

Supporting Information

Synthesis of α,β -Unsaturated Aldehydes Based on a One Pot Phase-Switch Dehydrogenative Cross-Coupling of Primary Alcohols

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1. General Experimental Method

Commercially available reagents were used as received. Silica-grafted primary amine (Si-Amine non endcapped) specifications: particle size 40-63 µm, loading 1.73 mmol/g, lot 35964. The solvents were purchased in sure/sealedTM bottles over molecular sieves. Flash column chromatography was performed with Merck silica gel 60, particle size 0.040–0.063 mm (230–400 mesh). All reactions were monitored by thin-layer chromatography (TLC) performed on glass-backed silica gel 60 F₂₅₄, 0.2 mm plates (Merck), and compounds were visualized under UV light (254 nm) or using 2,4-dinitrophenylhydrazine (otherwise KMnO₄) solution with subsequent heating. The eluents were technical grade and distilled prior to use. Melting points were determined in a Büchi 504 apparatus. IR spectra were recorded in a Nicolet (Impact 410) FT-IR spectrophotometer using neat samples. High-resolution mass spectra (HRMS) were obtained with a JEOL JMS-700 spectrometer using electrospray (ESI). ¹H NMR spectra were recorded at 25 °C. ¹H and ¹³C liquid NMR spectra were recorded on Bruker Avance III (400 MHz) NMR spectrometer at 25 °C. Proton chemical shifts are expressed in parts per million (ppm, δ scale) and are referred to the residual hydrogen in the solvent (CHCl₃, 7.27 ppm). Data are represented as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, pent = pentuplet, sex = sextuplet, sept = septuplet, m = multiplet and/or multiple resonances, br s = broad singlet), coupling constant (*J*) in Hertz and integration. Carbon chemical shifts are expressed in parts per million (ppm, δ scale) and are referenced to the carbon resonances of the NMR solvent (CDCl₃, δ 77.0 ppm). All the experiments were carried out in duplicate to ensure reproducibility of the experimental data.

2. Microwave Irradiation Experiments

All the reactions involving microwave dielectric heating were performed with a microwave oven (Discover from CEM) under mono-mode irradiation in a 10-mL glass tube sealed with a CEM's proprietary "snap-on" cap. The microwave heating was carried out at 120 °C using "dynamic power" mode with maximum power of 250 W for 3 hrs and operating at a frequency of 2.45GHz. The reaction mixtures were stirred with a magnetic stir bar during the irradiation. During the course of reactions, the internal temperature was monitored through an IR sensor (standard infrared temperature sensor). The maximal internal pressure was monitored and maintained under the value of 300 psi using the provided software. At the end of the reaction, the tube was cooled to room temperature with air-compressed jet cooling.

3. Table S1

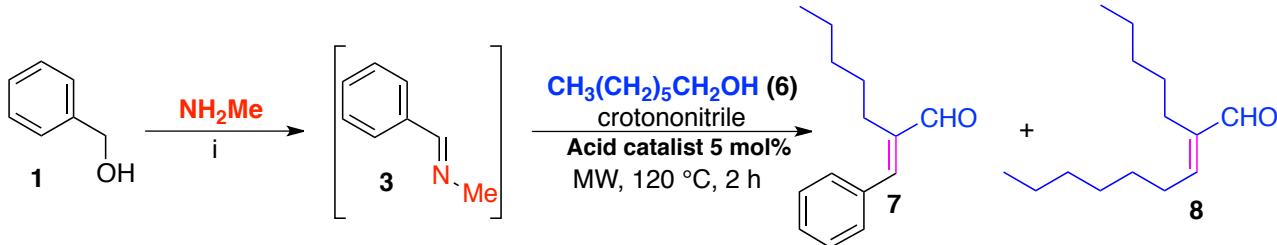
Synthesis of Imine **3**: an Assessment of the Reaction Conditions

entry	catalyst	ligand	T (°C)	conv. (%) ^b
1	RuH ₂ (PPh ₃) ₄	-	150	42
2	RuHCl(CO)(PPh ₃) ₃	-	150	54
3	[Ru(<i>p</i> -cymene)Cl ₂] ₂	-	150	49
4	[Ru ₃ (CO) ₁₂]	-	150	52
5	RuH ₂ CO(PPh ₃) ₃	-	150	63
6	RuH ₂ CO(PPh ₃) ₃	-	150	49 ^c
7	RuH ₂ CO(PPh ₃) ₃	-	120	61
8	RuH ₂ CO(PPh ₃) ₃	-	110	50
9	RuH ₂ CO(PPh ₃) ₃	PCy ₃ ^d	120	83
10	RuH ₂ CO(PPh ₃) ₃	cataCXium ^e	120	74
11	RuH ₂ CO(PPh ₃) ₃	dppf ^f	120	81
12	RuH ₂ CO(PPh ₃) ₃	Xantphos ^g	120	97
13	RuH ₂ CO(PPh ₃) ₃	Xantphos ^g	120	97 ^h

^aReaction conditions: benzyl alcohol **1** (1.0 mmol), methylamine **2** (1.3 mmol), crotononitrile (1.1 mmol), catalyst (4 mol%) in THF (2.0 mL) at the indicated temperature for 24 h. Unless otherwise specified, the reactions were carried out in a closed vessel inserted in a preheated oil bath. ^bConversion into imine **3** as determined by ¹H NMR spectroscopic analysis. ^cThe catalyst loading has been reduced to 3 mol%. ^d4 mol % of ligand have been added. ^e4 mol% of cataCXium (Di-adamantylalkylphosphine) have been used. ^f4 mol % of dppf [1,1'-Bis(diphenylphosphino)ferrocene] has been used. ^g4 mol % of Xantphos (4,5-Bis(diphenylphosphino)-9,9-dimethylxanthene) have been used. ^hReaction performed under microwave dielectric heating at 120 °C for 1h.

4. Table S2

Scheme 3. Screening of Different Acid Catalysts in the Chemoselective Dehydrogenative Cross-Coupling Reaction of the Benzyl Alcohol **1** with Heptanol **6** via a Cascade Catalytic Sequence.



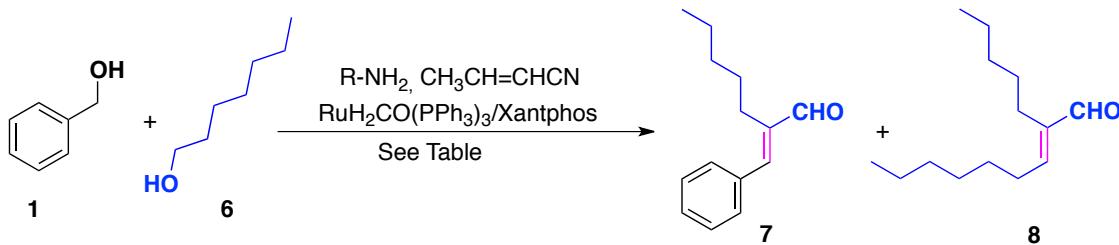
i: RuH₂CO(PPh₃)₃/Xantphos (4 mol%), CH₃CH=CHCN, acid catalyst (10 mol%), THF, MW, 120 °C, 1 h

Entry ^a	Acid catalyst	cross-coupling product (7): self-coupling product (8) ^b
1	pTsOH	78:22
2	CH ₃ COOH	80:20
3	tBuCOOH	81:19
4	CH ₃ CH ₂ COOH	82:18
5	CH ₃ CH ₂ COOH (10 mol%)	83:17
6	Silica gel ^c (50 mg)	82:18
7	Silica gel^c (100 mg)	85:15
8	Silica gel ^c (150 mg)	86:14

^aRatio imine **3**/alcohol **6** = 2/1. ^bRatio determined by ¹H NMR spectroscopy analysis. ^cReaction performed by adding silica gel with particle size 40-63 µm.

5. Table S3

Chemoselective Dehydrogenative Cross-Coupling Reaction of the Benzyl Alcohol **1** with Heptanol **6** via a One-Pot Reaction Sequence: Fine-tuning of the Reaction Conditions.^a



entry	Amine	Ratio 1/6 ^b	Cat. (mol %)	Solvent	T (°C)	t (h)	Conv. 6 (%) ^c	Ratio 7/8	Yield 7 (%) ^d
1	MeNH ₂ (2.0 mmol)	2/1	4	THF	130	3	< 10	-	-
2	MeNH ₂ (2.0 mmol)	2/1	4	THF	130	6	15	-	-
3	MeNH ₂ (0.5 mmol)	2/1	4	THF	130	3	100	76/24	40
4	MeNH ₂ (0.25 mmol)	2/1	4	THF	130	6	100	66/44	28
5	(Si)-NH ₂ (0.4 mmol) ^e	2/1	4	neat	130	3	>90	75/25	35
6	(Si)-NH ₂ (0.6 mmol)	2/1	4	neat	130	3	>90	77/23	44
7	(Si)-NH ₂ (0.9 mmol)	2/1	4	neat	130	3	>90	83/17	60
8	(Si)-NH ₂ (1.5 mmol)	2/1	4	neat	130	3	51	-	-
9	(Si)-NH ₂ (0.9 mmol)	3/1	4	neat	130	3	100	90/10	70
10	(Si)-NH ₂ (0.9 mmol)	3/1	4	neat	120	3	100	90/10	75
11	(Si)-NH ₂ (0.9 mmol)	3/1	2	neat	120	3	100	90/10	75
12	(Si)-NH ₂ (0.9 mmol)	3/1	2	neat	110	3	88	88/12	59
13	(Si)-NH ₂ (0.9 mmol)	3/1	2	neat	120	2	72	85/15	-
14	no amine	3/1	2	neat	120	3	<5	-	-
15	(Si)-NH ₂ (0.9 mmol)	3/1	2	toluene	120	3	<5	-	-
16	(Si)-NH ₂ (0.9 mmol)	3/1	2	THF	120	3	<5	-	-

^aReaction performed under microwave dielectric heating at the stated temperature using **RuH₂CO(PPh₃)₃/Xantphos** as catalyst and crotononitrile (5.0 mmol) as hydrogen acceptor. ^bHeptanol (1.0 mmol). ^cConversion determined by ¹H NMR spectroscopy analysis with an internal standard (2,5-dimethylfuran). ^dYields of isolated product after column chromatography. ^eReaction performed by using amine-grafted silica gel with particle size 40–63 µm.

6. Silica-grafted amine recycling

The reusability of the silica-grafted amine was studied by using “*Si-Amine non endcapped*” in five consecutive dehydrogenative cross-coupling experiments of benzyl alcohol **1** with heptanol **2**, under the optimised reaction condition. For this purpose, at the end of each reaction, the solid supported amine was recovered by filtration, washed with CH₂Cl₂ (3x5 mL), dried under vacuum at 40 °C overnight, and then subjected to the next run. The reaction was repeated with further addition of substrates in appropriate amounts under optimum reaction conditions. The results summarized in Table S4 show that the silica-grafted amine can be reused at least 5 consecutive cycles without significant loss of its efficiency.

Table S4

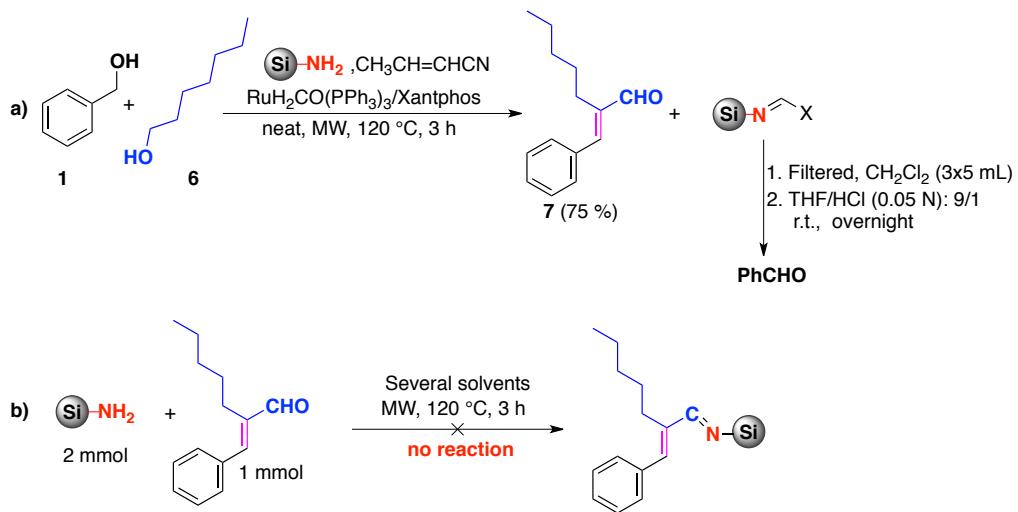
Reusability of the catalyst^a

Reaction cycles	Conversion (%) ^b	Ratio 7/8	Yield 7 (%) ^c
Fresh	100	90/10	75
1	100	92/08	73
2	100	90/10	71
3	100	91/9	72
4	100	89/11	70
5	100	90/10	71

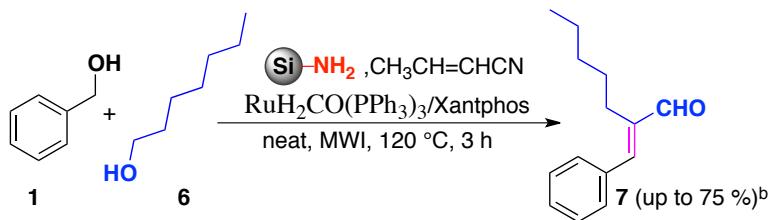
^aReaction condition: benzyl alcohol (3.0 mmol), 1-heptanol (1.0 mmol), crotononitrile (5.0 mmol), (Si)-NH₂ (0.9 mmol), RuH₂CO(PPh₃)₃/Xantphos (2 mol%), solvent-free, 120 °C, 3 h. ^bConversion of 1-heptanol **6** determined by ¹H NMR spectroscopy analysis. ^cYields of isolated product after column chromatography.

7. Analysis of the Solid Support (Silica gel) After Dehydrogenative Cross-Coupling Reaction Between Benzyl Alcohol 1 and Heptanol 6 Via a One-Pot Reaction Sequence.

In order to determine the presence of any residual unsaturated aldehyde on the solid support at the end of the cross-coupling reaction, the resulting silica-grafted amine was first filtered off and washed several times with DCM (3x5 mL) and then treated with an acid aqueous solution THF/HCl (0.05 N): 9/1 (5 ml). The mixture has shaken overnight at room temperature releasing in solution only benzaldehyde (Scheme 7a). Even 24 h after, there were no detectable amounts of the compound **7** in the solution. In a separate experiment, the unsaturated aldehyde **7** (1 mmol) was treated with an excess of fresh silica-grafted amine (2 mmol) under the optimised reaction condition described for the cross-coupling reaction between benzyl alcohol **1** and heptanol **6** (Scheme 7b). Even after a prolonged reaction time (overnight), the compound **7** was almost quantitatively recovered from the solution. Any attempt of end-attachment of the unsaturated aldehyde **7** to solid-supported failed even when more suitable solvents (THF, Toluene etc) were used. FT-IR spectra on the solid support showed the absence of a C=N band on the silica gel.

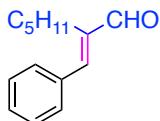


8. General Procedure for the preparation of α,β -Unsaturated Aldehydes

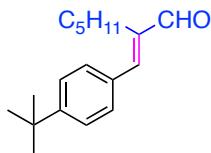


The model reaction of benzyl alcohol with 1-heptanol is described here to exemplify the general reaction procedure. $\text{RuH}_2(\text{CO})(\text{PPh}_3)_3$ (0.055 g, 0.06 mmol) and Xantphos (0.035 g, 0.06 mmol) were added to an oven dried 10-mL microwave reaction vessel, followed by crotononitrile (0.33 g, 0.40 ml, 5.0 mmol), benzyl alcohol (0.32 g, 0.31 ml, 3.0 mmol), 1-heptanol (0.12 g, 0.14 ml, 1.0 mmol) and grafted amine (0.520 g, 0.9 mmol). The tube was then sealed under nitrogen or argon and the reaction mixture was irradiated at 120°C for 3 hrs in a microwave oven. After completion of the reaction, the silica-grafted amine was filtered out and washed with CH_2Cl_2 (3×5 mL). The collected filtrate was condensed under reduced pressure to get the crude product, which was analysed by ^1H NMR spectroscopy integrating the aldehyde CHO peaks. The crude mixture was purified by chromatography on a silica gel column using hexane/ethyl acetate: 9/1 as eluent to give α,β -unsaturated aldehyde 7 (0.152 g, 75%). All products prepared by the above procedure were characterized spectroscopically as shown below.

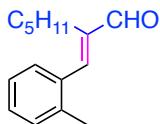
9. Characterization data for compounds 7-37.



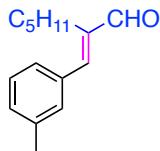
(E)-2-benzylideneheptanal (7). Colourless oil (152 mg, 75 %); $R_f = 0.43$ (hexane/AcOEt: 9/1). IR (film) cm^{-1} : 2955, 2928, 2858, 2711, 1682, 1624, 1455, 1088. $^1\text{H-NMR}$ (400 MHz; CDCl_3): $\delta = 9.54$ (s, 1H), 7.50-7.38 (m, ArH, 5H), 7.20 (s, 1H), 2.52 (t, $J = 7.6$ Hz, 2H), 1.54-1.46 (m, 2H), 1.38-1.31 (m, 4H), 0.89 (t, $J = 7.2$ Hz, 3H). $^{13}\text{C-NMR}$ (100 MHz; CDCl_3): $\delta = 195.6, 149.7, 143.3, 134.9, 129.6, 129.5, 128.7, 32.0, 27.9, 24.7, 22.3, 13.9$. These assignments matched with those previously published.¹



(E)-2-(4-(Tert-butyl)benzylidene)heptanal (9). A colourless oil (147 mg, 57 %); $R_f = 0.54$ (hexane/AcOEt: 9/1); IR (film) cm^{-1} : 2963, 2928, 2858, 1681, 1619, 1453, 1081. $^1\text{H-NMR}$ (400 MHz; CDCl_3): $\delta = 9.53$ (s, 1H), 7.47 (s, 4H), 7.17 (s, 1H), 2.54 (t, $J = 8.0$ Hz, 2H), 1.53-1.47 (m, 2H), 1.35 (bs, 13H), 0.90 (t, $J = 6.8$ Hz, 3H). $^{13}\text{C-NMR}$ (100 MHz; CDCl_3): $\delta = 195.8, 153.2, 149.8, 142.7, 132.2, 129.8, 125.8, 34.9, 32.2, 31.2, 28.0, 24.8, 22.5, 14.1$. HRMS: m/z calcd for $C_{18}H_{27}O$: 259.2062 [$M+H]^+$. Found: 259.2065.

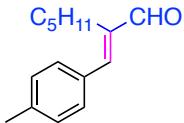


(E)-2-(2-methylbenzylidene)heptanal (10). A pale yellow oil (138 mg, 64 %); $R_f = 0.56$ (hexane/AcOEt: 9/1); IR (film) cm^{-1} : 2928, 2858, 2710, 1686, 1624, 1458, 1372, 1292, 1087. $^1\text{H-NMR}$ (400 MHz; CDCl_3): $\delta = 9.61$ (s, 1H), 7.39 (s, 1H), 7.30-7.24 (m, 4H), 2.37 (t, $J = 7.9$ Hz, 2H), 2.33 (s, 3H), 1.46-1.39 (m, 2H), 1.26-1.24 (m, 4H), 0.84 (t, $J = 6.7$ Hz, 3H). $^{13}\text{C-NMR}$ (100 MHz; CDCl_3): $\delta = 195.6, 148.8, 144.2, 136.7, 134.2, 130.4, 129.1, 128.2, 125.9, 31.9, 28.2, 24.8, 22.3, 20.0, 14.0$. HRMS: m/z calcd for $C_{15}H_{21}O$: 217.1592 [$M+H]^+$. Found: 217.1597.

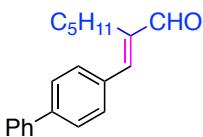


(E)-2-(3-methylbenzylidene)heptanal (11). A light yellow oil (134 mg, 62 %); $R_f = 0.55$ (hexane/AcOEt: 9/1); IR (film) cm^{-1} : 2927, 1681, 1624, 1172, 1086, 784; $^1\text{H-NMR}$ (400 MHz; CDCl_3): $\delta = 9.53$ (s, 1H), 7.34-7.31 (m, 3H), 7.21 (d, $J = 6.8$ Hz, 1H), 7.18 (s, 1H), 2.54-2.50 (m, 2H), 2.40 (s, 3H), 1.54-1.46 (m, 2H), 1.40-1.32 (m, 4H), 0.90 (t, $J = 7.0$ Hz, 3H). $^{13}\text{C-NMR}$ (100

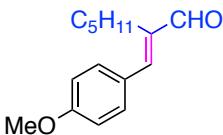
MHz; CDCl₃): δ = 195.8, 150.0, 143.3, 138.4, 135.0, 130.5, 130.3, 128.7, 126.7, 32.1, 28.0, 24.8, 22.4, 21.5, 14.0. These assignments matched with those previously published.³



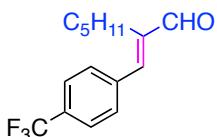
(E)-2-(4-Methylbenzylidene)heptanal (12). A colourless oil (132 mg, 61 %); R_f = 0.55 (hexane/AcOEt: 9/1); IR (film) cm⁻¹: 2963, 2927, 2864, 2715, 1681, 1619, 1458, 1083; ¹H-NMR (400 MHz; CDCl₃): δ = 9.52 (s, 1H), 7.41 (d, J = 8.1 Hz, 2H), 7.26 (d, J = 8.1 Hz, 2H), 7.16 (s, 1H), 2.53 (t, J = 8.0 Hz, 2H), 2.40 (s, 3H), 1.53-1.46 (m, 2H), 1.39-1.32 (m, 4H), 0.90 (t, J = 6.9 Hz, 3H). ¹³C-NMR (100 MHz; CDCl₃): δ = 195.8, 150.0, 142.6, 140.0, 132.2, 129.8, 129.6, 32.1, 27.9, 24.8, 22.4, 21.5, 14.0. HRMS (ESI): m/z calcd for C₁₅H₂₁O: 217.1592 [M+H]⁺. Found: 217.1589.



(E)-2-((1,1'-Biphenyl)-4-ylmethylene)heptanal (13). A light yellow oil (153 mg, 55 %); R_f = 0.42 (hexane/AcOEt: 9/1); IR (film) cm⁻¹: 2946, 2857, 1679, 1615, 1487, 1286, 1085. ¹H-NMR (400 MHz; CDCl₃): δ = 9.57 (s, 1H), 7.69 (d, J = 8.2 Hz, 2H), 7.64 (d, J = 8.2 Hz, 2H), 7.59 (d, J = 8.2 Hz, 2H), 7.47 (t, J = 7.6 Hz, 2H), 7.41-7.39 (m, 1H), 7.23 (s, 1H), 2.58 (t, J = 8.0 Hz, 2H), 1.54-1.52 (m, 2H), 1.42-1.35 (m, 4H), 0.91 (t, J = 6.8 Hz, 3H). ¹³C NMR (100 MHz; CDCl₃): δ = 195.7, 149.3, 143.3, 142.3, 140.1, 134.0, 130.3, 129.0, 127.9, 127.4, 127.1, 32.2, 28.0, 24.9, 22.5, 14.1. HRMS: m/z calcd for C₂₀H₂₃O: 279.1749 [M+H]⁺. Found: 279.1745.

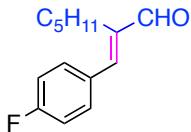


(E)-2-(4-Methoxybenzylidene)heptanal (14). An amber oil (112 mg, 48 %); R_f = 0.28 (hexane/AcOEt: 9/1); IR (film) cm⁻¹: 2972, 2870, 1682, 1615, 1170, 1087, 1031. ¹H-NMR (400 MHz; CDCl₃): δ = 9.49 (s, 1H), 7.49 (d, J = 8.7 Hz, 2H), 7.12 (s, 1H), 6.97 (d, J = 8.7 Hz, 2H), 3.86 (s, 3H), 2.54 (t, J = 7.9 Hz, 2H), 1.53-1.46 (m, 2H), 1.40-1.33 (m, 4H), 0.90 (t, J = 6.8 Hz, 3H). ¹³C-NMR (100 MHz; CDCl₃): δ = 195.7, 160.8, 149.7, 141.3, 131.8, 127.7, 114.3, 55.4, 32.2, 27.8, 24.7, 22.5, 14.1. These assignments matched with those previously published.²



(E)-2-(4-(Trifluoromethyl)benzylidene)heptanal (15). A pale yellow oil (151 mg, 56 %); R_f = 0.31 (hexane/AcOEt: 9/1); IR (film) cm⁻¹: 2958, 2931, 1687, 1324, 1167, 1128, 1068. ¹H-NMR

(400 MHz; CDCl₃): δ = 9.59 (s, 1H), 7.71 (d, *J* = 8.2 Hz, 2H), 7.58 (d, *J* = 8.2 Hz, 2H), 7.24 (s, 1H), 2.50 (t, *J* = 8 Hz, 2H), 1.51-1.47 (m, 2H), 1.36-1.32 (m, 4H), 0.89 (t, *J* = 6.9 Hz, 3H). ¹³C-NMR (100 MHz; CDCl₃): δ 195.2, 147.4, 145.2, 138.4, [131.5, 131.2, 130.9, 130.6] (q, *J* = 32.7 Hz, C-CF₃, 1C), 129.6, [125.79, 125.75, 125.72, 125.68] (q, *J* = 3.7 Hz, CF₃CC(Ar)C, 1C), [127.9, 125.2, 122.5, 119.8] (q, *J* = 270 Hz, CF₃, 1C), 32.0, 28.1, 24.8, 22.4, 14.0. ¹⁹F NMR (376 MHz; CDCl₃, decoupled): δ = -62.9. These assignments matched with those previously published.³



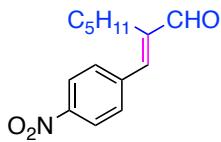
(E)-2-(4-Fluorobenzylidene)heptanal (16). A light yellow oil (161 mg, 73 %); R_f = 0.42 (hexane/AcOEt: 9/1); IR (film) cm⁻¹: 2930, 2860, 1683, 1624, 1600, 1508, 1465, 1399, 1294, 1236, 1159, 1086, 829. ¹H-NMR (400 MHz; CDCl₃): δ = 9.53 (s, 1H), 7.49 (dd, *J* = 8.6, 5.5 Hz, 2H), 7.16-7.12 (m, 3H), 2.51 (t, *J* = 8.0 Hz, 2H), 1.52-1.44 (m, 2H), 1.39-1.31 (m, 4H), 0.89 (t, *J* = 6.9 Hz, 3H). ¹³C-NMR (100 MHz; CDCl₃): δ = 195.5, [164.5, 162.0] (d, J_{C-F} = 250.0 Hz, C(Ar)-F, 1C), 148.4, 143.1, [131.7, 131.6] (d, J_{C-F} = 8.3 Hz, CH(Ar), 1C), [131.22, 131.19] (d, J_{C-F} = 3.6 Hz, C(Ar)C=C, 1C), [116.1, 115.9] (d, J_{C-F} = 21.6 Hz, F-C(Ar)CH(Ar), 1C), 32.1, 27.9, 24.7, 22.4, 14.0. ¹⁹F-NMR (376 MHz; CDCl₃, decoupled): δ = -110.3. HRMS: m/z calcd for C₁₄H₁₈FO: 221.1342 [M+H]⁺. Found: 221.1345.



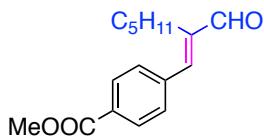
(E)-2-(4-Chlorobenzylidene)heptanal (17). A brown oil (156 mg, 66 %); R_f = 0.37 (hexane/AcOEt: 9/1); IR (film) cm⁻¹: 2957, 2931, 2862, 1703, 1592, 1490, 1092, 1014, 821. ¹H-NMR (400 MHz; CDCl₃): δ = 9.54 (s, 1H), 7.42 (s, 4H), 7.15 (s, 1H), 2.50 (t, *J* = 8.0 Hz, 2H), 1.51-1.44 (m, 2H), 1.37-1.33 (m, 4H), 0.89 (t, *J* = 6.9 Hz, 3H). ¹³C-NMR (100 MHz; CDCl₃): δ 195.4, 148.1, 143.8, 135.5, 133.4, 130.8, 129.1, 32.1, 27.9, 24.7, 22.4, 14.0. HRMS: m/z calcd for C₁₄H₁₈ClO: 237.1046 [M+H]⁺. Found: 237.1040.



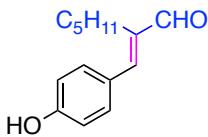
(E)-2-(4-Bromobenzylidene)heptanal (18). A light yellow oil (174 mg, 62 %); R_f = 0.37 (hexane/AcOEt: 9/1); IR (film) cm⁻¹: 2968, 2934, 2858, 1681, 1619, 1585, 1478, 1395, 1112, 1078. ¹H-NMR (400 MHz; CDCl₃): δ = 9.54 (s, 1H), 7.58 (d, *J* = 8.3 Hz, 2H), 7.35 (d, *J* = 8.4 Hz, 2H), 7.13 (s, 1H), 2.49 (t, *J* = 8.0 Hz, 2H), 1.51-1.43 (m, 2H), 1.36-1.32 (m, 4H), 0.89 (t, *J* = 6.8 Hz, 3H). ¹³C-NMR (100 MHz; CDCl₃): δ = 195.4, 148.1, 143.9, 133.9, 132.1, 131.0, 123.9, 32.1, 27.9, 24.8, 22.4, 14.0. These assignments matched with those previously published.³



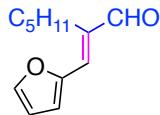
(E)-2-(4-Nitrobenzylidene)heptanal (19). A brown oil (124 mg, 50 %); $R_f = 0.18$ (hexane/AcOEt: 9/1); IR (film) cm^{-1} : 2930, 2859, 1685, 1596, 1520, 1346, 1085, 896. $^1\text{H-NMR}$ (400 MHz; CDCl_3): $\delta = 9.61$ (s, 1H), 8.31 (d, $J = 8.6$ Hz, 2H), 7.63 (d, $J = 8.7$ Hz, 2H), 7.27 (s, 1H), 2.50 (t, $J = 8.0$ Hz, 2H), 1.53-1.45 (m, 2H), 1.36-1.30 (m, 4H), 0.89 (t, $J = 6.9$ Hz, 3H). $^{13}\text{C-NMR}$ (100 MHz; CDCl_3): $\delta = 194.8, 147.8, 146.2, 146.0, 141.3, 130.0, 124.0, 32.0, 28.1, 24.9, 22.3, 14.0$. These assignments matched with those previously published.²



(E)-Methyl 4-(2-formylhept-1-en-1-yl)benzoate (20). A light yellow oil (180 mg, 69 %); $R_f = 0.23$ (hexane/AcOEt: 9/1); IR (film) cm^{-1} : 2955, 2931, 2863, 1724, 1686, 1507, 1435, 1279, 1109, 972. $^1\text{H-NMR}$ (400 MHz; CDCl_3): $\delta = 9.58$ (s, 1H), 8.11 (d, $J = 8.2$ Hz, 2H), 7.54 (d, $J = 8.2$ Hz, 2H), 7.24 (s, 1H), 3.95 (s, 3H), 2.51 (t, $J = 8$ Hz, 2H), 1.50-1.45 (m, 2H), 1.36-1.29 (m, 4H), 0.89 (t, $J = 6.9$ Hz, 3H). $^{13}\text{C-NMR}$ (100 MHz; CDCl_3): $\delta = 195.3, 166.4, 148.0, 145.0, 139.3, 130.6, 129.9, 129.4, 52.3, 32.0, 28.1, 24.8, 22.4, 14.0$. HRMS: m/z calcd for $\text{C}_{16}\text{H}_{21}\text{O}_3$: 261.1491 $[\text{M}+\text{H}]^+$. Found: 216.1497.

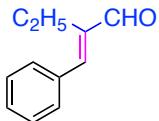


(E)-2-(4-Hydroxybenzylidene)heptanal (21). A crystalline white solid (124 mg, 57 %), m.p. 118-120 °C; $R_f = 0.5$ (hexane/AcOEt: 7/3); IR (film) cm^{-1} : 3241, 2921, 1654, 1600, 1574, 1509, 1285, 1230, 1176, 1084. $^1\text{H-NMR}$ (400 MHz; CDCl_3): $\delta = 9.49$ (s, 1H), 7.45 (d, $J = 8.5$ Hz, 2H), 7.12 (s, 1H), 6.92 (d, $J = 8.6$ Hz, 2H), 5.68 (bs, Ar-OH, 1H), 2.54 (t, $J = 8.0$ Hz, 2H), 1.51-1.46 (m, 2H), 1.42-1.29 (m, 4H), 0.90 (t, $J = 6.9$ Hz, 3H). $^{13}\text{C-NMR}$ (100 MHz; CDCl_3): $\delta = 196.1, 157.2, 150.2, 141.2, 132.0, 127.7, 115.9, 32.1, 27.8, 24.7, 22.5, 14.0$. HRMS: m/z calcd for $\text{C}_{14}\text{H}_{18}\text{O}_2\text{Na}$: 241.1204 $[\text{M}+\text{Na}]^+$. Found: 241.1209.

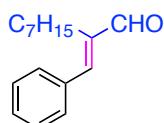


(E)-2-(Furan-2-ylmethylen)eptanal (22). A light yellow oil (81 mg, 42 %); $R_f = 0.40$ (hexane/AcOEt: 9/1); IR (film) cm^{-1} : 2968, 2929, 2866, 2717, 1678, 1625, 1472, 1384, 1366, 1279, 1088, 1026, 886. $^1\text{H-NMR}$ (400 MHz; CDCl_3): $\delta = 9.47$ (s, 1H), 7.61 (d, $J = 1.5$ Hz, 1H), 6.94 (s, 1H), 6.75 (d, $J = 3.5$ Hz, 1H), 6.56 (dd, $J = 3.2, 1.8$ Hz, 1H), 2.62 (t, $J = 7.7$ Hz, 2H), 1.47-1.43 (m, 2H), 1.36-1.33 (m, 4H), 0.89 (t, $J = 6.9$ Hz, 3H). $^{13}\text{C-NMR}$ (100 MHz; CDCl_3): $\delta = 194.4, 151.4, 145.3, 140.1, 135.1, 116.4, 112.6, 32.1, 28.0, 24.8, 22.5, 14.0$. HRMS: m/z calcd for $\text{C}_{12}\text{H}_{17}\text{O}_2$:

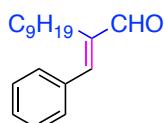
193.1229 [M+H]⁺. Found: 193.1226.



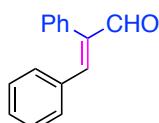
(E)-2-Benzylidenebutanal (23). A light yellow oil (93 mg, 58 %); R_f = 0.42 (hexane/AcOEt: 9/1); IR (film) cm^{-1} : 2943, 2929, 1677, 1602, 1510, 1258, 1176, 1031. ¹H-NMR (400 MHz; CDCl₃): δ = 9.55 (s, 1H), 7.52-7.40 (m, 5H), 7.21 (s, 1H), 2.57 (q, J = 7.5 Hz, 2H), 1.15 (t, J = 7.5 Hz, 3H). ¹³C NMR (100 MHz; CDCl₃): δ = 195.6, 149.6, 144.5, 134.9, 129.65, 129.58, 128.8, 18.1, 12.9. These assignments matched with those previously published.⁴



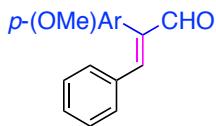
(E)-2-Benzylidenenonanal (24). A light yellow oil (166 mg, 72 %); R_f = 0.48 (hexane/AcOEt: 9/1); IR (film) cm^{-1} : 2951, 2925, 2854, 1687, 1602, 1506, 1465, 1095, 870. ¹H-NMR (400 MHz; CDCl₃): δ = 9.58 (s, 1H), 7.55-7.42 (m, 5H), 7.23 (s, 1H), 2.56 (t, J = 7.8 Hz, 2H), 1.53-1.50 (m, 2H), 1.35-1.30 (m, 8H), 0.91 (t, J = 6.7 Hz, 3H). ¹³C-NMR (100 MHz; CDCl₃): δ = 195.7, 149.8, 143.4, 135.0, 129.7, 129.5, 128.8, 31.8, 29.9, 29.0, 28.3, 24.8, 22.7, 14.1. These assignments matched with those previously published.⁵



(E)-2-Benzylideneundecanal (25). A light yellow oil (178 mg, 69 %); R_f = 0.56 (hexane/AcOEt: 9/1); IR (film) cm^{-1} : 2924, 2854, 1683, 1624, 1464, 1082, 755. ¹H-NMR (400 MHz; CDCl₃): δ = 9.56 (s, 1H), 7.46 (ddd, J = 22.6, 15.1, 7.3 Hz, 5H), 7.22 (s, 1H), 2.54 (t, J = 7.9 Hz, 2H), 1.54-1.46 (m, 2H), 1.40-1.36 (m, 2H), 1.34-1.22 (m, J = 15.6 Hz, 10H), 0.89 (t, J = 6.7 Hz, 3H). ¹³C-NMR (100 MHz; CDCl₃): δ = 195.7, 149.8, 143.4, 135.0, 129.7, 129.5, 128.8, 31.9, 29.9, 29.5, 29.4, 29.3, 28.3, 24.8, 22.7, 14.1. HRMS: *m/z* calcd for C₁₈H₂₇O: 259.2062 [M+H]⁺. Found: 259.2066.



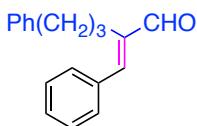
(E)-2,3-diphenylacrylaldehyde (26). A crystalline white solid (123 mg, 59 %), m.p. 64-66 °C; R_f = 0.26 (hexane/AcOEt: 9/1); IR (film) cm^{-1} : 3048, 2847, 2738, 1666, 1625, 1597, 1499, 1447, 1094, 1070. ¹H-NMR (400 MHz; CDCl₃): δ = 9.78 (s, 1H), 7.40 (dt, J = 6.4, 3.5 Hz, 4H), 7.28 (dd, J = 5.5, 2.6 Hz, 1H), 7.23 (s, 1H), 7.21-7.19 (m, 5H). ¹³C NMR (100 MHz; CDCl₃): δ = 193.9, 150.2, 141.8, 134.1, 133.4, 130.8, 130.3, 129.4, 128.9, 128.5, 128.3. These assignments matched with those previously published.⁶



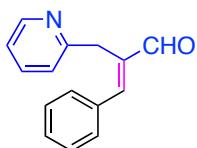
(E)-2-(4-Methoxyphenyl)-3-phenylacrylaldehyde (27). A yellow solid (117 mg, 49 %), m.p. 90-92 °C (lit.,⁷ mp 91–91.5 °C); R_f = 0.16 (hexane/AcOEt: 9/1); IR (film) cm^{-1} : 2932, 2851, 1691, 1513, 1246, 1082. $^1\text{H-NMR}$ (400 MHz; CDCl_3): δ = 9.75 (s, 1H), 7.34 (s, 1H), 7.28-7.24 (m, 5H), 7.13 (d, J = 8.5 Hz, 2H), 6.94 (d, J = 8.5 Hz, 2H), 3.84 (s, 3H). $^{13}\text{C-NMR}$ (100 MHz; CDCl_3): δ = 194.3, 159.6, 149.9, 141.4, 134.3, 130.7, 130.6, 130.1, 128.5, 125.2, 114.4, 55.3. These assignments matched with those previously published.⁷



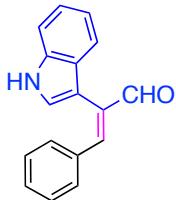
(E)-2-Benzyl-3-phenylacrylaldehyde (28). A light yellow oil (120 mg, 54 %); R_f = 0.3 (hexane/AcOEt: 9/1); IR (film) cm^{-1} : 3027, 1680, 1624, 1494, 1451, 1144, 1085. $^1\text{H-NMR}$ (400 MHz; CDCl_3): δ = 9.68 (s, 1H), 7.49-7.45 (m, 3H), 7.37 (t, J = 3.1 Hz, 3H), 7.26 (t, J = 7.6 Hz, 2H), 7.17 (t, J = 8.6 Hz, 3H), 3.94 (s, 2H). $^{13}\text{C-NMR}$ (100 MHz; CDCl_3): δ 195.2, 151.6, 140.5, 138.4, 134.6, 130.0, 129.8, 128.9, 128.7, 128.0, 126.3, 30.5. These assignments matched with those previously published.⁸



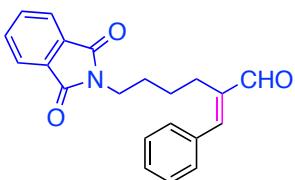
(E)-2-benzylidene-5-phenylpentanal (29). A light yellow oil (150 mg, 60 %); R_f = 0.4 (hexane/AcOEt: 9/1); IR (film) cm^{-1} : 3111, 3055, 2833, 1680, 1612, 1498, 1449, 1195, 1125, 1083, 750. $^1\text{H-NMR}$ (400 MHz; CDCl_3): δ 9.54 (s, 1H), 7.35-7.32 (m, 5H), 7.31-7.26 (m, 2H), 7.20-7.18 (m, 4H), 2.71 (t, J = 7.4 Hz, 2H), 2.58-2.54 (m, 2H), 1.86-1.79 (m, 2H). $^{13}\text{C-NMR}$ (100 MHz; CDCl_3): δ = 195.7, 150.0, 142.8, 141.8, 134.7, 129.7, 129.6, 128.80, 128.6, 128.4, 125.9, 36.0, 29.7, 24.2. HRMS: m/z calcd for $\text{C}_{18}\text{H}_{19}\text{O}$: 251.1436 [$\text{M}+\text{H}]^+$. Found: 251.1431.



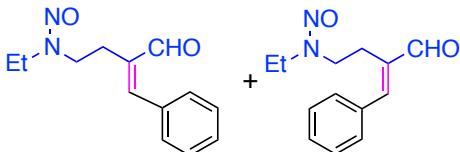
(E)-3-Phenyl-2-(pyridin-2-ylmethyl)acrylaldehyde (30). A light yellow oil (116 mg, 52 %); R_f = 0.13 (hexane/AcOEt: 9/1); IR (film) cm^{-1} : 3069, 3037, 2958, 2905, 1679, 1625, 1424, 1274, 1089, 712. $^1\text{H-NMR}$ (400 MHz; CDCl_3): δ = 9.69 (s, 1H), 8.45-8.43 (m, 2H), 7.55 (s, 1H), 7.45-7.40 (m, 6H), 7.18 (dd, J = 7.9, 4.8 Hz, 1H), 3.93 (s, 2H). $^{13}\text{C-NMR}$ (100 MHz; CDCl_3): δ = 194.8, 152.0, 149.7, 147.8, 139.6, 135.5, 134.20, 134.00, 130.2, 129.6, 129.0, 123.5, 27.9. HRMS: m/z calcd for $\text{C}_{15}\text{H}_{14}\text{NO}$: 224.1075 [$\text{M}+\text{H}]^+$. Found: 224.1069.



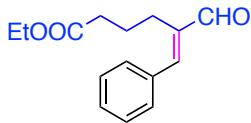
(E)-2-(1H-Indol-3-yl)-3-phenylacrylaldehyde (31). A brown oil (124 mg, 50 %); $R_f = 0.28$ (hexane/AcOEt: 8/2); IR (film) cm^{-1} : 3378, 3055, 2833, 1672, 1639, 1528, 1014, 853, 744. $^1\text{H-NMR}$ (400 MHz; CDCl_3): $\delta = 9.85$ (s, 1H), 8.49 (bs, 1H), 7.48 (s, 1H), 7.38-7.34 (m, 4H), 7.26-7.22 (m, 1H), 7.19-7.14 (m, 3H), 6.92 (s, 1H), 6.91 (s, 1H). $^{13}\text{C NMR}$ (100 MHz; CDCl_3): δ 194.8, 149.5, 136.2, 135.0, 134.3, 130.5, 129.9, 128.4, 125.6, 125.2, 122.2, 120.8, 119.9, 111.3, 107.6. HRMS: m/z calcd for $\text{C}_{17}\text{H}_{14}\text{NO}$: 248.1075 [$\text{M}+\text{H}]^+$. Found: 248.1081.



(E)-2-Benzylidene-6-(1,3-dioxoisodolin-2-yl)hexanal (32). A yellow crystalline solid (217 mg, 65 %); m.p. 88-90 °C; $R_f = 0.2$ (hexane/AcOEt: 8/2; IR (film) cm^{-1} : 3060, 2938, 2864, 2715, 1770, 1712, 1680, 1622, 1397, 1098, 1036. $^1\text{H-NMR}$ (400 MHz; CDCl_3): $\delta = 9.53$ (s, 1H), 7.82 (dd, $J = 5.3, 3.1$ Hz, 2H), 7.70 (dd, $J = 5.3, 3.1$ Hz, 2H), 7.42-7.36 (m, 5H), 7.23 (s, 1H), 3.68 (t, $J = 7.2$ Hz, 2H), 2.58 (t, $J = 7.9$ Hz, 2H), 1.75 (pent, $J = 7.5$ Hz, 2H), 1.54 (pent, $J = 7.5$ Hz, 2H). $^{13}\text{C NMR}$ (100 MHz; CDCl_3): $\delta = 195.5, 168.4, 150.2, 142.5, 134.8, 133.9, 132.1, 129.7, 129.6, 128.9, 123.2, 37.8, 28.7, 25.5, 24.3$. HRMS: m/z calcd for $\text{C}_{21}\text{H}_{20}\text{NO}_3$: 334.1443 [$\text{M}+\text{H}]^+$. Found: 334.1438.



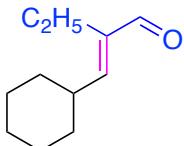
(E)-*N*-ethyl-*N*-(3-formyl-4-phenylbut-3-en-1-yl)nitrous amide (33) and (Z)-*N*-ethyl-*N*-(3-formyl-4-phenylbut-3-en-1-yl)nitrous amide (33). Recovered as an inseparable mixture of E/Z isomers (65/35). An amber oil (109 mg, 47 %); $R_f = 0.26$ (hexane/AcOEt: 8/2); IR (film) cm^{-1} : 2981, 1680, 1625, 1454, 1353, 1232, 1129, 1070, 1022, 754. $^1\text{H-NMR}$ (400 MHz; CDCl_3): $\delta = 9.59$ (s, 0.35 H), 9.56 (s, 0.65 H), 7.72 (d, $J = 7.8$ Hz, 1H), 7.55 (t, $J = 7.7$ Hz, 1H), 7.48-7.45 (m, 3H), 7.36 (s, 1H), 4.23-4.17 (m, 2H), 3.69-3.65 (m, 1H), 3.57-3.52 (m, 1H), 3.03-2.99 (m, 1H), 2.76-2.72 (m, 1H), 1.45 (t, $J = 7.3$ Hz, 2H), 1.07 (t, $J = 7.2$ Hz, 1H). $^{13}\text{C NMR}$ (100 MHz; CDCl_3): $\delta = 195.3, 195.0, 152.54, 152.53, 138.1, 137.8, 134.1, 133.9, 130.4, 130.2, 130.1, 129.3, 129.2, 129.1, 49.5, 47.7, 41.8, 38.8, 24.6, 22.0, 14.0, 11.2$. HRMS: m/z calcd for $\text{C}_{13}\text{H}_{17}\text{N}_2\text{O}_2$: 233.1290 [$\text{M}+\text{H}]^+$. Found: 233.1297.



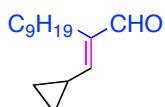
(E)-Ethyl 5-formyl-6-phenylhex-5-enoate (34). A colourless oil (150 mg, 61 %); $R_f = 0.24$ (hexane/AcOEt: 9/1); IR (film) cm^{-1} : 2979, 1731, 1680, 1624, 1374, 1182, 1131, 757. $^1\text{H-NMR}$ (400 MHz; CDCl_3): $\delta = 9.56$ (s, 1H), 7.54 (d, $J = 7.6$ Hz, 2H), 7.48-7.39 (m, 3H), 7.26 (s, 1H), 4.12 (q, $J = 7.1$ Hz, 2H), 2.59 (t, $J = 8.0$ Hz, 2H), 2.38 (t, $J = 7.3$ Hz, 2H), 1.84 (q, $J = 7.6$ Hz, 2H), 1.25 (t, $J = 7.1$ Hz, 3H). $^{13}\text{C-NMR}$ (100 MHz; CDCl_3): δ 195.5, 173.2, 150.6, 142.1, 134.7, 129.8, 128.9, 128.4, 60.4, 34.2, 24.2, 23.4, 14.3. HRMS: m/z calcd for $\text{C}_{15}\text{H}_{19}\text{O}_3$: 247.1334 $[\text{M}+\text{H}]^+$. Found: 247.1327.



(E)-2-Benzylidene-4-methoxybutanal (35). An amber oil (107 mg, 56 %); $R_f = 0.25$ (hexane/AcOEt: 9/1); IR (film) cm^{-1} : 2626, 1681, 1626, 1112, 1084, 756. $^1\text{H-NMR}$ (400 MHz; CDCl_3): $\delta = 9.58$ (s, 1H), 7.61 (d, $J = 7.4$ Hz, 2H), 7.43 (dt, $J = 11.8, 6.2$ Hz, 3H), 7.36 (s, 1H), 3.56 (t, $J = 7.0$ Hz, 2H), 3.34 (s, 3H), 2.85 (t, $J = 7.0$ Hz, 2H). $^{13}\text{C NMR}$ (100 MHz; CDCl_3): δ 195.5, 151.8, 139.3, 134.6, 129.8, 129.7, 128.8, 70.4, 58.7, 25.6. HRMS: m/z calcd for $\text{C}_{12}\text{H}_{15}\text{O}_2$: 191.1072 $[\text{M}+\text{H}]^+$. Found: 191.1069.



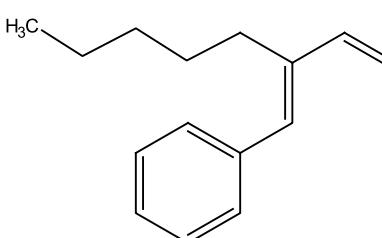
(E)-2-(Cyclohexylmethylene)butanal (36). A light yellow oil (73 mg, 44 %); $R_f = 0.5$ (hexane/AcOEt: 9/1); IR (film) cm^{-1} : 2957, 2928, 2853, 1689, 1643, 1602, 1441, 1124, 1049. $^1\text{H-NMR}$ (400 MHz; CDCl_3): $\delta = 9.33$ (s, 1H), 6.23 (d, $J = 9.9$ Hz, 1H), 2.57-2.49 (m, 1H), 2.26 (q, $J = 7.5$ Hz, 2H), 1.81-1.76 (m, 2H), 1.70-1.67 (m, 3H), 1.37-1.31 (m, 2H), 1.26-1.18 (m, 3H), 0.98 (t, $J = 7.5$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz; CDCl_3): δ = 195.6, 159.6, 143.3, 37.9, 32.2, 25.8, 25.4, 17.5, 14.0. HRMS: m/z calcd for $\text{C}_{11}\text{H}_{19}\text{O}$: 167.1436 $[\text{M}+\text{H}]^+$. Found: 167.1445.



(E)-2-(cyclopropylmethylene)undecanal (37). A colourless oil (85 mg, 38 %); $R_f = 0.4$ (hexane/AcOEt: 9/1); IR (film) cm^{-1} : 2924, 2854, 1681, 1635, 1456, 1096, 1052. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 9.26 (s, 1H), 5.74 (d, $J = 10.6$ Hz, 1H), 2.34 (t, $J = 7.6$ Hz, 2H), 1.83 (dtd, $J = 15.9, 8.1, 4.2$ Hz, 1H), 1.40 (q, $J = 7.0$ Hz, 2H), 1.36-1.18 (m, $J = 10.4$ Hz, 12H), 1.10 (td, $J = 7.1, 4.5$ Hz, 2H), 0.88 (t, $J = 6.6$ Hz, 3H), 0.72 (p, $J = 4.6$ Hz, 2H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 194.2, 160.3, 142.1, 31.9, 29.6, 29.6, 29.5, 29.3, 28.9, 24.1, 22.7, 14.1, 12.3, 9.5. HRMS: m/z calcd for $\text{C}_{15}\text{H}_{27}\text{O}$: 223.2062 $[\text{M}+\text{H}]^+$. Found: 223.2060.

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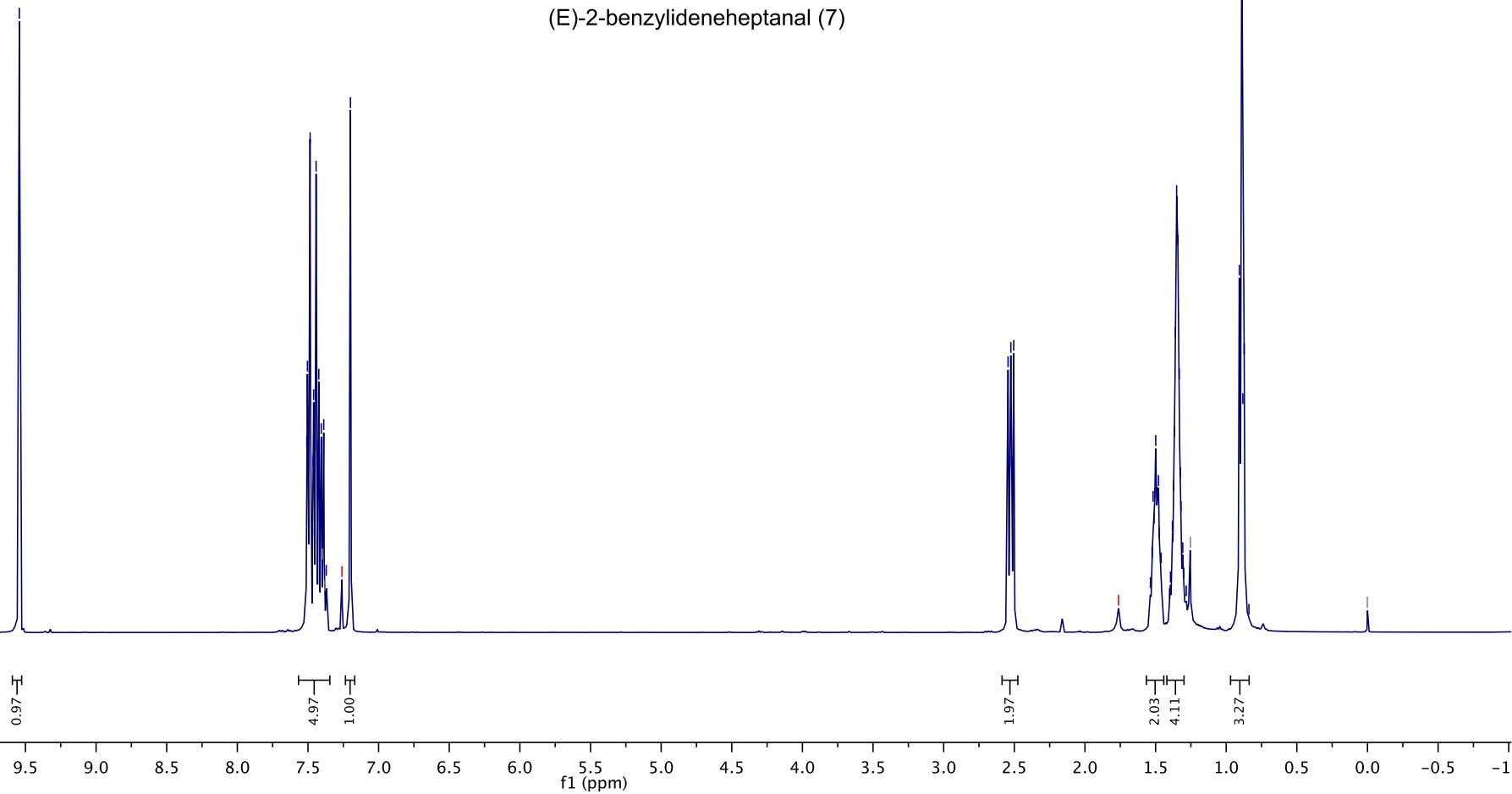


— 1.76 HDO

2.54
2.53
2.52
2.50

9.54
7.51
7.49
7.48
7.46
7.46
7.45
7.44
7.42
7.41
7.41
7.40
7.40
7.38
7.37
7.26 CDCl₃
7.20

(E)-2-benzylideneheptanal (7)

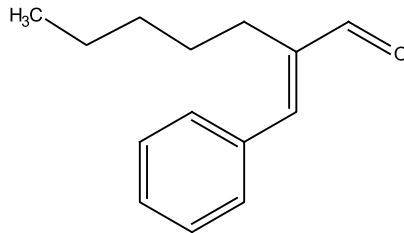


— 32.08
— 27.99
— 24.76
— 22.41

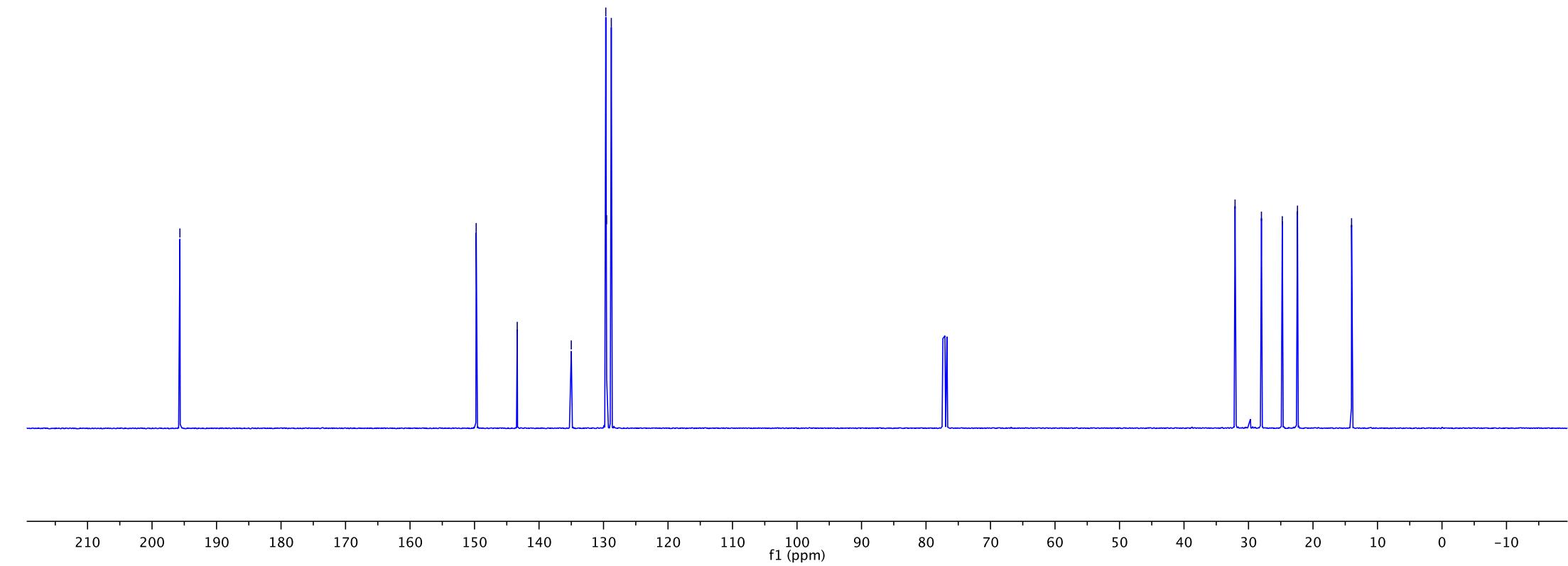
— 14.02

— 195.68

— 149.74
— 143.38
— 135.00
— 129.65
— 129.53
— 128.79



(E)-2-benzylideneheptanal (7)



S21

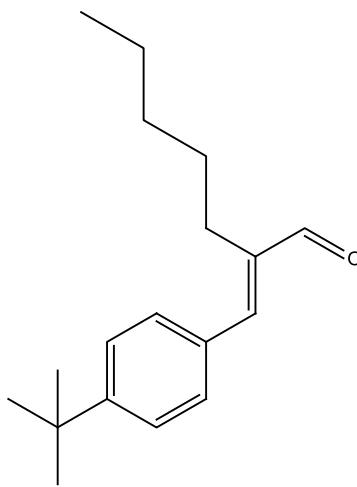
0.00

-0.00

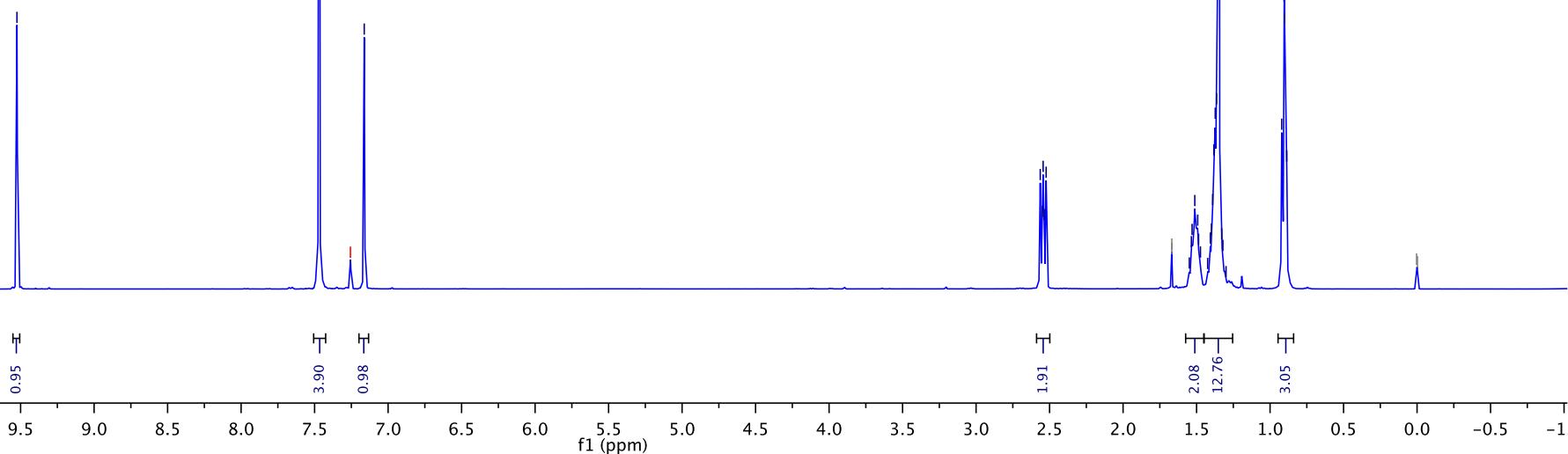
2.56
2.55
2.54
2.54
2.52
2.52
1.67
1.67
1.55
1.55
1.54
1.54
1.53
1.51
1.49
1.49
1.47
1.47
1.42
1.42
1.41
1.40
1.39
1.38
1.38
1.37
1.37
1.36
1.35
1.35
1.33
1.32
1.30
0.92
0.90
0.90
0.89

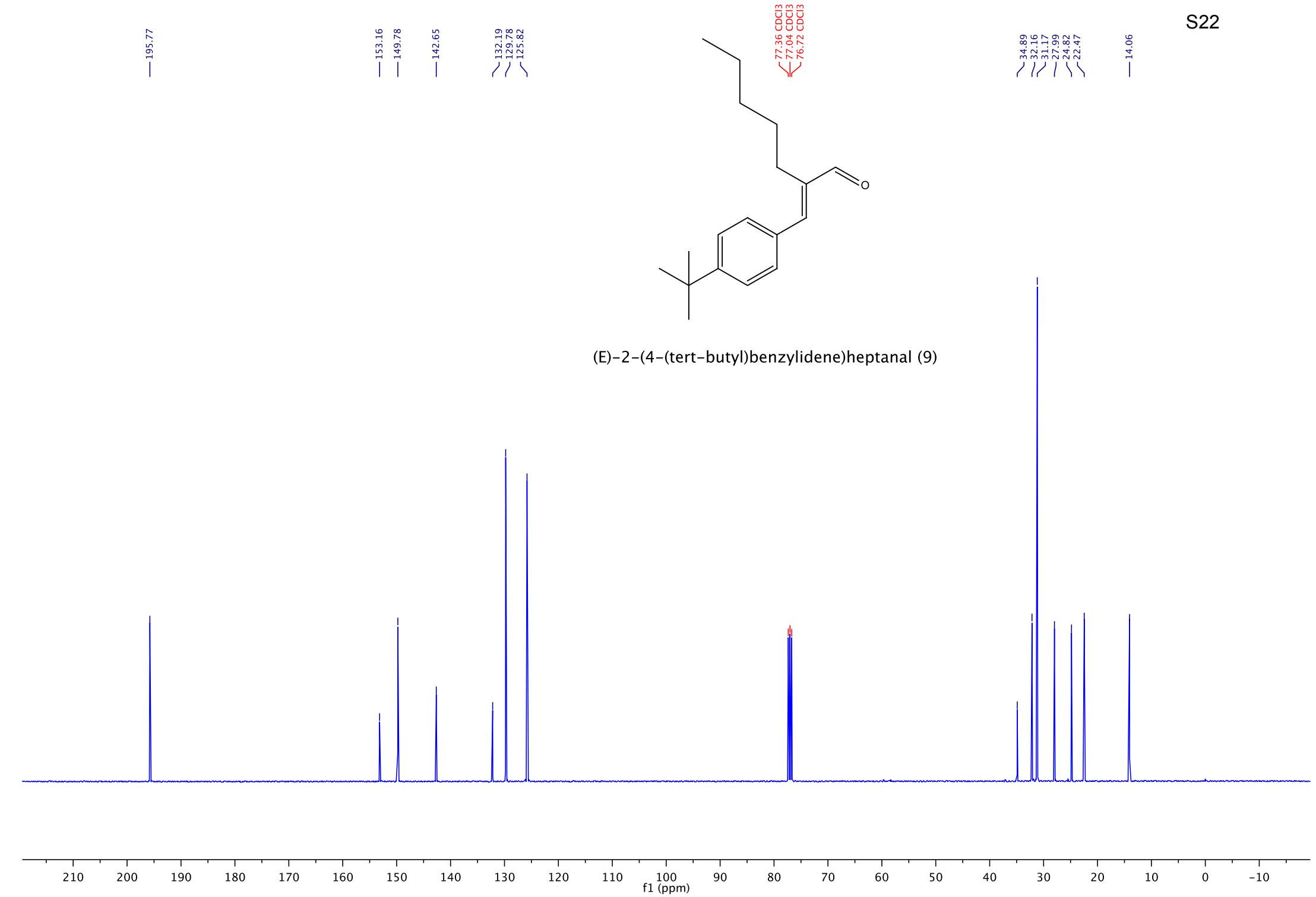
7.47
7.47
7.26 CDCl₃
7.16

9.52



(E)-2-(4-(tert-butyl)benzylidene)heptanal (9)

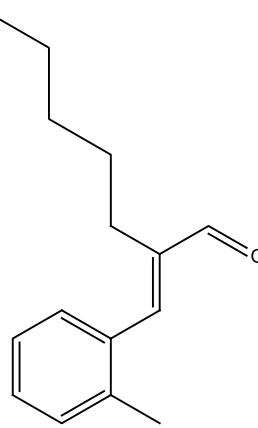




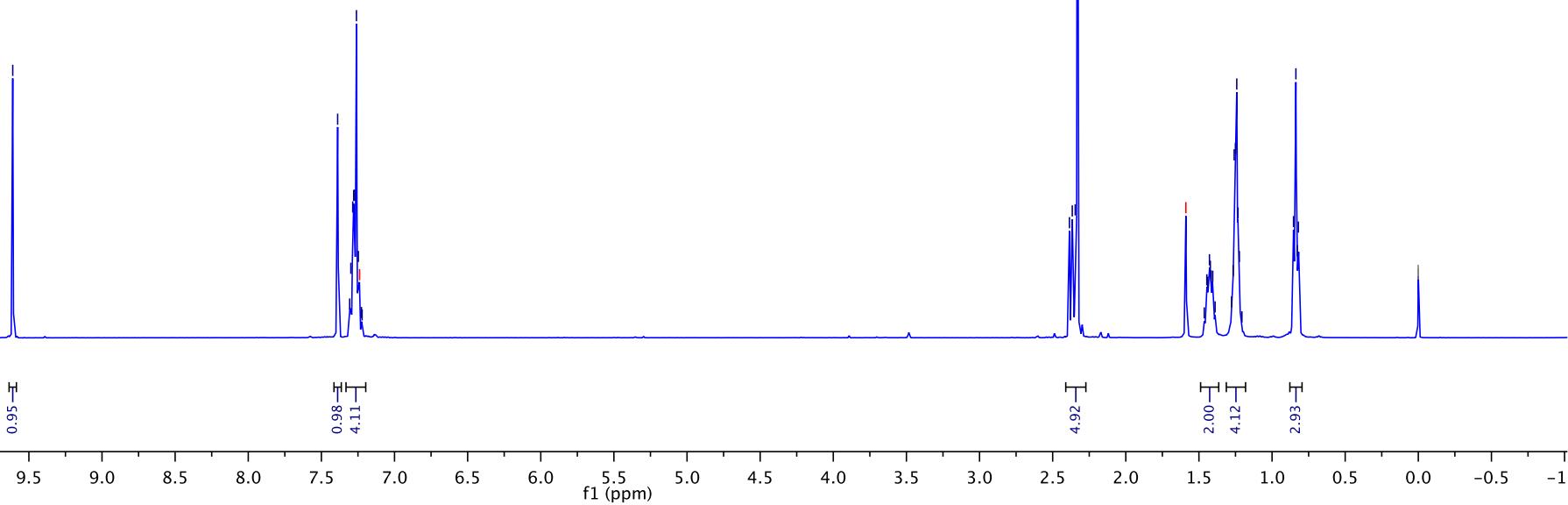
— 9.61

7.39
7.31
7.30
7.29
7.28
7.27
7.26
7.24 CDCl₃
7.23
7.22

2.39
2.37
2.35
2.33
1.59 HDO
1.46
1.45
1.44
1.43
1.42
1.41
1.39
1.28
1.27
1.26
1.25
1.24
1.23
1.22
1.21
0.85
0.84
0.83
0.82



(E)-2-(2-methylbenzylidene)heptanal (10)



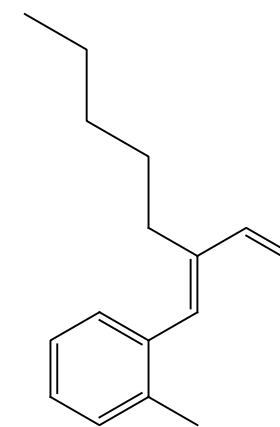
— 195.58

— 148.81
— 144.15
— 136.64
— 134.21
— 130.34
— 129.03
— 128.15
— 125.85

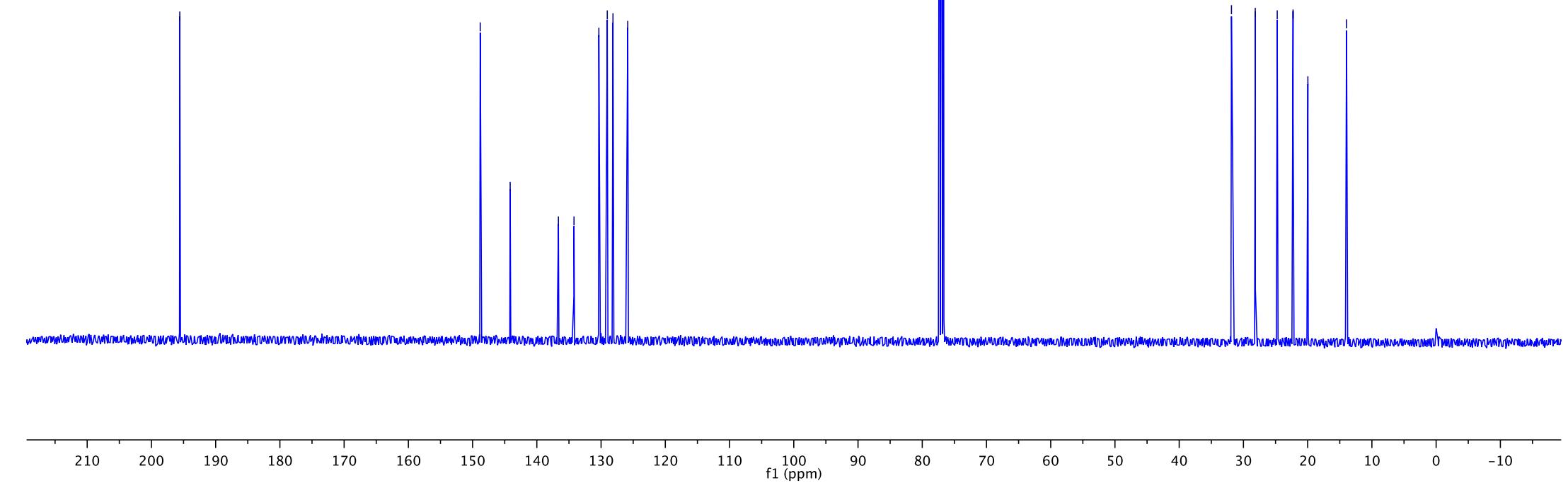
— 31.86
— 28.16
— 24.74
— 22.27
— 19.97

— 13.95

S24

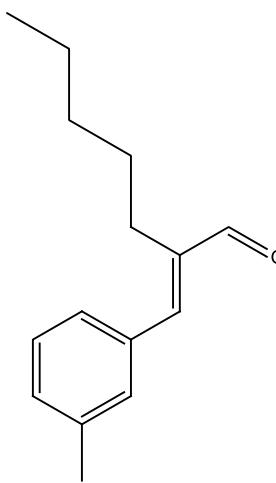


(E)-2-(2-methylbenzylidene)heptanal (10)

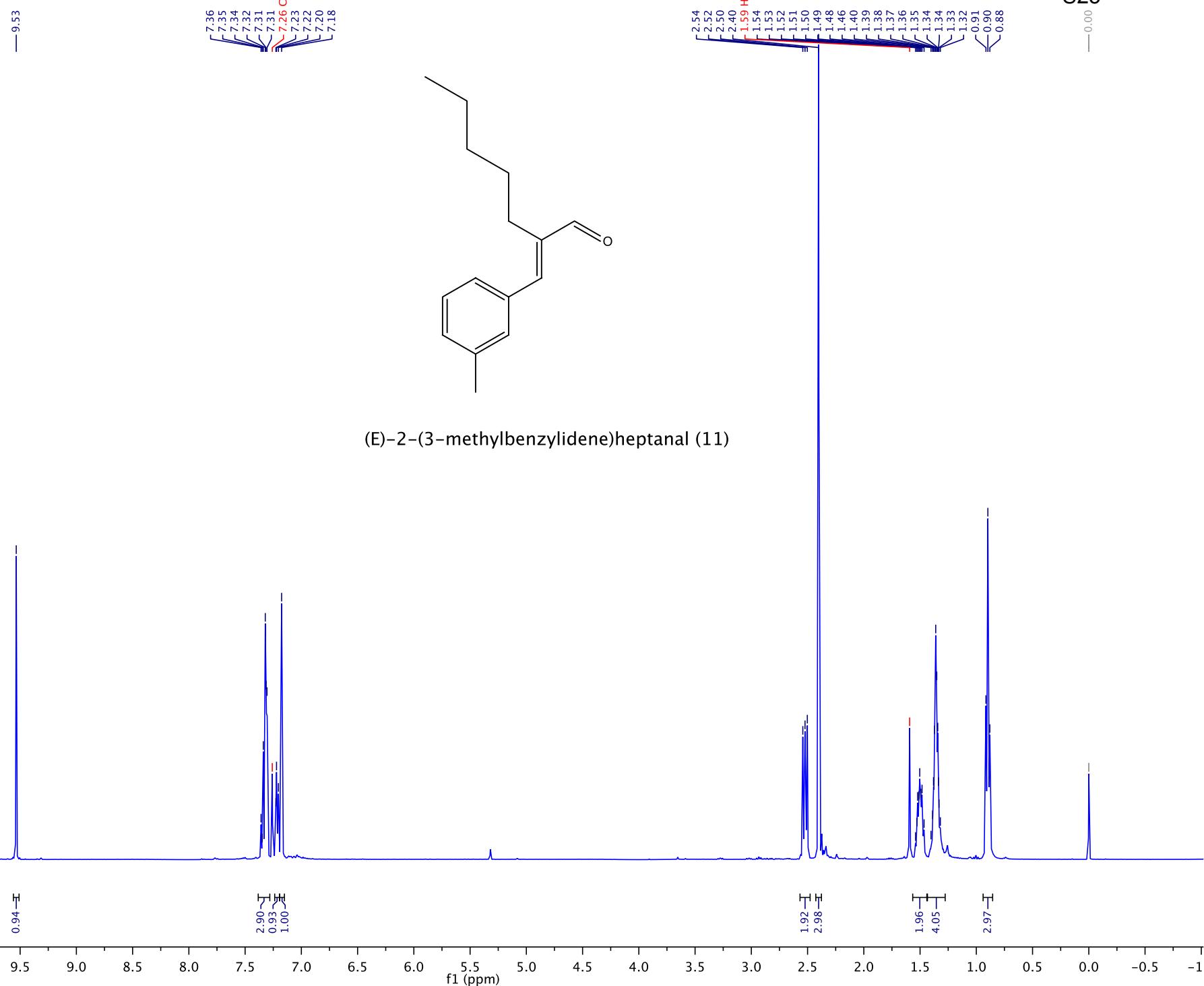


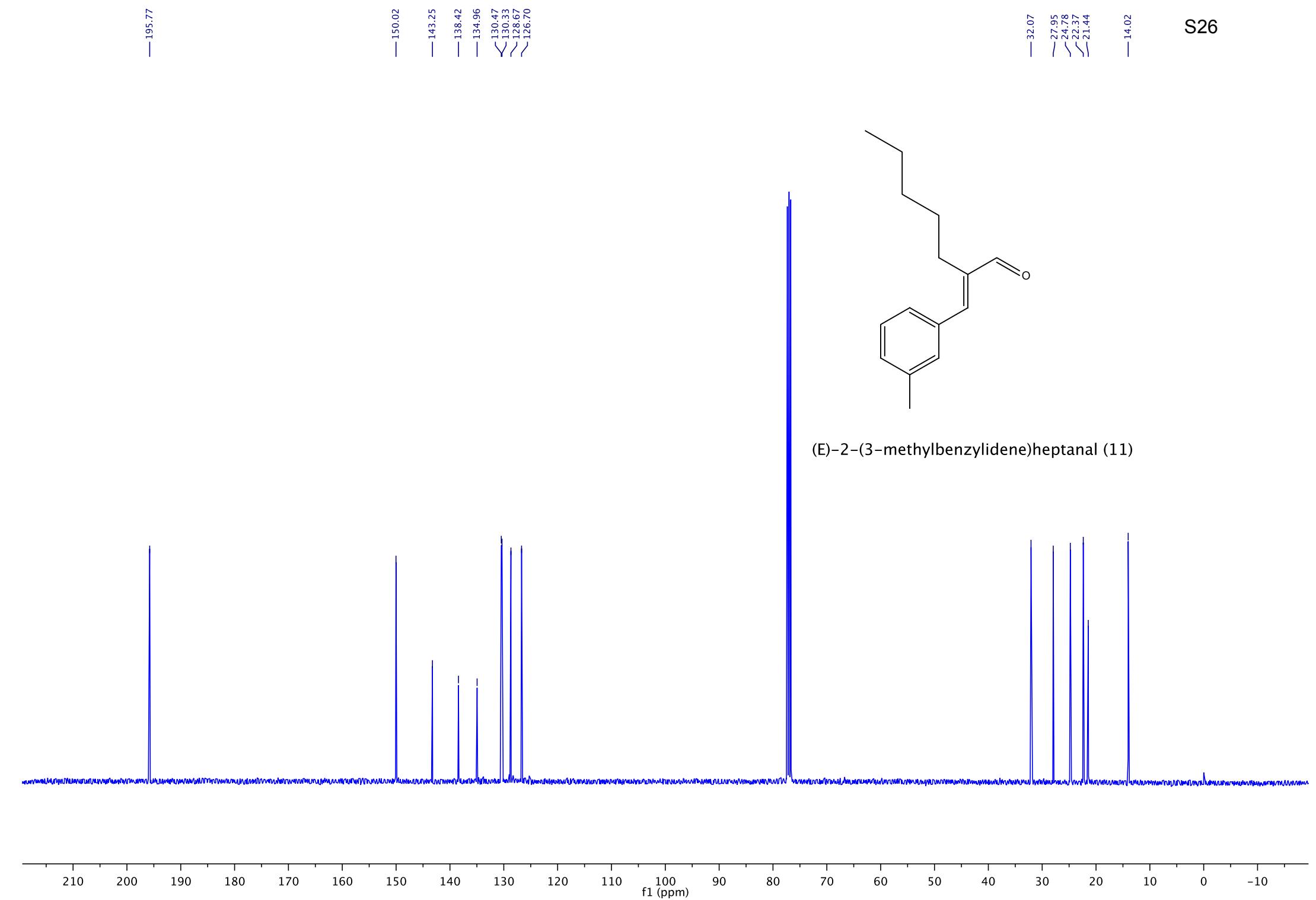
7.36
7.35
7.34
7.32
7.31
7.26 CDCl₃
7.23
7.22
7.20
7.18

2.54
2.52
2.50
2.40
1.59 HDO
1.54
1.53
1.52
1.51
1.50
1.49
1.48
1.46
1.40
1.39
1.38
1.37
1.36
1.35
1.34
1.33
1.32
0.91
0.90
0.88



(E)-2-(3-methylbenzylidene)heptanal (11)

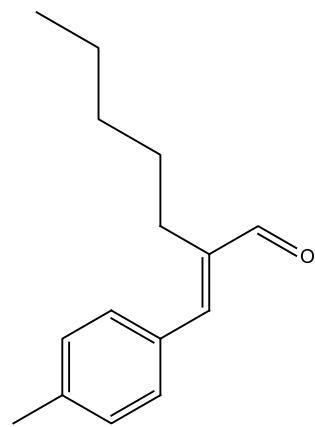




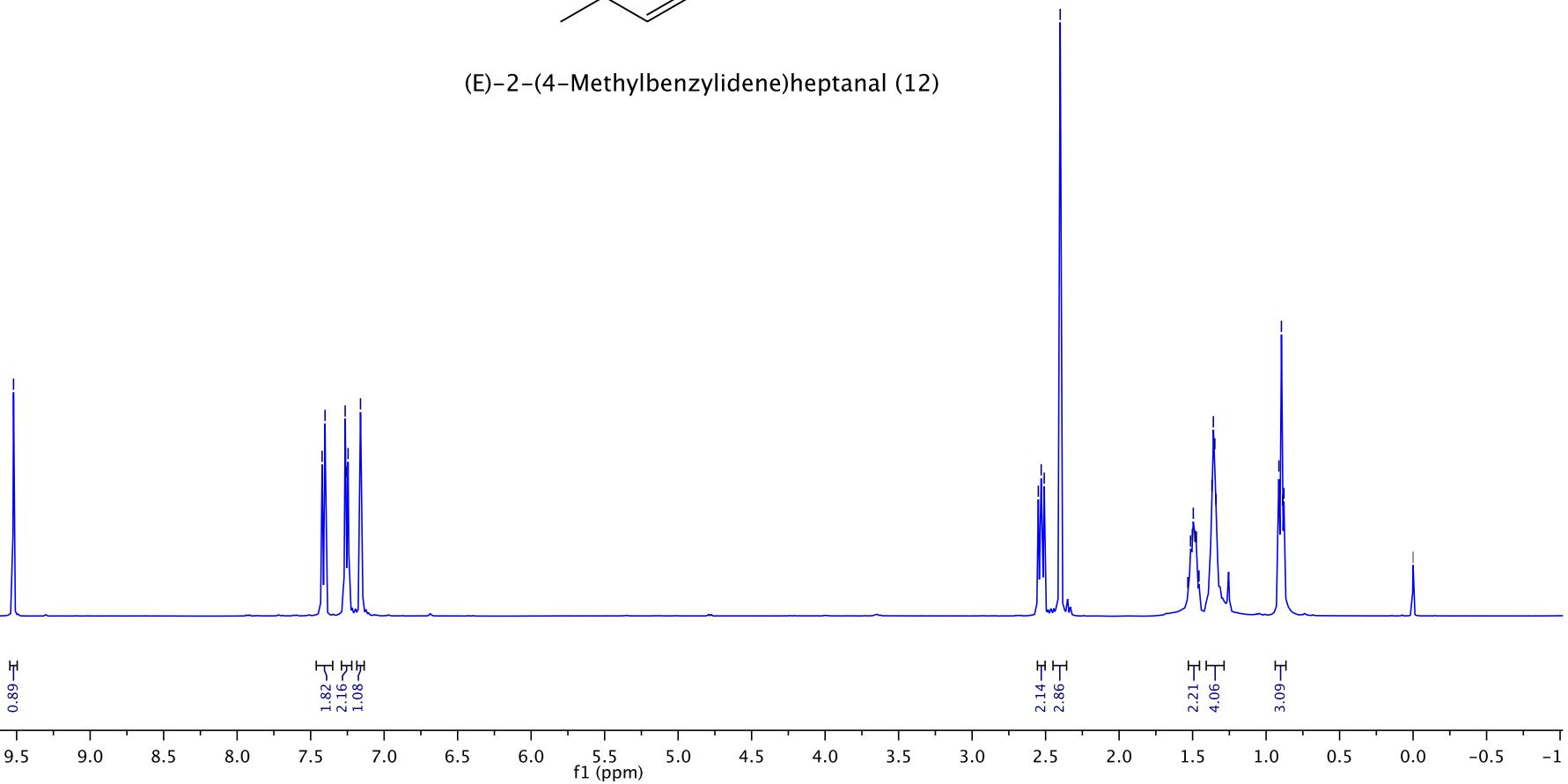
— 9.52 —

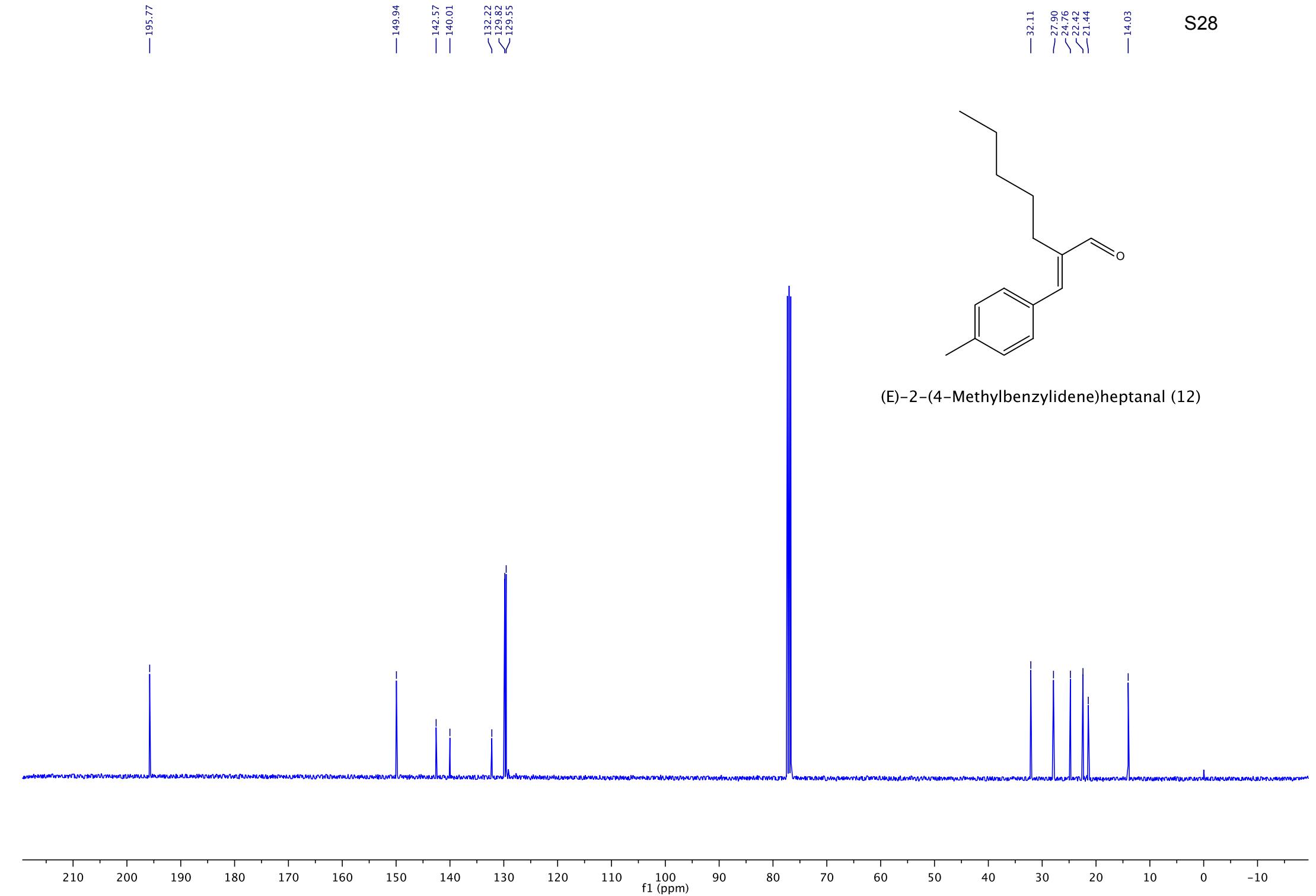
7.42
7.27
7.26
7.25
7.16

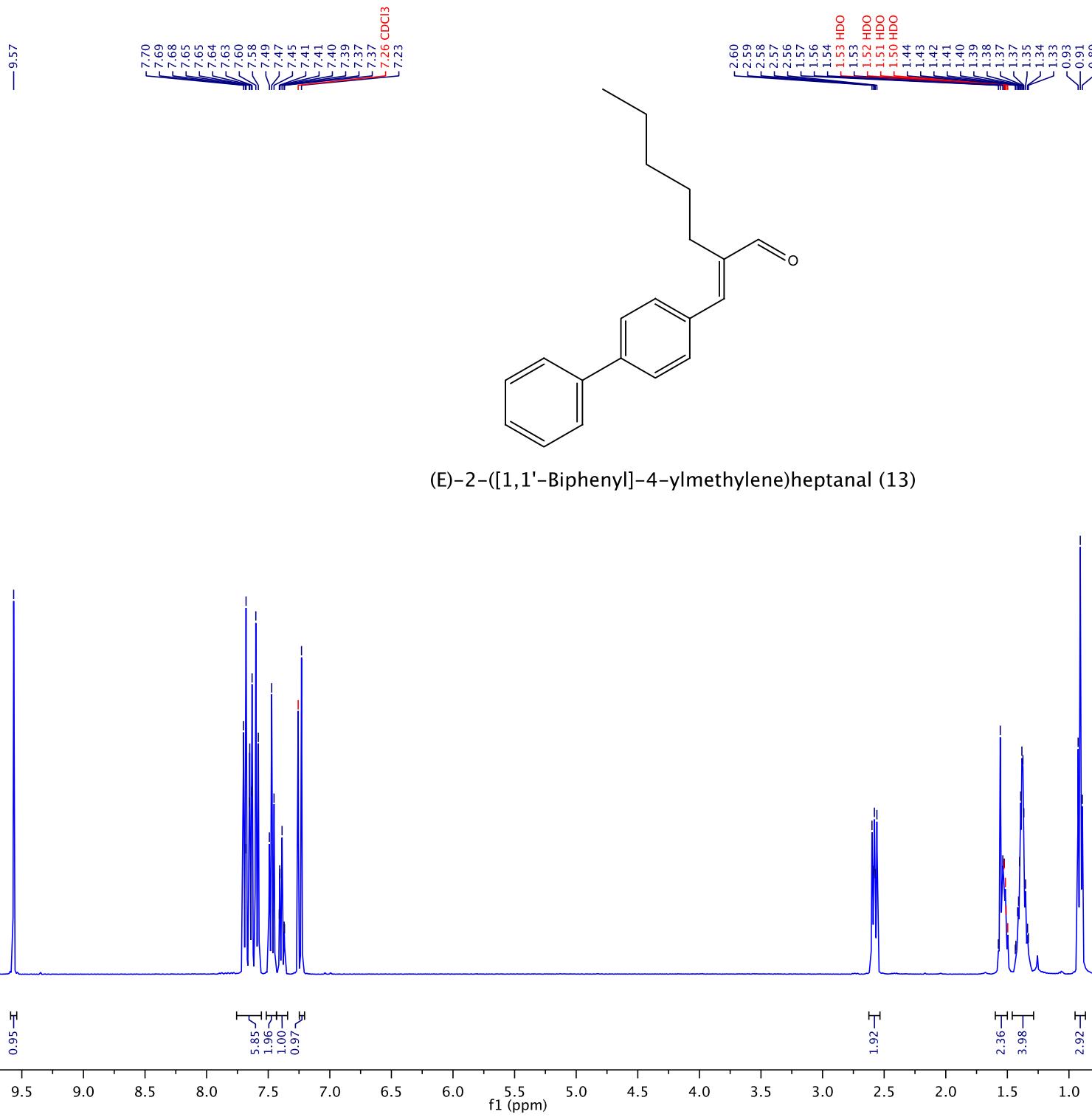
2.55
2.53
2.51
2.40
1.53
1.51
1.50
1.50
1.48
1.47
1.46
1.37
1.36
1.35
1.34
0.91
0.90
0.89
0.88



(E)-2-(4-Methylbenzylidene)heptanal (12)







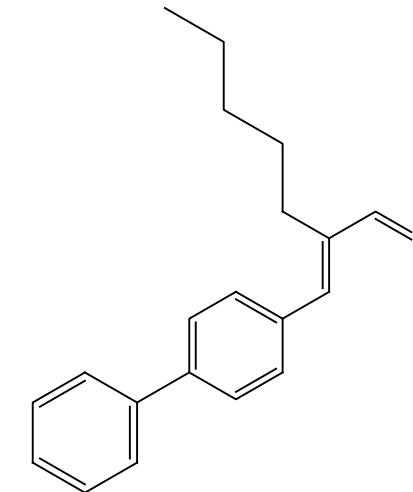
— 195.64

— 149.29
— 143.30
— 142.31
— 140.03
— 133.93
— 130.30
— 128.94
— 127.91
— 127.42
— 127.07

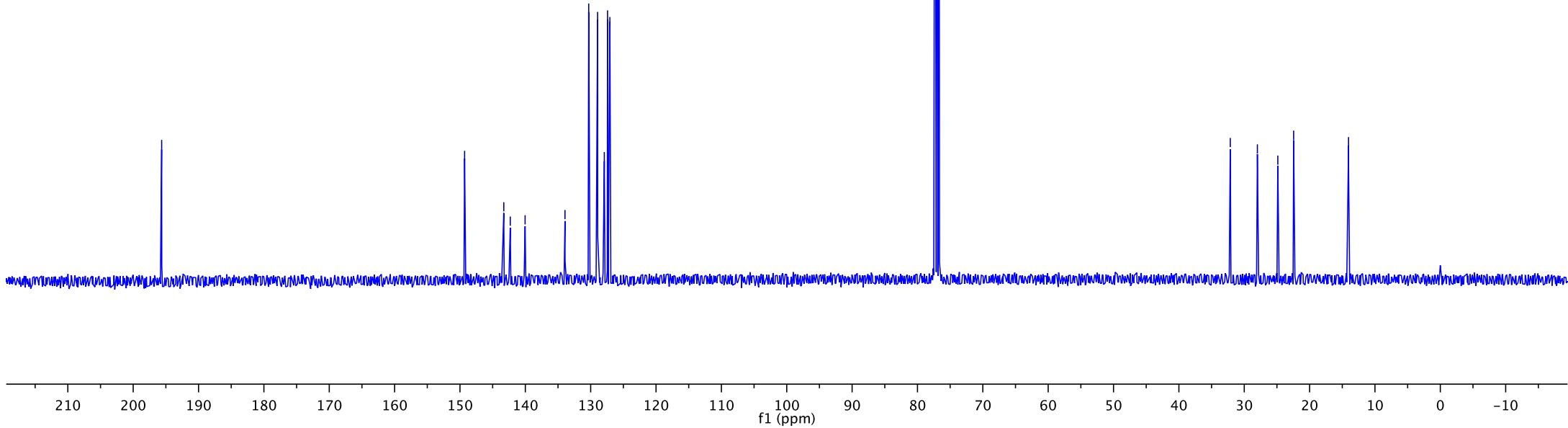
— 32.14
— 27.99
— 24.86
— 22.44

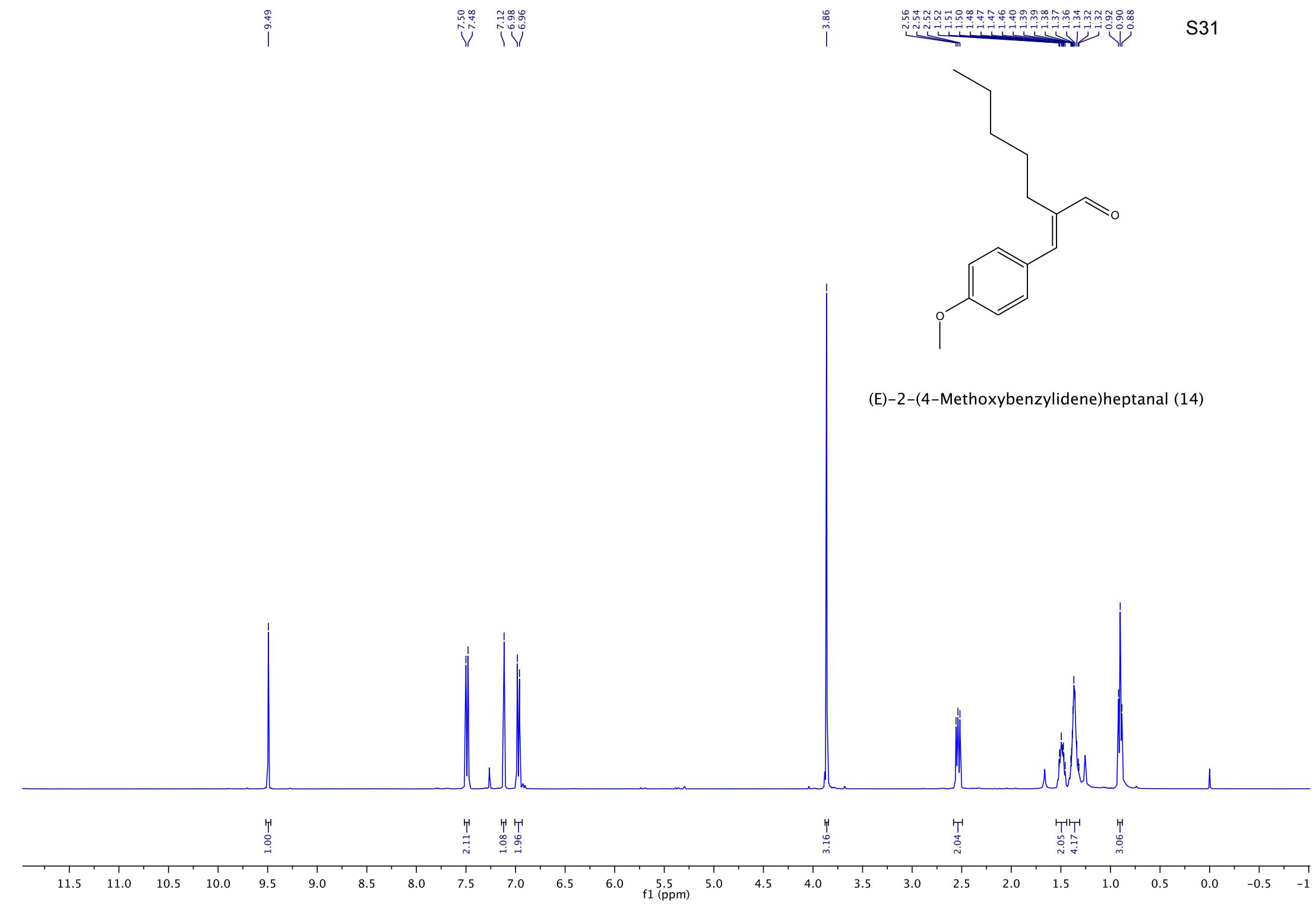
— 14.04

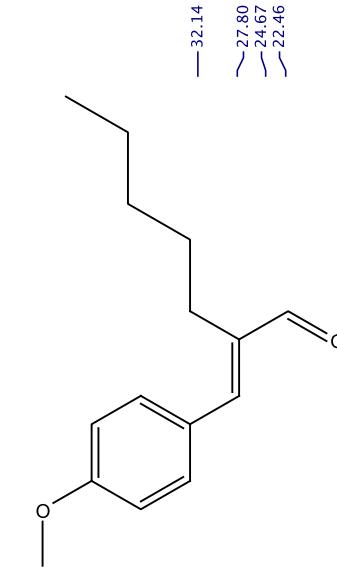
S30



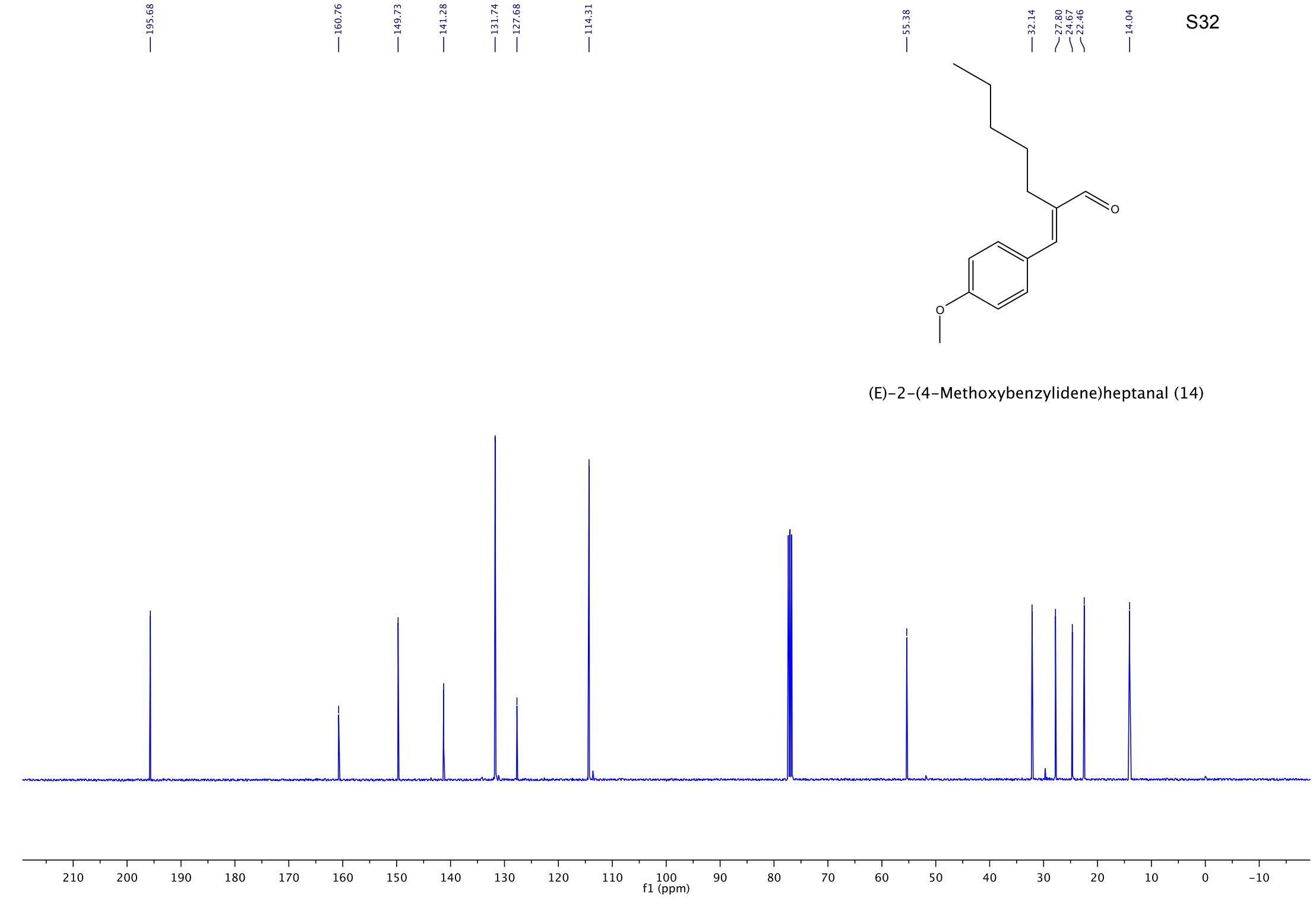
(E)-2-([1,1'-Biphenyl]-4-ylmethylene)heptanal (13)





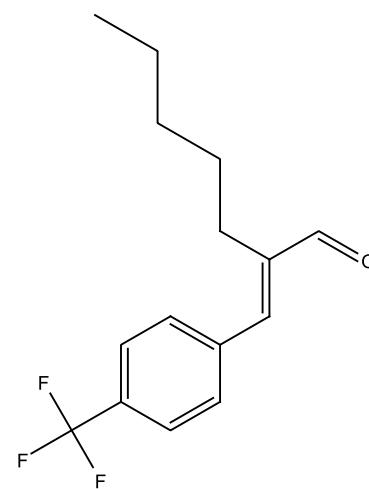


(E)-2-(4-Methoxybenzylidene)heptanal (14)



9.59
7.72
7.70
7.59
7.57
7.26 CDCl₃
7.24

2.52
2.50
2.48
1.58 HDO
1.52
1.51
1.49
1.48
1.47
1.45
1.38
1.37
1.36
1.35
1.34
1.33
1.32
1.31
1.30
1.26
1.24
0.91
0.89
0.87



(E)-2-(4-(Trifluoromethyl)benzylidene)heptanal (15)

— 9.59 —

1.90 H
1.91 H
0.95 H1.91 H
1.98 H
4.06 H
2.99 H

11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1

f1 (ppm)

— 195.18

— 147.34

— 145.19

— 138.44

— 138.42

— 131.51

— 131.18

— 130.86

— 130.53

— 129.57

— 127.89

— 125.77

— 125.73

— 125.70

— 125.66

— 125.18

— 122.48

— 119.77

— 77.34

— 77.02

— 76.70

— 32.02

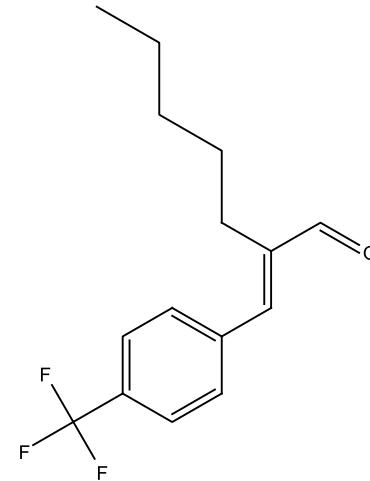
— 28.08

— 24.81

— 22.36

— 13.97

S34

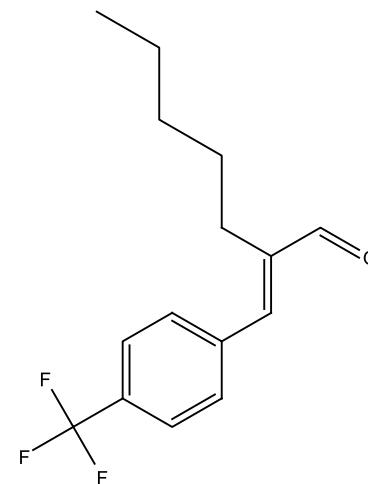


(E)-2-(4-(Trifluoromethyl)benzylidene)heptanal (15)

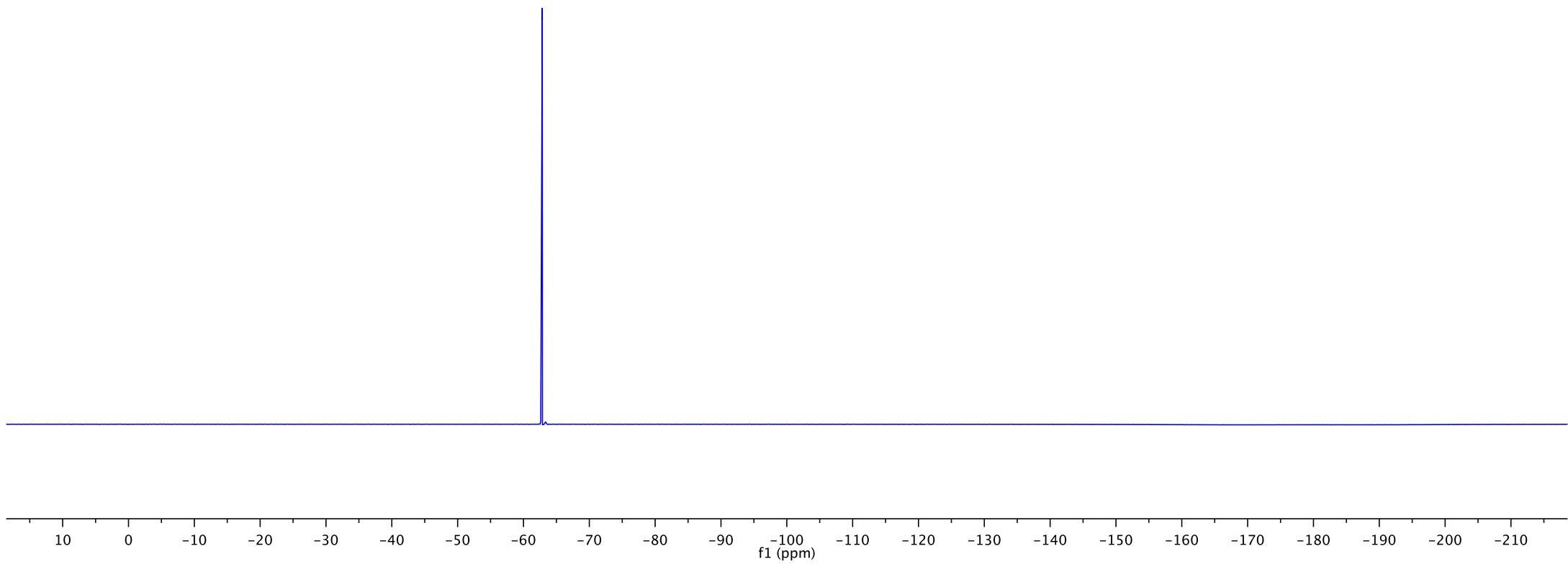
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

f1 (ppm)

— -62.85



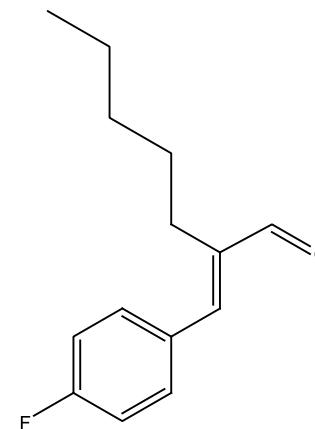
(E)-2-(4-(Trifluoromethyl)benzylidene)heptanal (15)



— 9.53 —

7.51
7.50
7.49
7.47
7.26 CDCl₃
7.16
7.14
7.13
7.12

2.53
2.51
2.51
2.50
2.49
1.59 HDO
1.52
1.51
1.50
1.48
1.47
1.46
1.44
1.39
1.38
1.37
1.36
1.35
1.34
1.33
1.32
1.31
1.26
0.91
0.89
0.88



(E)-2-(4-Fluorobenzylidene)heptanal (16)

— 9.56 —

— 1.96 —

— 2.91 —

— 1.97 —

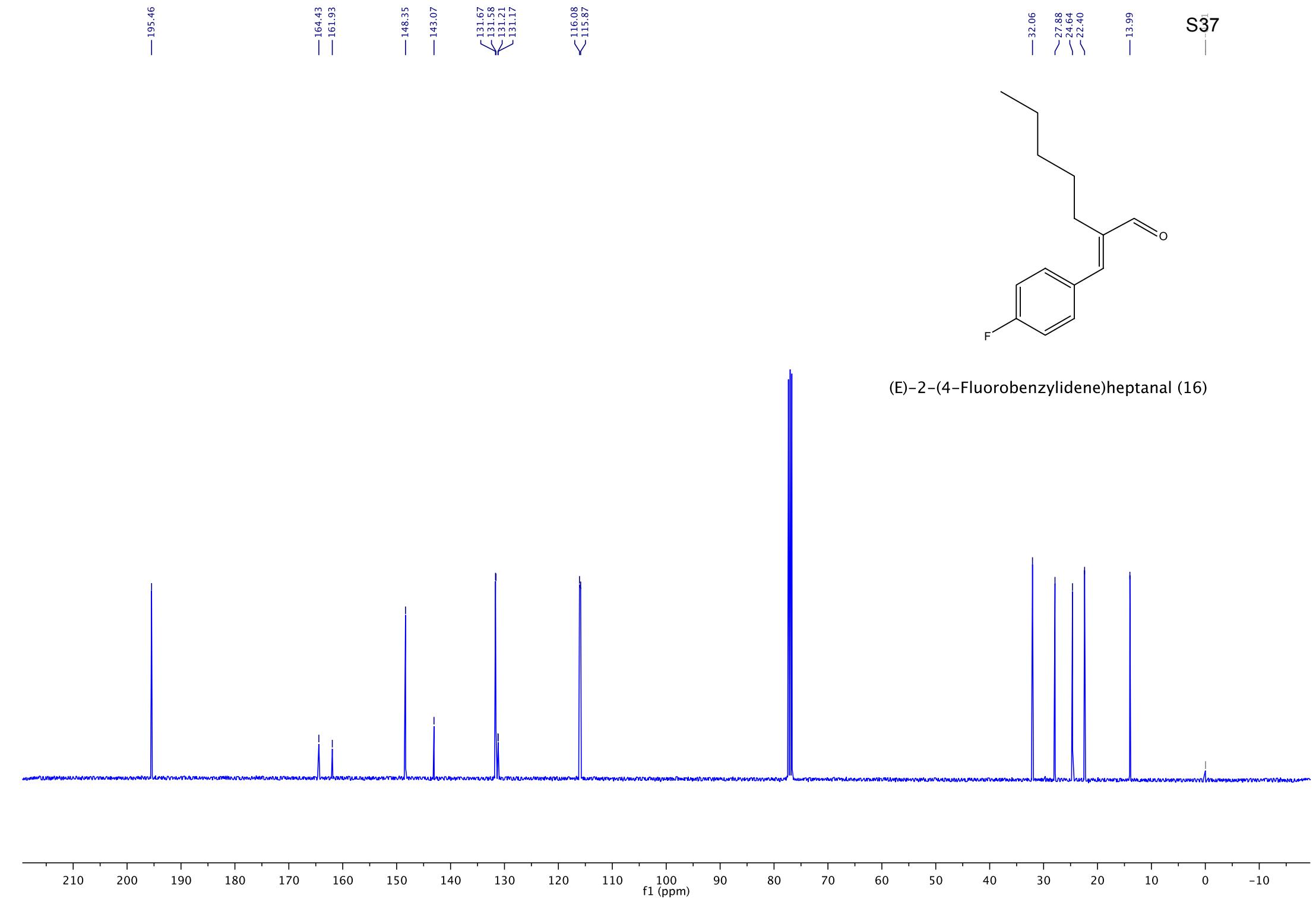
— 2.00 —

— 4.16 —

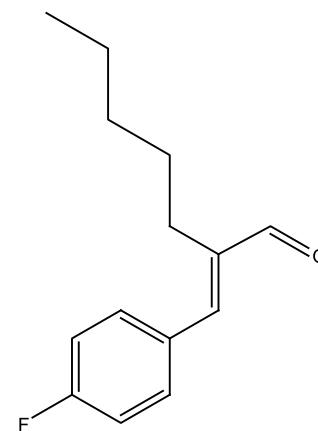
— 3.03 —

11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1

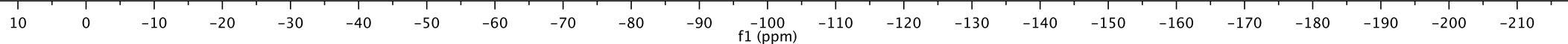
f1 (ppm)



-110.26



(E)-2-(4-Fluorobenzylidene)heptanal (16)

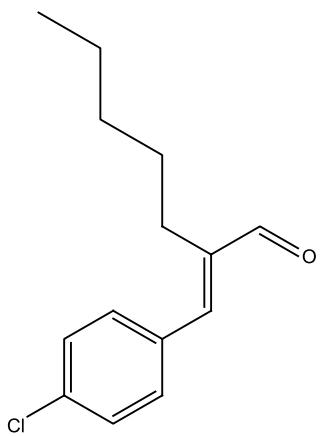


— 9.54 —

— 7.42 —

— 7.15 —

2.52
2.50
2.48
1.51
1.50
1.49
1.48
1.48
1.47
1.47
1.46
1.44
1.39
1.38
1.37
1.36
1.36
1.35
1.34
1.33
1.33
1.32
1.32
1.30
1.30
0.89
0.88
0.87



(E)-2-(4-Chlorobenzylidene)heptanal (17)

1.03 —H

4.11 —H

1.05 —H

2.09 —H

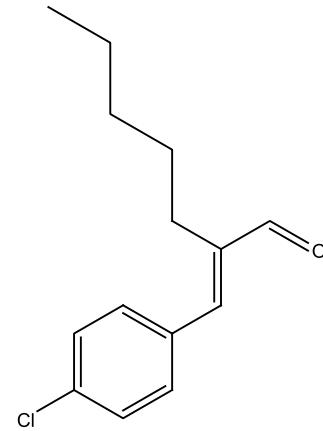
2.13 —H

4.24 —H

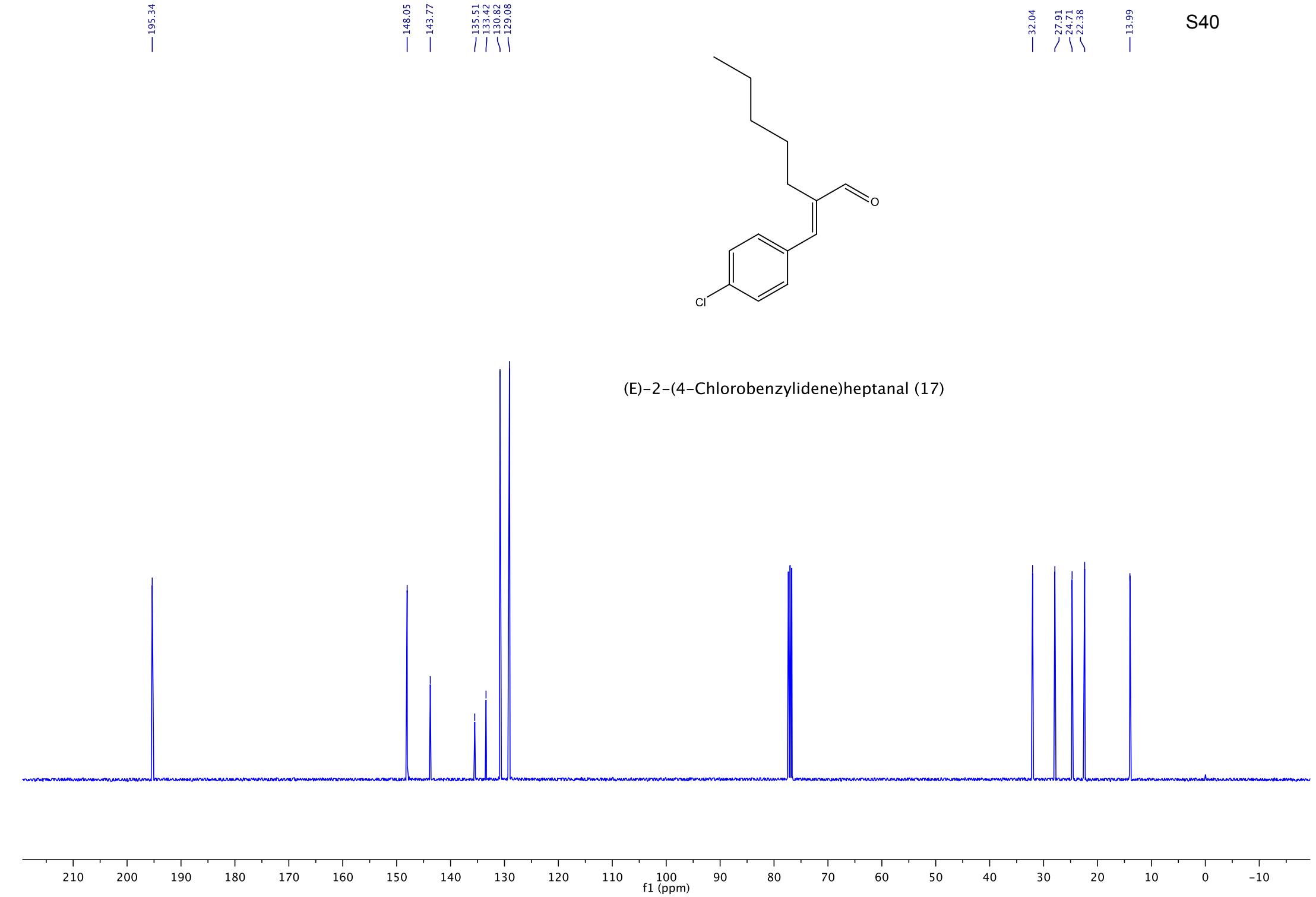
3.19 —H

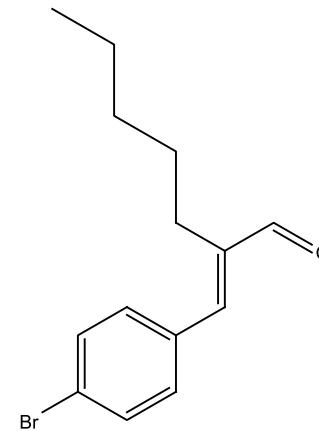
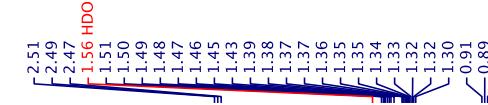
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1

f1 (ppm)

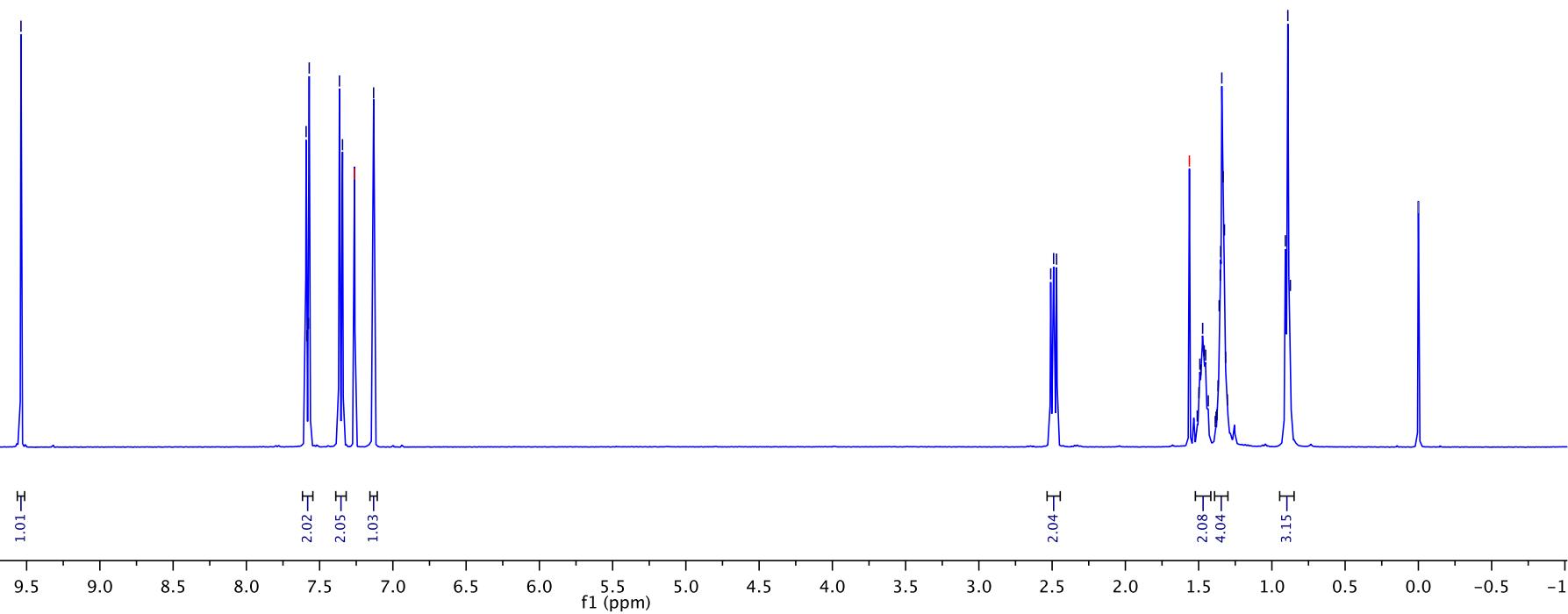


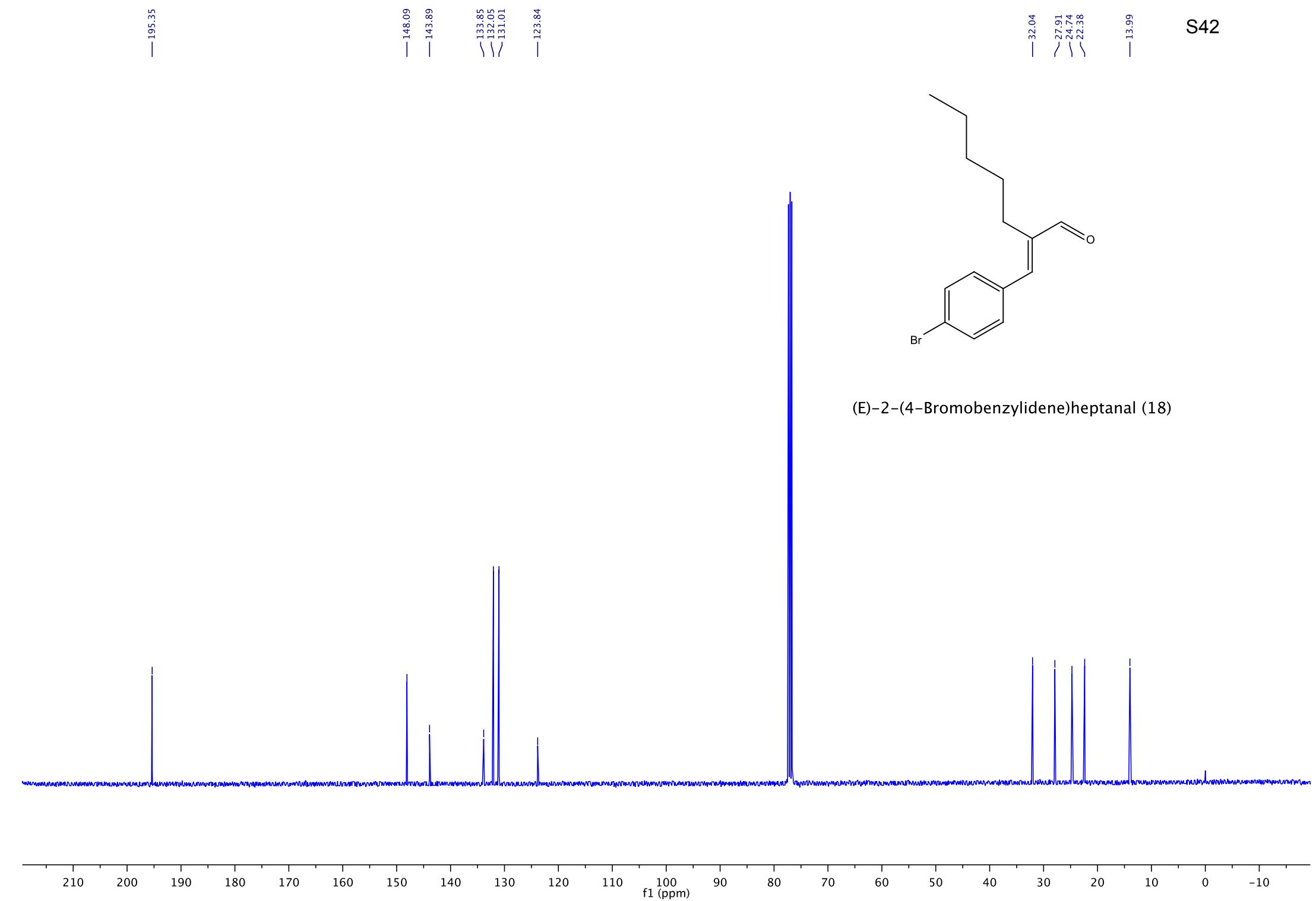
(E)-2-(4-Chlorobenzylidene)heptanal (17)





(E)-2-(4-Bromobenzylidene)heptanal (18)

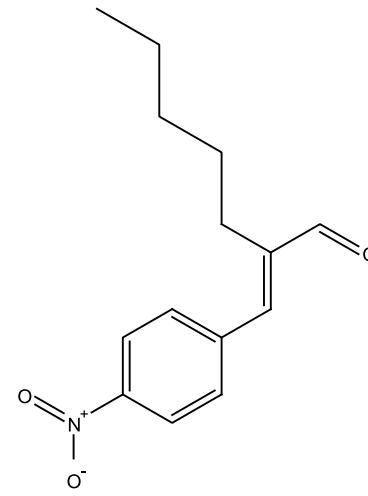




2.52
2.50
2.48
2.46
1.53
1.51
1.50
1.49
1.48
1.47
1.45
1.38
1.37
1.36
1.35
1.34
1.33
1.32
1.31
1.30
0.91
0.89
0.87

— 7.27 CDCl₃

— 9.61

8.32
8.307.64
7.62

(E)-2-(4-Nitrobenzylidene)heptanal (19)

1.03—H

2.06—H

2.10—H

2.09—H

2.13—H
4.11—H

3.19—H

f1 (ppm)

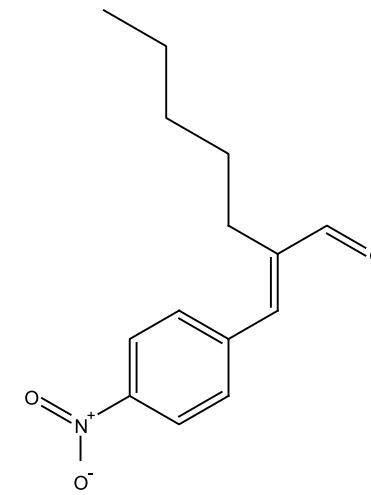
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 -1

— 194.77

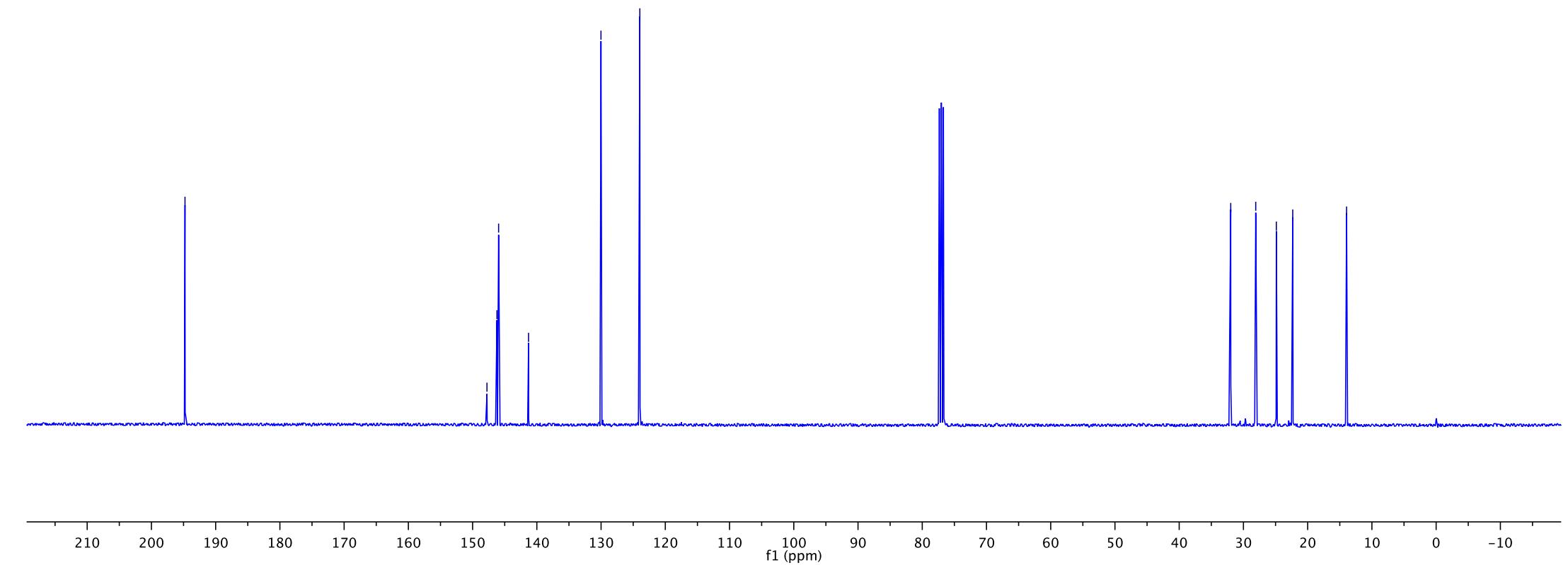
— 147.76
— 146.21
— 145.95
— 141.29

— 130.02
— 123.97

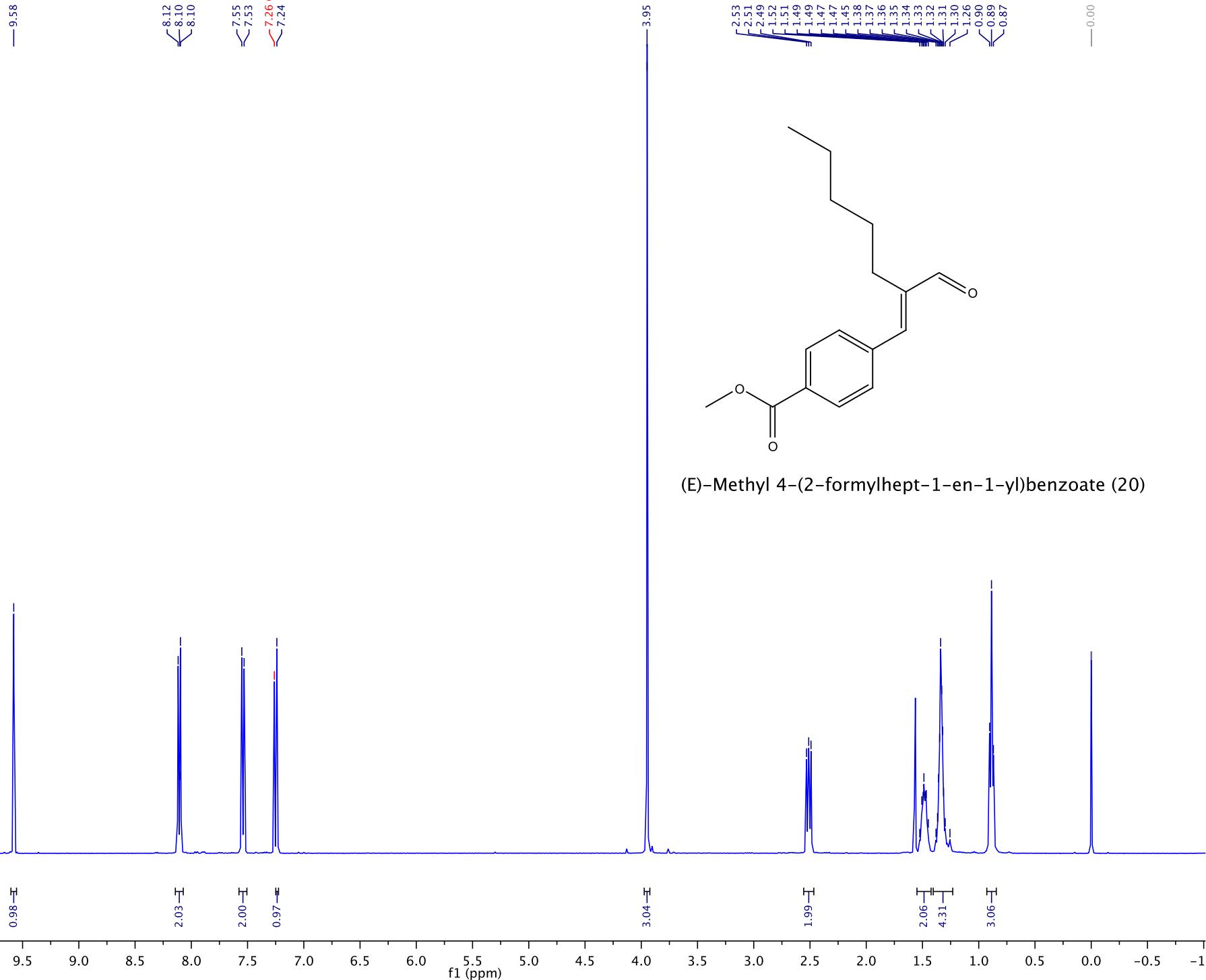
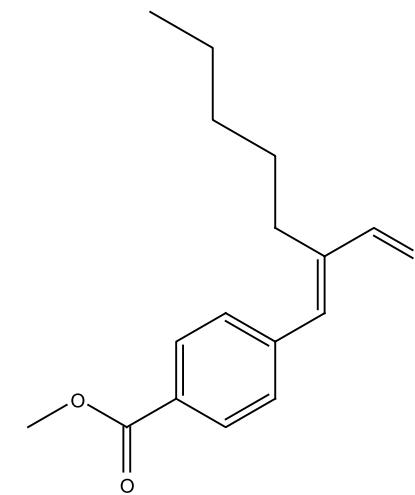
— 31.98
— 28.98
— 24.88
— 22.32
— 13.94



(E)-2-(4-Nitrobenzylidene)heptanal (19)



2.53
2.51
2.49
2.47
1.52
1.51
1.49
1.47
1.45
1.38
1.37
1.36
1.35
1.34
1.33
1.32
1.31
1.30
1.26
1.26
0.90
0.89
0.87



— 195.28

— 166.43

— 147.94

— 145.02

— 139.32

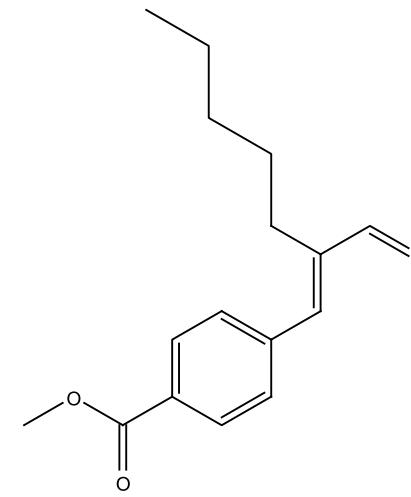
130.59
129.93
129.33

77.33 CDCl₃
77.21
77.01 CDCl₃
76.69 CDCl₃

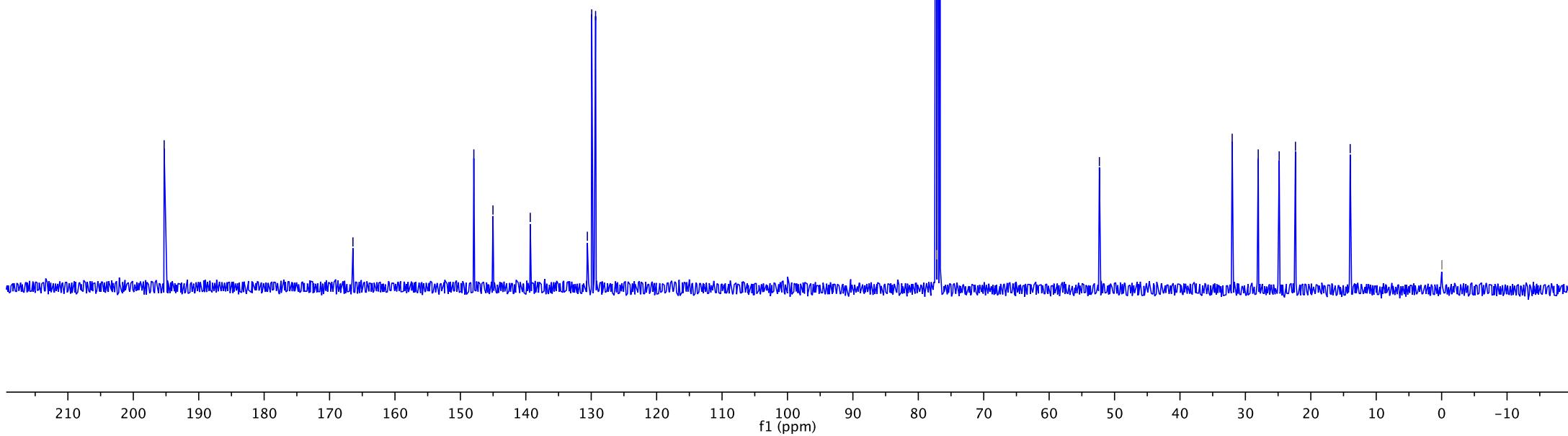
— 52.32

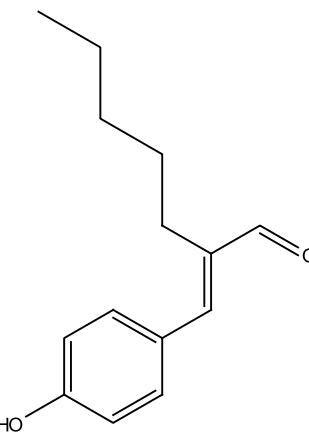
32.02
28.04
24.83
22.35

— 13.98

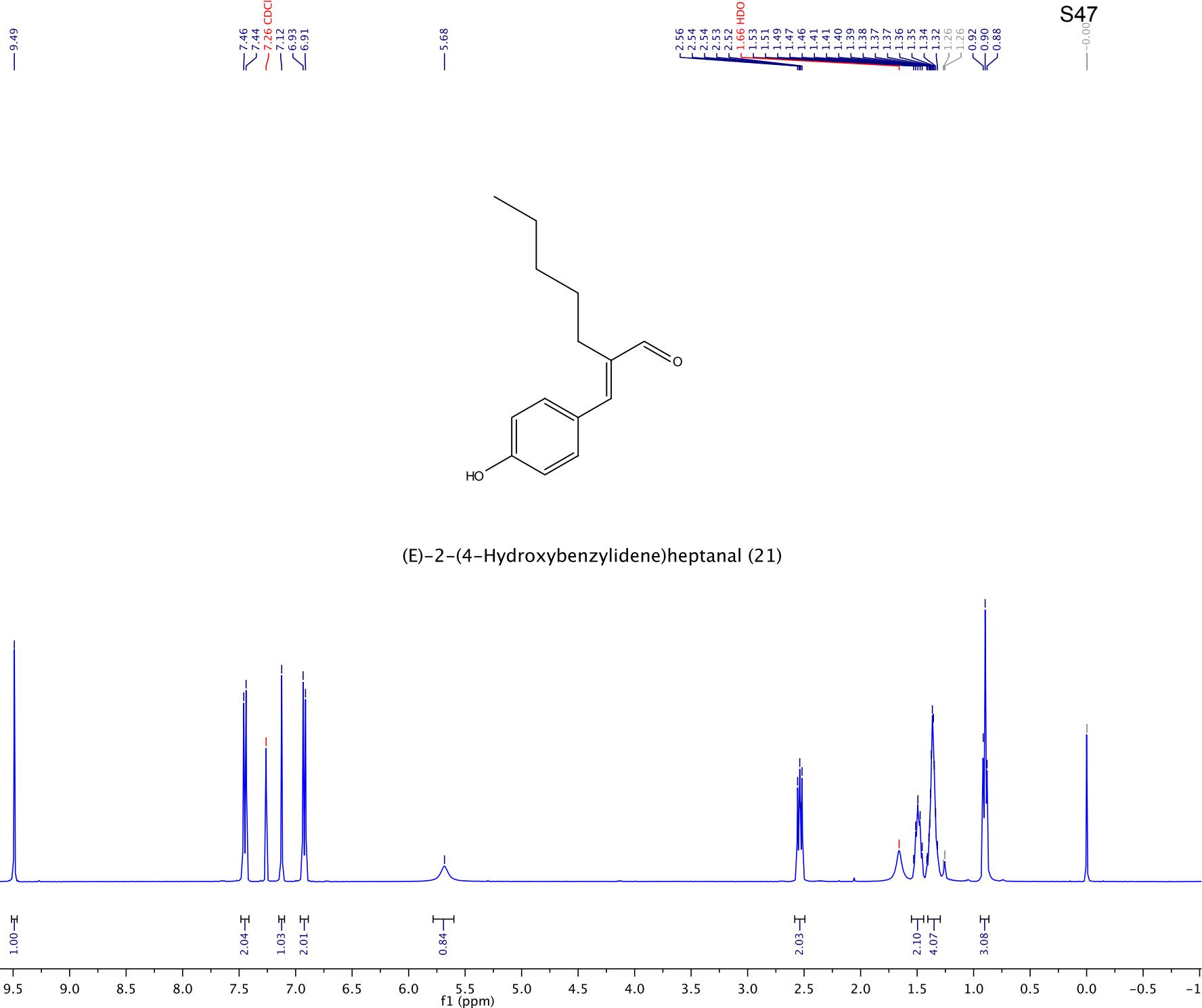


(E)-Methyl 4-(2-formylhept-1-en-1-yl)benzoate (20)





(E)-2-(4-Hydroxybenzylidene)heptanal (21)



—196.05

—157.23

—150.13

—141.22

—132.00

—127.72

—115.89

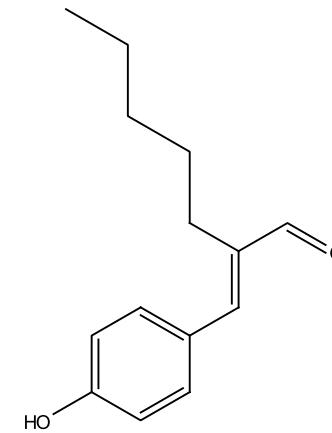
—32.11

~27.77

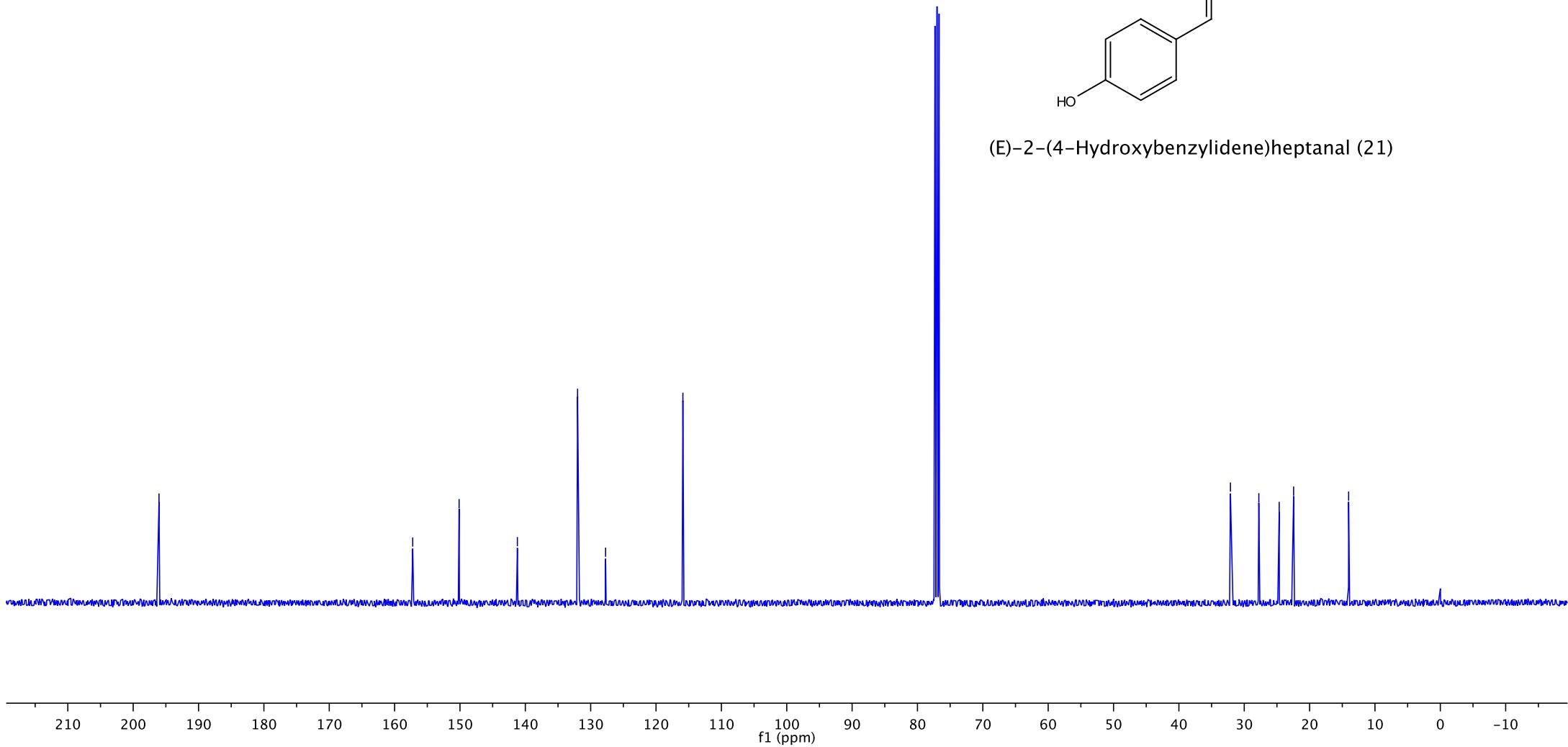
~24.65

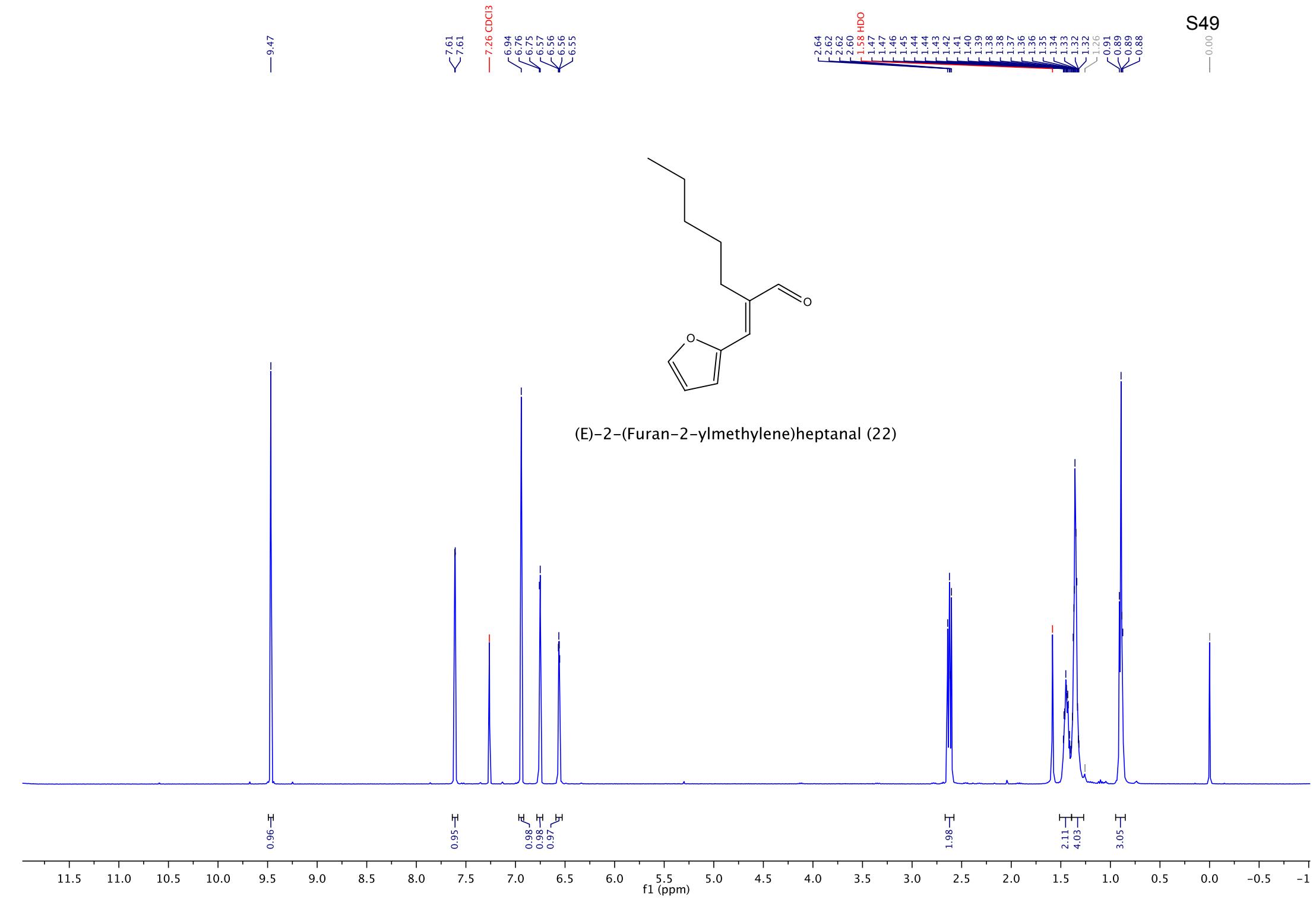
~22.45

—14.03



(E)-2-(4-Hydroxybenzylidene)heptanal (21)

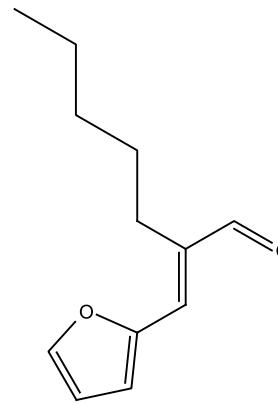




— 32.05
— 28.00
— 24.80
— 22.48

— 14.01

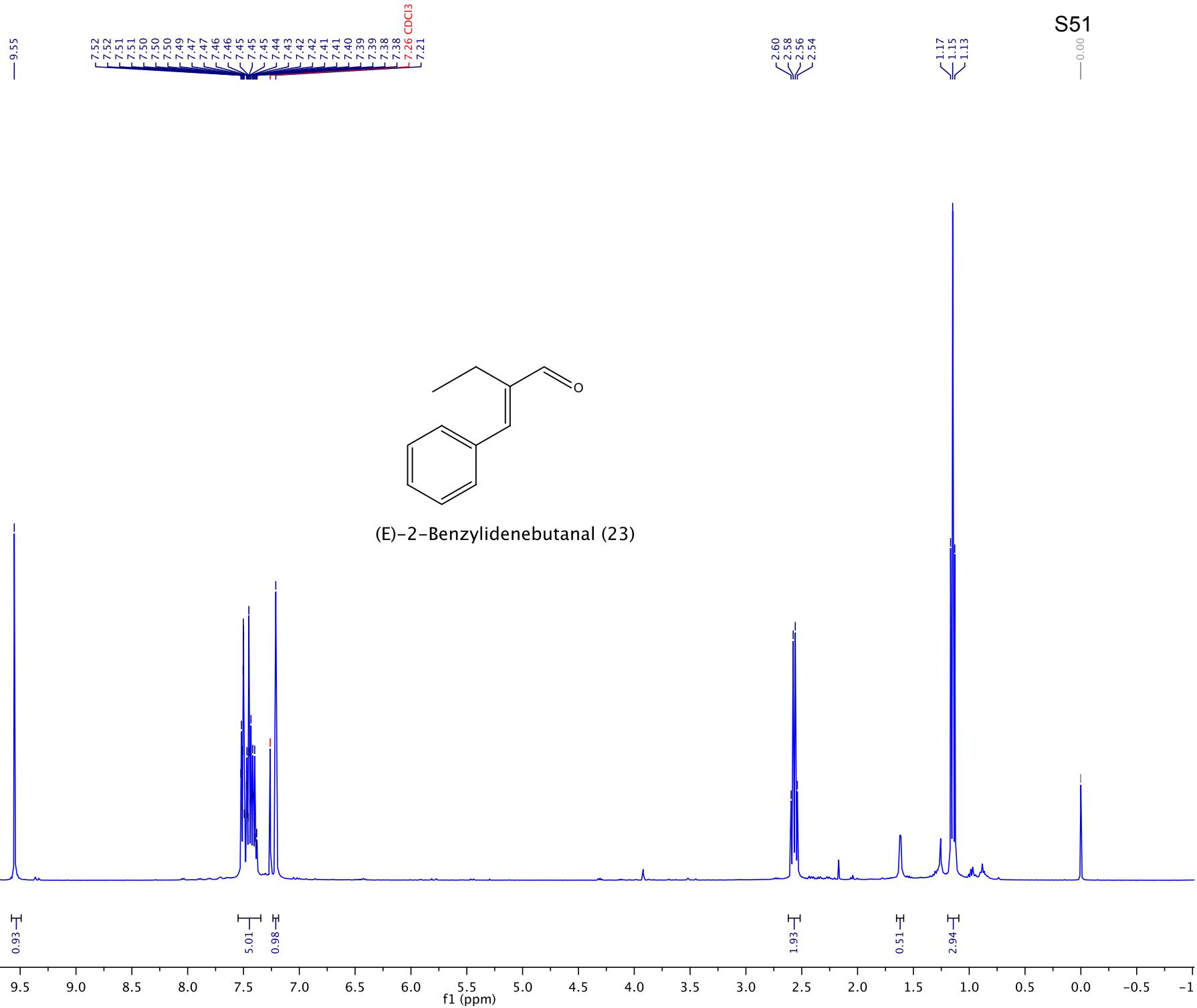
— 32.05
— 28.00
— 24.80
— 22.48



(E)-2-(Furan-2-ylmethylene)heptanal (22)

— 194.36
— 151.36
— 145.30
— 140.04
— 135.07

— 116.41
— 112.60



— 195.56

— 149.55

— 144.46

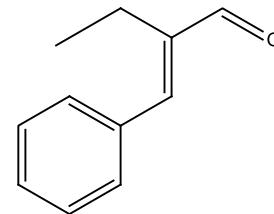
— 134.91

— 129.63
— 129.56
— 128.81

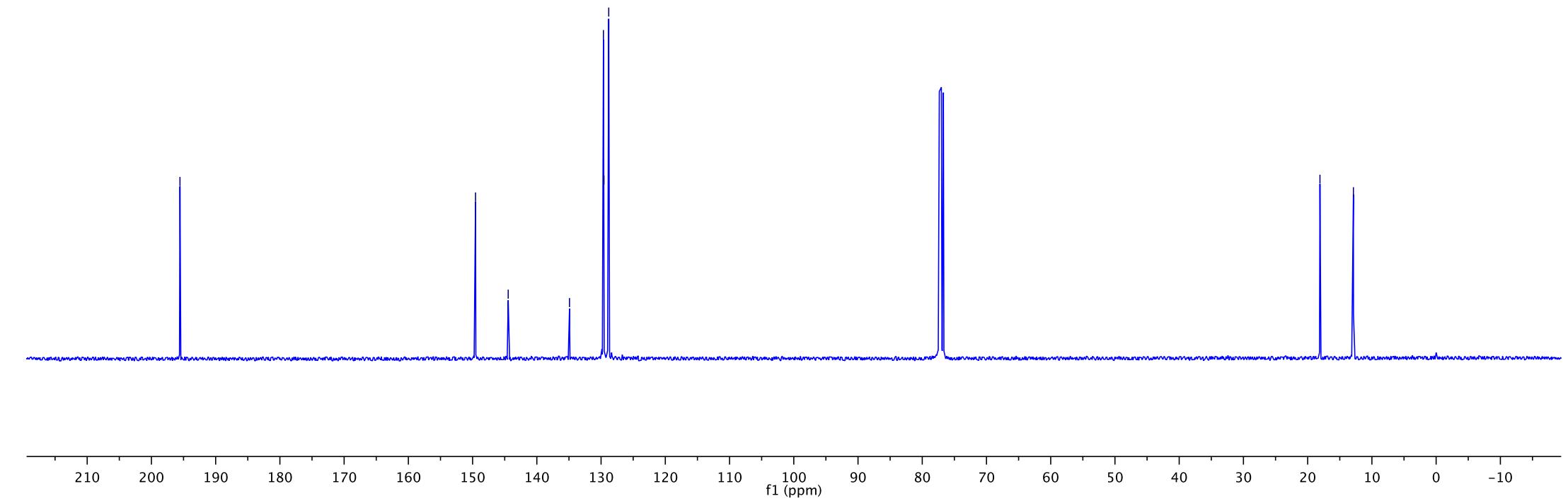
— 18.09

— 12.88

S52

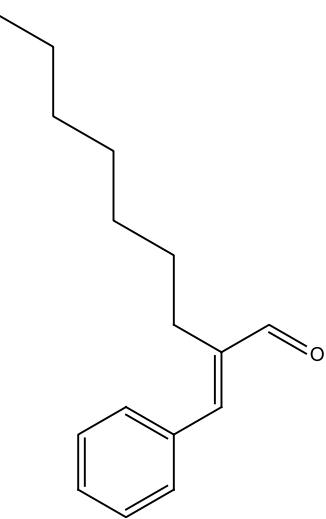


(E)-2-Benzylidenebutanal (23)

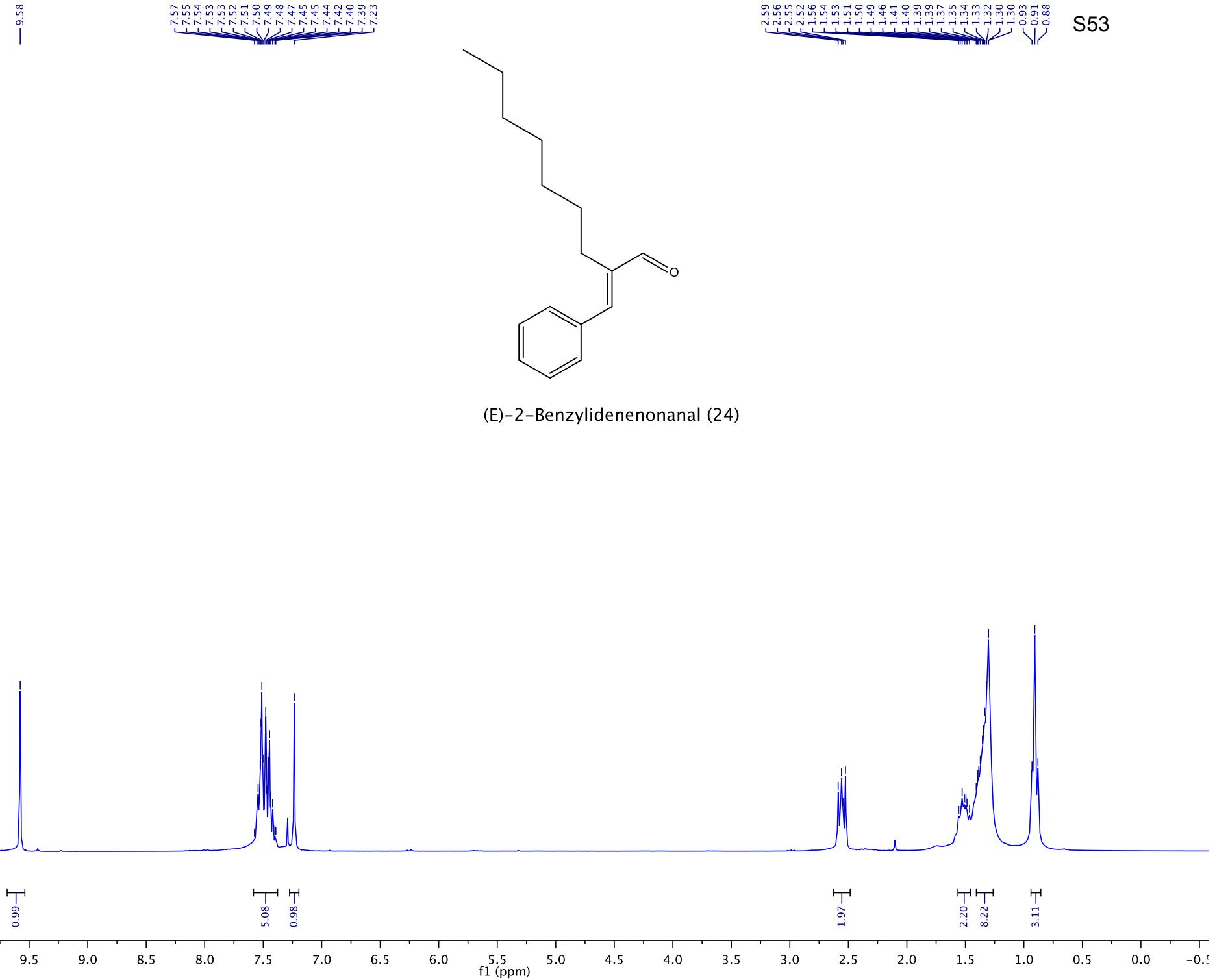


7.57
7.55
7.54
7.53
7.52
7.51
7.50
7.49
7.48
7.47
7.45
7.45
7.44
7.42
7.40
7.39
7.23

2.59
2.56
2.55
2.52
1.56
1.54
1.53
1.51
1.50
1.49
1.46
1.41
1.40
1.39
1.39
1.37
1.35
1.34
1.33
1.32
1.30
1.30
0.93
0.91
0.88

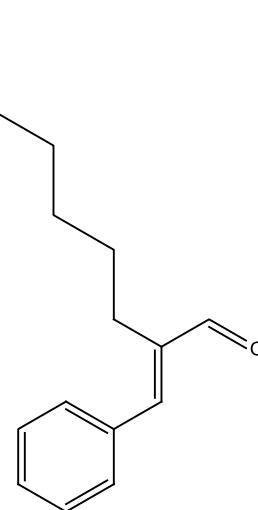


(E)-2-Benzylidenenonanal (24)

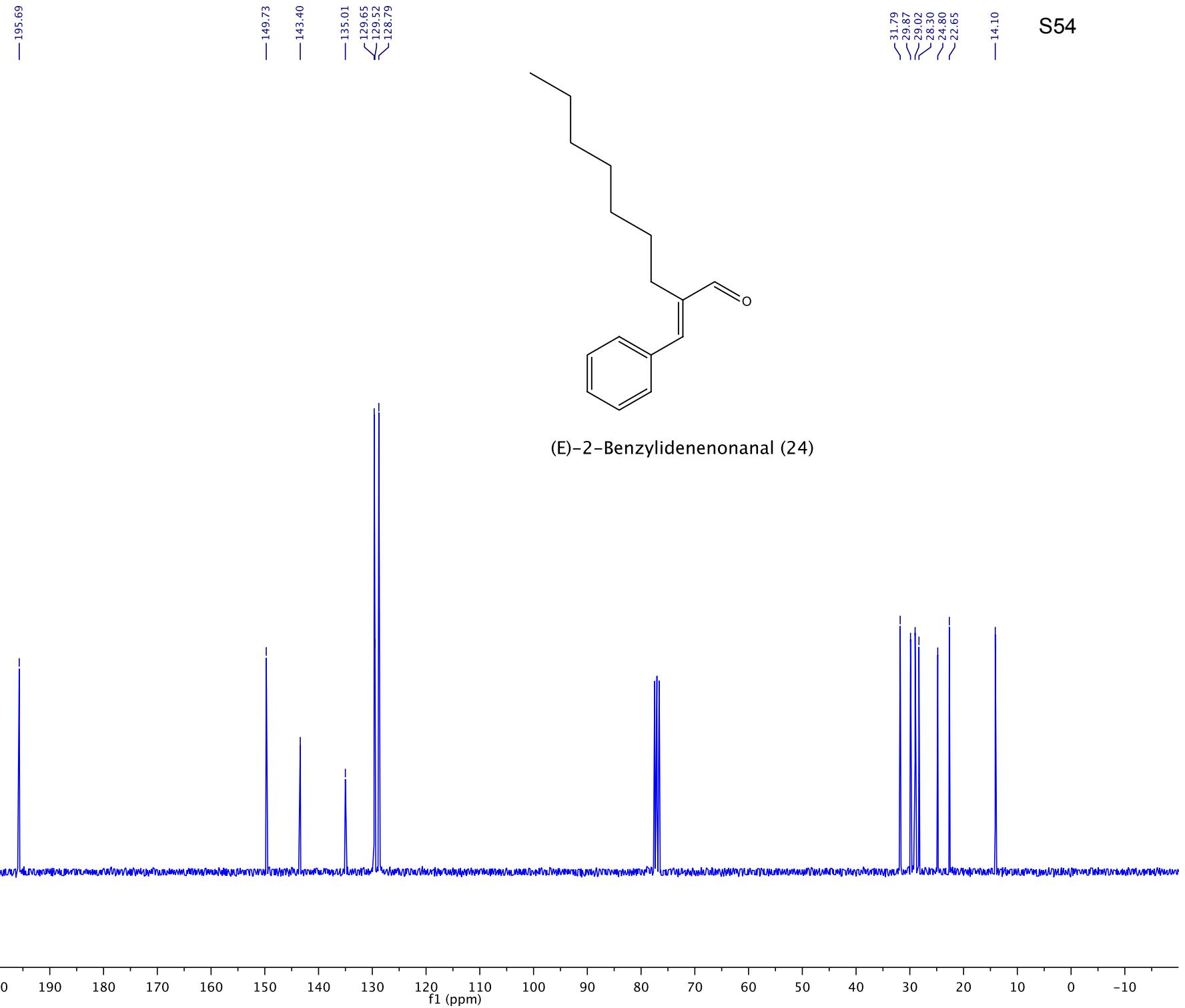


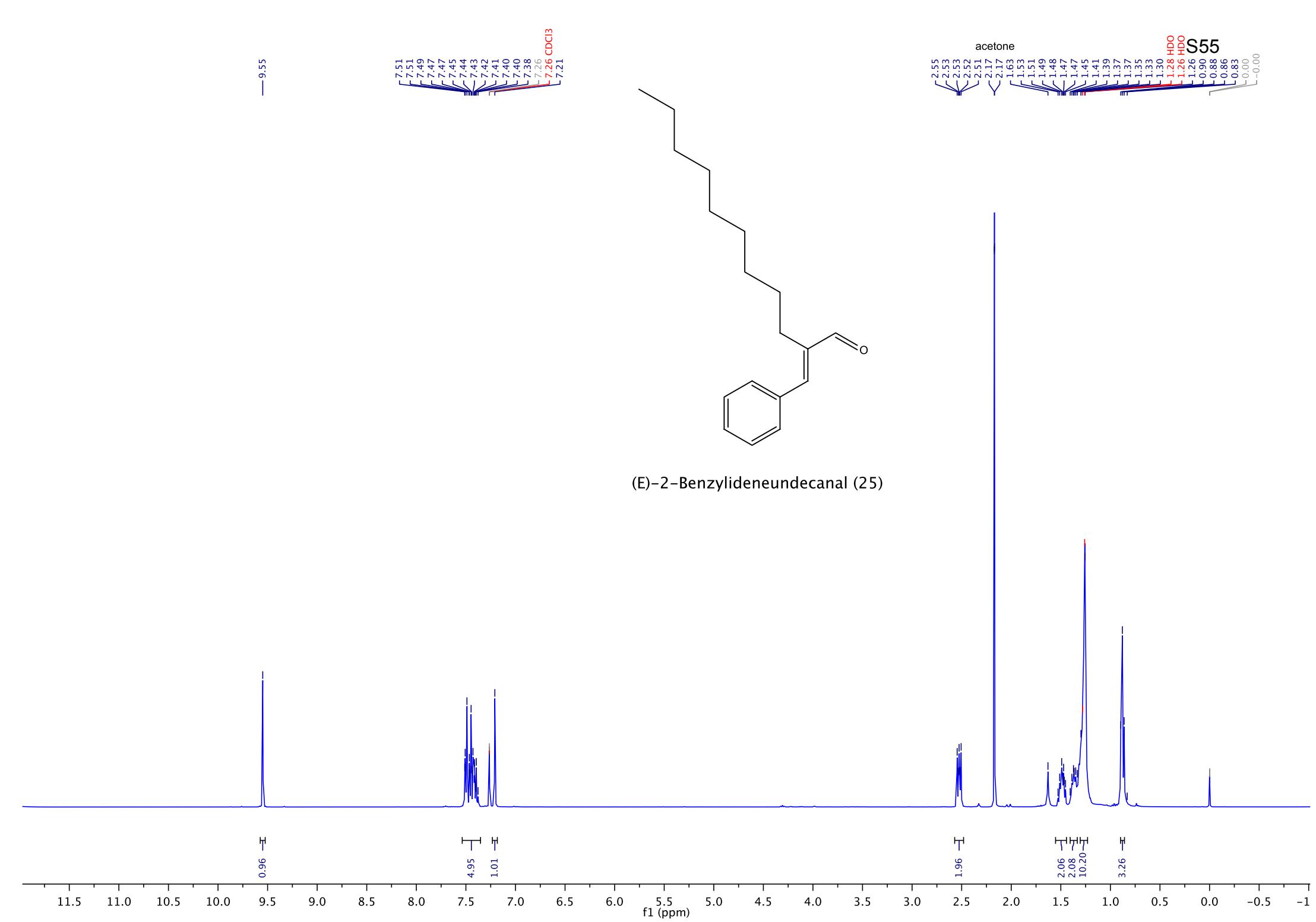
— 31.79
— 29.87
— 29.02
— 28.30
— 24.80
— 22.65

— 14.10

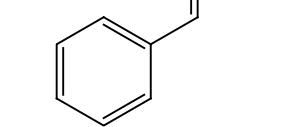


(E)-2-Benzylidenenonanal (24)





31.88
30.92
29.90
29.53
29.30
28.29
24.80
22.68



(E)-2-Benzylideneundecanal (25)

— 195.71

— 149.74

— 143.40

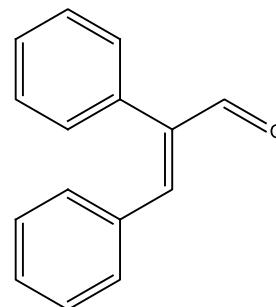
— 135.01

129.64
129.51
128.78

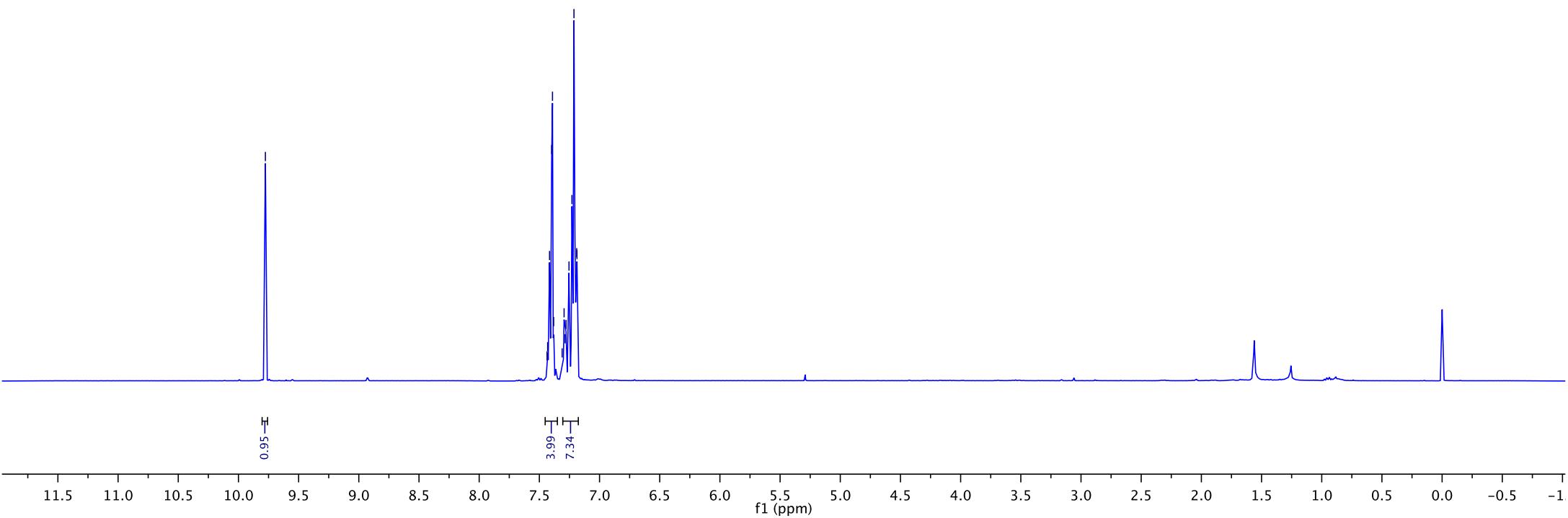
acetone

— 9.78 —

7.44
7.43
7.42
7.40
7.39
7.38
7.31
7.29
7.28
7.25
7.23
7.21
7.19
7.19



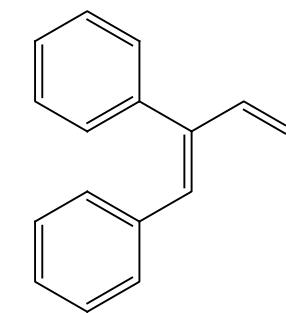
(E)-2,3-diphenylacrylaldehyde (26)



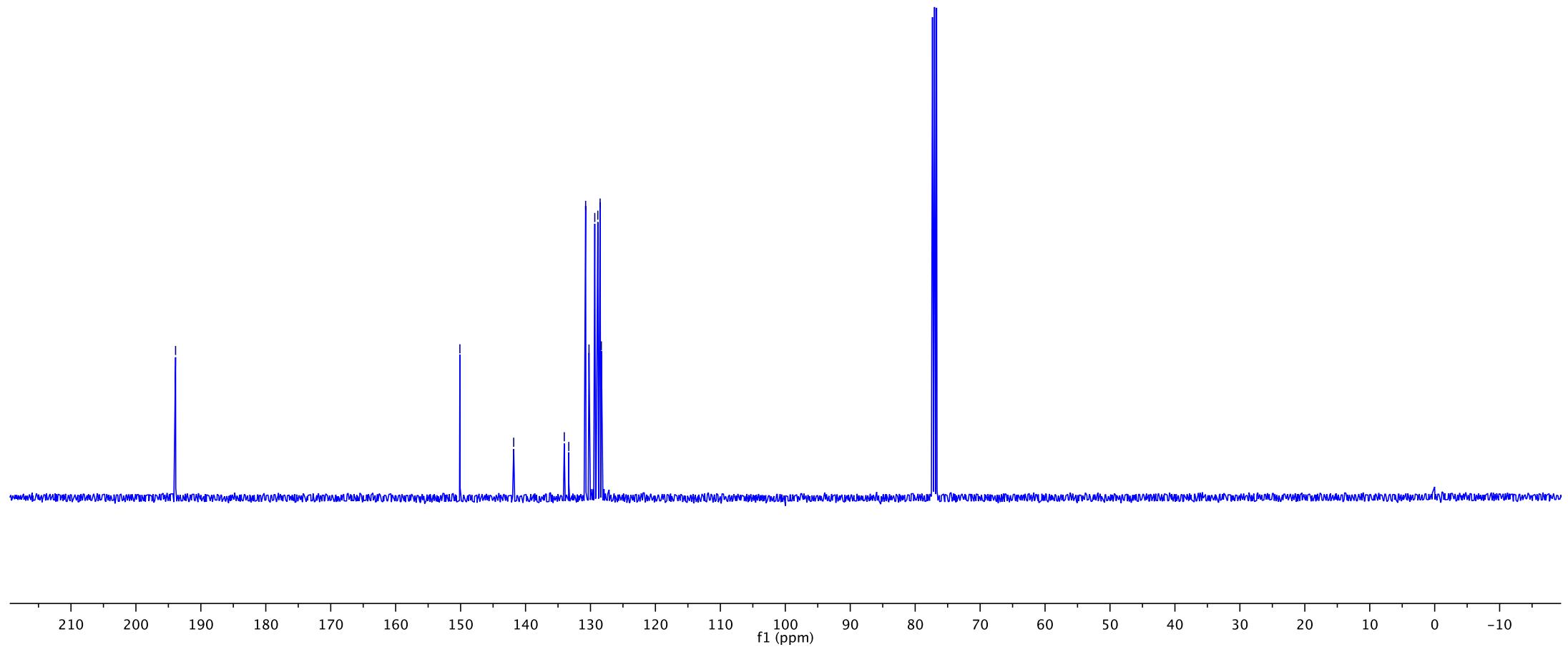
— 193.89

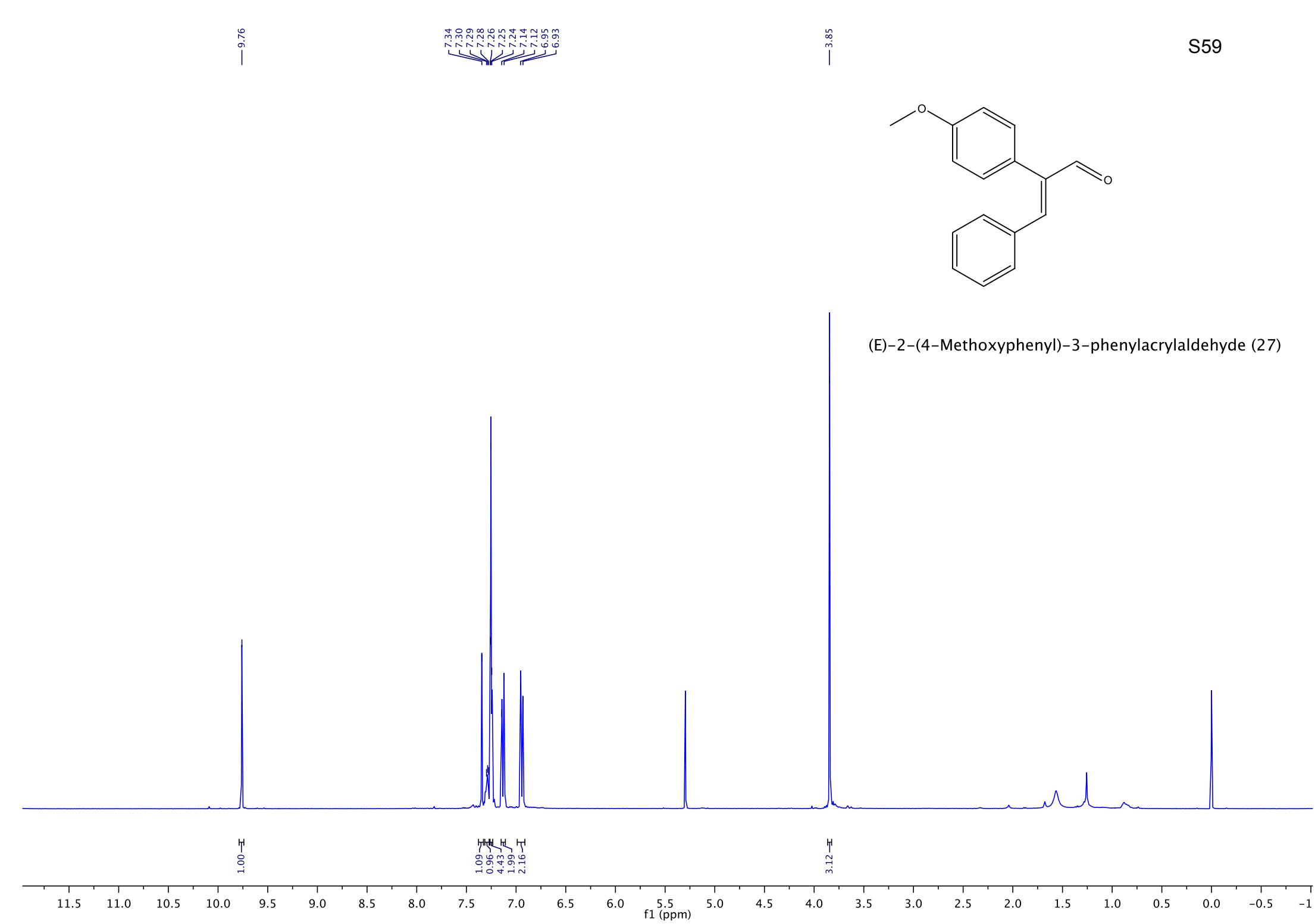
— 150.11

— 141.82
— 134.03
— 133.34
— 130.75
— 130.23
— 129.34
— 128.87
— 128.51
— 128.33



(E)-2,3-diphenylacrylaldehyde (26)





— 194.28

— 159.61

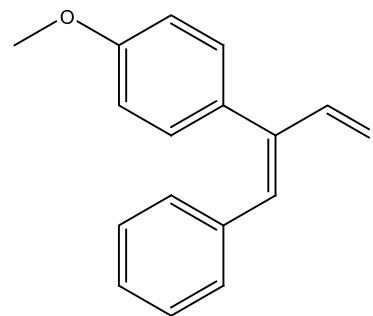
— 149.92

— 141.37

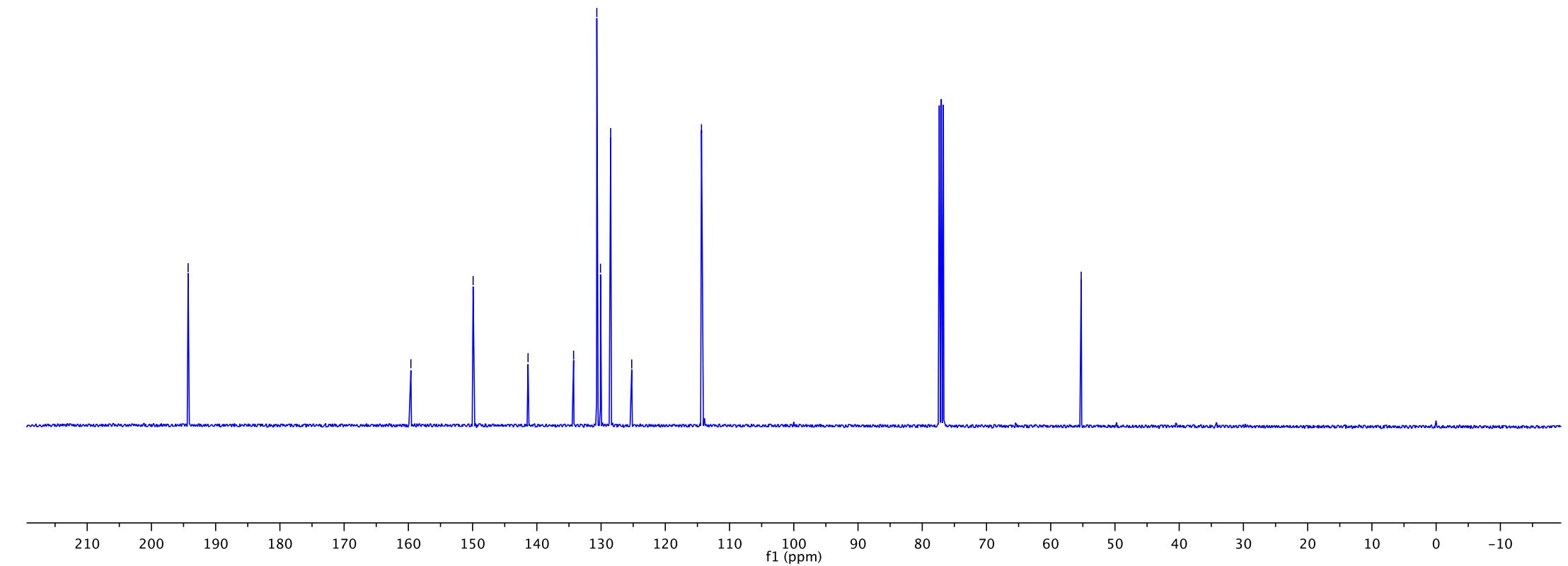
— 134.28
— 130.66
— 130.09
— 128.51
— 125.23

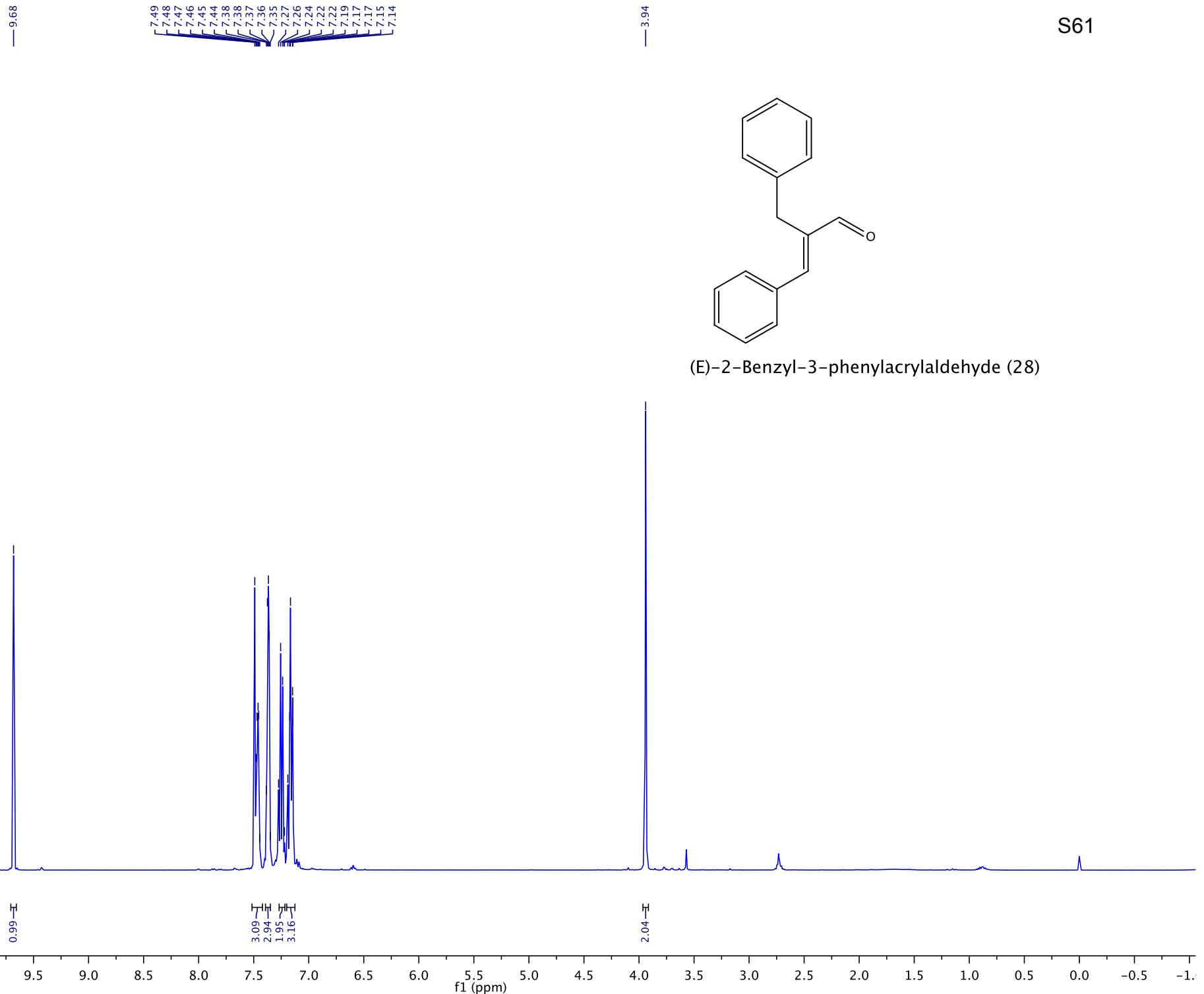
— 114.37

— 55.26

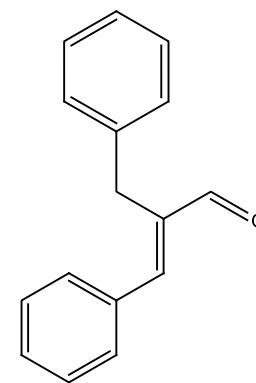


(E)-2-(4-Methoxyphenyl)-3-phenylacrylaldehyde (27)

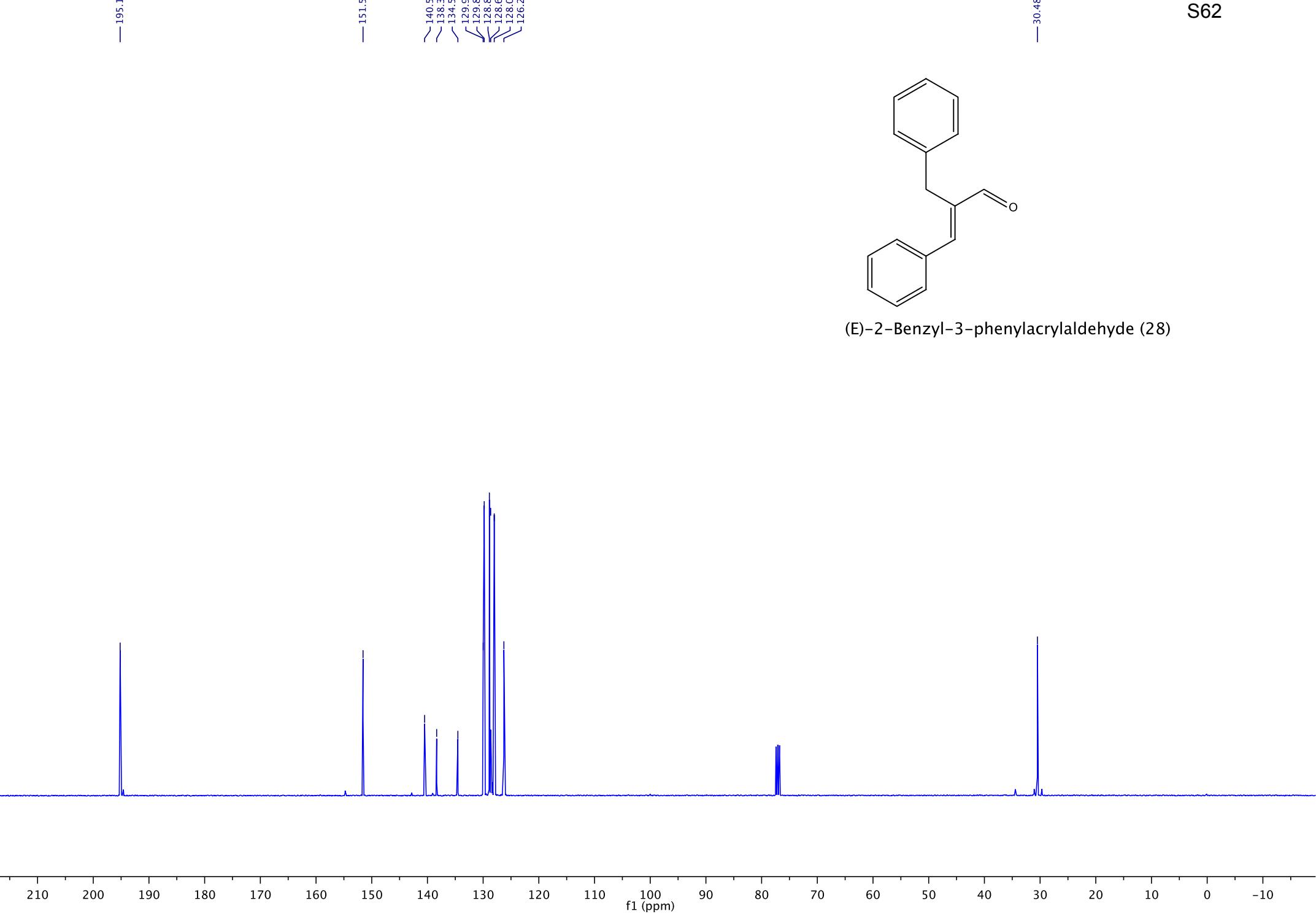


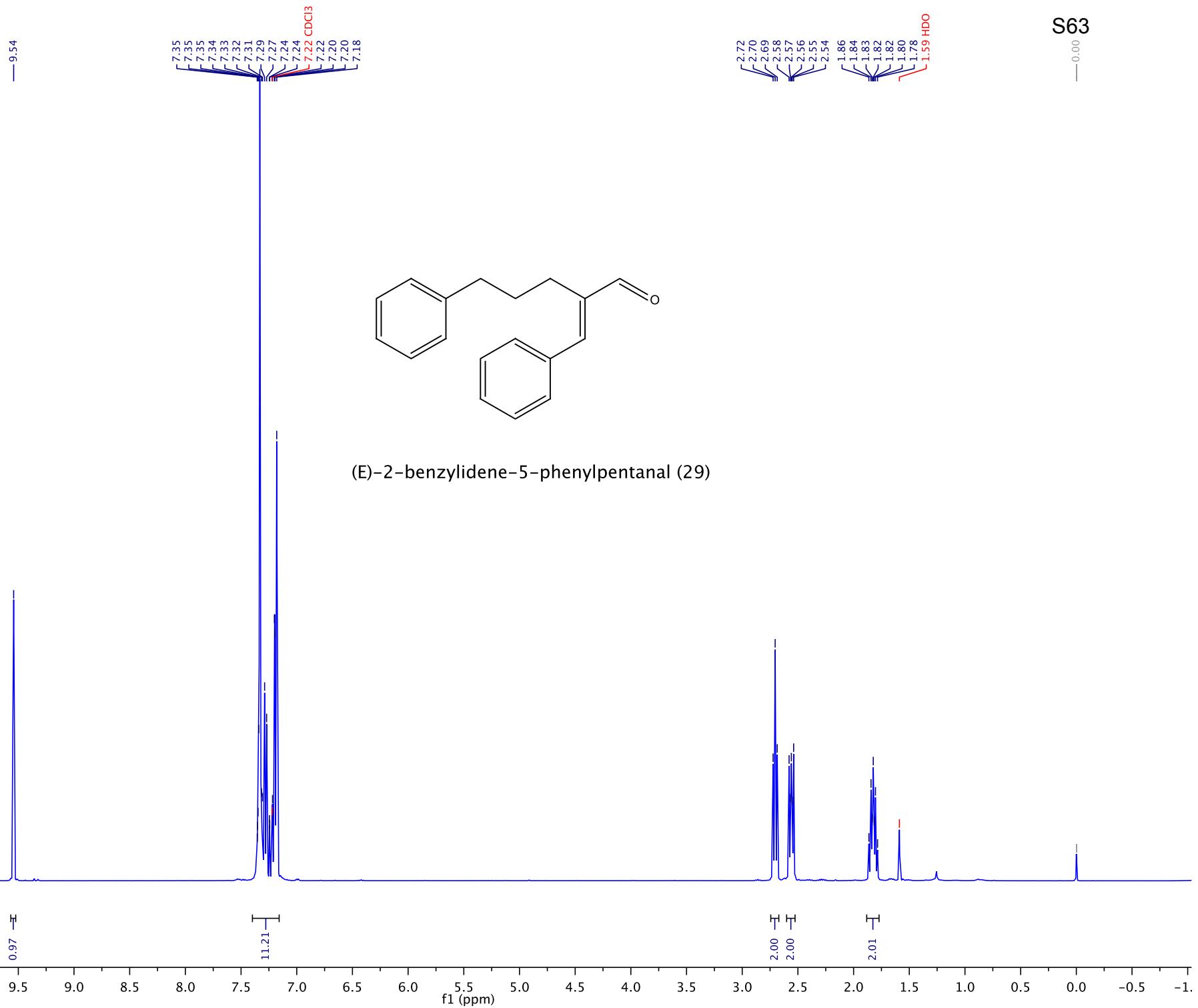


— 30.48



(E)-2-Benzyl-3-phenylacrylaldehyde (28)





— 195.64

— 149.97

— 142.80

— 141.75

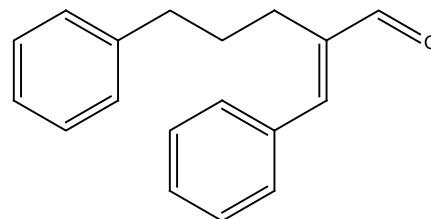
— 134.73
— 129.71
— 129.59
— 128.78
— 128.64
— 128.35
— 125.92

— 77.36 CDCl₃
— 77.05 CDCl₃
— 76.73 CDCl₃

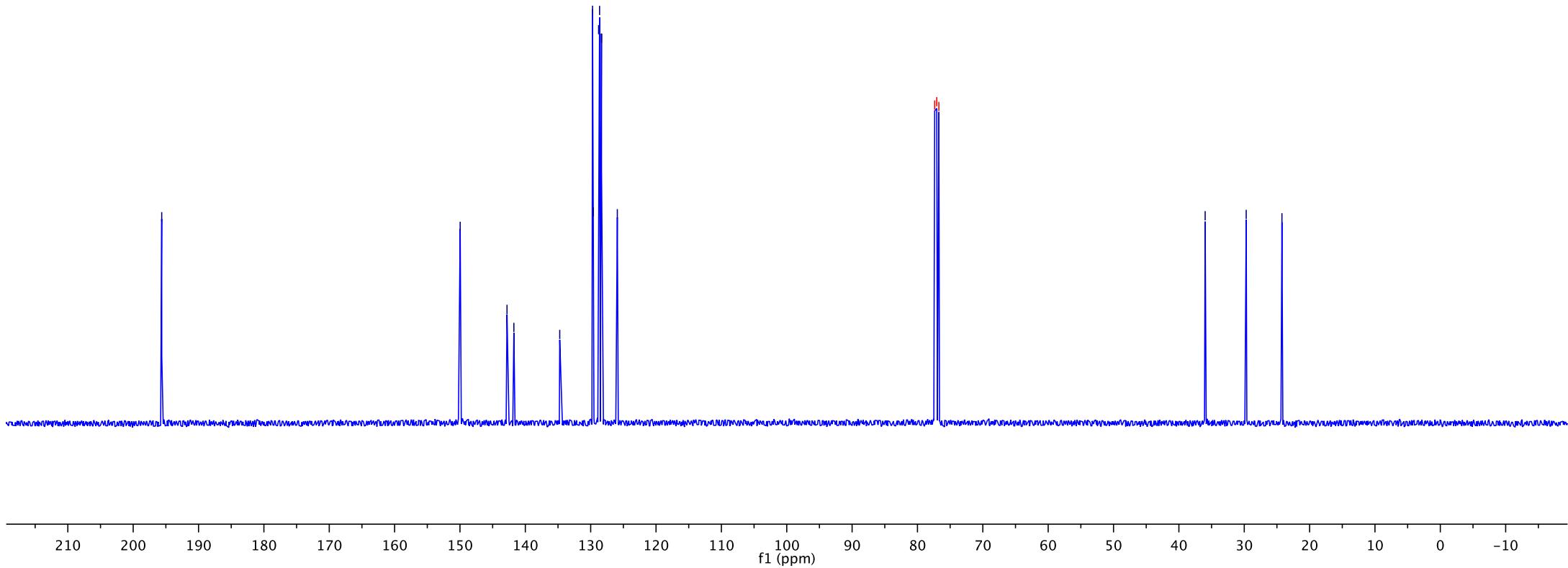
— 35.98

— 29.70

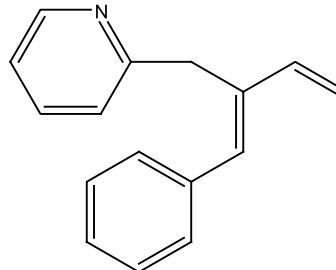
— 24.23



(E)-2-benzylidene-5-phenylpentanal (29)



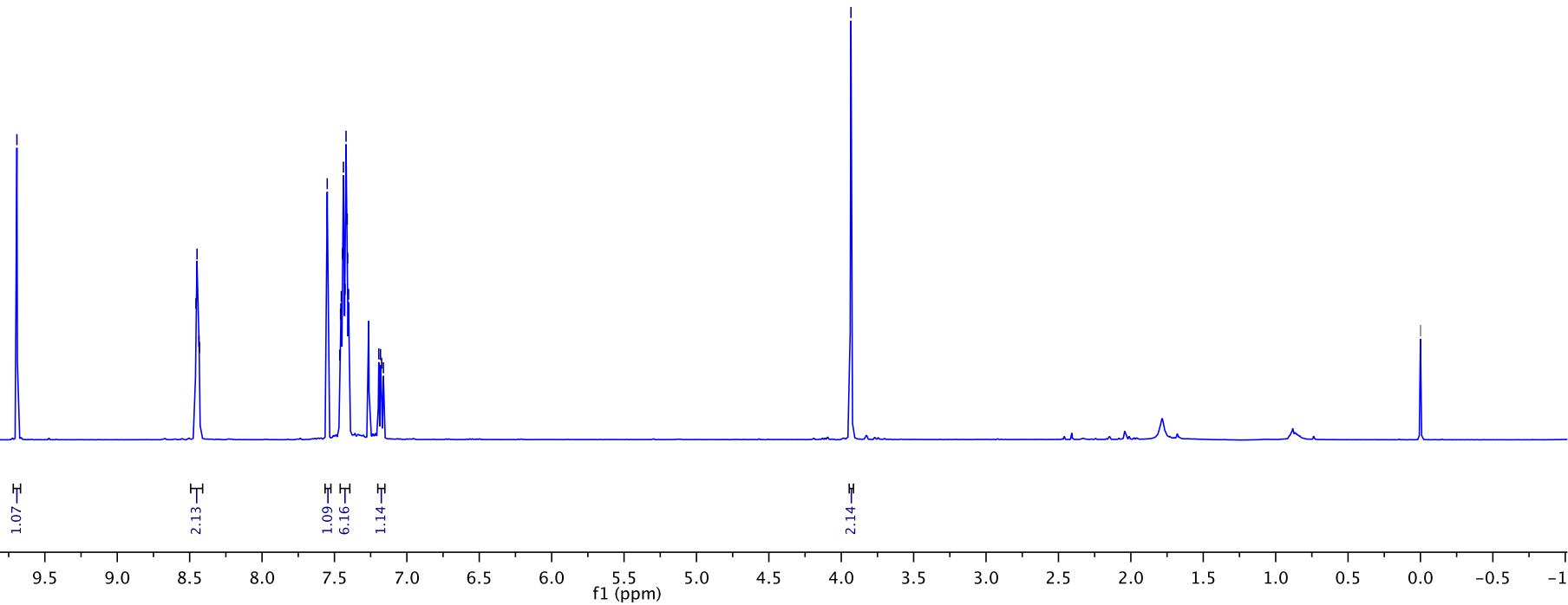
— 9.69 —



8.46
8.45
8.44
8.43
7.55
7.46
7.46
7.45
7.44
7.44
7.43
7.42
7.41
7.41
7.40
7.19
7.18
7.17
7.16

— 3.93 —

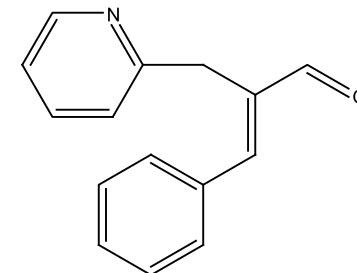
(E)-3-phenyl-2-(pyridin-2-ylmethyl)acrylaldehyde (30)



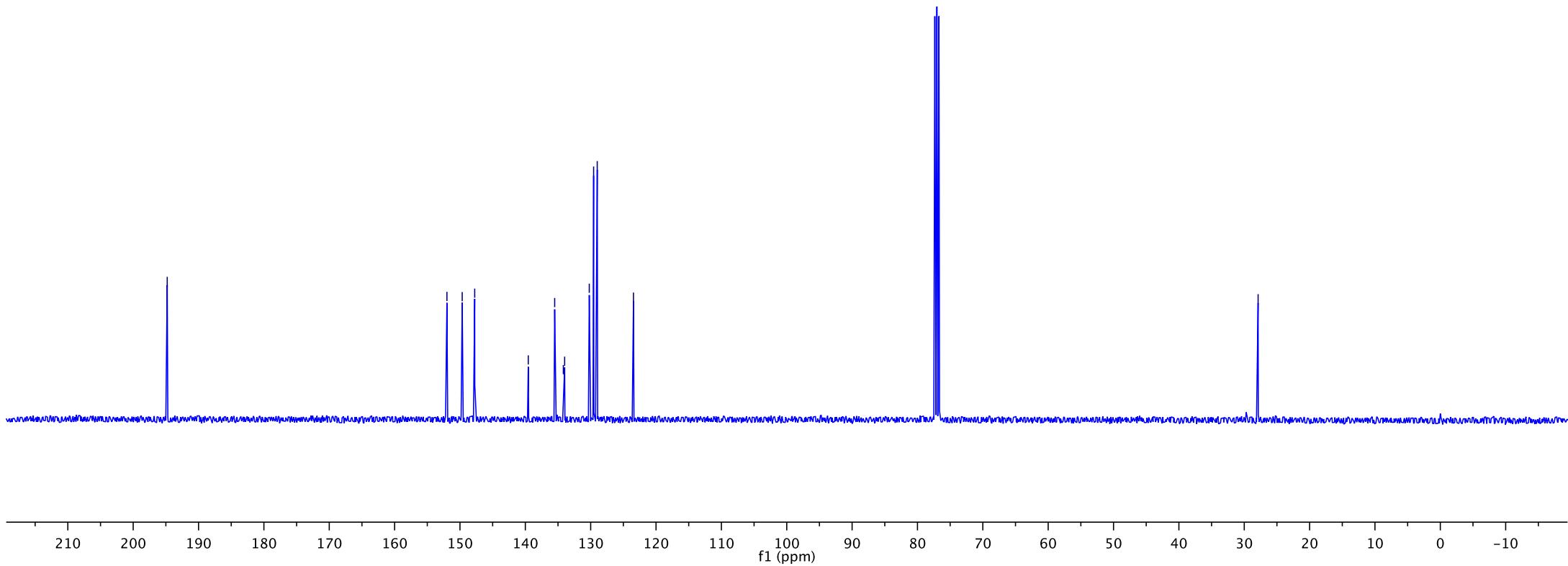
— 194.79

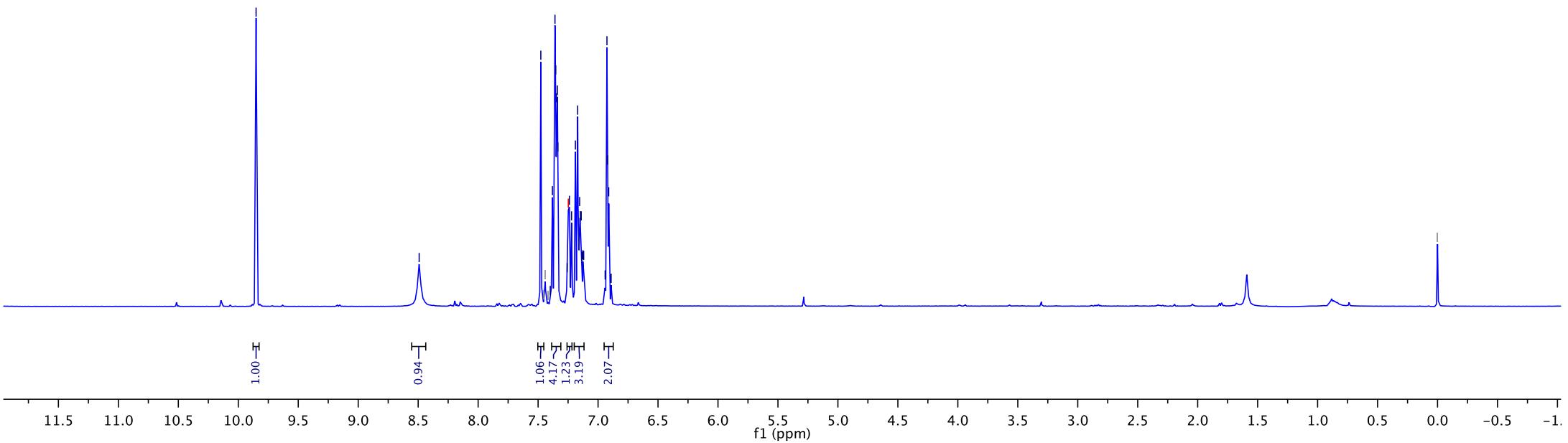
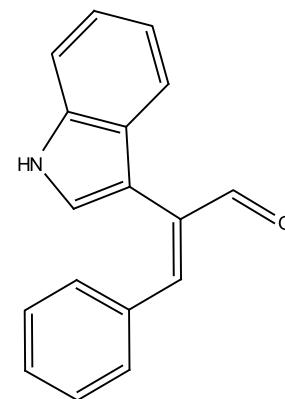
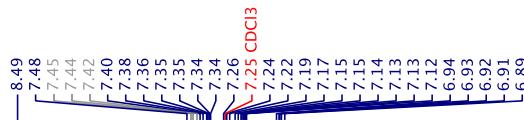
— 151.99
— 149.66
— 147.74
— 139.55
— 135.51
— 134.18
— 133.98
— 130.21
— 129.55
— 129.00
— 123.45

— 27.88



(E)-3-phenyl-2-(pyridin-2-ylmethyl)acrylaldehyde (30)



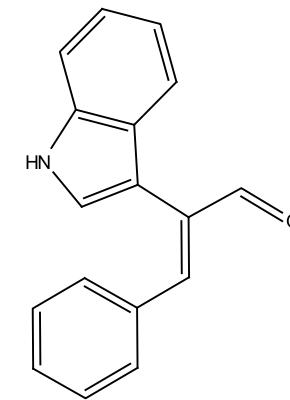


— 194.80

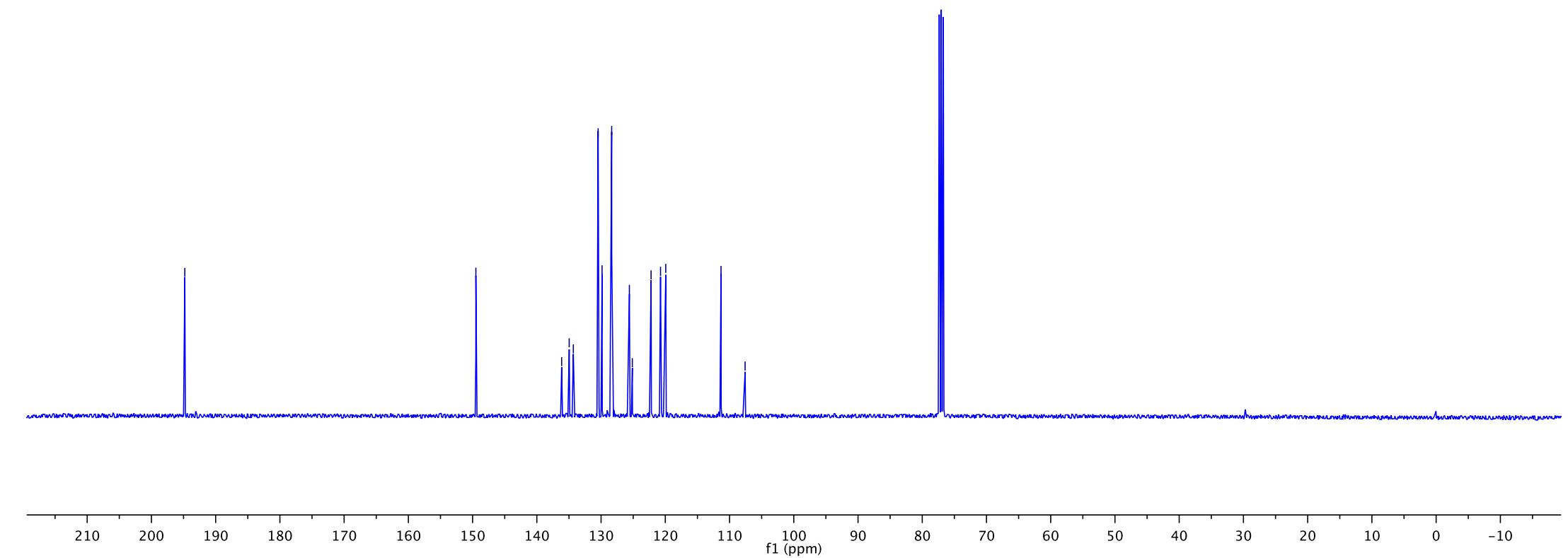
— 149.49

136.14
134.96
134.32
130.46
129.85
128.35
125.59
125.13
122.22
120.74
119.93

— 111.33
— 107.58



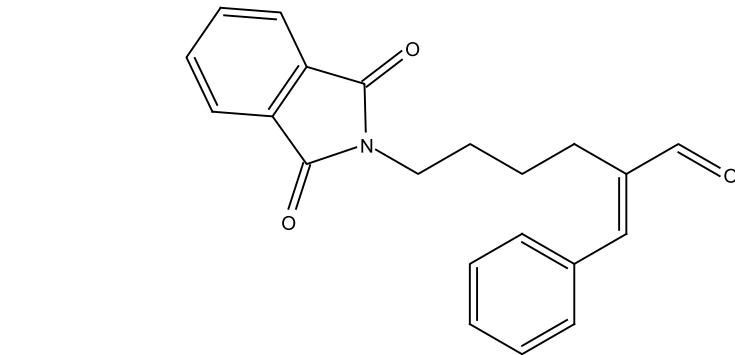
(E)-2-(1H-Indol-3-yl)-3-phenylacrylaldehyde (31)



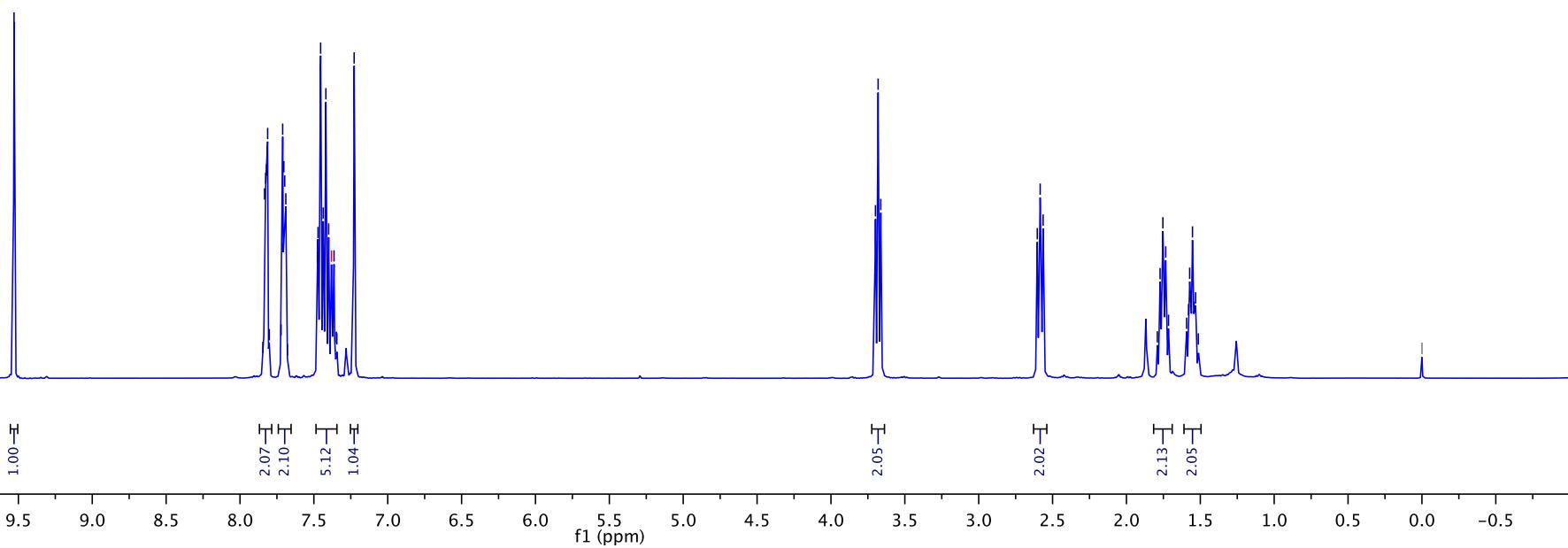
2.60
2.58
2.56
1.79
1.77
1.75
1.73
1.72
1.59
1.58
1.57
1.55
1.54
1.53
1.51

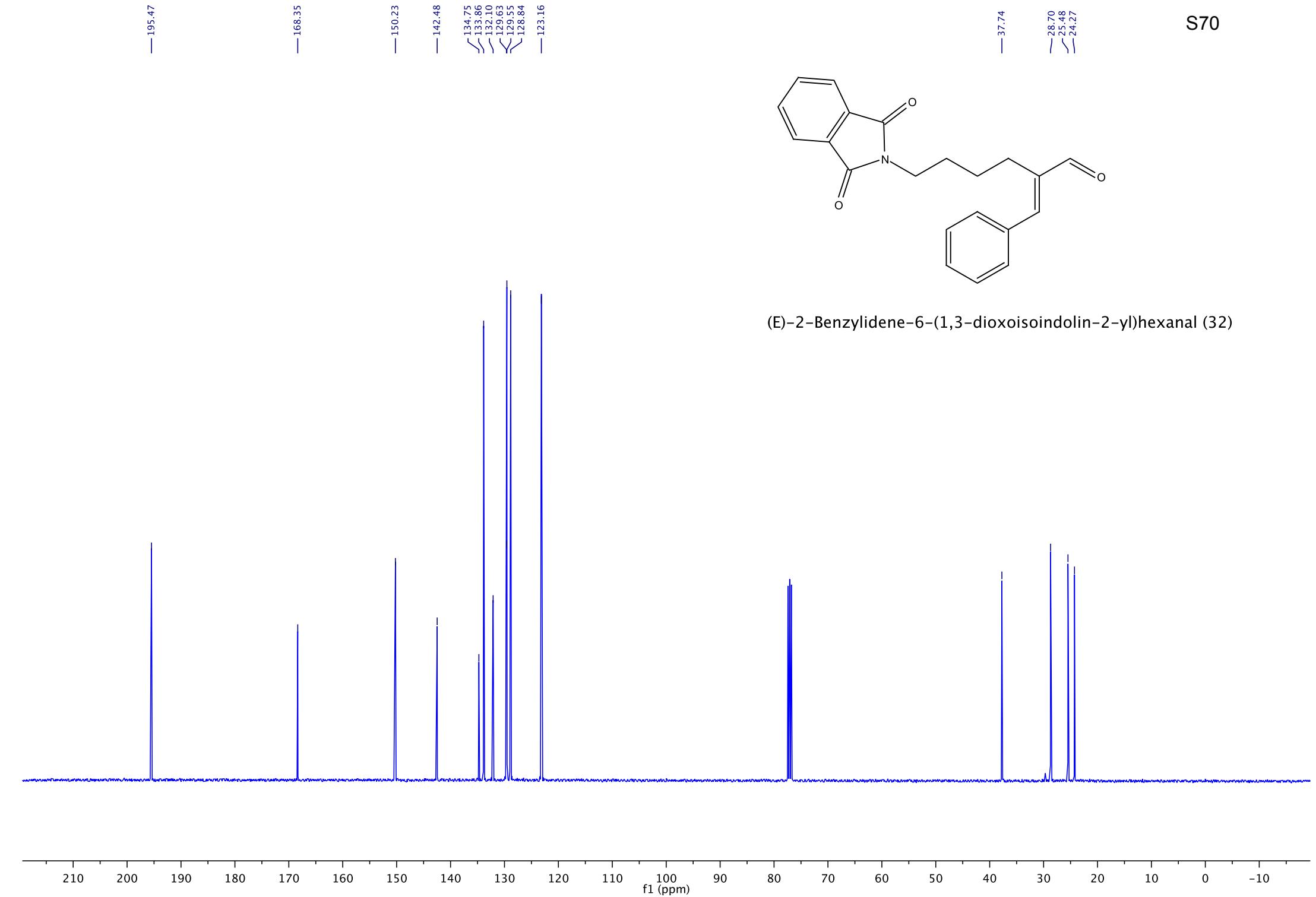
3.70
3.68
3.66

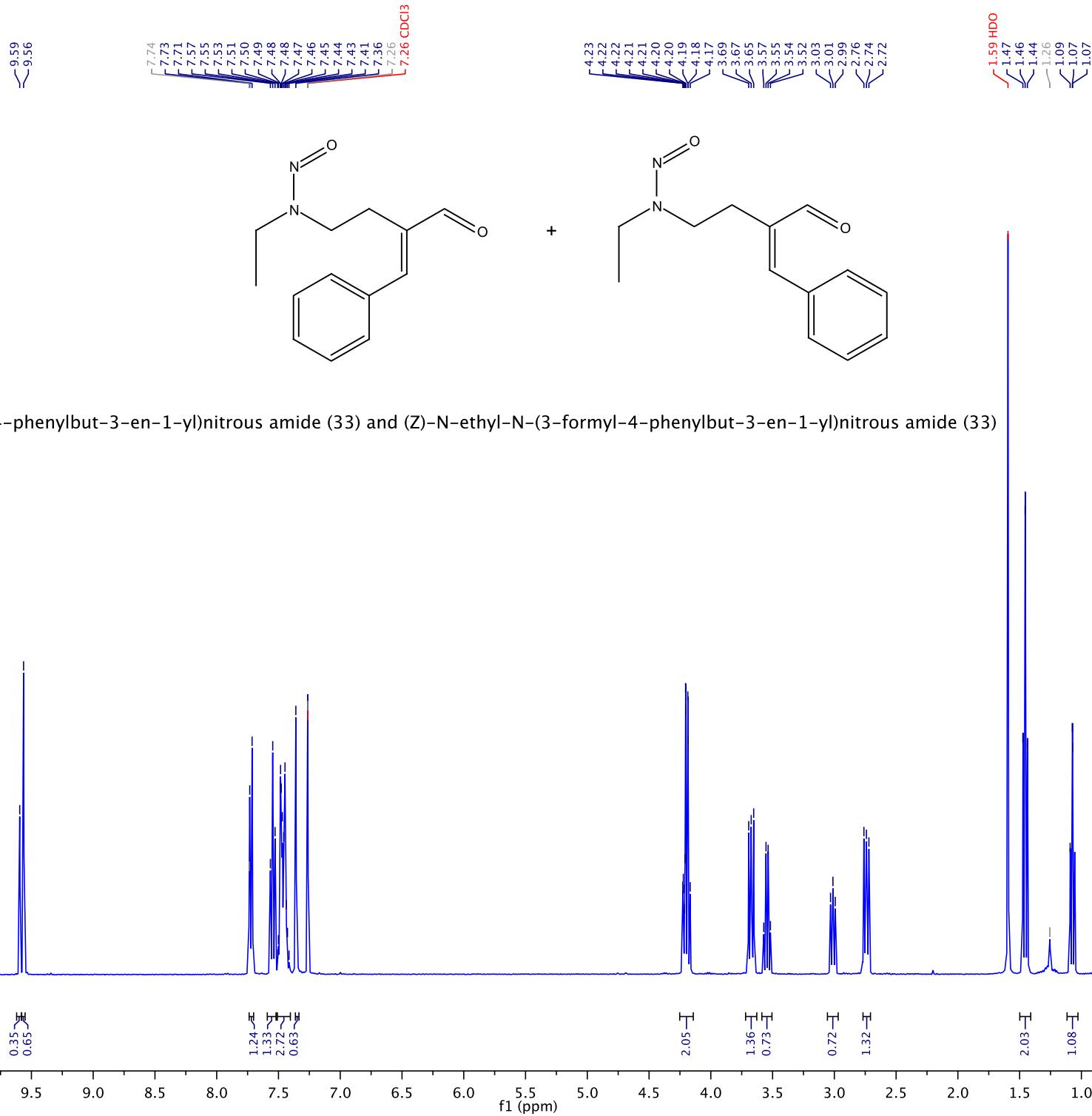
7.85
7.83
7.83
7.82
7.82
7.81
7.81
7.80
7.80
7.72
7.71
7.71
7.70
7.70
7.69
7.68
7.48
7.47
7.45
7.44
7.42
7.40
7.37
7.36
7.35
7.34
7.23

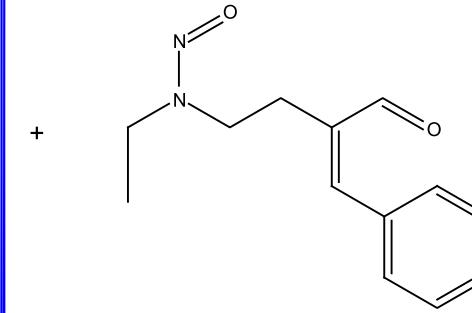
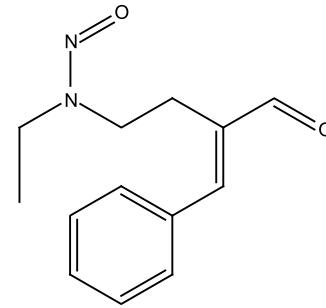


(E)-2-Benzylidene-6-(1,3-dioxoisindolin-2-yl)hexanal (32)

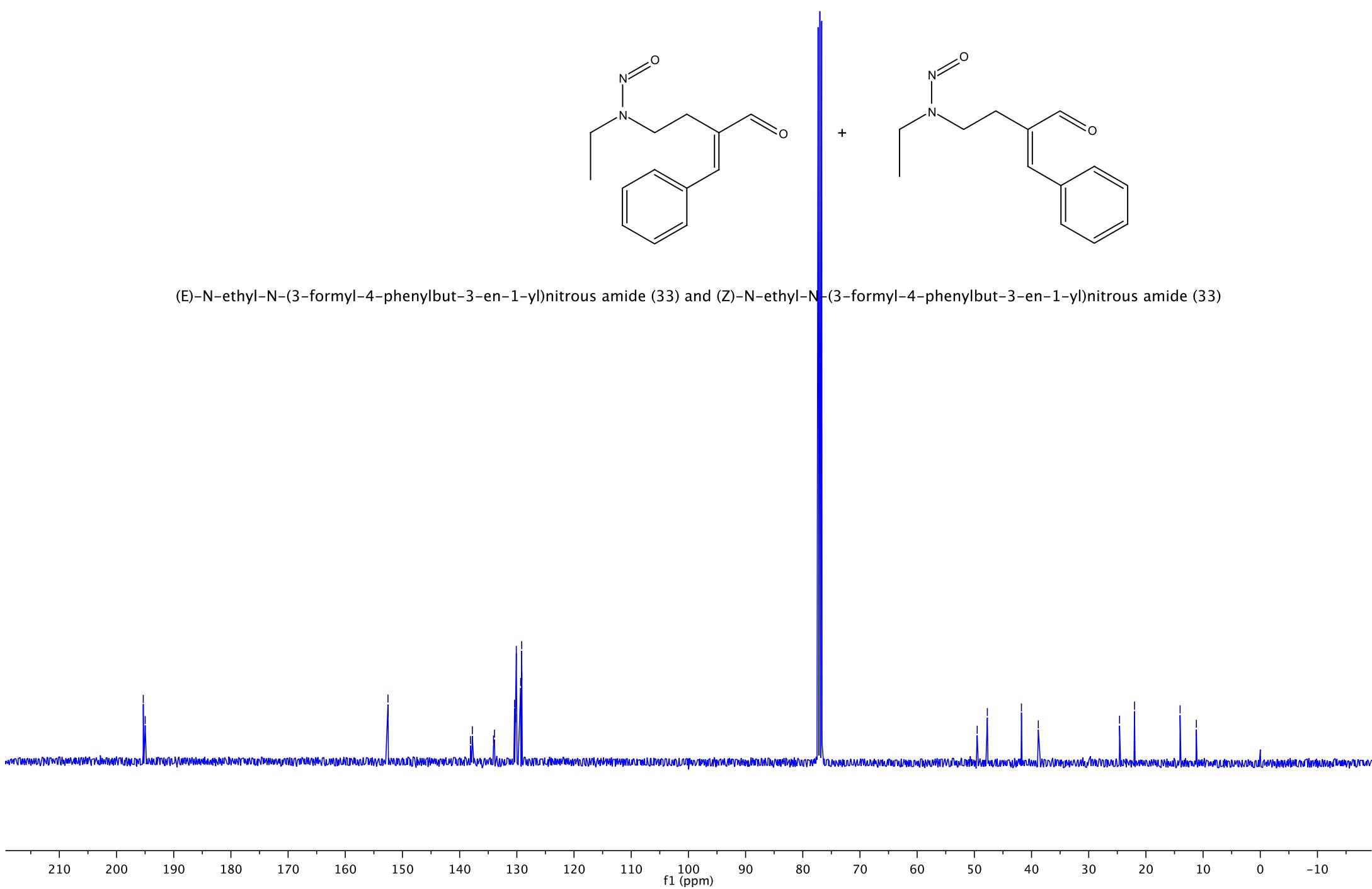






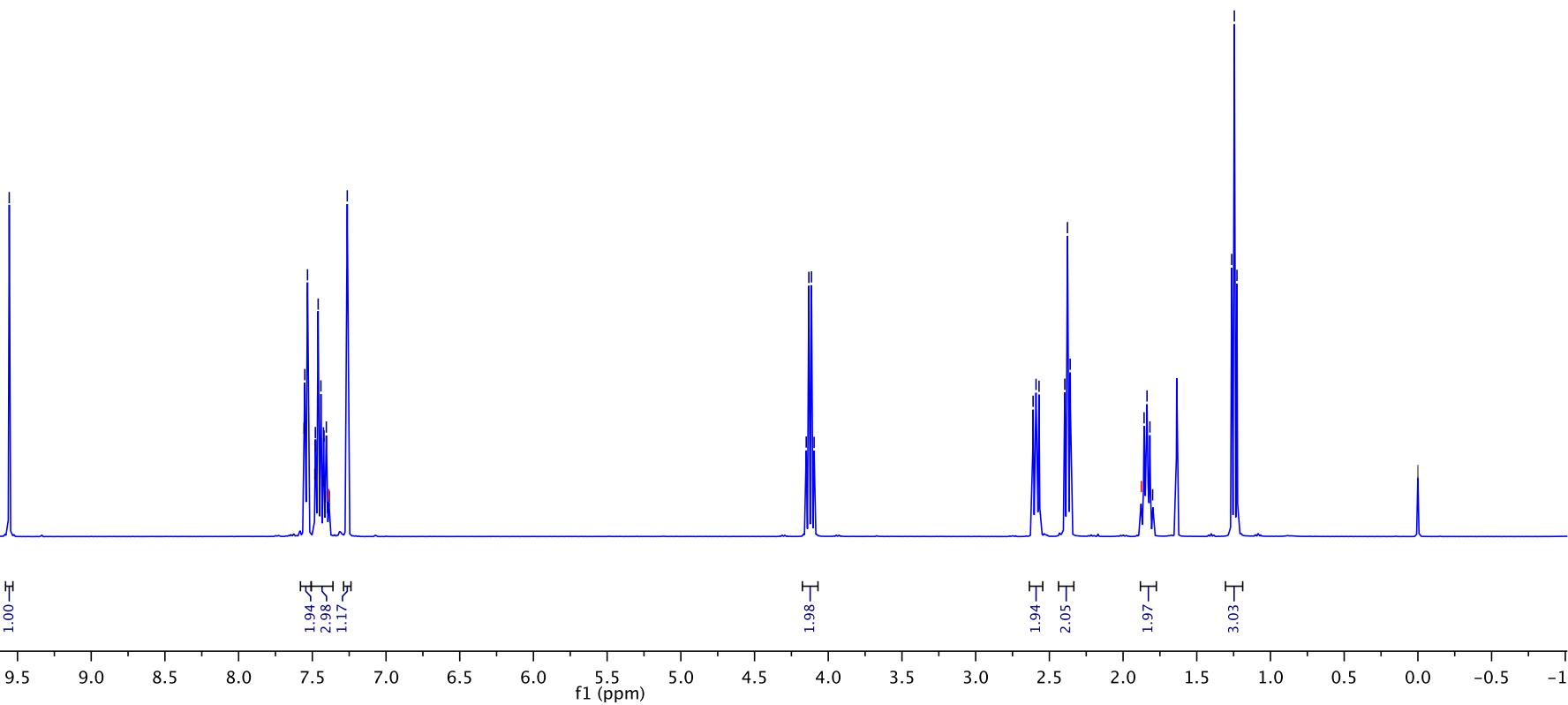
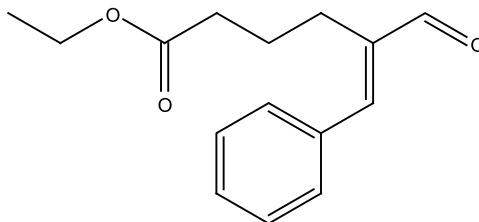
<195.32
<194.98<152.53
<152.52138.11
137.79
134.07
133.91
130.40
130.21
130.10
129.33
129.15
129.13—49.50
—47.72
—41.76
—38.81
—24.61
—22.00
—14.02
—11.17

(E)-N-ethyl-N-(3-formyl-4-phenylbut-3-en-1-yl)nitrous amide (33) and (Z)-N-ethyl-N-(3-formyl-4-phenylbut-3-en-1-yl)nitrous amide (33)

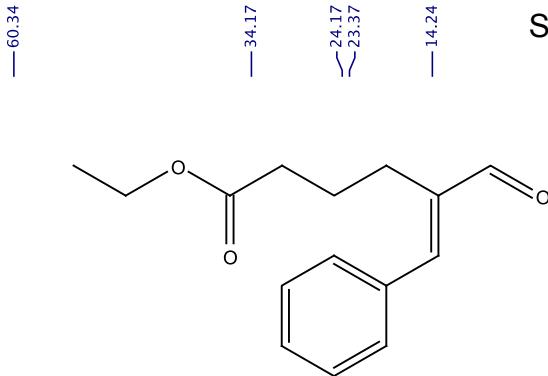




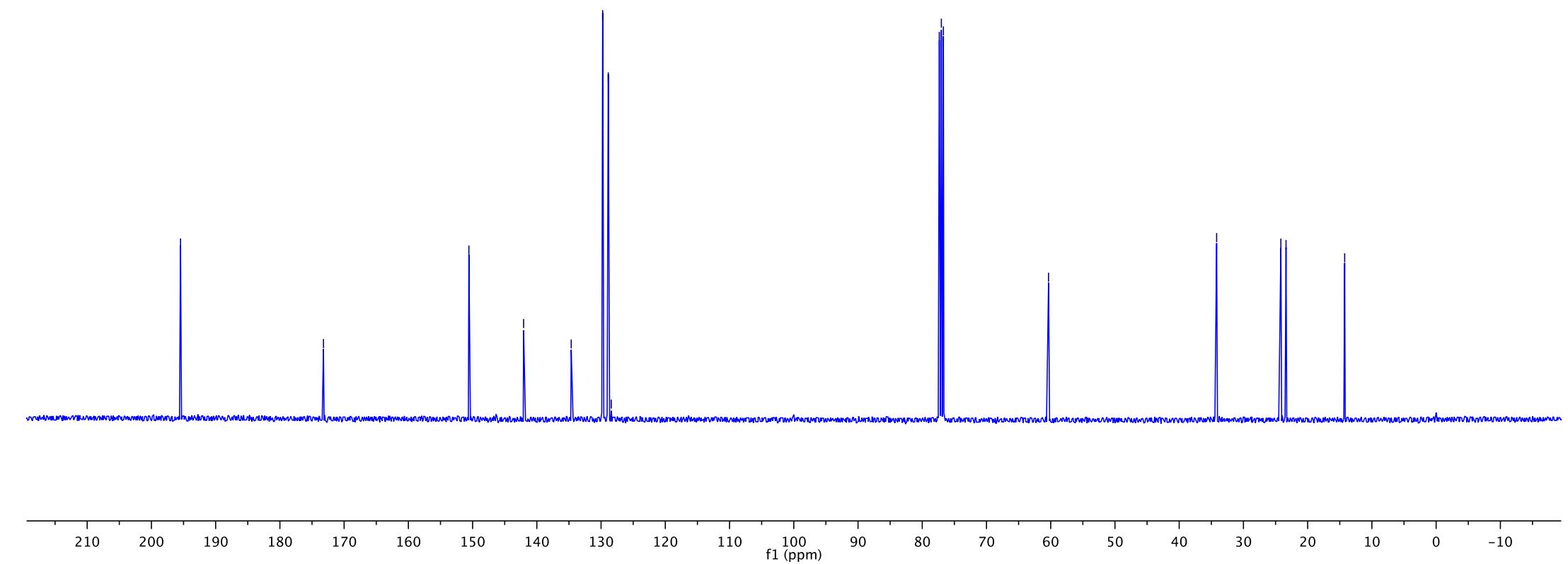
(E)-Ethyl 5-formyl-6-phenylhex-5-enoate (34)

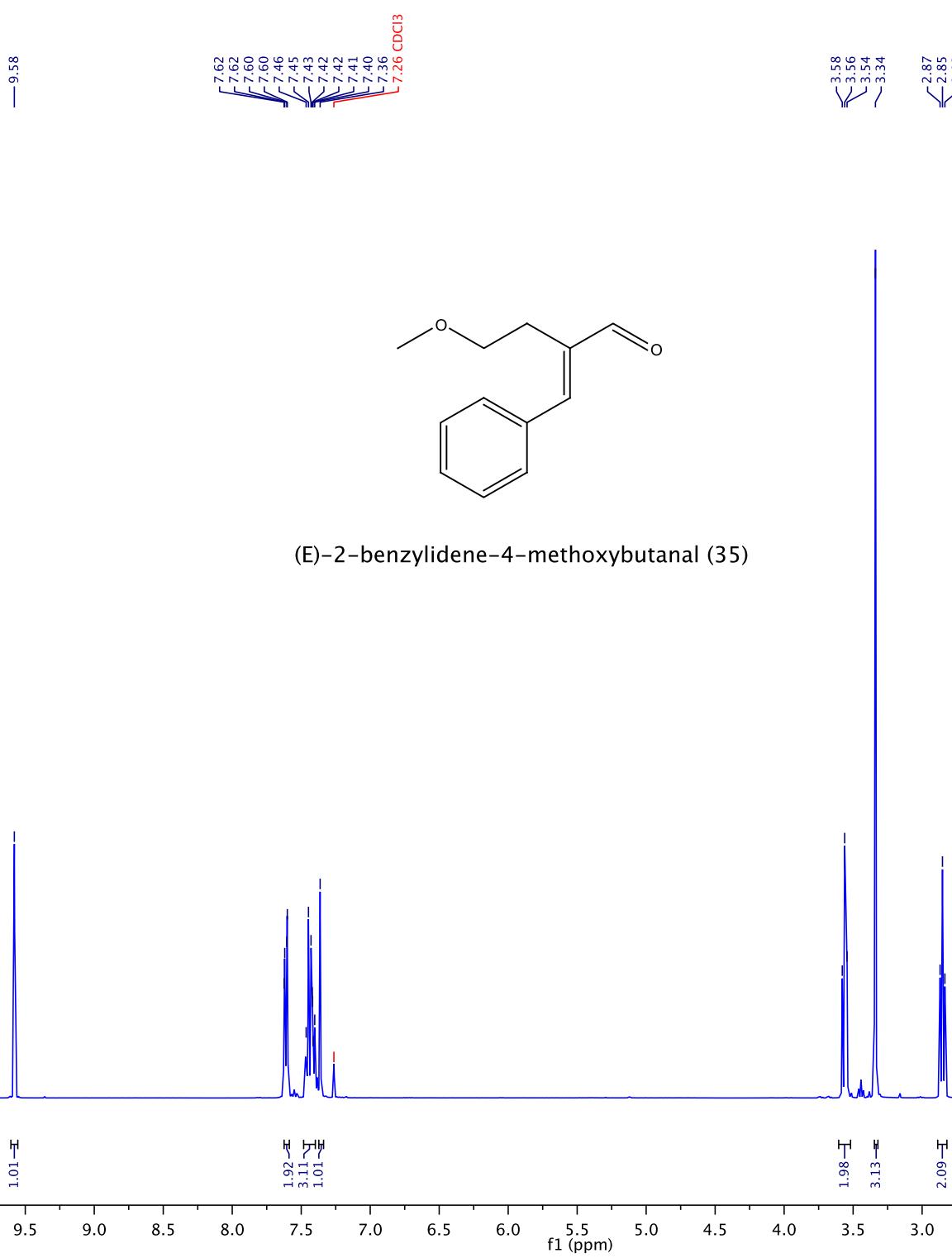


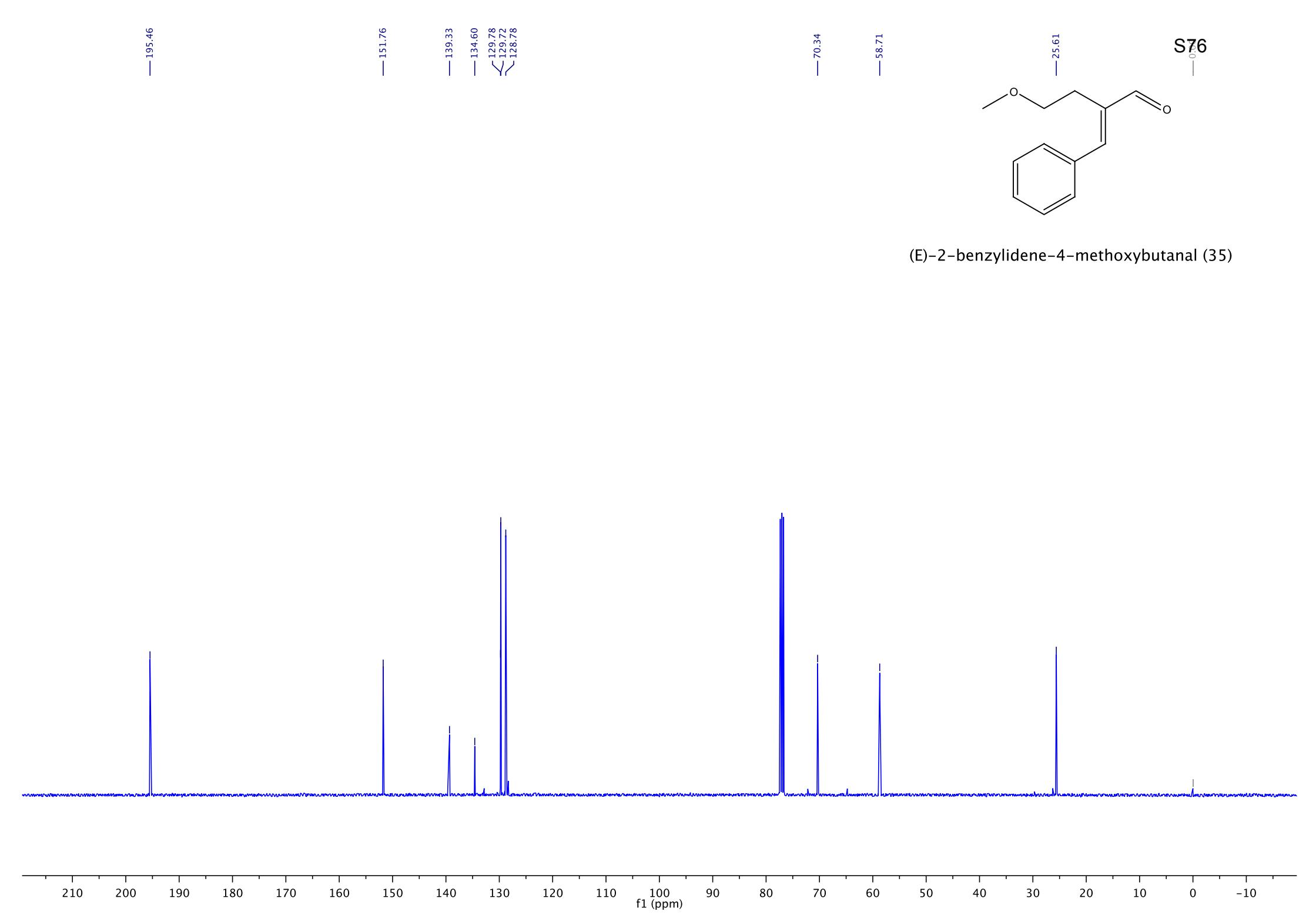
S74



(E)-Ethyl 5-formyl-6-phenylhex-5-enoate (34)

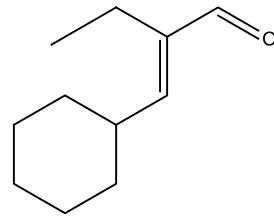
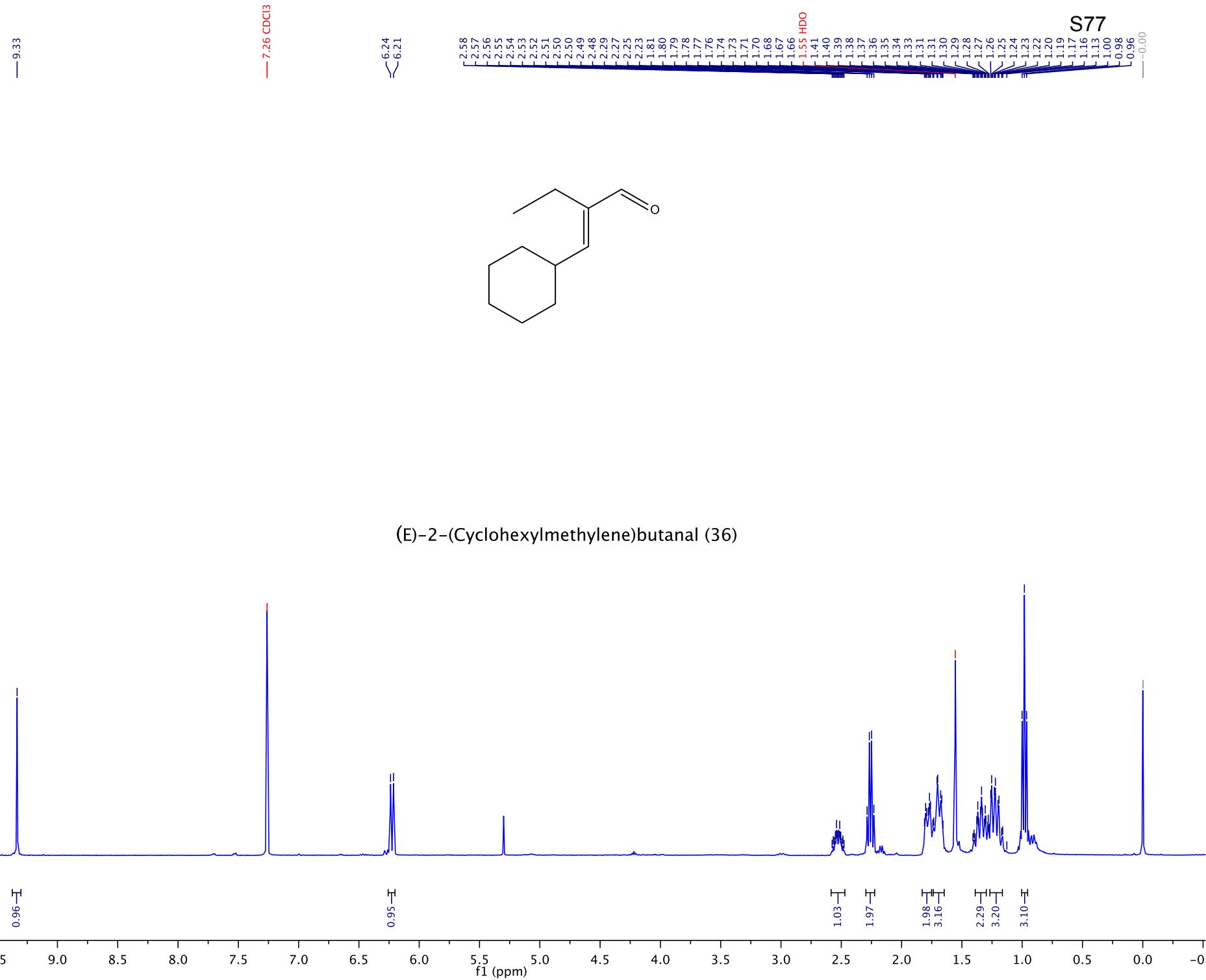






S77

0.96
0.98
1.00

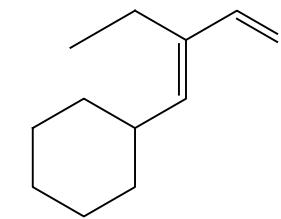


(E)-2-(Cyclohexylmethylene)butanal (36)

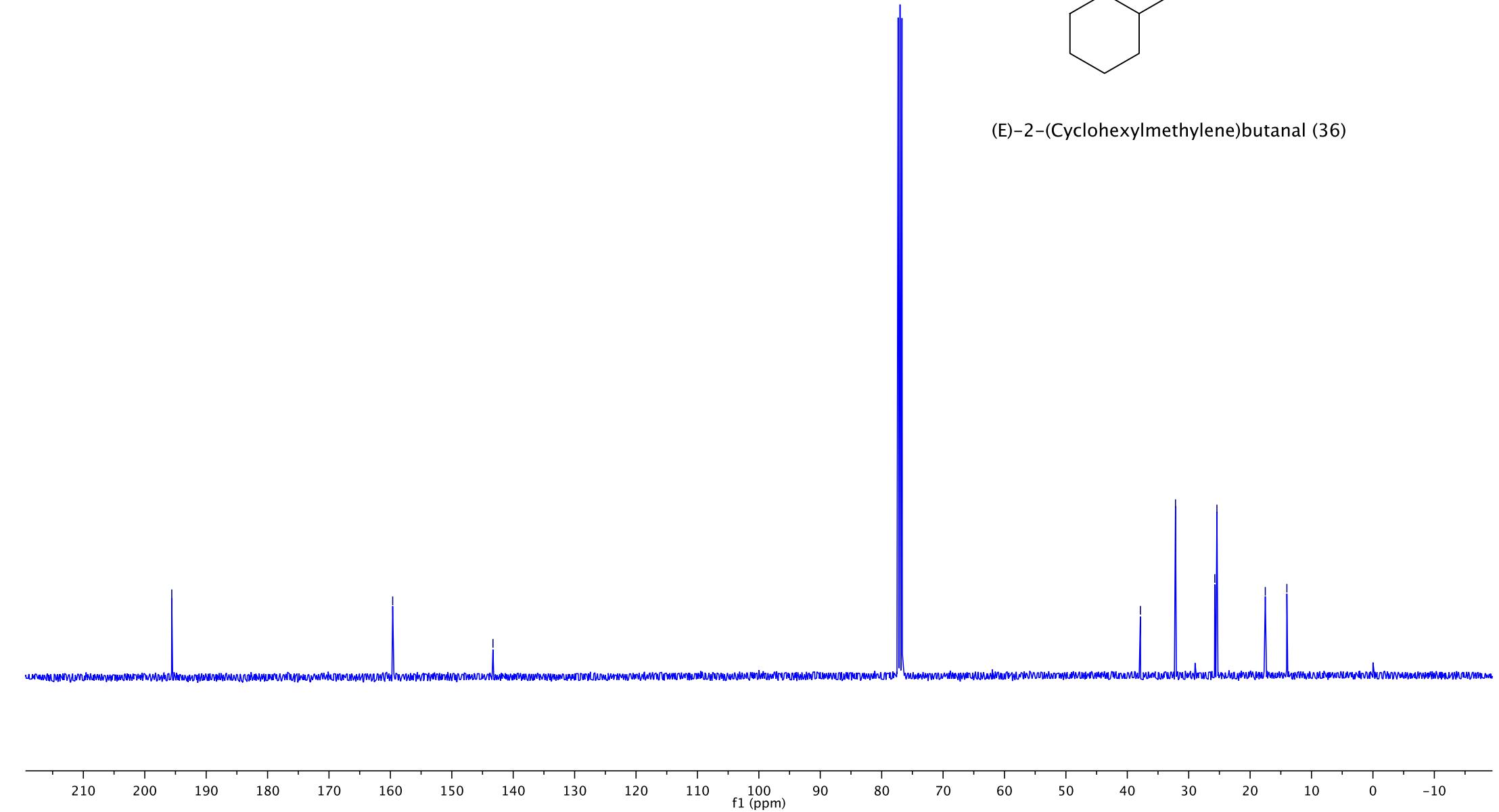
— 195.60
— 159.63
— 143.30

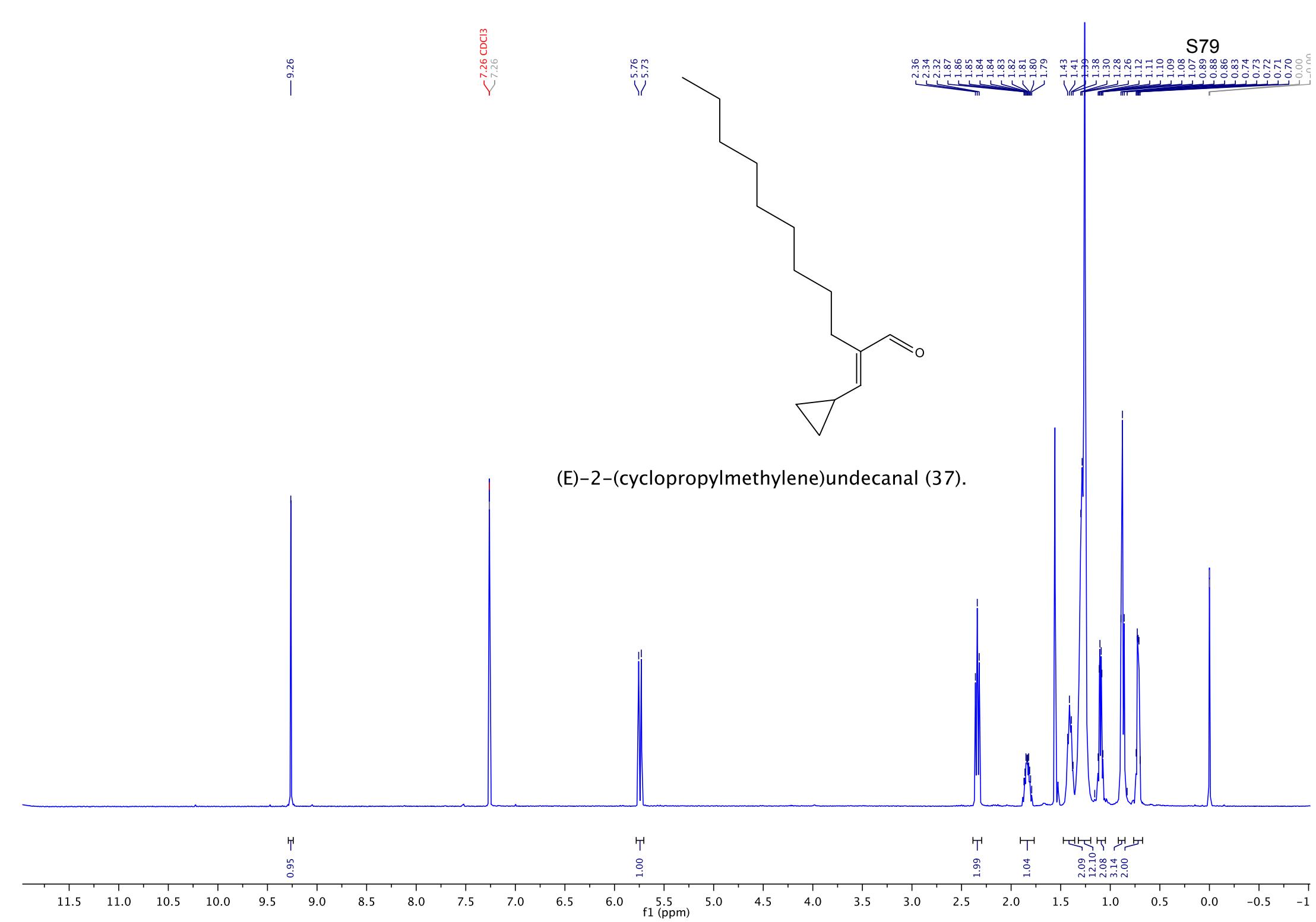
— 37.87
— 32.15
— 25.76
— 25.41

— 17.53
— 14.01



(E)-2-(Cyclohexylmethylene)butanal (36)





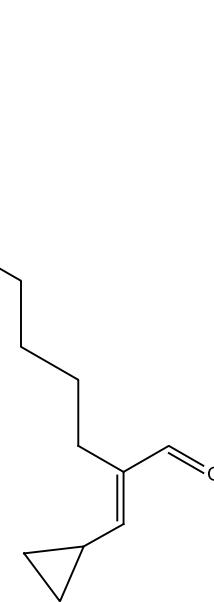
— 194.23

— 160.31 —

— 142.07

31.89
29.65
29.58
29.50
29.31
28.91
24.11
22.68

s80



(E)-2-(cyclopropylmethylene)undecanal (37)

