Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Iron-Catalyzed Stereospecific Arylation of Enol Tosylates with Grignard reagents

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1. General experimental details

^{1}HNMR (400, 500 or 600 MHz) and ^{13}CNMR (100, 125 or 150 MHz) spectrawere recorded on Bruker400 Hz or 500 Hz and JNM-ECE 400R or 600R spectrometers unless otherwise noted. The chemical shifts (δ) were quoted in parts per million from tetramethylsilane for ^{1}H and $CDCl_3$ for ^{13}C spectroscopy. High resolution mass spectra (HRMS) were obtained with a Bruker microTOF (ESI). Infrared data were acquired using an AVATAR 360 FT-IRspectrophotometer. Melting points were recorded on a TECH X-4 microscopic instrument and uncorrected.

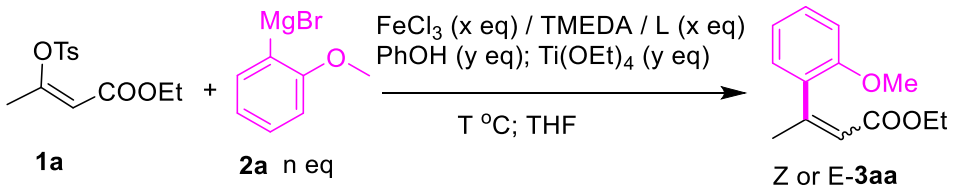
2. General remarks for the preparation of starting materials

All reagents and solvents used for aryl magnesium reagents or lithium reagents and reactions were freshly dehydrated before use. The corresponding glassware was oven dried (120 ^{o}C) and cooled under a stream of argon gas.

Common aryl Grignard reagents such as phenyl magnesium or 4-methoxyphenyl magnesium bromides were prepared according to standard procedure. Functionalized aryl Grignard reagents such as 2-cyanophenyl magnesium chloride or 4-(ethoxycarbonyl)phenyl magnesium chloride were prepared *via* iodine-magnesium exchange using *i*-PrMgCl·LiCl according to Knochel's method.[1] All the Grignard reagents were titrated before use.[2]

All enoltosylates were prepared according to the reported methods[3],[4] and their characterization data were consistent with those reported in the literature.

3. Optimization of reaction conditions



1a **2a** n eq Z or E-**3aa**

entry	n	x	y	T (°C)	L	yield[b](Z:E)
1	1.6[c]	0.1	0.2	0	--	13% (>98:2)
2	1.6[c]	0.1	0.2	25	--	31% (85:15)
3	1.6[c]	0.1	0.2	45	--	65% (50:50)
4	1.6[c]	0.1	0.2	65	--	88% (<2:98)
5	1.6[c]	0.1	0.2	0	PBu_3	8% (>98:2)
6	1.6[c]	0.1	0.2	0	PPh_3	5% (>98:2)
7	1.6[c]	0.1	0.2	0	SIMes·HCl[d]	10% (>98:2)
8	1.6[c]	0.1	0.2	0	SIPr·HCl[d]	53% (>98:2)
9	1.6[c]	0.1	0[e]	0	SIPr·HCl	trace
10	2.0[c]	0.1	0.2	0	SIPr·HCl	63% (>98:2)
11	2.0[c]	0.15	0.2	0	SIPr·HCl	68% (>98:2)
12	2.3[c]	0.15	0.2	0	SIPr·HCl	77% (>98:2)

[a]The reaction was carried out on 2 mmol scale and the amount of TMEDA was twice that of $FeCl_3$ unless indicated otherwise. [b]The yields referred to total yields of two isolated products and ">98:2" meant that only a single isomer was observed by TLC check and ^{1}HNMR of reaction mixtures and isolated products. [c]Total amount of Grignard reagents including those to neutralize phenol as well as NHC·HCl and reduce $FeCl_3$. [d]SIMes = 1,3-bis(2,4,6-trimethyl-phenyl)imida-zolin-2-ylidene; SIPr = 1,3-bis(2,6-di-isopropylphenyl)imidazolin-2-ylidene. [e]The reaction was conducted without either $Ti(OEt)_4$ or PhOH.

4. General Procedure for method A

4.1 Typical procedure of Method A using commonGrignard reagents (**Z-3ab**)

Under Ar atmosphere, $Ti(OEt)_4$ (228 mg, 1.0 mmol) and PhOH (94 mg, 1.0 mmol) were dissolved in 3 mL THF and stirred at room temperature for 20 to 30 min. A solution of C_6H_5MgBr (12.0 mL; 12.0 mmol, 1.0 M in THF) was added dropwise through a syringe to the resulting mixture during 10–15 min, and then stirred at room temperature for 30 to 40 min.

To another three-necked round-bottom flask were added ethyl (Z)-3-(tosyloxy)-but-2-enoate (1.42g, 5mmol) and 5 mL THF. Under stirring, $FeCl_3$ (122 mg; 0.75 mmol), TMEDA (174 mg, 1.5 mmol) and SIPr·HCl (308 mg, 0.75 mmol) were added to the resulting solution and stirred for 10 min at room temperature.The mixture was cooled to 0^{o}C and the above-prepared mixed titanate was added dropwise. The stirring was continued at 0 ^{o}C until the reaction finished (monitored by TLC, about 6–8 hr).The reaction was quenched by adding 100 mL distilled water. After being filtered, the solid and filtrate were extracted with CH_2Cl_2. The organic layer was washed with 50 mL saturated NaCl aqueous solution and dried over Na_2SO_4,then concentrated to yield the crude compound, which was purified by column chromatography to affordthe desired product **Z-3ab** (779 mg, 82% yield).

4.2 Typical procedure of Method A using functionalized Grignard reagents (Z-3ag)

Under Ar atmosphere, the solution of 2-IC_6H_4CN (2.75 g, 12 mmol) in 5 mL THF was cooled to -40 ^{o}C under stirring. To this solution was added *i*-PrMgCl·LiCl (12.0 mL; 12 mmol, 1.0 M in THF)dropwise. The stirring was continued at that temperature until the exchange reaction was completed (monitored by TLC).

Under Ar atmosphere, THF (3 mL); $Ti(OEt)_4$ (228 mg, 1.0 mmol) and PhOH (94 mg, 1.0 mmol) were added to another round-bottom flask, and the mixture was stirred and cooled to -45 ～ -50^{o}C. To this solution, the above-prepared Knochel-type functionalized Grignard reagent was added dropwisethrough a syringeduring 10-15 min with the temperature being kept below -40^{o}C. After the addition, the mixture was

allowed to come to room temperature in 2 hr, and stirred at that temperature for 30 min. This mixture could be used directly for the arylation.

In the reactions where the concomitant *i*-PrI was removed, the solvent as well as *i*-PrI were taken off the mixture under vacuum (about 10 mmHg) during which the temperature was below 20 ^{o}C until the mixture became a paste. THF (10 mL) was added to the paste and the stirring was continued until a solution was formed.

To another three-necked round-bottom flask were added ethyl (Z)-3-(tosyloxy)-but-2-enoate (1.42 g, 5 mmol) and 5 mL THF. Under stirring, $FeCl_3$ (122 mg, 0.75 mmol), TMEDA (174 mg, 1.5 mmol) and SIPr·HCl (308 mg, 0.75 mmol) were added to the resulting solution and stirred for 10 min at room temperature.The mixture was cooled to 0 ^{o}C and the above-prepared mixed titanate was added. The stirring was continued at 0 ^{o}C until the reaction finished (monitored by TLC, about 6–8 hr). The reaction was quenched by adding 100 mL distilled water. After being filtered, the solid and filtrate were extracted with CH_2Cl_2. The organic layer was washed with 50 mL saturated NaCl aqueous solution and dried over Na_2SO_4, then concentrated to yield the crude compound, which was purified by column chromatography to afford the desired product **Z-3ag** (714 mg, 71% yield).

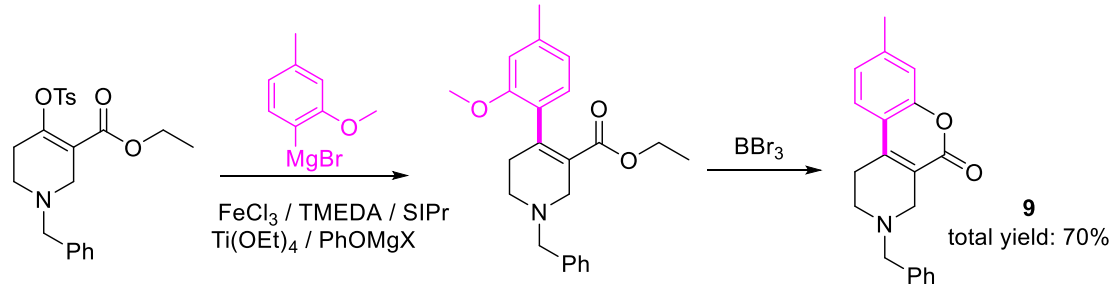
5. Typical procedure of Method B (E-3ab)

Under Ar atmosphere, $Ti(OEt)_4$ (228 mg, 1.0 mmol) and PhOH (94 mg,1.0 mmol) were dissolved in 3 mL THF and stirred at room temperature for 20 to 30 min. A solution of C_6H_5MgBr (12 mL; 12.0 mmol, 1.0 M in THF) was added dropwise through a syringe to the resulting mixtureduring 10–15 min, and then stirred at room temperature for 30 to 40 min.

To another three-necked round-bottom flask were added ethyl (E)-3-(tosyloxy)-but-2-enoate (1.42 g, 5mmol) and 5 mL THF. Under stirring, $FeCl_3$ (122 mg, 0.75mmol), TMEDA (174 mg, 1.5mmol) were added to the resulting solution and stirred for 10 min at room temperature. The above-prepared mixed titanate was added dropwise. The mixture was heated to 65^{o}C under stirring. The stirring was continued at 65 ^{o}C until the reaction finished (monitored by TLC, about 6hr).The reaction was quenched by adding 100 mL distilled water. After being filtered, the solid and filtrate were extracted with CH_2Cl_2. The organic layer was washed with 50 mL saturated NaCl aqueous solution and dried over Na_2SO_4,thenconcentrated to yield the crude compound, which was

purified by column chromatography to afford the desired product **E-3ab** (817 mg, 86% yield).

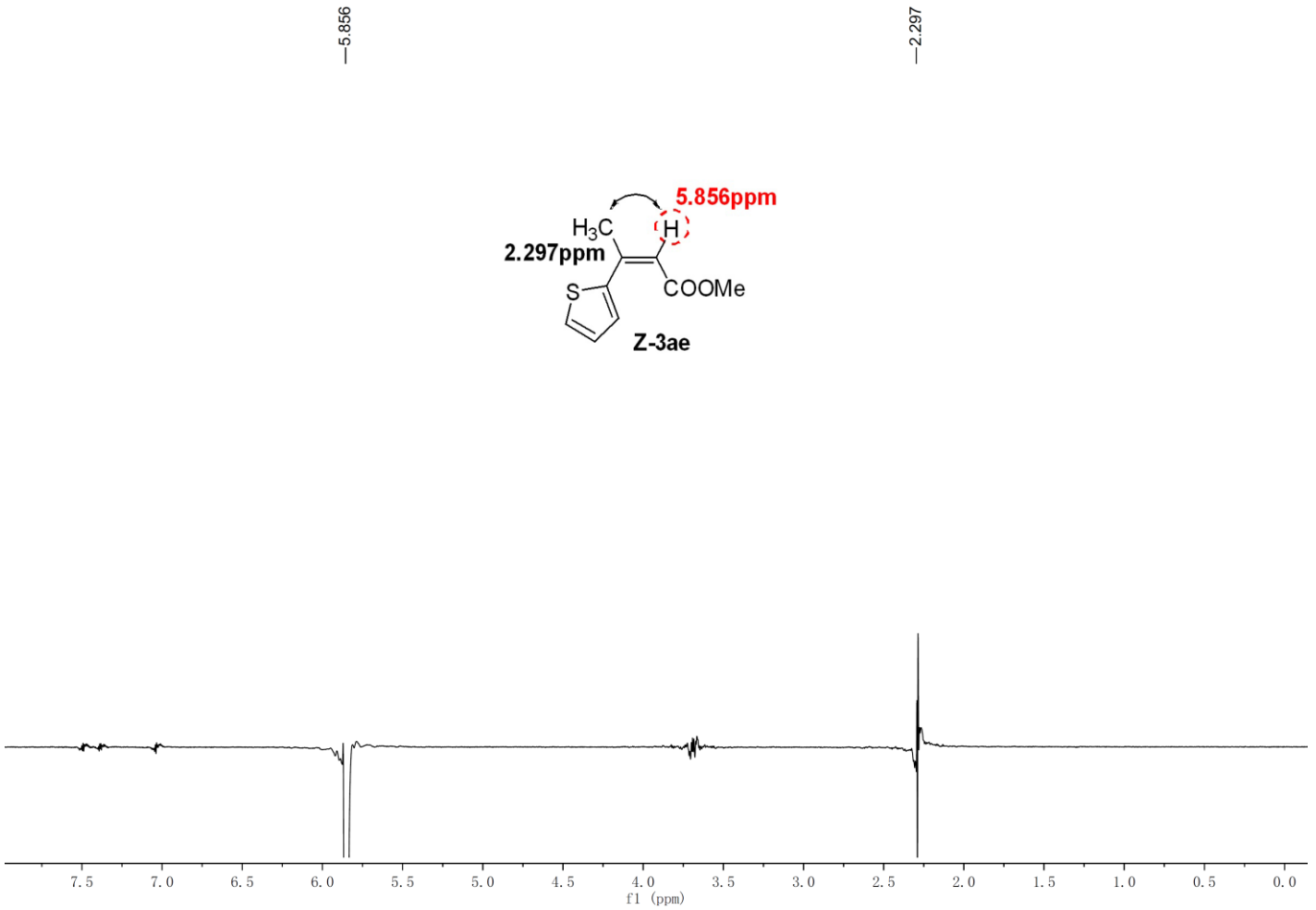
6. Preparations of 9



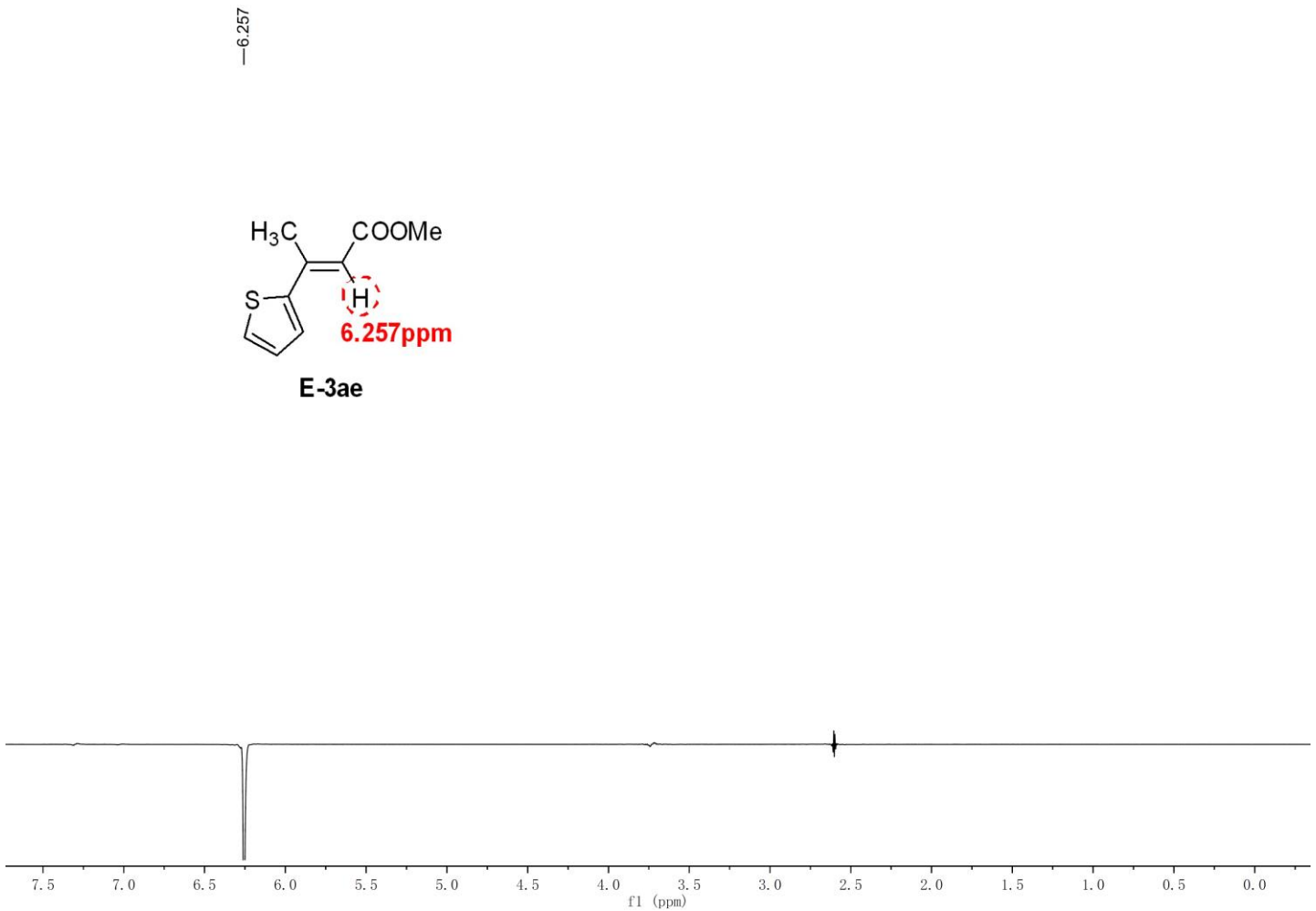
According to typical procedure 3.1, the crude products of arylation reaction were obtained and dissolved in 20 mL CH_2Cl_2. The mixture was stirred and cooled to -78 ^oC, and BBr_3 (2.5 g, 10mmol) was added dropwise. After addition, the mixture was allowed to come to room temperature and stirred for 24 h. The reaction was quenched with methanol (20 mL). The product was taken up with CH_2Cl_2 (4 × 50 mL) and washed with saturated $NaHCO_3$aqueous solution (50 mL). After dried over Na_2SO_4 and concentrated by rotary evaporator, crude product was obtained, which was purified by column chromatography to afford the desired product **9** (1068 mg, 70% yield).

7. Confirmation of configurational retention in arylations by NMR studies

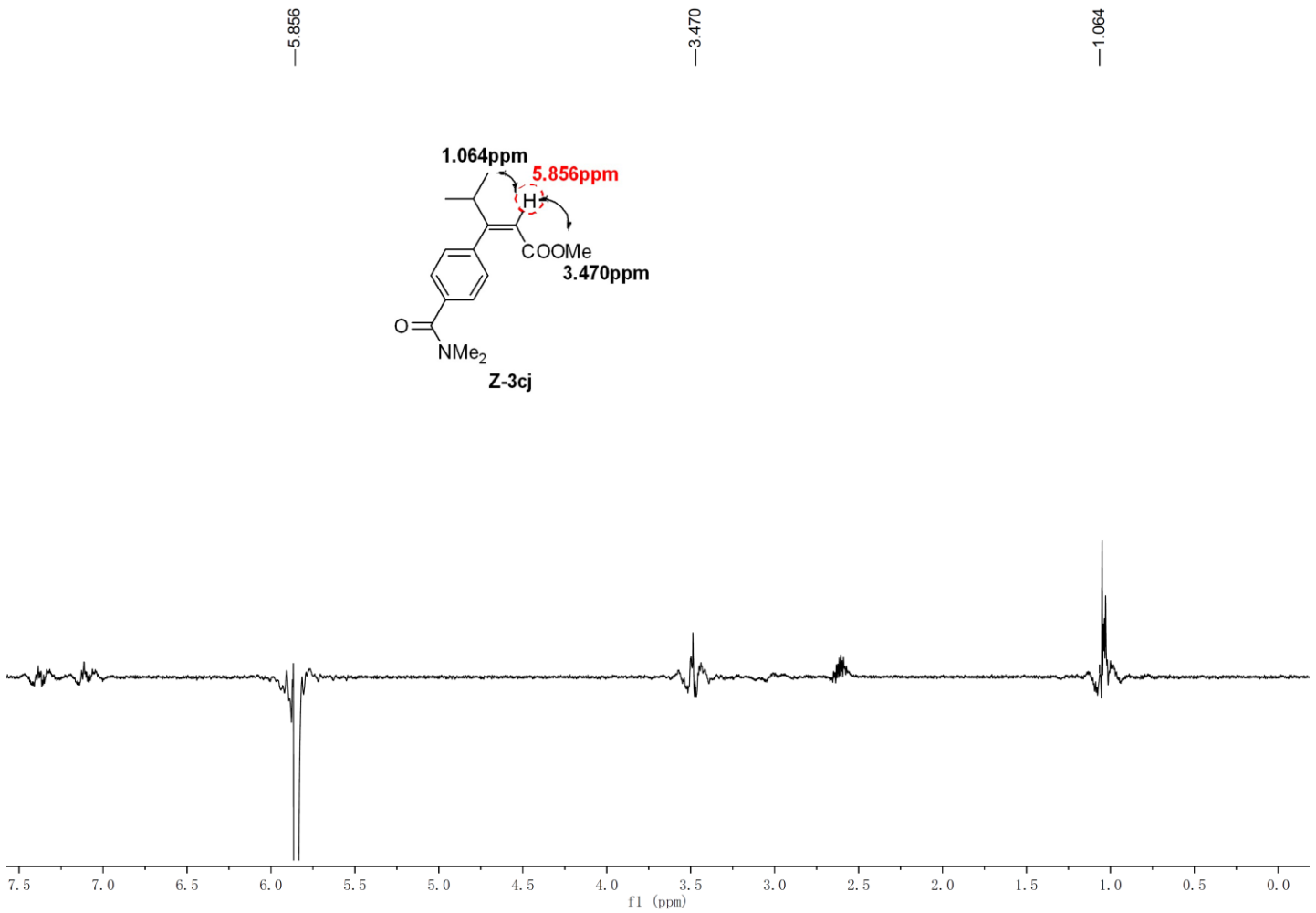
In this paper, almost all products were synthesized in E/Z isomer pairs. Meanwhile, we also successfully distinguished two isomers in every E/Z pair on TLC (see the R_f values in **Characterization data**), which allowed us to determine that only one isomer was generated during all our arylations. On this basis, we further confirmed that the arylations using method A proceeded with configuration retention by comparing the ^1H NOESY spectra of the products in Z/E pairs. Below are ^1H NOESY spectra for five Z/E pairs of arylation products.



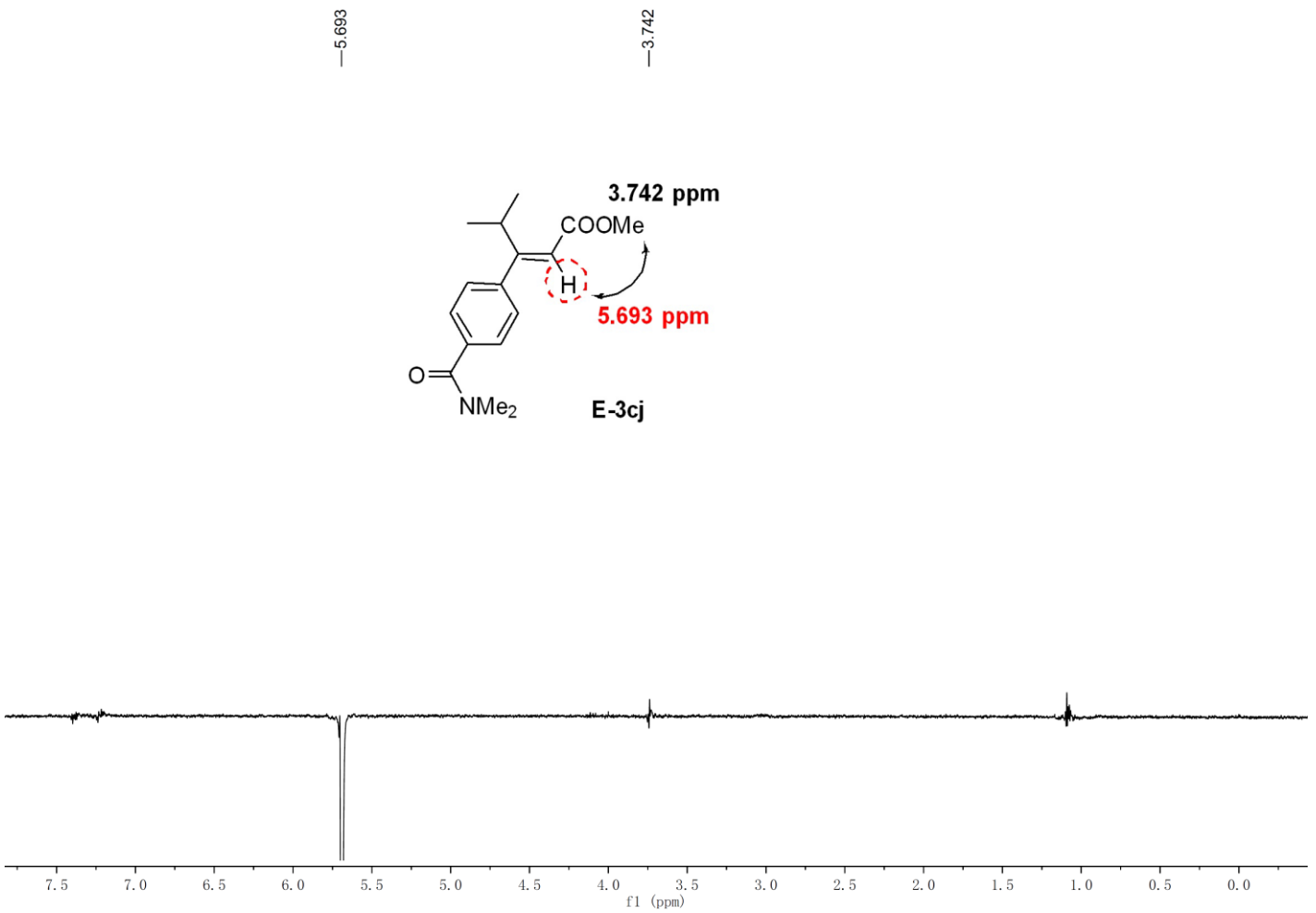
1D selective ^{1}H NOESY spectra (BF1 = 400.0 MHz;*selnogp*; NS = 128; DS = 2; TD = 65536; D1 = 2; SW =20 ppm; O1P = 4, mixing time D8 = 300 ms) of the **Z-3ae** in $CDCl_3$ at 300K irradiated at δ 5.86 ppm.



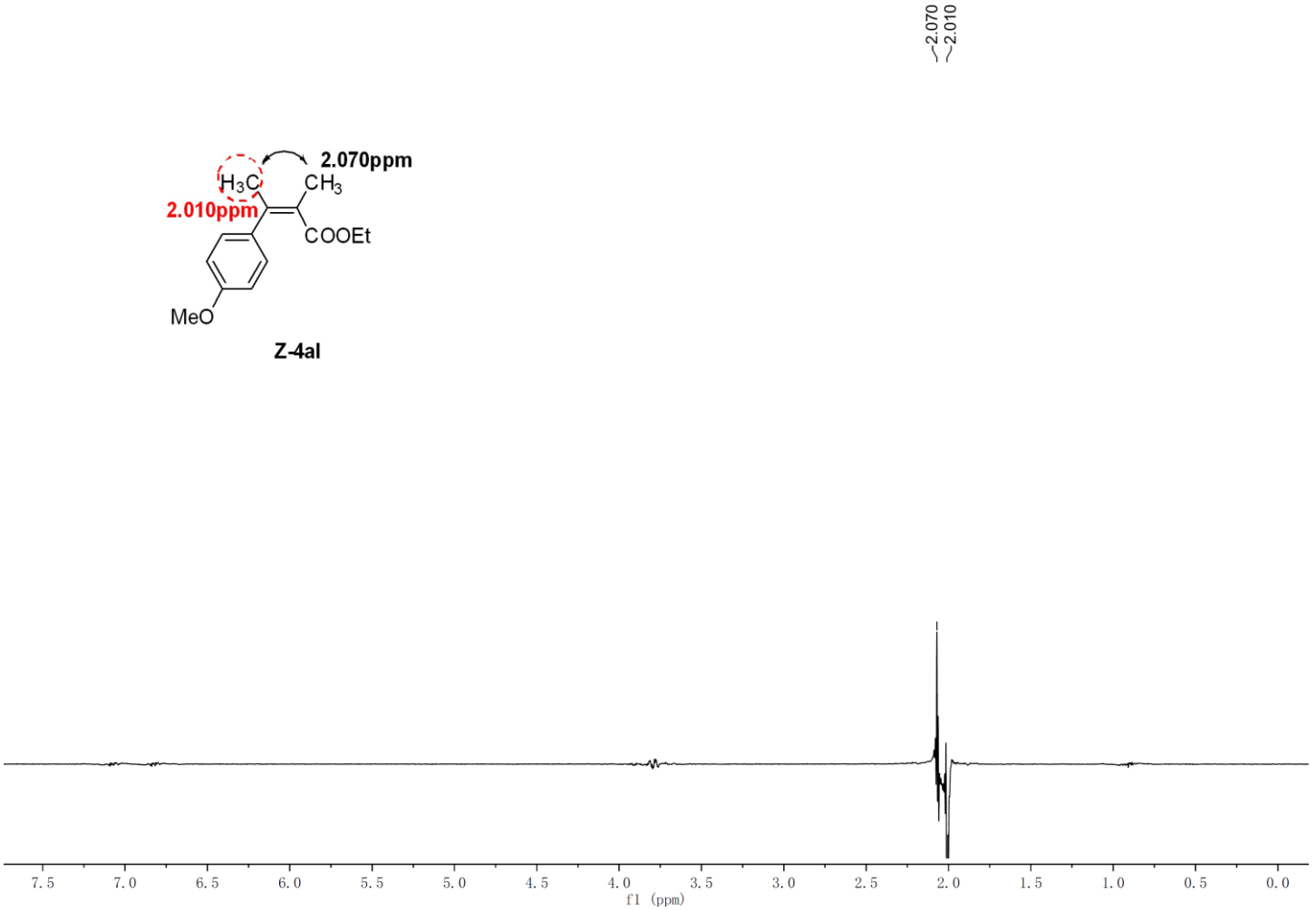
1D selective ^{1}H NOESY spectra (BF1 = 400.0 MHz;*selnogp*; NS = 128; DS = 2; TD = 65536; D1 = 2; SW =20 ppm; O1P = 4, mixing time D8 = 300 ms) of the **E-3ae** in $CDCl_3$ at 300K irradiated at δ 6.26 ppm.



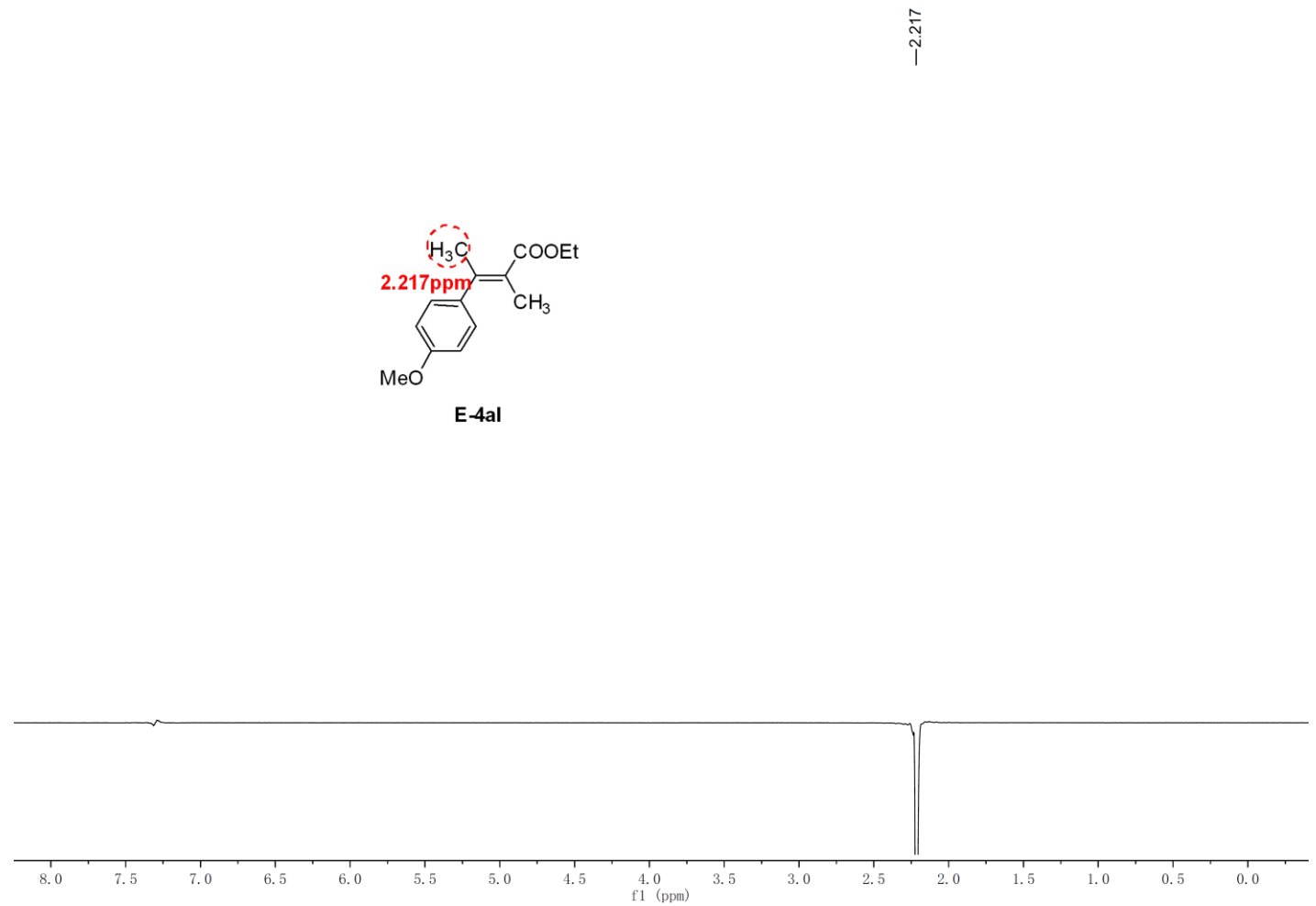
1D selective ^{1}H NOESY spectra (BF1 = 400.0 MHz;*selnogp*; NS = 128; DS = 2; TD = 65536; D1 = 2; SW =20 ppm; O1P = 4, mixing time D8 = 300 ms) of the**Z-3cj** in $CDCl_3$ at 300K irradiated at δ 5.86 ppm.



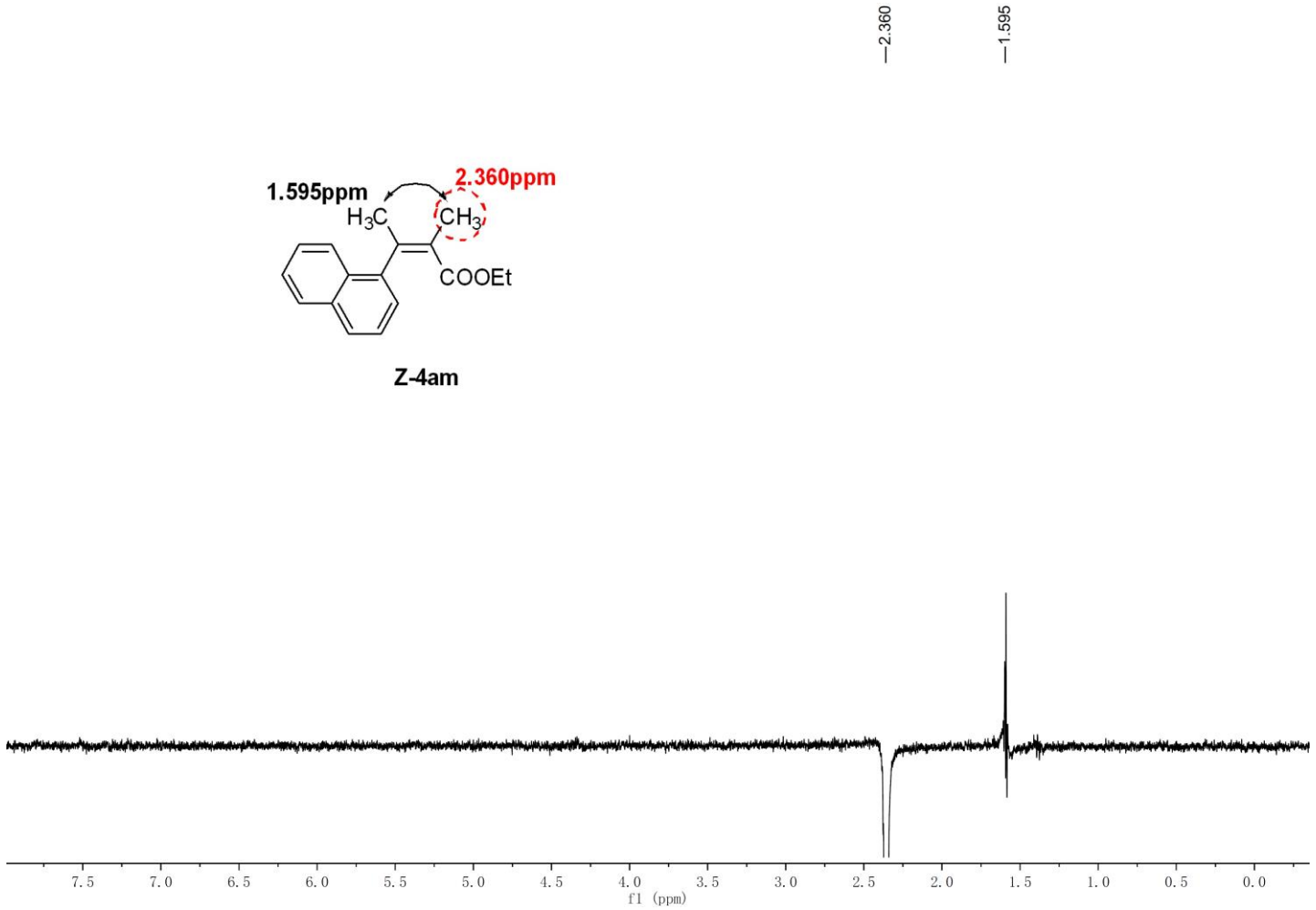
1D selective ^{1}H NOESY spectra (BF1 = 400.0 MHz;*selnogp*; NS = 128; DS = 2; TD = 65536; D1 = 2; SW =20 ppm; O1P = 4, mixing time D8 = 300 ms) of the **E-3cj** in $CDCl_3$ at 300K irradiated at δ 5.69 ppm.



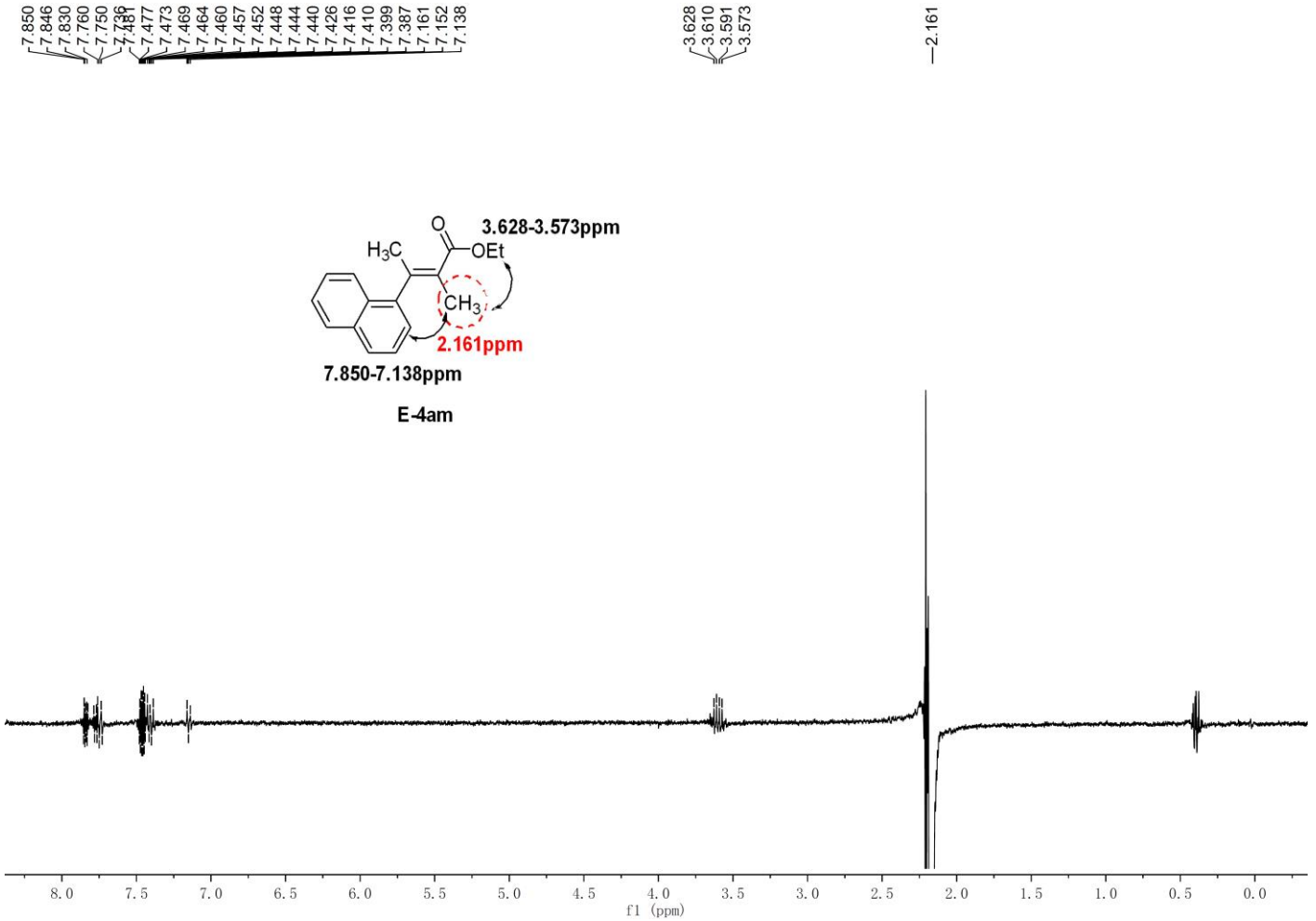
1D selective ^{1}H NOESY spectra (BF1 = 400.0 MHz;*selnogp*; NS = 128; DS = 2; TD = 65536; D1 = 2; SW = 20 ppm; O1P = 4, mixing time D8 = 300 ms) of the **Z-4al** in $CDCl_3$ at 300K irradiated at δ 2.01 ppm.



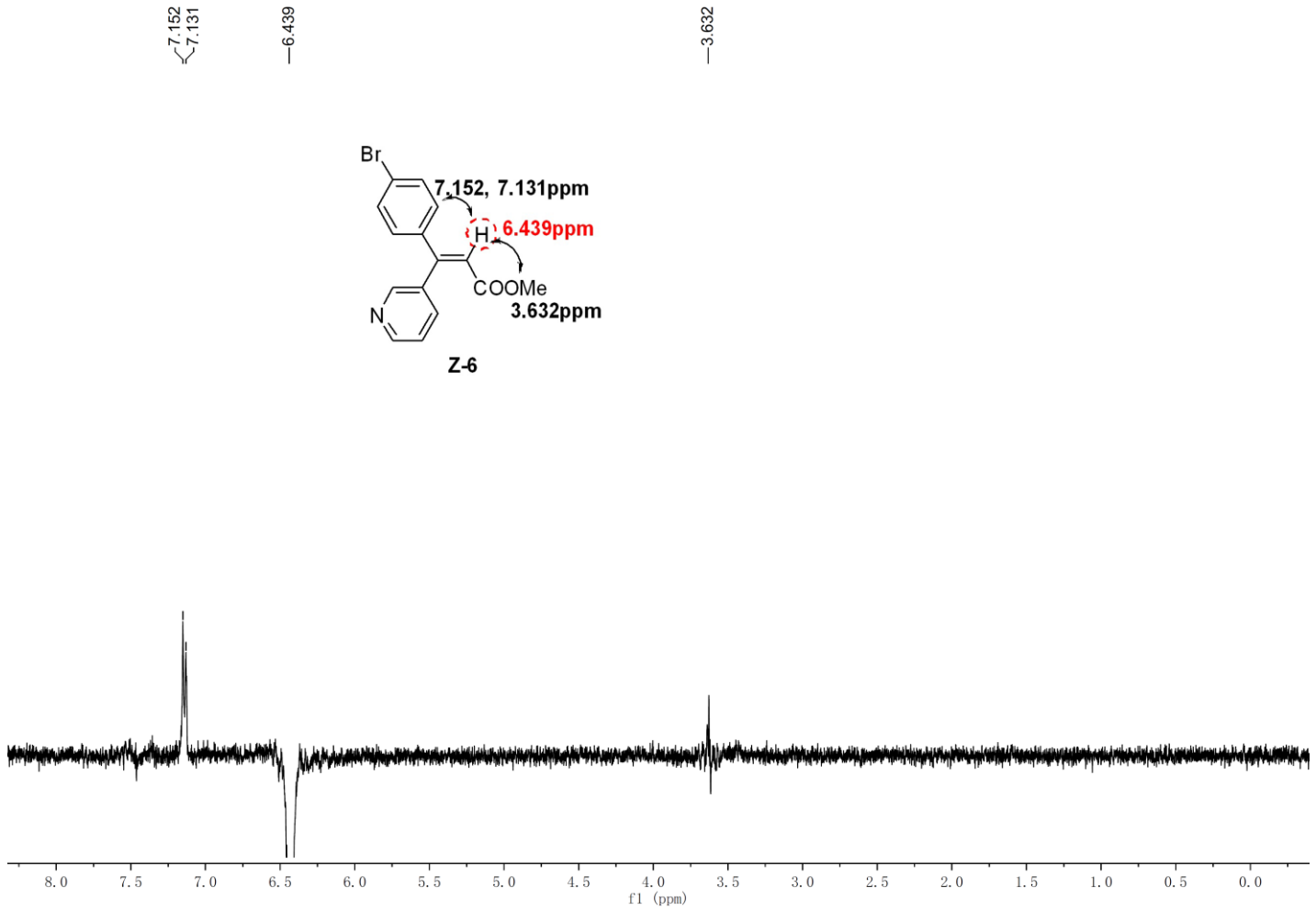
1D selective ^{1}H NOESY spectra (BF1 = 400.0 MHz;*selnogp*; NS = 128; DS = 2; TD = 65536; D1 = 2; SW = 20 ppm; O1P = 4, mixing time D8 = 300 ms) of the **E-4al** in $CDCl_3$ at 300K irradiated at δ 2.22 ppm.



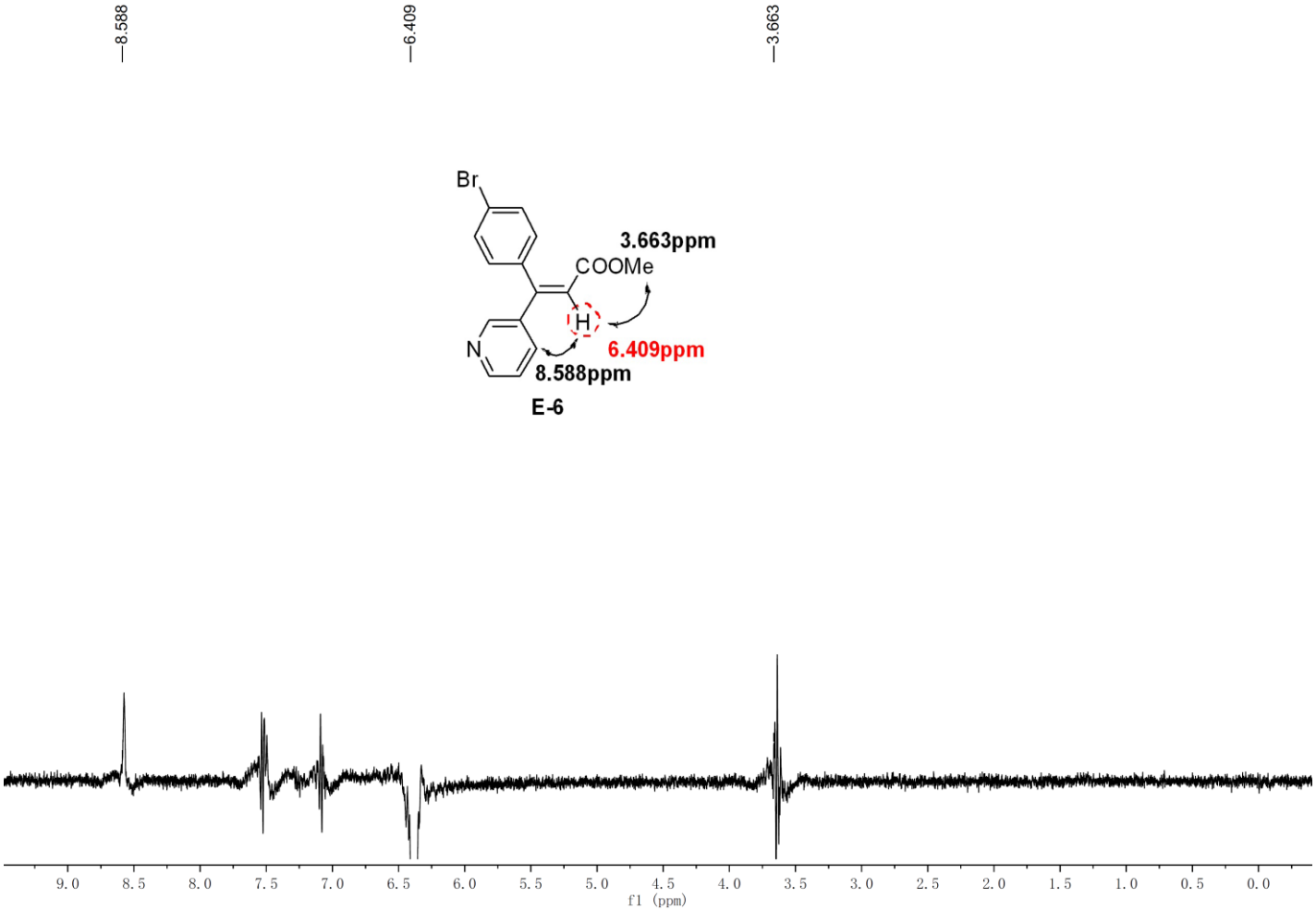
1D selective ^{1}H NOESY spectra (BF1 = 400.0 MHz;*selnogp*; NS = 128; DS = 2; TD = 65536; D1 = 2; SW = 20 ppm; O1P = 4, mixing time D8 = 300 ms) of the **Z-4am** in $CDCl_3$ at 300K irradiated at δ 2.36 ppm.



1D selective ^{1}H NOESY spectra (BF1 = 400.0 MHz;*selnogp*; NS = 128; DS = 2; TD = 65536; D1 = 2; SW =20 ppm; O1P = 4, mixing time D8 = 300 ms) of the **E-4am** in $CDCl_3$ at 300K irradiated at δ 2.16 ppm.

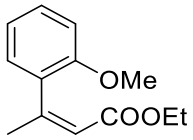


1D selective ^{1}H NOESY spectra (BF1 = 400.0 MHz;*selnogp*; NS = 128; DS = 2; TD = 65536; D1 = 2; SW = 20 ppm; O1P = 4, mixing time D8 = 300 ms) of the **Z-6** in $CDCl_3$ at 300K irradiated at δ 6.44 ppm.



1D selective ^{1}H NOESY spectra (BF1 = 400.0 MHz;*selnogp*; NS = 128; DS = 2; TD = 65536; D1 = 2; SW =20 ppm; O1P = 4, mixing time D8 = 300 ms) of the **E-6** in $CDCl_3$ at 300K irradiated at δ 6.41 ppm.

8. Characterization data

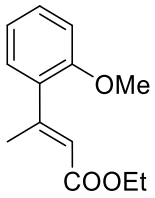


Z-3aa

Ethyl (Z)-3-(2-methoxyphenyl)but-2-enoate[5]

Yellow oil; 88% yield (968 mg); R_f = 0.22 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3028, 2951, 1750, 1722, 1333, 1181, 752, 690; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.283-7.237 (m, 1H), 7.011 (dd, J = 7.5, 1.8 Hz, 1H), 6.948-6.907 (m, 1H), 6.889 (d, J = 8.3 Hz, 1H), 5.949-5.945 (m, 1H), 4.001-3.931 (m, 2H), 3.787 (s, 3H), 2.139 (dd, J = 1.5, 0.7 Hz, 3H), 1.040 (td, J = 7.1, 0.7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 165.8, 155.5, 153.3, 130.5, 128.8, 128.1, 120.4, 119.0, 110.8, 59.6, 55.6, 26.2, 14.1.

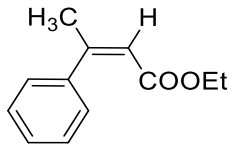


E-3aa

Ethyl (E)-3-(2-methoxyphenyl)but-2-enoate[6]

Yellow oil; 88% yield (968 mg); R_f = 0.35 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3036, 2903, 1756, 1722, 1338, 1177, 970, 762; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.289-7.232 (m, 1H), 7.116 (dd, J = 7.5, 1.8 Hz, 1H), 6.925-6.862 (m, 2H), 5.874-5.869 (m, 1H), 4.206-4.152 (m, 2H), 3.797 (s, 3H), 2.470 (d, J = 1.3 Hz, 3H), 1.280 (td, J = 7.1, 0.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 166.9, 156.7, 156.4, 133.2, 129.5, 128.9, 120.6, 119.3, 111.1, 59.8, 55.5, 19.9, 14.4.

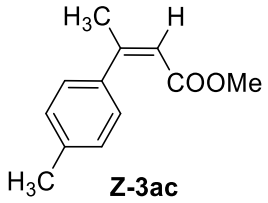


Z-3ab

Ethyl (Z)-3-phenylbut-2-enoate[3]

Yellow oil; 82% yield (779 mg); R_f = 0.25 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm^{-1}, KBr): 3029, 2891, 1747, 1728, 1250, 1190, 750, 681; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.371-7.289 (m, 3H), 7.220-7.198 (m, 2H), 5.914 (s, 1H), 4.002 (q, J = 7.2 Hz, 2H), 2.184 (s, 3H), 1.081 (t, J = 7.2 Hz, 3H);^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 166.0, 155.4, 141.0, 128.0, 127.8, 126.9, 117.9, 59.8, 27.2, 14.0.

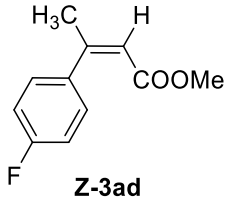


Z-3ac

Methyl (Z)-3-(p-tolyl)but-2-enoate[3]

Yellow oil; 74% yield (703 mg); R_f = 0.23 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm^{-1}, KBr): 3030, 2890, 1786, 1728, 1201, 1167, 840, 684; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.169 (d, J = 8.2 Hz, 2H), 7.120 (d, J = 8.0 Hz, 2H), 5.901 (s, 1H), 3.574 (s, 3H), 2.361 (s, 3H), 2.177 (s, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 166.5, 156.2, 137.8, 137.7, 128.7, 126.9, 116.9, 51.1, 27.3, 21.4.

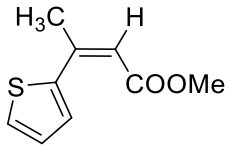


Z-3ad

Methyl (Z)-3-(4-fluorophenyl)but-2-enoate[7]

Yellow oil; 76% yield (737 mg); R_f = 0.20 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm^{-1}, KBr): 3038, 2904, 1760, 1721, 1248, 1150, 827, 676; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.221-7.171 (m, 2H), 7.068-7.010 (m, 2H), 5.926 (d, J = 1.5 Hz, 1H), 3.572 (s, 3H), 2.170 (d, J = 1.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 166.2, 162.5 (d, J = 245.0 Hz, 1C), 155.0, 136.5, 128.8 (d, J = 8.2 Hz, 1C), 117.6, 115.0 (d, J = 22.0 Hz, 1C), 51.1, 27.3.



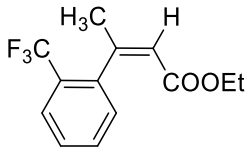
Z-3ae

Methyl (Z)-3-(thiophen-2-yl)but-2-enoate[8]

Yellow oil; 71% yield (646 mg); R_f = 0.30 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3029, 2899, 1758, 1722, 1231, 1146, 819, 661; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.479 (dd, J = 3.7, 1.2 Hz, 1H), 7.373 (dd, J = 5.1, 1.2 Hz, 1H),

7.021 (dd, *J* = 5.1, 3.7 Hz, 1H), 5.842 (d, *J* = 1.3 Hz, 1H), 3.674 (s, 3H), 2.276 (d, *J* = 1.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 166.5, 144.8, 140.2, 129.4, 127.8, 126.8, 116.0, 51.3, 27.6.

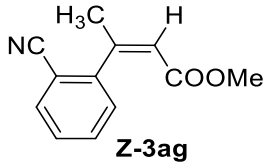


Z-3af

Ethyl (Z)-3-(2-(trifluoromethyl)phenyl)but-2-enoate

Yellow oil; 75% yield (968 mg); R_f = 0.29 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm^{-1}, KBr): 3011, 2891, 1761, 1710, 1221, 1130, 764, 701; ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.536 (d, *J* = 5.3 Hz, 1H), 7.427 (d, *J* = 5.0 Hz, 1H), 7.330 (t, *J* = 5.1 Hz, 1H), 7.202 (d, *J* = 5.1 Hz, 1H), 5.884 (d, *J* = 0.8 Hz, 1H), 4.012 (q, *J* = 4.7 Hz, 2H), 2.354 (s, 3H), 1.297 (d, *J* = 4.7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 165.8, 156.3, 155.1, 140.5 (d, *J* = 5.3 Hz, 1C), 131.3, 128.9, 128.6, 125.9 (q, *J* = 13.7 Hz, 1C), 120.1 (d, *J* = 4.1 Hz, 1C), 118.3, 111.4, 59.9, 27.4, 14.0. HRMS calcd for $C_{13}H_{13}F_3NaO_2^+$ $[M+Na]^+$ 281.0765, Found 281.0758.

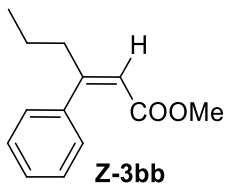


Z-3ag

Methyl (Z)-3-(2-cyanophenyl)but-2-enoate

Yellow oil; 71% yield (714 mg); R_f = 0.30 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3020, 2890, 1751, 1720, 1198, 1120, 771, 693; ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.720 (ddd, *J* = 8.1, 1.8, 0.8 Hz, 1H), 7.609 (dt, *J* = 7.9, 1.5 Hz, 1H), 7.365 (q, *J* = 6.3 Hz, 1H), 7.277 (td, *J* = 7.8, 1.8 Hz, 1H), 5.902 (s, 1H), 3.572 (s, 3H), 2.192 (s, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 166.3, 157.3, 136.8, 128.2, 126.5, 126.3, 126.1, 124.3, 118.3, 111.4, 51.2, 27.1. HRMS calcd for $C_{12}H_{12}NO_2^+$ $[M+H]^+$ 202.0868, Found 202.0866.

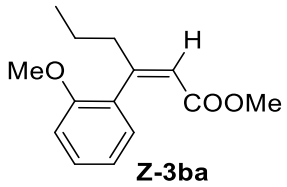


Z-3bb

Methyl (Z)-3-phenylhex-2-enoate

Yellow oil; 74% yield (755 mg); R_f = 0.24 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm^{-1}, KBr): 3033, 2948, 1749, 1710, 1233, 1109, 741, 689; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.369-7.307 (m, 3H), 7.168-7.144 (m, 2H), 5.885 (s, 1H), 3.544 (s, 3H), 2.445-2.405 (m, 2H), 1.415 (dd, J = 14.9, 7.4 Hz, 2H), 0.912 (t, J = 7.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 166.5, 160.3, 140.2, 127.9, 127.7, 127.1, 116.8, 51.1, 42.6, 20.6, 13.6.HRMS calcd for $C_{13}H_{16}NaO_2^+$ $[M+Na]^+$ 227.1048, Found 227.1045.

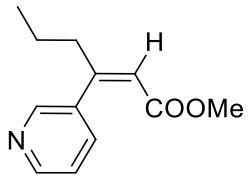


Z-3ba

Methyl (Z)-3-(2-methoxyphenyl)hex-2-enoate

Yellow oil; 72% yield (842 mg); R_f = 0.20 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3021, 2910, 1762, 1728, 1204, 1131, 752, 679; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.268-7.242 (m, 1H), 6.952 (ddd, J = 7.9, 7.3, 1.4 Hz, 2H), 6.893 (d, J = 8.3 Hz, 1H), 5.925 (s, 1H), 3.771 (s, 3H), 3.517 (s, 3H), 2.424-2.385 (m, 2H), 1.416 (dd, J = 15.0, 7.4 Hz, 2H), 0.904 (t, J = 7.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 166.3, 157.9, 155.7, 129.5, 128.8, 128.5, 120.3, 117.7, 110.8, 55.6, 50.6, 41.5, 20.6, 13.8.HRMS calcd for $C_{14}H_{18}NaO_3^+$ $[M+Na]^+$ 257.1154, Found 257.1150.

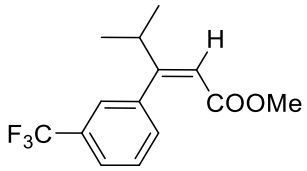


Z-3bh

Methyl (Z)-3-(pyridin-3-yl)hex-2-enoate

Yellow oil; 75% yield (768 mg); R_f = 0.28 (petroleum ether/ethyl acetate = 10:1, v/v).

IR (cm^{-1}, KBr): 3033, 2956, 1766, 1722, 1433, 1289, 1128, 680; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 8.684 (d, J = 0.9 Hz, 1H), 8.605-8.588 (m, 1H), 7.740-7.717 (m, 1H), 7.323 (dd, J = 5.7, 1.5 Hz, 1H), 6.057 (s, 1H), 3.758 (s, 3H), 3.088 (t, J = 7.7 Hz, 2H), 1.457 (dd, J = 15.2, 7.6 Hz, 2H), 0.936 (t, J = 8.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm)166.3, 157.4, 149.9, 147.8, 136.9, 134.0, 123.3, 118.5, 51.2, 32.6, 22.1, 13.9.HRMS calcd for $C_{12}H_{16}NO_2^+$ $[M+H]^+$ 206.1181, Found 206.1178.

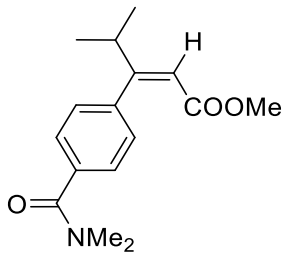


Z-3ci

Methyl (Z)-4-methyl-3-(3-(trifluoromethyl)phenyl)pent-2-enoate

Yellow oil; 80% yield (1088 mg); R_f = 0.3 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm^{-1}, KBr): 3029, 2899, 1768, 1720, 1281, 1170, 776, 683; ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.570 (ddd, J = 3.5, 1.5, 0.9 Hz, 1H), 7.489-7.449 (m, 1H), 7.353 (s, 1H), 7.300-7.281 (m, 1H), 5.939 (d, J = 1.2 Hz, 1H), 3.516 (s, 3H), 2.690-2.619 (m, 1H), 1.088 (d, J = 6.8 Hz, 6H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 166.4, 164.1, 140.9, 130.9 (d, J = 1.5 Hz, 1C), 130.3 (d, J = 32.0 Hz, 1C), 128.2, 125.6, 124.2 (q, J = 7.9 Hz, 1C), 122.9, 116.3, 51.2, 37.3, 21.1.HRMS calcd for $C_{14}H_{16}F_3O_2^+$ $[M+H]^+$ 273.1102, Found 273.1100.

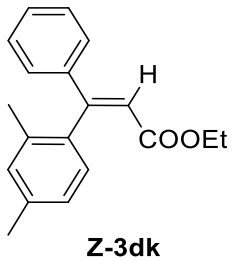


Z-3cj

Methyl (Z)-3-(4-(dimethylcarbamoyl)phenyl)-4-methylpent-2-enoate

Yellow solid; 70% yield (962.5 mg); R_f = 0.29 (petroleum ether/ethyl acetate = 3:1, v/v).

IR (cm^{-1}, KBr): 3018, 2860, 1766, 1723, 1630, 1319, 1277, 1163, 821, 692; ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.373-7.350 (m, 2H), 7.087 (dd, J = 7.9, 0.5 Hz, 2H), 5.838 (s, 1H), 3.474-3.470 (m, 3H), 3.019 (d, J = 35.2 Hz, 6H), 2.590 (hept, J = 6.8 Hz, 1H), 1.016 (dd, J = 6.8, 0.5 Hz, 6H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 171.5, 166.6, 165.0, 141.6, 135.2, 127.3, 126.7, 115.6, 51.1, 39.8, 37.4, 35.4, 21.1.HRMS calcd for $C_{16}H_{21}NNaO_3^+$ $[M+Na]^+$298.1419, Found 298.1416.

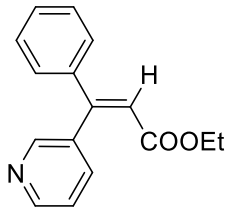


Z-3dk

Ethyl (Z)-3-(2,4-dimethylphenyl)-3-phenylacrylate

Yellow oil; 85% yield (1190 mg); R_f = 0.31 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm^{-1}, KBr): 3022, 2871, 1760, 1719, 1238, 1154, 870, 752, 701; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.354-7.299 (m, 5H), 7.061-7.036 (m, 2H), 6.958 (d, J = 7.6 Hz, 1H), 6.506 (s, 1H), 4.043 (q, J = 7.1 Hz, 2H), 2.367 (s, 3H), 2.046 (s, 3H), 1.118 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 166.0, 156.4, 139.8, 137.5, 135.8, 135.3, 130.8, 129.5, 128.7, 128.5, 127.6, 126.3, 117.7, 60.1, 21.4, 19.7, 14.2.HRMS calcd for $C_{19}H_{20}NaO_2^+$ $[M+Na]^+$ 303.1361, Found 303.1354.

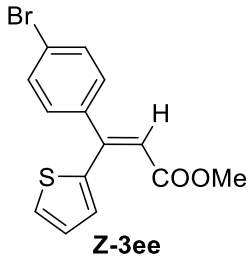


Z-3dh

Ethyl (Z)-3-phenyl-3-(pyridin-3-yl)acrylate

Yellow oil; 70% yield (885 mg); R_f = 0.22 (petroleum ether/ethyl acetate = 10:1, v/v).

IR (cm^{-1}, KBr): 3029, 2944, 1768, 1721, 1233, 1180, 742, 691; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 8.620 (dd, J = 4.8, 1.5 Hz, 1H), 8.478 (d, J = 1.7 Hz, 1H), 7.549-7.520 (m, 1H), 7.373-7.263 (m, 6H), 6.458 (s, 1H), 4.063 (q, J = 7.1 Hz, 2H), 1.127 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 165.7, 153.0, 149.8, 149.2, 140.0, 136.7, 134.9, 129.9, 128.7, 128.2, 122.8, 119.0, 60.4, 14.1.HRMS calcd for $C_{16}H_{16}NO_2^+$ $[M+H]^+$ 254.1181, Found 254.1180.

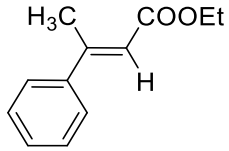


Z-3ee

Methyl (Z)-3-(4-bromophenyl)-3-(thiophen-2-yl)acrylate

Yellow oil; 75% yield (1211 mg); R_f = 0.36 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3026, 2913, 1763, 1728, 1288, 1162, 868, 692; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm)7.668-7.637 (m, 2H), 7.456 (dd, J = 5.1, 1.2 Hz, 1H), 7.355 (dd, J = 5.1, 1.2 Hz, 1H), 7.284 (dd, J = 5.2, 3.6 Hz, 1H), 7.096-7.060 (m, 2H), 6.224 (s, 1H), 3.698 (s, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 166.3, 148.4, 143.5, 140.6, 135.7, 129.2, 128.3, 126.9, 125.7, 123.8, 117.6, 51.6. HRMS calcd for $C_{14}H_{11}BrNaO_2S^+$ $[M+Na]^+$ 344.9561, Found 344.9552.

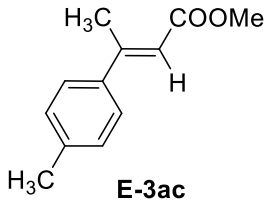


E-3ab

Ethyl (E)-3-phenylbut-2-enoate[3]

Yellow oil; 83% yield by method A (788 mg); 86% yield by method B (817 mg); R_f = 0.30 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm^{-1}, KBr): 3026, 2889, 1751, 1722, 1248, 1177, 966, 751; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.491-7.467 (m, 2H), 7.388-7.351 (m, 3H), 6.141 (d, J = 1.3 Hz, 1H), 4.222 (q, J = 7.1 Hz, 2H), 2.584 (d, J = 1.3 Hz, 3H), 1.322 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 166.9, 155.6, 142.4, 129.0, 128.6, 126.4, 117.3, 59.9, 18.0, 14.4.

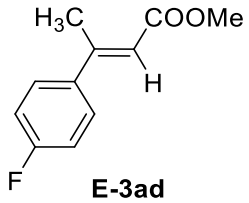


E-3ac

Methyl (E)-3-(p-tolyl)but-2-enoate[3]

Yellow oil; 74%yield by method A (703 mg); 82% yield by method B (779 mg); R_f = 0.31 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm^{-1}, KBr): 3031, 2892, 1783, 1723, 1244, 1166, 962, 842; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.387 (d, J = 8.2 Hz, 2H), 7.185 (d, J = 8.5 Hz, 2H), 6.167 (s, 1H), 3.752 (s, 3H), 2.574 (d, J = 1.3 Hz, 3H), 2.371 (s, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 167.5, 155.9, 139.3, 129.3, 126.3, 115.9, 51.1, 21.3, 17.9.

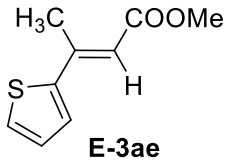


E-3ad

Methyl (E)-3-(4-fluorophenyl)but-2-enoate[7]

Yellow oil; 75% yield by method A (727 mg); 80% yield by method B (776 mg); R_f = 0.28 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm^{-1}, KBr): 3032, 2910, 1760, 1722, 1245, 1155, 976, 827; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.454 (dd, J = 9.0, 5.3 Hz, 2H), 7.074-7.031 (m, 2H), 6.092 (d, J = 1.3 Hz, 1H), 3.748 (s, 3H), 2.556 (d, J = 1.3 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 167.2, 163.3 (d, J = 248.1 Hz, 1C), 154.7, 138.22 (d, J = 3.4 Hz, 1C), 128.19 (d, J = 8.3 Hz, 1C), 116.69 (d, J = 1.5 Hz, 1C), 115.5 (d, J = 21.2 Hz, 1C), 51.2, 18.1.

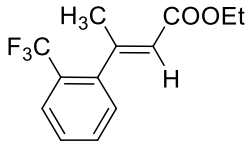


E-3ae

Methyl (E)-3-(thiophen-2-yl)but-2-enoate[8]

Yellow oil; 70% yield by method A (637 mg); 76% yield by method B (692 mg); R_f = 0.33 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3022, 2896, 1752, 1711, 1233, 1144, 960, 817; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.288 (d, J = 4.2 Hz, 2H), 7.012 (dd, J = 4.7, 4.1 Hz, 1H), 6.242 (d, J = 1.2 Hz, 1H), 3.716 (s, 3H), 2.587 (d, J = 1.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 167.2, 148.1, 145.6, 128.0, 127.2, 126.9, 113.9, 51.2, 17.4.

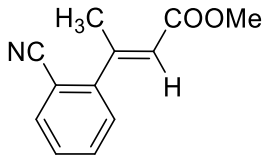


E-3af

Ethyl (E)-3-(2-(trifluoromethyl)phenyl)but-2-enoate

Yellow oil; 77% yield by method A (993 mg); 82% yield by method B (1058 mg); R_f = 0.34 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm^{-1}, KBr): 3013, 2893, 1762, 1712, 1221, 1130, 867, 764; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.677-7.656 (m, 1H), 7.533 (ddd, J = 7.6, 3.8, 0.6 Hz, 1H), 7.439-7.410 (m, 1H), 7.223-7.203 (m, 1H), 5.768 (d, J = 1.4 Hz, 1H), 4.217 (q, J = 7.1 Hz, 2H), 2.468-2.465 (m, 3H), 1.309 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 168.2, 166.2, 162.4, 155.1, 142.8 (d, J = 2.3 Hz, 1C), 137.0, 132.5(d, J = 148.0 Hz, 1C), 128.9 (d, J = 86.5 Hz, 1C), 126.3 (d, J = 5.0 Hz, 1C), 120.44 (q, J = 2.1 Hz, 1C), 60.1, 21.3, 14.3.HRMS calcd for $C_{13}H_{14}F_3O_2^+$ $[M+H]^+$ 259.0946, Found 259.0938.

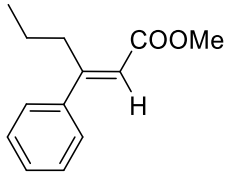


E-3ag

Methyl (E)-3-(2-cyanophenyl)but-2-enoate

Yellow oil; 68% yield by method A (683 mg); 70% yield by method B (704 mg); R_f = 0.35 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3022, 2889, 1765, 1720, 1179, 1123, 871, 771; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.644 (ddd, J = 8.1, 1.8, 0.8 Hz, 1H), 7.539 (dt, J = 7.9, 1.5 Hz, 1H), 7.355 (t, J = 4.0 Hz, 1H), 7.288 (q, J = 6.1 Hz, 1H), 6.124 (s, 1H), 3.763 (s, 3H), 2.573 (s, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 167.5, 155.8, 139.3, 129.2, 128.3, 127.2, 126.3, 126.2, 116.4, 111.3, 51.2, 17.3.HRMS calcd for $C_{12}H_{12}NO_2^+$ $[M+H]^+$ 202.0868, Found 202.0867.

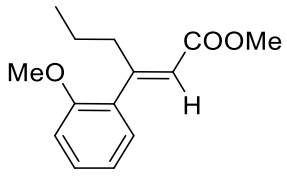


E-3bb

Methyl (E)-3-phenylhex-2-enoate[9]

Yellow oil; 74% yield by method A (755 mg); 80% yield by method B (816 mg); R_f = 0.30 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm^{-1}, KBr): 3032, 2945, 1721, 1707, 1231, 1109, 877, 741; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.443-7.418 (m, 2H), 7.378-7.350 (m, 2H), 6.051 (s, 1H), 3.747 (s, 3H), 3.101 (dd, J = 8.6, 6.9 Hz, 2H), 1.518-1.424 (m, 2H), 0.946 (t, J = 7.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 167.0, 161.2, 141.5, 128.9, 128.6, 126. 8, 117.1, 51.1, 32.9, 22.3, 14.1.

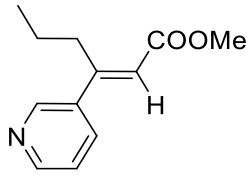


E-3ba

Methyl (E)-3-(2-methoxyphenyl)hex-2-enoate

Yellow oil; 73% yield by method A (854 mg); 78% yield by method B (913 mg); R_f = 0.31 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3021, 2913, 1766, 1728, 1204, 1131, 869, 752; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.304-7.260 (m, 1H), 7.086 (dd, J = 7.4, 1.8 Hz, 1H), 6.927 (dt, J = 7.5, 3.7 Hz, 1H), 6.881 (d, J = 8.3 Hz, 1H), 5.819 (s, 1H), 3.798 (s, 3H), 3.719 (s, 3H), 3.039-3.000 (m, 2H), 1.382-1.325 (m, 2H), 0.880 (t, J = 7.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 167.0, 161.6, 156.4, 131.8, 129.5, 129.4, 120.5, 118.9, 110.9, 55.5, 51.1, 34.1, 21.8, 14.2.HRMS calcd for $C_{14}H_{18}NaO_3^+$ $[M+Na]^+$ 257.1154, Found 257.1150.



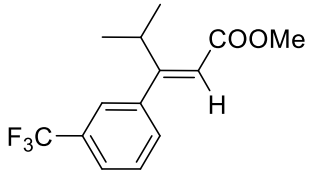
E-3bh

Methyl (E)-3-(pyridin-3-yl)hex-2-enoate

Yellow oil; 77% yield by method A (789 mg); 81% yield by method B (830 mg); R_f = 0.27 (petroleum ether/ethyl acetate = 10:1, v/v).

IR (cm^{-1}, KBr): 3033, 2953, 1732, 1718, 1430, 1286, 1128, 879; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 8.472 (dt, J = 4.8, 1.6 Hz, 1H), 8.335 (s, 1H), 7.442-7.418 (m, 1H), 7.217-7.192 (m, 1H), 5.904 (s, 1H), 3.478 (s, 3H), 2.358 (td, J = 7.8, 1.3 Hz, 2H), 1.373-

1.314 (m, 2H), 0.862-0.822 (m, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 165.9, 156.4, 148.7, 147.9, 135.7, 134.7, 122.7, 118.4, 51.1, 42.2, 20.4, 13.4.HRMS calcd for $C_{12}H_{16}NO_2^+$ $[M+H]^+$ 206.1181, Found 206.1178.

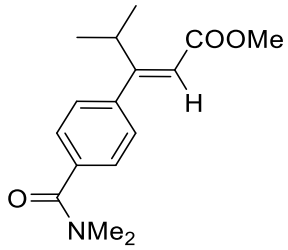


E-3ci

Methyl (E)-4-methyl-3-(3-(trifluoromethyl)phenyl)pent-2-enoate

Yellow oil; 81% yield by method A (1101 mg); 86% yield by method B (1034 mg); R_f = 0.36 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm^{-1}, KBr): 3032, 2899, 1761, 1720, 1281, 1170, 877, 776; ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.589 (d, J = 7.8 Hz, 1H), 7.481-7.440 (m, 2H), 7.381 (d, J = 7.7 Hz, 1H), 5.709 (s, 1H), 4.137 (dq, J = 13.7, 6.8 Hz, 1H), 3.751 (s, 3H), 1.081 (d, J = 7.0 Hz, 6H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 166.3, 165.6, 141.4, 131.1, 128.4, 124.5 (d, J = 2.9 Hz, 1C), 119.2, 100.0, 51.3, 29.6, 21.3.HRMS calcd for $C_{14}H_{16}F_3O_2^+$ $[M+H]^+$ 273.1102, Found 273.1100.

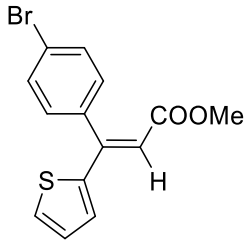


E-3cj

Methyl (E)-3-(4-(dimethylcarbamoyl)phenyl)-4-methylpent-2-enoate

Yellow oil; 71% yield by method A (976 mg); 71% yield by method B (976 mg); R_f = 0.32 (petroleum ether/ethyl acetate = 3:1, v/v).

IR (cm^{-1}, KBr): 3019, 2877, 1769, 1722, 1639, 1316, 1273, 1163, 866, 821; ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.392-7.370 (m, 2H), 7.233-7.204 (m, 2H), 5.686 (d, J = 0.5 Hz, 1H), 4.162-4.058 (m, 1H), 3.731 (s, 3H), 3.053 (s, 6H), 1.072 (d, J = 7.0 Hz, 6H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 171.3, 166.6, 166.5, 142.0, 135.7, 127.8, 126.7, 118.5, 51.2, 39.7, 35.5, 29.6, 21.4.HRMS calcd for $C_{16}H_{22}NO_3^+$ $[M+H]^+$ 276.1600, Found 276.1591.

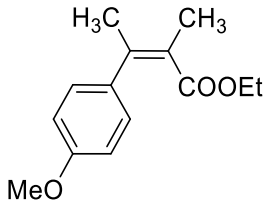


E-3ee

Methyl (E)-3-(4-bromophenyl)-3-(thiophen-2-yl)acrylate

Yellow oil; 76% yield by method A (1227 mg); 78% yield by method B (1260 mg); R_f =0.39 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3020, 2918, 1760, 1720, 1285, 1162, 877, 868; ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.560-7.539 (m, 2H), 7.378 (dd, J = 5.1, 1.2 Hz, 1H), 7.173-7.151 (m, 2H), 6.984 (dd, J = 5.1, 3.8 Hz, 1H), 6.842 (dd, J = 3.7, 1.2 Hz, 1H), 6.409 (s, 1H), 3.612 (s, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 165.9, 149.4, 144.5, 137.0, 131.3, 130.41, 130.36, 128.6, 128.1, 122.7, 114.4, 51.4.HRMS calcd for $C_{14}H_{11}BrNaO_2S^+$ $[M+Na]^+$ 344.9561, Found 344.9553.

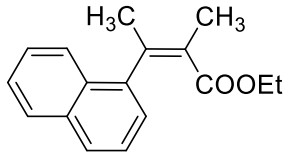


Z-4al

Ethyl (Z)-3-(4-methoxyphenyl)-2-methylbut-2-enoate[10]

Yellow oil; 67% yield (784 mg); R_f = 0.23 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3018, 2950, 2825, 1766, 1721, 1127, 860; ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.074 (d, J = 8.8 Hz, 2H), 6.822 (d, J = 8.8 Hz, 2H), 3.886 (q, J = 7.1 Hz, 2H), 3.787 (s, 3H), 2.063 (d, J = 1.1 Hz, 3H), 2.004 (d, J = 1.1 Hz, 3H), 0.900 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 171.0, 158.8, 142.2, 136.5, 128.2 125.8, 113.5, 60.2, 55.3, 21.7, 16.6, 13.8.



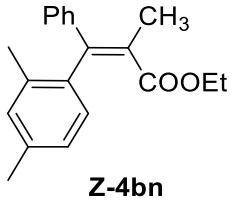
Z-4am

Ethyl (Z)-2-methyl-3-(naphthalen-1-yl)but-2-enoate

Yellow oil; 66% yield (838 mg); R_f = 0.22 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm^{-1}, KBr): 3010, 2980, 1788, 1723, 1129, 760; ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.873-7.849 (m, 1H), 7.783-7.742 (m, 2H), 7.487-7.442 (m, 3H), 7.188 (dd, J =

7.0, 1.2 Hz, 1H), 4.309 (q, *J* = 7.1 Hz, 2H), 2.345 (s, 3H), 1.571 (s, 3H), 1.376 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 169.6, 145.0, 141.5, 133.8, 129.8, 128.5, 127.3, 126.7, 126.4, 126.0, 125.7, 125.1, 124.0, 60.5, 23.5, 17.4, 14.5.HRMS calcd for $C_{17}H_{18}NaO_2^+$ $[M+Na]^+$ 277.1204, Found 277.1201.

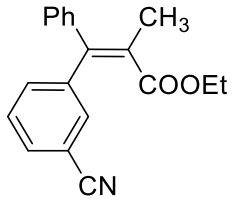


Z-4bn

Ethyl (Z)-3-(2,4-dimethylphenyl)-2-methyl-3-phenylacrylate

Yellow oil; 77% yield (1132 mg); R_f = 0.31 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3021, 2868, 1760, 1719, 1234, 1154, 873, 751;^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm)7.210-7.174 (m, 3H), 7.180 (dd, *J* = 7.7, 1.8 Hz, 2H), 7.029-6.984 (m, 3H), 3.973 (qd, *J* = 7.1, 1.4 Hz, 2H), 2.305 (s, 3H), 2.099 (s, 3H), 1.819 (s, 3H), 0.925 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 171.3, 145.8, 141.6, 137.5, 137.3, 135.5, 131.2, 129.4, 129.0, 128.8, 128.3, 127.9, 127.2, 126.6, 60.6, 21.2, 19.6, 18.2, 13.7.HRMS calcd for $C_{20}H_{22}NaO_2^+$ $[M+Na]^+$ 317.1517, Found 317.1510.

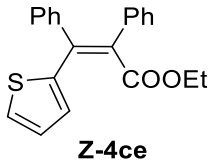


Z-4bo

Ethyl (Z)-3-(3-cyanophenyl)-2-methyl-3-phenylacrylate

Yellow oil; 65% yield (946 mg); R_f =0.32 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3089, 2879, 1762, 1720, 1179, 1122, 781;^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.446-7.394 (m, 5H), 7.343 (d, *J* = 7.6 Hz, 1H), 7.302 (dd, *J* = 10.3, 4.7 Hz, 2H), 7.238 (d, *J* = 4.2 Hz, 1H), 3.856 (q, *J* = 3.9 Hz, 2H), 2.212 (s, 3H), 1.055 (t, *J* = 3.9 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 169.3, 145.8, 144.6, 132.5, 128.4, 127.6, 126.6, 126.4, 126.2, 126.1, 125.4, 122.5, 122.1, 111.6, 60.1, 27.5, 13.7.HRMS calcd for $C_{19}H_{18}NO_2^+$ $[M+H]^+$ 292.1337, Found 292.1339.

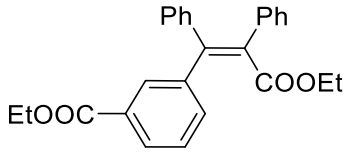


Z-4ce

Ethyl (Z)-2,3-diphenyl-3-(thiophen-2-yl)acrylate

Yellow oil; 71% yield (1186 mg); R_f = 0.31 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3042, 3011, 2881, 1769, 1720, 1179, 1123, 772, 741;^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.372-7.340 (m, 3H), 7.181-7.094 (m, 9H), 6.992 (dd, J = 5.1, 3.6 Hz, 1H), 4.175 (q, J = 7.1 Hz, 2H), 1.148 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 170.5, 144.4, 140.2, 136.9, 130.9, 130.0, 129.7, 129.4, 128.9, 128.6, 128.2, 128.1, 127.9, 127.6, 127.4, 126.9, 100.0, 61.4, 13.9. HRMS calcd for $C_{21}H_{18}NaO_2S^+$ $[M+Na]^+$ 357.0925, Found 357.0920.

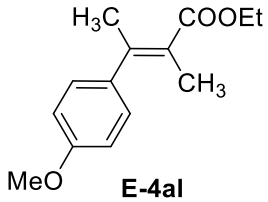


Z-4cp

Ethyl (Z)-3-(3-ethoxy-3-oxo-1,2-diphenylprop-1-en-1-yl)benzoate

Yellow oil; 71% yield (1420 mg); R_f = 0.34 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3043, 3011, 2881, 1769, 1720, 1179, 1123, 772, 741; ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm) 8.313 (s, 2H), 8.066 (d, J = 7.7 Hz, 2H), 7.969 (d, J = 7.6 Hz, 1H), 7.810 (d, J = 7.6 Hz, 2H), 7.536 (t, J = 7.7 Hz, 3H), 7.439-7.408 (m, 2H), 7.376-7.266 (m, 2H), 4.429 (q, J = 7.1 Hz, 4H), 1.429 (t, J = 7.1 Hz, 6H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 168.9, 166.5, 140.6, 133.6, 131.59, 131.56, 130.3, 129.7, 129.3, 129.04, 128.99, 128.89, 128.85, 128.8, 128.5, 128.3, 128.1, 128.0, 61.2, 60.7, 14.4, 14.1.HRMS calcd for $C_{26}H_{25}O_4^+$ $[M+H]^+$ 401.1753, Found 401.1751.

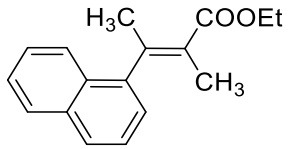


E-4al

Ethyl (E)-3-(4-methoxyphenyl)-2-methylbut-2-enoate[10]

Yellow oil; 70% yield (819 mg); R_f = 0.28 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3029, 2951, 2825, 1762, 1722, 1134, 862;^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.085-7.070 (m, 2H), 6.887-6.873 (m, 2H), 4.247 (q, J = 4.7 Hz, 2H), 3.809 (s, 3H), 2.225 (s, 3H), 1.771 (s, 3H), 1.331 (d, J = 4.8 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 170.3, 158.7, 145, 135.8, 128.7, 124.8, 113.7, 60.4, 55.3, 23.3, 17.5, 14.4.

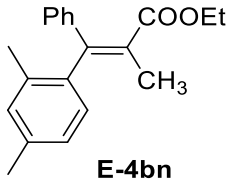


E-4am

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

Yellow oil; 68% yield (864 mg); R_f = 0.27 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm^{-1}, KBr): 3023, 2989, 1783, 1726, 1139, 758;^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.826 (dd, *J* = 6.2, 3.3 Hz, 1H), 7.761 (dd, *J* = 6.1, 3.5 Hz, 1H), 7.733 (d, *J* = 8.2 Hz, 1H), 7.447 (dd, *J* = 6.3, 3.3 Hz, 2H), 7.392 (dd, *J* = 8.0, 7.2 Hz, 1H), 7.135 (d, *J* = 6.9 Hz, 1H), 3.629-3.538 (m, 2H), 2.193 (s, 3H), 2.166 (s, 3H), 0.375 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 169.4, 143.4, 142.8, 133.6, 130.9, 128.3, 127.7, 126.9, 125.9, 125.7, 125.5, 125.4, 123.6, 59.8, 22.8, 15.9, 13.1.HRMS calcd for $C_{17}H_{18}NaO_2^+$ $[M+Na]^+$ 277.1204, Found 277.1202.

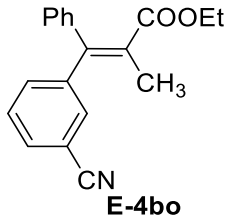


E-4bn

Ethyl (E)-3-(2,4-dimethylphenyl)-2-methyl-3-phenylacrylate

Yellow oil; 76% yield (1117 mg); R_f =0.33 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3018, 2865, 1766, 1713, 1230, 1152, 870, 748;^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.313-7.275 (m, 2H), 7.246 (dd, J = 5.1, 3.6 Hz, 1H), 7.198-7.174 (m, 2H), 6.995 (d, J = 7.5 Hz, 1H), 6.928 (d, J = 8.3 Hz, 2H), 3.874 (q, J = 7.1 Hz, 2H), 2.287 (s, 3H), 2.096 (s, 3H), 2.044 (s, 3H), 0.843 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm)170.7, 146.8, 140.1, 139.5, 137.0, 135.8, 130.8, 129.4, 128.8, 128.7, 128.0, 127.5, 126.0, 60.3, 21.2, 19.9, 17.7, 13.5.HRMS calcd for $C_{20}H_{22}NaO_2^+$ $[M+Na]^+$ 317.1517, Found 317.1509.

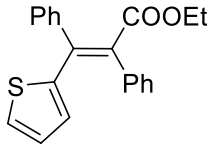


E-4bo

Ethyl (E)-3-(3-cyanophenyl)-2-methyl-3-phenylacrylate

Yellow oil; 66% yield (960 mg); R_f = 0.36 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3086, 2873, 1764, 1718, 1176, 1122, 779; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.407-7.356 (m, 5H), 7.305 (dt, J = 13.3, 7.7 Hz, 3H), 7.220 (s, 1H), 4.042 (q, J = 4.3 Hz, 2H), 2.100 (s, 3H), 1.041 (t, J = 4.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 170.3, 145.7, 144.6, 132.6, 128.3, 127.7, 126.6, 126.4, 126.2, 126.1, 125.3, 122.3, 122.2, 111.6, 59.3, 26.4, 14.1.HRMS calcd for $C_{19}H_{18}NO_2^+$ $[M+H]^+$ 292.1337, Found 292.1339.

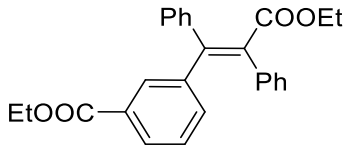


E-4ce

Ethyl (E)-2,3-diphenyl-3-(thiophen-2-yl)acrylate

Yellow oil; 69% yield (1152 mg); R_f =0.33 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3048, 3012, 2880, 1769, 1720, 1176, 1123, 778, 739; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.385-7.354 (m, 5H), 7.352-7.323 (m, 5H), 7.177 (dd, J = 5.1, 1.2 Hz, 1H), 6.768 (dd, J = 5.1, 3.7 Hz, 1H), 6.557 (dd, J = 3.7, 1.2 Hz, 1H), 3.895 (q, J = 7.1 Hz, 2H), 0.862 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 169.5, 143.0, 141.8, 139.7, 137.4, 131.4, 130.9, 130.0, 129.4, 128.9, 128.5, 128.3, 128.1, 126.3, 100.0, 60.9, 13.7.HRMS calcd for $C_{21}H_{18}NaO_2S^+$ $[M+Na]^+$ 357.0925, Found 357.0921.

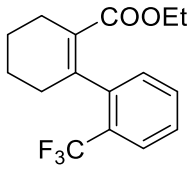


E-4cp

Ethyl (E)-3-(3-ethoxy-3-oxo-1,2-diphenylprop-1-en-1-yl)benzoate

Yellow oil; 72% yield (1440 mg); R_f =0.36 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3043, 3010, 2871, 1766, 1720, 1179, 1113, 769, 731;^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 8.023 (s, 1H), 7.693 (d, J = 7.0 Hz, 1H), 7.452-7.266 (m, 4H), 7.150 (dd, J = 16.3, 6.5 Hz, 7H), 6.993 (d, J = 6.6 Hz, 1H), 4.392 (q, J = 6.9 Hz, 2H), 4.037 (q, J = 6.9 Hz, 2H), 1.382 (t, J = 7.1 Hz, 3H), 1.007 (t, J = 5.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 170.1, 166.4, 144.9, 137.3, 137.2, 135.2, 133.7, 132.0, 130.9, 130.1, 130.0, 129.9, 129.2, 128.4, 128.34, 128.29, 128.0, 127.9, 61.1, 61.0, 14.4, 13.8.HRMS calcd for $C_{26}H_{25}O_4^+$ $[M+H]^+$ 401.1753, Found 401.1754.

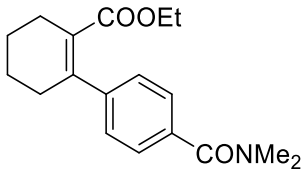


5a

Ethyl 2'-(trifluoromethyl)-3,4,5,6-tetrahydro-[1,1'-biphenyl]-2-carboxylate

Yellow oil; 79% yield by method A (1177 mg); 70% yield by method B (1043mg); R_f = 0.33 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm^{-1}, KBr): 3029, 2896, 1759, 1720, 1277, 1167, 873, 776; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.613 (d, J = 7.9 Hz, 1H), 7.485 (t, J = 7.5 Hz, 1H), 7.351 (t, J = 7.7 Hz, 1H), 7.120 (d, J = 7.7 Hz, 1H), 3.824 (qd, J = 7.1, 1.3 Hz, 2H), 2.502-2.269 (m, 4H), 1.732 (dd, J = 6.9, 4.4 Hz, 4H), 0.797 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 167.5, 146.3, 143.4 (d, J = 2.4 Hz, 1C), 131.3, 128.8, 127.8, 126.5, 125.9 (q, J = 49.6 Hz, 1C), 125.3, 123.5, 59.8, 34.6, 26.0, 22.3, 22.0, 13.5.HRMS calcd for $C_{16}H_{17}F_3NaO_2^+$ $[M+Na]^+$ 321.1078, Found 321.1072.

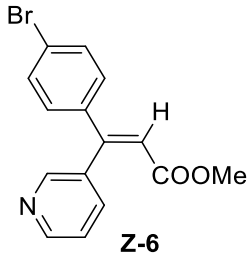


5b

Ethyl 4'-(dimethylcarbamoyl)-3,4,5,6-tetrahydro-[1,1'-biphenyl]-2-carboxylate

Yellow oil; 73% yield by method A (1099 mg); 66% yield by method B (993 mg); R_f = 0.23 (petroleum ether/ethyl acetate = 3:1, v/v).

IR (cm^{-1}, KBr): 3022, 2893, 1769, 1720, 1644, 1267, 1162, 863, 767; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.358-7.330 (m, 2H), 7.160-7.132 (m, 2H), 3.872-3.846 (m, 2H), 3.032 (d, J = 36.7 Hz, 6H), 2.408-2.333 (m, 4H), 1.723 (d, J = 3.0 Hz, 4H), 0.877-0.833 (m, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 171.5, 169.7, 145.1, 144.8, 134.8, 128.6, 127.0, 126.9, 60.2, 39.7, 35.4, 32.6, 26.7, 22.5, 21.9, 13.7.HRMS calcd for $C_{18}H_{23}NNaO_3^+$ $[M+Na]^+$324.1576, Found 324.1573.

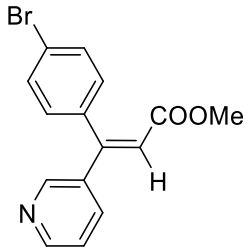


Z-6

Methyl (Z)-3-(4-bromophenyl)-3-(pyridin-3-yl)acrylate

Yellow oil; 76% yield (1205 mg); R_f = 0.24 (petroleum ether/ethyl acetate = 10:1, v/v).

IR (cm^{-1}, KBr): 3030, 2950, 1735, 1716, 1430, 1286, 1128, 840; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 8.631 (dd, J = 4.9, 1.7 Hz, 1H), 8.442 (dd, J = 2.2, 0.8 Hz, 1H), 7.536-7.526 (m, 1H), 7.489-7.478 (m, 1H), 7.466-7.455 (m, 1H), 7.337 (ddd, J = 7.8, 4.9, 0.9 Hz, 1H), 7.142-7.120 (m, 2H), 6.430 (s, 1H), 3.618 (s, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 165.8, 152.3, 149.7, 149.5, 138.9, 136.7, 134.2, 132.0, 129.8, 124.6, 123.0, 118.8, 51.5.HRMS calcd for $C_{15}H_{13}BrNO_2^+$ $[M+H]^+$ 318.0130, Found 318.0121.

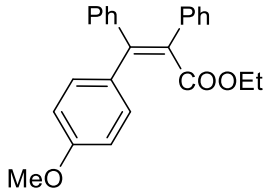


E-6

Methyl (E)-3-(4-bromophenyl)-3-(pyridin-3-yl)acrylate

Yellow oil; 78% yield (1236 mg); R_f = 0.28 (petroleum ether/ethyl acetate = 10:1, v/v).

IR (cm^{-1}, KBr): 3033, 2953, 1732, 1718, 1430, 1286, 1125, 876; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 8.607 (dd, J = 5.6, 1.8 Hz, 2H), 7.537 (ddd, J = 6.4, 4.5, 1.3 Hz, 3H), 7.283 (d, J = 5.1 Hz, 1H), 7.113-7.092 (m, 2H), 6.406 (s, 1H), 3.661 (s, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 165.7, 152.7, 150.6, 149.0, 136.6, 136.2, 135.7, 131.5, 130.9, 123.4, 123.2, 118.7, 51.6.HRMS calcd for $C_{15}H_{13}BrNO_2^+$ $[M+H]^+$318.0130, Found 318.0125.

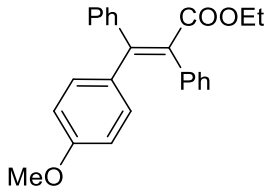


Z-7

Ethyl (Z)-3-(4-methoxyphenyl)-2,3-diphenylacrylate[11]

Yellow oil; 76% yield (1360 mg); R_f = 0.30 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3023, 2954, 2821, 1759, 1719, 1130, 861, 749; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.223-7.201 (m, 2H), 7.174-7.091 (m, 8H), 7.015 (ddd, J = 7.2, 2.0, 0.8 Hz, 2H), 6.868-6.846 (m, 2H), 4.081 (q, J = 7.1 Hz, 2H), 3.822 (s, 3H), 1.048 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 170.9, 159.7, 145.7, 141.0, 137.9, 135.0, 133.0, 131.1, 130.6, 130.0, 128.2, 127.9, 127.7, 127.3, 113.6, 61.0, 55.4, 14.0.

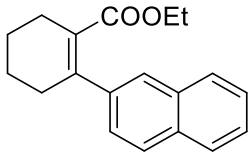


E-7

Ethyl (E)-3-(4-methoxyphenyl)-2,3-diphenylacrylate[11]

Yellow oil; 75% yield (1342 mg); R_f = 0.35 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3030, 2954, 2824, 1759, 1729, 1130, 861, 746; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.318 (dd, J = 4.4, 2.0 Hz, 3H), 7.281-7.260 (m, 2H), 7.201-7.170 (m, 3H), 7.139-7.123 (m, 2H), 6.913-6.899 (m, 2H), 6.654-6.640 (m, 2H), 3.997 (q, J = 7.1 Hz, 2H), 3.737 (s, 3H), 0.941 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 170.8, 159.2, 146.0, 143.0, 138.1, 133.0, 132.8, 132.5, 130.0, 129.3, 128.3, 128.1, 128.1, 127.3, 113.3, 60.9, 55.2, 13.8.

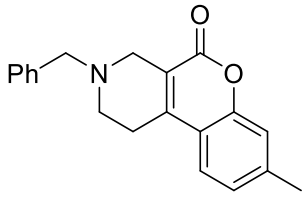


8

Ethyl 2-(naphthalen-2-yl)cyclohex-1-ene-1-carboxylate

Yellow oil; 77% yield (1078 mg); R_f =0.37 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm^{-1}, KBr): 3028, 2976, 1780, 1736, 1137, 758; ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.856-7.804 (m, 2H), 7.766-7.745 (m, 1H), 7.471-7.413 (m, 3H), 7.180 (dd, J = 7.0, 1.0 Hz, 1H), 3.680-3.580 (m, 2H), 2.615-2.405 (m, 4H), 1.922-1.833 (m, 4H), 0.411 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 168.7, 146.2, 142.2, 133.6, 131.0, 129.4, 128.3, 126.8, 125.9, 125.7, 125.4, 125.2, 123.6, 59.7, 34.1, 26.3, 22.7, 22.4, 13.2; HRMS calcd for $C_{19}H_{20}NaO_2^+$ $[M+Na]^+$303.1361, Found 303.1356.



9

1,2,3,4-Tetrahydro-8-methyl-3-(phenylmethyl)-5H-[1]benzopyrano[3,4-c] pyridin-5-one[12]

Yellow solid; 70% yield (1068 mg); R_f =0.3 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm^{-1}, KBr): 3021, 2981, 1786, 1726, 1619, 1453, 1139, 758;^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.586 (d, J = 7.4 Hz, 1H), 7.364-7.193 (m, 7H), 3.710 (s, 2H), 3.266 (s, 2H), 2.898 (t, J = 14.8 Hz, 2H), 2.750 (t, J = 14.6 Hz, 2H), 2.406 (s, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz,) δ (ppm) 170.1, 167.1, 156.2, 144.7, 138.4, 129.4, 129.1, 128.6, 128.4, 128.0, 127.2, 121.1, 111.6, 60.4, 59.6, 56.3, 28.8, 20.1.

9. References

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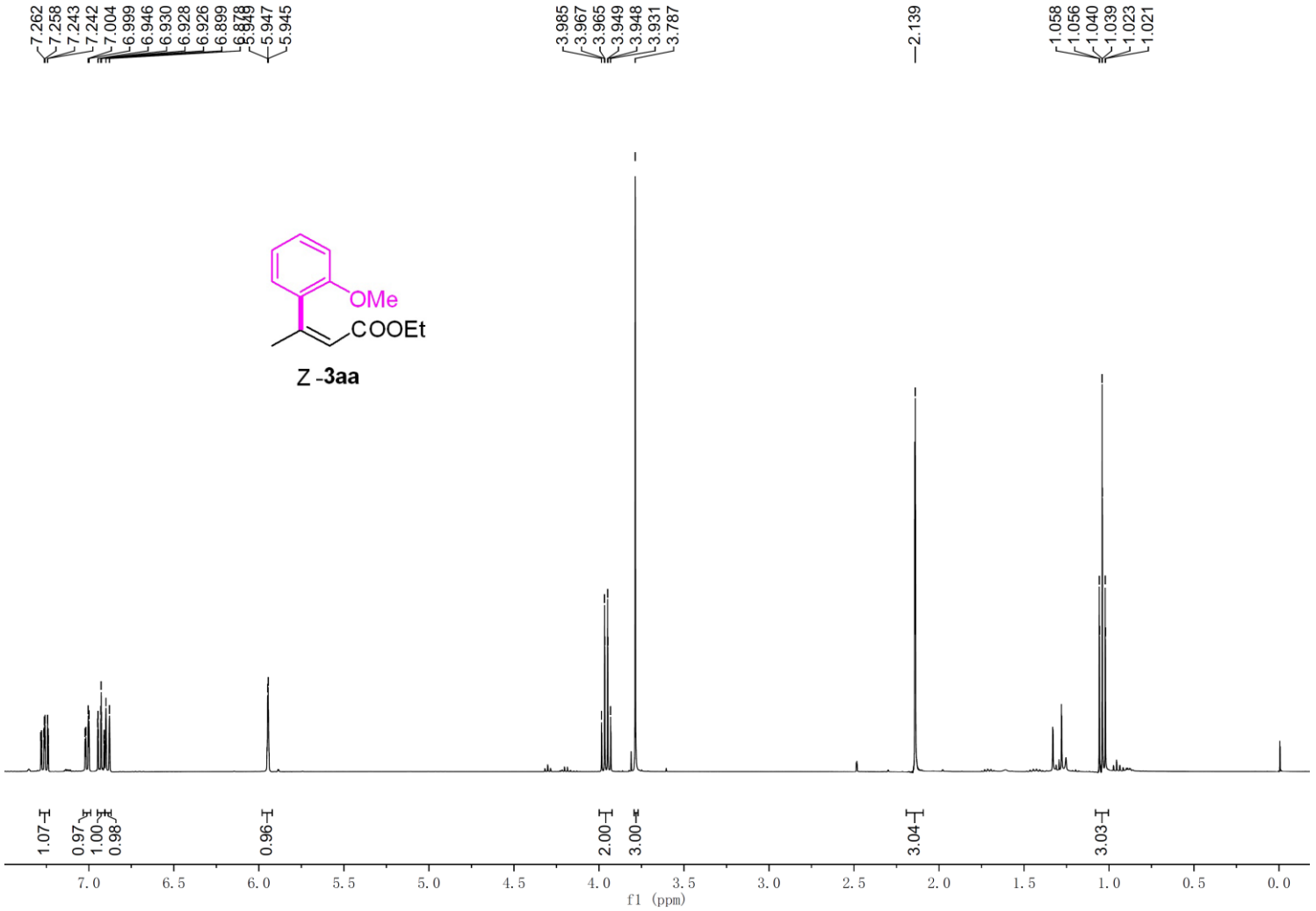
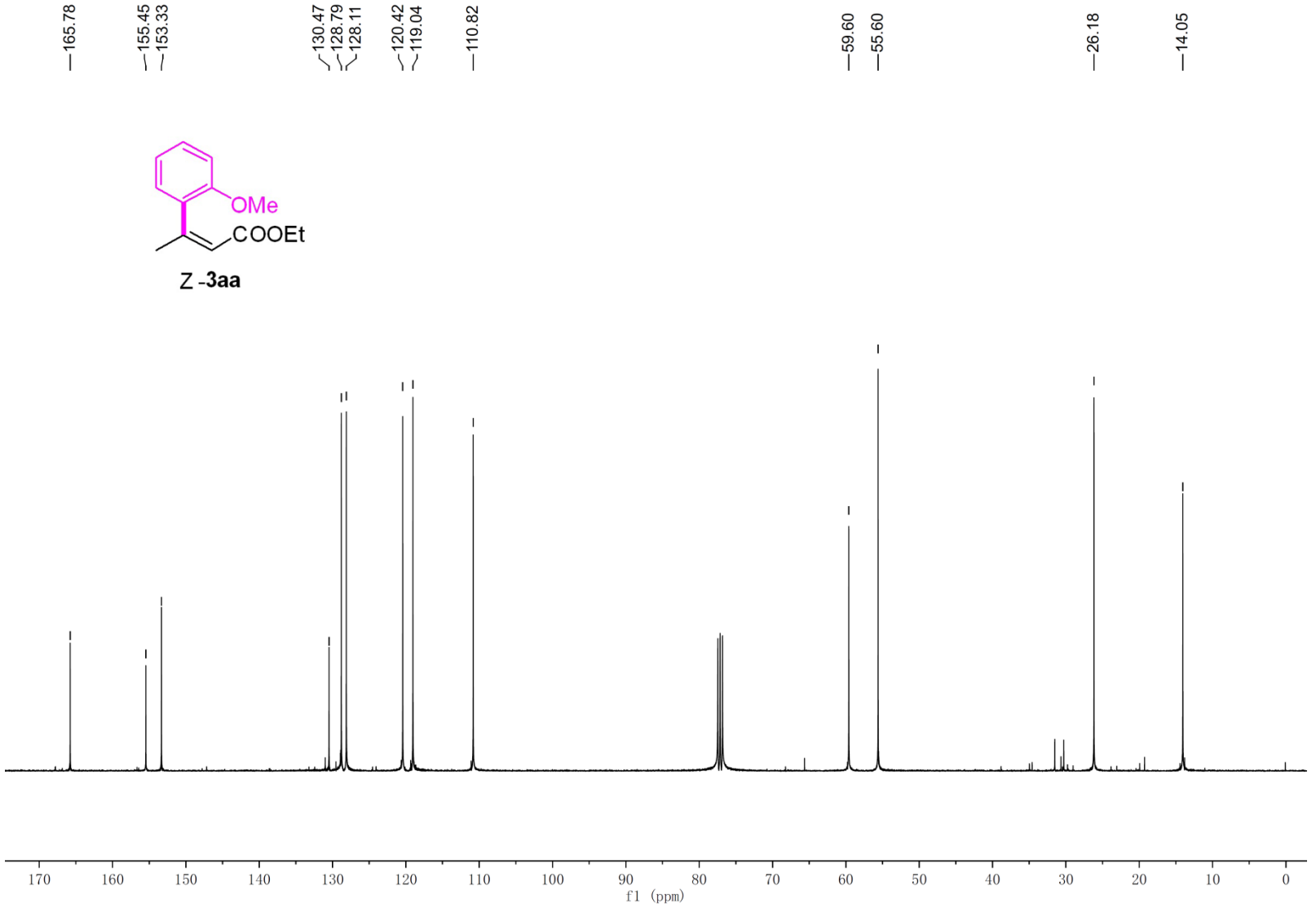
10. ^{1}H and ^{13}C NMR spectra for products

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OMe
COOEt
Z -3aa
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50
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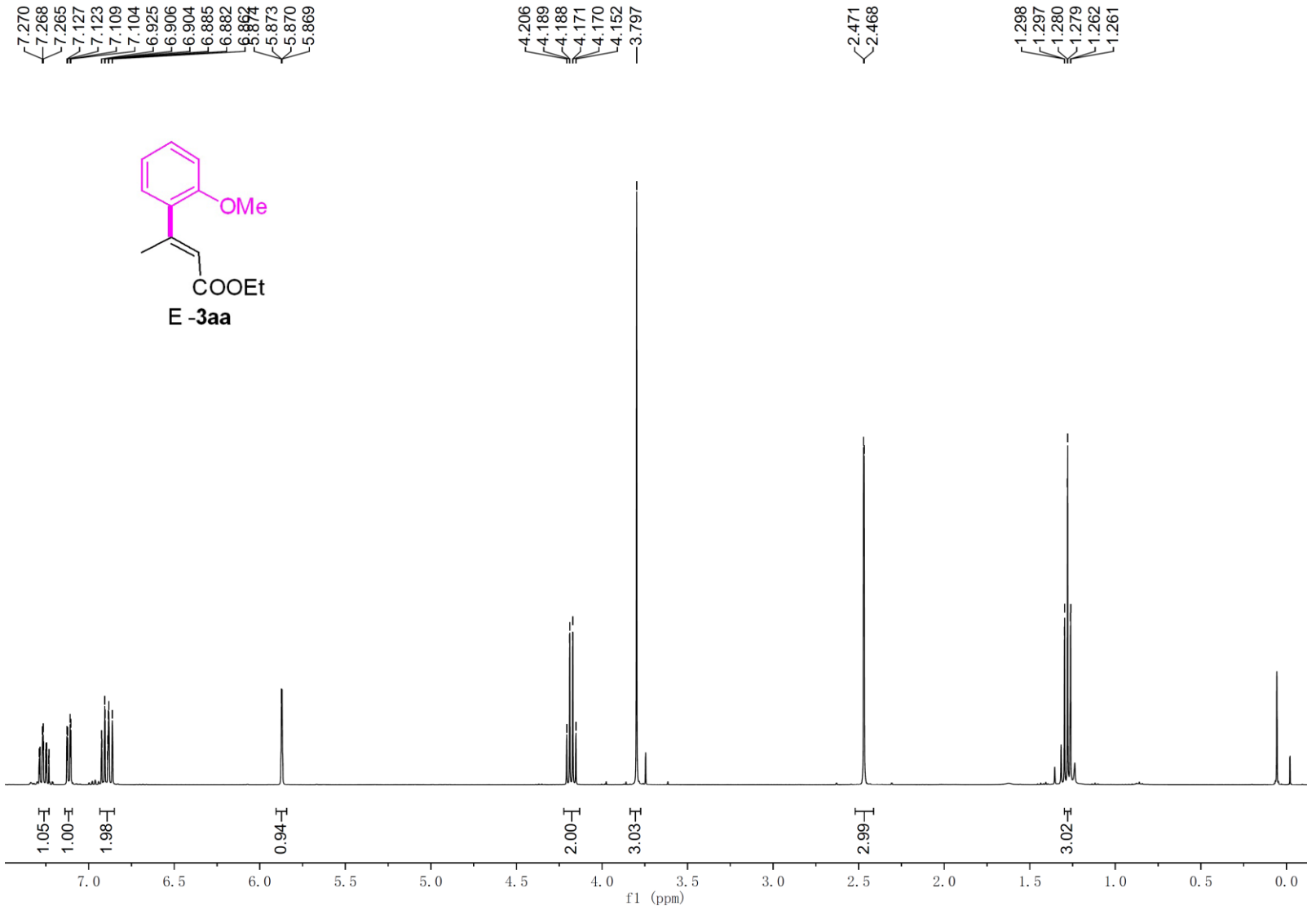
10
0
f1 (ppm)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4.188
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2.468
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1.297
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COOEt
E -3aa
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2.00
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2.99
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6.0
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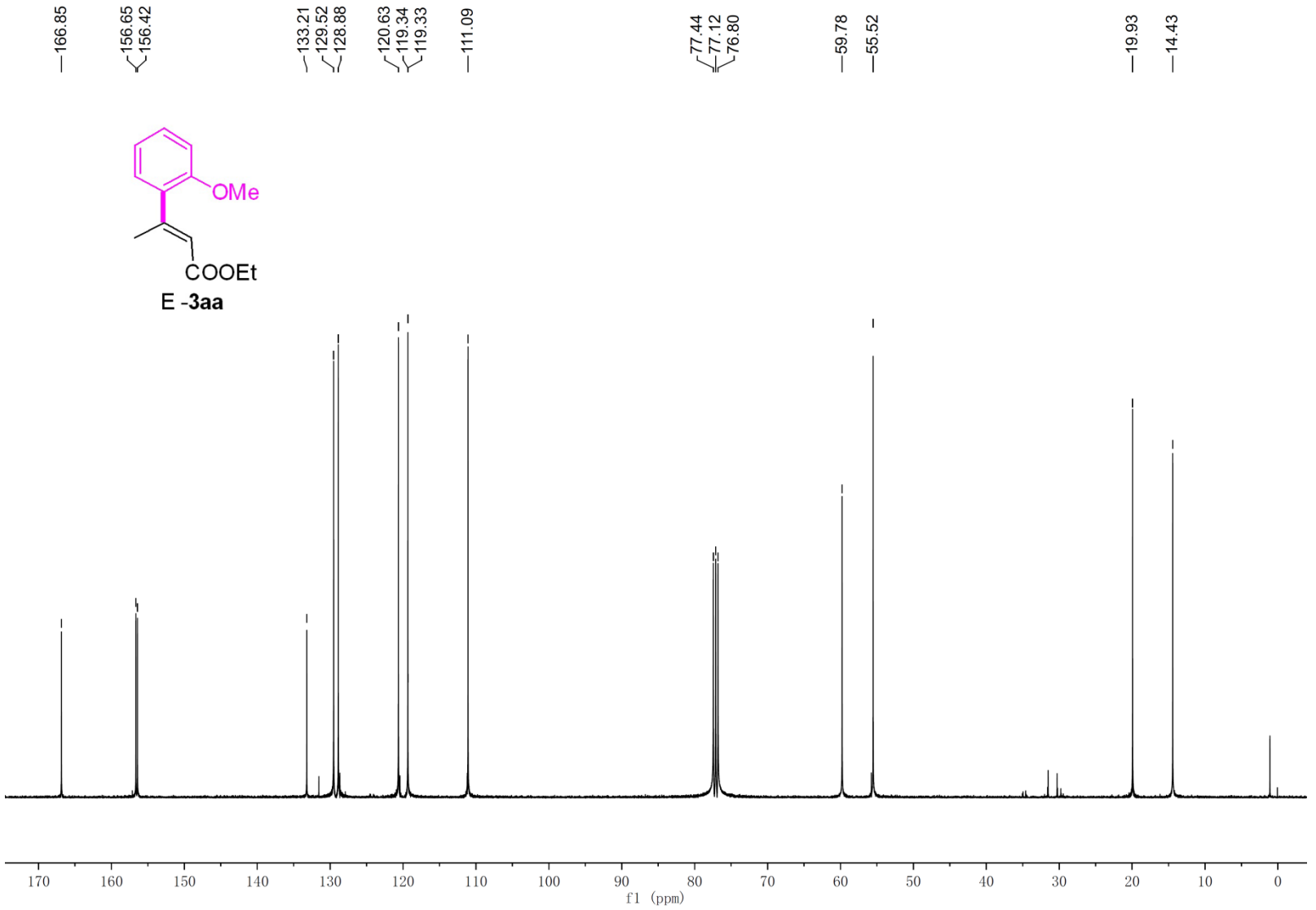
f1 (ppm)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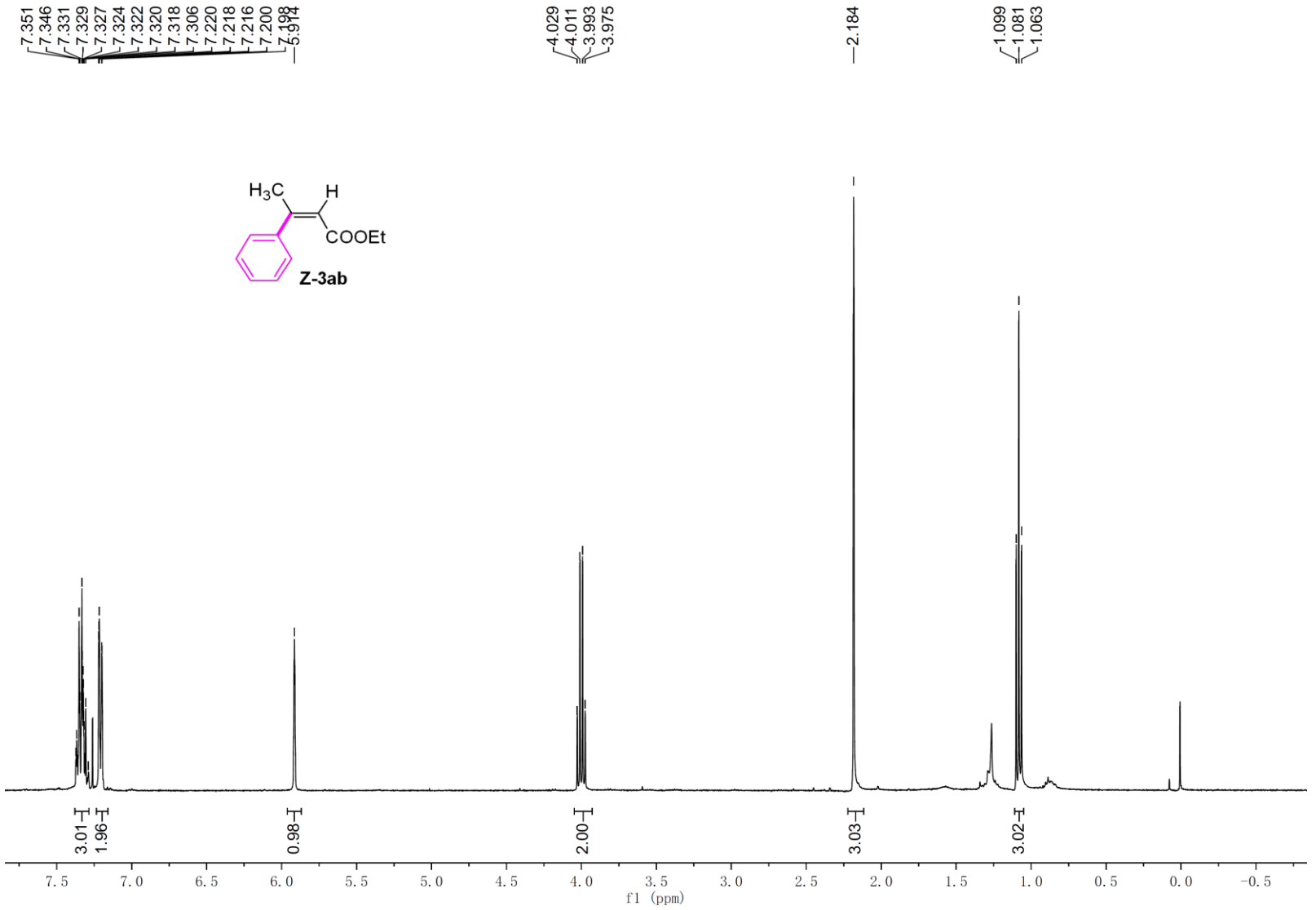
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3.02
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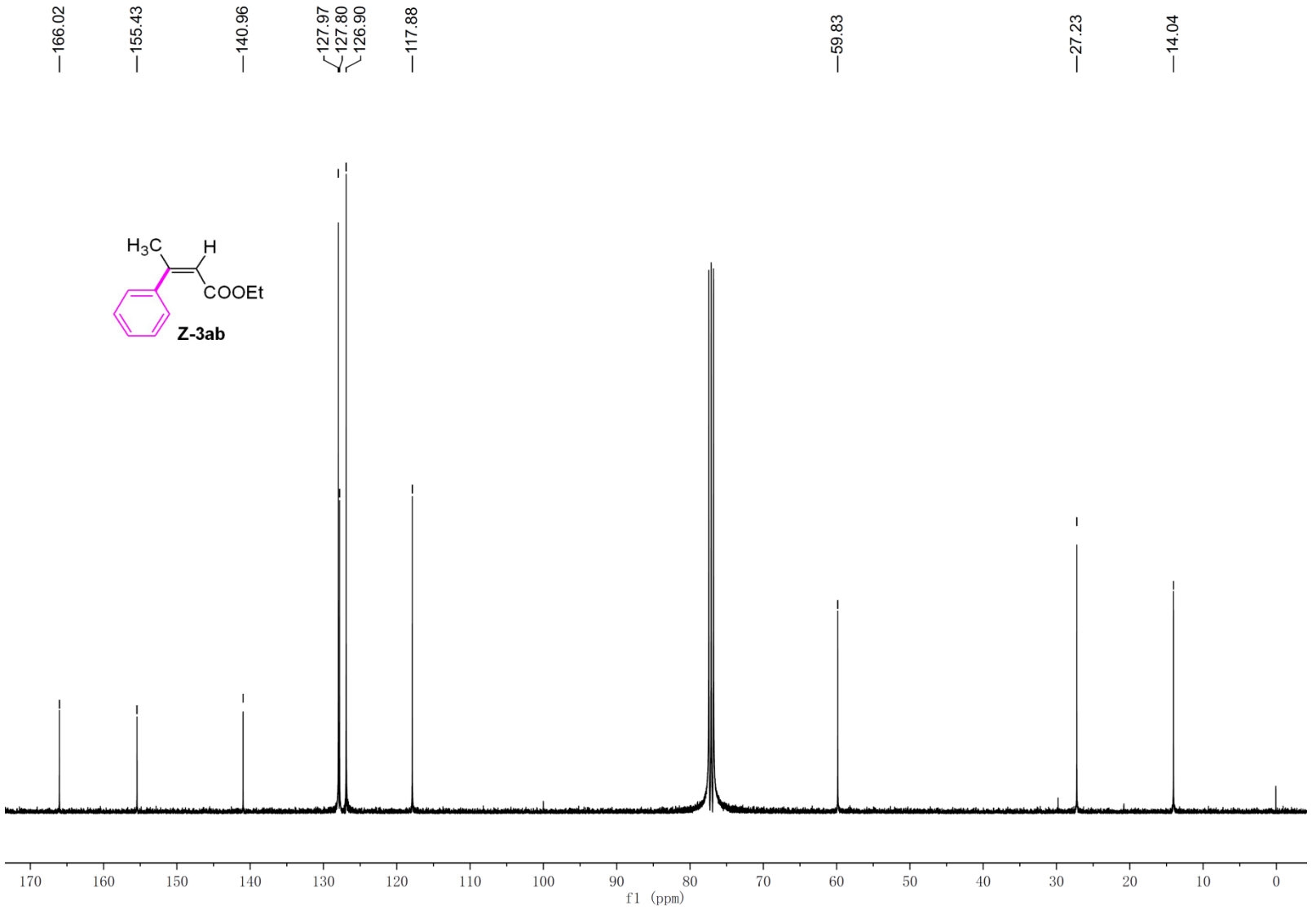
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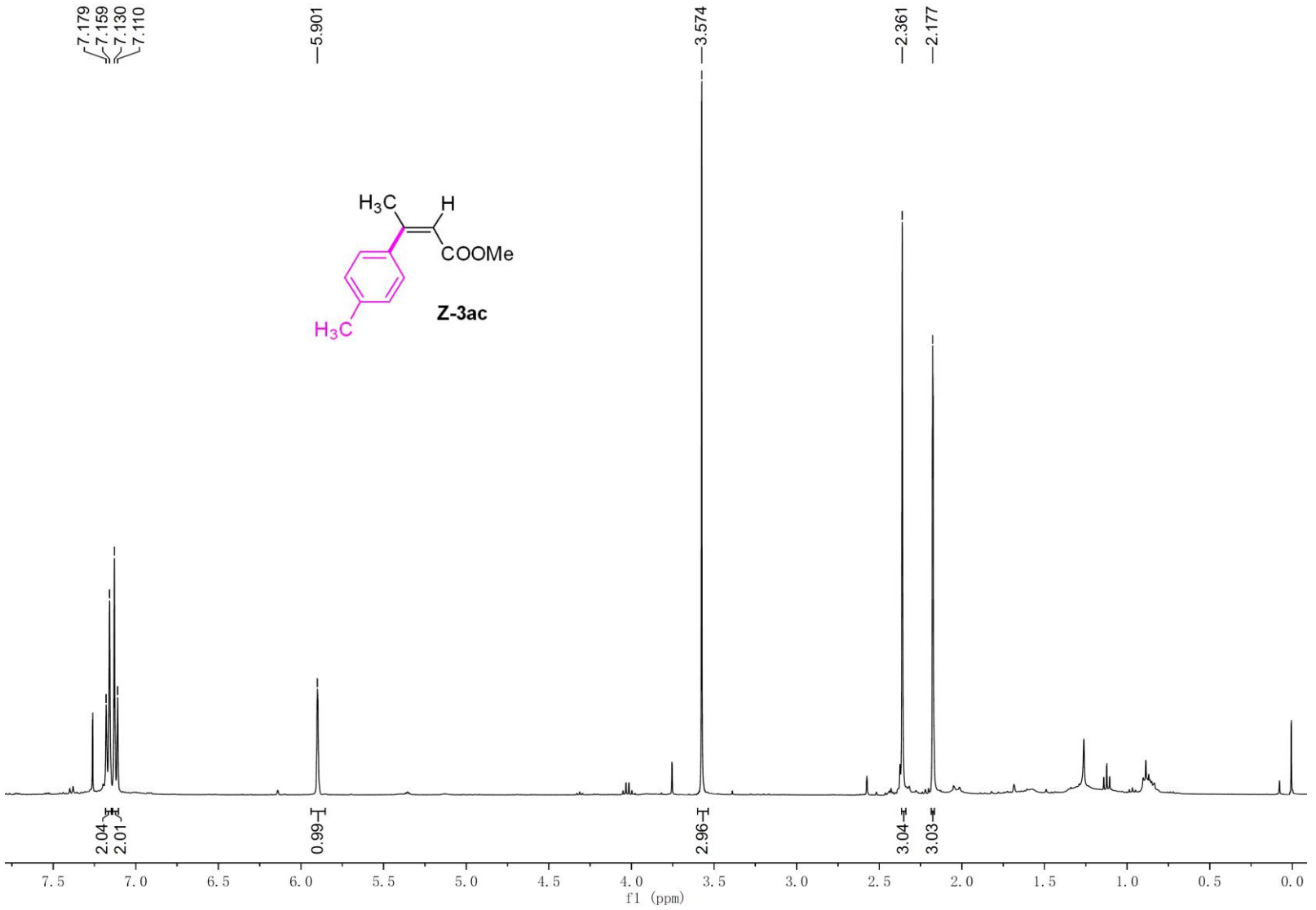
f1 (ppm)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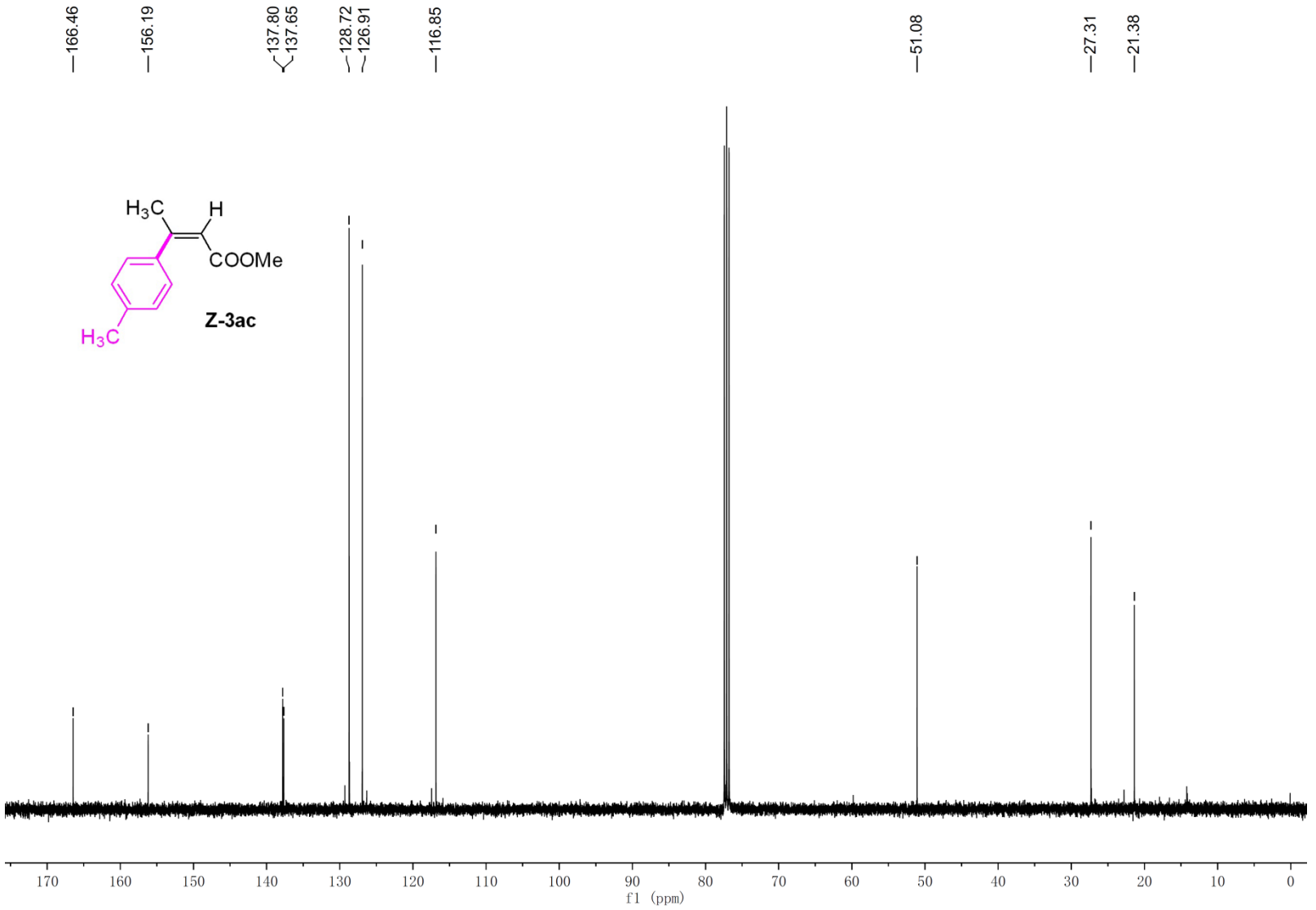
f1 (ppm)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H3C
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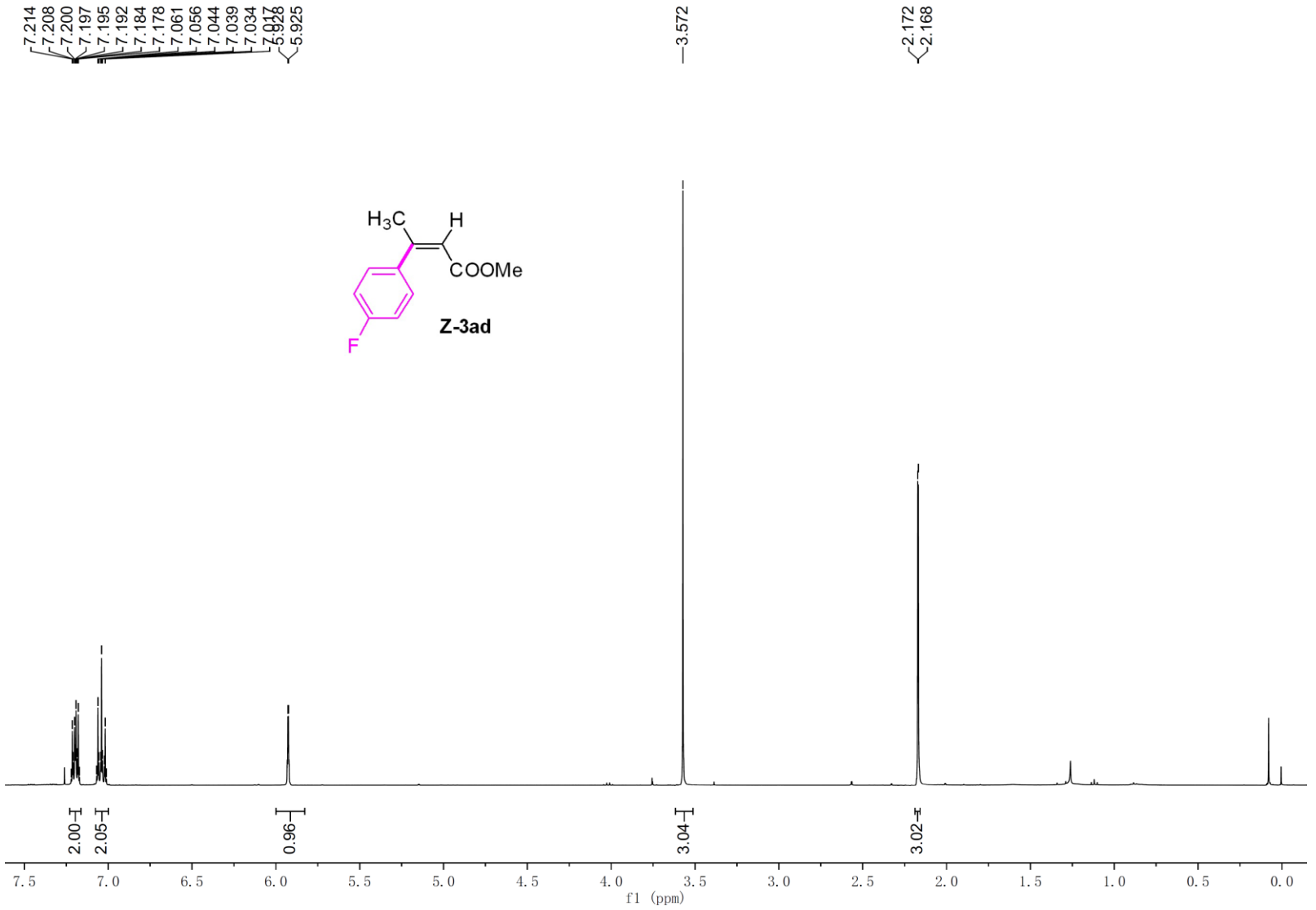
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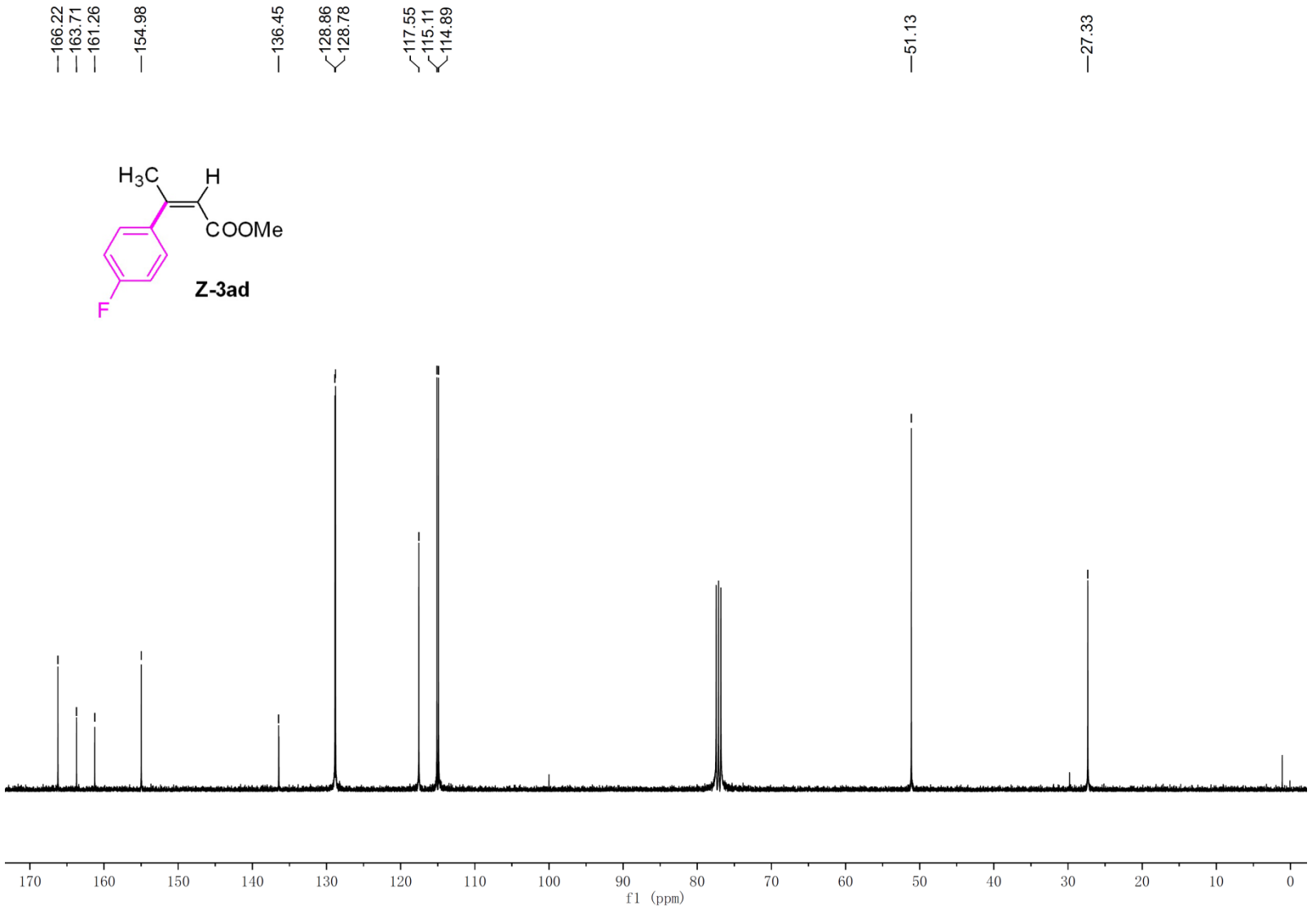
f1 (ppm)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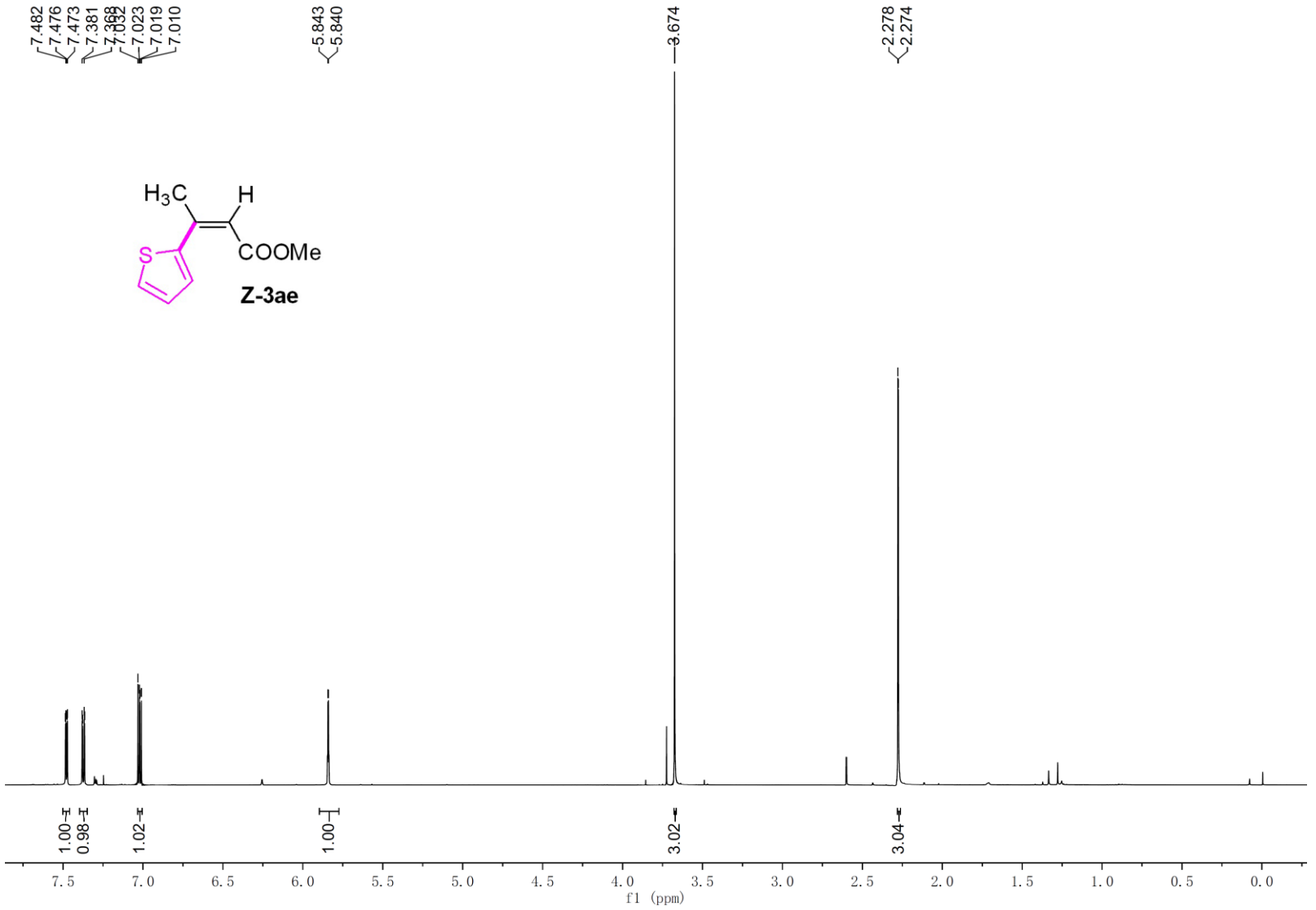
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2.278
2.274
H3C
H
COOMe
S
Z-3ae
1.00
0.98
1.02
1.00
3.02
3.04
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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3.0
2.5
2.0
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1.0
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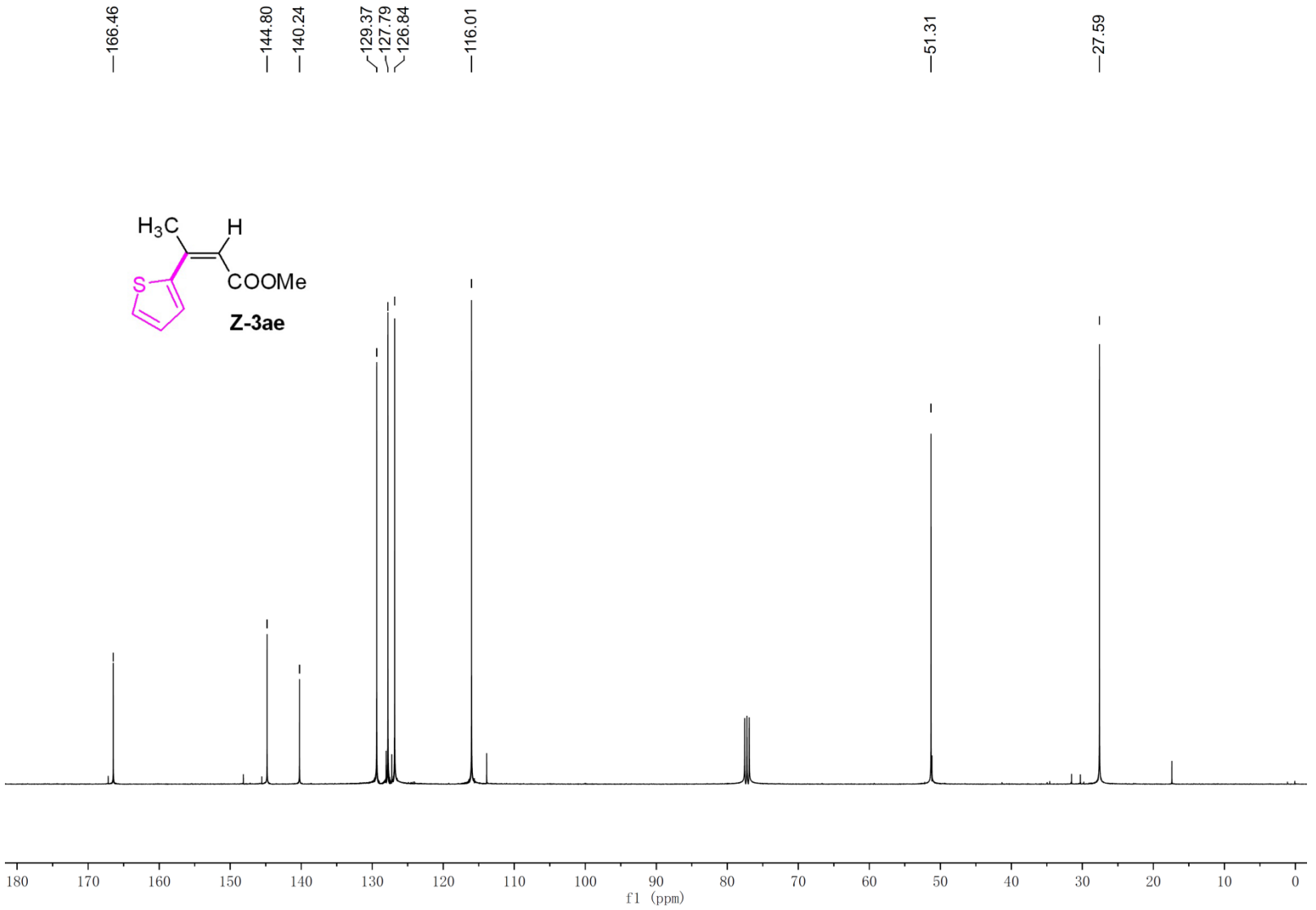
0.0
f1 (ppm)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144.80
140.24
129.37
127.79
126.84
116.01
51.31
27.59
H3C
H
S
COOMe
Z-3ae
180
170
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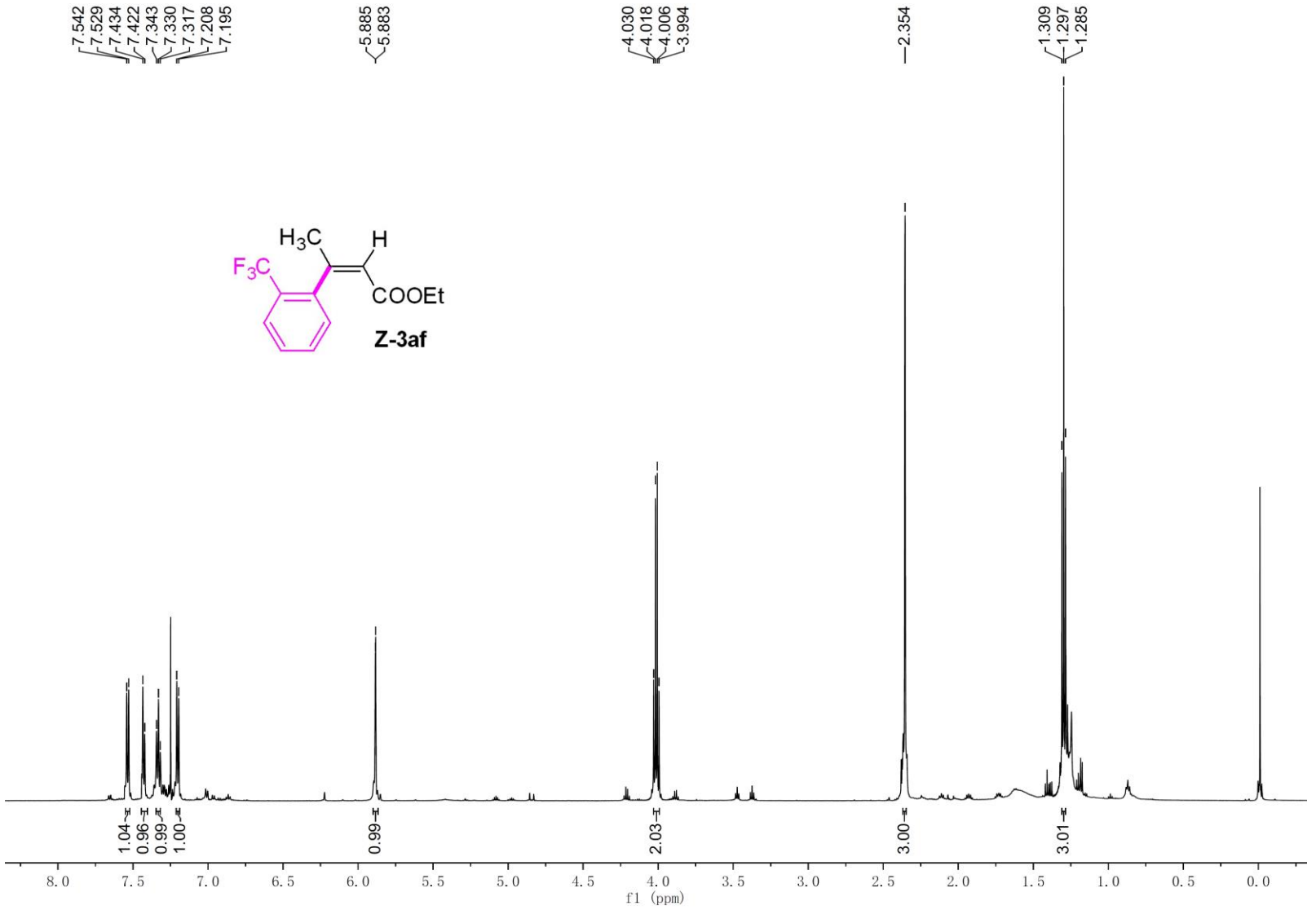
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.317
7.208
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5.883
4.030
4.018
4.006
3.994
2.354
1.309
1.297
1.285
H3C
H
F3C
COOEt
Z-3af
1.04
0.96
0.99
1.00
0.99
2.03
3.00
3.01
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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3.0
2.5
2.0
1.5
1.0
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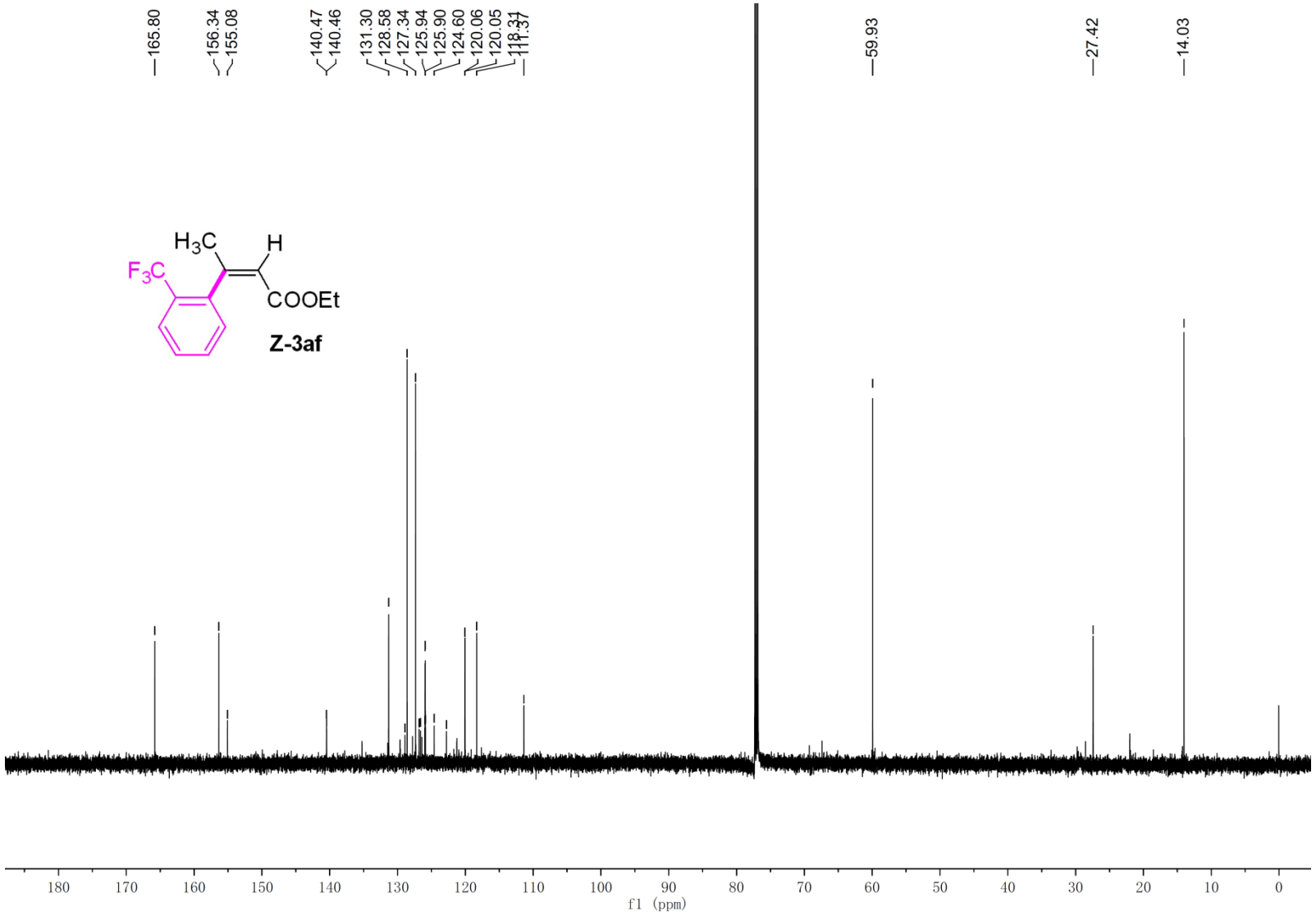
0.0
f1 (ppm)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156.34
155.08
140.47
140.46
131.30
128.58
127.34
125.94
125.90
124.60
120.06
120.05
118.31
111.37
59.93
27.42
14.03
H3C
H
F3C
COOEt
Z-3af
180
170
160
150
140
130
120
110
100
90
80
70
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50
40
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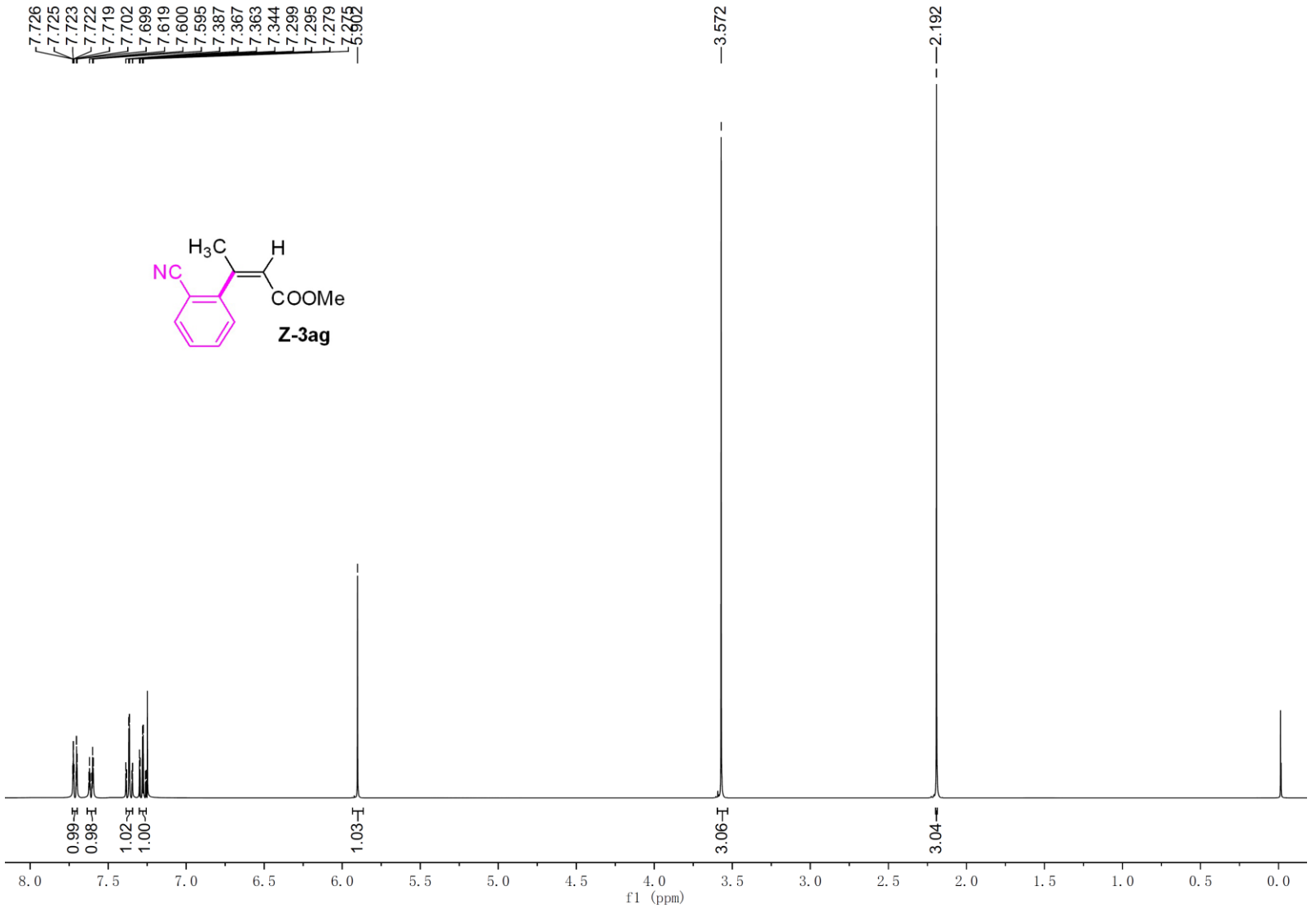
f1 (ppm)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7.619
7.600
7.595
7.387
7.367
7.363
7.344
7.299
7.295
7.279
7.275
5.902
3.572
2.192
H3C
H
NC
COOMe
Z-3ag
0.99
0.98
1.02
1.00
1.03
3.06
3.04
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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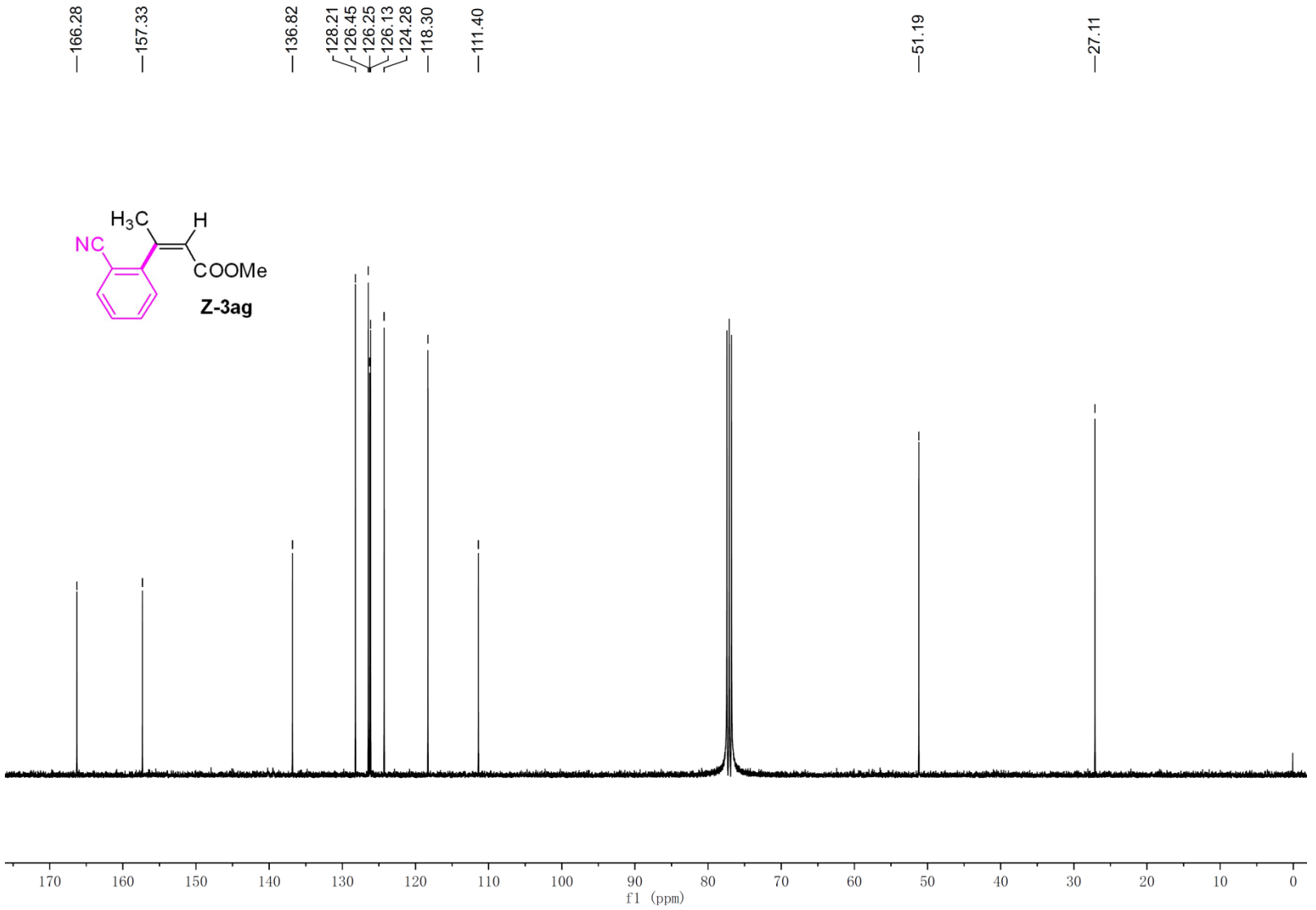
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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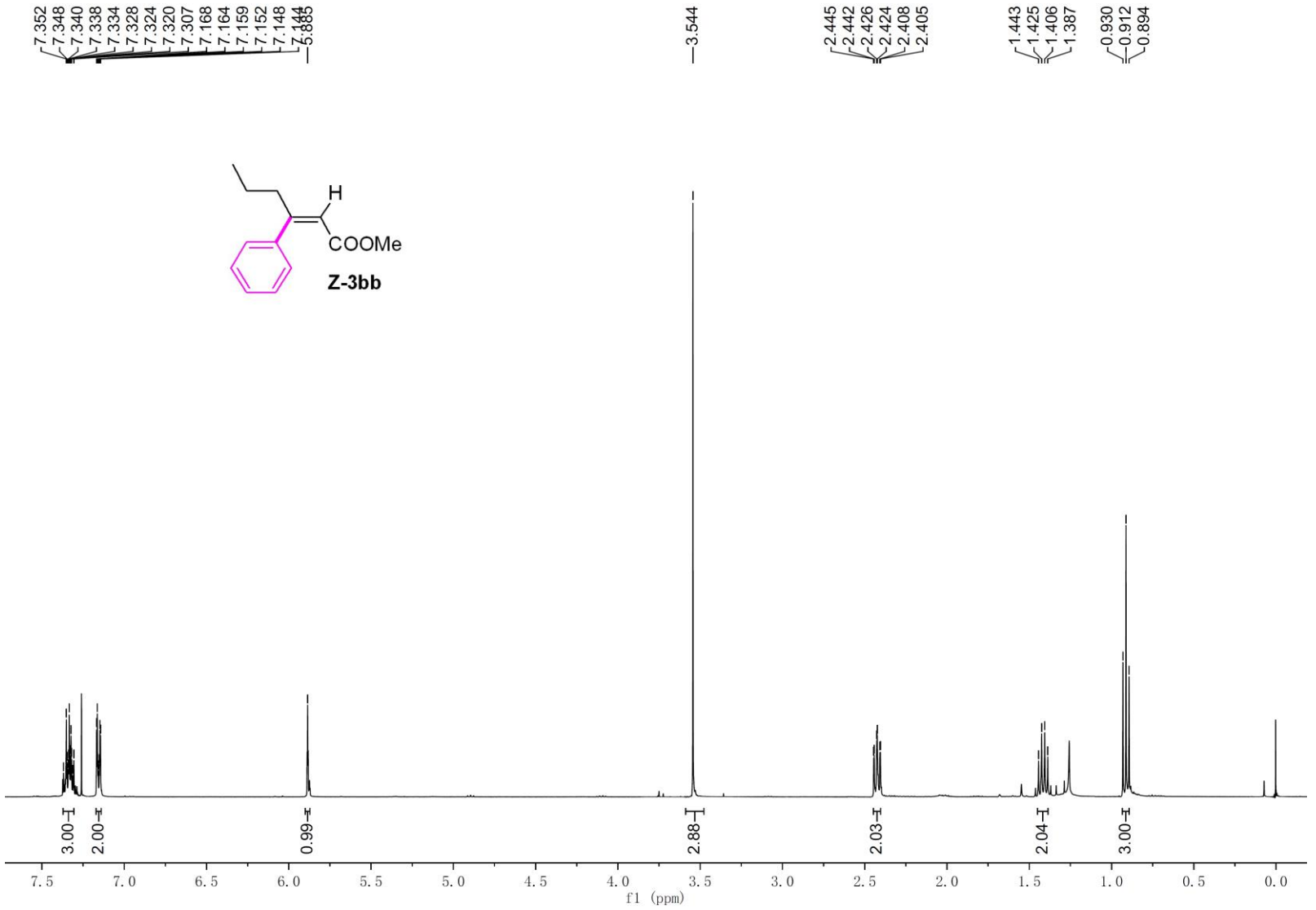
157.33
136.82
128.21
126.45
126.25
126.13
124.28
118.30
111.40
51.19
27.11
H3C
H
NC
COOMe
Z-3ag
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.328
7.324
7.320
7.307
7.168
7.164
7.159
7.152
7.148
7.144
5.885
3.544
2.445
2.442
2.426
2.424
2.408
2.405
1.443
1.425
1.406
1.387
0.930
0.912
0.894
H
COOMe
Z-3bb
3.00
2.00
0.99
2.88
2.03
2.04
3.00
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
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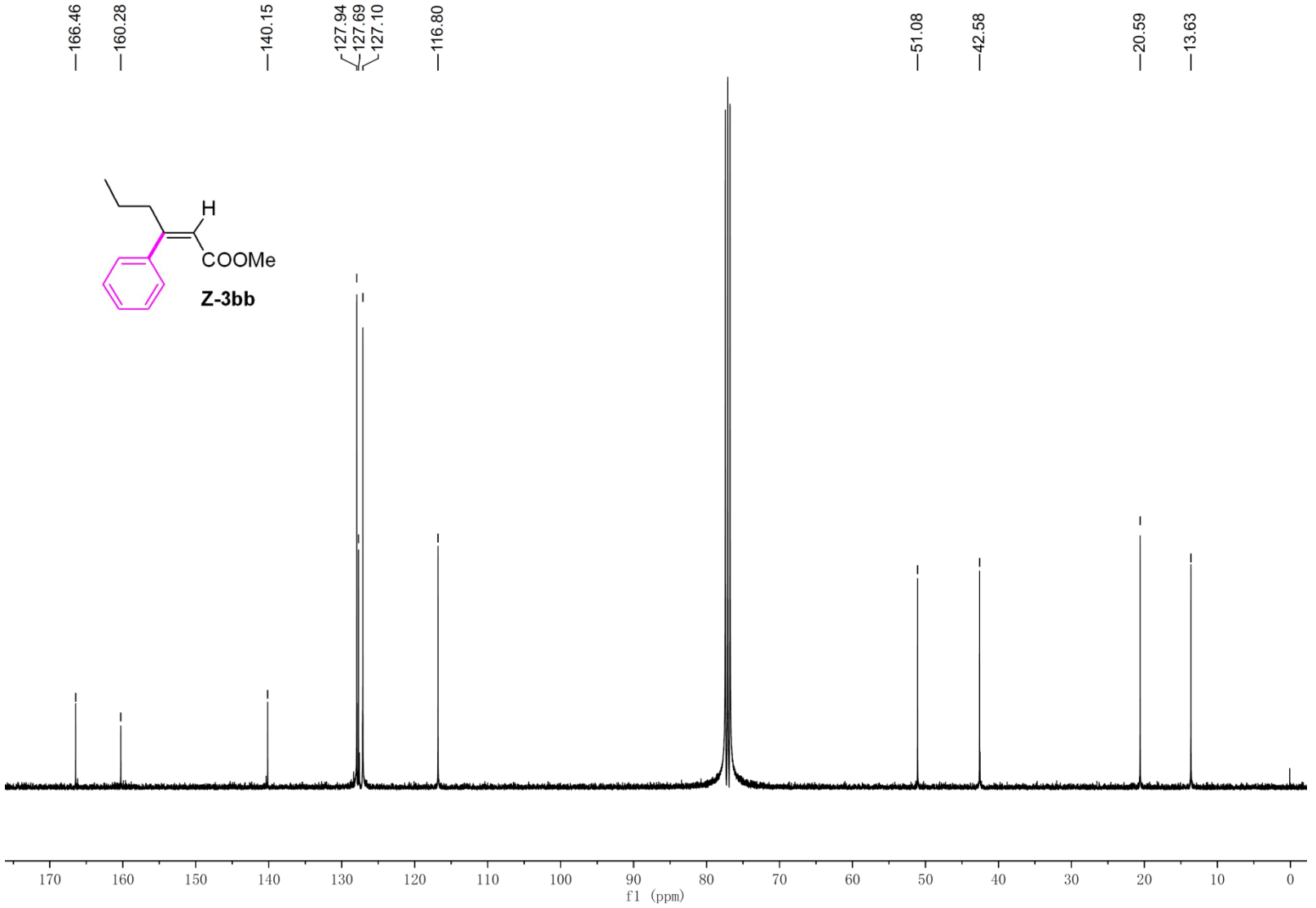
0.0
f1 (ppm)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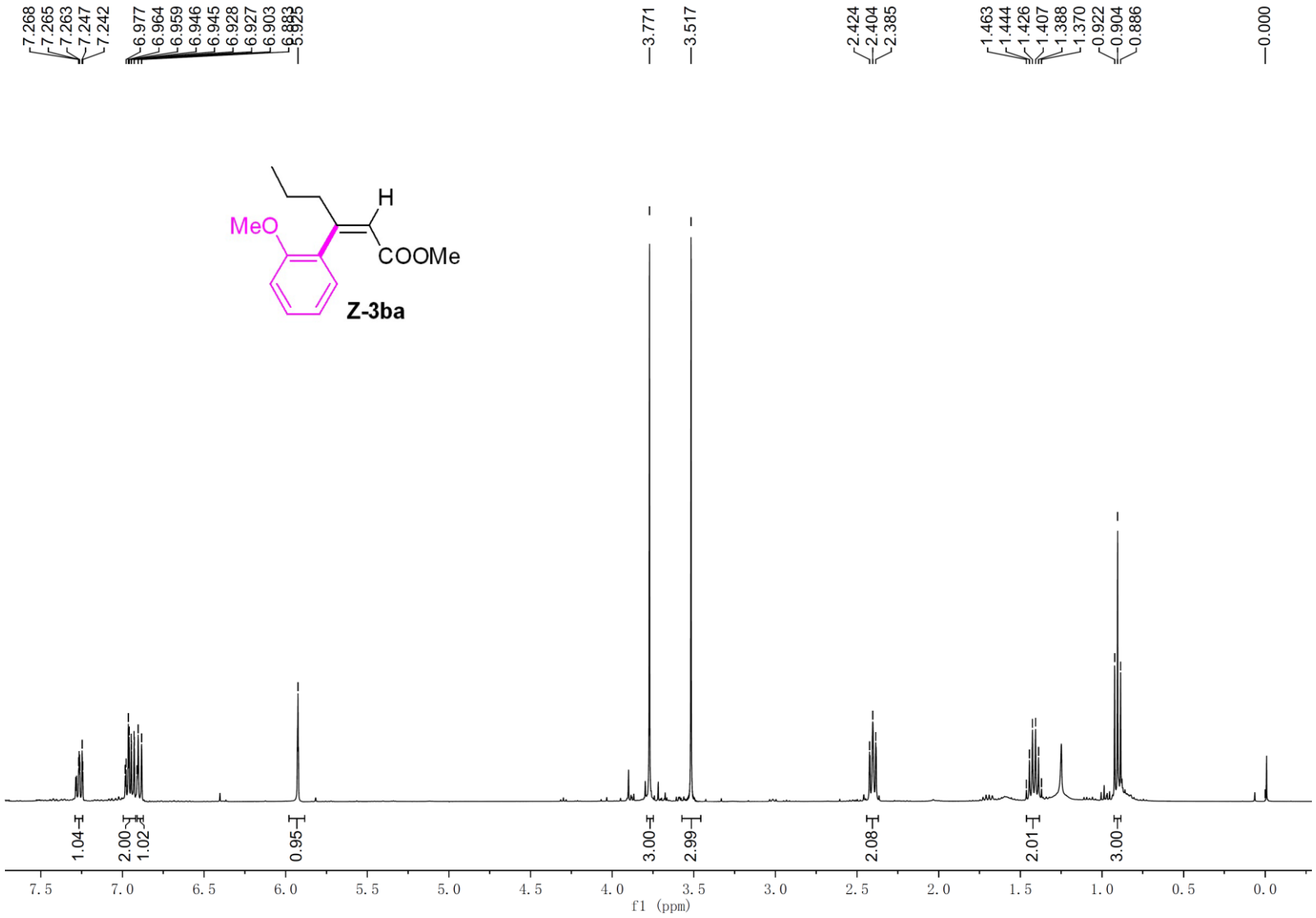
160.28
140.15
127.94
127.69
127.10
116.80
51.08
42.58
20.59
13.63
H
COOMe
Z-3bb
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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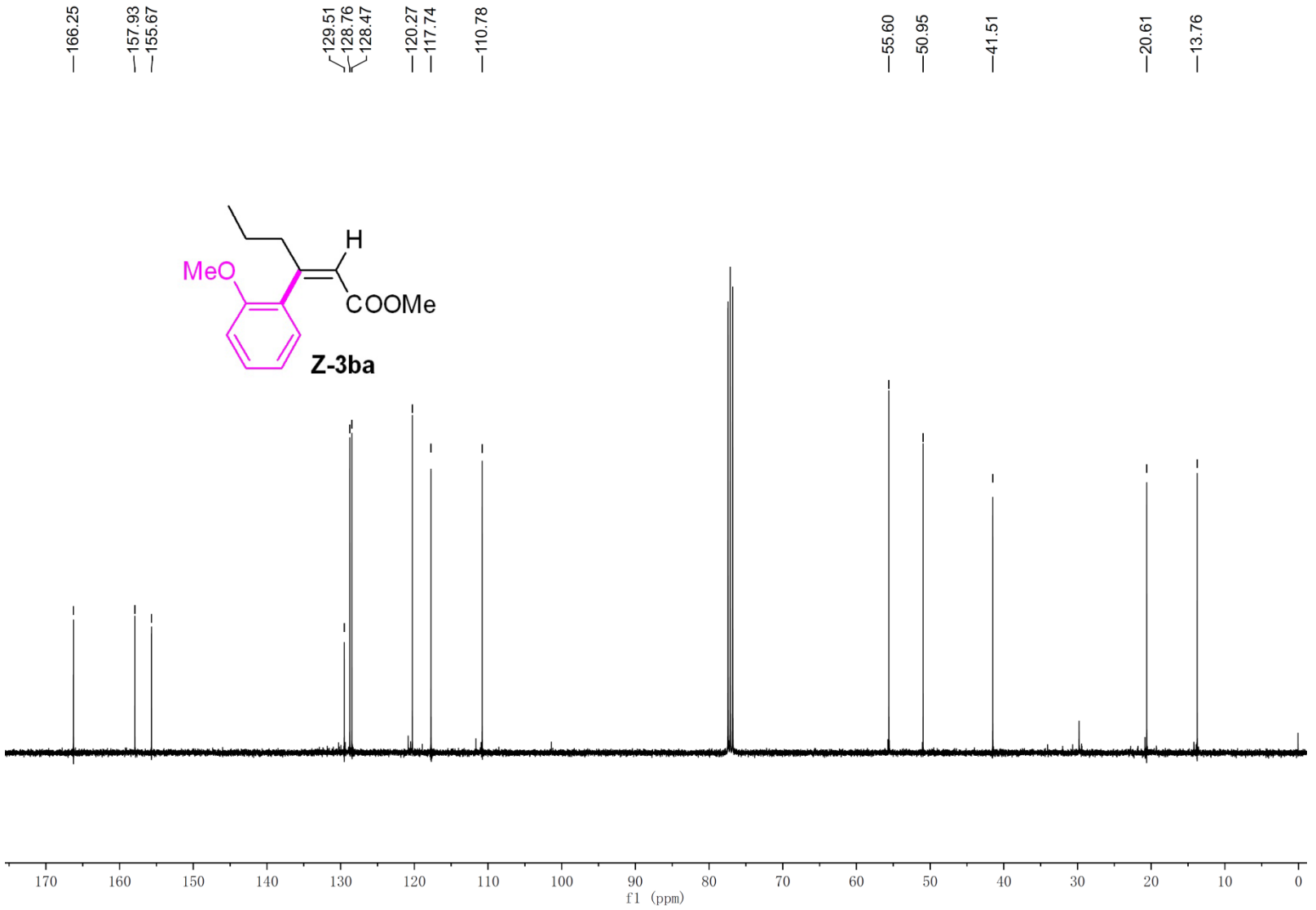
7.263
7.247
7.242
6.977
6.964
6.959
6.946
6.945
6.928
6.927
6.903
3.771
3.517
2.424
2.404
2.385
1.463
1.444
1.426
1.407
1.388
1.370
0.922
0.904
0.886
0.000
MeO
H
COOMe
Z-3ba
1.04
2.00
1.02
0.95
3.00
2.99
2.08
2.01
3.00
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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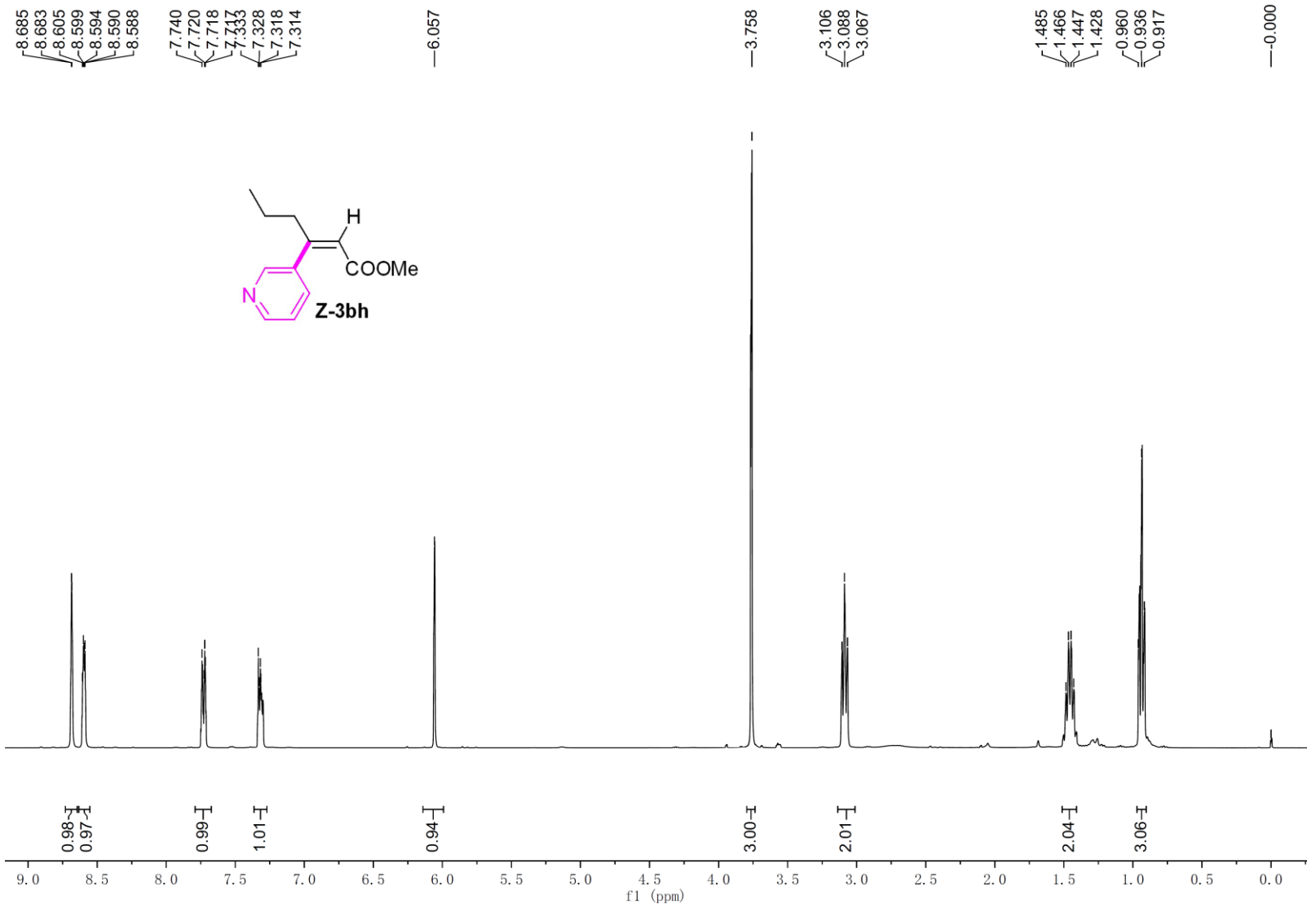
128.47
120.27
117.74
110.78
55.60
50.95
41.51
20.61
13.76
MeO
H
COOMe
Z-3ba
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.717
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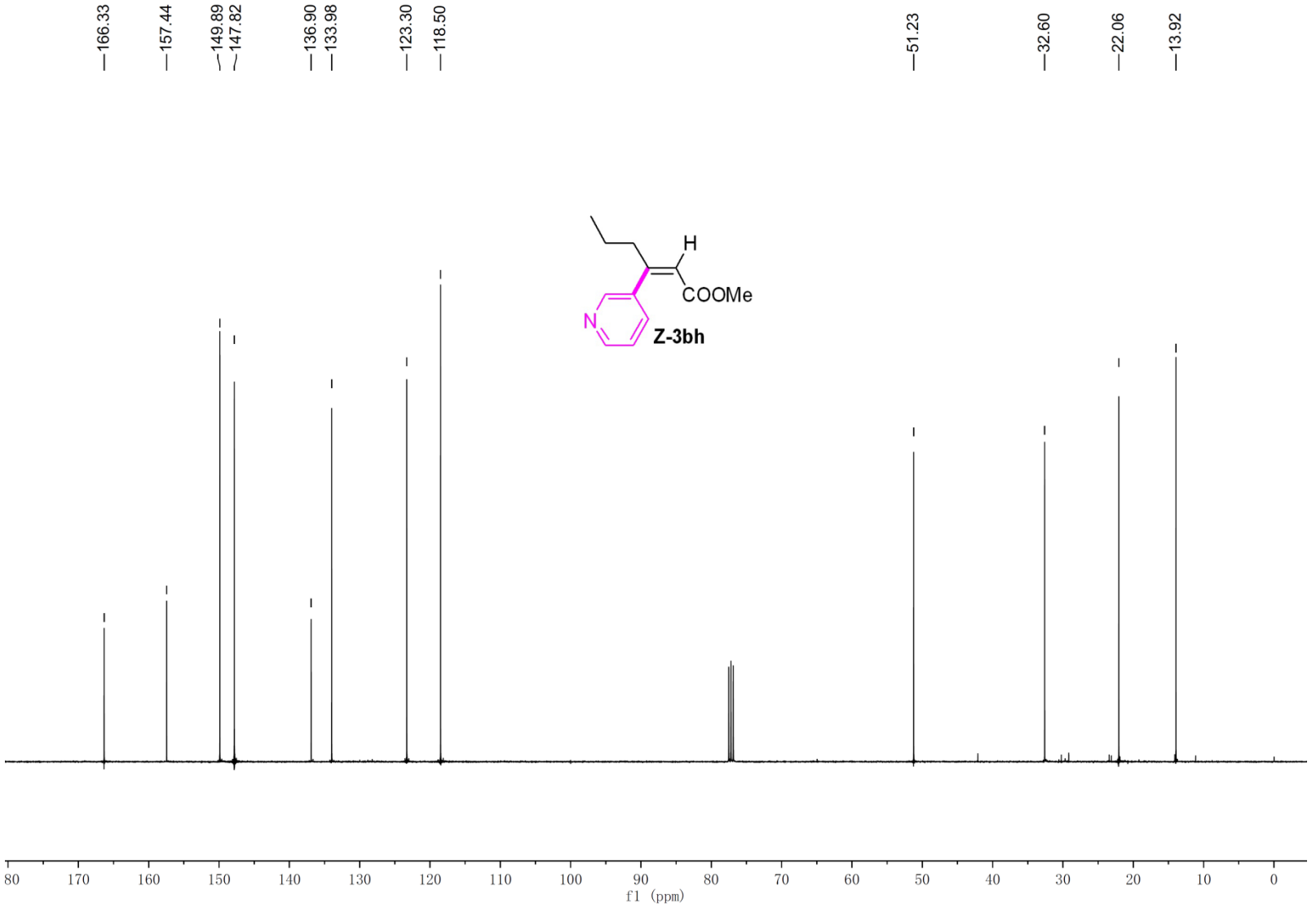
7.328
7.318
7.314
6.057
3.758
3.106
3.088
3.067
1.485
1.466
1.447
1.428
0.960
0.936
0.917
-0.000
H
COOMe
N
Z-3bh
0.98
0.97
0.99
1.01
0.94
3.00
2.01
2.04
3.06
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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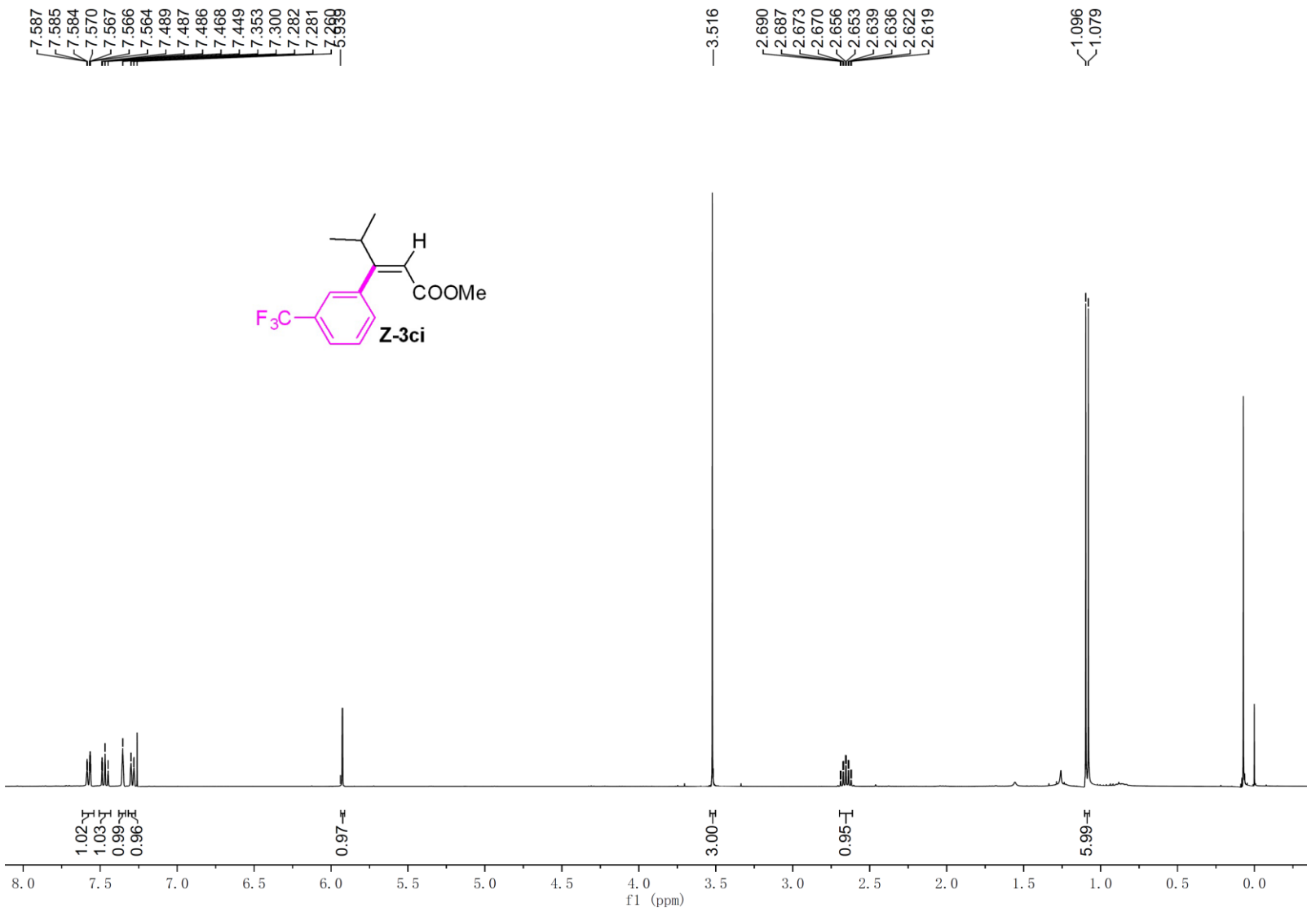
136.90
133.98
123.30
118.50
51.23
32.60
22.06
13.92
H
COOMe
N
Z-3bh
80
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.489
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7.486
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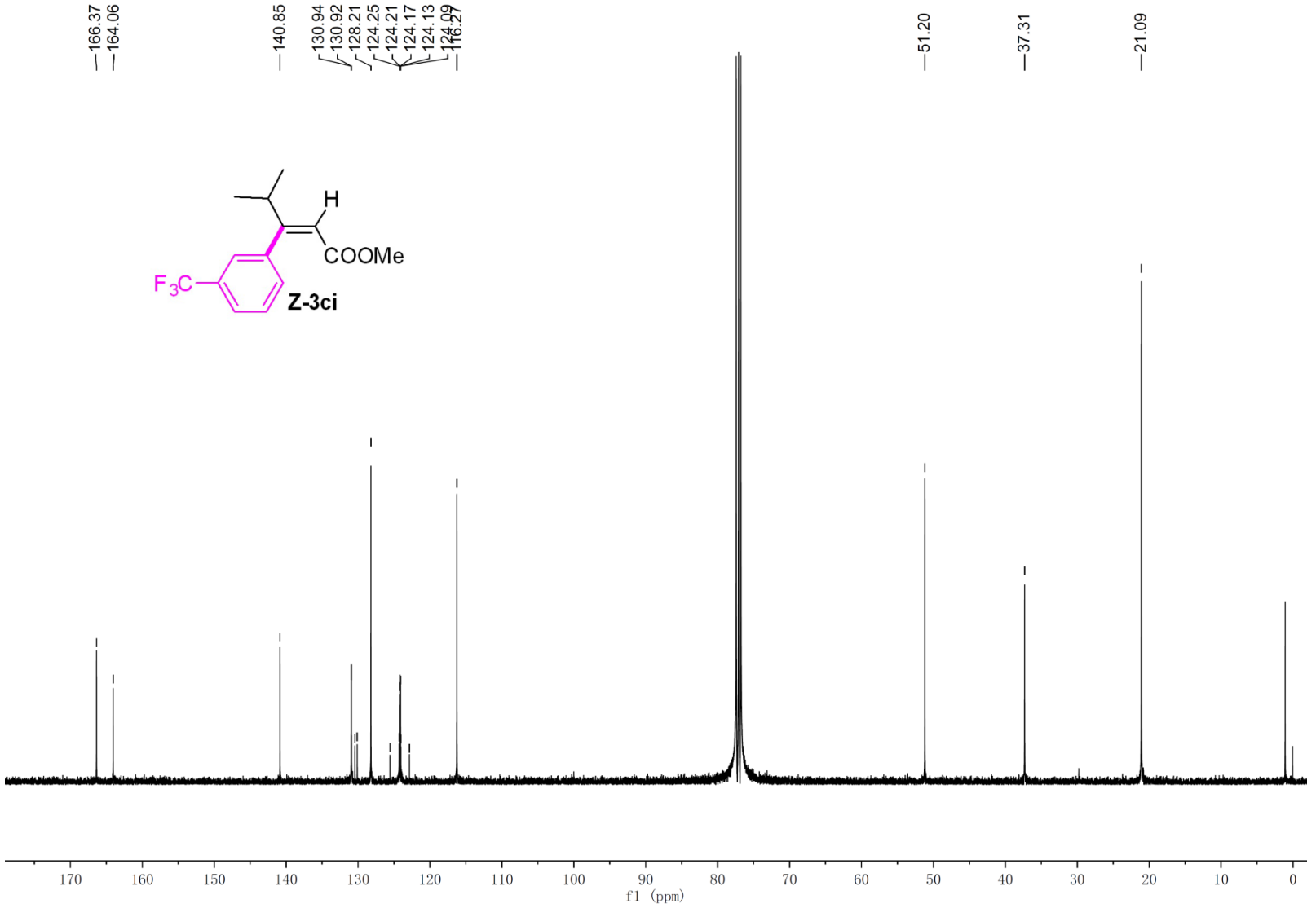
7.449
7.353
7.300
7.282
7.281
7.260
5.939
3.516
2.690
2.687
2.673
2.670
2.656
2.653
2.639
2.636
2.622
2.619
1.096
1.079
H
COOMe
F3C
Z-3ci
1.02
1.03
0.99
0.96
0.97
3.00
0.95
5.99
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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164.06
140.85
130.94
130.92
128.21
124.25
124.21
124.17
124.13
124.09
116.27
51.20
37.31
21.09
H
COOMe
F3C
Z-3ci
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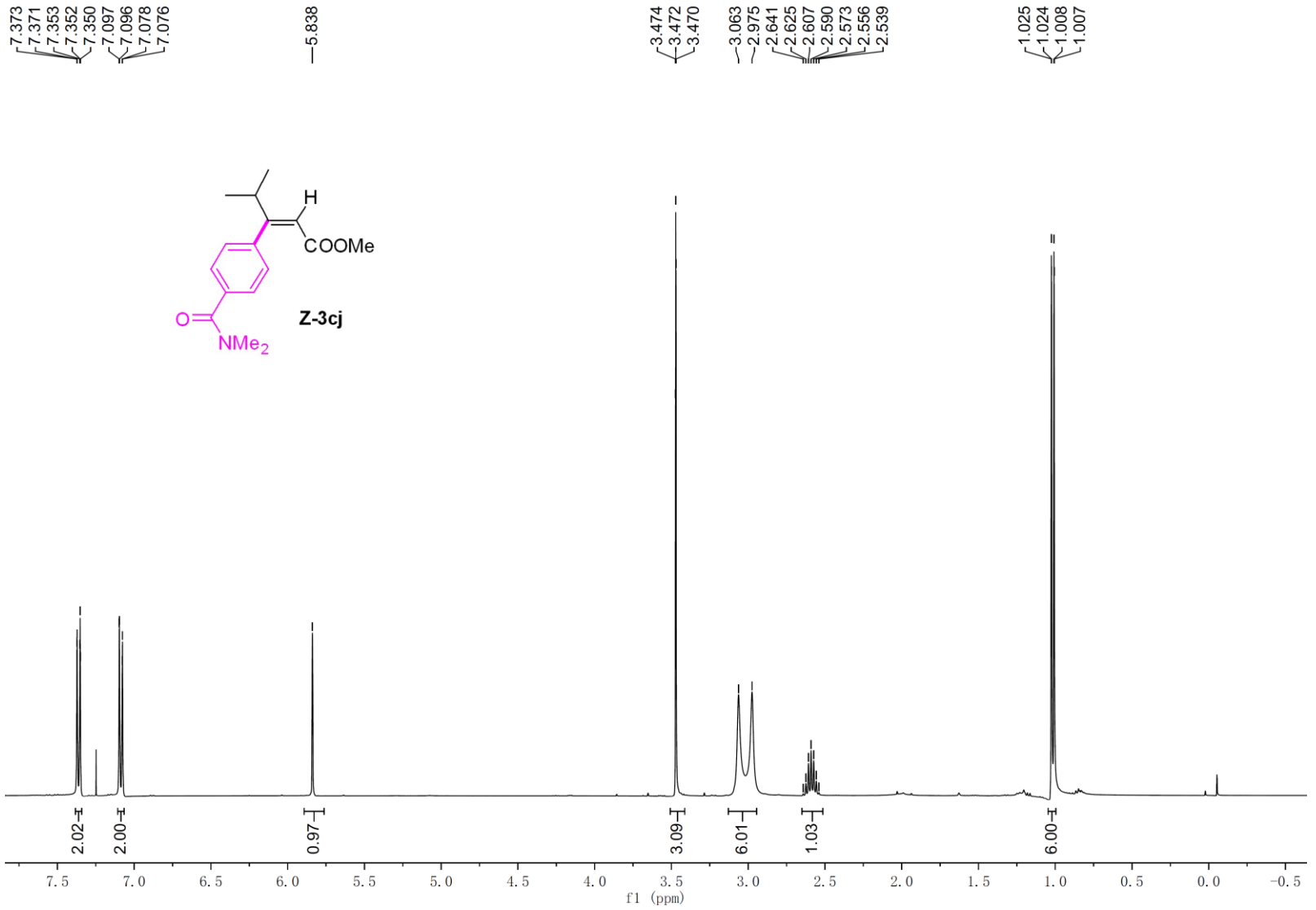
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.096
7.078
7.076
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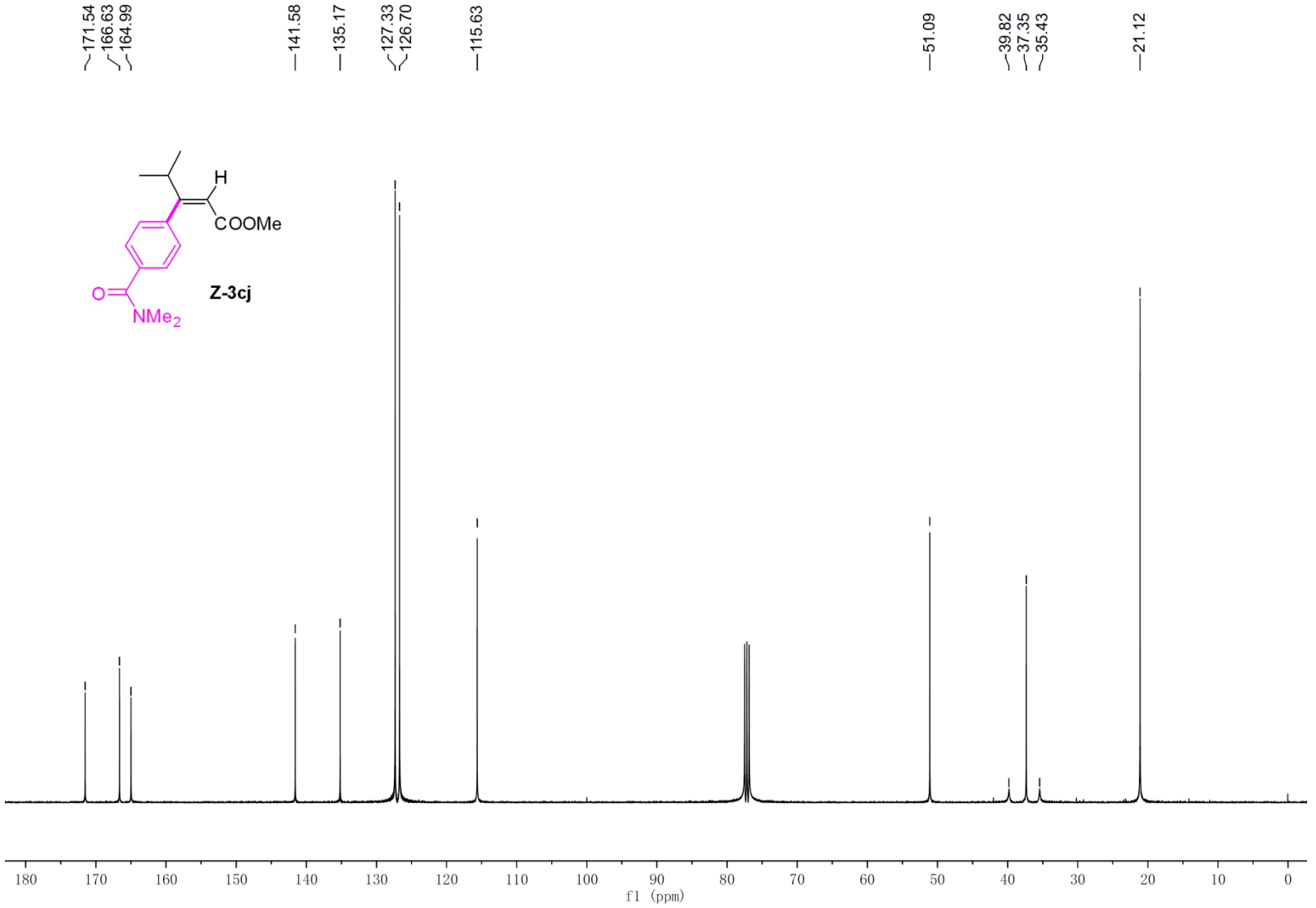
3.472
3.470
3.063
2.975
2.641
2.625
2.607
2.590
2.573
2.556
2.539
1.025
1.024
1.008
1.007
H
COOMe
O
NMe2
Z-3cj
2.02
2.00
0.97
3.09
6.01
1.03
6.00
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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127.33
126.70
115.63
51.09
39.82
37.35
35.43
21.12
H
COOMe
O
NMe2
Z-3cj
180
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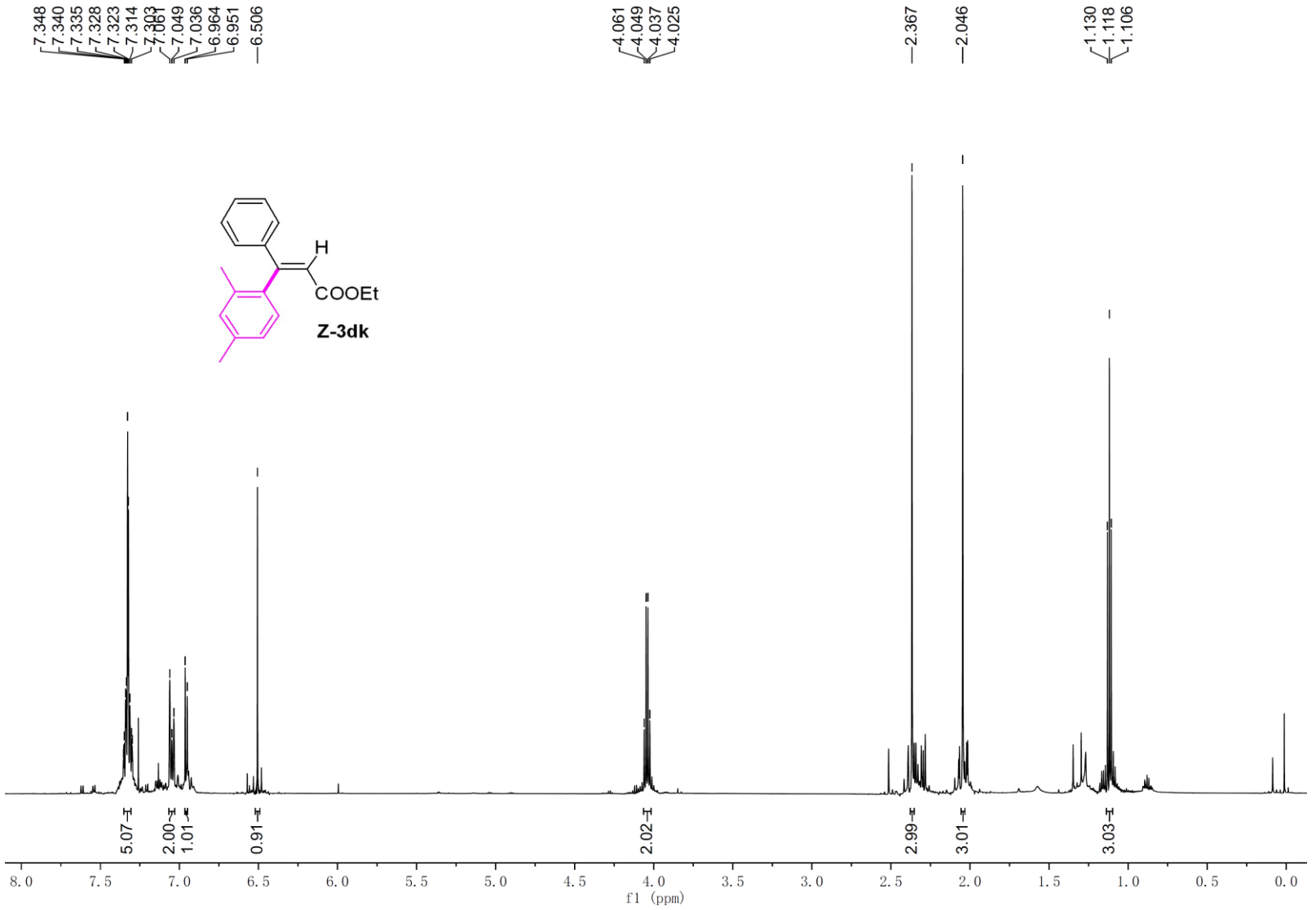
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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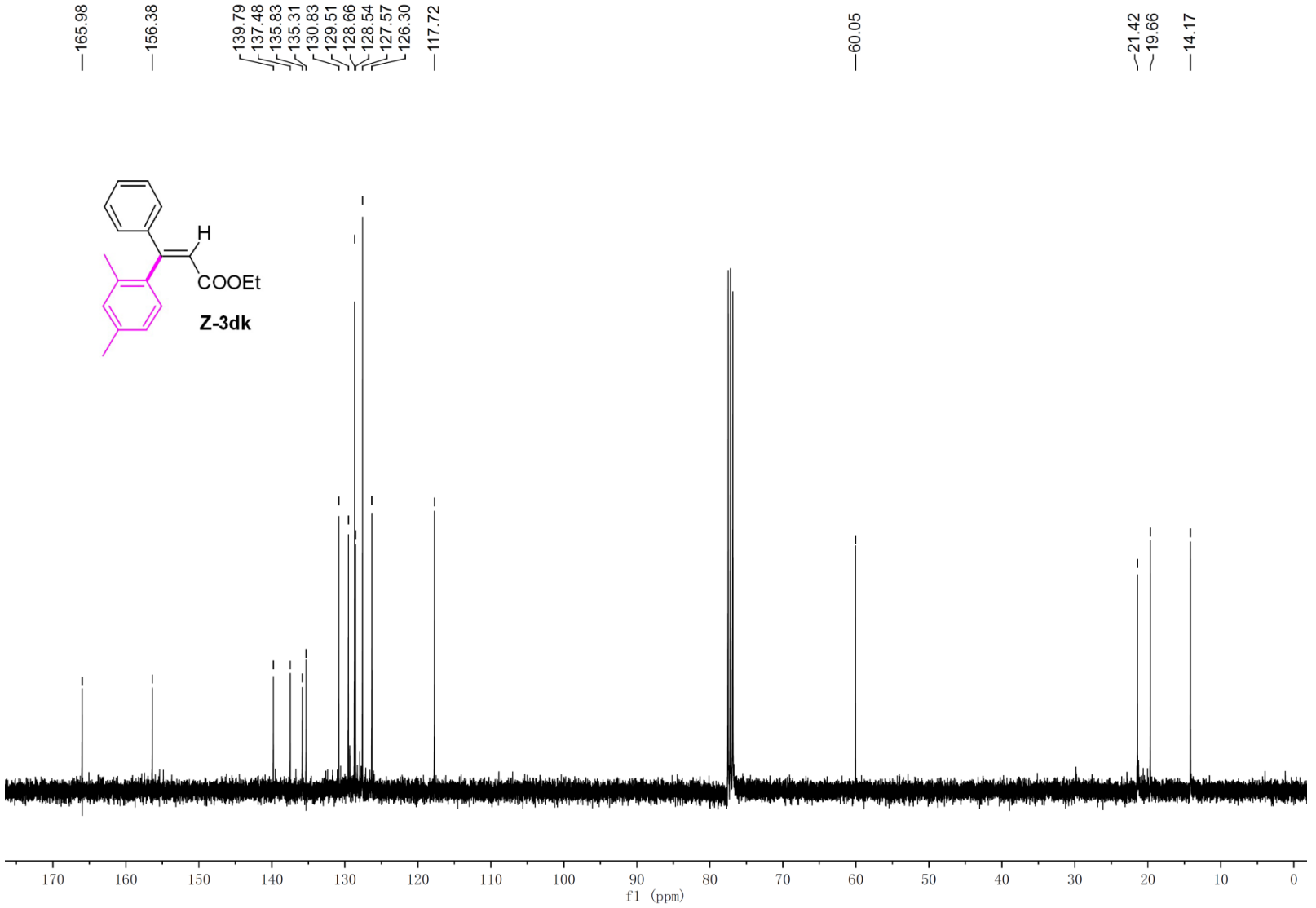
7.049
7.036
6.964
6.951
6.506
4.061
4.049
4.037
4.025
2.367
2.046
1.130
1.118
1.106
H
COOEt
Z-3dk
5.07
2.00
1.01
0.91
2.02
2.99
3.01
3.03
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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135.31
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129.51
128.66
128.54
127.57
126.30
117.72
60.05
21.42
19.66
14.17
H
COOEt
Z-3dk
170
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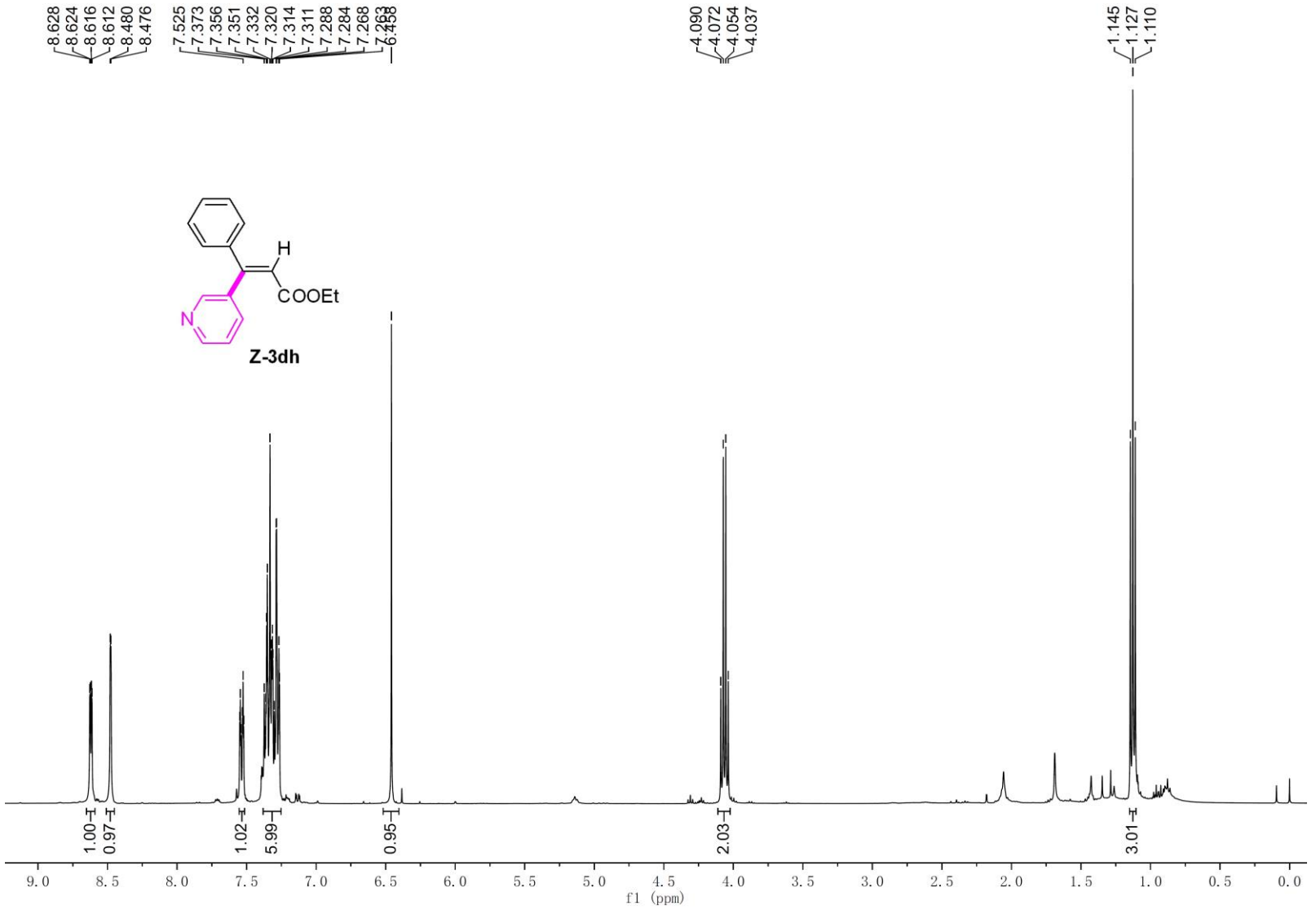
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.332
7.320
7.314
7.311
7.288
7.284
7.268
7.263
6.458
4.090
4.072
4.054
4.037
1.145
1.127
1.110
H
COOEt
N
Z-3dh
1.00
0.97
1.02
5.99
0.95
2.03
3.01
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
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2.0
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1.0
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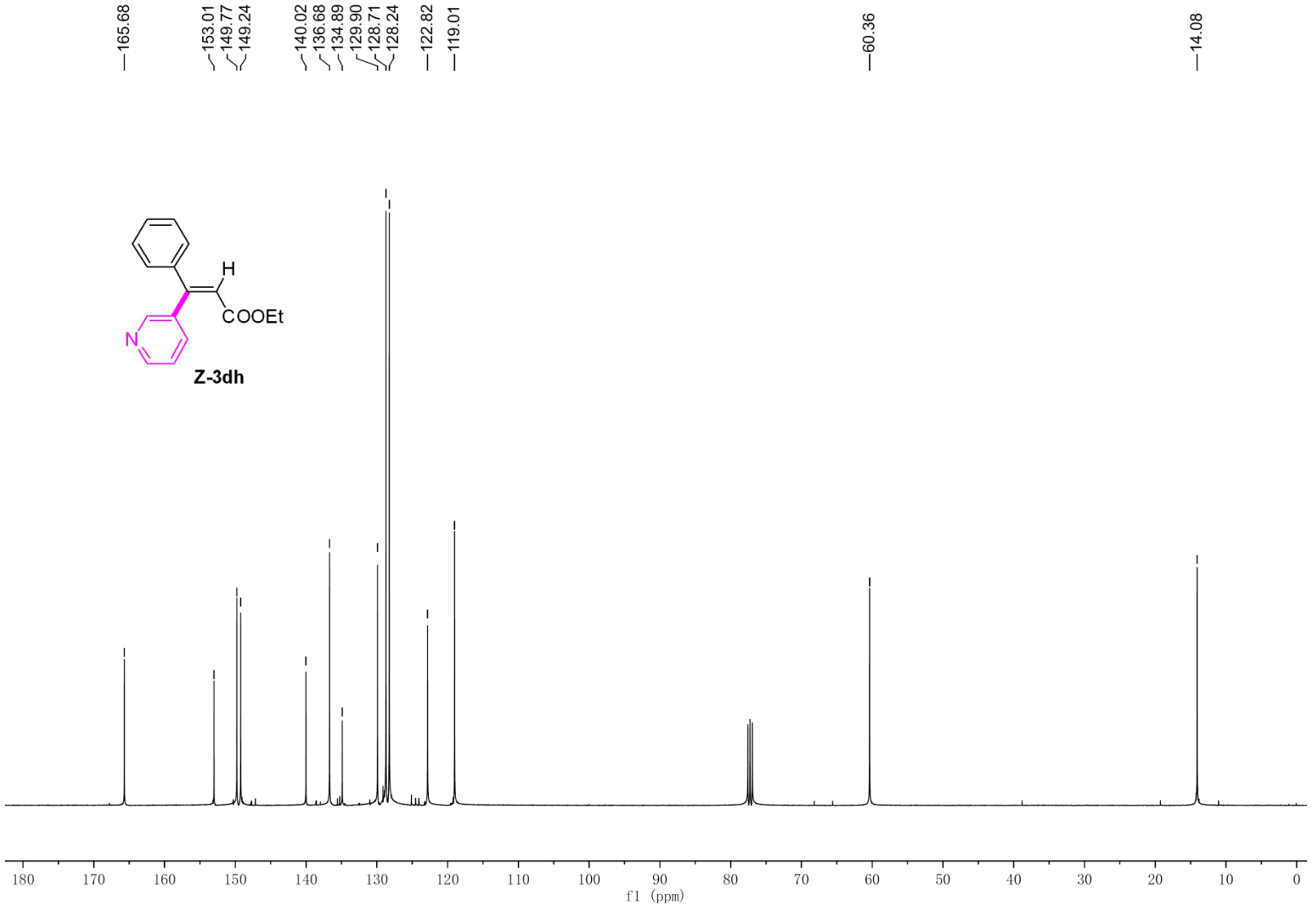
f1 (ppm)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136.68
134.89
129.90
128.71
128.24
122.82
119.01
60.36
14.08
H
COOEt
N
Z-3dh
180
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130
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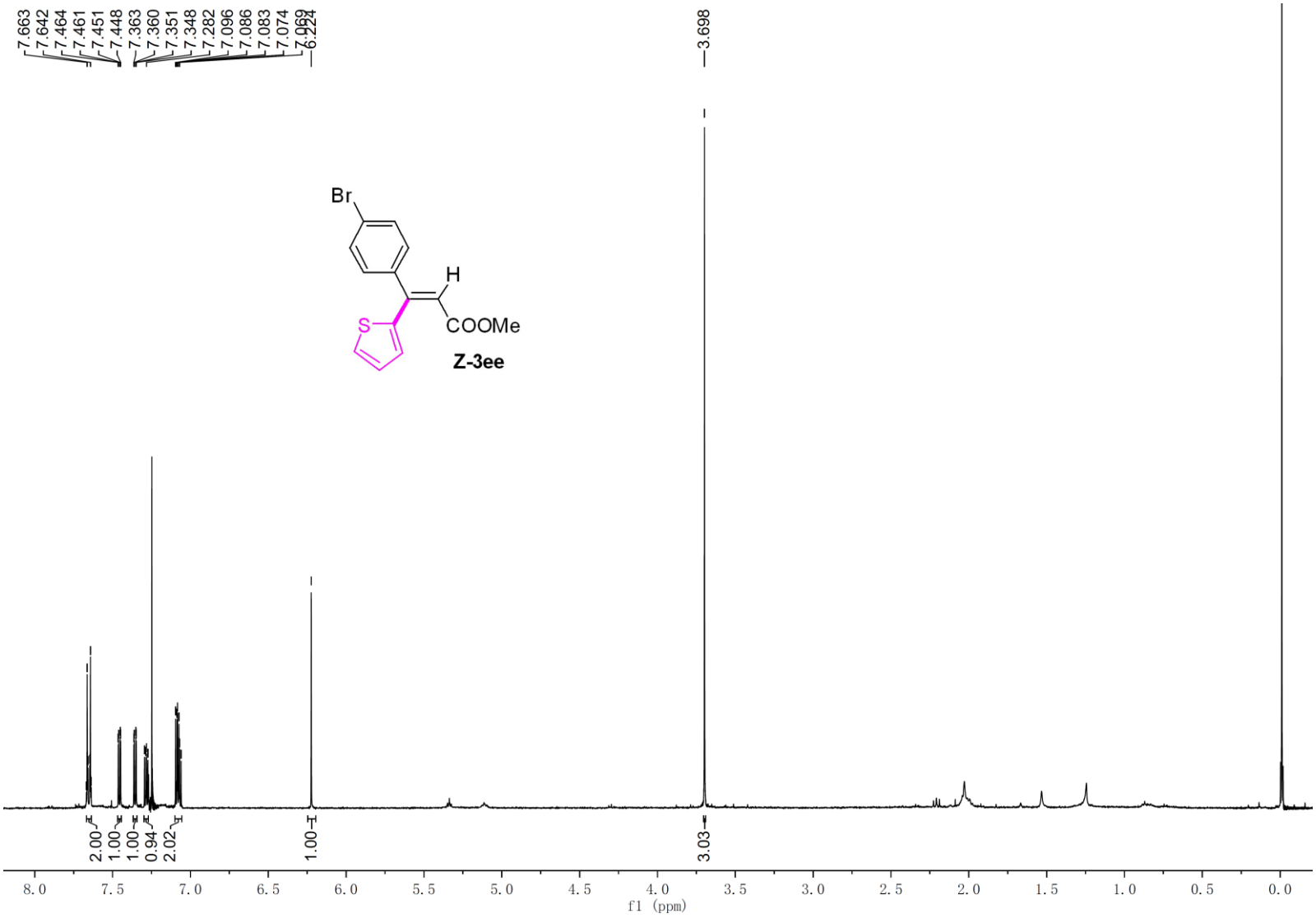
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.351
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7.282
7.096
7.086
7.083
7.074
6.224
3.698
Br
H
COOMe
S
Z-3ee
2.00
1.00
1.00
0.94
2.02
1.00
3.03
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
2.5
2.0
1.5
1.0
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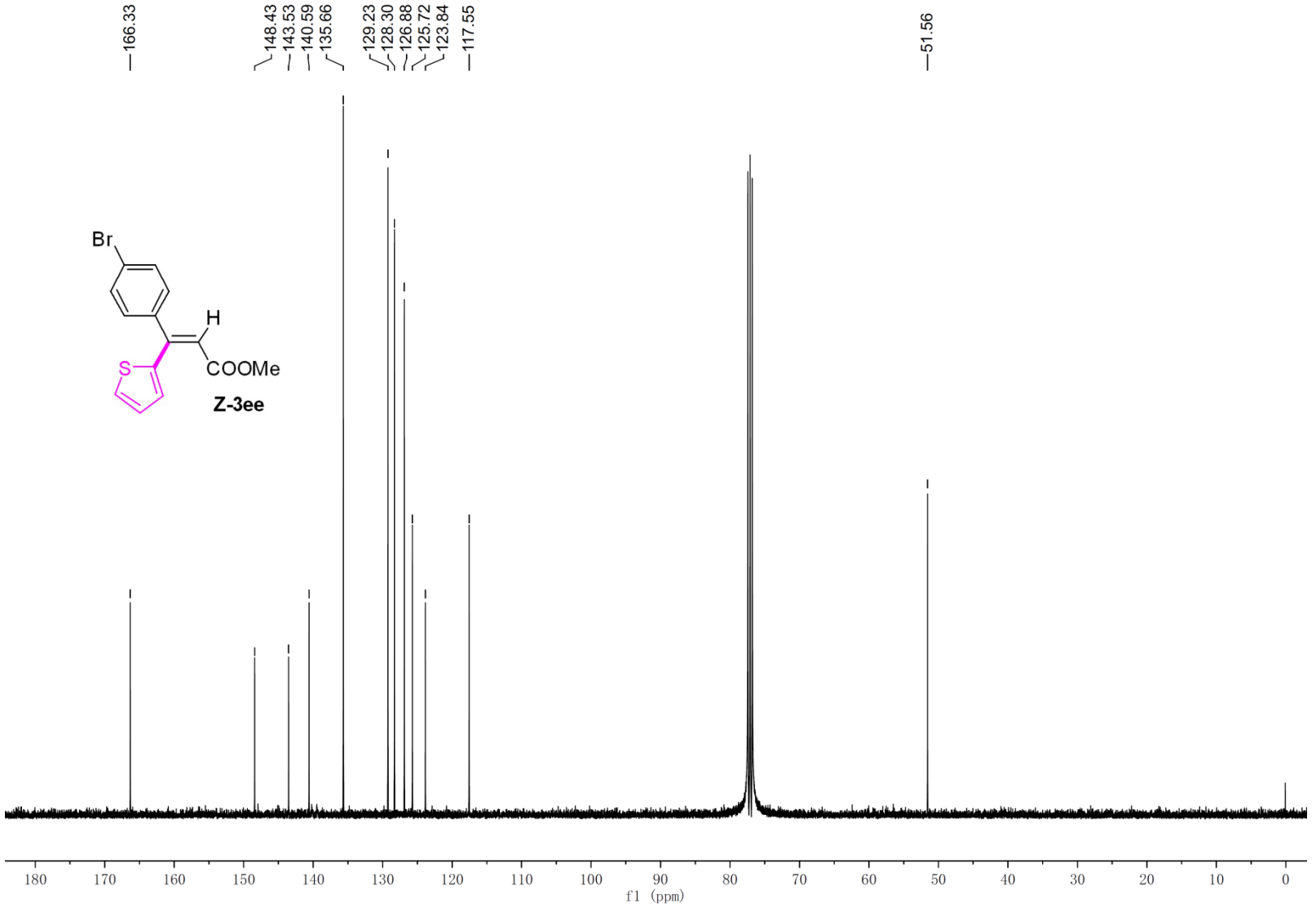
0.0
f1 (ppm)


166.33
148.43
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140.59
135.66
129.23
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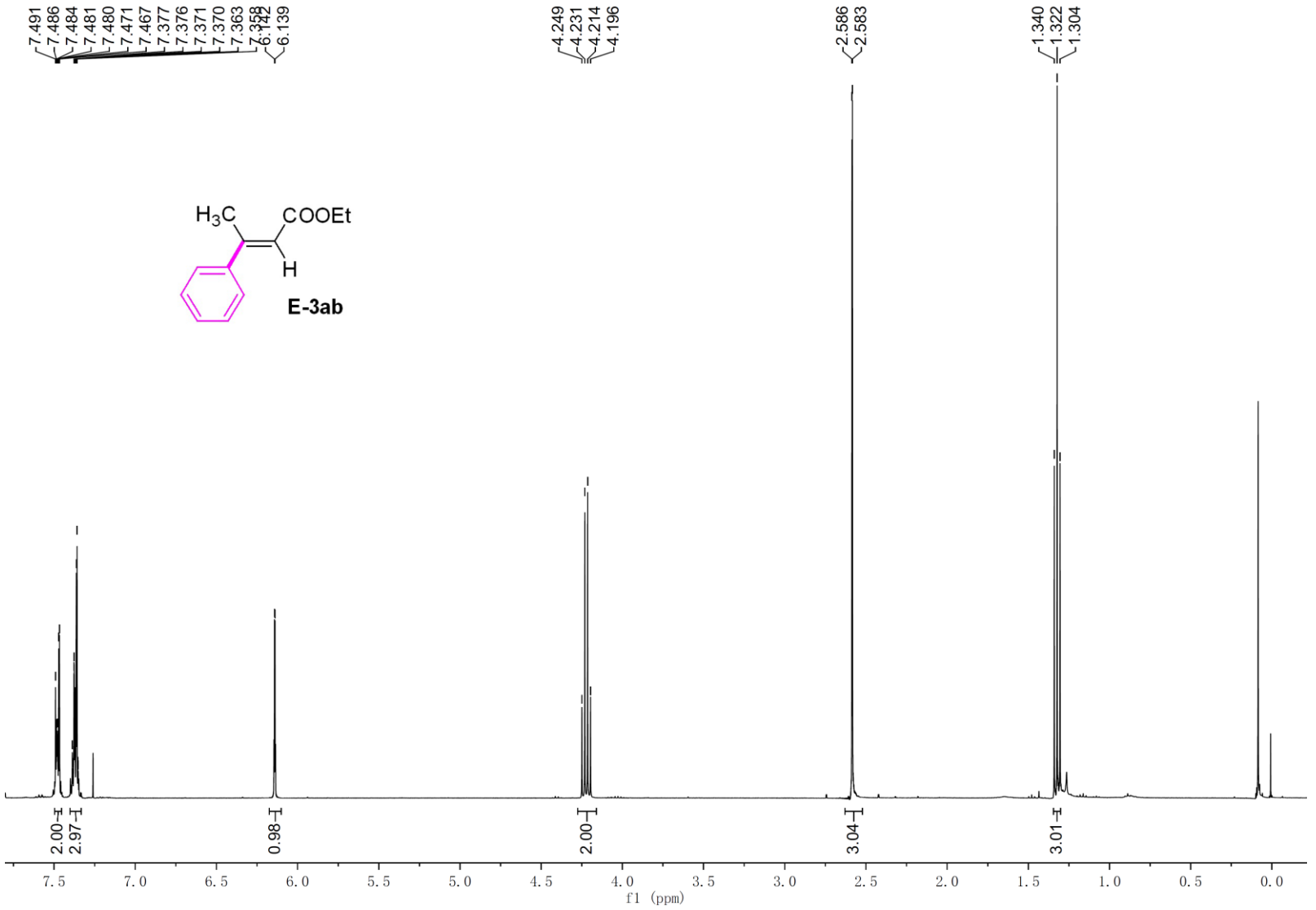
126.88
125.72
123.84
117.55
51.56
Br
H
COOMe
S
Z-3ee
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.371
7.370
7.363
7.358
6.142
6.139
4.249
4.231
4.214
4.196
2.586
2.583
1.340
1.322
1.304
H3C
COOEt
H
E-3ab
2.00
2.97
0.98
2.00
3.04
3.01
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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3.5
3.0
2.5
2.0
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1.0
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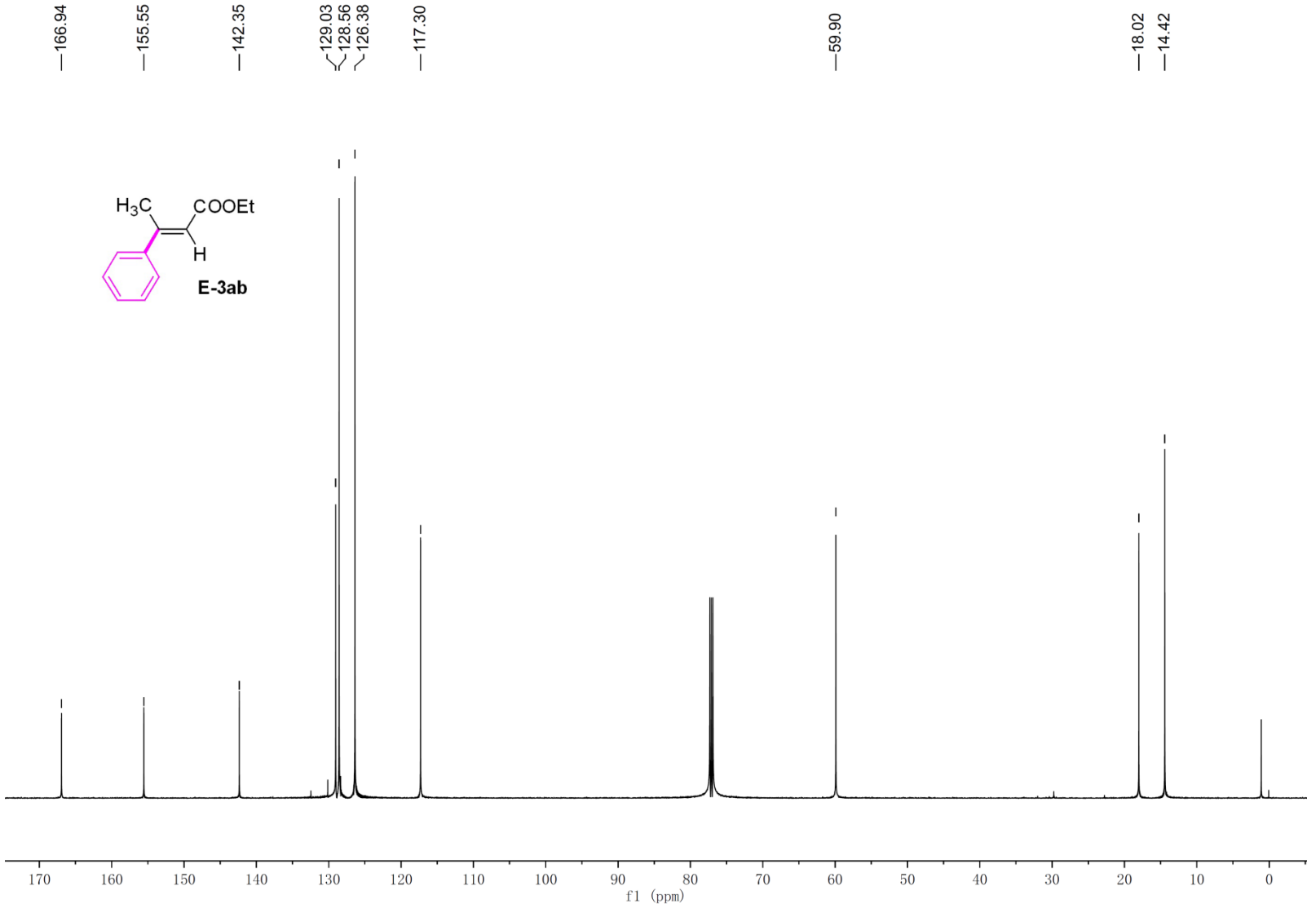
0.0
f1 (ppm)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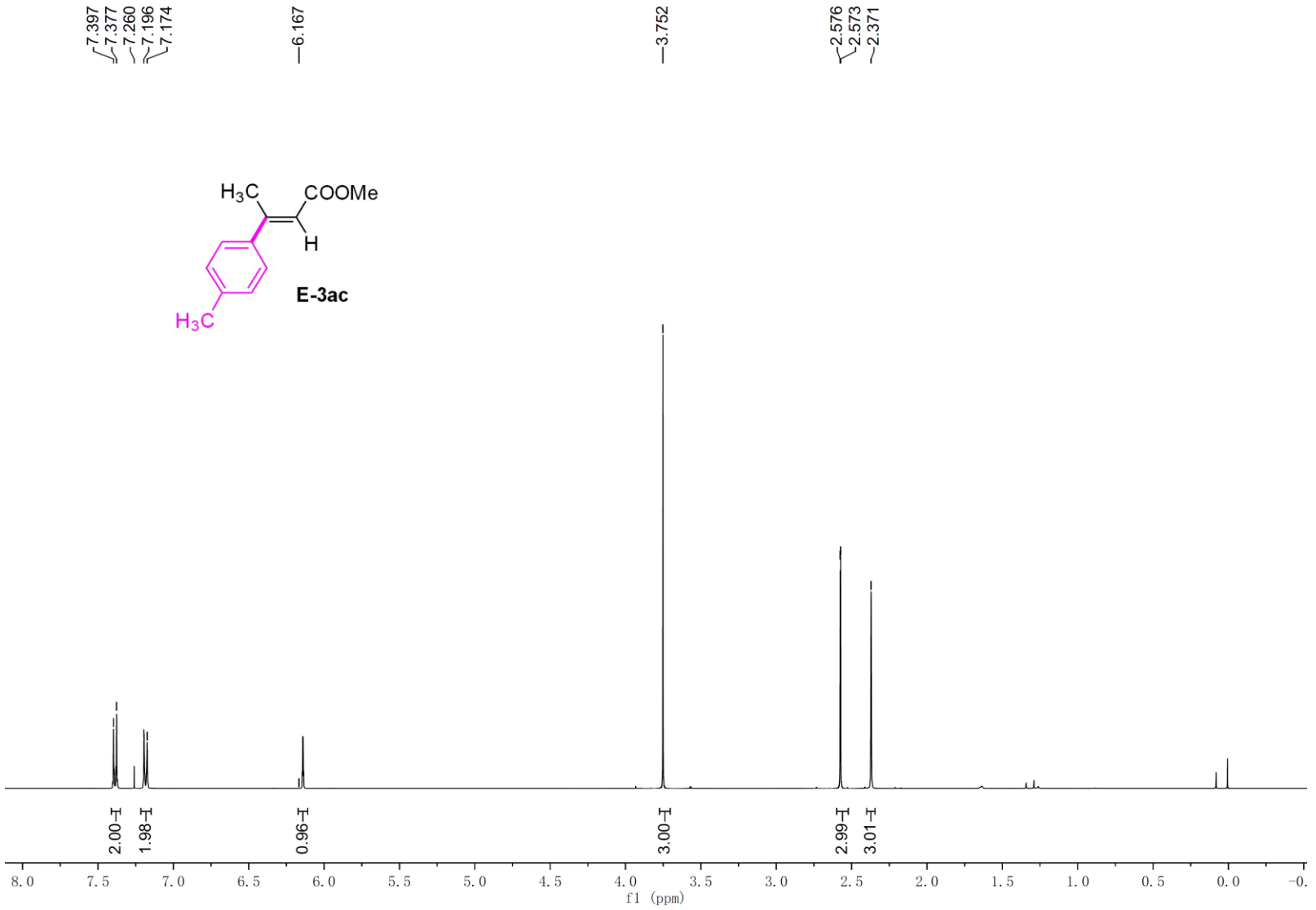
142.35
129.03
128.56
126.38
117.30
59.90
18.02
14.42
H3C
COOEt
H
E-3ab
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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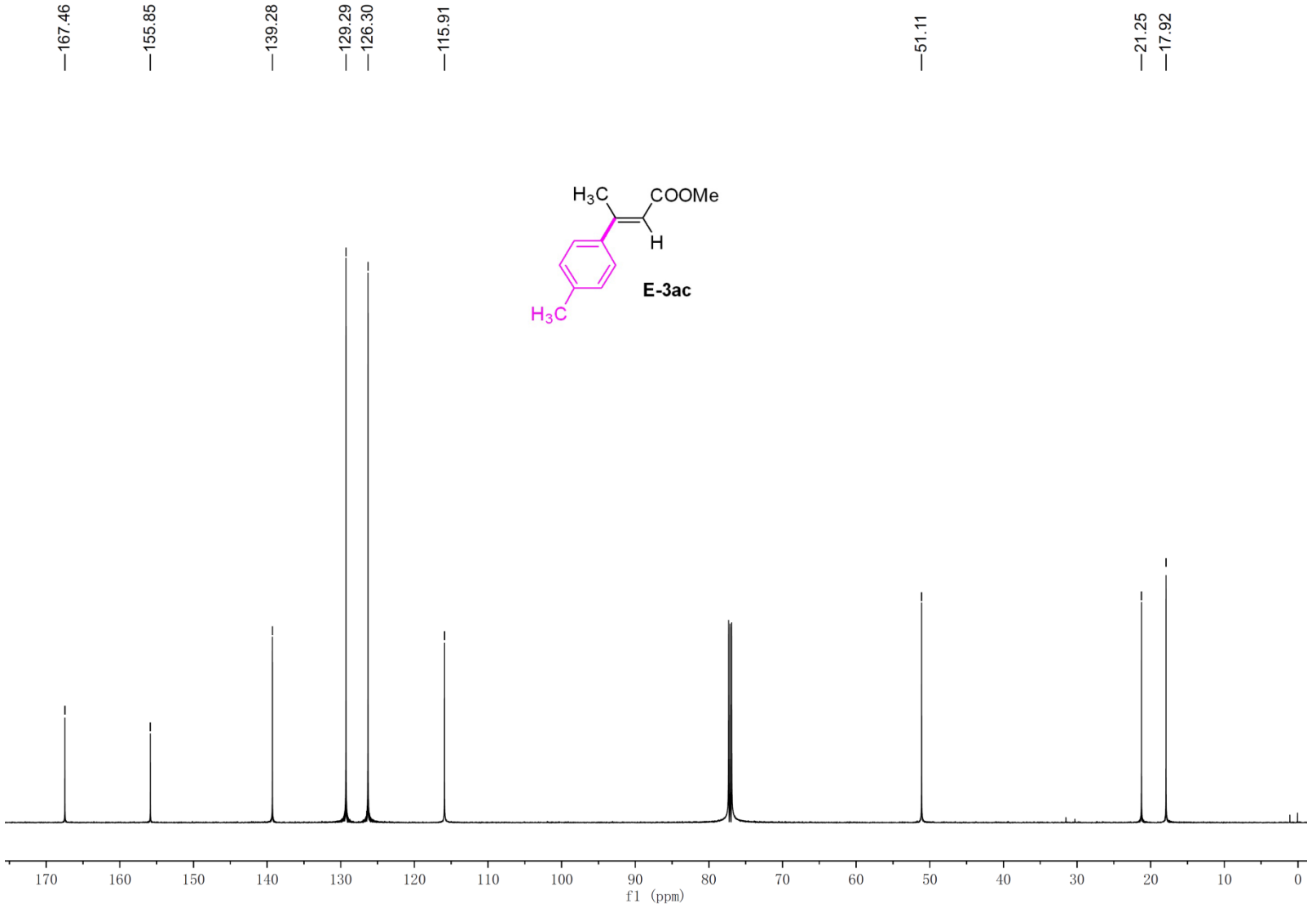
7.377
7.260
7.196
7.174
6.167
3.752
2.576
2.573
2.371
H3C
COOMe
H
H3C
E-3ac
2.00
1.98
0.96
3.00
2.99
3.01
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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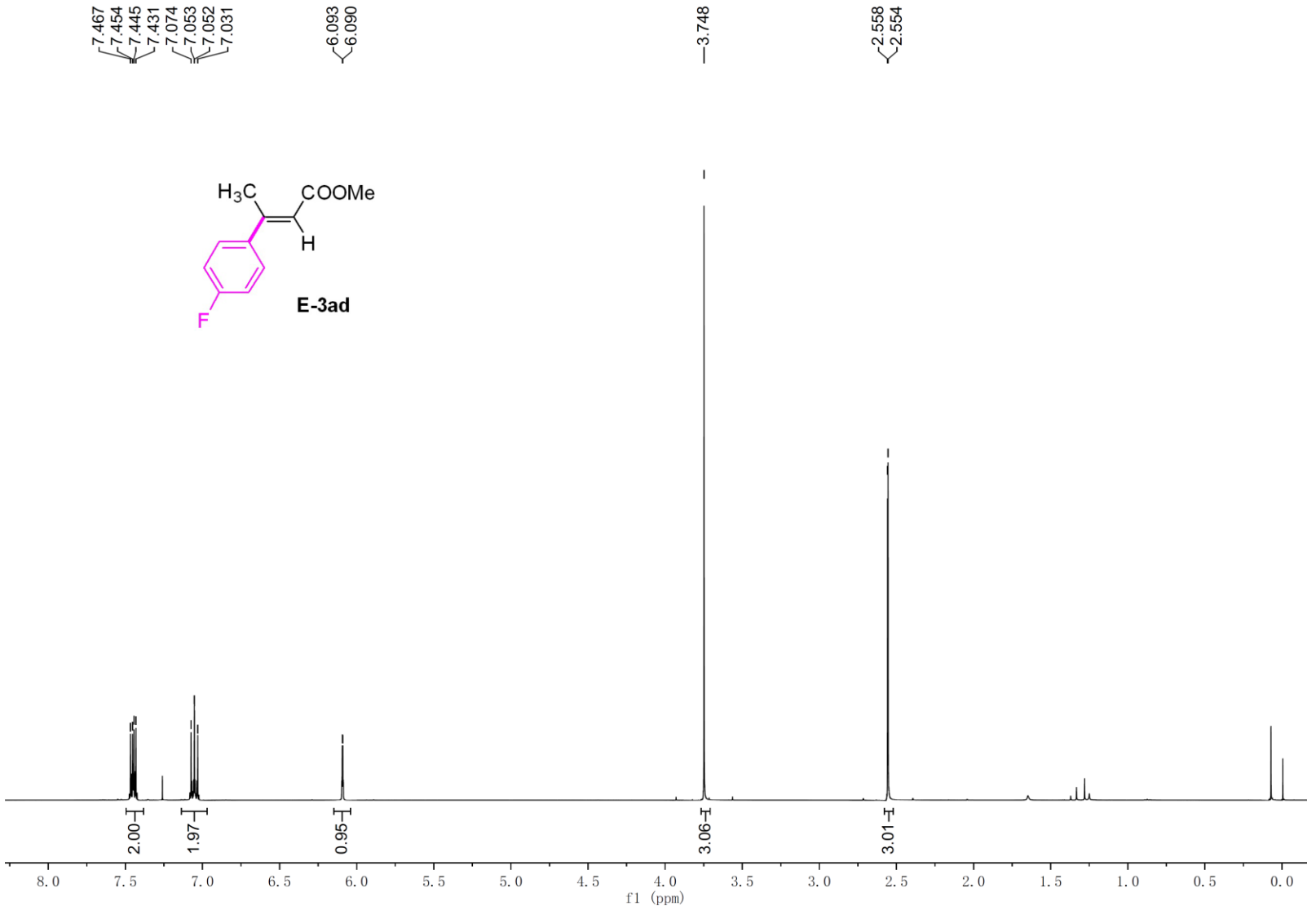
155.85
139.28
129.29
126.30
115.91
51.11
21.25
17.92
H3C
COOMe
H
H3C
E-3ac
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.454
7.445
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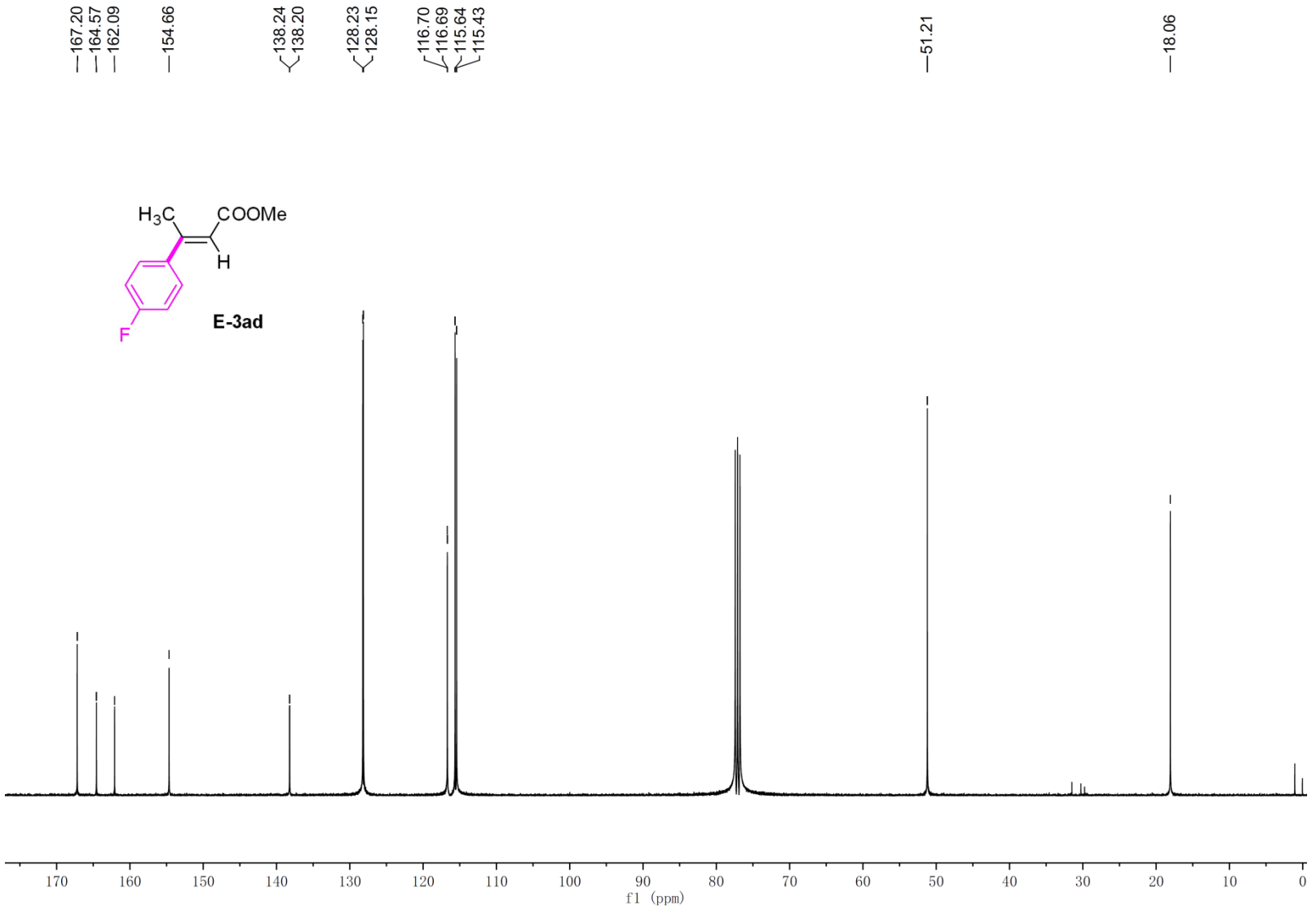
7.074
7.053
7.052
7.031
6.093
6.090
3.748
2.558
2.554
H3C
COOMe
H
F
E-3ad
2.00
1.97
0.95
3.06
3.01
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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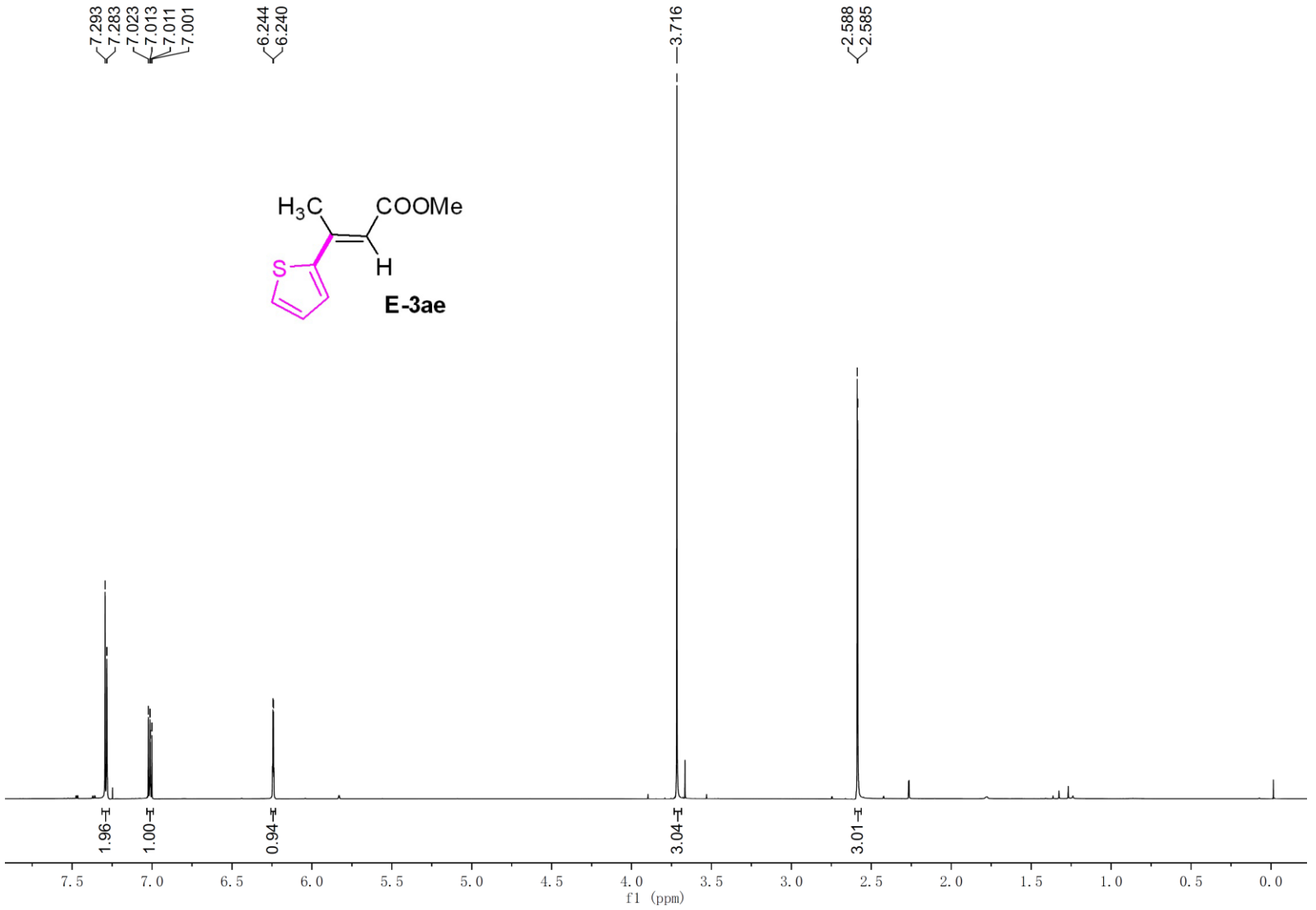
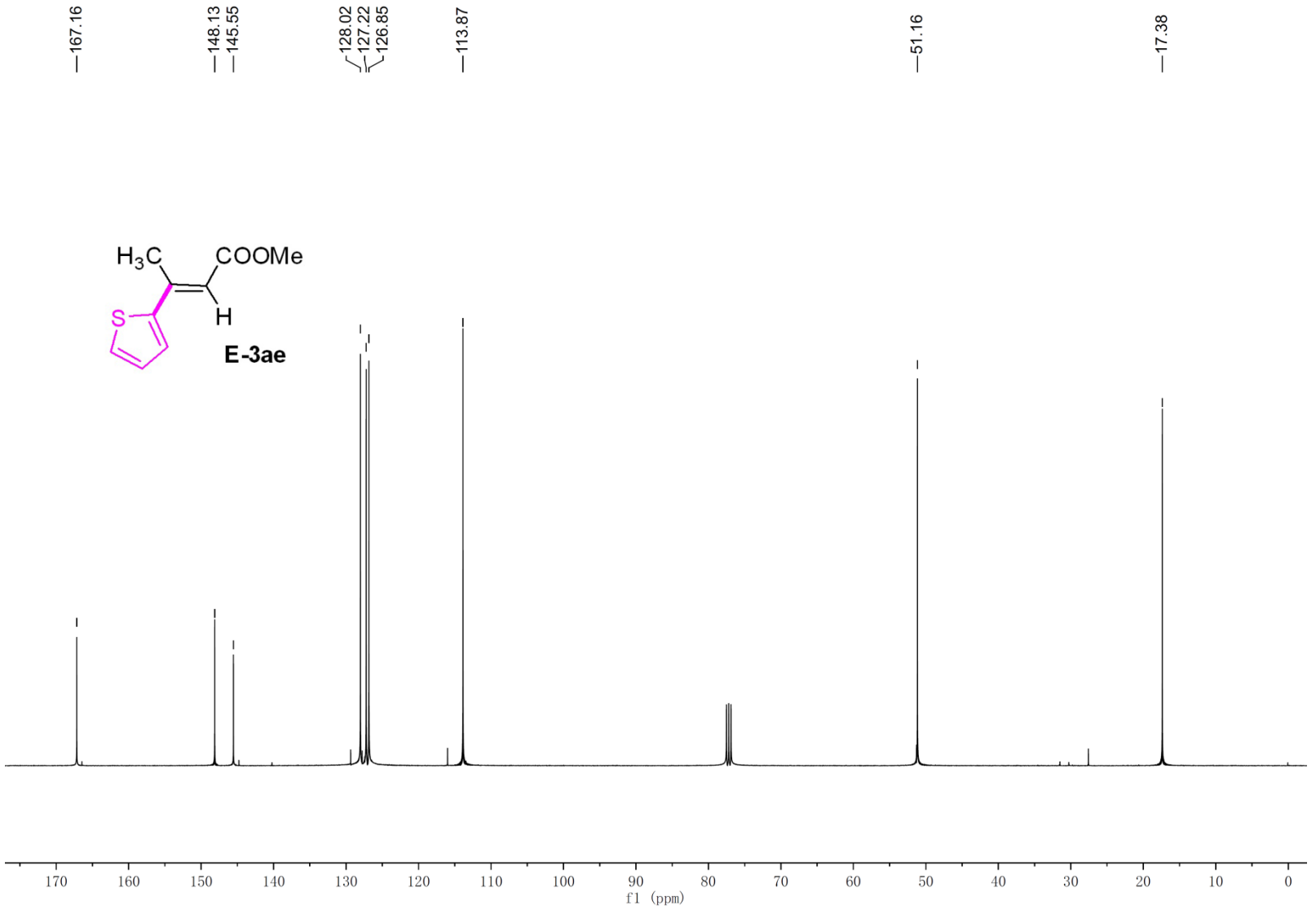
164.57
162.09
154.66
138.24
138.20
128.23
128.15
116.70
116.69
115.64
115.43
51.21
18.06
H3C
COOMe
H
F
E-3ad
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.293
7.283
7.023
7.013
7.011
7.001
6.244
6.240
3.716
2.588
2.585
H3C
COOMe
S
H
E-3ae
1.96
1.00
0.94
3.04
3.01
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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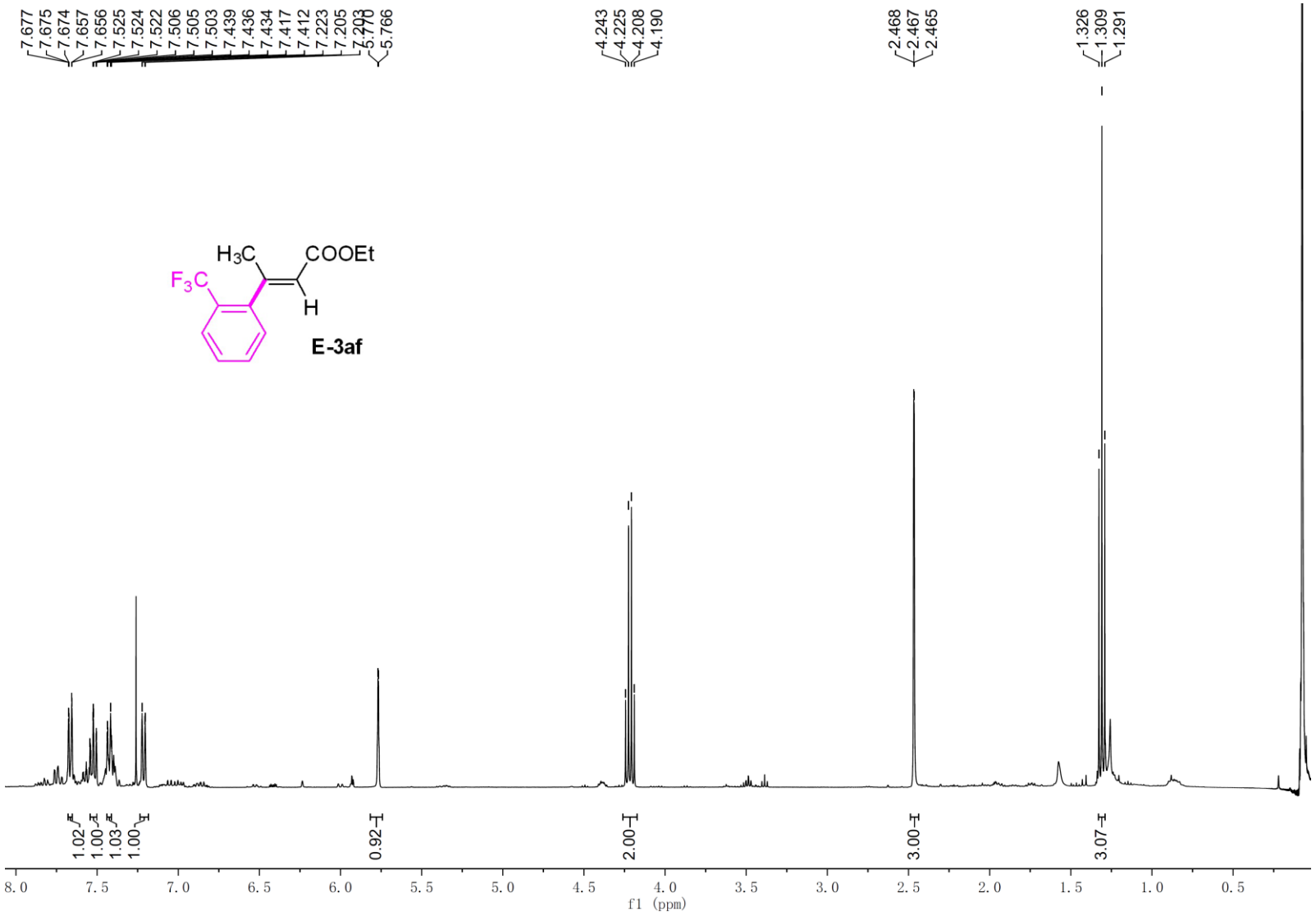
148.13
145.55
128.02
127.22
126.85
113.87
51.16
17.38
H3C
COOMe
S
H
E-3ae
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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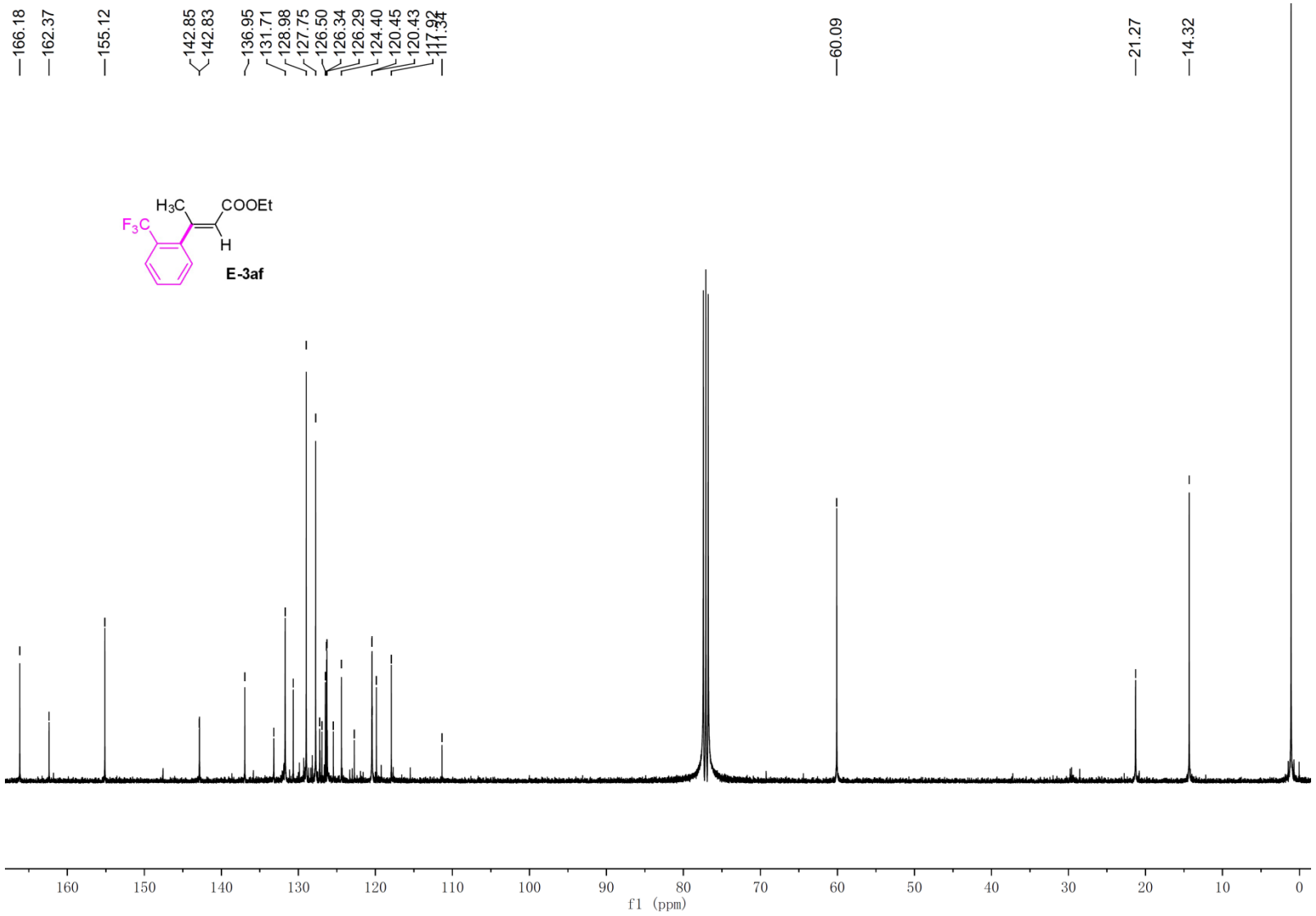
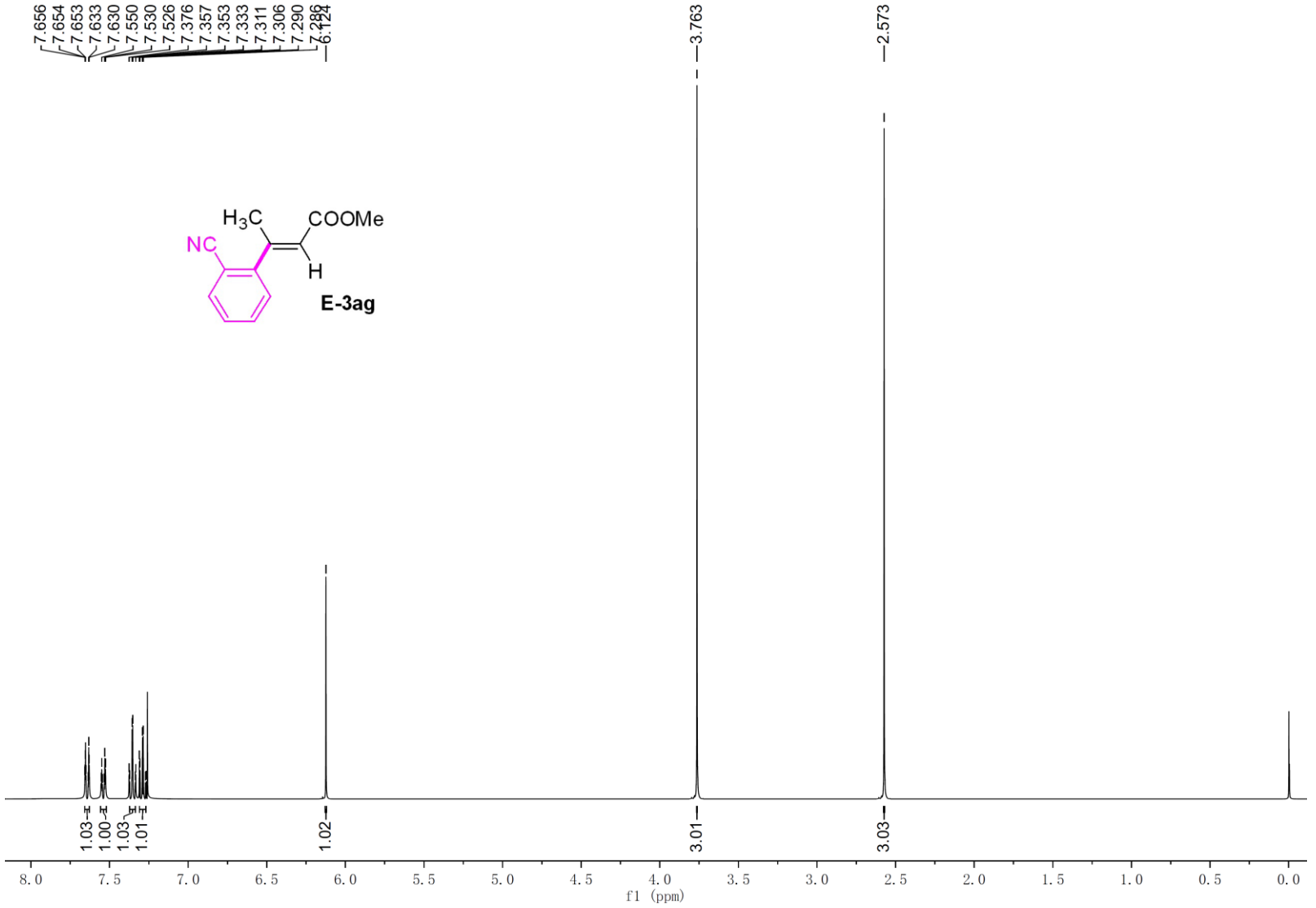
7.675
7.674
7.657
7.656
7.525
7.524
7.522
7.506
7.505
7.503
7.439
7.436
7.434
7.417
7.412
7.223
7.205
5.770
5.766
4.243
4.225
4.208
4.190
2.468
2.467
2.465
1.326
1.309
1.291
H3C
COOEt
F3C
H
E-3af
1.02
1.00
1.03
1.00
0.92
2.00
3.00
3.07
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


166.18
162.37
155.12
142.85
142.83
136.95
131.71
128.98
127.75
126.50
126.34
126.29
124.40
120.45
120.43
117.92
117.34
60.09
21.27
14.32
H3C
COOEt
F3C
H
E-3af
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.653
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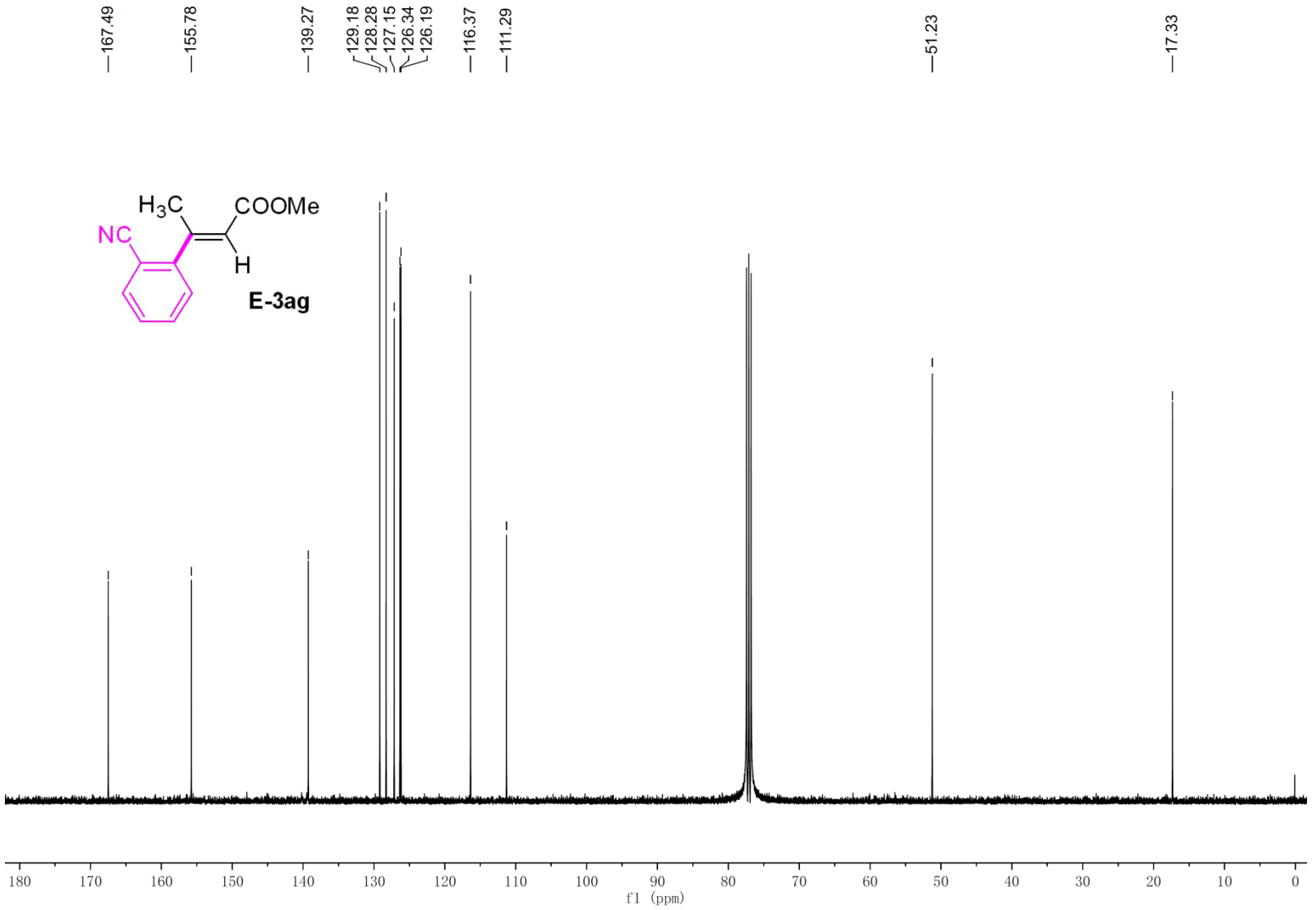
7.630
7.550
7.530
7.526
7.376
7.357
7.353
7.333
7.311
7.306
7.290
7.286
7.284
6.124
3.763
2.573
H3C
COOMe
NC
H
E-3ag
1.03
1.00
1.03
1.01
1.02
3.01
3.03
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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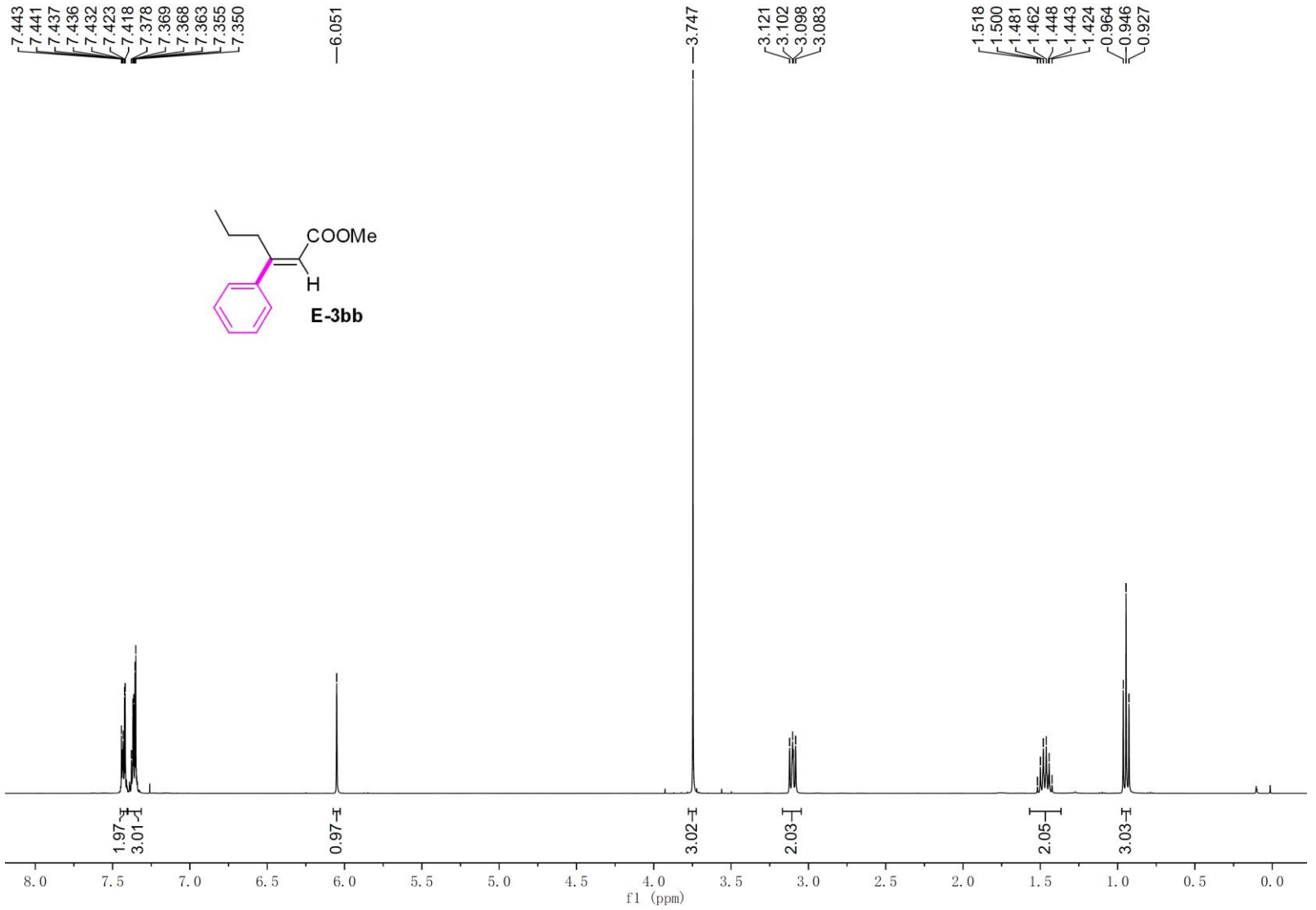
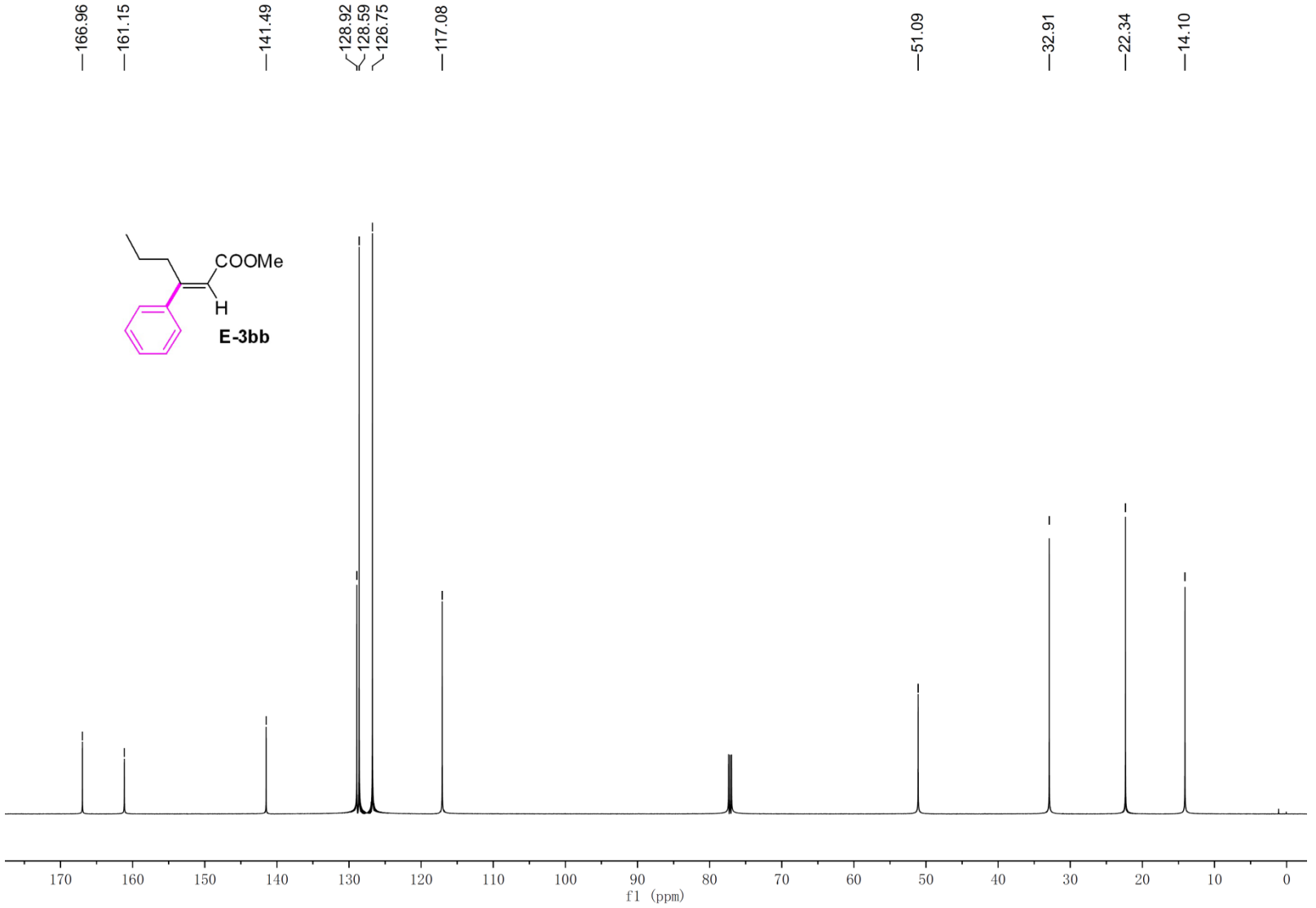
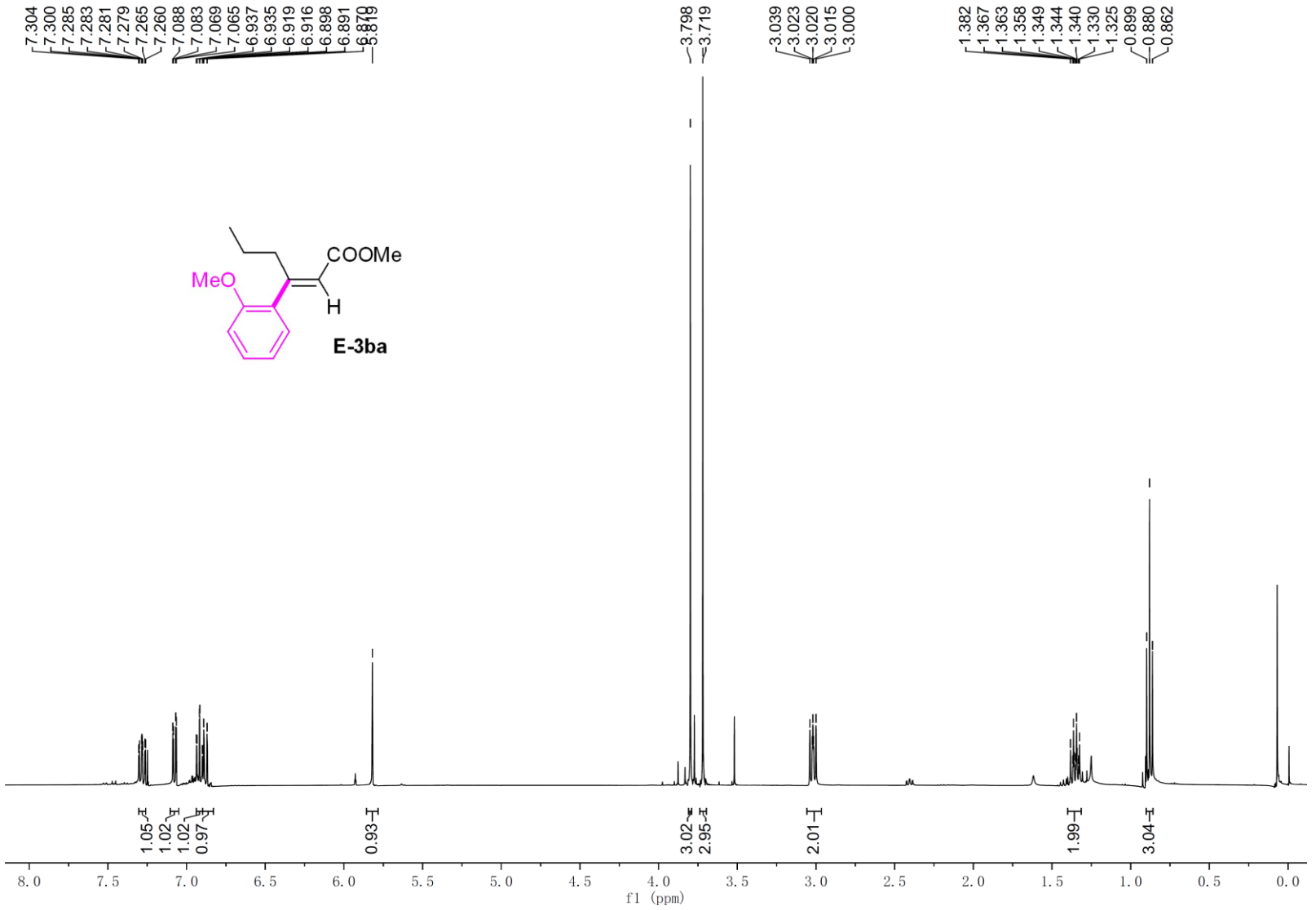
155.78
139.27
129.18
128.28
127.15
126.34
126.19
116.37
111.29
51.23
17.33
H3C
COOMe
NC
H
E-3ag
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.443
7.441
7.437
7.436
7.432
7.423
7.418
7.378
7.369
7.368
7.363
7.355
7.350
6.051
3.747
3.121
3.102
3.098
3.083
1.518
1.500
1.481
1.462
1.448
1.443
1.424
0.964
0.946
0.927
COOMe
H
E-3bb
1.97
3.01
0.97
3.02
2.03
2.05
3.03
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


166.96
161.15
141.49
128.92
128.59
126.75
117.08
51.09
32.91
22.34
14.10
COOMe
H
E-3bb
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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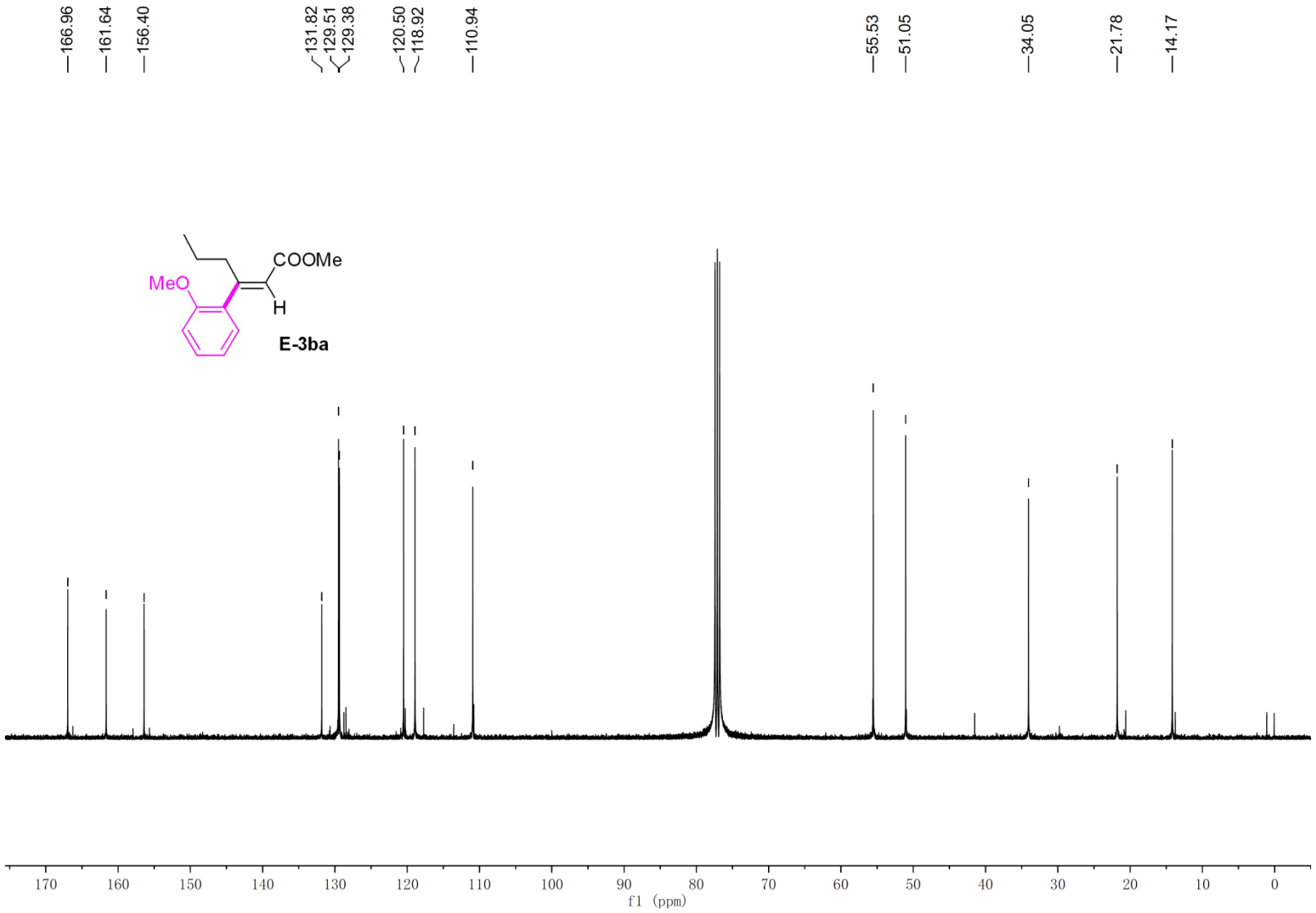
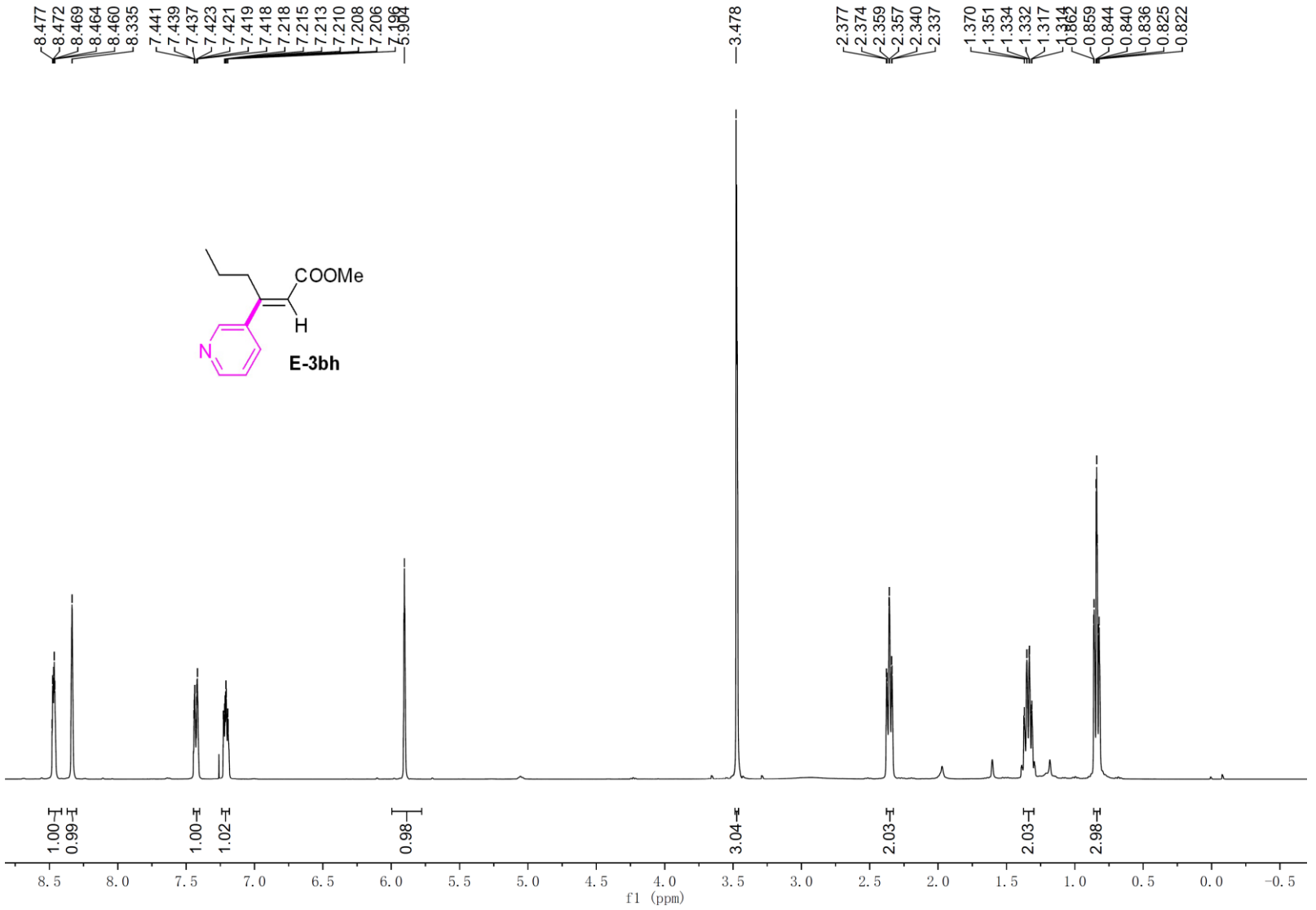
7.283
7.281
7.279
7.265
7.260
7.088
7.083
7.069
7.065
6.937
6.935
6.919
6.916
6.898
6.891
6.870
6.819
3.798
3.719
3.039
3.023
3.020
3.015
3.000
1.382
1.367
1.363
1.358
1.349
1.344
1.340
1.330
1.325
0.899
0.880
0.862
COOMe
MeO
H
E-3ba
1.05
1.02
1.02
0.97
0.93
3.02
2.95
2.01
1.99
3.04
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


166.96
161.64
156.40
131.82
129.51
129.38
120.50
118.92
110.94
55.53
51.05
34.05
21.78
14.17
COOMe
MeO
H
E-3ba
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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8.460
8.335
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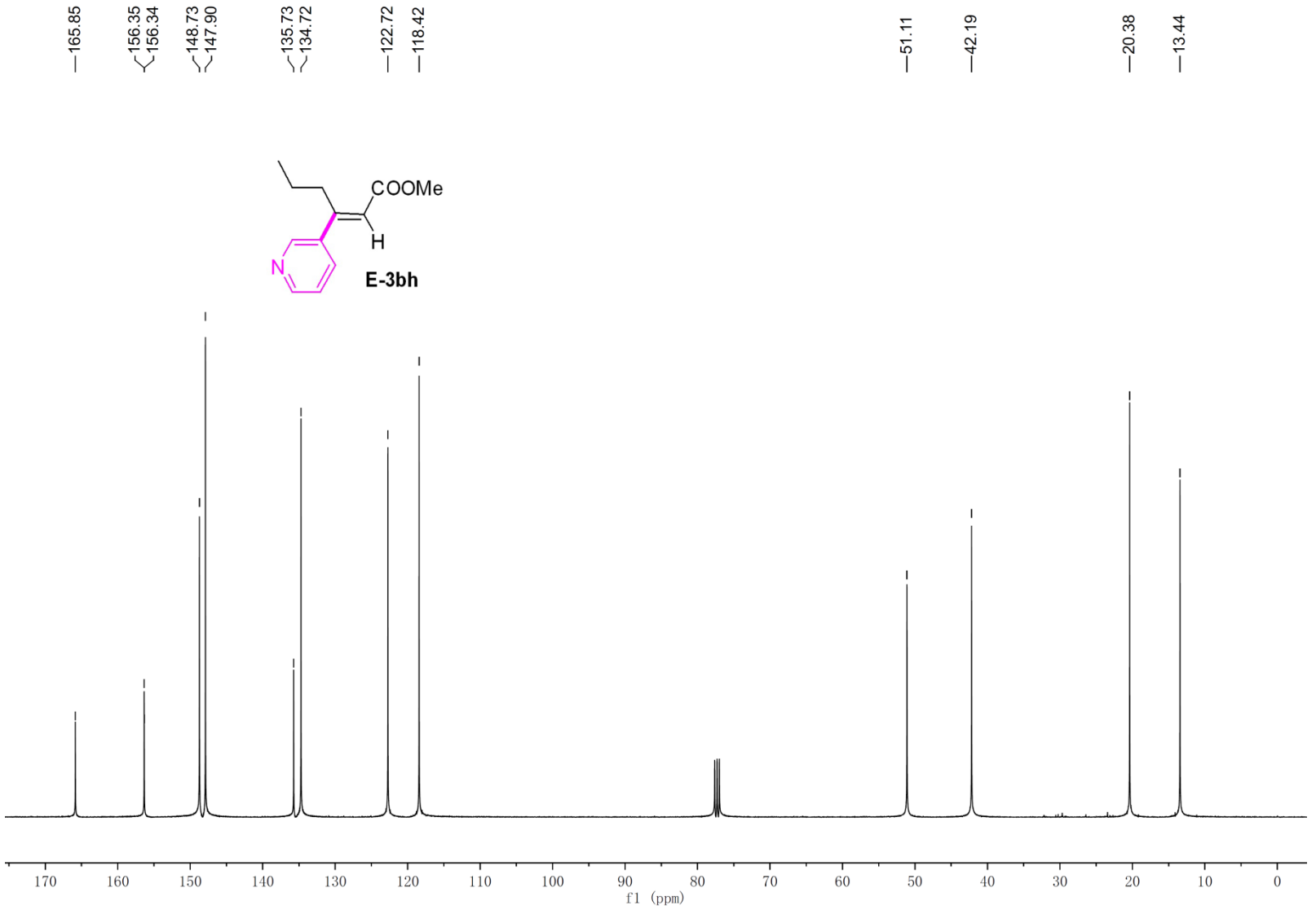
7.439
7.437
7.423
7.421
7.419
7.418
7.218
7.215
7.213
7.210
7.208
7.206
7.196
7.004
5.904
3.478
2.377
2.374
2.359
2.357
2.340
2.337
1.370
1.351
1.334
1.332
1.317
1.314
0.862
0.859
0.844
0.840
0.836
0.825
0.822
COOMe
H
N
E-3bh
1.00
0.99
1.00
1.02
0.98
3.04
2.03
2.03
2.98
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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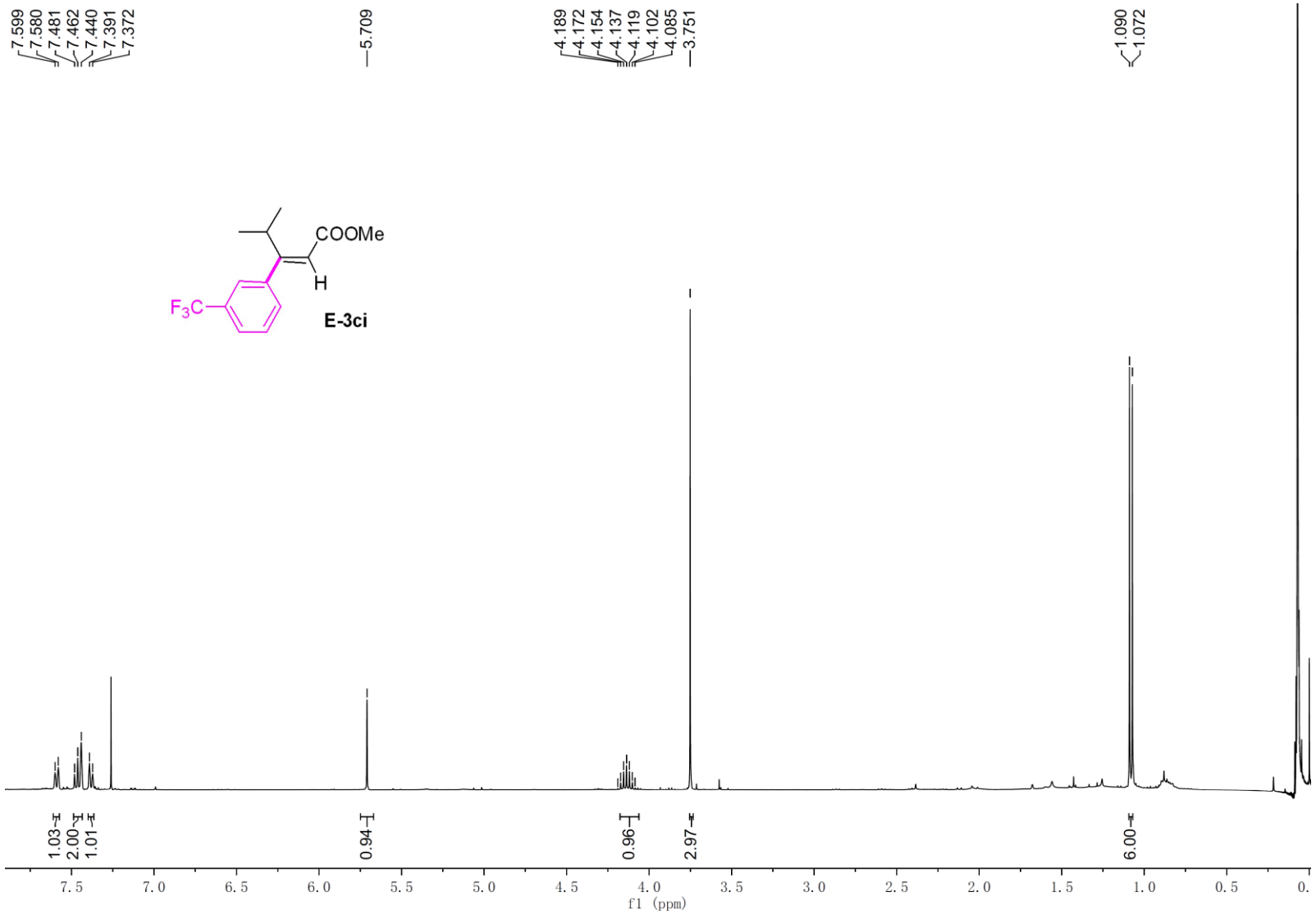
156.35
156.34
148.73
147.90
135.73
134.72
122.72
118.42
51.11
42.19
20.38
13.44
COOMe
H
N
E-3bh
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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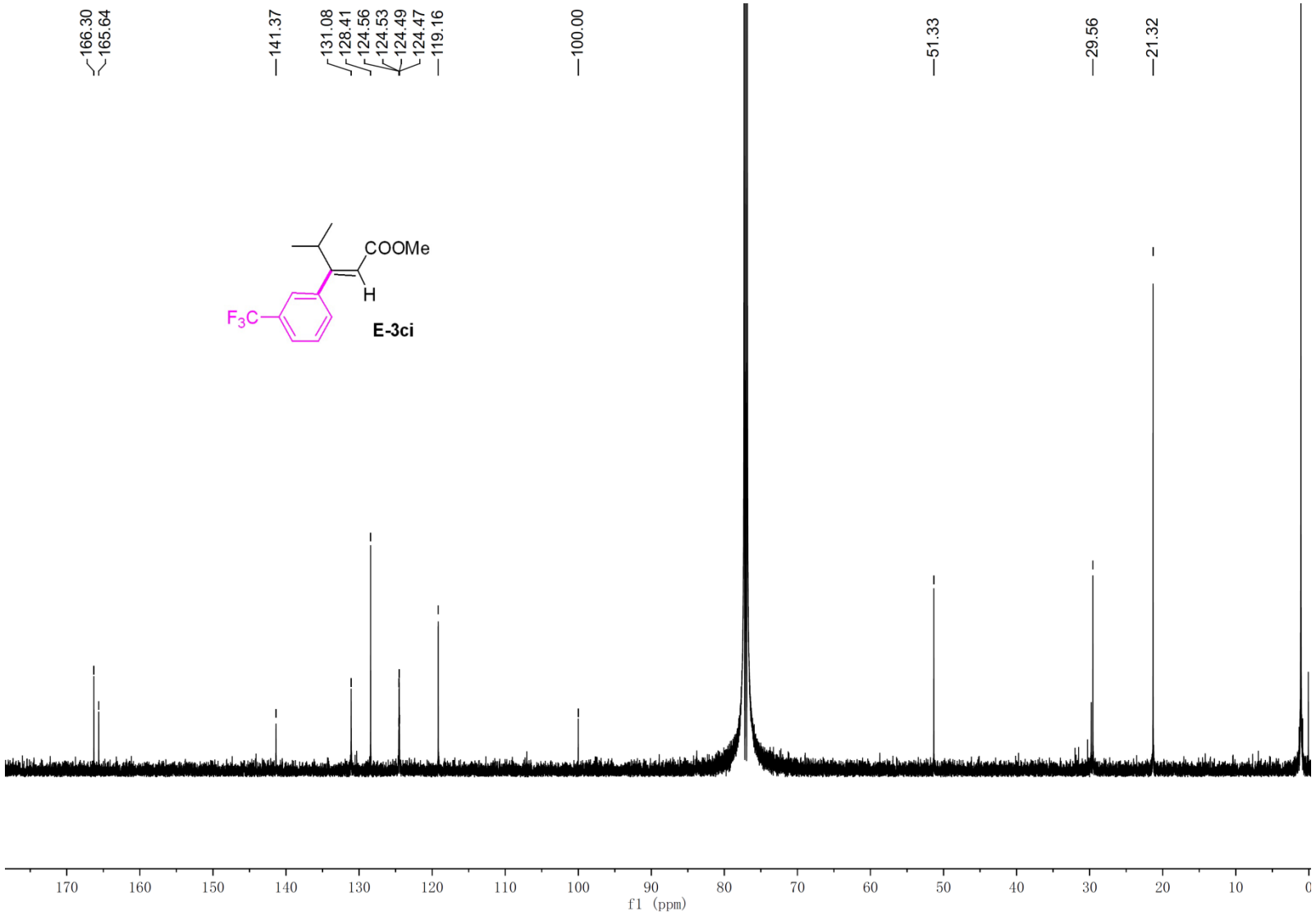
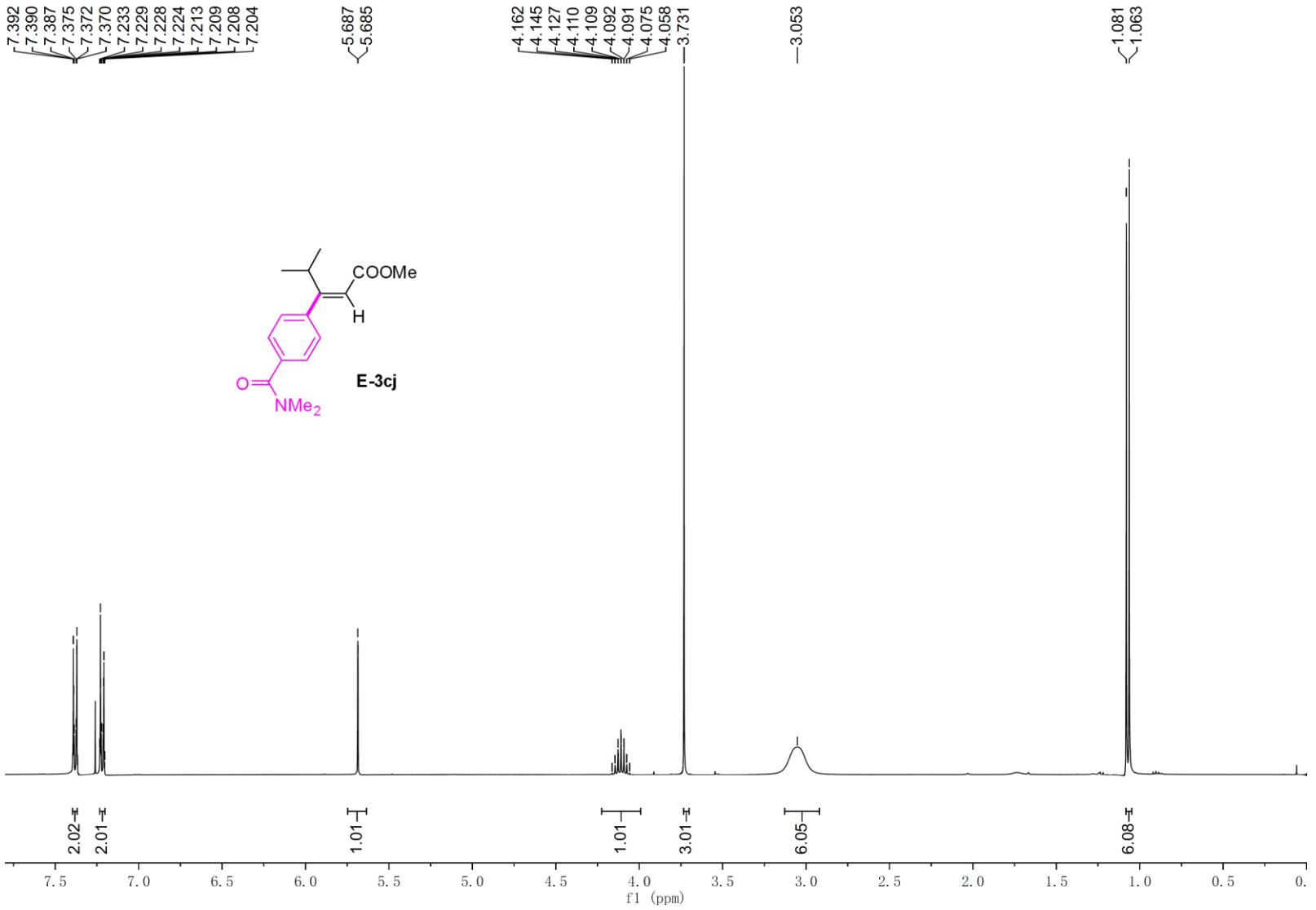
7.440
7.391
7.372
5.709
4.189
4.172
4.154
4.137
4.119
4.102
4.085
3.751
1.090
1.072
COOMe
H
F3C
E-3ci
1.03
2.00
1.01
0.94
0.96
2.97
6.00
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.
f1 (ppm)


166.30
165.64
141.37
131.08
128.41
124.56
124.53
124.49
124.47
119.16
100.00
51.33
29.56
21.32
COOMe
H
F3C
E-3ci
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.370
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7.229
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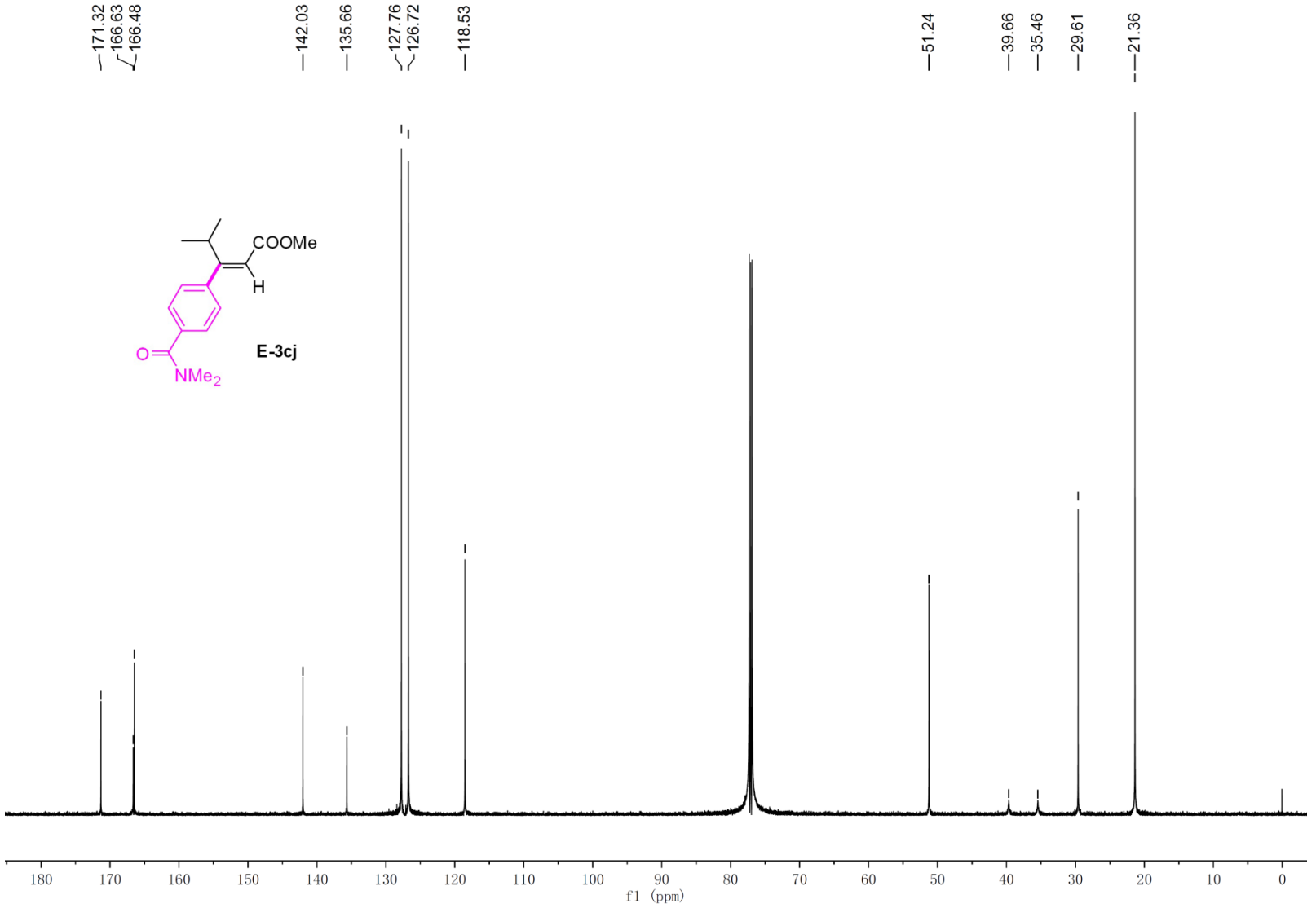
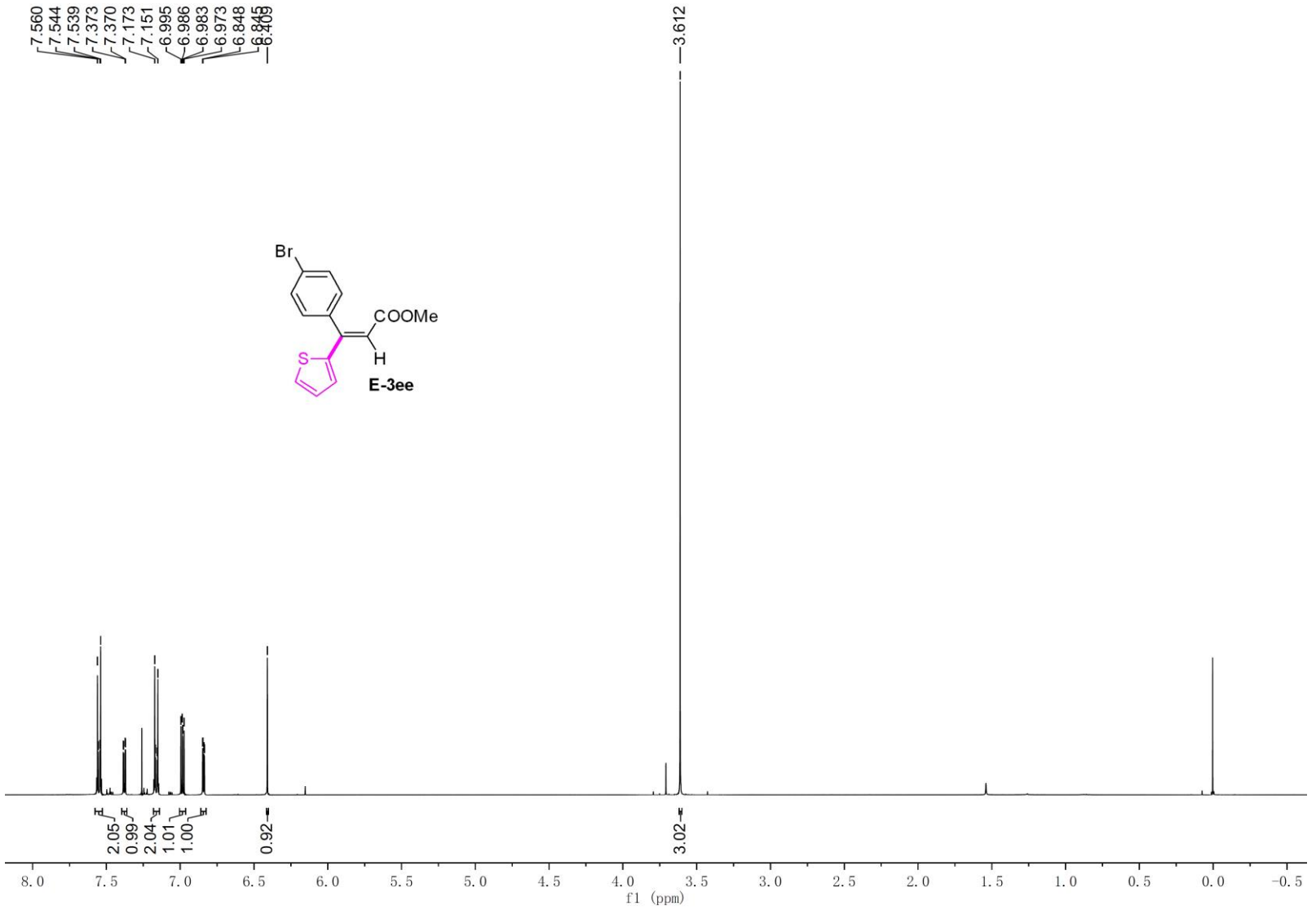
7.224
7.213
7.209
7.208
7.204
5.687
5.685
4.162
4.145
4.127
4.110
4.109
4.092
4.091
4.075
4.058
3.731
3.053
1.081
1.063
COOMe
H
O
NMe2
E-3cj
2.02
2.01
1.01
1.01
3.01
6.05
6.08
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.
f1 (ppm)


171.32
166.63
166.48
142.03
135.66
127.76
126.72
118.53
51.24
39.66
35.46
29.61
21.36
COOMe
H
O
NMe2
E-3cj
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.560
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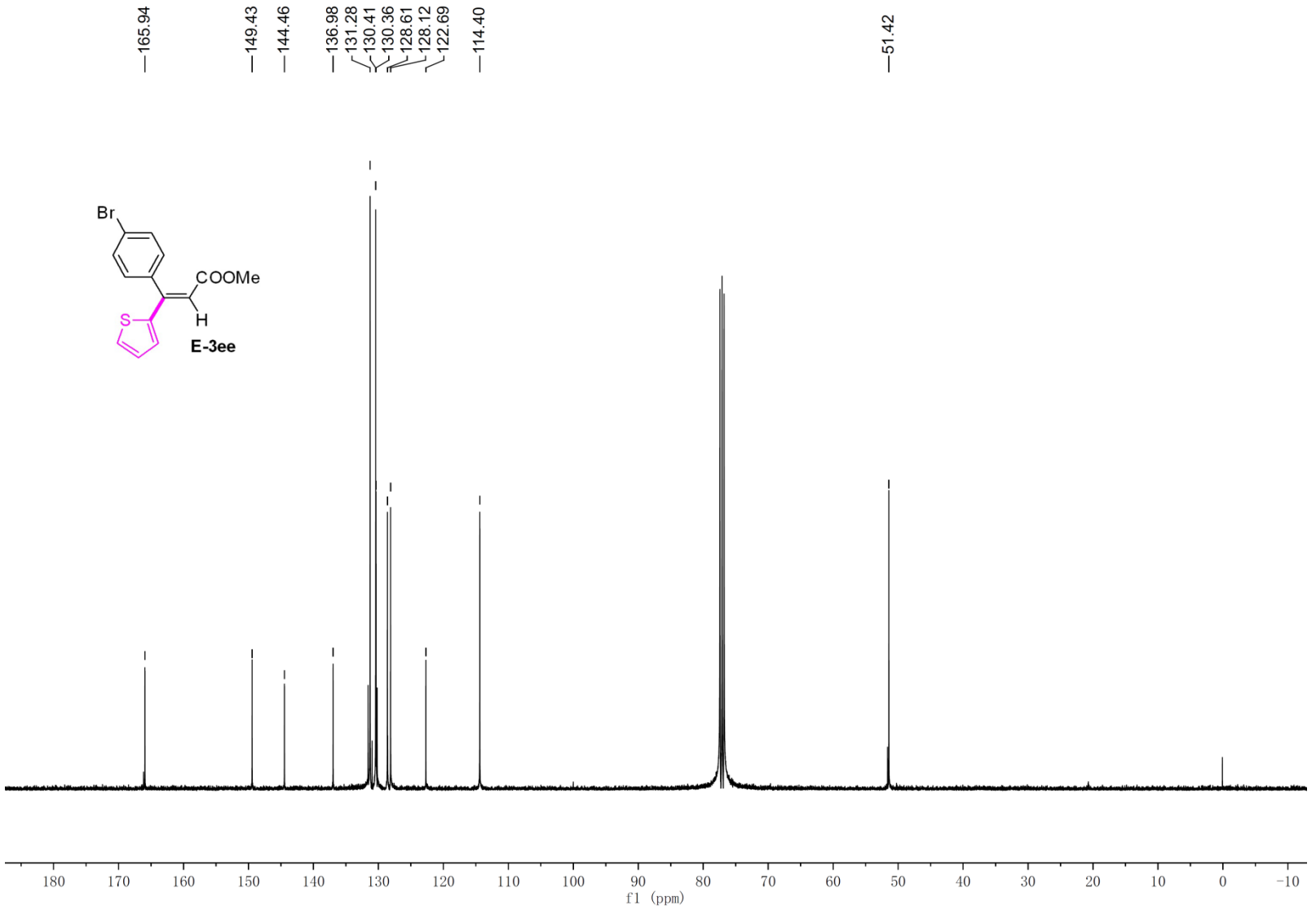
7.539
7.373
7.370
7.173
7.151
6.995
6.986
6.983
6.973
6.848
6.845
6.409
3.612
Br
COOMe
H
S
E-3ee
2.05
0.99
2.04
1.01
1.00
0.92
3.02
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


165.94
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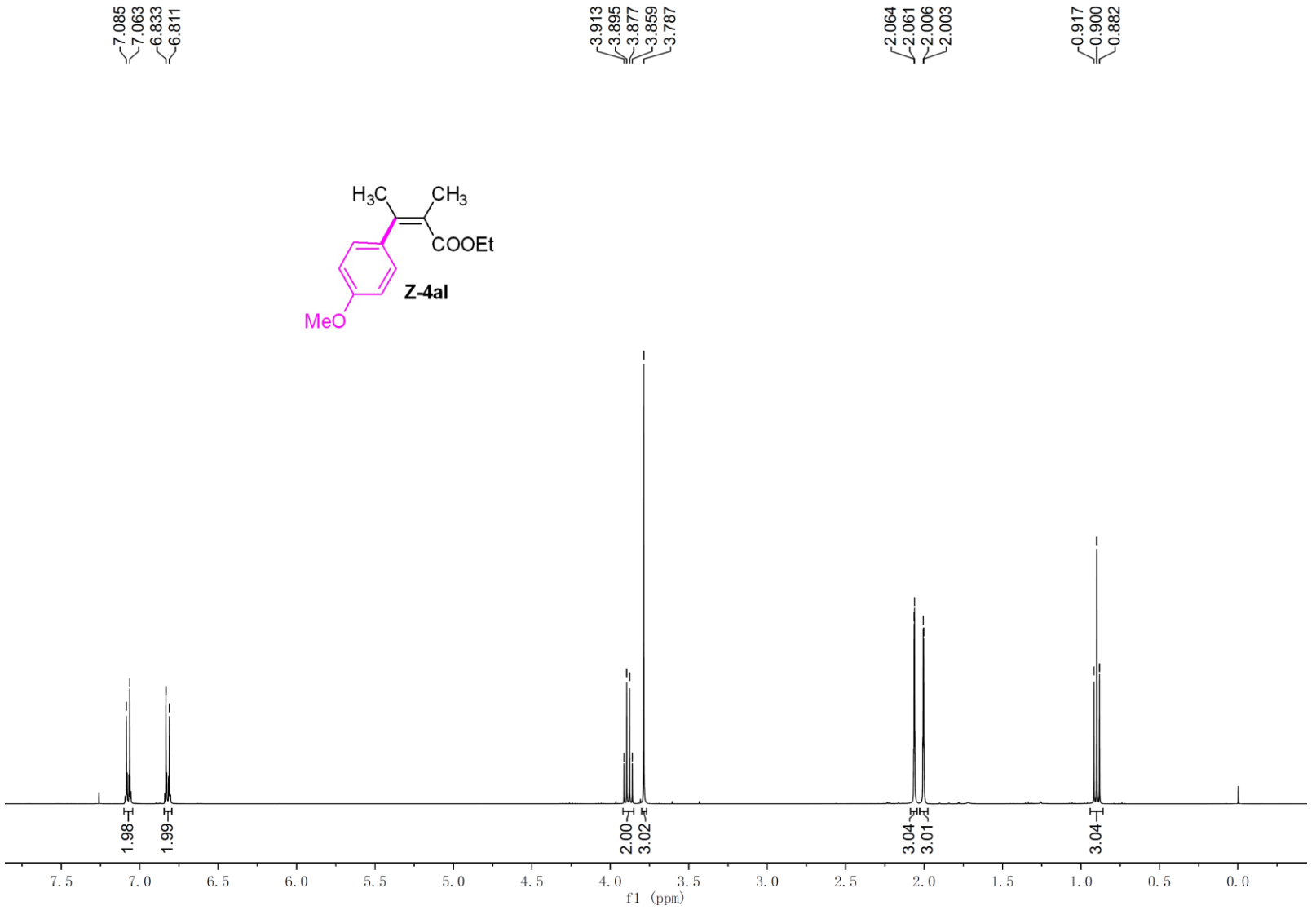
144.46
136.98
131.28
130.41
130.36
128.61
128.12
122.69
114.40
51.42
Br
COOMe
H
S
E-3ee
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)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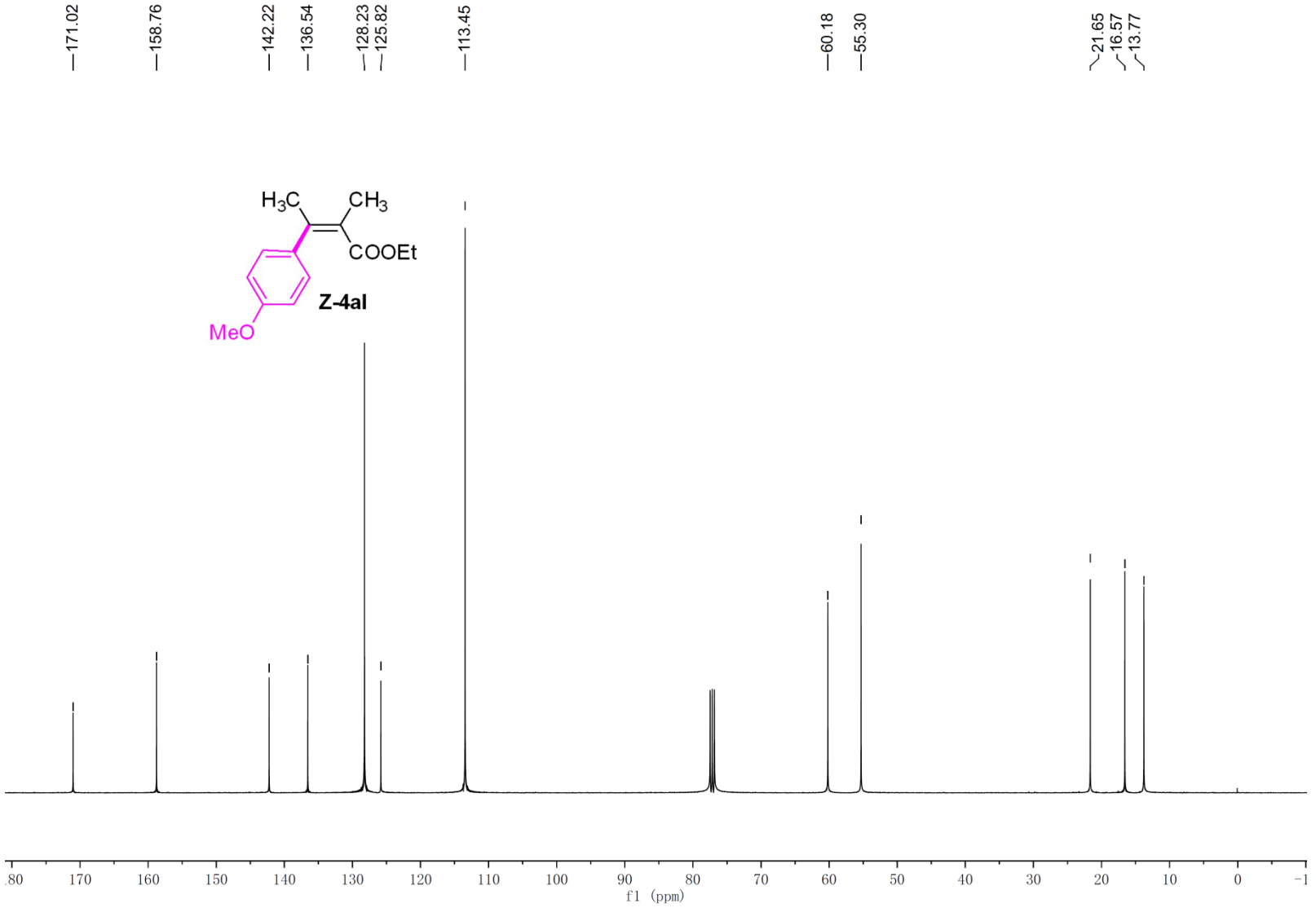
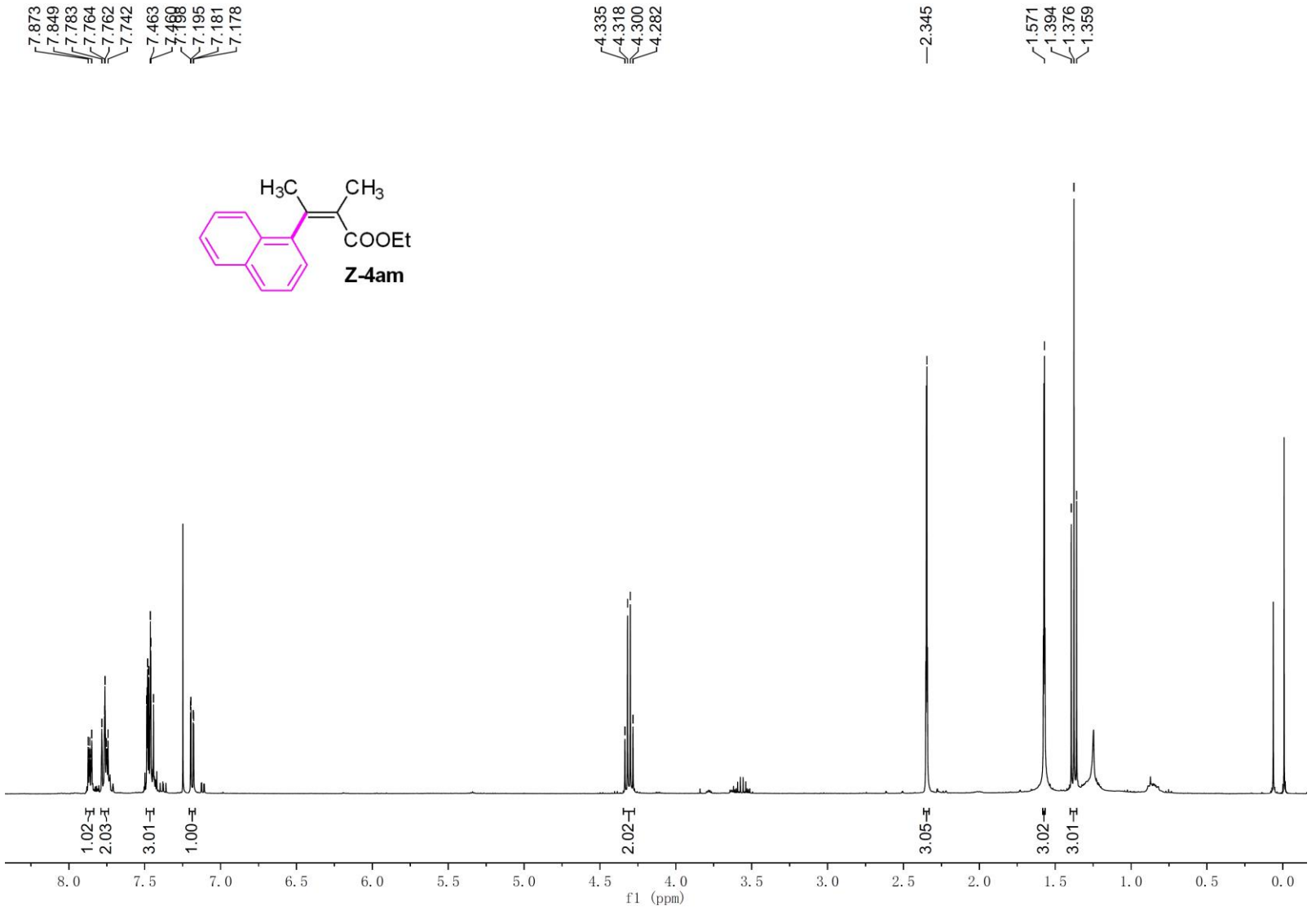
7.063
6.833
6.811
3.913
3.895
3.877
3.859
3.787
2.064
2.061
2.006
2.003
0.917
0.900
0.882
H3C
CH3
COOEt
MeO
Z-4al
1.98
1.99
2.00
3.02
3.04
3.01
3.04
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


171.02
158.76
142.22
136.54
128.23
125.82
113.45
60.18
55.30
21.65
16.57
13.77
H3C
CH3
COOEt
MeO
Z-4al
f1 (ppm)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7.849
7.783
7.764
7.762
7.742
7.463
7.460
7.195
7.181
7.178
4.335
4.318
4.300
4.282
2.345
1.571
1.394
1.376
1.359
H3C
CH3
COOEt
Z-4am
1.02
2.03
3.01
1.00
2.02
3.05
3.02
3.01
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
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2.5
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1.0
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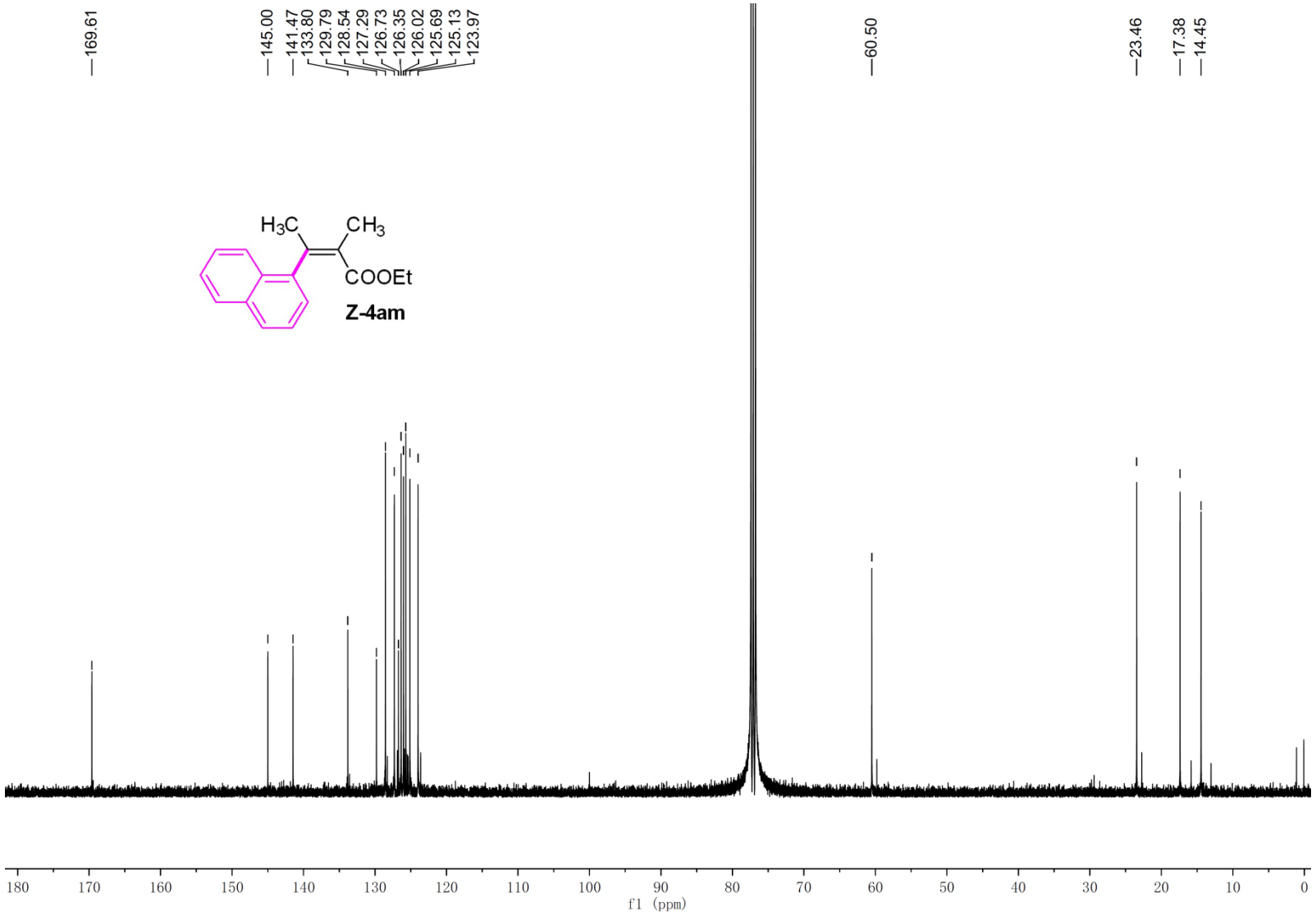
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126.02
125.69
125.13
123.97
60.50
23.46
17.38
14.45
H3C
CH3
COOEt
Z-4am
180
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140
130
120
110
100
90
80
70
60
50
40
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10
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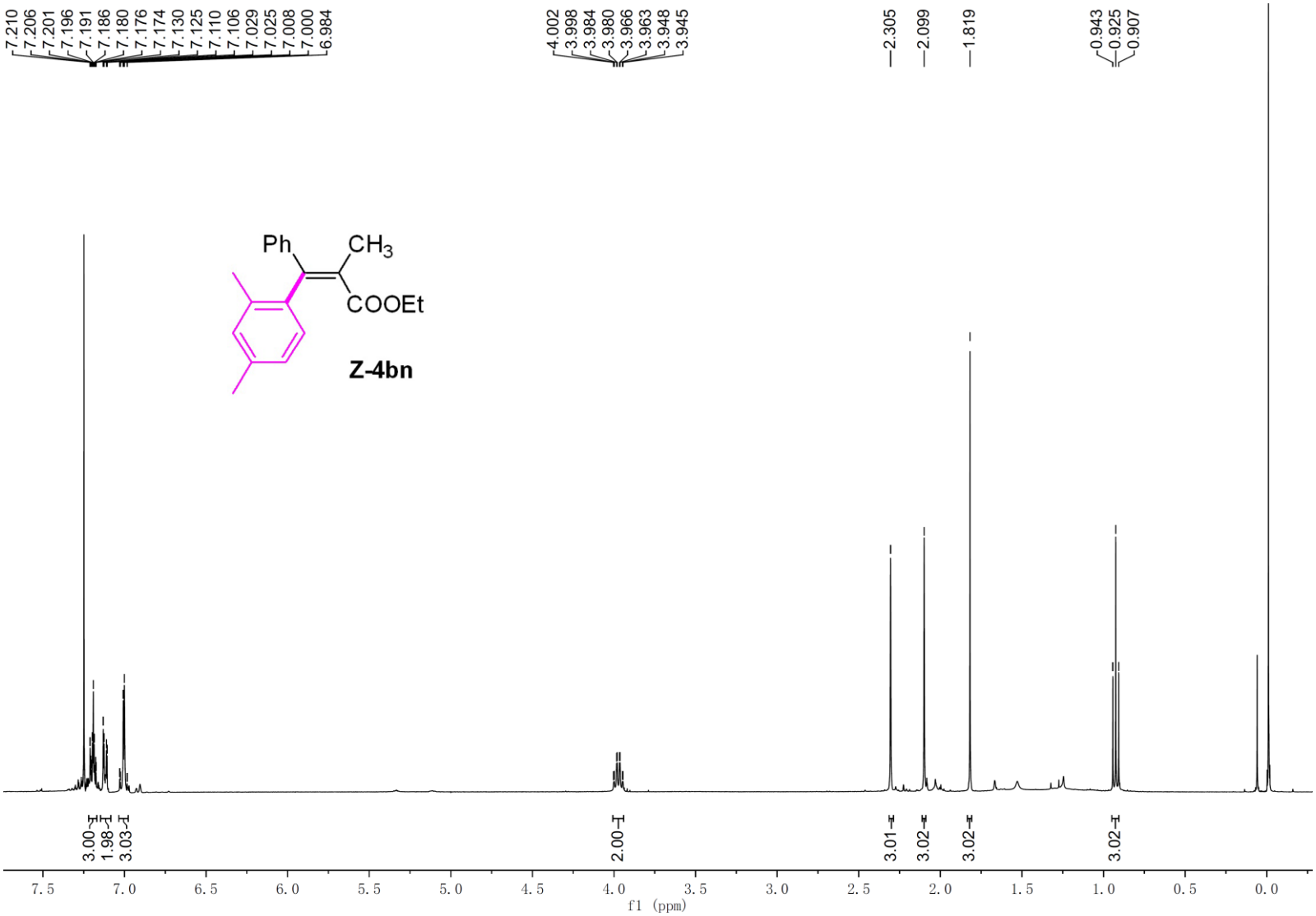
f1 (ppm)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7.110
7.106
7.029
7.025
7.008
7.000
6.984
4.002
3.998
3.984
3.980
3.966
3.963
3.948
3.945
2.305
2.099
1.819
0.943
0.925
0.907
Ph
CH3
COOEt
Z-4bn
3.00
1.98
3.03
2.00
3.01
3.02
3.02
3.02
7.5
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6.5
6.0
5.5
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2.5
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1.5
1.0
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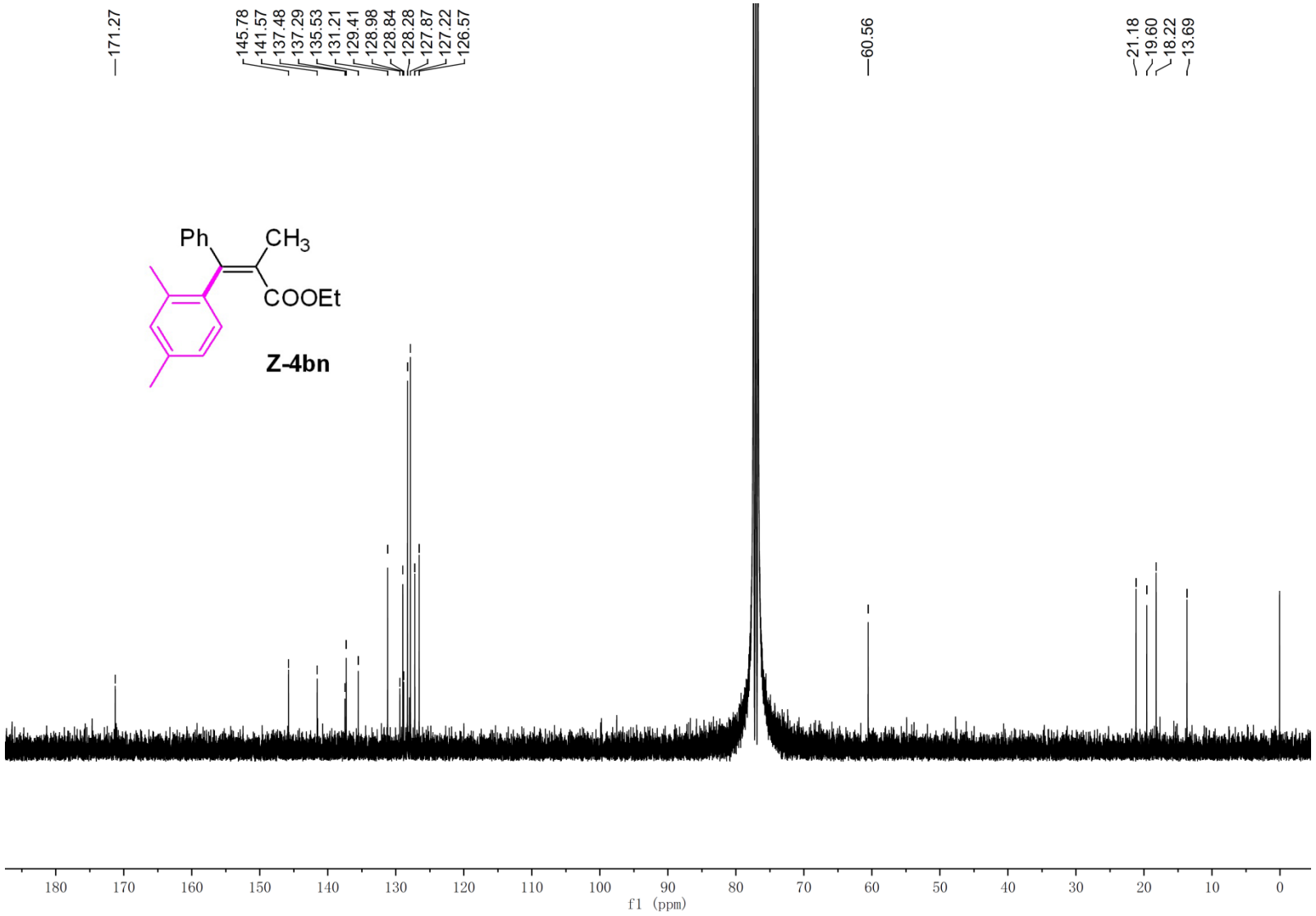
0.0
f1 (ppm)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135.53
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128.84
128.28
127.87
127.22
126.57
60.56
21.18
19.60
18.22
13.69
Ph
CH3
COOEt
Z-4bn
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
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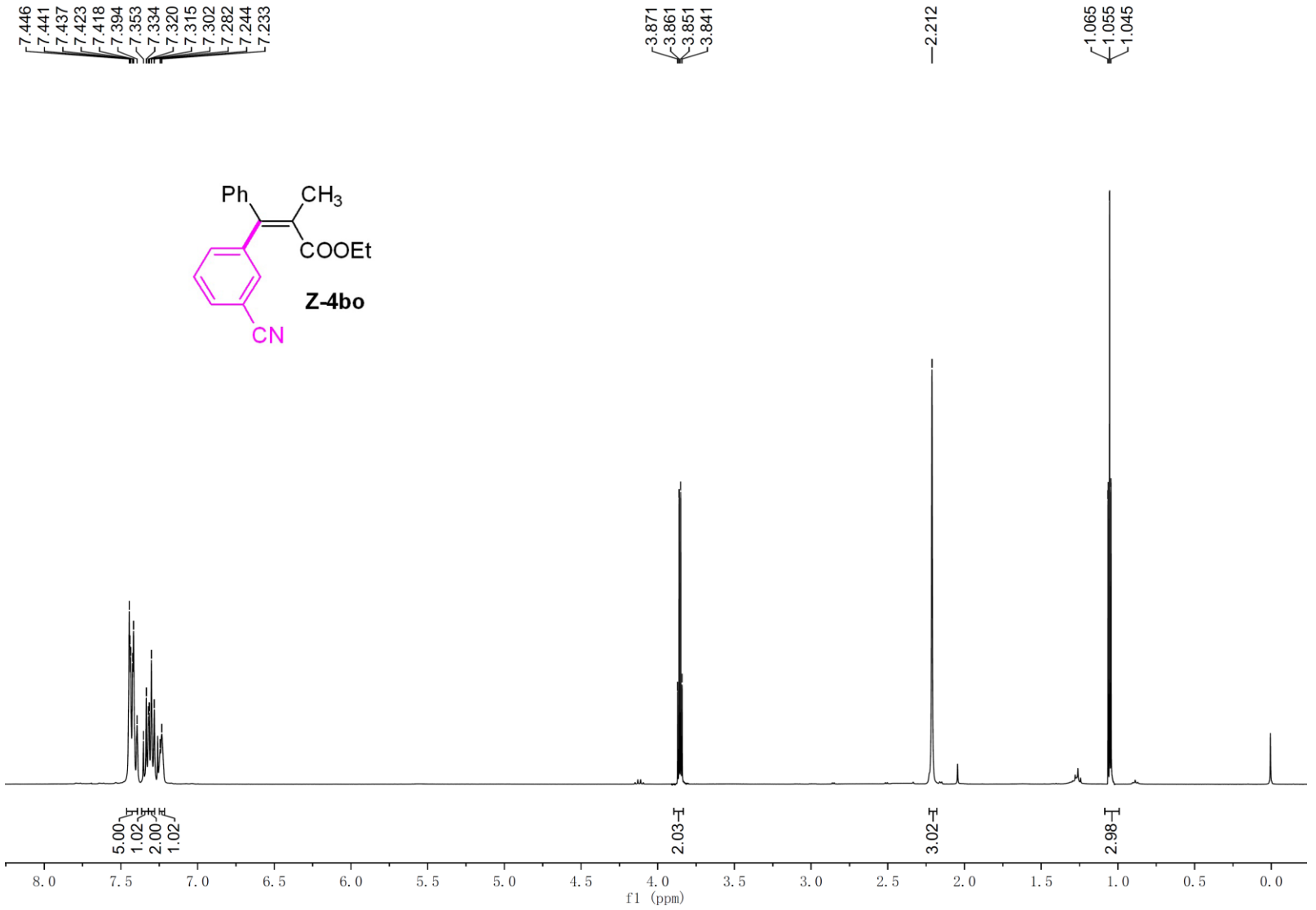
f1 (ppm)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7.394
7.353
7.334
7.320
7.315
7.302
7.282
7.244
7.233
3.871
3.861
3.851
3.841
2.212
1.065
1.055
1.045
Ph
CH3
COOEt
Z-4bo
CN
5.00
1.02
2.00
1.02
2.03
3.02
2.98
8.0
7.5
7.0
6.5
6.0
5.5
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3.0
2.5
2.0
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1.0
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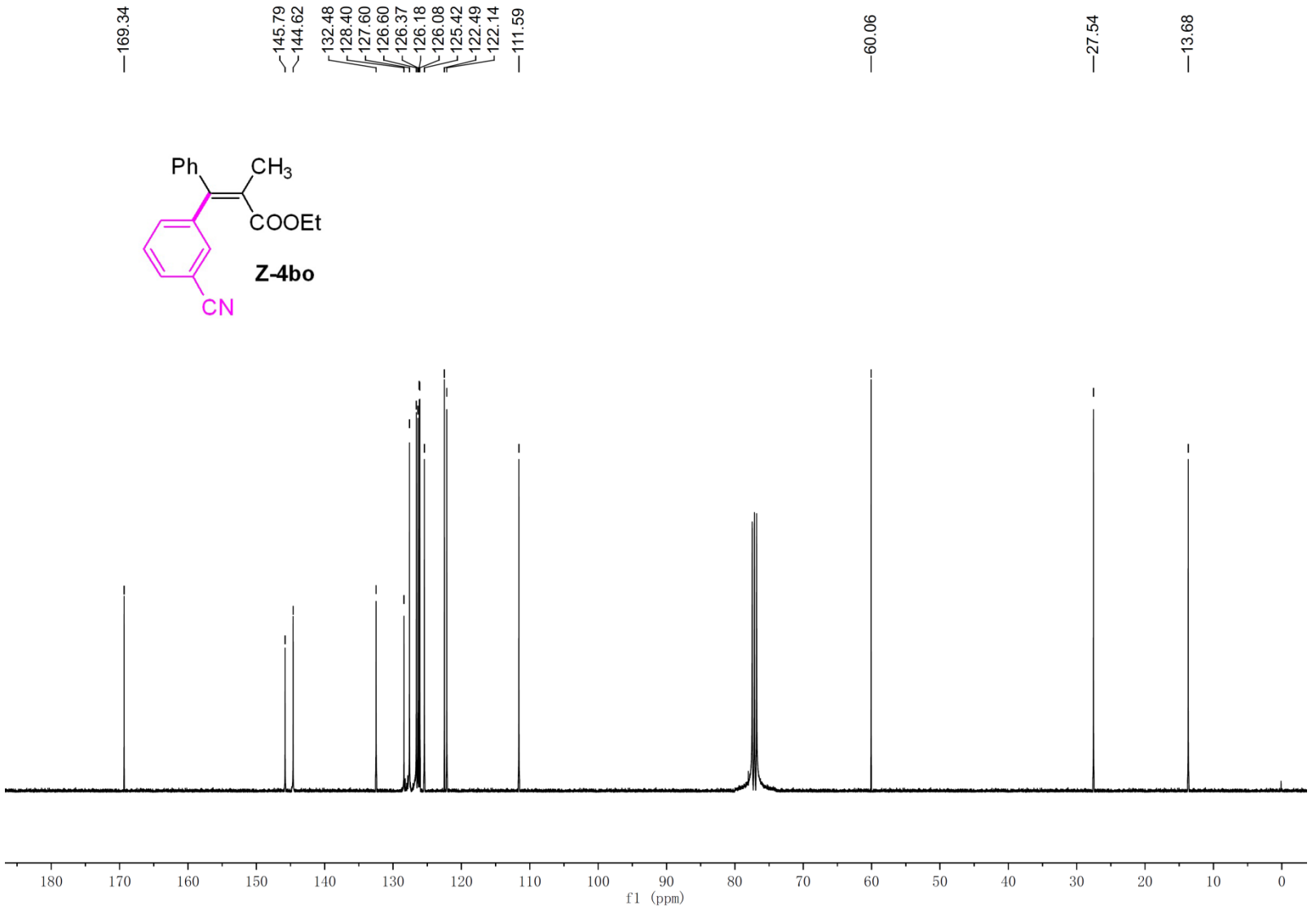
0.0
f1 (ppm)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126.37
126.18
126.08
125.42
122.49
122.14
111.59
60.06
27.54
13.68
Ph
CH3
COOEt
Z-4bo
CN
180
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150
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130
120
110
100
90
80
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50
40
30
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10
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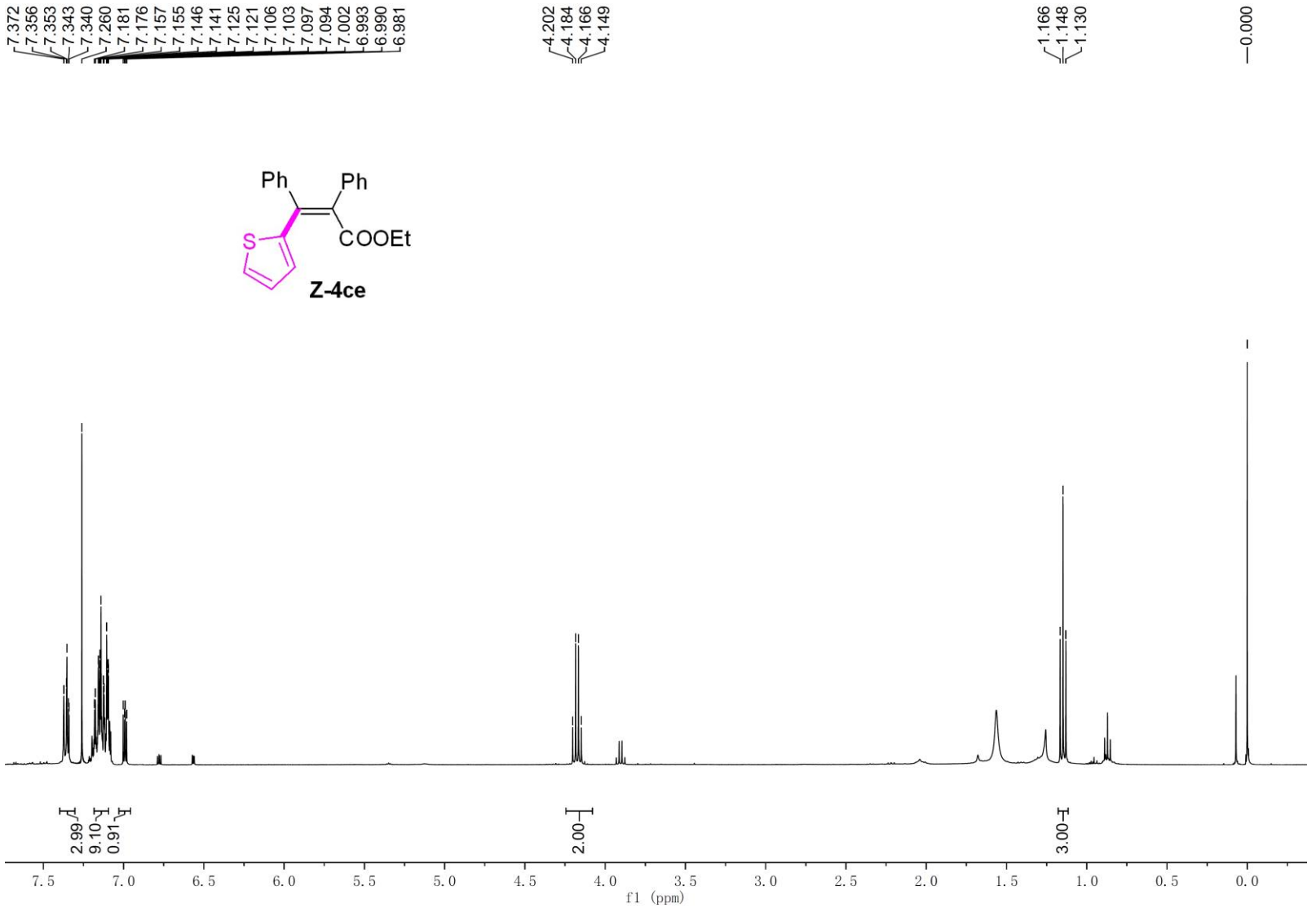
f1 (ppm)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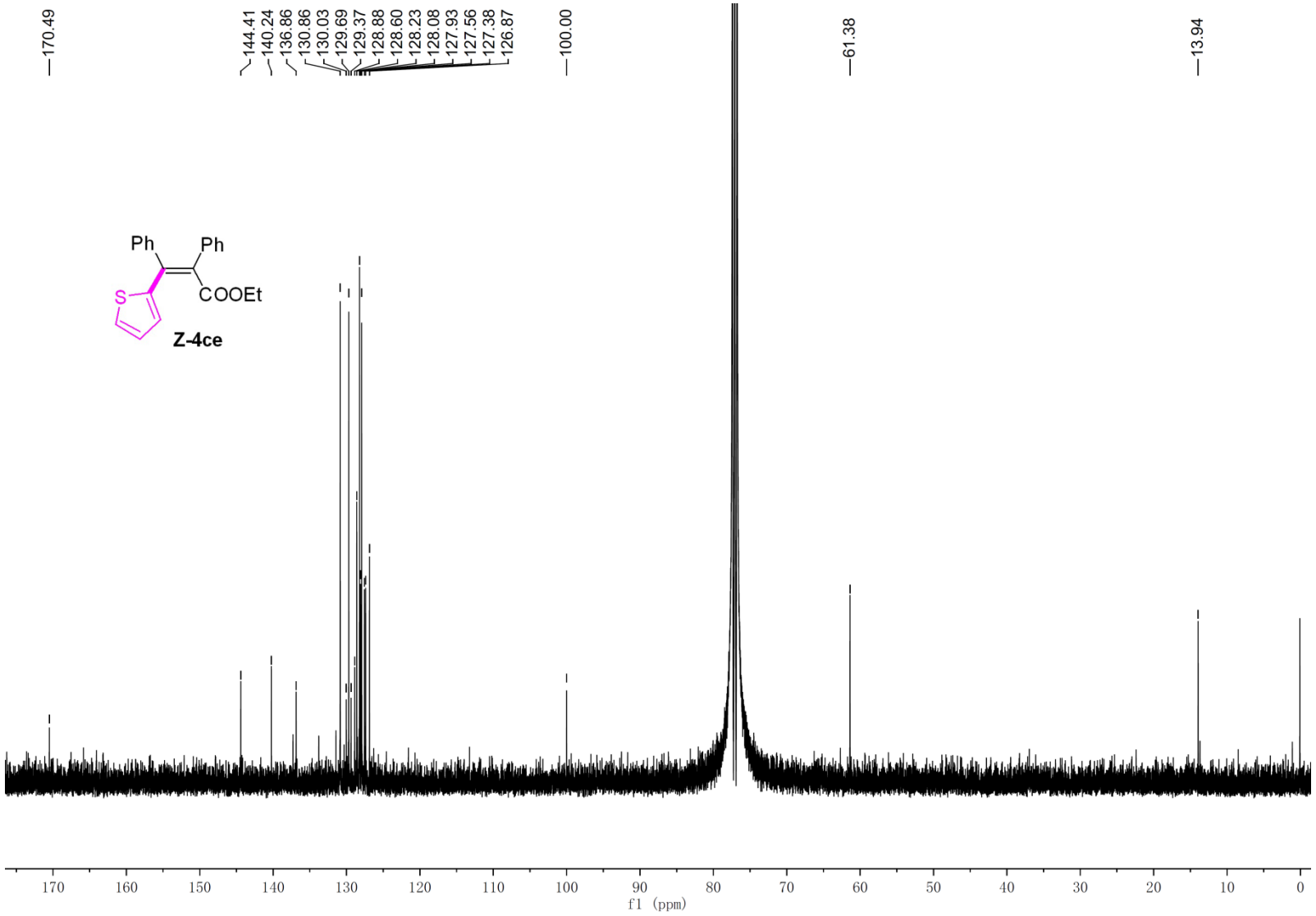
7.260
7.181
7.176
7.157
7.155
7.146
7.141
7.125
7.121
7.106
7.103
7.097
7.094
7.002
6.993
6.990
6.981
4.202
4.184
4.166
4.149
1.166
1.148
1.130
0.000
Ph
Ph
COOEt
S
Z-4ce
2.99
9.10
0.91
2.00
3.00
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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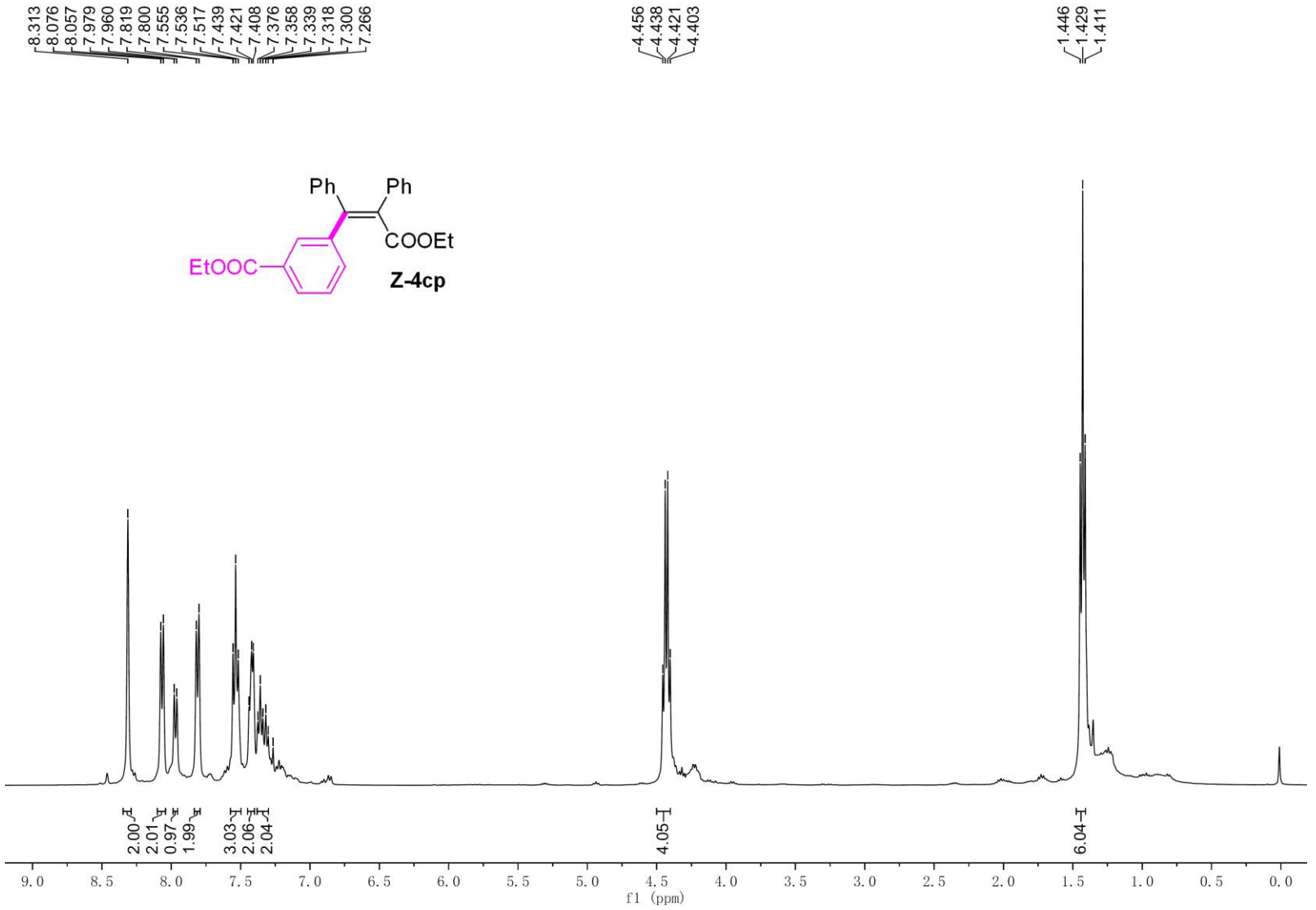
144.41
140.24
136.86
130.86
130.03
129.69
129.37
128.88
128.60
128.23
128.08
127.93
127.56
127.38
126.87
100.00
61.38
13.94
Ph
Ph
COOEt
S
Z-4ce
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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8.076
8.057
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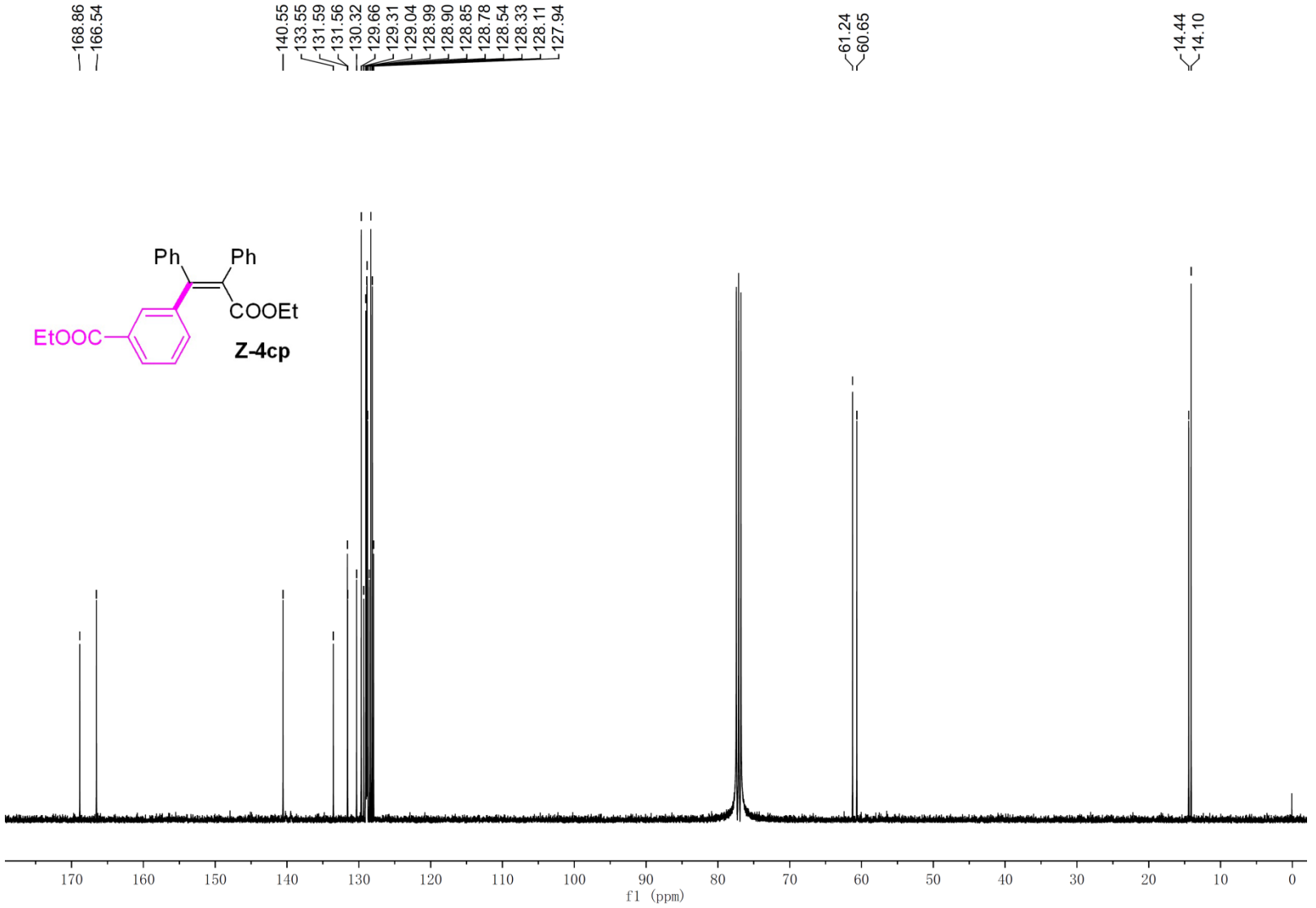
7.960
7.819
7.800
7.555
7.536
7.517
7.439
7.421
7.408
7.376
7.358
7.339
7.318
7.300
7.266
4.456
4.438
4.421
4.403
1.446
1.429
1.411
Ph
Ph
COOEt
EtOOC
Z-4cp
2.00
2.01
0.97
1.99
3.03
2.06
2.04
4.05
6.04
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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166.54
140.55
133.55
131.59
131.56
130.32
129.66
129.31
129.04
128.99
128.90
128.85
128.78
128.54
128.33
128.11
127.94
61.24
60.65
14.44
14.10
Ph
Ph
COOEt
EtOOC
Z-4cp
170
160
150
140
130
120
110
100
90
80
70
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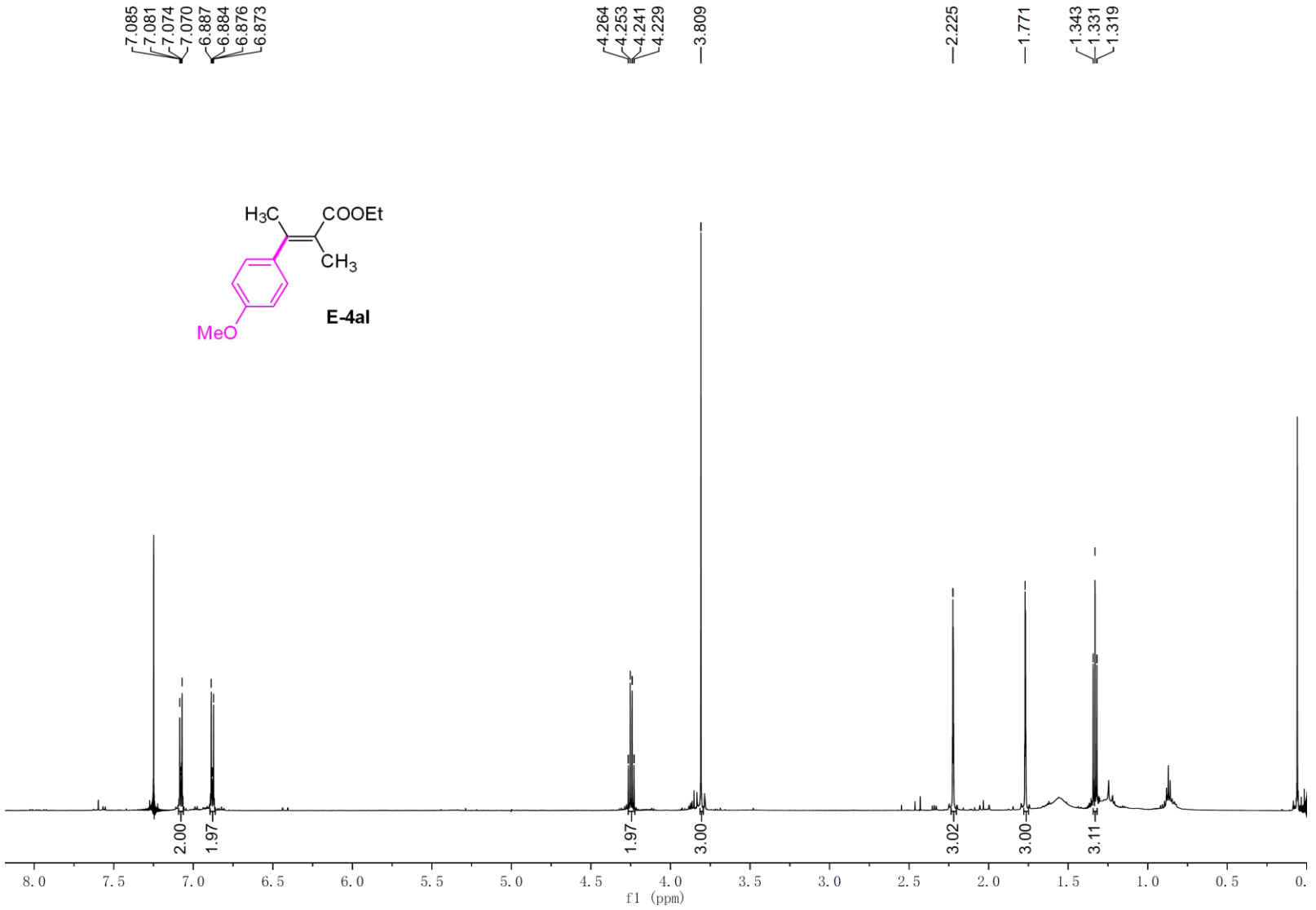
50
40
30
20
10
0
f1 (ppm)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6.884
6.876
6.873
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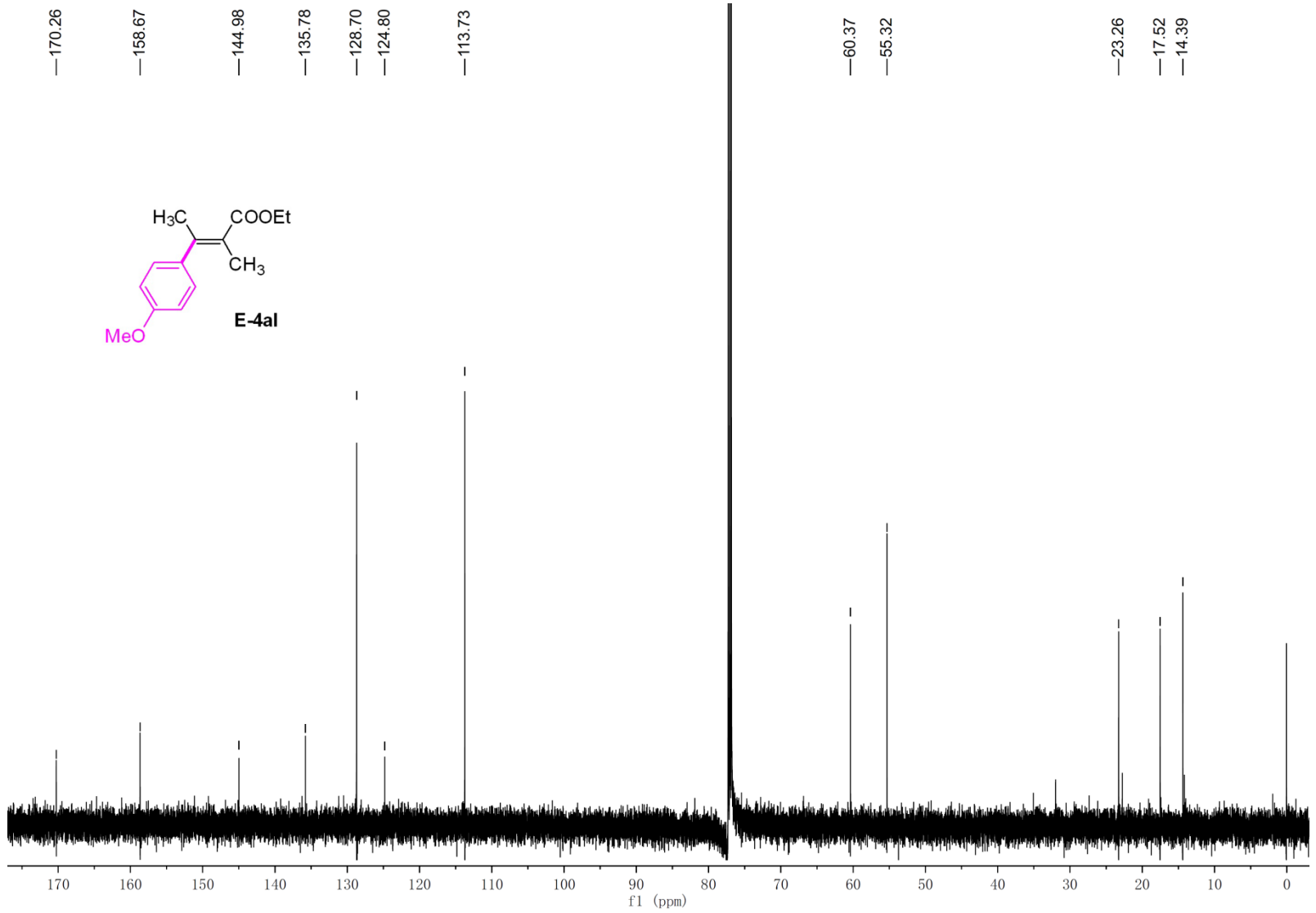
4.253
4.241
4.229
3.809
2.225
1.771
1.343
1.331
1.319
H3C
COOEt
CH3
MeO
E-4al
2.00
1.97
1.97
3.00
3.02
3.00
3.11
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.
f1 (ppm)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158.67
144.98
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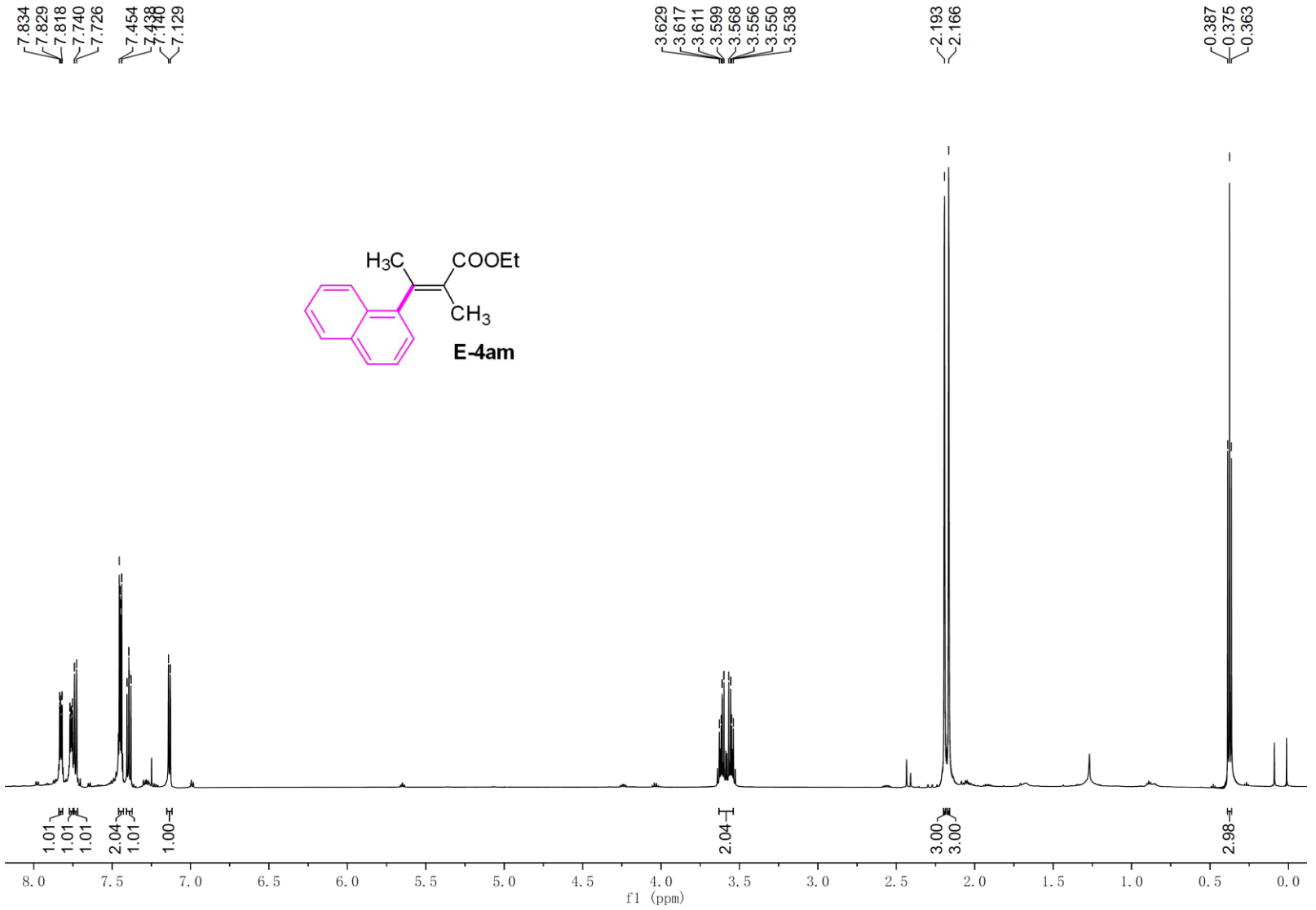
128.70
124.80
113.73
60.37
55.32
23.26
17.52
14.39
H3C
COOEt
CH3
MeO
E-4al
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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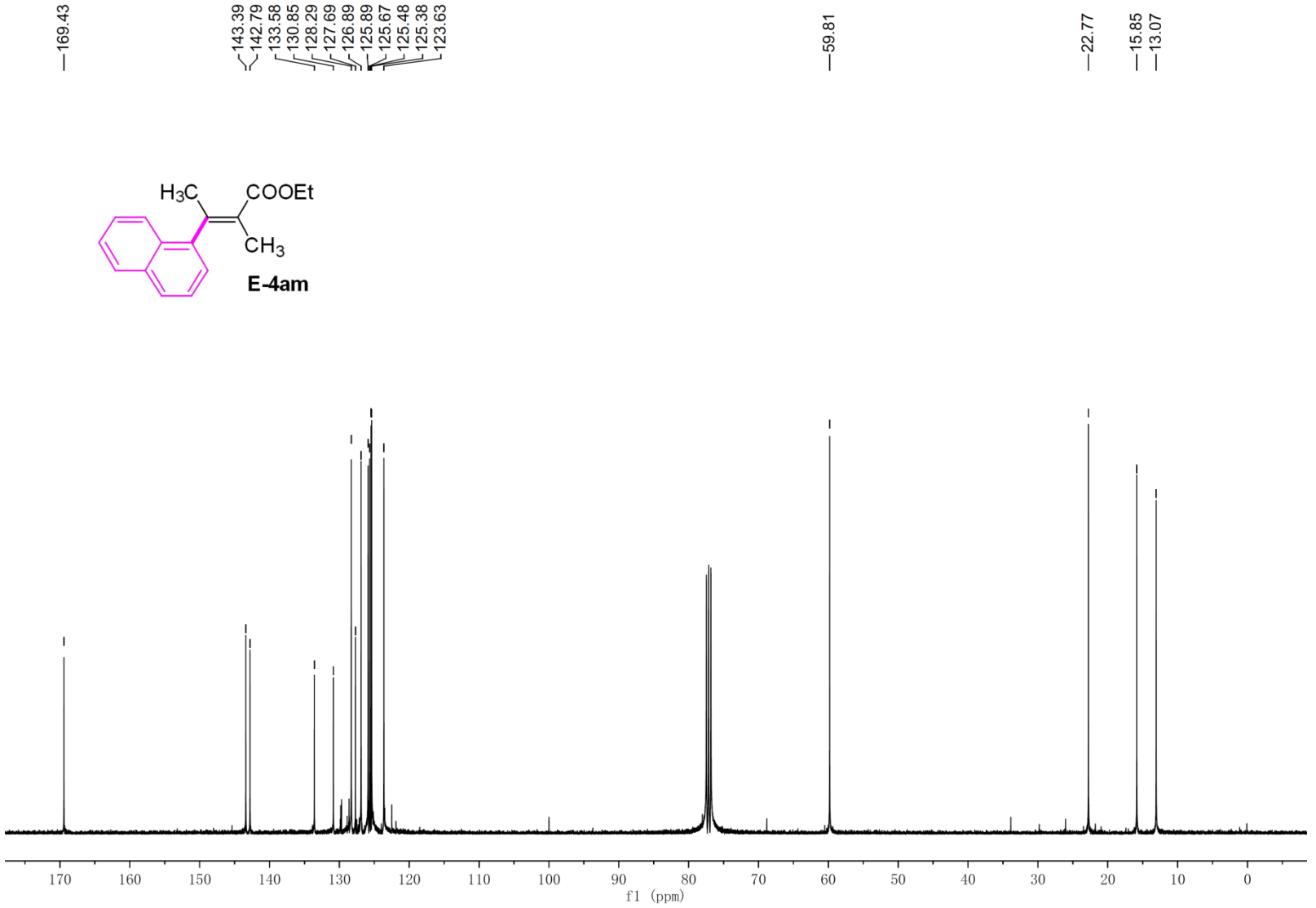
7.438
7.129
3.629
3.617
3.611
3.599
3.568
3.556
3.550
3.538
2.193
2.166
0.387
0.375
0.363
H3C
COOEt
CH3
E-4am
1.01
1.01
1.01
2.04
1.01
1.00
2.04
3.00
3.00
2.98
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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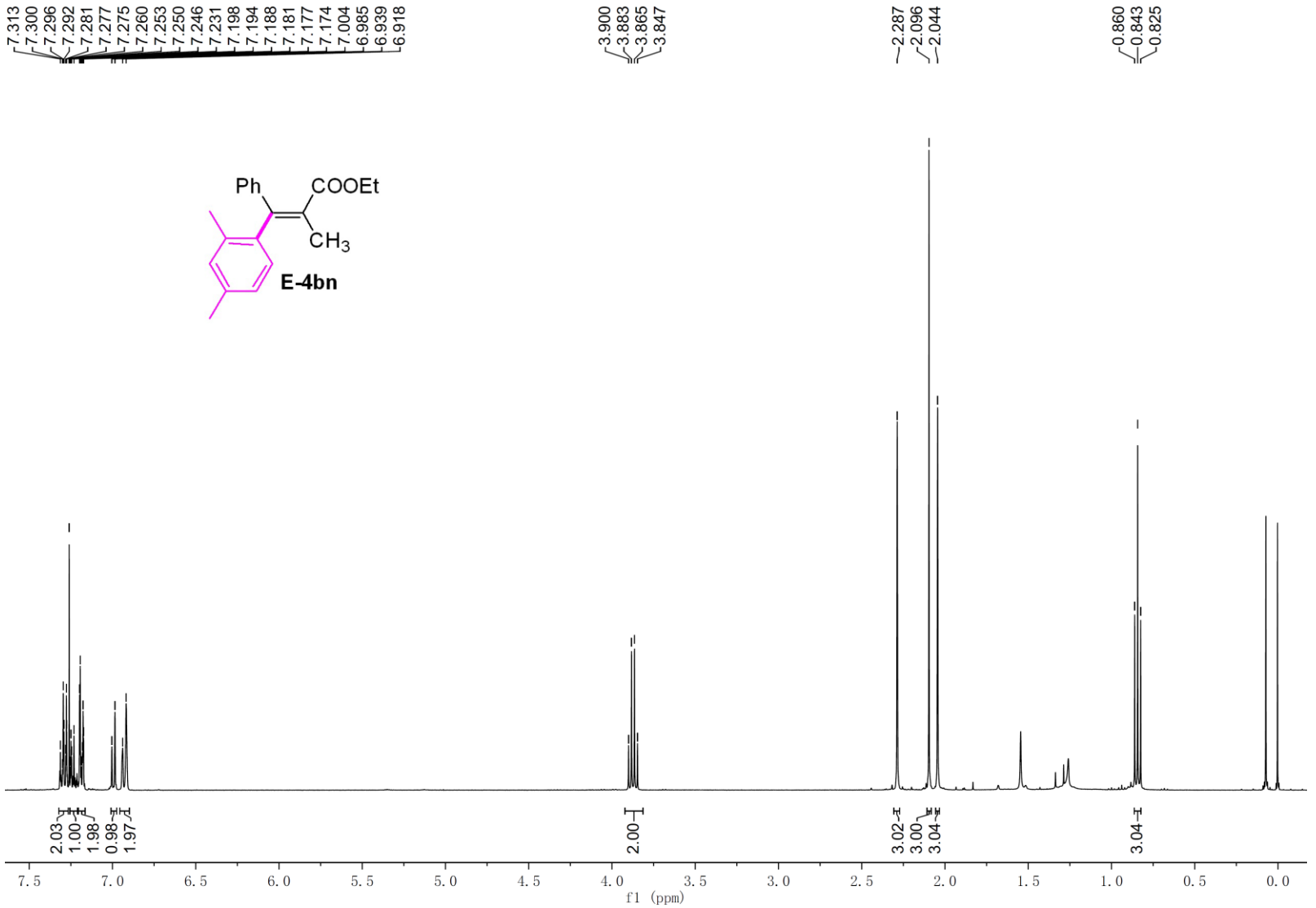
128.29
127.69
126.89
125.89
125.67
125.48
125.38
123.63
59.81
22.77
15.85
13.07
H3C
COOEt
CH3
E-4am
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.253
7.250
7.246
7.231
7.198
7.194
7.188
7.181
7.177
7.174
7.004
6.985
6.939
6.918
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3.883
3.865
3.847
2.287
2.096
2.044
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0.843
0.825
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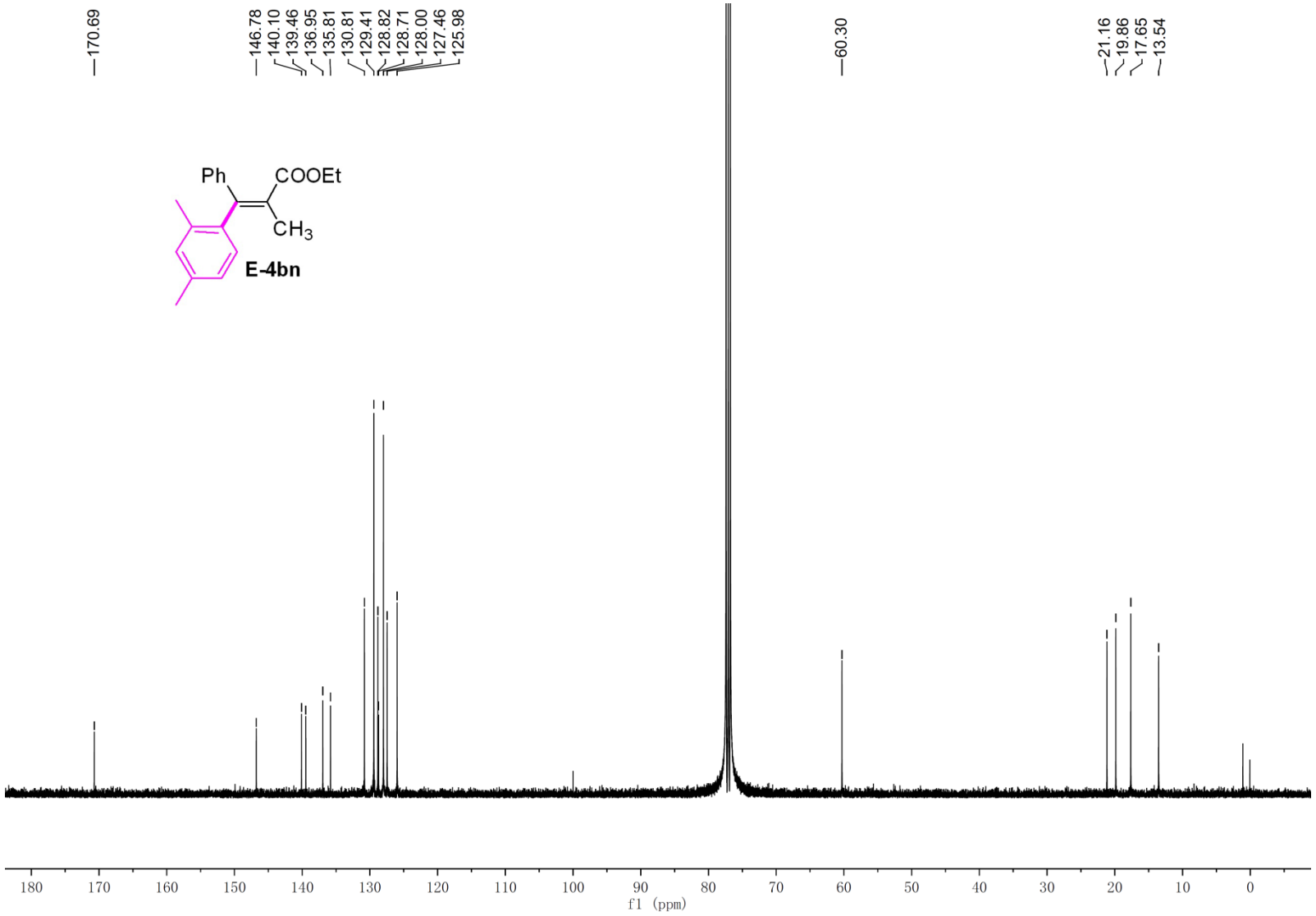
CH3
E-4bn
2.03
1.00
1.98
0.98
1.97
2.00
3.02
3.00
3.04
3.04
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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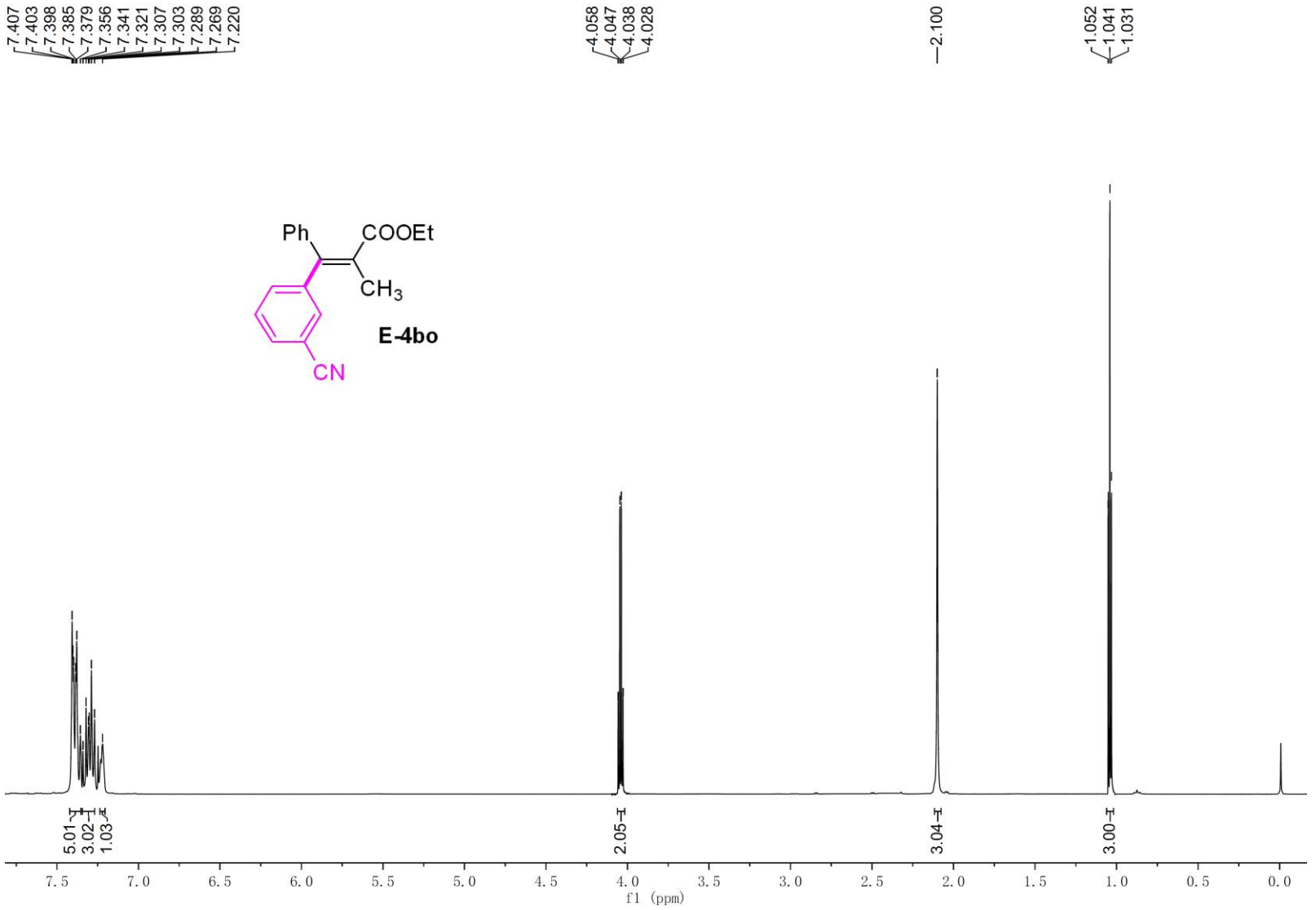
129.41
128.82
128.71
128.00
127.46
125.98
60.30
21.16
19.86
17.65
13.54
Ph
COOEt
CH3
E-4bn
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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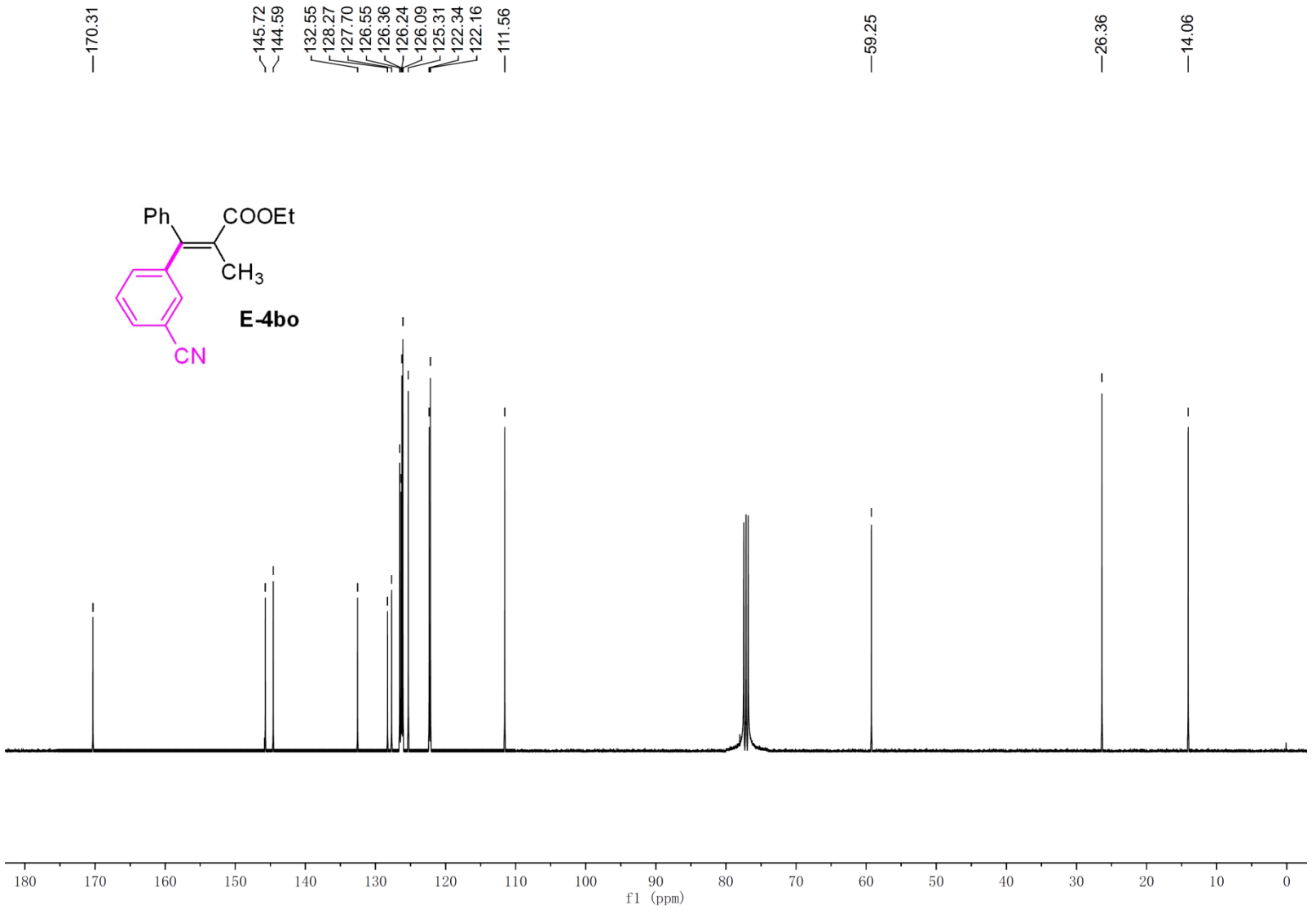
7.269
7.220
4.058
4.047
4.038
4.028
2.100
1.052
1.041
1.031
Ph
COOEt
CH3
E-4bo
CN
5.01
3.02
1.03
2.05
3.04
3.00
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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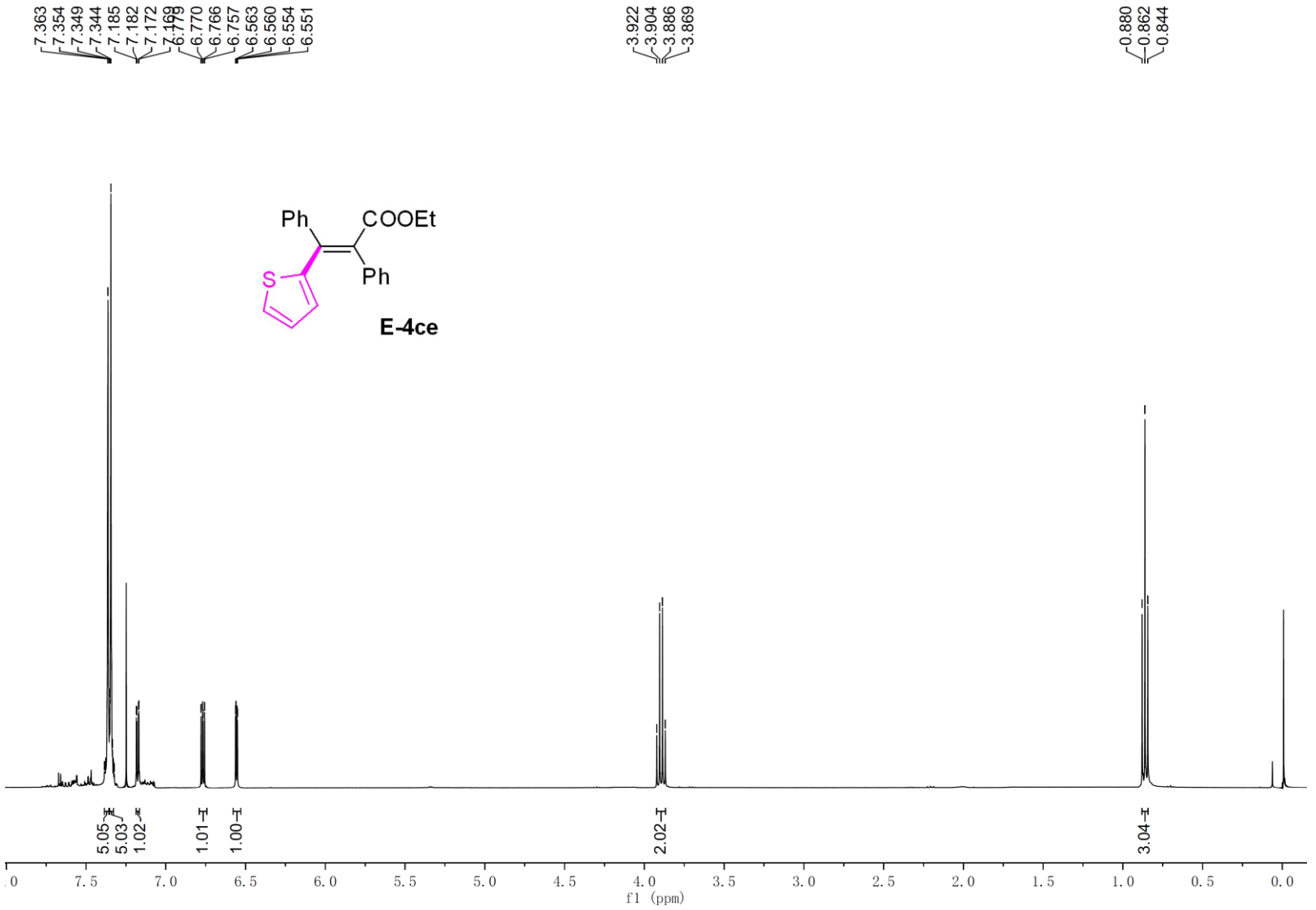
126.55
126.36
126.24
126.09
125.31
122.34
122.16
111.56
59.25
26.36
14.06
Ph
COOEt
CH3
CN
E-4bo
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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6.757
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6.560
6.554
6.551
3.922
3.904
3.886
3.869
0.880
0.862
0.844
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S
Ph
E-4ce
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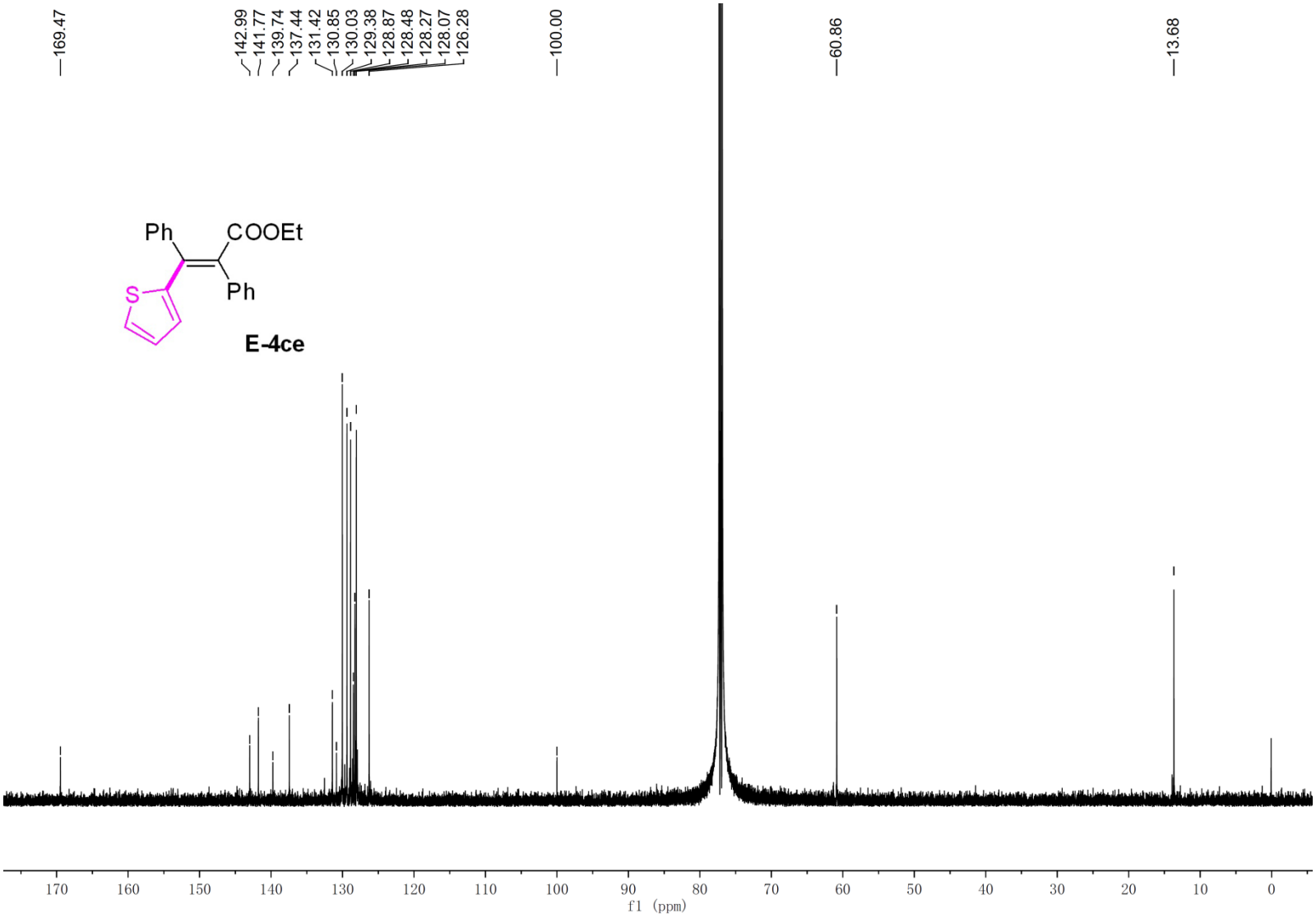
1.00
2.02
3.04
f1 (ppm)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130.03
129.38
128.87
128.48
128.27
128.07
126.28
100.00
60.86
13.68
Ph
COOEt
Ph
S
E-4ce
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130
120
110
100
90
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70
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50
40
30
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10
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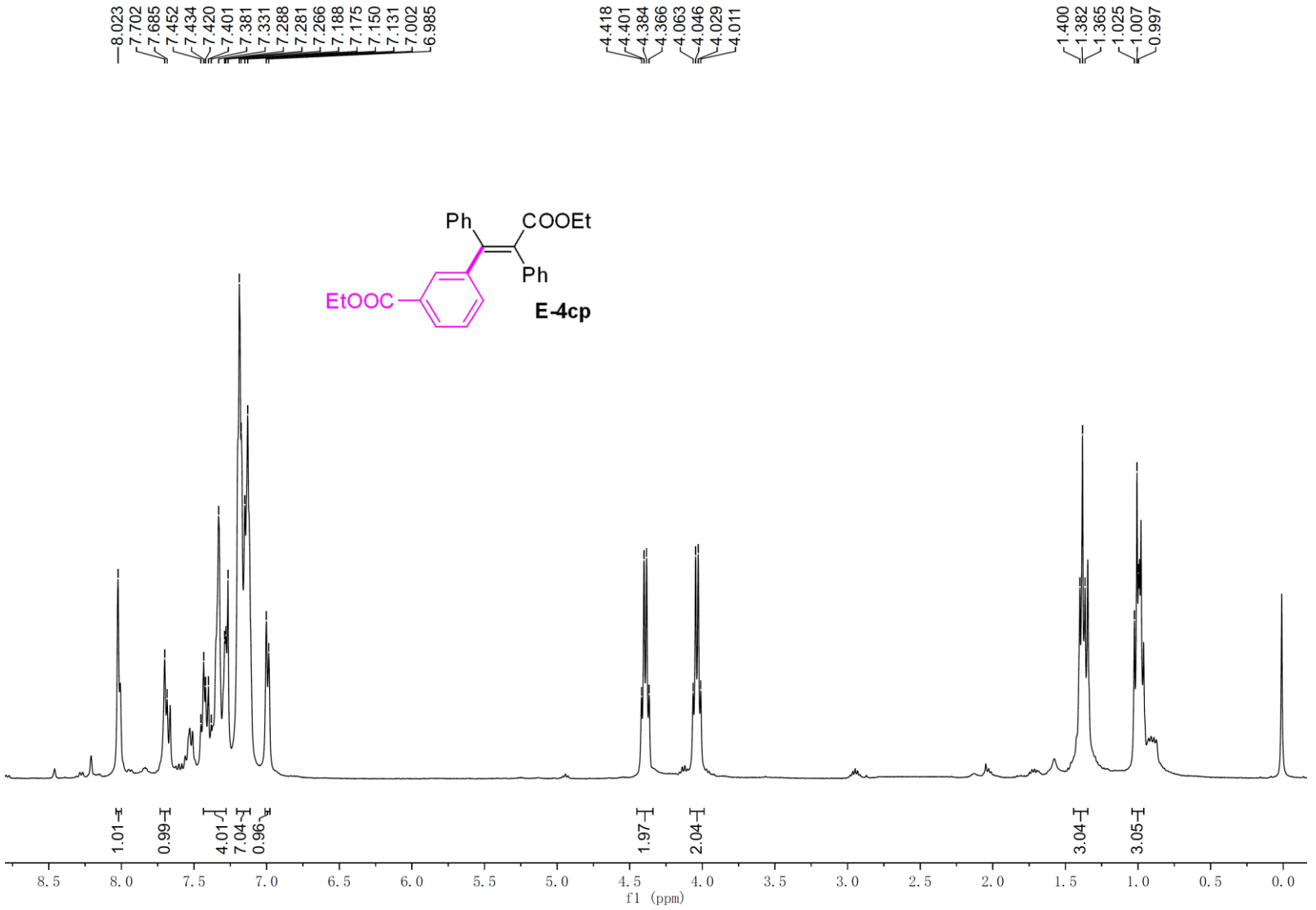
f1 (ppm)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7.266
7.188
7.175
7.150
7.131
7.002
6.985
4.418
4.401
4.384
4.366
4.063
4.046
4.029
4.011
1.400
1.382
1.365
1.025
1.007
0.997
Ph
COOEt
Ph
EtOOC
E-4cp
1.01
0.99
4.01
7.04
0.96
1.97
2.04
3.04
3.05
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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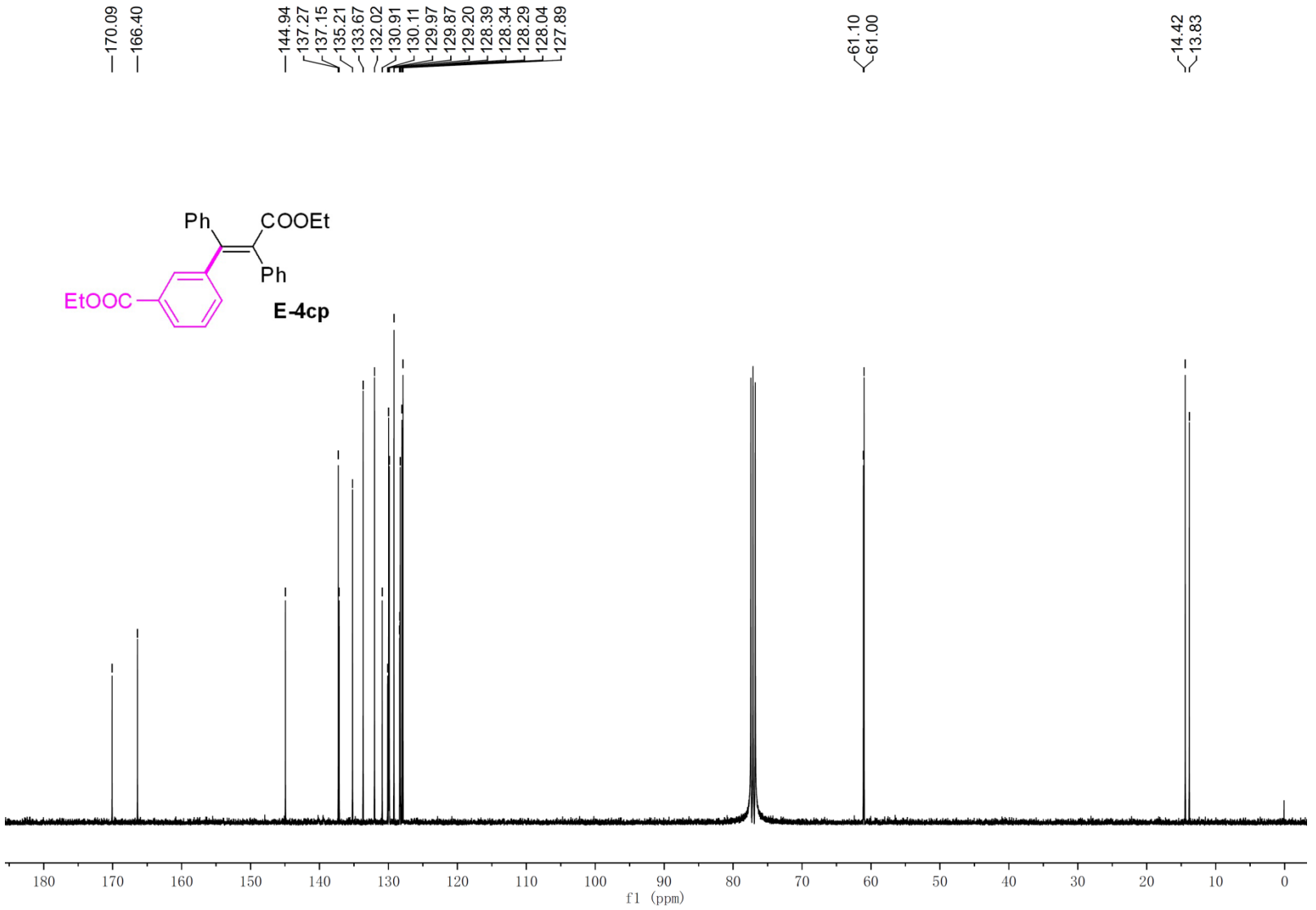
f1 (ppm)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133.67
132.02
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129.97
129.87
129.20
128.39
128.34
128.29
128.04
127.89
61.10
61.00
14.42
13.83
Ph
COOEt
Ph
EtOOC
E-4cp
180
170
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120
110
100
90
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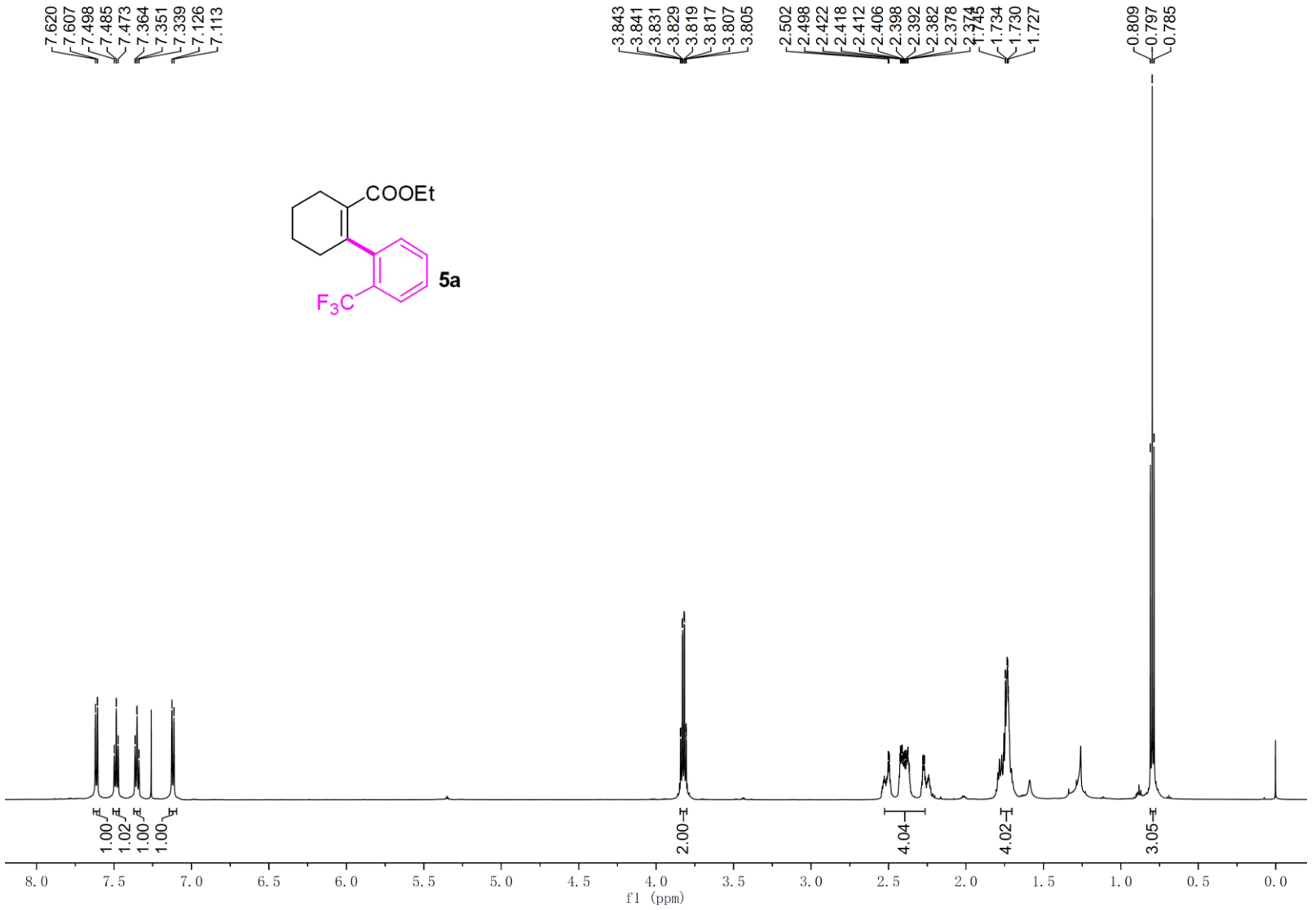
50
40
30
20
10
0
f1 (ppm)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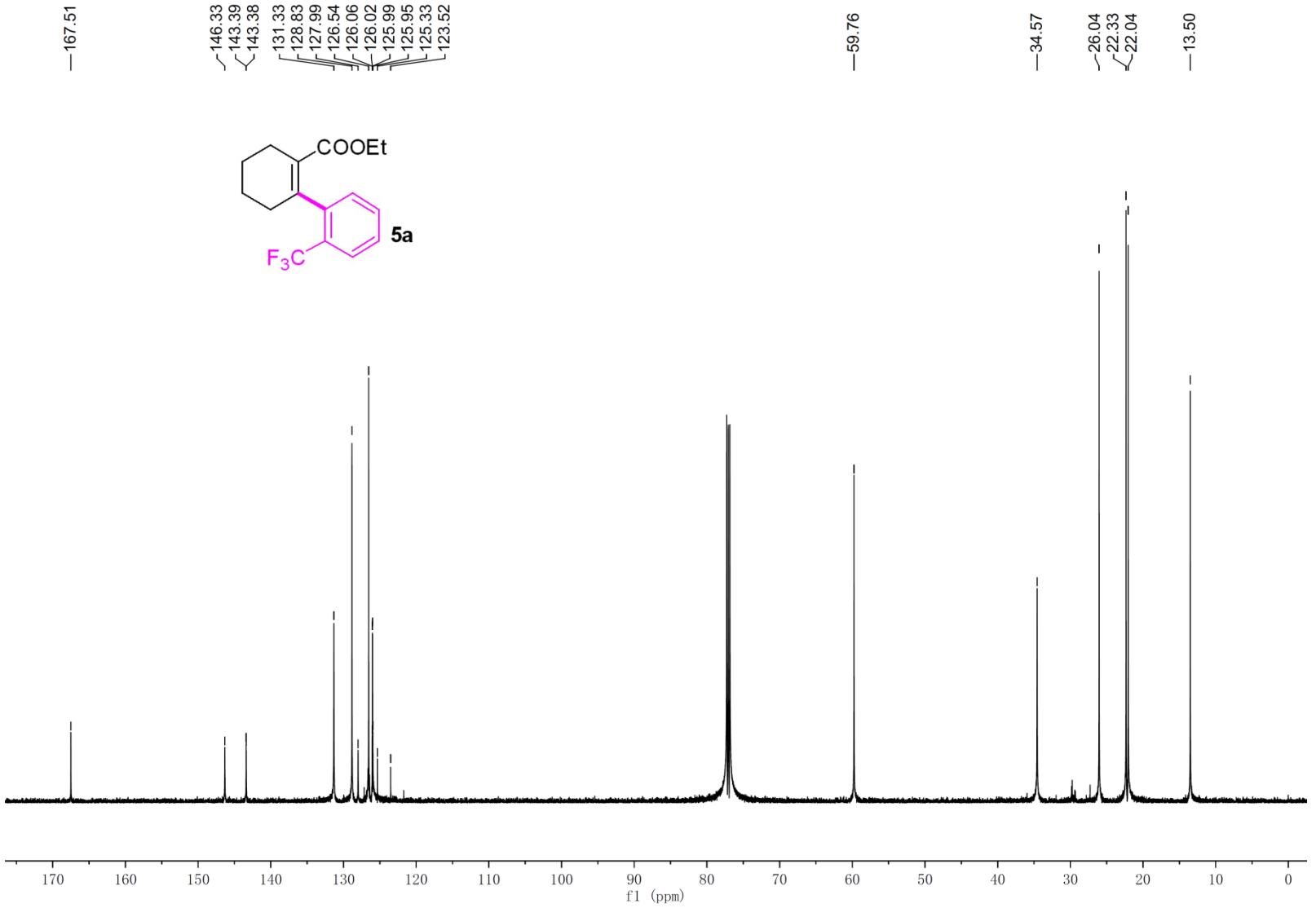
3.831
3.829
3.819
3.817
3.807
3.805
2.502
2.498
2.422
2.418
2.412
2.406
2.398
2.392
2.382
2.378
1.734
1.730
1.727
0.809
0.797
0.785
COOEt
F3C
5a
1.00
1.02
1.00
1.00
2.00
4.04
4.02
3.05
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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143.39
143.38
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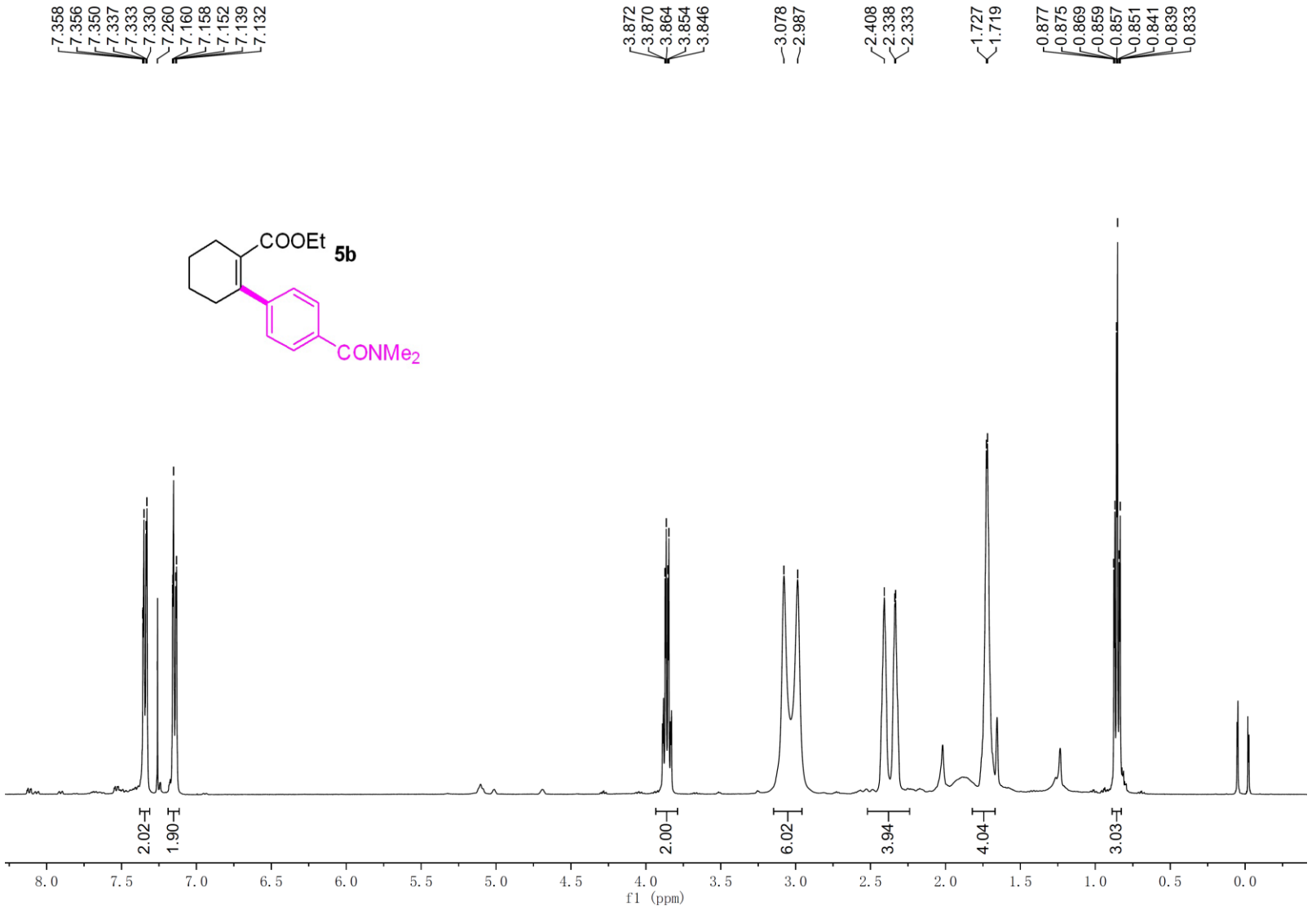
128.83
127.99
126.54
126.06
126.02
125.99
125.95
125.33
123.52
59.76
34.57
26.04
22.33
22.04
13.50
COOEt
F3C
5a
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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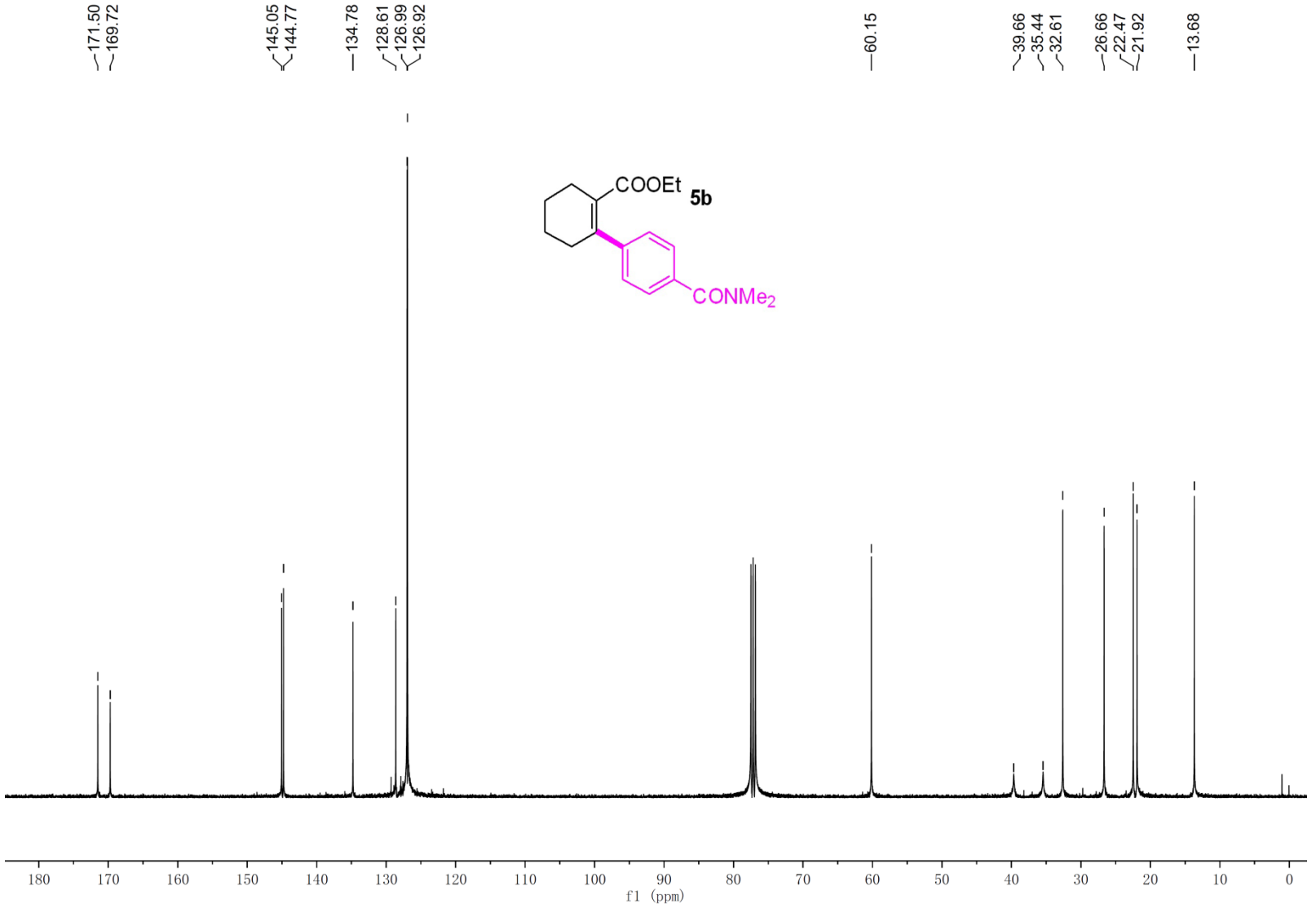
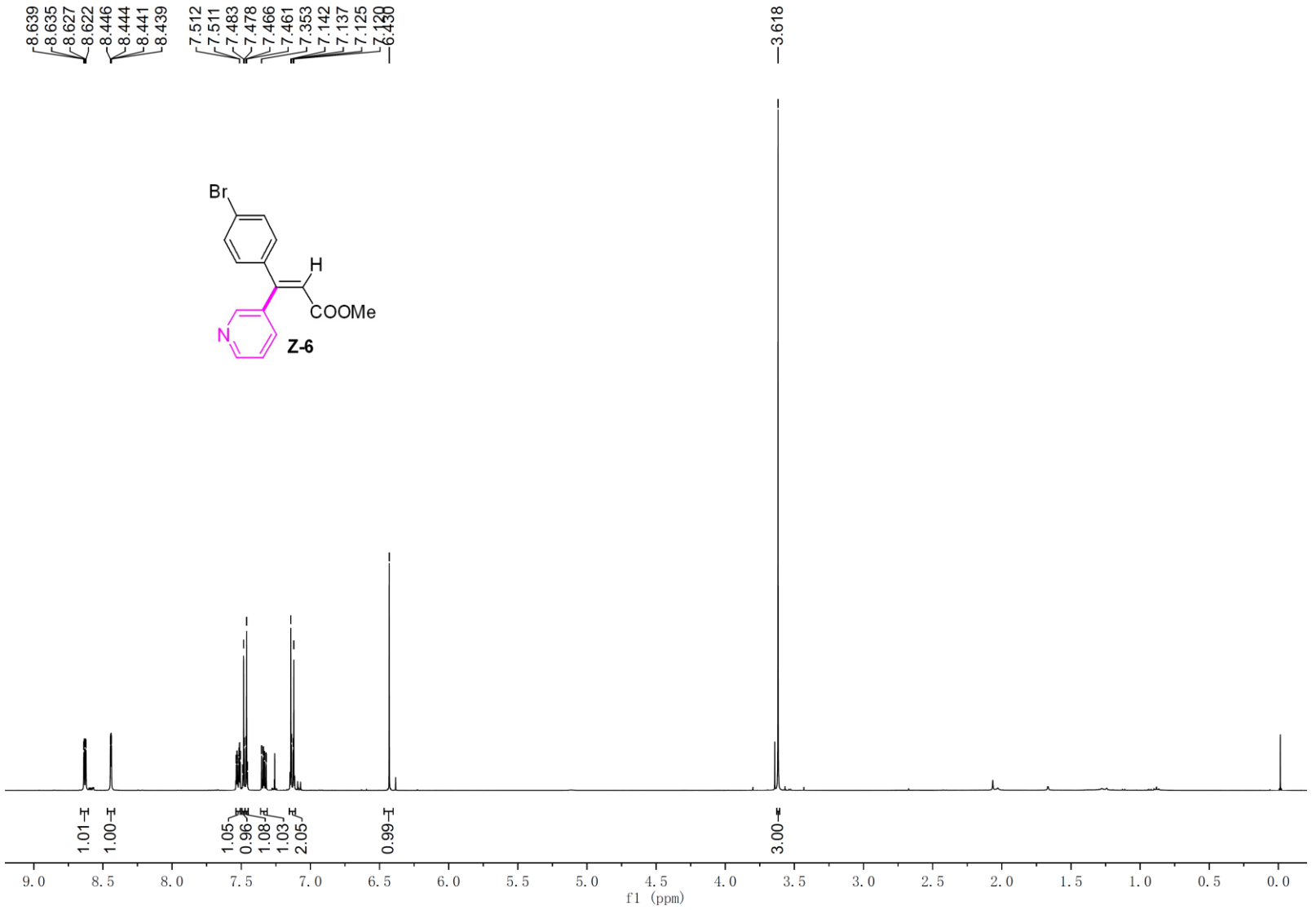
7.356
7.350
7.337
7.333
7.330
7.260
7.160
7.158
7.152
7.139
7.132
3.872
3.870
3.864
3.854
3.846
3.078
2.987
2.408
2.338
2.333
1.727
1.719
0.877
0.875
0.869
0.859
0.857
0.851
0.841
0.839
0.833
COOEt
5b
CONMe2
2.02
1.90
2.00
6.02
3.94
4.04
3.03
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


171.50
169.72
145.05
144.77
134.78
128.61
126.99
126.92
60.15
39.66
35.44
32.61
26.66
22.47
21.92
13.68
COOEt
5b
CONMe2
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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8.635
8.627
8.622
8.446
8.444
8.441
8.439
7.512
7.511
7.483
7.478
7.466
7.461
7.353
7.142
7.137
7.125
7.120
6.430
3.618
Br
H
COOMe
N
Z-6
1.01
1.00
1.05
0.96
1.08
1.03
2.05
0.99
3.00
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
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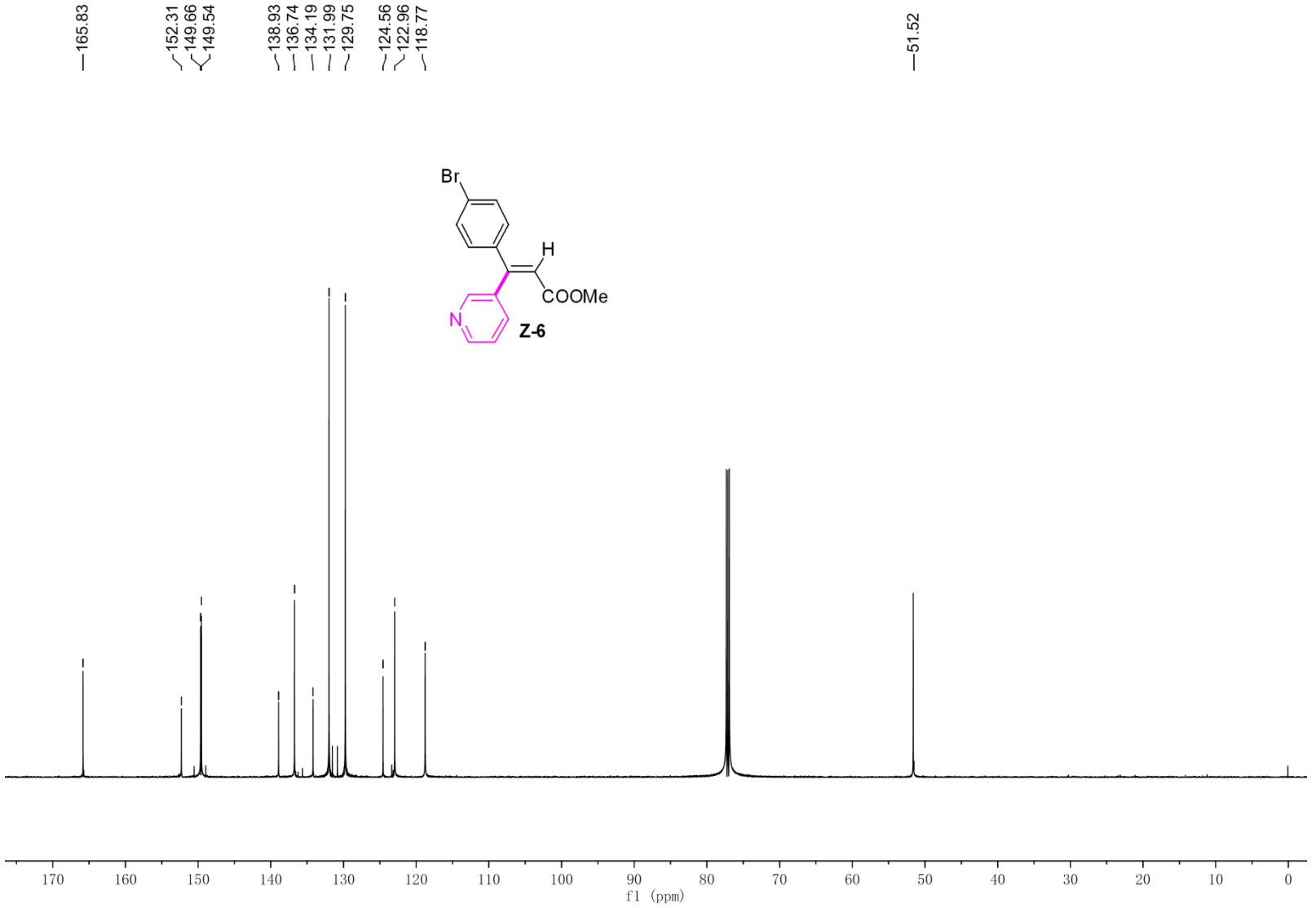
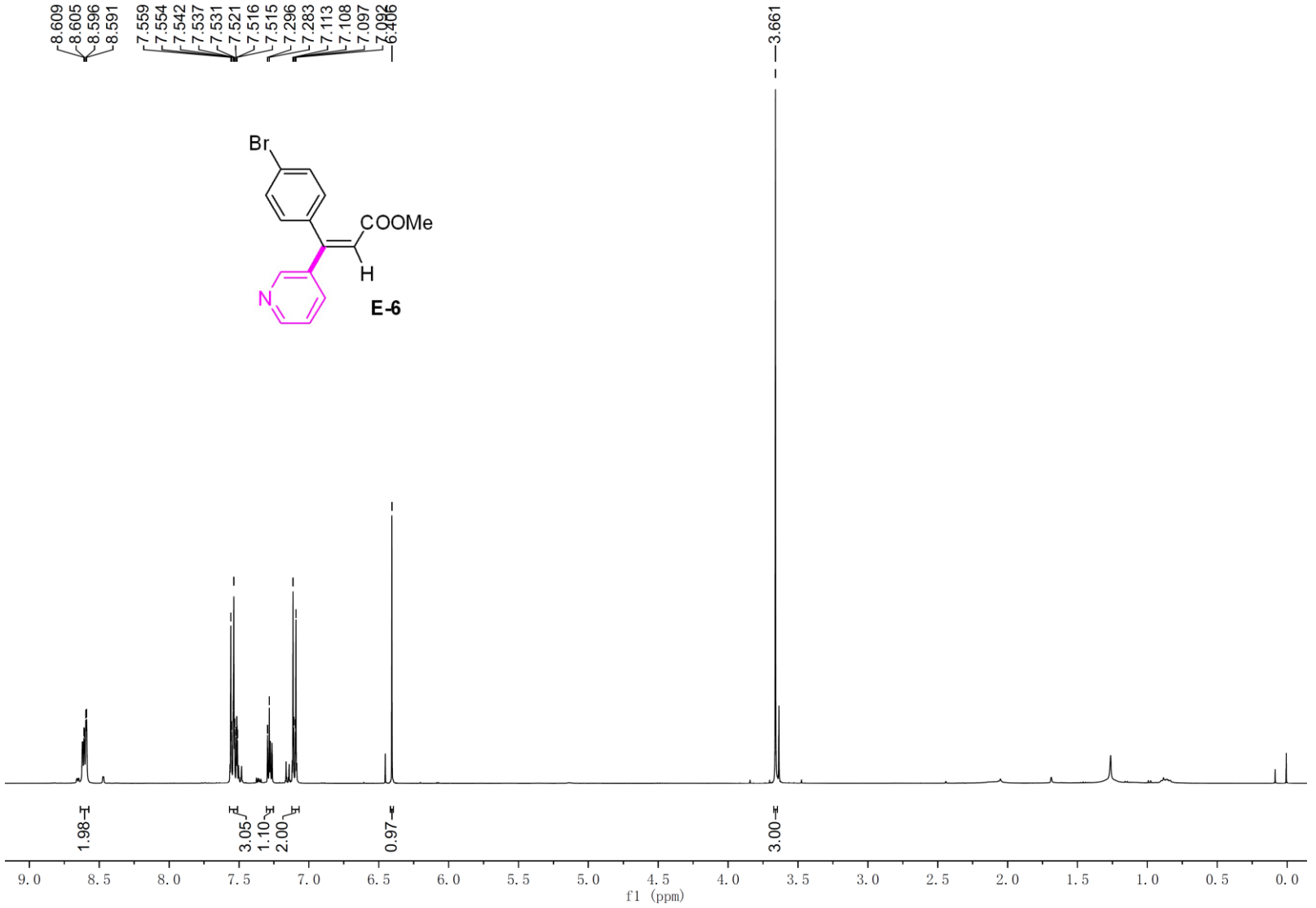
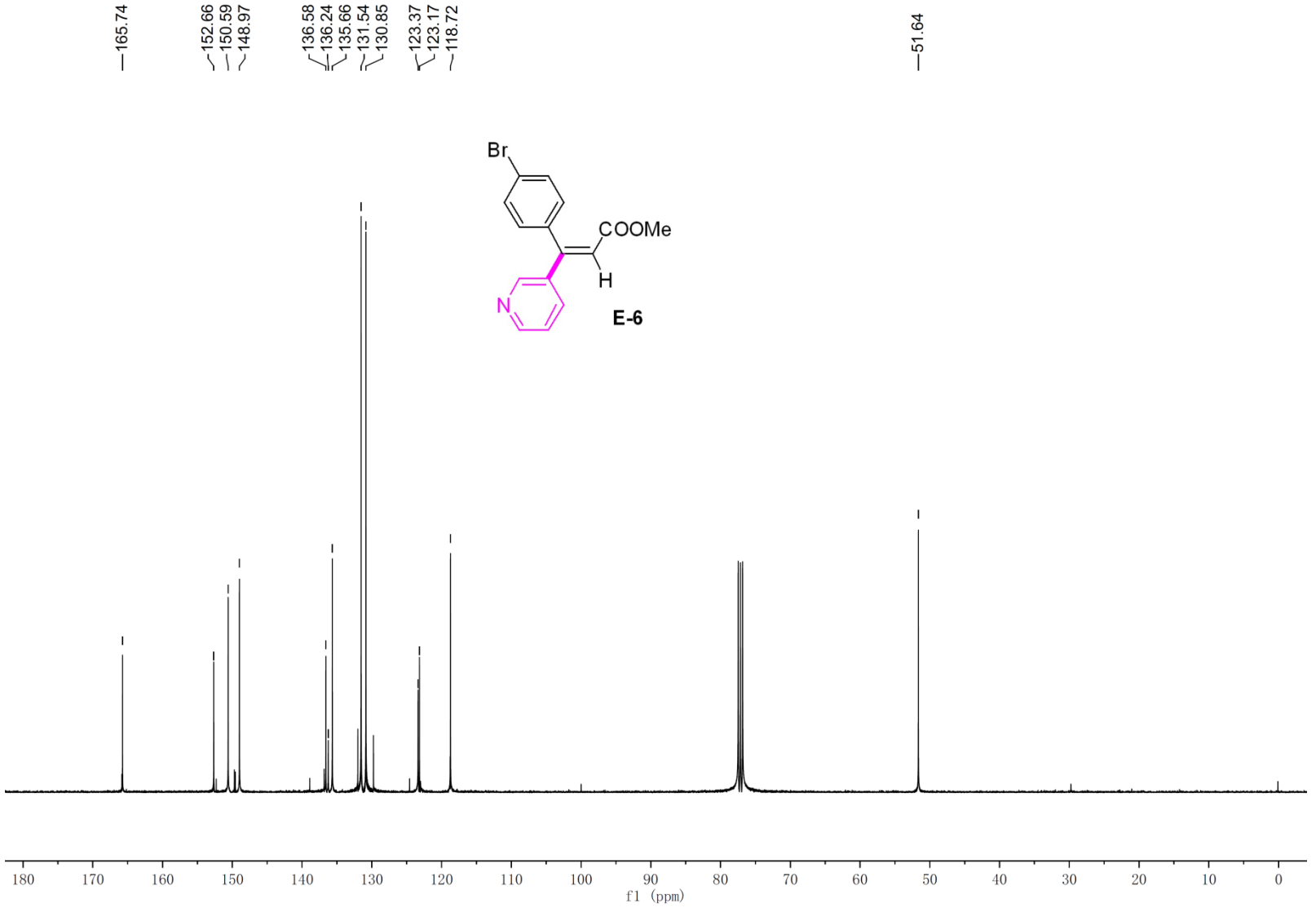
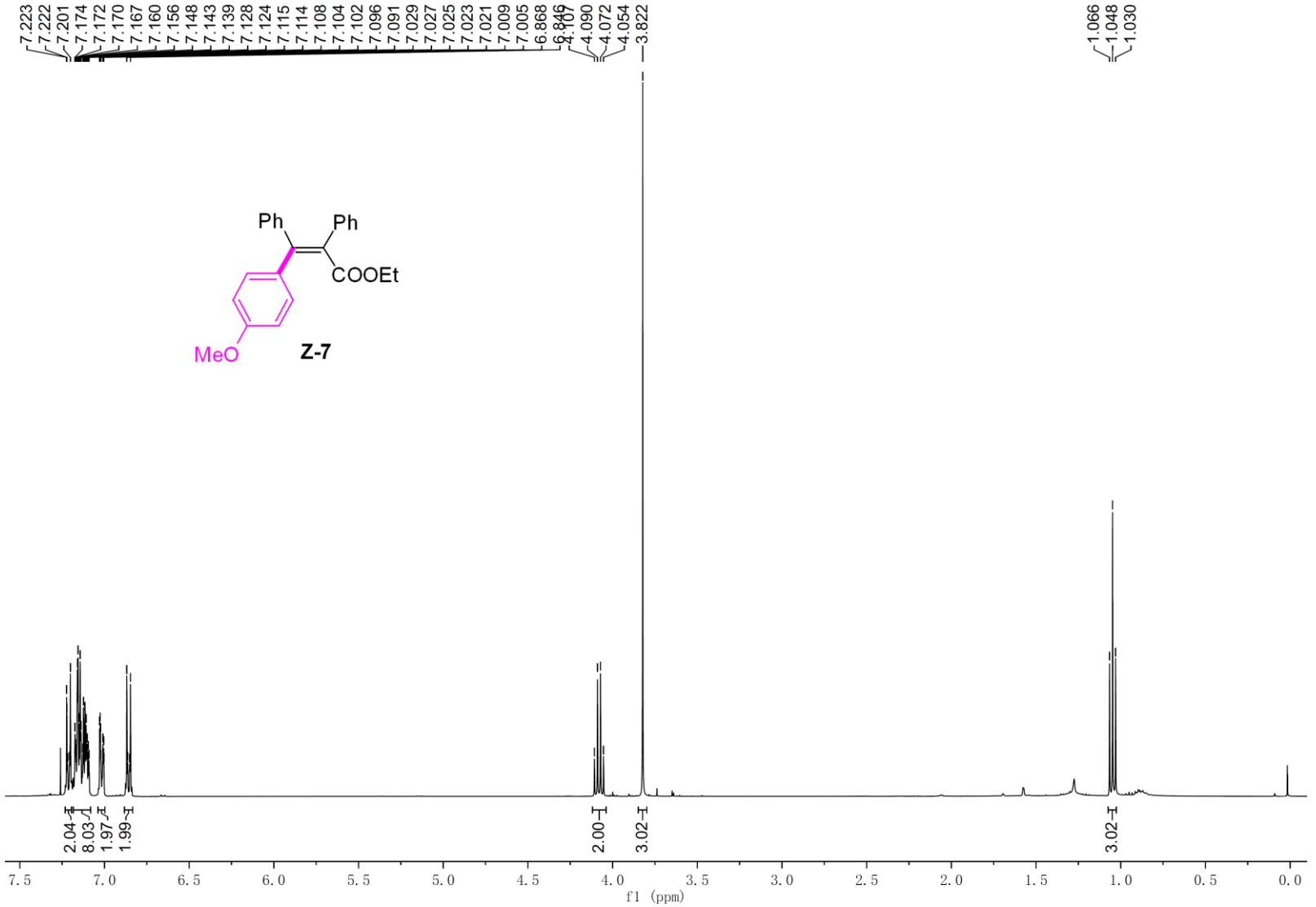
0.0
f1 (ppm)


165.83
152.31
149.66
149.54
138.93
136.74
134.19
131.99
129.75
124.56
122.96
118.77
51.52
Br
H
COOMe
N
Z-6
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.609
8.605
8.596
8.591
7.559
7.554
7.542
7.537
7.531
7.521
7.516
7.515
7.296
7.283
7.113
7.108
7.097
7.092
7.086
6.406
3.661
Br
COOMe
H
N
E-6
1.98
3.05
1.10
2.00
0.97
3.00
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
f1 (ppm)


165.74
152.66
150.59
148.97
136.58
136.24
135.66
131.54
130.85
123.37
123.17
118.72
51.64
Br
COOMe
H
N
E-6
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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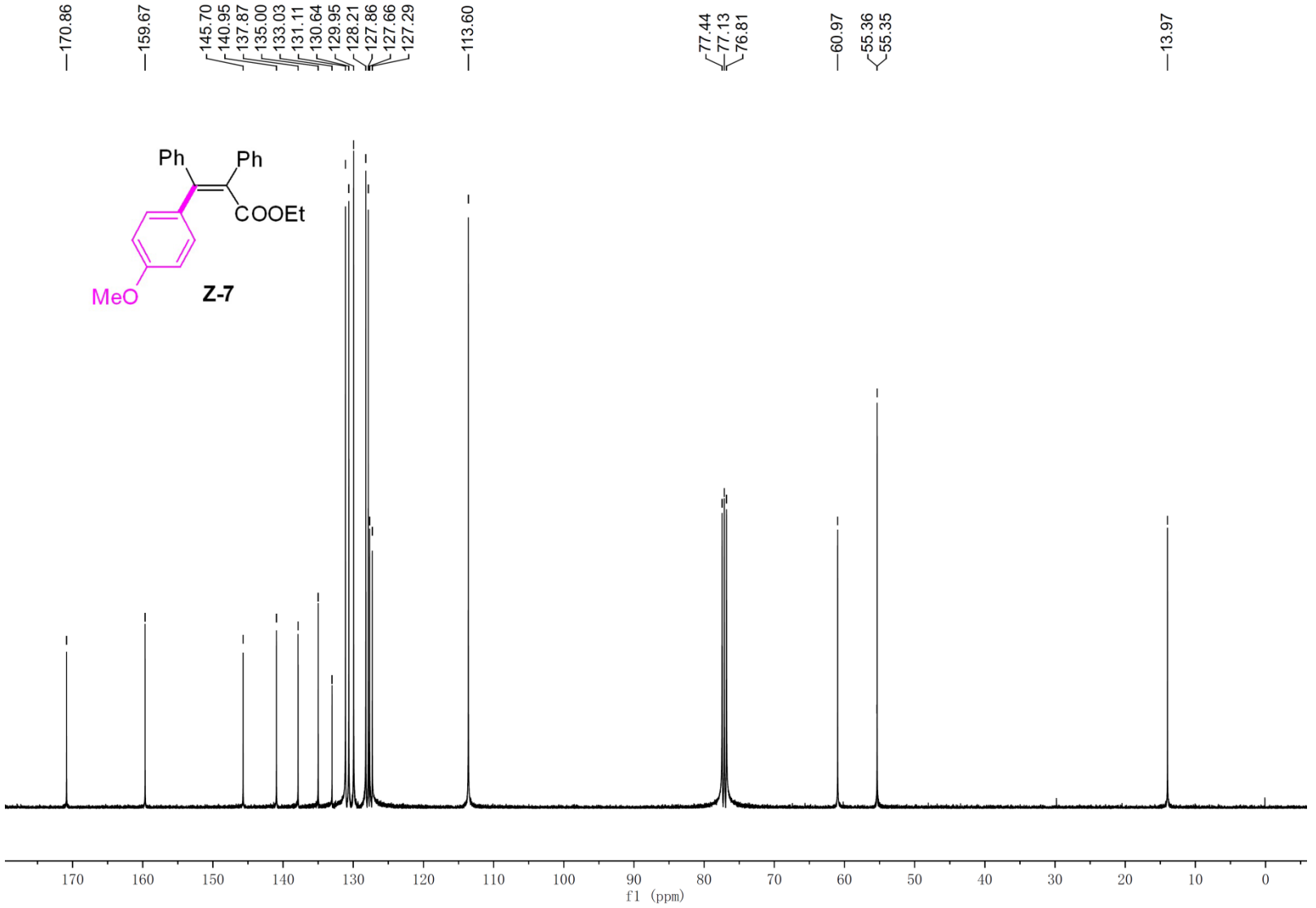
7.222
7.201
7.174
7.172
7.170
7.167
7.160
7.156
7.148
7.143
7.139
7.128
7.124
7.115
7.114
7.108
7.104
7.102
7.096
7.091
7.029
7.027
7.025
7.023
7.021
7.009
7.005
6.868
6.846
4.107
4.090
4.072
4.054
3.822
1.066
1.048
1.030
Ph
Ph
COOEt
MeO
Z-7
2.04
8.03
1.97
1.99
2.00
3.02
3.02
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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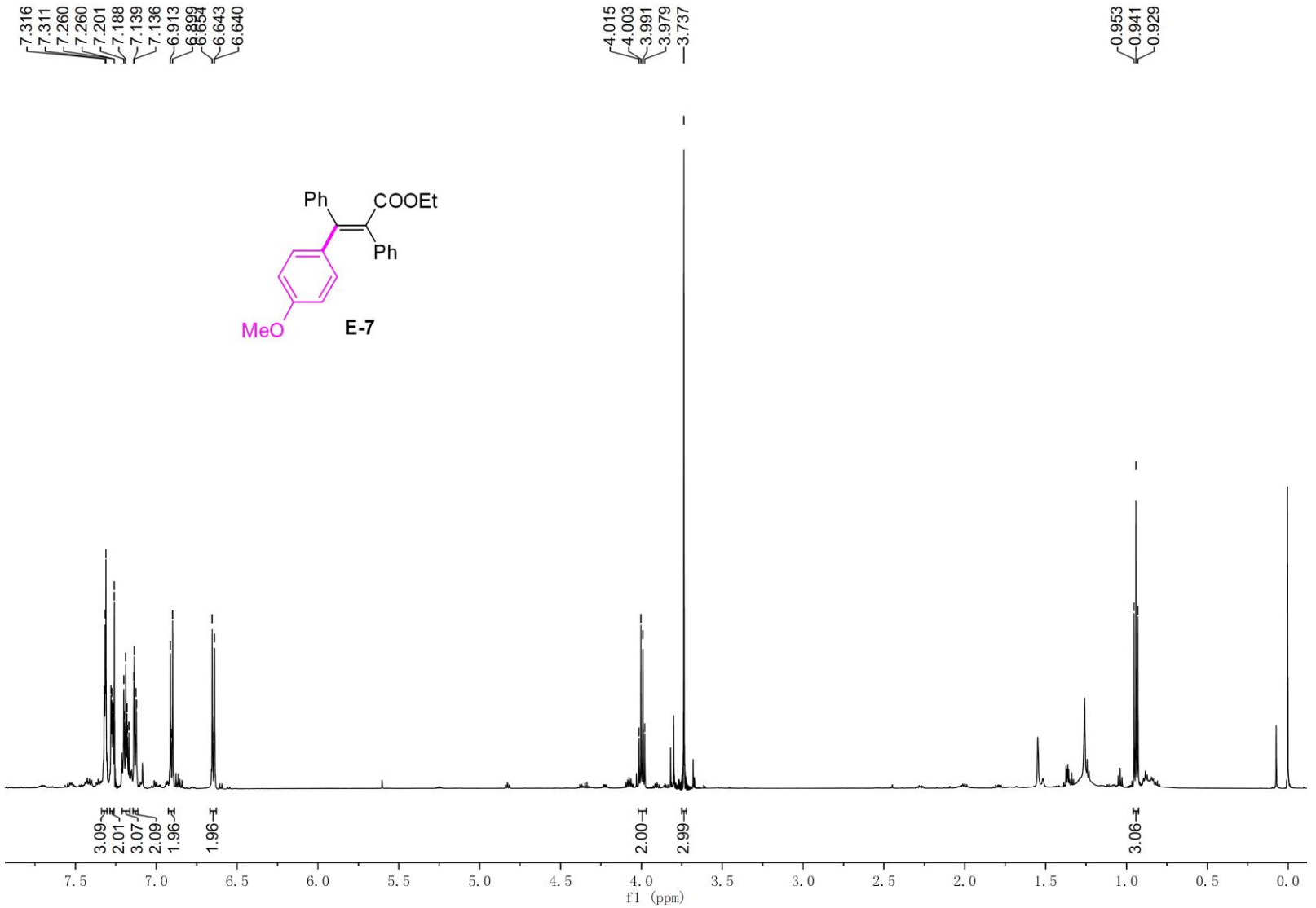
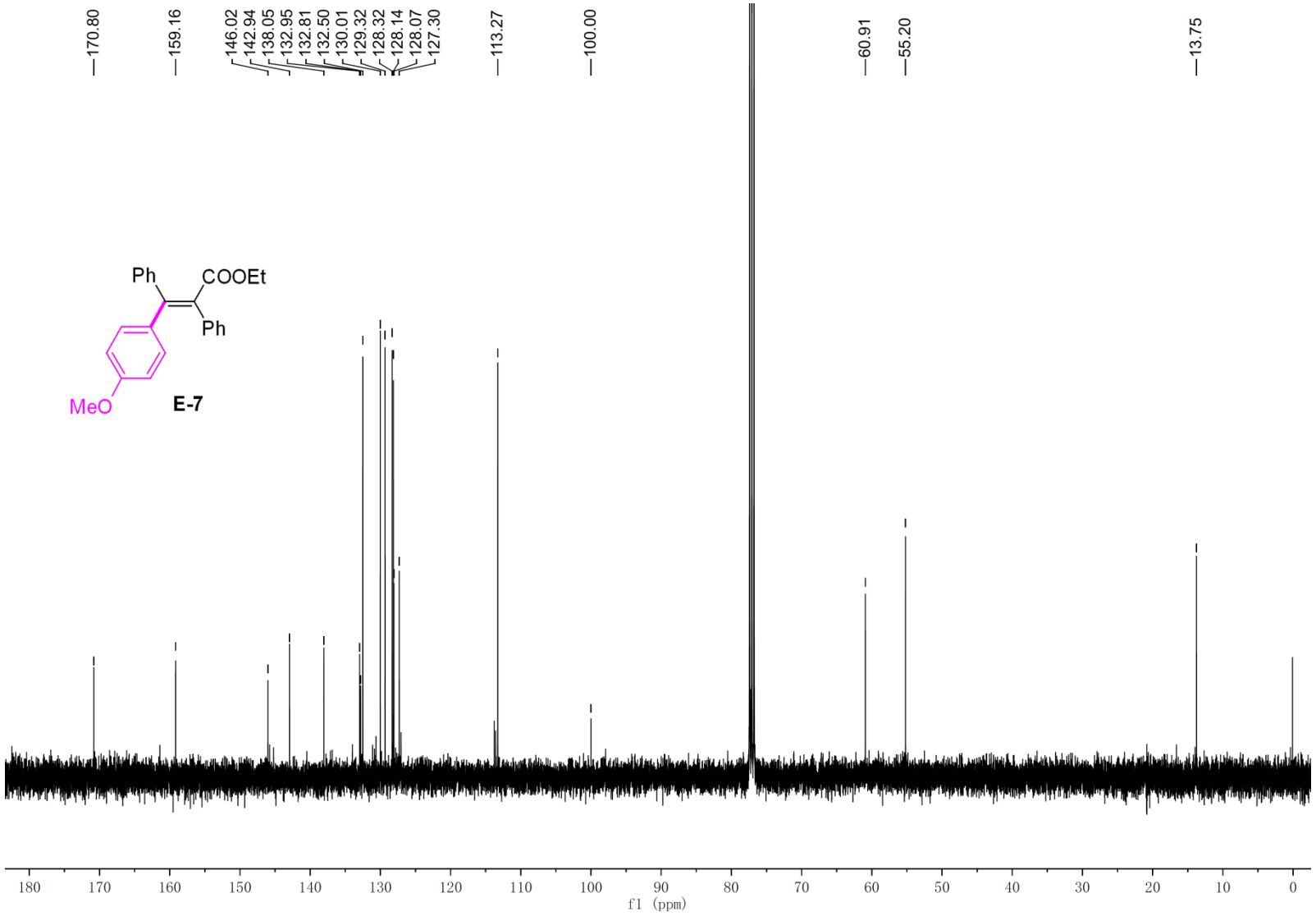
145.70
140.95
137.87
135.00
133.03
131.11
130.64
129.95
128.21
127.86
127.66
127.29
113.60
77.44
77.13
76.81
60.97
55.36
55.35
13.97
Ph
Ph
COOEt
MeO
Z-7
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.316
7.311
7.260
7.260
7.201
7.188
7.139
7.136
6.913
6.899
6.854
6.643
6.640
4.015
4.003
3.991
3.979
3.737
0.953
0.941
0.929
Ph
COOEt
Ph
MeO
E-7
3.09
2.01
3.07
2.09
1.96
1.96
2.00
2.99
3.06
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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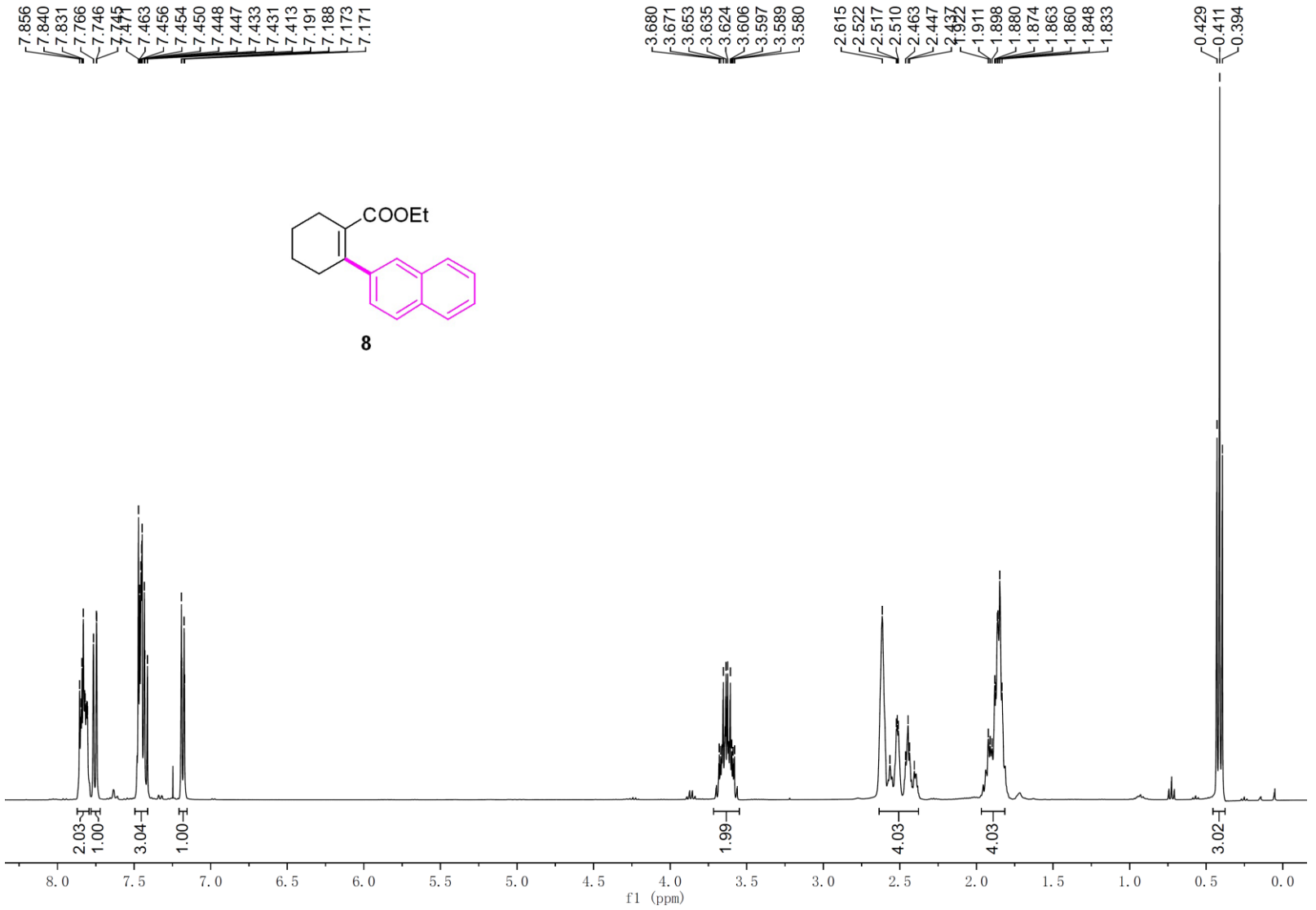
138.05
132.95
132.81
132.50
130.01
129.32
128.32
128.14
128.07
127.30
113.27
100.00
60.91
55.20
13.75
Ph
COOEt
Ph
MeO
E-7
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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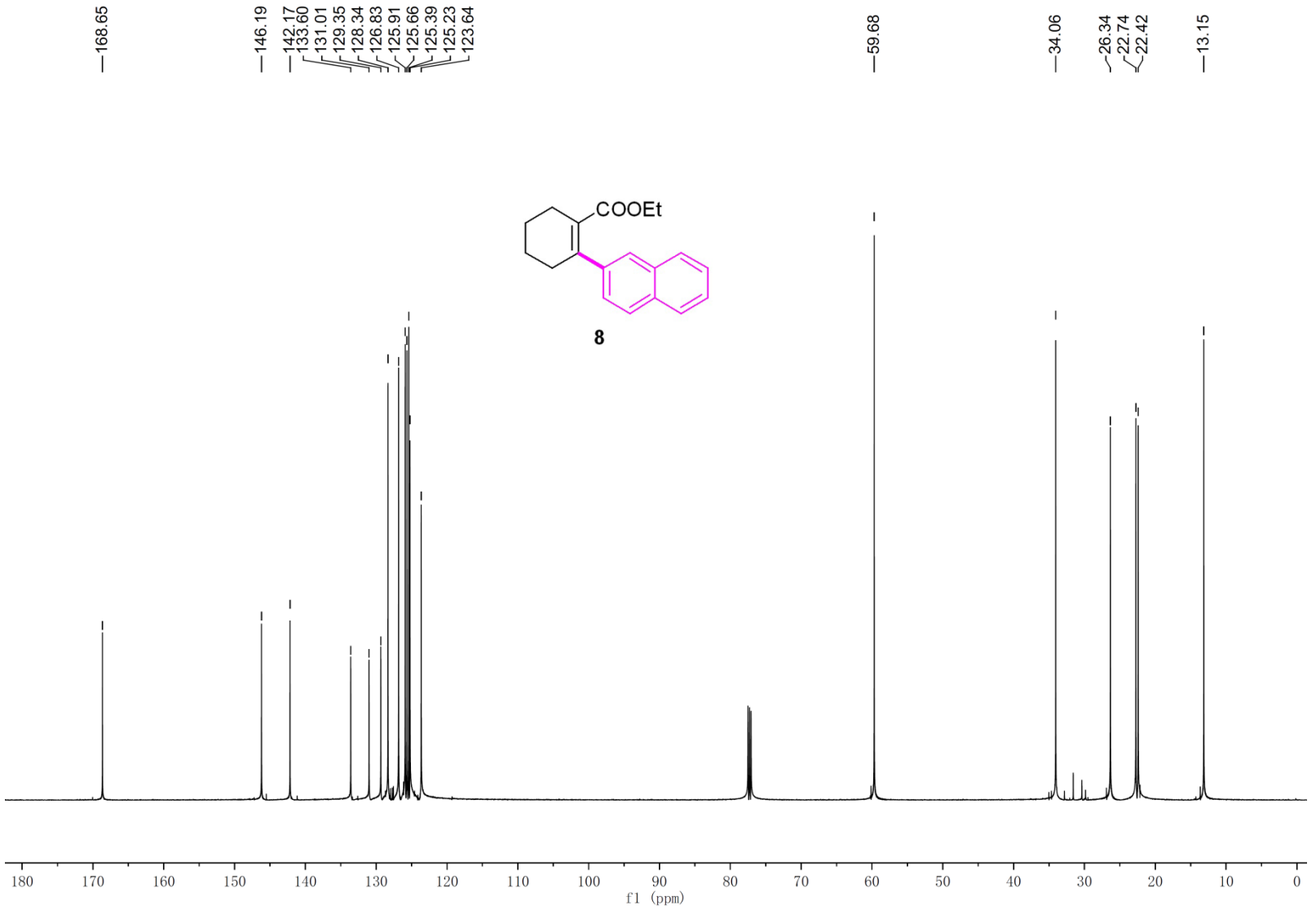
7.456
7.454
7.450
7.448
7.447
7.433
7.431
7.413
7.191
7.188
7.173
7.171
3.680
3.671
3.653
3.635
3.624
3.606
3.597
3.589
3.580
2.615
2.522
2.517
2.510
2.463
2.447
1.922
1.911
1.898
1.880
1.874
1.863
1.860
1.848
1.833
0.429
0.411
0.394
COOEt
8
2.03
1.00
3.04
1.00
1.99
4.03
4.03
3.02
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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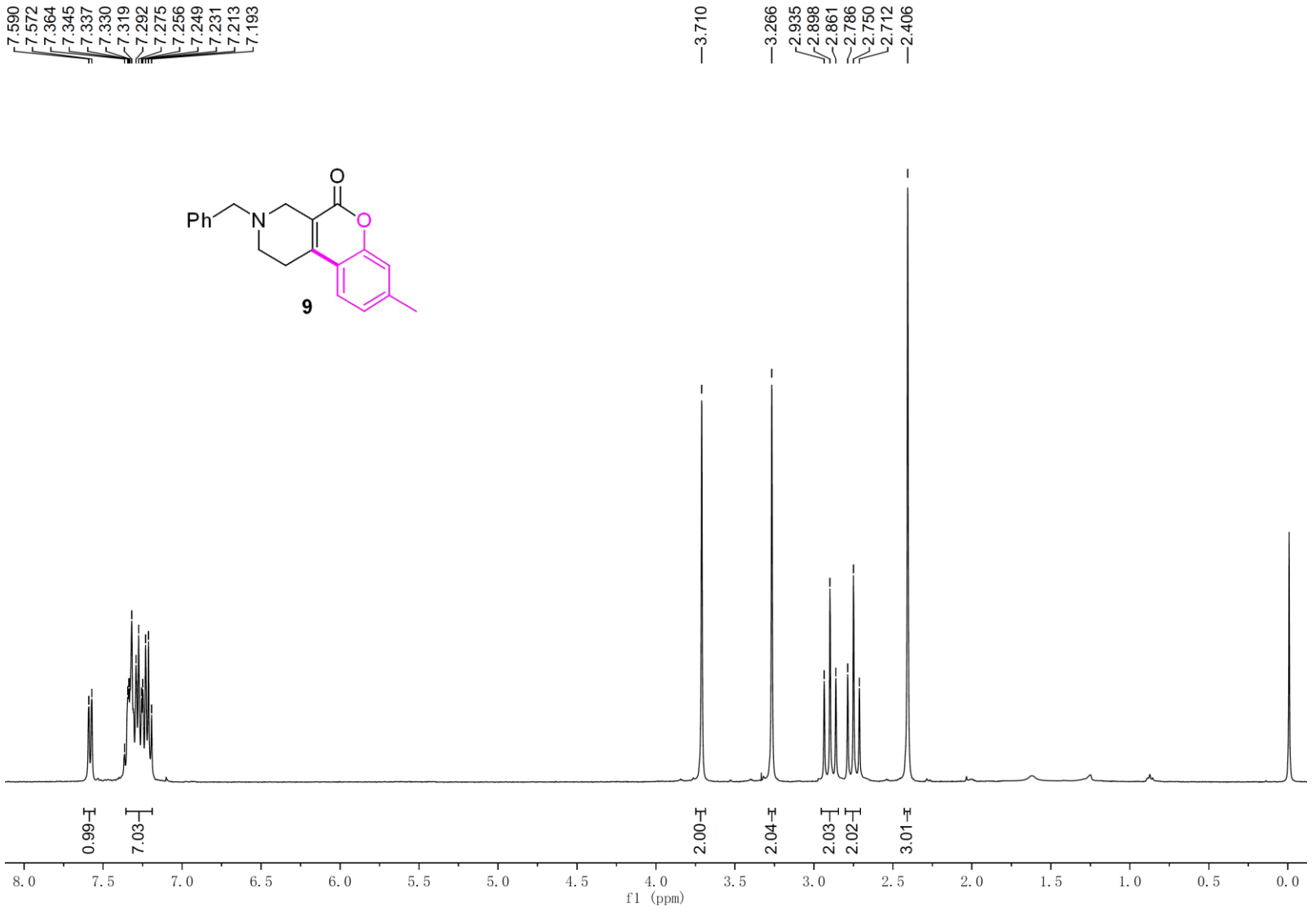
146.19
142.17
133.60
131.01
129.35
128.34
126.83
125.91
125.66
125.39
125.23
123.64
59.68
34.06
26.34
22.74
22.42
13.15
COOEt
8
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.292
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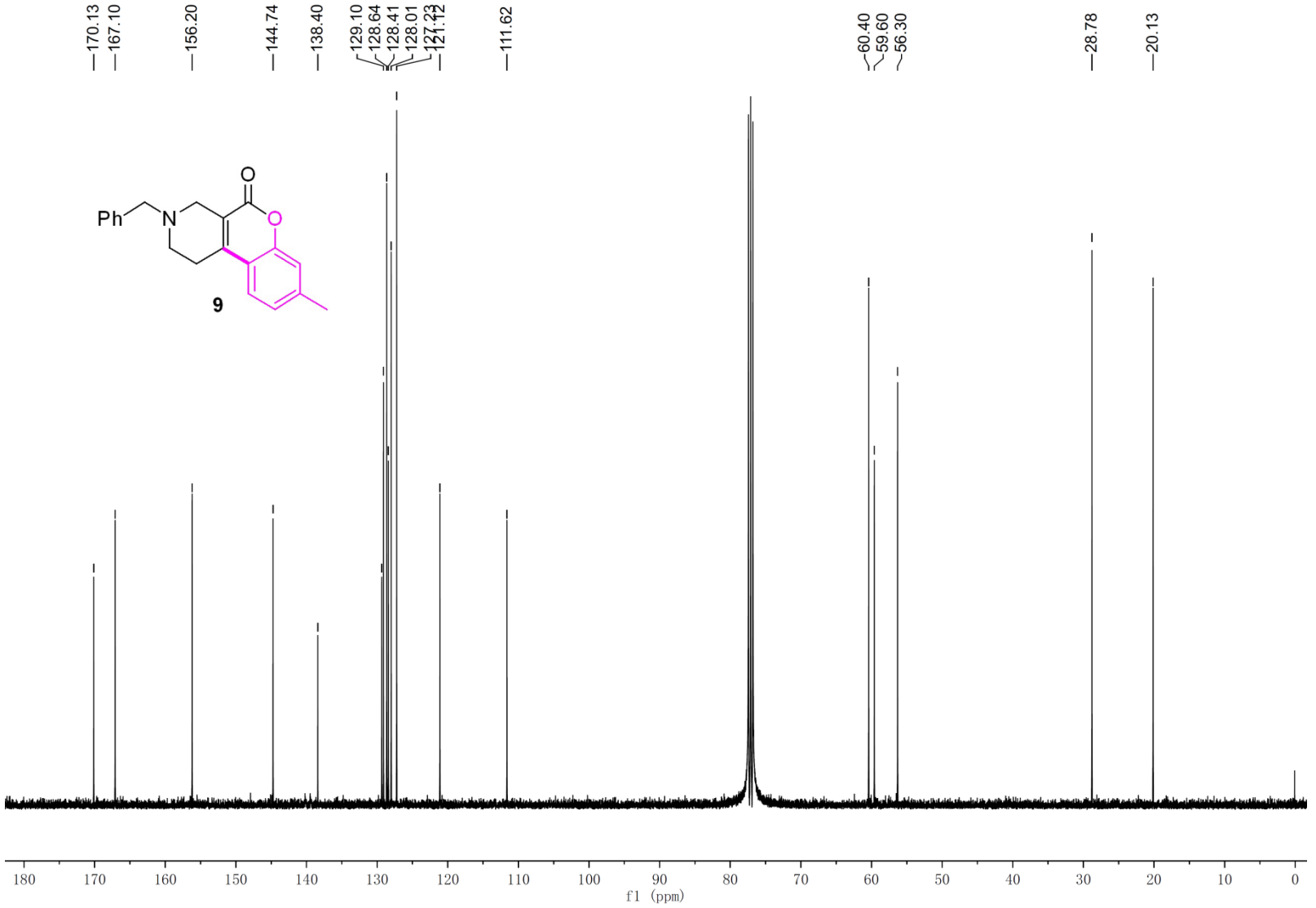
7.249
7.231
7.213
7.193
3.710
3.266
2.935
2.898
2.861
2.786
2.750
2.712
2.406
Ph
N
O
O
9
0.99
7.03
2.00
2.04
2.03
2.02
3.01
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


170.13
167.10
156.20
144.74
138.40
129.10
128.64
128.41
128.01
121.12
111.62
60.40
59.60
56.30
28.78
20.13
Ph
N
O
O
9
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)