$Me_2(CH_2{=}CH)SiCN$: a bifunctional ethylene equivalent for Diels-Alder reaction based controllable tandem synthesis

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Supporting Information

1. General information

Reactions were monitored by thin layer chromatography (TLC) using UV light or $KMnO_4$ to visualize the course of reaction. Purification of reaction products was carried out by flash chromatography on silica gel. Chemical yields refer to pure isolated substances. Infrared (IR) spectra were obtained using a Bruker tensor 27 infrared spectrometer. The HRMS spectra were measured on Waters GCT Premier™, JEOL-AccuTOF-GCv4G-GCT MS or Bruker maXis impact spectrometer using electron spray ionization (ESI) method. ^{1}H, ^{13}C, and ^{19}F NMR spectra were obtained using Bruker DPX-300, DPX-400 or DPX-500 MHz spectrometer. Chemical shifts are reported in ppm from $CDCl_3$ or CD_2Cl_2 with the solvent resonance or $Si(CH_3)_4$ as the internal standard. The following abbreviations are used to designate chemical shift multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad.

Unless mentioned, all reactions were performed under an atmosphere of air. Anhydrous $ClCH_2CH_2Cl$, CH_2Cl_2 and CH_3CN was prepared by first distillation from P_2O_5 and then freshly distilled from CaH_2 prior to use. Anhydrous EtOAc was prepared by distillation over activated $CaSO_4$ and then freshly distilled from 5Å MS prior to use. Anhydrous toluene, THF and 1,4-dioxane were prepared by distillation over sodium-benzophenone ketyl prior to use. The α,β-unsaturated aldehydes **8u**[1], **8w**[2], **8x**[3] and **8aa**[4] were prepared according to the literature reports, and all other aldehydes were commercially available.

List of abbreviation:

Entry	Chemical name	Abbreviation
1	Petroleum ether	PE
2	Tetrahydrofuran	THF
3	*N*,*N*-Dimethylformamide	DMF
4	Tetrabutylammonium fluoride	TBAF
5	Butylated hydroxytoluene	BHT
6	2,3-Dichloro-5,6-dicyano-1,4-benzoquinone	DDQ

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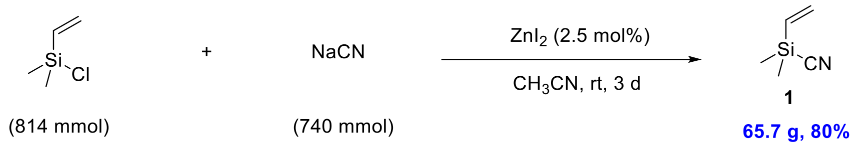
[1] J. R. Wolstenhulme, J. Rosenqvist, O. Lozano, J. Ilupeju, N. Wurz, K. M. Engle, G. W. Pidgeon, P. R. Moore, G. Sandford, V. Gouverneur, *Angew. Chem. Int. Ed*. **2013**, *52*, 9796-9800.

[2] S. Dey, D. Mal, *Tetrahedron Lett*. **2005**, *46*, 5483-5486.

[3] P. Panda, S. Nayak, S. K. Sahoo, S. Mohapatra, D. Nayak, R. Pradhan, C. N. Kundu, *RSC Adv*. **2018**, *8*, 16802-16814.

[4] B. S. Donslund, A. Monleln, T. A. Palazzo, M. L. Christensen, A. Dahlgaard, J. D. Erickson, K. A. Jørgensen, *Angew. Chem. Int. Ed*. **2018**, *57*, 1246-1250.

2. The synthesis of $Me_2(CH_2{=}CH)SiCN$



The newly designed bifunctional ethylene equivalent reagent $Me_2(CH_2{=}CH)SiCN$ was prepared according to our previous procedure for the synthesis of $Me_2(CH_2Cl)SiCN$.[5] To a 250 mL three-necked flask was added fine ground NaCN (740 mmol, 36.2 g), which was dried under vacuum at 125 °C for 12 h. After cooling down to room temperature, anhydrous CH_3CN (80 mL) was added, followed by the sequential addition of freshly distilled $Me_2(CH_2{=}CH)SiCl$ (814 mmol, 112 mL) and ZnI_2 (18.5 mmol, 5.9 g) under N_2 atmosphere. The resulting mixture was stirred at room temperature for about three days. After removing the solvent CH_3CN, the crude product was purified by distillation under reduced pressure to afford the analytically pure $Me_2(CH_2{=}CH)SiCN$ **1** as a colorless liquid (80%, 65.7 g). Boiling point: 133-135 °C. ^1H NMR (400 MHz, $CDCl_3$): δ 6.21 (ABd, J = 14.0, 3.6 Hz, 1H), 6.15-6.06 (m, 1H), 5.98 (ABd, J = 20.0, 3.6 Hz, 1H), 0.43 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.2, 131.1, 126.0, -3.2; MS (EI): 111 (M^+), 85; HRMS (EI): Exact mass calcd for C_5H_9NSi $[M]^+$: 111.0499, found: 111.0501.

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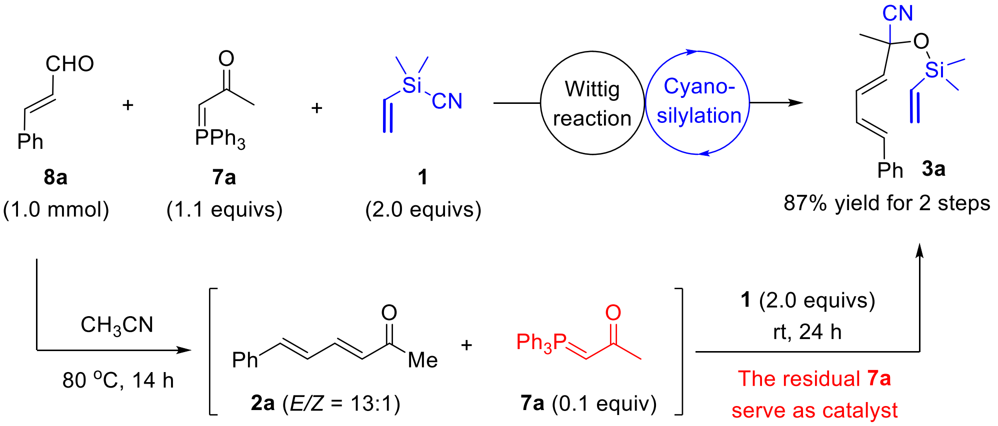
[5] X.-P. Zeng, J. Zhou, *J. Am. Chem. Soc.* **2016**, *138*, 8730-8733.

3. Initial attempt and condition optimizations

3.1 The initial attempt of the tandem Wittig-cyanosilylation reaction

With this novel bifunctional reagent $Me_2(CH_2{=}CH)SiCN$ **1** in hand, we first examined its performance in the cyanosilylation of conjugated dienone **2a** using our previously developed *P*-ylide catalytic system.[6] To our delight, the desired silylated cyanohydrin **3a**, bearing both a diene and a vinyl moiety, could be obtained in 85% yield, under the catalysis of acetone derived *P*-ylide **7a** (10 mol%) (Scheme 2b of the main text). Furthermore, it was found that the tandem Wittig/cyanosilylation sequence starting from enal **8a**, ylide **7a** and reagent **1** also proceeded well, and provided the targeted product **3a** in 87% yield, wherein the residual ylide **7a** (0.1 equiv) from the Wittig step played a role in enabling the downstream cyanosilylation step, as depicted in Scheme S1.



Scheme S1. The initial attempt of the tandem Wittig-cyanosilylation reaction.

3.2 The studies of the intramolecular Diels-Alder reaction

After successfully obtaining the silicon-tethered triene **3a**, we explored its intramolecular Diels-Alder (DA) reaction using toluene as the solvent. We were delighted to find that full conversion of **3a** was observed at 120 °C, and the NMR yield was 95%. However, the targeted product **4a** was obtained in only 15% isolated yield as a single diastereomer (Table S1, entry 1). Further evaluation of both reaction temperature and additive effects failed to improve its isolated yield (entries 2-7). To understand the actual reason, we conducted the ^{1}H NMR and GC-MS analysis, as shown in Figure S1-S2. First, full consumption of starting material **3a** by TLC, ^{1}H NMR and GC-MS analysis excluded the possibility of low conversion. Second, four diastereomers of the desired product could be

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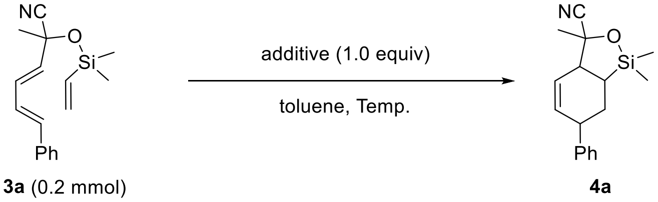
[6] W.-B. Wu, X.-P. Zeng, J. Zhou, *J. Org. Chem*. **2020**, *85*, 14342−14350.

observed in both ^{1}H NMR (Figure S1B) and GC-MS spectra (Figure S2B). Moreover, the ^{1}H NMR and GC-MS spectra of the sole one isomer **4a** obtained by column chromatography purification were also accord with one of four isomer, as compared in Figure S1A vs S1B and Figure S2A vs S2B. Based on these analysis, we concluded that the poor stability of other three isomers during silica gel column chromatography purification process might be responded to the low isolated yield. Therefore, we decided to develop a tandem DA reaction and *retro*-cyanosilylation sequence that will avoid the isolation of DA adducts and facilitate the removal of silyl group to provide the cyclohexene derivatives via tandem synthesis.

Table S1. Influence of temperature and additives in the intramolecular Diels-Alder reaction.



Entry	Temp. (°C)	t (h)	Additive	Isolated yield (%)
1[a]	120	11.0	-	15
2	150	10.0	-	16
3	185	8.0	-	11
4	120	11.0	BHT	16
5	120	11.0	Hydroquinone	15
6	120	0.5	Et_2AlCl	mess
7	120	0.5	$BF_3\cdot Et_2O$	mess

[a] 95% NMR yield, using 1,3,5-trimethoxybenzene as internal standard.

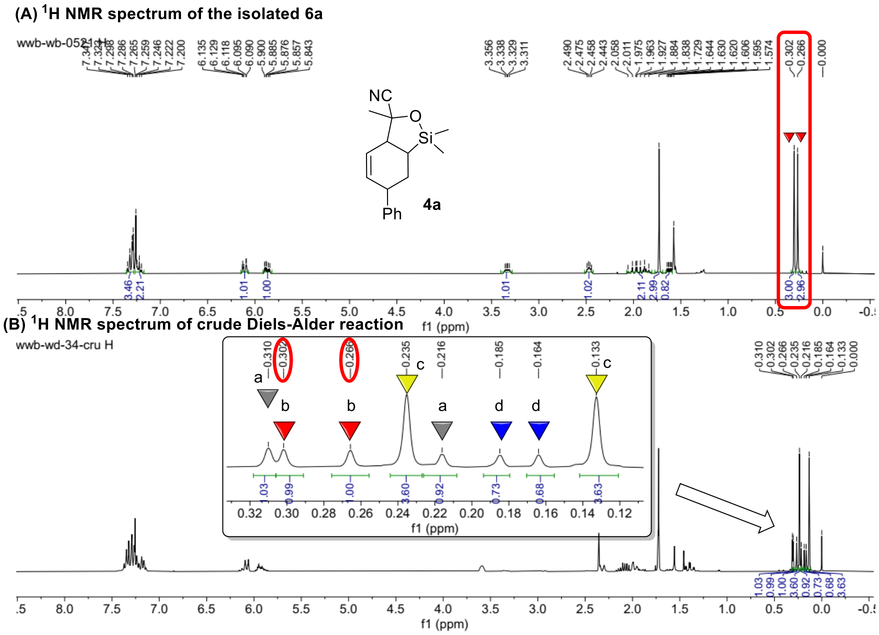
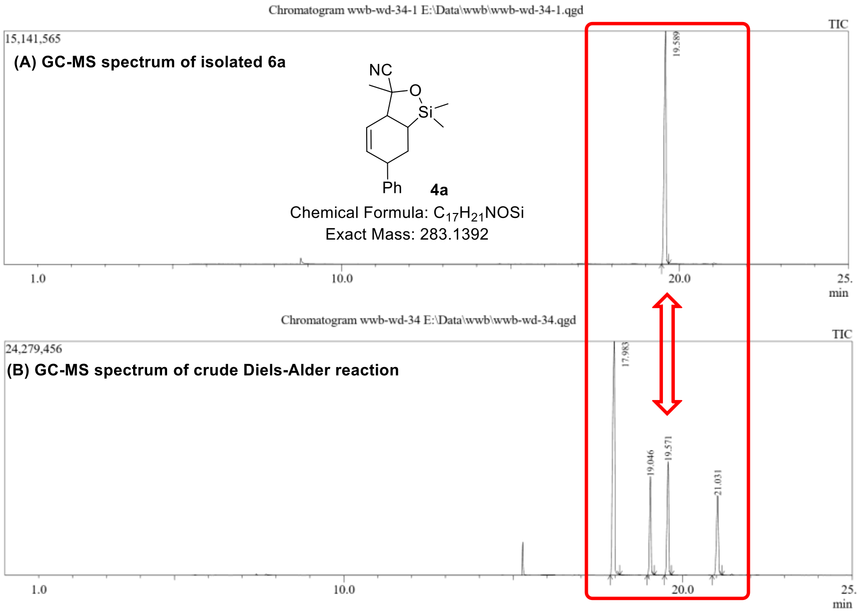


Figure S1. ^{1}H NMR analysis of Diels-Alder reaction mixture and the isolated product **4a**.



Four new peaks with a molecular weight of 283 observed.

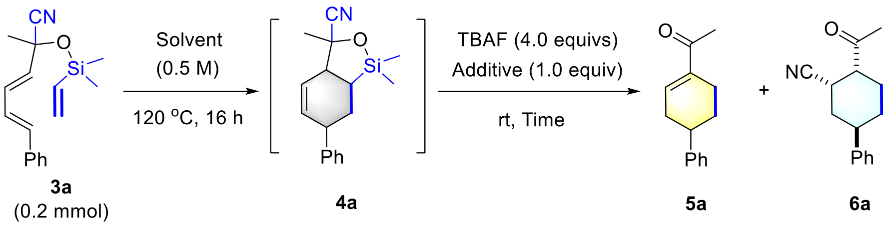
Figure S2. GC-MS analysis of Diels-Alder reaction mixture and the isolated product **4a**.

3.3 Condition optimization for the controllable synthesis of cyclohexene derivatives

With this idea in mind, we explored the desilylative condition after the DA reaction of **3a** reached completion. Upon treated the crude reaction mixture with a solution of TBAF in THF (1.0 M), and found that such sequence produced two products—cyclohexenyl methyl ketone **5a** with 11% yield and

2-acetyl-5-phenylcyclohexancarbonitrile **6a** with 52% yield and 1.2:1 dr (Table S2, entry 1). Encouraged by this result, further optimization was conducted to realize the controllable synthesis of **5a** and **6a** given that both are useful synthons. First, a series of metal additives, including $CuCl_2·2H_2O$, $FeCl_2$, AgOAc, $NiCl_2·6H_2O$, $Zn(OAc)_2·4H_2O$, and $Mn(OAc)_2·4H_2O$, were investigated to improve the yield of **5a** via capturing the resulting cyano anion (entries 2-7). It was found that all of them could inhibit the formation of **6a**, and $Mn(OAc)_2·4H_2O$ turned out to be optimal, affording **5a** in 73% yield (entry 7). Further investigation of solvent effect revealed that the use of EtOAc slightly improved the yield and dr value of **6a** (entry 11 vs entries 8-10). By using TBAF (70 wt.% in H_2O) instead of its THF solution, the dr value of **6a** could be greatly improved to >20:1, accompanied with a slightly increased 64% yield (entry 12).

Table S2. Selected condition optimization[a]



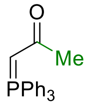
Entry	Solvent	Additive	Time (h)	Yield of **5a** (%)	Yield of **6a** (%)	dr of **6a**[b]
1	toluene	-	4	11	52	1.2:1
2	toluene	$CuCl_2·2H_2O$	4	35	<5	-
3	toluene	$FeCl_2$	4	34	<5	-
4	toluene	AgOAc	4	12	<5	-
5	toluene	$NiCl_2·6H_2O$	4	62	<5	-
6	toluene	$Zn(OAc)_2·2H_2O$	4	60	<5	-
7	**toluene**	**$Mn(OAc)_2·4H_2O$**	**4**	**73**	**<5**	-
8	DCE	-	12	58	29	>20:1
9	MeCN	-	12	<5	58	2.6:1
10	dioxane	-	12	<5	50	1.1:1
11	EtOAc	-	12	<5	57	4.0:1
12[c]	**EtOAc**	**-**	**24**	**13**	**64**	**>20:1**

[a] Reaction conditions: **3a** (0.2 mmol), TBAF (0.8 mL, 1.0 M in THF), additive (0.2 mmol), solvent (0.4 mL). The yield was determined by ^1H NMR analysis using 1,3,5-trimethoxybenzene as internal standard. [b] Determined by ^1H NMR analysis of the crude reaction mixture. [c] TBAF (70 wt.% in H_2O) was used instead of TBAF (1.0 M in THF).

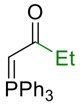
4. The correlation of serial number of α,β-unsaturated aldehydes & *P*-ylides, and their structure

Because the space limitation of the manuscript, we showed the structure of each α,β-unsaturated aldehydes and *P*-ylides, and its serial number in this section.

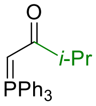
P-ylides **7**



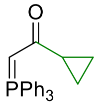
7a



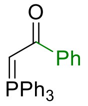
7b



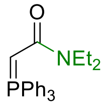
7c



7d

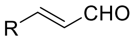


7e



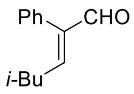
7f

α,β-Unsaturated aldehydes **8**

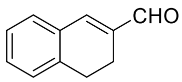


8a-o

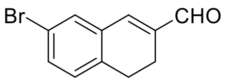
8a, R = C_6H_5
8b, R = 4-MeC_6H_4
8c, R = 4-$MeOC_6H_4$
8d, R = 2-$MeOC_6H_4$
8e, R = 4-ClC_6H_4
8f, R = 4-BrC_6H_4
8g, R = 4-FC_6H_4
8h, R = 3-FC_6H_4
8i, R = 2-FC_6H_4
8j, R = 2-$NO_2C_6H_4$
8k, R = 2-furyl
8l, R = Et
8m, R = *n*-Pr
8n, R = *n*-Bu
8o, R = *n*-C_5H_{11}



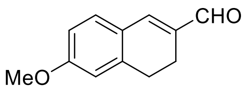
8t



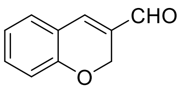
8u



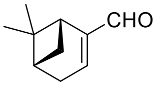
8v



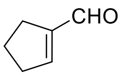
8w



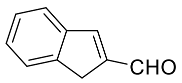
8x



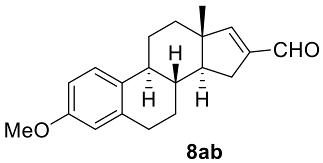
8y



8z

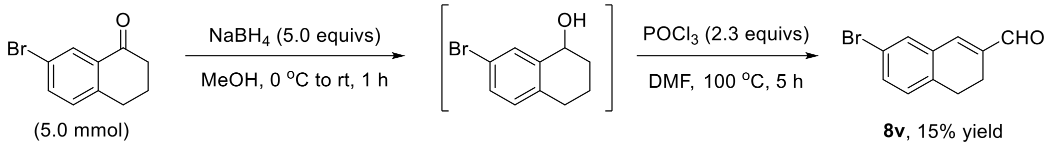


8aa



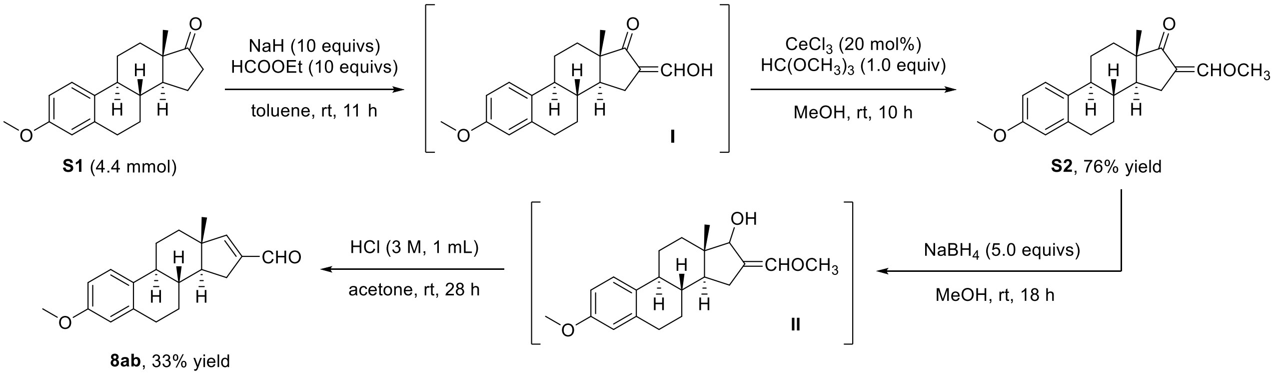
8ab

5. The synthesis of unknown α,β-unsaturated aldehydes and ketones



To a solution of 7-bromo-3,4-dihydro-2*H*-naphthalen-1-one (1.250 g, 5.0 mmol) in methanol (10 mL) was added $NaBH_4$ (0.950 g, 25.0 mmol) at 0 °C. The reaction mixture was then stirred at room temperature for 1 hour before quenching with 1M HCl. The aqueous layer was extracted with EtOAc (20 mL × 3) and the combined organic layers were washed with brine, dried over Na_2SO_4, and evaporated under vacuo to give the crude 7-bromo-1,2,3,4-tetrahydronaphthalen-1-ol as a pale yellow oil, which was used directly in the next step.

To a solution of above crude 7-bromo-1,2,3,4-tetrahydronaphthalen-1-ol in DMF (5 mL) was added $POCl_3$ (1.0 mL, 11.5 mmol) slowly at 0 °C. The resulting reaction solution was then heated at 100 °C for 5 hour. After cooling to room temperature, the reaction was quenched with water and adjusted the pH to around 7.0 with 3M NaOH aqueous solution. The aqueous phase was extracted with EtOAc (20 mL × 3). The combined organic phases were washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash column chromatography using PE/EtOAc (5/1, v/v) as the eluent to afford the desired α,β-unsaturated aldehydes **8v** as yellow oil in 15% yield (176.3 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.67 (s, 1H), 7.42-7.40 (m, 2H), 7.20-7.19 (m, 1H), 7.10-7.08 (m, 1H), 2.82 (t, *J* = 12.0 Hz, 2H), 2.59-2.54 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 192.7, 144.0, 140.4, 137.0, 134.2, 133.2, 131.4, 129.7, 120.4, 26.7, 19.3. IR (neat): ν 1672.3, 1552.7, 1301.9, 1193.9, 1114.9, 1074.4, 904.6, 829.4 cm^{-1}; MS (EI): 238 [M(^{81}Br)$^+$, 33], 236 [M(^{79}Br)$^+$, 35], 207 (7), 157 (26), 128 (100), 102 (9), 77 (11), 63 (10); HRMS (EI): Exact mass calcd for $C_{11}H_9O^{79}Br$ [M]$^+$: 235.9837, Found: 235.9835.

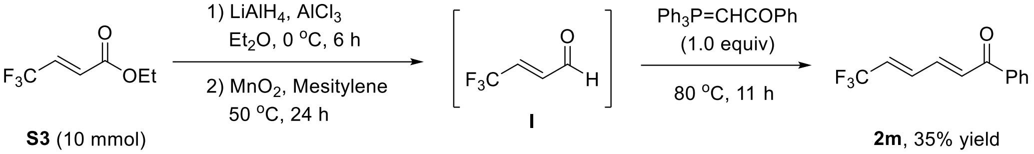


NaH (1.760 g, 60% in mineral oil, 44 mmol) was added to a 100 mL flame-dried flask under N_2 atmosphere, which was washed with anhydrous *n*-hexane (10 mL × 3). Then anhydrous toluene (10 mL) was added, followed by the addition of estrone 3-methyl ether **S1** (1.240 g, 4.4 mmol) and ethyl formate (3.5 mL, 44 mmol). The suspension was stirred at room temperature (rt) for 11 h until full conversion of **S1** by TLC analysis. The reaction mixture was poured in to 2M HCl (10 mL), and then extracted with EtOAc (20 mL × 3). The combined organic phases were washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure to give the intermediate **I**.

Under N_2 atmosphere, to a solution of $CeCl_3$ (0.217 g, 0.88 mmol) in anhydrous MeOH (10 mL) was successively added all the above intermediate **I** (ca. 4.4 mmol) and trimethoxymethane (0.467 g, 4.4 mmol) at rt. After being stirred overnight at rt, H_2O (5 mL) was added to the resulting solution, and exacted with $CHCl_3$ (20 mL × 3). The combined organic phases were washed with brine, and dried over Na_2SO_4. After evaporation, the crude residue was purified by flash column chromatography using PE/EtOAc (4:1, v/v) as the eluent to afford **S2** in 76% yield (1.084 g) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.21-7.19 (m, 2H), 6.73-6.70 (m, 1H), 6.64-6.63 (m, 1H), 3.85 (s, 3H), 3.78 (s, 3H), 2.91-2.88 (m, 2H), 2.70-2.64 (m, 1H), 2.40-2.37 (m, 1H), 2.28-2.24 (m, 1H), 2.11-2.07 (m, 1H), 2.00-1.96 (m, 2H), 1.60-1.46 (m, 5H), 0.92 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 210.3, 157.7, 154.6, 137.9, 132.4, 126.4, 115.5, 114.0, 111.7, 61.6, 55.3, 48.9, 48.8, 44.2, 38.0, 31.7, 29.8, 26.9, 26.2, 24.7, 14.8. HRMS (ESI): Exact mass calcd for $C_{21}H_{26}NaO_3$ $[M+Na]^+$: 349.1774; Found: 349.1777.

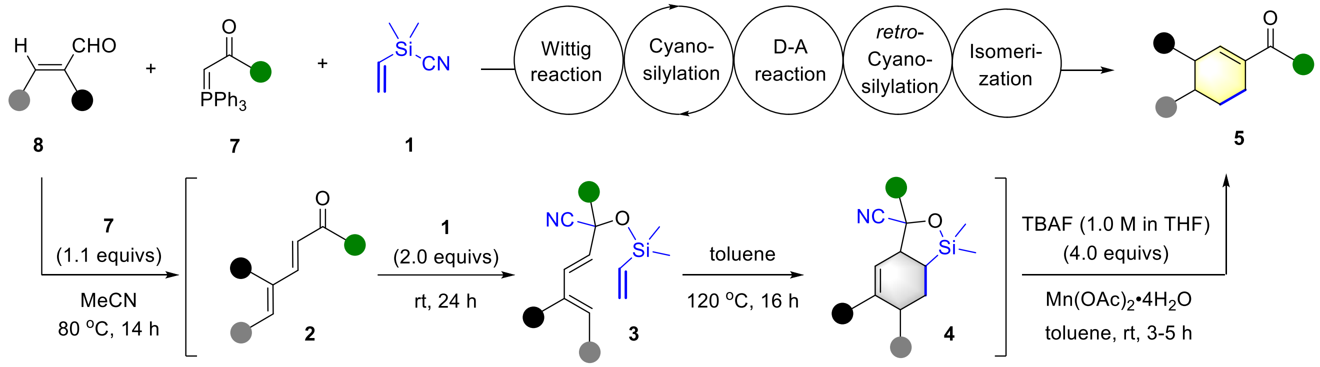
To a solution of **S2** (1.000 g, 3.3 mmol) in methanol (5 mL) was added $NaBH_4$ (0.623 g, 16.5 mmol). The reaction mixture was stirred at rt for 30 minutes before quenching with 1M HCl. The aqueous layer was extracted with EtOAc (20 mL × 3) and the combined organic layers were washed with brine, dried over Na_2SO_4, and concentrated. The crude product was dried under vacuo to give the intermediate **II**, which was dissolved in acetone (5.0 mL). To this solution was added 3M HCl (10 mL), the reaction mixture was stirred at rt for 28 h, and extracted with EtOAc (20 mL × 3). The combined organic phases were washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure to give the residue, which was purified by flash column chromatography using PE/EtOAc (4/1, v/v) as the eluent to give the product **8ab** as white solid in 33% yield (0.321 g). ^{1}H NMR (400 MHz, $CDCl_3$): 9.73 (s, 1H), 7.20-7.17 (s, 1H), 6.98 (d, J = 2.0 Hz, 1H), 6.73-6.70 (m, 1H), 6.65 (d, J = 2.8 Hz, 1H), 3.78 (s, 3H), 2.93-2.88 (m, 2H), 2.62-2.57 (m, 1H), 2.40-2.28 (m, 2H), 2.18-2.11 (m, 1H), 1.99-1.93 (m, 2H), 1.76-1.69 (m, 2H), 1.64-1.59 (m, 2H), 1.51-1.40 (m, 1H), 0.91 (s, 3H); ^{13}C

NMR (125 MHz, $CDCl_3$): δ 190.8, 163.6, 157.7, 146.3, 138.0, 132.5, 126.1, 114.0, 111.7, 55.3, 54.7, 47.7, 44.3, 37.5, 34.9, 29.8, 28.3, 27.8, 26.4, 16.3; HRMS (ESI): Exact mass calcd for $C_{20}H_{24}O_2Na$ $[M+Na]^+$: 319.1669, Found: 319.1670.

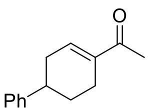


Under N_2 atmosphere, to a solution of $LiAlH_4$ (0.570 g, 15 mmol) and $AlCl_3$ (1.000 g, 0.75 mmol) in Et_2O (10 mL) was added ethyl 4,4,4-trifluorocrotonate **S3** (1.680 g, 10 mmol) at 0 °C. The suspension was stirred at 0 °C for 6 h until full conversion of **S3** by TLC analysis. The reaction mixture was poured in to saturated $NaHCO_3$ (aq., 10 mL), and aqueous solution of HCl (4 N) was added slowly until the pH was 7. The resulting mixture was extracted with Et_2O (20 mL × 3), and the combined organic phases were washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The obtained crude alcohol was dissolved in mesitylene (10 mL), followed by the addition of MnO_2 (6.950 g, 80 mmol). The reaction mixture was stirred at 50 °C for 24 h until full conversion of alcohol by TLC analysis. After filtrating through Celite, to the filtrate was added $Ph_3P{=}CHCOPh$ (3.800 g, 10 mmol), and the suspension was stirred at 80 °C until full conversion of aldehyde **I** by TLC analysis (ca. 11 h). The crude residue was purified by flash column chromatography using PE/EtOAc (20:1, v/v) as the eluent to afford **2m** in 35% yield (0.160 g) as yellow solid. ^1H NMR (400 MHz, $CDCl_3$): δ 7.97-7.95 (m, 2H), 7.63-7.59 (m, 1H), 7.53-7.49 (m, 2H), 7.40-7.33 (m, 1H), 7.25-7.21 (m, 1H), 7.01-6.94 (m, 1H), 6.19-6.10 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.7, 139.3, 137.4, 135.3 (q, J = 6 Hz), 133.5, 131.6, 129.0, 128.7, 126.3 (q, J = 34 Hz), 122.7 (q, J = 268 Hz). ^{19}F NMR (376 MHz, $CDCl_3$): δ -64.408; IR (neat): ν 1668.4, 1643.4, 1448.5, 1172.7, 1095.6, 995.3, 769.6, 984.7 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{12}H_9F_3NaO$ $[M+Na]^+$: 249.0498; Found: 249.0501.

6. General procedure for the tandem synthesis of cyclohexenyl ketones 5

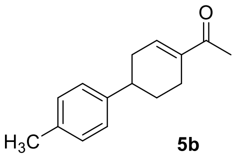


Under N_2 atmosphere, to a 10 mL sealed tube was added α,β-unsaturated aldehydes **8** (1.0 mmol) and *P*-ylide **7** (1.1 mmol), followed by the addition of anhydrous acetonitrile (1.0 mL). The resulting mixture was heated to 80 °C until full conversion of **8** by TLC analysis (*ca.* 14 h). After cooling to rt, $Me_2(CH{=}CH_2)SiCN$ **1** (250 µL, 2.0 mmol) was added. After being stirred at the same temperature for 24 h, the reaction mixture was rapidly passed through a short pad of silica gel, and washed with ether. The organic solution was concentrated in vacuo to give the crude silicon tethered triene **3**, which was dissolved in toluene (2.0 mL). The reaction solution was heated to 120 °C for 16 h. After full conversion of **3** by TLC analysis, the reaction mixture was cooled to rt, and $Mn(OAc)_2{\cdot}4H_2O$ (0.245 g, 1.0 mmol), TBAF (4.0 mL, 1.0 M in THF) was added. The suspension was stirred at rt until full conversion of **4** by TLC analysis (*ca.* 3~5 h), which was then poured in to H_2O (10 mL), and extracted with EtOAc (20 mL × 3). The combined organic phases were washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash column chromatography using PE/EtOAc (4/1, v/v) as the eluent to give the cyclohexenyl ketones **5**. (Note: *P*-ylide $Ph_3P{=}CHCONEt_2$ **7f** (3.8 mg, 0.01 mmol) was added as an extra catalyst in the cyanosilylation step in the cases of **5q-s**.)



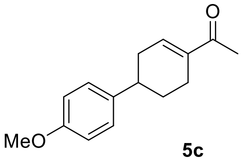
5a

Product **5a** was obtained in 65% yield (130.0 mg) as colorless oil. ^1H NMR (400 MHz, $CDCl_3$): δ 7.34-7.30 (m, 2H), 7.24-7.20 (m, 3H), 6.99-6.97 (m, 1H), 2.82-2.75 (m, 1H), 2.62-2.53 (m, 2H), 2.43-2.35 (m, 1H), 2.33 (s, 3H), 2.30-2.20 (m, 1H), 2.08-2.02 (m, 1H), 1.76-1.66 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.2, 146.1, 140.2, 139.6, 128.7, 126.9, 126.5, 39.4, 34.3, 29.4, 25.5, 23.9; IR (neat): ν 1786.1, 1658.8, 1639.5, 1386.8, 1249.9, 960.6, 767.7, 704.0 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{14}H_{16}NaO$ $[M+Na]^+$: 223.1093; Found: 223.1083.



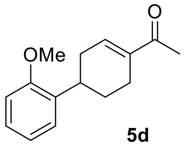
5b

Product **5b** was obtained in 51% yield (109.1 mg) as white solid. Mp: 51-53 °C. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.15-7.10 (m, 4H), 6.98-6.97 (m, 1H), 2.78-2.71 (m, 1H), 2.59-2.53 (m, 2H), 2.41-2.36 (m, 1H), 2.33 (s, 3H), 2.32 (s, 3H), 2.28-2.23 (m, 1H), 2.05-1.99 (m, 1H), 1.72-1.63 (m, 1H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 199.2, 143.1, 140.4, 139.6, 136.0, 129.3, 126.8, 39.0, 34.4, 29.5, 25.5, 23.9, 21.1; IR (neat): ν 1666.5, 1386.8, 1249.9, 1070.5, 958.6, 623.0, 599.9, 561.3 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{15}H_{18}NaO$ [M+Na]$^+$: 237.1250; Found: 237.1254.



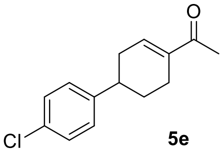
5c

Product **5c** was obtained in 63% yield (144.5 mg) as white solid. Mp: 45-47 °C. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.15-7.13 (m, 2H), 6.97 (t, J = 1.6 Hz, 1H), 6.87-6.85 (m, 2H), 3.80 (s, 3H), 2.78-2.70 (m, 1H), 2.59-2.52 (m, 2H), 2.38-2.23 (m, 5H), 2.04-1.99 (m, 1H), 1.70-1.63 (m, 1H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 199.2, 158.2, 140.3, 139.6, 138.2, 127.8, 114.1, 55.4, 38.5, 34.5, 29.6, 25.5, 23.9; IR (neat): ν 1710.9, 1653.0, 1508.3, 1244.1, 1035.8, 958.6, 621.1, 599.9 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{15}H_{18}NaO_2$ [M+Na]$^+$: 253.1199; Found: 253.1206.



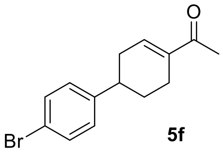
5d

Product **5d** was obtained in 80% yield (184.3 mg) as colorless oil. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.23-7.15 (m, 2H), 7.00-6.87 (m, 3H), 3.83 (s, 3H), 3.23-3.17 (m, 1H), 2.64-2.53 (m, 2H), 2.33 (s, 3H), 2.30-2.21 (m, 2H), 1.98-1.94 (m, 1H), 1.80-1.71 (m, 1H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 199.3, 157.1, 141.0, 139.5, 134.2, 127.3, 126.6, 120.8, 110.5, 55.4, 33.0, 32.4, 27.8, 25.5, 24.0; IR (neat): ν 1784.2, 1664.6, 1492.9, 1384.9, 1028.1, 960.6, 752.2, 594.1 cm^{-1}; HRMS (ESI):Exact mass calcd for $C_{15}H_{18}NaO_2$ [M+Na]$^+$: 253.1199; Found: 253.1215.



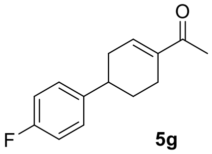
5e

Product **5e** was obtained in 52% yield (121.6 mg) as white solid. Mp: 66-68 °C. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.30-7.27 (m, 2H), 7.16-7.14 (m, 2H), 6.97-6.96 (m, 1H), 2.79-2.73 (m, 1H), 2.59-2.53 (m, 2H), 2.37-2.35 (m, 1H), 2.33 (s, 3H), 2.29-2.23 (m, 1H), 2.03-1.99 (m, 1H), 1.72-1.62 (m, 1H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 199.1, 144.5, 139.8, 139.6, 132.1, 128.8, 128.3, 38.9, 34.2, 29.3, 25.5, 23.7; IR (neat): ν 1710.9, 1658.8, 1354.0, 1249.9, 1089.8, 960.6, 812.0, 702.1 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{14}H_{15}ClNaO$ [M+Na]$^+$: 257.0704; Found: 257.0706.



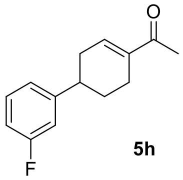
5f

Product **5f** was obtained in 53% yield (148.3 mg) as white solid. Mp: 104-106 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45-7.43 (m, 2H), 7.11-7.09 (m 2H), 6.96-6.95 (m, 1H), 2.79-2.72 (m, 1H), 2.59-2.52 (m, 2H), 2.38-2.31 (m, 4H), 2.29-2.21 (m, 1H), 2.04-1.99 (m, 1H), 1.72-1.65 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.1, 145.0, 139.8, 139.6, 131.7, 128.7, 120.2, 38.9, 34.1, 29.2, 25.5, 23.7; IR (neat): ν 1714.7, 1384.9, 1352.1, 1249.9, 1070.5, 960.6, 810.1, 599.9 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{14}H_{15}BrNaO$ [M+Na]$^+$: 301.0198; Found: 301.0200.



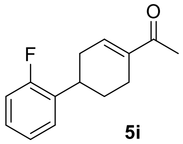
5g

Product **5g** was obtained in 60% yield (131.0 mg) as white solid. Mp: 56-58 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.19-7.16 (m, 2H), 7.02-6.98 (m, 2H), 6.97-6.96 (m, 1H), 2.80-2.74 (m, 1H), 2.59-2.53 (m, 2H), 2.38-2.32 (m, 4H), 2.29-2.24 (m, 1H), 2.04-2.00 (m, 1H), 1.73-1.64 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.1, 161.6 (d, *J* = 242 Hz), 141.7 (d, *J* = 3 Hz), 139.9, 139.6, 128.3 (d, *J* = 7 Hz), 115.4 (d, *J* = 21 Hz), 38.7, 34.4, 29.5, 25.5, 23.8; ^{19}F NMR (376 MHz, $CDCl_3$): δ -116.943; IR (neat): ν 1712.8, 1641.4, 1508.3, 1386.8, 1354.0, 833.3, 623.0, 597.9 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{14}H_{15}FNaO$ [M+Na]$^+$: 241.0999; Found: 241.1000.



5h

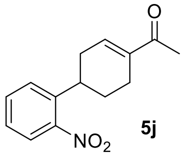
Product **5h** was obtained in 39% yield (81.5 mg) as white solid. Mp: 53-55 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31-7.25 (m, 1H), 7.01-6.99 (m, 1H), 6.97-6.56 (m, 1H), 6.94-6.90 (m, 2H), 2.84-2.76 (m, 1H), 2.59-2.54 (m, 2H), 2.40-2.37 (m, 1H), 2.33 (s, 3H), 2.29-2.24 (m, 1H), 2.07-2.02 (m, 1H), 1.74-1.65 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.0, 163.2 (d, *J* = 244 Hz), 148.7 (d, *J* = 7 Hz), 139.7, 139.6, 130.1 (d, *J* = 9 Hz), 122.6 (d, *J* = 2 Hz), 113.7 (d, *J* = 21 Hz), 113.3 (d, *J* = 20 Hz), 39.2, 34.0, 29.2, 25.5, 23.7; ^{19}F NMR (376 MHz, $CDCl_3$): δ -113.258; IR (neat): ν 1784.2, 1662.6, 1485.2, 1384.9, 1249.8, 1070.5, 702.1, 597.9 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{14}H_{15}FNaO_2$ [M+Na]$^+$: 241.0999; Found: 241.1012.



5i

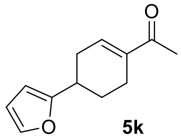
Product **5i** was obtained in 45% yield (98.0 mg) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23-7.18 (m, 2H), 7.12-7.08 (m, 1H), 7.05-7.01 (m, 1H), 6.98-6.97 (m, 1H), 3.17-3.09 (m, 1H), 2.63-2.55 (m, 2H), 2.41-2.36 (m, 1H), 2.33 (s, 3H), 2.30-2.25 (m, 1H), 2.03-1.99 (m, 1H), 1.83-1.76 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.1, 160.9 (d, *J* = 243 Hz), 144.0, 139.6, 132.6 (d, *J* = 15 Hz), 127.9 (d, *J* = 8 Hz), 127.7 (d, *J* = 5 Hz), 124.4 (d, *J* = 3 Hz), 115.6 (d, *J* = 23 Hz), 32.8, 32.6 (d, *J* = 2 Hz), 27.9, 25.5, 23.7; ^{19}F NMR (376

MHz, $CDCl_3$): δ -118.974; IR (neat): ν 1666.5, 1489.1, 1384.9, 1352.1, 1070.5, 960.6, 754.2, 638.4 cm^{-1};HRMS (ESI):Exact mass calcd for $C_{14}H_{15}FNaO_2$ $[M+Na]^+$: 241.0999; Found: 241.1004.



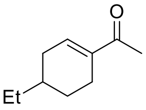
5j

Product **5j** was obtained in 48% yield (117.7 mg) as white solid. Mp: 95-97 °C. ^1H NMR (400 MHz, $CDCl_3$): δ 7.77-7.75 (m, 1H), 7.60-7.56 (m, 1H), 7.45-7.43 (m, 1H), 7.39-7.35 (m, 1H), 6.97-6.95 (m, 1H), 3.31-3.24 (m, 1H), 2.77-2.70 (m, 1H), 2.65-2.59 (m, 1H), 2.39-2.22 (m, 5H), 2.05-2.01 (m, 1H), 1.84-1.77 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.8, 150.2, 140.0, 139.5, 139.4, 132.9, 128.0, 127.3, 124.2, 34.3, 34.0, 28.8, 25.5, 23.8; IR (neat): ν 1710.9, 1431.2, 1217.1, 995.3, 850.6, 817.8, 684.7, 636.5 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{14}H_{15}NNaO_3$ $[M+Na]^+$: 268.0944; Found: 268.0957.



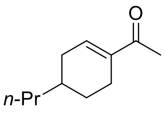
5k

Product **5k** was obtained in 77% yield (145.8 mg) as colorless oil. ^1H NMR (400 MHz, $CDCl_3$): δ 7.33 (d, *J* = 1.2 Hz, 1H), 6.93-6.92 (m, 1H), 6.31-6.29 (m, 1H), 6.02 (d, *J* = 1.6 Hz, 1H), 2.94-2.88 (m, 1H), 2.70-2.62 (m, 1H), 2.52-2.37 (m, 2H), 2.31 (s, 3H), 2.28-2.12 (m, 2H), 1.71-1.61 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.1, 159.0, 141.2, 139.6, 139.1, 110.1, 103.8, 32.6, 31.0, 27.0, 25.4, 22.8; IR (neat): ν 1585.5, 1384.9, 1303.9, 1166.9, 962.5, 883.4, 804.3, 752.2 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{12}H_{14}NaO_2$ $[M+Na]^+$: 213.0886; Found: 213.0889.



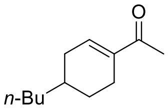
5l

Product **5l** was obtained in 30% yield (45.9 mg) as colorless oil. ^1H NMR (400 MHz, $CDCl_3$): δ 6.88 (s, 1H), 2.46-2.35 (m, 2H), 2.28 (s, 3H), 2.12-2.06 (m, 1H), 1.91-1.82 (m, 2H), 1.47-1.40 (m, 1H), 1.36-1.29 (m, 2H), 1.21-1.14 (m, 1H), 0.93 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.4, 140.7, 139.8, 34.6, 32.6, 28.9, 28.0, 25.4, 23.2, 11.5; IR (neat): ν 1714.7, 1641.4, 1386.8, 1352.1, 1246.0, 1070.5, 827.5, 632.7 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{10}H_{16}NaO$ $[M+Na]^+$: 175.1093; Found: 175.1088.



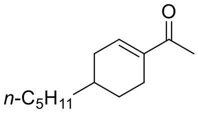
5m

Product **5m** was obtained in 43% yield (71.0 mg) as colorless oil. ^1H NMR (400 MHz, $CDCl_3$): δ 6.87 (s, 1H), 2.46-2.34 (m, 2H), 2.28 (s, 3H), 2.12-2.05 (m, 1H), 1.90-1.80 (m, 2H), 1.54-1.52 (m, 1H), 1.39-1.34 (m, 2H), 1.30-1.25 (m, 2H), 1.21-1.13 (m, 1H), 0.91 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.4, 140.7, 139.8, 38.5, 33.0, 32.5, 28.4, 25.4, 23.2, 20.1, 14.4; IR (neat): ν 1786.1, 1641.4, 1386.8, 1352.1, 1072.4, 760.0, 638.4, 560.0 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{11}H_{18}NaO$ $[M+Na]^+$: 189.1250; Found: 189.1242.



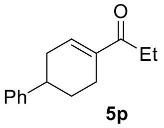
5n

Product **5n** was obtained in 40% yield (73.0 mg) as colorless oil.[7] ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.88-6.87 (m, 1H), 2.46-2.34 (m, 2H), 2.28 (s, 3H), 2.12-2.03 (m, 1H), 1.90-1.82 (m, 2H), 1.51 (s, br, 1H), 1.31-1.29 (m, 6H), 1.21-1.11 (m, 1H), 0.90 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.4, 140.7, 139.8, 36.0, 33.0, 32.8, 29.3, 28.4, 25.4, 23.2, 23.0, 14.2; IR (neat): ν 1784.2, 1641.4, 1352.1, 1147.7, 1070.5, 910.4, 760.0, 640.4 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{12}H_{20}NaO$ $[M+Na]^+$: 203.1406; Found: 203.1402.



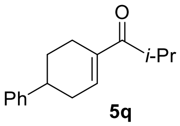
5o

Product **5o** was obtained in 40% yield (77.6 mg) as colorless oil.[7] ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.87 (t, *J* = 2.8 Hz, 1H), 2.45-2.34 (m, 2H), 2.28 (s, 1H), 2.13-2.04 (m, 1H), 1.90-1.81 (m, 2H), 1.51 (s, 1H), 1.32-1.26 (m, 8H), 1.21-1.11 (m, 1H), 0.89 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.4, 140.7, 139.8, 36.2, 33.0, 32.9, 32.2, 28.4, 26.7, 25.4, 23.2, 22.8, 14.2; IR (neat): ν 1668.4, 1386.8, 1247.7, 1070.5, 952.8, 906.5, 640.4, 600.0 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_{22}NaO$ $[M+Na]^+$: 217.1563; Found: 217.1556.



5p

Product **5p** was obtained in 65% yield (139.1 mg) as white solid. Mp: 43-45 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34-7.30 (m, 2H), 7.23-7.20 (m, 3H), 6.97-6.96 (m, 1H), 2.82-2.79 (m, 1H), 2.71 (q, *J* = 7.2 Hz, 2H), 2.60-2.54 (m, 2H), 2.41-2.26 (m, 2H), 2.06-2.02 (m, 1H), 1.77-1.66 (m, 1H), 1.12 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 201.9, 146.2, 139.0, 138.6, 128.7, 126.9, 126.5, 39.5, 34.2, 30.4, 29.5, 24.1, 8.8; IR (neat): ν 1780.3, 1712.8, 1496.8, 1220.9, 954.8, 896.9, 873.8, 702.1 cm^{-1}; MS (EI): 214 (M^+, 48), 185 (36), 157 (5), 129 (9), 123 (21), 104 (100), 91 (20), 81 (48); HRMS (EI): Exact mass calcd for $C_{15}H_{18}O$ $[M]^+$: 214.1358, Found: 214.1362.



5q

Product **5q** was obtained in 75% yield (171.6 mg) as white solid. Mp: 41-43 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34-7.30 (m, 2H), 7.24-7.20 (m, 3H), 6.98-6.97 (m, 1H), 3.35-3.28 (m, 1H), 2.84-2.76 (m, 1H), 2.60-2.54 (m, 2H), 2.42-2.37 (m, 1H), 2.30-2.22 (m, 1H), 2.07-2.02 (m, 1H), 1.78-1.67 (m, 1H), 1.12 (d, *J* = 1.2 Hz, 3H), 1.11 (d, *J* = 1.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 205.5, 146.2, 138.4, 138.0, 128.7, 126.9, 126.5, 39.5, 34.3, 33.9, 29.5, 24.4, 19.7; IR (neat): ν 1714.7, 1653.0, 1359.8, 1220.9, 1199.7, 964.4, 763.8, 700.2 cm^{-1}; MS (EI): 228 (M^+, 37), 185 (100), 157 (4), 141 (4), 129 (14), 115 (9), 104 (31), 91 (24); HRMS (EI):

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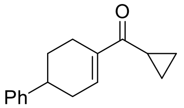
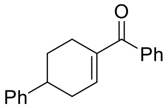
[7] A. I. Pavlyuchenko, G. V. Purvanetskas, V. K. Daukshas, N. I. Smirnova, N. I. Korotkova, E. I. Kovshev, *Chem. Heterocycl. Compd*. **1984**, *20*, 515-518.

Exact mass calcd for $C_{16}H_{20}O$ [M]$^+$: 228.1517, Found: 228.1514.



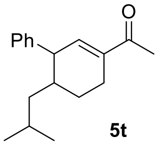
5r

Product **5r** was obtained in 54% yield (122.0 mg) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35-7.31 (m, 2H), 7.26-7.23 (m, 3H), 7.13-7.12 (m, 1H), 2.86-2.78 (m, 1H), 2.65-2.57 (m, 2H), 2.45-2.37 (m, 2H), 2.33-2.25 (m, 1H), 2.08-2.03 (m, 1H), 1.79-1.69 (m, 1H), 1.08-1.05 (m, 2H), 0.91-0.86 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.9, 146.2, 139.9, 138.6, 128.7, 126.9, 126.5, 39.5, 34.4, 29.5, 24.4, 15.8, 10.9, 10.7; IR (neat): ν 1668.4, 1641.4, 1492.9, 1352.1, 964.4, 910.4, 758.0, 702.1 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{16}H_{18}NaO$ [M+Na]$^+$: 249.1250; Found: 249.1246.



5s

Product **5s** was obtained in 50% yield (128.9 mg) as white solid.[8] Mp: 44-46 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68-7.66 (m, 2H), 7.53-7.49 (m, 1H), 7.45-7.41 (m, 2H), 7.35-7.31 (m, 2H), 7.26-7.21 (m, 3H), 6.66-6.65 (m, 1H), 2.92-2.85 (m, 1H), 2.76-2.70 (m, 1H), 2.62-2.54 (m, 1H), 2.51-2.36 (m, 2H), 2.15-2.10 (m, 1H), 1.89-1.78 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.0, 146.1, 143.1, 138.8, 138.7, 131.6, 129.3, 128.7, 128.2, 127.0, 126.5, 39.6, 34.3, 29.5, 24.9; IR (neat): ν 1784.2, 1599.0, 1577.8, 1311.6, 1213.2, 925.8, 758.8, 667.4 cm^{-1}; MS (EI): 262 (M$^+$, 41), 244 (1), 171 (11), 158 (10), 145 (11), 129 (4), 104 (100), 91 (10); HRMS (EI): Exact mass calcd for $C_{19}H_{18}O$ [M]$^+$: 262.1358, Found: 262.1362.



5t

Product **5t** was obtained in 32% yield (81.9 mg) as colorless oil. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 1.5:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35-7.31 (m, 2H), 7.27-7.23 (m, 1H), 7.16-7.14 (m, 2H), 6.75-6.73 (m, 1H), 3.17-3.14 (m, 1H), 2.52-2.45 (m, 1H), 2.27 (s, 3H), 2.25-2.20 (m, 1H), 1.99-1.94 (m, 1H), 1.68-1.59 (m, 2H), 1.28-1.24 (m, 1H), 1.13-1.09 (m, 2H), 0.81 (d, J = 6.8 Hz, 3H), 0.68 (d, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.7, 143.9, 143.4, 139.4, 128.71, 128.70, 126.8, 50.3, 43.1, 38.3, 26.4, 25.5, 25.2, 24.0, 22.8, 21.6; IR (neat): ν 1668.4, 1602.9, 1562.3, 1365.6, 960.6, 864.1, 700.2, 582.5 cm^{-1}. MS (EI): 256 (M$^+$, 95), 213 (18), 199 (51), 171 (17), 157 (49), 129 (100), 117 (12), 91 (39); HRMS (EI): Exact mass calcd for $C_{18}H_{24}O$ [M]$^+$: 256.1827, Found: 256.1823.

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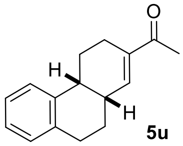
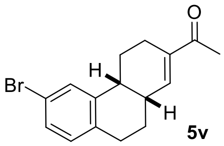
[8] F.-F. Pan, P. Guo, C.-L. Li, P. Su, X.-Z. Shu, *Org. Lett.* **2019**, *21*, 3701-3705.



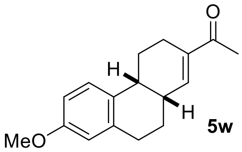
5u

Product **5u** was obtained in 40% yield (90.4 mg) as white solid. Mp: 68-70 °C. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 16:1. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.24-7.15 (m, 2H), 7.14-7.07 (m, 2H), 6.89-6.87 (m, 1H), 2.95-2.90 (m, 1H), 2.86-2.83 (m, 2H), 2.68-2.67 (m, 1H), 2.49-2.43 (m, 1H), 2.33 (s, 3H), 2.27-2.20 (m, 1H), 1.98-1.94 (m, 2H), 1.74-1.63 (m, 2H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 199.4, 143.8, 139.9, 139.4, 136.3, 129.4, 129.1, 126.2, 126.0, 36.9, 35.9, 29.7, 28.5, 25.8, 25.5, 23.7; IR (neat): ν 1683.9, 1658.8, 1500.6, 1352.1, 1286.5, 1037.7, 819.8, 611.4 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{16}H_{18}NaO$ [M+Na]$^+$: 249.1250; Found: 249.1244.



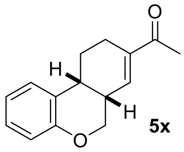
5v

Product **5v** was obtained in 50% yield (76.3 mg) as colorless oil. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 7:1. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.36 (d, *J* = 2.0 Hz, 1H), 7.22 (dd, *J* = 8.0, 2.0 Hz, 1H), 6.95 (d, *J* = 8.8 Hz, 1H), 6.86-6.84 (m, 1H), 2.92-2.86 (m, 1H), 2.78-2.74 (m, 2H), 2.65-2.63 (m, 1H), 2.48-2.42 (m, 1H), 2.32 (s, 3H), 2.27-2.19 (m, 1H), 1.98-1.93 (m, 2H), 1.73-1.68 (m, 1H), 1.66-1.60 (m, 1H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 199.2, 143.1, 142.2, 139.4, 135.2, 132.1, 130.7, 129.1, 119.7, 36.8, 35.5, 29.1, 28.4, 25.6, 25.5, 23.5; IR (neat): ν 1660.7, 1344.4, 1246.0, 887.3, 827.5, 607.6, 584.4, 553.6 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{16}H_{17}{}^{79}BrNaO$ [M+Na]$^+$: 327.0355; Found: 327.0362.



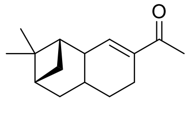
5w

Product **5w** was obtained in 43% yield (110.9 mg) as colorless oil. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 9:1. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.14 (d, *J* = 8.4 Hz, 1H), 6.88-6.86 (m, 1H), 6.77-6.74 (m, 1H), 6.61 (d, *J* = 2.8 Hz, 1H), 3.78 (s, 3H), 2.87-2.79 (m, 3H), 2.66-2.64 (m, 1H), 2.47-2.42 (m, 1H), 2.32 (s, 3H), 2.26-2.17 (m, 1H), 1.96-1.89 (m, 2H), 1.69-1.63 (m, 2H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 199.4, 157.4, 143.9, 139.4, 137.4, 132.0, 130.3, 113.4, 112.8, 55.4, 36.2, 36.1, 29.9, 28.5, 25.8, 25.5, 23.6; IR (neat): ν 1664.6, 1562.3, 1211.3, 1109.1, 977.9, 856.4, 659.7, 597.9 cm^{-1}; MS (EI): 256 (M$^+$, 89), 241 (13), 213 (21), 199 (7), 172 (31), 160 (100), 147 (11), 115 (17); HRMS (EI): Exact mass calcd for $C_{17}H_{20}O_2$ [M]$^+$: 256.1463, Found: 256.1461.



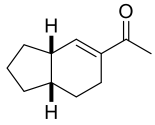
5x

Product **5x** was obtained in 30% yield (68.4 mg) as white solid. Mp: 101-103 °C. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 6:1. ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.20 (dd, *J* = 7.5, 1.5 Hz, 1H), 7.14-7.10 (m, 1H), 6.93 (td, *J* = 7.5, 1.0 Hz, 1H), 6.83 (dd, *J* = 8.5, 1.5 Hz, 1H), 6.77-6.76 (m, 1H), 4.27-4.24 (m, 1H), 3.86 (dd, *J* = 11.0, 8.5 Hz, 1H), 2.96-2.94 (m, 2H), 2.47-2.42 (m, 1H), 2.33 (s, 3H), 2.26-2.20 (m, 1H), 2.11-2.07 (m, 1H), 1.80-1.72 (m, 1H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 198.9, 154.5, 142.3, 137.3, 129.7, 127.7, 125.2, 121.2, 117.0, 65.9, 34.9, 33.0, 28.0, 25.6, 22.5; IR (neat): ν 1668.4, 1487.1, 1456.3, 1386.8, 1352.1, 1112.9, 1043.5, 582.5 cm^{-1}; MS (EI): 228 (M$^+$, 100), 185 (18), 132 (97), 131 (68), 115 (10), 107 (18), 97 (18), 77 (14); HRMS (EI): Exact mass calcd for $C_{15}H_{16}O_2$ [M]$^+$: 228.1150, Found: 228.1153.



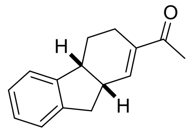
5y

Product **5y** was obtained in 34% yield (26.3 mg) as colorless oil. ^{1}H NMR analysis of the crude mixture revealed that the dr value was > 20:1. ^{1}H NMR (400 MHz, CDCl$_3$): δ 6.65 (t, *J* = 2.8 Hz, 1H), 2.84-2.82 (m, 1H), 2.46 (dt, *J* = 16.8, 4.4 Hz, 1H), 2.29 (s, 3H), 2.21-2.16 (m, 2H), 2.10-2.04 (m, 1H), 1.99-1.96 (m, 1H), 1.93-1.88 (m, 2H), 1.82-1.75 (m, 1H), 1.62-1.58 (m, 1H), 1.51-1.47 (m, 1H), 1.26 (s, 3H), 1.06 (d, *J* = 10.0 Hz, 1H), 0.97 (s, 3H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 198.7, 146.6, 141.3, 47.0, 41.4, 40.1, 38.6, 33.4, 33.2, 26.9, 26.8, 26.5, 25.3, 21.6, 20.8; IR (neat): ν 1660.7, 1633.7, 1500.6, 1037.7, 879.5, 819.8, 740.7, 609.5 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{15}H_{22}NaO$ [M+Na]$^+$: 241.1563; Found: 241.1554.



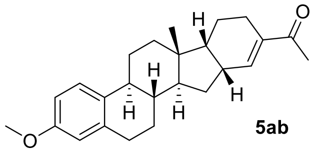
5z

Product **5z** was obtained in 49% yield (80.9 mg) as colorless oil. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 11:1. ^{1}H NMR (400 MHz, CDCl$_3$): δ 6.87-6.96 (m, 1H), 2.61-2.59 (m, 1H), 2.36-2.33 (m, 1H), 2.29 (s, 3H), 2.14-2.04 (m, 2H), 2.00-1.93 (m, 1H), 1.87-1.78 (m, 1H), 1.69-1.62 (m, 2H), 1.60-1.53 (m, 1H), 1.43-1.33 (m, 3H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 199.5, 144.4, 139.0, 40.6, 36.3, 31.9, 31.0, 26.0, 25.4, 24.4, 21.4; IR (neat): ν 1784.2, 1668.4, 1386.4, 1352.1, 1249.9, 1072.4, 858.3, 607.6 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{11}H_{16}NaO$ [M+Na]$^+$: 187.1093; Found: 187.1094.



5aa

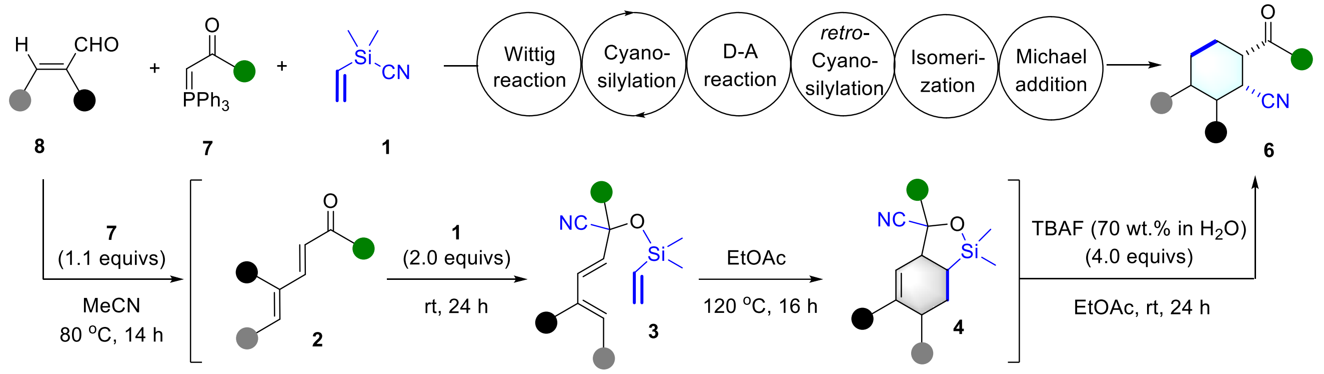
Product **5aa** was obtained in 40% yield (76.2 mg) as colorless oil. ^{1}H NMR analysis of the crude mixture revealed that the dr value was >20:1. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.25-7.16 (m, 4H), 6.89 (t, *J* = 2.0 Hz, 1H), 3.29-3.24 (m, 3H), 2.84-2.79 (m, 1H), 2.29 (s, 3H), 2.25-2.20 (m, 2H), 1.95-1.88 (m, 1H), 1.79-1.73 (m, 1H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 199.2, 145.9, 142.9, 142.0, 139.8, 126.8, 126.7, 125.0, 123.8, 42.6, 40.4, 37.8, 25.4, 25.1, 20.9; IR (neat): ν 2358.9, 1685.8, 1662.6, 1477.5, 1246.0, 842.9, 740.7, 597.9 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{15}H_{16}NaO$ [M+Na]$^+$: 235.1093; Found: 235.1084.



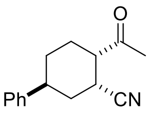
5ab

Product **5ab** was obtained in 69% yield (75.3 mg) as white solid. Mp: 118-120 °C. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 12:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.22-7.20 (m, 1H), 6.94-6.92 (m, 1H), 6.71-6.70 (m, 1H), 6.63-6.62 (m, 1H), 3.77 (s, 3H), 2.99-2.93 (m, 1H), 2.88-2.80 (m, 2H), 2.69-2.63 (m, 1H), 2.33-2.25 (m, 4H), 2.19-2.15 (m, 1H), 1.87-1.75 (m, 5H), 1.65-1.50 (m, 6H), 1.43-1.29 (m, 3H), 0.91 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.0, 157.6, 145.6, 139.3, 138.1, 132.9, 126.4, 114.0, 111.6, 55.3, 48.0, 47.3, 44.6, 44.0, 39.2, 36.9, 33.3, 32.6, 30.1, 28.3, 26.4, 22.5, 22.4, 22.0, 20.4; IR (neat): *v* 1683.9, 1658.9, 1500.6, 1311.6, 1234.4, 1037.7, 819.8, 651.9 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{25}H_{32}NaO_2$ $[M+Na]^+$: 387.2295; Found: 387.2291.

7. General procedure for the tandem synthesis of 2-acetyl cyclohexancarbonitriles 6

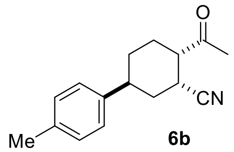


Under N_2 atmosphere, to a 10 mL sealed tube was added α,β-unsaturated aldehydes **8** (1.0 mmol) and ylide **7** (1.1 mmol), followed by the addition of anhydrous acetonitrile (1.0 mL). The resulting mixture was heated to 80 °C until full conversion of **8** by TLC analysis (ca. 14 h). After cooling to rt, $Me_2(CH{=}CH_2)SiCN$ **1** (250 μL, 2.0 mmol) was added. After being stirred at the same temperature for 24 h, the reaction mixture was rapidly passed through a short pad of silica gel, and washed with ether. The organic solution was concentrated in vacuo to give the crude silicon-tethered triene **3**, which was dissolved in EtOAc (2.0 mL). The resulting mixture was heated to 120 °C for 16 h. After full conversion of **3** by TLC analysis, the reaction mixture was cooled to rt, and TBAF (1.491 g, 70 wt.% in H_2O) was added. The suspension was stirred at rt until full conversion of **4** by TLC analysis (ca. 24 h), which was then poured in to H_2O (10 mL), and extracted with EtOAc (20 mL × 3). The combined organic phases were washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash column chromatography using PE/EtOAc (2/1, v/v) as the eluent to give the desired products **6**. (Note: *P*-ylide $Ph_3P{=}CHCONEt_2$ **7f** (3.8 mg, 0.01 mmol) was added as an extra catalyst in the cyanosilylation step in the cases of **6i-o**.)



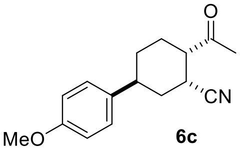
6a

Product **6a** was obtained in 55% yield (125.4 mg) as colorless oil. ^1H NMR analysis of the crude mixture revealed that the dr value was >20:1. ^1H NMR (400 MHz, $CDCl_3$): δ 7.27-7.23 (m, 2H), 7.19-7.13 (m, 3H), 3.35-3.34 (m, 1H), 2.92-2.86 (m, 1H), 2.42 (dt, *J* = 12.8, 3.6 Hz, 1H), 2.26-2.16 (m, 5H), 2.08-2.03 (m, 1H), 1.89-1.78 (m, 1H), 1.72-1.65 (m, 1H), 1.58-1.47 (m, 1H); ^{13}C NMR (100 MHz, CD_2Cl_2): δ 207.2, 145.1, 129.0, 127.2, 127.0, 120.4, 51.8, 39.9, 35.9, 33.6, 29.5, 27.9, 25.6; IR (neat): ν 1708.9, 1494.8, 1363.7, 1178.5, 760.0, 700.2, 592.2, 570.9 cm^{-1}; MS (EI): 227 (M^+, 100), 209 (10), 184 (47), 180 (19), 156 (23), 143 (21), 131 (17), 104 (97); HRMS (EI): Exact mass calcd for $C_{15}H_{17}NO$ $[M]^+$: 227.1310, Found: 227.1308.



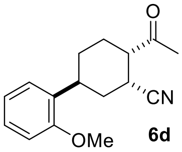
6b

Product **6b** was obtained in 63% yield (151.8 mg) as white solid. Mp: 101-103 °C. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 4.7:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.15-7.13 (m, 2H), 7.11-7.09 (m, 2H), 3.42-3.40 (m, 1H), 2.96-2.90 (m, 1H), 2.50-2.45 (m, 1H), 2.33 (s, 3H), 2.31-2.26 (m, 1H), 2.25 (s, 3H), 2.22-2.21 (m, 1H), 2.13-2.09 (m, 1H), 1.95-1.85 (m, 1H), 1.77-1.72 (m, 1H), 1.59-1.52 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 206.7, 141.5, 136.5, 129.5, 126.8, 120.1, 51.8, 39.1, 35.9, 33.3, 29.4, 27.7, 25.4, 21.1; IR (neat): ν 1708.0, 1515.1, 1454.4, 1363.7, 1177.6, 816.9, 805.3, 721.4 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{16}H_{19}NNaO$ [M+Na]$^+$: 264.1359; Found: 264.1359.



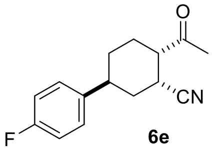
6c

Product **6c** was obtained in 77% yield (197.9 mg) as white solid. Mp: 58-60 °C. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 5.2:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.16-7.14 (m, 2H), 6.90-6.87 (m, 2H), 3.82 (s, 3H), 3.43-3.42 (m, 1H), 2.97-2.91 (m, 1H), 2.51-2.45 (m, 1H), 2.33-2.22 (m, 5H), 2.15-2.10 (m, 1H), 1.98-1.87 (m, 1H), 1.77-1.69 (m, 1H), 1.59-1.52 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 206.8, 158.5, 136.6, 127.8, 120.1, 114.2, 55.4, 51.8, 38.7, 36.1, 33.5, 29.5, 27.7, 25.4; IR (neat): ν 1702.2, 1511.3, 1245.1, 1177.6, 1031.9, 826.5, 805.3, 783.1 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{16}H_{19}NNaO_2$ [M+Na]$^+$: 280.1308; Found: 280.1308.



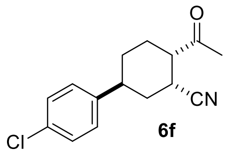
6d

Product **6d** was obtained in 67% yield (172.3 mg) as colorless oil. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 2.5:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.22-7.19 (m, 1H), 7.13-7.11 (m, 1H), 6.94-6.90 (m, 1H), 6.88-6.86 (m, 1H), 3.83 (s, 3H), 3.41-3.34 (m, 2H), 2.50-2.46 (m, 1H), 2.30-2.22 (m, 5H), 2.10-2.06 (m, 1H), 1.97-1.87 (m, 1H), 1.81-1.73 (m, 1H), 1.66-1.55 (m, 1H); ^{13}C NMR (100 MHz, CD_2Cl_2): δ 207.1, 157.2, 132.5, 127.7, 126.6, 120.7, 120.2, 110.7, 55.4, 52.0, 34.1, 33.2, 31.4, 29.5, 27.8, 25.5; IR (neat): ν 1683.9, 1653.0, 1367.5, 1332.8, 1157.3, 785.0, 752.2, 651.9 cm^{-1}; MS (EI): 257 (M$^+$, 100), 242 (5), 213 (64), 202 (21), 186 (20), 173 (15), 161 (20), 147 (19); HRMS (EI): Exact mass calcd for $C_{16}H_{19}NO_2$ [M]$^+$: 257.1416, Found: 257.1413.



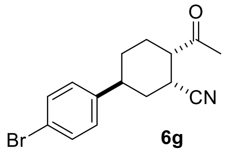
6e

Product **6e** was obtained in 51% yield (125.0 mg) as white solid. Mp: 64-66 °C. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 8.4:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.19-7.15 (m, 2H), 7.03-6.99 (m, 2H), 3.42-3.41 (m, 1H), 2.99-2.92 (m, 1H), 2.51-2.46 (m, 1H), 2.33-2.29 (m, 1H), 2.26 (s, 3H), 2.23-2.21 (m, 1H), 2.13-2.09 (m, 1H), 1.96-1.85 (m, 1H), 1.75-1.68 (m, 1H), 1.60-1.50 (m, 1H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 206.6, 161.8 (d, J = 243 Hz), 140.1, 128.3 (d, J = 8 Hz), 120.0, 115.6 (d, J = 21 Hz), 51.7, 38.9, 36.0, 33.4, 29.4, 27.7, 25.3; ^{19}F NMR (376 MHz, CDCl$_3$): δ -116.152; IR (neat): ν 1704.1, 1507.4, 1362.7, 1218.1, 1160.2, 847.7, 826.5, 811.1 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{15}H_{16}FNNaO$ [M+Na]$^+$: 268.1108; Found: 268.1103.



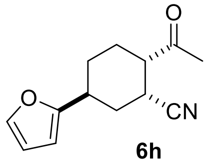
6f

Product **6f** was obtained in 67% yield (176.0 mg) as colorless oil. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 4.6:1. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.31-7.28 (m, 2H), 7.15-7.13 (m, 2H), 3.43-3.42 (m, 1H), 2.98-2.91 (m, 1H), 2.50-2.46 (m, 1H), 2.33-2.29 (m, 1H), 2.26 (s, 3H), 2.24-2.21 (m, 1H), 2.13-2.08 (m, 1H), 1.96-1.85 (m, 1H), 1.75-1.67 (m, 1H), 1.61-1.50 (m, 1H); ^{13}C NMR (100 MHz, CD$_2$Cl$_2$): δ 206.5, 142.9, 132.6, 129.0, 128.3, 120.0, 51.7, 39.0, 35.7, 33.2, 29.3, 27.8, 25.3; IR (neat): ν 1703.1, 1651.1, 1494.8, 1361.7, 1091.7, 814.0, 719.5, 621.1 cm^{-1}; MS (EI): 261 (M$^+$, 100), 243 (14), 218 (61), 206 (19), 190 (21), 177 (20), 138 (91), 125 (72); HRMS (EI): Exact mass calcd for $C_{15}H_{16}NO^{35}Cl$ [M]$^+$: 261.0920, Found: 261.0917.



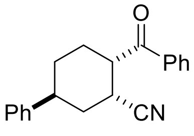
6g

Product **6g** was obtained in 60% yield (183.6 mg) as white solid. Mp: 85-87 °C. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 9.9:1. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.46-7.43 (m, 2H), 7.10-7.08 (m, 2H), 3.43-3.41 (m, 1H), 2.98-2.90 (m, 1H), 2.50-2.45 (m, 1H), 2.34-2.28 (m, 1H), 2.25 (s, 3H), 2.23-2.20 (m, 1H), 2.13-2.08 (m, 1H), 1.96-1.85 (m, 1H), 1.75-1.67 (m, 1H), 1.57-1.50 (m, 1H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 206.4, 143.4, 131.9, 128.7, 120.7, 119.9, 51.7, 39.1, 35.7, 33.1, 29.3, 27.7, 25.3; IR (neat): ν 1703.2, 1490.0, 1361.8, 1178.5, 1072.4, 990.5, 810.1, 614.3 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{15}H_{16}{}^{79}BrNNaO$ [M+Na]$^+$: 328.0307; Found: 328.0308.



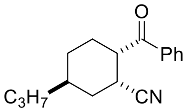
6h

Product **6h** was obtained in 83% yield (180.6 mg) as colorless oil. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 2.0:1. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.32 (dd, J = 2.0, 0.8 Hz, 1H), 6.30 (dd, J = 3.2, 2.0 Hz, 1H), 6.03-6.02 (m, 1H), 3.40-3.38 (m, 1H), 3.11-3.04 (m, 1H), 2.49-2.44 (m, 1H), 2.43-2.38 (m, 1H), 2.32-2.26 (m, 1H), 2.25-2.22 (m, 4H), 1.92-1.81 (m, 1H), 1.76-1.68 (m, 1H), 1.62-1.51 (m, 1H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 206.6, 157.5, 141.4, 119.8, 110.1, 104.1, 51.6, 33.2, 32.9, 30.4, 28.7, 27.7, 24.6; IR (neat): ν 1666.5, 1560.4, 1396.5, 1275.0, 1153.4, 883.4, 734.9, 600.0 cm^{-1}; MS (EI): 217 (M$^+$, 98), 199 (11), 184 (18), 173 (54), 159 (14), 147 (37), 133 (17), 94 (100); HRMS (EI): Exact mass calcd for $C_{13}H_{15}NO_2$ [M]$^+$: 217.1103, Found: 217.1102.



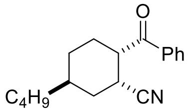
6i

Product **6i** was obtained in 99% yield (286.1 mg) as white solid. Mp: 130-132 °C. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 4.0:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93-7.90 (m, 2H), 7.63-7.59 (m, 1H), 7.53-7.49 (m, 2H), 7.37-7.33 (m, 2H), 7.27-7.26 (m, 3H), 3.46-3.41 (m, 1H), 3.39-3.36 (m, 1H), 3.12-3.04 (m, 1H), 2.35-2.30 (m, 1H), 2.26-2.21 (m, 1H), 2.17-2.12 (m, 1H), 2.09-1.99 (m, 1H), 1.97-1.89 (m, 1H), 1.78-1.68 (m, 1H); ^{13}C NMR (100 MHz, CD_2Cl_2): δ 199.3, 144.5, 135.3, 133.6, 129.1, 128.8, 128.4, 127.0, 126.9, 120.2, 46.8, 39.7, 36.2, 33.2, 30.4, 26.6; IR (neat): ν 1681.9, 1651.1, 1446.6, 1224.8, 1062.8, 949.0, 761.9, 700.2 cm^{-1}; MS (EI): 289 (M$^+$, 6), 245 (4), 184 (2), 172 (15), 131 (3), 105 (100), 91 (6), 77 (27); HRMS (EI): Exact mass calcd for $C_{20}H_{19}NO$ [M]$^+$: 289.1467, Found: 289.1465.



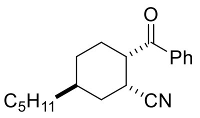
6j

Product **6j** was obtained in 32% yield (81.6 mg) as white solid. Mp: 69-71 °C. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 3.0:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88-7.86 (m, 2H), 7.58-7.56 (m, 1H), 7.50-7.46 (m, 2H), 3.30-3.26 (m, 1H), 3.25-3.23 (m, 1H), 2.19-2.13 (m, 1H), 2.11-2.05 (m, 1H), 2.01-1.96 (m, 1H), 1.90-1.75 (m, 2H), 1.42-1.33 (m, 3H), 1.30-1.24 (m, 2H), 1.18-1.08 (m, 1H), 0.92 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.6, 135.5, 133.5, 129.0, 128.4, 120.4, 47.4, 38.7, 35.6, 33.0, 32.2, 30.2, 26.3, 19.9, 14.4; IR (neat): ν 1683.9, 1447.6, 1239.3, 1222.9, 943.2, 939.4, 761.9, 698.2 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{17}H_{21}NNaO$ [M+Na]$^+$: 278.1515; Found: 278.1512.



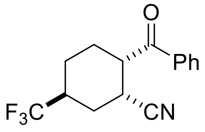
6k

Product **6k** was obtained in 34% yield (91.5 mg) as colorless oil. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 2.8:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88-7.86 (m, 2H), 7.60-7.57 (m, 1H), 7.50-7.46 (m, 2H), 3.30-3.25 (m, 1H), 3.24-3.23 (m, 1H), 2.19-2.14 (m, 1H), 2.11-2.06 (m, 1H), 2.01-1.96 (m, 1H), 1.91-1.83 (m, 1H), 1.81-1.76 (m, 1H), 1.42-1.36 (m, 1H), 1.34-1.26 (m, 6H), 1.18-1.09 (m, 1H), 0.91 (t, *J* =6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.5, 135.5, 133.6, 129.0, 128.5, 122.4, 45.6, 33.6, 32.7, 32.0, 29.3, 28.4, 26.7, 25.6, 22.9, 14.2; IR (neat): ν 1675.2, 1595.2, 1450.5, 1212.3, 1187.2, 956.7, 768.7, 694.4 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{18}H_{23}NNaO$ [M+Na]$^+$: 292.1672; Found: 292.1664.



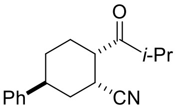
6l

Product **6l** was obtained in 50% yield (141.5 mg) as white solid. Mp: 75-77 °C. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 3.0:1. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.88-7.86 (m, 2H), 7.60-7.56 (m, 1H), 7.50-7.46 (m, 2H), 3.30-3.25 (m, 1H), 3.24-3.23 (m, 1H), 2.19-2.15 (m, 1H), 2.11-2.06 (m, 1H), 2.00-1.97 (m, 1H), 1.91-1.83 (m, 1H), 1.81-1.74 (m, 1H), 1.43-1.27 (m, 9H), 1.18-1.08 (m, 1H), 0.90 (t, J = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 199.6, 135.5, 133.5, 129.0, 128.4, 120.4, 47.4, 36.5, 35.6, 33.3, 32.3, 32.1, 30.2, 26.5, 26.4, 22.7, 14.2; IR (neat): ν 2925.1, 1670.4, 1596.1, 1448.6, 1223.9, 927.8, 693.4, 602.8 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{19}H_{25}NNaO$ [M+Na]$^+$: 306.1828; Found: 306.1827.



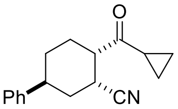
6m

Product **6m** was obtained in 90% yield (252.9 mg) as colorless oil. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 5.0:1. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.88-7.86 (m, 2H), 7.64-7.60 (m, 1H), 7.53-7.49 (m, 2H), 3.37-3.32 (m, 2H), 2.61-2.54 (m, 1H), 2.41-2.37 (m, 1H), 2.24-2.16 (m, 2H), 1.96-1.88 (m, 1H), 1.83-1.75 (m, 1H), 1.64-1.58 (m, 1H); ^{13}C NMR (125 MHz, CDCl$_3$): δ 198.5, 135.0, 133.9, 129.2, 128.4, 127.1 (q, J = 276 Hz), 119.1, 46.4, 38.2 (q, J = 27.5 Hz), 29.0, 28.1 (d, J = 2.5 Hz), 24.7, 24.3 (d, J = 2.5 Hz); ^{19}F NMR (376 MHz, CDCl$_3$): δ -73.371; IR (neat): ν 1681.9, 1651.1, 1338.6, 1273.0, 1151.5, 954.8, 785.0, 692.4 cm^{-1}; MS (EI): 281 (M$^+$, 2), 262 (1), 158 (1), 133 (1), 105 (100), 78 (3), 77 (32), 65 (1); HRMS (EI): Exact mass calcd for $C_{15}H_{14}NOF_3$ [M]$^+$: 281.1027, Found: 281.1025.



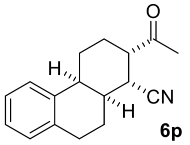
6n

Product **6n** was obtained in 58% yield (147.9 mg) as white solid. Mp: 91-93 °C. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 6.4:1. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.35-7.31 (m, 2H), 7.26-7.21 (m, 3H), 3.37-3.34 (m, 1H), 3.02-2.96 (m, 1H), 2.91-2.84 (m, 1H), 2.67-2.63 (m, 1H), 2.29-2.23 (m, 2H), 2.16-2.10 (m, 1H), 1.97-1.86 (m, 1H), 1.81-1.74 (m, 1H), 1.66-1.55 (m, 1H), 1.15 (d, J = 6.8 Hz, 3H), 1.12 (d, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 212.6, 144.5, 128.8, 126.93, 126.90, 120.2, 49.6, 39.6, 37.7, 36.0, 33.3, 29.5, 25.4, 19.1, 18.2; IR (neat): ν 1705.1, 1492.9, 1455.3, 1360.8, 1093.7, 984.7, 958.6, 758.0 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{17}H_{21}NNaO$ [M+Na]$^+$: 278.1515; Found: 278.1516.

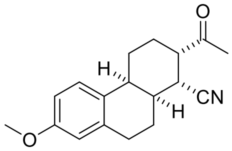


6o

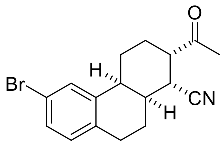
Product **6o** was obtained in 73% yield (184.7 mg) as colorless oil. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 4.8:1. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.35-7.31 (m, 2H), 7.25-7.22 (m, 3H), 3.44-3.41 (m, 1H), 3.03-2.97 (m, 1H), 2.72-2.67 (m, 1H), 2.41-2.36 (m, 1H), 2.30-2.24 (m, 1H), 2.17-2.12 (m, 1H), 2.09-2.02 (m, 1H), 2.01-1.90 (m, 1H), 1.83-1.76 (m, 1H), 1.68-1.57 (m, 1H), 1.15-1.12 (m, 2H), 0.98-0.94 (m, 2H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 208.6, 144.6, 128.8, 126.9, 126.8, 120.2, 51.8, 39.7, 36.0, 33.3, 29.7, 25.3, 18.7, 11.7, 11.6; IR (neat): ν 1702.2, 1511.3, 1388.8, 1247.0, 1084.9, 986.6, 981.8, 700.2 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{17}H_{19}NNaO$ [M+Na]$^+$: 276.1359; Found: 276.1353.


6p

Product **6p** was obtained in 57% yield (71.1 mg) as colorless oil. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 3.4:1. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.17-7.12 (m, 3H), 7.11-7.07 (m, 1H), 3.22 (s, 1H), 3.15-3.11 (m, 1H), 2.92-2.89 (m, 2H), 2.62-2.58 (m, 1H), 2.53-2.48 (m, 1H), 2.25 (s, 3H), 2.20-2.17 (m, 1H), 2.00-1.89 (m, 3H), 1.68-1.63 (m, 2H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 207.3, 139.6, 134.8, 129.3, 129.2, 126.5, 126.3, 120.2, 46.6, 37.4, 36.4, 34.4, 31.1, 29.5, 27.9, 25.1, 23.2; IR (neat): ν 2933.7, 2360.9, 1707.0, 1489.1, 1182.4, 910.4, 756.0, 733.0 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{17}H_{19}NNaO$ [M+Na]$^+$: 276.1359; Found: 276.1356.


6q

Product **6q** was obtained in 66% yield (93.4 mg) as white solid. Mp: 118-120 °C. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 1.9:1. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.06-7.03 (m, 1H), 6.75-6.72 (m, 1H), 6.62-6.61 (m, 1H), 3.77 (s, 3H), 3.21 (s, br, 1H), 3.09-3.04 (m, 1H), 2.88-2.85 (m, 2H), 2.61-2.56 (m, 1H), 2.51-2.45 (m, 1H), 2.24 (s, 3H), 2.18-2.14 (m, 1H), 1.98-1.88 (m, 3H), 1.65-1.62 (m, 2H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 207.3, 158.1, 136.0, 131.9, 130.2, 120.3, 113.6, 112.7, 55.4, 46.7, 37.6, 35.7, 34.4, 31.1, 29.8, 27.9, 25.1, 23.2; IR (neat): ν 2923.2, 1705.1, 1610.6, 1498.7, 1248.9, 1129.3, 1034.8, 818.8 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{18}H_{21}NNaO_2$ [M+Na]$^+$: 306.1464; Found: 306.1462.

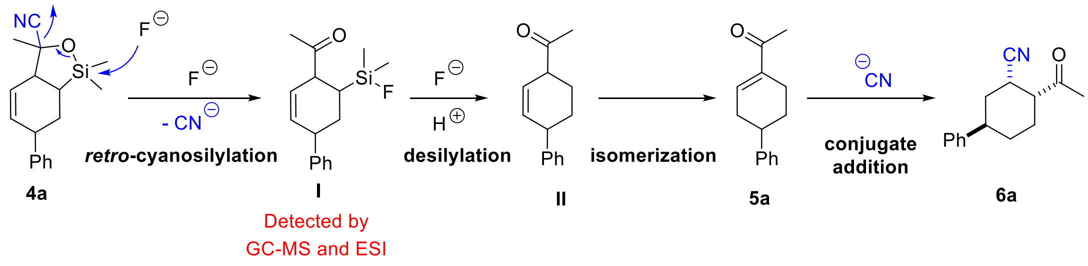

6r

Product **6r** was obtained in 54% yield (89.6 mg) as white solid. Mp: 194-196 °C. ^{1}H NMR analysis of the crude mixture revealed that the dr value was 3.0:1. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.28-7.27 (m, 1H), 7.25-7.23 (m, 1H), 6.97-6.95 (m, 1H), 3.22 (s, br, 1H), 3.12-3.07 (m, 1H), 2.86-2.81 (m, 2H), 2.61-2.56 (m, 1H), 2.50-2.44 (m, 1H), 2.25 (s, 3H), 2.21-2.18 (m, 1H), 2.00-1.87 (m, 3H), 1.68-1.61 (m, 2H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 207.1, 141.9, 133.8, 132.0 , 130.9, 129.6, 120.0, 119.7, 46.5, 37.0, 36.3, 34.2, 30.9, 29.0, 27.9, 25.0, 23.0; IR (neat): ν 1704.1, 1507.4, 1250.9, 1218.1, 1160.2, 847.7, 811.1, 642.3 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{17}H_{18}{}^{79}BrNNaO$ [M+Na]$^+$: 354.0464; Found: 354.0456.

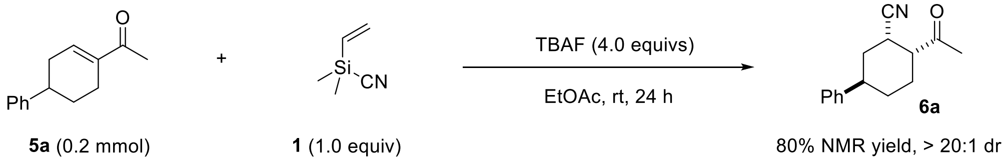
8. The possible pathway for the generation of 5a and 6a

Based on our results, we tentatively proposed a pathway for the generation of **5a** and **6a** from the DA adduct **4a** (Scheme S2A). First, the *retro*-cyanosilylation reaction of **4a** occurred to give the intermediate **I** and cyanide anion (CN^-) in the presence of fluoride ion (F^-), and the intermediate **I** could be detected by GC-MS and ESI when NH_4F was used as the fluoride ion source. Under the action of another F^-, the intermediate **I** subsequently underwent desilylation to provide the intermediate **II**, which then rapidly isomerized to give enone **5a**. On the other hand, the desired **6a** might generate from **5a** via a conjugate addition with the residual cyanide anion, in the absence of $Mn(OAc)_4$ as the CN^- capturing agent. This result was further confirmed by the control experiment that **5a** could be readily converted into **6a** in 80% NMR yield with >20:1 dr in the presence of $Me_2(CH_2{=}CH)SiCN$ (1.0 equiv) and TBAF (4.0 equivs), as shown in Scheme S2B.

A) Proposed mechanism for generation of 7a and 8a from 6a



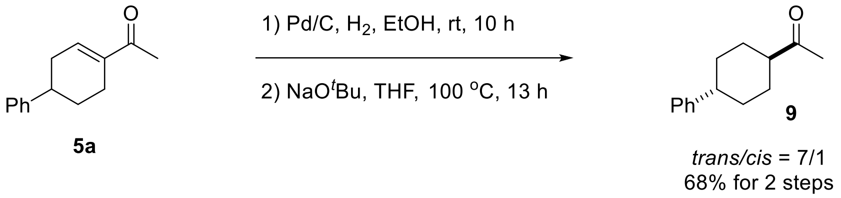
B) Control experiment



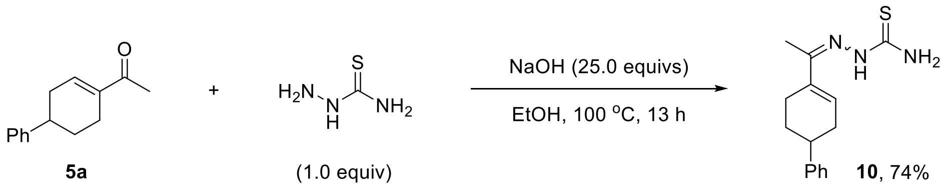
Scheme S2. Proposed mechanism and control experiment.

9. Synthetic applications

9.1 Synthetic transformations of 5a



To a 25 mL flask was added ketone **5a** (40.0 mg, 0.2 mmol), followed by the addition of EtOH (1.0 mL) and Pd/C (10 wt.%, 4.0 mg). This resulting mixture was in a vacuum and back-filled with H_2 (1 atm). After being stirred at rt for 10 h, the reaction solution was filtered, and the filtered-cake was washed with EtOH (10.0 mL). The filtrate was evaporated under vacuo to give the crude product, which was re-dissolved in THF (1.0 mL). To this solution was added NaO^{t}Bu (48.0 mg, 0.5 mmol), and then stirred at 100 ^{o}C for 13 h. After removal of solvent THF under the reduced pressure, the crude residue was purified by silica gel column chromatography using PE/EtOAc (10:1, v/v) as the eluent to give 1-(4-phenylcyclohexyl) ethanone **9** in 68% yield (27.5 mg) as colorless oil.[9] ^{1}H NMR analysis of the crude residue revealed that the ratio of *trans*- and *cis*-isomer was 7:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32-7.25 (m, 2H), 7.21-7.19 (m, 3H), 2.53-2.48 (m, 1H), 2.44-2.38 (m, 1H), 2.18 (s, 3H), 2.06-2.00 (m, 4H), 1.56-1.45 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 212.2, 147.0, 128.5, 126.9, 126.3, 51.2, 43.9, 33.5, 28.8, 28.2.



To a sealed tube was added **5a** (0.20 mmol, 40.0 mg) and thiosemicarbazide (0.20 mmol, 18.2 mg), followed by the addition of EtOH (1.0 mL) and NaOH (5.0 mmol, 200.0 mg) at rt. The reaction mixture was stirred at 100 ^{o}C until full conversion of **5a** (ca. 13 h). The 4M HCl aqueous solution was then added slowly until the pH was 2, and the resulting mixture was filtered to afford analytically pure **10** in 74% yield (40.6 mg) as brown solid. Mp: 161-163 ^{o}C. ^{1}H NMR analysis revealed the *Z*/*E* value was >20:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.65 (s, 1H), 7.34-7.31 (m, 2H), 7.24-7.21 (m, 3H), 6.43-6.41 (m, 1H), 6.33 (s, br, 1H), 2.85-2.78 (m, 1H), 2.65-2.53 (m, 2H), 2.39-2.32 (m, 2H), 2.04 (s, 3H), 1.80-1.75 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 179.3, 149.2, 146.3, 136.1, 132.1, 128.7,

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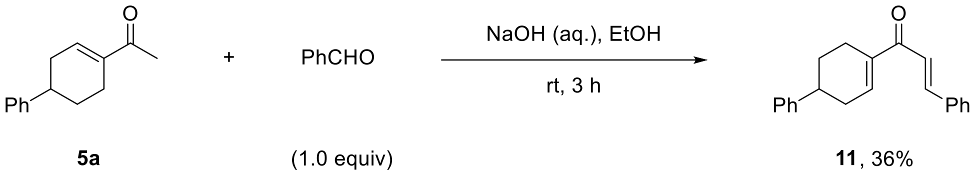
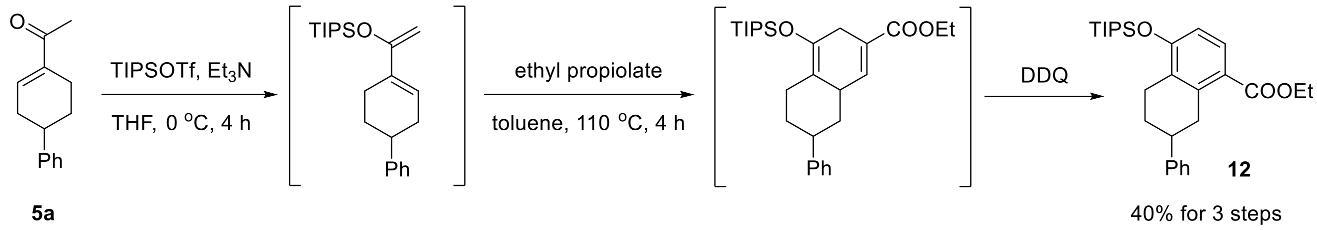
[9] H. E. Zimmerman, P. S. Mariano, *J. Am. Chem. Soc.* **1968**, *90*, 6091-6096.

126.9, 126.5, 39.8, 34.5, 29.7, 25.2, 11.5; IR (neat): *v* 1714.7, 1365.6, 1224.8, 1093.6, 840.9, 744.5, 698.2, 601.8 cm^{-1}; MS (EI): 273 (M$^+$, 69), 256 (45), 240 (100), 214 (35), 128 (28), 104 (62), 91 (82), 77 (34); HRMS (EI): Exact mass calcd for $C_{15}H_{19}N_3S$ [M]$^+$: 273.1300, Found: 273.1302.

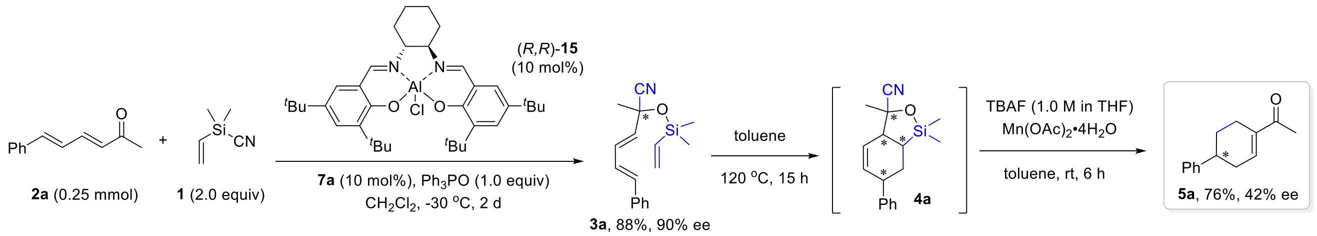


To a solution of **5a** (0.20 mmol, 40.0 mg) in EtOH (0.5 mL) was added NaOH (0.5 mL, 0.4 M in H_2O) at rt. After being stirred for 10 min, benzaldehyde (0.20 mmol, 21 μL) was added. The reaction mixture was stirred for about 3 h until full conversion of **5a** by TLC analysis. Then H_2O (5 mL) was added to the resulting mixture, and extracted with EtOAc (20 mL × 3). The combined organic phases were washed with brine, and dried over Na_2SO_4. After evaporation, the residue was purified by silica gel column chromatography using PE/EtOAc (20/1, v/v) as the eluent to afford the product **11** in 36% yield (20.7 mg) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68-7.64 (m, 1H), 7.61-7.58 (m, 2H), 7.41-7.38 (m, 3H), 7.36-7.32 (m, 3H), 7.25-7.21 (m, 3H), 7.11-7.09 (m, 1H), 2.89-2.82 (m, 1H), 2.72-2.60 (m, 2H), 2.48-2.37 (m, 2H), 2.12-2.07 (m, 1H), 1.84-1.73 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.8, 146.1, 142.9, 140.3, 139.3, 135.4, 130.2, 129.0, 128.7, 128.3, 127.0, 126.5, 121.5, 39.5, 34.4, 29.5, 24.5; IR (neat): *v* 1654.9, 1597.1, 1492.9, 1448.5, 1384.9, 974.1, 754.2, 696.3 cm^{-1}; MS (EI): 287 (M$^+$, 41), 245 (100), 203 (89), 161 (30), 119 (20), 91 (13), 73 (14), 52 (3); HRMS (EI): Exact mass calcd for $C_{21}H_{20}O$ [M]$^+$: 288.1514, Found: 288.1512.



Under N_2 atmosphere, to a solution of **5a** (0.20 mmol, 40.0 mg) in anhydrous THF (1.0 mL) was added Et_3N (0.22 mmol, 60 μL) at 0 °C, followed by the addition of TIPSOTf (0.50 mmol, 70 μL). The reaction mixture was stirred at the same temperature for about 4 h until full conversion of **5a** by TLC analysis. *n*-Hexane (10 mL) was added slowly, and the resulting mixture was washed with saturated $NaHCO_3$ (aq., 5 mL) and brine, successively. The organic phases were evaporated to give the crude silyl enol ether, which was directly dissolved in anhydrous toluene (0.5 mL). To this solution were added ethyl propiolate (0.30 mmol, 30 μL). After being stirred at 110 °C for 4 h and cooled to rt, DDQ (0.20 mmol, 45.4 mg) was added to the reaction mixture, and then stirred at rt for 2 h. After full conversion, the reaction mixture was directly purified by silica gel column chromatography using PE/EtOAc (20/1, v/v) as the eluent to deliver the product **12** in 40% yield (36.3 mg) as colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.71-7.70 (m, 1H), 7.33-7.27 (m, 4H), 7.22-7.19 (m, 1H), 6.68-6.67 (m, 1H), 4.26 (q, *J* = 7.0 Hz, 2H), 3.53-3.48 (m, 1H), 3.12-3.04 (m, 2H), 2.88-2.84 (m, 1H), 2.76-2.70 (m, 1H), 2.17-2.14 (m, 1H), 1.91-1.83 (m, 1H), 1.35-1.31 (m, 6H), 1.13-1.12 (m, 18H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 167.7, 157.4, 146.8, 141.1, 129.6, 128.6, 128.5, 127.1, 126.2, 122.6, 114.5, 60.4, 40.0, 36.6, 29.4, 25.4, 18.2, 14.5, 13.2; IR (neat): *v* 825.5, 1030.0, 1134.1, 1170.8, 1255.7, 1471.7, 1587.4, 1712.8 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{28}H_{40}O_3SiNa$ $[M+Na]^+$: 475.2639; Found: 475.2649.

9.2 Initial attempt to access chiral cyclohexyl ketone 5a



To a 5 mL vial was added ketone **2a** (86.0 mg, 0.5 mmol), (*R*,*R*)-**15** (30.4 mg, 0.05 mmol, 10 mol%), and ylide **7a** (15.9 mg, 0.05 mmol, 10 mol%), followed by the addition of anhydrous CH_2Cl_2 (1.0 mL) and Ph_3PO (139.0 mg, 1.0 equiv), successively. The resulting mixture was cooled down to -30 °C for 0.5 h, and $Me_2(CH_2{=}CH)SiCN$ **1** (125 μL, 1.0 mmol) was added. After being stirred at -30 °C for 2 days, the reaction mixture was rapidly passed through a short pad of silica gel, and washed with ether. The obtained organic solution was concentrated in vacuo to give the crude product, which was purified by silica gel column chromatography using PE/Et_2O (10:1, v/v) as the eluent to afford the desired cyanohydrin **3a** in 88% yield (124.5 mg). HPLC analysis (Chiralcel OJ-H, *i*-PrOH/hexane = 1/99, 1.0 mL/min, 230 nm; tr (minor) = 15.45 min, tr (major) = 20.13 min) gave the isomeric composition of the product: 90% ee. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43-7.41 (m, 2H), 7.36-7.32 (m, 2H), 7.28-7.24 (m, 1H), 6.77-6.62 (m, 3H), 6.25-6.16 (m, 1H), 6.07-6.02 (m, 1H), 5.84-5.73 (m, 2H), 1.71 (s, 3H), 0.31 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.6, 136.7, 135.8, 133.8, 132.9, 131.7, 128.9, 128.3, 126.8, 126.5, 120.7, 70.0, 30.8, -0.3; IR (neat): ν 1714.7, 1616.4, 1406.1, 1251.8, 960.5, 835.2, 748.4, 636.5 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{17}H_{21}NSiNaO$ $[M+Na]^+$: 306.1285; Found: 306.1288.

The cyanohydrin **3a** (56.6 mg, 0.2 mmol) was dissolved in toluene (1.0 mL). The reaction solution was heated to 120 ^{o}C for 15 h. After full conversion of **3a** by TLC analysis, the reaction mixture was cooled to room temperature, and $Mn(OAc)_2{\cdot}4H_2O$ (49.0 mg, 0.2 mmol), TBAF (0.8 mL, 1.0 M in THF) were added. The suspension was stirred at room temperature for 6 h until full conversion of **4a** by TLC analysis. The reaction mixture was poured in to H_2O (10 mL), and extracted with EtOAc (20 mL × 3). The combined organic phases were then washed with brine, dried over Na_2SO_4 and concentrated under the reduced pressure. The residue was purified by flash column chromatography using PE/EtOAc (4/1, v/v) as the eluent to give the cyclohexenyl ketone **5a**. HPLC analysis (Chiralpak AS-H, *i*-PrOH/hexane = 5/95, 1.0 mL/min, 205 nm; tr (major) = 7.29 min, tr (minor) = 8.63 min) gave the isomeric composition of the product: 42% ee. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.35-7.29 (m, 3H), 7.27-7.20 (m, 2H), 6.14-6.09 (m, 1H), 5.90-5.84 (m, 1H), 3.36-3.31 (m, 1H), 2.49-2.44 (m, 1H), 2.06-1.84 (m, 2H), 1.73 (s, 3H), 1.64-1.59 (m, 1H), 0.30 (s, 3H), 0.27 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.7, 136.9, 128.8, 127.8, 126.6, 123.2, 122.0, 77.5, 48.2, 43.5, 30.0, 28.0, 23.9, 0.01, -3.0; MS (EI): 283 (M^+, 55), 256 (35), 214 (87), 154 (81), 135 (100), 128 (29), 101 (27), 85 (56); HRMS (EI): Exact mass calcd for $C_{17}H_{21}NOSi$ $[M]^+$: 283.1392, Found: 283.1390.

10. X-ray crystallographic data

10.1 X-ray crystallographic data of 5u.[10]

Data intensity of **5u** was collected using a XtaLAB AFC12 (RINC): Kappa dual home/near diffractometer at 293(2) K. Data collection and reduction were done by using Olex2 and the structure was solved with the ShelXS structure solution program using direct methods and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for non-H atoms using SHELX-97. Hydrogen atoms were added at their geometrically idea positions and refined isotropically. Crystal data for **5u**: $C_{16}H_{18}O$, T = 296(2) K, monoclinic, space group P21/n, a = 7.27630(10) Å, b = 13.18790(10) Å, c = 12.60950(10) Å, α = 90 deg, β = 94.1790(10) deg, γ = 90 deg, V = 1206.78(2) Å^3. Z = 4, d_{calc} = 1.246 g/cm^3. 25070 reflections measured, 2436 [R_{int} = 0.0430, R_{sigma} = 0.0171], ($I > 2\sigma(I)$, final), R_1 = 0.0433, wR_2 = 0.1109 (all data).

5u

Table S3. Crystal data and structure refinement for **5u**.

Identification code	**5u**	
Empirical formula	$C_{16}H_{18}O$	
Formula weight	226.30	
Temperature	293(2) K	
Crystal system	Monoclinic	
Space group	P21/n	
Unit cell dimensions	a = 7.27630(10) Å	α = 90°
	b = 13.18790(10) Å	β = 94.1790(10)°
	c = 12.60950(10) Å	γ = 90°
Volume	1206.78(2) Å^3	
Z	4	
ρ_{calc}g	1.246(cm^3)	
μ	0.581(mm^{-1})	
F(000)	488.0	

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[10] Supplementary crystallographic data have been deposited at the Cambridge Crystallographic Data Center (CCDC number: 1921832).

Crystal size	0.32 × 0.28 × 0.22 mm^3
Radiation	CuKα (λ = 1.54184)
Theta range for data collection	9.718 to 148.868°.
Index ranges	$-9 \leq h \leq 9$, $-16 \leq k \leq 16$, $-15 \leq l \leq 15$
Reflections collected	25070
Independent reflections	2436 [R_{int} = 0.0430, R_{sigma} = 0.0171]
Data / restraints / parameters	2436/0/156
Goodness-of-fit on F^2	1.038
Final R indices [I>2sigma(I)]	R_1 = 0.0433, wR_2 = 0.1102
R indices (all data)	R_1 = 0.0441, wR_2 = 0.1109
Largest diff. peak and hole	0.42 and -0.20 e.Å^{-3}

Table S4. Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for **5u**. Ueq is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	***x***	***y***	***z***	**U(eq)**
O1	8858.6(13)	7389.1(8)	5663.9(8)	27.9(3)
C1	798.0(17)	6168.7(9)	3455.2(10)	19.2(3)
C2	400.9(17)	5132.4(10)	3497.1(11)	22.9(3)
C3	-1060.5(18)	4714.4(10)	2881.5(11)	24.0(3)
C4	-2175.1(17)	5338.0(10)	2219.3(11)	23.3(3)
C5	-1810.6(17)	6368.0(10)	2176.3(10)	21.1(3)
C6	-325.8(16)	6795.6(9)	2786.2(10)	18.8(3)
C7	6.1(18)	7925.7(9)	2740.0(10)	21.8(3)
C8	1976.8(18)	8211.3(10)	3109.0(11)	23.6(3)
C9	2514.5(17)	7734.2(9)	4195.4(10)	20.6(3)
C10	4361.2(18)	8108.4(10)	4646.2(10)	22.2(3)
C11	5861.9(17)	7530.6(9)	4847.9(10)	19.2(3)
C12	5859.0(17)	6417.6(10)	4574.8(11)	22.1(3)
C13	4260.7(17)	6163.2(10)	3764.9(10)	21.3(3)
C14	2442.7(16)	6569.5(9)	4144.1(10)	19.5(3)
C15	7573.2(17)	7950.6(10)	5394.9(10)	21.1(3)
C16	7700.6(19)	9070.5(10)	5643.2(11)	27.4(3)

Table S5. Anisotropic Displacement Parameters ($Å^2×10^3$) for **5u**. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	19.4(5)	33.1(5)	30.2(5)	3.3(4)	-4.6(4)	-0.9(4)
C1	15.7(6)	20.4(6)	21.3(6)	-1.0(5)	-0.5(5)	0.7(5)
C2	18.6(6)	20.4(6)	28.9(7)	1.1(5)	-2.9(5)	1.9(5)
C3	20.2(6)	19.2(6)	32.2(7)	-3.0(5)	-0.7(5)	-1.1(5)
C4	17.3(6)	27.0(7)	24.9(6)	-5.5(5)	-2.8(5)	-1.5(5)
C5	17.2(6)	25.5(6)	20.1(6)	0.5(5)	-1.6(5)	2.8(5)
C6	17.2(6)	20.8(6)	18.4(6)	-0.5(5)	0.6(5)	1.7(5)
C7	21.8(6)	19.9(6)	23.0(6)	2.1(5)	-3.8(5)	3.5(5)
C8	23.8(7)	18.2(6)	28.2(7)	1.9(5)	-3.3(5)	-0.6(5)
C9	17.6(6)	21.5(6)	22.4(6)	-1.9(5)	-0.4(5)	1.7(5)
C10	22.8(7)	21.3(6)	22.3(6)	-1.6(5)	-0.7(5)	-1.7(5)
C11	17.3(6)	22.5(6)	17.7(6)	1.1(5)	1.0(5)	-2.2(5)
C12	16.3(6)	23.1(6)	26.5(6)	1.4(5)	-1.4(5)	1.2(5)
C13	18.5(6)	20.4(6)	24.6(6)	-0.5(5)	-0.5(5)	1.7(5)
C14	17.0(6)	20.9(6)	20.1(6)	1.8(5)	-1.4(5)	0.4(5)
C15	19.0(6)	27.0(7)	17.4(6)	2.9(5)	0.9(5)	-3.3(5)
C16	24.6(7)	27.6(7)	29.1(7)	0.3(5)	-4.9(5)	-7.6(5)

Table S6. Bond Lengths for **5u**.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O1	C15	1.2215(16)	C8	C9	1.5322(18)
C1	C2	1.3986(17)	C9	C10	1.5036(17)
C1	C6	1.4012(17)	C9	C14	1.5381(17)
C1	C14	1.5214(16)	C10	C11	1.3406(18)
C2	C3	1.3845(18)	C11	C12	1.5077(17)
C3	C4	1.3902(18)	C11	C15	1.4859(17)
C4	C5	1.3858(19)	C12	C13	1.5281(17)
C5	C6	1.3984(17)	C13	C14	1.5355(17)
C6	C7	1.5116(17)	C15	C16	1.5112(18)

C7	C8	1.5223(17)

Table S7. Bond Angles for **5u**.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C2	C1	C6	119.09(11)	C10	C9	C14	111.74(10)
C2	C1	C14	118.29(11)	C11	C10	C9	125.33(12)
C6	C1	C14	122.62(11)	C10	C11	C12	121.50(11)
C3	C2	C1	121.35(12)	C10	C11	C15	121.30(12)
C2	C3	C4	119.46(12)	C15	C11	C12	117.14(11)
C5	C4	C3	119.89(12)	C11	C12	C13	110.77(10)
C4	C5	C6	121.08(12)	C12	C13	C14	110.26(10)
C1	C6	C7	121.14(11)	C1	C14	C9	113.20(10)
C5	C6	C1	119.12(11)	C1	C14	C13	111.14(10)
C5	C6	C7	119.71(11)	C13	C14	C9	109.52(10)
C6	C7	C8	112.44(10)	O1	C15	C11	120.11(12)
C7	C8	C9	110.19(11)	O1	C15	C16	120.06(12)
C8	C9	C14	111.52(10)	C11	C15	C16	119.81(11)
C10	C9	C8	111.48(11)				

Table S8. Hydrogen coordinates ($\times10^4$) and isotropic displacement parameters ($Å^2\times10^3$) for **5u**.

Atom	*x*	*y*	*z*	U(eq)
H2	1133.42	4715.67	3947.69	27
H3	-1294.53	4022.1	2910.77	29
H4	-3163.72	5064.4	1805.74	28
H5	-2565.84	6781.54	1734.74	25
H7A	-833.72	8267.97	3184.34	26
H7B	-258.54	8158.82	2015.36	26
H8A	2084.17	8943.15	3159.52	28
H8B	2808.19	7975.84	2594.23	28
H9	1599.05	7952.37	4680.75	25
H10	4471.3	8796.89	4797.37	27
H12A	5750.24	6020.99	5215.06	27

H12B	7015.02	6240.64	4284.42	27
H13A	4177.39	5434.04	3672.75	26
H13B	4481.32	6463.25	3083.38	26
H14	2334.8	6319.07	4868.63	23
H16A	6837.46	9239.62	6156.09	41
H16B	7418.81	9452.9	5004.12	41
H16C	8926.3	9230.53	5927.08	41
H2	1133.42	4715.67	3947.69	27
H3	-1294.53	4022.1	2910.77	29

10.2 X-ray crystallographic data of 5x.[11]

Data intensity of **5x** was collected using a XtaLAB AFC12 (RINC): Kappa dual home/near diffractometer at 100.00(10) K. Data collection and reduction were done by using Olex2 and the structure was solved with the ShelXS structure solution program using direct methods and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for non-H atoms using SHELX-97. Hydrogen atoms were added at their geometrically idea positions and refined isotropically. Crystal Data for **5x**, $C_{15}H_{16}O_2$ (M =228.28 g/mol): monoclinic, space group Cc (no. 9), a = 8.78830(10) Å, b = 12.6773(2) Å, c = 10.4803(2) Å, β = 99.0970(10)°, V = 1152.94(3) Å^3, Z = 4, T = 100.00(10) K, μ(CuKα) = 0.682 mm^{-1}, *Dcalc* = 1.315 g/cm^3, 10931 reflections measured ($12.36° \leqslant 2\Theta \leqslant 148.886°$), 2186 unique ($R_{int}$ = 0.0431, R_{sigma} = 0.0216) which were used in all calculations. The final R_1 was 0.0314 (I > 2σ(I)) and wR_2 was 0.0845 (all data).

5x

Table S9. Crystal data and structure refinement for **5x**.

Identification code	**5x**
Empirical formula	$C_{15}H_{16}O_2$
Formula weight	228.28
Temperature	100.00(10) K

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[11] Supplementary crystallographic data have been deposited at the Cambridge Crystallographic Data Center (CCDC number: 1917989).

Crystal system, space group	monoclinic, Cc	
Unit cell dimensions	a = 8.78830(10) Å	α = 90 deg
	b = 12.6773(2) Å	β = 99.0970(10) deg
	c = 10.4803(2) Å	γ = 90 deg
Volume	1152.94(3) Å^3	
Z, Calculated density	4, 1.315 Mg/m^3	
ρcalcg/cm^3	1.315	
μ/mm^{-1}	0.682	
F(000)	488.0	
Crystal size	0.32 × 0.26 × 0.22 mm^3	
Radiation	CuKα (λ = 1.54184)	
2Θ range for data collection/°	12.36 to 148.886	
Index ranges	$-10 \leqslant h \leqslant 10$, $-15 \leqslant k \leqslant 15$, $-12 \leqslant l \leqslant 12$	
Reflections collected	10931	
Independent reflections	2186 [R_{int} = 0.0431, R_{sigma} = 0.0216]	
Data/restraints/parameters	2186/2/156	
Goodness-of-fit on F2	1.046	
Final R indexes [I>=2σ (I)]	R_1 = 0.0314, wR_2 = 0.0844	
Final R indexes [all data]	R_1 = 0.0314, wR_2 = 0.0845	
Largest diff. peak/hole / e Å^{-3}	0.18/-0.20	
Flack parameter	0.14(12)	

Table S10. Fractional Atomic coordinates (×10^4) and equivalent isotropic displacement parameters (A^2×10^3) for **5x**. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

Atom	***x***	***y***	***z***	**U(eq)**
O1	3310.3(16)	4325.4(10)	3714.8(13)	22.0(3)
O2	5677.9(19)	9454.2(11)	5834.0(15)	27.5(4)
C1	4423(2)	3824.0(15)	4565(2)	19.4(4)
C13	3252(2)	5456.2(14)	3842(2)	22.0(4)
C7	5875(2)	5530.2(14)	5179.8(19)	18.5(4)
C12	4849(2)	5950.8(14)	3968.7(19)	19.9(4)
C11	4746(2)	7133.7(15)	3965.8(19)	20.4(4)
C10	5222(2)	7747.8(14)	4989(2)	19.1(4)

C5	6751(2)	3744.7(16)	6100(2)	21.3(4)
C3	5349(2)	2156.3(14)	5485(2)	23.2(4)
C14	5173(2)	8919.7(13)	4893(2)	19.9(4)
C9	5871(3)	7295.5(14)	6292(2)	21.2(4)
C6	5675(2)	4347.5(14)	5286(2)	19.4(4)
C8	5517(2)	6120.3(15)	6374.3(19)	20.0(4)
C2	4269(2)	2730.4(15)	4661(2)	22.3(4)
C4	6593(2)	2662.6(16)	6211(2)	23.5(4)
C15	4498(3)	9436.9(15)	3637(2)	25.3(5)

Table S11. Anisotropic displacement parameters ($A^2\times10^3$) for **5x**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$

Atom	**U_{11}**	**U_{22}**	**U_{33}**	**U_{23}**	**U_{13}**	**U_{12}**
O1	26.3(7)	13.9(7)	24.5(8)	-0.2(5)	-0.1(6)	-1.8(5)
O2	40.5(9)	16.2(7)	25.0(8)	-3.0(5)	2.7(6)	-1.7(6)
C1	21.7(9)	17.5(9)	20.0(9)	-0.4(7)	6.8(8)	0.4(7)
C13	26.2(10)	12.5(9)	25.9(11)	1.3(7)	-0.7(8)	-0.7(7)
C7	17.7(9)	14.4(9)	23.6(10)	0.0(7)	4.1(7)	-0.4(7)
C12	27.1(10)	14.1(9)	19.1(10)	-1.6(7)	5.9(7)	-2.2(7)
C11	24.9(10)	16.2(9)	20.3(10)	1.4(7)	3.7(8)	-2.1(7)
C10	20.3(8)	14.9(8)	22.4(10)	0.9(7)	5.0(8)	-0.1(7)
C5	20.6(9)	19.7(10)	24.2(10)	-3.0(8)	4.7(8)	1.9(7)
C3	32.4(11)	12.5(8)	26.5(10)	-1.0(7)	10.4(9)	1.1(7)
C14	21.1(9)	14.8(8)	24.2(11)	0.1(8)	4.9(7)	0.6(8)
C9	29.3(11)	15.4(9)	18.8(10)	-1.2(7)	3.4(8)	-0.9(7)
C6	22.8(10)	14.3(8)	22.4(10)	-0.8(7)	7.8(8)	1.5(7)
C8	23.3(10)	16.1(9)	20.0(10)	0.1(7)	1.8(8)	0.0(7)
C2	27.1(11)	17.9(10)	22.6(10)	-1.6(7)	6.4(8)	-2.6(8)
C4	27.9(10)	16.9(9)	26.1(11)	1.0(8)	5.5(8)	7.0(8)
C15	31.7(11)	14.3(8)	28.2(11)	2.1(8)	-0.5(8)	-0.6(7)

Table S12. Bond lengths [Å] for **5x**.

Atom	**Atom**	**Length/Å**	**Atom**	**Atom**	**Length/Å**
O1	C1	1.371(2)	C11	C10	1.338(3)

Atom	**Atom**	**Length/Å**	**Atom**	**Atom**	**Length/Å**
O1	C13	1.441(2)	C10	C14	1.489(2)
O2	C14	1.221(2)	C10	C9	1.507(3)
C1	C6	1.399(3)	C5	C6	1.397(3)
C1	C2	1.398(3)	C5	C4	1.385(3)
C13	C12	1.523(3)	C3	C2	1.385(3)
C7	C12	1.532(3)	C3	C4	1.386(3)
C7	C6	1.516(2)	C14	C15	1.506(3)
C7	C8	1.533(3)	C9	C8	1.527(2)
C12	C11	1.502(2)			

Table S13. Bond Angles for **5x**.

Atom	**Atom**	**Atom**	**Angle/˚**	**Atom**	**Atom**	**Atom**	**Angle/˚**
C1	O1	C13	115.67(15)	C14	C10	C9	116.35(15)
O1	C1	C6	123.38(16)	C4	C5	C6	121.73(18)
O1	C1	C2	116.04(17)	C2	C3	C4	120.14(17)
C2	C1	C6	120.57(18)	O2	C14	C10	119.72(17)
O1	C13	C12	111.83(16)	O2	C14	C15	120.47(16)
C12	C7	C8	109.70(15)	C10	C14	C15	119.80(16)
C6	C7	C12	110.20(15)	C10	C9	C8	111.78(15)
C6	C7	C8	112.44(16)	C1	C6	C7	121.32(16)
C13	C12	C7	110.03(16)	C5	C6	C1	117.95(16)
C11	C12	C13	110.87(16)	C5	C6	C7	120.73(17)
C11	C12	C7	112.12(15)	C9	C8	C7	111.11(16)
C10	C11	C12	124.62(17)	C3	C2	C1	120.07(19)
C11	C10	C14	121.59(17)	C5	C4	C3	119.54(18)
C11	C10	C9	122.04(16)				

Table S14. Hydrogen coordinates (×10^4) and isotropic displacement parameters (A^2×10^3) for **5x**.

Atom	***x***	***y***	***z***	**U(eq)**
H13A	2793.57	5632.4	4597.55	26

H13B	2602.42	5748.27	3090.34	26
H7	6950.44	5670.62	5094.49	22
H12	5308.7	5736.77	3215.84	24
H11	4320.99	7462.37	3198.32	25
H5	7594.52	4078.47	6581.18	26
H3	5240.02	1429.68	5552.53	28
H9A	5437.39	7669.69	6957.65	25
H9B	6978.37	7398.99	6450.9	25
H8A	4438.08	6027.88	6444.89	24
H8B	6128.1	5825.2	7143.29	24
H2	3439.25	2388.13	4171.43	27
H4	7317.3	2278.41	6767.13	28
H15A	4582.71	10189.16	3726.54	38
H15B	3431.91	9244.68	3418.11	38
H15C	5048.14	9206.68	2965.7	38

10.3 X-ray crystallographic data of 5ab.[12]

Data intensity of **5ab** was collected using a XtaLAB AFC12 (RINC): Kappa dual home/near diffractometer. The X-ray condition of was 50 kV × 30 mA. Data collection and reduction were done by using the Bruker ApexII software package. The structure was solved by direct methods, and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for non-hydrogen atoms using SHELX-97. Hydrogen atoms were added at their geometrically idea positions and refined isotropically. Crystal data for **5ab**: $C_{25}H_{32}O_2$, (M =364.50 g/mol): monoclinic, space group P21 (no. 4), a = 12.1960(5) Å, b = 6.2725(2) Å, c = 13.2067(7) Å, β = 97.530(4)°, V = 1001.59(7) Å3, Z = 2, T = 100.00(10) K, μ(CuKα) = 0.574 mm^{-1}, *Dcalc* = 1.209 g/cm^3, 21715 reflections measured (9.284° ≤ 2Θ ≤ 150.772°), 3981 unique (R_{int} = 0.0843, R_{sigma} = 0.0419) which were used in all calculations. The final R_1 was 0.0858 (I > 2σ(I)) and wR_2 was 0.2587 (all data).

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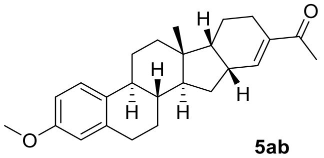
[12] Supplementary crystallographic data have been deposited at the Cambridge Crystallographic Data Center (CCDC number: 2076484).



5ab

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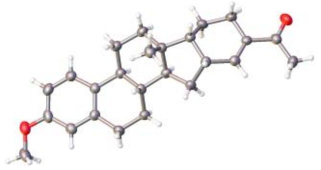


Table S15. Crystal data and structure refinement for **5ab**.

Identification code	**5ab**	
Empirical formula	$C_{25}H_{32}O_2$	
Formula weight	364.50	
Temperature/K	100.00(10)	
Crystal system	monoclinic	
Space group	P21	
Unit cell dimensions	a = 12.1960(5) Å	α = 90 deg
	b = 6.2725(2) Å	β = 97.530(4) deg
	c = 13.2067(7) Å	γ = 90 deg
Volume/Å^3	1001.59(7)	
Z	2	
ρcalcg/cm^3	1.209	
μ/mm^{-1}	0.574	
F(000)	396.0	
Crystal size/mm^3	0.46 × 0.44 × 0.28	
Radiation	CuKα (λ = 1.54184)	
2Θ range for data collection/°	9.284 to 150.772	
Index ranges	$-15 \leqslant h \leqslant 15, -7 \leqslant k \leqslant 7, -16 \leqslant l \leqslant 15$	
Reflections collected	21715	
Independent reflections	3981 [R_{int} = 0.0843, R_{sigma} = 0.0419]	
Data/restraints/parameters	3981/1/247	
Goodness-of-fit on F2	1.083	
Final R indexes [I>=2σ (I)]	R_1 = 0.0858, wR_2 = 0.2572	
Final R indexes [all data]	R_1 = 0.0874, wR_2 = 0.2587	
Largest diff. peak/hole / e Å^{-3}	0.37/-0.33	
Flack parameter	0.2(3)	

Table S16. Fractional Atomic Coordinates ($\times10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2\times10^3$) for **5ab**. U(eq) is defined as 1/3 of of the trace of the orthogonalised Uij tensor.

Atom	***x***	***y***	***z***	**U(eq)**
O1	529(4)	6542(9)	7421(4)	53.2(13)
O2	8025(3)	5303(8)	-177(3)	44.5(11)
C1	1980(4)	3393(9)	4514(4)	31.0(11)
C2	1776(4)	3483(9)	5613(4)	31.5(11)
C3	1319(4)	5130(9)	6032(4)	29.9(11)
C4	1061(5)	7172(9)	5453(4)	37.1(13)
C5	1767(5)	7410(9)	4595(4)	34.9(12)
C6	1648(4)	5456(9)	3897(4)	31.2(11)
C7	2408(4)	5387(9)	3047(4)	30.6(11)
C8	2636(4)	7497(9)	2522(4)	33.0(11)
C9	3538(4)	7256(10)	1826(4)	35.2(12)
C10	4600(4)	6191(8)	2349(4)	30.3(11)
C11	5479(4)	5846(9)	1648(4)	30.7(11)
C12	5592(4)	7223(10)	833(4)	34.8(12)
C13	6415(5)	6994(10)	232(4)	39.2(13)
C14	7190(4)	5338(10)	410(4)	36.0(12)
C15	7073(4)	3913(10)	1195(4)	34.7(12)
C16	6229(4)	4152(9)	1815(4)	31.4(11)
C17	6174(5)	2511(9)	2649(4)	35.6(12)
C18	5453(4)	3234(9)	3451(4)	32.2(11)
C19	4368(4)	4119(8)	2925(4)	30.5(11)
C20	3520(4)	4632(8)	3640(4)	29.3(11)
C21	3201(4)	2883(8)	4362(4)	29.6(11)
C22	1005(4)	5044(10)	7091(4)	36.1(12)
C23	1287(5)	3093(11)	7729(4)	42.7(14)
C24	1913(5)	3769(11)	2249(4)	38.5(13)
C25	8870(5)	3765(14)	55(5)	52.0(18)

Table S17. Anisotropic Displacement Parameters (Å^2×10^3) for **5ab**. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$

Atom	**U_{11}**	**U_{22}**	**U_{33}**	**U_{23}**	**U_{13}**	**U_{12}**
O1	55(3)	64(3)	43(3)	-4(2)	14(2)	21(2)
O2	36(2)	63(3)	35(2)	-1(2)	8.3(16)	-5(2)
C1	35(3)	29(3)	29(3)	-1(2)	4(2)	-3(2)
C2	29(2)	30(3)	35(3)	-1(2)	4(2)	-2.6(19)
C3	26(2)	32(3)	31(3)	2(2)	2.1(18)	3(2)
C4	39(3)	36(3)	37(3)	4(2)	8(2)	15(2)
C5	34(3)	32(3)	40(3)	1(2)	8(2)	9(2)
C6	29(2)	34(3)	30(3)	1(2)	1.5(19)	3(2)
C7	29(2)	34(3)	28(3)	-2(2)	1.6(19)	2(2)
C8	38(3)	32(3)	29(3)	4(2)	1(2)	2(2)
C9	37(3)	36(3)	32(3)	5(2)	2(2)	3(2)
C10	31(2)	31(3)	28(3)	-1(2)	2(2)	-1(2)
C11	29(2)	34(3)	29(3)	-2(2)	1(2)	-3(2)
C12	32(3)	40(3)	32(3)	2(2)	3(2)	1(2)
C13	38(3)	48(3)	30(3)	7(2)	0(2)	-4(3)
C14	32(2)	52(3)	25(3)	-7(2)	5(2)	-10(2)
C15	30(3)	40(3)	33(3)	-7(2)	1(2)	-1(2)
C16	31(2)	32(3)	31(3)	0(2)	2.6(19)	-4(2)
C17	34(3)	36(3)	37(3)	-2(2)	7(2)	5(2)
C18	34(3)	30(3)	33(3)	2(2)	5(2)	2(2)
C19	31(2)	28(2)	32(3)	-3(2)	5(2)	-1.5(19)
C20	31(2)	26(2)	30(3)	0(2)	1.5(19)	-1.6(19)
C21	32(3)	24(2)	33(3)	0.4(19)	6(2)	0.0(19)
C22	33(3)	43(3)	32(3)	-4(2)	4(2)	6(2)
C23	46(3)	51(3)	31(3)	8(3)	8(2)	3(3)
C24	32(3)	49(3)	34(3)	-4(2)	3(2)	-3(2)
C25	33(3)	83(5)	42(3)	-5(3)	12(2)	3(3)

Table S18. Bond Lengths for **5ab**.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O1	C22	1.215(7)		C9	C10
O2	C14	1.359(6)		C10	C11
O2	C25	1.416(9)		C10	C19
C1	C2	1.506(7)		C11	C12
C1	C6	1.554(7)		C11	C16
C1	C21	1.561(7)		C12	C13
C2	C3	1.329(7)		C13	C14
C3	C4	1.504(7)		C14	C15
C3	C22	1.498(7)		C15	C16
C4	C5	1.517(8)		C16	C17
C5	C6	1.529(8)		C17	C18
C6	C7	1.546(7)		C18	C19
C7	C8	1.537(7)		C19	C20
C7	C20	1.548(7)		C20	C21
C7	C24	1.529(8)		C22	C23
C8	C9	1.531(7)			

Table S19. Bond Angles for **5ab**.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C14	O2	C25	117.8(5)		C12	C11	C10
C2	C1	C6	114.2(4)		C16	C11	C10
C2	C1	C21	114.4(4)		C16	C11	C12
C6	C1	C21	106.9(4)		C13	C12	C11
C3	C2	C1	124.2(5)		C12	C13	C14
C2	C3	C4	121.4(5)		O2	C14	C13
C2	C3	C22	122.2(5)		O2	C14	C15
C4	C3	C22	116.3(5)		C15	C14	C13
C3	C4	C5	111.2(4)		C14	C15	C16
C4	C5	C6	110.7(5)		C11	C16	C15
C5	C6	C1	110.7(4)		C11	C16	C17
C5	C6	C7	116.2(4)		C15	C16	C17

C7	C6	C1	102.5(4)	C16	C17	C18
C6	C7	C20	102.1(4)	C19	C18	C17
C8	C7	C6	117.7(4)	C18	C19	C10
C8	C7	C20	107.0(4)	C18	C19	C20
C24	C7	C6	107.4(4)	C20	C19	C10
C24	C7	C8	109.8(4)	C19	C20	C7
C24	C7	C20	112.7(4)	C19	C20	C21
C9	C8	C7	111.9(4)	C21	C20	C7
C8	C9	C10	113.7(4)	C20	C21	C1
C9	C10	C19	112.8(4)	O1	C22	C3
C11	C10	C9	114.1(4)	O1	C22	C23
C11	C10	C19	111.6(4)	C23	C22	C3

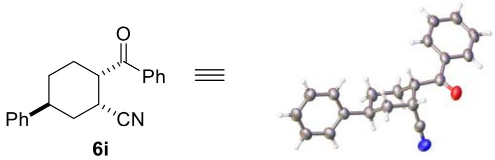
Table S20. Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for **5ab**.

Atom	***x***	***y***	***z***	**U(eq)**
H1	1520.85	2237.31	4189.1	37
H2	1983.63	2310.5	6024.71	38
H4A	1194.03	8370.26	5917.34	45
H4B	285.67	7187.31	5170.89	45
H5A	2535.63	7580.71	4881.08	42
H5B	1541.48	8675.71	4199.74	42
H6	876.27	5334.98	3584.24	37
H8A	1960.4	7989.74	2121.12	40
H8B	2863.18	8564.92	3038.54	40
H9A	3721.91	8655.37	1585.19	42
H9B	3247.67	6415.2	1234.33	42
H10	4921.89	7195.59	2873.83	36
H12	5088.79	8333.39	696.43	42
H13	6462.66	7945.67	-300.59	47
H15	7564.14	2777.26	1311.17	42
H17A	6916.36	2227.24	2982.18	43
H17B	5879.05	1190.24	2342.27	43

H18A	5309.99	2036.06	3879.25	39
H18B	5837.98	4321.9	3882.5	39
H19	4043.93	3069.2	2423.07	37
H20	3816.22	5826.95	4069.53	35
H21A	3679.18	2925.63	5008.76	36
H21B	3253.73	1483.54	4059.12	36
H23A	2074.68	2901.54	7831.65	64
H23B	943.4	1868.57	7385.41	64
H23C	1023.57	3260.87	8378.16	64
H24A	1880.46	2394.43	2563.64	58
H24B	2368.04	3686.48	1709.85	58
H24C	1181.01	4210.26	1973.82	58
H25A	9409.48	3921.54	-408.01	78
H25B	8554.65	2361.81	-13.67	78
H25C	9218.11	3969.6	742.63	78

10.4 X-ray crystallographic data of 6i.[13]

Data intensity of **6i** was collected using a XtaLAB AFC12 (RINC): Kappa dual home/near diffractometer. The X-ray condition of was 50 kV × 30 mA. Data collection and reduction were done by using the Bruker ApexII software package. The structure was solved by direct methods, and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for non-hydrogen atoms using SHELX-97. Hydrogen atoms were added at their geometrically idea positions and refined isotropically. Crystal data for **6i**: $C_{20}H_{17}NO$ (M =287.35 g/mol): orthorhombic, space group P212121 (no. 19), a = 6.55952(19) Å, b = 7.19955(14) Å, c = 33.3740(6) Å, V = 1576.11(6) Å^3, Z = 4, T = 100.00(10) K, μ(CuKα) = 0.580 mm^{-1}, *Dcalc* = 1.211 g/cm^3, 32998 reflections measured (13.394° ≤ 2Θ ≤ 134.15°), 2806 unique (R_{int} = 0.0588, R_{sigma} = 0.0246) which were used in all calculations. The final R_1 was 0.0392 (I > 2σ(I)) and wR_2 was 0.1052 (all data).



6i

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[13] Supplementary crystallographic data have been deposited at the Cambridge Crystallographic Data Center (CCDC number: 2083519).

Table S21. Crystal data and structure refinement for **6i**

Identification code	**6i**	
Empirical formula	$C_{20}H_{17}NO$	
Formula weight	287.35	
Temperature/K	100.00(10)	
Crystal system	orthorhombic	
Space group	P212121	
Unit cell dimensions	a = 6.55952(19) Å	α = 90 deg
	b = 7.19955(14) Å	β = 90 deg
	c = 33.3740(6) Å	γ = 90 deg
Volume/Å^3	1576.11(6)	
Z	4	
ρcalcg/cm^3	1.211	
μ/mm^{-1}	0.580	
F(000)	608.0	
Crystal size/mm^3	0.3 × 0.25 × 0.25	
Radiation	CuKα (λ = 1.54184)	
2Θ range for data collection/°	13.394 to 134.15	
Index ranges	$-7 \leq h \leq 7$, $-8 \leq k \leq 8$, $-39 \leq l \leq 39$	
Reflections collected	32998	
Independent reflections	2806 [R_{int} = 0.0588, R_{sigma} = 0.0246]	
Data/restraints/parameters	2806/0/217	
Goodness-of-fit on F2	1.078	
Final R indexes [I>=2σ (I)]	R_1 = 0.0392, wR_2 = 0.1032	
Final R indexes [all data]	R_1 = 0.0425, wR_2 = 0.1052	
Largest diff. peak/hole / e Å^{-3}	0.32/-0.13	
Flack parameter	0.08(19)	

Table S22. Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for **6i**. U(eq) is defined as 1/3 of of the trace of the orthogonalised Uij tensor.

Atom	***x***	***y***	***z***	**U(eq)**
C1	-7470(5)	-5160(4)	-2194.1(7)	45.6(6)

C01	-3755(9)	458(7)	-1449.4(16)	42.0(12)
C02	-4945(10)	-2173(6)	-1040.5(13)	43.9(13)
C2	-8786(5)	-6609(4)	-2277.7(7)	52.4(7)
C3	-10700(5)	-6671(4)	-2104.2(8)	52.8(7)
C4	-11311(5)	-5264(4)	-1849.8(7)	47.6(6)
C5	-10017(4)	-3800(4)	-1765.2(7)	42.8(6)
C6	-8075(4)	-3736(3)	-1934.8(7)	40.3(6)
C7	-6616(4)	-2172(4)	-1859.7(7)	42.6(6)
C8	-7128(4)	-719(3)	-1544.8(7)	41.2(6)
C9	-5732(4)	979(4)	-1564.8(8)	44.5(6)
C10	-6479(5)	2476(3)	-1273.6(7)	44.9(6)
C11	-6585(4)	1770(3)	-840.2(7)	43.1(6)
C12	-7821(5)	-28(3)	-816.3(7)	44.5(6)
C13	-7114(5)	-1534(3)	-1113.7(7)	44.6(6)
C14	-7447(4)	3224(3)	-558.5(7)	41.2(6)
C15	-9351(4)	4029(4)	-628.6(8)	45.5(6)
C16	-10144(5)	5341(3)	-367.7(8)	48.5(7)
C17	-9065(5)	5869(4)	-32.1(9)	49.4(7)
C18	-7198(5)	5071(4)	43.3(8)	50.6(7)
C19	-6390(4)	3758(3)	-218.0(8)	45.7(6)
N01	-2096(8)	126(8)	-1342.7(17)	65.2(14)
N02	-3265(9)	-2643(7)	-988.0(13)	60.8(14)
O1	-5032(3)	-2053(3)	-2049.7(6)	55.3(5)

Table S23. Anisotropic Displacement Parameters (Å^2×10^3) for **6i**. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	**U_{11}**	**U_{22}**	**U_{33}**	**U_{23}**	**U_{13}**	**U_{12}**
C1	60.5(17)	46.0(14)	30.3(11)	2.1(11)	-2.3(11)	13.4(13)
C01	45(3)	30(2)	52(3)	-2(2)	10(2)	-1(2)
C02	79(4)	30(2)	23(2)	2.3(17)	-1(2)	13(3)
C2	78(2)	46.1(15)	33.1(12)	-9.1(11)	-7.4(13)	12.3(15)
C3	68.2(19)	50.9(15)	39.2(13)	-5.9(12)	-10.6(13)	-0.9(15)
C4	53.2(17)	50.5(15)	39.2(12)	-1.5(12)	-6.1(12)	2.3(13)

C5	53.2(15)	41.8(13)	33.6(12)	-2.4(10)	-2.9(11)	13.0(12)
C6	54.8(16)	37.3(13)	28.7(11)	1.9(9)	-4.5(10)	11.4(11)
C7	51.2(16)	42.2(13)	34.6(12)	4.9(10)	0.8(11)	12.4(11)
C8	50.1(16)	36.0(12)	37.4(12)	2.5(10)	-0.1(11)	9.7(11)
C9	57.8(17)	36.8(12)	38.9(12)	5.4(10)	3.9(12)	5.4(12)
C10	57.8(17)	31.4(12)	45.6(14)	3.4(11)	4.4(12)	5.5(12)
C11	54.6(16)	33.8(12)	40.9(12)	1.3(10)	-4.5(11)	6.3(12)
C12	68.0(18)	30.6(12)	34.9(11)	2.4(10)	1.4(11)	3.3(12)
C13	66.8(18)	33.0(13)	33.8(12)	2.5(10)	-0.6(11)	8.7(12)
C14	53.1(16)	30.2(11)	40.4(12)	2.3(10)	-0.3(11)	1.1(12)
C15	53.8(17)	37.8(13)	45.0(13)	-0.7(11)	-3.5(12)	6.4(12)
C16	51.6(16)	34.9(13)	58.9(15)	1.0(12)	5.7(13)	5.0(12)
C17	59.6(18)	30.8(12)	57.7(16)	-7.8(12)	8.3(13)	-6.7(12)
C18	58.6(19)	38.0(13)	55.3(15)	-10.8(13)	-2.1(13)	-8.8(13)
C19	50.9(16)	34.6(13)	51.6(14)	-2.3(11)	-2.8(12)	-1.9(12)
N01	45(3)	62(3)	89(4)	-21(3)	2(3)	6(3)
N02	81(4)	62(3)	39(2)	-1(2)	-6(2)	33(3)
O1	60.9(12)	54.6(11)	50.3(10)	-0.6(8)	13.8(10)	7.7(10)

Table S24. Bond Lengths for **6i**.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C1	C2	1.383(4)		C8	C9
C1	C6	1.399(3)		C8	C13
C01	C9	1.403(6)		C9	C10
C01	N01	1.170(7)		C10	C11
C02	C13	1.516(7)		C11	C12
C02	N02	1.166(7)		C11	C14
C2	C3	1.383(4)		C12	C13
C3	C4	1.381(4)		C14	C15
C4	C5	1.383(4)		C14	C19
C5	C6	1.395(4)		C15	C16
C6	C7	1.499(4)		C16	C17
C7	C8	1.520(3)		C17	C18
C7	O1	1.220(3)		C18	C19

Table S25. Bond Angles for **6i**.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C2	C1	C6	120.0(3)	C8	C9	C10	110.1(2)
N01	C01	C9	175.9(5)	C9	C10	C11	112.3(2)
N02	C02	C13	179.0(5)	C12	C11	C10	110.7(2)
C1	C2	C3	120.4(3)	C14	C11	C10	111.84(19)
C4	C3	C2	119.8(3)	C14	C11	C12	110.8(2)
C3	C4	C5	120.4(3)	C11	C12	C13	113.7(2)
C4	C5	C6	120.2(2)	C02	C13	C8	105.6(3)
C1	C6	C7	118.2(2)	C02	C13	C12	113.1(3)
C5	C6	C1	119.1(3)	C12	C13	C8	109.18(19)
C5	C6	C7	122.7(2)	C15	C14	C11	121.1(2)
C6	C7	C8	119.4(2)	C19	C14	C11	120.9(2)
O1	C7	C6	120.6(2)	C19	C14	C15	118.0(2)
O1	C7	C8	119.9(3)	C16	C15	C14	120.9(3)
C7	C8	C9	112.8(2)	C17	C16	C15	120.4(3)
C7	C8	C13	112.27(19)	C18	C17	C16	119.4(3)
C9	C8	C13	109.8(2)	C17	C18	C19	120.5(3)
C01	C9	C8	109.1(3)	C14	C19	C18	120.8(3)
C01	C9	C10	108.1(3)				

Table S26. Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for **6i**.

Atom	*x*	*y*	*z*	U(eq)
H1	-6182.2	-5131.88	-2310.43	55
H2	-8382.31	-7548.59	-2451.67	63
H3	-11572.83	-7657.01	-2158.73	63
H4	-12601.9	-5302.08	-1734.7	57
H5	-10442.67	-2854.7	-1594.68	51
H8	-8517.56	-287.78	-1598.94	49
H9	-5710.54	1473.91	-1838.3	53
H10A	-7822.23	2891.78	-1355.88	54
H10B	-5565.92	3534.43	-1285.81	54

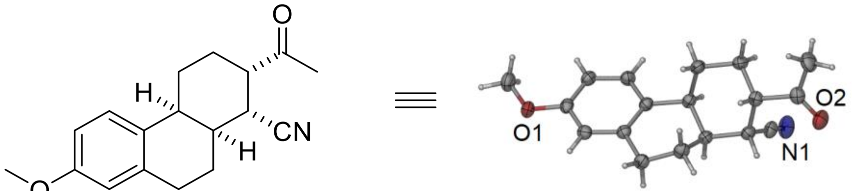
H11	-5191.81	1490.61	-753.01	52
H12A	-9242.8	255.24	-868.01	53
H12B	-7725.95	-518.74	-546.28	53
H15	-10096.31	3679.71	-853.33	55
H16	-11412.12	5867.24	-419.31	58
H17	-9594.3	6756.26	141.77	59
H18	-6469.58	5413.08	270.77	61
H19	-5124.77	3232.42	-163.48	55

Table S27. Atomic Occupancy for **6i**.

Atom	***Occupancy***	**Atom**	***Occupancy***	**Atom**	***Occupancy***
C01	0.5	C02	0.5	N01	0.5
N02	0.5				

10.5 X-ray crystallographic data of 6q.[14]

Data intensity of **6q** was collected using a XtaLAB AFC12 (RINC): Kappa dual home/near diffractometer. The X-ray condition of was 50 kV × 30 mA. Data collection and reduction were done by using the Bruker ApexII software package. The structure was solved by direct methods, and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for non-hydrogen atoms using SHELX-97. Hydrogen atoms were added at their geometrically idea positions and refined isotropically. Crystal data for **6q**: $C_{18}H_{21}NO_2$ (M =283.36 g/mol): triclinic, space group P-1 (no. 2), a = 7.2466(2) Å, b = 8.3731(2) Å, c = 12.6472(3) Å, α = 95.047(2)°, β = 96.626(2)°, γ = 94.254(2)°, V = 756.54(3) Å^3, Z = 2, T = 170.00(10) K, μ(CuKα) = 0.638 mm^{-1}, *Dcalc* = 1.244 g/cm^3, 14785 reflections measured (7.07° ≤ 2Θ ≤ 134.126°), 2694 unique (R_{int} = 0.0241, R_{sigma} = 0.0162) which were used in all calculations. The final R_1 was 0.0406 (I > 2σ(I)) and wR_2 was 0.1077 (all data).



6q

Table S28. Crystal data and structure refinement for **6q**.

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[14] Supplementary crystallographic data have been deposited at the Cambridge Crystallographic Data Center (CCDC number: 2101568).

Identification code	**6q**	
Empirical formula	$C_{18}H_{21}NO_2$	
Formula weight	283.36	
Temperature/K	170.00(10)	
Crystal system	triclinic	
Space group	P-1	
Unit cell dimensions	a = 7.2466(2) Å	α = 95.047(2) deg
	b = 8.3731(2) Å	β = 96.626(2) deg
	c = 12.6472(3) Å	γ = 94.254(2) deg
Volume/Å^3	756.54(3)	
Z	2	
ρcalcg/cm^3	1.244	
μ/mm^{-1}	0.638	
F(000)	304.0	
Crystal size/mm^3	0.32 × 0.26 × 0.22	
Radiation	CuKα (λ = 1.54184)	
2Θ range for data collection/°	7.07 to 134.126	
Index ranges	$-8 \leqslant h \leqslant 8, -9 \leqslant k \leqslant 9, -15 \leqslant l \leqslant 15$	
Reflections collected	14785	
Independent reflections	2694 [R_{int} = 0.0241, R_{sigma} = 0.0162]	
Data/restraints/parameters	2694/0/192	
Goodness-of-fit on F^2	1.033	
Final R indexes [I>=2σ (I)]	R_1 = 0.0406, wR_2 = 0.1064	
Final R indexes [all data]	R_1 = 0.0423, wR_2 = 0.1077	
Largest diff. peak/hole / e Å^{-3}	0.28/-0.20	

Table S29. Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for **6q**. U(eq) is defined as 1/3 of of the trace of the orthogonalised Uij tensor.

Atom	***x***	***y***	***z***	**U(eq)**
O1	4767.4(14)	1314.5(12)	2068.7(7)	41.1(3)
O2	2790.3(16)	4649.4(13)	10110.0(8)	49.6(3)
N1	931(2)	1011.7(16)	8631.4(10)	53.1(4)
C1	5010.5(17)	2224.0(15)	4945.3(10)	30.5(3)

C2	5467.0(18)	1889.0(15)	3916.2(10)	33.4(3)
C3	4115.0(19)	1614.4(15)	3038.5(10)	32.9(3)
C4	2243.4(19)	1630.1(17)	3181.0(10)	36.9(3)
C5	1789.6(19)	1940.9(17)	4211.9(10)	36.5(3)
C6	3131.2(18)	2255.6(15)	5102.5(10)	30.8(3)
C7	2526.0(17)	2674.7(15)	6198.6(10)	30.0(3)
C8	1836.7(19)	4360.0(16)	6266.9(10)	35.2(3)
C9	1290(2)	4905.7(17)	7363.4(11)	37.6(3)
C10	2910.4(18)	4808.0(15)	8244.7(10)	32.2(3)
C11	3550.6(18)	3094.4(15)	8205.0(10)	32.5(3)
C12	4096.8(18)	2507.3(15)	7098.2(10)	31.5(3)
C13	5923.7(18)	3360.2(17)	6874.8(11)	38.0(3)
C14	6553.4(18)	2515.7(18)	5871.7(11)	37.1(3)
C15	2481.9(19)	5421.1(16)	9350.6(11)	36.8(3)
C16	1700(3)	7014.1(19)	9470.2(13)	53.1(4)
C17	2076(2)	1939.6(16)	8469.9(10)	37.3(3)
C18	3430(2)	1102(2)	1141.2(11)	49.5(4)

Table S30. Anisotropic Displacement Parameters (Å2×103) for **6q**. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	**U_{11}**	**U_{22}**	**U_{33}**	**U_{23}**	**U_{13}**	**U_{12}**
O1	47.0(6)	48.4(6)	29.9(5)	7.3(4)	8.9(4)	6.2(4)
O2	65.7(7)	48.5(6)	32.9(5)	0.7(4)	4.0(5)	0.5(5)
N1	68.4(9)	46.0(7)	43.4(7)	1.6(6)	15.5(6)	-16.2(7)
C1	31.1(7)	27.3(6)	33.0(7)	7.6(5)	2.6(5)	-1.5(5)
C2	32.3(7)	31.3(7)	37.7(7)	8.5(5)	7.0(5)	-0.4(5)
C3	42.1(7)	27.4(6)	30.6(7)	7.1(5)	7.8(5)	1.9(5)
C4	38.1(7)	41.2(7)	29.9(7)	3.1(5)	-1.6(5)	2.9(6)
C5	30.3(7)	45.2(8)	33.0(7)	2.6(6)	1.2(5)	2.1(6)
C6	31.3(7)	30.5(6)	30.0(7)	5.5(5)	1.5(5)	-0.7(5)
C7	27.0(6)	33.4(7)	28.3(6)	4.7(5)	0.7(5)	-3.4(5)
C8	34.5(7)	39.0(7)	32.4(7)	9.0(5)	0.7(5)	4.5(6)
C9	38.0(7)	39.0(7)	36.7(7)	5.7(6)	3.0(6)	8.5(6)

C10	32.2(7)	30.1(7)	33.0(7)	2.2(5)	3.6(5)	-4.6(5)
C11	32.0(7)	35.3(7)	28.3(6)	2.9(5)	-2.3(5)	0.1(5)
C12	32.5(7)	30.5(6)	30.7(7)	4.8(5)	0.4(5)	1.1(5)
C13	30.2(7)	45.2(8)	36.1(7)	3.8(6)	-2.1(5)	-4.0(6)
C14	27.3(7)	45.9(8)	37.6(7)	8.1(6)	2.6(5)	-2.2(6)
C15	33.6(7)	36.7(7)	37.4(7)	-2.2(6)	4.4(5)	-8.4(6)
C16	65.2(11)	46.1(9)	49.6(9)	-3.3(7)	17.4(8)	7.6(8)
C17	48.0(8)	36.0(7)	26.6(6)	1.3(5)	3.7(6)	-1.3(6)
C18	55.6(9)	60.5(10)	30.9(7)	-1.4(7)	6.3(6)	0.6(8)

Table S31. Bond Lengths for **6q**.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O1	C3	1.3742(15)	C7	C8	1.5302(18)
O1	C18	1.4219(17)	C7	C12	1.5370(17)
O2	C15	1.2128(17)	C8	C9	1.5268(18)
N1	C17	1.1425(18)	C9	C10	1.5351(18)
C1	C2	1.3896(18)	C10	C11	1.5388(18)
C1	C6	1.4006(18)	C10	C15	1.5211(18)
C1	C14	1.5130(17)	C11	C12	1.5471(17)
C2	C3	1.3840(19)	C11	C17	1.4749(19)
C3	C4	1.390(2)	C12	C13	1.5245(18)
C4	C5	1.3884(19)	C13	C14	1.5302(19)
C5	C6	1.3929(18)	C15	C16	1.490(2)
C6	C7	1.5214(17)			

Table S32. Bond Angles for **6q**.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
C3	O1	C18	117.28(11)	C8	C9	C10	110.71(11)
C2	C1	C6	119.21(12)	C9	C10	C11	110.36(10)
C2	C1	C14	119.22(11)	C15	C10	C9	113.09(11)
C6	C1	C14	121.57(11)	C15	C10	C11	112.60(11)
C3	C2	C1	121.75(12)	C10	C11	C12	112.64(10)
O1	C3	C2	115.49(12)	C17	C11	C10	110.86(11)

O1	C3	C4	124.72(12)	C17	C11	C12	108.38(10)
C2	C3	C4	119.78(12)	C7	C12	C11	111.30(10)
C5	C4	C3	118.35(12)	C13	C12	C7	110.67(10)
C4	C5	C6	122.72(13)	C13	C12	C11	112.38(10)
C1	C6	C7	122.24(11)	C12	C13	C14	109.37(11)
C5	C6	C1	118.17(12)	C1	C14	C13	112.78(11)
C5	C6	C7	119.56(11)	O2	C15	C10	121.11(13)
C6	C7	C8	110.16(10)	O2	C15	C16	121.27(13)
C6	C7	C12	111.37(10)	C16	C15	C10	117.61(12)
C8	C7	C12	111.93(10)	N1	C17	C11	177.02(14)
C9	C8	C7	113.44(10)				

Table S33. Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for **6q**.

Atom	***x***	***y***	***z***	**U(eq)**
H2	6713.95	1848.16	3814.5	40
H4	1317.74	1437.25	2600.28	44
H5	538.05	1938.92	4311.96	44
H7	1476.28	1907.05	6281.29	36
H8A	2810.87	5121.18	6099.53	42
H8B	766.3	4374.43	5732.24	42
H9A	214.87	4228.76	7500.41	45
H9B	947.75	6004.56	7370.8	45
H10	3956.7	5515.47	8080.38	39
H11	4644.36	3093.46	8738.69	39
H12	4278.13	1359.42	7101.96	38
H13A	6870.6	3337.81	7480.33	46
H13B	5750.4	4475.57	6769.78	46
H14A	7584.86	3168.15	5659.29	44
H14B	6999.59	1492.38	6039.39	44
H16A	386.67	6888.45	9232.08	80
H16B	2306.78	7741.34	9047.33	80
H16C	1904.44	7439.25	10208.18	80

H18A	4051.98	903.7	518.01	74
H18B	2781.69	2055.64	1086.78	74
H18C	2554.63	202.01	1192.55	74

10.6 X-ray crystallographic data of 6r.[15]

Data intensity of **6r** was collected using a XtaLAB AFC12 (RINC): Kappa dual home/near diffractometer. The X-ray condition of was 50 kV × 30 mA. Data collection and reduction were done by using the Bruker ApexII software package. The structure was solved by direct methods, and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for non-hydrogen atoms using SHELX-97. Hydrogen atoms were added at their geometrically idea positions and refined isotropically. Crystal data for **6r**: $C_{17}H_{18}BrNO$ (M =332.23 g/mol): monoclinic, space group P21/c (no. 14), a = 19.7377(2) Å, b = 9.57380(10) Å, c = 7.61300(10) Å, β = 99.0900(10)°, V = 1420.52(3) Å^3, Z = 4, T = 169.99(10) K, μ(CuKα) = 3.891 mm^{-1}, Dcalc = 1.553 g/cm^3, 13942 reflections measured (9.074° ≤ 2Θ ≤ 149.246°), 2860 unique (R_{int} = 0.0396, R_{sigma} = 0.0248) which were used in all calculations. The final R_1 was 0.0283 (I > 2σ(I)) and wR_2 was 0.0740 (all data).

6r

Table S34. Crystal data and structure refinement for **6r**.

Identification code	**6r**	
Empirical formula	$C_{17}H_{18}BrNO$	
Formula weight	332.23	
Temperature/K	169.99(10)	
Crystal system	monoclinic	
Space group	P21/c	
Unit cell dimensions	a = 19.7377(2) Å	α = 90 deg
	b = 9.57380(10) Å	β = 99.0900(10) deg
	c = 7.61300(10) Å	γ = 90 deg
Volume/Å^3	1420.52(3)	
Z	4	

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[15] Supplementary crystallographic data have been deposited at the Cambridge Crystallographic Data Center (CCDC number: 2089395).

ρcalcg/cm^3	1.553
μ/mm^{-1}	3.891
F(000)	680.0
Crystal size/mm^3	0.42 × 0.13 × 0.11
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	9.074 to 149.246
Index ranges	$-24 \leq h \leq 23$, $-11 \leq k \leq 11$, $-6 \leq l \leq 9$
Reflections collected	13942
Independent reflections	2860 [R_{int} = 0.0396, R_{sigma} = 0.0248]
Data/restraints/parameters	2860/0/183
Goodness-of-fit on F^2	1.079
Final R indexes [I>=2σ (I)]	R_1 = 0.0283, wR_2 = 0.0734
Final R indexes [all data]	R_1 = 0.0290, wR_2 = 0.0740
Largest diff. peak/hole / e Å^{-3}	0.52/-0.48

Table S35. Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for **6r**. U(eq) is defined as 1/3 of of the trace of the orthogonalised Uij tensor.

Atom	***x***	***y***	***z***	**U(eq)**
Br1	923.8(2)	-429.7(2)	-336.3(3)	23.70(10)
O1	4454.6(8)	7113.8(18)	4265(2)	39.6(4)
N1	3269.8(10)	5341.5(19)	6341(2)	30.4(4)
C1	1809.0(9)	3483.6(19)	727(2)	17.5(3)
C2	1686.7(9)	2044.3(19)	663(2)	18.9(3)
C3	1076.8(10)	1536(2)	-256(2)	19.3(4)
C4	568.1(9)	2421(2)	-1102(2)	22.1(4)
C5	694.5(9)	3837(2)	-1031(2)	22.2(4)
C6	1312.1(10)	4392.8(19)	-145(2)	19.3(4)
C7	1426.2(10)	5957(2)	-160(3)	23.2(4)
C8	2180.2(10)	6354(2)	373(2)	22.7(4)
C9	2460.7(10)	5582.8(18)	2090(3)	19.2(4)
C10	3161.4(9)	6144.4(18)	3018(2)	18.5(4)
C11	3771.6(9)	5683.7(19)	2114(2)	18.9(4)
C12	3777.6(9)	4093(2)	1882(3)	21.5(4)

C13	3096.8(9)	3580.7(19)	852(2)	21.1(4)
C14	2485.5(9)	4001.4(18)	1758(2)	17.1(3)
C15	4448.0(10)	6271(2)	3076(3)	23.5(4)
C16	5096.5(10)	5784(2)	2494(3)	29.2(4)
C17	3250.0(9)	5697.1(19)	4898(3)	21.4(4)

Table S36. Anisotropic Displacement Parameters (Å2×103) for **6r**. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Br1	20.66(14)	18.91(14)	32.55(15)	-3.41(7)	7.30(9)	-4.43(6)
O1	28.5(8)	42.8(9)	47.2(9)	-21.7(8)	5.1(7)	-9.9(7)
N1	29.6(10)	35.3(10)	26.8(9)	-1.8(7)	6.0(7)	1.6(7)
C1	16.5(8)	19.9(8)	16.7(8)	0.1(6)	4.9(7)	-0.4(7)
C2	16.9(8)	18.3(8)	21.8(8)	0.4(7)	4.4(7)	0.0(7)
C3	19.7(8)	19.2(9)	20.5(8)	-0.6(6)	7.4(7)	-3.2(7)
C4	16.7(8)	27.2(10)	22.3(9)	-0.1(7)	3.2(7)	-4.8(7)
C5	17.9(9)	25.1(9)	23.3(9)	3.5(7)	2.2(7)	1.5(7)
C6	19.5(9)	19.0(9)	20.0(9)	1.2(7)	4.9(7)	-0.5(7)
C7	21.7(9)	18.7(9)	27.7(9)	4.8(7)	-0.4(7)	1.4(7)
C8	23.9(9)	18.1(9)	25.8(9)	3.9(7)	3.2(7)	-0.4(7)
C9	17.2(9)	17.1(8)	23.3(9)	-1.5(7)	3.4(7)	0.4(6)
C10	16.8(8)	15.2(8)	23.6(9)	-2.0(6)	3.4(7)	-0.2(6)
C11	17.5(8)	18.6(8)	21.1(9)	-0.8(7)	4.5(7)	-1.1(7)
C12	17.1(8)	19.0(9)	29.0(9)	-3.5(7)	5.8(7)	0.3(7)
C13	19.3(9)	18.9(8)	25.5(9)	-4.3(7)	5.2(7)	-1.5(7)
C14	17.0(8)	15.1(8)	18.9(8)	0.5(6)	2.1(7)	-0.7(6)
C15	20.7(9)	20.8(9)	28.7(9)	2.5(7)	3.5(8)	-3.0(7)
C16	18.7(9)	29.5(10)	39.2(11)	0.5(9)	4.1(8)	-1.2(8)
C17	17.0(8)	18.8(8)	28.5(10)	-7.0(7)	3.9(7)	0.4(7)

Table S37. Bond Lengths for **6r**.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Br1	C3	1.9054(19)	C7	C8	1.527(3)
O1	C15	1.211(2)	C8	C9	1.527(3)

N1	C17	1.145(3)	C9	C10	1.547(2)
C1	C2	1.398(3)	C9	C14	1.537(2)
C1	C6	1.398(3)	C10	C11	1.543(2)
C1	C14	1.521(2)	C10	C17	1.478(3)
C2	C3	1.382(3)	C11	C12	1.533(2)
C3	C4	1.392(3)	C11	C15	1.525(3)
C4	C5	1.378(3)	C12	C13	1.526(2)
C5	C6	1.402(3)	C13	C14	1.535(2)
C6	C7	1.515(2)	C15	C16	1.494(3)

Table S38. Bond Angles for **6r**.

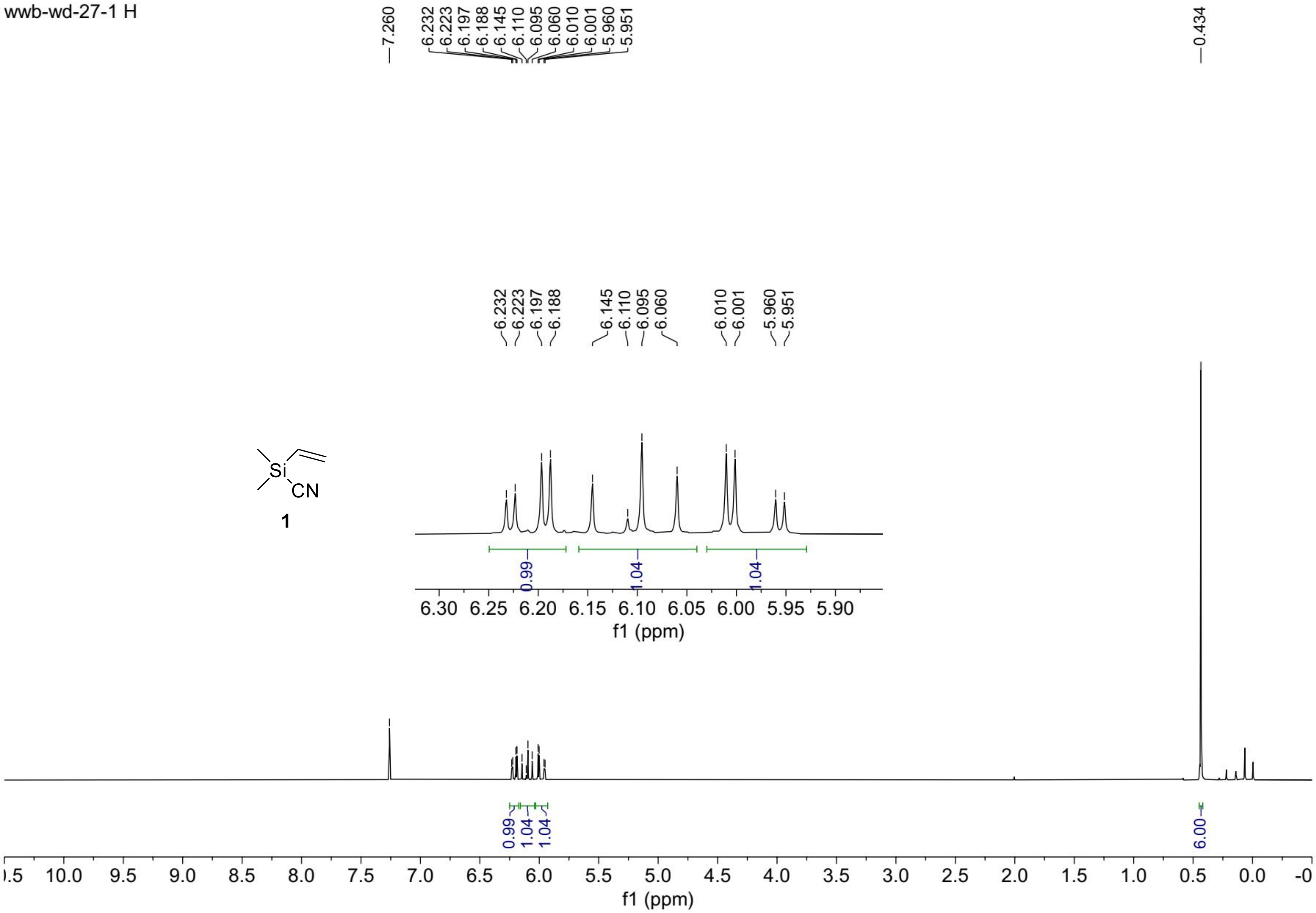
Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C2	C1	C14	118.13(15)	C14	C9	C10	111.60(14)
C6	C1	C2	119.53(16)	C11	C10	C9	113.70(15)
C6	C1	C14	122.33(16)	C17	C10	C9	107.57(15)
C3	C2	C1	119.76(17)	C17	C10	C11	111.58(15)
C2	C3	Br1	119.02(14)	C12	C11	C10	110.86(15)
C2	C3	C4	121.81(17)	C15	C11	C10	111.49(15)
C4	C3	Br1	119.17(14)	C15	C11	C12	113.55(15)
C5	C4	C3	117.90(17)	C13	C12	C11	110.87(15)
C4	C5	C6	122.05(17)	C12	C13	C14	112.07(15)
C1	C6	C5	118.93(17)	C1	C14	C9	110.96(14)
C1	C6	C7	121.69(16)	C1	C14	C13	111.76(14)
C5	C6	C7	119.38(16)	C13	C14	C9	112.34(15)
C6	C7	C8	112.55(15)	O1	C15	C11	120.65(17)
C9	C8	C7	108.27(15)	O1	C15	C16	121.43(18)
C8	C9	C10	113.57(15)	C16	C15	C11	117.92(17)
C8	C9	C14	110.60(15)	N1	C17	C10	175.2(2)

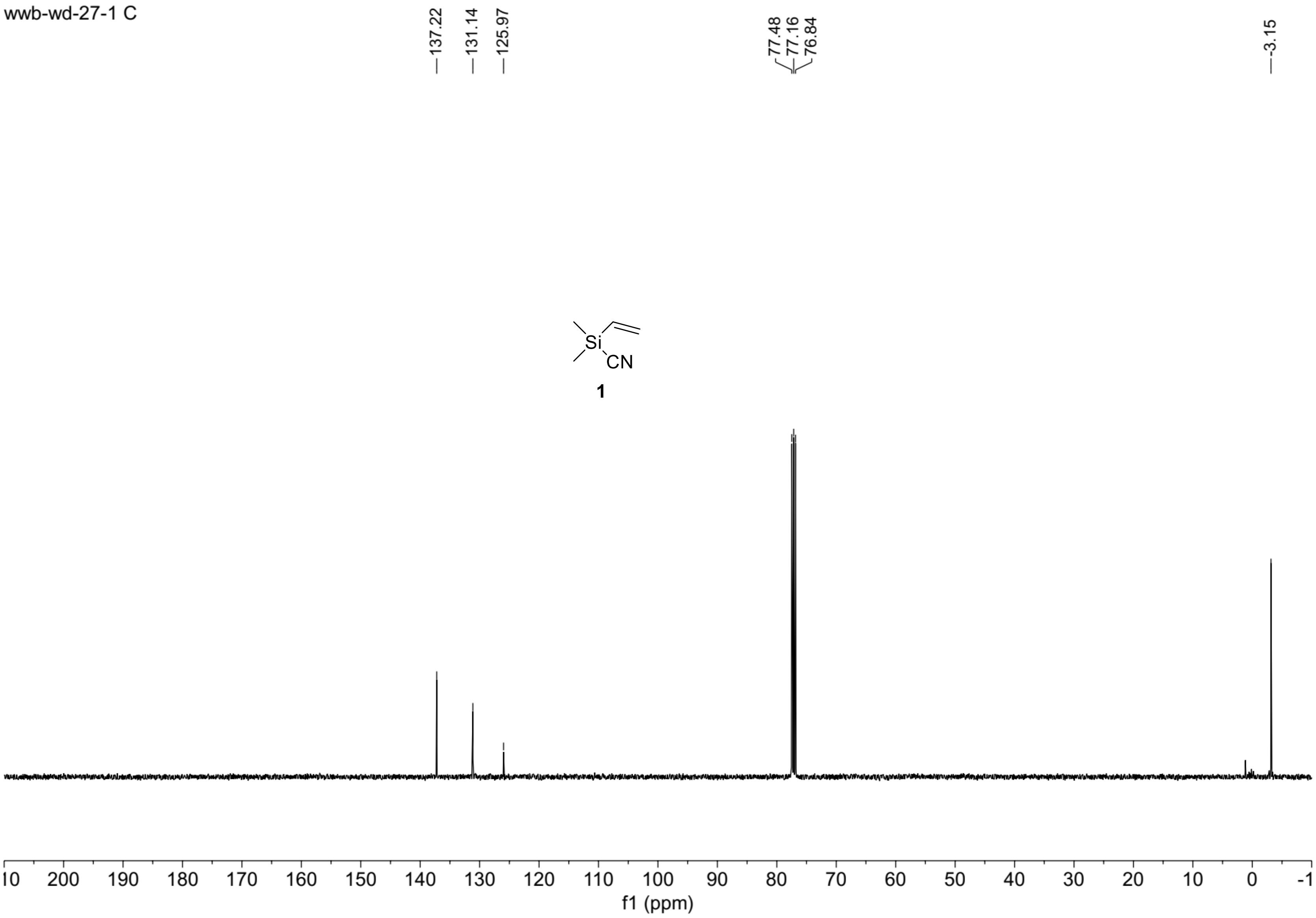
Table S39. Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for **6r**.

Atom	*x*	*y*	*z*	U(eq)
H2	2014.7	1431.49	1237.11	23
H4	155.83	2067.95	-1697.1	26

H5	359.51	4442.76	-1588.82	27
H7A	1159.49	6396.21	653.63	28
H7B	1262.5	6313.84	-1344.03	28
H8A	2437.76	6093.38	-561.82	27
H8B	2223.02	7354.63	555.82	27
H9	2132.81	5732.27	2913.88	23
H10	3141.52	7167.11	2991.17	22
H11	3698.12	6089.89	917.71	23
H12A	4147.95	3831.95	1248.49	26
H12B	3859.35	3649.22	3041.92	26
H13A	3109.63	2571.37	752.14	25
H13B	3034.97	3967.11	-340.15	25
H14	2547.34	3542.84	2923.53	21
H16A	5476.44	6310.63	3101.65	44
H16B	5065.76	5916.68	1233.57	44
H16C	5163.86	4810.48	2771.11	44

11. NMR spectra and HPLC trace

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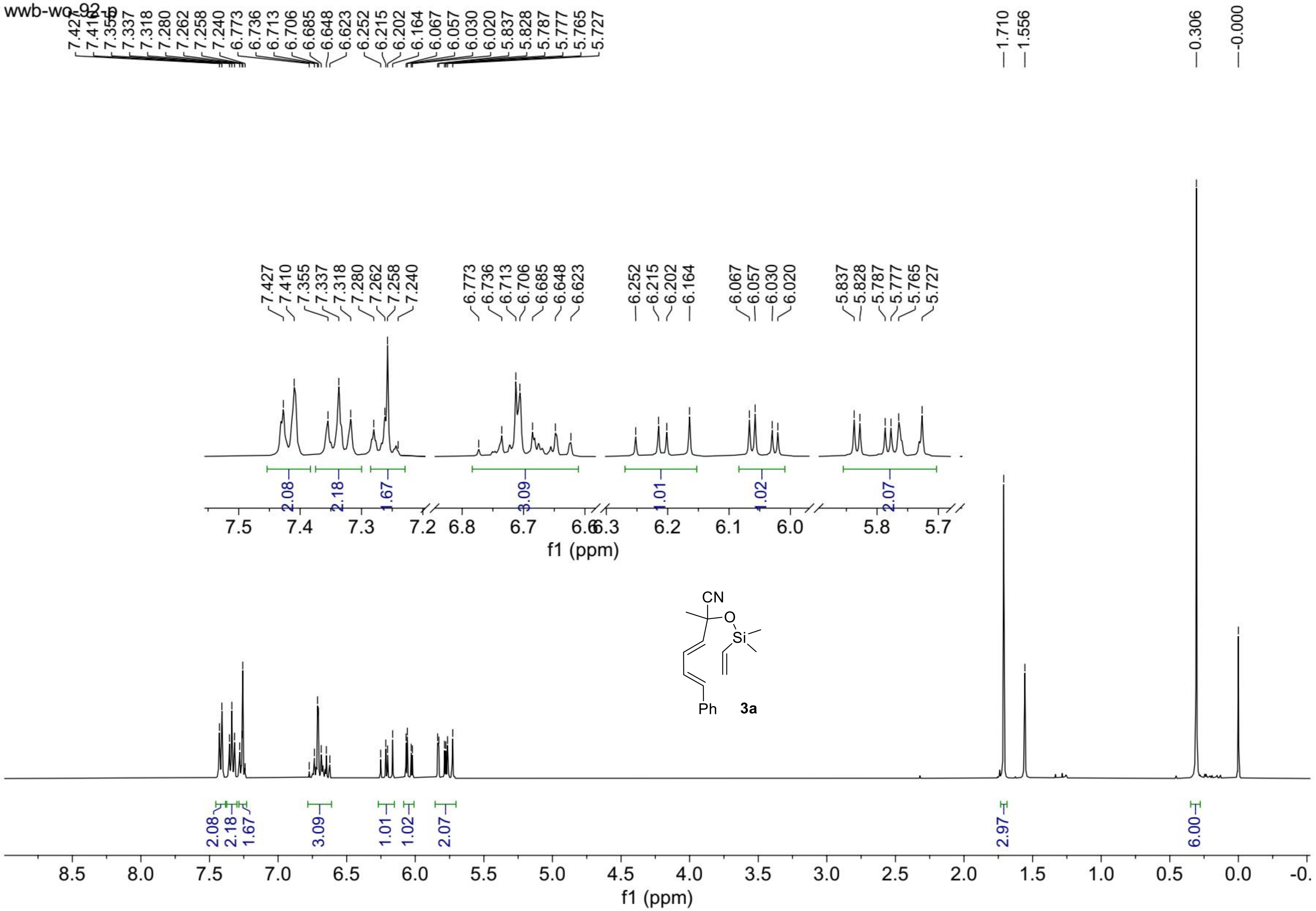
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1.01
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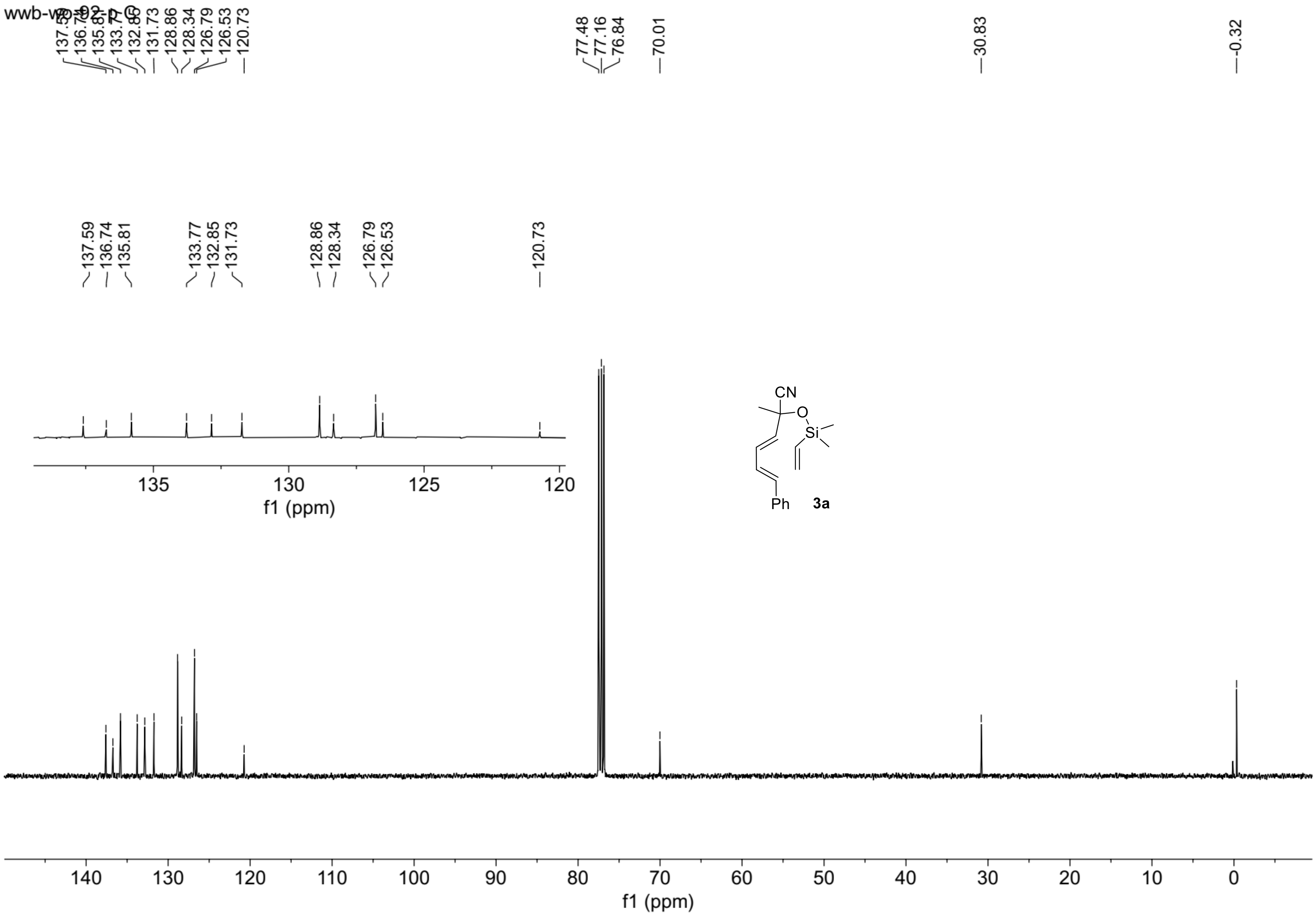
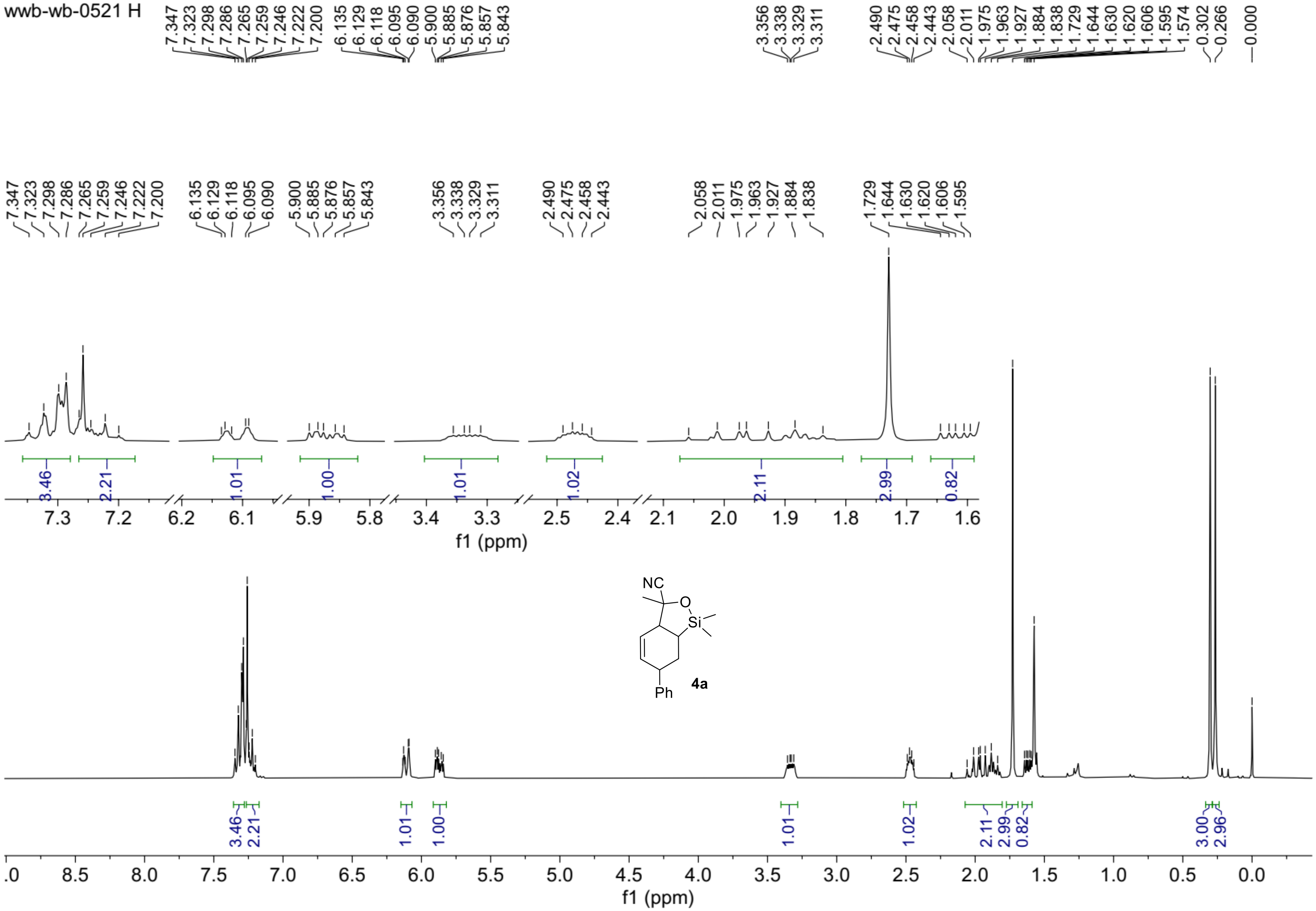
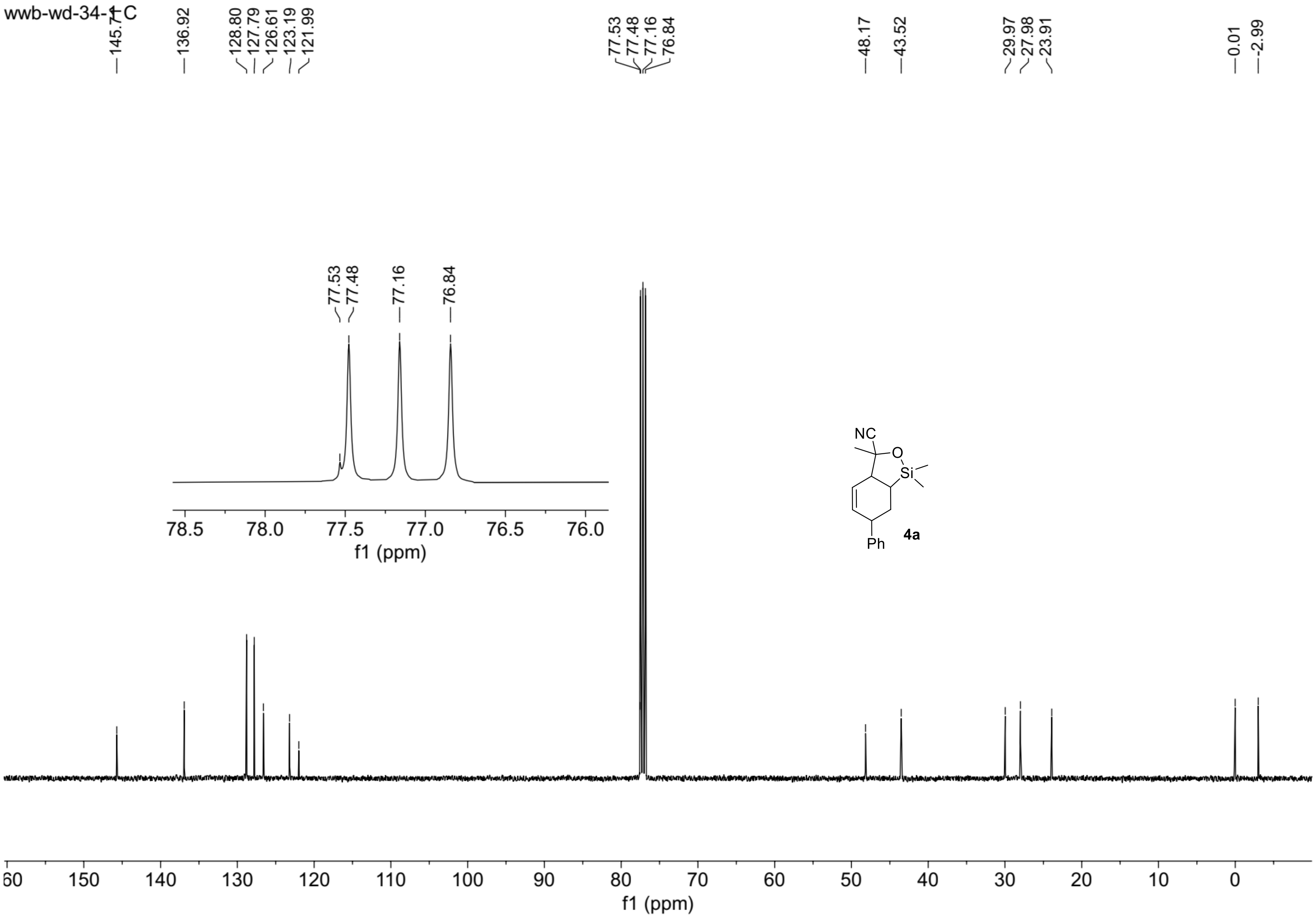
f1 (ppm)
CN
O
Si
Ph

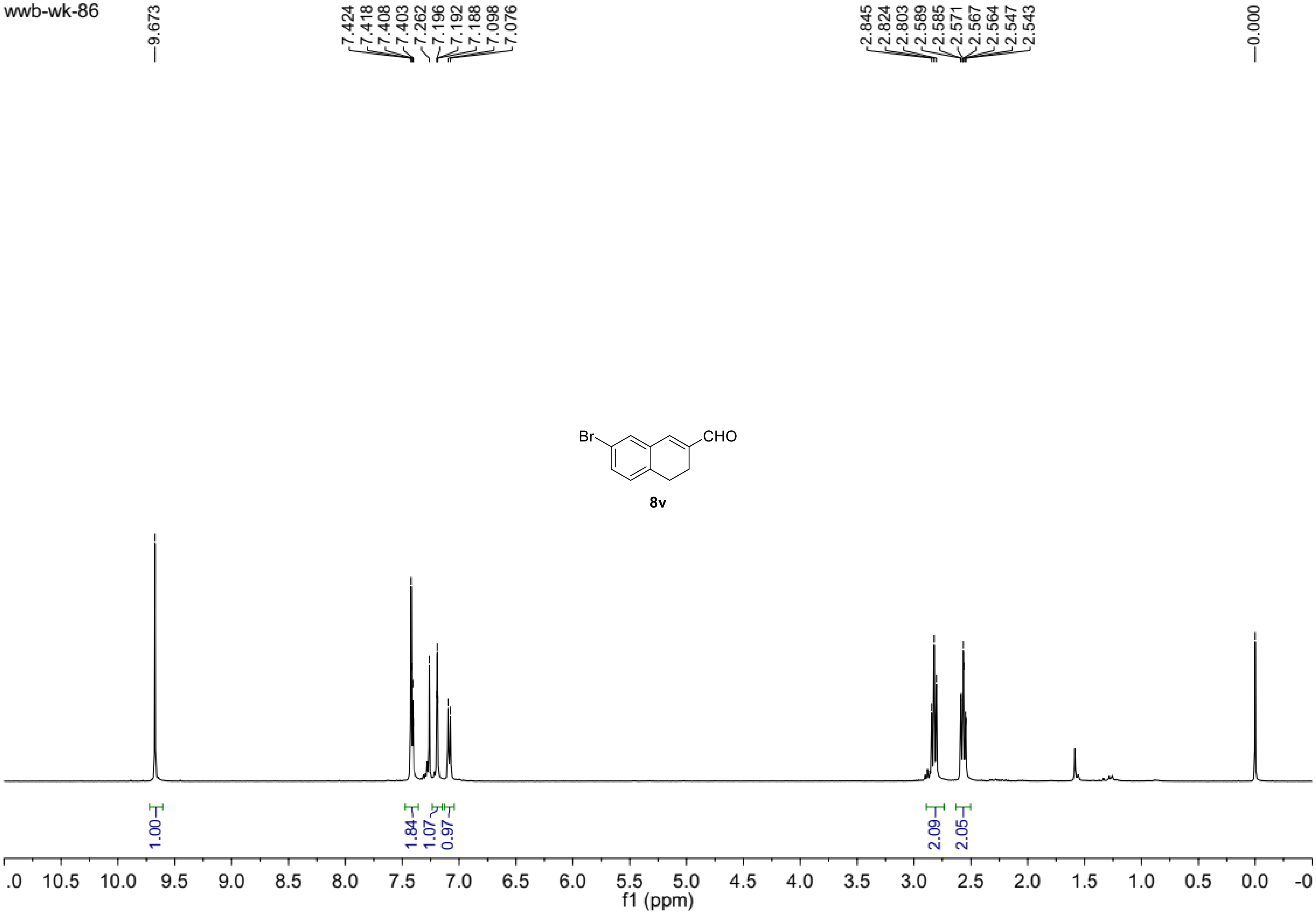
3a

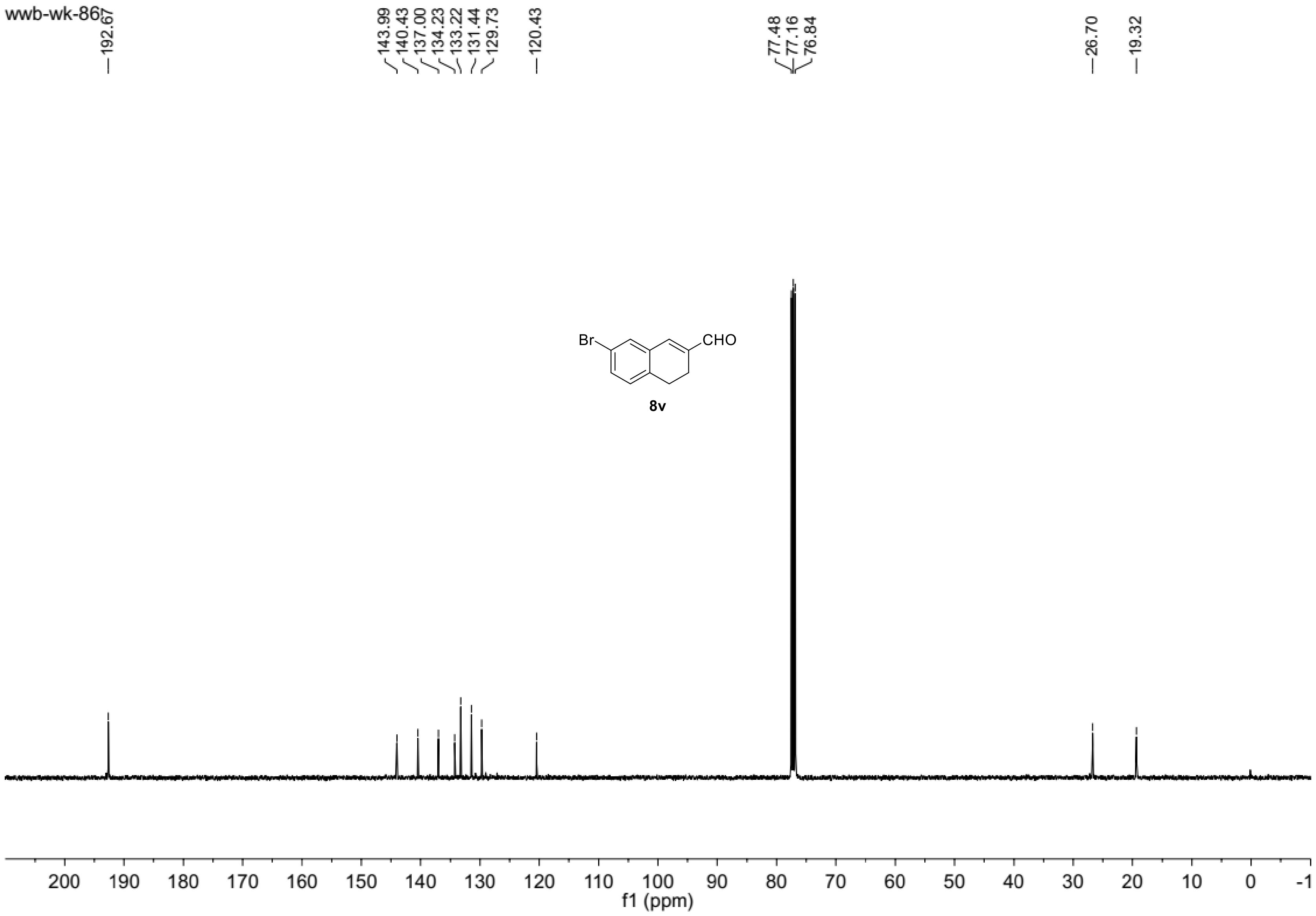

wwb-wo-92-p-C
137.59
136.74
135.81
133.77
132.85
131.73
128.86
128.34
126.79
126.53
120.73
77.48
77.16
76.84
70.01
30.83
-0.32
135
130
125
120
f1 (ppm)
CN
O
Si
Ph
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

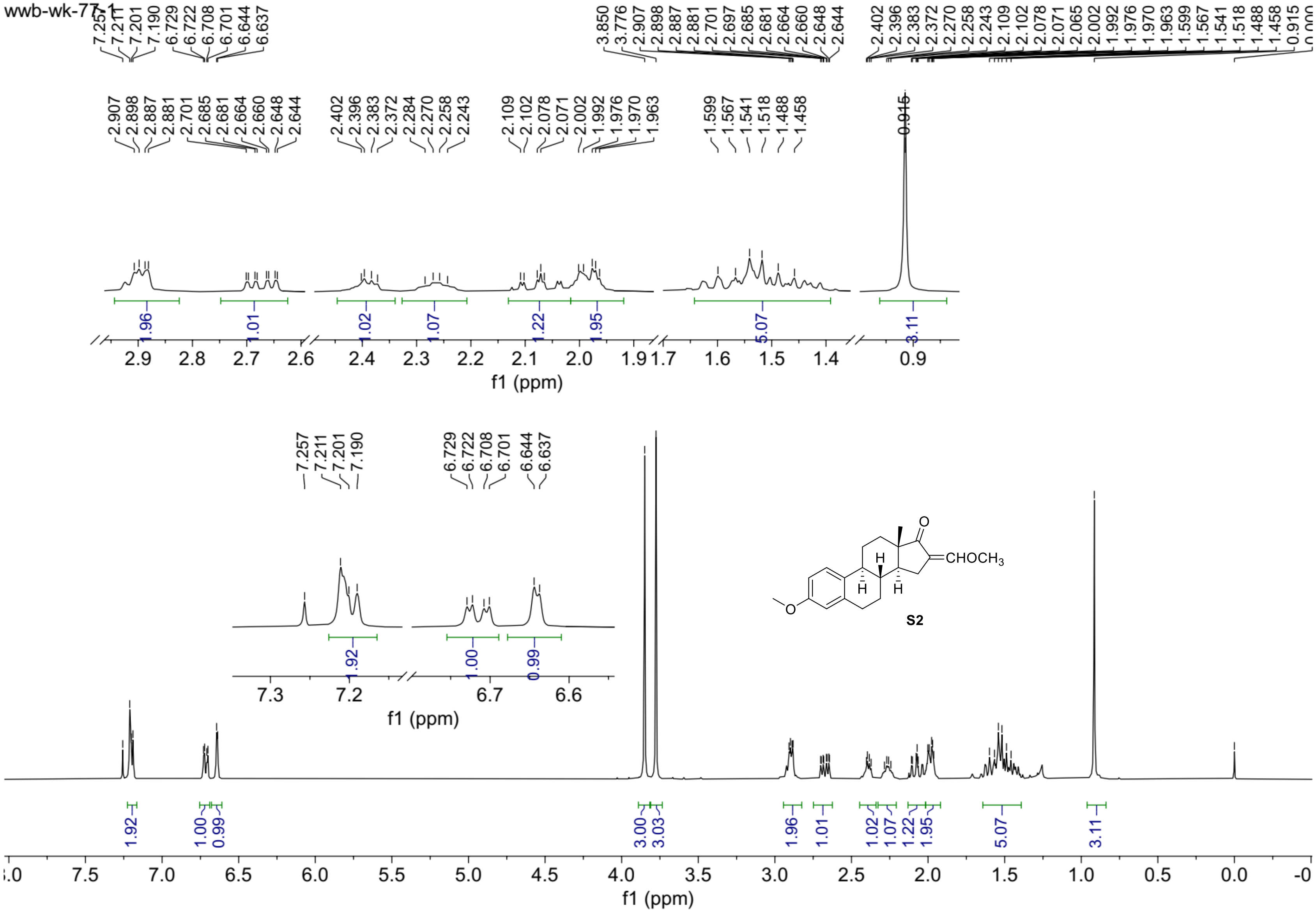
3a


wwb-wb-0521 H
7.347
7.323
7.298
7.286
7.265
7.259
7.246
7.222
7.200
6.135
6.129
6.118
6.095
6.090
5.900
5.885
5.876
5.857
5.843
3.356
3.338
3.329
3.311
2.490
2.475
2.458
2.443
2.058
2.011
1.975
1.963
1.927
1.884
1.838
1.729
1.644
1.630
1.620
1.606
1.595
1.574
0.302
0.266
0.000
3.46
2.21
1.01
1.00
1.01
1.02
2.11
2.99
0.82
3.00
2.96
7.3
7.2
6.2
6.1
5.9
5.8
3.4
3.3
2.5
2.4
2.1
2.0
1.9
1.8
1.7
1.6
f1 (ppm)
NC
O
Si
Ph
4a
.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
f1 (ppm)

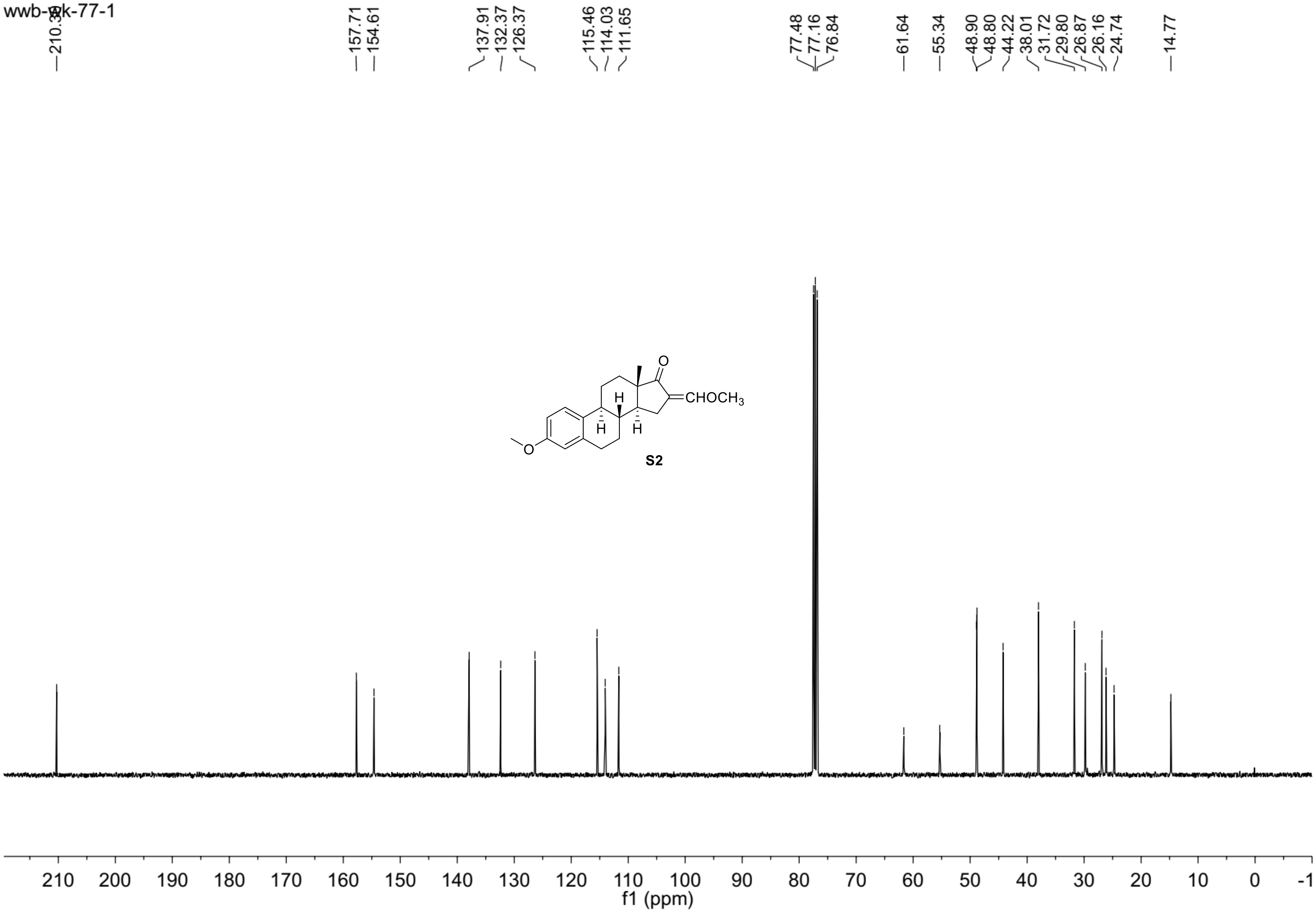

wwb-wd-34-1C
145.7
136.92
128.80
127.79
126.61
123.19
121.99
77.53
77.48
77.16
76.84
48.17
43.52
29.97
27.98
23.91
0.01
-2.99
77.53
77.48
77.16
76.84
78.5
78.0
77.5
77.0
76.5
76.0
f1 (ppm)
NC
O
Si
Ph
4a
60
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

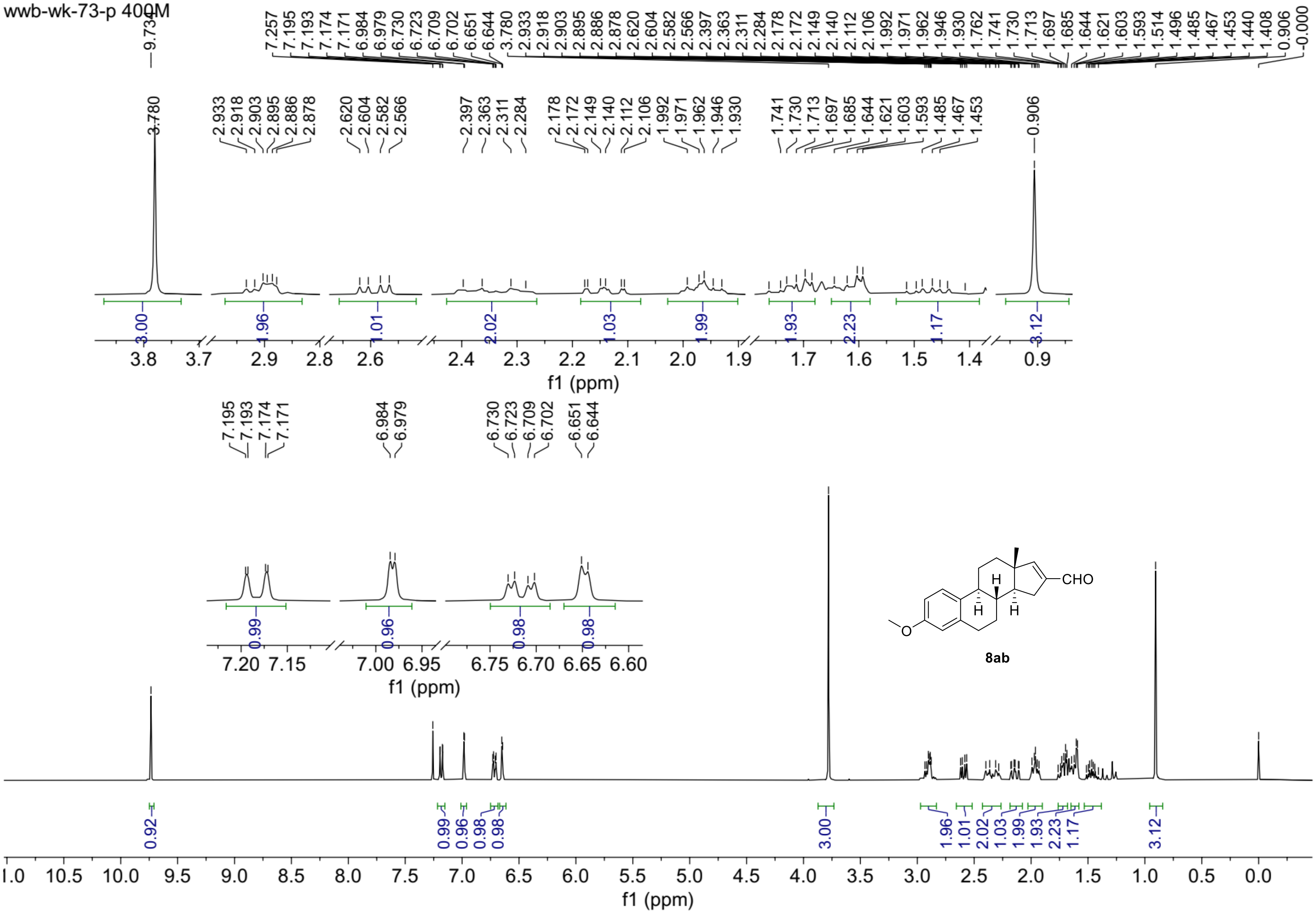

wwb-wk-86
9.673
7.424
7.418
7.408
7.403
7.262
7.196
7.192
7.188
7.098
7.076
2.845
2.824
2.803
2.589
2.585
2.571
2.567
2.564
2.547
2.543
0.000
Br
CHO
8v
1.00
1.84
1.07
0.97
2.09
2.05
.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

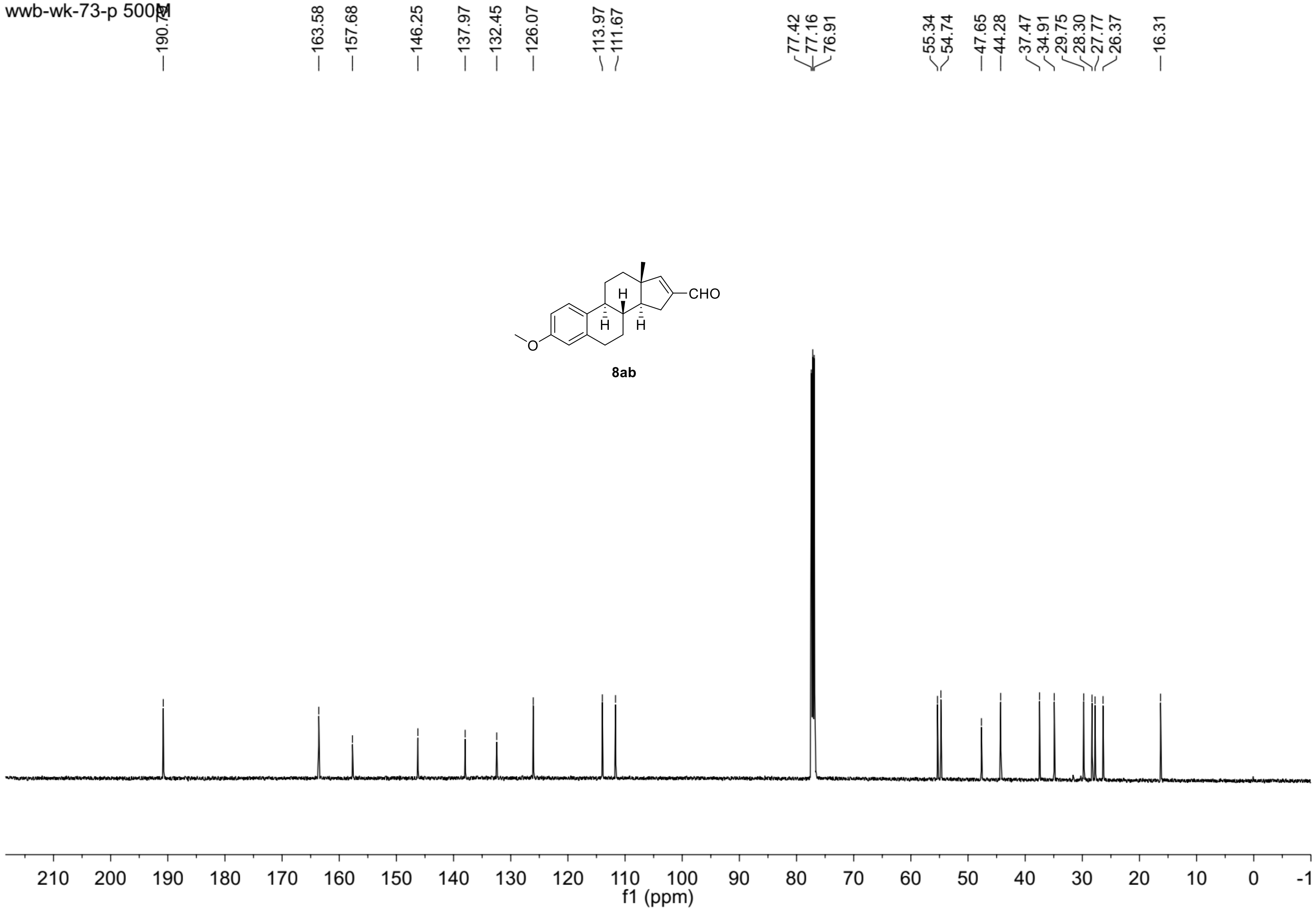

wwb-wk-86
192.67
143.99
140.43
137.00
134.23
133.22
131.44
129.73
120.43
77.48
77.16
76.84
26.70
19.32
Br
CHO
8v
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)


wwb-wk-77-1
7.257 7.211 7.201 7.190 6.729 6.722 6.708 6.701 6.644 6.637
3.850 3.776 2.907 2.898 2.887 2.881 2.701 2.697 2.685 2.681 2.664 2.660 2.648 2.644
2.402 2.396 2.383 2.372 2.270 2.258 2.243 2.109 2.102 2.078 2.071 2.065 2.002 1.992 1.976 1.970 1.963 1.599 1.567 1.541 1.518 1.488 1.458 0.915 0.000
2.907 2.898 2.887 2.881 2.701 2.685 2.681 2.664 2.660 2.648 2.644
2.402 2.396 2.383 2.372 2.284 2.270 2.258 2.243
2.109 2.102 2.078 2.071 2.002 1.992 1.976 1.970 1.963
1.599 1.567 1.541 1.518 1.488 1.458
0.915
1.96 1.01 1.02 1.07 1.22 1.95 5.07 3.11
2.9 2.8 2.7 2.6 2.4 2.3 2.2 2.1 2.0 1.9 1.7 1.6 1.5 1.4 0.9
f1 (ppm)
7.257 7.211 7.201 7.190
6.729 6.722 6.708 6.701 6.644 6.637
1.92 1.00 0.99
7.3 7.2 6.7 6.6
f1 (ppm)
O
H
H
H
CHOCH3
1.92 1.00 0.99 3.00 3.03 1.96 1.01 1.02 1.07 1.22 1.95 5.07 3.11
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)

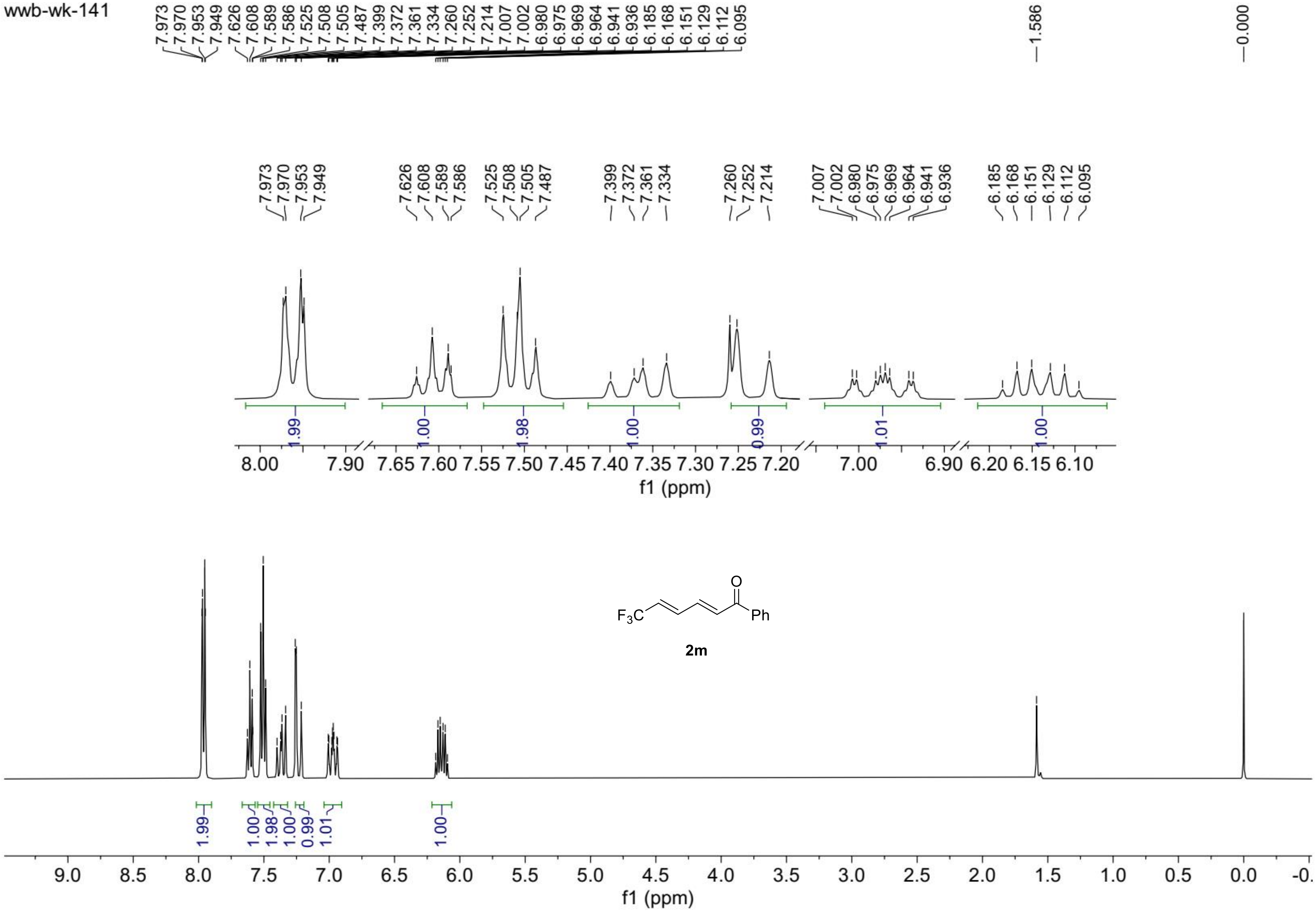
S2

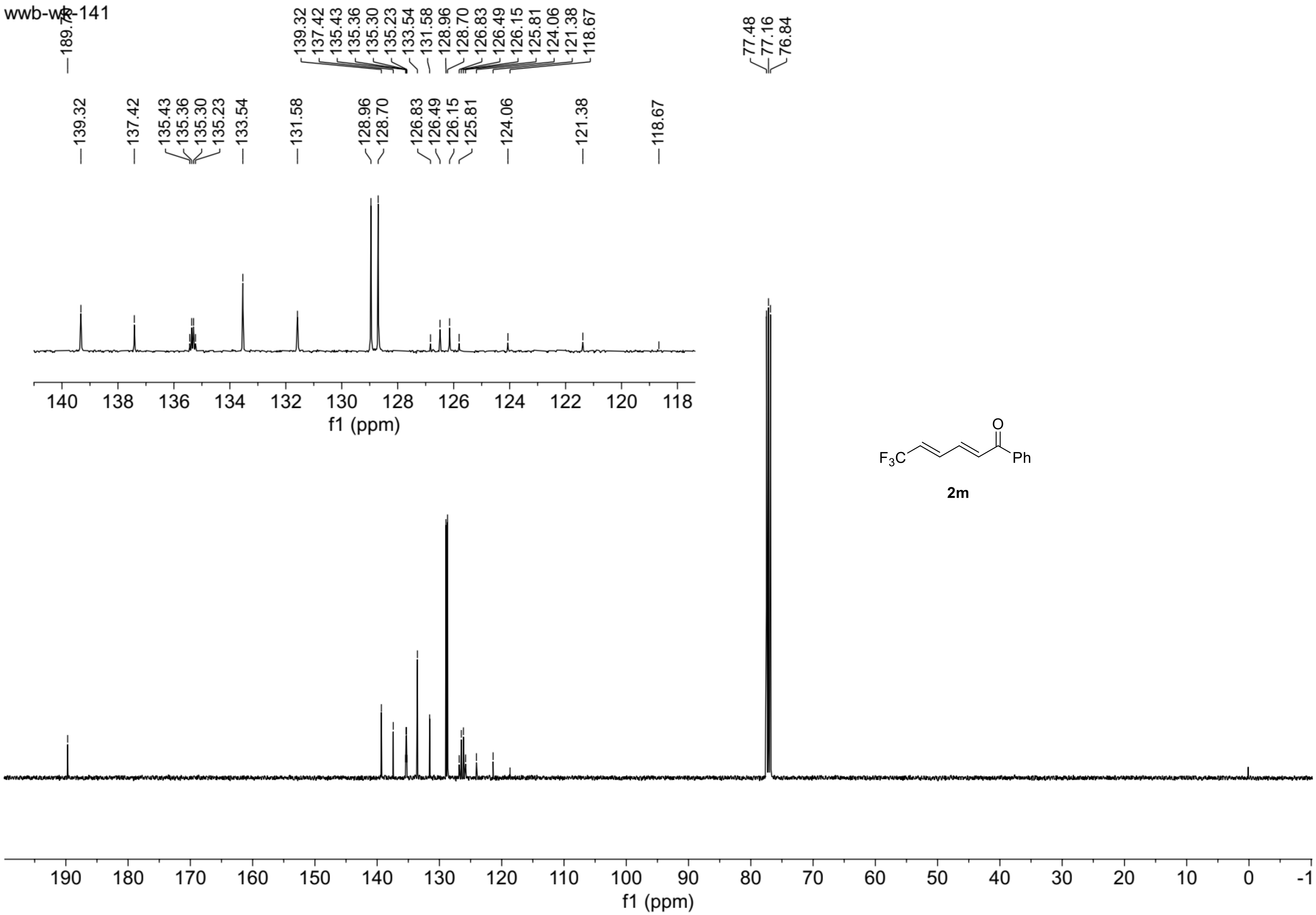

wwb-wk-77-1
210.3
157.71
154.61
137.91
132.37
126.37
115.46
114.03
111.65
77.48
77.16
76.84
61.64
55.34
48.90
48.80
44.22
38.01
31.72
29.80
26.87
26.16
24.74
14.77
O
H
H
H
CHOCH3
O
S2
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

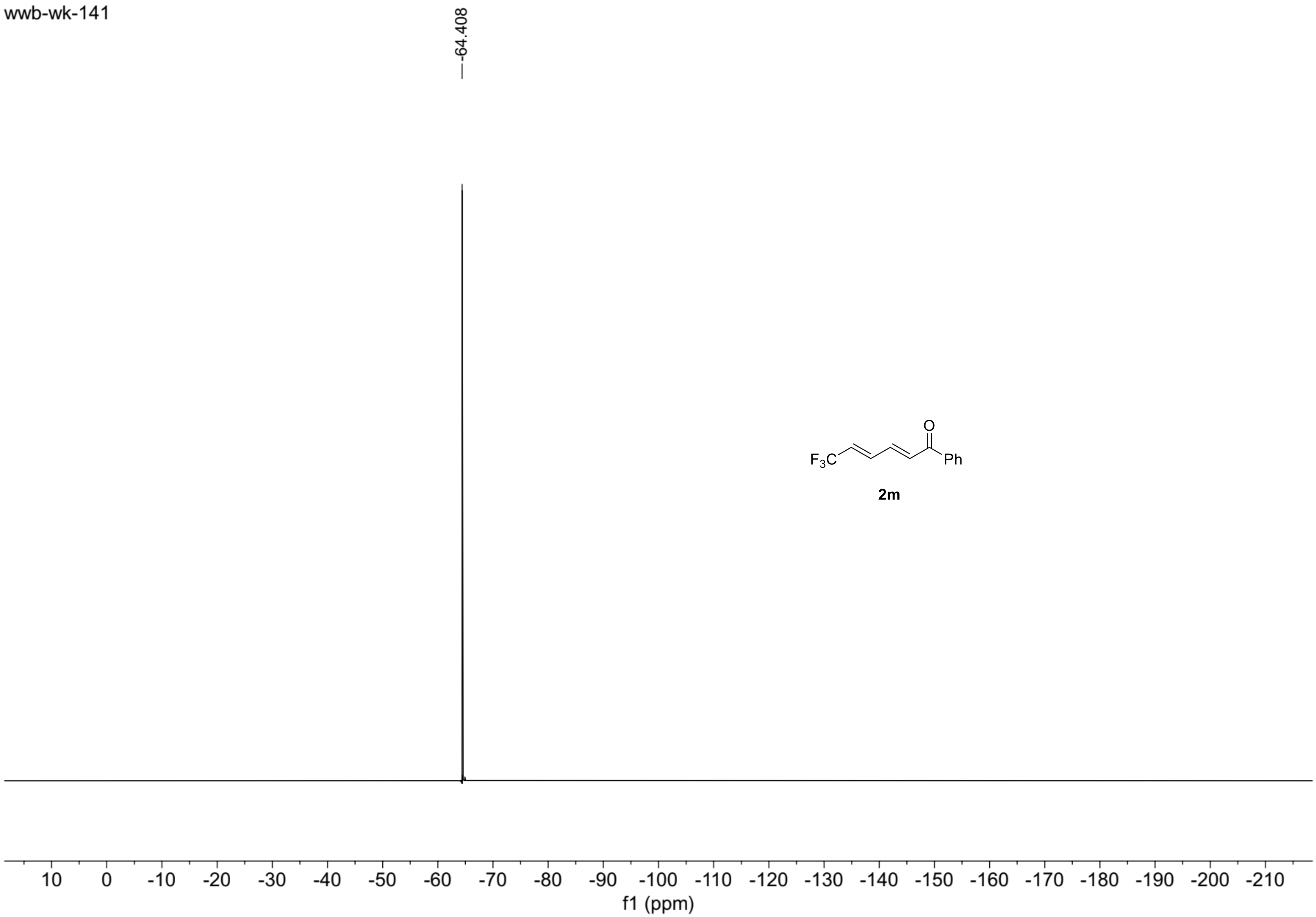

wwb-wk-73-p 400M
9.734
7.257
7.195
7.193
7.174
7.171
6.984
6.979
6.730
6.723
6.709
6.702
6.651
6.644
3.780
2.933
2.918
2.903
2.895
2.886
2.878
2.620
2.604
2.582
2.566
2.397
2.363
2.311
2.284
2.178
2.172
2.149
2.140
2.112
2.106
1.992
1.971
1.962
1.946
1.930
1.762
1.741
1.730
1.713
1.697
1.685
1.644
1.621
1.603
1.593
1.514
1.496
1.485
1.467
1.453
1.440
1.408
0.906
-0.000
3.00
1.96
1.01
2.02
1.03
1.99
1.93
2.23
1.17
3.12
0.99
0.96
0.98
0.98
0.92
f1 (ppm)
CHO
H
H
H
O
8ab

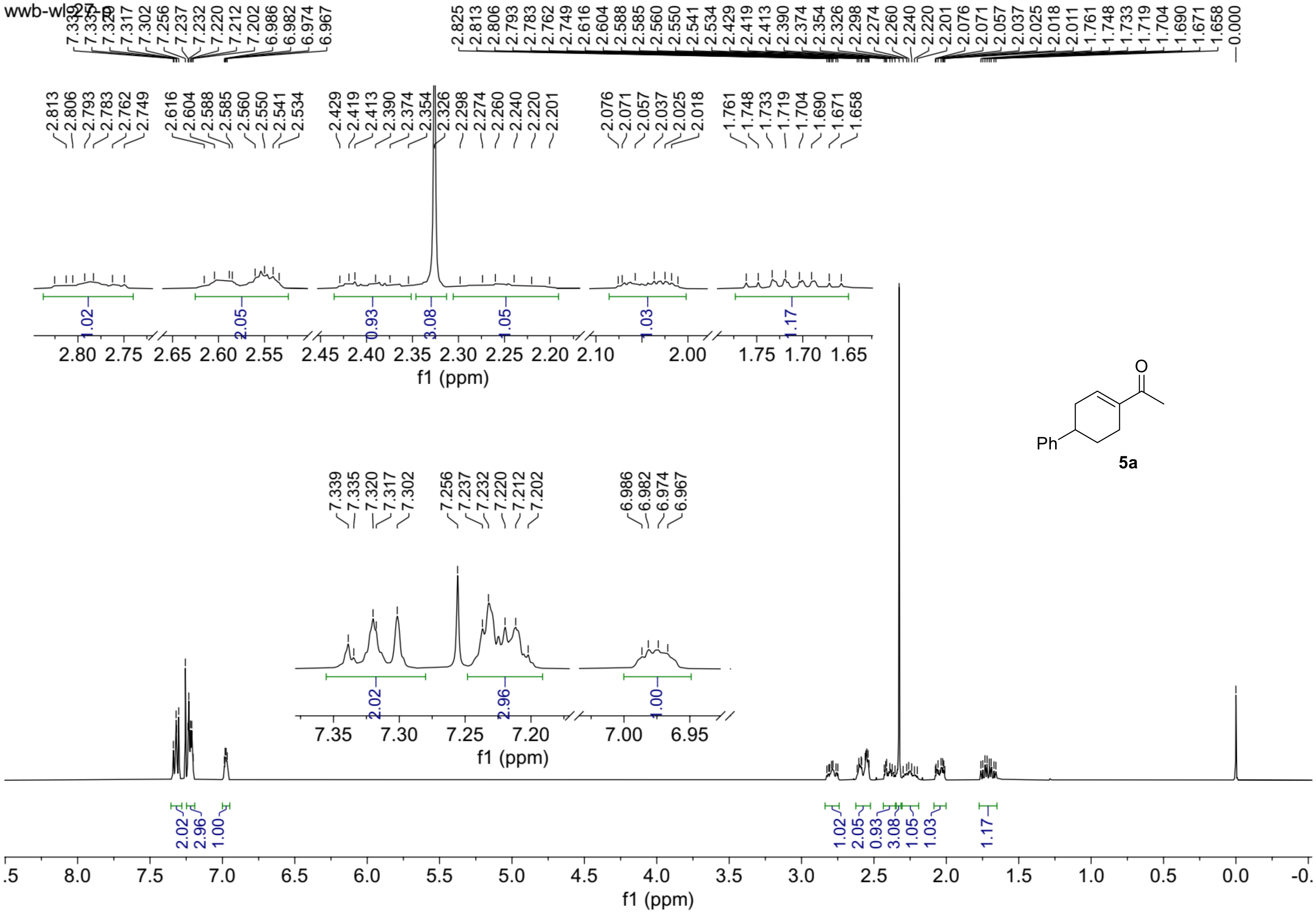

wwb-wk-73-p 500M
190.75
163.58
157.68
146.25
137.97
132.45
126.07
113.97
111.67
77.42
77.16
76.91
55.34
54.74
47.65
44.28
37.47
34.91
29.75
28.30
27.77
26.37
16.31
CHO
H
H
H
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

8ab

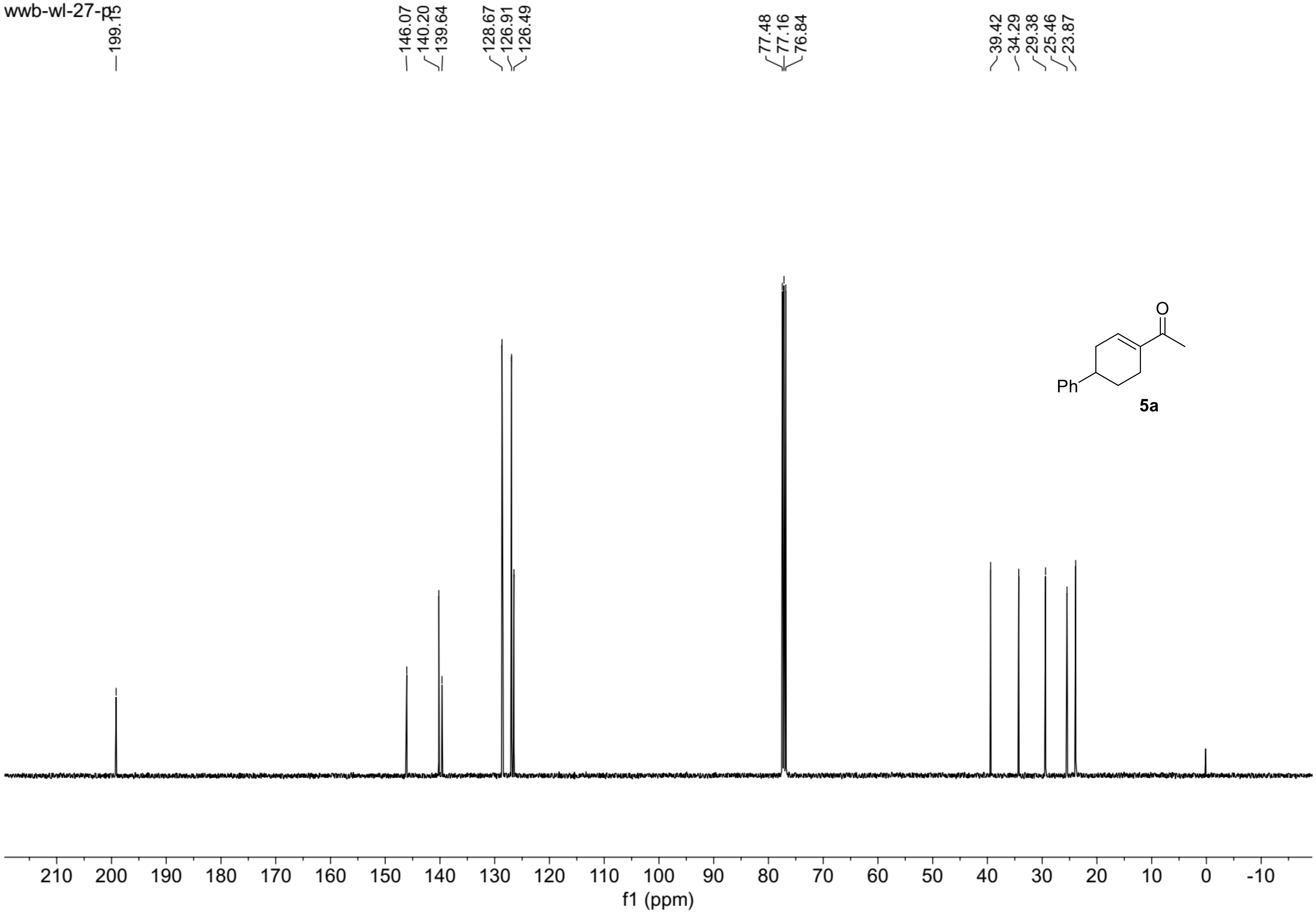

wwb-wk-141
7.973
7.970
7.953
7.949
7.626
7.608
7.589
7.586
7.525
7.508
7.505
7.487
7.399
7.372
7.361
7.334
7.260
7.252
7.214
7.007
7.002
6.980
6.975
6.969
6.964
6.941
6.936
6.185
6.168
6.151
6.129
6.112
6.095
1.586
0.000
1.99
1.00
1.98
1.00
0.99
1.01
1.00
8.00
7.90
7.65
7.60
7.55
7.50
7.45
7.40
7.35
7.30
7.25
7.20
7.00
6.90
6.20
6.15
6.10
f1 (ppm)
O
F3C
Ph
2m
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.
f1 (ppm)

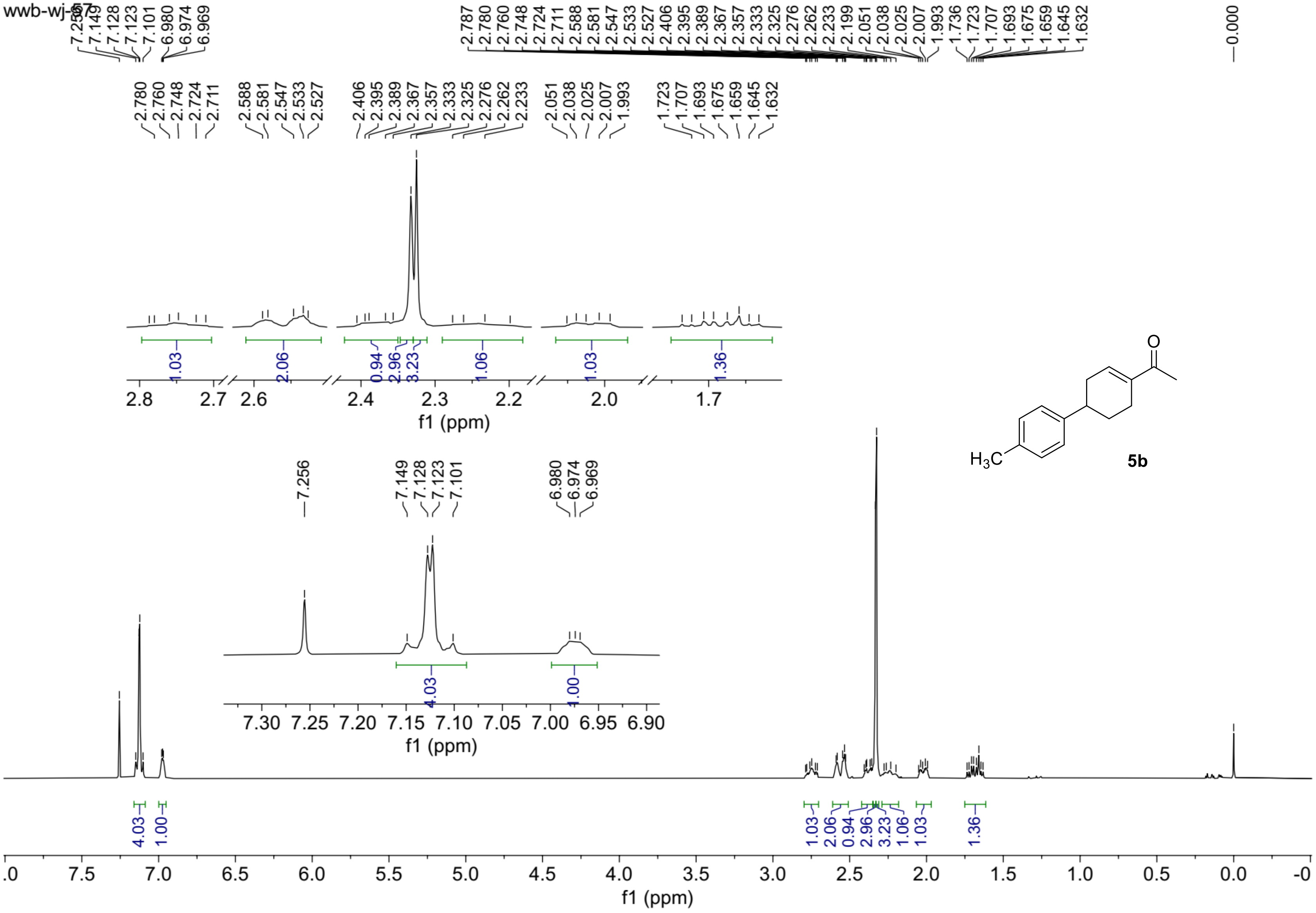

wwb-wk-141
189.78
139.32
137.42
135.43
135.36
135.30
135.23
133.54
131.58
128.96
128.70
126.83
126.49
126.15
125.81
124.06
121.38
118.67
77.48
77.16
76.84
140
138
136
134
132
130
128
126
124
122
120
118
f1 (ppm)
O
F3C
Ph
2m
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)


wwb-wk-141
-64.408
F3C
O
Ph
2m
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

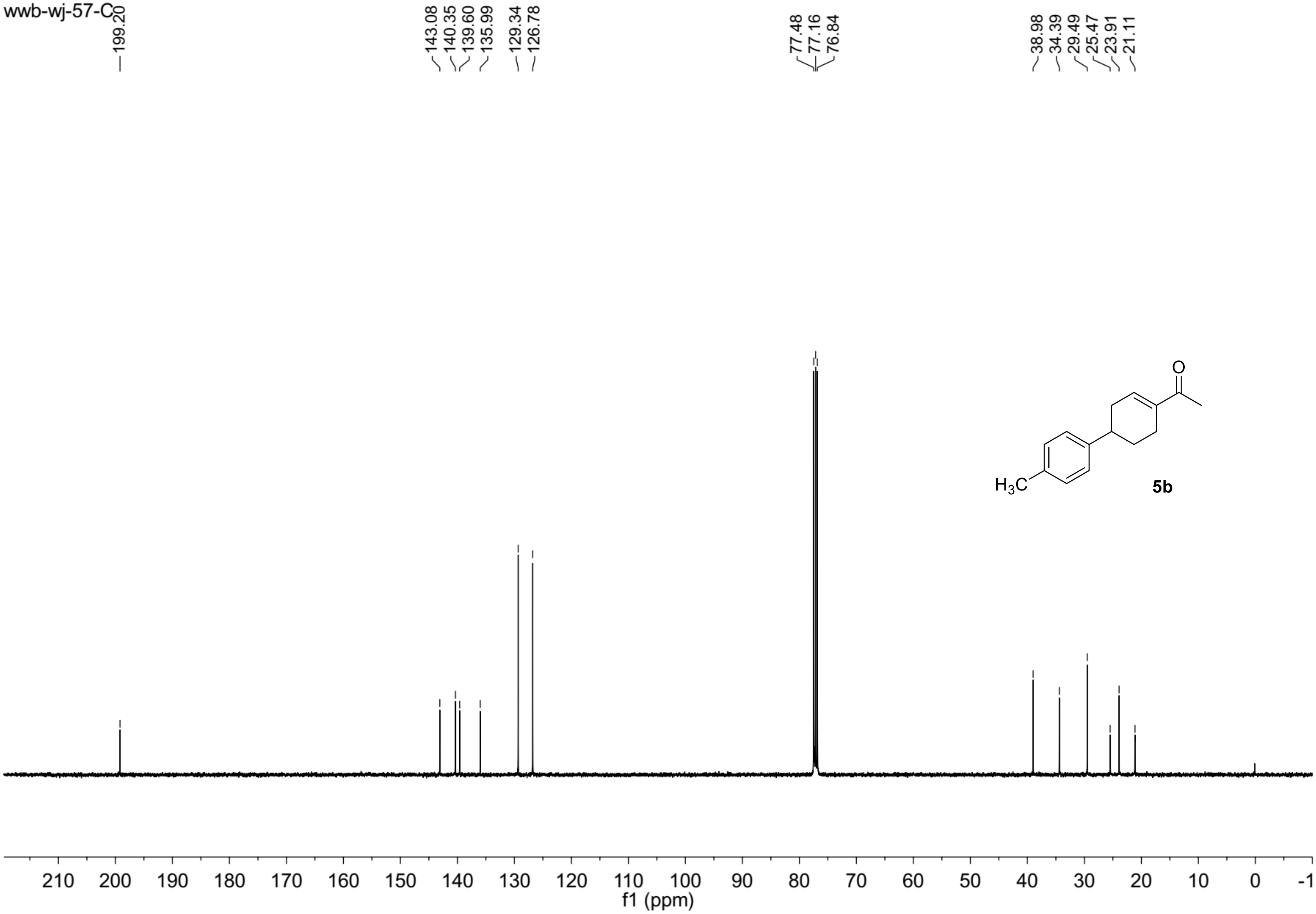

wwb-wl927-p
7.339
7.335
7.320
7.317
7.302
7.256
7.237
7.232
7.220
7.212
7.202
6.986
6.982
6.974
6.967
2.825
2.813
2.806
2.793
2.783
2.762
2.749
2.616
2.604
2.588
2.585
2.560
2.550
2.541
2.534
2.429
2.419
2.413
2.390
2.374
2.354
2.326
2.298
2.274
2.260
2.240
2.220
2.201
2.076
2.071
2.057
2.037
2.025
2.018
2.011
1.761
1.748
1.733
1.719
1.704
1.690
1.671
1.658
0.000
1.02
2.05
0.93
3.08
1.05
1.03
1.17
2.80 2.75 2.65 2.60 2.55 2.45 2.40 2.35 2.30 2.25 2.20 2.10 2.00 1.75 1.70 1.65
f1 (ppm)
O
Ph
2.02
2.96
1.00
7.35 7.30 7.25 7.20 7.00 6.95
f1 (ppm)
.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.
f1 (ppm)

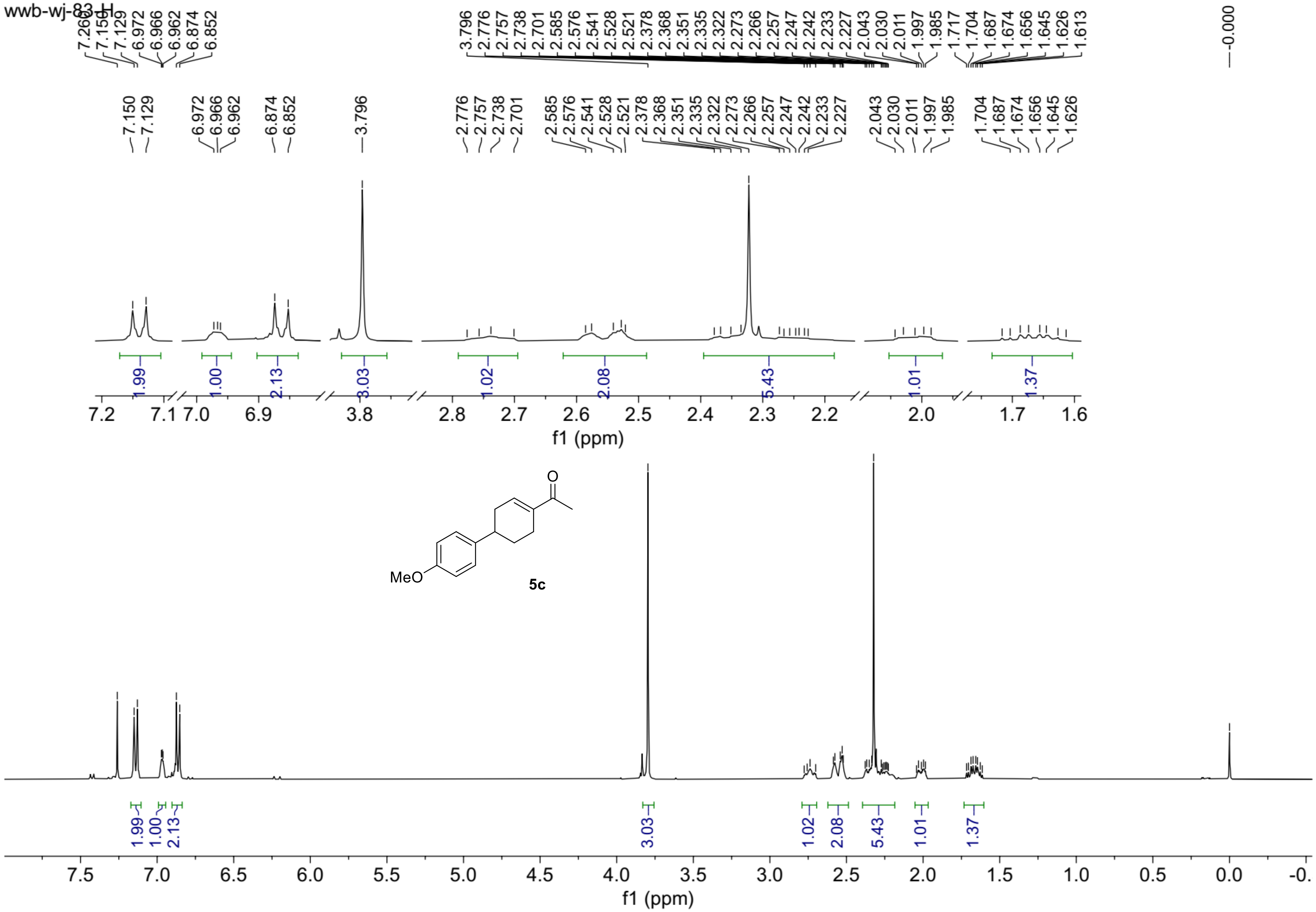
5a

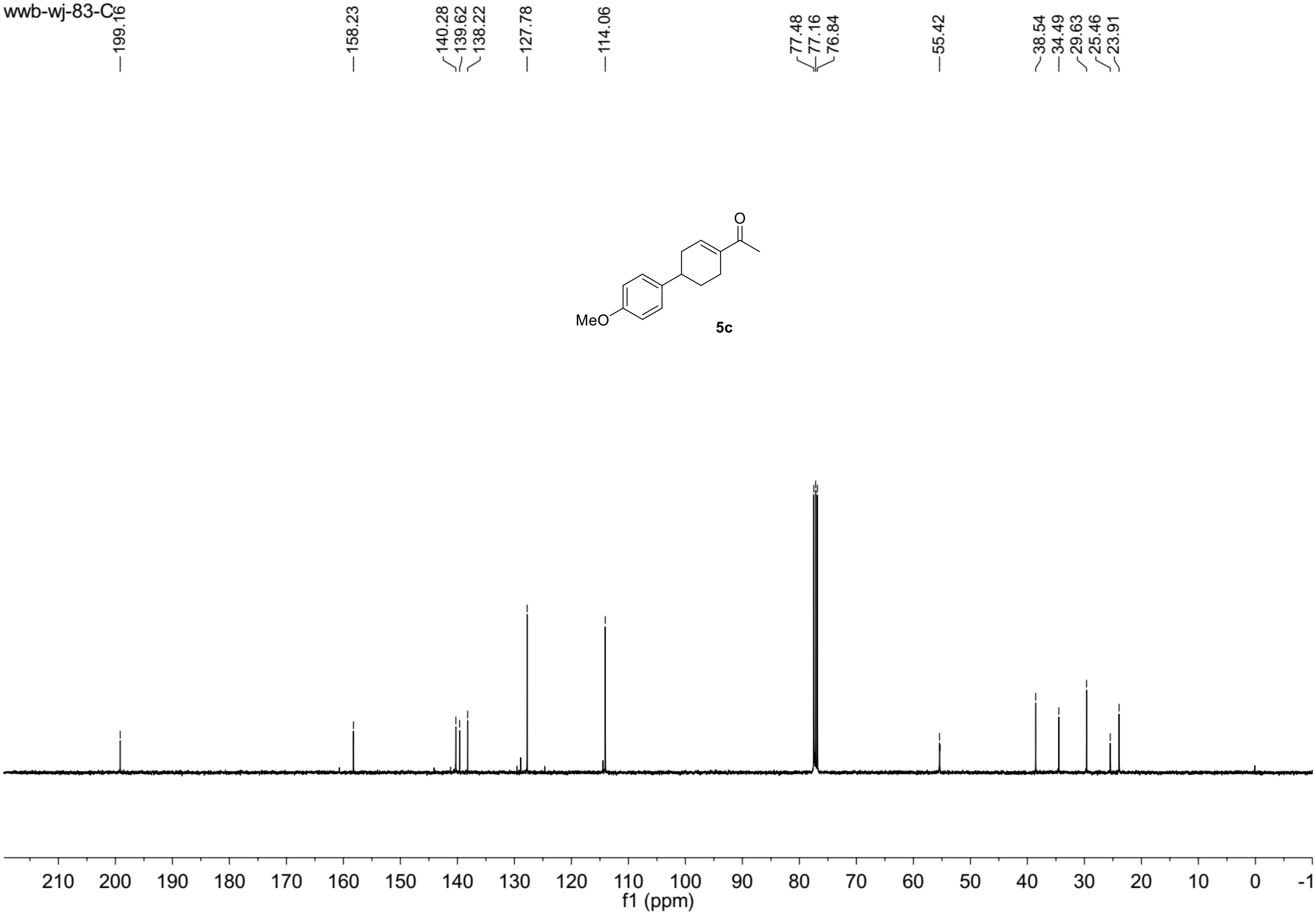

wwb-wl-27-p5
199.15
146.07
140.20
139.64
128.67
126.91
126.49
77.48
77.16
76.84
39.42
34.29
29.38
25.46
23.87
O
Ph
5a
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

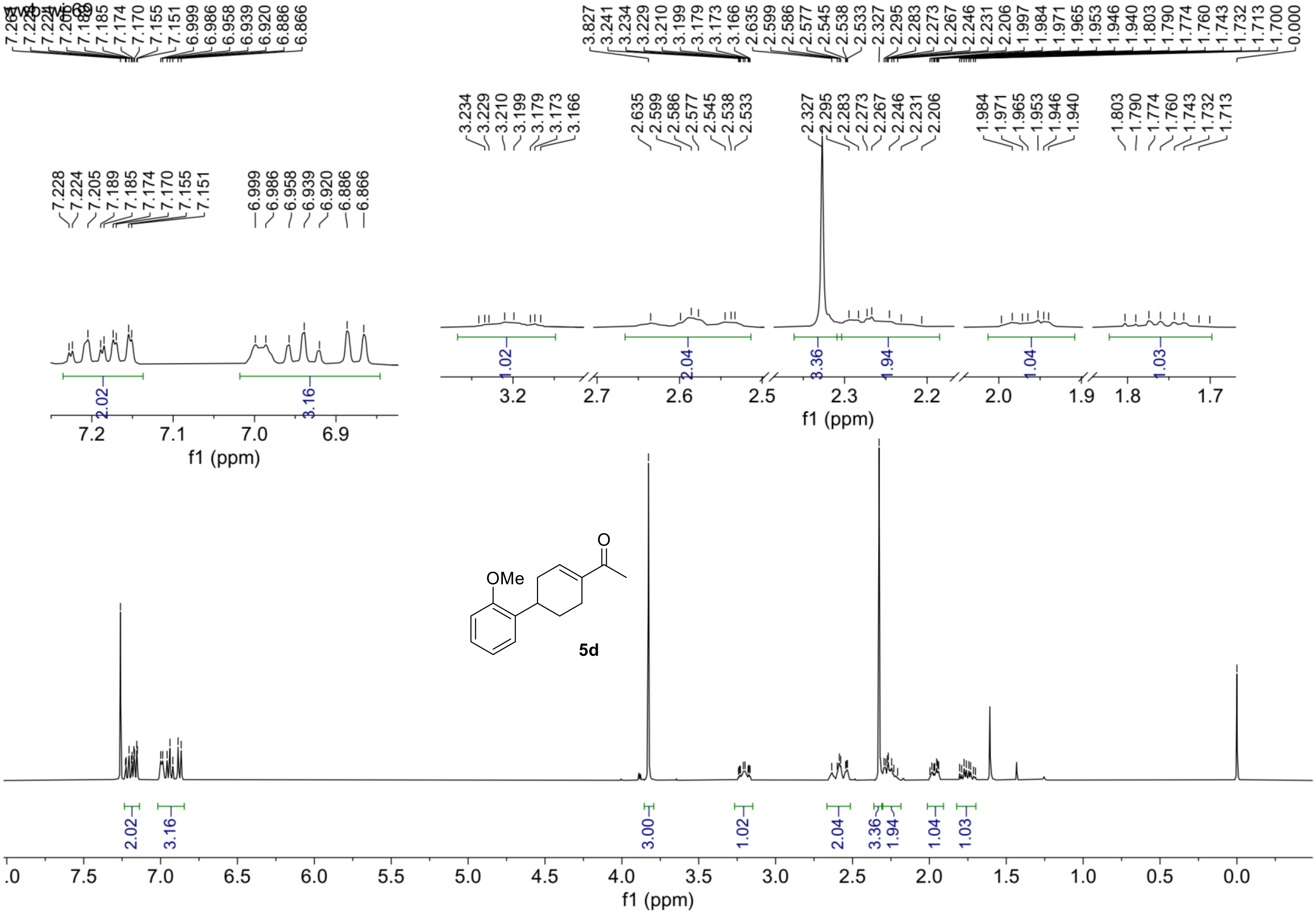

wwb-wj-57
7.256 7.149 7.128 7.123 7.101 6.980 6.974 6.969
2.787 2.780 2.760 2.748 2.724 2.711 2.588 2.581 2.547 2.533 2.527 2.406 2.395 2.389 2.367 2.357 2.333 2.325 2.276 2.262 2.233 2.199 2.051 2.038 2.025 2.007 1.993 1.736 1.723 1.707 1.693 1.675 1.659 1.645 1.632
0.000
2.780 2.760 2.748 2.724 2.711
2.588 2.581 2.547 2.533 2.527
2.406 2.395 2.389 2.367 2.357 2.333 2.325 2.276 2.262 2.233
2.051 2.038 2.025 2.007 1.993
1.723 1.707 1.693 1.675 1.659 1.645 1.632
1.03 2.06 0.94 2.96 3.23 1.06 1.03 1.36
2.8 2.7 2.6 2.4 2.3 2.2 2.0 1.7
f1 (ppm)
O
H_3C
7.256
7.149 7.128 7.123 7.101
6.980 6.974 6.969
4.03 1.00
7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90
f1 (ppm)
4.03 1.00 1.03 2.06 0.94 2.96 3.23 1.06 1.03 1.36
.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)

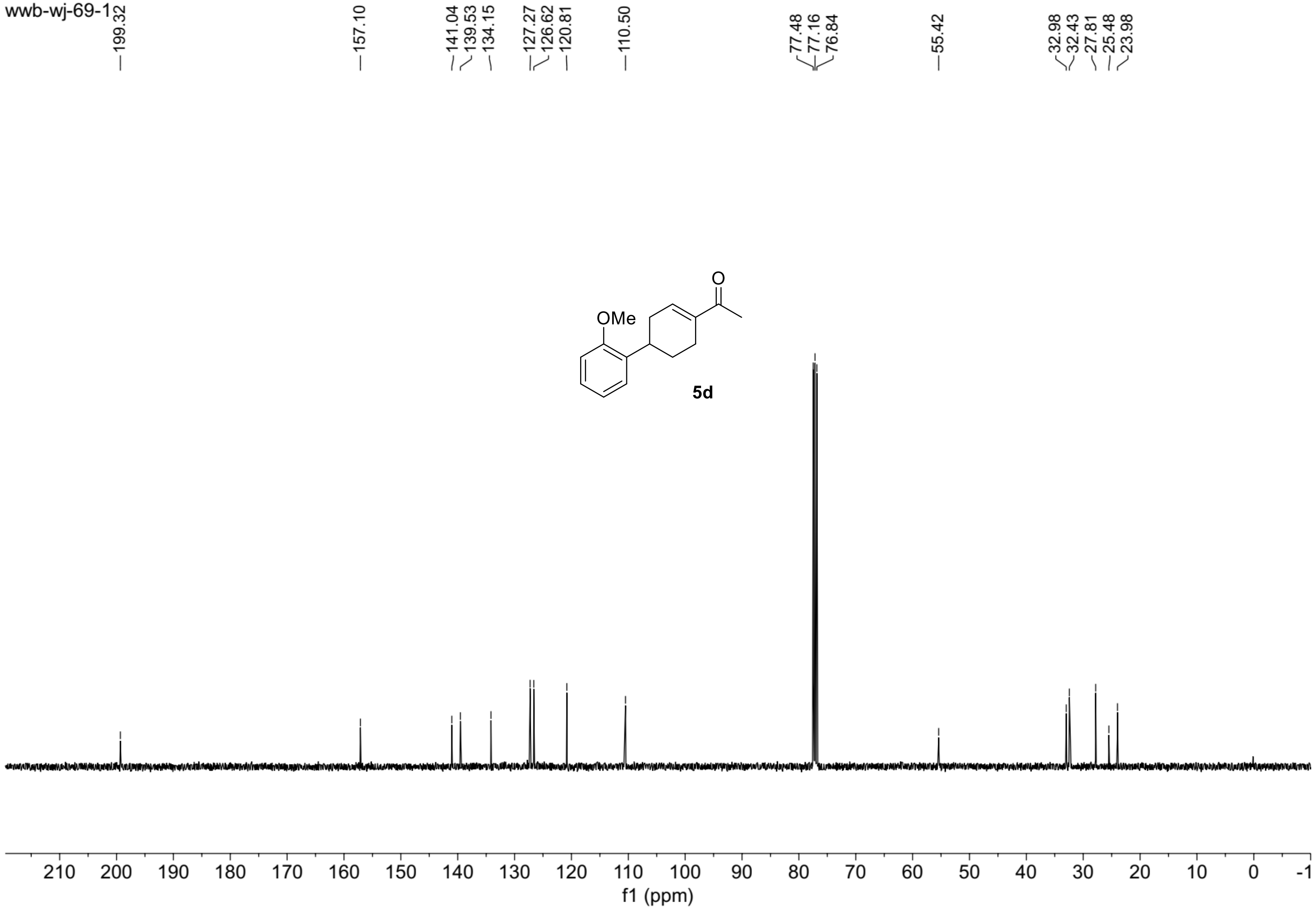
5b

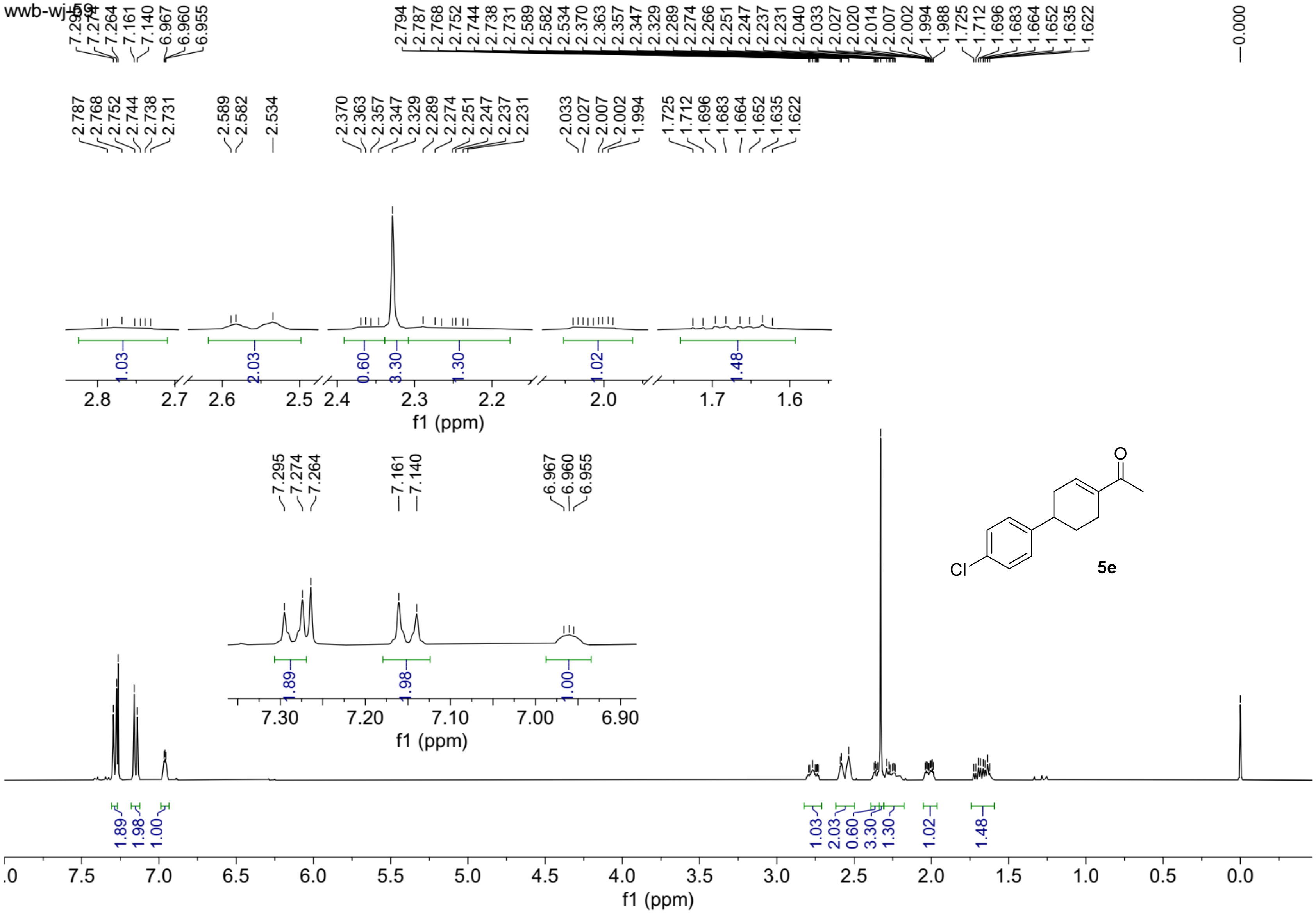

wwb-wj-57-C
199.20
143.08
140.35
139.60
135.99
129.34
126.78
77.48
77.16
76.84
38.98
34.39
29.49
25.47
23.91
21.11
O
H_3C
5b
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

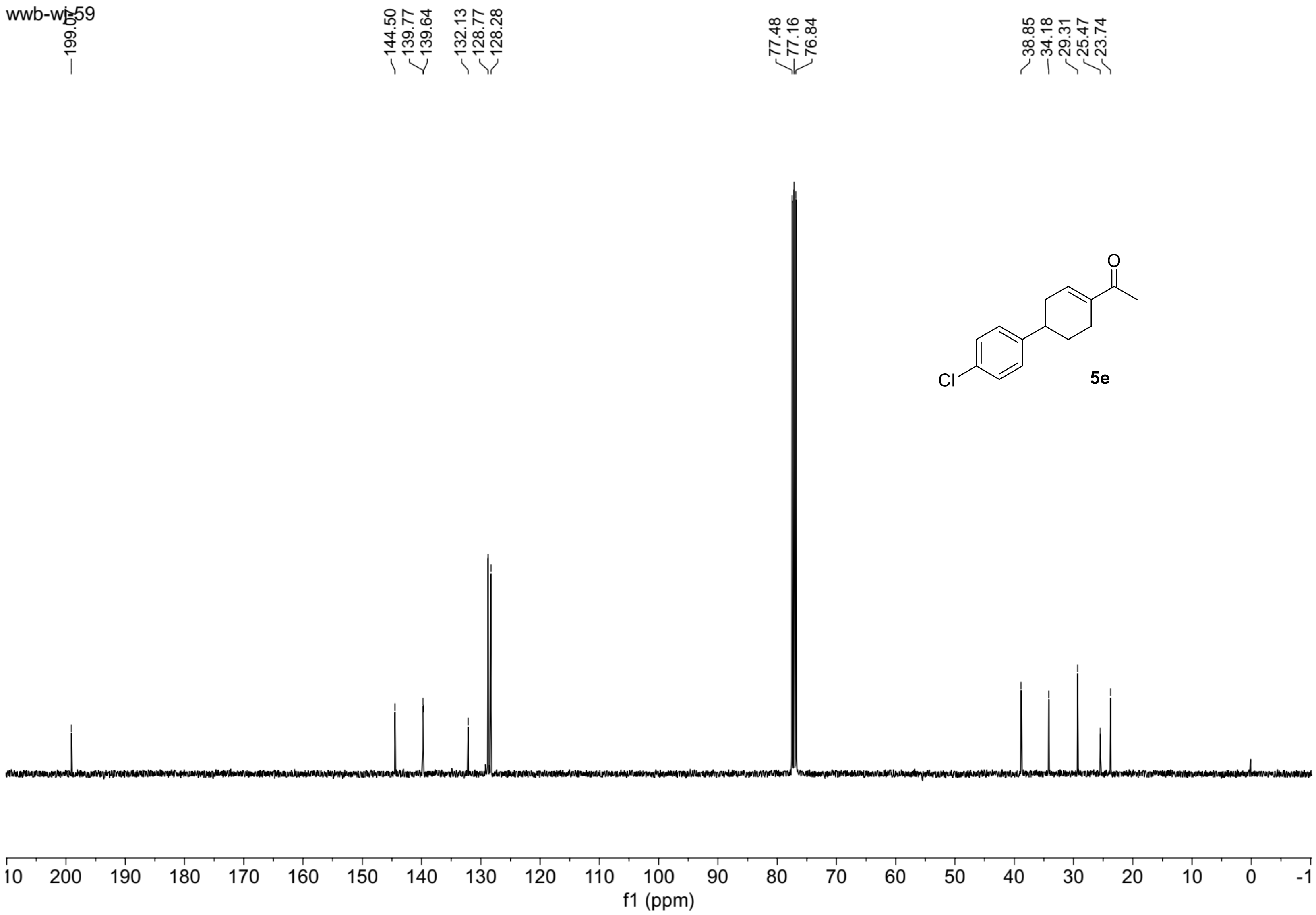

wwb-wj-83-H
7.260
7.150
7.129
6.972
6.966
6.962
6.874
6.852
3.796
2.776
2.757
2.738
2.701
2.585
2.576
2.541
2.528
2.521
2.378
2.368
2.351
2.335
2.322
2.273
2.266
2.257
2.247
2.242
2.233
2.227
2.043
2.030
2.011
1.997
1.985
1.717
1.704
1.687
1.674
1.656
1.645
1.626
1.613
0.000
1.99
1.00
2.13
3.03
1.02
2.08
5.43
1.01
1.37
f1 (ppm)
O
MeO
5c

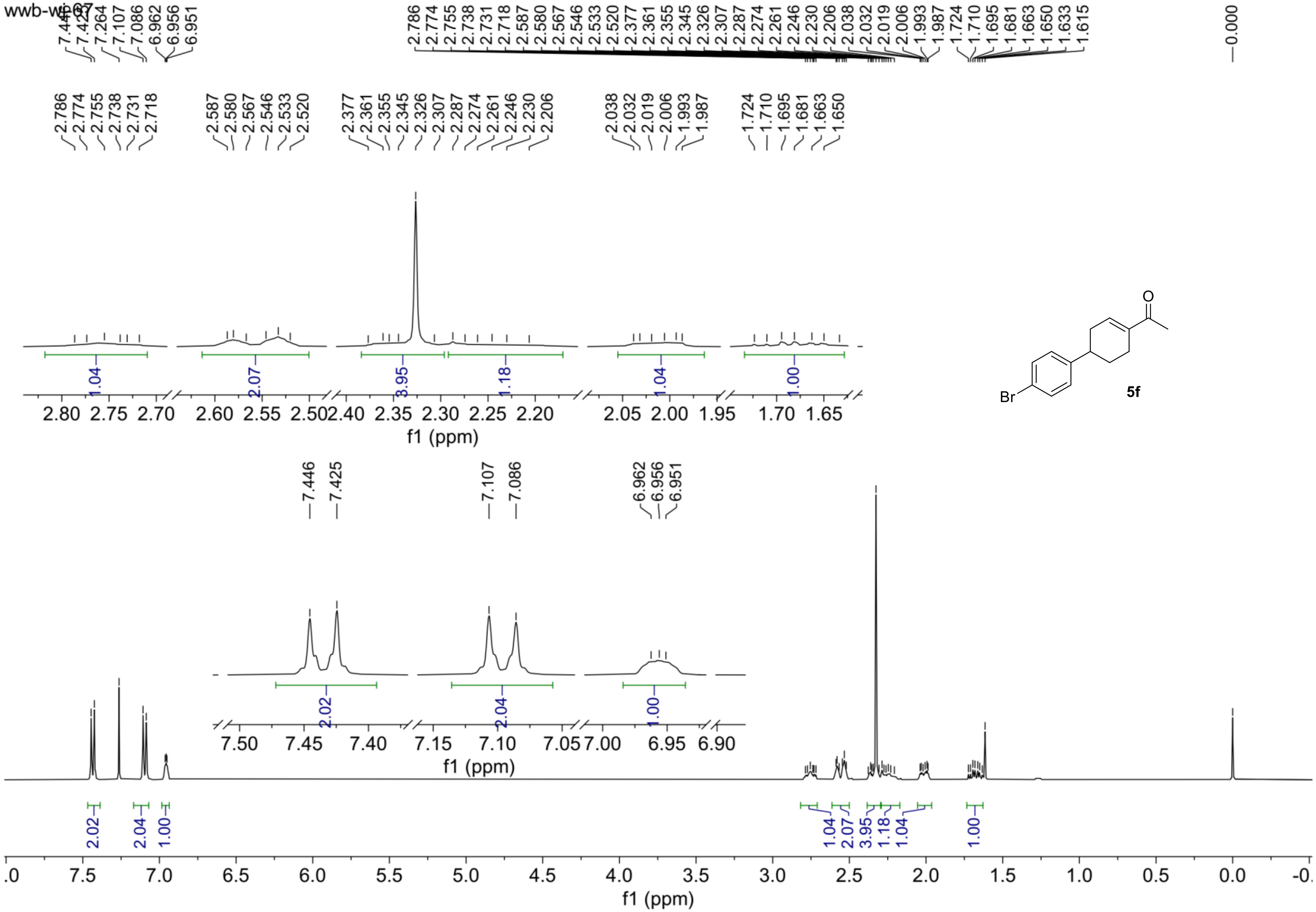

wwb-wj-83-C
199.16
158.23
140.28
139.62
138.22
127.78
114.06
77.48
77.16
76.84
55.42
38.54
34.49
29.63
25.46
23.91
O
MeO
5c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)


wwb-wj-69
7.260
7.228
7.224
7.205
7.189
7.185
7.174
7.170
7.155
7.151
6.999
6.986
6.958
6.939
6.920
6.886
6.866
3.827
3.241
3.234
3.229
3.210
3.199
3.179
3.173
3.166
2.635
2.599
2.586
2.577
2.545
2.538
2.533
2.327
2.295
2.283
2.273
2.267
2.246
2.231
2.206
1.997
1.984
1.971
1.965
1.953
1.946
1.940
1.803
1.790
1.774
1.760
1.743
1.732
1.713
1.700
0.000
2.02
3.16
1.02
2.04
3.36
1.94
1.04
1.03
3.00
f1 (ppm)
OMe
O
5d


wwb-wj-69-1
199.32
157.10
141.04
139.53
134.15
127.27
126.62
120.81
110.50
77.48
77.16
76.84
55.42
32.98
32.43
27.81
25.48
23.98
O
OMe
5d
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)

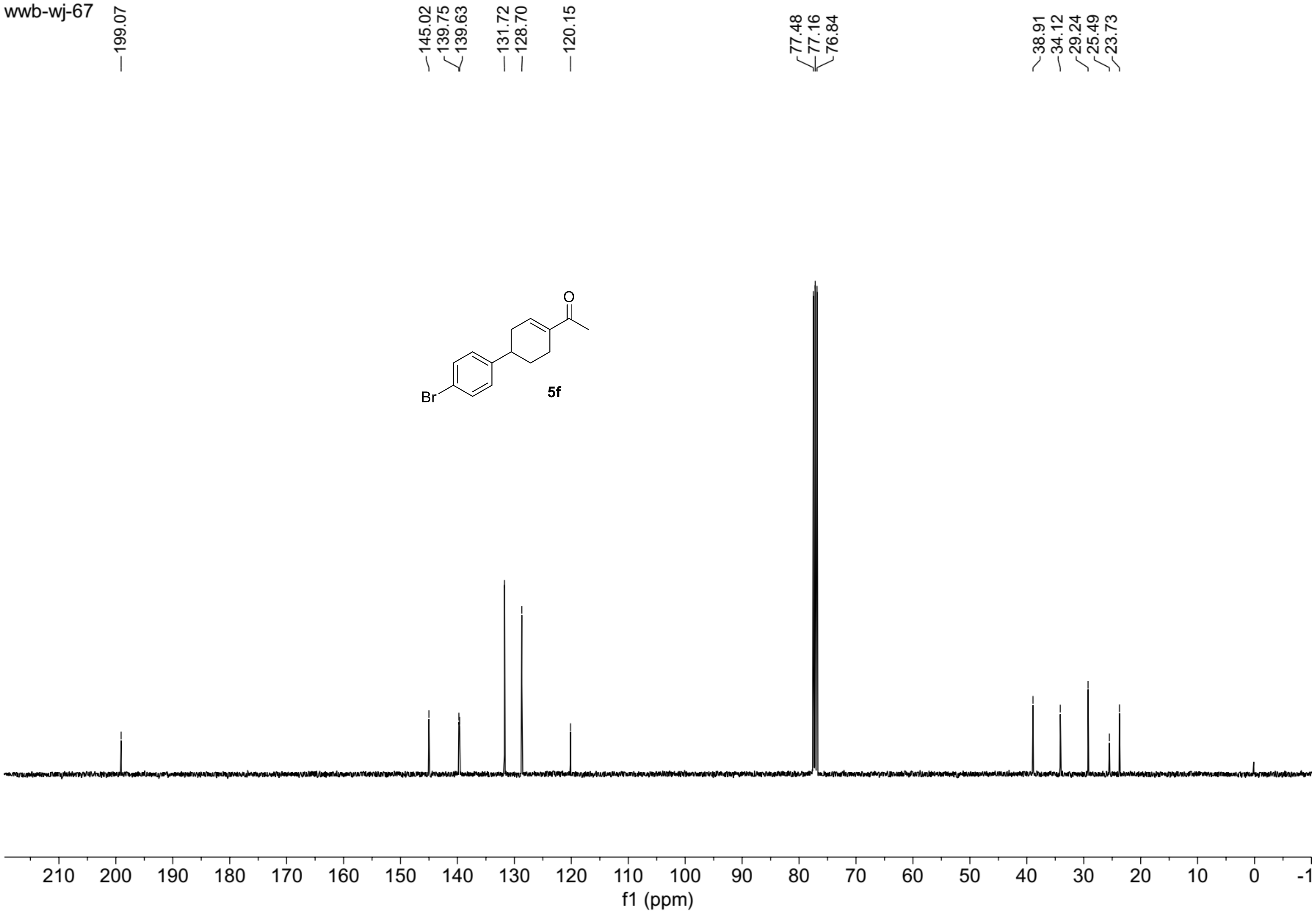

wwb-wj-59
7.295
7.274
7.264
7.161
7.140
6.967
6.960
6.955
2.794
2.787
2.768
2.752
2.744
2.738
2.731
2.589
2.582
2.534
2.370
2.363
2.357
2.347
2.329
2.289
2.274
2.266
2.251
2.247
2.237
2.231
2.040
2.033
2.027
2.020
2.014
2.007
2.002
1.994
1.988
1.725
1.712
1.696
1.683
1.664
1.652
1.635
1.622
0.000
1.03
2.03
0.60
3.30
1.30
1.02
1.48
1.89
1.98
1.00
f1 (ppm)
O
Cl
5e

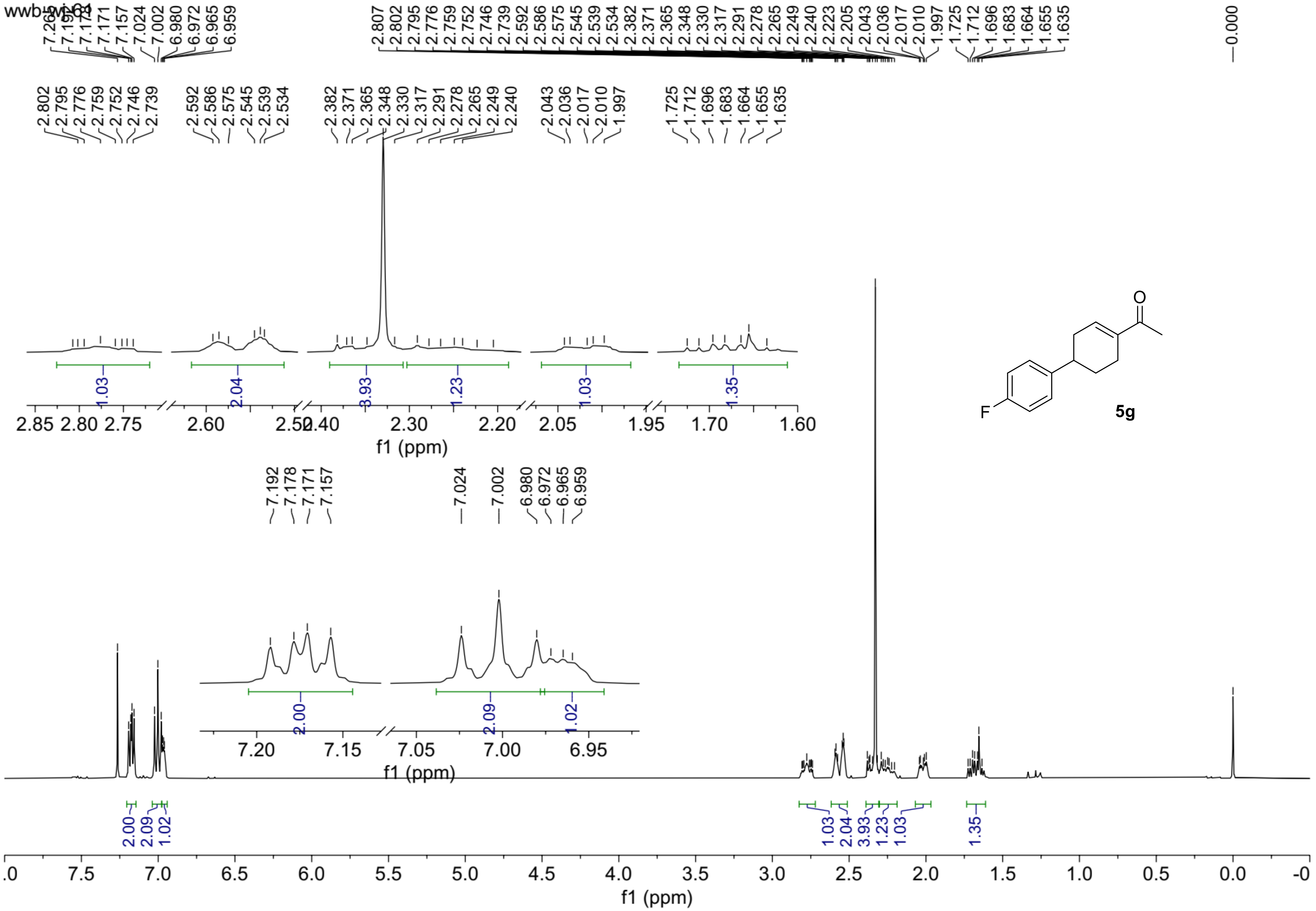

wwb-wj-59
199.07
144.50
139.77
139.64
132.13
128.77
128.28
77.48
77.16
76.84
38.85
34.18
29.31
25.47
23.74
O
Cl
5e
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

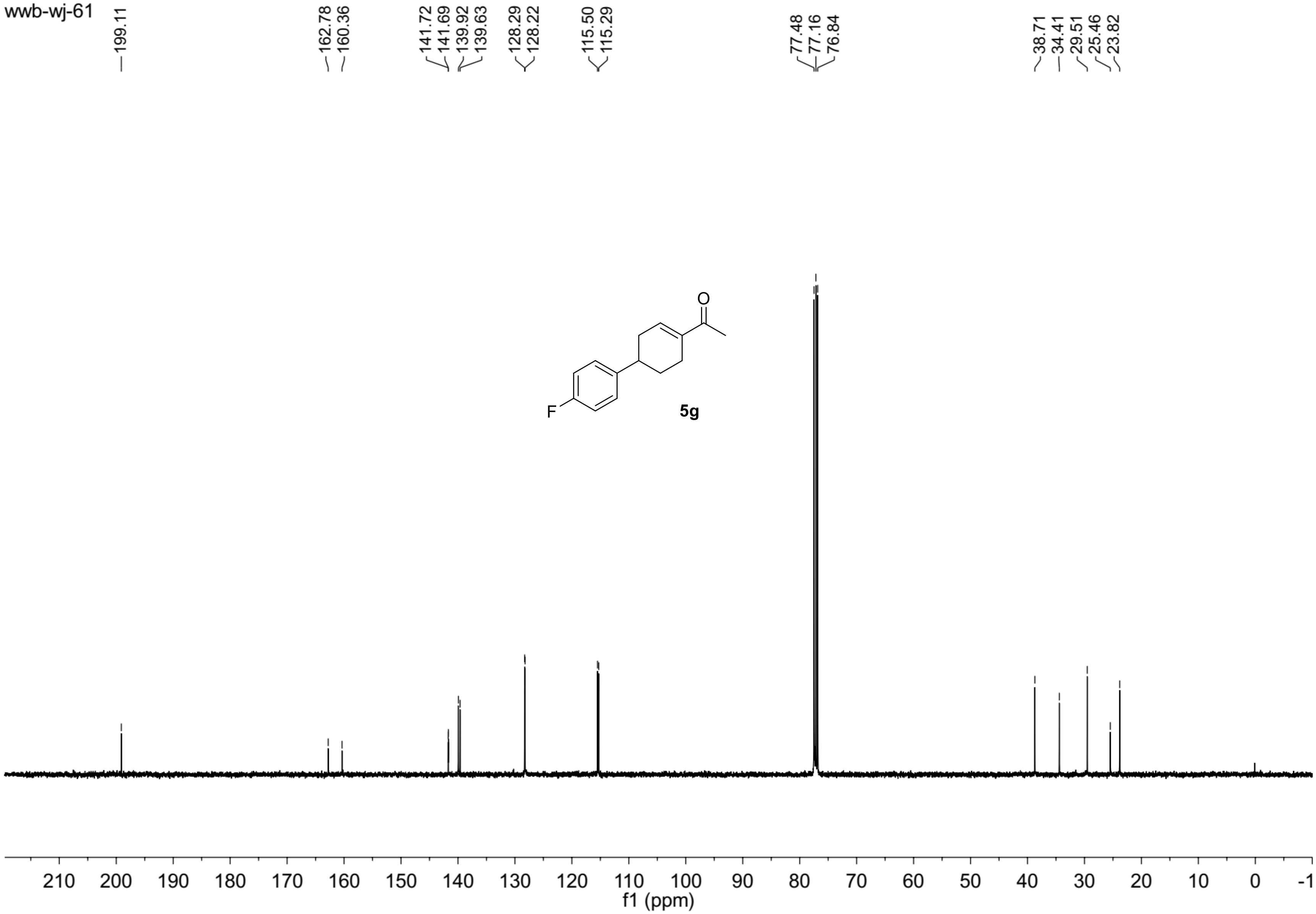


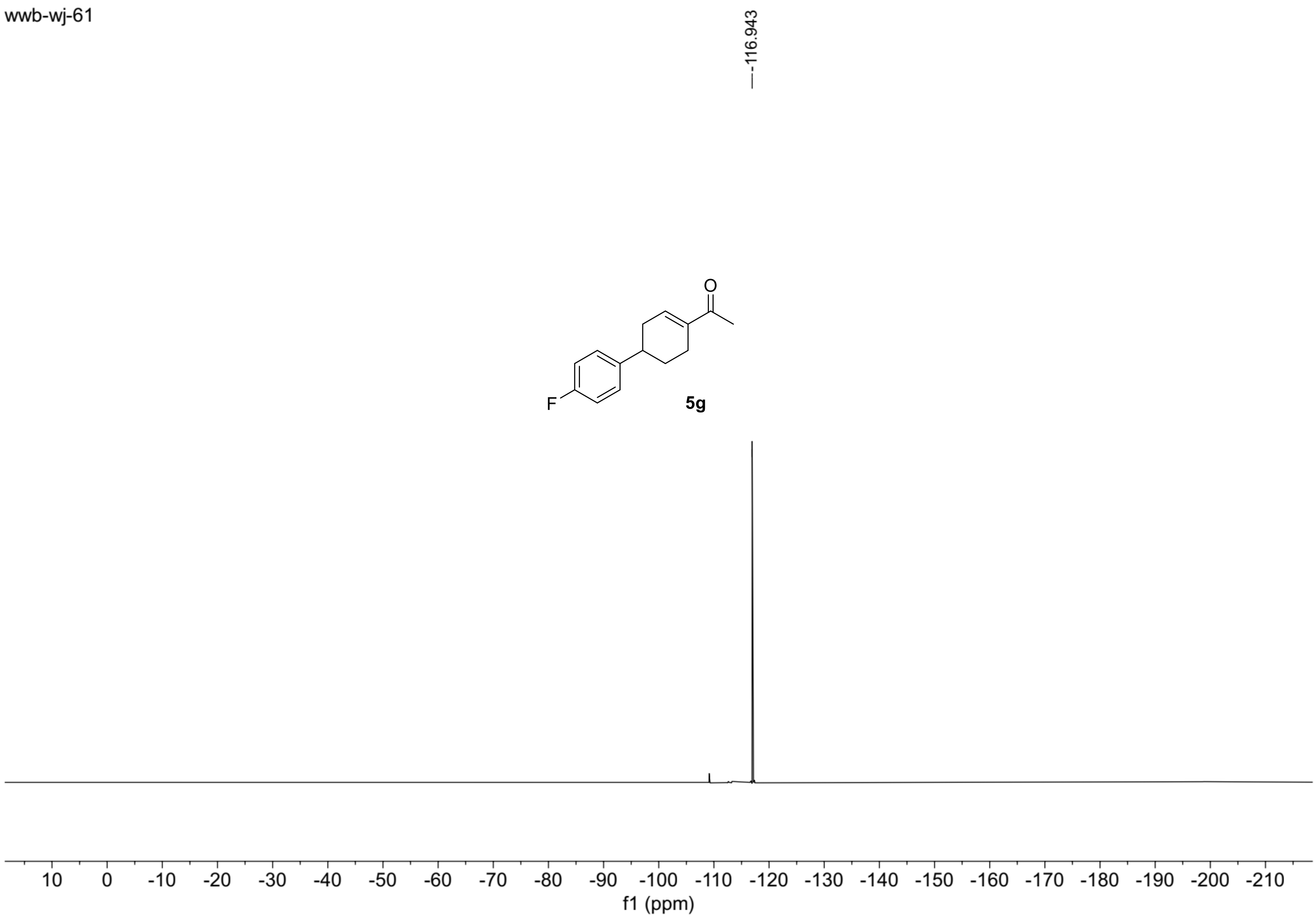
wwb-wj67
7.446
7.425
7.264
7.107
7.086
6.962
6.956
6.951
2.786
2.774
2.755
2.738
2.731
2.718
2.587
2.580
2.567
2.546
2.533
2.520
2.377
2.361
2.355
2.345
2.326
2.307
2.287
2.274
2.261
2.246
2.230
2.206
2.038
2.032
2.019
2.006
1.993
1.987
1.724
1.710
1.695
1.681
1.663
1.650
1.633
1.615
0.000
1.04
2.07
3.95
1.18
1.04
1.00
2.80 2.75 2.70
2.60 2.55 2.50
2.40 2.35 2.30 2.25 2.20
2.05 2.00 1.95
1.70 1.65
f1 (ppm)
2.02
2.04
1.00
7.50 7.45 7.40
7.15 7.10 7.05
7.00 6.95 6.90
f1 (ppm)
O
Br
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)

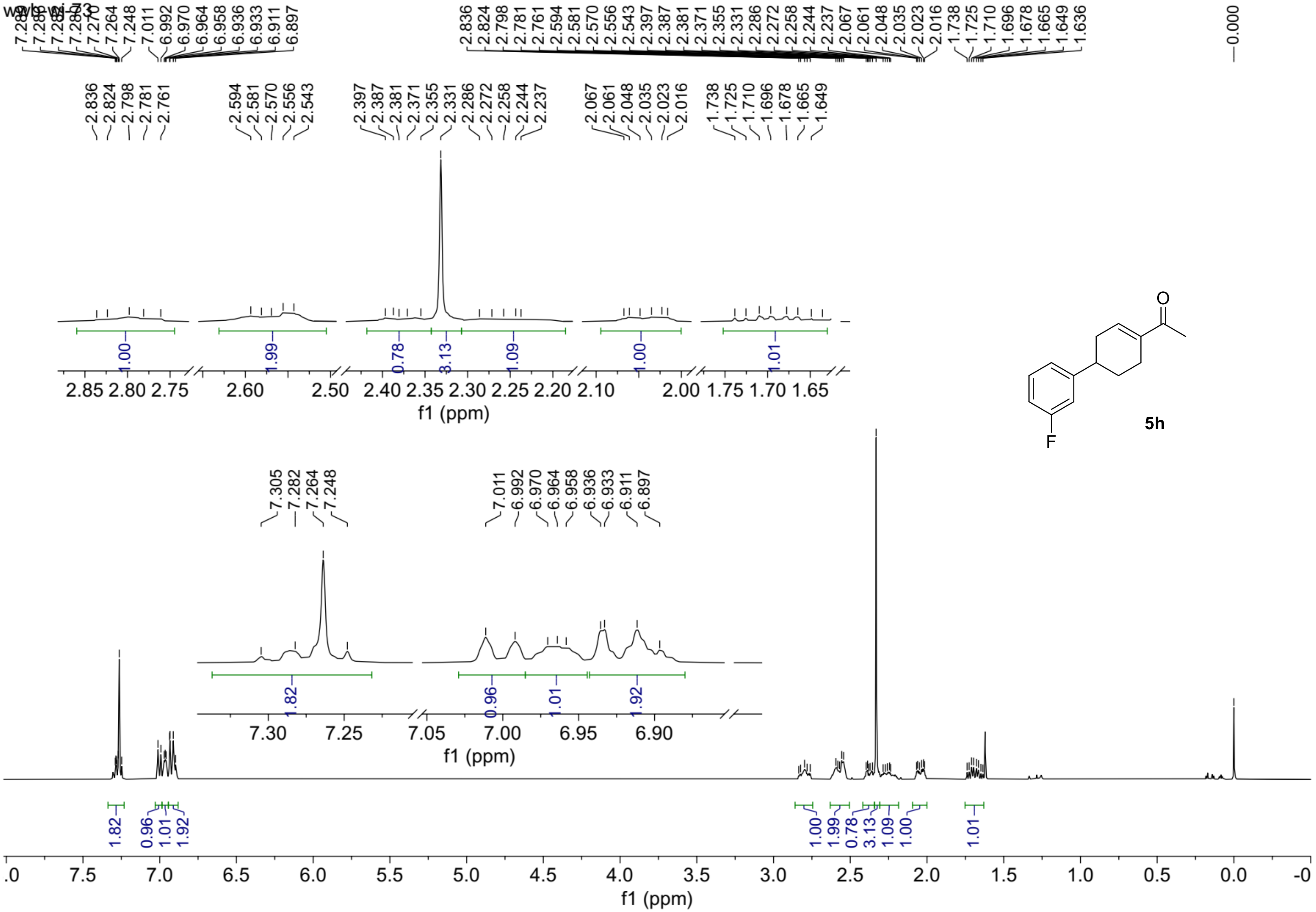
5f


wwb-wj-67
199.07
145.02
139.75
139.63
131.72
128.70
120.15
77.48
77.16
76.84
38.91
34.12
29.24
25.49
23.73
O
Br
5f
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)

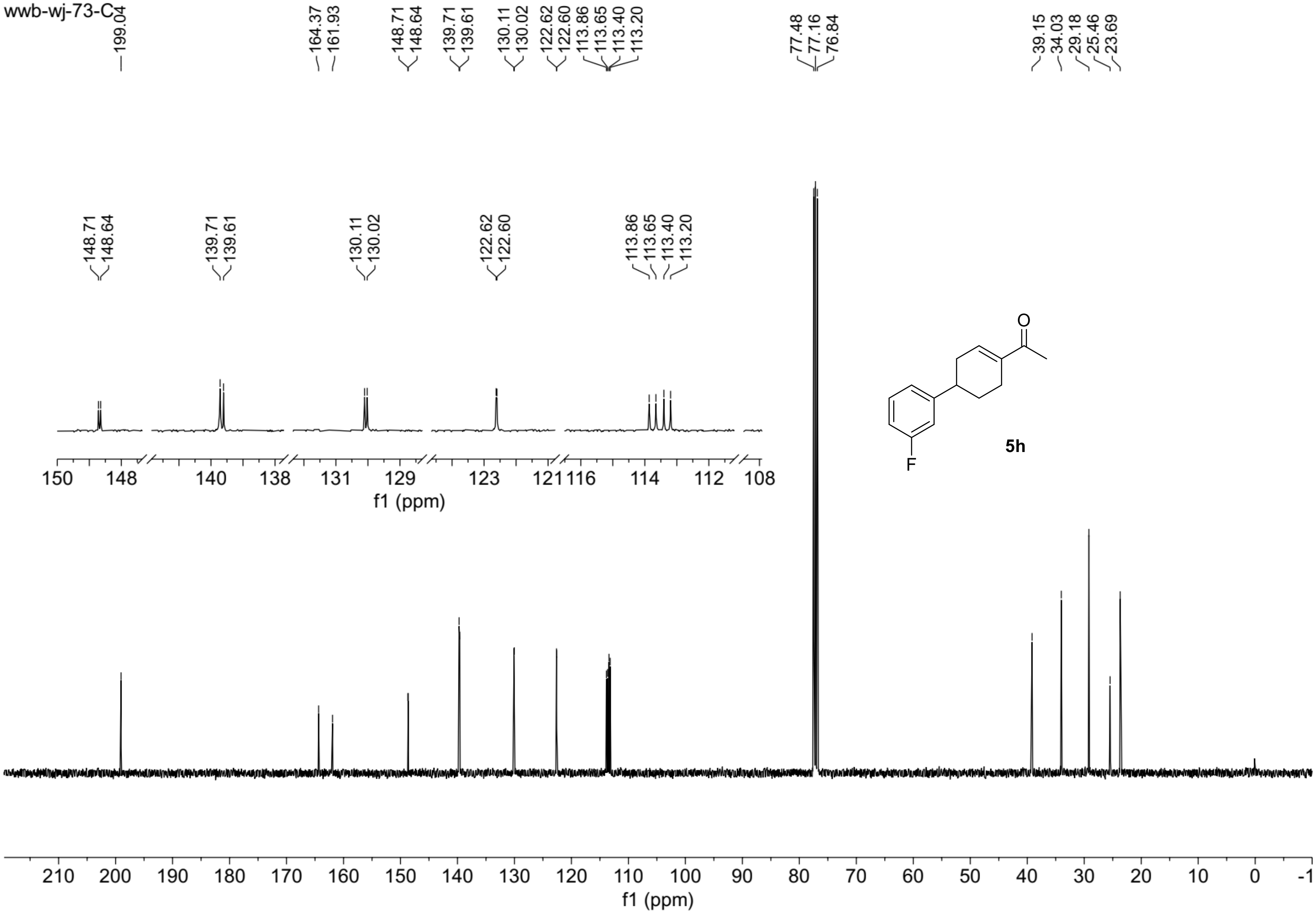

7.260 7.192 7.178 7.171 7.157 7.024 7.002 6.980 6.972 6.965 6.959
2.807 2.802 2.795 2.776 2.759 2.752 2.746 2.739 2.592 2.586 2.575 2.545 2.539 2.534 2.382 2.371 2.365 2.348 2.330 2.317 2.291 2.278 2.265 2.249 2.240 2.223 2.205 2.043 2.036 2.017 2.010 1.997 1.725 1.712 1.696 1.683 1.664 1.655 1.635
0.000
1.03
2.04
3.93
1.23
1.03
1.35
2.00
2.09
1.02
f1 (ppm)
O
F
5g

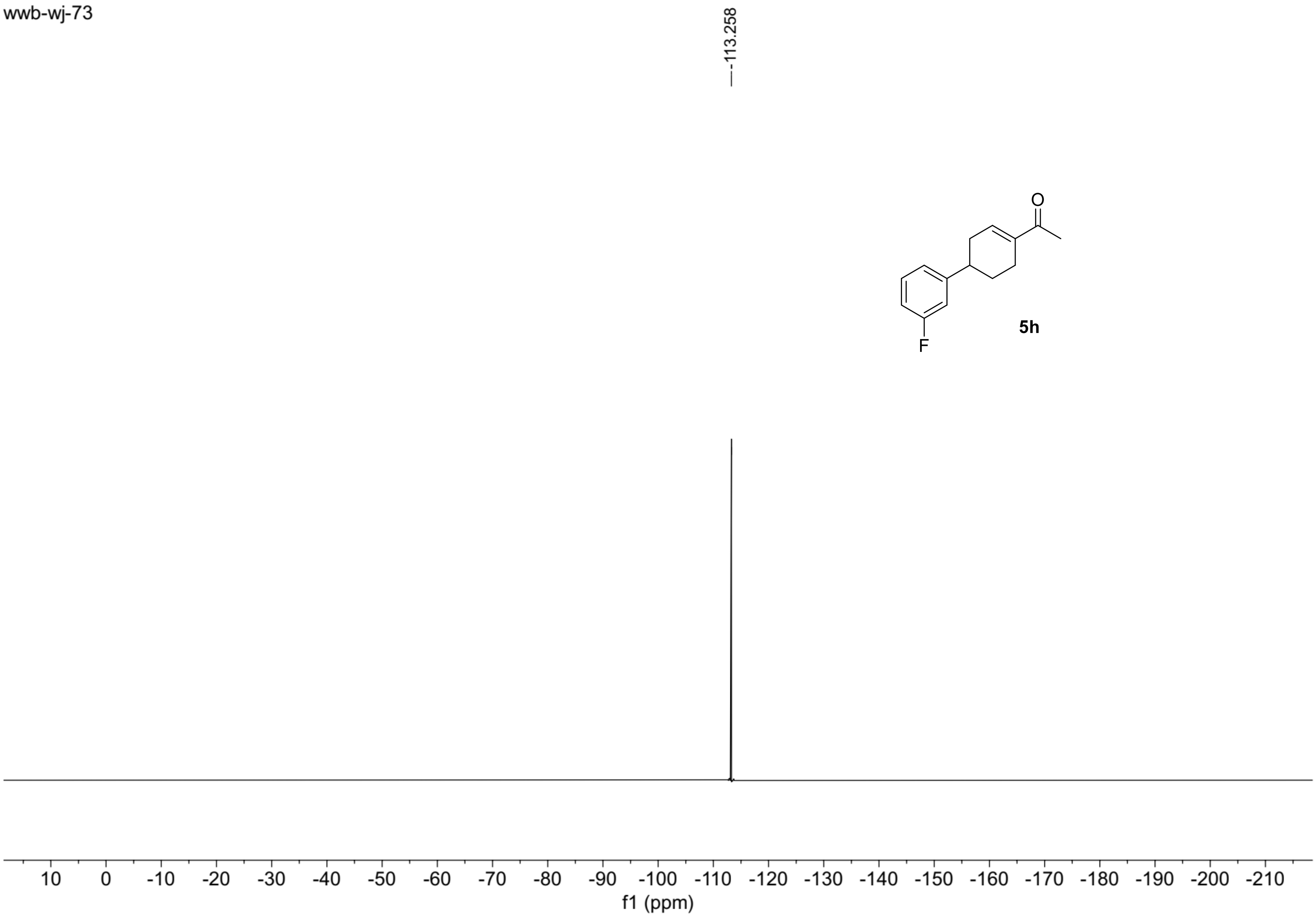

wwb-wj-61
199.11
162.78
160.36
141.72
141.69
139.92
139.63
128.29
128.22
115.50
115.29
77.48
77.16
76.84
38.71
34.41
29.51
25.46
23.82
O
F
5g
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

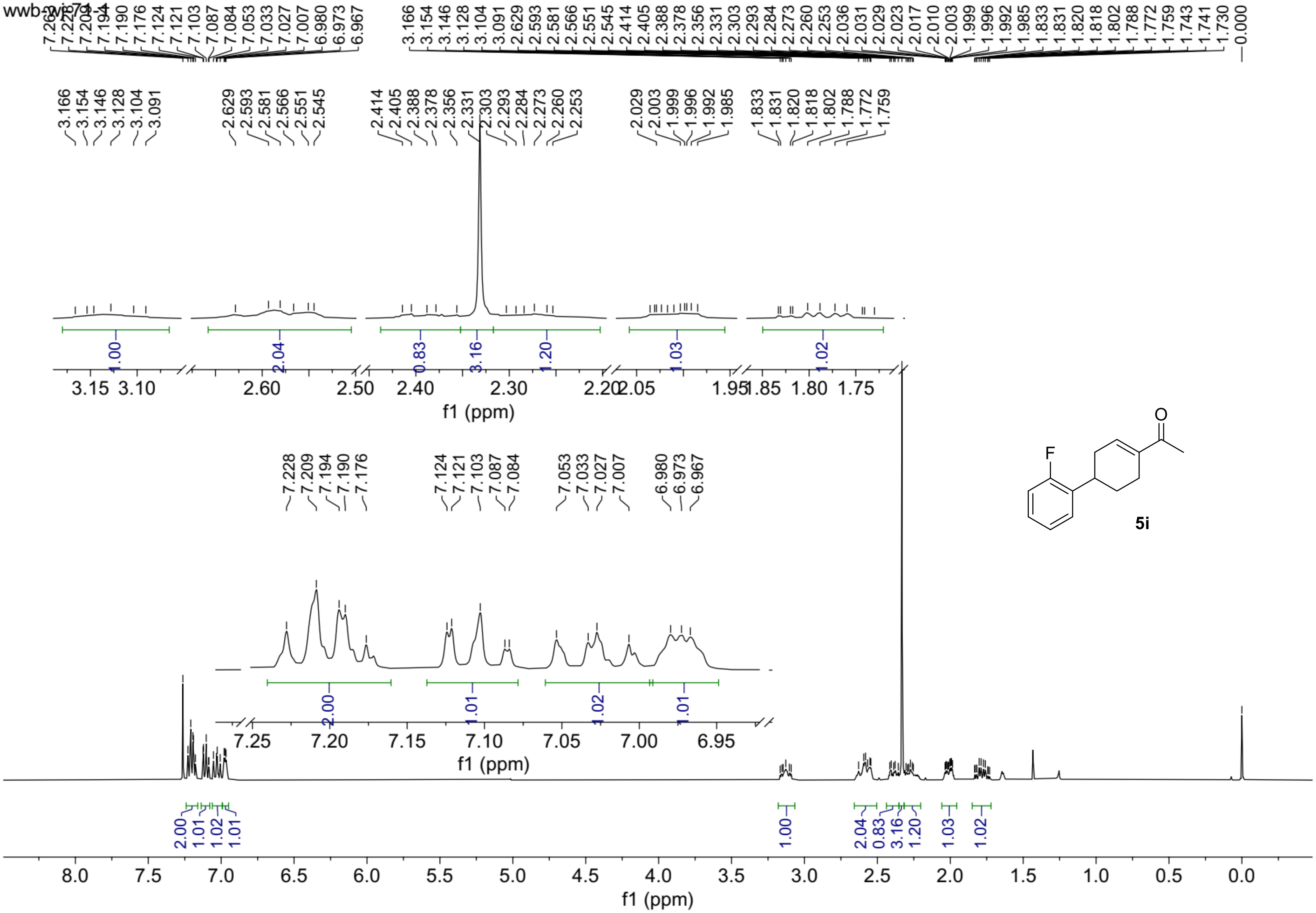

wwb-wj-61
-116.943
O
F
5g
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

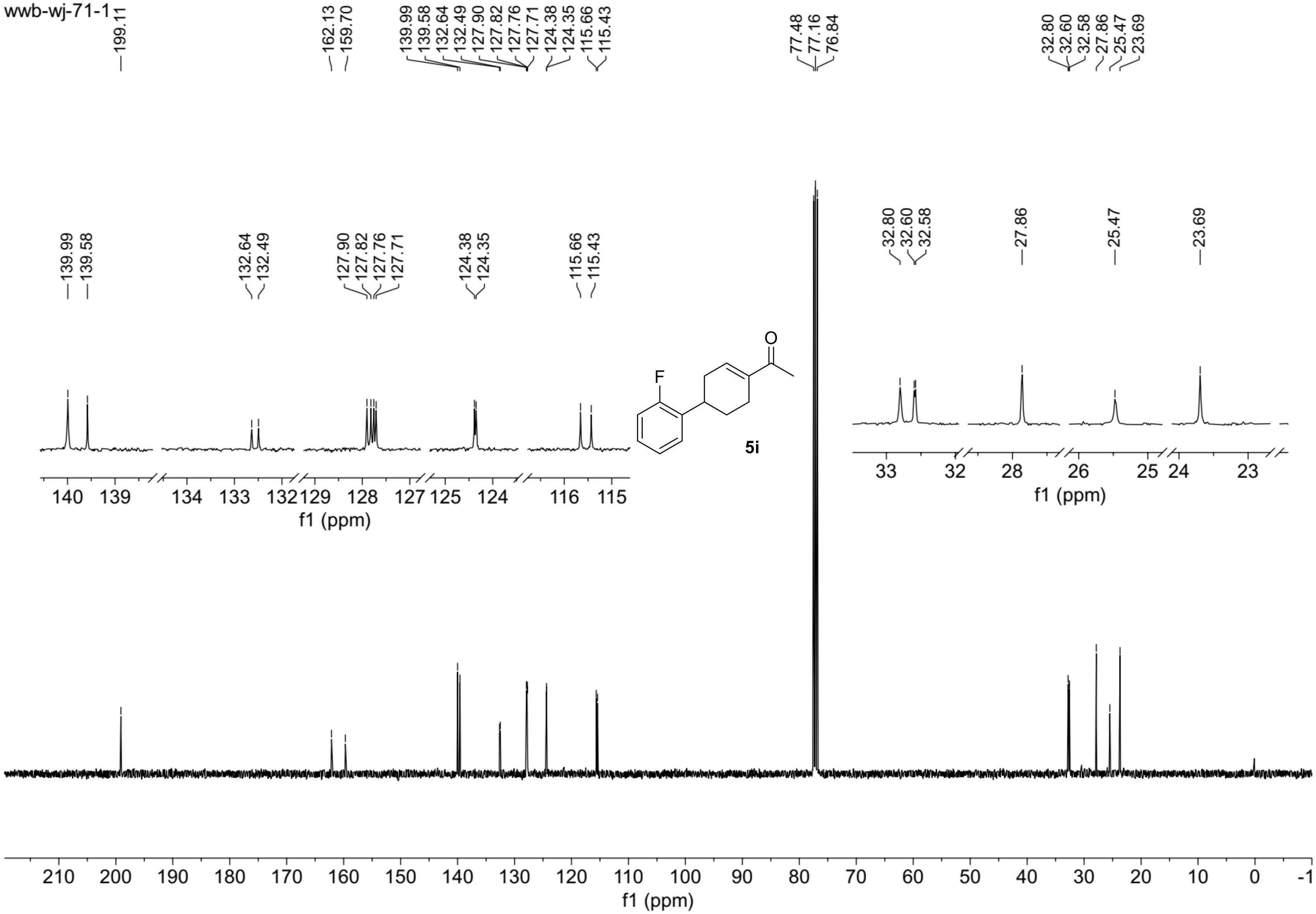

wgk-wj-73
7.305 7.282 7.270 7.264 7.248 7.011 6.992 6.970 6.964 6.958 6.936 6.933 6.911 6.897
2.836 2.824 2.798 2.781 2.761 2.594 2.581 2.570 2.556 2.543 2.397 2.387 2.381 2.371 2.355 2.331 2.286 2.272 2.258 2.244 2.237 2.067 2.061 2.048 2.035 2.023 2.016 1.738 1.725 1.710 1.696 1.678 1.665 1.649 1.636
0.000
1.00
1.99
0.78
3.13
1.09
1.00
1.01
2.85 2.80 2.75 2.60 2.50 2.40 2.35 2.30 2.25 2.20 2.10 2.00 1.75 1.70 1.65
f1 (ppm)
1.82
0.96
1.01
1.92
7.30 7.25 7.05 7.00 6.95 6.90
f1 (ppm)
O
F
.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)

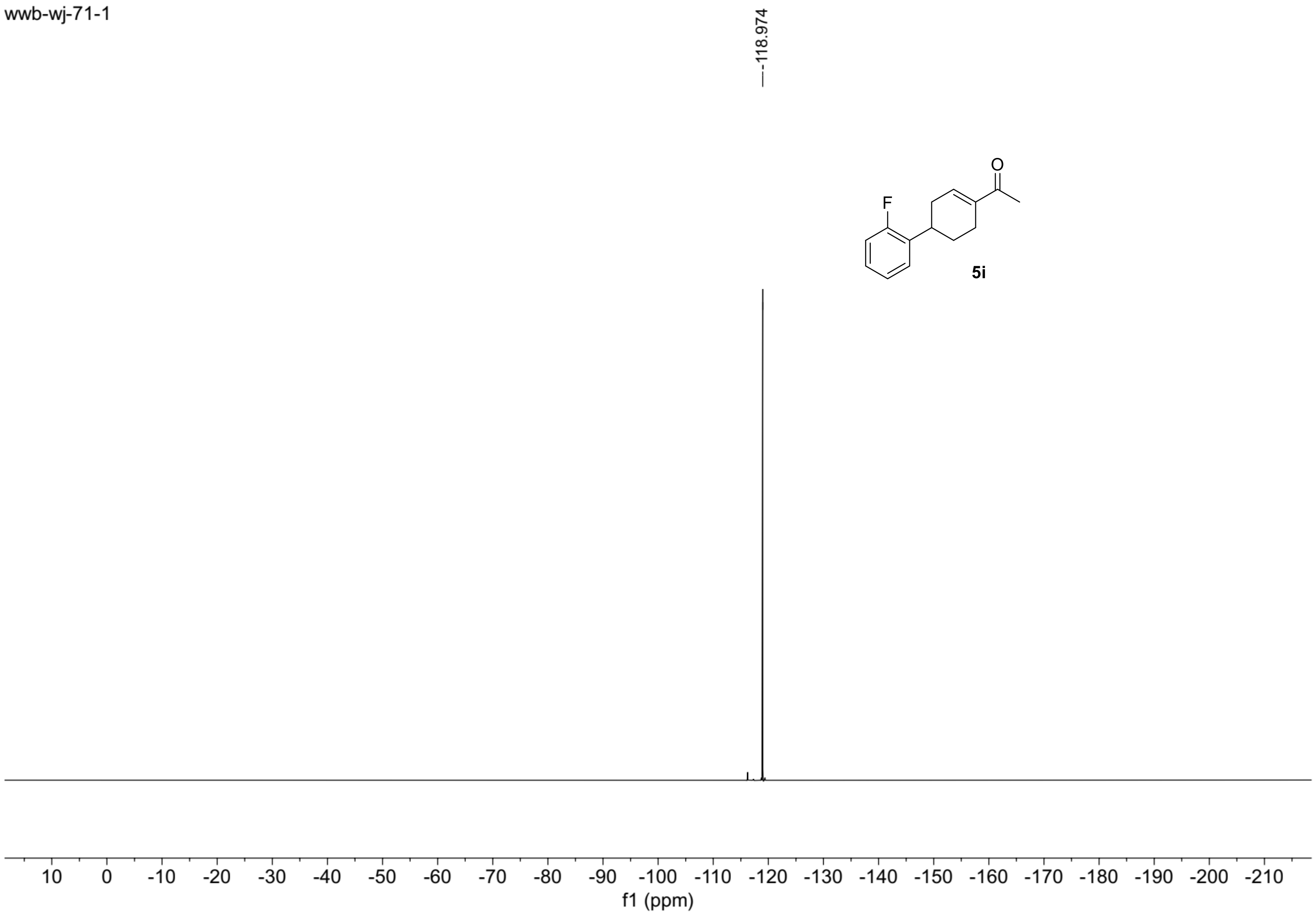
5h

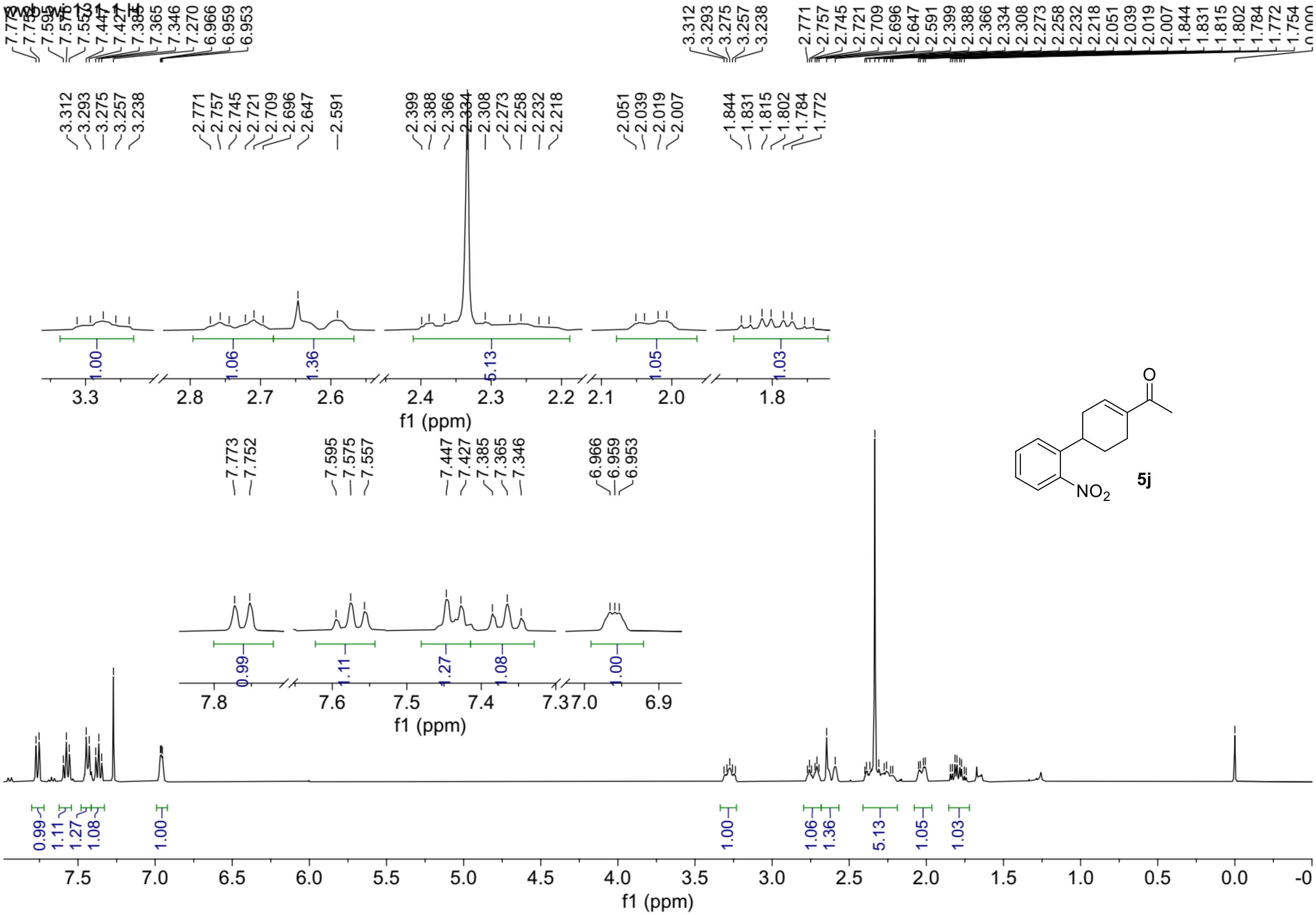

wwb-wj-73-C
199.04
164.37
161.93
148.71
148.64
139.71
139.61
130.11
130.02
122.62
122.60
113.86
113.65
113.40
113.20
77.48
77.16
76.84
39.15
34.03
29.18
25.46
23.69
150 148 140 138 131 129 123 121 116 114 112 108
f1 (ppm)
O
F
5h
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)


wwb-wj-73
-113.258
O
F
5h
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

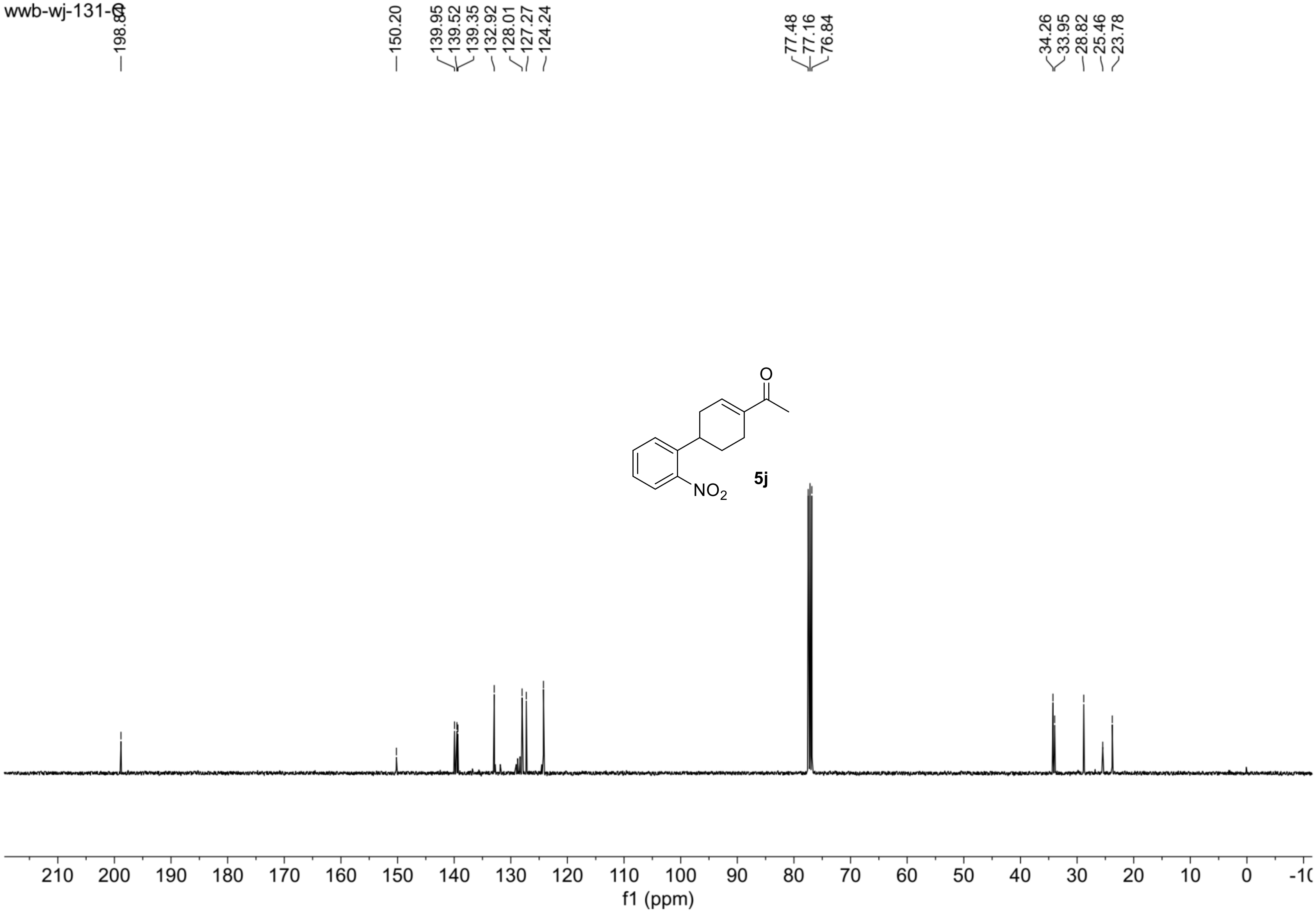

wwb-wjc71-1
7.228
7.209
7.194
7.190
7.176
7.124
7.121
7.103
7.087
7.084
7.053
7.033
7.027
7.007
6.980
6.973
6.967
3.166
3.154
3.146
3.128
3.104
3.091
2.629
2.593
2.581
2.566
2.551
2.545
2.414
2.405
2.388
2.378
2.356
2.331
2.303
2.293
2.284
2.273
2.260
2.253
2.036
2.031
2.029
2.023
2.017
2.010
2.003
1.999
1.996
1.992
1.985
1.833
1.831
1.820
1.818
1.802
1.788
1.772
1.759
1.743
1.741
1.730
0.000
2.00
1.01
1.02
1.01
1.00
2.04
0.83
3.16
1.20
1.03
1.02
f1 (ppm)
F
O
5i

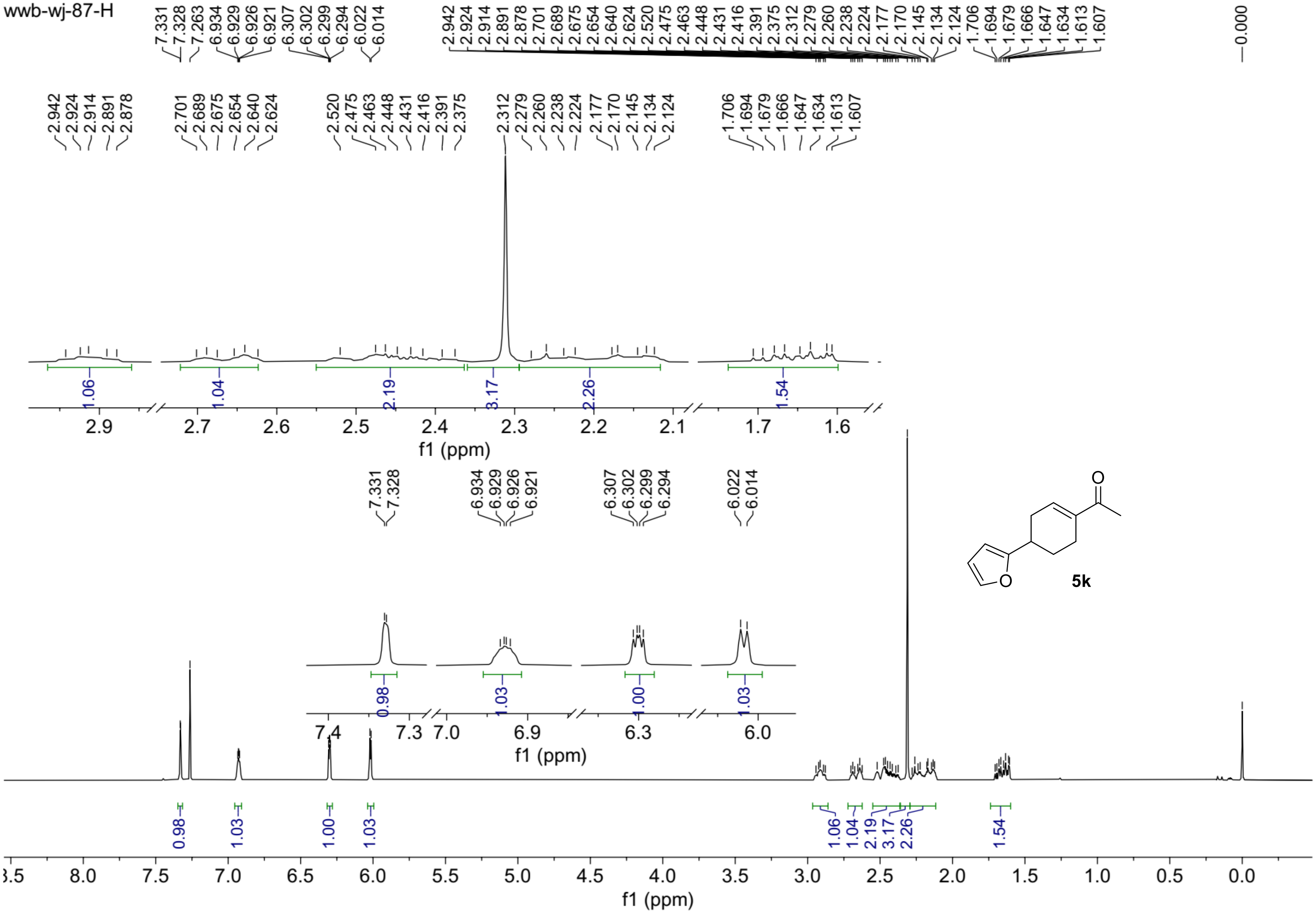

wwb-wj-71-1
199.11
162.13
159.70
139.99
139.58
132.64
132.49
127.90
127.82
127.76
127.71
124.38
124.35
115.66
115.43
77.48
77.16
76.84
32.80
32.60
32.58
27.86
25.47
23.69
F
O
5i
140 139 134 133 132 129 128 127 125 124 116 115
33 32 28 26 25 24 23
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

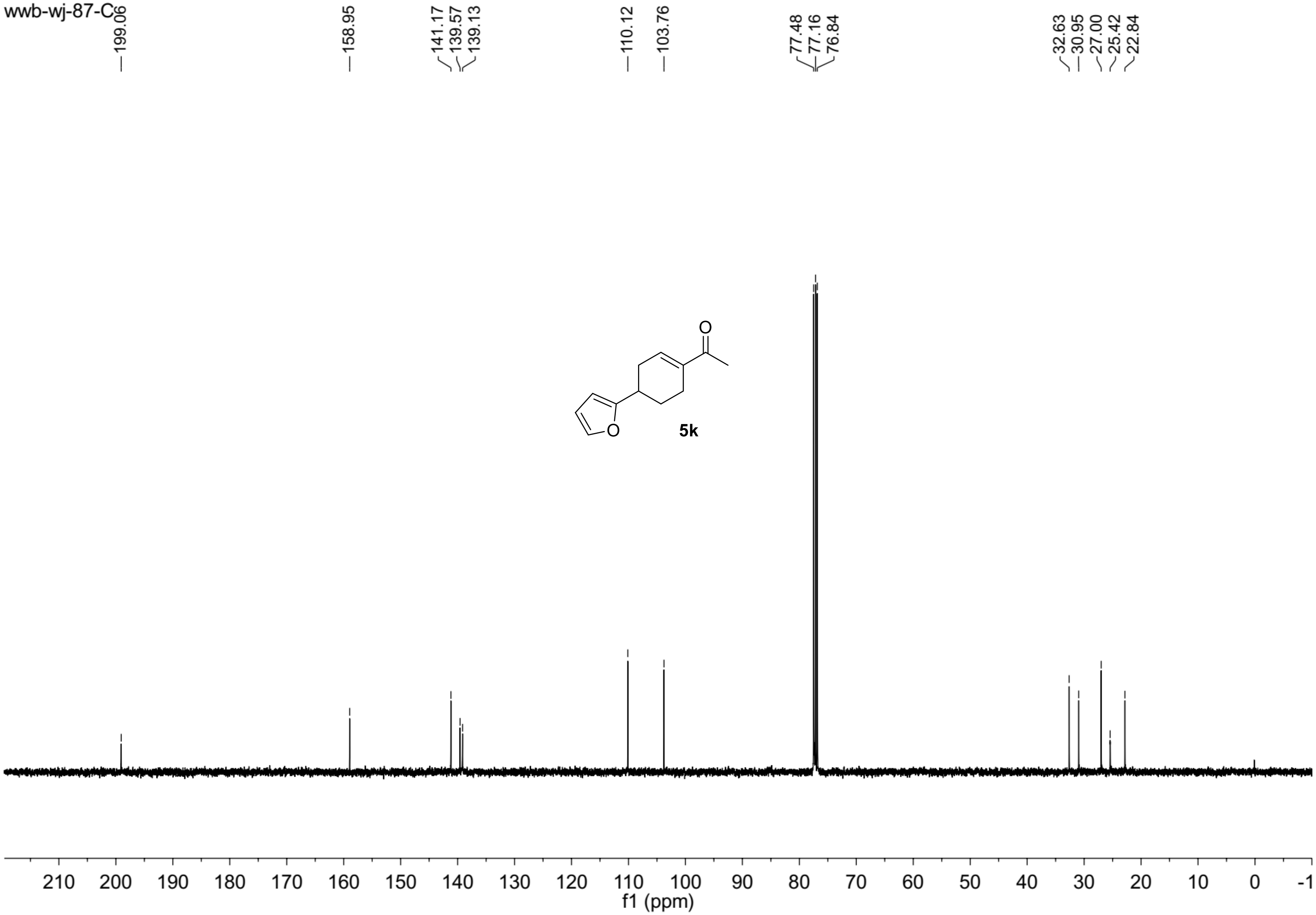

wwb-wj-71-1
-118.974
F
O
5i
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

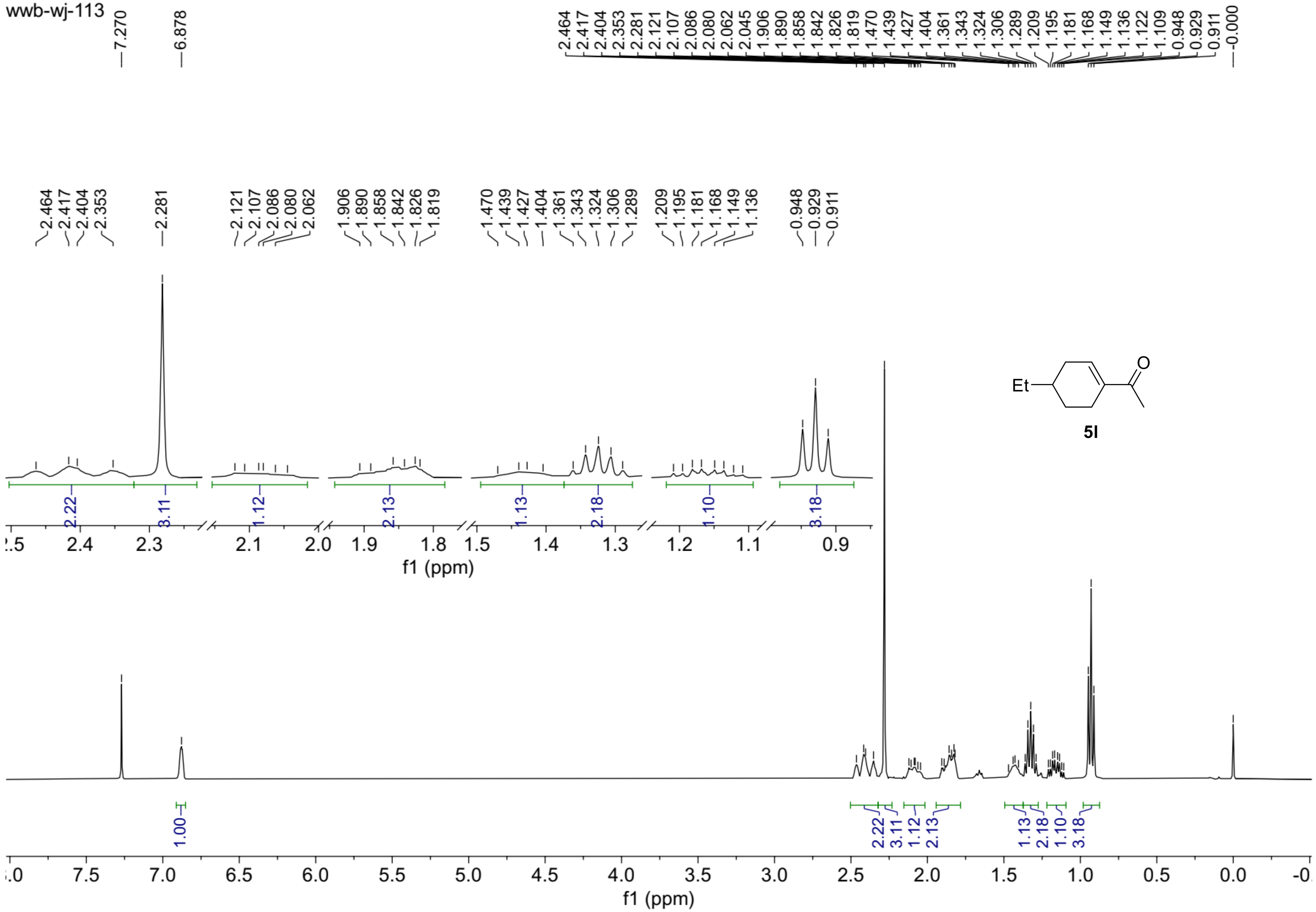

wyb-wj-131-1-H
3.312 3.293 3.275 3.257 3.238
2.771 2.757 2.745 2.721 2.709 2.696 2.647 2.591
2.399 2.388 2.366 2.334 2.308 2.273 2.258 2.232 2.218
2.051 2.039 2.019 2.007
1.844 1.831 1.815 1.802 1.784 1.772 1.754
0.000
1.00 1.06 1.36 5.13 1.05 1.03
3.3 2.8 2.7 2.6 2.4 2.3 2.2 2.1 2.0 1.8
f1 (ppm)
7.773 7.752
7.595 7.575 7.557
7.447 7.427 7.385 7.365 7.346
7.270
6.966 6.959 6.953
0.99 1.11 1.27 1.08 1.00
7.8 7.6 7.5 7.4 7.3 7.0 6.9
f1 (ppm)
O
NO2
0.99 1.11 1.27 1.08 1.00 1.00 1.06 1.36 5.13 1.05 1.03
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)

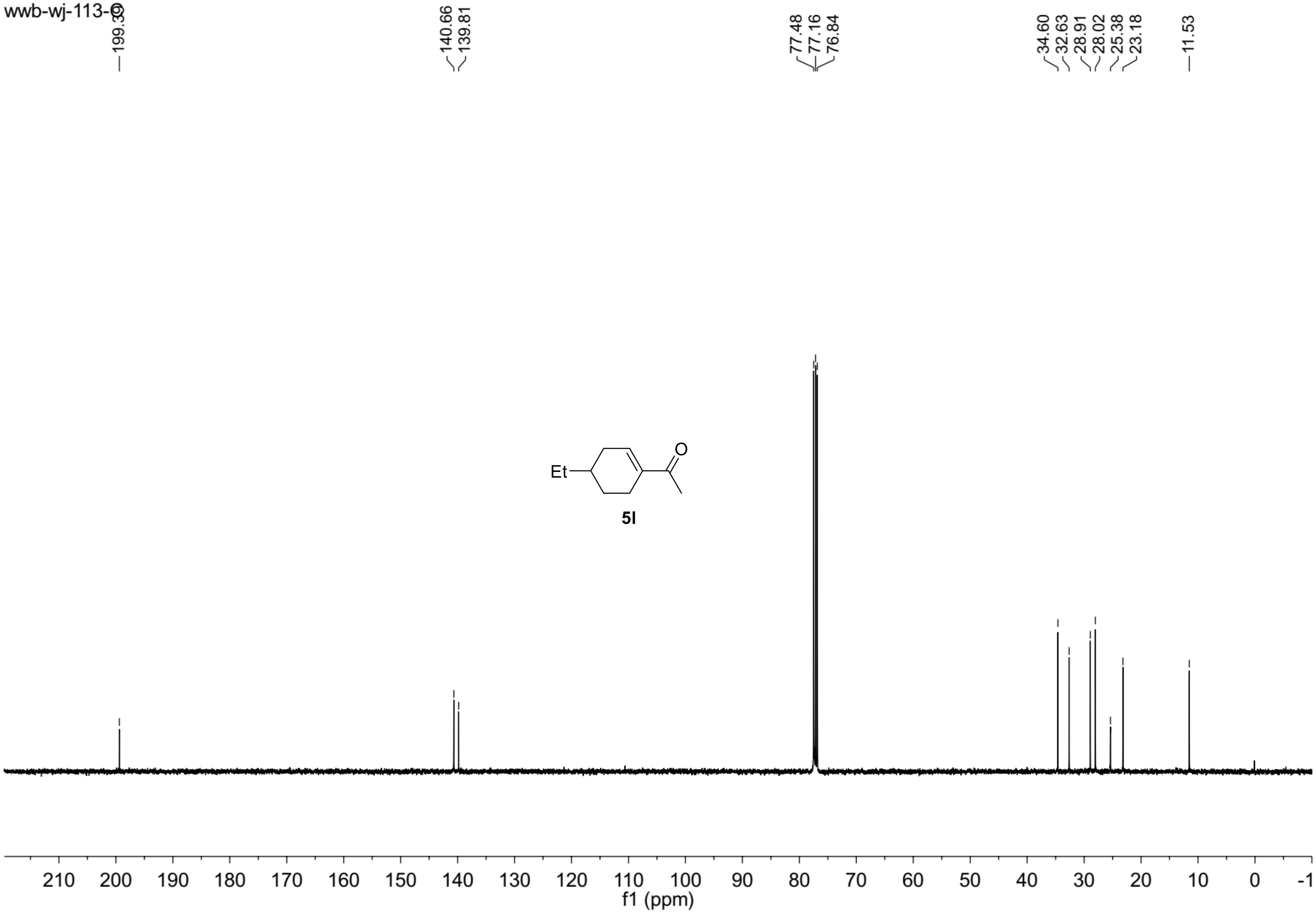
5j

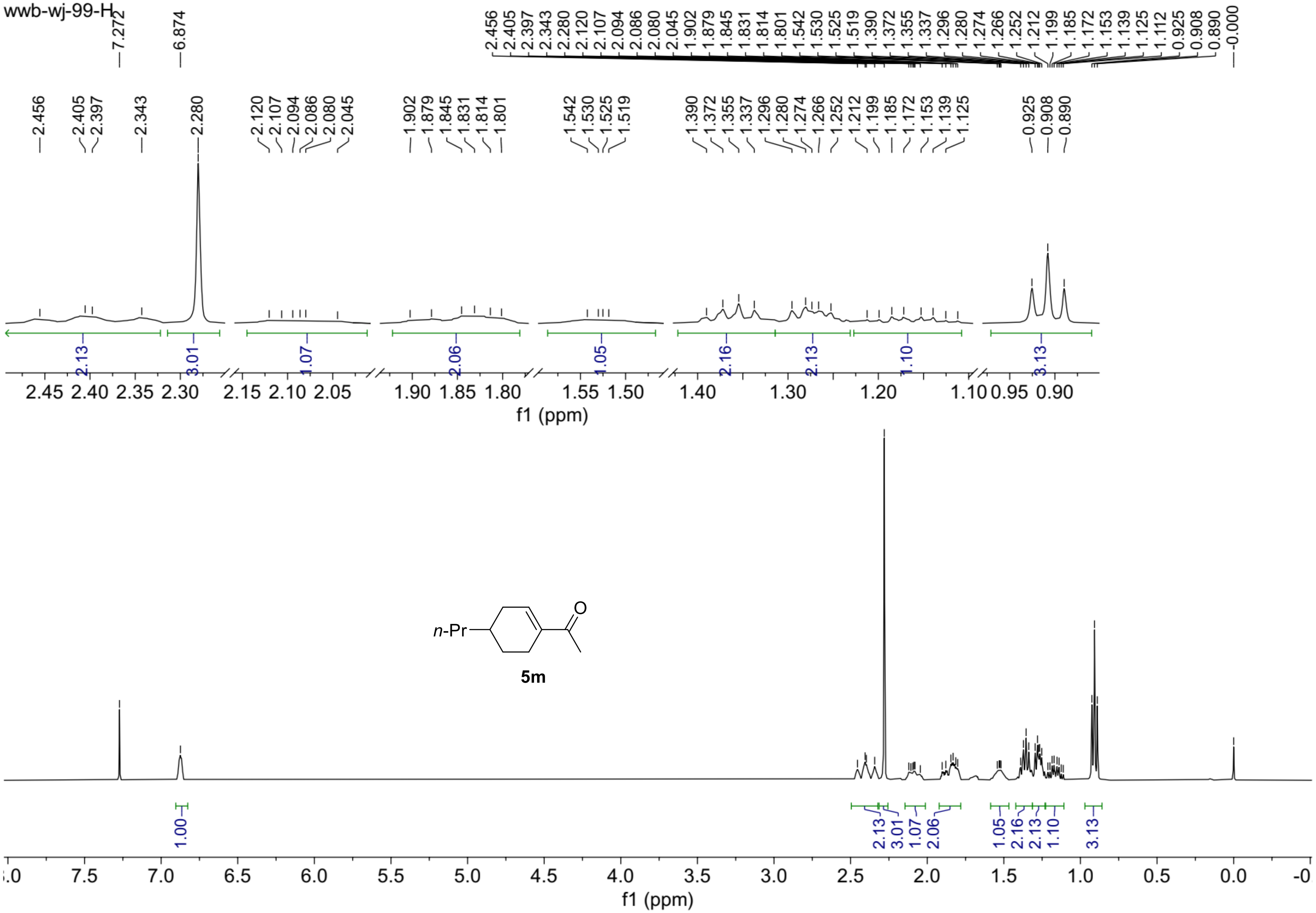

wwb-wj-131-C
198.84
150.20
139.95
139.52
139.35
132.92
128.01
127.27
124.24
77.48
77.16
76.84
34.26
33.95
28.82
25.46
23.78
O
NO_2
5j
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1(
f1 (ppm)

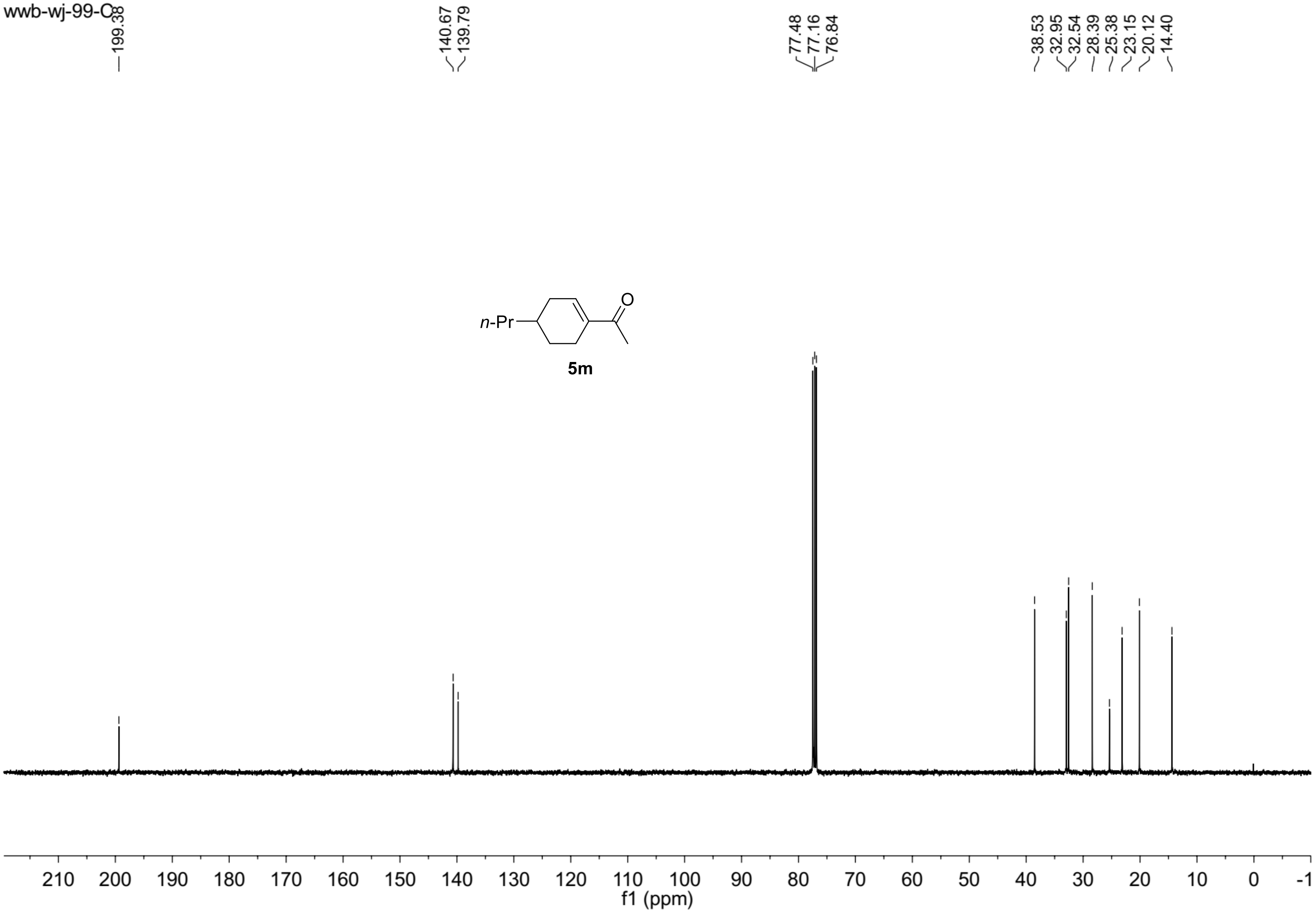

wwb-wj-87-H
7.331
7.328
7.263
6.934
6.929
6.926
6.921
6.307
6.302
6.299
6.294
6.022
6.014
2.942
2.924
2.914
2.891
2.878
2.701
2.689
2.675
2.654
2.640
2.624
2.520
2.475
2.463
2.448
2.431
2.416
2.391
2.375
2.312
2.279
2.260
2.238
2.224
2.177
2.170
2.145
2.134
2.124
1.706
1.694
1.679
1.666
1.647
1.634
1.613
1.607
0.000
1.06
1.04
2.19
3.17
2.26
1.54
0.98
1.03
1.00
1.03
f1 (ppm)
O
O
5k

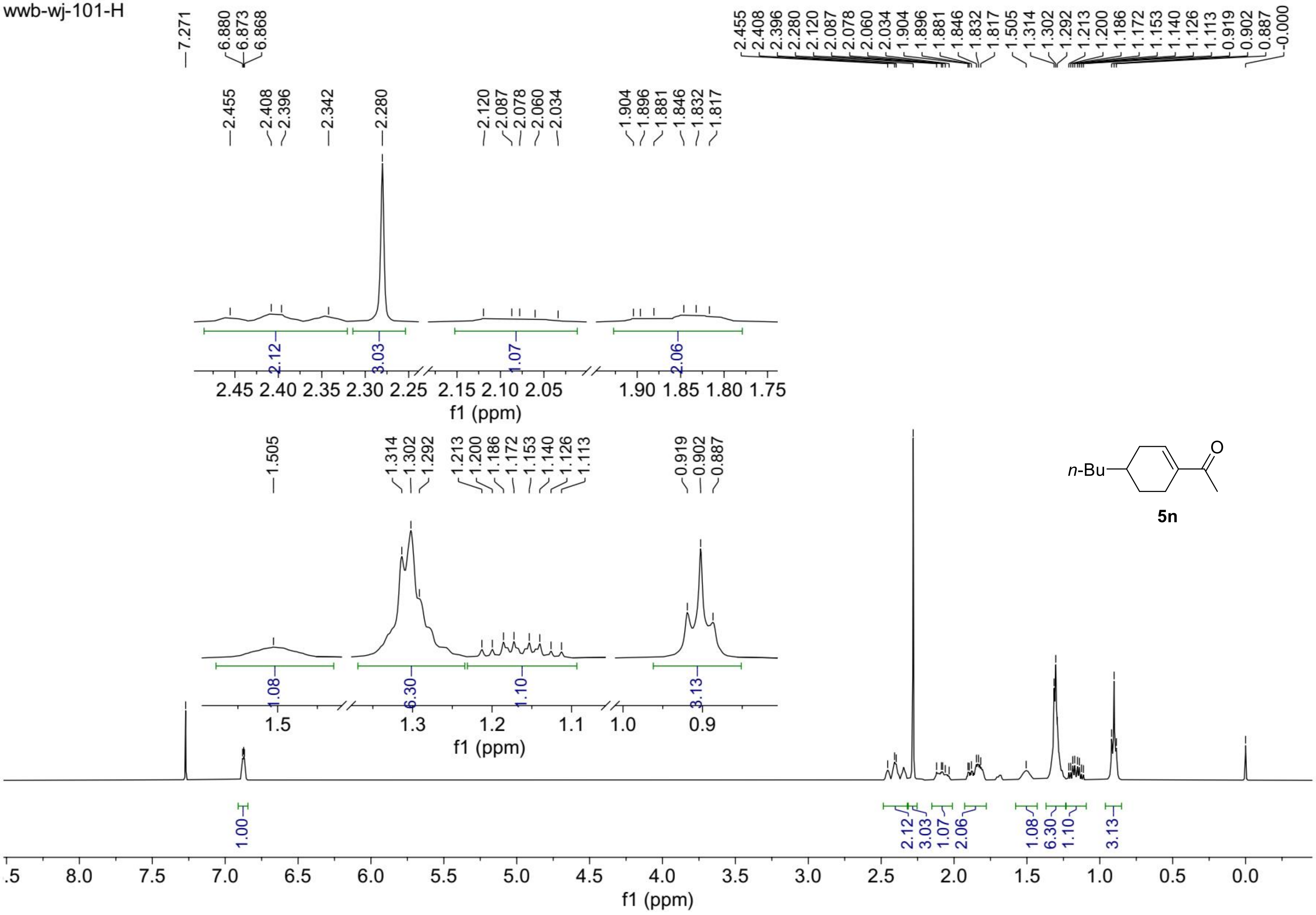

wwb-wj-87-C
199.06
158.95
141.17
139.57
139.13
110.12
103.76
77.48
77.16
76.84
32.63
30.95
27.00
25.42
22.84
O
O
5k
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

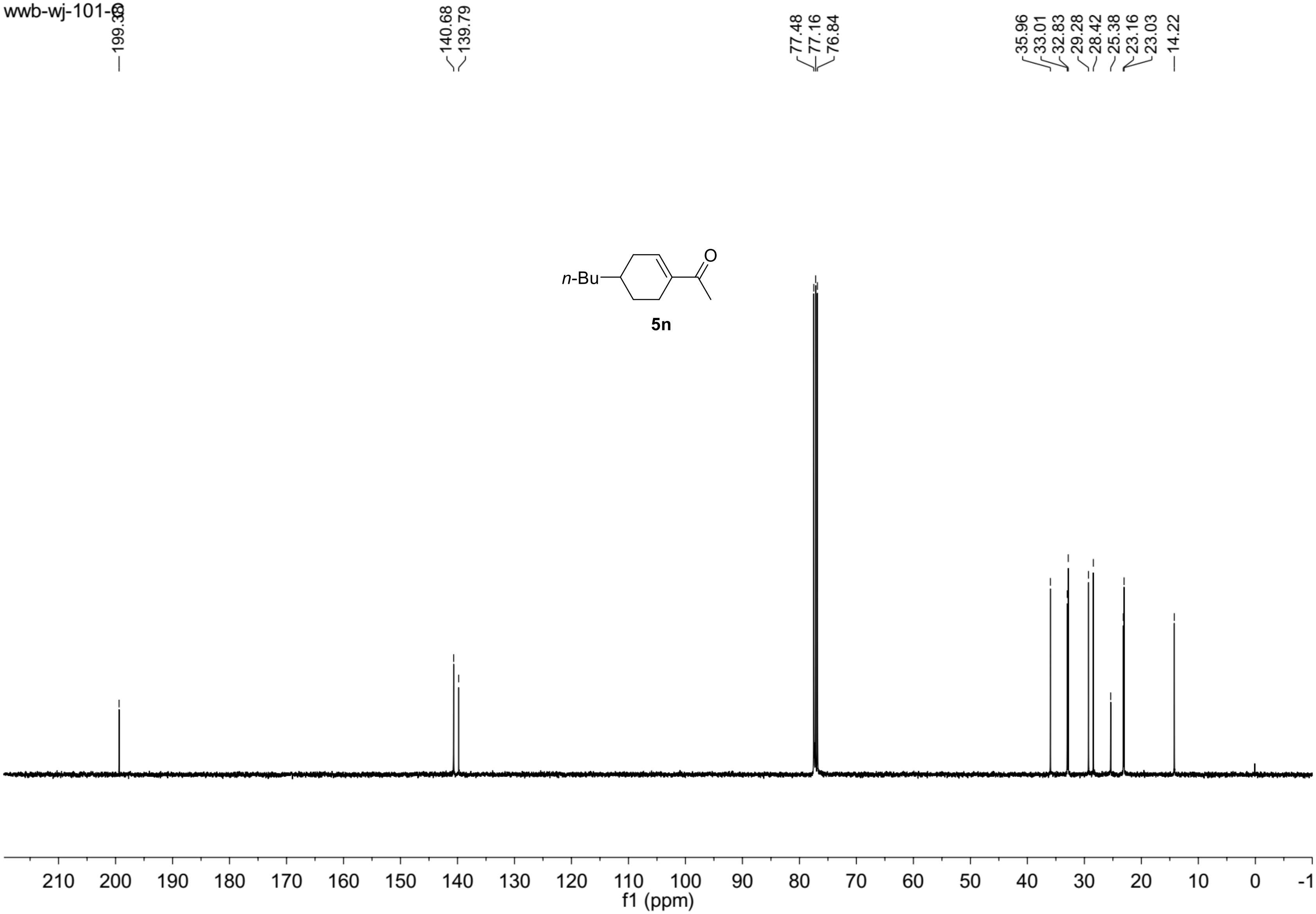

wwb-wj-113
7.270
6.878
2.464
2.417
2.404
2.353
2.281
2.121
2.107
2.086
2.080
2.062
2.045
1.906
1.890
1.858
1.842
1.826
1.819
1.470
1.439
1.427
1.404
1.361
1.343
1.324
1.306
1.289
1.209
1.195
1.181
1.168
1.149
1.136
1.122
1.109
0.948
0.929
0.911
-0.000
2.22
3.11
1.12
2.13
1.13
2.18
1.10
3.18
1.00
f1 (ppm)
Et
O
5l

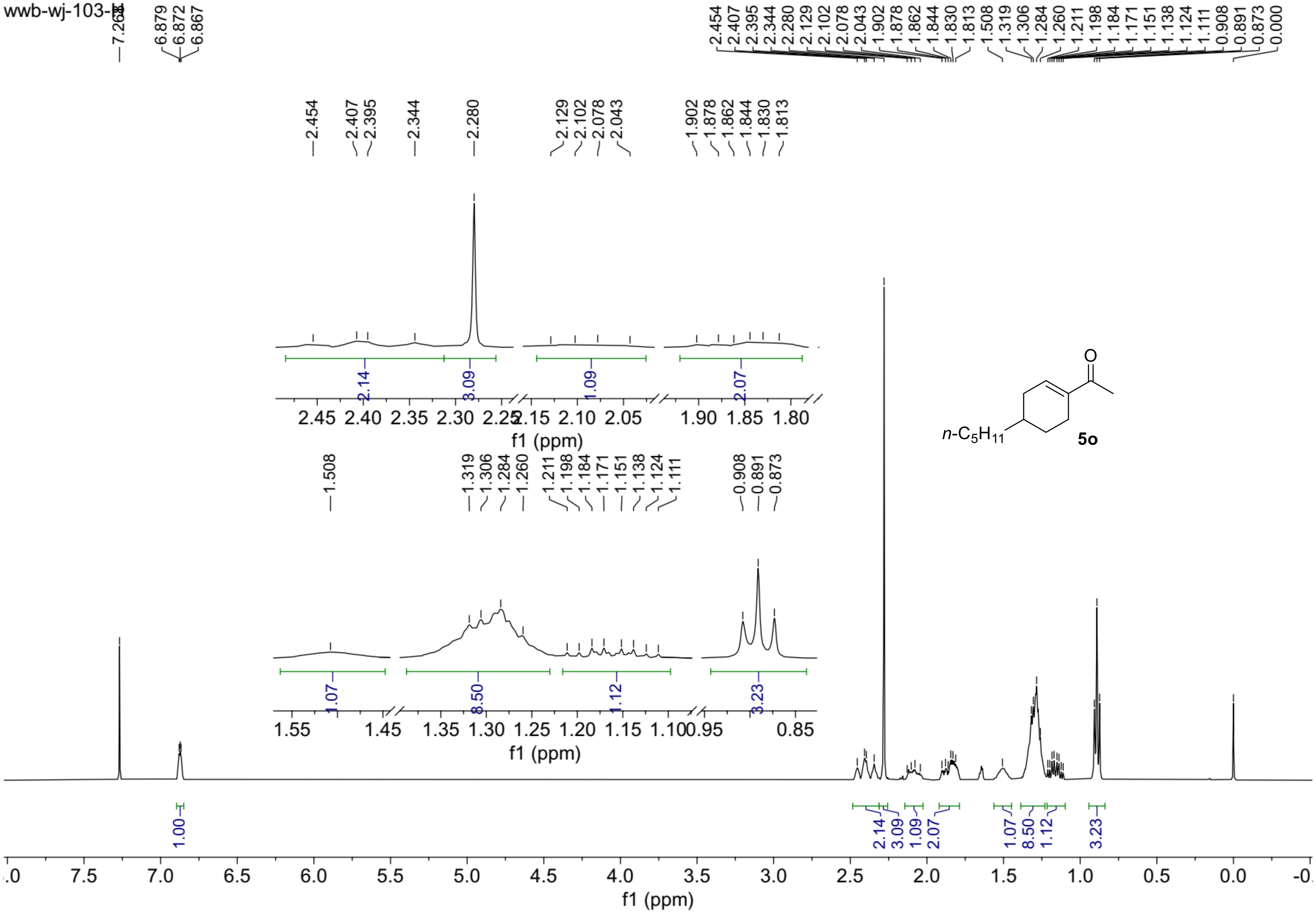

wwb-wj-113-C
199.30
140.66
139.81
77.48
77.16
76.84
34.60
32.63
28.91
28.02
25.38
23.18
11.53
Et
O
5l
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

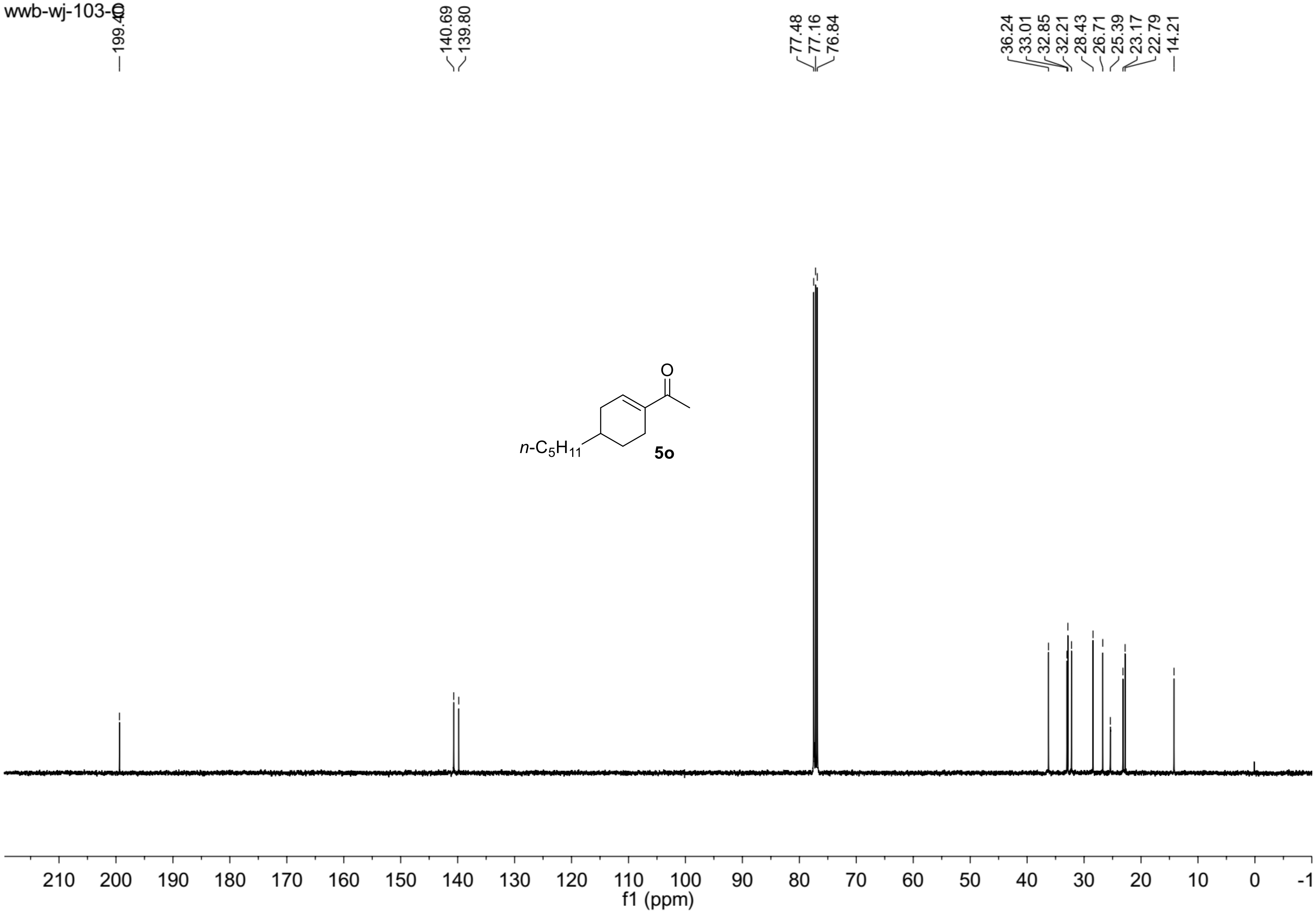

wwb-wj-99-H
7.272
6.874
2.456
2.405
2.397
2.343
2.280
2.120
2.107
2.094
2.086
2.080
2.045
1.902
1.879
1.845
1.831
1.814
1.801
1.542
1.530
1.525
1.519
1.390
1.372
1.355
1.337
1.296
1.280
1.274
1.266
1.252
1.212
1.199
1.185
1.172
1.153
1.139
1.125
1.112
0.925
0.908
0.890
-0.000
2.13
3.01
1.07
2.06
1.05
2.16
2.13
1.10
3.13
1.00
2.45 2.40 2.35 2.30 2.15 2.10 2.05 1.90 1.85 1.80 1.55 1.50 1.40 1.30 1.20 1.10 0.95 0.90
f1 (ppm)
n-Pr
O
5m
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)

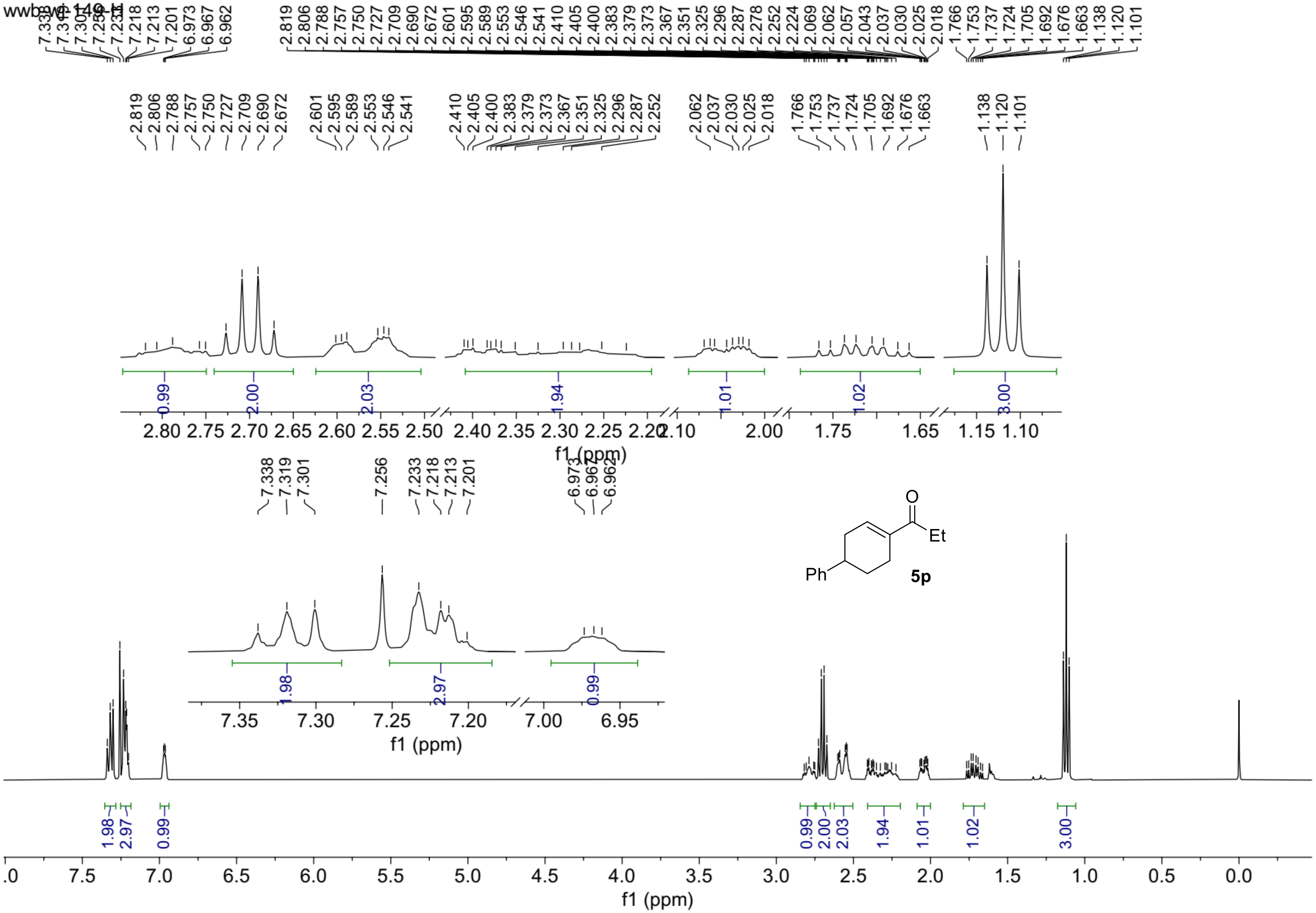

wwb-wj-99-C
199.38
140.67
139.79
77.48
77.16
76.84
38.53
32.95
32.54
28.39
25.38
23.15
20.12
14.40
n-Pr
O
5m
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

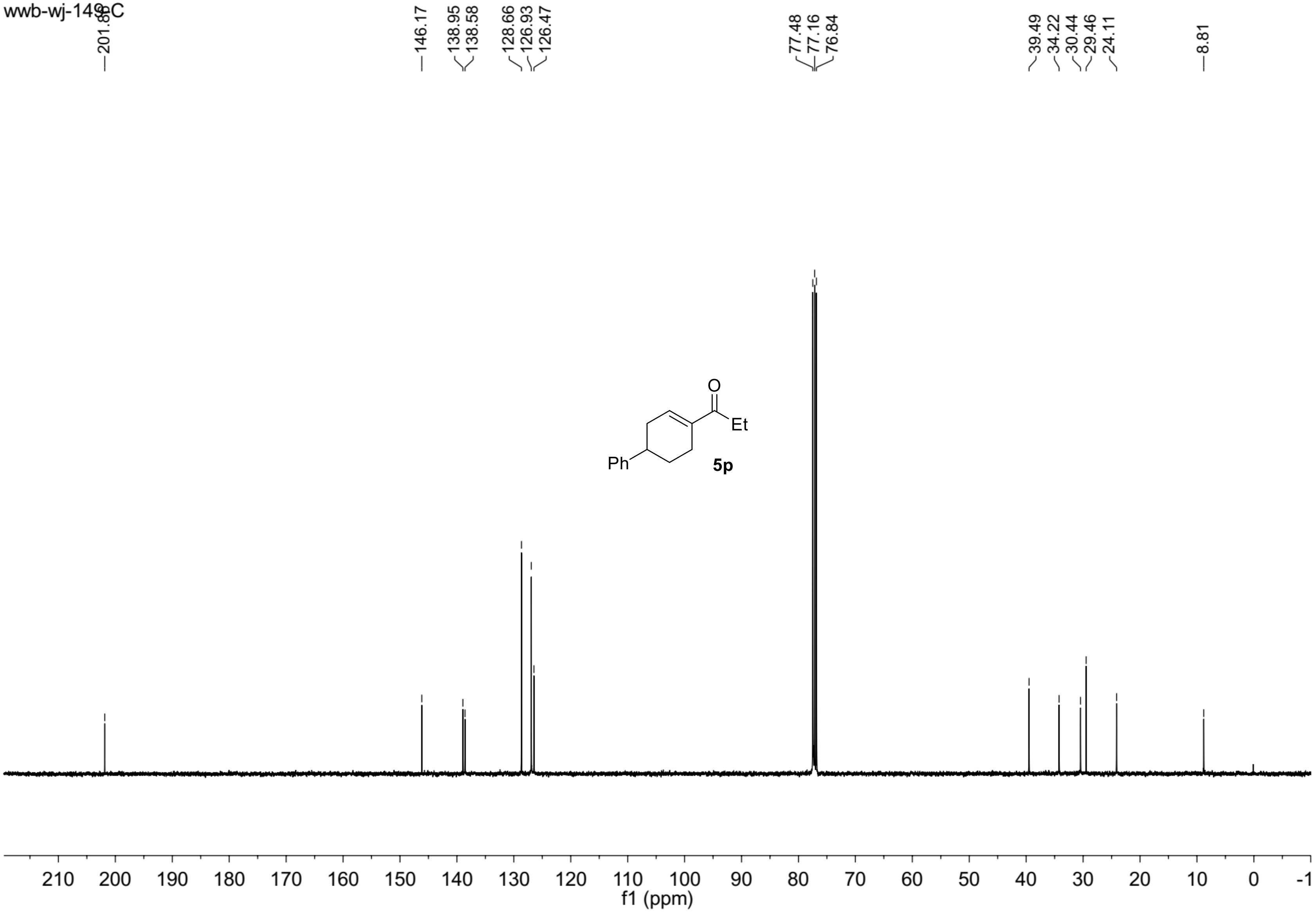

wwb-wj-101-H
7.271
6.880
6.873
6.868
2.455
2.408
2.396
2.280
2.120
2.087
2.078
2.060
2.034
1.904
1.896
1.881
1.846
1.832
1.817
1.505
1.314
1.302
1.292
1.213
1.200
1.186
1.172
1.153
1.140
1.126
1.113
0.919
0.902
0.887
-0.000
2.455
2.408
2.396
2.342
2.280
2.120
2.087
2.078
2.060
2.034
1.904
1.896
1.881
1.846
1.832
1.817
2.12
3.03
1.07
2.06
2.45 2.40 2.35 2.30 2.25 2.15 2.10 2.05 1.90 1.85 1.80 1.75
f1 (ppm)
1.505
1.314
1.302
1.292
1.213
1.200
1.186
1.172
1.153
1.140
1.126
1.113
0.919
0.902
0.887
1.08
6.30
1.10
3.13
1.5 1.3 1.2 1.1 1.0 0.9
f1 (ppm)
n-Bu
O
5n
1.00
2.12
3.03
1.07
2.06
1.08
6.30
1.10
3.13
.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)

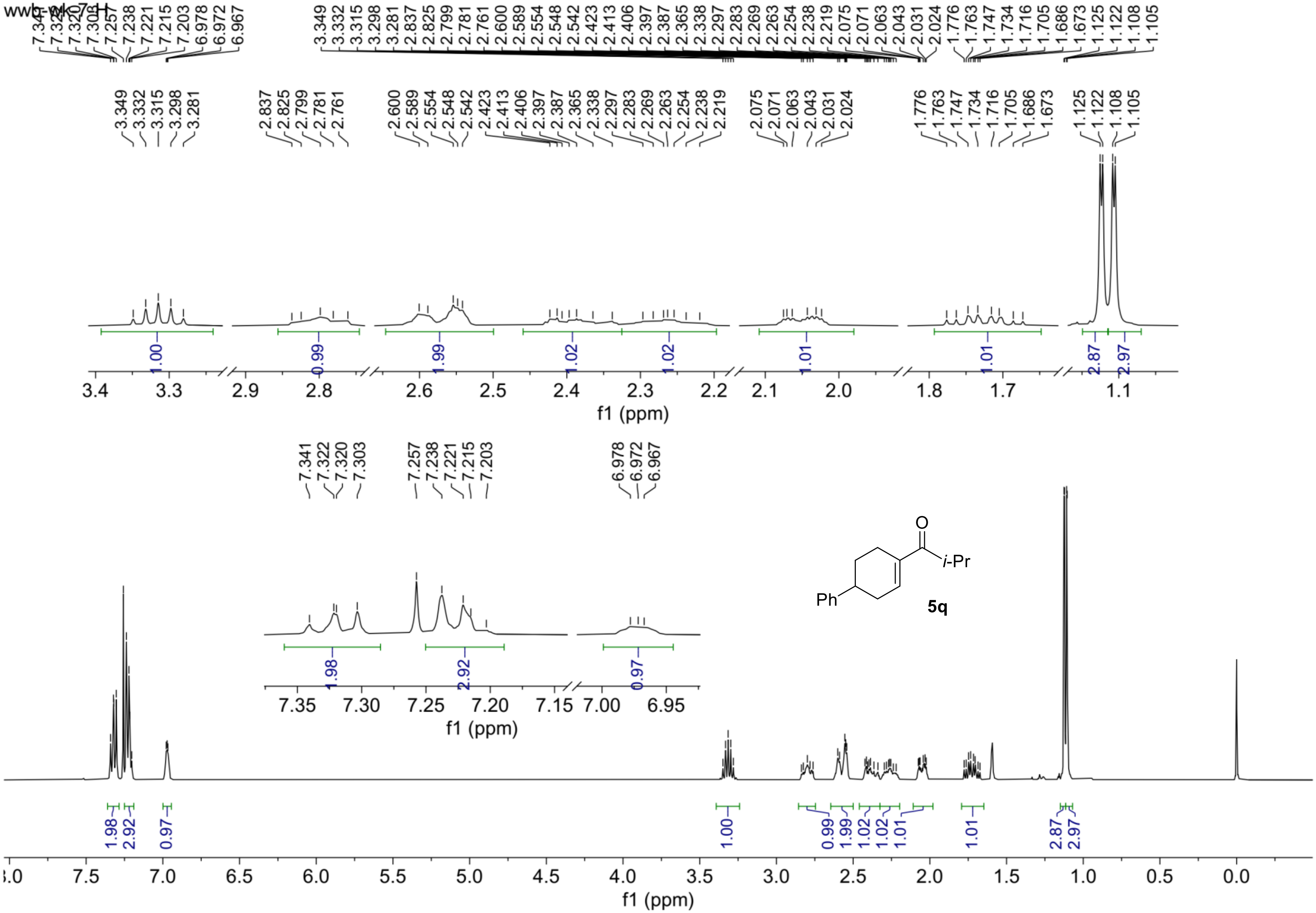

wwb-wj-101-6
199.30
140.68
139.79
77.48
77.16
76.84
35.96
33.01
32.83
29.28
28.42
25.38
23.16
23.03
14.22
n-Bu
O
5n
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)


wwb-wj-103-H
7.260
6.879 6.872 6.867
2.454 2.407 2.395 2.344 2.280 2.129 2.102 2.078 2.043 1.902 1.878 1.862 1.844 1.830 1.813 1.508 1.319 1.306 1.284 1.260 1.211 1.198 1.184 1.171 1.151 1.138 1.124 1.111 0.908 0.891 0.873 0.000
2.14
3.09
1.09
2.07
2.45 2.40 2.35 2.30 2.25 2.15 2.10 2.05 1.90 1.85 1.80
f1 (ppm)
1.07
8.50
1.12
3.23
1.55 1.45 1.35 1.30 1.25 1.20 1.15 1.10 0.95 0.85
f1 (ppm)
O
n-C5H11
5o
1.00
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)

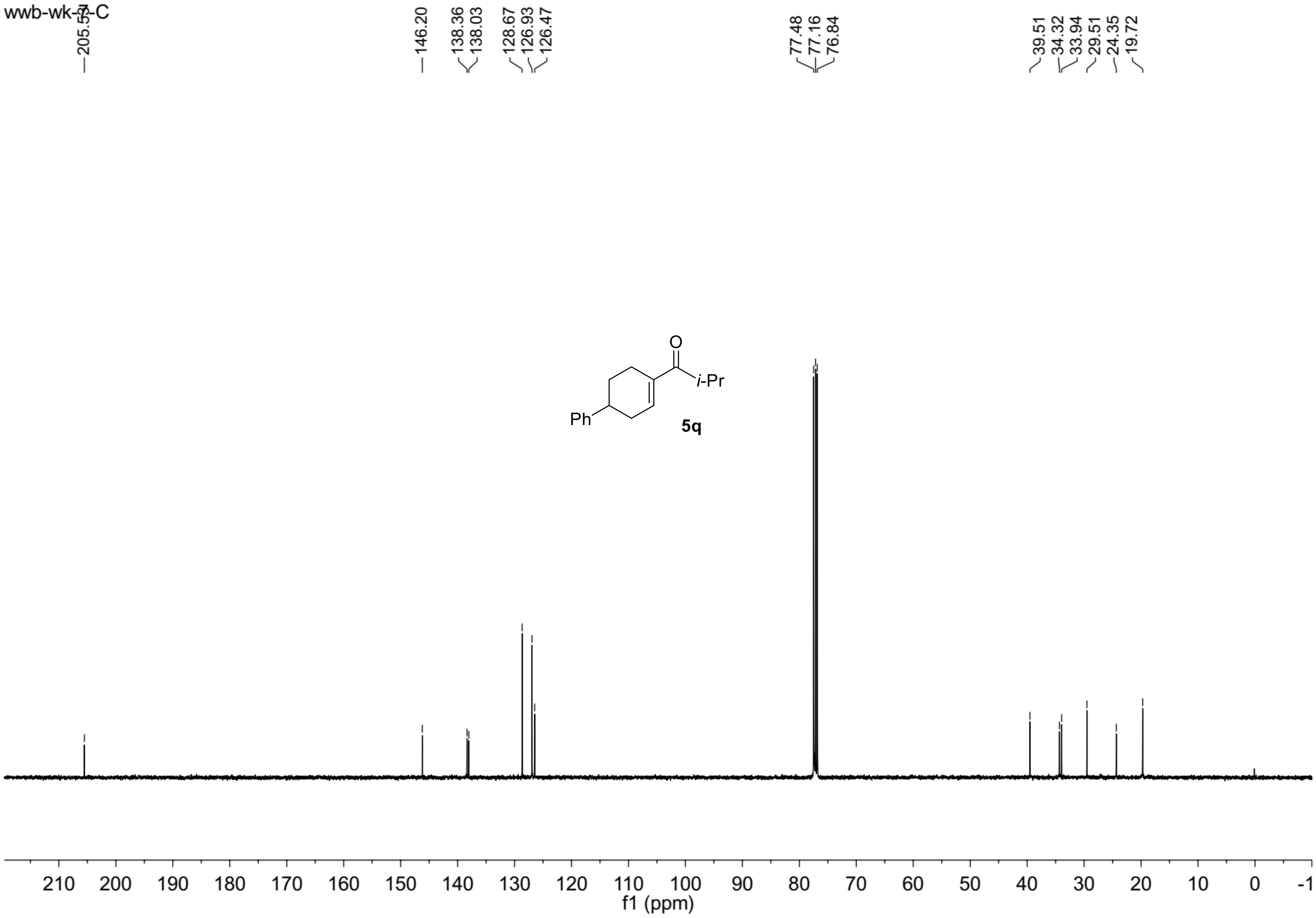

wwb-wj-103-C
199.40
140.69
139.80
77.48
77.16
76.84
36.24
33.01
32.85
32.21
28.43
26.71
25.39
23.17
22.79
14.21
O
n-C5H11
5o
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

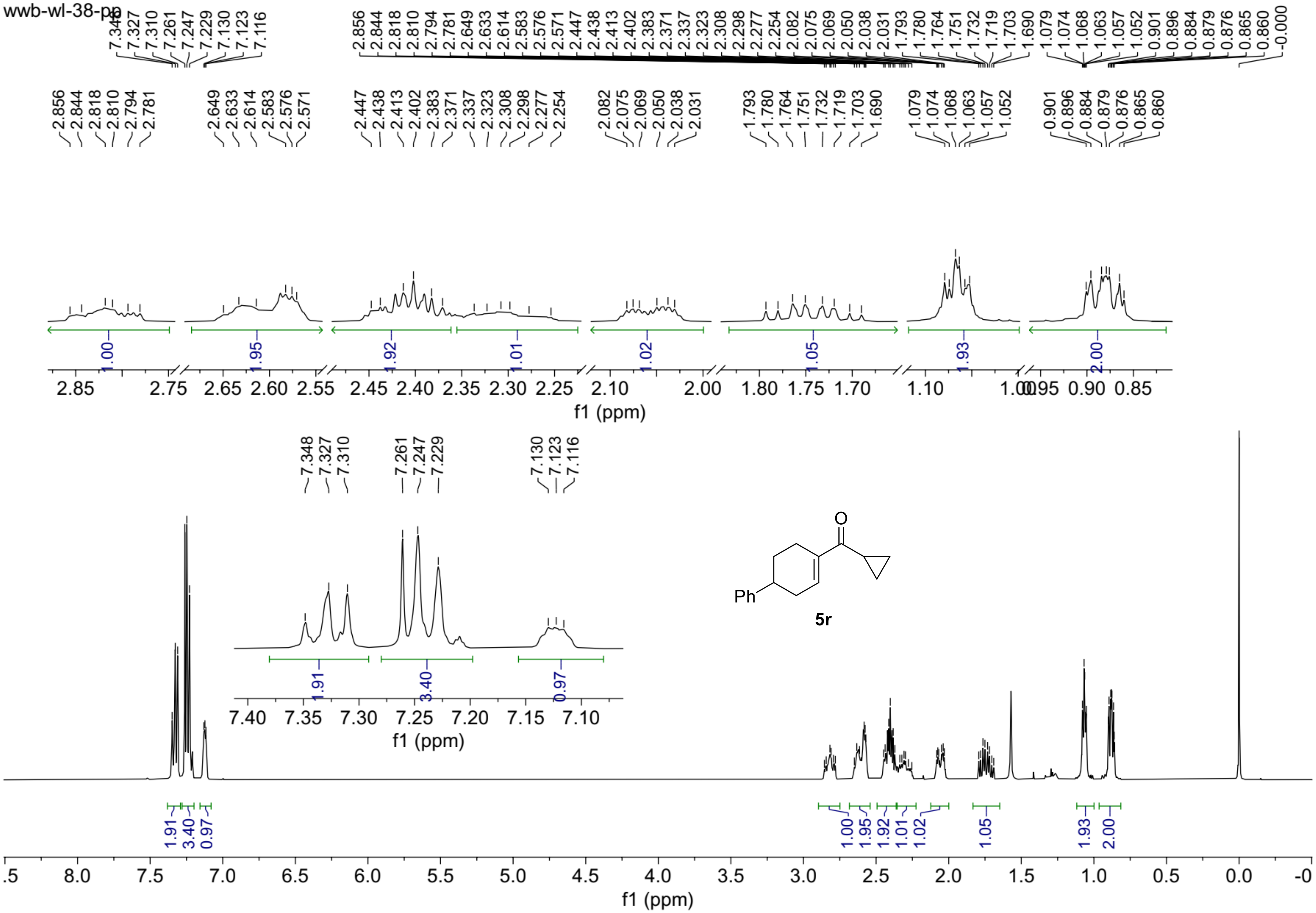

wwbwyl-149-H
7.338 7.319 7.301 7.256 7.233 7.218 7.213 7.201 6.973 6.967 6.962
2.819 2.806 2.788 2.757 2.750 2.727 2.709 2.690 2.672 2.601 2.595 2.589 2.553 2.546 2.541 2.410 2.405 2.400 2.383 2.379 2.373 2.367 2.351 2.325 2.296 2.287 2.278 2.252 2.224 2.069 2.062 2.057 2.043 2.037 2.030 2.025 2.018 1.766 1.753 1.737 1.724 1.705 1.692 1.676 1.663 1.138 1.120 1.101
0.99
2.00
2.03
1.94
1.01
1.02
3.00
1.98
2.97
0.99
O
Et
Ph
5p
f1 (ppm)

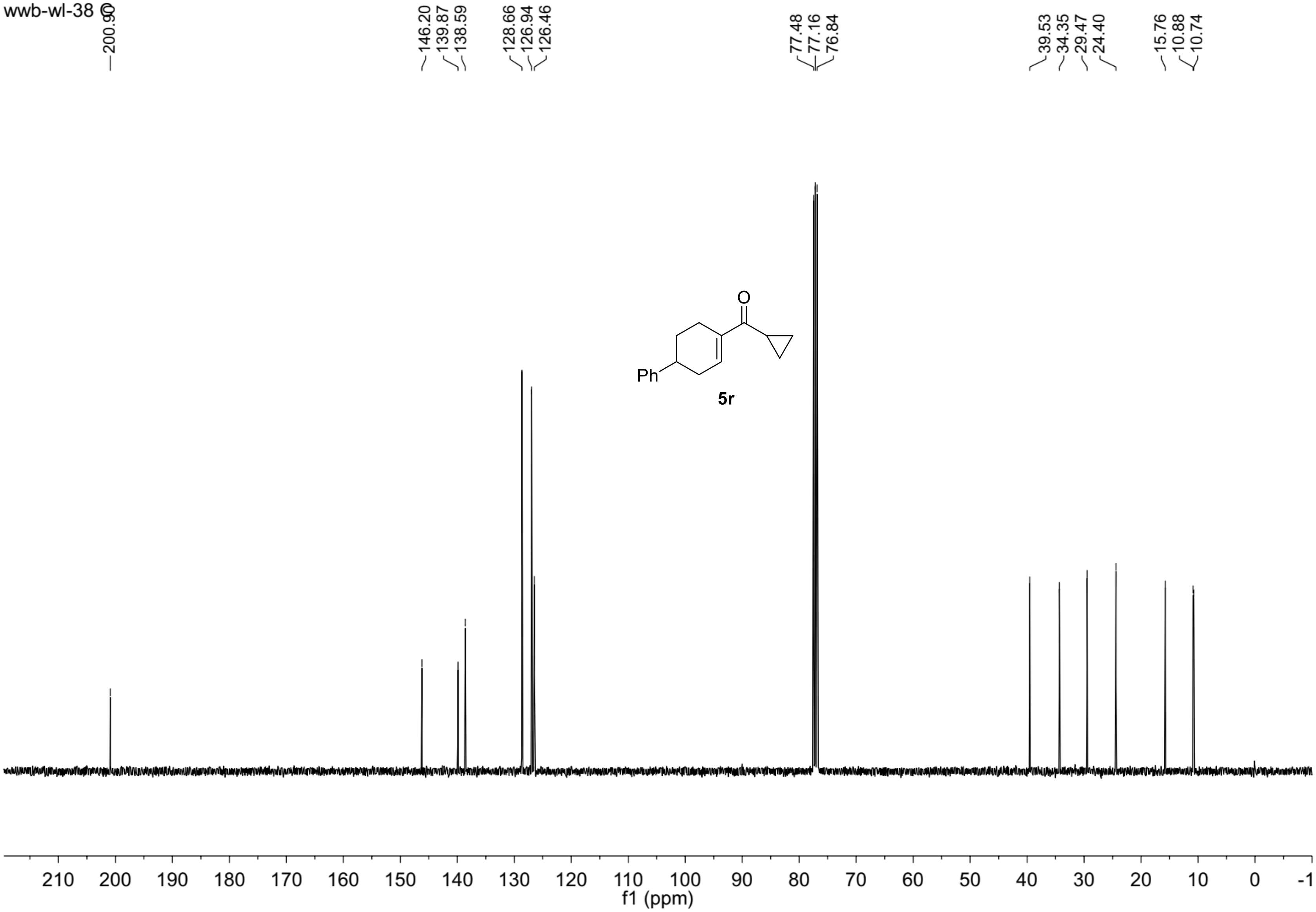
wwb-wj-149-C
201.8
146.17
138.95
138.58
128.66
126.93
126.47
77.48
77.16
76.84
39.49
34.22
30.44
29.46
24.11
8.81
O
Et
Ph
5p
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

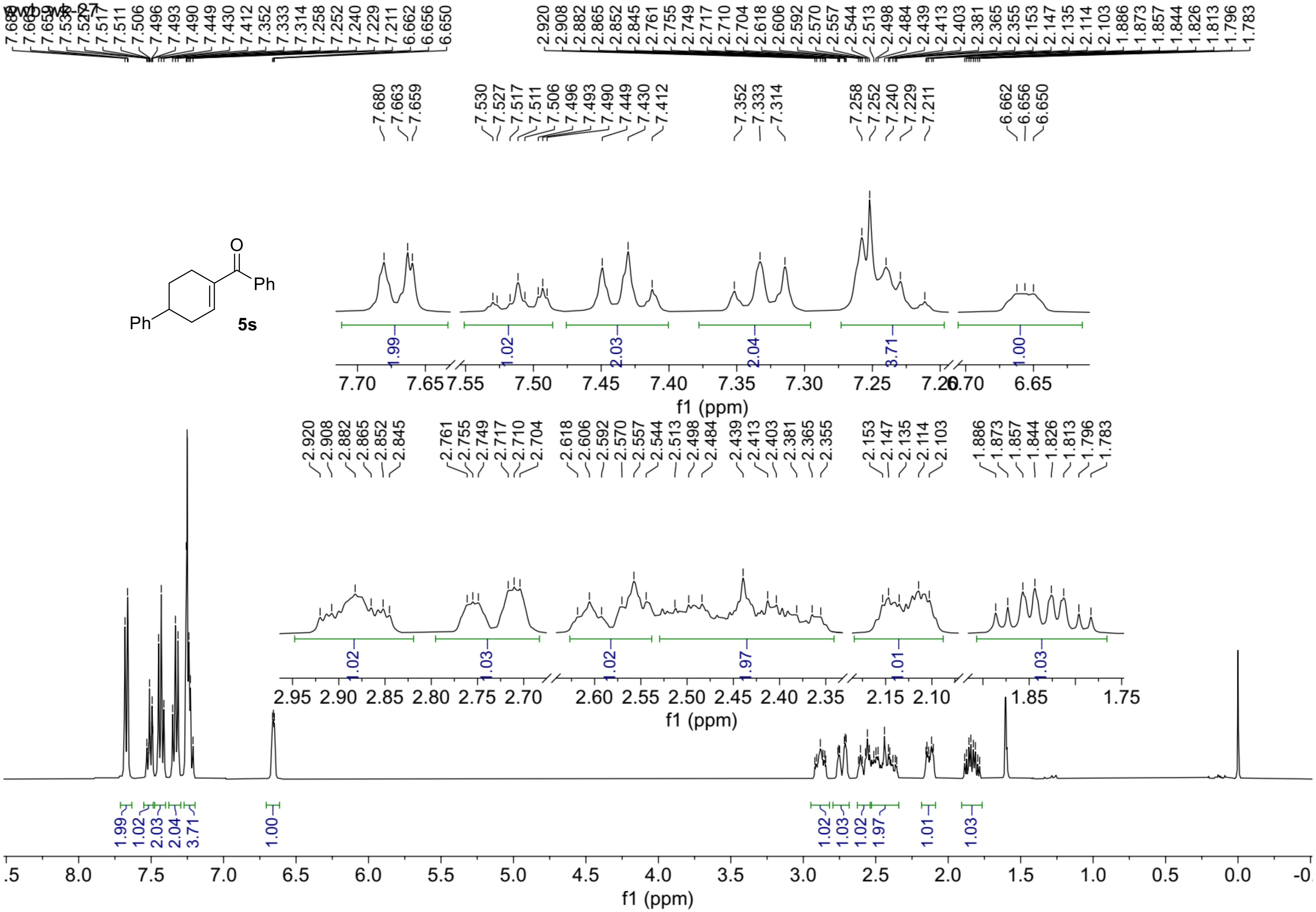

wwb-wk-7-1H
7.341
7.322
7.320
7.303
7.257
7.238
7.221
7.215
7.203
6.978
6.972
6.967
3.349
3.332
3.315
3.298
3.281
2.837
2.825
2.799
2.781
2.761
2.600
2.589
2.554
2.548
2.542
2.423
2.413
2.406
2.397
2.387
2.365
2.338
2.297
2.283
2.269
2.263
2.254
2.238
2.219
2.075
2.071
2.063
2.043
2.031
2.024
1.776
1.763
1.747
1.734
1.716
1.705
1.686
1.673
1.125
1.122
1.108
1.105
1.00
0.99
1.99
1.02
1.02
1.01
1.01
2.87
2.97
1.98
2.92
0.97
O
i-Pr
Ph
f1 (ppm)

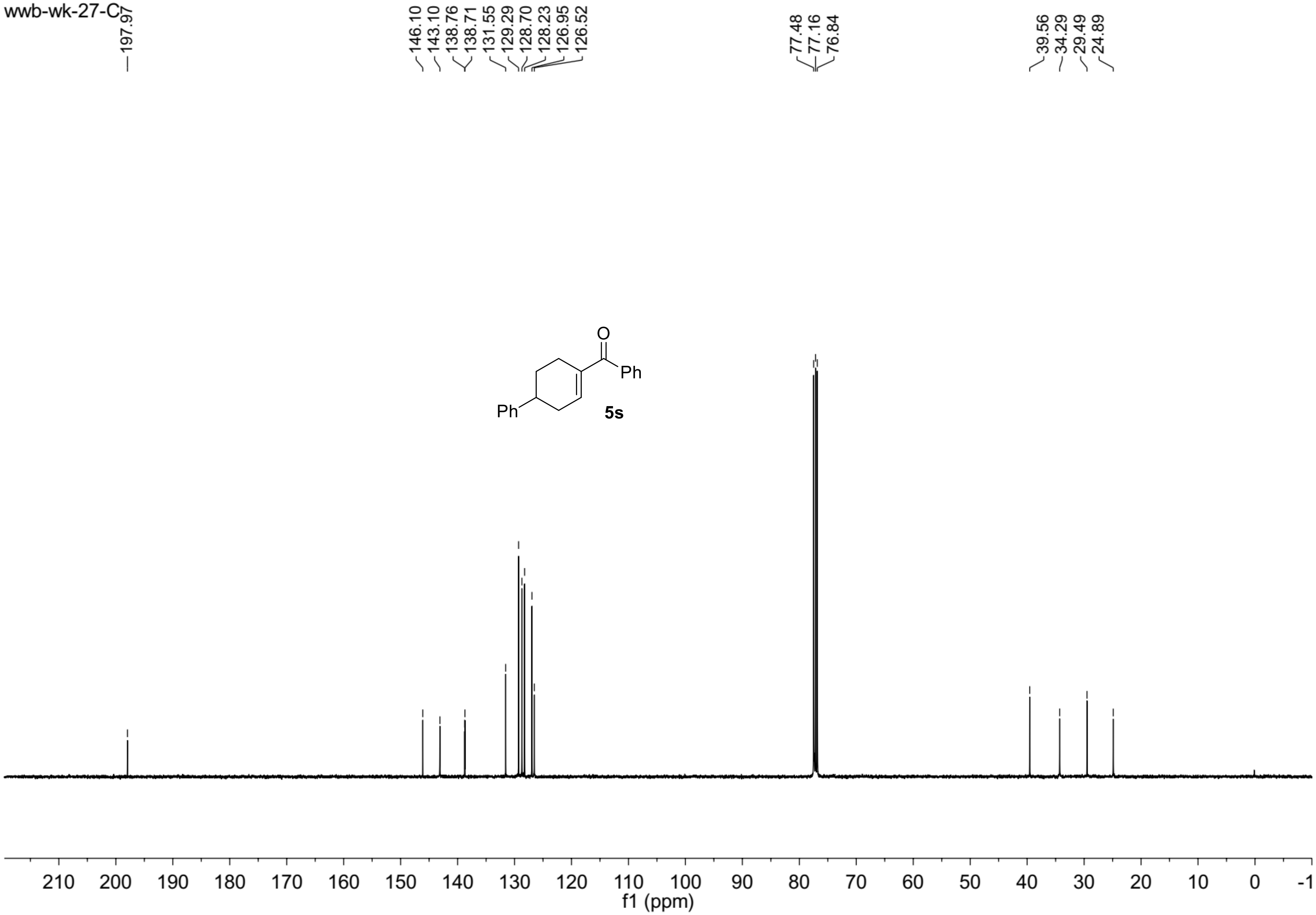
5q

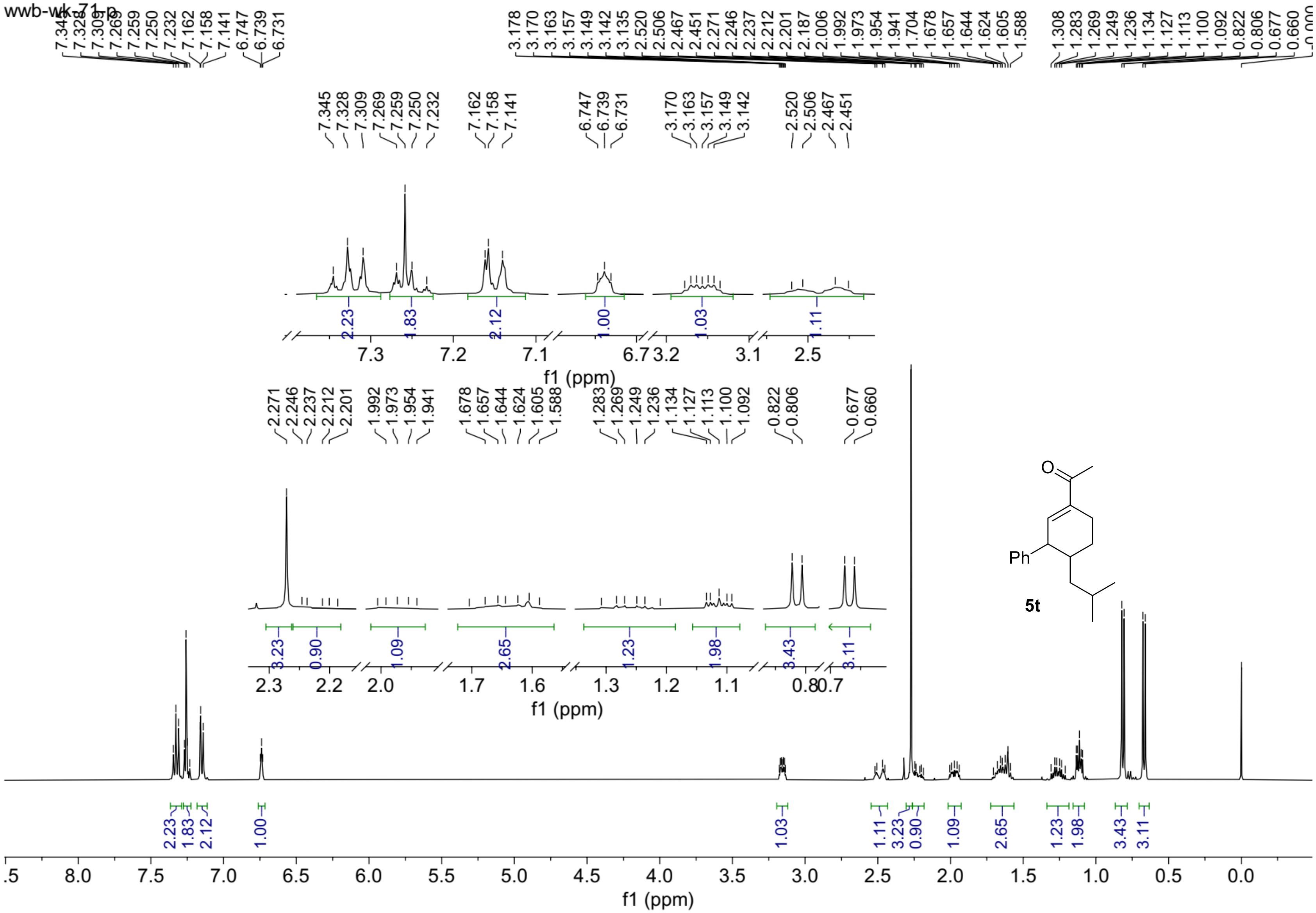

wwb-wk-7-C
205.57
146.20
138.36
138.03
128.67
126.93
126.47
77.48
77.16
76.84
39.51
34.32
33.94
29.51
24.35
19.72
O
i-Pr
Ph
5q
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

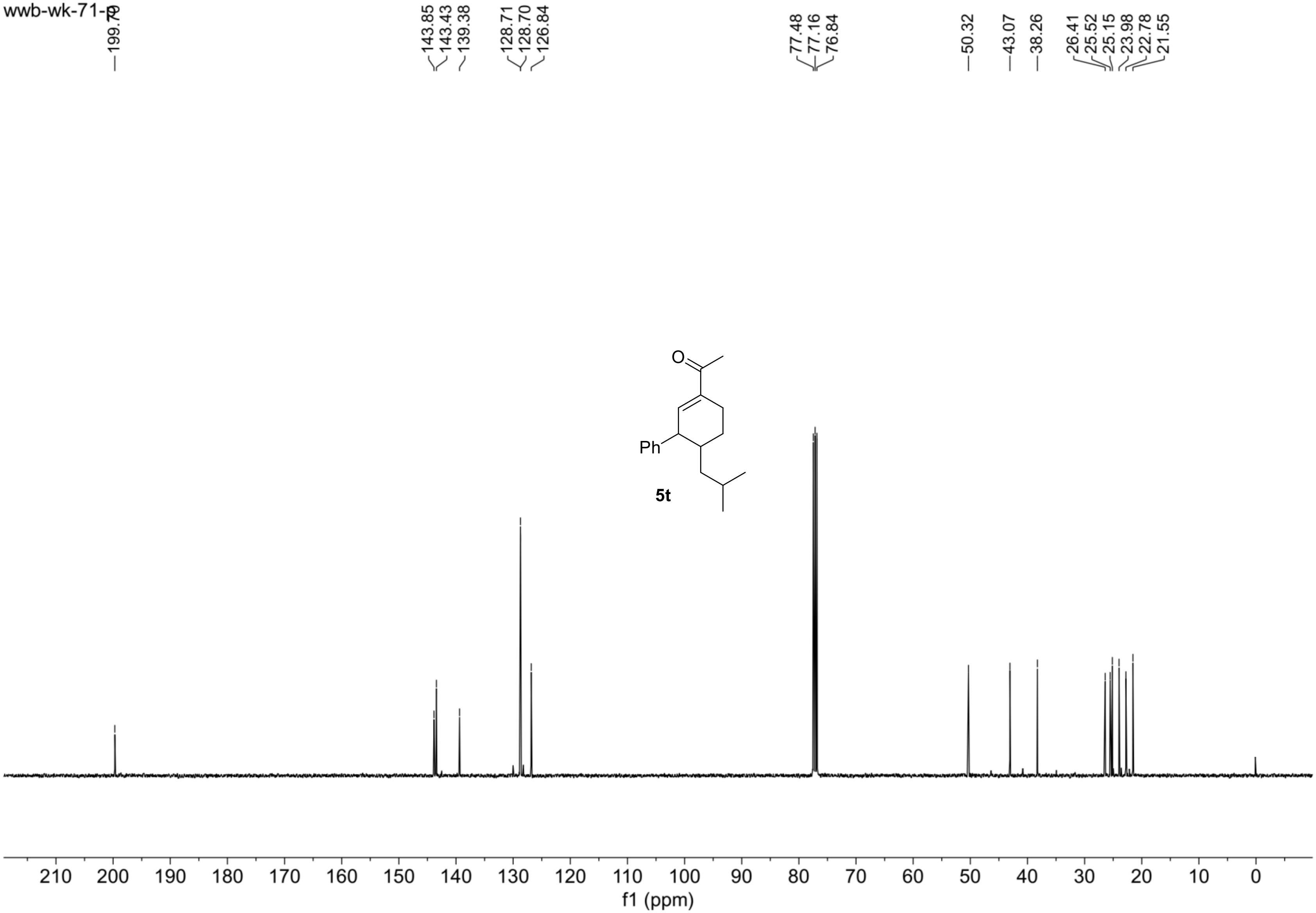

wwb-wl-38-pp
7.348
7.327
7.310
7.261
7.247
7.229
7.130
7.123
7.116
2.856
2.844
2.818
2.810
2.794
2.781
2.649
2.633
2.614
2.583
2.576
2.571
2.447
2.438
2.413
2.402
2.383
2.371
2.337
2.323
2.308
2.298
2.277
2.254
2.082
2.075
2.069
2.050
2.038
2.031
1.793
1.780
1.764
1.751
1.732
1.719
1.703
1.690
1.079
1.074
1.068
1.063
1.057
1.052
0.901
0.896
0.884
0.879
0.876
0.865
0.860
-0.000
1.00
1.95
1.92
1.01
1.02
1.05
1.93
2.00
2.85
2.75
2.65
2.60
2.55
2.45
2.40
2.35
2.30
2.25
2.10
2.00
1.80
1.75
1.70
1.10
1.00
0.95
0.90
0.85
f1 (ppm)
1.91
3.40
0.97
7.40
7.35
7.30
7.25
7.20
7.15
7.10
f1 (ppm)
O
Ph
5r
.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
f1 (ppm)

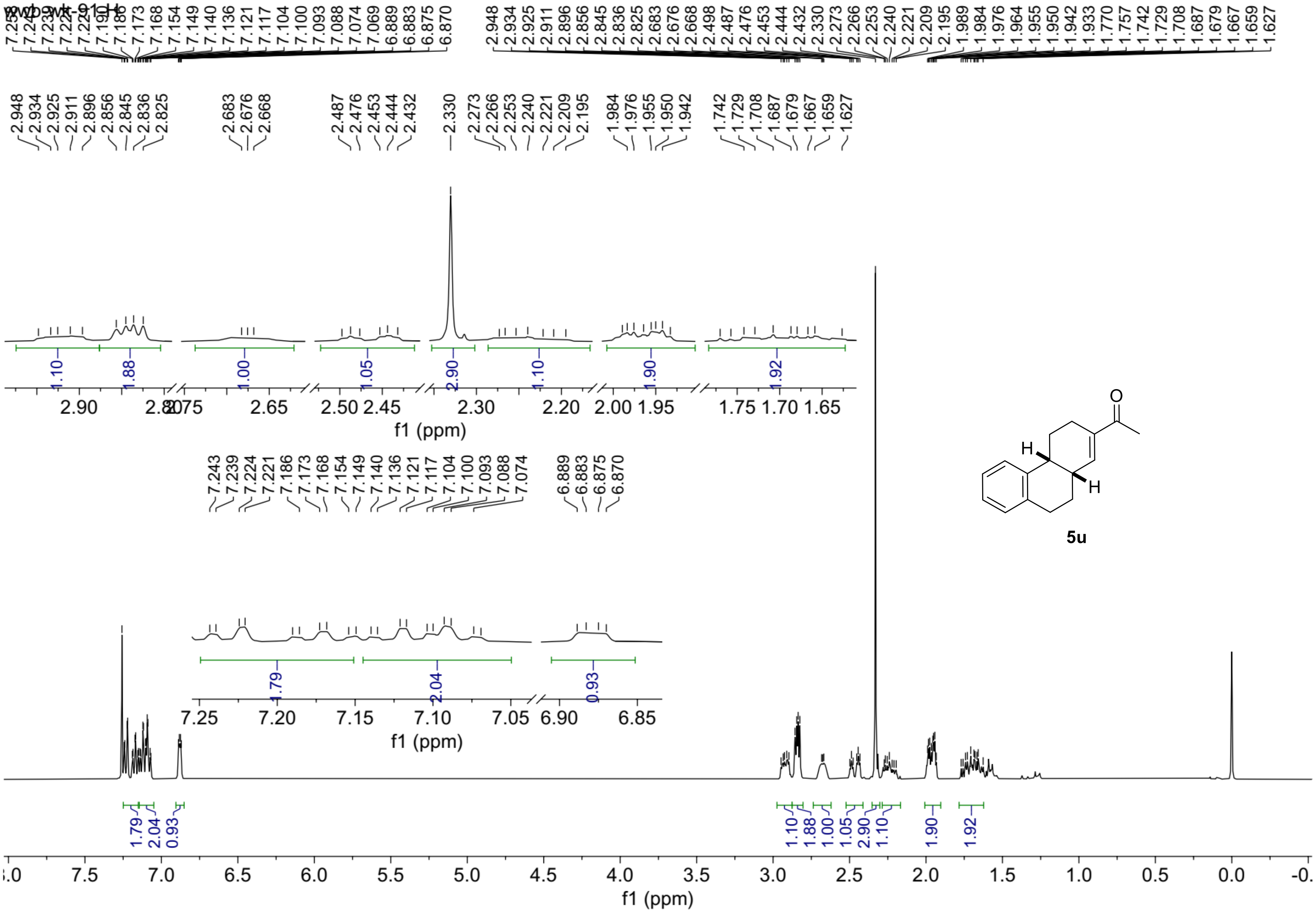

wwb-wl-38 C
200.90
146.20
139.87
138.59
128.66
126.94
126.46
77.48
77.16
76.84
39.53
34.35
29.47
24.40
15.76
10.88
10.74
O
Ph
5r
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)


wwb-wk-27
7.680 7.663 7.659 7.530 7.527 7.517 7.511 7.506 7.496 7.493 7.490 7.449 7.430 7.412 7.352 7.333 7.314 7.258 7.252 7.240 7.229 7.211 6.662 6.656 6.650
2.920 2.908 2.882 2.865 2.852 2.845 2.761 2.755 2.749 2.717 2.710 2.704 2.618 2.606 2.592 2.570 2.557 2.544 2.513 2.498 2.484 2.439 2.413 2.403 2.381 2.365 2.355 2.153 2.147 2.135 2.114 2.103 1.886 1.873 1.857 1.844 1.826 1.813 1.796 1.783
O
Ph
Ph
5s
1.99
1.02
2.03
2.04
3.71
1.00
7.70 7.65 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 6.70 6.65
f1 (ppm)
1.02
1.03
1.02
1.97
1.01
1.03
2.95 2.90 2.85 2.80 2.75 2.70 2.60 2.55 2.50 2.45 2.40 2.35 2.15 2.10 1.85 1.75
f1 (ppm)
.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.
f1 (ppm)

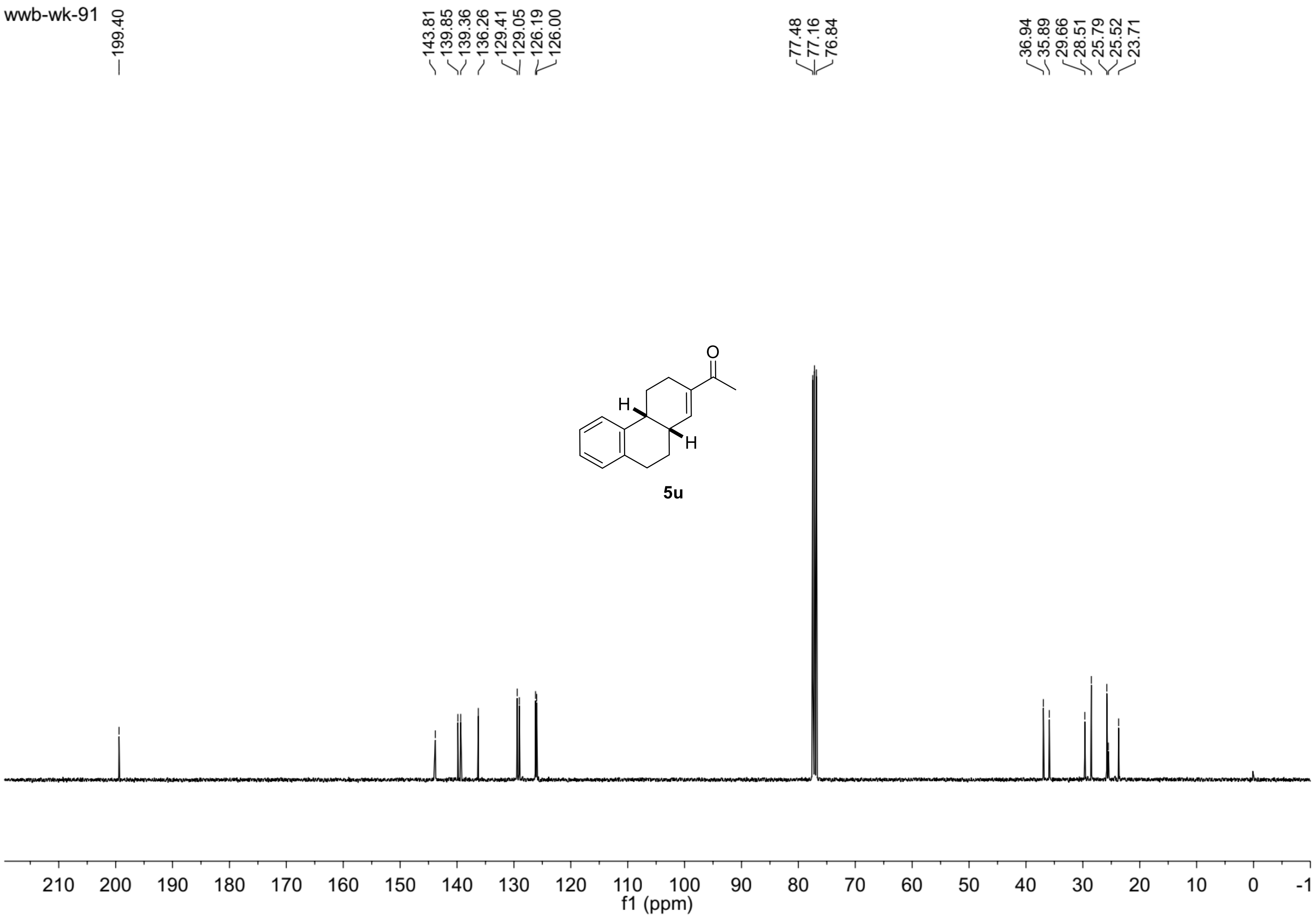

wwb-wk-27-C
197.97
146.10
143.10
138.76
138.71
131.55
129.29
128.70
128.23
126.95
126.52
77.48
77.16
76.84
39.56
34.29
29.49
24.89
O
Ph
Ph
5s
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

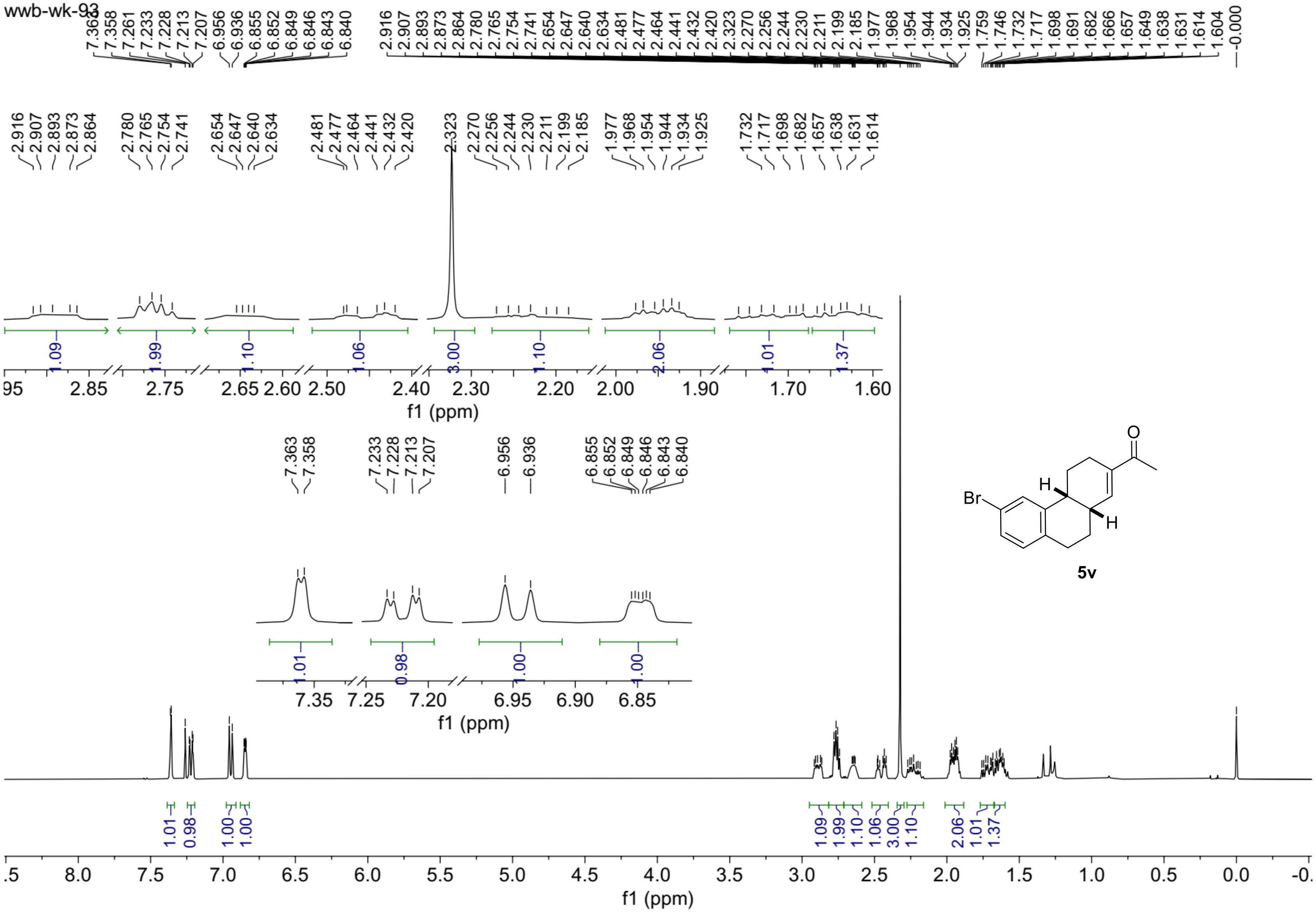

wwb-wk-71-p
7.345
7.328
7.309
7.269
7.259
7.250
7.232
7.162
7.158
7.141
6.747
6.739
6.731
3.178
3.170
3.163
3.157
3.149
3.142
3.135
2.520
2.506
2.467
2.451
2.271
2.246
2.237
2.212
2.201
2.187
2.006
1.992
1.973
1.954
1.941
1.704
1.678
1.657
1.644
1.624
1.605
1.588
1.308
1.283
1.269
1.249
1.236
1.134
1.127
1.113
1.100
1.092
0.822
0.806
0.677
0.660
0.000
2.23
1.83
2.12
1.00
1.03
1.11
3.23
0.90
1.09
2.65
1.23
1.98
3.43
3.11
7.3
7.2
7.1
6.7
3.2
3.1
2.5
2.3
2.2
2.0
1.7
1.6
1.3
1.2
1.1
0.8
0.7
f1 (ppm)
O
Ph
5t
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

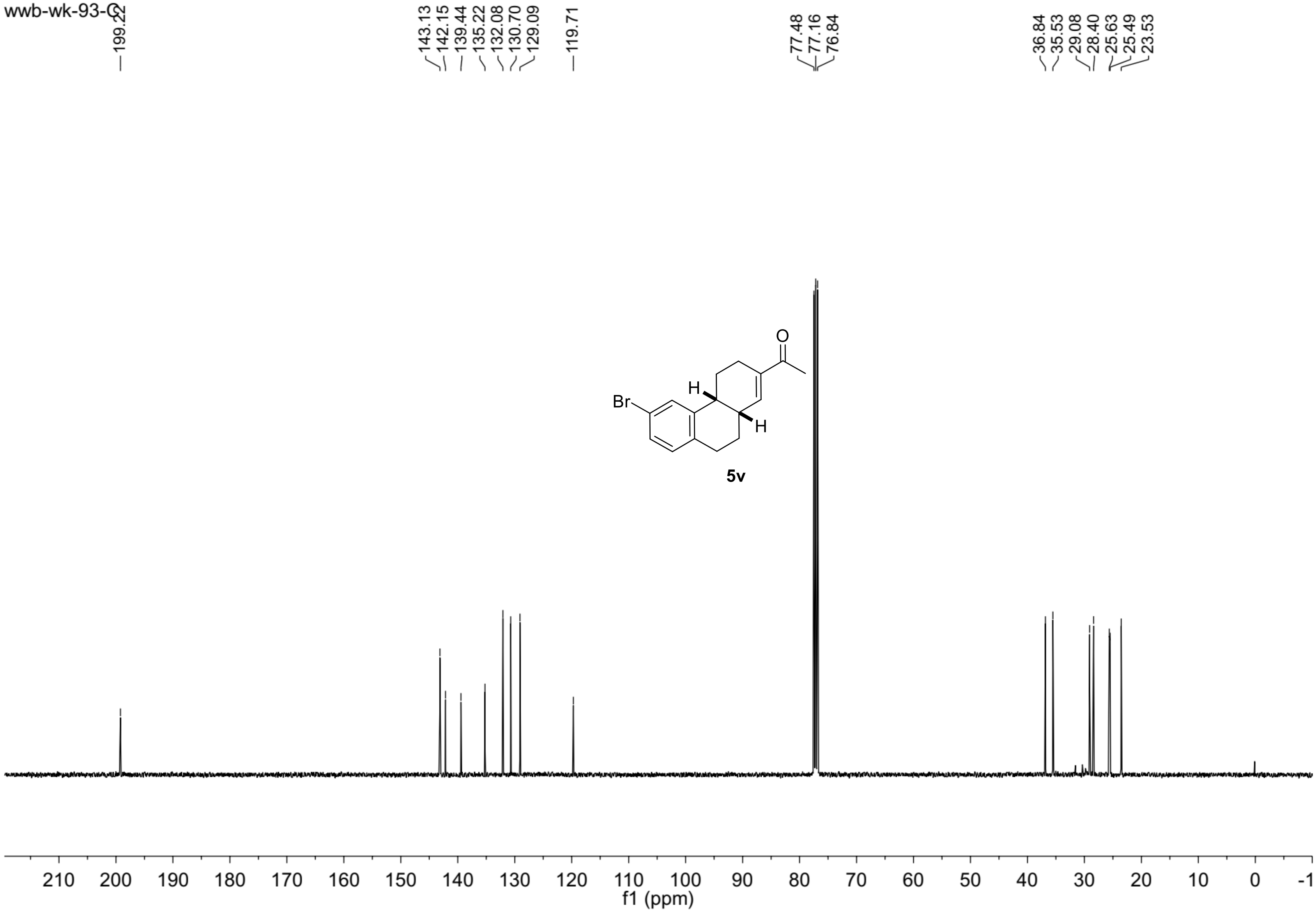

wwb-wk-71-p
199.70
143.85
143.43
139.38
128.71
128.70
126.84
77.48
77.16
76.84
50.32
43.07
38.26
26.41
25.52
25.15
23.98
22.78
21.55
O
Ph
5t
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

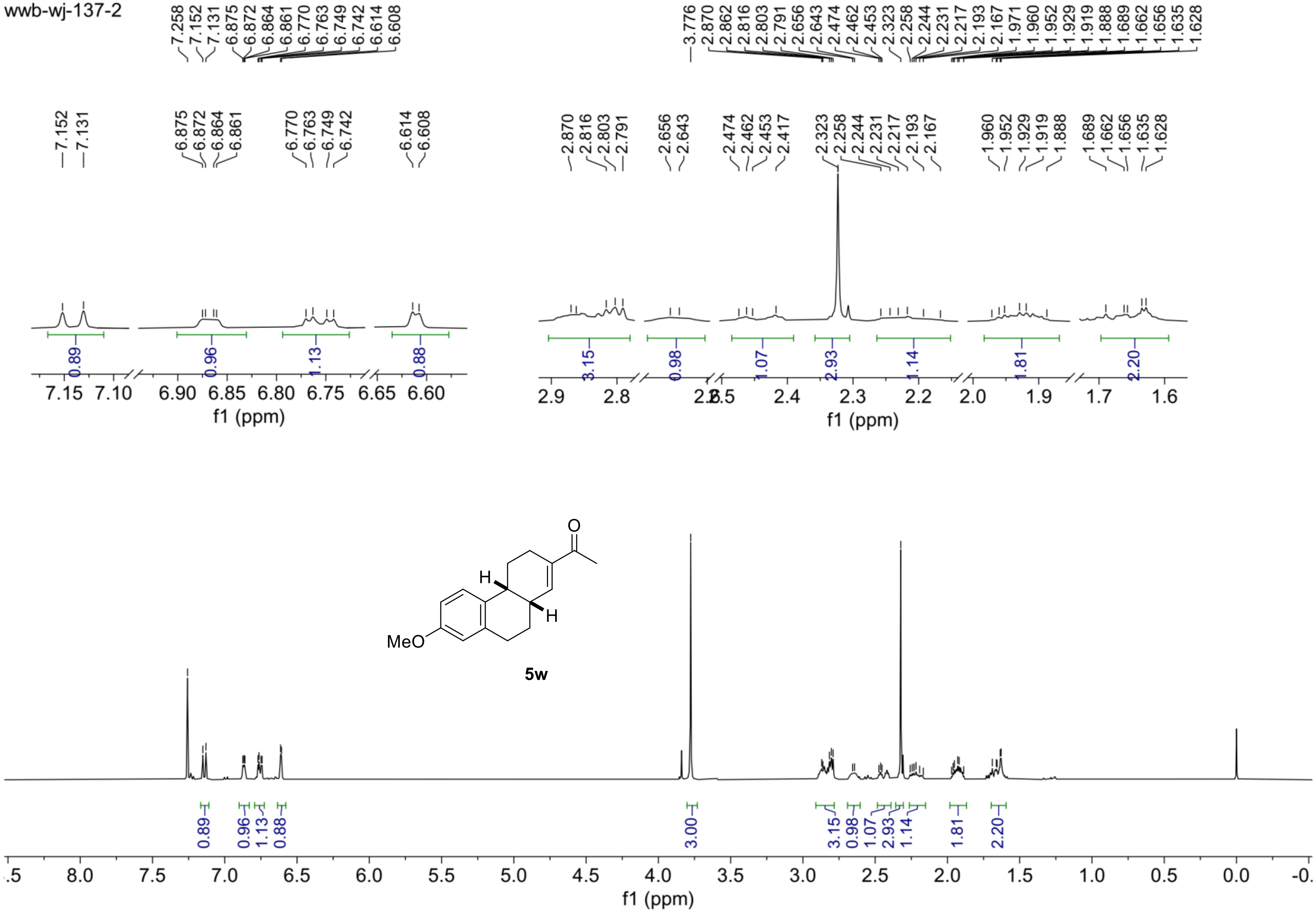

wwb-wk-91-H
7.250 7.243 7.239 7.224 7.221 7.191 7.186 7.173 7.168 7.154 7.149 7.140 7.136 7.121 7.117 7.104 7.100 7.093 7.088 7.074 7.069 6.889 6.883 6.875 6.870
2.948 2.934 2.925 2.911 2.896 2.856 2.845 2.836 2.825 2.683 2.676 2.668 2.498 2.487 2.476 2.453 2.444 2.432 2.330 2.273 2.266 2.253 2.240 2.221 2.209 2.195 1.989 1.984 1.976 1.964 1.955 1.950 1.942 1.933 1.770 1.757 1.742 1.729 1.708 1.687 1.679 1.667 1.659 1.627
1.10
1.88
1.00
1.05
2.90
1.10
1.90
1.92
2.90 2.85 2.80 2.75 2.65 2.50 2.45 2.30 2.20 2.00 1.95 1.75 1.70 1.65
f1 (ppm)
1.79
2.04
0.93
7.25 7.20 7.15 7.10 7.05 6.90 6.85
f1 (ppm)
O
H
H
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)

5u

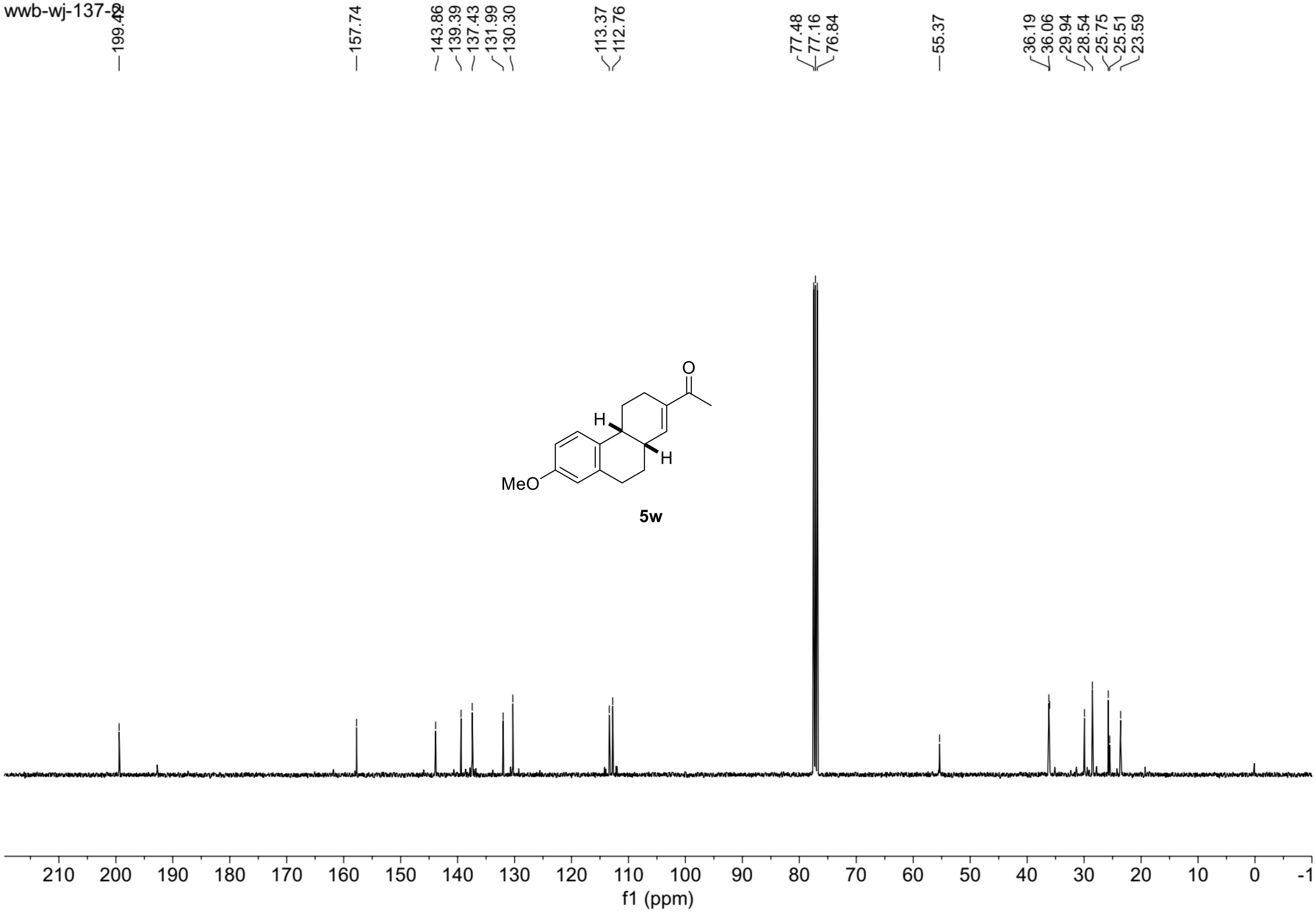

wwb-wk-91
199.40
143.81
139.85
139.36
136.26
129.41
129.05
126.19
126.00
77.48
77.16
76.84
36.94
35.89
29.66
28.51
25.79
25.52
23.71
O
H
H
5u
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

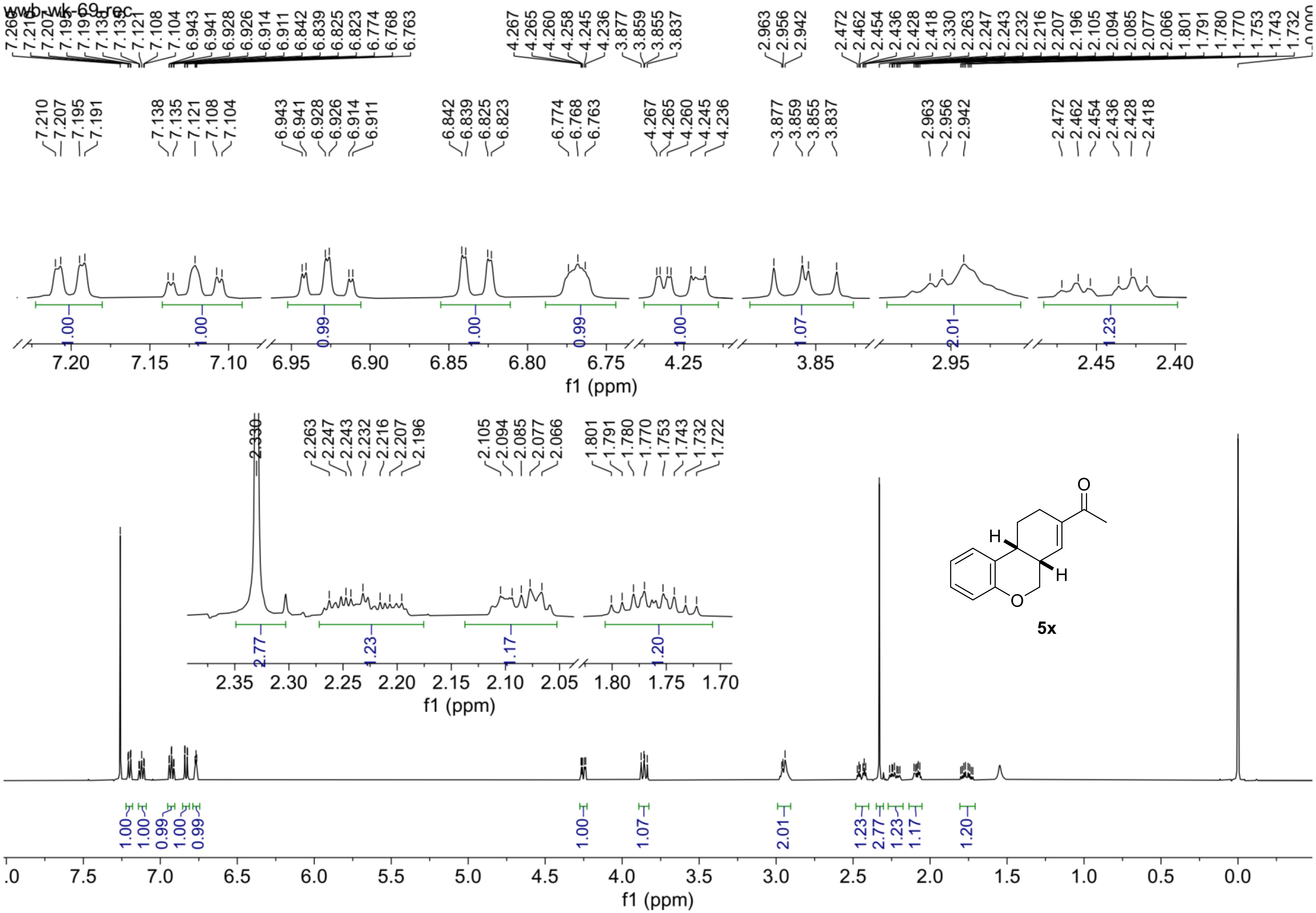

wwb-wk-93
7.363 7.358 7.261 7.233 7.228 7.213 7.207 6.956 6.936 6.855 6.852 6.849 6.846 6.843 6.840
2.916 2.907 2.893 2.873 2.864 2.780 2.765 2.754 2.741 2.654 2.647 2.640 2.634 2.481 2.477 2.464 2.441 2.432 2.420 2.323 2.270 2.256 2.244 2.230 2.211 2.199 2.185 1.977 1.968 1.954 1.944 1.934 1.925 1.759 1.746 1.732 1.717 1.698 1.691 1.682 1.666 1.657 1.649 1.638 1.631 1.614 1.604 -0.000
1.09
1.99
1.10
1.06
3.00
1.10
2.06
1.01
1.37
f1 (ppm)
1.01
0.98
1.00
1.00
f1 (ppm)
Br
H
H
O
5v
f1 (ppm)


wwb-wk-93-C
199.22
143.13
142.15
139.44
135.22
132.08
130.70
129.09
119.71
77.48
77.16
76.84
36.84
35.53
29.08
28.40
25.63
25.49
23.53
O
H
Br
H
5v
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)

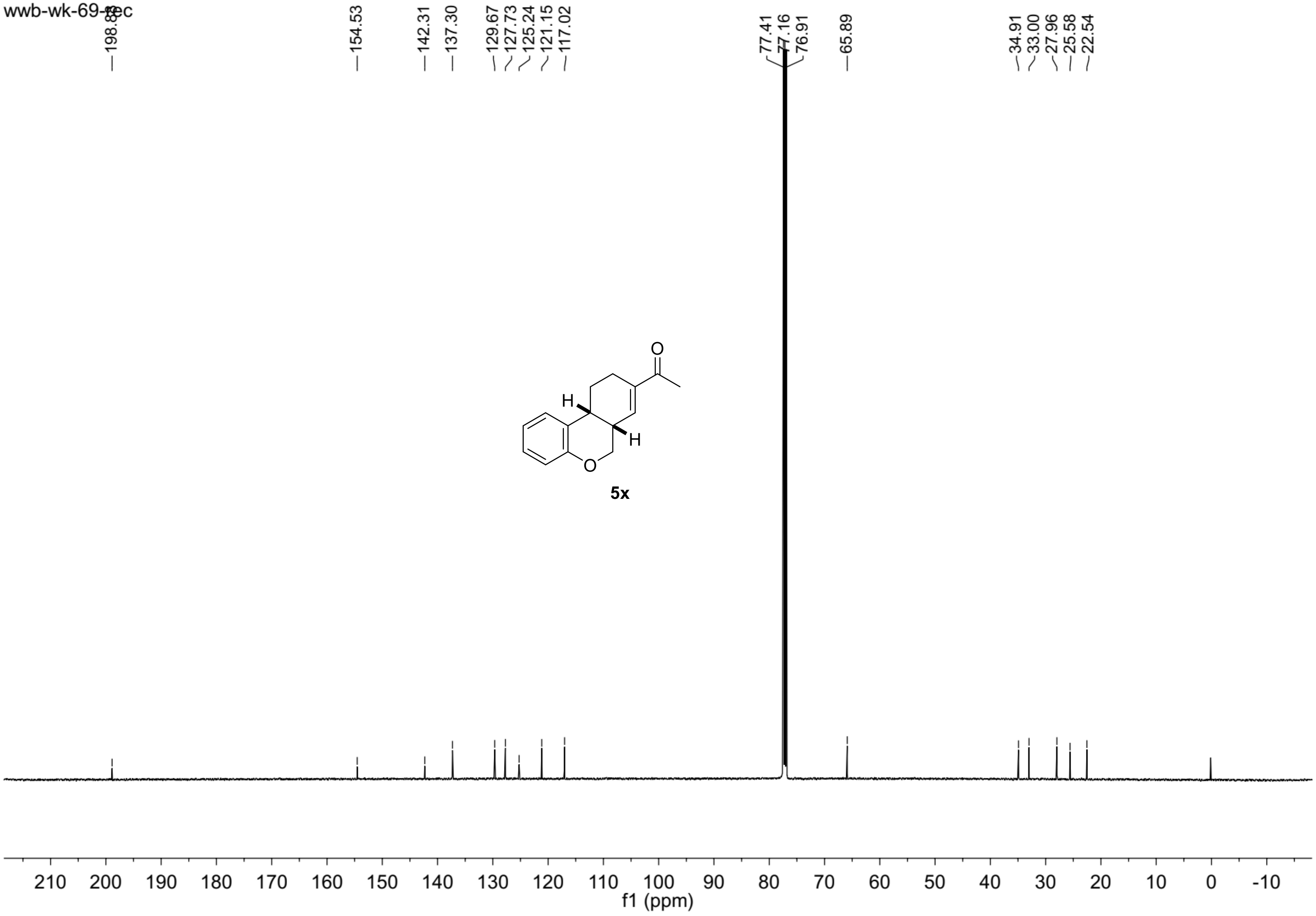

wwb-wj-137-2
7.258
7.152
7.131
6.875
6.872
6.864
6.861
6.770
6.763
6.749
6.742
6.614
6.608
3.776
2.870
2.862
2.816
2.803
2.791
2.656
2.643
2.474
2.462
2.453
2.323
2.258
2.244
2.231
2.217
2.193
2.167
1.971
1.960
1.952
1.929
1.919
1.888
1.689
1.662
1.656
1.635
1.628
0.89
0.96
1.13
0.88
3.15
0.98
1.07
2.93
1.14
1.81
2.20
3.00
7.15 7.10 6.90 6.85 6.80 6.75 6.65 6.60
2.9 2.8 2.5 2.4 2.3 2.2 2.0 1.9 1.7 1.6
.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.
f1 (ppm)
O
H
H
MeO

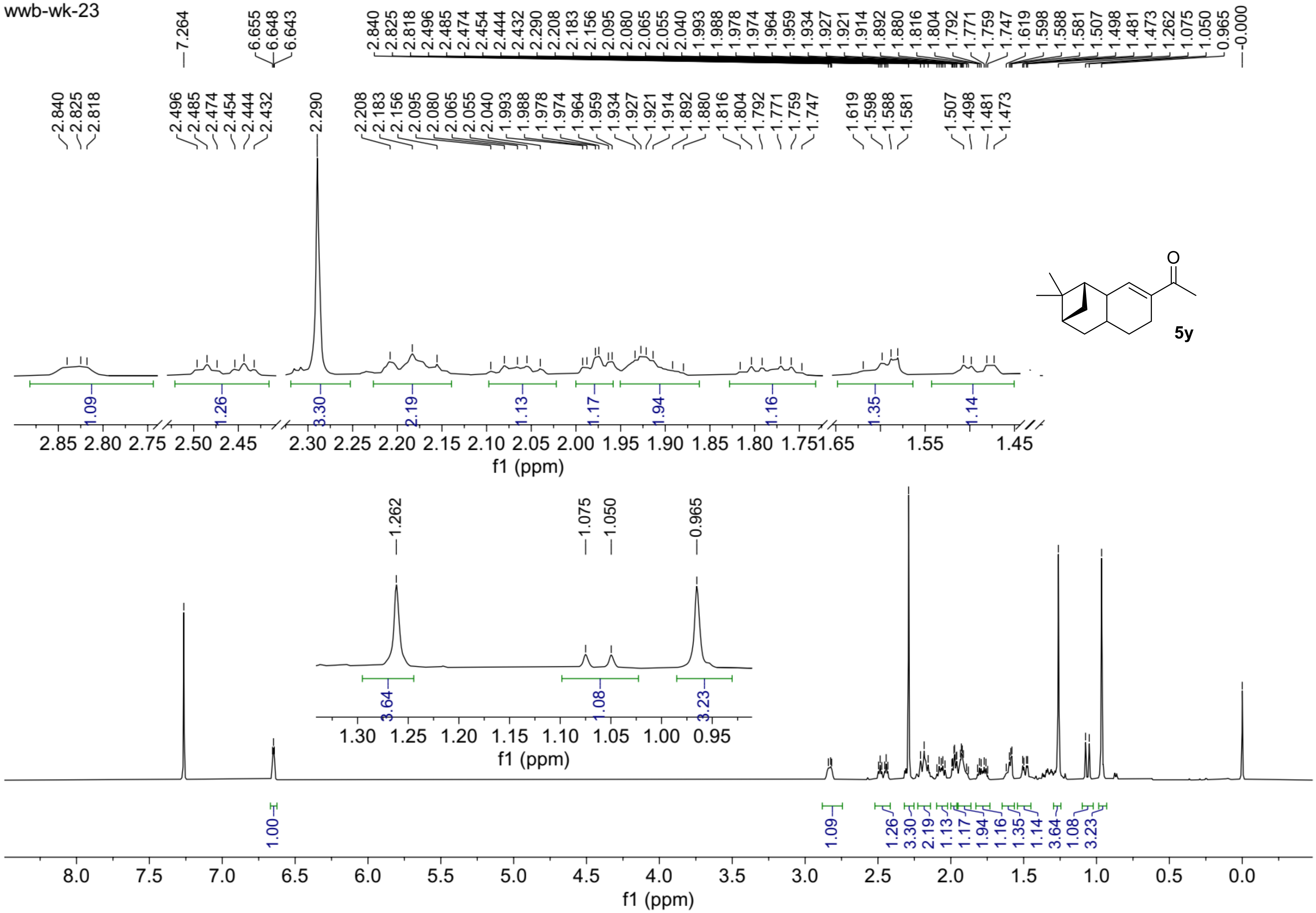
5w

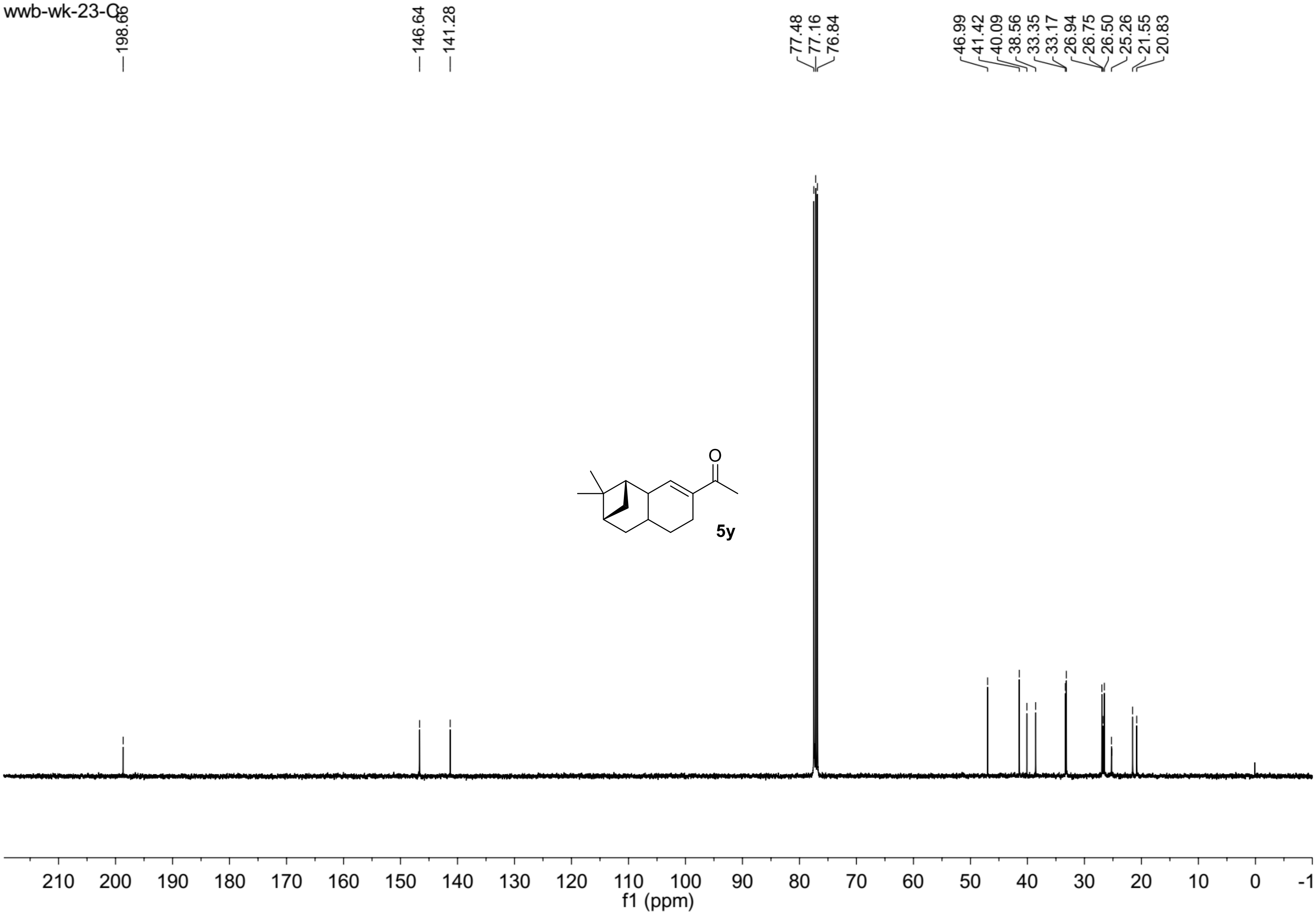

wwb-wj-137-2
199.42
157.74
143.86
139.39
137.43
131.99
130.30
113.37
112.76
77.48
77.16
76.84
55.37
36.19
36.06
29.94
28.54
25.75
25.51
23.59
O
H
H
MeO
5w
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

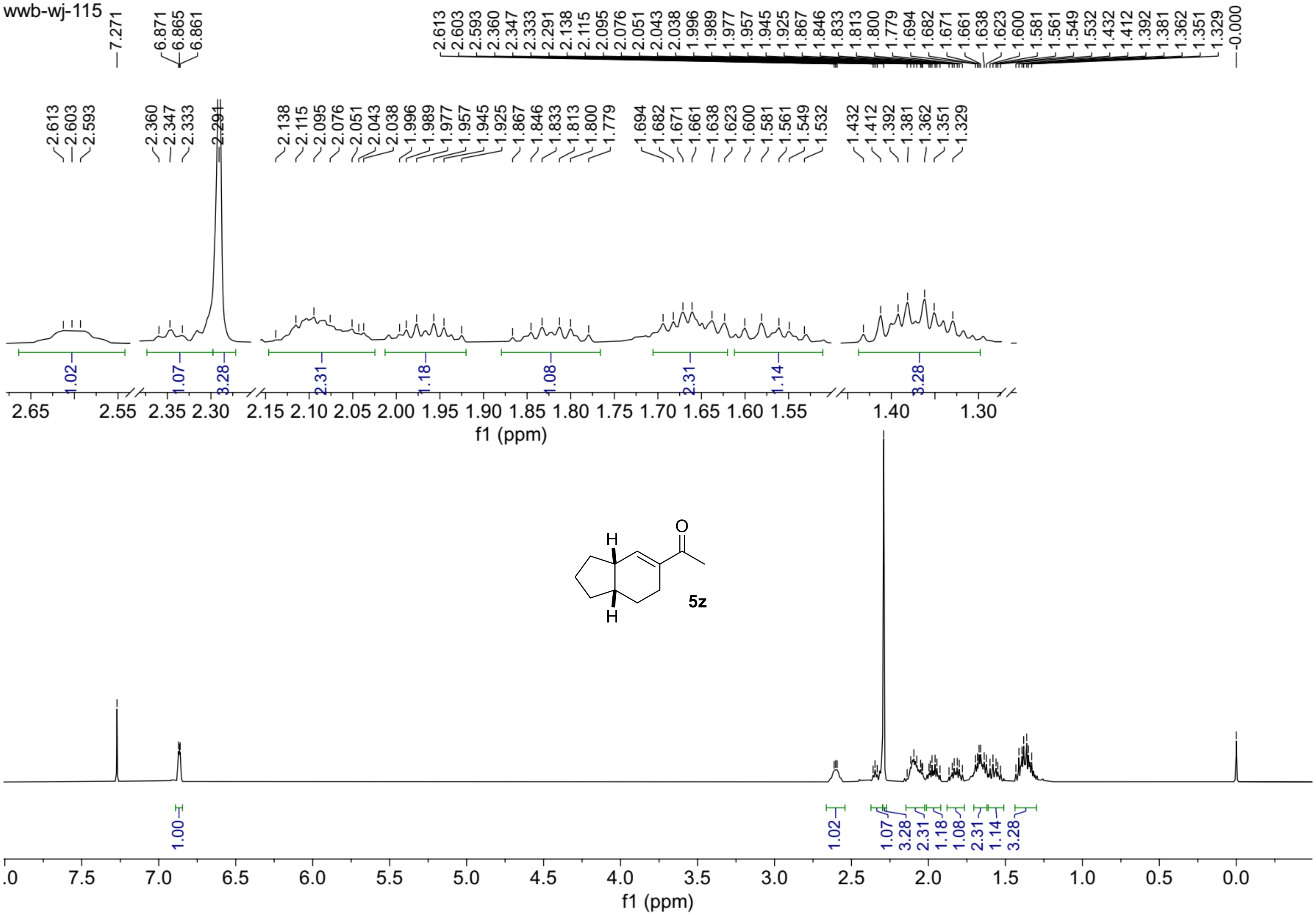

wwb-wk-69.rec
7.210 7.207 7.195 7.191
7.138 7.135 7.121 7.108 7.104
6.943 6.941 6.928 6.926 6.914 6.911
6.842 6.839 6.825 6.823
6.774 6.768 6.763
4.267 4.265 4.260 4.258 4.245 4.236
3.877 3.859 3.855 3.837
2.963 2.956 2.942
2.472 2.462 2.454 2.436 2.428 2.418
2.330
2.263 2.247 2.243 2.232 2.216 2.207 2.196
2.105 2.094 2.085 2.077 2.066
1.801 1.791 1.780 1.770 1.753 1.743 1.732 1.722
0.000
1.00
1.00
0.99
1.00
0.99
1.00
1.07
2.01
1.23
2.77
1.23
1.17
1.20
f1 (ppm)
O
H
H
O

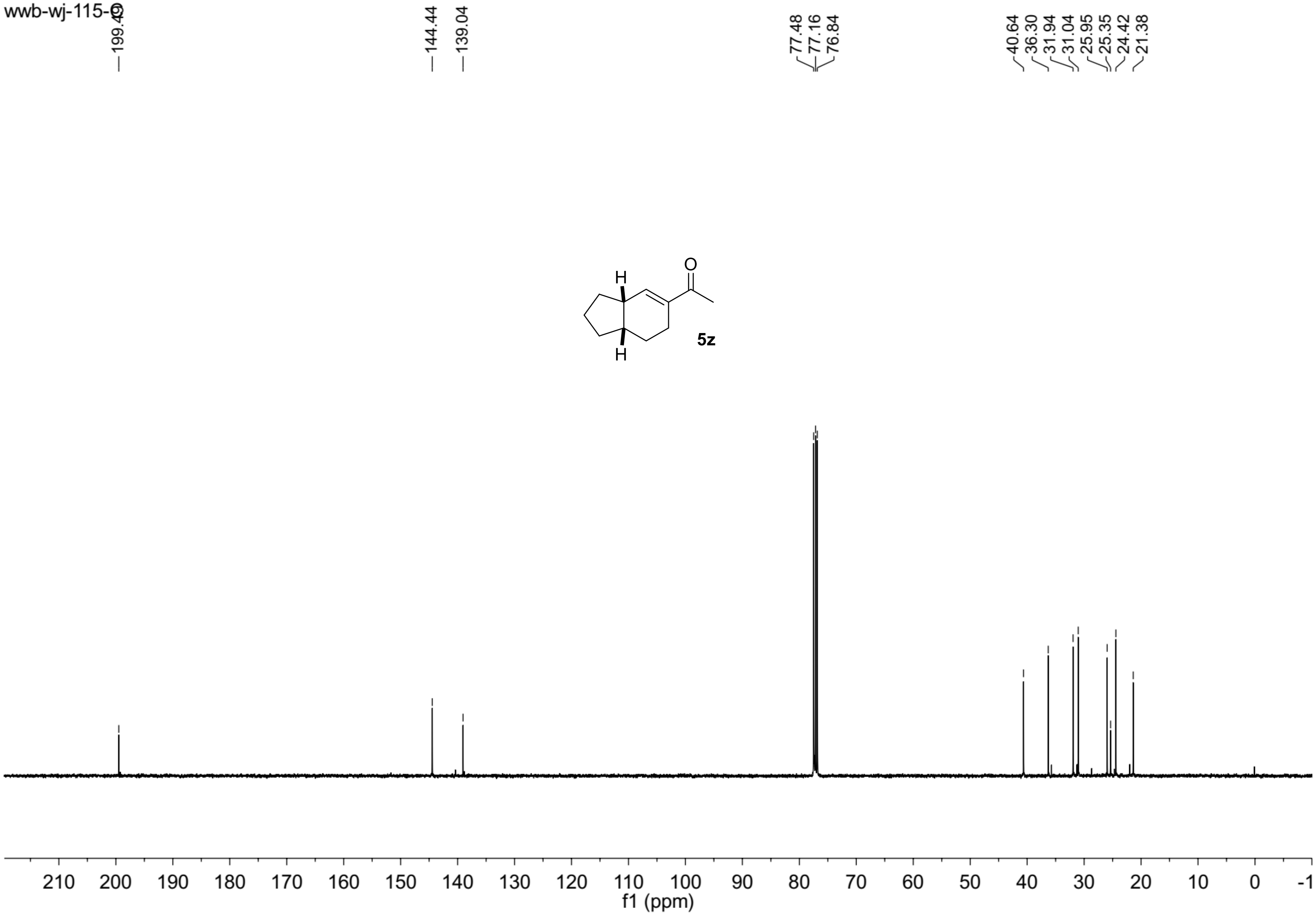
5x

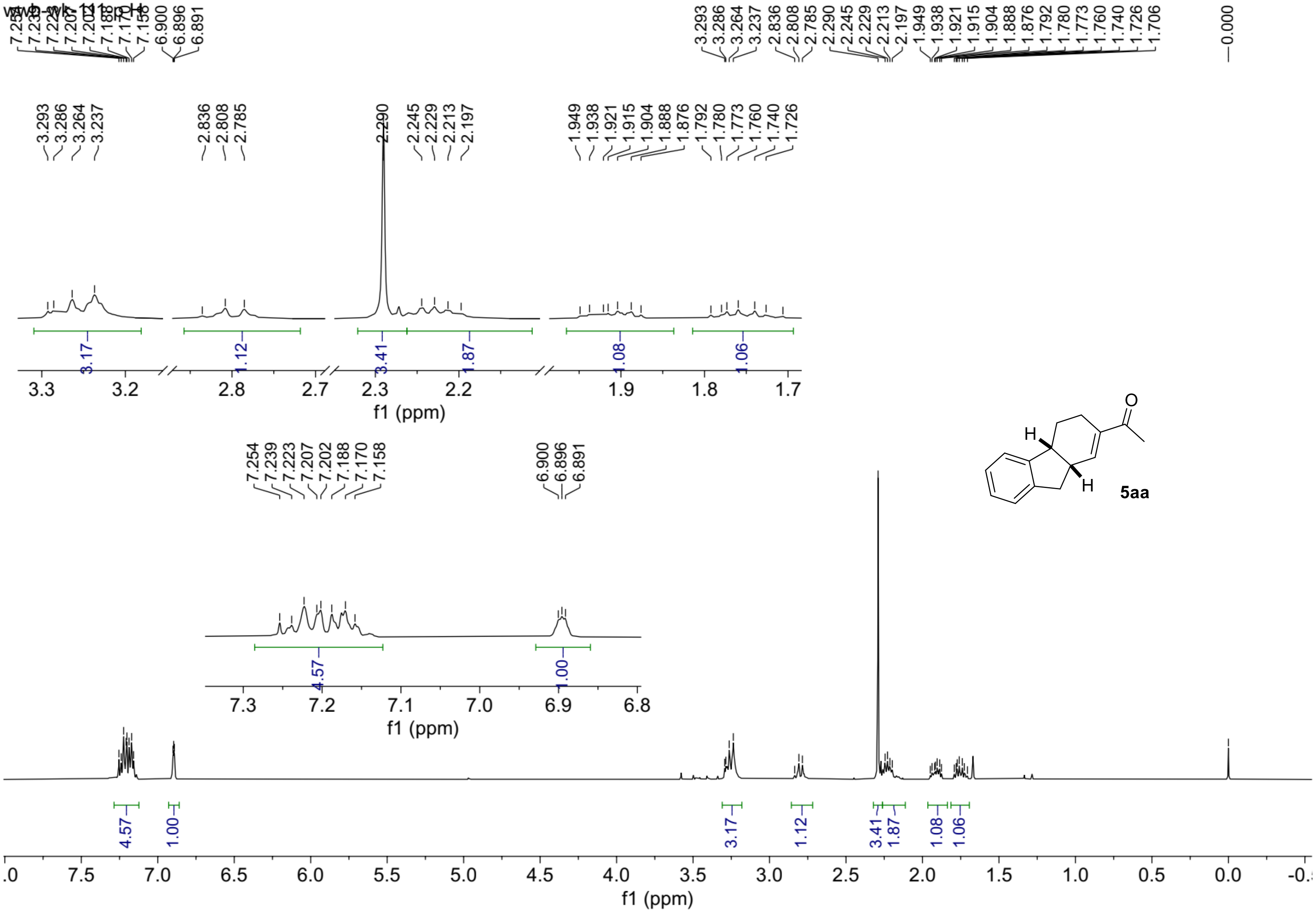

wwb-wk-69-rec
198.88
154.53
142.31
137.30
129.67
127.73
125.24
121.15
117.02
77.41
77.16
76.91
65.89
34.91
33.00
27.96
25.58
22.54
O
H
H
O
5x
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


wwb-wk-23
7.264
6.655
6.648
6.643
2.840
2.825
2.818
2.496
2.485
2.474
2.454
2.444
2.432
2.290
2.208
2.183
2.156
2.095
2.080
2.065
2.055
2.040
1.993
1.988
1.978
1.974
1.964
1.959
1.934
1.927
1.921
1.914
1.892
1.880
1.816
1.804
1.792
1.771
1.759
1.747
1.619
1.598
1.588
1.581
1.507
1.498
1.481
1.473
1.262
1.075
1.050
0.965
-0.000
O
5y
1.09
1.26
3.30
2.19
1.13
1.17
1.94
1.16
1.35
1.14
2.85 2.80 2.75 2.50 2.45 2.30 2.25 2.20 2.15 2.10 2.05 2.00 1.95 1.90 1.85 1.80 1.75 1.65 1.55 1.45
f1 (ppm)
3.64
1.08
3.23
1.30 1.25 1.20 1.15 1.10 1.05 1.00 0.95
f1 (ppm)
1.00
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)

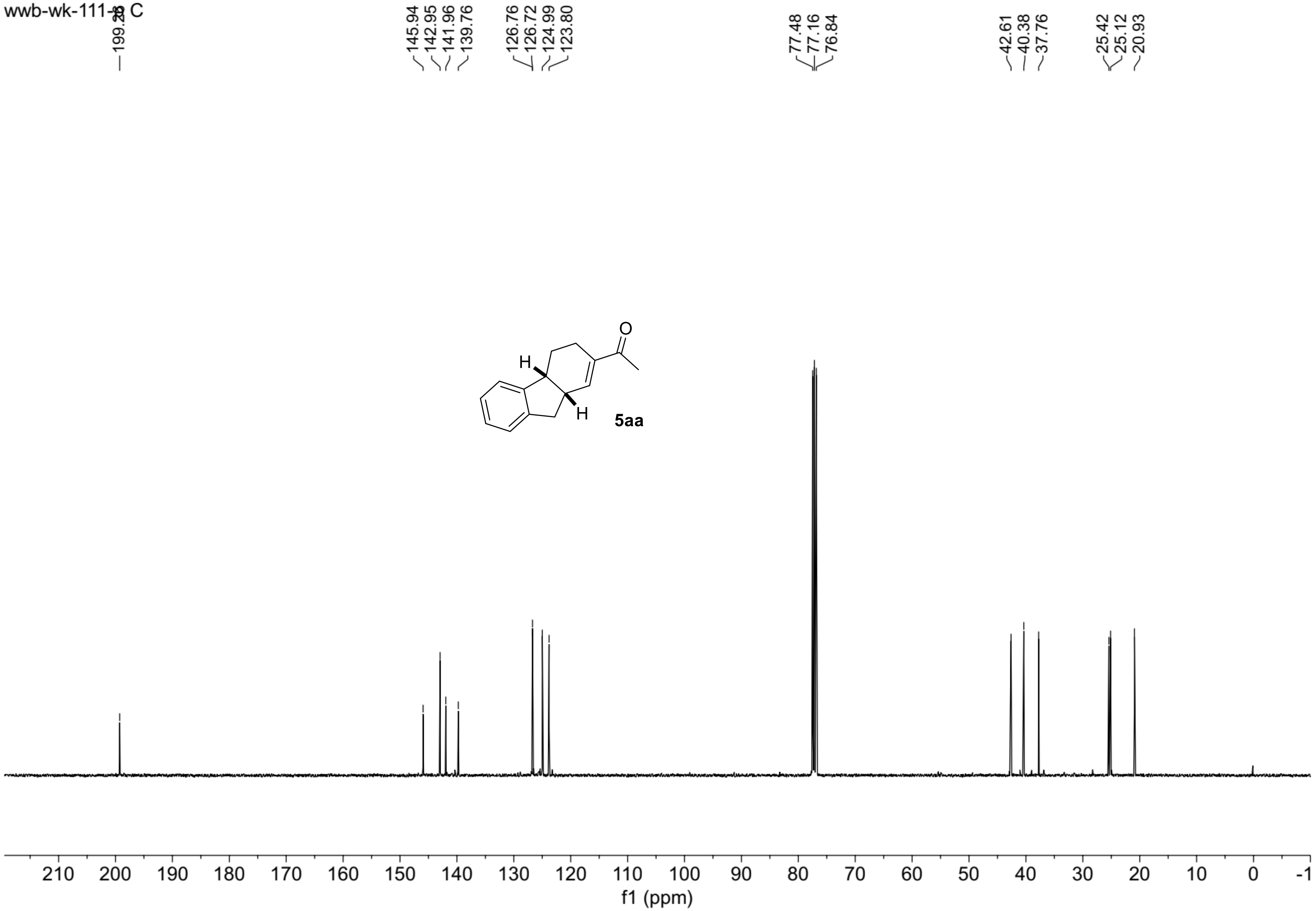

wwb-wk-23-C
198.60
146.64
141.28
77.48
77.16
76.84
46.99
41.42
40.09
38.56
33.35
33.17
26.94
26.75
26.50
25.26
21.55
20.83
O
5y
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

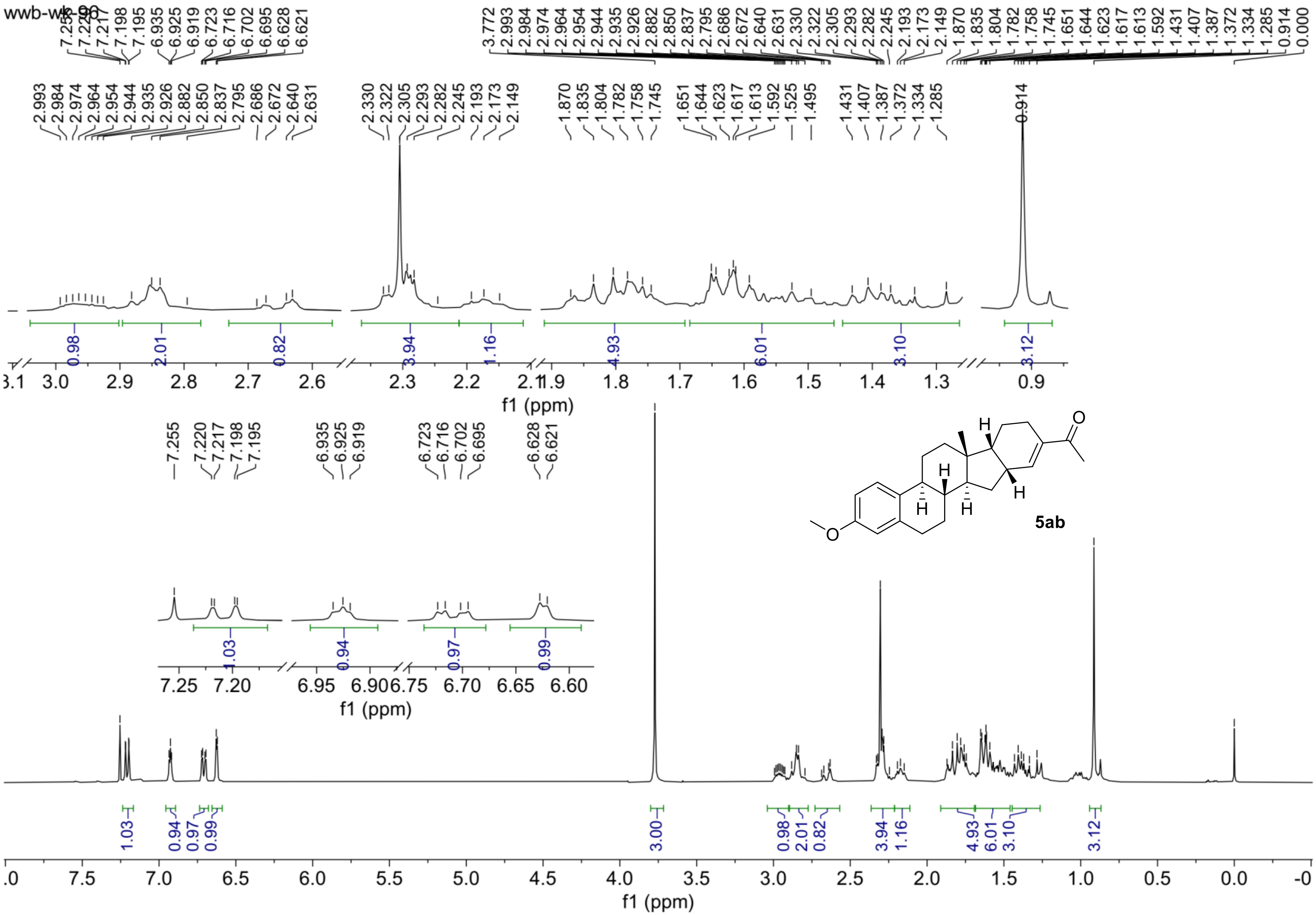

wwb-wj-115
7.271
6.871
6.865
6.861
2.613
2.603
2.593
2.360
2.347
2.333
2.291
2.138
2.115
2.095
2.076
2.051
2.043
2.038
1.996
1.989
1.977
1.957
1.945
1.925
1.867
1.846
1.833
1.813
1.800
1.779
1.694
1.682
1.671
1.661
1.638
1.623
1.600
1.581
1.561
1.549
1.532
1.432
1.412
1.392
1.381
1.362
1.351
1.329
-0.000
1.02
1.07
3.28
2.31
1.18
1.08
2.31
1.14
3.28
2.65
2.55
2.35
2.30
2.15
2.10
2.05
2.00
1.95
1.90
1.85
1.80
1.75
1.70
1.65
1.60
1.55
1.40
1.30
f1 (ppm)
O
H
H
5z
1.00
.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)

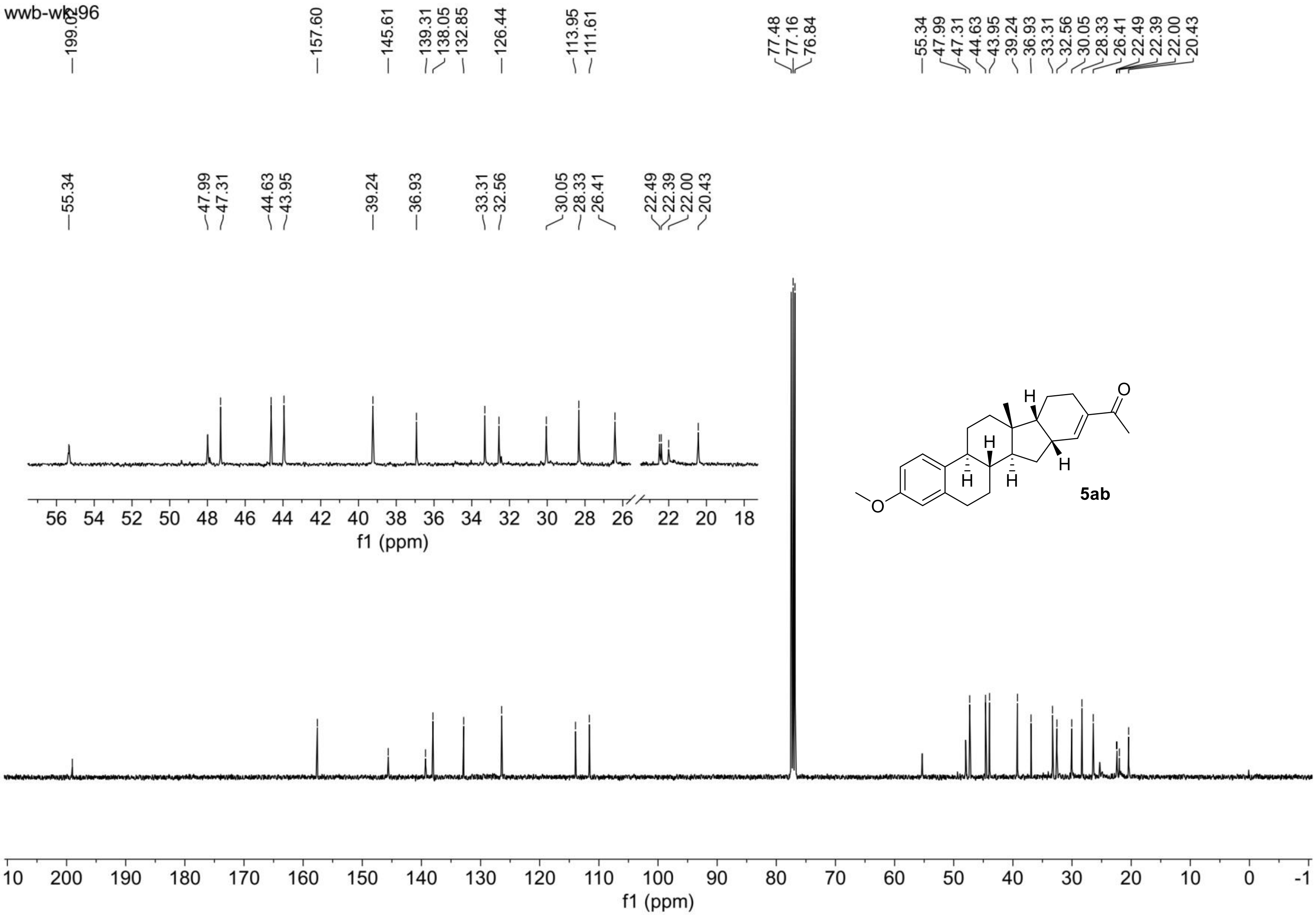

wwb-wj-115-C
199.40
144.44
139.04
77.48
77.16
76.84
40.64
36.30
31.94
31.04
25.95
25.35
24.42
21.38
H
H
O
5z
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

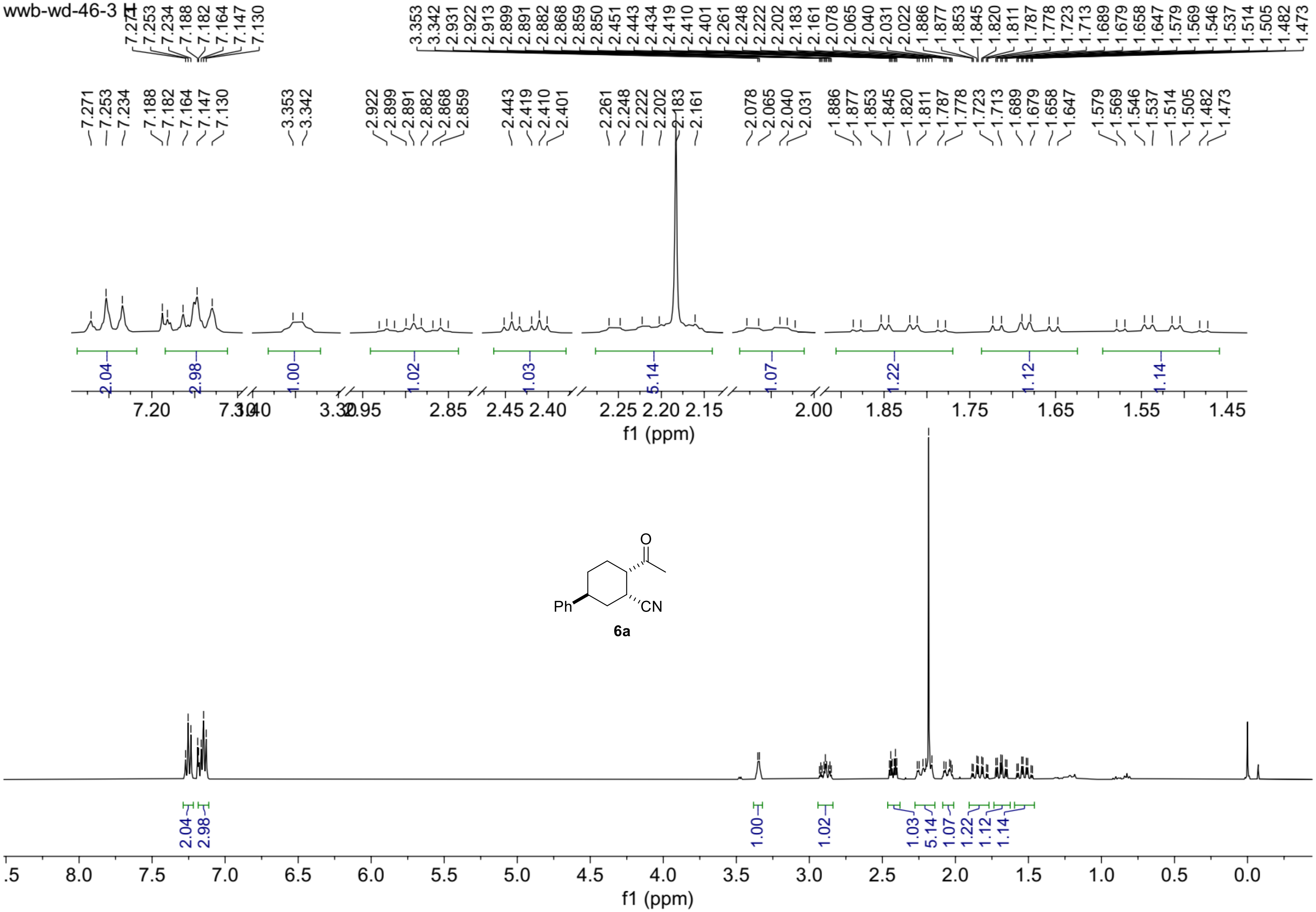


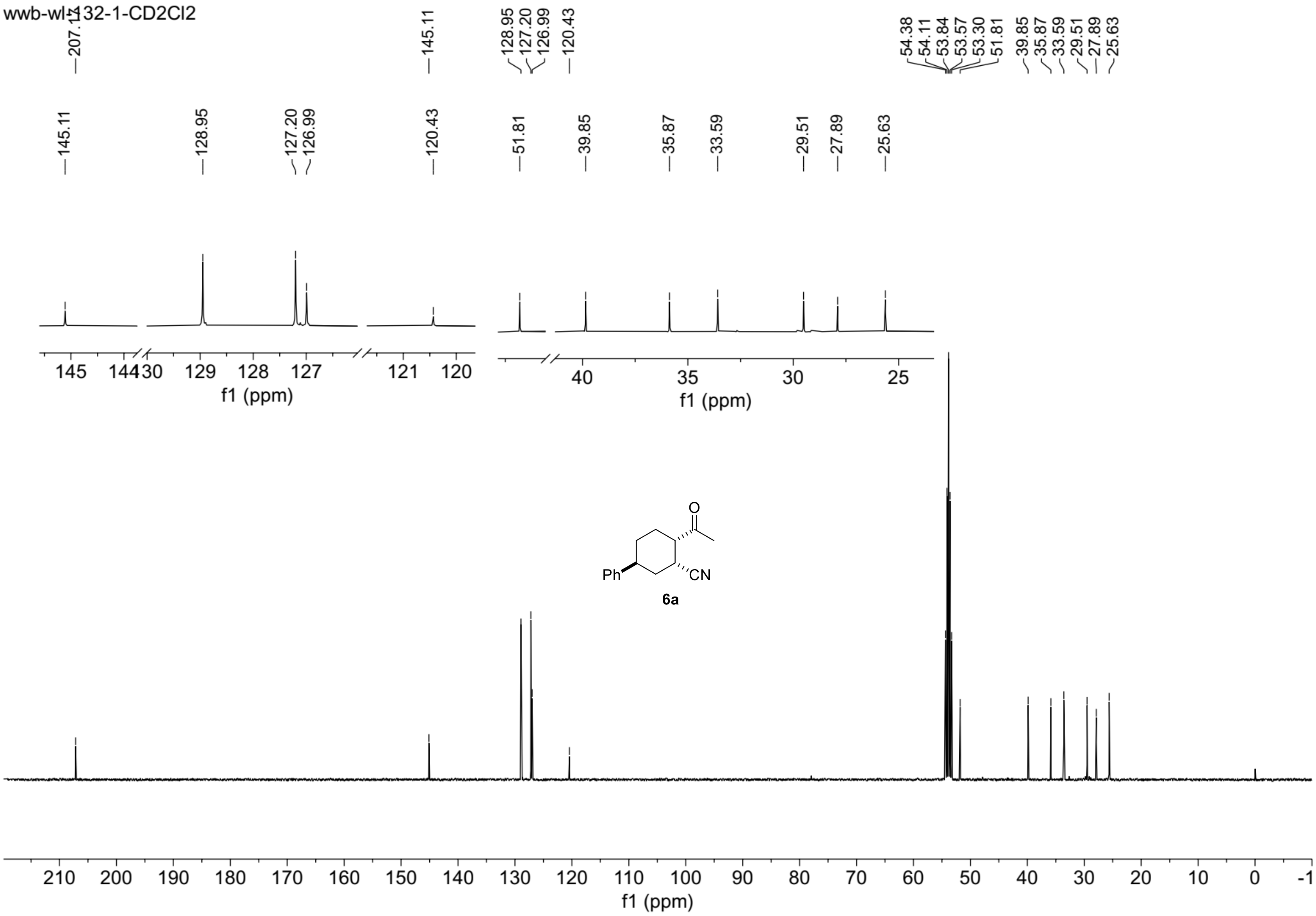
7.254
7.239
7.223
7.207
7.202
7.188
7.170
7.158
6.900
6.896
6.891
3.293
3.286
3.264
3.237
2.836
2.808
2.785
2.290
2.245
2.229
2.213
2.197
1.949
1.938
1.921
1.915
1.904
1.888
1.876
1.792
1.780
1.773
1.760
1.740
1.726
1.706
0.000
3.17
1.12
3.41
1.87
1.08
1.06
4.57
1.00
f1 (ppm)
H
H
O
5aa

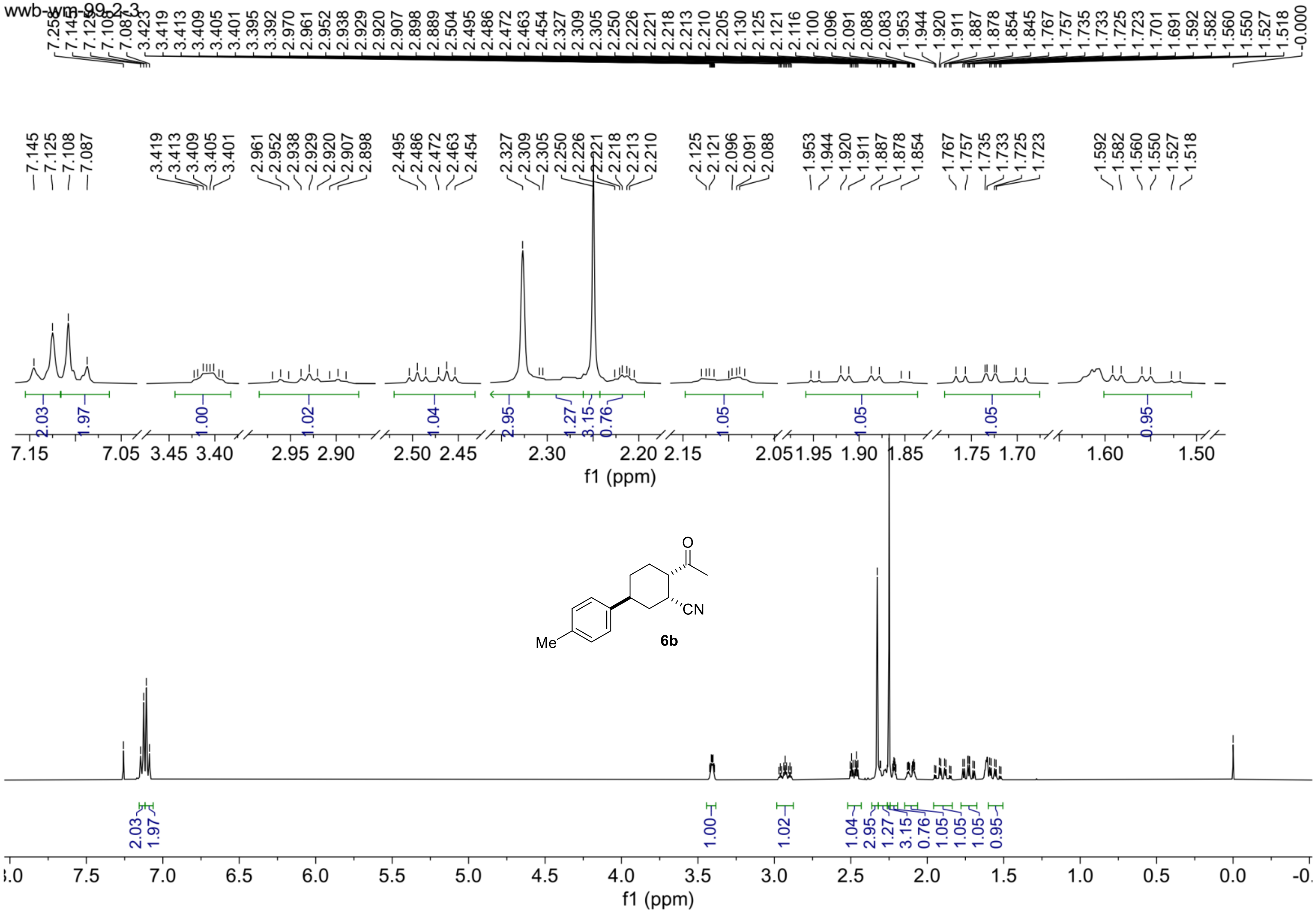

wwb-wk-111-p C
199.26
145.94
142.95
141.96
139.76
126.76
126.72
124.99
123.80
77.48
77.16
76.84
42.61
40.38
37.76
25.42
25.12
20.93
O
H
H
5aa
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

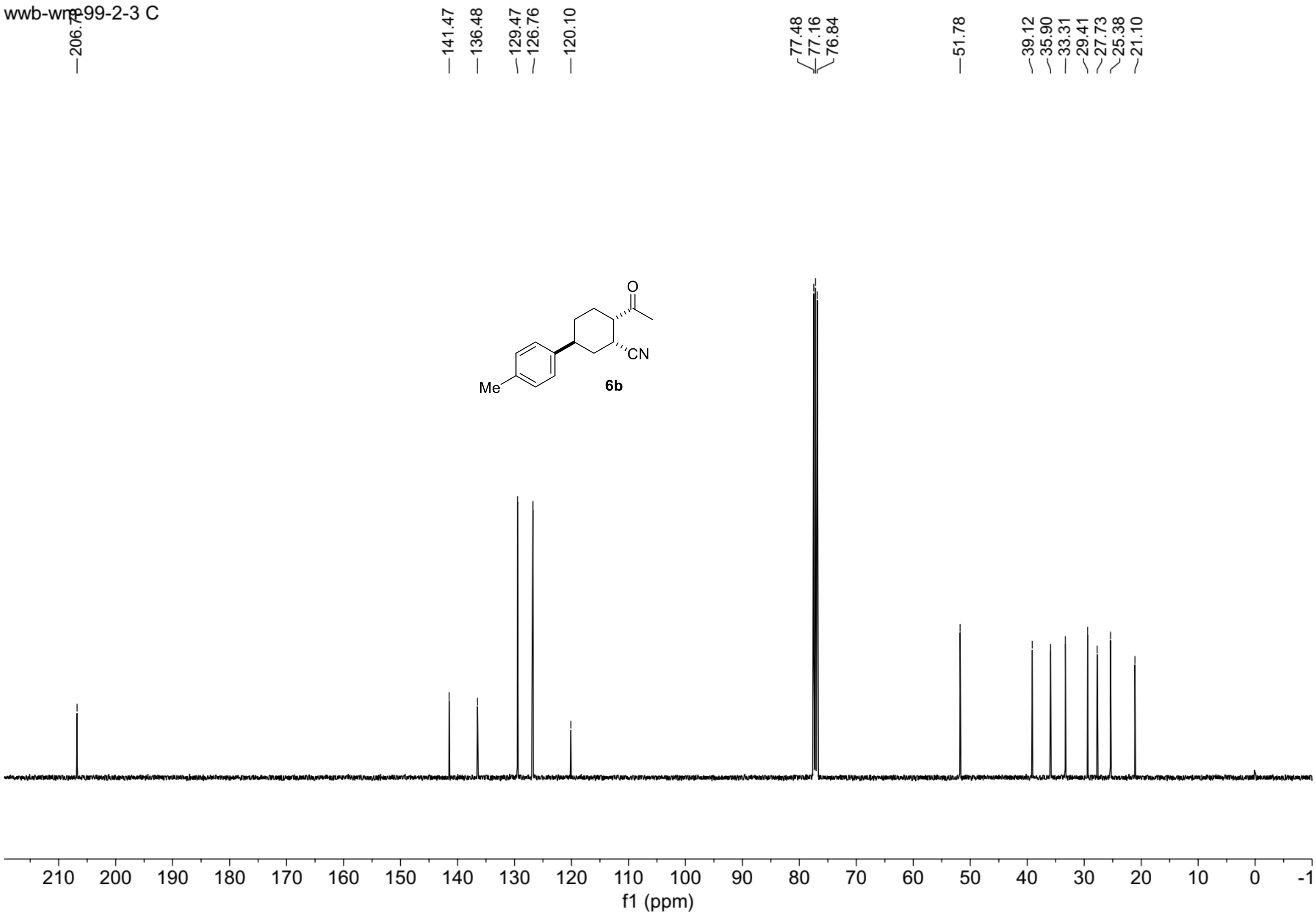

wwb-wk-96
7.255 7.220 7.217 7.198 7.195 6.935 6.925 6.919 6.723 6.716 6.702 6.695 6.628 6.621
3.772 2.993 2.984 2.974 2.964 2.954 2.944 2.935 2.926 2.882 2.850 2.837 2.795 2.686 2.672 2.640 2.631 2.330 2.322 2.305 2.293 2.282 2.245 2.193 2.173 2.149 1.870 1.835 1.804 1.782 1.758 1.745 1.651 1.644 1.623 1.617 1.613 1.592 1.431 1.407 1.387 1.372 1.334 1.285 0.914 0.000
2.993 2.984 2.974 2.964 2.954 2.944 2.935 2.926 2.882 2.850 2.837 2.795 2.686 2.672 2.640 2.631
2.330 2.322 2.305 2.293 2.282 2.245 2.193 2.173 2.149
1.870 1.835 1.804 1.782 1.758 1.745
1.651 1.644 1.623 1.617 1.613 1.592 1.525 1.495
1.431 1.407 1.387 1.372 1.334 1.285
0.914
0.98 2.01 0.82 3.94 1.16 4.93 6.01 3.10 3.12
3.1 3.0 2.9 2.8 2.7 2.6 2.3 2.2 2.1 1.9 1.8 1.7 1.6 1.5 1.4 1.3 0.9
f1 (ppm)
7.255 7.220 7.217 7.198 7.195
6.935 6.925 6.919
6.723 6.716 6.702 6.695
6.628 6.621
1.03 0.94 0.97 0.99
7.25 7.20 6.95 6.90 6.75 6.70 6.65 6.60
f1 (ppm)
H
O
H
H
H
H
O
5ab
1.03 0.94 0.97 0.99 3.00 0.98 2.01 0.82 3.94 1.16 4.93 6.01 3.10 3.12
.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)

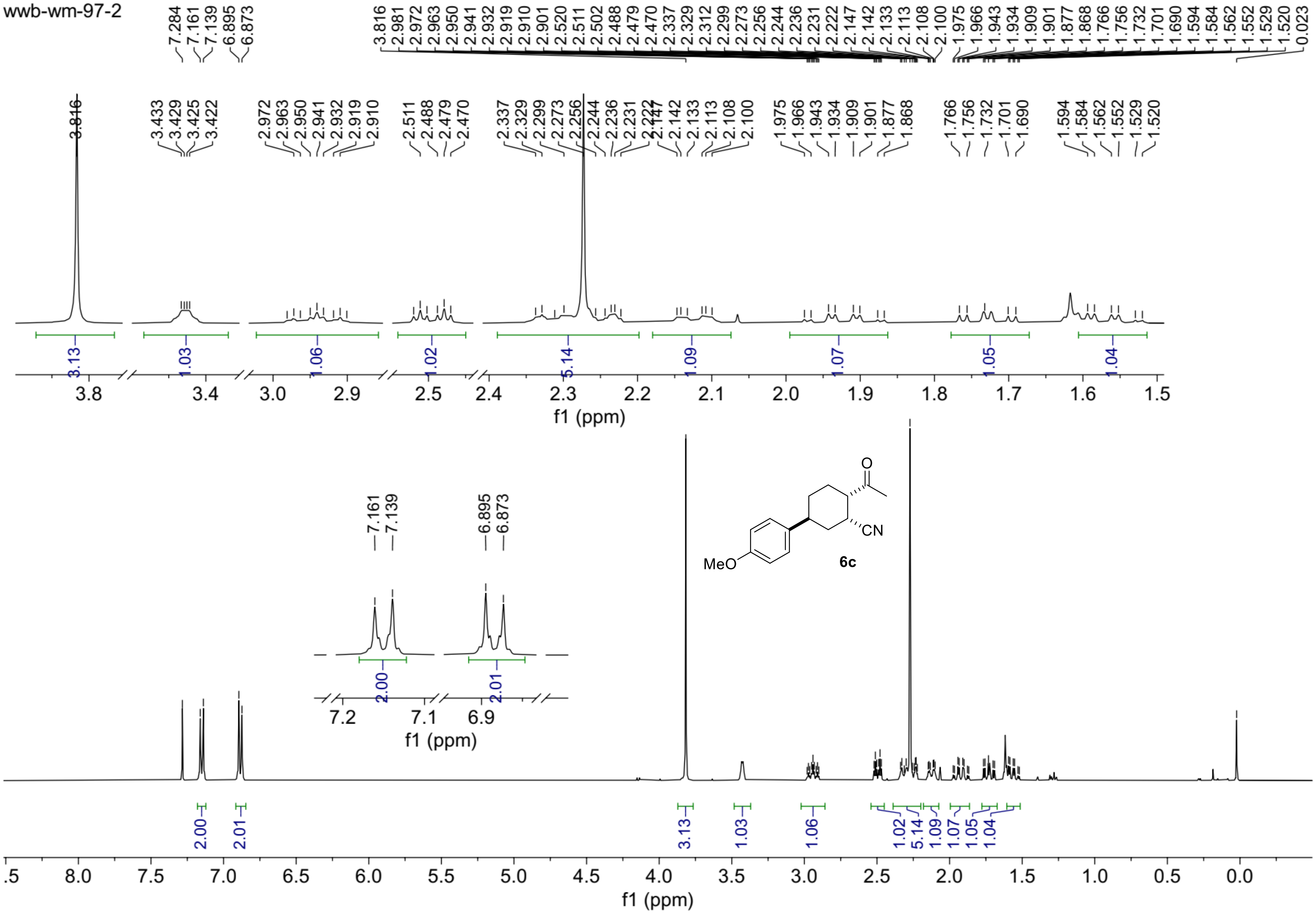

wwb-wb-96
199.02
157.60
145.61
139.31
138.05
132.85
126.44
113.95
111.61
77.48
77.16
76.84
55.34
47.99
47.31
44.63
43.95
39.24
36.93
33.31
32.56
30.05
28.33
26.41
22.49
22.39
22.00
20.43
55.34
47.99
47.31
44.63
43.95
39.24
36.93
33.31
32.56
30.05
28.33
26.41
22.49
22.39
22.00
20.43
56 54 52 50 48 46 44 42 40 38 36 34 32 30 28 26 22 20 18
f1 (ppm)
H
H
H
H
H
O
O
5ab
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)


wwb-wd-46-3 H
7.271
7.253
7.234
7.188
7.182
7.164
7.147
7.130
3.353
3.342
2.931
2.922
2.913
2.899
2.891
2.882
2.868
2.859
2.850
2.451
2.443
2.434
2.419
2.410
2.401
2.261
2.248
2.222
2.202
2.183
2.161
2.078
2.065
2.040
2.031
1.886
1.877
1.853
1.845
1.820
1.811
1.787
1.778
1.723
1.713
1.689
1.679
1.658
1.647
1.579
1.569
1.546
1.537
1.514
1.505
1.482
1.473
2.04
2.98
1.00
1.02
1.03
5.14
1.07
1.22
1.12
1.14
f1 (ppm)
O
Ph
CN
6a
.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)

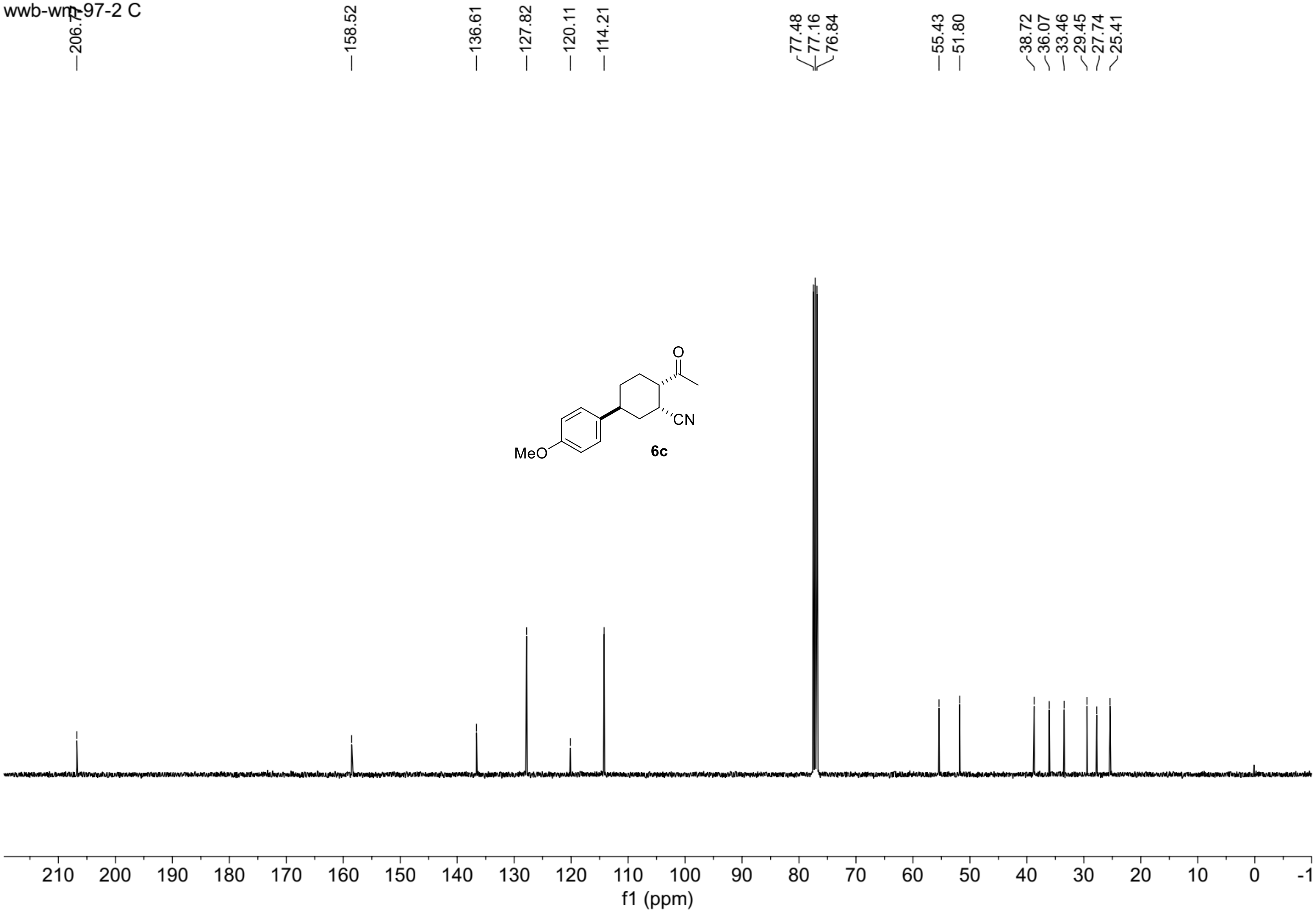

wwb-wl-132-1-CD2Cl2
207.11
145.11
128.95
127.20
126.99
120.43
54.38
54.11
53.84
53.57
53.30
51.81
39.85
35.87
33.59
29.51
27.89
25.63
145.11
128.95
127.20
126.99
120.43
51.81
39.85
35.87
33.59
29.51
27.89
25.63
145 144 130 129 128 127 121 120
f1 (ppm)
40 35 30 25
f1 (ppm)
O
Ph
CN
6a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

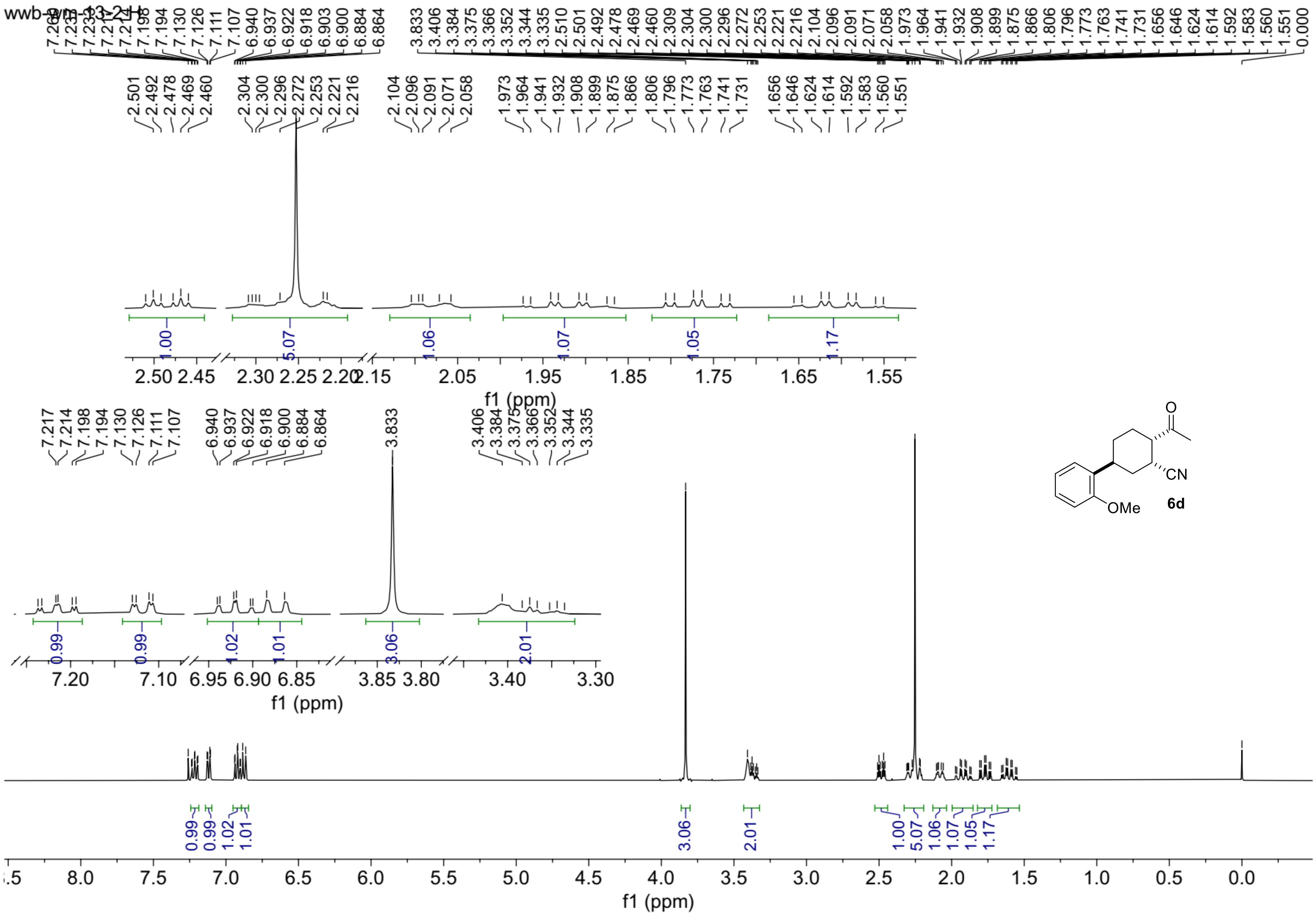

wwb-wm-99-2-3
7.258
7.145
7.125
7.108
7.087
3.423
3.419
3.413
3.409
3.405
3.401
3.395
3.392
2.970
2.961
2.952
2.938
2.929
2.920
2.907
2.898
2.889
2.504
2.495
2.486
2.472
2.463
2.454
2.327
2.309
2.305
2.250
2.226
2.221
2.218
2.213
2.210
2.205
2.130
2.125
2.121
2.116
2.100
2.096
2.091
2.088
2.083
1.953
1.944
1.920
1.911
1.887
1.878
1.854
1.845
1.767
1.757
1.735
1.733
1.725
1.723
1.701
1.691
1.592
1.582
1.560
1.550
1.527
1.518
-0.000
2.03
1.97
1.00
1.02
1.04
2.95
1.27
3.15
0.76
1.05
1.05
1.05
0.95
7.15
7.05
3.45
3.40
2.95
2.90
2.50
2.45
2.30
2.20
2.15
2.05
1.95
1.90
1.85
1.75
1.70
1.60
1.50
f1 (ppm)
O
CN
Me
6b
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)

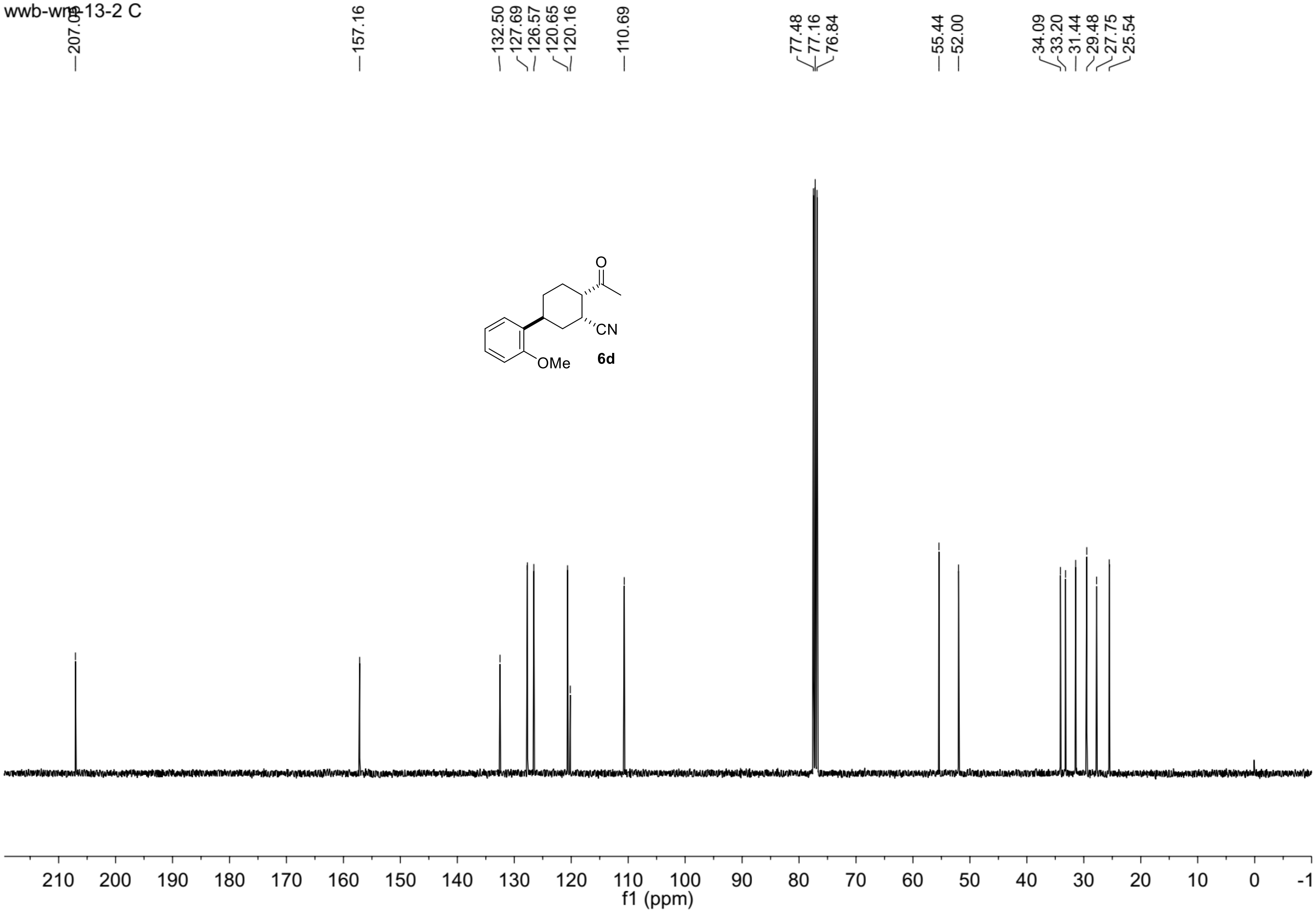

wwb-wm-99-2-3 C
206.7
141.47
136.48
129.47
126.76
120.10
77.48
77.16
76.84
51.78
39.12
35.90
33.31
29.41
27.73
25.38
21.10
O
CN
Me
6b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

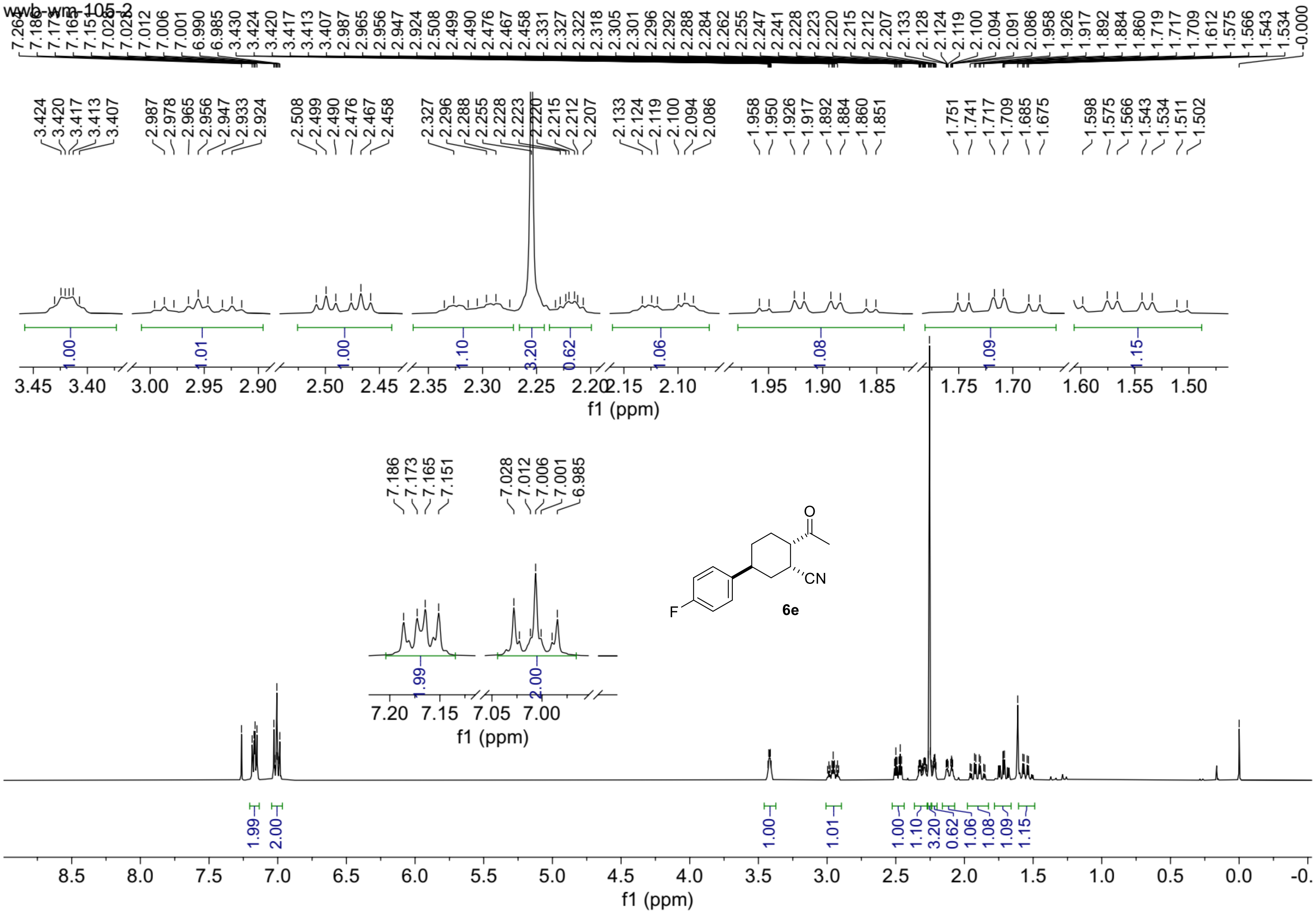

wwb-wm-97-2
7.284
7.161
7.139
6.895
6.873
3.816
2.981
2.972
2.963
2.950
2.941
2.932
2.919
2.910
2.901
2.520
2.511
2.502
2.488
2.479
2.470
2.337
2.329
2.312
2.299
2.273
2.256
2.244
2.236
2.231
2.222
2.147
2.142
2.133
2.113
2.108
2.100
1.975
1.966
1.943
1.934
1.909
1.901
1.877
1.868
1.766
1.756
1.732
1.701
1.690
1.594
1.584
1.562
1.552
1.529
1.520
0.023
3.816
3.433
3.429
3.425
3.422
3.13
1.03
1.06
1.02
5.14
1.09
1.07
1.05
1.04
3.8
3.4
3.0
2.9
2.5
2.4
2.3
2.2
2.1
2.0
1.9
1.8
1.7
1.6
1.5
f1 (ppm)
O
CN
MeO
7.161
7.139
6.895
6.873
2.00
2.01
7.2
7.1
6.9
f1 (ppm)
2.00
2.01
3.13
1.03
1.06
1.02
5.14
1.09
1.07
1.05
1.04
.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)

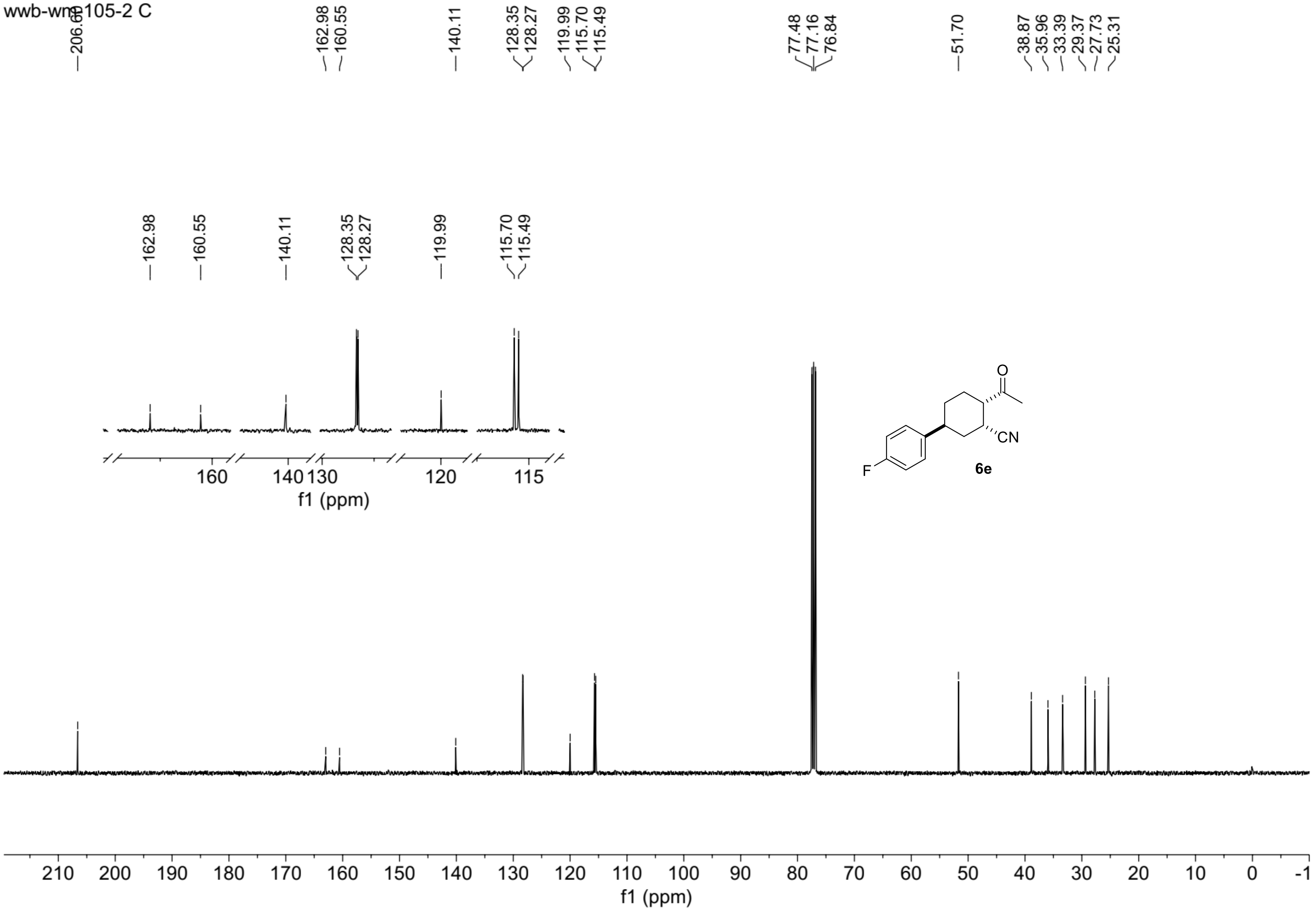
6c

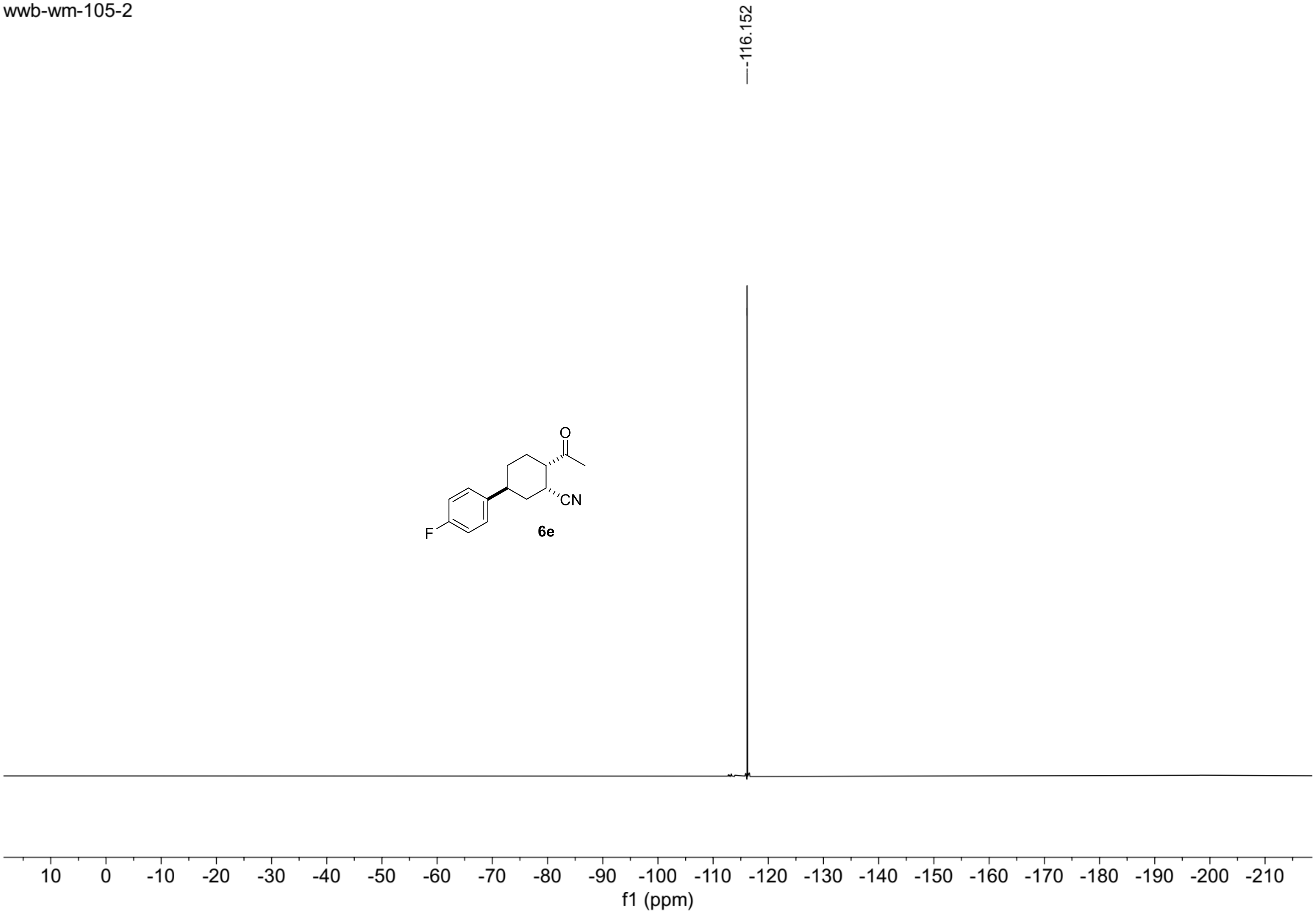

wwb-wm-97-2 C
206.7
158.52
136.61
127.82
120.11
114.21
77.48
77.16
76.84
55.43
51.80
38.72
36.07
33.46
29.45
27.74
25.41
O
CN
MeO
6c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

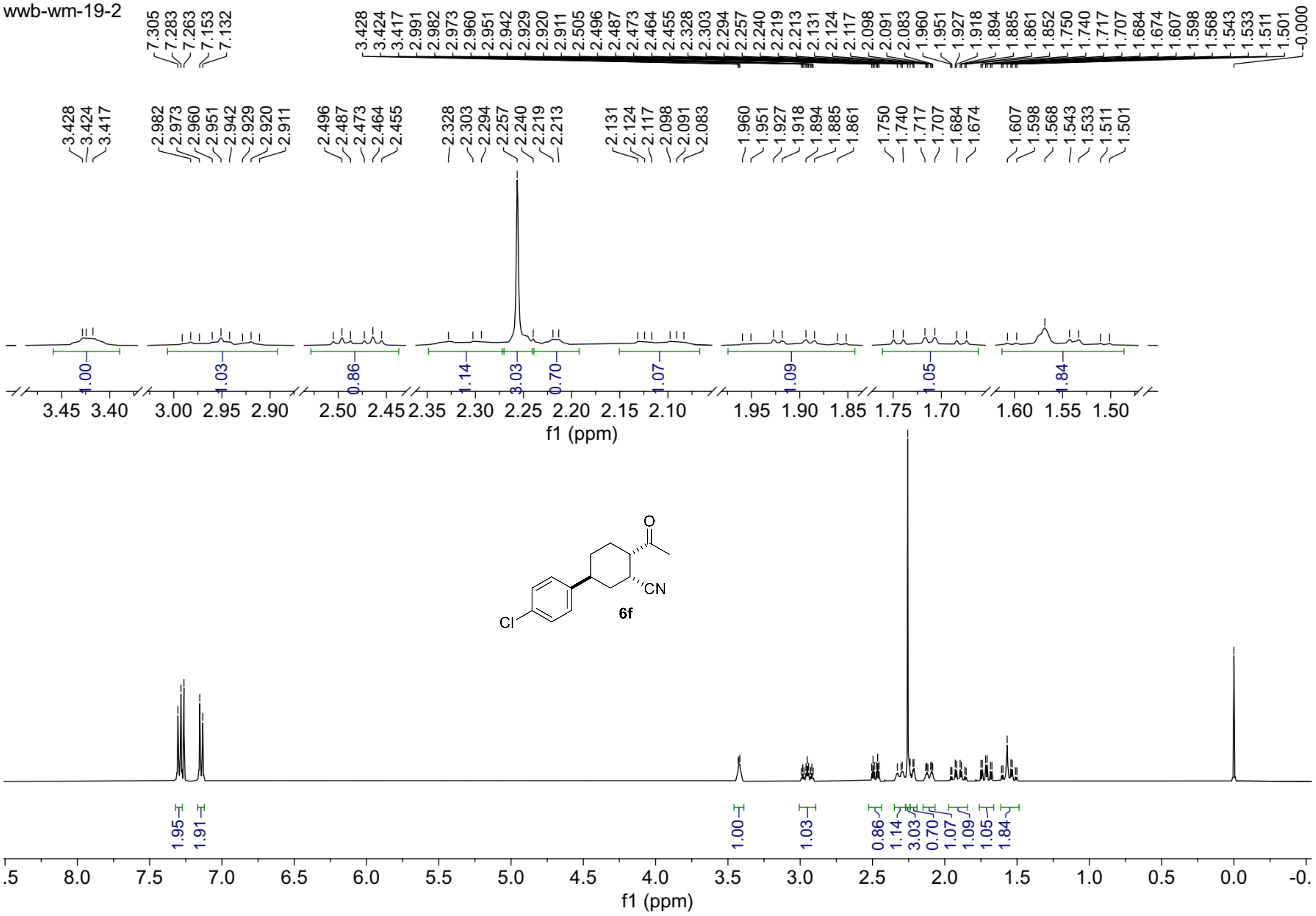

wwb-ym-13-2-1H
7.198 7.194 7.130 7.126 7.111 7.107 6.940 6.937 6.922 6.918 6.903 6.900 6.884 6.864 3.833 3.406 3.384 3.375 3.366 3.352 3.344 3.335 2.510 2.501 2.492 2.478 2.469 2.460 2.309 2.304 2.300 2.296 2.272 2.253 2.221 2.216 2.104 2.096 2.091 2.071 2.058 1.973 1.964 1.941 1.932 1.908 1.899 1.875 1.866 1.806 1.796 1.773 1.763 1.741 1.731 1.656 1.646 1.624 1.614 1.592 1.583 1.560 1.551 0.000
2.501 2.492 2.478 2.469 2.460
2.304 2.300 2.296 2.272 2.253 2.221 2.216
2.104 2.096 2.091 2.071 2.058
1.973 1.964 1.941 1.932 1.908 1.899 1.875 1.866
1.806 1.796 1.773 1.763 1.741 1.731
1.656 1.646 1.624 1.614 1.592 1.583 1.560 1.551
1.00 5.07 1.06 1.07 1.05 1.17
2.50 2.45 2.30 2.25 2.20 2.15 2.05 1.95 1.85 1.75 1.65 1.55
f1 (ppm)
7.217 7.214 7.198 7.194 7.130 7.126 7.111 7.107
6.940 6.937 6.922 6.918 6.900 6.884 6.864
3.833
3.406 3.384 3.375 3.366 3.352 3.344 3.335
0.99 0.99 1.02 1.01 3.06 2.01
7.20 7.10 6.95 6.90 6.85 3.85 3.80 3.40 3.30
f1 (ppm)
O
CN
OMe
6d
0.99 0.99 1.02 1.01 3.06 2.01 1.00 5.07 1.06 1.07 1.05 1.17
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)

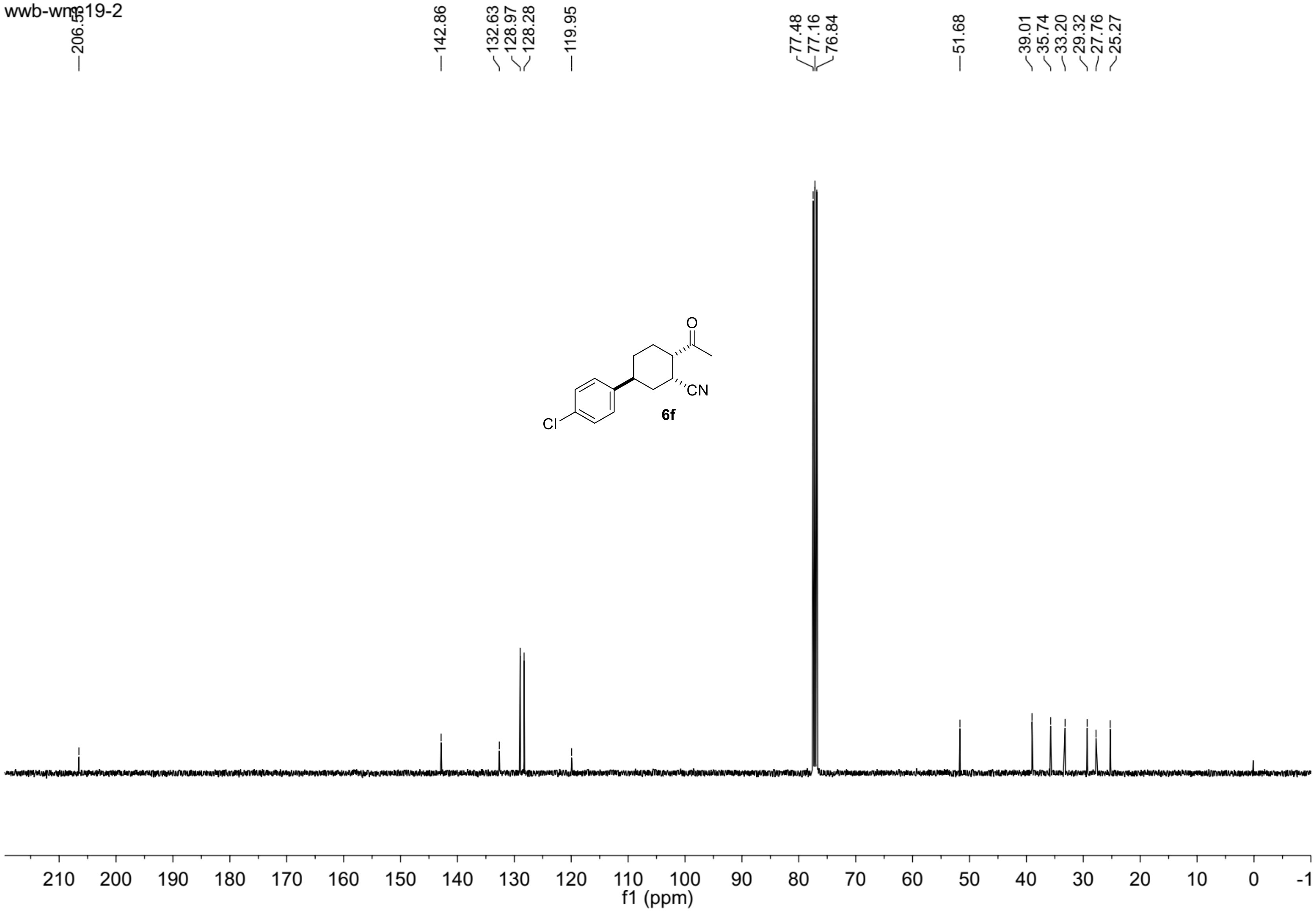

wwb-wm-13-2 C
207.04
157.16
132.50
127.69
126.57
120.65
120.16
110.69
77.48
77.16
76.84
55.44
52.00
34.09
33.20
31.44
29.48
27.75
25.54
O
CN
OMe
6d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

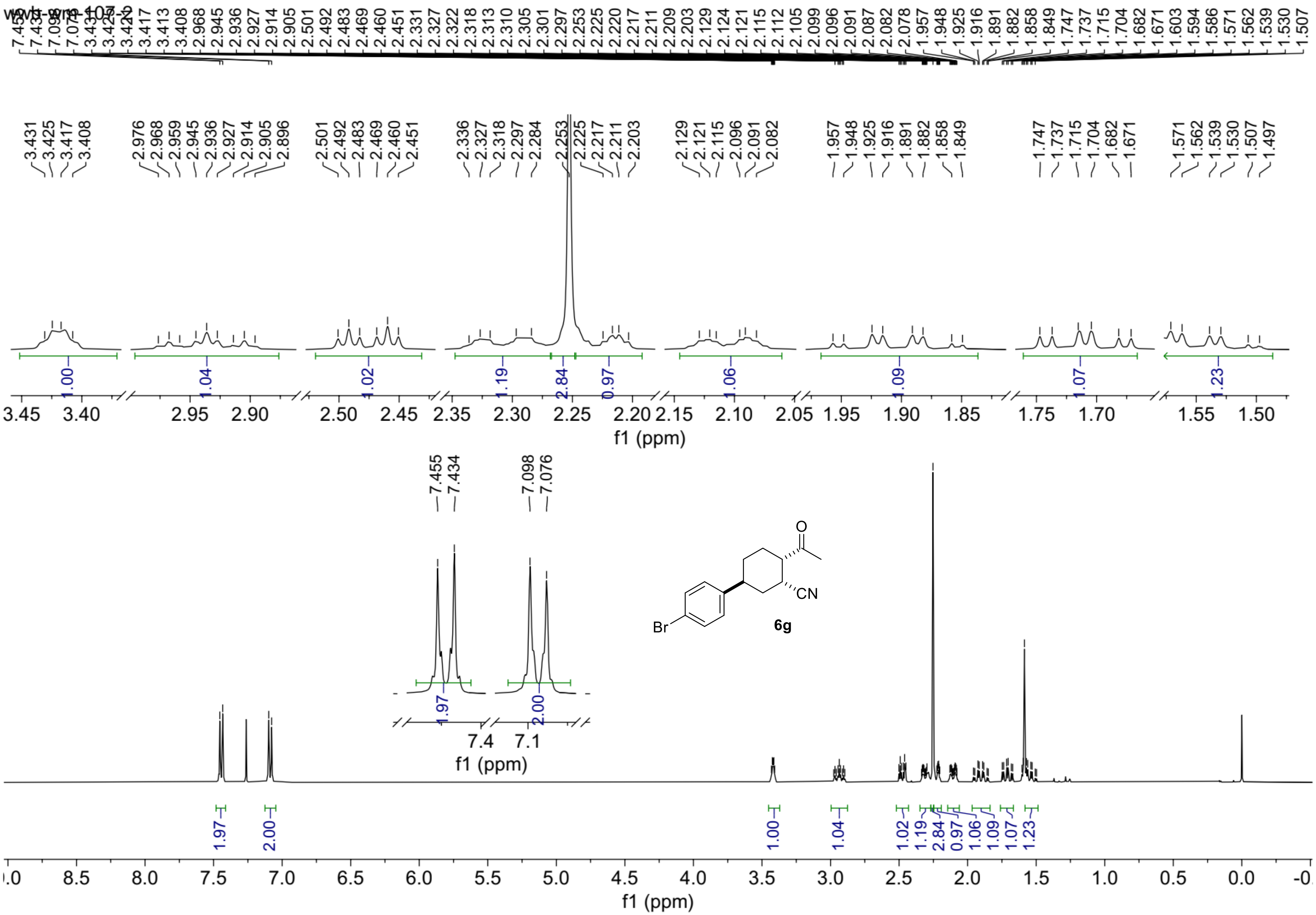

wwb-wm-105-2
3.424 3.420 3.417 3.413 3.407
2.987 2.978 2.965 2.956 2.947 2.933 2.924
2.508 2.499 2.490 2.476 2.467 2.458
2.327 2.296 2.288 2.255 2.228 2.223 2.220 2.215 2.212 2.207
2.133 2.124 2.119 2.100 2.094 2.086
1.958 1.950 1.926 1.917 1.892 1.884 1.860 1.851
1.751 1.741 1.717 1.709 1.685 1.675
1.598 1.575 1.566 1.543 1.534 1.511 1.502
-0.000
1.00
1.01
1.00
1.10
3.20
0.62
1.06
1.08
1.09
1.15
f1 (ppm)
7.186 7.173 7.165 7.151
7.028 7.012 7.006 7.001 6.985
1.99
2.00
f1 (ppm)
O
CN
F
6e
f1 (ppm)

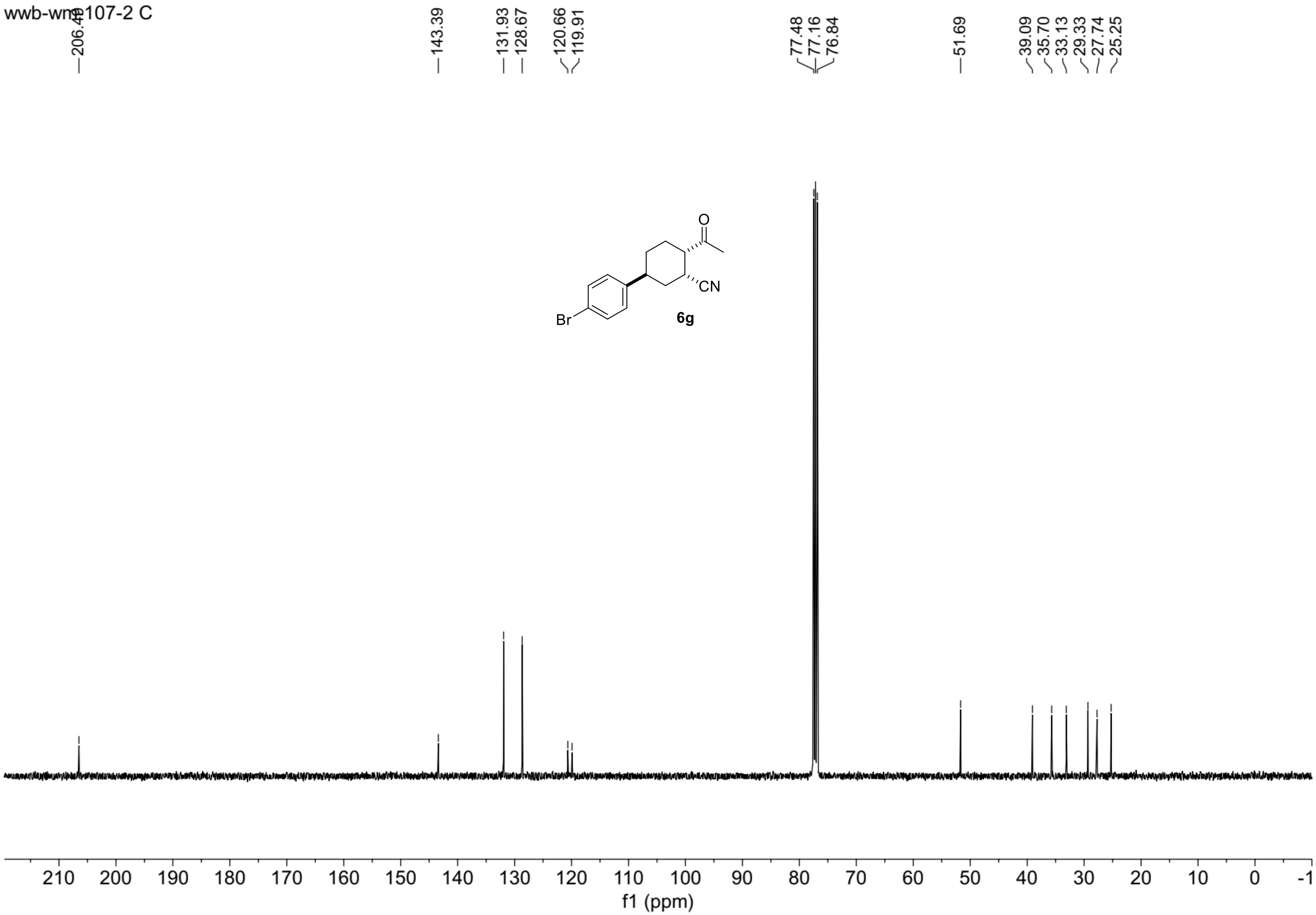

wwb-wm-105-2 C
206.61
162.98
160.55
140.11
128.35
128.27
119.99
115.70
115.49
77.48
77.16
76.84
51.70
38.87
35.96
33.39
29.37
27.73
25.31
162.98
160.55
140.11
128.35
128.27
119.99
115.70
115.49
160
140 130
120
115
f1 (ppm)
O
CN
F
6e
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

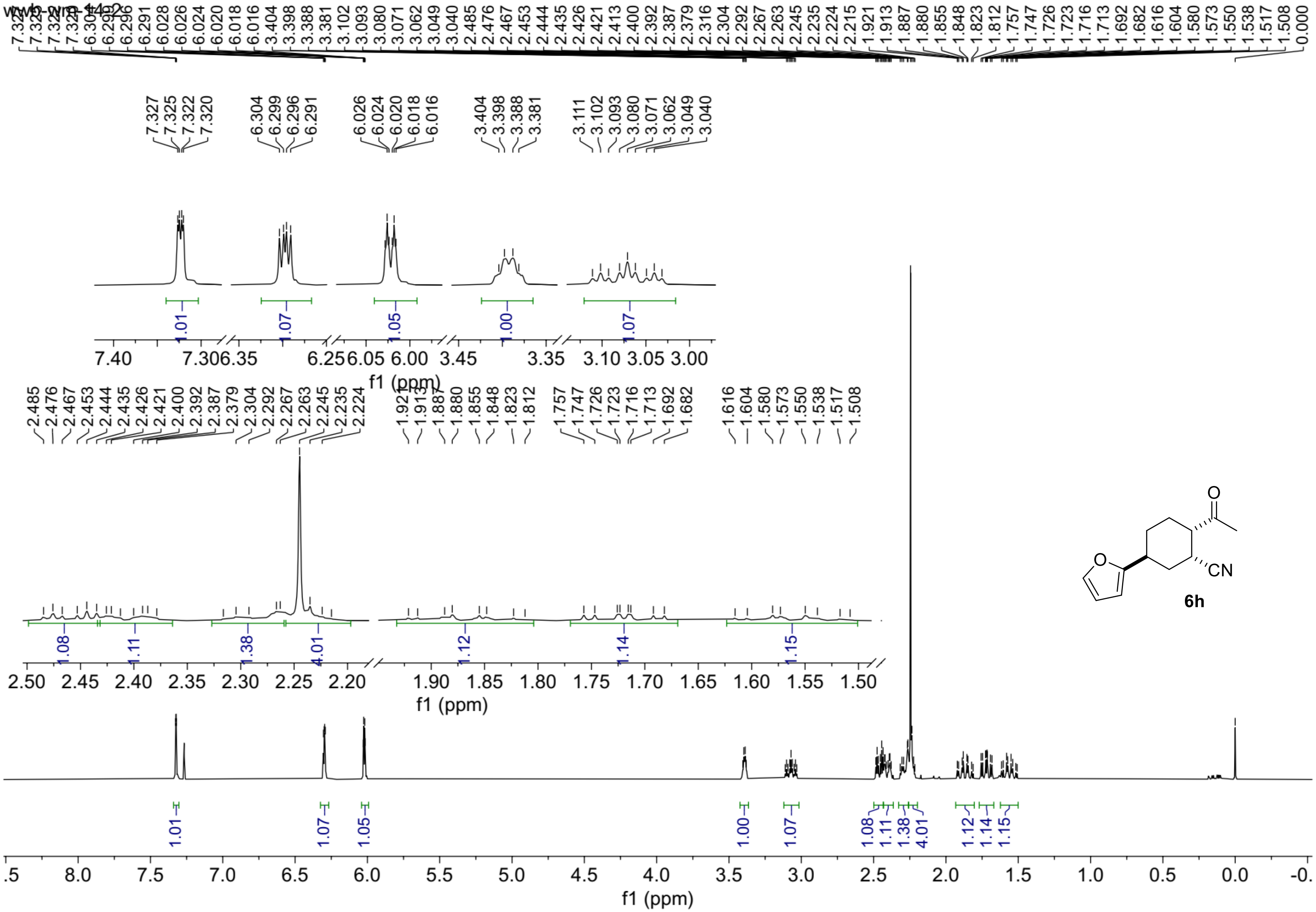

wwb-wm-105-2
-116.152
O
CN
F
6e
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

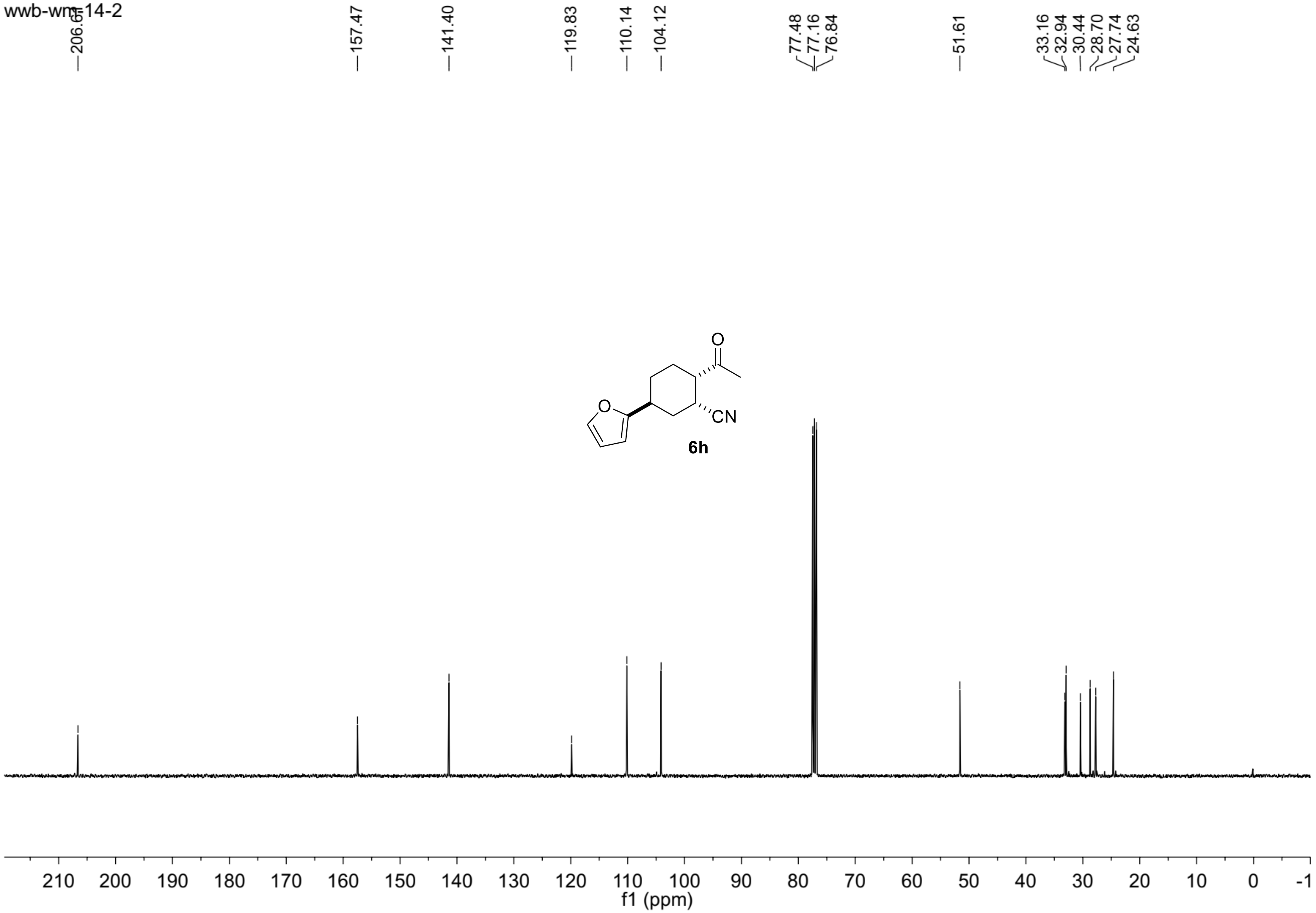

wwb-wm-19-2
7.305
7.283
7.263
7.153
7.132
3.428
3.424
3.417
2.991
2.982
2.973
2.960
2.951
2.942
2.929
2.920
2.911
2.505
2.496
2.487
2.473
2.464
2.455
2.328
2.303
2.294
2.257
2.240
2.219
2.213
2.131
2.124
2.117
2.098
2.091
2.083
1.960
1.951
1.927
1.918
1.894
1.885
1.861
1.852
1.750
1.740
1.717
1.707
1.684
1.674
1.607
1.598
1.568
1.543
1.533
1.511
1.501
-0.000
1.00
1.03
0.86
1.14
3.03
0.70
1.07
1.09
1.05
1.84
3.45 3.40
3.00 2.95 2.90
2.50 2.45
2.35 2.30 2.25 2.20 2.15 2.10
1.95 1.90 1.85
1.75 1.70
1.60 1.55 1.50
f1 (ppm)
O
CN
Cl
6f
1.95
1.91
.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.
f1 (ppm)

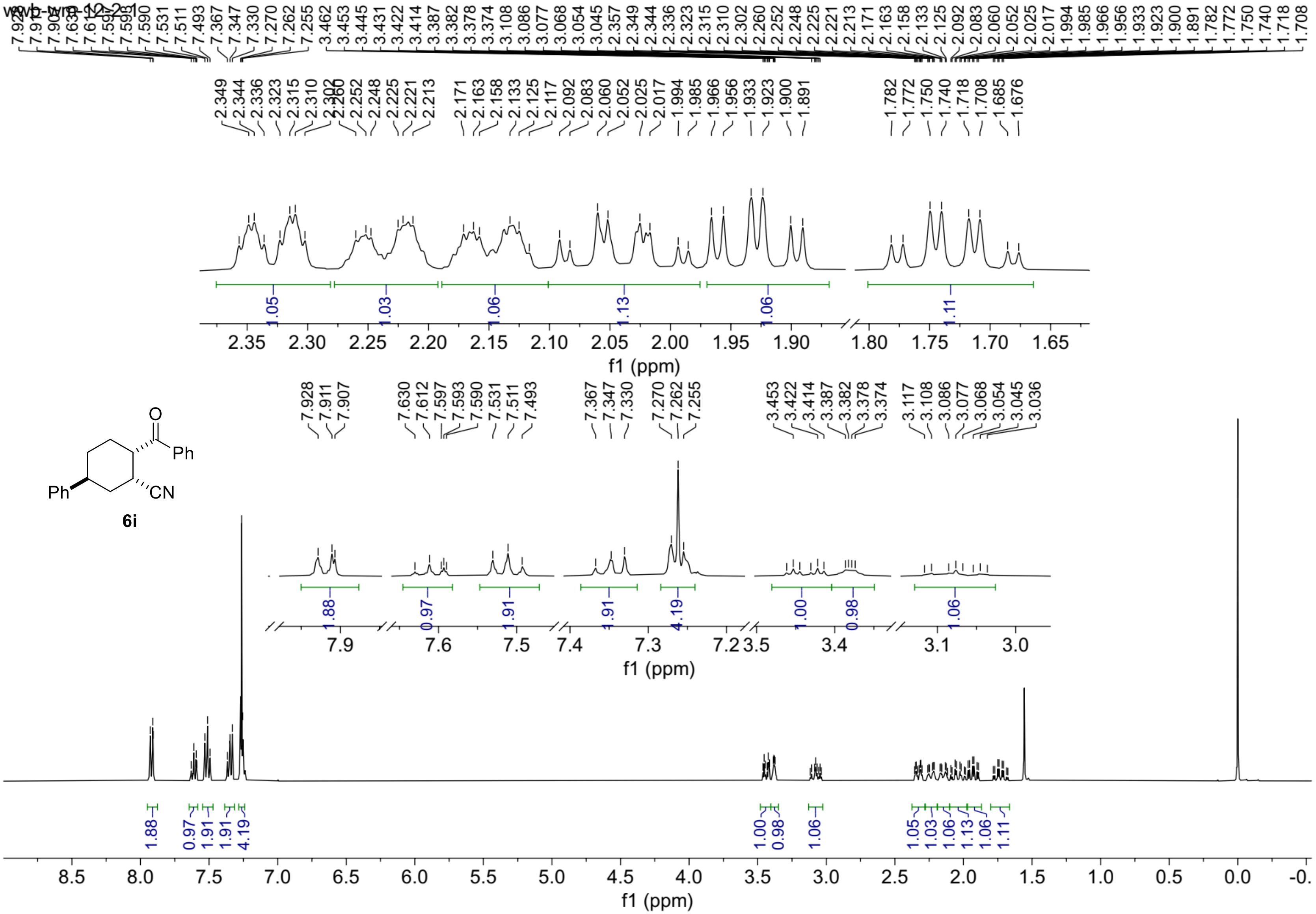

wwb-wm-19-2
206.5
142.86
132.63
128.97
128.28
119.95
77.48
77.16
76.84
51.68
39.01
35.74
33.20
29.32
27.76
25.27
O
CN
Cl
6f
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

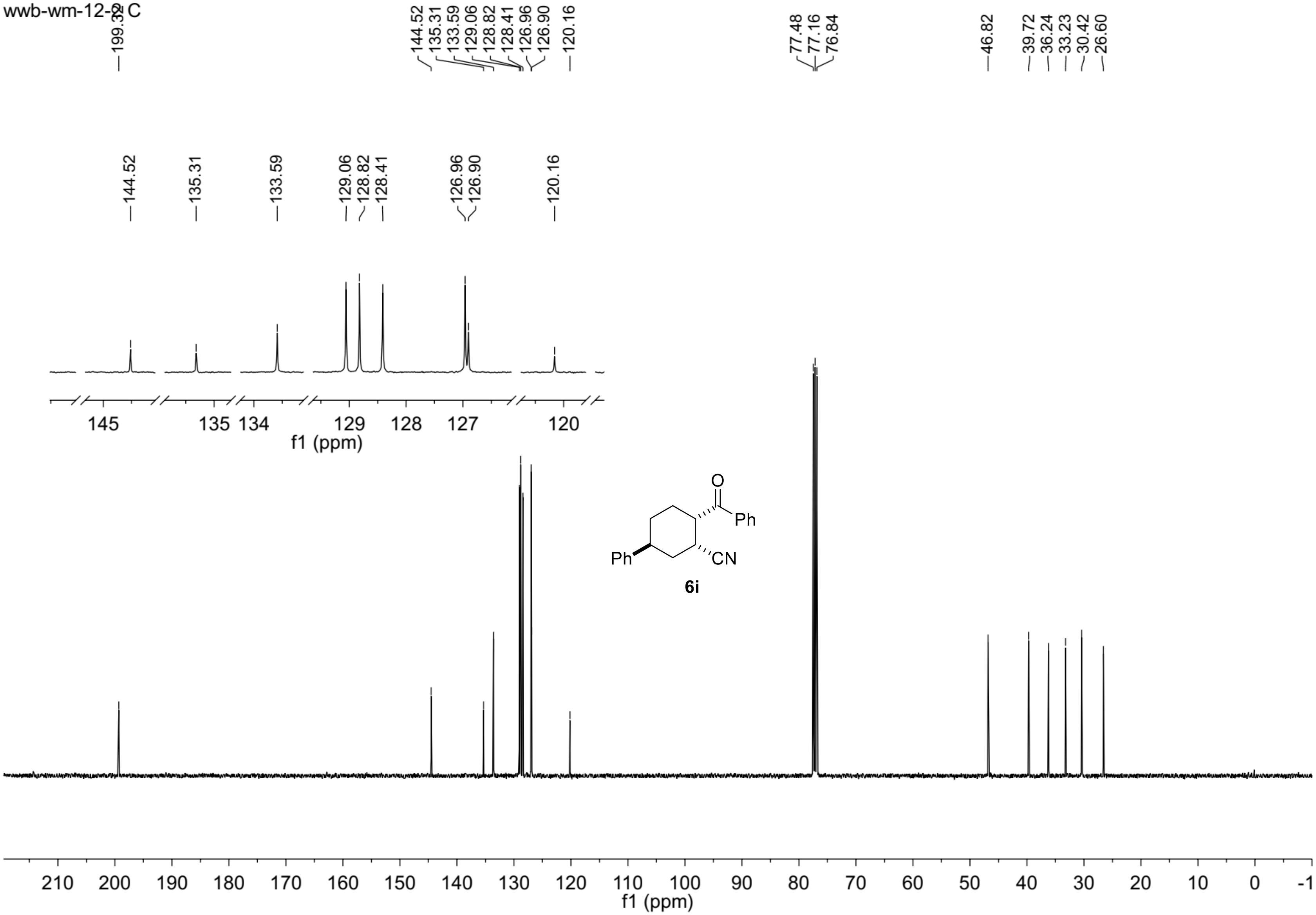

wwb-wn-107-2
3.431 3.425 3.417 3.408
2.976 2.968 2.959 2.945 2.936 2.927 2.914 2.905 2.896
2.501 2.492 2.483 2.469 2.460 2.451
2.336 2.327 2.318 2.297 2.284 2.253 2.225 2.217 2.211 2.203
2.129 2.121 2.115 2.096 2.091 2.082
1.957 1.948 1.925 1.916 1.891 1.882 1.858 1.849
1.747 1.737 1.715 1.704 1.682 1.671
1.571 1.562 1.539 1.530 1.507 1.497
1.00
1.04
1.02
1.19
2.84
0.97
1.06
1.09
1.07
1.23
f1 (ppm)
7.455 7.434
7.098 7.076
1.97
2.00
f1 (ppm)
O
CN
Br
6g
f1 (ppm)

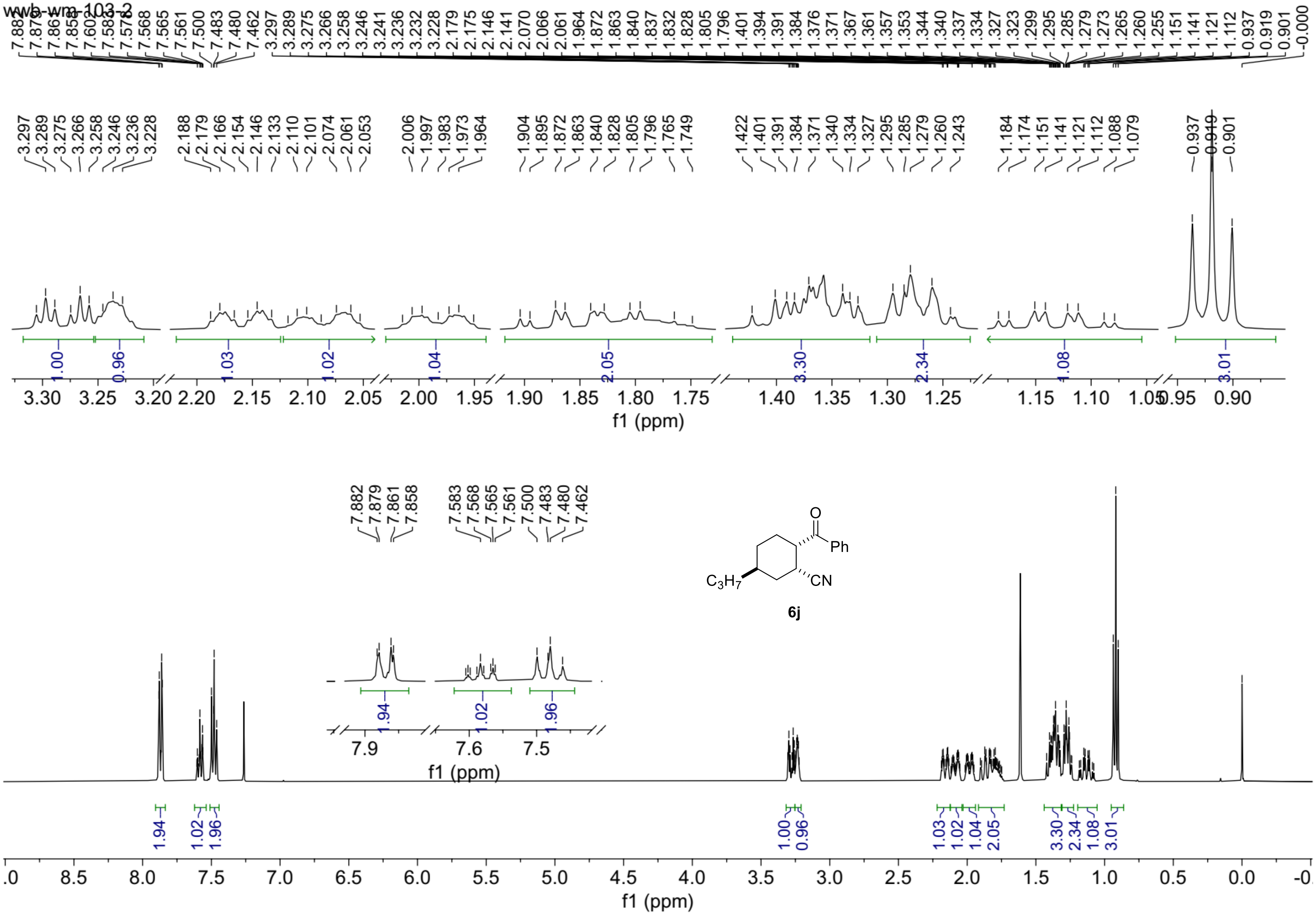

wwb-wm-107-2 C
206.4
143.39
131.93
128.67
120.66
119.91
77.48
77.16
76.84
51.69
39.09
35.70
33.13
29.33
27.74
25.25
O
CN
Br
6g
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

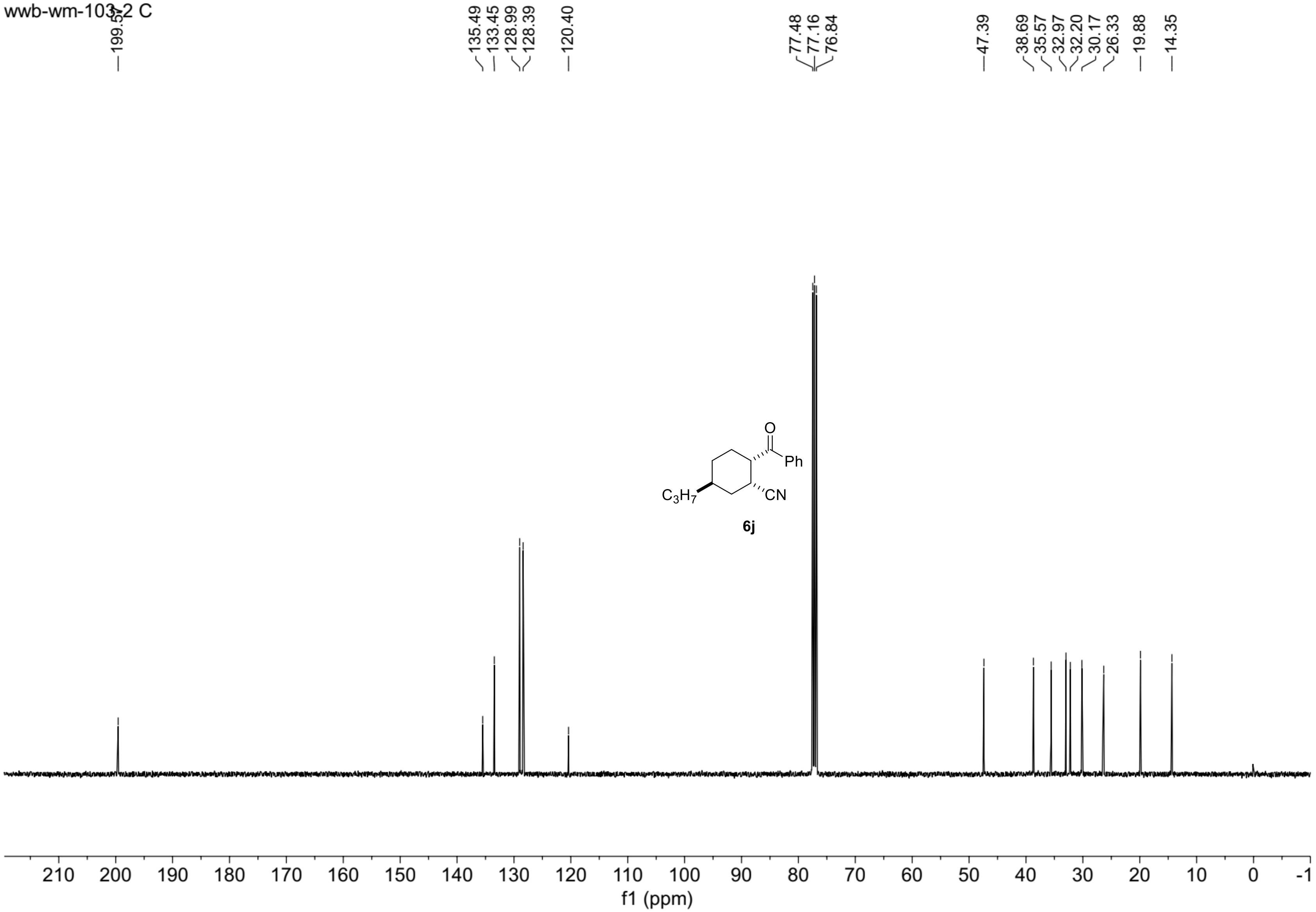

wwb-wm-1432
7.327 7.325 7.322 7.320 6.304 6.299 6.296 6.291 6.028 6.026 6.024 6.020 6.018 6.016 3.404 3.398 3.388 3.381 3.102 3.093 3.080 3.071 3.062 3.049 3.040 2.485 2.476 2.467 2.453 2.444 2.435 2.426 2.421 2.413 2.400 2.392 2.387 2.379 2.316 2.304 2.292 2.267 2.263 2.245 2.235 2.224 2.215 1.921 1.913 1.887 1.880 1.855 1.848 1.823 1.812 1.757 1.747 1.726 1.723 1.716 1.713 1.692 1.682 1.616 1.604 1.580 1.573 1.550 1.538 1.517 1.508 0.000
7.327 7.325 7.322 7.320
6.304 6.299 6.296 6.291
6.026 6.024 6.020 6.018 6.016
3.404 3.398 3.388 3.381
3.111 3.102 3.093 3.080 3.071 3.062 3.049 3.040
1.01
1.07
1.05
1.00
1.07
7.40 7.30 6.35 6.25 6.05 6.00 3.45 3.35 3.10 3.05 3.00
f1 (ppm)
2.485 2.476 2.467 2.453 2.444 2.435 2.426 2.421 2.400 2.392 2.387 2.379 2.304 2.292 2.267 2.263 2.245 2.235 2.224
1.921 1.913 1.887 1.880 1.855 1.848 1.823 1.812
1.757 1.747 1.726 1.723 1.716 1.713 1.692 1.682
1.616 1.604 1.580 1.573 1.550 1.538 1.517 1.508
1.08
1.11
1.38
4.01
1.12
1.14
1.15
2.50 2.45 2.40 2.35 2.30 2.25 2.20 1.90 1.85 1.80 1.75 1.70 1.65 1.60 1.55 1.50
f1 (ppm)
O
O
CN
6h
1.01 1.07 1.05 1.00 1.07 1.08 1.11 1.38 4.01 1.12 1.14 1.15
.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.
f1 (ppm)

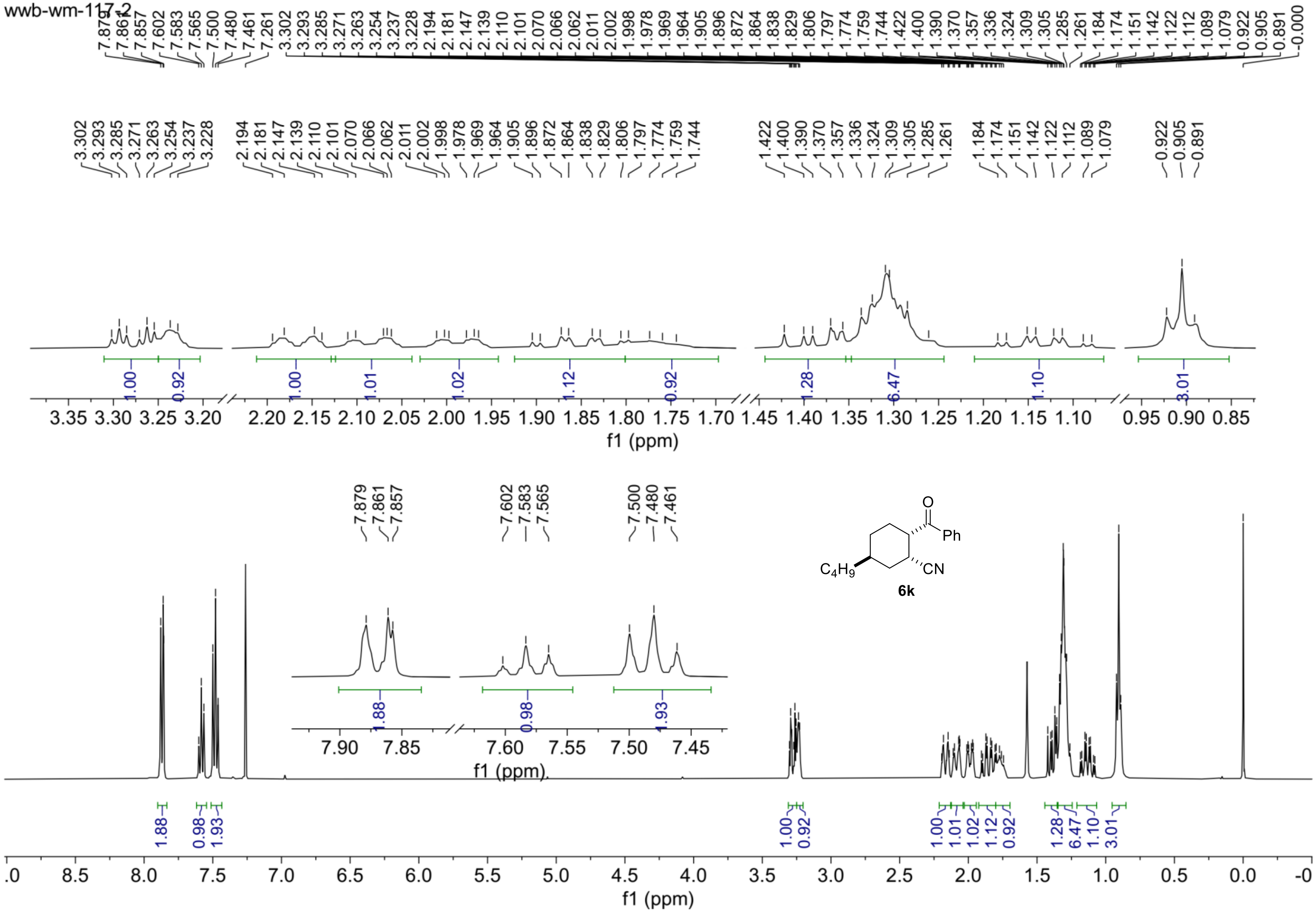

wwb-wm-14-2
206.61
157.47
141.40
119.83
110.14
104.12
77.48
77.16
76.84
51.61
33.16
32.94
30.44
28.70
27.74
24.63
O
O
CN
6h
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

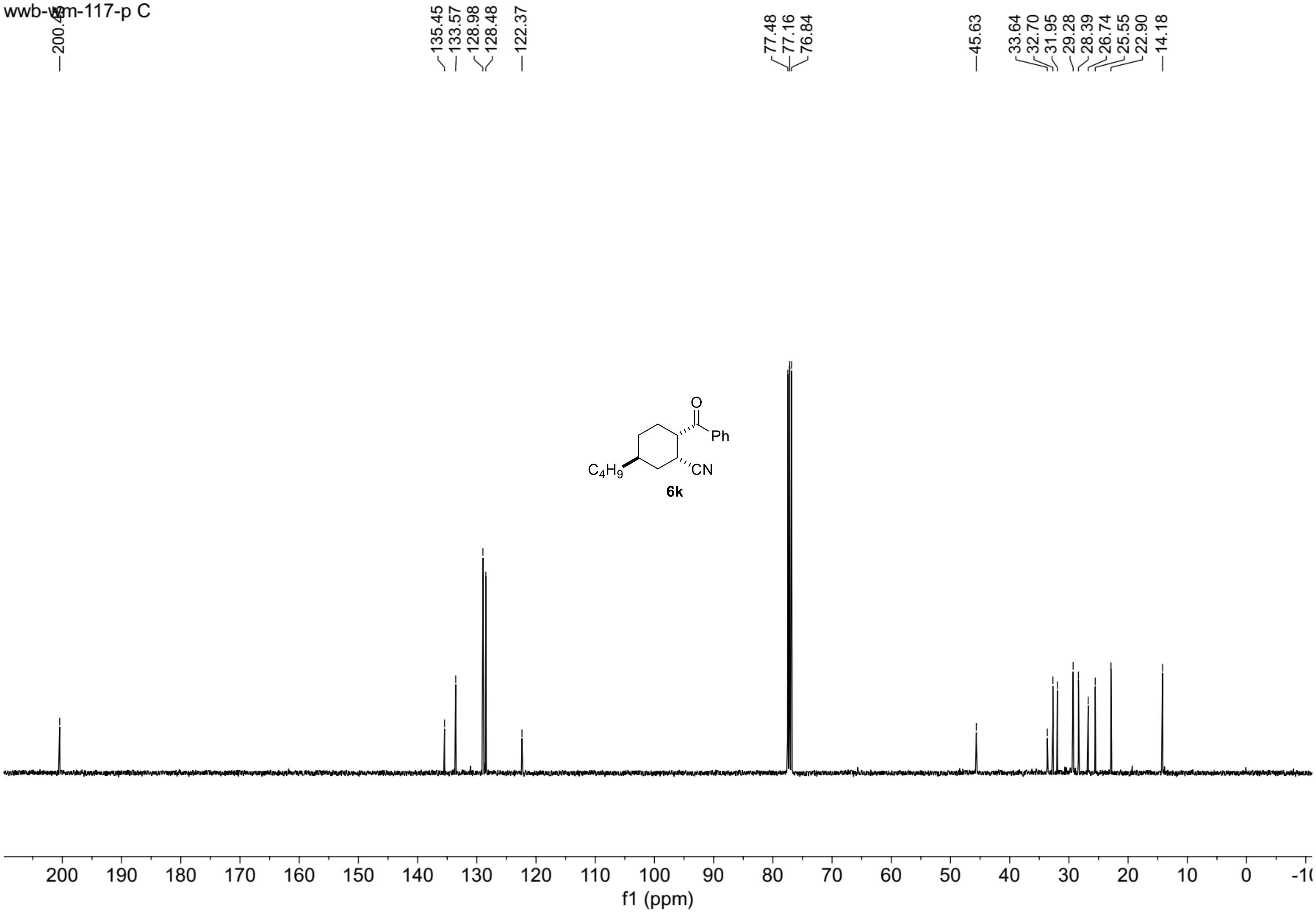

wwb-wm-12-2-1
7.928 7.911 7.907 7.630 7.612 7.597 7.593 7.590 7.531 7.511 7.493 7.367 7.347 7.330 7.270 7.262 7.255
3.462 3.453 3.445 3.431 3.422 3.414 3.387 3.382 3.378 3.374 3.108 3.086 3.077 3.068 3.054 3.045
2.357 2.349 2.344 2.336 2.323 2.315 2.310 2.302 2.260 2.252 2.248 2.225 2.221 2.213 2.171 2.163 2.158 2.133 2.125 2.092 2.083 2.060 2.052 2.025 2.017 1.994 1.985 1.966 1.956 1.933 1.923 1.900 1.891 1.782 1.772 1.750 1.740 1.718 1.708 1.685 1.676
3.117 3.036
O
Ph
Ph
CN
6i
1.05 1.03 1.06 1.13 1.06 1.11
1.88 0.97 1.91 1.91 4.19 1.00 0.98 1.06
f1 (ppm)

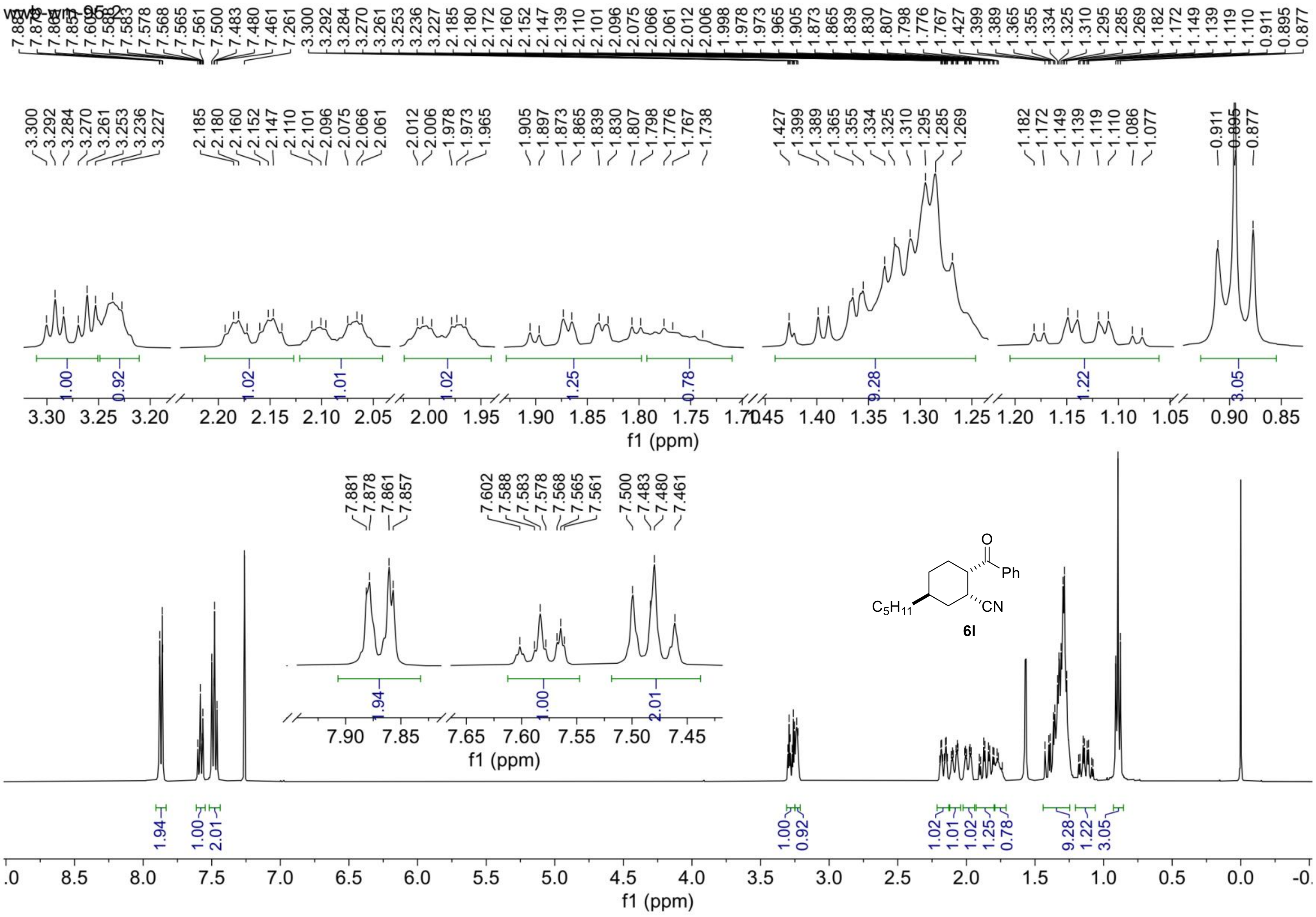

wwb-wm-12-9 C
199.32
144.52
135.31
133.59
129.06
128.82
128.41
126.96
126.90
120.16
77.48
77.16
76.84
46.82
39.72
36.24
33.23
30.42
26.60
144.52
135.31
133.59
129.06
128.82
128.41
126.96
126.90
120.16
145
135
134
129
128
127
120
f1 (ppm)
O
Ph
Ph
CN
6i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

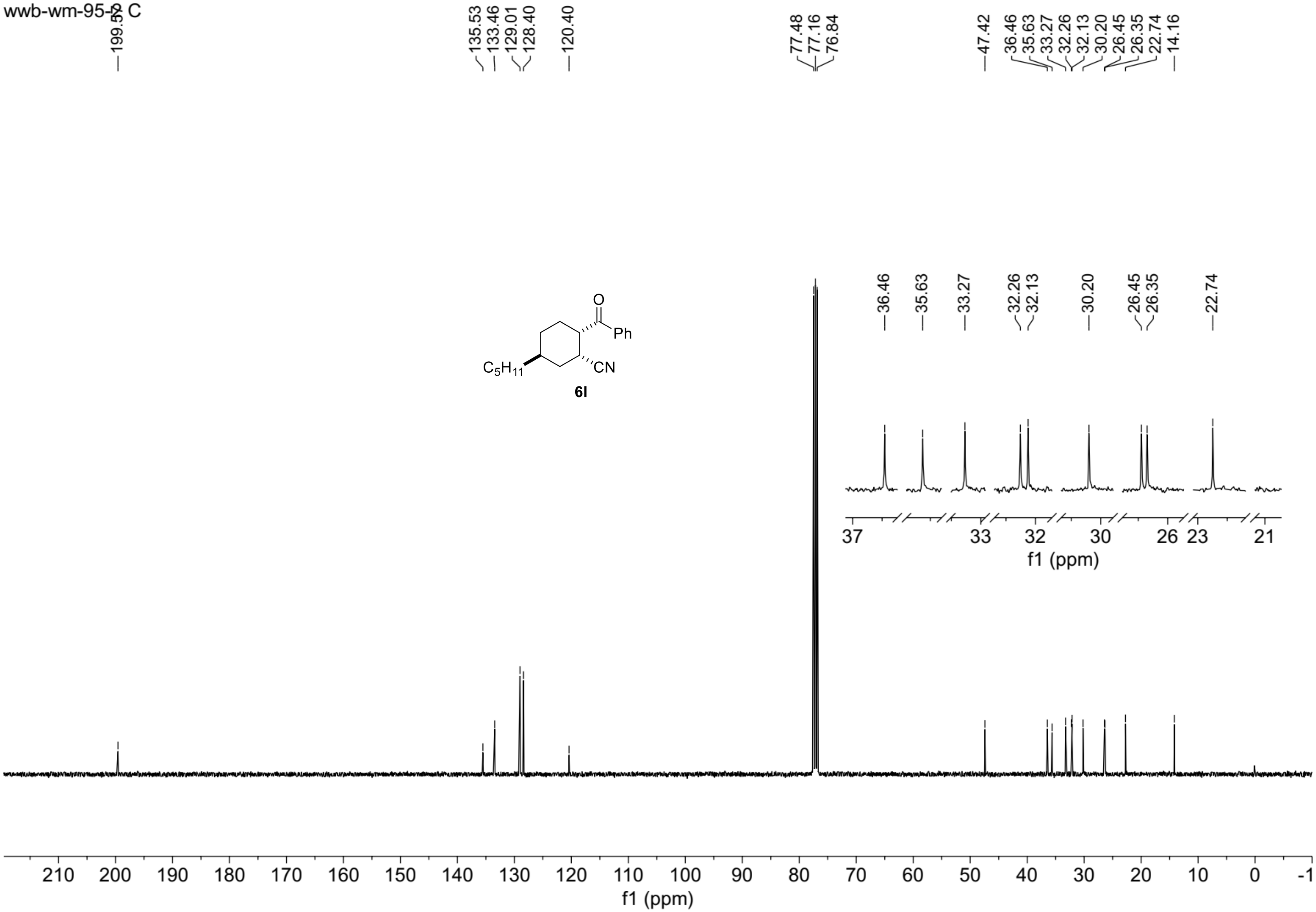

wwb-wm-103-2
7.882 7.879 7.861 7.858 7.583 7.568 7.565 7.561 7.500 7.483 7.480 7.462 3.297 3.289 3.275 3.266 3.258 3.246 3.241 3.236 3.232 3.228 2.179 2.175 2.146 2.141 2.070 2.066 2.061 1.964 1.872 1.863 1.840 1.837 1.832 1.828 1.805 1.796 1.401 1.394 1.391 1.384 1.376 1.371 1.367 1.361 1.357 1.353 1.344 1.340 1.337 1.334 1.327 1.323 1.299 1.295 1.285 1.279 1.273 1.265 1.260 1.255 1.151 1.141 1.121 1.112 0.937 0.919 0.901 -0.000
3.297 3.289 3.275 3.266 3.258 3.246 3.236 3.228
2.188 2.179 2.166 2.154 2.146 2.133 2.110 2.101 2.074 2.061 2.053
2.006 1.997 1.983 1.973 1.964
1.904 1.895 1.872 1.863 1.840 1.828 1.805 1.796 1.765 1.749
1.422 1.401 1.391 1.384 1.371 1.340 1.334 1.327 1.295 1.285 1.279 1.260 1.243
1.184 1.174 1.151 1.141 1.121 1.112 1.088 1.079
0.937 0.919 0.901
1.00 0.96 1.03 1.02 1.04 2.05 3.30 2.34 1.08 3.01
3.30 3.25 3.20 2.20 2.15 2.10 2.05 2.00 1.95 1.90 1.85 1.80 1.75 1.40 1.35 1.30 1.25 1.15 1.10 1.05 0.95 0.90
f1 (ppm)
O
Ph
C3H7
CN
6j
1.94 1.02 1.96
7.9 7.6 7.5
f1 (ppm)
1.94 1.02 1.96 1.00 0.96 1.03 1.02 1.04 2.05 3.30 2.34 1.08 3.01
.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.
f1 (ppm)

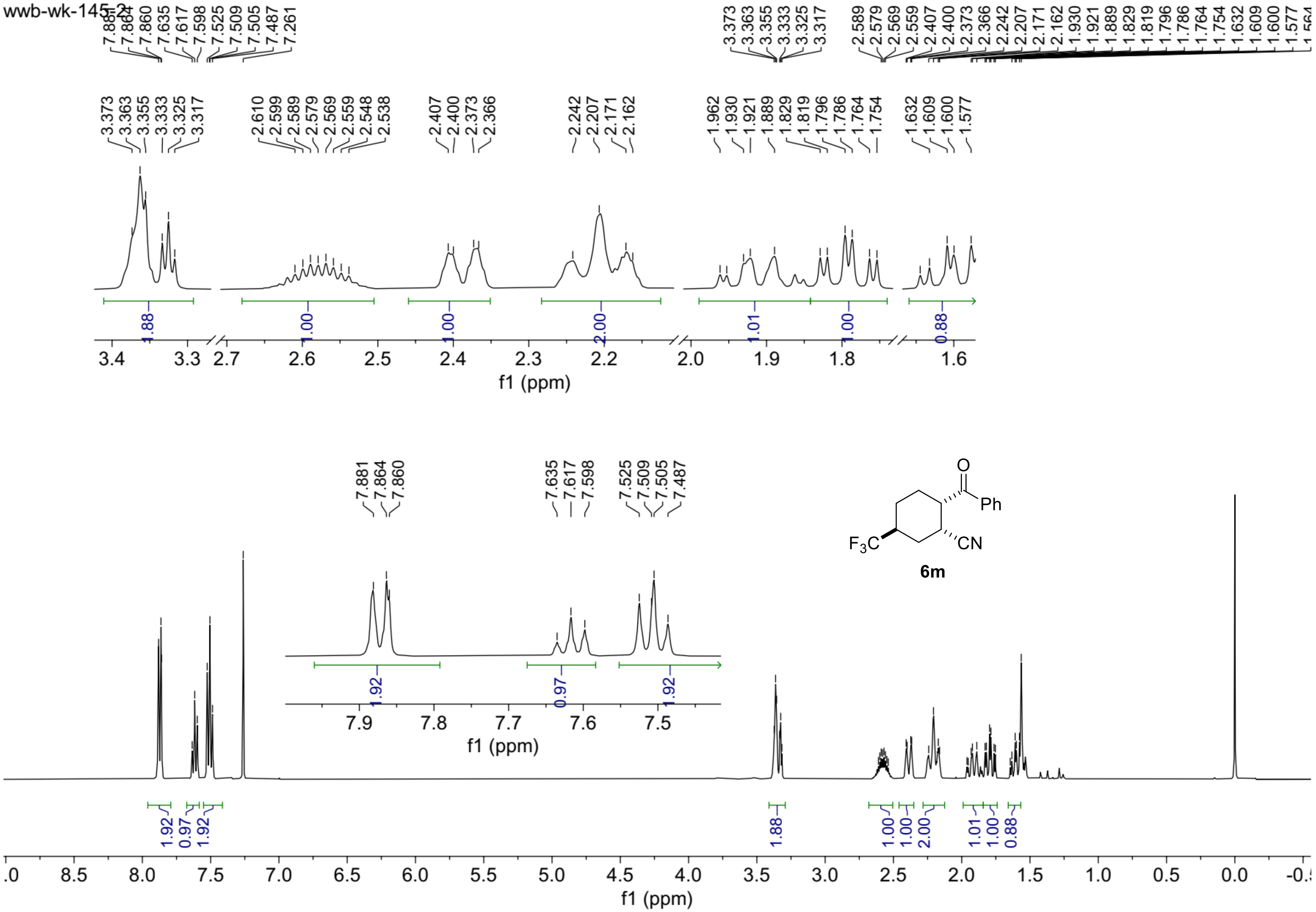

wwb-wm-103-2 C
199.52
135.49
133.45
128.99
128.39
120.40
77.48
77.16
76.84
47.39
38.69
35.57
32.97
32.20
30.17
26.33
19.88
14.35
O
Ph
C$_3$H$_7$
CN
6j
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)


wwb-wm-117-2
7.879
7.861
7.857
7.602
7.583
7.565
7.500
7.480
7.461
7.261
3.302
3.293
3.285
3.271
3.263
3.254
3.237
3.228
2.194
2.181
2.147
2.139
2.110
2.101
2.070
2.066
2.062
2.011
2.002
1.998
1.978
1.969
1.964
1.905
1.896
1.872
1.864
1.838
1.829
1.806
1.797
1.774
1.759
1.744
1.422
1.400
1.390
1.370
1.357
1.336
1.324
1.309
1.305
1.285
1.261
1.184
1.174
1.151
1.142
1.122
1.112
1.089
1.079
0.922
0.905
0.891
-0.000
1.00
0.92
1.00
1.01
1.02
1.12
0.92
1.28
6.47
1.10
3.01
1.88
0.98
1.93
f1 (ppm)
O
Ph
CN
C_4H_9
6k

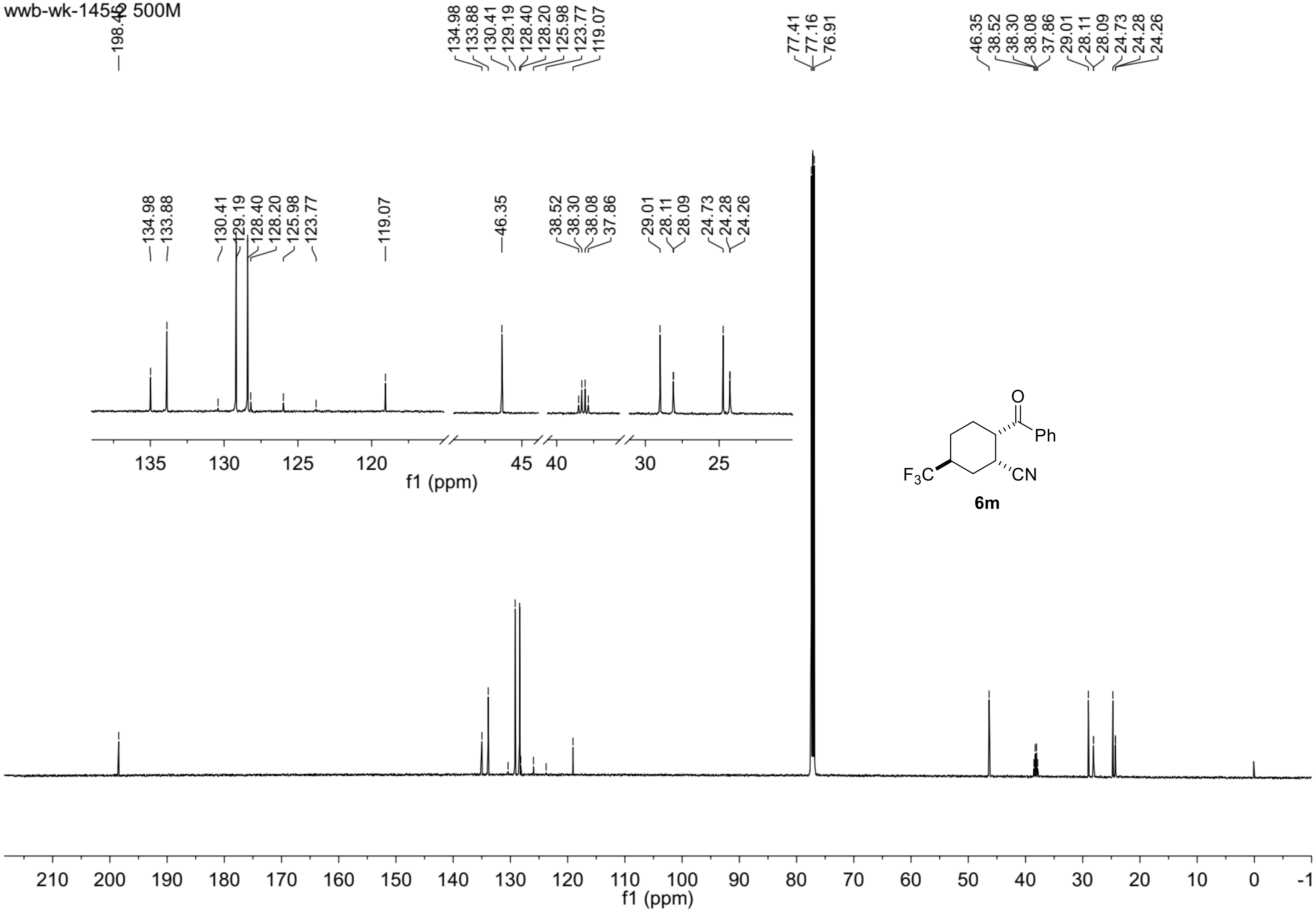

wwb-wm-117-p C
200.42
135.45
133.57
128.98
128.48
122.37
77.48
77.16
76.84
45.63
33.64
32.70
31.95
29.28
28.39
26.74
25.55
22.90
14.18
O
Ph
C_4H_9
CN
6k
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1(
f1 (ppm)


wwb-wm-95-2
O
Ph
CN
C_5H_{11}
6l
3.300 3.292 3.284 3.270 3.261 3.253 3.236 3.227
2.185 2.180 2.160 2.152 2.147 2.110 2.101 2.096 2.075 2.066 2.061
2.012 2.006 1.978 1.973 1.965
1.905 1.897 1.873 1.865 1.839 1.830 1.807 1.798 1.776 1.767 1.738
1.427 1.399 1.389 1.365 1.355 1.334 1.325 1.310 1.295 1.285 1.269
1.182 1.172 1.149 1.139 1.119 1.110 1.086 1.077
0.911 0.895 0.877
7.881 7.878 7.861 7.857
7.602 7.588 7.583 7.578 7.568 7.565 7.561
7.500 7.483 7.480 7.461
7.261
1.00 0.92 1.02 1.01 1.02 1.25 0.78 9.28 1.22 3.05
1.94 1.00 2.01
f1 (ppm)

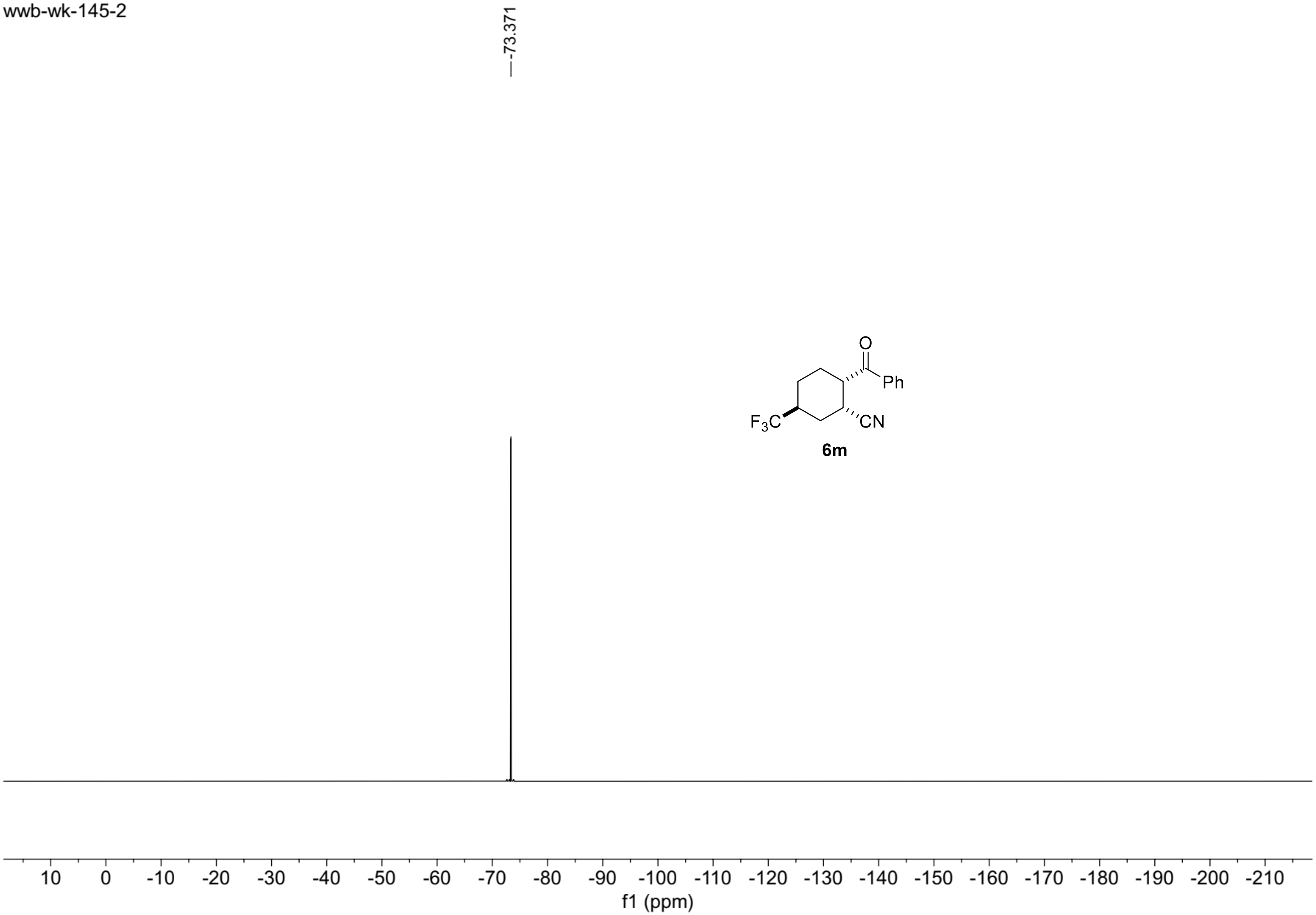

wwb-wm-95-2 C
199.52
135.53
133.46
129.01
128.40
120.40
77.48
77.16
76.84
47.42
36.46
35.63
33.27
32.26
32.13
30.20
26.45
26.35
22.74
14.16
O
Ph
C_5H_{11}
CN
6l
36.46
35.63
33.27
32.26
32.13
30.20
26.45
26.35
22.74
37
33
32
30
26
23
21
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

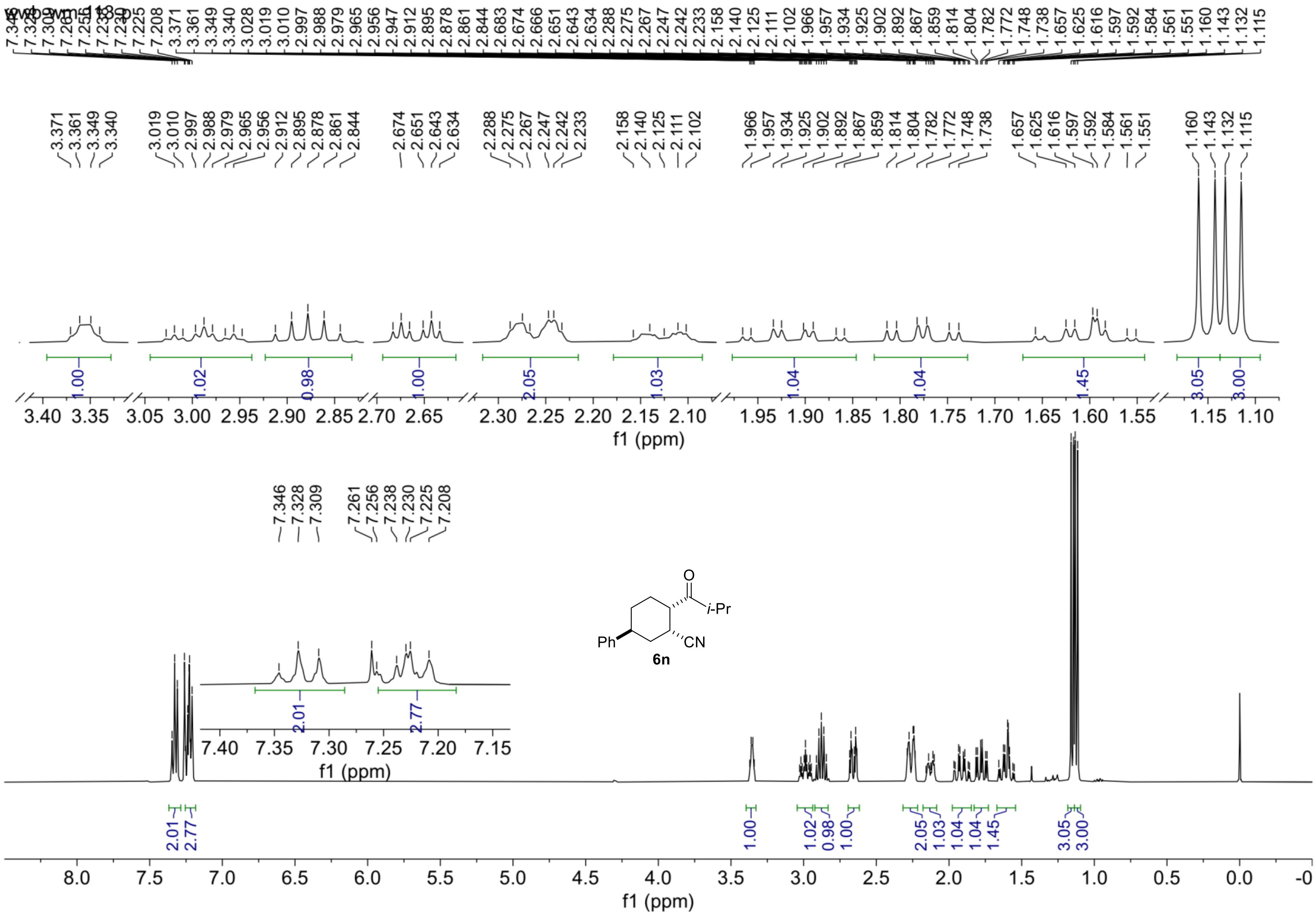


wwb-wk-145-2
7.881
7.864
7.860
7.635
7.617
7.598
7.525
7.509
7.505
7.487
7.261
3.373
3.363
3.355
3.333
3.325
3.317
2.610
2.599
2.589
2.579
2.569
2.559
2.548
2.538
2.407
2.400
2.373
2.366
2.242
2.207
2.171
2.162
1.962
1.930
1.921
1.889
1.829
1.819
1.796
1.786
1.764
1.754
1.632
1.609
1.600
1.577
1.88
1.00
1.00
2.00
1.01
1.00
0.88
3.4
3.3
2.7
2.6
2.5
2.4
2.3
2.2
2.0
1.9
1.8
1.6
f1 (ppm)
1.92
0.97
1.92
7.9
7.8
7.7
7.6
7.5
f1 (ppm)
O
Ph
F3C
CN
6m
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)

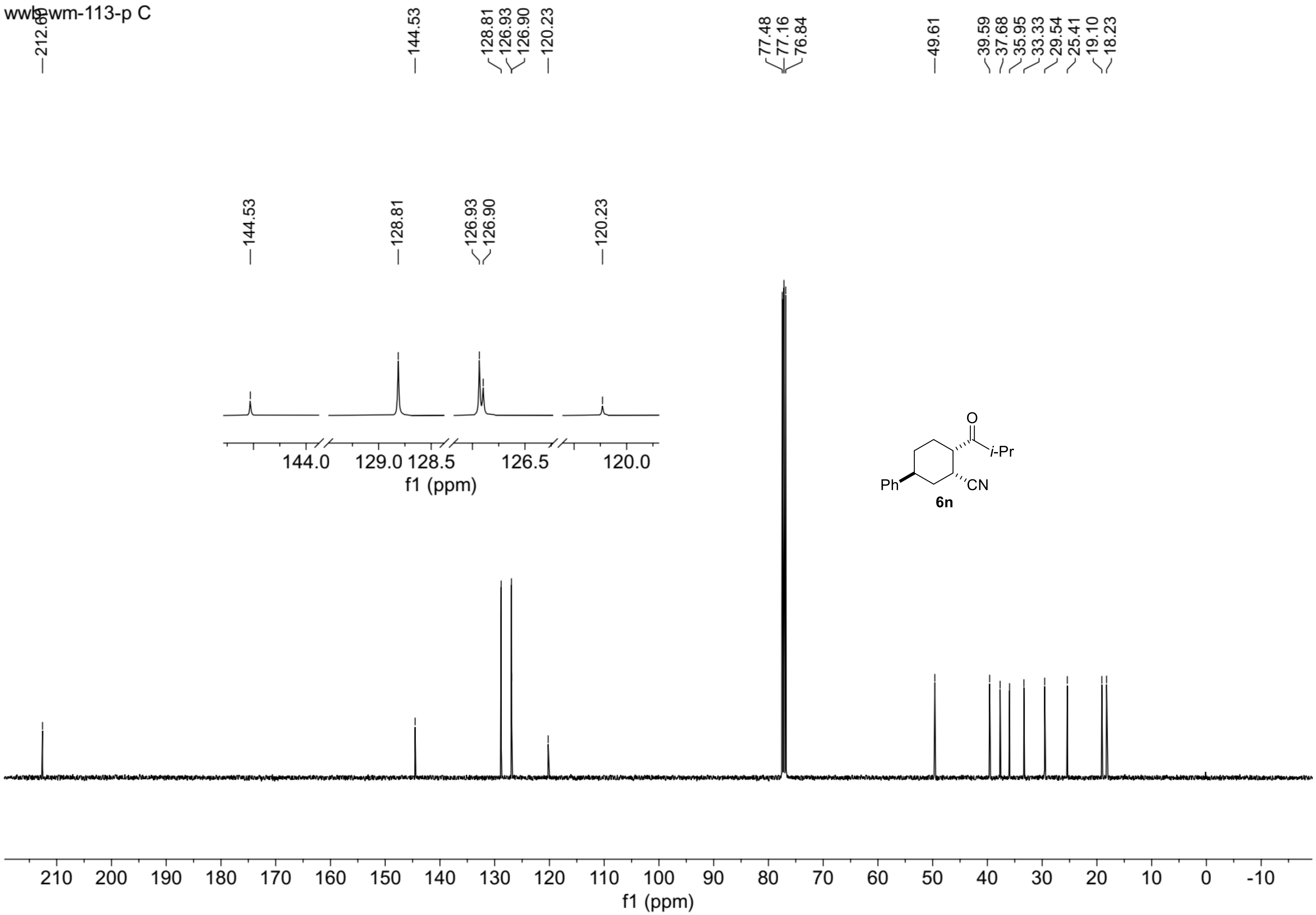

wwb-wk-145-2 500M
198.42
134.98
133.88
130.41
129.19
128.40
128.20
125.98
123.77
119.07
77.41
77.16
76.91
46.35
38.52
38.30
38.08
37.86
29.01
28.11
28.09
24.73
24.28
24.26
135
130
125
120
45
40
30
25
f1 (ppm)
O
Ph
F3C
CN
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

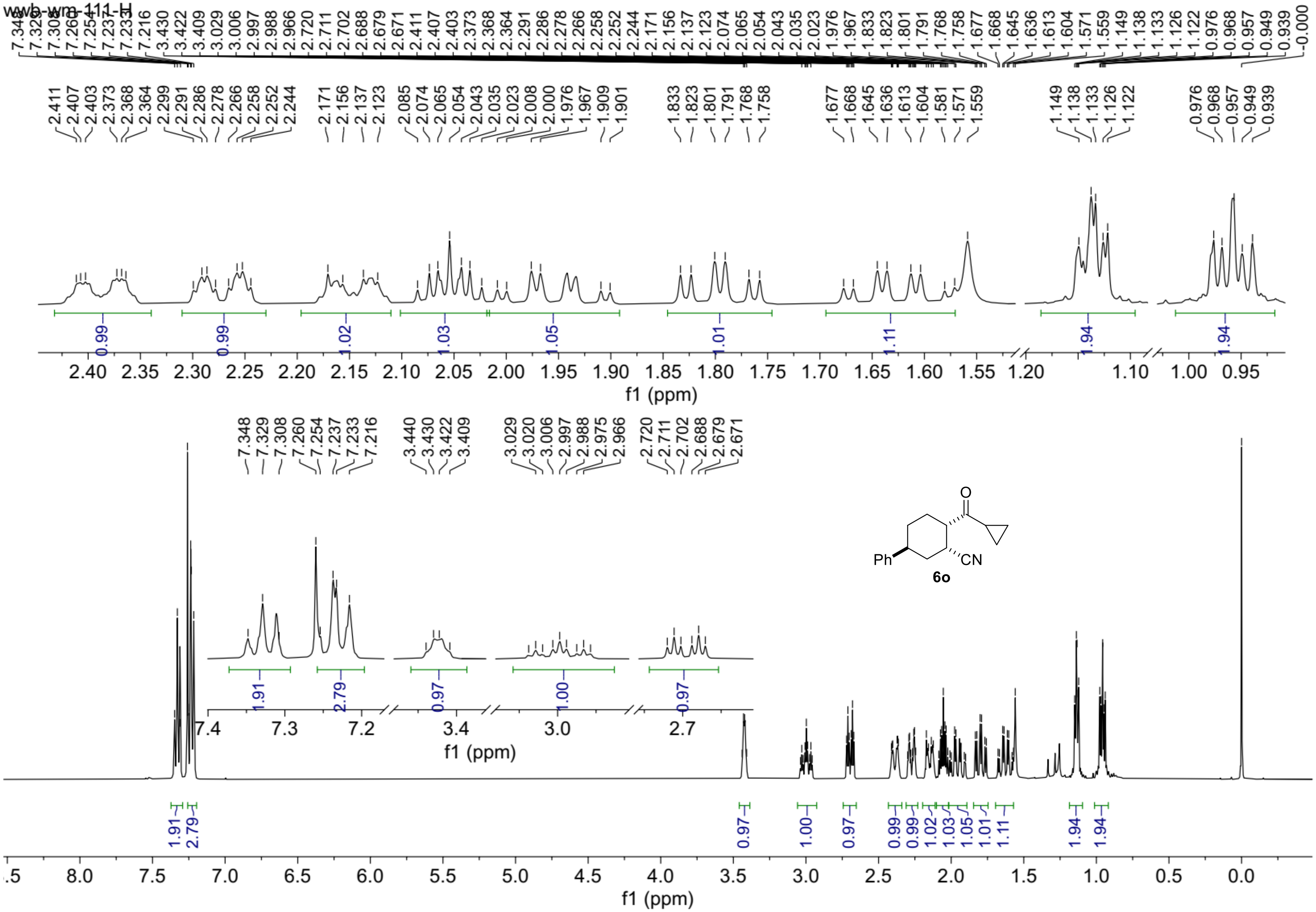
6m

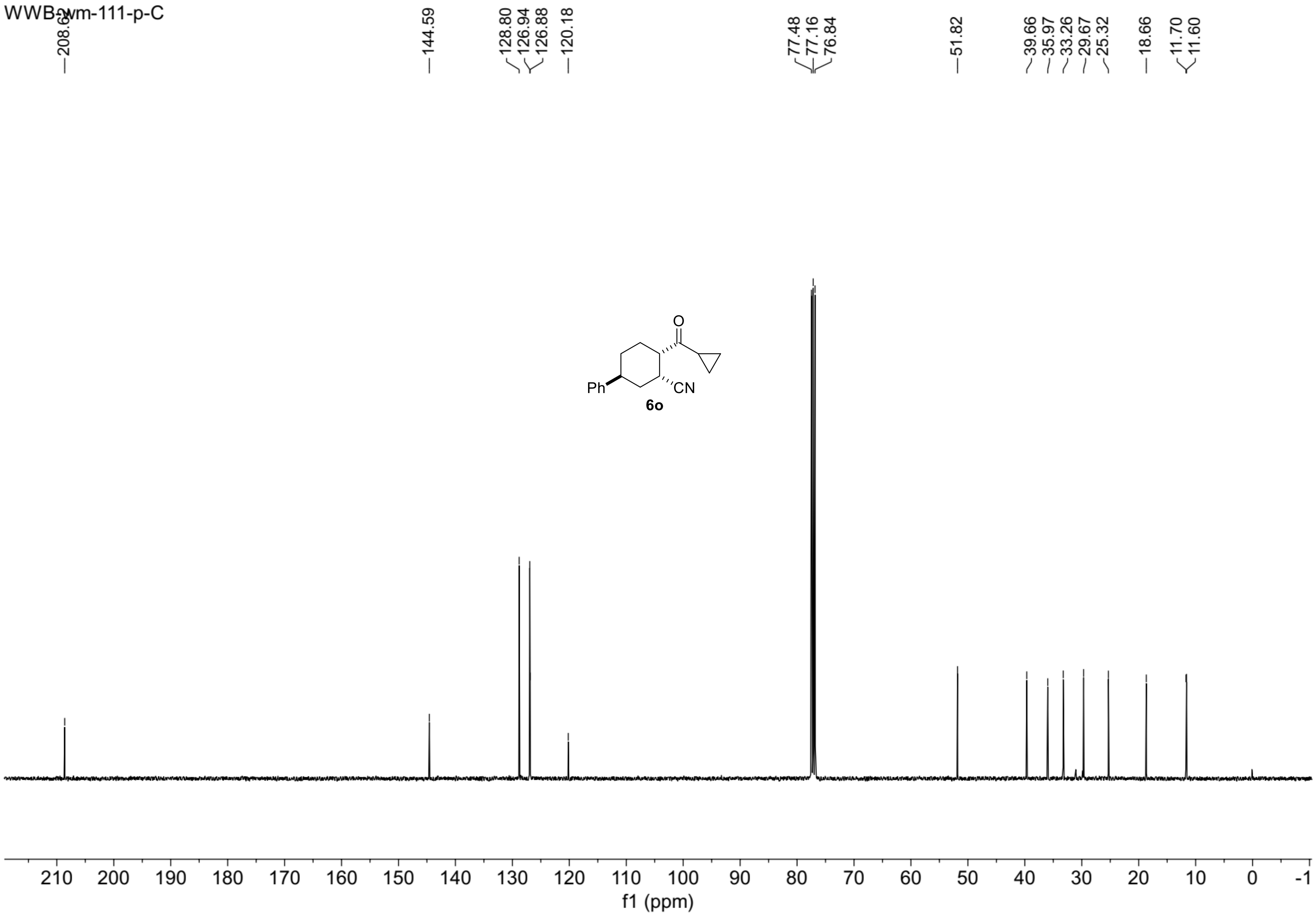

wwb-wk-145-2
-73.371
O
Ph
F3C
CN
6m
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

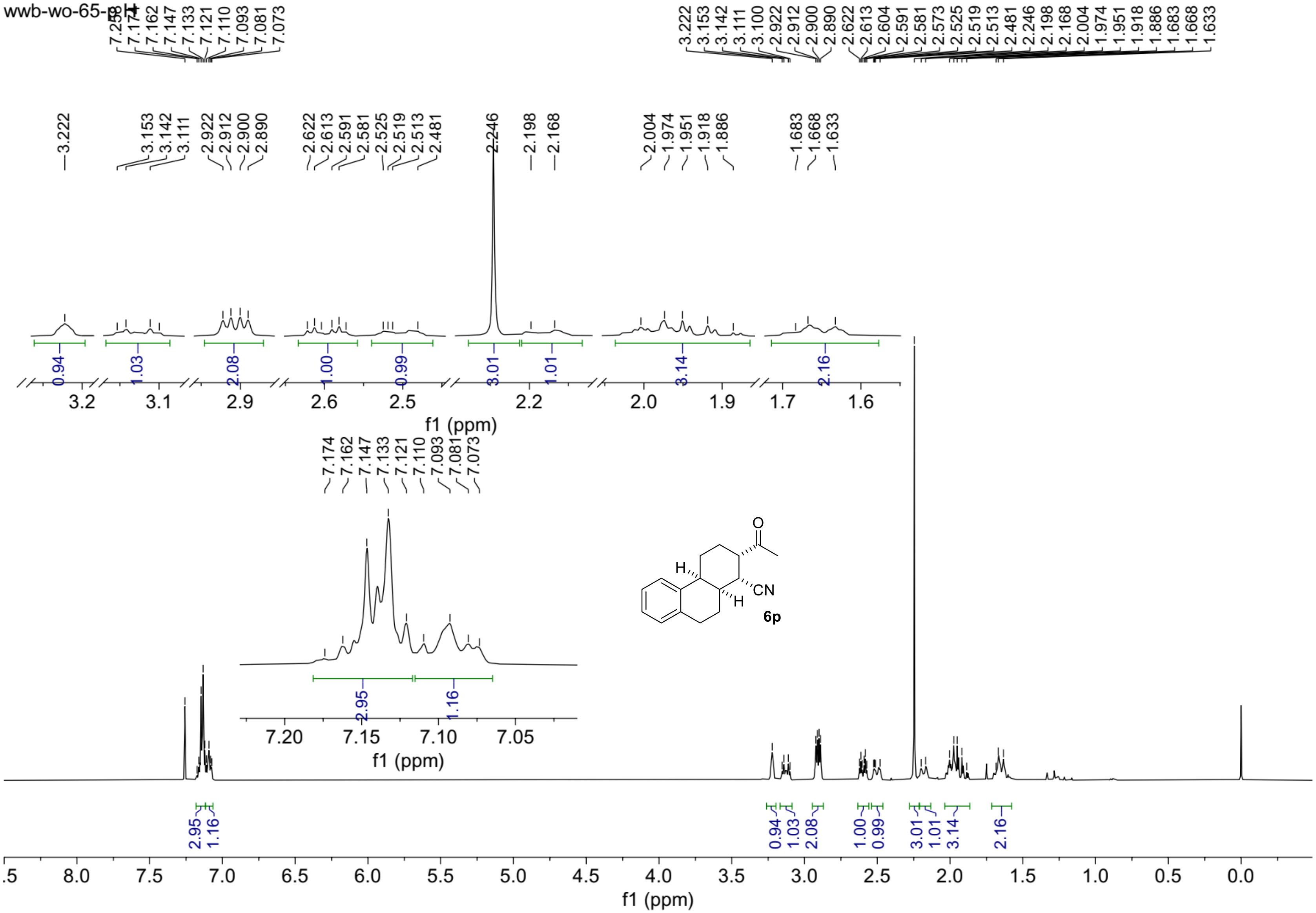


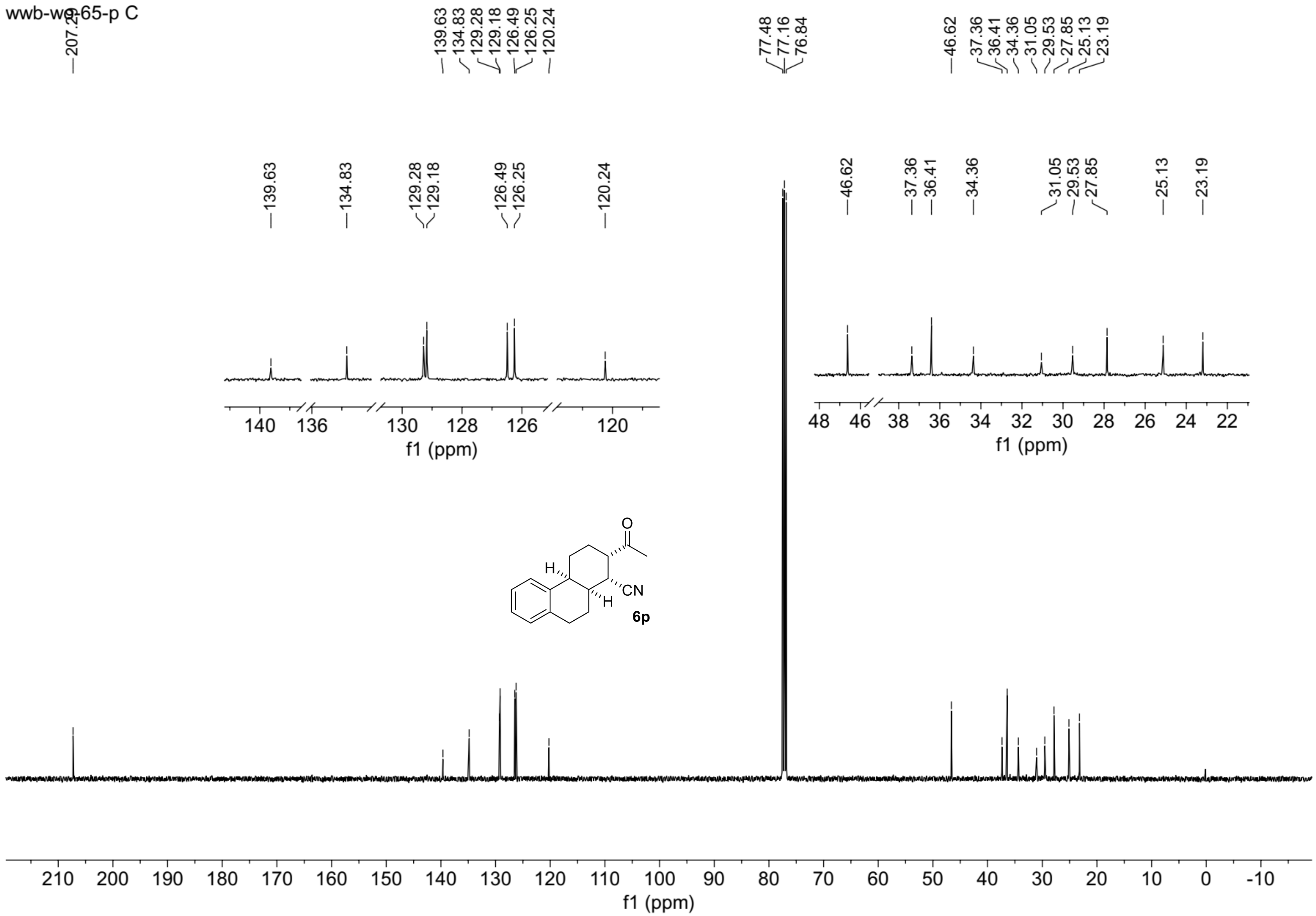
wyb-wm-113
7.346 7.328 7.309 7.261 7.256 7.238 7.230 7.225 7.208
3.371 3.361 3.349 3.340
3.019 3.010 2.997 2.988 2.979 2.965 2.956 2.912 2.895 2.878 2.861 2.844
2.683 2.674 2.666 2.651 2.643 2.634
2.288 2.275 2.267 2.247 2.242 2.233
2.158 2.140 2.125 2.111 2.102
1.966 1.957 1.934 1.925 1.902 1.892 1.867 1.859 1.814 1.804 1.782 1.772 1.748 1.738
1.657 1.625 1.616 1.597 1.592 1.584 1.561 1.551
1.160 1.143 1.132 1.115
1.00 1.02 0.98 1.00 2.05 1.03 1.04 1.04 1.45 3.05 3.00
2.01 2.77
f1 (ppm)
O
i-Pr
Ph
CN
6n

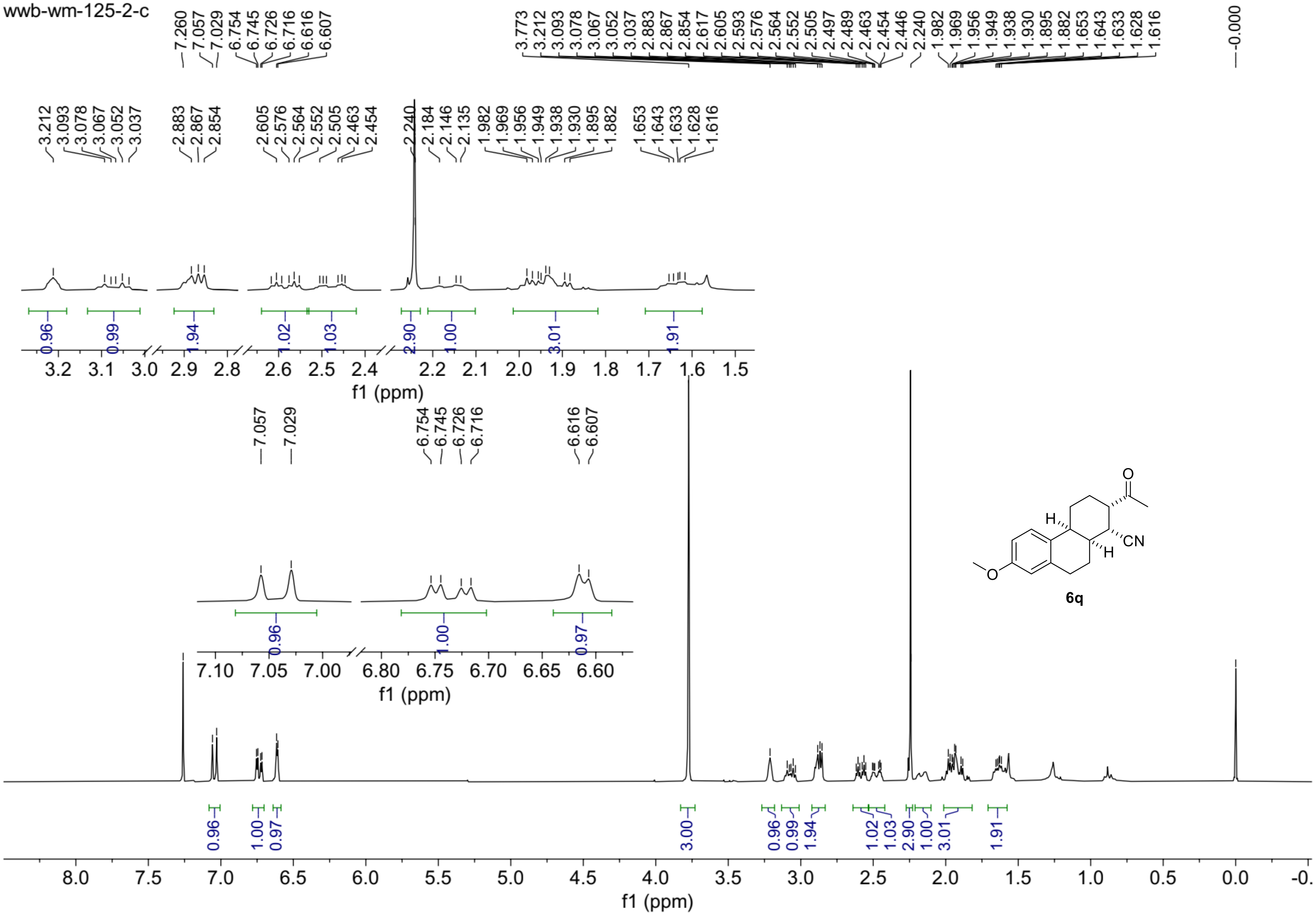

wwb-wm-113-p C
212.6
144.53
128.81
126.93
126.90
120.23
77.48
77.16
76.84
49.61
39.59
37.68
35.95
33.33
29.54
25.41
19.10
18.23
144.53
128.81
126.93
126.90
120.23
144.0
129.0 128.5
126.5
120.0
f1 (ppm)
O
i-Pr
Ph
CN
6n
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


wwb-wm-111-H
7.348 7.329 7.308 7.260 7.254 7.237 7.233 7.216 3.430 3.422 3.409 3.029 3.006 2.997 2.988 2.966 2.720 2.711 2.702 2.688 2.679 2.671 2.411 2.407 2.403 2.373 2.368 2.364 2.291 2.286 2.278 2.266 2.258 2.252 2.244 2.171 2.156 2.137 2.123 2.074 2.065 2.054 2.043 2.035 2.023 1.976 1.967 1.833 1.823 1.801 1.791 1.768 1.758 1.677 1.668 1.645 1.636 1.613 1.604 1.571 1.559 1.149 1.138 1.133 1.126 1.122 0.976 0.968 0.957 0.949 0.939 -0.000
2.411 2.407 2.403 2.373 2.368 2.364 2.299 2.291 2.286 2.278 2.266 2.258 2.252 2.244 2.171 2.156 2.137 2.123 2.085 2.074 2.065 2.054 2.043 2.035 2.023 2.008 2.000 1.976 1.967 1.909 1.901 1.833 1.823 1.801 1.791 1.768 1.758 1.677 1.668 1.645 1.636 1.613 1.604 1.581 1.571 1.559 1.149 1.138 1.133 1.126 1.122 0.976 0.968 0.957 0.949 0.939
0.99 0.99 1.02 1.03 1.05 1.01 1.11 1.94 1.94
2.40 2.35 2.30 2.25 2.20 2.15 2.10 2.05 2.00 1.95 1.90 1.85 1.80 1.75 1.70 1.65 1.60 1.55 1.20 1.10 1.00 0.95
f1 (ppm)
7.348 7.329 7.308 7.260 7.254 7.237 7.233 7.216 3.440 3.430 3.422 3.409 3.029 3.020 3.006 2.997 2.988 2.975 2.966 2.720 2.711 2.702 2.688 2.679 2.671
1.91 2.79 0.97 1.00 0.97
7.4 7.3 7.2 3.4 3.0 2.7
f1 (ppm)
O
Ph
CN
6o
1.91 2.79 0.97 1.00 0.97 0.99 0.99 1.02 1.03 1.05 1.01 1.11 1.94 1.94
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)

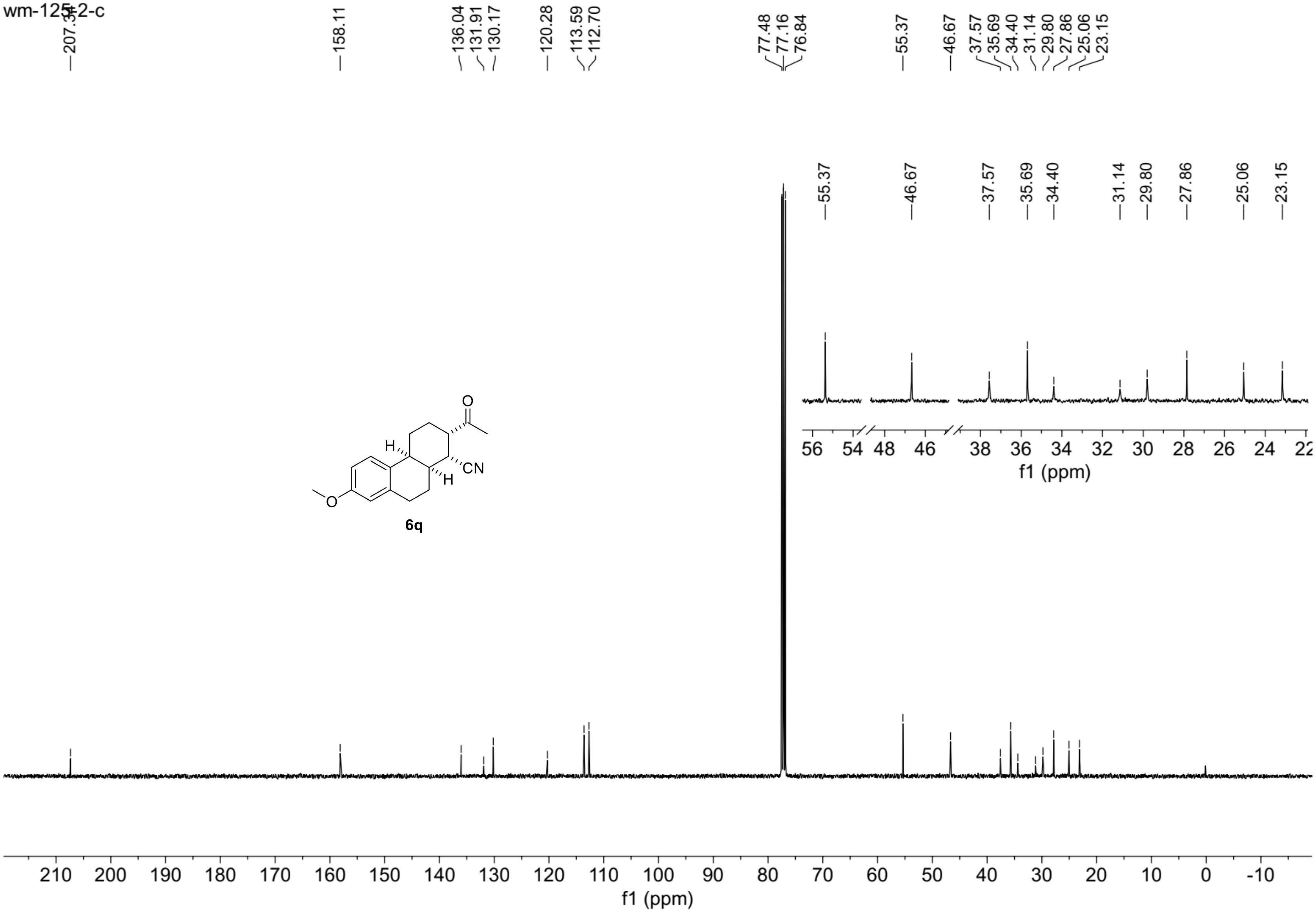

WWB-wm-111-p-C
208.6
144.59
128.80
126.94
126.88
120.18
77.48
77.16
76.84
51.82
39.66
35.97
33.26
29.67
25.32
18.66
11.70
11.60
O
Ph
CN
6o
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)


wwb-wo-65-pH
7.259
7.174
7.162
7.147
7.133
7.121
7.110
7.093
7.081
7.073
3.222
3.153
3.142
3.111
3.100
2.922
2.912
2.900
2.890
2.622
2.613
2.604
2.591
2.581
2.573
2.525
2.519
2.513
2.481
2.246
2.198
2.168
2.004
1.974
1.951
1.918
1.886
1.683
1.668
1.633
0.94
1.03
2.08
1.00
0.99
3.01
1.01
3.14
2.16
3.2
3.1
2.9
2.6
2.5
2.2
2.0
1.9
1.7
1.6
f1 (ppm)
2.95
1.16
7.20
7.15
7.10
7.05
f1 (ppm)
O
H
CN
H
6p
.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)

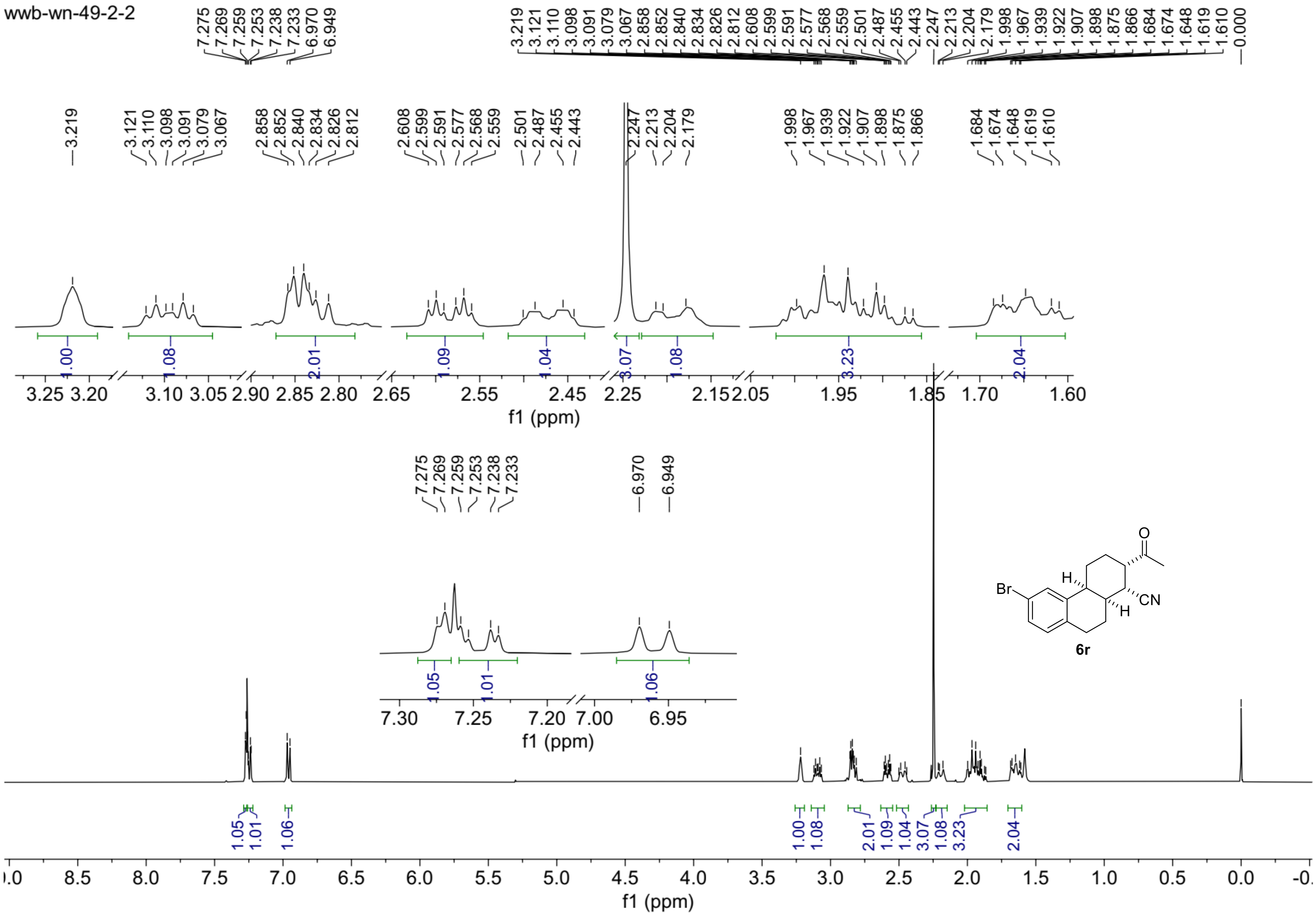

wwb-wq-65-p C
207.2
139.63
134.83
129.28
129.18
126.49
126.25
120.24
77.48
77.16
76.84
46.62
37.36
36.41
34.36
31.05
29.53
27.85
25.13
23.19
140
136
130
128
126
120
f1 (ppm)
48
46
38
36
34
32
30
28
26
24
22
f1 (ppm)
O
H
CN
H
6p
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

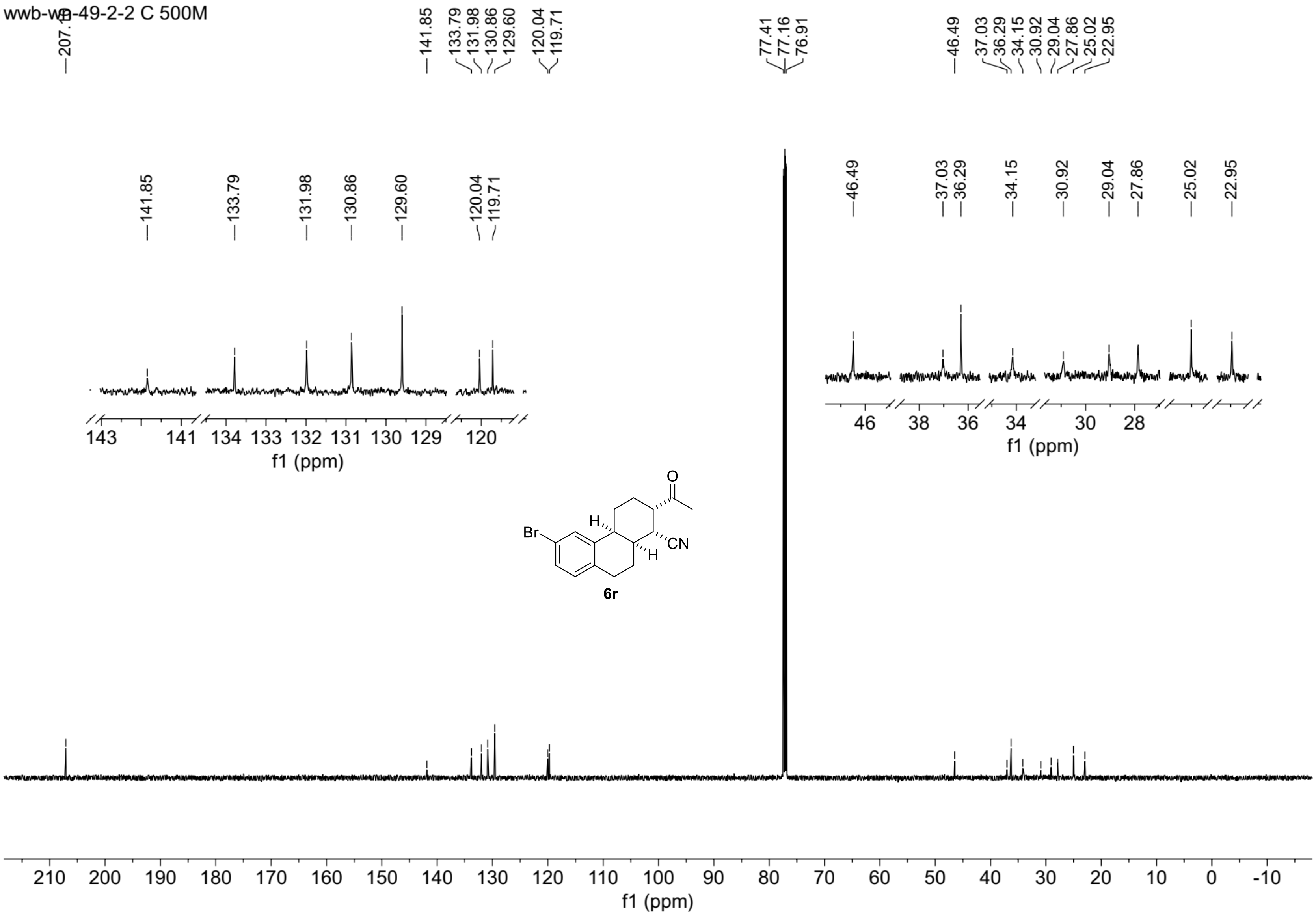

wwb-wm-125-2-c
7.260
7.057
7.029
6.754
6.745
6.726
6.716
6.616
6.607
3.773
3.212
3.093
3.078
3.067
3.052
3.037
2.883
2.867
2.854
2.617
2.605
2.593
2.576
2.564
2.552
2.505
2.497
2.489
2.463
2.454
2.446
2.240
1.982
1.969
1.956
1.949
1.938
1.930
1.895
1.882
1.653
1.643
1.633
1.628
1.616
-0.000
3.212
3.093
3.078
3.067
3.052
3.037
2.883
2.867
2.854
2.605
2.576
2.564
2.552
2.505
2.463
2.454
2.240
2.184
2.146
2.135
1.982
1.969
1.956
1.949
1.938
1.930
1.895
1.882
1.653
1.643
1.633
1.628
1.616
0.96
0.99
1.94
1.02
1.03
2.90
1.00
3.01
1.91
3.2 3.1 3.0 2.9 2.8 2.6 2.5 2.4 2.2 2.1 2.0 1.9 1.8 1.7 1.6 1.5
f1 (ppm)
7.057
7.029
6.754
6.745
6.726
6.716
6.616
6.607
0.96
1.00
0.97
7.10 7.05 7.00 6.80 6.75 6.70 6.65 6.60
f1 (ppm)
O
H
CN
H
O
0.96
1.00
0.97
3.00
0.96
0.99
1.94
1.02
1.03
2.90
1.00
3.01
1.91
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)

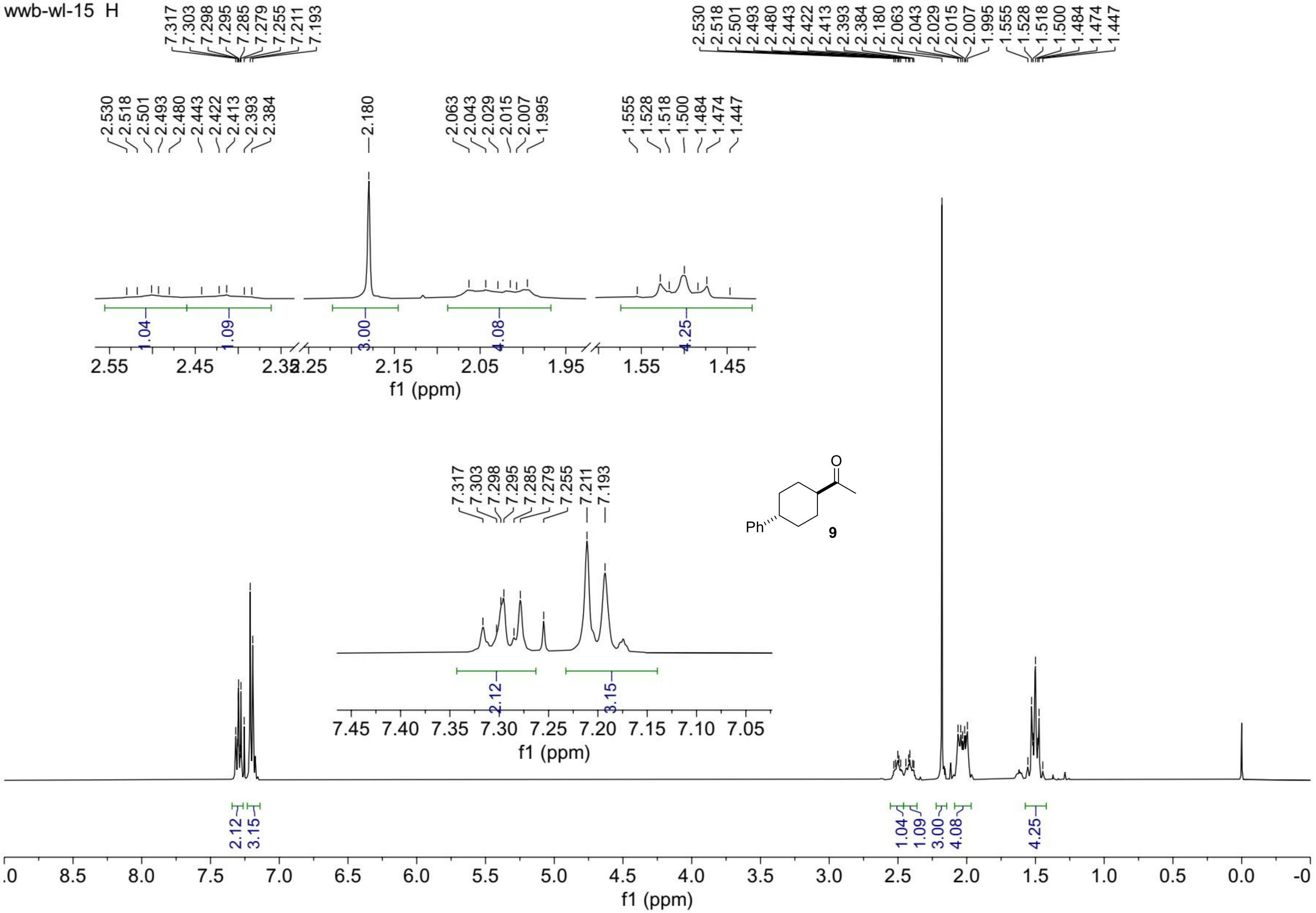
6q

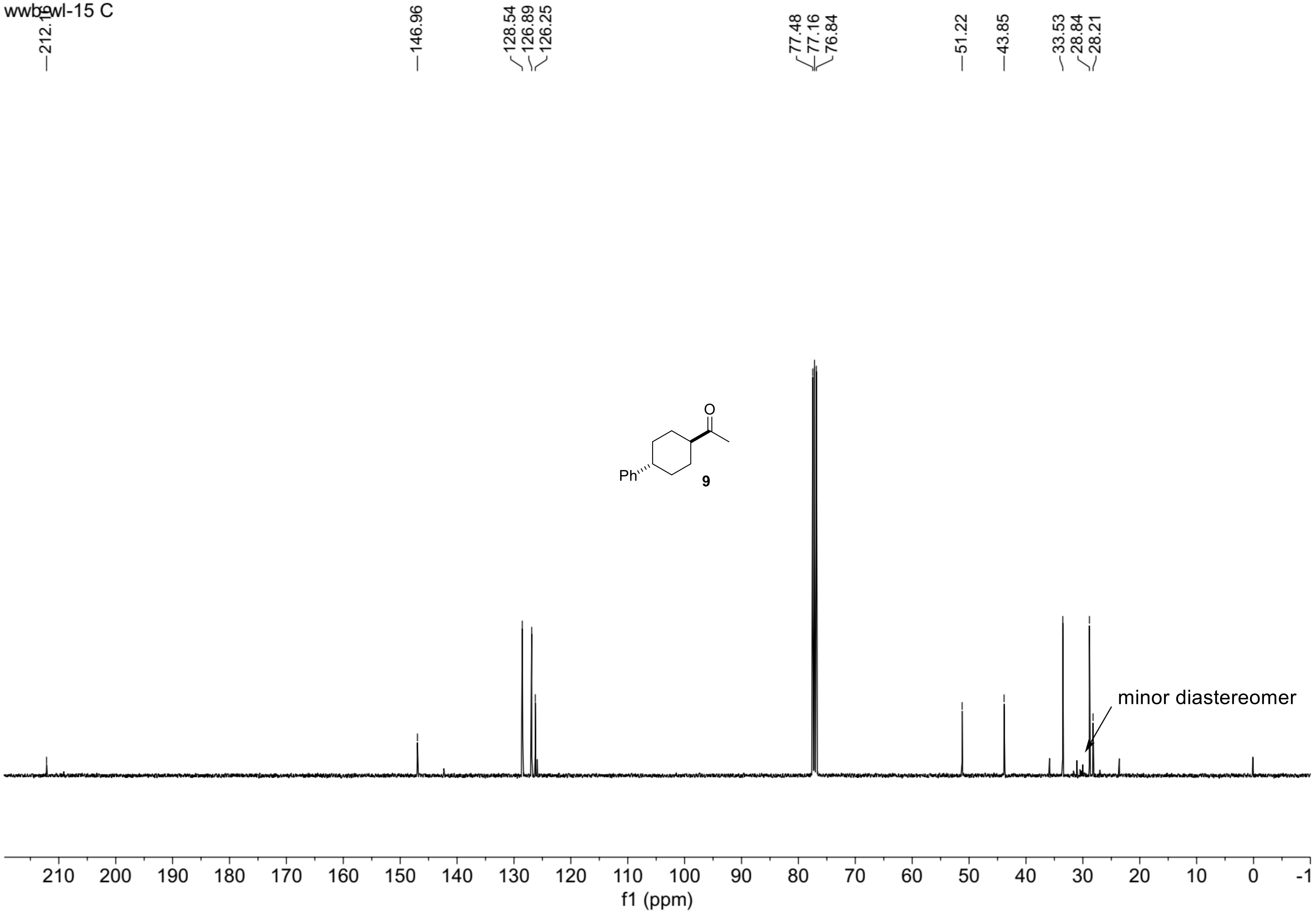

wm-125-2-c
207.3
158.11
136.04
131.91
130.17
120.28
113.59
112.70
77.48
77.16
76.84
55.37
46.67
37.57
35.69
34.40
31.14
29.80
27.86
25.06
23.15
O
H
CN
H
O
56
54
48
46
38
36
34
32
30
28
26
24
22
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

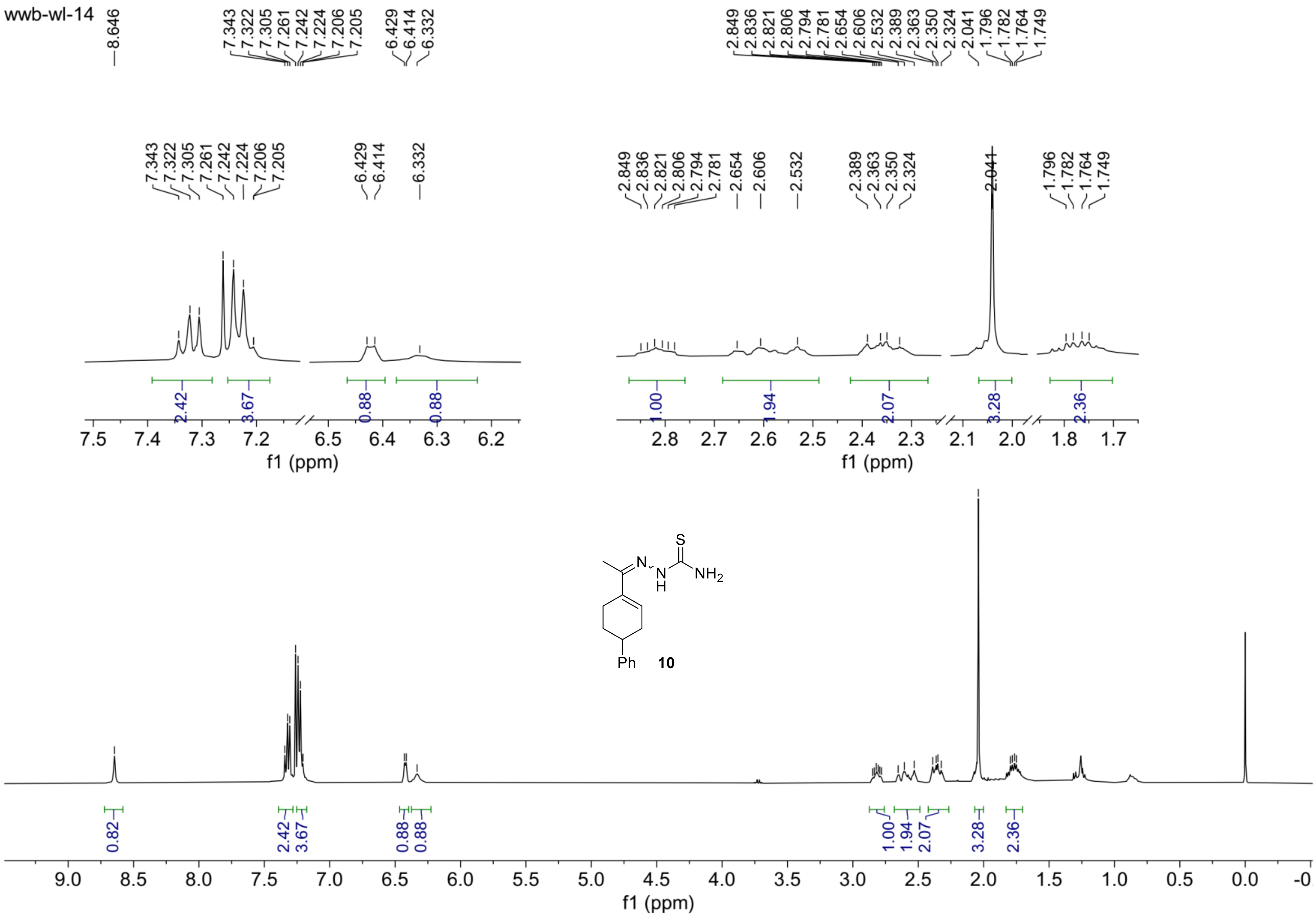
6q

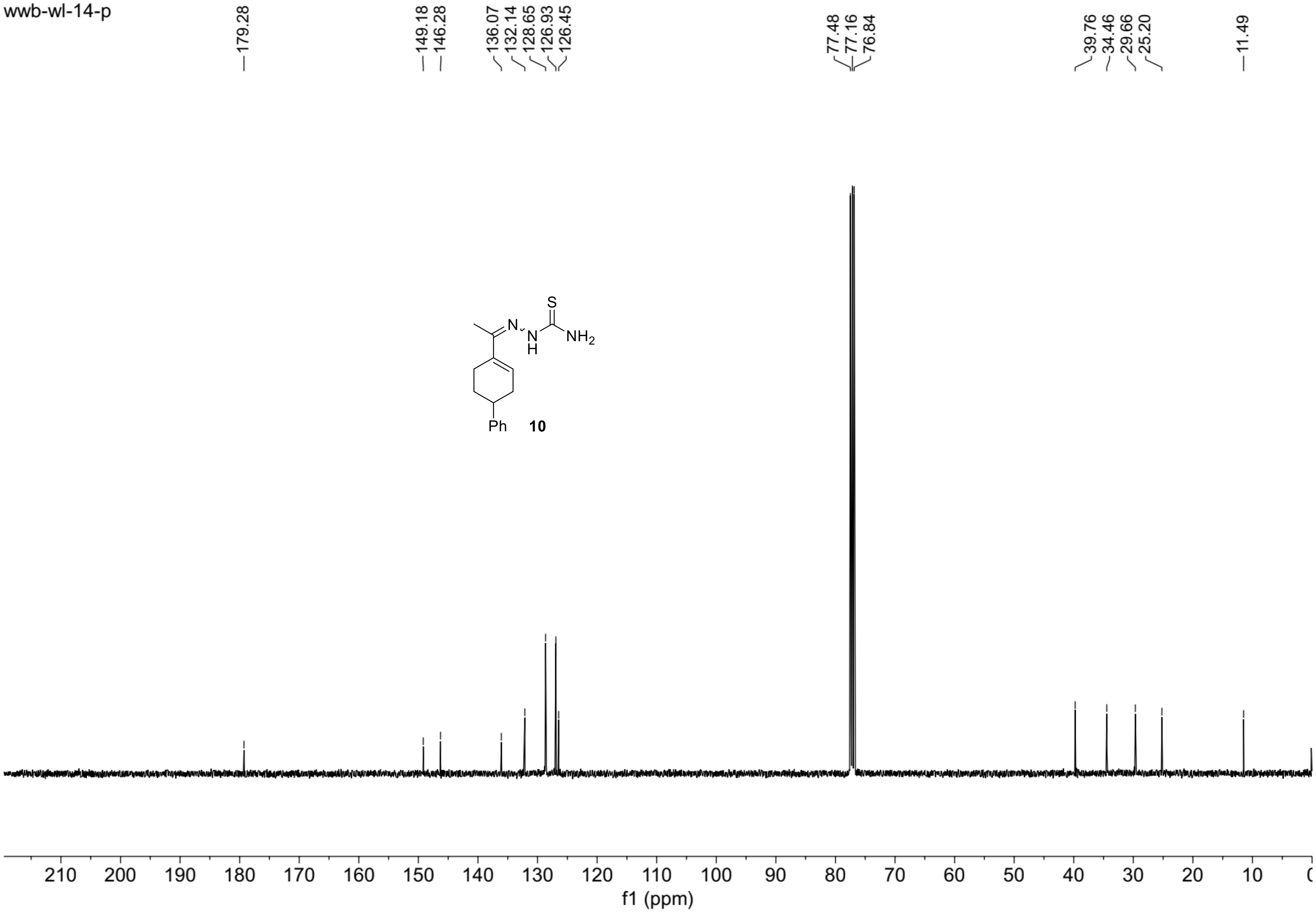

wwb-wn-49-2-2
7.275 7.269 7.259 7.253 7.238 7.233 6.970 6.949
3.219 3.121 3.110 3.098 3.091 3.079 3.067 2.858 2.852 2.840 2.834 2.826 2.812 2.608 2.599 2.591 2.577 2.568 2.559 2.501 2.487 2.455 2.443 2.247 2.213 2.204 2.179 1.998 1.967 1.939 1.922 1.907 1.898 1.875 1.866 1.684 1.674 1.648 1.619 1.610 0.000
1.00 1.08 2.01 1.09 1.04 3.07 1.08 3.23 2.04
1.05 1.01 1.06
f1 (ppm)
Br
H
H
O
CN
6r

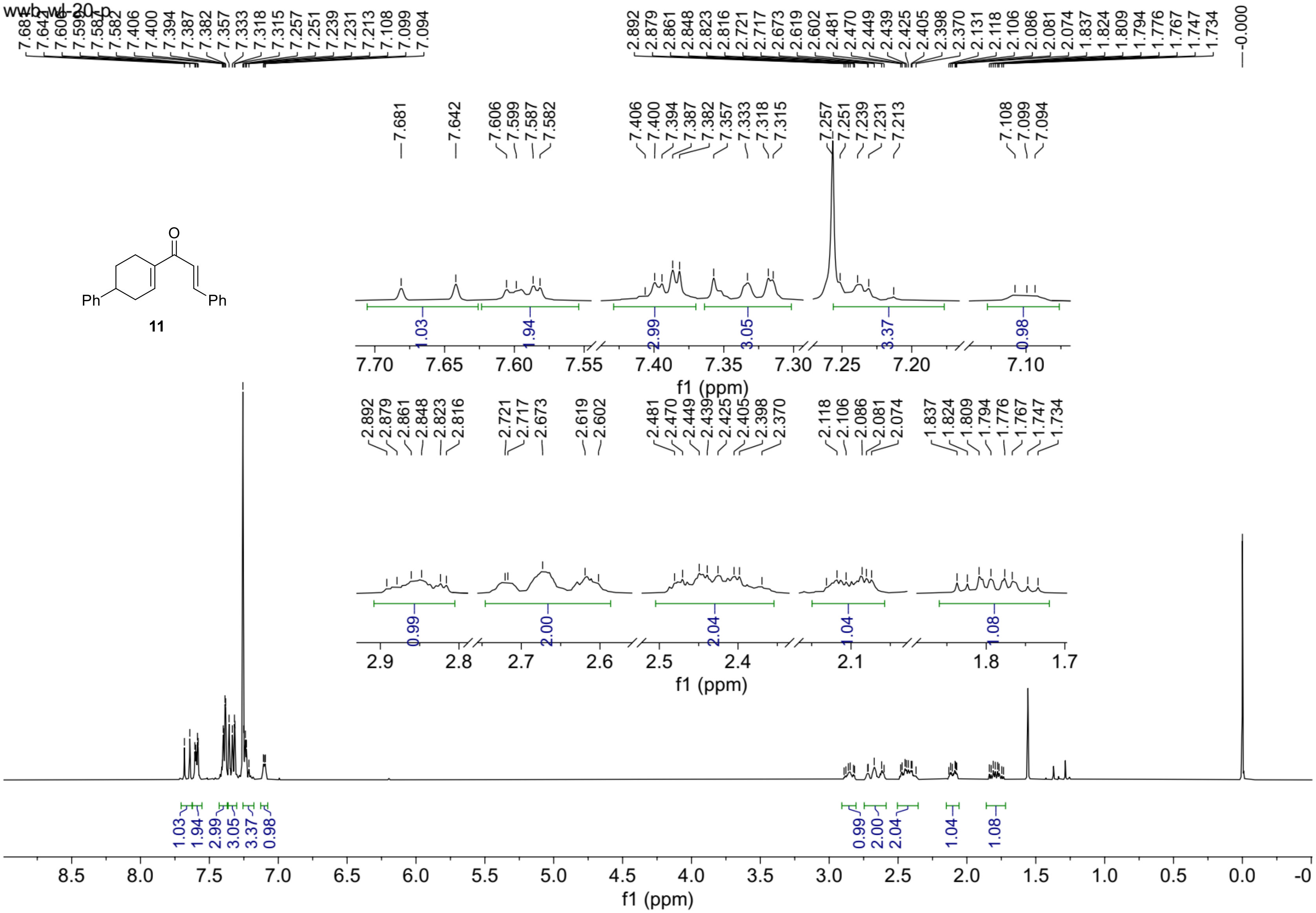

wwb-wn-49-2-2 C 500M
207.1
141.85
133.79
131.98
130.86
129.60
120.04
119.71
77.41
77.16
76.91
46.49
37.03
36.29
34.15
30.92
29.04
27.86
25.02
22.95
143
141
134
133
132
131
130
129
120
f1 (ppm)
46
38
36
34
30
28
f1 (ppm)
Br
H
H
O
CN
6r
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


wwb-wl-15 H
7.317
7.303
7.298
7.295
7.285
7.279
7.255
7.211
7.193
2.530
2.518
2.501
2.493
2.480
2.443
2.422
2.413
2.393
2.384
2.180
2.063
2.043
2.029
2.015
2.007
1.995
1.555
1.528
1.518
1.500
1.484
1.474
1.447
1.04
1.09
3.00
4.08
4.25
2.55 2.45 2.35 2.25 2.15 2.05 1.95 1.55 1.45
f1 (ppm)
2.12
3.15
7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05
f1 (ppm)
O
Ph
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
f1 (ppm)

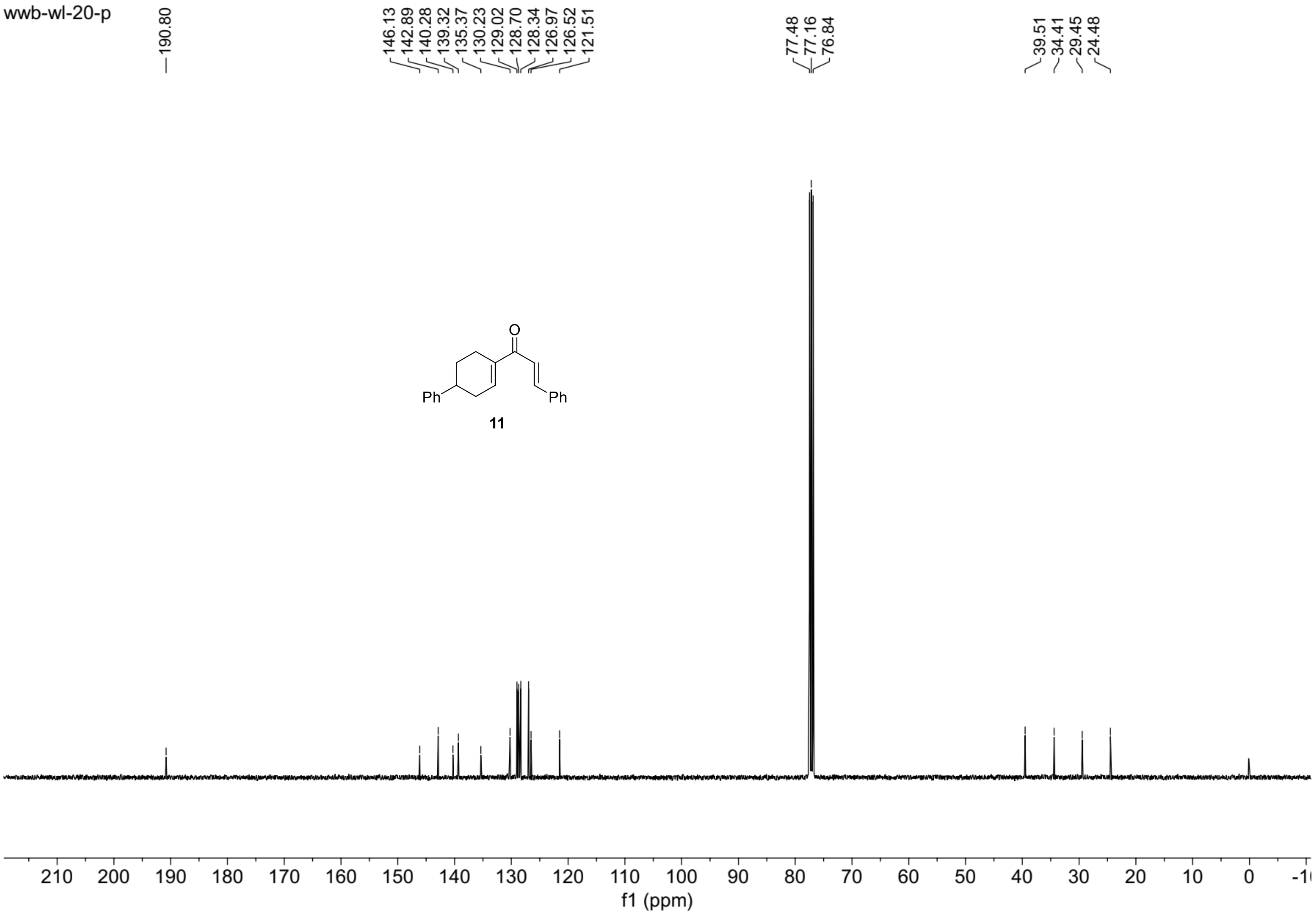

wwbewl-15 C
212.1
146.96
128.54
126.89
126.25
77.48
77.16
76.84
51.22
43.85
33.53
28.84
28.21
O
Ph
9
minor diastereomer
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

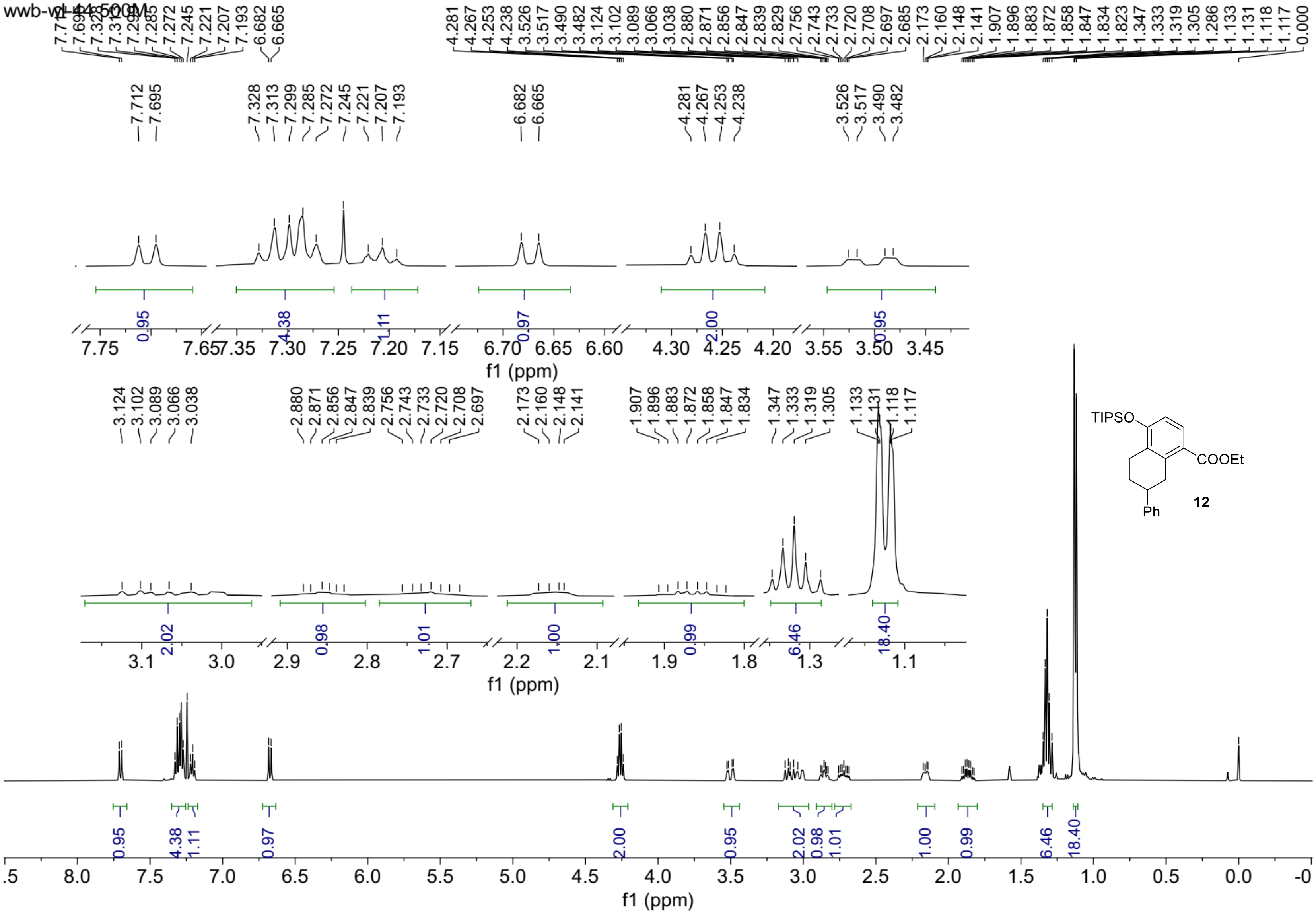

wwb-wl-14
8.646
7.343
7.322
7.305
7.261
7.242
7.224
7.206
7.205
6.429
6.414
6.332
2.849
2.836
2.821
2.806
2.794
2.781
2.654
2.606
2.532
2.389
2.363
2.350
2.324
2.041
1.796
1.782
1.764
1.749
2.42
3.67
0.88
0.88
1.00
1.94
2.07
3.28
2.36
0.82
7.5 7.4 7.3 7.2 6.5 6.4 6.3 6.2
2.8 2.7 2.6 2.5 2.4 2.3 2.1 2.0 1.8 1.7
f1 (ppm)
S
N
N
H
NH$_2$
Ph
10
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

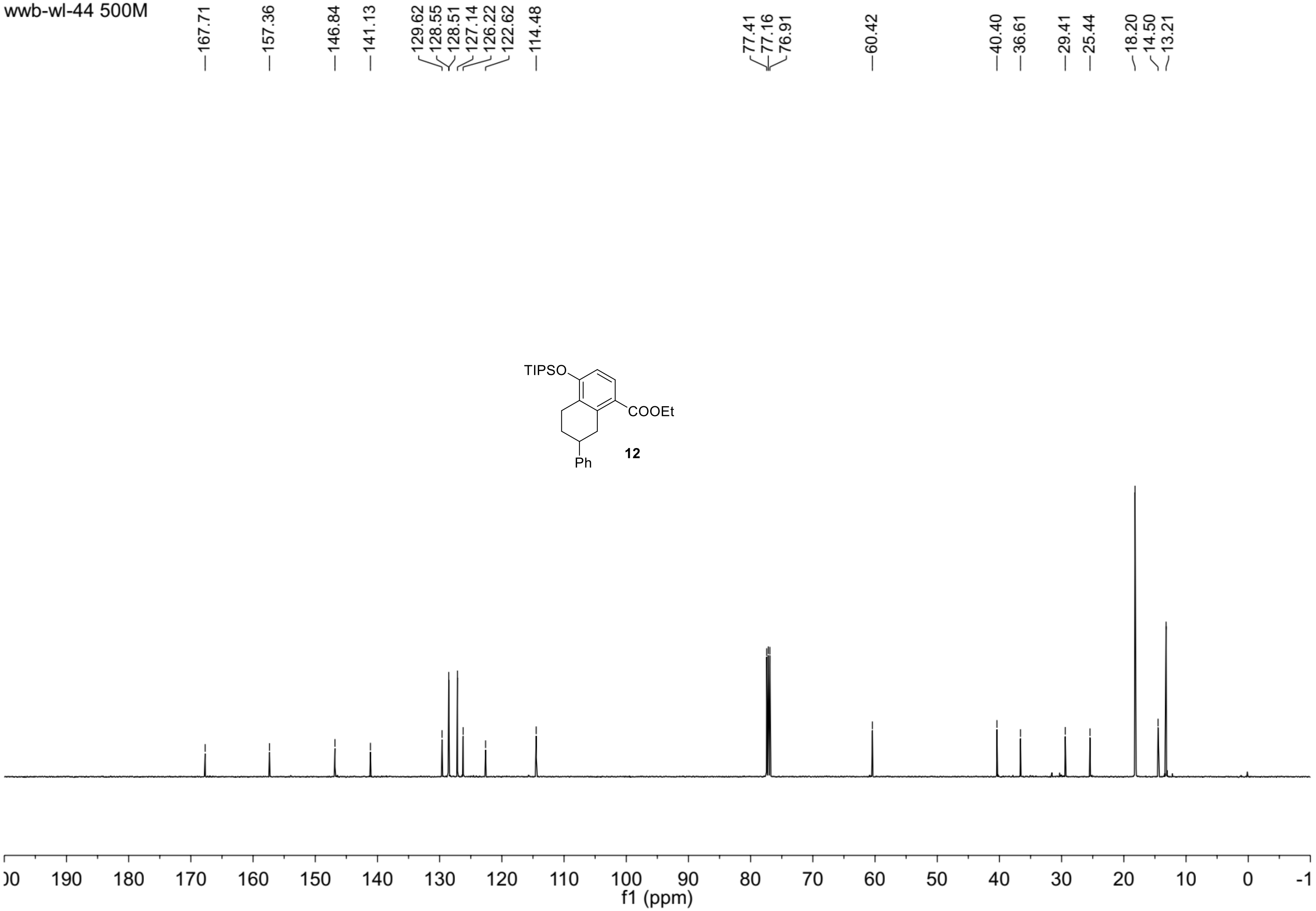

wwb-wl-14-p
179.28
149.18
146.28
136.07
132.14
128.65
126.93
126.45
77.48
77.16
76.84
39.76
34.46
29.66
25.20
11.49
S
N
N
H
NH2
Ph
10
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



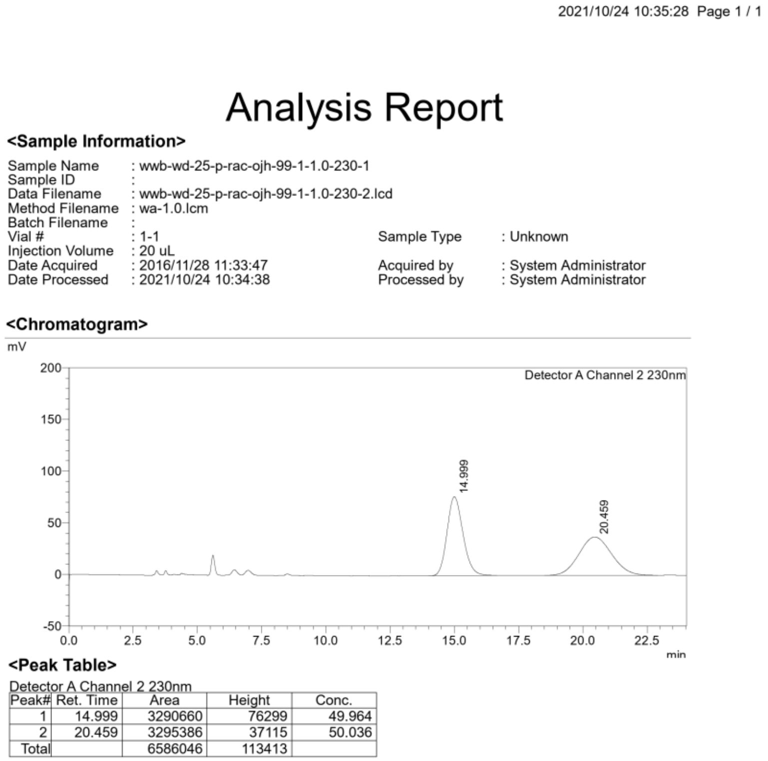
wwb-wl-20-p
7.681
7.642
7.606
7.599
7.587
7.582
7.406
7.400
7.394
7.387
7.382
7.357
7.333
7.318
7.315
7.257
7.251
7.239
7.231
7.213
7.108
7.099
7.094
2.892
2.879
2.861
2.848
2.823
2.816
2.721
2.717
2.673
2.619
2.602
2.481
2.470
2.449
2.439
2.425
2.405
2.398
2.370
2.131
2.118
2.106
2.086
2.081
2.074
1.837
1.824
1.809
1.794
1.776
1.767
1.747
1.734
-0.000
O
Ph
Ph
11
1.03
1.94
2.99
3.05
3.37
0.98
7.70
7.65
7.60
7.55
7.40
7.35
7.30
7.25
7.20
7.10
f1 (ppm)
0.99
2.00
2.04
1.04
1.08
2.9
2.8
2.7
2.6
2.5
2.4
2.1
1.8
1.7
f1 (ppm)
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
f1 (ppm)


wwb-wl-20-p
190.80
146.13
142.89
140.28
139.32
135.37
130.23
129.02
128.70
128.34
126.97
126.52
121.51
77.48
77.16
76.84
39.51
34.41
29.45
24.48
O
Ph
Ph
11
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)


wwb-wl-44-500M
7.712
7.695
7.328
7.313
7.299
7.285
7.272
7.245
7.221
7.207
7.193
6.682
6.665
4.281
4.267
4.253
4.238
3.526
3.517
3.490
3.482
3.124
3.102
3.089
3.066
3.038
2.880
2.871
2.856
2.847
2.839
2.829
2.756
2.743
2.733
2.720
2.708
2.697
2.685
2.173
2.160
2.148
2.141
1.907
1.896
1.883
1.872
1.858
1.847
1.834
1.823
1.347
1.333
1.319
1.305
1.286
1.133
1.131
1.118
1.117
0.000
0.95
4.38
1.11
0.97
2.00
0.95
2.02
0.98
1.01
1.00
0.99
6.46
18.40
f1 (ppm)
TIPSO
COOEt
Ph
12


wwb-wl-44 500M
167.71
157.36
146.84
141.13
129.62
128.55
128.51
127.14
126.22
122.62
114.48
77.41
77.16
76.91
60.42
40.40
36.61
29.41
25.44
18.20
14.50
13.21
TIPSO
COOEt
Ph
12
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

HPLC spectra



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Analysis Report

<Sample Information>

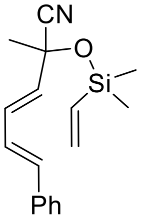
Sample Name : wwb-wd-25-p-rac-ojh-99-1-1.0-230-1
Sample ID :
Data Filename : wwb-wd-25-p-rac-ojh-99-1-1.0-230-2.lcd
Method Filename : wa-1.0.lcm
Batch Filename :
Vial # : 1-1
Injection Volume : 20 uL
Date Acquired : 2016/11/28 11:33:47
Date Processed : 2021/10/24 10:34:38
Sample Type : Unknown
Acquired by : System Administrator
Processed by : System Administrator

<Chromatogram>

<Peak Table>

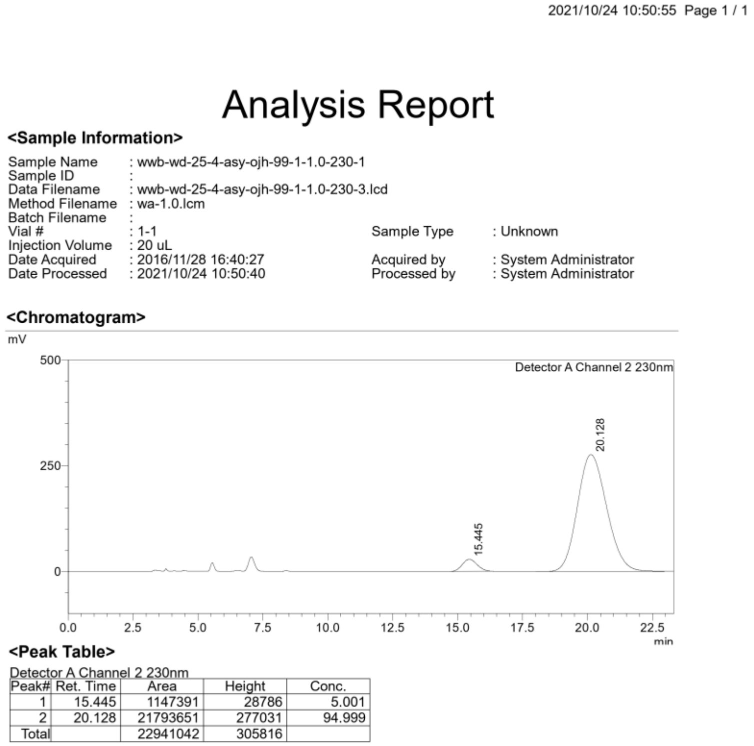
Detector A Channel 2 230nm

Peak#	Ret. Time	Area	Height	Conc.
1	14.999	3290660	76299	49.964
2	20.459	3295386	37115	50.036
Total		6586046	113413	



(*rac*)-**3a**

D:\Data\wuwenbiao\wd\wwb-wd-25-p-rac-ojh-99-1-1.0-230-2.lcd



2021/10/24 10:50:55 Page 1 / 1

Analysis Report

<Sample Information>

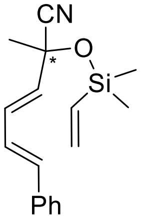
Sample Name : wwb-wd-25-4-asy-ojh-99-1-1.0-230-1
Sample ID :
Data Filename : wwb-wd-25-4-asy-ojh-99-1-1.0-230-3.lcd
Method Filename : wa-1.0.lcm
Batch Filename :
Vial # : 1-1
Injection Volume : 20 uL
Date Acquired : 2016/11/28 16:40:27
Date Processed : 2021/10/24 10:50:40
Sample Type : Unknown
Acquired by : System Administrator
Processed by : System Administrator

<Chromatogram>

<Peak Table>

Detector A Channel 2 230nm

Peak#	Ret. Time	Area	Height	Conc.
1	15.445	1147391	28786	5.001
2	20.128	21793651	277031	94.999
Total		22941042	305816	

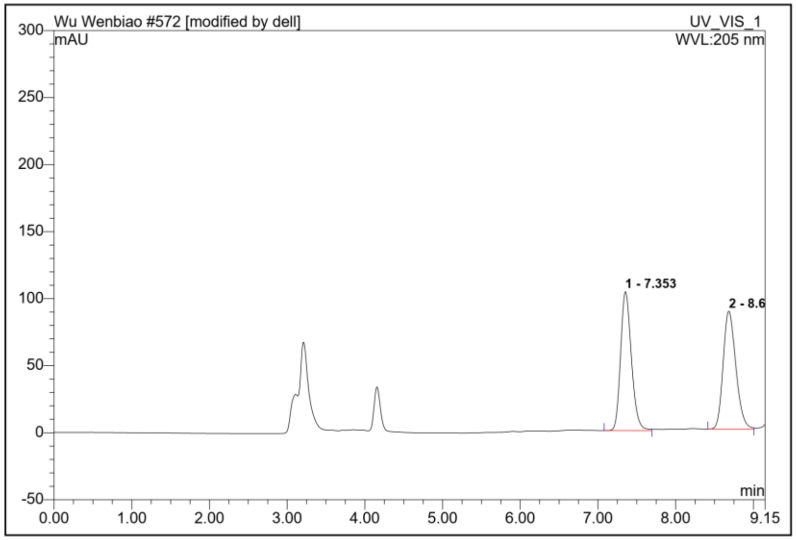


3a

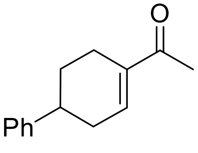
D:\Data\wuwenbiao\wd\wwb-wd-25-4-asy-ojh-99-1-1.0-230-3.lcd

572 wwb-wg-89-rac-ash-95-5-1.0-205-1

Sample Name:	**wwb-wg-89-rac-ash-95-5-1.0-205-1**	*Injection Volume:*	**20.0**
Vial Number:	**710**	*Channel:*	**UV_VIS_1**
Sample Type:	**standard**	*Wavelength:*	**205**
Control Program:	**Wu Wenbiao**	*Bandwidth:*	**n.a.**
Quantif. Method:	**Wu Wenbiao**	*Dilution Factor:*	**1.0000**
Run Time (min):	**20.18**	*Sample Amount:*	**1.0000**



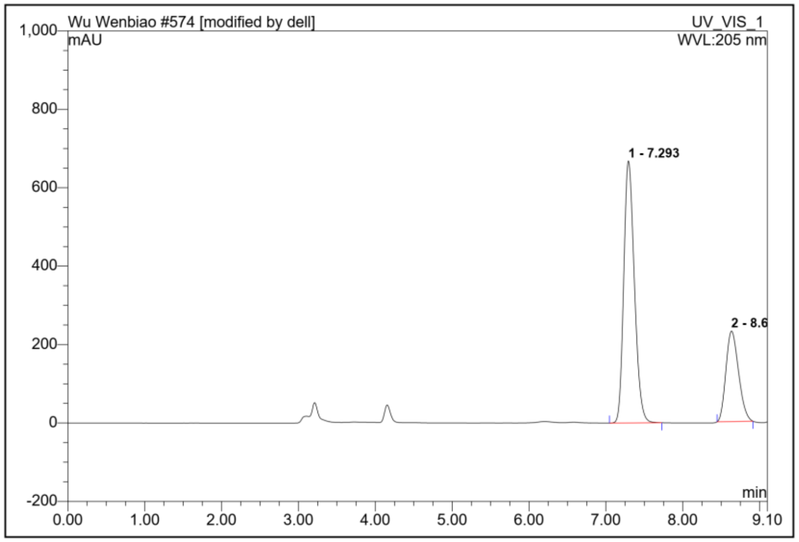
No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.35	n.a.	103.624	16.784	50.23	n.a.	BM *
2	8.69	n.a.	87.863	16.627	49.77	n.a.	BM *
Total:			191.488	33.410	100.00	0.000	



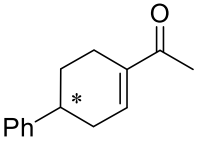
(*rac*)-**5a**

574 wwb-wg-89-2-asy-ash-95-5-1.0-205-1

Sample Name:	**wwb-wg-89-2-asy-ash-95-5-1.0-205-1**	*Injection Volume:*	**20.0**
Vial Number:	**712**	*Channel:*	**UV_VIS_1**
Sample Type:	**standard**	*Wavelength:*	**205**
Control Program:	**Wu Wenbiao**	*Bandwidth:*	**n.a.**
Quantif. Method:	**Wu Wenbiao**	*Dilution Factor:*	**1.0000**
Run Time (min):	**38.95**	*Sample Amount:*	**1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.29	n.a.	668.610	107.214	70.94	n.a.	BMB*
2	8.63	n.a.	230.971	43.914	29.06	n.a.	BMB*
Total:			899.581	151.128	100.00	0.000	



5a