Supporting Information

Ag_3PO_4 Nanoparticles Enable the Generation of Long-lived Radical Cations for Visible Light-Driven [2+2] and [4+2] Pericyclic Reactions

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S1. Experimental details

Materials. All reagents were of analytical grade and used without further purification unless otherwise stated. $AgNO_3$, Na_2HPO_4, AgCl, AgBr, and AgI were purchased from Energy Organics Co. Ltd. TiO_2 (P25), Bi_2MoO_6 and polyvinyl pyrrolidone (PVP, MW = 58,000) were purchased from Sigma-Aldrich Pharmaceutical Group Co. Ltd. If the solid raw chemicals agglomerated (such as AgCl), they were carefully ground into powders before use. Ammonium hydroxide, ascorbic acid, hydrogen peroxide, 2,6-bis(1,1-dimethylethyl)-4-methylphenol (BHT), and 2,2,6,6-tetramethylpiperidinooxy (TEMPO) were purchased from Macklin Organics Co. Ltd. The commercially available styrenes were purchased from Sigma-Aldrich Pharmaceutical Group Co. Ltd. and used without purification. The anethole derivatives not commercially available were synthesized using the literature methods described in Supporting Information[1-2]. All solvents, such as HFIP, nitromethane, dichloromethane, 2,2,2-trifluoroethanol, acetonitrile, tetrahydrofuran, n-hexane, and ethyl acetate, were dried by distillation with CaH as the desiccant.

Instruments. The emission spectra of all the LEDs and light intensity irradiated on the reaction vial were reported previously.[3] ^1H and ^{13}C NMR spectra were obtained on a Bruker AV-500 spectrometer with $CDCl_3$ as the solvent. High-resolution mass spectra were recorded on an Agilent Q-TOF 6510 instrument. A JEOL JSM-7000F SEM and a JEOL JEM–1011 TEM were used to investigate the morphology of the materials. The UV–vis diffusive reflectance spectra were recorded using the HITACHI U-3900 UV–vis spectrophotometer.

Preparation of Ag_3PO_4. A home-prepared Ag_3PO_4 was chosen for most studies for the following reasons. (1) The synthesis is simple. (2) The recycled and regenerated samples can be easily compared with the as-prepared sample. It was prepared following the literature method.[4-5] Briefly, a 100 mL aqueous solution containing 717 mg of NaH_2PO_4 was added dropwise to a 100 mL aqueous solution containing PVP (800 mg) and $AgNO_3$ (1.70 g). The yellow suspension was heated at 60 °C for 30 min under vigorous stirring. It was then filtered, rinsed with absolute alcohol and distilled water (Ag_3PO_4 is purified in this step), and dried under vacuum overnight to yield the Ag_3PO_4 spheres as a powder. According to our experience, spherical Ag_3PO_4 NPs of similar size could also be synthesized without using PVP. However,

PVP was routinely used in the synthesis. The tetrahedral, cubic, and rhomboid dodecahedral Ag_3PO_4 NPs were prepared following the literature procedures and characterized by SEM.[6]

Laser flash photolysis experiments. A laser flash photolysis system (LP980, Edinburgh Instruments, U.K.) equipped with an Nd: YAG laser (Quanta Ray, Spectra-Physics, U.S.) was used for laser flash photolysis experiments. The third harmonic mode (355 nm, laser duration of 10 ns, ~30 mJ/pulse) and the fourth harmonic mode (266 nm, laser duration of 10 ns, ~10 mJ/pulse) of the laser were employed as the pump source. A xenon lamp (150 W) was used as the probe light. The pulsed laser output was directed onto a quartz cuvette at a right angle to the probe light. All experiments were conducted at room temperature under an air atmosphere, as described in the literature.[7-8] The Ag_3PO_4 suspension was ultrasonicated for 30 min before the experiment. Because Ag_3PO_4 settled very quickly, all operations should be completed as quickly as possible.

DFT calculations. The PBE density functional[9] and PAW basis[10-11] in the Vienna ab initio Simulation Package (VASP)[12] were used for the DFT calculations. The DFT-D3 method with Becke-Jonson damping was used for weak interactions between molecules and substrates.[13-14] A 3×3×3 supercell of Ag_3PO_4 was constructed, and the lattice parameter of one direction was set to 35 Å for the adsorption of molecules.[6] The distance between image molecules in such a two-dimensional structure was set in the 10-15 Å to avoid possible interactions between them. Only Γ point was used for such a large supercell. Both PO_4^{3-}-terminated and Ag^+-terminated (100) facets, with various initial configurations of the adsorbed molecules on the facets, were used in the calculations. During geometric optimization, the bottom 2/3 of the two-dimensional supercell was kept fixed while the other atoms were relaxed. Several optimized structures are shown in Figures S9-S10.

General procedure for the photocatalytic reactions. The reactions were carried out in 10-mL Pyrex vials. Olefin and Ag_3PO_4 were dispersed in a solvent in the vial, which was then purged for 10 min with high-purity N_2 (99.999%). The vial was immersed in a cryogenic reaction bath to maintain the reaction temperature. The reaction suspension was stirred in the dark for 30 min to achieve adsorption-desorption equilibrium. The mixture was then exposed to the lamp from the side. To monitor the reaction progress, a syringe was used to withdraw 10 μL of the solution for analysis by thin-layer chromatography (TLC).

After the reaction, the suspension was centrifuged to separate the solid catalyst from the solution. To reuse Ag_3PO_4, the paste was rinsed with absolute alcohol and distilled water before being vacuum-dried overnight. On the other hand, the solution was dried to give a residue by rotary evaporation. The residue was either dissolved in a certain amount of $CDCl_3$ for ^1H NMR analysis (an internal standard was added at this stage) or purified with column chromatography to afford the desired pure products. The yields of the model reactions are the average values of three parallel experiments. The following are the details for several types of reactions.

Details for the homo-dimerization of styrenes. To a solution of styrene **1** (1.0 mmol) in 3.0 mL of HFIP, Ag_3PO_4 (12 mol%) was added in one portion. The remaining steps are the same as the general procedure.

Details for the cross-dimerization of styrenes. To a solution of styrene **3** (1.0 mmol, 2.0 eq) in 2.0 mL of HFIP was added Ag_3PO_4 (20 mol%). Then, the reaction mixture was degassed by purging with high-purity nitrogen for 10 min. An ice bath was used to maintain the reaction temperature. The mixture was stirred in the dark at 0 °C for half an hour to achieve adsorption-desorption equilibrium. The photocatalytic reaction was then initiated by irradiating the dispersion from the side with the LED lamps. During the reaction, a solution of styrene **1** (0.5 mmol) in 2.0 mL HFIP was added using a syringe pump (at a rate of 4.0 mL/h). The remaining steps are the same as the general procedure.

Details for the intramolecular [2+2] reactions. To a solution of styrene **5** (0.3 mmol) in 2.0 mL of HFIP, Ag_3PO_4 (10 mol%) was added in one portion. The remaining steps are the same as the general procedure.

Details for the Diels–Alder cycloadditions. To a solution of the diene **7** (2.0 mmol, 2.0 eq) in 2.0 mL of HFIP was added Ag_3PO_4 (8 mol%). Then, the reaction mixture was degassed by purging with high-purity nitrogen for 10 min. An ice bath was used to maintain the reaction temperature. The mixture was then stirred in the dark at 0 °C for half an hour to achieve adsorption-desorption equilibrium. The photocatalytic reaction was initiated by irradiating the dispersion from the side with an LED lamp. During the reaction, a solution of styrene **1** (1.0 mmol) in 2.0 mL of HFIP was added using a syringe pump (at a rate of 4.0 mL/h). The remaining steps are the same as the general procedure.

Details for the apparent quantum efficiency (AQY) calculation:

The AQY can be defined using the following equation:

$$AQY = \frac{N_e}{N_p} \times 100\% = \frac{10^9 \times (rate \times N_A \times K) \times (h \times c)}{I \times A \times \lambda}$$

Where *Ne* is the number of electrons calculated according to the quantity of **2a**; *Np* is the total number of the incident photon. To get reliable data about AQY, we assume that all UV photons reaching the solution were absorbed. *Rate* is the reaction rate (mol s^{-1}); N_A is the Avogadro constant (6.022×10^{23} mol^{-1}); *K* is the electron number of the reaction; *h* is the Planck constant (6.626×10^{-34} J·s); *c* is the speed of light (3×10^8 m s^{-1}); *I* is the intensity of irradiation light; *A* is the irradiation area (6.28 cm^2); λ is the wavelength of the monochromatic light (420 nm).

The AQY is then calculated using the following equation.

$$AQY = \frac{1.2 \times 10^8 \times rate}{0.12 \times 6.28 \times 420}$$

S2. Additional results and discussion

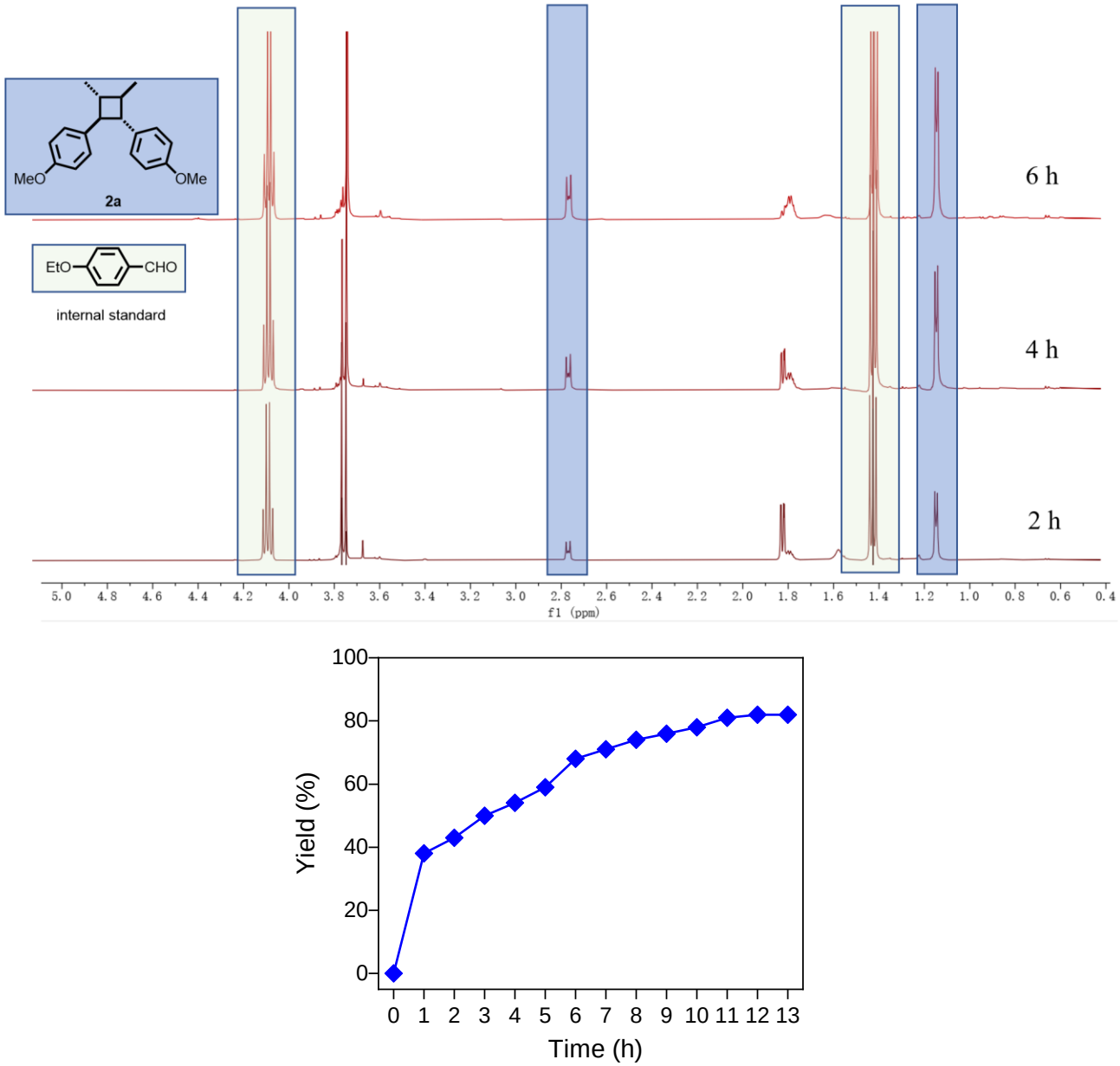
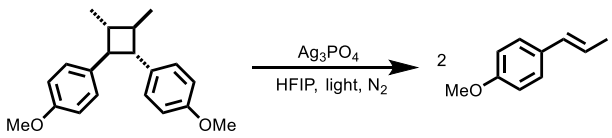


Figure S1. Time-resolved ^{1}H NMR spectra and kinetics of **2a** formation during anethole's [2+2] cycloaddition reaction.



0 h, 90 mg in 3 mL — 0

12 h, 82% left — 18%

Figure S2. The decomposition of **2a** by Ag_3PO_4 photocatalysis.

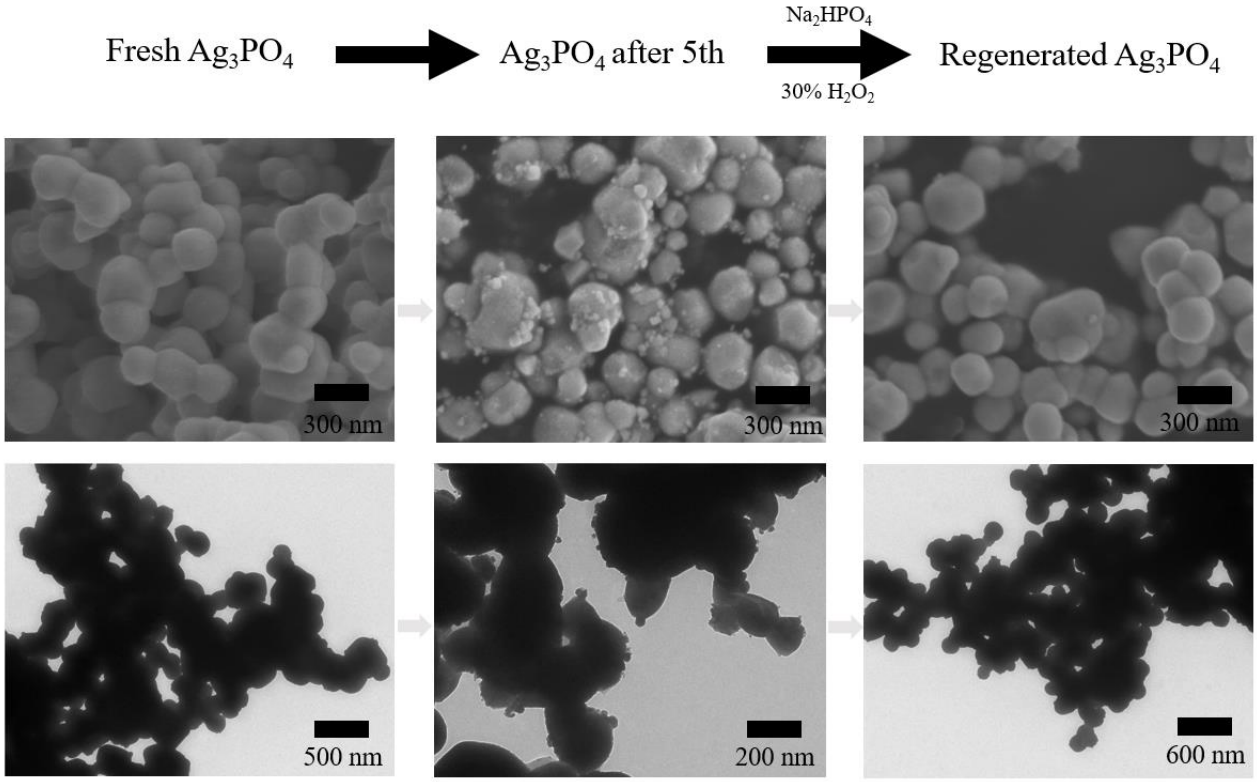


Figure S3. The TEM and SEM images of fresh, recycled, and regenerated Ag_3PO_4.

Figure S4. The color change of TiO_2 before and after irradiation.

A discussion on the rate-limiting step of the [2+2] cycloaddition by Ag_3PO_4 photocatalysis.

First of all, assume substrate incoming (step I in Figure 6 of the text) is the rate-limiting step, then the overall rate should be equal to the rate of adsorption, namely,

$$\text{overall rate} = k_{ads} \bullet [\mathbf{1a}] \bullet (1-\theta) = k_{ads} \bullet [\mathbf{1a}] \bullet (1-\Gamma / \Gamma_m)$$

Next, assume the 1e-oxidation of **1a** (step III) is the rate-limiting step, then the overall rate should be determined by the amounts of both h^+ and adsorbed **1a**, namely,

$$\text{overall rate} = k_{\text{III}} \bullet [h^+] \bullet \Gamma$$

The above assumptions could be ruled out by the linear correlation of AQY with the square of the adsorbed amount of **1a** (recall eq 2 in the text)**.**

Finally, if the 1e-reduction of $\mathbf{2a}^{\bullet+}$ (step V) is the rate-limiting step, the overall rate should be determined by the amounts of both e_{CB}^- and $\mathbf{2a}^{\bullet+}$, namely,

$$\text{overall rate} = k_{\text{V}} \bullet [e_{\text{CB}}^-] \bullet [\mathbf{2a}^{\bullet+}]$$

Both the amounts of e_{CB}^- and $\mathbf{2a}^{\bullet+}$ should not have direct connections with Γ. Therefore, step V should not be the rate-limiting step.

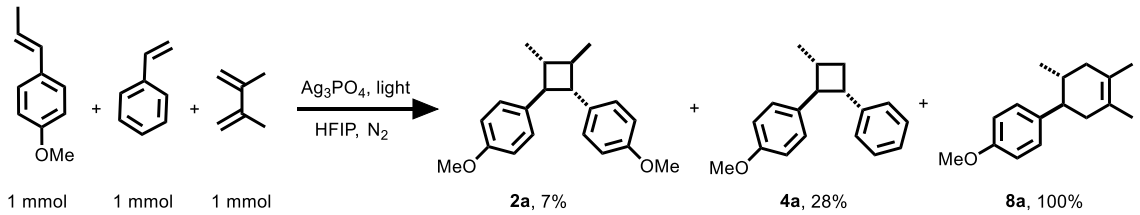


Figure S5. Comparison of the reaction rates.

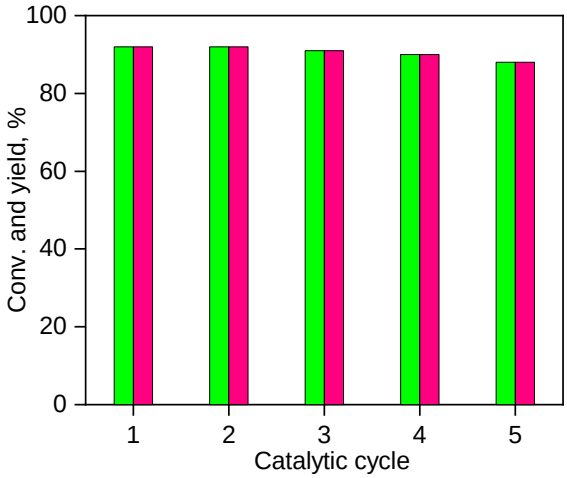


Figure S6. Reuse of Ag_3PO_4 in the Diels-Alder cycloaddition.

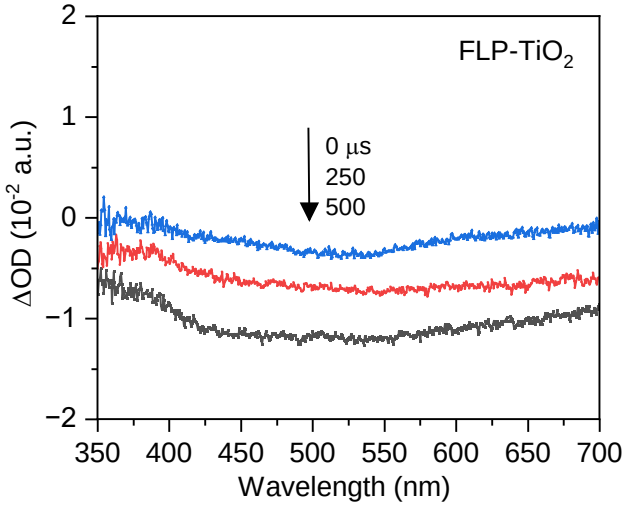


Figure S7. Transient spectra (transmission mode) of the **1a**/TiO_2/HFIP system after 355 nm excitation.

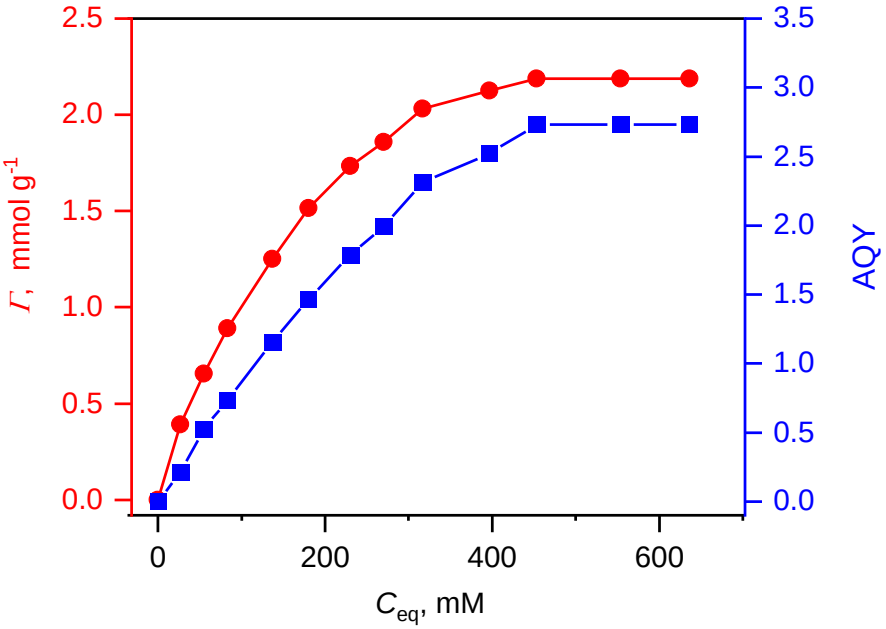


Figure S8. The AQY as a function of substrate concentration.

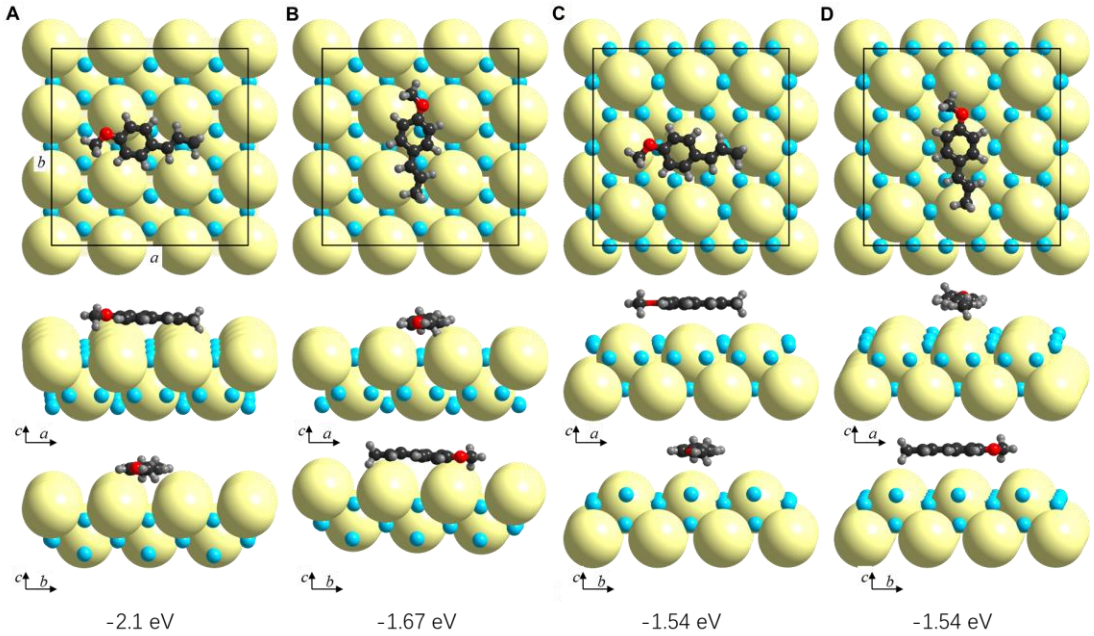


Figure S9. The adsorption energy of **1a** from vacuum to Ag_3PO_4: (**A**) and (**B**), PO_4^{3-}-terminated surfaces; (**C**) and (**D**), Ag^+-terminated surfaces. The optimum configuration of **1a** is **A**, which is reported as Figure 5 in the main text.

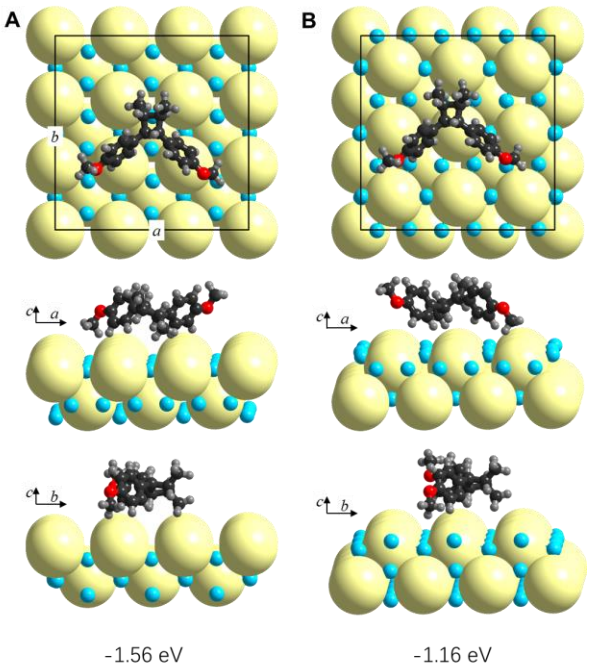


Figure S10. The adsorption energy of **2a** from vacuum to Ag_3PO_4: (**A**) PO_4^{3-}-terminated surface and (**B**) Ag^+-terminated surface. The optimum configuration of **2a** is **A**, as shown in Figure 5 in the main text.

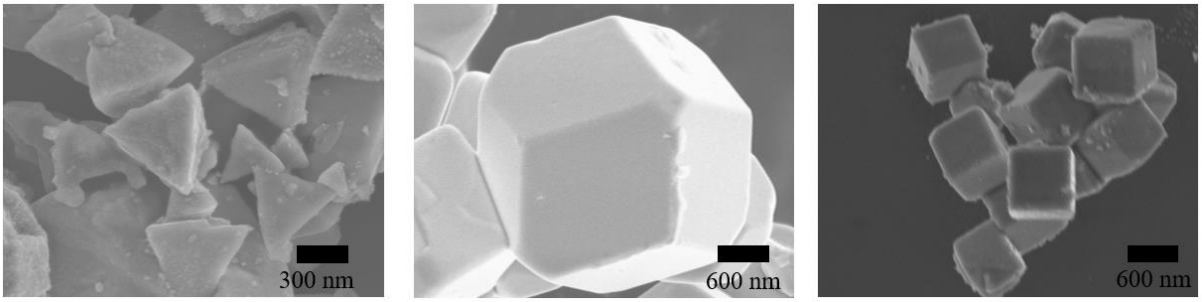
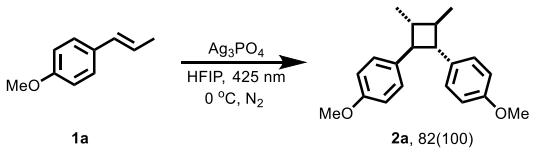


Figure S11. SEM images of the various faceted Ag_3PO_4 samples. The BET area values of all the samples are ranging 1-2 $m^2\ g^{-1}$ with uncertainties of ca. 0.5 $m^2\ g^{-1}$.

Table S1. Screening of reaction conditions.

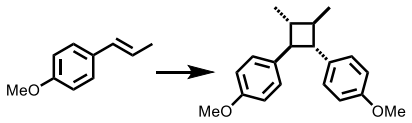


1a **2a**, 82(100)

entry	change from standard conditions	conversion	yield (%)
1	No change	82	82
2	No light, 80 °C	0	n.p.
3	No catalyst	0	n.p.
4	Air	100	13
5	1 eq Vatamin C	0	0
6	1 eq TEMPO	0	0
7	1 eq BHT	0	0

Standard condition: substrate, 1 mmol; photocatalyst, 12 mol%; solvent, 3.0 mL; LED (425 nm); 1 atm N_2; 0 °C; 12 h. The yield was determined by ^{1}H NMR analysis using 4-ethoxybenzaldehyde as an internal standard. From entry 5, we can conclude that holes are indispensable in this reaction. The results of entries 6 and 7 are consistent with the radical cation mechanism.

Table S2. Comparison of the performance of Ag_3PO_4 with various catalysts in the cycloadditions.



1a 2a

catalyst	yield	conditions	catalyst reuse	gram-scale reaction	ref
Ag_3PO_4	1.0 mmol, 82%	12 mol%, 425 nm LED, HFIP, 0 °C	5 runs, > 64%	12.0 g, 80%	this study
triarylpyrylium-based polymer	0.1 mmol, 66%	2 mol% PC, 427 nm Kessil lamp, MeCN, r.t.	no	no	*ACS Catal.* **2020**, *10*, 13251.
cross-linked poly(benzothiadiazole) network	0.24 mmol, 80%	1 mg/mL PC, 460 nm LED, $MeNO_2$, r.t.	no	no	*ACS Catal.* **2017**, *7*, 3097.
2,4,6-tris(4-methoxyphenyl)pyrylium tetrafluoroborate	0.7 mmol, 54%	3 mol% PC, 450 nm LED. 50 mol% naphthalene as electron relays, MeCN, r.t.	no	no	*Chem. Sci.* **2013**, *4*, 2625.
$Ru(bpm)_3(BAr^F)_2$	1.38 mmol, 81%	0.5 mol% PC, fluorescent lightbulb, DCM, 0 °C	no	no	*Chem. Sci.* **2012**, *3*, 2807.
Cage-quinone	0.006 mmol, 34% in the NMR tube	5 mol% catalyst,CD_2Cl_2, r.t.	no	no	*J. Am. Chem. Soc.* **2020**, *142*, 2134.
phenyliodine diacetate	0.2 mmol, 76%	5 mol% catalyst, HFIPA, r.t.	no	no	*Angew. Chem., Int. Ed.* **2016**, *55*, 4748.
$Fe(ClO_4)_3$	1.0 mmol, 68%	10 mol% catalyst, MeCN 30°C	no	no	*Green Chem.* **2018**, *20*, 1743.
fluorenone	0.5 mmol, 76%	3 mol% PC, blue LED, $MeNO_2$, r.t.	no	no	*Green Chem.* **2019,** *21*, 1916.

Table S3. Challenging substrates in the [2+2] cycloaddition.

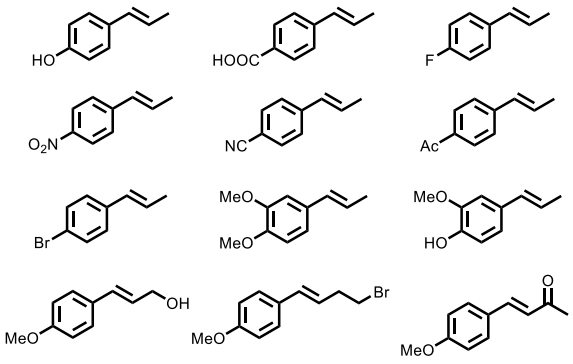
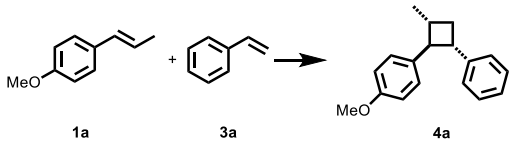


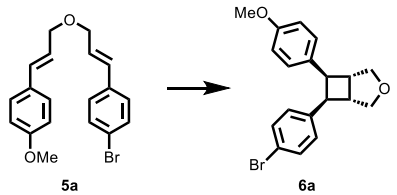
Table S4. Comparison of the performance of Ag_3PO_4 with various catalysts in the cycloadditions.



1a 3a 4a

catalyst	yield	conditions	catalyst reuse	gram-scale reaction	ref
Ag_3PO_4	1.0 mmol, 78%	20 mol%, 425 nm LED, HFIP, 0 °C	yes	no	this study
triarylpyrylium-based polymer	0.1 mmol, 44%	2 mol% PC, 427 nm Kessil lamp, MeCN, r.t.	no	no	*ACS Catal*. **2020**, *10*, 13251.
cross-linked poly(benzothiadiazole) network	0.24 mmol, 85%	1 mg/mL PC, 460 nm LED, $MeNO_2$, r.t.	no	no	*ACS Catal*. **2017,** *7*, 3097.
UCN-coating glass beads	10 mmol, flow chemistry equipment, 82%	1 mg/mL PC, white LED, $MeNO_2$,r.t.	5th, 80%	1.5 g, 82%	*Nat. Commun.* **2020,** *11*, 1239.
$Ru(bpm)_3(BArF)_2$	0.67 mmol, 79%	0.25 mol% PC, fluorescent lightbulb, DCM, -15 °C	no	1.45 g, 81%	*Chem. Sci.* **2012,** *3*, 2807.
Thioxanthylium-based catalysts	0.5 mmol, 99%	1 mol% PC, green LED,$MeNO_2$, r.t.	no	no	*Org. Lett.* **2020,** *22*, 5207.
$Fe(Me_4phen)_3(PF_6)_3$	0.1 mmol, 86%	3 mol% catalyst, DCE/TFE 4:1, r.t.	no	no	*Org. Lett.* **2018,** *20*, 5872.
Cage-Quinone	0.006 mmol, 56% in the NMR tube	5 mol% catalyst, CD_2Cl_2, r.t.	no	no	*J. Am. Chem. Soc.* **2020,** *142*, 2134.
Dess–Martin periodinane	1 mmol, 70%	10 mol% catalyst, HFIPA, r.t.	no	no	*Angew. Chem., Int. Ed.* **2016,** *55*, 4748.
$Fe(ClO_4)_3$	1.0 mmol, 85%	10 mol% catalyst, EtOAc, 40 °C	no	2.16 g, 85%	*Green Chem.* **2018,** *20*, 1743.

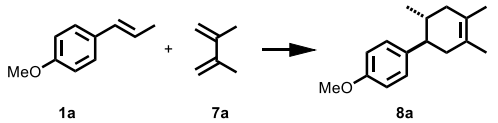
Table S5. Comparison of the performance of Ag_3PO_4 with various catalysts in the cycloadditions.



5a **6a**

catalyst	**yield**	**conditions**	**catalyst reuse**	**gram-scale reaction**	**ref**
Ag_3PO_4	0.3 mmol, 79%	10 mol%, 425 nm LED, HFIP, 0 °C	yes	no	this study
$Ru(bpy)_3(PF_6)_2$	0.32 mmol, 92%	1 mol% PC, 15 mol % $MV(PF_6)_2$, visible light, $MeNO_2$, r.t.	no	1.0 g, 69%	*J. Am. Chem. Soc.* **2010,** *132*, 8572.
phenyliodine diacetate	0.0489 mmol, 75%	10 mol% catalyst, HFIP, r.t.	no	no	*Chem. Commun.* **2019,** *55*, 10316.

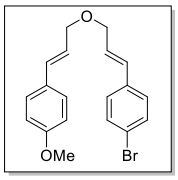
Table S6. Comparison of the performance of Ag_3PO_4 with various catalysts in the cycloadditions.



1a 7a 8a

catalyst	yield	conditions	catalyst reuse	gram-scale reaction	ref
Ag_3PO_4	1.0 mmol, 98%	8 mol%, 425 nm LED, HFIP, 0 °C	5 runs, 90%	41.5 g, 90%	this study
g-C_3N_4	0.5 mmol, 97%	25 mg PC, 30 mg $MgSO_4$, >420 nm LED, $MeNO_2$, r.t.	5 th, 94%	1.0 g, 95%	*Angew. Chem., Int. Ed.* **2017,** *56*, 9336.
TiO_2	0.2 mmol, 97%	100 mg PC, 1 M $LiClO_4$,365 nm LED, $MeNO_2$, r.t.	no	no	*Org. Lett*. **2019,** *21*, 2246-2250.
$Ru(bpz)_3(BArF)_2$	0.68 mmol, 96%	0.5 mol% PC, CFL bulb, DCM, r.t.	no	no	*J. Am. Chem. Soc*. **2011,** *133*, 19350.
$[Cr(Ph_2phen)_3](BF_4)_3$	0.12 mmol, 86%	1 mol% PC, 300-419 nm, $MeNO_2$, r.t.	no	no	*Angew. Chem., Int. Ed.* **2015,** *54*, 6506.
Thioxanthylium-based catalysts	0.5 mmol, 95%	1 mol% PC, green light, $MeNO_2$, r.t.	no	1.5 g, 84%	*Tetrahedron Lett.* **2018,** 59, 3361.
$Fe(Me_4phen)_3(PF_6)_3$	0.1 mmol, 95%	1 mol% catalyst, DCE/TFE 9:1, r.t.	no	no	*Org. Lett*. **2018,** *20*, 5872.
Thioxanthylium-based catalysts	0.5 mmol, 88%	1 mol% PC, green light, $MeNO_2$, r.t.	no	1.5 g, 84%	*J. Org. Chem.* **2022,** *87*, 3319.
$[Cr(Ph_2phen)_3]_3BF_4$	0.12 mmol, 88%	1 mol% PC, 300-419 nm, $MeNO_2$, r.t.	no	no	*J. Am. Chem. Soc.* **2016,** *138*, 5451.
fluorenone	0.5 mmol, 74%	3 mol% PC, blue LED, $MeNO_2$, r.t.	no	no	*Green Chem.* **2019,** *21*, 1916.
$FeCl_3$	0.18 mmol, 98%	3 mol% catalyst, MeCN, r.t.	0	103.5 g, 95%	*J. Am. Chem. Soc.* **2019,** *141*, 1877.
$Fe(ClO_4)_3$	1.0 mmol, 68%	10 mol% catalyst, MeCN, r.t.	0	1.26 g, 97%	*Green Chem.* **2018,** *20*, 1743.
electrochemistry	1.6 mmol, 96%	$MeNO_2$, $LiClO_4$ electrolyte solution	0	0	*Chem. Sci*. **2016,** *7*, 6387.

S3. Analysis data of the substrates and products

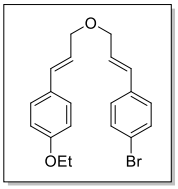


5a

1-bromo-4-((E)-3-(((E)-3-(4-methoxyphenyl)allyl)oxy)prop-1-en-1-yl)benzene (5a)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.4 Hz, 2H), 7.37 – 7.30 (m, 2H), 7.29 – 7.21 (m, 2H), 6.86 (t, *J* = 5.8 Hz, 2H), 6.58 (d, *J* = 15.9 Hz, 2H), 6.31 (dt, *J* = 15.9, 5.9 Hz, 1H), 6.19 (dt, *J* = 15.9, 6.3 Hz, 1H), 4.22 – 4.13 (m, 4H), 3.81 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 159.35, 135.71, 132.46, 131.65, 131.09, 129.41, 127.99, 127.70, 127.00, 123.56, 121.40, 113.98, 71.14, 70.38, 55.28.

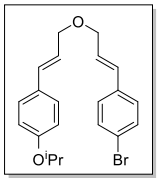


5b

1-bromo-4-((E)-3-(((E)-3-(4-ethoxyphenyl)allyl)oxy)prop-1-en-1-yl)benzene (5b)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.6 Hz, 2H), 7.25 (d, *J* = 8.3 Hz, 2H), 6.84 (d, *J* = 8.6 Hz, 2H), 6.57 (d, *J* = 15.9 Hz, 2H), 6.31 (dt, *J* = 15.9, 5.9 Hz, 1H), 6.18 (dt, *J* = 15.8, 6.3 Hz, 1H), 4.26 – 4.13 (m, 4H), 4.03 (q, *J* = 7.0 Hz, 2H), 1.41 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 158.72, 135.72, 132.52, 131.64, 131.05, 129.25, 127.98, 127.68, 127.02, 123.43, 121.38, 114.53, 71.14, 70.34, 63.44, 14.80.

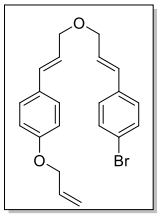


5c

1-bromo-4-((E)-3-(((E)-3-(4-isopropoxyphenyl)allyl)oxy)prop-1-en-1-yl)benzene (5c)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48 – 7.38 (m, 2H), 7.37 – 7.29 (m, 2H), 7.25 (d, *J* = 8.1 Hz, 2H), 6.89 – 6.77 (m, 2H), 6.64 – 6.49 (m, 2H), 6.31 (dt, *J* = 15.9, 5.9 Hz, 1H), 6.17 (dt, *J* = 15.9, 6.3 Hz, 1H), 4.54 (dt, *J* = 12.1, 6.1 Hz, 1H), 4.24 – 4.10 (m, 4H), 1.56 (s, 1H), 1.33 (d, *J* = 6.1 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.70, 135.73, 132.56, 131.65, 131.06, 129.19, 127.99, 127.71, 127.04, 123.42, 121.39, 115.89, 71.16, 70.33, 69.92, 22.03.

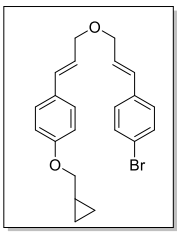


5d

1-(allyloxy)-4-((E)-3-(((E)-3-(4-bromophenyl)allyl)oxy)prop-1-en-1-yl)benzene (5d)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48 – 7.40 (m, 2H), 7.37 – 7.29 (m, 2H), 7.28 – 7.22 (m, 3H), 6.94 – 6.77 (m, 2H), 6.57 (d, *J* = 15.9 Hz, 2H), 6.31 (dt, *J* = 15.9, 5.9 Hz, 1H), 6.19 (dt, *J* = 15.9, 6.3 Hz, 1H), 6.05 (ddd, *J* = 11.9, 10.5, 5.3 Hz, 1H), 5.41 (dq, *J* = 17.3, 1.6 Hz, 1H), 5.29 (dq, *J* = 10.5, 1.4 Hz, 1H), 4.54 (dt, *J* = 5.3, 1.5 Hz, 2H), 4.24 – 4.11 (m, 4H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 158.33, 135.69, 133.15, 132.43, 131.64, 131.09, 129.54, 127.98, 127.67, 126.97, 123.62, 121.40, 117.73, 114.78, 71.12, 70.37, 68.80.

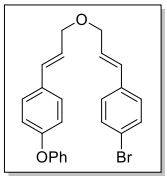


5e

1-bromo-4-((E)-3-(((E)-3-(4-(cyclopropylmethoxy)phenyl)allyl)oxy)prop-1-en-1-yl)benzene (5e)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.7 Hz, 2H), 7.25 (d, *J* = 8.0 Hz, 3H), 6.85 (d, *J* = 8.7 Hz, 2H), 6.57 (d, *J* = 15.7 Hz, 2H), 6.32 (dd, *J* = 13.9, 8.0 Hz, 1H), 6.25 – 6.12 (m, 1H), 4.18 (d, *J* = 6.1 Hz, 4H), 3.80 (d, *J* = 6.9 Hz, 2H), 1.27 (ddd, *J* = 11.9, 7.5, 5.2 Hz, 1H), 0.73 – 0.58 (m, 2H), 0.35 (q, *J* = 4.8 Hz, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 158.79, 135.70, 132.52, 131.65, 131.09, 129.30, 127.99, 127.68, 126.98, 123.44, 121.40, 114.63, 72.79, 71.15, 70.37, 10.24, 3.17.

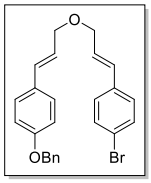


5f

1-bromo-4-((E)-3-(((E)-3-(4-phenoxyphenyl)allyl)oxy)prop-1-en-1-yl)benzene (5f)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 – 7.41 (m, 2H), 7.39 – 7.31 (m, 4H), 7.29 – 7.22 (m, 3H), 7.11 (t, *J* = 7.4 Hz, 1H), 7.04 – 6.99 (m, 2H), 6.98 – 6.92 (m, 2H), 6.65 – 6.53 (m, 2H), 6.36 – 6.29 (m, 1H), 6.24 (dt, *J* = 15.9, 6.1 Hz, 1H), 4.25 – 4.15 (m, 4H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.02, 156.99, 135.68, 131.99, 131.84, 131.67, 131.15, 129.75, 127.99, 127.85, 126.92, 124.90, 123.39, 121.44, 118.98, 118.82, 70.97, 70.48.

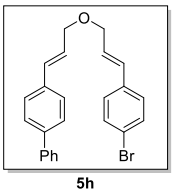


5g

1-(benzyloxy)-4-((E)-3-(((E)-3-(4-bromophenyl)allyl)oxy)prop-1-en-1-yl)benzene (5g)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.3 Hz, 4H), 7.38 (dd, *J* = 8.0, 6.8 Hz, 2H), 7.32 (dd, *J* = 7.4, 5.9 Hz, 3H), 7.29 – 7.21 (m, 2H), 6.93 (dd, *J* = 8.7, 1.7 Hz, 2H), 6.57 (d, *J* = 15.9 Hz, 2H), 6.31 (dtd, *J* = 15.9, 5.9, 1.4 Hz, 1H), 6.25 – 6.13 (m, 1H), 5.06 (s, 2H), 4.24 – 4.12 (m, 4H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 158.55, 136.89, 135.71, 132.39, 131.65, 131.07, 129.67, 128.57, 127.98, 127.97, 127.71, 127.43, 127.00, 123.72, 121.40, 114.95, 71.11, 70.37, 70.03.

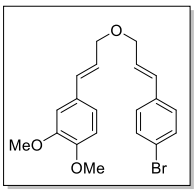


5h

4-((E)-3-(((E)-3-(4-bromophenyl)allyl)oxy)prop-1-en-1-yl)-1,1'-biphenyl (5h)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 (dd, *J* = 16.6, 7.8 Hz, 4H), 7.50 – 7.39 (m, 6H), 7.34 (t, *J* = 7.4 Hz, 1H), 7.26 (d, *J* = 8.2 Hz, 2H), 6.68 (d, *J* = 15.9 Hz, 1H), 6.59 (d, *J* = 16.0 Hz, 1H), 6.34 (ddt, *J* = 19.8, 15.9, 6.0 Hz, 2H), 4.28 – 4.11 (m, 4H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 140.64, 140.50, 135.68, 132.21, 131.67, 131.19, 128.77, 128.00, 127.32, 127.25, 126.92, 125.99, 121.45, 70.94, 70.54.

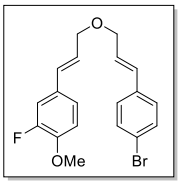


5i

4-((E)-3-(((E)-3-(4-bromophenyl)allyl)oxy)prop-1-en-1-yl)-1,2-dimethoxybenzene (5i)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 – 7.37 (m, 2H), 7.29 – 7.17 (m, 2H), 6.94 (dd, *J* = 17.9, 4.7 Hz, 2H), 6.85 – 6.76 (m, 1H), 6.57 (d, *J* = 15.9 Hz, 2H), 6.40 – 6.26 (m, 1H), 6.19 (dt, *J* = 15.8, 6.2 Hz, 1H), 4.18 (dd, *J* = 5.8, 4.6 Hz, 4H), 3.95 – 3.81 (m, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 149.08, 149.01, 135.72, 132.64, 131.68, 131.09, 129.77, 128.02, 127.02, 123.92, 121.44, 119.81, 111.17, 108.93, 71.09, 70.49, 55.93, 55.83.

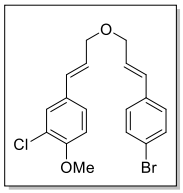


5j

4-((E)-3-(((E)-3-(4-bromophenyl)allyl)oxy)prop-1-en-1-yl)-2-fluoro-1-methoxybenzene (5j)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48 – 7.40 (m, 2H), 7.26 (t, *J* = 4.1 Hz, 3H), 7.15 (dd, *J* = 12.5, 2.0 Hz, 1H), 7.07 (d, *J* = 8.5 Hz, 1H), 6.90 (t, *J* = 8.6 Hz, 1H), 6.56 (dd, *J* = 21.3, 16.0 Hz, 2H), 6.31 (dt, *J* = 15.9, 5.9 Hz, 1H), 6.18 (dt, *J* = 15.9, 6.1 Hz, 1H), 4.18 (d, *J* = 6.0 Hz, 4H), 3.89 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 147.34, 135.66, 131.68, 131.29, 131.21, 128.00, 126.86, 125.09, 122.78, 121.47, 113.66, 113.51, 113.29, 70.79, 70.58, 56.30.

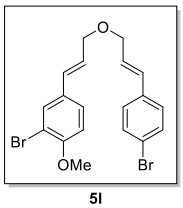


5k

4-((E)-3-(((E)-3-(4-bromophenyl)allyl)oxy)prop-1-en-1-yl)-2-chloro-1-methoxybenzene (5k)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 – 7.39 (m, 3H), 7.23 (dd, *J* = 13.7, 5.2 Hz, 3H), 6.87 (d, *J* = 8.5 Hz, 1H), 6.54 (dd, *J* = 26.7, 15.9 Hz, 2H), 6.30 (dt, *J* = 15.9, 5.9 Hz, 1H), 6.19 (dt, *J* = 15.9, 6.0 Hz, 1H), 4.22 – 4.14 (m, 4H), 3.89 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 154.57, 135.69, 131.69, 131.19, 130.96, 130.45, 128.08, 128.02, 126.89, 126.03, 125.16, 122.70, 121.48, 112.03, 70.82, 70.58, 56.21.

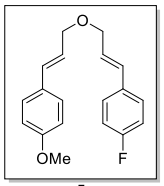


5l

2-bromo-4-((E)-3-(((E)-3-(4-bromophenyl)allyl)oxy)prop-1-en-1-yl)-1-methoxybenzene (5l)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.60 (d, *J* = 2.1 Hz, 1H), 7.48 – 7.37 (m, 2H), 7.33 – 7.21 (m, 4H), 6.85 (d, *J* = 8.5 Hz, 1H), 6.55 (dd, *J* = 29.2, 15.9 Hz, 2H), 6.31 (dt, *J* = 15.9, 5.9 Hz, 1H), 6.19 (dt, *J* = 15.9, 6.1 Hz, 1H), 4.18 (dd, *J* = 6.0, 1.4 Hz, 4H), 3.89 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 155.45, 135.68, 131.69, 131.20, 130.93, 130.83, 128.02, 126.88, 126.74, 125.18, 121.48, 111.94, 111.85, 70.81, 70.57, 56.31.

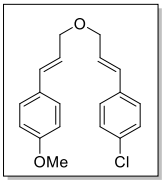


5m

1-fluoro-4-((E)-3-(((E)-3-(4-methoxyphenyl)allyl)oxy)prop-1-en-1-yl)benzene (5m)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 (ddt, *J* = 9.5, 7.1, 2.5 Hz, 4H), 7.03 – 6.95 (m, 2H), 6.89 – 6.81 (m, 2H), 6.58 (dd, *J* = 15.9, 6.7 Hz, 2H), 6.24 (dd, *J* = 14.1, 8.1 Hz, 1H), 6.20 – 6.13 (m, 1H), 4.20 – 4.14 (m, 4H), 3.78 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 163.37, 161.41, 159.37, 132.97, 132.95, 132.44, 131.32, 129.48, 128.07, 128.00, 127.75, 125.90, 125.89, 123.67, 115.58, 115.41, 114.03, 71.08, 70.55, 55.28.

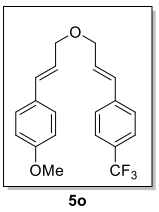


5n

1-chloro-4-((E)-3-(((E)-3-(4-methoxyphenyl)allyl)oxy)prop-1-en-1-yl)benzene (5n)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 – 7.23 (m, 6H), 6.92 – 6.82 (m, 2H), 6.58 (dd, *J* = 15.9, 5.6 Hz, 2H), 6.30 (dt, *J* = 15.9, 5.9 Hz, 1H), 6.19 (dt, *J* = 15.9, 6.3 Hz, 1H), 4.18 (d, *J* = 5.9 Hz, 4H), 3.81 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 159.36, 135.28, 133.28, 132.49, 131.11, 129.43, 128.73, 127.73, 127.70, 126.85, 123.58, 114.00, 71.16, 70.43, 55.31.

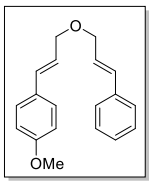


5o

1-methoxy-4-((E)-3-(((E)-3-(4-(trifluoromethyl)phenyl)allyl)oxy)prop-1-en-1-yl)benzene (5o)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.2 Hz, 2H), 7.48 (d, *J* = 8.2 Hz, 2H), 7.38 – 7.29 (m, 2H), 6.90 – 6.81 (m, 2H), 6.63 (dd, *J* = 44.0, 15.9 Hz, 2H), 6.42 (dt, *J* = 16.0, 5.7 Hz, 1H), 6.20 (dt, *J* = 15.9, 6.3 Hz, 1H), 4.21 (ddd, *J* = 9.5, 6.0, 1.3 Hz, 4H), 3.81 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 159.39, 140.27, 132.60, 130.65, 129.55, 129.37, 129.29, 128.98, 127.74, 126.61, 125.55, 125.52, 125.28, 123.45, 114.01, 71.31, 70.21, 55.30.

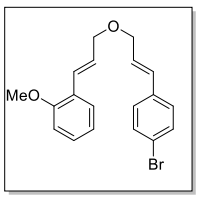


5p

1-((E)-3-(cinnamyloxy)prop-1-en-1-yl)-4-methoxybenzene(5p)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 (dd, *J* = 12.2, 4.8 Hz, 2H), 7.32 (dt, *J* = 13.7, 5.3 Hz, 4H), 7.23 (t, *J* = 7.3 Hz, 1H), 6.91 – 6.78 (m, 2H), 6.60 (dd, *J* = 27.7, 15.9 Hz, 2H), 6.32 (dt, *J* = 15.9, 6.1 Hz, 1H), 6.19 (dt, *J* = 15.9, 6.2 Hz, 1H), 4.23 – 4.16 (m, 4H), 3.79 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 159.35, 136.78, 132.56, 132.41, 129.52, 128.63, 128.60, 127.75, 127.71, 126.54, 126.50, 126.14, 123.74, 114.02, 71.00, 70.66, 55.31.

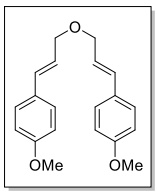


5q

1-((E)-3-(((E)-3-(4-bromophenyl)allyl)oxy)prop-1-en-1-yl)-2-methoxybenzene (5q)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 – 7.39 (m, 3H), 7.30 – 7.20 (m, 4H), 6.94 (dd, *J* = 18.1, 11.2 Hz, 2H), 6.87 (d, *J* = 8.2 Hz, 1H), 6.58 (d, *J* = 16.0 Hz, 1H), 6.33 (ddt, *J* = 15.9, 7.2, 6.1 Hz, 2H), 4.20 (ddd, *J* = 15.5, 6.1, 1.3 Hz, 4H), 3.85 (s, 3H).

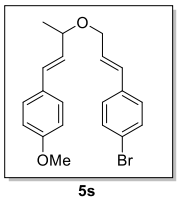
^{13}C NMR (126 MHz, $CDCl_3$) δ 156.78, 135.77, 131.67, 131.10, 128.82, 128.02, 127.86, 127.09, 127.05, 126.49, 125.66, 121.39, 120.66, 110.84, 71.51, 70.36, 55.45.



5r

4,4'-((1E,1'E)-oxybis(prop-1-ene-3,1-diyl))bis(methoxybenzene) (5r)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 – 7.26 (m, 4H), 6.95 – 6.76 (m, 4H), 6.57 (d, *J* = 15.9 Hz, 2H), 6.19 (dt, *J* = 15.9, 6.2 Hz, 2H), 4.17 (dd, *J* = 6.2, 1.3 Hz, 4H), 3.79 (s, 6H).

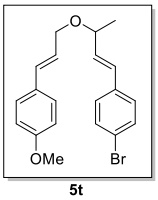


5s

1-bromo-4-((E)-3-(((E)-4-(4-methoxyphenyl)but-3-en-2-yl)oxy)prop-1-en-1-yl)benzene (5s)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 – 7.39 (m, 2H), 7.36 – 7.30 (m, 2H), 7.27 – 7.22 (m, 2H), 6.90 – 6.82 (m, 2H), 6.58 – 6.45 (m, 2H), 6.35 – 6.24 (m, 1H), 5.99 (dd, *J* = 15.9, 7.9 Hz, 1H), 4.20 – 4.16 (m, 1H), 4.09 – 4.02 (m, 2H), 3.79 (d, *J* = 7.9 Hz, 3H), 1.37 (d, *J* = 6.3 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 159.37, 135.87, 131.72, 131.69, 131.64, 131.29, 131.07, 130.70, 129.34, 129.30, 128.04, 128.01, 127.70, 127.51, 126.78, 121.31, 114.05, 76.45, 70.73, 68.50, 55.34, 21.86.

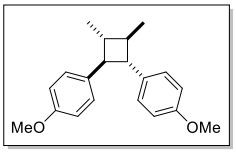


5t

1-bromo-4-((E)-3-(((E)-3-(4-methoxyphenyl)allyl)oxy)but-1-en-1-yl)benzene (5t)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.4 Hz, 2H), 7.28 (dd, *J* = 31.8, 8.6 Hz, 4H), 6.84 (d, *J* = 8.7 Hz, 2H), 6.50 (dd, *J* = 29.9, 15.9 Hz, 2H), 6.22 – 6.08 (m, 2H), 4.24 – 4.16 (m, 1H), 4.13 – 4.00 (m, 2H), 3.79 (d, *J* = 3.1 Hz, 3H), 1.36 (d, *J* = 6.4 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 159.27, 135.64, 132.64, 132.01, 131.70, 129.97, 129.56, 128.27, 128.02, 127.69, 124.04, 121.41, 113.96, 113.90, 75.65, 69.12, 55.29, 21.60.



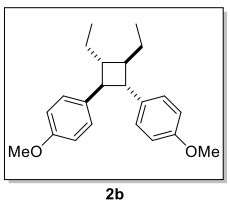
2a

(±)-4,4'-((*1S,2S,3R,4R*)-3,4-dimethylcyclobutane-1,2-diyl)bis(methoxybenzene) (2a)

Following the general procedure A; 82% yield; dr:>19:1; White Oil; R_f = 0.4 (*n*-hexane : EA = 20:1);

^{1}H NMR (500 MHz, $CDCl_3$) 7.12 (d, *J* = 8.7 Hz, 4H), 6.82 (d, *J* = 8.7 Hz, 4H), 3.76 (s, 6H), 2.84 – 2.76 (m, 2H), 1.82 (m, 2H), 1.18 (d, *J* = 6.1 Hz, 6H).^{13}C NMR (126 MHz, $CDCl_3$) δ 157.93, 135.90, 127.70, 113.68, 55.20, 52.47, 43.19, 18.85.

HRMS (ESI) m/z calcd for $[C_{20}H_{25}O_2]^+$ $[M+H]^+$: 297.1849, found 297.1852.



2b

(±)-4,4'-((*1S,2S,3R,4R*)-3,4-diethylcyclobutane-1,2-diyl)bis(methoxybenzene) (2b)

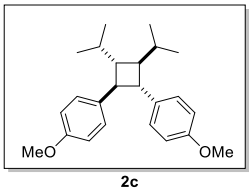
Following the general procedure A; 83% yield; dr:>19:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 20:1);

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.14 (d, *J* = 8.6 Hz, 4H), 6.82 (d, *J* = 8.6 Hz, 4H), 3.77 (s, 6H), 2.80 (dd, *J* = 5.7, 3.2 Hz, 2H), 1.98 – 1.87 (m, 2H), 1.72 – 1.53 (m, 4H), 0.87 (t, *J* = 7.5 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.75, 136.47, 127.87, 113.52, 55.13, 51.24,

47.26, 28.53, 11.87.

HRMS (ESI) m/z calcd for $[C_{22}H_{29}O_2]^+$ $[M+H]^+$: 325.2162, found 325.2163.



2c

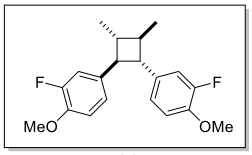
(±)-4,4'-((*1S,2S,3R,4R*)-3,4-diisopropylcyclobutane-1,2-diyl)bis(methoxybenzene) (2c)

Following the general procedure A; 42% yield; dr:>19:1; White Oil; R_f = 0.2 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.10 (d, *J* = 8.7 Hz, 4H), 6.80 (d, *J* = 8.7 Hz, 4H), 3.77 (s, 6H), 2.78 (d, *J* = 9.0 Hz, 2H), 2.05 – 1.98 (m, 2H), 1.82 (dd, *J* = 13.3, 6.7 Hz, 2H), 0.87 (dd, *J* = 8.7, 6.8 Hz, 12H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.71, 137.12, 128.13, 113.52, 55.16, 49.35, 49.18, 32.98, 21.19, 19.70.

HRMS (ESI) m/z calcd for $[C_{24}H_{33}O_2]^+$ $[M+H]^+$: 353.2475, found 353.2481.



2d

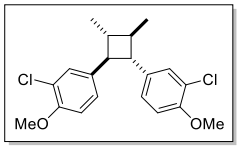
(±)-4,4'-((*1S,2S,3R,4R*)-3,4-dimethylcyclobutane-1,2-diyl)bis(2-fluoro-1-methoxybenzene) (2d)

Following the general procedure A; 54% yield; dr:>19:1; White Oil; R_f = 0.4 (*n*-hexane : EA = 20:1);

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.93 (d, *J* = 12.8 Hz, 2H), 6.87 (d, *J* = 4.5 Hz, 4H), 3.85 (s, 6H), 2.81 – 2.67 (m, 2H), 1.81 (d, *J* = 4.9 Hz, 2H), 1.17 (d, *J* = 5.5 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 153.35, 151.39, 145.94, 145.85, 136.58, 136.53, 122.27, 122.24, 114.41, 114.27, 113.37, 56.33, 52.33, 43.10, 18.69.

HRMS (ESI) m/z calcd for $[C_{20}H_{23}F_2O_2]^+$ $[M+H]^+$: 333.1661, found 333.1661.



2e

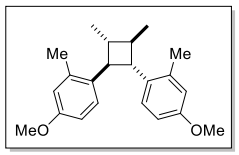
(±)-4,4'-((*1S,2S,3R,4R*)-3,4-dimethylcyclobutane-1,2-diyl)bis(2-chloro-1-methoxybenzene) (2e)

Following the general procedure A; 50% yield; dr:>19:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 20:1);

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.21 (d, *J* = 1.9 Hz, 2H), 7.02 (dd, *J* = 8.4, 1.9 Hz, 2H), 6.84 (d, *J* = 8.4 Hz, 2H), 3.86 (s, 6H), 2.74 (dd, *J* = 5.6, 3.3 Hz, 2H), 1.81 (dd, *J* = 9.8, 4.9 Hz, 2H), 1.17 (d, *J* = 5.7 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 153.37, 136.48, 128.40, 126.03, 122.24, 112.01, 56.15, 52.20, 43.16, 18.68.

HRMS (ESI) m/z calcd for $[C_{20}H_{23}Cl_2O_2]^+$ $[M+H]^+$: 365.1070, found 365.1058.



2f

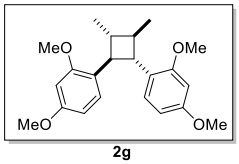
(±)-4,4'-((*1S,2S,3R,4R*)-3,4-dimethylcyclobutane-1,2-diyl)bis(1-methoxy-3-methylbenzene) (2f)

Following the general procedure A; 63% yield; dr:>19:1; White Oil; R_f = 0.2 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 (d, *J* = 8.5 Hz, 2H), 6.74 (dd, *J* = 8.5, 2.6 Hz, 2H), 6.60 (d, *J* = 2.6 Hz, 2H), 3.75 (s, 6H), 3.05 – 2.87 (m, 2H), 2.01 (s, 6H), 1.99 – 1.90 (m, 2H), 1.15 (d, *J* = 5.8 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.46, 137.61, 133.97, 127.06, 115.51, 111.18, 55.15, 50.42, 42.33, 20.40, 18.95.

HRMS (ESI) m/z calcd for $[C_{22}H_{29}O_2]^+$ $[M+H]^+$: 325.2162, found 325.2167.



2g

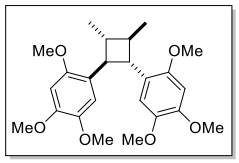
(±)-4,4'-((*1S,2S,3R,4R*)-3,4-dimethylcyclobutane-1,2-diyl)bis(1,3-dimethoxybenzene) (2g)

Following the general procedure A; 51% yield; dr:>19:1; White Oil; R_f = 0.4 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.20 (d, *J* = 8.4 Hz, 2H), 6.43 (dd, *J* = 8.3, 2.4 Hz, 2H), 6.38 (d, *J* = 2.3 Hz, 2H), 3.76 (s, 6H), 3.68 (s, 6H), 3.30 – 3.17 (m, 2H), 1.74 (q, *J* = 5.0 Hz, 2H), 1.16 (d, *J* = 5.9 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 158.73, 158.43, 127.71, 124.96, 103.69, 98.16, 55.25, 55.06, 44.84, 43.41, 19.19.

HRMS (ESI) m/z calcd for $[C_{22}H_{29}O_4]^+$ $[M+H]^+$: 357.2060, found 357.2060.



2h

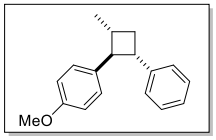
(±)-5,5'-((*1S,2S,3R,4R*)-3,4-dimethylcyclobutane-1,2-diyl)bis(1,2,4-trimethoxybenzene) (2h)

Following the general procedure A; 70% yield; dr:>19:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 6.95 (s, 2H), 6.47 (s, 2H), 3.86 (d, *J* = 12.0 Hz, 12H), 3.69 (s, 6H), 3.27 (d, *J* = 9.0 Hz, 2H), 1.77 (q, *J* = 5.1 Hz, 2H), 1.19 (d, *J* = 5.9 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 151.57, 147.44, 143.01, 123.79, 112.17, 97.77, 56.63, 56.44, 56.08, 45.26, 43.38, 18.98.

HRMS (ESI) m/z calcd for $[C_{24}H_{33}O_6]^+$ $[M+H]^+$: 417.2272, found 417.2275.



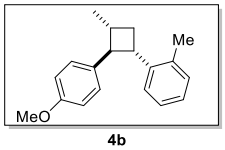
4a

(±)-1-methoxy-4-((*1S,2R,4S*)-2-methyl-4-phenylcyclobutyl)benzene (4a)

Following the general procedure B; 78% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.37 (t, *J* = 7.5 Hz, 2H), 7.27 (q, *J* = 7.9, 6.8 Hz, 5H), 6.95 (d, *J* = 8.6 Hz, 2H), 3.88 (s, 3H), 3.49 (q, *J* = 9.9 Hz, 1H), 3.05 (t, *J* = 9.5 Hz, 1H), 2.68 – 2.56 (m, 1H), 2.43 (dtd, *J* = 16.5, 9.5, 8.3, 5.1 Hz, 1H), 1.80 (q, *J* = 10.1 Hz, 1H), 1.29 (d, *J* = 6.5 Hz, 3H).

HRMS (ESI) m/z calcd for $[C_{18}H_{21}O]^+$ $[M+H]^+$: 253.1587, found 253.1590.



4b

(±)-1-((*1S,2S,3R*)-2-(4-methoxyphenyl)-3-methylcyclobutyl)-2-methylbenzene (4b)

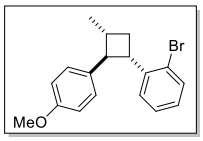
Following the general procedure B; 76% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.29 (d, *J* = 7.7 Hz, 1H), 7.20 – 7.11 (m, 3H), 7.09 – 7.04 (m, 2H), 6.81 (d, *J* = 8.6 Hz, 2H), 3.73 (s, 3H), 3.51 (q, *J* = 9.9 Hz, 1H), 3.11 (t, *J* = 9.5 Hz, 1H), 2.64 – 2.51 (m, 1H), 2.38 – 2.26 (m, 1H), 2.17 (s, 3H), 1.53 (q, *J* = 10.1 Hz, 1H), 1.18 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 158.01, 142.24, 135.97, 135.86, 129.92, 127.70, 125.84, 125.77, 125.67, 113.68, 55.13, 53.31, 41.67, 35.61, 34.64,

20.56, 19.77.

HRMS (ESI) m/z calcd for $[C_{19}H_{23}O]^+$ $[M+H]^+$: 267.1743, found 267.1748.



4c

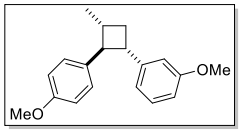
(±)-1-bromo-2-((*1S,2S,3R*)-2-(4-methoxyphenyl)-3-methylcyclobutyl)benzene (4c)

Following the general procedure B; 64% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 15:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.55 – 7.43 (m, 1H), 7.35 (d, *J* = 7.1 Hz, 1H), 7.21 (dd, *J* = 31.1, 8.1 Hz, 3H), 7.11 – 6.90 (m, 1H), 6.83 (d, *J* = 8.6 Hz, 2H), 3.75 (s, 3H), 3.70 (q, *J* = 10.0 Hz, 1H), 3.13 (t, *J* = 9.6 Hz, 1H), 2.84 – 2.68 (m, 1H), 2.42 – 2.23 (m, 1H), 1.45 (q, *J* = 10.1 Hz, 1H), 1.20 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 158.11, 143.10, 135.20, 132.64, 127.74, 127.72, 127.43, 127.34, 124.12, 113.73, 55.19, 52.72, 43.47, 35.72, 35.32, 20.56.

HRMS (ESI) m/z calcd for $[C_{18}H_{20}BrO]^+$ $[M+H]^+$: 331.0692, found 331.0529.



4d

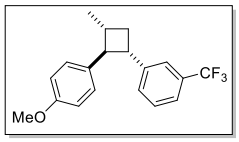
(±)-1-methoxy-3-((*1S,2S,3R*)-2-(4-methoxyphenyl)-3-methylcyclobutyl)benzene (4d)

Following the general procedure B; 67% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 15:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.19 – 7.13 (m, 3H), 6.83 (d, *J* = 8.7 Hz, 2H), 6.78 (d, *J* = 7.6 Hz, 1H), 6.75 – 6.67 (m, 2H), 3.76 (s, 3H), 3.75 (s, 3H), 3.35 (q, *J* = 9.8 Hz, 1H), 2.93 (t, *J* = 9.5 Hz, 1H), 2.54 – 2.42 (m, 1H), 2.31 (ddd, *J* = 14.1, 9.5, 7.4 Hz, 1H), 1.68 (q, *J* = 10.1 Hz, 1H), 1.17 (d, *J* = 6.5 Hz, 4H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 159.54, 158.04, 146.36, 135.73, 129.17, 127.73, 127.68, 119.03, 113.72, 113.67, 112.55, 110.94, 55.40, 55.18, 55.05, 44.13, 35.32, 33.88, 20.42.

HRMS (ESI) m/z calcd for $[C_{19}H_{23}O_2]^+$ $[M+H]^+$: 283.1693, found 283.1698.



4e

(±)-1-((*1S,2S,3R*)-2-(4-methoxyphenyl)-3-methylcyclobutyl)-3-(trifluoromethyl)benzene (4e)

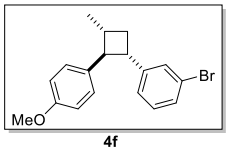
Following the general procedure B; 60% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.38 – 7.29 (m, 2H), 7.28 – 7.20 (m, 2H), 7.07 (d, *J* = 8.6 Hz, 2H), 6.80 – 6.74 (m, 2H), 3.68 (s, 3H), 3.40 – 3.26 (m, 1H), 2.84 (t, *J* = 9.6 Hz, 1H), 2.44 (dt, *J* = 10.1, 7.8 Hz, 1H), 2.27 (ddd, *J* = 9.2, 6.7, 2.2 Hz, 1H), 1.60 (q, *J* = 10.1 Hz, 1H), 1.09 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 158.24, 145.47, 135.10, 130.62, 130.37, 130.09, 128.62, 127.74, 125.37, 123.25, 123.22, 122.78, 122.75, 113.85, 55.55, 55.18, 43.93, 35.54, 33.78, 20.32.

HRMS (ESI) m/z calcd for $[C_{19}H_{20}F_3O]^+$ $[M+H]^+$: 321.1461, found

321.1461.



4f

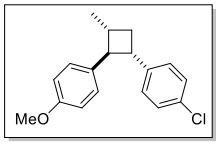
(±)-1-bromo-3-((*1S,2S,3R*)-2-(4-methoxyphenyl)-3-methylcyclobutyl)benzene (4f)

Following the general procedure B; 77% yield; dr:>10:1; White Oil; R_f = 0.2 (*n*-hexane : EA = 15:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.24 (s, 1H), 7.19 (dt, *J* = 7.2, 1.8 Hz, 1H), 7.05 (d, *J* = 8.6 Hz, 2H), 7.03 – 6.96 (m, 2H), 6.79 – 6.73 (m, 2H), 3.68 (s, 3H), 3.24 (q, *J* = 9.9 Hz, 1H), 2.81 (t, *J* = 9.5 Hz, 1H), 2.40 (dt, *J* = 10.1, 7.8 Hz, 1H), 2.29 – 2.17 (m, 1H), 1.56 (q, *J* = 10.1 Hz, 1H), 1.08 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 158.15, 146.96, 135.18, 129.76, 129.64, 128.97, 127.69, 125.33, 122.42, 113.80, 55.46, 55.18, 43.75, 35.45, 33.81, 20.35.

HRMS (ESI) m/z calcd for $[C_{18}H_{20}BrO]^+$ $[M+H]^+$: 331.0692, found 331.0679.



4g

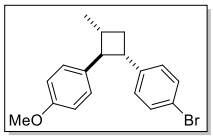
(±)-1-chloro-4-((*1S,2S,3R*)-2-(4-methoxyphenyl)-3-methylcyclobutyl)benzene (4g)

Following the general procedure B; 71% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.20 (d, *J* = 8.4 Hz, 2H), 7.12 (d, *J* = 8.6 Hz, 2H), 7.08 (d, *J* = 8.4 Hz, 2H), 6.83 (d, *J* = 8.7 Hz, 2H), 3.75 (s, 3H), 3.31 (td, *J* = 10.0, 7.9 Hz, 1H), 2.86 (t, *J* = 9.5 Hz, 1H), 2.47 (dt, *J* = 10.2, 7.7 Hz, 1H), 2.37 – 2.25 (m, 1H), 1.63 (q, *J* = 10.1 Hz, 1H), 1.16 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 158.13, 143.00, 135.28, 131.49, 128.25, 127.92, 127.68, 113.78, 55.72, 55.13, 43.61, 35.36, 33.78, 20.35.

HRMS (ESI) m/z calcd for $[C_{18}H_{20}ClO]^+$ $[M+H]^+$: 287.1197, found 287.1200.



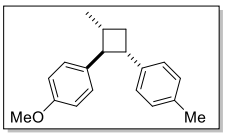
4h

(±)-1-bromo-4-((*1S,2S,3R*)-2-(4-methoxyphenyl)-3-methylcyclobutyl)benzene (4h)

Following the general procedure B; 45% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.45 – 7.40 (m, 2H), 7.21 (d, *J* = 8.6 Hz, 2H), 7.13 – 7.08 (m, 2H), 6.95 – 6.89 (m, 2H), 3.84 (s, 3H), 3.43 – 3.31 (m, 1H), 2.94 (t, *J* = 9.5 Hz, 1H), 2.56 (dt, *J* = 10.0, 7.8 Hz, 1H), 2.45 – 2.32 (m, 1H), 1.71 (q, *J* = 10.1 Hz, 1H), 1.24 (d, *J* = 6.5 Hz, 3H).

HRMS (ESI) m/z calcd for $[C_{18}H_{20}BrO]^+$ $[M+H]^+$: 331.0692, found 331.0538.



4i

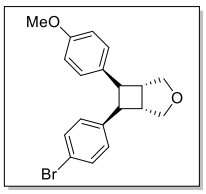
(±)-1-methoxy-4-((*1S,2R,4S*)-2-methyl-4-(p-tolyl)cyclobutyl)benzene (4i)

Following the general procedure B; 82% yield; dr:>10:1; White Oil; R_f = 0.5 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.20 (d, *J* = 8.6 Hz, 2H), 7.16 – 7.08 (m, 4H), 6.88 (d, *J* = 8.6 Hz, 2H), 3.81 (s, 3H), 3.38 (q, *J* = 9.8 Hz, 1H), 2.96 (t, *J* = 9.5 Hz, 1H), 2.58 – 2.47 (m, 1H), 2.34 (s, 4H), 1.71 (q, *J* = 10.1 Hz, 1H), 1.22 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.98, 141.61, 135.90, 135.34, 128.88, 127.72, 126.50, 113.68, 55.56, 55.18, 43.81, 35.35, 34.06, 20.98, 20.47.

HRMS (ESI) m/z calcd for $[C_{19}H_{23}O]^+$ $[M+H]^+$: 267.1743, found 267.1747.



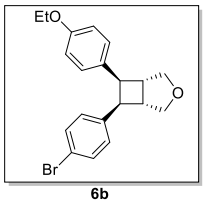
6a

(*1S,5R,6R,7S*)-6-(4-bromophenyl)-7-(4-methoxyphenyl)-3-oxabicyclo[3.2.0]heptane (6a)

Following the general procedure C; 79% yield; dr:>10:1; White Oil; R_f = 0.2 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.23 – 7.16 (m, 2H), 6.93 – 6.77 (m, 4H), 6.68 – 6.64 (m, 2H), 4.08 (dd, *J* = 9.4, 4.0 Hz, 2H), 3.76 – 3.63 (m, 7H), 3.22 (dq, *J* = 4.1, 1.9 Hz, 2H).

HRMS (ESI) m/z calcd for $[C_{19}H_{20}BrO_2]^+$ $[M+H]^+$: 359.0641, found 359.0641.



6b

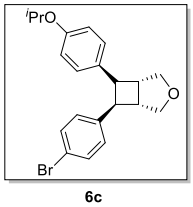
(*1S,5R,6S,7R*)-6-(4-bromophenyl)-7-(4-ethoxyphenyl)-3-oxabicyclo[3.2.0]heptane (6b)

Following the general procedure C; 71% yield; dr:>10:1; White Oil; R_f = 0.2 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.20 (d, *J* = 8.3 Hz, 2H), 6.81 (dd, *J* = 14.2, 8.5 Hz, 4H), 6.64 (d, *J* = 8.5 Hz, 2H), 4.08 (dd, *J* = 9.4, 4.4 Hz, 2H), 3.92 (q, *J* = 7.0 Hz, 2H), 3.67 (dq, *J* = 11.4, 4.1 Hz, 4H), 3.22 (s, 2H), 1.35 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 156.98, 140.02, 132.40, 130.73, 129.76, 128.97, 119.36, 113.96, 73.92, 73.83, 63.28, 46.61, 46.38, 42.46, 42.14, 14.76.

HRMS (ESI) m/z calcd for $[C_{20}H_{22}BrO_2]^+$ $[M+H]^+$: 373.0798, found 373.0793.



6c

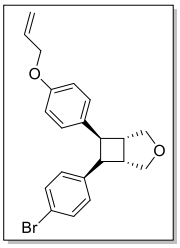
(*1S,5R,6S,7S*)-6-(4-bromophenyl)-7-(4-isopropoxyphenyl)-3-oxabicyclo[3.2.0]heptane (6c)

Following the general procedure C; 77% yield; dr:>10:1; White Oil; R_f = 0.2 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.19 (d, *J* = 8.4 Hz, 2H), 6.80 (dd, *J* = 11.3, 8.5 Hz, 4H), 6.63 (d, *J* = 8.6 Hz, 2H), 4.41 (p, *J* = 6.1 Hz, 1H), 4.08 (dd, *J* = 9.4, 4.2 Hz, 2H), 3.67 (dq, *J* = 13.4, 4.4 Hz, 4H), 3.22 (t, *J* = 4.2 Hz, 2H), 1.25 (d, *J* = 6.1 Hz, 7H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 155.87, 139.99, 132.46, 130.68, 129.78, 128.98, 119.33, 115.69, 73.93, 73.83, 69.96, 46.64, 46.42, 42.33, 42.04, 29.67, 21.92.

HRMS (ESI) m/z calcd for $[C_{21}H_{24}BrO_2]^+$ $[M+H]^+$: 387.0954, found 387.0947.



6d

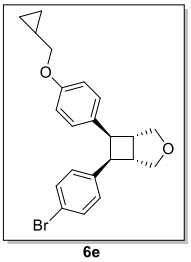
(*1R,5S,6R,7R*)-6-(4-(allyloxy)phenyl)-7-(4-bromophenyl)-3-oxabicyclo[3.2.0]heptane (6d)

Following the general procedure C; 75% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.20 (d, *J* = 8.4 Hz, 2H), 6.81 (dd, *J* = 18.4, 8.5 Hz, 4H), 6.66 (d, *J* = 8.7 Hz, 2H), 6.00 (ddt, *J* = 17.2, 10.6, 5.3 Hz, 1H), 5.35 (dd, *J* = 17.3, 1.6 Hz, 1H), 5.30 – 5.19 (m, 1H), 4.43 (dt, *J* = 5.3, 1.5 Hz, 2H), 4.08 (dd, *J* = 9.5, 4.0 Hz, 2H), 3.74 – 3.60 (m, 4H), 3.22 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 156.55, 139.94, 133.23, 132.68, 130.70, 129.74, 128.94, 119.33, 117.52, 114.16, 73.88, 73.79, 68.64, 46.57, 46.32, 42.34, 42.04.

HRMS (ESI) m/z calcd for $[C_{21}H_{22}BrO_2]^+$ $[M+H]^+$: 385.0798, found 385.0789.



6e

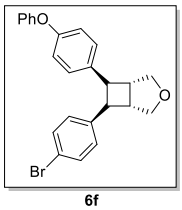
(*1S,5R,6S,7R*)-6-(4-bromophenyl)-7-(4-(cyclopropylmethoxy)phenyl)-3-oxabicyclo[3.2.0]heptane (6e)

Following the general procedure C; 83% yield; dr:>10:1; White Oil; R_f = 0.2 (*n*-hexane : EA = 15:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.20 (d, *J* = 8.4 Hz, 2H), 6.81 (dd, *J* = 12.2, 8.6 Hz, 4H), 6.65 (d, *J* = 8.7 Hz, 2H), 4.07 (dd, *J* = 9.5, 3.2 Hz, 2H), 3.74 – 3.59 (m, 6H), 3.28 – 3.14 (m, 2H), 1.26 – 1.14 (m, 1H), 0.68 – 0.53 (m, 2H), 0.38 – 0.23 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.01, 139.99, 132.47, 130.71, 129.74, 128.93, 119.32, 114.09, 73.89, 73.80, 72.65, 46.56, 46.32, 42.42, 42.07, 10.21, 3.14, 3.07.

HRMS (ESI) m/z calcd for $[C_{22}H_{24}BrO_2]^+$ $[M+H]^+$: 399.0954, found 399.0946.



6f

(*1S,5R,6R,7S*)-6-(4-bromophenyl)-7-(4-phenoxyphenyl)-3-oxabicyclo[3.2.0]heptane (6f)

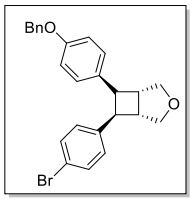
Following the general procedure C; 82% yield; dr:>10:1; White Oil; R_f = 0.2 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.35 – 7.25 (m, 2H), 7.22 (d, *J* = 8.4 Hz, 2H), 7.03 (t, *J* = 7.4 Hz, 1H), 6.89 (d, *J* = 8.5 Hz, 2H), 6.84 – 6.72 (m, 5H), 4.17 – 4.01 (m, 2H), 3.80 – 3.61 (m, 4H), 3.25 (dq, *J* = 8.3, 4.2, 3.5 Hz, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.94, 154.72, 139.81, 135.73, 130.80, 130.76, 129.89, 129.69, 129.33, 122.69, 119.50, 119.19, 117.95, 73.95,

73.87, 46.81, 46.67, 41.90, 41.84.

HRMS (ESI) m/z calcd for $[C_{24}H_{22}BrO_2]^+$ $[M+H]^+$: 421.0798, found 421.0795.



6g

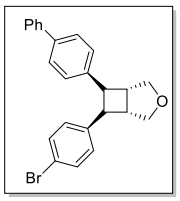
(*1R,5S,6S,7S*)-6-(4-(benzyloxy)phenyl)-7-(4-bromophenyl)-3-oxabicyclo[3.2.0]heptane (6g)

Following the general procedure C; 67% yield; dr:>10:1; White Oil; R_f = 0.2 (*n*-hexane : EA = 15:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.39 – 7.33 (m, 4H), 7.33 – 7.27 (m, 1H), 7.19 (d, *J* = 8.4 Hz, 2H), 6.83 (d, *J* = 8.6 Hz, 2H), 6.79 (d, *J* = 8.4 Hz, 2H), 6.71 (d, *J* = 8.7 Hz, 2H), 4.96 (s, 2H), 4.07 (dd, *J* = 9.4, 3.4 Hz, 2H), 3.67 (td, *J* = 9.1, 4.5 Hz, 4H), 3.21 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 156.82, 139.98, 137.04, 132.85, 130.74, 129.77, 129.00, 128.48, 127.83, 127.41, 119.37, 114.41, 73.91, 73.82, 69.91, 46.61, 46.38, 42.41, 42.13.

HRMS (ESI) m/z calcd for $[C_{25}H_{24}BrO_2]^+$ $[M+H]^+$: 435.0954, found 435.0950.



6h

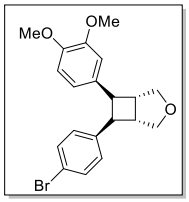
(*1R,5S,6S,7R*)-6-([1,1'-biphenyl]-4-yl)-7-(4-bromophenyl)-3-oxabicyclo[3.2.0]heptane (6h)

Following the general procedure C; 74% yield; dr:>10:1; White Oil; R_f = 0.2 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.53 – 7.47 (m, 2H), 7.42 – 7.32 (m, 4H), 7.31 – 7.26 (m, 1H), 7.23 – 7.16 (m, 2H), 6.99 (d, *J* = 8.2 Hz, 2H), 6.83 (d, *J* = 8.4 Hz, 2H), 4.10 (dd, *J* = 9.3, 7.2 Hz, 2H), 3.77 (dd, *J* = 10.1, 5.0 Hz, 1H), 3.70 (dt, *J* = 9.3, 4.6 Hz, 3H), 3.37 – 3.20 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 140.68, 139.86, 139.56, 138.57, 130.81, 129.75, 128.64, 128.43, 127.04, 126.84, 126.51, 119.49, 73.89, 73.82, 46.72, 46.61, 42.35, 42.27.

HRMS (ESI) m/z calcd for $[C_{24}H_{22}BrO]^+$ $[M+H]^+$: 405.0849, found 405.0842.



6i

(*1S,5R,6R,7S*)-6-(4-bromophenyl)-7-(3,4-dimethoxyphenyl)-3-oxabicyclo[3.2.0]heptane (6i)

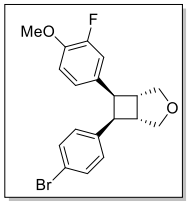
Following the general procedure C; 67% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.21 (d, *J* = 8.4 Hz, 2H), 6.82 (d, *J* = 8.4 Hz, 2H), 6.65 (d, *J* = 8.2 Hz, 1H), 6.56 (dd, *J* = 8.2, 1.9 Hz, 1H), 6.27 (d, *J* = 1.9 Hz, 1H), 4.08 (d, *J* = 10.0 Hz, 2H), 3.79 (s, 3H), 3.68 (ddd, *J* = 12.2, 6.6, 2.7 Hz, 4H), 3.62 (s, 3H), 3.22 (q, *J* = 4.3 Hz, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 148.39, 147.21, 140.14, 133.08, 131.71, 130.83, 129.79, 127.98, 119.94, 119.53, 111.99, 110.61, 73.90, 73.86, 55.78, 55.73, 46.72, 46.62, 42.61, 42.12.

HRMS (ESI) m/z calcd for $[C_{20}H_{22}BrO_3]^+$ $[M+H]^+$: 389.0747, found

389.0745.



6j

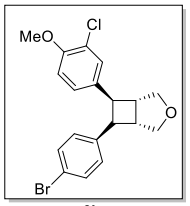
(*1S,5R,6S,7R*)-6-(4-bromophenyl)-7-(3-fluoro-4-methoxyphenyl)-3-oxabicyclo[3.2.0]heptane (6j)

Following the general procedure C; 90% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.22 (d, *J* = 8.4 Hz, 2H), 6.80 (d, *J* = 8.4 Hz, 2H), 6.74 – 6.64 (m, 2H), 6.61 (d, *J* = 8.5 Hz, 1H), 4.07 (dd, *J* = 9.4, 3.2 Hz, 2H), 3.78 (s, 3H), 3.72 – 3.56 (m, 4H), 3.28 – 3.12 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 152.92, 150.97, 145.60, 145.51, 139.63, 133.72, 133.68, 130.92, 129.70, 123.66, 123.63, 119.63, 115.73, 115.58, 112.90, 73.80, 73.79, 56.20, 46.60, 46.21, 42.30, 42.07, 29.70.

HRMS (ESI) m/z calcd for $[C_{19}H_{19}BrFO_2]^+$ $[M+H]^+$: 377.0547, found 377.0546.



6k

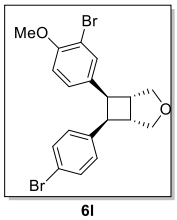
(*1S,5R,6R,7R*)-6-(4-bromophenyl)-7-(3-chloro-4-methoxyphenyl)-3-oxabicyclo[3.2.0]heptane (6k)

Following the general procedure C; 85% yield; dr:>10:1; White Oil; R_f = 0.4 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.26 – 7.19 (m, 2H), 6.97 (d, *J* = 2.2 Hz, 1H), 6.84 – 6.77 (m, 2H), 6.71 (dd, *J* = 8.5, 2.2 Hz, 1H), 6.64 (d, *J* = 8.5 Hz, 1H), 4.07 (d, *J* = 9.4 Hz, 2H), 3.79 (s, 3H), 3.72 – 3.57 (m, 4H), 3.21 (q, *J* = 3.7 Hz, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 152.97, 139.54, 133.69, 130.88, 129.70, 129.60, 127.28, 121.74, 119.61, 111.55, 73.75, 56.00, 46.56, 45.97, 42.31, 42.01.

HRMS (ESI) m/z calcd for $[C_{19}H_{19}BrClO_2]^+$ $[M+H]^+$: 393.0251, found 393.0247.



6l

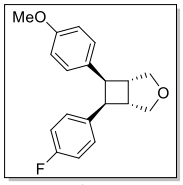
(*1R,5S,6R,7S*)-6-(3-bromo-4-methoxyphenyl)-7-(4-bromophenyl)-3-oxabicyclo[3.2.0]heptane (6l)

Following the general procedure C; 68% yield; dr:>10:1; White Oil; R_f = 0.4 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.23 (d, *J* = 8.3 Hz, 2H), 7.14 (d, *J* = 2.0 Hz, 1H), 6.81 (d, *J* = 8.3 Hz, 2H), 6.74 (dd, *J* = 8.4, 2.0 Hz, 1H), 6.61 (d, *J* = 8.5 Hz, 1H), 4.07 (d, *J* = 9.5 Hz, 2H), 3.78 (s, 3H), 3.71 – 3.60 (m, 4H), 3.21 (q, *J* = 4.0 Hz, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 153.87, 139.53, 134.17, 132.66, 130.89, 129.72, 128.06, 119.63, 111.39, 111.03, 73.76, 56.11, 46.61, 45.91, 42.38, 42.00.

HRMS (ESI) m/z calcd for $[C_{19}H_{19}Br_2O_2]^+$ $[M+H]^+$: 436.9746, found 436.9738.



6m

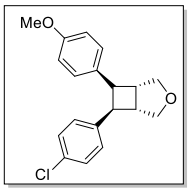
(*1S,5R,6S,7R*)-6-(4-fluorophenyl)-7-(4-methoxyphenyl)-3-oxabicyclo[3.2.0]heptane (6m)

Following the general procedure C; 92% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 6.90 – 6.85 (m, 2H), 6.85 – 6.80 (m, 2H), 6.80 – 6.72 (m, 2H), 6.67 – 6.60 (m, 2H), 4.08 (d, *J* = 9.5 Hz, 2H), 3.68 (d, *J* = 10.1 Hz, 7H), 3.28 – 3.17 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 161.84, 159.90, 157.51, 136.57, 136.54, 132.63, 129.38, 129.32, 128.98, 128.76, 114.52, 114.36, 113.17, 73.90, 73.84, 55.05, 46.42, 42.28, 42.22.

HRMS (ESI) m/z calcd for $[C_{19}H_{20}FO_2]^+$ $[M+H]^+$: 299.1442, found 299.1439.



6n

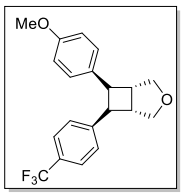
(*1S,5R,6S,7S*)-6-(4-chlorophenyl)-7-(4-methoxyphenyl)-3-oxabicyclo[3.2.0]heptane (6n)

Following the general procedure C; 87% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.10 – 7.00 (m, 2H), 6.90 – 6.80 (m, 4H), 6.69 – 6.60 (m, 2H), 4.08 (dd, *J* = 9.4, 3.0 Hz, 2H), 3.76 – 3.60 (m, 7H), 3.28 – 3.15 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.56, 139.44, 132.52, 131.21, 129.34, 128.97, 127.79, 113.26, 73.90, 73.81, 55.08, 46.51, 46.37, 42.42, 42.12.

HRMS (ESI) m/z calcd for $[C_{19}H_{20}ClO_2]^+$ $[M+H]^+$: 315.1146, found 315.1146.



6o

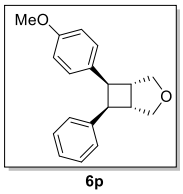
(*1R,5S,6S,7R*)-6-(4-methoxyphenyl)-7-(4-(trifluoromethyl)phenyl)-3-oxabicyclo[3.2.0]heptane (6o)

Following the general procedure C; 77% yield; dr:>10:1; White Oil; R_f = 0.4 (*n*-hexane : EA = 15:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.33 (d, *J* = 8.2 Hz, 2H), 7.03 (d, *J* = 8.1 Hz, 2H), 6.83 (d, *J* = 8.7 Hz, 2H), 6.63 (d, *J* = 8.7 Hz, 2H), 4.09 (dd, *J* = 9.4, 4.7 Hz, 2H), 3.79 – 3.65 (m, 7H), 3.27 (dtd, *J* = 18.2, 8.4, 5.0 Hz, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.68, 145.13, 132.31, 128.93, 128.26, 124.61, 124.58, 124.55, 124.52, 113.33, 73.88, 73.76, 55.08, 46.89, 46.45, 42.55, 41.96.

HRMS (ESI) m/z calcd for $[C_{20}H_{20}F_3O_2]^+$ $[M+H]^+$: 349.1410, found 349.1406.



6p

(*1R,5S,6R,7S*)-6-(4-methoxyphenyl)-7-phenyl-3-oxabicyclo[3.2.0]heptane (6p)

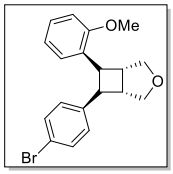
Following the general procedure C; 61% yield; dr:>10:1; White Oil; R_f = 0.4 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.09 (t, *J* = 7.5 Hz, 2H), 7.01 (d, *J* = 7.3 Hz, 1H), 6.94 (d, *J* = 7.2 Hz, 2H), 6.88 – 6.81 (m, 2H), 6.68 – 6.58 (m,

2H), 4.09 (d, *J* = 9.4 Hz, 2H), 3.69 (d, *J* = 7.7 Hz, 7H), 3.30 (dt, *J* = 9.7, 4.9 Hz, 1H), 3.24 (dt, *J* = 8.3, 4.8 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.45, 140.86, 132.97, 129.02, 128.08, 127.70, 125.54, 113.10, 73.97, 55.08, 47.15, 46.48, 42.60, 41.97.

HRMS (ESI) m/z calcd for $[C_{19}H_{21}O_2]^+$ $[M+H]^+$: 281.1536, found 281.1535.



6q

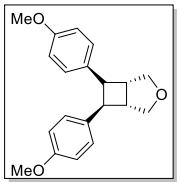
(*1S,5R,6R,7S*)-6-(4-bromophenyl)-7-(2-methoxyphenyl)-3-oxabicyclo[3.2.0]heptane (6q)

Following the general procedure C; 83% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.22 – 7.08 (m, 3H), 7.07 – 6.98 (m, 1H), 6.80 (dd, *J* = 7.2, 5.1 Hz, 3H), 6.49 (d, *J* = 8.1 Hz, 1H), 4.10 (d, *J* = 9.4 Hz, 1H), 4.03 (d, *J* = 9.2 Hz, 1H), 3.93 (dd, *J* = 10.0, 6.1 Hz, 1H), 3.68 (dd, *J* = 9.3, 5.5 Hz, 2H), 3.62 (dd, *J* = 10.1, 4.8 Hz, 1H), 3.50 (s, 4H), 3.03 (dt, *J* = 9.3, 5.4 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 156.52, 141.02, 130.06, 129.65, 128.17, 127.22, 127.01, 119.67, 119.08, 109.55, 73.97, 73.94, 54.54, 47.43, 42.94, 41.97, 39.64.

HRMS (ESI) m/z calcd for $[C_{19}H_{20}BrO_2]^+$ $[M+H]^+$: 359.0641, found 359.0642.



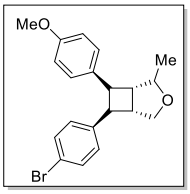
6r

(*1R,5S,6S,7S*)-6,7-bis(4-methoxyphenyl)-3-oxabicyclo[3.2.0]heptane (6r)

Following the general procedure C; 50% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 20:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 6.89 – 6.82 (m, 4H), 6.68 – 6.61 (m, 4H), 4.07 (d, *J* = 9.4 Hz, 2H), 3.86 (s, 1H), 3.74 – 3.61 (m, 10H), 3.26 – 3.18 (m, 2H).

HRMS (ESI) m/z calcd for $[C_{20}H_{23}O_3]^+$ $[M+H]^+$: 311.1642 found 311.1640.



6s

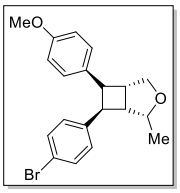
(*1S,2R,5R,6R,7S*)-6-(4-bromophenyl)-7-(4-methoxyphenyl)-2-methyl-3-oxabicyclo[3.2.0]heptane (6s)

Following the general procedure C; 56% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.20 (d, *J* = 8.4 Hz, 2H), 6.81 (dd, *J* = 13.4, 8.5 Hz, 4H), 6.65 (d, *J* = 8.7 Hz, 2H), 4.35 (q, *J* = 6.5 Hz, 1H), 4.01 (qd, *J* = 9.6, 3.7 Hz, 2H), 3.71 (s, 4H), 3.66 (dd, *J* = 9.9, 5.1 Hz, 1H), 3.30 – 3.20 (m, 1H), 2.92 (dd, *J* = 8.0, 5.5 Hz, 1H), 1.16 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.62, 140.06, 132.55, 130.76, 129.74, 128.95, 119.37, 113.31, 81.01, 71.54, 55.11, 48.75, 46.67, 46.10, 42.06, 19.36.

HRMS (ESI) m/z calcd for $[C_{20}H_{22}BrO_2]^+$ $[M+H]^+$: 373.0798, found 373.0796.



6t

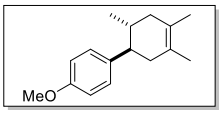
(*1S,2R,5S,6S,7S*)-7-(4-bromophenyl)-6-(4-methoxyphenyl)-2-methyl-3-oxabicyclo[3.2.0]heptane (6t)

Following the general procedure C; 67% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.20 (d, *J* = 8.4 Hz, 2H), 6.84 (d, *J* = 8.6 Hz, 2H), 6.78 (d, *J* = 8.4 Hz, 2H), 6.65 (d, *J* = 8.7 Hz, 2H), 4.34 (q, *J* = 6.5 Hz, 1H), 4.07 – 3.97 (m, 2H), 3.70 (d, *J* = 7.4 Hz, 5H), 3.24 (dtd, *J* = 7.6, 4.9, 3.7, 1.8 Hz, 1H), 2.92 (dd, *J* = 8.1, 4.6 Hz, 1H), 1.17 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.60, 140.02, 132.56, 130.73, 129.70, 128.95, 119.36, 113.31, 80.87, 71.61, 55.08, 48.37, 46.41, 46.34, 42.37, 19.30.

HRMS (ESI) m/z calcd for $[C_{20}H_{22}BrO_2]^+$ $[M+H]^+$: 373.0798, found 373.0795.



8a

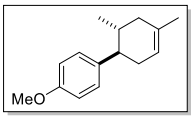
(±)-(*1R,2R*)-4'-methoxy-2,4,5-trimethyl-1,2,3,6-tetrahydro-1,1'-biphenyl (8a)

Following the general procedure D; 98% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 30:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.09 (d, *J* = 8.6 Hz, 2H), 6.85 (d, *J* = 8.7 Hz, 2H), 3.79 (s, 3H), 2.35 (td, *J* = 10.6, 5.7 Hz, 1H), 2.24 – 2.02 (m, 3H), 1.94 – 1.78 (m, 2H), 1.64 (d, *J* = 13.8 Hz, 6H), 0.71 (d, *J* = 6.2 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.70, 138.11, 128.39, 125.40, 125.24, 113.63, 55.11, 47.78, 41.80, 41.60, 34.21, 19.97, 18.69, 18.61.

HRMS (ESI) m/z calcd for $[C_{16}H_{23}O]^+$ $[M+H]^+$: 231.1743, found 231.1747.



8b

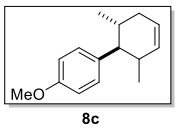
(±)-(*1R,2R*)-4'-methoxy-2,4-dimethyl-1,2,3,6-tetrahydro-1,1'-biphenyl (8b)

Following the general procedure D; 96% yield; dr:>10:1; White Oil; R_f = 0.4 (*n*-hexane : EA = 20:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.13 (d, *J* = 8.5 Hz, 2H), 6.89 (d, *J* = 8.5 Hz, 2H), 5.50 (s, 1H), 3.82 (s, 3H), 2.35 (td, *J* = 10.7, 5.3 Hz, 1H), 2.30 – 2.09 (m, 3H), 2.00 – 1.91 (m, 1H), 1.89 – 1.81 (m, 1H), 1.74 (s, 3H), 0.76 (d, *J* = 6.4 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.82, 138.19, 133.81, 128.54, 121.01, 113.74, 55.19, 47.02, 39.93, 35.37, 34.02, 23.49, 20.33.

HRMS (ESI) m/z calcd for $[C_{15}H_{21}O]^+$ $[M+H]^+$: 217.1587, found 217.1591.



8c

(±)-(*1S,2R*)-4'-methoxy-2,6-dimethyl-1,2,3,6-tetrahydro-1,1'-biphenyl (8c)

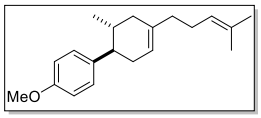
Following the general procedure D; 97% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 20:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.06 (d, *J* = 8.0 Hz, 2H), 6.83 (d, *J* = 8.0 Hz, 2H), 5.68 (d, *J* = 39.8 Hz, 2H), 3.78 (s, 3H), 2.66 (dd, *J* = 10.4, 5.0 Hz, 1H), 2.36 – 2.21 (m, 2H), 2.13 (s, 1H), 1.80 (dd, *J* = 16.7, 9.3 Hz, 1H),

0.79 (dd, *J* = 45.9, 6.6 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.55, 135.32, 133.41, 130.04, 125.05, 113.15, 55.06, 50.42, 35.50, 34.73, 26.40, 20.51, 16.75.

HRMS (ESI) m/z calcd for $[C_{15}H_{21}O]^+$ $[M+H]^+$: 217.1587, found 217.1591.



8d

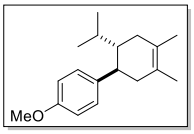
(±)-(*1R,2R*)-4'-methoxy-2-methyl-4-(4-methylpent-3-en-1-yl)-1,2,3,6-tetrahydro-1,1'-biphenyl (8d)

Following the general procedure D; 93% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 30:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.08 (d, *J* = 8.6 Hz, 2H), 6.83 (d, *J* = 8.6 Hz, 2H), 5.45 (s, 1H), 5.14 (t, *J* = 6.9 Hz, 1H), 3.77 (s, 3H), 2.41 – 2.06 (m, 6H), 1.99 (t, *J* = 7.7 Hz, 2H), 1.93 – 1.76 (m, 2H), 1.70 (s, 3H), 1.61 (d, *J* = 14.4 Hz, 3H), 0.71 (d, *J* = 6.3 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.69, 138.14, 137.37, 131.30, 128.42, 124.35, 120.40, 113.61, 55.08, 47.02, 38.07, 37.51, 35.14, 33.92, 26.43, 25.70, 20.25, 17.68.

HRMS (ESI) m/z calcd for $[C_{20}H_{29}O]^+$ $[M+H]^+$: 285.2213, found 285.2219.



8e

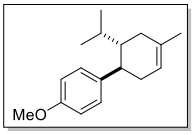
(±)-(*1R,2S*)-2-isopropyl-4'-methoxy-4,5-dimethyl-1,2,3,6-tetrahydro-1,1'-biphenyl (8e)

Following the general procedure D; 95% yield; dr:>10:1; White Oil; R_f = 0.4 (*n*-hexane : EA = 20:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.00 (d, *J* = 8.6 Hz, 2H), 6.75 (d, *J* = 8.6 Hz, 2H), 3.68 (s, 3H), 2.49 (td, *J* = 10.7, 6.0 Hz, 1H), 2.08 – 1.94 (m, 2H), 1.83 (d, *J* = 10.2 Hz, 2H), 1.73 (ddt, *J* = 10.5, 8.4, 4.0 Hz, 1H), 1.58 (s, 4H), 1.52 (s, 3H), 1.46 (td, *J* = 6.9, 3.4 Hz, 1H), 0.75 (d, *J* = 7.0 Hz, 3H), 0.57 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.62, 137.81, 128.56, 125.25, 125.05, 113.65, 55.07, 43.91, 43.78, 42.74, 30.70, 27.03, 21.30, 18.99, 18.57, 14.84.

HRMS (ESI) m/z calcd for $[C_{18}H_{27}O]^+$ $[M+H]^+$: 259.2056, found 259.2054.



8f

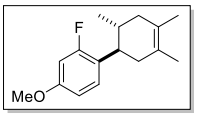
(±)-(*1R,2S*)-2-isopropyl-4'-methoxy-4-methyl-1,2,3,6-tetrahydro-1,1'-biphenyl (8f)

Following the general procedure D; 92% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 30:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.09 (d, *J* = 8.6 Hz, 2H), 6.83 (d, *J* = 8.6 Hz, 2H), 3.77 (s, 3H), 2.53 (td, *J* = 10.7, 5.3 Hz, 1H), 2.20 (d, *J* = 17.3 Hz, 1H), 2.16 – 2.06 (m, 1H), 1.95 – 1.78 (m, 3H), 1.71 (s, 3H), 1.55 (td, *J* = 6.9, 3.2 Hz, 1H), 0.84 (d, *J* = 7.0 Hz, 3H), 0.66 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.62, 137.83, 133.84, 128.59, 120.51, 113.63, 55.08, 43.46, 43.12, 36.21, 29.09, 27.07, 23.65, 21.23, 14.80.

HRMS (ESI) m/z calcd for $[C_{17}H_{25}O]^+$ $[M+H]^+$: 245.1900, found 245.1903.



8g

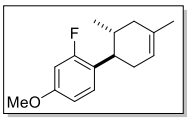
(±)-(*1R,2R*)-2'-fluoro-4'-methoxy-2,4,5-trimethyl-1,2,3,6-tetrahydro-1,1'-biphenyl (8g)

Following the general procedure D; 96% yield; dr:>10:1; White Oil; R_f = 0.3 (*n*-hexane : EA = 20:1);

^{1}H NMR (500 MHz, Chloroform-*d*) δ 6.97 (t, *J* = 8.5 Hz, 1H), 6.64 – 6.35 (m, 2H), 3.67 (s, 3H), 2.63 (td, *J* = 10.9, 5.3 Hz, 1H), 2.10 (d, *J* = 12.0 Hz, 1H), 1.97 (d, *J* = 17.3 Hz, 2H), 1.89 – 1.70 (m, 2H), 1.54 (d, *J* = 11.6 Hz, 6H), 0.64 (d, *J* = 6.3 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 162.42, 160.48, 158.68, 158.60, 128.99, 128.93, 125.29, 125.19, 124.22, 124.09, 109.91, 109.89, 101.37, 101.16, 55.33, 41.46, 40.48, 39.91, 33.32, 19.64, 18.70, 18.60.

HRMS (ESI) m/z calcd for $[C_{16}H_{22}FO]^+$ $[M+H]^+$: 249.1649, found 249.1644.



8h

(±)-(*1R,2R*)-2'-fluoro-4'-methoxy-2,4-dimethyl-1,2,3,6-tetrahydro-1,1'-biphenyl (8h)

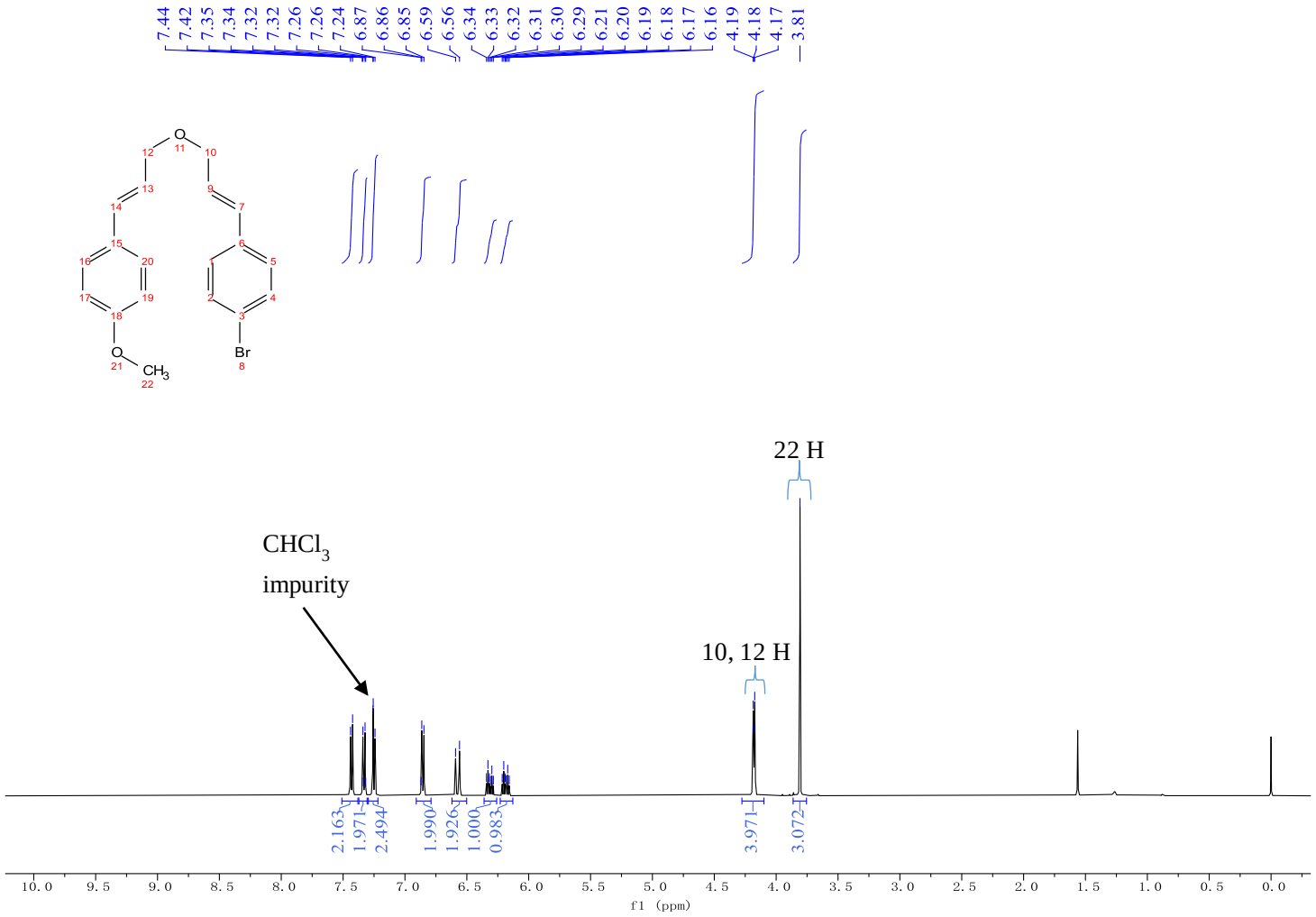
Following the general procedure D; 94% yield; dr:>10:1; White Oil; R_f = 0.5 (*n*-hexane : EA = 10:1);

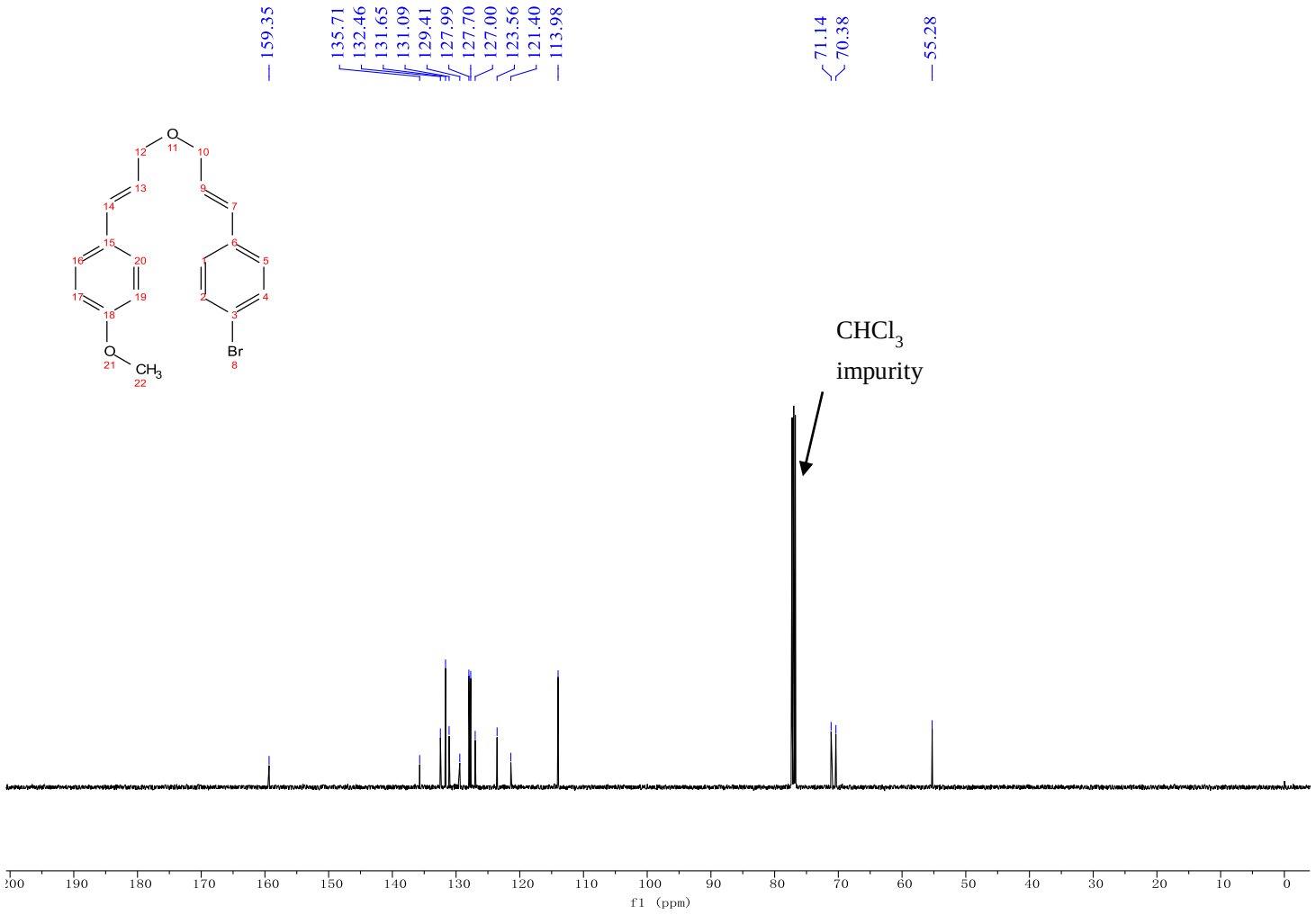
^{1}H NMR (500 MHz, Chloroform-*d*) δ 6.98 (t, *J* = 8.5 Hz, 1H), 6.63 – 6.43 (m, 2H), 5.34 (s, 1H), 3.67 (s, 3H), 2.59 (q, *J* = 9.9 Hz, 1H), 2.09 (s, 2H), 2.04 – 1.93 (m, 1H), 1.87 (dd, *J* = 10.5, 5.3 Hz, 1H), 1.81 – 1.66 (m, 1H), 1.60 (s, 3H), 0.66 (d, *J* = 6.4 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 162.49, 160.55, 158.77, 158.68, 133.82, 129.08, 129.02, 124.28, 124.16, 120.72, 109.97, 109.95, 101.44, 101.22, 55.41, 39.74, 33.50, 33.06, 23.43, 19.90.

HRMS (ESI) m/z calcd for $[C_{15}H_{20}FO]^+$ $[M+H]^+$: 235.1493, found 235.1495.

S4. The spectra of the compounds

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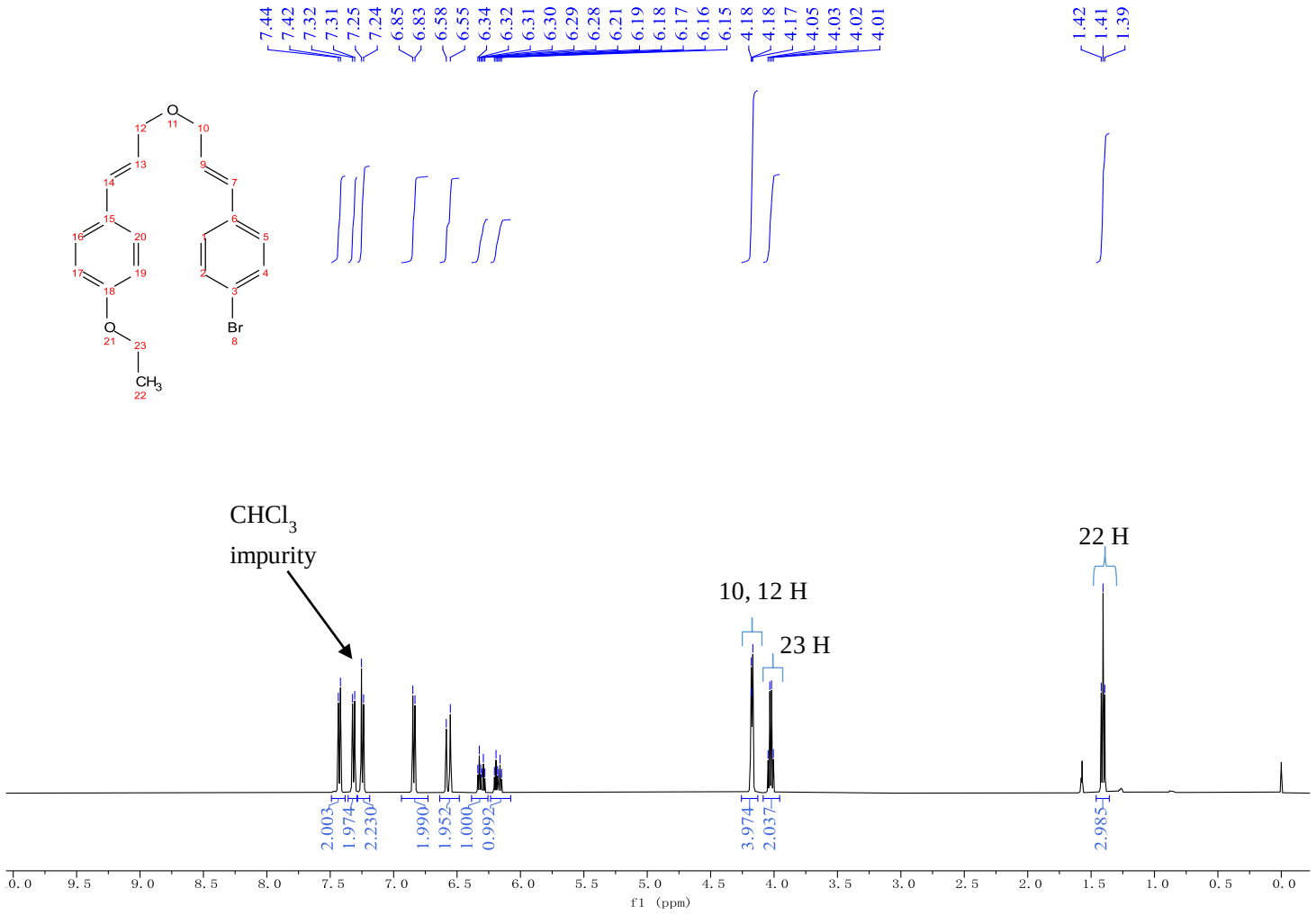
131.65
131.09
129.41
127.99
127.70
127.00
123.56
121.40
113.98
71.14
70.38
55.28
O
11
12
10
13
9
14
7
15
6
16
20
1
5
17
19
2
4
18
3
O
21
CH3
22
Br
8
CHCl3
impurity
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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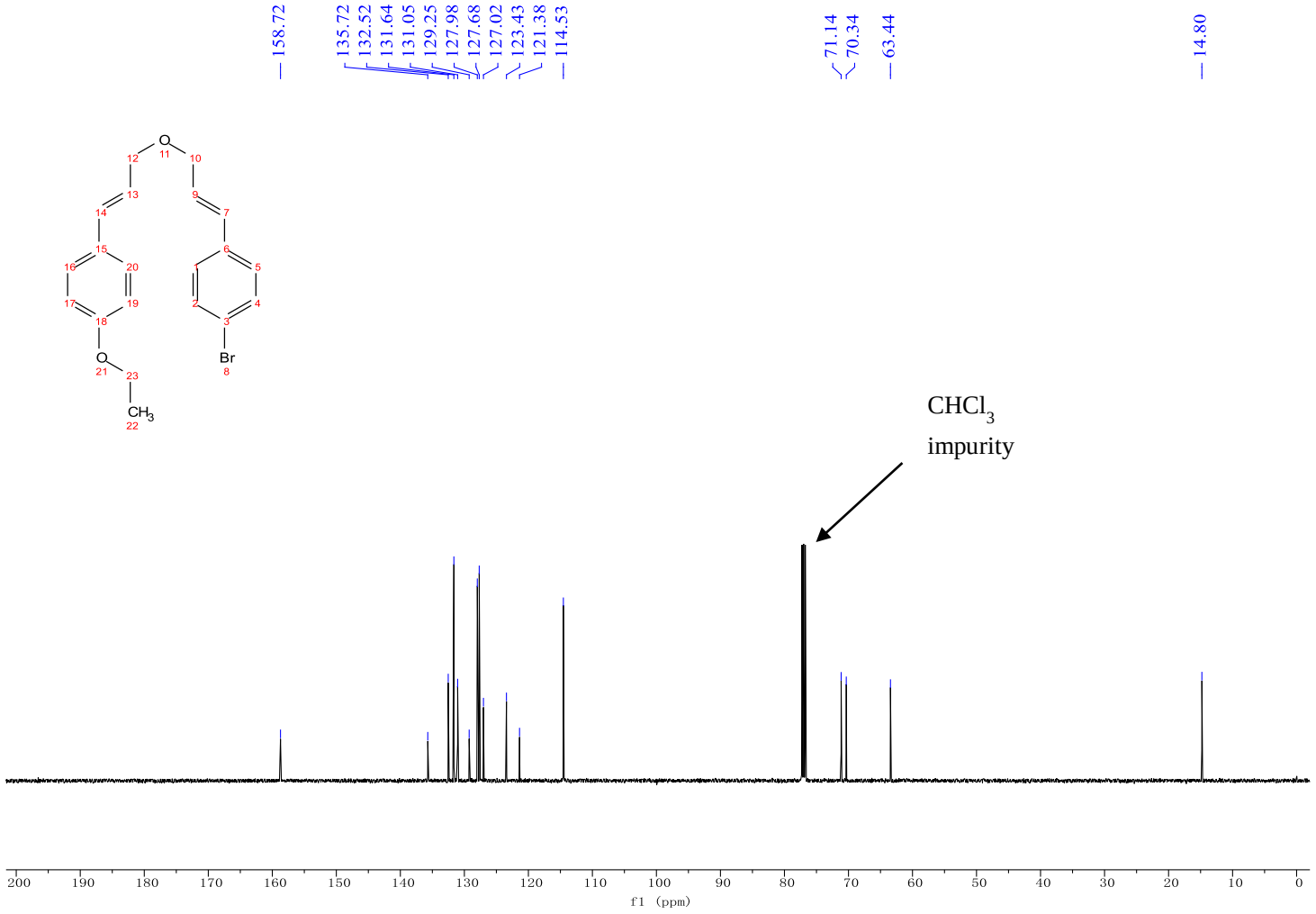
7.42
7.32
7.31
7.25
7.24
6.85
6.83
6.58
6.55
6.34
6.32
6.31
6.30
6.29
6.28
6.21
6.19
6.18
6.17
6.16
6.15
4.18
4.18
4.17
4.05
4.03
4.02
4.01
1.42
1.41
1.39
Br
CHCl3
impurity
10, 12 H
23 H
22 H
2.003
1.974
2.230
1.990
1.952
1.000
0.992
3.974
2.037
2.985
f1 (ppm)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132.52
131.64
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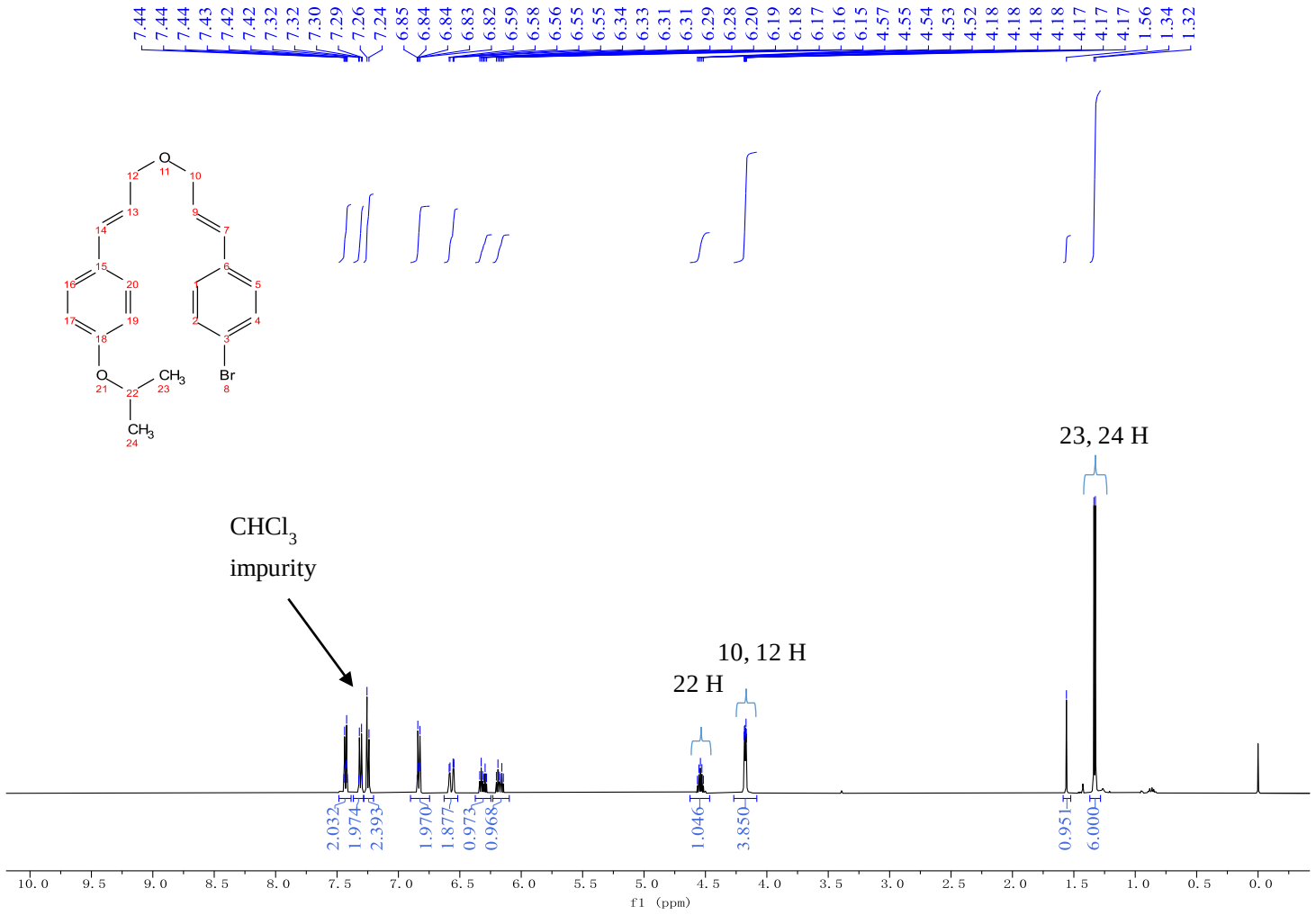
129.25
127.98
127.68
127.02
123.43
121.38
114.53
71.14
70.34
63.44
14.80
O
11
12
10
13
9
14
7
15
6
16
20
1
5
17
19
2
4
18
3
O
21
Br
8
23
CH3
22
CHCl3
impurity
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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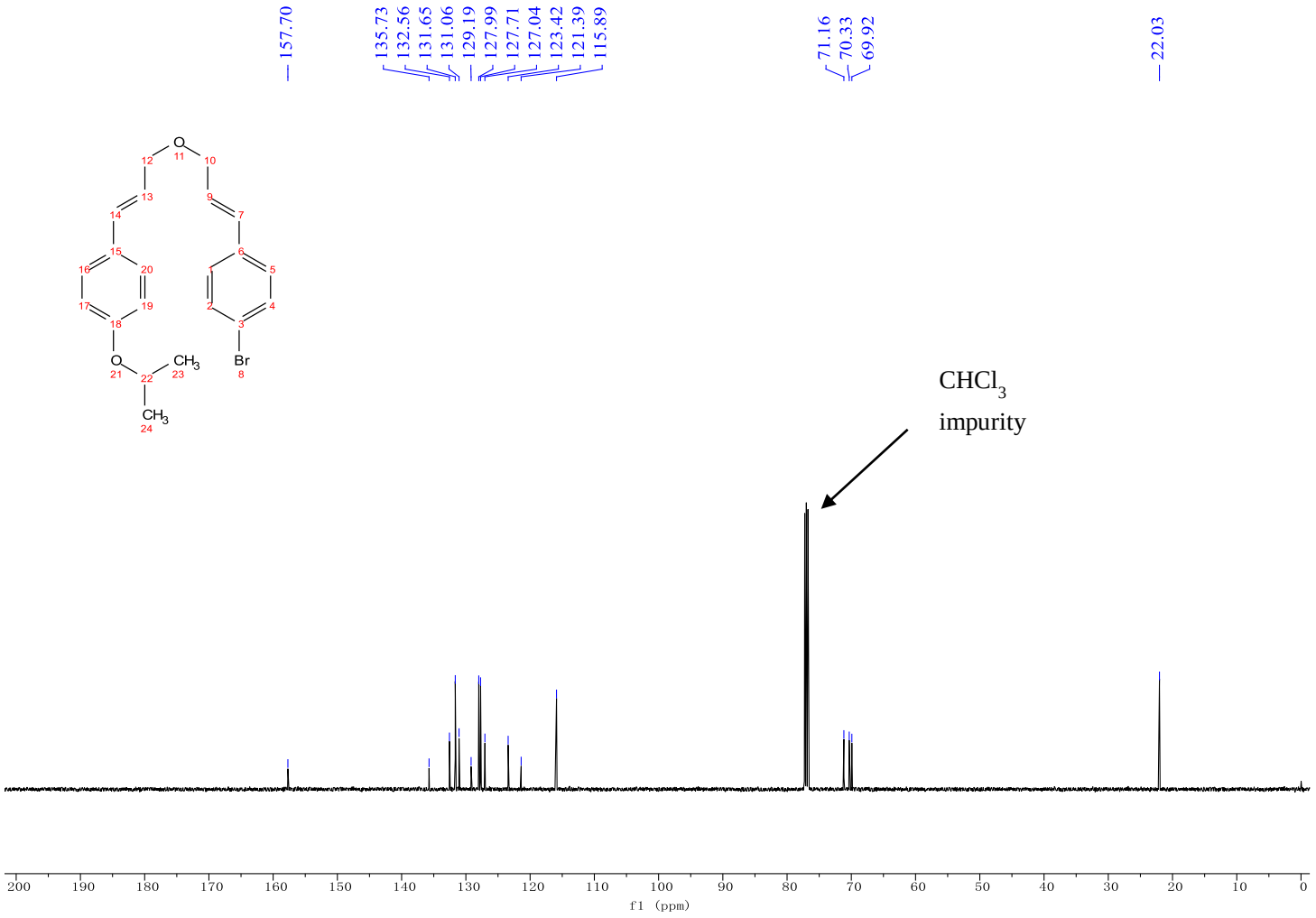
O
11
12
10
13
9
14
7
15
6
16
20
1
5
17
19
2
4
18
3
O
21
22
CH3
23
Br
8
CH3
24
23, 24 H
CHCl3
impurity
10, 12 H
22 H
2.032
1.974
2.393
1.970
1.877
0.973
0.968
1.046
3.850
0.951
6.000
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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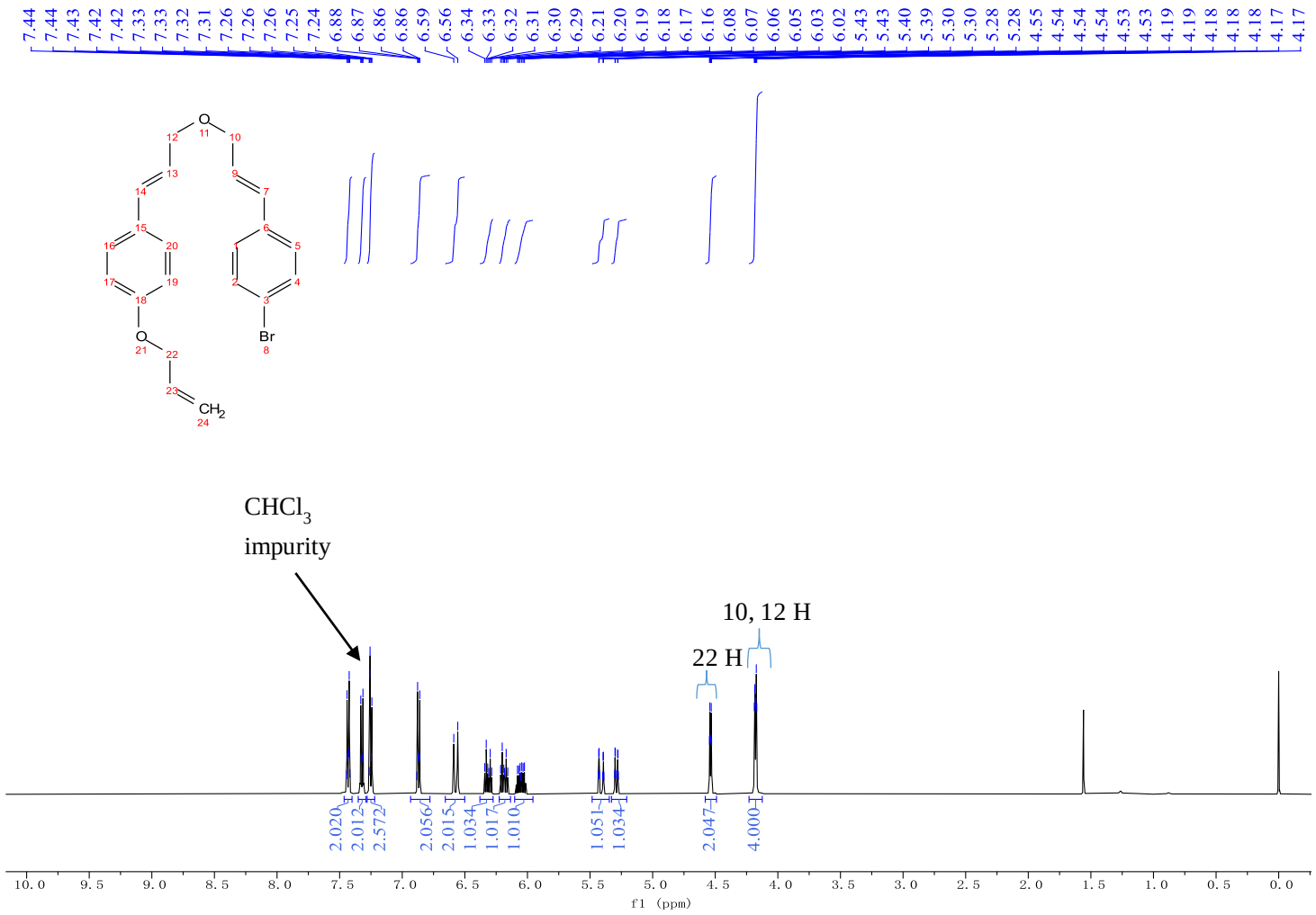
132.56
131.65
131.06
129.19
127.99
127.71
127.04
123.42
121.39
115.89
71.16
70.33
69.92
22.03
O
11
12
10
13
9
14
7
15
6
16
20
5
17
19
2
4
18
3
O
21
22
CH3
23
Br
8
CH3
24
CHCl3
impurity
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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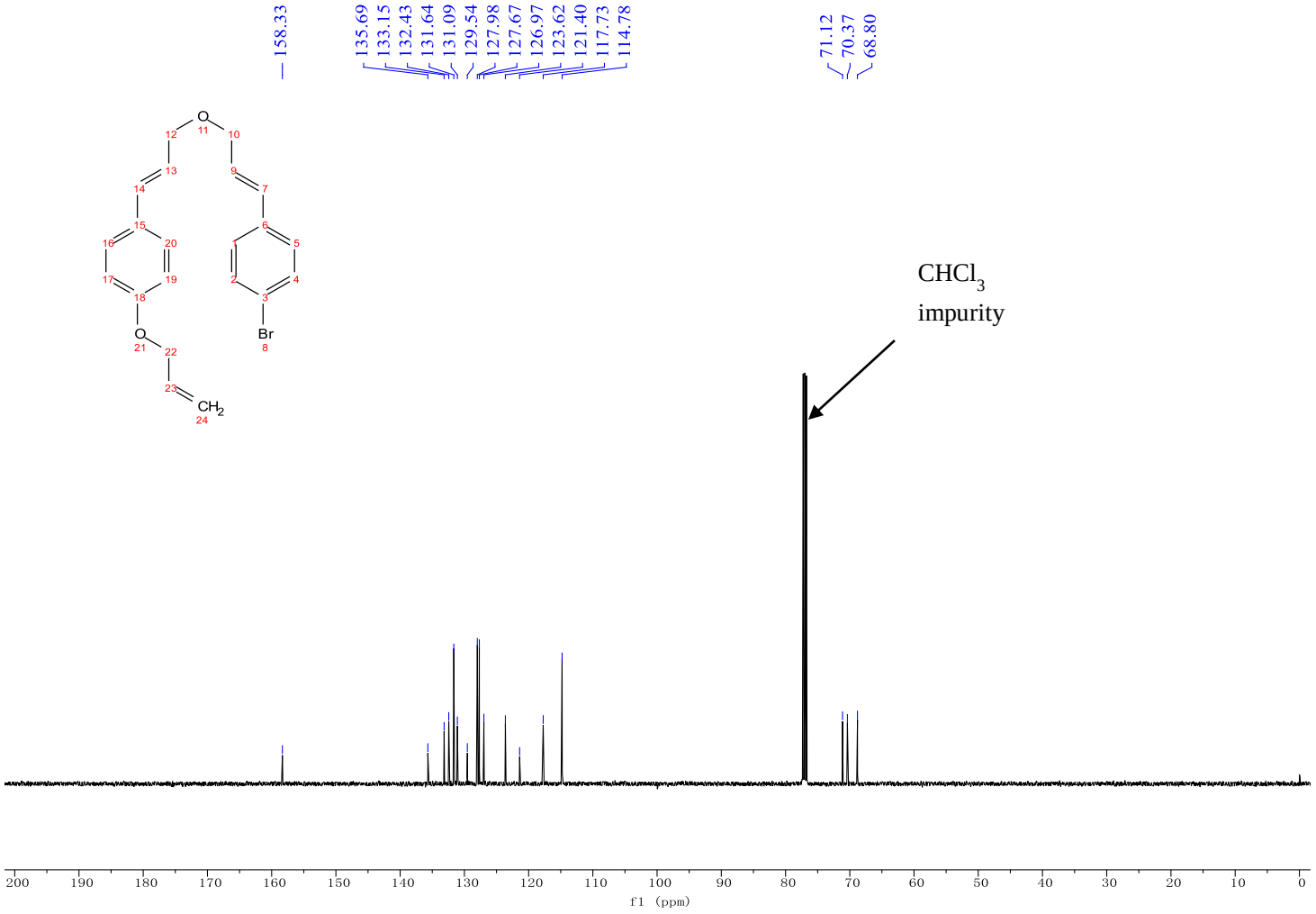
O
11
12
10
13
9
14
7
6
15
16
20
1
5
17
19
2
4
18
3
O
21
22
Br
8
23
CH2
24
CHCl3
impurity
10, 12 H
22 H
2.020
2.012
2.572
2.056
2.015
1.034
1.017
1.010
1.051
1.034
2.047
4.000
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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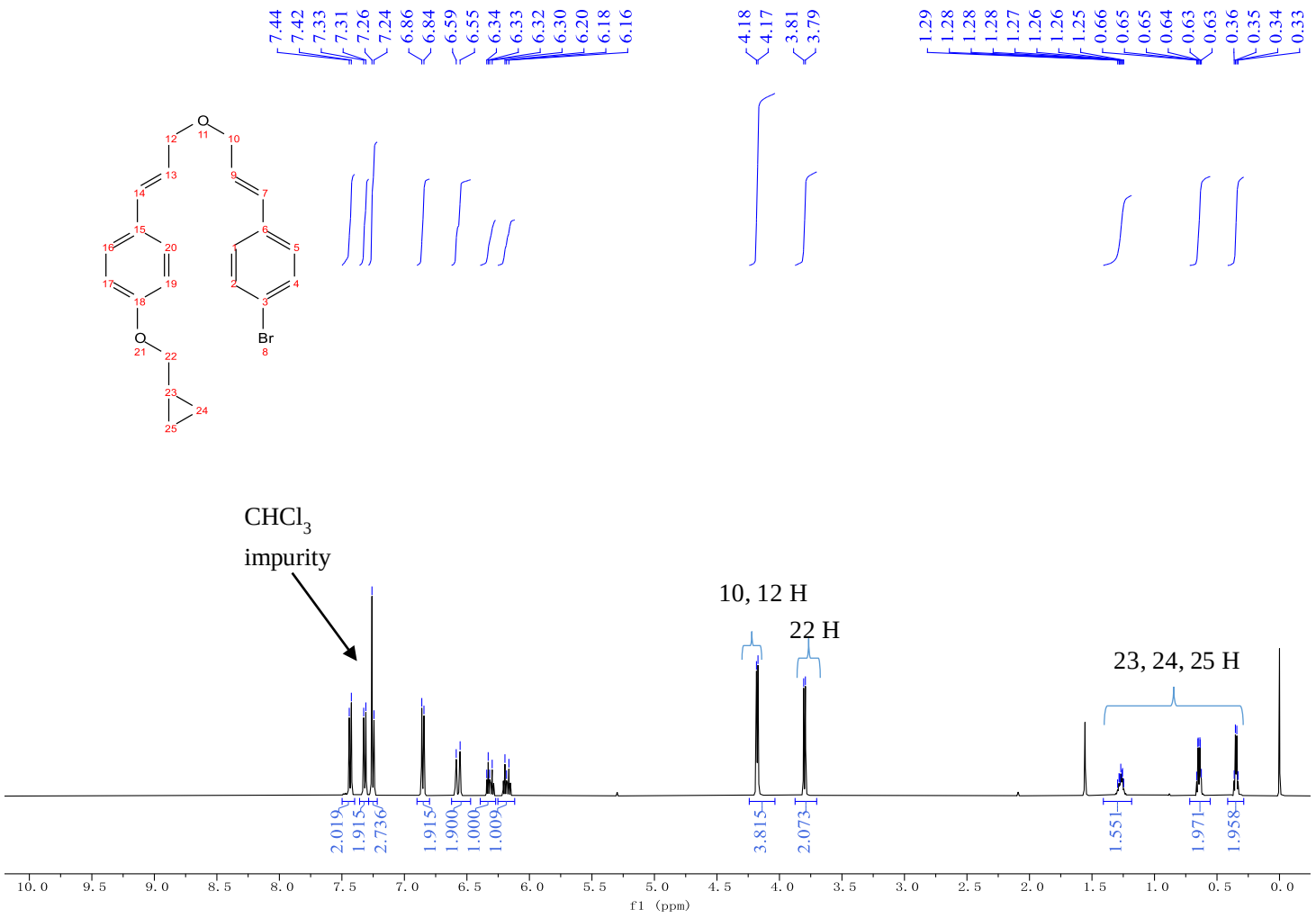
132.43
131.64
131.09
129.54
127.98
127.67
126.97
123.62
121.40
117.73
114.78
71.12
70.37
68.80
CHCl3
impurity
Br
CH2
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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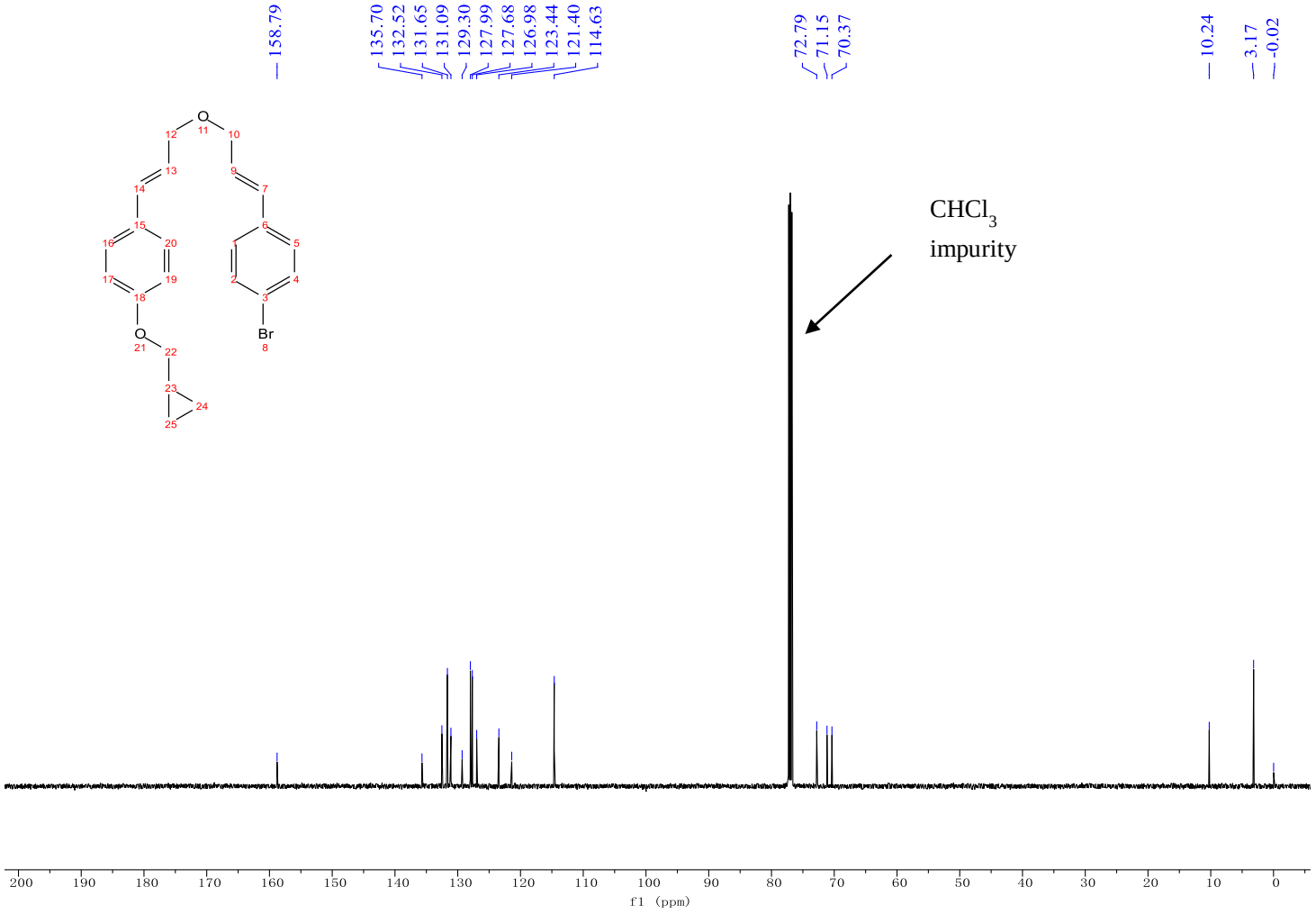
7.42
7.33
7.31
7.26
7.24
6.86
6.84
6.59
6.55
6.34
6.33
6.32
6.30
6.20
6.18
6.16
4.18
4.17
3.81
3.79
1.29
1.28
1.28
1.28
1.27
1.26
1.26
1.25
0.66
0.65
0.64
0.63
0.63
0.36
0.35
0.34
0.33
Br
CHCl3
impurity
10, 12 H
22 H
23, 24, 25 H
2.019
1.915
2.736
1.915
1.900
1.000
1.009
3.815
2.073
1.551
1.971
1.958
f1 (ppm)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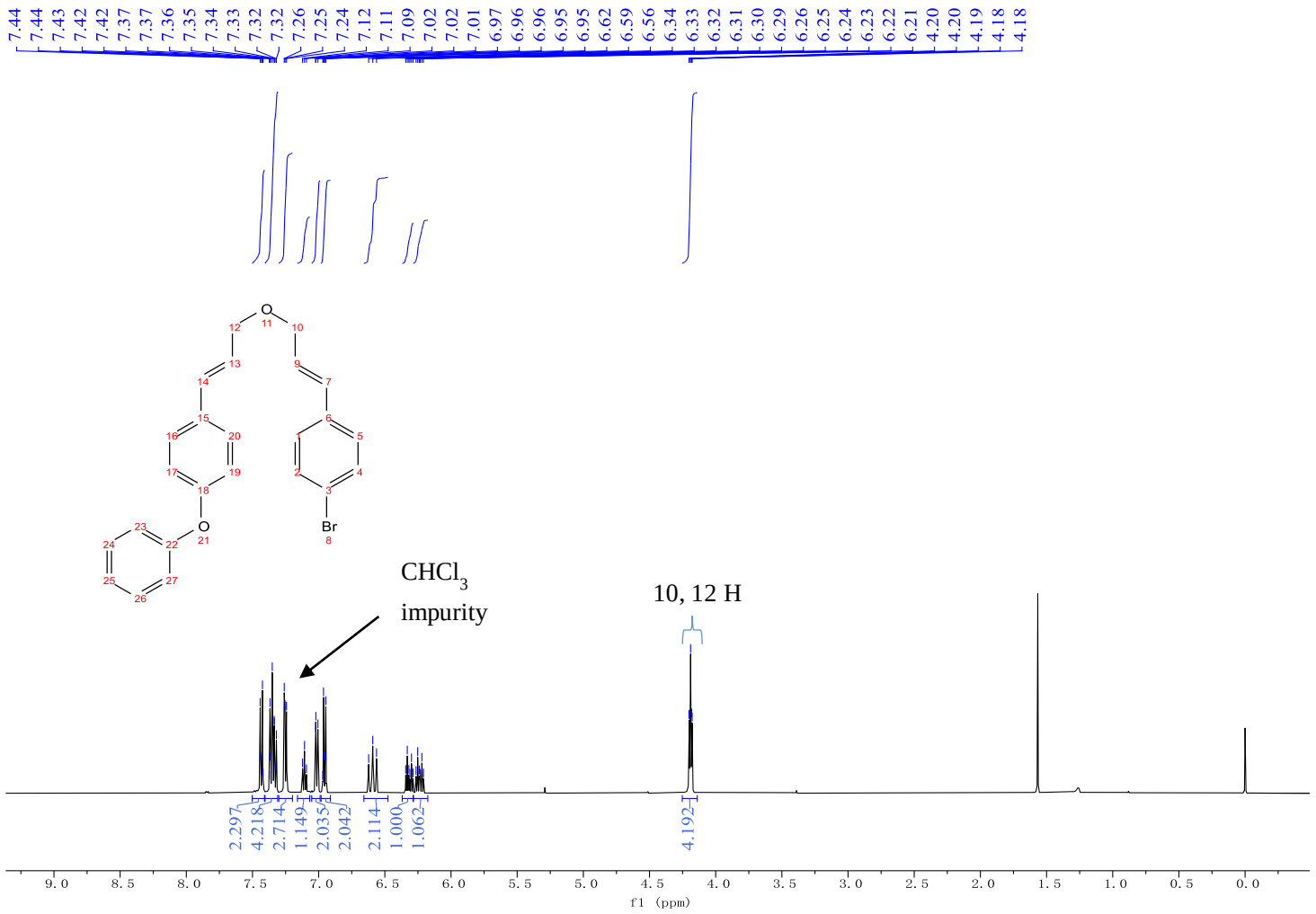
132.52
131.65
131.09
129.30
127.99
127.68
126.98
123.44
121.40
114.63
72.79
71.15
70.37
10.24
3.17
-0.02
CHCl3
impurity
Br
f1 (ppm)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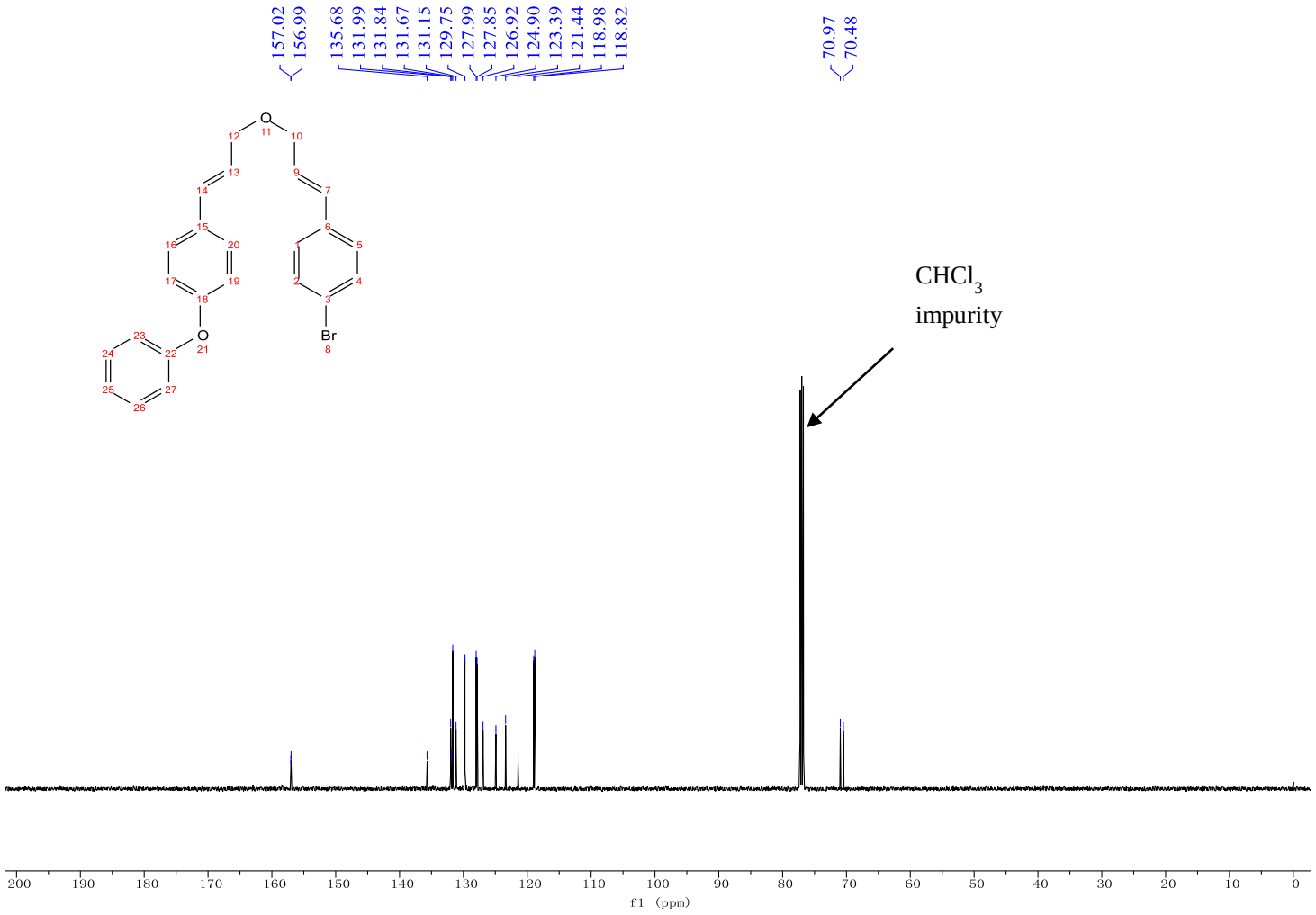
O
Br
CHCl3
impurity
10, 12 H
2.297 4.218 2.714 1.149 2.035 2.042 2.114 1.000 1.062 4.192
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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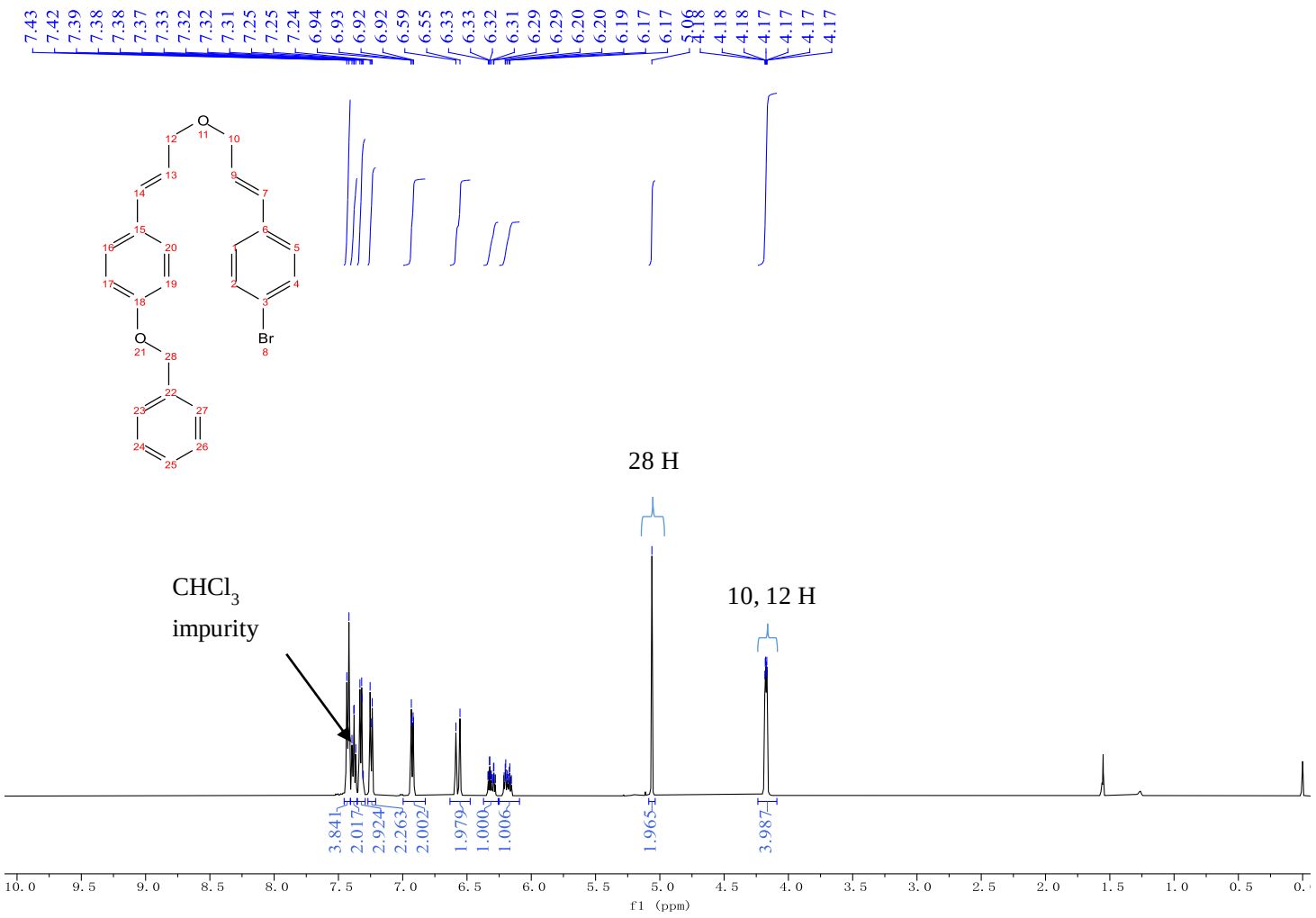
131.67
131.15
129.75
127.99
127.85
126.92
124.90
123.39
121.44
118.98
118.82
70.97
70.48
CHCl3
impurity
Br
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.38
7.37
7.33
7.32
7.32
7.31
7.25
7.25
7.24
6.94
6.93
6.92
6.92
6.59
6.55
6.33
6.33
6.32
6.31
6.29
6.29
6.20
6.20
6.19
6.17
6.17
5.06
4.18
4.18
4.17
4.17
4.17
4.17
Br
28 H
CHCl3
impurity
10, 12 H
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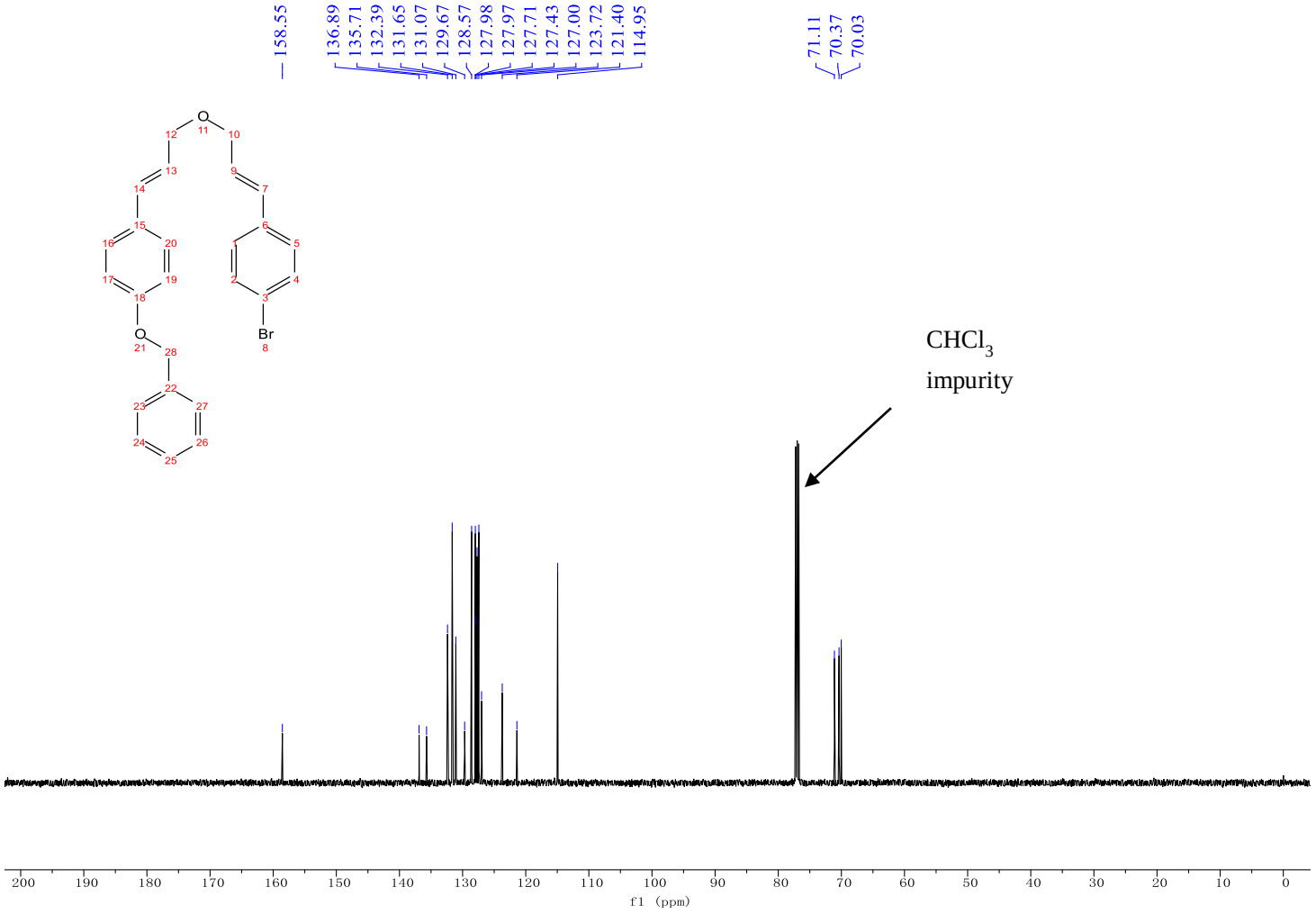
2.924
2.263
2.002
1.979
1.000
1.006
1.965
3.987
f1 (ppm)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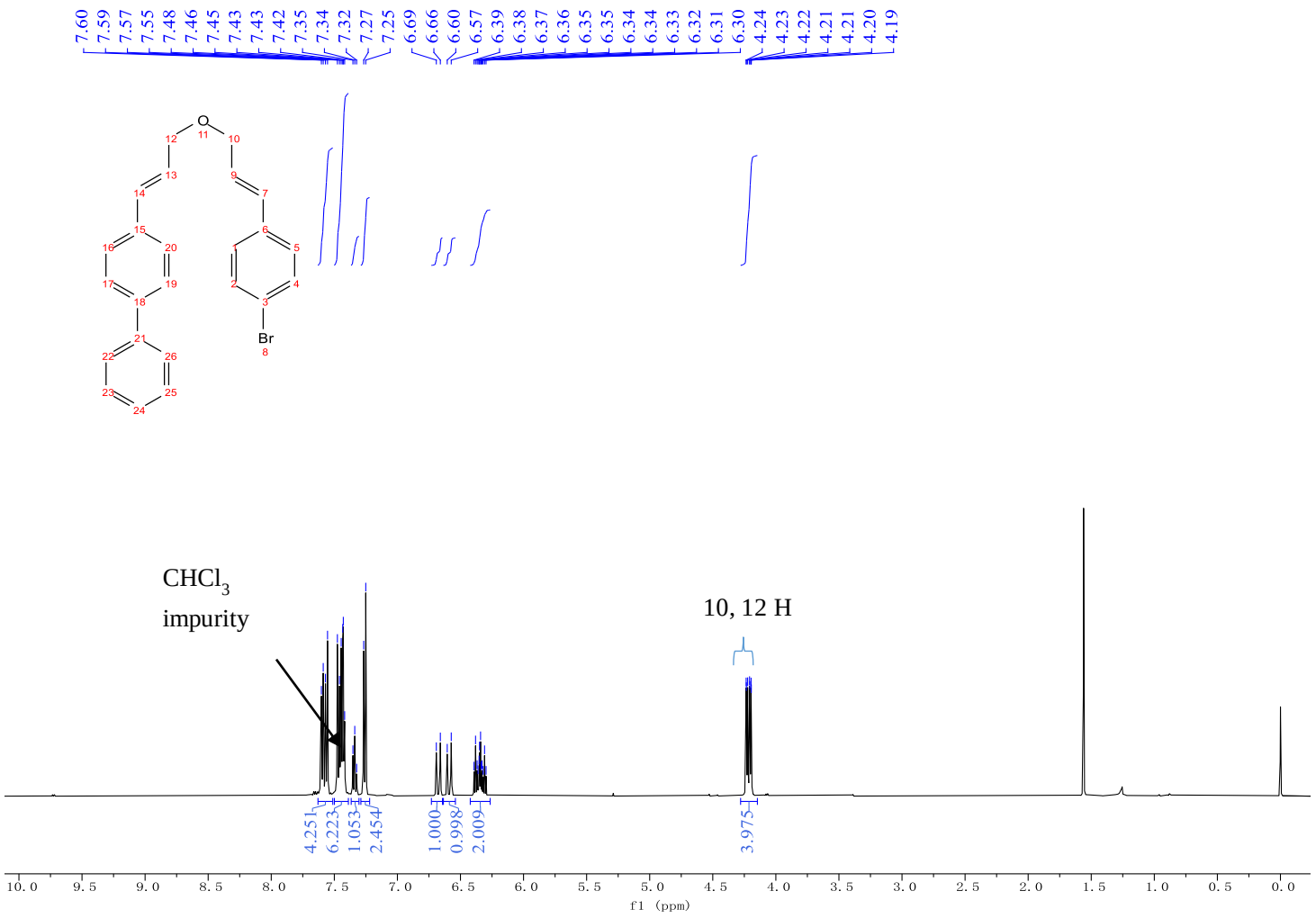
132.39
131.65
131.07
129.67
128.57
127.98
127.97
127.71
127.43
127.00
123.72
121.40
114.95
71.11
70.37
70.03
CHCl3
impurity
Br
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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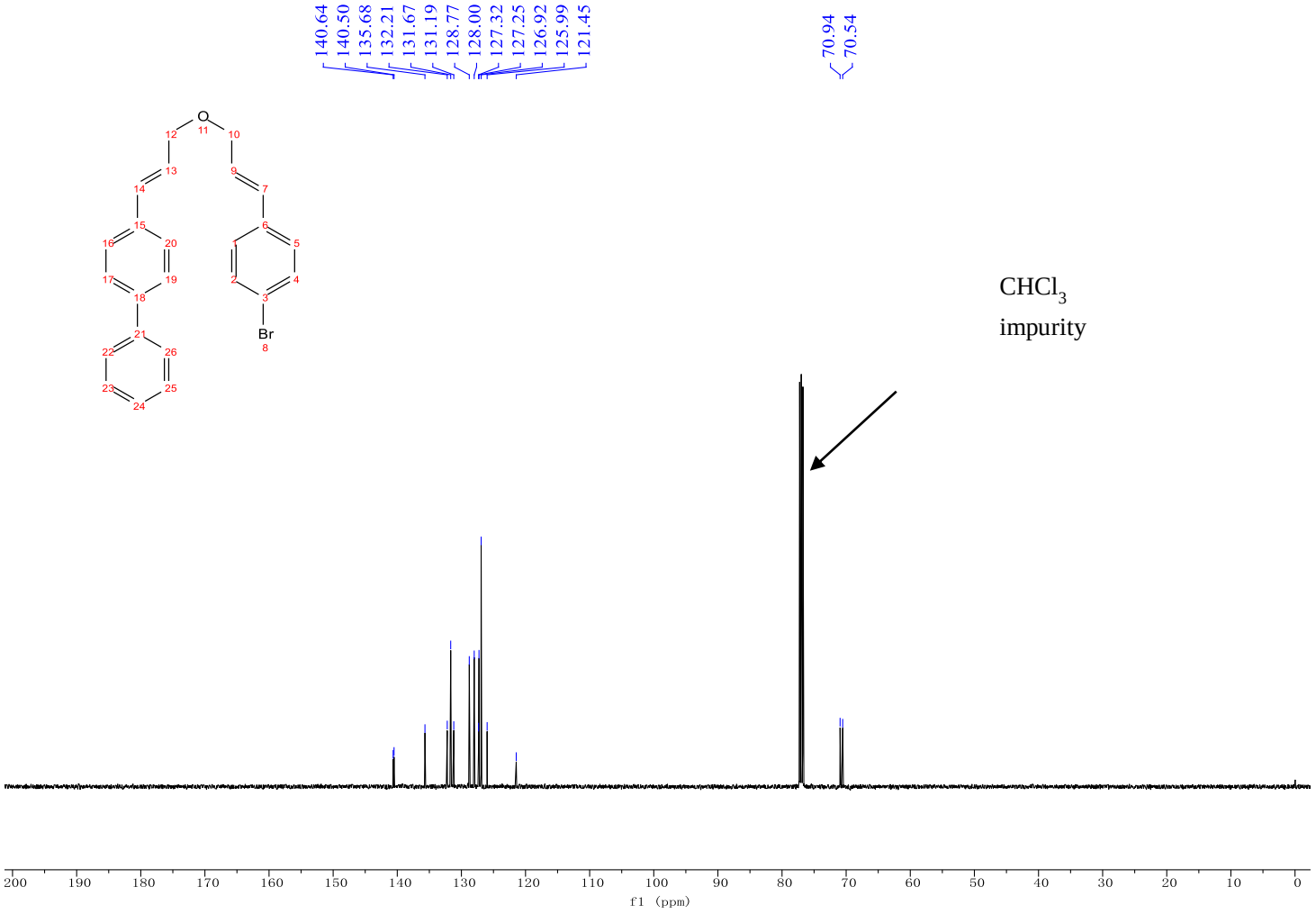
7.32
7.27
7.25
6.69
6.66
6.60
6.57
6.39
6.38
6.37
6.36
6.35
6.35
6.34
6.34
6.33
6.32
6.31
6.30
4.24
4.23
4.22
4.21
4.21
4.20
4.19
Br
CHCl3
impurity
10, 12 H
4.251
6.223
1.053
2.454
1.000
0.998
2.009
3.975
f1 (ppm)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140.50
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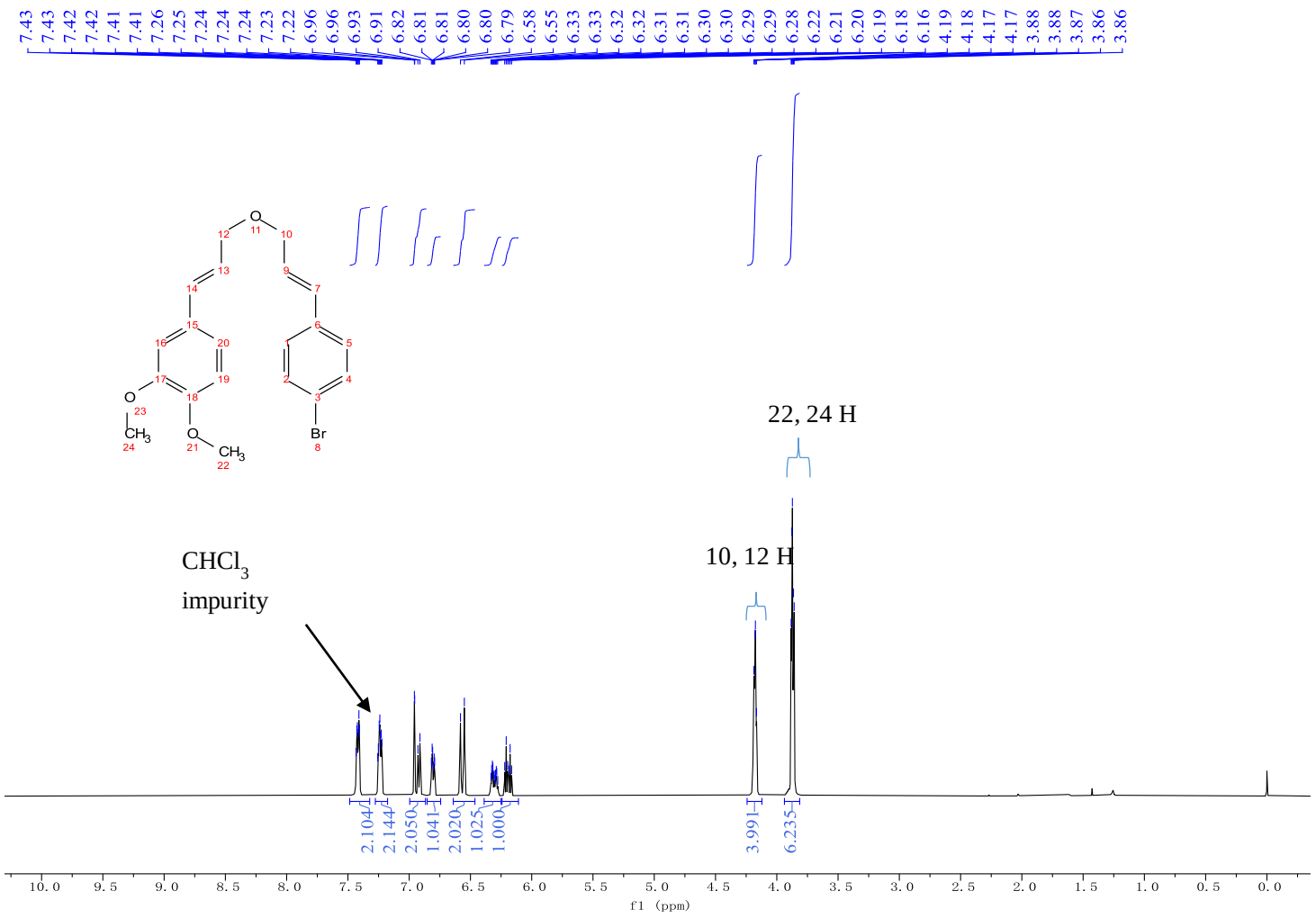
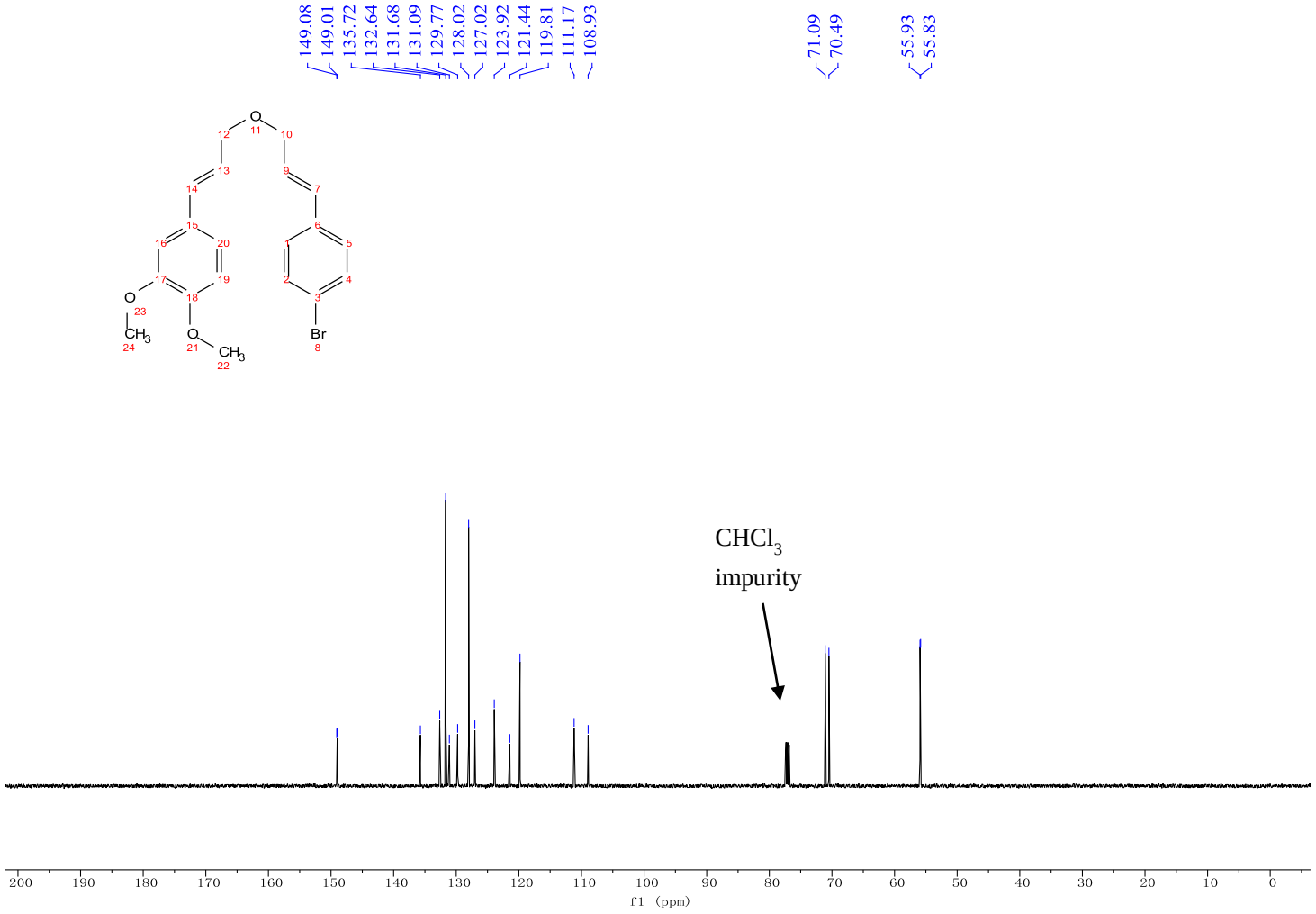
132.21
131.67
131.19
128.77
128.00
127.32
127.25
126.92
125.99
121.45
70.94
70.54
CHCl3
impurity
Br
f1 (ppm)


7.43 7.43 7.42 7.42 7.41 7.41 7.26 7.25 7.24 7.24 7.24 7.23 7.22 6.96 6.96 6.93 6.91 6.82 6.81 6.81 6.80 6.80 6.79 6.58 6.55 6.33 6.33 6.32 6.32 6.31 6.31 6.30 6.30 6.29 6.29 6.28 6.22 6.21 6.20 6.19 6.18 6.16 4.19 4.18 4.17 4.17 3.88 3.88 3.87 3.86 3.86
22, 24 H
10, 12 H
CHCl3
impurity
2.104 2.144 2.050 1.041 2.020 1.025 1.000 3.991 6.235
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
f1 (ppm)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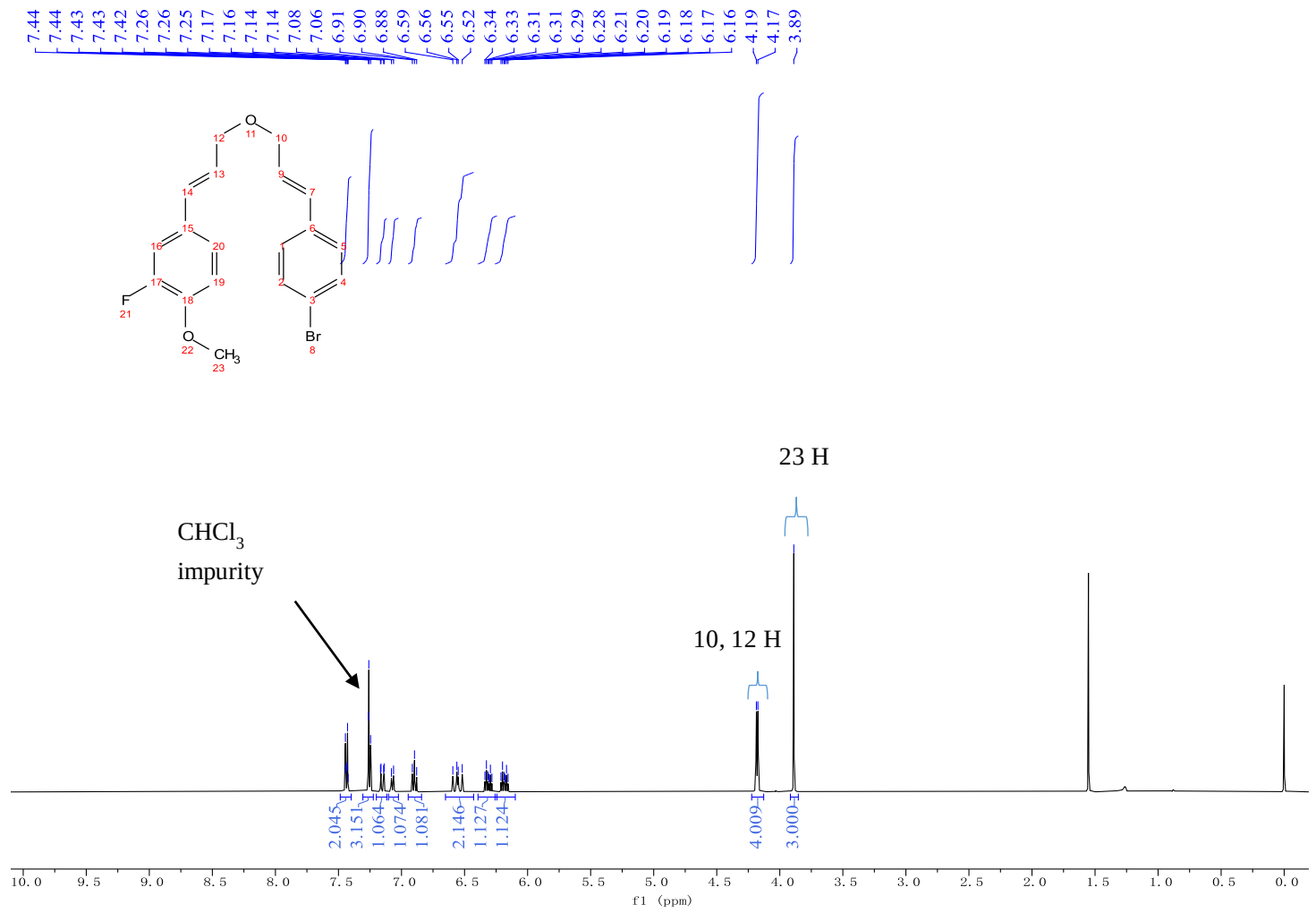
131.09
129.77
128.02
127.02
123.92
121.44
119.81
111.17
108.93
71.09
70.49
55.93
55.83
O
11
12
10
13
9
14
7
15
6
16
20
5
1
17
19
2
4
18
3
O
23
CH3
24
O
21
CH3
22
Br
8
CHCl3
impurity
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.44
7.43
7.43
7.42
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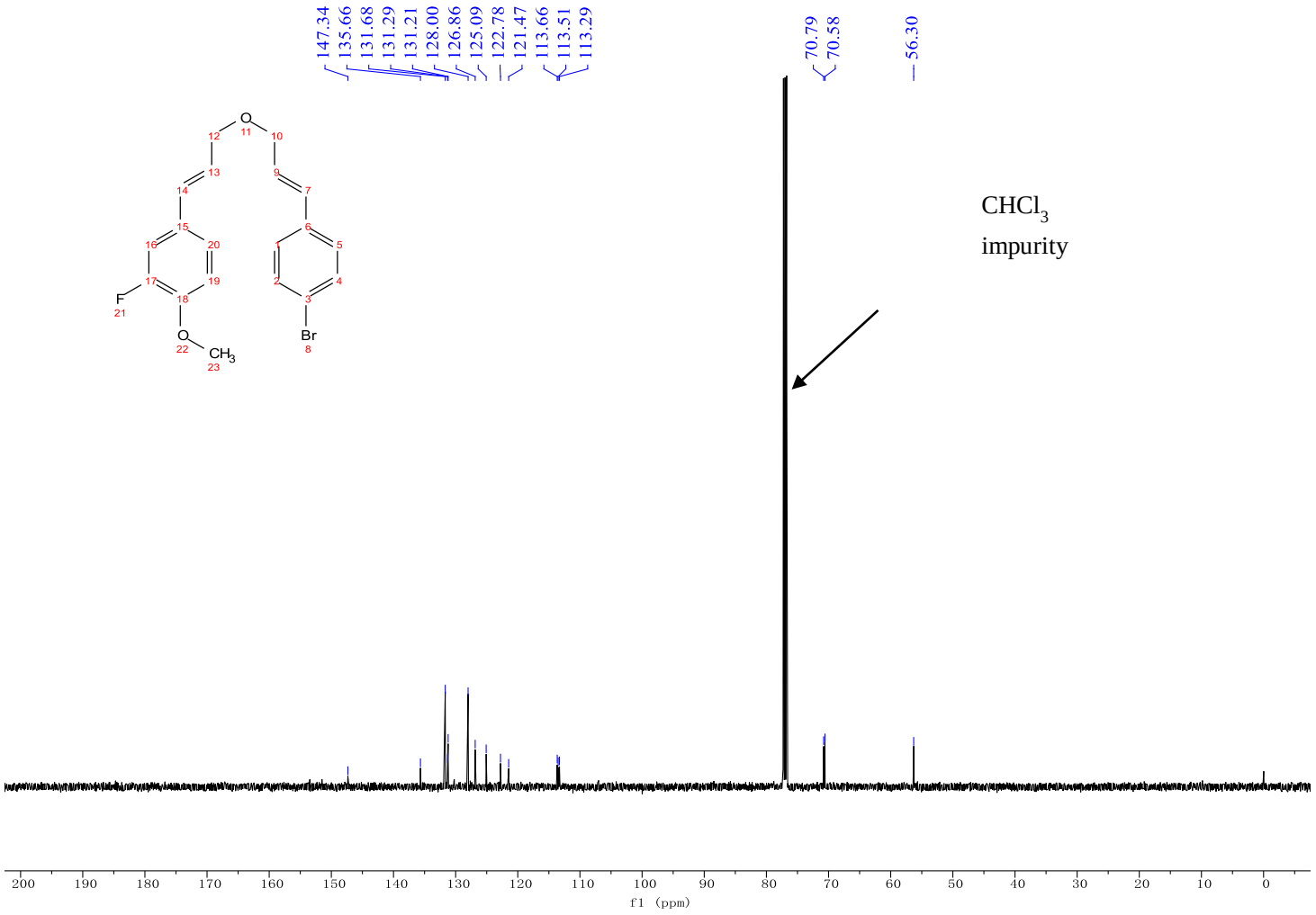
7.26
7.25
7.17
7.16
7.14
7.14
7.08
7.06
6.91
6.90
6.88
6.59
6.56
6.55
6.52
6.34
6.33
6.31
6.31
6.29
6.28
6.21
6.20
6.19
6.18
6.17
6.16
4.19
4.17
3.89
O
11
12
10
13
9
14
7
15
6
16
20
1
5
17
19
2
4
18
3
F
21
O
22
CH3
23
Br
8
23 H
CHCl3
impurity
10, 12 H
2.045
3.151
1.064
1.074
1.081
2.146
1.127
1.124
4.009
3.000
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
f1 (ppm)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128.00
126.86
125.09
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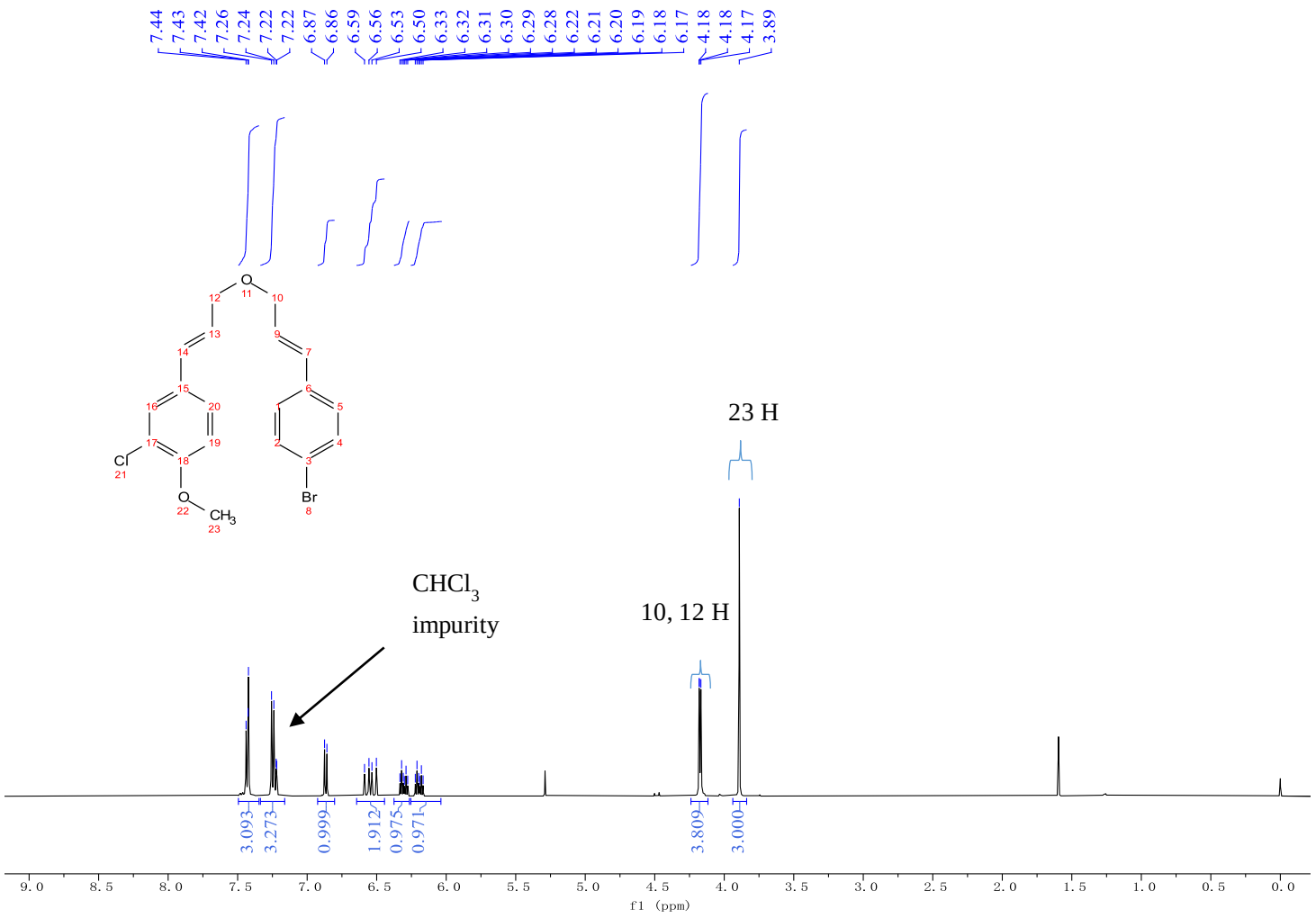
121.47
113.66
113.51
113.29
70.79
70.58
56.30
CHCl3
impurity
F
21
O
22
CH3
23
Br
8
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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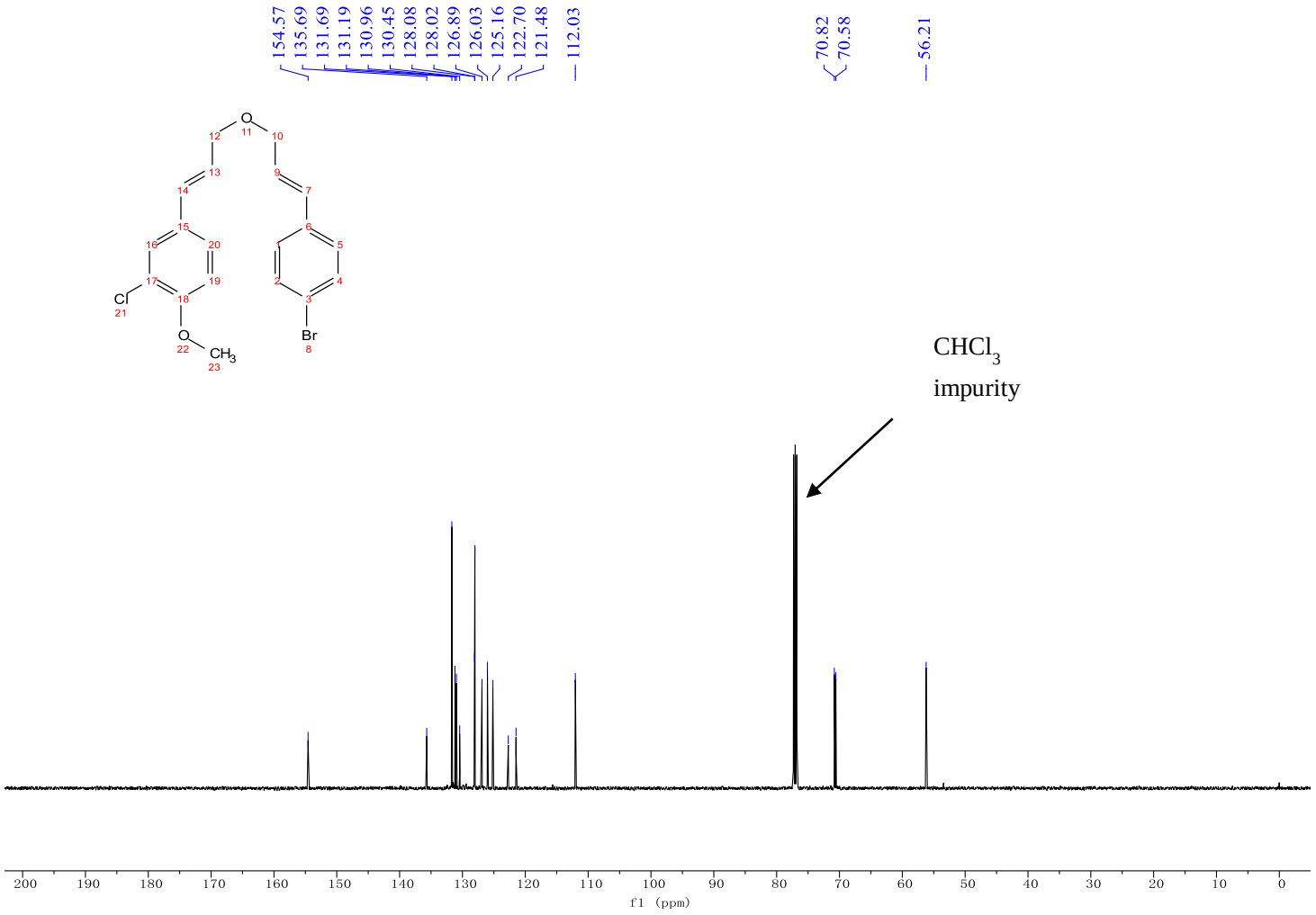
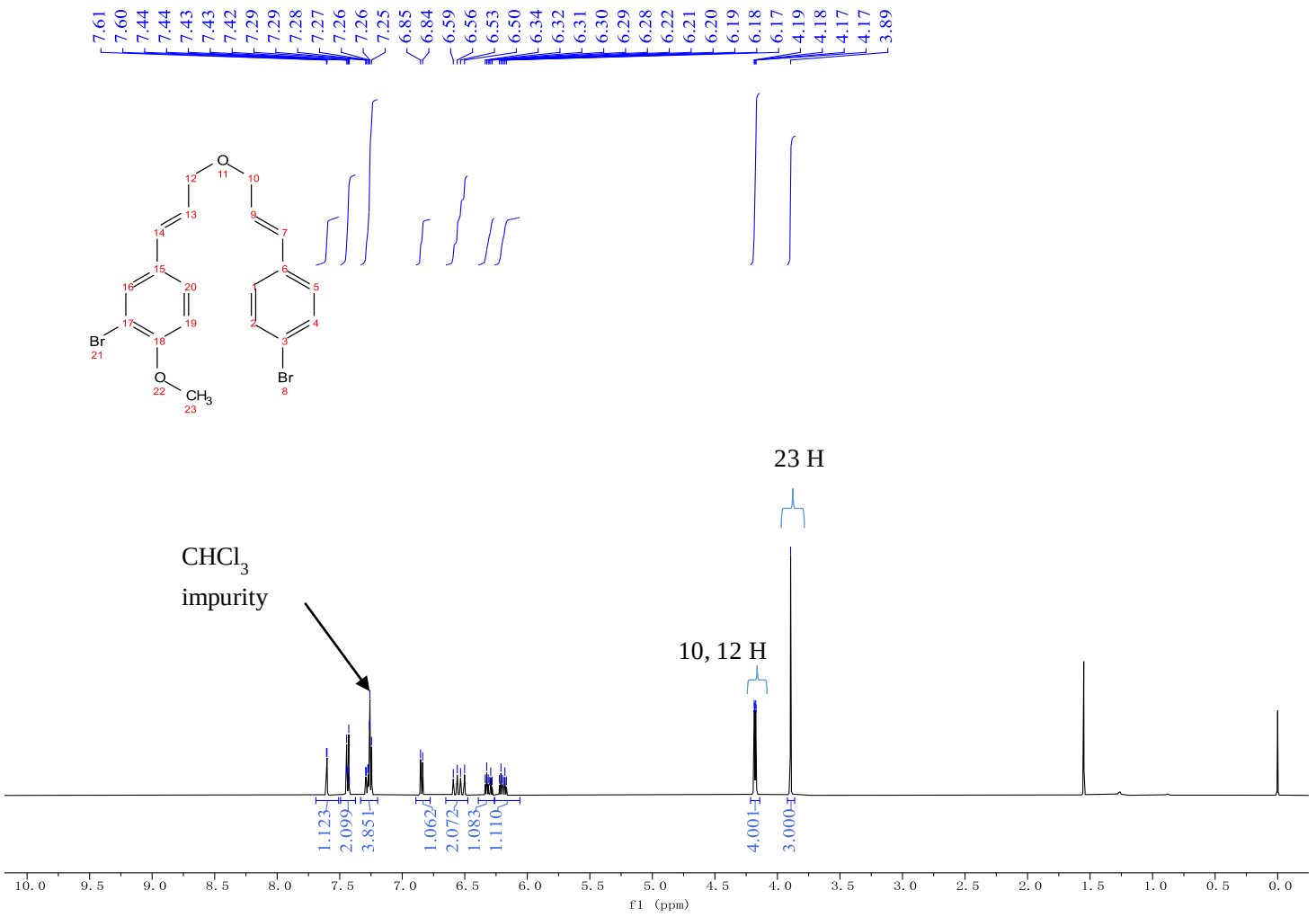
7.42
7.26
7.24
7.22
7.22
6.87
6.86
6.59
6.56
6.53
6.50
6.33
6.32
6.31
6.30
6.29
6.28
6.22
6.21
6.20
6.19
6.18
6.17
4.18
4.18
4.17
3.89
23 H
CHCl3
impurity
10, 12 H
3.093
3.273
0.999
1.912
0.975
0.971
3.809
3.000
f1 (ppm)


154.57
135.69
131.69
131.19
130.96
130.45
128.08
128.02
126.89
126.03
125.16
122.70
121.48
112.03
70.82
70.58
56.21
CHCl3
impurity
f1 (ppm)


7.61
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7.44
7.43
7.43
7.42
7.29
7.29
7.28
7.27
7.26
7.26
7.25
6.85
6.84
6.59
6.56
6.53
6.50
6.34
6.32
6.31
6.30
6.29
6.28
6.22
6.21
6.20
6.19
6.18
6.17
4.19
4.18
4.17
4.17
3.89
Br
21
Br
8
O
22
CH3
23
23 H
CHCl3
impurity
10, 12 H
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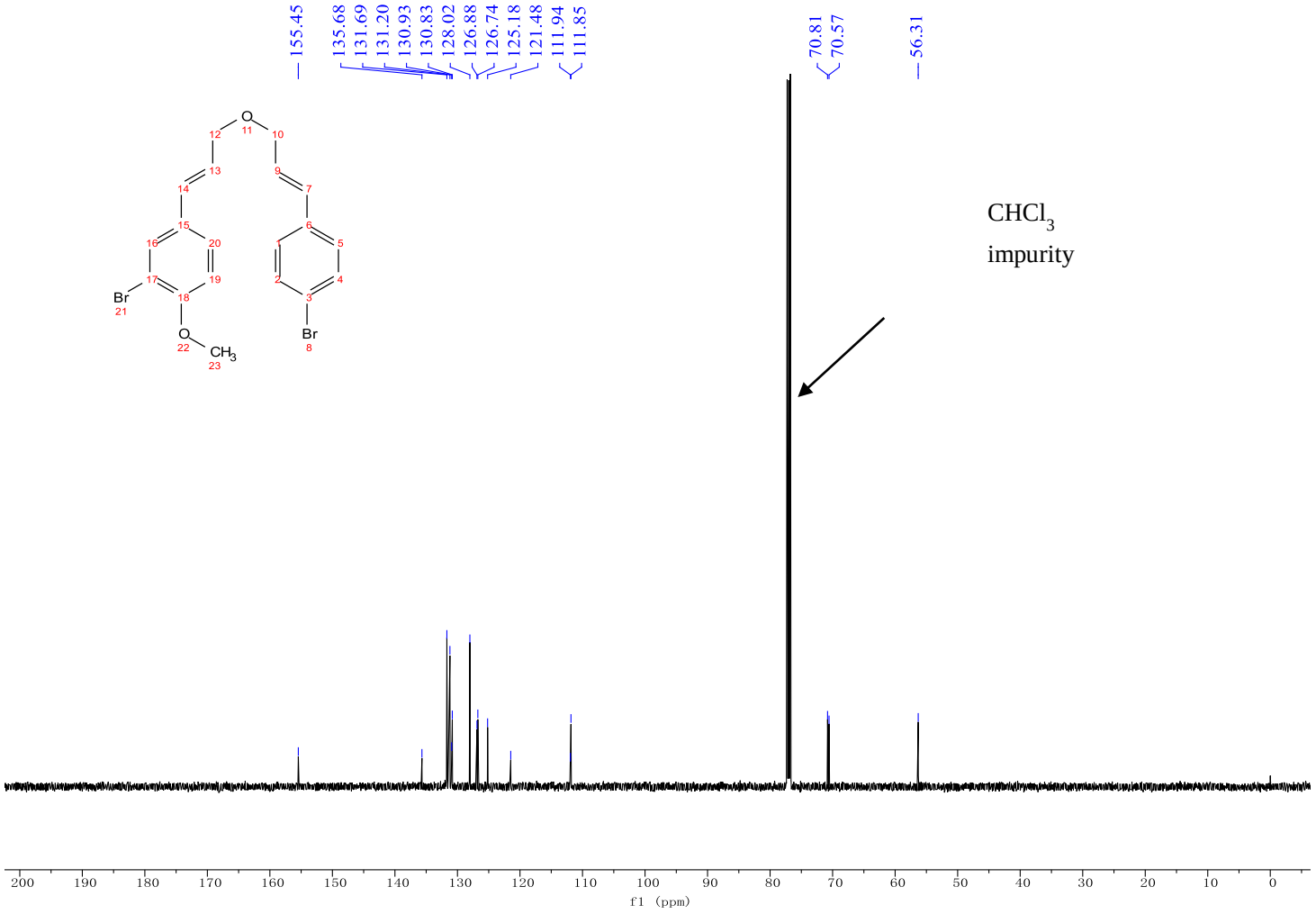
3.851
1.062
2.072
1.083
1.110
4.001
3.000
f1 (ppm)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125.18
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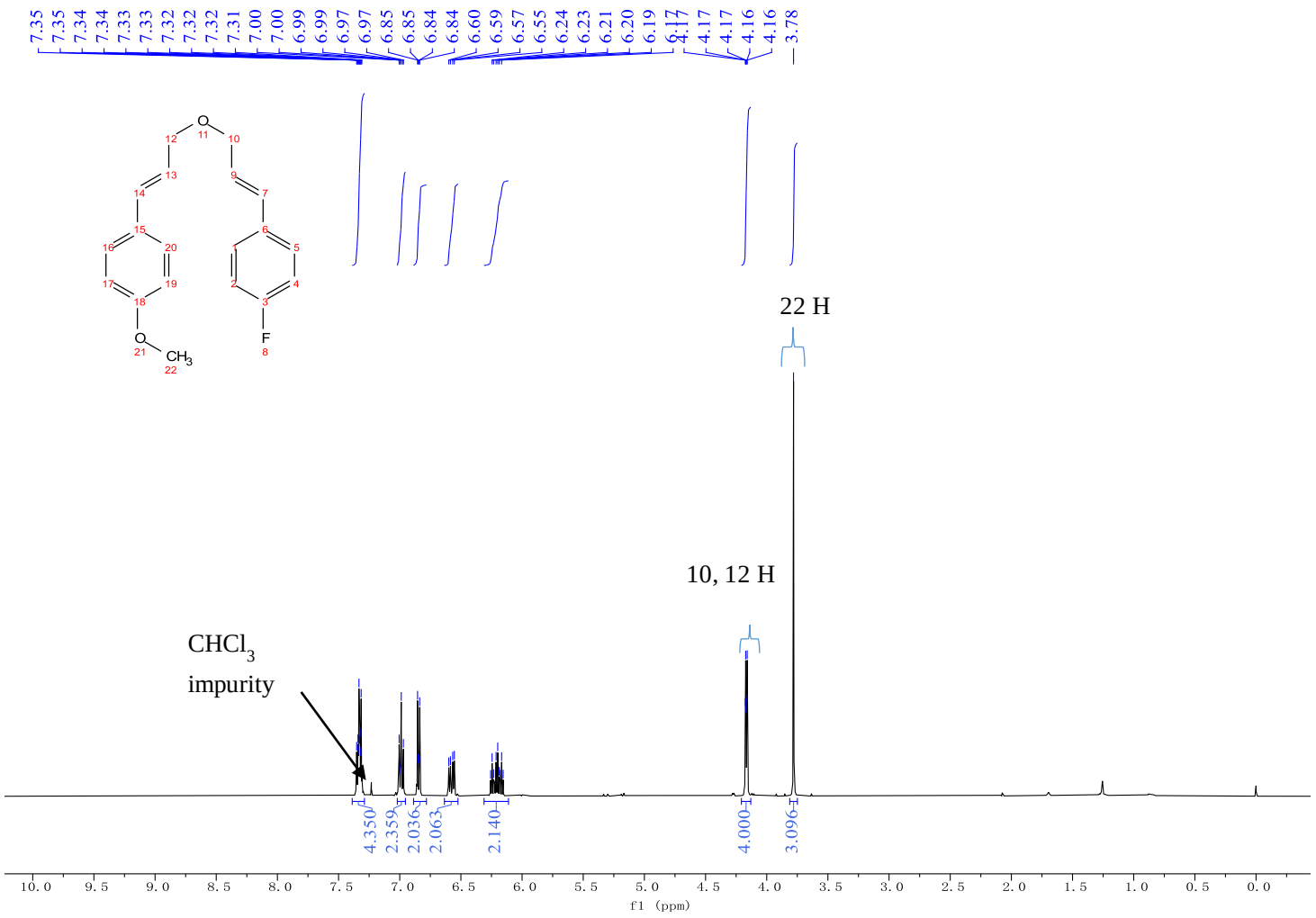
111.85
70.81
70.57
56.31
CHCl3
impurity
Br
Br
O
CH3
f1 (ppm)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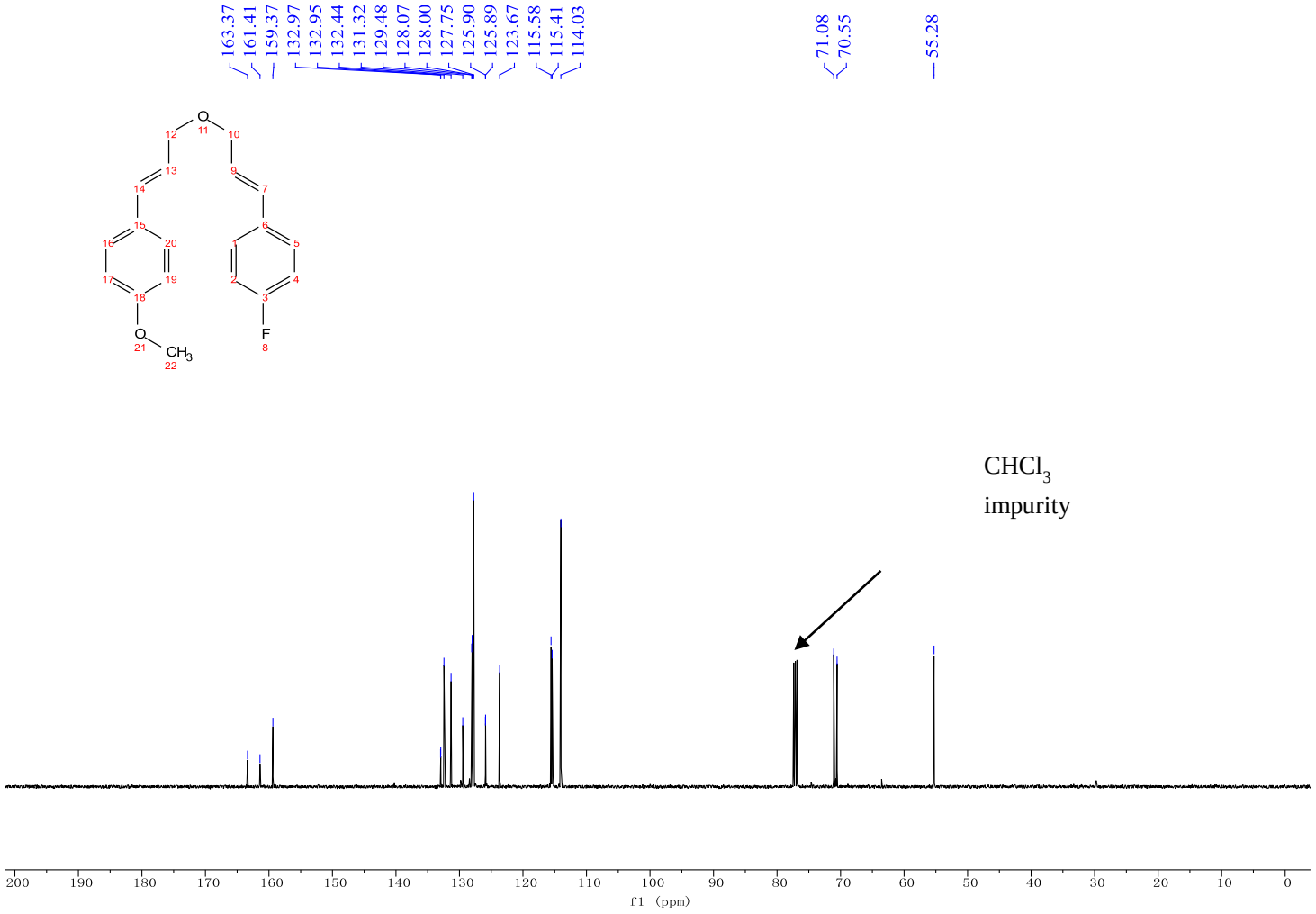
22 H
10, 12 H
CHCl3
impurity
4.350 2.359 2.036 2.063 2.140 4.000 3.096
f1 (ppm)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125.89
123.67
115.58
115.41
114.03
71.08
70.55
55.28
O
11
12
10
13
9
14
7
15
6
16
20
5
1
17
19
2
4
18
3
O
21
CH3
22
F
8
CHCl3
impurity
200
190
180
170
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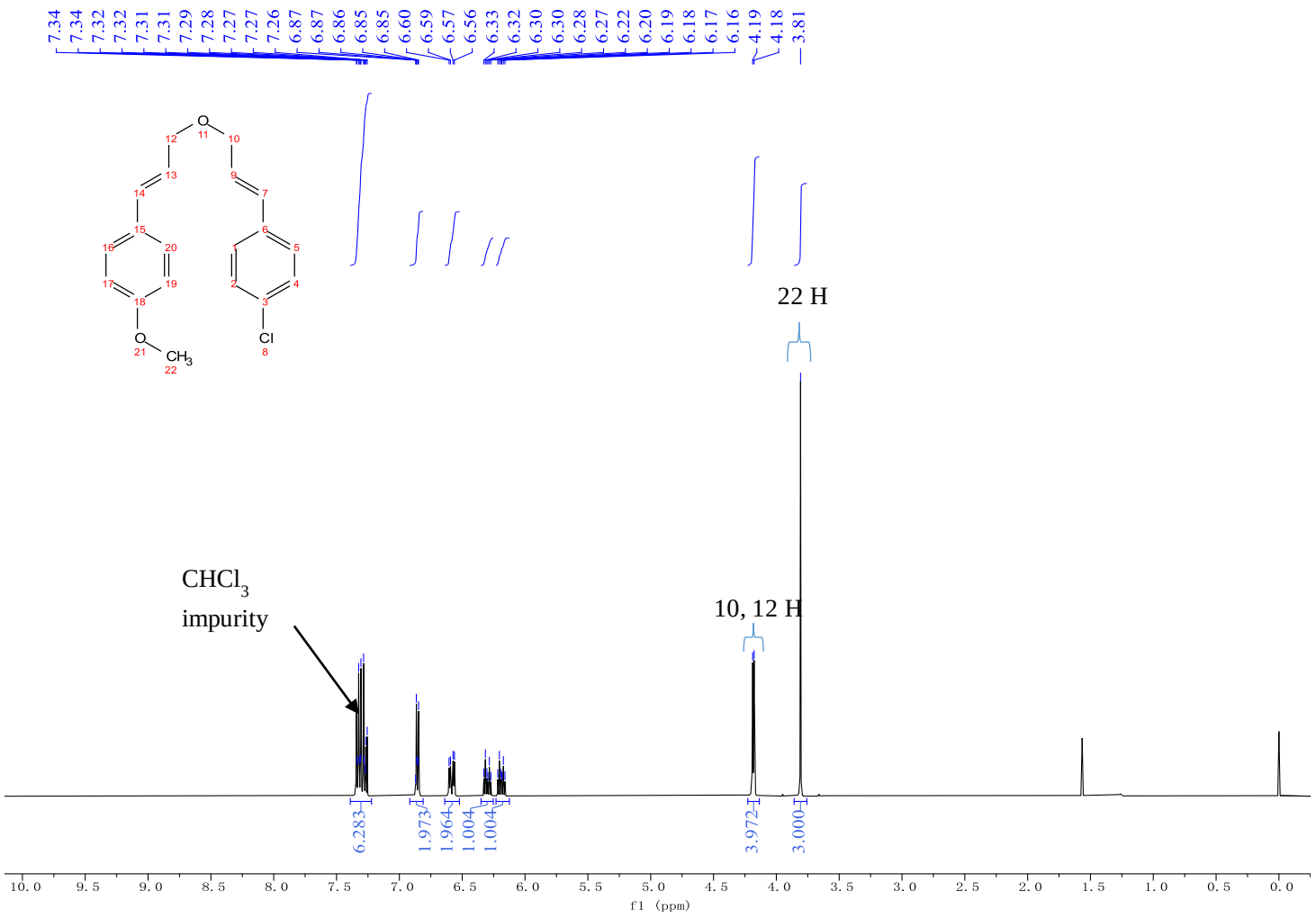
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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6.86
6.85
6.85
6.60
6.59
6.57
6.56
6.33
6.32
6.30
6.30
6.28
6.27
6.22
6.20
6.19
6.18
6.17
6.16
4.19
4.18
3.81
O
11
12
10
13
9
14
7
15
6
16
20
1
5
17
19
2
4
18
3
O
21
CH3
22
Cl
8
22 H
CHCl3
impurity
10, 12 H
6.283
1.973
1.964
1.004
1.004
3.972
3.000
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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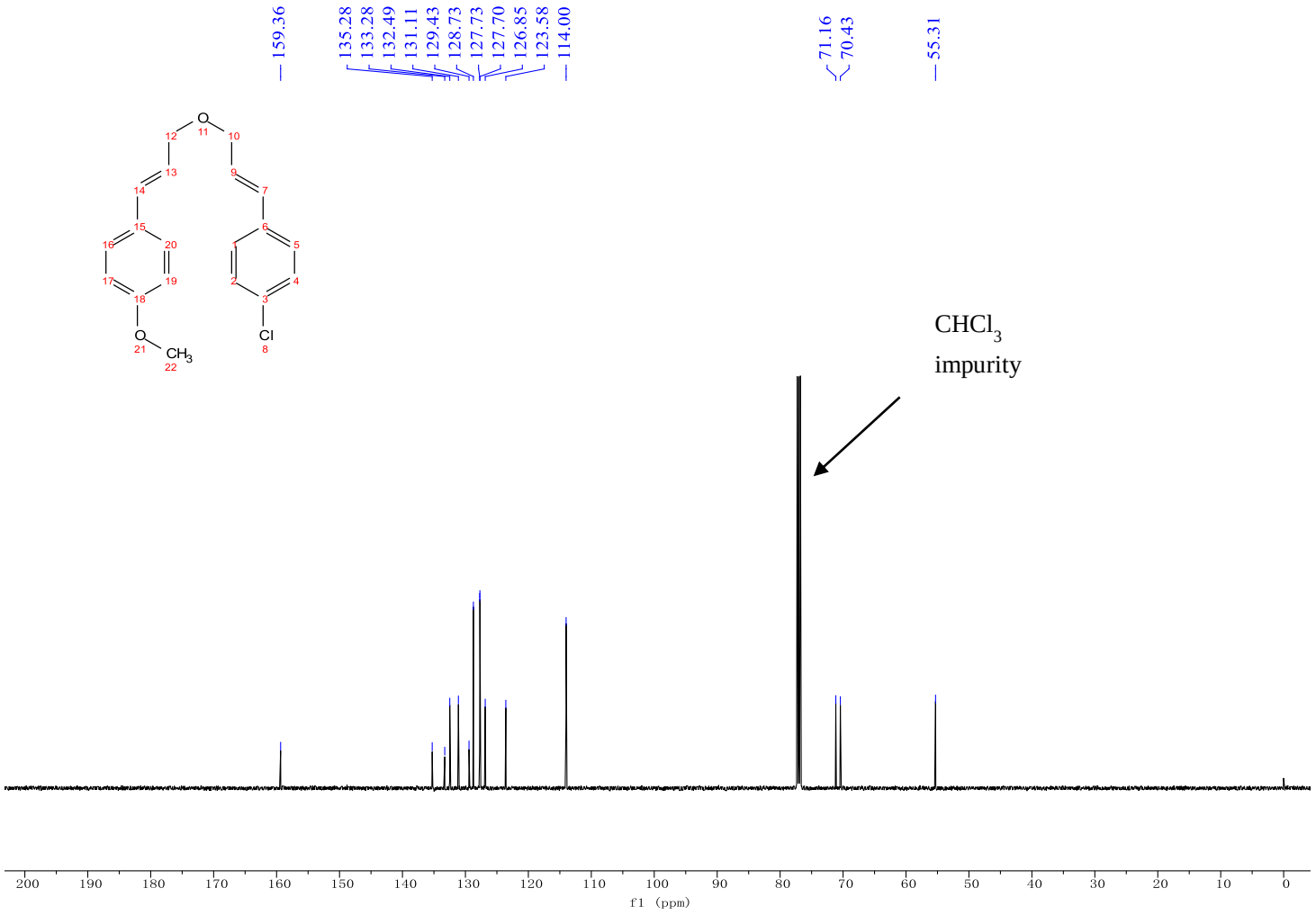
0.0
f1 (ppm)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123.58
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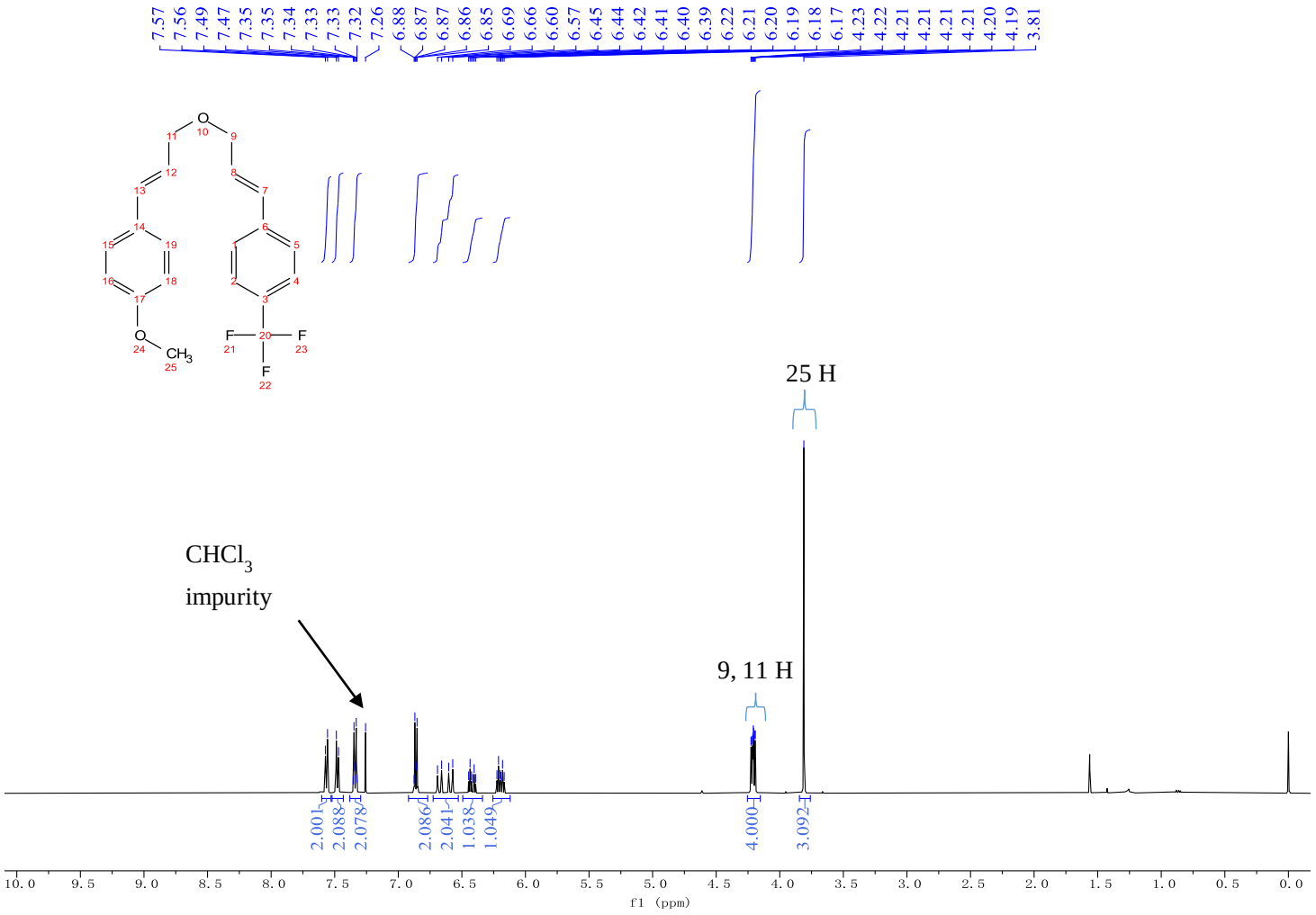
71.16
70.43
55.31
CHCl3
impurity
f1 (ppm)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7.33
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7.32
7.26
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6.87
6.86
6.85
6.69
6.66
6.60
6.57
6.45
6.44
6.42
6.41
6.40
6.39
6.22
6.21
6.20
6.19
6.18
6.17
4.23
4.22
4.21
4.21
4.21
4.20
4.19
3.81
25 H
CHCl3
impurity
9, 11 H
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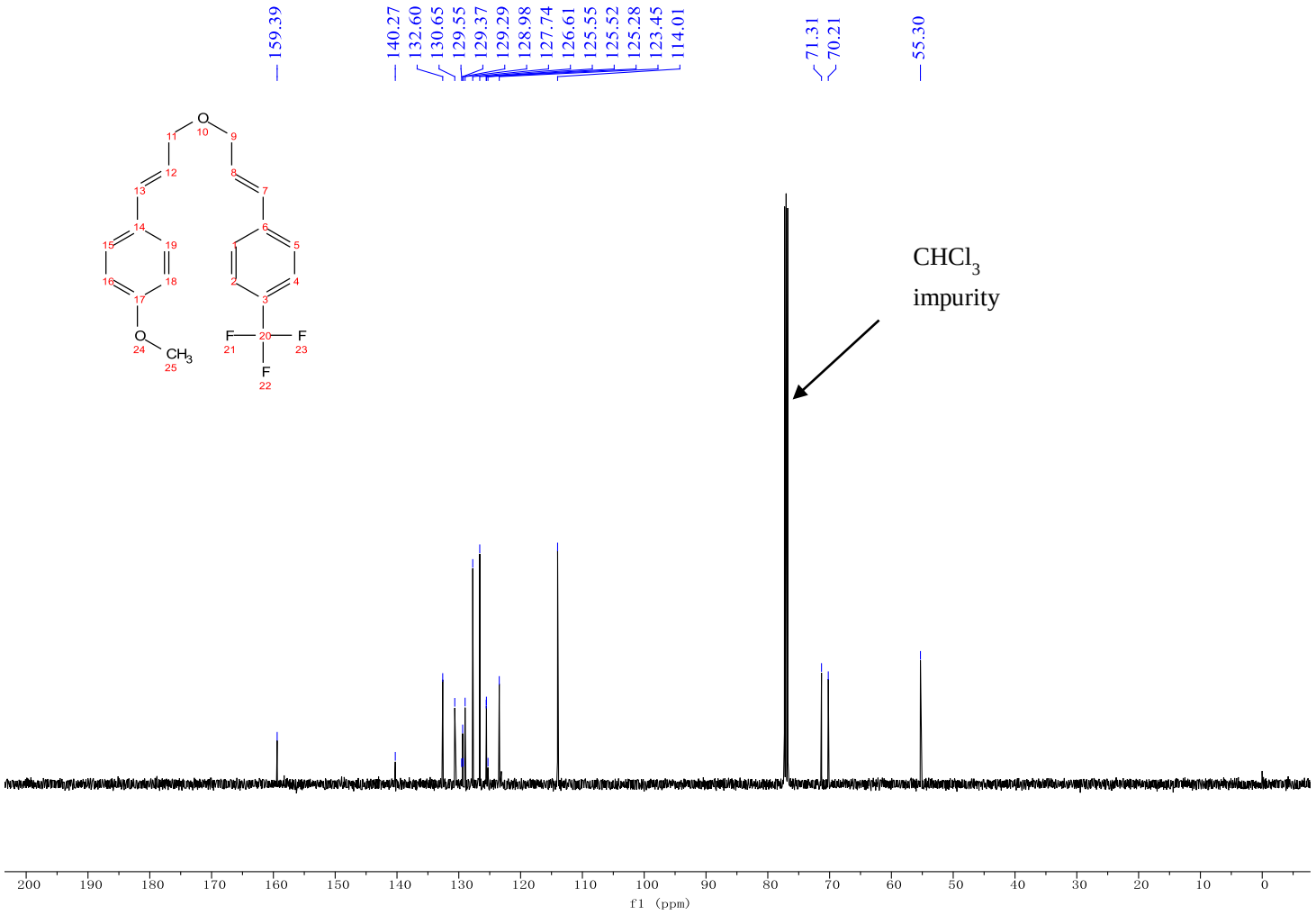
2.088
2.078
2.086
2.041
1.038
1.049
4.000
3.092
f1 (ppm)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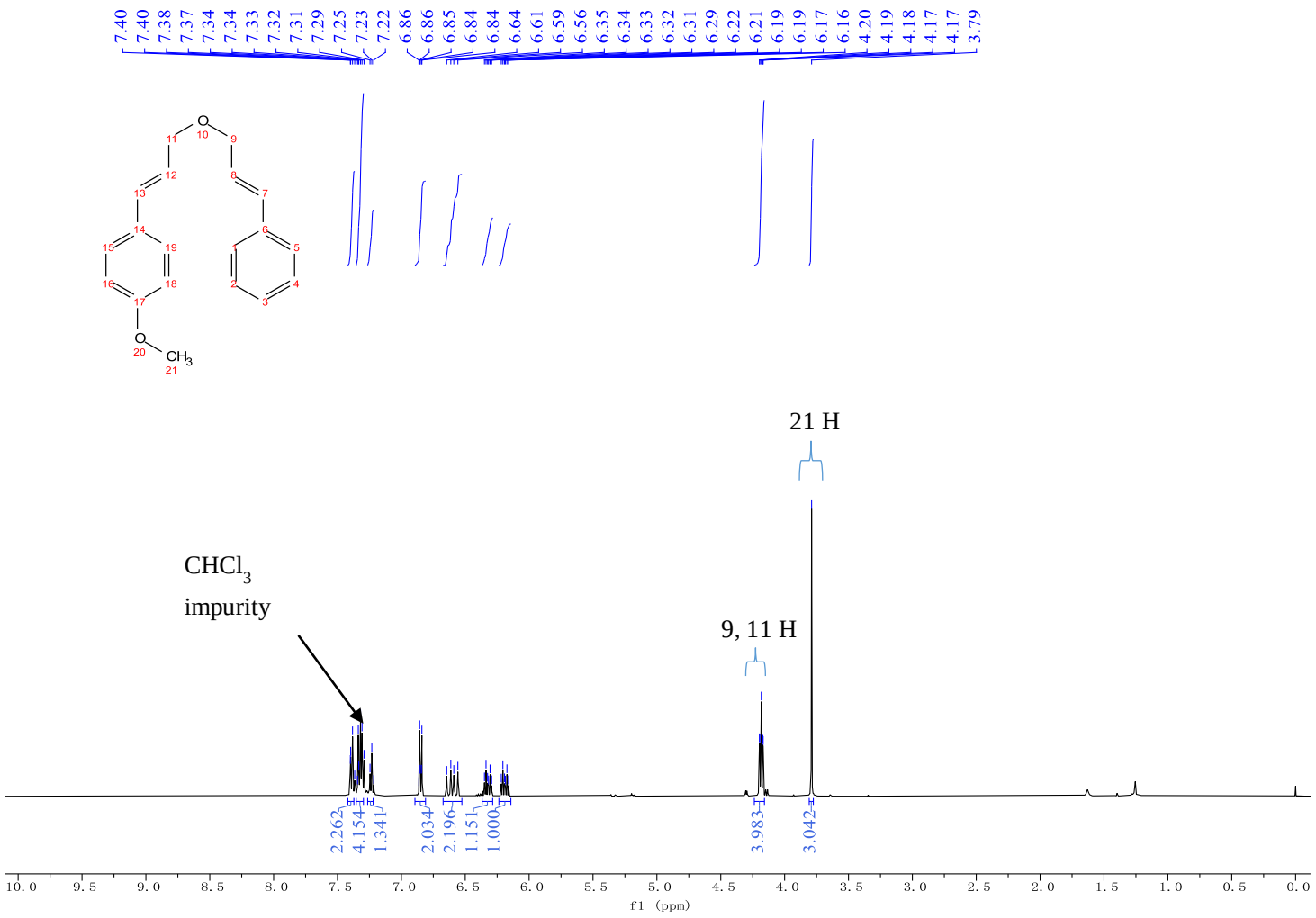
128.98
127.74
126.61
125.55
125.52
125.28
123.45
114.01
71.31
70.21
55.30
CHCl3
impurity
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.40
7.38
7.37
7.34
7.34
7.33
7.32
7.31
7.29
7.25
7.23
7.22
6.86
6.86
6.85
6.84
6.84
6.64
6.61
6.59
6.56
6.35
6.34
6.33
6.32
6.31
6.29
6.22
6.21
6.19
6.19
6.17
6.16
4.20
4.19
4.18
4.17
4.17
3.79
21 H
CHCl3
impurity
9, 11 H
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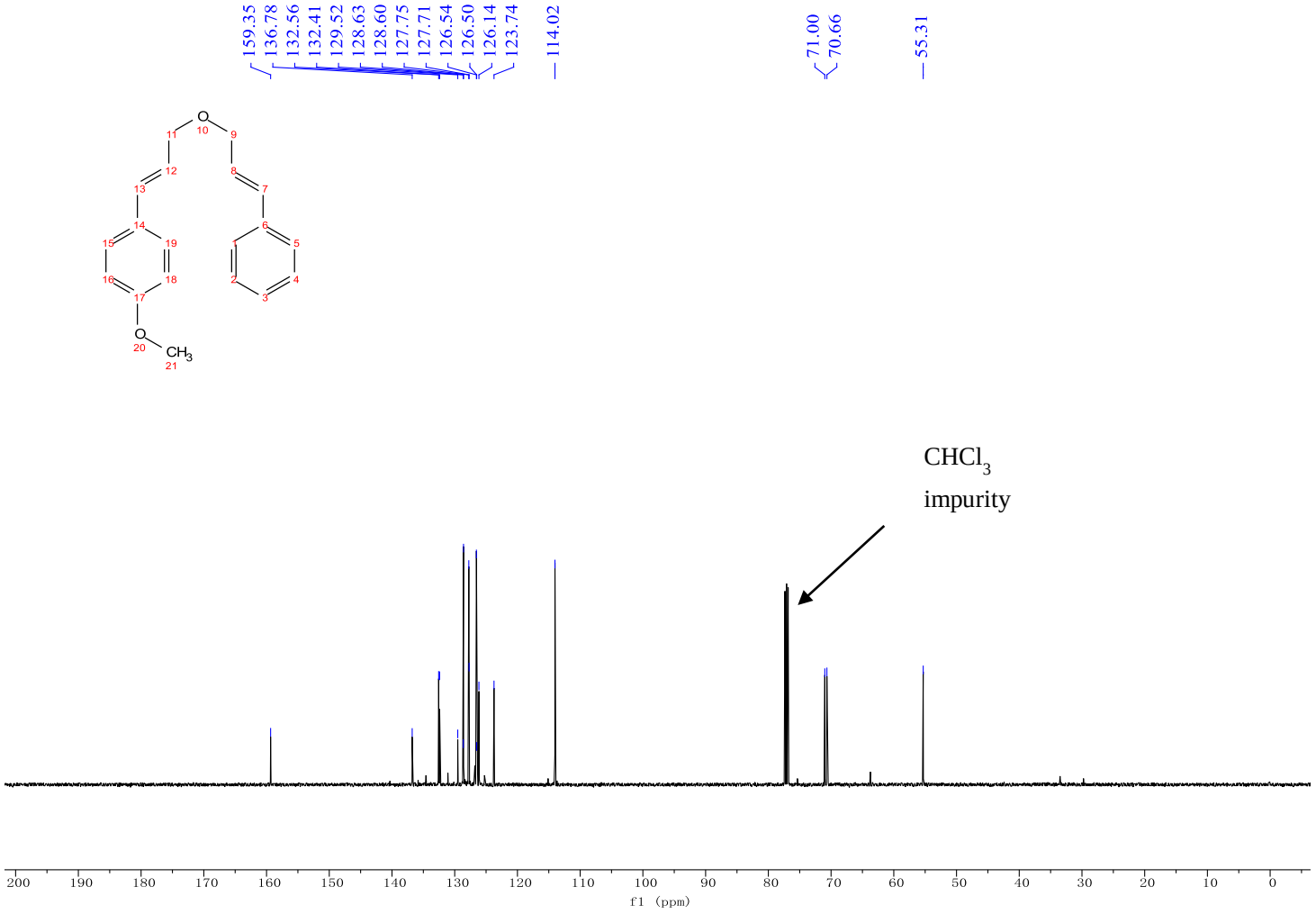
4.154
1.341
2.034
2.196
1.151
1.000
3.983
3.042
f1 (ppm)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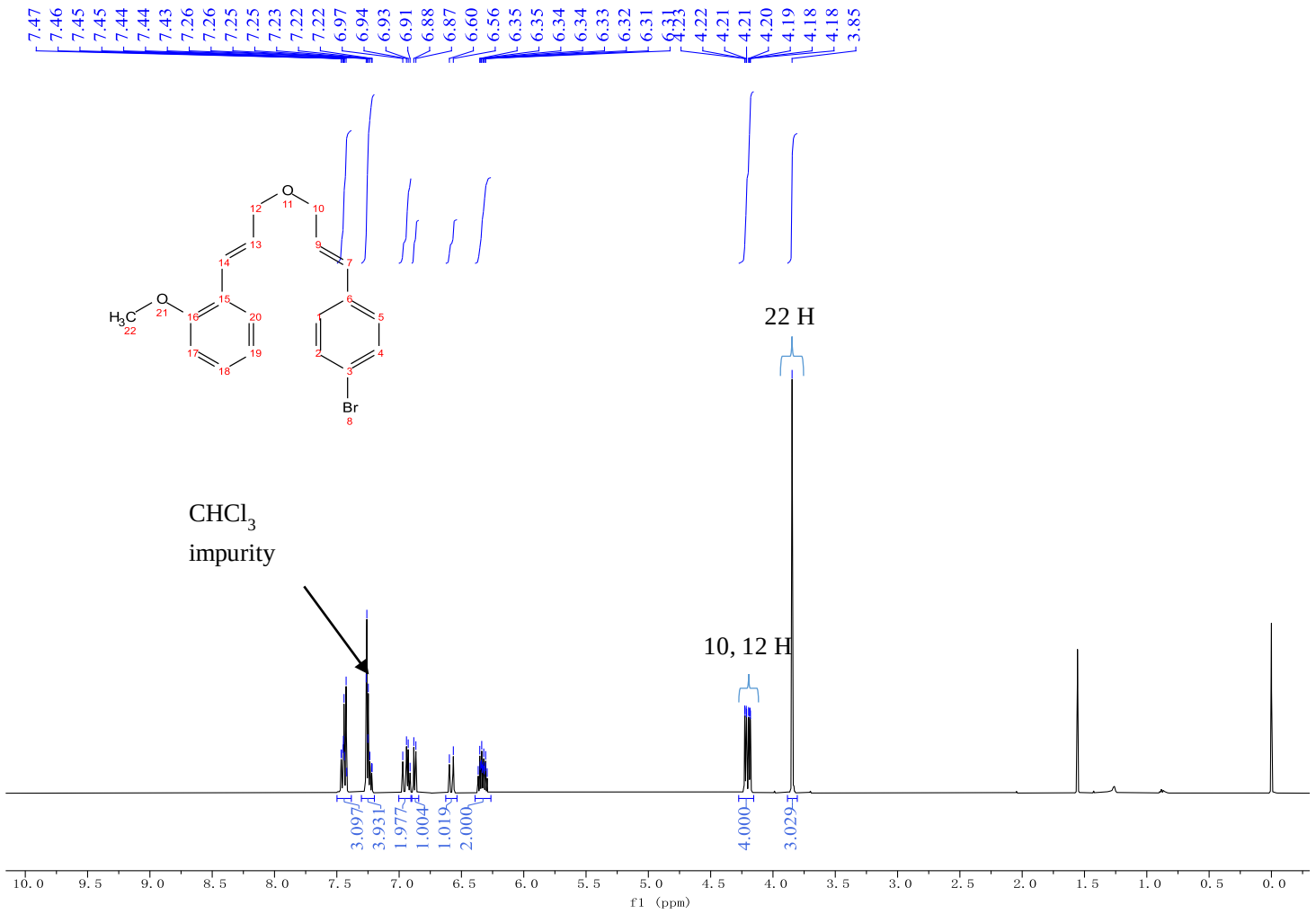
126.14
123.74
114.02
71.00
70.66
55.31
O
10
11
9
12
8
13
7
14
6
15
19
1
5
16
18
2
4
17
3
O
20
CH3
21
CHCl3
impurity
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.43
7.26
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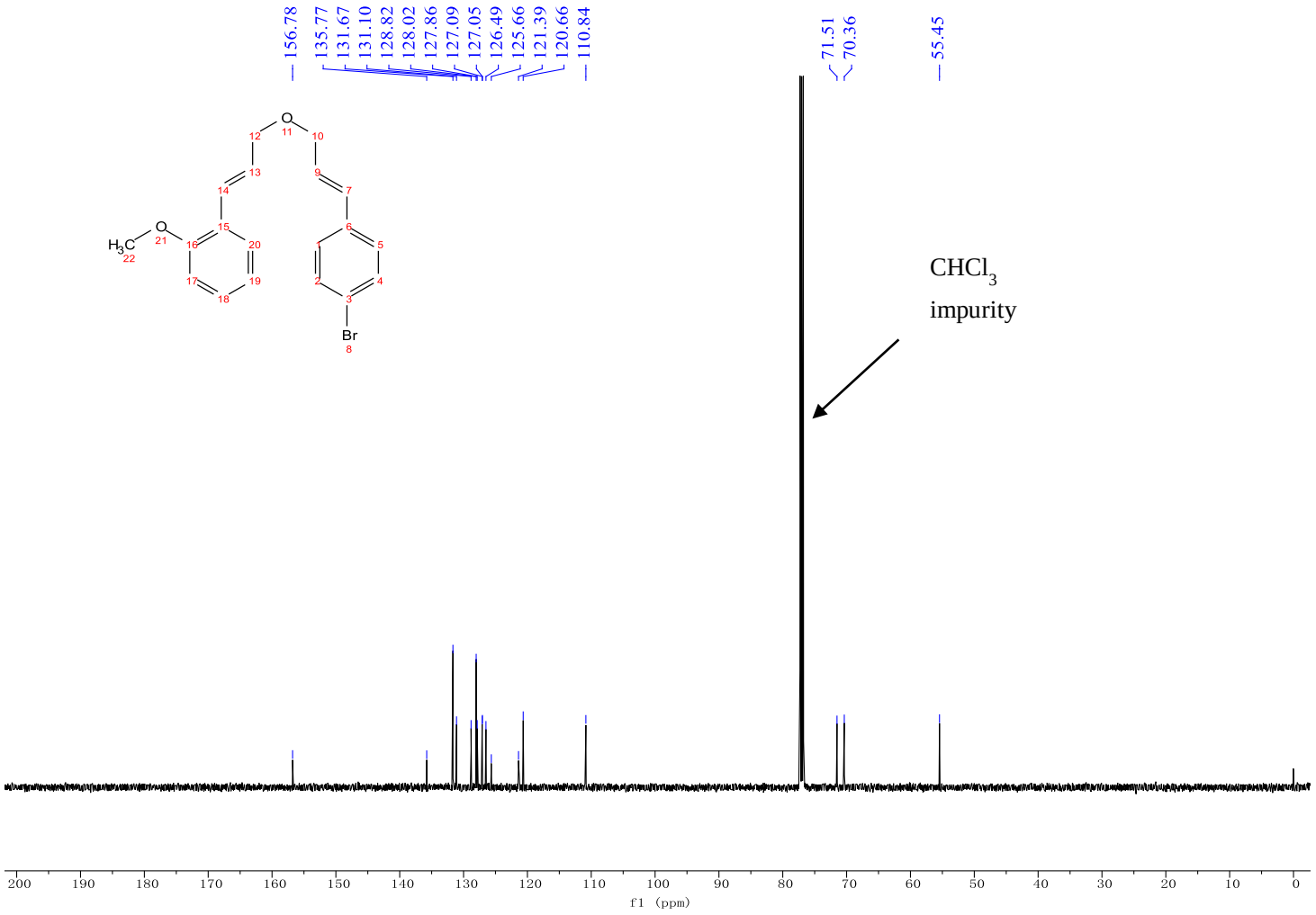
7.25
7.25
7.23
7.22
7.22
6.97
6.94
6.93
6.91
6.88
6.87
6.60
6.56
6.35
6.35
6.34
6.34
6.33
6.32
6.31
6.31
4.23
4.22
4.21
4.21
4.20
4.19
4.18
4.18
3.85
22 H
CHCl3
impurity
10, 12 H
3.097
3.931
1.977
1.004
1.019
2.000
4.000
3.029
f1 (ppm)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128.02
127.86
127.09
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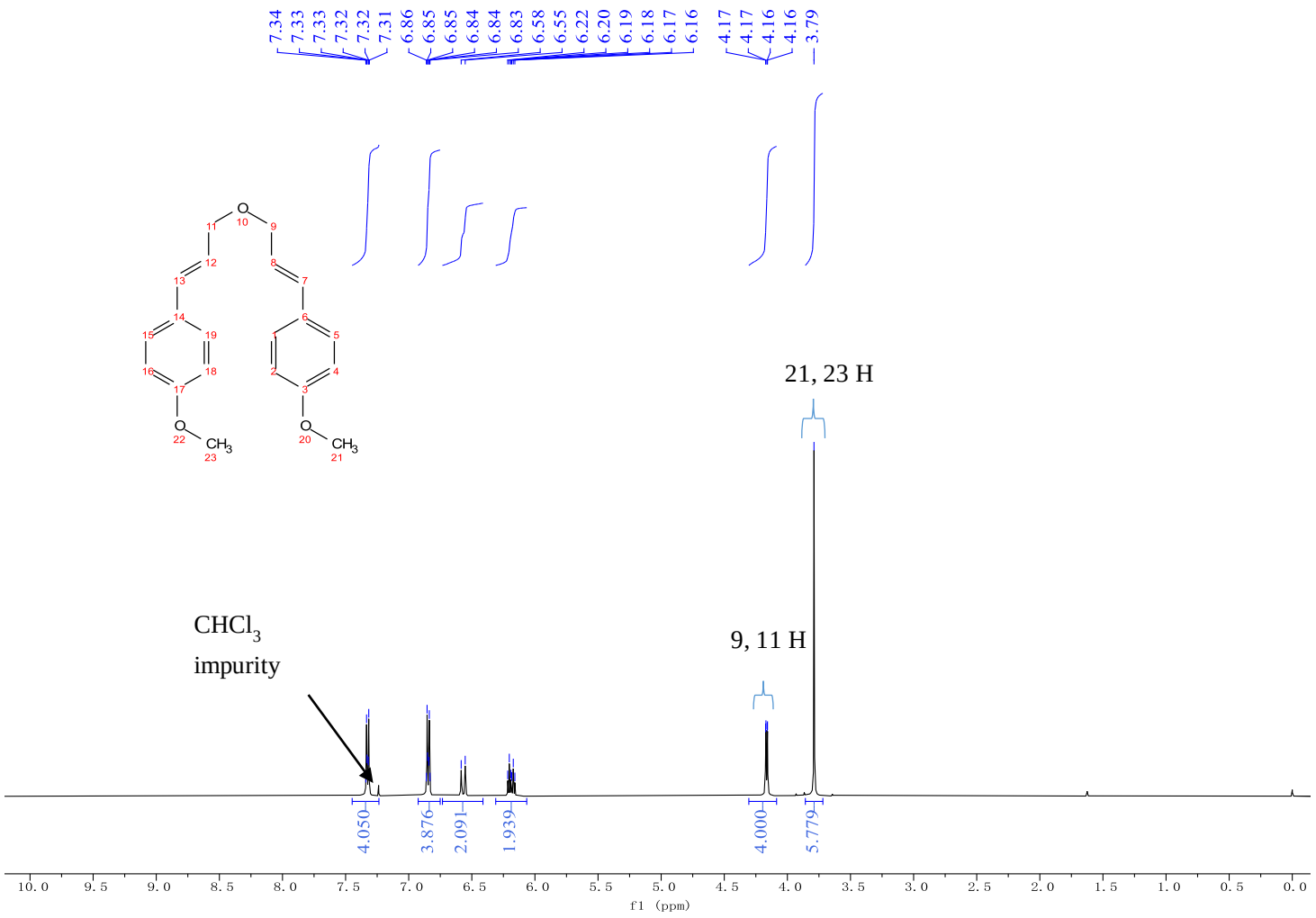
121.39
120.66
110.84
71.51
70.36
55.45
CHCl3
impurity
f1 (ppm)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7.31
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6.85
6.85
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6.84
6.83
6.58
6.55
6.22
6.20
6.19
6.18
6.17
6.16
4.17
4.17
4.16
4.16
3.79
21, 23 H
9, 11 H
CHCl3
impurity
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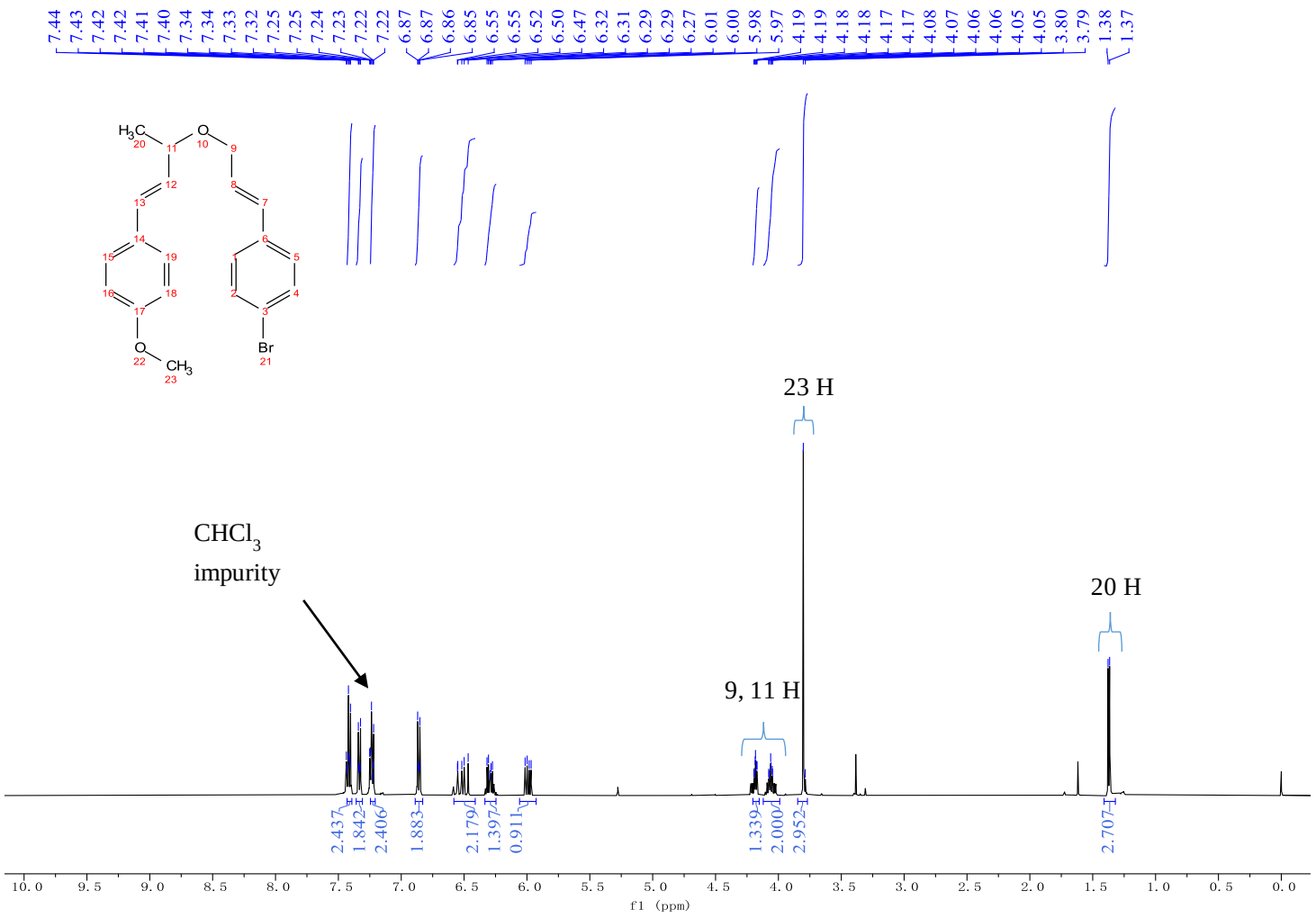
1.939
4.000
5.779
f1 (ppm)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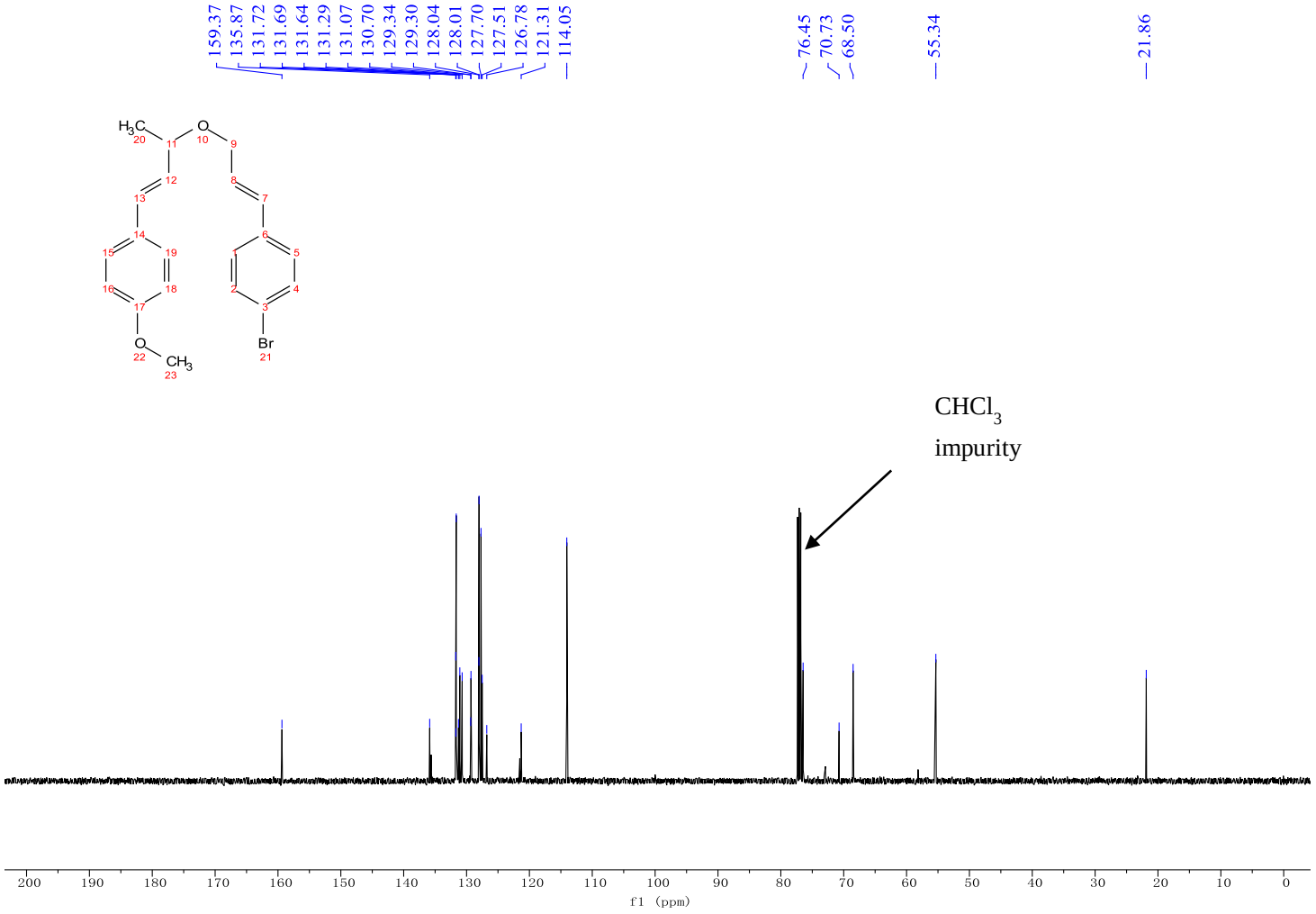
6.87 6.87 6.86 6.85 6.55 6.55 6.52 6.50 6.47 6.32 6.31 6.29 6.29 6.27 6.01 6.00 5.98 5.97
4.19 4.19 4.18 4.18 4.17 4.17 4.08 4.07 4.06 4.06 4.05 4.05 3.80 3.79 1.38 1.37
H3C
O
Br
CH3
23 H
CHCl3
impurity
20 H
9, 11 H
2.437
1.842
2.406
1.883
2.179
1.397
0.911
1.339
2.000
2.952
2.707
10.0 9.5 9.0 8.5 8.0 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
f1 (ppm)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128.01
127.70
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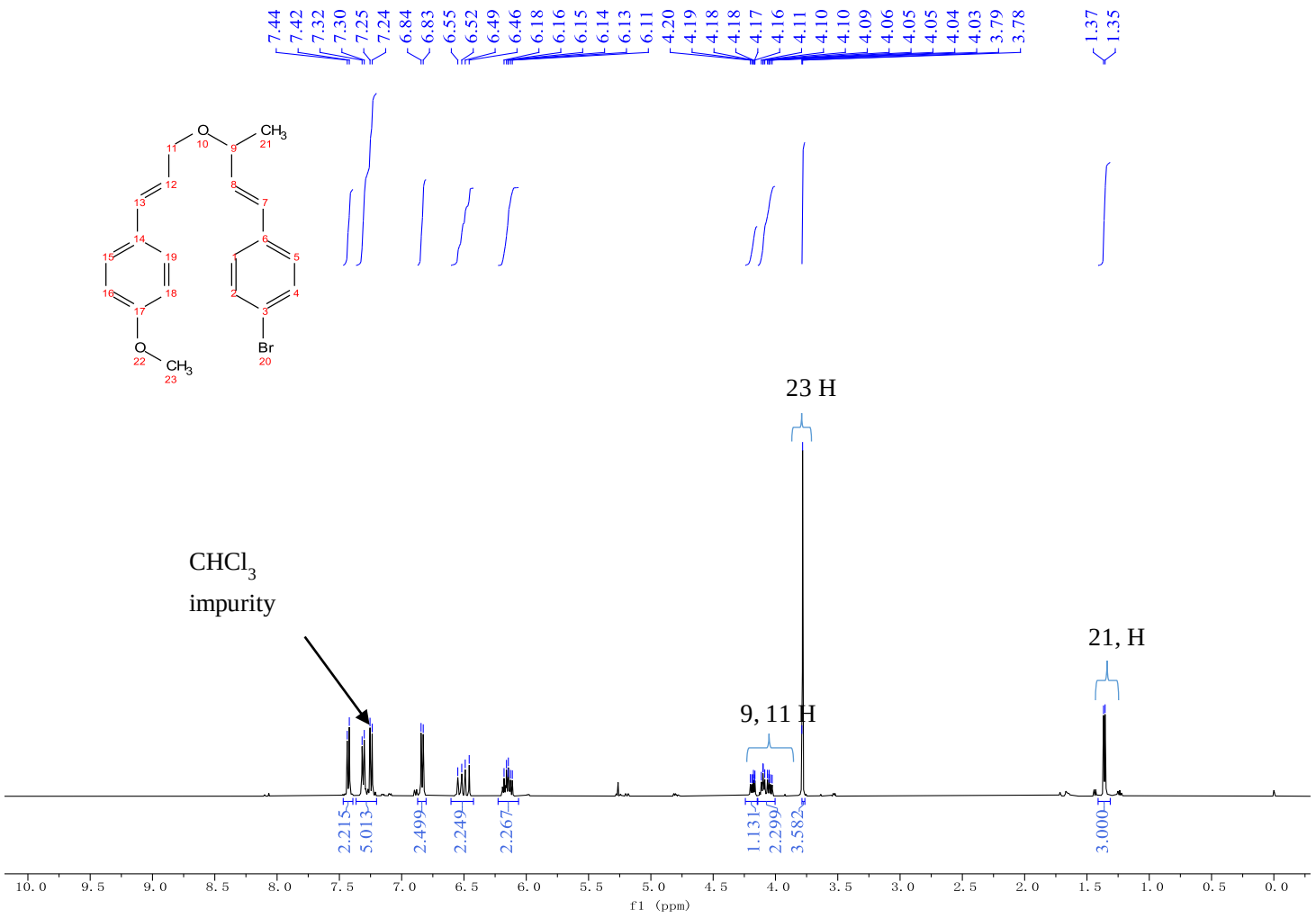
68.50
55.34
21.86
CHCl3
impurity
f1 (ppm)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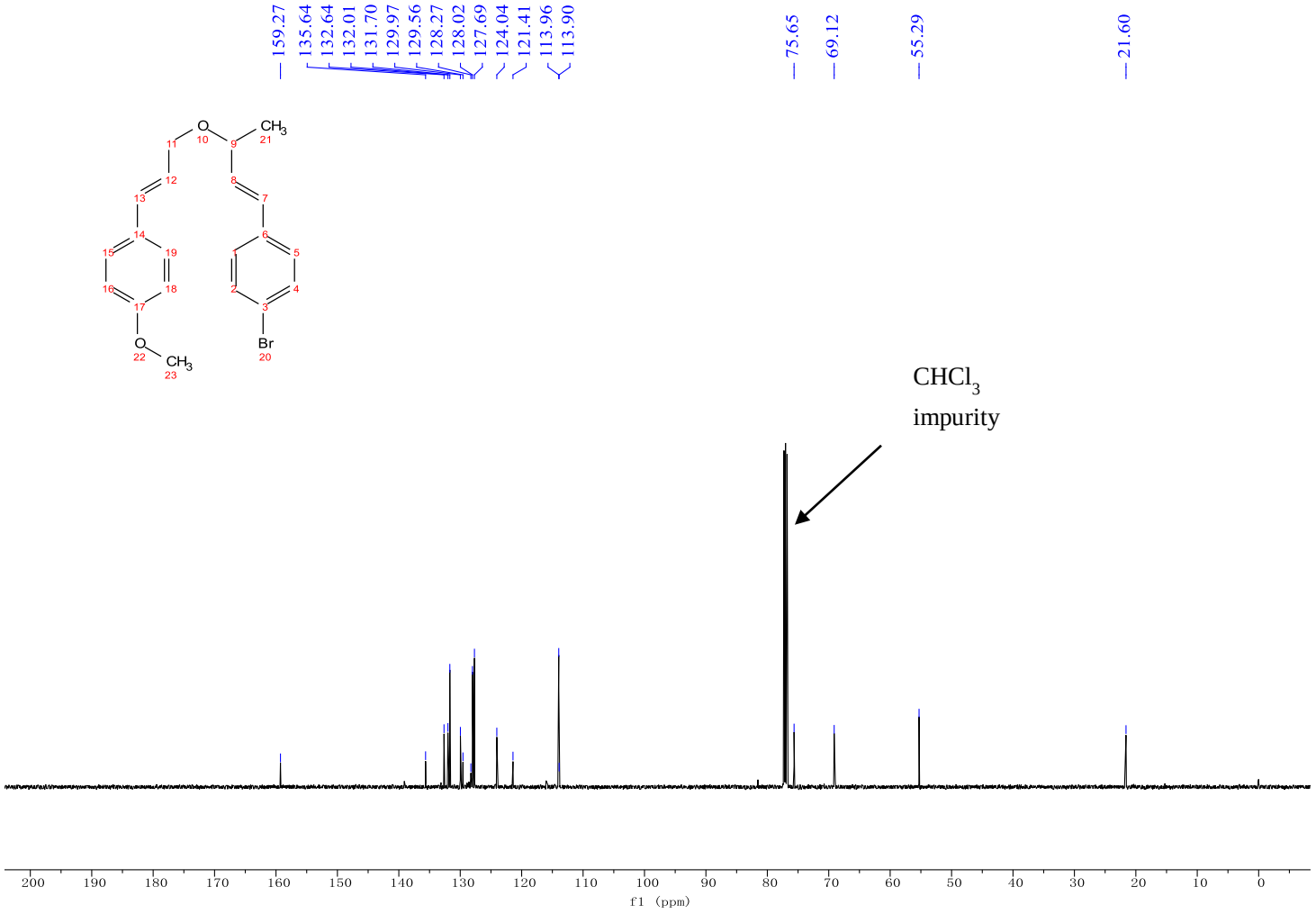
6.55
6.52
6.49
6.46
6.18
6.16
6.15
6.14
6.13
6.11
4.20
4.19
4.18
4.18
4.17
4.16
4.11
4.10
4.10
4.09
4.06
4.05
4.05
4.04
4.03
3.79
3.78
1.37
1.35
O
10
CH3
21
11
12
9
8
13
7
14
6
15
19
1
5
16
18
2
4
17
3
O
22
CH3
23
Br
20
23 H
CHCl3
impurity
21, H
9, 11 H
2.215
5.013
2.499
2.249
2.267
1.131
2.299
3.582
3.000
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
f1 (ppm)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128.27
128.02
127.69
124.04
121.41
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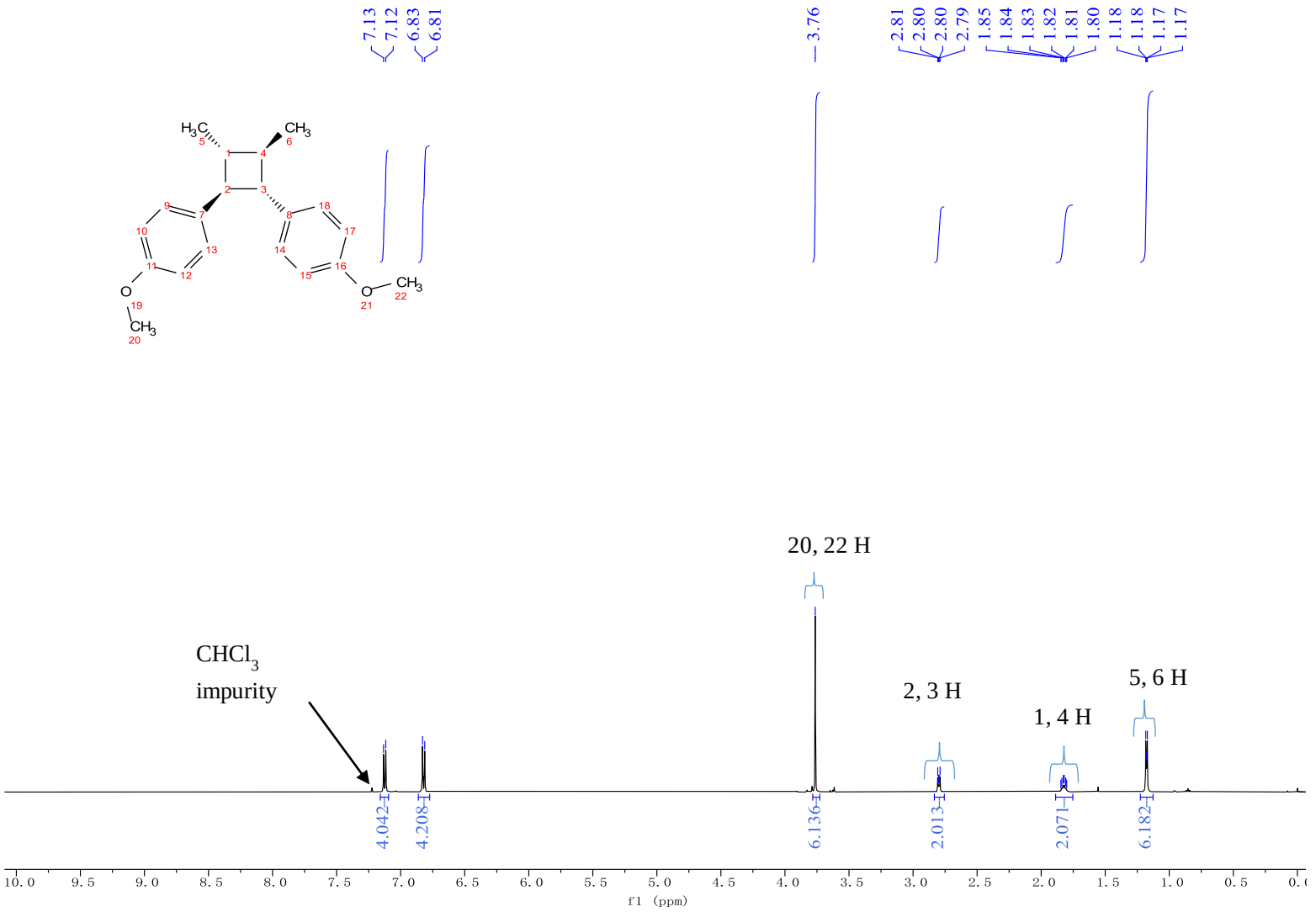
113.90
75.65
69.12
55.29
21.60
O
10
CH3
21
11
12
13
14
15
16
17
18
19
9
8
7
6
1
2
3
4
5
O
22
CH3
23
Br
20
CHCl3
impurity
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.12
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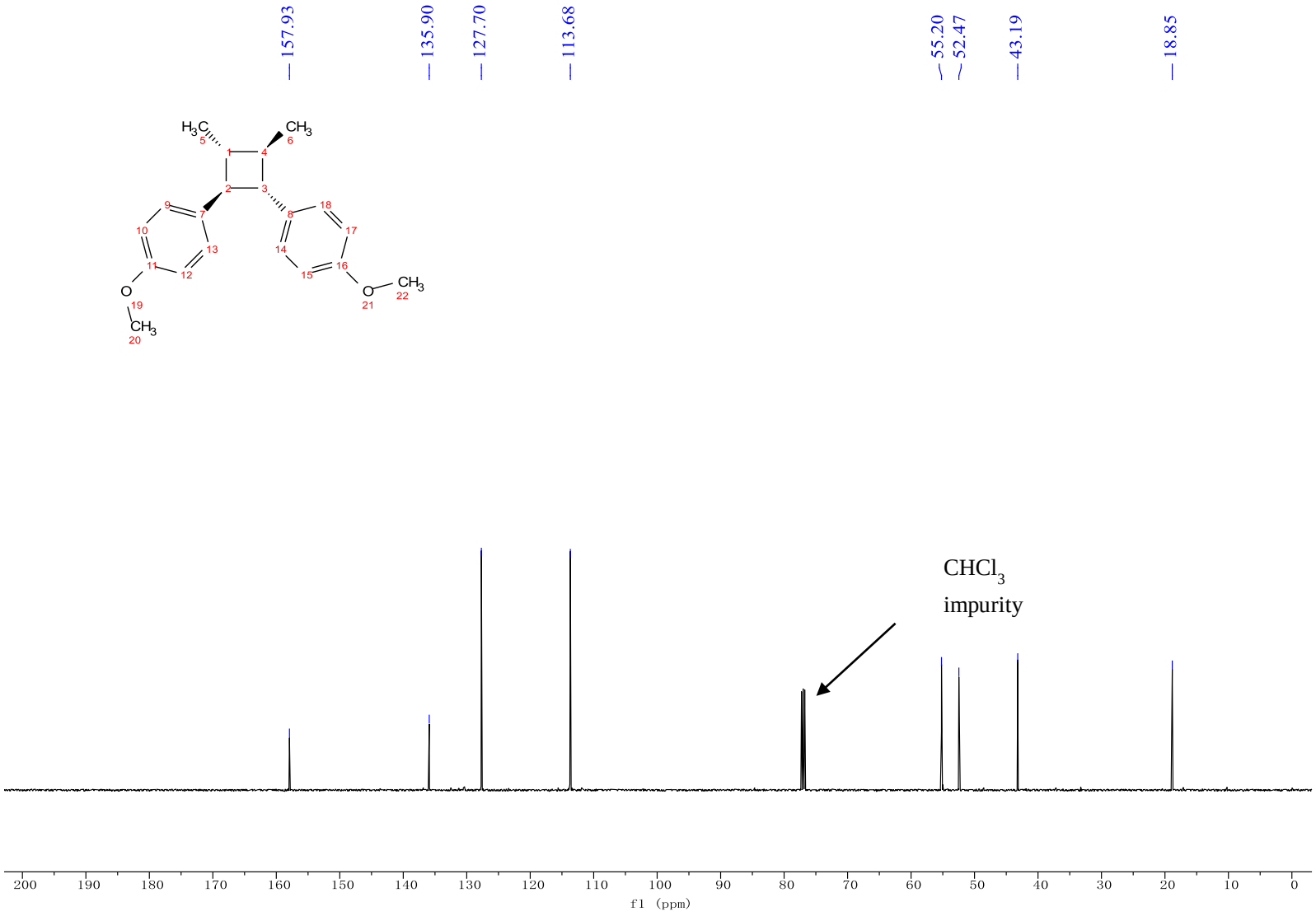
3.76
2.81
2.80
2.80
2.79
1.85
1.84
1.83
1.82
1.81
1.80
1.18
1.18
1.17
1.17
20, 22 H
CHCl3
impurity
2, 3 H
1, 4 H
5, 6 H
4.042
4.208
6.136
2.013
2.071
6.182
f1 (ppm)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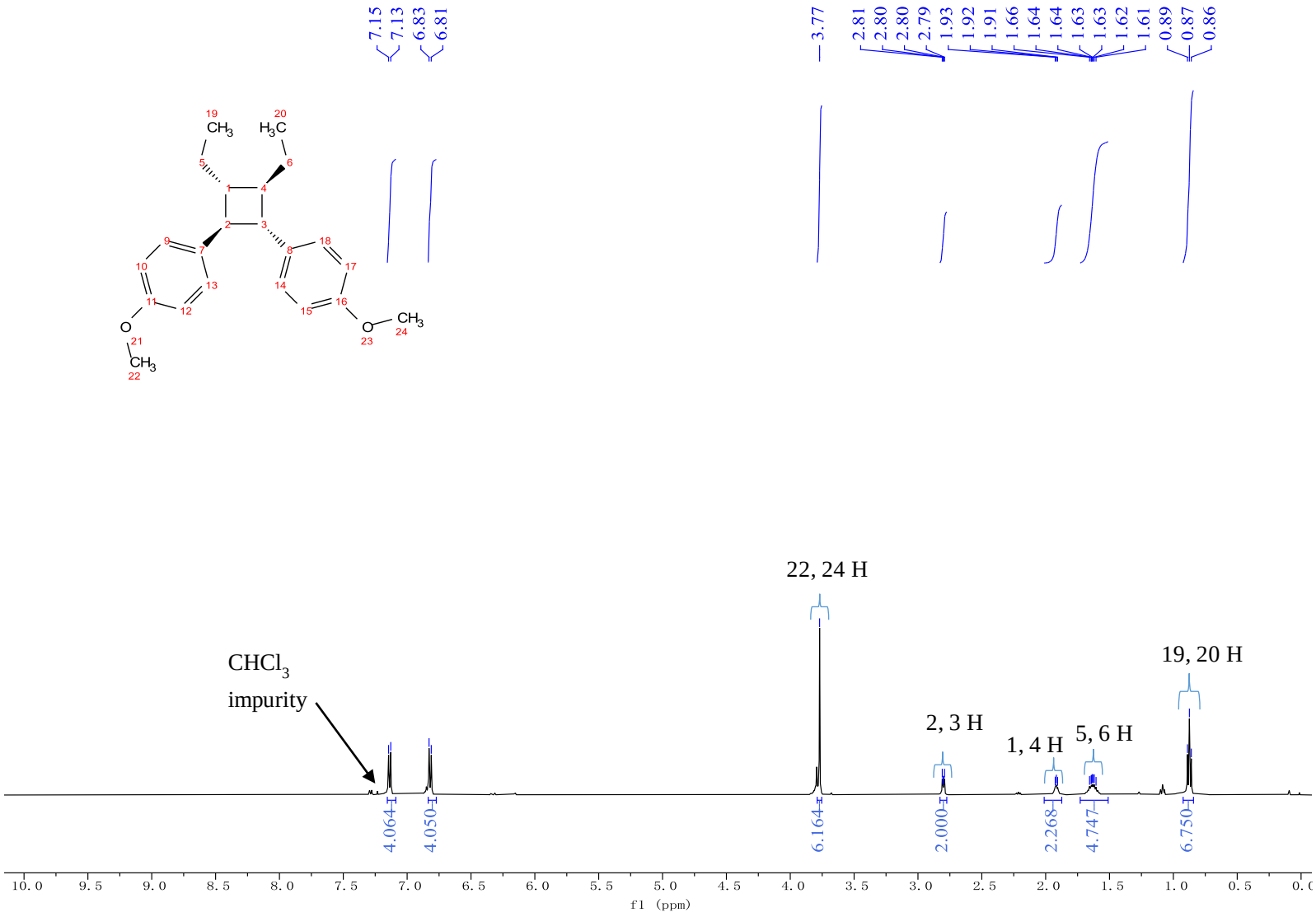
52.47
43.19
18.85
CHCl3
impurity
f1 (ppm)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2.80
2.80
2.79
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1.92
1.91
1.66
1.64
1.64
1.63
1.63
1.62
1.61
0.89
0.87
0.86
22, 24 H
CHCl3
impurity
2, 3 H
1, 4 H
5, 6 H
19, 20 H
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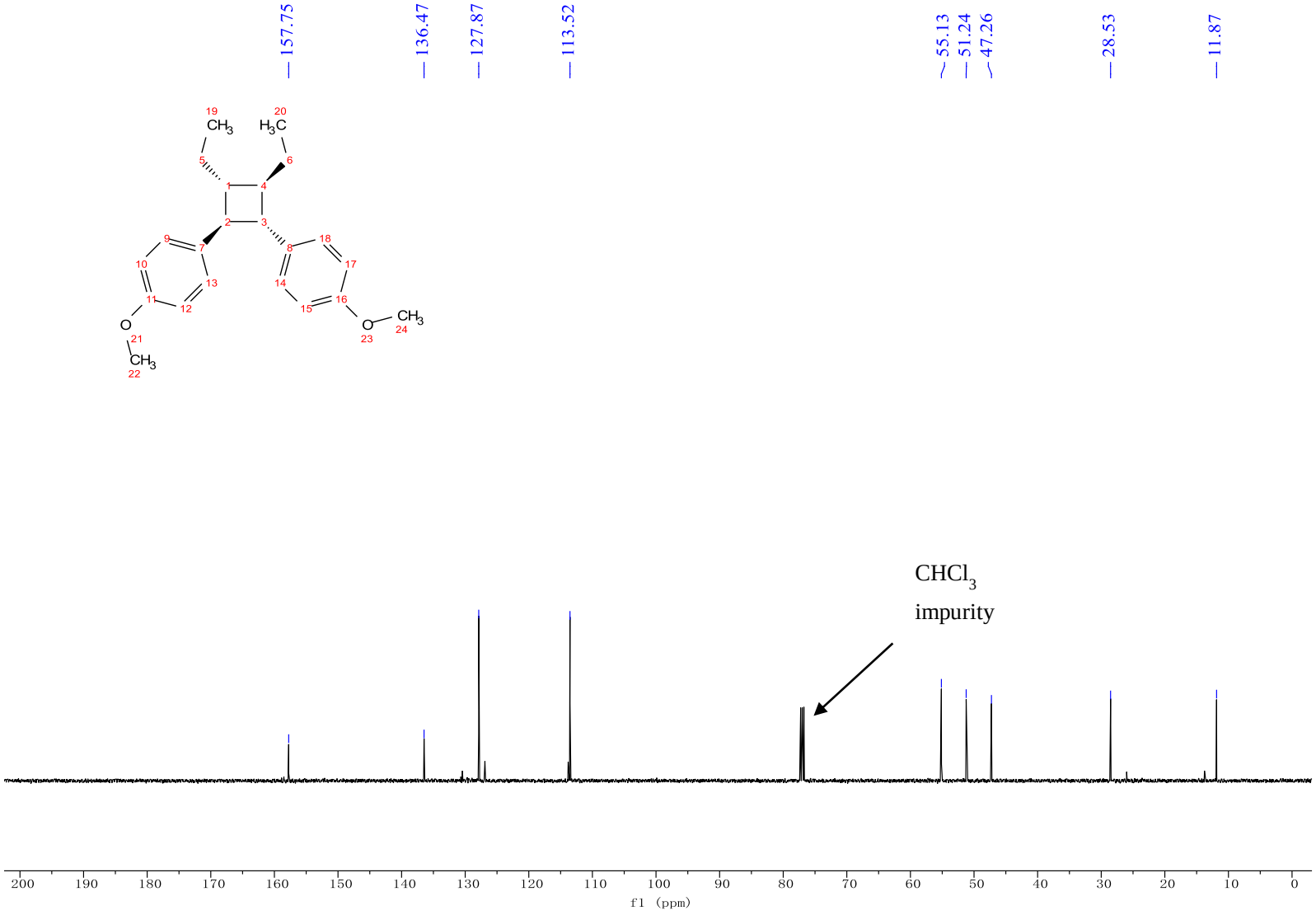
4.050
6.164
2.000
2.268
4.747
6.750
f1 (ppm)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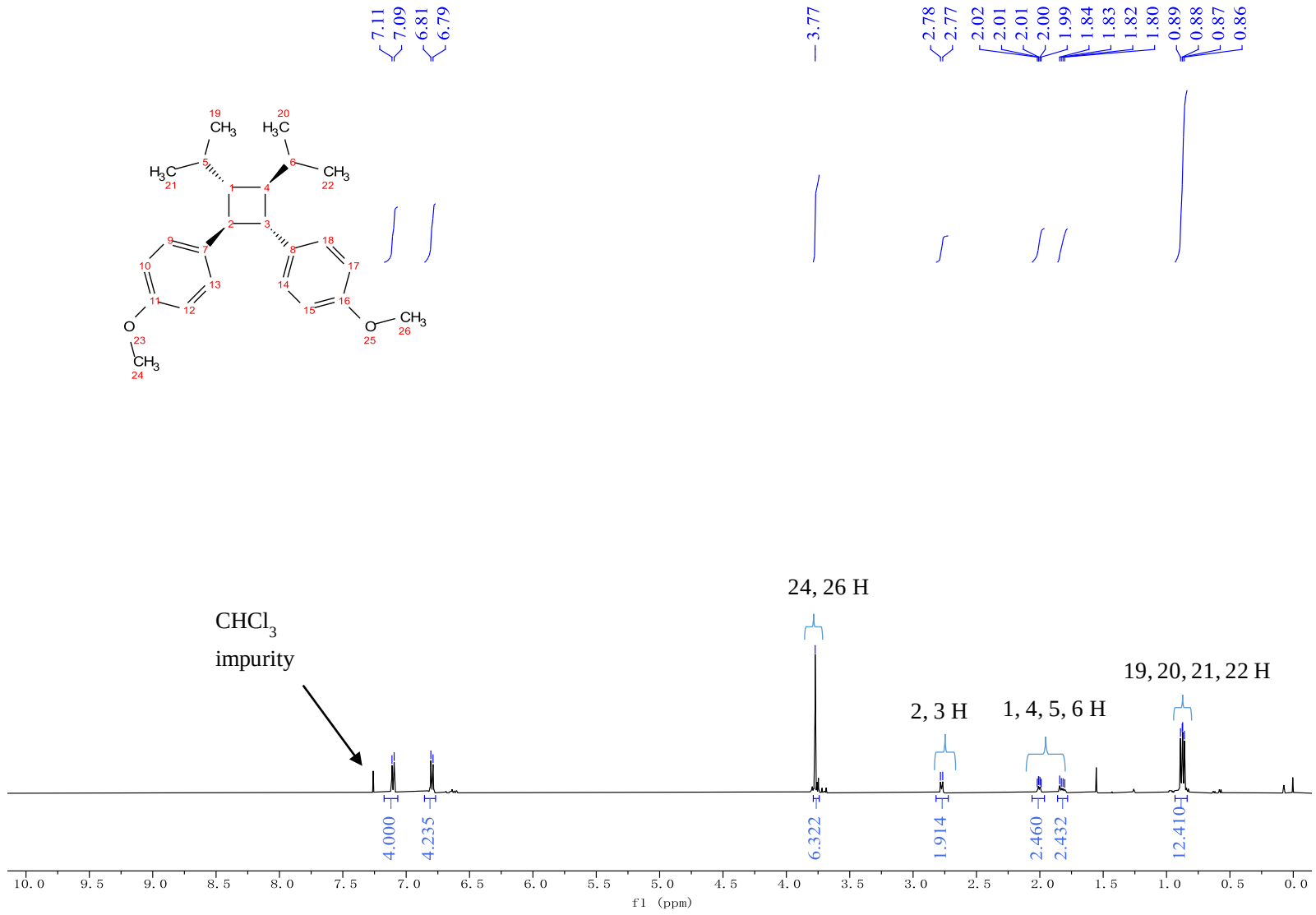
51.24
47.26
28.53
11.87
CHCl3
impurity
f1 (ppm)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7.09
6.81
6.79
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2.78
2.77
2.02
2.01
2.01
2.00
1.99
1.84
1.83
1.82
1.80
0.89
0.88
0.87
0.86
CHCl3
impurity
24, 26 H
2, 3 H
1, 4, 5, 6 H
19, 20, 21, 22 H
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2.460
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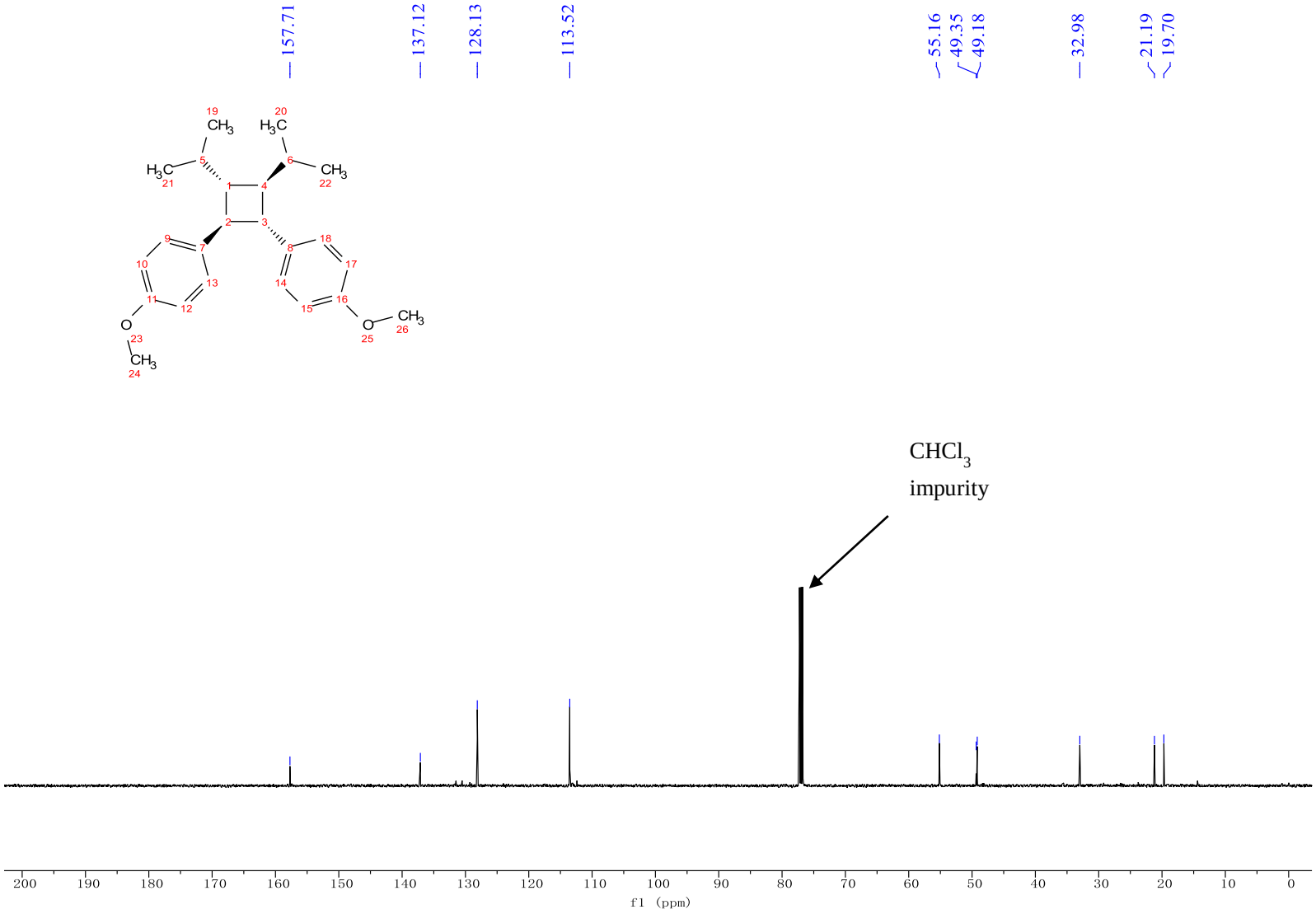
12.410
f1 (ppm)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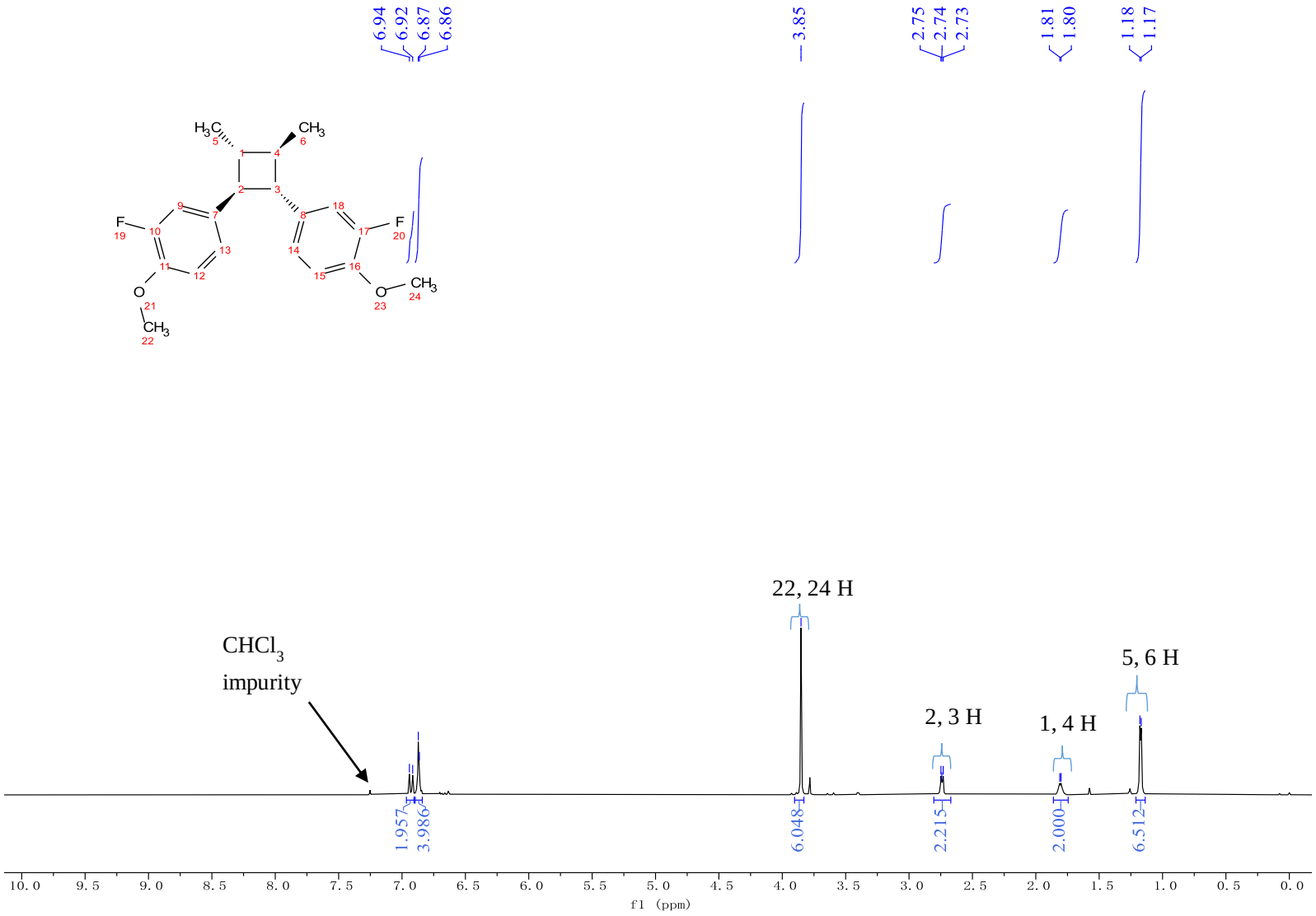
49.18
32.98
21.19
19.70
CHCl3
impurity
f1 (ppm)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6.86
3.85
2.75
2.74
2.73
1.81
1.80
1.18
1.17
H3C
5
CH3
6
1
4
2
3
9
7
8
18
F
19
10
17
F
20
13
14
11
16
12
15
O
21
O
23
CH3
24
CH3
22
22, 24 H
CHCl3
impurity
2, 3 H
1, 4 H
5, 6 H
1.957
3.986
6.048
2.215
2.000
6.512
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
2.5
2.0
1.5
1.0
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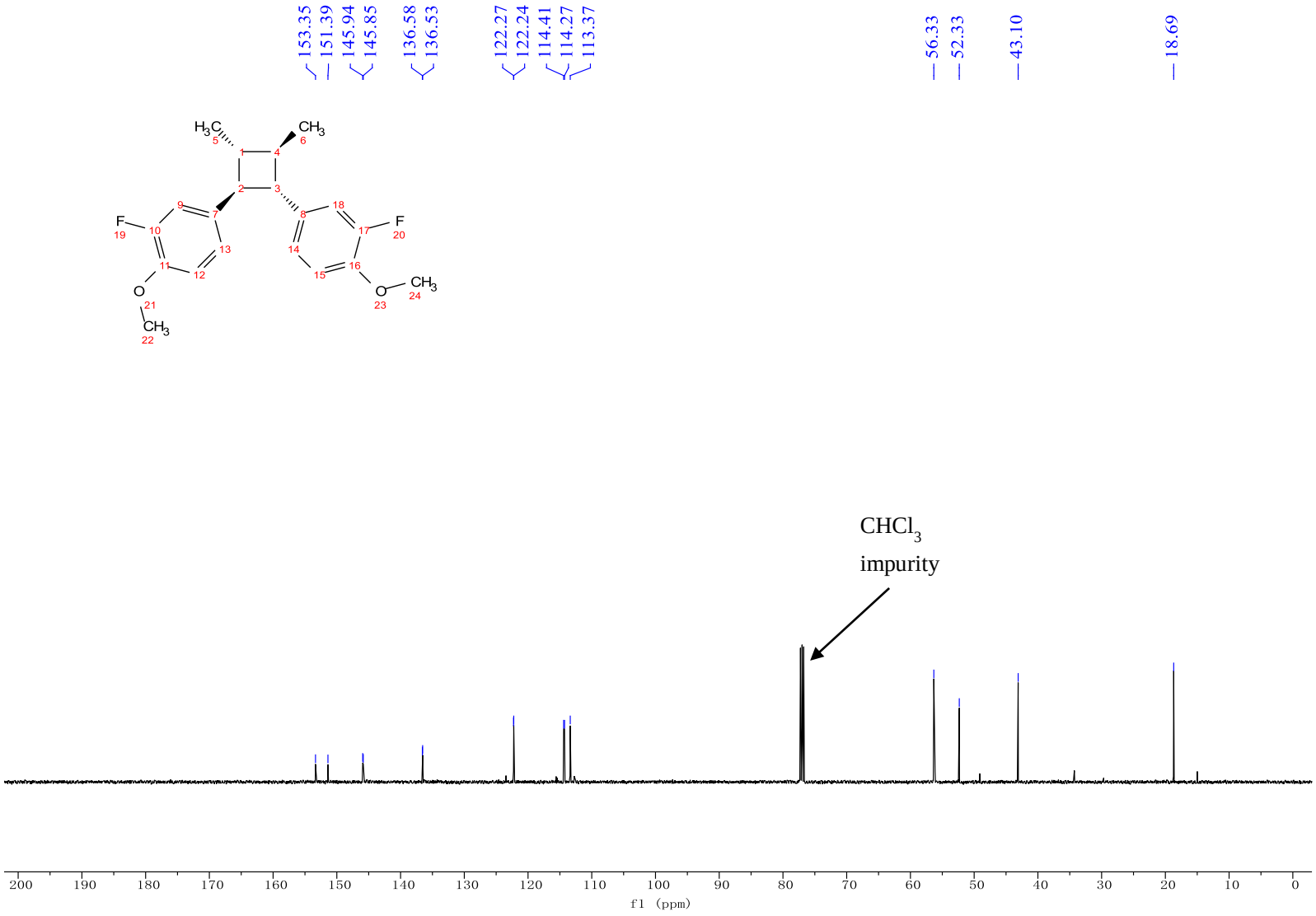
0.0
f1 (ppm)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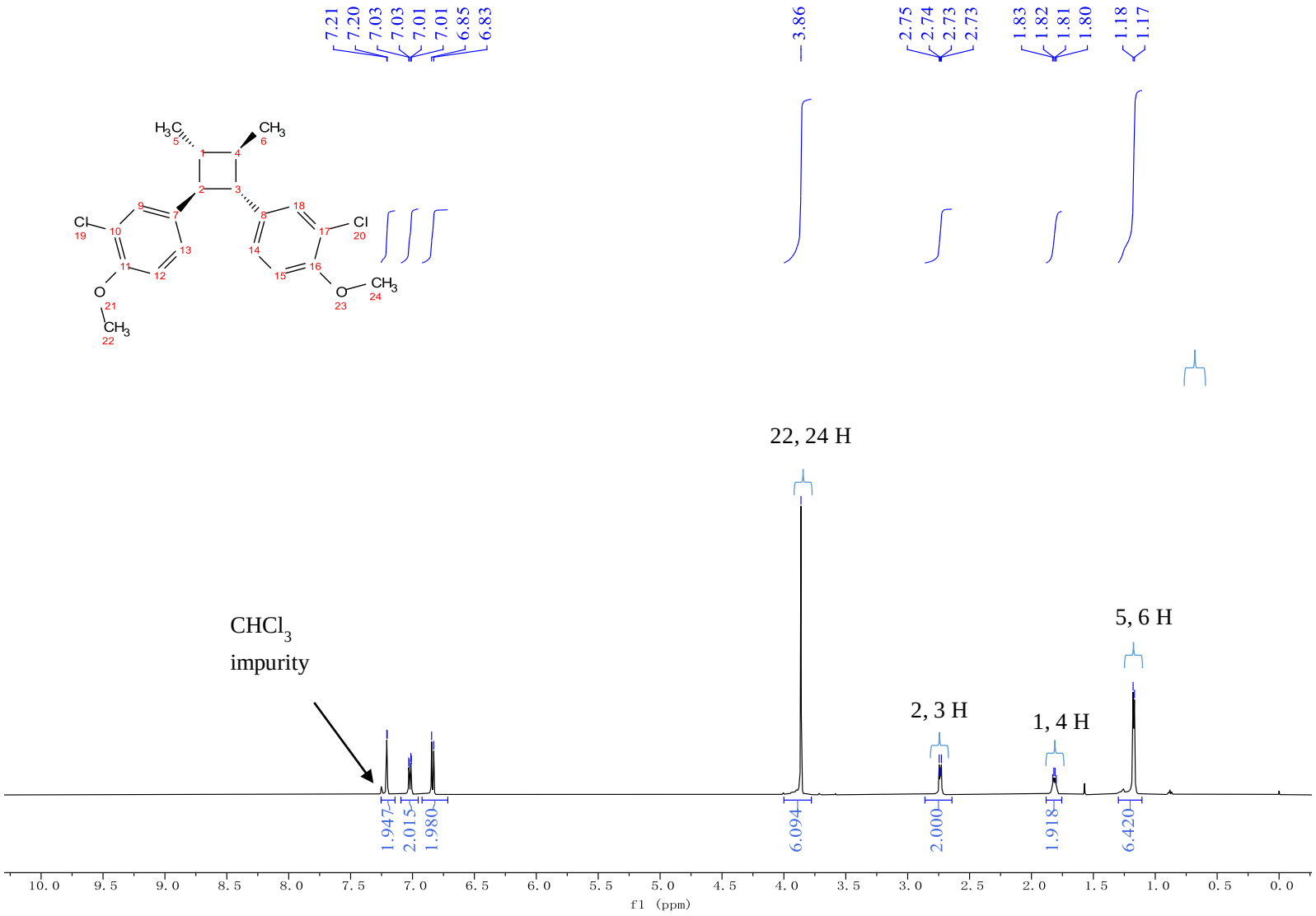
114.27
113.37
56.33
52.33
43.10
18.69
CHCl3
impurity
f1 (ppm)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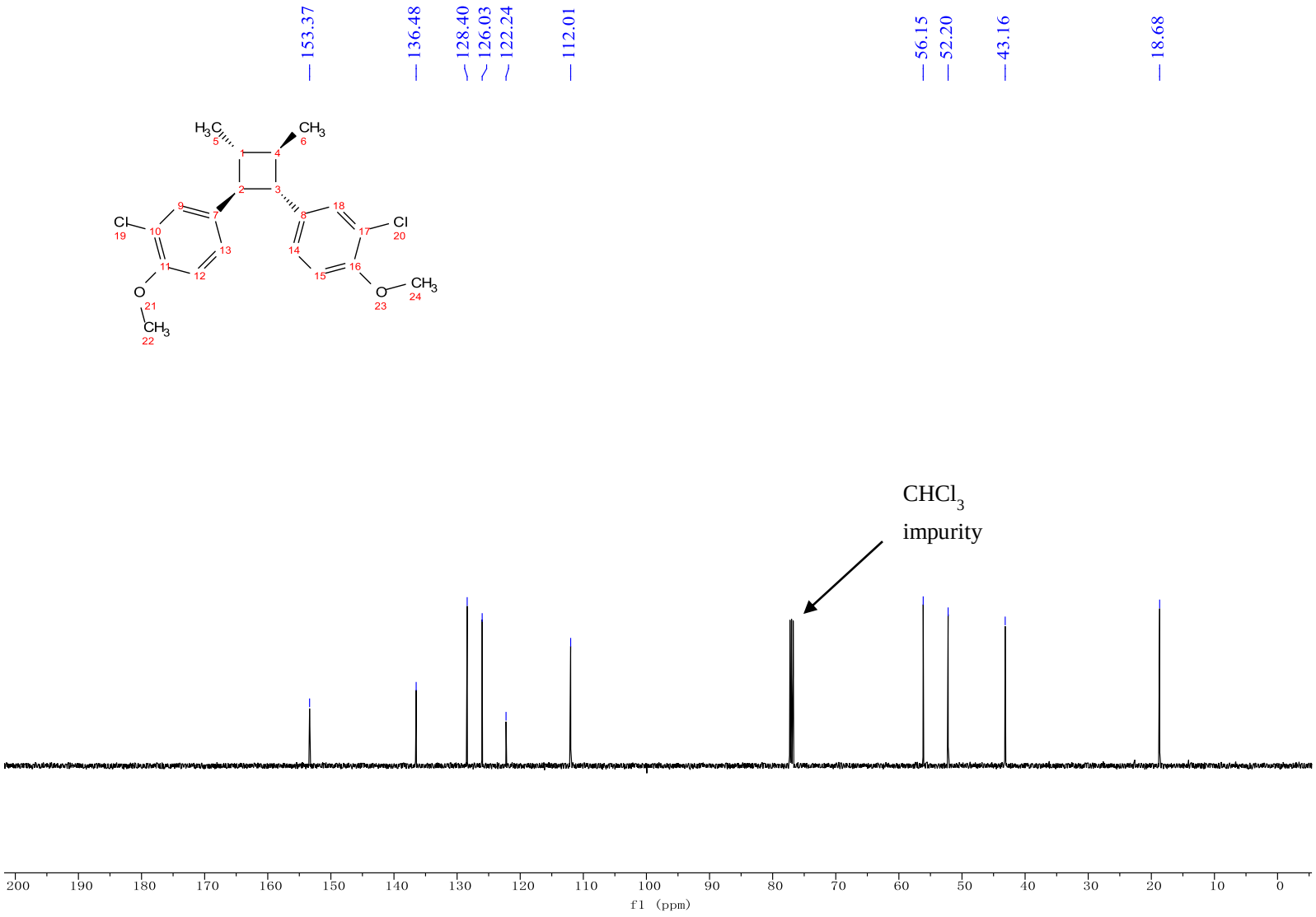
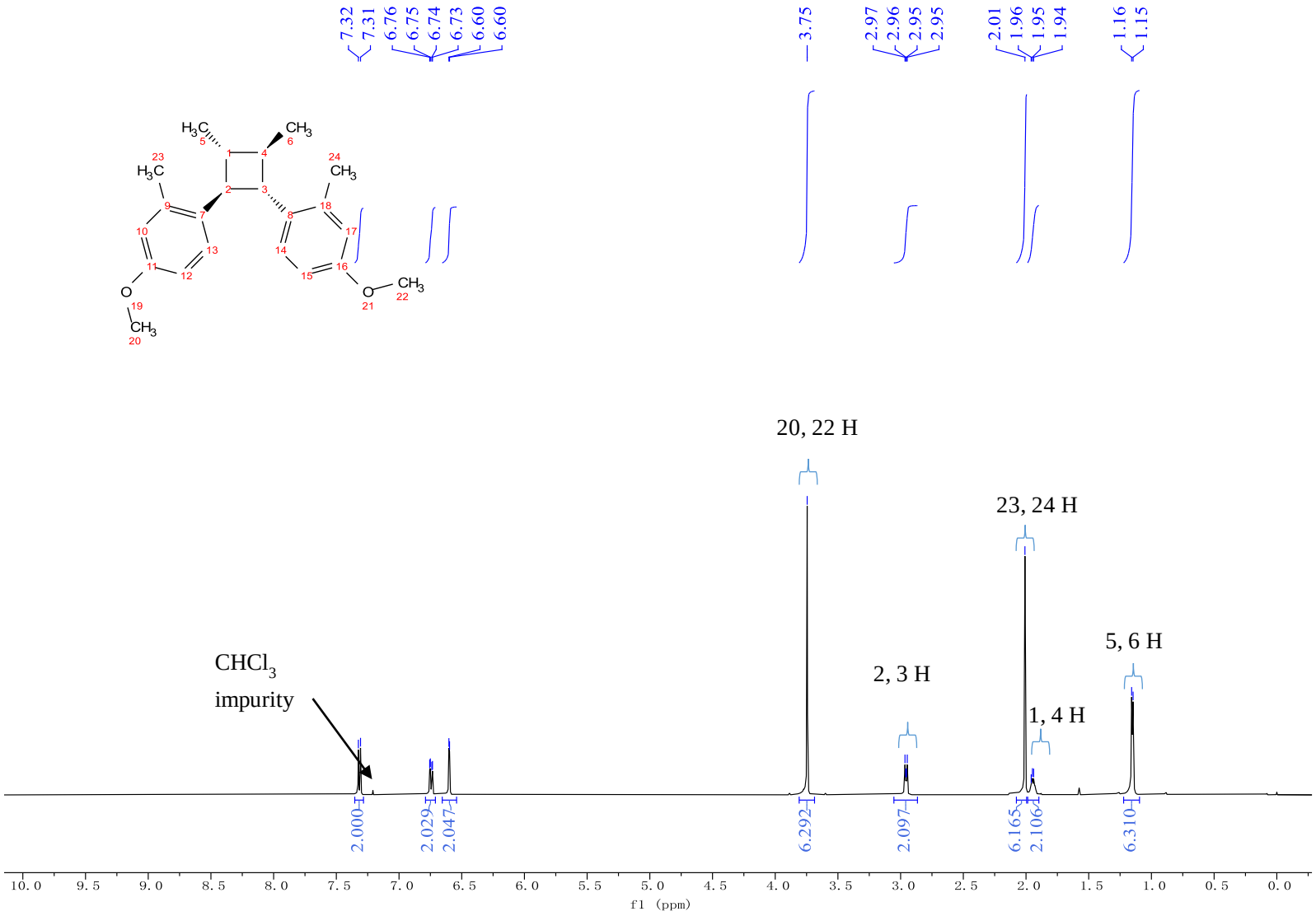
7.20
7.03
7.03
7.01
7.01
6.85
6.83
3.86
2.75
2.74
2.73
2.73
1.83
1.82
1.81
1.80
1.18
1.17
H3C
CH3
Cl
Cl
O
O
CH3
CH3
22, 24 H
CHCl3
impurity
2, 3 H
1, 4 H
5, 6 H
1.947
2.015
1.980
6.094
2.000
1.918
6.420
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


153.37
136.48
128.40
126.03
122.24
112.01
56.15
52.20
43.16
18.68
CHCl3
impurity
f1 (ppm)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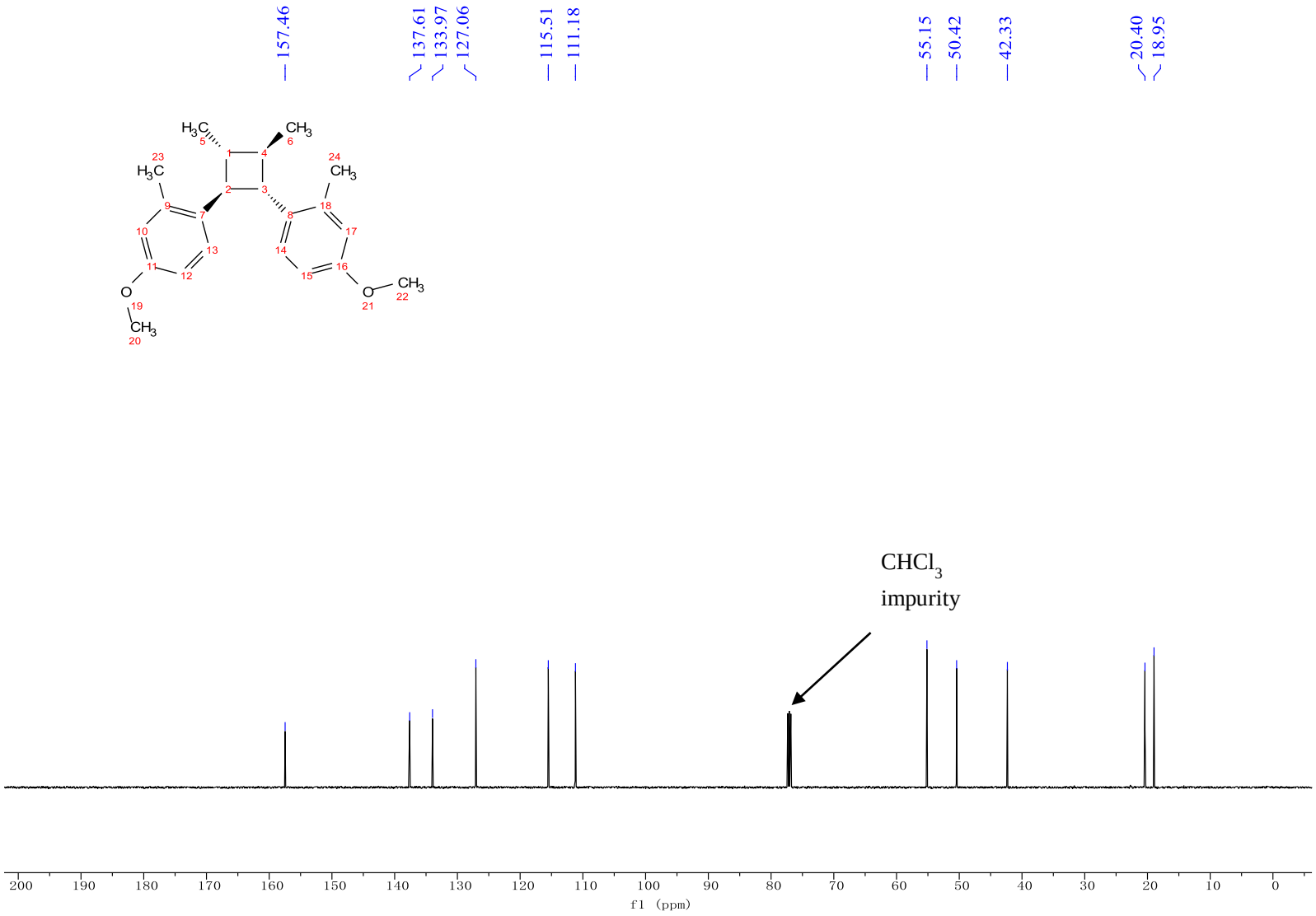
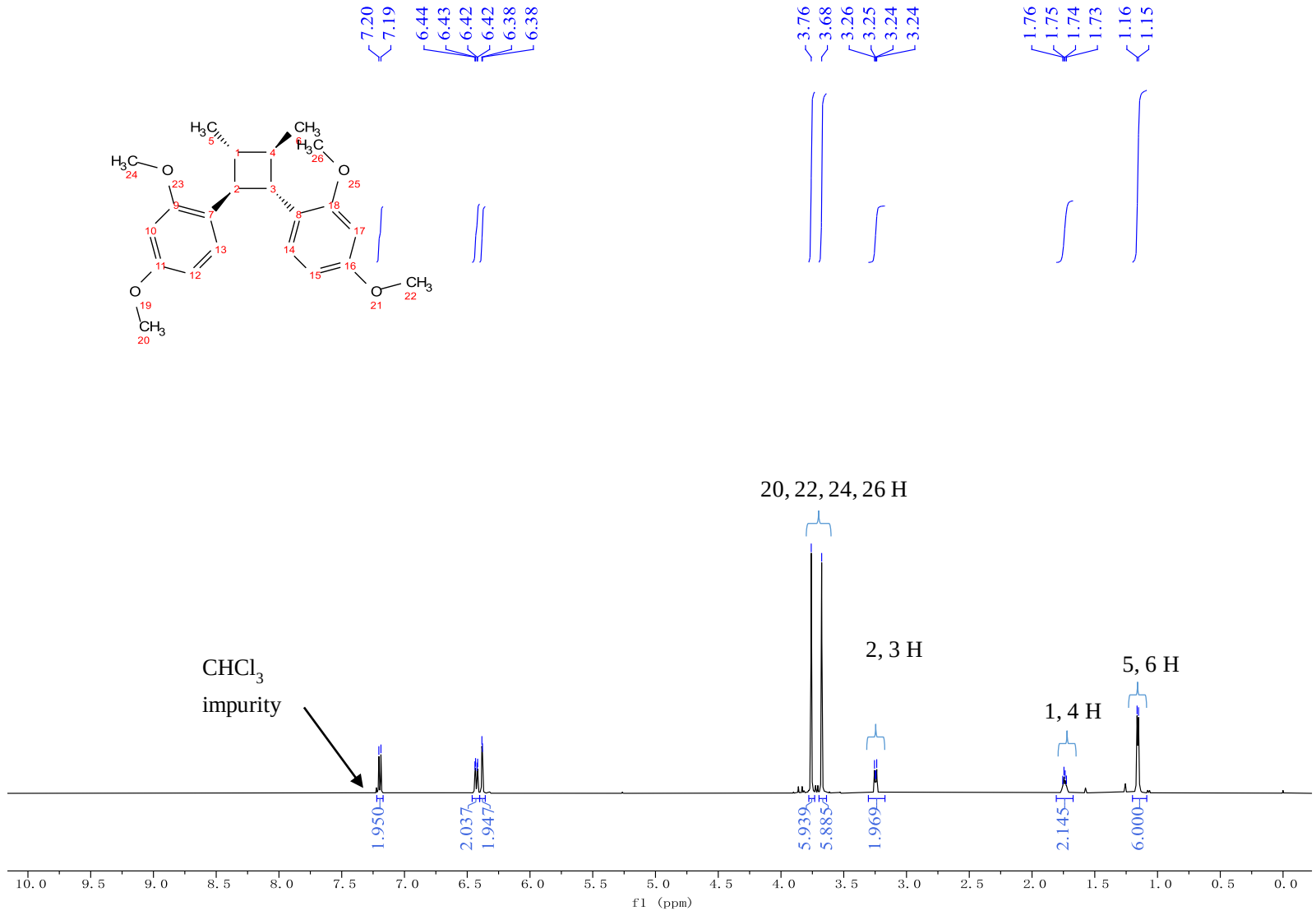
7.31
6.76
6.75
6.74
6.73
6.60
6.60
3.75
2.97
2.96
2.95
2.95
2.01
1.96
1.95
1.94
1.16
1.15
20, 22 H
23, 24 H
5, 6 H
2, 3 H
1, 4 H
CHCl3
impurity
2.000
2.029
2.047
6.292
2.097
6.165
2.106
6.310
f1 (ppm)


157.46
137.61
133.97
127.06
115.51
111.18
55.15
50.42
42.33
20.40
18.95
CHCl3
impurity
f1 (ppm)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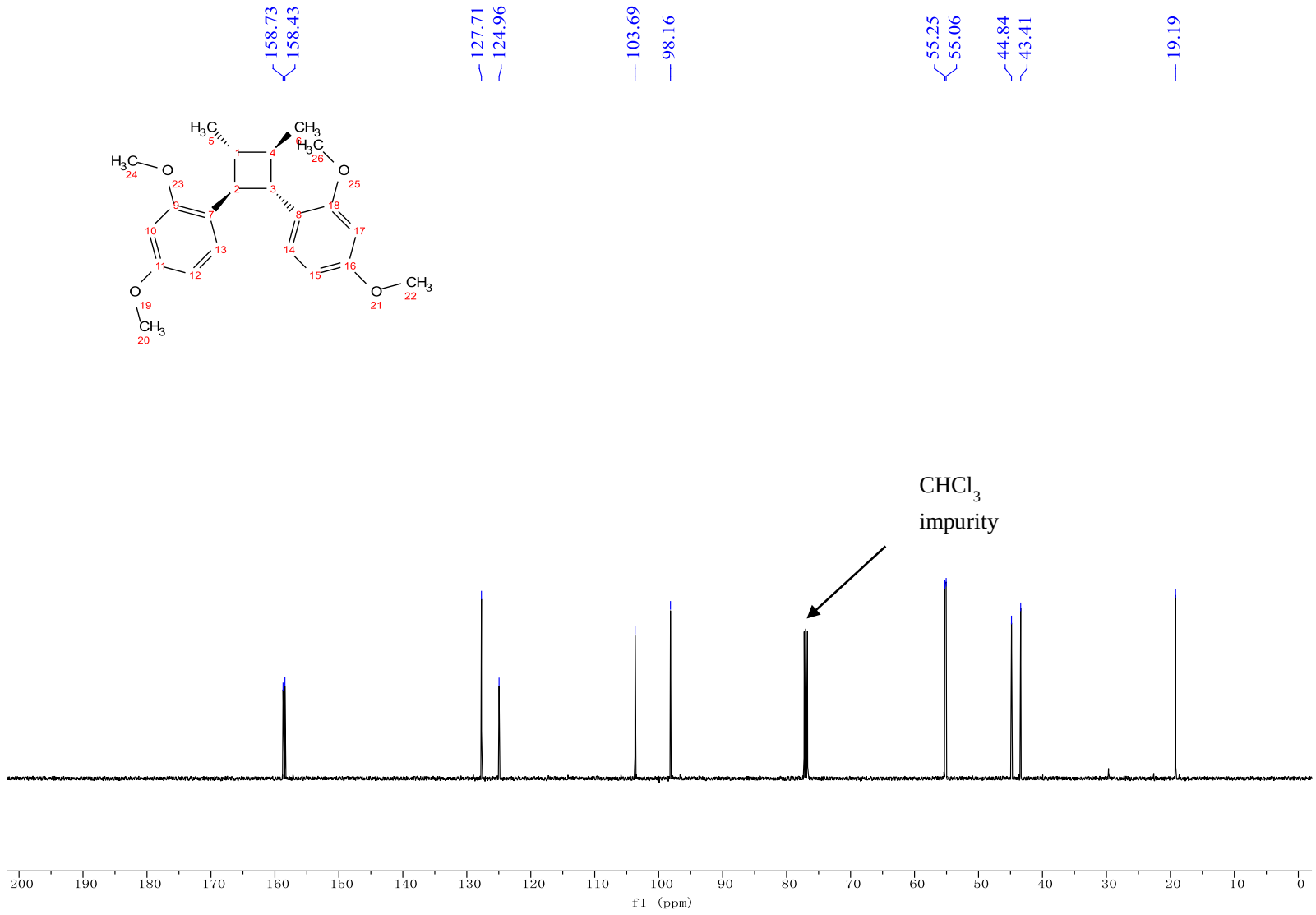
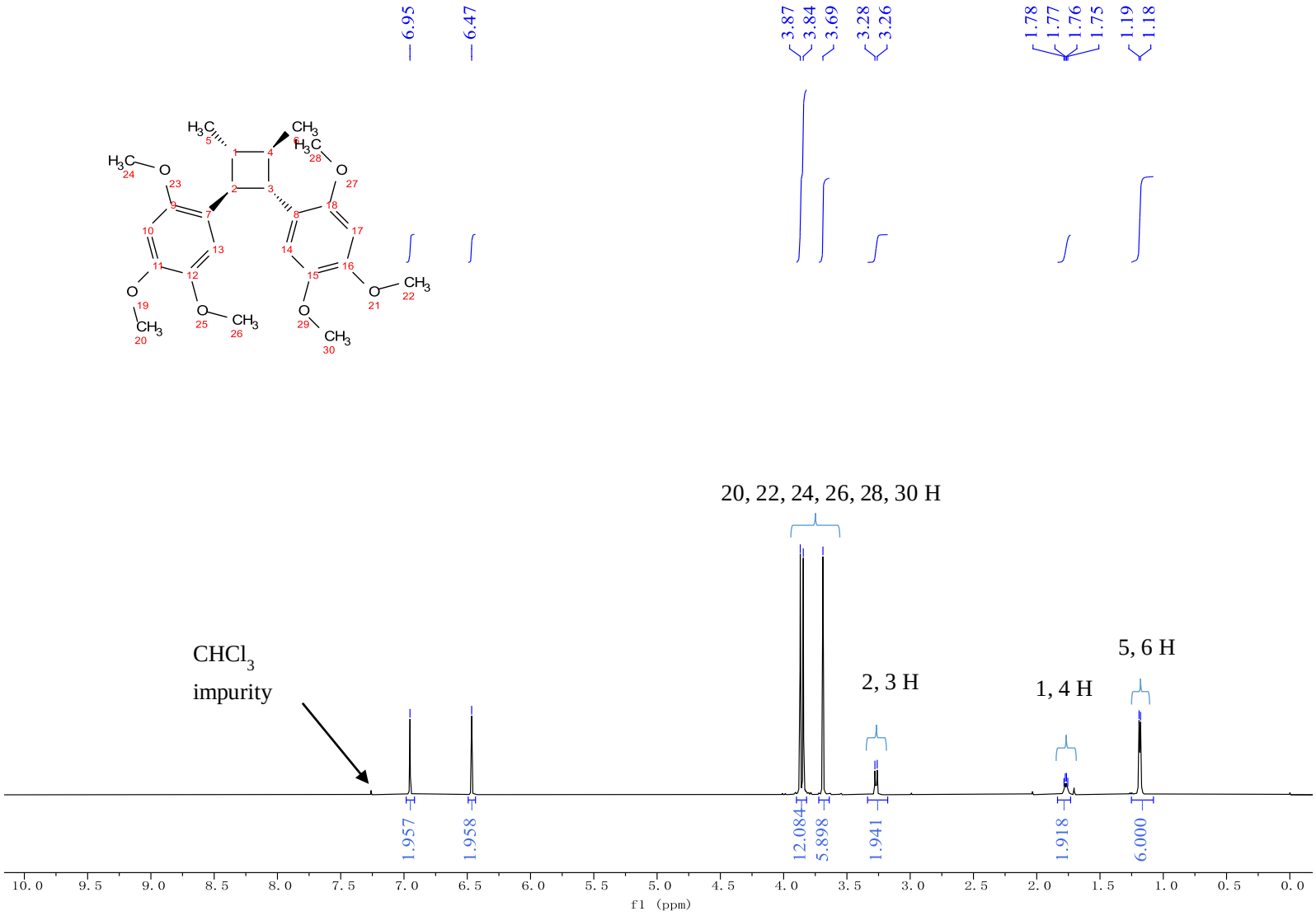
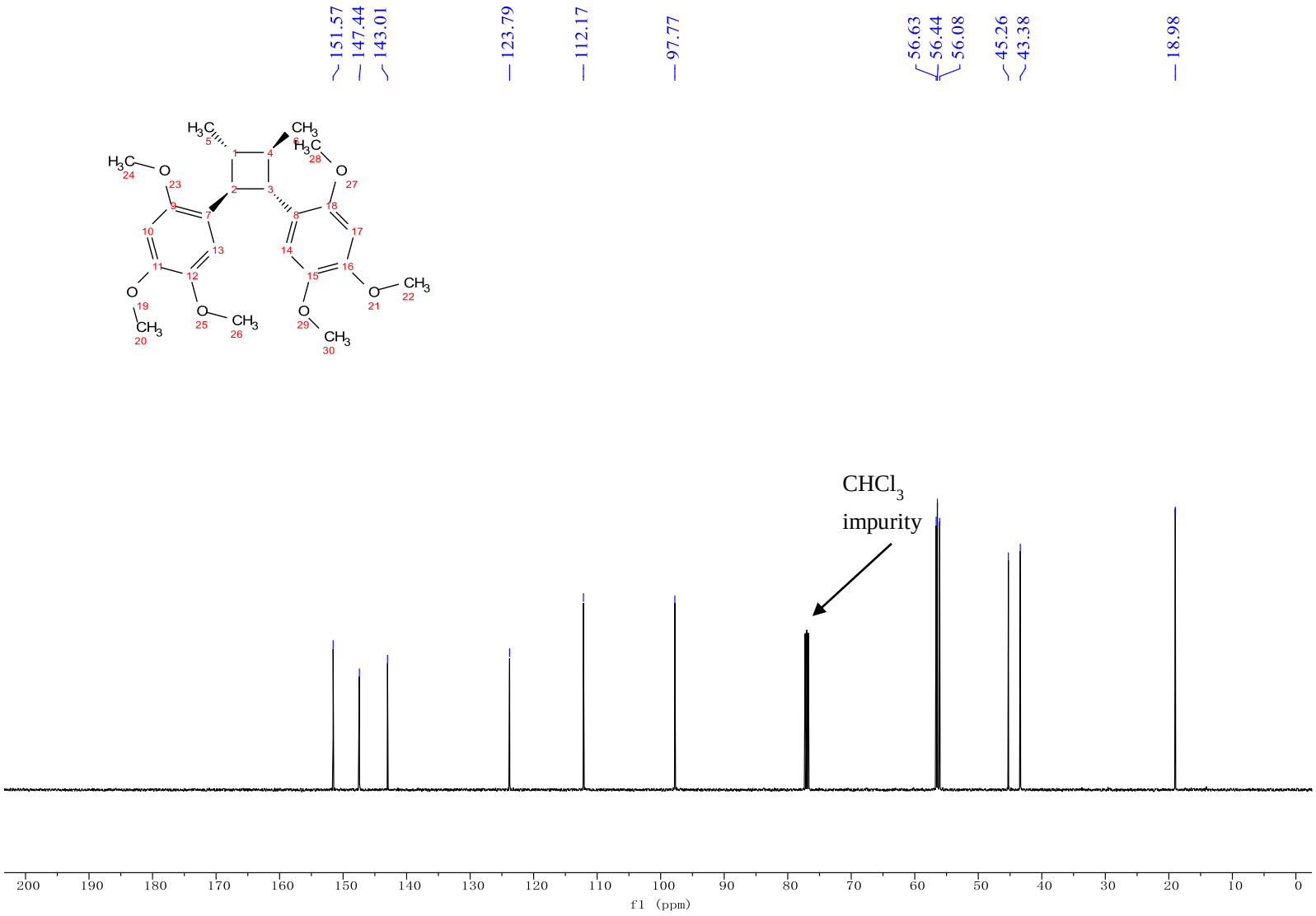
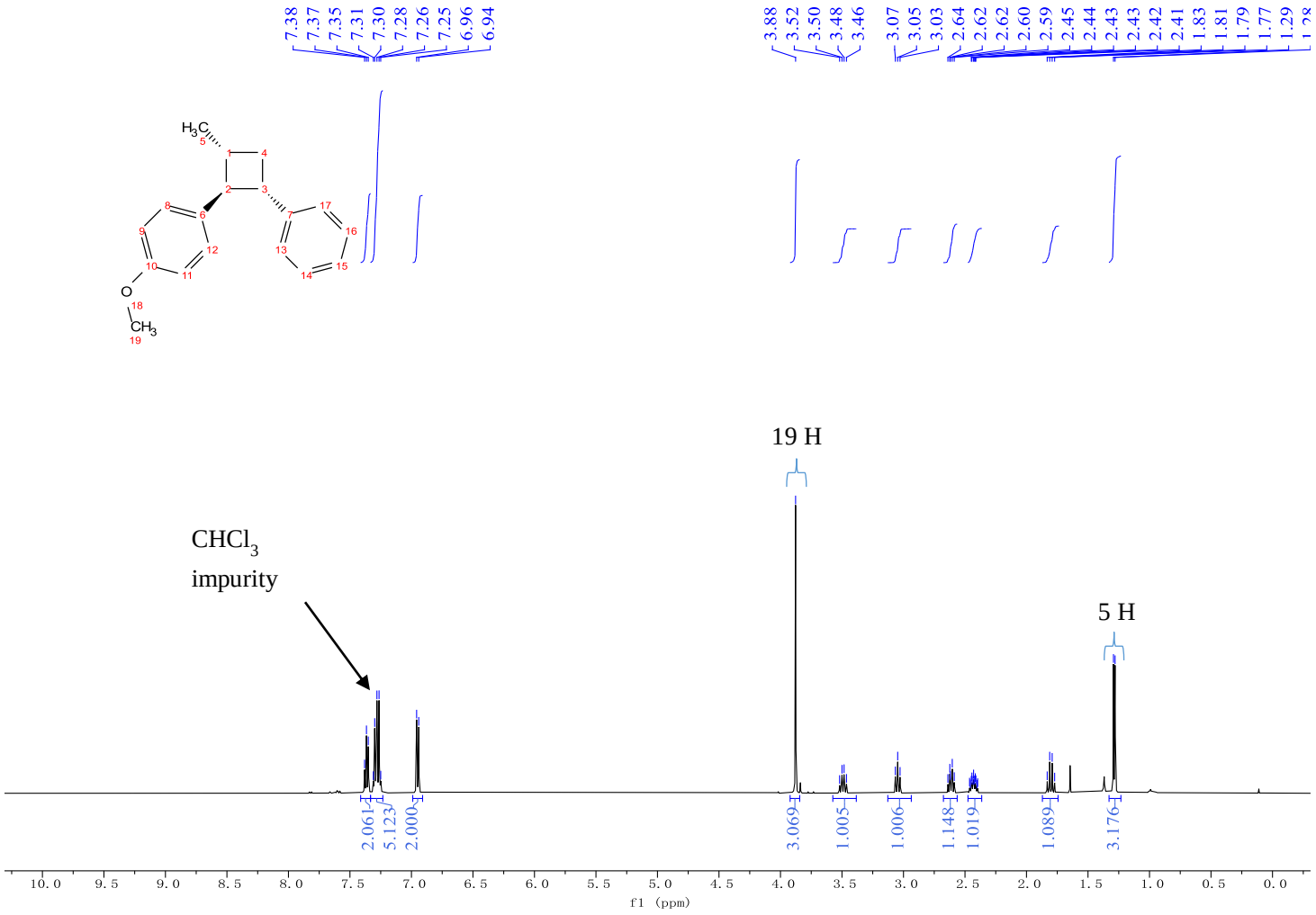
7.19
6.44
6.43
6.42
6.42
6.38
6.38
3.76
3.68
3.26
3.25
3.24
3.24
1.76
1.75
1.74
1.73
1.16
1.15
20, 22, 24, 26 H
2, 3 H
5, 6 H
1, 4 H
CHCl3
impurity
1.950
2.037
1.947
5.939
5.885
1.969
2.145
6.000
f1 (ppm)


158.73
158.43
127.71
124.96
103.69
98.16
55.25
55.06
44.84
43.41
19.19
CHCl3
impurity
f1 (ppm)


6.95
6.47
3.87
3.84
3.69
3.28
3.26
1.78
1.77
1.76
1.75
1.19
1.18
20, 22, 24, 26, 28, 30 H
CHCl3
impurity
2, 3 H
1, 4 H
5, 6 H
1.957
1.958
12.084
5.898
1.941
1.918
6.000
f1 (ppm)


151.57
147.44
143.01
123.79
112.17
97.77
56.63
56.44
56.08
45.26
43.38
18.98
CHCl3
impurity
f1 (ppm)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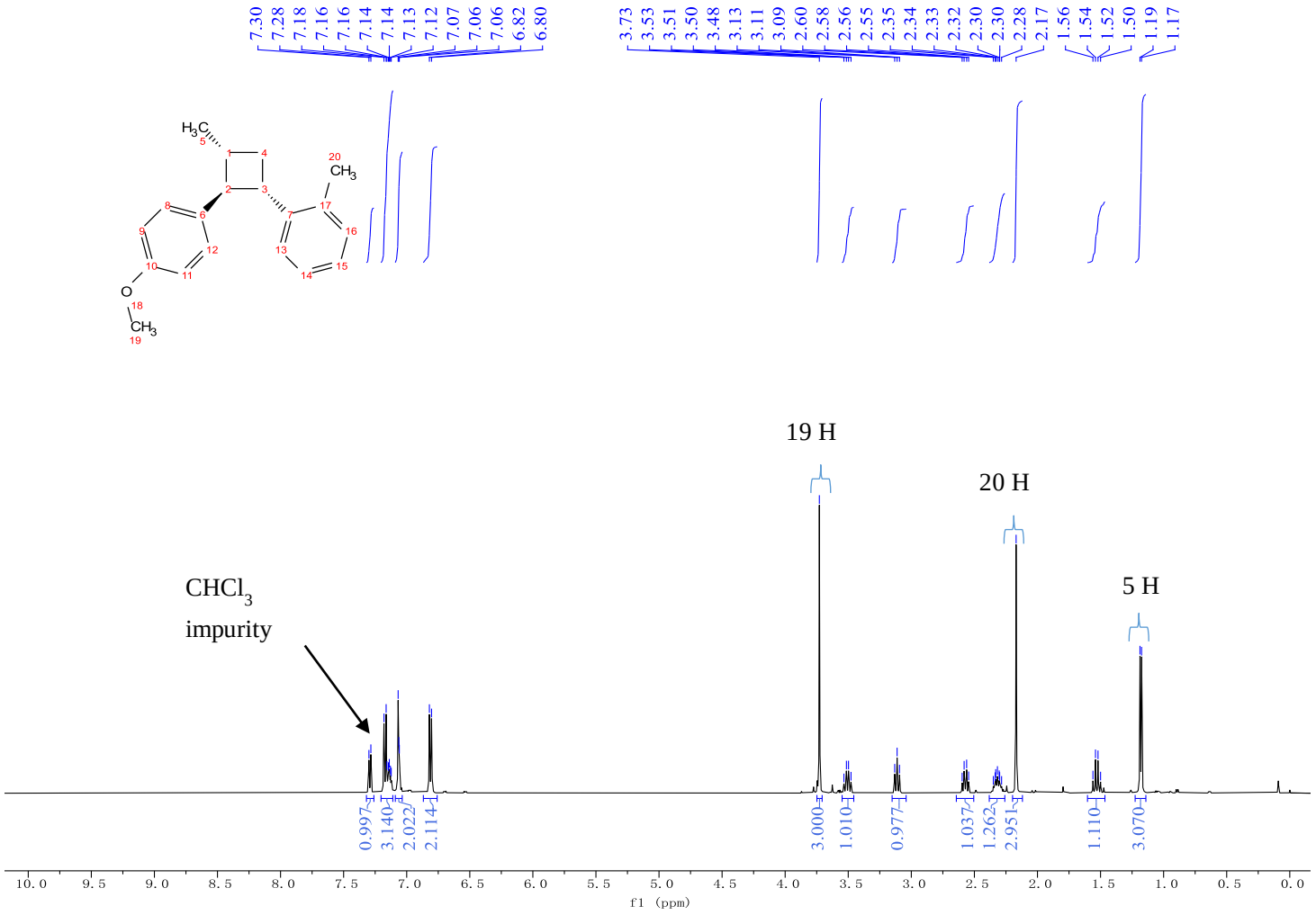
7.37
7.35
7.31
7.30
7.28
7.26
7.25
6.96
6.94
3.88
3.52
3.50
3.48
3.46
3.07
3.05
3.03
2.64
2.62
2.62
2.60
2.59
2.45
2.44
2.43
2.43
2.42
2.41
1.83
1.81
1.79
1.77
1.29
H3C
5
1
4
2
3
8
6
7
17
16
9
12
13
10
11
14
15
O
18
CH3
19
19 H
CHCl3
impurity
5 H
2.061
5.123
2.000
3.069
1.005
1.006
1.148
1.019
1.089
3.176
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
f1 (ppm)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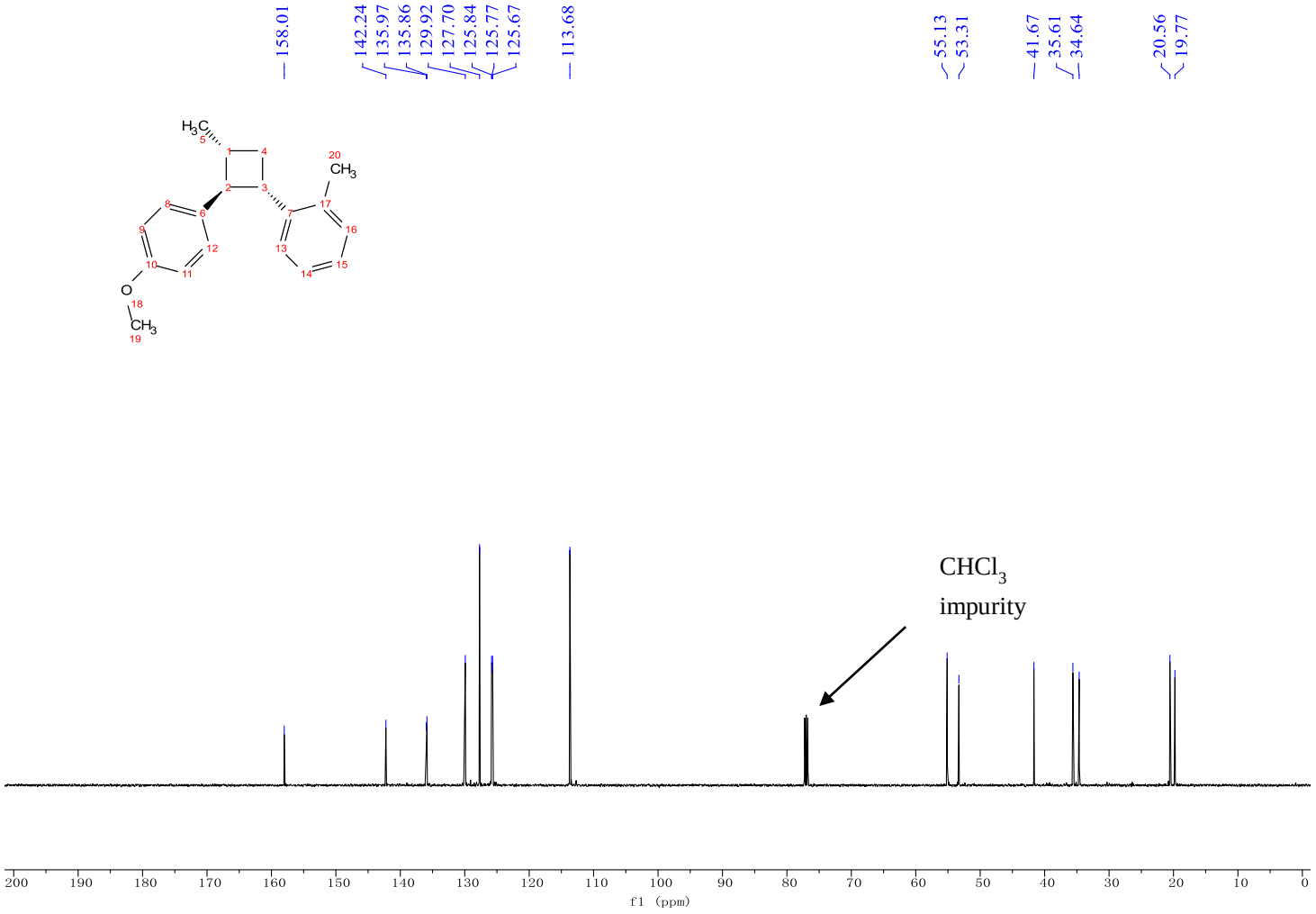
7.28
7.18
7.16
7.16
7.14
7.14
7.13
7.12
7.07
7.06
7.06
6.82
6.80
3.73
3.53
3.51
3.50
3.48
3.13
3.11
3.09
2.60
2.58
2.56
2.55
2.35
2.34
2.33
2.32
2.30
2.30
2.28
2.17
1.56
1.54
1.52
1.50
1.19
1.17
19 H
20 H
5 H
CHCl3
impurity
0.997
3.140
2.022
2.114
3.000
1.010
0.977
1.037
1.262
2.951
1.110
3.070
f1 (ppm)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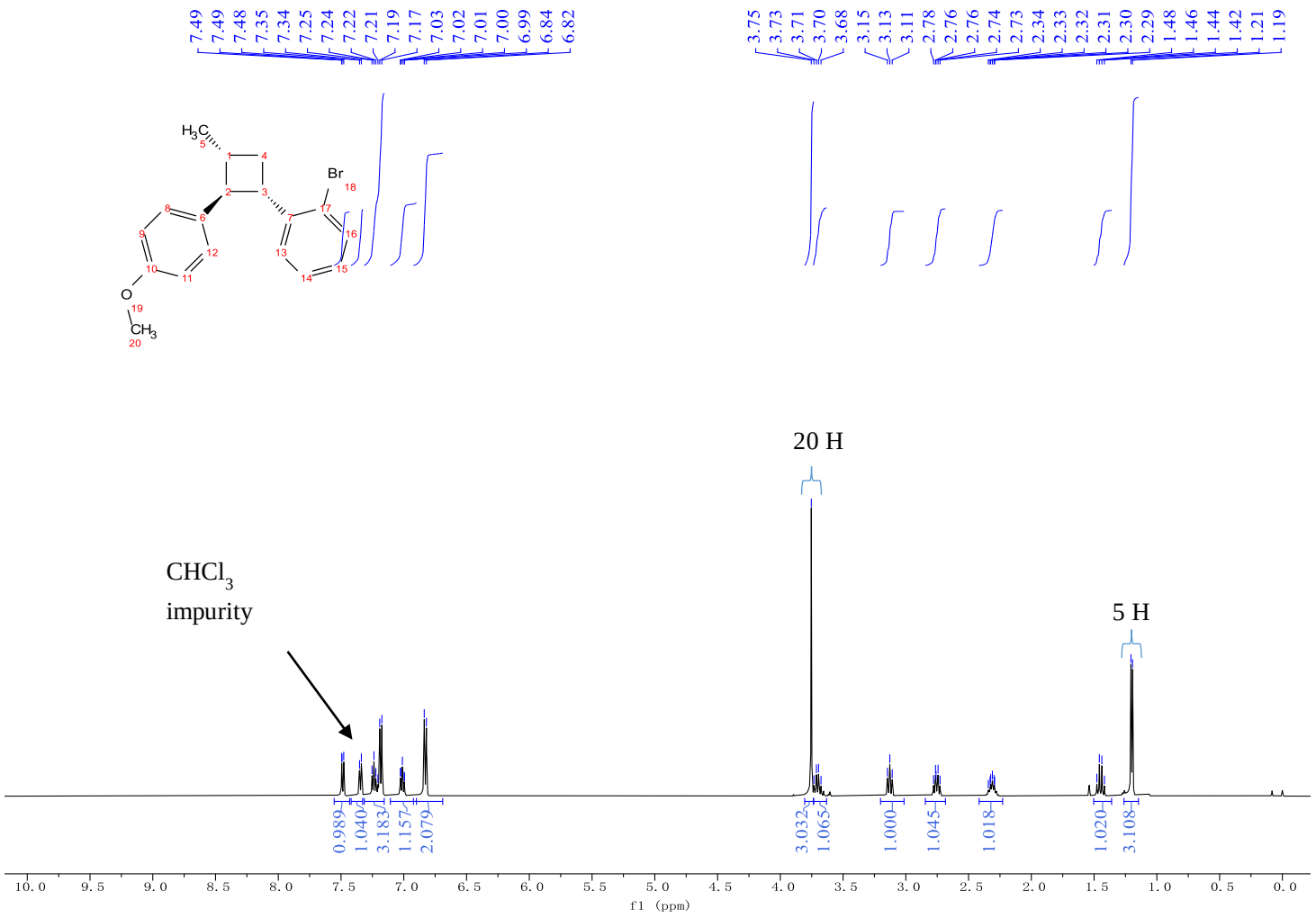
142.24
135.97
135.86
129.92
127.70
125.84
125.77
125.67
113.68
55.13
53.31
41.67
35.61
34.64
20.56
19.77
CHCl3
impurity
f1 (ppm)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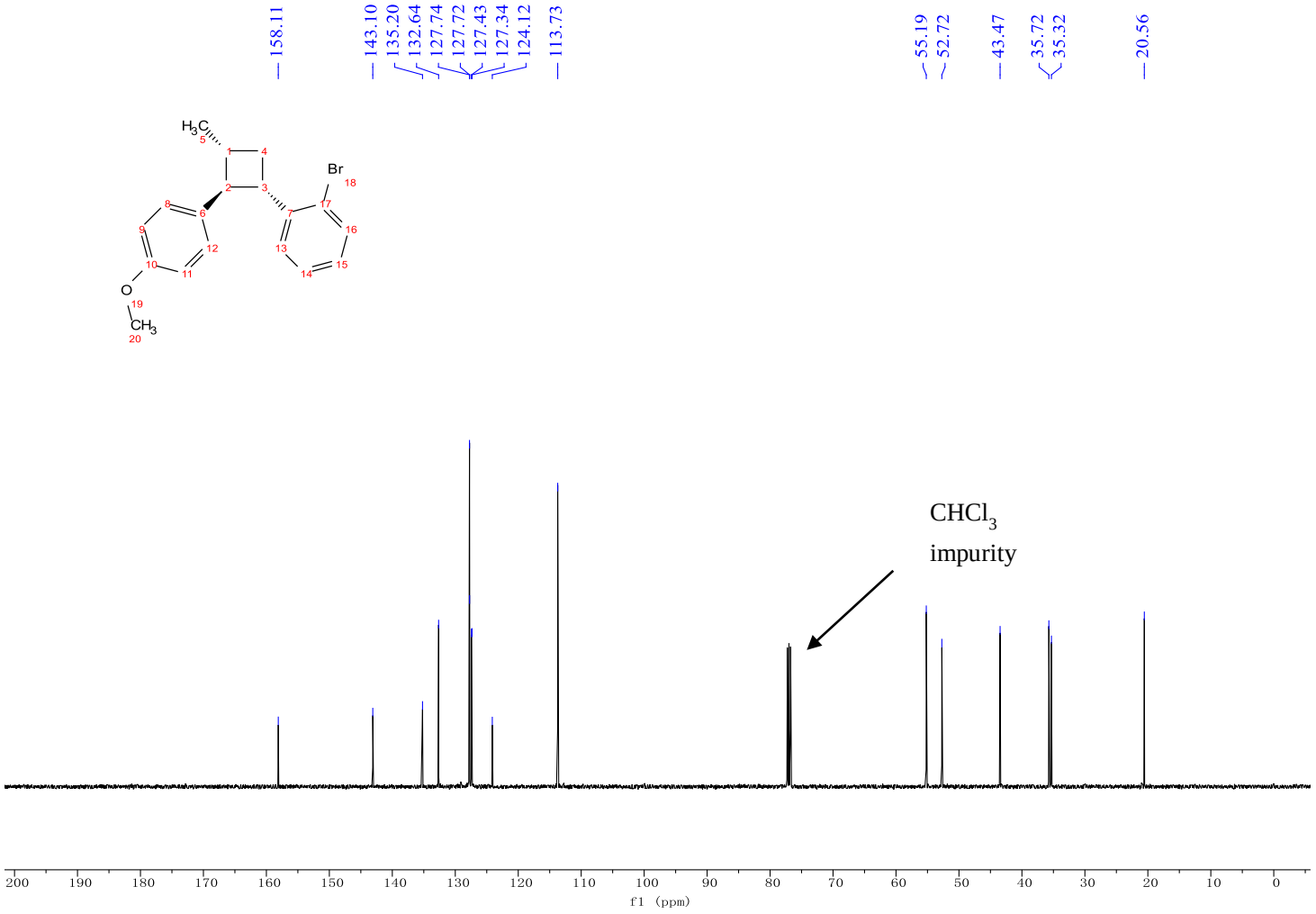
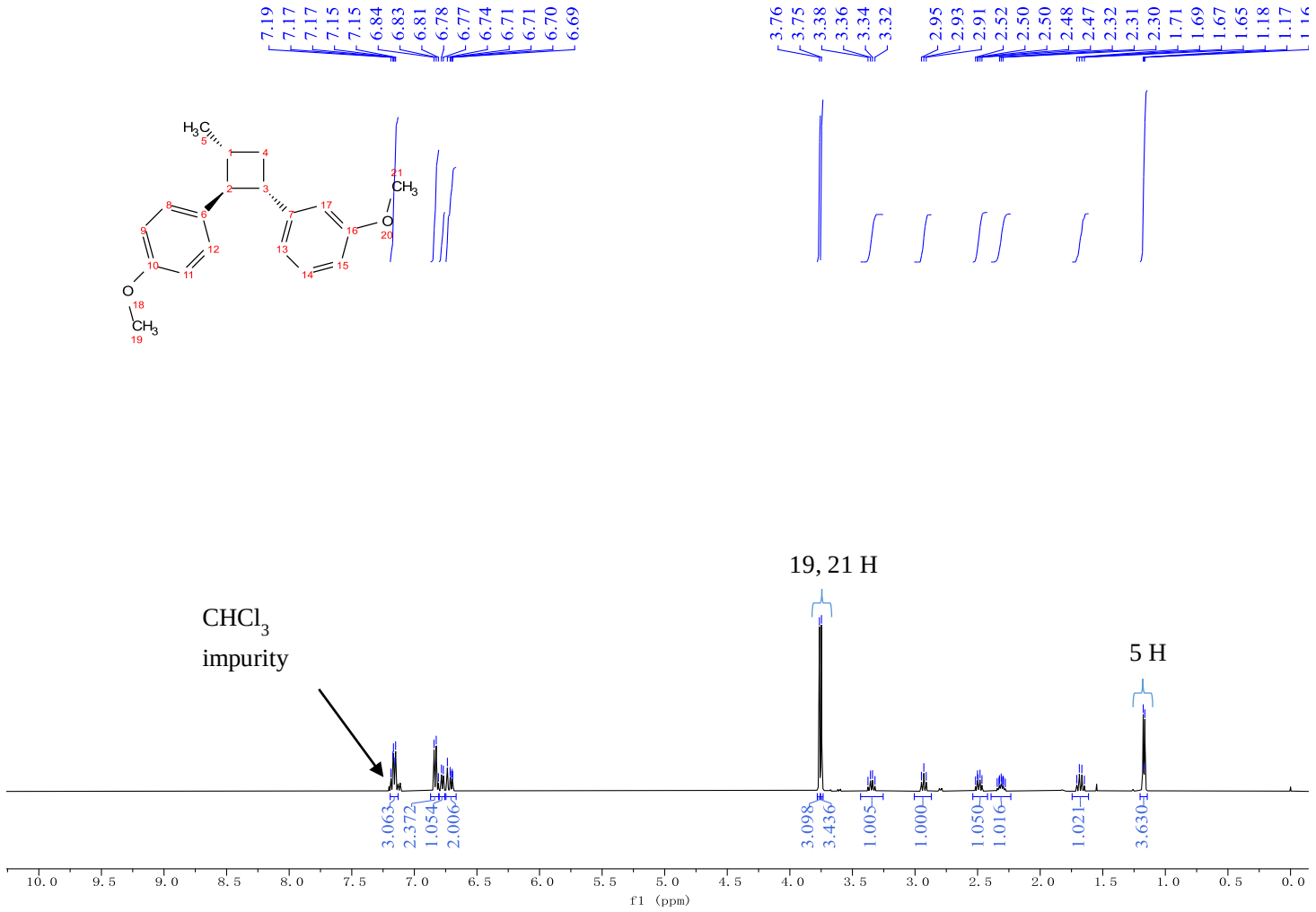
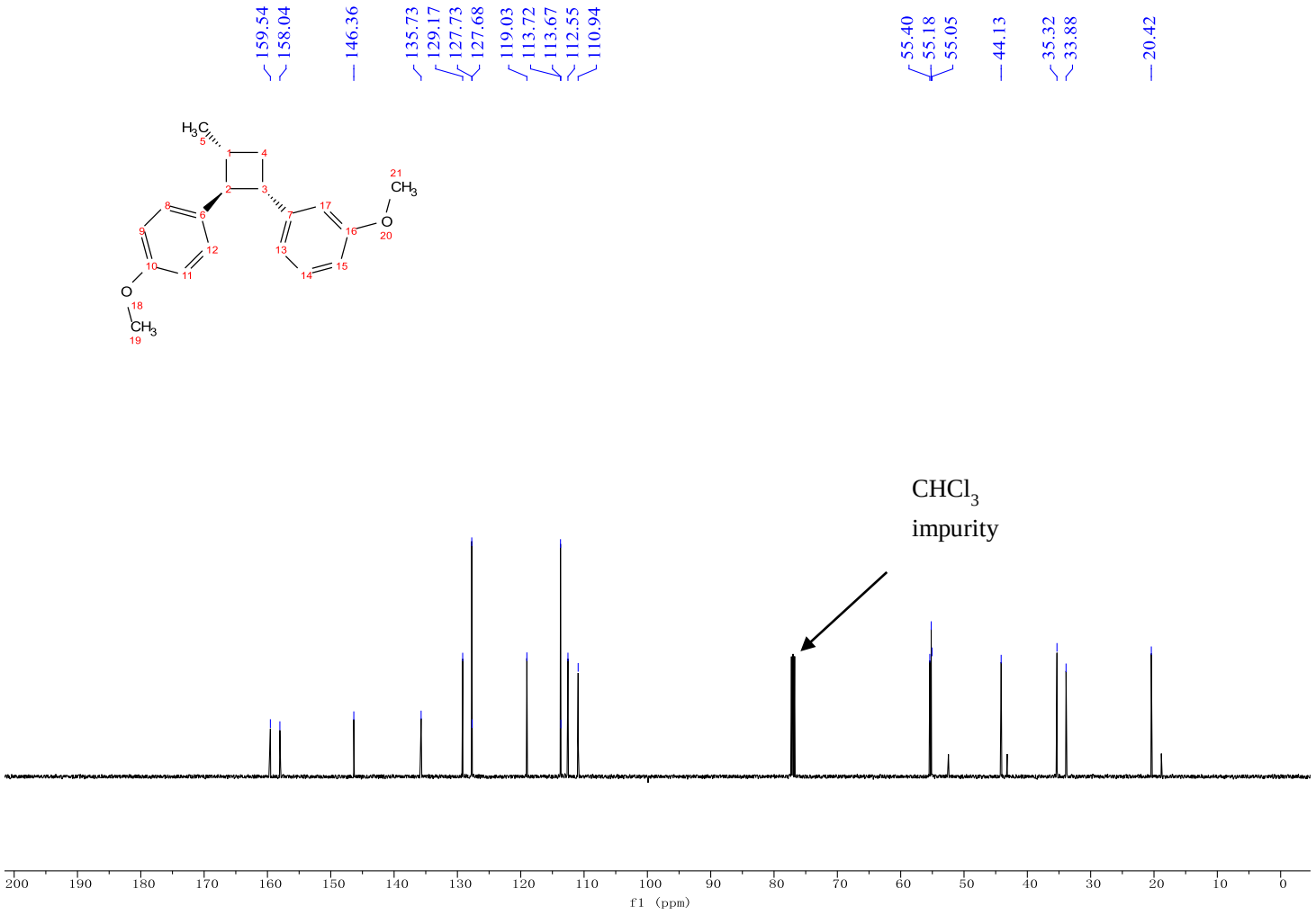
7.49
7.48
7.35
7.34
7.25
7.24
7.22
7.21
7.19
7.17
7.03
7.02
7.01
7.00
6.99
6.84
6.82
3.75
3.73
3.71
3.70
3.68
3.15
3.13
3.11
2.78
2.76
2.76
2.74
2.73
2.34
2.33
2.32
2.31
2.30
2.29
1.48
1.46
1.44
1.42
1.21
1.19
Br
20 H
CHCl3
impurity
5 H
0.989
1.040
3.183
1.157
2.079
3.032
1.065
1.000
1.045
1.018
1.020
3.108
f1 (ppm)


158.11
143.10
135.20
132.64
127.74
127.72
127.43
127.34
124.12
113.73
55.19
52.72
43.47
35.72
35.32
20.56
Br
O
CH3
CHCl3
impurity
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.19
7.17
7.17
7.15
7.15
6.84
6.83
6.81
6.78
6.77
6.74
6.71
6.71
6.70
6.69
3.76
3.75
3.38
3.36
3.34
3.32
2.95
2.93
2.91
2.52
2.50
2.50
2.48
2.47
2.32
2.31
2.30
1.71
1.69
1.67
1.65
1.18
1.17
H3C
CH3
O
CH3
19, 21 H
CHCl3
impurity
5 H
3.063
2.372
1.054
2.006
3.098
3.436
1.005
1.000
1.050
1.016
1.021
3.630
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
f1 (ppm)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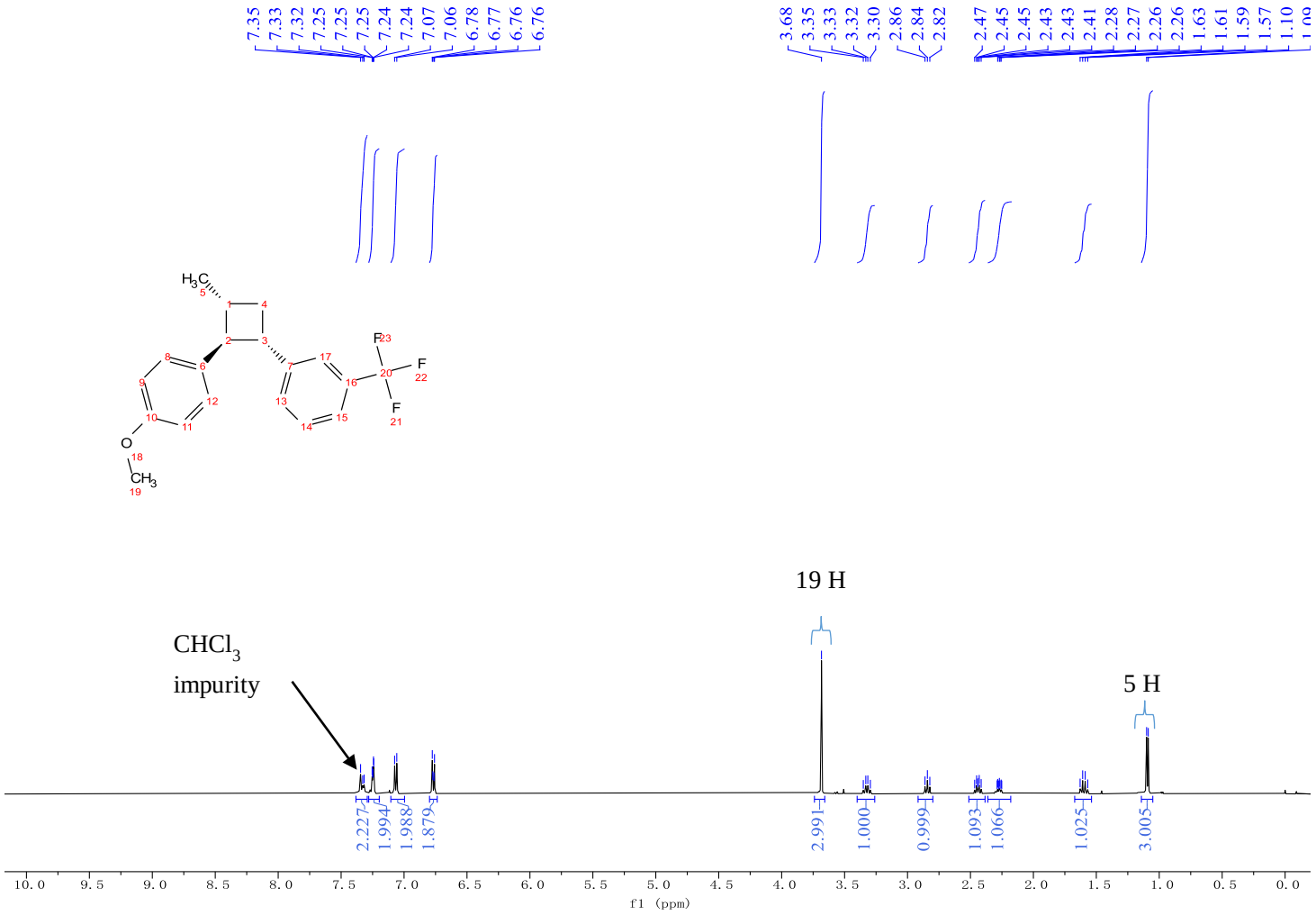
158.04
146.36
135.73
129.17
127.73
127.68
119.03
113.72
113.67
112.55
110.94
55.40
55.18
55.05
44.13
35.32
33.88
20.42
CHCl3
impurity
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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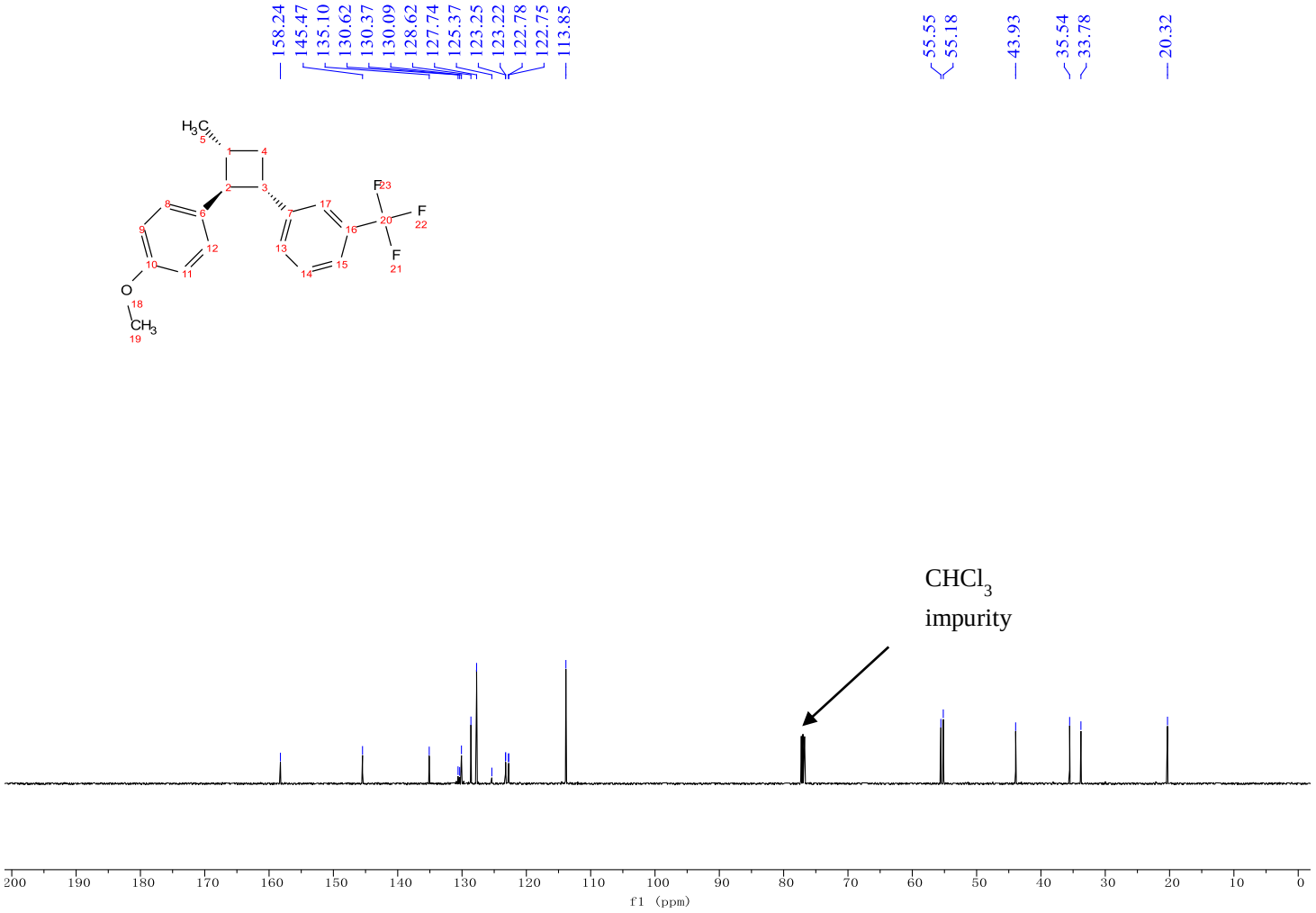
7.32
7.25
7.25
7.25
7.24
7.24
7.07
7.06
6.78
6.77
6.76
6.76
3.68
3.35
3.33
3.32
3.30
2.86
2.84
2.82
2.47
2.45
2.45
2.43
2.43
2.41
2.28
2.27
2.26
2.26
1.63
1.61
1.59
1.57
1.10
H3C
O
CH3
F
F
F
19 H
5 H
CHCl3
impurity
2.227
1.994
1.988
1.879
2.991
1.000
0.999
1.093
1.066
1.025
3.005
10.0 9.5 9.0 8.5 8.0 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
f1 (ppm)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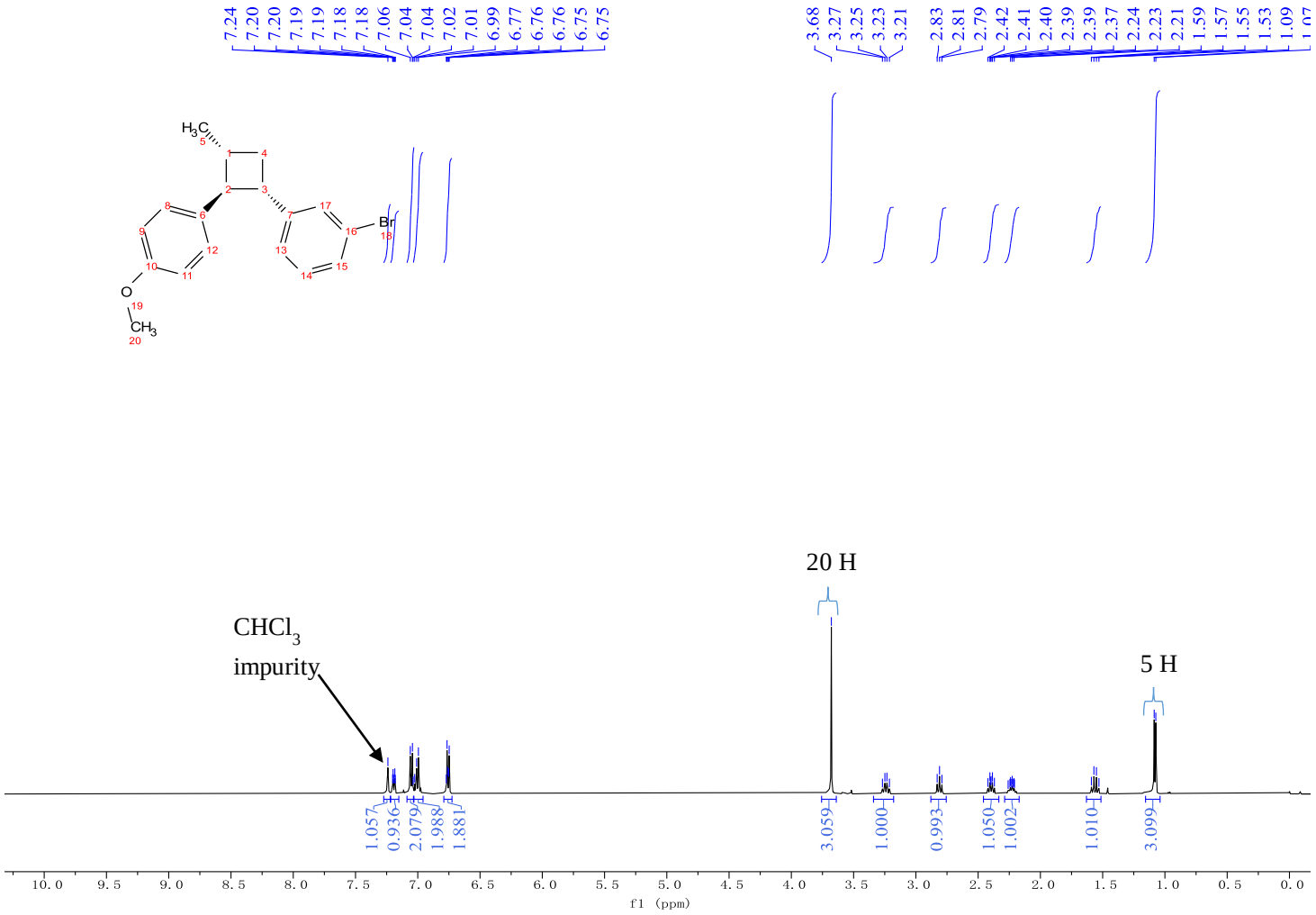
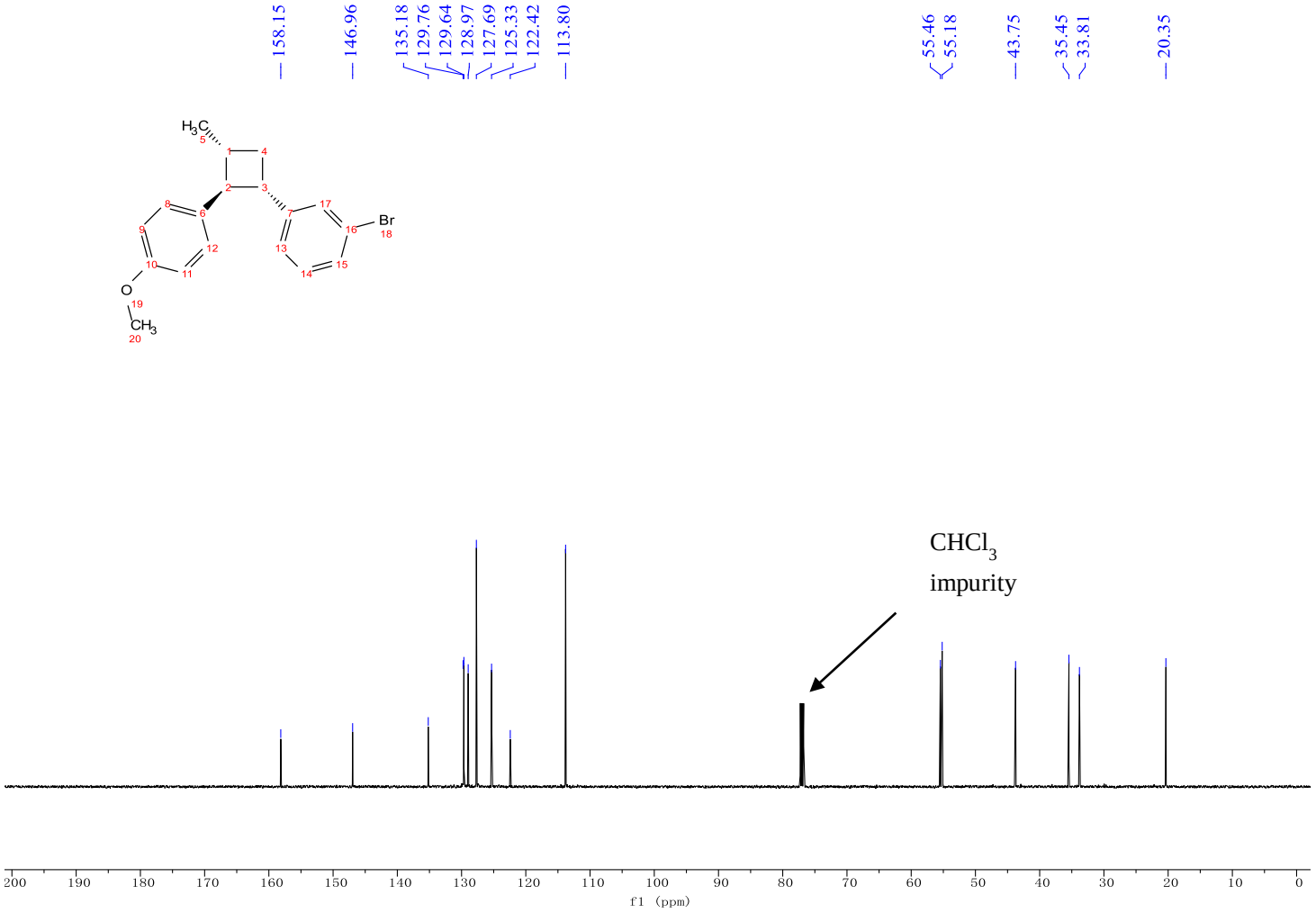
130.09
128.62
127.74
125.37
123.25
123.22
122.78
122.75
113.85
55.55
55.18
43.93
35.54
33.78
20.32
CHCl3
impurity
f1 (ppm)


7.24
7.20
7.20
7.19
7.19
7.18
7.18
7.06
7.04
7.04
7.02
7.01
6.99
6.77
6.76
6.76
6.75
6.75
3.68
3.27
3.25
3.23
3.21
2.83
2.81
2.79
2.42
2.41
2.40
2.39
2.39
2.37
2.24
2.23
2.21
1.59
1.57
1.55
1.53
1.09
H3C
Br
O
CH3
CHCl3
impurity
20 H
5 H
1.057
0.936
2.079
1.988
1.881
3.059
1.000
0.993
1.050
1.002
1.010
3.099
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
f1 (ppm)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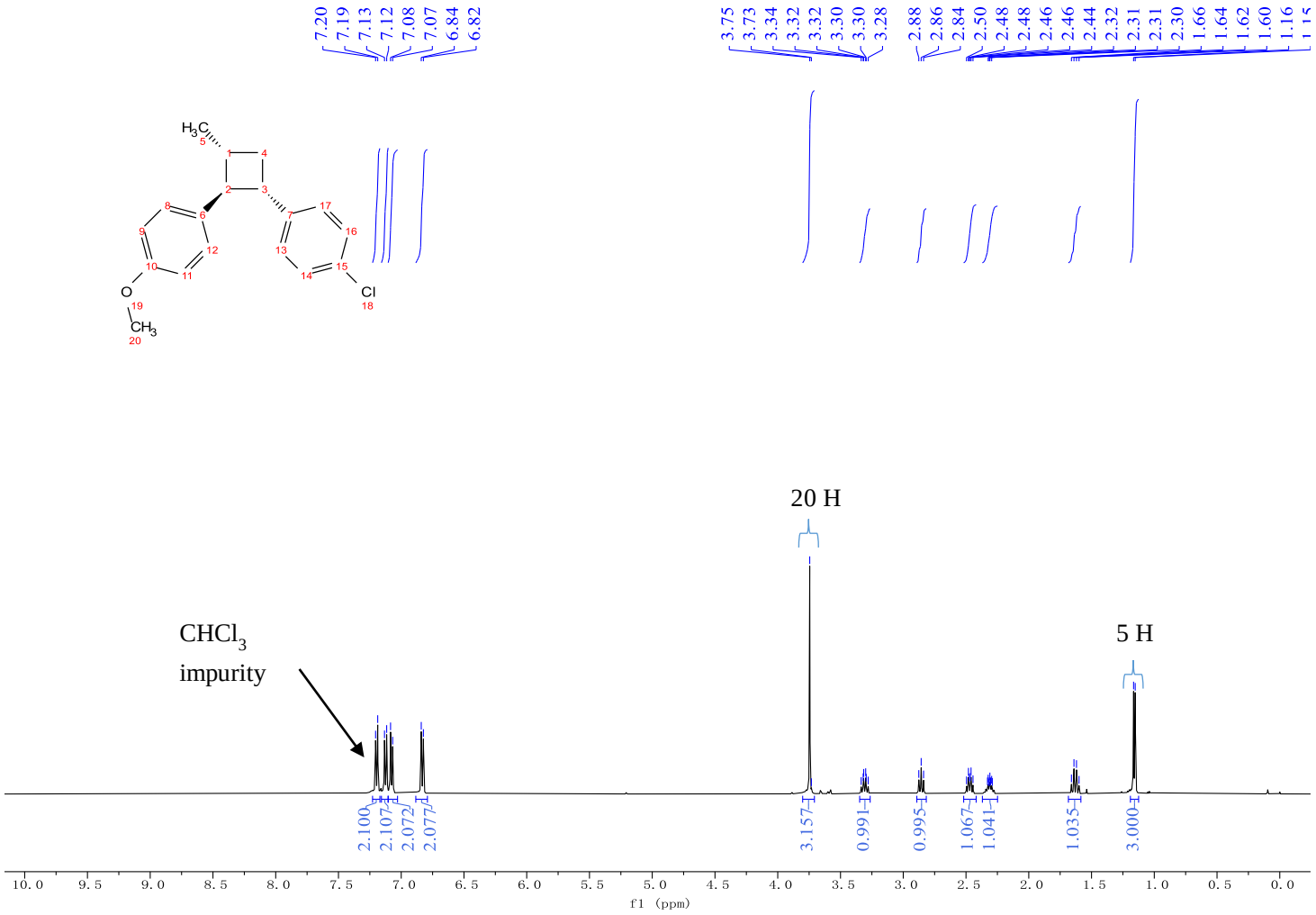
128.97
127.69
125.33
122.42
113.80
55.46
55.18
43.75
35.45
33.81
20.35
Br
O
CH3
CHCl3
impurity
f1 (ppm)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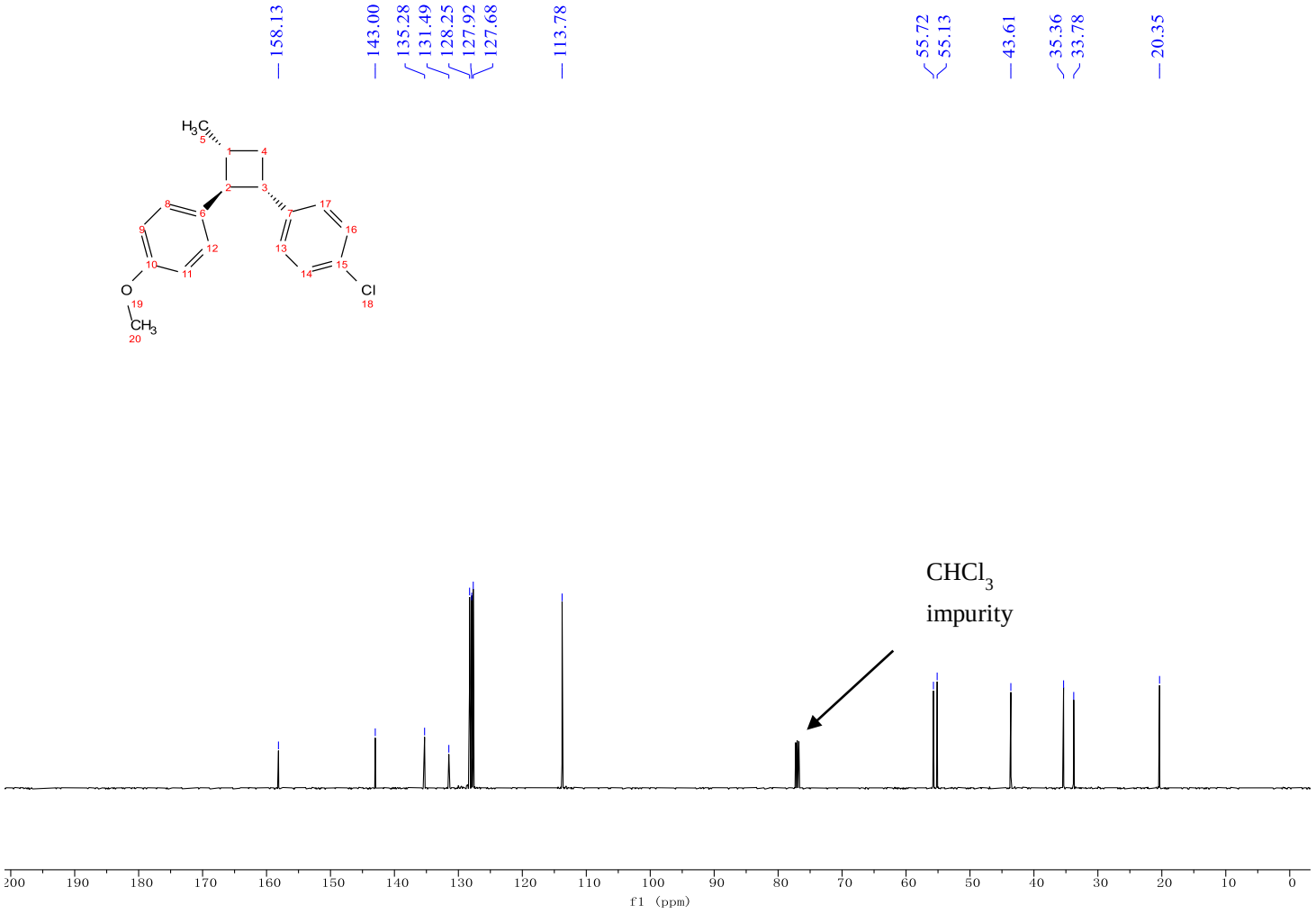
7.12
7.08
7.07
6.84
6.82
3.75
3.73
3.34
3.32
3.32
3.30
3.30
3.28
2.88
2.86
2.84
2.50
2.48
2.48
2.46
2.46
2.44
2.32
2.31
2.31
2.30
1.66
1.64
1.62
1.60
1.16
H3C
5
1
4
2
3
8
6
7
17
9
12
16
13
10
11
14
15
O
19
Cl
18
CH3
20
20 H
CHCl3
impurity
5 H
2.100
2.107
2.072
2.077
3.157
0.991
0.995
1.067
1.041
1.035
3.000
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
f1 (ppm)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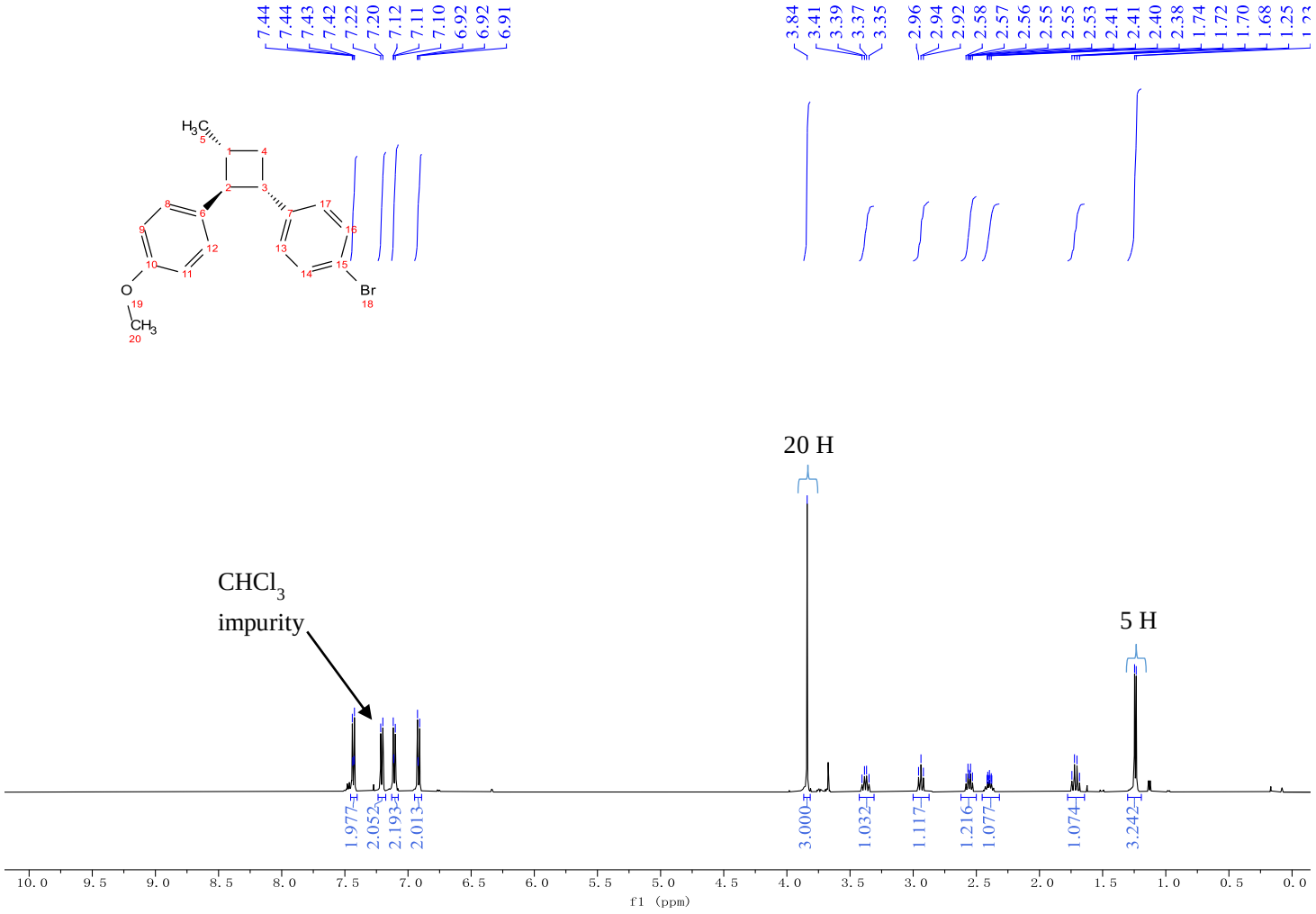
127.68
113.78
55.72
55.13
43.61
35.36
33.78
20.35
CHCl3
impurity
f1 (ppm)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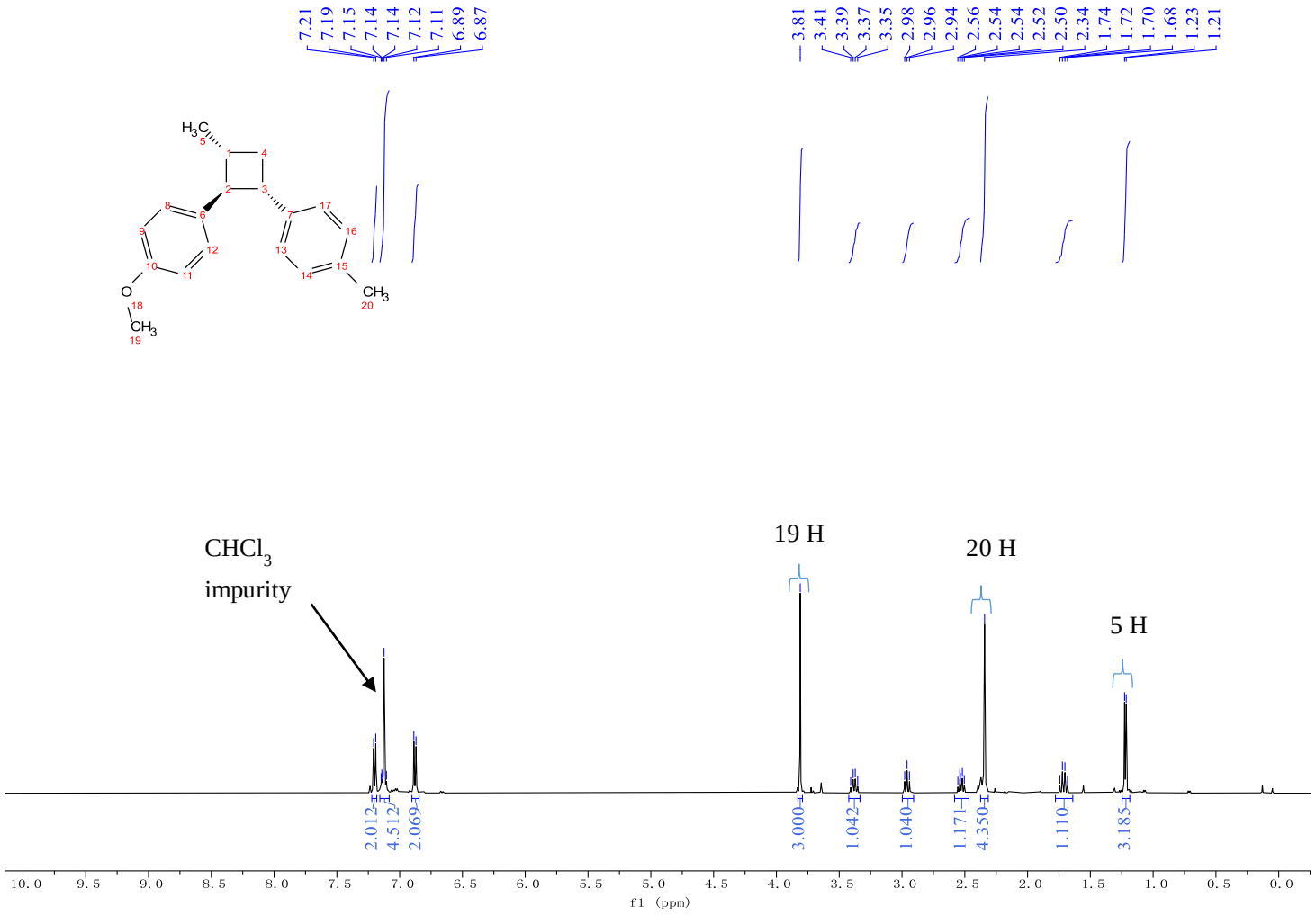
7.44
7.43
7.42
7.22
7.20
7.12
7.11
7.10
6.92
6.92
6.91
3.84
3.41
3.39
3.37
3.35
2.96
2.94
2.92
2.58
2.57
2.56
2.55
2.55
2.53
2.41
2.41
2.40
2.38
1.74
1.72
1.70
1.68
1.25
H3C
5
1
4
2
3
6
8
9
10
11
12
7
17
16
13
14
15
Br
18
O
19
CH3
20
20 H
CHCl3
impurity
5 H
1.977
2.052
2.193
2.013
3.000
1.032
1.117
1.216
1.077
1.074
3.242
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
f1 (ppm)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7.19
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7.14
7.14
7.12
7.11
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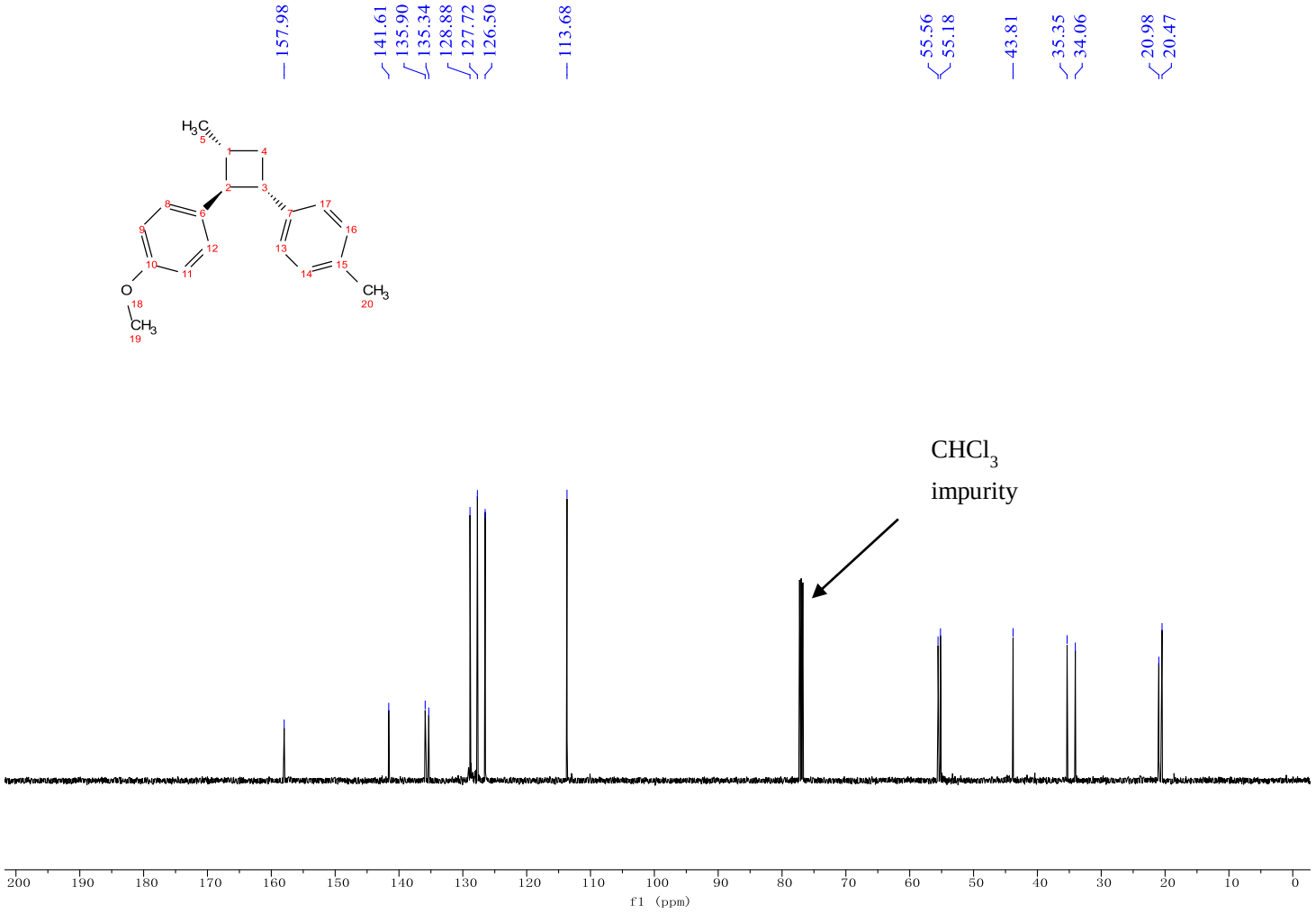
6.87
3.81
3.41
3.39
3.37
3.35
2.98
2.96
2.94
2.56
2.54
2.54
2.52
2.50
2.34
1.74
1.72
1.70
1.68
1.23
1.21
CHCl3
impurity
19 H
20 H
5 H
2.012
4.512
2.069
3.000
1.042
1.040
1.171
4.350
1.110
3.185
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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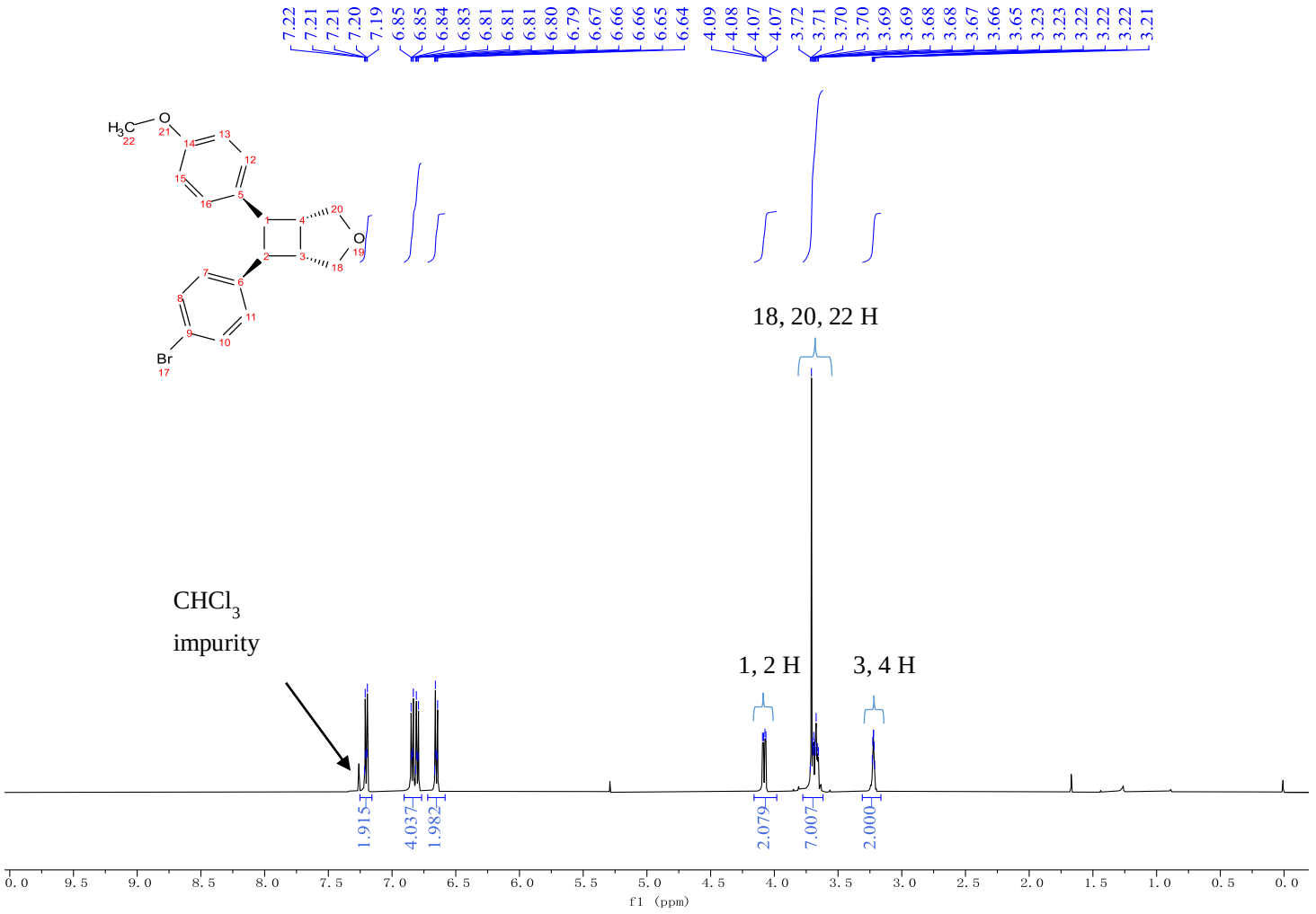
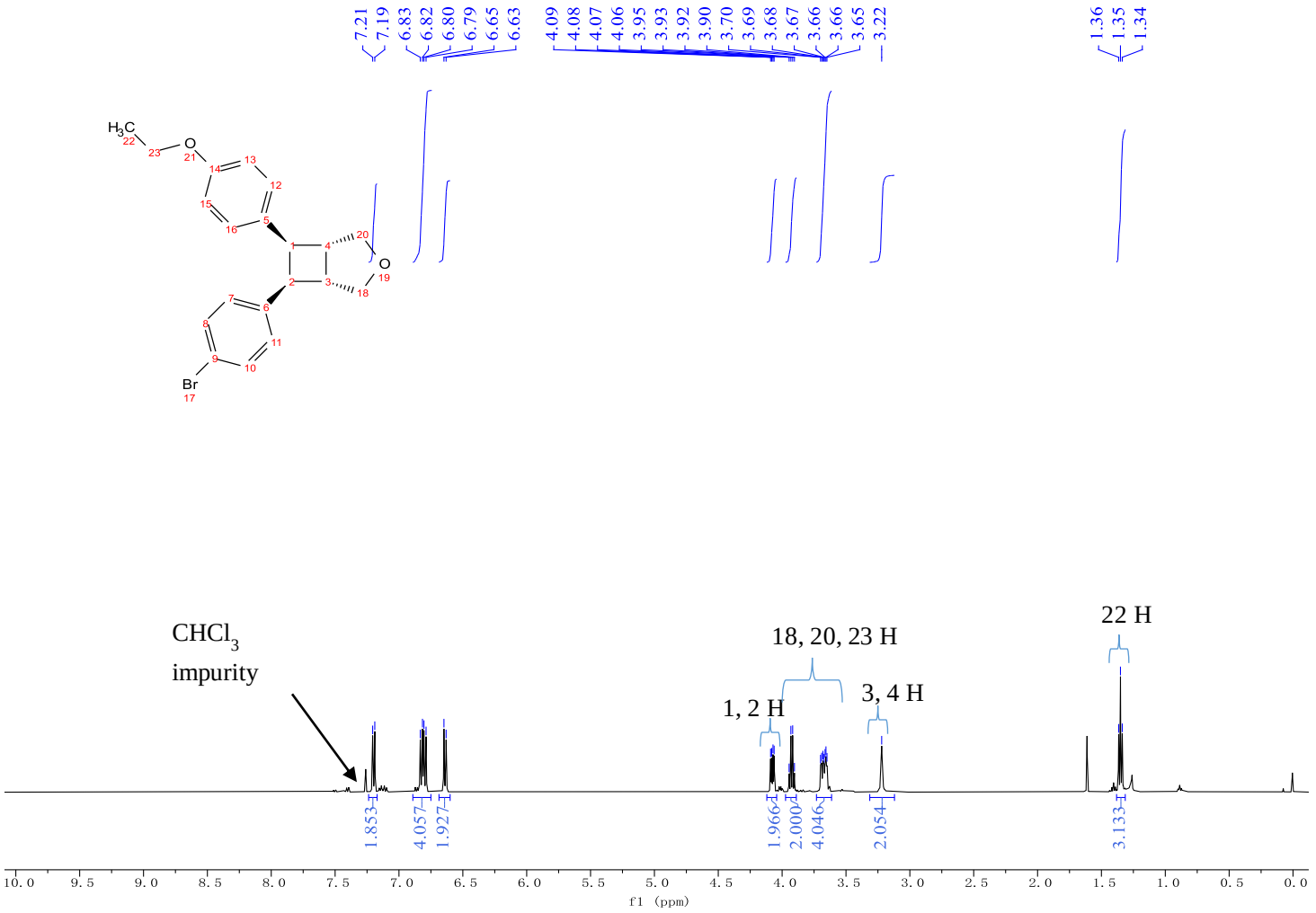
135.34
128.88
127.72
126.50
113.68
55.56
55.18
43.81
35.35
34.06
20.98
20.47
CHCl3
impurity
f1 (ppm)


7.22 7.21 7.21 7.20 7.19 6.85 6.85 6.84 6.83 6.81 6.81 6.81 6.80 6.79 6.67 6.66 6.66 6.65 6.64 4.09 4.08 4.07 4.07 3.72 3.71 3.70 3.70 3.69 3.69 3.68 3.68 3.67 3.66 3.65 3.23 3.23 3.22 3.22 3.22 3.21
18, 20, 22 H
CHCl3
impurity
1, 2 H
3, 4 H
1.915
4.037
1.982
2.079
7.007
2.000
f1 (ppm)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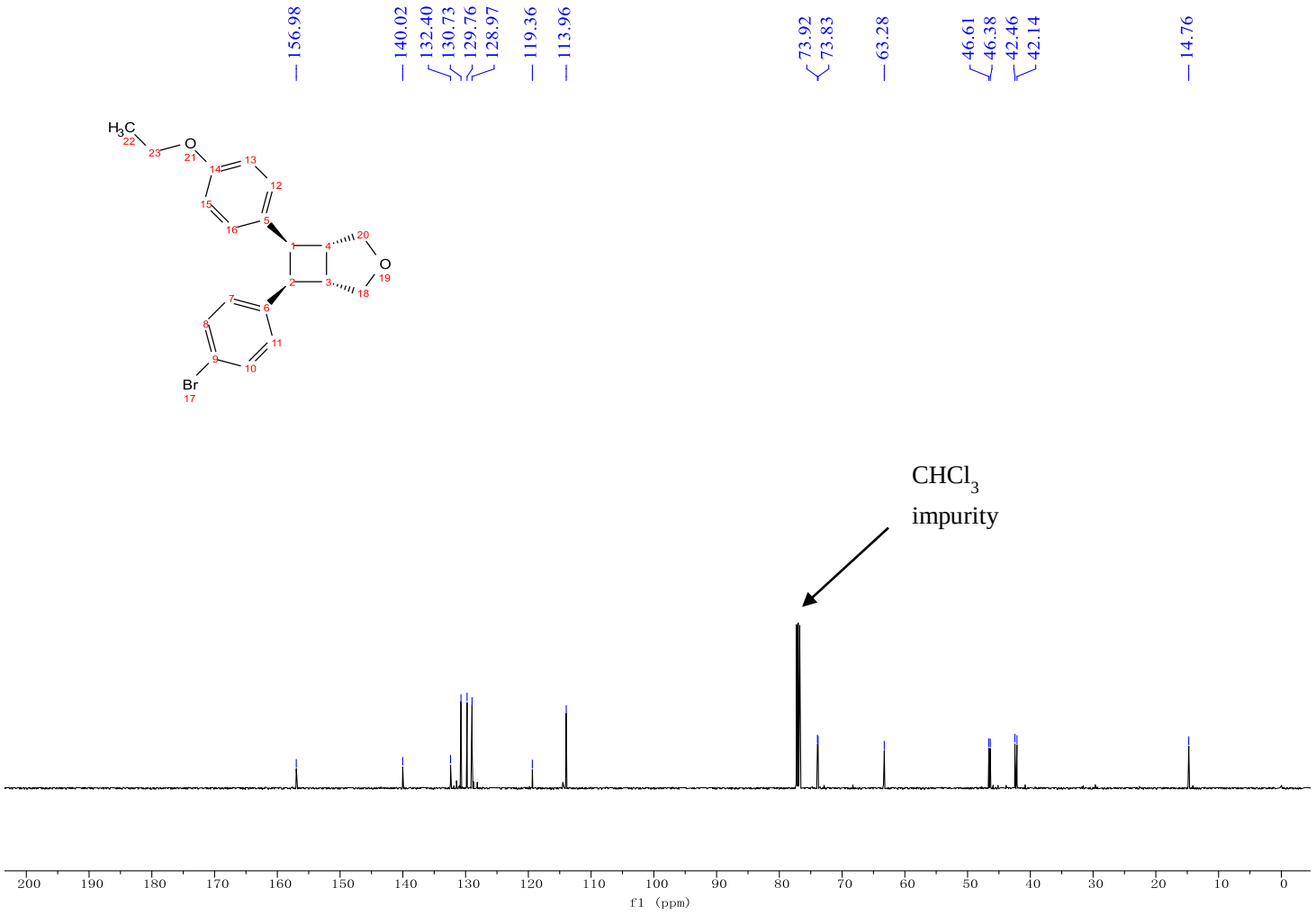
7.19
6.83
6.82
6.80
6.79
6.65
6.63
4.09
4.08
4.07
4.06
3.95
3.93
3.92
3.90
3.70
3.69
3.68
3.67
3.66
3.66
3.65
3.22
1.36
1.35
1.34
H3C
22
O
21
23
Br
17
O
19
CHCl3
impurity
18, 20, 23 H
22 H
1, 2 H
3, 4 H
1.853
4.057
1.927
1.966
2.000
4.046
2.054
3.133
f1 (ppm)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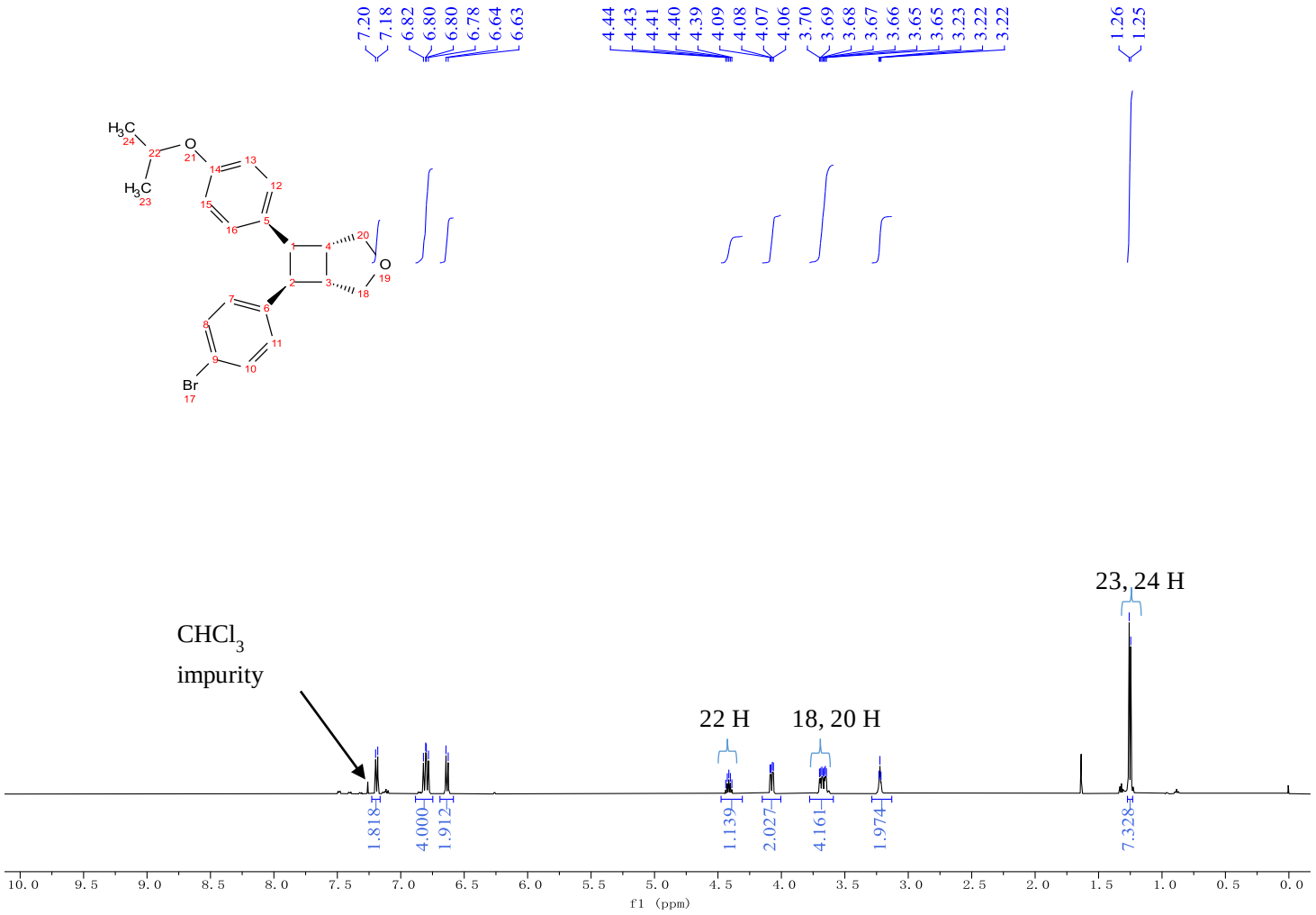
140.02
132.40
130.73
129.76
128.97
119.36
113.96
73.92
73.83
63.28
46.61
46.38
42.46
42.14
14.76
CHCl3
impurity
f1 (ppm)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7.18
6.82
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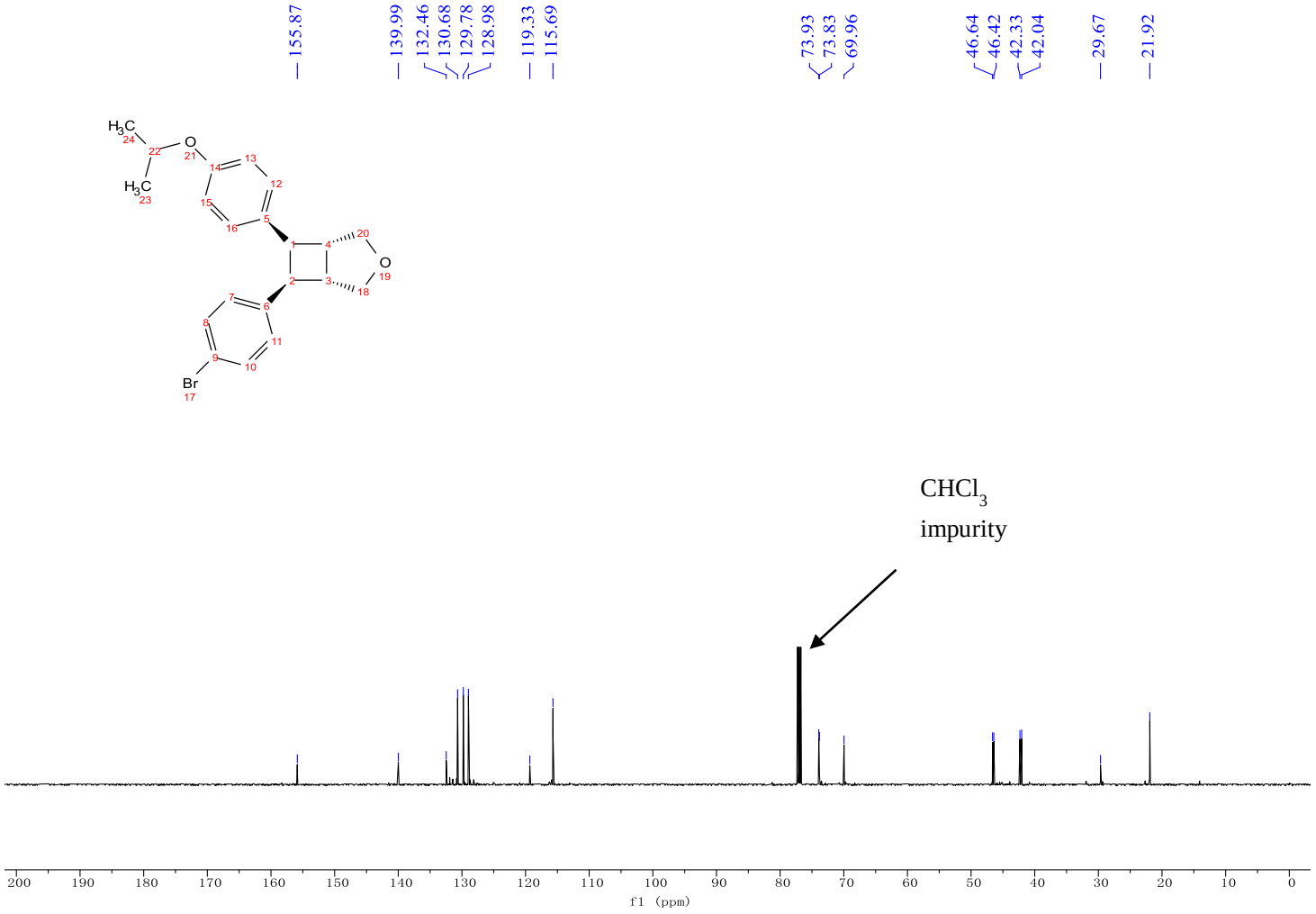
6.80
6.78
6.64
6.63
4.44
4.43
4.41
4.40
4.39
4.09
4.08
4.07
4.06
3.70
3.69
3.68
3.67
3.66
3.65
3.65
3.23
3.22
3.22
1.26
1.25
H3C
24
O
21
H3C
23
22
13
14
12
15
5
16
1
4
20
O
19
2
3
18
7
6
8
11
9
10
Br
17
CHCl3
impurity
22 H
18, 20 H
23, 24 H
1.818
4.000
1.912
1.139
2.027
4.161
1.974
7.328
10.0
9.5
9.0
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
f1 (ppm)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128.98
119.33
115.69
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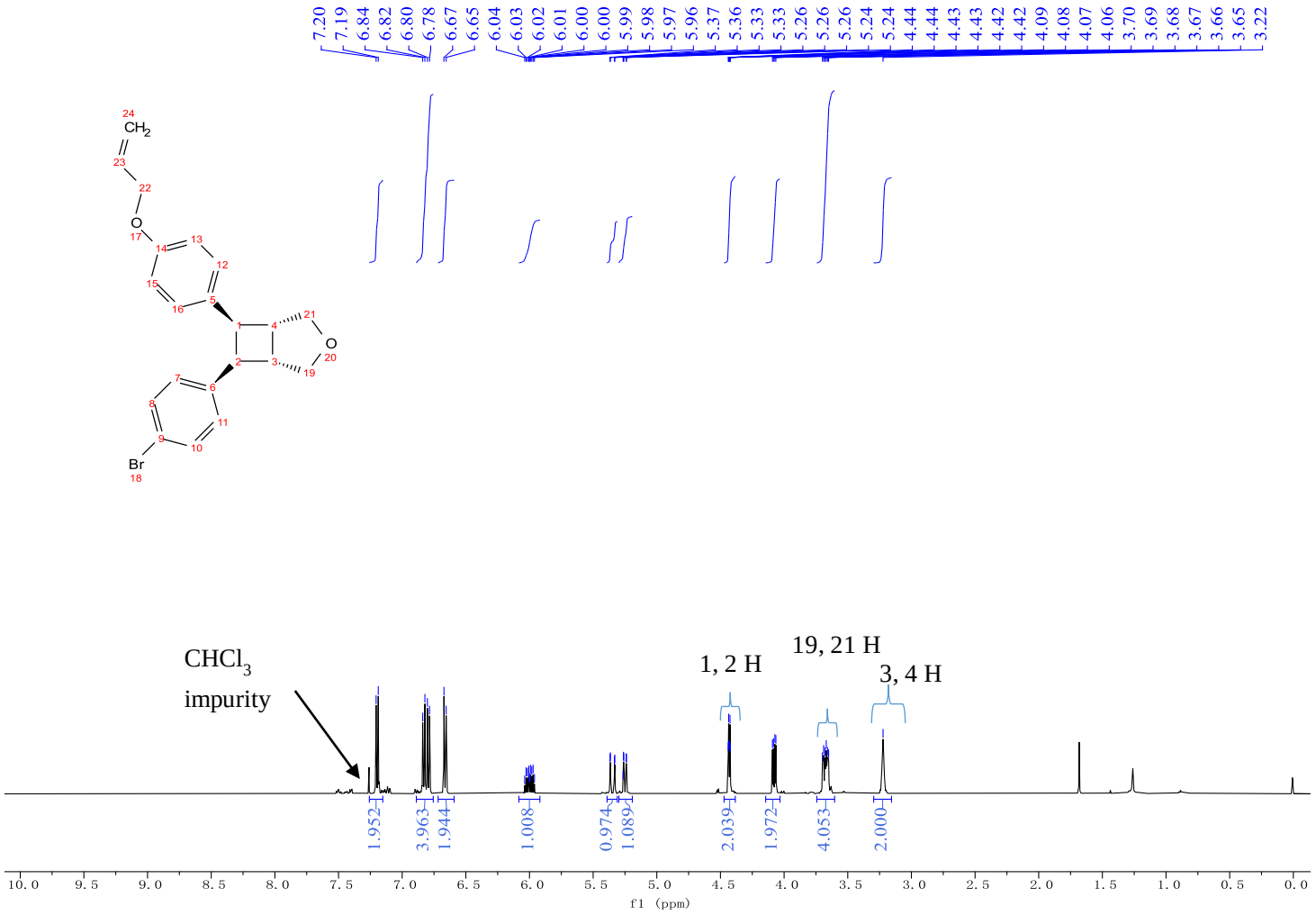
46.64
46.42
42.33
42.04
29.67
21.92
CHCl3
impurity
f1 (ppm)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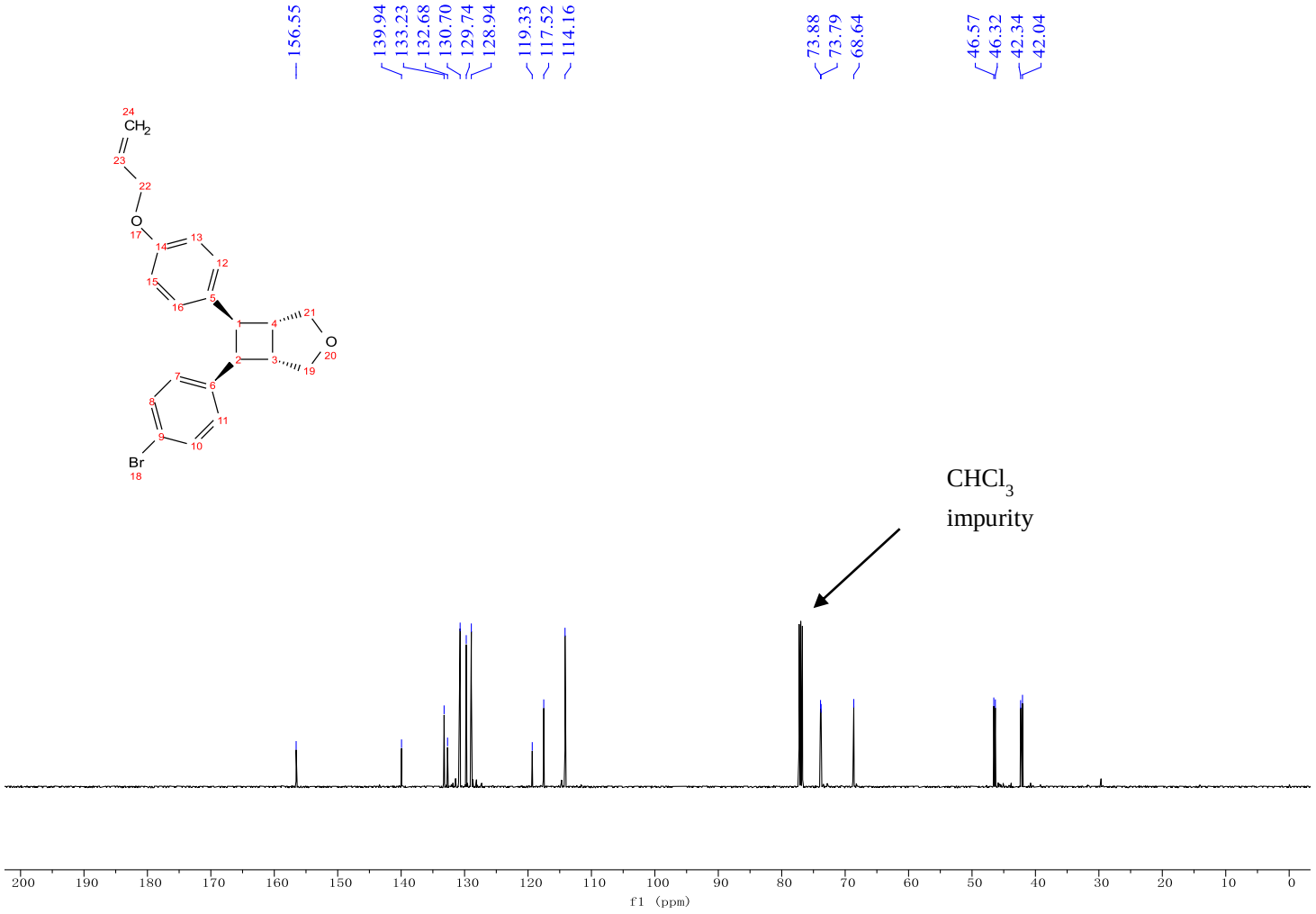
7.19
6.84
6.82
6.80
6.78
6.67
6.65
6.04
6.03
6.02
6.01
6.00
6.00
5.99
5.98
5.97
5.96
5.37
5.36
5.33
5.33
5.26
5.26
5.24
5.24
4.44
4.44
4.43
4.43
4.42
4.42
4.09
4.08
4.07
4.06
3.70
3.69
3.68
3.67
3.66
3.65
3.22
CHCl3
impurity
1, 2 H
19, 21 H
3, 4 H
1.952
3.963
1.944
1.008
0.974
1.089
2.039
1.972
4.053
2.000
f1 (ppm)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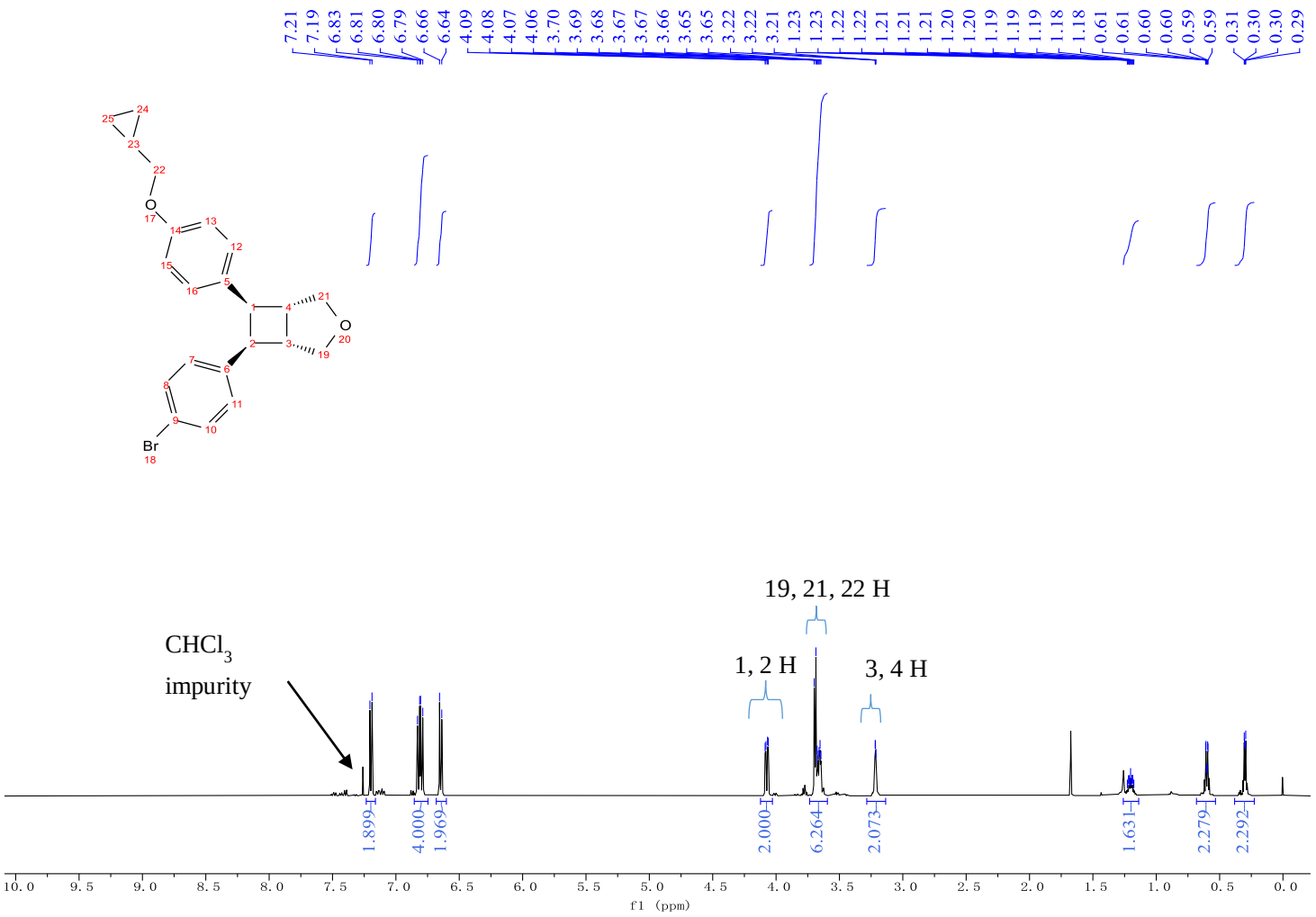
139.94
133.23
132.68
130.70
129.74
128.94
119.33
117.52
114.16
73.88
73.79
68.64
46.57
46.32
42.34
42.04
CHCl3
impurity
f1 (ppm)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Br
CHCl3
impurity
19, 21, 22 H
1, 2 H
3, 4 H
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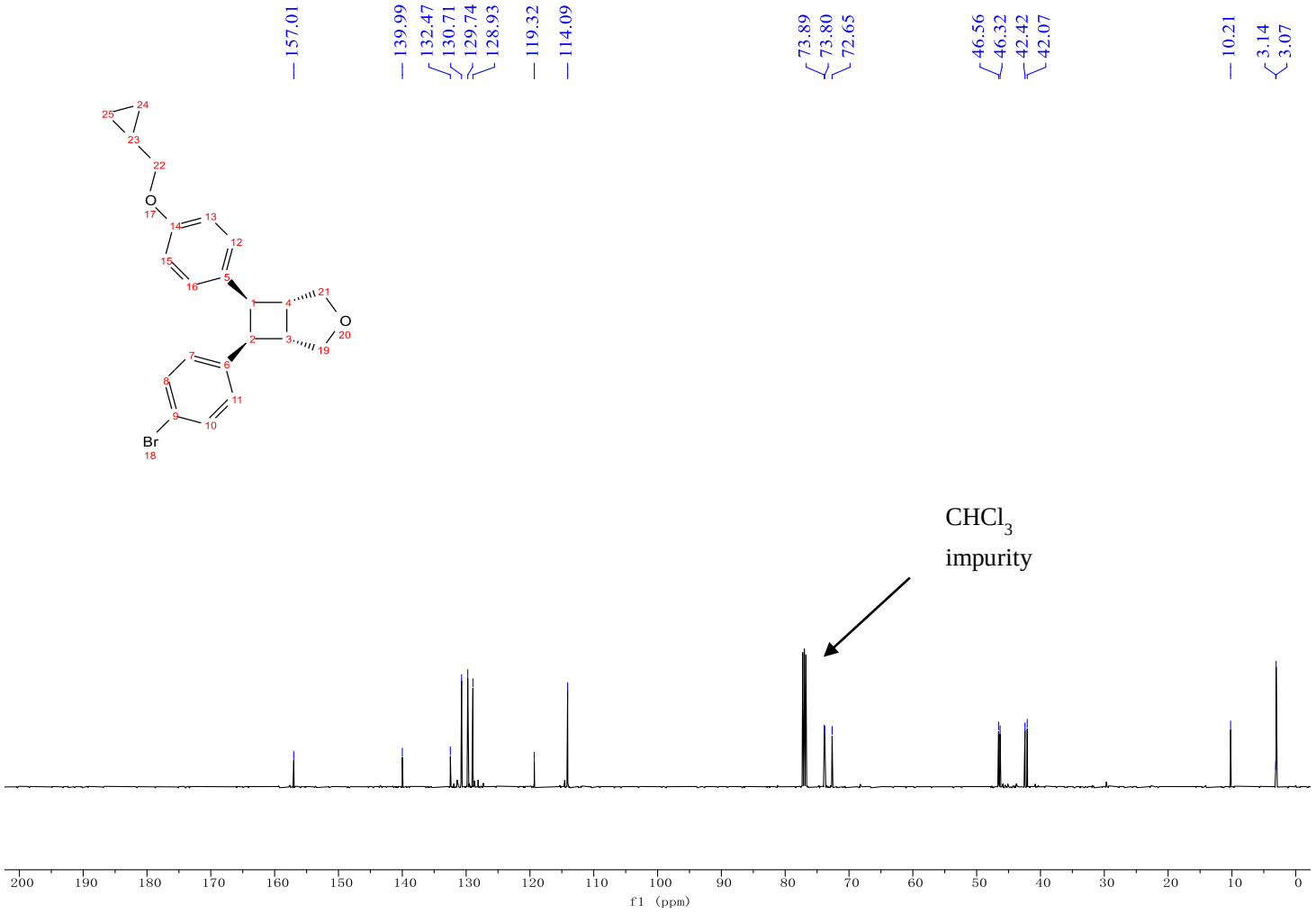
4.000
1.969
2.000
6.264
2.073
1.631
2.279
2.292
f1 (ppm)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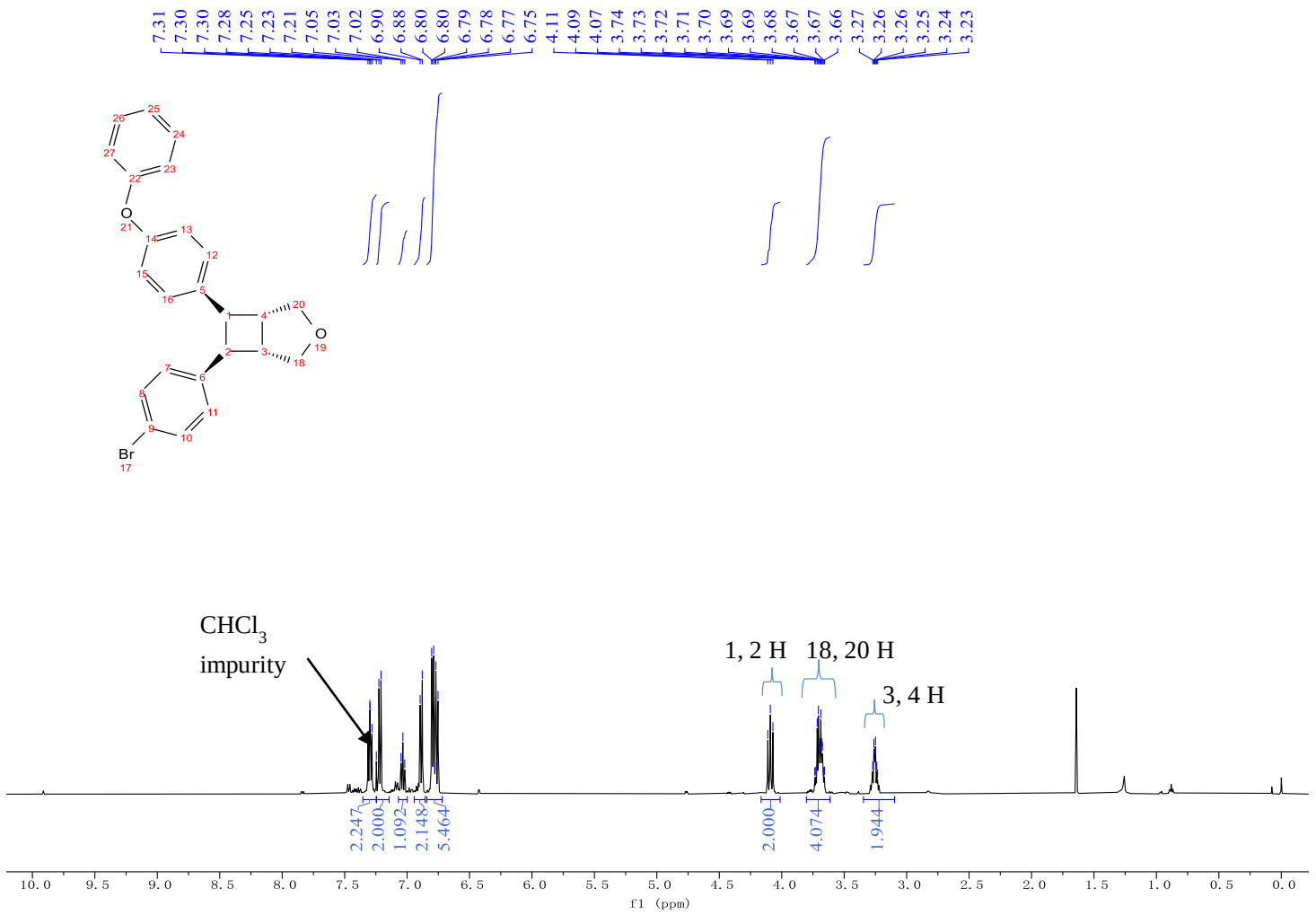
130.71
129.74
128.93
119.32
114.09
73.89
73.80
72.65
46.56
46.32
42.42
42.07
10.21
3.14
3.07
CHCl3
impurity
f1 (ppm)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7.30
7.30
7.28
7.25
7.23
7.21
7.05
7.03
7.02
6.90
6.88
6.80
6.80
6.79
6.78
6.77
6.75
4.11
4.09
4.07
3.74
3.73
3.72
3.71
3.70
3.69
3.69
3.68
3.67
3.67
3.66
3.27
3.26
3.26
3.25
3.24
3.23
Br
O
O
CHCl3
impurity
1, 2 H
18, 20 H
3, 4 H
2.247
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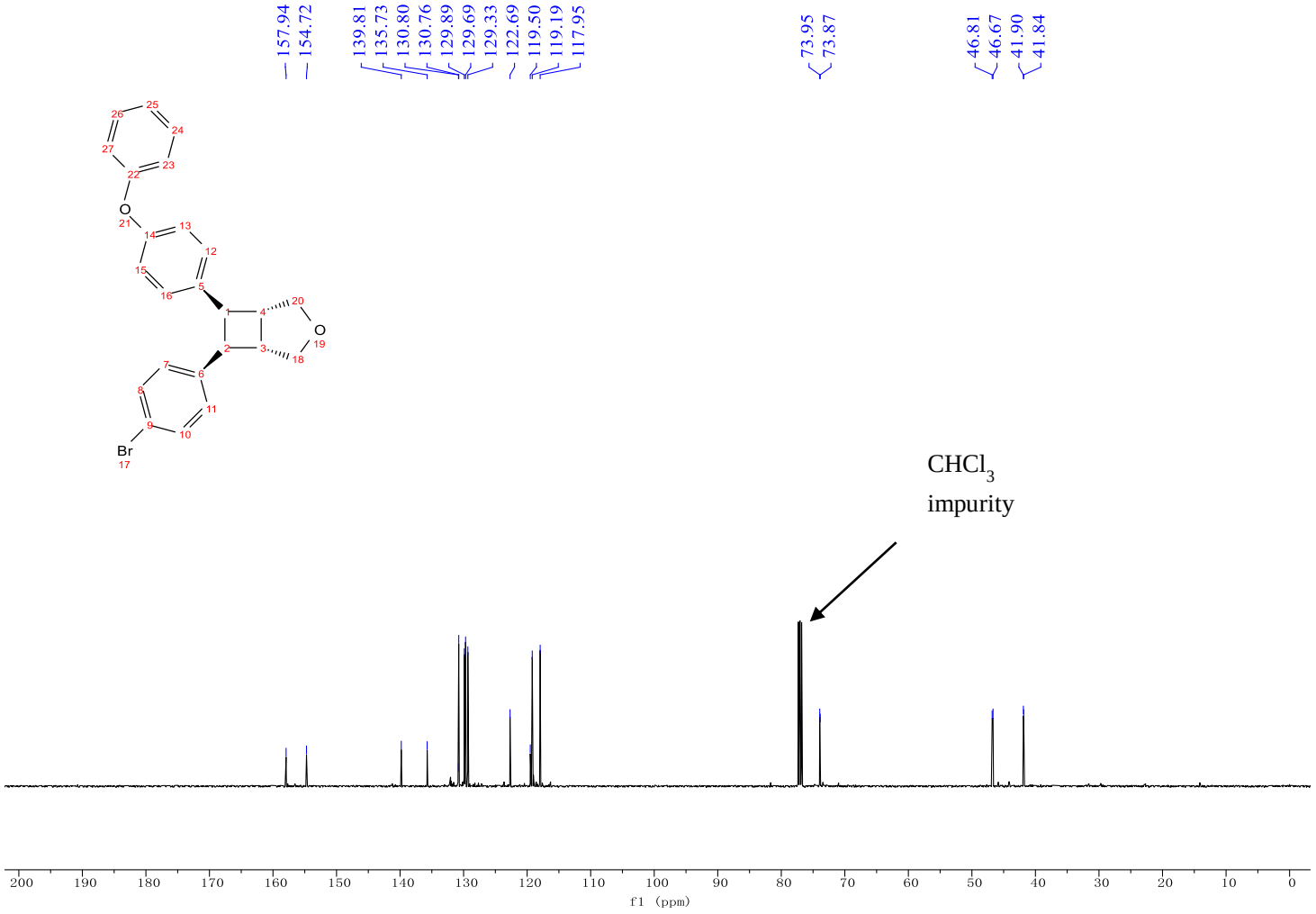
1.092
2.148
5.464
2.000
4.074
1.944
f1 (ppm)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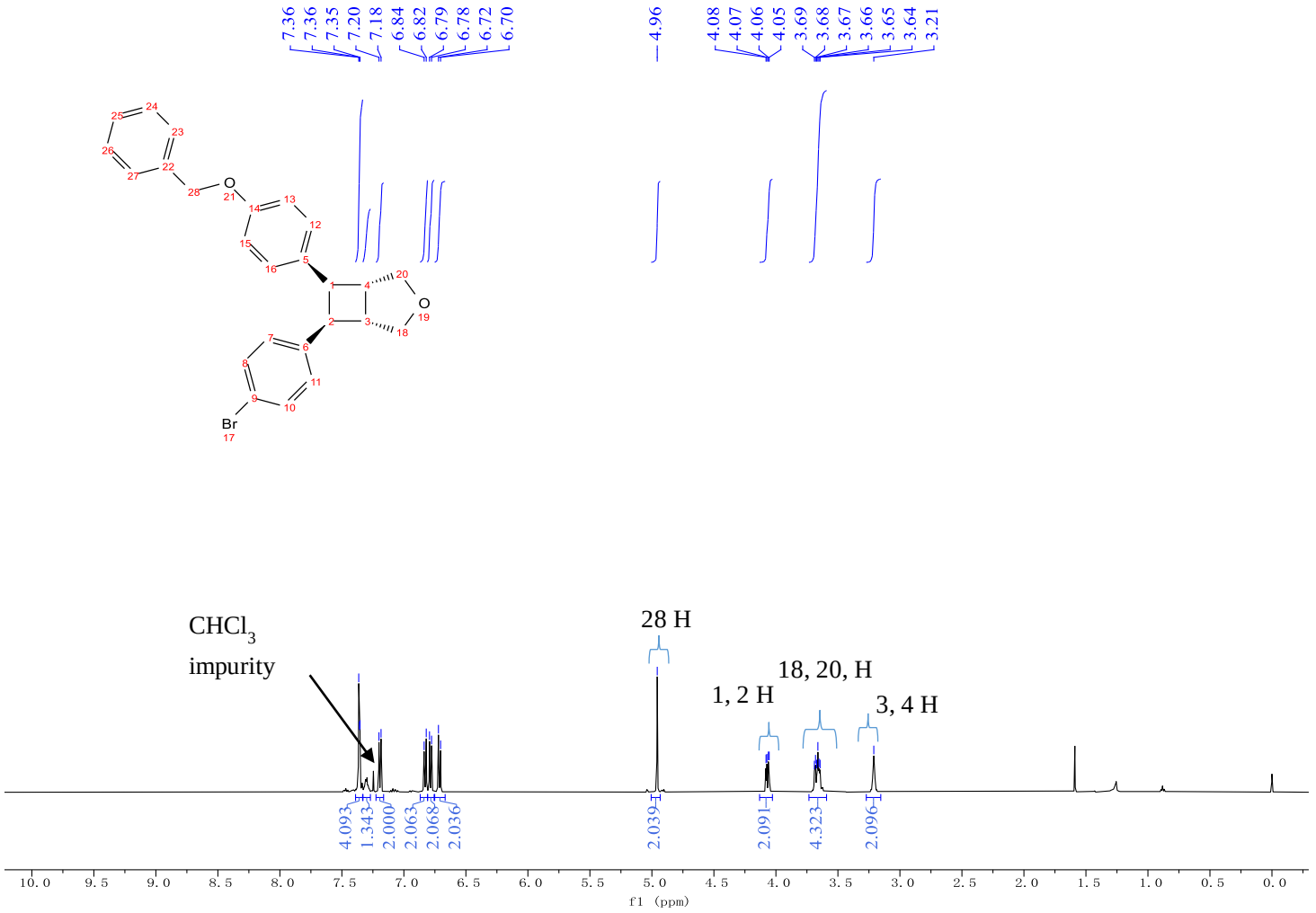
154.72
139.81
135.73
130.80
130.76
129.89
129.69
129.33
122.69
119.50
119.19
117.95
73.95
73.87
46.81
46.67
41.90
41.84
CHCl3
impurity
f1 (ppm)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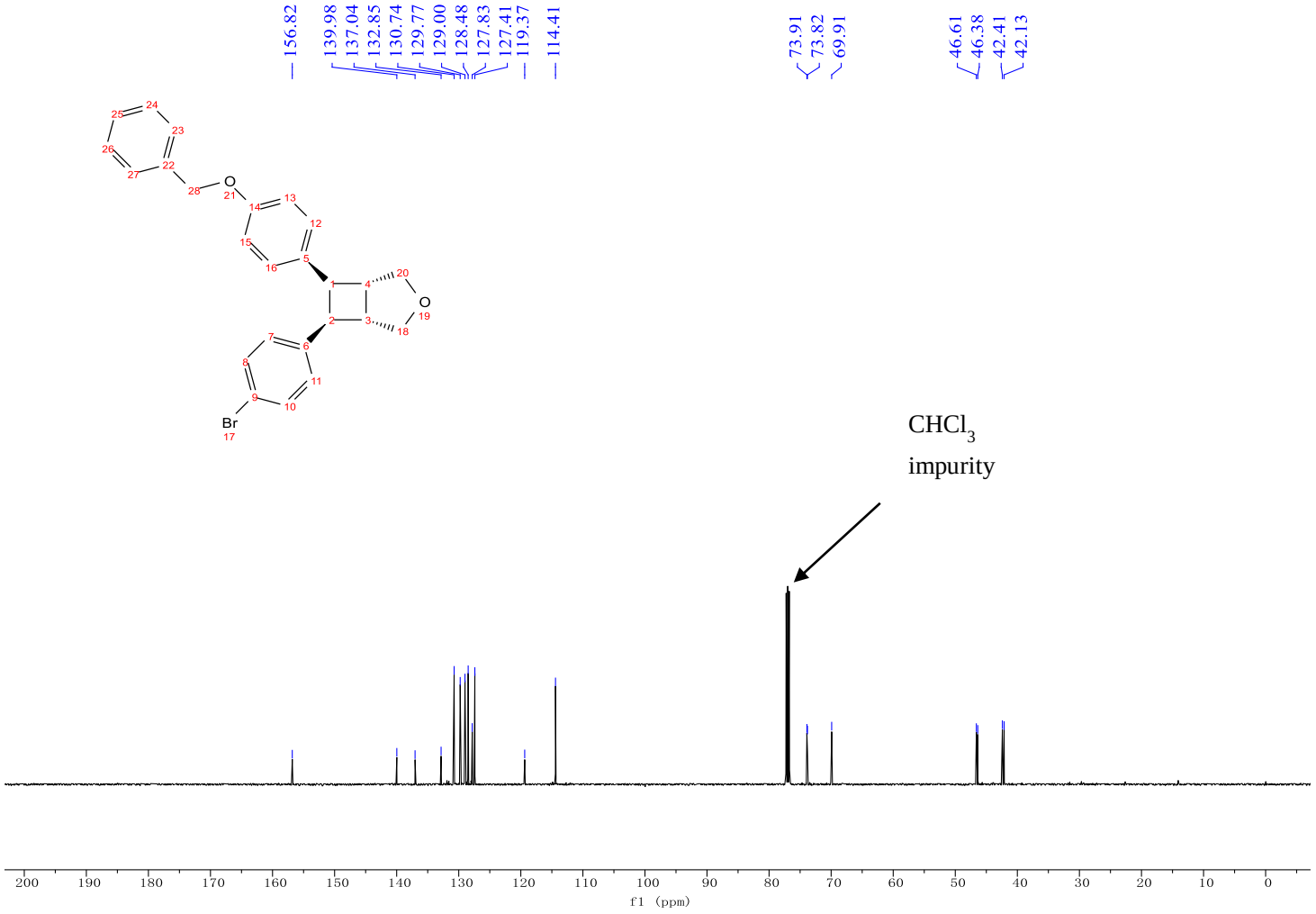
6.84
6.82
6.79
6.78
6.72
6.70
4.96
4.08
4.07
4.06
4.05
3.69
3.68
3.67
3.66
3.65
3.64
3.21
Br
17
O
21
O
19
CHCl3
impurity
28 H
18, 20, H
1, 2 H
3, 4 H
4.093
1.343
2.000
2.063
2.068
2.036
2.039
2.091
4.323
2.096
f1 (ppm)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127.83
127.41
119.37
114.41
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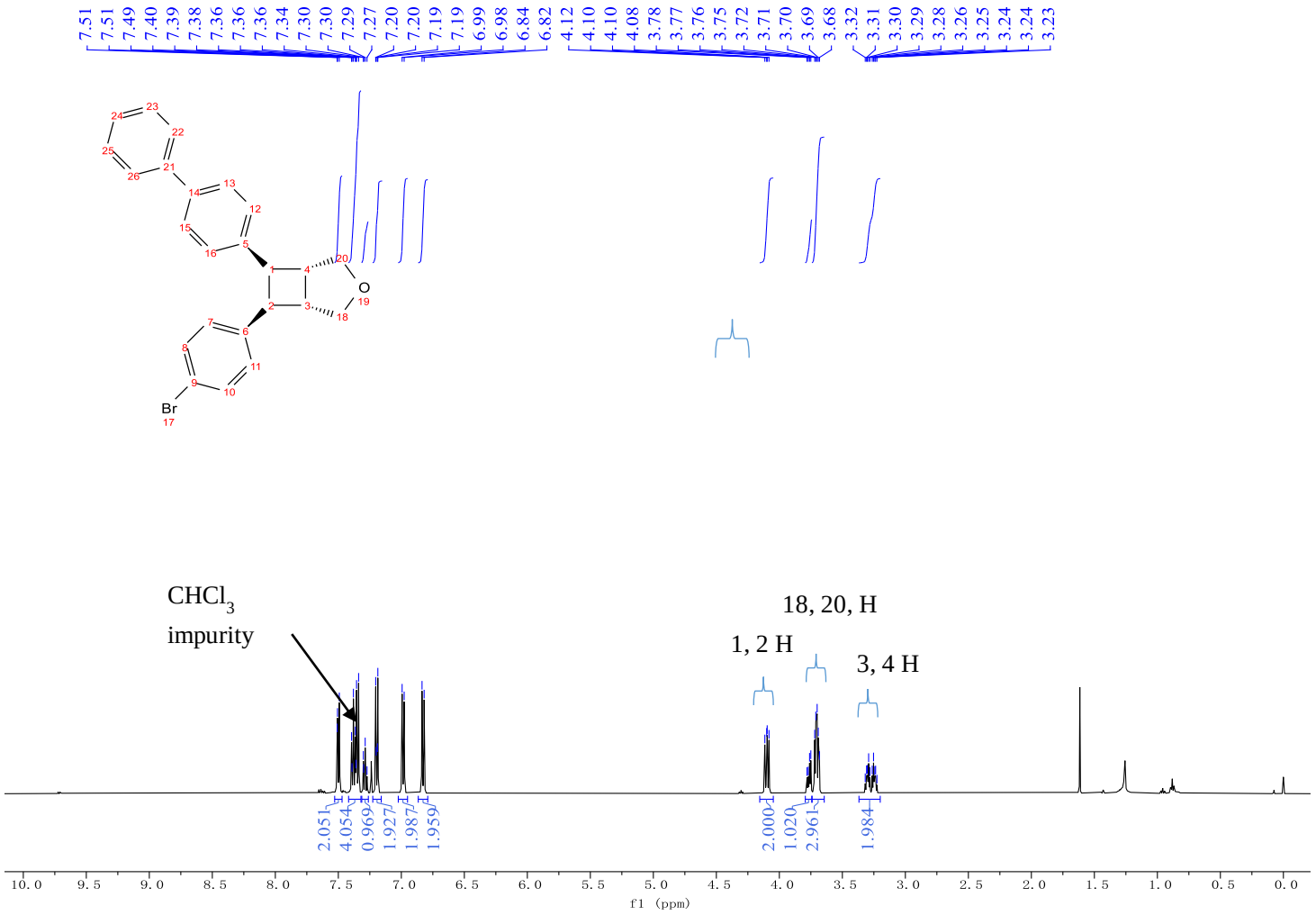
73.82
69.91
46.61
46.38
42.41
42.13
Br
O
CHCl3
impurity
f1 (ppm)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7.51
7.49
7.40
7.39
7.38
7.36
7.36
7.36
7.34
7.30
7.30
7.29
7.27
7.20
7.20
7.19
7.19
6.99
6.98
6.84
6.82
4.12
4.10
4.10
4.08
3.78
3.77
3.76
3.75
3.72
3.71
3.70
3.69
3.68
3.32
3.31
3.30
3.29
3.28
3.26
3.25
3.24
3.24
3.23
Br
O
CHCl3
impurity
18, 20, H
1, 2 H
3, 4 H
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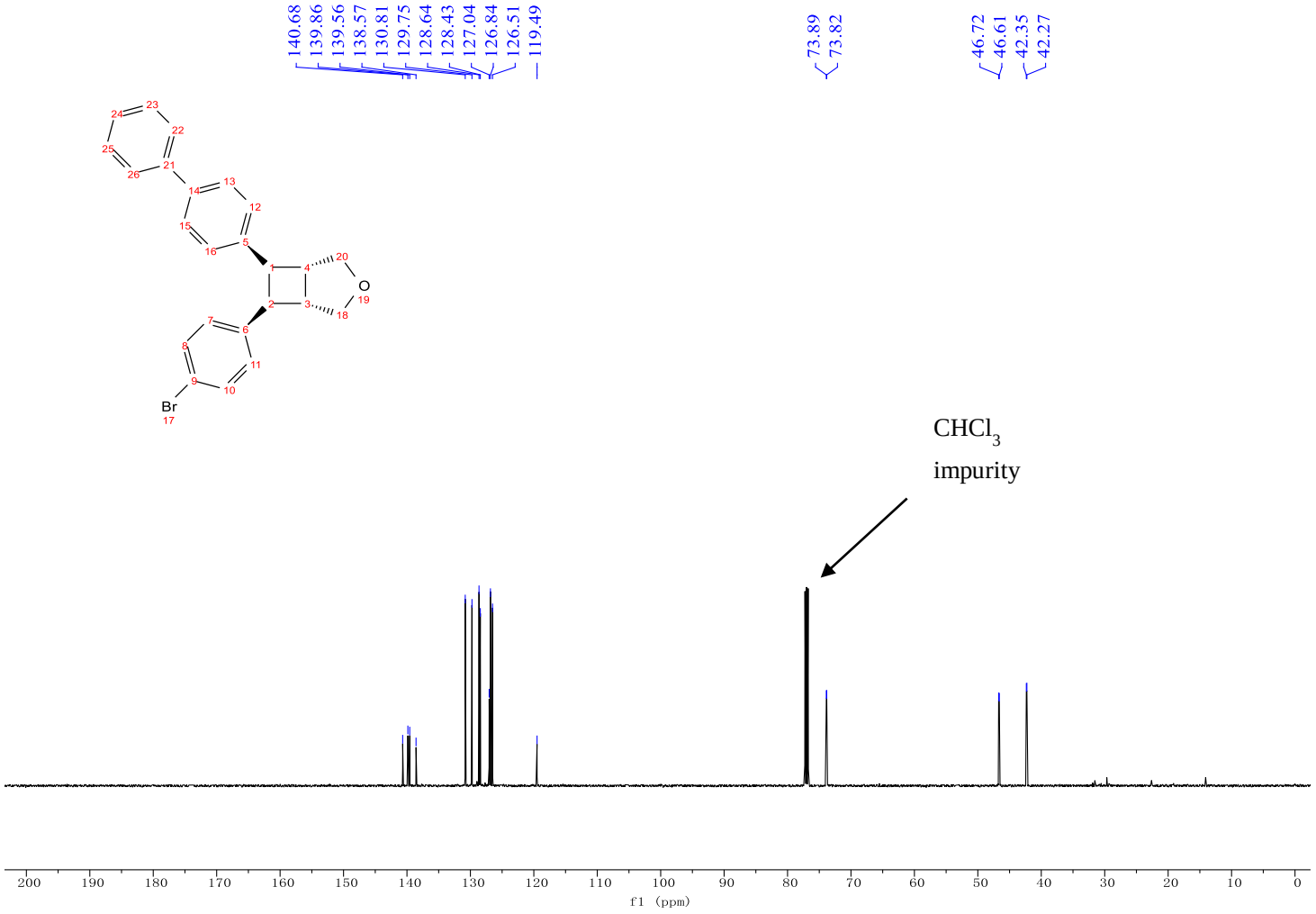
0.969
1.927
1.987
1.959
2.000
1.020
2.961
1.984
f1 (ppm)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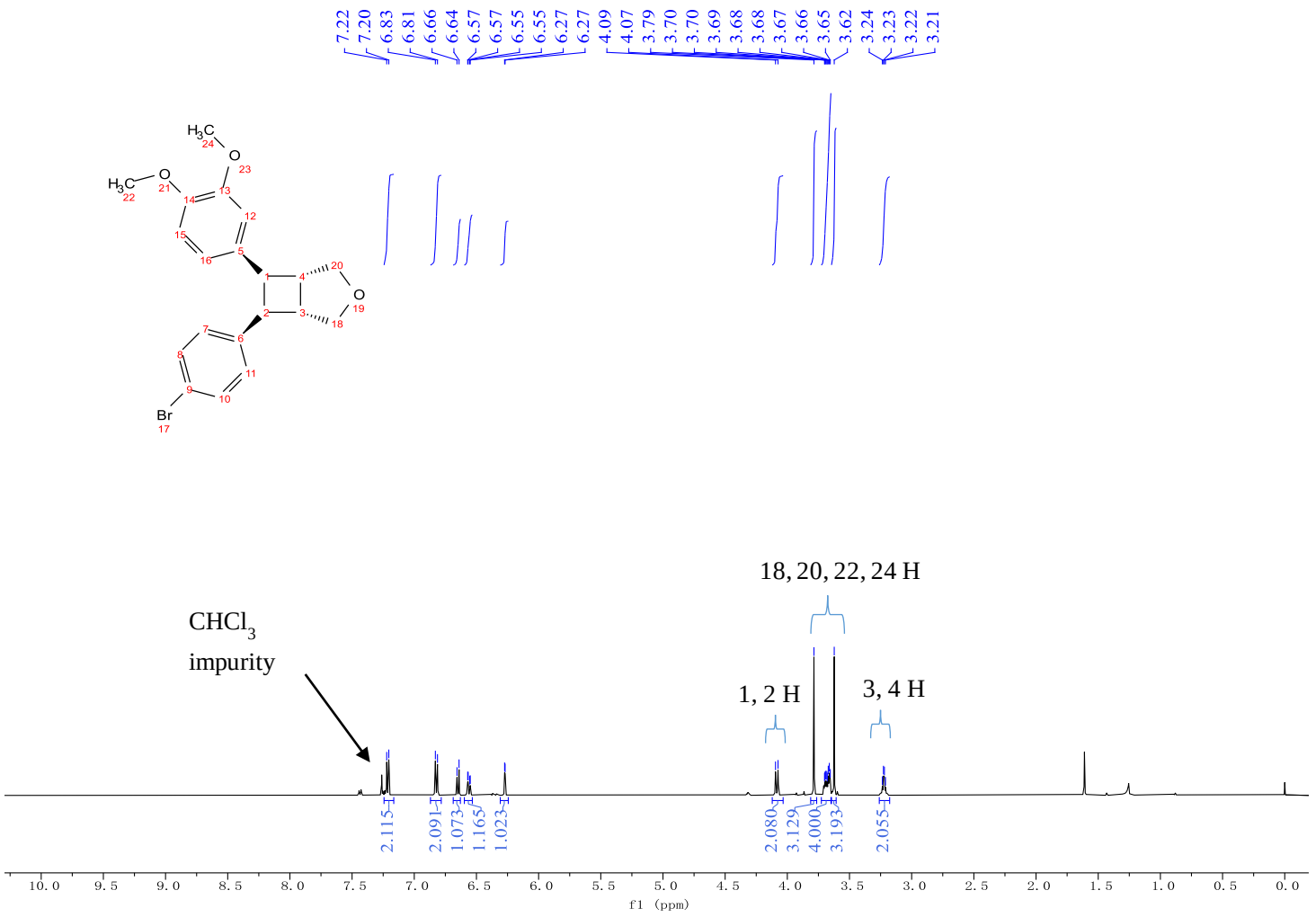
139.86
139.56
138.57
130.81
129.75
128.64
128.43
127.04
126.84
126.51
119.49
73.89
73.82
46.72
46.61
42.35
42.27
Br
O
CHCl3
impurity
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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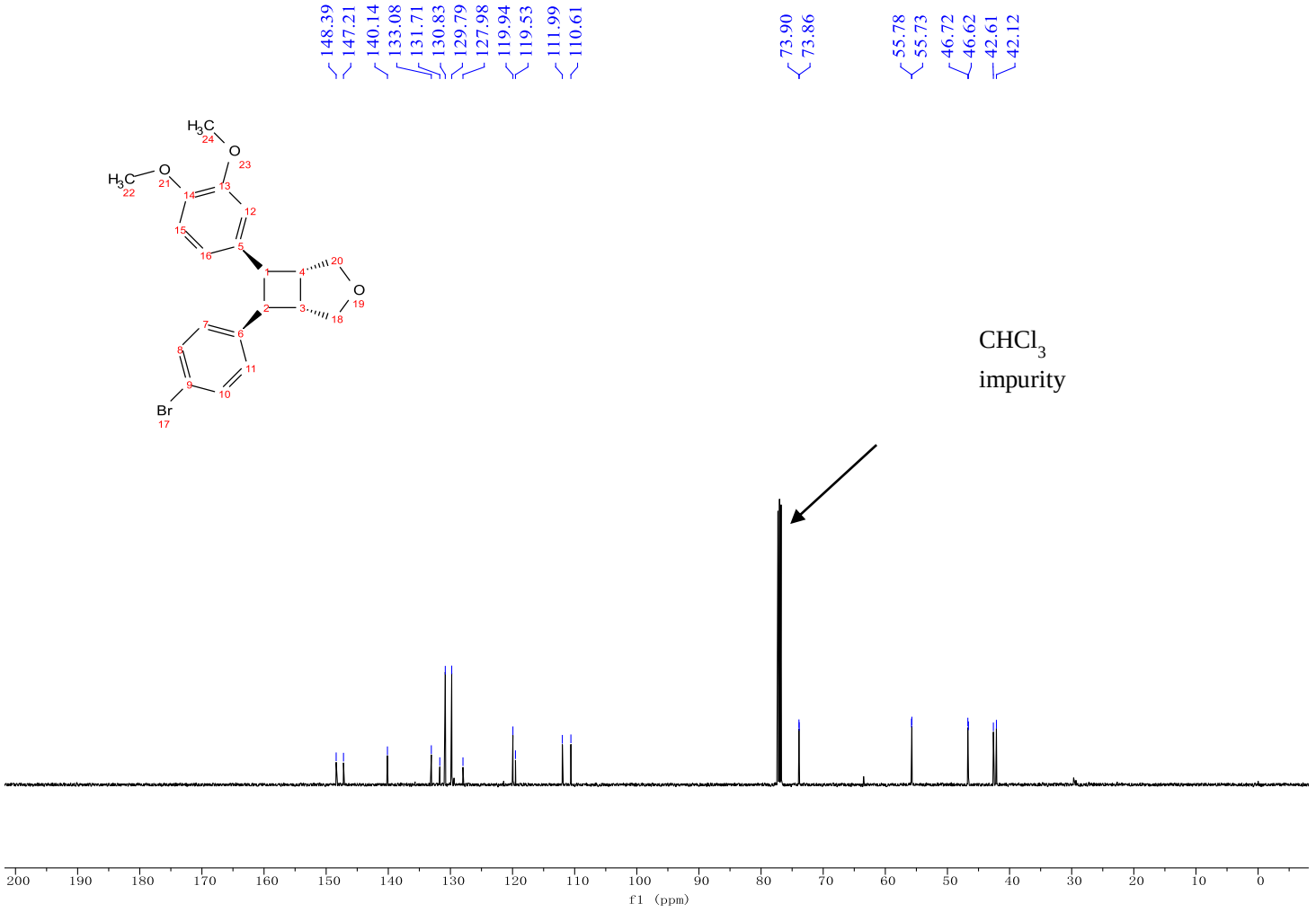
6.64
6.57
6.57
6.55
6.55
6.27
6.27
4.09
4.07
3.79
3.70
3.70
3.69
3.68
3.68
3.67
3.66
3.65
3.62
3.24
3.23
3.22
3.21
H3C
24
O
23
H3C
22
O
21
14
13
12
15
5
16
1
4
20
O
19
2
3
18
7
6
8
11
9
10
Br
17
18, 20, 22, 24 H
CHCl3
impurity
1, 2 H
3, 4 H
2.115
2.091
1.073
1.165
1.023
2.080
3.129
4.000
3.193
2.055
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
f1 (ppm)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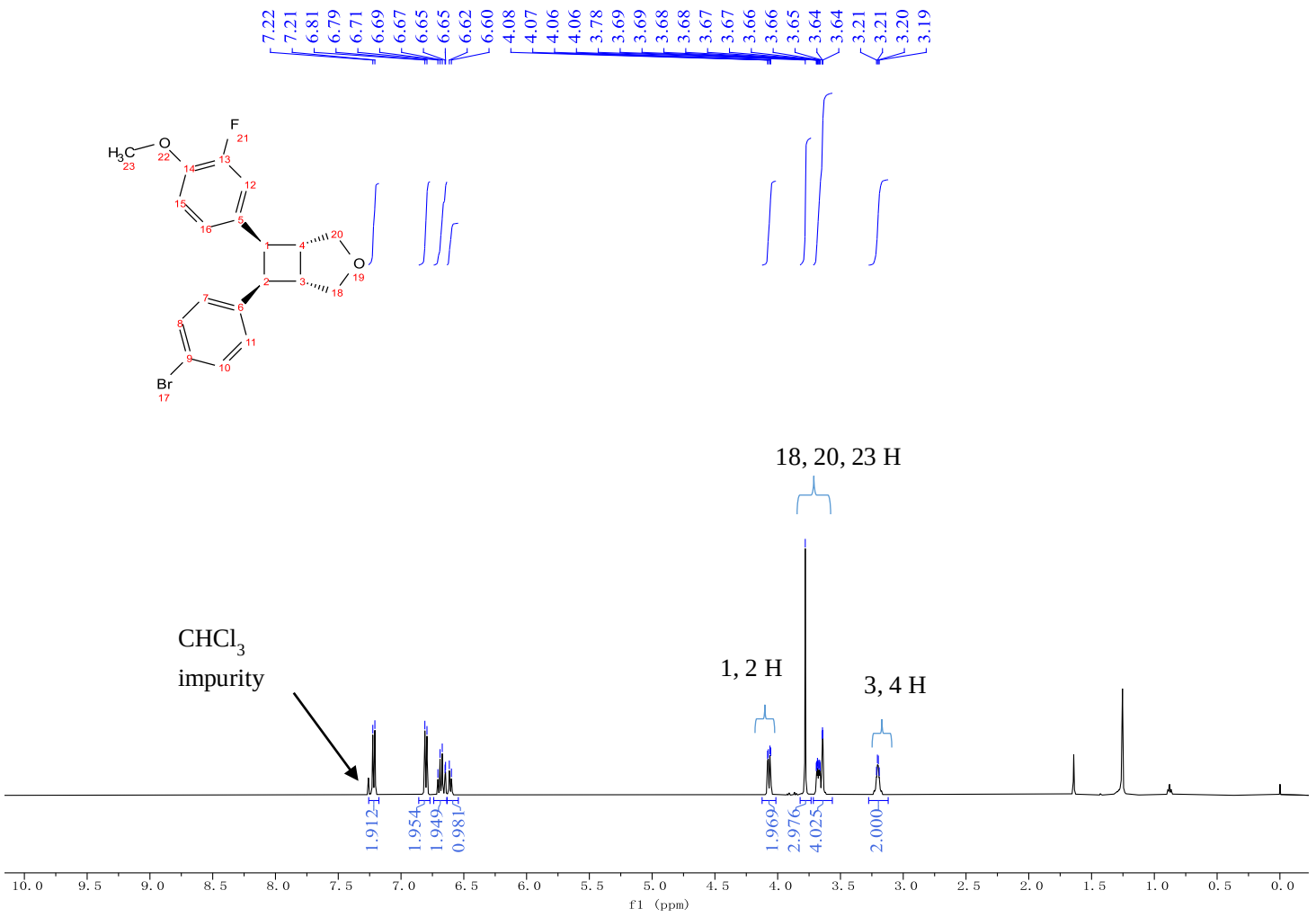
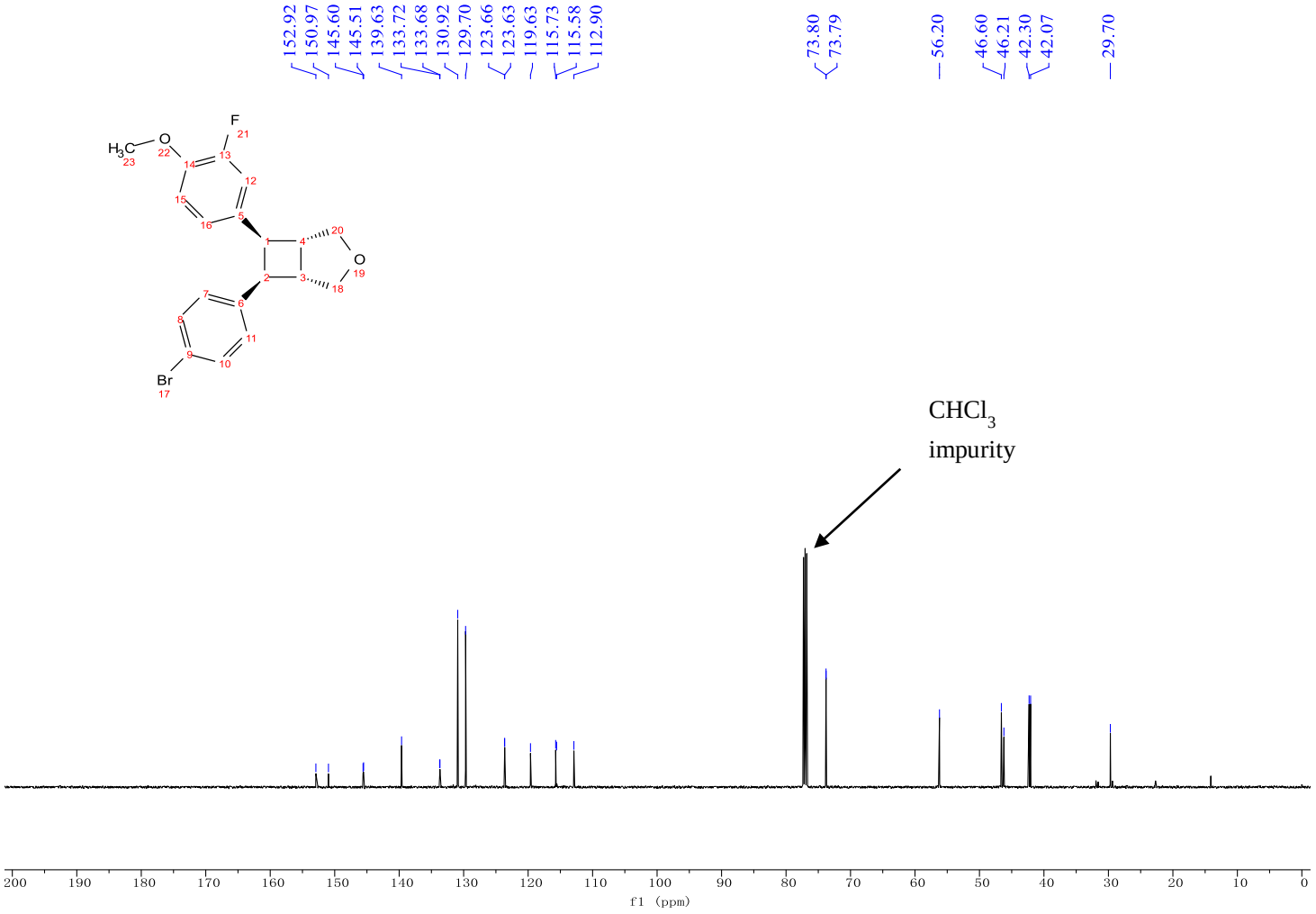
130.83
129.79
127.98
119.94
119.53
111.99
110.61
73.90
73.86
55.78
55.73
46.72
46.62
42.61
42.12
CHCl3
impurity
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.22
7.21
6.81
6.79
6.71
6.69
6.67
6.65
6.65
6.62
6.60
4.08
4.07
4.06
4.06
3.78
3.69
3.69
3.68
3.68
3.67
3.67
3.66
3.66
3.65
3.64
3.64
3.21
3.21
3.20
3.19
F
21
H3C
23
O
22
O
19
Br
17
18, 20, 23 H
CHCl3
impurity
1, 2 H
3, 4 H
1.912
1.954
1.949
0.981
1.969
2.976
4.025
2.000
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
f1 (ppm)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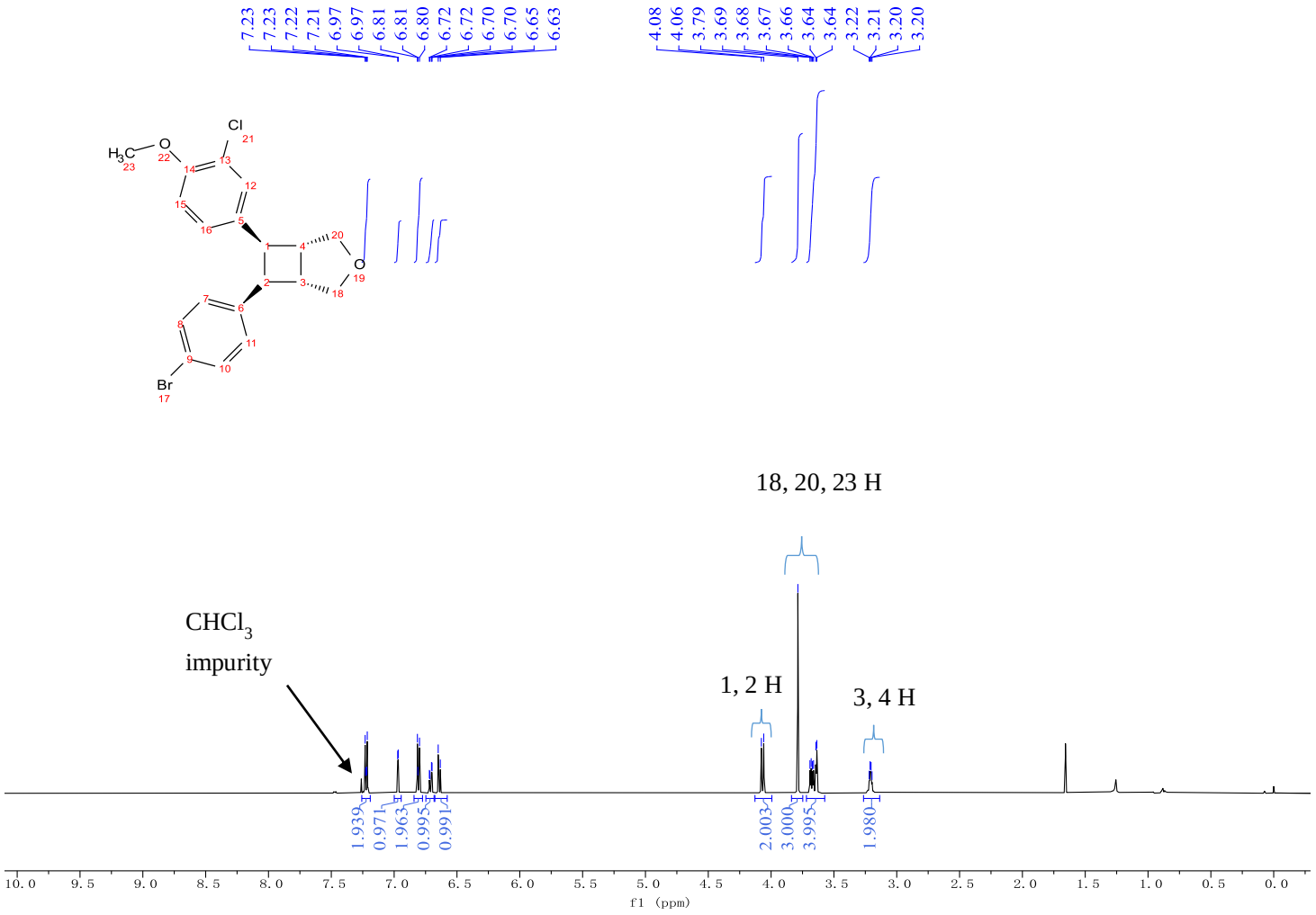
133.68
130.92
129.70
123.66
123.63
119.63
115.73
115.58
112.90
73.80
73.79
56.20
46.60
46.21
42.30
42.07
29.70
F
21
H3C
23
O
22
O
19
Br
17
CHCl3
impurity
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.23
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7.21
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6.97
6.81
6.81
6.80
6.72
6.72
6.70
6.70
6.65
6.63
4.08
4.06
3.79
3.69
3.68
3.67
3.66
3.64
3.64
3.22
3.21
3.20
3.20
Cl
21
H3C
23
O
22
Br
17
O
19
18, 20, 23 H
CHCl3
impurity
1, 2 H
3, 4 H
1.939
0.971
1.963
0.995
0.991
2.003
3.000
3.995
1.980
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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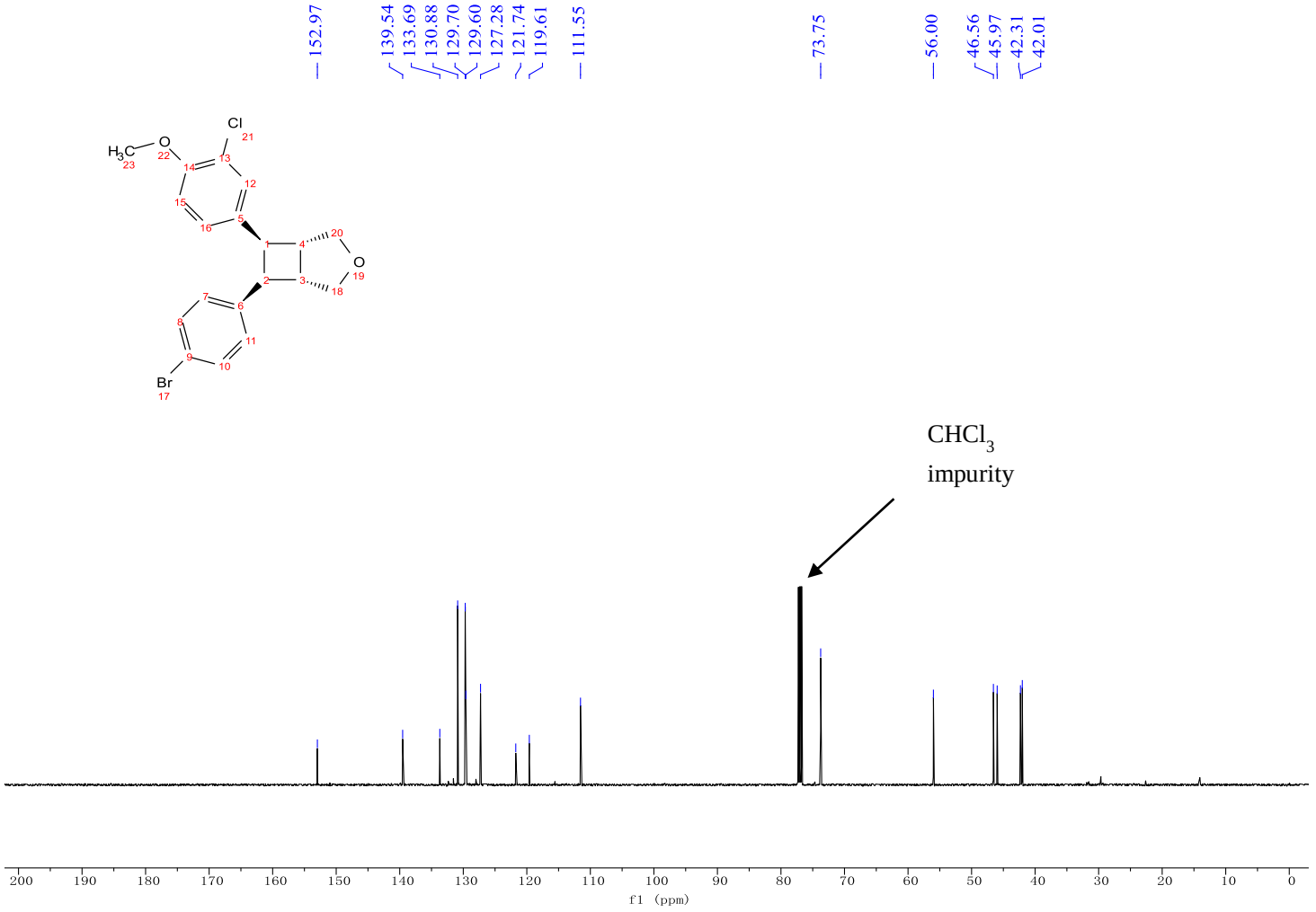
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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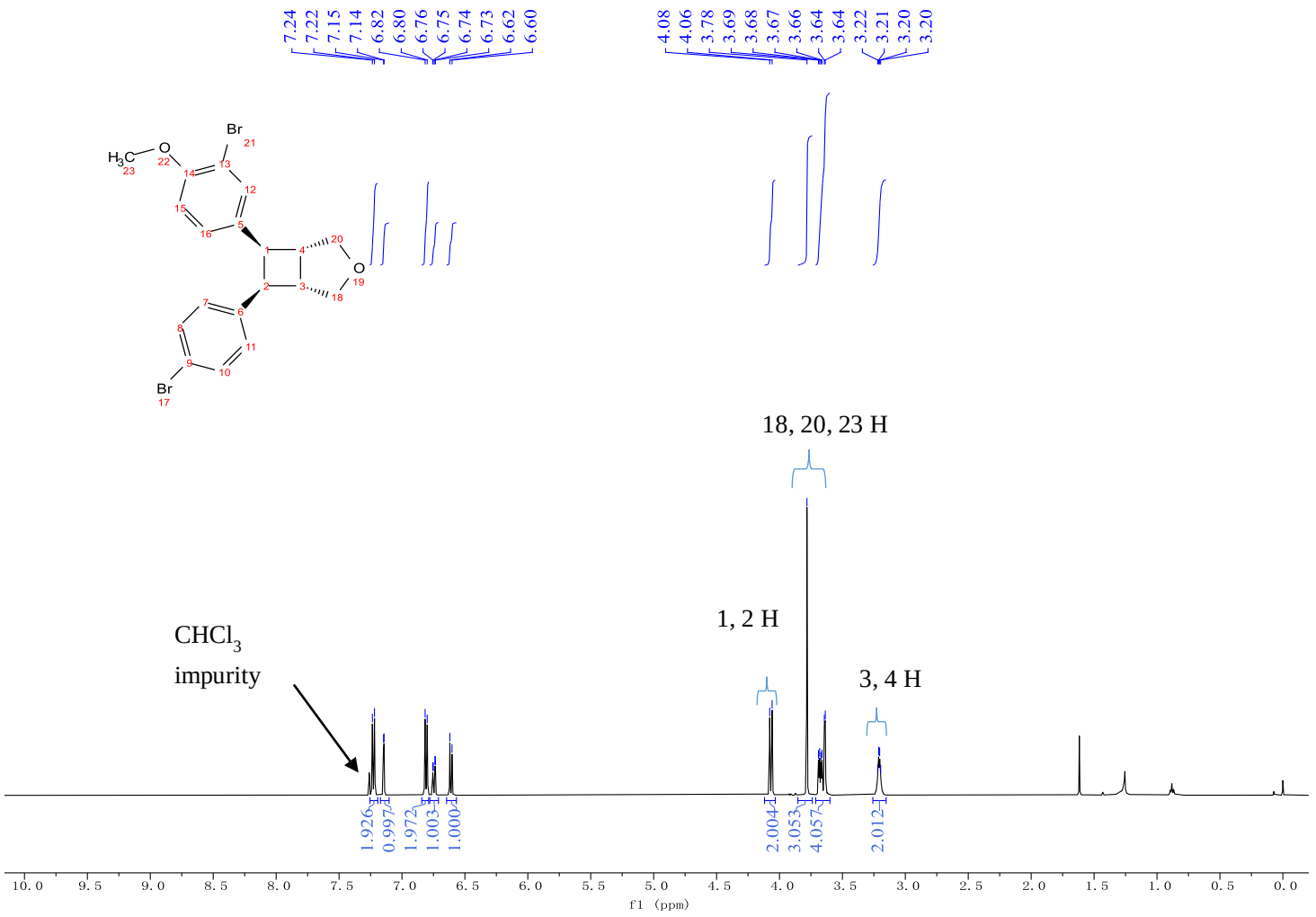
111.55
73.75
56.00
46.56
45.97
42.31
42.01
Cl
21
O
22
H3C
23
O
19
Br
17
CHCl3
impurity
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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6.82
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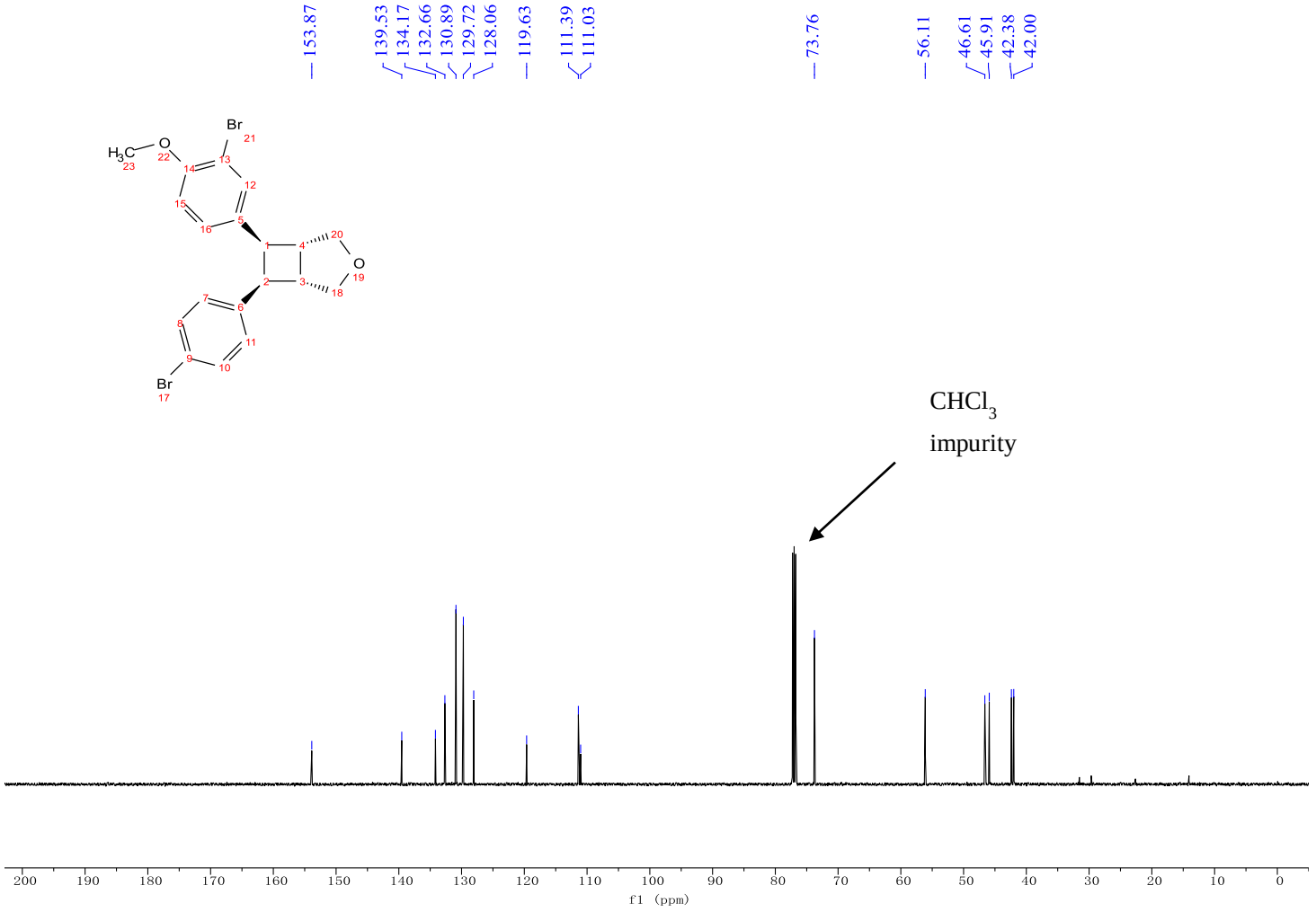
6.76
6.75
6.74
6.73
6.62
6.60
4.08
4.06
3.78
3.69
3.68
3.67
3.66
3.64
3.64
3.22
3.21
3.20
3.20
Br
21
H3C
23
O
22
14
13
12
15
5
16
1
4
20
O
19
2
3
18
7
6
8
11
9
10
Br
17
18, 20, 23 H
1, 2 H
3, 4 H
CHCl3
impurity
1.926
0.997
1.972
1.003
1.000
2.004
3.053
4.057
2.012
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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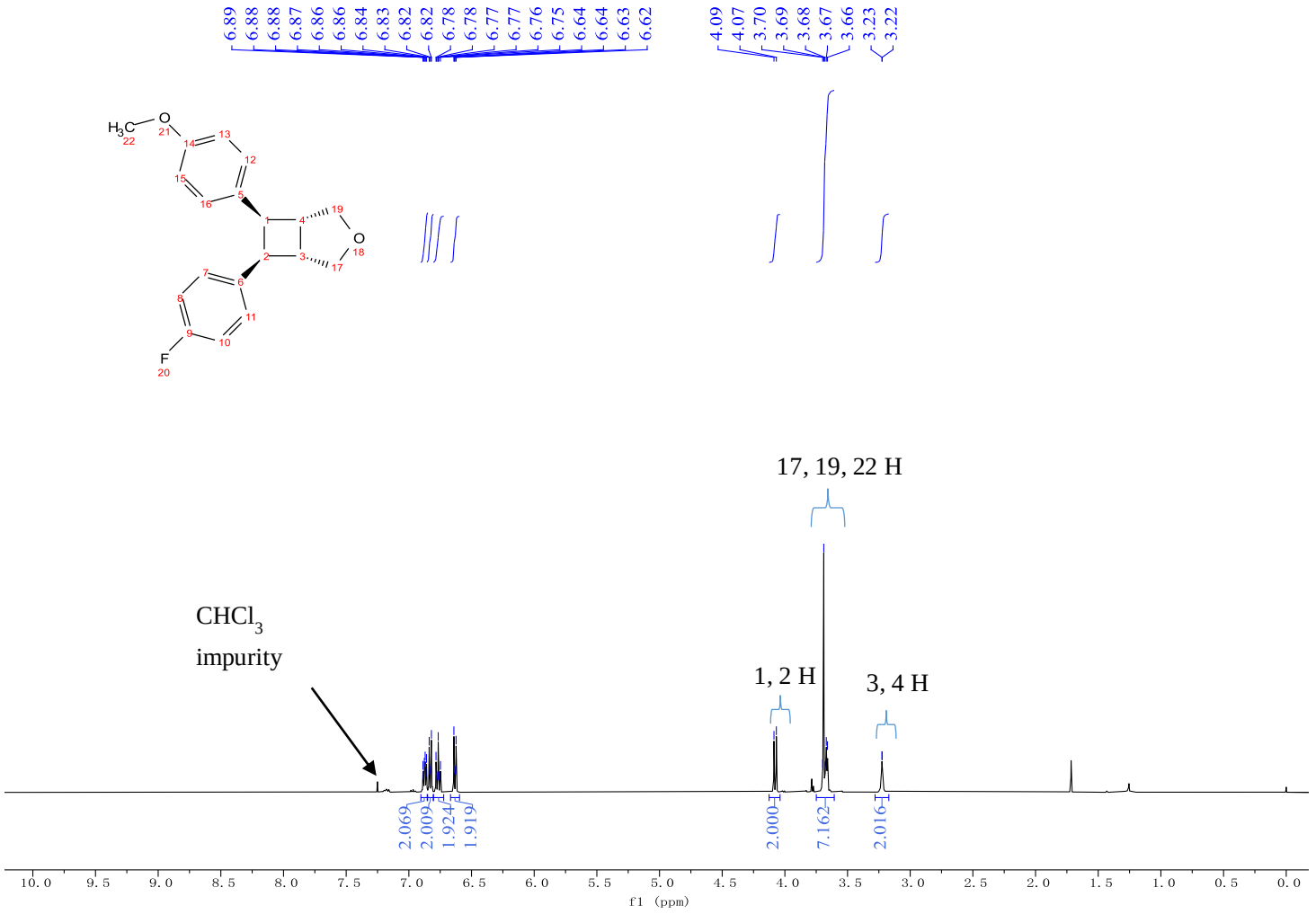
134.17
132.66
130.89
129.72
128.06
119.63
111.39
111.03
73.76
56.11
46.61
45.91
42.38
42.00
Br
21
H3C
23
O
22
14
13
12
15
5
16
1
4
20
O
19
2
3
18
7
6
8
11
9
10
Br
17
CHCl3
impurity
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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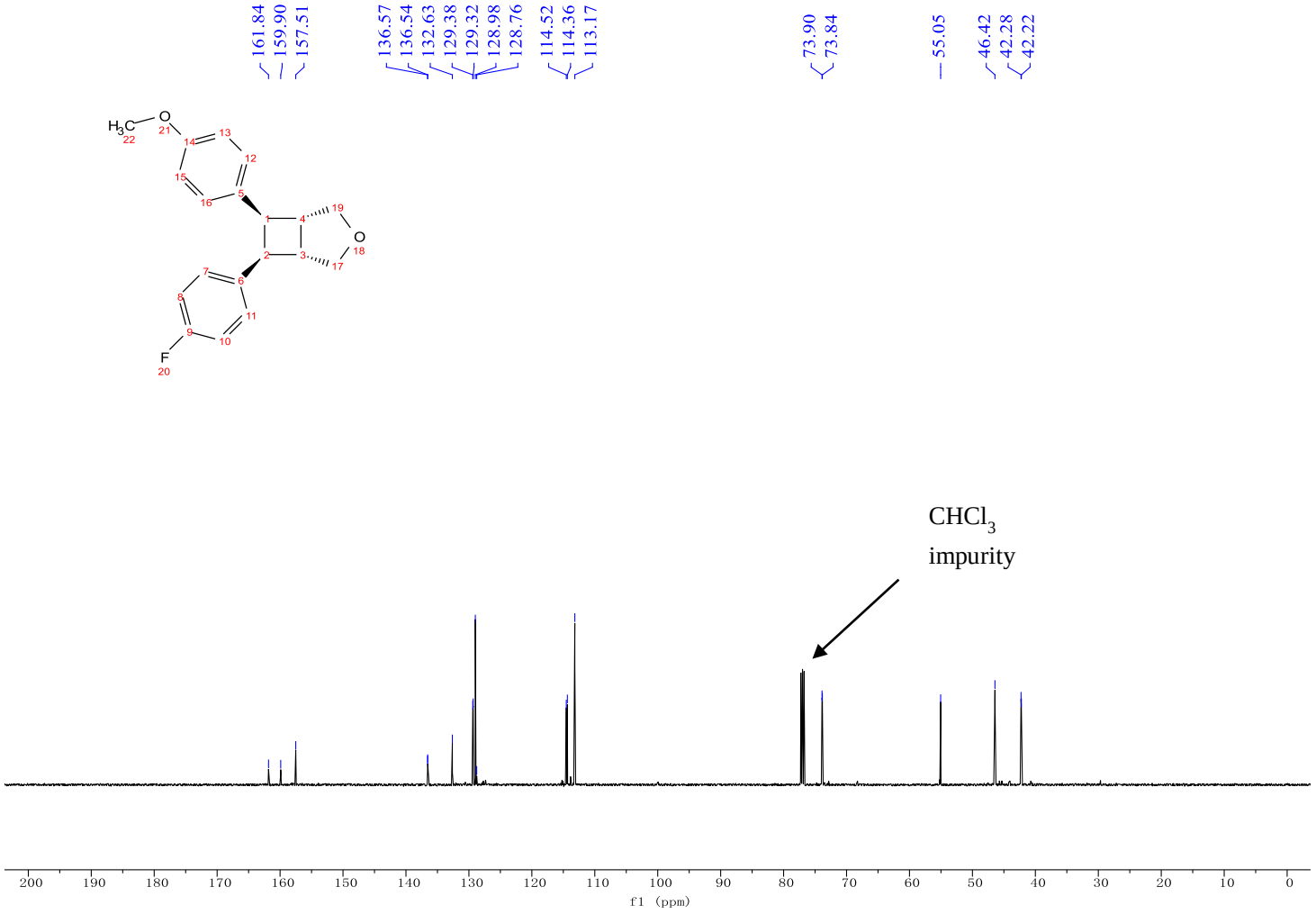
6.88
6.88
6.87
6.86
6.86
6.84
6.83
6.82
6.82
6.78
6.78
6.77
6.77
6.76
6.75
6.64
6.64
6.63
6.62
4.09
4.07
3.70
3.69
3.68
3.67
3.66
3.23
3.22
17, 19, 22 H
CHCl3
impurity
1, 2 H
3, 4 H
2.069
2.009
1.924
1.919
2.000
7.162
2.016
f1 (ppm)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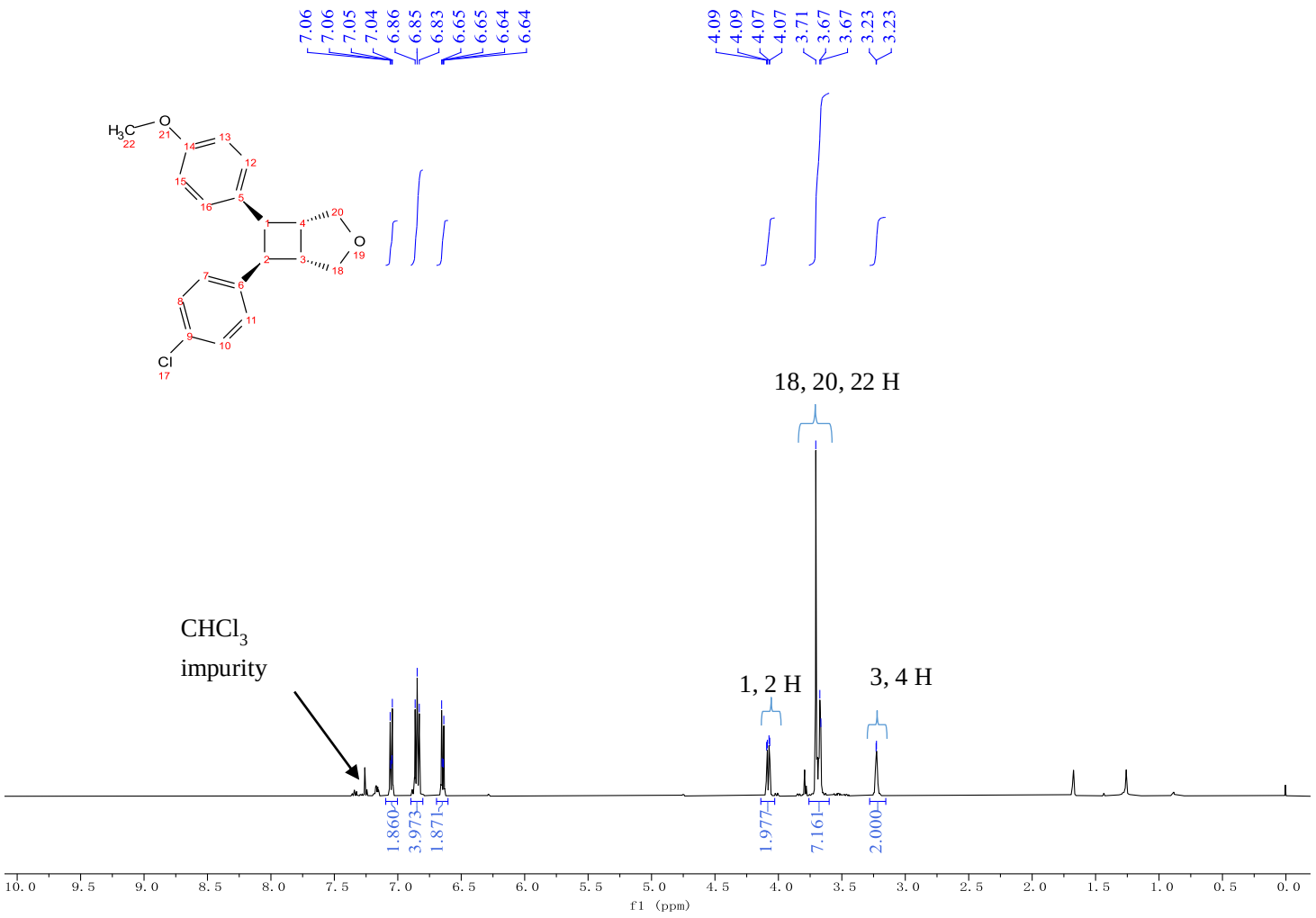
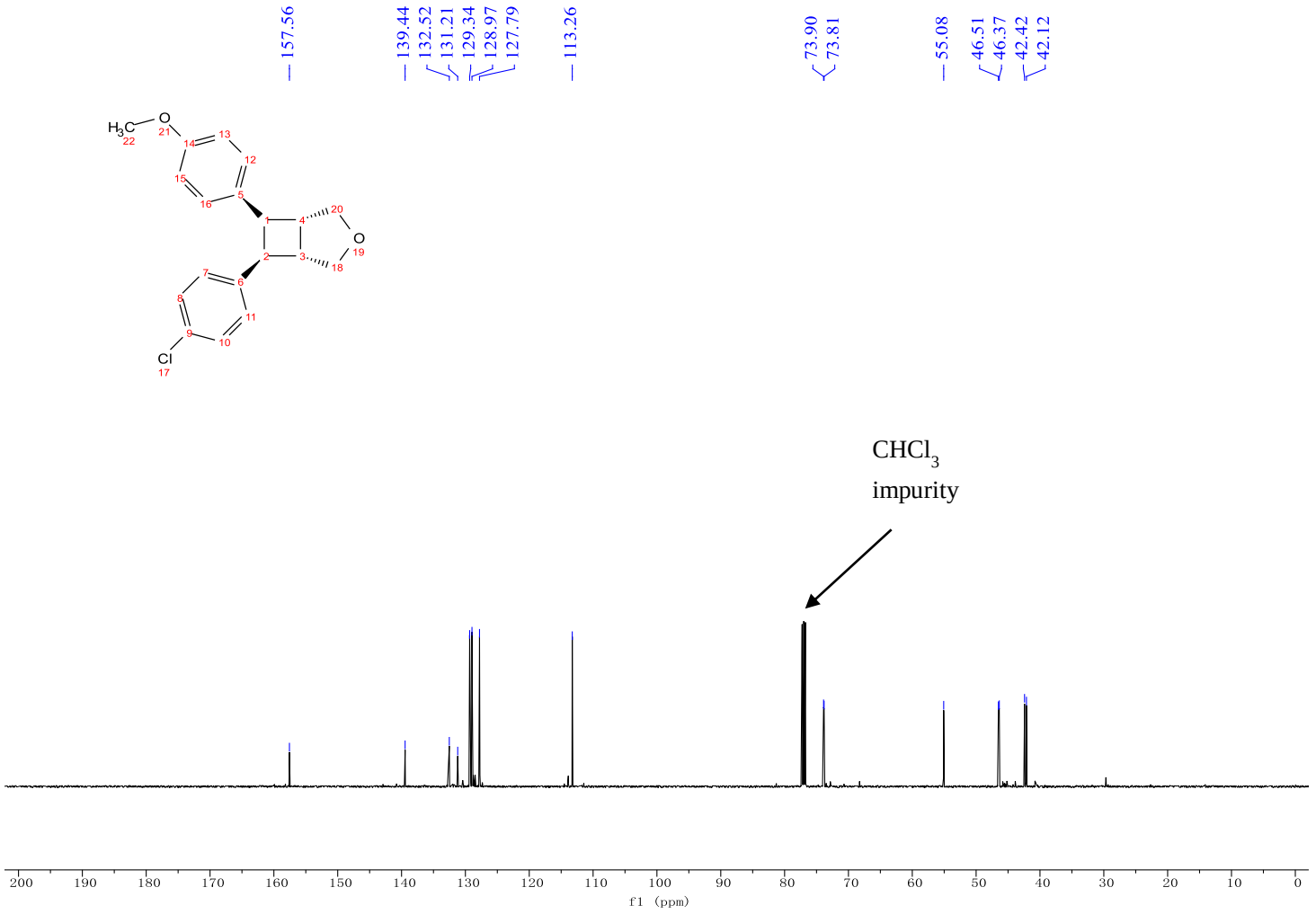
159.90
157.51
136.57
136.54
132.63
129.38
129.32
128.98
128.76
114.52
114.36
113.17
73.90
73.84
55.05
46.42
42.28
42.22
CHCl3
impurity
f1 (ppm)


7.06
7.06
7.05
7.04
6.86
6.85
6.83
6.65
6.65
6.64
6.64
4.09
4.09
4.07
4.07
3.71
3.67
3.67
3.23
3.23
18, 20, 22 H
CHCl3
impurity
1, 2 H
3, 4 H
1.860
3.973
1.871
1.977
7.161
2.000
f1 (ppm)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139.44
132.52
131.21
129.34
128.97
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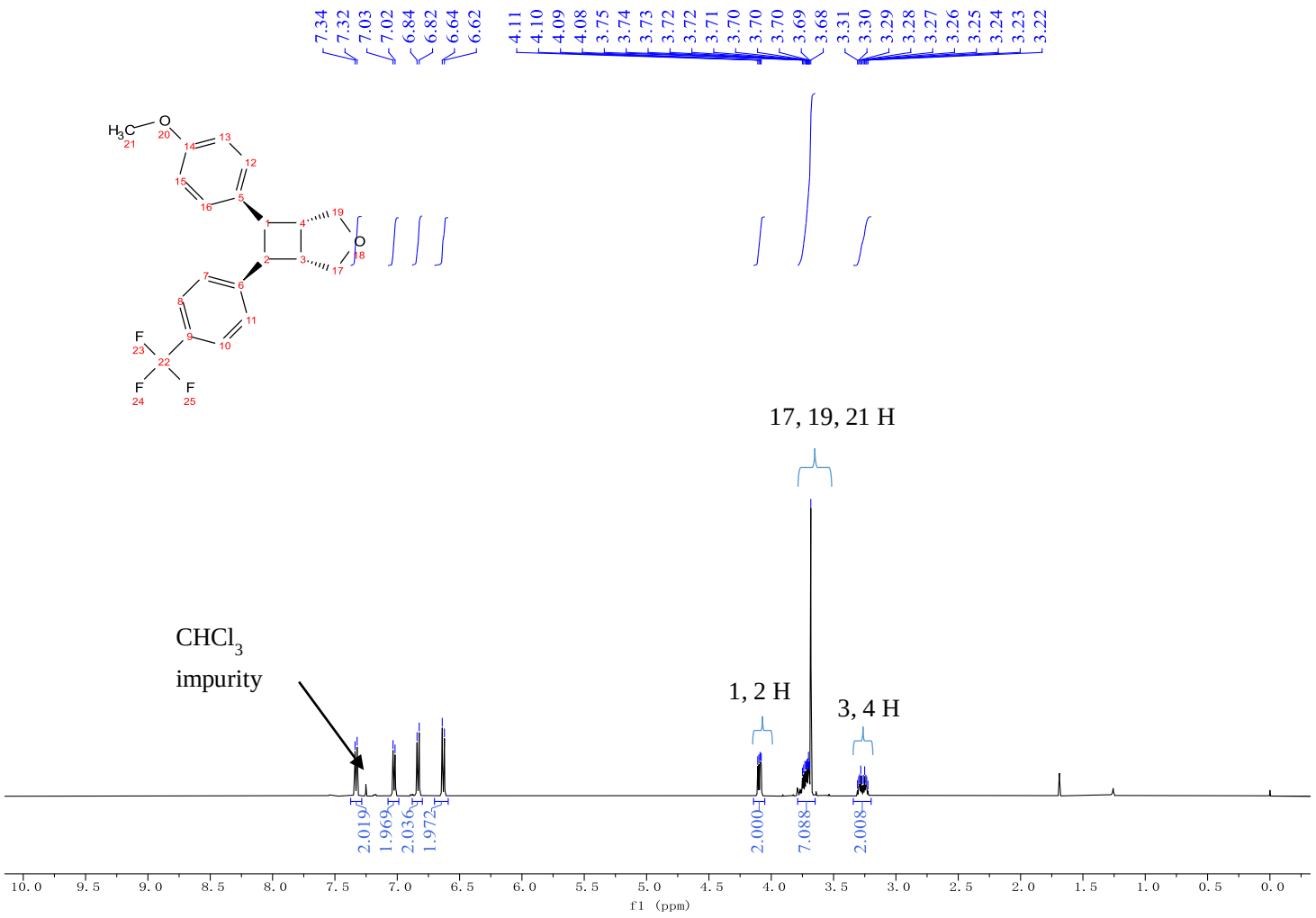
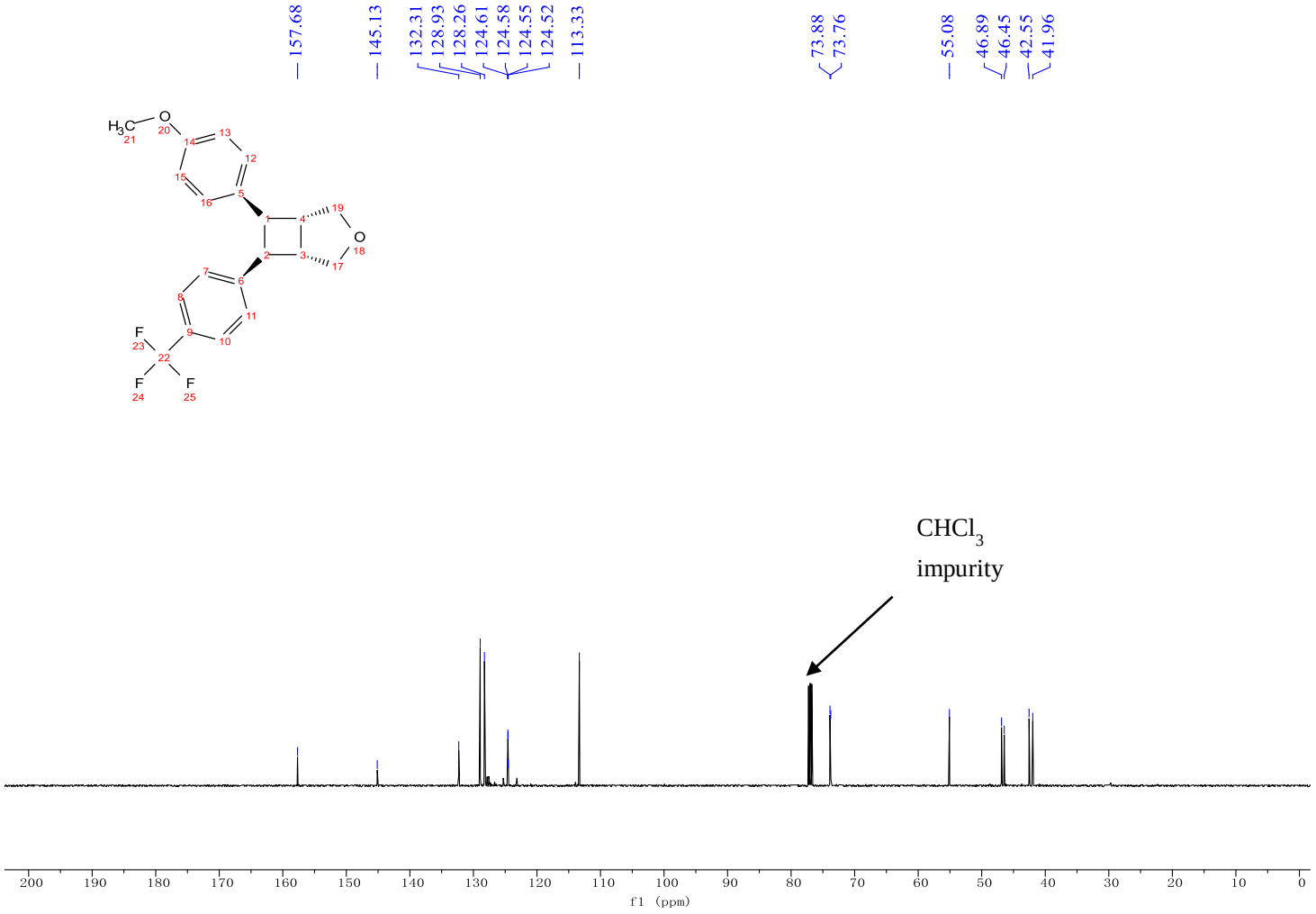
113.26
73.90
73.81
55.08
46.51
46.37
42.42
42.12
CHCl3
impurity
f1 (ppm)


7.34
7.32
7.03
7.02
6.84
6.82
6.64
6.62
4.11
4.10
4.09
4.08
3.75
3.74
3.73
3.72
3.72
3.71
3.70
3.70
3.70
3.69
3.68
3.31
3.30
3.29
3.28
3.27
3.26
3.25
3.24
3.23
3.22
17, 19, 21 H
CHCl3
impurity
1, 2 H
3, 4 H
2.019
1.969
2.036
1.972
2.000
7.088
2.008
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
f1 (ppm)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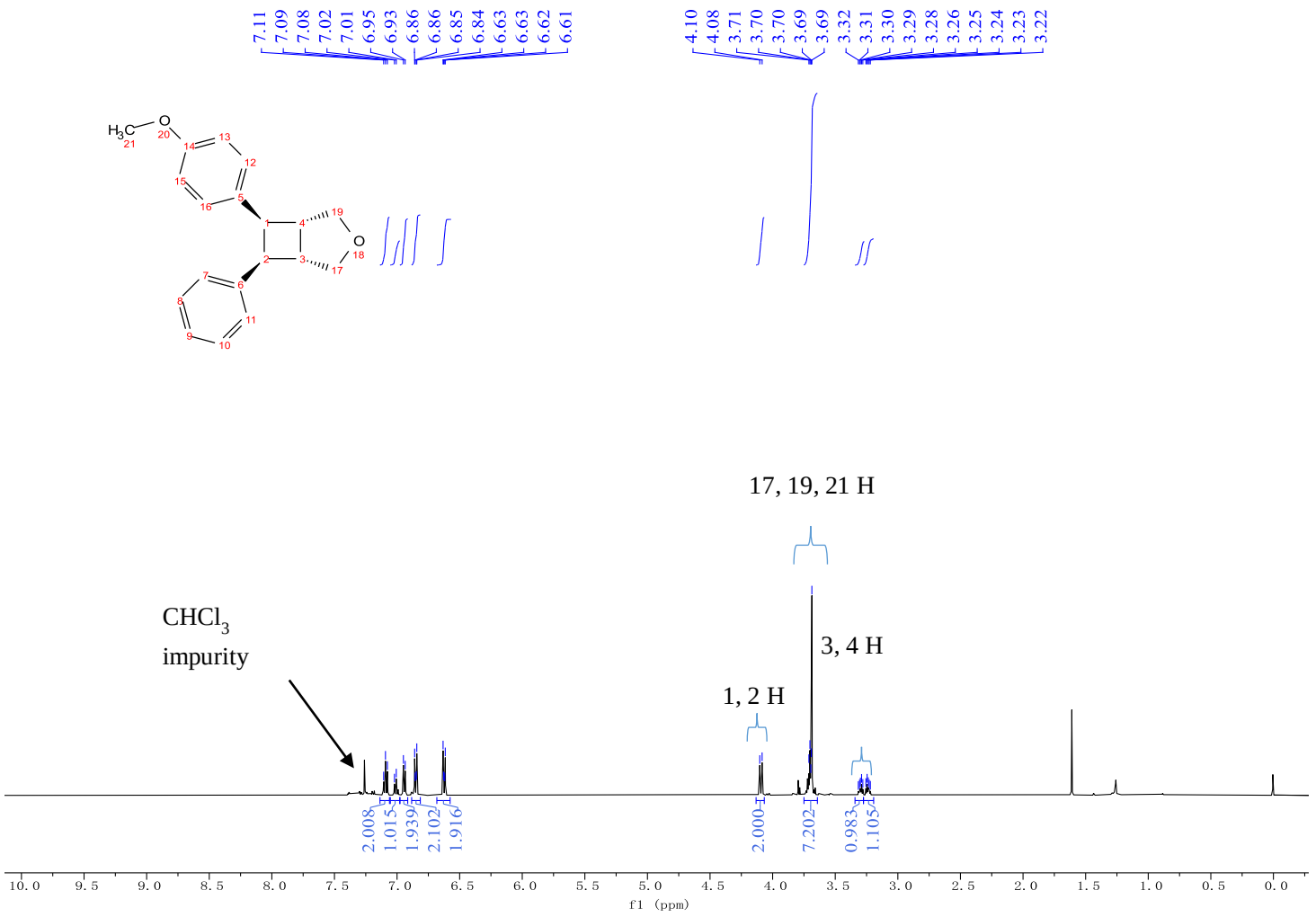
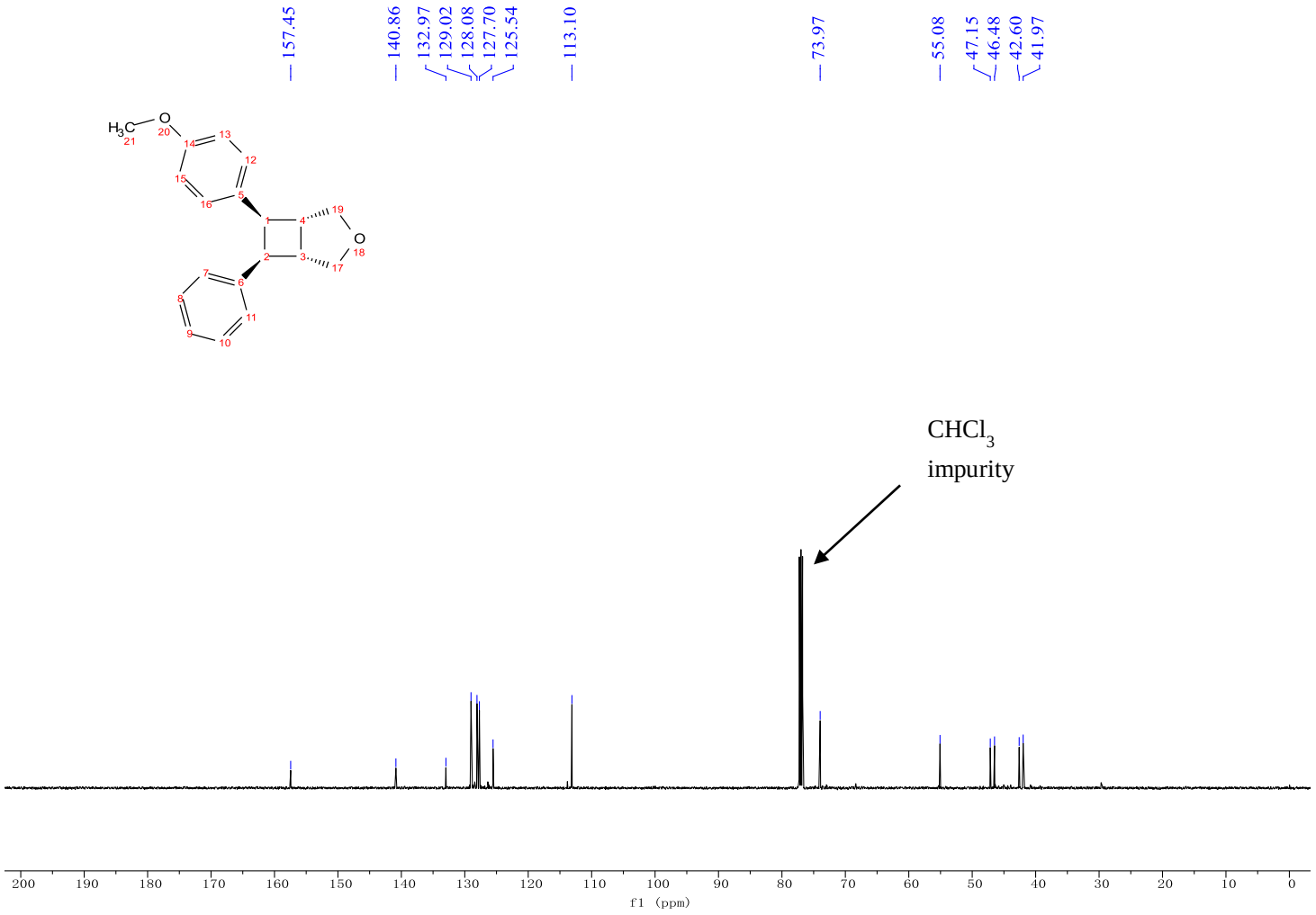
145.13
132.31
128.93
128.26
124.61
124.58
124.55
124.52
113.33
73.88
73.76
55.08
46.89
46.45
42.55
41.96
CHCl3
impurity
f1 (ppm)


7.11
7.09
7.08
7.02
7.01
6.95
6.93
6.86
6.86
6.85
6.84
6.63
6.63
6.62
6.61
4.10
4.08
3.71
3.70
3.70
3.69
3.69
3.32
3.31
3.30
3.29
3.28
3.26
3.25
3.24
3.23
3.22
17, 19, 21 H
CHCl3
impurity
3, 4 H
1, 2 H
2.008
1.015
1.939
2.102
1.916
2.000
7.202
0.983
1.105
f1 (ppm)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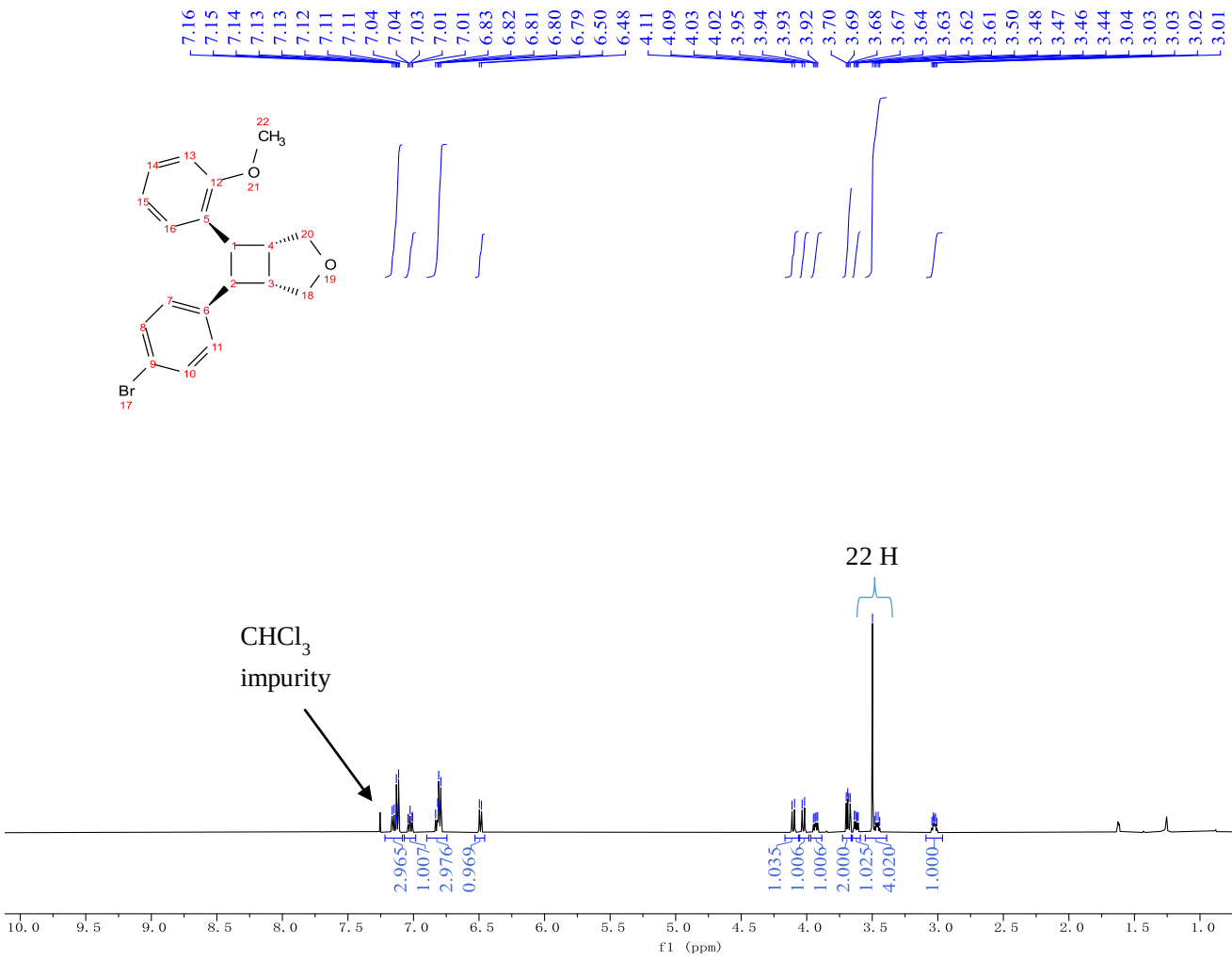
140.86
132.97
129.02
128.08
127.70
125.54
113.10
73.97
55.08
47.15
46.48
42.60
41.97
CHCl3
impurity
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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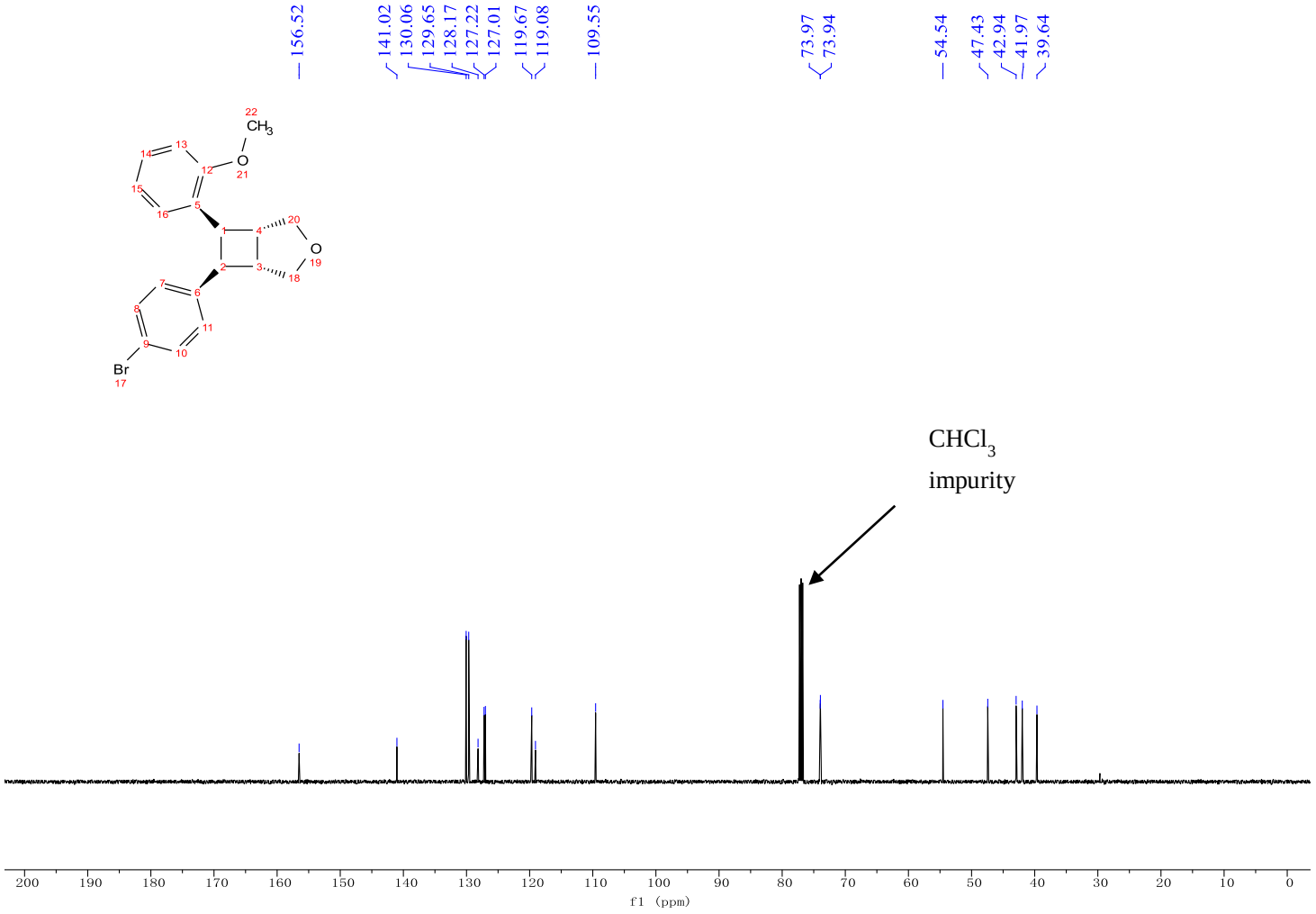
7.15
7.14
7.13
7.13
7.12
7.11
7.11
7.04
7.04
7.03
7.01
7.01
6.83
6.82
6.81
6.80
6.79
6.50
6.48
4.11
4.09
4.03
4.02
3.95
3.94
3.93
3.92
3.70
3.69
3.68
3.67
3.64
3.63
3.62
3.61
3.50
3.48
3.47
3.46
3.44
3.04
3.03
3.03
3.02
3.01
22
CH3
O
21
Br
17
O
19
22 H
CHCl3
impurity
2.965
1.007
2.976
0.969
1.035
1.006
1.006
2.000
1.025
4.020
1.000
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
f1 (ppm)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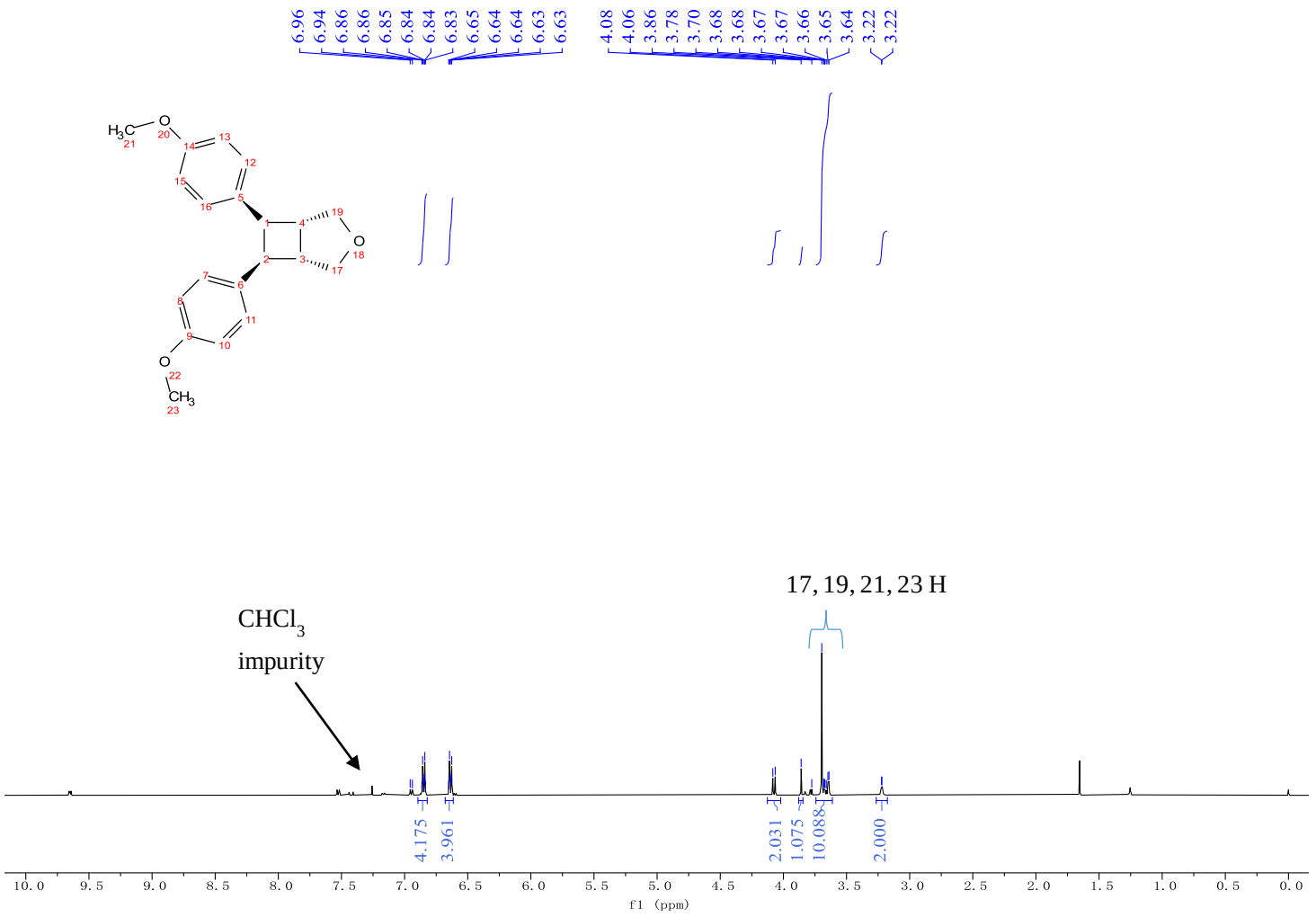
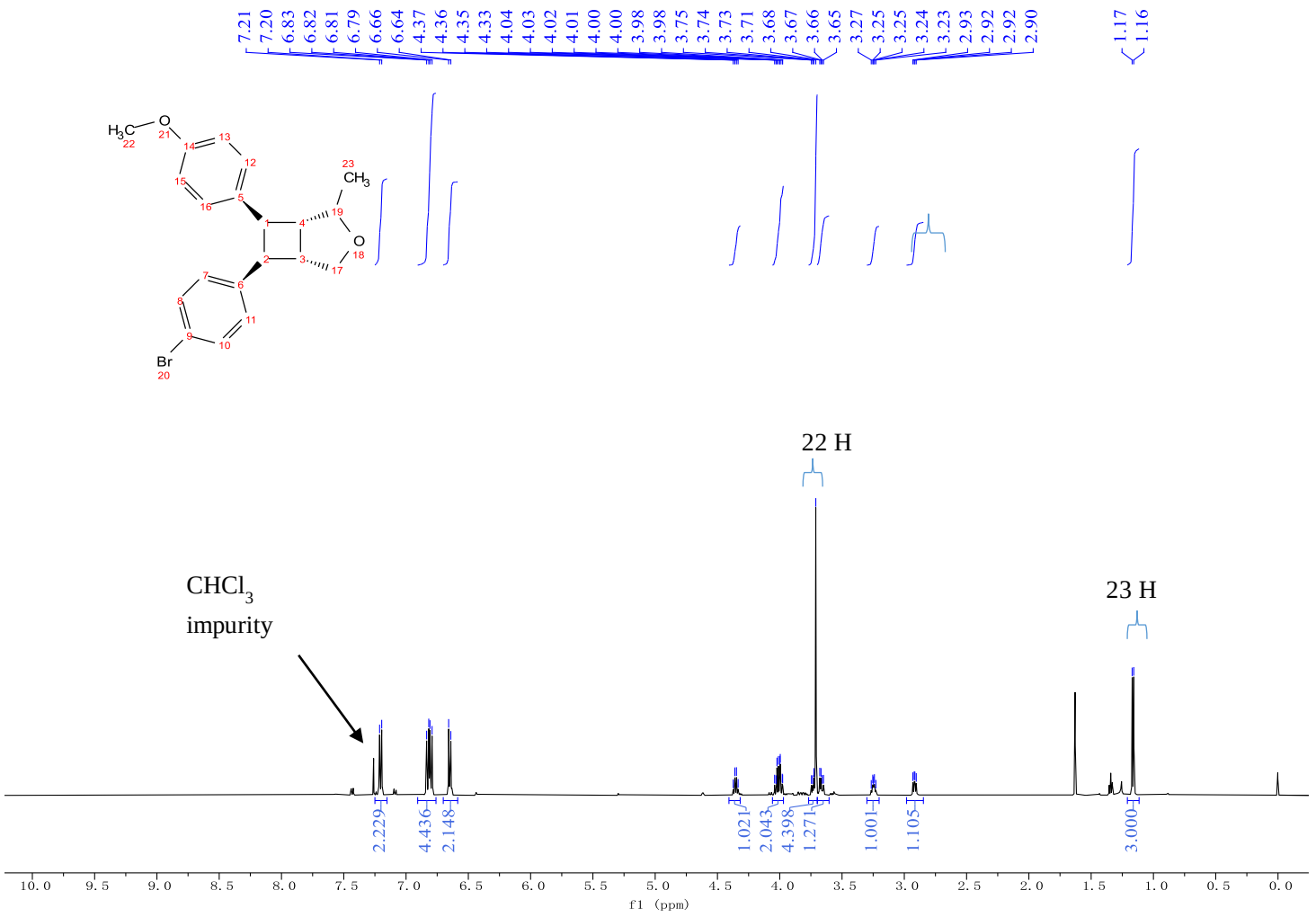
141.02
130.06
129.65
128.17
127.22
127.01
119.67
119.08
109.55
73.97
73.94
54.54
47.43
42.94
41.97
39.64
CH3
O
Br
CHCl3
impurity
f1 (ppm)


6.96
6.94
6.86
6.86
6.85
6.84
6.84
6.83
6.65
6.64
6.64
6.63
6.63
4.08
4.06
3.86
3.78
3.70
3.68
3.68
3.67
3.67
3.66
3.65
3.64
3.22
3.22
17, 19, 21, 23 H
CHCl3
impurity
4.175
3.961
2.031
1.075
10.088
2.000
f1 (ppm)


7.21
7.20
6.83
6.82
6.81
6.79
6.66
6.64
4.37
4.36
4.35
4.33
4.04
4.03
4.02
4.01
4.00
4.00
3.98
3.98
3.75
3.74
3.73
3.71
3.68
3.67
3.66
3.65
3.27
3.25
3.25
3.24
3.23
2.93
2.92
2.92
2.90
1.17
1.16
22 H
23 H
CHCl3
impurity
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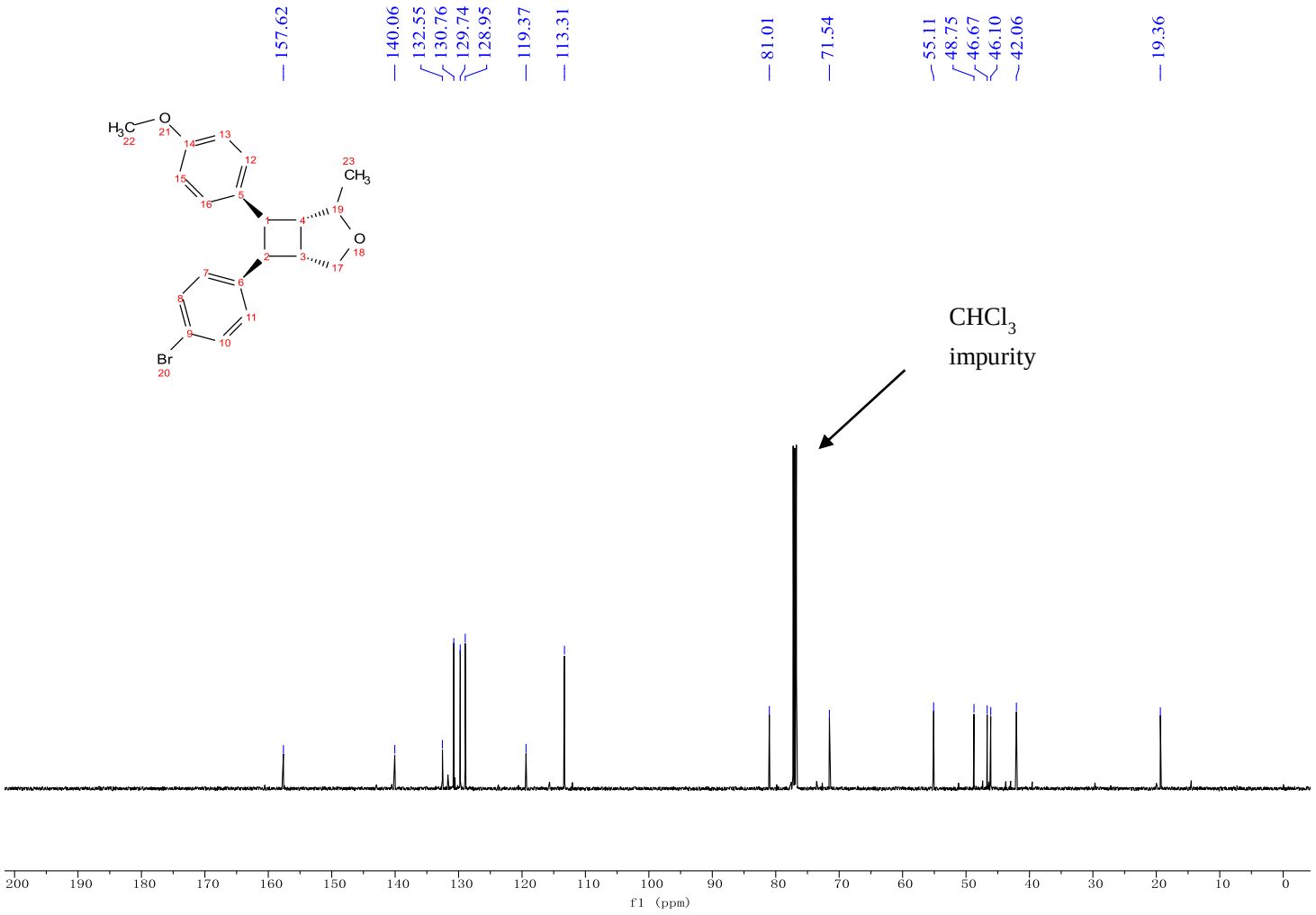
4.436
2.148
1.021
2.043
4.398
1.271
1.001
1.105
3.000
f1 (ppm)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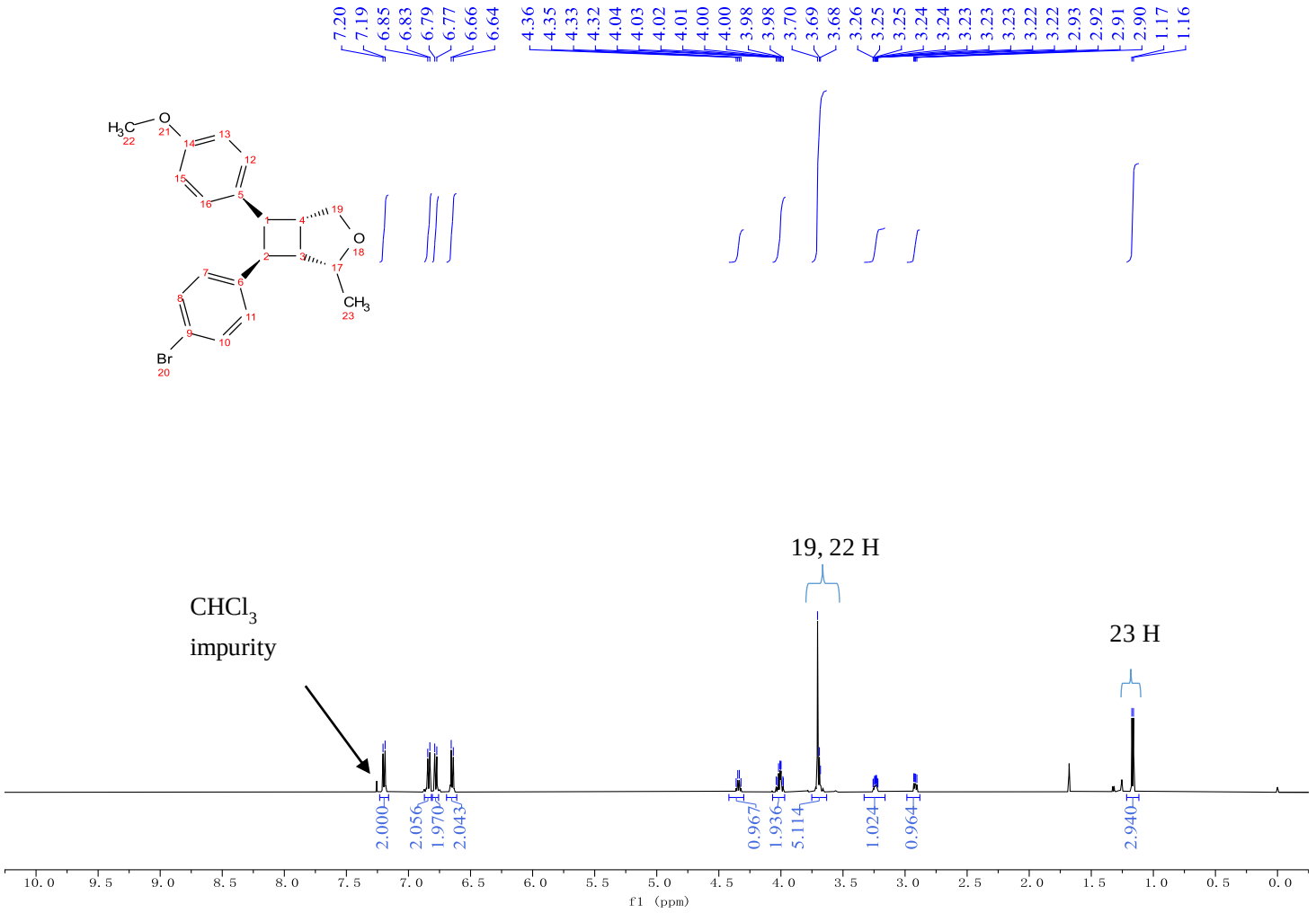
140.06
132.55
130.76
129.74
128.95
119.37
113.31
81.01
71.54
55.11
48.75
46.67
46.10
42.06
19.36
CHCl3
impurity
f1 (ppm)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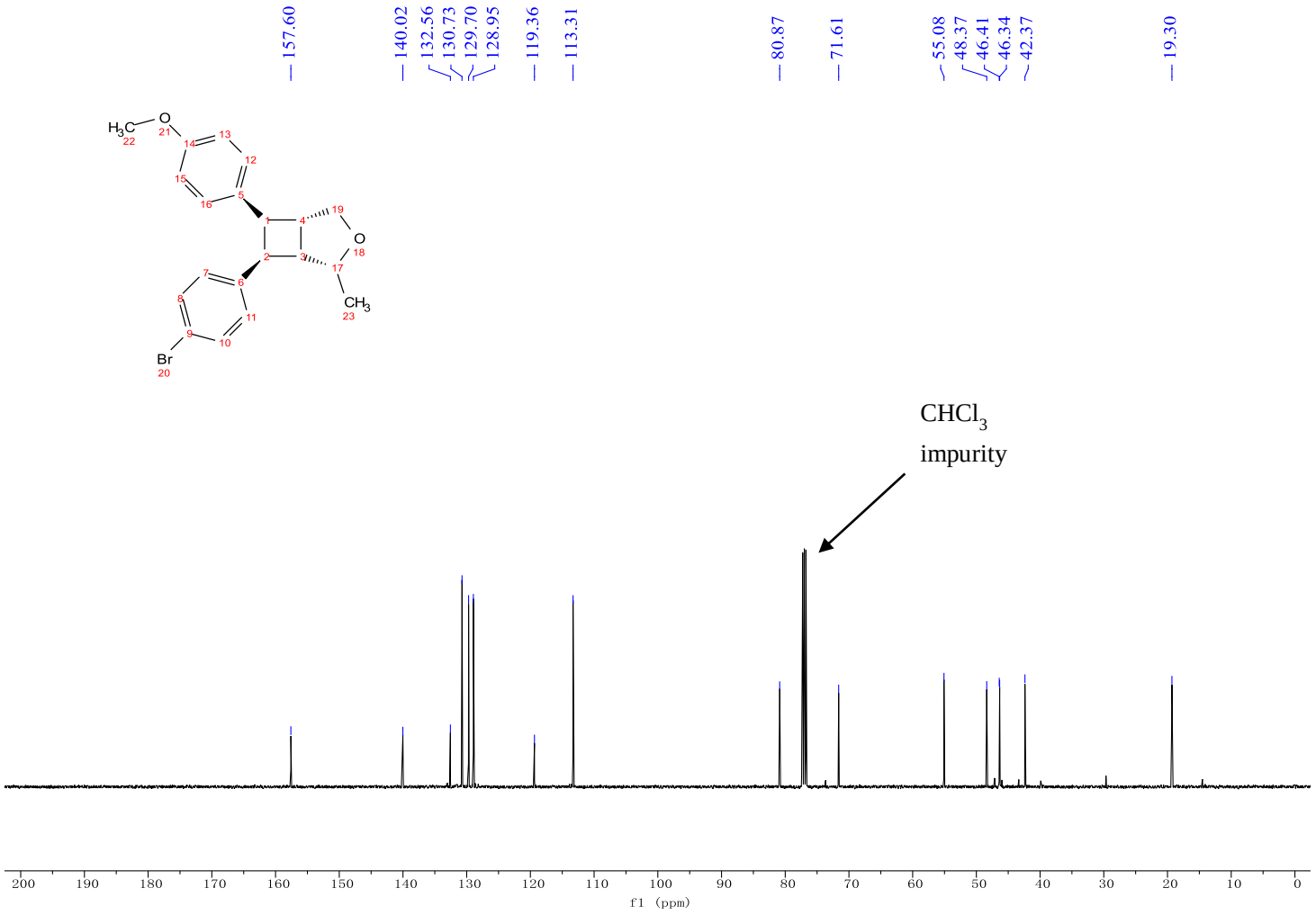
7.19
6.85
6.83
6.79
6.77
6.66
6.64
4.36
4.35
4.33
4.32
4.04
4.03
4.02
4.01
4.00
4.00
3.98
3.98
3.70
3.69
3.68
3.26
3.25
3.25
3.24
3.24
3.23
3.23
3.23
3.22
3.22
2.93
2.92
2.91
2.90
1.17
1.16
H3C
O
Br
CH3
19, 22 H
23 H
CHCl3
impurity
2.000
2.056
1.970
2.043
0.967
1.936
5.114
1.024
0.964
2.940
f1 (ppm)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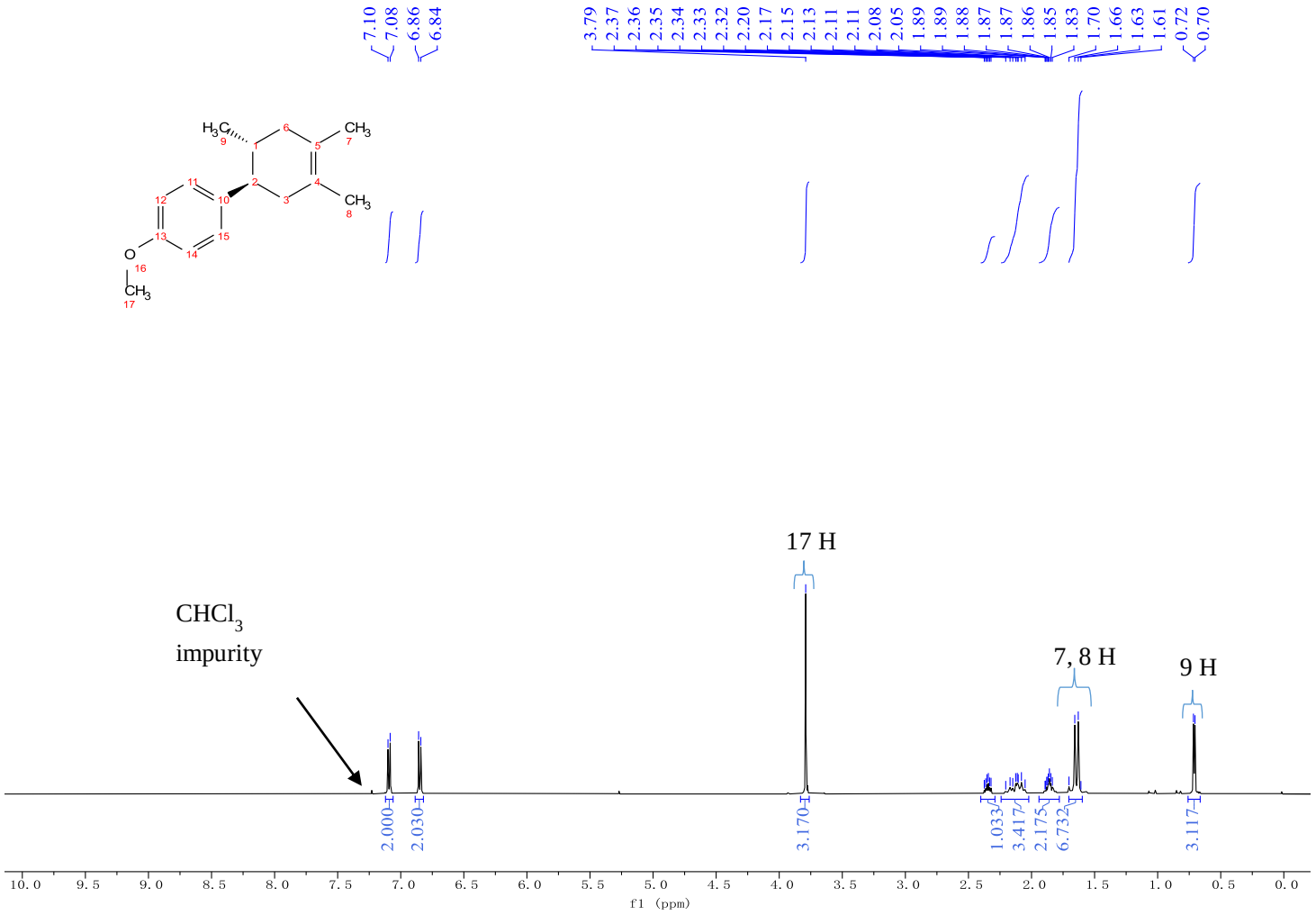
130.73
129.70
128.95
119.36
113.31
80.87
71.61
55.08
48.37
46.41
46.34
42.37
19.30
CHCl3
impurity
f1 (ppm)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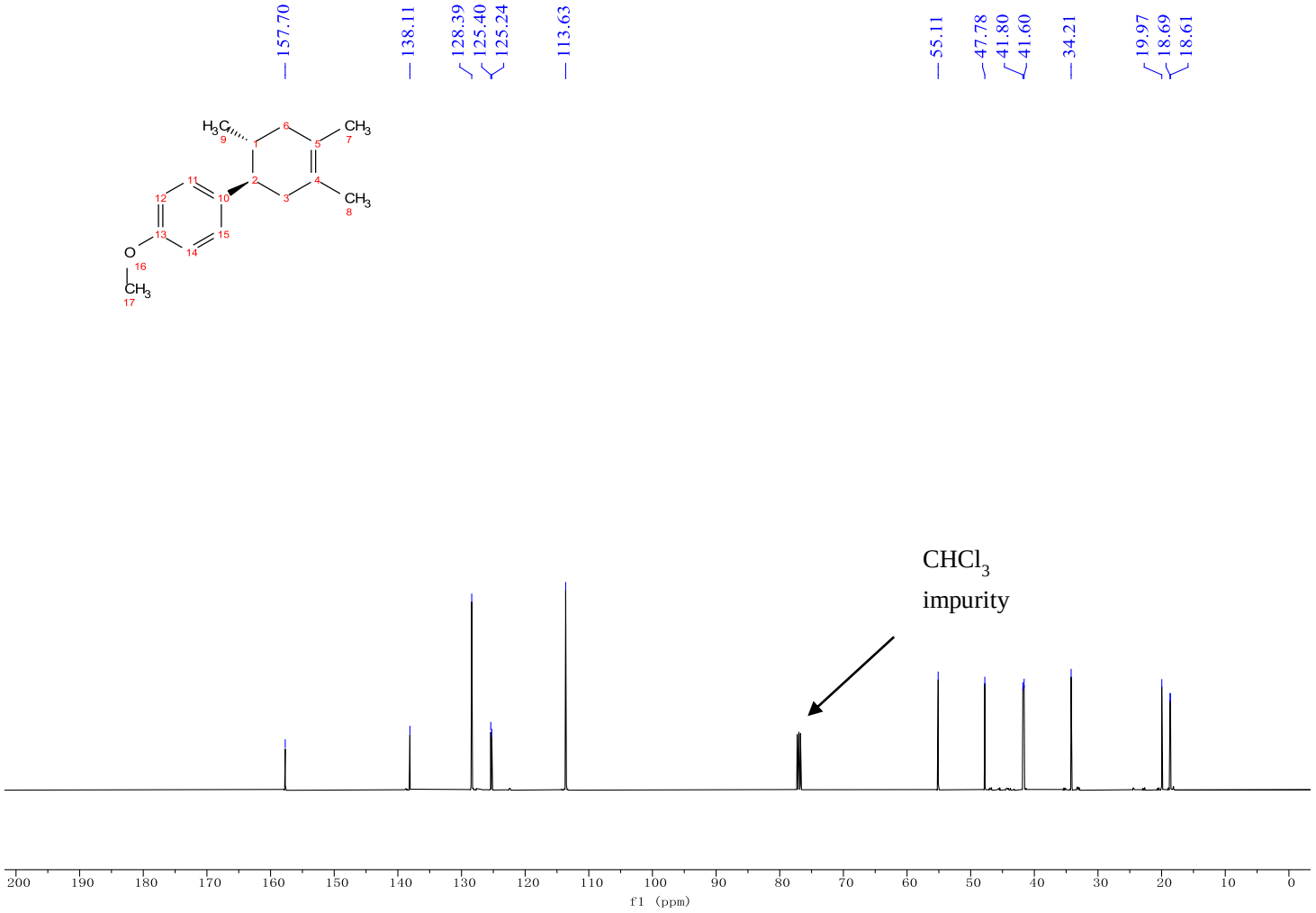
7.08
6.86
6.84
3.79
2.37
2.36
2.35
2.34
2.33
2.32
2.20
2.17
2.15
2.13
2.11
2.11
2.08
2.05
1.89
1.89
1.88
1.87
1.87
1.86
1.85
1.83
1.70
1.66
1.63
1.61
0.72
0.70
17 H
CHCl3
impurity
7, 8 H
9 H
2.000
2.030
3.170
1.033
3.417
2.175
6.732
3.117
f1 (ppm)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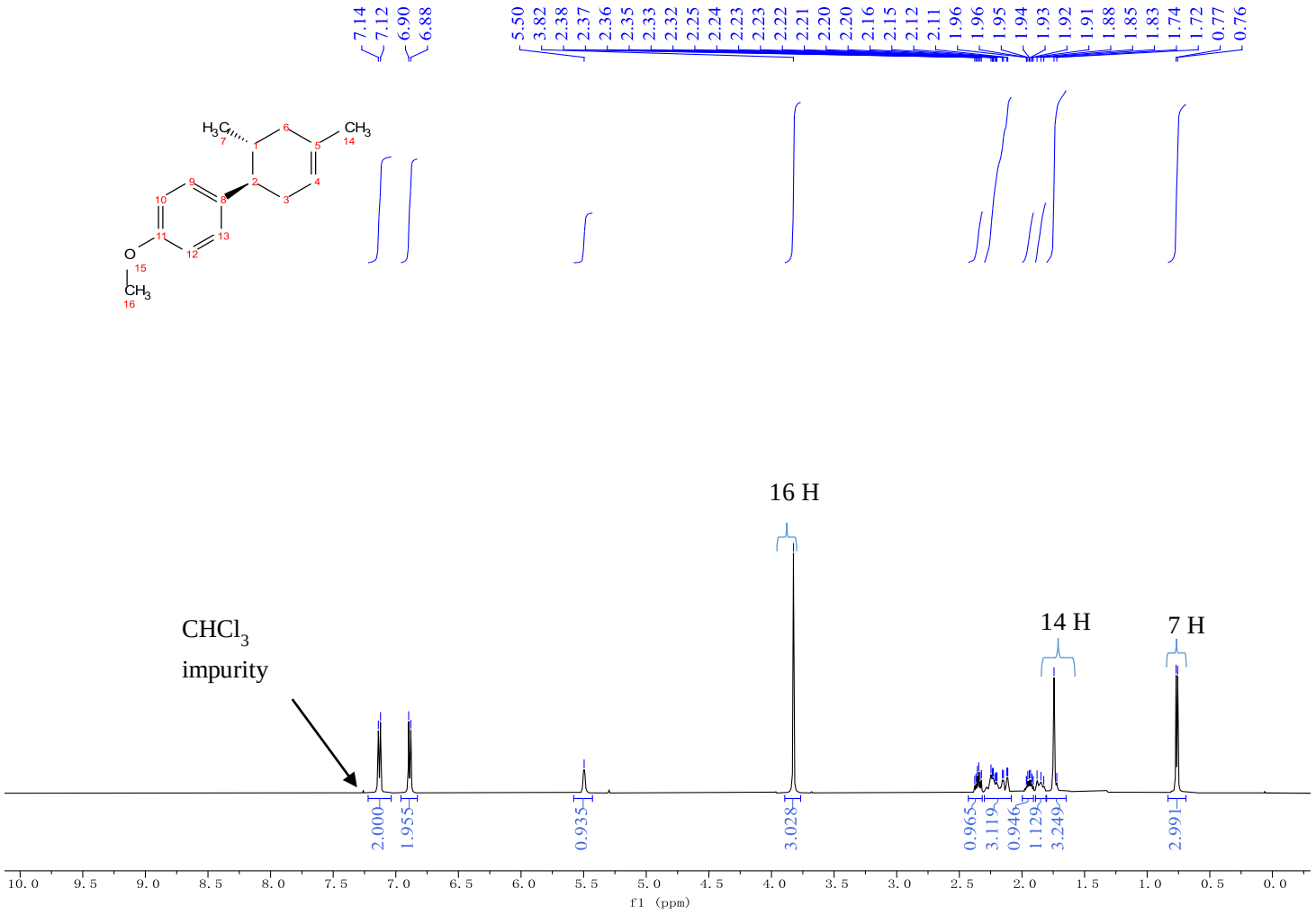
138.11
128.39
125.40
125.24
113.63
55.11
47.78
41.80
41.60
34.21
19.97
18.69
18.61
CHCl3
impurity
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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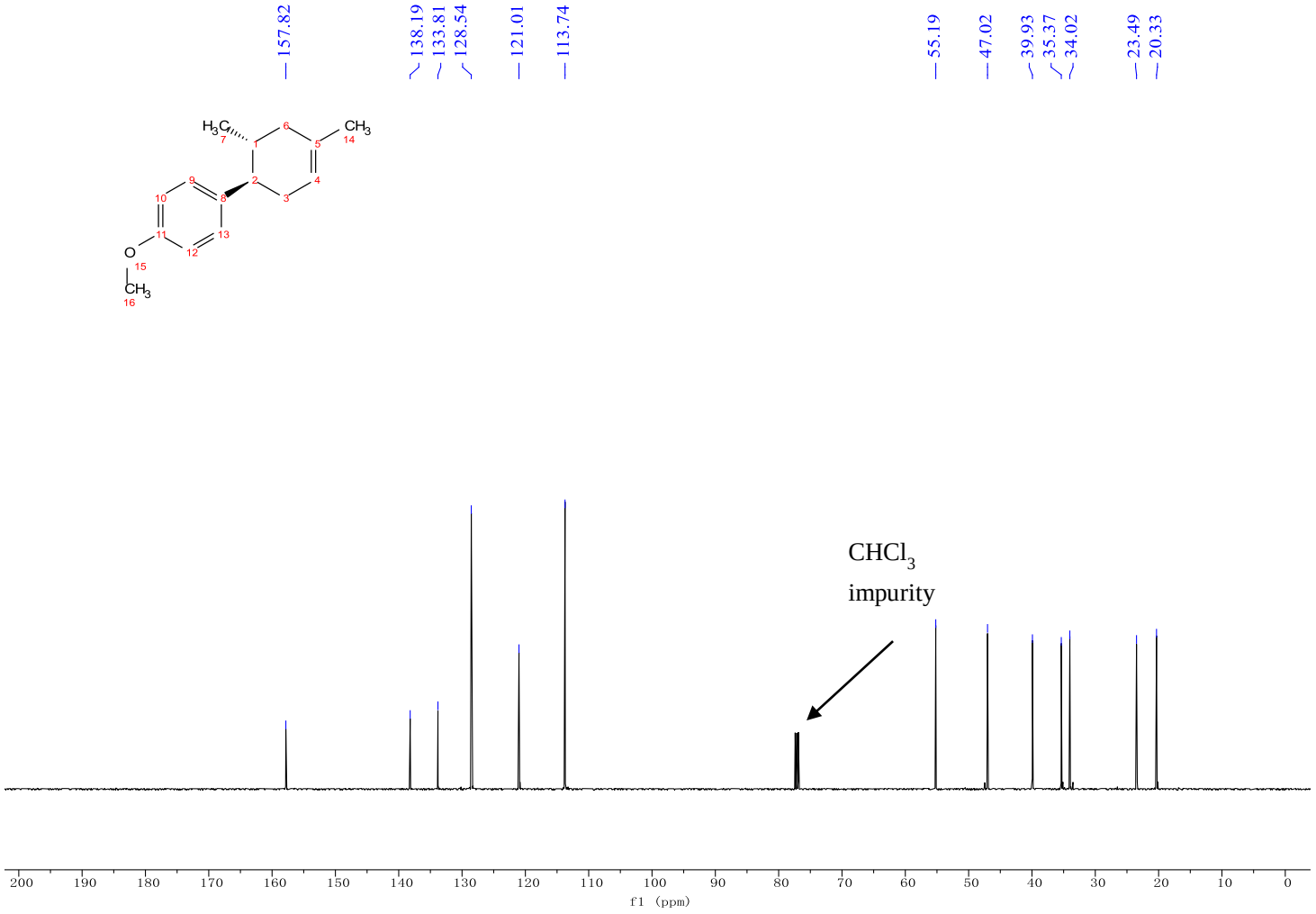
7.12
6.90
6.88
5.50
3.82
2.38
2.37
2.36
2.35
2.33
2.32
2.25
2.24
2.23
2.23
2.22
2.21
2.20
2.20
2.16
2.15
2.12
2.11
1.96
1.96
1.95
1.94
1.93
1.92
1.91
1.88
1.85
1.83
1.74
1.72
0.77
0.76
16 H
14 H
7 H
CHCl3
impurity
2.000
1.955
0.935
3.028
0.965
3.119
0.946
1.129
3.249
2.991
f1 (ppm)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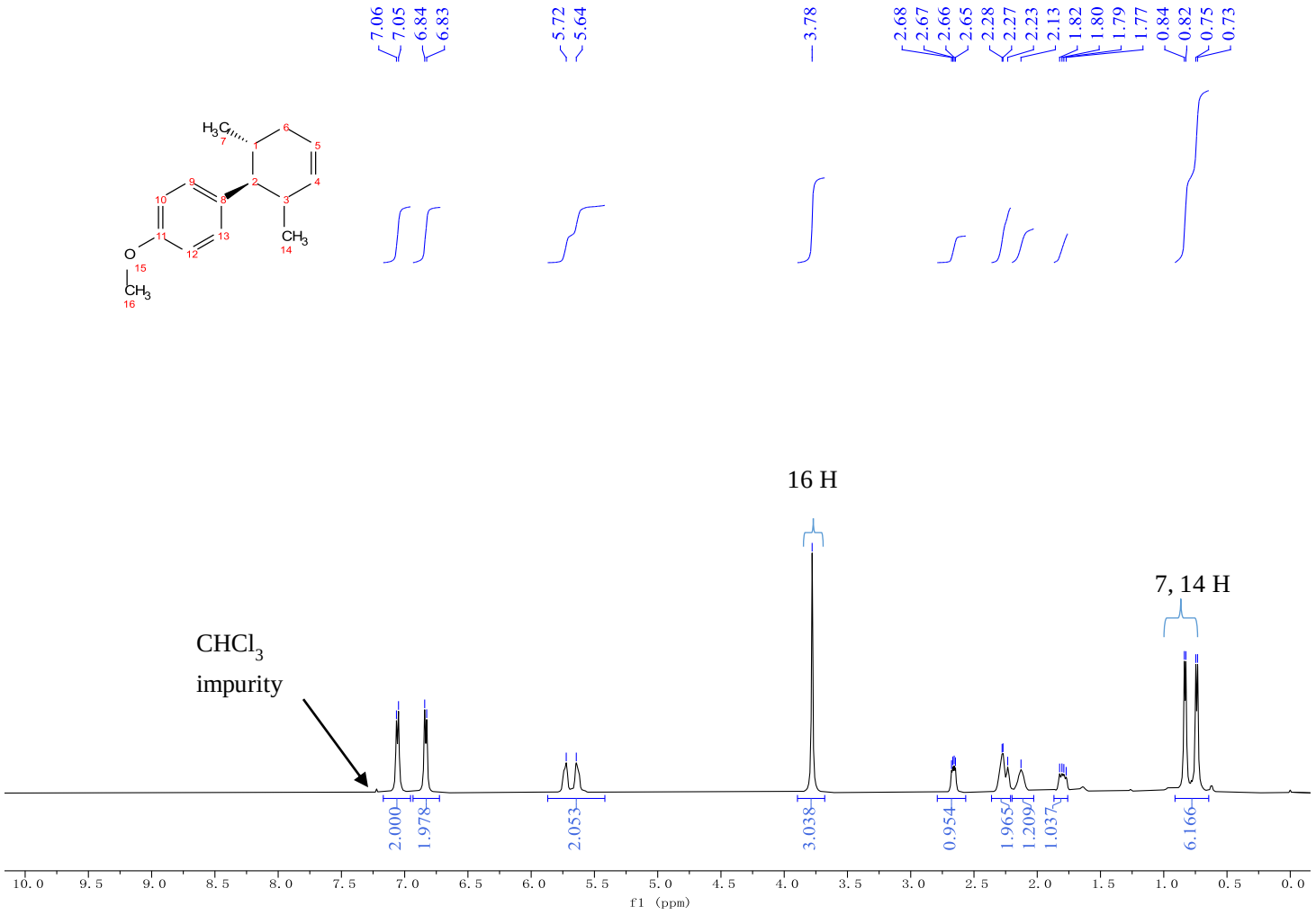
133.81
128.54
121.01
113.74
55.19
47.02
39.93
35.37
34.02
23.49
20.33
CHCl3
impurity
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.05
6.84
6.83
5.72
5.64
3.78
2.68
2.67
2.66
2.65
2.28
2.27
2.23
2.13
1.82
1.80
1.79
1.77
0.84
0.82
0.75
0.73
16 H
7, 14 H
CHCl3
impurity
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1.978
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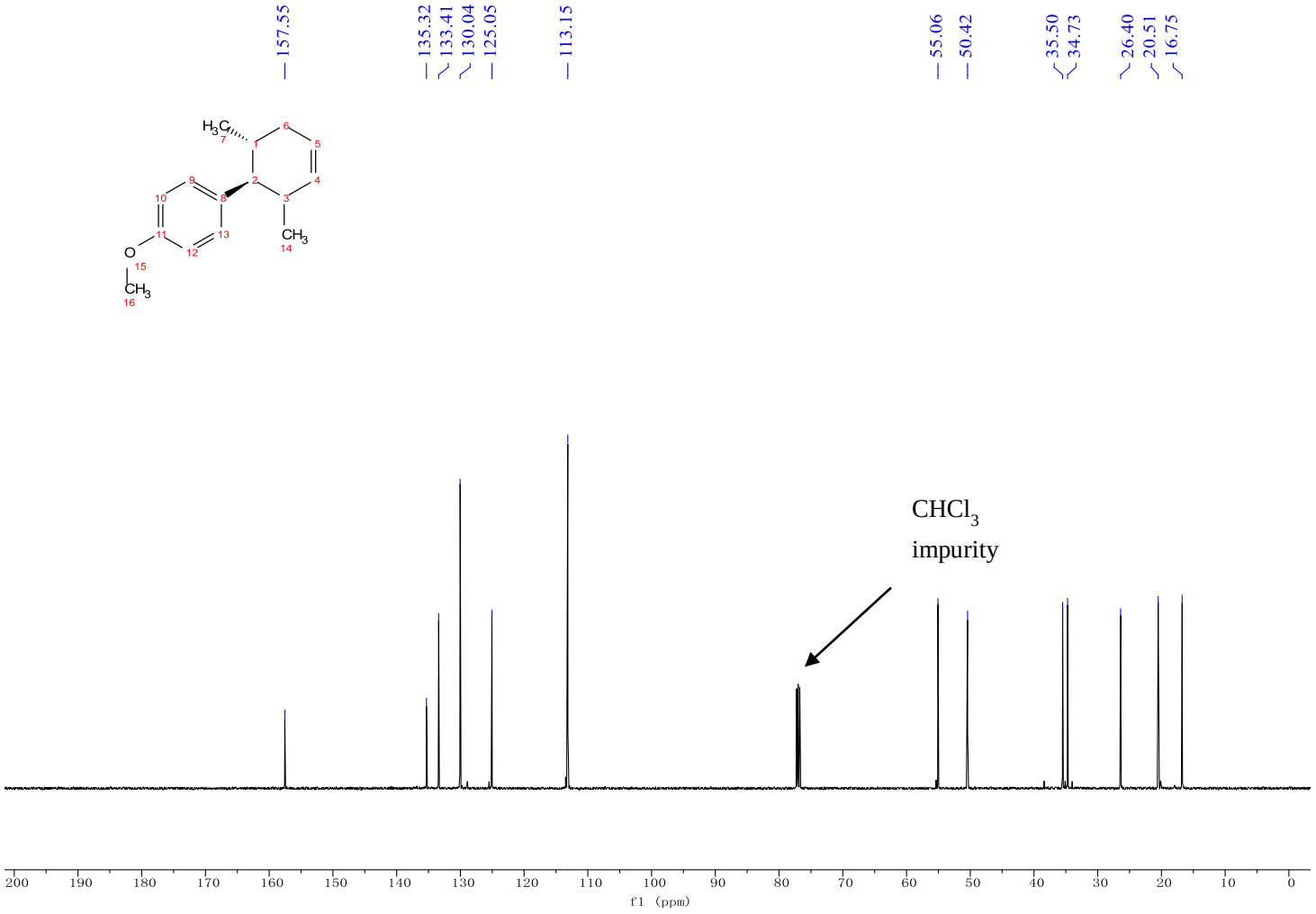
3.038
0.954
1.965
1.209
1.037
6.166
f1 (ppm)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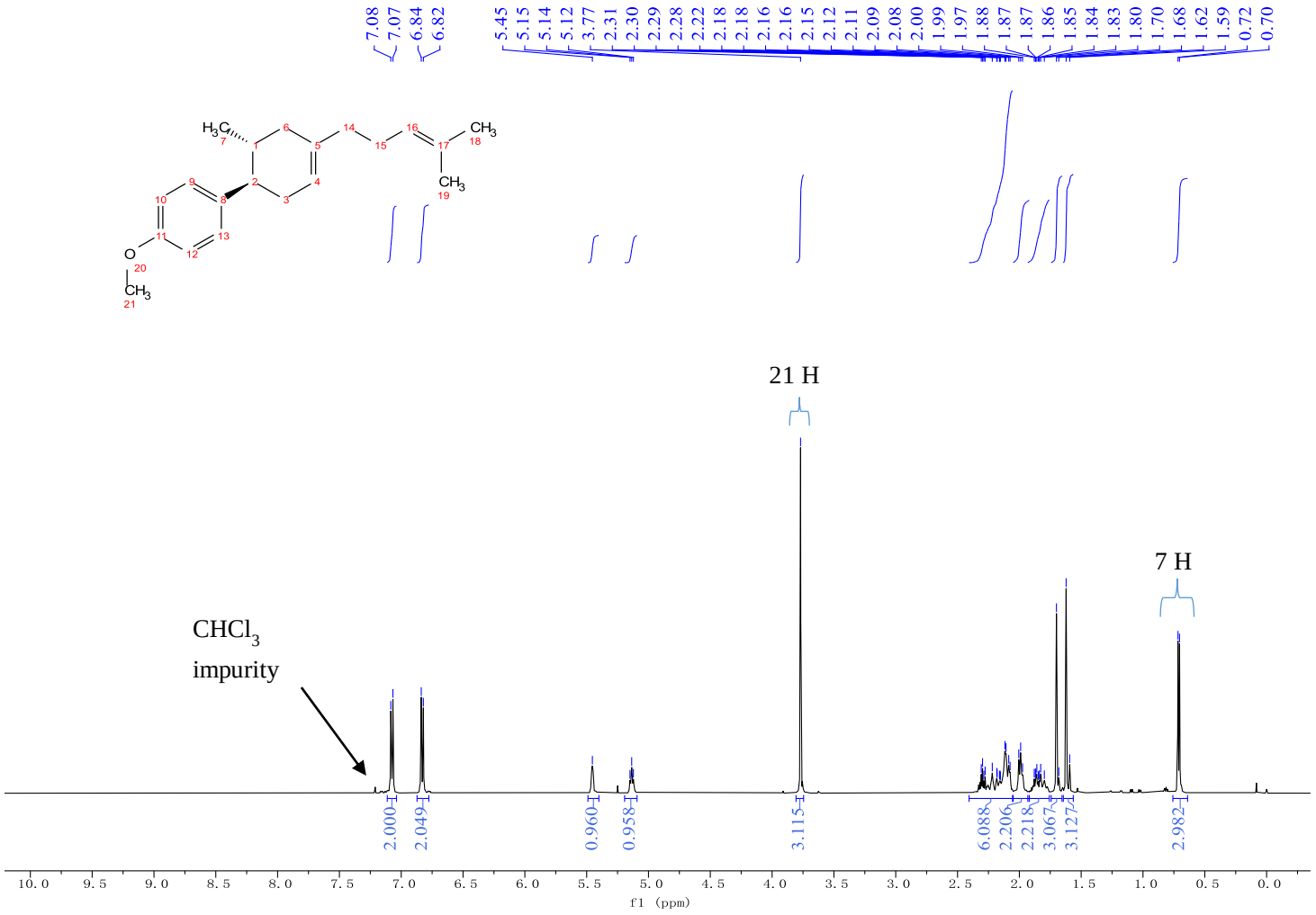
130.04
125.05
113.15
55.06
50.42
35.50
34.73
26.40
20.51
16.75
CHCl3
impurity
f1 (ppm)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7.07
6.84
6.82
5.45
5.15
5.14
5.12
3.77
2.31
2.30
2.29
2.28
2.22
2.18
2.18
2.16
2.16
2.15
2.12
2.11
2.09
2.08
2.00
1.99
1.97
1.88
1.87
1.87
1.86
1.85
1.84
1.83
1.80
1.70
1.68
1.62
1.59
0.72
0.70
21 H
7 H
CHCl3
impurity
2.000
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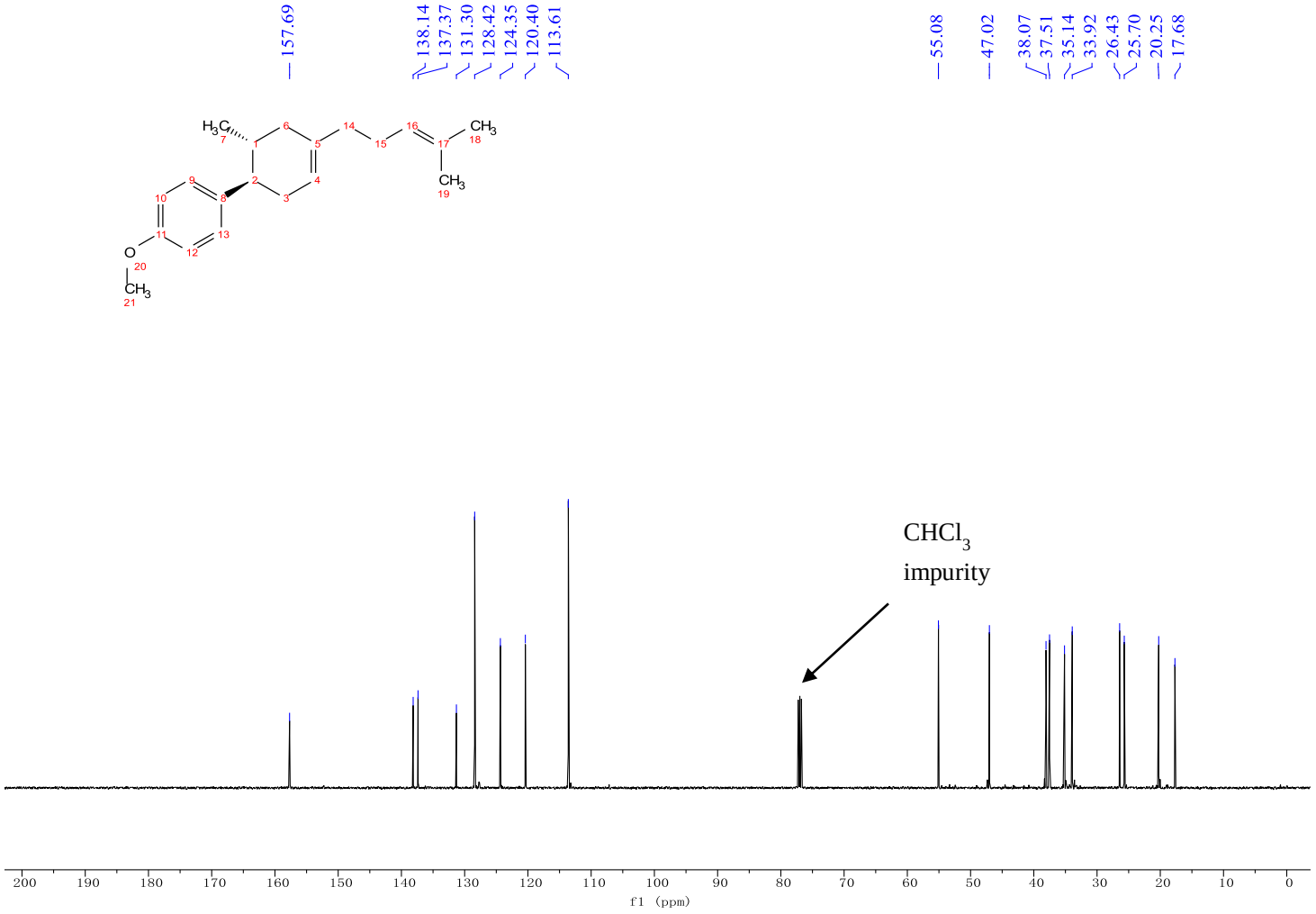
0.958
3.115
6.088
2.206
2.218
3.067
3.127
2.982
f1 (ppm)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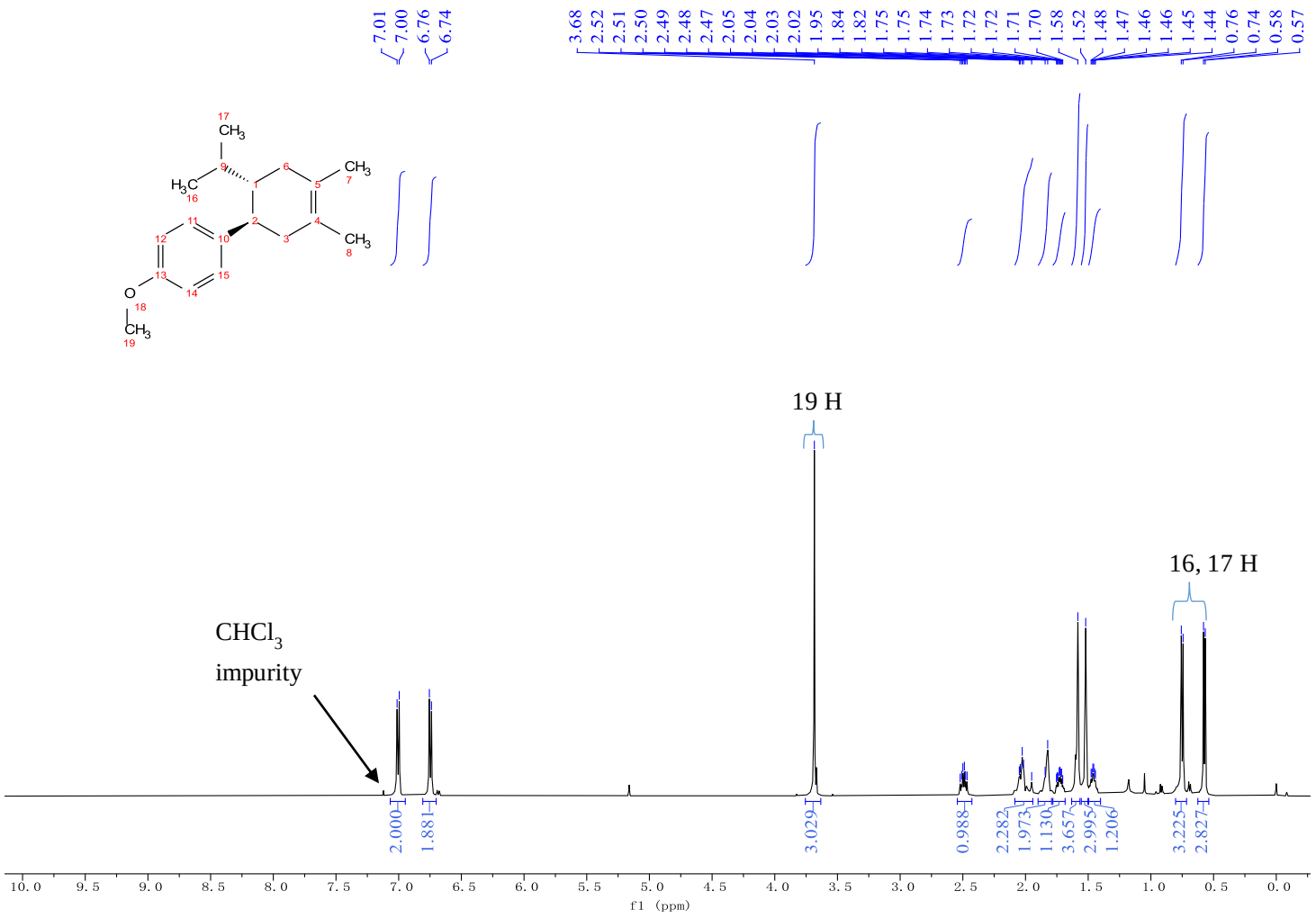
138.14
137.37
131.30
128.42
124.35
120.40
113.61
55.08
47.02
38.07
37.51
35.14
33.92
26.43
25.70
20.25
17.68
CHCl3
impurity
f1 (ppm)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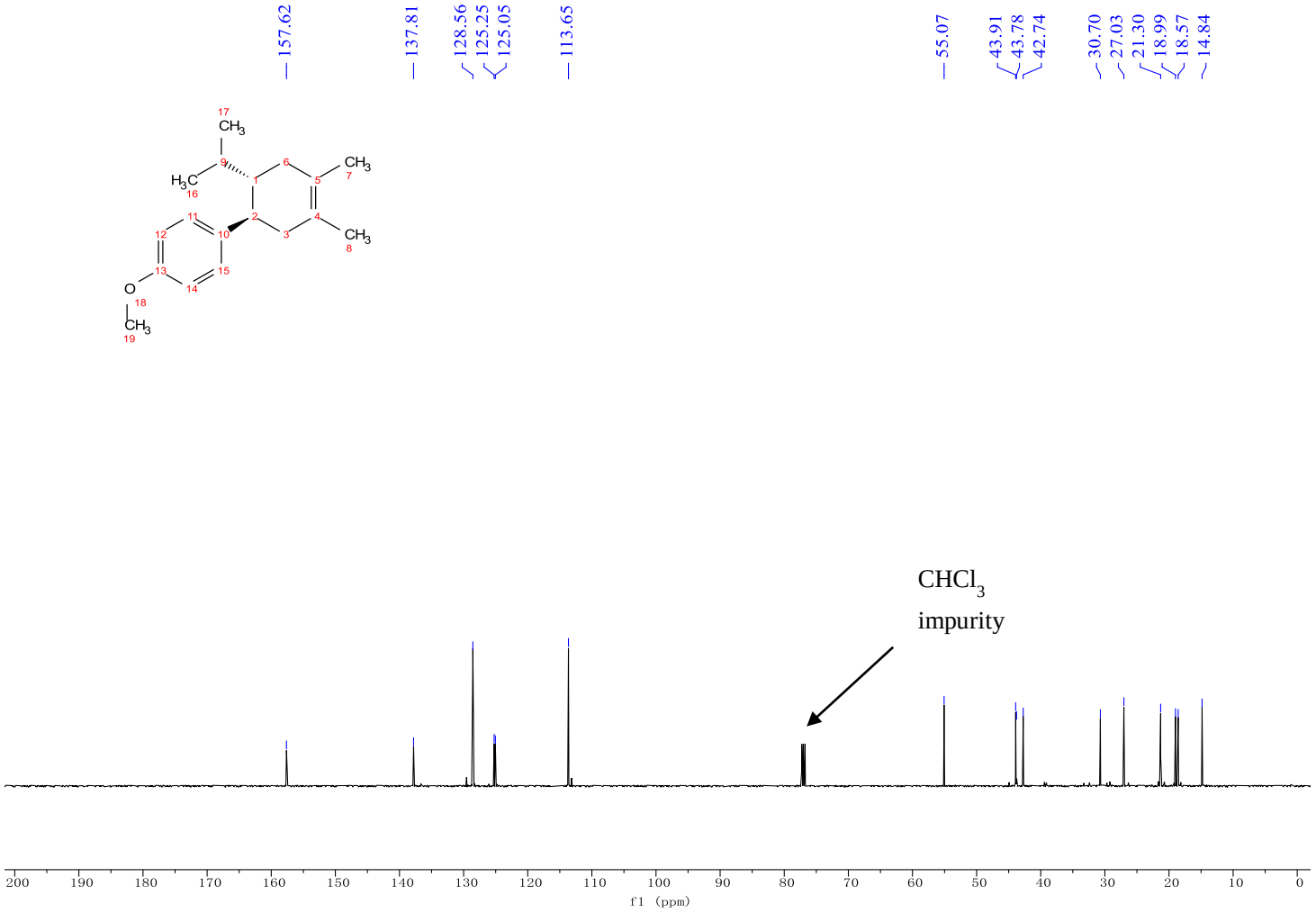
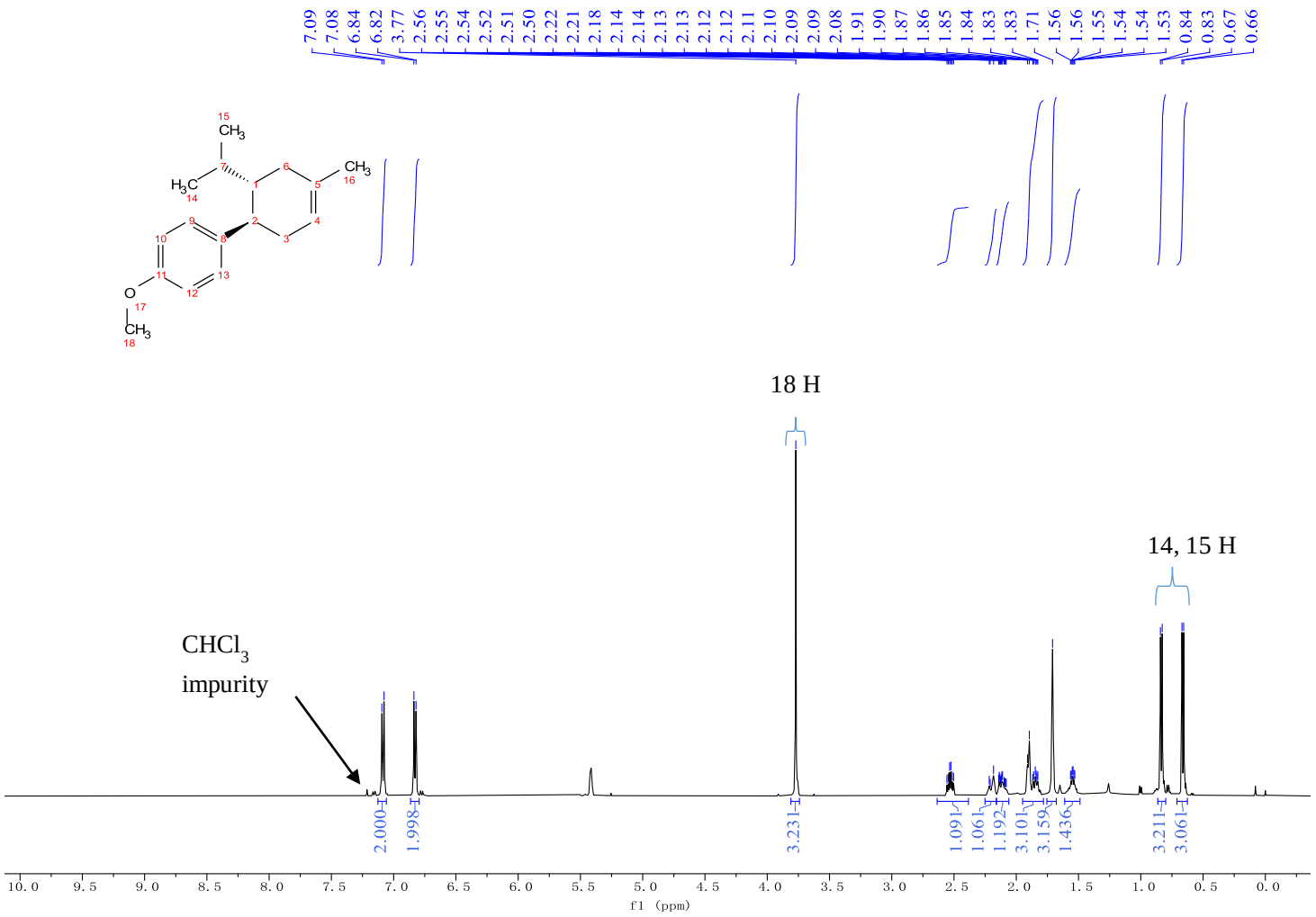
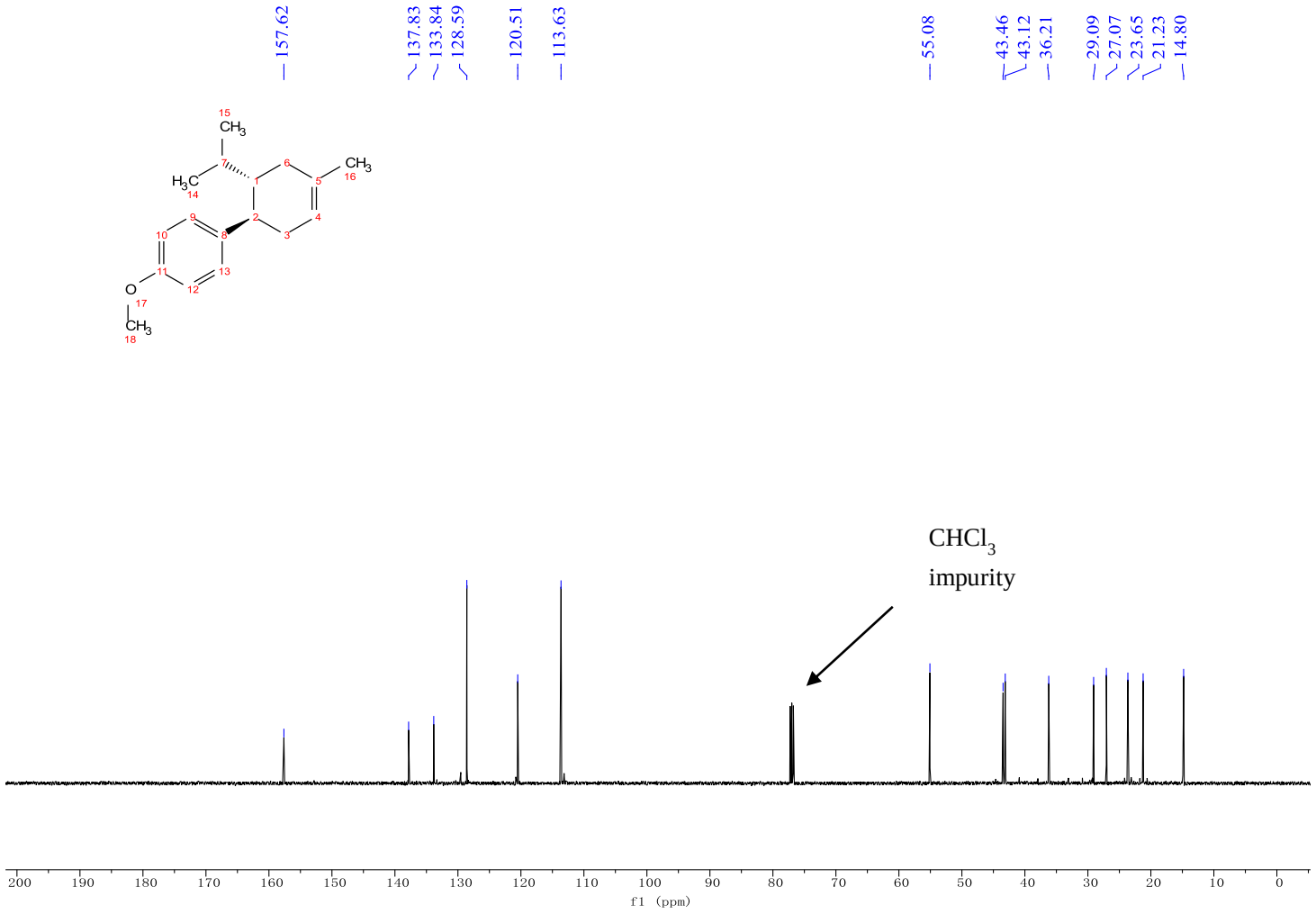
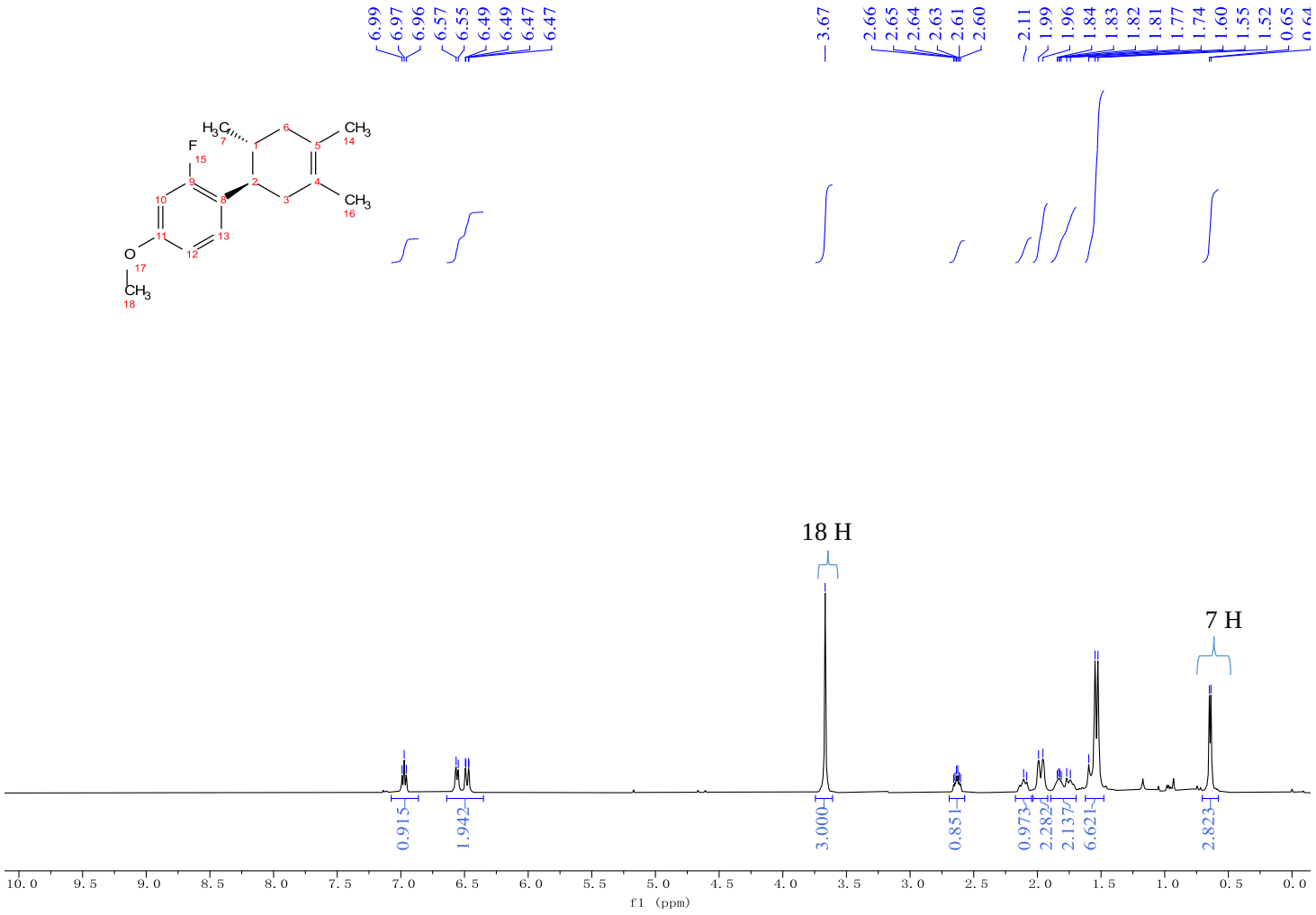
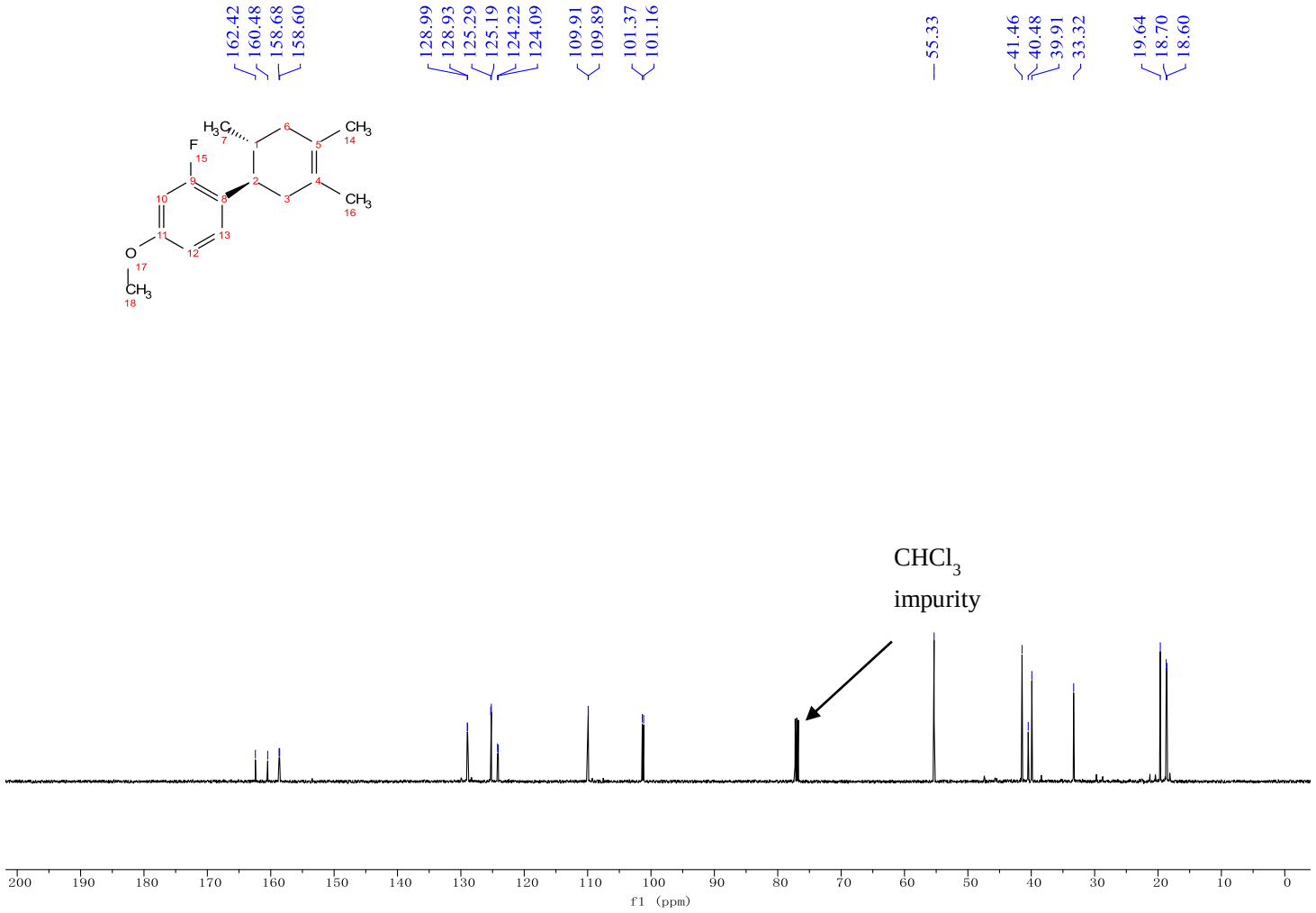
7.00
6.76
6.74
3.68
2.52
2.51
2.50
2.49
2.48
2.47
2.05
2.04
2.03
2.02
1.95
1.84
1.82
1.75
1.75
1.74
1.73
1.72
1.72
1.71
1.70
1.58
1.52
1.48
1.47
1.46
1.46
1.45
1.44
0.76
0.74
0.58
0.57
19 H
16, 17 H
CHCl3
impurity
2.000
1.881
3.029
0.988
2.282
1.973
1.130
3.657
2.995
1.206
3.225
2.827
f1 (ppm)

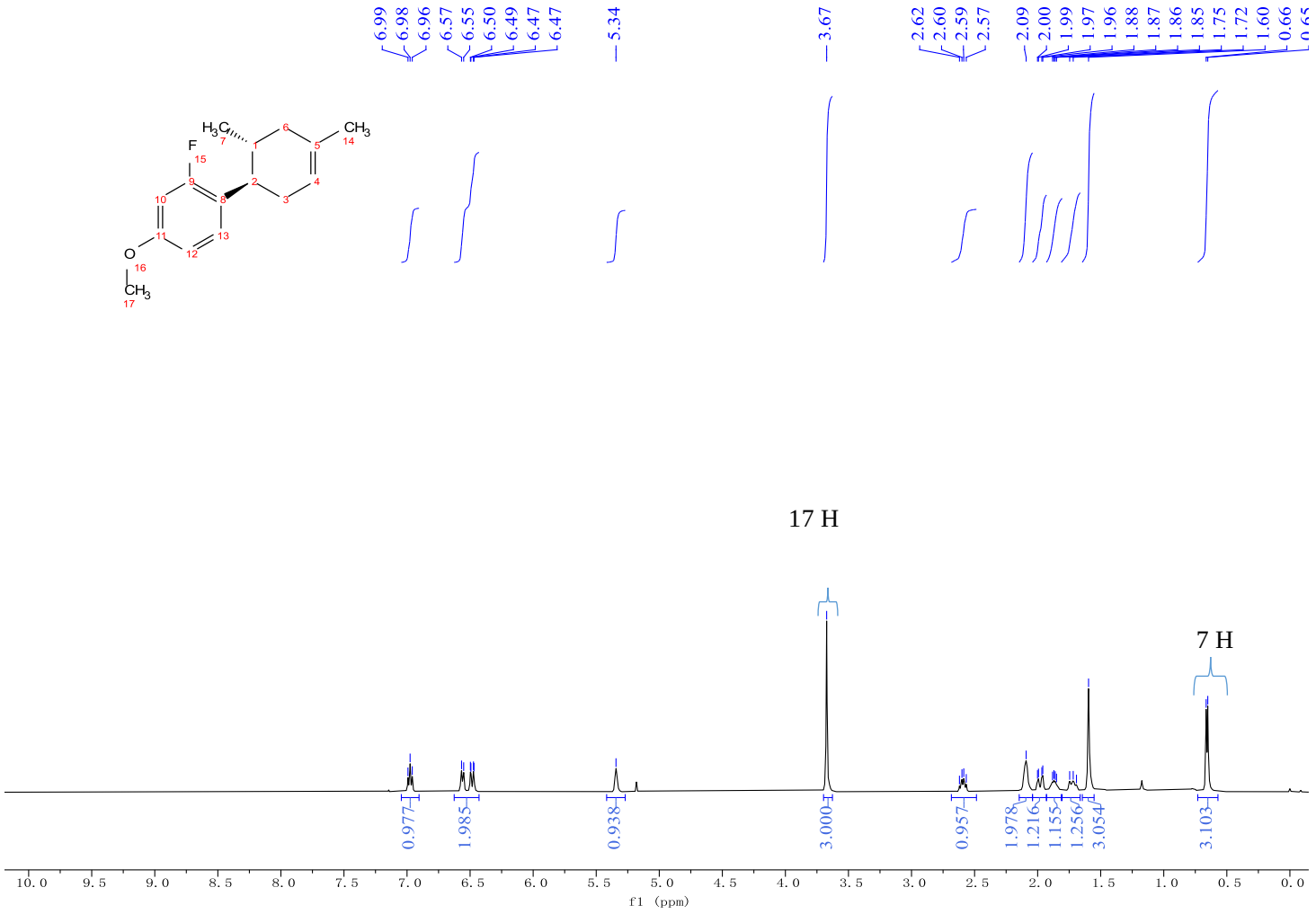

157.62
137.81
128.56
125.25
125.05
113.65
55.07
43.91
43.78
42.74
30.70
27.03
21.30
18.99
18.57
14.84
CHCl3
impurity
f1 (ppm)

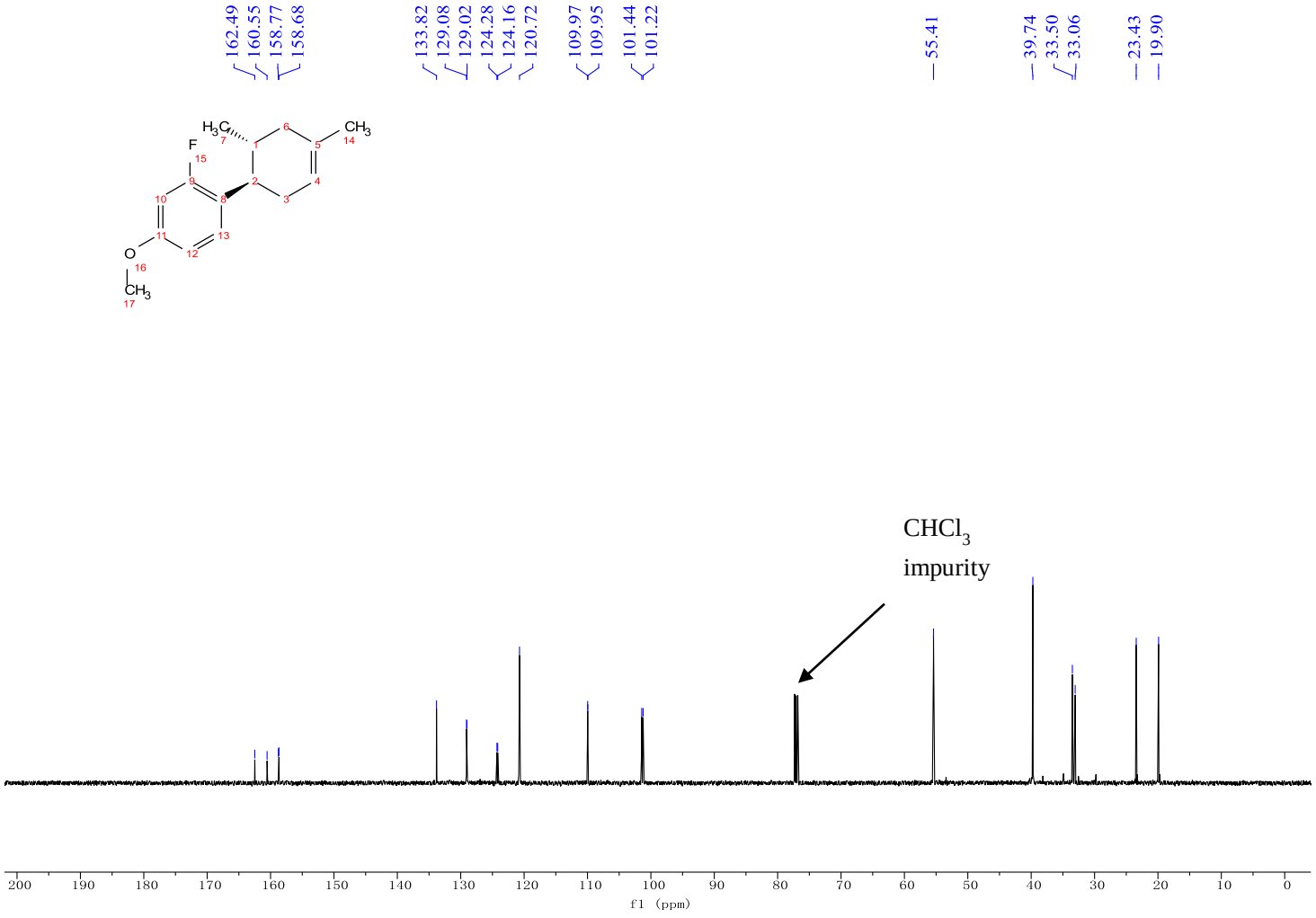

7.09 7.08 6.84 6.82 3.77 2.56 2.55 2.54 2.52 2.51 2.50 2.22 2.21 2.18 2.14 2.14 2.13 2.13 2.12 2.12 2.11 2.10 2.09 2.09 2.08 1.91 1.90 1.87 1.86 1.85 1.84 1.83 1.83 1.71 1.56 1.56 1.55 1.54 1.54 1.53 0.84 0.83 0.67 0.66
18 H
14, 15 H
CHCl3
impurity
2.000 1.998 3.231 1.091 1.061 1.192 3.101 3.159 1.436 3.211 3.061
f1 (ppm)


157.62
137.83
133.84
128.59
120.51
113.63
55.08
43.46
43.12
36.21
29.09
27.07
23.65
21.23
14.80
CHCl3
impurity
f1 (ppm)


6.99
6.97
6.96
6.57
6.55
6.49
6.49
6.47
6.47
3.67
2.66
2.65
2.64
2.63
2.61
2.60
2.11
1.99
1.96
1.84
1.83
1.82
1.81
1.77
1.74
1.60
1.55
1.52
0.65
18 H
7 H
0.915
1.942
3.000
0.851
0.973
2.282
2.137
6.621
2.823
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
f1 (ppm)


162.42
160.48
158.68
158.60
128.99
128.93
125.29
125.19
124.22
124.09
109.91
109.89
101.37
101.16
55.33
41.46
40.48
39.91
33.32
19.64
18.70
18.60
CHCl3
impurity
f1 (ppm)


6.99
6.98
6.96
6.57
6.55
6.50
6.49
6.47
6.47
5.34
3.67
2.62
2.60
2.59
2.57
2.09
2.00
1.99
1.97
1.96
1.88
1.87
1.86
1.85
1.75
1.72
1.60
0.66
0.65
17 H
7 H
0.977
1.985
0.938
3.000
0.957
1.978
1.216
1.155
1.256
3.054
3.103
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
f1 (ppm)


162.49
160.55
158.77
158.68
133.82
129.08
129.02
124.28
124.16
120.72
109.97
109.95
101.44
101.22
55.41
39.74
33.50
33.06
23.43
19.90
CHCl3
impurity
f1 (ppm)

S5. References

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