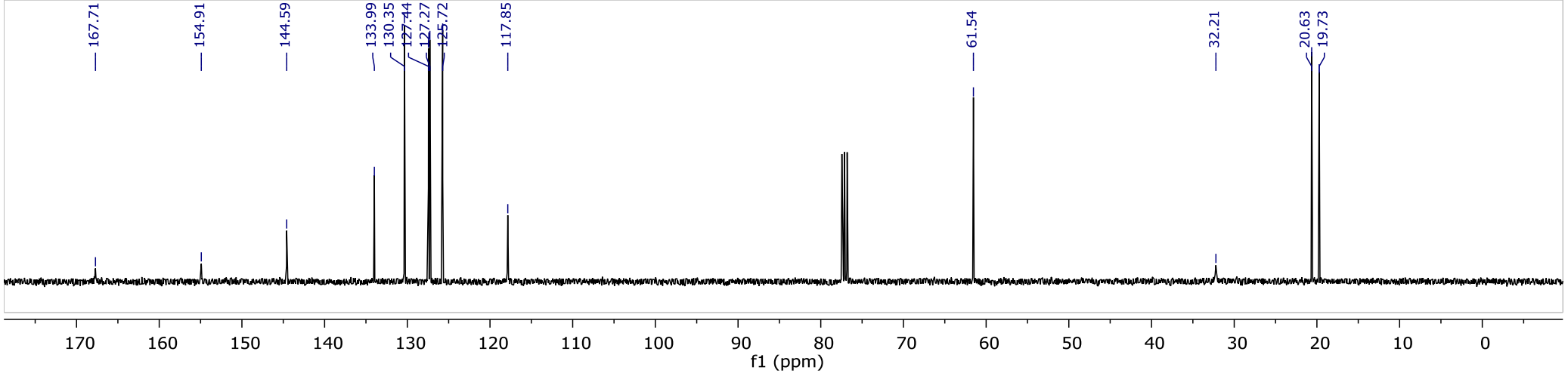
128.53
128.43
126.26
116.05
61.54
32.37
17.99
f1 (ppm)
7.48 7.47 7.47 7.47 7.46 7.46 7.46 7.45 7.45 7.45 7.45 7.44 7.38 7.38 7.38 7.38 7.37 7.37 7.36 7.36 7.36 7.35 7.35 7.34 7.34 7.34 7.34 7.33 7.32 7.32 7.32 7.32 7.31 7.30 7.30 6.56
3.70 3.69 3.69 3.69 3.68 3.67 3.67 3.25 3.25 3.25 3.24
2.54 2.54 2.52 2.52 2.52 2.51
O
N
OMe
Me
2.19
3.40
1.00
3.49
3.48
3.44
f1 (ppm)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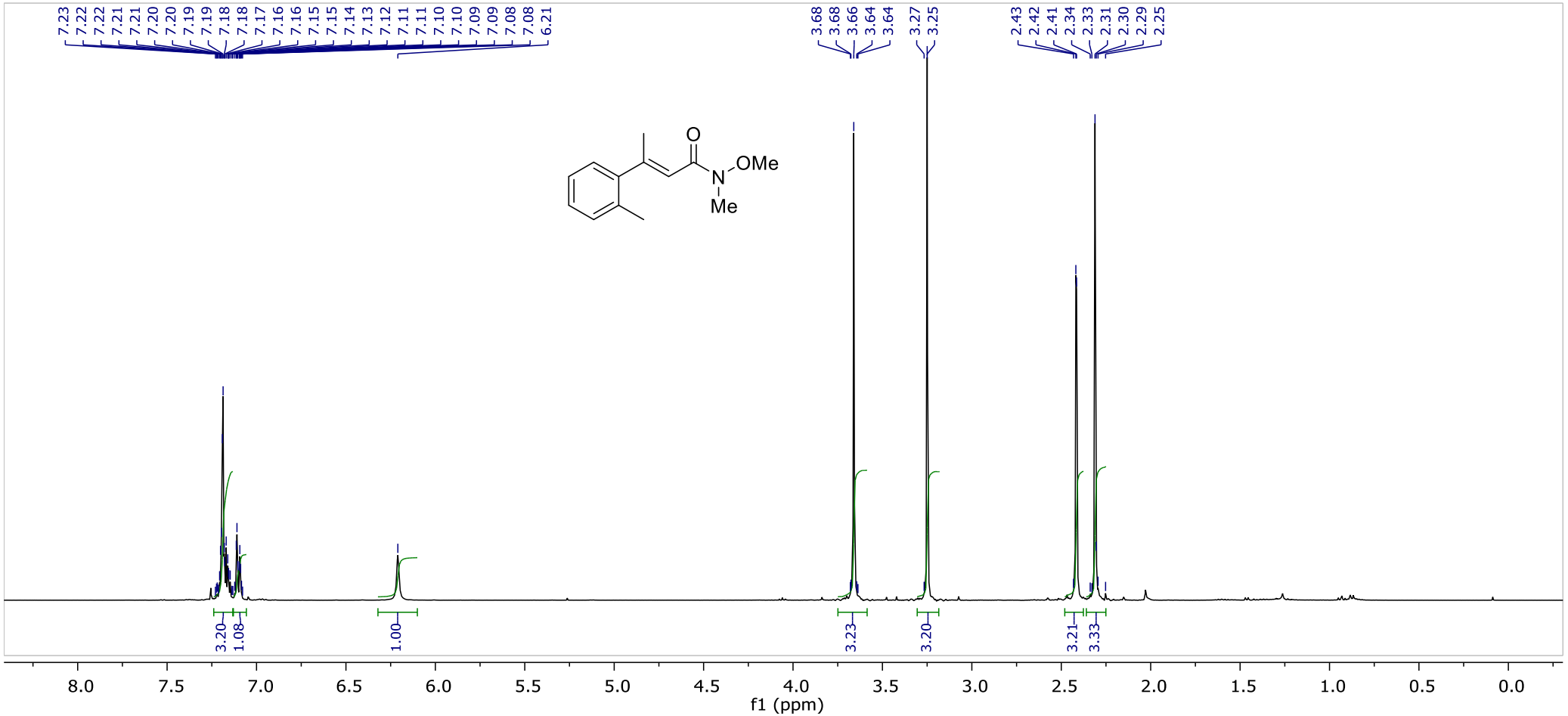
125.72
117.85
61.54
32.21
20.63
19.73
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.22
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7.18
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7.10
7.10
7.09
7.09
7.08
7.08
6.21
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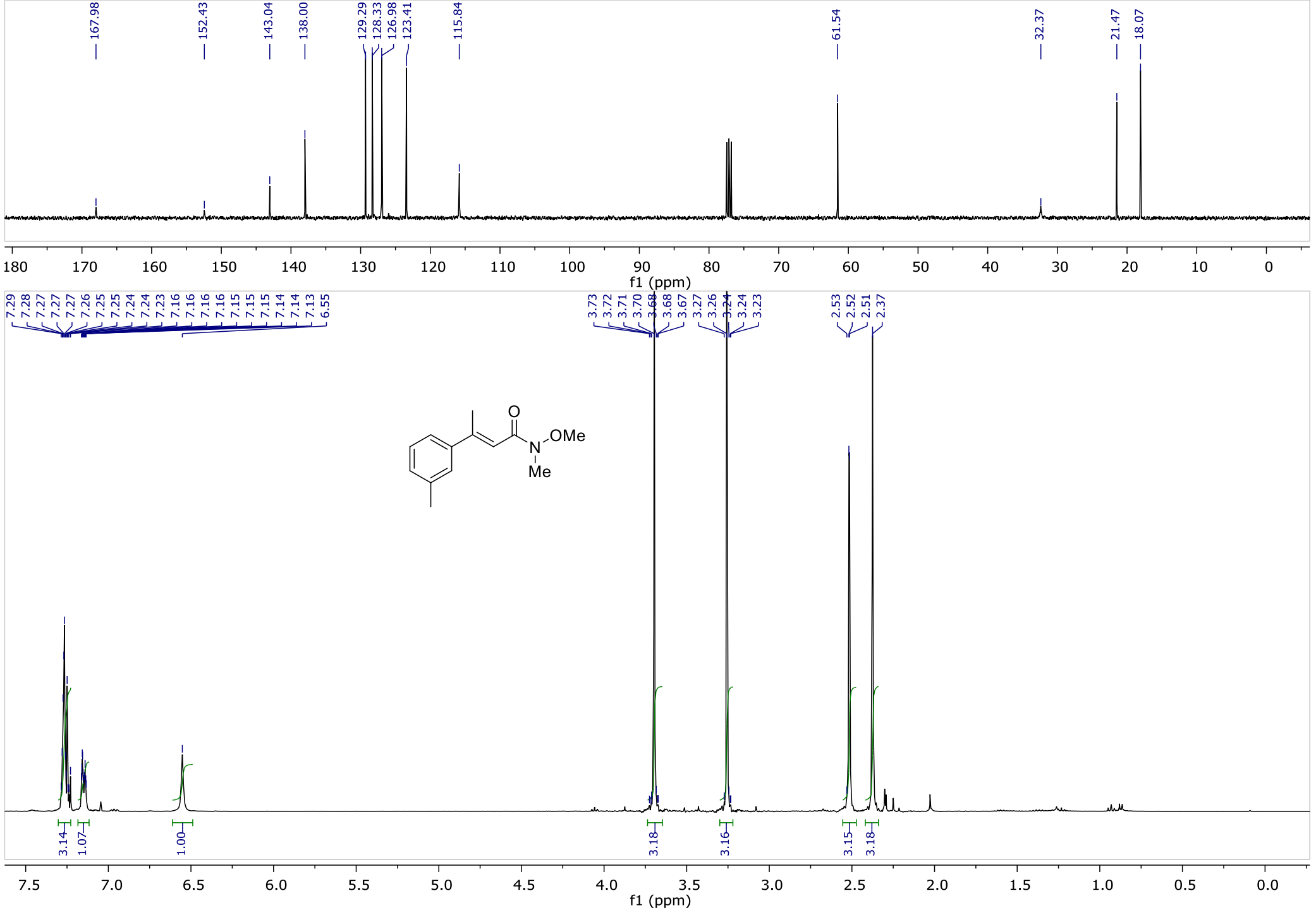
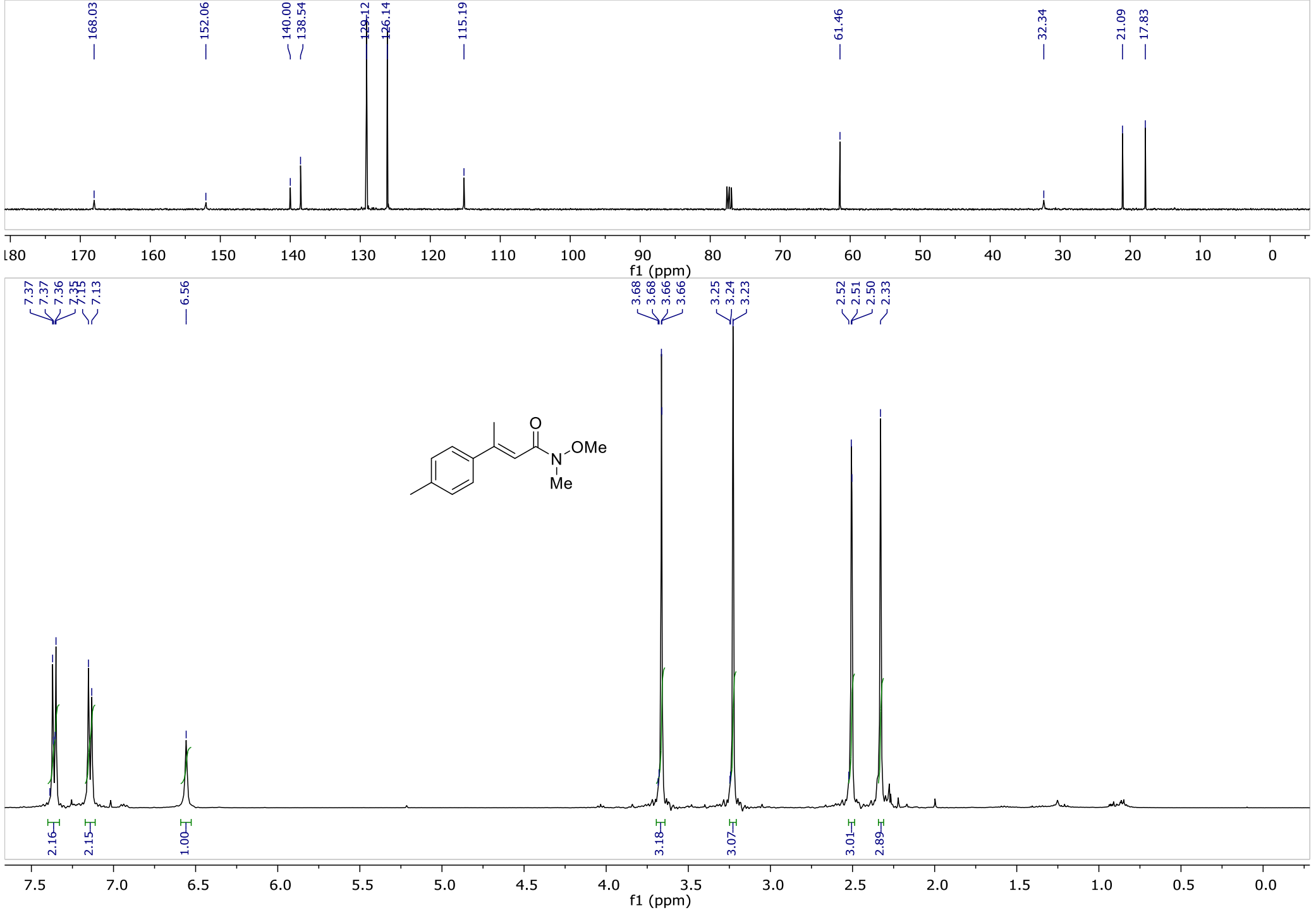
3.68
3.66
3.64
3.64
3.27
3.25
2.43
2.42
2.41
2.34
2.33
2.31
2.30
2.29
2.25
O
N
OMe
Me
3.20
1.08
1.00
3.23
3.20
3.21
3.33
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


167.98
152.43
143.04
138.00
129.29
128.33
126.98
123.41
115.84
61.54
32.37
21.47
18.07
f1 (ppm)
7.29
7.28
7.27
7.27
7.27
7.26
7.25
7.25
7.24
7.24
7.23
7.16
7.16
7.16
7.15
7.15
7.15
7.14
7.14
7.13
6.55
3.73
3.72
3.71
3.70
3.68
3.68
3.67
3.27
3.26
3.24
3.24
3.23
2.53
2.52
2.51
2.37
O
N
OMe
Me
3.14
1.07
1.00
3.18
3.16
3.15
3.18
f1 (ppm)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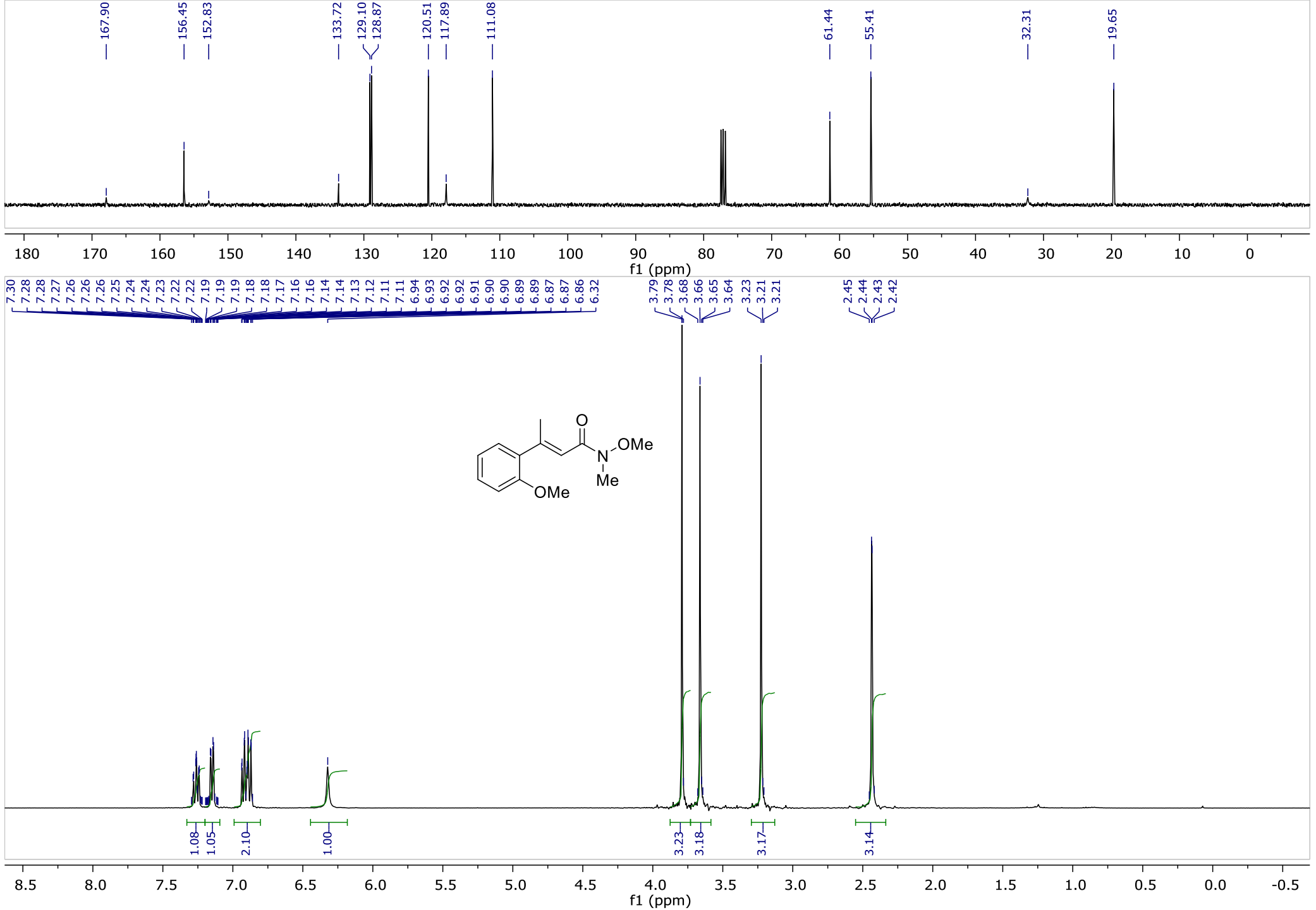
152.06
140.00
138.54
129.12
126.14
115.19
61.46
32.34
21.09
17.83
f1 (ppm)
7.37
7.37
7.36
7.35
7.15
7.13
6.56
3.68
3.68
3.66
3.66
3.25
3.24
3.23
2.52
2.51
2.50
2.33
O
N
OMe
Me
2.16
2.15
1.00
3.18
3.07
3.01
2.89
f1 (ppm)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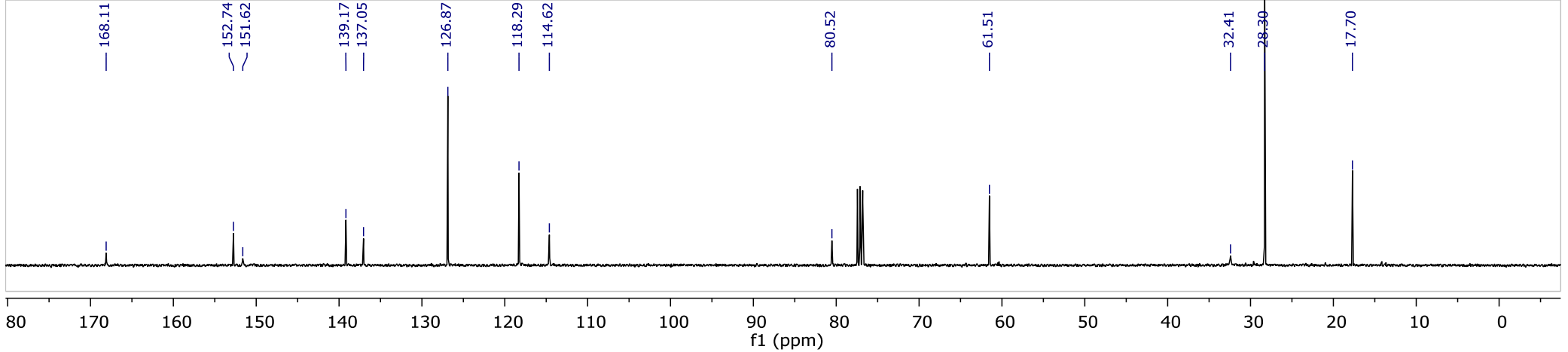
156.45
152.83
133.72
129.10
128.87
120.51
117.89
111.08
61.44
55.41
32.31
19.65
f1 (ppm)
7.30 7.28 7.28 7.27 7.26 7.26 7.26 7.25 7.24 7.24 7.23 7.22 7.22 7.19 7.19 7.19 7.18 7.18 7.17 7.16 7.16 7.14 7.14 7.13 7.12 7.11 7.11 6.94 6.93 6.92 6.92 6.91 6.90 6.90 6.89 6.89 6.87 6.87 6.86 6.32
3.79 3.78 3.68 3.66 3.65 3.64 3.23 3.21 3.21
2.45 2.44 2.43 2.42
O
N
OMe
Me
OMe
1.08 1.05 2.10 1.00 3.23 3.18 3.17 3.14
f1 (ppm)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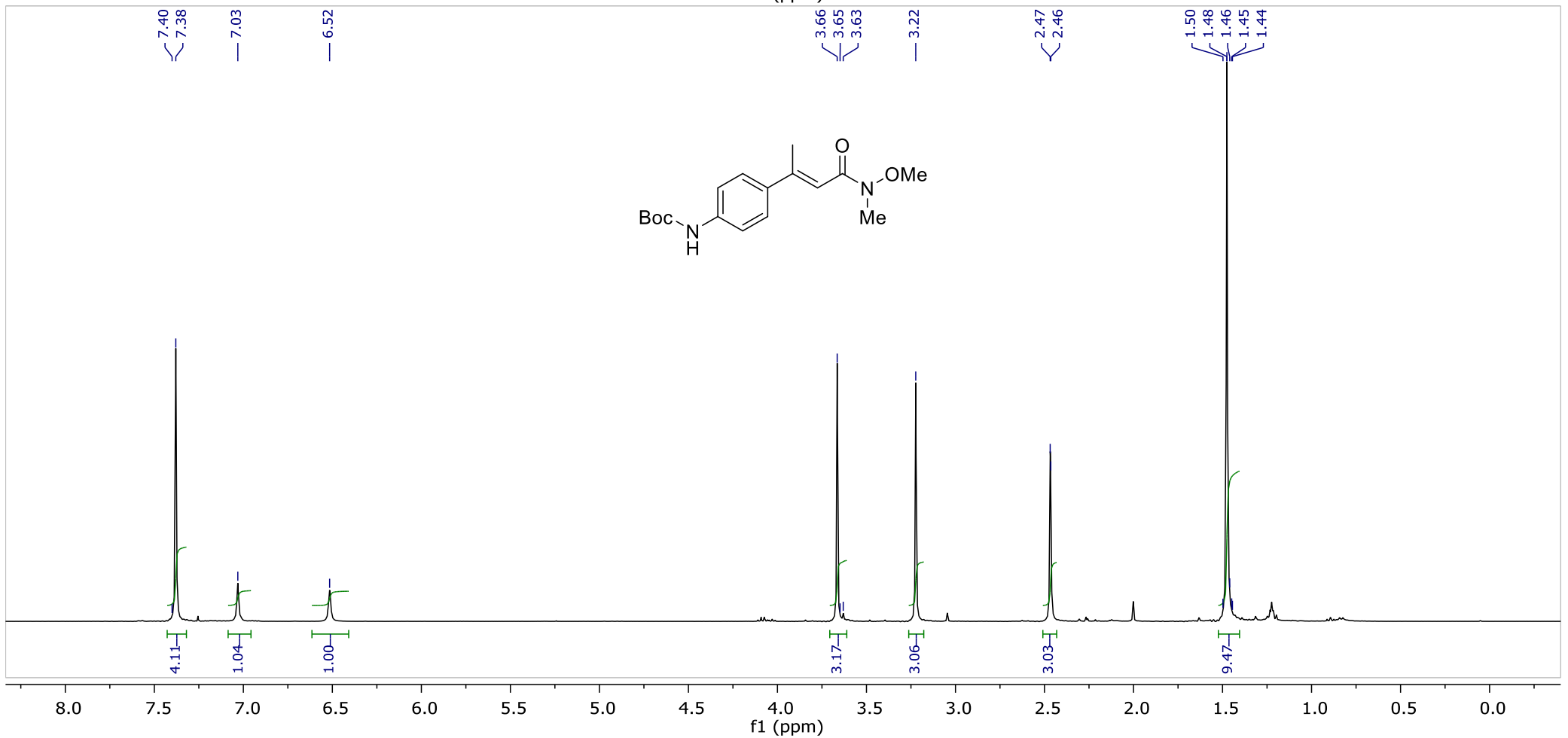
152.74
151.62
139.17
137.05
126.87
118.29
114.62
80.52
61.51
32.41
28.30
17.70
80 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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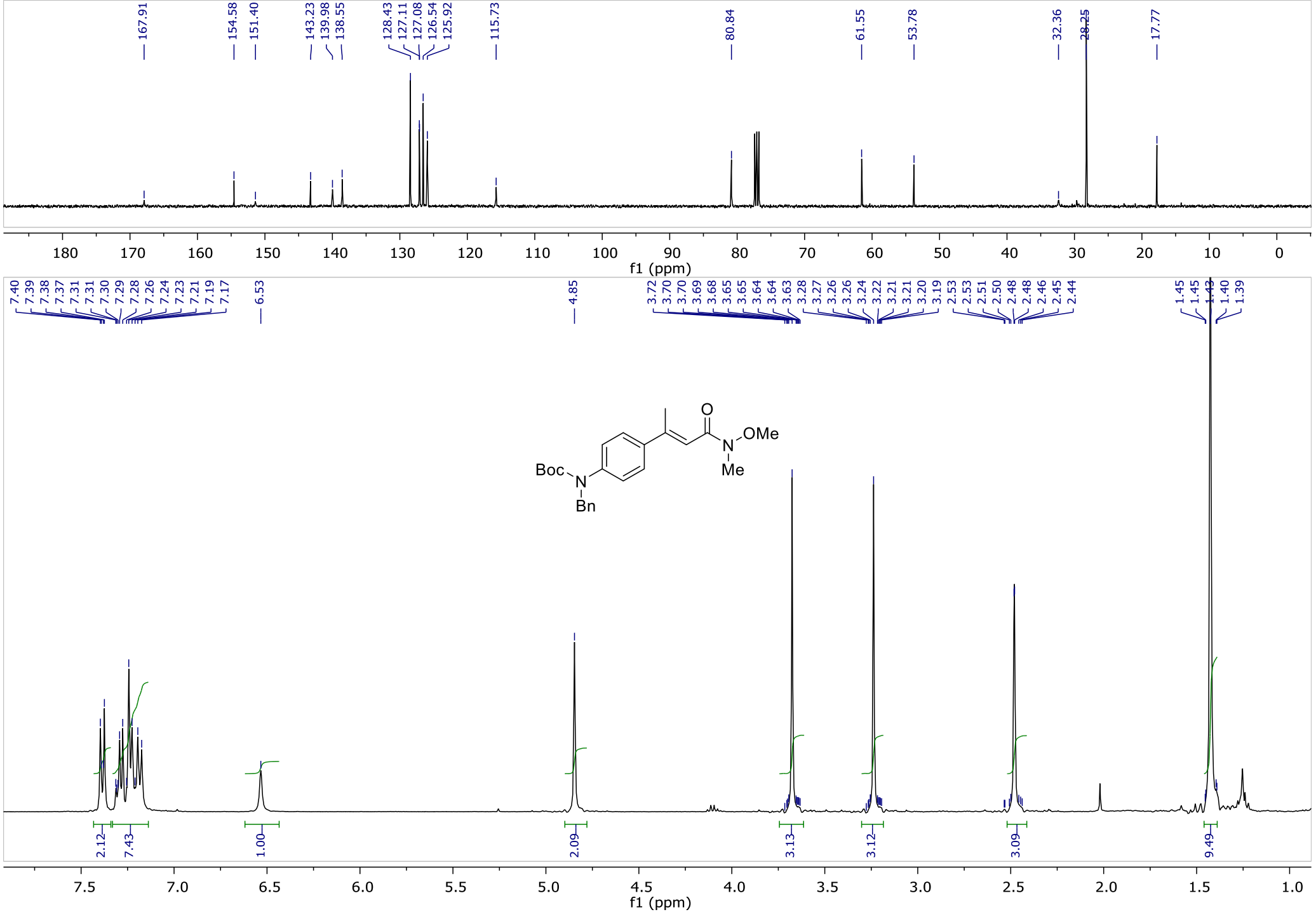
7.38
7.03
6.52
3.66
3.65
3.63
3.22
2.47
2.46
1.50
1.48
1.46
1.45
1.44
O
OMe
N
Me
Boc
N
H
4.11
1.04
1.00
3.17
3.06
3.03
9.47
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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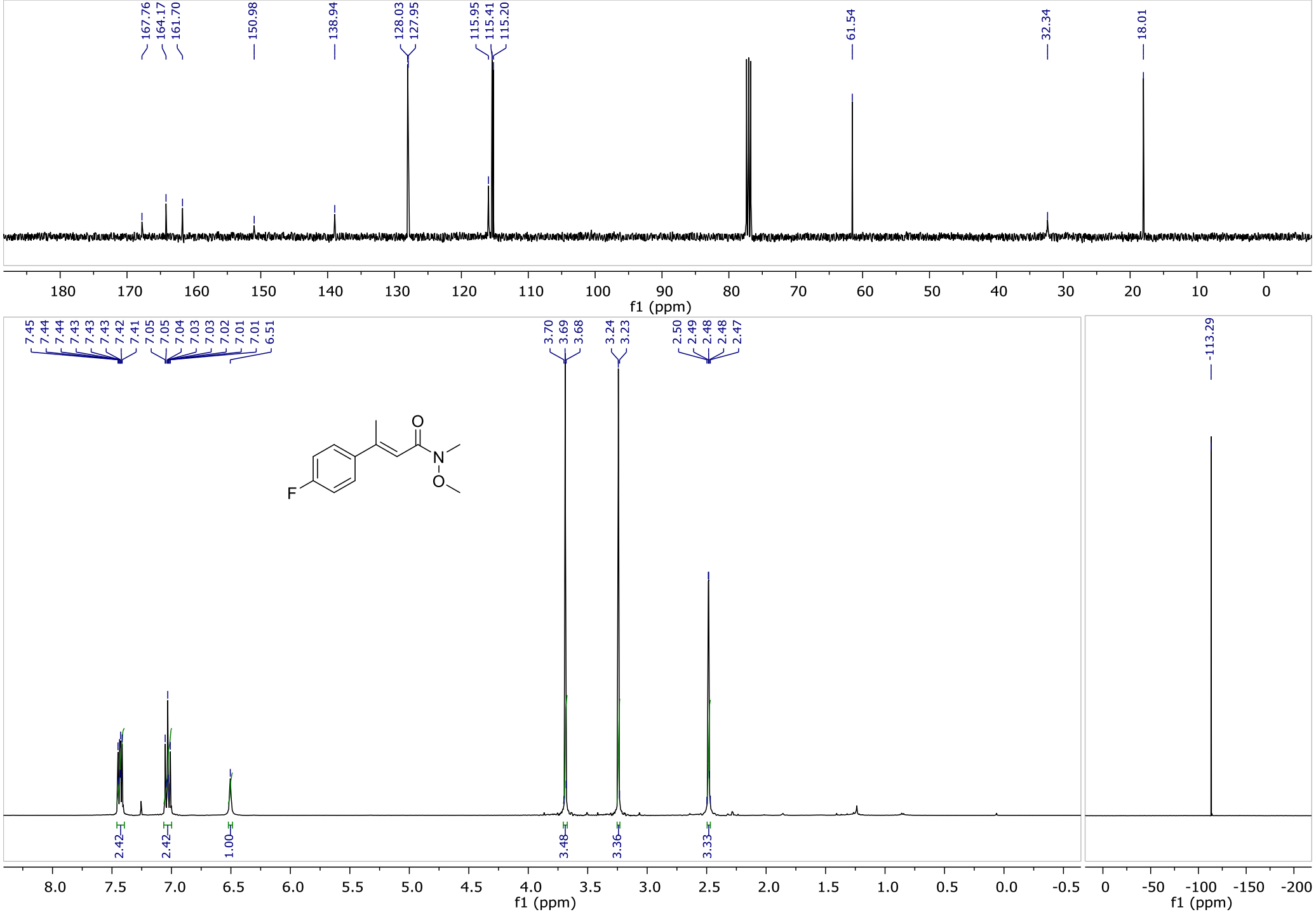
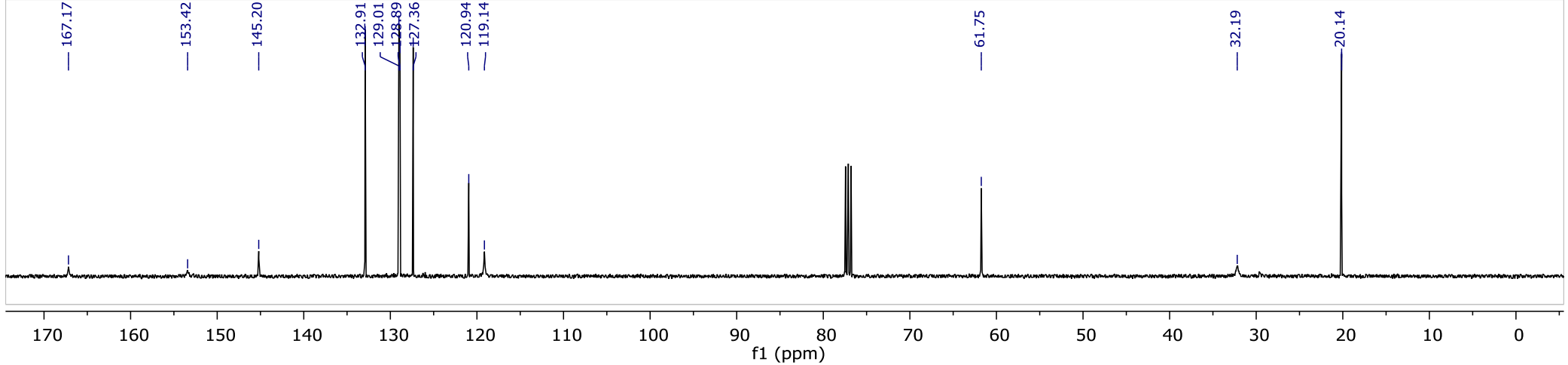
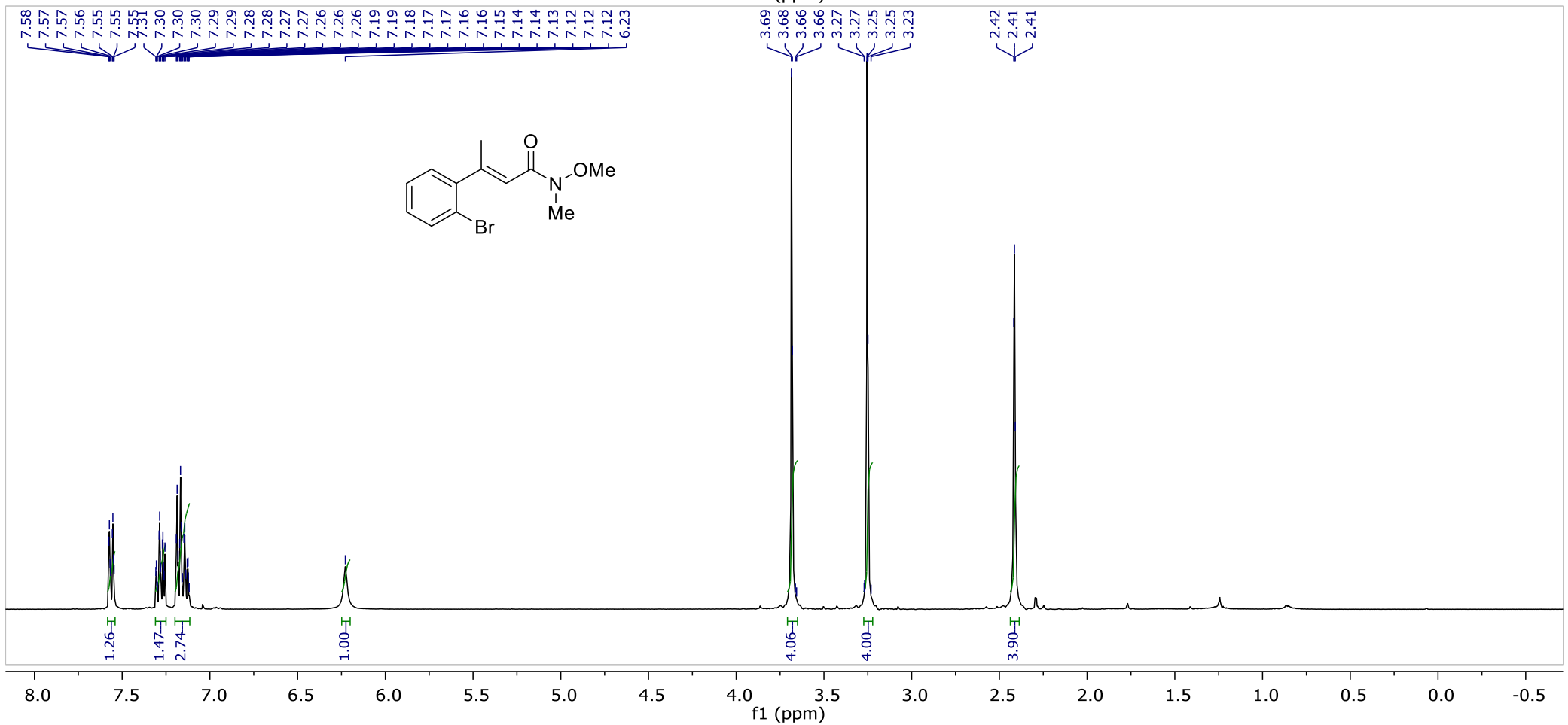
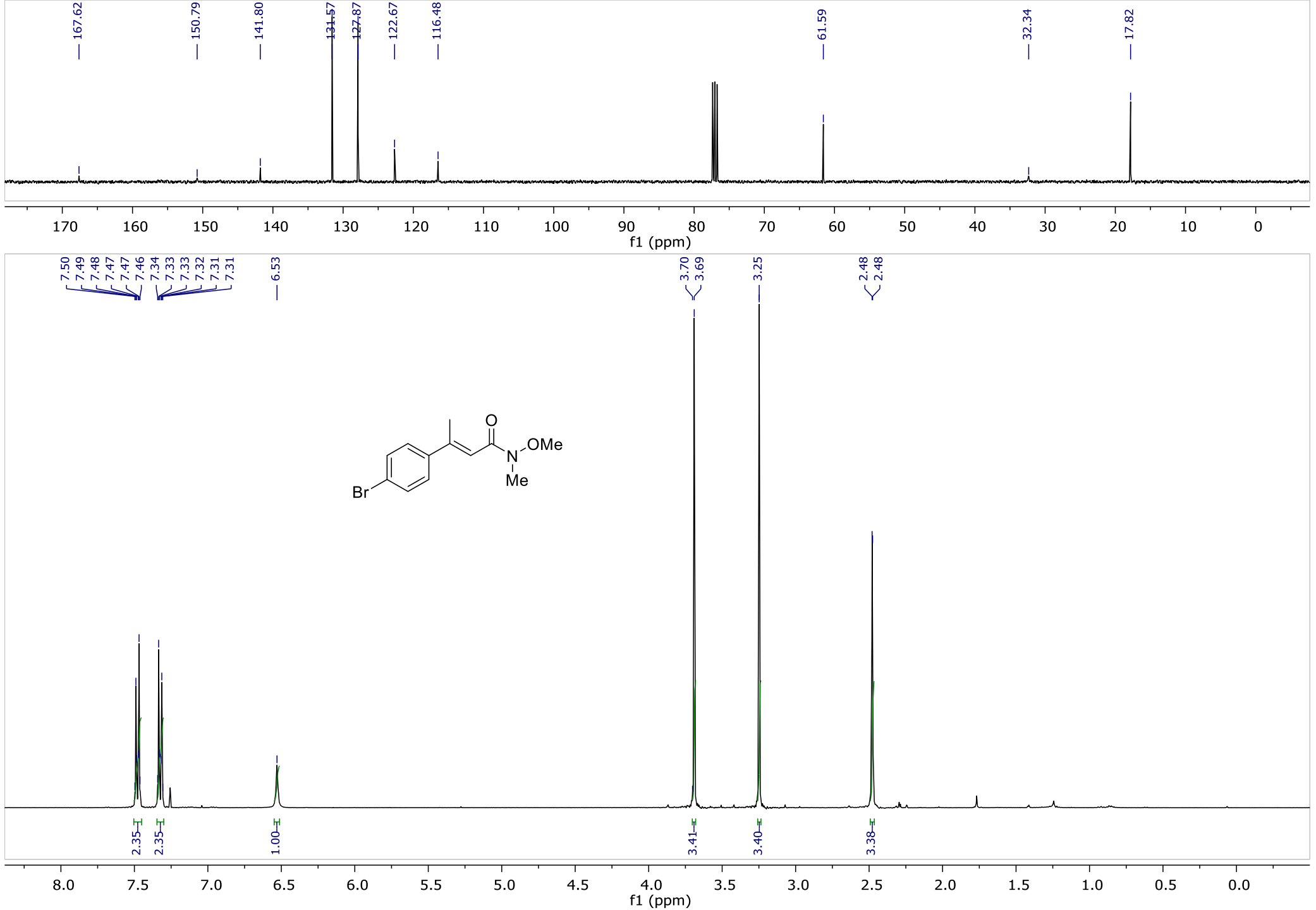
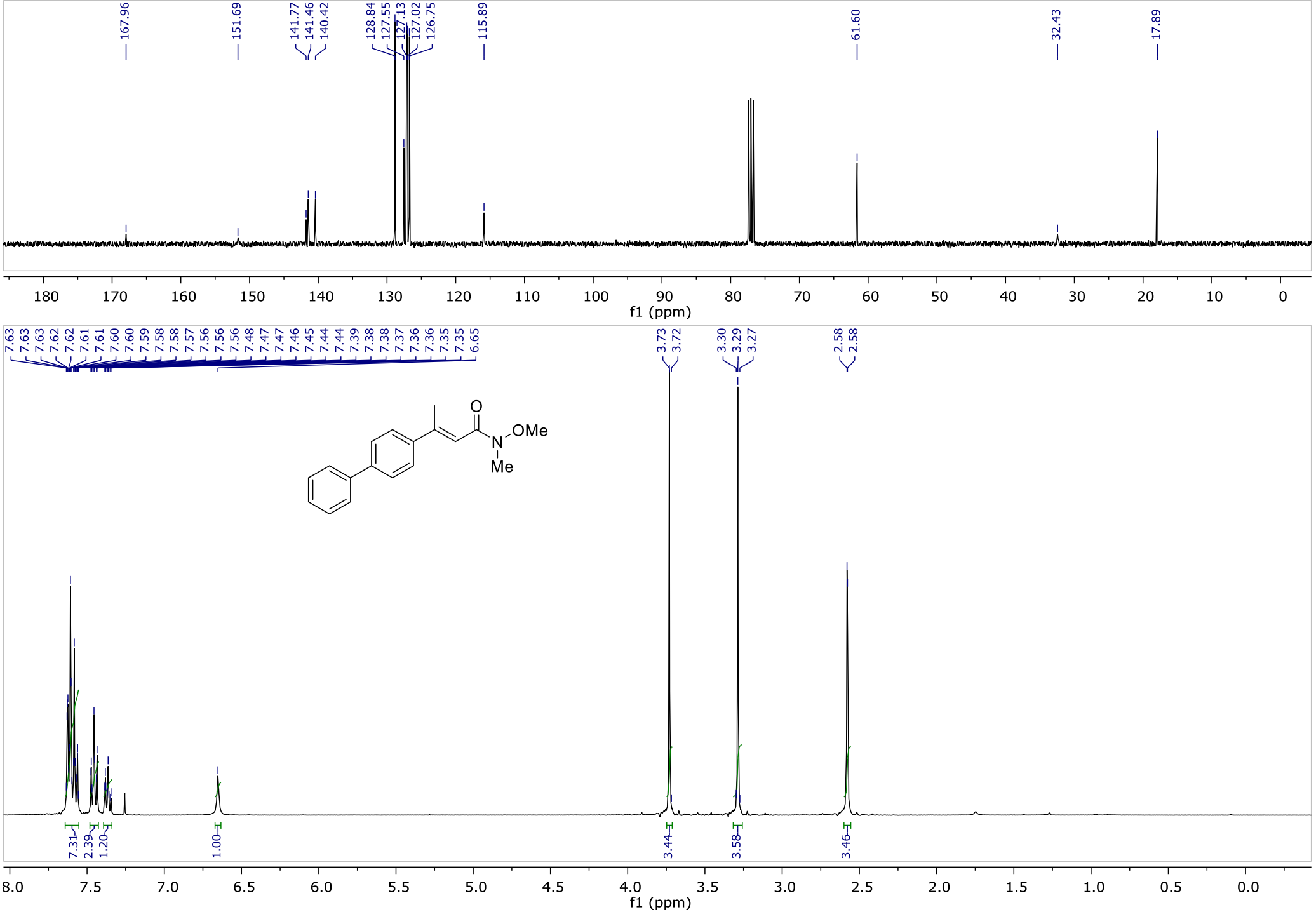
154.58
151.40
143.23
139.98
138.55
128.43
127.11
127.08
126.54
125.92
115.73
80.84
61.55
53.78
32.36
28.25
17.77
f1 (ppm)
7.40
7.39
7.38
7.37
7.31
7.31
7.30
7.29
7.28
7.26
7.24
7.23
7.21
7.19
7.17
6.53
4.85
3.72
3.70
3.70
3.69
3.68
3.65
3.65
3.64
3.64
3.63
3.28
3.27
3.26
3.26
3.24
3.22
3.21
3.21
3.20
3.19
2.53
2.53
2.51
2.50
2.48
2.48
2.46
2.45
2.44
1.45
1.45
1.43
1.40
1.39
Boc
N
Bn
O
N
OMe
Me
2.12
7.43
1.00
2.09
3.13
3.12
3.09
9.49
f1 (ppm)


167.76
164.17
161.70
150.98
138.94
128.03
127.95
115.95
115.41
115.20
61.54
32.34
18.01
f1 (ppm)
7.45
7.44
7.44
7.43
7.43
7.43
7.42
7.41
7.05
7.05
7.04
7.03
7.03
7.02
7.01
7.01
6.51
3.70
3.69
3.68
3.24
3.23
2.50
2.49
2.48
2.48
2.47
O
N
O
F
2.42
2.42
1.00
3.48
3.36
3.33
f1 (ppm)
-113.29
f1 (ppm)


167.17
153.42
145.20
132.91
129.01
128.89
127.36
120.94
119.14
61.75
32.19
20.14
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.58 7.57 7.57 7.56 7.55 7.55 7.31 7.30 7.30 7.30 7.29 7.29 7.28 7.28 7.27 7.27 7.26 7.26 7.26 7.19 7.19 7.18 7.17 7.17 7.16 7.16 7.15 7.14 7.14 7.13 7.12 7.12 7.12 6.23
3.69 3.68 3.66 3.66 3.27 3.27 3.25 3.25 3.23
2.42 2.41 2.41
O
N
OMe
Me
Br
1.26 1.47 2.74 1.00 4.06 4.00 3.90
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)


167.62
150.79
141.80
131.57
127.87
122.67
116.48
61.59
32.34
17.82
f1 (ppm)
7.50
7.49
7.48
7.47
7.47
7.46
7.34
7.33
7.33
7.32
7.31
7.31
6.53
3.70
3.69
3.25
2.48
2.48
O
N
OMe
Me
Br
2.35
2.35
1.00
3.41
3.40
3.38
f1 (ppm)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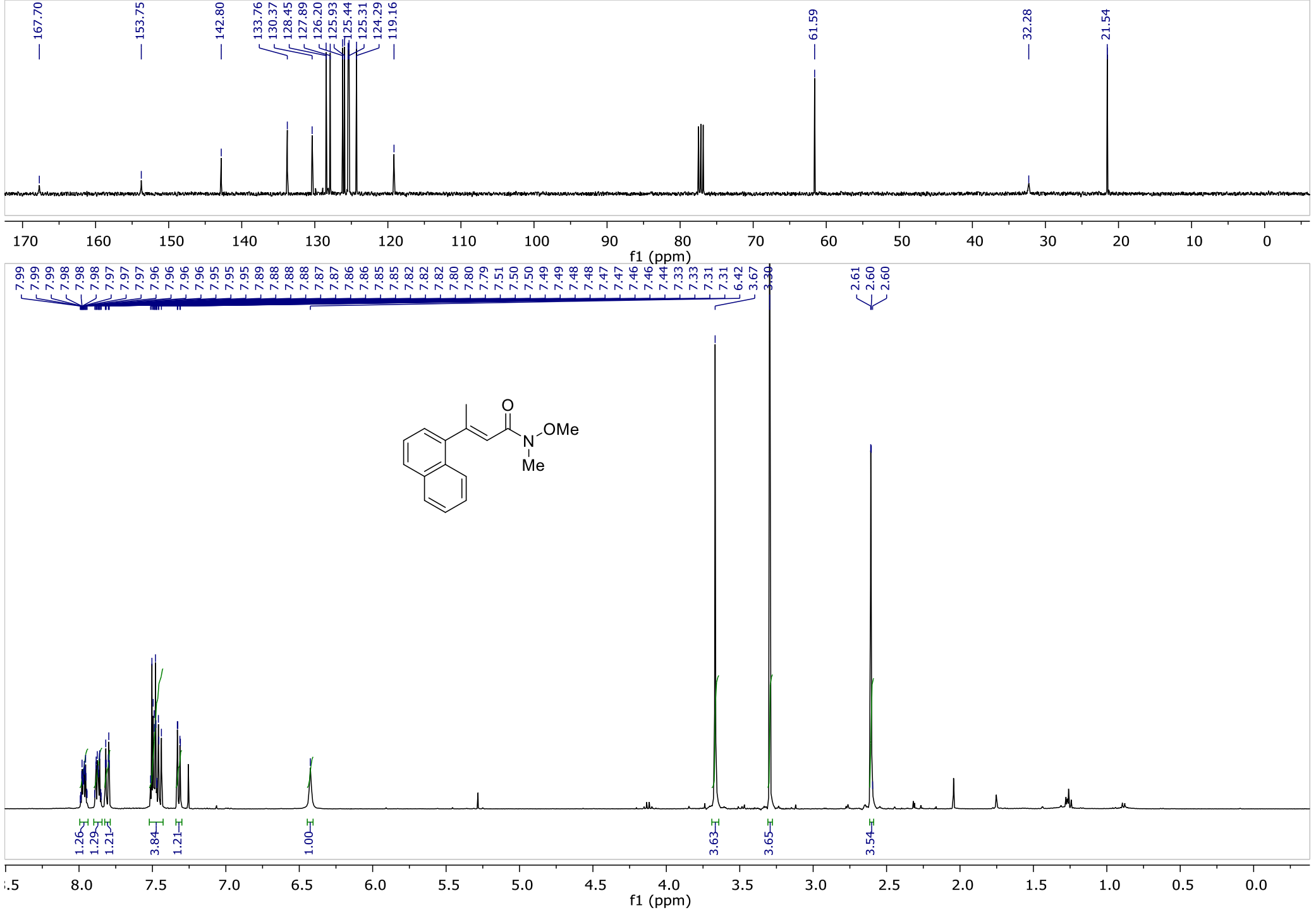
151.69
141.77
141.46
140.42
128.84
127.55
127.13
127.02
126.75
115.89
61.60
32.43
17.89
f1 (ppm)
7.63
7.63
7.63
7.62
7.62
7.61
7.61
7.60
7.60
7.59
7.58
7.58
7.57
7.56
7.56
7.56
7.48
7.47
7.47
7.46
7.45
7.44
7.44
7.39
7.38
7.38
7.37
7.36
7.36
7.35
7.35
6.65
3.73
3.72
3.30
3.29
3.27
2.58
2.58
O
N
OMe
Me
7.31
2.39
1.20
1.00
3.44
3.58
3.46
f1 (ppm)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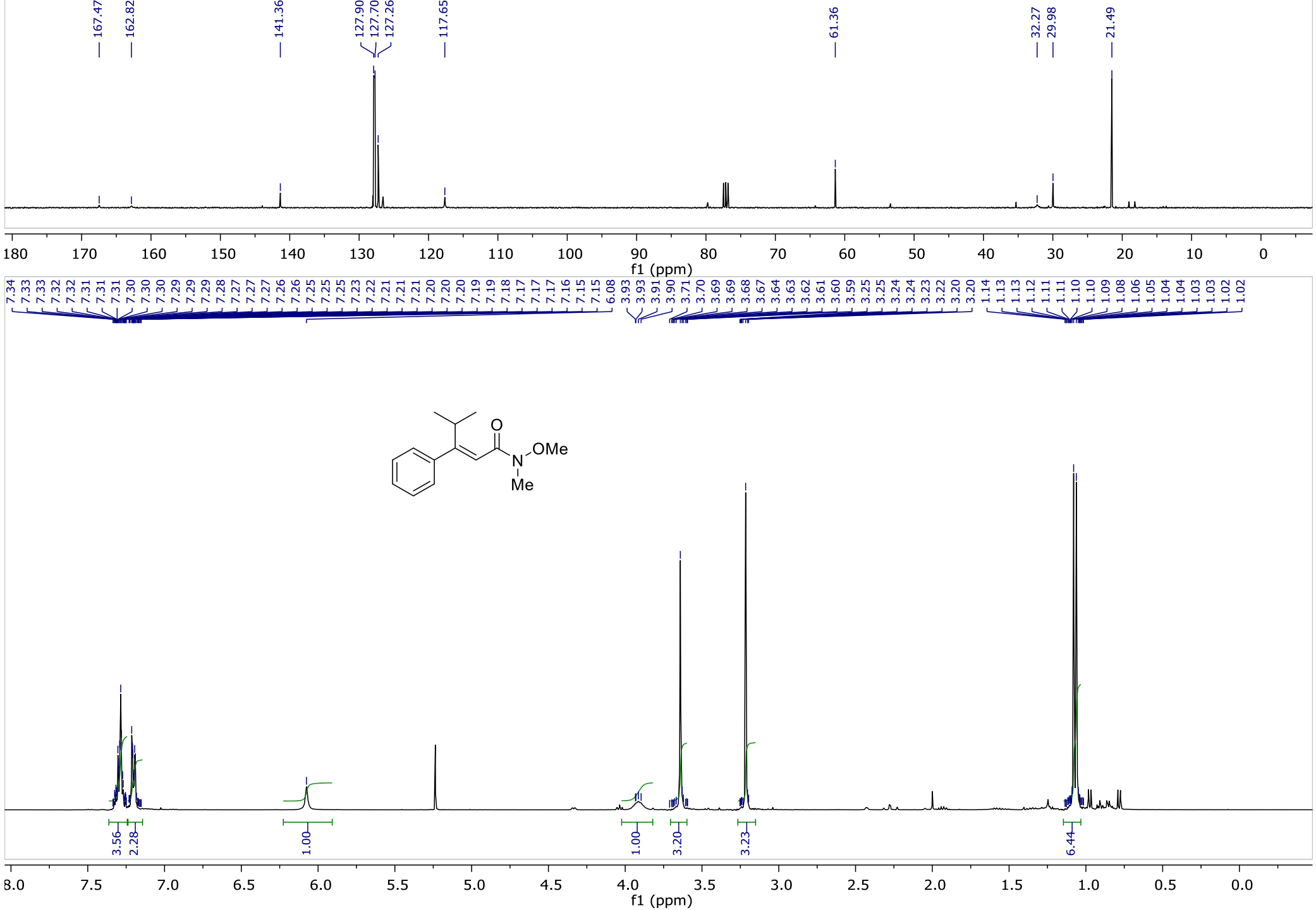
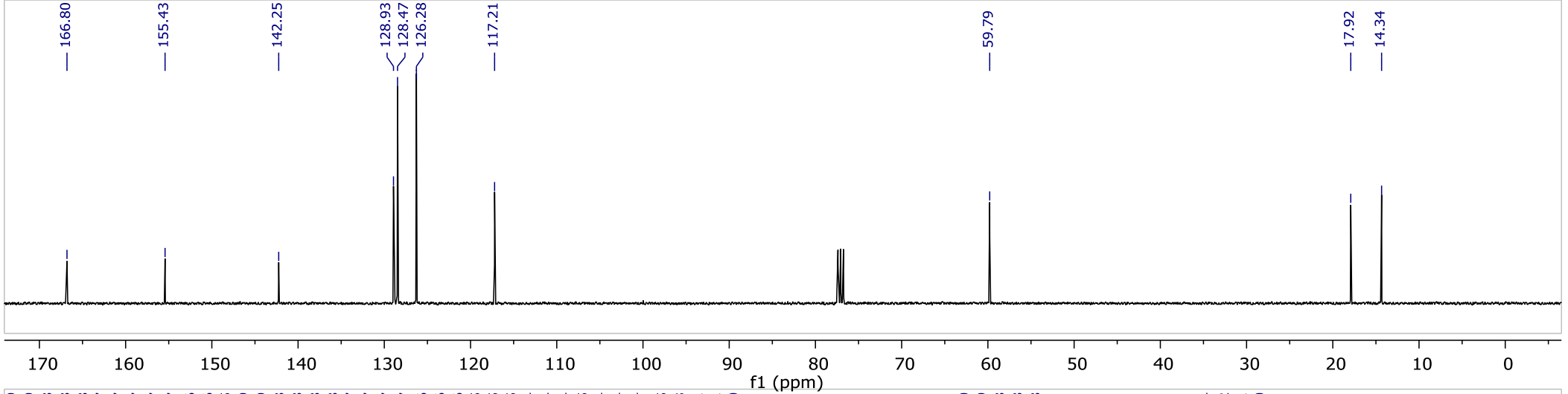
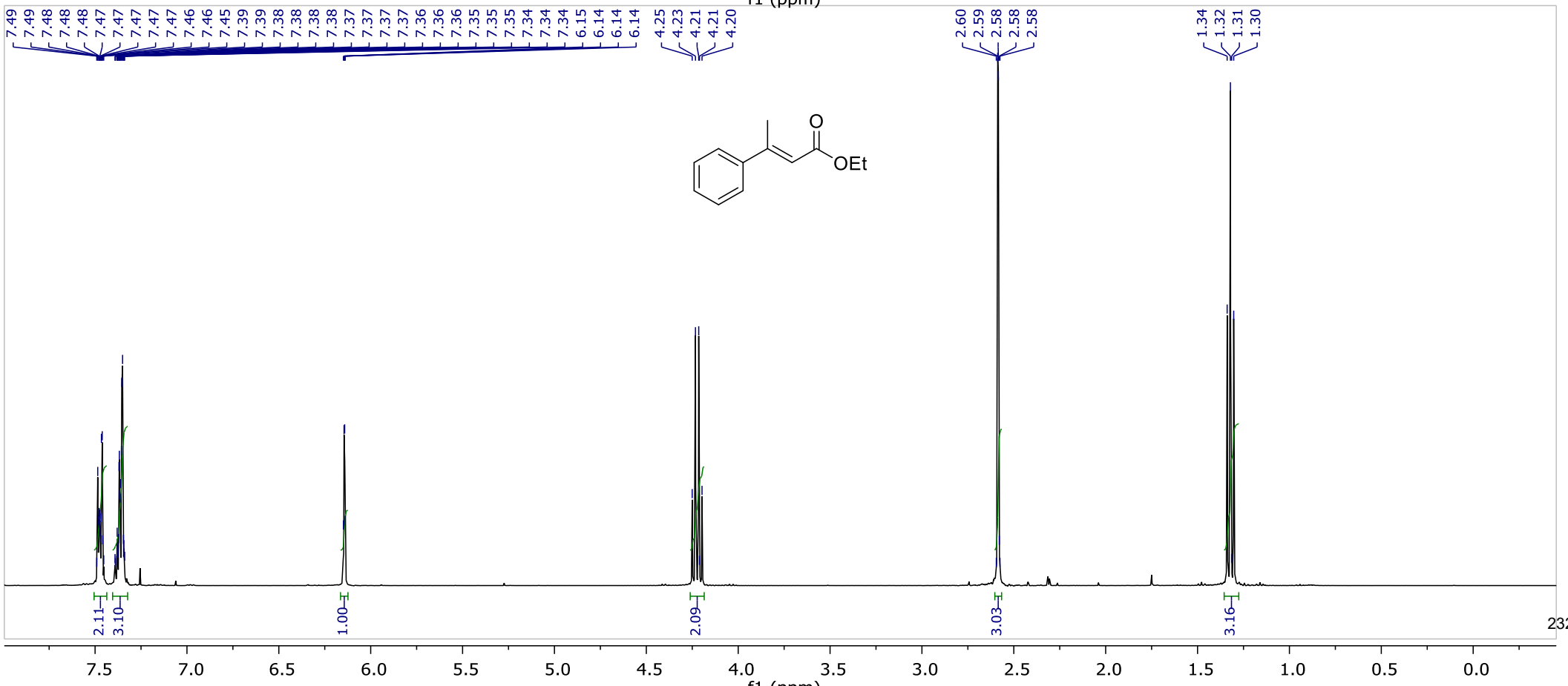
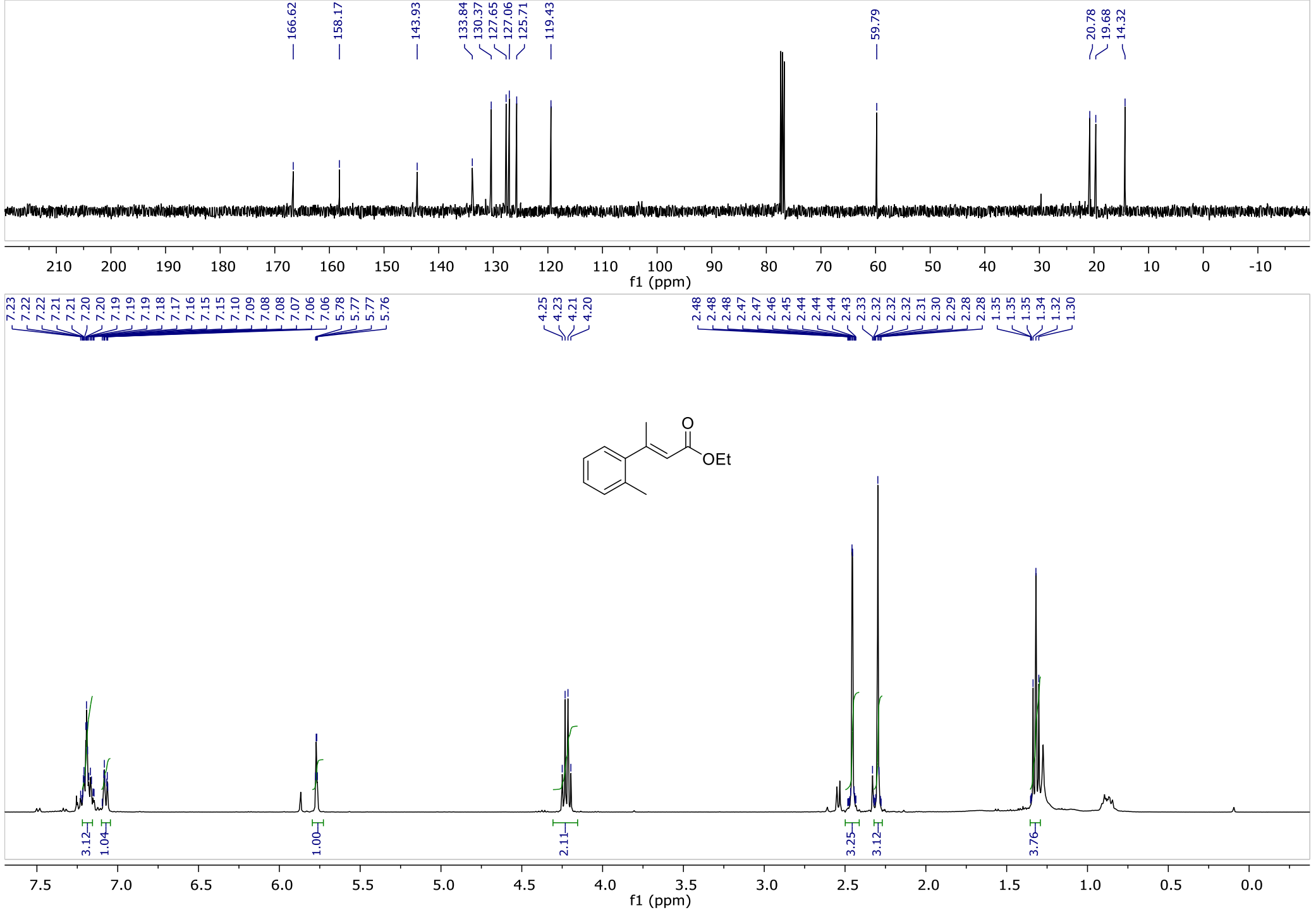
153.75
142.80
133.76
130.37
128.45
127.89
126.20
125.93
125.44
125.31
124.29
119.16
61.59
32.28
21.54
f1 (ppm)
O
N
OMe
Me
2.61
2.60
2.60
1.26
1.29
1.21
3.84
1.21
1.00
3.63
3.65
3.54
f1 (ppm)


167.47
162.82
141.36
127.90
127.70
127.26
117.65
61.36
32.27
29.98
21.49
f1 (ppm)
O
N
OMe
Me
3.56
2.28
1.00
1.00
3.20
3.23
6.44
f1 (ppm)


166.80
155.43
142.25
128.93
128.47
126.28
117.21
59.79
17.92
14.34
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.49 7.49 7.48 7.48 7.48 7.47 7.47 7.47 7.47 7.47 7.46 7.46 7.45 7.39 7.39 7.38 7.38 7.38 7.38 7.37 7.37 7.37 7.37 7.36 7.36 7.36 7.35 7.35 7.35 7.34 7.34 7.34 6.15 6.14 6.14 6.14
4.25 4.23 4.21 4.21 4.20
2.60 2.59 2.58 2.58 2.58
1.34 1.32 1.31 1.30
O
OEt
2.11
3.10
1.00
2.09
3.03
3.16
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
f1 (ppm)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158.17
143.93
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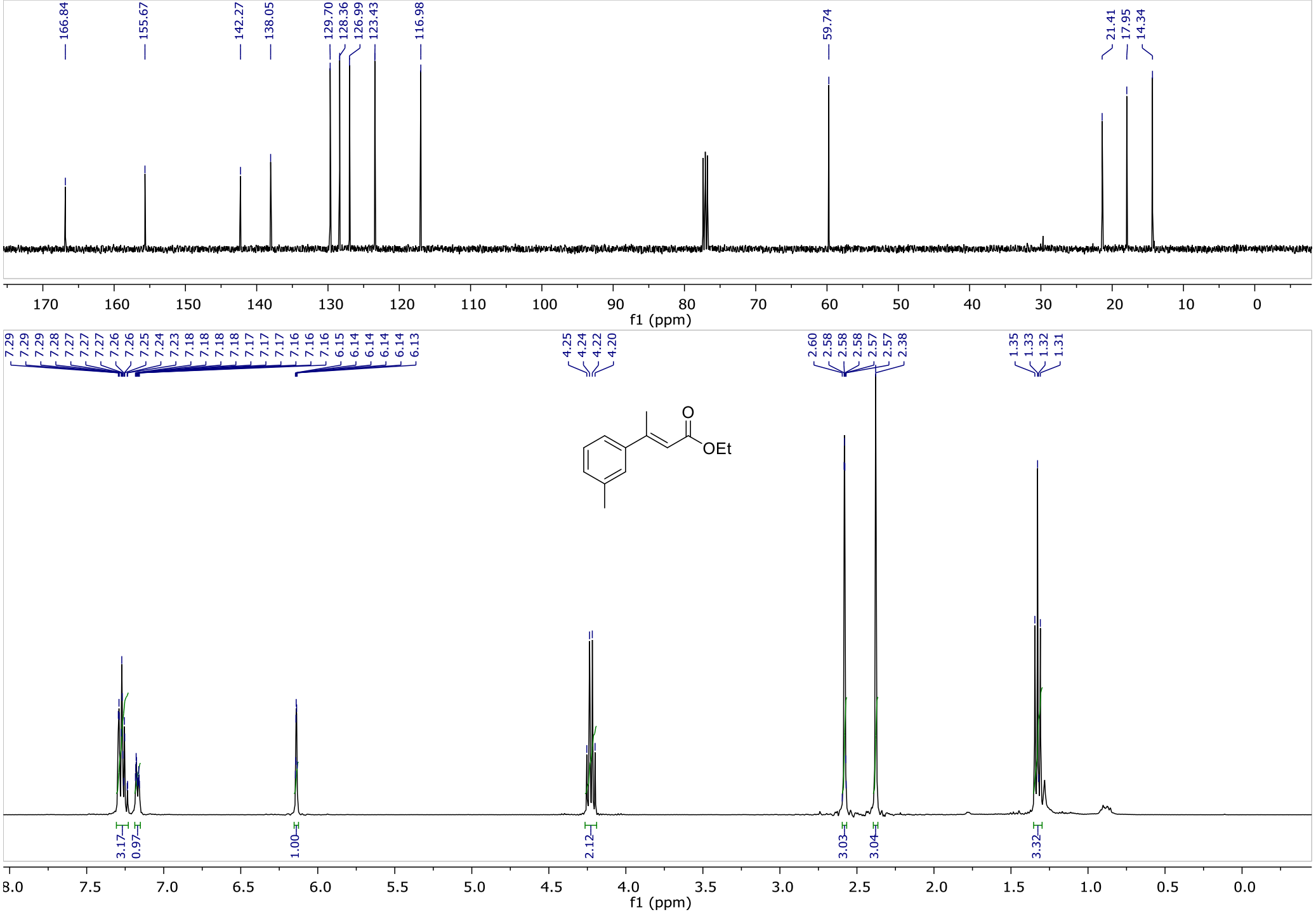
130.37
127.65
127.06
125.71
119.43
59.79
20.78
19.68
14.32
f1 (ppm)
7.23 7.22 7.22 7.21 7.21 7.20 7.20 7.19 7.19 7.19 7.18 7.17 7.16 7.15 7.15 7.10 7.09 7.08 7.08 7.07 7.06 7.06 5.78 5.77 5.77 5.76
4.25 4.23 4.21 4.20
2.48 2.48 2.48 2.47 2.47 2.46 2.45 2.44 2.44 2.44 2.43 2.33 2.32 2.32 2.32 2.31 2.30 2.29 2.28 2.28
1.35 1.35 1.35 1.34 1.32 1.30
O
OEt
3.12
1.04
1.00
2.11
3.25
3.12
3.76
f1 (ppm)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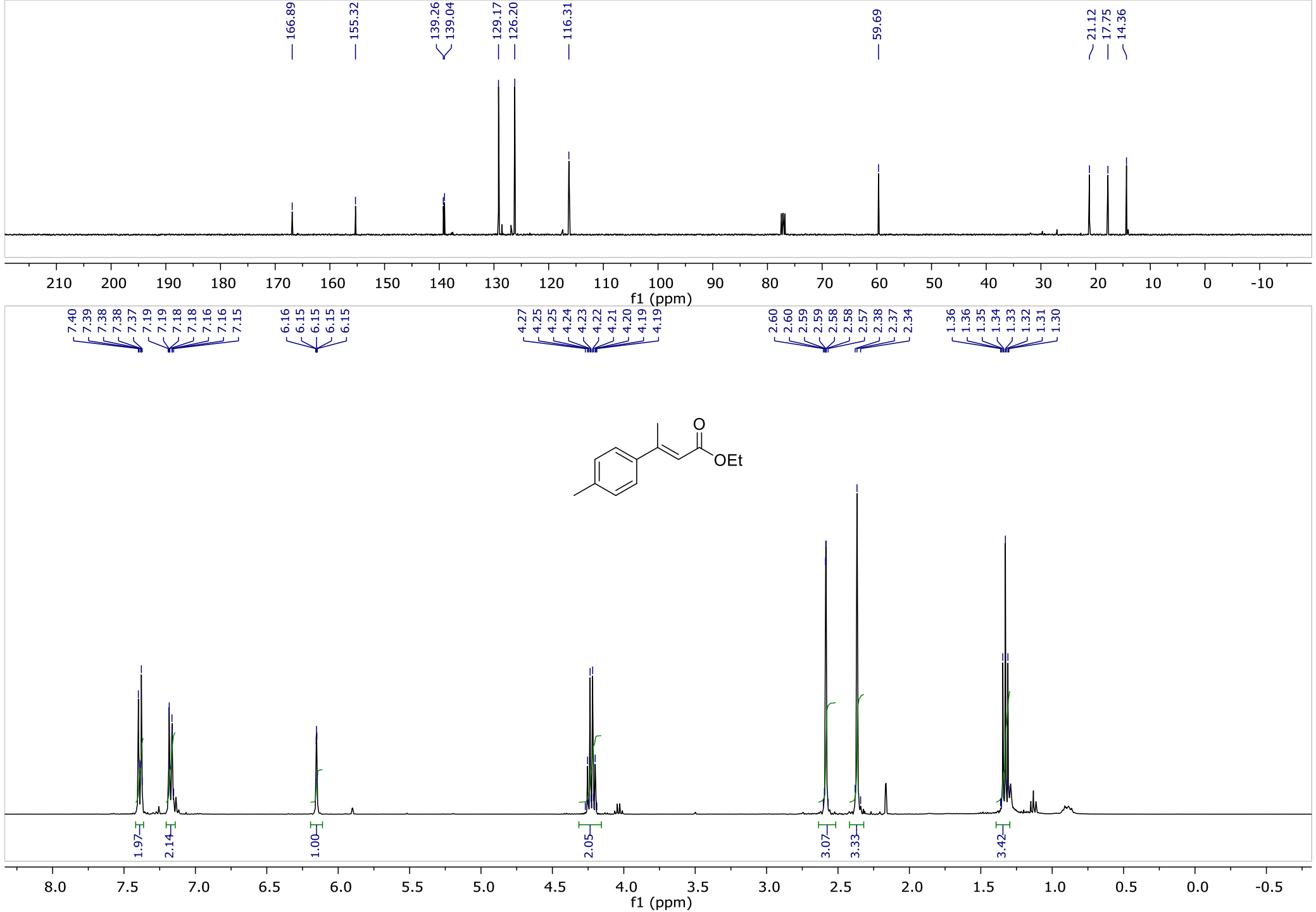
128.36
126.99
123.43
116.98
59.74
21.41
17.95
14.34
f1 (ppm)
7.29
7.29
7.29
7.28
7.27
7.27
7.27
7.26
7.26
7.25
7.24
7.23
7.18
7.18
7.18
7.18
7.17
7.17
7.17
7.16
7.16
6.15
6.14
6.14
6.14
6.14
6.13
4.25
4.24
4.22
4.20
2.60
2.58
2.58
2.58
2.57
2.57
2.38
1.35
1.33
1.32
1.31
O
OEt
3.17
0.97
1.00
2.12
3.03
3.04
3.32
f1 (ppm)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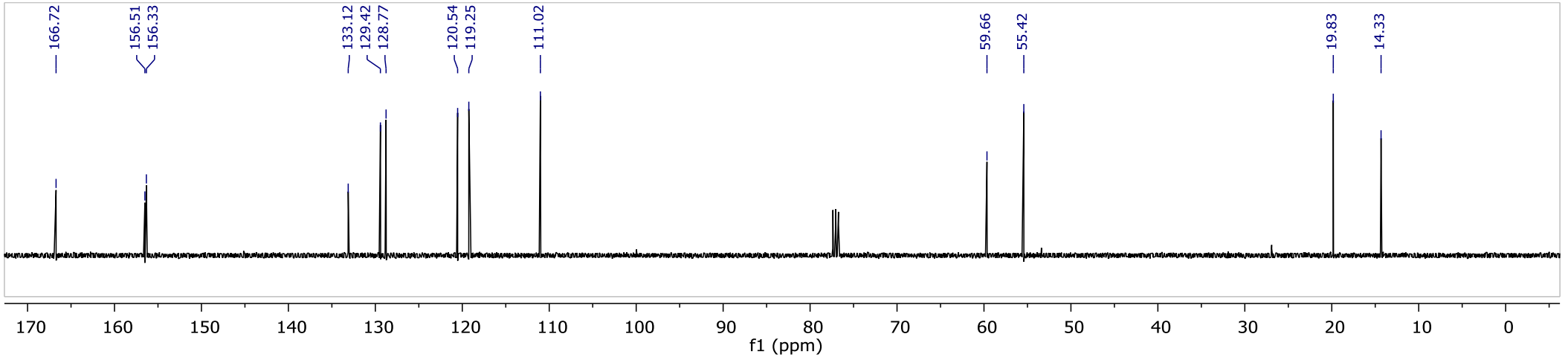
139.26
139.04
129.17
126.20
116.31
59.69
21.12
17.75
14.36
f1 (ppm)
7.40 7.39 7.38 7.38 7.37 7.19 7.19 7.18 7.18 7.16 7.16 7.15
6.16 6.15 6.15 6.15 6.15
4.27 4.25 4.25 4.24 4.23 4.22 4.21 4.20 4.19 4.19
2.60 2.60 2.59 2.59 2.58 2.58 2.57 2.38 2.37 2.34
1.36 1.36 1.35 1.34 1.33 1.32 1.31 1.30
O
OEt
1.97
2.14
1.00
2.05
3.07
3.33
3.42
f1 (ppm)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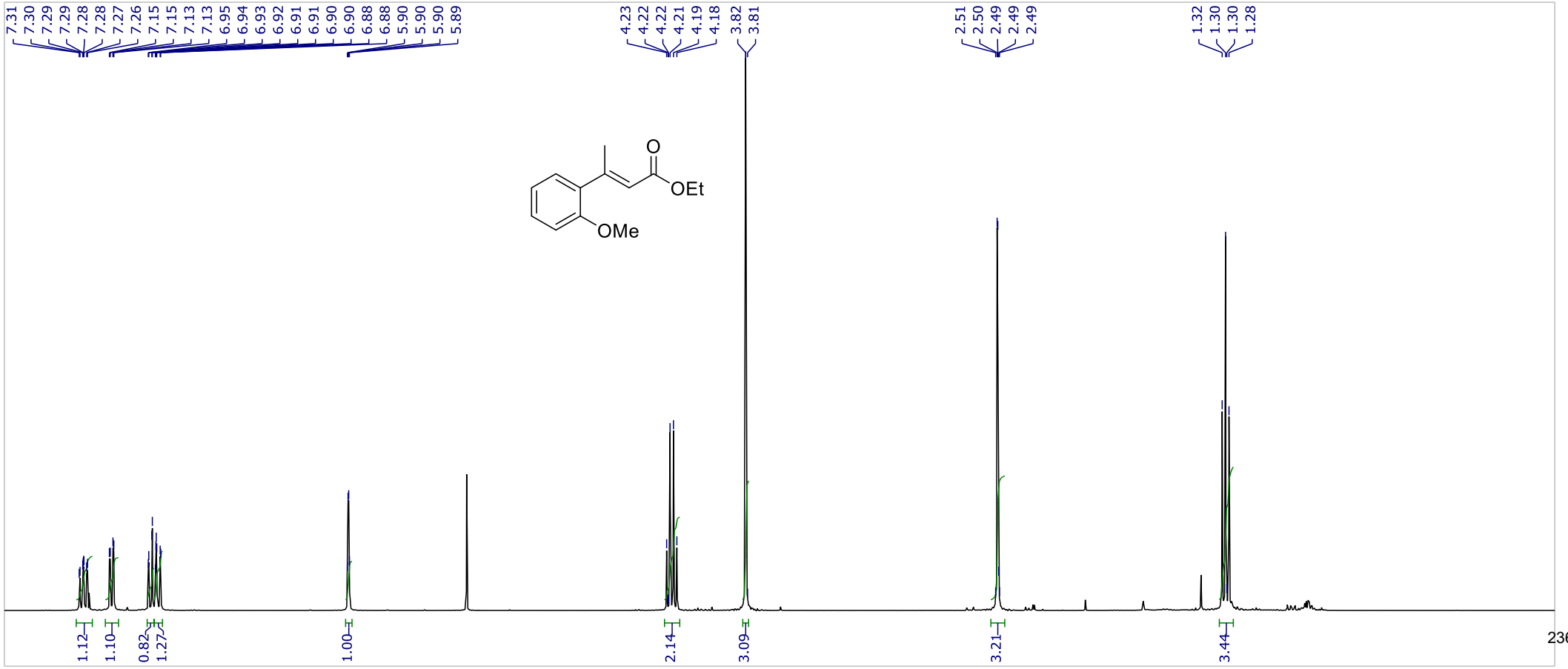
129.42
128.77
120.54
119.25
111.02
59.66
55.42
19.83
14.33
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.29
7.29
7.28
7.28
7.27
7.26
7.15
7.15
7.13
7.13
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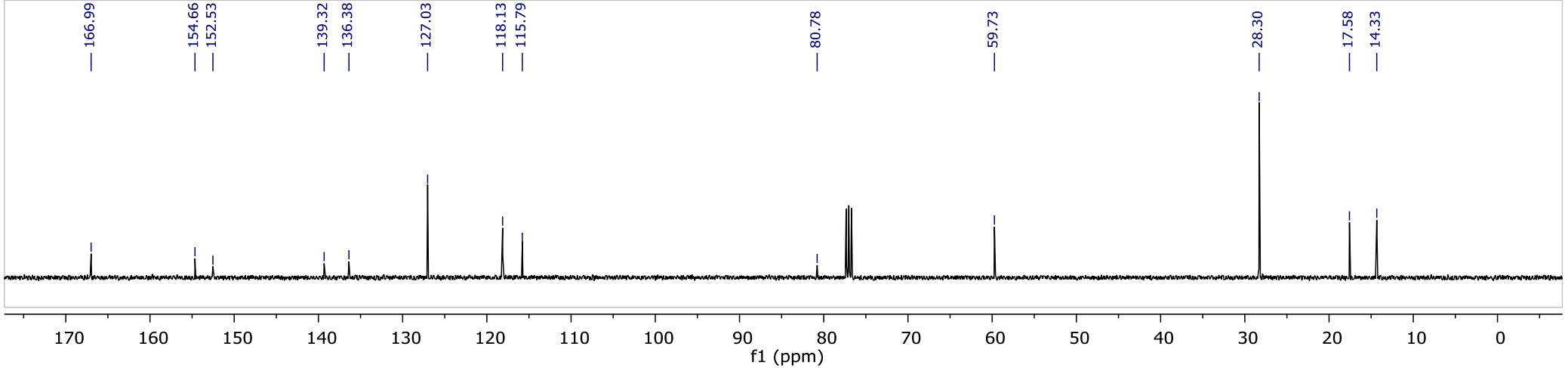
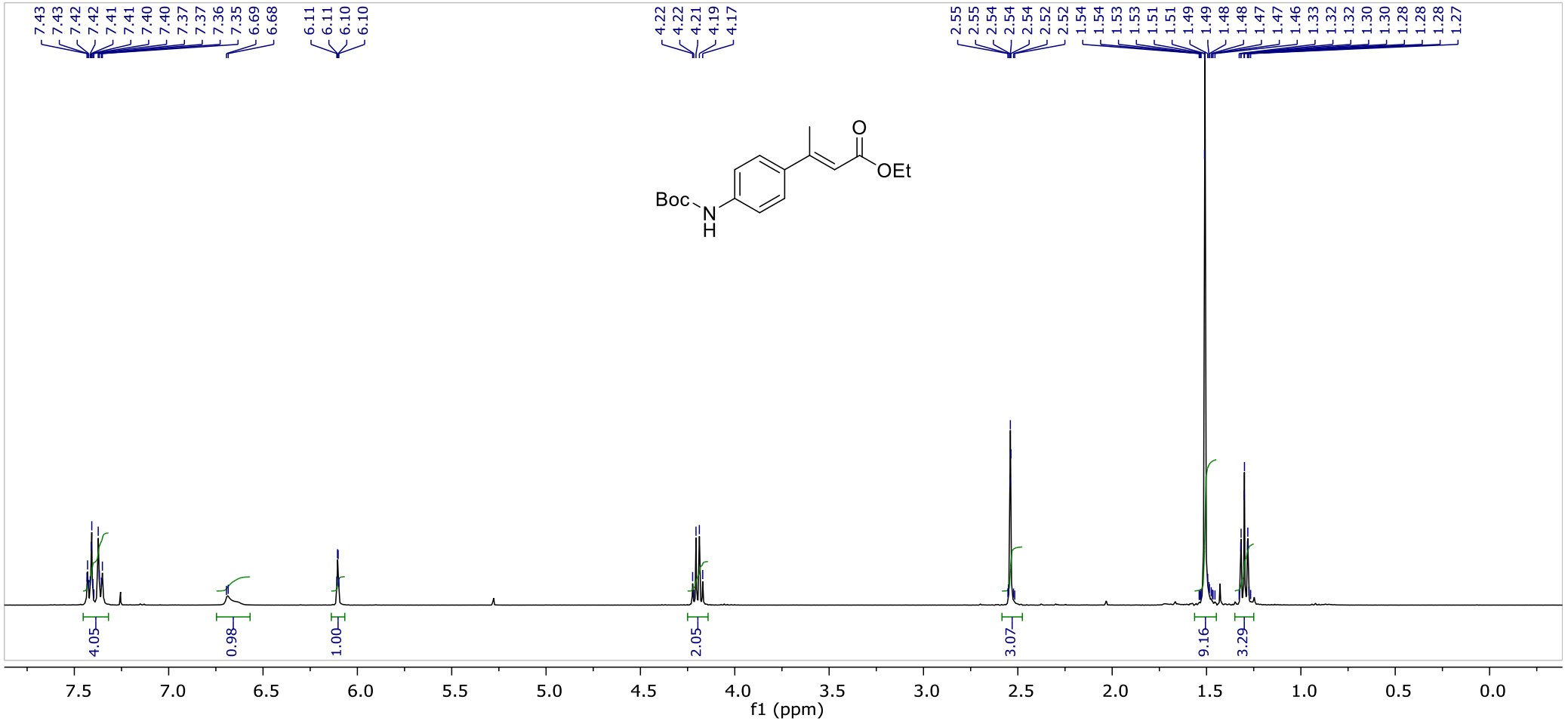
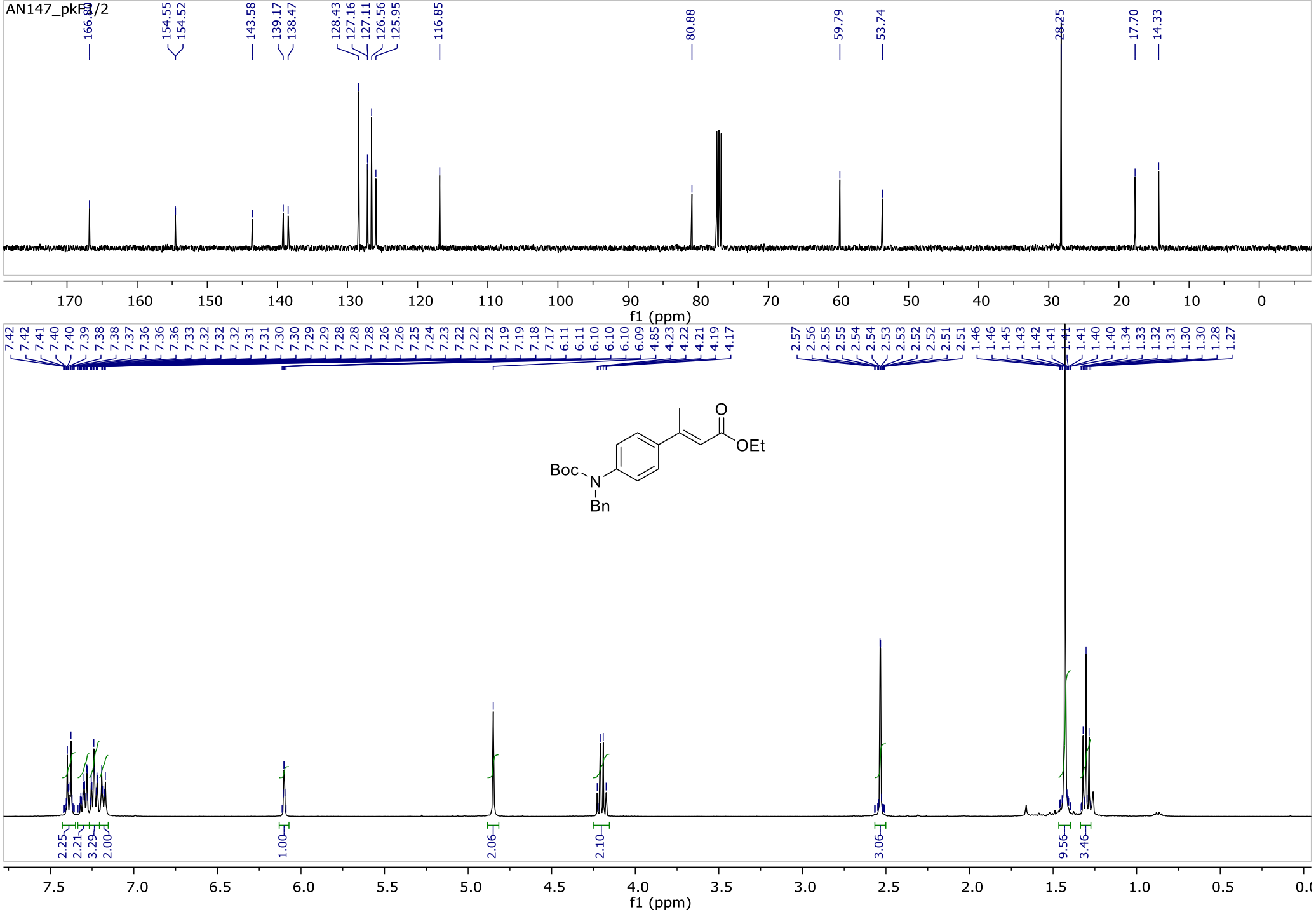
6.94
6.93
6.92
6.91
6.91
6.90
6.90
6.88
6.88
5.90
5.90
5.90
5.89
4.23
4.22
4.22
4.21
4.19
4.18
3.82
3.81
2.51
2.50
2.49
2.49
2.49
1.32
1.30
1.30
1.28
O
OEt
OMe
1.12
1.10
0.82
1.27
1.00
2.14
3.09
3.21
3.44
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0


166.99
154.66
152.53
139.32
136.38
127.03
118.13
115.79
80.78
59.73
28.30
17.58
14.33
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.43
7.43
7.42
7.42
7.41
7.41
7.40
7.40
7.37
7.37
7.36
7.35
6.69
6.68
6.11
6.11
6.10
6.10
4.22
4.22
4.21
4.19
4.17
2.55
2.55
2.54
2.54
2.52
2.52
1.54
1.54
1.53
1.53
1.51
1.51
1.49
1.49
1.48
1.48
1.47
1.47
1.46
1.33
1.32
1.32
1.30
1.28
1.28
1.28
1.27
O
OEt
Boc
N
H
4.05
0.98
1.00
2.05
3.07
9.16
3.29
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
f1 (ppm)


AN147_pkf3/2
166.84
154.55
154.52
143.58
139.17
138.47
128.43
127.16
127.11
126.56
125.95
116.85
80.88
59.79
53.74
28.25
17.70
14.33
f1 (ppm)
O
OEt
Boc
N
Bn
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3.29
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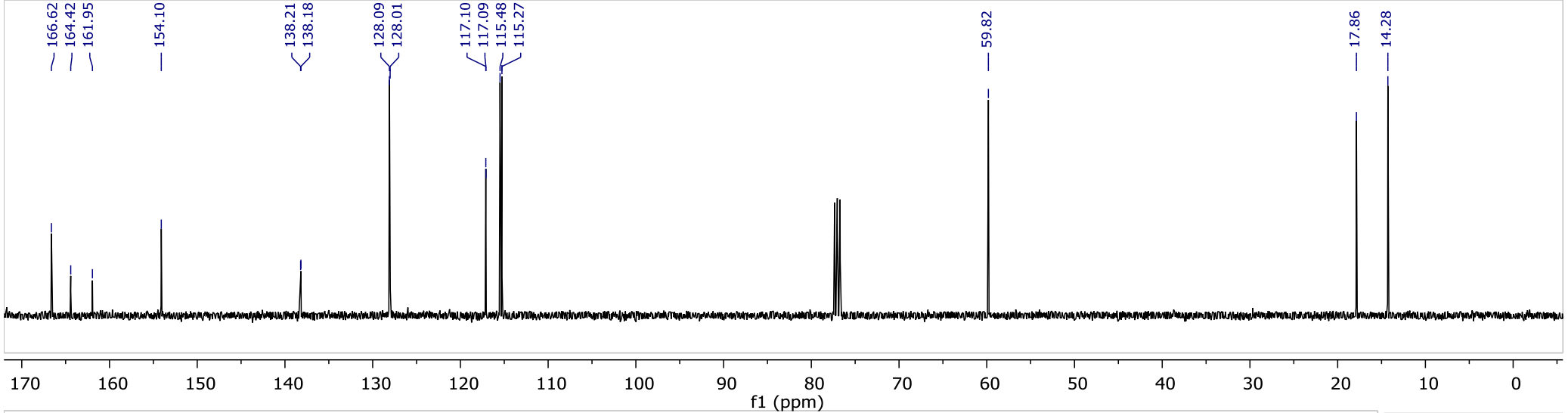
1.00
2.06
2.10
3.06
9.56
3.46
f1 (ppm)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138.18
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128.01
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117.09
115.48
115.27
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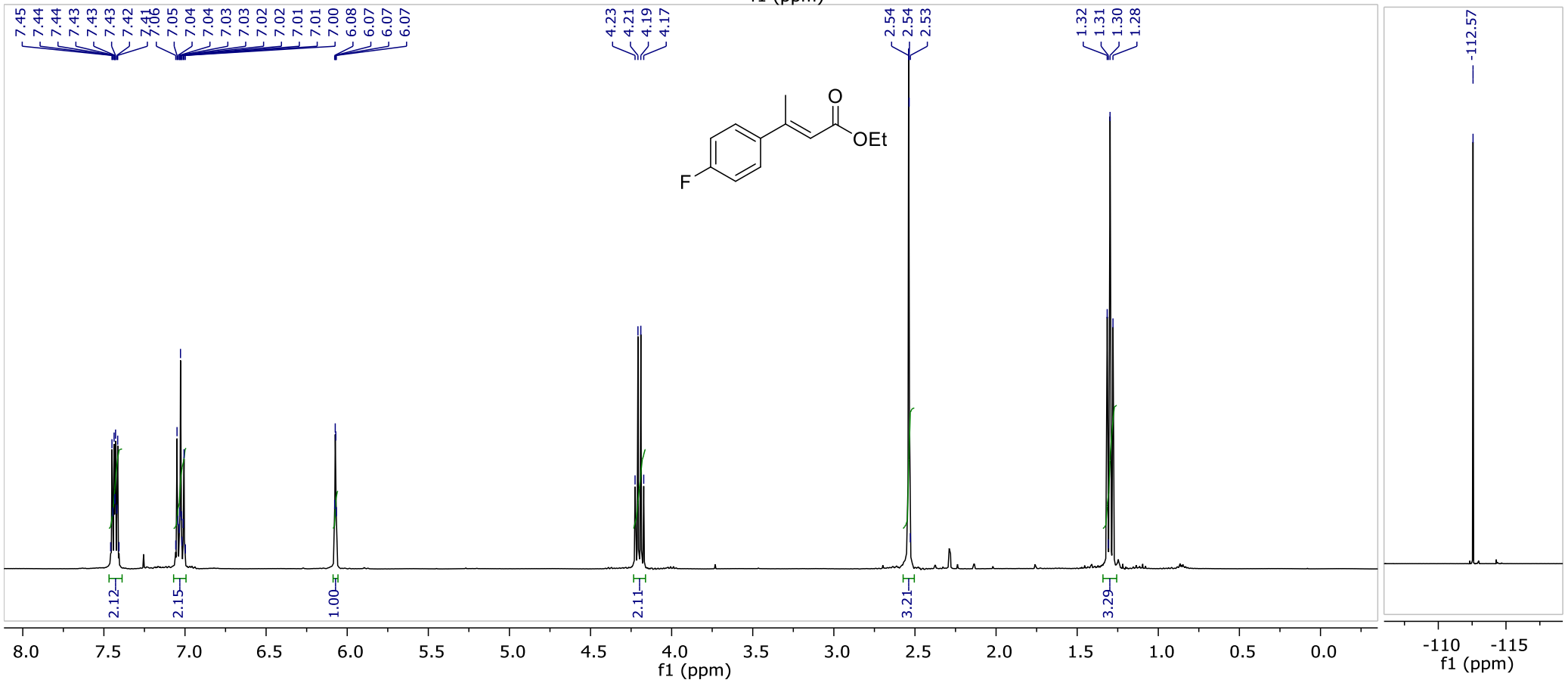
17.86
14.28
f1 (ppm)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7.44
7.44
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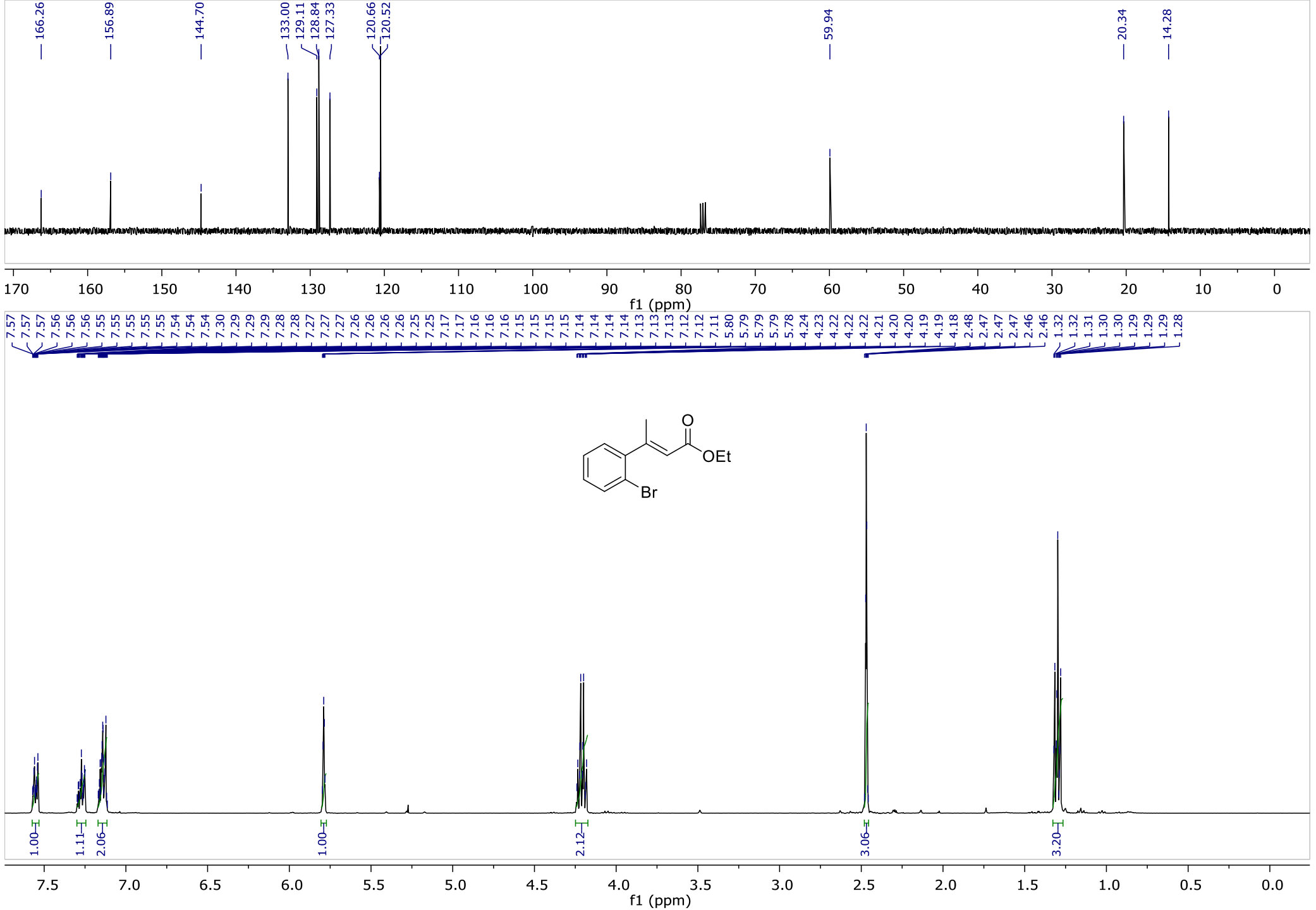
7.43
7.43
7.42
7.41
7.06
7.05
7.04
7.04
7.04
7.03
7.03
7.03
7.02
7.02
7.02
7.01
7.01
7.01
7.00
6.08
6.07
6.07
6.07
4.23
4.21
4.19
4.17
2.54
2.54
2.53
1.32
1.31
1.30
1.28
O
OEt
F
2.12
2.15
1.00
2.11
3.21
3.29
f1 (ppm)
-112.57
f1 (ppm)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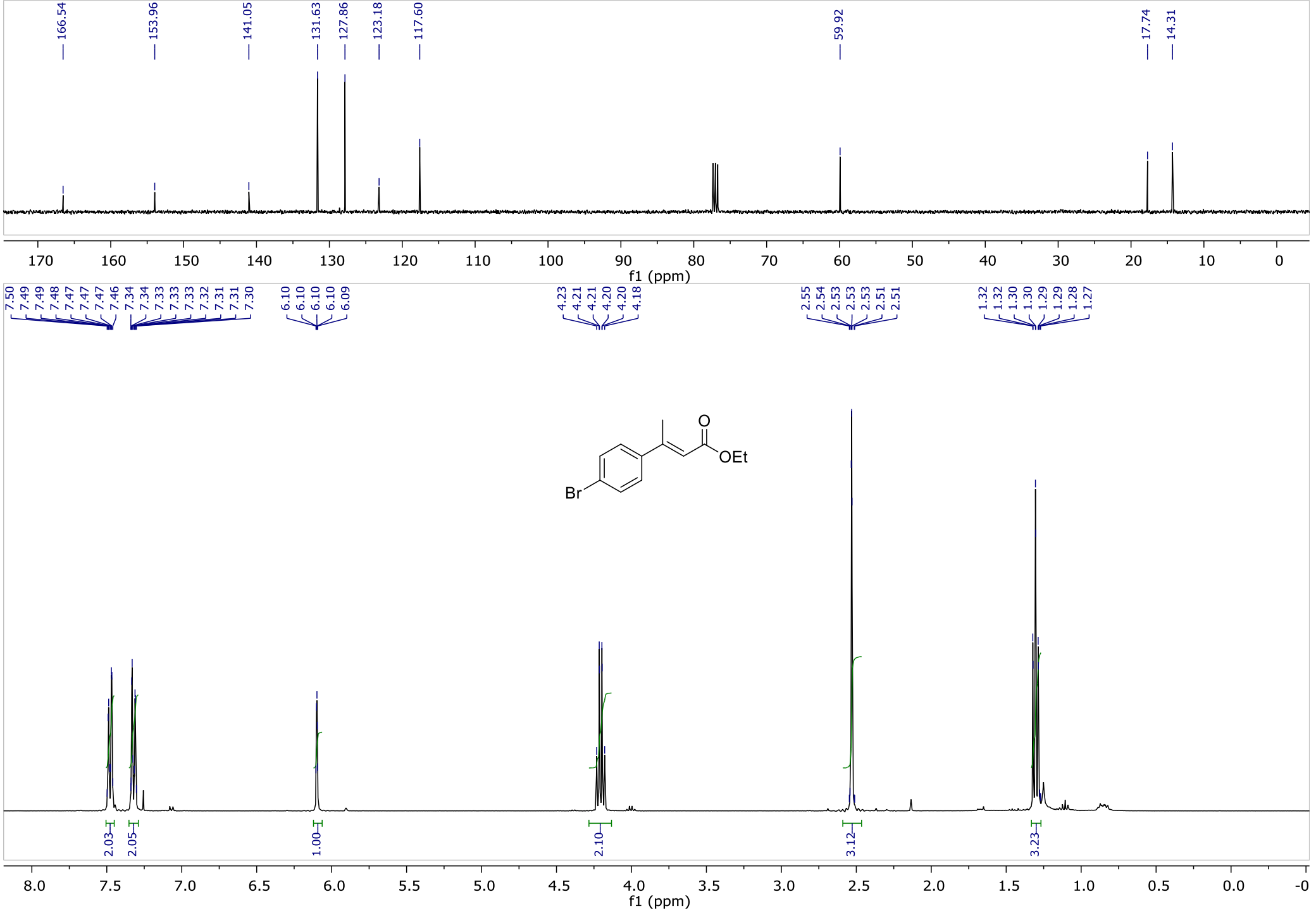
144.70
133.00
129.11
128.84
127.33
120.66
120.52
59.94
20.34
14.28
f1 (ppm)
O
OEt
Br
1.00
1.11
2.06
1.00
2.12
3.06
3.20
f1 (ppm)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131.63
127.86
123.18
117.60
59.92
17.74
14.31
f1 (ppm)
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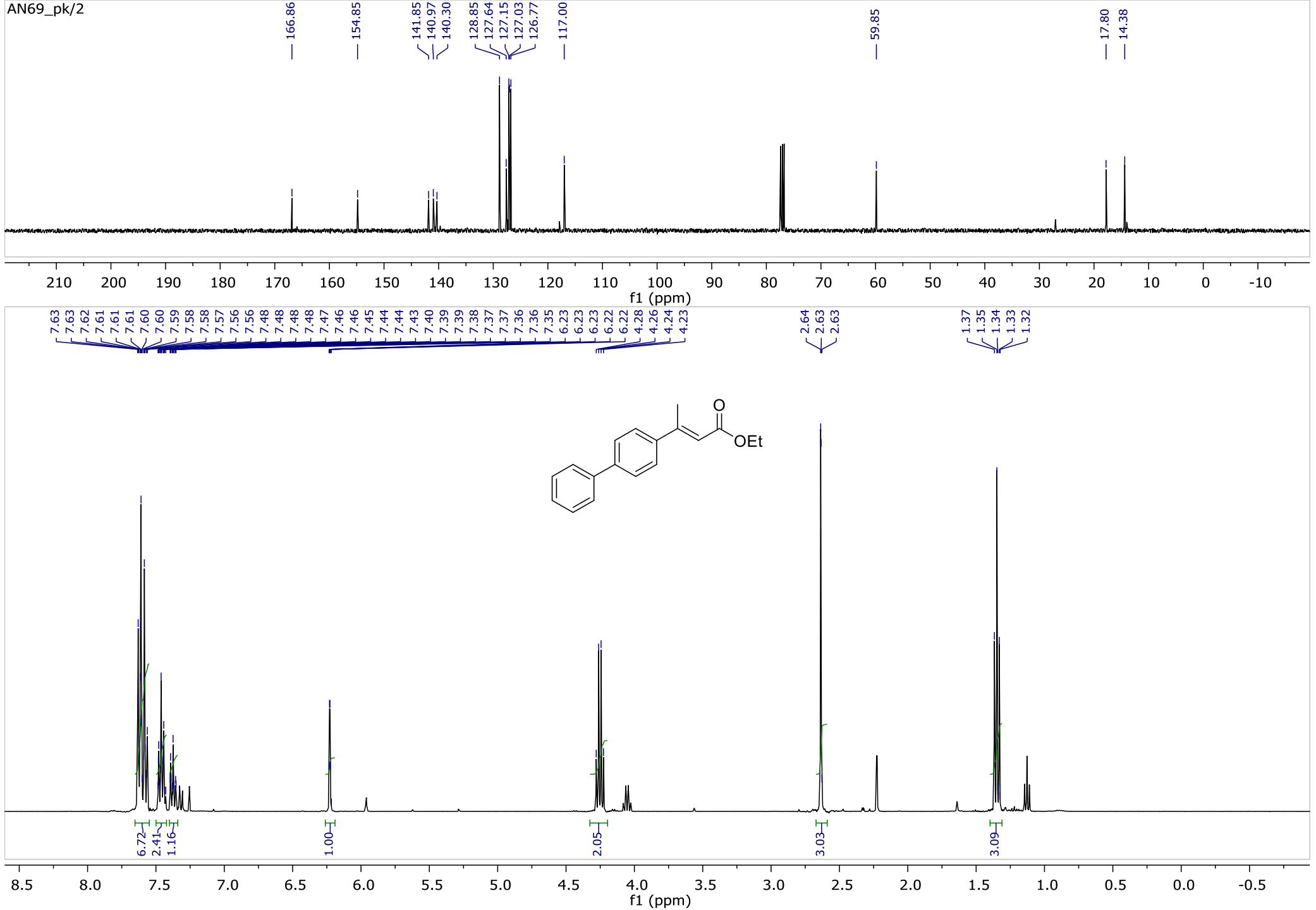
6.10 6.10 6.10 6.10 6.09
4.23 4.21 4.21 4.20 4.20 4.18
2.55 2.54 2.53 2.53 2.53 2.51 2.51
1.32 1.32 1.30 1.30 1.29 1.29 1.28 1.27
O
OEt
Br
2.03
2.05
1.00
2.10
3.12
3.23
f1 (ppm)


AN69_pk/2
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140.97
140.30
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127.64
127.15
127.03
126.77
117.00
59.85
17.80
14.38
f1 (ppm)
7.63
7.62
7.61
7.60
7.59
7.58
7.57
7.56
7.48
7.47
7.46
7.45
7.44
7.43
7.40
7.39
7.38
7.37
7.36
7.35
6.23
6.22
4.28
4.26
4.24
4.23
2.64
2.63
1.37
1.35
1.34
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6.72
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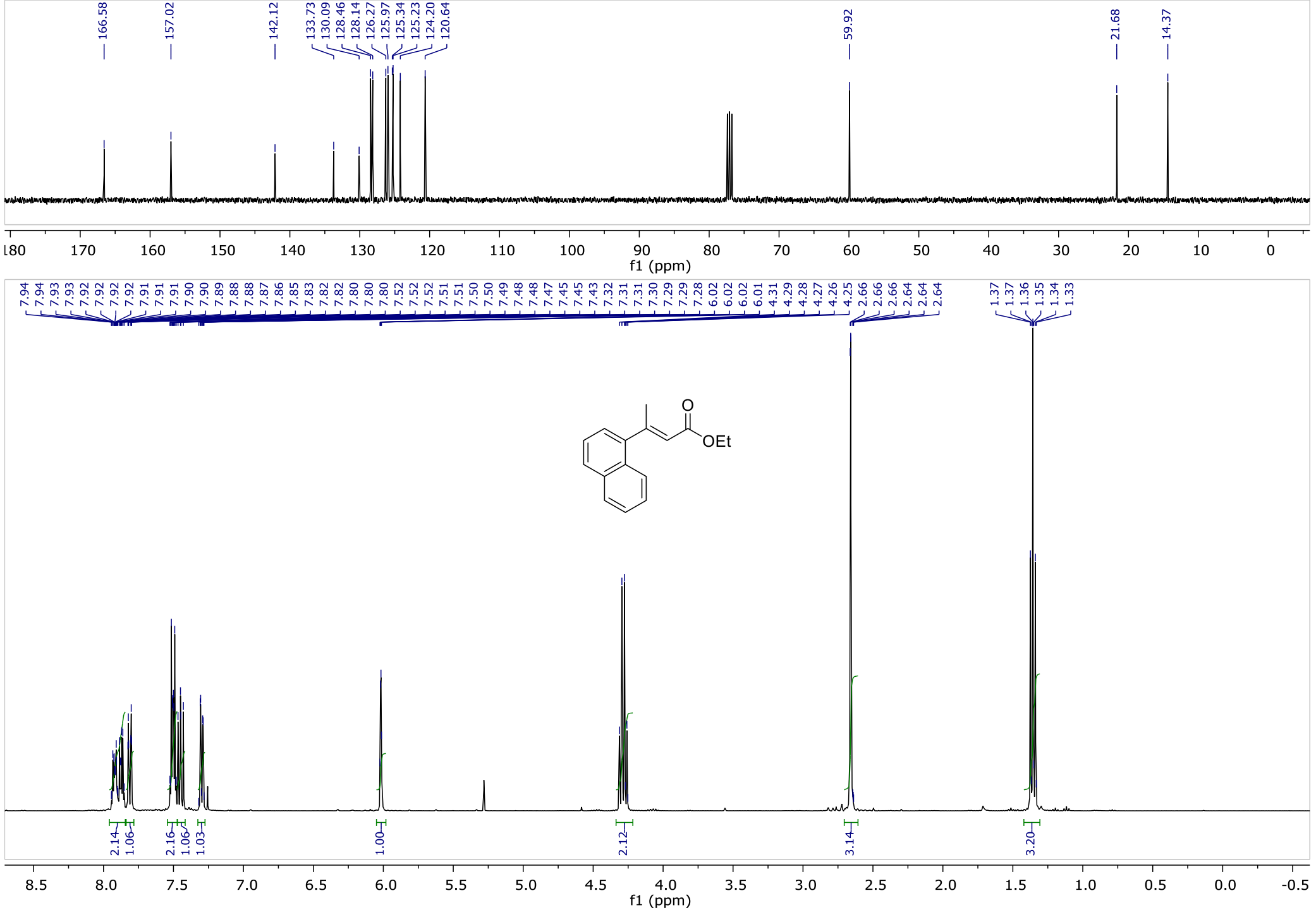
1.00
2.05
3.03
3.09
f1 (ppm)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128.46
128.14
126.27
125.97
125.34
125.23
124.20
120.64
59.92
21.68
14.37
f1 (ppm)
7.94
7.94
7.93
7.93
7.92
7.92
7.92
7.92
7.91
7.91
7.91
7.90
7.90
7.89
7.88
7.88
7.87
7.86
7.85
7.83
7.82
7.82
7.80
7.80
7.80
7.52
7.52
7.52
7.51
7.51
7.50
7.50
7.49
7.48
7.48
7.47
7.45
7.45
7.43
7.32
7.31
7.31
7.30
7.29
7.29
7.28
6.02
6.02
6.02
6.01
4.31
4.29
4.28
4.27
4.26
4.25
2.66
2.66
2.66
2.64
2.64
2.64
1.37
1.37
1.36
1.35
1.34
1.33
O
OEt
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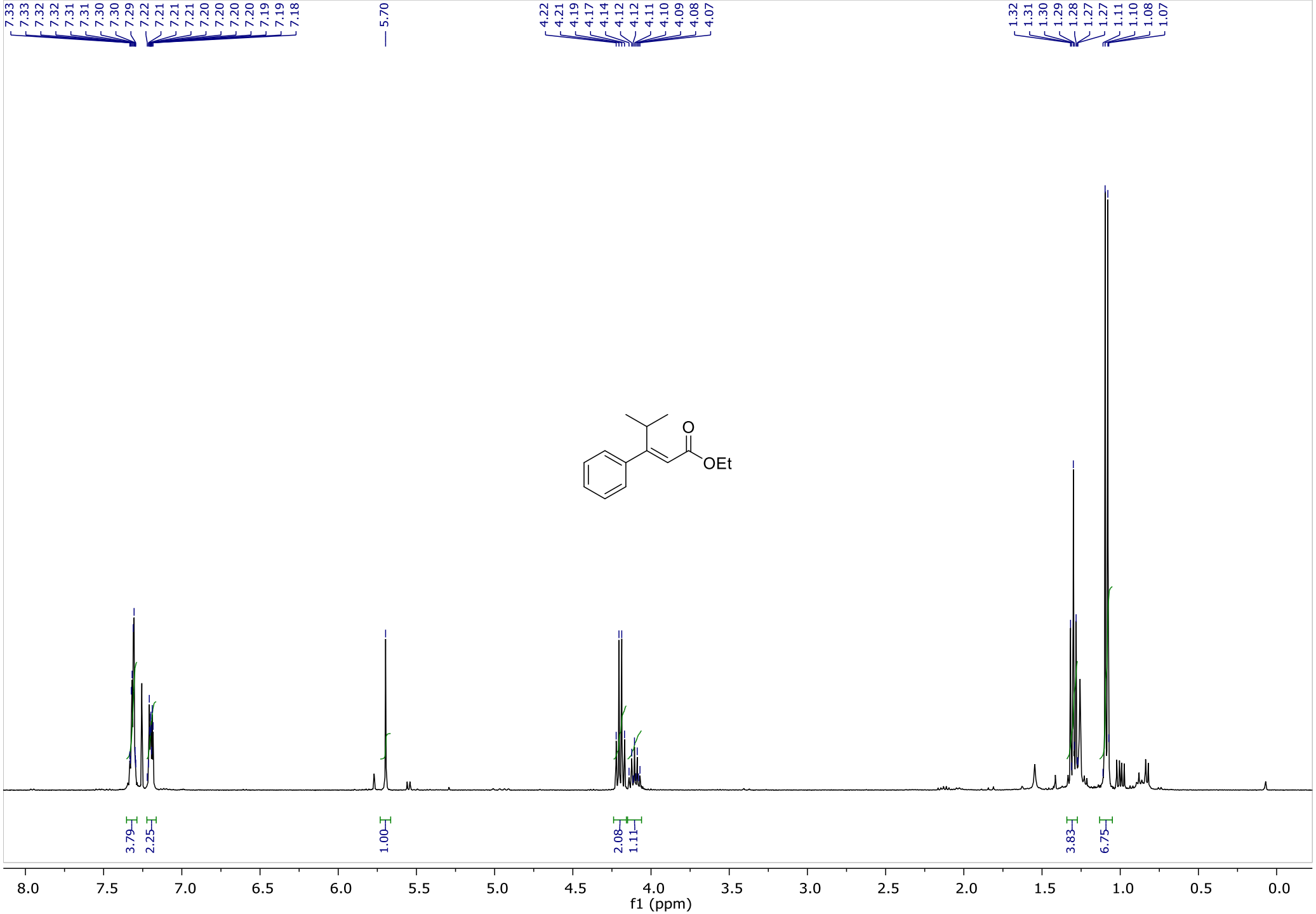
2.16
1.06
1.03
1.00
2.12
3.14
3.20
f1 (ppm)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7.33
7.32
7.32
7.31
7.31
7.30
7.30
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7.22
7.21
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7.21
7.20
7.20
7.20
7.19
7.19
7.18
5.70
4.22
4.21
4.19
4.17
4.14
4.12
4.12
4.11
4.10
4.09
4.08
4.07
1.32
1.31
1.30
1.29
1.28
1.27
1.27
1.11
1.10
1.08
1.07
O
OEt
3.79
2.25
1.00
2.08
1.11
3.83
6.75
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
f1 (ppm)