Electronic Supplementary Material (ESI) for Green Chemistry.

Supporting Information

Visible-light-driven [3 + 2] cyclization of phenols with indoles and olefins using recyclable Ag_3PO_4 nanoparticles

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Contents:

S1. Experimental details

Instruments. The emission spectra of all the LEDs and light intensity irradiated on the reaction vial were reported previously.[1] Proton (^{1}H) and carbon (^{13}C) NMR spectra were recorded on a Bruker AV-500 spectrometer operating at 500 MHz for proton and 126 MHz for carbon nuclei using $CDCl_3$ as solvent, respectively. Chemical shifts are expressed as parts per million (δ, ppm) and are referenced to 7.26 ($CDCl_3$) for ^{1}H NMR and 77.00 ($CDCl_3$) for ^{13}C NMR. Proton signal data uses the following abbreviations: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet and *J* = coupling constant. High Resolution Mass Spectrometry was performed on a Bruker Apex II mass instrument under the conditions of electrospray ionization (ESI) in both positive and negative mode. 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. 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.

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) was 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. 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 and phenols were purchased from Sigma-Aldrich Pharmaceutical Group Co. Ltd. and used without purification.

All reactions were carried out in oven-dried glassware under air with magnetic stirring. Solvents were dried by passage through an activated alumina column under argon. Liquids and solutions were transferred via syringe. All reactions were monitored by thin-layer chromatography (TLC) with E. Merck silica gel 60 pre-coated plates (0.25 mm). Silica gel (particle size 0.032 - 0.063 mm) purchased from SiliCycle was used for flash chromatography.

Synthesis of *N*-Acetyl indoles. To a solution of *N*-H indole (5.0 mmol) in dry dichloromethane (30 mL) were added tetrabutylammonium hydrogen sulfate (0.5 mmol) and sodium hydroxide (25.0 mmol). The resulting solution was stirred for 15 minutes and acetyl chloride (15.0 mmol) was added dropwise over 10 minutes. The mixture was vigorously stirred for 2 hours and quenched by addition of water. The aqueous layer was extracted with dichloromethane. Organic layers were washed with brine, and dried over Na_2SO_4. Flash column chromatography purification (hexane / EtOAc : 15/1) led to *N*-acetyl-indole.[2]

General procedure for the photocatalytic reactions. The reactions were carried out in 10-mL Pyrex vials. Olefin, phenol and Ag_3PO_4 were dispersed in a solvent in the vial. 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.

S2. Additional results and discussion

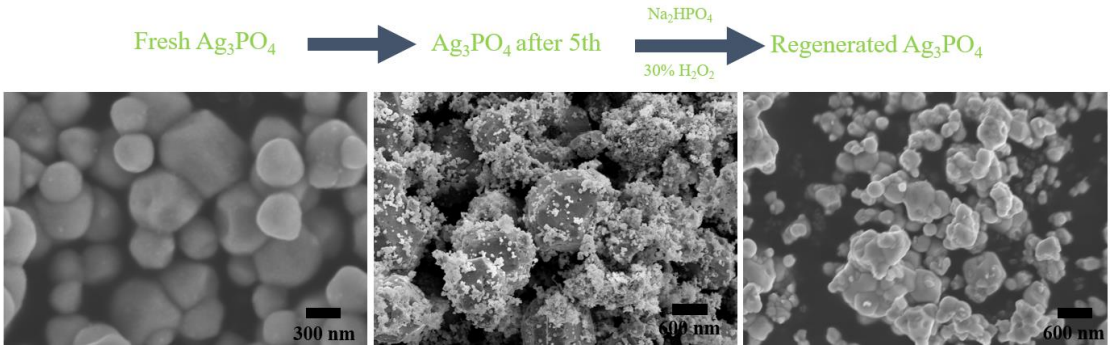


Figure S1. SEM images of fresh, recycled, and regenerated Ag_3PO_4.

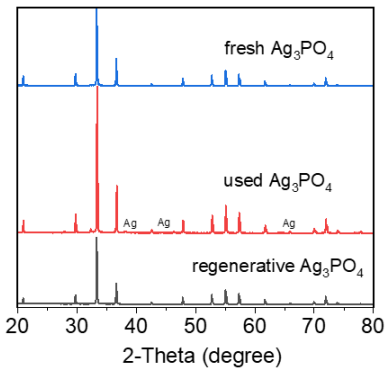


Figure S2. XRD of fresh, recycled, and regenerated Ag_3PO_4.

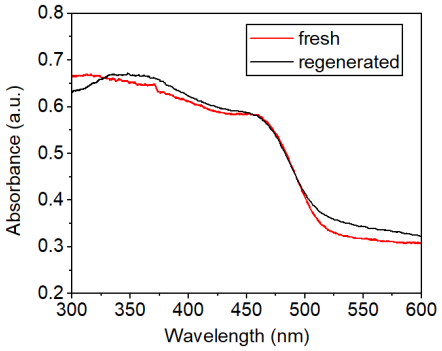


Figure S3. Uv-vis spectrum of fresh, and regenerated Ag_3PO_4.

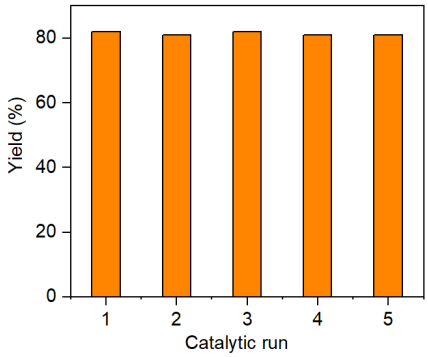


Figure S4. Reuse of Ag_3PO_4 in the [3 + 2] cycloaddition.

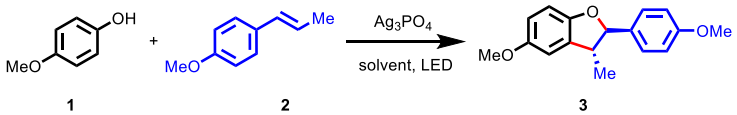
Table S1. Additional optimization of the reaction conditions.[a]

1a 2a 3a

entry	change from standard conditions	Yield (%)
1	no change	82
2	Acetone	n.r.
3	Dimethyl sulfoxide	n.r.
4	Tetrahydrofuran	n.r.
5	Et_2O	n.r.
6	CF_3Ph	<5
7	Toluene	n.r.
8	$AgNO_3$	<5
9	AgF	<5
10	Ag_2CO_3	18

[a] standard condition: **1a**, 0.2 mmol; **2a**, 0.4 mmol; Ag_3PO_4, 0.4 mmol; DCM/HFIP (1:3) mixed solvent, 2 mL; 420 nm blue LED; 0 °C; 4 h.

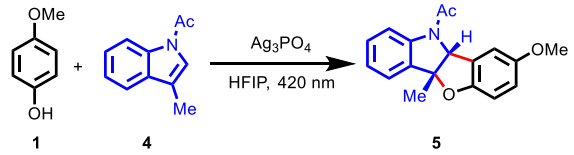
Table S2. Comparison of the performance of Ag_3PO_4 with various catalysts in the cycloadditions.[3]



1 2 3

catalyst	**yield**	**conditions**	**catalyst reuse**	**gram-scale reaction**	**ref**
Ag_3PO_4	0.2 mmol, 82%	420 nm LED, HFIP/DCM, 0 °C	5 runs, > 80%	10.2 g, 75%	this study
$Ru(bpz)_3^{2+}$	0.2 mmol, 93%	$(NH_4)_2S_2O_8$ (2.0 eq), Kessil lamp, MeCN, r.t.	no	no	*Angew. Chem. Int. Ed.* **2014**, *53*, 11056–11059
covalent organic frameworks	0.24 mmol, 80%	$(NH_4)_2S_2O_8$, (2.0 eq), white LED, MeCN, r.t.	no	no	*J. Am. Chem. Soc.* **2023,** *145*, 5074−5082

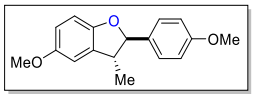
$Na_2S_2O_8$	0.15 mmol, 82%	$Na_2S_2O_8$ (3.0 eq), MeCN, 50 °C	no	no	*Synthesis* **2015**, *47*, 2731–2737



1 4 5

catalyst	yield	conditions	catalyst reuse	gram-scale reaction	ref
$FeCl_3$ (1.1 eq)	52%	DDQ (1.5 eq), DCM, r.t.	no	no	*Angew. Chem. Int. Ed.* **2014,** *53*, 11881 –11885

S3. Analysis data of the products

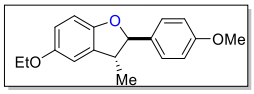


82% yield; White Oil; R_f = 0.4 (*n*-hexane : EA = 10:1); flash chromatography: *n*-hexane : EA = 15:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.6 Hz, 2H), 6.92 (d, *J* = 8.7 Hz, 2H), 6.81 – 6.65 (m, 3H), 5.08 (d, *J* = 9.1 Hz, 1H), 3.80 (d, *J* = 16.1 Hz, 6H), 3.42 (p, *J* = 6.9 Hz, 1H), 1.39 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 159.59, 154.36, 153.22, 133.08, 132.64, 127.62, 113.96, 112.82, 110.04, 109.30, 92.56, 56.01, 55.28, 45.64, 17.54.

HRMS (ESI) m/z calcd for $[C_{17}H_{19}O_3]^+$ $[M+H]^+$: 271.1329, found 271.1327.

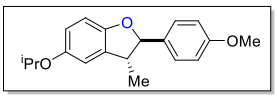


73% yield; White Oil; R_f = 0.4 (*n*-hexane : EA = 10:1); flash chromatography: *n*-hexane : EA = 15:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.31 (m, 2H), 6.92 – 6.82 (m, 2H), 6.79 – 6.65 (m, 3H), 5.05 (d, *J* = 9.1 Hz, 1H), 3.96 (qd, *J* = 7.0, 2.3 Hz, 2H), 3.77 (s, 3H), 3.38 (ddt, *J* = 9.0, 6.8, 1.0 Hz, 1H), 1.40 – 1.29 (m, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 159.54, 153.61, 153.15, 132.96, 132.66, 127.52, 113.89, 113.62, 110.81, 109.19, 92.44, 64.23, 55.14, 45.57, 17.45, 14.91.

HRMS (ESI) m/z calcd for $[C_{18}H_{21}O_3]^+$ $[M+H]^+$: 285.1485, found 285.1483.

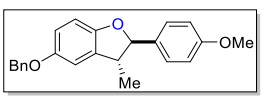


67% yield; White Oil; R_f = 0.5 (*n*-hexane : EA = 10:1); flash chromatography: *n*-hexane : EA = 15:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 – 7.32 (m, 2H), 6.92 – 6.82 (m, 2H), 6.75 – 6.66 (m, 3H), 5.05 (d, *J* = 9.1 Hz, 1H), 4.39 (p, *J* = 6.1 Hz, 1H), 3.78 (s, 3H), 3.38 (dd, *J* = 9.0, 6.7 Hz, 1H), 1.35 (d, *J* = 6.8 Hz, 3H), 1.30 (d, *J* = 6.2 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 159.58, 153.37, 152.41, 132.98, 132.71, 127.55, 115.87, 113.93, 112.85, 109.23, 92.51, 71.28, 55.19, 45.59, 22.14, 22.12, 17.47.

HRMS (ESI) m/z calcd for $[C_{19}H_{23}O_3]^+$ $[M+H]^+$: 299.1642, found 299.1640.



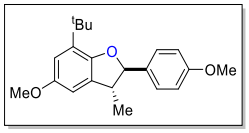
53% yield; White Oil; R_f = 0.2 (*n*-hexane : EA = 10:1); flash chromatography: *n*-hexane : EA = 12:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 (d, *J* = 7.3 Hz, 2H), 7.33 (td, *J* = 15.4, 7.2 Hz, 5H), 6.89 (d, *J* = 8.7 Hz, 2H), 6.76 (dt, *J* = 18.0, 7.9 Hz, 3H), 5.06 (d, *J* = 9.1 Hz, 1H), 4.99 (s, 2H), 3.77 (s, 3H), 3.39 (p, *J* = 6.8 Hz, 1H), 1.35 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 159.59, 153.57, 153.47, 137.36, 133.09,

132.64, 128.45, 127.78, 127.56, 127.44, 114.03, 113.95, 111.28, 109.26, 92.54, 71.03, 55.20, 45.58, 17.51.

HRMS (ESI) m/z calcd for $[C_{23}H_{23}O_3]^+$ $[M+H]^+$: 347.1642, found 347.1638.

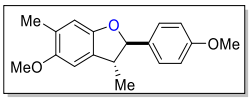


80% yield; White Oil; R_f = 0.4 (*n*-hexane : EA = 10:1); flash chromatography: *n*-hexane : EA = 10:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35 (d, *J* = 8.7 Hz, 2H), 6.90 (d, *J* = 8.7 Hz, 2H), 6.71 (dd, *J* = 2.7, 0.9 Hz, 1H), 6.55 (dd, *J* = 2.7, 1.1 Hz, 1H), 5.07 (d, *J* = 9.3 Hz, 1H), 3.78 (s, 3H), 3.76 (s, 3H), 3.29 (ddt, *J* = 9.1, 6.9, 1.0 Hz, 1H), 1.40 – 1.34 (m, 12H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 159.51, 154.17, 151.28, 133.84, 133.72, 132.93, 127.79, 127.31, 114.02, 111.64, 106.29, 91.81, 56.00, 55.32, 45.93, 34.36, 29.35, 17.76.

HRMS (ESI) m/z calcd for $[C_{21}H_{27}O_3]^+$ $[M+H]^+$: 327.1955, found 327.1947.

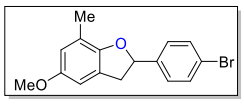


52% yield; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1); flash chromatography: *n*-hexane : EA = 10:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 – 7.29 (m, 2H), 6.93 – 6.87 (m, 2H), 6.65 (d, *J* = 3.5 Hz, 2H), 5.04 (d, *J* = 8.8 Hz, 1H), 3.79 (d, *J* = 1.5 Hz, 6H), 3.39 (p, *J* = 7.1 Hz, 1H), 2.20 (s, 3H), 1.37 (d, *J* = 6.9 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 159.64, 152.88, 152.49, 133.09, 129.42, 127.60, 126.61, 114.03, 111.54, 106.81, 92.51, 56.38, 55.31, 45.89, 18.04, 16.52.

HRMS (ESI) m/z calcd for $[C_{18}H_{21}O_3]^+$ $[M+H]^+$: 285.1485, found 285.1483.

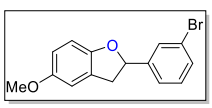


81% yield; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1); flash chromatography: *n*-hexane : EA = 10:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.51 – 7.43 (m, 2H), 7.30 – 7.18 (m, 2H), 6.57 (dd, *J* = 19.7, 2.6 Hz, 2H), 5.66 (dd, *J* = 9.4, 8.0 Hz, 1H), 3.74 (s, 3H), 3.58 (dd, *J* = 15.6, 9.4 Hz, 1H), 3.10 (dd, *J* = 15.6, 8.0 Hz, 1H), 2.24 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 154.32, 152.06, 141.48, 131.67, 127.40, 126.02, 121.66, 119.81, 114.83, 108.20, 82.90, 56.00, 39.20, 15.52.

HRMS (ESI) m/z calcd for $[C_{16}H_{16}BrO_2]^+$ $[M+H]^+$: 319.0328, found 319.0192.

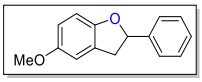


66% yield; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1); flash chromatography: *n*-hexane : EA = 10:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.54 (s, 1H), 7.41 (d, *J* = 7.9 Hz, 1H), 7.30 (d, *J* = 7.7 Hz, 1H), 7.24 – 7.17 (m, 1H), 6.79 – 6.74 (m, 2H), 6.70 (dd, *J* = 8.6, 2.6 Hz, 1H), 5.67 (t, *J* = 8.7 Hz, 1H), 3.75 (s, 3H), 3.58 (dd, *J* = 15.7, 9.4 Hz, 1H), 3.13 (dd, *J* = 15.7, 8.0 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 154.53, 153.56, 144.53, 131.01, 130.26, 128.79, 127.03, 124.31, 122.76, 113.23, 111.26, 109.34, 83.21, 56.06, 38.90.

HRMS (ESI) m/z calcd for $[C_{15}H_{14}BrO_2]^+$ $[M+H]^+$: 305.0172, found 305.0166.

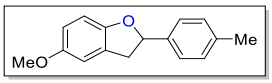


63% yield; White Oil; R_f = 0.5 (*n*-hexane : EA = 10:1); flash chromatography: *n*-hexane : EA = 20:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 – 7.32 (m, 4H), 7.31 – 7.23 (m, 2H), 6.81 – 6.73 (m, 2H), 6.69 (dt, *J* = 8.7, 1.7 Hz, 1H), 5.71 (dd, *J* = 9.3, 8.3 Hz, 1H), 3.74 (s, 3H), 3.63 – 3.54 (m, 1H), 3.17 (ddt, *J* = 15.6, 8.1, 1.1 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 154.39, 153.86, 142.09, 128.67, 128.02, 127.55, 125.81, 113.10, 111.28, 109.25, 84.27, 56.07, 38.92.

HRMS (ESI) m/z calcd for $[C_{15}H_{15}O_2]^+$ $[M+H]^+$: 227.1067, found 227.1061.

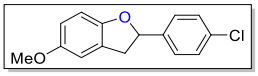


65% yield; White Oil; R_f = 0.4 (*n*-hexane : EA = 10:1); flash chromatography: *n*-hexane : EA = 15:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28 (d, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 6.79 – 6.72 (m, 2H), 6.68 (dd, *J* = 8.6, 2.6 Hz, 1H), 5.68 (t, *J* = 8.8 Hz, 1H), 3.74 (s, 3H), 3.54 (dd, *J* = 15.7, 9.3 Hz, 1H), 3.17 (dd, *J* = 15.7, 8.3 Hz, 1H), 2.33 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 154.22, 153.79, 138.94, 137.68, 129.22, 127.60, 125.76, 112.95, 111.17, 109.12, 84.20, 55.97, 38.77, 21.09.

HRMS (ESI) m/z calcd for $[C_{16}H_{17}O_2]^+$ $[M+H]^+$: 241.1223, found 241.1219.

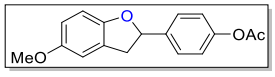


90% yield; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1); flash chromatography: *n*-hexane : EA = 15:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 (s, 4H), 6.78 – 6.72 (m, 2H), 6.68 (dd, *J* = 8.7, 2.5 Hz, 1H), 5.66 (dd, *J* = 9.3, 8.1 Hz, 1H), 3.73 (s, 3H), 3.56 (dd, *J* = 15.6, 9.4 Hz, 1H), 3.10 (dd, *J* = 15.6, 8.0 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 154.52, 153.64, 140.70, 133.71, 128.81, 127.20, 127.17, 113.21, 111.29, 109.30, 83.43, 56.04, 38.91.

HRMS (ESI) m/z calcd for $[C_{15}H_{14}ClO_2]^+$ $[M+H]^+$: 261.0677, found 261.0674.

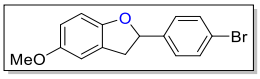


85% yield; White Oil; R_f = 0.2 (*n*-hexane : EA = 10:1); flash chromatography: *n*-hexane : EA = 9:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 (d, *J* = 8.5 Hz, 2H), 7.07 (d, *J* = 8.6 Hz, 2H), 6.79 – 6.73 (m, 2H), 6.68 (dd, *J* = 8.6, 2.6 Hz, 1H), 5.69 (t, *J* = 8.7 Hz, 1H), 3.73 (s, 3H), 3.56 (dd, *J* = 15.7, 9.3 Hz, 1H), 3.15 (dd, *J* = 15.7, 8.0 Hz, 1H), 2.26 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 169.25, 154.30, 153.55, 150.21, 139.55, 127.23, 126.77, 121.61, 113.01, 111.12, 109.10, 83.49, 55.89, 38.72, 20.95.

HRMS (ESI) m/z calcd for $[C_{17}H_{17}O_4]^+$ $[M+H]^+$: 285.1121, found 285.1116.

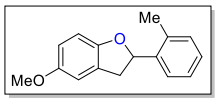


89% yield; White Oil; R_f = 0.4 (*n*-hexane : EA = 10:1); flash chromatography: *n*-hexane : EA = 10:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 – 7.42 (m, 2H), 7.25 (d, *J* = 8.4 Hz, 2H), 6.76 (d, *J* = 8.5 Hz, 2H), 6.69 (dd, *J* = 8.9, 2.4 Hz, 1H), 5.70 – 5.57 (m, 1H), 3.74 (s, 3H), 3.57 (dd, *J* = 15.5, 9.4 Hz, 1H), 3.10 (dd, *J* = 15.5, 8.0 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 154.40, 153.51, 141.11, 131.64, 127.36, 127.05, 121.70, 113.10, 111.17, 109.19, 83.33, 55.95, 38.76.

HRMS (ESI) m/z calcd for $[C_{15}H_{14}BrO_2]^+$ $[M+H]^+$: 305.0172, found 305.0166.

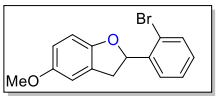


74% yield; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1); flash chromatography: *n*-hexane : EA = 15:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34 (dd, *J* = 6.0, 2.8 Hz, 1H), 7.12 – 7.01 (m, 3H), 6.73 – 6.55 (m, 3H), 5.78 (dd, *J* = 9.5, 8.1 Hz, 1H), 3.62 (s, 3H), 3.48 (dd, *J* = 15.6, 9.5 Hz, 1H), 2.93 (ddt, *J* = 15.6, 8.1, 1.1 Hz, 1H), 2.22 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 154.23, 153.77, 140.22, 134.08, 130.40, 127.49, 127.26, 126.15, 124.88, 112.96, 112.94, 111.19, 109.08, 81.63, 55.88, 37.77, 19.10, 19.08.

HRMS (ESI) m/z calcd for $[C_{16}H_{17}O_2]^+$ $[M+H]^+$: 241.1223, found 241.1218.

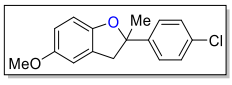


57% yield; White Oil; R_f = 0.2 (*n*-hexane : EA = 10:1); flash chromatography: *n*-hexane : EA = 9:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52 (ddd, *J* = 20.9, 7.9, 1.2 Hz, 2H), 7.30 – 7.23 (m, 1H), 7.12 (td, *J* = 7.7, 1.6 Hz, 1H), 6.82 (d, *J* = 8.6 Hz, 1H), 6.76 – 6.65 (m, 2H), 5.98 (dd, *J* = 9.6, 7.2 Hz, 1H), 3.80 (dd, *J* = 15.9, 9.6 Hz, 1H), 3.73 (s, 3H), 2.99 (dd, *J* = 15.9, 7.1 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 154.57, 153.78, 141.94, 132.76, 129.06, 127.69, 126.96, 126.66, 120.89, 113.17, 111.38, 109.29, 82.93, 56.05, 38.20.

HRMS (ESI) m/z calcd for $[C_{15}H_{14}BrO_2]^+$ $[M+H]^+$: 305.0172, found 305.0171.

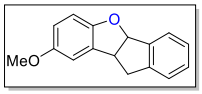


47% yield; White Oil; R_f = 0.4 (*n*-hexane : EA = 10:1); flash chromatography: *n*-hexane : EA = 10:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 (d, *J* = 8.6 Hz, 2H), 7.31 – 7.24 (m,

2H), 6.77 (d, *J* = 8.5 Hz, 1H), 6.73 – 6.65 (m, 2H), 3.71 (s, 3H), 3.32 (s, 2H), 1.71 (s, 3H).

HRMS (ESI) m/z calcd for $[C_{16}H_{16}ClO_2]^+$ $[M+H]^+$: 275.0833, found 275.0794.

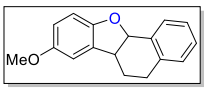


69% yield; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1); flash chromatography: *n*-hexane : EA = 10:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 – 7.49 (m, 1H), 7.29 – 7.15 (m, 3H), 6.79 (d, *J* = 2.4 Hz, 1H), 6.66 – 6.57 (m, 2H), 6.15 (d, *J* = 8.2 Hz, 1H), 4.24 (tt, *J* = 8.4, 1.6 Hz, 1H), 3.72 (s, 3H), 3.48 (dd, *J* = 16.4, 8.6 Hz, 1H), 3.18 (dd, *J* = 16.4, 2.2 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 154.38, 153.00, 142.12, 140.88, 131.79, 129.16, 127.19, 125.85, 125.02, 113.52, 110.87, 109.75, 90.68, 56.00, 45.23, 38.92.

HRMS (ESI) m/z calcd for $[C_{16}H_{15}O_2]^+$ $[M+H]^+$: 239.1067, found 239.1062.

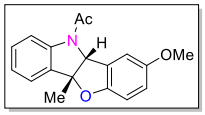


71% yield; White Oil; R_f = 0.3 (*n*-hexane : EA = 10:1); flash chromatography: *n*-hexane : EA = 10:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52 (dd, *J* = 7.4, 1.7 Hz, 1H), 7.25 (dtd, *J* = 17.8, 7.3, 1.6 Hz, 2H), 7.13 (dd, *J* = 7.3, 1.5 Hz, 1H), 6.82 (d, *J* = 2.5 Hz, 1H), 6.72 – 6.60 (m, 2H), 5.63 (d, *J* = 8.4 Hz, 1H), 3.76 (s, 3H), 3.63 (td, *J* = 8.2, 5.2 Hz, 1H), 2.72 (ddd, *J* = 15.7, 7.5, 3.9 Hz, 1H), 2.63 (ddd, *J* = 15.6, 8.8, 4.0 Hz, 1H), 2.04 (dddd, *J* = 13.0, 7.4, 5.5, 4.0 Hz, 1H), 1.81 (dtd, *J* = 12.8, 8.5, 4.0 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 154.25, 153.46, 138.73, 133.53, 132.37, 130.10, 128.36, 128.17, 126.60, 112.99, 110.77, 109.38, 81.94, 56.00, 41.60, 27.83, 27.58.

HRMS (ESI) m/z calcd for $[C_{17}H_{17}O_2]^+$ $[M+H]^+$: 253.1223, found 253.1216.

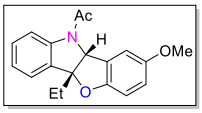


60% yield; White Oil; R_f = 0.3 (*n*-hexane : EA = 3:1); flash chromatography: *n*-hexane : EA = 3:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52 (d, *J* = 7.5 Hz, 1H), 7.40 – 7.27 (m, 2H), 7.17 – 7.10 (m, 2H), 6.83 – 6.68 (m, 1H), 6.65 (d, *J* = 8.8 Hz, 1H), 5.96 (s, 1H), 3.74 (s, 3H), 2.50 (s, 3H), 1.83 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 168.72, 154.37, 152.86, 140.35, 135.02, 129.92, 126.93, 125.01, 124.85, 124.01, 123.44, 118.08, 116.90, 116.12, 114.47, 112.47, 111.02, 110.18, 92.79, 90.59, 71.84, 55.98, 25.01, 24.78.

HRMS (ESI) m/z calcd for $[C_{18}H_{18}NO_3]^+$ $[M+H]^+$: 296.1281, found 296.1289.

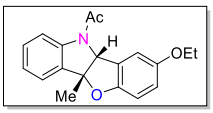


49% yield; White Oil; R_f = 0.4 (*n*-hexane : EA = 3:1); flash chromatography: *n*-hexane : EA = 3:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49 (d, *J* = 7.5 Hz, 1H), 7.34 – 7.25 (m, 2H), 7.18 – 7.08 (m, 2H), 6.74 (dd, *J* = 8.7, 2.8 Hz, 1H), 6.66 (d, *J* = 8.7 Hz, 1H), 6.07 (s, 1H), 3.74 (s, 3H), 2.55 (d, *J* = 43.3 Hz, 3H), 2.22 (dt, *J* = 14.6, 7.3 Hz, 1H), 2.07 (dt, *J* = 14.2, 7.1 Hz, 1H), 0.94 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 168.74, 154.39, 153.24, 140.85, 134.35, 129.99, 127.19, 125.29, 124.06, 116.93, 114.60, 112.30, 110.13, 93.93, 69.17, 56.06, 30.61, 25.03, 7.79.

HRMS (ESI) m/z calcd for $[C_{19}H_{20}NO_3]^+$ $[M+H]^+$: 310.1438, found 310.1434.

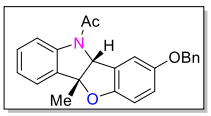


61% yield; White Oil; R_f = 0.3 (*n*-hexane : EA = 3:1); flash chromatography: *n*-hexane : EA = 3:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52 (d, *J* = 7.5 Hz, 1H), 7.37 – 7.27 (m, 2H), 7.19 – 7.09 (m, 2H), 6.74 (dd, *J* = 8.7, 2.8 Hz, 1H), 6.64 (d, *J* = 8.7 Hz, 1H), 5.96 (s, 1H), 3.95 (q, *J* = 6.9 Hz, 2H), 2.54 (d, *J* = 39.5 Hz, 3H), 1.84 (d, *J* = 7.7 Hz, 3H), 1.36 (q, *J* = 6.9, 6.4 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 168.75, 153.77, 152.88, 140.45, 135.13, 129.99, 127.00, 125.10, 124.06, 117.59, 114.53, 113.49, 110.20, 92.82, 90.62, 72.48, 71.92, 64.40, 25.11, 24.87, 14.93.

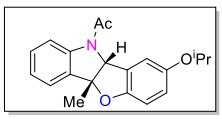
HRMS (ESI) m/z calcd for $[C_{19}H_{20}NO_3]^+$ $[M+H]^+$: 310.1438, found 310.1431.



31% yield; White Solid; R_f = 0.3 (*n*-hexane : EA = 3:1); flash chromatography: *n*-hexane : EA = 3:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53 (d, *J* = 7.5 Hz, 1H), 7.39 (dt, *J* = 20.8, 7.6 Hz, 5H), 7.32 (q, *J* = 4.5, 2.4 Hz, 2H), 7.18 – 7.11 (m, 2H), 6.82 (dd, *J* = 8.7, 2.8 Hz, 1H), 6.65 (d, *J* = 8.7 Hz, 1H), 5.97 (s, 1H), 4.98 (s, 2H), 2.52 (d, *J* = 10.6 Hz, 3H), 1.84 (s, 3H).

HRMS (ESI) m/z calcd for $[C_{24}H_{22}NO_3]^+$ $[M+H]^+$: 372.1594, found 372.1573.

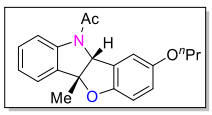


62% yield; White Oil; R_f = 0.3 (*n*-hexane : EA = 3:1); flash chromatography: *n*-hexane : EA = 3:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48 (dd, *J* = 39.5, 7.5 Hz, 1H), 7.34 – 7.27 (m, 2H), 7.18 – 7.08 (m, 2H), 6.73 (dd, *J* = 8.7, 2.8 Hz, 1H), 6.63 (d, *J* = 8.7 Hz, 1H), 5.95 (s, 1H), 4.49 – 4.29 (m, 1H), 2.53 (d, *J* = 39.6 Hz, 3H), 1.82 (s, 3H), 1.27 (d, *J* = 6.0 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 168.72, 153.05, 152.52, 140.46, 135.09, 130.00, 127.13, 125.10, 124.05, 123.54, 119.41, 118.67, 118.16, 115.85, 114.52, 114.14, 111.00, 110.12, 90.64, 77.36, 77.11, 76.85, 71.86, 71.29, 60.41, 25.12, 24.89, 22.20, 22.06, 21.08, 14.23.

HRMS (ESI) m/z calcd for $[C_{20}H_{22}NO_3]^+$ $[M+H]^+$: 324.1594, found 324.1576.

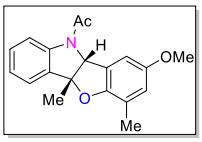


54% yield; White Solidl; R_f = 0.3 (*n*-hexane : EA = 3:1); flash chromatography: *n*-hexane : EA = 3:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48 (dd, *J* = 41.7, 7.8 Hz, 1H), 7.33 – 7.26 (m, 2H), 7.19 – 7.08 (m, 2H), 6.74 (dd, *J* = 8.8, 2.8 Hz, 1H), 6.63 (d, *J* = 8.7 Hz, 1H), 5.96 (s, 1H), 3.84 (t, *J* = 6.5 Hz, 2H), 2.54 (d, *J* = 40.6 Hz, 3H), 1.83 (d, *J* = 5.4 Hz, 3H), 1.75 (q, *J* = 7.1 Hz, 2H), 1.00 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 168.75, 154.00, 152.84, 140.46, 135.15, 129.98, 127.00, 125.09, 124.90, 124.06, 123.52, 118.16, 117.48, 116.85, 114.54, 113.51, 111.91, 111.01, 110.17, 90.61, 72.49, 71.93, 70.63, 70.46, 25.10, 24.88, 24.38, 22.71, 10.57.

HRMS (ESI) m/z calcd for $[C_{20}H_{22}NO_3]^+$ $[M+H]^+$: 324.1594, found 324.1584.

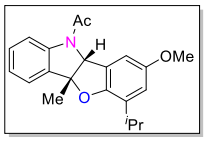


48% yield; White Oil; R_f = 0.3 (*n*-hexane : EA = 3:1); flash chromatography: *n*-hexane : EA = 3:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49 (dd, *J* = 41.8, 7.5 Hz, 1H), 7.34 – 7.24 (m, 1H), 7.17 – 7.05 (m, 2H), 6.67 – 6.53 (m, 1H), 5.96 (s, 1H), 3.72 (s, 3H), 2.53 (d, *J* = 37.8 Hz, 3H), 2.14 (d, *J* = 15.6 Hz, 3H), 1.83 (d, *J* = 8.6 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 168.73, 154.26, 151.73, 141.15, 140.44, 135.60, 134.27, 129.96, 129.87, 126.03, 125.18, 125.04, 124.85, 124.00, 123.55, 121.66, 120.72, 118.21, 118.12, 117.57, 114.54, 109.33, 107.90, 92.28, 90.09, 72.86, 72.29, 56.00, 25.30, 25.11, 25.08, 15.52.

HRMS (ESI) m/z calcd for $[C_{19}H_{20}NO_3]^+$ $[M+H]^+$: 310.1438, found 310.1439.

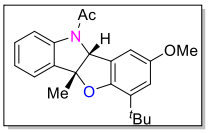


64% yield; White Oil; R_f = 0.3 (*n*-hexane : EA = 4:1); flash chromatography: *n*-hexane : EA = 4:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48 (dd, *J* = 43.6, 7.5 Hz, 1H), 7.32 – 7.23 (m, 1H), 7.11 (td, *J* = 7.8, 7.2, 4.4 Hz, 2H), 6.78 – 6.61 (m, 1H), 5.98 (s, 1H), 3.73 (s, 3H), 3.01 (dt, *J* = 13.8, 6.3 Hz, 1H), 2.53 (d, *J* = 39.3 Hz, 3H), 1.82 (d, *J* = 9.2 Hz, 3H), 1.17 (ddd, *J* = 36.1, 11.3, 6.9 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 168.74, 168.02, 154.54, 150.79, 140.37, 135.78, 134.42, 132.59, 131.74, 129.90, 129.81, 126.25, 125.30, 125.05, 124.87, 123.99, 123.41, 118.14, 114.60, 114.51, 114.00, 108.73, 107.22, 92.15, 89.97, 72.74, 72.18, 55.98, 28.58, 28.39, 25.24, 25.09, 25.07, 24.32, 22.06, 21.99, 21.86.

HRMS (ESI) m/z calcd for $[C_{21}H_{24}NO_3]^+$ $[M+H]^+$: 338.1751, found 338.1756.

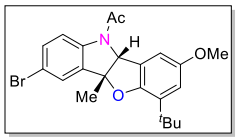


69% yield; White Solid; R_f = 0.4 (*n*-hexane : EA = 3:1); flash chromatography: *n*-hexane : EA = 3:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53 (d, *J* = 7.5 Hz, 1H), 7.31 – 7.20 (m, 1H), 7.18 – 6.99 (m, 2H), 6.73 (dd, *J* = 9.9, 3.7 Hz, 1H), 5.99 (s, 1H), 3.74 (s, 3H), 2.57 (s, 1H), 2.49 (s, 2H), 1.83 (d, *J* = 11.1 Hz, 3H), 1.30 (d, *J* = 14.5 Hz, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 168.67, 154.08, 150.91, 140.17, 135.86, 134.12, 129.64, 127.01, 124.86, 124.78, 123.93, 123.27, 118.07, 114.85, 114.58, 114.41, 108.36, 106.75, 91.78, 89.61, 72.27, 71.69, 60.29, 55.83, 34.16, 28.93, 24.94, 20.95, 14.12.

HRMS (ESI) m/z calcd for $[C_{22}H_{26}NO_3]^+$ $[M+H]^+$: 352.1907, found 352.1917.

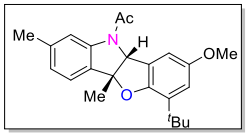


40% yield; White Solid; R_f = 0.4 (*n*-hexane : EA = 3:1); flash chromatography: *n*-hexane : EA = 3:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.70 – 7.49 (m, 1H), 7.38 (dd, *J* = 8.6, 2.1 Hz, 1H), 7.04 (dd, *J* = 36.8, 5.7 Hz, 1H), 6.82 – 6.66 (m, 1H), 5.97 (s, 1H), 3.74 (s, 3H), 2.52 (d, *J* = 44.2 Hz, 3H), 1.82 (d, *J* = 13.3 Hz, 3H), 1.30 (d, *J* = 14.9 Hz, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 168.57, 154.32, 150.82, 138.13, 134.44, 132.81, 132.60, 128.05, 126.63, 126.44, 125.65, 119.73, 116.46, 116.09, 115.16, 114.68, 108.41, 106.82, 91.32, 89.18, 72.57, 72.14, 55.94, 34.40, 34.27, 29.05, 25.01, 24.94, 24.18, 14.22.

HRMS (ESI) m/z calcd for $[C_{22}H_{25}BrNO_3]^+$ $[M+H]^+$: 430.1012, found 430.0994.

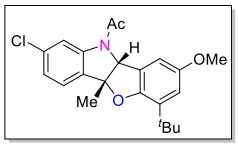


44% yield; White Solid; R_f = 0.3 (*n*-hexane : EA = 3:1); flash chromatography: *n*-hexane : EA = 3:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 (dd, *J* = 46.2, 7.7 Hz, 1H), 7.10 (d, *J* = 2.7 Hz, 1H), 6.97 – 6.87 (m, 2H), 6.72 (dd, *J* = 10.7, 3.3 Hz, 1H), 5.98 (s, 1H), 3.73 (s, 3H), 2.53 (d, *J* = 29.9 Hz, 3H), 2.32 (d, *J* = 13.7 Hz, 3H), 1.81 (d, *J* = 11.9 Hz, 3H), 1.29 (d, *J* = 14.0 Hz, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 168.76, 167.92, 154.27, 154.15, 151.13, 151.03, 141.16, 140.48, 140.07, 139.89, 135.04, 134.18, 133.29, 131.82, 127.23, 126.26, 125.76, 124.89, 124.59, 123.02, 118.65, 115.44, 114.92, 114.48, 108.45, 106.85, 91.76, 89.59, 72.66, 72.09, 55.99, 55.93, 34.39, 34.27, 29.05, 25.22, 25.10, 25.06, 24.33, 22.00, 21.78.

HRMS (ESI) m/z calcd for $[C_{23}H_{28}NO_3]^+$ $[M+H]^+$: 366.2064, found 366.2044.

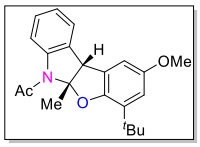


56% yield; White Oil; R_f = 0.4 (*n*-hexane : EA = 3:1); flash chromatography: *n*-hexane : EA = 3:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 (dd, *J* = 48.5, 8.1 Hz, 1H), 7.14 – 7.02 (m, 2H), 6.81 – 6.63 (m, 1H), 5.99 (s, 1H), 3.74 (s, 3H), 2.53 (d, *J* = 35.8 Hz, 3H), 1.81 (d, *J* = 12.1 Hz, 3H), 1.29 (d, *J* = 12.9 Hz, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 168.63, 168.08, 154.30, 150.86, 141.94, 141.38, 135.47, 135.23, 134.58, 134.37, 133.07, 126.71, 125.78, 125.05, 124.13, 118.48, 115.13, 114.67, 108.38, 106.80, 91.31, 89.07, 72.78, 72.32, 55.95, 34.27, 29.01, 25.07, 24.93, 24.21, 14.15.

HRMS (ESI) m/z calcd for $[C_{22}H_{25}ClNO_3]^+$ $[M+H]^+$: 386.1517, found 386.1514.

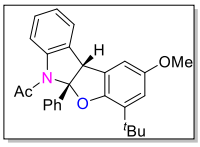


60% yield; White Oil; R_f = 0.4 (*n*-hexane : EA = 3:1); flash chromatography: *n*-hexane : EA = 3:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.34 (d, *J* = 8.2 Hz, 1H), 7.42 (ddd, *J* = 8.5, 7.5, 1.7 Hz, 1H), 7.29 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.24 – 7.18 (m, 1H), 7.14 (s, 1H), 6.81 (d, *J* = 2.5 Hz, 1H), 6.52 (d, *J* = 2.6 Hz, 1H), 3.73 (s, 3H), 2.41 (s, 3H), 1.98 (s, 3H), 1.52 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 168.33, 156.10, 152.63, 147.31, 136.27, 135.92, 130.94, 128.99, 128.73, 124.32, 122.28, 121.69, 112.74, 110.88, 98.44, 55.81, 34.44, 29.86, 24.87, 12.92.

HRMS (ESI) m/z calcd for $[C_{22}H_{26}NO_3]^+$ $[M+H]^+$: 352.1907, found 352.1893.



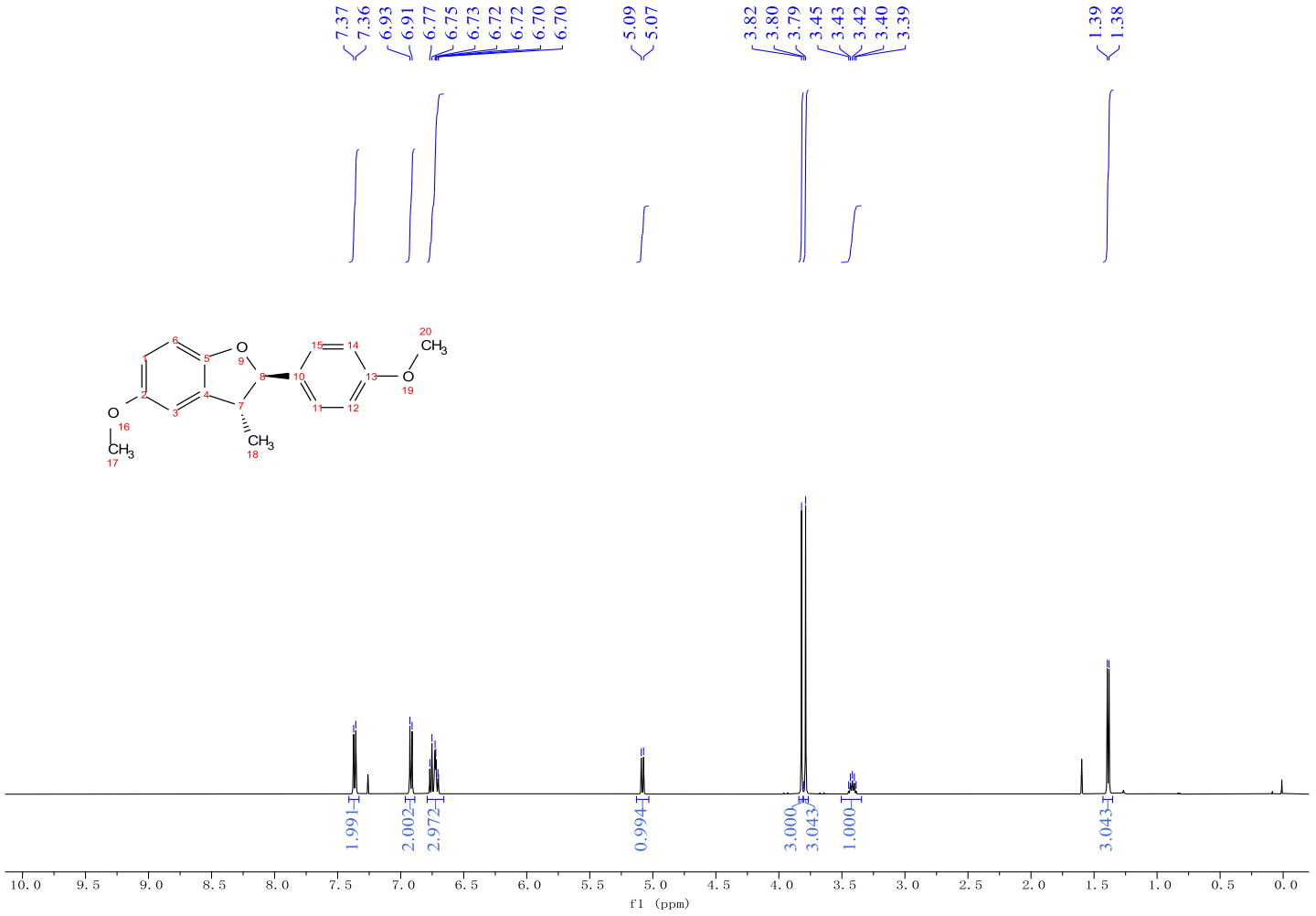
69% yield; White Oil; R_f = 0.3 (*n*-hexane : EA = 3:1); flash chromatography: *n*-hexane : EA = 3:1.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.38 (d, *J* = 8.3 Hz, 1H), 7.63 – 7.56 (m, 2H), 7.48 (ddd, *J* = 8.5, 7.5, 1.6 Hz, 1H), 7.41 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.33 (qt, *J* = 5.1, 2.1 Hz, 3H), 7.27 – 7.21 (m, 1H), 7.14 (s, 1H), 6.90 (d, *J* = 2.5 Hz, 1H), 6.56 (d, *J* = 2.5 Hz, 1H), 3.75 (s, 3H), 1.70 (s, 3H), 1.60 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 168.41, 156.44, 150.91, 147.36, 136.15, 136.03, 130.97, 130.61, 130.29, 129.15, 128.89, 128.86, 128.77, 126.42, 125.96, 124.62, 122.25, 121.91, 112.47, 98.77, 55.85, 34.50, 29.97, 24.44.

HRMS (ESI) m/z calcd for $[C_{27}H_{28}NO_3]^+$ $[M+H]^+$: 414.2064, found 414.2056.

S4. The spectra of the compounds



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154.36
153.22
133.08
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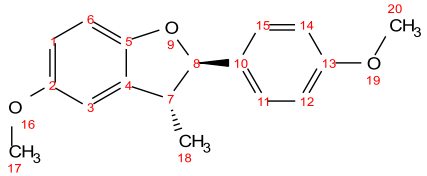
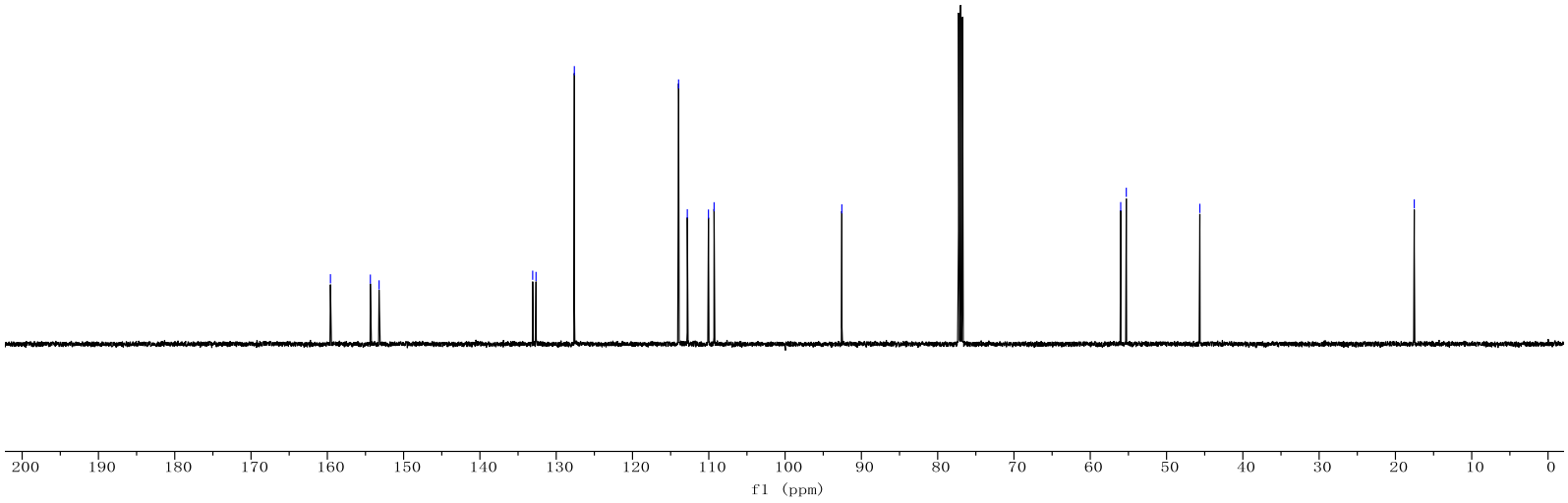
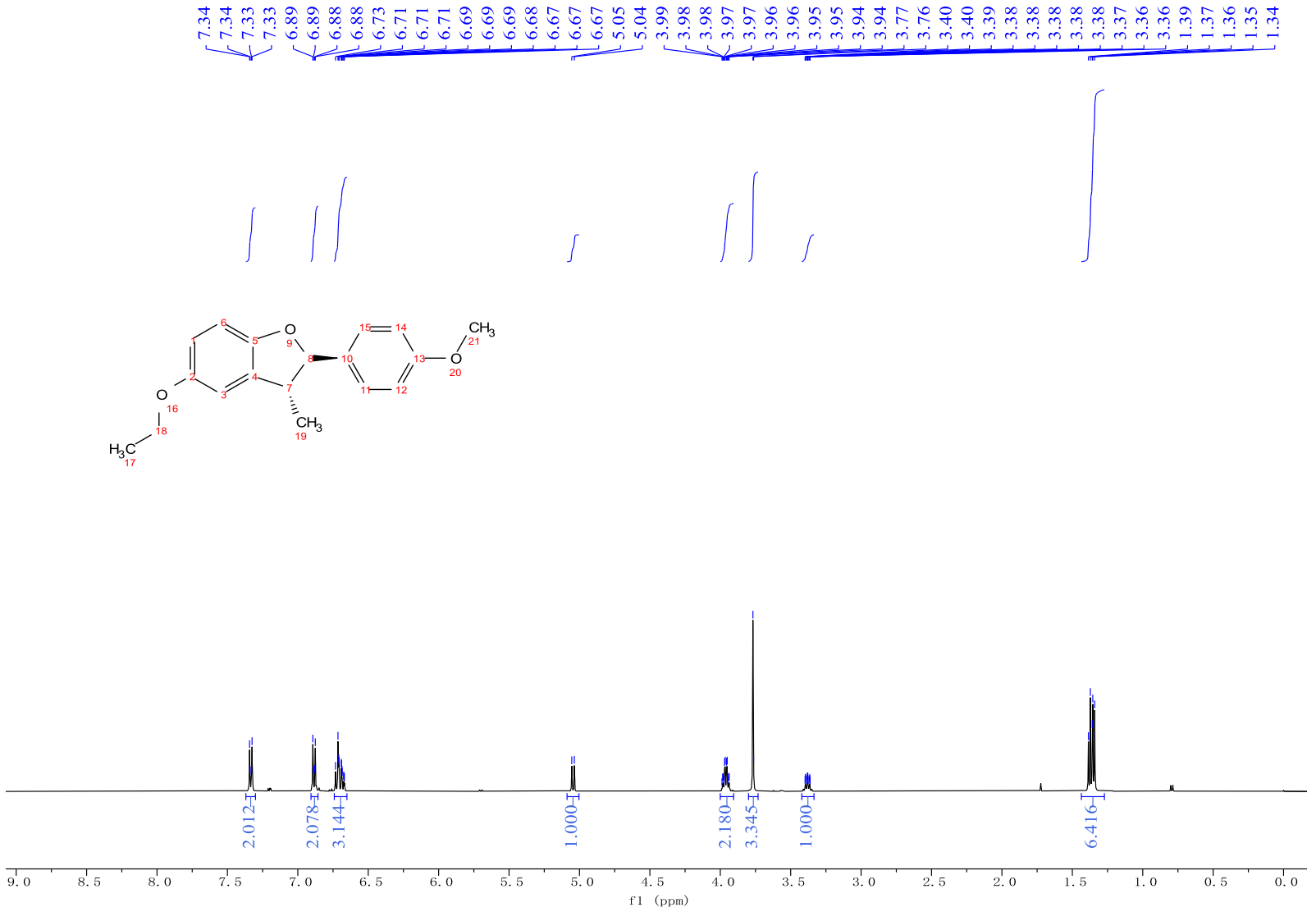
127.62
113.96
112.82
110.04
109.30
92.56
56.01
55.28
45.64
17.54



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7.33
7.33
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6.89
6.88
6.88
6.73
6.71
6.71
6.71
6.69
6.69
6.69
6.68
6.67
6.67
6.67
5.05
5.04
3.99
3.98
3.98
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3.96
3.96
3.95
3.95
3.94
3.94
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3.76
3.40
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3.39
3.38
3.38
3.38
3.38
3.38
3.37
3.36
3.36
1.39
1.37
1.36
1.35
1.34
O
9
CH3
21
O
20
O
16
H3C
17
CH3
19
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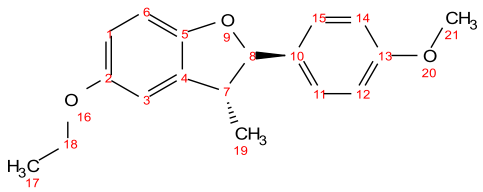
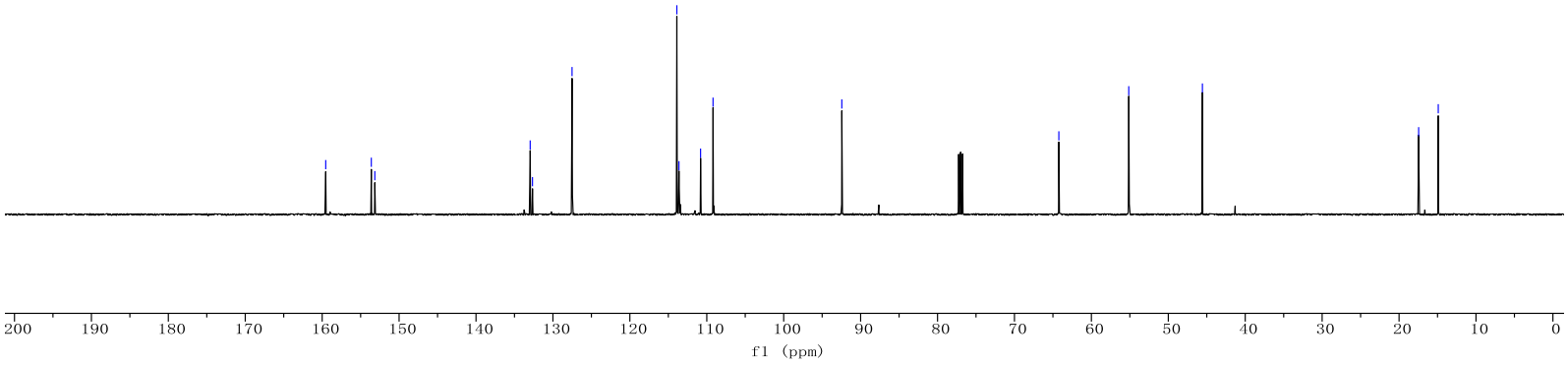
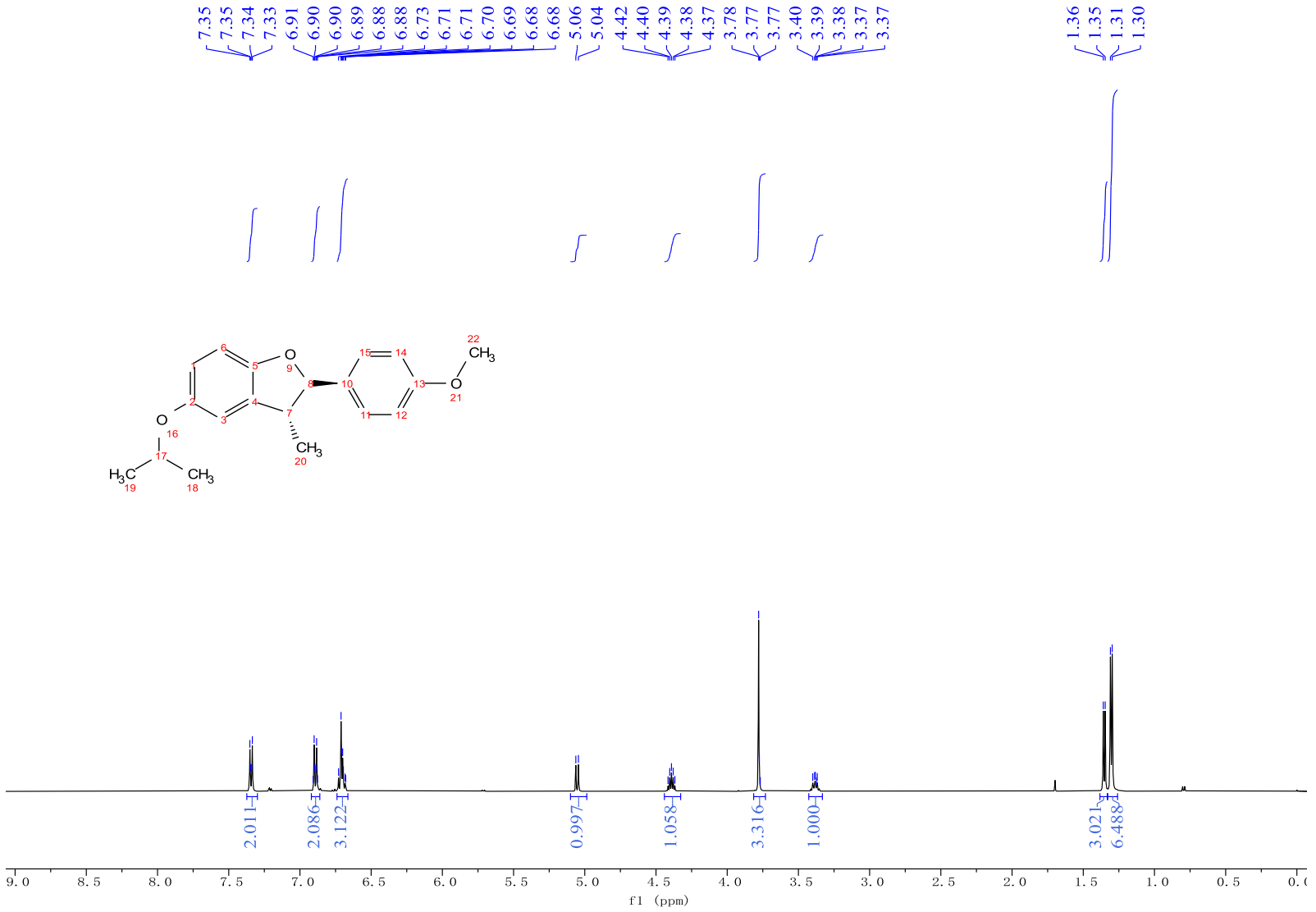
2.078
3.144
1.000
2.180
3.345
1.000
6.416
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

159.54
153.61
153.15
132.96
132.66
127.52
113.89
113.62
110.81
109.19
92.44
64.23
55.14
45.57
17.45
14.91



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5.06
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4.38
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3.39
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1.35
1.31
1.30
O
CH3
O
O
O
CH3
H3C
CH3
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2.086
3.122
0.997
1.058
3.316
1.000
3.021
6.488
f1 (ppm)

159.58
153.37
152.41
132.98
132.71
127.55
115.87
113.93
112.85
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92.51
71.28
55.19
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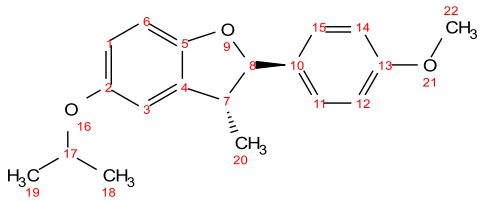
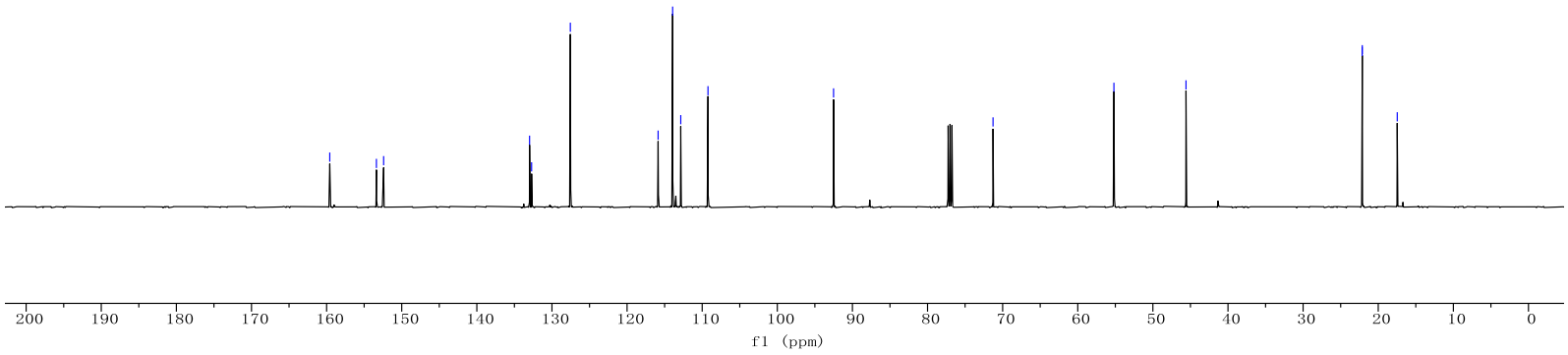
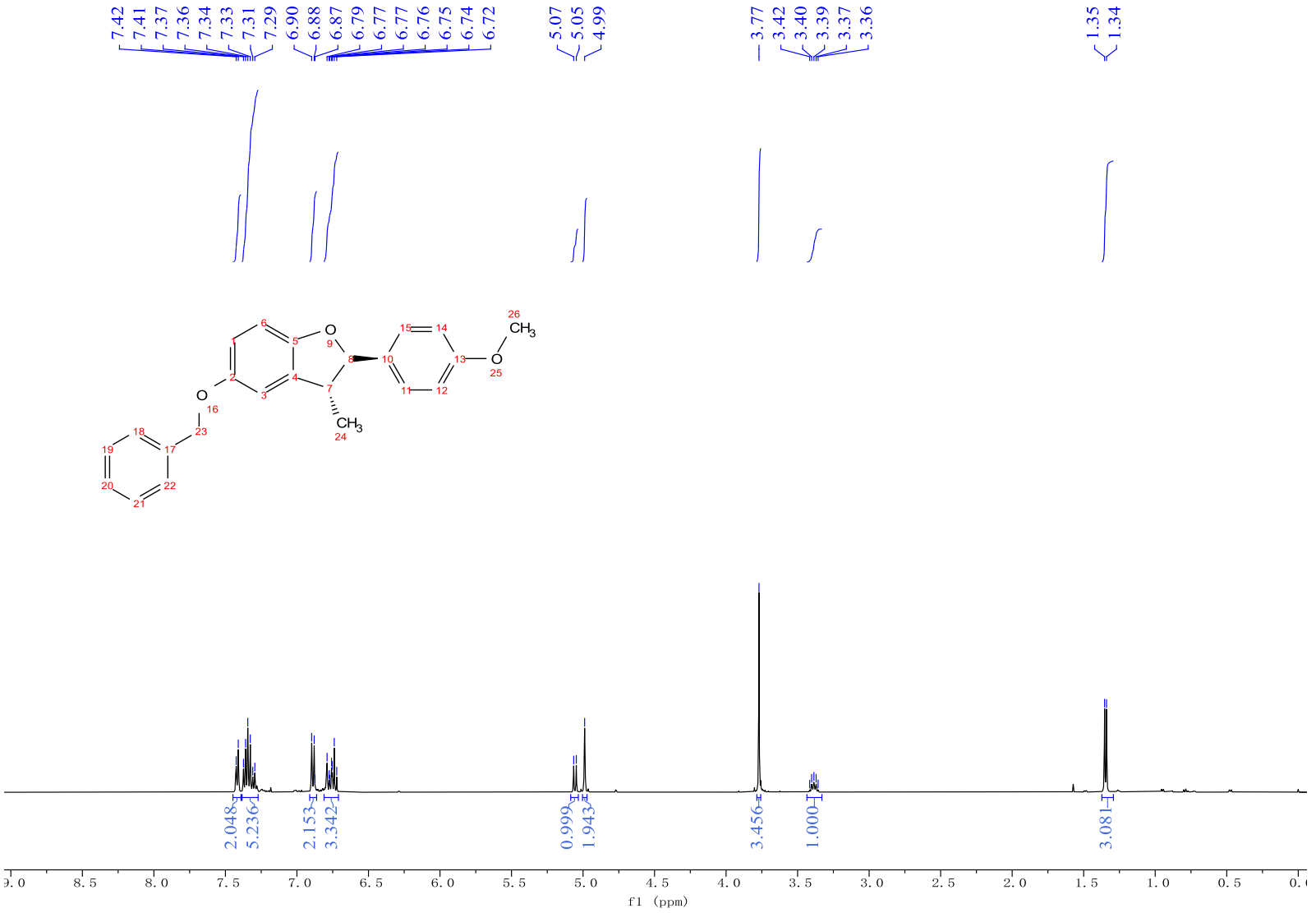
22.14
22.12
17.47



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6.76
6.75
6.74
6.72
5.07
5.05
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3.37
3.36
1.35
1.34
O
O
O
O
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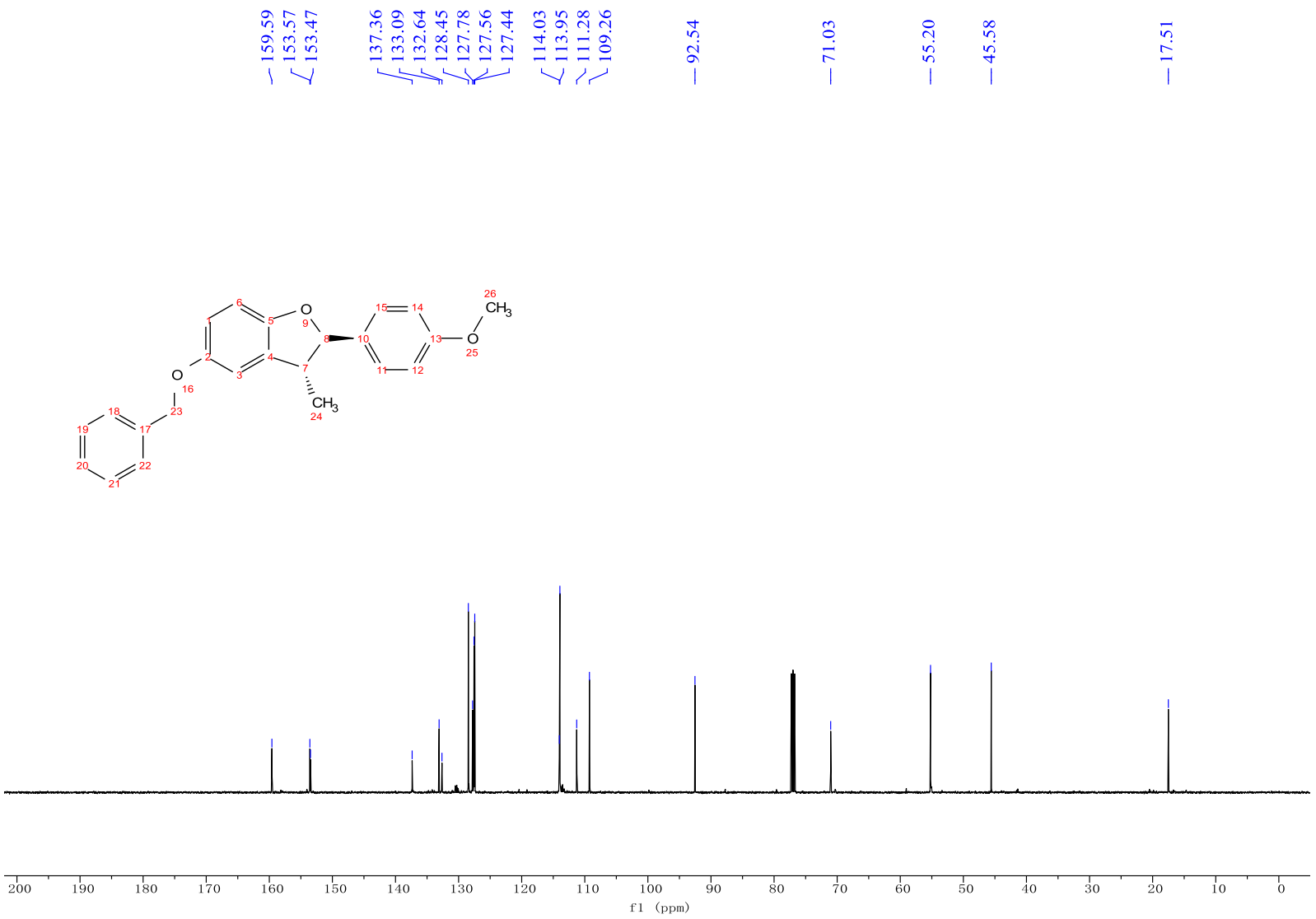
3.081
f1 (ppm)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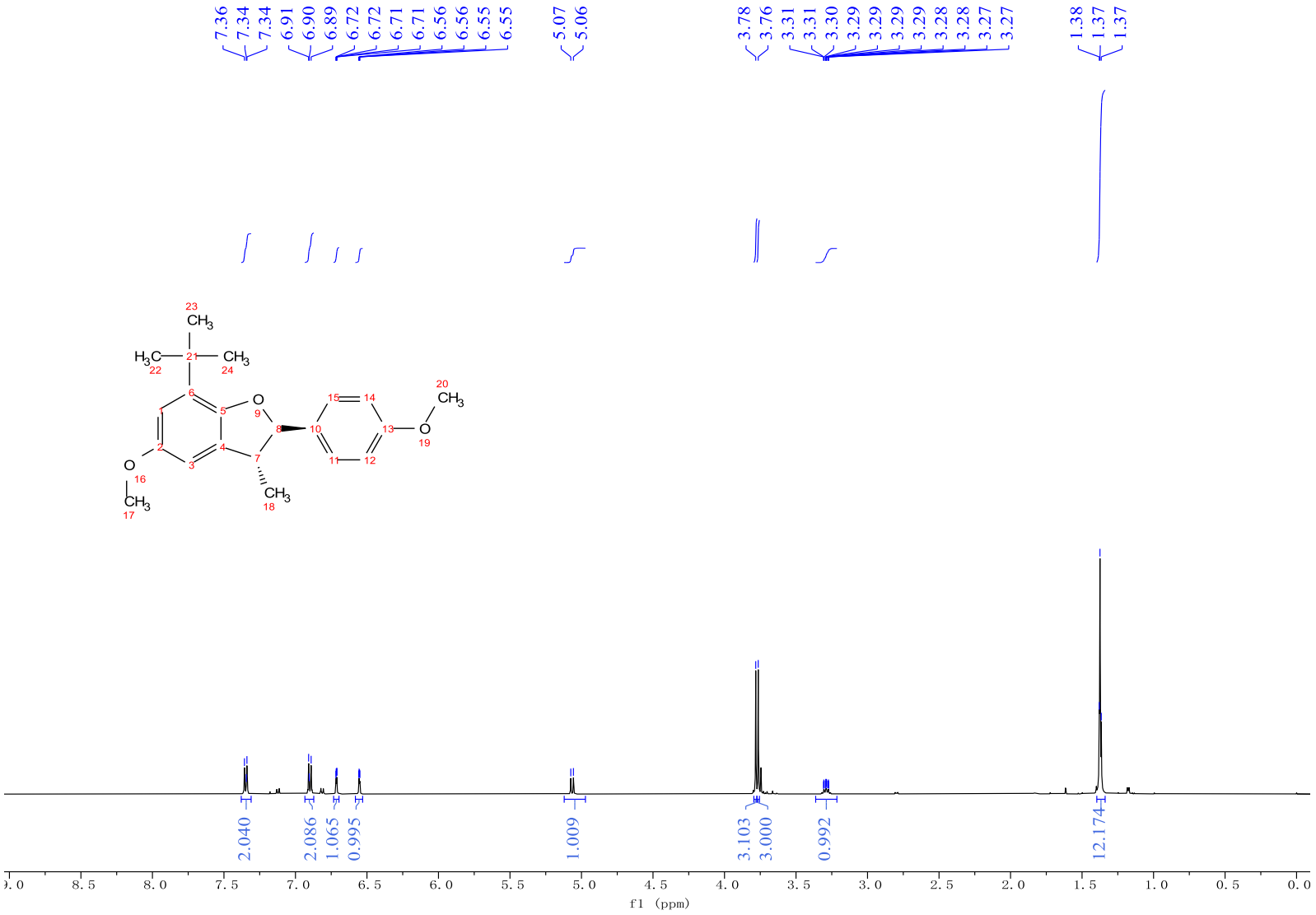
113.95
111.28
109.26
92.54
71.03
55.20
45.58
17.51
O
CH3
O
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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3.29
3.29
3.28
3.28
3.27
3.27
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1.37
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3.000
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12.174
f1 (ppm)

159.51
154.17
151.28
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133.72
132.93
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91.81
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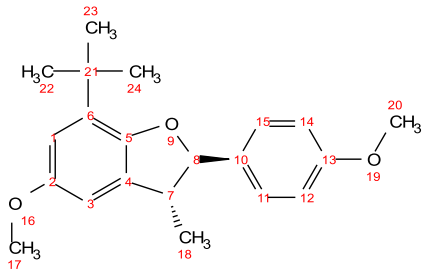
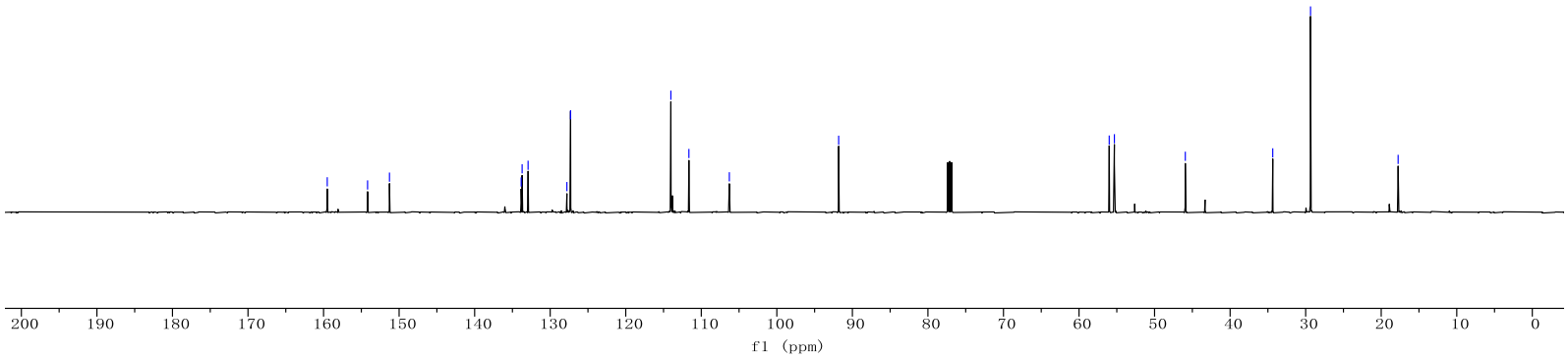
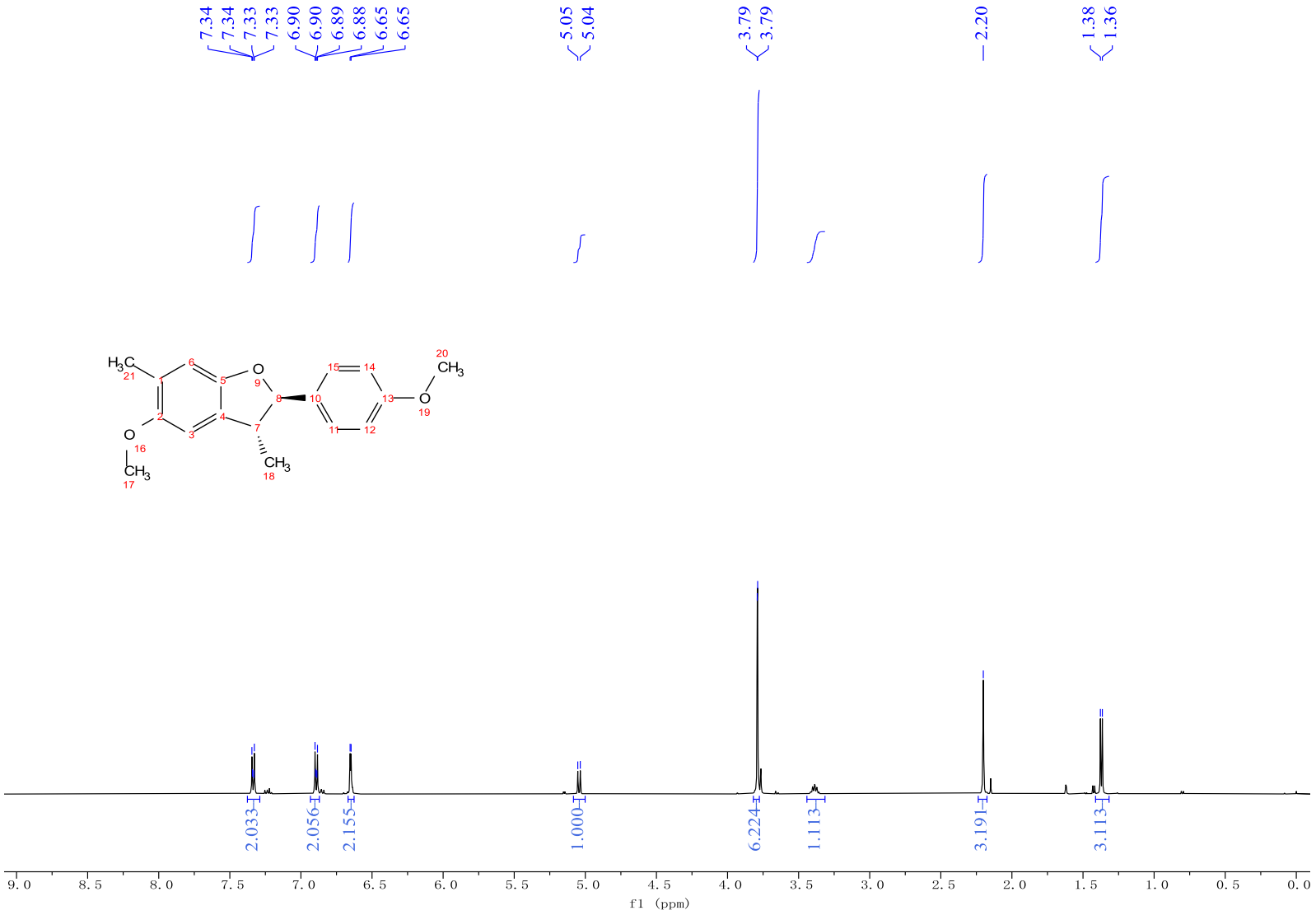
29.35
17.76



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3.79
2.20
1.38
1.36
H3C
21
6
5
O
9
15
14
20
CH3
8
10
13
O
19
2
4
3
7
11
12
O
16
CH3
17
CH3
18
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1.000
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3.113
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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)

159.64
152.88
152.49
133.09
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106.81
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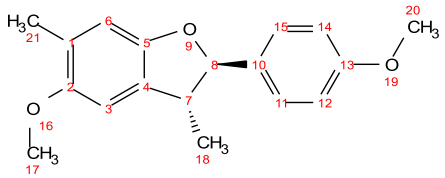
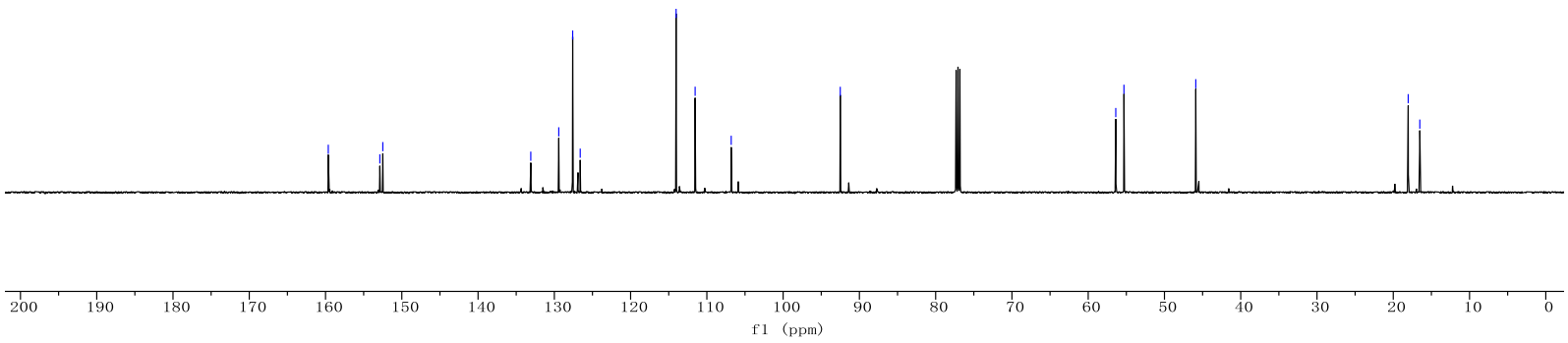
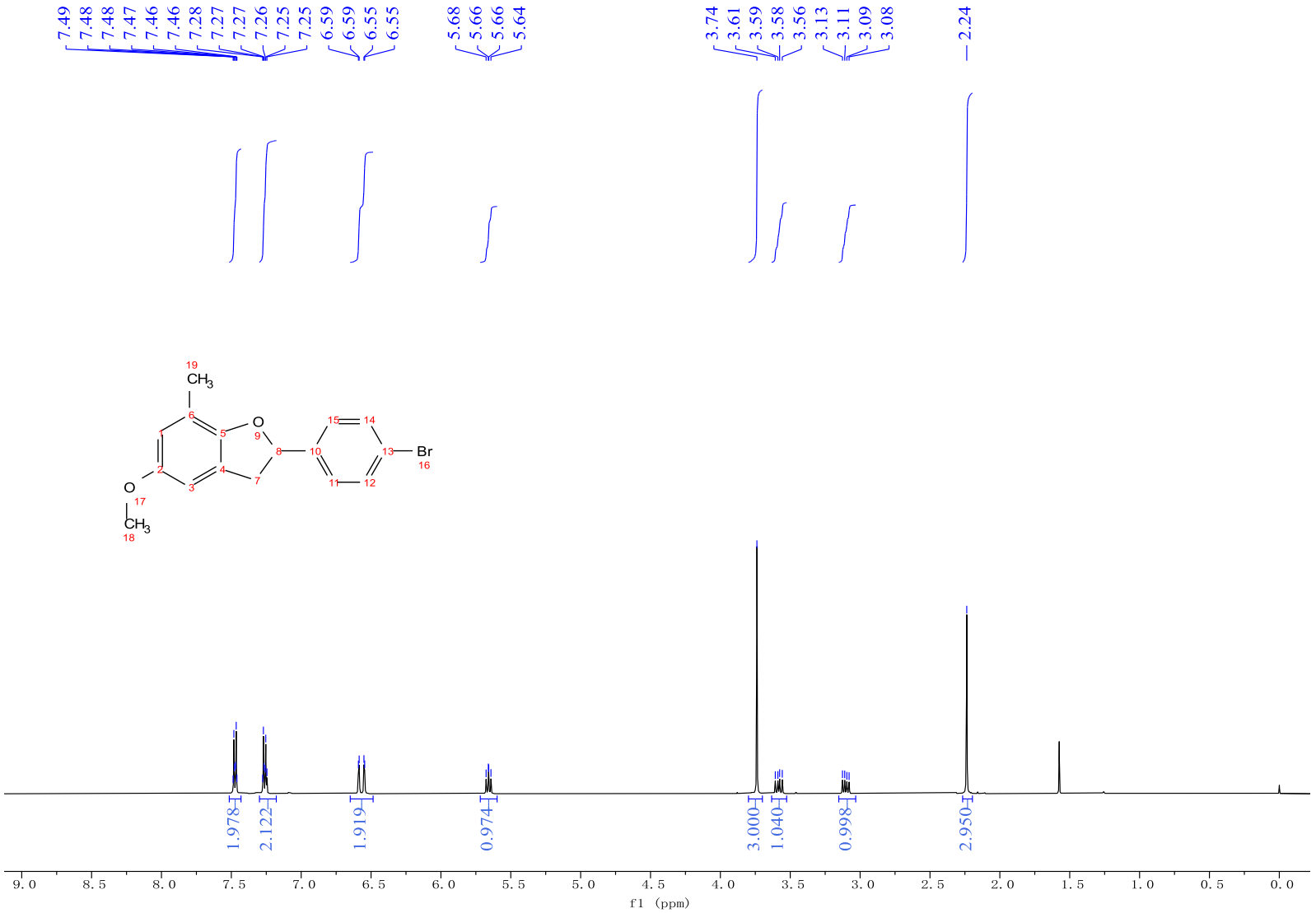
18.04
16.52



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7.26
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7.25
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5.68
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5.66
5.64
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3.61
3.59
3.58
3.56
3.13
3.11
3.09
3.08
2.24
1.978
2.122
1.919
0.974
3.000
1.040
0.998
2.950
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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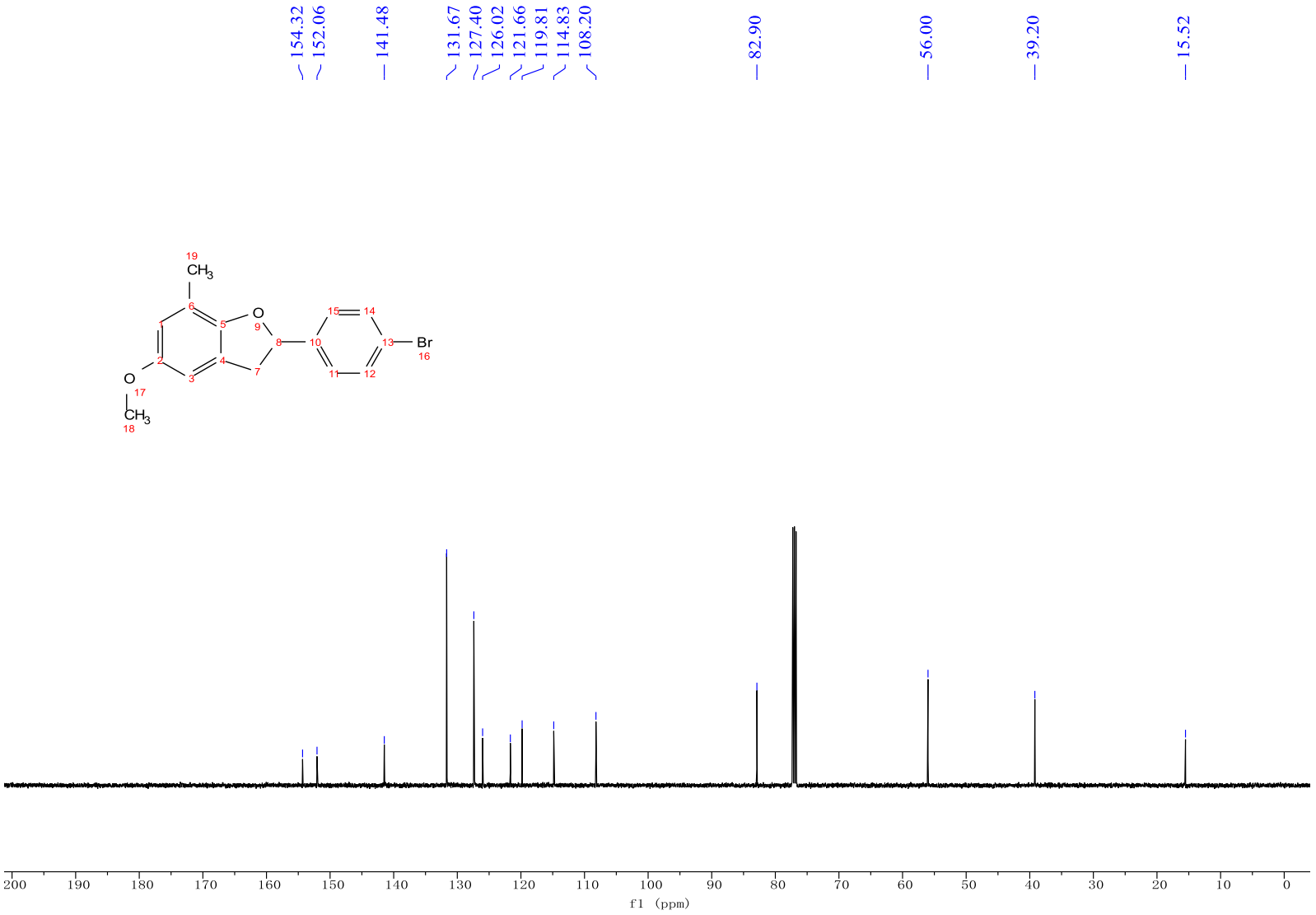
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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121.66
119.81
114.83
108.20
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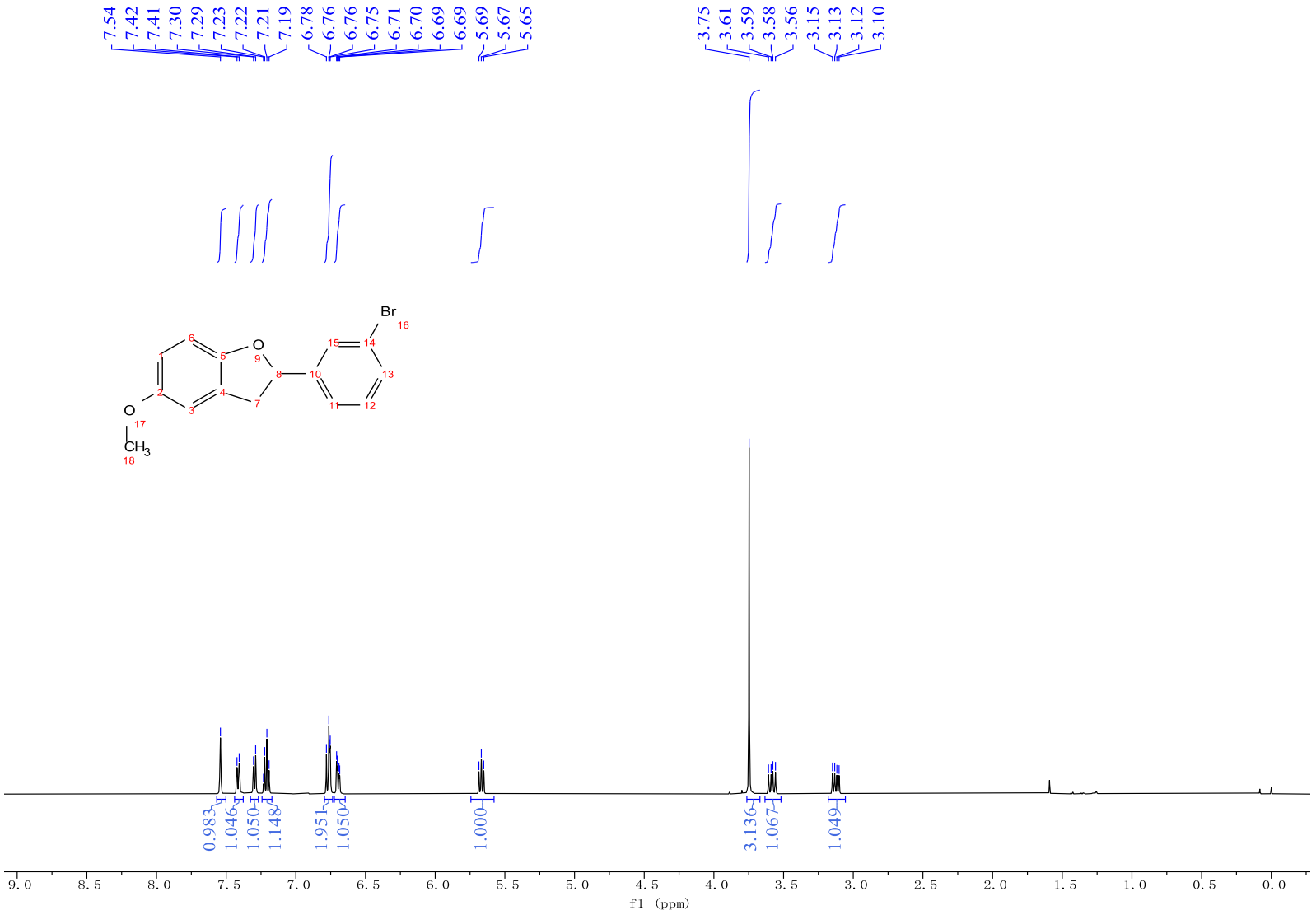
56.00
39.20
15.52
Br
f1 (ppm)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6.76
6.76
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6.69
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3.59
3.58
3.56
3.15
3.13
3.12
3.10
Br
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9
O
17
CH3
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1.050
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1.951
1.050
1.000
3.136
1.067
1.049
9.0
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8.0
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
2.5
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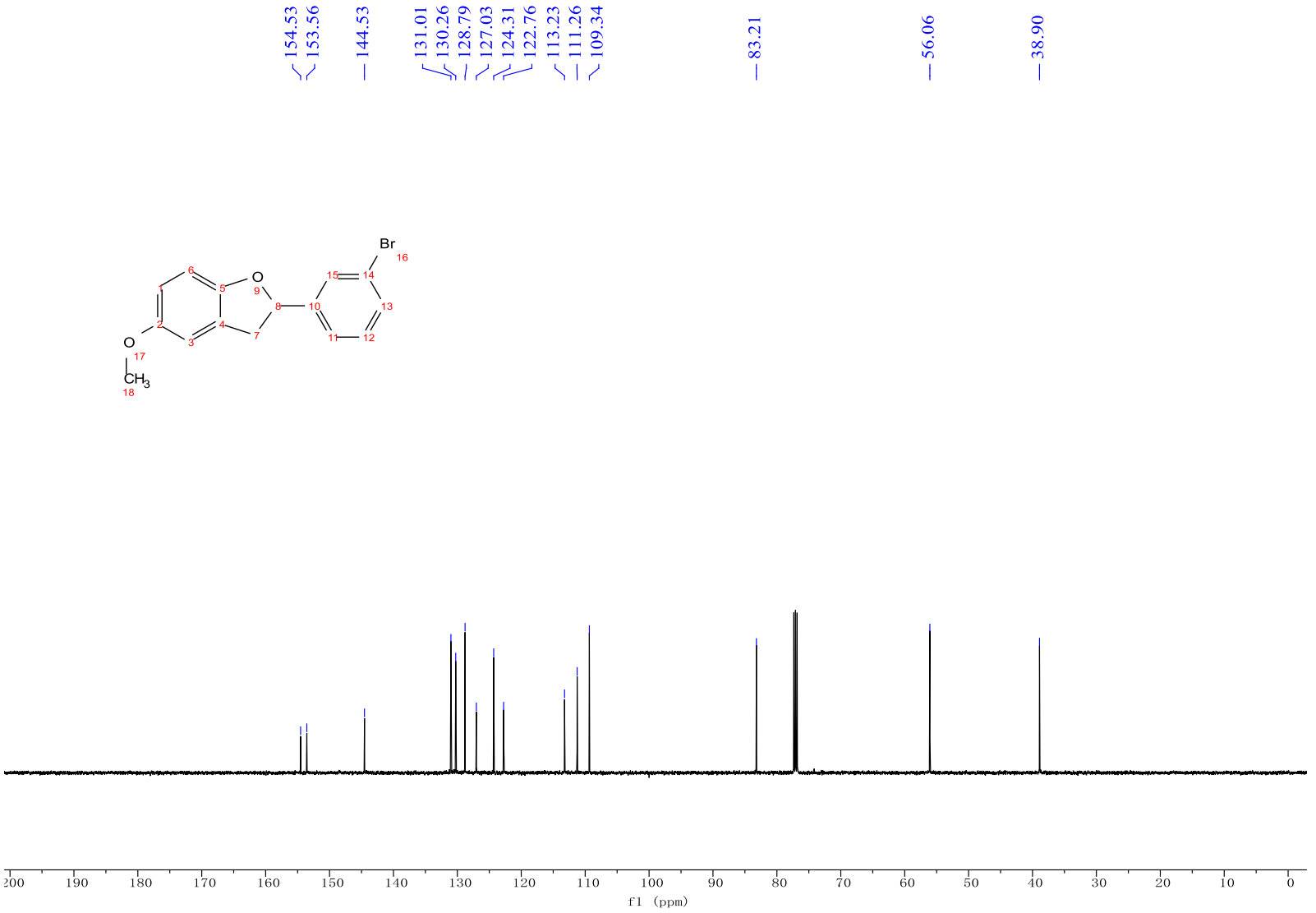
1.0
0.5
0.0
f1 (ppm)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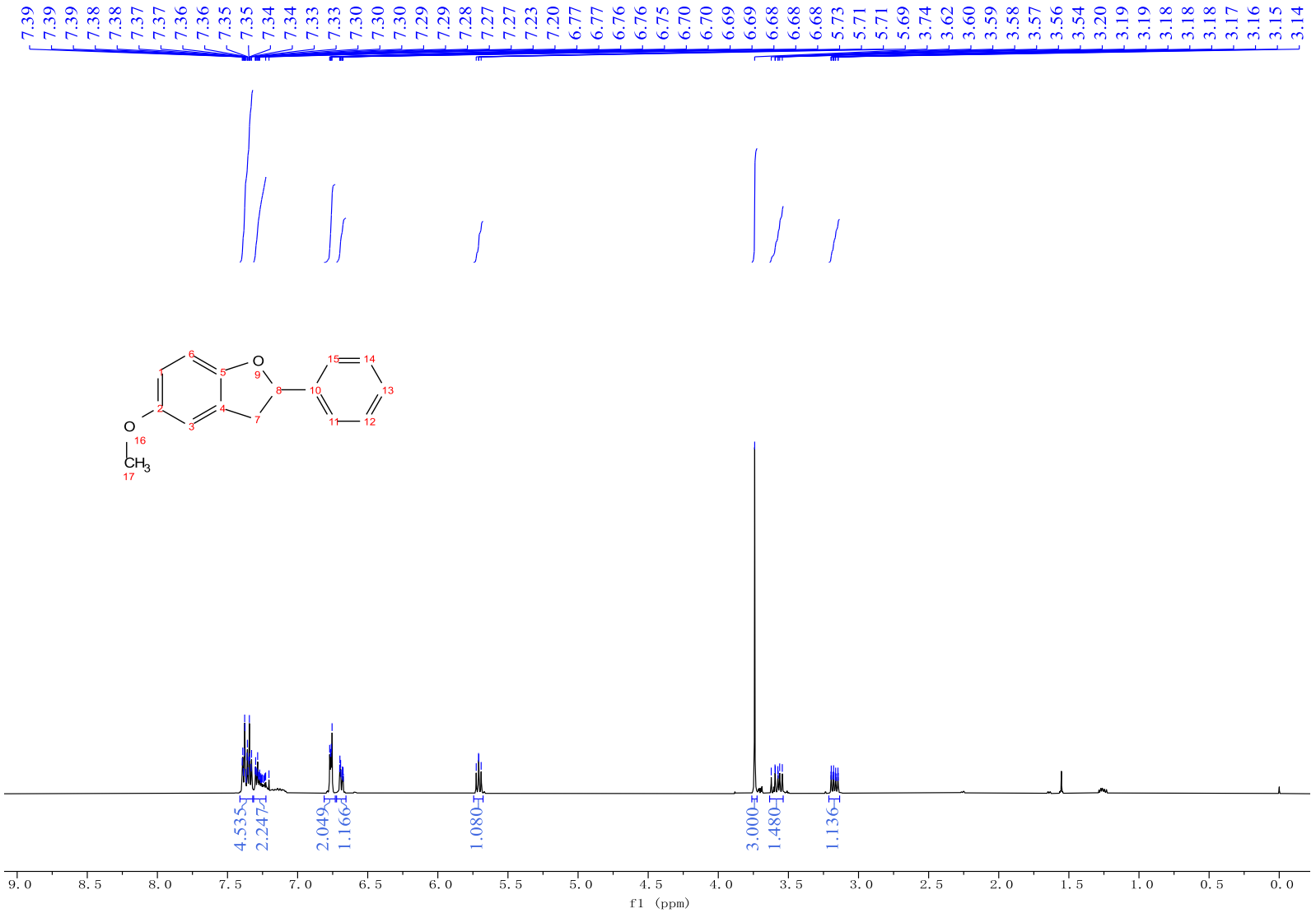
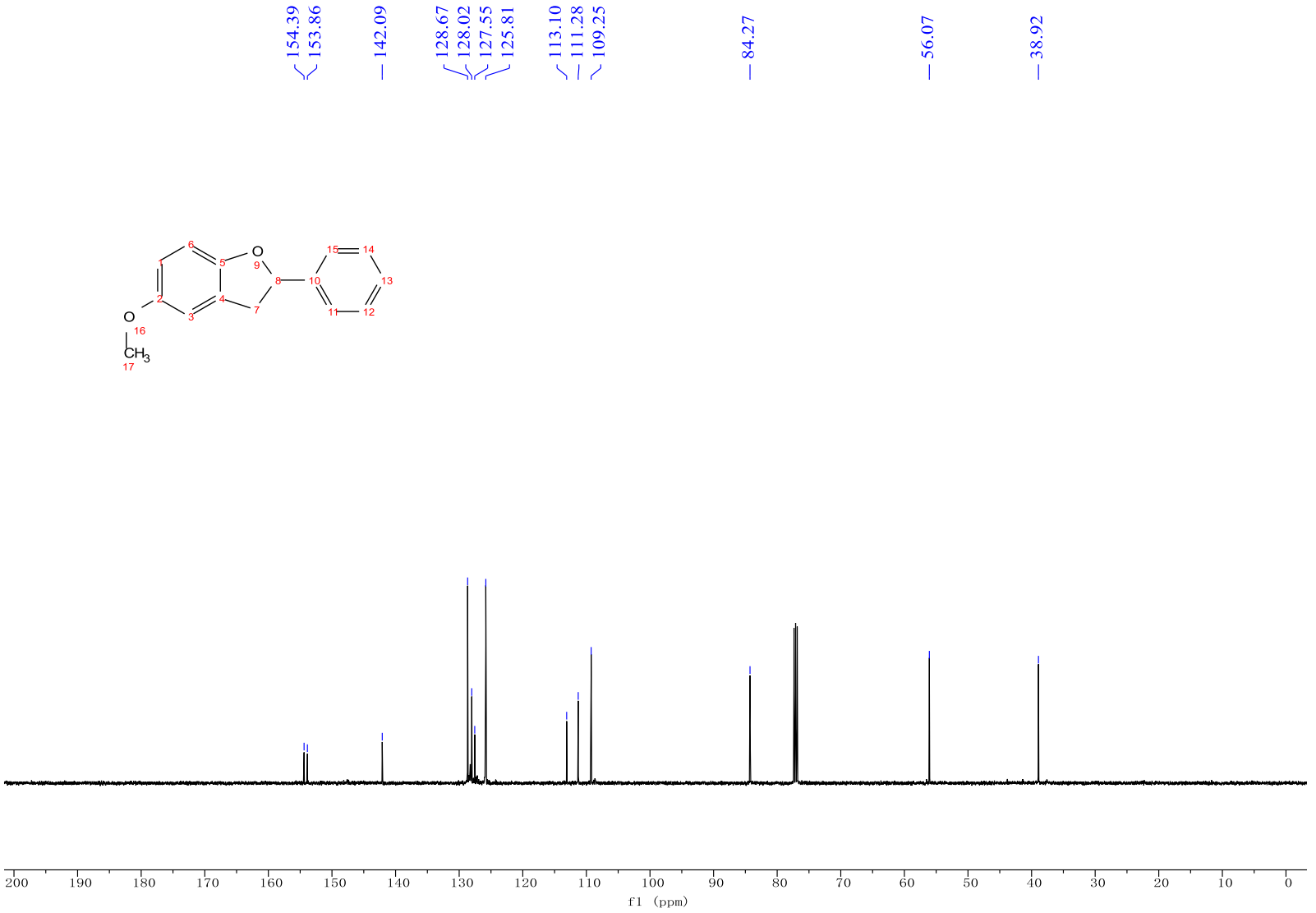
128.79
127.03
124.31
122.76
113.23
111.26
109.34
83.21
56.06
38.90
Br
16
O
9
O
17
CH3
18
f1 (ppm)


7.39 7.39 7.39 7.38 7.38 7.37 7.37 7.36 7.36 7.35 7.35 7.34 7.34 7.33 7.33 7.30 7.30 7.30 7.29 7.29 7.28 7.27 7.27 7.23 7.20 6.77 6.77 6.76 6.76 6.75 6.70 6.70 6.69 6.69 6.68 6.68 6.68 5.73 5.71 5.71 5.69 3.74 3.62 3.60 3.59 3.58 3.57 3.56 3.54 3.20 3.19 3.19 3.18 3.18 3.18 3.17 3.16 3.15 3.14
O
CH3
4.535
2.247
2.049
1.166
1.080
3.000
1.480
1.136
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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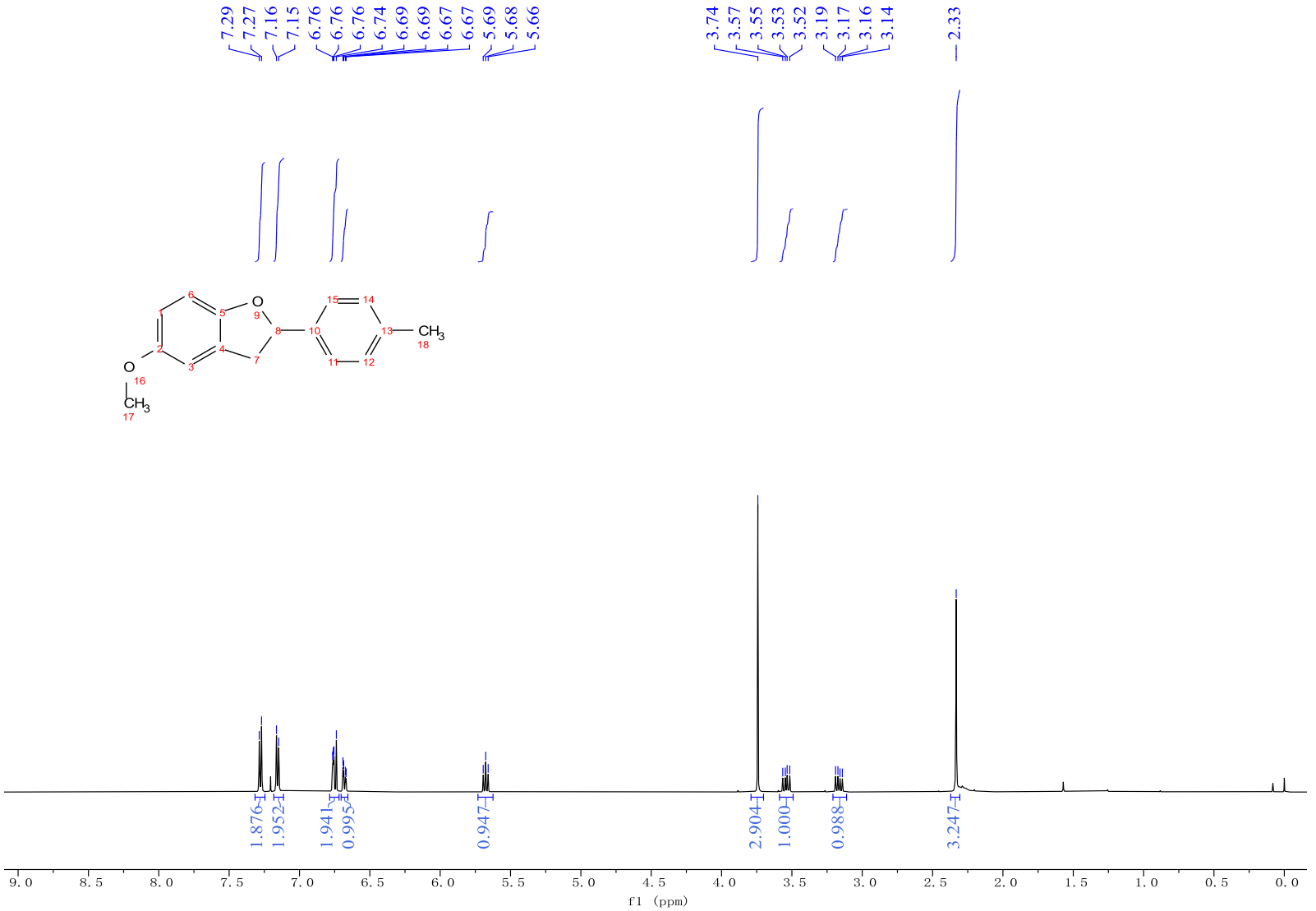
127.55
125.81
113.10
111.28
109.25
84.27
56.07
38.92
O
O
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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6.76
6.74
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6.69
6.67
6.67
5.69
5.68
5.66
3.74
3.57
3.55
3.53
3.52
3.19
3.17
3.16
3.14
2.33
1.876
1.952
1.941
0.995
0.947
2.904
1.000
0.988
3.247
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
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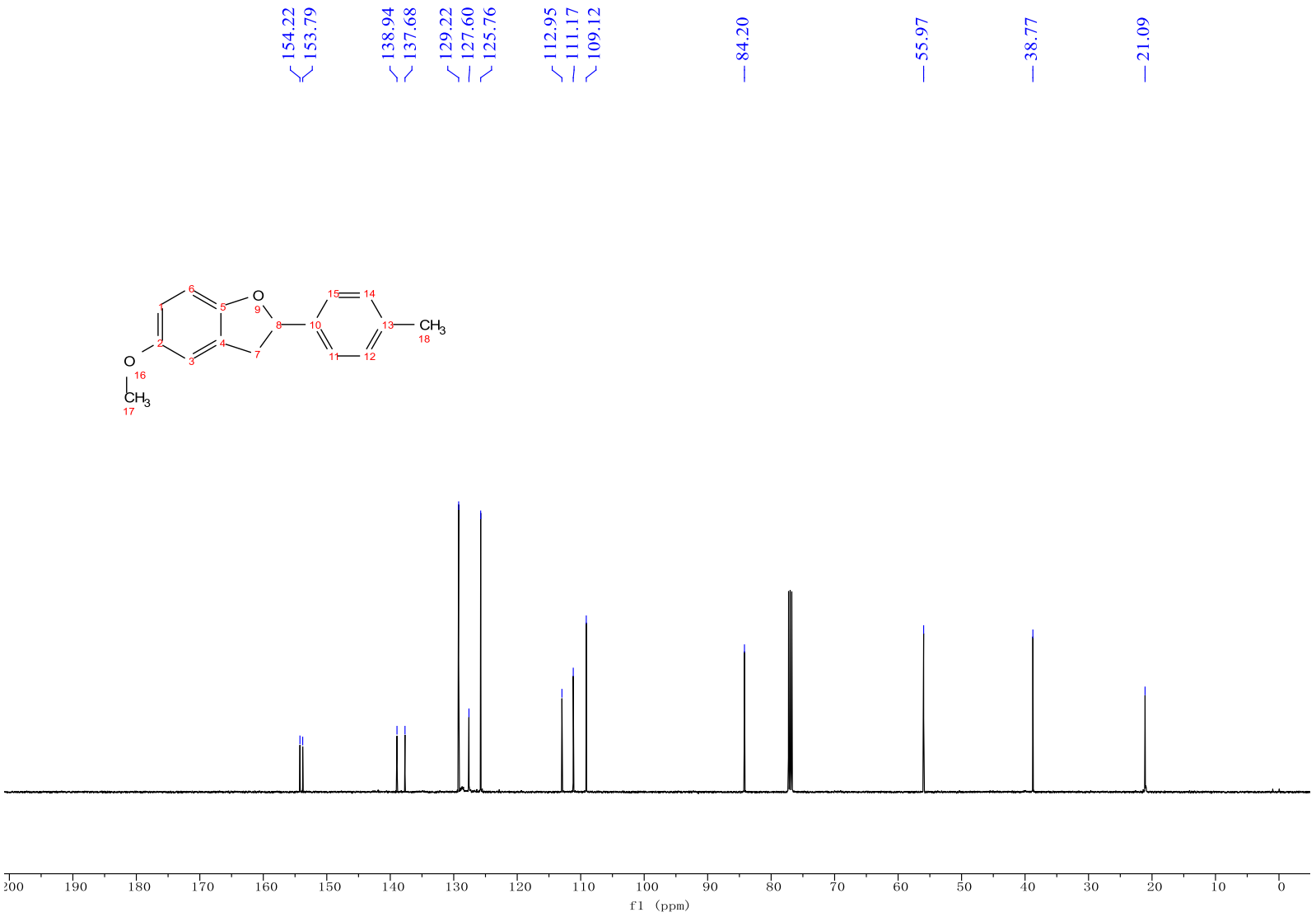
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138.94
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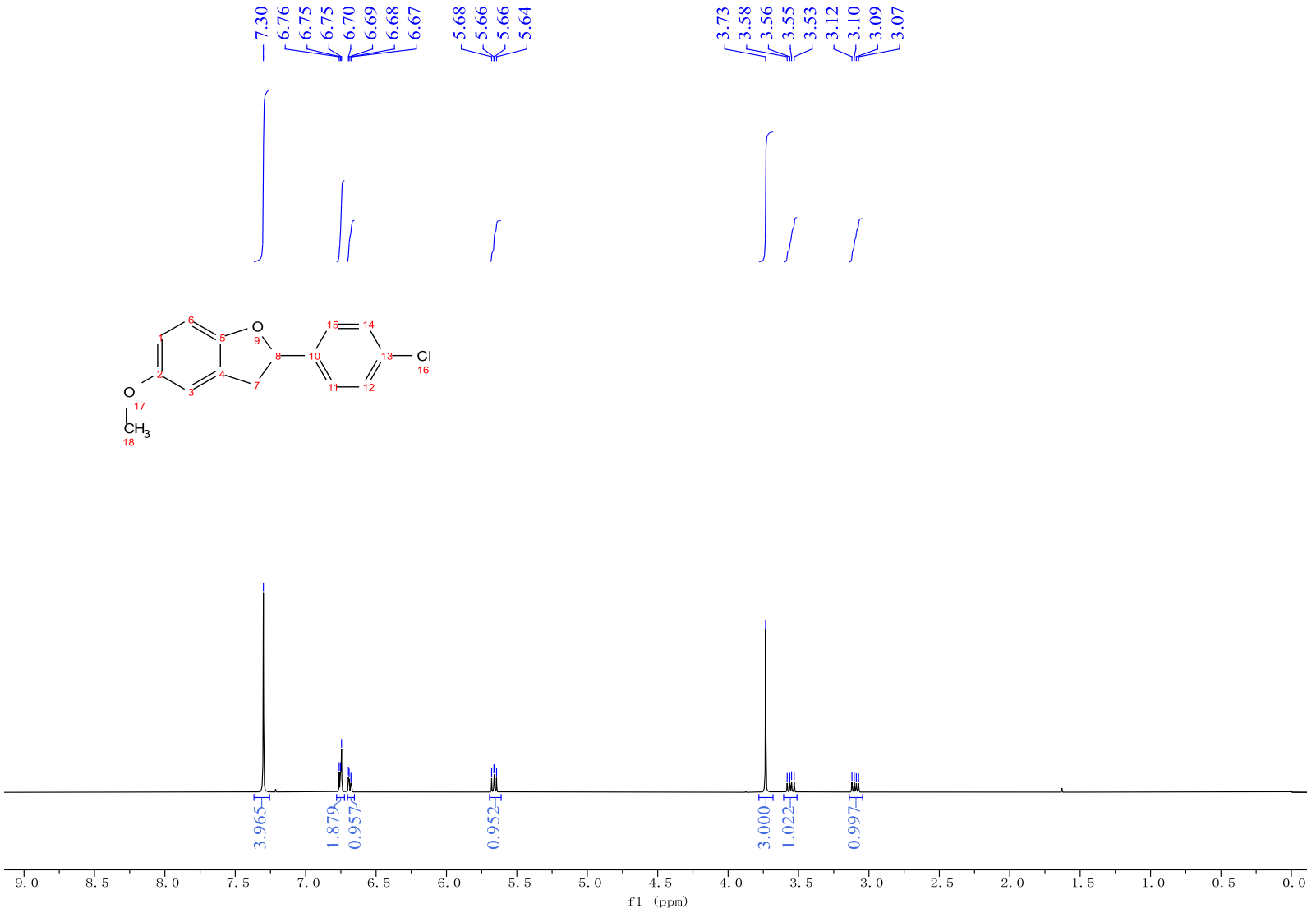
112.95
111.17
109.12
84.20
55.97
38.77
21.09
O
CH3
f1 (ppm)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6.69
6.68
6.67
5.68
5.66
5.66
5.64
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3.58
3.56
3.55
3.53
3.12
3.10
3.09
3.07
O
Cl
CH3
3.965
1.879
0.957
0.952
3.000
1.022
0.997
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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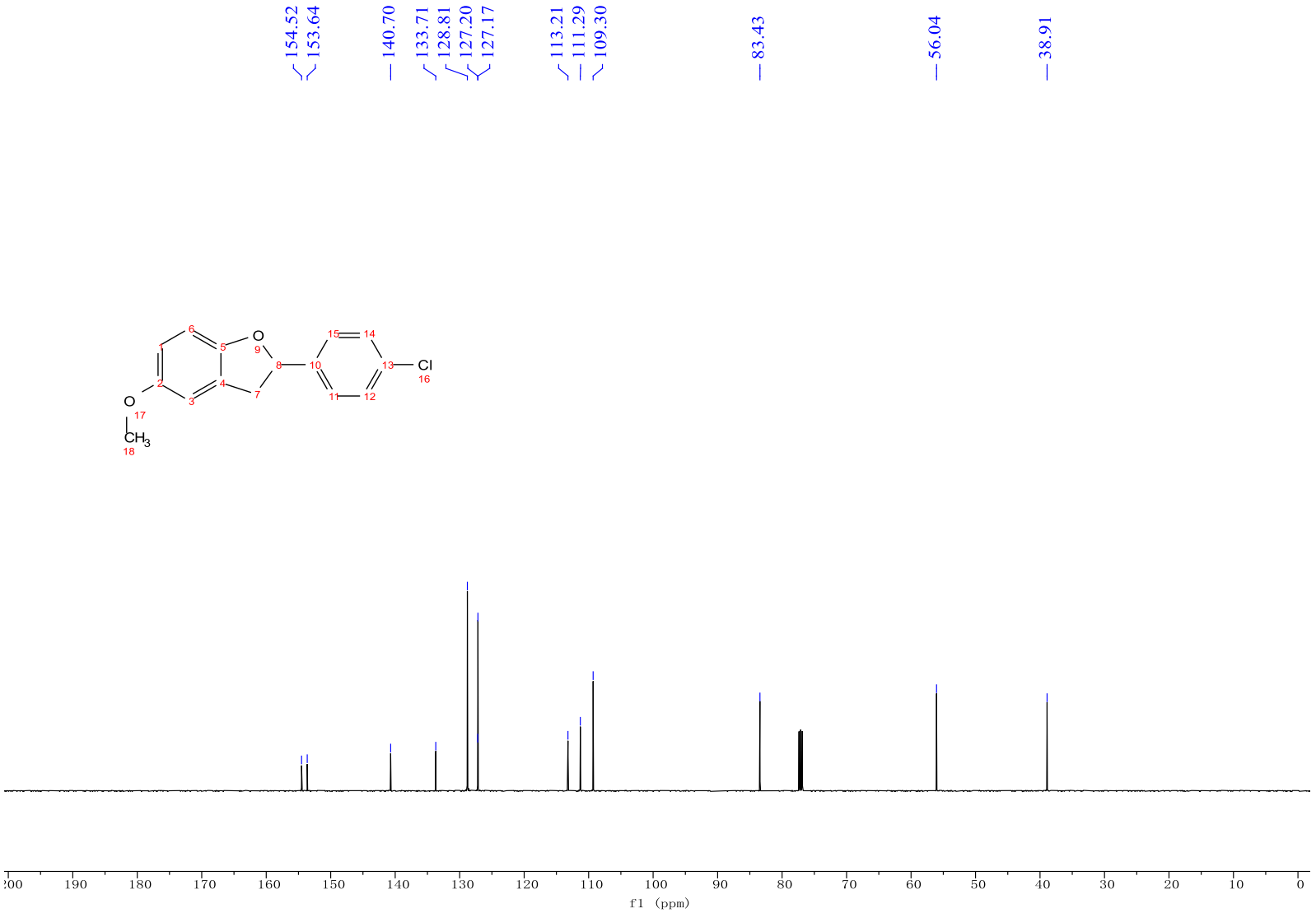
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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113.21
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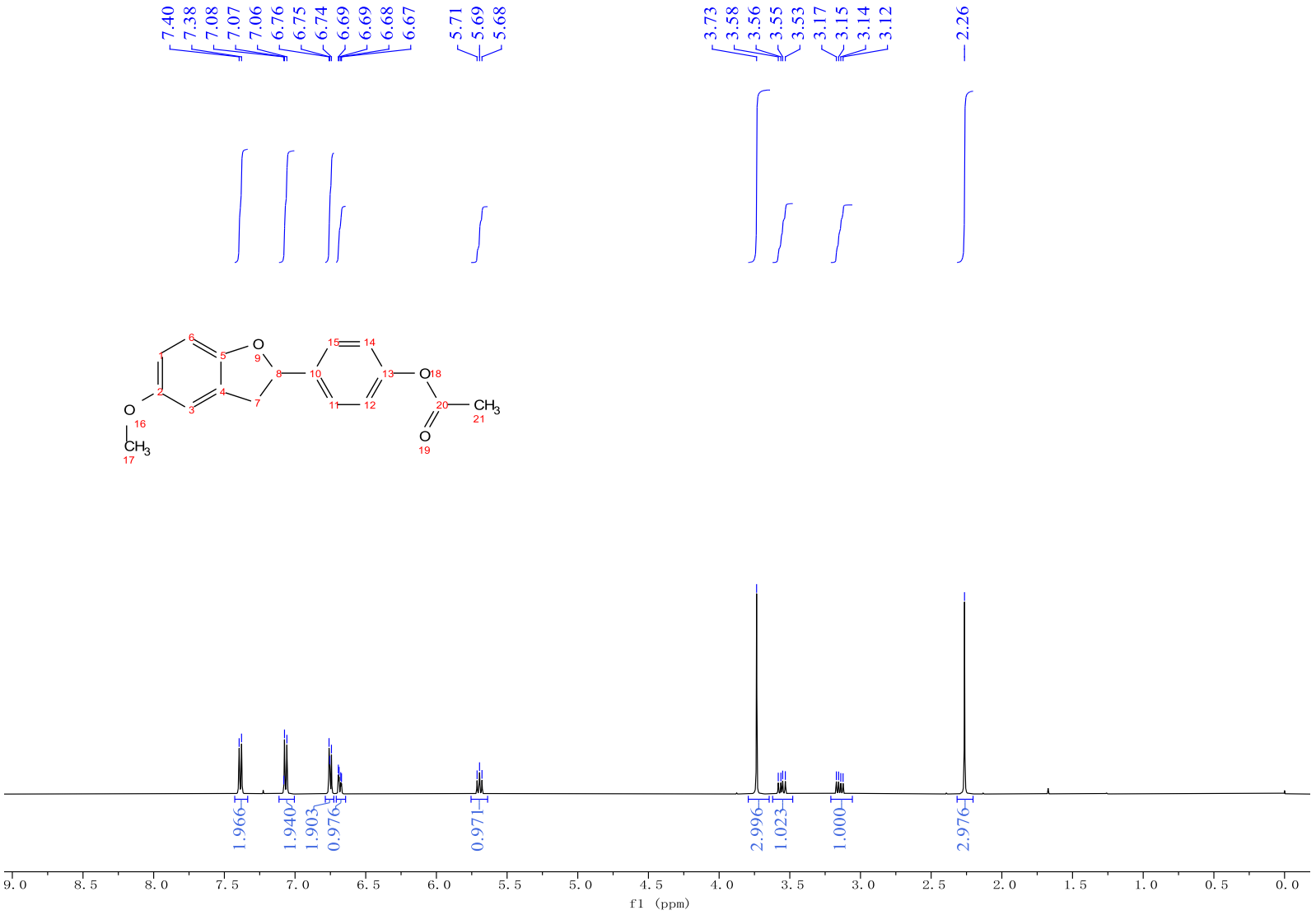
109.30
83.43
56.04
38.91
O
Cl
CH3
f1 (ppm)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6.74
6.69
6.69
6.68
6.67
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5.69
5.68
3.73
3.58
3.56
3.55
3.53
3.17
3.15
3.14
3.12
2.26
1.966
1.940
1.903
0.976
0.971
2.996
1.023
1.000
2.976
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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4.5
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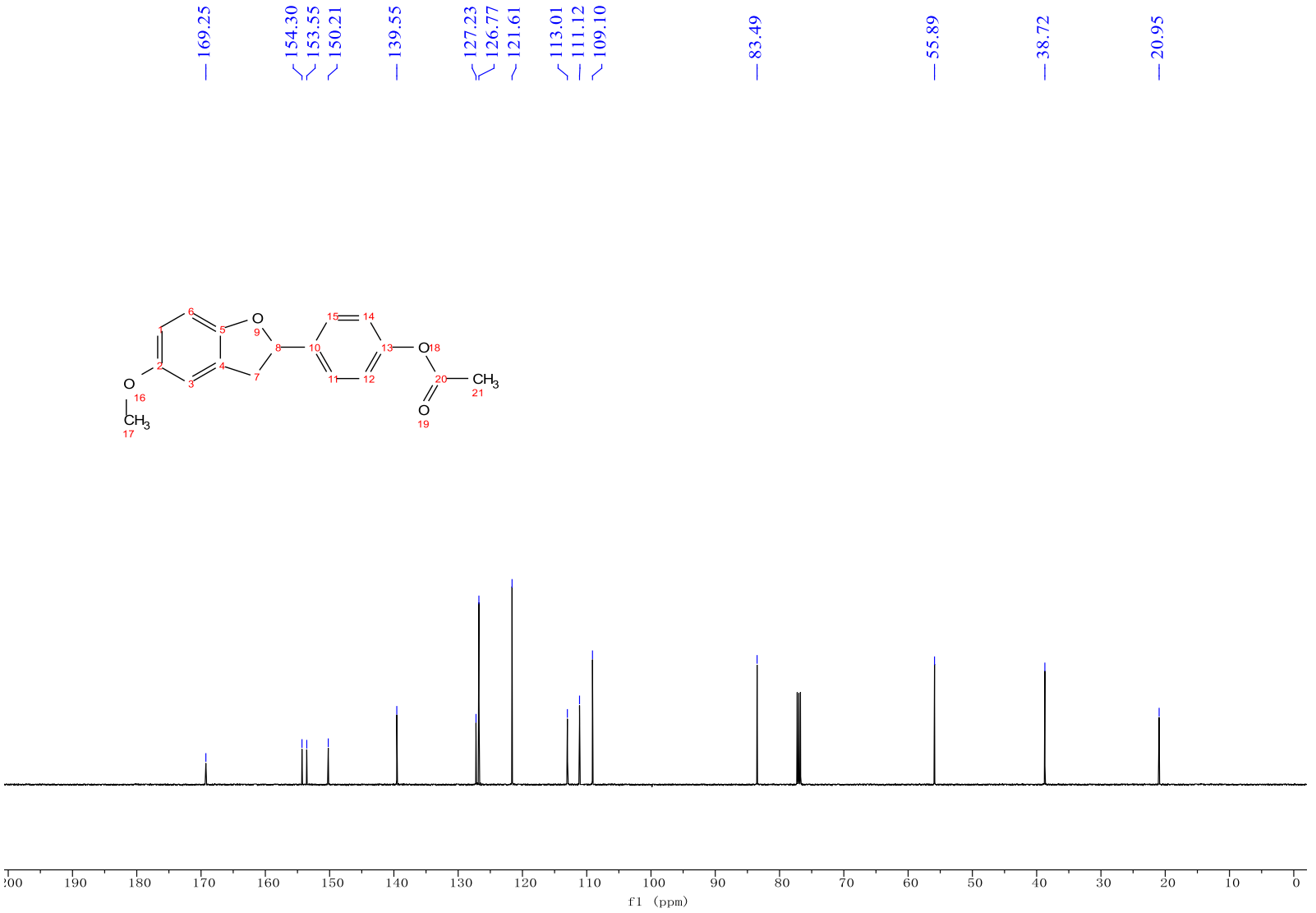
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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154.30
153.55
150.21
139.55
127.23
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121.61
113.01
111.12
109.10
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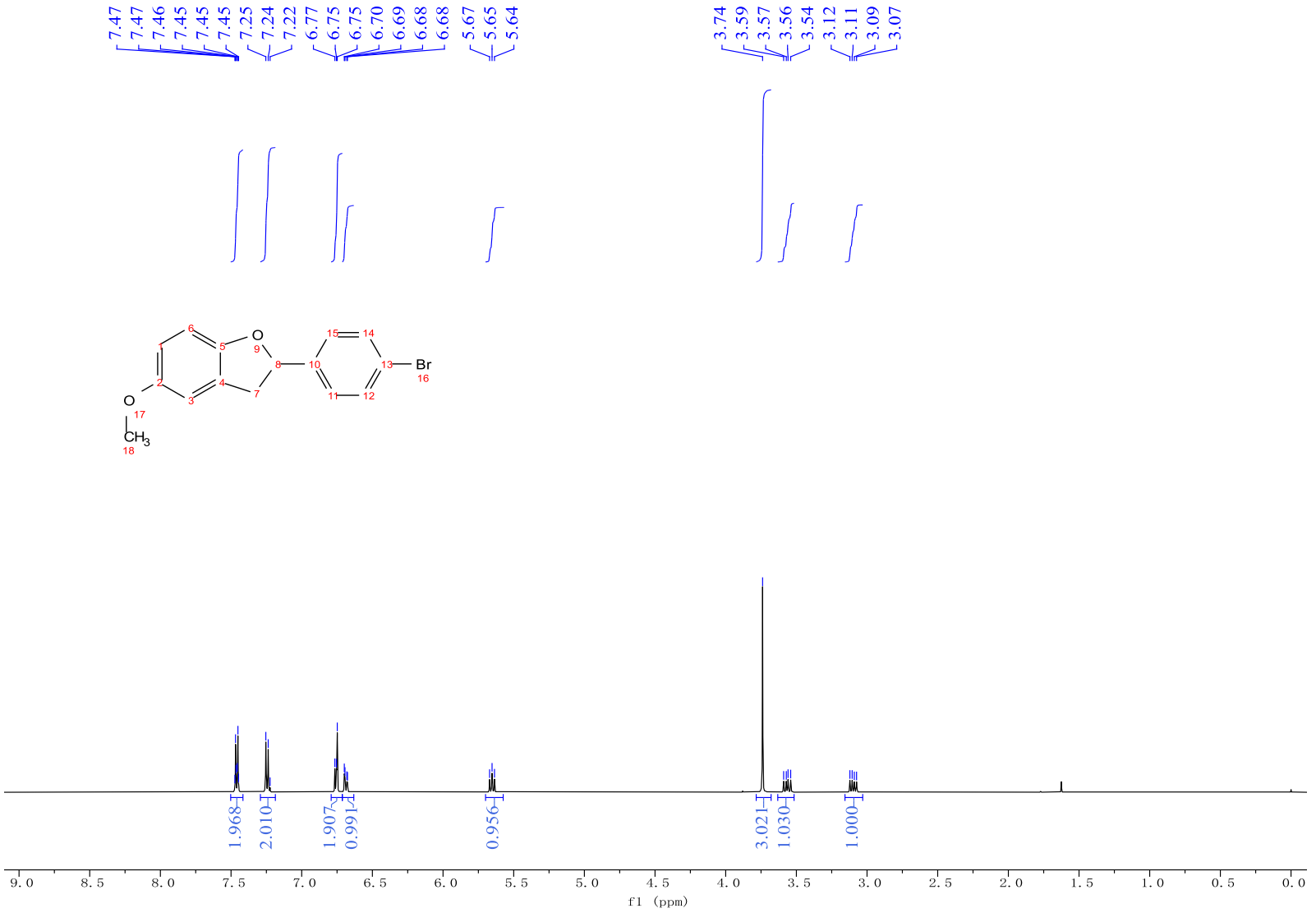
55.89
38.72
20.95
O
CH3
f1 (ppm)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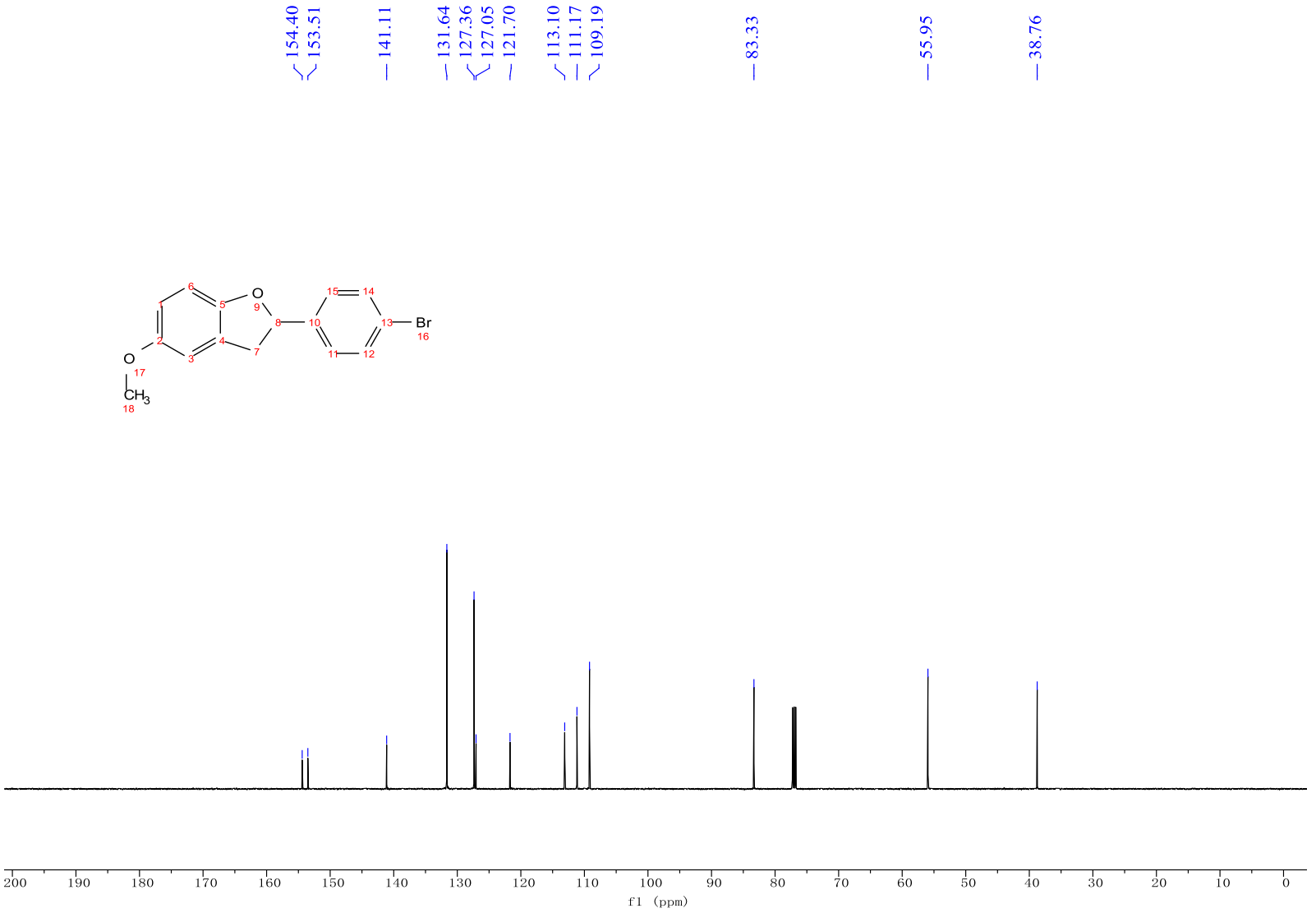
7.45
7.45
7.25
7.24
7.22
6.77
6.75
6.75
6.70
6.69
6.68
6.68
5.67
5.65
5.64
3.74
3.59
3.57
3.56
3.54
3.12
3.11
3.09
3.07
O
Br
CH3
1.968
2.010
1.907
0.991
0.956
3.021
1.030
1.000
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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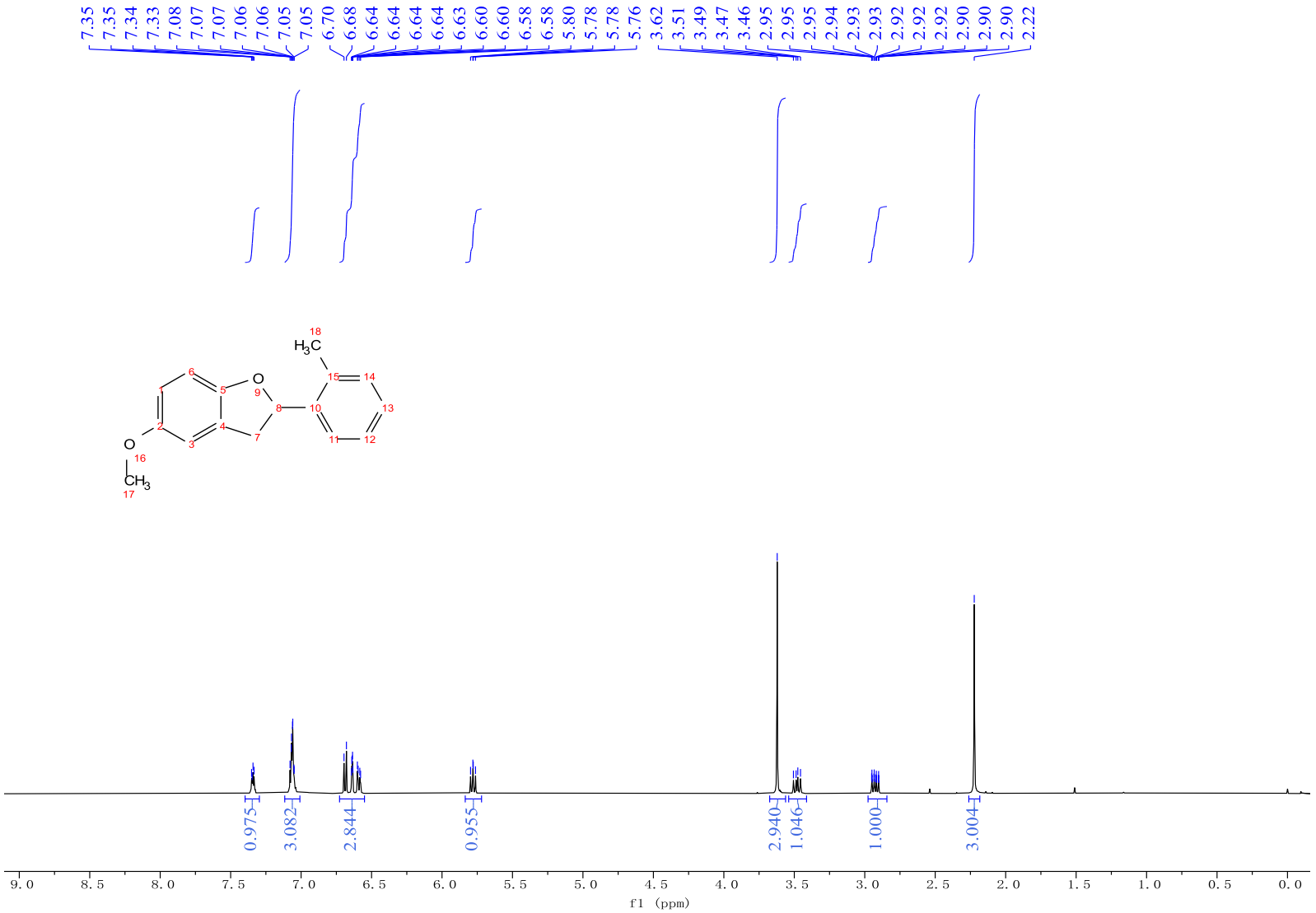
141.11
131.64
127.36
127.05
121.70
113.10
111.17
109.19
83.33
55.95
38.76
O
Br
CH3
f1 (ppm)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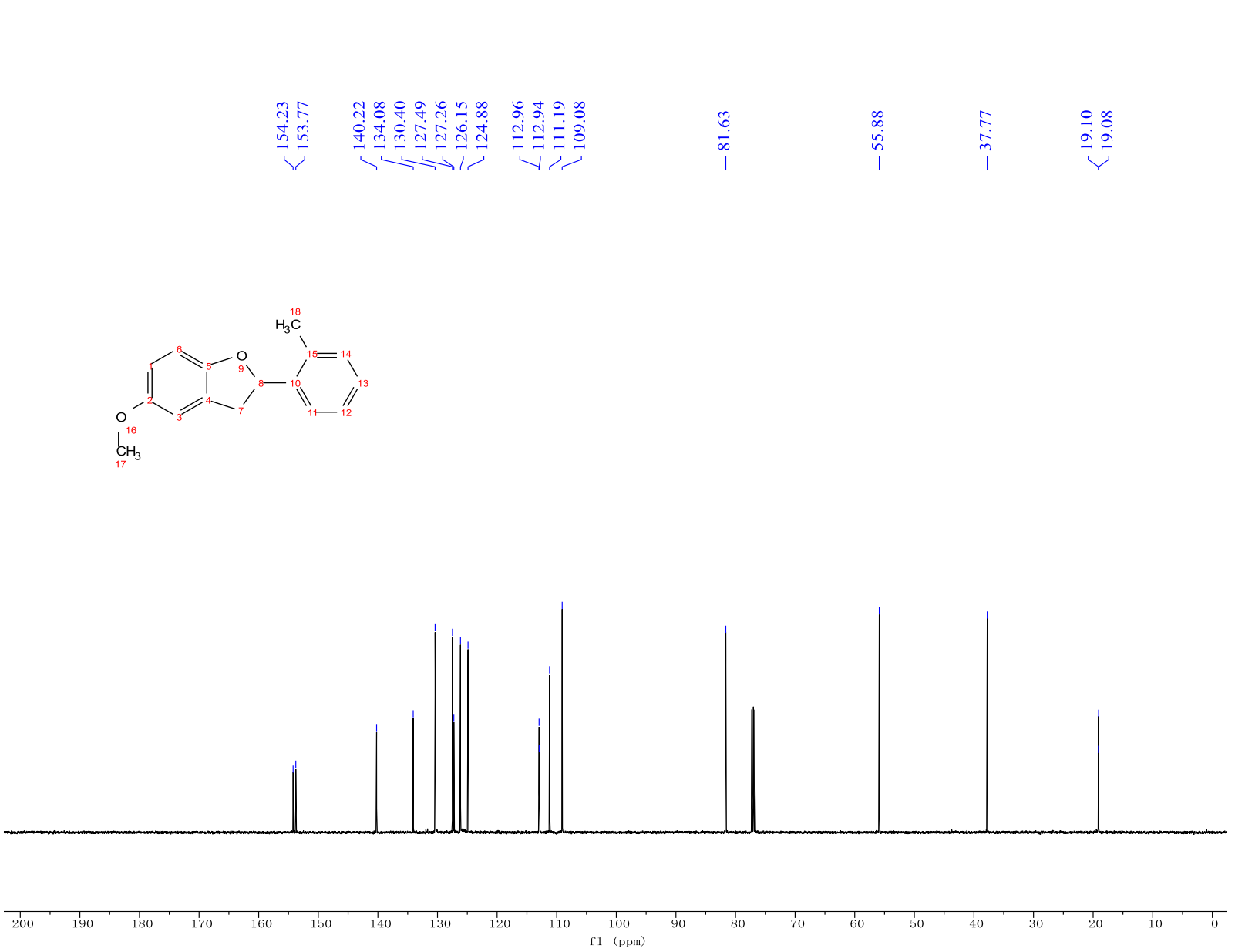
6.70 6.68 6.64 6.64 6.64 6.64 6.63 6.60 6.60 6.58 6.58 5.80 5.78 5.78 5.76
3.62 3.51 3.49 3.47 3.46 2.95 2.95 2.95 2.94 2.93 2.93 2.92 2.92 2.92 2.90 2.90 2.90 2.22
H3C
O
CH3
0.975
3.082
2.844
0.955
2.940
1.046
1.000
3.004
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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140.22
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127.26
126.15
124.88
112.96
112.94
111.19
109.08
81.63
55.88
37.77
19.10
19.08
H3C
18
O
9
O
16
CH3
17
200
190
180
170
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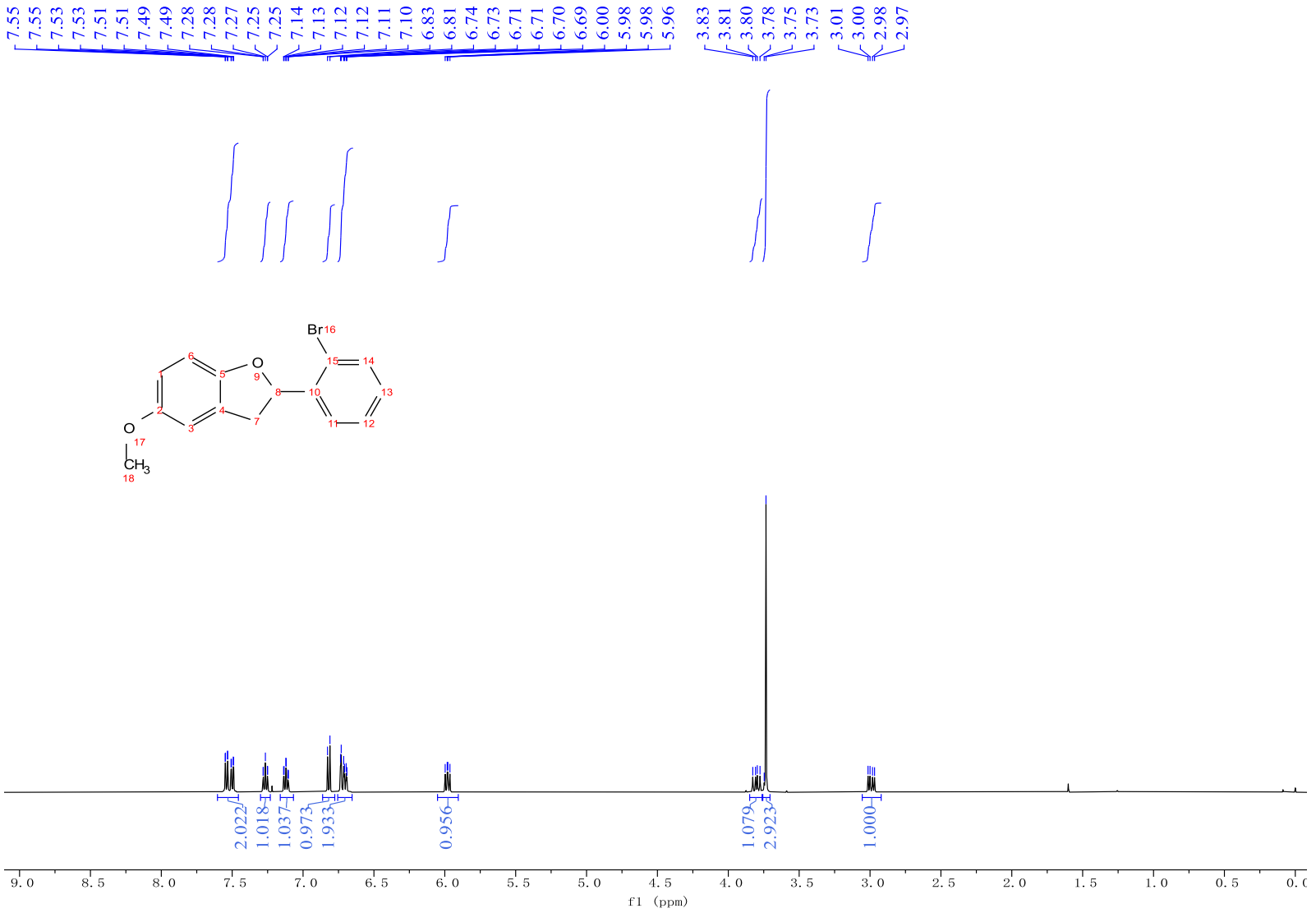
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.49
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7.28
7.27
7.25
7.25
7.14
7.13
7.12
7.12
7.11
7.10
6.83
6.81
6.74
6.73
6.71
6.71
6.70
6.69
6.00
5.98
5.98
5.96
3.83
3.81
3.80
3.78
3.75
3.73
3.01
3.00
2.98
2.97
Br16
O9
O17
CH3
18
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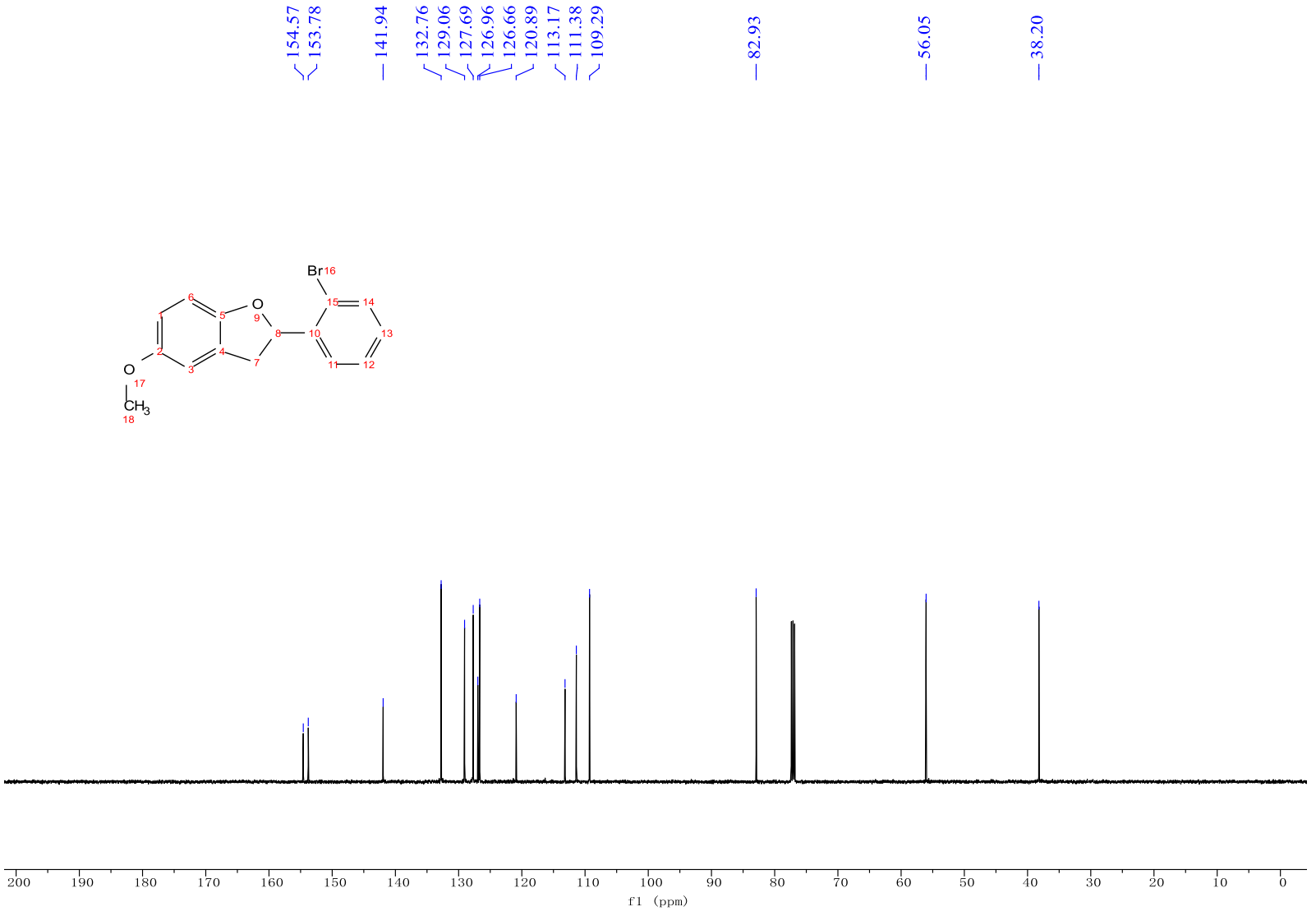
1.018
1.037
0.973
1.933
0.956
1.079
2.923
1.000
f1 (ppm)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126.66
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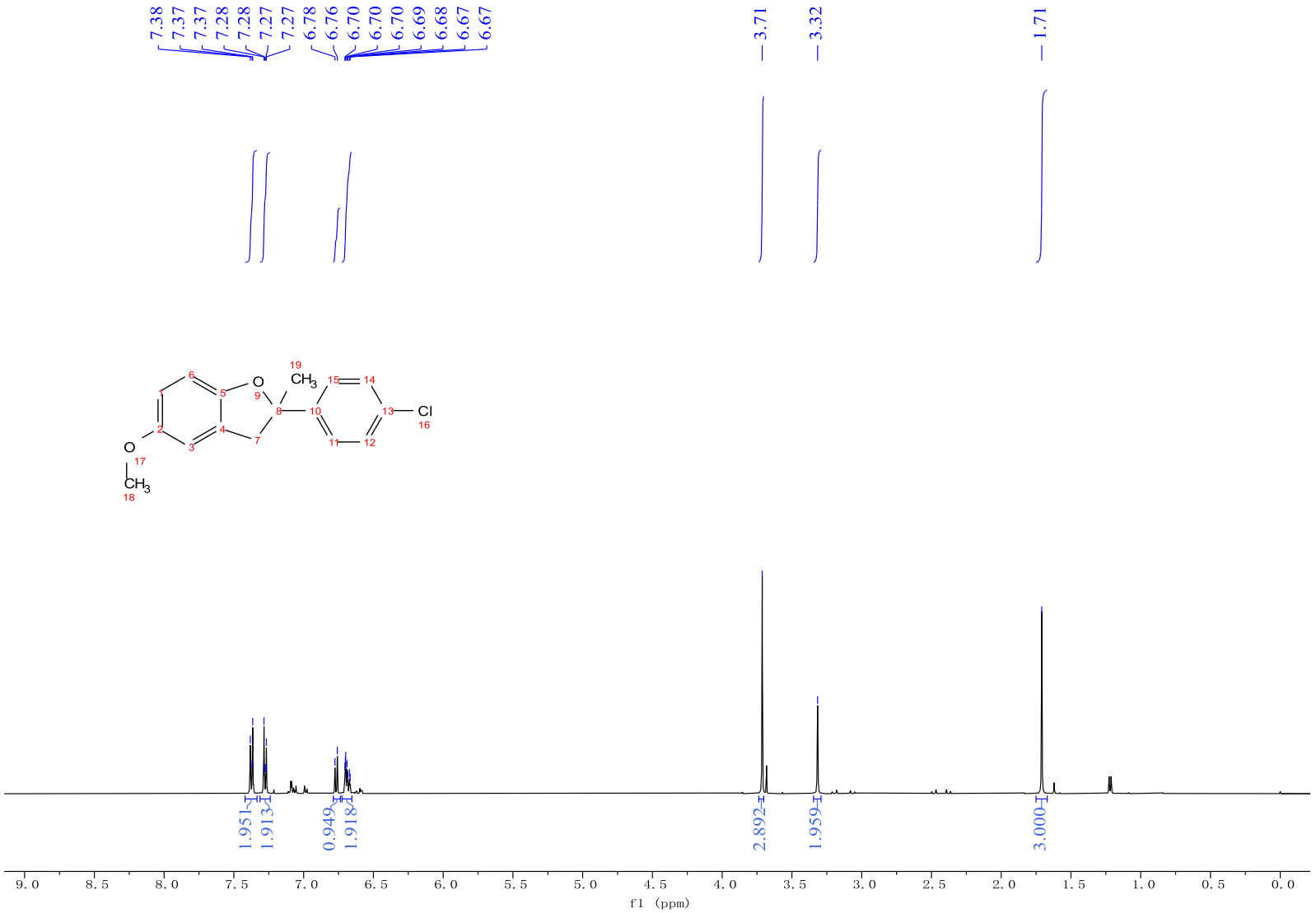
56.05
38.20
Br16
O9
O17
CH3
18
f1 (ppm)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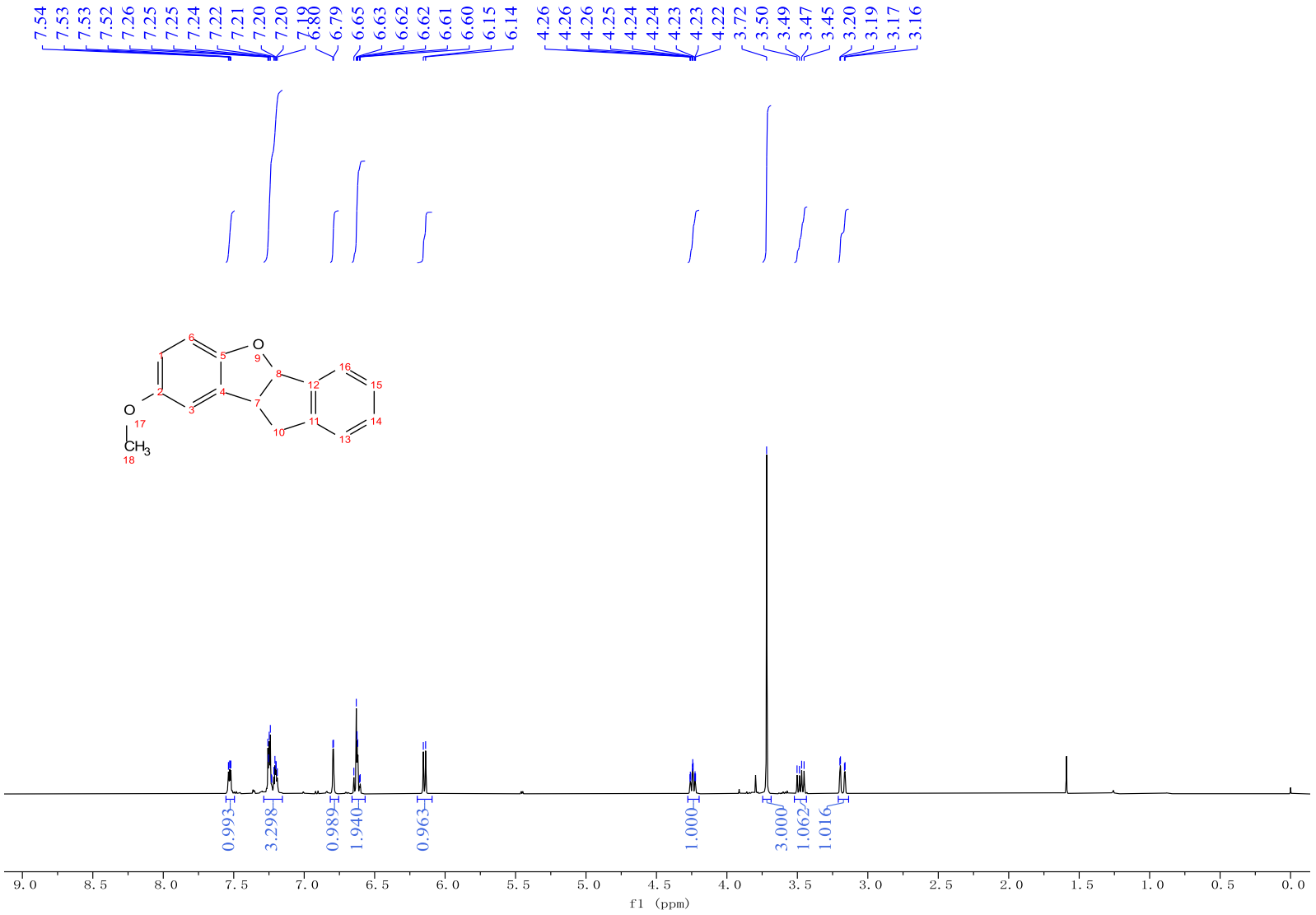
6.70
6.70
6.70
6.69
6.68
6.67
6.67
3.71
3.32
1.71
O
CH3
Cl
1.951
1.913
0.949
1.918
2.892
1.959
3.000
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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7.19
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6.79
6.65
6.63
6.62
6.62
6.61
6.60
6.15
6.14
4.26
4.26
4.26
4.25
4.24
4.24
4.23
4.23
4.22
3.72
3.50
3.49
3.47
3.45
3.20
3.19
3.17
3.16
O
9
O
17
CH3
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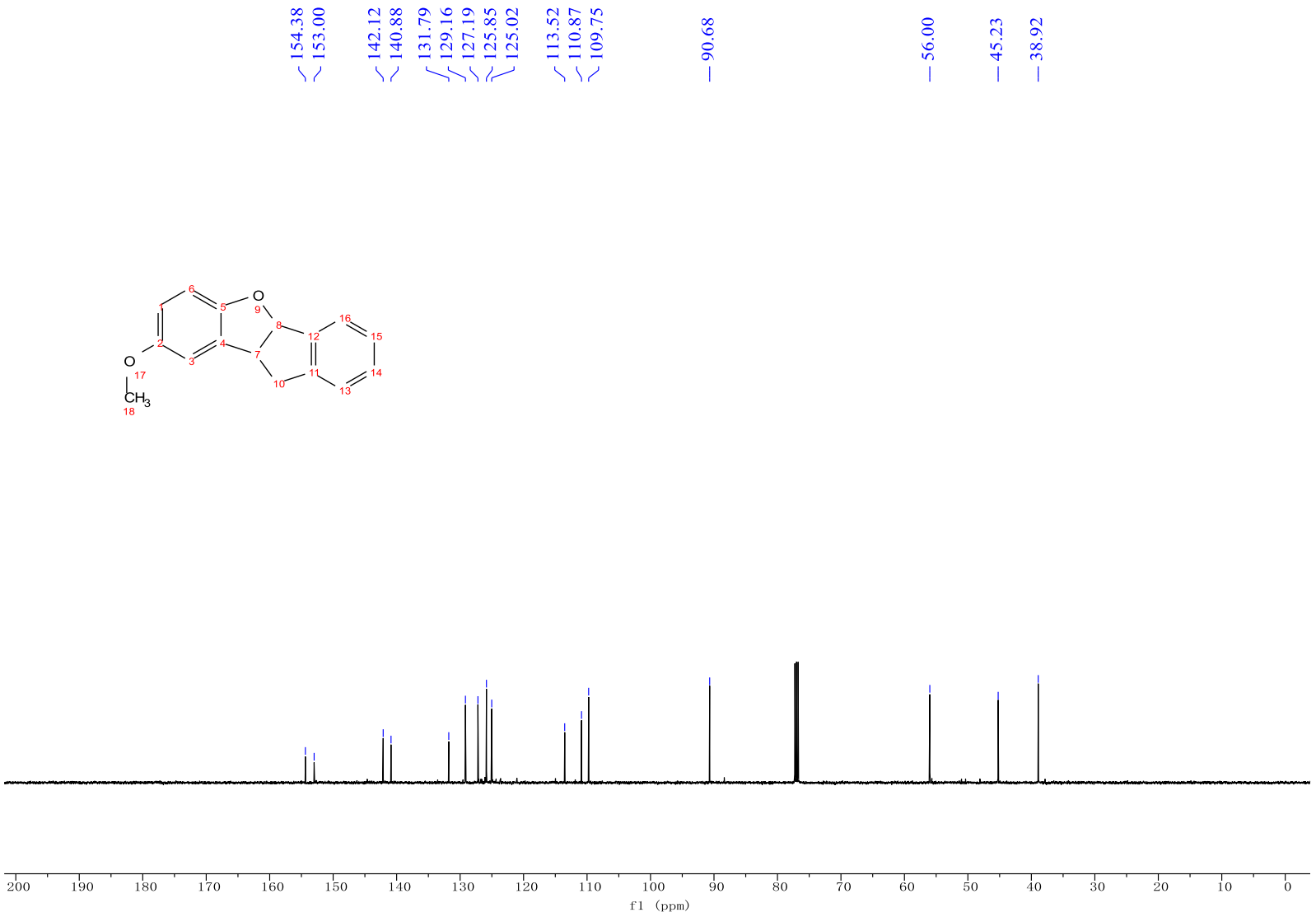
3.298
0.989
1.940
0.963
1.000
3.000
1.062
1.016
f1 (ppm)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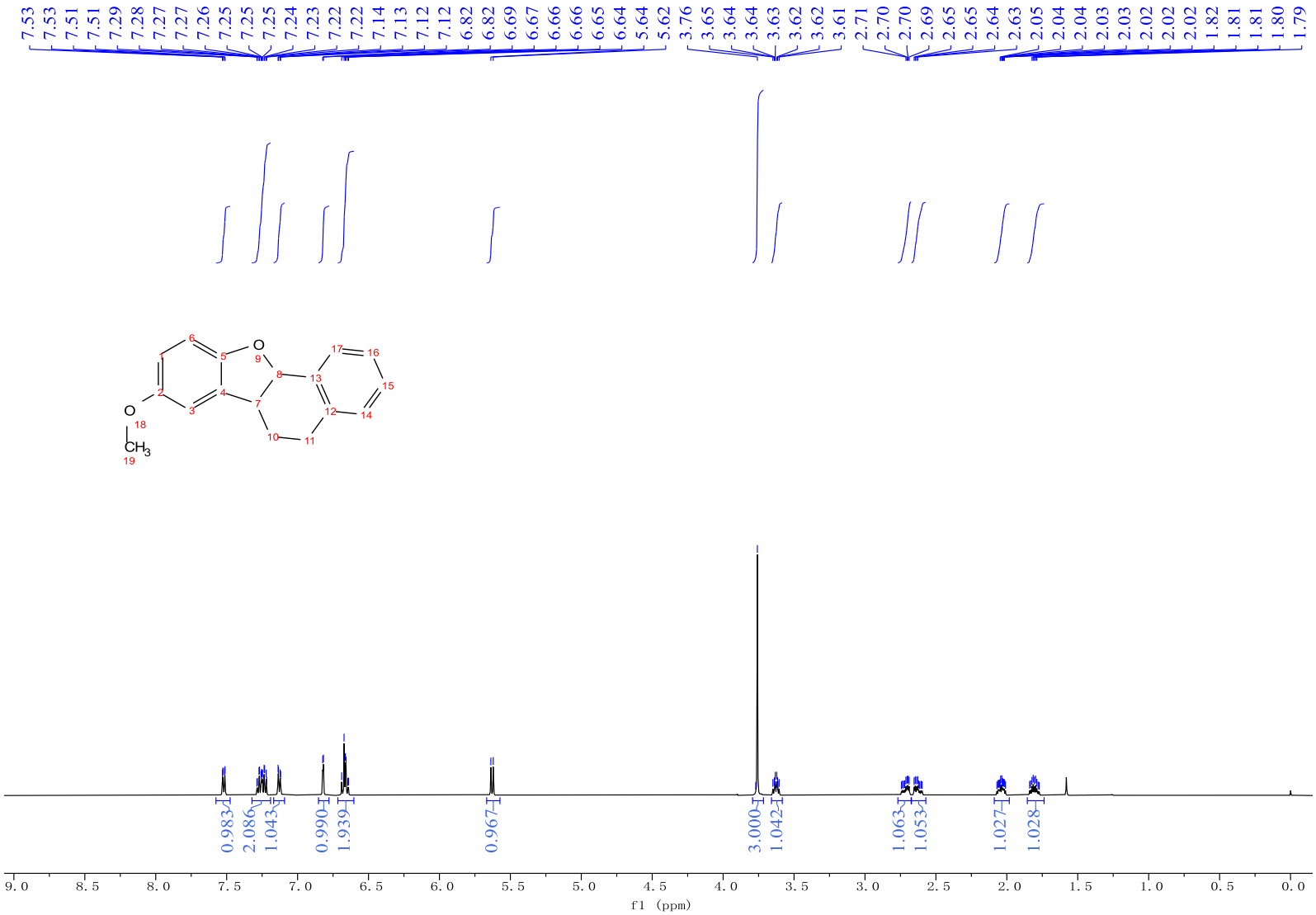
125.02
113.52
110.87
109.75
90.68
56.00
45.23
38.92
O
O
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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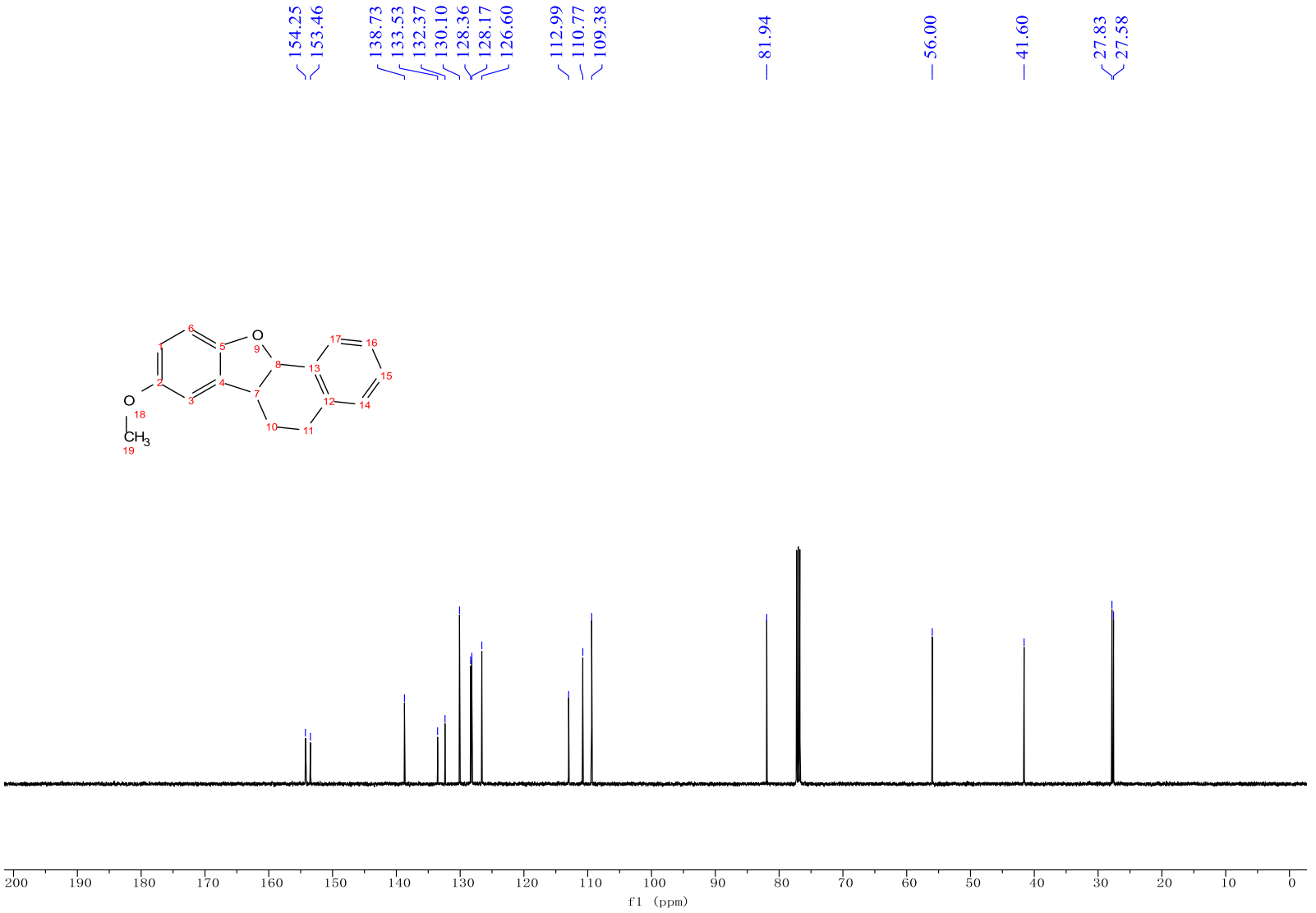
3.76 3.65 3.64 3.64 3.63 3.62 3.62 3.61
2.71 2.70 2.70 2.69 2.65 2.65 2.64 2.63 2.05 2.04 2.04 2.03 2.03 2.02 2.02 2.02 1.82 1.81 1.81 1.80 1.79
O
O
CH3
0.983 2.086 1.043 0.990 1.939 0.967 3.000 1.042 1.063 1.053 1.027 1.028
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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128.17
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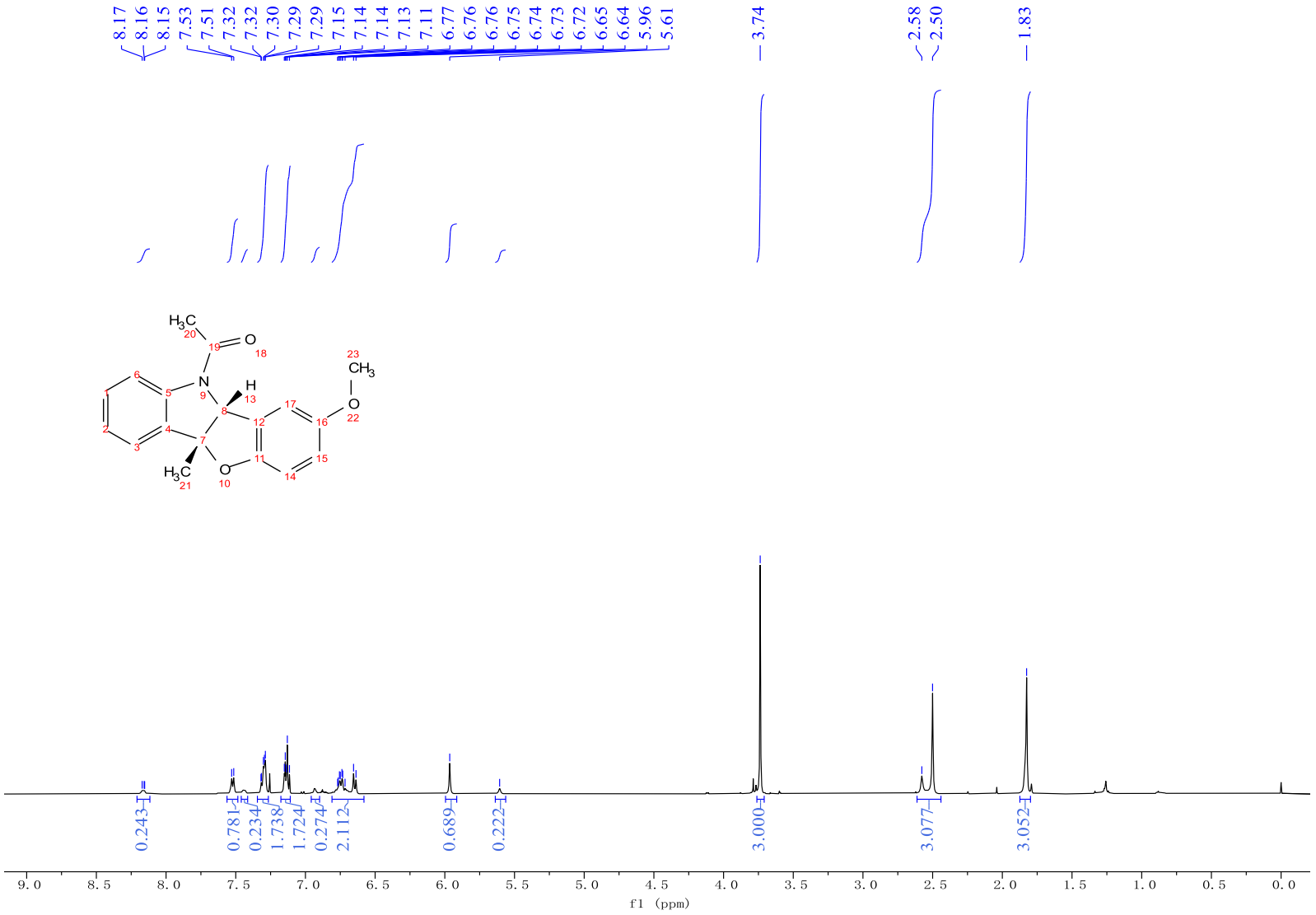
109.38
81.94
56.00
41.60
27.83
27.58
O
O
CH3
f1 (ppm)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7.29
7.29
7.15
7.14
7.14
7.13
7.11
6.77
6.76
6.76
6.75
6.74
6.73
6.72
6.65
6.64
5.96
5.61
3.74
2.58
2.50
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0.243
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0.234
1.738
1.724
0.274
2.112
0.689
0.222
3.000
3.077
3.052
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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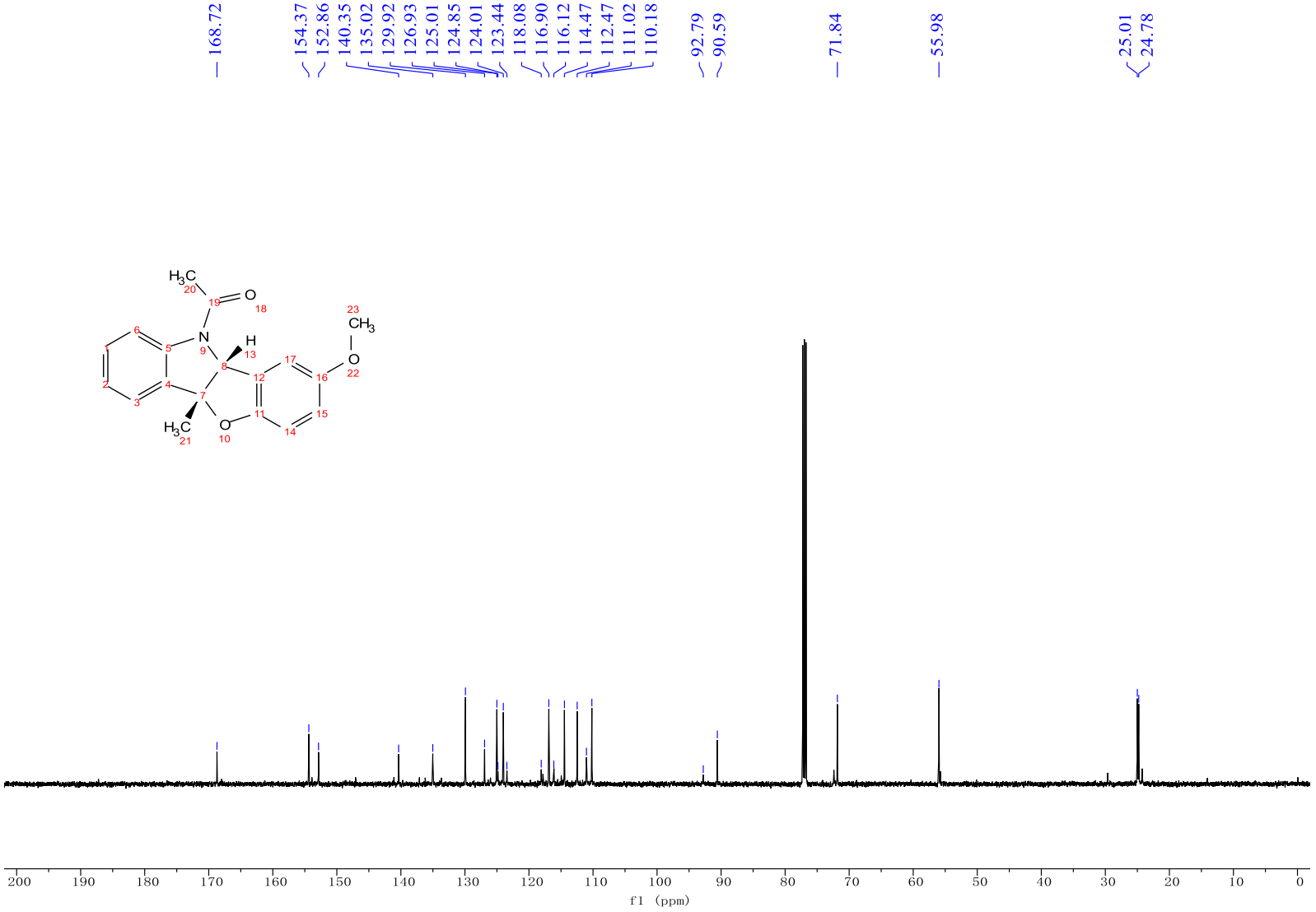
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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125.01
124.85
124.01
123.44
118.08
116.90
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111.02
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92.79
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71.84
55.98
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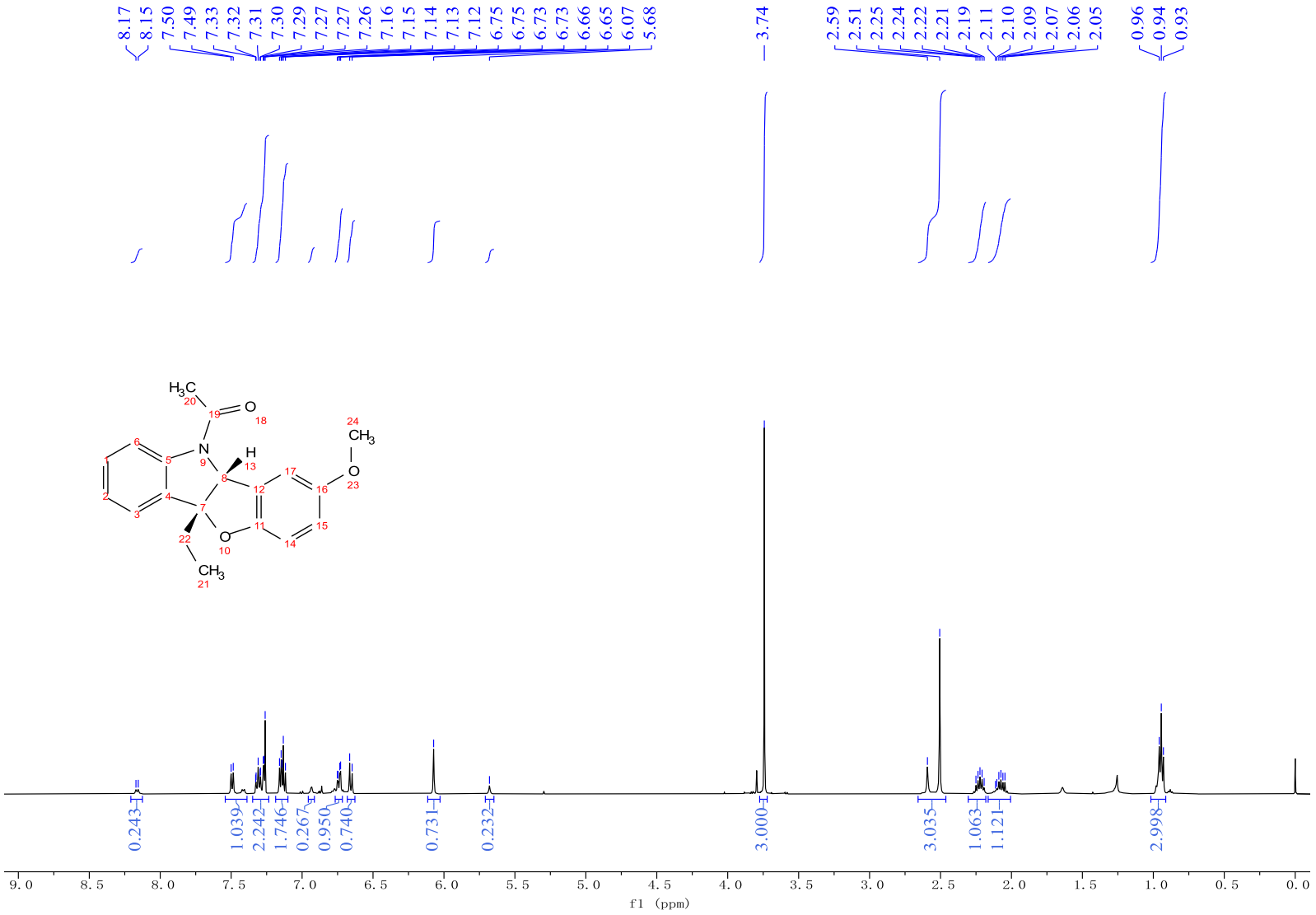
24.78
f1 (ppm)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7.27
7.27
7.26
7.16
7.15
7.14
7.13
7.12
6.75
6.75
6.73
6.73
6.66
6.65
6.07
5.68
3.74
2.59
2.51
2.25
2.24
2.22
2.21
2.19
2.11
2.10
2.09
2.07
2.06
2.05
0.96
0.94
0.93
H3C
20
19
O
18
24
CH3
6
N
9
H
13
5
1
8
17
16
O
23
2
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12
3
7
11
15
22
14
O
10
CH3
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0.950
0.740
0.731
0.232
3.000
3.035
1.063
1.121
2.998
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8.5
8.0
7.5
7.0
6.5
6.0
5.5
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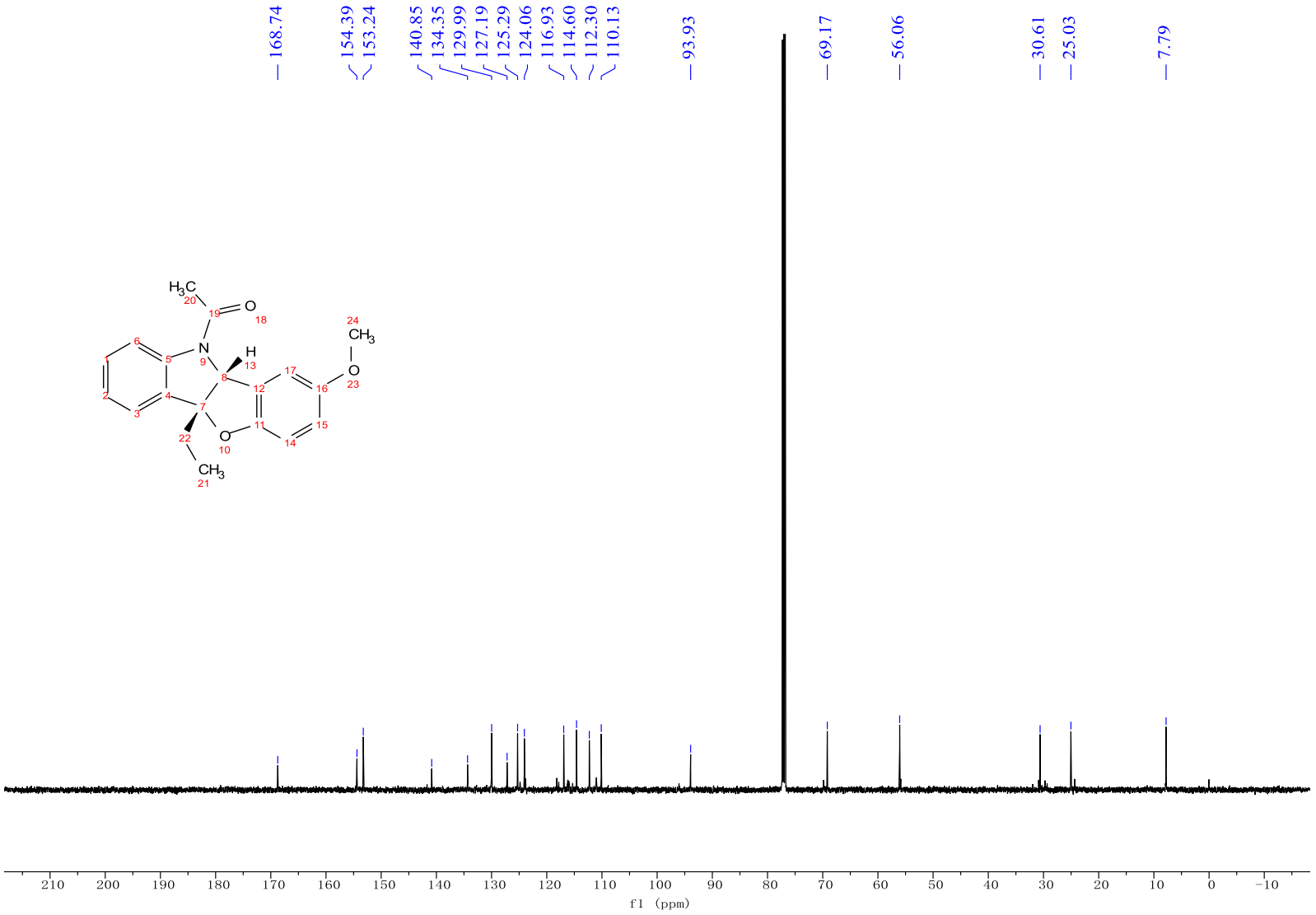
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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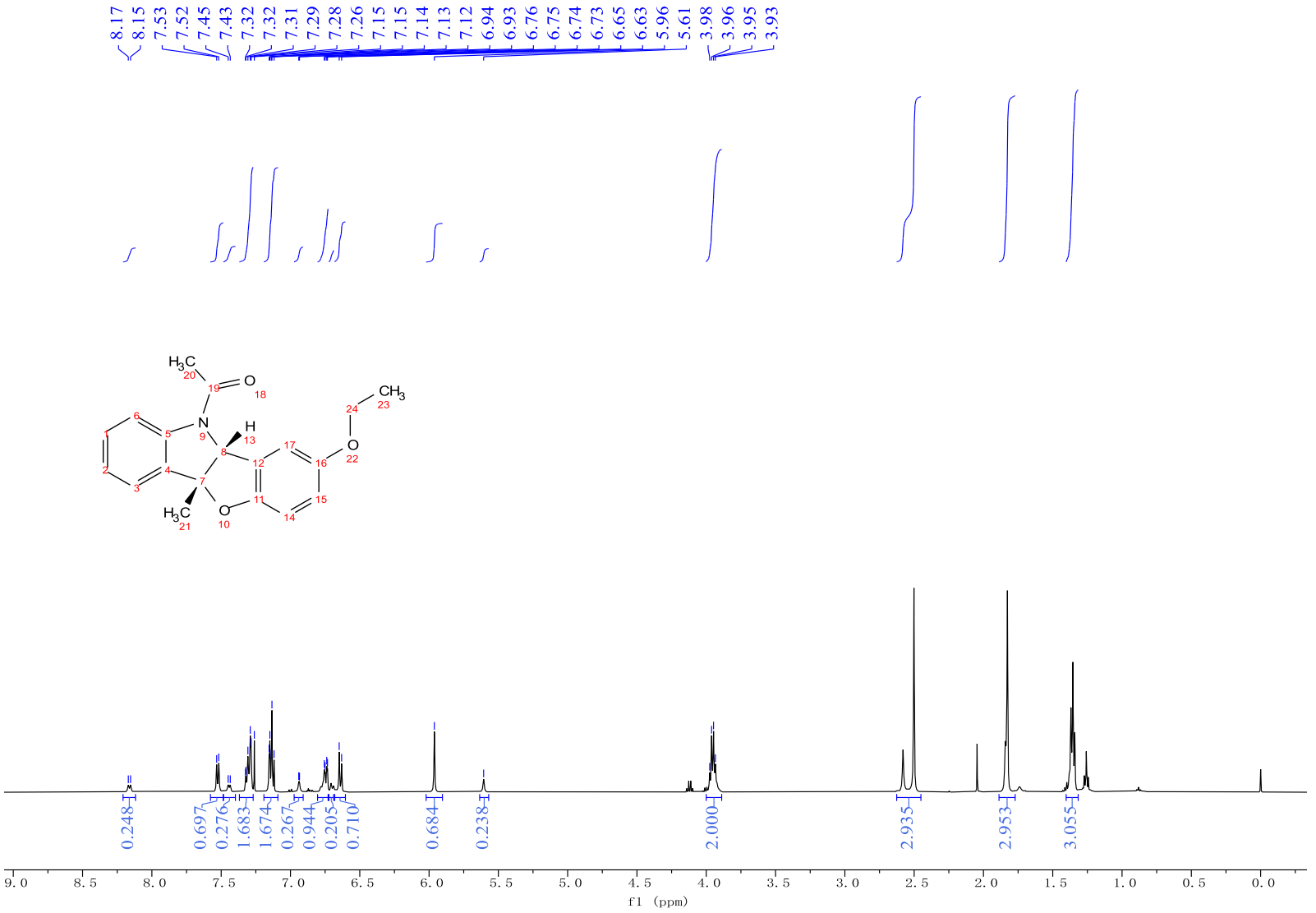
114.60
112.30
110.13
93.93
69.17
56.06
30.61
25.03
7.79
H3C
20
19
O
18
N
9
H
13
8
6
5
1
2
3
4
7
22
CH3
21
O
10
11
12
14
15
16
17
O
23
24
CH3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)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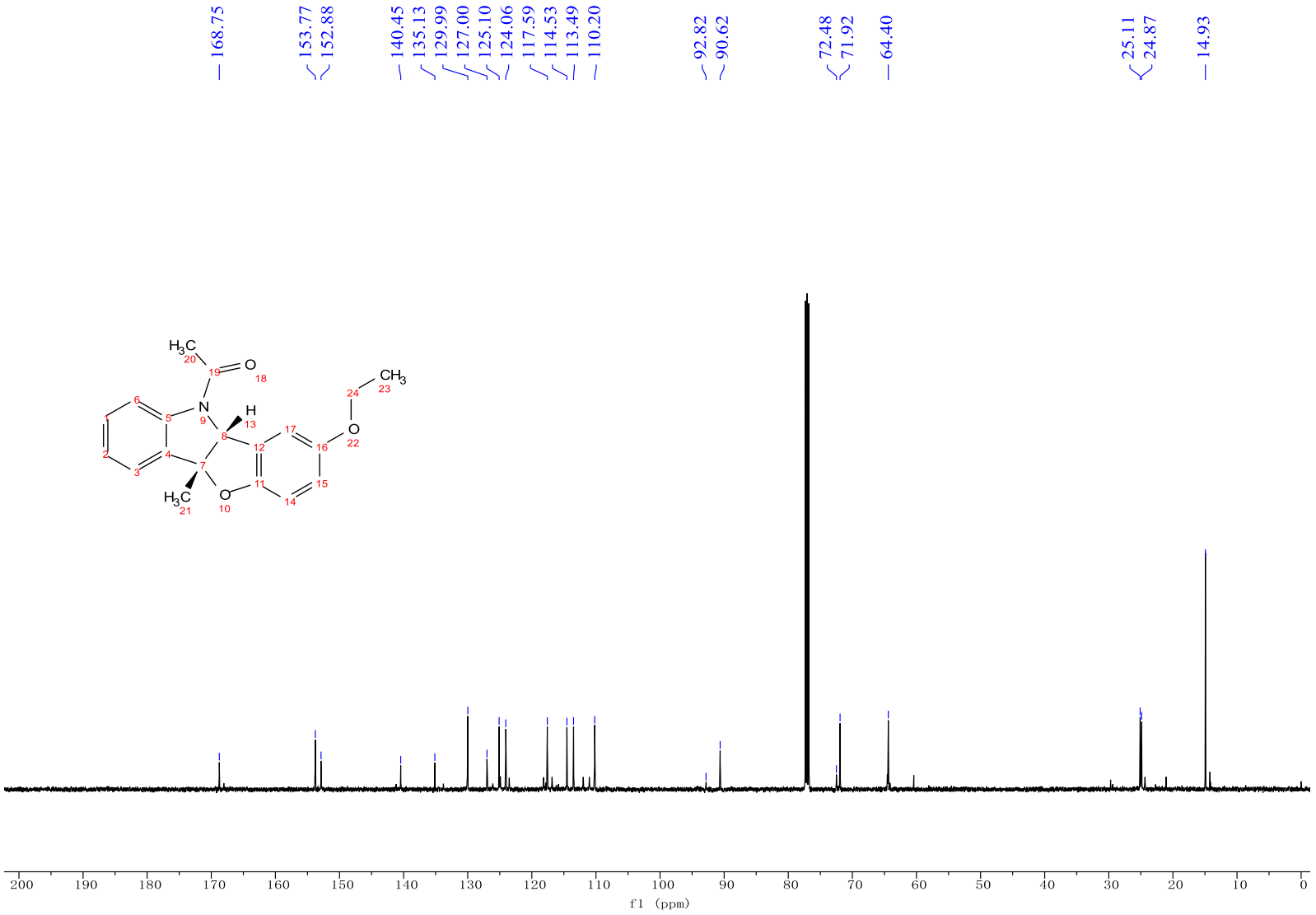
7.32
7.31
7.29
7.28
7.26
7.15
7.15
7.14
7.13
7.12
6.94
6.93
6.76
6.75
6.74
6.73
6.65
6.63
5.96
5.61
3.98
3.96
3.95
3.93
H3C
20
19
O
18
CH3
23
24
N
9
H
13
O
22
H3C
21
O
10
0.248
0.697
0.276
1.683
1.674
0.267
0.944
0.205
0.710
0.684
0.238
2.000
2.935
2.953
3.055
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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114.53
113.49
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72.48
71.92
64.40
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24.87
14.93
H3C
20
19
O
18
CH3
23
24
N
9
H
13
8
17
O
22
6
5
1
2
4
7
12
16
3
11
15
14
H3C
21
O
10
200
190
180
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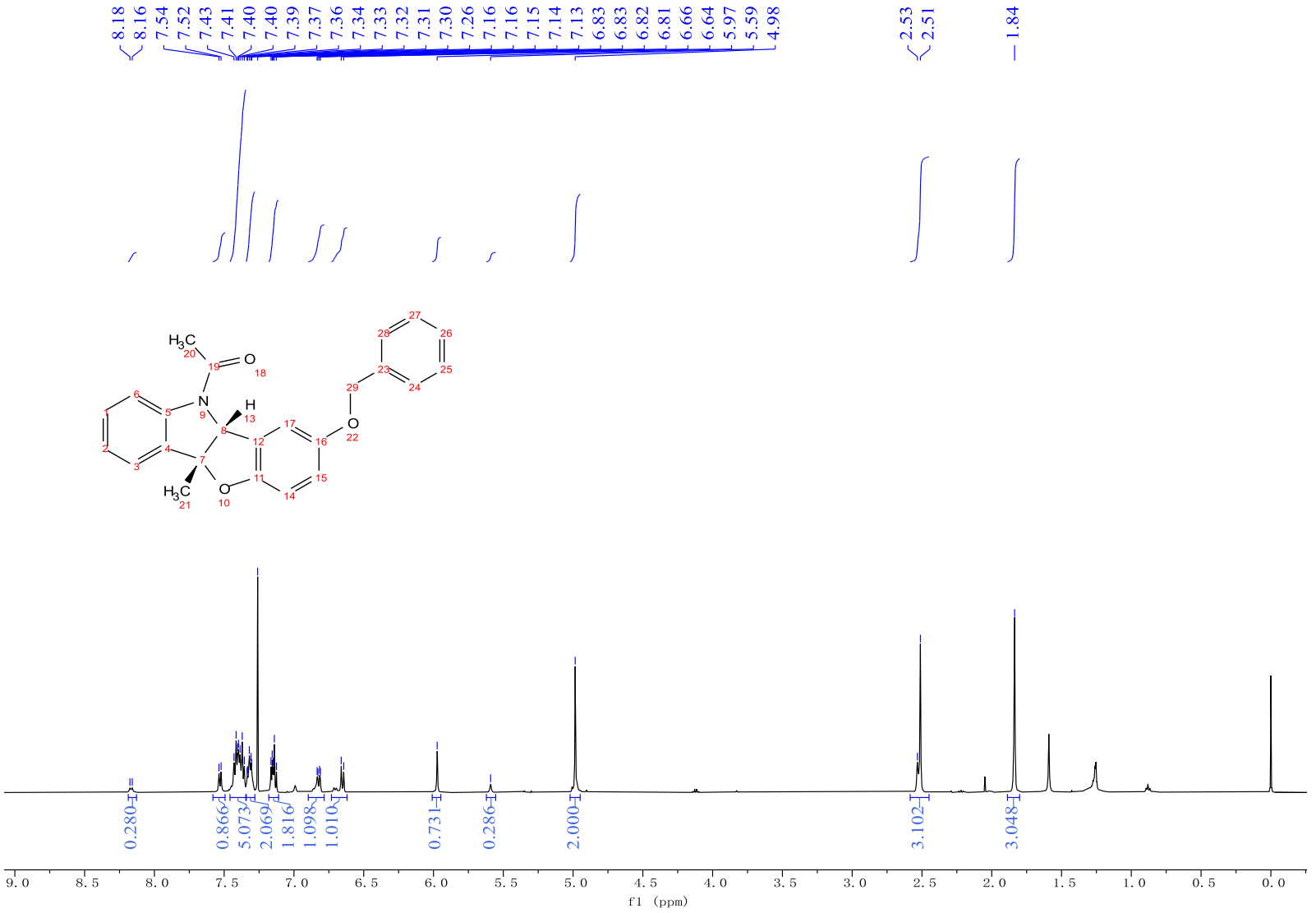
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6.83
6.82
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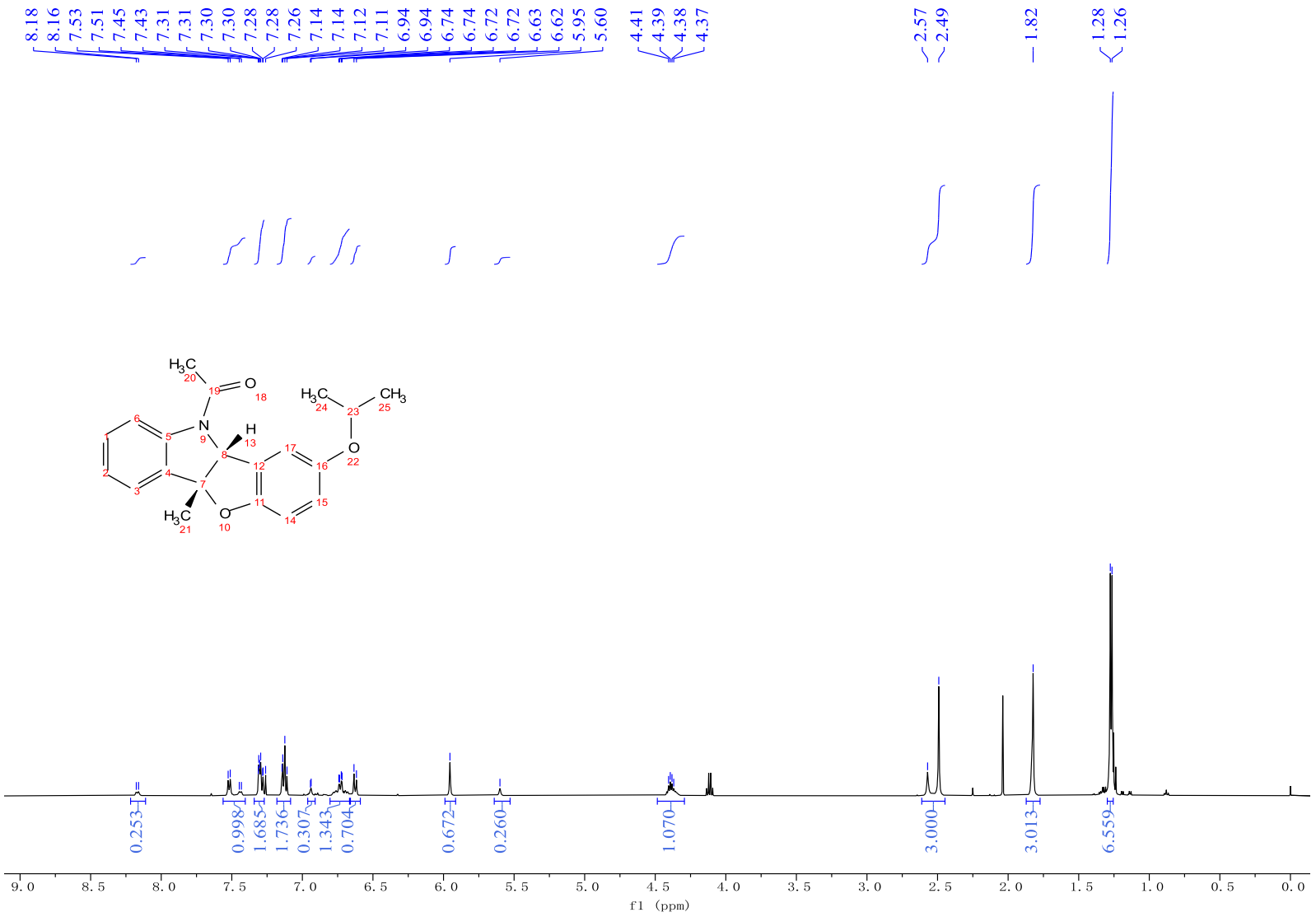
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7.12
7.11
6.94
6.94
6.74
6.74
6.72
6.72
6.63
6.62
5.95
5.60
4.41
4.39
4.38
4.37
2.57
2.49
1.82
1.28
1.26
H3C
20
19
O
18
H3C
24
23
CH3
25
6
5
N
9
H
13
17
O
22
2
4
8
12
16
7
3
11
15
H3C
21
O
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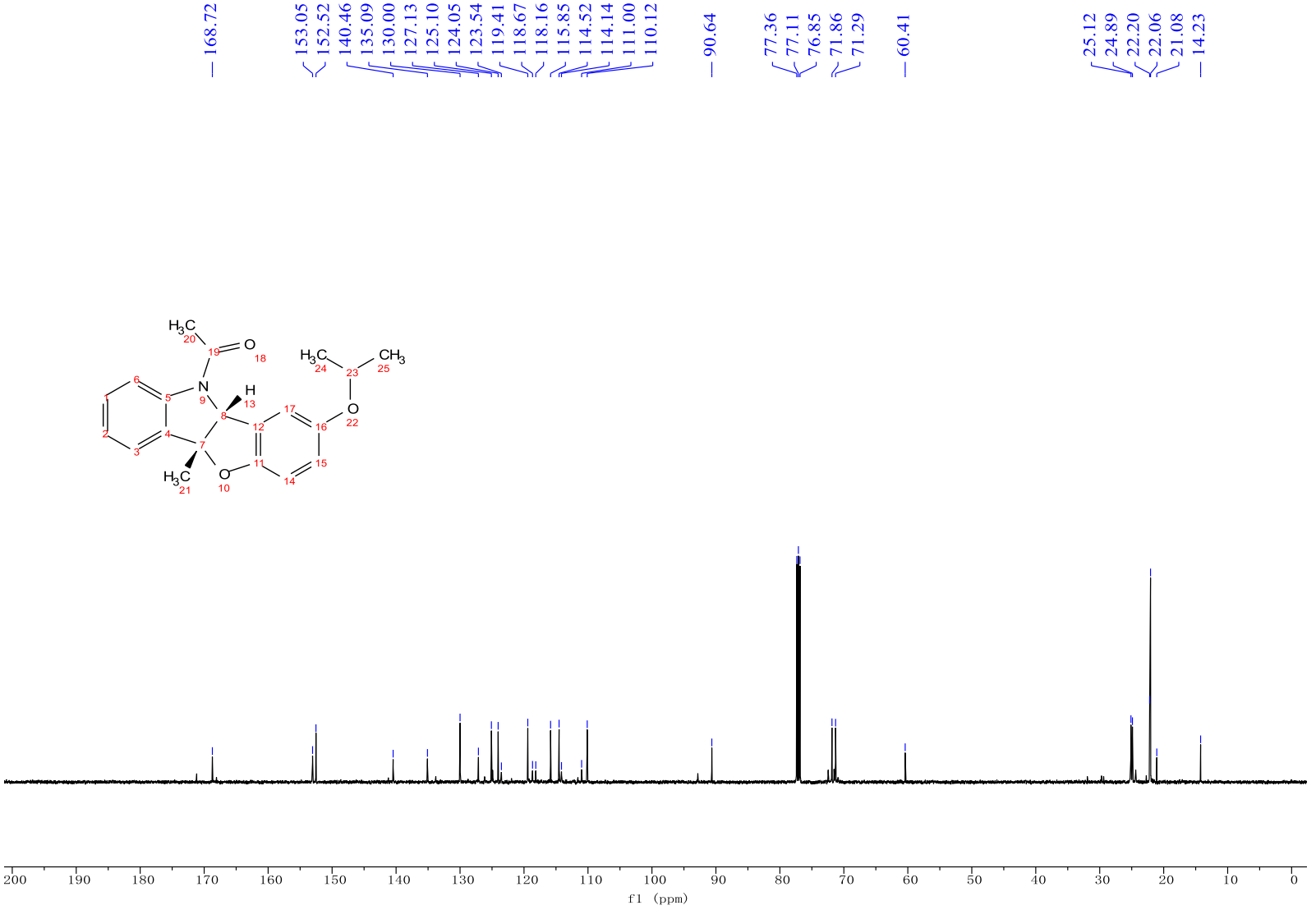
1.0
0.5
0.0
f1 (ppm)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114.52
114.14
111.00
110.12
90.64
77.36
77.11
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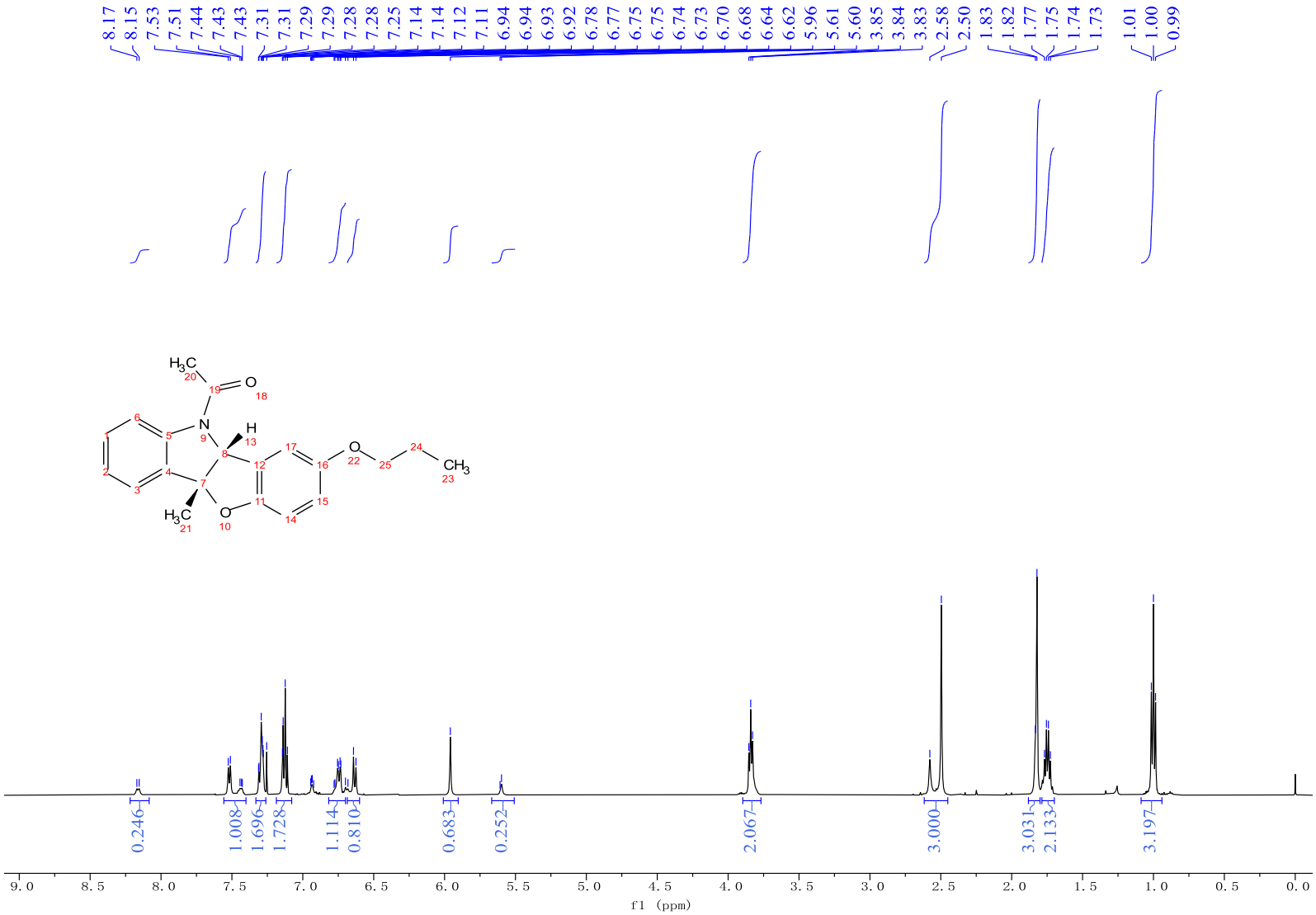
24.89
22.20
22.06
21.08
14.23
f1 (ppm)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7.14
7.12
7.11
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6.94
6.93
6.92
6.78
6.77
6.75
6.75
6.74
6.73
6.70
6.68
6.64
6.62
5.96
5.61
5.60
3.85
3.84
3.83
2.58
2.50
1.83
1.82
1.77
1.75
1.74
1.73
1.01
1.00
0.99
H3C
20
19
O
18
N
9
H
13
O
22
25
24
CH3
23
H3C
21
O
10
0.246
1.008
1.696
1.728
1.114
0.810
0.683
0.252
2.067
3.000
3.031
2.133
3.197
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8.0
7.5
7.0
6.5
6.0
5.5
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2.5
2.0
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1.0
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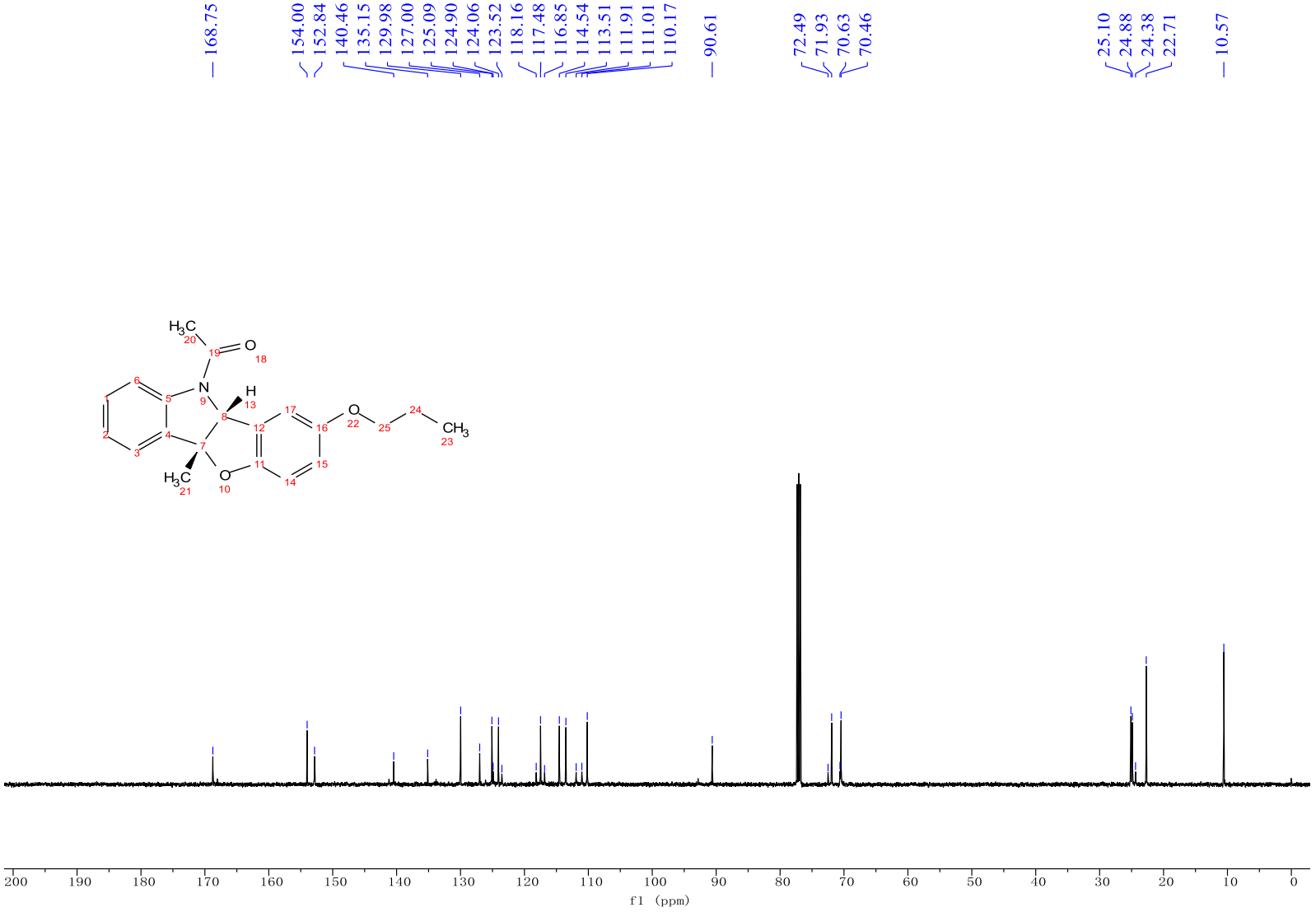
0.0
f1 (ppm)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114.54
113.51
111.91
111.01
110.17
90.61
72.49
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70.63
70.46
25.10
24.88
24.38
22.71
10.57
H3C
20
19
O
18
N
9
H
13
6
5
1
2
4
3
7
8
12
17
16
O
22
25
24
CH3
23
11
15
14
H3C
21
O
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200
190
180
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150
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130
120
110
100
90
80
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50
40
30
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10
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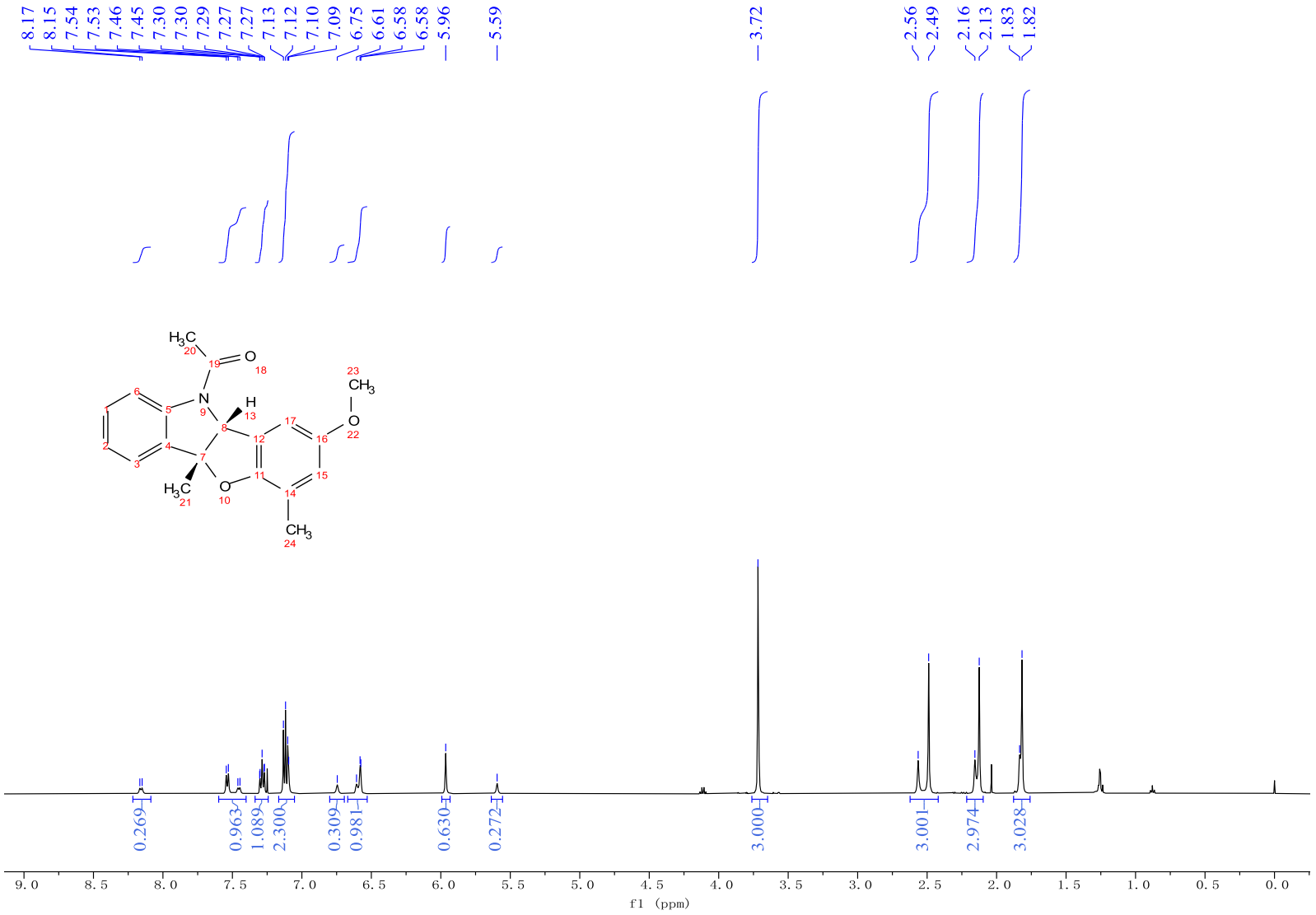
f1 (ppm)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7.30
7.29
7.27
7.27
7.13
7.12
7.10
7.09
6.75
6.61
6.58
6.58
5.96
5.59
3.72
2.56
2.49
2.16
2.13
1.83
1.82
0.269
0.963
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0.981
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0.272
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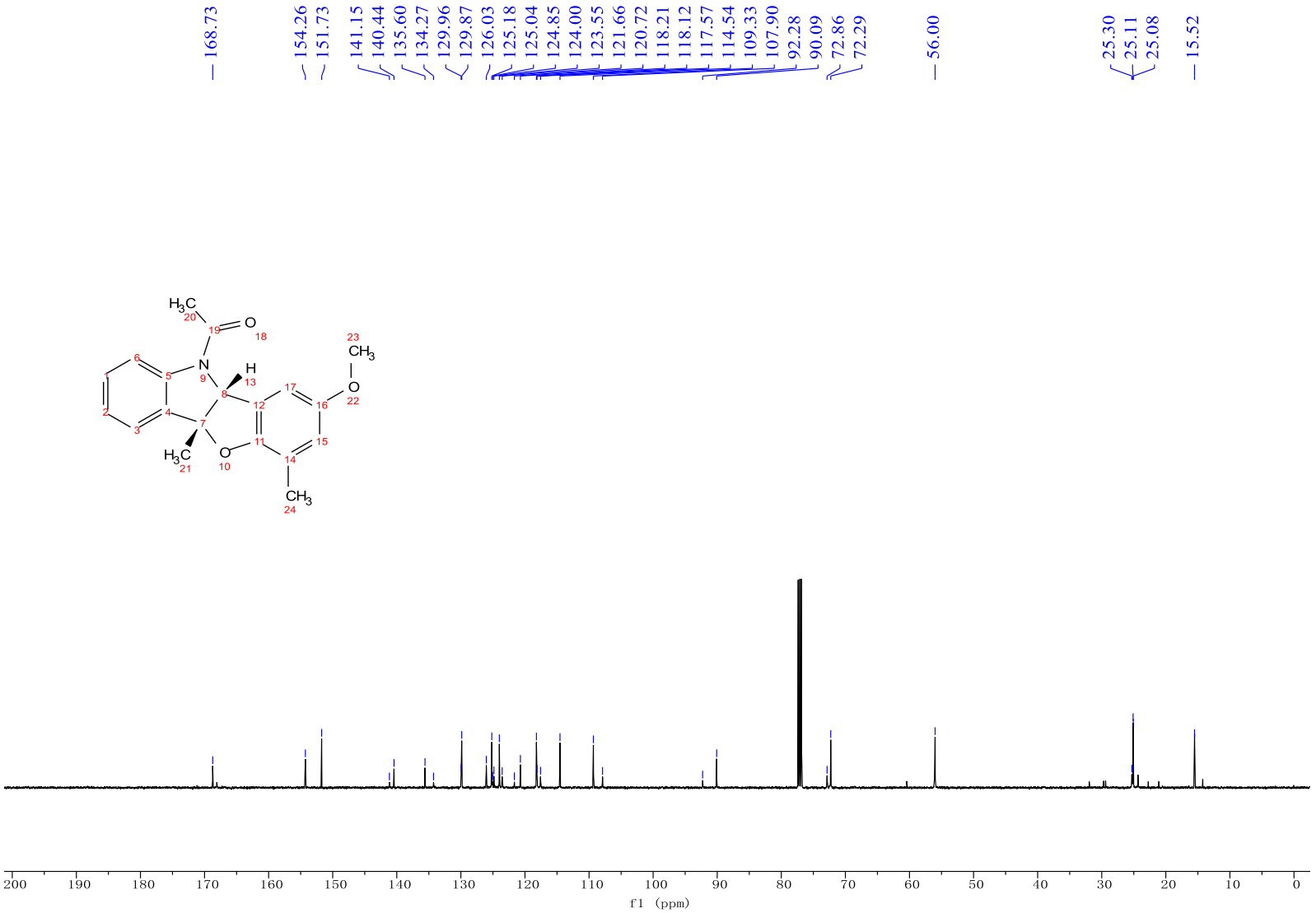
3.001
2.974
3.028
f1 (ppm)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124.00
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25.30
25.11
25.08
15.52
H3C
20
19
O
18
N
9
H
13
6
5
1
2
4
3
8
7
12
17
16
23
CH3
O
22
15
14
11
H3C
21
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10
CH3
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200
190
180
170
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130
120
110
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90
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70
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50
40
30
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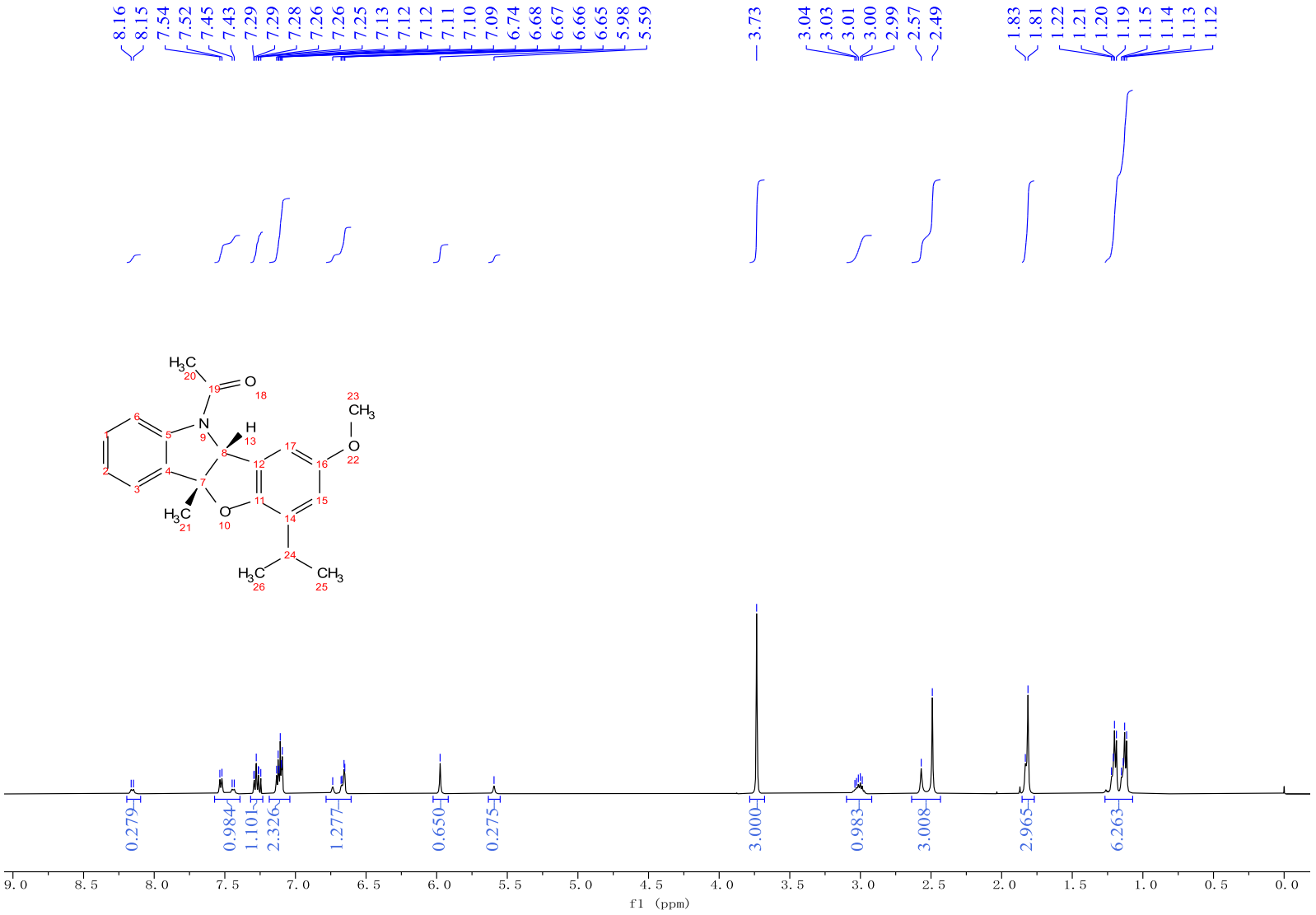
10
0
f1 (ppm)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7.13
7.12
7.12
7.11
7.10
7.09
6.74
6.68
6.67
6.66
6.65
5.98
5.59
3.73
3.04
3.03
3.01
3.00
2.99
2.57
2.49
1.83
1.81
1.22
1.21
1.20
1.19
1.15
1.14
1.13
1.12
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0.984
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0.275
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0.983
3.008
2.965
6.263
f1 (ppm)

168.74
168.02
154.54
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135.78
134.42
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129.81
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123.41
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114.60
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114.00
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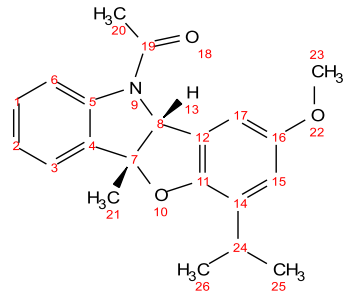
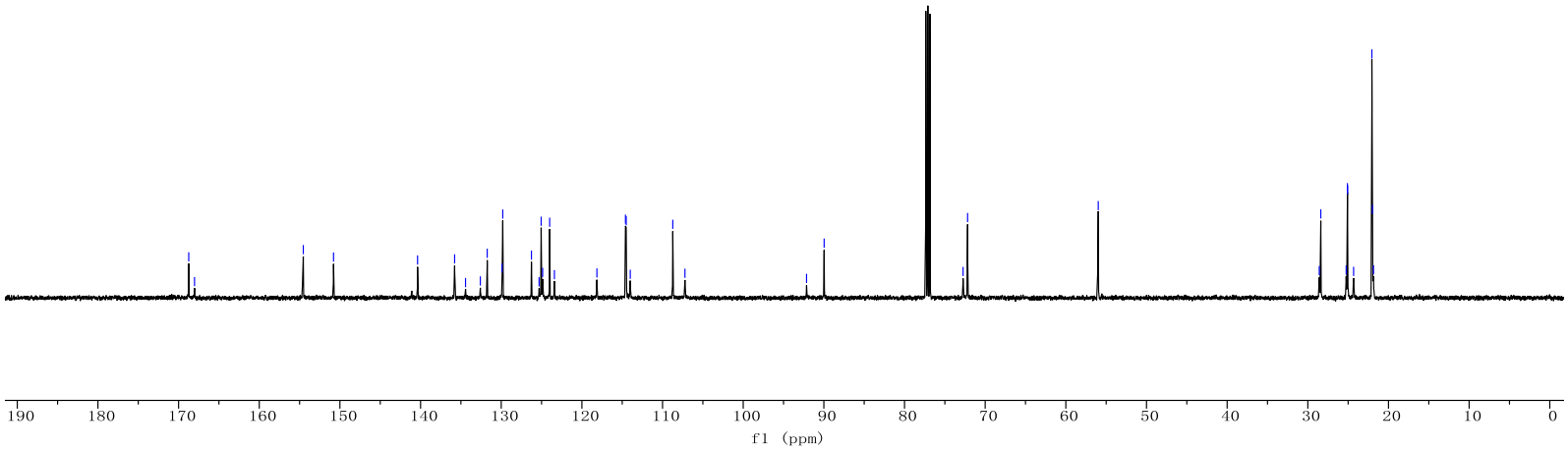
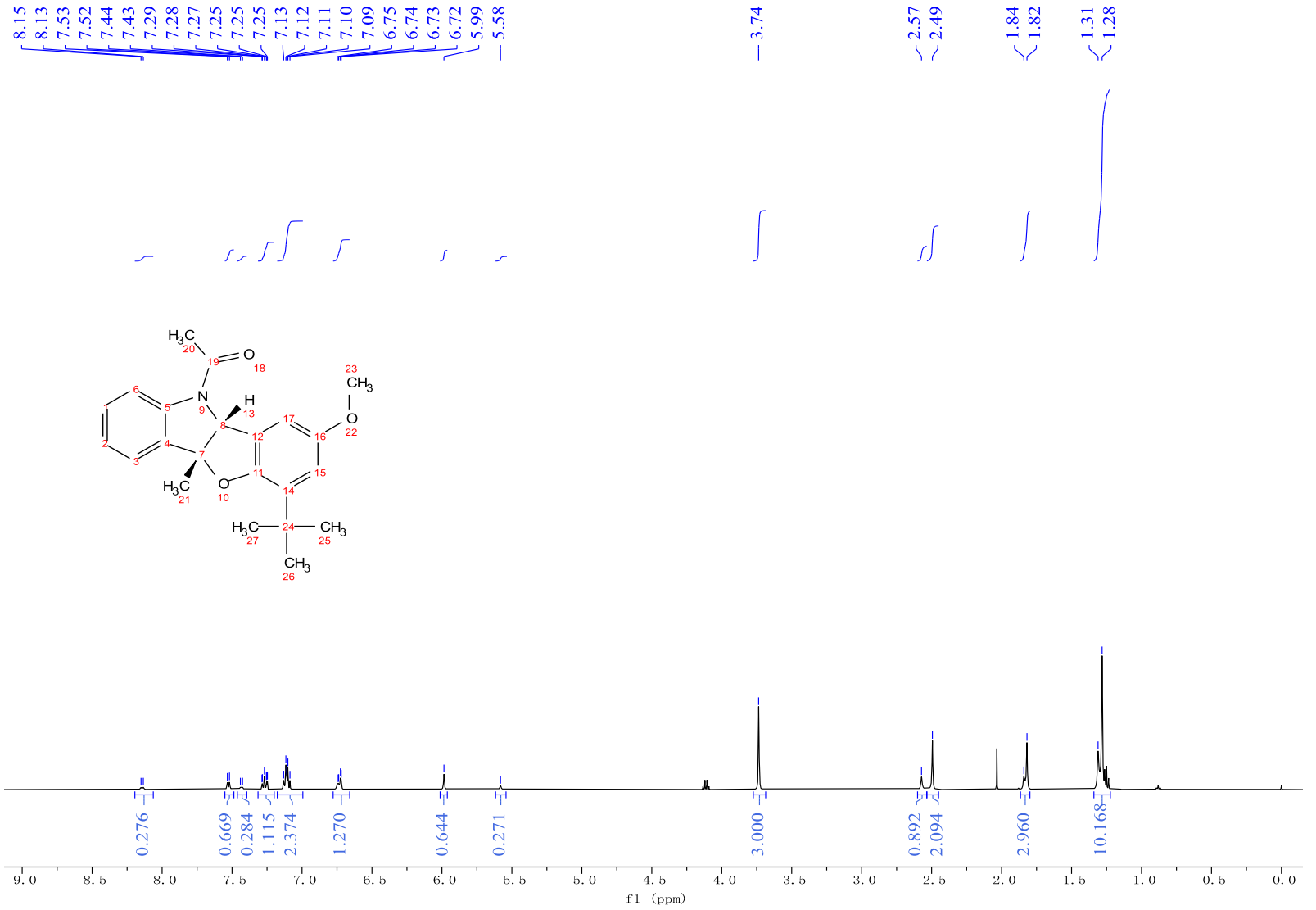
21.86



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7.10
7.09
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6.73
6.72
5.99
5.58
3.74
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0.669
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0.271
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2.094
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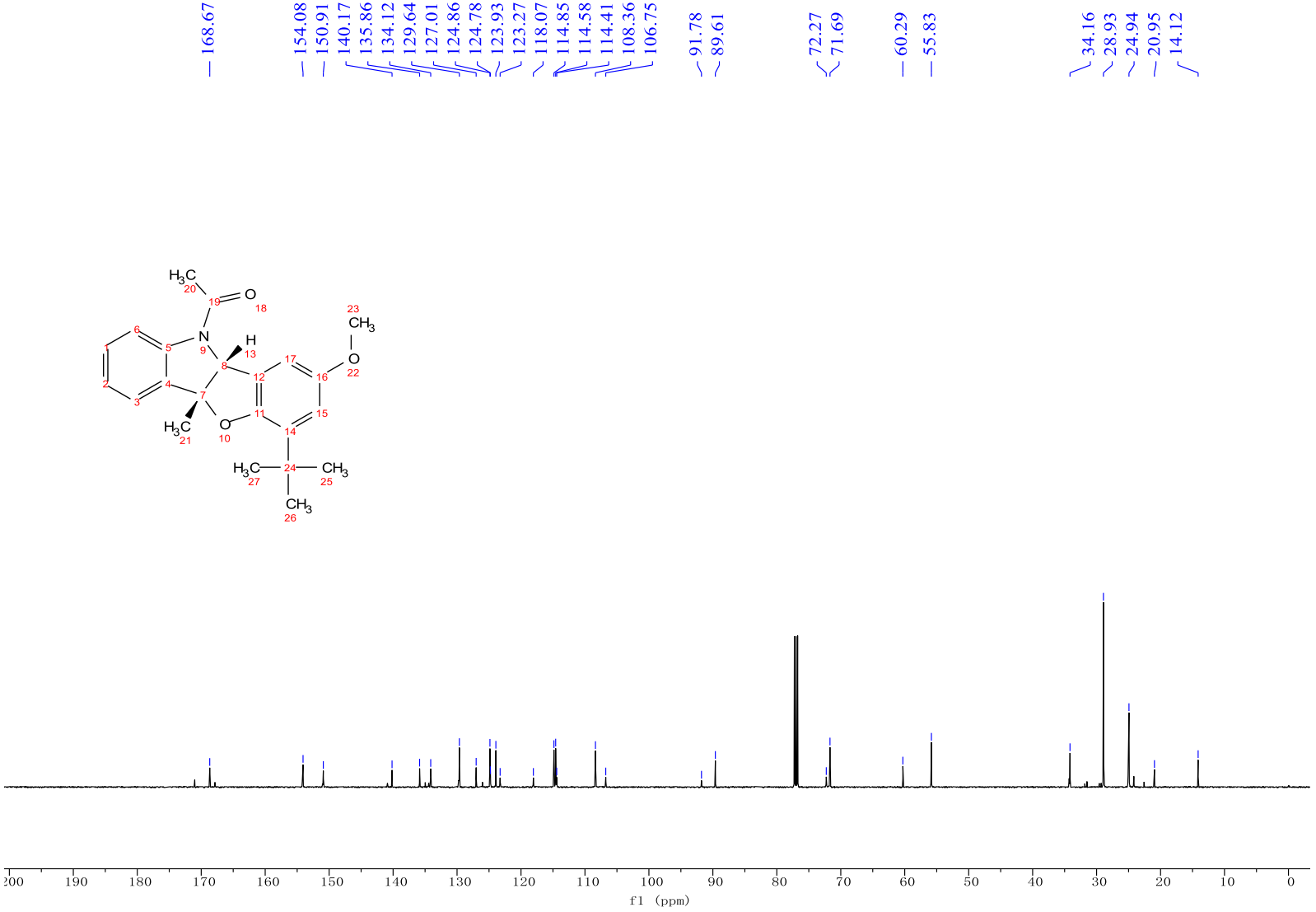
10.168
f1 (ppm)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114.58
114.41
108.36
106.75
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71.69
60.29
55.83
34.16
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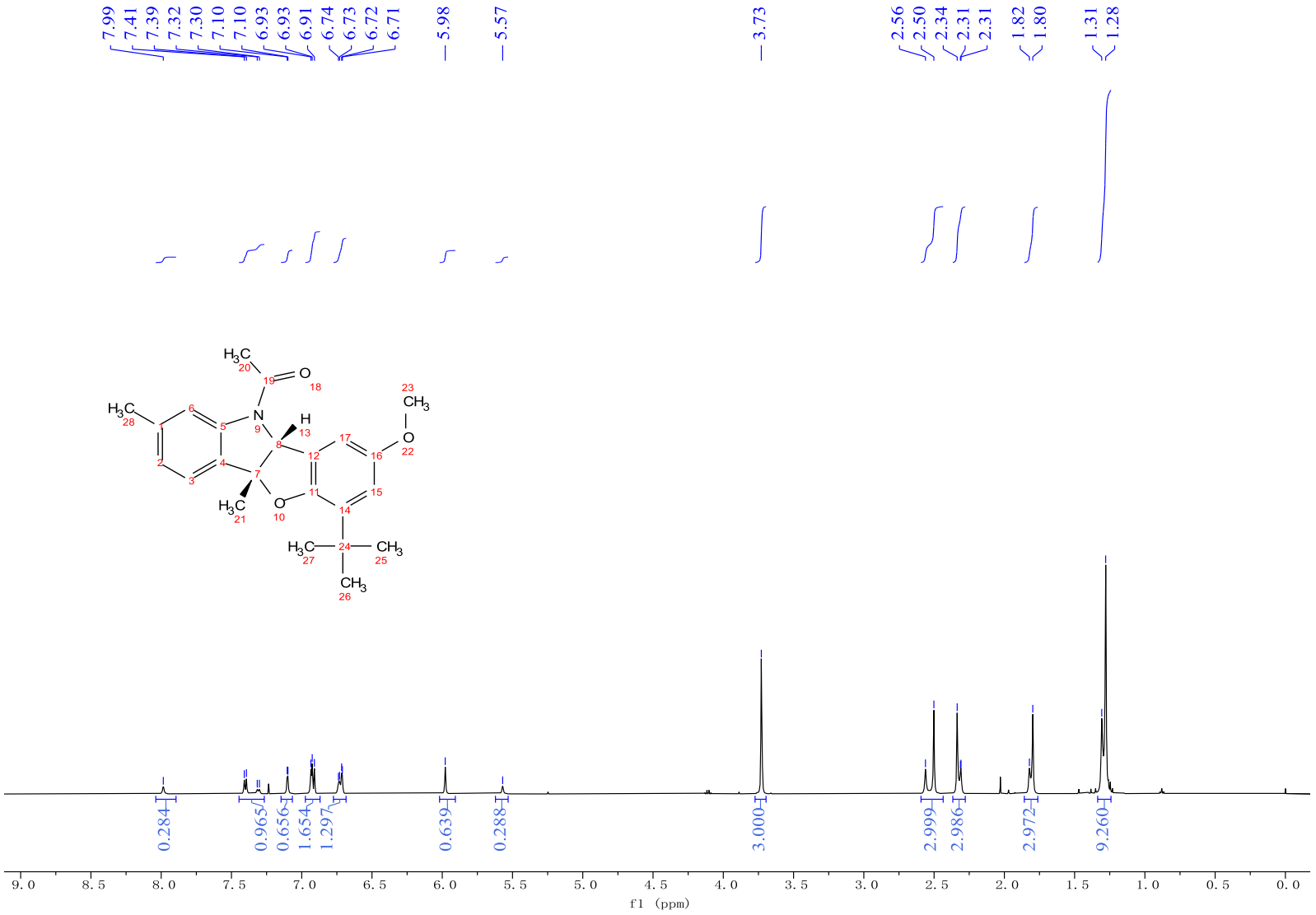
24.94
20.95
14.12
f1 (ppm)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6.91
6.74
6.73
6.72
6.71
5.98
5.57
3.73
2.56
2.50
2.34
2.31
2.31
1.82
1.80
1.31
1.28
0.284
0.965
0.656
1.654
1.297
0.639
0.288
3.000
2.999
2.986
2.972
9.260
f1 (ppm)

168.76
167.92
154.27
154.15
151.13
151.03
141.16
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140.07
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135.04
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133.29
131.82
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126.26
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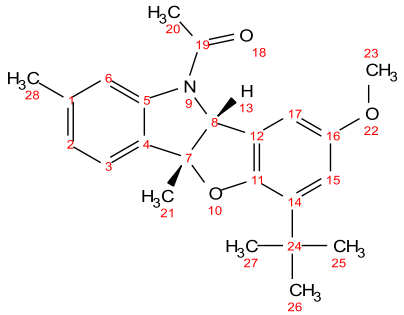
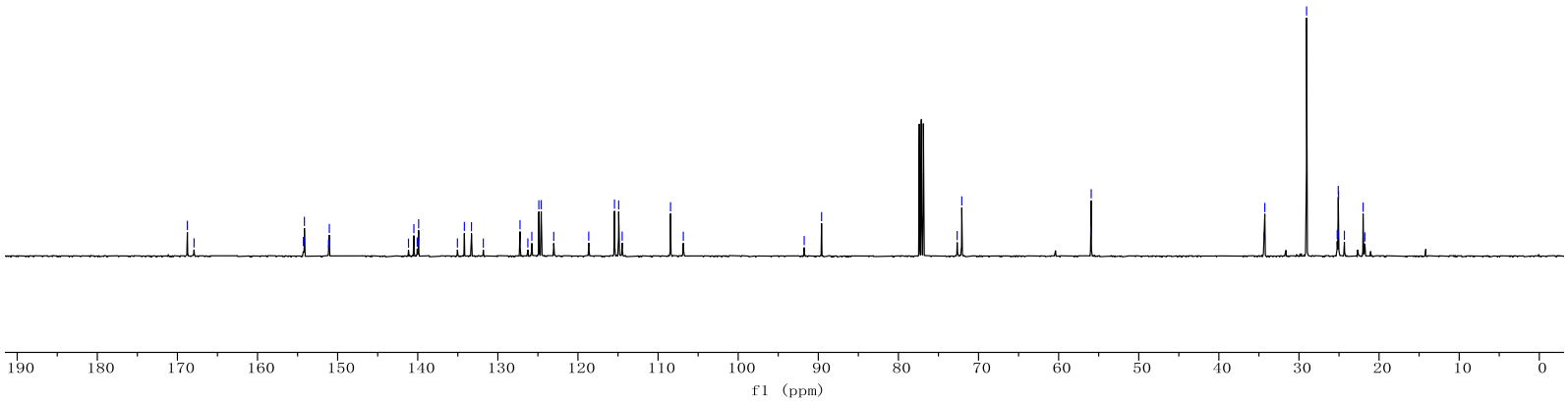
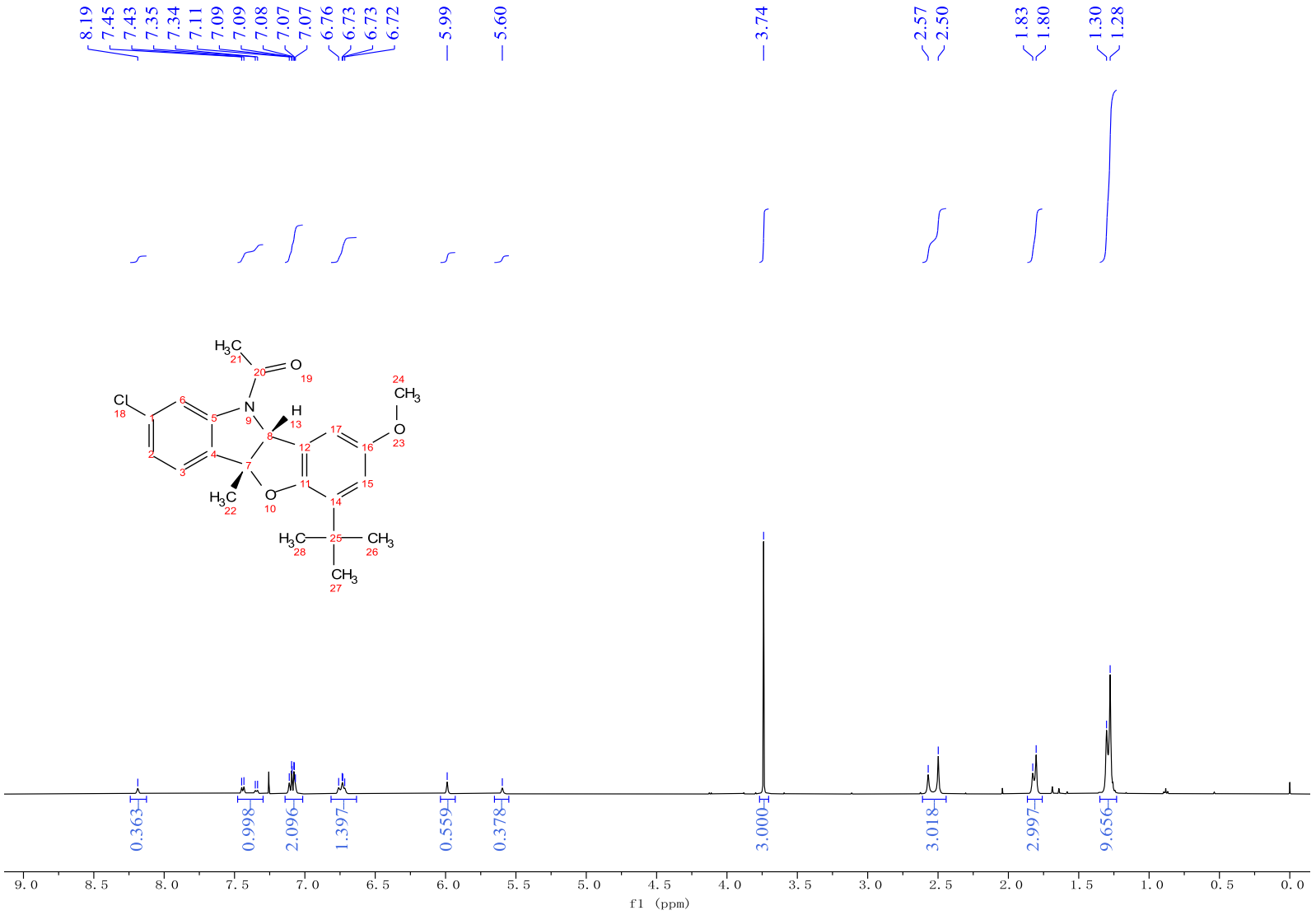
21.78



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7.09
7.08
7.07
7.07
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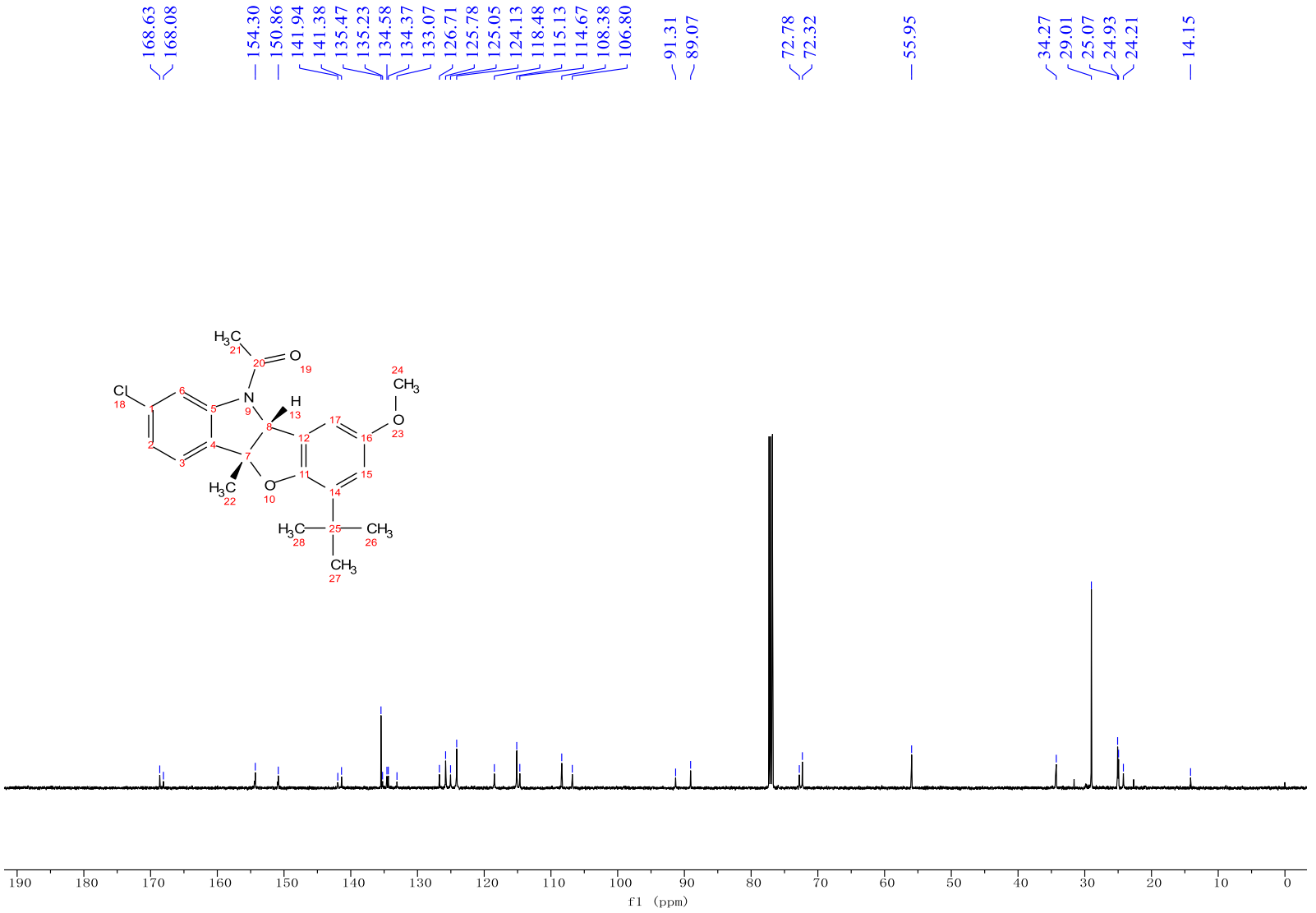
6.73
6.72
5.99
5.60
3.74
2.57
2.50
1.83
1.80
1.30
1.28
0.363
0.998
2.096
1.397
0.559
0.378
3.000
3.018
2.997
9.656
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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125.05
124.13
118.48
115.13
114.67
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106.80
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29.01
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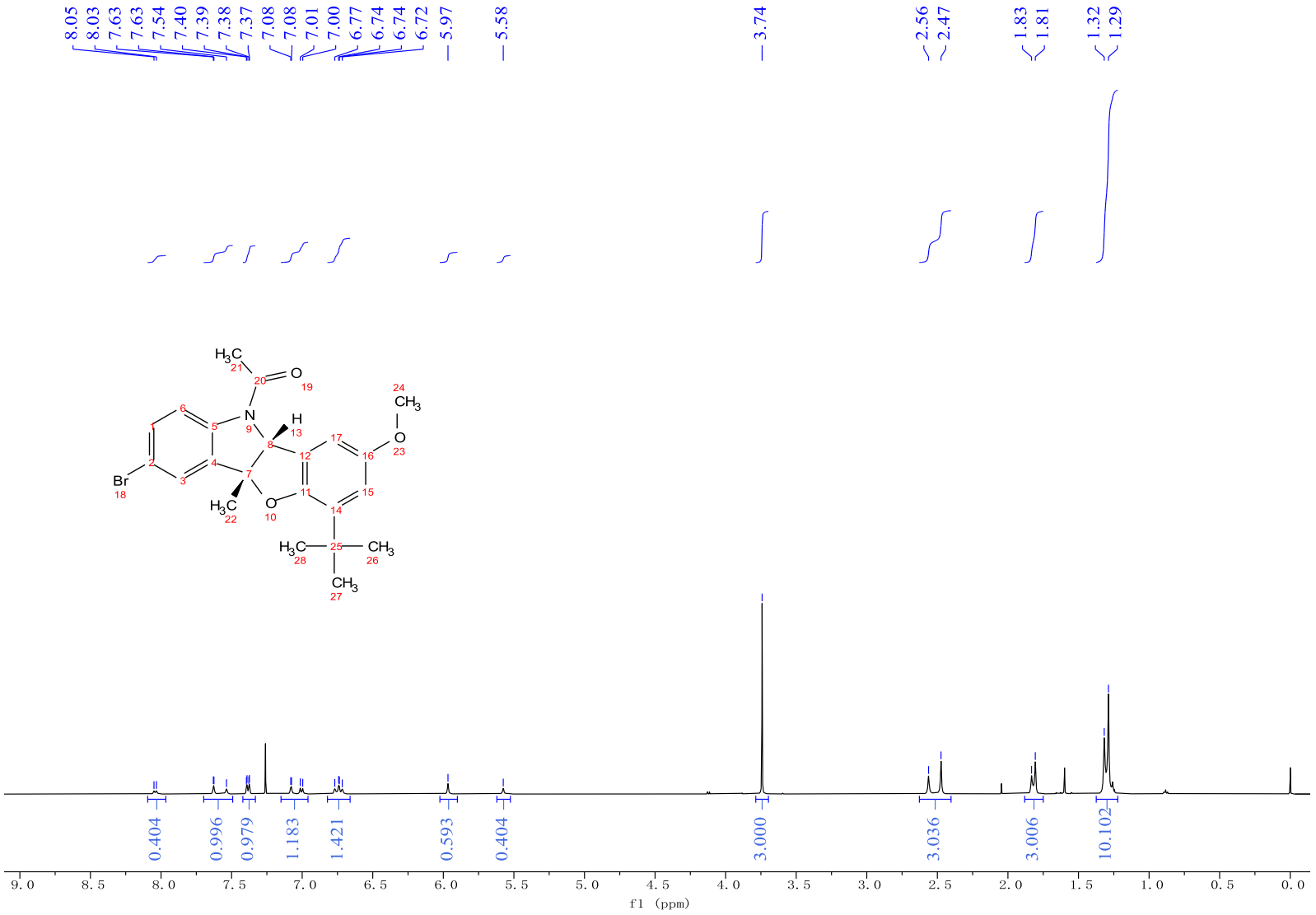
24.21
14.15
f1 (ppm)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6.74
6.72
5.97
5.58
3.74
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2.47
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1.81
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1.29
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0.996
0.979
1.183
1.421
0.593
0.404
3.000
3.036
3.006
10.102
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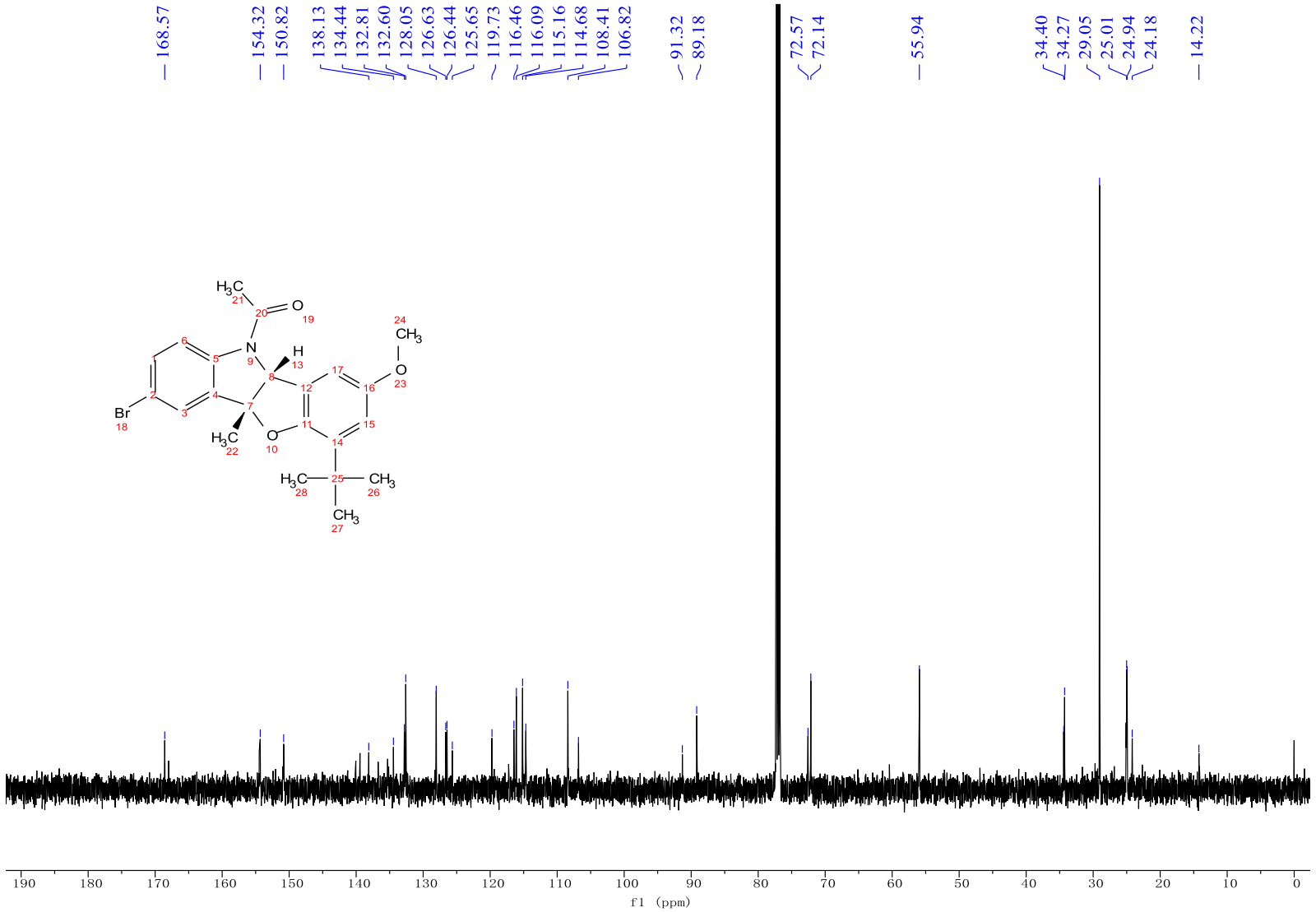
f1 (ppm)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115.16
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108.41
106.82
91.32
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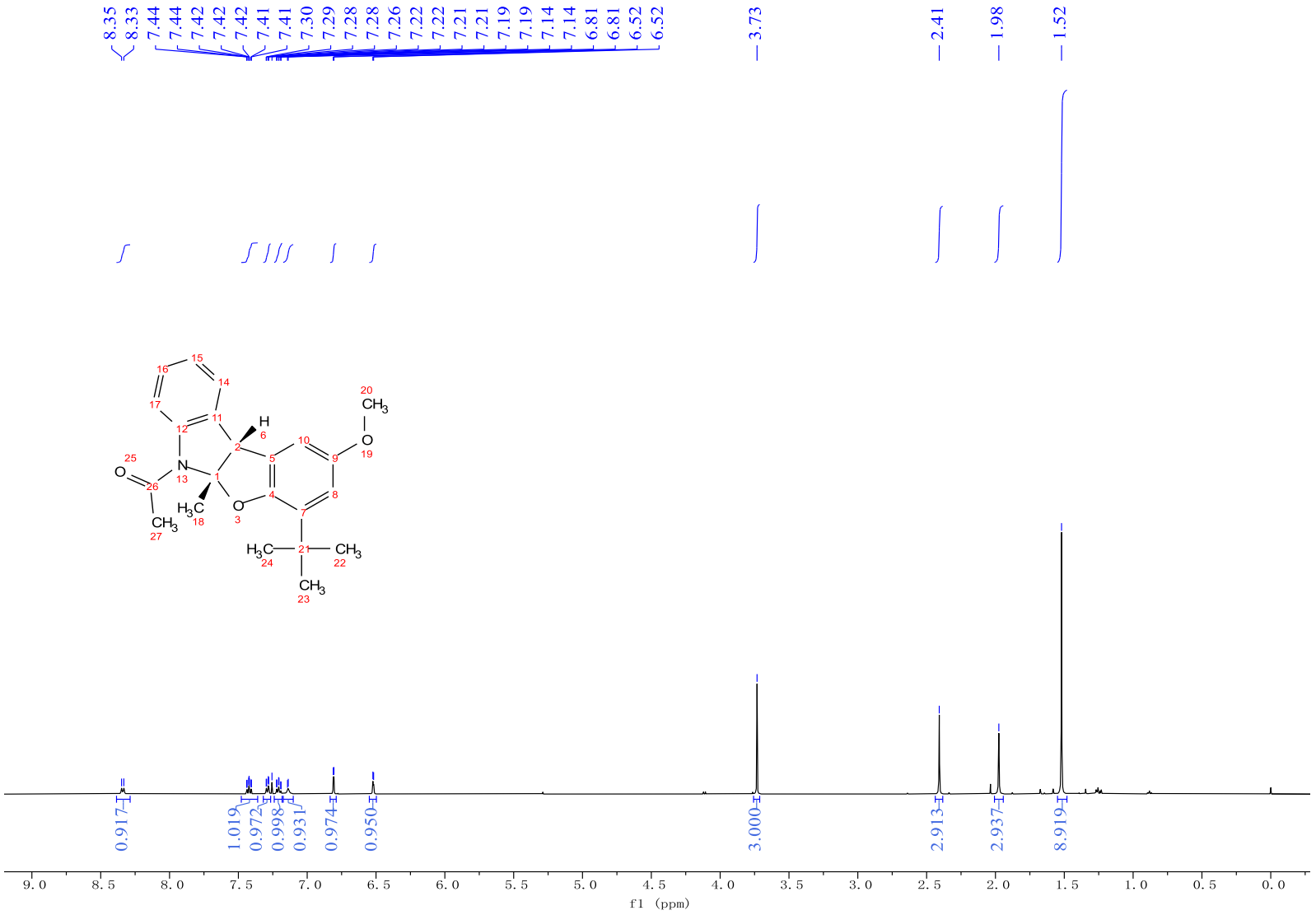
34.27
29.05
25.01
24.94
24.18
14.22
f1 (ppm)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1.019
0.972
0.998
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0.950
3.000
2.913
2.937
8.919
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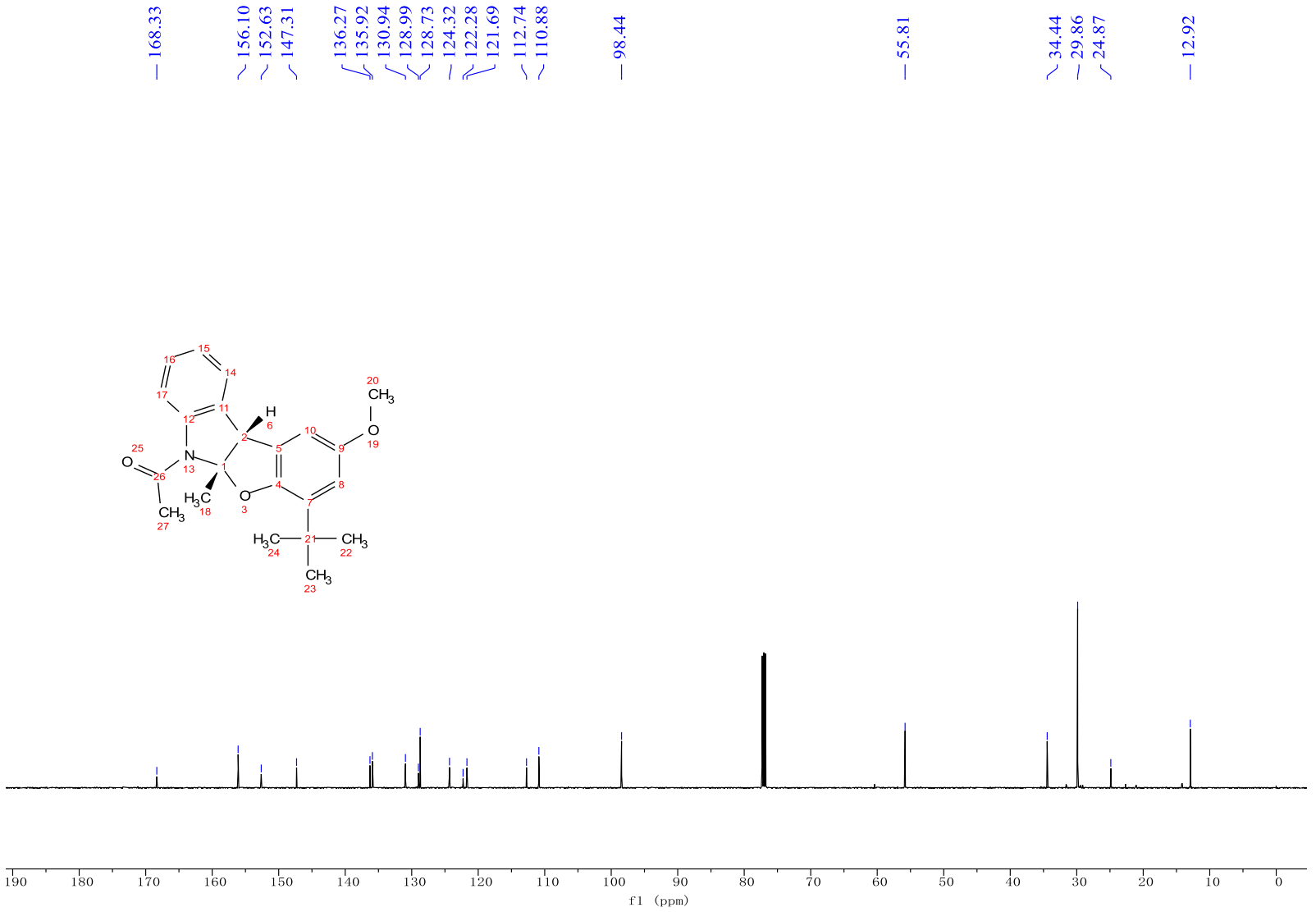
f1 (ppm)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112.74
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180
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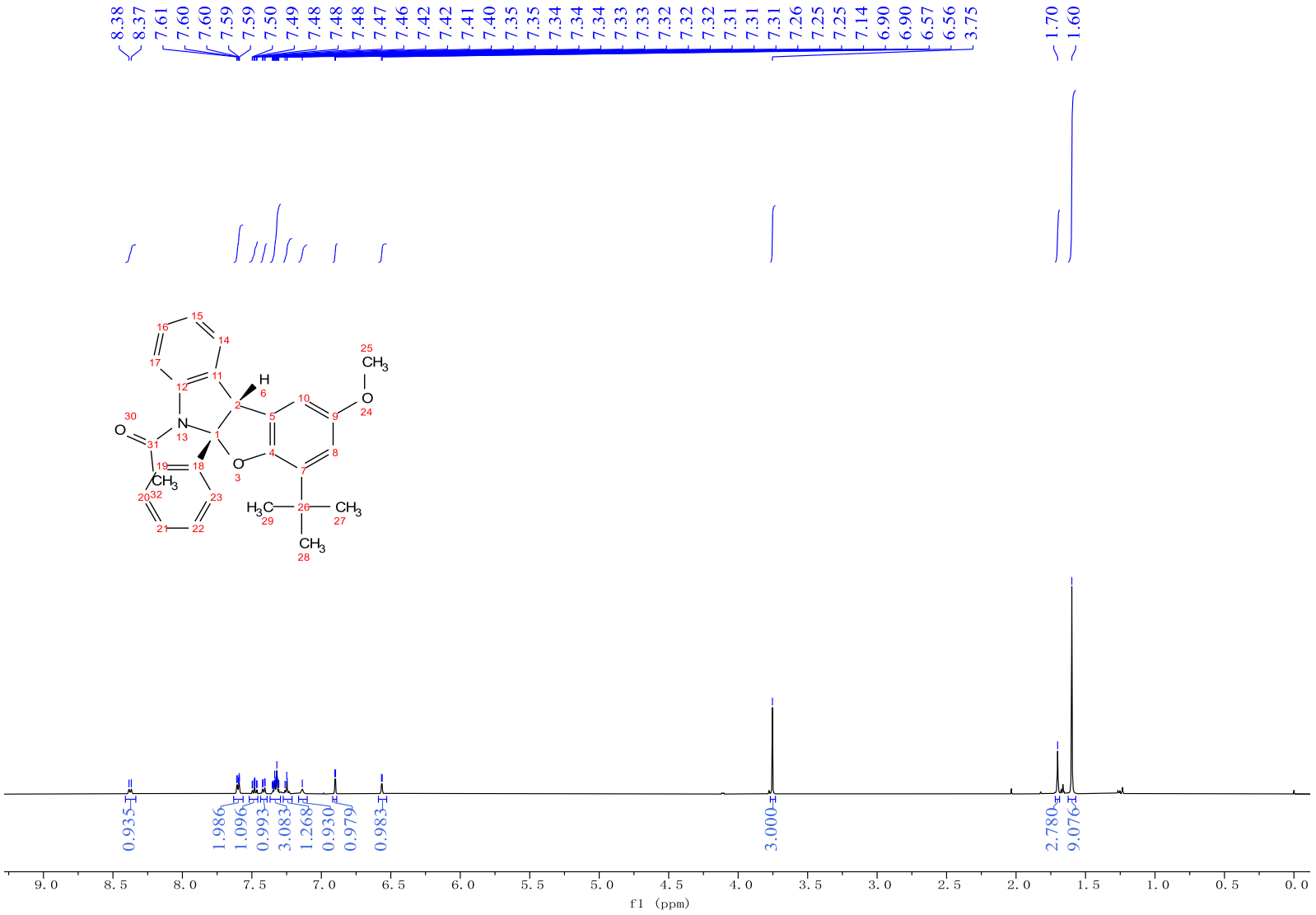
f1 (ppm)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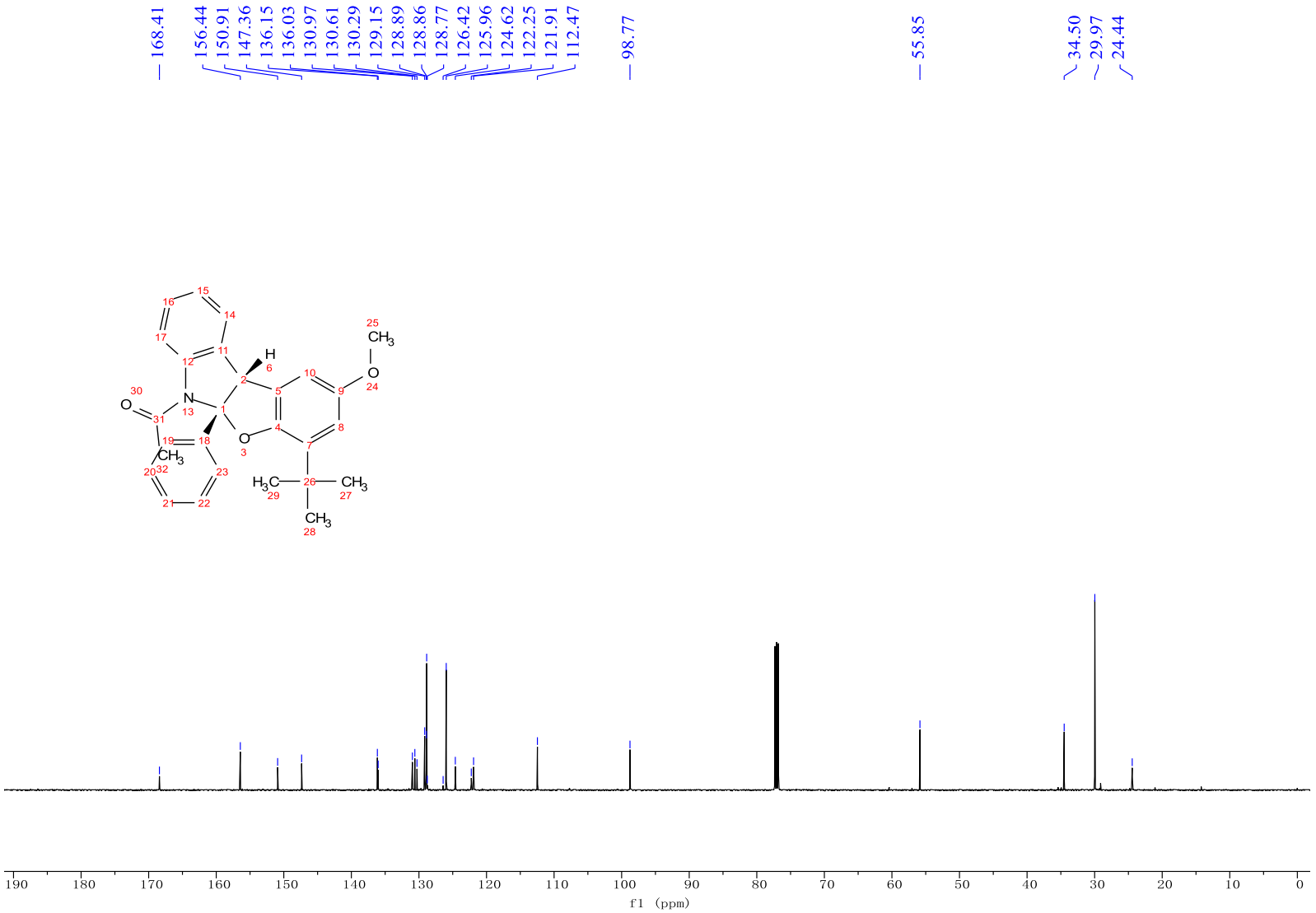
2.780
9.076
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


168.41
156.44
150.91
147.36
136.15
136.03
130.97
130.61
130.29
129.15
128.89
128.86
128.77
126.42
125.96
124.62
122.25
121.91
112.47
98.77
55.85
34.50
29.97
24.44
f1 (ppm)

S5. References

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