Supporting Information

Highly Efficient Cascade Reaction for Selective Formation of Spirocyclobutenes from Dienallenes via Palladium-Catalyzed Oxidative Double Carbocyclization-Carbonylation-Alkynylation

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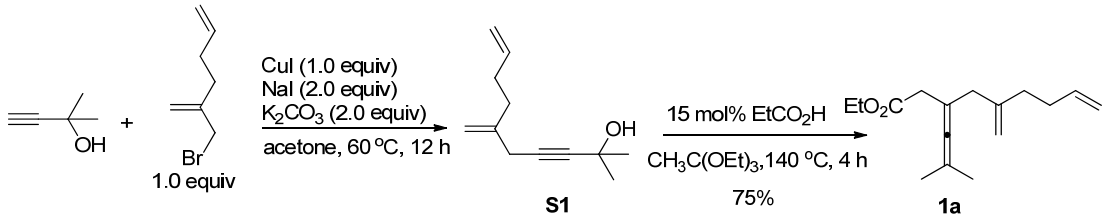
General information

Unless otherwise noted, all reagents were used as received from commercial suppliers. $Pd(TFA)_2$ was obtained from Pressure Chemicals and used without further purification. THF and toluene were obtained from a VAC Solvent Purifier. Reactions were monitored using thin-layer chromatography (SiO_2). TLC plates were visualized with UV light (254 nm) or $KMnO_4$ stain. Flash chromatography was carried out with 60Å (particle size 35-70 μm) normal flash silica gel. NMR spectra were recorded at 400 MHz (^1H) or 500 MHz (^1H) and at 100 MHz (^{13}C) or 125 MHz (^{13}C), respectively. Chemical shifts (δ) are reported in ppm, using the residual solvent peak in $CDCl_3$ (H = 7.26 and C = 77.0 ppm) as internal standard, and coupling constants (*J*) are given in Hz. HRMS were recorded using ESI-TOF techniques.

General procedure for the preparation of starting materials[1]

Preparation of allenes 1a~1f and 1k.

Ethyl 5-methylene-3-(2-methylprop-1-en-1-ylidene)non-8-enoate (**1a**)



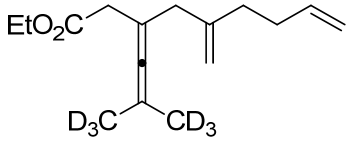
To a three-necked flask were added 2-(bromomethyl)hexa-1,5-diene[2] (522 mg, 3 mmol), 2-methylbut-3-yn-2-ol (252 mg, 3 mmol), CuI (571 mg, 3 mmol), NaI (899 mg, 6 mmol), K_2CO_3 (829 mg, 6 mmol), and acetone (10 mL). After full consumption of starting material as monitored by TLC at 60 °C (12 h) the reaction mixture was filtered to remove precipitates. Evaporation of the solvent afforded the crude product **S1**, which was used as the starting material in the next step without further purification and characterization.

A dry round-bottomed flask was equipped with a distillation receiver and a condenser. Propargylic alcohol **S1** (493 mg, 3 mmol), triethyl orthoacetate (6 mL), and propanoic acid (33 mg. 0.45 mmol) were added sequentially. After the reaction was refluxed for 4 h, the mixture was cooled down to 0 °C in an ice bath. Et_2O (50 mL) and HCl (aq., 1 M, 20 mL) were added. The organic layer was separated, and the aqueous layer was extracted with Et_2O (2 × 50 mL). The combined organic layers were dried over Na_2SO_4, filtered, concentrated in vacuo, and purified via column chromatography on silica gel (eluent: petroleum ether/ethyl ether = 100/1~50/1) to afford the desired product **1a** (558 mg, 75% for two steps): colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.87-5.75 (m, 1H), 5.06-4.92 (m, 2H), 4.84-4.78 (m, 2H), 4.11 (q, J = 7.1 Hz, 2H), 2.87 (s, 2H), 2.75 (s, 2H), 2.23-2.08 (m, 4H), 1.67 (s, 6H), 1.25 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.2, 171.7, 146.2, 138.5, 114.4, 111.7, 95.7, 93.2, 60.4, 41.0, 38.1, 34.3, 31.8, 20.5, 14.2; HRMS (ESI): calc. for $C_{16}H_{24}NaO_2$ $[M+Na]^+$: 271.1669; found: 271.1678.

The general method from above was used for the preparation of the following

dinenes:

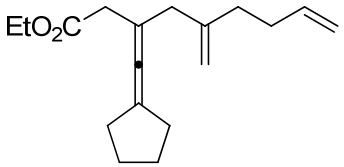
[D_6]- Ethyl 5-methylene-3-(2-methylprop-1-en-1-ylidene)non-8-enoate (**1a-*d*$_6$**)



1a-*d*$_6$

58% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.87-5.74 (m, 1H), 5.05-4.92 (m, 2H), 4.84-4.79 (m, 2H), 4.11 (q, *J* = 7.2 Hz, 2H), 2.87 (s, 2H), 2.75 (s, 2H), 2.24-2.08 (m, 4H), 1.25 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.2, 171.7, 146.2, 138.5, 114.4, 111.7, 93.2, 60.4, 41.0, 38.1, 34.3, 31.8, 14.2; HRMS (ESI): calc. for $C_{16}H_{18}D_6NaO_2$ $[M+Na]^+$: 277.2045; found: 277.2046.

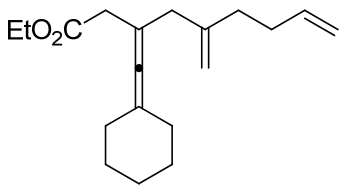
Ethyl 3-(cyclopentylidenemethylene)-5-methylenenon-8-enoate (**1b**)



1b

56% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.88-5.74 (m, 1H), 5.06-4.91 (m, 2H), 4.86-4.75 (m, 2H), 4.11 (q, *J* = 7.1 Hz, 2H), 2.90 (s, 2H), 2.77 (s, 2H), 2.36-2.27 (m, 4H), 2.24-2.08 (m, 4H), 1.68-1.61 (m, 4H), 1.25 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.7, 171.7, 146.2, 138.5, 114.4, 111.6, 104.4, 95.8, 60.4, 41.2, 38.3, 34.3, 31.8, 30.9, 27.0, 14.2; HRMS (ESI): calc. for $C_{18}H_{26}NaO_2$ $[M+Na]^+$: 297.1825; found: 297.1831.

Ethyl 3-(cyclohexylidenemethylene)-5-methylenenon-8-enoate (**1c**)

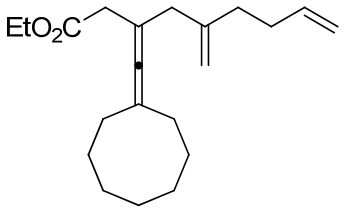


1c

55% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.86-5.74 (m, 1H), 5.05-4.89 (m, 2H), 4.82-4.72 (m, 2H), 4.11 (q, *J* = 7.2 Hz, 2H), 2.88 (s, 2H), 2.76 (s, 2H), 2.23-2.05 (m, 8H), 1.62-1.45 (m, 6H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100

MHz, $CDCl_3$) δ 197.7, 171.7, 146.3, 138.5, 114.4, 111.7, 103.2, 3.0, 60.4, 41.2, 38.4, 34.3, 31.8, 31.5, 27.6, 26.1, 14.2; HRMS (ESI): calc. for $C_{19}H_{28}NaO_2$ $[M+Na]^+$: 311.1982; found:311.1991.

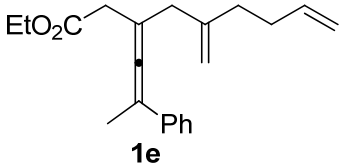
Ethyl 3-(cyclooctylidenemethylene)-5-methylenenon-8-enoate (**1d**)



1d

59% isolated yield, colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 5.89-5.74 (m, 1H), 5.07-4.91 (m, 2H), 4.87-4.78 (m, 2H), 4.12 (q, *J* = 7.2 Hz, 2H), 2.89 (s, 2H), 2.77 (s, 2H), 2.25-2.09 (m, 8H), 1.70-1.47 (m, 10H), 1.25 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.5, 171.8, 146.2, 138.5, 114.4, 111.8, 105.0, 93.6, 60.4, 40.7, 37.9, 34.3, 31.9, 31.7, 27.0, 26.8, 26.2, 14.2; HRMS (ESI): calc. for $C_{21}H_{32}NaO_2$ $[M+Na]^+$: 339.2295; found: 339.2287.

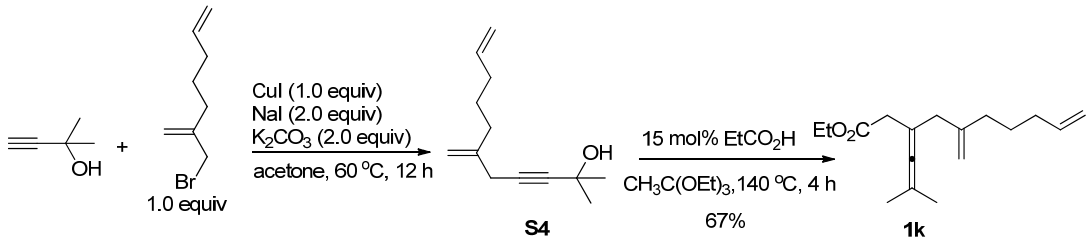
Ethyl 5-methylene-3-(2-phenylprop-1-en-1-ylidene)non-8-enoate (**1i**)



1e

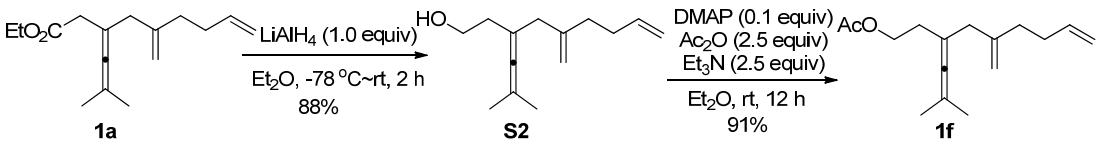
36% isolated yield, colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 7.47-7.40 (m, 2H), 7.34-7.29 (m, 2H), 7.23-7.17 (m, 1H), 5.86-5.70 (m, 1H), 5.04-4.84 (m, 4H), 4.19-4.05 (m, 2H), 3.06 (s, 2H), 2.93 (d, *J* = 14.8 Hz, 1H), 2.89 (d, *J* = 14.8 Hz, 1H), 2.22-2.14 (m, 4H), 2.1 (s, 3H), 1.23 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.8, 171.2, 145.6, 138.2, 137.3, 128.2, 126.6, 125.8, 114.5, 112.3, 101.1, 97.5, 60.6, 40.7, 37.9, 34.4, 31.7, 17.0, 14.1; HRMS (ESI): calc. for $C_{21}H_{26}NaO_2$ $[M+Na]^+$: 333.1825; found: 333.1831.

Ethyl 5-methylene-3-(2-methylprop-1-en-1-ylidene)dec-9-enoate (**1k**)



67% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.87-5.74 (m, 1H), 5.03-4.91 (m, 2H), 4.82-4.75 (m, 2H), 4.11 (q, *J* = 7.1 Hz, 2H), 2.87 (s, 2H), 2.73 (s, 2H), 2.08-2.01 (m, 4H), 1.67 (s, 6H), 1.56-1.47 (m, 2H), 1.24 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.2, 171.7, 146.7, 138.7, 114.5, 111.5, 95.6, 93.3, 60.4, 40.9, 38.1, 34.5, 33.5, 26.8, 20.5, 14.2; HRMS (ESI): calc. for $C_{17}H_{26}NaO_2$ $[M+Na]^+$: 285.1825; found: 285.1830.

Preparation of 5-Methylene-3-(2-methylprop-1-en-1-ylidene)non-8-en-1-yl acetate (**1e**)



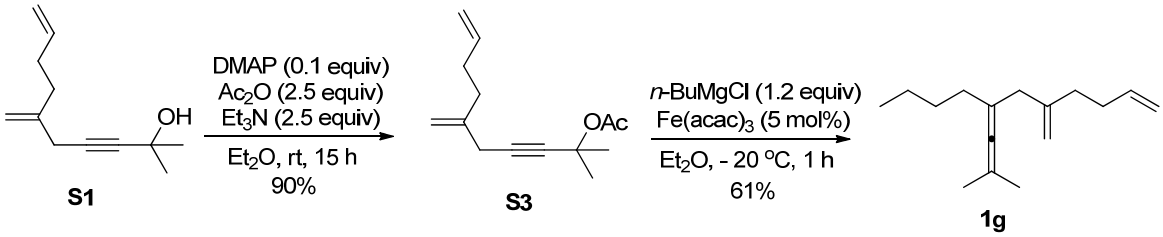
A solution of allene **1a** (496 mg, 2 mmol) in dry Et_2O (5 mL) was added dropwise to a stirred suspension of $LiAlH_4$ (76 mg, 2 mmol) in dry Et_2O (10 mL) at -78 °C under Ar atmosphere. The mixture was stirred for 2 h at room temperature, and then carefully quenched with H_2O (2 mL). The organic layer was separated, and the aqueous layer was extracted with Et_2O (2 × 30 mL). The combined organic layers were dried over Na_2SO_4, filtered, and concentrated in vacuo. The residue was dissolved in Et_2O (20 mL), and quickly filtered via a short column of silica gel (2 cm, eluent: 20 mL of Et_2O). Evaporation of the solvent afforded the pure product **S2** (363 mg, 88%): colorless oil, which was used as the starting material in the next step without further characterization.

To a three-necked flask were added propargyl alcohol **S2** (206 g, 1 mmol), DMAP (12.2 mg, 0.1 mmol), Et_2O (3 mL), Et_3N (0.35 mL, 2.5 mmol), and Ac_2O (0.24 mL, 2.5 mmol). After full consumption of starting material as monitored by TLC at room temperature (12 h), the reaction was carefully quenched with water (5 mL). The

organic layer was separated, and the aqueous layer was extracted with Et_2O (2 × 10 mL). The combined organic layers were dried over Na_2SO_4, filtered, and concentrated in vacuo. The crude product was purified via column chromatography on silica gel (eluent: petroleum ether/ethyl ether = 30/1) to afford the desired acetate **1f** (228 mg, 91%): colorless oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.87-5.74 (m, 1H), 5.05-4.91 (m, 2H), 4.83-4.75 (m, 2H), 4.10 (t, *J* = 6.8 Hz, 2H), 2.68 (s, 2H), 2.23-2.14 (m, 4H), 2.02 (s, 3H), 1.65 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.8, 171.0, 146.5, 138.4, 114.4, 111.3, 95.9, 95.3, 63.0, 41.5, 34.3, 31.8, 30.5, 20.9, 20.6; HRMS (ESI): calc. for $C_{16}H_{24}NaO$ $[M+Na]^+$: 271.1669; found: 271.1677.

Preparation of allenes 1g~1i.[3]

5-Methylene-7-(2-methylprop-1-en-1-ylidene)undec-1-ene (**1g**)



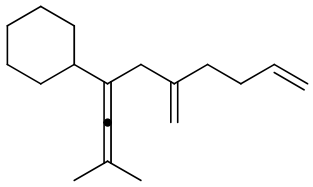
To a three-necked flask were added propargyl alcohol **S1** (712 mg, 4 mmol), DMAP (49 mg, 0.8 mmol), Et_2O (10 mL), Et_3N (1.4 mL, 10 mmol), and Ac_2O (0.95 mL, 10 mmol). After full consumption of starting material as monitored by TLC at room temperature (15 h), the reaction was carefully quenched with water (10 mL). The organic layer was separated, and the aqueous layer was extracted with Et_2O (2 × 30 mL). The combined organic layers were dried over Na_2SO_4, filtered, concentrated in vacuo. The crude product was purified via column chromatography on silica gel (eluent: petroleum ether/ethyl ether = 30/1) to afford the desired acetate **S3** (792 mg, 90%): colorless oil, which was used as the starting material in the next step without further characterization.

A solution of *n*-BuMgCl (0.9 mL, 1.8 mmol, 2.0 M/THF) was added dropwise to a stirred suspension of propargylic alcohol acetate **S3** (309 mg, 1.5 mmol) and $Fe(acac)_3$ (26.6 mg, 0.075 mmol) in dry Et_2O (7.5 mL) at -20 °C under Ar atmosphere. The mixture was stirred for 1 h at -20 °C, and then carefully quenched with citric acid

aqueous solution (2 mL, 10%). The organic layer was separated, and the aqueous layer was extracted with Et_2O (2 × 30 mL). The combined organic layers were dried over Na_2SO_4, filtered, concentrated in vacuo, and purified via column chromatography on silica gel (eluent: petroleum ether) to afford the desired product **1g** (199 mg, 61%): colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.89-5.77 (m, 1H), 5.07-4.91 (m, 2H), 4.82-4.75 (m, 2H), 2.66 (s, 2H), 2.26-2.08 (m, 4H), 1.87-1.81 (m, 2H), 1.66 (s, 6H), 1.40-1.27 (m, 4H), 0.89 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.8, 147.2, 138.7, 114.3, 110.8, 99.2, 94.5, 41.4, 34.4, 31.9, 31.4, 29.8, 22.3, 20.8, 14.1; HRMS (ESI): calc. for $C_{16}H_{26}Na$ $[M+Na]^+$: 244.1927; found: 244.1900.

The general method from above was used for the preparation of the following compounds:

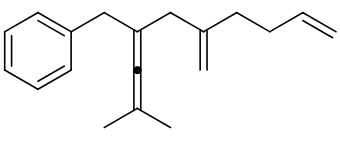
(2-Methyl-6-methylenedeca-2,3,9-trien-4-yl)cyclohexane (**1h**)



1h

59% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.90-5.75 (m, 1H), 5.06-4.91 (m, 2H), 4.81-4.73 (m, 2H), 2.69 (s, 2H), 2.24-2.07 (m, 4H), 1.82-1.58 (m, 12H), 1.31-0.97 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.5, 147.6, 138.7, 114.3, 110.7, 104.9, 95.4, 40.0, 39.5, 34.5, 32.6, 31.9, 26.5, 26.4, 20.9; HRMS (ESI): calc. for $C_{18}H_{28}Na$ $[M+Na]^+$:267.2063; found: 267.2138.

(4-Methylene-2-(2-methylprop-1-en-1-ylidene)oct-7-en-1-yl)benzene (**1i**)



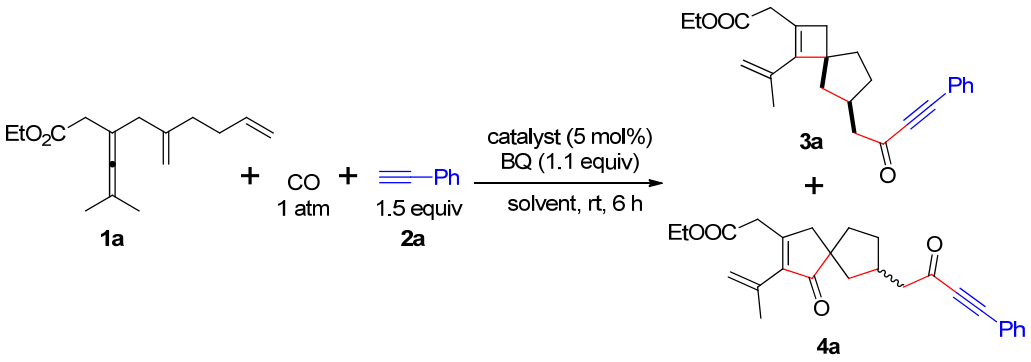
1i

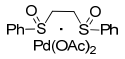
78% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31-7.25 (m, 2H), 7.22-7.17 (m, 3H), 5.90-5.76 (m, 1H), 5.07-4.93 (m, 2H), 4.86-4.79 (m, 2H), 3.21 (s, 2H), 2.64 (s, 2H), 2.23-2.10 (m, 4H), 1.64 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ

201.2, 146.8, 140.4, 138.6, 129.0, 128.0, 125.8, 114.3, 111.3, 98.8, 94.6, 40.1, 38.9, 34.5, 31.8, 20.6; HRMS (ESI): calc. for $C_{19}H_{24}Na$ $[M+Na]^+$: 275.1770; found: 275.1775.

Optimization of Oxidative Carbocyclization of Dienallenes:

Table S1. Optimization of the reaction conditions[a]



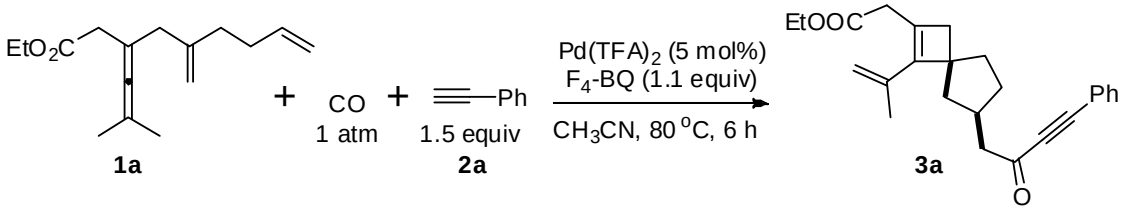
Entry	Catalyst	Solvent	Yield of **3a** (%)[b]	Yield of **4a** (%)[b]	Recovery of **1a** (%)[b]
1	$Pd(OAc)_2$	DCE	3	41	33
2	**$Pd(TFA)_2$**	**DCE**	**-**	**90**	**-**
3	$Pd(PPh_3)_2Cl_2$	DCE	-	-	90
4		DCE	-	77	-
5	$Pd(TFA)_2$	THF	4	69	-
6	$Pd(TFA)_2$	acetone	7	64	-
7	$Pd(TFA)_2$	toluene	3	81	-
8	$Pd(TFA)_2$	dioxane	2	85	-
9	$Pd(TFA)_2$	MeCN	12	20	45
10[c]	$Pd(TFA)_2$	MeCN	18	35	20
11[d]	$Pd(TFA)_2$	MeCN	35	38	-
12[e]	$Pd(TFA)_2$	MeCN	56	16	-
13[e,f]	$Pd(TFA)_2$	MeCN	65	9	-
14[e,f,g]	$Pd(TFA)_2$	MeCN	47	8	10
15[e,f,h]	**$Pd(TFA)_2$**	**MeCN**	**75**	**3**	**-**

[a]The reaction was conducted in the indicated solvent (1 mL) at room temperature using **1a** (0.1 mmol), **2a** (1.5 equiv), BQ (1.1 equiv) in the presence of the palladium catalyst (5 mol%). [b]Determined by ^{1}HNMR using anisole as the internal standard. [c]The reaction was conducted at 40 °C. [d]The reaction was conducted at 60 °C. [e]The reaction was conducted at 80 °C. [f]3 mL MeCN was used. [g]2,6-Dimethyl-BQ was used instead of BQ. [h]F_4-BQ was used instead of BQ.

General procedure for palladium-catalyzed oxidative carbocyclization of enallenes for the formation of 3

Representative procedure A for the synthesis of 3a-3i.

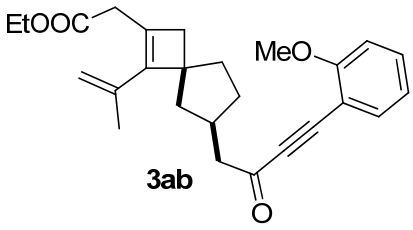
Ethyl 2-(6-(2-oxo-4-phenylbut-3-yn-1-yl)-1-(prop-1-en-2-yl)spiro[3.4]oct-1-en-2-yl) acetate (**3a**)



To a solution of $Pd(TFA)_2$ (3.3 mg, 0.01 mmol), F_4-BQ (39.6 mg, 0.22 mmol) and **2a** (30.6 mg, 0.3 mmol) in MeCN (6 mL) were added enallene **1a** (49.6 mg, 0.2 mmol). The tube was closed with a septum. The tube was evacuated and filled with carbon monoxide gas (repeated three times) using a balloon. The reaction was stirred at 80 ^oC for 6 h. After full consumption of starting material **1a** as monitored by TLC, the reaction mixture was evaporated and purified via column chromatography on silica gel (eluent: petroleum ether/ethyl ether = 15/1) afforded **3a** (56.4 mg, 75%): colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 7.60-7.54 (m, 2H, ArH), 7.48-7.42 (m, 1H, ArH), 7.41-7.35 (m, 2H, ArH), 4.91-4.84 (m, 1H, one proton of $=CH_2$), 4.67-4.62 (m, 1H, one proton of $=CH_2$), 4.08 (q, J = 7.1 Hz, 2H, OCH_2), 3.08 (d, J = 15.7 Hz, 1H, one proton of CH_2), 2.94 (d, J = 15.7 Hz, 1H, one proton of CH_2), 2.86 (d, J = 14.2 Hz, 1H, one proton of CH_2), 2.82 (d, J = 14.2 Hz, 1H, one proton of CH_2), 2.47 (t, J = 4.0 Hz, 1H, one proton of CH_2), 2.37 (d, J = 16.8 Hz, 1H, one proton of CH_2), 2.11 (d, J = 16.8 Hz, 1H, one proton of CH_2), 1.89-1.71 (m, 8H, CH_3 + CH_2 + one proton of CH_2), 1.64 (ddd, J = 10.7, 4.1, 1.0 Hz, 1H, CH), 1.22 (t, J = 7.1 Hz, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.1, 172.2, 145.4, 144.9, 133.0, 130.7, 128.6, 120.9, 120.1, 90.5, 89.3, 60.3, 56.2, 45.7, 42.0, 41.5, 39.5, 38.4, 35.8, 34.7, 22.1, 14.2; HRMS (ESI): calc. for $C_{25}H_{28}NaO_3$ $[M+Na]^+$: 399.1931; found: 399.1926.

The general method from above was used for the preparation of the following compounds **3**:

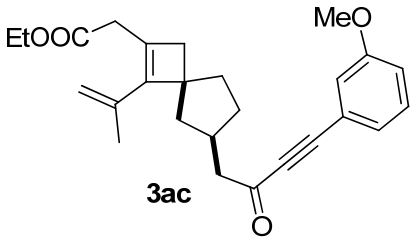
Ethyl 2-(6-(4-(2-methoxyphenyl)-2-oxobut-3-yn-1-yl)-1-(prop-1-en-2-yl)spiro[3.4]oc t-1-en-2-yl)acetate (**3ab**)



3ab

72% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.41 (ddd, *J* = 9.2, 7.5, 1.8 Hz, 1H), 6.94 (td, *J* = 7.6, 1.0 Hz, 1H), 6.90 (d, *J* = 8.4 Hz,1H), 4.91-4.83 (m, 1H), 4.68-4.61 (m, 1H), 4.08 (q, *J* = 7.1 Hz, 2H), 3.89 (s, 3H), 3.07 (d, *J* = 15.7 Hz, 1H), 2.93 (d, *J* = 15.7 Hz, 1H), 2.86 (d, *J* = 14.2 Hz, 1H), 2.82 (d, *J* = 14.2 Hz, 1H), 2.48-2.43 (m, 1H), 2.38 (d, *J* = 16.9 Hz, 1H), 2.10 (d, *J* = 16.9 Hz, 1H), 1.89-1.73 (m, 8H), 1.65 (ddd, *J* = 10.7, 4.1, 1.2 Hz, 1H), 1.22 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.1, 172.2, 161.6, 145.4, 145.0, 134.9, 132.4, 120.9, 120.5, 113.1, 110.8, 109.3, 93.4, 87.9, 60.3, 56.2, 55.8, 45.7, 41.9, 41.4, 39.6, 38.4, 35.8, 34.8, 22.1, 14.2; HRMS (ESI): calc. for $C_{26}H_{30}NaO_4$ $[M+Na]^+$: 429.2036; found: 429.2030.

Ethyl 2-(6-(4-(3-methoxyphenyl)-2-oxobut-3-yn-1-yl)-1-(prop-1-en-2-yl)spiro[3.4]oct -1-en-2-yl)acetate (**3ac**)

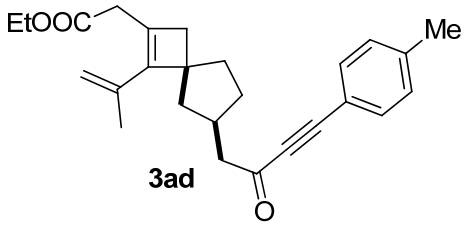


3ac

74% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (t, *J* = 8.0 Hz, 1H), 7.41 (td, *J* = 7.6, 1.3 Hz, 1H), 7.08 (dd, *J* = 2.5, 1.4 Hz, 1H), 7.00 (ddd, *J* = 8.3, 2.7, 1.0 Hz, 1H), 4.90-4.84 (m, 1H), 4.68-4.61 (m, 1H), 4.08 (q, *J* = 7.1 Hz, 2H), 3.82 (s, 3H), 3.08 (d, *J* = 15.7 Hz, 1H), 2.94 (d, *J* = 15.7 Hz, 1H), 2.86 (d, *J* = 14.3 Hz, 1H), 2.82 (d, *J* = 14.3 Hz, 1H), 2.47 (t, *J* = 4.1 Hz, 1H), 2.37 (d, *J* = 16.9 Hz, 1H), 2.11 (d, *J* = 16.9 Hz, 1H), 1.89-1.73 (m, 8H), 1.63 (ddd, *J* = 10.7, 4.1, 1.1 Hz, 1H), 1.22 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.1, 172.2, 159.4, 145.5, 144.9, 129.7, 125.5, 121.0, 120.9, 117.5, 117.4, 113.0, 90.4, 88.9, 60.3, 56.1, 55.4, 45.7, 42.0, 41.5,

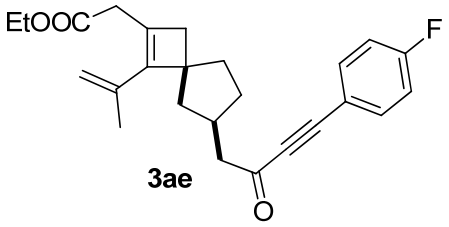
39.5, 38.4, 35.8, 34.7, 22.1, 14.2; HRMS (ESI): calc. for $C_{26}H_{30}NaO_4$ $[M+Na]^+$: 429.2036; found: 429.2033.

Ethyl e2-(6-(2-oxo-4-(p-tolyl)but-3-yn-1-yl)-1-(prop-1-en-2-yl)spiro[3.4]oct-1-en-2 -yl)acetate (**3ad**)



79% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49-7.44 (m, 2H), 7.21-7.16 (m, 2H), 4.89-4.84 (m, 1H), 4.66-4.62 (m, 1H), 4.08 (q, *J* = 7.1 Hz, 2H), 3.07 (d, *J* = 15.7 Hz, 1H), 2.93 (d, *J* = 15.7 Hz, 1H), 2.84 (d, *J* = 14.2 Hz, 1H), 2.82 (d, *J* = 14.2 Hz, 1H), 2.48-2.43 (m, 1H), 2.40-2.33 (m, 4H), 2.10 (d, *J* = 16.8 Hz, 1H), 1.89-1.71 (m, 8H), 1.63 (ddd, *J* = 10.6, 4.1, 1.0 Hz, 1H), 1.22 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.1, 172.2, 145.4, 144.9, 141.4, 133.0, 129.4, 120.9, 117.0, 113.2, 91.2, 89.2, 60.3, 56.2, 45.7, 42.0, 41.5, 39.5, 38.4, 35.8, 34.7, 22.1, 21.7, 14.2; HRMS (ESI): calc. for $C_{26}H_{30}NaO_3$ $[M+Na]^+$: 413.2087; found: 413.2096.

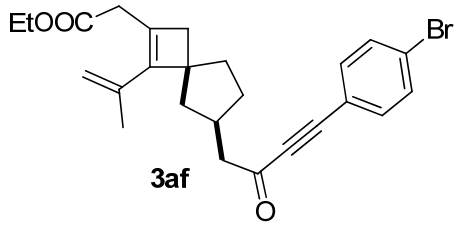
Ethyl 2-(6-(4-(4-fluorophenyl)-2-oxobut-3-yn-1-yl)-1-(prop-1-en-2-yl)spiro[3.4]oct -1-en-2-yl)acetate (**3ae**)



64% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61-7.53 (m, 2H), 7.12-7.03 (m, 2H), 4.90-4.84 (m, 1H), 4.66-4.62 (m, 1H), 4.08 (q, *J* = 7.1 Hz, 2H), 3.07 (d, *J* = 15.7 Hz, 1H), 2.93 (d, *J* = 15.8 Hz, 1H), 2.85 (d, *J* = 14.3 Hz, 1H), 2.80 (d, *J* = 14.3 Hz, 1H), 2.46 (t, *J* = 4.1 Hz, 1H), 2.36 (d, *J* = 16.9 Hz, 1H), 2.10 (d, *J* = 16.9 Hz, 1H), 1.90-1.69 (m, 8H), 1.62 (ddd, *J* = 10.6, 4.1, 1.1 Hz, 1H), 1.22 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.9, 172.2, 164 (d, *J* = 254 Hz), 145.4, 144.9, 135 (d, *J* = 254 Hz), 120.8, 116.18 (d, *J* = 3.5 Hz), 116.15 (d, *J* = 22.2 Hz), 113.2, 89 (d, *J* = 24.5 Hz), 60.3, 56.1, 45.7, 42.0, 41.5, 39.5, 38.4, 35.8, 34.7, 22.0, 14.2; ^{19}F

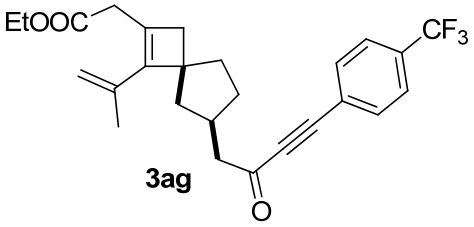
NMR (376 MHz, $CDCl_3$) δ -106.3; HRMS (ESI): calc. for $C_{25}H_{27}FNaO_3$ $[M+Na]^+$: 417.1836; found: 417.1831.

Ethyl 2-(6-(4-(4-bromophenyl)-2-oxobut-3-yn-1-yl)-1-(prop-1-en-2-yl)spiro[3.4]oct-1 -en-2-yl)acetate (**3af**)



66% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55-7.50 (m, 2H), 7.44-7.40 (m, 2H), 4.89-4.85 (m, 1H), 4.66-4.62 (m, 1H), 4.08 (q, *J* = 7.1 Hz, 2H), 3.07 (d, *J* = 15.7 Hz, 1H), 2.93 (d, *J* = 15.7 Hz, 1H), 2.85 (d, *J* = 14.4 Hz, 1H), 2.80 (d, *J* = 14.4 Hz, 1H), 2.46 (t, *J* = 4.2 Hz, 1H), 2.36 (d, *J* = 16.9 Hz, 1H), 2.10 (d, *J* = 16.9 Hz, 1H), 1.88-1.71 (m, 8H), 1.62 (ddd, *J* = 10.7, 4.0, 1.1 Hz, 1H), 1.22 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.9, 172.2, 145.5, 144.9, 134.2, 132.0, 125.5, 120.8, 119.0, 113.2, 90.0, 89.0, 60.3, 56.1, 45.6, 41.9, 41.5, 39.5, 38.4, 35.8, 34.7, 22.0, 14.2; HRMS (ESI): calc. for $C_{25}H_{27}Br^{81}NaO_3$ $[M+Na]^+$: 479.1017; found: 479.1007.

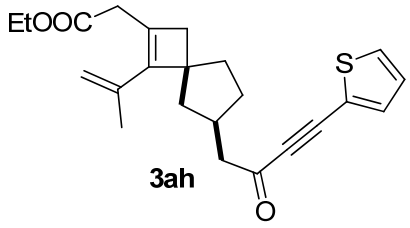
Ethyl 2-(6-(2-oxo-4-(4-(trifluoromethyl)phenyl)but-3-yn-1-yl)-1-(prop-1-en-2-yl)spiro [3.4]oct-1-en-2-yl)acetate (**3ag**)



77% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70-7.62 (m, 4H), 4.90-4.85 (m, 1H), 4.67-4.62 (m, 1H), 4.12-4.05 (m, 2H), 3.08 (d, *J* = 15.7 Hz, 1H), 2.94 (d, *J* = 15.6 Hz, 1H), 2.88 (d, *J* = 14.5 Hz, 1H), 2.83 (d, *J* = 14.5 Hz, 1H), 2.47 (t, *J* = 4.3 Hz, 1H), 2.37 (d, *J* = 16.8 Hz, 1H), 2.11 (d, *J* = 16.7 Hz, 1H), 1.89-1.72 (m, 8H), 1.63 (ddd, *J* = 10.5, 4.0, 1.1 Hz, 1H), 1.22 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.7, 172.2, 145.5, 144.8, 133.0, 132.2 (q, *J* = 33.1 Hz), 125.5 (q, *J* = 3.7 Hz), 122.9, 123.5 (q, *J* = 273 Hz), 120.8, 113.3, 90.3, 87.9, 60.3, 56.2, 45.6,

41.9, 41.5, 39.5, 38.4, 35.8, 34.7, 22.0, 14.2; ^{19}F NMR (376 MHz, $CDCl_3$) δ -63.2; HRMS (ESI): calc. for $C_{26}H_{27}F_3NaO_3$ $[M+Na]^+$: 467.1805; found: 467.1800.

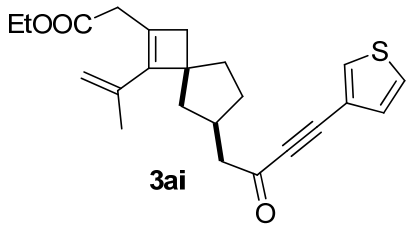
Ethyl 2-(6-(2-oxo-4-(thiophen-2-yl)but-3-yn-1-yl)-1-(prop-1-en-2-yl)spiro[3.4]oct-1 -en-2-yl)acetate (**3ah**)



3ah

83% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (ddd, *J* = 7.1, 5.1, 1.2 Hz, 2H), 7.07 (dd, *J* = 5.1, 3.7 Hz, 1H), 4.89-4.84 (m, 1H), 4.67-4.62 (m, 1H), 4.08 (q, *J* = 7.1 Hz, 2H), 3.07 (d, *J* = 15.6 Hz, 1H), 2.93 (d, *J* = 15.7 Hz, 1H), 2.84 (d, *J* = 14.3 Hz, 1H), 2.79 (d, *J* = 14.3 Hz, 1H), 2.46 (t, *J* = 4.0 Hz, 1H), 2.36 (d, *J* = 16.9 Hz, 1H), 2.09 (d, *J* = 16.9 Hz, 1H), 1.89-1.70 (m, 8H), 1.63 (ddd, *J* = 10.7, 4.0, 1.2 Hz, 1H), 1.22 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.6, 172.2, 145.4, 144.9, 136.5, 131.6, 127.7, 120.8, 119.9, 113.2, 93.8, 84.5, 60.3, 55.9, 45.7, 42.0, 41.5, 39.5, 38.4, 35.8, 34.7, 22.0, 14.2; ^{19}F NMR (376 MHz, $CDCl_3$) δ -106.3; HRMS (ESI): calc. for $C_{23}H_{26}NaO_3S$ $[M+Na]^+$: 405.1495; found: 405.1481.

Ethyl 2-(6-(2-oxo-4-(thiophen-3-yl)but-3-yn-1-yl)-1-(prop-1-en-2-yl)spiro[3.4]oct-1 -en-2-yl)acetate (**3ai**)

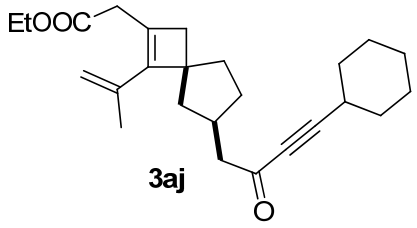


3ai

71% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (dd, *J* = 3.0, 1.2 Hz, 1H), 7.34 (dd, *J* = 5.0, 3.0 Hz, 1H), 7.34 (dd, *J* = 5.1, 1.2 Hz, 1H), 4.89-4.85 (m, 1H), 4.66-4.62 (m, 1H), 4.08 (q, *J* = 7.2 Hz, 2H), 3.07 (d, *J* = 15.6 Hz, 1H), 2.93 (d, *J* = 15.6 Hz, 1H), 2.84 (d, *J* = 14.3 Hz, 1H), 2.79 (d, *J* = 14.3 Hz, 1H), 2.46 (t, *J* = 4.1 Hz, 1H), 2.36 (d, *J* = 16.8 Hz, 1H), 2.10 (d, *J* = 16.9 Hz, 1H), 1.88-1.71 (m, 8H), 1.62 (ddd, *J* = 10.7, 4.1, 1.1 Hz, 1H), 1.22 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.1, 172.2, 145.4, 144.9, 133.7, 130.2, 126.2, 120.9, 119.4, 89.5, 85.9,

60.3, 56.1, 45.7, 42.0, 39.5, 38.5, 35.8, 34.7, 22.1, 14.2; HRMS (ESI): calc. for $C_{23}H_{26}NaO_3S$ $[M+Na]^+$: 405.1495; found: 405.1493.

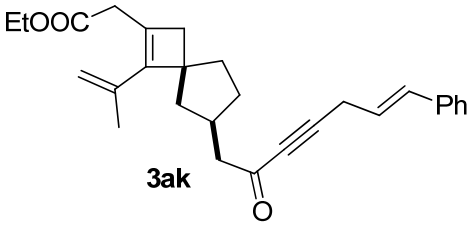
Ethyl 2-(6-(4-cyclohexyl-2-oxobut-3-yn-1-yl)-1-(prop-1-en-2-yl)spiro[3.4]oct-1-en-2 -yl)acetate (**3aj**)



3aj

66% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.88-4.85 (m, 1H), 4.65-4.62 (m, 1H), 4.09 (q, *J* = 7.1 Hz, 2H), 3.07 (d, *J* = 15.8 Hz, 1H), 2.92 (d, *J* = 15.7 Hz, 1H), 2.72 (d, *J* = 14.2 Hz, 1H), 2.68 (d, *J* = 14.2 Hz, 1H), 2.58-2.50 (m, 1H), 2.44 (t, *J* = 4.4 Hz, 1H), 2.31 (d, *J* = 16.9 Hz, 1H), 2.04 (d, *J* = 16.9 Hz, 1H), 1.88-1.65 (m, 12H), 1.60-1.45 (m, 4H), 1.40-1.29 (m, 3H), 1.24 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.5, 172.2, 145.4, 145.0, 120.9, 113.1, 97.8, 82.3, 60.3, 56.2, 45.7, 41.8, 41.5, 39.5, 38.4, 35.7, 34.7, 31.5, 29.1, 25.6, 24.7, 22.1, 14.2; HRMS (ESI): calc. for $C_{25}H_{34}NaO_3$ $[M+Na]^+$: 405.2400; found: 405.2401.

(E)-Ethyl 2-(6-(2-oxo-6-phenylhex-5-en-3-yn-1-yl)-1-(prop-1-en-2-yl)spiro[3.4]oct-1 -en-2-yl)acetate (**3ak**)

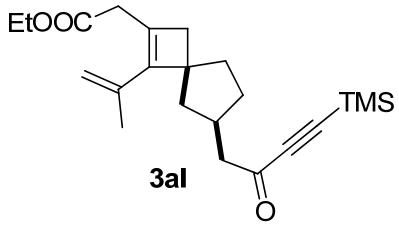


3ak

70% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39-7.29 (m, 4H), 7.27-7.21 (m, 1H), 6.67-6.59 (m, 1H), 6.13 (dt, *J* = 15.8, 5.9 Hz, 1H), 4.89-4.85 (m, 1H), 4.65-4.62 (m, 1H), 4.09 (q, *J* = 7.1 Hz, 2H), 3.31 (dd, *J* = 5.8, 1.8 Hz, 1H), 3.07 (d, *J* = 15.6 Hz, 1H), 2.92 (d, *J* = 15.8 Hz, 1H), 2.77 (d, *J* = 14.5 Hz, 1H), 2.73 (d, *J* = 14.2 Hz, 1H), 2.45 (t, *J* = 4.3 Hz, 1H), 2.32 (d, *J* = 16.9 Hz, 1H), 2.07 (d, *J* = 16.9 Hz, 1H), 1.87-1.69 (m, 8H), 1.59 (ddd, *J* = 10.5, 4.1, 1.1 Hz, 1H), 1.23 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.1, 172.2, 145.4, 144.9, 136.5, 132.7, 128.6, 127.7, 126.3, 121.6, 120.9, 113.2, 90.1, 83.9, 60.3, 56.0, 45.6, 41.8, 41.4, 39.5, 38.4, 35.8,

34.7, 22.5, 22.1, 14.2; HRMS (ESI): calc. for $C_{28}H_{32}NaO_3$ $[M+Na]^+$: 439.2244; found: 439.2238.

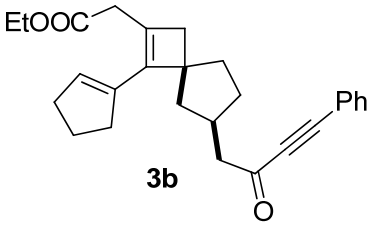
Ethyl 2-(6-(2-oxo-4-(trimethylsilyl)but-3-yn-1-yl)-1-(prop-1-en-2-yl)spiro[3.4]oct-1 -en-2-yl)acetate (**3al**)



3al

79% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.89-4.84 (m, 1H), 4.66-4.61 (m, 1H), 4.10 (q, *J* = 7.1 Hz, 2H), 3.06 (d, *J* = 15.7 Hz, 1H), 2.93 (d, *J* = 15.7 Hz, 1H), 2.76 (d, *J* = 14.5 Hz, 1H), 2.71 (d, *J* = 14.5 Hz, 1H), 2.44 (t, *J* = 4.5 Hz, 1H), 2.30 (d, *J* = 17.0 Hz, 1H), 2.05 (d, *J* = 17.0 Hz, 1H), 1.88-1.66 (m, 8H), 1.60-1.54 (m , 1H), 1.24 (t, *J* = 7.2 Hz, 3H), 0.24 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.9, 172.2, 145.4, 144.9, 120.8, 113.2, 103.5, 97.5, 60.3, 55.9, 45.6, 41.8, 41.4, 39.5, 38.4, 35.7, 34.7, 22.1, 14.2, -0.8; HRMS (ESI): calc. for $C_{22}H_{32}NaO_3Si$ $[M+Na]^+$: 395.2013; found: 395.2012.

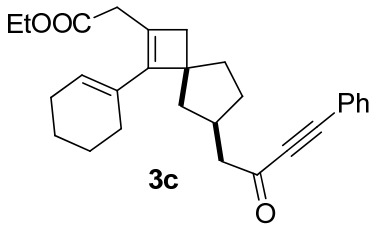
Ethyl 2-(1-(cyclopent-1-en-1-yl)-6-(2-oxo-4-phenylbut-3-yn-1-yl)spiro[3.4]oct-1-en -2-yl)acetate (**3b**)



3b

71% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59-7.54 (m, 2H), 7.48-7.42 (m, 1H), 7.41-7.35 (m, 2H), 5.50-5.42 (m, 1H), 4.09 (q, *J* = 7.1 Hz, 2H), 3.08 (d, *J* = 15.6 Hz, 1H), 2.97 (d, *J* = 15.6 Hz, 1H), 2.86 (d, *J* = 14.3 Hz, 1H), 2.81 (d, *J* = 14.3 Hz, 1H), 2.53-2.26 (m, 6H), 2.12 (d, *J* = 17.0 Hz, 1H), 1.92-1.73 (m, 7H), 1.63 (ddd, *J* = 10.6, 4.0, 1.0 Hz, 1H), 1.23 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.2, 172.3, 142.8, 140.6, 132.9, 130.7, 128.6, 127.7, 121.9, 120.1, 90.5, 89.3, 60.3, 56.2, 46.3, 41.9, 41.4, 39.6, 35.9, 34.9, 34.8, 32.7, 23.5, 14.2; HRMS (ESI): calc. for $C_{27}H_{30}NaO_3$ $[M+Na]^+$: 425.2087; found: 425.2078.

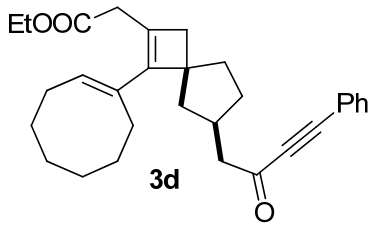
Ethyl 2-(1-(cyclohex-1-en-1-yl)-6-(2-oxo-4-phenylbut-3-yn-1-yl)spiro[3.4]oct-1-en-2-yl)acetate (**3c**)



3c

68% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59-7.54 (m, 2H), 7.48-7.42 (m, 1H), 7.41-7.35 (m, 2H), 5.38-5.32 (m, 1H), 4.11-4.05 (m, 2H), 3.04 (d, *J* = 15.7 Hz, 1H), 2.91 (d, *J* = 15.7 Hz, 1H), 2.85 (d, *J* = 14.2 Hz, 1H), 2.80 (d, *J* = 14.2 Hz, 1H), 2.46-2.40 (m, 1H), 2.36 (d, *J* = 16.9 Hz, 1H), 2.13-1.97 (m, 4H), 1.96-1.69 (m, 6H), 1.67-1.53 (m, 5H), 1.22 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.2, 172.2, 146.1, 138.0, 133.0, 130.6, 128.6, 124.0, 120.5, 120.1, 90.5, 89.3, 60.2, 56.3, 45.7, 42.0, 41.6, 39.7, 38.5, 35.9, 34.8, 27.7, 25.1, 22.9, 22.2, 14.2; HRMS (ESI): calc. for $C_{28}H_{32}NaO_3$ $[M+Na]^+$: 439.2244; found: 439.2248.

(E)-Ethyl 2-(1-(cyclooct-1-en-1-yl)-6-(2-oxo-4-phenylbut-3-yn-1-yl)spiro[3.4]oct-1-en-2-yl)acetate (**3d**)

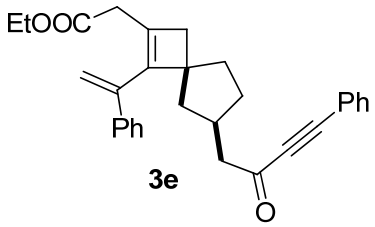


3d

80% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59-7.55 (m, 2H), 7.48-7.43 (m, 1H), 7.41-7.35 (m, 2H), 5.34 (t, *J* = 8.0 Hz, 1H), 4.12-4.05 (m, 2H), 3.15 (d, *J* = 15.8 Hz, 1H), 2.96 (d, *J* = 15.8 Hz, 1H), 2.85 (d, *J* = 14.2 Hz, 1H), 2.81 (d, *J* = 14.2 Hz, 1H), 2.49-2.43 (m, 1H), 2.38 (d, *J* = 16.8 Hz, 1H), 2.27-2.06 (m, 5H), 1.89-1.69 (m, 5H), 1.63 (ddd, *J* = 10.6, 4.1, 1.2 Hz, 1H), 1.59-1.45 (m, 8H), 1.22 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.2, 172.5, 146.5, 140.6, 133.0, 130.6, 128.6, 127.7, 120.6, 120.1, 90.5, 89.3, 60.2, 56.2, 45.8, 42.0, 41.5, 39.8, 38.7, 35.9, 34.6, 29.6, 29.0, 28.4, 26.6, 26.5, 26.4, 14.2; HRMS (ESI): calc. for $C_{30}H_{36}NaO_3$ $[M+Na]^+$: 467.2557; found: 467.2544.

Ethyl 2-(6-(2-oxo-4-phenylbut-3-yn-1-yl)-1-(1-phenylvinyl)spiro[3.4]oct-1-en-2-yl)

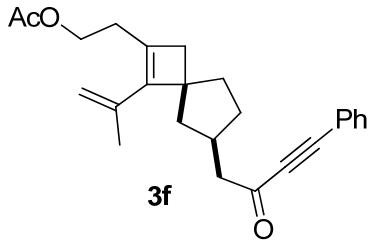
acetate (**3e**)



3e

62% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60-7.51 (m, 2H), 7.49-7.43 (m, 1H), 7.42-7.36 (m, 4H), 7.35-7.26 (m, 3H), 5.53 (d, *J* = 1.8 Hz, 1H), 5.04 (d, *J* = 1.8 Hz, 1H), 4.08 (q, *J* = 7.1 Hz, 2H), 3.13 (d, *J* = 15.9 Hz, 1H), 2.99 (d, *J* = 15.9 Hz, 1H), 2.89 (d, *J* = 14.4 Hz, 1H), 2.87 (d, *J* = 14.4 Hz, 1H), 2.51 (d, *J* = 17.1 Hz, 1H), 2.31 (d, *J* = 4.5 Hz, 1H), 2.25 (d, *J* = 17.1 Hz, 1H), 1.94-1.71 (m, 5H), 1.62 (ddd, *J* = 10.6, 3.9, 1.1 Hz, 1H), 1.22 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.1, 172.1, 147.6, 143.6, 139.1, 132.9, 130.7, 128.6, 128.4, 127.7, 126.7, 124.6, 120.1, 113.7, 90.6, 89.3, 60.4, 56.2, 45.7, 42.0, 41.6, 40.4, 38.5, 36.0, 34.3, 14.2; HRMS (ESI): calc. for $C_{30}H_{30}NaO_3$ $[M+Na]^+$: 461.2087; found: 461.2065.

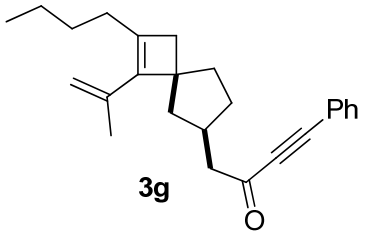
2-(6-(2-Oxo-4-phenylbut-3-yn-1-yl)-1-(prop-1-en-2-yl)spiro[3.4]oct-1-en-2-yl)ethyl acetate (**3f**)



3f

68% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60-7.55 (m, 2H), 7.49-7.43 (m, 1H), 7.42-7.36 (m, 2H), 4.88-4.82 (m, 1H), 4.62-4.56 (m, 1H), 4.03 (t, *J* = 7.3 Hz, 2H), 2.86 (d, *J* = 14.3 Hz, 1H), 2.82 (d, *J* = 14.3 Hz, 1H), 2.44-2.22 (m, 4H), 2.07 (d, *J* = 17.0 Hz, 1H), 2.01 (s, 3H), 1.85-1.74 (m, 6H), 1.74-1.66 (m, 2H), 1.61 (ddd, *J* = 10.7, 4.0, 1.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.1, 171.0, 145.4, 144.8, 133.0, 130.7, 128.6, 123.1, 120.1, 112.5, 90.5, 89.3, 63.5, 56.3, 45.2, 41.9, 39.9, 36.0, 34.8, 31.9, 22.5, 21.0; HRMS (ESI): calc. for $C_{25}H_{28}NaO_3$ $[M+Na]^+$: 399.1931; found: 399.1931.

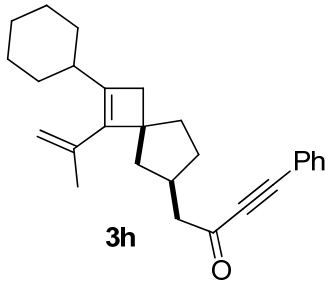
1-(2-Butyl-1-(prop-1-en-2-yl)spiro[3.4]oct-1-en-6-yl)-4-phenylbut-3-yn-2-one (**3g**)



3g

55% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61-7.55 (m, 2H), 7.49-7.43 (m, 1H), 7.42-7.35 (m, 2H), 4.86-4.78 (m, 1H), 4.59-4.53 (m, 1H), 2.85 (d, *J* = 14.0 Hz, 1H), 2.81 (d, *J* = 14.0 Hz, 1H), 2.42-2.36 (m, 1H), 2.32 (d, *J* = 17.0 Hz, 1H), 2.03 (d, *J* = 17.2 Hz, 1H), 2.00-1.89 (m, 2H), 1.86-1.65 (m, 8H), 1.60 (ddd, *J* = 10.5, 4.0, 1.2 Hz, 1H), 1.32-1.19 (m, 5H), 0.85 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.4, 146.0, 141.4, ,132.9, 130.7, 128.6, 127.7, 120.2, 90.5, 89.4, 56.7, 44.9, 42.0, 39.9, 36.0, 34.9, 32.5, 31.1, 22.69, 22.67, 14.1; HRMS (ESI): calc. for $C_{25}H_{30}NaO$ $[M+Na]^+$: 369.2189; found: 369.2182.

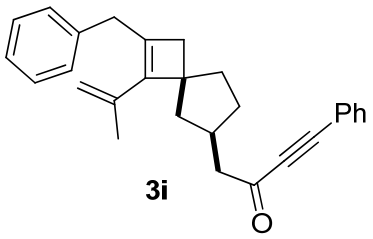
1-(2-Cyclohexyl-1-(prop-1-en-2-yl)spiro[3.4]oct-1-en-6-yl)-4-phenylbut-3-yn-2-one (**3h**)



3h

60% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61-7.55 (m, 2H), 7.49-7.43 (m, 1H), 7.42-7.35 (m, 2H), 4.83-4.76 (m, 1H), 4.57-4.51 (m, 1H), 2.85 (d, *J* = 13.9 Hz, 1H), 2.85 (d, *J* = 13.9 Hz, 1H), 2.40-2.22 (m, 3H), 2.04 (d, *J* = 16.8 Hz, 1H), 1.86-1.53 (m, 12H), 1.42-1.33 (m, 2H), 1.32-1.14 (m, 4H), 1.12-1.00 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.5, 146.4, 140.3, 132.9, 132.1, 130.6, 128.6, 120.2, 111.4, 90.5, 89.4, 56.8, 41.8, 41.7, 40.8, 40.7, 40.1, 35.6, 35.0, 32.3, 31.2, 26.6, 26.5, 26.2, 23.1; HRMS (ESI): calc. for $C_{27}H_{32}NaO$ $[M+Na]^+$: 38.2345; found: 395.2335.

1-(2-Benzyl-1-(prop-1-en-2-yl)spiro[3.4]oct-1-en-6-yl)-4-phenylbut-3-yn-2-one (**3i**)



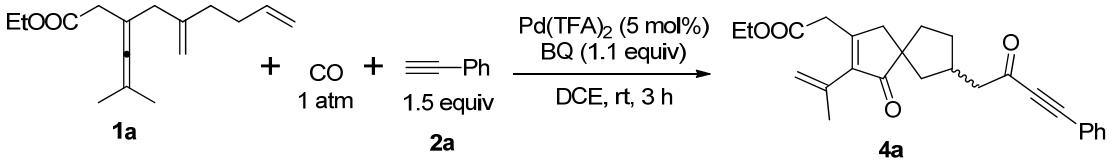
3i

63% isolated yield, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52-7.48 (m, 2H), 7.48-7.42 (m, 1H), 7.40-7.34 (m, 2H), 7.25-7.20 (m, 2H), 7.16-7.07 (m, 3H), 4.89-4.85 (m, 1H), 4.73-4.68 (m, 1H), 3.39 (d, *J* = 14.9 Hz, 1H), 3.34 (d, *J* = 14.9 Hz, 1H), 2.79 (d, *J* = 14.2 Hz, 1H), 2.76 (d, *J* = 14.2 Hz, 1H), 2.51 (t, *J* = 4.5 Hz, 1H), 2.24 (dd, *J* = 17.0, 1.3 Hz, 1H), 1.98-1.61 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.2, 145.5, 143.7, 140.9, 132.9, 130.6, 128.6, 128.4, 128.2, 125.8, 125.6, 120.1, 112.5, 90.5, 89.3, 56.2, 44.9, 42.0, 41.8, 40.0, 38.6, 35.8, 34.9, 22.8; HRMS (ESI): calc. for $C_{28}H_{28}NaO$ $[M+Na]^+$: 403.2032; found: 403.2036.

General procedure for palladium-catalyzed oxidative carbocyclization of enallenes for the formation of compounds 4

Representative procedure B for the synthesis of 4a-4i.

Ethyl 2-(4-oxo-7-(2-oxo-4-phenylbut-3-yn-1-yl)-3-(prop-1-en-2-yl)spiro[4.4]non-2-en-2-yl)acetate (**4a**)

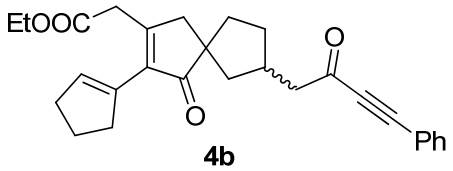


1a **2a** **4a**

To a solution of $Pd(TFA)_2$ (3.3 mg, 0.01 mmol), BQ (23.8 mg, 0.22 mmol) and **2a** (30.6 mg, 0.3 mmol) in DCE (2 mL) were added enallene **1a** (49.6 mg, 0.2 mmol). The tube was closed with a septum. The tube was evacuated and filled with carbon monoxide gas (repeated three times) using a balloon. The reaction was stirred at room temperature for 3 h. After full consumption of starting material **1a** as monitored by TLC, the reaction mixture was evaporated and purified via column chromatography on silica gel (eluent: petroleum ether/ethyl ether = 5/1) afforded **4a** (72.7 mg, 90%, d/r = 91/9 determined by ^{1}H NMR analysis): colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61-7.55 (m, 2H, ArH), 7.48-7.42 (m, 1H, ArH), 7.41-7.35 (m, 2H, ArH), 5.23-5.16 (m, 1H, one proton of $=CH_2$), 4.87-4.81 (m, 1H, one proton of $=CH_2$), 4.21-4.13 (m, 2H, OCH_2), 3.50 (d, *J* = 15.5 Hz, 1H, one proton of CH_2), 3.46 (d, *J* = 15.5 Hz, 1H, one proton of CH_2), 2.93-2.80 (m, 1H, one proton of CH_2), 2.76-2.57 (m, 4H, 2 × CH_2), 2.34-2.26 (m, 0.91H, one proton of CH_2), 2.22-2.12 (m, 0.91H, one proton of CH_2), 2.20-1.88 (m, 4H, CH_3 + one proton of CH_2), 1.66-1.54 (m, 1H, CH), 1.48-1.36 (m, 1H, one proton of CH_2), 1.34-1.23 (m, 4H, CH_3 + one proton of CH_2); ^{13}C NMR (100 M, Hz, $CDCl_3$) the following signal is discernible for major isomer: δ 211.1, 187.2, 169.2, 161.4, 143.4, 137.2, 133.1, 130.7, 128.6, 119.9, 117.1, 91.1, 87.8, 61.2, 54.2, 51.8, 48.3, 43.2, 38.4, 37.1, 35.9, 33.1, 21.9, 14.1; HRMS (ESI): calc. for $C_{26}H_{28}NaO_4$ $[M+Na]^+$: 427.1880, found: 427.1873.

The general method from above was used for preparation of the following compounds **4**:

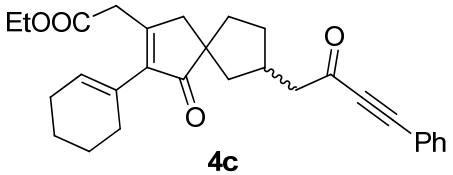
Ethyl 2-(3-(cyclopent-1-en-1-yl)-4-oxo-7-(2-oxo-4-phenylbut-3-yn-1-yl)spiro[4.4]non -2-en-2-yl)acetate (**4b**)



4b

90% isolated yield, d/r = 95/5, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61-7.55 (m, 2H), 7.48-7.42 (m, 1H), 7.41-7.35 (m, 2H), 6.14-6.06 (m, 1H), 4.22-4.13 (m, 2H), 3.54 (d, *J* = 15.5 Hz, 1H), 3.51 (d, *J* = 15.5 Hz, 1H), 2.92-2.80 (m, 1H), 2.77-2.53 (m, 6H), 2.48-2.39 (m, 2H), 2.31 (dd, *J* = 13.2, 8.7 Hz, 0.95H), 2.21-2.12 (m, 0.95H), 1.99-1.85 (m, 3H), 1.64-1.53 (m, 1H), 1.47-1.35 (m, 1H), 1.33-1.24 (m, 4H); ^{13}C NMR (100 M, Hz, $CDCl_3$) the following signal is discernible for major isomer: δ 211.6, 187.2, 169.3, 160.6, 137.4, 134.4, 133.1, 133.0, 130.7, 128.6, 119.9, 91.1, 87.9, 61.2, 54.1, 51.8, 48.7, 43.3, 38.6, 37.7, 35.9, 34.6, 33.1, 33.0, 23.4, 14.2; HRMS (ESI): calc. for $C_{28}H_{30}NaO_4$ $[M+Na]^+$: 453.2036; found: 453.2029.

Ethyl 2-(3-(cyclohex-1-en-1-yl)-4-oxo-7-(2-oxo-4-phenylbut-3-yn-1-yl)spiro[4.4]non -2-en-2-yl)acetate (**4c**)

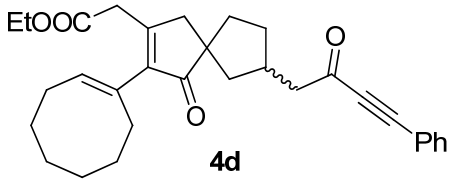


4c

93% isolated yield, d/r = 91/9, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61-7.54 (m, 2H), 7.48-7.41 (m, 1H), 7.41-7.35 (m, 2H), 5.60-5.53 (m, 1H), 4.21-4.13 (m, 2H), 3.46 (d, *J* = 15.5 Hz, 1H), 3.44 (d, *J* = 15.5 Hz, 1H), 2.93-2.79 (m, 1H), 2.74-2.56 (m, 4H), 2.29 (dd, *J* = 13.2, 8.7 Hz, 0.95H), 2.20-2.07 (m, 5H), 1.97-1.88 (m, 0.91H), 1.71-1.52 (m, 5H), 1.47-1.34 (m, 1H), 1.33-1.24 (m, 4H); ^{13}C NMR (100 M, Hz, $CDCl_3$) the following signal is discernible for major isomer: δ 211.7, 187.3, 169.4, 160.8, 143.8, 133.1, 130.7, 128.7, 128.6, 119.9, 91.1, 87.8, 61.1, 54.1, 51.8, 48.1, 43.2, 38.4, 37.2, 35.8, 33.1, 27.3, 25.3, 22.4, 21.9, 14.1; HRMS (ESI): calc. for $C_{29}H_{32}NaO_4$ $[M+Na]^+$: 467.2193; found: 467.2187.

(E)-Ethyl 2-(3-(cyclooct-1-en-1-yl)-4-oxo-7-(2-oxo-4-phenylbut-3-yn-1-yl)spiro[4.4]

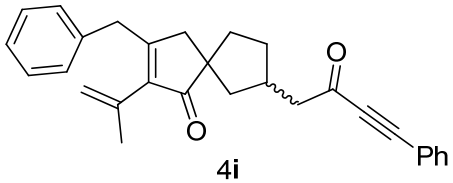
non-2-en-2-yl)acetate (**4d**)



4d

89% isolated yield, d/r = 93/7, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60-7.55 (m, 2H), 7.48-7.42 (m, 1H), 7.41-7.35 (m, 2H), 5.57-5.44 (m, 1H), 4.24-4.10 (m, 2H), 3.51 (d, *J* = 15.3 Hz, 1H), 3.48 (d, *J* = 15.3 Hz, 1H), 2.92-2.79 (m, 1H), 2.77-2.57 (m, 4H), 2.38-2.11 (m, 6H), 1.99-1.87 (m, 0.93H), 1.65-1.37 (m, 10H), 1.33-1.23 (m, 4H); ^{13}C NMR (100 M, Hz, $CDCl_3$) the following signal is discernible for major isomer: δ 211.9, 187.3, 169.4, 161.2, 144.3, 133.5, 133.1, 131.9, 130.7, 128.6, 119.9, 91.1, 87.8, 51.2, 54.0, 51.8, 48.1, 43.2, 38.4, 37.2, 35.9, 33.1, 29.5, 28.5, 28.1, 26.5, 26.4, 26.3, 14.1; HRMS (ESI): calc. for $C_{31}H_{36}NaO_4$ $[M+Na]^+$: 495.2506; found: 495.2516.

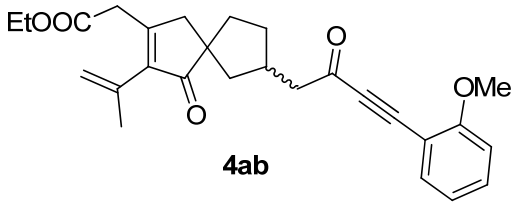
3-Benzyl-7-(2-oxo-4-phenylbut-3-yn-1-yl)-2-(prop-1-en-2-yl)spiro[4.4]non-2-en-1-on e (**4i**)



4i

88% isolated yield, d/r = 88/12, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) 7.60-7.53 (m, 2H), 7.48-7.42 (m, 1H), 7.41-7.35 (m, 2H), 7.35-7.29 (m, 2H), 7.28-7.22 (m, 1H), 7.16-7.10 (m, 2H), 5.28-5.23 (m, 1H), 4.94-4.90 (m, 1H), 3.82 (d, *J* = 14.8 Hz, 1H), 3.78 (d, *J* = 14.8 Hz, 1H), 2.90-2.77 (m, 1H), 2.71-2.59 (m, 2H), 2.50-2.36 (m, 2H), 2.28 (dd, *J* = 13.2, 8.7 Hz, 1H), 2.18-2.07 (m, 0.88H), 2.04-1.86 (m, 4H), 1.54-1.46 (m, 1H, CH), 1.39-1.16 (m, 2H); ^{13}C NMR (100 M, Hz, $CDCl_3$) the following signal is discernible for major isomer: δ 211.6, 187.3, 168.5, 137.5, 133.1, 130.7, 128.8, 128.7, 128.6, 126.7, 119.9, 116.8, 91.1, 87.8, 53.9, 51.7, 47.5, 43.2, 38.4, 37.4, 35.9, 33.1, 22.4; HRMS (ESI): calc. for $C_{29}H_{28}NaO_2$ $[M+Na]^+$: 431.1982; found: 431.1977.

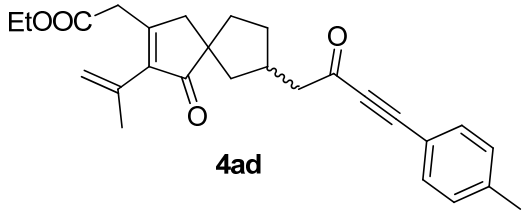
Ethyl 2-(7-(4-(2-methoxyphenyl)-2-oxobut-3-yn-1-yl)-4-oxo-3-(prop-1-en-2-yl) spiro[4.4]non-2-en-2-yl)acetate (**4ab**)



4ab

84% isolated yield, d/r = 90/10, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53-7.47 (m, 1H), 7.45-7.36 (m, 1H), 6.98-6.86 (m, 2H), 5.23-5.15 (m, 1H), 4.88-4.80 (m, 1H), 4.21-4.12 (m, 2H), [3.91 (s), 3.89 (s), 3H], 3.49 (d, *J* = 15.5 Hz, 1H), 3.46 (d, *J* = 15.5 Hz, 1H), 2.97-2.80 (m, 1H), 2.74-2.58 (m, 4H), 2.32 (dd, *J* = 13.2, 8.9 Hz, 0.9H), 2.23-2.13 (m, 0.91H), 1.98-1.88 (m, 4H), 1.64-1.56 (m, 1H), 1.46-1.35 (m, 1H), 1.35-1.23 (m, 4H); ^{13}C NMR (100 M, Hz, $CDCl_3$) the following signal is discernible for major isomer: δ 211.1, 187.4, 169.2, 161.4, 143.4, 137.2, 135.0, 132.5, 120.5, 117.1, 110.8, 109.1, 91.9, 88.7, 61.2, 55.8, 54.2, 51.9, 48.3, 43.2, 38.6, 37.1, 35.9, 33.1, 21.9, 14.1; HRMS (ESI): calc. for $C_{27}H_{30}NaO_5$ $[M+Na]^+$: 457.1985; found: 457.1981.

Ethyl 2-(4-oxo-7-(2-oxo-4-(p-tolyl)but-3-yn-1-yl)-3-(prop-1-en-2-yl)spiro[4.4]non -2-en-2-yl)acetate (**4ad**)

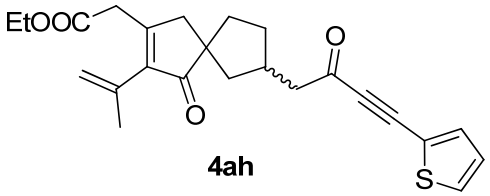


4ad

87% isolated yield, d/r = 92/8, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50-7.43 (m, 2H), 7.22-7.15 (m, 2H), 5.22-5.17 (m, 1H), 4.86-4.81 (m, 1H), 4.21-4.13 (m, 2H), 3.49 (d, *J* = 15.6 Hz, 1H), 3.47 (d, *J* = 15.6 Hz, 1H), 2.90-2.79 (m, 1H), 2.76-2.59 (m, 4H), 2.38 (s, 3H), 2.31 (dd, *J* = 13.2, 8.7 Hz, 0.92H), 2.21-2.12 (m, 0.92H), 1.99-1.88 (m, 4H), 1.65-1.53 (m, 1H), 1.46-1.35 (m, 1H), 1.34-1.22 (m, 4H); ^{13}C NMR (100 M, Hz, $CDCl_3$) the following signal is discernible for major isomer: δ 211.1, 187.3, 169.2, 161.4, 143.4, 141.5, 137.2, 133.1, 129.4, 117.1, 116.8, 91.8, 87.7, 61.2, 54.2, 51.8, 48.3, 43.2, 38.5, 37.1, 35.9, 33.1, 21.9, 21.7, 14.1; HRMS (ESI): calc. for $C_{27}H_{30}NaO_4$ $[M+Na]^+$: 441.2036; found: 441.2030.

Ethyl 2-(4-oxo-7-(2-oxo-4-(thiophen-2-yl)but-3-yn-1-yl)-3-(prop-1-en-2-yl)spiro[4.4]

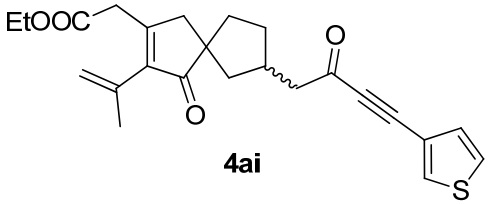
non-2-en-2-yl)acetate (**4ah**)



4ah

72% isolated yield, d/r = 91/9, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56-7.45 (m, 2H), 7.12-7.03 (m, 1H), 5.25-5.14 (m, 1H), 4.87-4.82 (m, 1H), 4.22-4.13 (m, 2H), 3.50 (d, *J* = 15.5 Hz, 1H), 3.47 (d, *J* = 15.5 Hz, 1H), 2.88-2.78 (m, 1H), 2.76-2.60 (m, 4H), 2.31 (dd, *J* = 13.3, 8.7 Hz, 1H), 2.21-2.12 (m, 0.91H), 1.99-1.89 (m, 4H), 1.66-1.58 (m, 1H), 1.46-1.37 (m, 1H), 1.34-1.24 (m, 4H); ^{13}C NMR (100 M, Hz, $CDCl_3$) the following signal is discernible for major isomer: δ 211.1, 186.7, 169.2, 161.4, 143.4, 137.2, 136.8, 131.7, 127.7, 119.7, 117.1, 92.5, 85.0, 61.3, 54.2, 51.5, 48.3, 43.2, 38.5, 37.1, 35.8, 33.1, 21.9, 14.1; HRMS (ESI): calc. for $C_{24}H_{26}NaO_4S$ $[M+Na]^+$: 433.1444; found: 433.1442.

Ethyl 2-(4-oxo-7-(2-oxo-4-(thiophen-3-yl)but-3-yn-1-yl)-3-(prop-1-en-2-yl)spiro[4.4] non-2-en-2-yl)acetate (**4ai**)



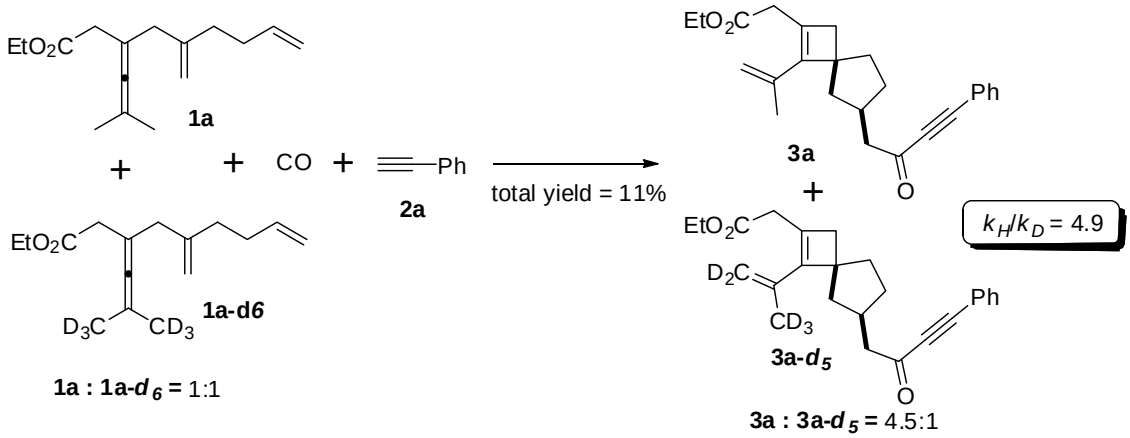
4ai

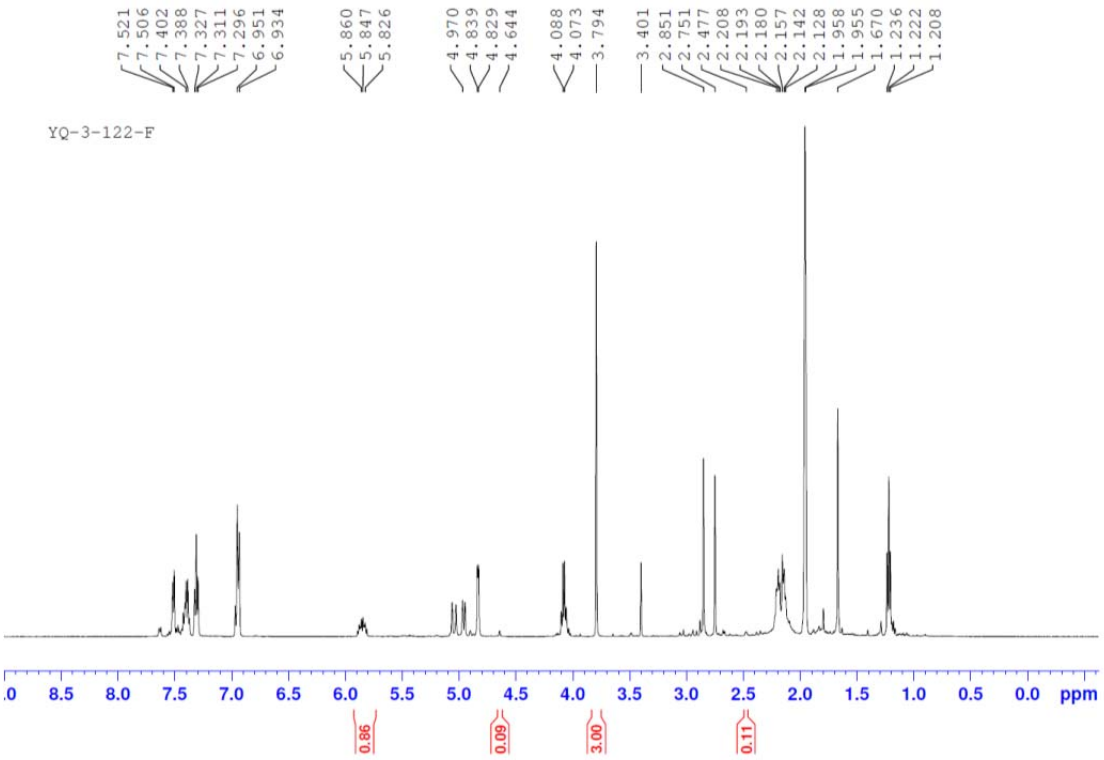
78% isolated yield, d/r = 88/12, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ [7.78 (dd, *J* = 2.9, 1.2 Hz), 7.75 (dd, *J* = 3.0, 1.2 Hz), 1H], 7.35-7.31 (m, 1H), 7.25-7.21 (m, 1H), 5.22-5.19 (m, 1H), 4.86-4.82 (m, 1H), 4.21-4.14 (m, 2H), 3.49 (d, *J* = 15.5 Hz, 1H), 3.47 (d, *J* = 15.5 Hz, 1H), 2.90-2.79 (m, 1H), 2.75-2.59 (m, 4H), 2.30 (dd, *J* = 13.2, 8.6 Hz, 1H), 2.20-2.12 (m, 1H), 2.00-1.89 (m, 4H), 1.66-1.51 (m, 1H), 1.46-1.36 (m, 1H), 1.33-1.24 (m, 4H); ^{13}C NMR (100 M, Hz, $CDCl_3$) the following signal is discernible for major isomer: δ 211.1, 187.2, 169.2, 161.4, 143.4, 137.2, 134.0, 130.3, 126.2, 119.2, 117.1, 88.1, 86.6, 61.3, 54.1, 51.6, 48.3, 43.2, 38.4, 37.1, 35.9, 33.1, 21.8, 14.1; HRMS (ESI): calc. for $C_{24}H_{26}NaO_4S$ $[M+Na]^+$: 433.1444; found: 433.1437.

Kinetic Isotope Effect (KIE) Experiments

1. Determination of Intermolecular Competition KIE

To a solution of $Pd(TFA)_2$ (3.3 mg, 0.01 mmol) and F_4-BQ (39.6 mg, 0.22 mmol) in CD_3CN (3.0 mL) were added enallene **1a** (24.8 mg, 0.1 mmol), enallene **1a-*d*$_6$** (25.4 mg, 0.1 mmol), phenylacetylene (30.6 mg, 0.3 mmol), and CD_3CN (3.0 mL) sequentially. The tube was closed with a septum. The tube was evacuated and filled with carbon monoxide gas using a balloon for three times. The reaction was stirred at 80 °C for 90 seconds, then quickly evaporated. The yields of **3a** and **3a-*d*$_5$** were determined by ^{1}H NMR measurement using anisole as the internal standard (22 μL, 0.2 mmol).





As shown in the attached spectra, the combined yield of **3a** and **3a-*d*$_5$** was 11%, and the yield of **3a** was 9%, thus the yield of **3a-*d*$_5$** was 2%. Therefore, the ratio of **3a** and **3a-*d*$_5$** was determined as 4.5:1. Furthermore, the combined recovery of **1a** and **1a-*d*$_6$** was 86% [Integration of (H^a+H^b)], so the reaction conversion was 11.3%. Finally, the isotope effect value calculated from the product ratio and conversion of the reaction is 4.9 according to Sih's equation.[4]

2. Intermolecular KIE Experiments (Separate experiments)

To a solution of $Pd(TFA)_2$ (3.3 mg, 0.01 mmol) and F_4-BQ (39.6 mg, 0.22 mmol) in CD_3CN (3.0 mL) were added enallene **1a** (49.6 mg, 0.2 mmol), enallene **1a-*d*$_6$** (50.8 mg, 0.2 mmol), phenylacetylene (30.6 mg, 0.3 mmol), and CD_3CN (3.0 mL) sequentially. The tube was closed with a septum. The tube was evacuated and filled with carbon monoxide gas using a balloon for three times. The reaction was stirred at room temperature and recorded at different time (see **Table S2** and **S3** respectively). The yields were determined by ^{1}H NMR measurement using anisole as the internal standard.

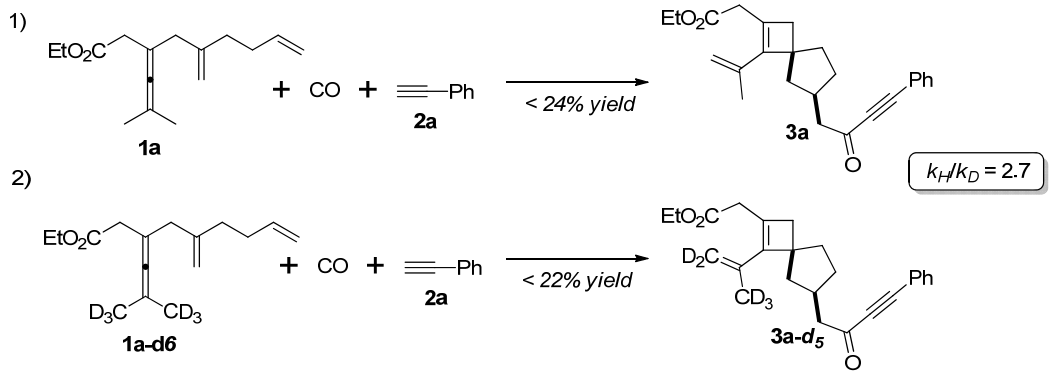


Table S2. For **1a**:

Time/s	20	35	60	90	120
Yield of **3a**/%	4	8	14	19	24

Due to the nature of the experiment, plots to determine the KIE were taken for **1a** (Figures S1).

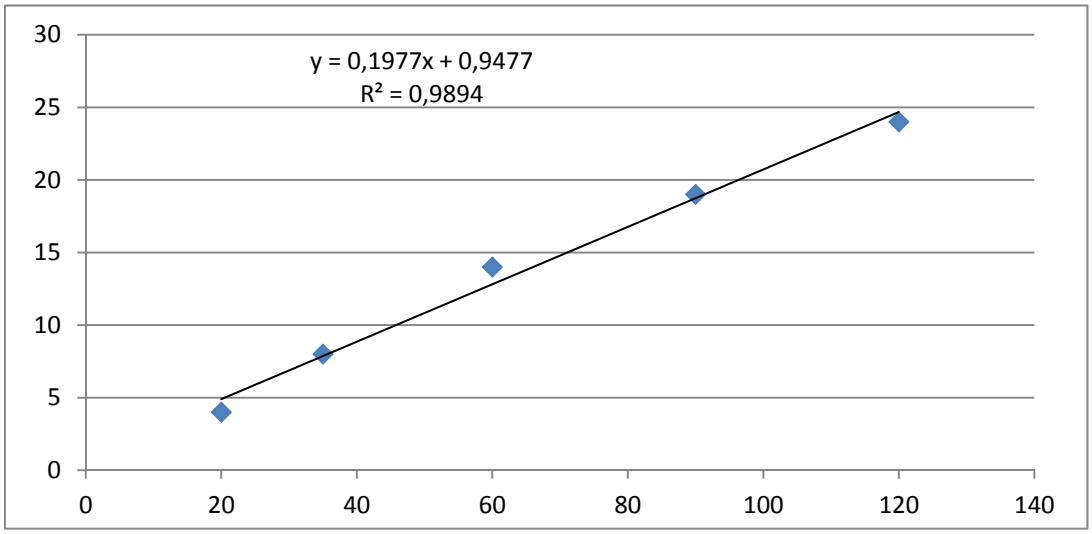


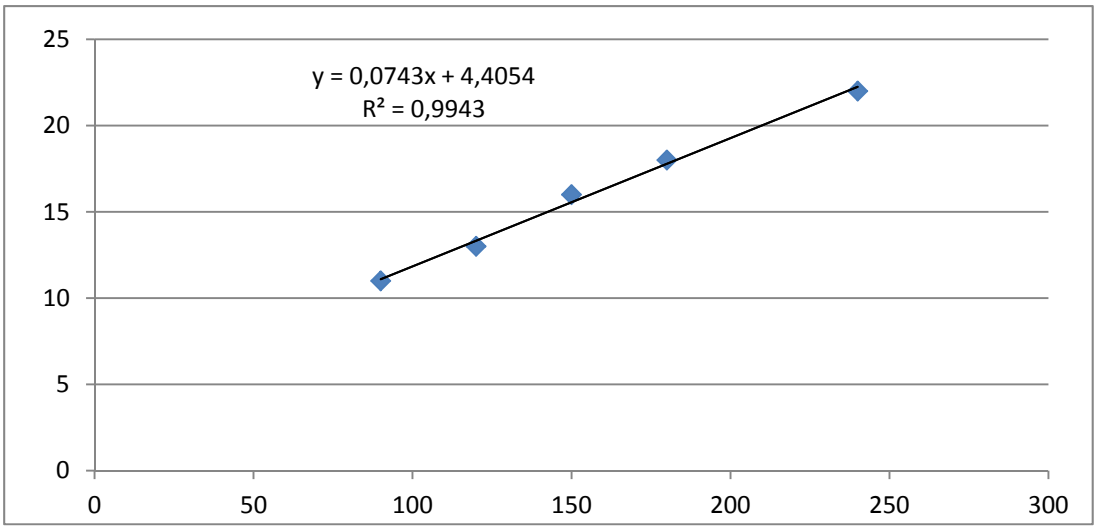
Figure S1. Linear function fit for reaction rate of **1a**.

Table S3. For **1a-*d*$_6$**:

Time/s	90	120	150	180	240
Yield of **3a-*d*$_5$**/%	11	13	16	18	22

Due to the nature of the experiment, plots to determine the KIE were taken for **1a-*d*$_6$** (Figures S2).

Figure S2. Linear function fit for reaction rate of **1a-*d*$_6$**.



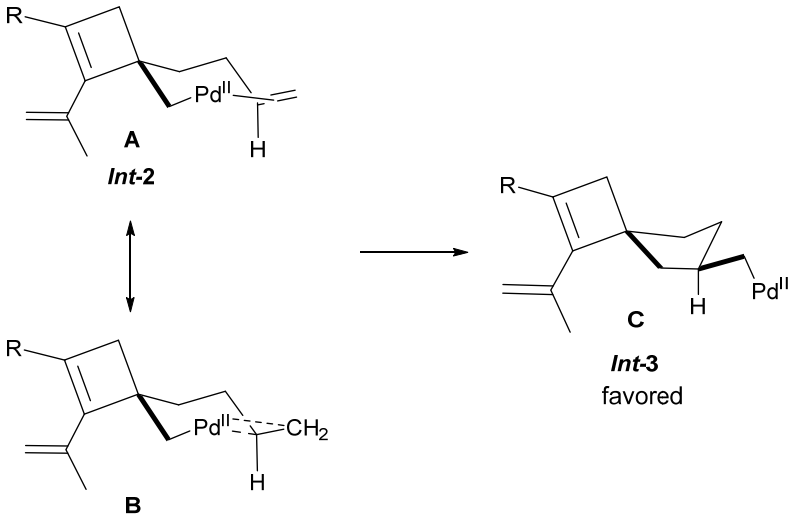
Finally, the intermolecular isotope effect value is determined as 2.7.

$k_H/k_D = (0.1977)/(0.0743) = 2.7$

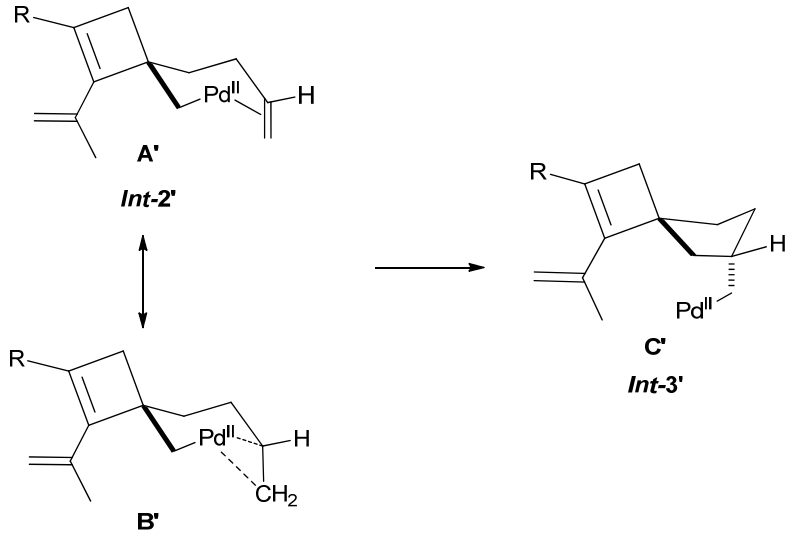
The spiro[3,4]octene derivatives 3 as single isomers

Re-face coordination is favored over si-face coordination.

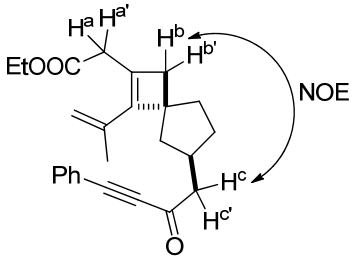
Re-face coordination:



Si-face coordination:



Determination of relative seterochemistry of 3a



3a

The assignment of ^{1}H NMR signals for **3a** was as follows: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60-7.54 (m, 2H, ArH), 7.48-7.42 (m, 1H, ArH), 7.41-7.35 (m, 2H, ArH), 4.91-4.84 (m, 1H, one proton of $=CH_2$), 4.67-4.62 (m, 1H, one proton of $=CH_2$), 4.08 (q, J = 7.1 Hz, 2H, OCH_2), 3.08 (d, J = 15.7 Hz, 1H, H^a), 2.94 (d, J = 15.7 Hz, 1H, $H^{a'}$), 2.86 (d, J = 14.2 Hz, 1H, H^b), 2.82 (d, J = 14.2 Hz, 1H, $H^{b'}$), 2.47 (t, J = 4.0 Hz, 1H, one proton of CH_2), 2.37 (br. d, J = 16.8 Hz, 1H, H^c), 2.11 (br. d, J = 16.8 Hz, 1H, $H^{c'}$), 1.89-1.71 (m, 8H, CH_3 + CH_2 + one proton of CH_2), 1.64 (ddd, J = 10.7, 4.1, 1.0 Hz, 1H, CH), 1.22 (t, J = 7.1 Hz, 3H, CH_3).

We were able to assign H^a,$H^{a'}$ in **3a** by comparing the ^{1}H NMR spectra of products **3a** and **3g**. A clear NOE was observed between H^b($H^{b'}$) and H^c in **3a** (see p. S57). Thus, the relative sterochemistry of $CH^bH^{b'}$ and $CH^cH^{c'}$ in **3a** could be determined to be cis to one another as shown in the above structure.

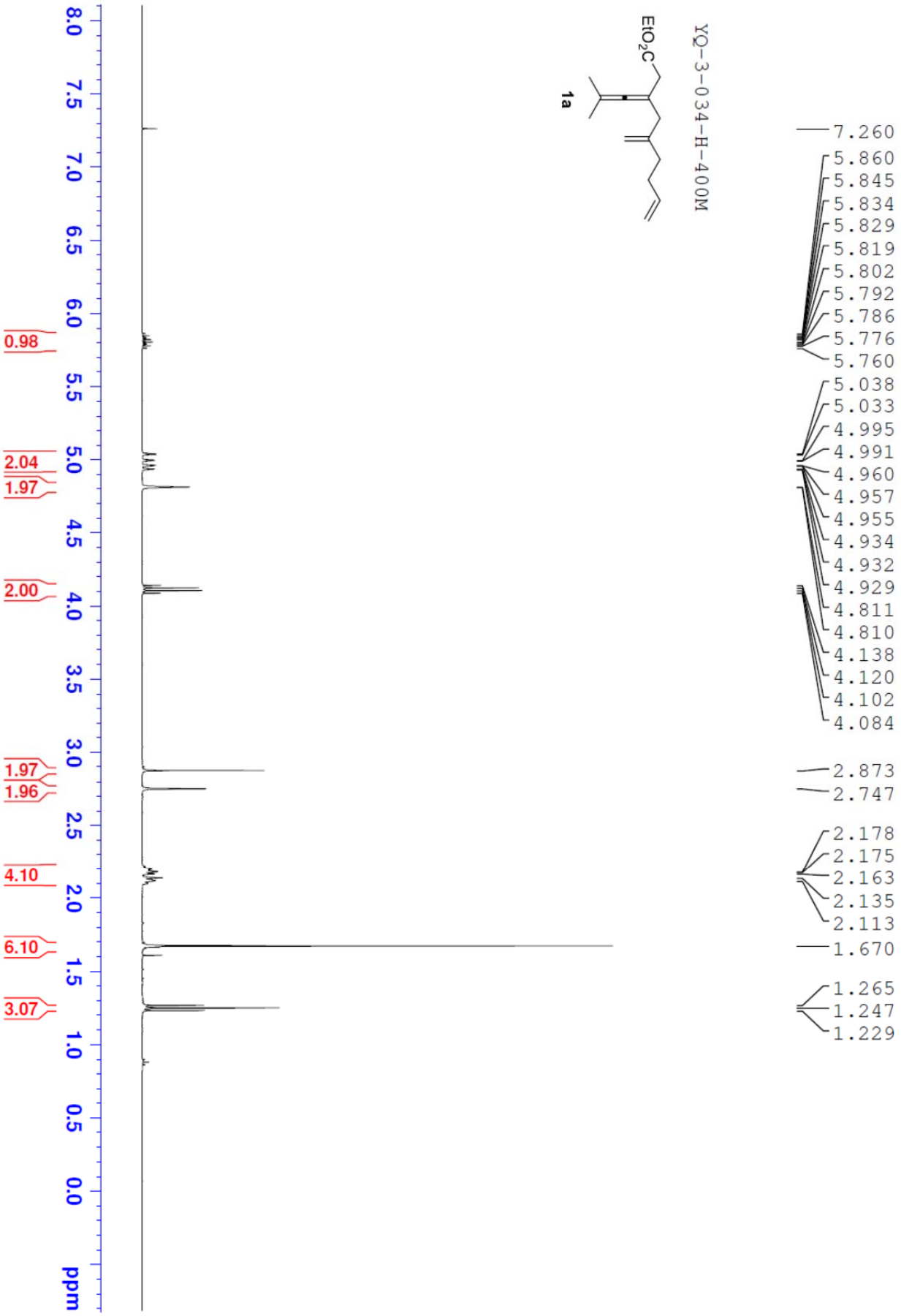
References:

(1) (a) Zhu, C.; Yang, B.; Jiang, T.; Bäckvall, J.-E. *Angew. Chem. Int. Ed*. **2015**, *54*, 9066. (b) Zhu, C.; Yang, B.; Bäckvall, J.-E. *J. Am. Chem. Soc*. **2015**, *137*, 11868. (c) Qiu, Y.; Yang, B.; Zhu, C.; Bäckvall, J.-E. *Angew. Chem. Int. Ed*. **2016**, *55*, 6520.

(2) Sherwood, T. C.; Trotta, A. H.; Snyder, S. A. *J. Am. Chem. Soc*. **2014**, *136*, 9743.

(3) Kessler, S. N.; Bäckvall, J.-E. *Angew. Chem. Int. Ed*. **2016**, *55*, 3734.

(4) Chen, C.-S.; Fujimoto, Y.; Girdaukas, G.; Sih, C. J. *J. Am. Chem. Soc*. **1982**, *104*, 7294.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5.786
5.776
5.760
5.038
5.033
4.995
4.991
4.960
4.957
4.955
4.934
4.932
4.929
4.811
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4.084
2.873
2.747
2.178
2.175
2.163
2.135
2.113
1.670
1.265
1.247
1.229
0.98
2.04
1.97
2.00
1.97
1.96
4.10
6.10
3.07
8.0
7.5
7.0
6.5
6.0
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1.5
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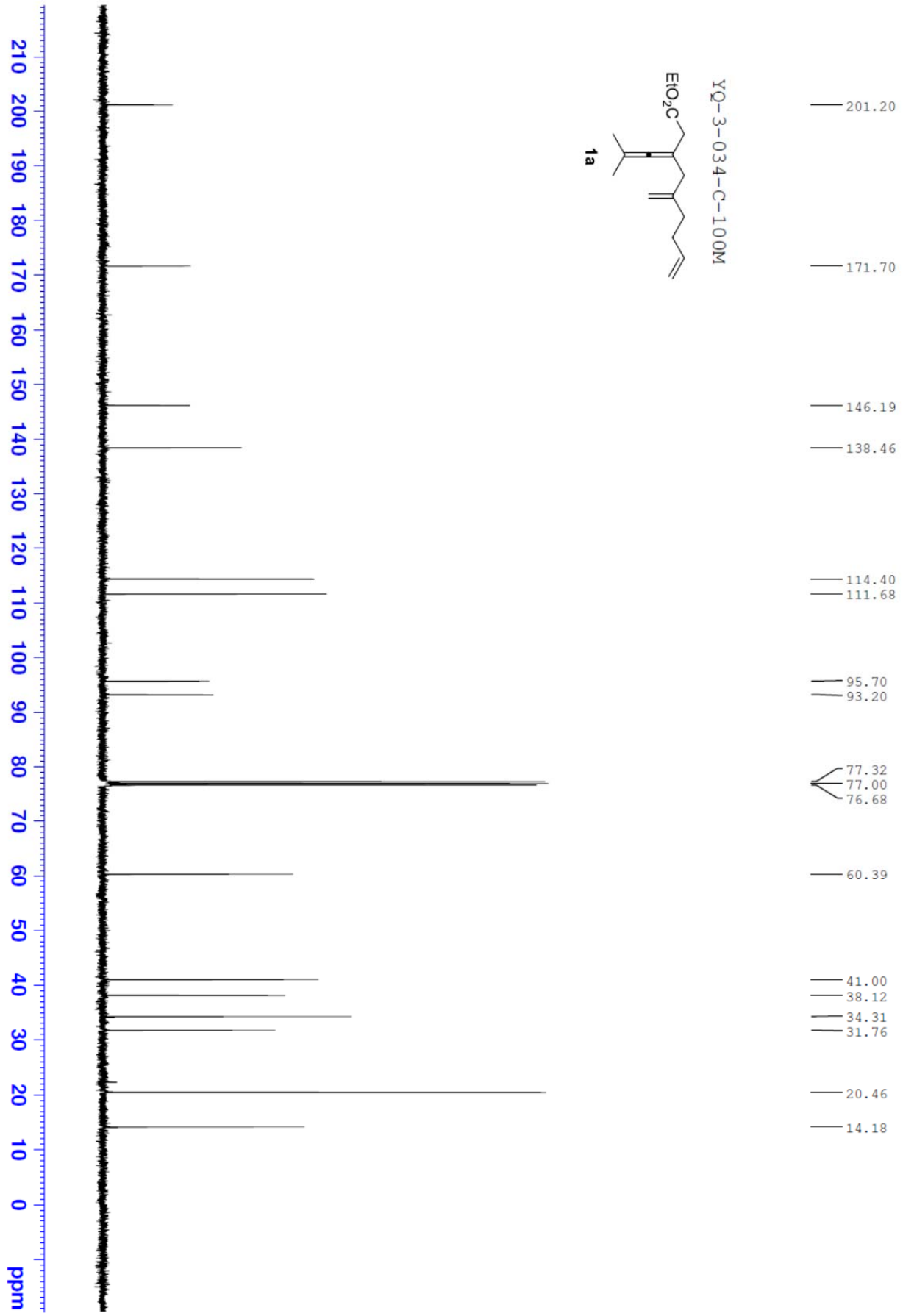
ppm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77.32
77.00
76.68
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14.18
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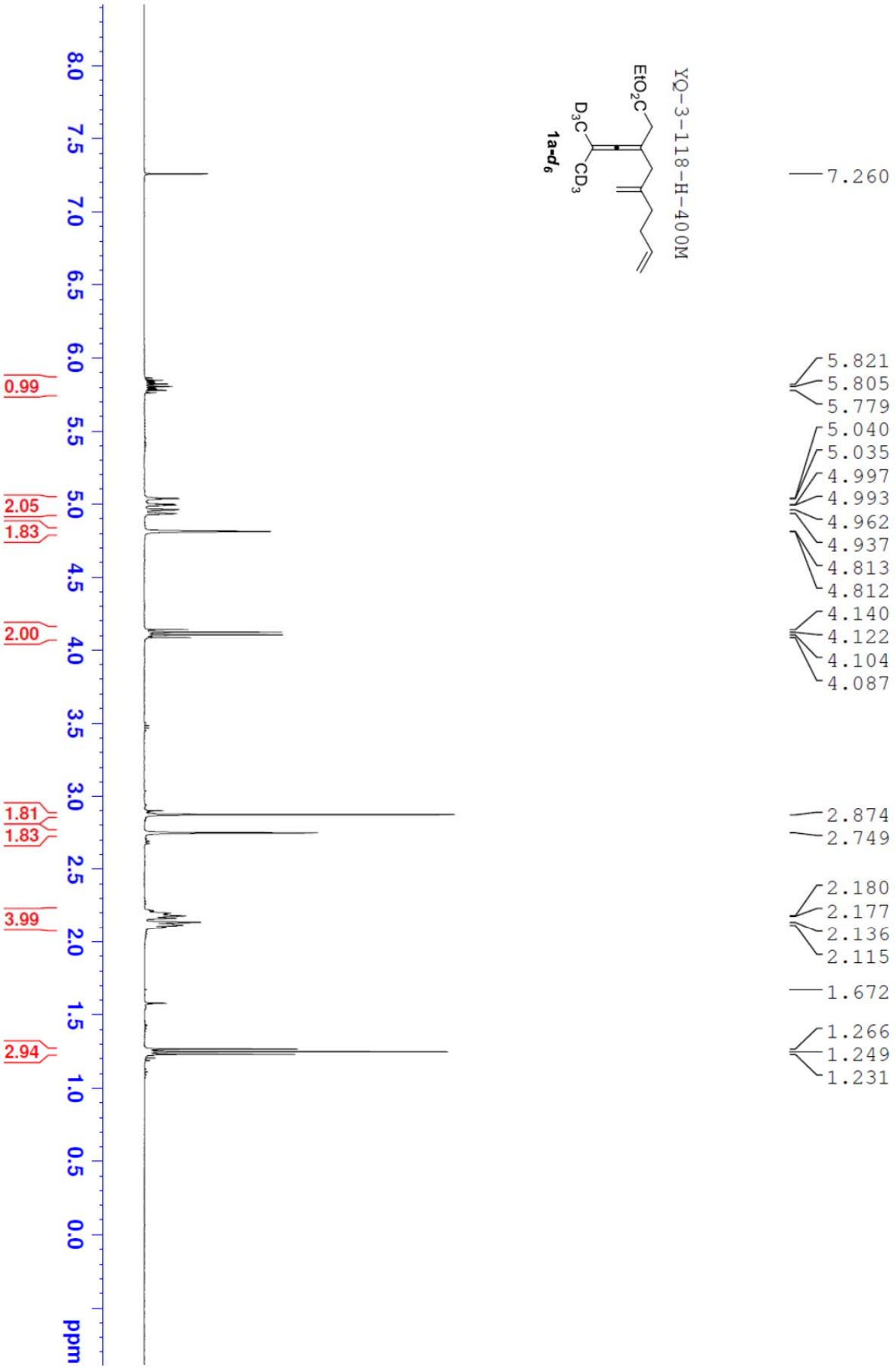
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4.104
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7.0
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6.0
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1.0
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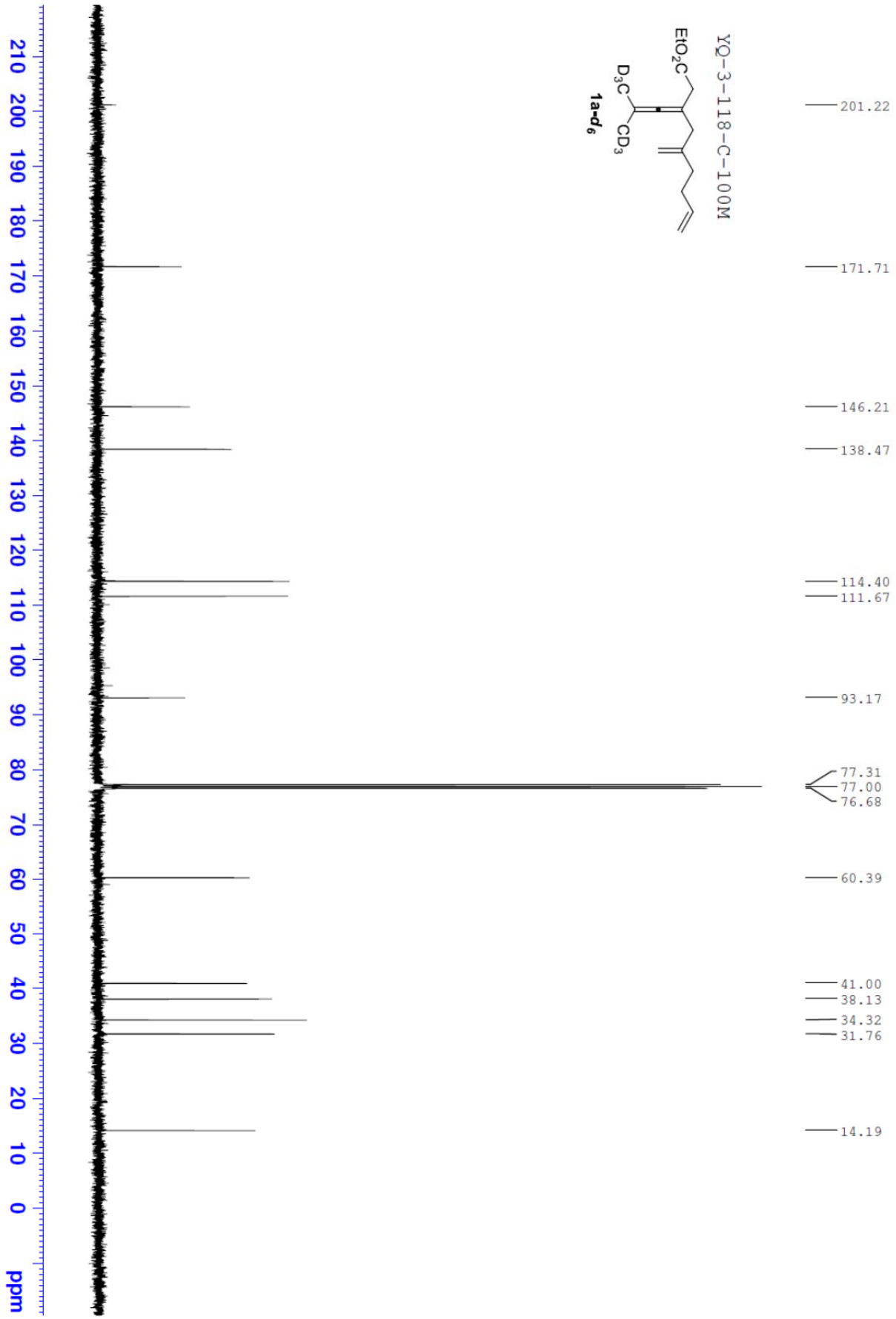
ppm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14.19
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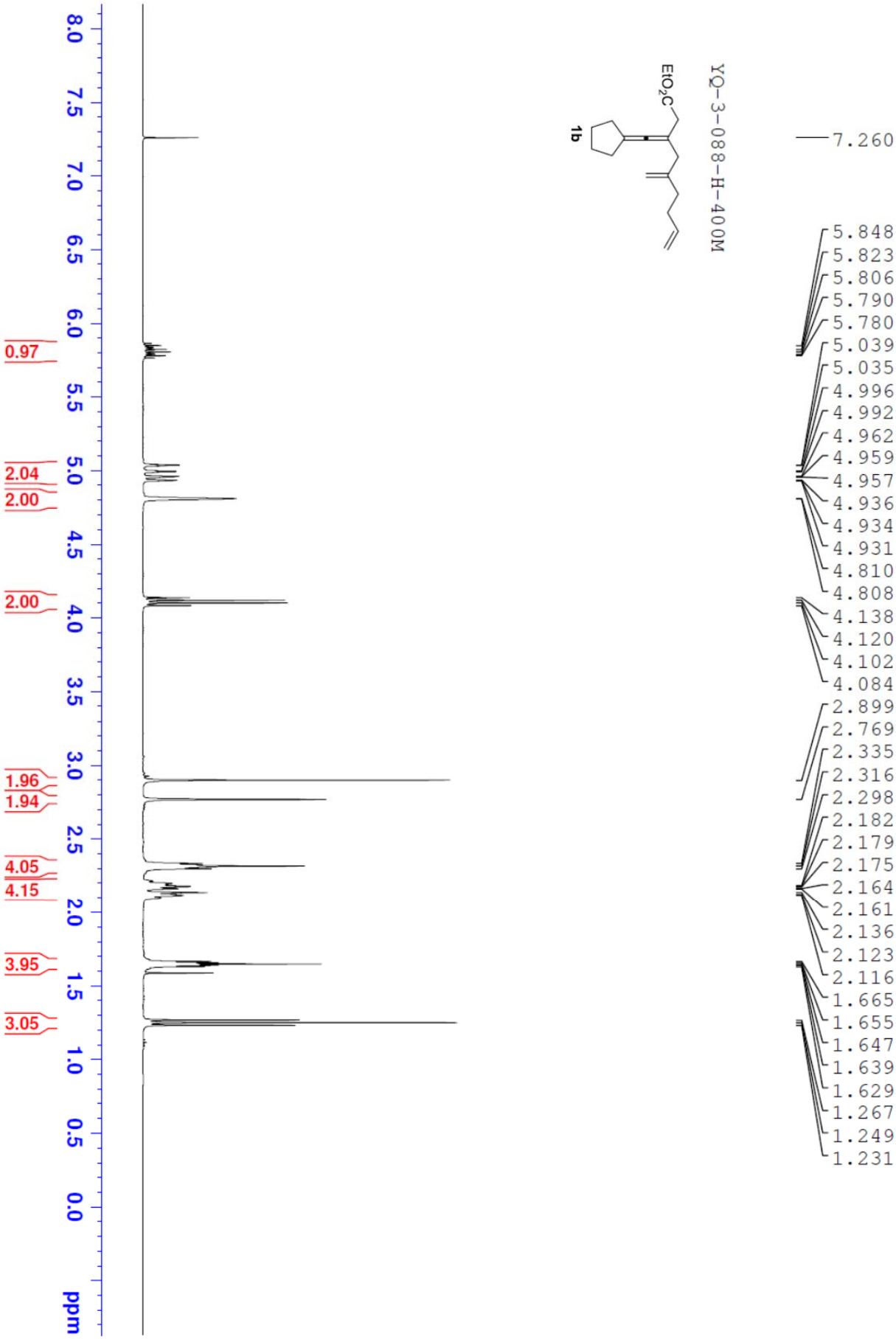
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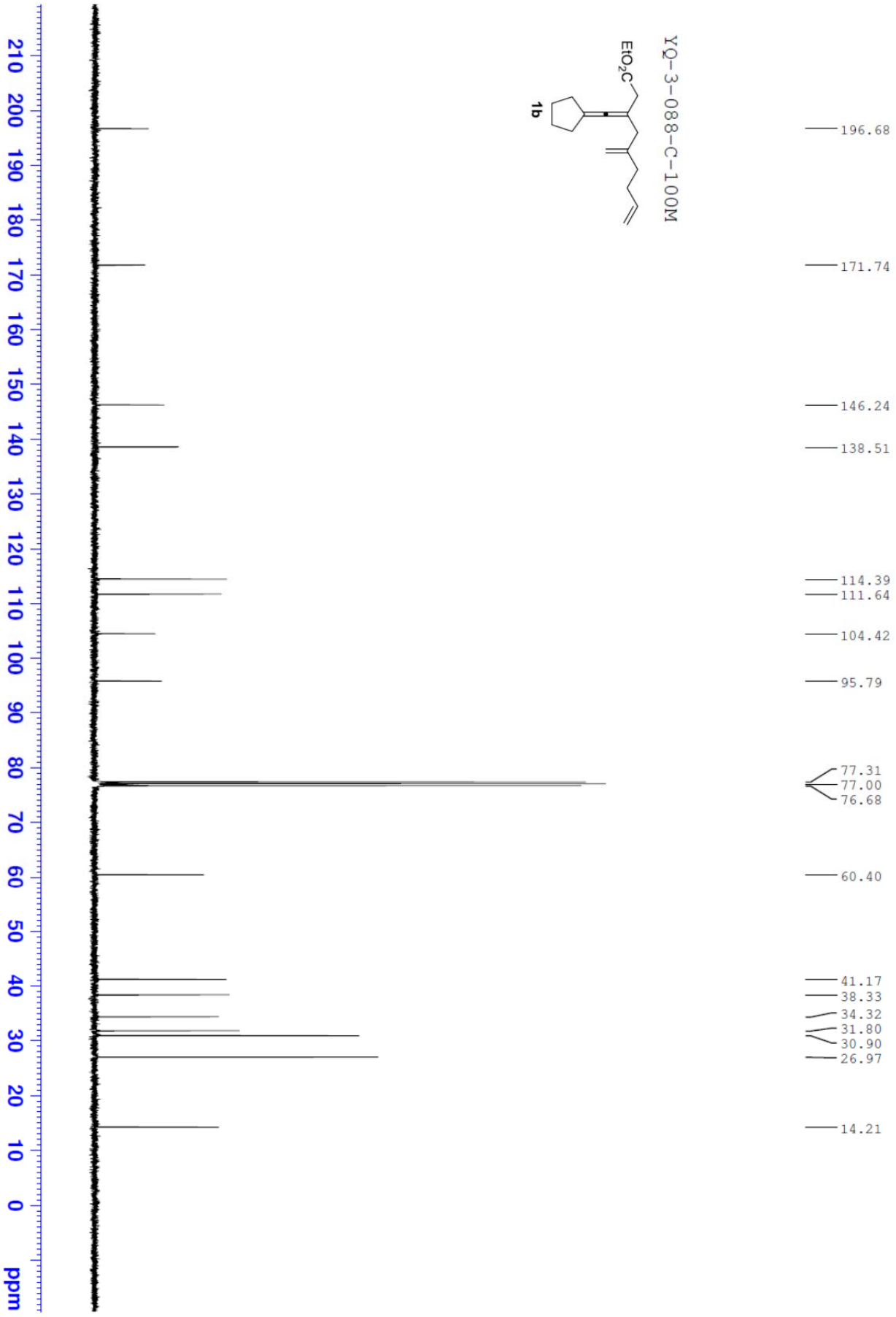
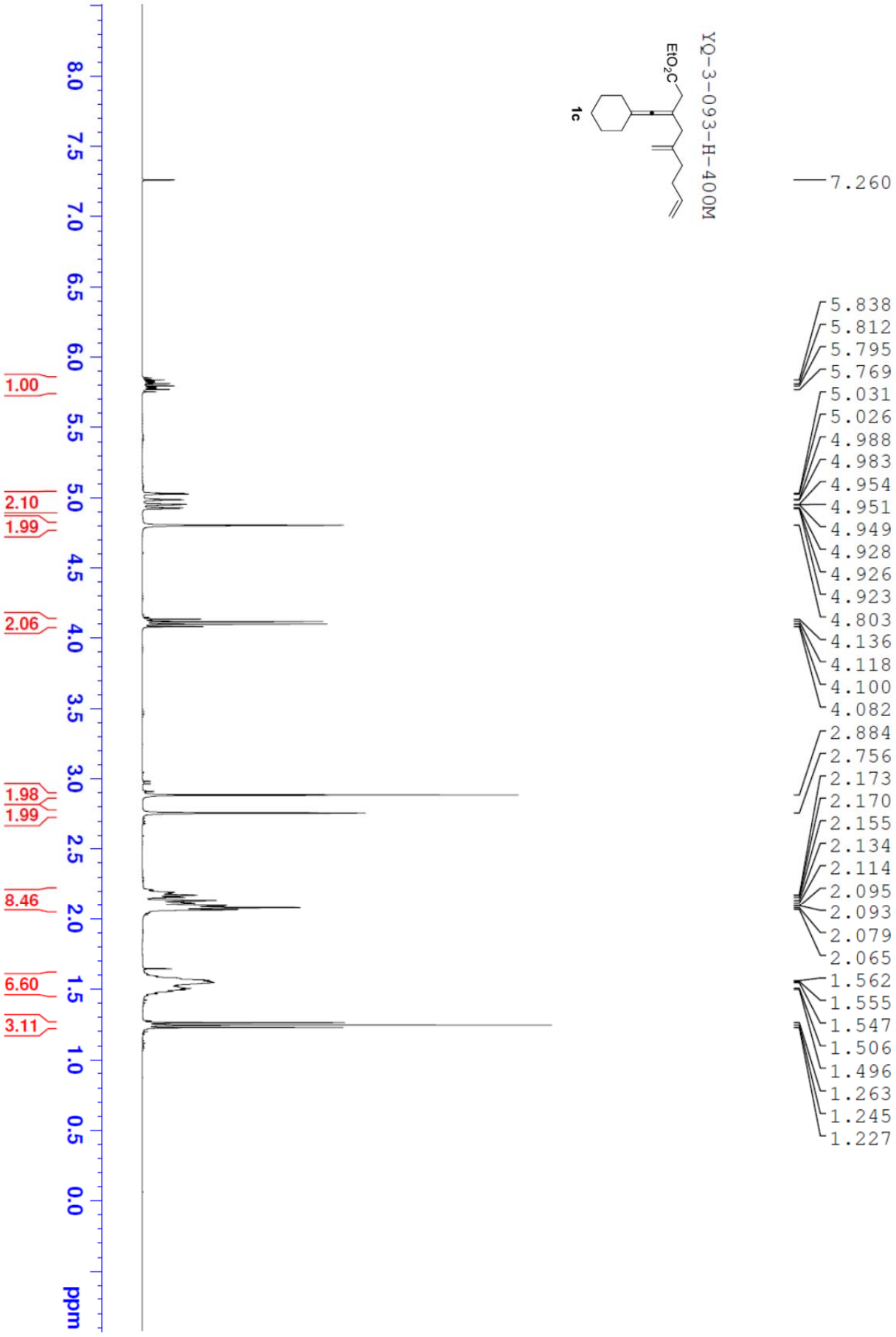
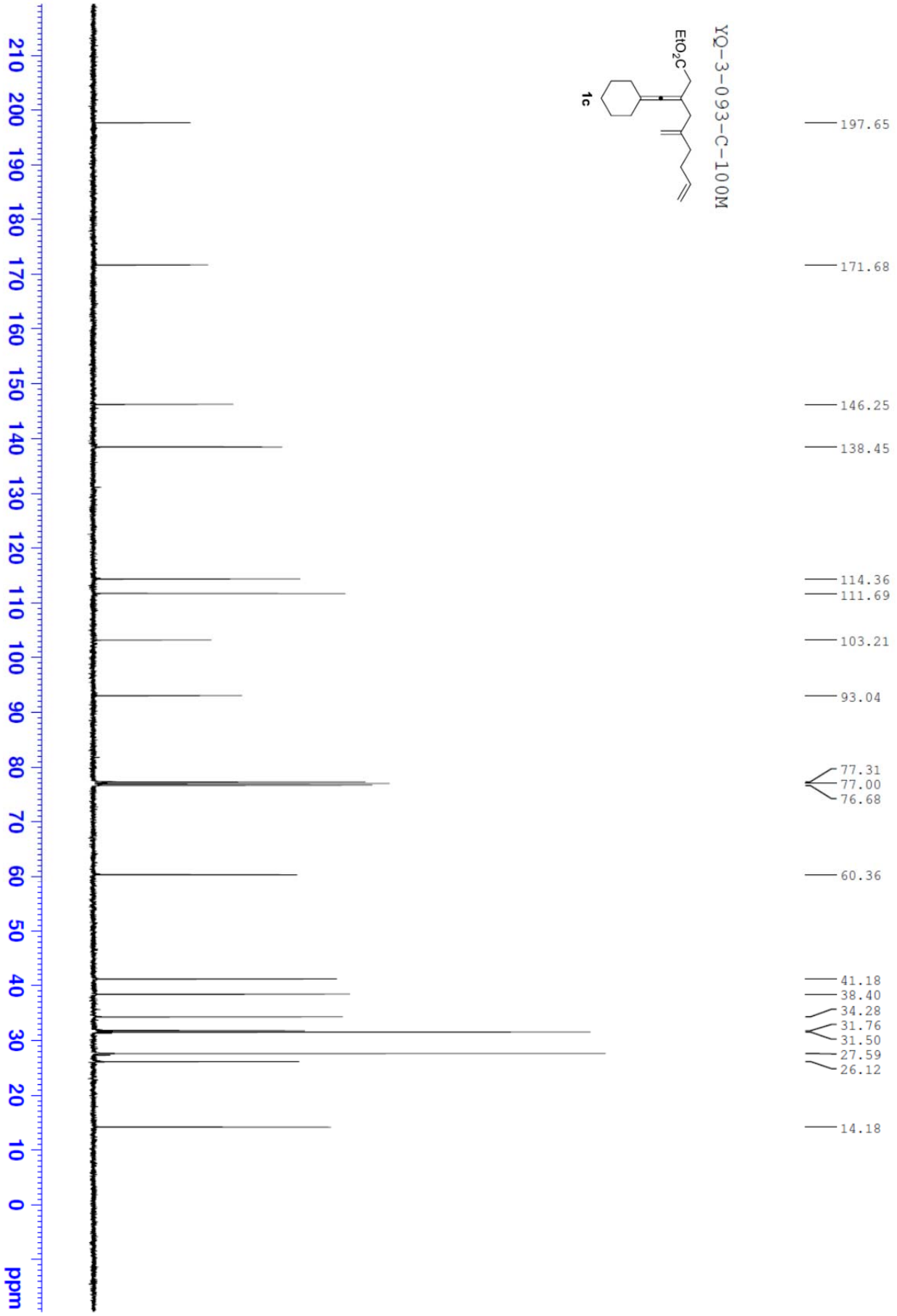
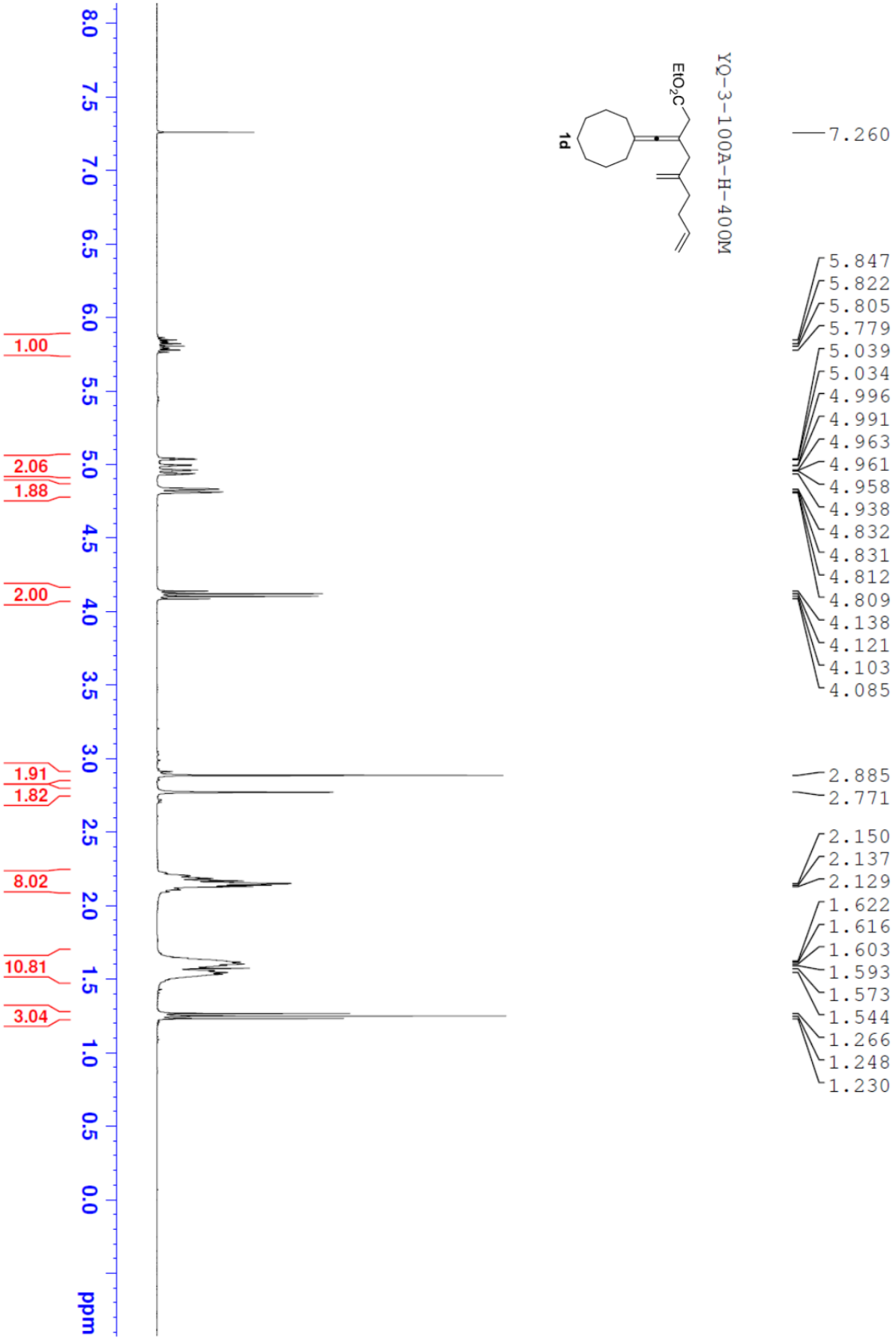
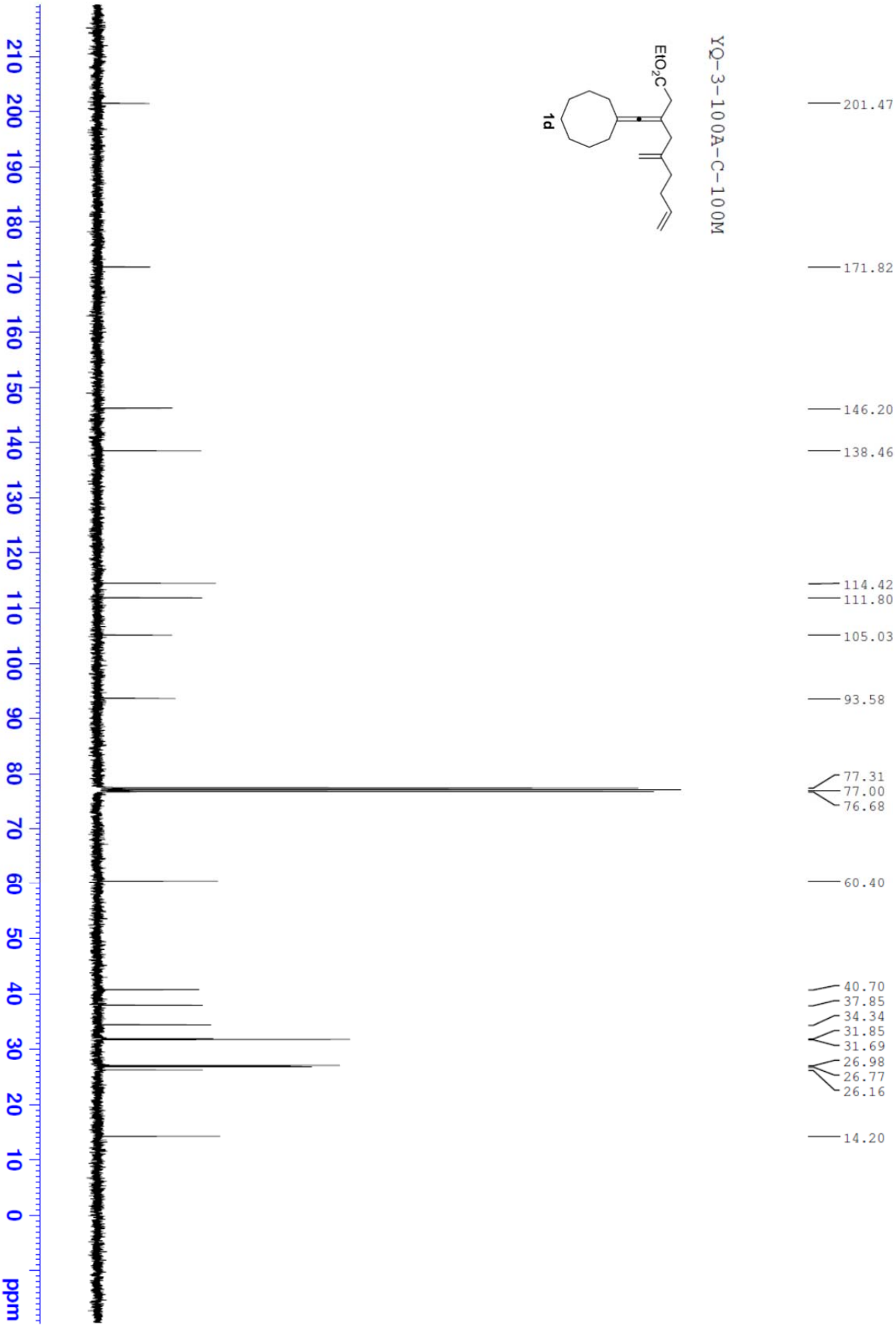
4.934
4.931
4.810
4.808
4.138
4.120
4.102
4.084
2.899
2.769
2.335
2.316
2.298
2.182
2.179
2.175
2.164
2.161
2.136
2.123
2.116
1.665
1.655
1.647
1.639
1.629
1.267
1.249
1.231
0.97
2.04
2.00
2.00
1.96
1.94
4.05
4.15
3.95
3.05
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

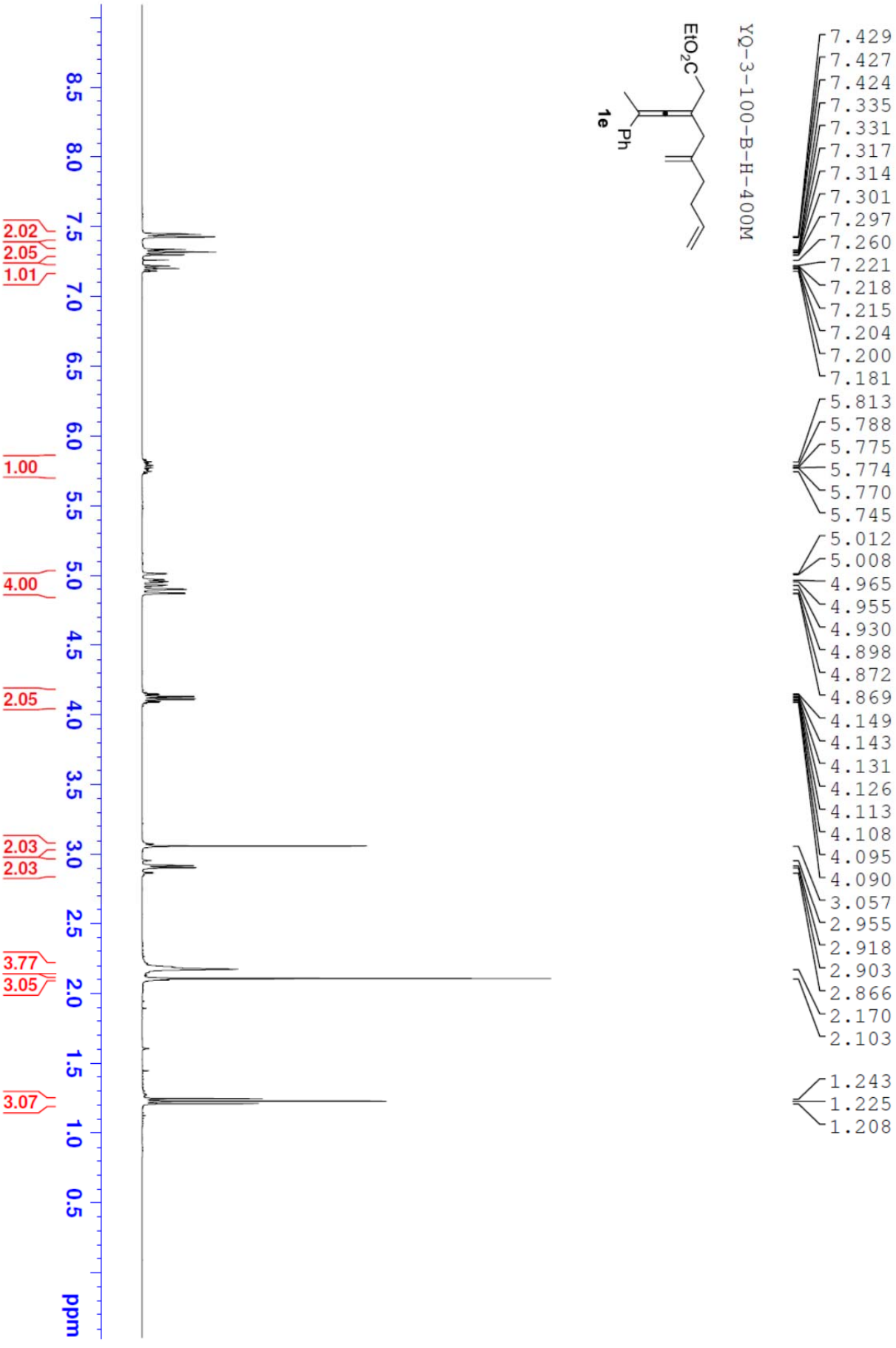

YQ-3-088-C-100M
EtO2C
1b
196.68
171.74
146.24
138.51
114.39
111.64
104.42
95.79
77.31
77.00
76.68
60.40
41.17
38.33
34.32
31.80
30.90
26.97
14.21
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

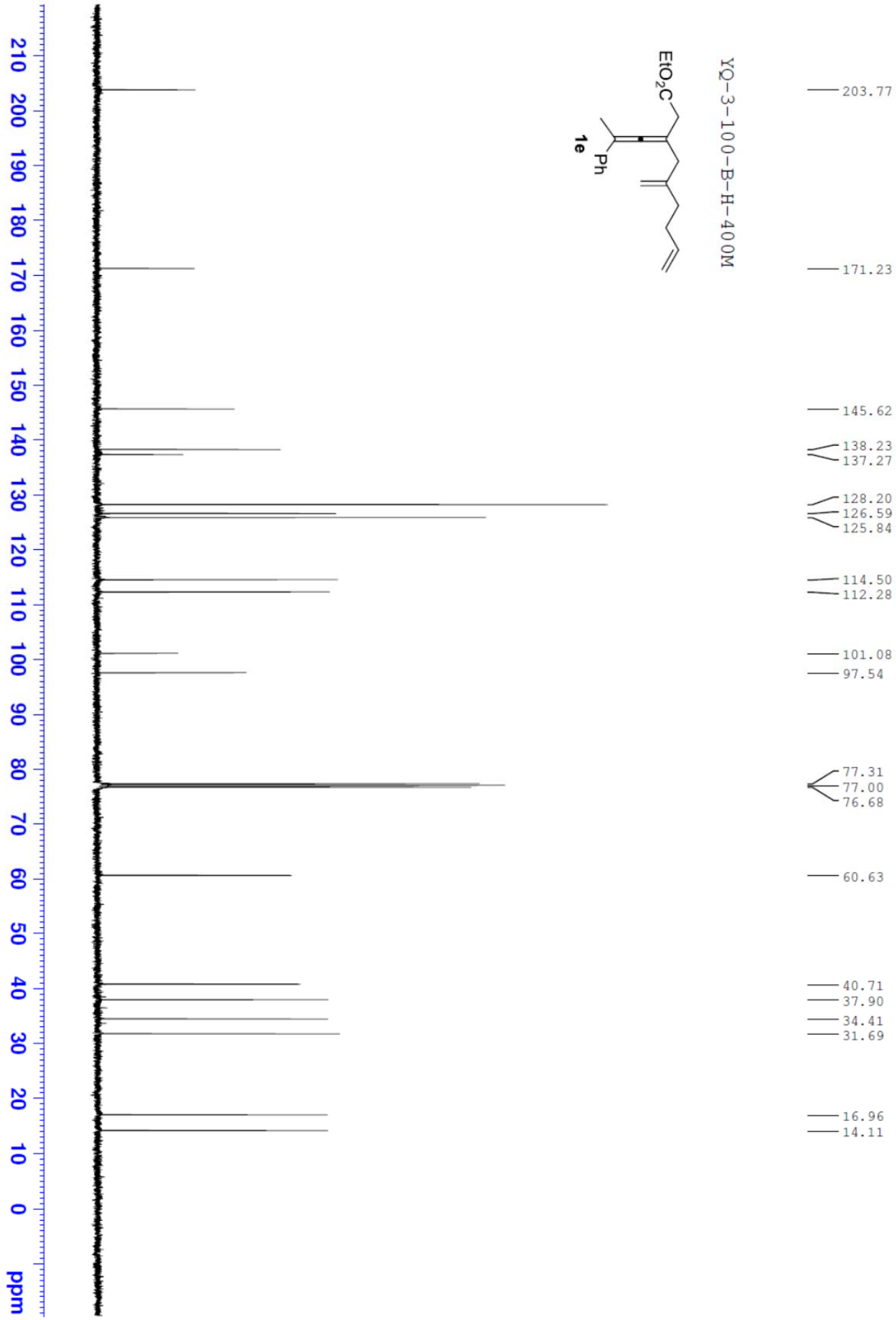

YQ-3-093-H-400M
EtO2C
1c
7.260
5.838
5.812
5.795
5.769
5.031
5.026
4.988
4.983
4.954
4.951
4.949
4.928
4.926
4.923
4.803
4.136
4.118
4.100
4.082
2.884
2.756
2.173
2.170
2.155
2.134
2.114
2.095
2.093
2.079
2.065
1.562
1.555
1.547
1.506
1.496
1.263
1.245
1.227
1.00
2.10
1.99
2.06
1.98
1.99
8.46
6.60
3.11
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

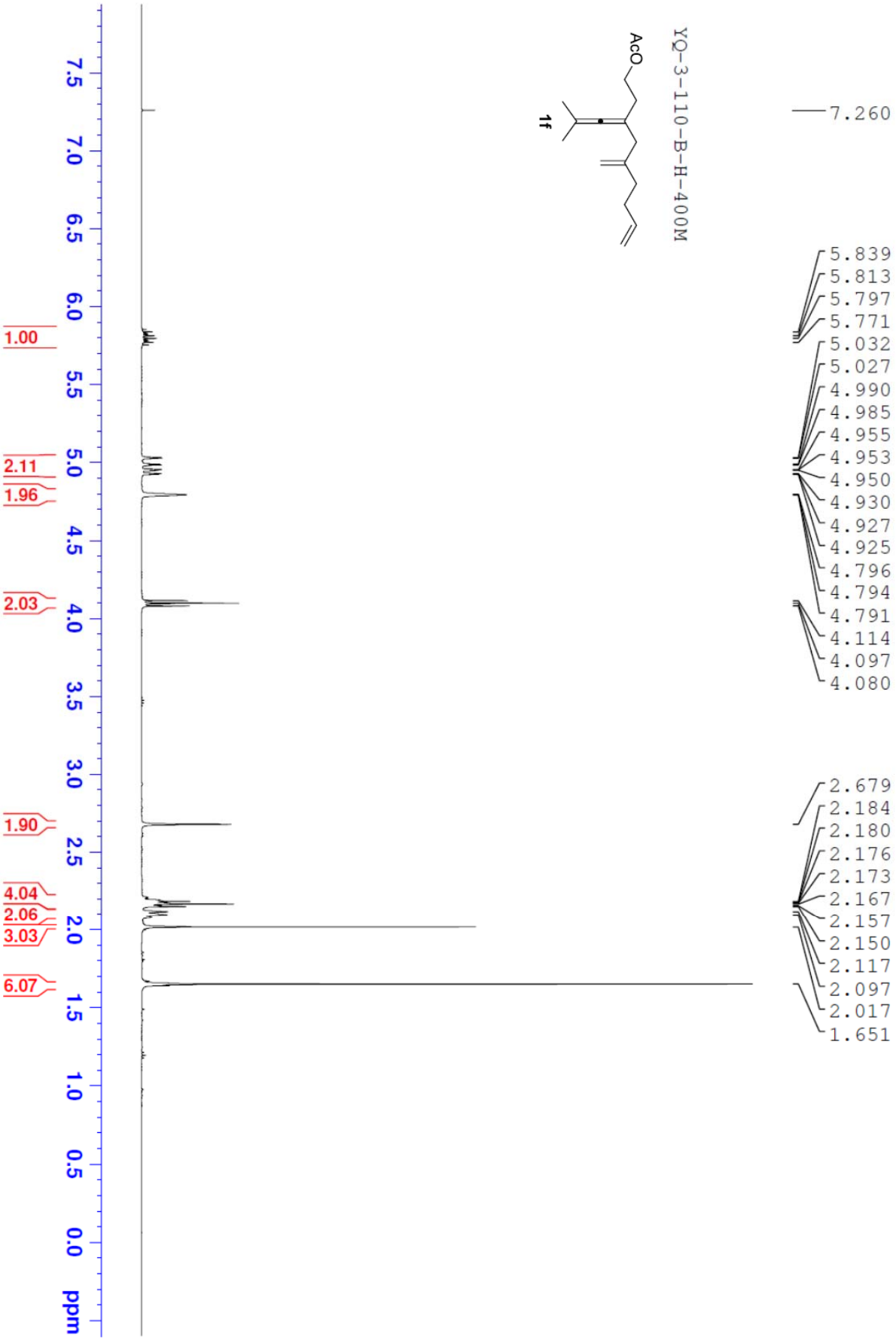

YQ-3-093-C-100M
EtO2C
1c
197.65
171.68
146.25
138.45
114.36
111.69
103.21
93.04
77.31
77.00
76.68
60.36
41.18
38.40
34.28
31.76
31.50
27.59
26.12
14.18
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

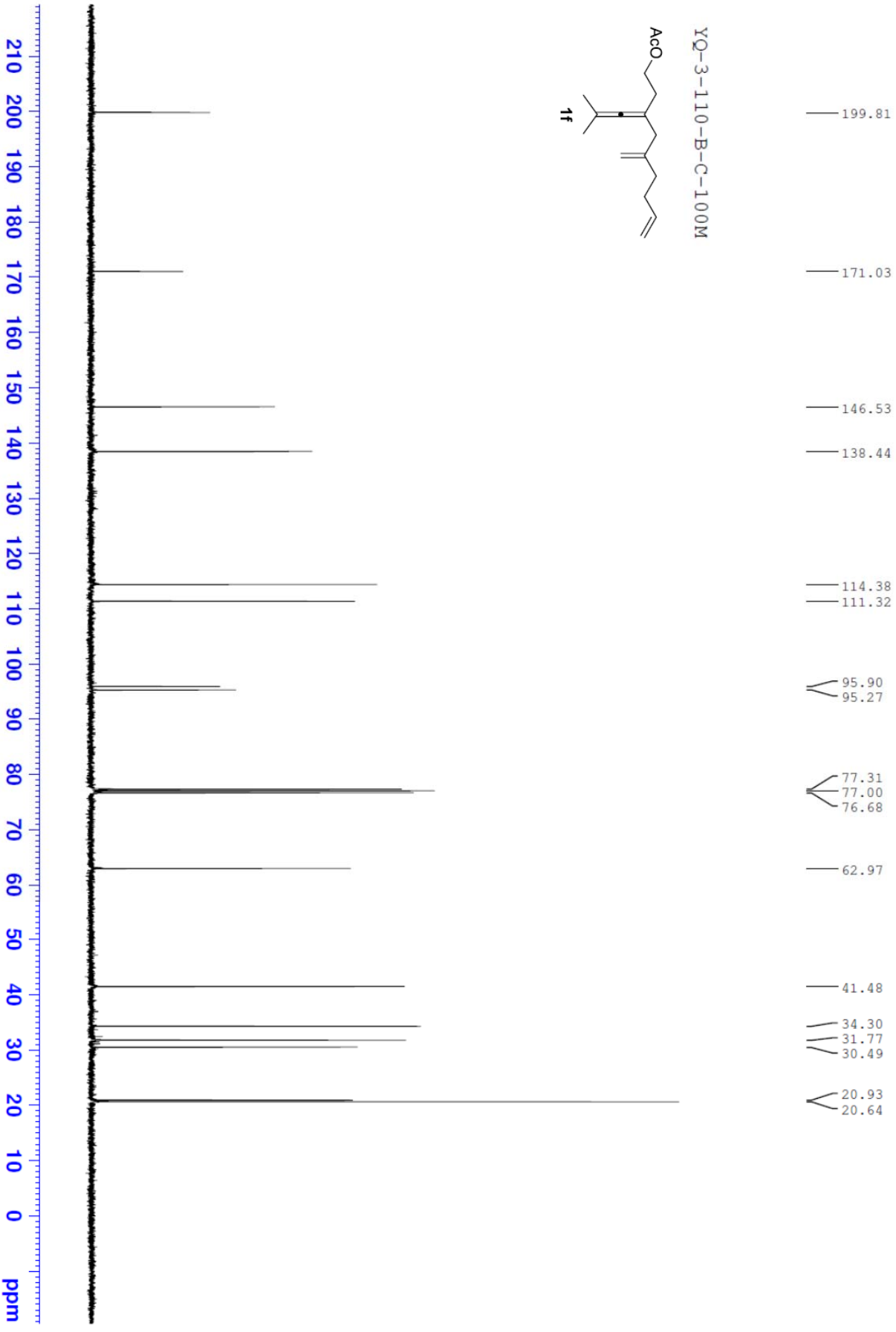

YQ-3-100A-H-400M
EtO2C
1d
7.260
5.847
5.822
5.805
5.779
5.039
5.034
4.996
4.991
4.963
4.961
4.958
4.938
4.832
4.831
4.812
4.809
4.138
4.121
4.103
4.085
2.885
2.771
2.150
2.137
2.129
1.622
1.616
1.603
1.593
1.573
1.544
1.266
1.248
1.230
1.00
2.06
1.88
2.00
1.91
1.82
8.02
10.81
3.04
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

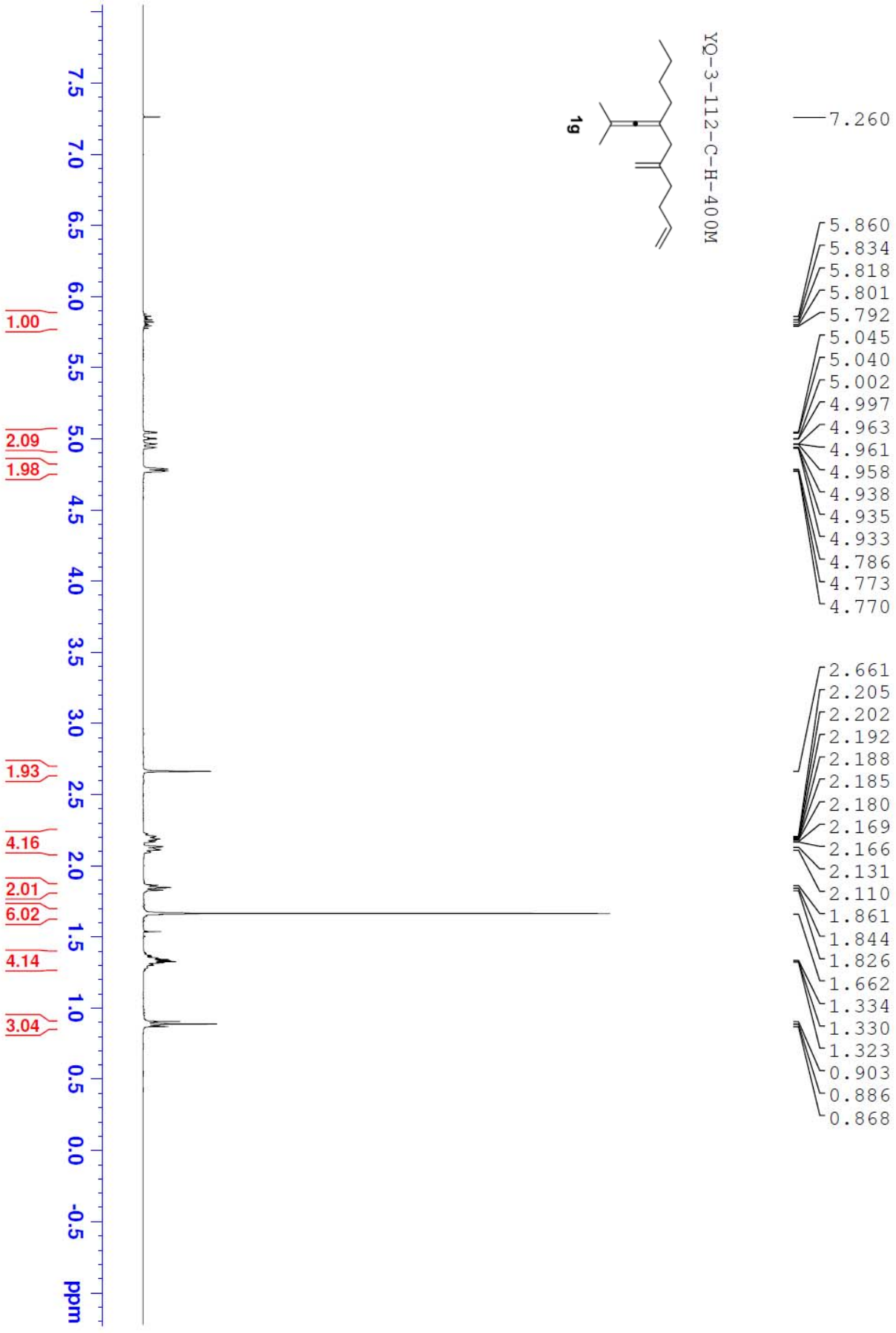

YQ-3-100A-C-100M
EtO2C
1d
201.47
171.82
146.20
138.46
114.42
111.80
105.03
93.58
77.31
77.00
76.68
60.40
40.70
37.85
34.34
31.85
31.69
26.98
26.77
26.16
14.20
ppm

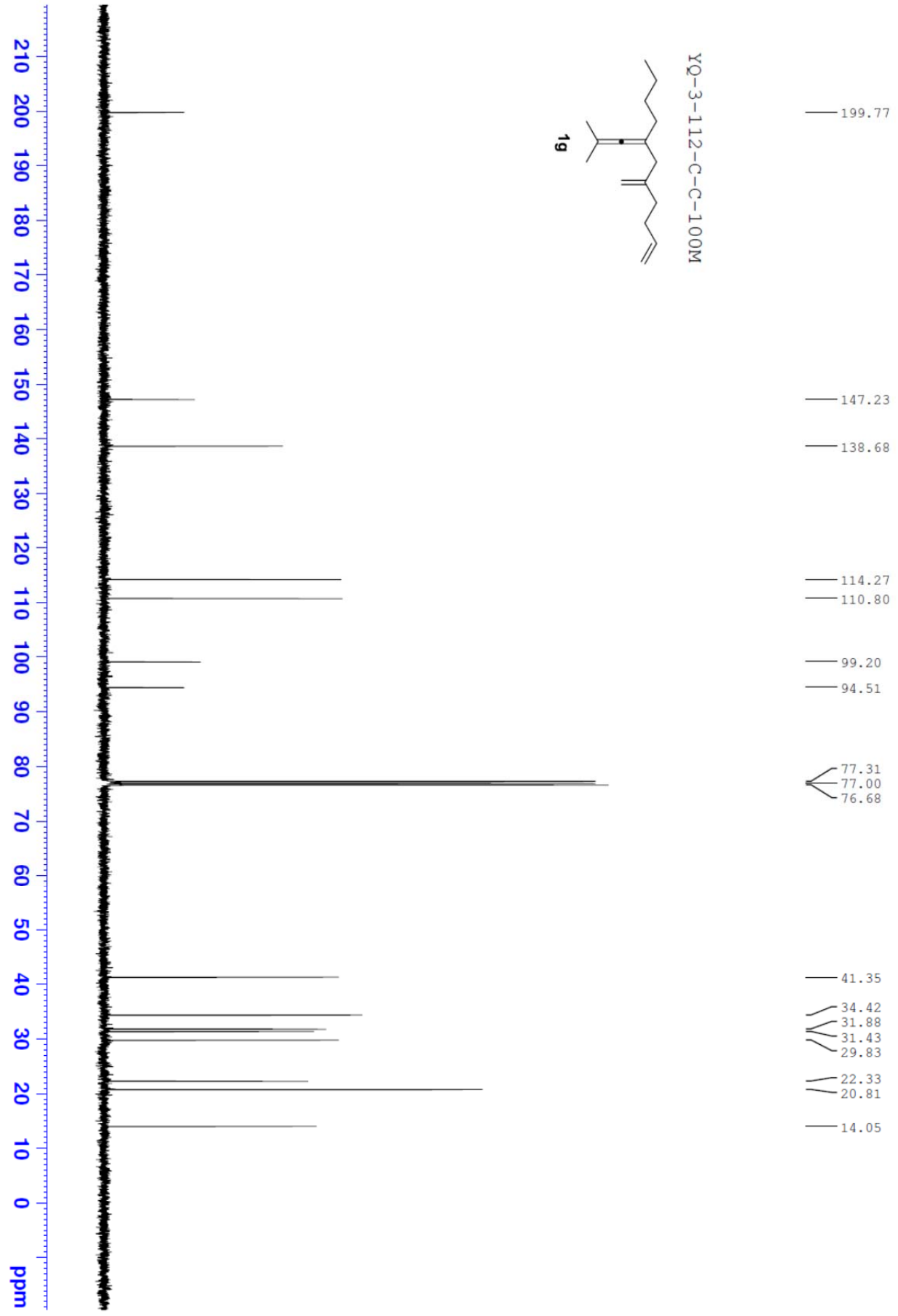

YQ-3-100-B-H-400M
EtO2C
Ph
1e
7.429
7.427
7.424
7.335
7.331
7.317
7.314
7.301
7.297
7.260
7.221
7.218
7.215
7.204
7.200
7.181
5.813
5.788
5.775
5.774
5.770
5.745
5.012
5.008
4.965
4.955
4.930
4.898
4.872
4.869
4.149
4.143
4.131
4.126
4.113
4.108
4.095
4.090
3.057
2.955
2.918
2.903
2.866
2.170
2.103
1.243
1.225
1.208
2.02
2.05
1.01
1.00
4.00
2.05
2.03
2.03
3.77
3.05
3.07
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
ppm

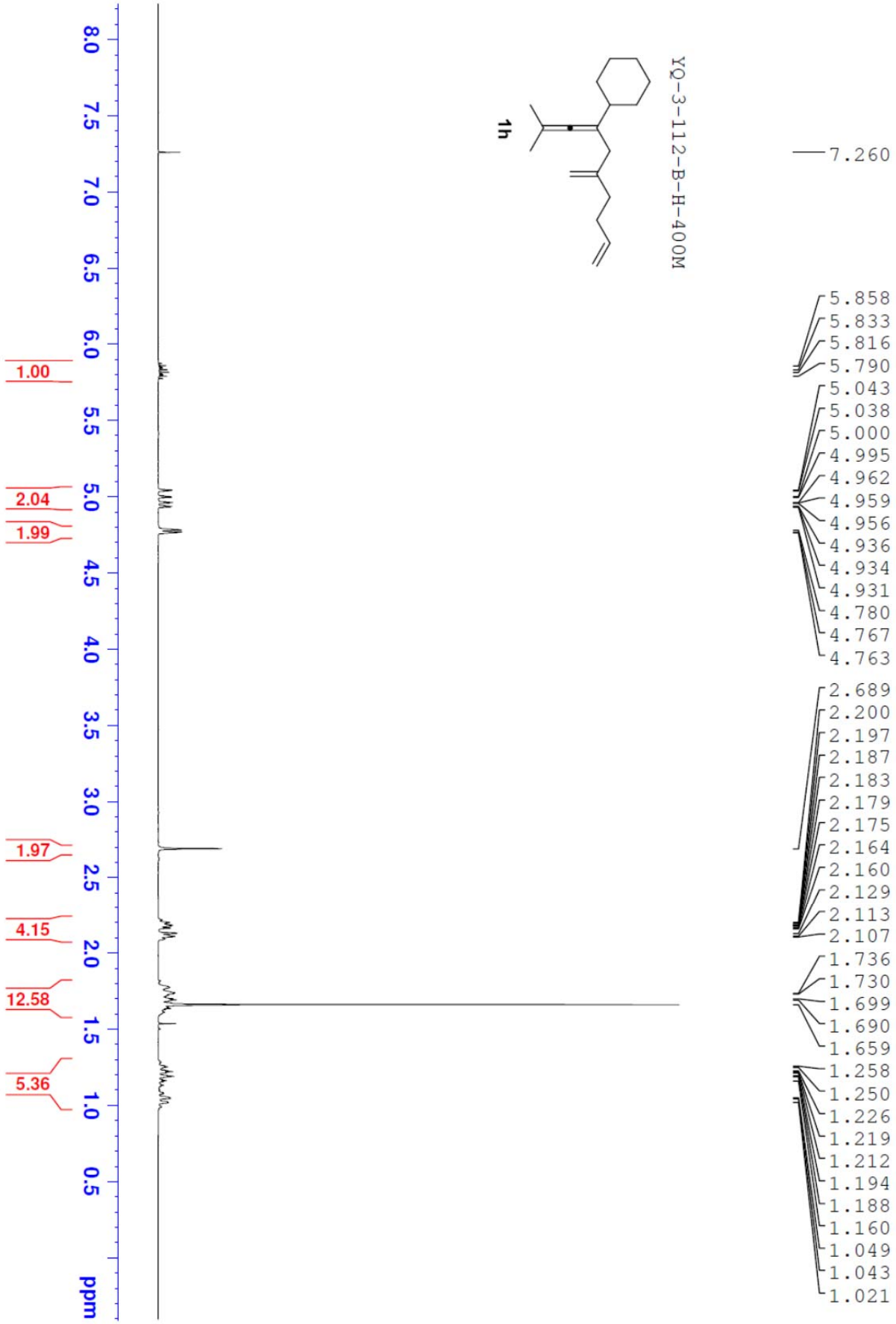

YQ-3-100-B-H-400M
EtO2C
Ph
1e
203.77
171.23
145.62
138.23
137.27
128.20
126.59
125.84
114.50
112.28
101.08
97.54
77.31
77.00
76.68
60.63
40.71
37.90
34.41
31.69
16.96
14.11
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

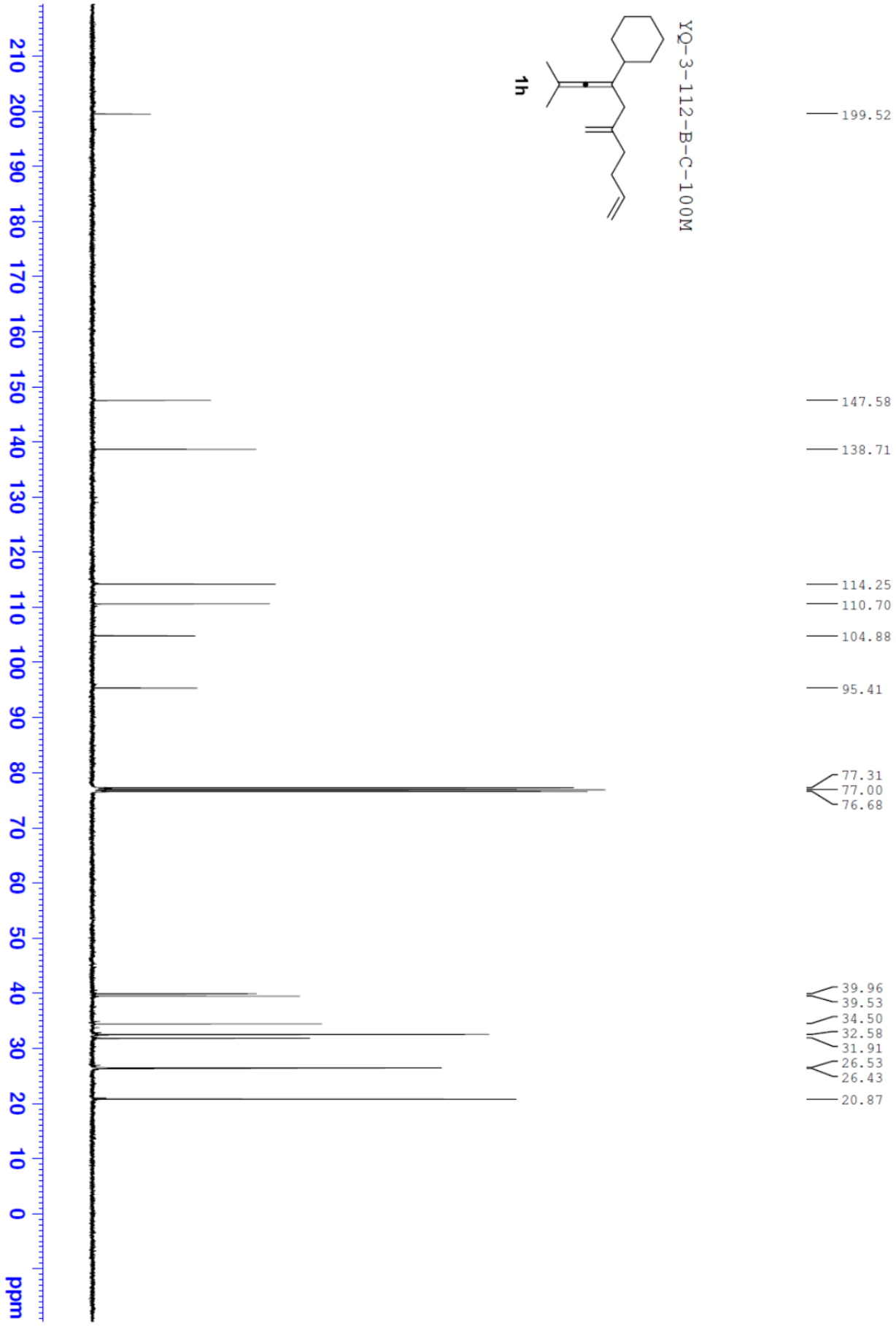

YQ-3-110-B-H-400M
AcO
1f
7.260
5.839
5.813
5.797
5.771
5.032
5.027
4.990
4.985
4.955
4.953
4.950
4.930
4.927
4.925
4.796
4.794
4.791
4.114
4.097
4.080
2.679
2.184
2.180
2.176
2.173
2.167
2.157
2.150
2.117
2.097
2.017
1.651
1.00
2.11
1.96
2.03
1.90
4.04
2.06
3.03
6.07
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

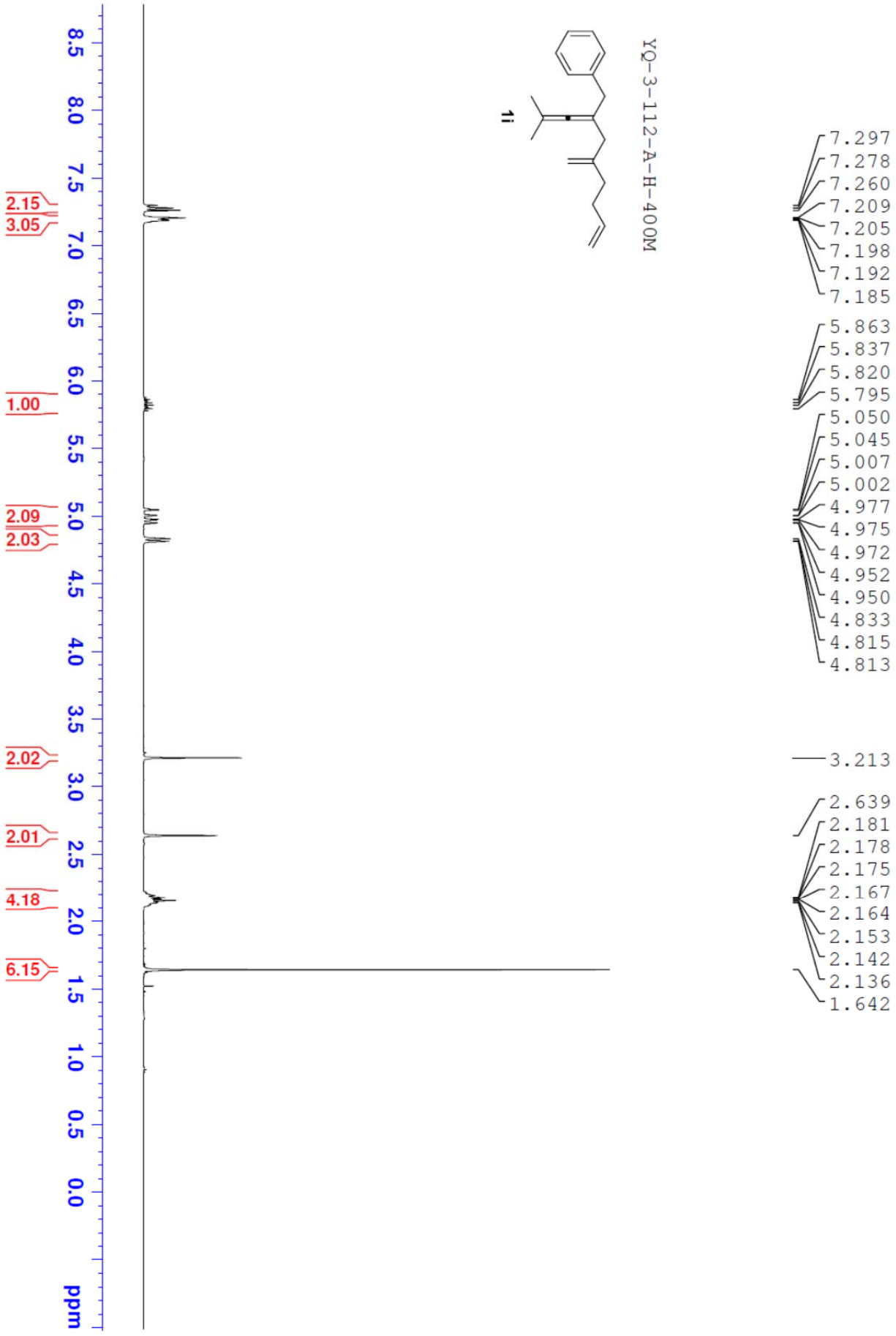

YQ-3-110-B-C-100M
AcO
1f
199.81
171.03
146.53
138.44
114.38
111.32
95.90
95.27
77.31
77.00
76.68
62.97
41.48
34.30
31.77
30.49
20.93
20.64
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

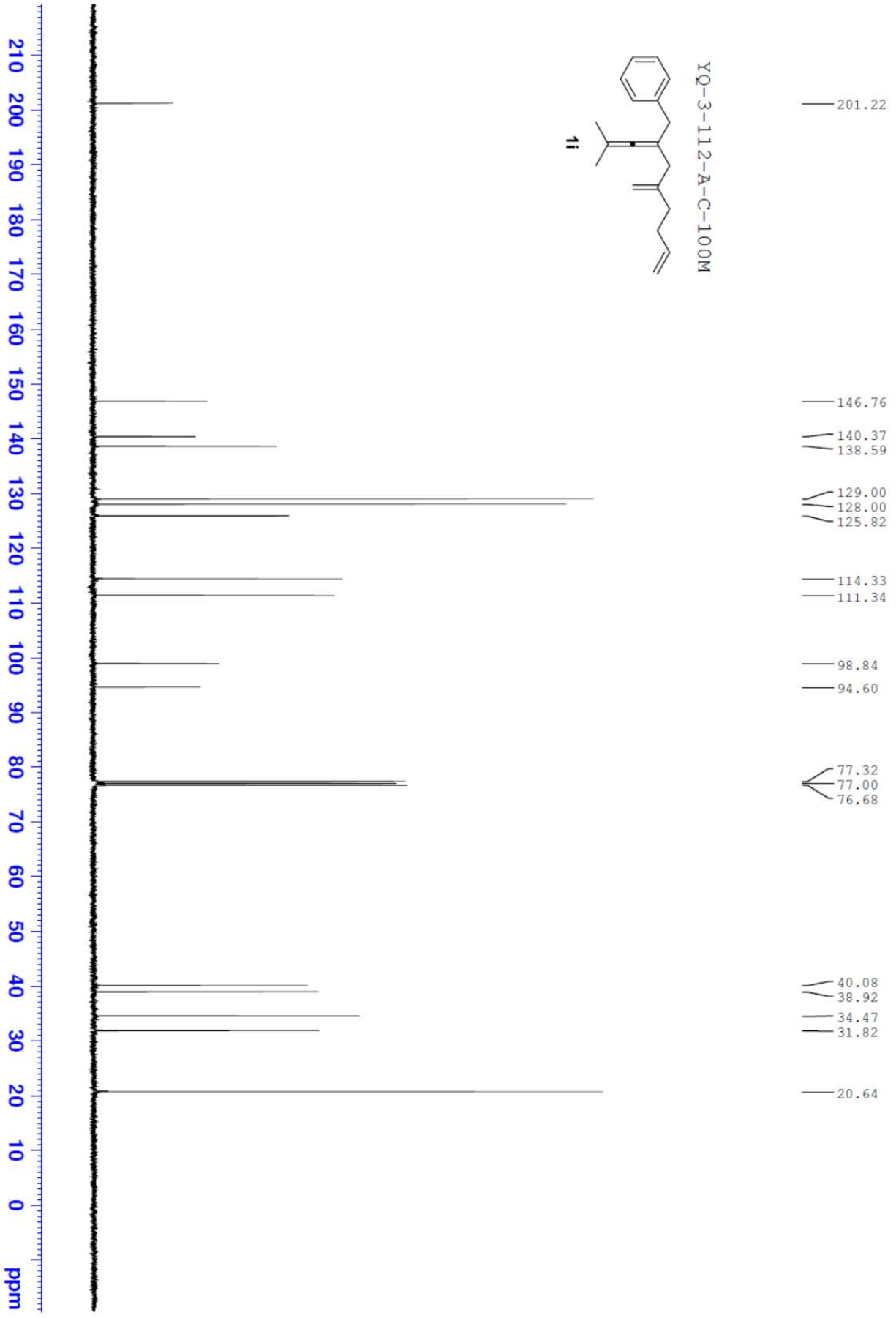

YQ-3-112-C-H-400M
1g
7.260
5.860
5.834
5.818
5.801
5.792
5.045
5.040
5.002
4.997
4.963
4.961
4.958
4.938
4.935
4.933
4.786
4.773
4.770
2.661
2.205
2.202
2.192
2.188
2.185
2.180
2.169
2.166
2.131
2.110
1.861
1.844
1.826
1.662
1.334
1.330
1.323
0.903
0.886
0.868
1.00
2.09
1.98
1.93
4.16
2.01
6.02
4.14
3.04
ppm

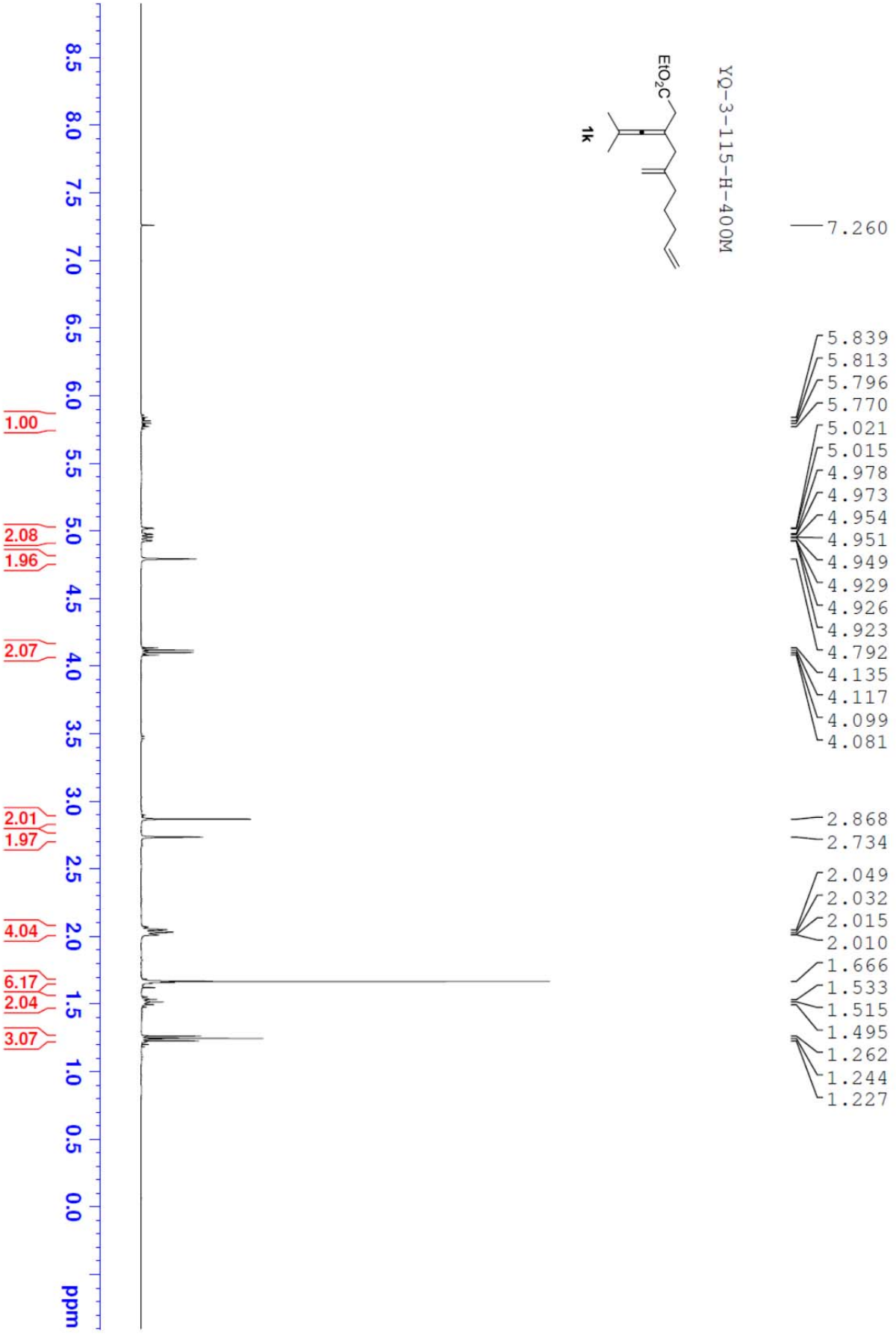

YQ-3-112-C-C-100M
1g
199.77
147.23
138.68
114.27
110.80
99.20
94.51
77.31
77.00
76.68
41.35
34.42
31.88
31.43
29.83
22.33
20.81
14.05
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

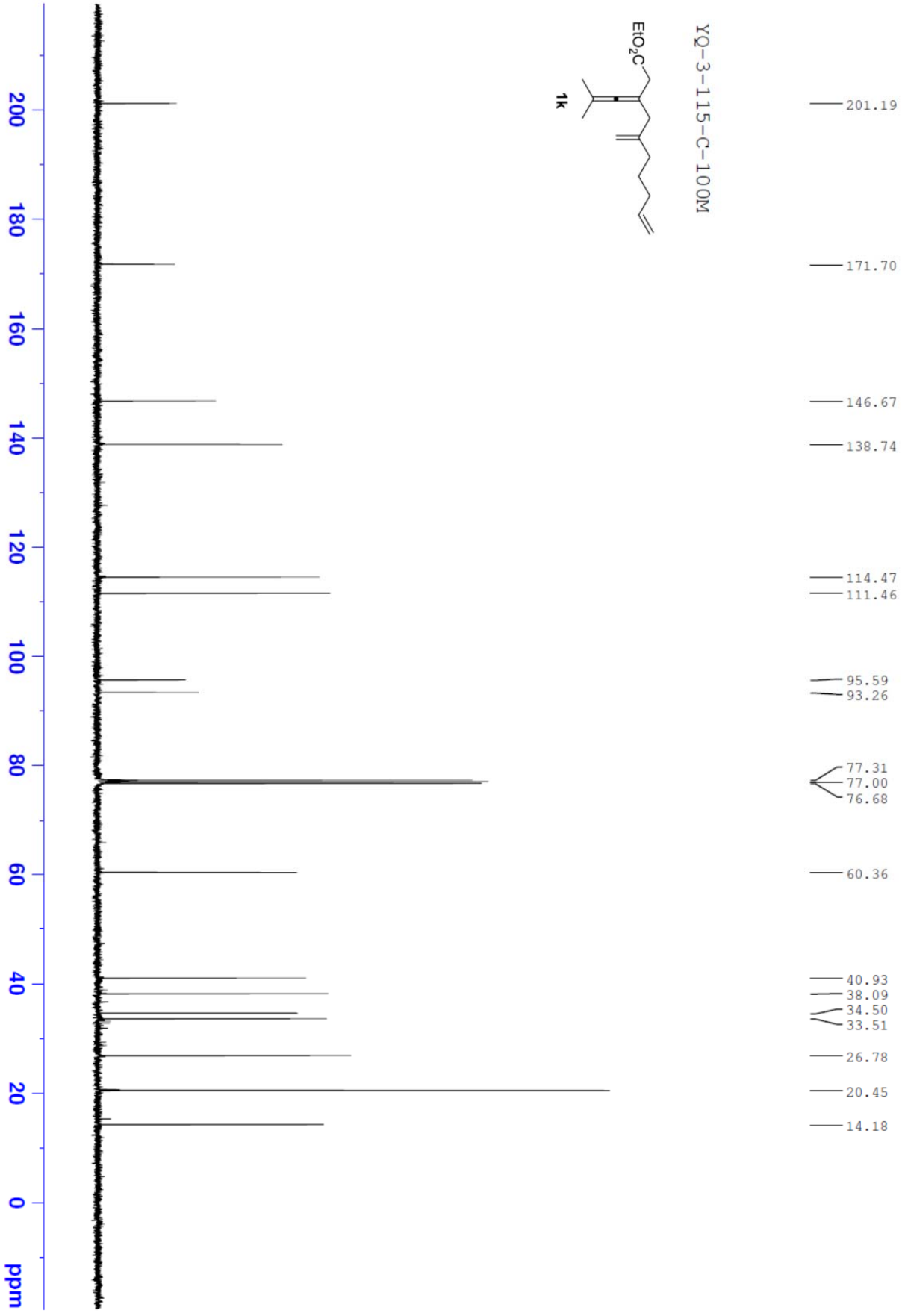

YQ-3-112-B-H-400M
1h
7.260
5.858
5.833
5.816
5.790
5.043
5.038
5.000
4.995
4.962
4.959
4.956
4.936
4.934
4.931
4.780
4.767
4.763
2.689
2.200
2.197
2.187
2.183
2.179
2.175
2.164
2.160
2.129
2.113
2.107
1.736
1.730
1.699
1.690
1.659
1.258
1.250
1.226
1.219
1.212
1.194
1.188
1.160
1.049
1.043
1.021
1.00
2.04
1.99
1.97
4.15
12.58
5.36
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
ppm

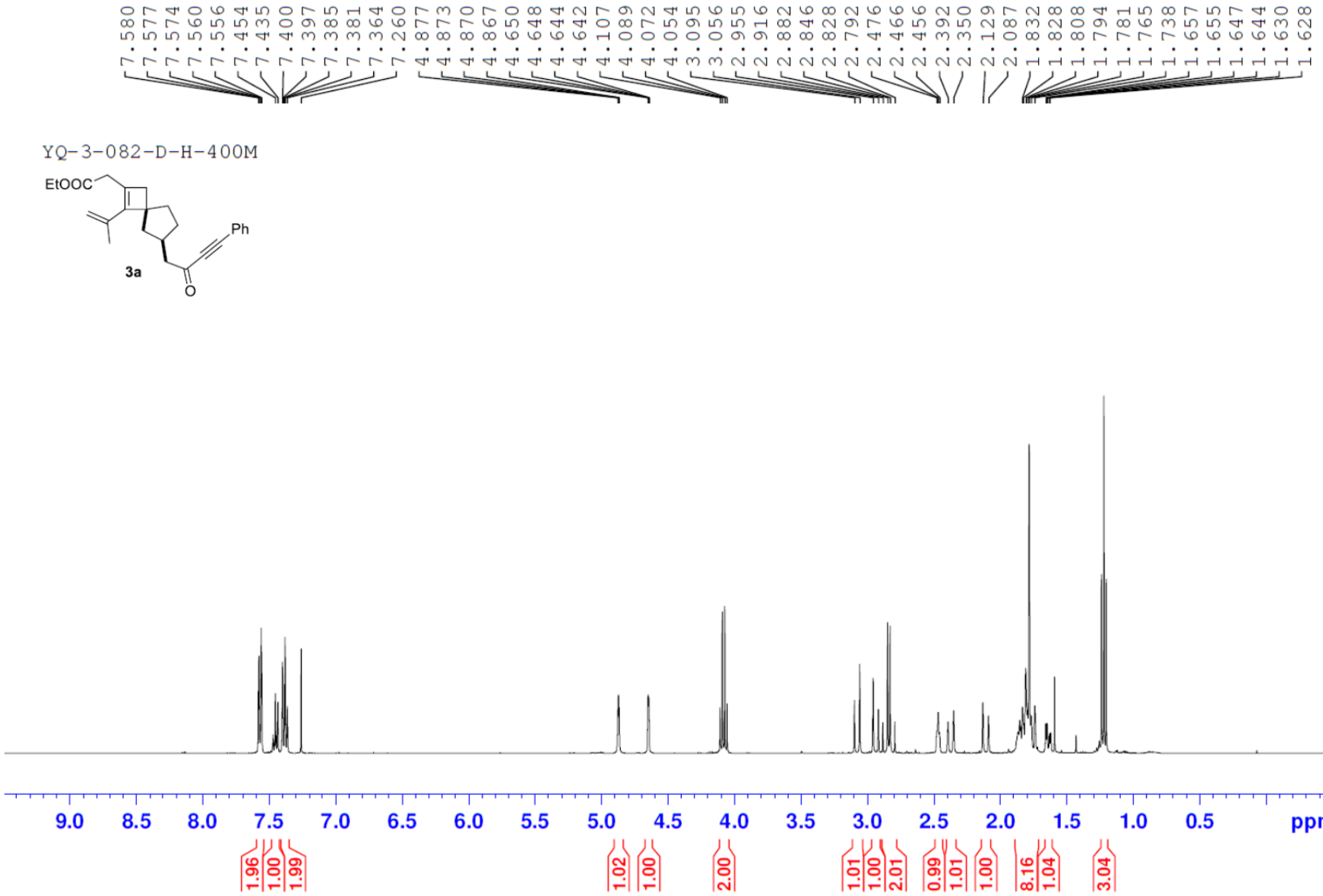

YQ-3-112-B-C-100M
1h
199.52
147.58
138.71
114.25
110.70
104.88
95.41
77.31
77.00
76.68
39.96
39.53
34.50
32.58
31.91
26.53
26.43
20.87
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

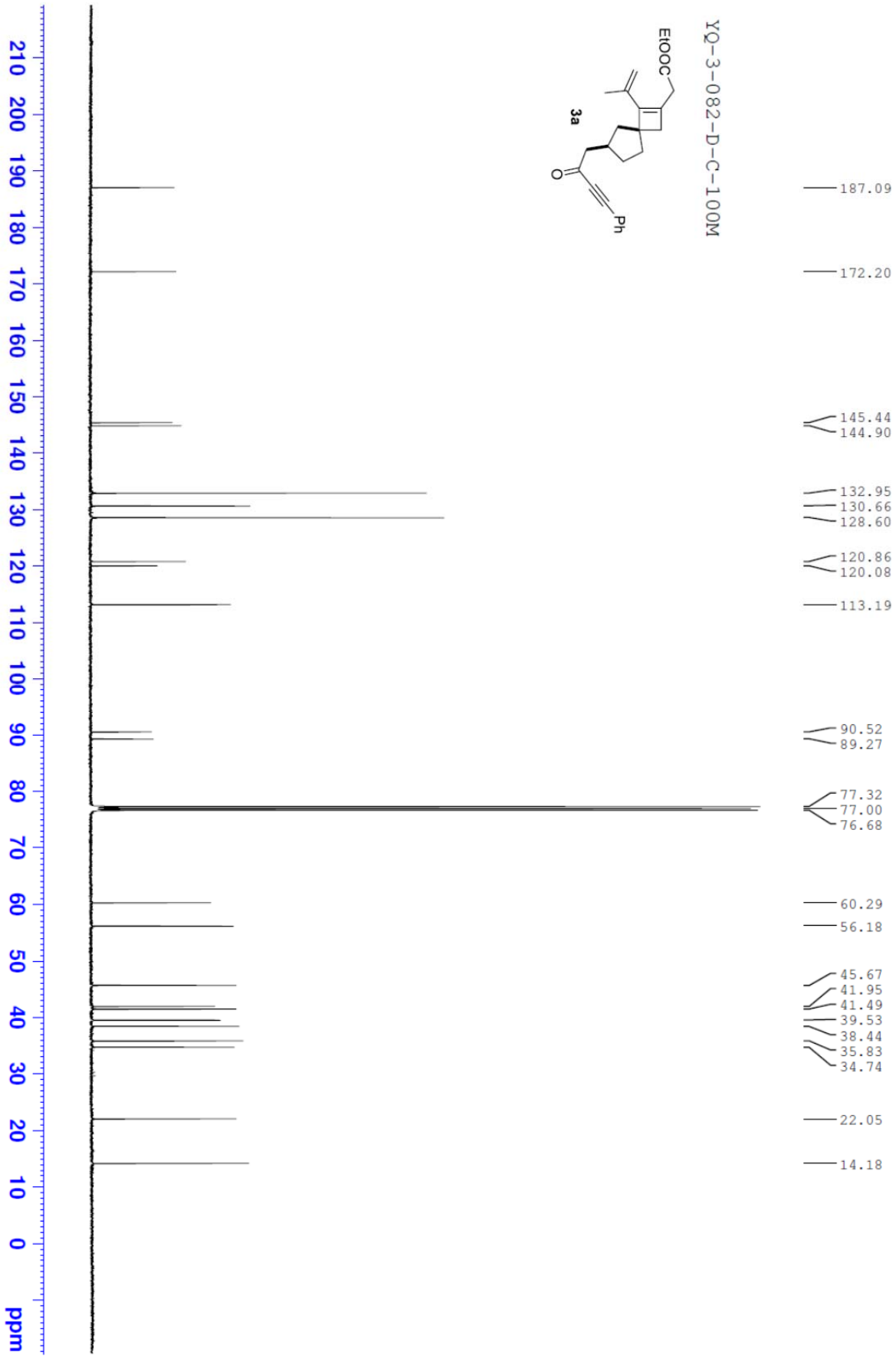

YQ-3-112-A-H-400M
1i
7.297
7.278
7.260
7.209
7.205
7.198
7.192
7.185
5.863
5.837
5.820
5.795
5.050
5.045
5.007
5.002
4.977
4.975
4.972
4.952
4.950
4.833
4.815
4.813
3.213
2.639
2.181
2.178
2.175
2.167
2.164
2.153
2.142
2.136
1.642
2.15
3.05
1.00
2.09
2.03
2.02
2.01
4.18
6.15
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

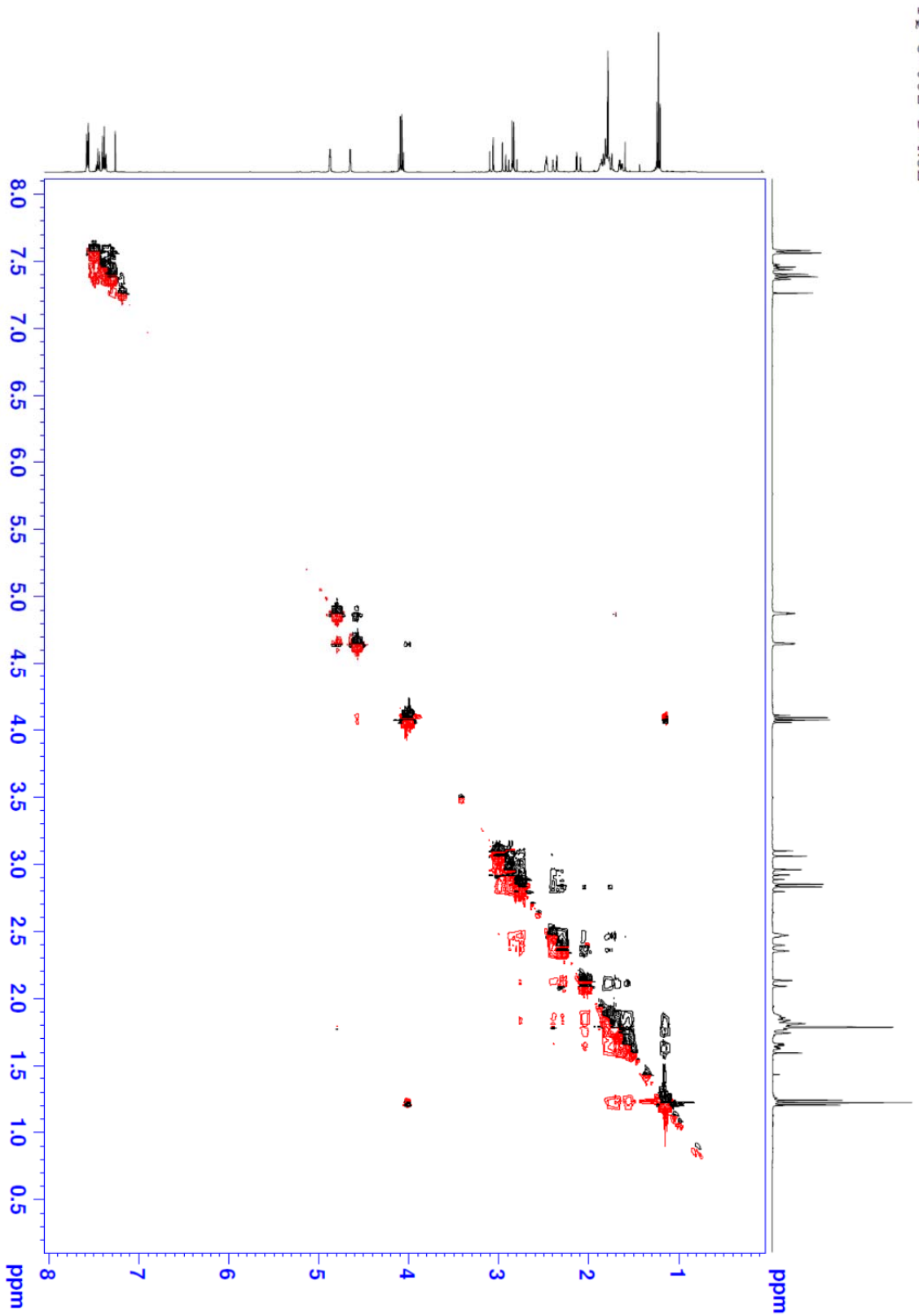

YQ-3-112-A-C-100M
1i
201.22
146.76
140.37
138.59
129.00
128.00
125.82
114.33
111.34
98.84
94.60
77.32
77.00
76.68
40.08
38.92
34.47
31.82
20.64
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

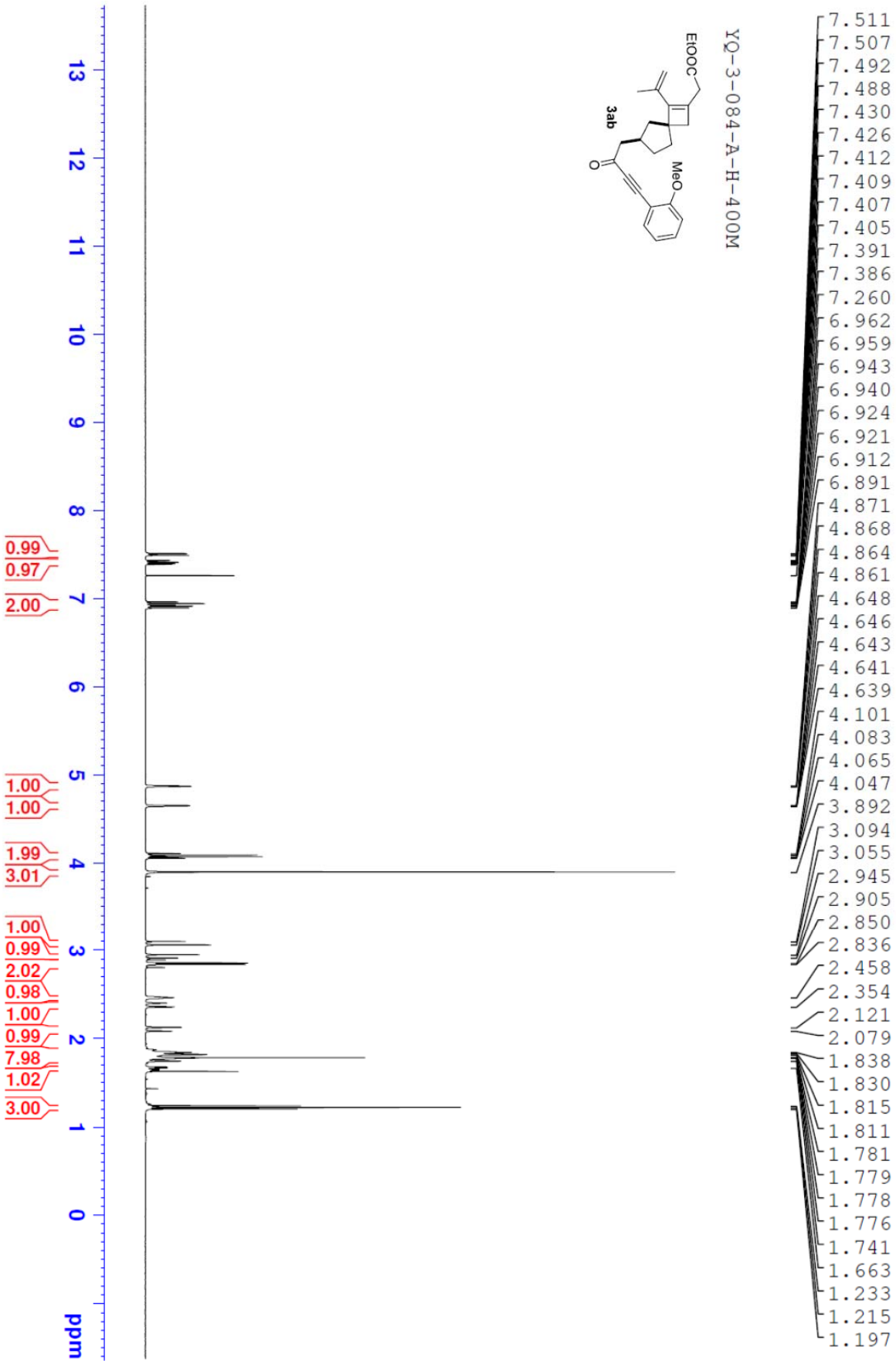

YQ-3-115-H-400M
EtO2C
1k
7.260
5.839
5.813
5.796
5.770
5.021
5.015
4.978
4.973
4.954
4.951
4.949
4.929
4.926
4.923
4.792
4.135
4.117
4.099
4.081
2.868
2.734
2.049
2.032
2.015
2.010
1.666
1.533
1.515
1.495
1.262
1.244
1.227
1.00
2.08
1.96
2.07
2.01
1.97
4.04
6.17
2.04
3.07
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

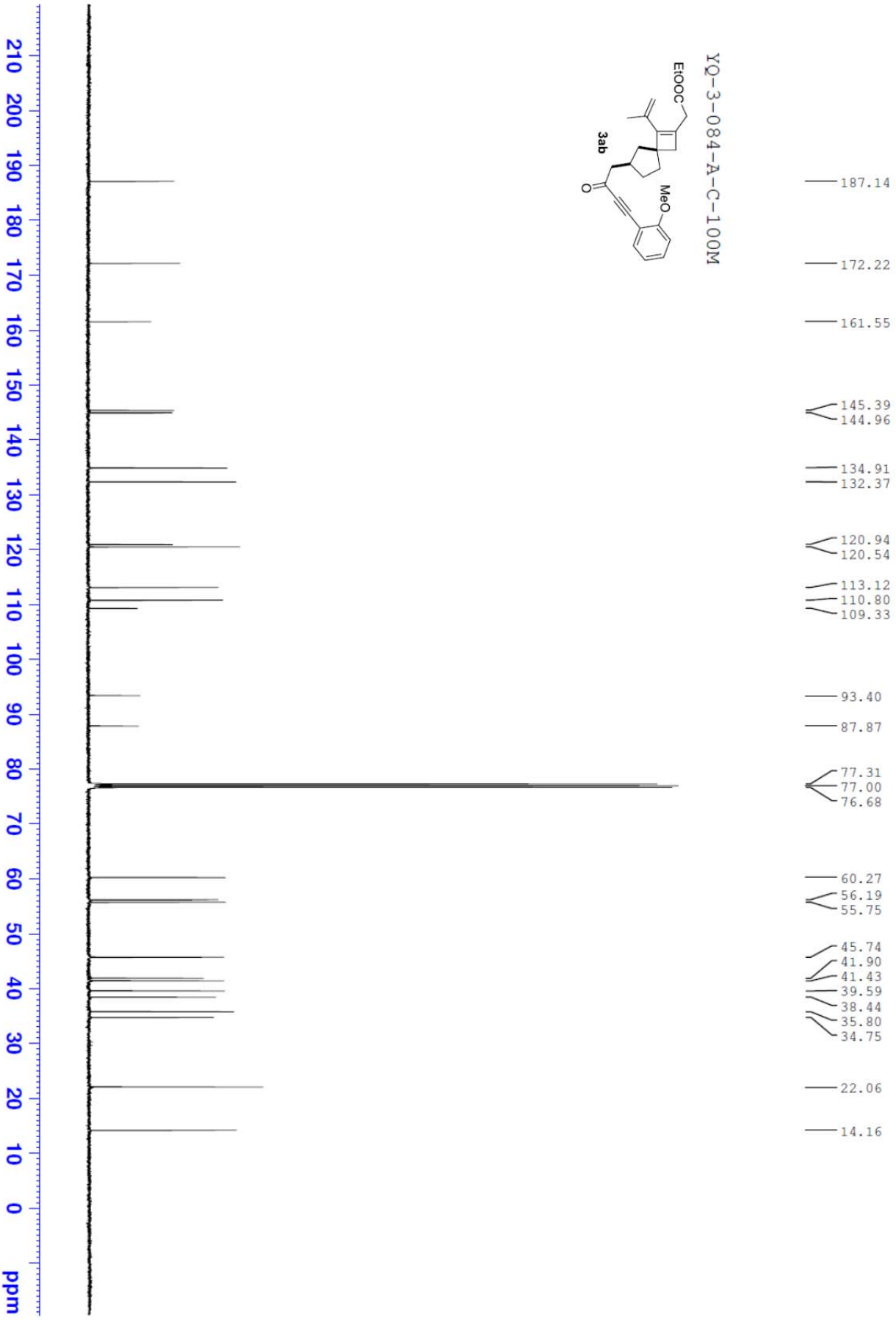

YQ-3-115-C-100M
EtO2C
1k
201.19
171.70
146.67
138.74
114.47
111.46
95.59
93.26
77.31
77.00
76.68
60.36
40.93
38.09
34.50
33.51
26.78
20.45
14.18
200
180
160
140
120
100
80
60
40
20
0
ppm

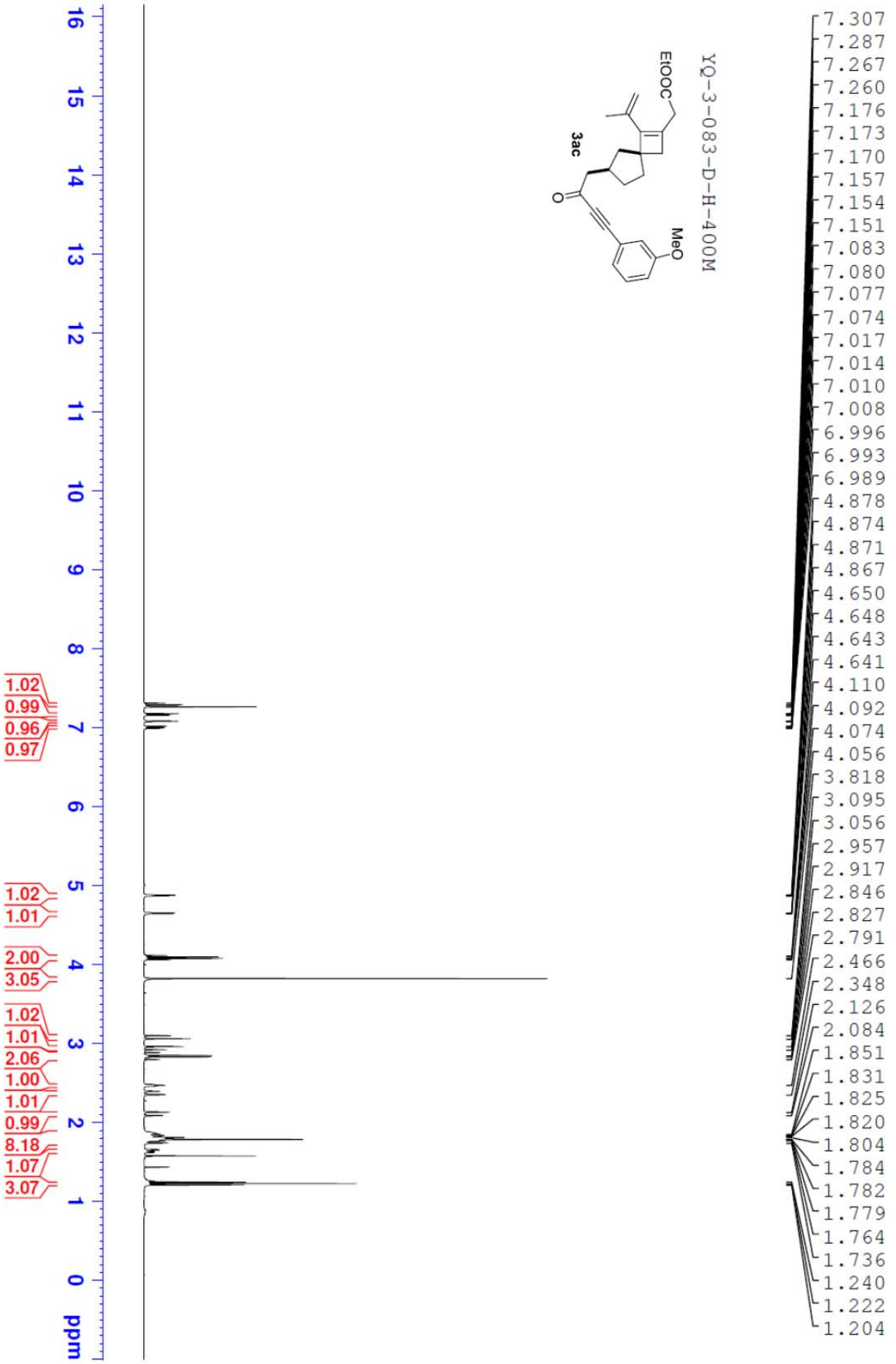

7.580
7.577
7.574
7.560
7.556
7.454
7.435
7.400
7.397
7.385
7.381
7.364
7.260
4.877
4.873
4.870
4.867
4.650
4.648
4.644
4.642
4.107
4.089
4.072
4.054
4.095
3.056
2.955
2.916
2.882
2.846
2.828
2.792
2.476
2.466
2.456
2.392
2.350
2.129
2.087
1.832
1.828
1.808
1.794
1.781
1.765
1.738
1.657
1.655
1.647
1.644
1.630
1.628
YQ-3-082-D-H-400M
EtOOC
Ph
3a
O
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
ppm
1.96
1.00
1.99
1.02
1.00
2.00
1.01
1.00
2.01
0.99
1.01
1.00
8.16
1.04
3.04

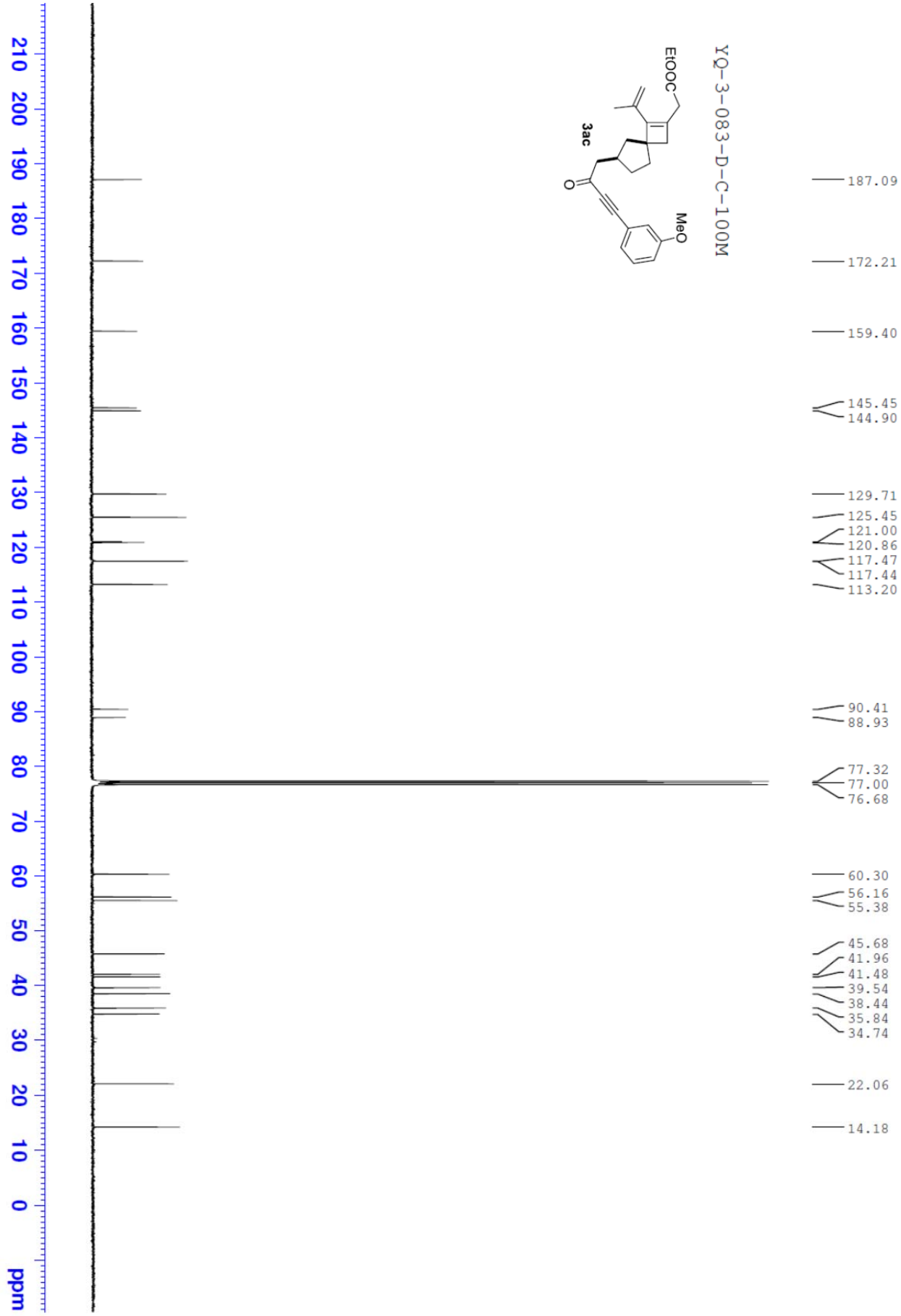

YQ-3-082-D-C-100M
EtOOC
3a
O
Ph
187.09
172.20
145.44
144.90
132.95
130.66
128.60
120.86
120.08
113.19
90.52
89.27
77.32
77.00
76.68
60.29
56.18
45.67
41.95
41.49
39.53
38.44
35.83
34.74
22.05
14.18
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

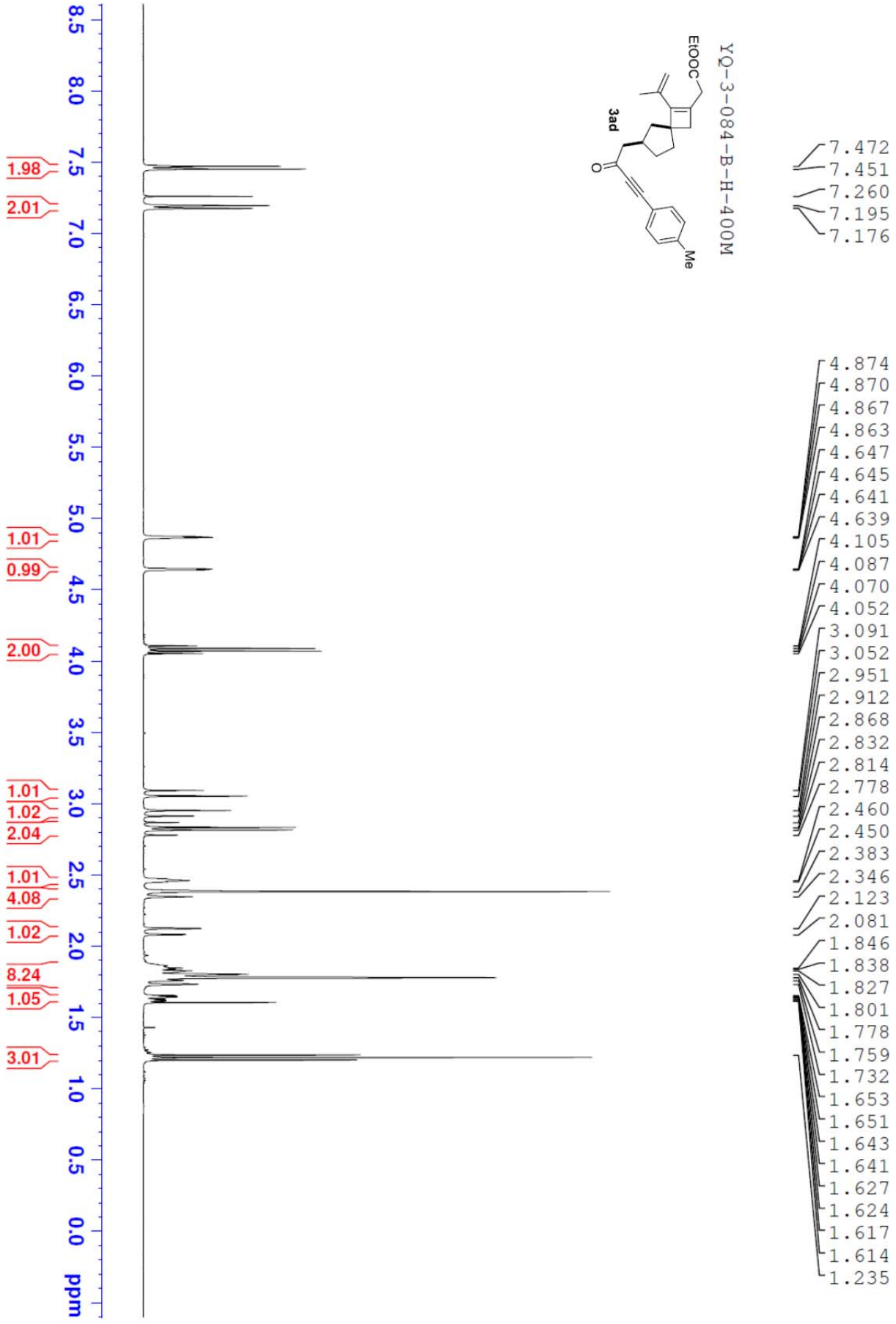

YQ-3-082-D-NOE
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
8
7
6
5
4
3
2
1
ppm


YQ-3-084-A-H-400M
EtOOC
3ab
MeO
O
7.511
7.507
7.492
7.488
7.430
7.426
7.412
7.409
7.407
7.405
7.391
7.386
7.260
6.962
6.959
6.943
6.940
6.924
6.921
6.912
6.891
4.871
4.868
4.864
4.861
4.648
4.646
4.643
4.641
4.639
4.101
4.083
4.065
4.047
3.892
3.094
3.055
2.945
2.905
2.850
2.836
2.458
2.354
2.121
2.079
1.838
1.830
1.815
1.811
1.781
1.779
1.778
1.776
1.741
1.663
1.233
1.215
1.197
0.99
0.97
2.00
1.00
1.00
1.99
3.01
1.00
0.99
2.02
0.98
1.00
0.99
7.98
1.02
3.00
ppm


YQ-3-084-A-C-100M
EtOOC
3ab
MeO
O
187.14
172.22
161.55
145.39
144.96
134.91
132.37
120.94
120.54
113.12
110.80
109.33
93.40
87.87
77.31
77.00
76.68
60.27
56.19
55.75
45.74
41.90
41.43
39.59
38.44
35.80
34.75
22.06
14.16
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


YQ-3-083-D-H-400M
3ac
EtOOC
MeO
O
7.307
7.287
7.267
7.260
7.176
7.173
7.170
7.157
7.154
7.151
7.083
7.080
7.077
7.074
7.017
7.014
7.010
7.008
6.996
6.993
6.989
4.878
4.874
4.871
4.867
4.650
4.648
4.643
4.641
4.110
4.092
4.074
4.056
3.818
3.095
3.056
2.957
2.917
2.846
2.827
2.791
2.466
2.348
2.126
2.084
1.851
1.831
1.825
1.820
1.804
1.784
1.782
1.779
1.764
1.736
1.240
1.222
1.204
1.02
0.99
0.96
0.97
1.02
1.01
2.00
3.05
1.02
1.01
2.06
1.00
1.01
0.99
8.18
1.07
3.07
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm


YQ-3-083-D-C-100M
3ac
EtOOC
O
MeO
187.09
172.21
159.40
145.45
144.90
129.71
125.45
121.00
120.86
117.47
117.44
113.20
90.41
88.93
77.32
77.00
76.68
60.30
56.16
55.38
45.68
41.96
41.48
39.54
38.44
35.84
34.74
22.06
14.18
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


YQ-3-084-B-H-400M
EtOOC
3ad
O
Me
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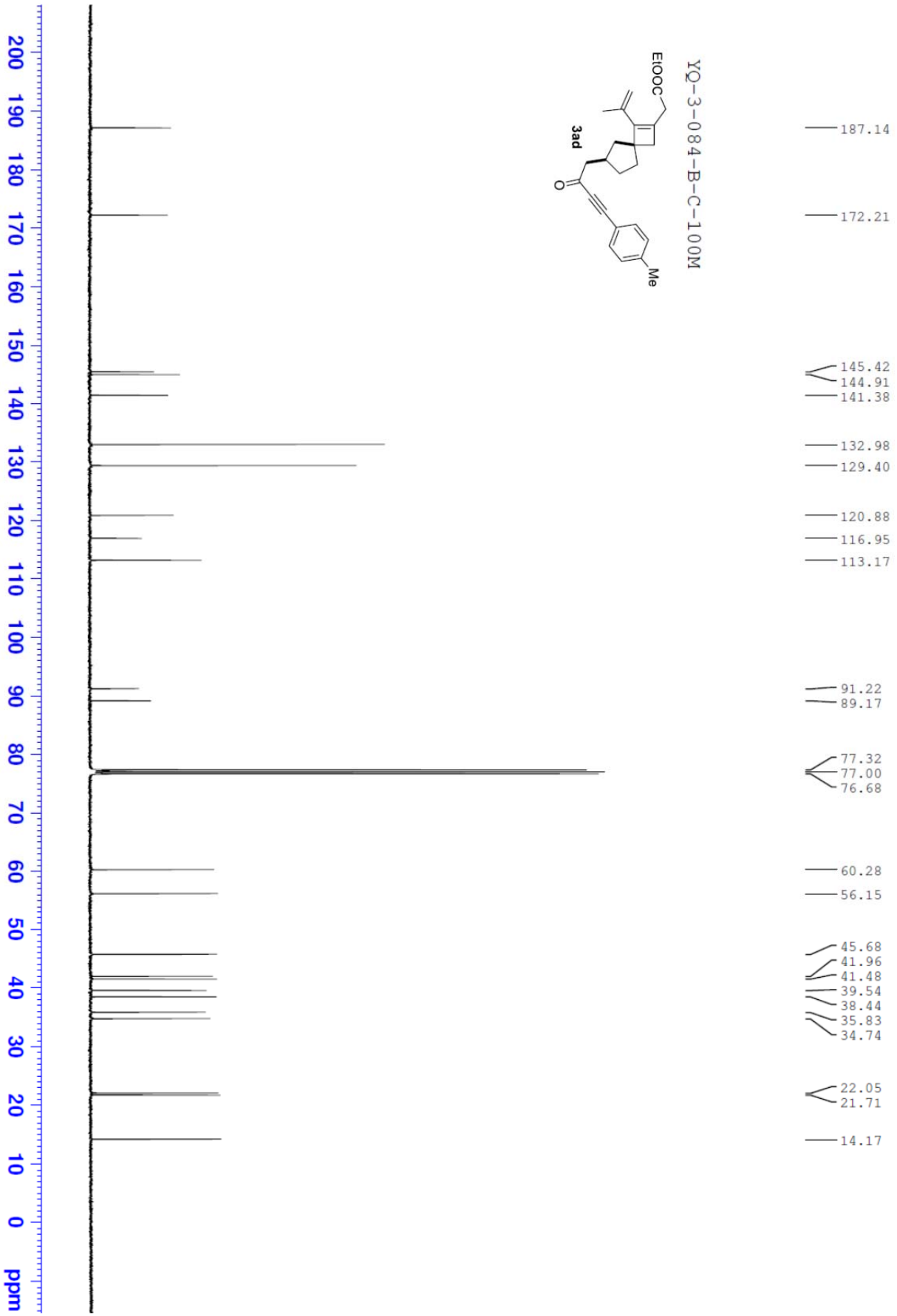
7.451
7.260
7.195
7.176
4.874
4.870
4.867
4.863
4.647
4.645
4.641
4.639
4.105
4.087
4.070
4.052
3.091
3.052
2.951
2.912
2.868
2.832
2.814
2.778
2.460
2.450
2.383
2.346
2.123
2.081
1.846
1.838
1.827
1.801
1.778
1.759
1.732
1.653
1.651
1.643
1.641
1.627
1.624
1.617
1.614
1.235
1.98
2.01
1.01
0.99
2.00
1.01
1.02
2.04
1.01
4.08
1.02
8.24
1.05
3.01
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
ppm


YQ-3-084-B-C-100M
EtOOC
3ad
O
Me
187.14
172.21
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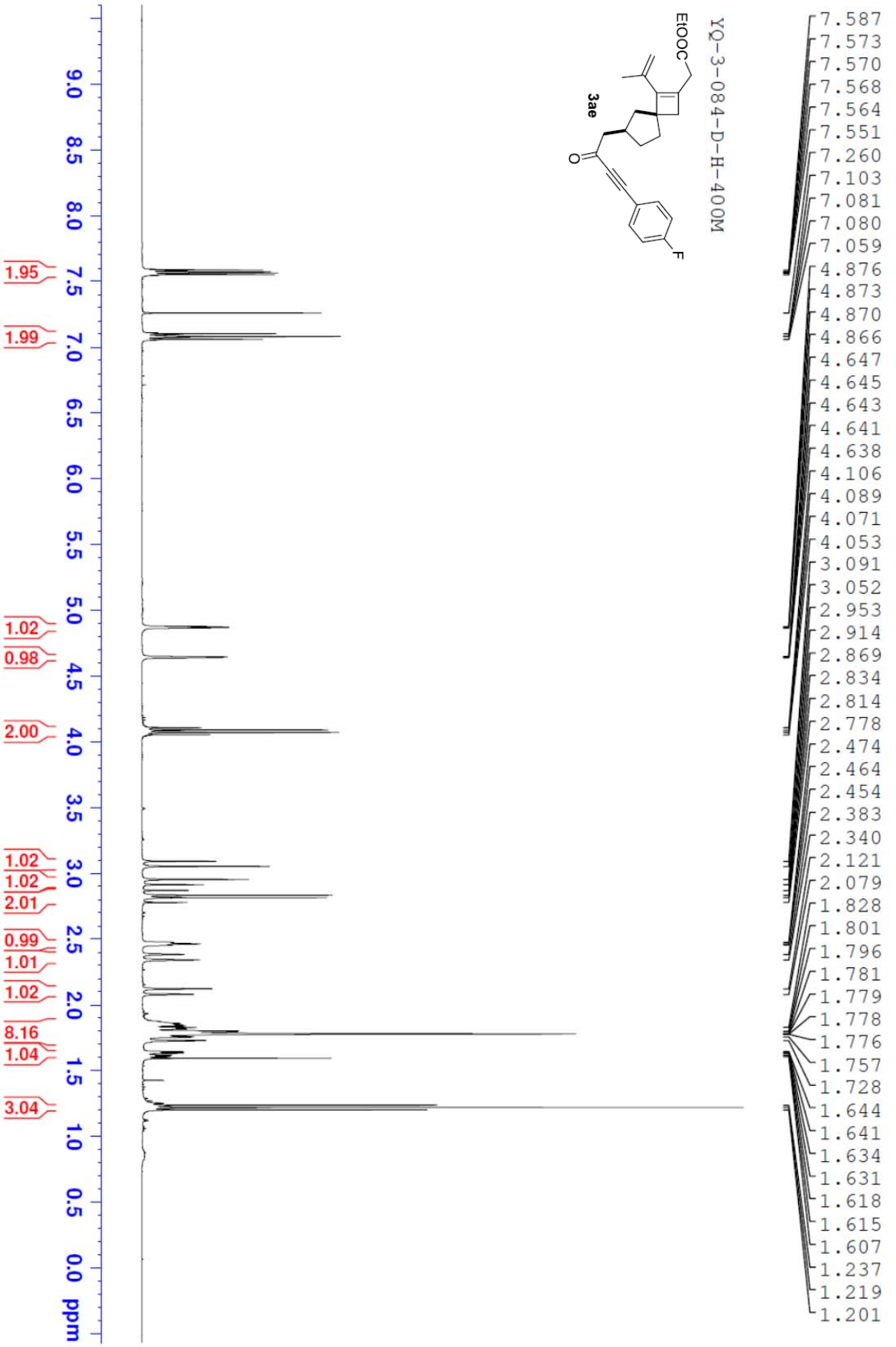
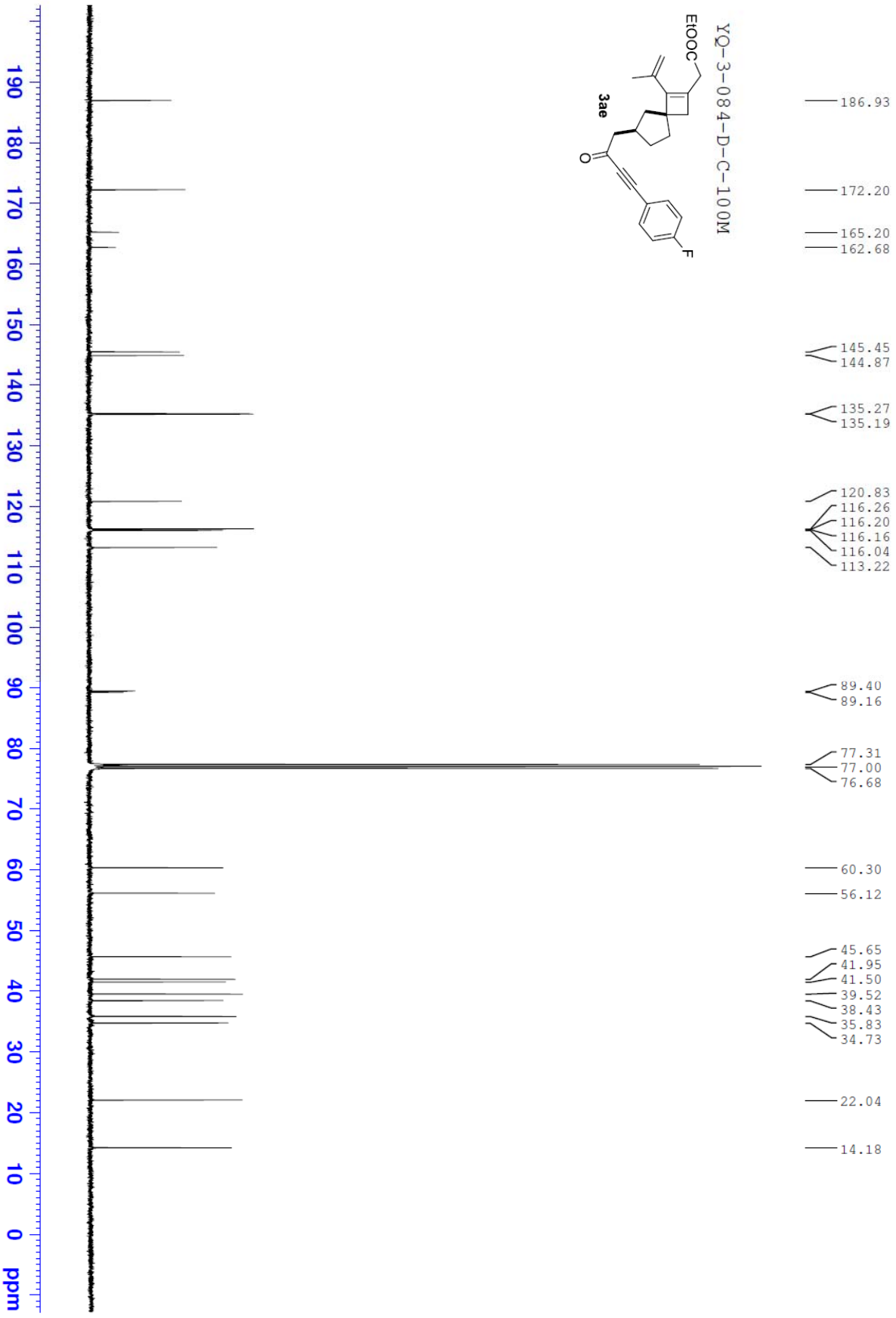
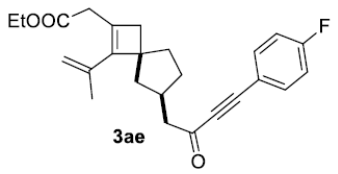
144.91
141.38
132.98
129.40
120.88
116.95
113.17
91.22
89.17
77.32
77.00
76.68
60.28
56.15
45.68
41.96
41.48
39.54
38.44
35.83
34.74
22.05
21.71
14.17
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


YQ-3-084-D-H-400M
EtOOC
3ae
O
F
7.587
7.573
7.570
7.568
7.564
7.551
7.260
7.103
7.081
7.080
7.059
4.876
4.873
4.870
4.866
4.647
4.645
4.643
4.641
4.638
4.106
4.089
4.071
4.053
3.091
3.052
2.953
2.914
2.869
2.834
2.814
2.778
2.474
2.464
2.454
2.383
2.340
2.121
2.079
1.828
1.801
1.796
1.781
1.779
1.778
1.776
1.757
1.728
1.644
1.641
1.634
1.631
1.618
1.615
1.607
1.237
1.219
1.201
1.95
1.99
1.02
0.98
2.00
1.02
1.02
2.01
0.99
1.01
1.02
8.16
1.04
3.04
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
ppm

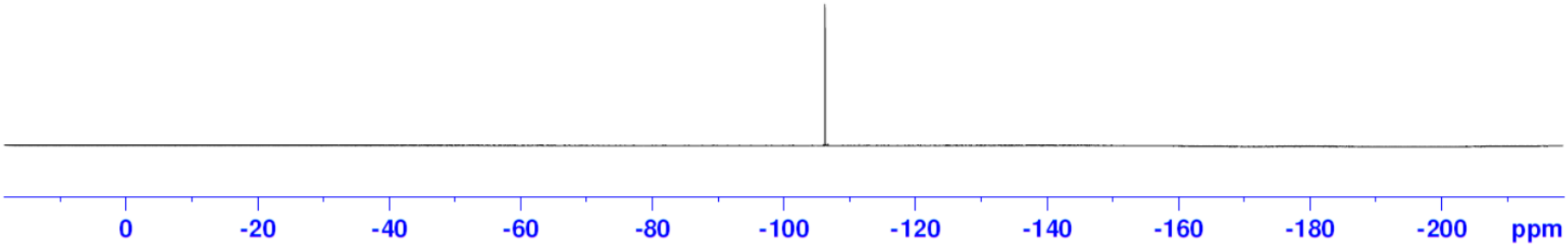

YQ-3-084-D-C-100M
EtOOC
3ae
O
F
186.93
172.20
165.20
162.68
145.45
144.87
135.27
135.19
120.83
116.26
116.20
116.16
116.04
113.22
89.40
89.16
77.31
77.00
76.68
60.30
56.12
45.65
41.95
41.50
39.52
38.43
35.83
34.73
22.04
14.18
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

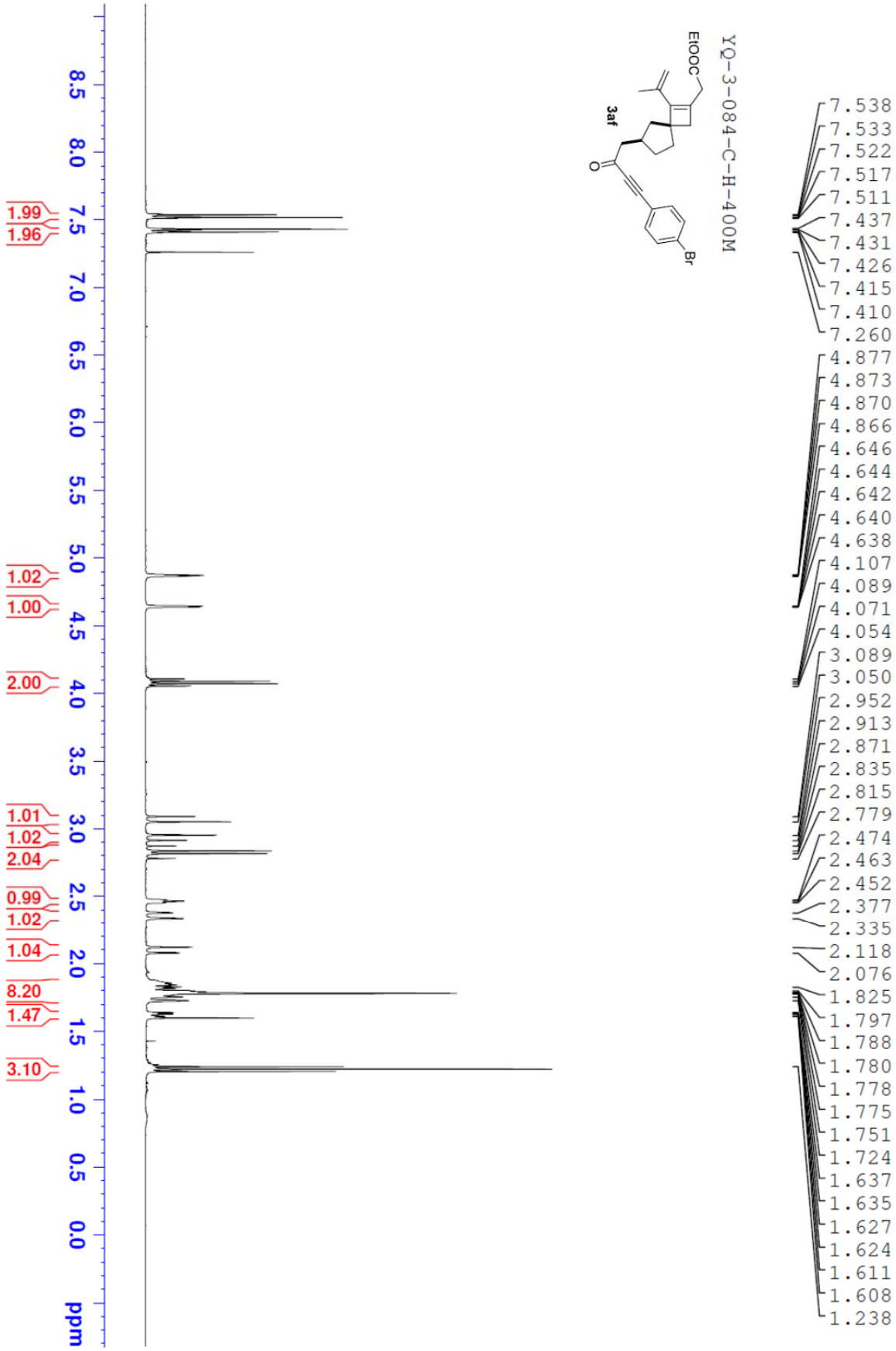
YQ-3-084-D-F

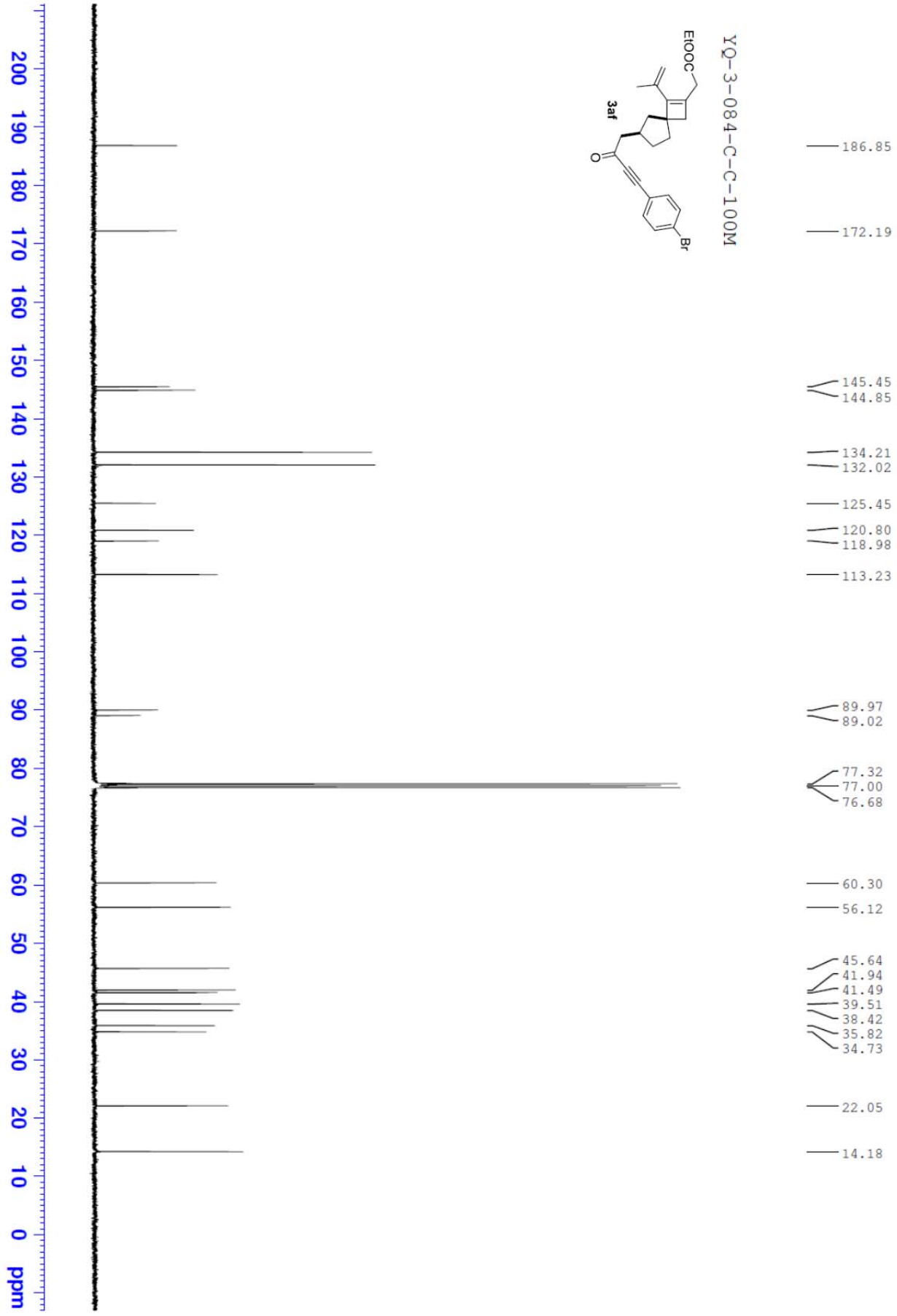


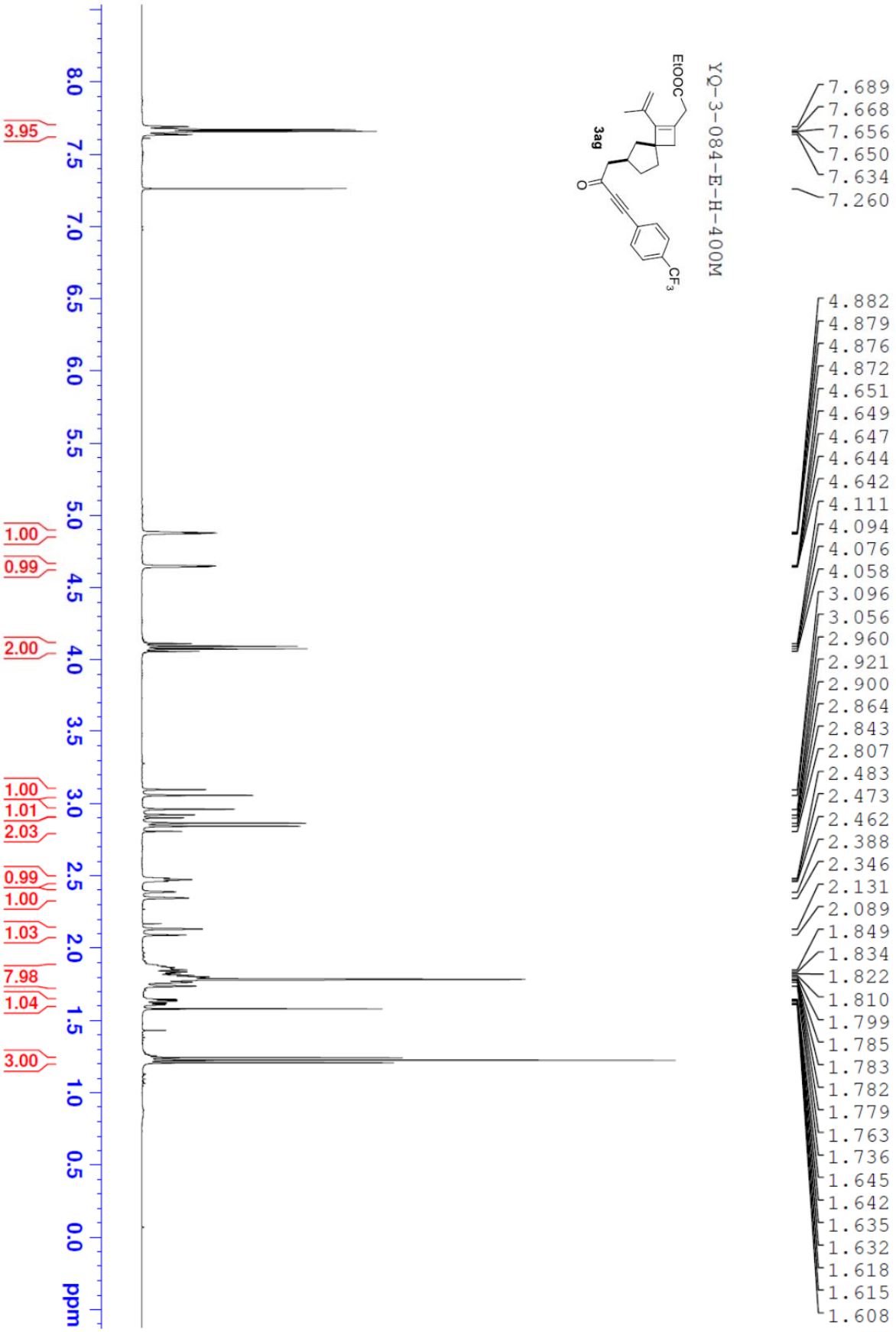
3ae

-106.31




YQ-3-084-C-H-400M
EtOOC
3af
O
Br
7.538
7.533
7.522
7.517
7.511
7.437
7.431
7.426
7.415
7.410
7.260
4.877
4.873
4.870
4.866
4.646
4.644
4.642
4.640
4.638
4.107
4.089
4.071
4.054
3.089
3.050
2.952
2.913
2.871
2.835
2.815
2.779
2.474
2.463
2.452
2.377
2.335
2.118
2.076
1.825
1.797
1.788
1.780
1.778
1.775
1.751
1.724
1.637
1.635
1.627
1.624
1.611
1.608
1.238
1.99
1.96
1.02
1.00
2.00
1.01
1.02
2.04
0.99
1.02
1.04
8.20
1.47
3.10
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
ppm


YQ-3-084-C-C-100M
EtOOC
3af
O
Br
186.85
172.19
145.45
144.85
134.21
132.02
125.45
120.80
118.98
113.23
89.97
89.02
77.32
77.00
76.68
60.30
56.12
45.64
41.94
41.49
39.51
38.42
35.82
34.73
22.05
14.18
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


YQ-3-084-E-H-400M
3ag
EtOOC
CF3
O
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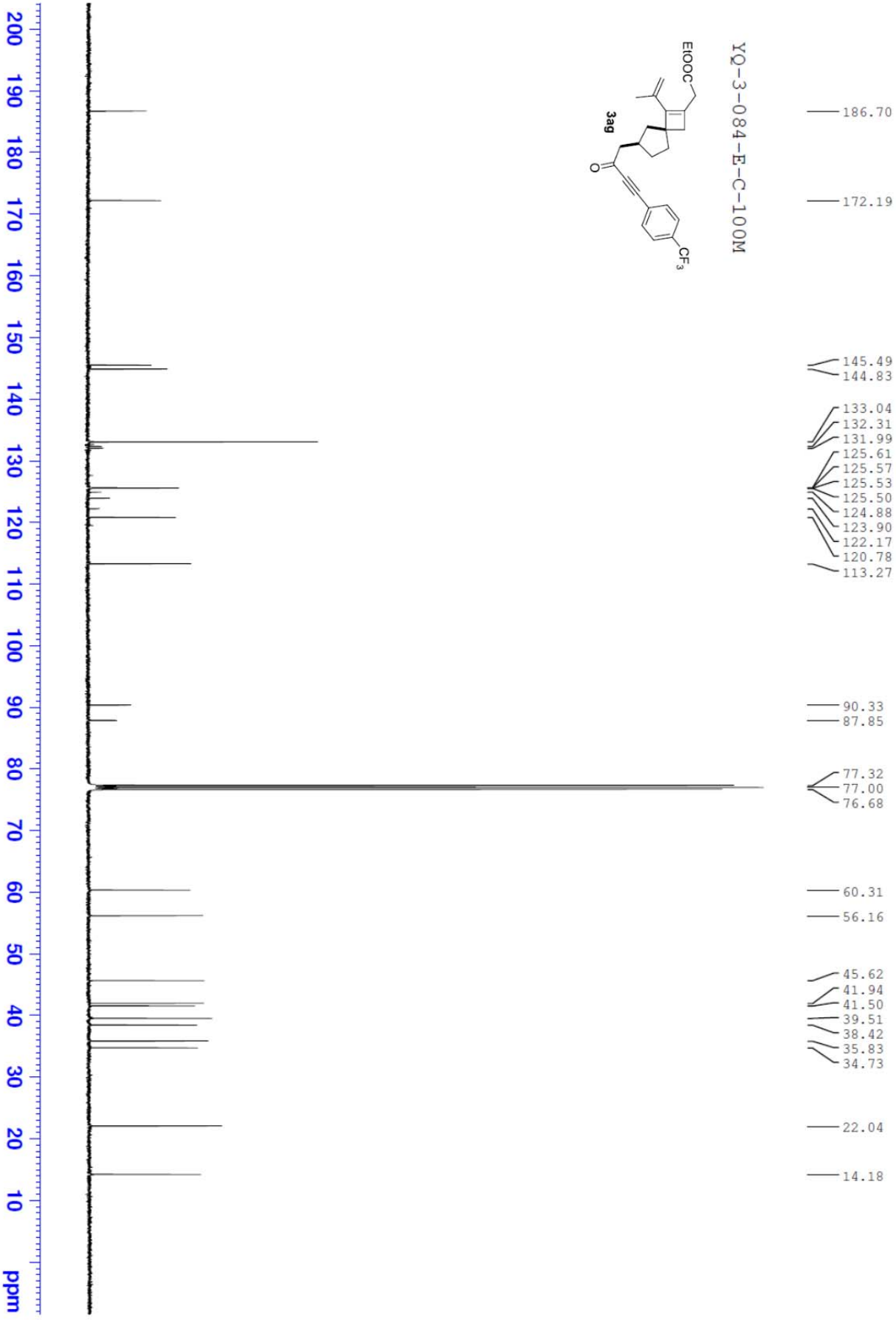
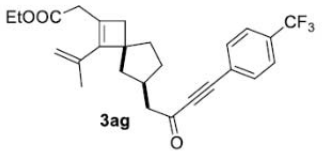
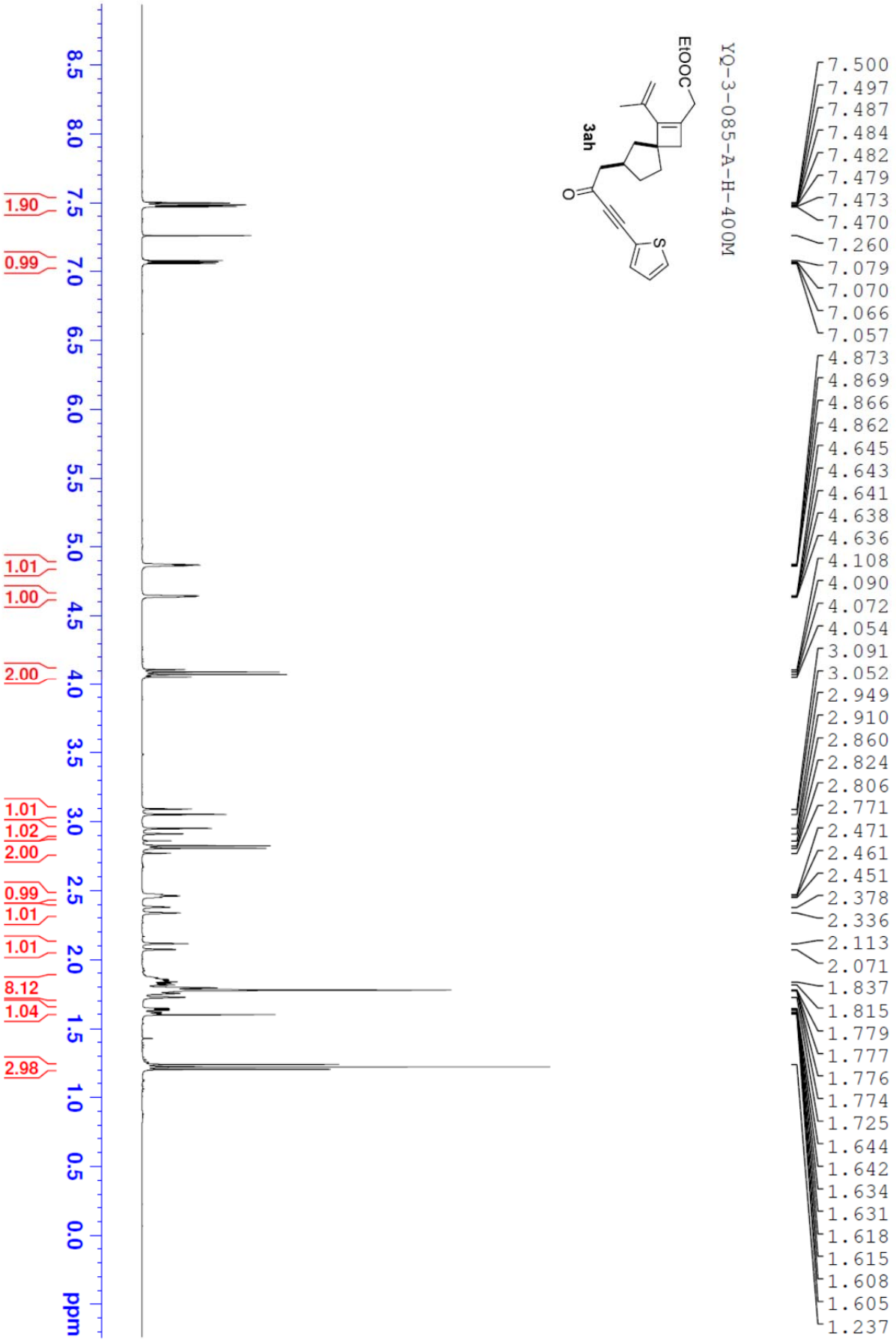
7.668
7.656
7.650
7.634
7.260
4.882
4.879
4.876
4.872
4.651
4.649
4.647
4.644
4.642
4.111
4.094
4.076
4.058
3.096
3.056
2.960
2.921
2.900
2.864
2.843
2.807
2.483
2.473
2.462
2.388
2.346
2.131
2.089
1.849
1.834
1.822
1.810
1.799
1.785
1.783
1.782
1.779
1.763
1.736
1.645
1.642
1.635
1.632
1.618
1.615
1.608
3.95
1.00
0.99
2.00
1.00
1.01
2.03
0.99
1.00
1.03
7.98
1.04
3.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

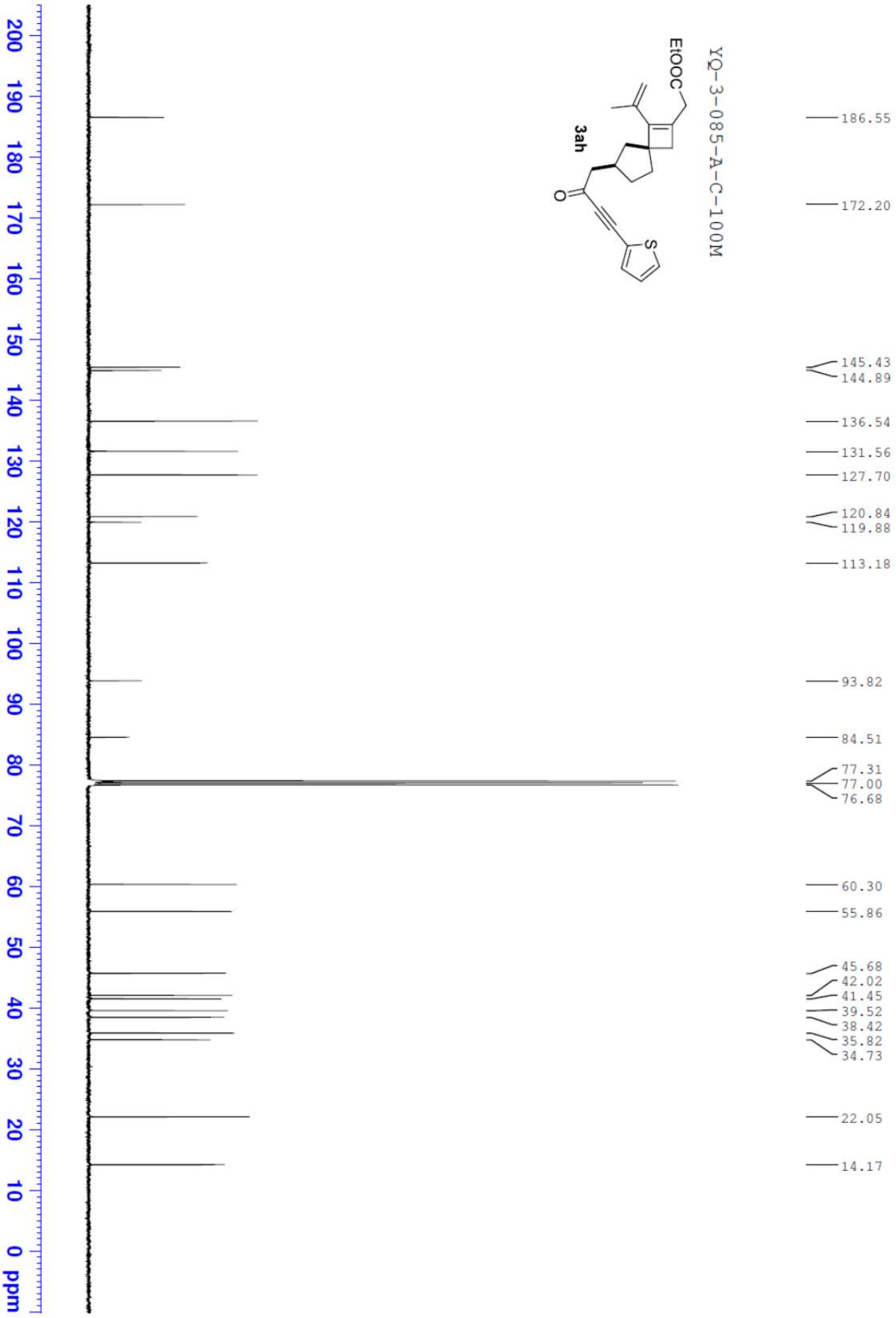

YQ-3-084-E-C-100M
EtOOC
3ag
O
CF3
186.70
172.19
145.49
144.83
133.04
132.31
131.99
125.61
125.57
125.53
125.50
124.88
123.90
122.17
120.78
113.27
90.33
87.85
77.32
77.00
76.68
60.31
56.16
45.62
41.94
41.50
39.51
38.42
35.83
34.73
22.04
14.18
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

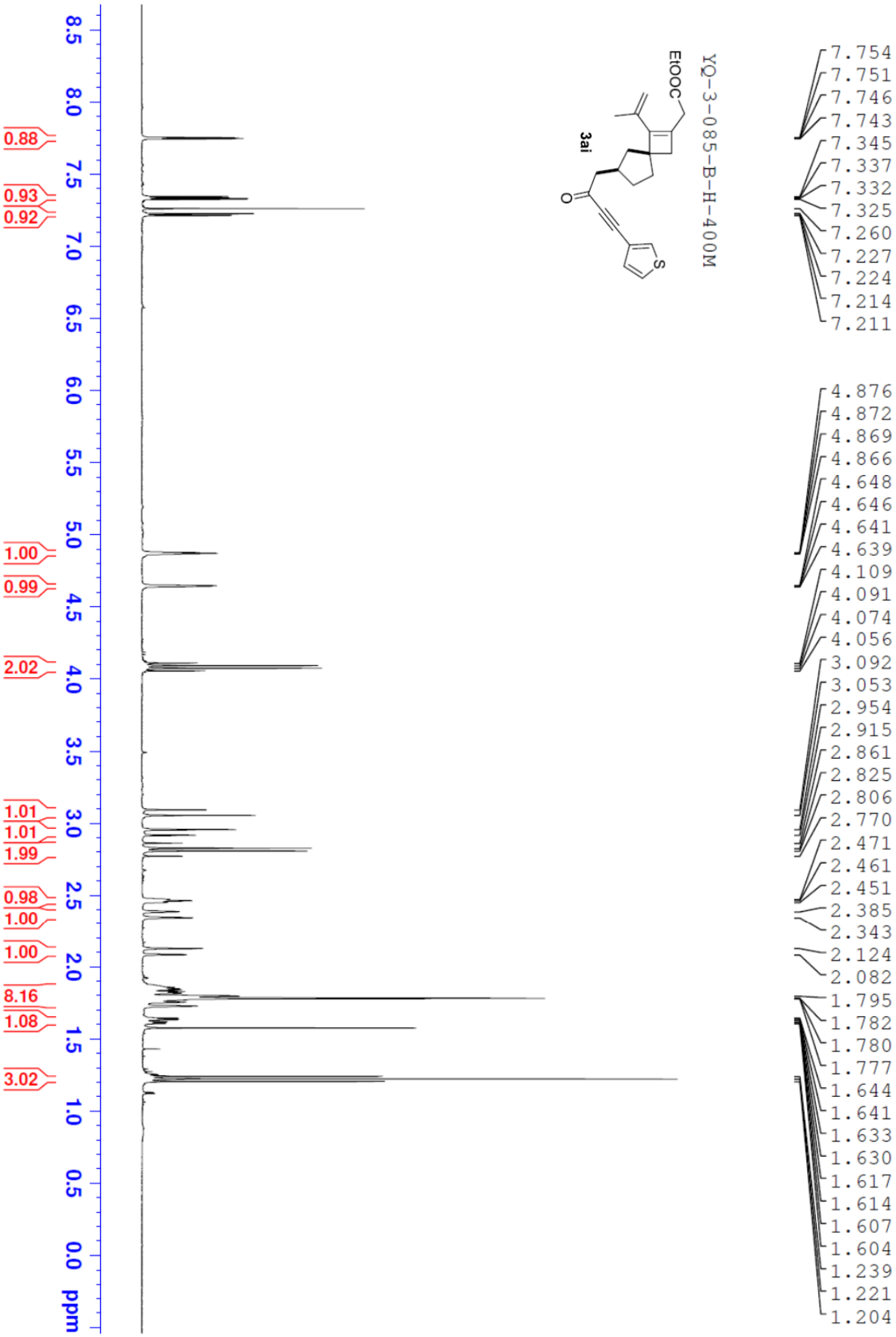
YQ-3-084-E-F

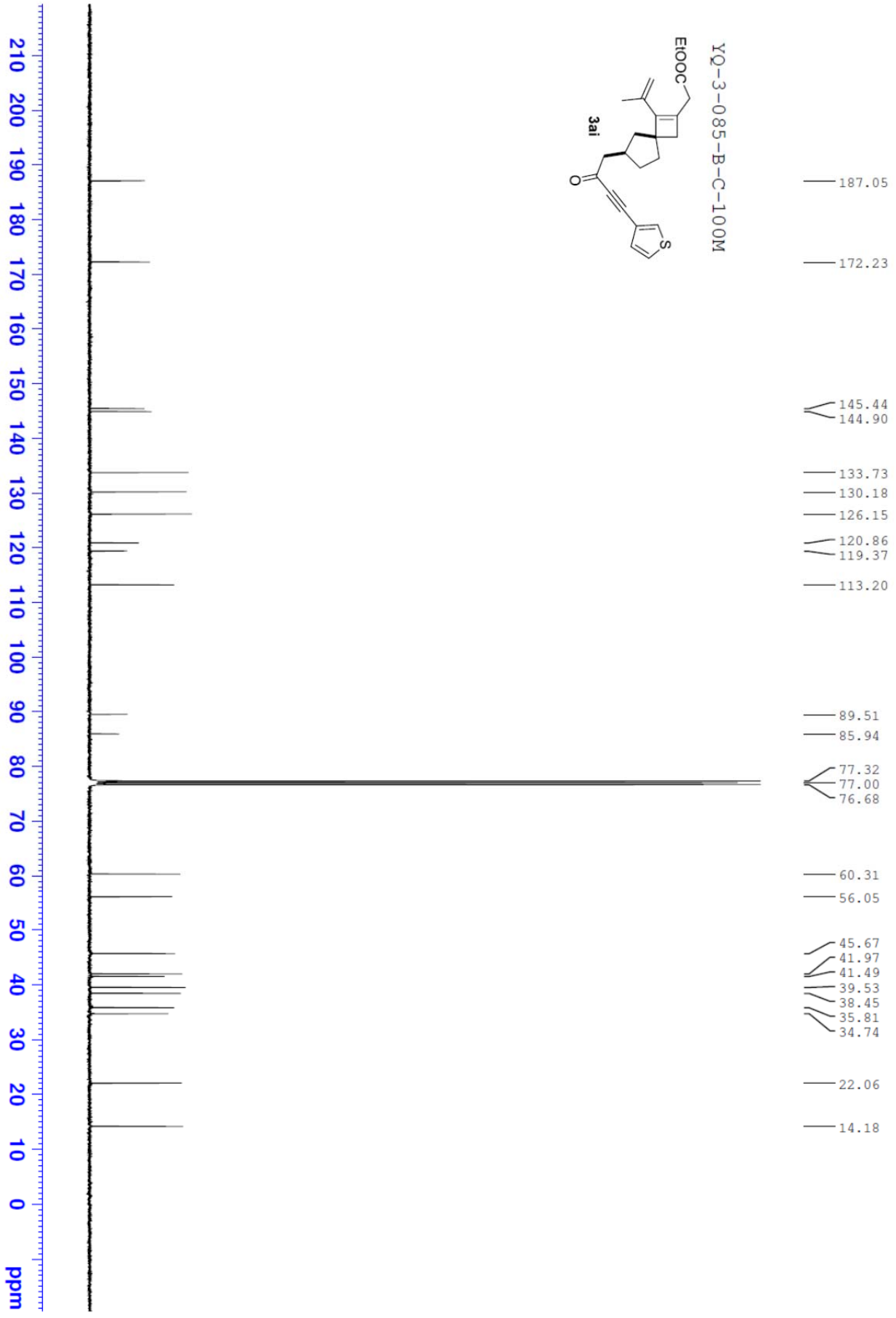


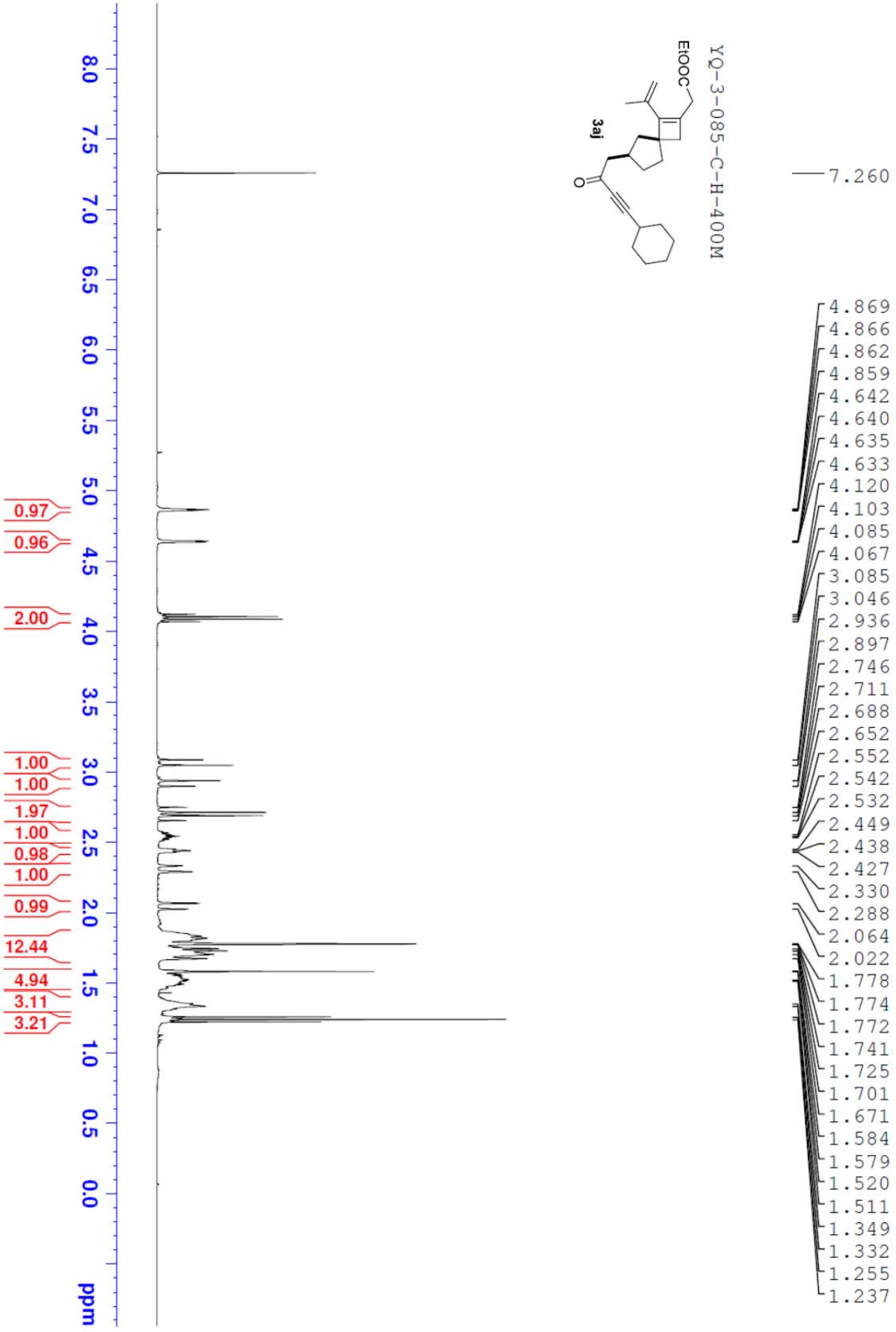
3ag

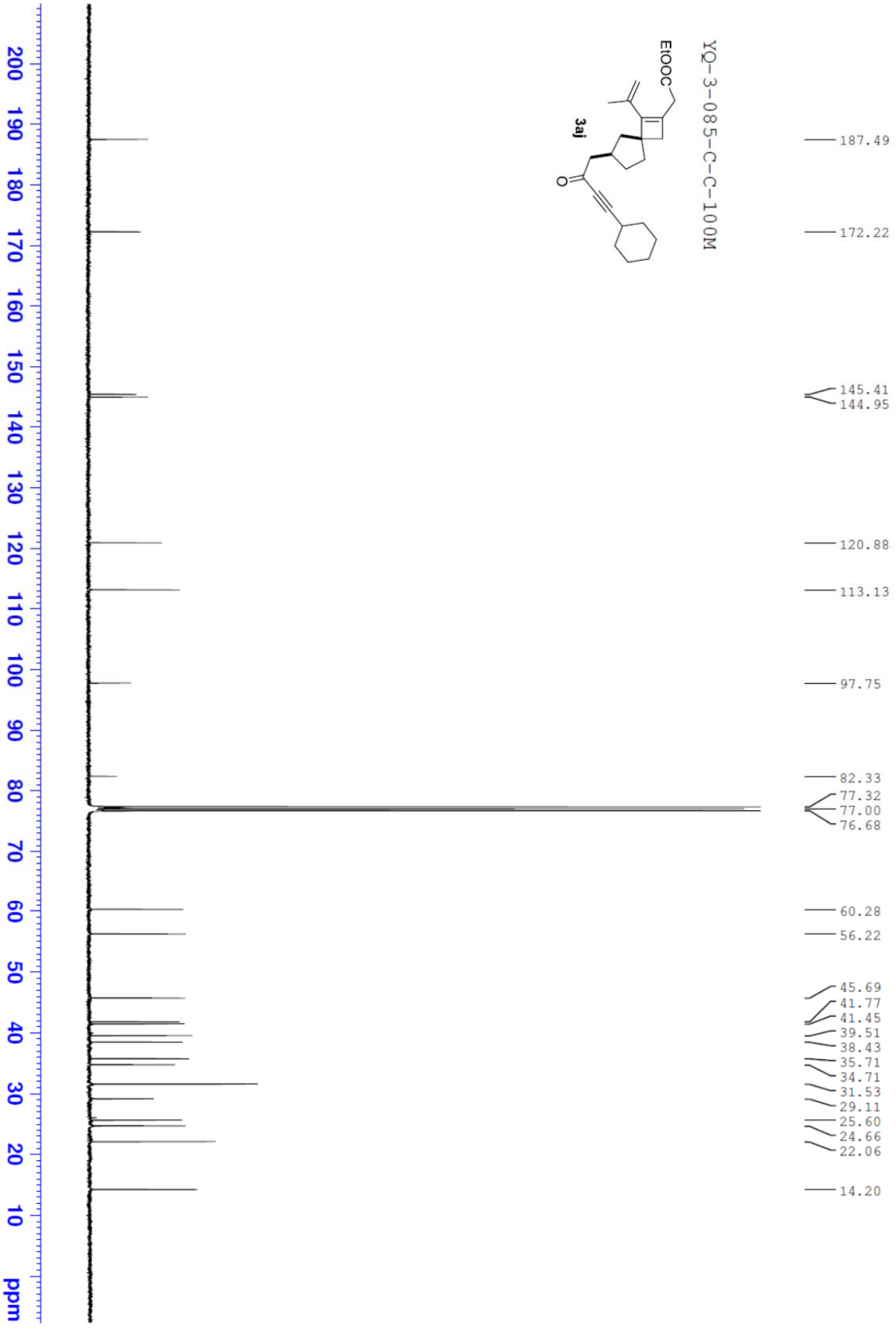

YQ-3-085-A-H-400M
3ah
EtOOC
O
S
7.500
7.497
7.487
7.484
7.482
7.479
7.473
7.470
7.260
7.079
7.070
7.066
7.057
4.873
4.869
4.866
4.862
4.645
4.643
4.641
4.638
4.636
4.108
4.090
4.072
4.054
3.091
3.052
2.949
2.910
2.860
2.824
2.806
2.771
2.471
2.461
2.451
2.378
2.336
2.113
2.071
1.837
1.815
1.779
1.777
1.776
1.774
1.725
1.644
1.642
1.634
1.631
1.618
1.615
1.608
1.605
1.237
1.90
0.99
1.01
1.00
2.00
1.01
1.02
2.00
0.99
1.01
1.01
8.12
1.04
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
ppm


YQ-3-085-A-C-100M
EtOOC
3ah
O
S
186.55
172.20
145.43
144.89
136.54
131.56
127.70
120.84
119.88
113.18
93.82
84.51
77.31
77.00
76.68
60.30
55.86
45.68
42.02
41.45
39.52
38.42
35.82
34.73
22.05
14.17
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm


YQ-3-085-B-H-400M
3ai
EtOOC
O
S
7.754
7.751
7.746
7.743
7.345
7.337
7.332
7.325
7.260
7.227
7.224
7.214
7.211
4.876
4.872
4.869
4.866
4.648
4.646
4.641
4.639
4.109
4.091
4.074
4.056
3.092
3.053
2.954
2.915
2.861
2.825
2.806
2.770
2.471
2.461
2.451
2.385
2.343
2.124
2.082
1.795
1.782
1.780
1.777
1.644
1.641
1.633
1.630
1.617
1.614
1.607
1.604
1.239
1.221
1.204
0.88
0.93
0.92
1.00
0.99
2.02
1.01
1.01
1.99
0.98
1.00
1.00
8.16
1.08
3.02
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
ppm


YQ-3-085-B-C-100M
3ai
EtOOC
O
S
187.05
172.23
145.44
144.90
133.73
130.18
126.15
120.86
119.37
113.20
89.51
85.94
77.32
77.00
76.68
60.31
56.05
45.67
41.97
41.49
39.53
38.45
35.81
34.74
22.06
14.18
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


YQ-3-085-C-H-400M
EtOOC
3aj
O
7.260
4.869
4.866
4.862
4.859
4.642
4.640
4.635
4.633
4.120
4.103
4.085
4.067
3.085
3.046
2.936
2.897
2.746
2.711
2.688
2.652
2.552
2.542
2.532
2.449
2.438
2.427
2.330
2.288
2.064
2.022
1.778
1.774
1.772
1.741
1.725
1.701
1.671
1.584
1.579
1.520
1.511
1.349
1.332
1.255
1.237
0.97
0.96
2.00
1.00
1.00
1.97
1.00
0.98
1.00
0.99
12.44
4.94
3.11
3.21
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm


YQ-3-085-C-C-100M
EtOOC
3aj
O
187.49
172.22
145.41
144.95
120.88
113.13
97.75
82.33
77.32
77.00
76.68
60.28
56.22
45.69
41.77
41.45
39.51
38.43
35.71
34.71
31.53
29.11
25.60
24.66
22.06
14.20
200
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150
140
130
120
110
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90
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70
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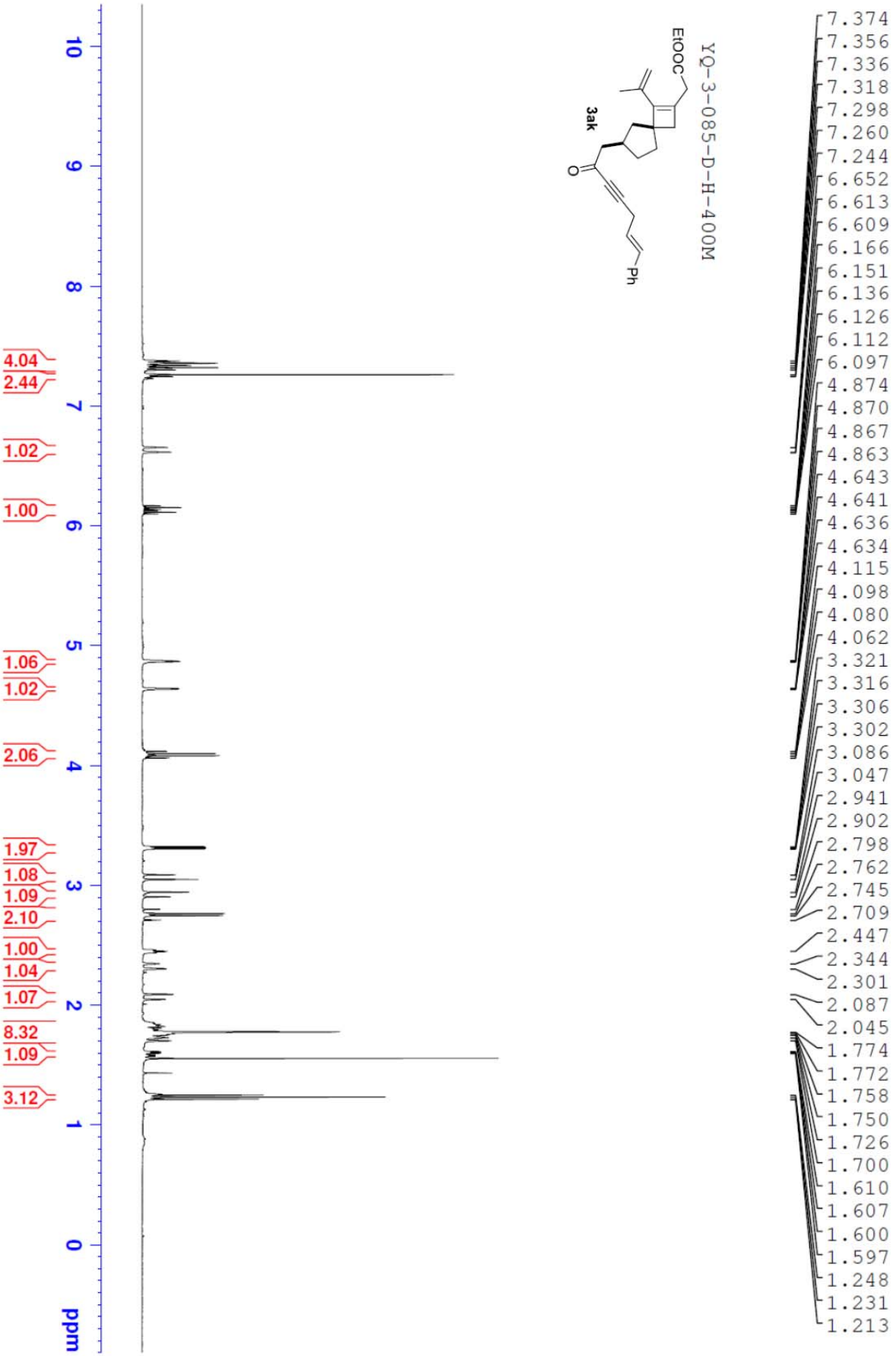
30
20
10
ppm


YQ-3-085-D-H-400M
3ak
EtOOC
O
Ph
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7.298
7.260
7.244
6.652
6.613
6.609
6.166
6.151
6.136
6.126
6.112
6.097
4.874
4.870
4.867
4.863
4.643
4.641
4.636
4.634
4.115
4.098
4.080
4.062
3.321
3.316
3.306
3.302
3.086
3.047
2.941
2.902
2.798
2.762
2.745
2.709
2.447
2.344
2.301
2.087
2.045
1.774
1.772
1.758
1.750
1.726
1.700
1.610
1.607
1.600
1.597
1.248
1.231
1.213
4.04
2.44
1.02
1.00
1.06
1.02
2.06
1.97
1.08
1.09
2.10
1.00
1.04
1.07
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1.09
3.12
10
9
8
7
6
5
4
3
2
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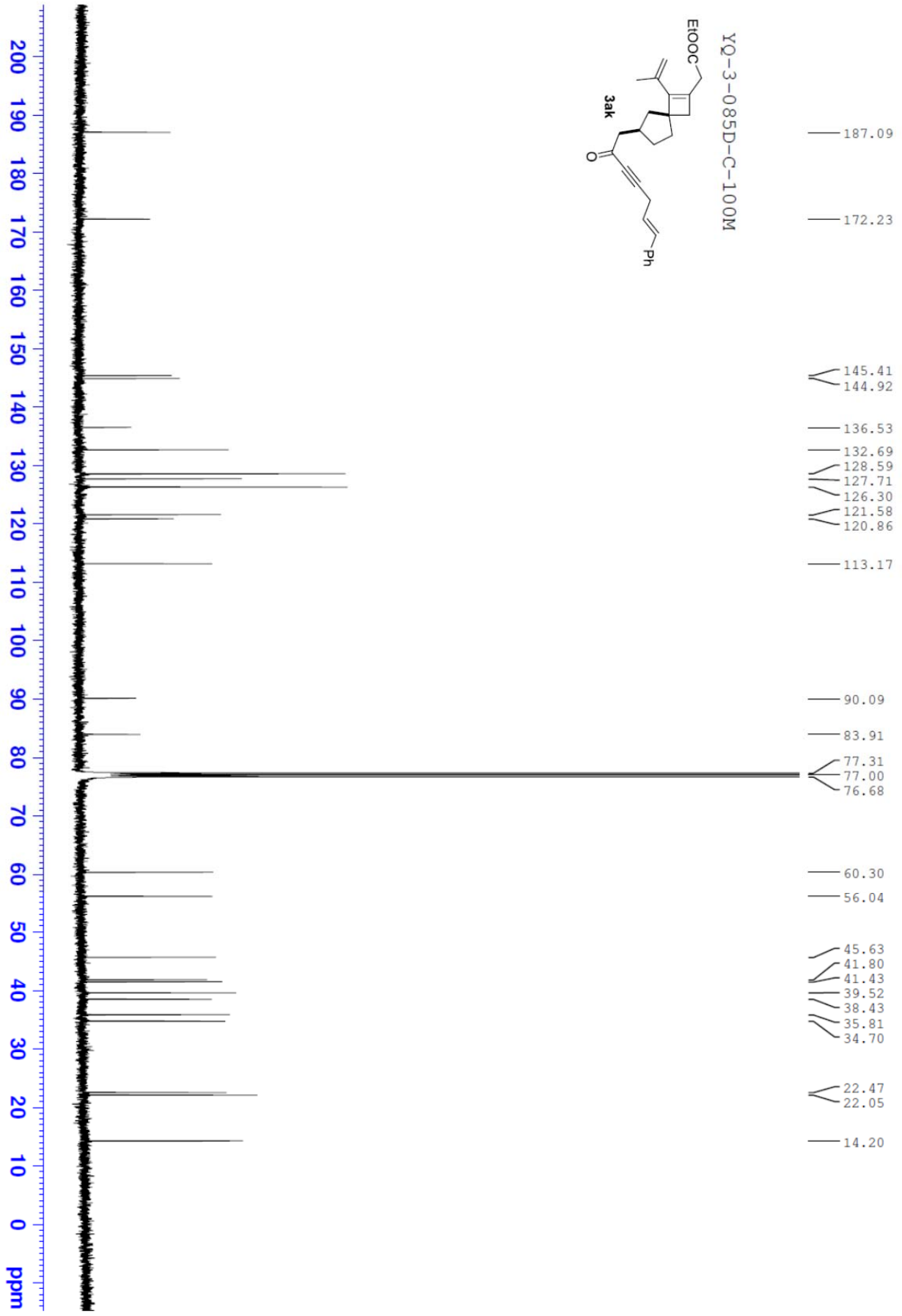
0
ppm


YQ-3-085D-C-100M
EtOOC
3ak
O
Ph
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172.23
145.41
144.92
136.53
132.69
128.59
127.71
126.30
121.58
120.86
113.17
90.09
83.91
77.31
77.00
76.68
60.30
56.04
45.63
41.80
41.43
39.52
38.43
35.81
34.70
22.47
22.05
14.20
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140
130
120
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80
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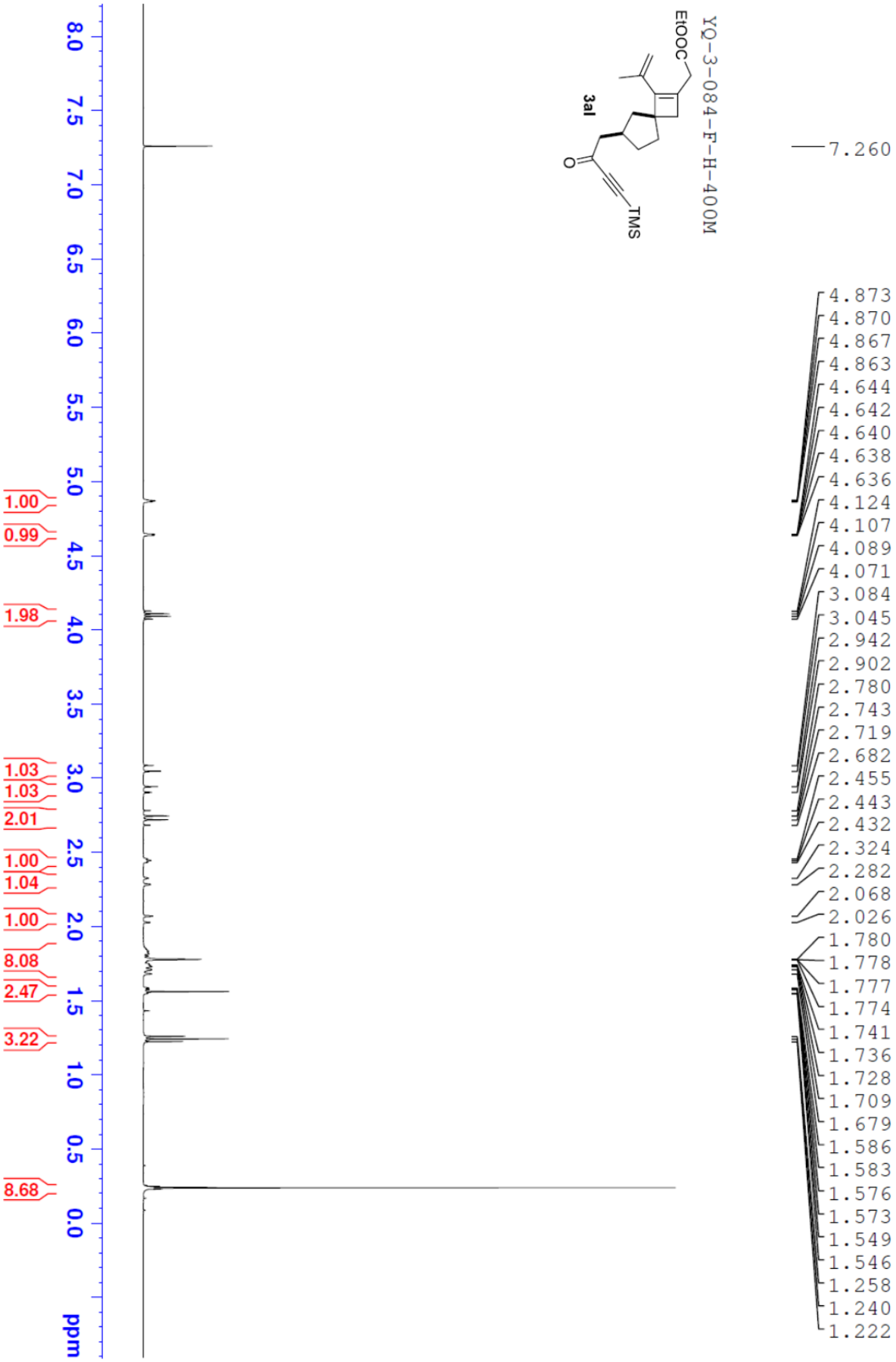
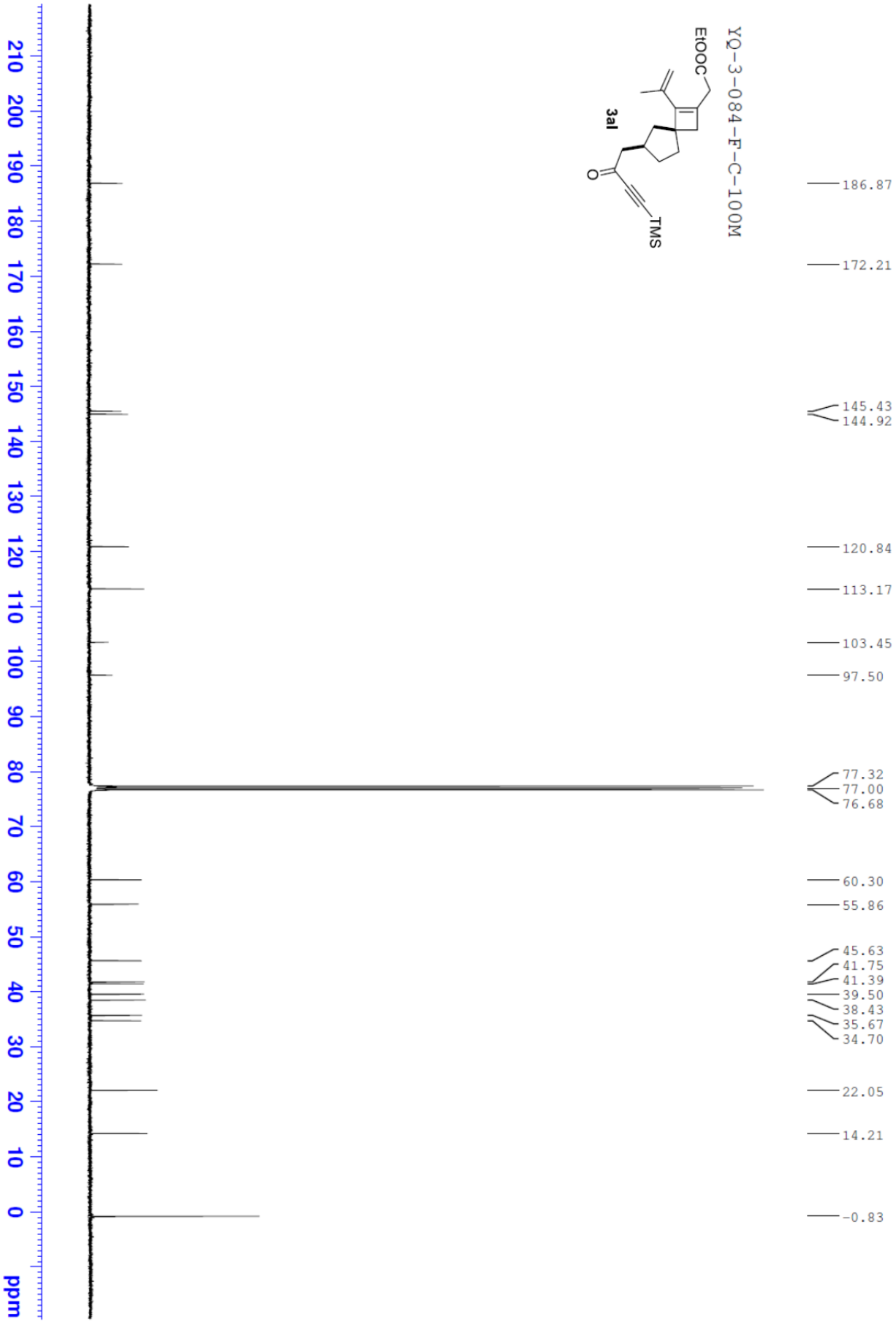

YQ-3-084-F-H-400M
EtOOC
3al
O
TMS
7.260
4.873
4.870
4.867
4.863
4.644
4.642
4.640
4.638
4.636
4.124
4.107
4.089
4.071
3.084
3.045
2.942
2.902
2.780
2.743
2.719
2.682
2.455
2.443
2.432
2.324
2.282
2.068
2.026
1.780
1.778
1.777
1.774
1.741
1.736
1.728
1.709
1.679
1.586
1.583
1.576
1.573
1.549
1.546
1.258
1.240
1.222
1.00
0.99
1.98
1.03
1.03
2.01
1.00
1.04
1.00
8.08
2.47
3.22
8.68
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
ppm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3al
O
TMS
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103.45
97.50
77.32
77.00
76.68
60.30
55.86
45.63
41.75
41.39
39.50
38.43
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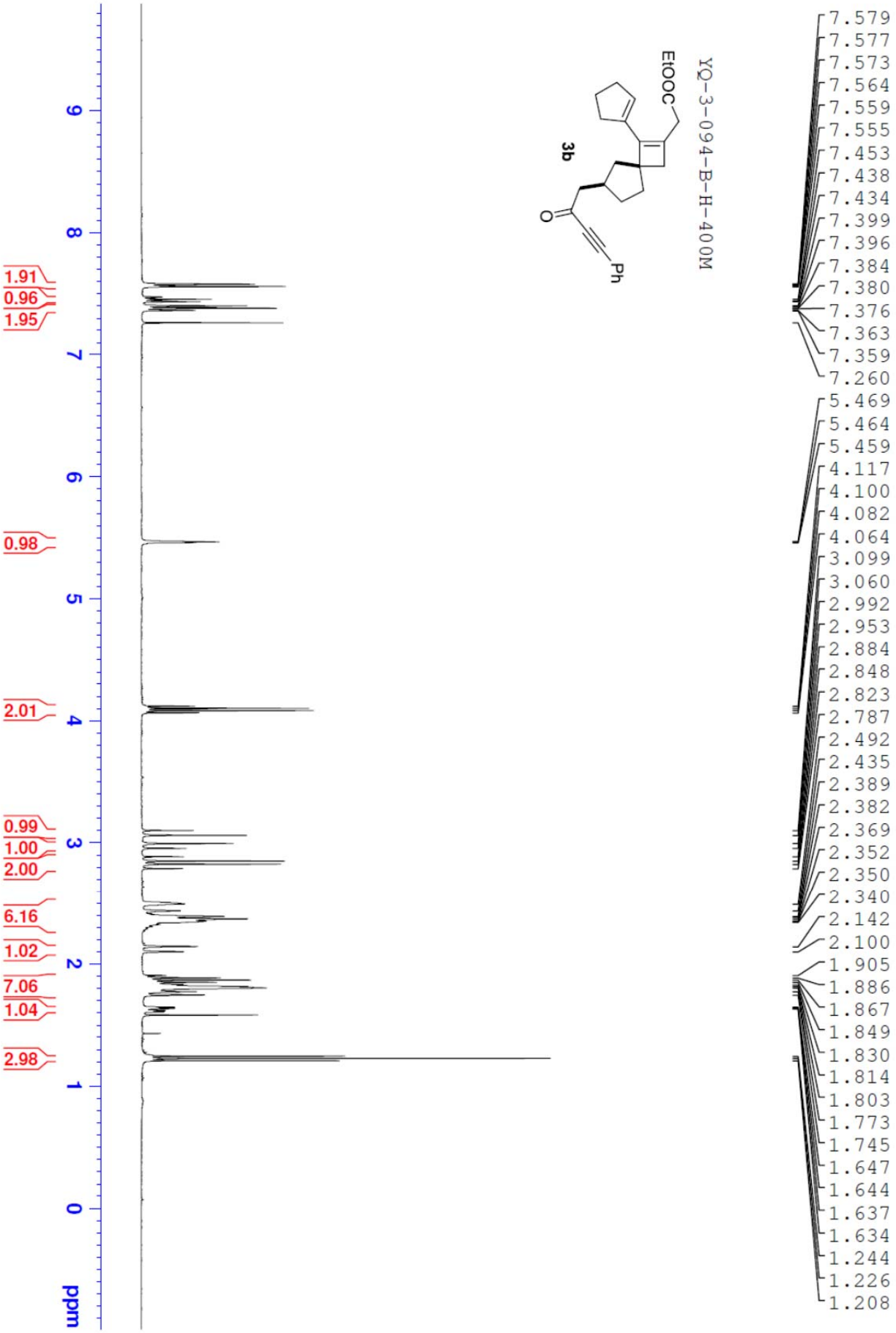
34.70
22.05
14.21
-0.83
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


YQ-3-094-B-H-400M
EtOOC
3b
O
Ph
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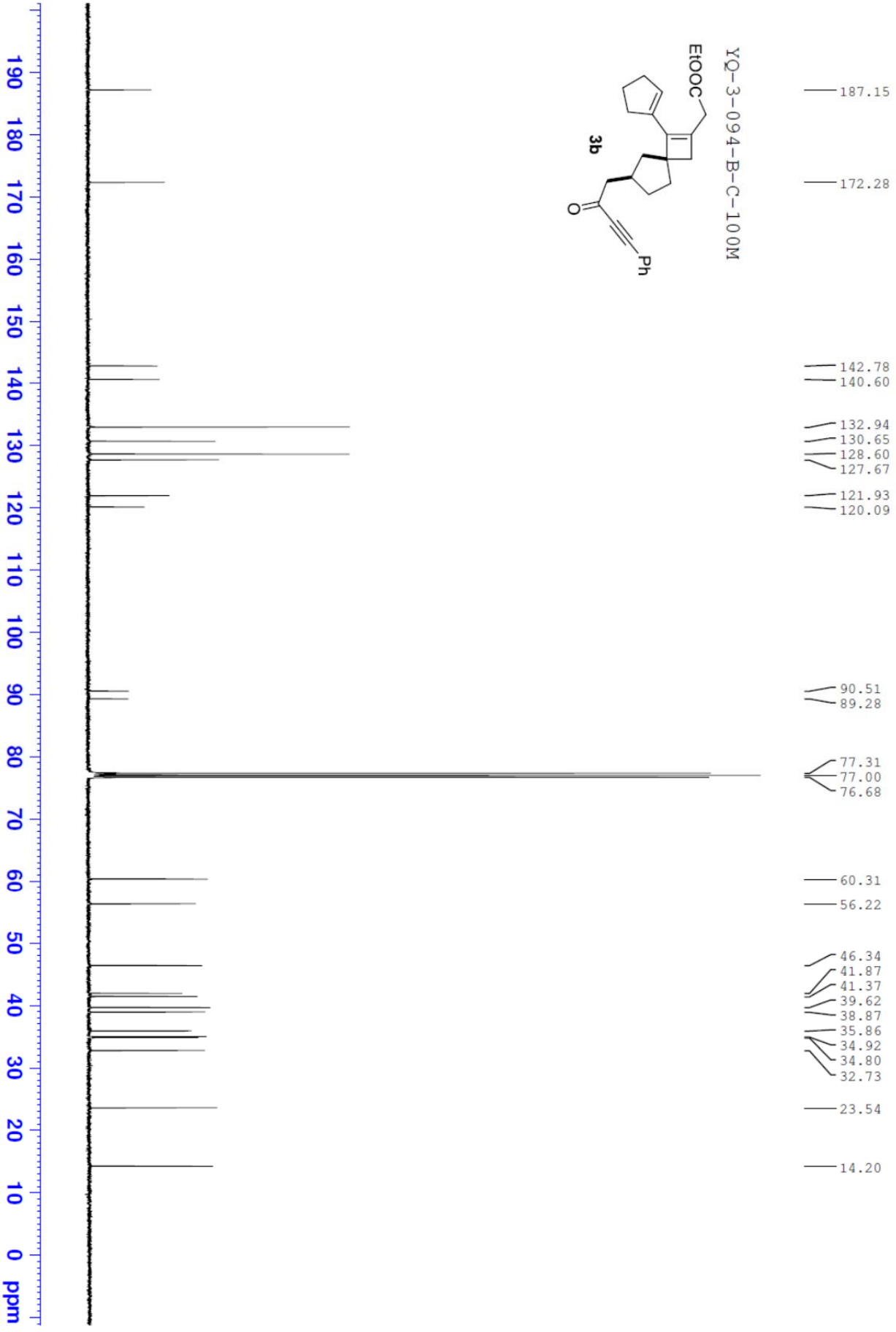
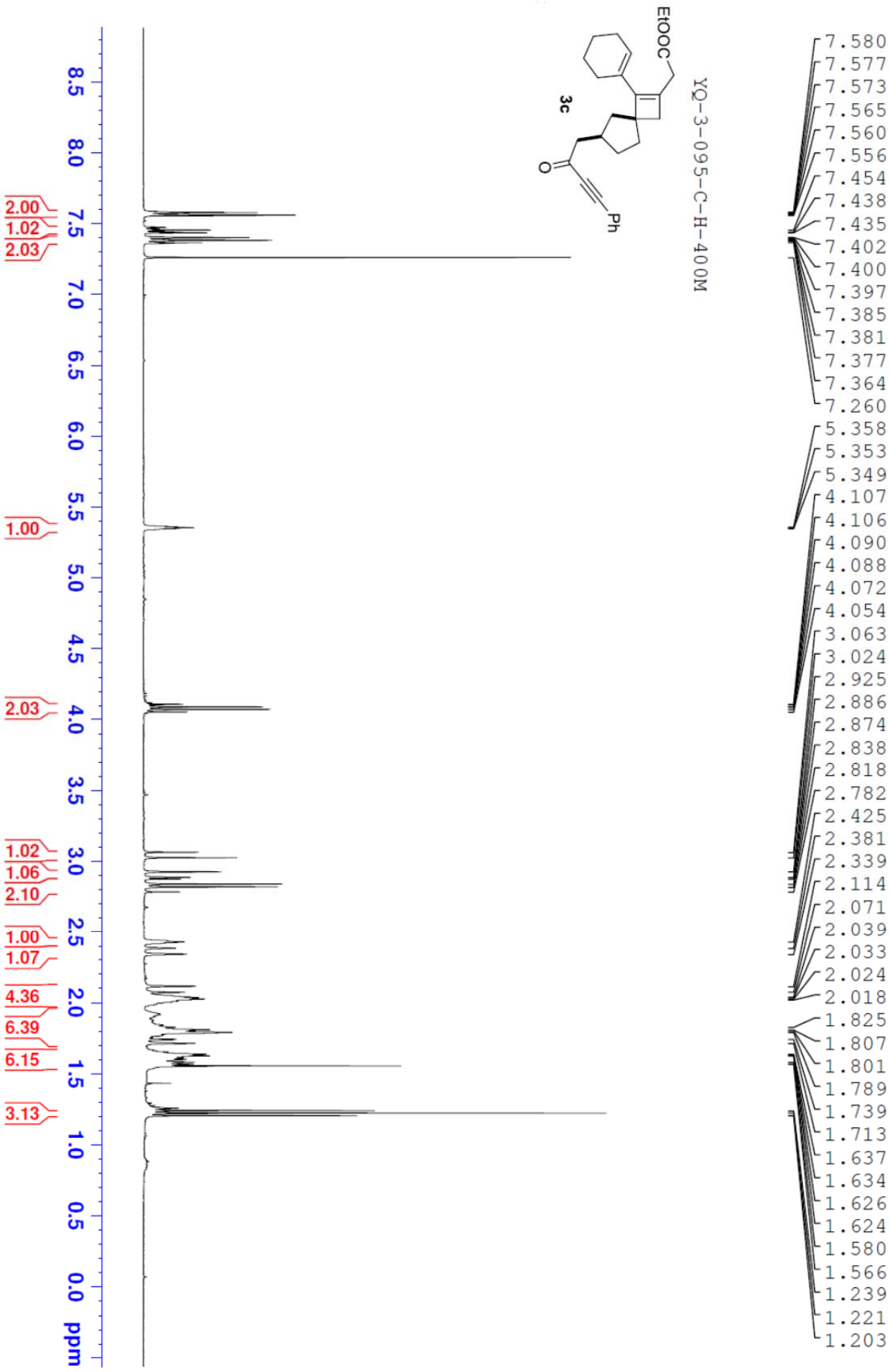
7.577
7.573
7.564
7.559
7.555
7.453
7.438
7.434
7.399
7.396
7.384
7.380
7.376
7.363
7.359
7.260
5.469
5.464
5.459
4.117
4.100
4.082
4.064
3.099
3.060
2.992
2.953
2.884
2.848
2.823
2.787
2.492
2.435
2.389
2.382
2.369
2.352
2.350
2.340
2.142
2.100
1.905
1.886
1.867
1.849
1.830
1.814
1.803
1.773
1.745
1.647
1.644
1.637
1.634
1.244
1.226
1.208
1.91
0.96
1.95
0.98
2.01
0.99
1.00
2.00
6.16
1.02
7.06
1.04
2.98
ppm


YQ-3-094-B-C-100M
EtOOC
3b
O
Ph
187.15
172.28
142.78
140.60
132.94
130.65
128.60
127.67
121.93
120.09
90.51
89.28
77.31
77.00
76.68
60.31
56.22
46.34
41.87
41.37
39.62
38.87
35.86
34.92
34.80
32.73
23.54
14.20
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


YQ-3-095-C-H-400M
3c
EtOOC
Ph
O
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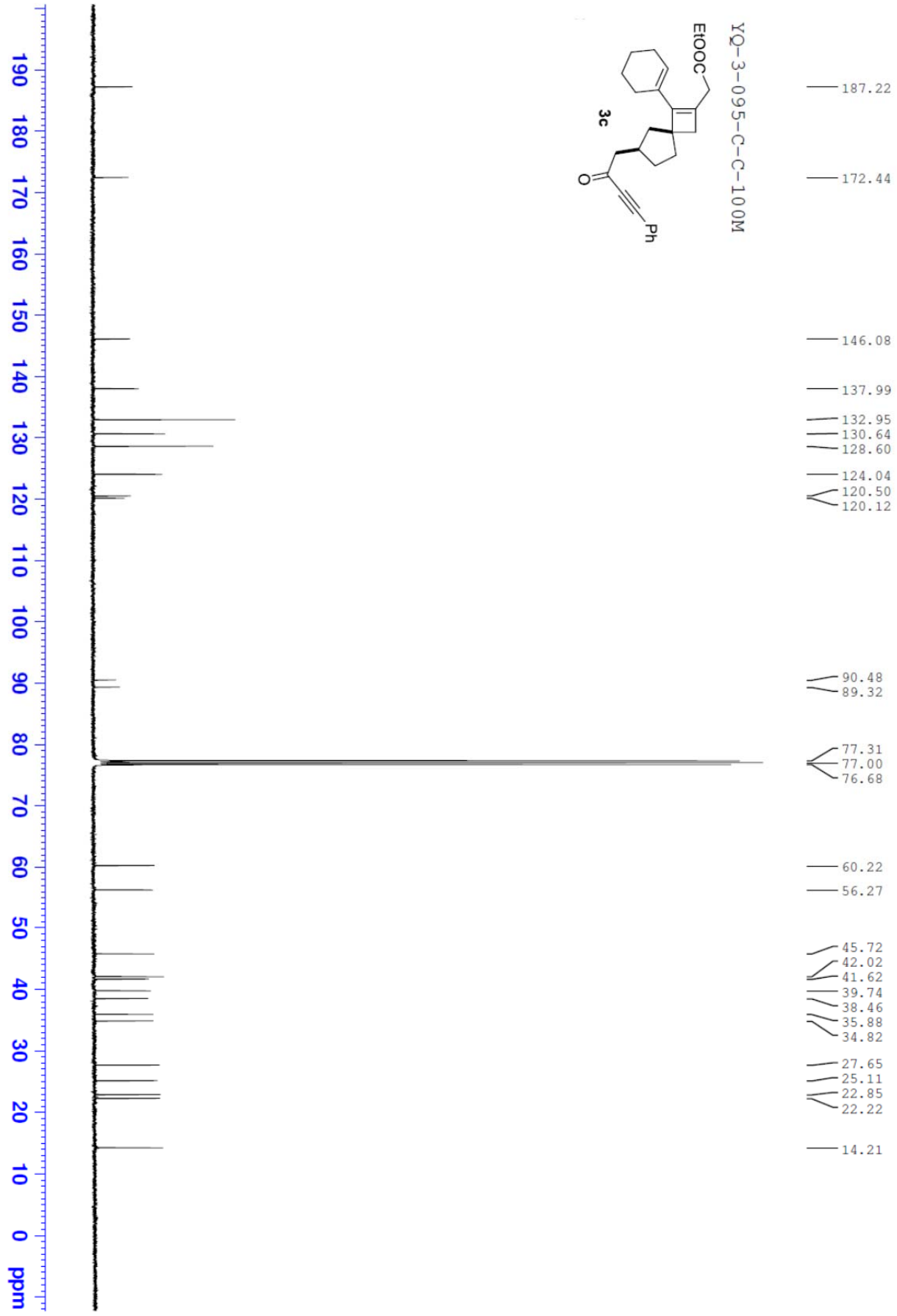
7.565
7.560
7.556
7.454
7.438
7.435
7.402
7.400
7.397
7.385
7.381
7.377
7.364
7.260
5.358
5.353
5.349
4.107
4.106
4.090
4.088
4.072
4.054
3.063
3.024
2.925
2.886
2.874
2.838
2.818
2.782
2.425
2.381
2.339
2.114
2.071
2.039
2.033
2.024
2.018
1.825
1.807
1.801
1.789
1.739
1.713
1.637
1.634
1.626
1.624
1.580
1.566
1.239
1.221
1.203
2.00
1.02
2.03
1.00
2.03
1.02
1.06
2.10
1.00
1.07
4.36
6.39
6.15
3.13
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
ppm


YQ-3-095-C-C-100M
EtOOC
3c
O
Ph
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172.44
146.08
137.99
132.95
130.64
128.60
124.04
120.50
120.12
90.48
89.32
77.31
77.00
76.68
60.22
56.27
45.72
42.02
41.62
39.74
38.46
35.88
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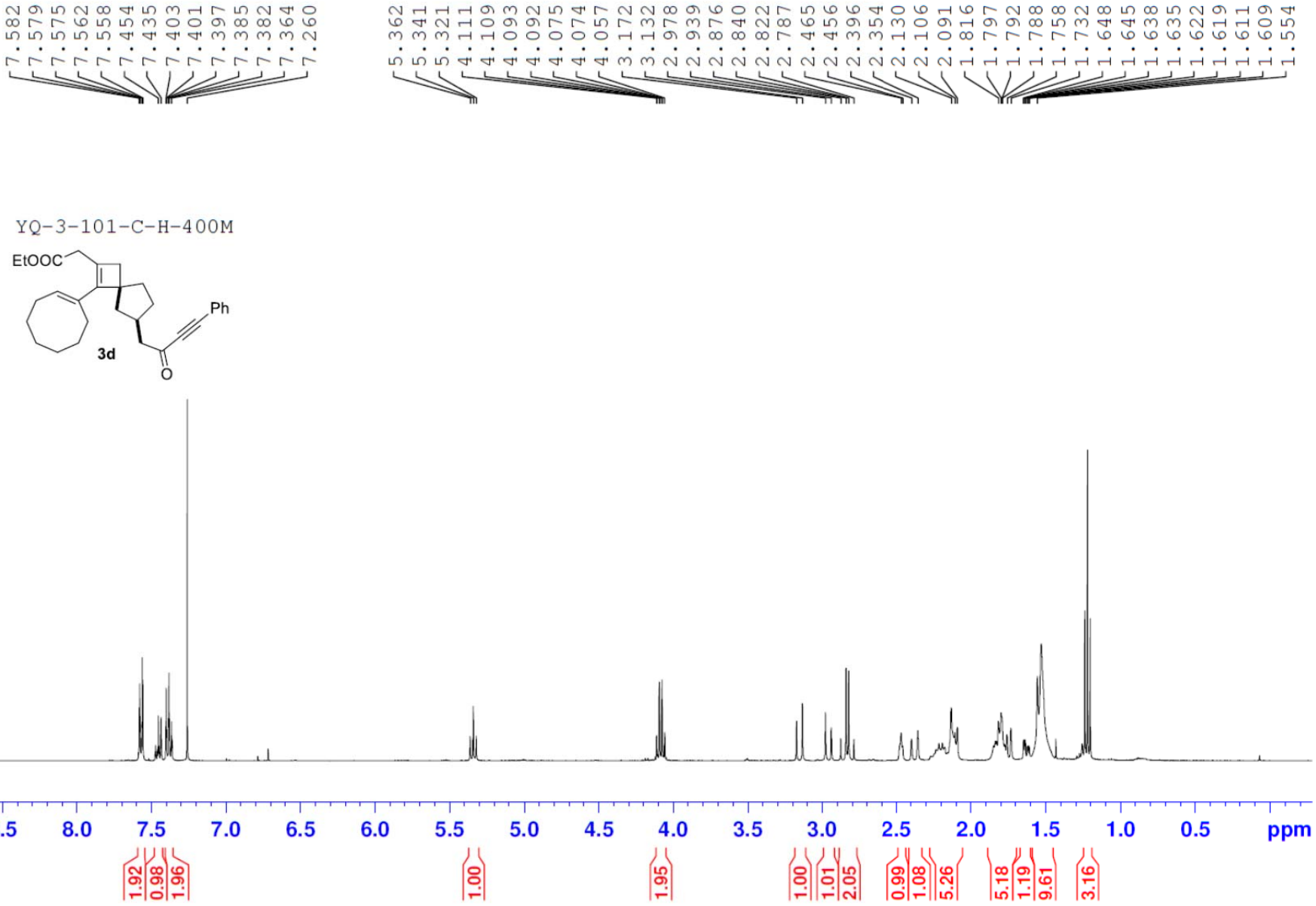
27.65
25.11
22.85
22.22
14.21
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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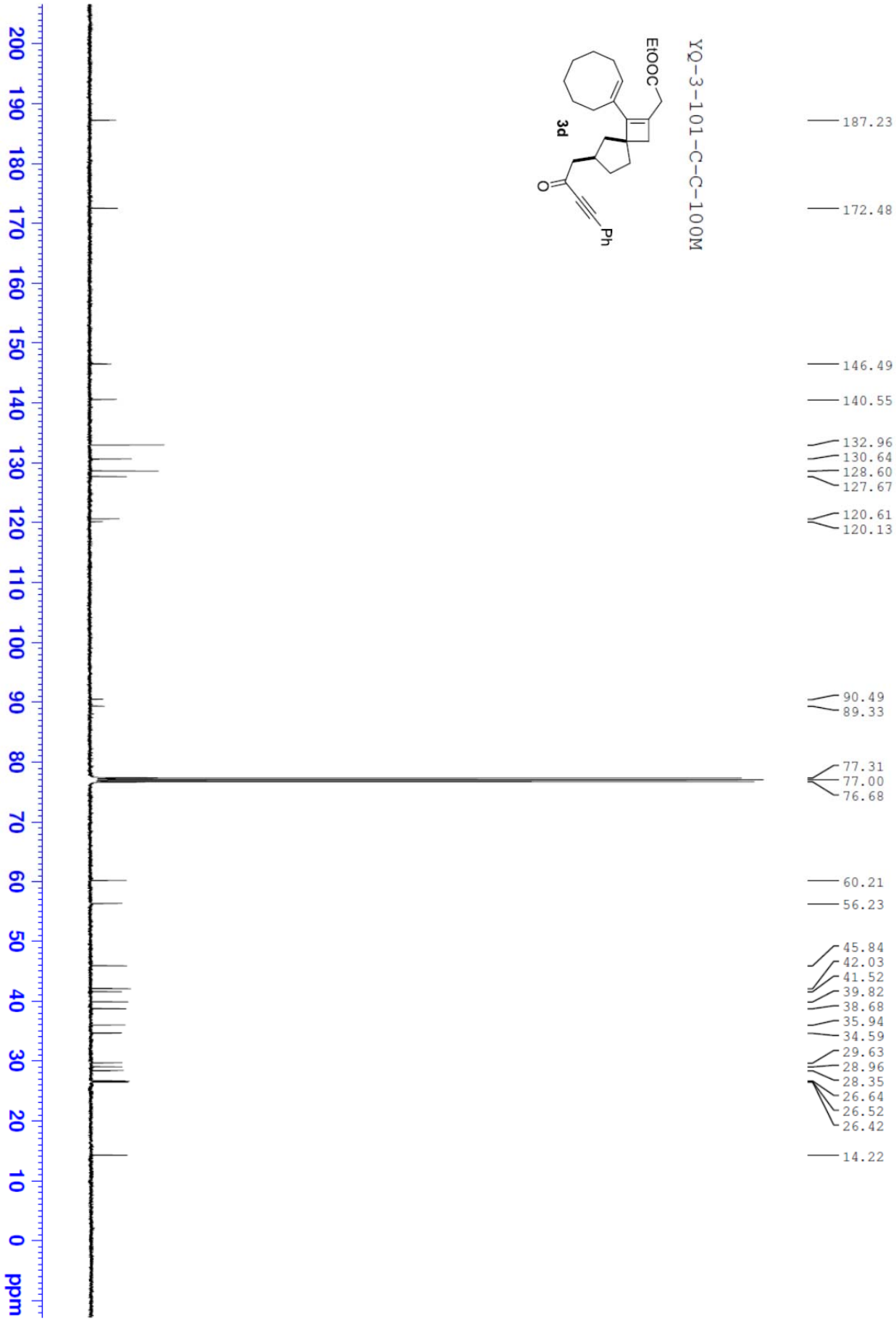
7.579
7.575
7.562
7.558
7.454
7.435
7.403
7.401
7.397
7.385
7.382
7.364
7.260
5.362
5.341
5.321
4.111
4.109
4.093
4.092
4.075
4.074
4.057
3.172
3.132
2.978
2.939
2.876
2.840
2.822
2.787
2.465
2.456
2.396
2.354
2.130
2.106
2.091
1.816
1.797
1.792
1.788
1.758
1.732
1.648
1.645
1.638
1.635
1.622
1.619
1.611
1.609
1.554
YQ-3-101-C-H-400M
EtOOC
Ph
O
3d
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
ppm
1.92
0.98
1.96
1.00
1.95
1.00
1.01
2.05
0.99
1.08
5.26
5.18
1.19
9.61
3.16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EtOOC
3d
O
Ph
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140.55
132.96
130.64
128.60
127.67
120.61
120.13
90.49
89.33
77.31
77.00
76.68
60.21
56.23
45.84
42.03
41.52
39.82
38.68
35.94
34.59
29.63
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28.35
26.64
26.52
26.42
14.22
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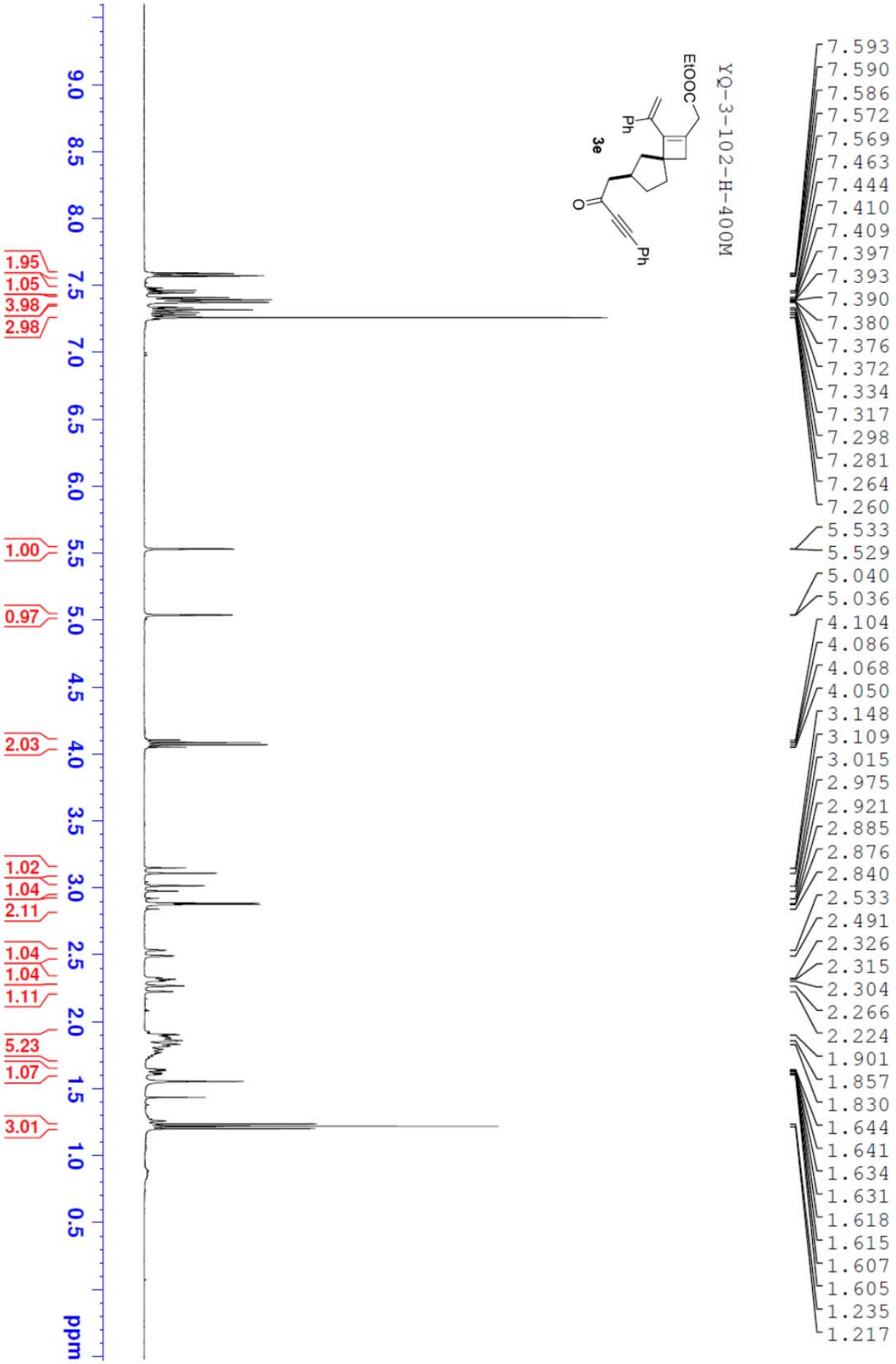
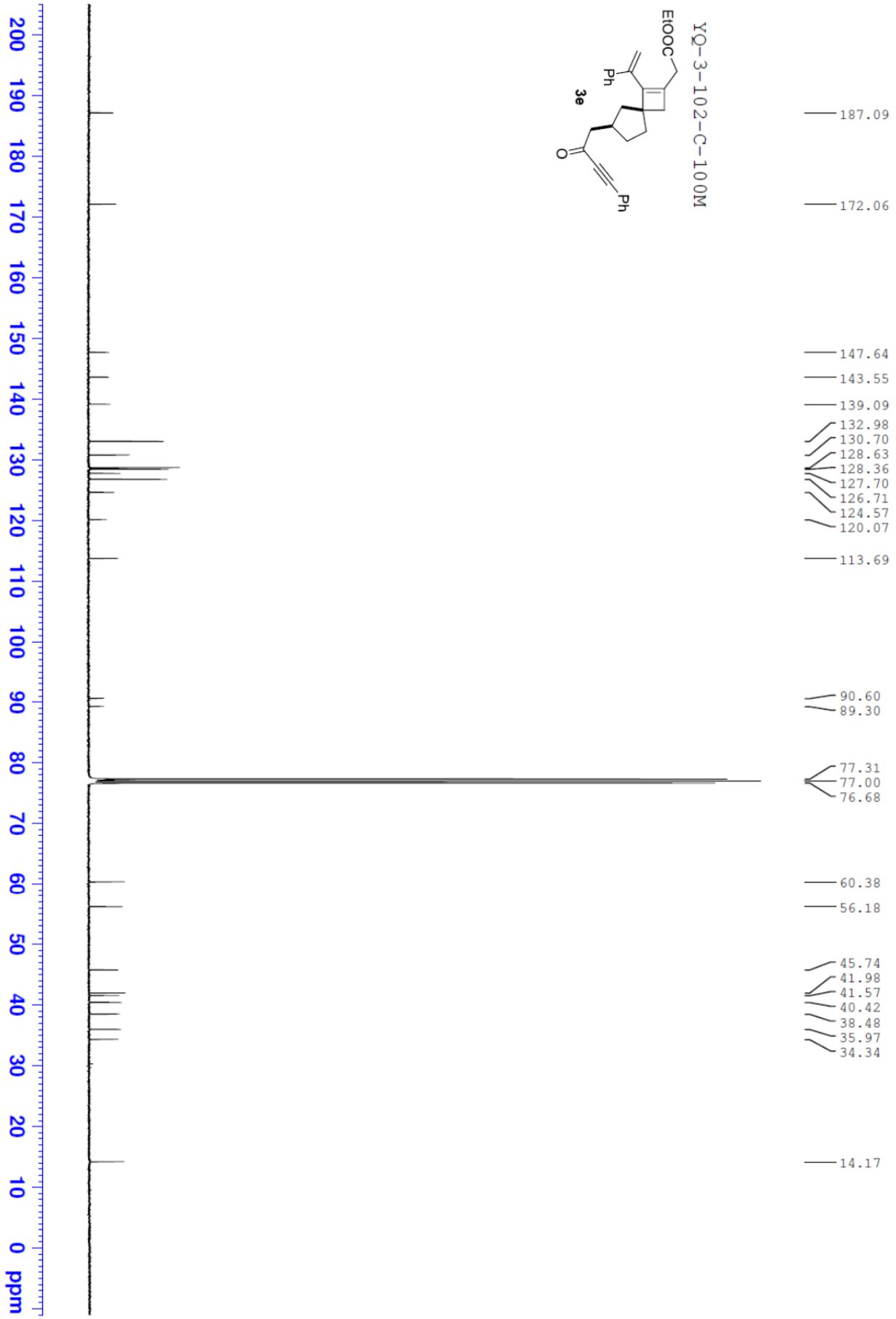

YQ-3-102-H-400M
EtOOC
Ph
3e
O
Ph
7.593
7.590
7.586
7.572
7.569
7.463
7.444
7.410
7.409
7.397
7.393
7.390
7.380
7.376
7.372
7.334
7.317
7.298
7.281
7.264
7.260
5.533
5.529
5.040
5.036
4.104
4.086
4.068
4.050
3.148
3.109
3.015
2.975
2.921
2.885
2.876
2.840
2.533
2.491
2.326
2.315
2.304
2.266
2.224
1.901
1.857
1.830
1.644
1.641
1.634
1.631
1.618
1.615
1.607
1.605
1.235
1.217
1.95
1.05
3.98
2.98
1.00
0.97
2.03
1.02
1.04
2.11
1.04
1.04
1.11
5.23
1.07
3.01
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
ppm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EtOOC
Ph
3e
O
Ph
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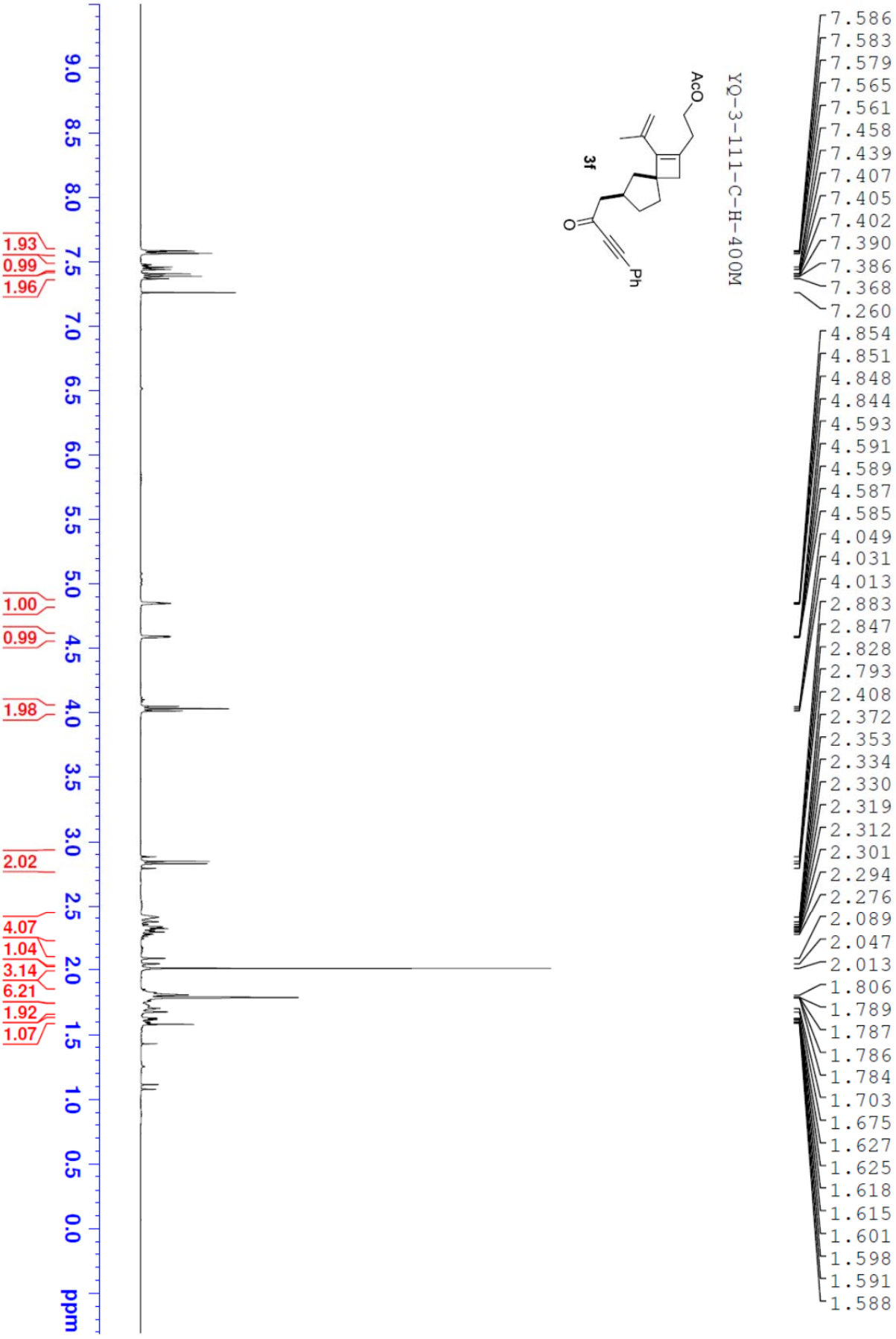
143.55
139.09
132.98
130.70
128.63
128.36
127.70
126.71
124.57
120.07
113.69
90.60
89.30
77.31
77.00
76.68
60.38
56.18
45.74
41.98
41.57
40.42
38.48
35.97
34.34
14.17
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


YQ-3-111-C-H-400M
AcO
3f
O
Ph
7.586
7.583
7.579
7.565
7.561
7.458
7.439
7.407
7.405
7.402
7.390
7.386
7.368
7.260
4.854
4.851
4.848
4.844
4.593
4.591
4.589
4.587
4.585
4.049
4.031
4.013
2.883
2.847
2.828
2.793
2.408
2.372
2.353
2.334
2.330
2.319
2.312
2.301
2.294
2.276
2.089
2.047
2.013
1.806
1.789
1.787
1.786
1.784
1.703
1.675
1.627
1.625
1.618
1.615
1.601
1.598
1.591
1.588
1.93
0.99
1.96
1.00
0.99
1.98
2.02
4.07
1.04
3.14
6.21
1.92
1.07
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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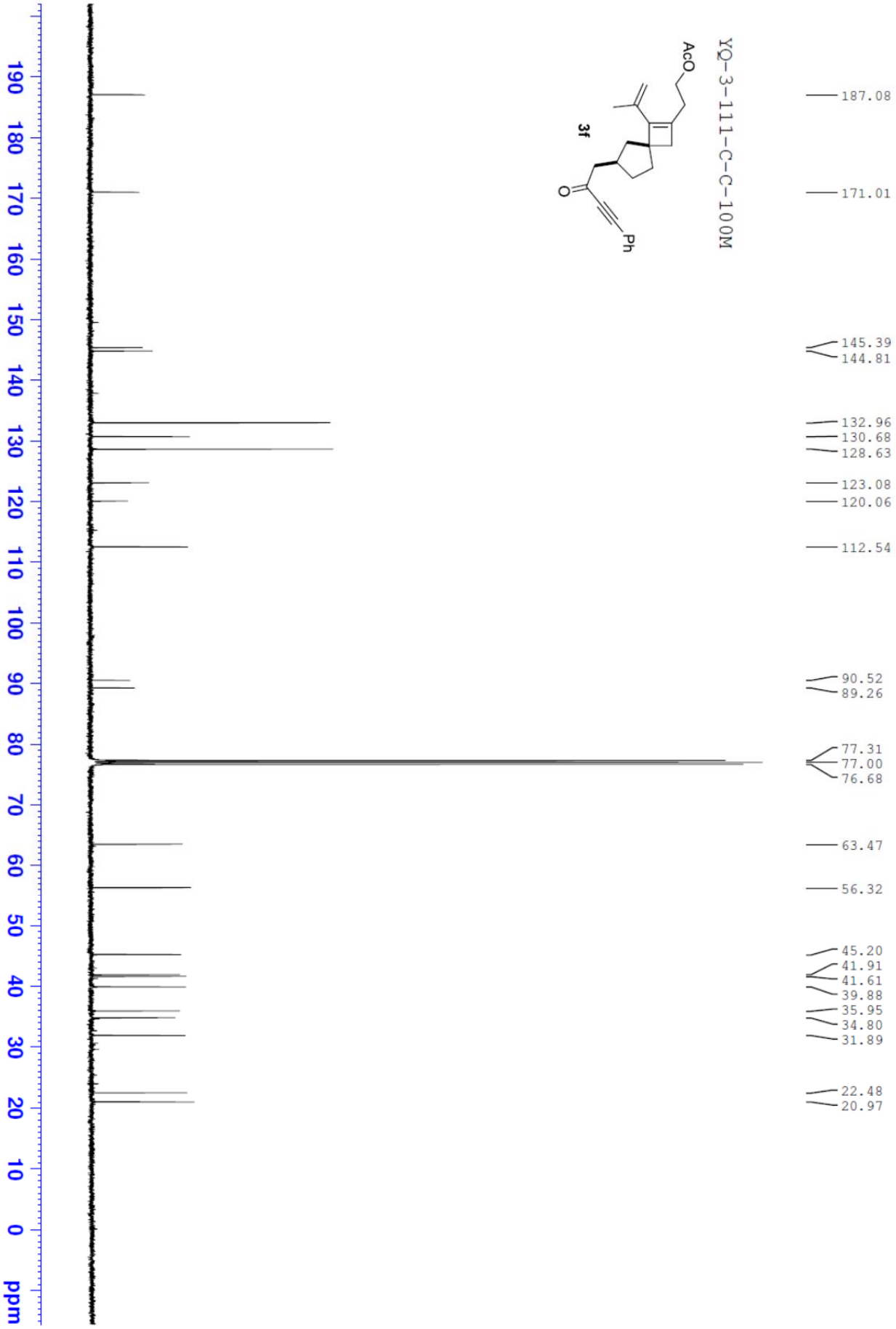
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm


YQ-3-111-C-C-100M
AcO
3f
O
Ph
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171.01
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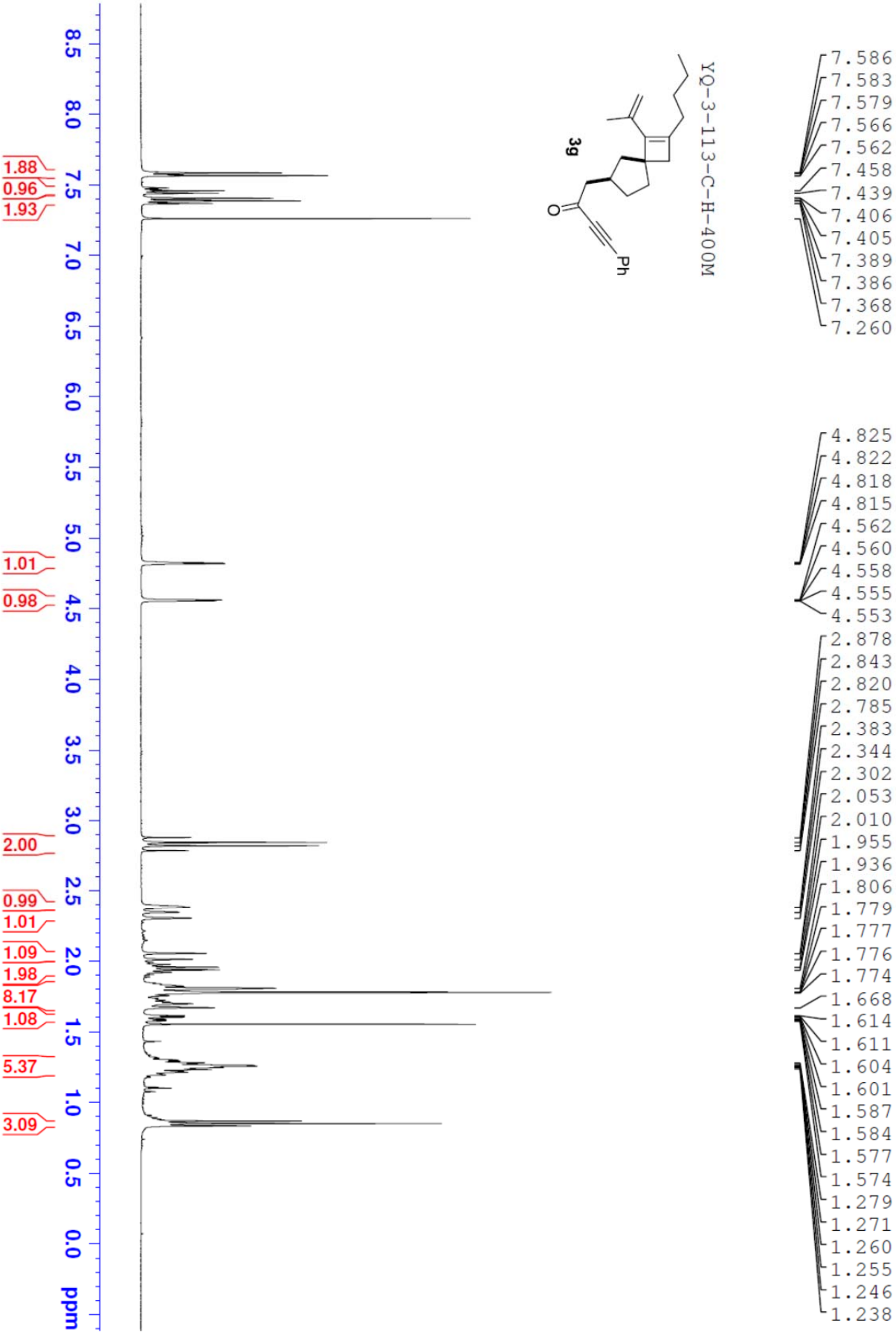
144.81
132.96
130.68
128.63
123.08
120.06
112.54
90.52
89.26
77.31
77.00
76.68
63.47
56.32
45.20
41.91
41.61
39.88
35.95
34.80
31.89
22.48
20.97
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


YQ-3-113-C-H-400M
3g
Ph
O
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7.579
7.566
7.562
7.458
7.439
7.406
7.405
7.389
7.386
7.368
7.260
4.825
4.822
4.818
4.815
4.562
4.560
4.558
4.555
4.553
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2.843
2.820
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2.383
2.344
2.302
2.053
2.010
1.955
1.936
1.806
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1.777
1.776
1.774
1.668
1.614
1.611
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1.584
1.577
1.574
1.279
1.271
1.260
1.255
1.246
1.238
1.88
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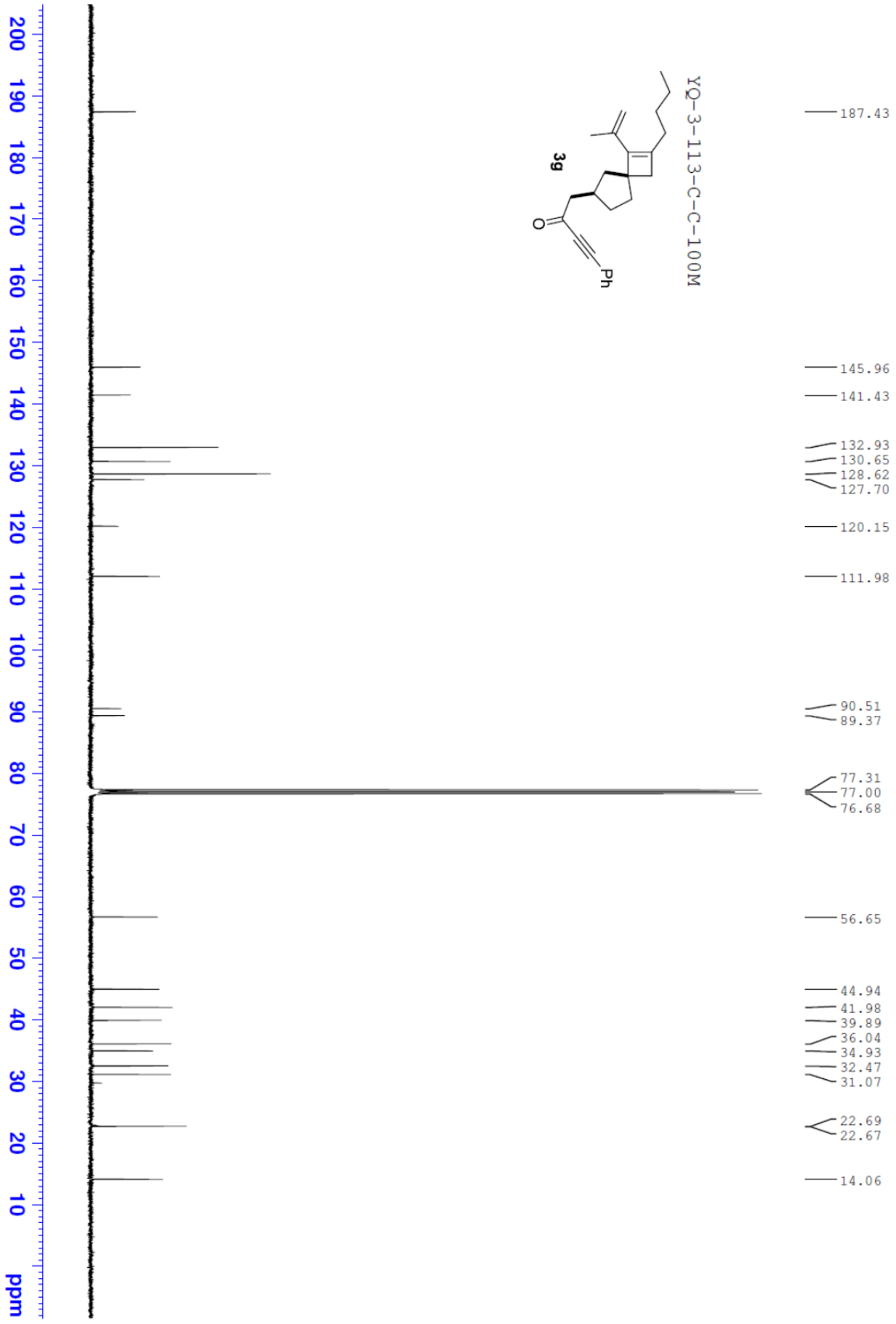
1.93
1.01
0.98
2.00
0.99
1.01
1.09
1.98
8.17
1.08
5.37
3.09
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm


YQ-3-113-C-C-100M
3g
O
Ph
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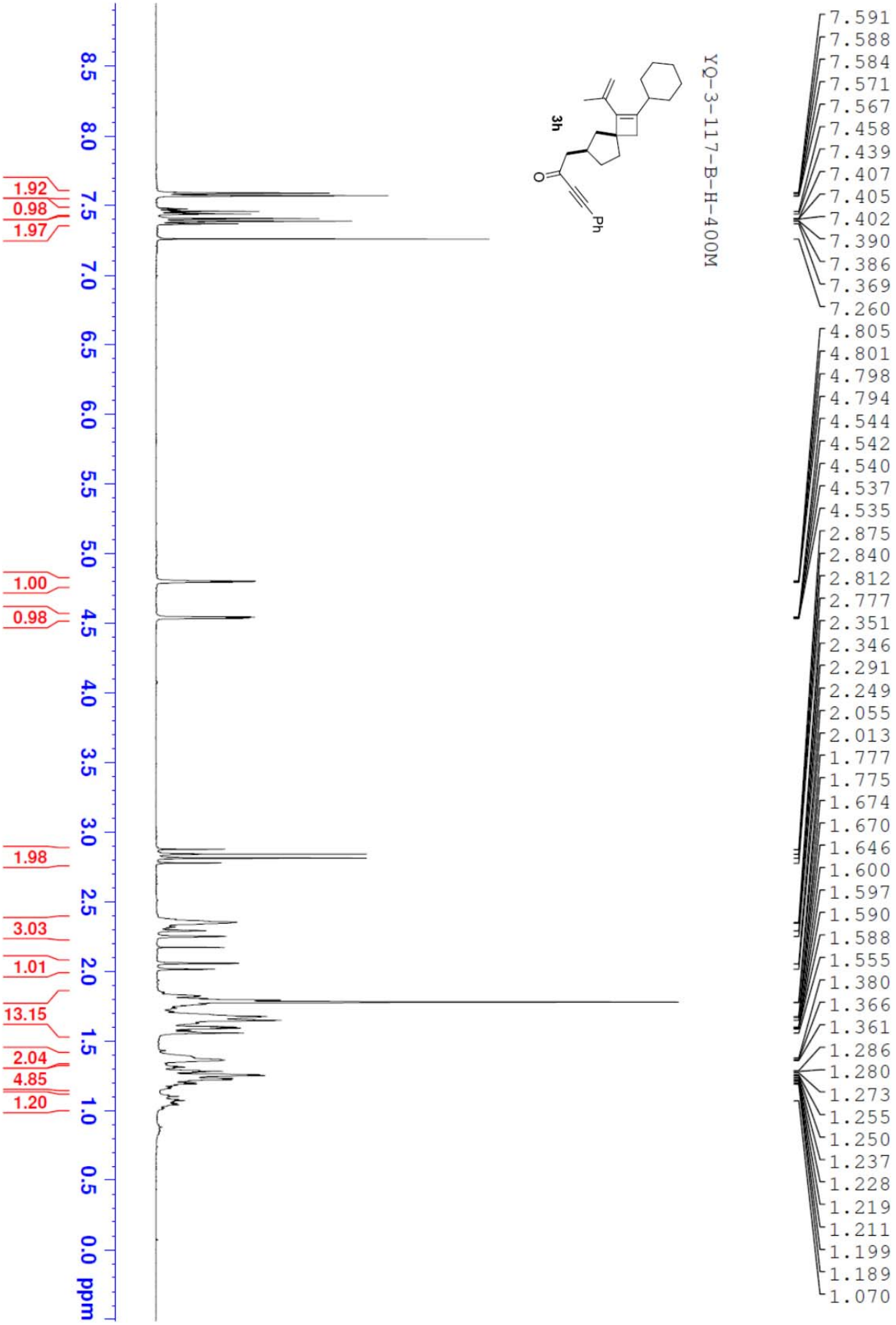
130.65
128.62
127.70
120.15
111.98
90.51
89.37
77.31
77.00
76.68
56.65
44.94
41.98
39.89
36.04
34.93
32.47
31.07
22.69
22.67
14.06
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


YQ-3-117-B-H-400M
3h
O
Ph
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7.584
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7.567
7.458
7.439
7.407
7.405
7.402
7.390
7.386
7.369
7.260
4.805
4.801
4.798
4.794
4.544
4.542
4.540
4.537
4.535
2.875
2.840
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2.777
2.351
2.346
2.291
2.249
2.055
2.013
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1.97
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0.98
1.98
3.03
1.01
13.15
2.04
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1.20
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
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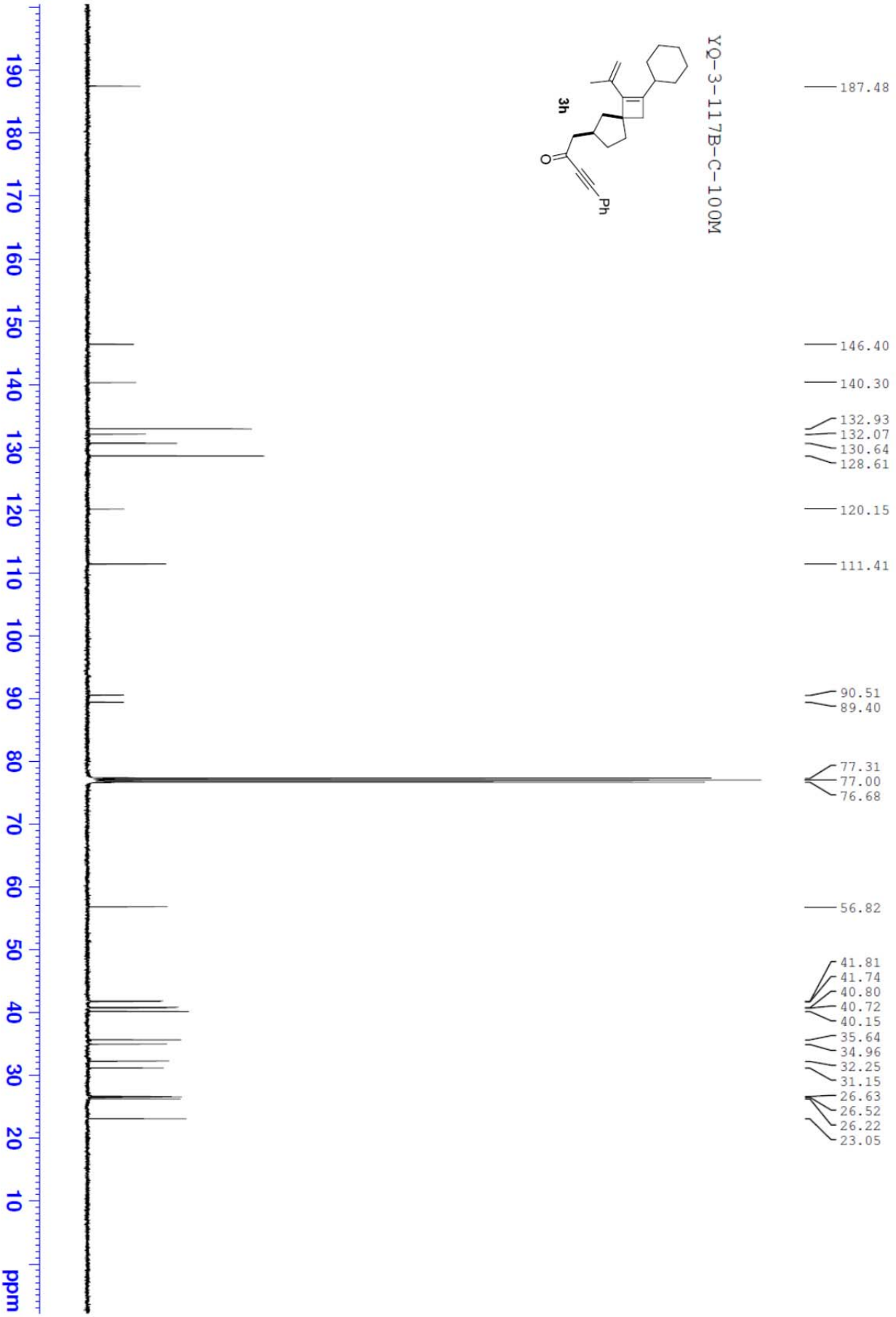
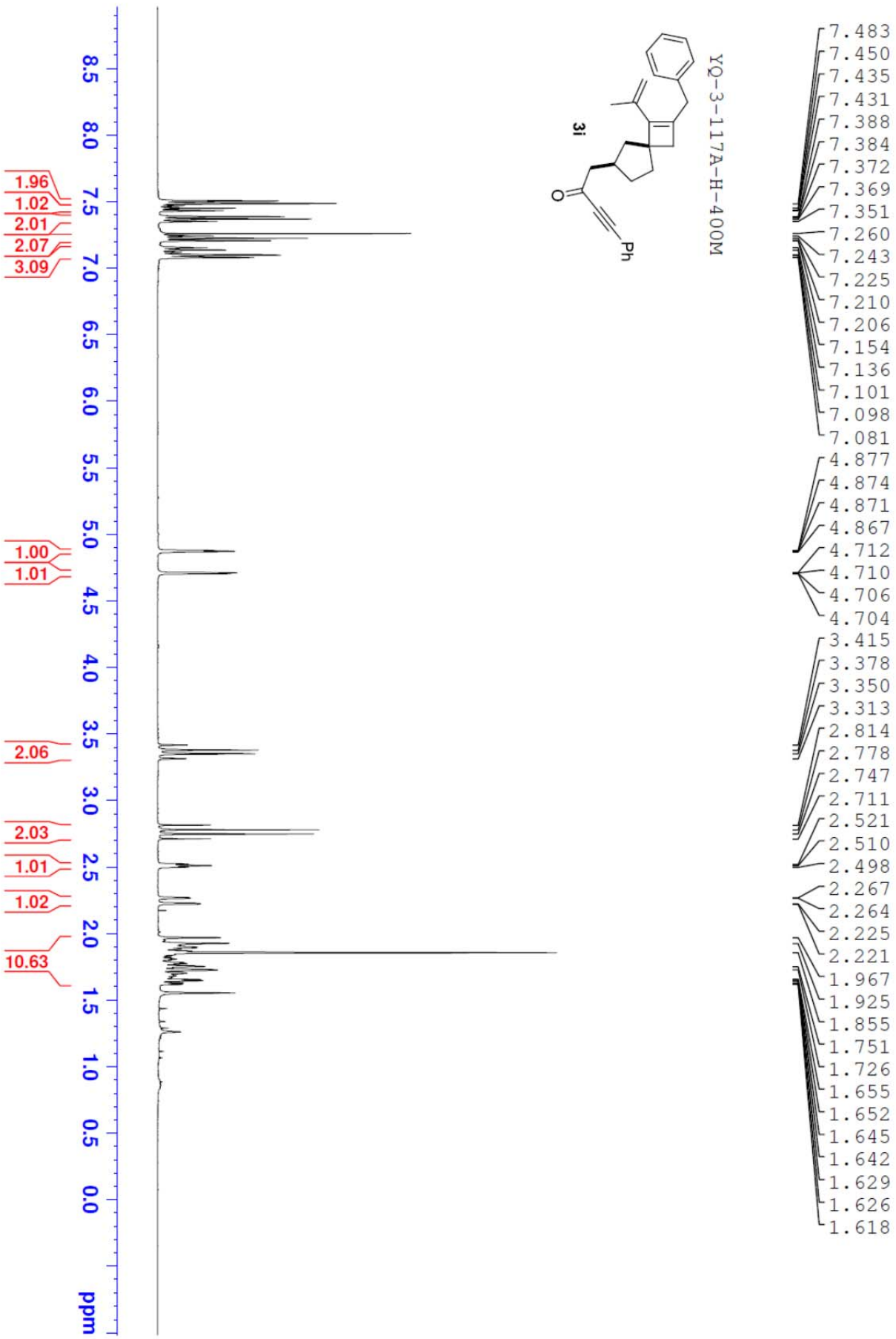
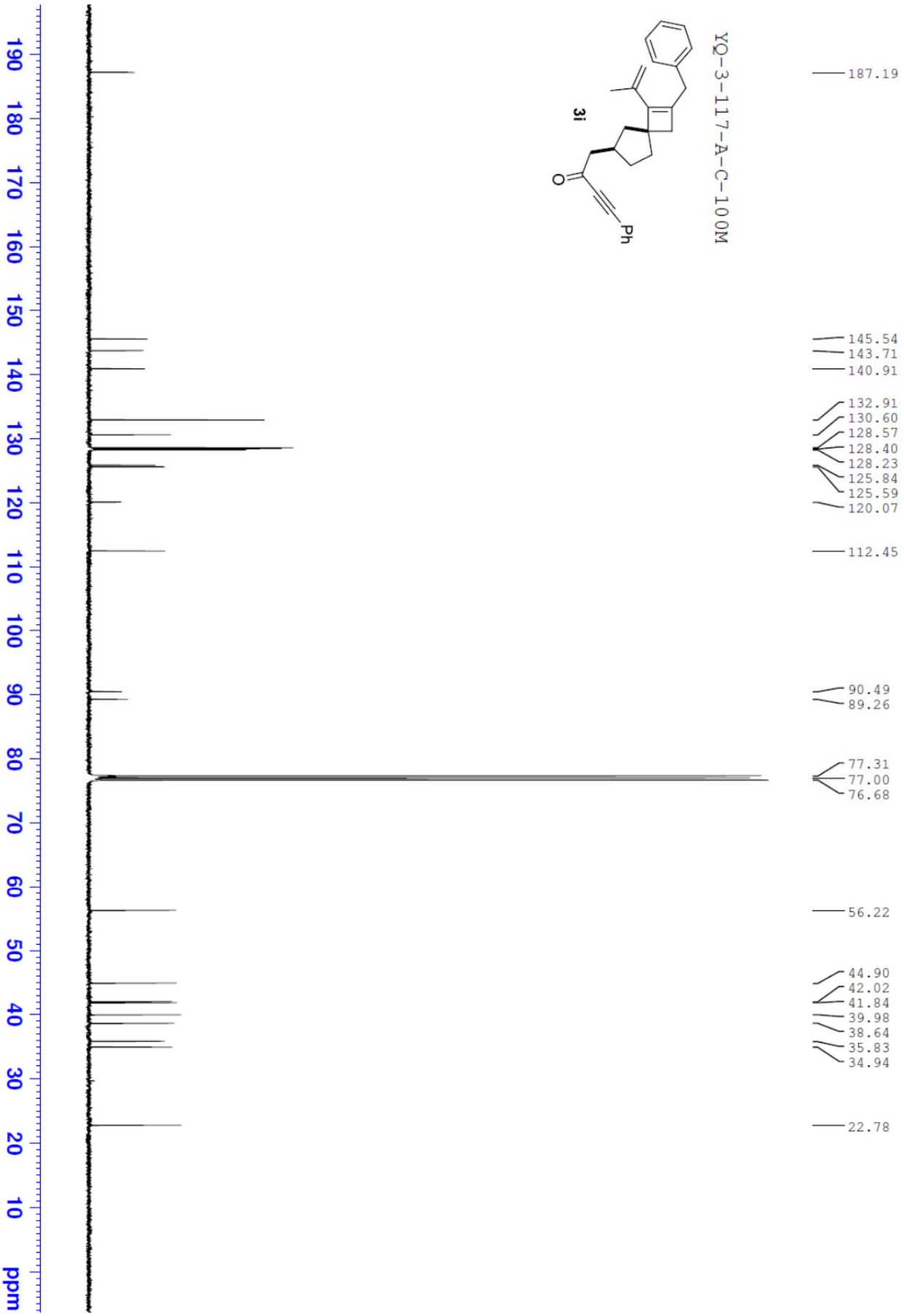
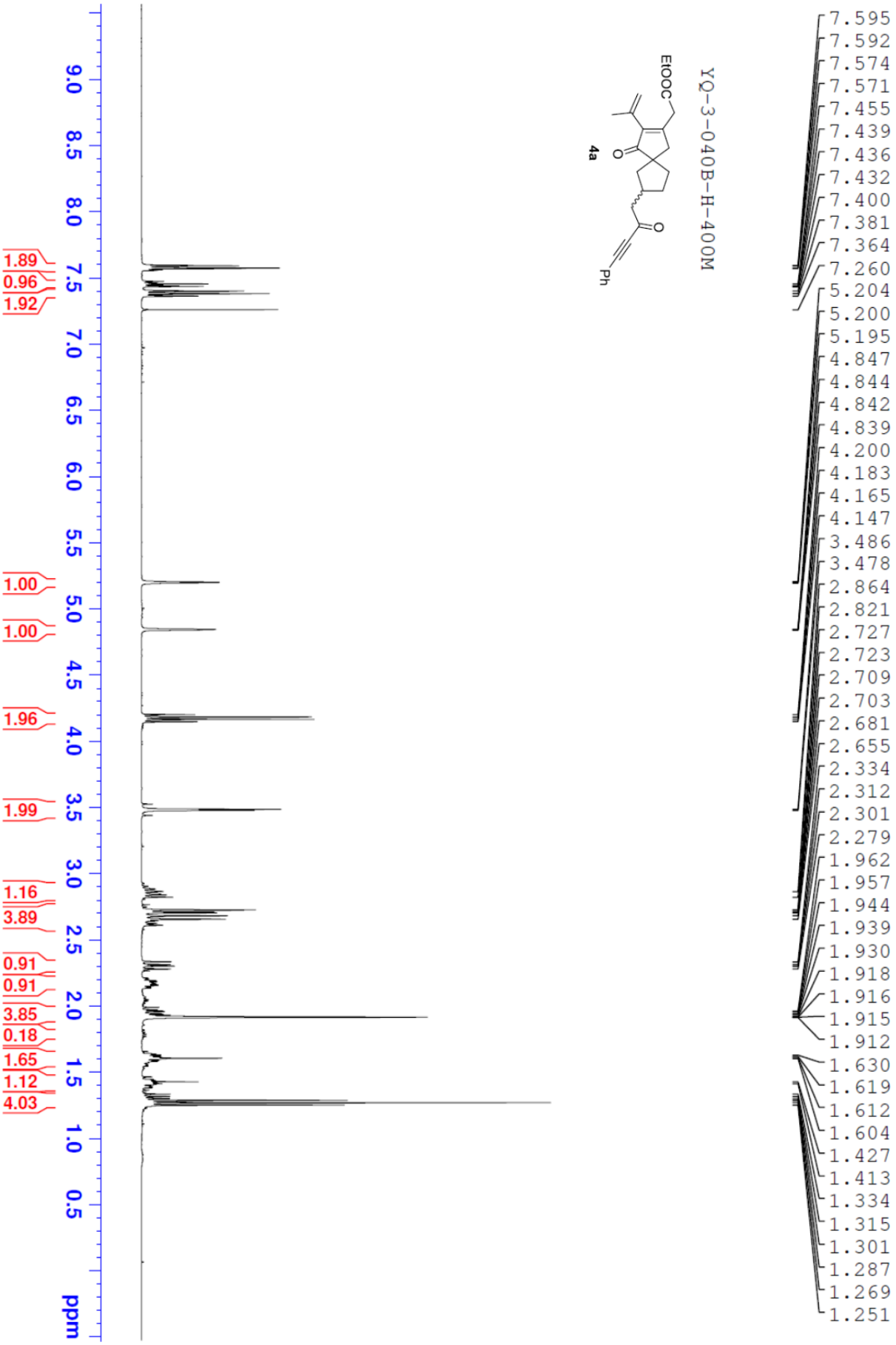
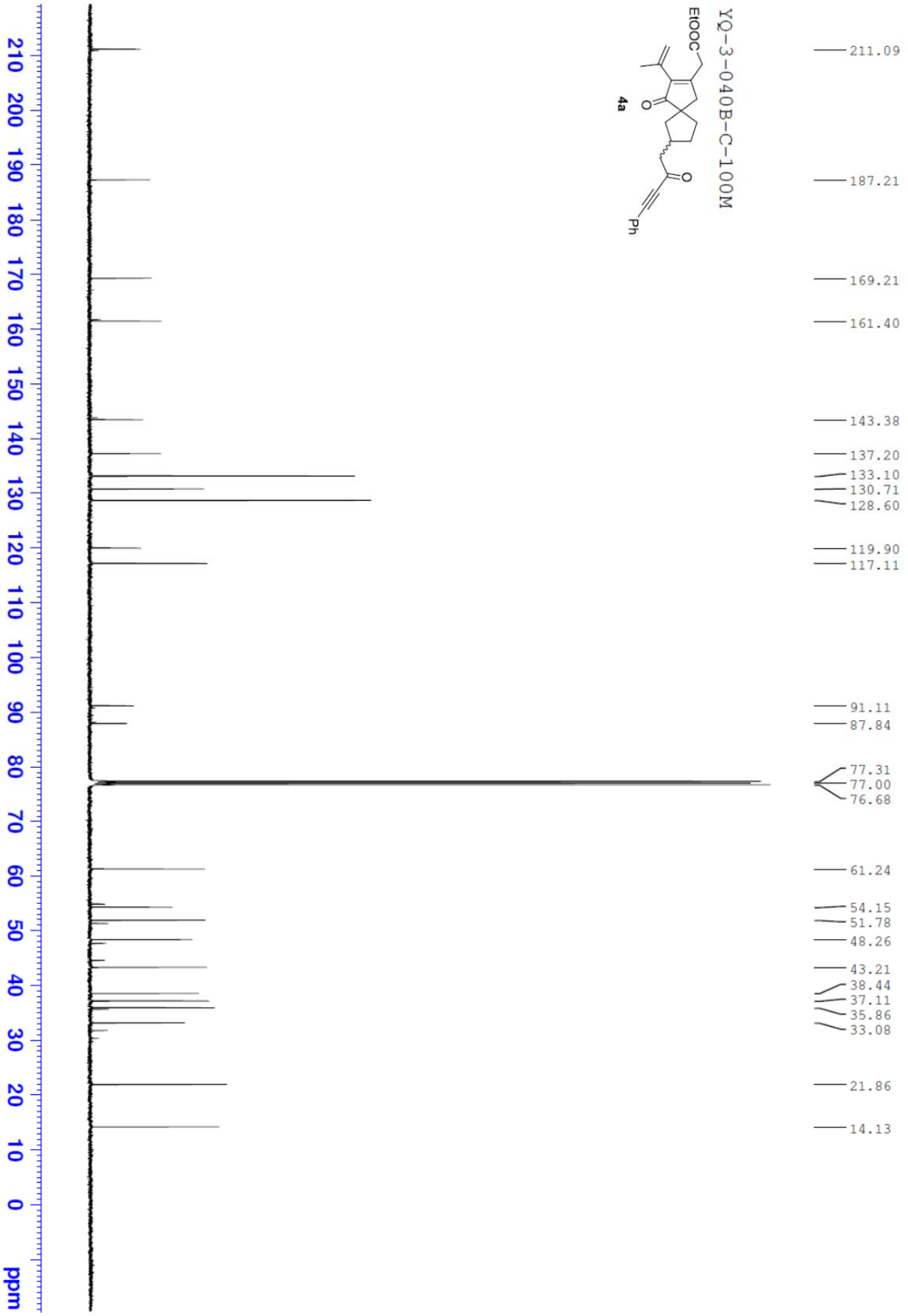
0.5
0.0
ppm

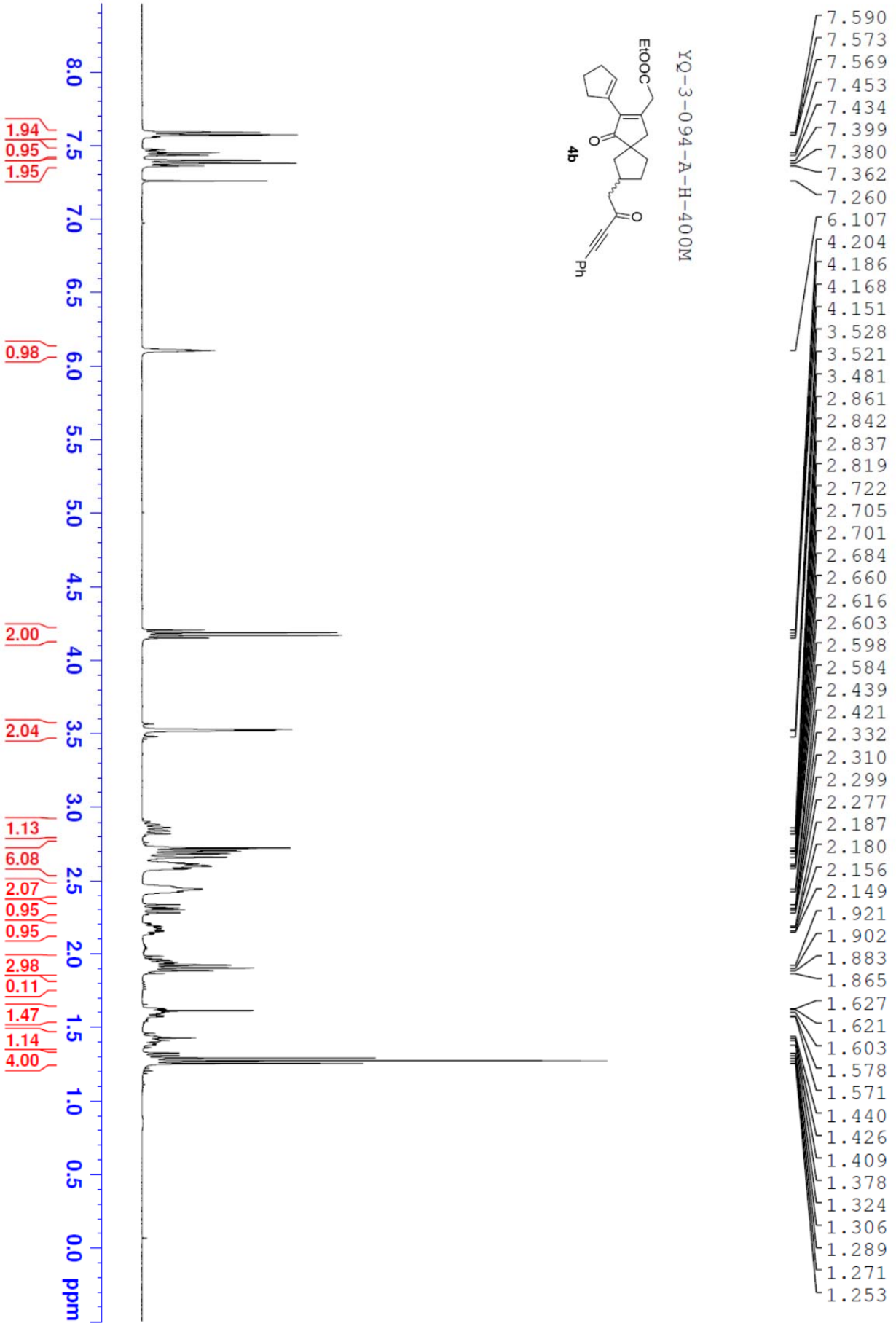

YQ-3-117B-C-100M
3h
Ph
187.48
146.40
140.30
132.93
132.07
130.64
128.61
120.15
111.41
90.51
89.40
77.31
77.00
76.68
56.82
41.81
41.74
40.80
40.72
40.15
35.64
34.96
32.25
31.15
26.63
26.52
26.22
23.05
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

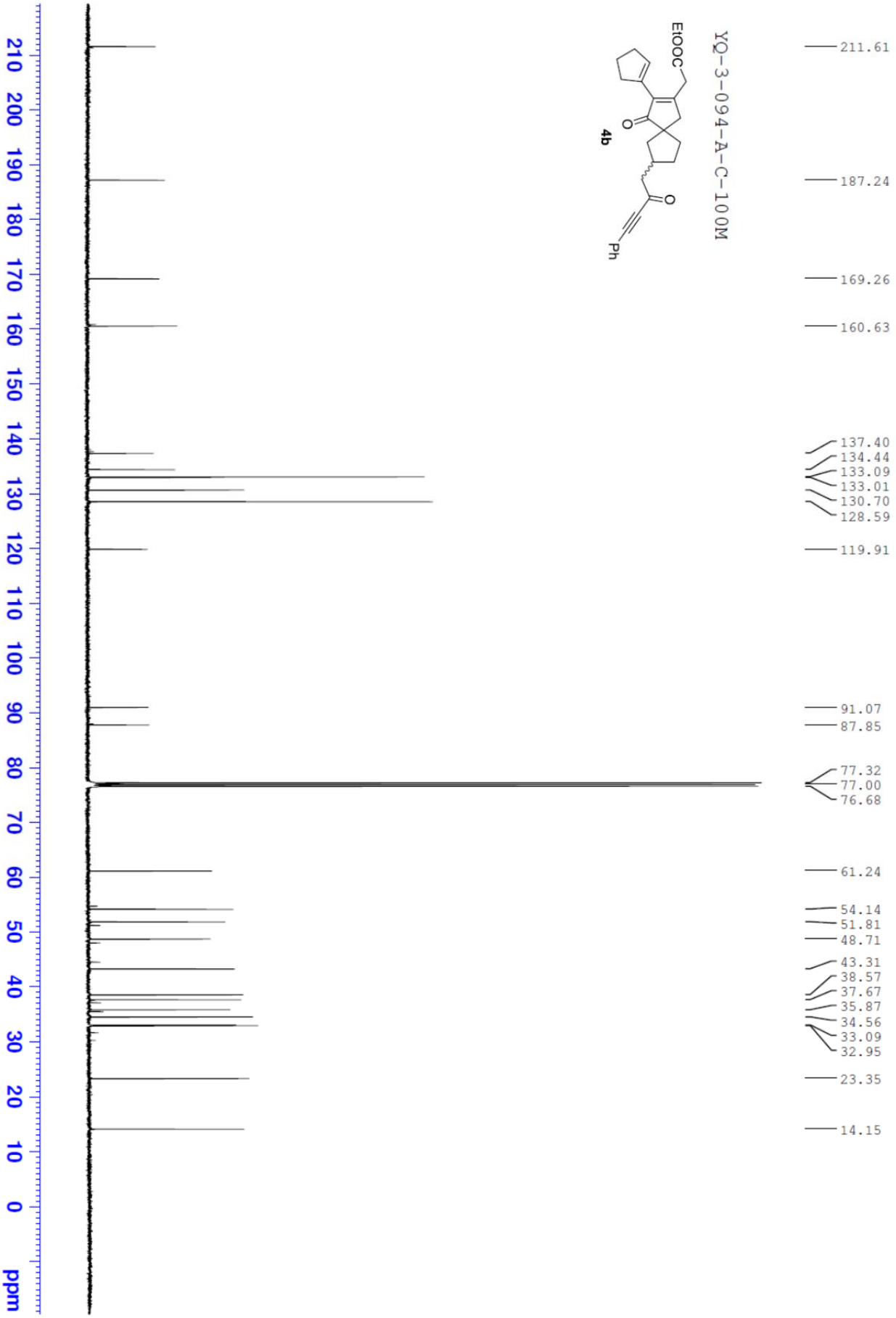

YQ-3-117A-H-400M
3i
Ph
O
7.483
7.450
7.435
7.431
7.388
7.384
7.372
7.369
7.351
7.260
7.243
7.225
7.210
7.206
7.154
7.136
7.101
7.098
7.081
4.877
4.874
4.871
4.867
4.712
4.710
4.706
4.704
3.415
3.378
3.350
3.313
2.814
2.778
2.747
2.711
2.521
2.510
2.498
2.267
2.264
2.225
2.221
1.967
1.925
1.855
1.751
1.726
1.655
1.652
1.645
1.642
1.629
1.626
1.618
1.96
1.02
2.01
2.07
3.09
1.00
1.01
2.06
2.03
1.01
1.02
10.63
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
ppm

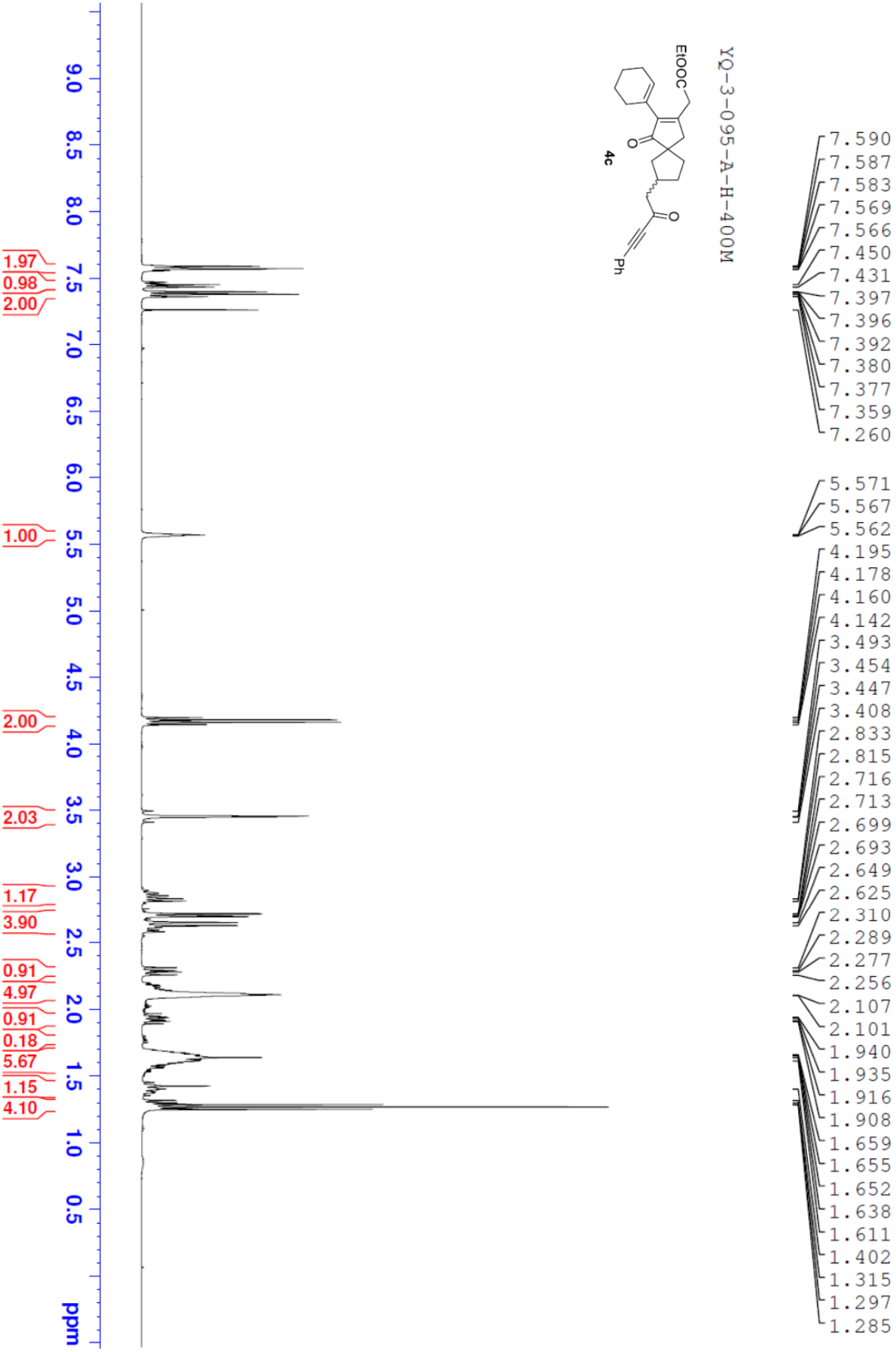

YQ-3-117-A-C-100M
3i
O
Ph
187.19
145.54
143.71
140.91
132.91
130.60
128.57
128.40
128.23
125.84
125.59
120.07
112.45
90.49
89.26
77.31
77.00
76.68
56.22
44.90
42.02
41.84
39.98
38.64
35.83
34.94
22.78
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

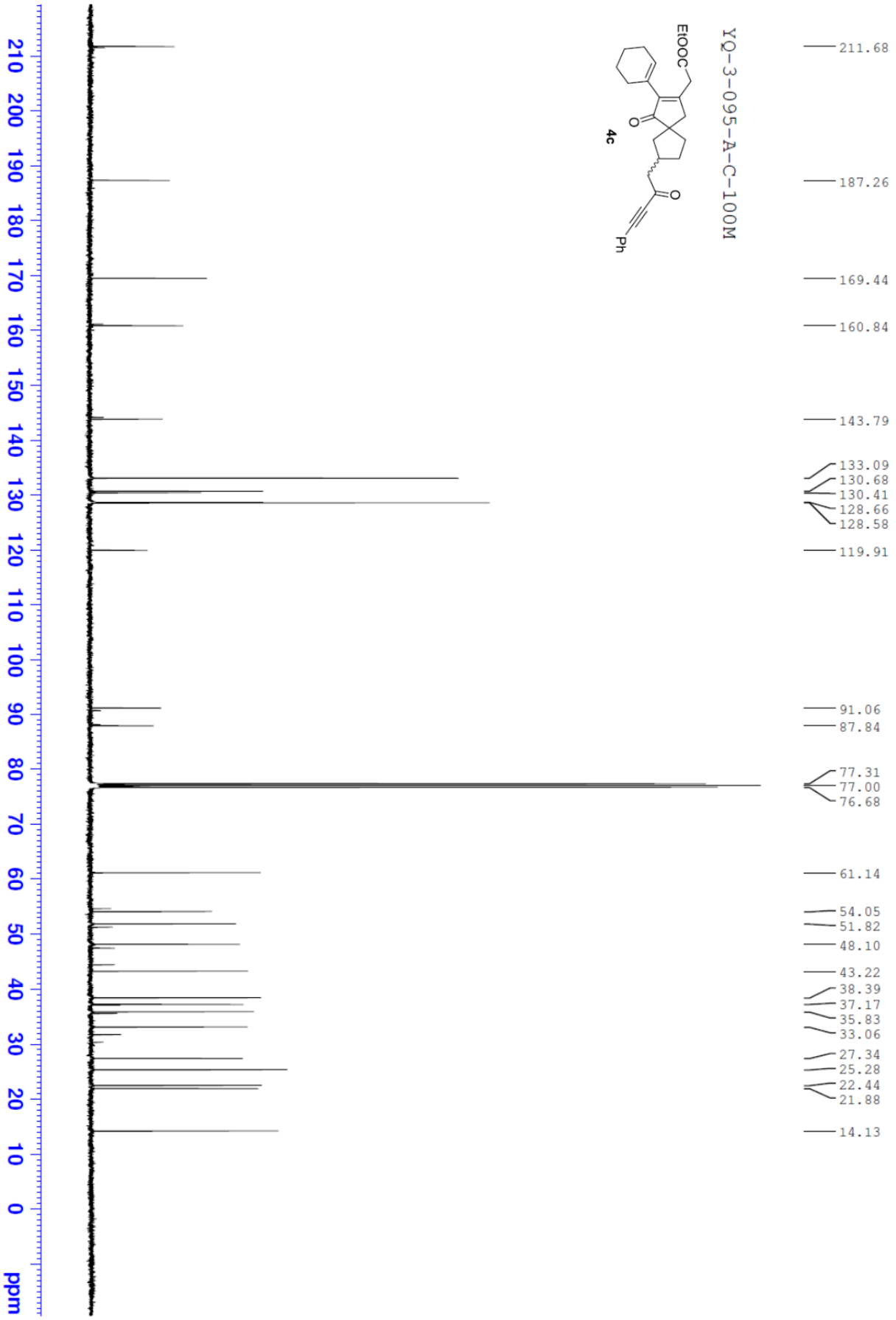

YQ-3-040B-H-400M
EtOOC
O
O
O
Ph
4a
7.595
7.592
7.574
7.571
7.455
7.439
7.436
7.432
7.400
7.381
7.364
7.260
5.204
5.200
5.195
4.847
4.844
4.842
4.839
4.200
4.183
4.165
4.147
3.486
3.478
2.864
2.821
2.727
2.723
2.709
2.703
2.681
2.655
2.334
2.312
2.301
2.279
1.962
1.957
1.944
1.939
1.930
1.918
1.916
1.915
1.912
1.630
1.619
1.612
1.604
1.427
1.413
1.334
1.315
1.301
1.287
1.269
1.251
1.89
0.96
1.92
1.00
1.00
1.96
1.99
1.16
3.89
0.91
0.91
3.85
0.18
1.65
1.12
4.03
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
ppm

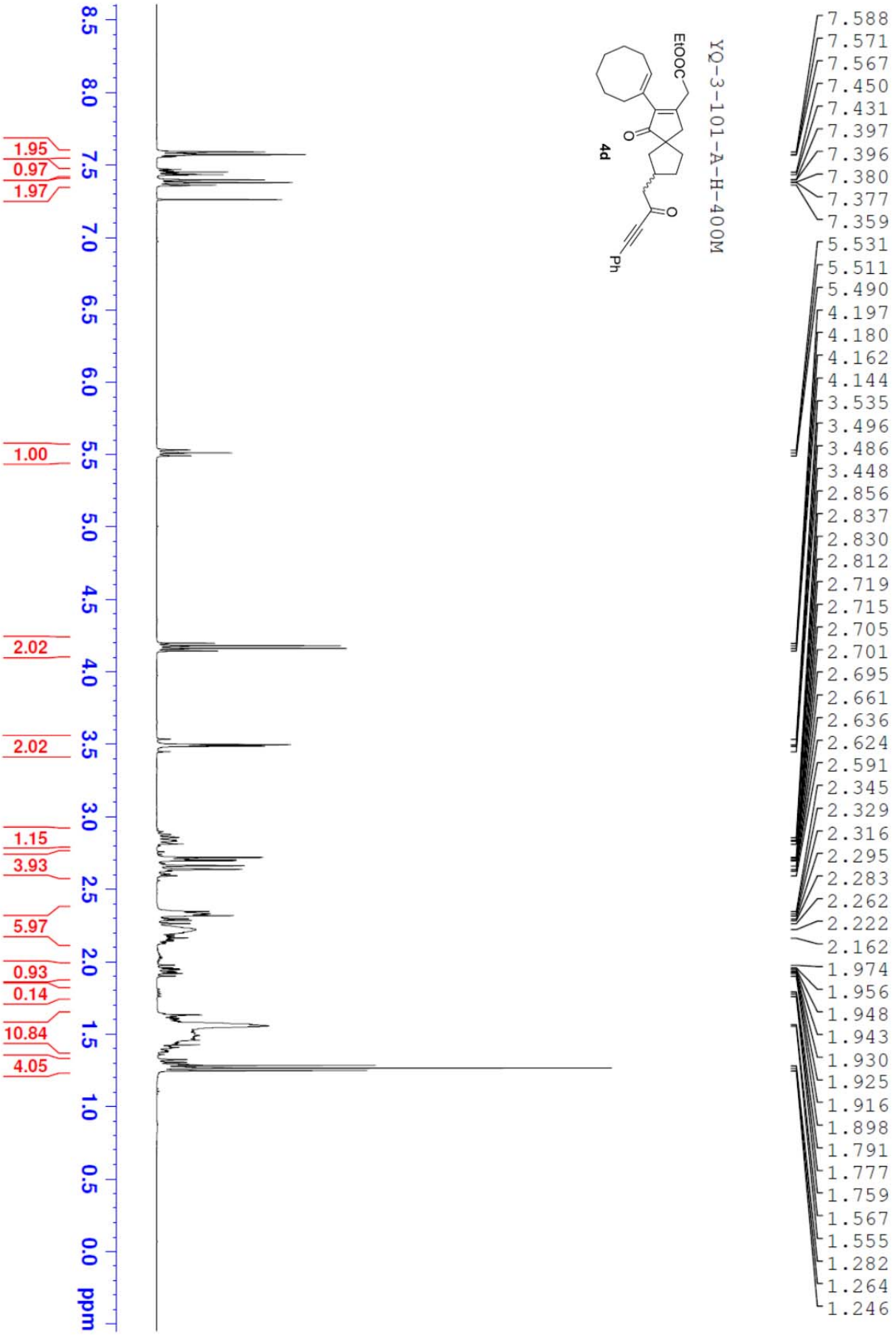

YQ-3-040B-C-100M
EtOOC
O
O
O
Ph
4a
211.09
187.21
169.21
161.40
143.38
137.20
133.10
130.71
128.60
119.90
117.11
91.11
87.84
77.31
77.00
76.68
61.24
54.15
51.78
48.26
43.21
38.44
37.11
35.86
33.08
21.86
14.13
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

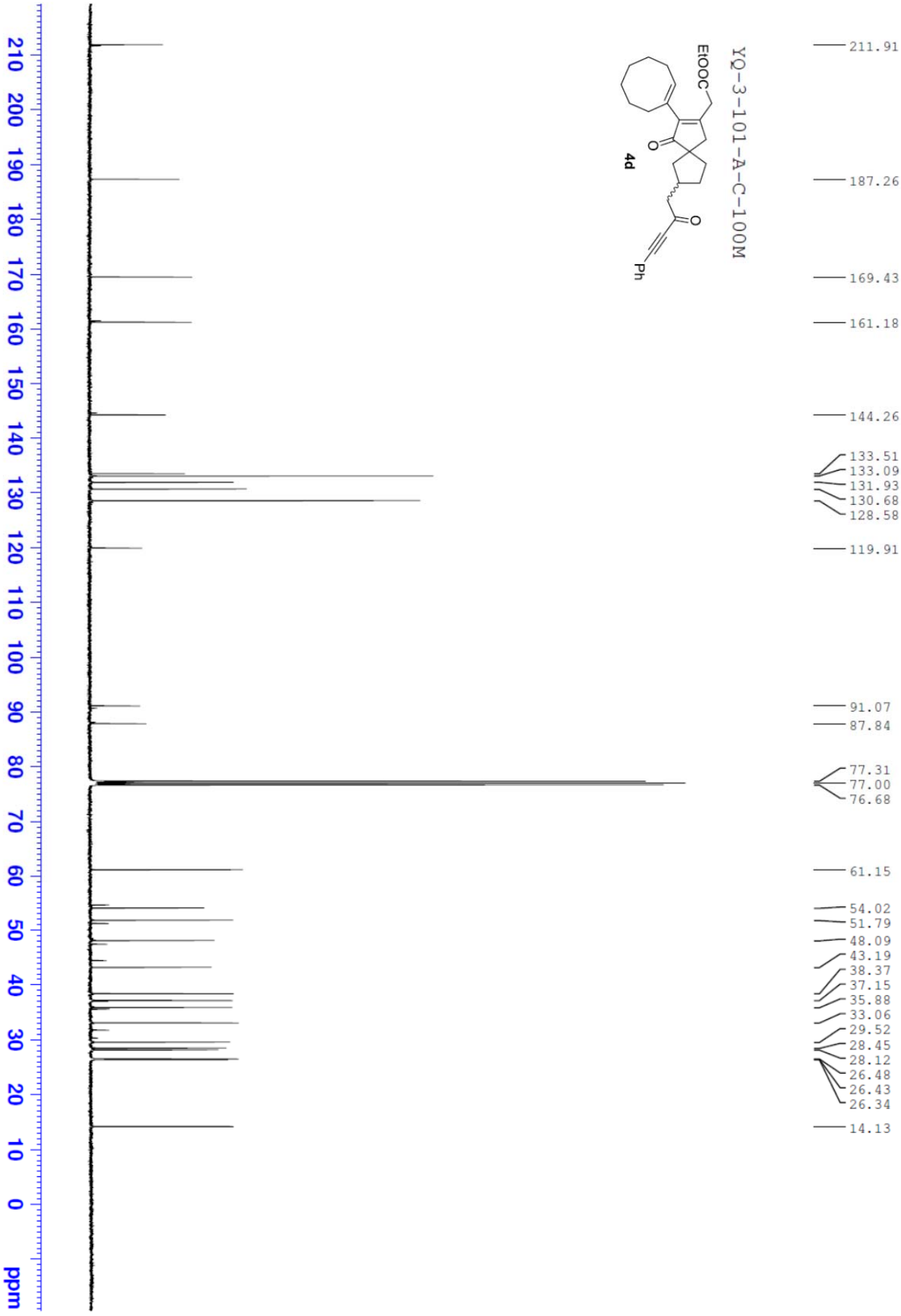

YQ-3-094-A-H-400M
EtOOC
O
O
O
Ph
4b
7.590
7.573
7.569
7.453
7.434
7.399
7.380
7.362
7.260
6.107
4.204
4.186
4.168
4.151
3.528
3.521
3.481
2.861
2.842
2.837
2.819
2.722
2.705
2.701
2.684
2.660
2.616
2.603
2.598
2.584
2.439
2.421
2.332
2.310
2.299
2.277
2.187
2.180
2.156
2.149
1.921
1.902
1.883
1.865
1.627
1.621
1.603
1.578
1.571
1.440
1.426
1.409
1.378
1.324
1.306
1.289
1.271
1.253
1.94
0.95
1.95
0.98
2.00
2.04
1.13
6.08
2.07
0.95
0.95
2.98
0.11
1.47
1.14
4.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

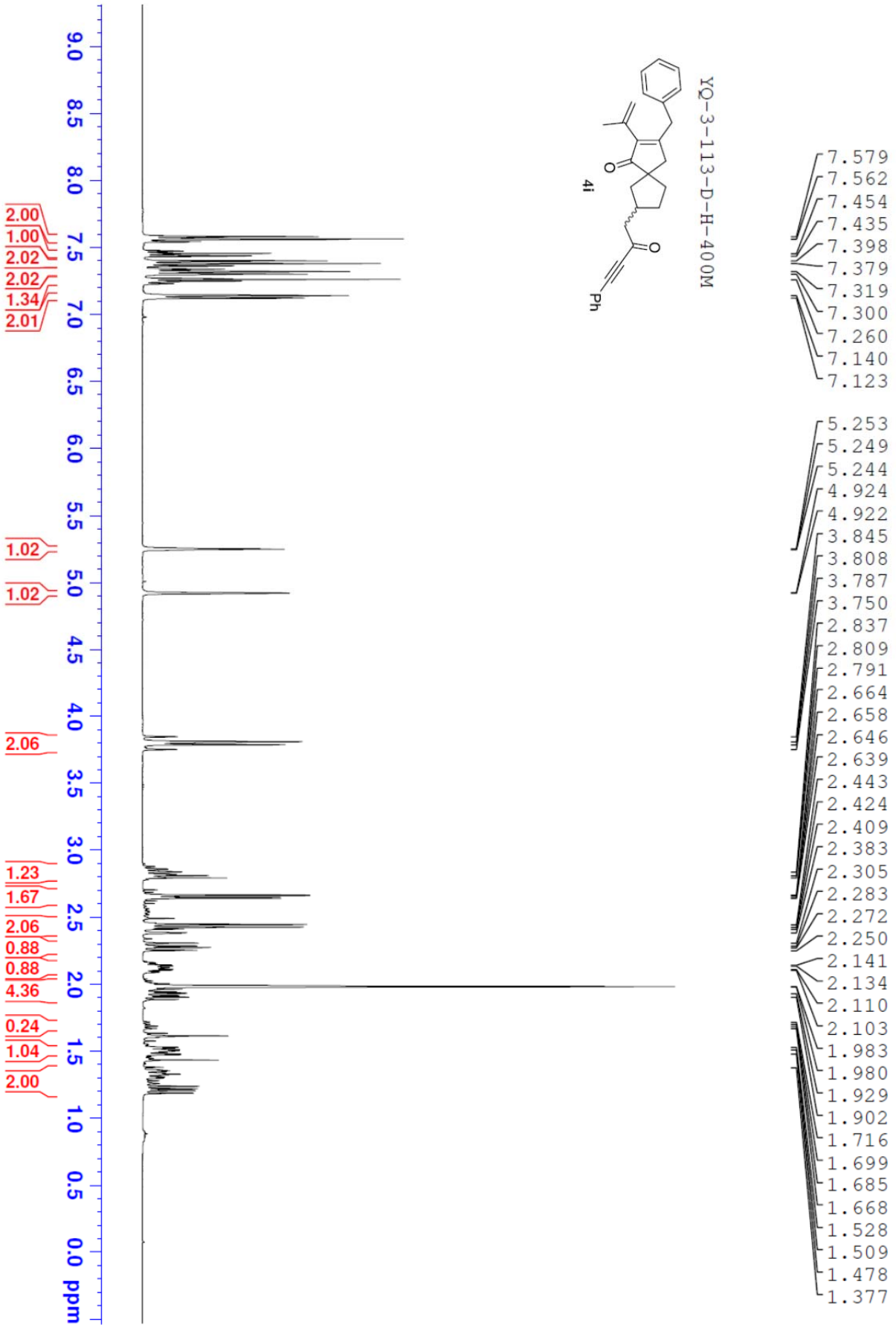

YQ-3-094-A-C-100M
EtOOC
O
O
Ph
4b
211.61
187.24
169.26
160.63
137.40
134.44
133.09
133.01
130.70
128.59
119.91
91.07
87.85
77.32
77.00
76.68
61.24
54.14
51.81
48.71
43.31
38.57
37.67
35.87
34.56
33.09
32.95
23.35
14.15
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

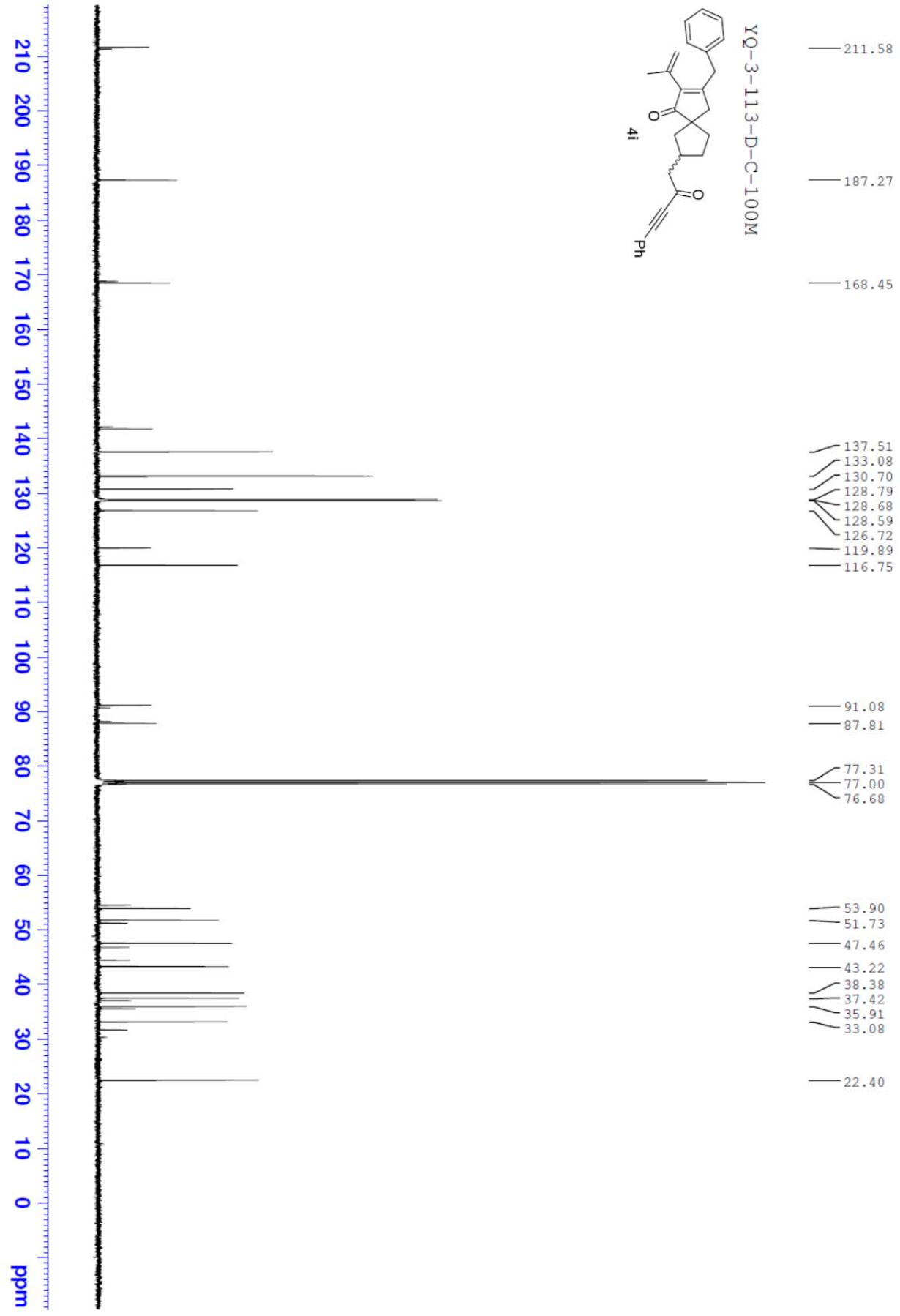

YQ-3-095-A-H-400M
EtOOC
O
O
Ph
4c
7.590
7.587
7.583
7.569
7.566
7.450
7.431
7.397
7.396
7.392
7.380
7.377
7.359
7.260
5.571
5.567
5.562
4.195
4.178
4.160
4.142
3.493
3.454
3.447
3.408
2.833
2.815
2.716
2.713
2.699
2.693
2.649
2.625
2.310
2.289
2.277
2.256
2.107
2.101
1.940
1.935
1.916
1.908
1.659
1.655
1.652
1.638
1.611
1.402
1.315
1.297
1.285
1.97
0.98
2.00
1.00
2.00
2.03
1.17
3.90
0.91
4.97
0.91
0.18
5.67
1.15
4.10
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

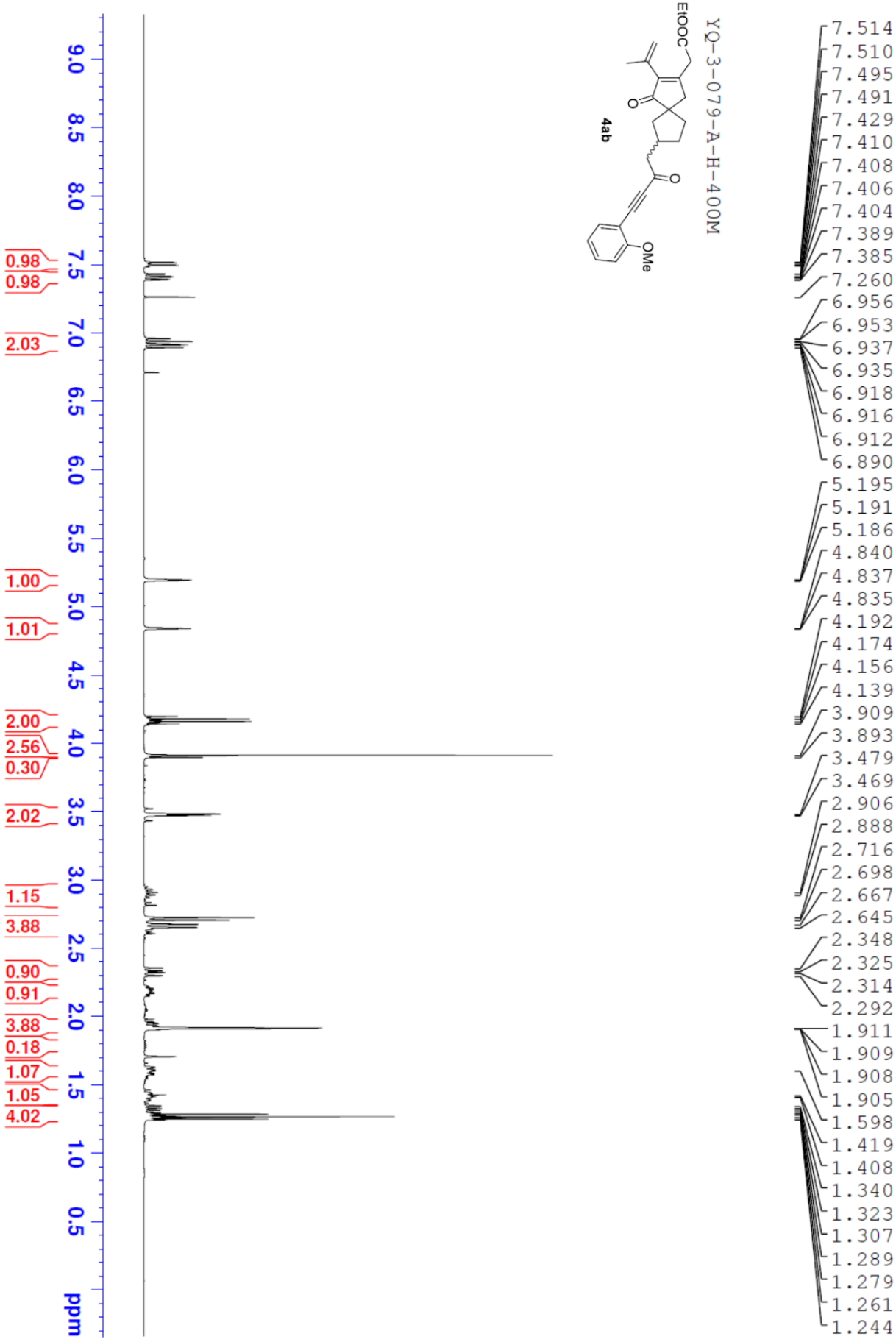

YQ-3-095-A-C-100M
EtOOC
O
O
Ph
4c
211.68
187.26
169.44
160.84
143.79
133.09
130.68
130.41
128.66
128.58
119.91
91.06
87.84
77.31
77.00
76.68
61.14
54.05
51.82
48.10
43.22
38.39
37.17
35.83
33.06
27.34
25.28
22.44
21.88
14.13
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

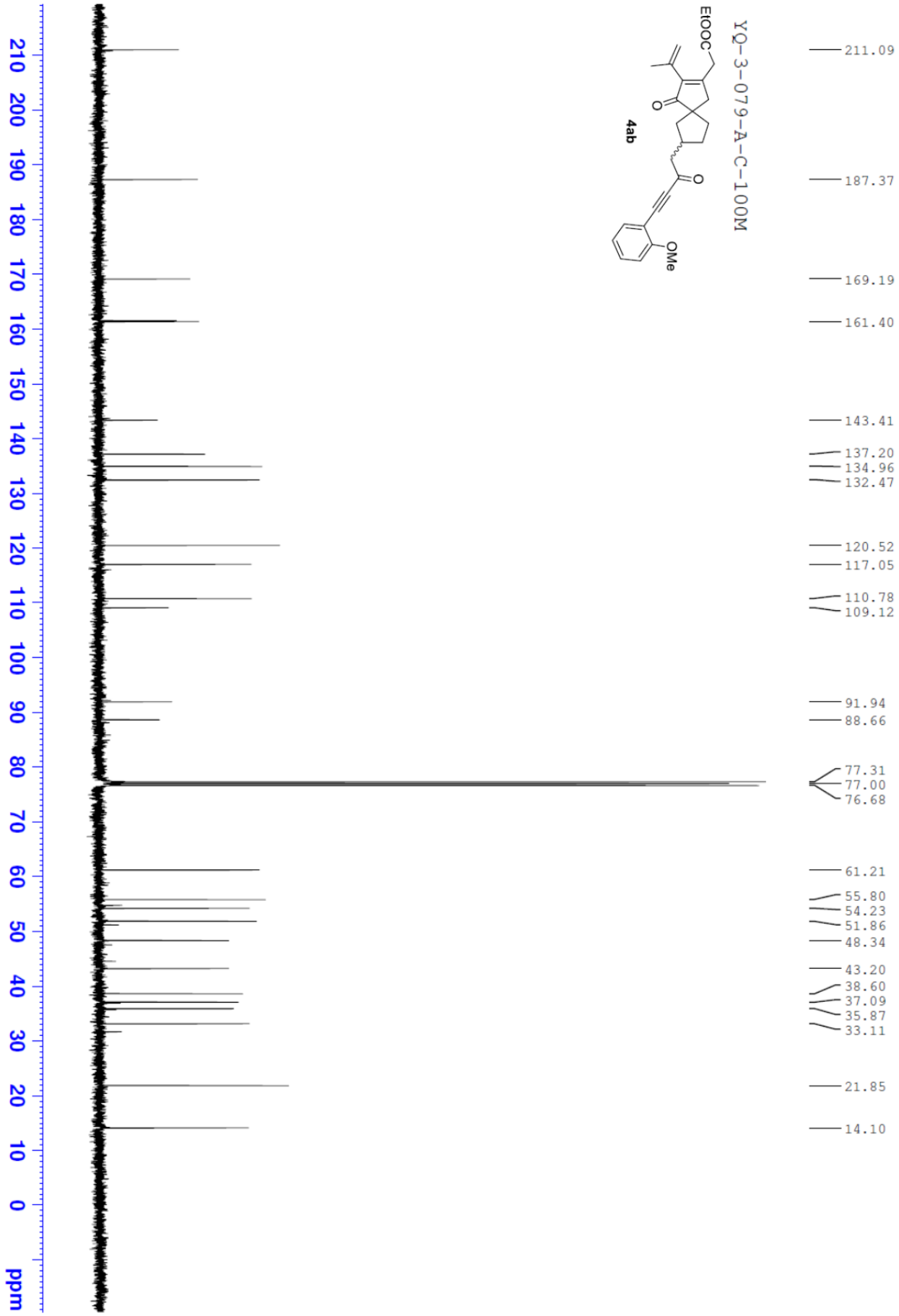

YQ-3-101-A-H-400M
EtOOC
O
O
Ph
4d
7.588
7.571
7.567
7.450
7.431
7.397
7.396
7.380
7.377
7.359
5.531
5.511
5.490
4.197
4.180
4.162
4.144
3.535
3.496
3.486
3.448
2.856
2.837
2.830
2.812
2.719
2.715
2.705
2.701
2.695
2.661
2.636
2.624
2.591
2.345
2.329
2.316
2.295
2.283
2.262
2.222
2.162
1.974
1.956
1.948
1.943
1.930
1.925
1.916
1.898
1.791
1.777
1.759
1.567
1.555
1.282
1.264
1.246
1.95
0.97
1.97
1.00
2.02
2.02
1.15
3.93
5.97
0.93
0.14
10.84
4.05
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
ppm

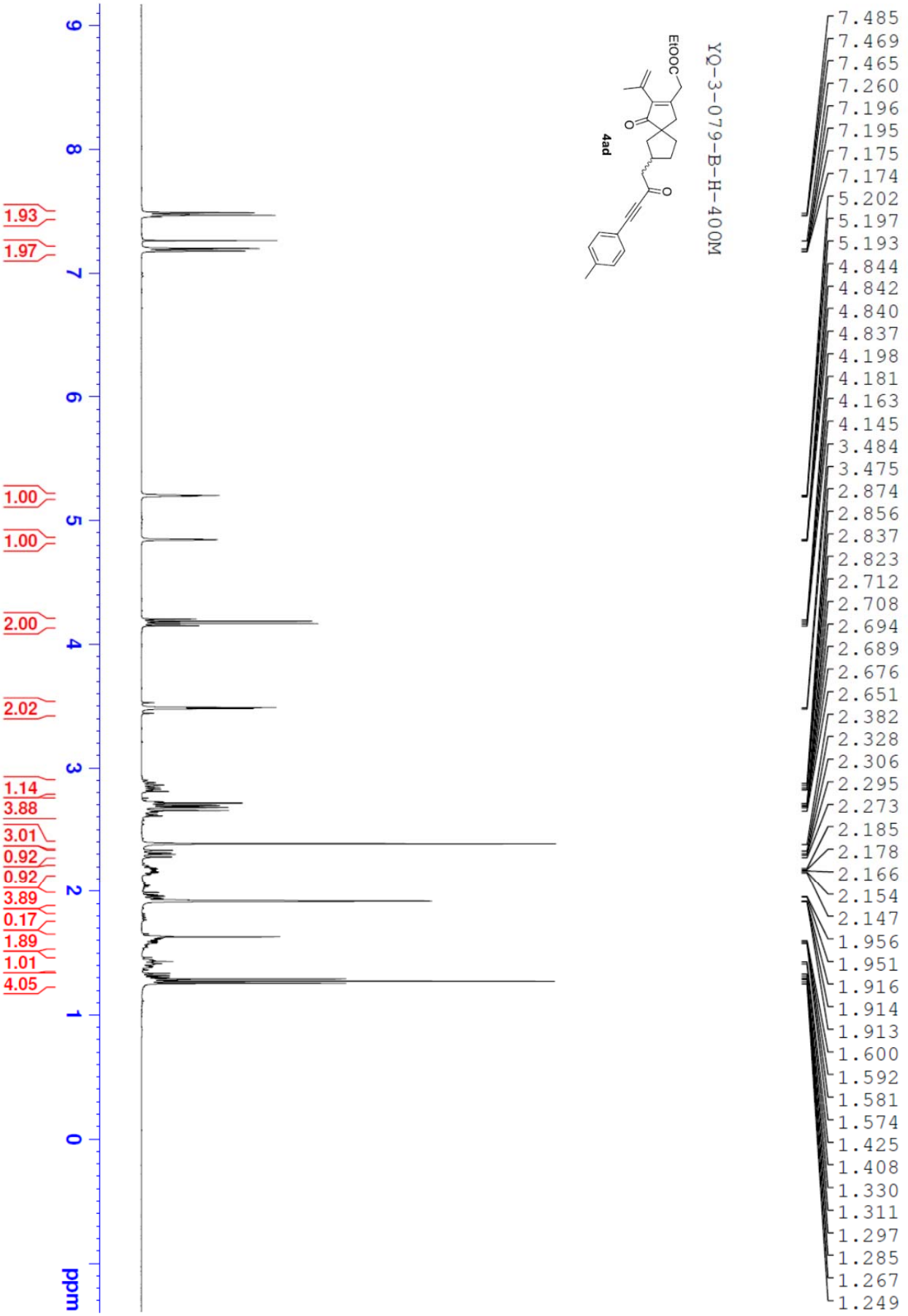

YQ-3-101-A-C-100M
EtOOC
O
O
Ph
4d
211.91
187.26
169.43
161.18
144.26
133.51
133.09
131.93
130.68
128.58
119.91
91.07
87.84
77.31
77.00
76.68
61.15
54.02
51.79
48.09
43.19
38.37
37.15
35.88
33.06
29.52
28.45
28.12
26.48
26.43
26.34
14.13
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

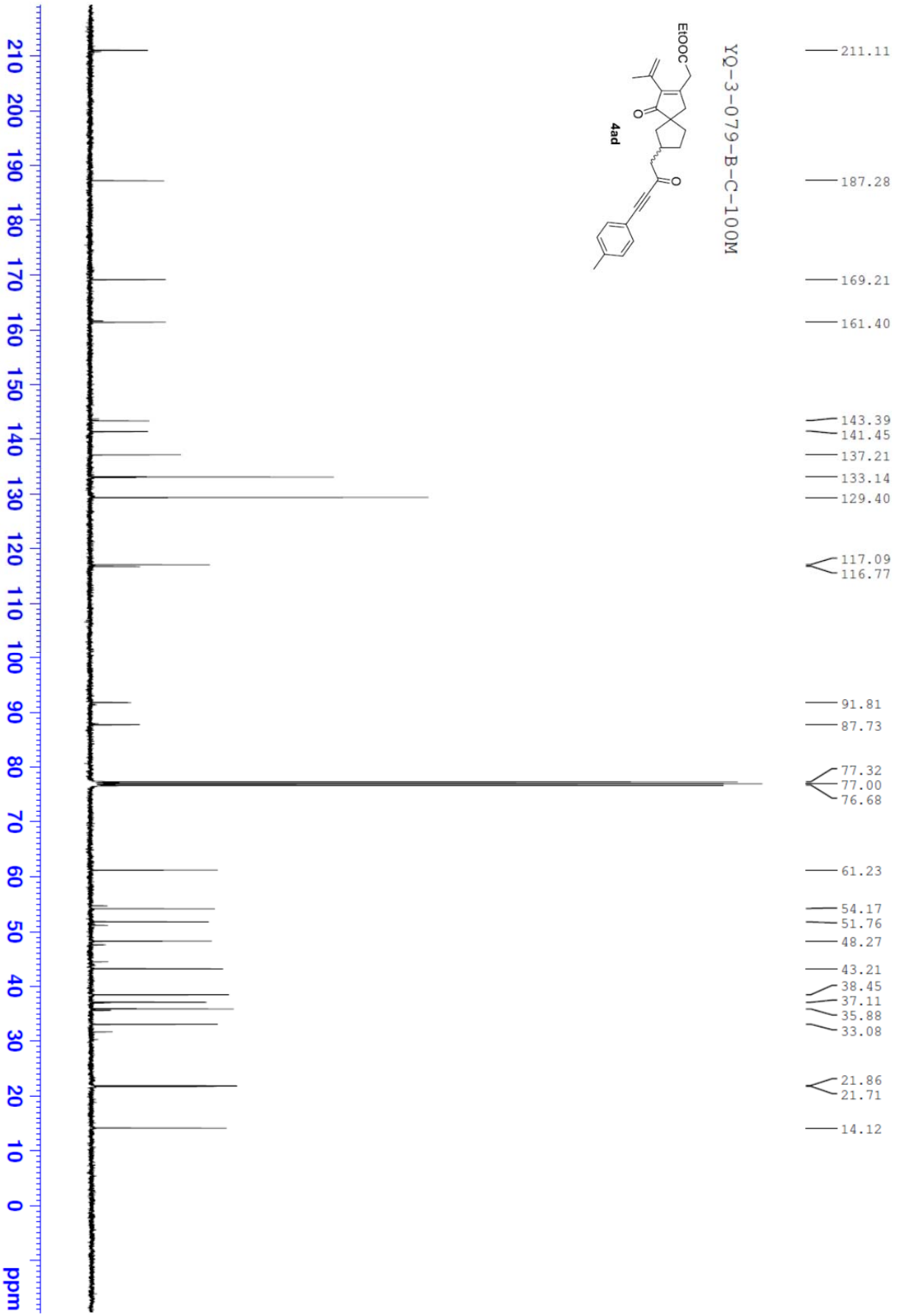

YQ-3-113-D-H-400M
4i
Ph
O
O
7.579
7.562
7.454
7.435
7.398
7.379
7.319
7.300
7.260
7.140
7.123
5.253
5.249
5.244
4.924
4.922
3.845
3.808
3.787
3.750
2.837
2.809
2.791
2.664
2.658
2.646
2.639
2.443
2.424
2.409
2.383
2.305
2.283
2.272
2.250
2.141
2.134
2.110
2.103
1.983
1.980
1.929
1.902
1.716
1.699
1.685
1.668
1.528
1.509
1.478
1.377
2.00
1.00
2.02
2.02
1.34
2.01
1.02
1.02
2.06
1.23
1.67
2.06
0.88
0.88
4.36
0.24
1.04
2.00
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
ppm

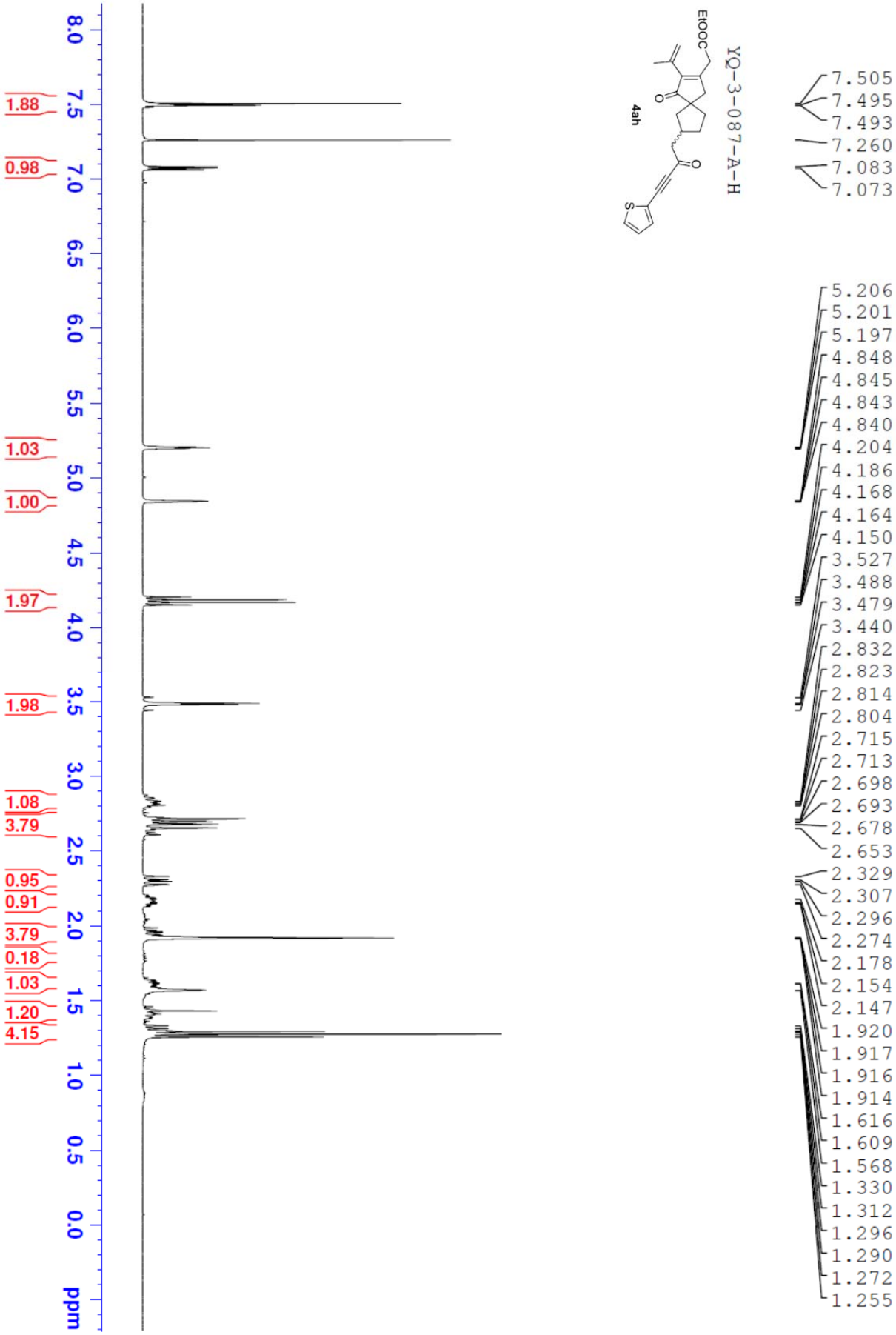

YQ-3-113-D-C-100M
4i
O
O
Ph
211.58
187.27
168.45
137.51
133.08
130.70
128.79
128.68
128.59
126.72
119.89
116.75
91.08
87.81
77.31
77.00
76.68
53.90
51.73
47.46
43.22
38.38
37.42
35.91
33.08
22.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


YQ-3-079-A-H-400M
EtOOC
O
O
OMe
4ab
7.514
7.510
7.495
7.491
7.429
7.410
7.408
7.406
7.404
7.389
7.385
7.260
6.956
6.953
6.937
6.935
6.918
6.916
6.912
6.890
5.195
5.191
5.186
4.840
4.837
4.835
4.192
4.174
4.156
4.139
3.909
3.893
3.479
3.469
2.906
2.888
2.716
2.698
2.667
2.645
2.348
2.325
2.314
2.292
1.911
1.909
1.908
1.905
1.598
1.419
1.408
1.340
1.323
1.307
1.289
1.279
1.261
1.244
0.98
0.98
2.03
1.00
1.01
2.00
2.56
0.30
2.02
1.15
3.88
0.90
0.91
3.88
0.18
1.07
1.05
4.02
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
ppm


YQ-3-079-A-C-100M
EtOOC
O
O
OMe
4ab
211.09
187.37
169.19
161.40
143.41
137.20
134.96
132.47
120.52
117.05
110.78
109.12
91.94
88.66
77.31
77.00
76.68
61.21
55.80
54.23
51.86
48.34
43.20
38.60
37.09
35.87
33.11
21.85
14.10
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm


YQ-3-079-B-H-400M
EtOOC
4ad
7.485
7.469
7.465
7.260
7.196
7.195
7.175
7.174
5.202
5.197
5.193
4.844
4.842
4.840
4.837
4.198
4.181
4.163
4.145
3.484
3.475
2.874
2.856
2.837
2.823
2.712
2.708
2.694
2.689
2.676
2.651
2.382
2.328
2.306
2.295
2.273
2.185
2.178
2.166
2.154
2.147
1.956
1.951
1.916
1.914
1.913
1.600
1.592
1.581
1.574
1.425
1.408
1.330
1.311
1.297
1.285
1.267
1.249
1.93
1.97
1.00
1.00
2.00
2.02
1.14
3.88
3.01
0.92
0.92
3.89
0.17
1.89
1.01
4.05
ppm


YQ-3-079-B-C-100M
EtOOC
O
O
4ad
211.11
187.28
169.21
161.40
143.39
141.45
137.21
133.14
129.40
117.09
116.77
91.81
87.73
77.32
77.00
76.68
61.23
54.17
51.76
48.27
43.21
38.45
37.11
35.88
33.08
21.86
21.71
14.12
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


YQ-3-087-A-H
4ah
EtOOC
O
O
S
7.505
7.495
7.493
7.260
7.083
7.073
5.206
5.201
5.197
4.848
4.845
4.843
4.840
4.204
4.186
4.168
4.164
4.150
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6.5
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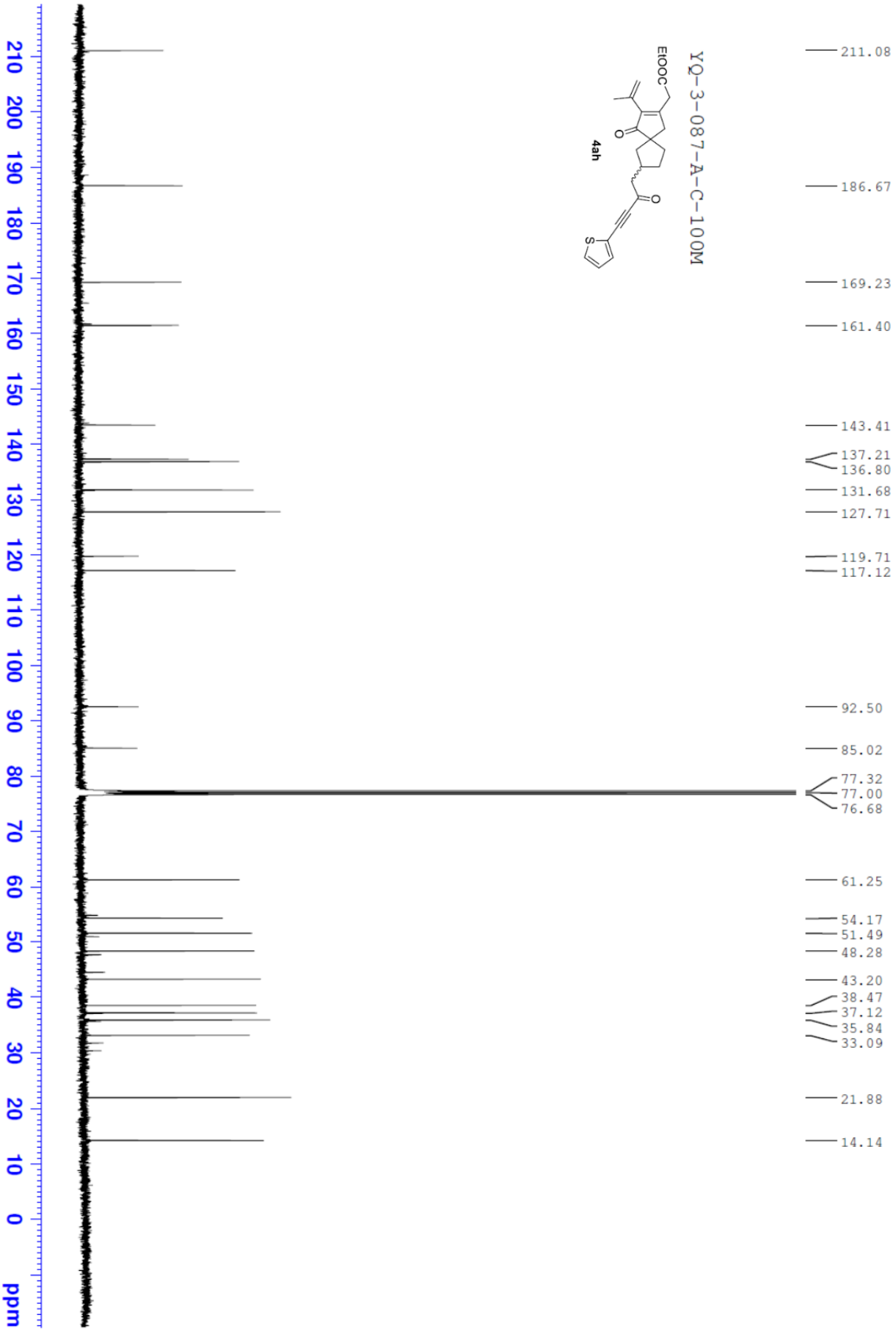
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131.68
127.71
119.71
117.12
92.50
85.02
77.32
77.00
76.68
61.25
54.17
51.49
48.28
43.20
38.47
37.12
35.84
33.09
21.88
14.14
210
200
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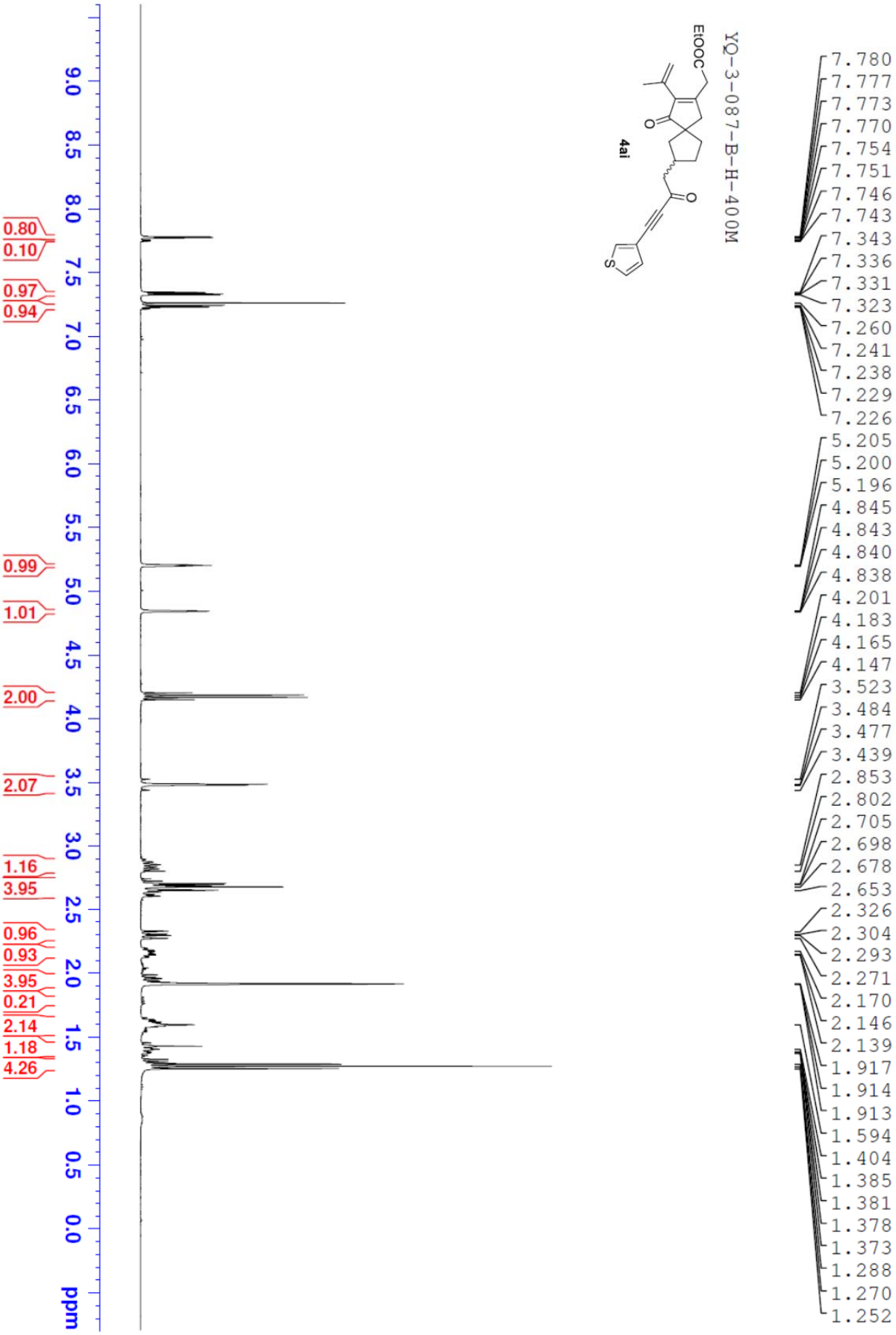
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7.751
7.746
7.743
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7.226
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4.147
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2.705
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0.99
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2.00
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3.95
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0.93
3.95
0.21
2.14
1.18
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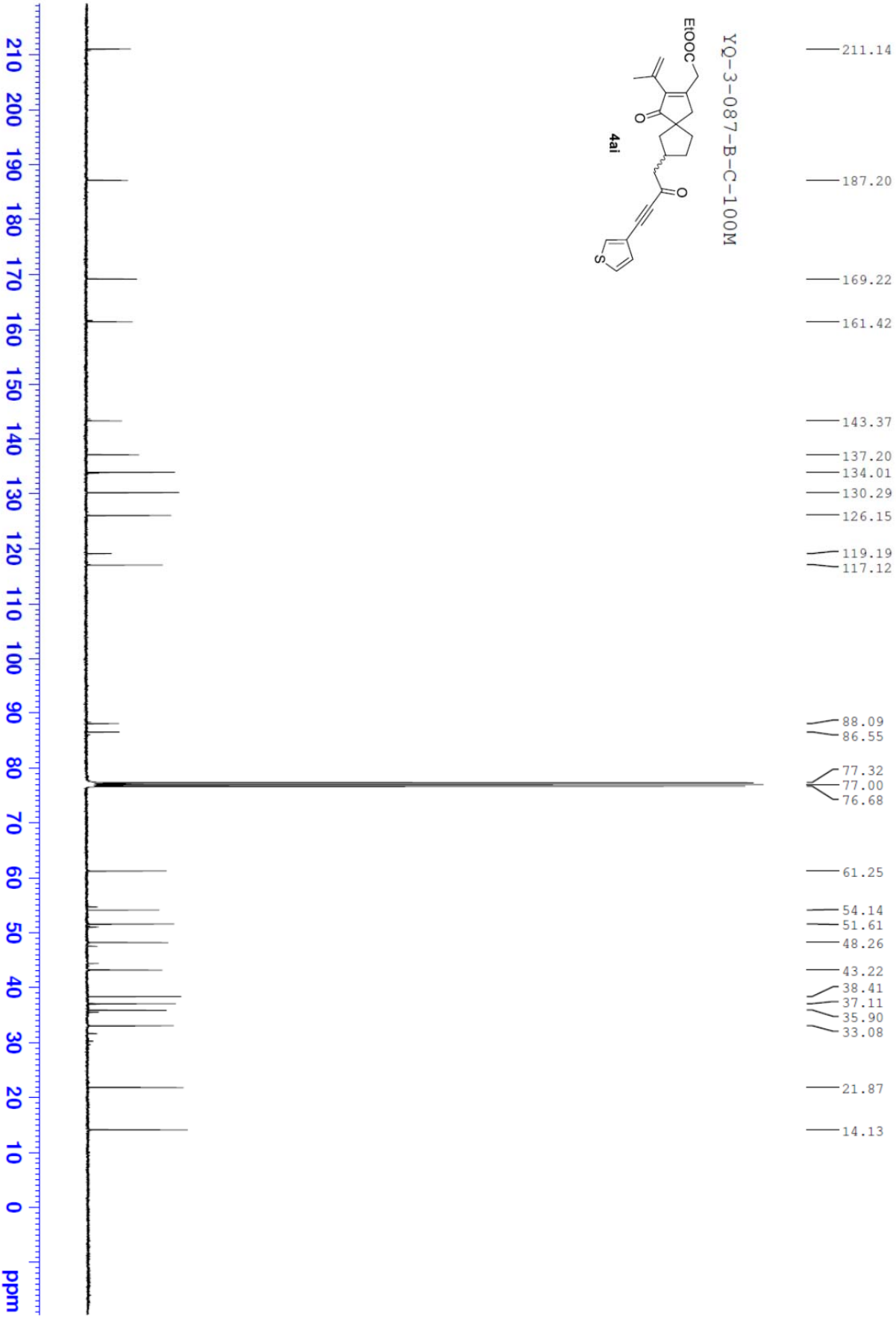
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O
S
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126.15
119.19
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77.00
76.68
61.25
54.14
51.61
48.26
43.22
38.41
37.11
35.90
33.08
21.87
14.13
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm