

Supporting Information

Diastereoselective 1,2-Difunctionalization of 1,3-Enynes Enabled by Merging Photoexcited Hantzsch Ester with Chromium Catalysis

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05 December 2023 - Note added after first publication: This Supplementary Information file replaces that originally published on 20 October 2023. The spectra for compounds 4a-4z, 4aa-4ac, 5 and 7a-7b have been updated with the correct data.

1. General remarks

Unless otherwise noted, the commercially available reagents were used directly without further purification. All reactions were performed under nitrogen gas atmosphere. Anhydrous solvents were directly purchased from Energy Chemical and stored under nitrogen gas. Thin layer chromatography (TLC) was performed using Huanghai TLC silica gel plates HSGF254 and visualized using UV light or potassium permanganate (KMnO_4). Flash column chromatography was performed using 200-300 mesh silica gel. The ^1H NMR, ^{13}C NMR and ^{19}F NMR spectra were recorded on a Bruker AV-400 MHz with CDCl_3 (7.26 ppm for ^1H NMR, 77.0 ppm for ^{13}C NMR) as solvent. Data are reported as follows: chemical shift δ/ppm , integration (^1H only), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or combinations thereof; ^{13}C signals are singlets unless otherwise stated), coupling constants J in Hz, assignment. High Resolution Mass Spectrometry (HRMS) were recorded on HPLC-Q-TOF mass spectrometer. The measured HRMS values are reported to 4 decimal places of the calculated value. The UV-Vis data were obtained from HP 8453 spectrometer.

2. Photochemical reaction setup

Household blue LED strips (30 W, 400-410 nm) were coiled around the inside of an aluminium ware with 15 cm diameter (Figure S1, left). The maximum emission of the LED strips at 405 nm (Figure S1, right). The Quartz glass was used as reaction vessel at room temperature.

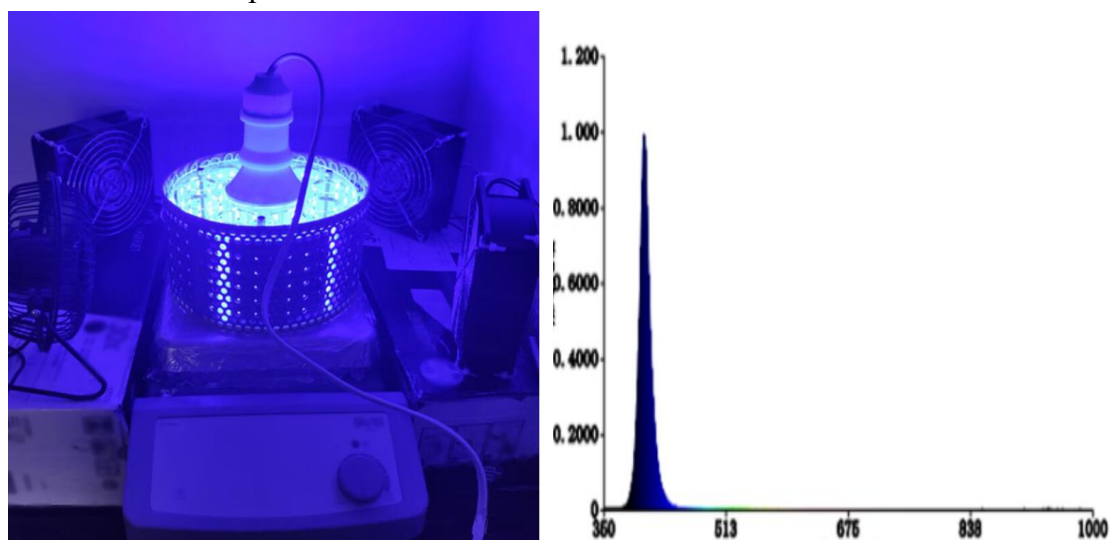
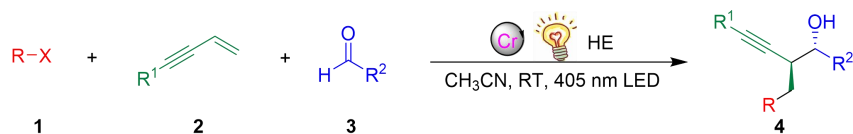


Figure S1. Reaction setup (left) and the emission spectra (right)

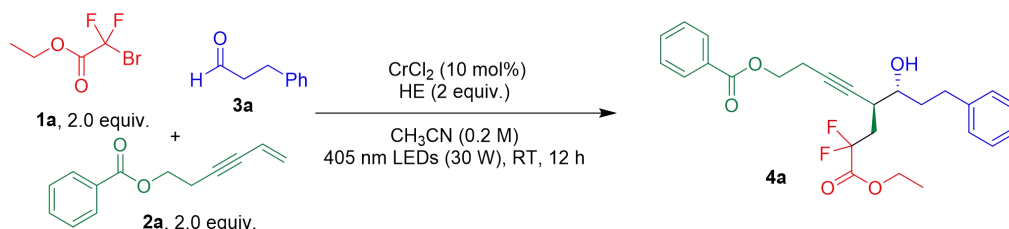
3. General procedures of the reaction



An oven-dried Schlenk tube equipped with an stirring bar was charged under nitrogen gas with the corresponding alkyl halides (**1**, 2.0 equiv.), 1,3-enyne (**2**, 2.0 equiv.), aldehyde (**3**, 0.2 mmol), Hantzsch ester (HE, 2.0 equiv.), $CrCl_2$ (10 mol%), and CH_3CN (0.2 M, 1.0 mL). The reaction mixture was then degassed by freeze-pump-thaw for three times, then the vessel was back-filled with nitrogen gas. The substrate was added after degassed when it's a liquid. Subsequently, the reaction was stirred at room temperature under blue LEDs irradiation (30 W, 400-410 nm) for 12 h. The reaction solution was concentrated in vacuo and filtered through a pad of silica gel by washing with EtOAc. The obtained crude reaction mixture was then concentrated for 1H NMR spectroscopy to determine the diastereomeric ratio. The mixture solution was purified by column chromatography on silica gel (pentane/EtOAc) to afford the corresponding compounds.

All 1,3-enynes were prepared from the alkynes and vinyl bromide according to the previous reports.^[1, 2]

4. The procedure of large-scale



Based on the general procedure, 3-phenylpropionaldehyde **3a** (10 mmol, 1.34 g), 1,3-enyne **2a** (20 mmol), and ethyl bromodifluoroacetate **1a** (20 mmol), Hantzsch ester (HE, 20 mmol), $CrCl_2$ (1 mmol), and CH_3CN (0.2 M, 50 mL) were stirred at room temperature under blue LEDs irradiation (30 W, 400-410 nm) for 12 h. The reaction solution was concentrated in vacuo and filtered through a pad of silica gel by washing with EtOAc. The obtained crude reaction mixture was then concentrated for 1H NMR spectroscopy to determine the diastereomeric ratio. The mixture solution was purified by column chromatography on silica gel (pentane/EtOAc) to afford the corresponding compounds **4a** (3.57 g, 78%).

5. Mechanistic investigation

5.1 UV-visible spectra absorption analysis

A variety of solution of $BrCF_2COOEt$, HE, and HE+ $BrCF_2COOEt$ with different concentration in CH_3CN were prepared. Then the UV-visible absorption spectra of a series of solutions were recorded on HP 8453 spectrometer.

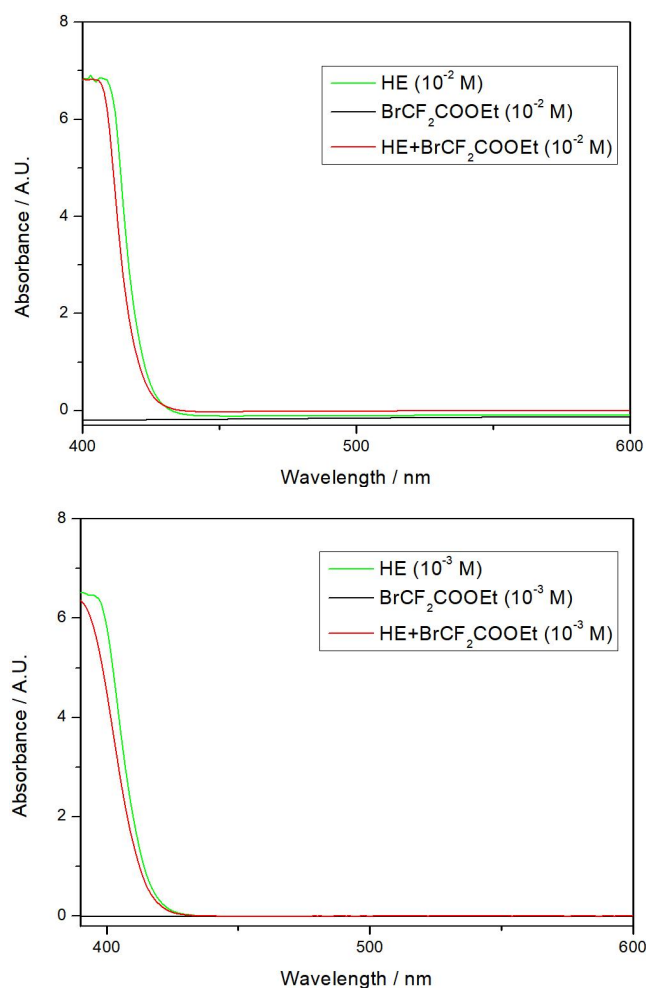
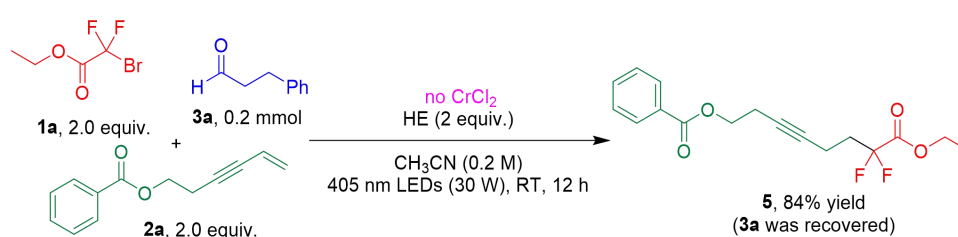


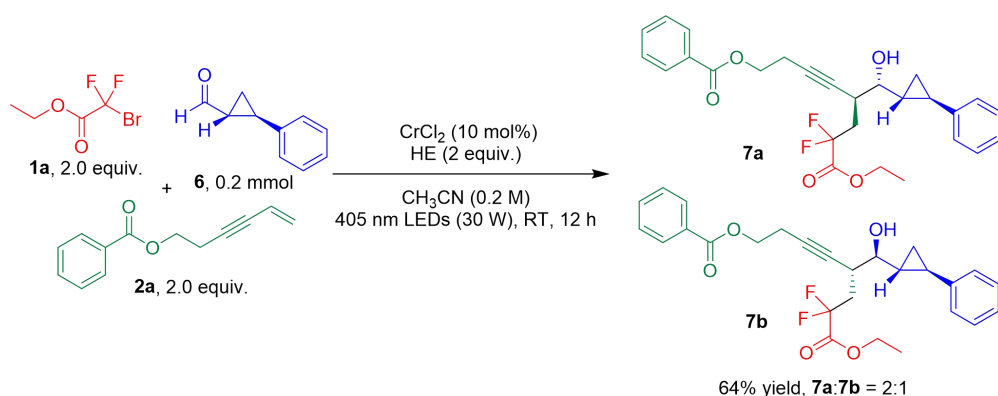
Figure S2. UV-visible absorption spectra of BrCF₂COOEt, HE, and HE+BrCF₂COOEt in CH₃CN

5.2 Radical probing experiment I



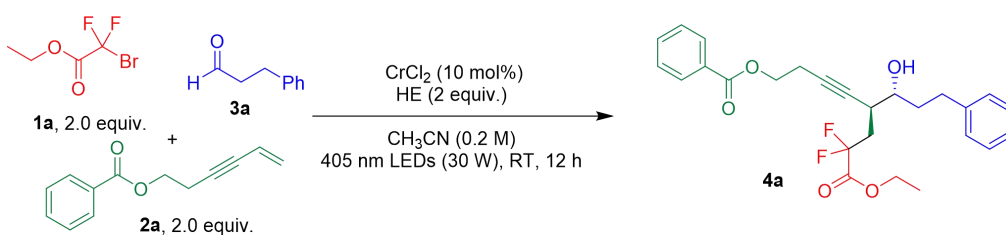
According to the general procedure, the reaction was equipped with 3-phenylpropionaldehyde **3a**, 1,3-enyne **2a**, and ethyl bromodifluoroacetate **1a**, Hantzsch ester (HE, 2.0 equiv.), and CH₃CN (0.2 M, 1.0 mL) and stirred at room temperature under blue LEDs irradiation for 12 h. The reaction solution was concentrated in vacuo and then purified by column chromatography on silica gel (pentane/EtOAc = 50:1) to afford **5** as a colorless oil.

5.3 Radical probing experiment II



2-phenylcyclopropane-1-carboxaldehyde **6** was used to further explore the reaction mechanism. An oven-dried Schlenk tube equipped with a stirring bar was charged under nitrogen gas with the corresponding **6**, 1,3-enyne **2a**, and ethyl bromodifluoroacetate **1a**, Hantzsch ester (HE, 2.0 equiv.), CrCl_2 (10 mol%), and CH_3CN (0.2 M, 1.0 mL) and stirred at room temperature under blue LEDs irradiation for 12 h. The reaction solution was concentrated in vacuo and then purified by column chromatography on silica gel (pentane/EtOAc = 10:1) to afford colorless oil **7a** and **7b** in 2:1 ratio.

5.4 Light on/off experiment



The light on/off experiment of the model reaction was performed parallelly on a 1.0 mmol scale according to the general procedure by adding 1.0 equiv. 1,3,5-trimethoxybenzene as internal standard. The reaction started with successive irradiation and black periods to study the influence of continuous irradiation of the visible-light for the progress photochemical reaction. After being irradiated with 30 W blue LEDs for 1 h, an aliquot (200 μL) from the reaction mixture was transferred into a flask and then concentrated. The yield of the desired product **4a** was determined by ^1H NMR. The reaction was then stirred with light-off for 1 h and the yield was determined by ^1H NMR. All of the following yields were analyzed in the identical way after a 1 h light on or light-off. These results revealed that light is a necessary component of the reaction.

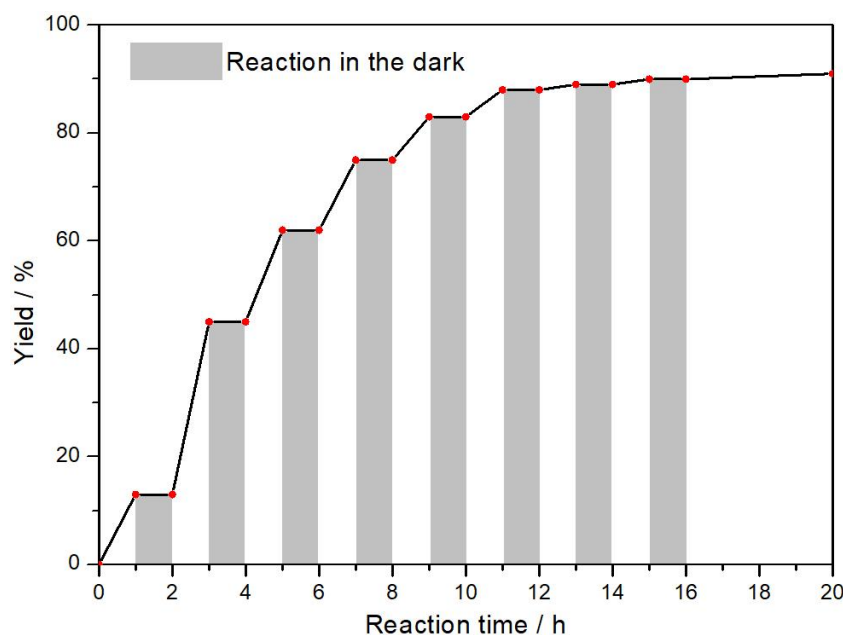


Figure S3. Time profile of the transformation with light on/off over time

5.5 Quantum yield calculation^[4]

(A) Determination of light intensity at 436 nm:

0.737 g of potassium ferrioxalate trihydrate was dissolved in 10 mL H₂SO₄ (0.05 M) and stored in the dark. Then, a buffer solution was prepared by dissolving 2.25 g of sodium acetate and 10 mg of phenanthroline in 10 mL of H₂SO₄ (0.5 M).

General protocol to assess the photon flux of the 436 nm blue LEDs: 3.0 mL of the ferrioxalate solution was placed in a cuvette containing a stirring bar and irradiated for 90.0 seconds at $\lambda = 436$ nm with an emission slit width at 10.0 nm. After irradiation, 0.525 mL of the phenanthroline solution was added to the cuvette. The solution was then allowed to stir for 1.0 h in the dark to allow the ferrous ions to completely coordinate to the phenanthroline. The absorbance of this solution was then measured at 510 nm by UV-vis spectrophotometry. The number of moles of Fe²⁺ produced by light irradiation is obtained by:

$$n(\text{Fe}^{2+}) = \frac{V \times \Delta A}{l \times \epsilon} \quad (1)$$

Where:

V is the total volume (0.003525 L) of the solution after addition of phenanthroline;

ΔA is the difference in absorbance at 510 nm between the irradiated and non-irradiated solutions;

l is the path length (1.000 cm);

ϵ is the molar absorptivity at 510 nm (11,100 L mol⁻¹ cm⁻¹).

The photon flux (F) is obtained by using the following equation:

$$F = \frac{n(\text{Fe}^{2+})}{\Phi \times t \times f} \quad (2)$$

Where:

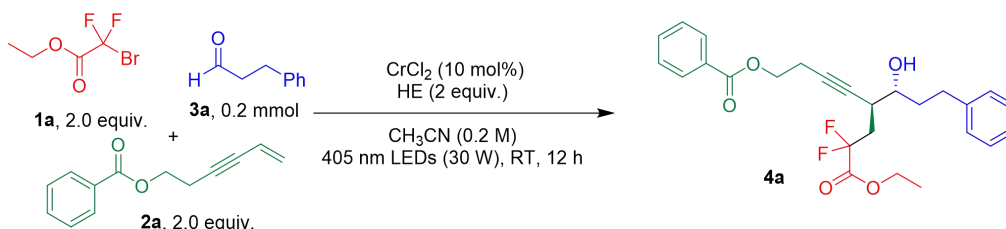
Φ is the quantum yield for the ferrioxalate actinometer (1.01 for a 0.15 M solution at $\lambda = 436$ nm);

t is the time (90.0 s);

f is the fraction of light absorbed at $\lambda = 436$ nm.

The photon flux was calculated to be 1.58×10^{-9} einstein s^{-1} .

(B) Quantum yield calculation of the reaction:



Following the standard procedure, an oven-dried Schlenk tube equipped with an stirring bar was charged under nitrogen gas with the corresponding alkyl halides (**1**, 2.0 equiv.), 1,3-enyne (**2**, 2.0 equiv.), aldehyde (**3**, 0.2 mmol), Hantzsch ester (HE, 2.0 equiv.), CrCl₂ (10 mol%), and CH₃CN (0.2 M, 1.0 mL). The reaction mixture was then degassed by freeze-pump-thaw for three times, then the vessel was back-filled with nitrogen gas. The substrate was added after degassed when it's a liquid. Subsequently, the reaction was stirred at room temperature under blue LEDs irradiation ($\lambda = 405$ nm, slit width = 10.0 nm) for 4 h. After irradiation, the yield was determined by ¹H NMR analysis using 1,3,5-trimethoxybenzene as an internal standard. The yield was determined to be 1.8% (3.6×10^{-6} mol). The reaction quantum yield (Φ) was determined using eq. 3 where the photon flux is 1.58×10^{-9} einsteins s^{-1} (determined by actinometry as described above), t is the reaction time (14400 s) and f is the fraction of incident light absorbed by the reaction mixture ($f = 1 - 10^{-A}$, 0.996).

$$\Phi = \frac{n(\text{product})}{F \times t \times f} \quad (3)$$

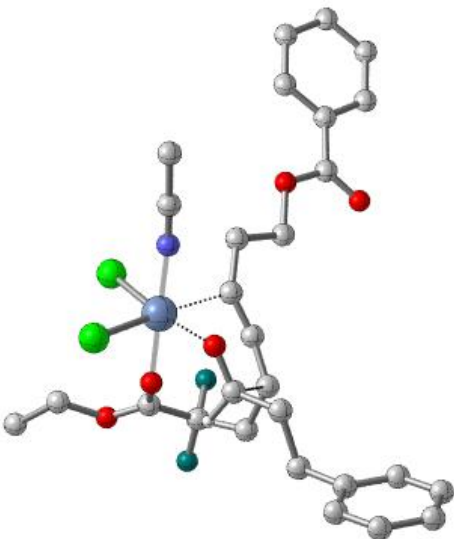
The reaction quantum yield (Φ) was thus determined to be $\Phi = 0.16$.

6. DFT calculations

Computational Methods: All of the density functional theory (DFT) calculations were performed with the Gaussian 16^[5] series of programs. The B3LYP functional^[6] including Grimme empirical dispersion correction (GD3BJ)^[7] with the standard 6-31G(d) basis set (SDD basis set for Cr) was used for the geometry optimizations in the gas phase. Harmonic vibrational frequency calculations were performed for all of the stationary points to determine whether they are transition structures and to derive the thermochemical corrections for free

energies. The M06L functional^[8] proposed by Truhlar et al. with the 6-311+G(d,p) basis set (SDD basis set for Cr) was used to calculate the single-point energies. The solvent effect was considered by applying the implicit SMD^[9] solvation model with MeCN as solvent in single-point energy calculations. The 3D images of the calculated structures were prepared using CYLview.^[10]

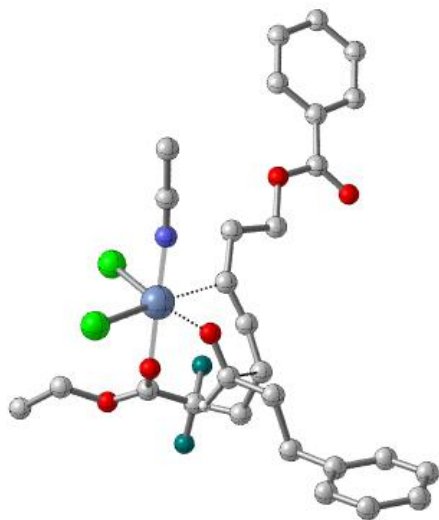
Cartesian (Å) Coordinates and Energies of Optimized Structures.



Cr	0.59783400	-1.56806900	1.24741900	C	4.34144500	1.95562400	-0.41542800
Cl	2.00632700	-2.80719100	2.53407700	H	3.84486100	2.01278000	-1.39111700
C	1.33525400	0.70244700	-1.40287400	H	5.05077700	1.11990800	-0.47072700
C	0.18479400	0.35972100	-0.77283500	H	3.05331300	-0.42531200	-0.10079200
C	-0.62215100	-0.06338700	0.10692100	Cl	-1.00347900	-3.11990800	0.75234600
C	2.57292700	0.35227500	0.49704500	C	-2.08685900	0.04063300	0.36341900
O	1.73707600	0.00812000	1.38801300	H	-2.25765600	0.38499900	1.39036800
H	1.59779100	1.75856900	-1.41341000	H	-2.53950100	-0.95288800	0.29232800
C	1.93265100	-0.09501800	-2.55020000	C	-2.75548500	0.99955900	-0.61210400
H	1.78178800	0.42846800	-3.50335900	H	-2.64172600	0.65770300	-1.64535000
H	3.01980800	-0.18780300	-2.42361900	H	-2.33816600	2.00790700	-0.54149400
C	1.38896000	-1.49751600	-2.78677200	C	1.56234500	-2.49932100	-1.62770200
C	3.30298900	1.65898700	0.67840600	O	1.60541800	-2.13249800	-0.46064000
H	3.80785400	1.60792600	1.65397700	O	1.65084700	-3.74079600	-2.01804600
H	2.57603400	2.47553500	0.76158800	C	1.73541100	-4.75726400	-0.95332800
				H	0.80848200	-4.69721800	-0.38043800
				H	2.56284800	-4.47781400	-0.29663100
				C	1.93629400	-6.09126200	-1.63537600
				H	1.09638500	-6.32390600	-2.29694600
				H	2.85992900	-6.10384400	-2.22250900
				F	0.04682500	-1.45555000	-3.05320500
				F	2.00421200	-2.00771500	-3.89473300
				C	-0.95086400	-0.77931500	3.86503700
				N	-0.40054000	-1.01117700	2.87764400
				C	-1.64345900	-0.50712200	5.11569300
				H	-1.11271600	-0.98621200	5.94427100
				H	-2.66456300	-0.89838900	5.07103400
				H	-1.68206100	0.57177000	5.29459600
				C	5.08335100	3.24455600	-0.14983200
				C	6.23955800	3.25549900	0.63954700
				C	4.59906200	4.46053300	-0.64644200
				C	6.89706300	4.45194400	0.92604300
				H	6.62820500	2.31739900	1.02948000
				C	5.25280600	5.65980800	-0.36274500
				H	3.70334800	4.46600000	-1.26392300
				C	6.40481500	5.65855600	0.42542000

TS-Re
 E(UB3LYP) = -2723.157401 Hartree
 E(UM06L) = -2723.414443 Hartree
 Thermal Correction to Energy = 0.575420 Hartree
 Thermal Correction to Enthalpy = 0.576365 Hartree
 Thermal Correction to Free Energy = 0.446891 Hartree

H	7.79562800	4.44140300	1.53668400	C	1.79158100	0.02837300	-2.17808400
H	4.86505600	6.59367200	-0.76021200	O	1.01915200	1.00269200	-1.91479600
H	6.91738800	6.59073300	0.64499200	H	0.85035200	-2.32546100	-1.95607700
O	-4.15562000	1.03782900	-0.27430100	C	1.77590700	-2.39865900	0.03779500
C	-4.90262600	1.93239900	-0.95737000	H	1.60628400	-3.48270300	0.08195500
O	-4.44270400	2.68179800	-1.79764800	H	2.83502600	-2.26947200	-0.21279100
C	-6.33697500	1.89233300	-0.56012600	C	1.61323600	-1.92437800	1.47544500
C	-6.82508000	0.98255500	0.38694100	Cl	-0.59521400	2.13772700	1.80648800
C	-7.21006200	2.80177900	-1.17031500	C	-2.41513600	0.17085800	-0.16154800
C	-8.17837900	0.98694700	0.71936200	H	-2.78195800	1.04743500	-0.70738100
H	-6.14375200	0.27849400	0.85028700	H	-2.53524900	0.40430900	0.90111500
C	-8.56109600	2.80405200	-0.83432500	C	-3.24812800	-1.04780900	-0.53576000
H	-6.80794200	3.49372800	-1.90241500	H	-2.94285200	-1.92916700	0.03653300
C	-9.04656800	1.89659000	0.11095700	H	-3.16083300	-1.28846900	-1.59891600
H	-8.55718900	0.27954800	1.45144100	C	1.90908700	-0.43360800	1.73201100
H	-9.23647500	3.51031200	-1.30838600	O	1.69951000	0.41920700	0.88057600
H	-10.10123800	1.89715900	0.37203100	O	2.39852000	-0.18765200	2.91676800
H	2.00063900	-6.87383300	-0.87219500	C	2.63965200	1.23004000	3.23923900



TS-Si

E(UB3LYP) = -2723.149836 Hartree

E(UM06L) = -2723.406769 Hartree

Thermal Correction to Energy = 0.575419 Hartree

Thermal Correction to Enthalpy = 0.576363 Hartree

Thermal Correction to Free Energy = 0.447940 Hartree

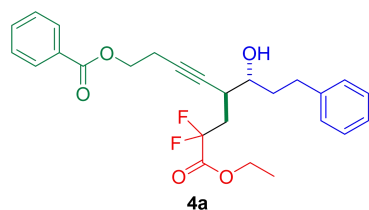
Cr	0.48950500	1.68475700	-0.15497200	C	5.38658900	-1.33597900	-1.77745400
Cl	2.08305700	3.31307200	-0.15980700	C	6.37551500	-0.34986100	-1.89487700
C	0.88134000	-1.76994900	-1.01981500	C	5.66857300	-2.47735700	-1.01813900
C	-0.21162200	-1.03317300	-0.68820600	C	7.61071400	-0.49936000	-1.26648000
C	-0.97159100	-0.05051200	-0.45591500	H	6.17420400	0.54200000	-2.48365200

C	6.90404100	-2.63138300	-0.38609100	C	-7.19902000	-0.06437800	0.45495200
H	4.91695500	-3.25830100	-0.92786200	C	-7.96618700	-2.11743000	-0.58418300
C	7.87856700	-1.64115100	-0.50737000	C	-8.51587700	0.25977900	0.77561800
H	8.36567400	0.27504500	-1.36998000	H	-6.38403200	0.59431500	0.73164500
H	7.10365800	-3.52570700	0.19760400	C	-9.28052700	-1.78925800	-0.26326000
H	8.84102000	-1.75805600	-0.01761100	H	-7.72424600	-3.03495400	-1.10963200
O	-4.62005300	-0.73165000	-0.22891000	C	-9.55662900	-0.60021600	0.41698400
C	-5.53456300	-1.65612400	-0.59437200	H	-8.73094500	1.18228400	1.30718100
O	-5.24522400	-2.69296600	-1.16053000	H	-10.08969400	-2.45849600	-0.54086300
C	-6.92099100	-1.25595800	-0.22755900	H	-10.58219500	-0.34475600	0.66888600

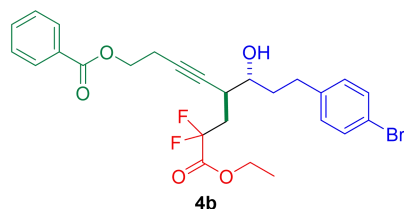
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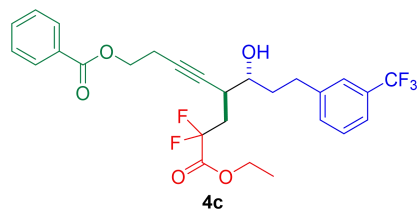
7. Report of the NMR spectra



4a, $R_f = 0.5$ (EA:PE=1:3), column solvent: hexane/EtOAc = 10/1, 85%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 8.05 (d, $J = 7.1$ Hz, 2H), 7.56 (t, $J = 7.4$ Hz, 1H), 7.43 (t, $J = 7.8$ Hz, 2H), 7.26 (t, $J = 7.8$ Hz, 2H), 7.18 (t, $J = 7.4$ Hz, 1H), 7.15 (d, $J = 7.1$ Hz, 2H), 4.43 (t, $J = 6.6$ Hz, 2H), 4.33 – 4.26 (m, 2H), 3.54 – 3.45 (m, 1H), 2.80 – 2.71 (m, 2H), 2.69 – 2.59 (m, 3H), 2.52 – 2.26 (m, 2H), 1.99 – 1.76 (m, 3H), 1.33 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 166.50, 163.85 (t, $J = 32.6$ Hz), 141.42, 133.15, 129.76, 129.61, 128.39, 128.35, 125.89, 115.51 (dd, $J = 251.8, 250.0$ Hz), 80.89, 78.81, 72.40, 62.85, 62.71, 37.07, 37.03 (t, $J = 22.9$ Hz), 32.68 – 32.37 (m), 31.94, 19.58, 13.87. ^{19}F NMR (471 MHz, CDCl_3) δ -103.12 (dt, $J = 260.3, 14.6$ Hz, 1F), -106.21 (dt, $J = 36.2, 17.8$ Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{26}\text{H}_{28}\text{F}_2\text{NaO}_5$, 481.1797, found 481.1795.

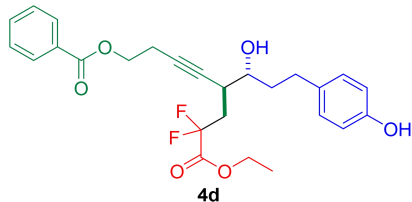


4b, $R_f = 0.6$ (EA:PE=1:3), column solvent: hexane/EtOAc = 10/1, 77%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 8.03 (d, $J = 7.4$ Hz, 2H), 7.56 (t, $J = 7.4$ Hz, 1H), 7.42 (t, $J = 7.7$ Hz, 2H), 7.35 (d, $J = 8.3$ Hz, 2H), 6.99 (d, $J = 8.3$ Hz, 2H), 4.43 (t, $J = 6.4$ Hz, 2H), 4.29 (q, $J = 7.0$ Hz, 2H), 3.53 – 3.42 (m, 1H), 2.78 – 2.70 (m, 1H), 2.69 – 2.53 (m, 4H), 2.49 – 2.26 (m, 2H), 1.90 – 1.69 (m, 2H), 1.33 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 166.52, 163.83 (t, $J = 32.6$ Hz), 140.40, 133.20, 131.41, 130.12, 129.72, 129.60, 128.40, 119.60, 115.46 (dd, $J = 251.8, 249.9$ Hz), 81.06, 78.70, 72.20, 62.89, 62.68, 37.02 (t, $J = 22.9$ Hz), 36.93, 32.72 – 32.28 (m), 31.35, 19.62, 13.88. ^{19}F NMR (471 MHz, CDCl_3) δ -102.74 – -103.78 (m, 1F), -106.22 (dt, $J = 36.0, 18.2$ Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{26}\text{H}_{27}\text{BrF}_2\text{NaO}_5$, 559.0902, found 559.0893.

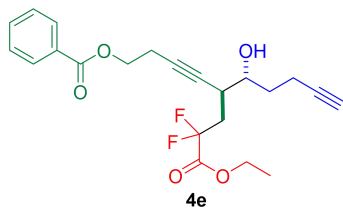


4c, $R_f = 0.5$ (EA:PE=1:3), column solvent: hexane/EtOAc = 10/1, 80%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 8.03 (d, $J = 7.2$ Hz, 2H), 7.55 (t, $J = 7.4$ Hz, 1H), 7.46 – 7.38 (m, 4H), 7.36 (t, $J = 7.7$ Hz, 1H), 7.31 (d, $J = 7.6$ Hz, 1H), 4.44 (t, $J = 6.5$ Hz, 2H), 4.34 – 4.24 (m, 2H), 3.56 – 3.43 (m, 1H), 2.85 – 2.60 (m, 5H), 2.49 – 2.27 (m, 2H), 1.96 (s, 1H), 1.91 – 1.75 (m, 2H), 1.33 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 166.55, 163.83 (t, $J = 32.6$ Hz), 142.39, 133.20, 131.79, 130.64 (q, $J = 31.9$ Hz), 129.72, 129.60, 128.80, 128.40, 124.17 (q, $J = 272.1$ Hz), 125.04 (q, $J = 3.7$ Hz), 122.81 (q, $J = 3.6$ Hz), 117.54 – 113.38 (m), 81.16, 78.68, 72.23, 62.91, 62.65, 37.01 (t, $J = 22.9$ Hz), 36.85, 32.88

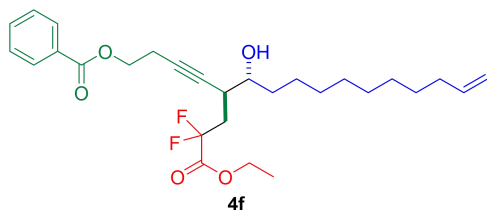
– 32.35 (m), 31.78, 19.63, 13.87. ^{19}F NMR (471 MHz, CDCl_3) δ -62.50 (s, 3F), -102.87 – -103.65 (m, 1F), -106.21 (dt, J = 261.4, 18.0 Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{27}\text{H}_{28}\text{F}_5\text{NaO}_5$, 527.1851, found 527.1862.



4d, R_f = 0.3 (EA:PE=1:1), column solvent: hexane/EtOAc = 2/1, 82%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 8.04 (d, J = 7.2 Hz, 2H), 7.56 (t, J = 7.4 Hz, 1H), 7.43 (t, J = 7.8 Hz, 2H), 6.99 (d, J = 8.4 Hz, 2H), 6.73 (d, J = 8.4 Hz, 2H), 5.30 (s, 1H), 4.42 (t, J = 6.6 Hz, 2H), 4.29 (qd, J = 7.1, 1.9 Hz, 2H), 3.58 – 3.42 (m, 1H), 2.79 – 2.70 (m, 1H), 2.68 – 2.52 (m, 4H), 2.47 – 2.25 (m, 2H), 1.92 (s, 1H), 1.86 – 1.71 (m, 2H), 1.33 (t, J = 7.2 Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 166.64, 163.92 (t, J = 32.6 Hz), 153.86, 133.37, 133.22, 129.72, 129.65, 129.43, 128.43, 115.52 (dd, J = 251.7, 250.2 Hz), 115.26, 80.86, 78.85, 72.39, 62.92, 62.78, 37.24, 37.02 (t, J = 23.0 Hz), 32.62 – 32.37 (m), 31.00, 19.59, 13.89. ^{19}F NMR (471 MHz, CDCl_3) δ -103.14 (ddd, J = 260.9, 17.4, 12.9 Hz, 1F), -106.23 (dt, J = 35.7, 18.1 Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{26}\text{H}_{28}\text{F}_2\text{NaO}_6$, 497.1746, found 497.1730.

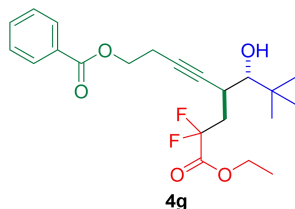


4e, R_f = 0.5 (EA:PE=1:3), column solvent: hexane/EtOAc = 10/1, 66%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 8.04 (d, J = 7.2 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.45 (t, J = 7.7 Hz, 2H), 4.43 (t, J = 6.6 Hz, 2H), 4.34 – 4.27 (m, 2H), 3.75 – 3.62 (m, 1H), 2.80 – 2.71 (m, 1H), 2.68 – 2.58 (m, 2H), 2.50 – 2.31 (m, 2H), 2.31 – 2.21 (m, 2H), 1.98 – 1.90 (m, 2H), 1.80 – 1.63 (m, 2H), 1.34 (t, J = 7.2 Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 166.49, 163.84 (t, J = 32.6 Hz), 133.18, 129.77, 129.62, 128.42, 115.47 (dd, J = 251.8, 250.1 Hz), 83.43, 80.99, 78.68, 71.84, 69.01, 62.90, 62.67, 36.91 (t, J = 23.0 Hz), 33.90, 32.56 – 32.34 (m), 19.58, 14.99, 13.90. ^{19}F NMR (471 MHz, CDCl_3) δ -102.84 – -103.65 (m, 1F), -106.19 (dt, J = 261.6, 18.0 Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{22}\text{H}_{24}\text{F}_2\text{NaO}_5$, 429.1484, found 429.1477.

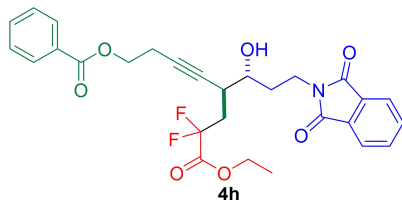


4f, R_f = 0.5 (EA:PE=1:3), column solvent: hexane/EtOAc = 10/1, 60%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 8.05 (d, J = 7.2 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.44 (t, J = 7.7 Hz, 2H), 5.90 – 5.71 (m, 1H), 4.96 (dd, J = 30.7, 13.6 Hz, 2H), 4.45 – 4.38 (m, 2H), 4.34 – 4.27 (m, 2H), 3.53 – 3.44 (m, 1H), 2.77 – 2.69 (m, 1H), 2.65 (td, J = 6.5, 1.8 Hz, 2H), 2.50 – 2.27 (m, 2H), 2.03 (dd, J = 14.4, 7.0 Hz, 2H), 1.79 – 1.61 (m, 2H), 1.55 – 1.45 (m, 2H), 1.39 – 1.30 (m, 6H), 1.28 – 1.18 (m, 8H). ^{13}C NMR

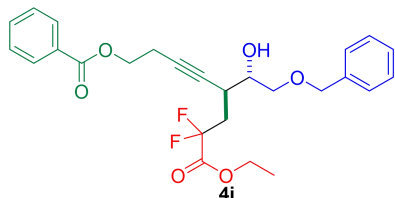
(126 MHz, CDCl₃) δ 166.47, 163.92 (t, *J* = 32.6 Hz), 139.18, 133.14, 129.82, 129.63, 128.38, 115.60 (dd, *J* = 251.8, 250.1 Hz), 114.10, 80.63, 78.96, 73.22, 62.85, 62.75, 37.14 (t, *J* = 22.9 Hz), 35.56, 33.77, 32.63 – 31.90 (m), 29.47, 29.39, 29.35, 29.06, 28.88, 25.78, 19.57, 13.90. ¹⁹F NMR (471 MHz, CDCl₃) δ -102.55 – -103.56 (m, 1F), -106.29 (dt, *J* = 36.3, 17.9 Hz, 1F). HRMS-ESI (*m/z*) [M+Na]⁺ calculated for C₂₈H₃₈F₂NaO₅, 515.2580, found 515.2572.



4g, *R_f* = 0.5 (EA:PE=1:3), column solvent: hexane/EtOAc = 10/1, 54%, colorless oil, ¹H NMR (500 MHz, CDCl₃) δ 8.04 (d, *J* = 7.2 Hz, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 2H), 4.44 – 4.35 (m, 2H), 4.33 – 4.26 (m, 2H), 3.11 (s, 1H), 3.02 (t, *J* = 6.3 Hz, 1H), 2.67 – 2.60 (m, 2H), 2.56 – 2.26 (m, 2H), 1.91 (s, 1H), 1.34 (t, *J* = 7.1 Hz, 3H), 0.94 (s, 9H). ¹³C NMR (126 MHz, CDCl₃) δ 166.38, 163.85 (t, *J* = 32.7 Hz), 133.13, 129.77, 129.62, 128.37, 115.48 (dd, *J* = 251.8, 250.0 Hz), 82.56, 80.38, 79.41, 62.87, 62.62, 39.71 (t, *J* = 22.6 Hz), 36.04, 28.24 – 27.83 (m), 26.30, 19.52, 13.90. ¹⁹F NMR (471 MHz, CDCl₃) δ -102.57 – -103.37 (m, 1F), -106.49 (dt, *J* = 261.8, 18.2 Hz, 1F). HRMS-ESI (*m/z*) [M+Na]⁺ calculated for C₂₂H₂₈F₂NaO₅, 433.1797, found 433.1794.

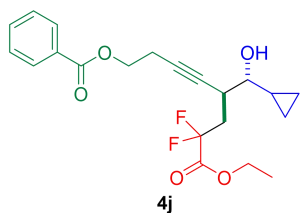


4h, *R_f* = 0.4 (EA:PE=1:3), column solvent: hexane/EtOAc = 5/1, 65%, colorless oil, ¹H NMR (500 MHz, CDCl₃) δ 8.02 (d, *J* = 7.4 Hz, 2H), 7.85 – 7.79 (m, 2H), 7.73 – 7.68 (m, 2H), 7.53 (t, *J* = 7.4 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 2H), 4.42 – 4.36 (m, 2H), 4.31 – 4.25 (m, 2H), 3.83 – 3.77 (m, 2H), 3.58 – 3.49 (m, 1H), 2.84 (s, 1H), 2.79 – 2.72 (m, 1H), 2.65 – 2.58 (m, 2H), 2.49 – 2.30 (m, 2H), 1.99 – 1.90 (m, 1H), 1.87 – 1.80 (m, 1H), 1.31 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 168.69, 166.31, 163.80 (t, *J* = 32.6 Hz), 134.06, 133.02, 131.88, 129.82, 129.58, 128.32, 123.31, 115.47 (dd, *J* = 251.4, 250.1 Hz), 80.59, 78.76, 69.98, 62.83, 62.69, 36.74 (t, *J* = 22.9 Hz), 34.62, 34.02, 32.30 – 31.84 (m), 19.44, 13.85. ¹⁹F NMR (471 MHz, CDCl₃) δ -102.73 – -103.79 (m, 1F), -106.15 (dt, *J* = 36.1, 17.8 Hz, 1F). HRMS-ESI (*m/z*) [M+Na]⁺ calculated for C₂₈H₂₇F₂NNaO₇, 550.1648, found 550.1645.

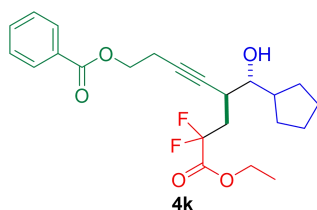


4i, *R_f* = 0.5 (EA:PE=1:3), column solvent: hexane/EtOAc = 10/1, 71%, colorless oil, ¹H NMR (500 MHz, CDCl₃) δ 8.05 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.36 – 7.31 (m, 2H), 7.31 – 7.25 (m, 3H), 4.50 – 4.42 (m, 2H), 4.38 (t, *J* = 6.7 Hz, 2H), 4.34 – 4.24 (m, 2H), 3.85 – 3.76 (m, 1H), 3.52 (d, *J* = 6.1 Hz, 2H), 3.04 – 2.89 (m, 1H), 2.62 (td, *J* = 6.7, 2.1 Hz, 2H), 2.49 – 2.32 (m, 2H), 1.33 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 166.39, 163.82 (t, *J* = 32.6 Hz),

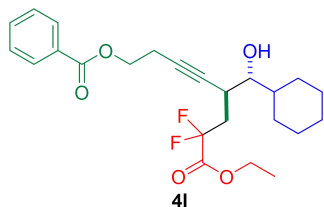
137.68, 133.11, 129.83, 129.62, 128.38, 128.32, 127.75, 127.68, 115.46 (dd, $J = 251.9, 250.0$ Hz), 80.51, 78.53, 73.29, 71.88, 71.29, 62.84, 62.68, 36.39 (t, $J = 23.2$ Hz), 29.74 – 28.58 (m), 19.47, 13.86. ^{19}F NMR (471 MHz, CDCl_3) δ -102.65 – -103.73 (m, 1F), -106.25 (dt, $J = 35.7, 18.1$ Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{26}\text{H}_{28}\text{F}_2\text{NaO}_6$, 497.1746, found 497.1746.



4j, $R_f = 0.5$ (EA:PE=1:3), column solvent: hexane/EtOAc = 10/1, 51%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 8.04 (d, $J = 7.7$ Hz, 2H), 7.56 (t, $J = 7.4$ Hz, 1H), 7.44 (t, $J = 7.6$ Hz, 2H), 4.43 (t, $J = 6.7$ Hz, 2H), 4.31 (q, $J = 7.1$ Hz, 2H), 2.95 – 2.86 (m, 1H), 2.74 (dd, $J = 8.8, 3.3$ Hz, 1H), 2.69 – 2.62 (m, 2H), 2.49 – 2.32 (m, 2H), 2.01 (s, 1H), 1.34 (t, $J = 7.1$ Hz, 3H), 1.13 – 0.98 (m, 1H), 0.55 – 0.42 (m, 2H), 0.40 – 0.16 (m, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 166.44, 163.89 (t, $J = 32.6$ Hz), 133.11, 129.83, 129.61, 128.35, 115.56 (dd, $J = 251.9, 249.9$ Hz), 80.28, 79.51, 78.01, 62.84, 62.73, 36.93 (t, $J = 23.0$ Hz), 32.89 – 32.68 (m), 19.56, 15.80, 13.89, 3.07, 2.89. ^{19}F NMR (471 MHz, CDCl_3) δ -102.44 – -103.33 (m, 1F), -106.34 (dt, $J = 36.4, 17.9$ Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{21}\text{H}_{24}\text{F}_2\text{NaO}_5$, 417.1484, found 417.1470.

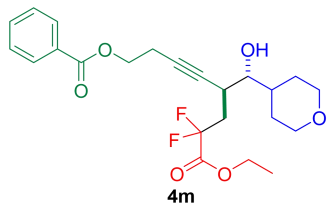


4k, $R_f = 0.5$ (EA:PE=1:3), column solvent: hexane/EtOAc = 10/1, 65%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 8.05 (d, $J = 7.3$ Hz, 2H), 7.57 (t, $J = 7.4$ Hz, 1H), 7.44 (t, $J = 7.6$ Hz, 2H), 4.47 – 4.38 (m, 2H), 4.31 (q, $J = 7.0, 2.1$ Hz, 2H), 3.20 – 3.08 (m, 1H), 2.88 – 2.79 (m, 1H), 2.69 – 2.61 (m, 2H), 2.55 – 2.25 (m, 2H), 2.06 – 1.94 (m, 1H), 1.80 – 1.70 (m, 2H), 1.69 – 1.62 (m, 1H), 1.62 – 1.51 (m, 2H), 1.49 – 1.40 (m, 2H), 1.34 (t, $J = 7.1$ Hz, 3H), 1.11 – 0.98 (m, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 166.47, 163.93 (t, $J = 32.6$ Hz), 133.14, 129.82, 129.60, 128.39, 115.63 (dd, $J = 251.7, 249.5$ Hz), 80.54, 78.85, 78.17, 62.83, 62.75, 45.00, 37.67 (t, $J = 22.8$ Hz), 31.82 – 30.41 (m), 29.79, 29.09, 25.41, 25.28, 19.60, 13.90. ^{19}F NMR (471 MHz, CDCl_3) δ -102.54 – -103.66 (m, 1F), -106.21 (dt, $J = 36.2, 17.8$ Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{23}\text{H}_{29}\text{F}_2\text{O}_5$, 423.1978, found 423.1977.

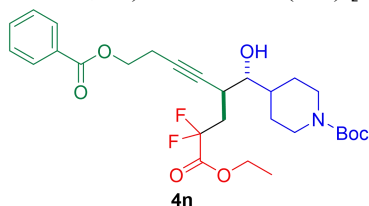


4l, $R_f = 0.5$ (EA:PE=1:3), column solvent: hexane/EtOAc = 10/1, 70%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 8.05 (d, $J = 8.1$ Hz, 2H), 7.56 (t, $J = 7.4$ Hz, 1H), 7.44 (t, $J = 7.7$ Hz, 2H), 4.42 (t, $J = 6.6$ Hz, 2H), 4.34 – 4.26 (m, 2H), 3.12 – 3.04 (m, 1H), 2.96 – 2.88 (m, 1H), 2.68 – 2.60 (m, 2H), 2.55 – 2.25 (m, 2H), 2.02 – 1.93 (m, 1H), 1.74 – 1.56 (m, 5H), 1.45 – 1.38 (m, 1H), 1.33 (t, $J = 7.1$ Hz, 3H),

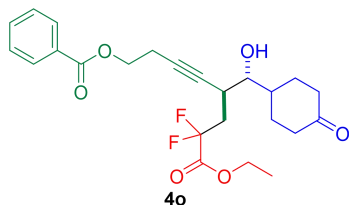
1.17 – 1.00 (m, 3H), 0.98 – 0.83 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 166.47, 163.92 (t, *J* = 32.7 Hz), 133.14, 129.80, 129.61, 128.39, 115.66 (dd, *J* = 251.7, 249.9 Hz), 80.59, 78.76, 77.81, 62.82, 62.79, 42.05, 37.73 (t, *J* = 22.8 Hz), 29.68 – 29.33 (m), 29.21, 28.99, 26.14, 25.79, 25.58, 19.58, 13.89. ¹⁹F NMR (471 MHz, CDCl₃) δ -102.48 – -103.48 (m, 1F), -106.36 (dt, *J* = 260.2, 18.1 Hz, 1F). HRMS-ESI (m/z) [M+Na]⁺ calculated for C₂₄H₃₀F₂NaO₅, 459.1954, found 459.1951.



4m, *R_f* = 0.4 (EA:PE=1:2), column solvent: hexane/EtOAc = 5/1, 62%, colorless oil, ¹H NMR (500 MHz, CDCl₃) δ 8.04 (d, *J* = 7.1 Hz, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 2H), 4.50 – 4.38 (m, 2H), 4.34 – 4.25 (m, 2H), 3.93 – 3.83 (m, 2H), 3.21 (td, *J* = 11.9, 2.1 Hz, 1H), 3.13 – 3.05 (m, 2H), 2.92 – 2.82 (m, 1H), 2.69 – 2.60 (m, 2H), 2.56 – 2.24 (m, 2H), 1.94 – 1.75 (m, 2H), 1.70 – 1.57 (m, 1H), 1.52 – 1.43 (m, 1H), 1.33 (t, *J* = 7.2 Hz, 3H), 1.30 – 1.15 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 166.52, 163.82 (t, *J* = 32.6 Hz), 133.25, 129.71, 129.58, 128.43, 115.51 (dd, *J* = 251.8, 250.1 Hz), 81.00, 78.26, 77.32, 67.59, 67.15, 62.87, 62.66, 39.47, 37.54 (t, *J* = 22.7 Hz), 29.50, 29.16 – 28.99 (m), 28.81, 19.64, 13.87. ¹⁹F NMR (471 MHz, CDCl₃) δ -102.71 – -103.63 (m, 1F), -106.39 (dt, *J* = 261.3, 18.3 Hz, 1F). HRMS-ESI (m/z) [M+Na]⁺ calculated for C₂₃H₂₈F₂NaO₆, 461.1746, found 461.1740.

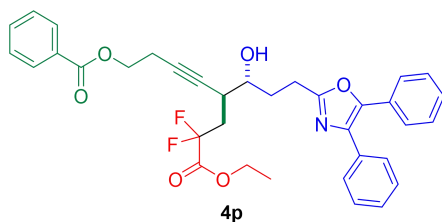


4n, *R_f* = 0.4 (EA:PE=1:3), column solvent: hexane/EtOAc = 5/1, 57%, colorless oil, ¹H NMR (500 MHz, CDCl₃) δ 8.03 (d, *J* = 7.0 Hz, 2H), 7.57 (t, *J* = 8.1 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 2H), 4.48 – 4.37 (m, 2H), 4.33 – 4.20 (m, 2H), 4.16 – 3.90 (m, 2H), 3.16 – 3.00 (m, 1H), 2.92 – 2.81 (m, 1H), 2.68 – 2.60 (m, 2H), 2.57 – 2.26 (m, 4H), 1.96 – 1.81 (m, 2H), 1.60 – 1.48 (m, 2H), 1.43 (s, 9H), 1.34 – 1.30 (m, 3H), 1.18 – 0.97 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 166.51, 163.80 (t, *J* = 32.5 Hz), 154.65, 133.26, 129.71, 129.57, 128.43, 115.49 (dd, *J* = 251.9, 250.0 Hz), 81.05, 79.30, 78.29, 62.87, 62.65, 43.28, 40.56, 37.54 (t, *J* = 22.8 Hz), 29.82 – 29.14 (m), 28.58, 28.38, 28.01, 19.64, 13.88. ¹⁹F NMR (471 MHz, CDCl₃) δ -102.21 – -103.83 (m, 1F), -105.69 – -107.10 (m, 1F). HRMS-ESI (m/z) [M+Na]⁺ calculated for C₂₈H₃₇F₂NNaO₇, 560.2430, found 560.2427.

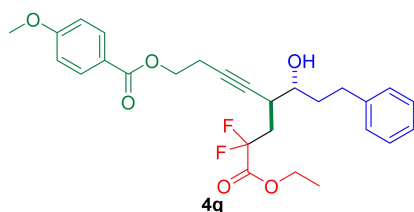


4o, *R_f* = 0.2 (EA:PE=1:3), column solvent: hexane/EtOAc = 3/1, 63%, colorless oil, ¹H NMR (500 MHz, CDCl₃) δ 8.05 (d, *J* = 7.3 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 2H), 4.54 – 4.40 (m, 2H), 4.31 (q, *J* = 7.1 Hz, 2H), 3.25 – 3.15 (m, 1H), 2.98 – 2.87 (m, 1H), 2.71 – 2.63 (m, 2H), 2.56 – 2.42 (m, 1H), 2.40 – 2.25 (m, 4H), 2.24 – 2.14 (m, 1H), 2.08 – 1.92 (m, 3H), 1.89 – 1.80 (m, 1H), 1.50 – 1.36 (m, 2H), 1.34 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 211.46, 166.60, 163.79 (t, *J* = 32.5 Hz), 133.38, 129.71, 129.58, 128.50, 115.45 (dd, *J* = 251.9, 250.2 Hz), 81.41, 78.22, 76.34, 62.95,

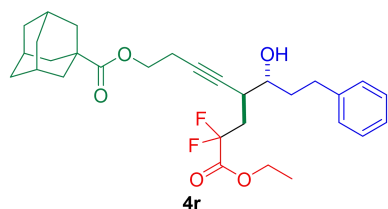
62.64, 40.35, 39.97, 39.95, 37.56 (t, $J = 22.7$ Hz), 30.08 – 29.88 (m), 28.94, 28.72, 19.74, 13.90. ^{19}F NMR (471 MHz, CDCl_3) δ -102.76 – -103.79 (m, 1F), -105.91 – -106.87 (m, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{24}\text{H}_{28}\text{F}_2\text{NaO}_6$, 473.1746, found 473.1732.



4p, $R_f = 0.3$ (EA:PE=1:2), column solvent: hexane/EtOAc = 3/1, 67%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 8.04 (d, $J = 7.2$ Hz, 2H), 7.61 (d, $J = 6.9$ Hz, 2H), 7.57 (d, $J = 6.9$ Hz, 2H), 7.52 (t, $J = 7.4$ Hz, 1H), 7.41 (t, $J = 7.7$ Hz, 2H), 7.38 – 7.29 (m, 6H), 4.42 (t, $J = 6.7$ Hz, 2H), 4.33 – 4.26 (m, 2H), 3.79 – 3.72 (m, 1H), 2.97 (t, $J = 7.0$ Hz, 2H), 2.89 – 2.77 (m, 1H), 2.70 – 2.59 (m, 2H), 2.51 – 2.38 (m, 2H), 2.15 – 2.05 (m, 2H), 1.33 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 166.41, 163.89 (t, $J = 32.6$ Hz), 163.25, 145.29, 134.67, 133.08, 132.09, 129.81, 129.59, 128.79, 128.62, 128.53, 128.49, 128.34, 128.09, 127.81, 126.43, 115.58 (dd, $J = 251.5, 250.3$ Hz), 80.56, 79.12, 72.71, 62.84, 62.72, 36.53 (t, $J = 22.9$ Hz), 33.08 – 32.45 (m), 31.68, 25.26, 19.55, 13.88. ^{19}F NMR (471 MHz, CDCl_3) δ -102.73 – -103.70 (m, 1F), -106.06 (dt, $J = 36.2, 17.8$ Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{35}\text{H}_{33}\text{F}_2\text{NNaO}_6$, 624.2168, found 624.2152.

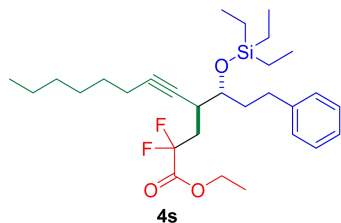


4q, $R_f = 0.4$ (EA:PE=1:3), column solvent: hexane/EtOAc = 5/1, 71%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 7.97 (d, $J = 8.8$ Hz, 2H), 7.24 (t, $J = 7.4$ Hz, 2H), 7.15 (t, $J = 7.3$ Hz, 1H), 7.12 (d, $J = 7.3$ Hz, 2H), 6.87 (d, $J = 8.9$ Hz, 2H), 4.40 – 4.35 (m, 2H), 4.30 – 4.23 (m, 2H), 3.82 (s, 3H), 3.54 – 3.38 (m, 1H), 2.76 – 2.69 (m, 2H), 2.66 – 2.57 (m, 3H), 2.47 – 2.25 (m, 2H), 1.95 – 1.74 (m, 3H), 1.31 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 166.25, 163.84 (t, $J = 32.6$ Hz), 163.49, 141.45, 131.67, 128.36, 128.33, 125.86, 122.12, 115.51 (dd, $J = 253.3, 248.3$ Hz), 113.63, 81.03, 78.71, 72.44, 62.84, 62.39, 55.37, 37.06, 37.38 – 36.75 (m), 37.38 – 36.75 (m), 31.95, 19.65, 13.86. ^{19}F NMR (471 MHz, CDCl_3) δ -102.38 – -103.60 (m, 1F), -106.29 (dt, $J = 260.5, 17.9$ Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{27}\text{H}_{30}\text{F}_2\text{NaO}_6$, 511.1903, found 511.1890.

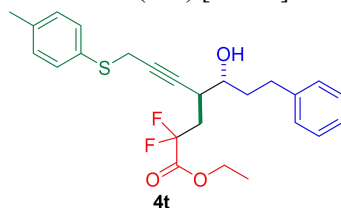


4r, $R_f = 0.5$ (EA:PE=1:3), column solvent: hexane/EtOAc = 10/1, 75%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 7.28 (t, $J = 7.5$ Hz, 2H), 7.22 – 7.16 (m, 3H), 4.30 (q, $J = 7.1$ Hz, 2H), 4.22 – 4.15 (m, 1H), 4.13 – 4.06 (m, 1H), 3.56 – 3.47 (m, 1H), 2.84 – 2.65 (m, 3H), 2.51 – 2.46 (m, 2H), 2.46 – 2.27 (m, 2H), 2.02 – 1.98 (m, 3H), 1.96 – 1.90 (m, 2H), 1.89 – 1.86 (m, 6H), 1.74 – 1.64 (m, 6H), 1.34 (t, J

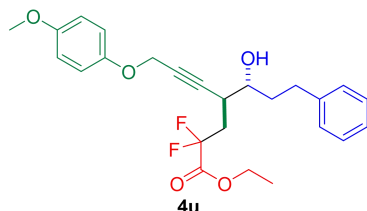
= 7.2 Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 177.60, 163.82 (t, $J = 32.6$ Hz), 141.44, 128.38, 128.35, 125.89, 115.49 (dd, $J = 251.8, 250.0$ Hz), 81.10, 78.39, 72.36, 62.83, 61.90, 40.63, 38.69, 37.10, 37.05 (t, $J = 22.9$ Hz), 36.36, 32.60 – 32.37 (m), 31.98, 27.80, 19.40, 13.86. ^{19}F NMR (471 MHz, CDCl_3) δ -102.76 – -103.55 (m, 1F), -106.19 (dt, $J = 261.1, 18.2$ Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{30}\text{H}_{38}\text{F}_2\text{NaO}_5$, 539.2580, found 539.2564.



4s, $R_f = 0.6$ (EA:PE=1:10), column solvent: hexane/EtOAc = 20/1, 82%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 7.32 – 7.27 (m, 2H), 7.22 – 7.16 (m, 3H), 4.30 (q, $J = 7.2$ Hz, 2H), 3.91 – 3.75 (m, 1H), 2.83 – 2.74 (m, 2H), 2.61 – 2.51 (m, 1H), 2.49 – 2.37 (m, 1H), 2.27 – 2.05 (m, 4H), 1.66 – 1.57 (m, 1H), 1.50 – 1.41 (m, 2H), 1.36 (t, $J = 7.2$ Hz, 5H), 1.31 – 1.24 (m, 4H), 0.98 (t, $J = 8.0$ Hz, 9H), 0.88 (t, $J = 6.9$ Hz, 3H), 0.63 (q, $J = 7.9$ Hz, 6H). ^{13}C NMR (126 MHz, CDCl_3) δ 164.08 (t, $J = 32.7$ Hz), 142.14, 128.35, 128.28, 125.80, 116.07 (dd, $J = 252.3, 248.0$ Hz), 84.13, 78.16, 73.67, 62.64, 34.82, 34.15 (t, $J = 23.2$ Hz), 32.81, 32.27 – 31.91 (m), 31.32, 28.75, 28.50, 22.53, 18.76, 14.04, 13.87, 6.86, 5.00. ^{19}F NMR (471 MHz, CDCl_3) δ -101.44 (dt, $J = 257.6, 13.3$ Hz, 1F), -107.38 – -108.34 (m, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{29}\text{H}_{46}\text{F}_2\text{NaO}_3\text{Si}$, 531.3076, found 531.3064.

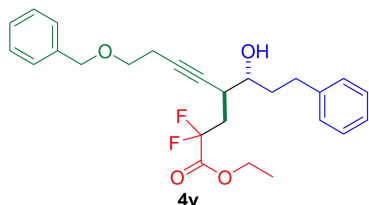


4t, $R_f = 0.5$ (EA:PE=1:3), column solvent: hexane/EtOAc = 10/1, 67%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 7.33 (d, $J = 8.1$ Hz, 2H), 7.29 (t, $J = 7.5$ Hz, 2H), 7.20 (t, $J = 7.4$ Hz, 1H), 7.16 (d, $J = 7.1$ Hz, 2H), 7.11 (d, $J = 8.0$ Hz, 2H), 4.31 (q, $J = 7.1$ Hz, 2H), 3.58 (d, $J = 2.1$ Hz, 2H), 3.50 – 3.38 (m, 1H), 2.78 – 2.65 (m, 2H), 2.64 – 2.55 (m, 1H), 2.46 – 2.27 (m, 5H), 1.77 – 1.67 (m, 2H), 1.35 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 163.80 (t, $J = 32.5$ Hz), 141.37, 137.39, 131.09, 130.93, 129.78, 128.40, 128.35, 125.93, 115.42 (dd, $J = 251.8, 250.1$ Hz), 80.84, 80.31, 72.31, 62.94, 37.08, 36.90 (t, $J = 23.1$ Hz), 32.76 – 32.37 (m), 31.90, 23.36, 21.02, 13.89. ^{19}F NMR (471 MHz, CDCl_3) δ -102.71 – -103.51 (m, 1F), -106.36 (dt, $J = 36.3, 17.8$ Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{25}\text{H}_{28}\text{F}_2\text{NaO}_3\text{S}$, 469.1619, found 469.1606.

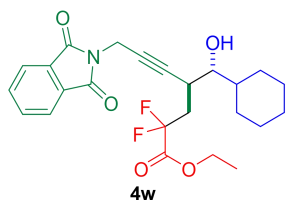


4u, $R_f = 0.3$ (EA:PE=1:3), column solvent: hexane/EtOAc = 5/1, 57%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 7.28 (t, $J = 7.5$ Hz, 2H), 7.20 (t, $J = 7.2$ Hz, 1H), 7.15 (d, $J = 7.6$ Hz, 2H), 6.89 (d, $J = 8.9$ Hz, 2H), 6.82 (d, $J = 8.9$ Hz, 2H), 4.64 (s, 2H), 4.30 (q, $J = 7.1$ Hz, 2H), 3.75 (s, 3H), 3.58 – 3.50 (m, 1H), 2.87 – 2.78 (m, 1H), 2.77 – 2.59 (m, 2H), 2.53 – 2.29 (m, 2H), 1.84 – 1.74 (m, 3H), 1.34 (t, J

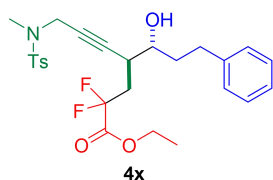
= 7.1 Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 163.79 (t, $J = 32.4$ Hz), 154.47, 151.47, 141.30, 128.44, 128.36, 125.98, 116.33, 115.40 (dd, $J = 251.7, 250.4$ Hz), 114.60, 84.60, 80.03, 72.33, 62.99, 56.90, 55.65, 36.95, 36.61 (t, $J = 23.1$ Hz), 32.73 – 32.40 (m), 31.96, 13.88. ^{19}F NMR (471 MHz, CDCl_3) δ -103.22 (ddd, $J = 261.4, 17.6, 13.2$ Hz, 1F), -106.26 (dt, $J = 261.5, 18.1$ Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{25}\text{H}_{28}\text{F}_2\text{NaO}_5$, 469.1797, found 469.1793.



4v, $R_f = 0.5$ (EA:PE=1:3), column solvent: hexane/EtOAc = 8/1, 82%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 7.37 – 7.31 (m, 4H), 7.31 – 7.26 (m, 3H), 7.22 – 7.16 (m, 3H), 4.53 (s, 2H), 4.28 (q, $J = 7.0$ Hz, 2H), 3.56 (t, $J = 6.8$ Hz, 2H), 3.54 – 3.46 (m, 1H), 2.85 – 2.63 (m, 3H), 2.52 – 2.24 (m, 4H), 1.97 – 1.79 (m, 2H), 1.33 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 163.93 (t, $J = 32.5$ Hz), 141.56, 137.89, 128.42, 128.41, 127.74, 127.70, 125.92, 115.62 (dd, $J = 251.8, 249.7$ Hz), 82.24, 78.10, 77.25, 77.00, 76.75, 72.99, 72.44, 68.39, 62.85, 37.17, 37.18 (t, $J = 23.0$ Hz), 32.83 – 32.50 (m), 32.01, 20.13, 13.88. ^{19}F NMR (471 MHz, CDCl_3) δ -102.76 (ddd, $J = 261.1, 16.9, 13.6$ Hz, 1F), -106.38 (dt, $J = 36.3, 18.6$ Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{26}\text{H}_{31}\text{F}_2\text{O}_4$, 445.2185, found 445.2185.

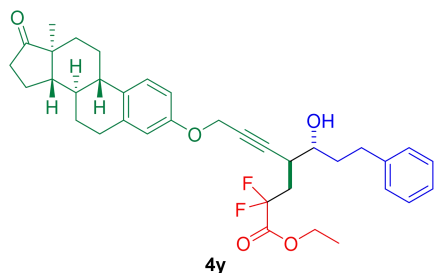


4w, $R_f = 0.4$ (EA:PE=1:3), column solvent: hexane/EtOAc = 5/1, 59%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 7.90 – 7.85 (m, 2H), 7.77 – 7.72 (m, 2H), 4.43 (d, $J = 1.8$ Hz, 2H), 4.31 (q, $J = 7.1$ Hz, 2H), 3.22 – 3.10 (m, 1H), 2.96 – 2.87 (m, 1H), 2.56 – 2.25 (m, 2H), 2.01 (d, $J = 12.6$ Hz, 1H), 1.76 – 1.61 (m, 5H), 1.48 – 1.39 (m, 1H), 1.34 (t, $J = 7.1$ Hz, 3H), 1.17 – 1.07 (m, 3H), 1.00 – 0.87 (m, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 167.11, 163.84 (t, $J = 32.3$ Hz), 134.24, 131.93, 123.54, 115.51 (dd, $J = 252.4, 250.1$ Hz), 81.33, 77.77, 62.96, 42.11, 37.22 (t, $J = 22.9$ Hz), 29.68 – 29.48 (m), 29.10, 29.08, 27.60, 26.19, 25.83, 25.73, 13.92. ^{19}F NMR (471 MHz, CDCl_3) δ -102.63 – -103.56 (m, 1F), -106.36 (dt, $J = 260.9, 18.5$ Hz, 1F). HRMS-ESI (m/z) $[\text{M}-\text{OH}]^+$ calculated for $\text{C}_{24}\text{H}_{26}\text{F}_2\text{NO}_4^+$, 430.1824, found 430.1827.

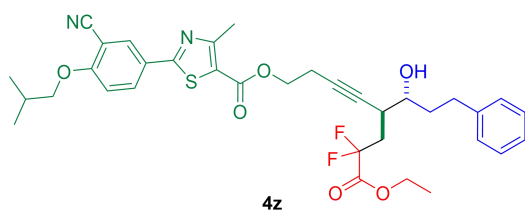


4x, $R_f = 0.3$ (EA:PE=1:3), column solvent: hexane/EtOAc = 5/1, 82%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 7.68 (d, $J = 8.3$ Hz, 2H), 7.30 (t, $J = 7.5$ Hz, 2H), 7.26 (d, $J = 8.0$ Hz, 2H), 7.21 (t, $J = 7.4$ Hz, 1H), 7.17 (d, $J = 7.0$ Hz, 2H), 4.31 (q, $J = 7.1$ Hz, 2H), 4.06 – 3.95 (m, 2H), 3.46 – 3.41 (m, 1H), 2.79 (s, 3H), 2.76 – 2.69 (m, 1H), 2.64 – 2.57 (m, 2H), 2.40 (s, 3H), 2.28 – 2.16 (m, 2H), 1.78 – 1.59 (m, 3H), 1.35 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 163.73 (t, $J = 32.4$ Hz), 143.79, 141.26, 134.17, 129.60, 128.50, 128.34, 127.86, 126.07, 115.28 (dd, $J = 254.0, 248.9$ Hz), 83.24, 72.08,

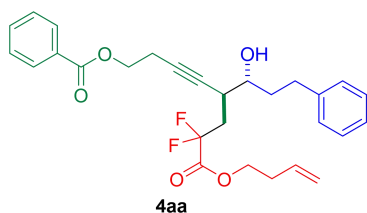
63.05, 39.95, 36.98, 36.37 (t, $J = 22.9$ Hz), 34.43, 32.47 – 32.11 (m), 31.95, 21.46, 13.91. ^{19}F NMR (471 MHz, CDCl_3) δ -103.83 – -104.81 (m, 1F), -104.97 – -106.01 (m, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{26}\text{H}_{31}\text{F}_2\text{NNaO}_5\text{S}$, 530.1783, found 530.1769.



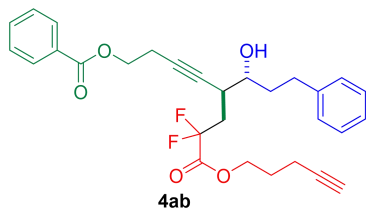
4y, $R_f = 0.2$ (EA:PE=1:3), column solvent: hexane/EtOAc = 3/1, 80%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 7.35 – 7.30 (m, 2H), 7.27 – 7.22 (m, 2H), 7.20 (d, $J = 7.4$ Hz, 2H), 6.81 (dd, $J = 8.5, 2.5$ Hz, 1H), 6.75 (d, $J = 2.4$ Hz, 1H), 4.71 (d, $J = 1.6$ Hz, 2H), 4.36 (q, $J = 7.1$ Hz, 2H), 3.67 – 3.54 (m, 1H), 2.96 – 2.85 (m, 3H), 2.83 – 2.75 (m, 1H), 2.72 – 2.64 (m, 1H), 2.60 – 2.49 (m, 2H), 2.46 – 2.35 (m, 2H), 2.33 – 2.24 (m, 1H), 2.25 – 2.14 (m, 1H), 2.14 – 2.07 (m, 1H), 2.07 – 1.96 (m, 2H), 1.93 – 1.82 (m, 2H), 1.75 (s, 1H), 1.71 – 1.43 (m, 7H), 1.40 (t, $J = 7.2$ Hz, 3H), 0.95 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 163.75 (t, $J = 32.5$ Hz), 155.40, 141.25, 137.83, 132.94, 128.39, 128.32, 128.31, 126.32, 125.95, 117.47 – 113.27 (m), 115.10, 115.05, 112.62, 112.59, 84.63, 79.93, 72.32, 62.96, 56.06, 50.30, 47.93, 43.88, 38.19, 36.84, 36.55 (t, $J = 23.1$ Hz), 35.81, 32.61 – 32.41 (m), 31.95, 31.48, 29.54, 26.43, 25.82, 21.51, 13.86, 13.77. ^{19}F NMR (471 MHz, CDCl_3) δ -102.66 – -103.71 (m, 1F), -105.63 – -106.86 (m, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{36}\text{H}_{42}\text{F}_2\text{NaO}_5$, 615.2893, found 615.2882.



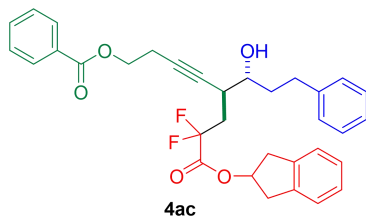
4z, $R_f = 0.3$ (EA:PE=1:3), column solvent: hexane/EtOAc = 3/1, 61%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 8.24 (d, $J = 2.3$ Hz, 1H), 8.15 (dd, $J = 8.8, 2.3$ Hz, 1H), 7.35 – 7.30 (m, 2H), 7.26 – 7.20 (m, 3H), 7.08 (d, $J = 8.9$ Hz, 1H), 4.48 (t, $J = 6.5$ Hz, 2H), 4.38 (q, $J = 7.2$ Hz, 2H), 3.98 (d, $J = 6.5$ Hz, 2H), 3.65 – 3.56 (m, 1H), 2.89 – 2.81 (m, 5H), 2.78 – 2.68 (m, 3H), 2.59 – 2.39 (m, 2H), 2.33 – 2.24 (m, 1H), 2.04 – 1.86 (m, 3H), 1.42 (t, $J = 7.2$ Hz, 3H), 1.17 (d, $J = 6.7$ Hz, 6H). ^{13}C NMR (126 MHz, CDCl_3) δ 167.57, 163.85 (t, $J = 32.5$ Hz), 162.52, 161.86, 161.78, 141.36, 132.58, 132.09, 128.41, 128.30, 125.93, 125.83, 121.11, 115.50 (dd, $J = 253.9, 248.0$ Hz), 115.33, 112.56, 102.93, 80.60, 79.02, 75.65, 72.40, 62.99, 62.91, 37.14, 37.00 (t, $J = 22.8$ Hz), 32.72 – 32.25 (m), 31.99, 28.11, 19.59, 19.02, 17.52, 13.91. ^{19}F NMR (471 MHz, CDCl_3) δ -103.05 – -104.04 (m, 1F), -105.97 (dt, $J = 35.9, 17.5$ Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{35}\text{H}_{38}\text{F}_2\text{N}_2\text{NaO}_6\text{S}$, 675.2311, found 675.2314.



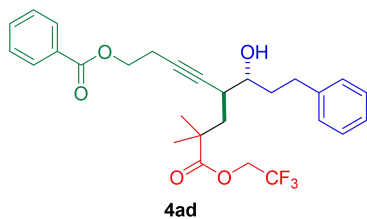
4aa, $R_f = 0.5$ (EA:PE=1:3), column solvent: hexane/EtOAc = 10/1, 56%, colorless oil, $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.05 (d, $J = 7.7$ Hz, 2H), 7.56 (t, $J = 7.4$ Hz, 1H), 7.43 (t, $J = 7.8$ Hz, 2H), 7.27 (t, $J = 7.5$ Hz, 2H), 7.18 (t, $J = 7.3$ Hz, 1H), 7.15 (d, $J = 7.2$ Hz, 2H), 5.83 – 5.70 (m, 1H), 5.17 – 5.06 (m, 2H), 4.44 (t, $J = 6.6$ Hz, 2H), 4.34 – 4.24 (m, 2H), 3.58 – 3.45 (m, 1H), 2.79 – 2.70 (m, 2H), 2.68 – 2.59 (m, 3H), 2.50 – 2.25 (m, 4H), 1.95 – 1.75 (m, 3H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 166.48, 163.79 (t, $J = 32.8$ Hz), 141.42, 133.15, 132.86, 129.76, 129.62, 128.40, 128.35, 125.89, 118.00, 115.49 (dd, $J = 254.7, 247.1$ Hz), 80.94, 78.79, 72.38, 65.61, 62.70, 37.10, 37.05 (t, $J = 22.9$ Hz), 32.61, 32.58 – 32.48 (m), 31.94, 19.60. $^{19}\text{F NMR}$ (471 MHz, CDCl_3) δ -102.57 – -103.39 (m, 1F), -105.98 (dt, $J = 260.9, 18.0$ Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{28}\text{H}_{30}\text{F}_2\text{NaO}_5$, 507.1954, found 507.1953.



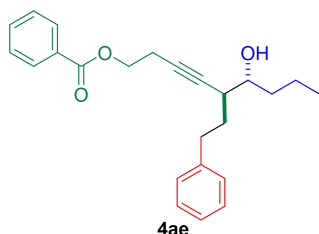
4ab, $R_f = 0.5$ (EA:PE=1:3), column solvent: hexane/EtOAc = 10/1, 61%, colorless oil, $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.05 (d, $J = 7.3$ Hz, 2H), 7.56 (t, $J = 7.4$ Hz, 1H), 7.43 (t, $J = 7.7$ Hz, 2H), 7.26 (t, $J = 7.5$ Hz, 2H), 7.18 (t, $J = 7.4$ Hz, 1H), 7.14 (d, $J = 7.3$ Hz, 2H), 4.44 (t, $J = 6.6$ Hz, 2H), 4.35 (t, $J = 6.3$ Hz, 2H), 3.56 – 3.44 (m, 1H), 2.79 – 2.70 (m, 2H), 2.69 – 2.58 (m, 3H), 2.51 – 2.26 (m, 4H), 2.01 – 1.97 (m, 1H), 1.95 – 1.76 (m, 5H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 166.48, 163.78 (t, $J = 32.8$ Hz), 141.40, 133.15, 129.75, 129.61, 128.40, 128.35, 125.89, 115.52 (dd, $J = 252.0, 249.9$ Hz), 82.37, 81.00, 78.73, 72.37, 69.49, 65.15, 62.69, 37.05, 37.02 (t, $J = 22.9$ Hz), 32.62 – 32.40 (m), 31.94, 27.02, 19.59, 14.93. $^{19}\text{F NMR}$ (471 MHz, CDCl_3) δ -102.73 (ddd, $J = 30.4, 16.4, 13.6$ Hz, 1F), -105.97 (dt, $J = 261.6, 18.2$ Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{29}\text{H}_{30}\text{F}_2\text{NaO}_5$, 519.1954, found 519.1955.



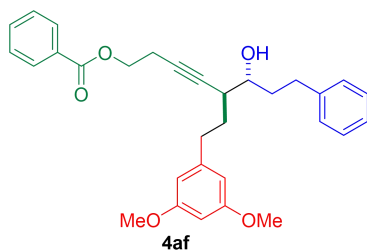
4ac, $R_f = 0.5$ (EA:PE=1:3), column solvent: hexane/EtOAc = 10/1, 66%, colorless oil, $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.04 (d, $J = 8.1$ Hz, 2H), 7.53 (t, $J = 7.4$ Hz, 1H), 7.40 (t, $J = 7.4$ Hz, 2H), 7.28 – 7.14 (m, 7H), 7.12 (d, $J = 7.6$ Hz, 2H), 5.70 – 5.51 (m, 1H), 4.41 (t, $J = 6.3$ Hz, 2H), 3.52 – 3.42 (m, 1H), 3.35 (dd, $J = 17.2, 6.5$ Hz, 2H), 3.12 – 3.00 (m, 2H), 2.76 – 2.66 (m, 2H), 2.66 – 2.53 (m, 3H), 2.46 – 2.20 (m, 2H), 1.94 – 1.71 (m, 3H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 166.47, 163.76 (t, $J = 32.7$ Hz), 141.41, 139.53, 133.13, 129.74, 129.60, 128.38, 128.33, 126.98, 125.87, 124.59, 115.36 (dd, $J = 255.1, 247.3$ Hz), 80.89, 78.84, 78.00, 72.31, 62.69, 39.25, 39.21, 37.01, 36.88 (t, $J = 22.8$ Hz), 32.61 – 32.39 (m), 31.92, 19.59. $^{19}\text{F NMR}$ (471 MHz, CDCl_3) δ -102.65 – -103.63 (m, 1F), -105.44 (ddd, $J = 35.0, 23.9, 10.7$ Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{33}\text{H}_{32}\text{F}_2\text{NaO}_5$, 569.2110, found 569.2119.



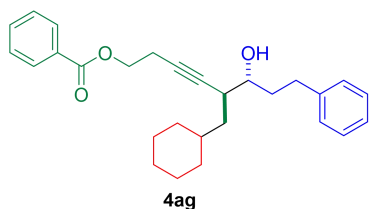
4ad, $R_f = 0.5$ (EA:PE=1:3), column solvent: hexane/EtOAc = 10/1, 53%, colorless oil, $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.12 (d, $J = 7.6$ Hz, 2H), 7.63 (t, $J = 7.4$ Hz, 1H), 7.50 (t, $J = 7.7$ Hz, 2H), 7.37 – 7.30 (m, 3H), 7.23 (d, $J = 7.3$ Hz, 2H), 4.58 – 4.36 (m, 4H), 3.53 – 3.38 (m, 1H), 2.88 – 2.62 (m, 4H), 2.53 – 2.41 (m, 1H), 2.11 – 1.75 (m, 4H), 1.66 (s, 1H), 1.33 (s, 3H), 1.29 (s, 3H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 176.01, 166.44, 141.75, 133.15, 129.81, 129.63, 128.42, 128.40, 128.37, 125.83, 123.01 (d, $J = 277.4$ Hz), 80.66, 73.39, 62.80, 60.29 (q, $J = 36.5$ Hz), 42.04, 41.94, 37.01, 35.58, 32.03, 25.80, 25.08, 19.56. $^{19}\text{F NMR}$ (471 MHz, CDCl_3) δ -73.71 (t, $J = 8.3$ Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{28}\text{H}_{32}\text{F}_3\text{O}_5$, 505.2196, found 505.2196.



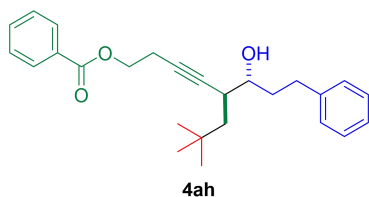
4ae, $R_f = 0.6$ (EA:PE=1:3), column solvent: hexane/EtOAc = 15/1, 62%, colorless oil, $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.09 (d, $J = 7.6$ Hz, 2H), 7.58 (t, $J = 7.4$ Hz, 1H), 7.45 (t, $J = 7.7$ Hz, 2H), 7.28 (d, $J = 5.7$ Hz, 2H), 7.19 (t, $J = 7.6$ Hz, 3H), 4.49 (t, $J = 6.6$ Hz, 2H), 3.59 – 3.42 (m, 1H), 2.94 – 2.62 (m, 4H), 2.54 – 2.34 (m, 1H), 1.99 – 1.74 (m, 2H), 1.64 (s, 1H), 1.56 – 1.26 (m, 4H), 0.89 (t, $J = 6.9$ Hz, 3H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 166.46, 141.80, 133.09, 129.92, 129.63, 128.45, 128.38, 128.30, 125.80, 81.00, 80.41, 73.01, 63.10, 38.64, 37.88, 33.72, 33.70, 19.64, 18.98, 13.96. HRMS-ESI (m/z) $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{24}\text{H}_{29}\text{O}_3$, 365.2111, found 365.2111.



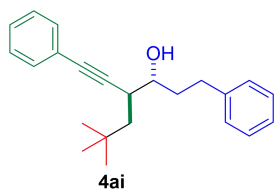
4af, $R_f = 0.4$ (EA:PE=1:3), column solvent: hexane/EtOAc = 5/1, 57%, colorless oil, $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.14 (d, $J = 7.9$ Hz, 2H), 7.63 (t, $J = 7.2$ Hz, 1H), 7.49 (t, $J = 7.6$ Hz, 2H), 7.33 (d, $J = 9.2$ Hz, 2H), 7.26 – 7.17 (m, 3H), 6.40 (s, 2H), 6.38 (s, 1H), 4.55 (t, $J = 6.3$ Hz, 2H), 3.84 (s, 6H), 3.70 – 3.42 (m, 1H), 2.93 – 2.61 (m, 6H), 2.59 – 2.43 (m, 1H), 2.02 – 1.73 (m, 5H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 166.46, 160.70, 144.10, 141.87, 133.10, 129.86, 129.61, 128.42, 128.39, 128.34, 125.77, 106.49, 97.83, 80.78, 80.67, 72.48, 63.06, 55.21, 38.70, 37.38, 33.95, 33.42, 32.03, 19.65. HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{31}\text{H}_{35}\text{O}_5$, 487.2479, found 487.2479.



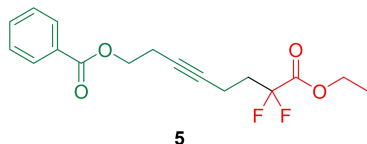
4ag, $R_f = 0.6$ (EA:PE=1:3), column solvent: hexane/EtOAc = 15/1, 46%, colorless oil, $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.04 (d, $J = 7.9$ Hz, 2H), 7.53 (t, $J = 7.2$ Hz, 1H), 7.40 (t, $J = 7.6$ Hz, 2H), 7.24 (d, $J = 7.4$ Hz, 2H), 7.15 (m, 3H), 4.41 (t, $J = 6.4$ Hz, 2H), 3.55 – 3.30 (m, 1H), 2.83 – 2.56 (m, 4H), 2.55 – 2.41 (m, 1H), 1.90 – 1.76 (m, 3H), 1.75 – 1.67 (m, 1H), 1.65 – 1.54 (m, 4H), 1.51 – 1.33 (m, 2H), 1.25 – 1.03 (m, 4H), 0.94 – 0.80 (m, 1H), 0.79 – 0.64 (m, 1H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 166.31, 141.88, 132.94, 129.80, 129.49, 128.30, 128.24, 128.20, 125.61, 81.12, 79.71, 72.65, 62.99, 39.32, 37.28, 36.32, 35.14, 33.85, 32.16, 31.97, 26.42, 26.06, 25.95, 19.48. HRMS-ESI (m/z) $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{28}\text{H}_{35}\text{O}_3$, 419.2581, found 419.2581.



4ah, $R_f = 0.6$ (EA:PE=1:3), column solvent: hexane/EtOAc = 15/1, 43%, colorless oil, $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.03 (d, $J = 7.4$ Hz, 2H), 7.53 (t, $J = 7.4$ Hz, 1H), 7.40 (t, $J = 7.7$ Hz, 2H), 7.27 – 7.21 (m, 3H), 7.16 (d, $J = 6.4$ Hz, 2H), 4.39 (t, $J = 8.5$ Hz, 2H), 3.49 – 3.28 (m, 1H), 2.87 – 2.72 (m, 1H), 2.69 – 2.58 (m, 3H), 2.57 – 2.33 (m, 1H), 1.86 – 1.74 (m, 2H), 1.61 – 1.51 (m, 2H), 1.36 – 1.17 (m, 2H), 0.89 (s, 9H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 166.44, 142.14, 142.00, 133.07, 129.94, 129.65, 128.44, 128.36, 125.77, 82.75, 79.86, 74.21, 63.06, 46.19, 37.07, 35.14, 32.20, 30.72, 29.75, 19.60. HRMS-ESI (m/z) $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{26}\text{H}_{33}\text{O}_3$, 393.2424, found 393.2424.

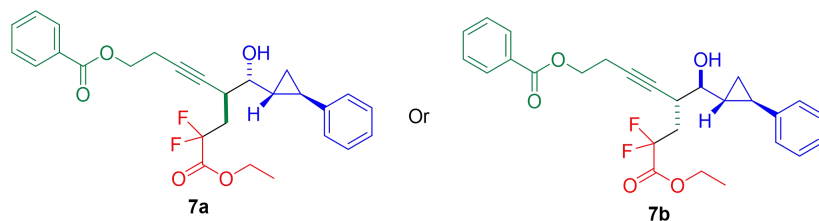


4ai, $R_f = 0.6$ (EA:PE=1:3), column solvent: hexane/EtOAc = 15/1, 62%, colorless oil, $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.37 – 7.30 (m, 2H), 7.26 – 7.21 (m, 5H), 7.19 (d, $J = 8.2$ Hz, 2H), 7.14 (t, $J = 7.3$ Hz, 1H), 3.59 – 3.44 (m, 1H), 2.88 – 2.60 (m, 3H), 1.97 – 1.87 (m, 2H), 1.85 – 1.80 (m, 1H), 1.72 – 1.62 (m, 1H), 1.47 – 1.37 (m, 1H), 0.96 (s, 9H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 141.95, 131.46, 128.48, 128.39, 128.24, 127.85, 125.83, 123.48, 90.57, 84.43, 74.26, 45.97, 37.08, 35.79, 32.25, 30.80, 29.83. HRMS-ESI (m/z) $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{23}\text{H}_{29}\text{O}$, 321.2213, found 321.2213.



5, $R_f = 0.6$ (EA:PE=1:3), column solvent: hexane/EtOAc = 20/1, 84%, colorless oil, $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.05 (d, $J = 8.5$ Hz, 2H), 7.56 (t, $J = 7.4$ Hz, 1H), 7.44 (t, $J = 7.7$ Hz, 2H), 4.37 (t, $J = 7.0$ Hz, 2H), 4.32 (q, $J = 7.1$ Hz, 2H), 2.66 – 2.56 (m, 2H), 2.42 – 2.34 (m, 2H), 2.34 – 2.19 (m, 2H), 1.35 (t, $J = 7.1$ Hz, 3H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 166.29, 163.73 (t, $J = 33.3$ Hz), 133.00, 130.01, 129.60,

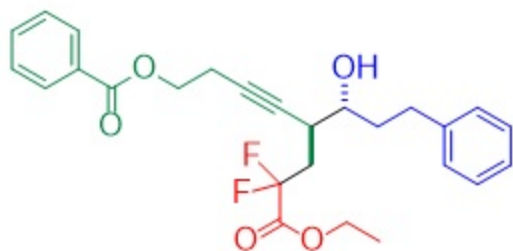
128.34, 115.14 (dd, $J = 251.0, 250.1$ Hz), 78.97, 76.79, 62.93, 62.91, 34.02 (t, $J = 23.5$ Hz), 19.27, 13.89, 11.74 (t, $J = 6.1$ Hz). ^{19}F NMR (471 MHz, CDCl_3) δ -101.57 – -103.57 (m, 1F), -105.37 – -106.74 (m, 1F). HRMS-ESI (m/z) $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{17}\text{H}_{19}\text{F}_2\text{O}_4$, 325.1246, found 325.1243.



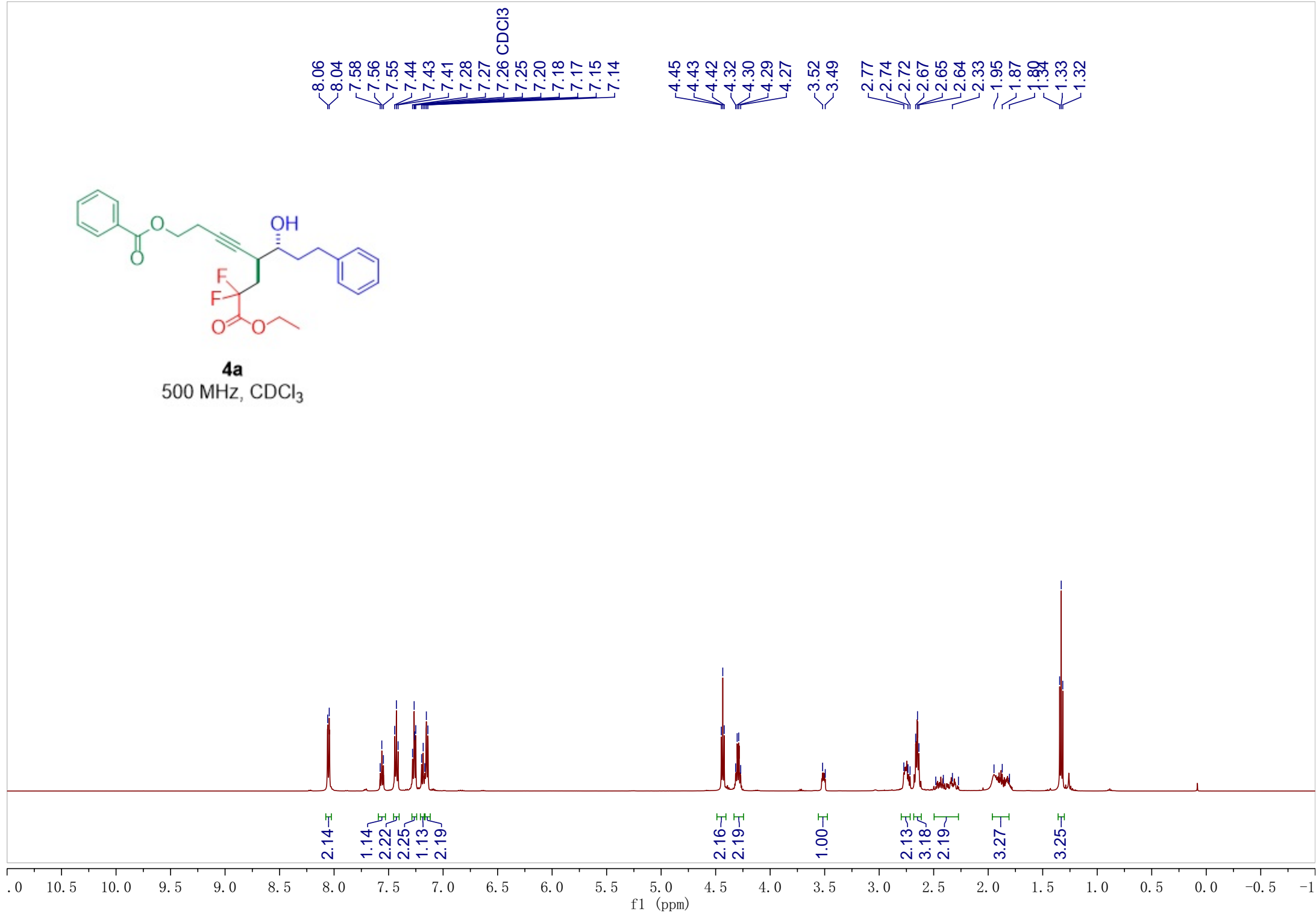
7a (major product), $R_f = 0.5$ (EA:PE=1:3), column solvent: hexane/EtOAc = 10/1, 43%, colorless oil, ^1H NMR (500 MHz, CDCl_3) δ 8.05 (d, $J = 7.0$ Hz, 2H), 7.57 (t, $J = 7.4$ Hz, 1H), 7.44 (t, $J = 7.8$ Hz, 2H), 7.22 (t, $J = 7.5$ Hz, 2H), 7.14 (t, $J = 7.3$ Hz, 1H), 6.98 (d, $J = 7.1$ Hz, 2H), 4.48 – 4.35 (m, 2H), 4.33 – 4.25 (m, 2H), 3.13 – 3.04 (m, 1H), 2.98 – 2.91 (m, 1H), 2.68 – 2.59 (m, 2H), 2.51 – 2.34 (m, 2H), 1.87 – 1.77 (m, 1H), 1.43 – 1.36 (m, 1H), 1.33 (t, $J = 7.2$ Hz, 3H), 1.10 – 1.02 (m, 1H), 0.93 – 0.83 (m, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 166.50, 163.84 (t, $J = 32.7$ Hz), 141.71, 133.17, 129.82, 129.69, 128.41, 128.33, 126.02, 125.78, 115.51 (dd, $J = 252.3, 249.9$ Hz), 80.81, 79.28, 76.61, 62.89, 62.67, 36.79 (t, $J = 23.0$ Hz), 33.18 – 32.56 (m), 26.81, 21.22, 19.64, 13.90, 13.70. ^{19}F NMR (471 MHz, CDCl_3) δ -102.53 – -103.40 (m, 1F), -106.20 (dt, $J = 36.3, 18.5$ Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{27}\text{H}_{28}\text{F}_2\text{NaO}_5$, 493.1797, found 493.1807.

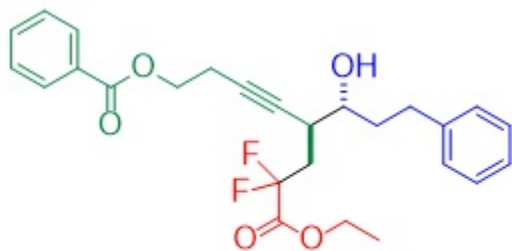
7b (minor product), $R_f = 0.5$ (EA:PE=1:3), column solvent: hexane/EtOAc = 10/1, 21%, ^1H NMR (500 MHz, CDCl_3) δ 8.02 (d, $J = 7.2$ Hz, 2H), 7.54 (t, $J = 7.4$ Hz, 1H), 7.40 (t, $J = 7.8$ Hz, 2H), 7.24 (t, $J = 7.5$ Hz, 2H), 7.15 (t, $J = 7.3$ Hz, 1H), 7.06 (d, $J = 7.3$ Hz, 2H), 4.44 – 4.37 (m, 2H), 4.31 (dd, $J = 14.1, 7.0$ Hz, 2H), 3.12 – 3.04 (m, 1H), 3.01 – 2.90 (m, 1H), 2.69 – 2.61 (m, 2H), 2.50 – 2.36 (m, 2H), 2.04 – 1.98 (m, 1H), 1.61 (s, 1H), 1.46 – 1.39 (m, 1H), 1.34 (t, $J = 7.2$ Hz, 3H), 1.02 – 0.90 (m, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 166.40, 163.86 (t, $J = 32.5$ Hz), 142.12, 133.11, 129.80, 129.60, 128.38, 128.33, 125.91, 125.72, 115.53 (dd, $J = 252.1, 249.8$ Hz), 80.71, 79.20, 76.90, 62.90, 62.71, 36.94 (t, $J = 23.0$ Hz), 32.75 – 32.37 (m), 27.39, 21.45, 19.57, 13.92, 13.31. ^{19}F NMR (471 MHz, CDCl_3) δ -102.62 – -103.48 (m, 1F), -106.24 (dt, $J = 36.3, 17.8$ Hz, 1F). HRMS-ESI (m/z) $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{27}\text{H}_{28}\text{F}_2\text{NaO}_5$, 493.1797, found 493.1807.

8. NMR spectra of the products



4a
500 MHz, CDCl₃





4a
126 MHz, CDCl₃

166.50
164.11
163.85
163.59

141.42
133.15
129.61
128.39
128.35
125.89
117.80
115.51
115.50
113.51

80.89
78.81
77.25
77.00 CDCl₃
76.75
72.40
62.85
62.71

37.22
37.07
37.03
36.85
32.53
32.50
32.48
31.94
19.58
13.87

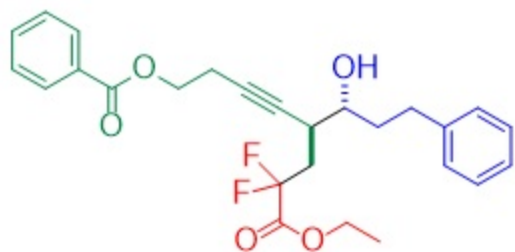
A (t)
163.85
H

B (dd)
115.51
H

D (t)
37.03
H

C (m)
32.50
H

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



4a

471 MHz, CDCl₃

102.81
102.84
102.88
103.37
103.40
103.43

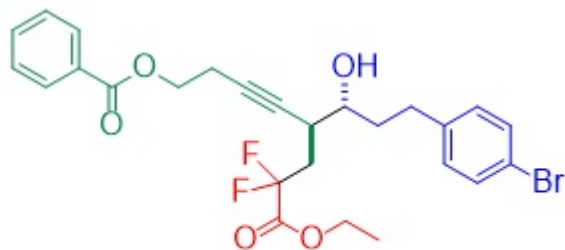
105.89
105.93
105.97
106.45
106.49
106.53

-96 -97 -98 -99 -100 -101 -102 -103 -104 -105 -106 -107 -108 -109 -110 -111 -112 -113 -114

f1 (ppm)

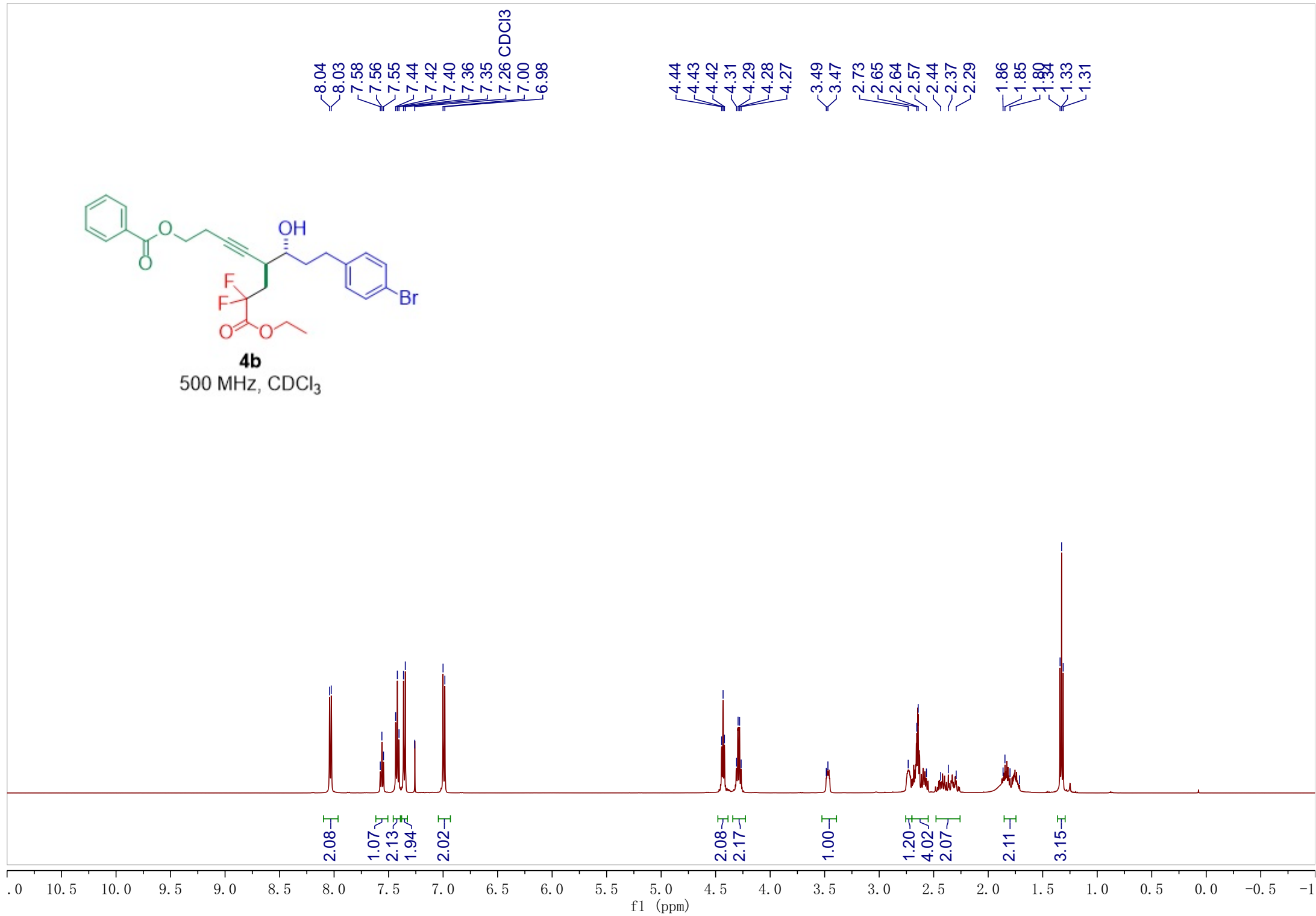
1.00

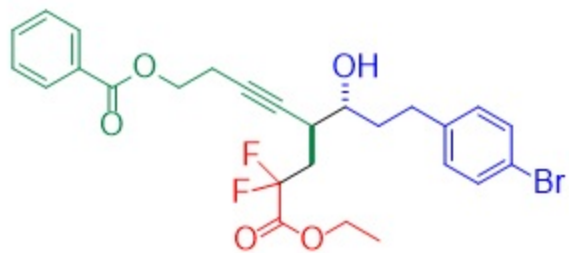
1.00



4b

500 MHz, CDCl₃





4b

126 MHz, CDCl₃

166.52
164.08
163.83
163.57

140.40
131.41
130.12
129.60
119.60
117.46
115.47
115.46
113.47

81.06
78.70
77.25
77.00 CDCl₃
76.75
72.20
62.89
62.68

37.20
37.02
36.93
36.84
32.56
32.53
31.35
19.62
13.88

A (t)
163.83
H

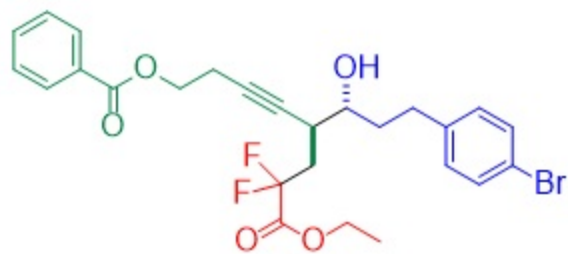
B (dd)
115.46
H

C (t)
37.02
H H

D (m)
32.54

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

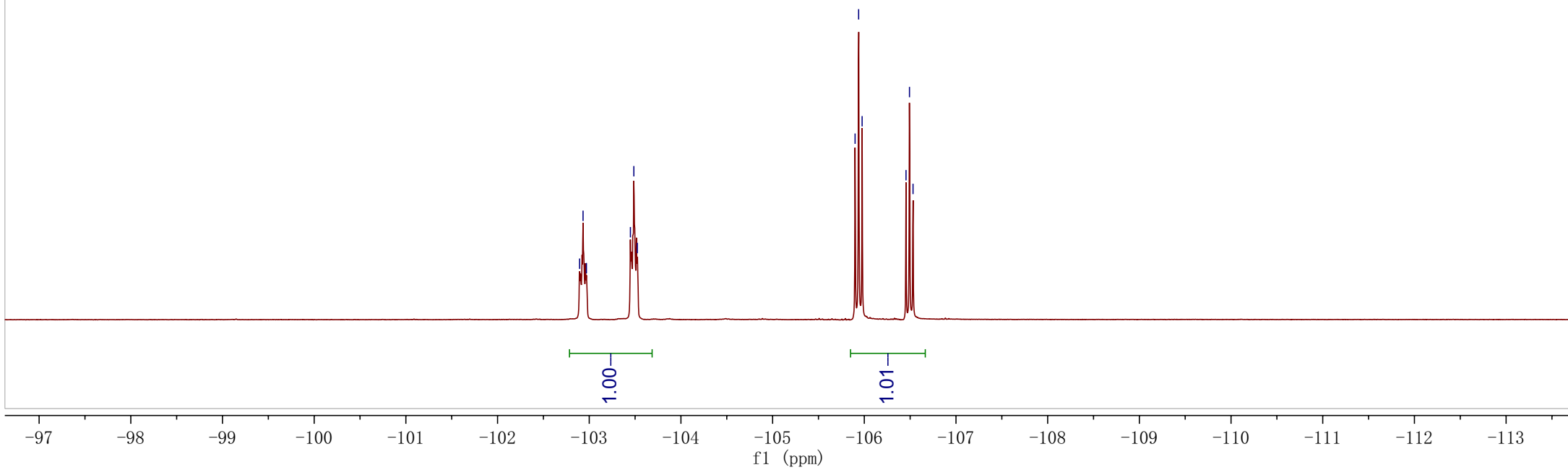
f1 (ppm)

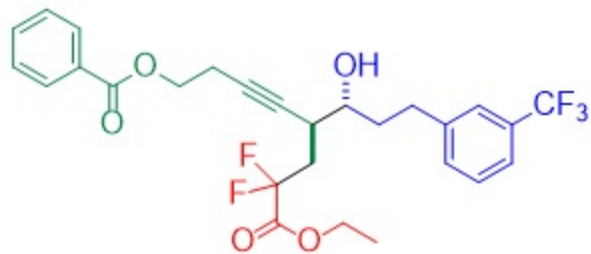


4b
471 MHz, CDCl₃

102.89
102.93
102.97
103.45
103.49
103.52

105.90
105.94
105.98
106.45
106.49
106.53



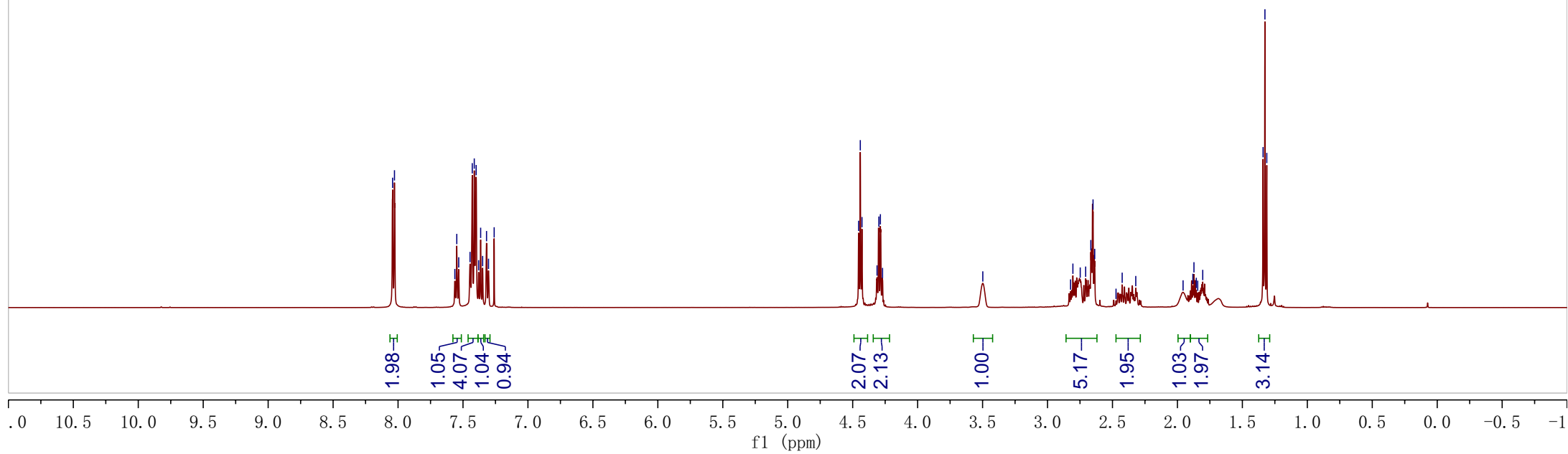


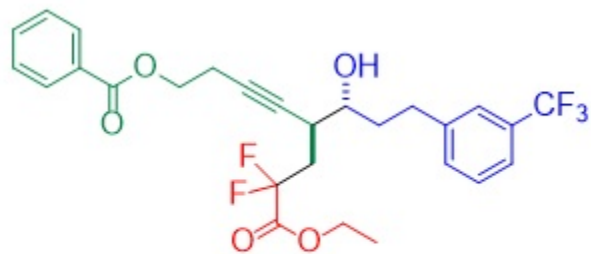
4c

500 MHz, CDCl₃

8.04
8.03
7.56
7.55
7.53
7.45
7.43
7.41
7.40
7.38
7.36
7.35
7.32
7.30
7.26 CDCl₃

4.45
4.44
4.43
4.31
4.30
4.29
4.27
— 3.50
2.80
2.75
2.71
2.67
2.65
2.64
2.43
1.88
1.87
1.84
1.33
1.31





4c
126 MHz, CDCl₃

166.55
164.09
163.83
163.57

142.39

133.20

131.79

130.76

130.51

129.72

129.60

128.80

128.40

125.08

125.05

125.02

124.99

122.83

122.80

122.77

81.16

78.68

77.25

77.00 CDCl₃

76.75

72.23

62.91

62.65

37.19

37.00

36.85

32.64

32.62

32.59

31.78

19.63

13.87

A (t)
163.83
H

B (q)
130.64
H

F (q)
124.17

C (q)
125.04

E (m)
115.46

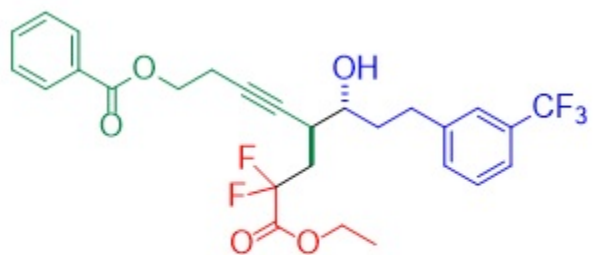
D (q)
122.81

H (t)
37.01

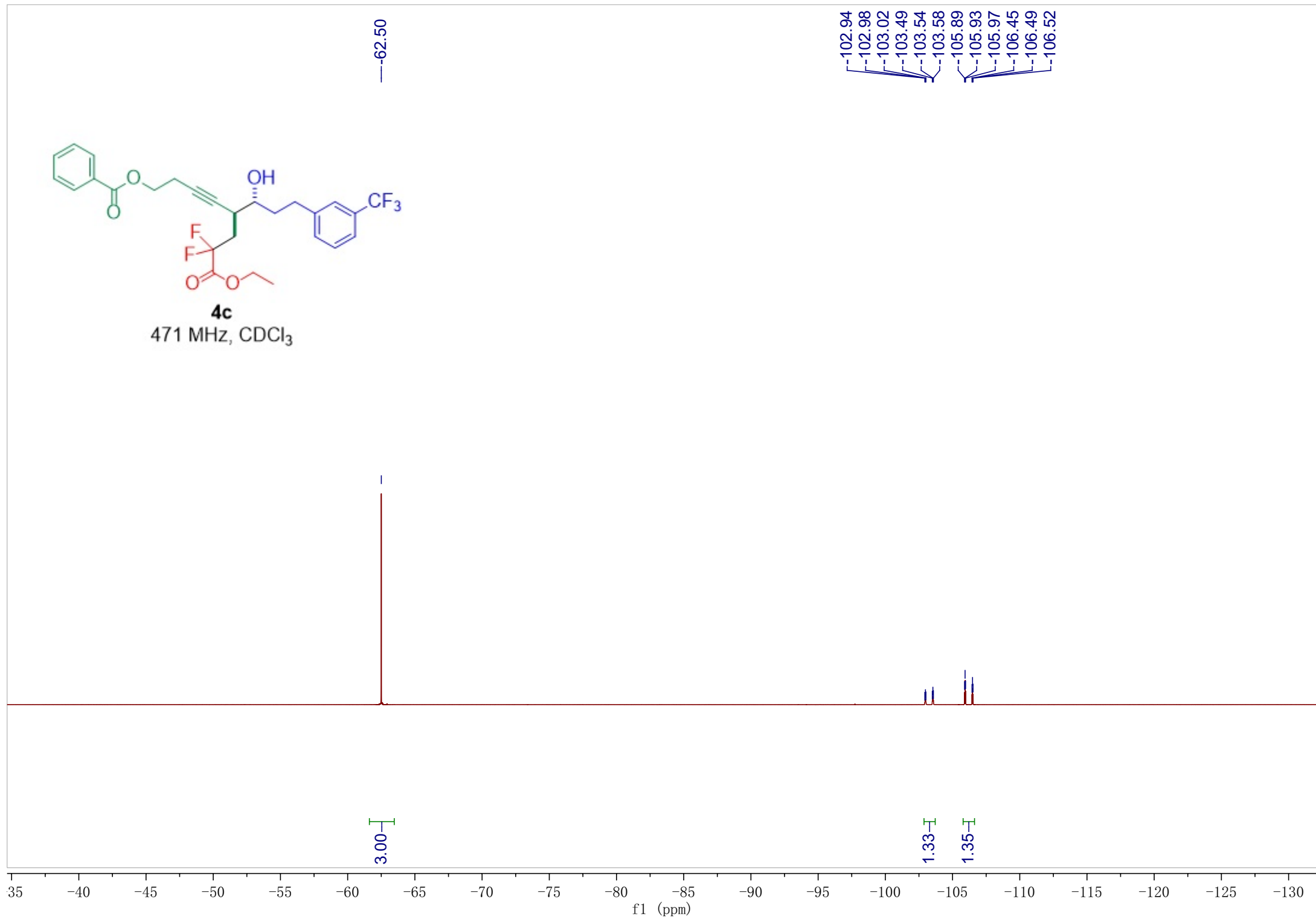
G (m)
32.60
H H

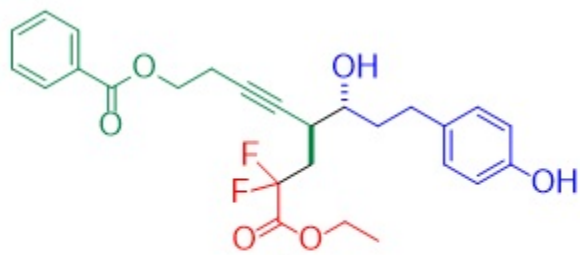
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

f1 (ppm)



4c
471 MHz, CDCl₃





4d
500 MHz, CDCl₃

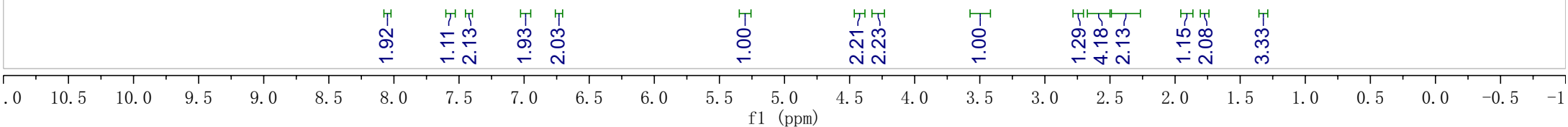
8.05
8.04
7.58
7.56
7.55
7.44
7.43
7.41
7.26 CDCl₃
7.00
6.98
6.74
6.72

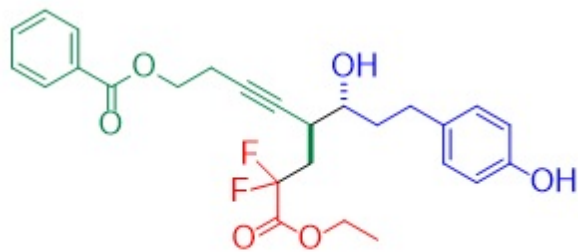
5.30

4.44
4.42
4.41
4.31
4.30
4.29
4.28
4.28
4.27
3.45

2.66
2.64
2.63
2.58
2.45
2.42
2.33

1.92
1.77
1.34
1.33
1.31





4d
126 MHz, CDCl₃

166.64
164.18
163.92
163.66
— 153.86

133.37
133.22
129.72
129.65
129.43
128.43
117.51
115.52
115.51
115.26
113.52

80.86
78.85
77.25
77.00 CDCl₃
76.75
72.39
62.92
62.78

37.24
37.20
37.02
36.84
32.47
31.00
— 19.59
— 13.89

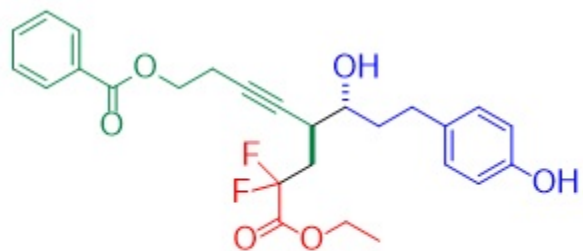
A (t)
163.92
H

B (dd)
115.52
H

D (t)
37.02
H

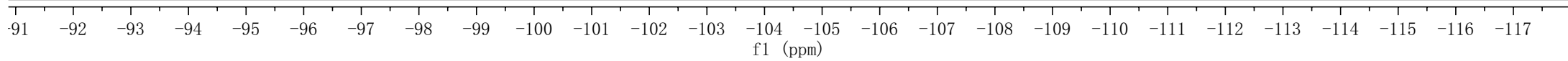
C (m)
32.47
H

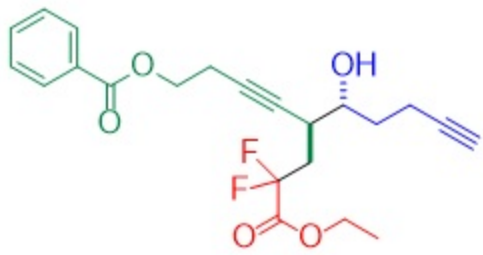
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



4d
471 MHz, CDCl₃

102.83
102.85
102.86
102.89
103.38
103.41
103.42
103.44
105.91
105.95
105.99
106.47
106.50
106.54

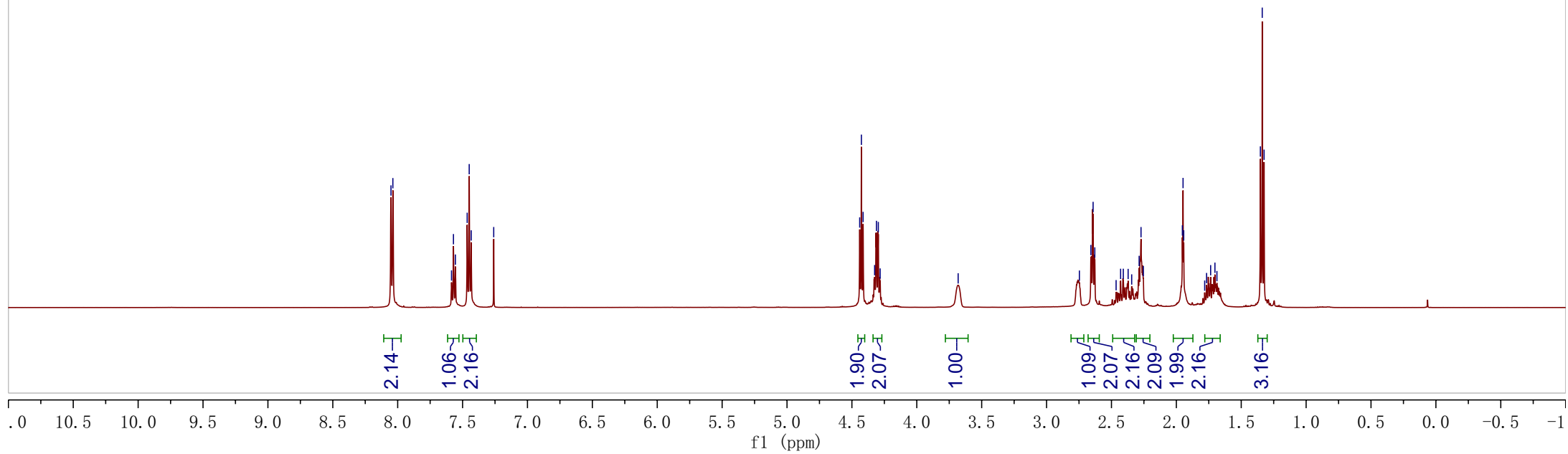


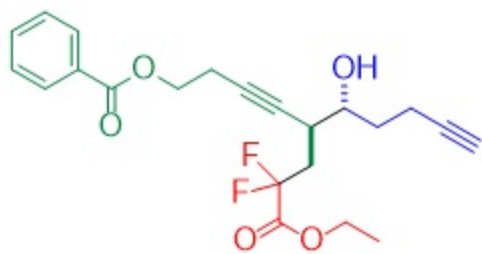


4e
500 MHz, CDCl₃

8.05
8.04
7.59
7.57
7.56
7.46
7.45
7.43
7.26 CDCl₃

4.44
4.43
4.41
4.32
4.31
4.30
4.28
3.68
2.75
2.66
2.64
2.63
2.46
2.43
2.41
2.37
2.34
2.29
2.27
2.25
1.95
1.95
1.94
1.78
1.77
1.74
1.70
1.69
1.35
1.34
1.32





4e
126 MHz, CDCl₃

166.49
164.10
163.84
163.58

133.18
129.77
129.62
128.42
117.47
115.48
115.46
113.47

83.43
80.99
78.68
77.25
77.00 CDCl₃
76.75
71.84
69.01
62.90
62.67

37.09
36.91
36.73
33.90
32.43
32.40
19.58
14.99
13.90

A (t)
163.84
H

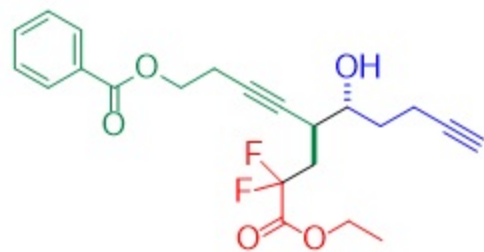
B (dd)
115.47
H

C (t)
36.91
H

D (m)
32.42
H

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

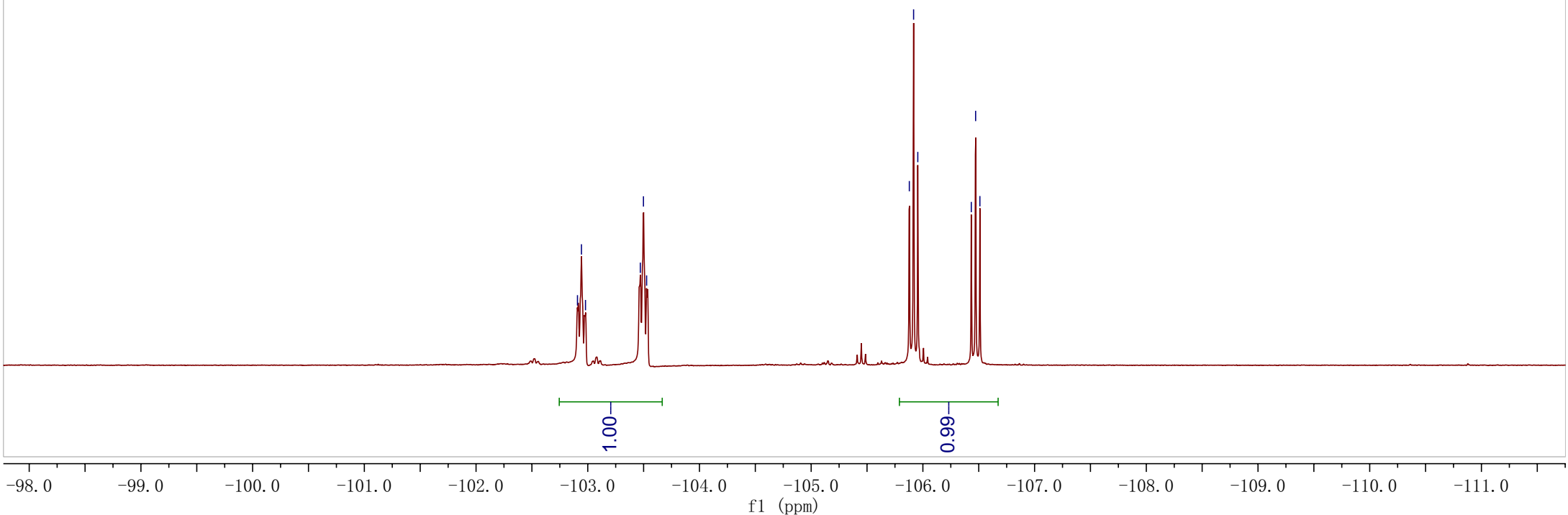
f1 (ppm)

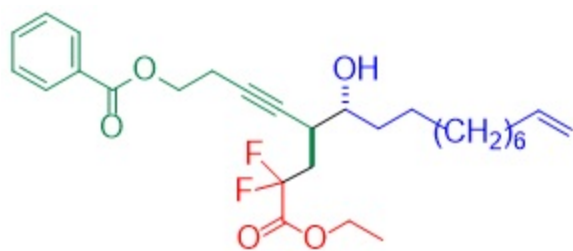


4e
471 MHz, CDCl₃

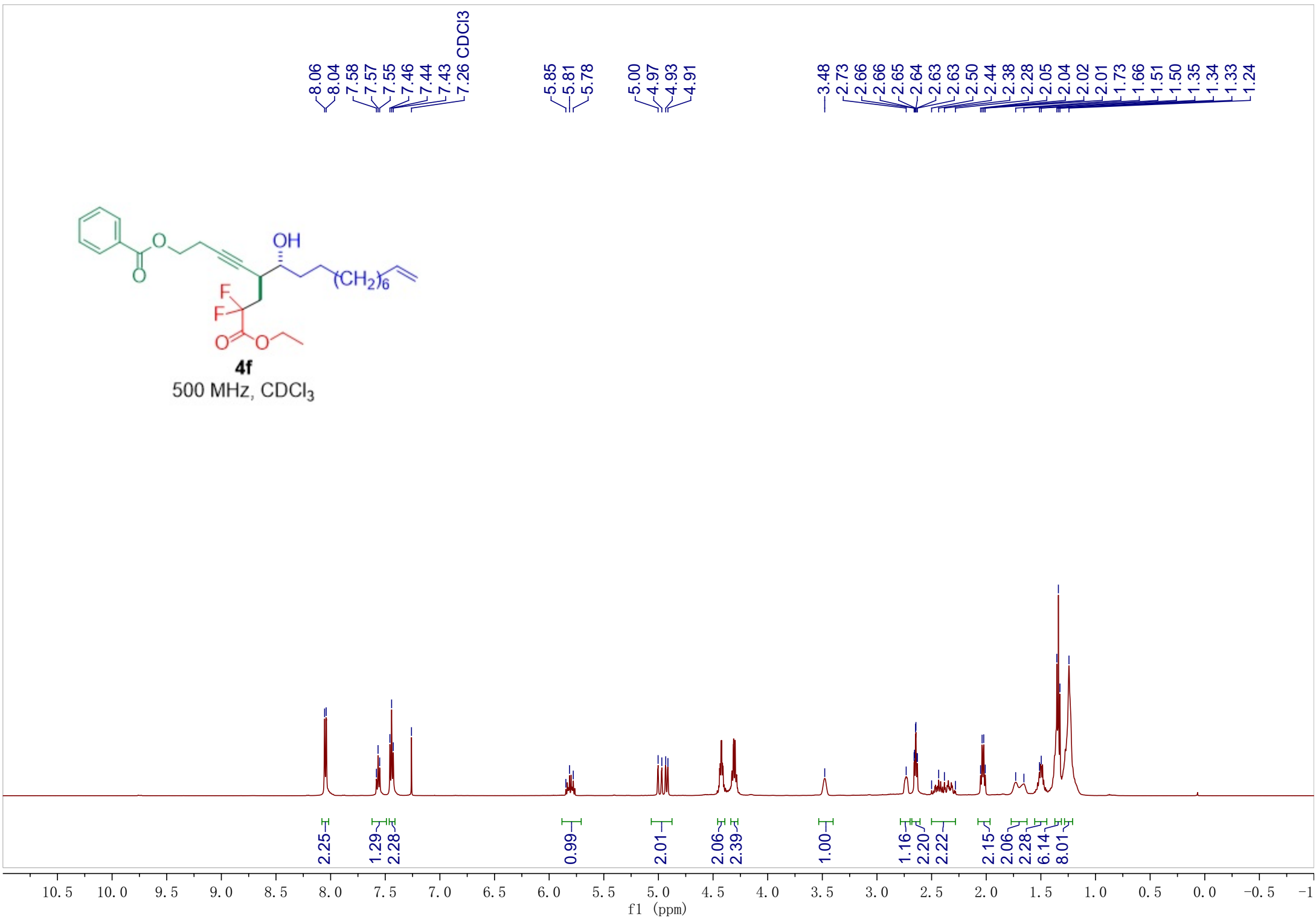
102.91
102.94
102.98
103.47
103.50
103.53

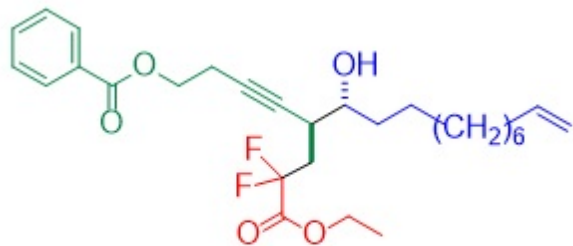
105.88
105.92
105.95
106.43
106.47
106.51





4f
500 MHz, CDCl₃





4f
126 MHz, CDCl₃

166.47
164.18
163.92
163.66

139.18
133.14
129.82
129.63
128.38
117.59
115.61
115.59
114.10
113.60

80.63
78.96
77.25
77.00 CDCl₃
76.75
73.22
62.85
62.75

37.33
37.14
36.96
35.56
33.77
32.35
29.47
29.39
29.35
29.06
28.88
25.78
19.57
13.90

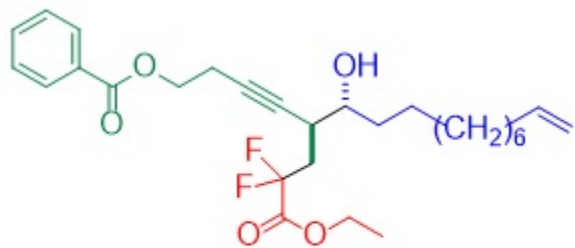
A (t)
163.92
H

B (dd)
115.60
H

C (t)
37.14
H H

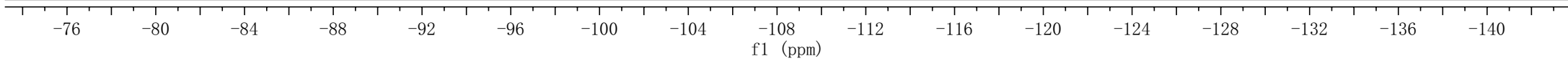
D (m)
32.35

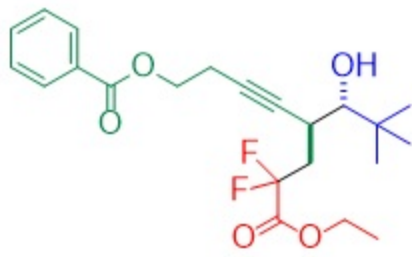
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



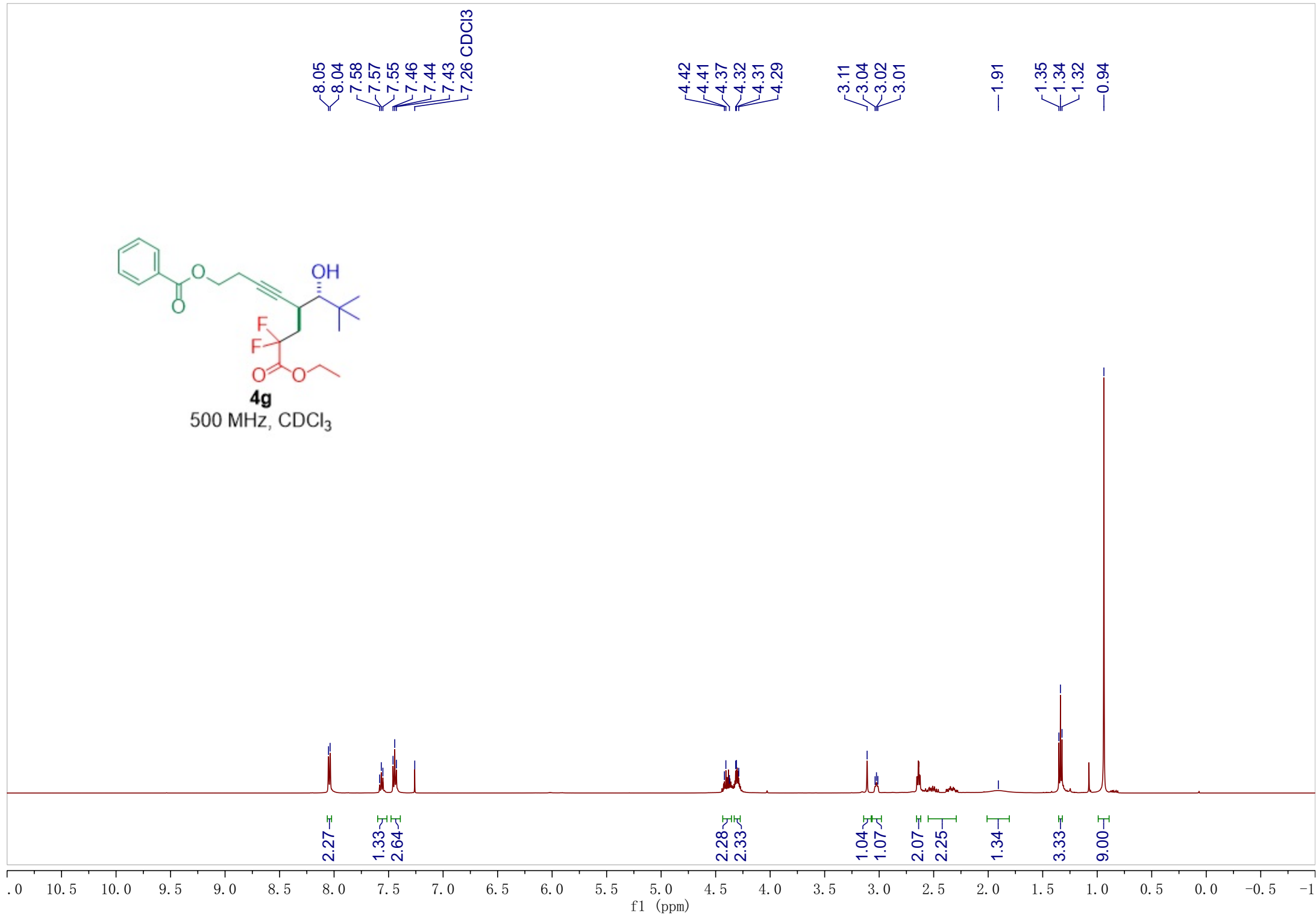
4f
471 MHz, CDCl₃

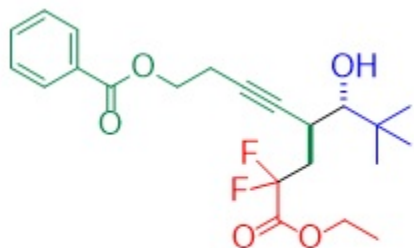
102.77
102.80
102.83
103.32
103.35
103.39
105.98
106.02
106.06
106.53
106.57
106.61





4g
500 MHz, CDCl₃





4g
126 MHz, CDCl₃

166.38
164.11
163.85
163.59

133.13
129.77
129.62
128.37
117.48
115.49
115.48
113.49

82.56
80.38
79.41
77.25
77.00 CDCl₃
76.75
62.87
62.62
62.34

39.89
39.71
39.53
36.04

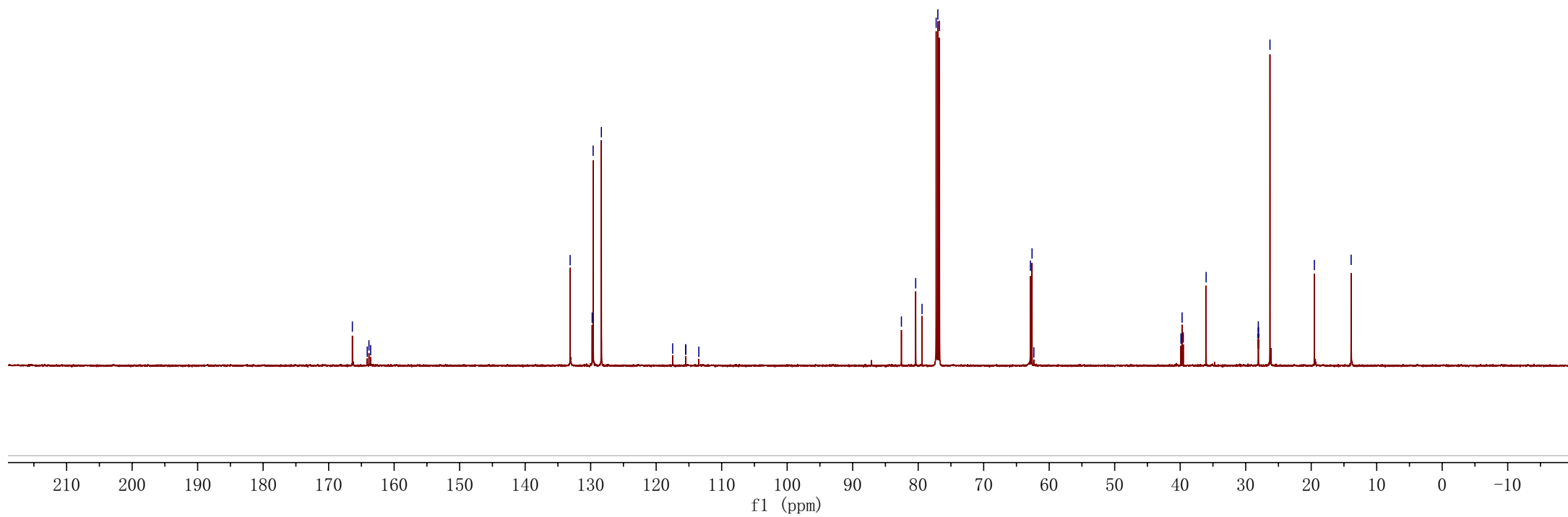
28.07
28.04
26.80
— 13.90

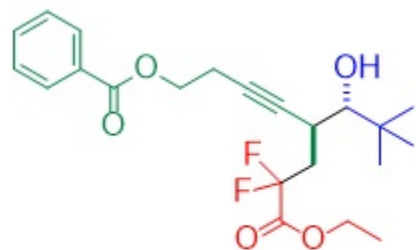
A (t)
163.85
H

B (dd)
115.48
H

D (t)
39.71
H

C (m)
28.07
H





4g

471 MHz, CDCl₃

102.66
102.69
102.73

103.22
103.25
103.29

106.17
106.21
106.25

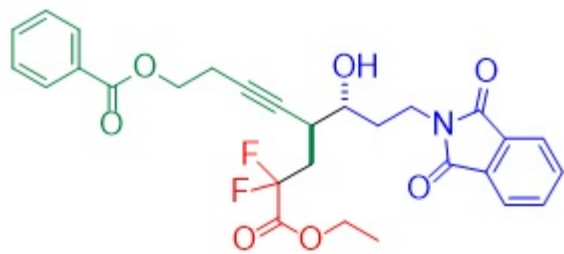
106.73
106.77
106.81

-99.5 -100.5 -101.5 -102.5 -103.5 -104.5 -105.5 -106.5 -107.5 -108.5 -109.5

f1 (ppm)

1.00

1.02



4h
500 MHz, CDCl₃

8.03
8.01
7.83
7.82
7.71
7.71
7.70
7.54
7.53
7.51
7.43
7.42
7.40
7.26 CDCl₃

4.40
4.39
4.38
4.30
4.29
4.27
4.26
3.81
3.80
3.78
3.54
2.84
2.75
2.64
2.63
2.61
2.42
2.36
1.98
1.95
1.85
1.31
1.30

2.04
2.17
2.17
1.21
2.37

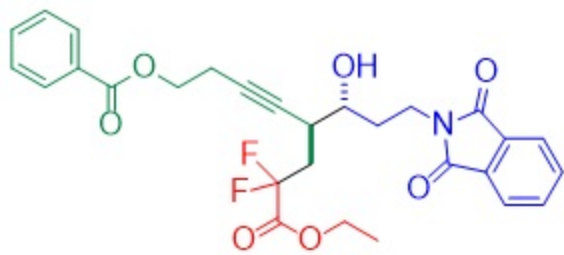
2.01
2.12
1.96
1.00

0.94
1.10
2.12
2.08

1.19
1.16
3.20

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

f1 (ppm)



4h
126 MHz, CDCl₃

168.69
166.31
164.06
163.80
163.54

134.06
133.02
131.88
129.82
129.58
128.32
123.31
117.47
115.48
115.47
113.48

80.59
78.76
77.25
77.00 CDCl₃
76.75
69.98
62.83
62.69

36.92
36.74
36.56
34.62
34.02
32.12
19.44
13.85

A (t)
163.80
H

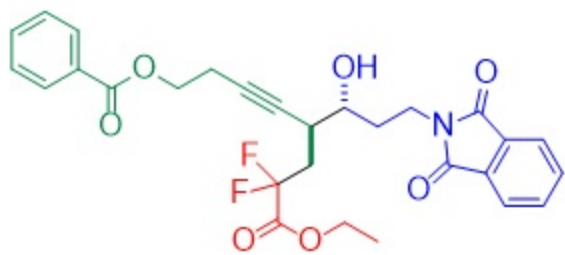
B (dd)
115.47
H

C (t)
36.74
H

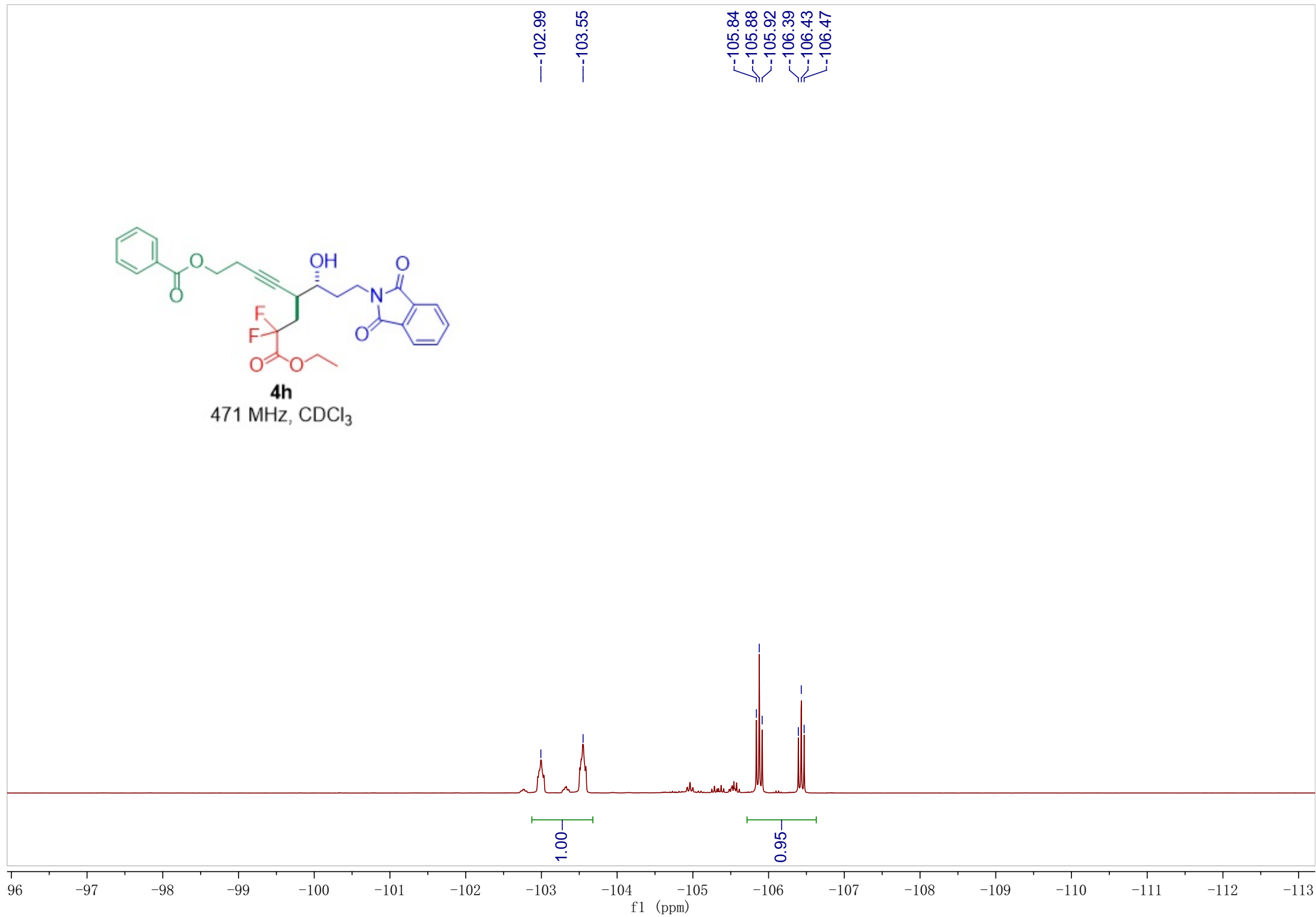
D (m)
32.10
H

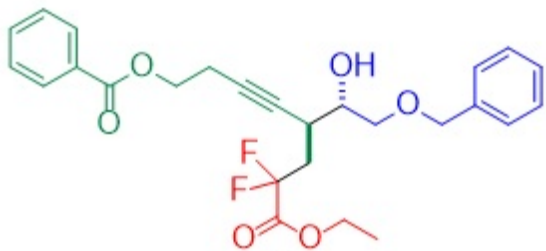
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

f1 (ppm)

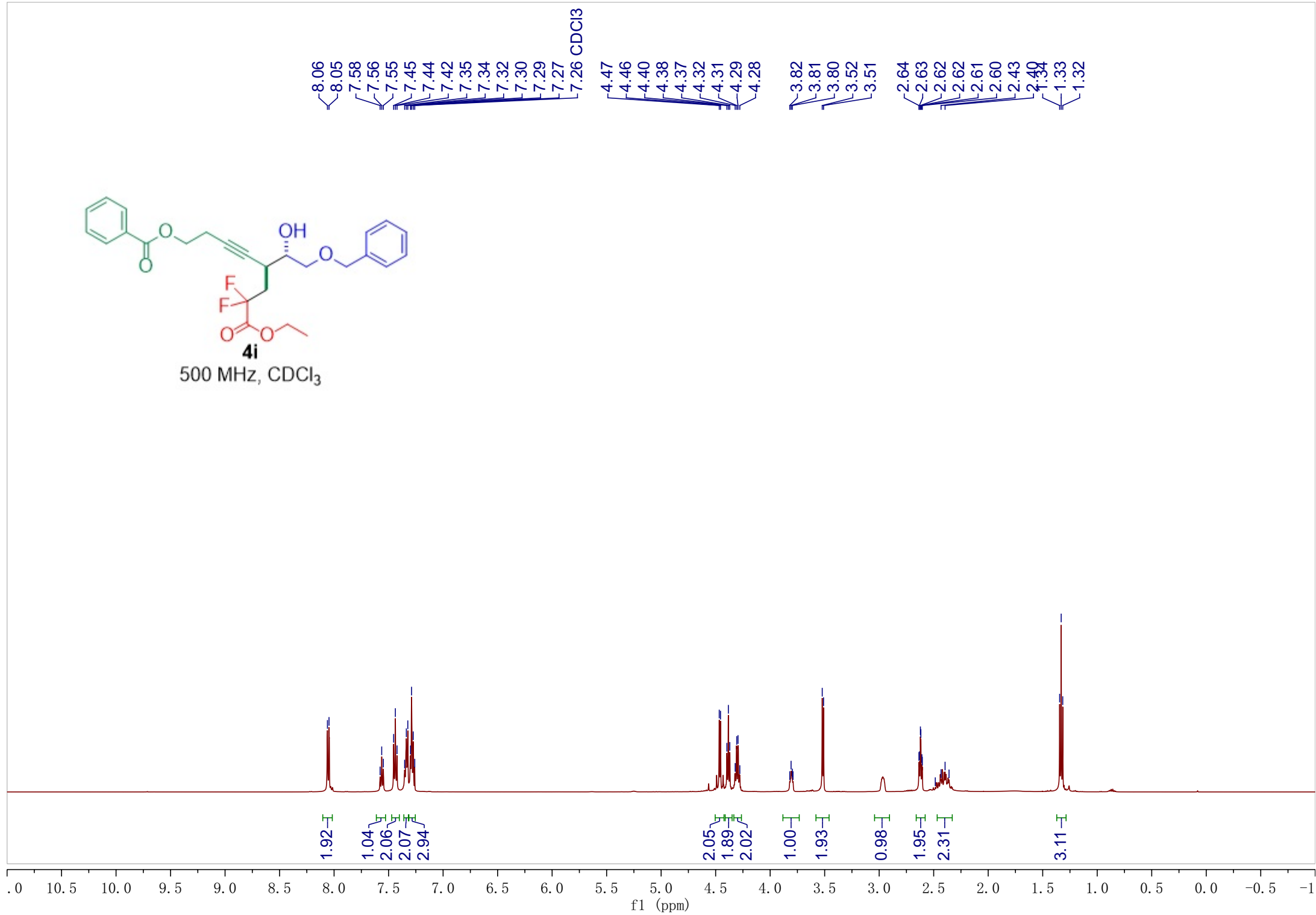


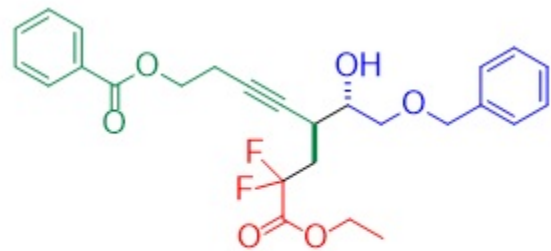
4h
471 MHz, CDCl₃





500 MHz, CDCl₃





4i

126 MHz, CDCl₃

166.39
164.08
163.82
163.56

137.68
133.11
129.83
129.62
128.38
128.32
127.75
127.68
117.46
115.47
115.45
113.46

80.51
78.53
77.25
77.00 CDCl₃
76.75
73.37
73.29
71.88
71.29
62.84
62.68

36.58
36.39
36.21
29.17
29.13
29.10
— 19.47
— 13.86

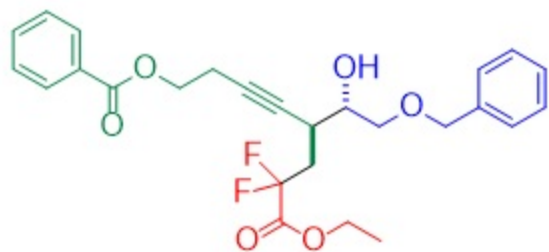
A (t)
163.82
H

B (dd)
115.46
H

C (t)
36.39
H

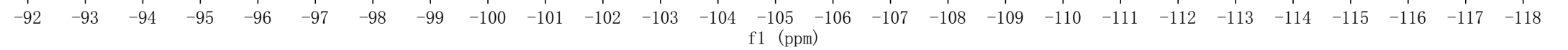
D (m)
29.14
H

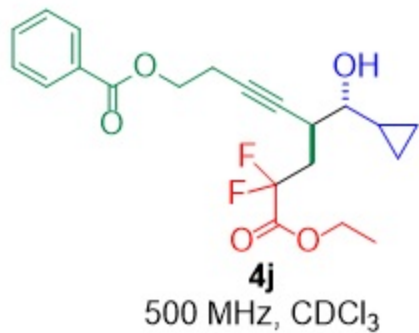
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



4i
471 MHz, CDCl₃

102.82
102.85
102.89
103.38
103.41
103.44
105.94
105.98
106.01
106.49
106.53
106.57



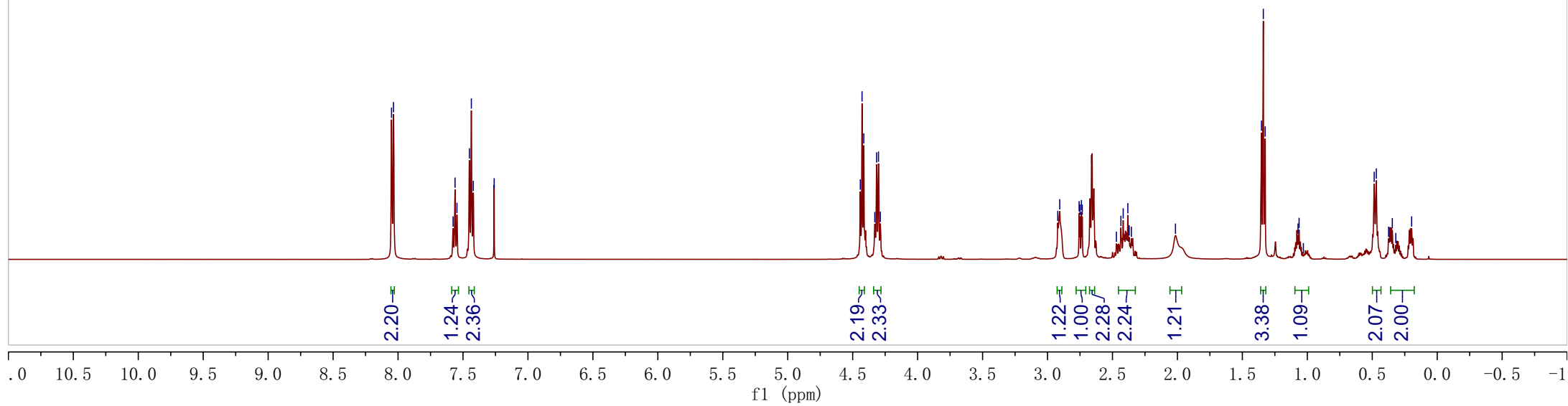


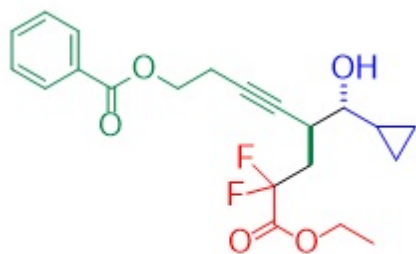
8.05
 8.04
 7.58
 7.56
 7.55
 7.45
 7.44
 7.42
 7.26 CDCl₃

4.44
 4.43
 4.41
 4.33
 4.32
 4.30
 4.29

2.92
 2.91
 2.76
 2.75
 2.74
 2.73
 2.43
 2.42
 2.38

1.35
 1.34
 1.32
 1.07
 1.06
 1.03
 0.48
 0.47
 0.37
 0.35
 0.32
 0.20





4j

126 MHz, CDCl₃

166.44
164.15
163.89
163.63

133.11
129.83
129.61
128.35
117.56
115.57
115.56
113.57

80.28
79.51
78.01
77.25
77.00 CDCl₃
76.75
62.84
62.73

37.12
36.93
36.75
32.85
32.82
32.78
19.56
15.80
13.89
3.07
2.89

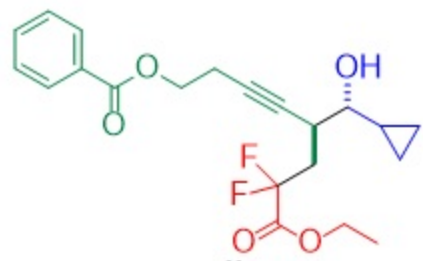
A (t)
163.89
H

B (dd)
115.56
H

C (t)
36.93
H

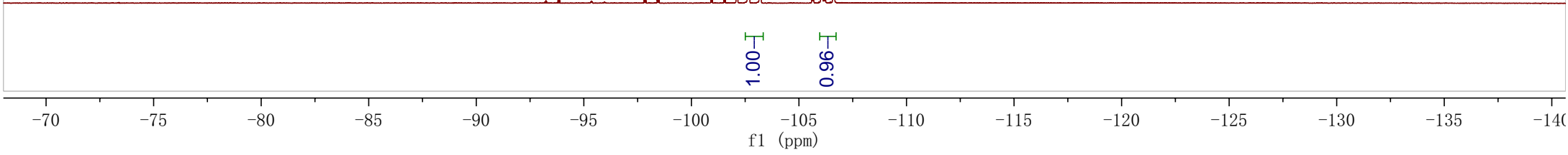
D (m)
32.80
H

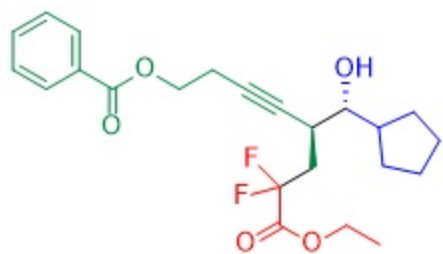
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



4j
471 MHz, CDCl₃

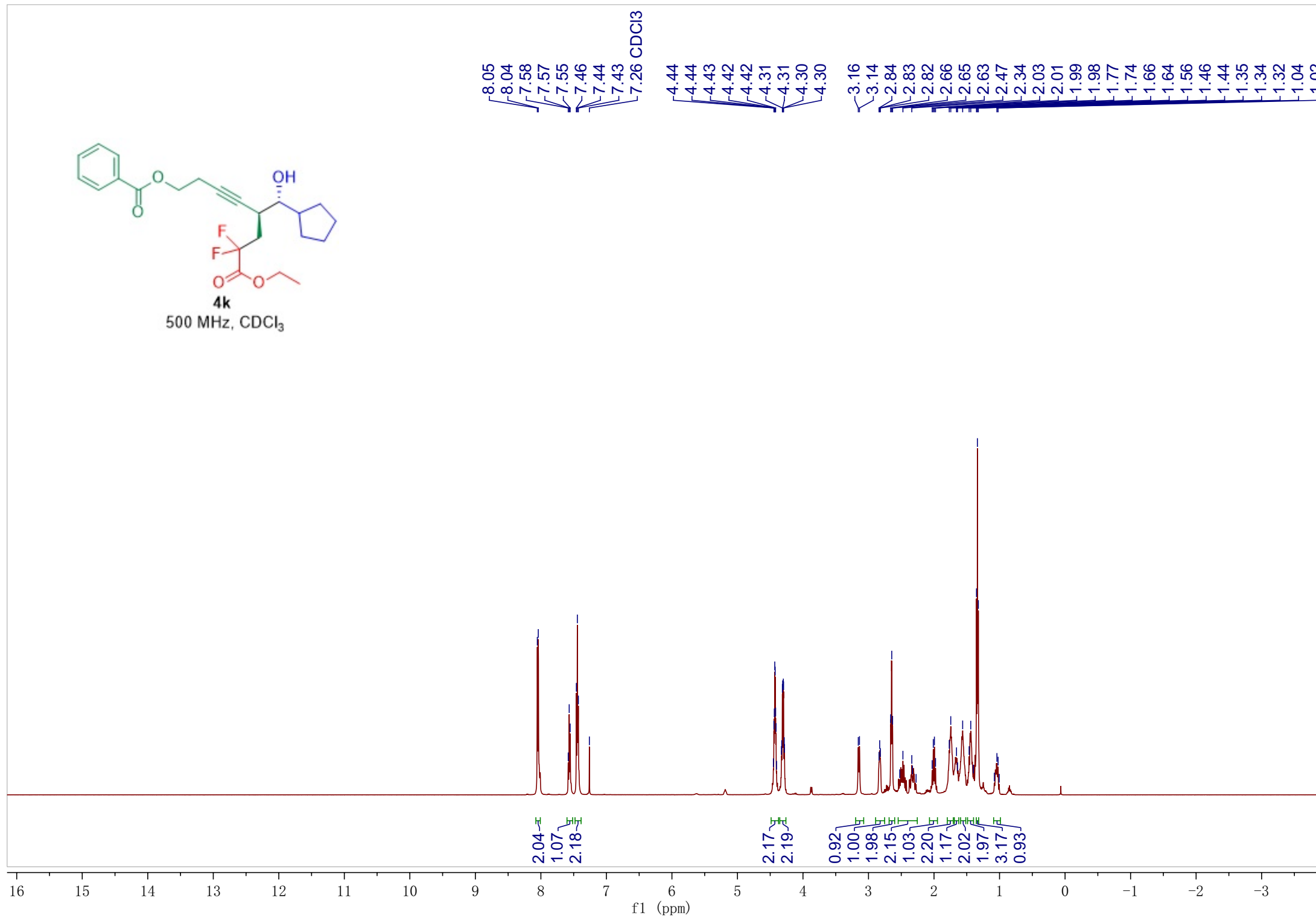
102.61
102.62
102.67
103.16
103.19
103.21
103.24
106.02
106.06
106.10
106.58
106.62
106.65

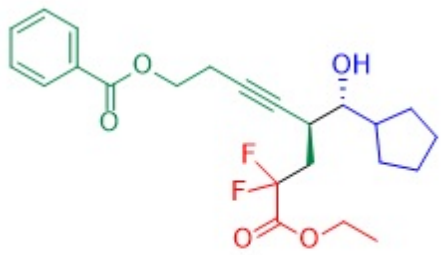




4k

500 MHz, CDCl₃





4k
126 MHz, CDCl₃

166.47
164.19
163.93
163.67

133.14
129.82
129.60
128.39
117.62
115.64
115.62
113.64

80.54
78.85
78.17
77.25
77.00 CDCl₃
76.75

62.83
62.75

45.00
37.86
37.67
37.49
31.37
31.35
31.34
31.32
29.79
29.09
25.41
25.28
19.60
13.90

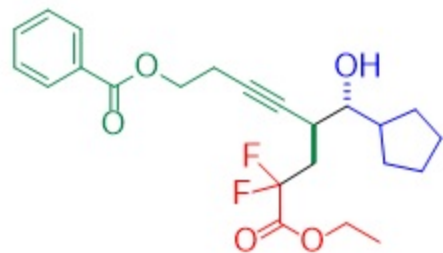
A (t)
163.93
H

B (dd)
115.63
H

C (t)
37.67
H

D (m)
31.34
H

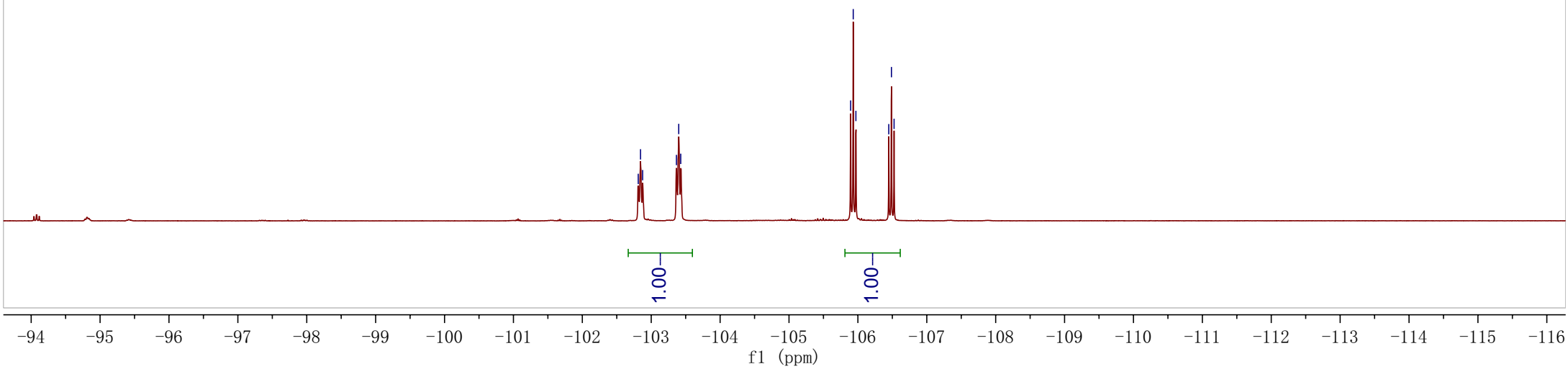
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

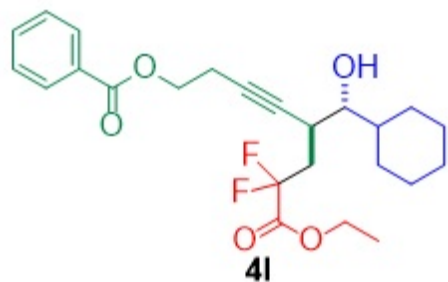


4k
471 MHz, CDCl₃

102.81
102.84
102.88
103.37
103.40
103.43

105.89
105.93
105.97
106.45
106.49
106.53



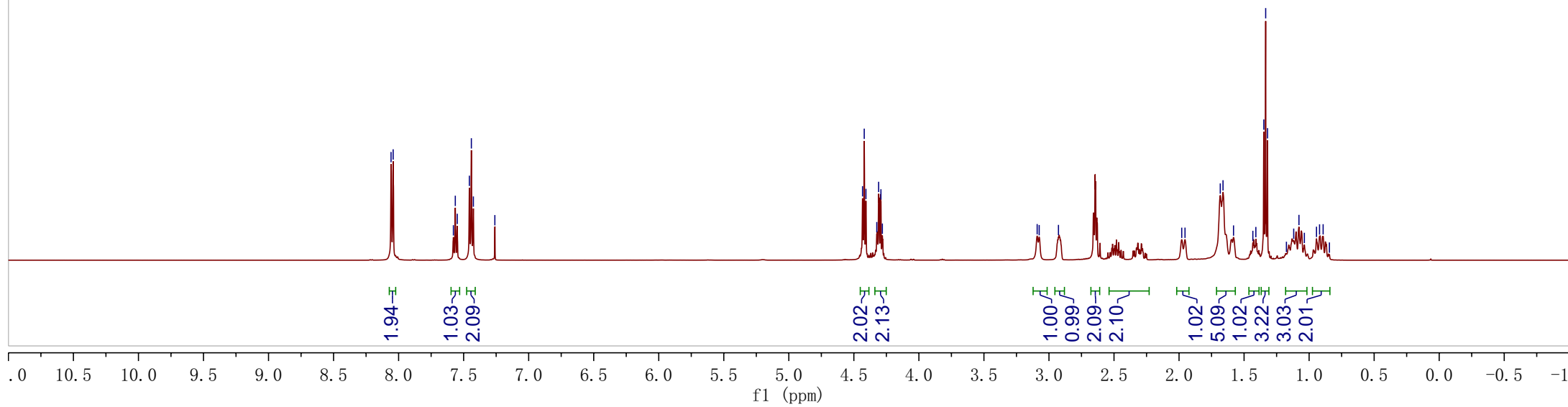


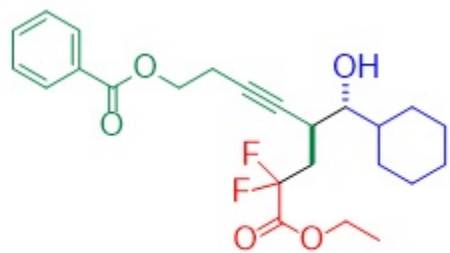
500 MHz, CDCl₃

8.06
8.04
7.58
7.56
7.55
7.46
7.44
7.43
7.26 CDCl₃

4.43
4.42
4.41
4.32
4.31
4.29
4.28

3.09
3.08
2.93
1.98
1.95
1.68
1.66
1.58
1.43
1.41
1.35
1.33
1.32
1.17
1.12
1.08
1.04
0.94
0.92
0.89
0.84





4I

126 MHz, CDCl₃

175.85
166.47
164.18
163.92
163.66

133.14
129.80
129.61
128.39
117.65
115.66
115.65
113.66

80.59
78.76
77.81
77.25
77.00 CDCl₃
76.75
62.82
62.79

42.05
37.91
37.73
37.55
29.49
29.47
29.45
29.43
29.21
28.99
26.14
25.79
25.58
19.58
13.89

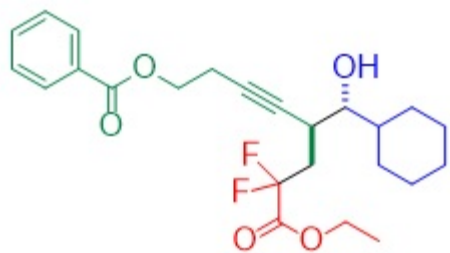
E (t)
163.92
H

B (dd)
115.66
H

C (t)
37.73
H

D (m)
29.46
H

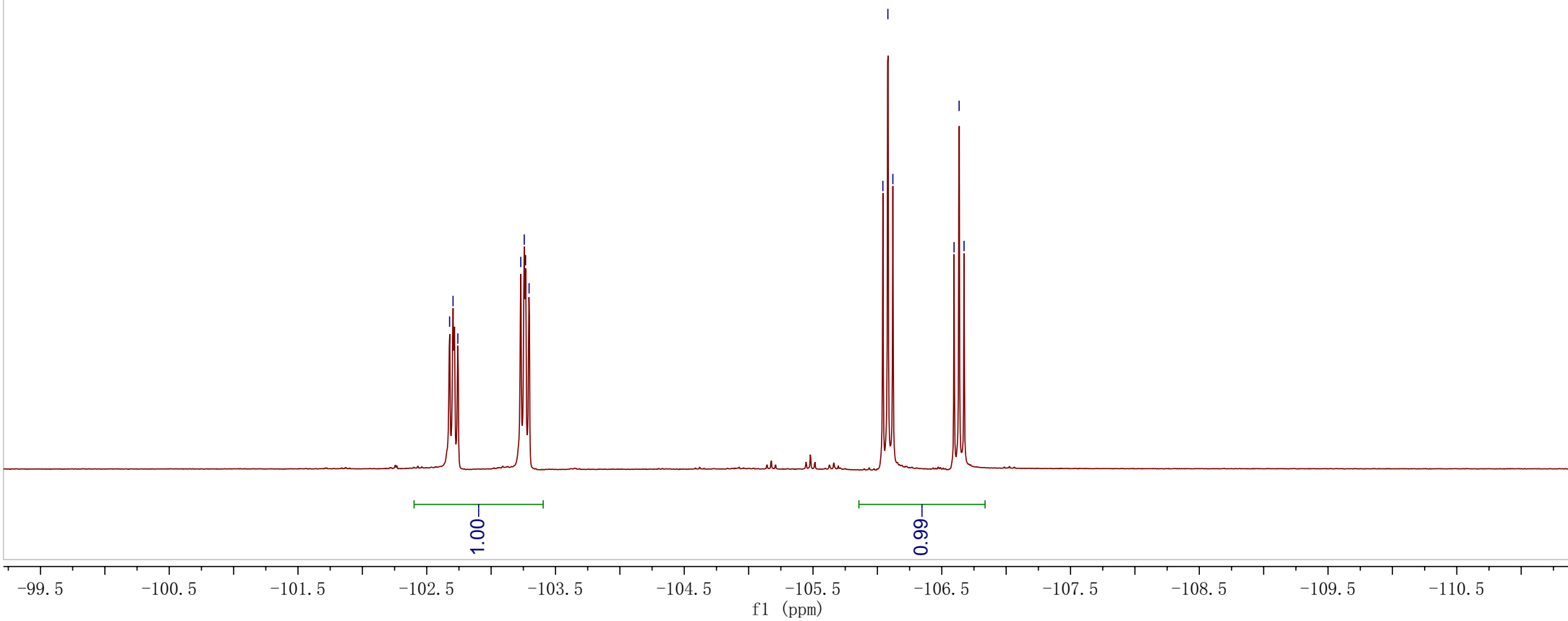
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

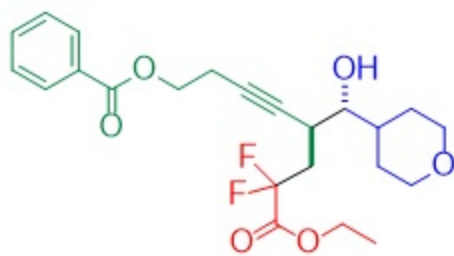


4I
471 MHz, CDCl₃

102.68
102.70
102.74
103.23
103.26
103.27
103.29

106.04
106.08
106.12
106.60
106.63
106.67





4m

500 MHz, CDCl₃

8.05
8.03
7.58
7.57
7.55
7.46
7.44
7.43
7.26 CDCl₃

4.49
4.45
4.42
4.39
4.32
4.30
4.29
4.27
3.90
3.87

3.21
3.21
3.19
3.12
3.09
2.87
2.66
2.64
2.63
1.88
1.82
1.80
1.64
1.63
1.49
1.46
1.34
1.33
1.31
1.27
1.24
1.20
1.19

2.00

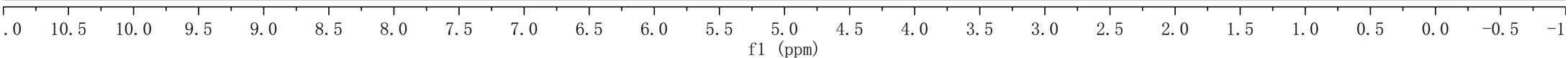
1.08
2.12

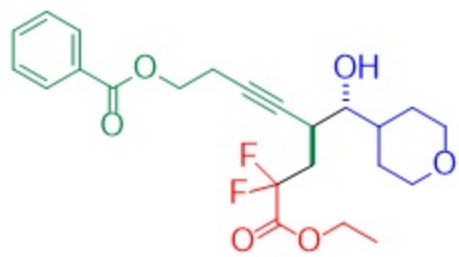
2.11
2.12

2.03

1.13
2.01
1.00
2.08
2.16

2.29
1.03
1.07
3.04
2.14





4m
126 MHz, CDCl₃

166.52
164.07
163.82
163.56

133.25
129.71
129.58
128.43
117.51
115.52
115.51
113.52

81.00
78.26
77.32
77.25
77.00 CDCl₃
76.75
67.59
67.15
62.87
62.66

39.47
37.73
37.54
37.36

29.50
29.08
29.04
13.87

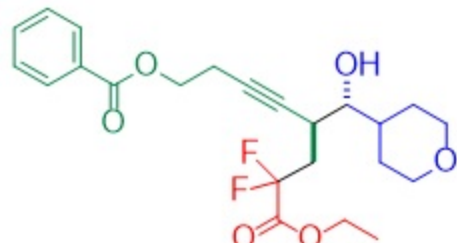
A (t)
163.82
H

B (dd)
115.51
H

C (t)
37.54
H

D (m)
29.08
H

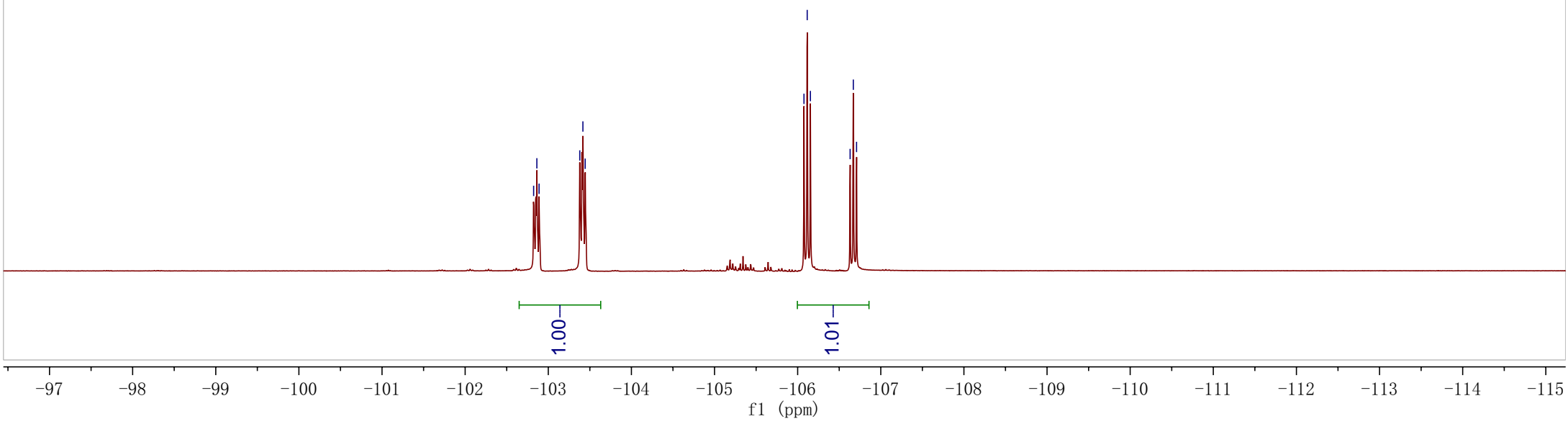
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

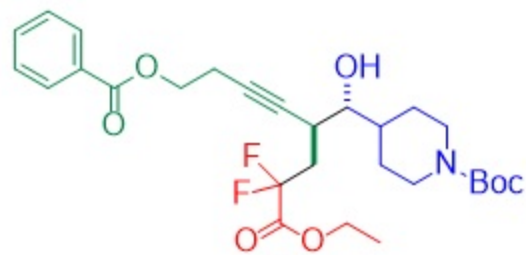


4m
471 MHz, CDCl₃

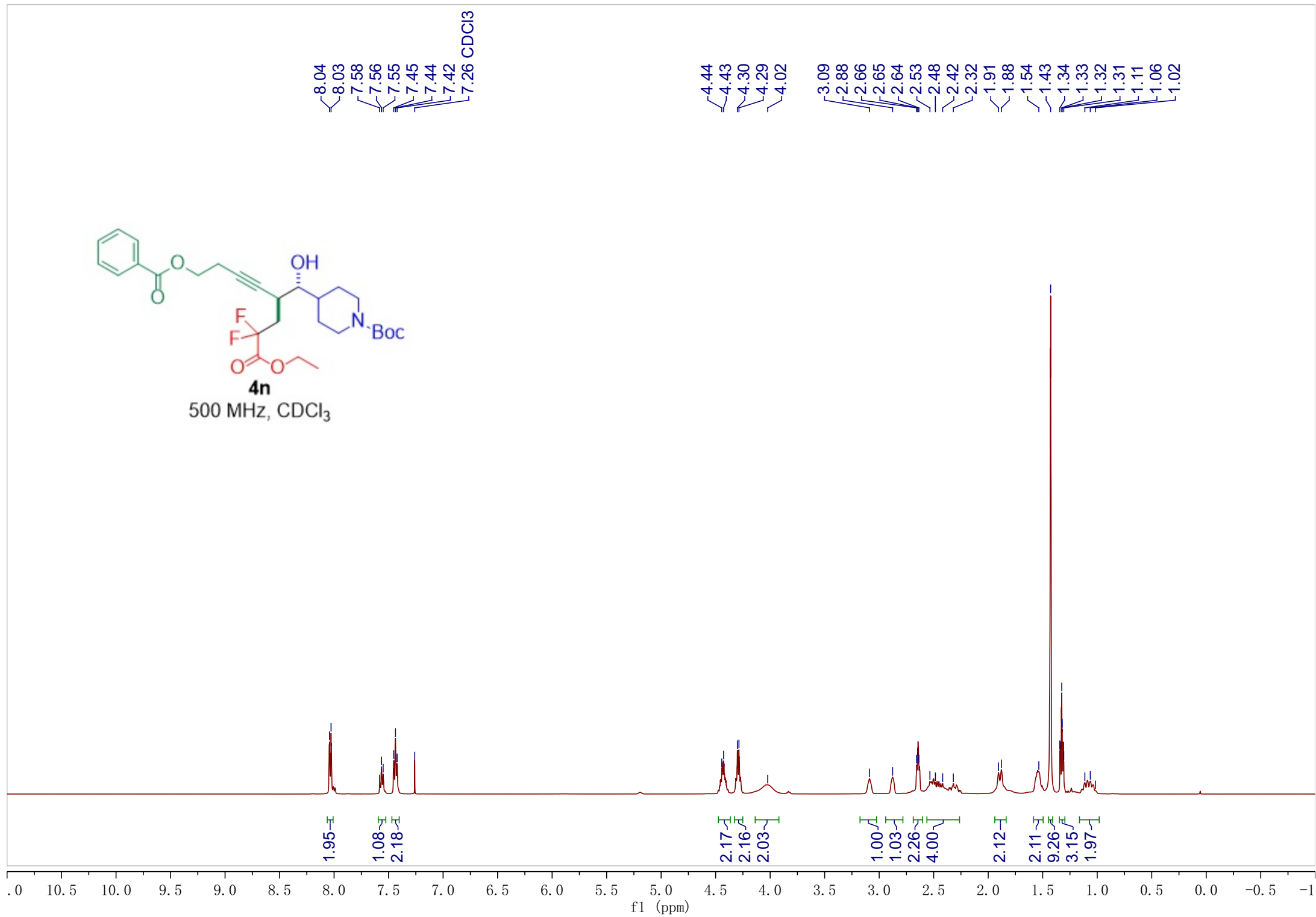
102.82
102.86
102.89
103.38
103.42
103.44

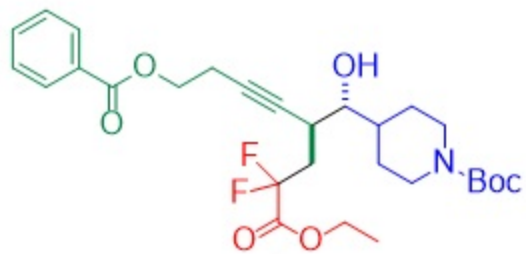
106.08
106.11
106.15
106.63
106.67
106.71



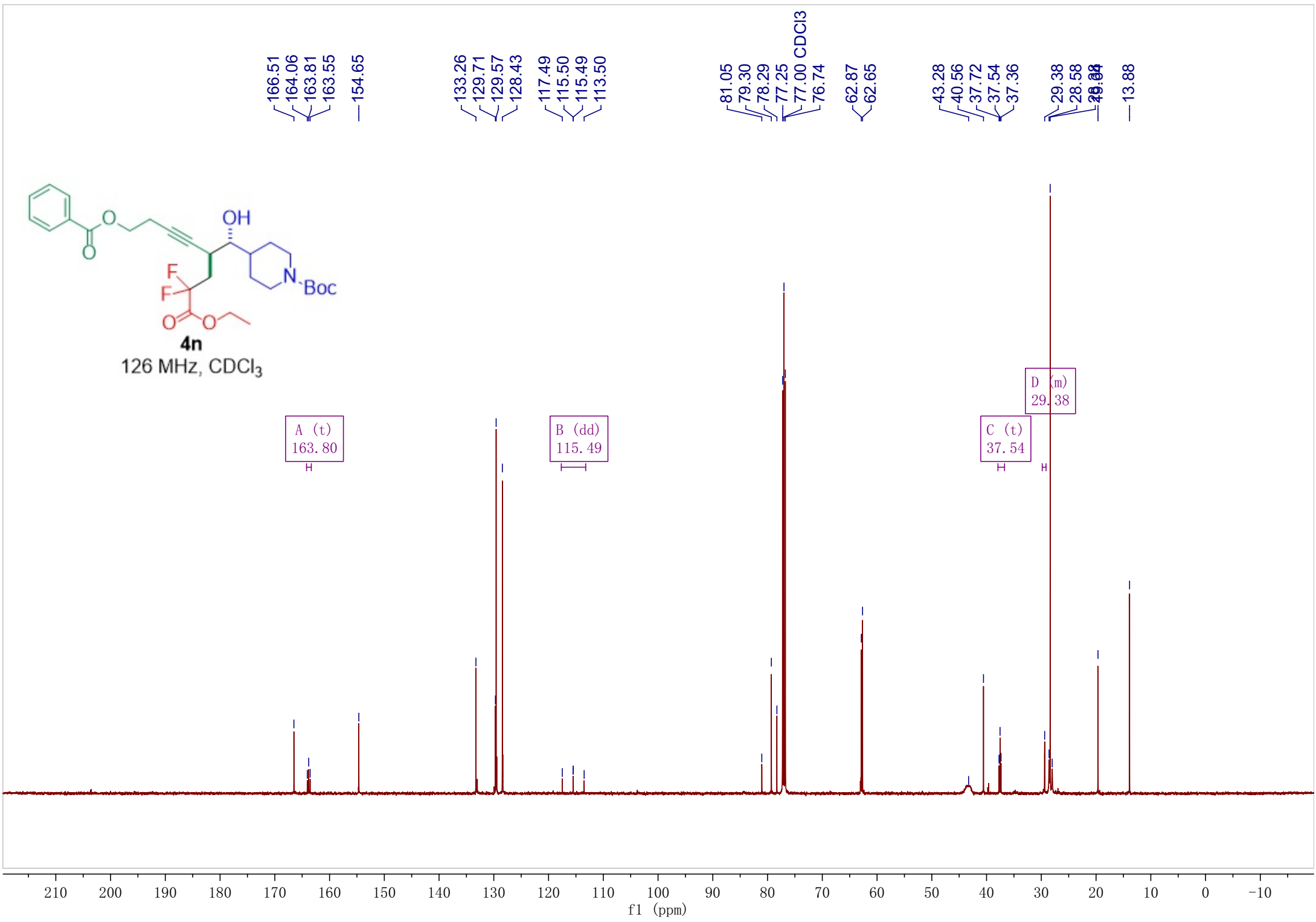


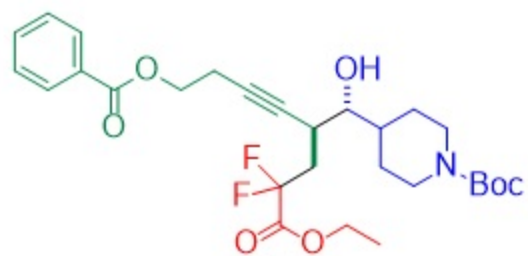
4n
500 MHz, CDCl₃





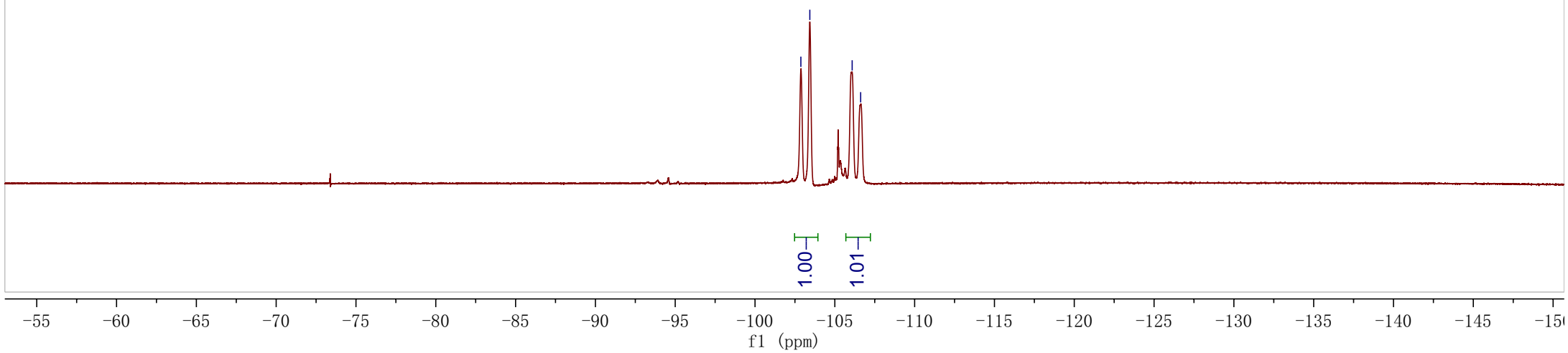
4n
126 MHz, CDCl₃

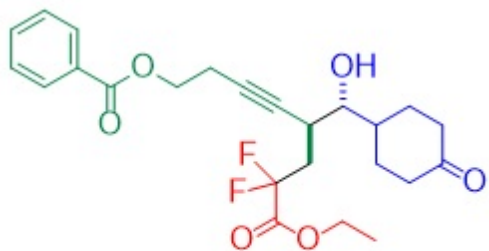




4n
471 MHz, CDCl₃

102.87
103.43
106.09
106.62





4o

126 MHz, CDCl₃

—211.46

166.60
164.05
163.80
163.53

133.38
129.71
129.58
128.50
117.45
115.46
115.45
113.46

81.41
78.22
77.25
77.00 CDCl₃
76.75
76.34

62.95
62.64

40.35
39.97
39.95
37.75
37.56
37.38
29.97
28.94
28.72

—19.74
—13.90

A (t)
163.79
H

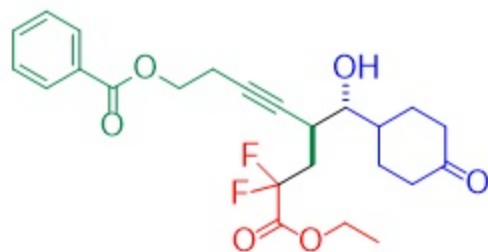
B (dd)
115.45
H

C (t)
37.56
H

D (m)
29.98
H

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

f1 (ppm)



4o

471 MHz, CDCl₃

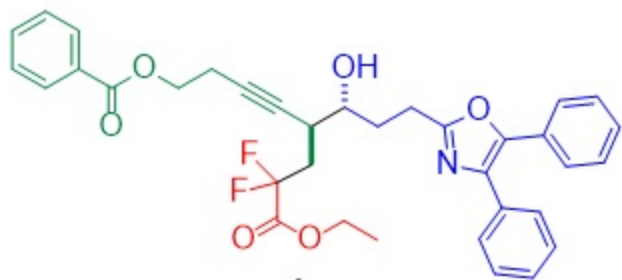
102.99
103.01
103.05
103.53
103.57
103.61
106.00
106.04
106.08
106.56
106.60
106.64

1.00

1.04

-65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 -145 -150

f1 (ppm)

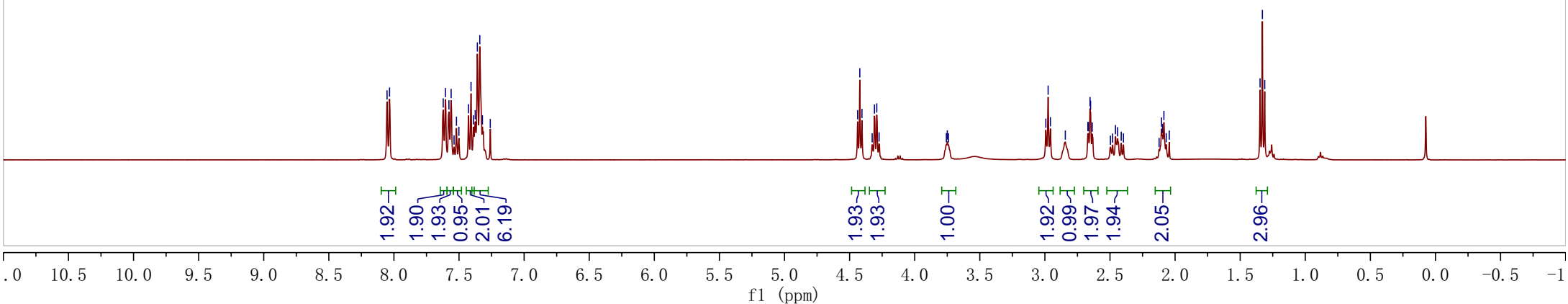


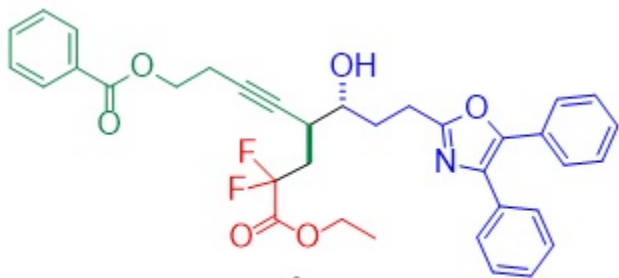
4p

500 MHz, CDCl₃

8.05
8.03
7.62
7.60
7.58
7.56
7.54
7.52
7.50
7.43
7.41
7.39
7.38
7.36
7.34
7.32
7.26 CDCl₃

4.44
4.42
4.40
4.33
4.31
4.29
4.27
3.76
3.75
3.74
2.99
2.98
2.96
2.67
2.67
2.65
2.65
2.64
2.10
2.10
1.99
1.33
1.31





4p
126 MHz, CDCl₃

166.41
164.15
163.89
163.63
163.25

— 145.29

129.59
128.62
128.53
128.34
127.81
126.43
117.57
115.58
115.57
113.58

80.56
79.12
77.25
77.00 CDCl₃
76.75
72.71
62.84
62.72

36.71
36.53
36.35
32.69
31.68
25.26
19.55
13.88

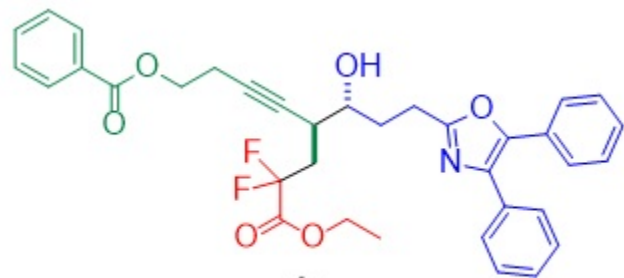
A (t)
163.89
H

B (dd)
115.58
H

D (m)
32.69
C (t)
36.53
H H

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

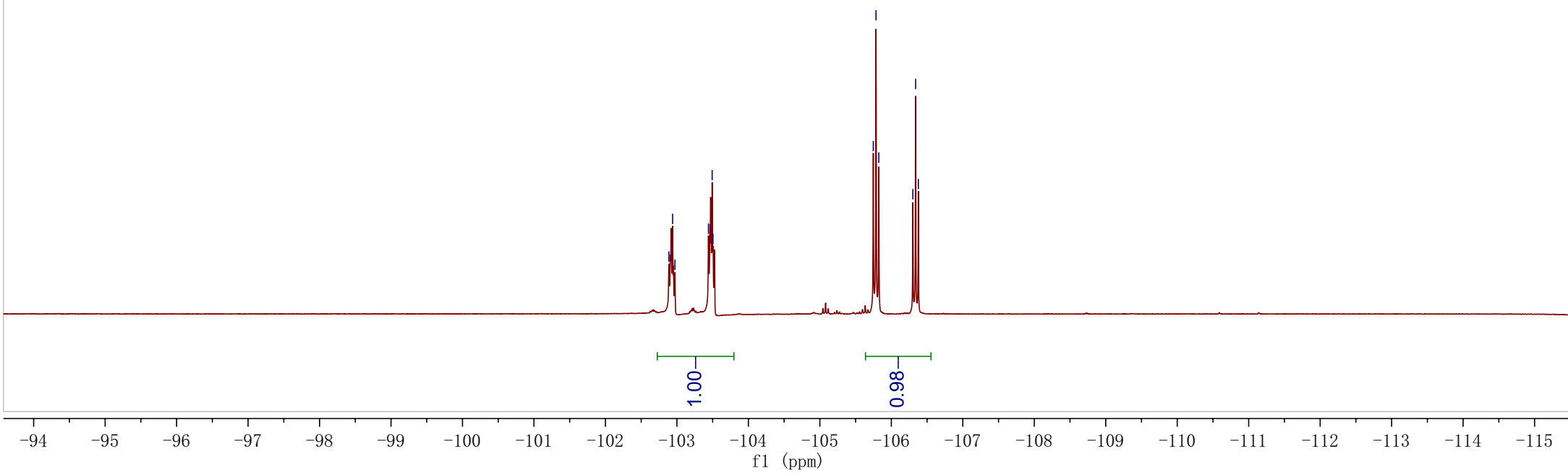
f1 (ppm)

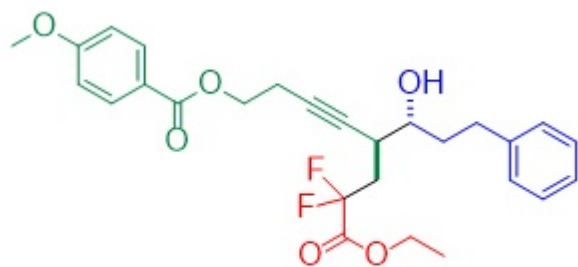


4p
471 MHz, CDCl₃

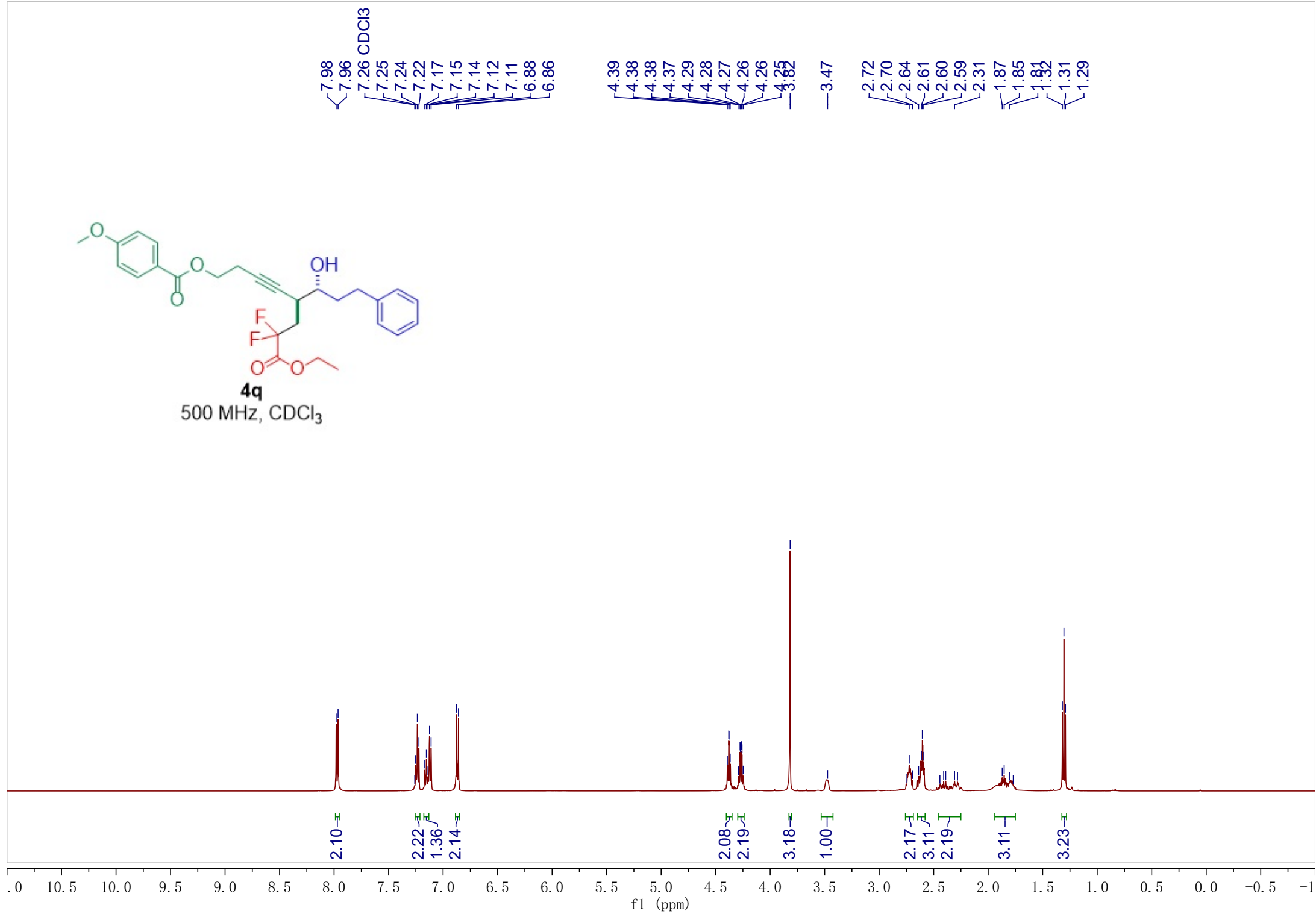
102.89
102.94
102.97
103.44
103.49
103.50

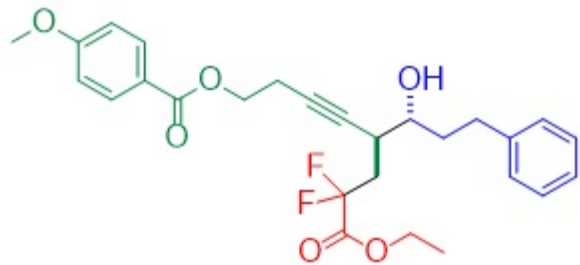
105.75
105.79
105.82
106.30
106.34
106.38





4q
500 MHz, CDCl₃





4q
126 MHz, CDCl₃

166.25
164.10
163.84
163.58
163.49

141.45
131.67
128.36
128.33
125.86
122.12
117.52
115.52
115.48
113.63
113.53

81.03
78.71
77.25
77.00 CDCl₃
76.75
72.44
62.84
62.39
55.37

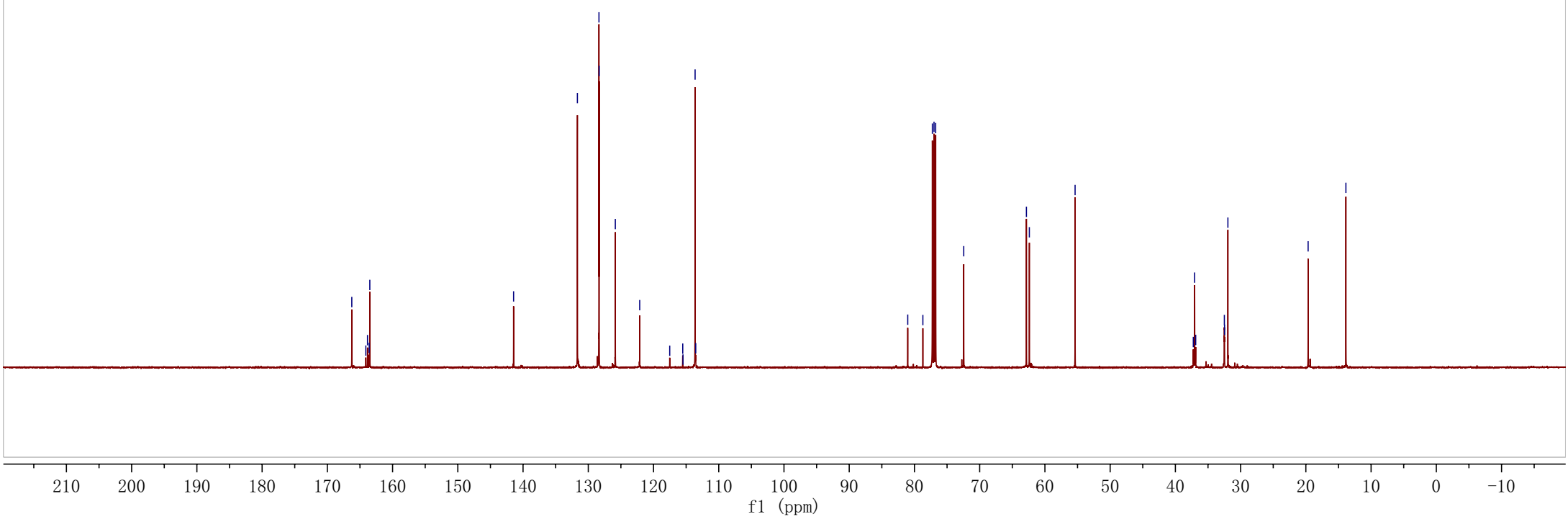
37.25
37.06
36.89
32.49
32.46
31.95
19.65
13.86

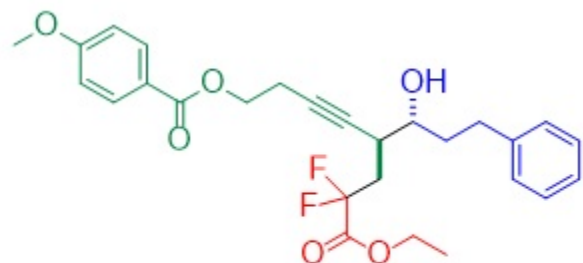
A (t)
163.84
H

B (dd)
115.51
H

D (m)
37.06
H

C (m)
32.48
H





4q
471 MHz, CDCl₃

102.73

103.29

105.98

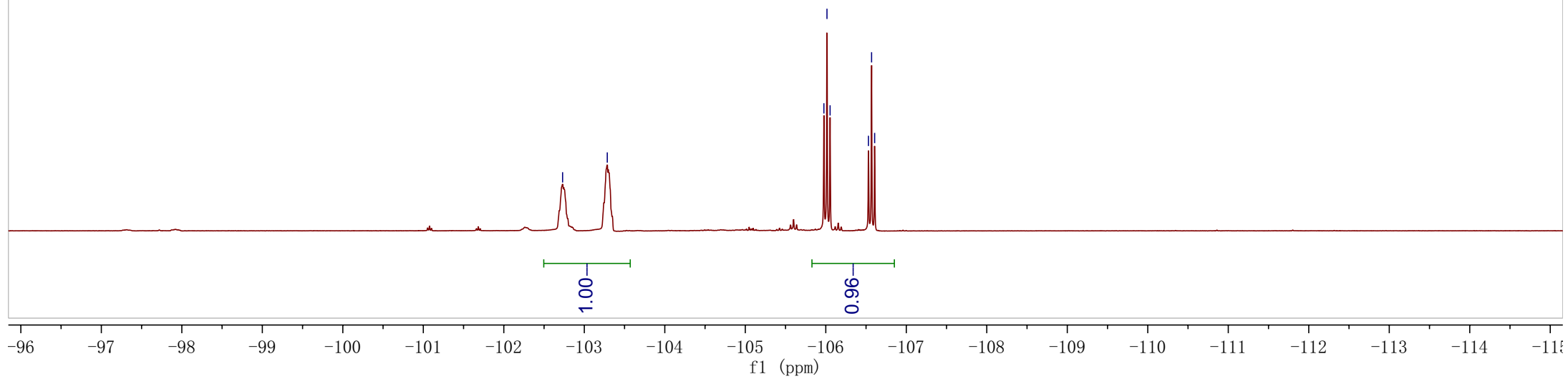
106.01

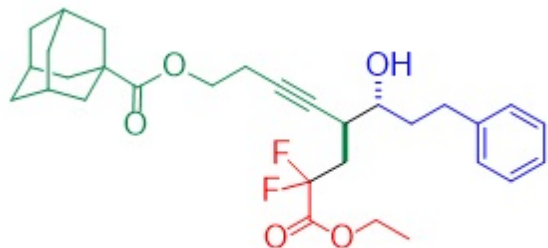
106.05

106.53

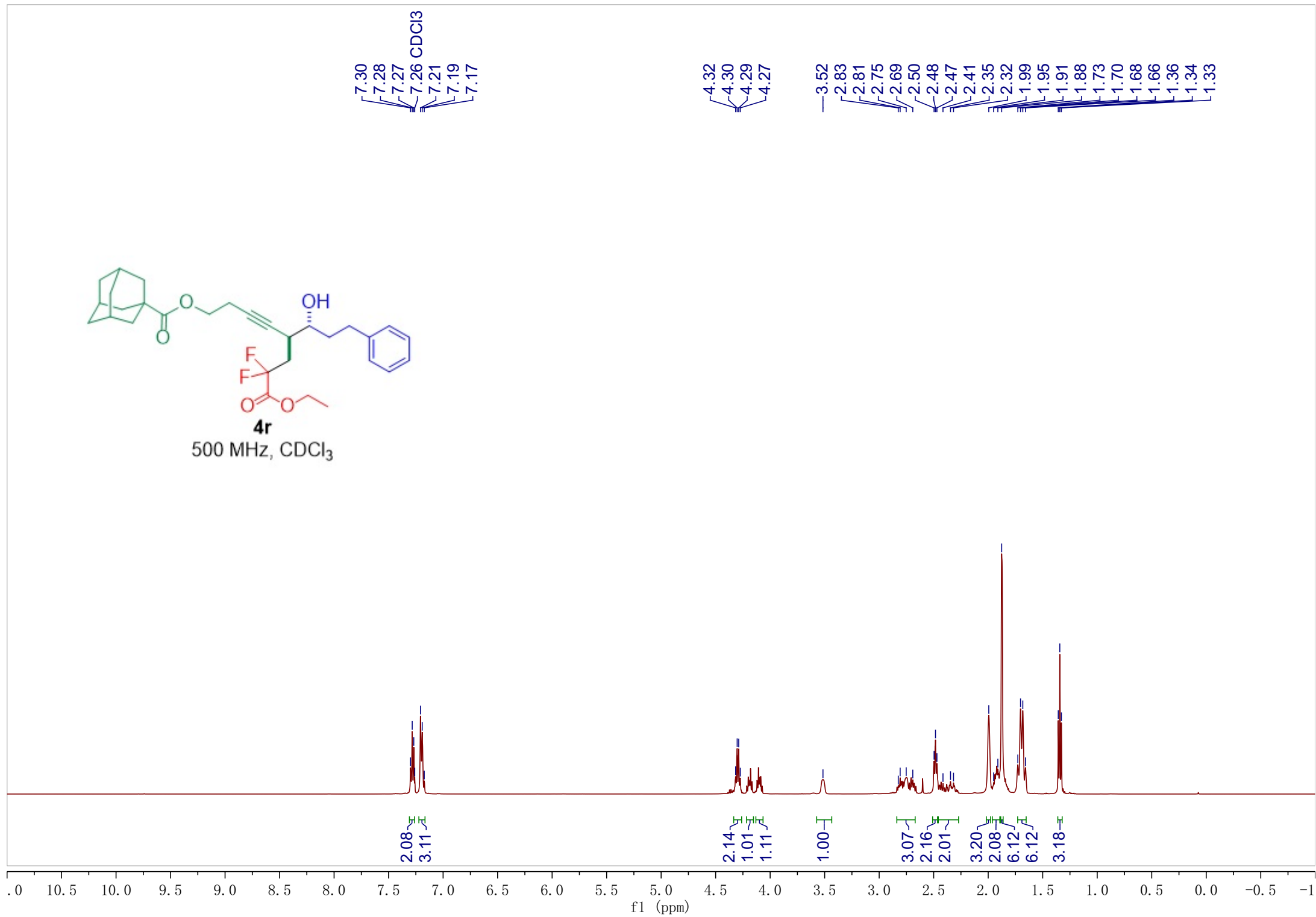
106.57

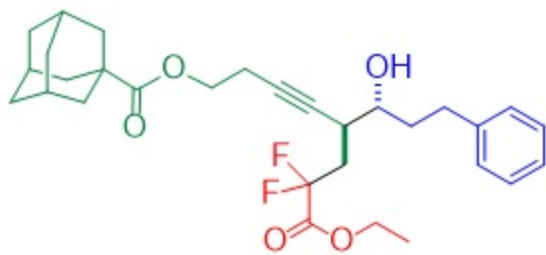
106.61





4r
500 MHz, CDCl₃





4r
126 MHz, CDCl₃

—177.60

164.08
163.82
163.56

—141.44

128.38
128.35
125.89
117.48
115.49
115.48
113.49

81.10
78.39
77.25
77.00 CDCl₃
76.74
72.36
62.83
61.90

40.63
38.69
37.23
37.10
37.05
36.86
36.36
32.48
31.98
27.80
19.40
—13.86

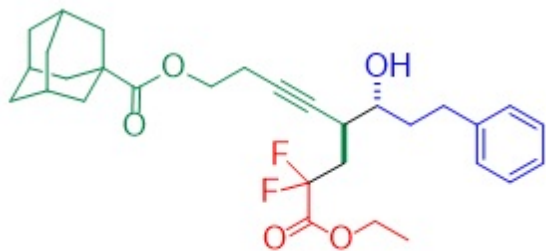
A (t)
163.82
H

B (dd)
115.49
H

D (t)
37.05
H

C (m)
32.48
H

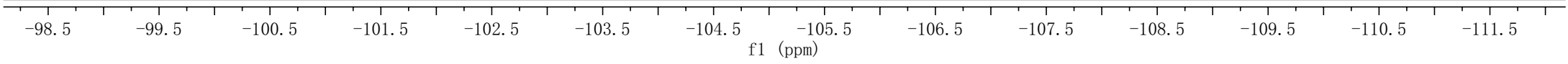
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

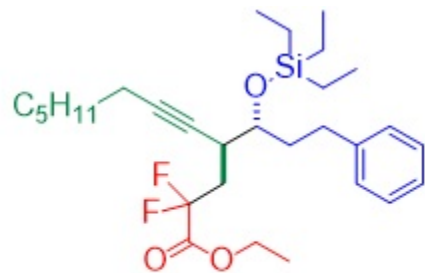


4r
471 MHz, CDCl₃

102.86
102.89
102.92
103.41
103.44
103.45
103.48

105.87
105.91
105.95
106.43
106.46
106.50





4s

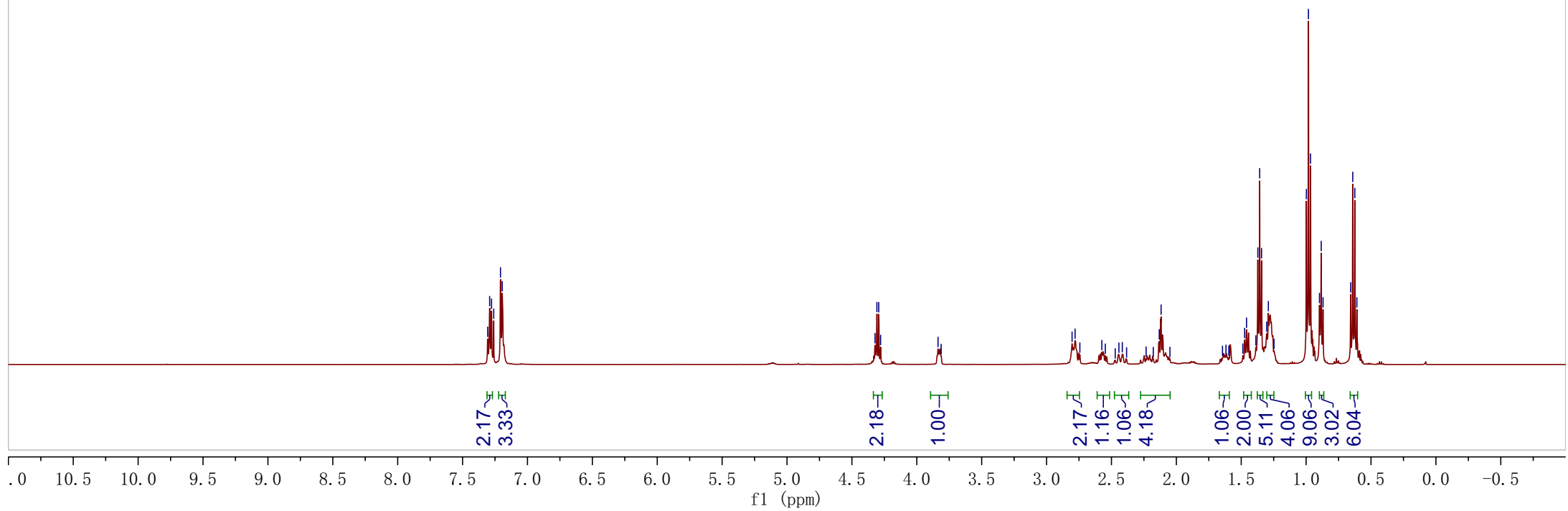
500 MHz, CDCl₃

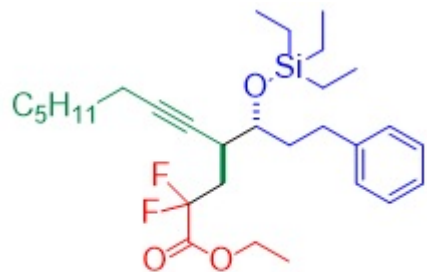
7.31
7.29
7.28
7.26
7.21
7.19

4.32
4.31
4.29
4.28
3.84
3.81

2.80
2.78
2.74
2.57
2.55
2.44
2.42
2.23
2.13
2.12

1.46
1.37
1.36
1.34
1.30
1.29
1.00
0.98
0.97
0.90
0.88
0.87
0.66
0.64
0.62
0.61





4s

126 MHz, CDCl₃

A (t)
164.08
H

D (dd)
116.07
H

B (t)
34.15
H

C (m)
32.09

164.34
164.08
163.82

142.14

128.35
128.28
125.80
118.06
116.09
116.06
114.08

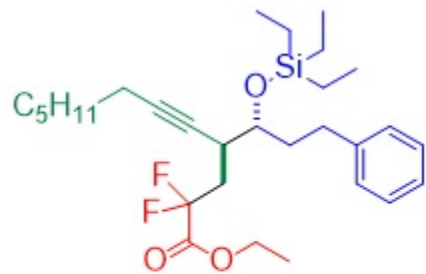
84.13
78.16
77.25
77.00 CDCl₃
76.75
73.67

62.64

34.82
34.34
34.15
33.97
32.81
32.11
31.32
28.75
28.50
22.53
18.76
14.04
13.87
6.86
5.00

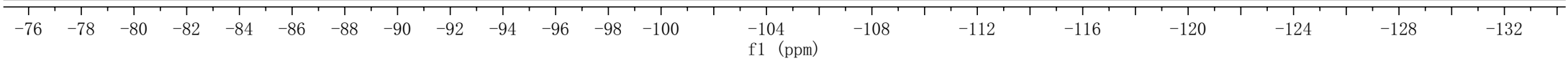
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

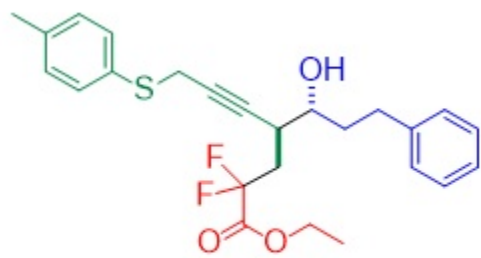
f1 (ppm)



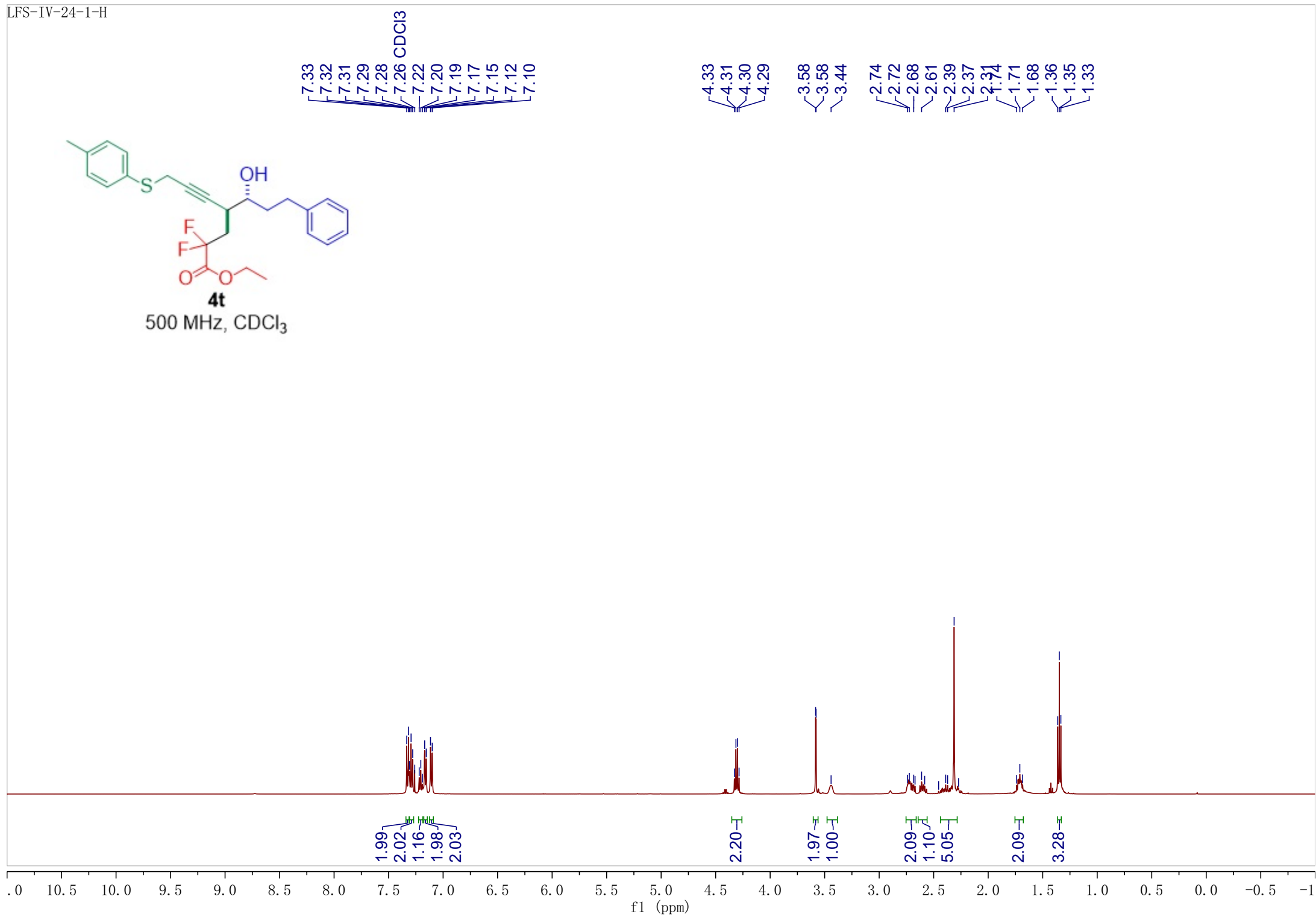
4s
471 MHz, CDCl₃

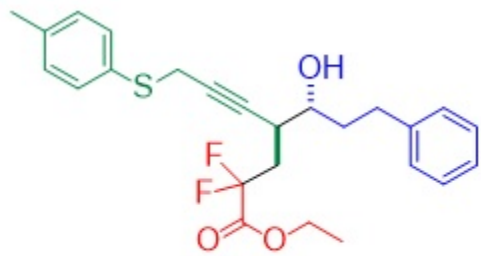
101.14
101.16
101.19
101.68
101.71
101.74
107.47
107.51
107.55
108.02
108.06
108.10





4t
500 MHz, CDCl₃





4t
126 MHz, CDCl₃

164.06
163.80
163.54

141.37
137.39
131.09
130.93
129.78
128.40
128.35
125.93
117.42
115.43
115.42
113.43

80.84
80.31
77.25
77.00 CDCl₃
76.75
72.31
— 62.94

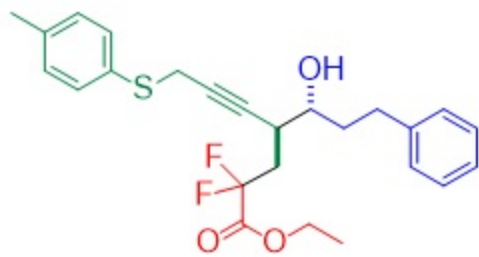
37.08
36.94
36.90
36.71
32.57
31.90
23.36
21.02
— 13.89

A (t)
163.80
H

B (dd)
115.42
H

D (m)
32.56
C (t)
36.90
H H

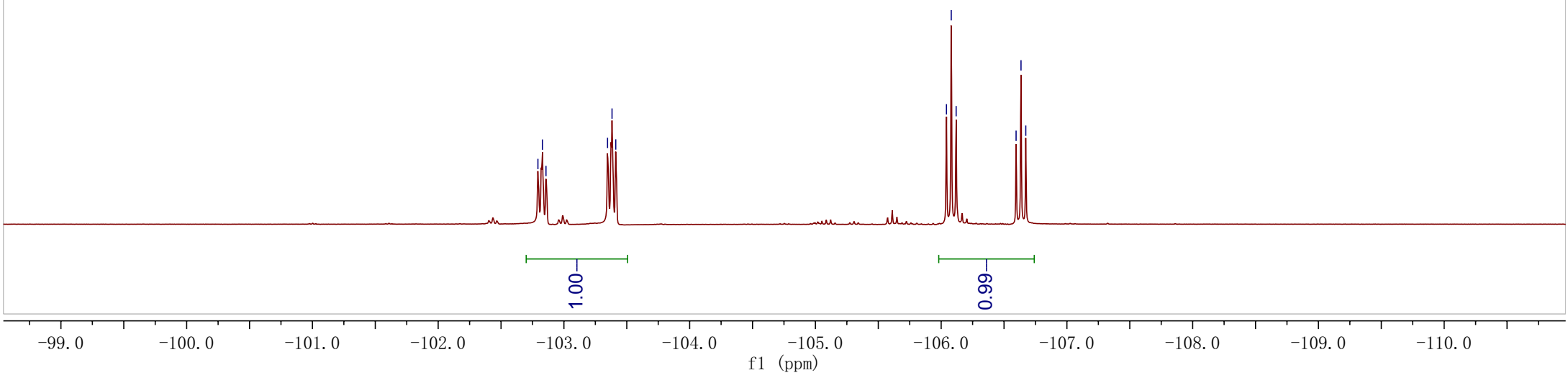
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

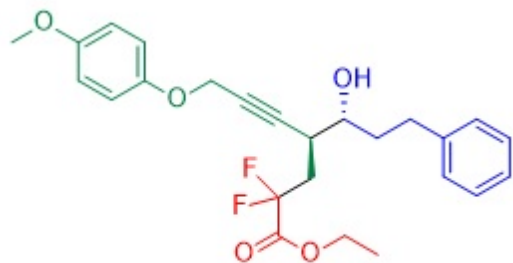


4t
471 MHz, CDCl₃

102.79
102.83
102.86
103.35
103.38
103.41

106.04
106.08
106.12
106.60
106.64
106.67



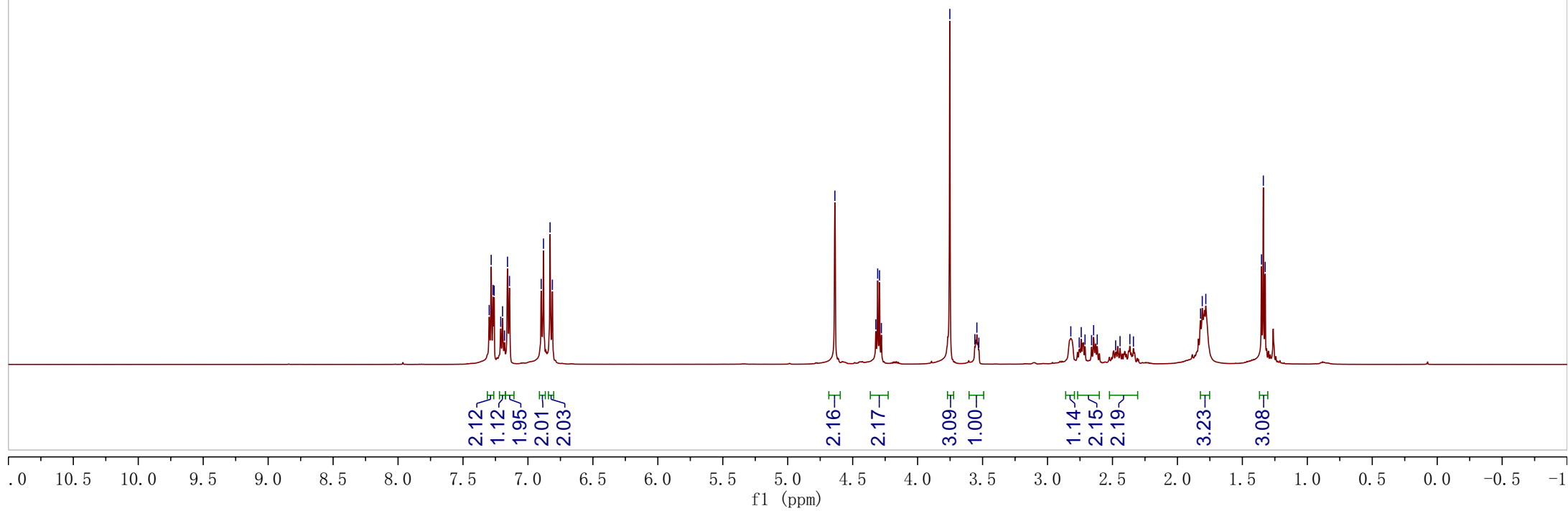


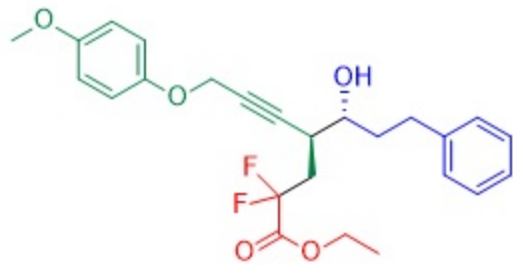
4u
500 MHz, CDCl₃

7.30
7.28
7.27
7.26 CDCl₃
7.21
7.20
7.18
7.16
7.14
6.90
6.88
6.83
6.81

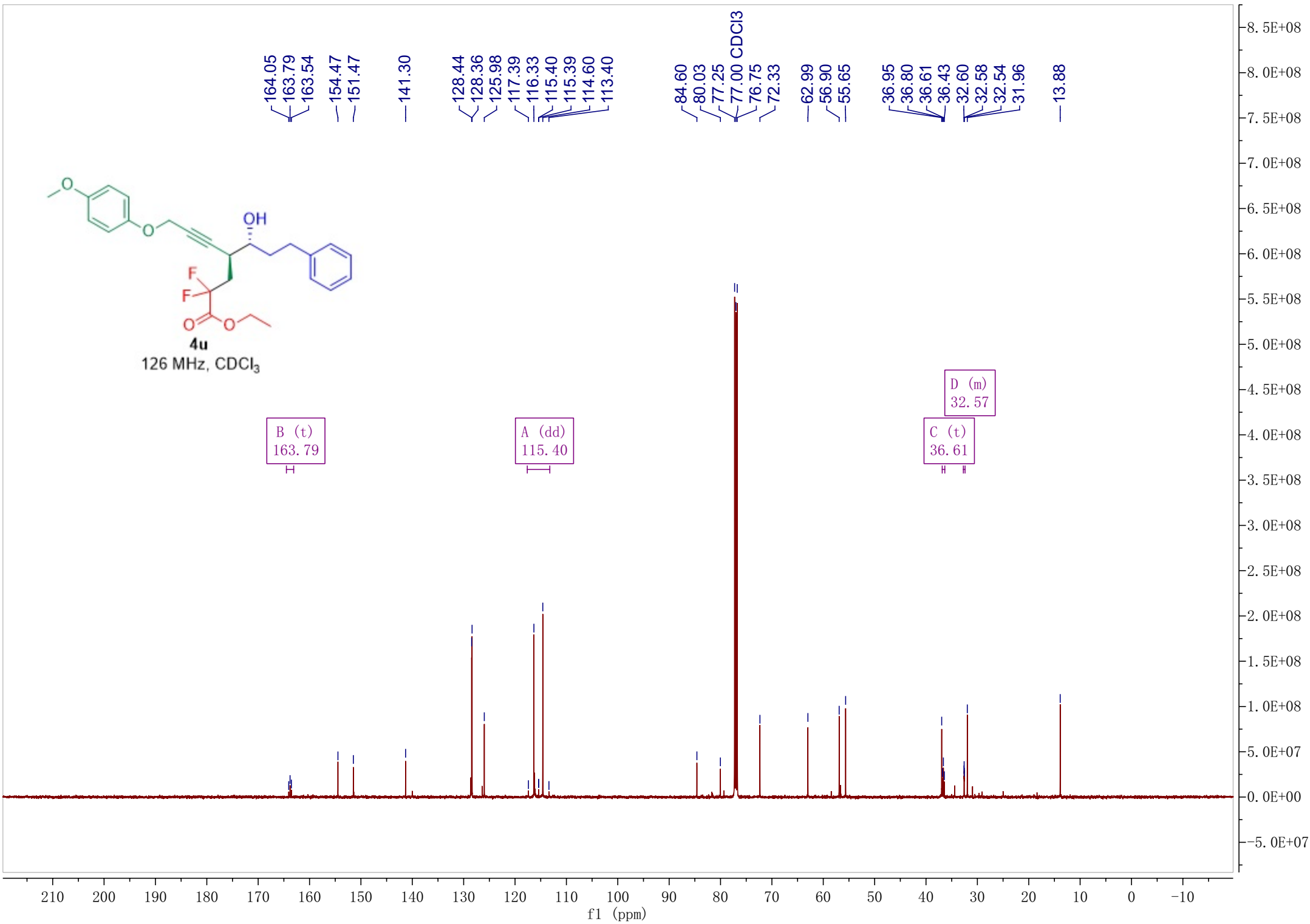
4.64
4.32
4.31
4.29
4.28
3.75
3.56
3.54
3.53

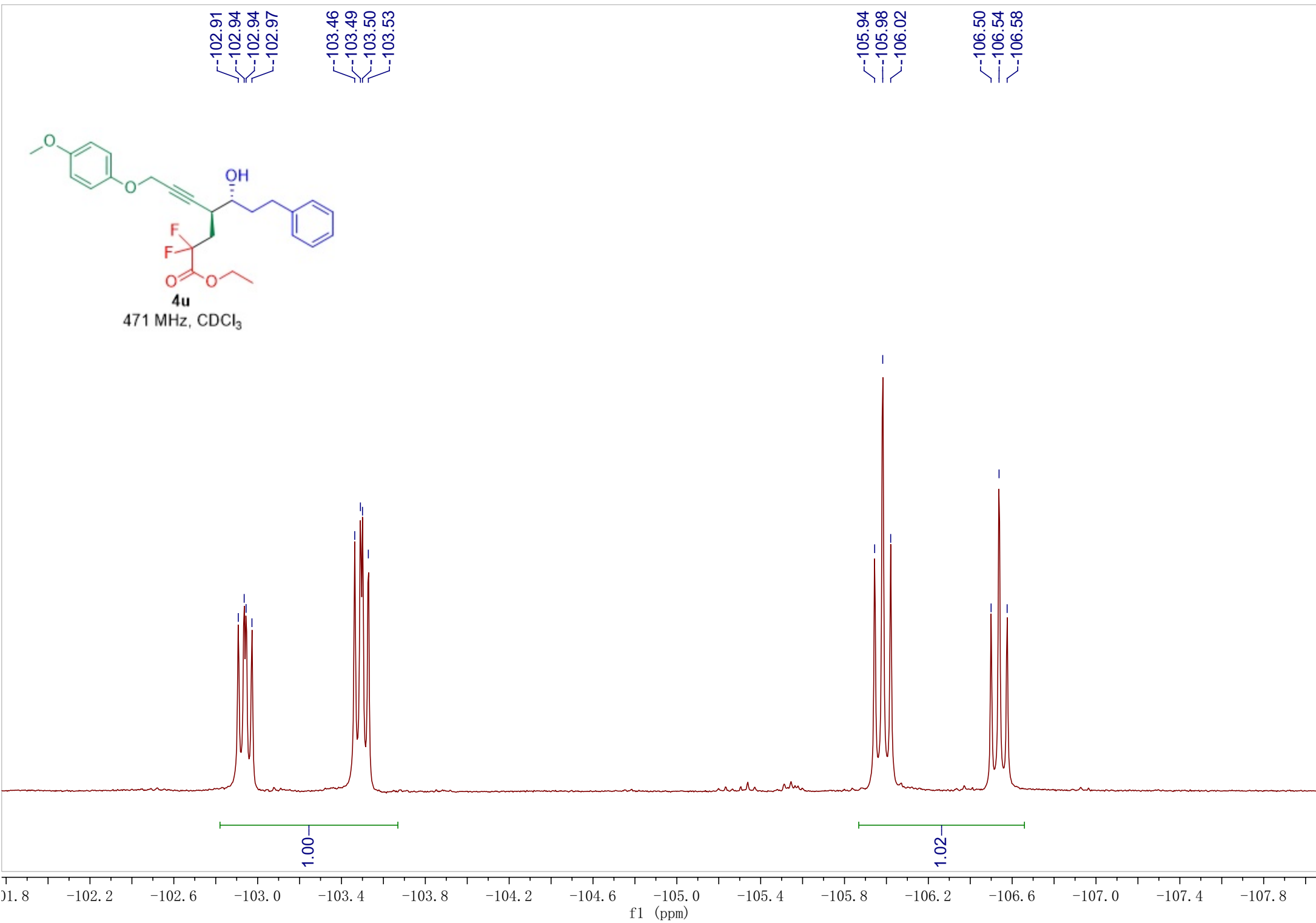
2.82
2.74
2.71
2.66
2.65
2.62
2.37
1.81
1.78
1.35
1.34
1.32

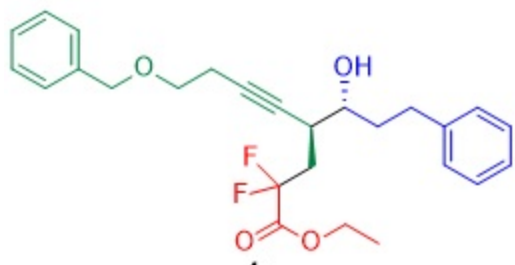




4u
126 MHz, CDCl₃



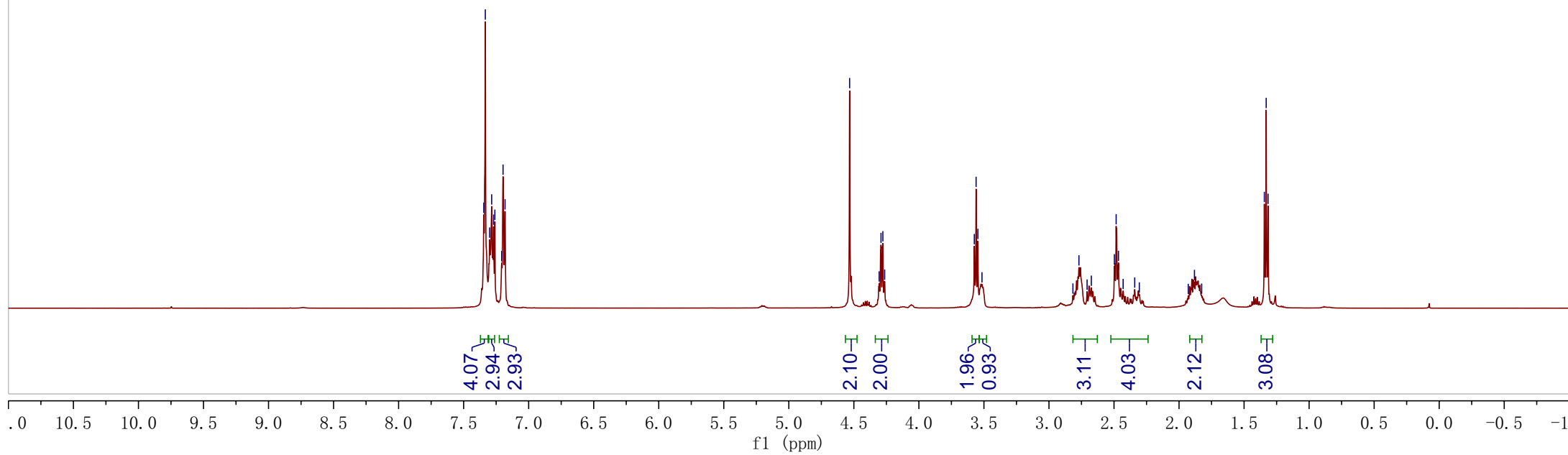


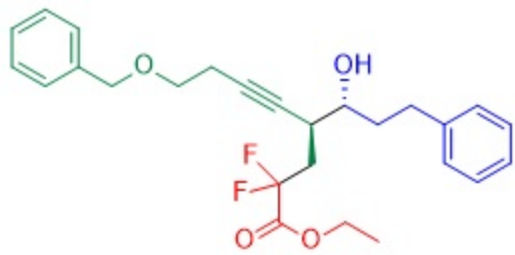


4v
500 MHz, CDCl₃

7.34
7.33
7.30
7.28
7.27
7.26 CDCl₃
7.21
7.20
7.18

4.53
4.31
4.29
4.28
4.26
3.57
3.56
3.55
3.52
2.77
2.67
2.50
2.48
2.47
1.93
1.88
1.83
1.34
1.33
1.32





4v
126 MHz, CDCl₃

164.15
163.89
163.63

141.56
137.89

128.42
128.41
127.74
127.70
115.58
115.56
113.58

82.24
78.10
77.25
77.00 CDCl₃
76.75
72.99
72.44
68.39
62.85

37.32
37.17
37.13
36.95
32.68
32.66
32.62
32.01
20.13
13.88

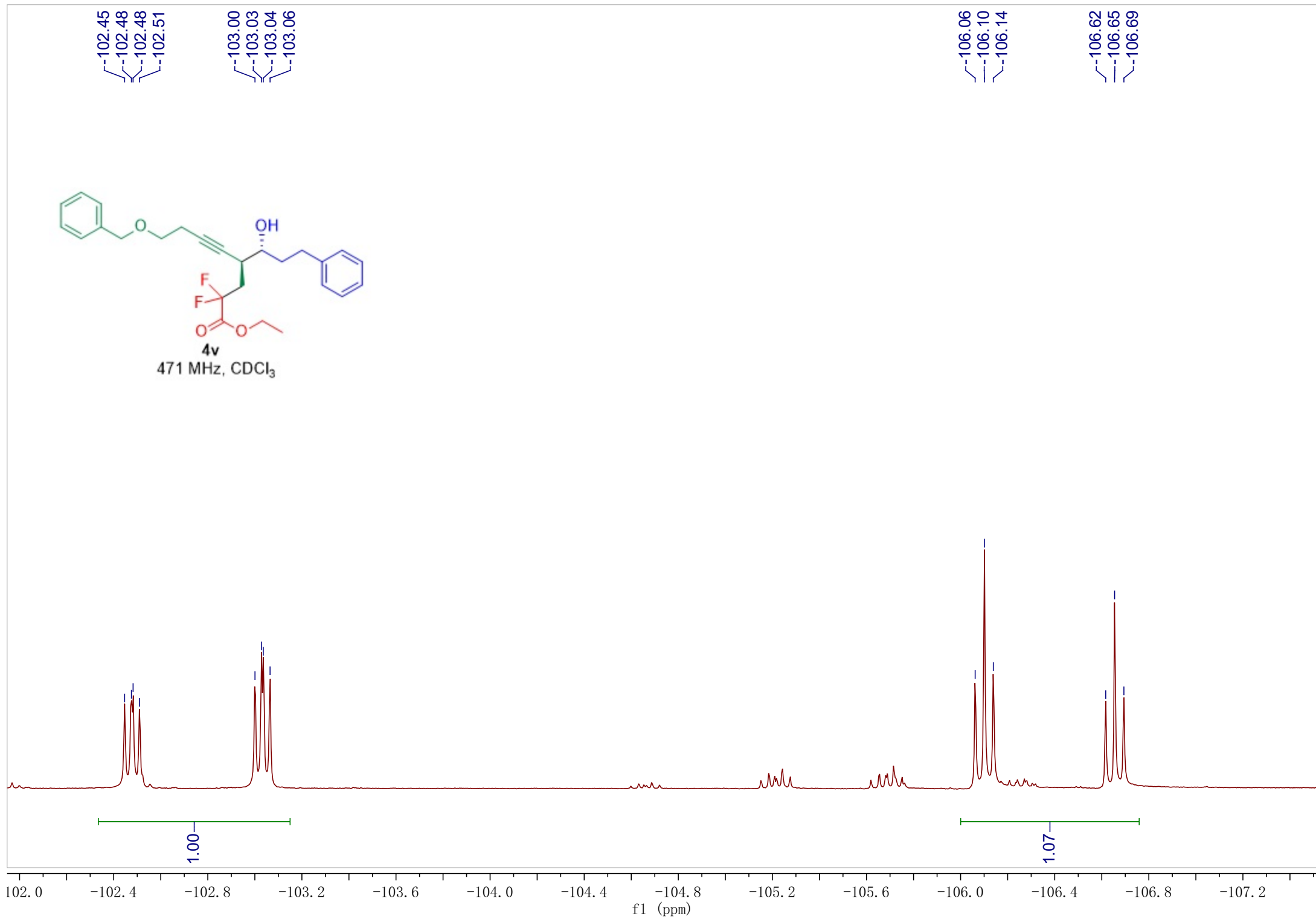
A (t)
163.89
H

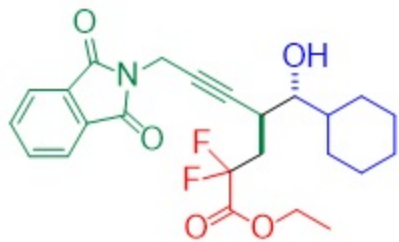
B (dd)
115.57
H

D (t)
37.13
H

C (m)
32.66
H

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



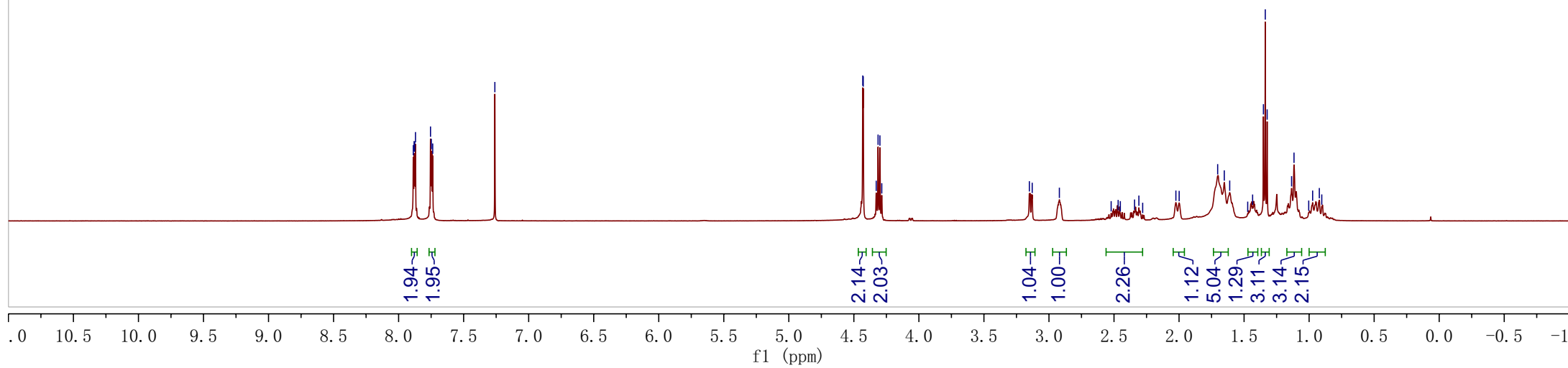


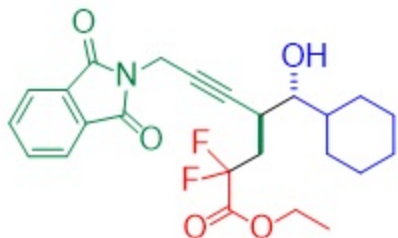
4w
500 MHz, CDCl₃

7.89
7.88
7.88
7.87
7.75
7.75
7.74
— 7.26 CDCl₃

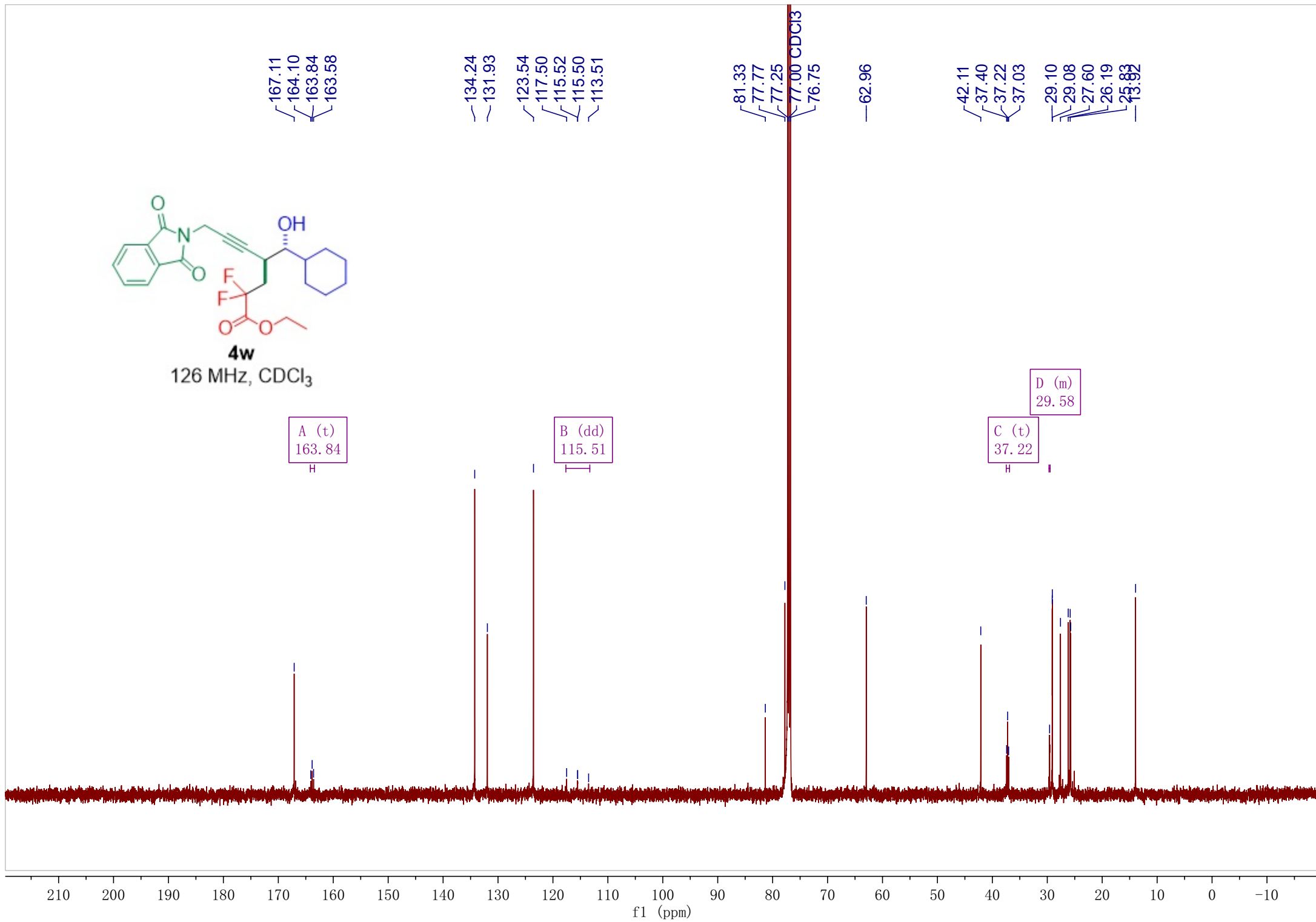
4.43
4.43
4.33
4.31
4.30
4.29

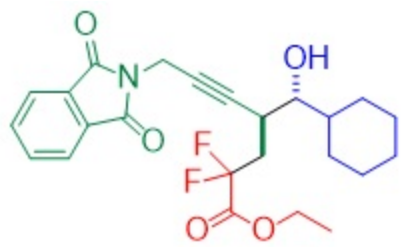
3.15
3.13
2.92
2.52
2.47
2.45
2.34
2.31
2.28
2.02
2.00
1.70
1.65
1.61
1.47
1.43
1.35
1.34
1.32
1.13
1.12
1.00
0.97
0.92
0.90





4w
126 MHz, CDCl₃

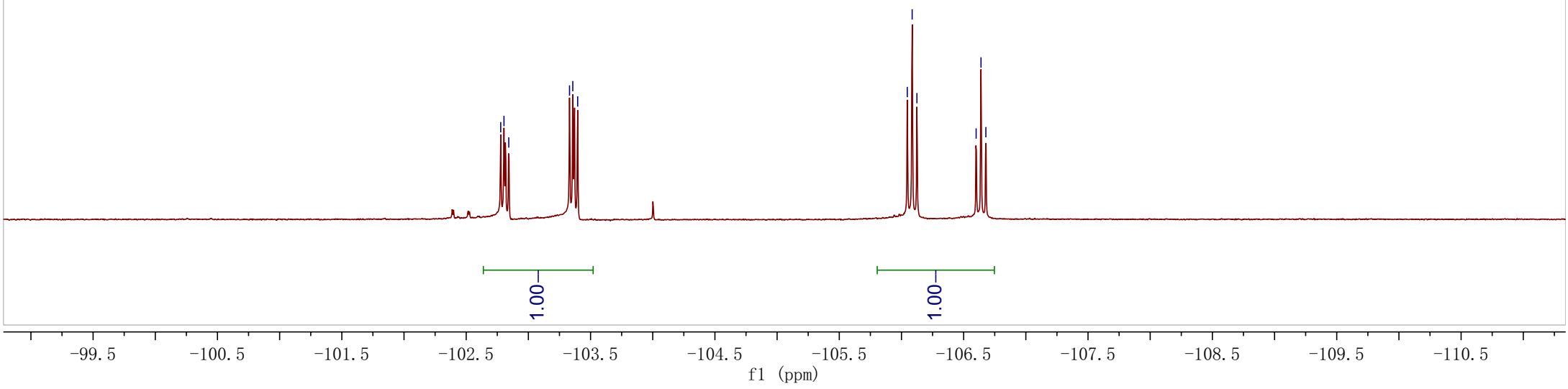


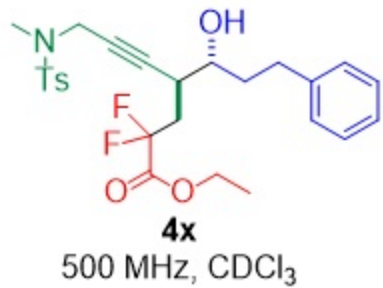


4w
471 MHz, CDCl₃

102.78
102.80
102.84
103.33
103.36
103.40

106.05
106.09
106.12
106.60
106.64
106.68





7.69
7.67
7.31
7.30
7.28
7.27
7.26 CDCl₃
7.25
7.22
7.21
7.19
7.17
7.16

4.33
4.31
4.30
4.28
4.05
4.01
4.00
3.96
3.45
3.43

2.79
2.74
2.62
2.61
2.40
2.24
2.21
1.74
1.71
1.66
1.62
1.36
1.35
1.33

2.16
2.17
2.34
1.05
2.03

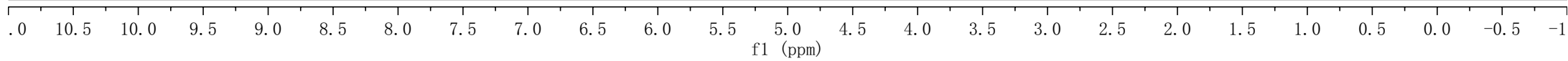
2.15
2.22

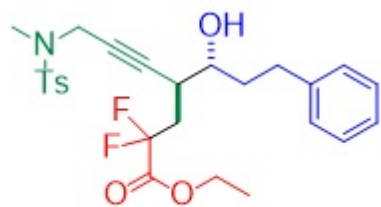
1.00

3.04
1.24
1.95
3.04
2.05

3.17

3.15





4x

126 MHz, CDCl₃

A (t)
163.73
H

B (dd)
115.28
H

C (t)
36.37
H

D (m)
32.31
H

163.99
163.73
163.47

143.79
141.26

129.60
128.50
128.34
127.86
126.97
117.27
115.31
115.27
113.27

83.24
77.25
77.00 CDCl₃
76.75
72.08

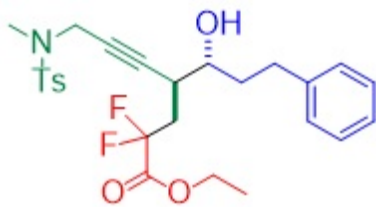
63.05

39.95
36.98
36.55
36.37
36.19
34.43
32.31
31.95
21.46

13.91

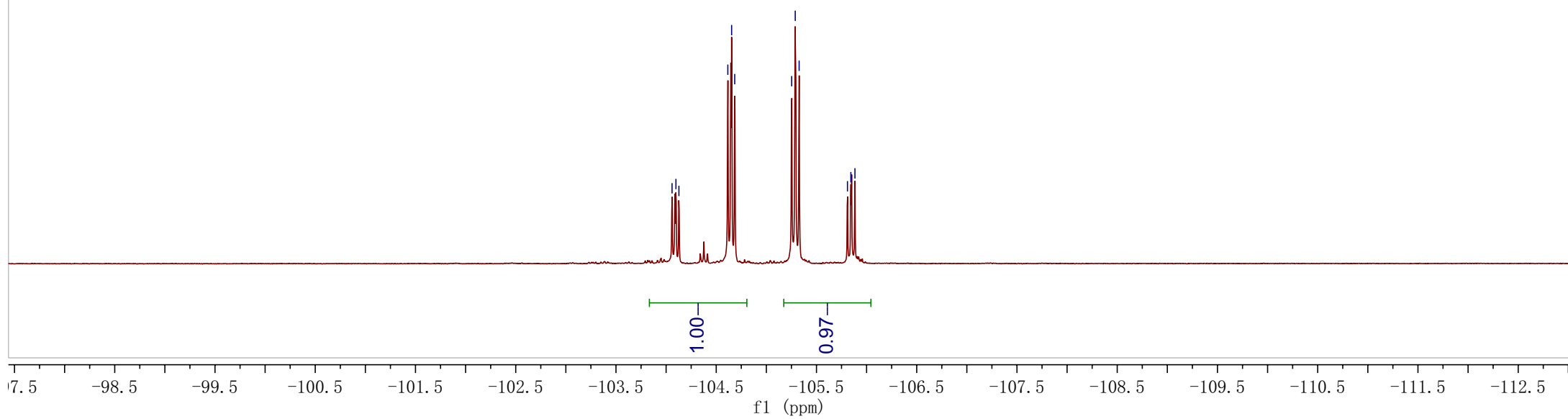
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

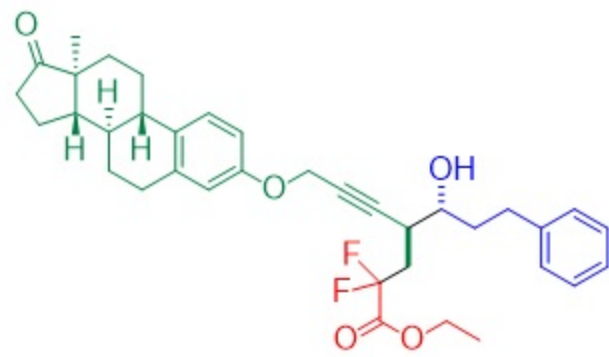
f1 (ppm)



4x
471 MHz, CDCl₃

104.06
104.10
104.13
104.62
104.65
105.29
105.33
105.81
105.84
105.88





4y
500 MHz, CDCl₃

7.34
7.33
7.31
7.26 CDCl₃
7.25
7.23
7.21
7.19
6.82
6.82
6.80
6.80
6.75
6.74

4.71
4.71
4.38
4.36
4.35
4.34

—3.61

2.92
2.91
2.53
2.43
2.22
2.18
2.01
1.89
1.87
1.54
1.52
1.41
1.40
1.38

2.25
2.09
1.94

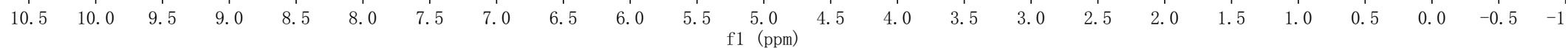
1.02
0.96

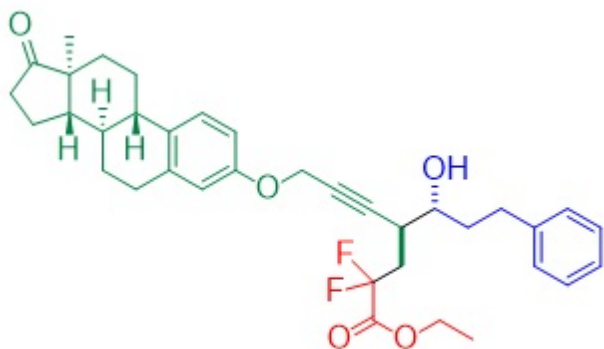
2.00

2.11

1.00

3.15
1.05
1.10
2.09
2.20
1.10
1.10
1.09
2.10
2.07
1.17
6.99
3.14
3.15





4y

126 MHz, CDCl₃

A (t)
163.75
H

B (m)
115.38
H

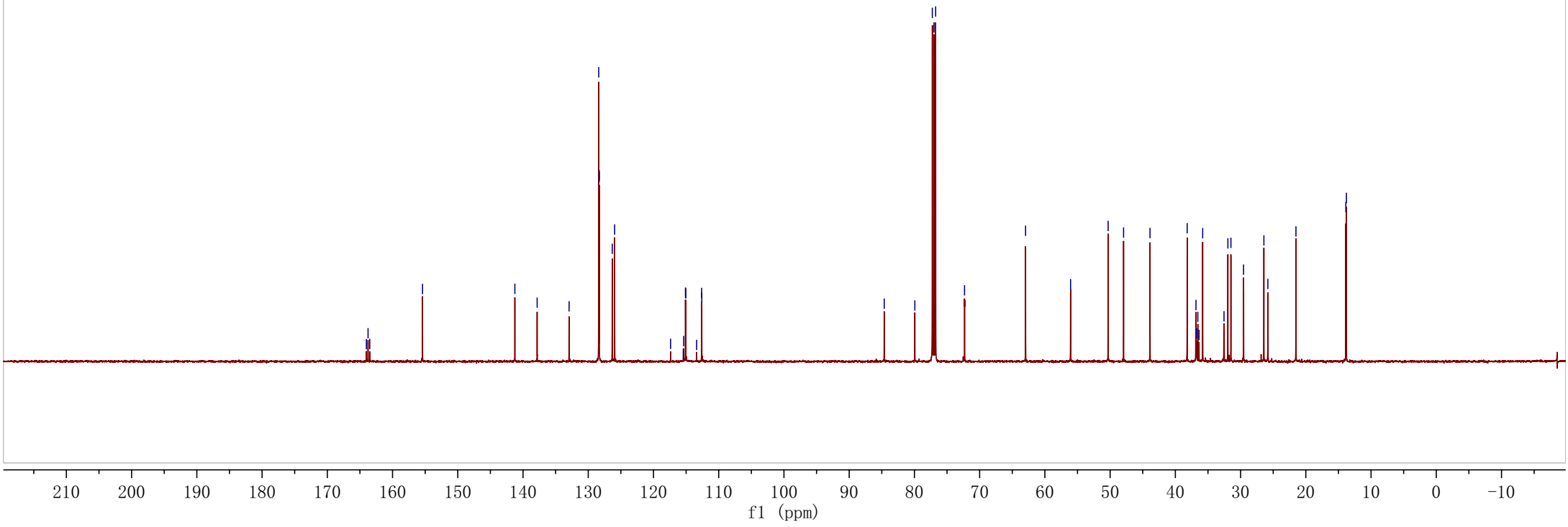
C (t)
36.55
H

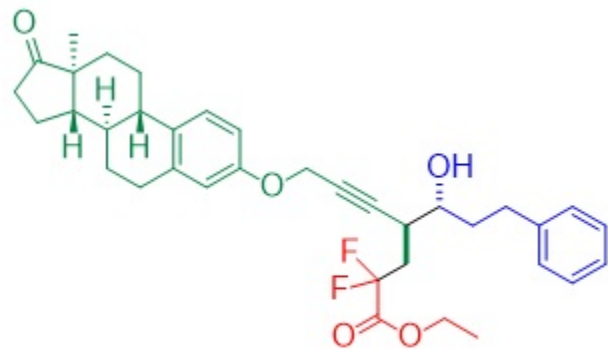
D (m)
32.53
H

- 164.01
- 163.75
- 163.50
- 155.40
- 141.25
- 137.83
- 132.94
- 128.39
- 128.32
- 128.31
- 126.32
- 125.95
- 117.38
- 115.44
- 115.37
- 115.10
- 115.05
- 113.38
- 112.62
- 112.59

- 84.63
- 79.93
- 77.25
- 77.00 CDCl₃
- 76.75
- 72.32

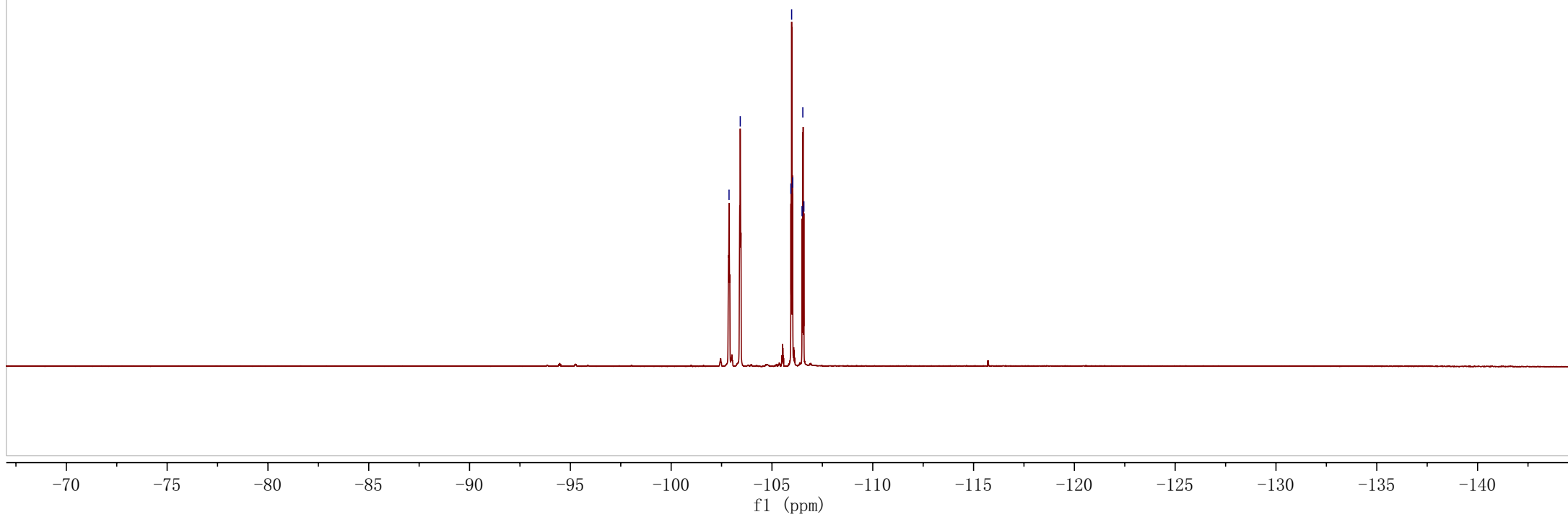
- 62.96
- 56.06
- 50.30
- 47.93
- 43.88
- 38.19
- 36.84
- 35.81
- 31.95
- 31.48
- 29.54
- 26.43
- 25.82
- 21.51
- 13.86
- 13.77

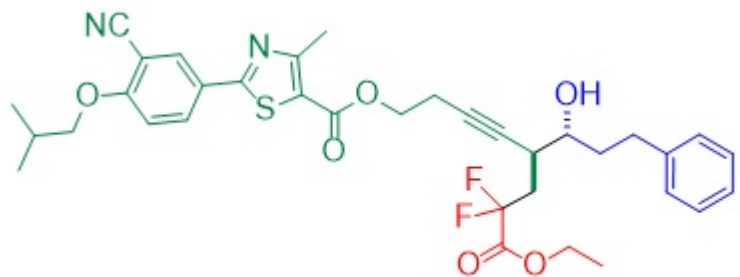




4y
471 MHz, CDCl₃

102.87
103.43
105.94
105.98
106.03
106.49
106.53
106.58

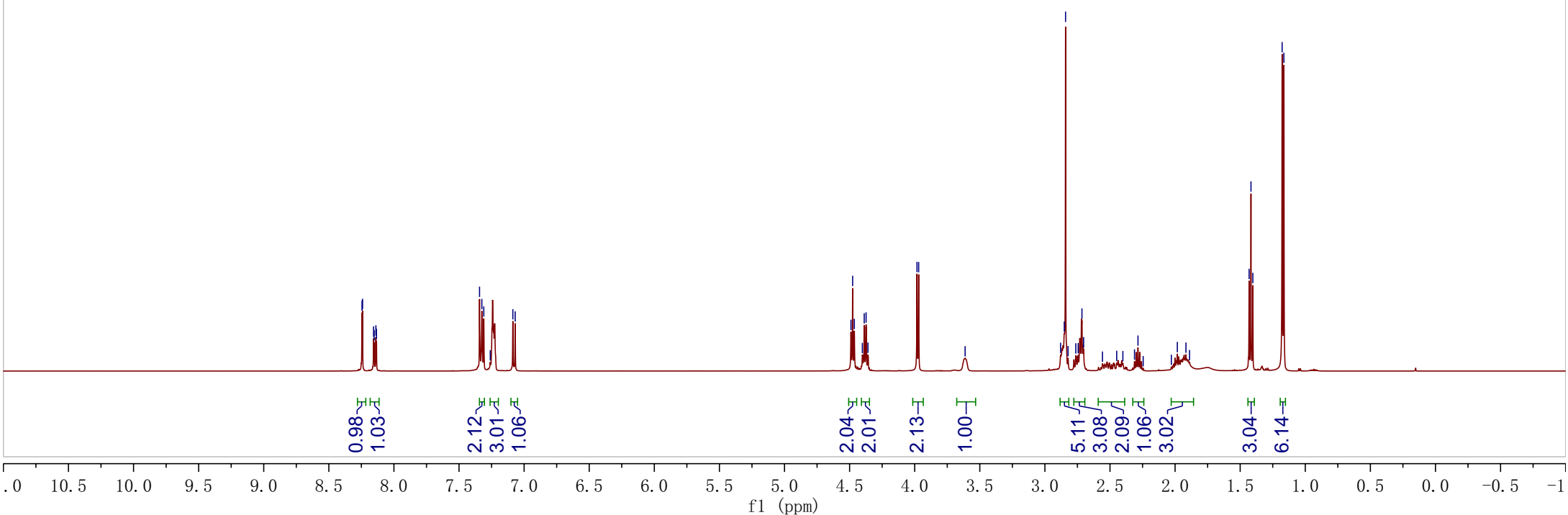


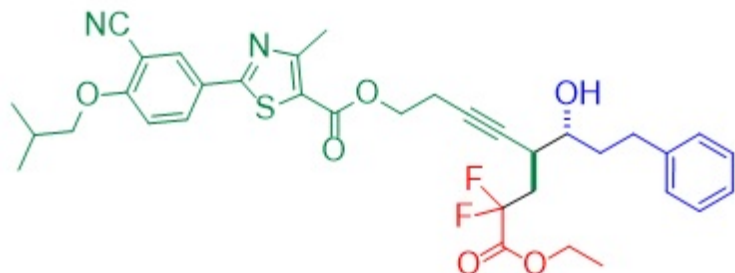


4z
500 MHz, CDCl₃

8.25
8.24
8.16
8.15
8.14
8.13
7.34
7.32
7.31
7.26 CDCl₃
7.09
7.07

4.49
4.48
4.46
4.40
4.39
4.37
4.36
3.98
3.97
— 3.61
2.88
2.85
2.84
2.82
2.76
2.74
2.72
2.70
2.29
1.98
1.92
1.43
1.42
1.40
1.18
1.16





4z

126 MHz, CDCl₃

167.57
164.11
163.85
163.59
162.52
161.86
161.78

141.36
132.58
132.09
128.41
128.30
125.93
125.83
121.11
115.48
115.33
102.59

80.60
79.02
77.25
77.00 CDCl₃
76.75
75.65
72.40
62.99
62.91

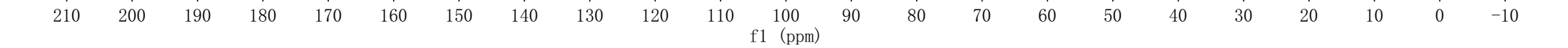
37.19
37.14
37.00
36.82
32.48
31.99
28.11
19.59
19.02
17.52
13.91

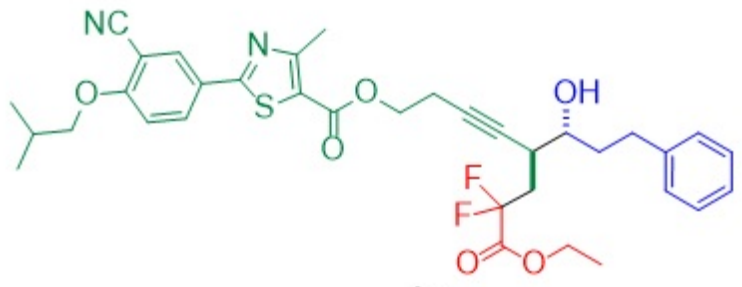
A (t)
163.85
H

B (dd)
115.50
H

C (t)
37.00
H H

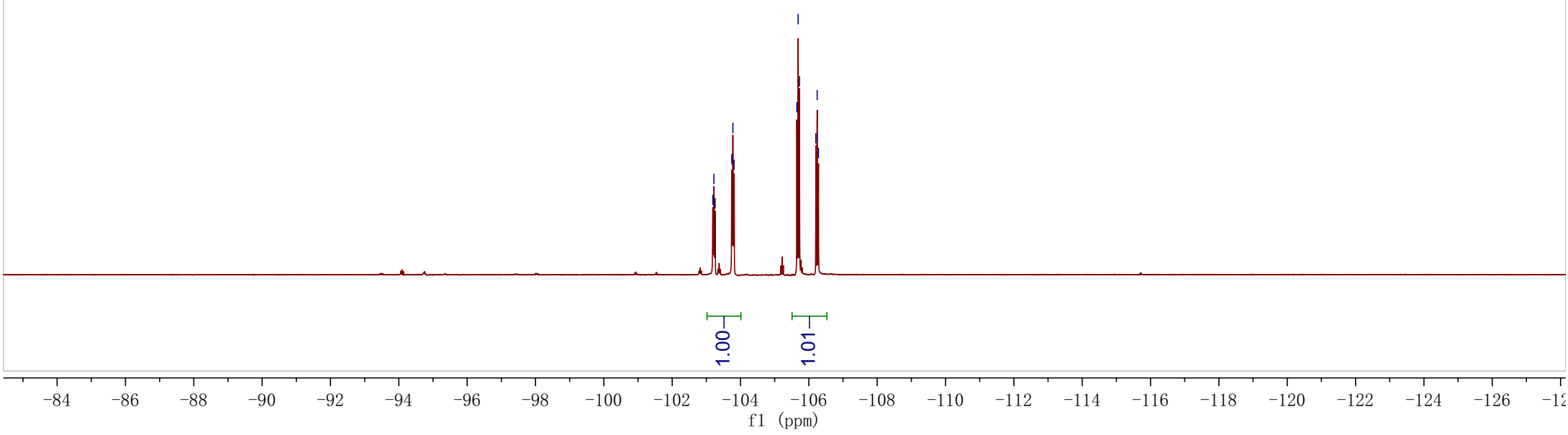
D (m)
32.48

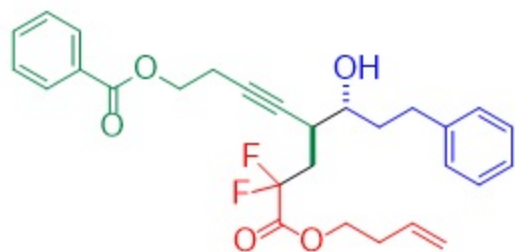




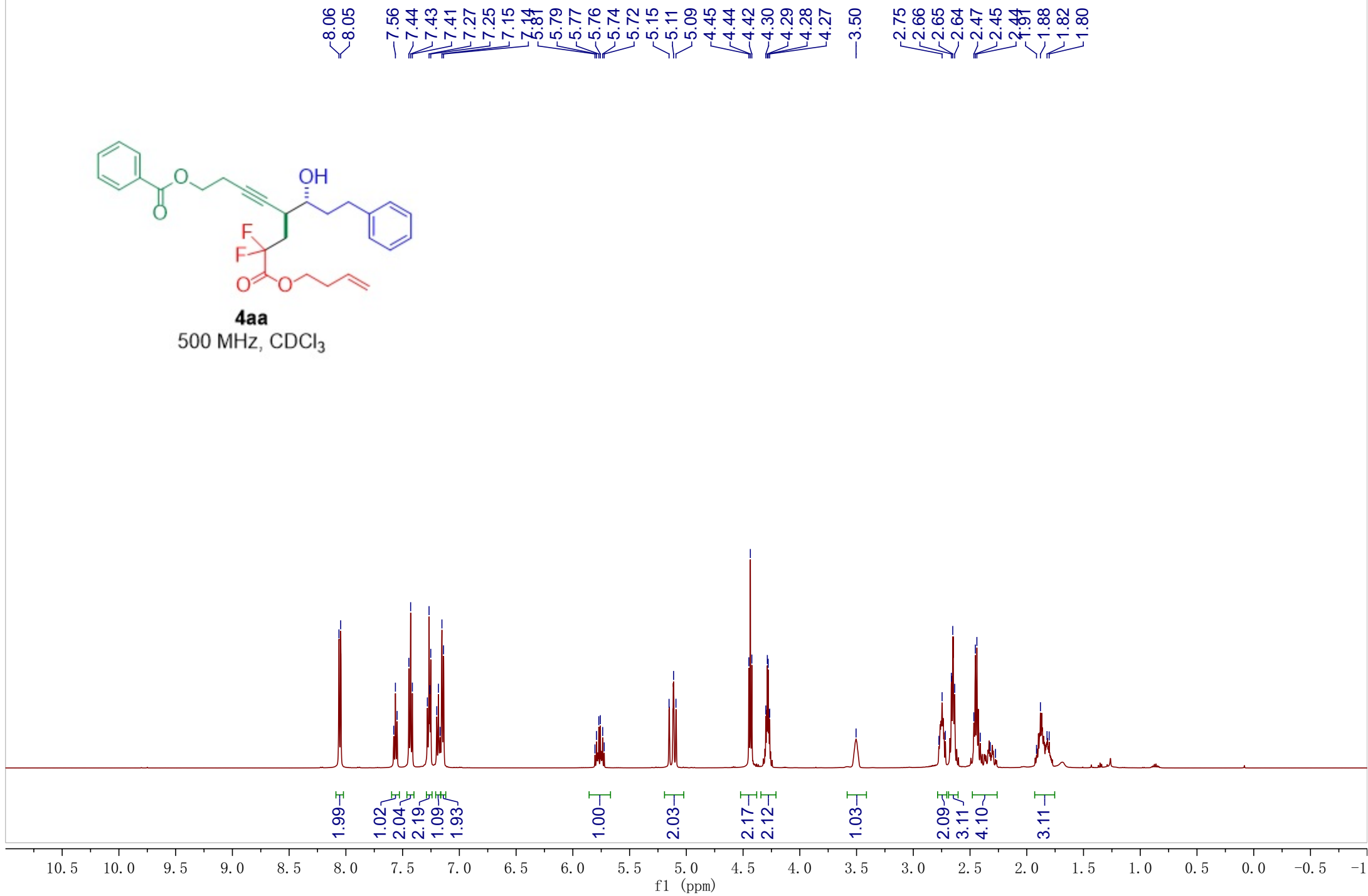
4z
471 MHz, CDCl₃

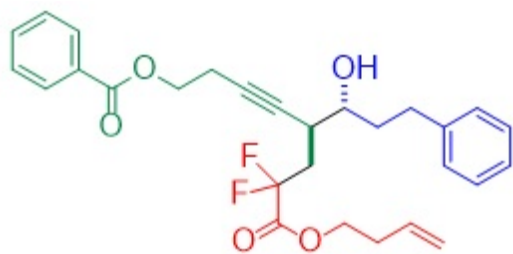
103.19
103.22
103.26
103.75
103.78
103.81
105.65
105.69
105.73
106.20
106.24
106.28





4aa
500 MHz, CDCl₃





4aa
126 MHz, CDCl₃

166.48
164.05
163.79
163.53

141.42
133.15
129.62
128.40
128.35
125.89
118.00
117.50
115.51
115.45
113.51

80.94
78.79
77.25
77.00 CDCl₃
76.75
72.38
65.61
62.70

37.23
37.10
37.05
36.87
32.61
32.57
32.54
32.51
31.94
19.60

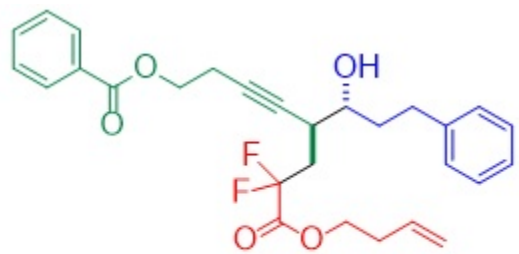
A (t)
163.79
H

B (dd)
115.49
H

C (t)
37.05
H

D (m)
32.54

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



4aa

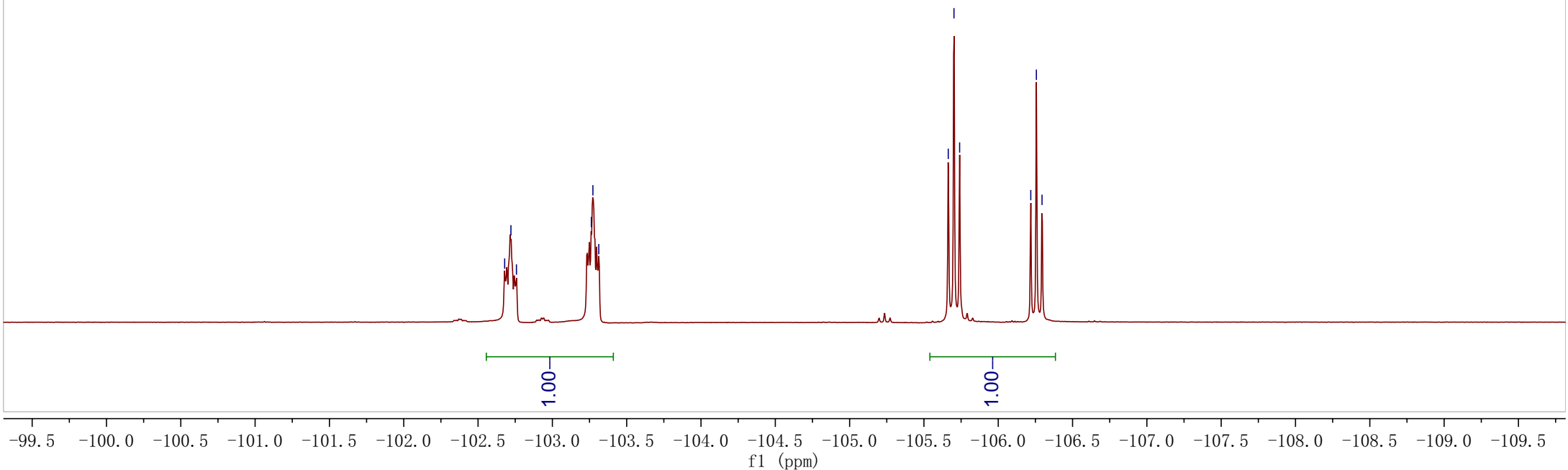
471 MHz, CDCl₃

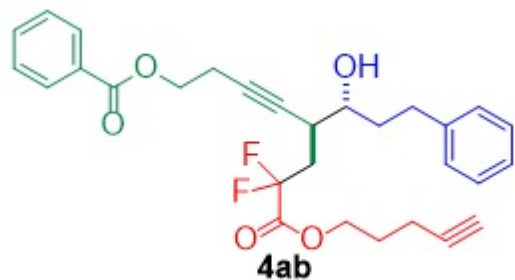
102.68
102.72
102.76

103.26
103.27
103.31

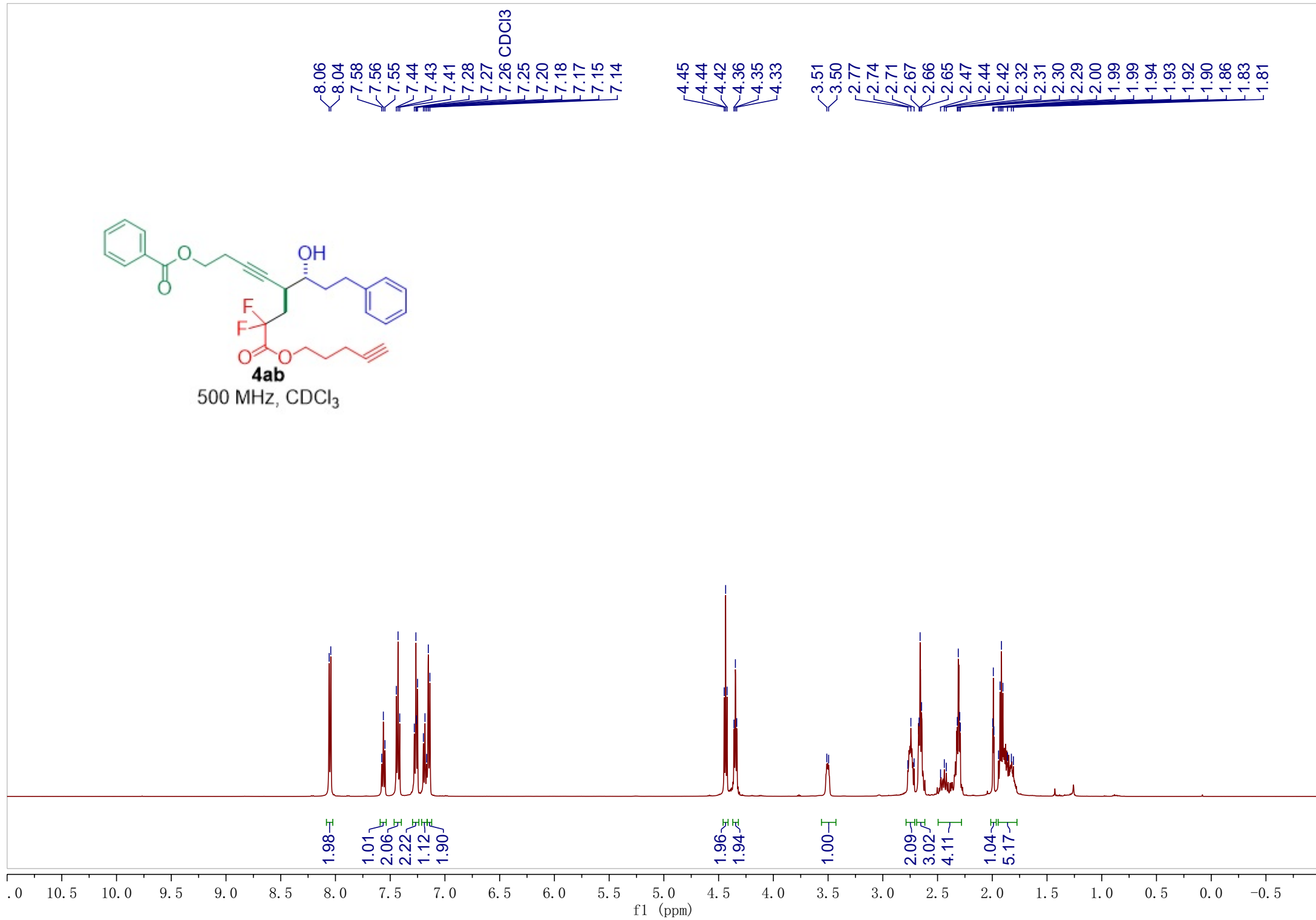
105.66
105.70
105.74

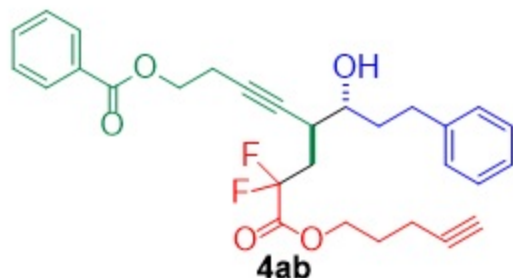
106.22
106.26
106.29





500 MHz, CDCl₃





126 MHz, CDCl₃

166.48
164.04
163.78
163.52

141.40
133.15
129.61
128.40
128.35
125.89
117.51
115.53
115.51
113.52

82.37
81.00
78.73
77.25
77.00 CDCl₃
76.75
72.37
69.49
65.15
62.69

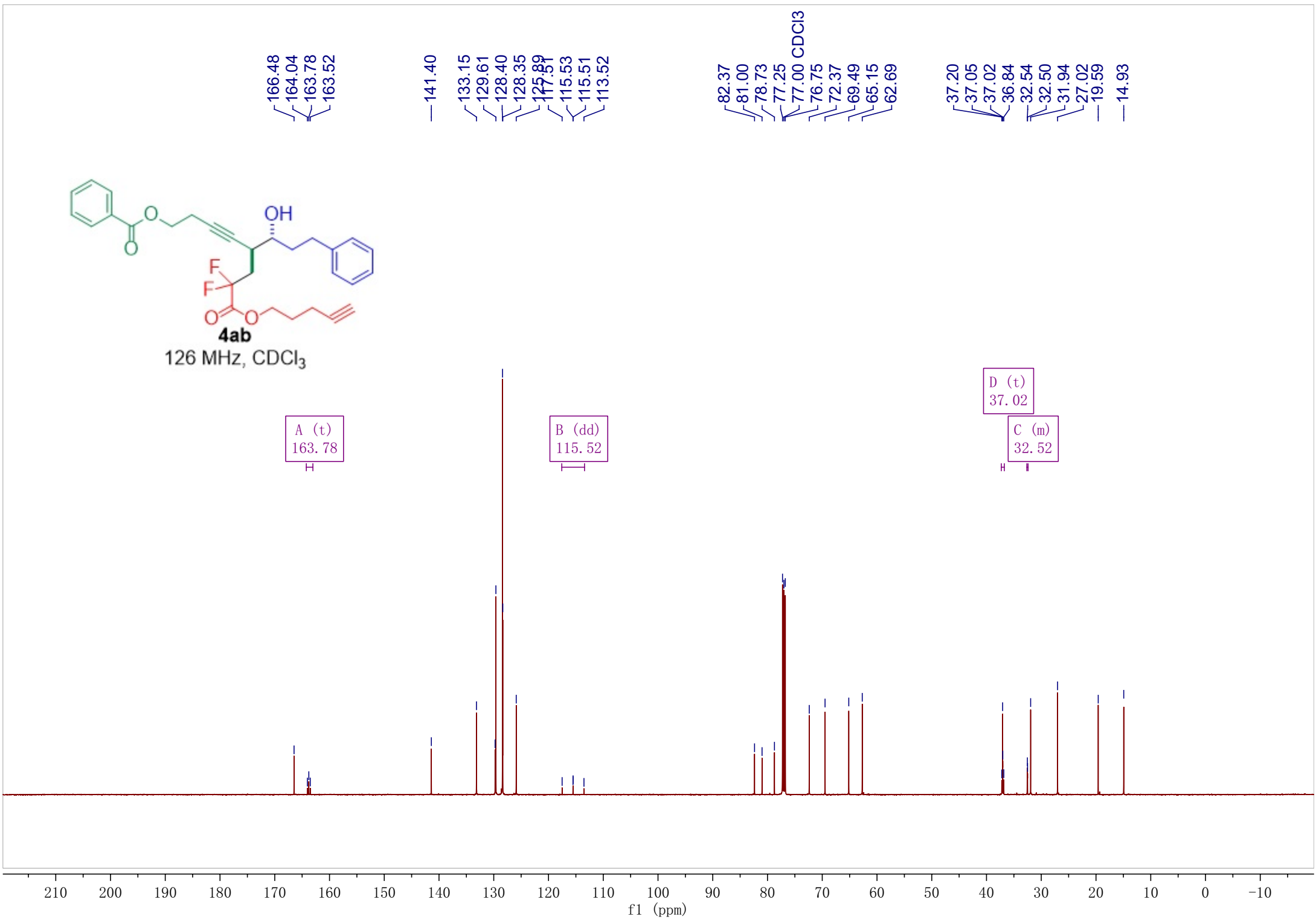
37.20
37.05
37.02
36.84
32.54
32.50
31.94
27.02
19.59
14.93

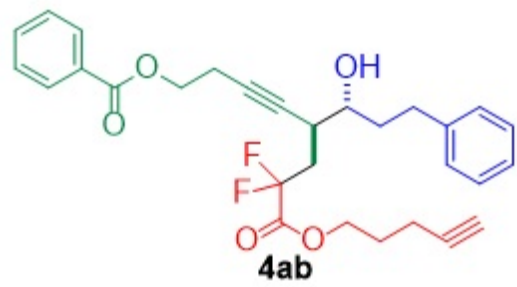
A (t)
163.78
H

B (dd)
115.52
H

D (t)
37.02
H

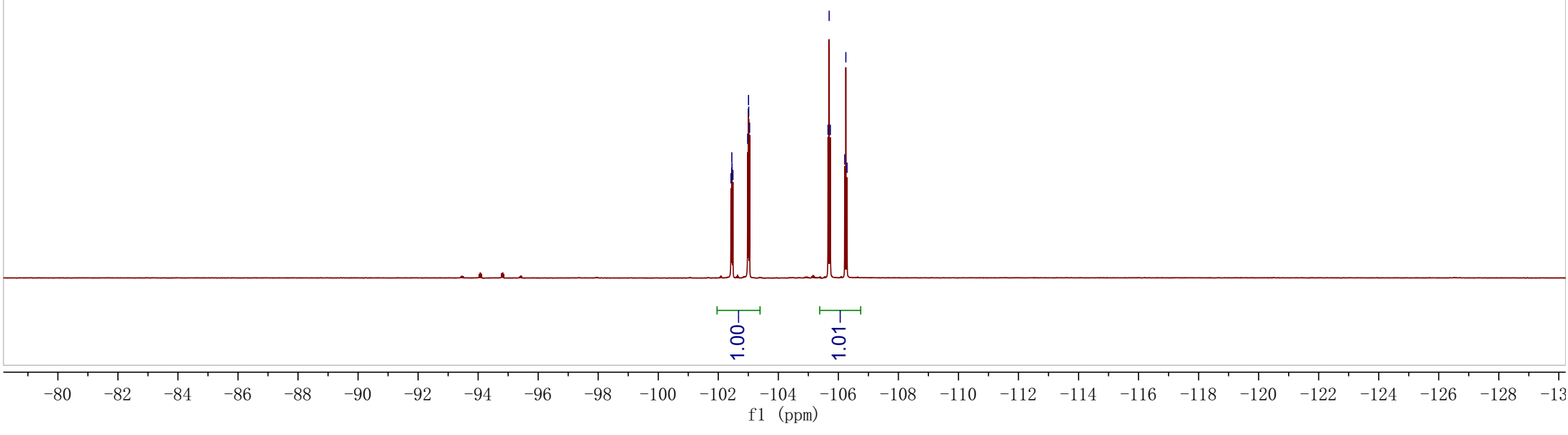
C (m)
32.52
H

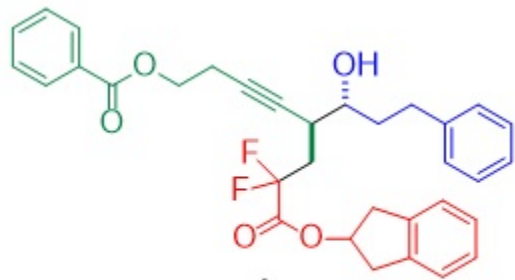




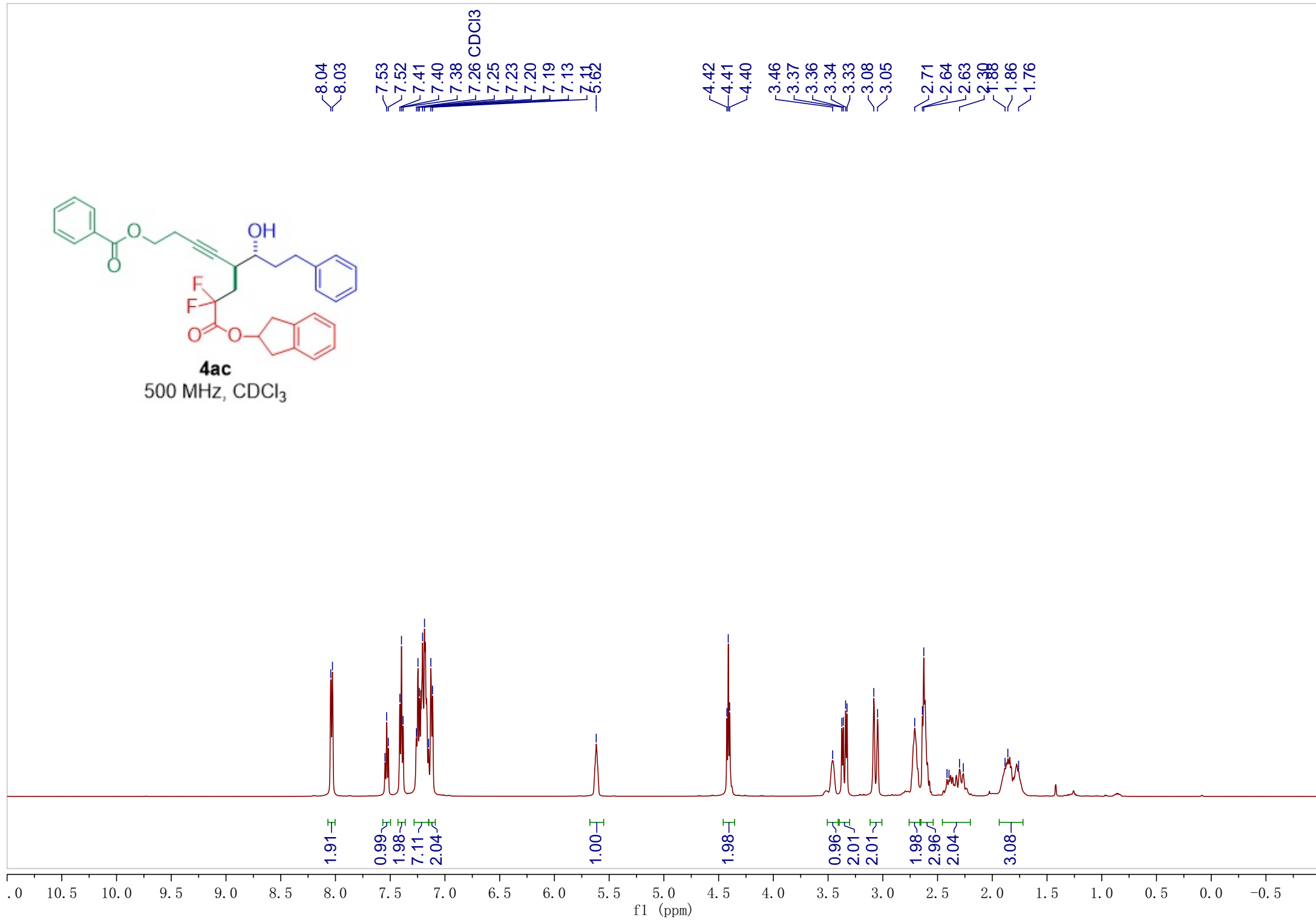
471 MHz, CDCl₃

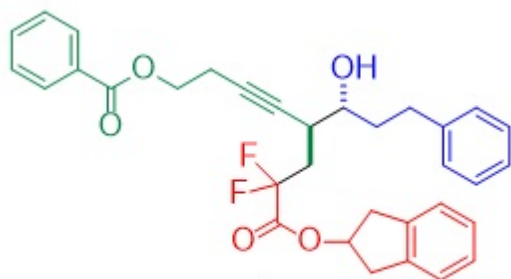
102.42
102.45
102.46
102.49
102.98
103.01
103.01
103.04
105.66
105.69
105.73
106.21
106.25
106.29





4ac
500 MHz, CDCl₃





4ac

126 MHz, CDCl₃

166.47
164.02
163.76
163.50

141.41
139.53
133.13
129.74
129.60
128.38
128.33
126.98
125.87
124.59
117.38
115.38
115.32
113.38

80.89
78.84
78.00
77.26
77.00 CDCl₃
76.75
72.31
—62.69

39.25
39.21
37.06
37.01
36.88
36.70
32.49
31.92
—19.59

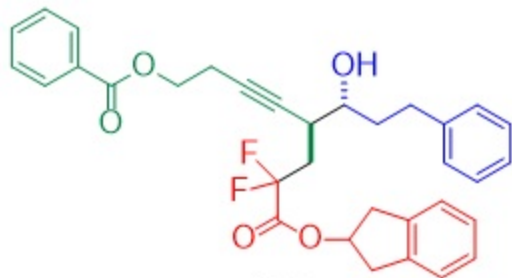
A (t)
163.76
H

B (dd)
115.36
H

C (t)
36.88
H

D (m)
32.49
H

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

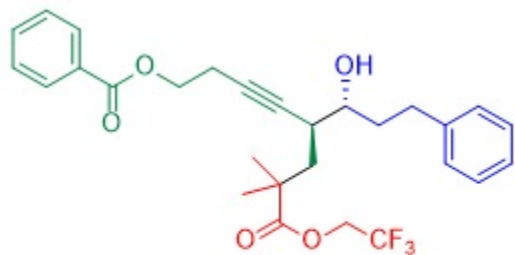


4ac
471 MHz, CDCl₃

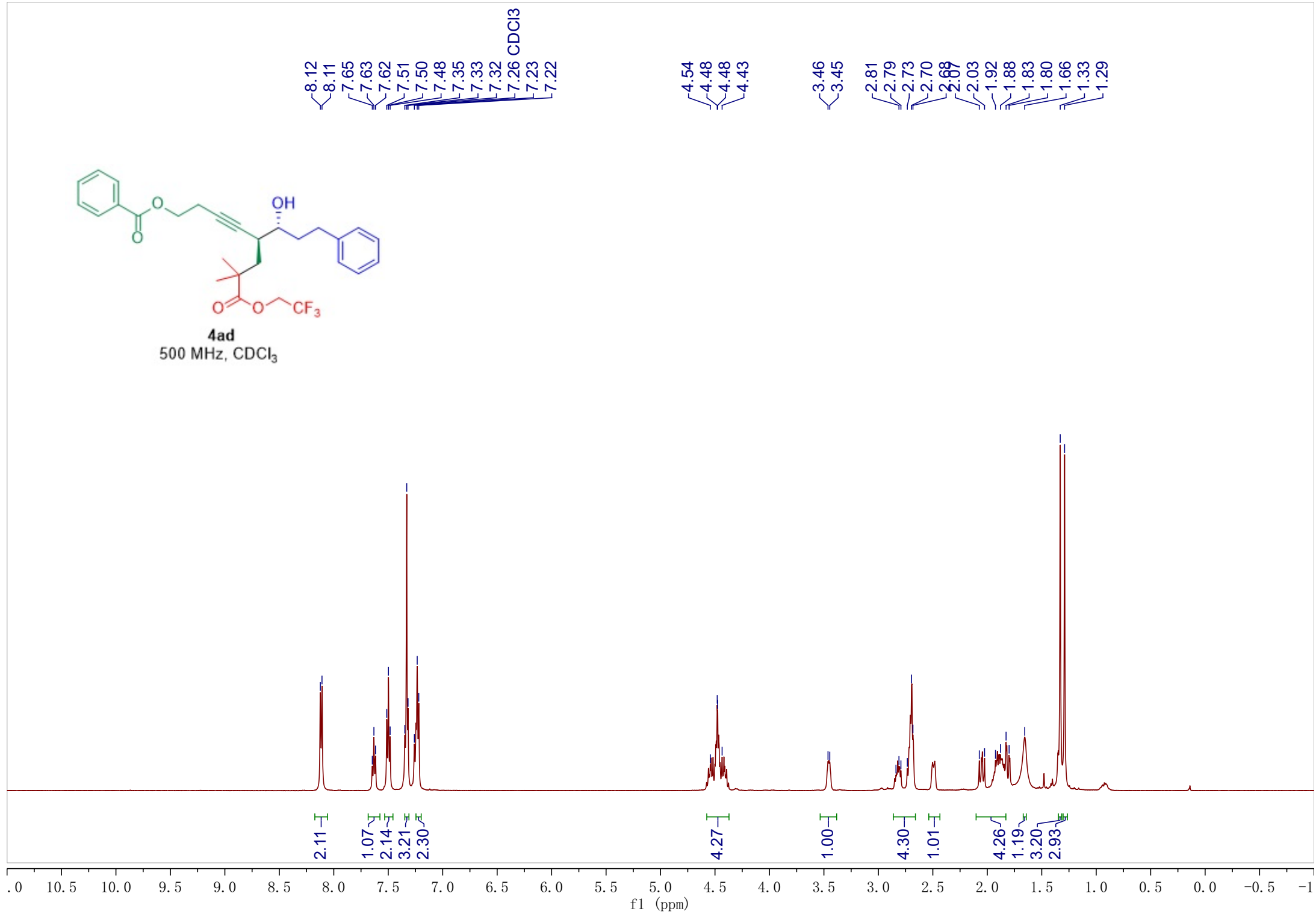
102.79
102.83
102.85
103.35
103.38
103.41
105.14
105.17
105.21
105.69
105.73
105.76

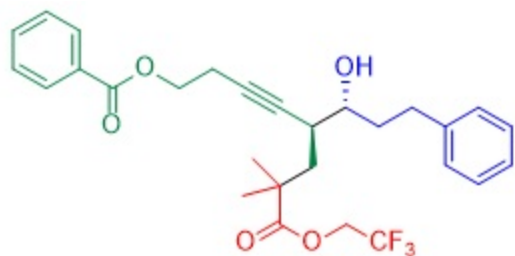
1.00
0.94

20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220
f1 (ppm)



4ad
500 MHz, CDCl₃





4ad
126 MHz, CDCl₃

—176.01

—166.44

—141.75

133.15

129.81

129.63

128.42

128.40

128.37

125.83

124.11

121.91

80.66

77.25

77.00 CDCl₃

76.75

73.39

62.80

60.72

60.43

60.14

59.85

42.04

41.94

37.01

35.58

32.03

25.80

25.08

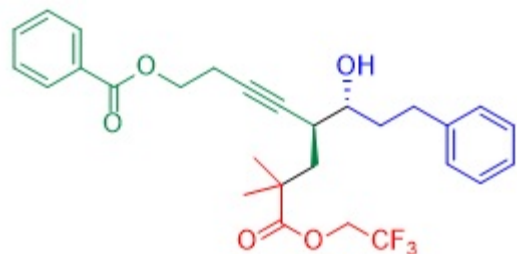
~19.56

B (d)
123.01

A (q)
60.29

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

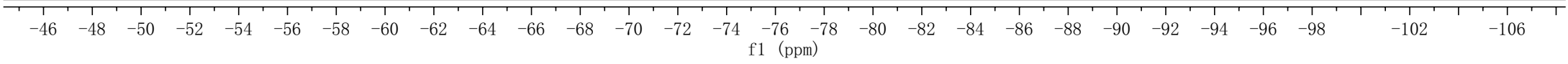
f1 (ppm)

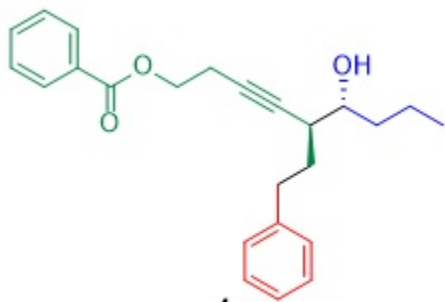


4ad
471 MHz, CDCl₃

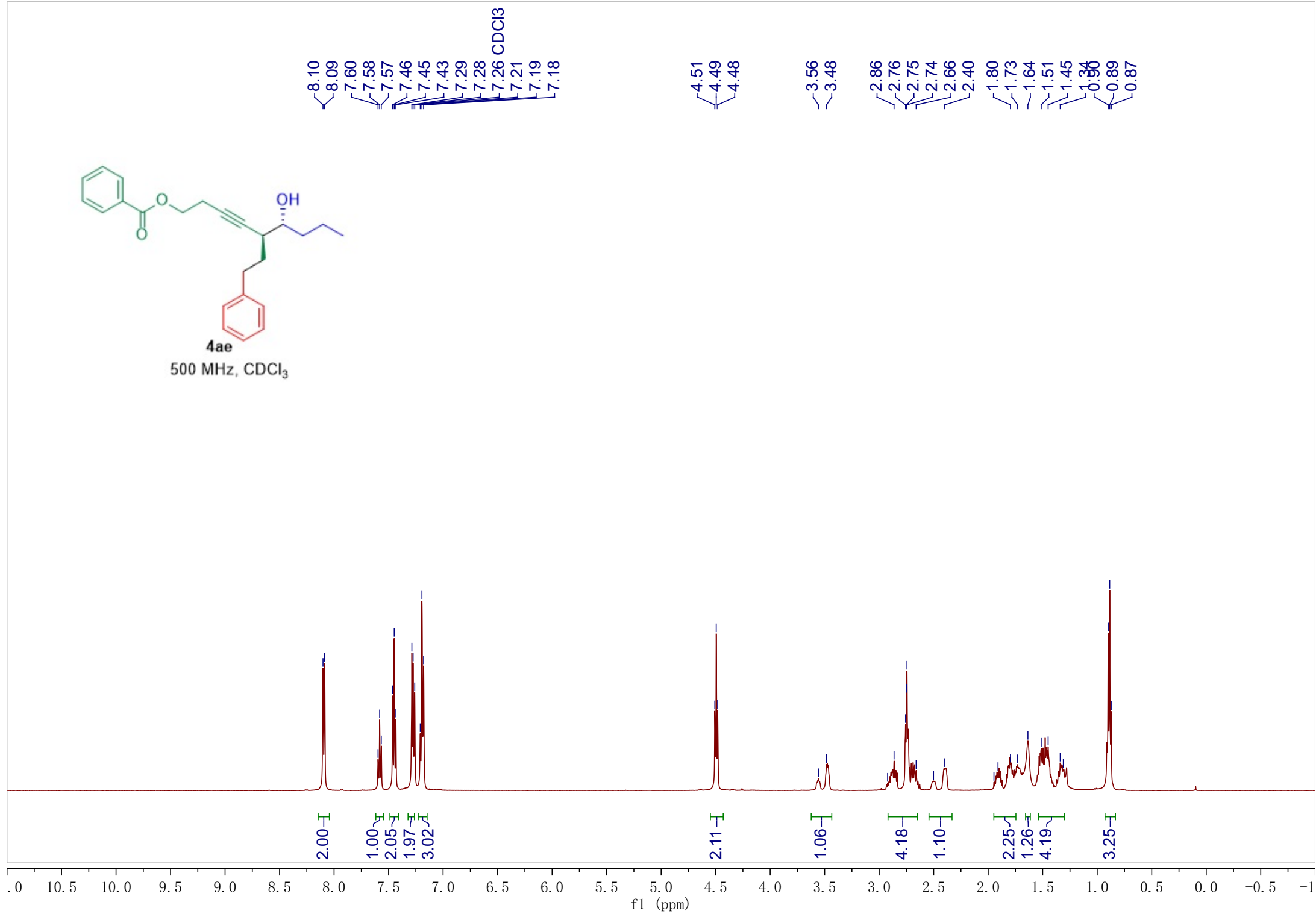
73.69
73.71
73.73

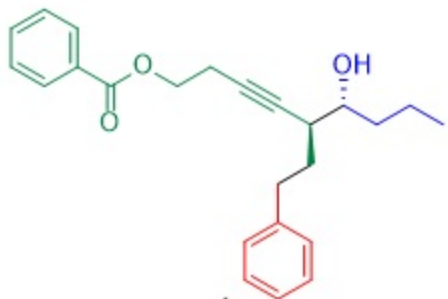
1.00





4ae
500 MHz, CDCl₃





4ae
126 MHz, CDCl₃

166.46

141.80
133.09
129.92
129.63
128.45
128.38
128.30
125.80

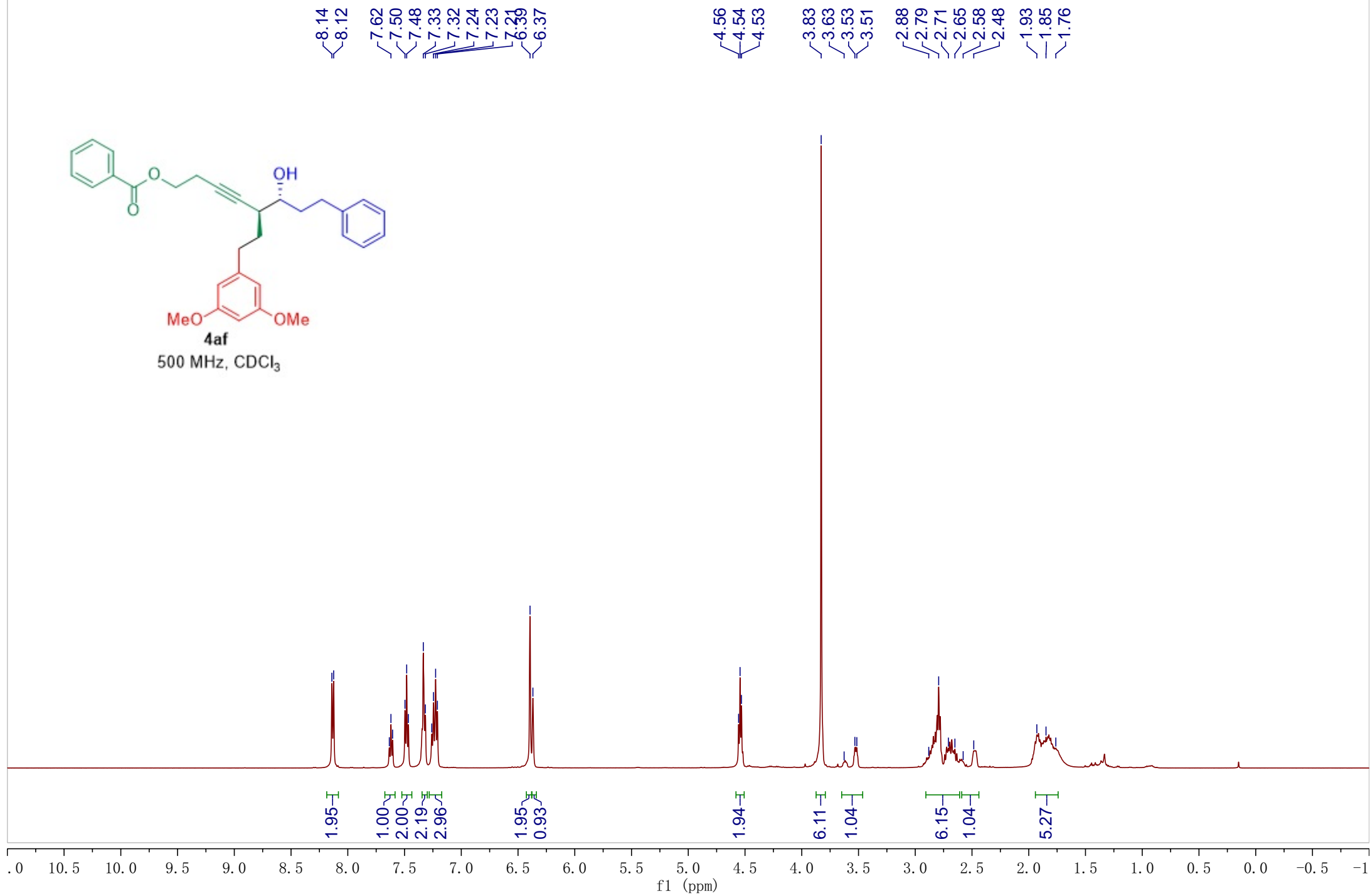
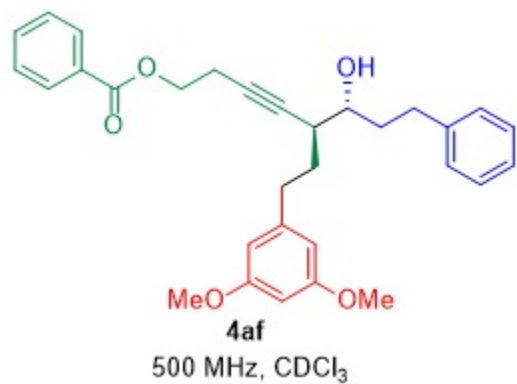
81.00
80.41
77.25
77.00 CDCl₃
76.75
73.01
63.10

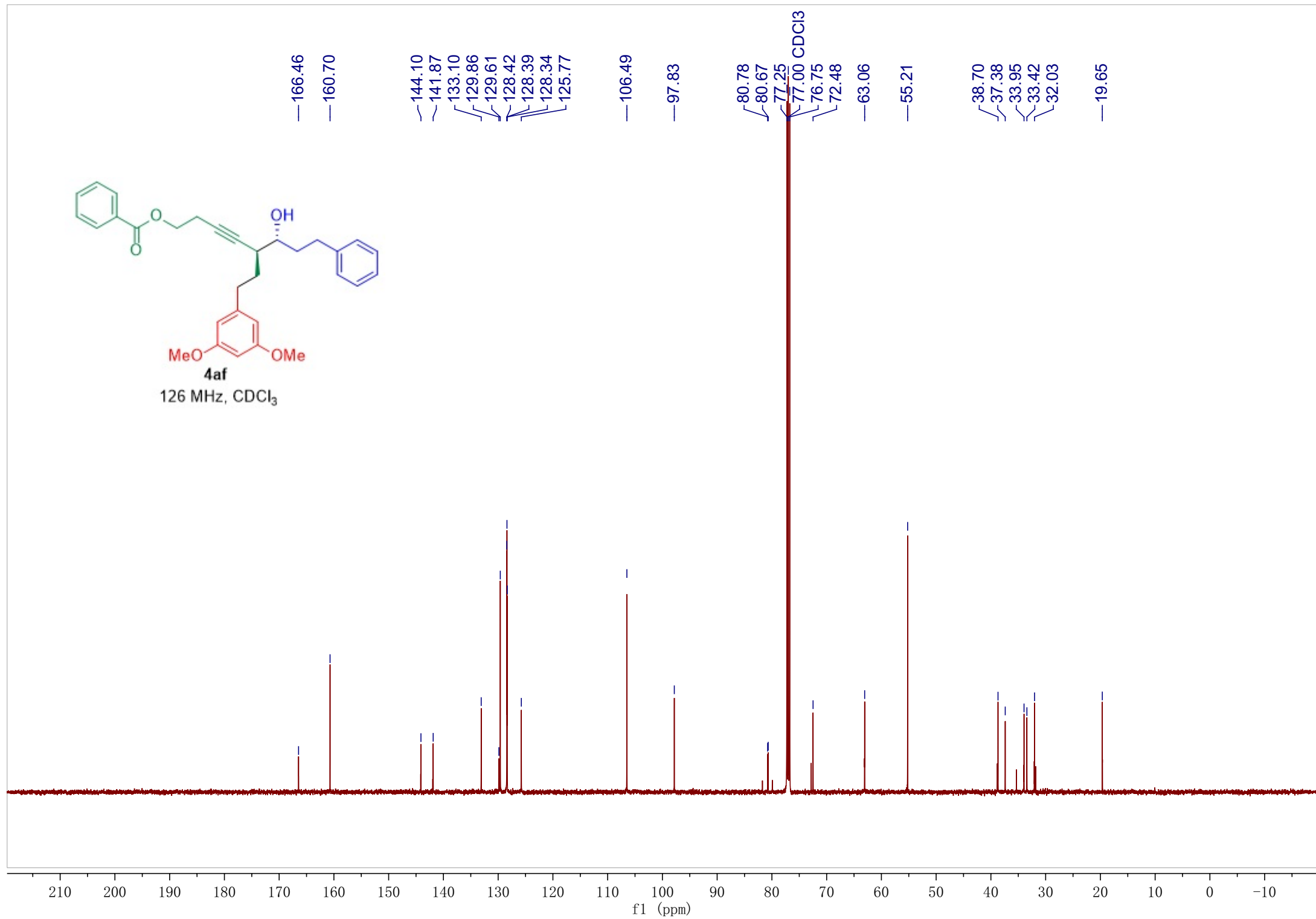
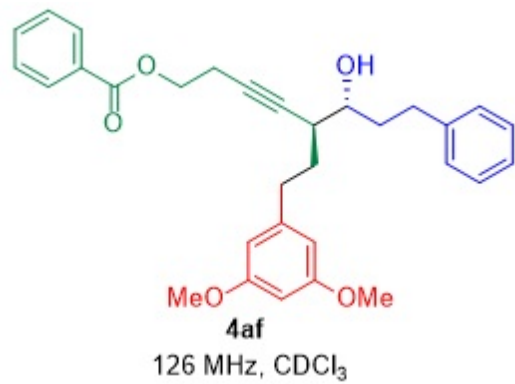
38.64
37.88
33.72
33.70

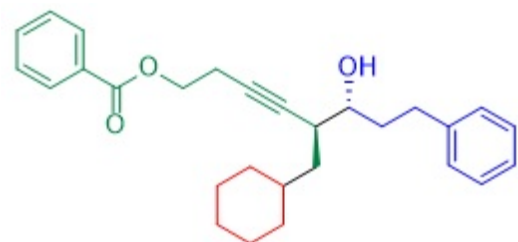
19.64
18.98
13.96

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

f1 (ppm)







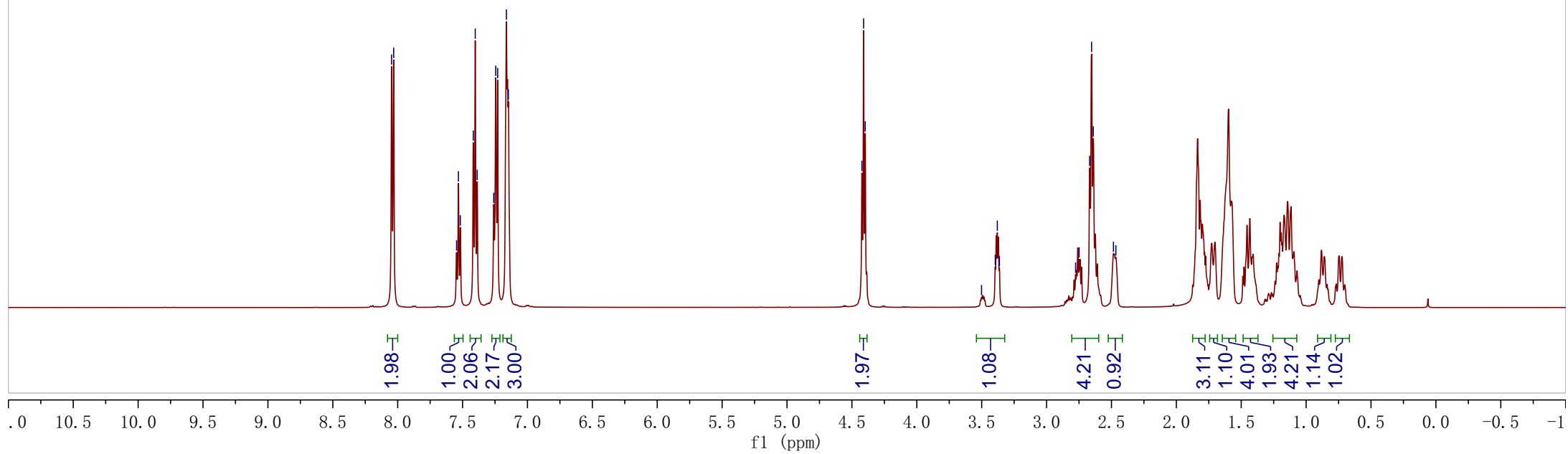
4ag

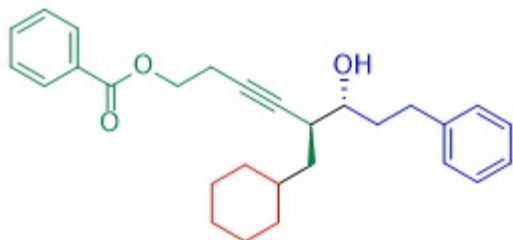
500 MHz, CDCl₃

8.05
8.03
7.55
7.53
7.52
7.42
7.40
7.39
7.26 CDCl₃
7.24
7.23
7.16
7.15

4.42
4.41
4.40

3.50
3.40
3.38
3.36
2.78
2.75
2.67
2.65
2.64
2.48
2.47





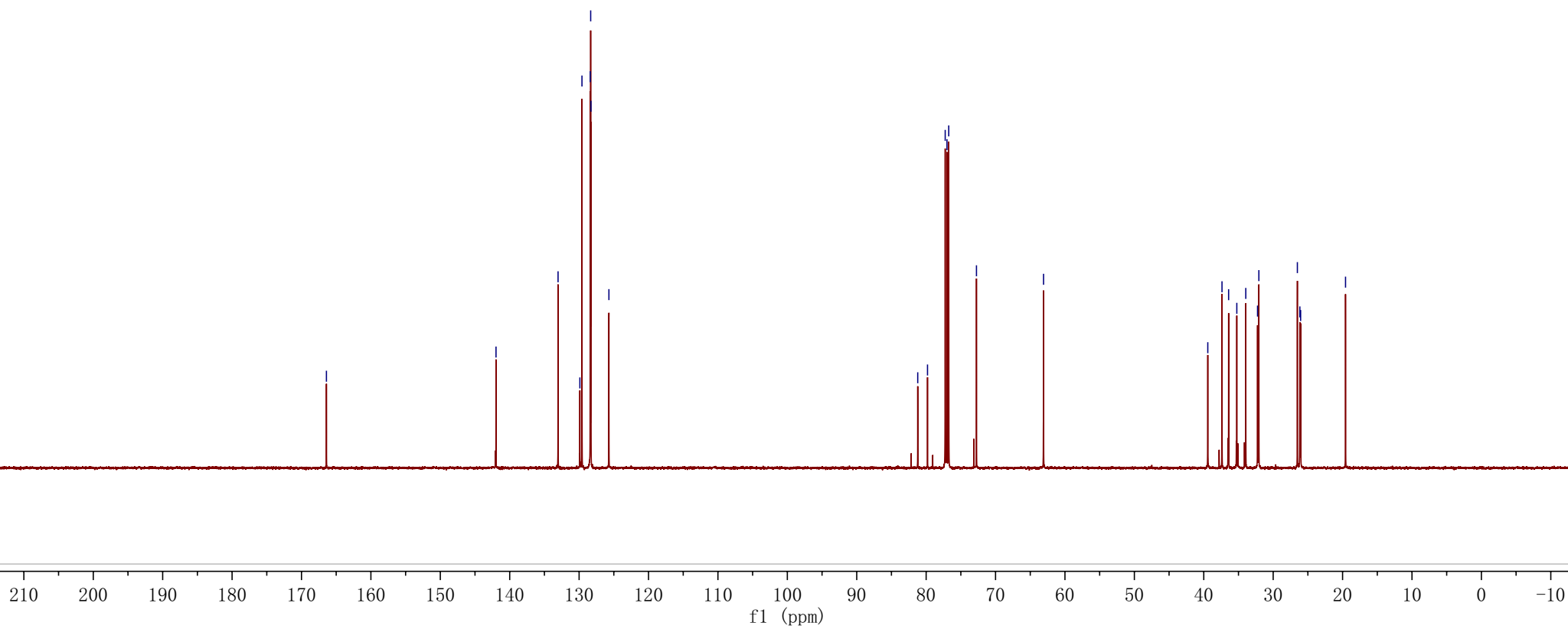
4ag
126 MHz, CDCl₃

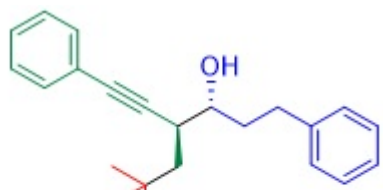
—166.41

—141.97
133.03
129.89
129.59
128.39
128.33
128.30
125.71

81.21
79.80
77.25
77.00 CDCl₃
76.75
72.75
—63.08

39.42
37.37
36.42
35.24
33.95
32.25
32.06
26.51
26.16
26.05
19.57





4ah

500 MHz, CDCl₃

7.35
7.34
7.33
7.26 CDCl₃
7.24
7.24
7.23
7.20
7.18
7.16
7.14
7.13

3.55
3.49
2.84
2.81
2.79
2.72
2.66
2.62
1.92
1.83
1.81
1.67
1.64
1.44
1.41

2.05
4.96
2.17
1.11

1.00

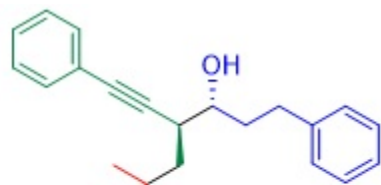
3.01

1.95
0.98
0.87
1.14

9.17

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

f1 (ppm)



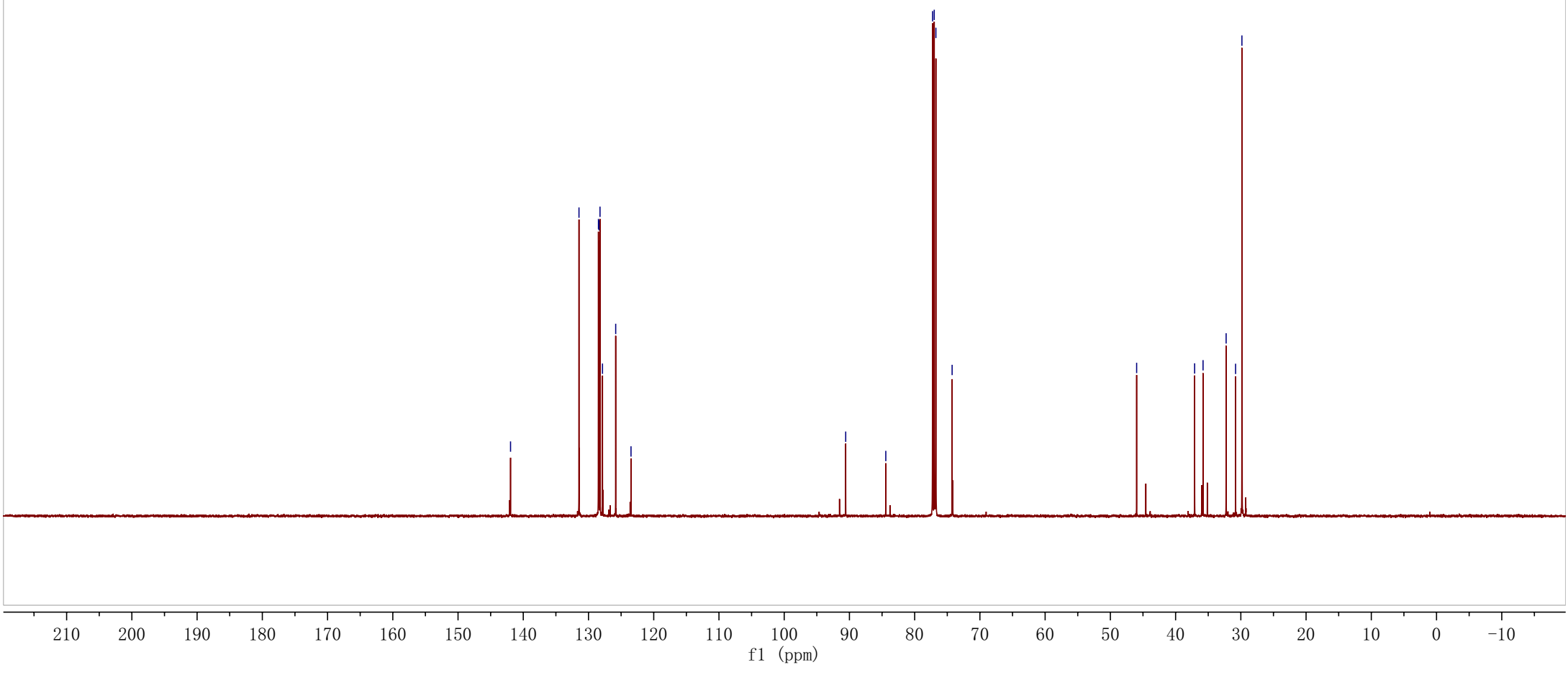
4ah

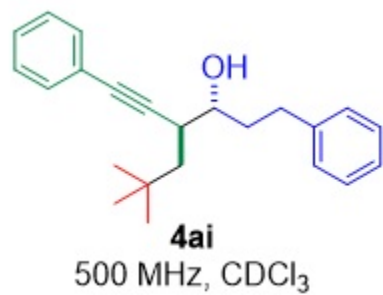
126 MHz, CDCl₃

141.95
131.46
128.48
128.39
128.24
127.85
125.83
123.48

90.57
84.43
77.25
77.00 CDCl₃
76.75
74.26

45.97
37.08
35.79
32.25
30.80
29.83





7.35
7.34
7.33
7.26 CDCl₃
7.24
7.24
7.23
7.20
7.18
7.16
7.14
7.13

3.55
3.49
2.84
2.81
2.79
2.72
2.66
2.62
1.92
1.83
1.81
1.67
1.64
1.44
1.41

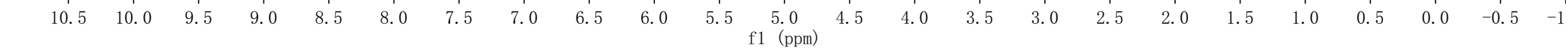
2.05
4.96
2.17
1.11

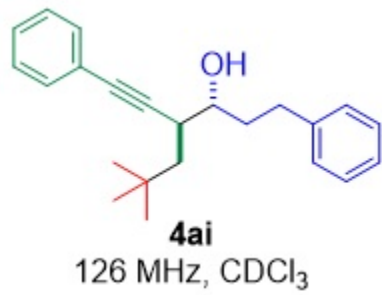
1.00

3.01

1.95
0.98
0.87
1.14

9.17

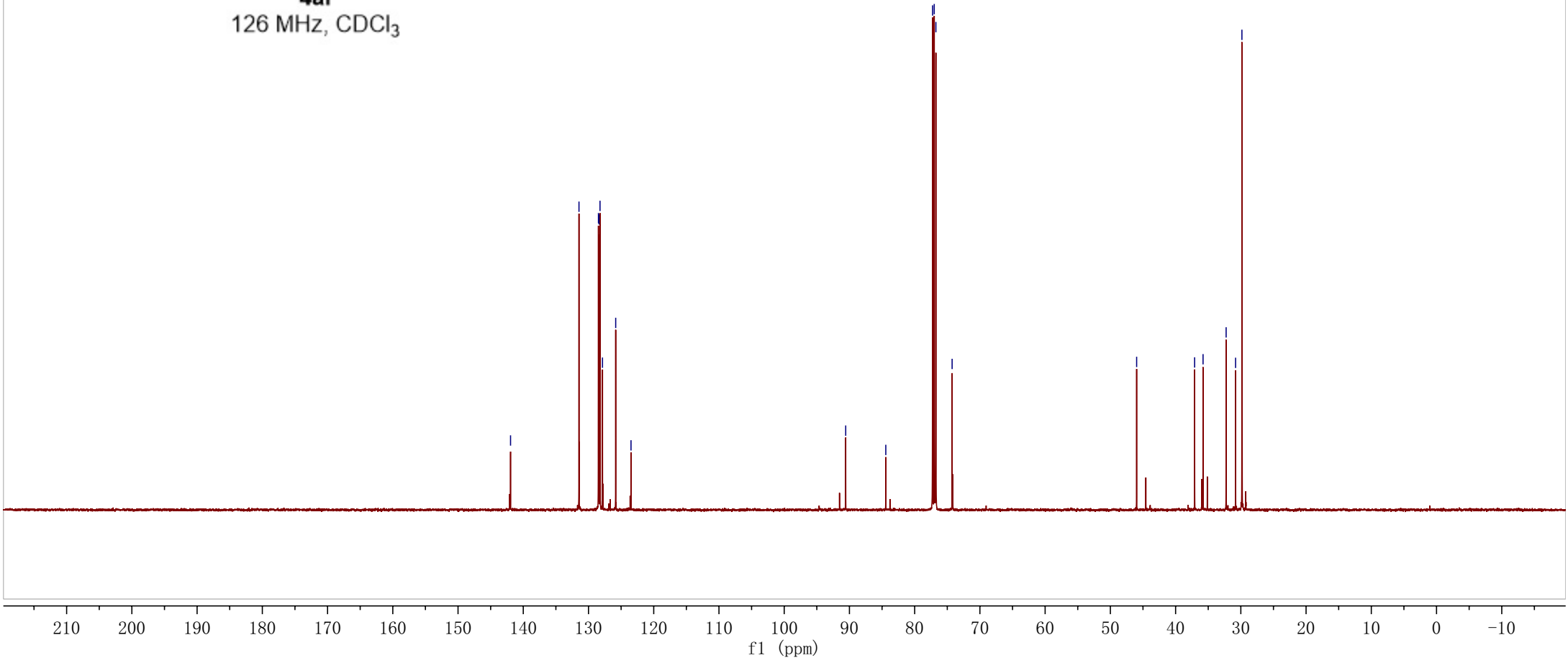




141.95
131.46
128.48
128.39
128.24
127.85
125.83
123.48

90.57
84.43
77.25
77.00 CDCl₃
76.75
74.26

45.97
37.08
35.79
32.25
30.80
29.83



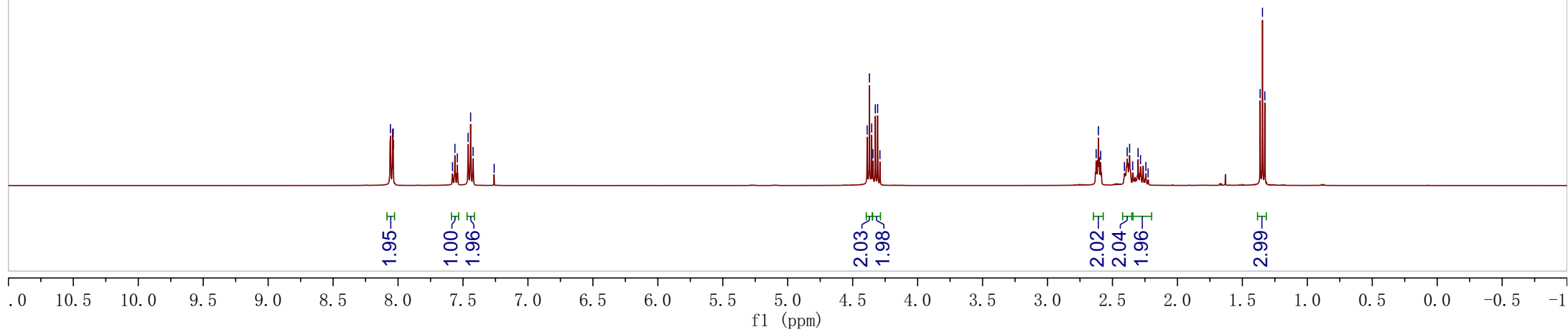


5
500 MHz, CDCl₃

8.06
8.04
7.58
7.56
7.54
7.46
7.44
7.42
7.26 CDCl₃

4.39
4.37
4.35
4.34
4.33
4.31
4.29

2.63
2.61
2.59
2.41
2.39
2.37
2.34
2.30
2.28
2.24
2.23
1.36
1.35
1.33





5
126 MHz, CDCl₃

166.29
164.06
163.73
163.41

133.00
130.01
129.60
128.34
117.63
115.18
115.14
112.64

78.97
77.32
77.00 CDCl₃
76.79
76.68

62.93
62.91

34.25
34.02
33.78

19.27
13.89
11.80
11.74
11.68

A (t)
163.73
H

B (dd)
115.14
H

C (t)
34.02
H

D (t)
11.74
H

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



5

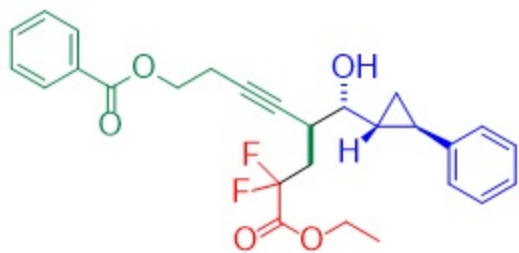
471 MHz, CDCl₃

101.88
102.44
103.03
105.63
105.67
105.74
106.19
106.23
106.30

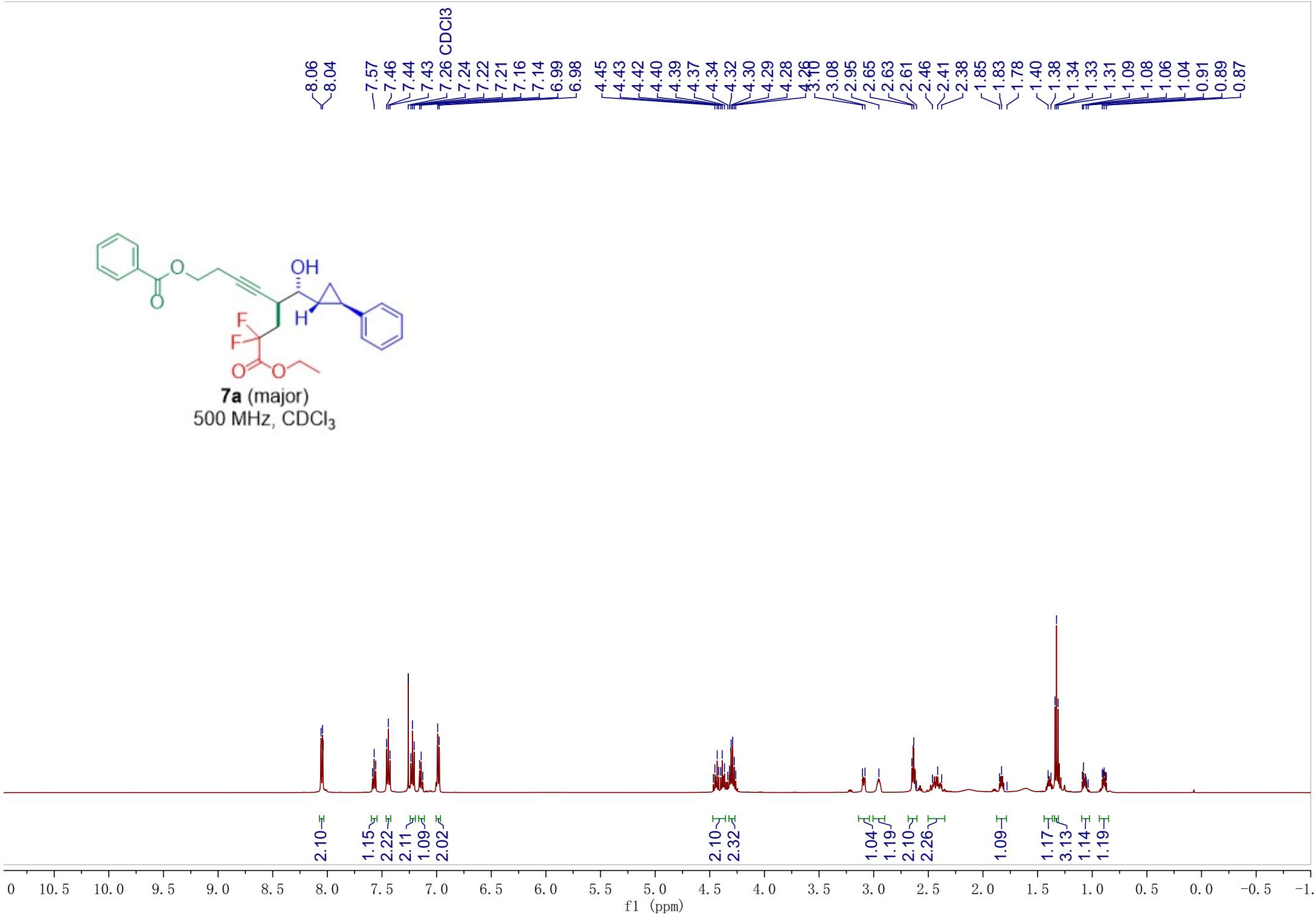
1.00
0.94

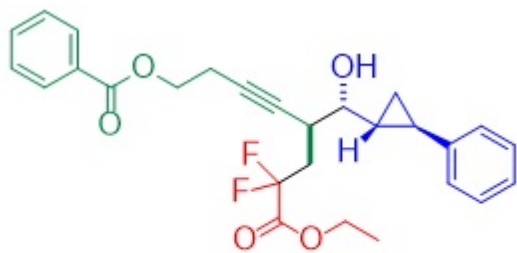
-10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -2

f1 (ppm)

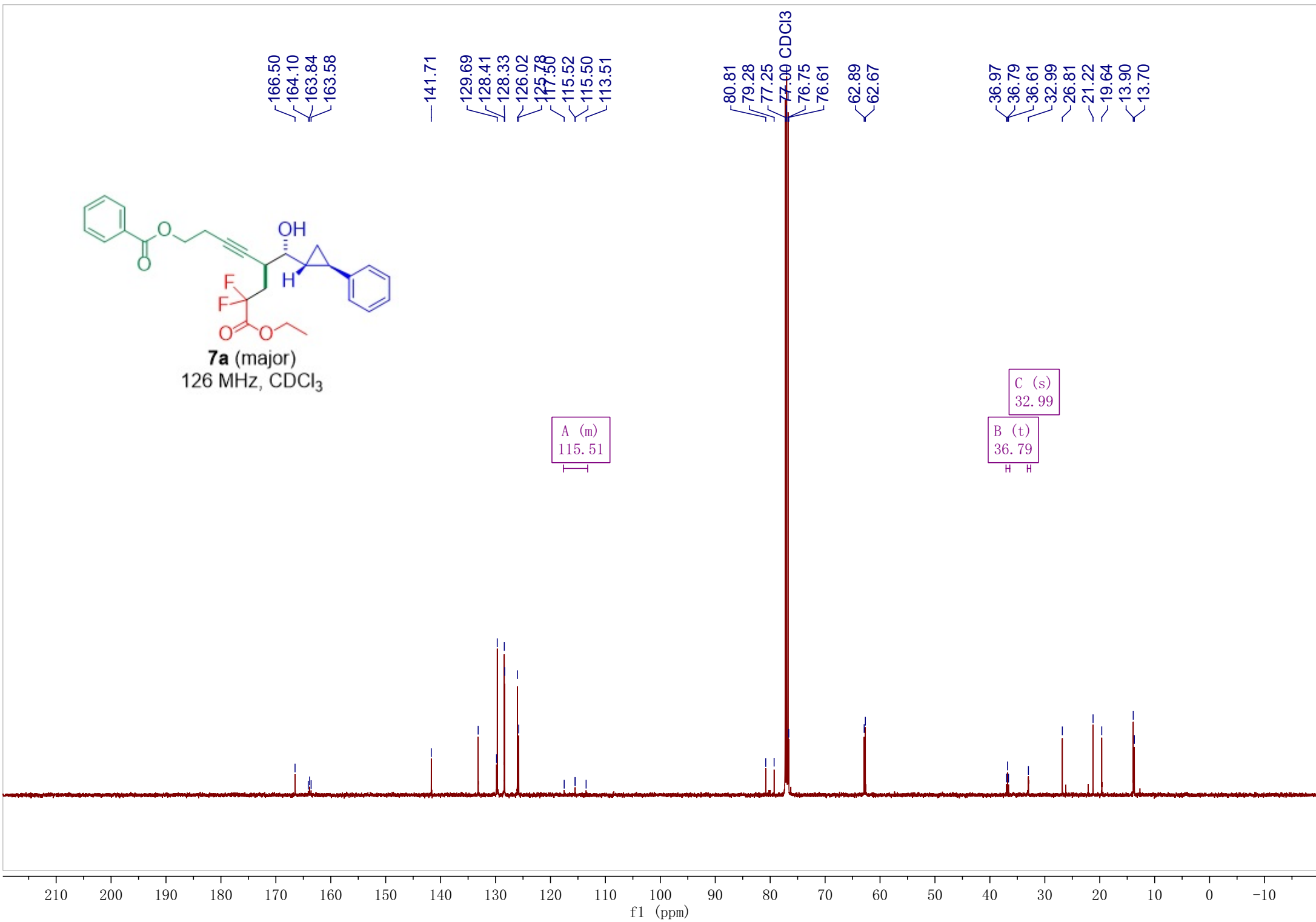


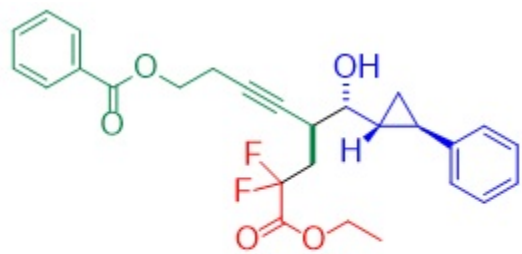
7a (major)
500 MHz, CDCl₃





7a (major)
126 MHz, CDCl₃





7a (major)
471 MHz, CDCl₃

102.66
102.69
102.72
103.21
103.25
103.28
105.89
105.93
105.97
106.44
106.48
106.52

1.00

0.99

f1 (ppm)

-85

-90

-95

-100

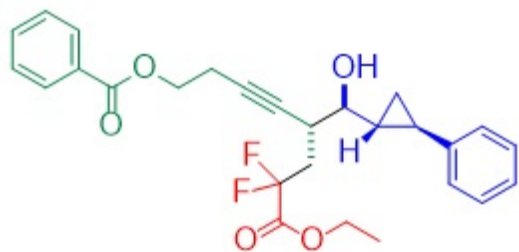
-105

-110

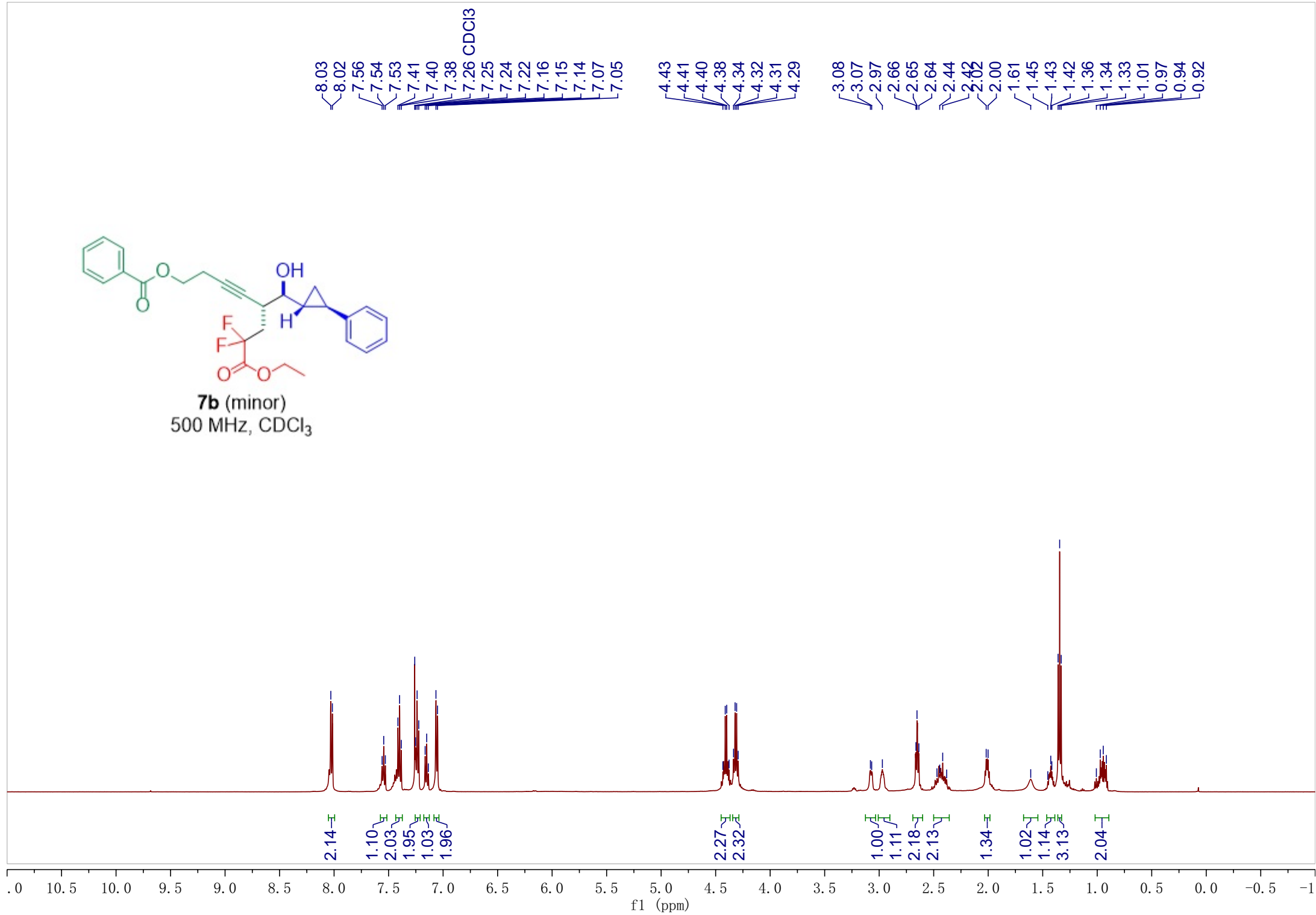
-115

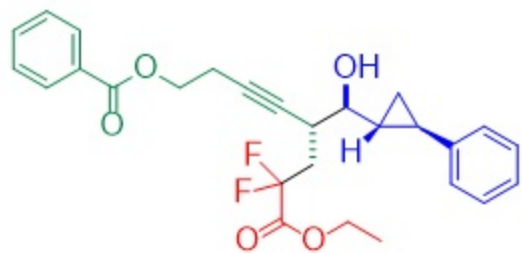
-120

-125

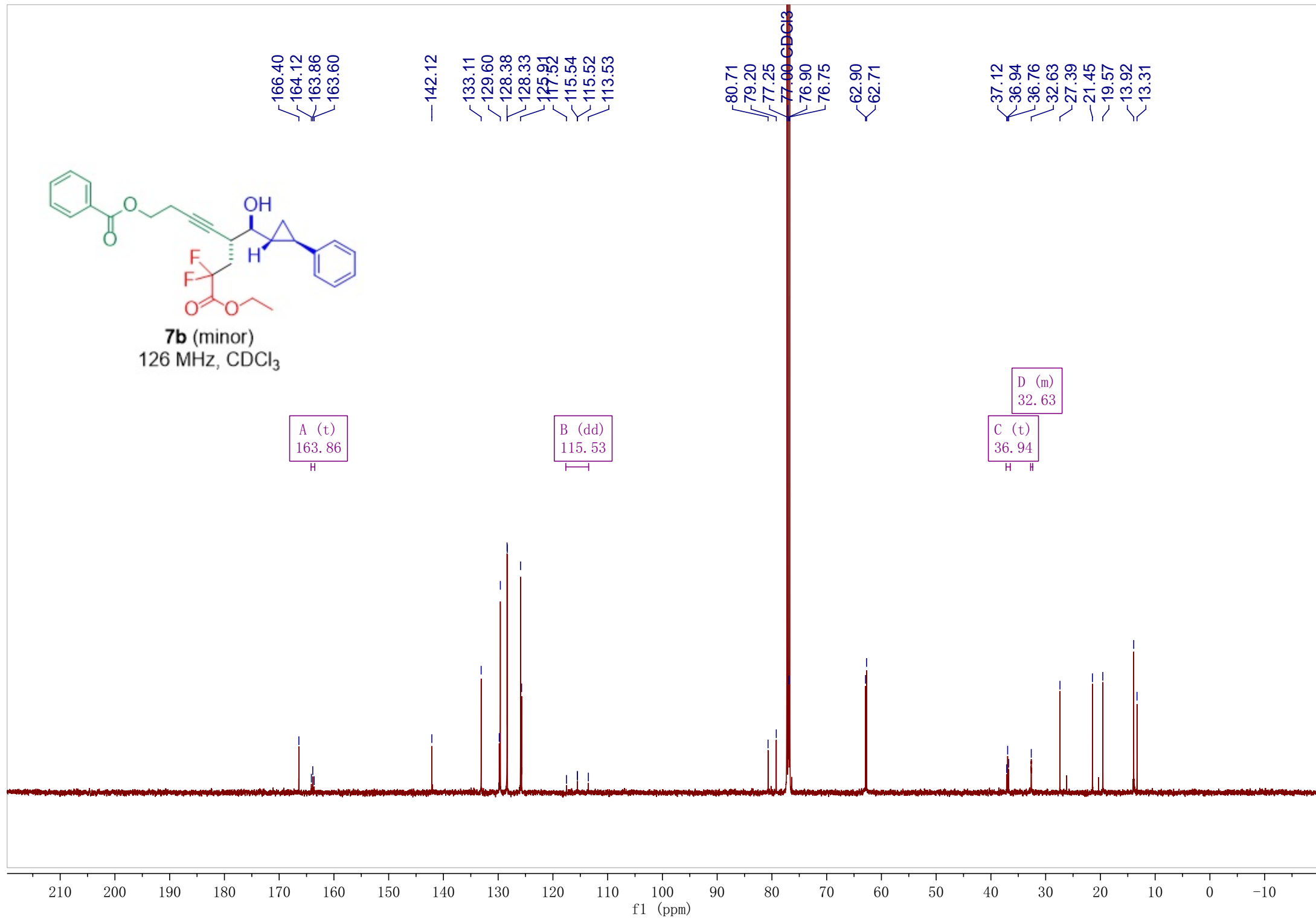


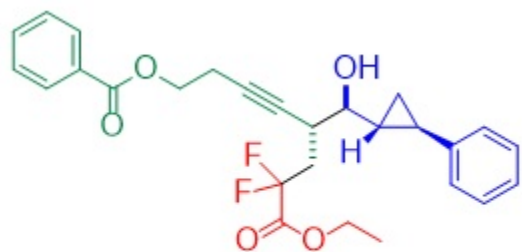
7b (minor)
500 MHz, CDCl₃





7b (minor)
126 MHz, CDCl₃





7b (minor)
471 MHz, CDCl₃

102.75
102.78
102.82
103.31
103.34
103.37
105.93
105.97
106.00
106.48
106.52
106.56

