Electronic Supplementary Material (ESI) for Green Chemistry.

Benign catalysis with iron: facile assembly of cyclobutanes and cyclohexenes via intermolecular radical cation cycloadditions

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Supporting Information

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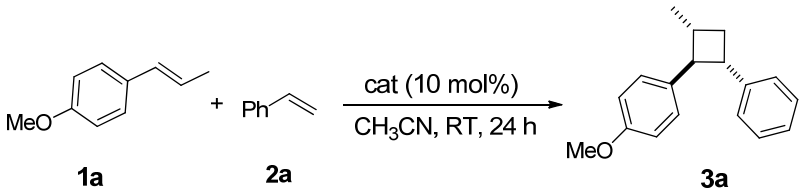
1. General Information.

Chemicals and solvents were purchased from commercial suppliers and used as received unless noted. All products were purified by flash chromatography on silica gel. The chemical yields referred are isolated products. ^{1}H NMR and ^{13}C NMR spectra were recorded on 400 MHz or 600 MHz Bruker spectrometers. Chemical shifts of 1 H were reported in part per million relative to the $CDCl_3$ residual peak (δ 7.26). Chemical shifts of ^{13}C NMR were reported relative to $CDCl_3$ (δ 77.16). The used abbreviations are as follows: s (singlet), d (doublet), t (triplet), q (quartet), quint.(quintet), m (multiplet), br (broad). Multiplets which arise from accidental equality of coupling constants of magnetically non-equivalent protons are marked as virtual (*virt.*). Infrared spectra were recorded on a Perkin Elmer FT-IR Spectrum. High resolution mass spectra (HRMS) data were measured on a APCI-micro TOF. Reactions were monitored by TLC analysis using silica gel 60 Å F-254 thin layer plates and compounds were visualized with a UV light at 254 nm or 365 nm. Flash column chromatography was performed on silica gel 60Å, 10-40μm.

2. Reaction Condition Optimizations.

2.1 Table S1. Catalyst screening.

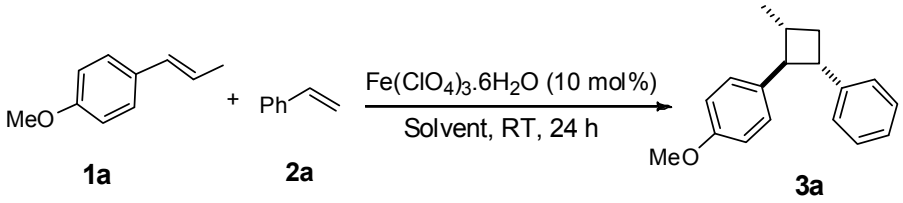


1a **2a** **3a**

Entry[a]	Catalyst	Yield(%)[b]
1	$Fe(ClO_4)_3.6H_2O$	52
2	$FeCl_3$	46
3	$FeCl_3{\cdot}6H_2O$	29
4	$Fe(NO_3)_3{\cdot}9H_2O$	20%
5	$Cu(ClO_4)_2{\cdot}6H_2O$	34%
6	$Cu(NO_3)_2{\cdot}3H_20$	0
7	Cu(TMEDA)Cl	0
8	$NiCl_2$	0
9	$AgNO_3$	0
10	MnO_2	0
11	$Fe(acac)_3$	0
12	$Fe_2(SO_4)_3{\cdot}XH_2O$	0
13	$FeBr_3$	0

[a] Reaction conditions: all of the reactions were performed with **2a** (286 μL, 2.0 mmol), catalyst (10 mol%) in 3.0 mL CH_3CN and a solution of **1a** (148 μL, 1.0 mmol) in 1.0 ml CH_3CN was added using a syringe pump (at a rate of 1.0 mL/h) under air. [b]Yields are for isolated products.

2.2 Table S2. Solvent screening.

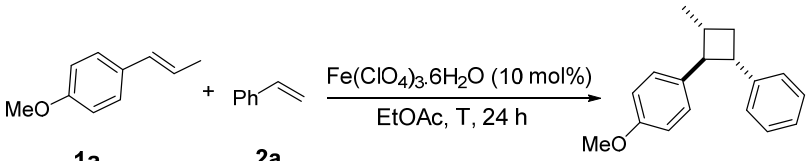


1a **2a** **3a**

Entry[a]	Solvent	Yield(%)[b]
1	$MeNO_2$	0
2	DMF	0
3	DMSO	0
4	THF	22
5	1,4-dioxane	0
6	EtOAc	62
7	MeOAc	49
8	2-Propyl acetate	52
9	CH_3CN	52
10	CH_2Cl_2	18
11	CH_3Cl	0
12	Et_2O	0
13	MeOH	0
14	HFIPA	0
15	Toluene	0
16	DCE	0
17	PhCl	0

[a]Reaction conditions: all of the reactions were performed with **2a** (286 μL, 2.0 mmol), $Fe(ClO_4)_3{\cdot}6H_2O$ (10 mol %, 35.4 mg) in 3.0 mL solvent and a solution of **1a** (148 μL, 1.0 mmol) in 1.0 mL solvent was added using a syringe pump (at a rate of 1.0 mL/h) under air. [b]Yields are for isolated products.

2.3 Table S3. The effect of reaction temperature.



1a 2a 3a

Entry[a]	T(°C)	Yield (%)[b]
1	35	87
2	40	88
3	45	83
4	50	79

[a]Reaction conditions: all of the reactions were performed with **2a** (576 μL, 5.0 mmol), $Fe(ClO_4)_3{\cdot}6H_2O$ (10 mol%, 35.4 mg) in 3.0 mL EtOAc and a solution of **1a** (148 μL, 1.0 mmol) in 1.0 mL EtOAc was added using a syringe pump (at a rate of 1.0 ml/h) at specified temperature. [b]Yields are for isolated products.

3. General procedures.

3.1. *General Procedure 1* for the homodimerization of styrenes.

To a solution of styrene **1** (1.0 mmol) in 4.0 mL CH_3CN, $Fe(ClO_4)_3{\cdot}6H_2O$ (35.4 mg, 10 mol%) was added in one portion. The mixture was stirred at 40 °C for 24 hours under Air. Then, the solvent was evaporated under reduced pressure to give the crude product, which was purified by chromatography on silica gel using the petroleum ether as the eluent.

3.2. *General procedure 2* for the cross dimerization of styrenes.

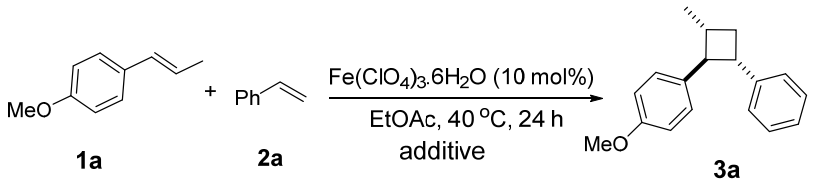
To a solution of the styrene **2** (5.0 mmol, 5.0 eq) in 3.0 mL EtOAc was added $Fe(ClO_4)_3{\cdot}6H_2O$ (35.4 mg, 10 mol%), then a solution of styrene **1** (1.0 mmol) in 1.0 mL EtOAc was added using a syringe pump (at a rate of 1.0 ml/h), The resulting mixture was stirred at 40 °C for 24 hours under air. Then, the solvent was evaporated under reduced pressure to give the crude product, which was purified by chromatography on silica gel using the petroleum ether as the eluent.

3.3. *General procedure 3* for the Diels–Alder Cycloadditions.

To a solution of the diene **4** (2.0 mmol, 2.0 eq) in 3.0 mL CH_3CN, was added $Fe(ClO_4)_3{\cdot}6H_2O$ (35.4 mg, 10 mol%), then a solution of styrene **1** (1.0 mmol) in1.0 mL CH_3CN was added using a syringe pump (at a rate of 1.0 mL/h), The resulting mixture was stirred at room temperature for 24 hours under air. Then, the solvent was evaporated under reduced pressure to give the crude product, which was purified by chromatography on silica gel using the petroleum ether as the eluent.

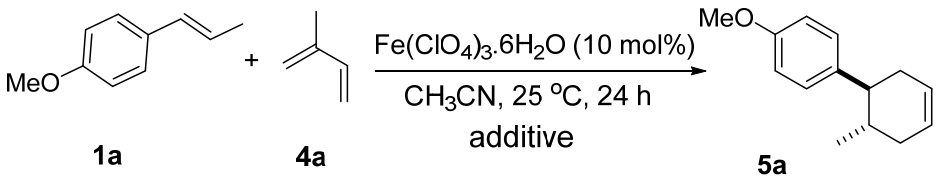
4. Mechanistic studies.

4.1 The radical trapping experiments.



additive (1.0 eq)	yield of **3a**
TEMPO	0%
BHT	0%

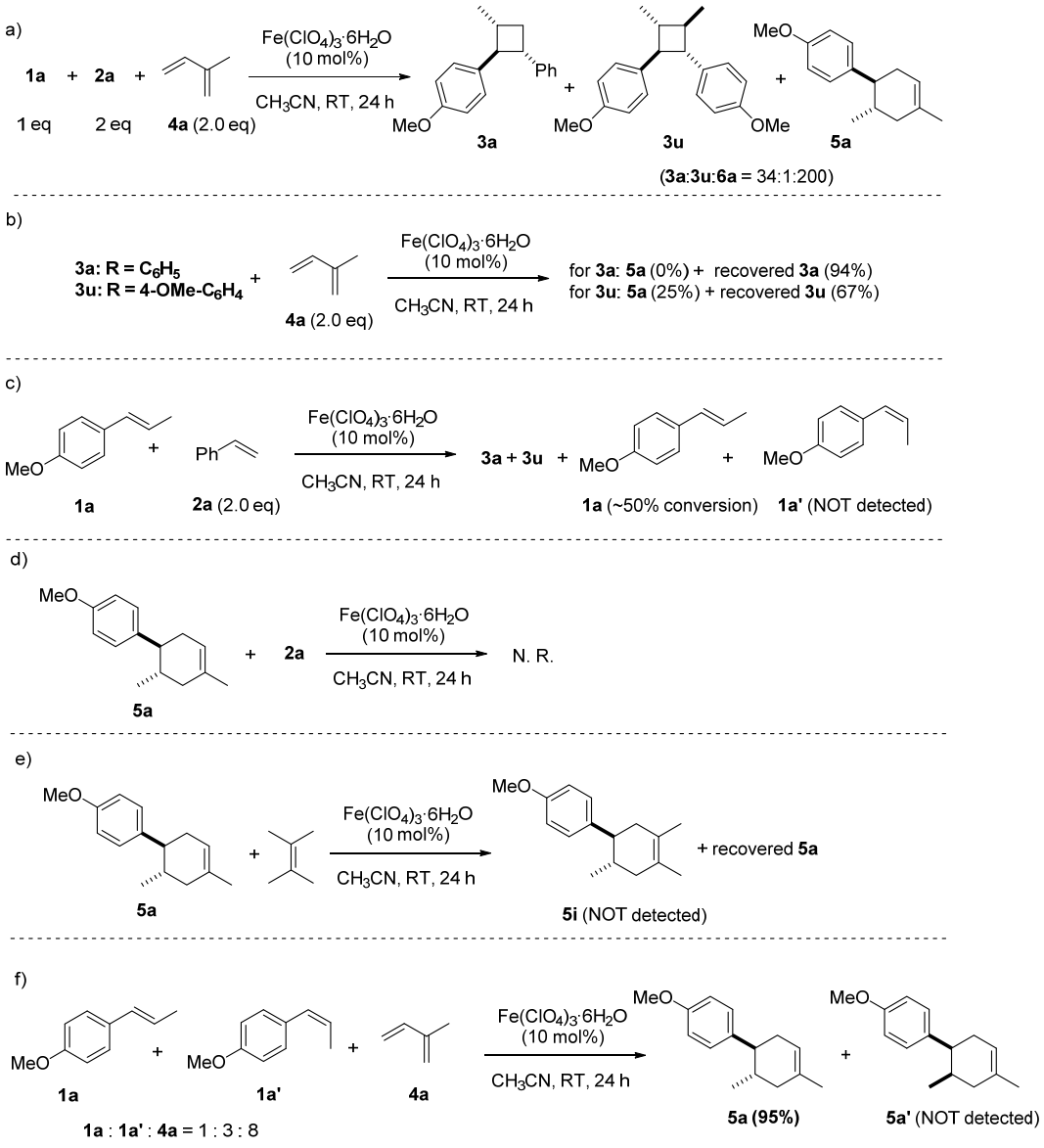
To a solution of the styrene **2a** (143 µL, 1.25 mmol, 5.0 eqiv) in 1.0 mL EtOAc, was added to anthole **1a** (37.5 µL, 0.25 mmol) and $Fe(ClO_4)_3{\cdot}6H_2O$ (8.8 mg, 10 mol%), then, the additive (0.25 mmol, 1.0 eq) was added to the mixture. TLC and ^1H NMR confirmed that no reaction took place and only starting materials were detected.



additive (1.0 eq)	yield of **5a**
TEMPO	0%
BHT	0%

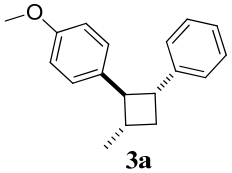
To a solution of the diene **4a** (50 µL, 0.5 mmol, 2.0 eqiv) in 1.0 mL CH_3CN, was added to anthole **1a** (37.5 µL, 0.25 mmol) and $Fe(ClO_4)_3{\cdot}6H_2O$ (8.8 mg, 10 mol%), then, the additive (0.25 mmol, 1.0 equiv) was added to the mixture. TLC and ^1H NMR confirmed that no reaction took place and only starting materials were detected.

4.2 Crossover experiments.



5. Analytical data of all of the products.

1-Methoxy-4-[(1*RS*,2*SR*,4*RS*)-2-methyl-4-phenylcyclobutyl]benzene (3a)[1]



3a

Compound **3a** was synthesized following the *General procedure* 2.

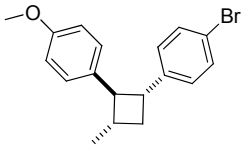
A colorless oil, 88% yield.

TLC: R_f = 0.53 (Hexane).

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.40 – 7.33 (m, 2H), 7.31 – 7.25 (m, 5H), 6.95 (d, *J* = 6.7 Hz, 2H), 3.87 (s, 3H), 3.54 – 3.43 (m, 1H), 3.05 (t, *J* = 9.5 Hz, 1H), 2.66 – 2.55 (m, 1H), 2.44 (d, *J* = 7.3 Hz, 1H), 1.80 (q, *J* = 10.1 Hz, 1H), 1.29 (d, *J* = 6.5 Hz, 3H).

The spectra data are matched with those reported[1].

1-Bromo-4-[(1*RS*,2*RS*,3*SR*)-2-(4-methoxyphenyl)-3-methylcyclobutyl]benzene (3b)[1]



3b

Compound **3b** was synthesized following the *General procedure* 2.

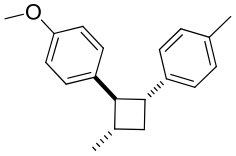
A colorless oil, 75% yield.

TLC: R_f = 0.49 (Hexane).

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.43 (d, *J* = 8.4 Hz, 2H), 7.20 (d, *J* = 8.6 Hz, 2H), 7.10 (d, *J* = 8.3 Hz, 2H), 6.91 (d, *J* = 8.7 Hz, 2H), 3.84 (s, 3H), 3.37 (q, *J* = 9.9 Hz, 1H), 2.93 (s, 1H), 2.63 – 2.51 (m, 1H), 2.45 – 2.32 (m, 1H), 1.70 (q, *J* = 10.1 Hz, 1H), 1.23 (d, *J* = 6.5 Hz, 3H).

The spectra data are matched with those reported[1].

1-Methoxy-4-[(1*RS*,2*SR*,4*RS*)-2-methyl-4-(p-tolyl)cyclobutyl]benzene (3c)[1]



3c

Compound **3c** was synthesized following the *General procedure* 2.

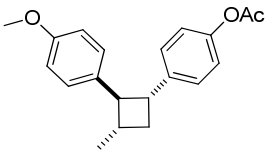
A colorless oil, 88% yield.

TLC: R_f = 0.51 (Hexane).

^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.18 (d, *J* = 8.5 Hz, 2H), 7.11 (s, 4H), 6.87 (d, *J* = 8.6 Hz, 2H), 3.81 (s, 3H), 3.36 (q, *J* = 9.9 Hz, 1H), 2.94 (t, *J* = 9.5 Hz, 1H), 2.51 (t, *J* = 7.7 Hz, 1H), 2.33 (s, 3H), 1.69 (q, *J* = 10.1 Hz, 2H), 1.20 (d, *J* = 6.5 Hz, 3H).

The spectra data are matched with those reported[1].

4-[(1*RS*,2*RS*,3*SR*)-2-(4-Methoxyphenyl)-3-methylcyclobutyl]phenyl acetate (3d)[1]



3d

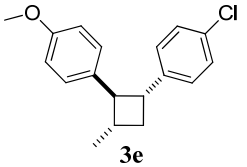
Compound **3d** was synthesized following the *General procedure* 2.

A colorless oil, 60% yield.

TLC: R_f = 0.39 (Hexane).

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.24 – 7.15 (m, 4H), 7.00 (d, *J* = 8.4 Hz, 2H), 6.88 (d, *J* = 8.6 Hz, 2H), 3.82 (s, 3H), 3.41 (q, *J* = 9.6 Hz, 1H), 2.94 (t, *J* = 9.6 Hz, 1H), 2.53 (q, *J* = 8.0 Hz, 1H), 2.36 (q, *J* = 8.2, 7.6 Hz, 1H), 2.31 (s, 3H), 1.70 (q, *J* = 10.1 Hz, 1H), 1.20 (d, *J* = 6.5 Hz, 3H). The spectra data are matched with those reported[1].

1-Chloro-4-[(1*RS*,2*RS*,3*SR*)-2-(4-methoxyphenyl)-3-methylcyclobutyl]benzene (3e)



3e

Compound **3e** was synthesized following the *General procedure* 2.

A colorless oil, 70% yield.

TLC: R_f = 0.46 (Hexane).

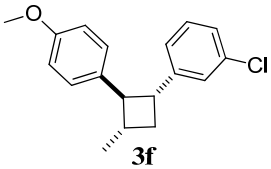
IR (KBr): 2954, 2927, 1512, 1249, 1091, 824 cm-1.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.25 (d, *J* = 8.2 Hz, 2H), 7.15 (dd, *J* = 16.2, 8.3 Hz, 4H), 6.88 (d, *J* = 8.4 Hz, 2H), 3.82 (s, 3H), 3.36 (q, *J* = 9.7 Hz, 1H), 2.90 (t, *J* = 9.5 Hz, 1H), 2.60 – 2.47 (m, 1H), 2.43 – 2.28 (m, 1H), 1.74 – 1.62 (m, 1H), 1.20 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 143.1, 135.4, 128.3, 127.9, 127.8, 113.8, 55.7, 55.2, 43.6, 35.4, 33.8, 20.4.

HRMS (APCI): $C_{18}H_{20}ClO$ $[M+H]^+$: calcd: 287.1197; found: 287.1190.

1-Chloro-3-[(1*RS*,2*RS*,3*SR*)-2-(4-methoxyphenyl)-3-methylcyclobutyl]benzene (3f)



3f

Compound **3f** was synthesized following the *General procedure* 2.

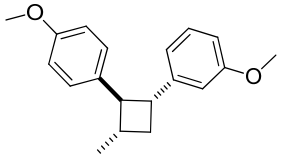
A colorless oil, 75% yield.

TLC: R_f = 0.43(Hexane).

IR (KBr): 2954, 2930, 1512, 1250, 1038, 782 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.24 – 7.13 (m, 5H), 7.07 (d, *J* = 7.3 Hz, 1H), 6.94 – 6.75 (m, 2H), 3.82 (s, 3H), 3.38 (q, *J* = 9.6 Hz, 1H), 2.94 (t, *J* = 9.6 Hz, 1H), 2.63 – 2.46 (m, 1H), 2.44 – 2.26 (m, 1H), 1.70 (q, *J* = 10.0 Hz, 1H), 1.21 (d, *J* = 6.5 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 158.2, 146.7, 135.3, 129.5, 127.7, 126.7, 126.1, 124.9, 113.8, 55.5, 55.2, 43.8, 35.5, 33.8, 20.4.
HRMS (APCI): $C_{18}H_{20}ClO$ $[M+H]^+$: calcd: 287.1197; found: 287.1190.

1-Methoxy-3-[(1*RS*,2*RS*,3*SR*)-2-(4-methoxyphenyl)-3-methylcyclobutyl]benzene (3g)[1]

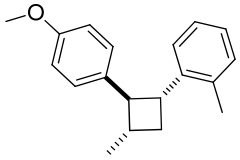


3g

Compound **3g** was synthesized following the *General procedure* 2.
A colorless oil, 68% yield.
TLC: R_f = 0.60(Hexane).
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.22 – 7.18 (m, 3H), 6.88 (d, *J* = 8.4 Hz, 2H), 6.81 (d, *J* = 7.8 Hz, 1H), 6.75 (d, *J* = 11.6 Hz, 2H), 3.81 (d, *J* = 7.7 Hz, 6H), 3.39 (q, *J* = 9.5 Hz, 1H), 2.96 (t, *J* = 9.5 Hz, 1H), 2.53 (d, *J* = 7.5 Hz, 1H), 2.46 – 2.27 (m, 1H), 1.71 (q, *J* = 10.1 Hz, 1H), 1.20 (d, *J* = 6.5 Hz, 3H).

The spectra data are matched with those reported[1].

1-[(1*RS*,2*RS*,3*SR*)-2-(4-Methoxyphenyl)-3-methylcyclobutyl]-2-methylbenzene (3h)[1]

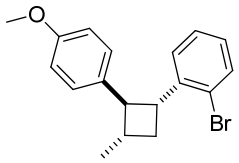


3h

Compound **3h** was synthesized following the *General procedure* 2.
A colorless oil, 85% yield.
TLC: R_f = 0.40 (Hexane).
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.33 (d, *J* = 7.6 Hz, 1H), 7.22 (d, *J* = 8.3 Hz, 3H), 7.12 (d, *J* = 4.4 Hz, 2H), 6.86 (d, *J* = 8.4 Hz, 2H), 3.81 (s, 3H), 3.65 – 3.46 (m, 1H), 3.16 (t, *J* = 9.5 Hz, 1H), 2.72 – 2.56 (m, 1H), 2.46 – 2.31 (m, 1H), 2.22 (s, 3H), 1.59 (q, J = 10.0 Hz, 1H), 1.22 (d, *J* = 6.5 Hz, 3H).

The spectra data are matched with those reported[1].

1-Bromo-2-[(1*RS*,2*RS*,3*SR*)-2-(4-methoxyphenyl)-3-methylcyclobutyl]benzene (3i)[1]



3i

Compound 3i was synthesized following the *General procedure* 2.

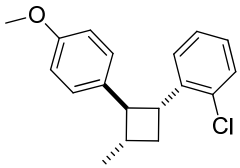
A colorless oil, 72% yield.

TLC: R_f = 0.45 (Hexane).

^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.53 (d, *J* = 9.5 Hz, 1H), 7.38 (d, *J* = 7.5 Hz, 1H), 7.29 (s, 1H), 7.22 (d, *J* = 8.5 Hz, 2H), 7.06 (t, *J* = 8.1 Hz, 1H), 6.87 (d, *J* = 8.6 Hz, 2H), 3.81 (s, 3H), 3.74 (q, *J* = 9.9 Hz, 1H), 3.16 (t, *J* = 9.6 Hz, 1H), 2.84 – 2.73 (m, 1H), 2.35 (t, *J* = 8.2 Hz, 1H), 1.52 – 1.45 (m, 1H), 1.24 (d, *J* = 6.5 Hz, 3H).

The spectra data are matched with those reported[1].

1-Chloro-2-[(1*RS*,2*RS*,3*SR*)-2-(4-methoxyphenyl)-3-methylcyclobutyl]benzene (3j)



3j

Compound 3j was synthesized following the *General procedure* 2.

A colorless oil, 77% yield.

TLC: R_f = 0.53 (Hexane).

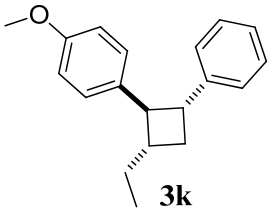
IR (KBr): 2950, 1512, 1249, 1177, 1036, 752 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.40 (d, *J* = 7.5 Hz, 1H), 7.37 – 7.32 (m, 1H), 7.24 (d, *J* = 8.6 Hz, 3H), 7.20 – 7.12 (m, 1H), 6.89 (d, *J* = 8.6 Hz, 2H), 3.82 (s, 3H), 3.77 (d, *J* = 10.2 Hz, 1H), 3.17 (t, *J* = 9.6 Hz, 1H), 2.81 – 2.71 (m, 1H), 2.37 (td, *J* = 7.2, 3.7 Hz, 1H), 1.61 – 1.49 (m, 1H), 1.25 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 158.1, 135.3, 129.3, 127.7, 127.6, 127.1, 126.7, 113.78, 55.2, 52.8, 41.1, 36.0, 35.1, 20.6.

HRMS (APCI): $C_{18}H_{20}ClO$ $[M+H]^+$: calcd: 287.1197; found: 287.1190.

1-[(1*RS*,2*SR*,4*RS*)-2-Ethyl-4-phenylcyclobutyl]-4-methoxybenzene (3k)



3k

Compound **3k** was synthesized following the *General procedure* 2.

A colorless oil, 62% yield.

TLC: Rf = 0.52(Hexane).

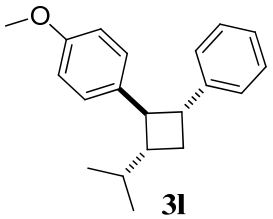
IR (KBr): 2957, 2927, 1512, 1248, 1038, 832, 698 cm^{-1}.

^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.32 – 7.27 (m, 3H), 7.20 (dd, *J* = 8.6, 6.6 Hz, 4H), 6.89 – 6.84 (m, 2H), 3.81 (s, 3H), 3.39 (q, *J* = 9.4 Hz, 1H), 3.02 (t, *J* = 9.5 Hz, 1H), 2.52 (dt, *J* = 10.2, 7.8 Hz, 1H), 2.29 – 2.20 (m, 1H), 1.75 – 1.64 (m, 2H), 1.49 (dt, *J* = 15.2, 7.6 Hz, 1H), 0.87 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 157.9, 144.7, 136.2, 128.2, 127.9, 126.6, 125.9, 113.7, 55.2, 54.0, 44.2, 41.9, 31.6, 28.8, 11.4.

HRMS (APCI): $C_{19}H_{23}O$ $[M+H]^+$: calcd: 267.1743; found: 267.1742.

1-[(1*RS*,2*RS*,4*RS*)-2-Isopropyl-4-phenylcyclobutyl]-4-methoxybenzene (3l)



3l

Compound **3l** was synthesized following the *General procedure* 2.

A colorless oil, 58% yield.

TLC: R_f = 0.54(Hexane).

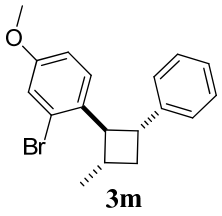
IR (KBr): 2955, 1512, 1249, 1177, 1038, 830, 698 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.38 – 7.15 (m, 7H), 6.88 (d, *J* = 8.7 Hz, 2H), 3.83 (s, 3H), 3.35 – 3.25 (m, 1H), 3.05 (t, *J* = 9.4 Hz, 1H), 2.54 – 2.43 (m, 1H), 2.22 – 2.07 (m, 1H), 1.82 – 1.66 (m, 2H), 0.93 (d, *J* = 6.7 Hz, 3H), 0.79 (d, *J* = 6.7 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 136.6, 128.2, 128.1, 126.5, 125.8, 113.6, 55.2, 53.6, 47.0, 44.5, 34.7, 29.8, 20.2, 19.8.

HRMS (APCI): $C_{20}H_{25}O$ $[M+H]^+$: calcd: 281.1900; found: 271.1892.

2-Bromo-4-methoxy-1-[(1*RS*,2*SR*,4*RS*)-2-methyl-4-phenylcyclobutyl]benzene (3m)



3m

Compound **3m** was synthesized following the *General procedure* 2.

A colorless oil, 50% yield.

TLC: R_f = 0.47 (Hexane).

IR (KBr): 2955.5, 1602.7, 1492.4, 1286.8, 1247.9, 1039.1, 846.6, 698.2 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.43 (d, *J* = 8.6 Hz, 1H), 7.32 – 7.25 (m, 2H), 7.19 (d, *J* = 7.4 Hz, 3H), 7.11 (d, *J* = 2.6 Hz, 1H), 6.93 (dd, *J* = 8.7, 2.7 Hz, 1H), 3.81 (s, 3H), 3.55 (t, *J* = 27.4,

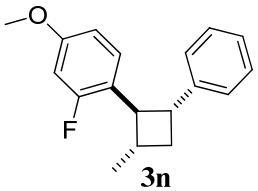
9.4 Hz, 2H), 2.54 (dd, *J* = 10.2, 7.5 Hz, 1H), 2.33 – 2.24 (m, 1H), 1.75 (q, *J* = 10.0 Hz, 1H), 1.22 (d, *J* = 6.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 158.2, 134.3, 128.5, 128.1, 126.5, 125.9, 117.5, 114.0, 55.5, 53.4, 43.7, 37.3, 33.6, 20.5.

HRMS (APCI): $C_{18}H_{20}{}^{79}BrO$ $[M+H]^+$: calcd: 331.0692; found: 331.0696.

$C_{18}H_{20}{}^{81}BrO$ $[M+H]^+$: calcd: 333.0677; found: 333.0676.

2-Fluoro-4-methoxy-1-[(1*RS*,2*SR*,4*RS*)-2-methyl-4-phenylcyclobutyl]benzene (3n)



3n

Compound **3n** was synthesized following the *General procedure* 2.

A colorless oil, 61% yield.

TLC: R_f = 0.65 (Hexane).

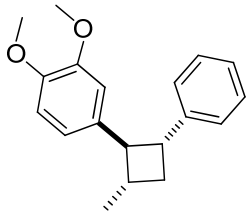
IR (KBr): 2955.6, 2926.1, 1624.5, 1508.1, 1154.4, 833.9, 744.9, 698.1 cm^{-1}.

^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.31 – 7.27 (m, 3H), 7.24 – 7.17 (m, 3H), 6.73 – 6.68 (m, 1H), 6.60 (d, *J* = 12.0 Hz, 1H), 3.80 (s, 3H), 3.57 – 3.49 (m, 1H), 3.26 (t, *J* = 9.7 Hz, 1H), 2.60 – 2.53 (m, 1H), 2.45 – 2.34 (m, 1H), 1.72 (q, *J* = 10.1 Hz, 1H), 1.20 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 161.4 (d, *J* = 224.5 Hz), 159.1 (d, *J* = 10.7 Hz), 144.4, 128.7 (d, *J* = 7.2 Hz), 122.0 (d, *J* = 16.2 Hz), 128.2, 126.4, 125.9, 109.7 (d, *J* = 3.7 Hz), 101.5 (d, *J* = 26.5 Hz), 55.5, 48.7, 42.6, 35.3, 34.2, 20.4.

HRMS (APCI): $C_{18}H_{20}FO$ $[M+H]^+$: calcd: 271.1493; found: 271.1492.

1,2-Dimethoxy-4-[(1*RS*,2*SR*,4*RS*)-2-methyl-4-phenylcyclobutyl]benzene (3o)



3o

Compound **3o** was synthesized following the *General procedure* 2.

A colorless oil, 65% yield.

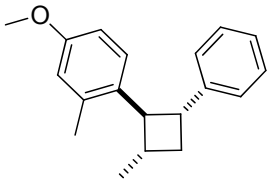
TLC: R_f = 0.60 (Hexane).

IR (KBr): 2952, 2931, 1515, 1453, 1263, 1030, 762, 699 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.33 – 7.29 (m, 2H), 7.26 – 7.18 (m, 3H), 6.84 (d, *J* = 2.2 Hz, 2H), 6.79 (s, 1H), 3.89 (d, *J* = 2.9 Hz, 6H), 3.50 – 3.36 (m, 1H), 2.97 (t, *J* = 9.5 Hz, 1H), 2.55 (dt, *J* = 10.3, 7.8 Hz, 1H), 2.41 – 2.34 (m, 1H), 1.75 (q, *J* = 10.1 Hz, 1H), 1.23 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 144.5, 136.4, 128.2, 126.6, 125.9, 118.6, 111.3, 110.29, 56.0, 55.9, 55.8, 44.1, 35.4, 33.7, 20.5.
HRMS (APCI): $C_{19}H_{23}O_2$ $[M+H]^+$: calcd: 283.1693; found: 283.1693.

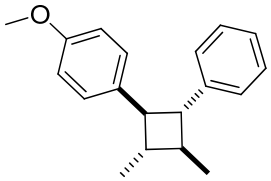
4-Methoxy-1-[(1*RS*,2*RS*,4*SR*)-2-(4-methoxyphenyl)-4-methylcyclobutyl]-2-methylbenzene (3p)



3p

Compound **3p** was synthesized following the *General procedure* 2.
A colorless oil, 52% yield.
TLC: R_f = 0.49(Hexane).
IR (KBr): 2924, 1609, 1502, 1251, 1052, 846 cm^{-1}.
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.41 (d, *J* = 8.5 Hz, 1H), 7.30 – 7.26 (m, 2H), 7.19 (d, *J* = 7.2 Hz, 3H), 6.85 – 6.78 (m, 1H), 6.70 (s, 1H), 3.82 (s, 3H), 3.46 – 3.37 (m, 1H), 3.20 (t, *J* = 9.5 Hz, 1H), 2.59 – 2.49 (m, 1H), 2.39 (t, *J* = 11.9, 5.4 Hz, 1H), 2.15 (s, 3H), 1.80 – 1.71 (m, 1H), 1.20 (d, *J* = 6.4 Hz, 3H).
^{13}C NMR (101 MHz, CDCl3) δ (ppm) 144.7, 137.4, 133.7, 128.2, 127.2, 126.5, 125.9, 115.6, 111.2, 55.1, 52.4, 44.6, 36.0, 33.3, 20.6, 20.3.
HRMS (APCI): $C_{19}H_{23}O$ $[M+H]^+$: calcd: 267.1743; found: 267.1745.

1-[(1*RS*,2*SR*,3*SR*,4*RS*)-2,3-Dimethyl-4-phenylcyclobutyl]-4-methoxybenzene (3q)[1]

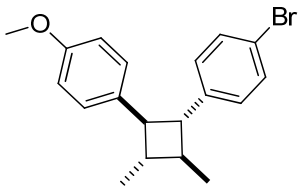


3q

Compound 3q was synthesized following the *General procedure* 2.
A colorless oil, 55% yield.
TLC: Rf = 0.49 (Hexane).
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.30 (d, *J* = 7.6 Hz, 3H), 7.23 (d, *J* = 7.3 Hz, 2H), 7.17 (d, *J* = 8.6 Hz, 2H), 6.86 (d, *J* = 8.5 Hz, 2H), 3.81 (s, 3H), 2.90 (dd, *J* = 8.6, 4.4 Hz, 2H), 2.00 – 1.79 (m, 2H), 1.22 (dd, *J* = 7.8, 6.1 Hz, 6H).

The spectra data are matched with those reported[1].

1-Bromo-4-[(1*RS*,2*RS*,3*SR*,4*SR*)-2-(4-methoxyphenyl)-3,4-dimethylcyclobutyl]benzene (3r)[1]



3r

Compound 3r was synthesized following the *General procedure* 2.

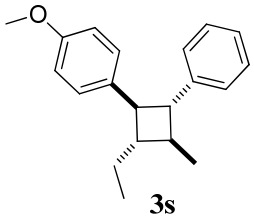
A colorless oil, 42% yield.

TLC: R_f = 0.46 (Hexane).

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.41 (d, *J* = 8.1 Hz, 2H), 7.12 (dd, *J* = 17.3, 8.2 Hz, 4H), 6.86 (d, *J* = 8.3 Hz, 2H), 3.81 (s, 3H), 2.88 – 2.77 (m, 2H), 1.87 (h, *J* = 6.9 Hz, 2H), 1.26 – 1.17 (m, 6H).

The spectra data are matched with those reported[1].

1-[(1*RS*,2*SR*,3*SR*,4*RS*)-2-Ethyl-3-methyl-4-phenylcyclobutyl]-4-methoxybenzene (3s)[1]



3s

Compound **3s** was synthesized following the *General procedure* 2.

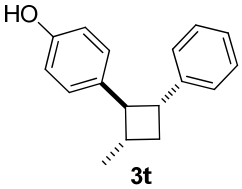
A colorless oil, 45% yield.

TLC: R_f = 0.62 (Hexane).

^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.32 – 7.27 (m, 2H), 7.22 (d, *J* = 7.3 Hz, 2H), 7.21 – 7.16 (m, 3H), 6.86 (d, *J* = 8.5 Hz, 2H), 3.81 (s, 3H), 2.93 (t, *J* = 9.4 Hz, 1H), 2.87 (t, *J* = 9.4 Hz, 1H), 1.99 (dd, *J* = 8.8, 6.4 Hz, 1H), 1.85 (dd, *J* = 15.7, 7.3 Hz, 1H), 1.67 (m, *J* = 13.8, 6.8 Hz, 1H), 1.63 – 1.56 (m, 1H), 1.28 (d, *J* = 6.5 Hz, 3H), 0.91 (t, *J* = 7.4 Hz, 3H).

The spectra data are matched with those reported[1].

4-[(1*RS*,2*SR*,4*RS*)-2-Methyl-4-phenylcyclobutyl]phenol (3t)



3t

Compound **3t** was synthesized following the *General procedure* 2.

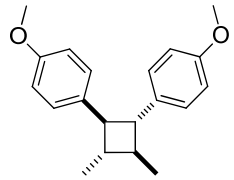
A colorless oil, 60% yield.

TLC: Rf = 0.35 (Hexane).

IR (KBr): 3300, 2957, 2752, 1257, 648, 876cm^{-1}.

^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.33 – 7.27 (m, 3H), 7.21 (d, *J* = 7.4 Hz, 2H), 7.15 (d, *J* = 6.3 Hz, 2H), 6.80 (d, *J* = 8.4 Hz, 2H), 4.67 (s, 1H), 3.41 (q, *J* = 9.5 Hz, 1H), 2.96 (t, *J* = 9.5 Hz, 1H), 2.57 – 2.50 (m, 1H), 2.40 – 2.27 (m, 1H), 1.73 (q, *J* = 10.1 Hz, 1H), 1.21 (d, *J* = 6.5 Hz, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 153.8, 128.2, 127.9, 126.6, 125.9, 115.1, 55.5, 44.14, 35.4, 33.9, 20.4.
HRMS (APCI): $C_{17}H_{19}O$ $[M+H]^+$: calcd: 239.1430; found: 239.1426.

4,4'-[(1*RS*,2*RS*,3*SR*,4*SR*)-3,4-Dimethylcyclobutane-1,2-diyl]bis(methoxybenzene) (3u)[1]

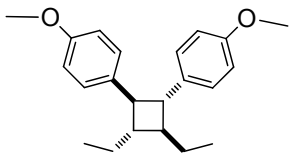


3u

Compound **3u** was synthesized following the *General procedure* 1.
A colorless oil, 68% yield.
TLC: R_f = 0.54 (Hexane).
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.20 (d, *J* = 8.6 Hz, 4H), 6.90 (d, *J* = 8.7 Hz, 4H), 3.84 (s, 6H), 2.95 – 2.75 (m, 2H), 1.90 (dp, *J* = 11.5, 3.7 Hz, 2H), 1.25 (d, *J* = 6.0 Hz, 6H).

The spectra data are matched with those reported[1].

4, 4'-[(1*RS*, 2*RS*, 3*SR*, 4*SR*)-3,4-Diethylcyclobutane-1,2-diyl]bis(methoxybenzene) (3v)[1]

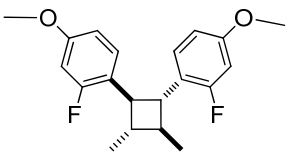


3v

Compound 3v was synthesized following the *General procedure* 1.
A colorless oil, 72% yield.
TLC: R_f = 0.50 (Hexane).
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.12 (d, *J* = 8.5 Hz, 4H), 6.81 (d, *J* = 8.5 Hz, 4H), 3.77 (s, 6H), 2.79 (d, *J* = 5.7 Hz, 2H), 1.90 (q, *J* = 4.3, 3.8 Hz, 2H), 1.63 (t, *J* = 7.1 Hz, 4H), 0.86 (t, *J* = 7.4 Hz, 6H).

The spectra data are matched with those reported[1].

4,4'-[(1*RS*,2*RS*,3*SR*,4*SR*)-3,4-dimethylcyclobutane-1,2-diyl]bis(3-fluoro-1-methoxybenzene) (3w)[1]



3w

Compound **3w** was synthesized following the *General procedure* 1.

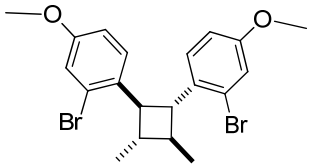
A colorless oil, 60% yield.

TLC: R_f = 0.58 (Hexane).

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.22 (dd, *J* = 17.9, 9.3 Hz, 2H), 6.68 – 6.59 (m, 2H), 6.55 (d, *J* = 11.9 Hz, 2H), 3.76 (s, 6H), 3.24 – 3.08 (m, 2H), 1.85 (h, *J* = 3.8 Hz, 2H), 1.18 (d, *J* = 5.0 Hz, 6H).

The spectra data are matched with those reported[1].

4,4'-[(1*RS*,2*RS*,3*SR*,4*SR*)-3,4-Dimethylcyclobutane-1,2-diyl]bis(3-bromo-1-methoxybenzene) (3x)[1]



3x

Compound **3x** was synthesized following the *procedure* 1.

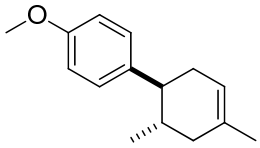
A colorless oil, 63% yield.

TLC: R_f = 0.50 (Hexane).

^{1}H NMR (600 MHz, $CDCl_3$) δ(ppm) 7.43 (d, *J* = 8.7 Hz, 2H), 7.05 (s, 2H), 6.97 – 6.84 (m, 2H), 3.78 (s, 6H), 3.50 – 3.32 (m, 2H), 1.84 (p, *J* = 5.6, 4.6 Hz, 2H), 1.22 (s, 6H).

The spectra data are matched with those reported[1].

(1*SR*,2*SR*)-4'-Methoxy-2,4-dimethyl-1,2,3,6-tetrahydro-1,1'-biphenyl (5a)[2]



5a

Compound **5a** was synthesized following the *General procedure* 3.

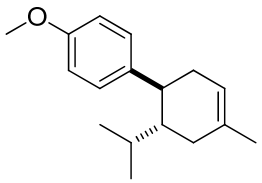
A yellow oil, 98% yield.

TLC: R_f = 0.60 (Hexane).

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.18 – 7.05 (m, 2H), 6.97 – 6.81 (m, 2H), 5.48 (s, 1H), 3.83 (s, 3H), 2.33 (dt, *J* = 10.4, 5.2 Hz, 1H), 2.26 – 2.08 (m, 3H), 1.93 (dt, *J* = 10.4, 5.1 Hz, 1H), 1.89 – 1.79 (m, 1H), 1.73 (s, 3H), 0.75 (d, *J* = 5.9 Hz, 3H).

The spectra data are matched with those reported[2].

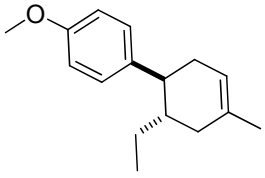
(1*SR*,2*RS*)-2-Isopropyl-4'-methoxy-4-methyl-1,2,3,6-tetrahydro-1,1'-biphenyl (5b)



5b

Compound **5b** was synthesized following the *General procedure* 3.
A yellow oil, 95% yield.
TLC: R_f = 0.62 (Hexane).
IR (KBr): 2956, 2899, 1512, 1245, 1177, 1040, 828 Cm^{-1}.
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.13 (d, *J* = 8.6 Hz, 2H), 6.87 (d, *J* = 8.6 Hz, 2H), 5.57 – 5.39 (m, 1H), 3.82 (s, 3H), 2.57 (td, *J* = 10.7, 5.4 Hz, 1H), 2.18 (d, *J* = 26.2 Hz, 2H), 1.94 (d, *J* = 10.1 Hz, 2H), 1.92 – 1.84 (m, 1H), 1.75 (s, 3H), 1.57 (q, *J* = 6.9, 3.5, 3.1 Hz, 1H), 0.87 (d, *J* = 7.0 Hz, 3H), 0.70 (d, *J* = 6.8 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 157.6, 137.9, 133.9, 128.6, 120.5, 113.6, 55.1, 43.5, 43.1, 36.2, 29.1, 27.1, 23.7, 21.2, 14.8.
HRMS (APCI): $C_{17}H_{25}O$ $[M+H]^+$: calcd: 245.1900; found: 245.1896.

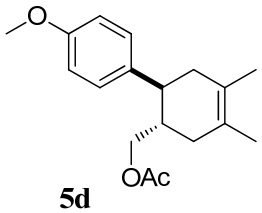
(1*SR*,2*SR*)-2-Ethyl-4'-methoxy-4-methyl-1,2,3,6-tetrahydro-1,1'-biphenyl (5c)



5c

Compound **5c** was synthesized following the *General procedure* 3.
A yellow oil, 96% yield.
TLC: R_f = 0.65 (Hexane).
IR (KBr): 2960, 2905, 1512, 1247, 1039, 825 cm^{-1}.
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.12 (d, *J* = 8.5 Hz, 2H), 6.88 (d, *J* = 8.4 Hz, 2H), 5.57 – 5.38 (m, 1H), 3.83 (s, 3H), 2.51 – 2.36 (m, 1H), 2.18 (dt, *J* = 21.0, 7.9 Hz, 3H), 1.79 (d, *J* = 9.0 Hz, 2H), 1.74 (s, 3H), 1.34 – 1.25 (m, 1H), 1.03 – 0.89 (m, 1H), 0.81 (t, *J* = 7.4 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 157.7, 138.2, 133.6, 128.5, 120.7, 113.6, 55.2, 45.2, 40.0, 35.9, 35.2, 26.6, 23.5, 10.8.
HRMS (APCI): $C_{16}H_{23}O$ $[M+H]^+$: calcd:231.1743; found: 231.1744.

{(1*SR*,2*SR*)-4'-Methoxy-4,5-dimethyl-1,2,3,6-tetrahydro-[1,1'-biphenyl]-2-yl}methylacetate (5d)[2]



5d

Compound **5d** was synthesized following the *General procedure* 3.

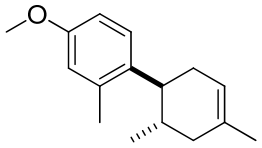
A yellow oil, 61% yield.

TLC: R_f = 0.56 (Hexane).

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.09 (d, *J* = 8.6 Hz, 2H), 6.83 (d, *J* = 8.6 Hz, 2H), 3.89 (dd, *J* = 10.9, 3.6 Hz, 1H), 3.78 (s, 3H), 3.65 (dd, *J* = 10.9, 7.0 Hz, 1H), 2.61 (d, *J* = 5.8 Hz, 1H), 2.16 (dd, *J* = 24.7, 12.1 Hz, 4H), 2.03 – 1.99 (m, 1H), 1.97 (s, 3H), 1.64 (d, *J* = 13.4 Hz, 6H).

The spectra data are matched with those reported[2].

(1*SR*,2*SR*)-4'-Methoxy-2,2',4-trimethyl-1,2,3,6-tetrahydro-1,1'-biphenyl (5e)



5e

Compound **5e** was synthesized following the *General procedure* 3.

A yellow oil, 95% yield.

TLC: R_f = 0.59 (Hexane).

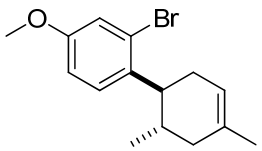
IR (KBr): 2592, 2924, 1609, 1502, 1251, 1052, 844 cm^{-1}

^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.08 (s, 1H), 6.78 – 6.74 (m, 1H), 6.72 (d, *J* = 2.8 Hz, 1H), 5.50 – 5.43 (m, 1H), 3.81 (s, 3H), 2.64 (td, *J* = 10.8, 5.2 Hz, 1H), 2.32 (s, 3H), 2.23 – 2.02 (m, 3H), 2.02 – 1.94 (m, 1H), 1.86 (dd, *J* = 17.0, 11.4 Hz, 1H), 1.73 (s, 3H), 0.75 (d, *J* = 6.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 157.0, 137.2, 136.3, 133.8, 127.0, 121.1, 115.3, 111.63, 55.1, 41.3, 39.9, 34.7, 33.6, 23.4, 20.1, 19.8.

HRMS (APCI): $C_{18}H_{20}ClO$ $[M+H]^+$: calcd: 231.1743; found: 231.1748.

(1*SR*,2*SR*)-2'-Bromo-4'-methoxy-2,4-dimethyl-1,2,3,6-tetrahydro-1,1'-biphenyl (5f)



5f

Compound **5f** was synthesized following the *General procedure* 3.

A yellow oil, 95% yield.

TLC: R_f = 0.63 (Hexane).

IR (KBr): 2957, 2923, 1603, 1492, 1439, 1279, 1279, 1239, 1041, 836 cm^{-1}.

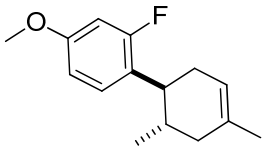
^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.14 –7.09 (m, 2H), 6.91 – 6.86 (m, 1H), 5.52 – 5.41 (m, 1H), 3.81 (s, 3H), 2.98 (td, *J* = 10.6, 5.3 Hz, 1H), 2.27 (dd, *J* = 17.4, 5.4 Hz, 1H), 2.15 – 2.08 (m, 1H), 2.05 – 1.96 (m, 2H), 1.94 – 1.84 (m, 1H), 1.73 (s, 3H), 0.79 (d, *J* = 6.4 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 157.8, 136.8, 133.8, 128.1, 120.6, 117.3, 114.2, 55.4, 44.5, 39.5, 33.9, 33.4, 23.4, 19.5.

HRMS (APCI): $C_{15}H_{20}{}^{79}BrO$ $[M+H]^+$: calcd: 295.0692; found: 295.0690.

$C_{15}H_{20}{}^{81}BrO$ $[M+H]^+$: calcd: 297.0672; found: 297.0670.

(1*SR*,2*SR*)-2'-Fluoro-4'-methoxy-2,4-dimethyl-1,2,3,6-tetrahydro-1,1'-biphenyl (5g)



5g

Compound **5g** *w*as synthesized following the *General procedure* 3.

A yellow oil, 90% yield.

TLC: R_f = 0.51 (Hexane).

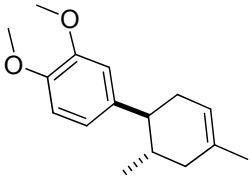
IR (KBr): 2957, 2924, 1625, 1507, 1293, 1152, 833 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.10 (t, *J* = 8.5 Hz, 1H), 6.77 – 6.64 (m, 1H), 6.61 (dd, *J* = 12.1, 2.6 Hz, 1H), 5.47 (s, 1H), 3.81 (s, 3H), 2.81 – 2.63 (m, 1H), 2.24 – 2.16 (m, 2H), 2.11 (dd, *J* = 17.0, 5.0 Hz, 1H), 2.03 – 1.93 (m, 1H), 1.91 – 1.77 (m, 1H), 1.72 (s, 3H), 0.78 (d, *J* = 6.4 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 161.5 (d, *J* = 245.0 Hz), 158.7 (d, *J* = 10.8 Hz), 133.8, 129.0 (d, *J* = 7.4 Hz), 124.1 (d, *J* = 15.3 Hz), 120.6, 109.9 (d, *J* = 3.2 Hz), 101.3 (d, *J* = 27.2 Hz), 58.5, 55.4, 39.7, 33.4, 33.0, 23.4, 19.8.

HRMS (APCI): $C_{15}H_{20}FO$ $[M+H]^+$: calcd: 235.1493; found: 235.1493.

(1*SR*,2*SR*)-3',4'-Dimethoxy-2,4-dimethyl-1,2,3,6-tetrahydro-1,1'-biphenyl (5h)



5h

Compound **5h** was synthesized following the *General procedure* 3.

A yellow oil, 60% yield.

TLC: R_f = 0.69 (Hexane).

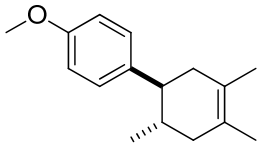
IR(KBr): 2952, 2925, 1516, 1258, 1238, 1140, 1031, 804 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 6.84 (dt, *J* = 7.9, 3.4 Hz, 1H), 6.79 – 6.69 (m, 2H), 5.48 (s, 1H), 3.90 (s, 6H), 2.31 (dd, *J* = 9.4, 4.6 Hz, 1H), 2.22 (d, *J* = 17.8 Hz, 2H), 2.12 (d, *J* = 17.6 Hz, 1H), 1.99 – 1.77 (m, 2H), 1.72 (s, 3H), 0.75 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 148.8, 147.1, 138.8, 133.83, 120.7, 119.6, 111.0, 110.7, 55.8, 55.8, 47.4, 39.8, 35.2, 34.0, 23.9, 20.2.

HRMS (APCI): $C_{16}H_{23}O_2$ $[M+H]^+$: calcd: 247.1693; found: 247.1692.

(1*SR*,2*SR*)-4'-Methoxy-2,4,5-trimethyl-1,2,3,6-tetrahydro-1,1'-biphenyl (5i)[2]



5i

Compound **5i** was synthesized following the *General procedure* 3.

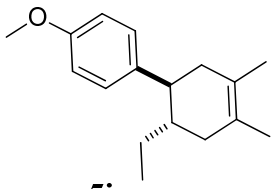
A yellow oil, 98% yield.

TLC: R_f = 0.55 (Hexane).

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.08 (d, *J* = 6.6 Hz, 2H), 6.85 (d, *J* = 2.2 Hz, 2H), 3.79 (s, 3H), 2.33 (td, *J* = 10.4, 5.8 Hz, 1H), 2.20 – 2.01 (m, 3H), 1.89 – 1.76 (m, 2H), 1.63 (d, *J* = 11.0 Hz, 6H), 0.69 (d, *J* = 5.7 Hz, 3H).

The spectra data are matched with those reported[2].

(1*SR*,2*SR*)-2-Ethyl-4'-methoxy-4,5-dimethyl-1,2,3,6-tetrahydro-1,1'-bipheny (5j)



5j

Compound **5j** was synthesized following the *General procedure* 3.

A yellow oil, 98% yield.

TLC: R_f = 0.62 (Hexane).

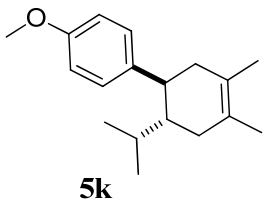
IR (KBr): 2959, 2909, 1512, 1246, 1177, 1039, 829 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.08 (d, *J* = 8.2 Hz, 2H), 6.84 (d, *J* = 8.4 Hz, 2H), 3.79 (s, 3H), 2.44 (td, *J* = 10.4, 5.9 Hz, 1H), 2.24 – 1.99 (m, 3H), 1.87 – 1.67 (m, 2H), 1.64 (d, *J* = 17.1 Hz, 6H), 1.25 (td, *J* = 7.5, 3.9 Hz, 1H), 0.96 – 0.84 (m, 1H), 0.76 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 157.7, 138.2, 128.5, 125.2, 125.1, 113.6, 55.2, 46.1, 41.8, 40.3, 37.6, 26.5, 18.9, 18.6, 10.9.

HRMS (APCI): $C_{17}H_{25}O$ $[M+H]^+$: calcd: 245.1900; found: 245.1901.

(1*SR*, 2*RS*)-2-Isopropyl-4'-methoxy-4, 5-dimethyl-1, 2, 3, 6-tetrahydro-1, 1'-biphenyl (5k)



5k

Compound **5k** was synthesized following the *General procedure* 3.

A yellow oil, 90% yield.

TLC: R_f = 0.60 (Hexane).

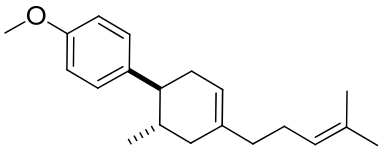
IR (KBr): 2956, 2899, 1512, 1245, 1177, 1040, 828 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.09 (d, *J* = 8.5 Hz, 2H), 6.84 (d, *J* = 8.3 Hz, 2H), 3.79 (s, 3H), 2.58 (td, *J* = 10.5, 6.2 Hz, 1H), 2.11 (t, *J* = 7.2 Hz, 2H), 1.92 (d, *J* = 9.9 Hz, 2H), 1.86 – 1.75 (m, 1H), 1.67 (s, 3H), 1.61 (s, 3H), 1.57 – 1.49 (m, 1H), 0.83 (d, *J* = 6.9 Hz, 3H), 0.66 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 157.6, 137.8, 128.6, 125.3, 125.1, 113.7, 55.1, 43.9, 43.8, 42.7, 30.7, 27.0, 21.3, 19.0, 18.6, 14.8.

HRMS (APCI): $C_{18}H_{27}O$ $[M+H]^+$: calcd: 259.2056; found: 259.2053.

(1*SR*,2*SR*)-4'-Methoxy-2-methyl-4-(4-methylpent-3-en-1-yl)-1,2,3,6-tetrahydro-1,1'-biphenyl (5l)[2]



5l

Compound **5l** was synthesized following the *General procedure* 3.

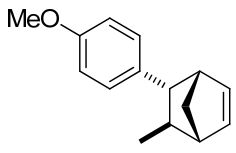
A yellow oil, 97% yield.

TLC: R_f = 0.65 (Hexane).

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.12 (d, *J* = 8.5 Hz, 2H), 6.88 (d, *J* = 8.4 Hz, 2H), 5.49 (d, *J* = 5.8 Hz, 1H), 5.25 – 5.09 (m, 1H), 3.83 (s, 3H), 2.33 (d, *J* = 10.4 Hz, 1H), 2.28 – 2.21 (m, 1H), 2.14 (q, *J* = 11.4, 6.5, 4.7 Hz, 4H), 2.03 (t, *J* = 8.1 Hz, 2H), 1.94 – 1.81 (m, 2H), 1.74 (s, 3H), 1.66 (s, 3H), 0.75 (d, *J* = 6.1 Hz, 3H).

The spectra data are matched with those reported[2].

(1*RS*,4*SR*,5*SR*,6*RS*)-5-(4-methoxyphenyl)-6-methylbicyclo[2.2.1]hept-2-ene(5m)[2]

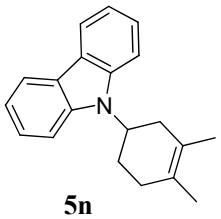


5m

Compound **5m** was synthesized following the *General procedure* 3.
A yellow oil, 72% yield.
TLC: R_f = 0.64 (Hexane).
^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.19 – 7.11 (m, 2H), 6.84 – 6.78 (m, 2H), 6.53 (t, *J* = 7.4 Hz, 1H), 6.25 (t, *J* = 7.1 Hz, 1H), 3.81 (s, *J* = 4.2 Hz, 3H), 2.54 – 2.49 (m, 1H), 2.39 – 2.31 (m, 1H), 2.19 (dd, *J* = 11.4, 6.7 Hz, 1H), 1.84 (m, *J* = 10.5, 5.0, 2.7 Hz, 1H), 1.70 – 1.62 (m, 2H), 1.08 (d, 3H).

The spectra data are matched with those reported[2].

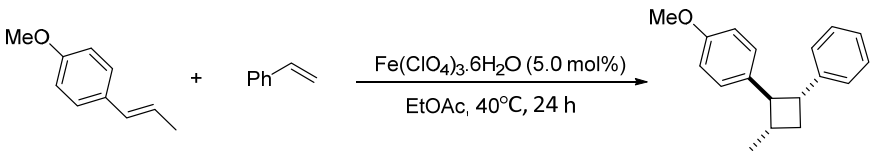
9-(3,4-Dimethylcyclohex-3-en-1-yl)-9H-carbazole (5n)



5n

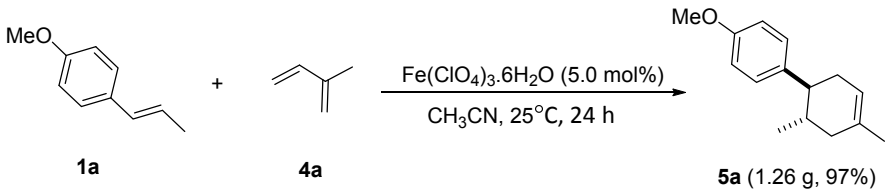
Compound **5n** was synthesized following the *General procedure* 3.
A yellow oil, 62% yield.
TLC: R_f = 0.70 (Hexane).
IR: 2956, 2925, 1452, 748, 722 cm^{-1}.
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.14 (d, *J* = 7.8 Hz, 2H), 7.56 (d, *J* = 8.3 Hz, 2H), 7.45 (t, *J* = 7.7 Hz, 2H), 7.24 (t, *J* = 7.4 Hz, 2H), 4.90 – 4.77 (m, 1H), 3.06 (t, *J* = 14.5 Hz, 1H), 2.71 (tt, *J* = 12.4, 6.1 Hz, 1H), 2.41 (s, 1H), 2.26 (dd, *J* = 15.1, 8.8 Hz, 2H), 2.13 – 2.00 (m, 1H), 1.77 (s, 3H), 1.71 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) 125.9, 125.3, 124.4, 123.3, 120.3, 118.5, 110.1, 52.4, 34.9, 32.5, 27.8, 19.1, 18.8.
HRMS (APCI): $C_{20}H_{22}N$ $[M+H]^+$: calcd: 276.1747; found: 276.1744.

6. Scale-up Experiments.



1a **2a** **3a** (1.28 g, 85%)

To a solution of the styrene **2a** (3.4 mL, 30 mmol, 5.0 eq) in 14 mL EtOAc, was added $Fe(ClO_4)_3{\cdot}6H_2O$ (106 mg, 5.0 mol%), then, a solution of anethole **1a** (890 mg, 6.0 mmol) in 10 ml EtOAc was added using a syringe pump (at a rate of 1.0 mL/h), The reaction was stirred under air. Then, the solvent was evaporated under reduced pressure to give the crude product, which was purified by chromatography on silica gel using the petroleum ether as the eluent.

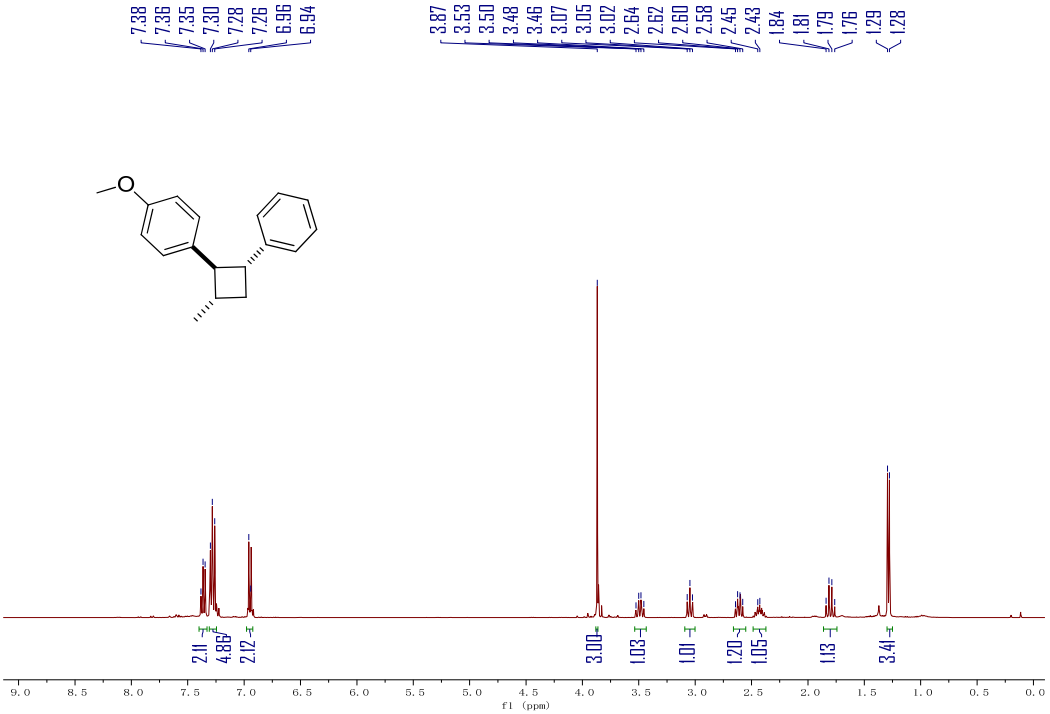


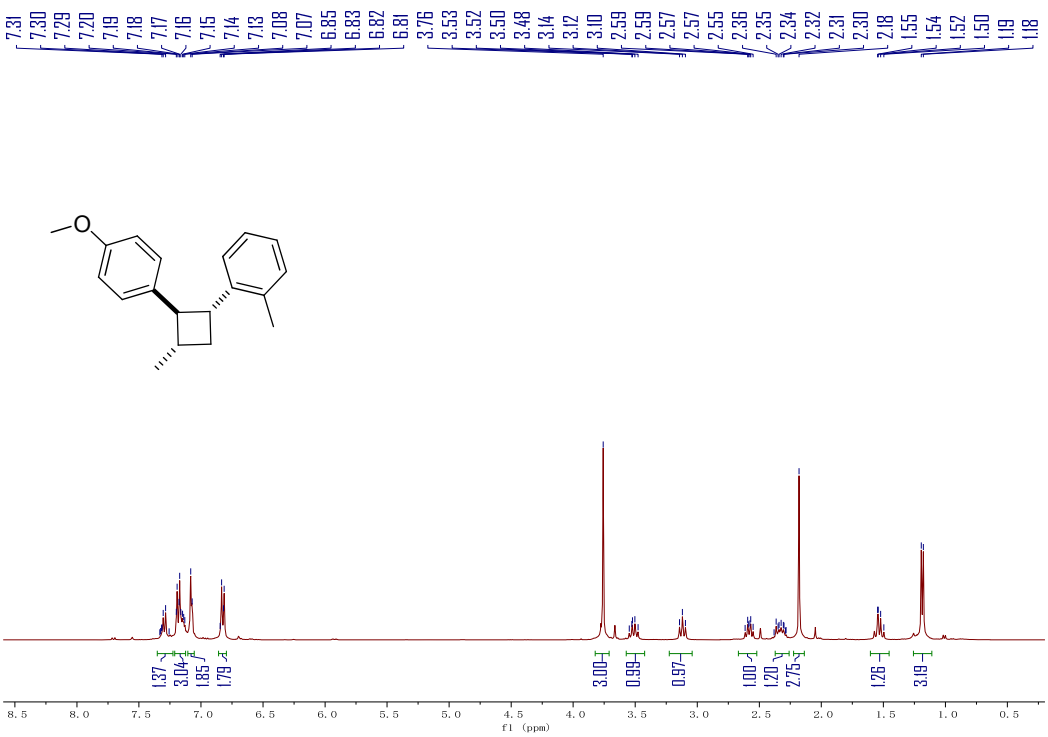
To a solution of the diene **4a** (1.2 mL, 12.0 mmol, 2.0 eq) in 14 mL CH_3CN, was added $Fe(ClO_4)_3{\cdot}6H_2O$ (5.0 mol%, 106 mg), then, a solution of anethole **1a** (890 mg, 6.0 mmol) in 10 mL CH_3CN was added using a syringe pump (at a rate of 1.0 mL/h), The reaction was stirred under air. Then, the solvent was evaporated under reduced pressure to give the crude product, which was purified by chromatography on silica gel using the petroleum ether.

Reference

1. I. Colomer, R. C. Barcelos and T. J. Donoho, *Angew. Chem.*, *Int. Ed*., 2016, **55**, 4748.
2. S.-S. Lin, M. A. Ischay, C. G. Fry and T. P. Yoon, *J. Am. Chem. Soc*., 2011, **133,** 19350.

7. NMR spectra of all products.

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7.08
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6.83
6.82
6.81
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1.55
1.54
1.52
1.50
1.19
1.18
O
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1.79
3.00
0.99
0.97
1.00
1.20
2.75
1.26
3.19
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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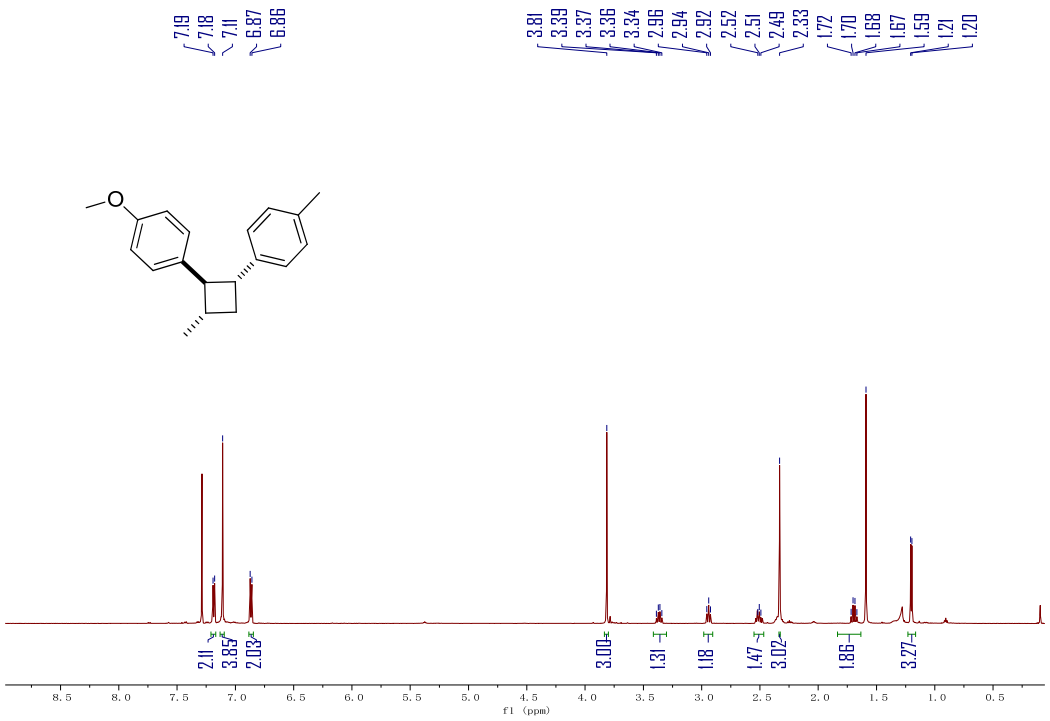
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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3.36
3.34
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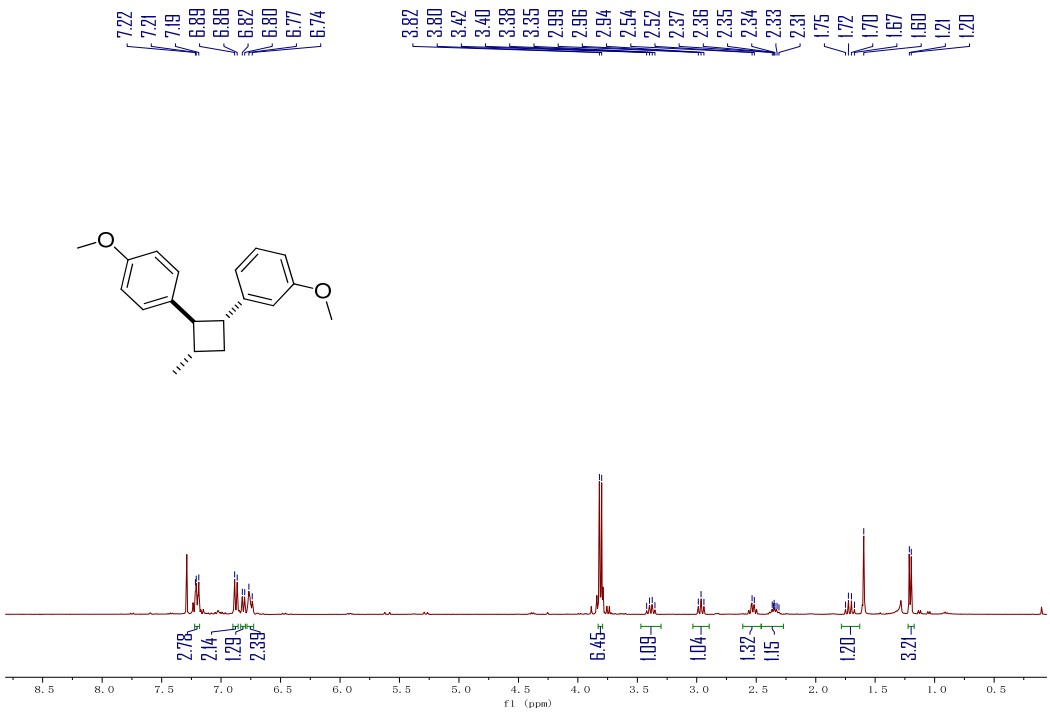
2.94
2.92
2.52
2.51
2.49
2.33
1.72
1.70
1.68
1.67
1.59
1.21
1.20
O
2.11
3.89
2.03
3.00
1.31
1.18
1.47
3.02
1.86
3.27
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


7.22
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6.74
3.82
3.80
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2.36
2.35
2.34
2.33
2.31
1.75
1.72
1.70
1.67
1.60
1.21
1.20
O
O
2.78
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2.39
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1.09
1.04
1.32
1.15
1.20
3.21
8.5
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7.5
7.0
6.5
6.0
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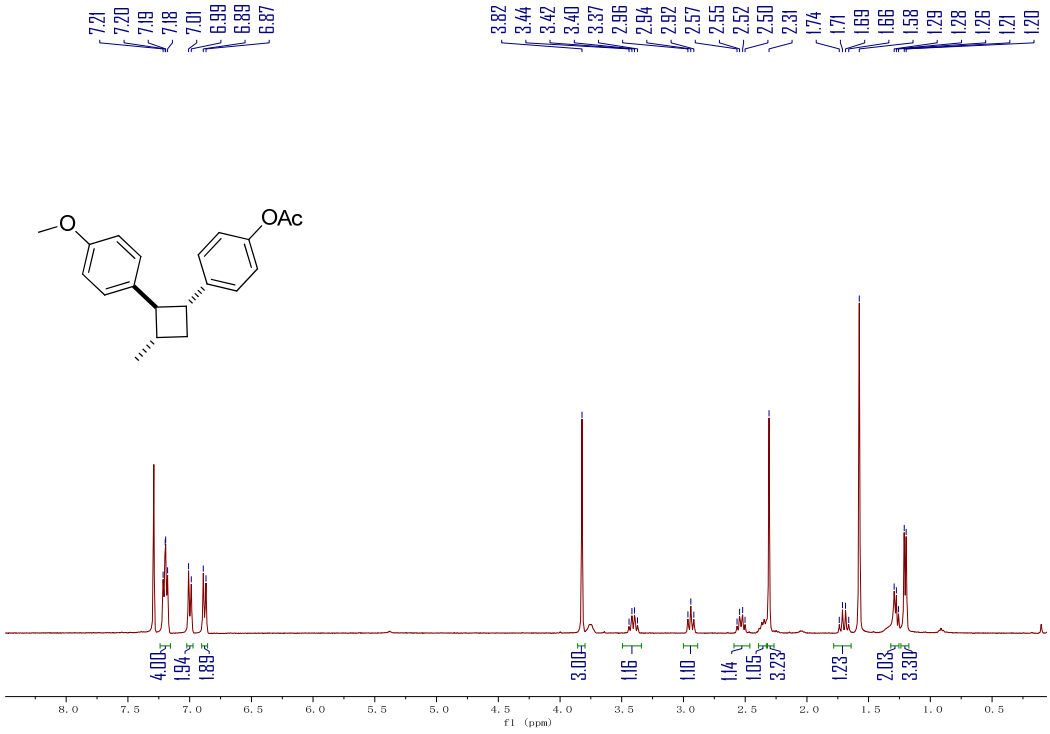
f1 (ppm)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2.94
2.92
2.57
2.55
2.52
2.50
2.31
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1.71
1.69
1.66
1.58
1.29
1.28
1.26
1.21
1.20
O
OAc
4.00
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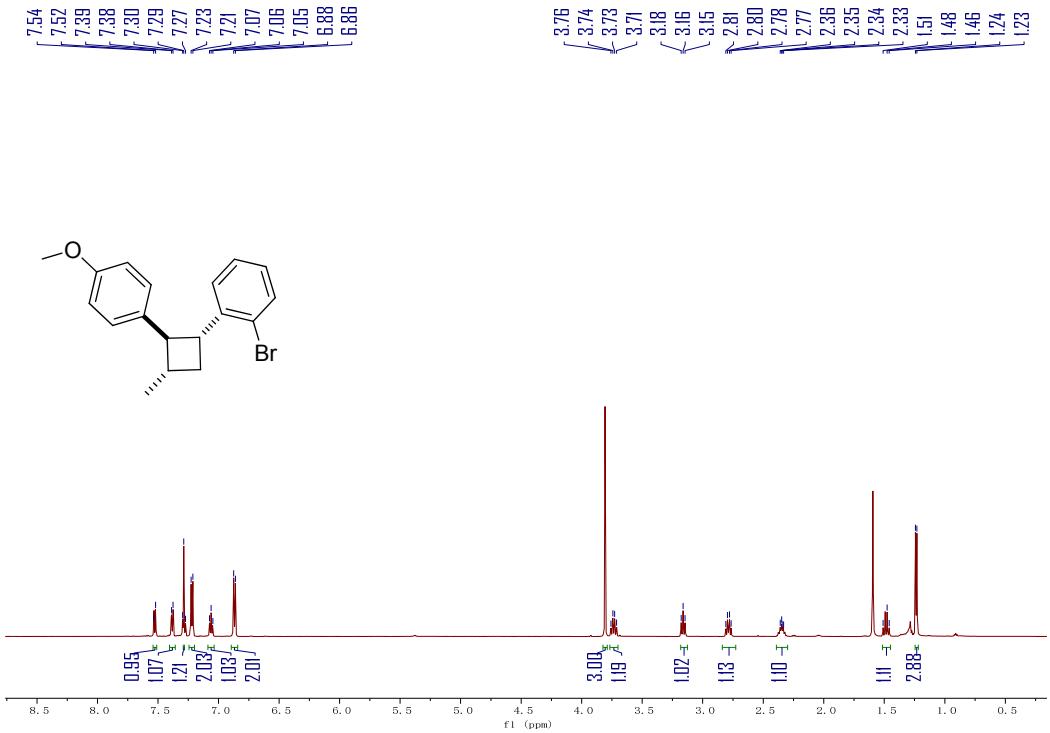
1.14
1.05
3.23
1.23
2.03
3.30
f1 (ppm)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3.74
3.73
3.71
3.18
3.16
3.15
2.81
2.80
2.78
2.77
2.36
2.35
2.34
2.33
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1.48
1.46
1.24
1.23
O
Br
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1.07
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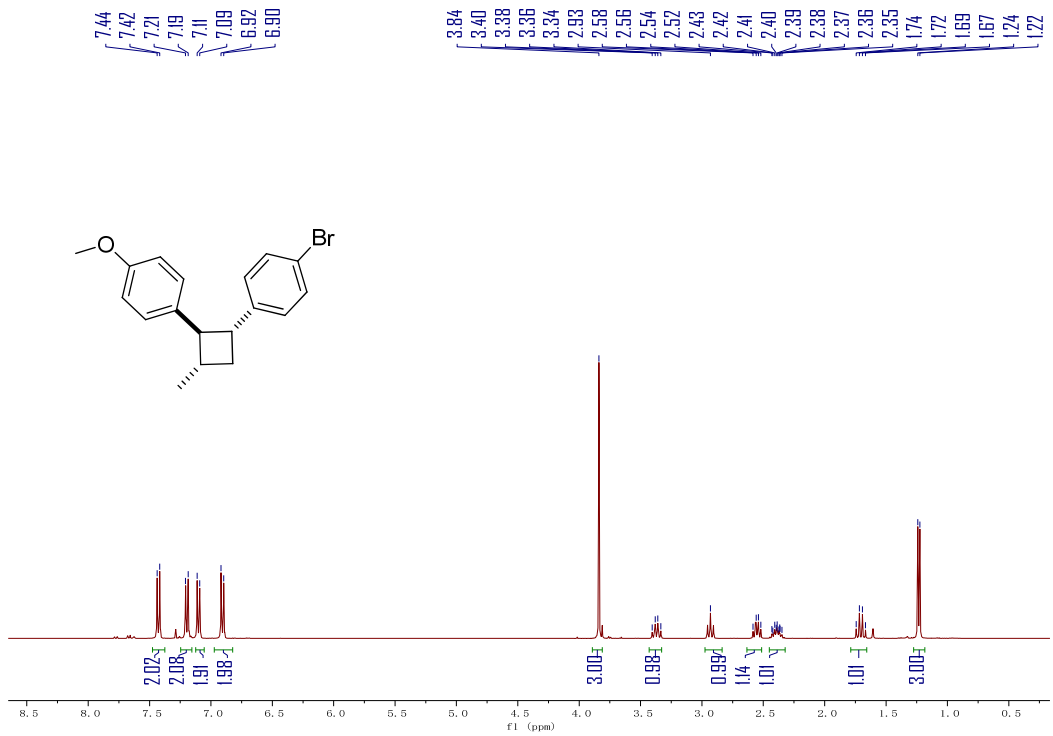
f1 (ppm)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1.69
1.67
1.24
1.22
O
Br
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0.98
0.99
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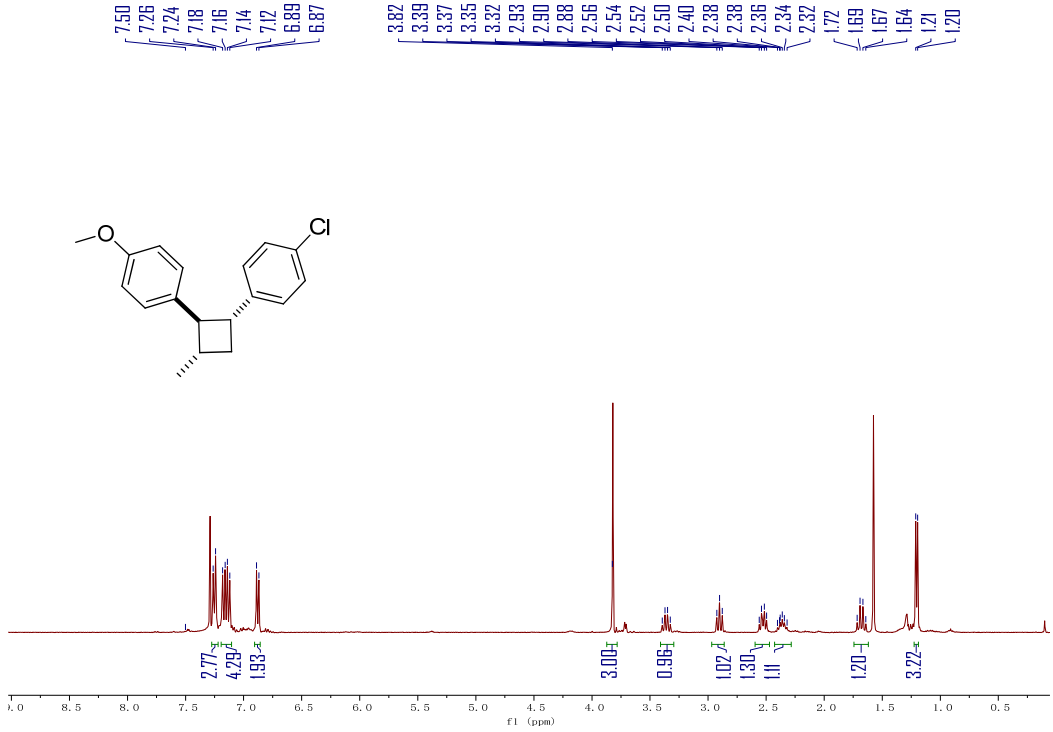
1.01
1.01
3.00
f1 (ppm)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3.32
2.93
2.90
2.88
2.56
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2.52
2.50
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2.38
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2.36
2.34
2.32
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1.69
1.67
1.64
1.21
1.20
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3.00
0.96
1.02
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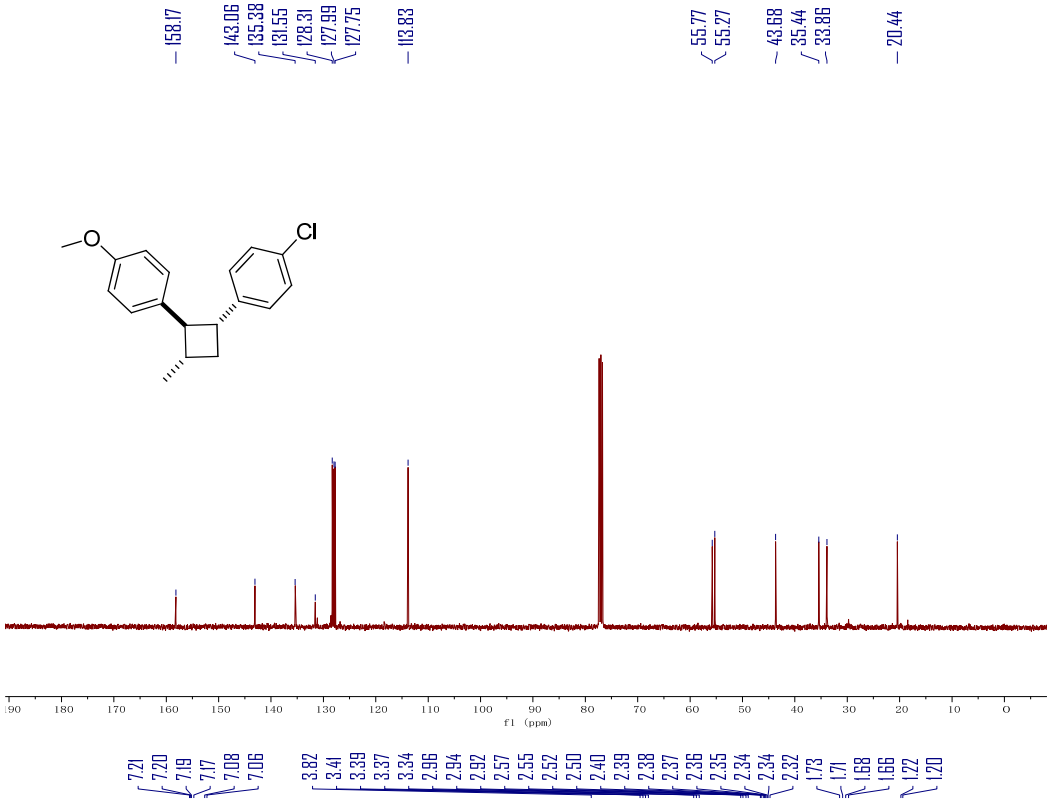
1.11
1.20
3.22
f1 (ppm)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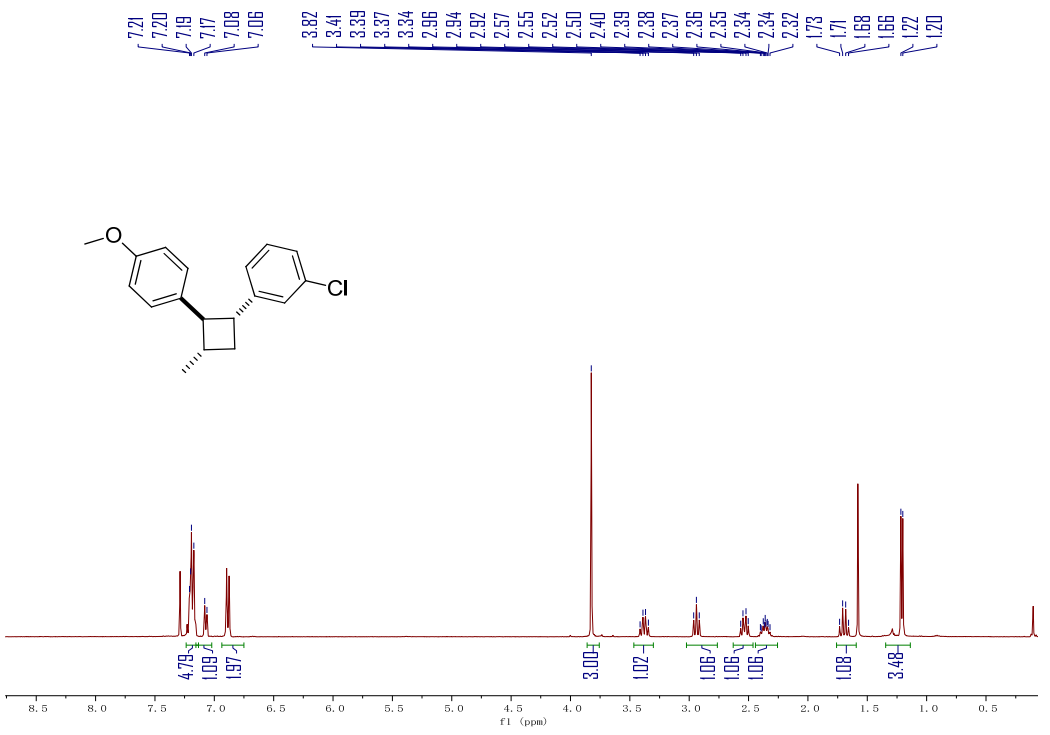
33.86
20.44
O
Cl
f1 (ppm)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3.41
3.39
3.37
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2.94
2.92
2.57
2.55
2.52
2.50
2.40
2.39
2.38
2.37
2.36
2.35
2.34
2.34
2.32
1.73
1.71
1.68
1.66
1.22
1.20
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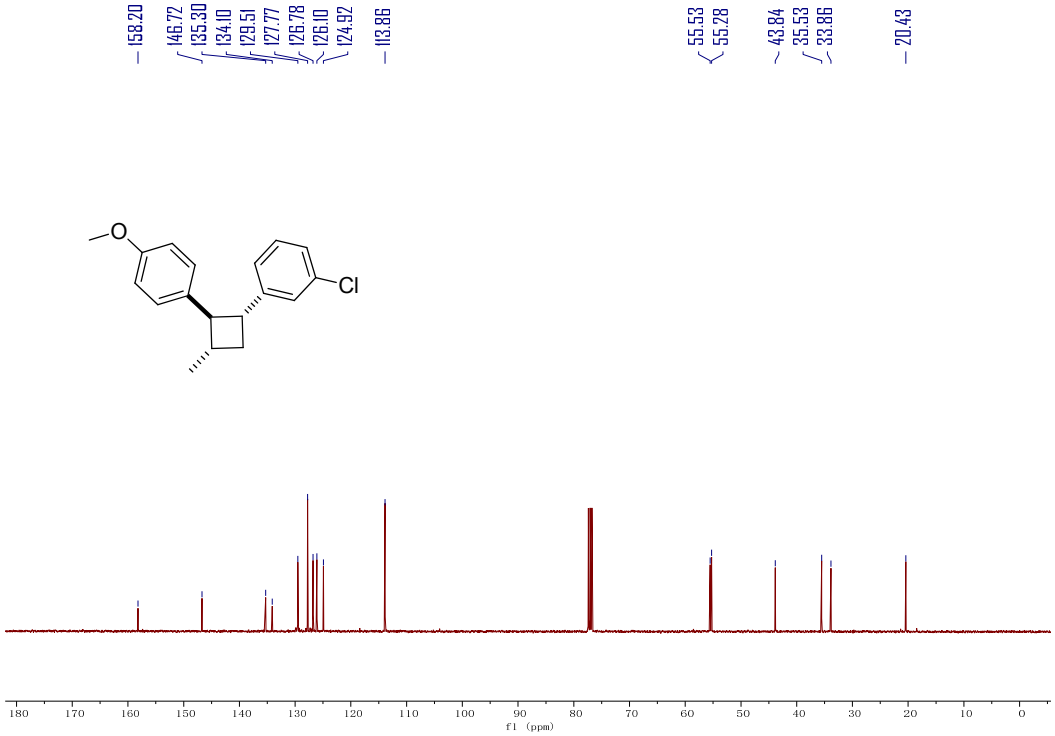
1.02
1.06
1.06
1.06
1.08
3.48
f1 (ppm)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126.10
124.92
113.86
55.53
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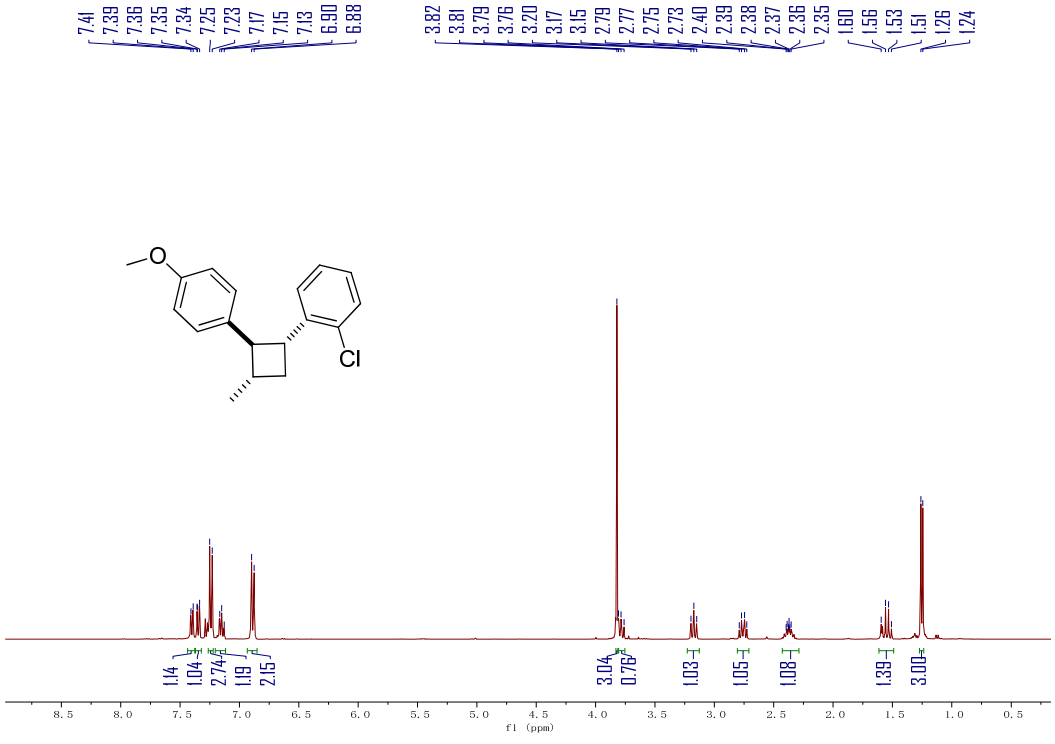
43.84
35.53
33.86
20.43
O
Cl
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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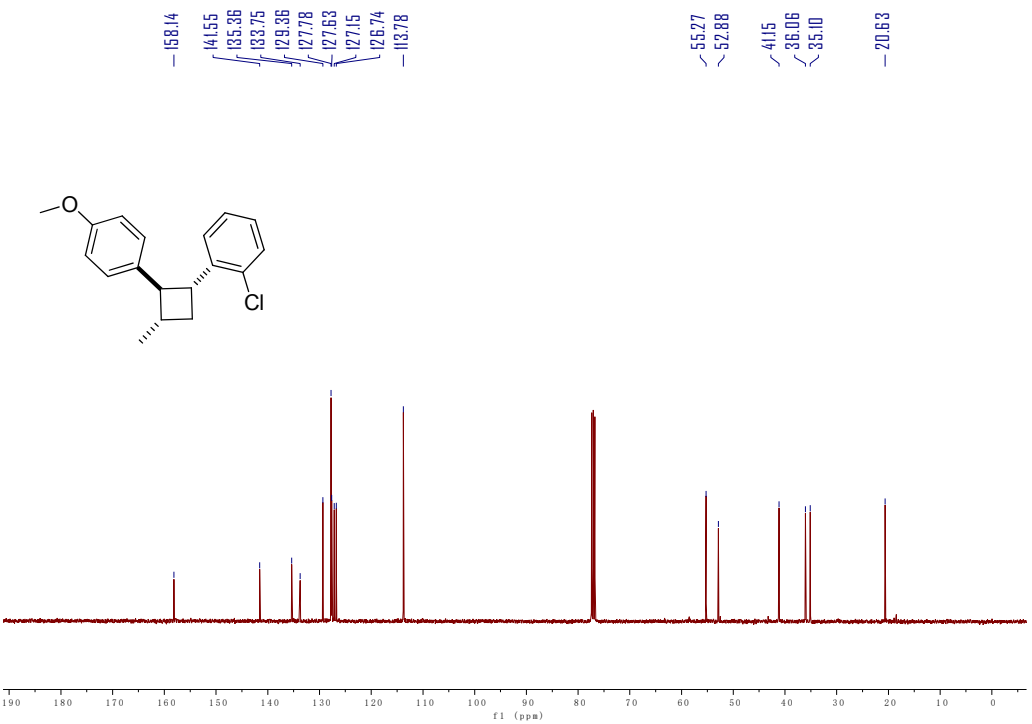
7.15
7.13
6.90
6.88
3.82
3.81
3.79
3.76
3.20
3.17
3.15
2.79
2.77
2.75
2.73
2.40
2.39
2.38
2.37
2.36
2.35
1.60
1.56
1.53
1.51
1.26
1.24
O
Cl
1.14
1.04
2.74
1.19
2.15
3.04
0.78
1.03
1.05
1.08
1.39
3.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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127.63
127.15
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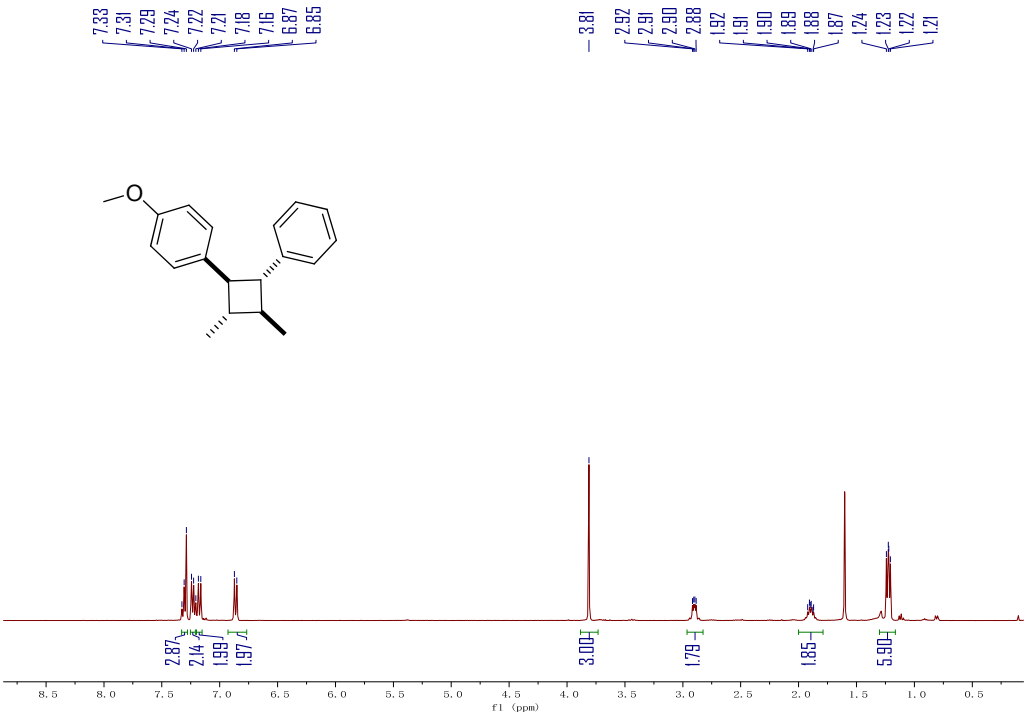
55.27
52.88
41.15
38.06
35.10
20.63
O
Cl
f1 (ppm)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7.31
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7.16
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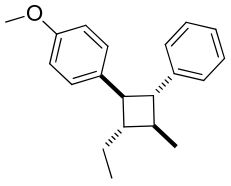
6.85
3.81
2.92
2.91
2.90
2.88
1.92
1.91
1.90
1.89
1.88
1.87
1.24
1.23
1.22
1.21
O
2.87
2.14
1.99
1.97
3.00
1.79
1.85
5.90
f1 (ppm)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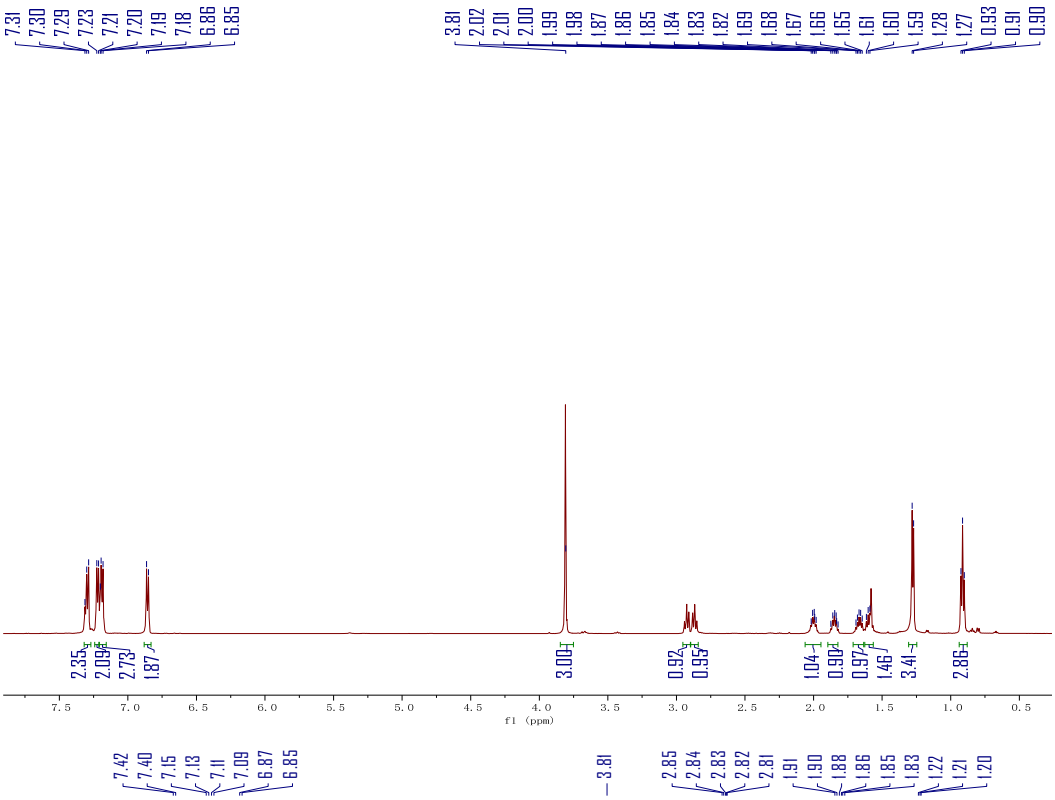
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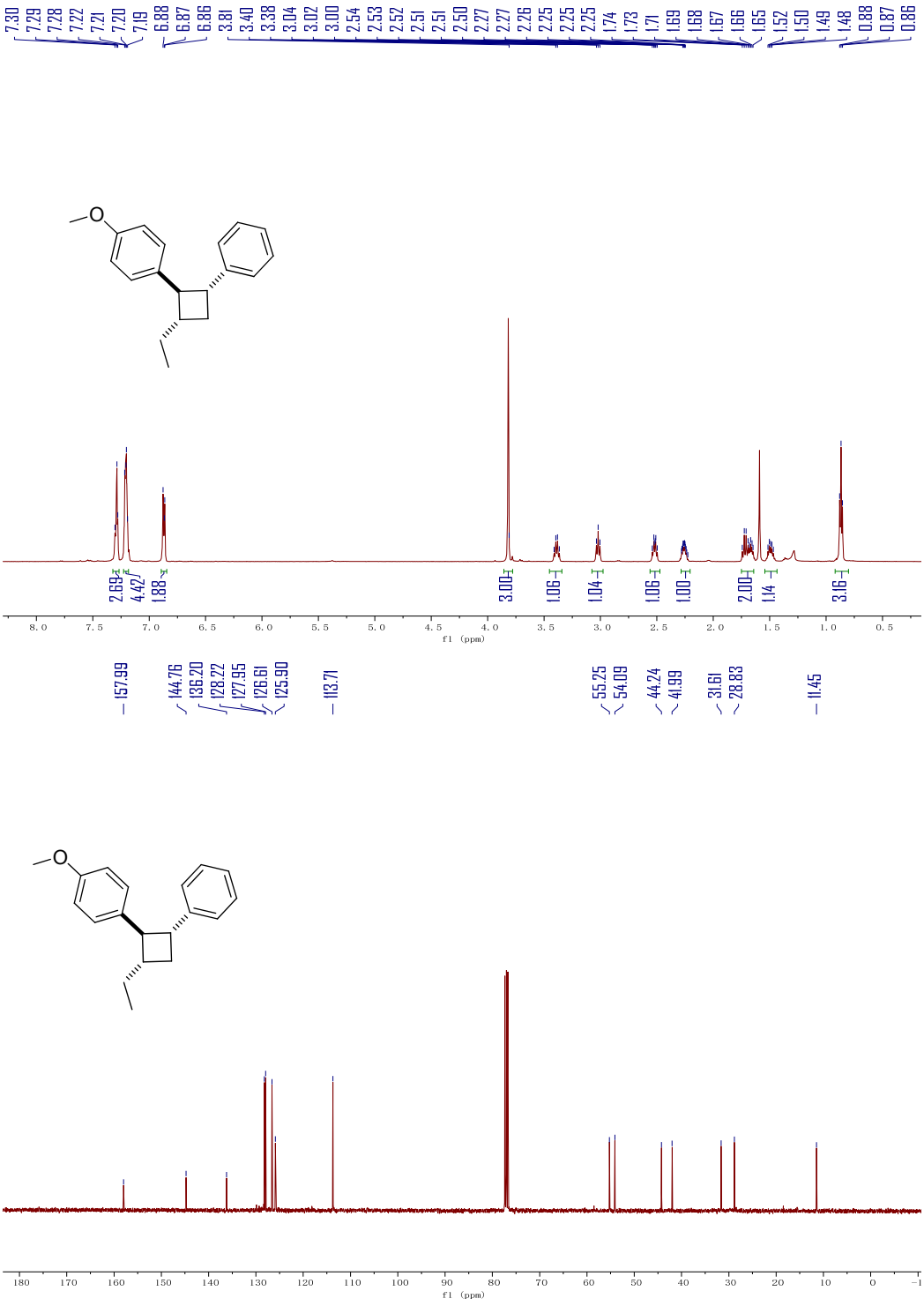
2.02
2.01
2.00
1.99
1.98
1.87
1.86
1.85
1.84
1.83
1.82
1.69
1.68
1.67
1.66
1.65
1.61
1.60
1.59
1.28
1.27
0.93
0.91
0.90
2.35
2.09
2.73
1.87
3.00
0.92
0.93
1.04
0.90
0.97
1.46
3.41
2.86
f1 (ppm)
7.42
7.40
7.15
7.13
7.11
7.09
6.87
6.85
3.81
2.85
2.84
2.83
2.82
2.81
1.91
1.90
1.88
1.86
1.85
1.83
1.22
1.21
1.20
O
Br
2.29
4.15
2.39
3.45
2.05
1.97
6.00
f1 (ppm)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6.87
6.86
3.81
3.40
3.38
3.04
3.02
3.00
2.54
2.53
2.52
2.51
2.51
2.50
2.27
2.27
2.26
2.25
2.25
2.25
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1.73
1.71
1.69
1.68
1.67
1.66
1.65
1.52
1.50
1.49
1.48
0.88
0.87
0.86
O
2.69
4.42
1.88
3.00
1.06
1.04
1.06
1.00
2.00
1.14
3.16
f1 (ppm)
157.99
144.76
136.20
128.22
127.95
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55.25
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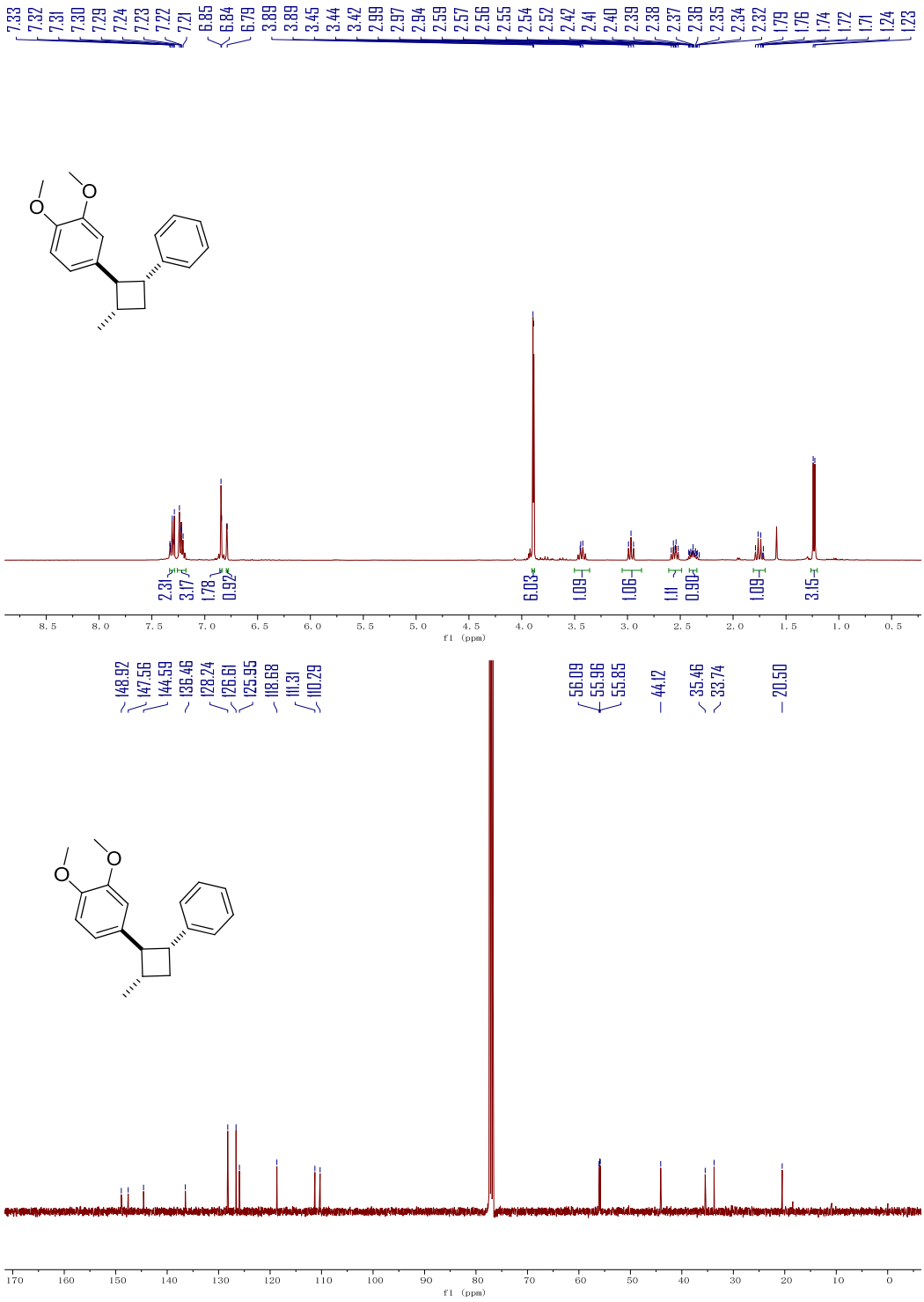
44.24
41.99
31.61
28.83
11.45
O
f1 (ppm)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7.22
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6.84
6.79
3.89
3.89
3.45
3.44
3.42
2.99
2.97
2.94
2.59
2.57
2.56
2.55
2.54
2.52
2.42
2.41
2.40
2.39
2.38
2.37
2.36
2.35
2.34
2.32
1.79
1.76
1.74
1.72
1.71
1.24
1.23
O
O
2.31
3.17
1.78
0.92
6.03
1.09
1.06
1.11
0.90
1.09
3.15
f1 (ppm)
148.92
147.56
144.59
136.46
128.24
126.61
125.95
118.68
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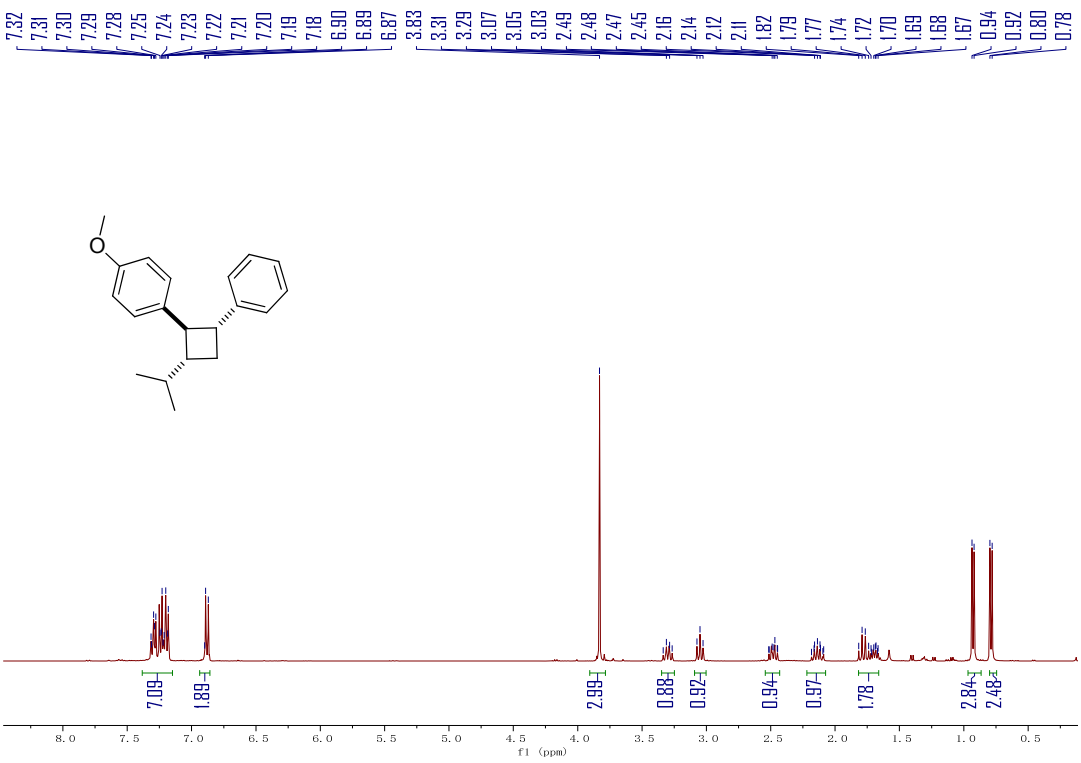
110.29
56.09
55.96
55.85
44.12
35.46
33.74
20.50
O
O
f1 (ppm)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7.22
7.21
7.20
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7.18
6.90
6.89
6.87
3.83
3.31
3.29
3.07
3.05
3.03
2.49
2.48
2.47
2.45
2.16
2.14
2.12
2.11
1.82
1.79
1.77
1.74
1.72
1.70
1.69
1.68
1.67
0.94
0.92
0.80
0.78
O
7.09
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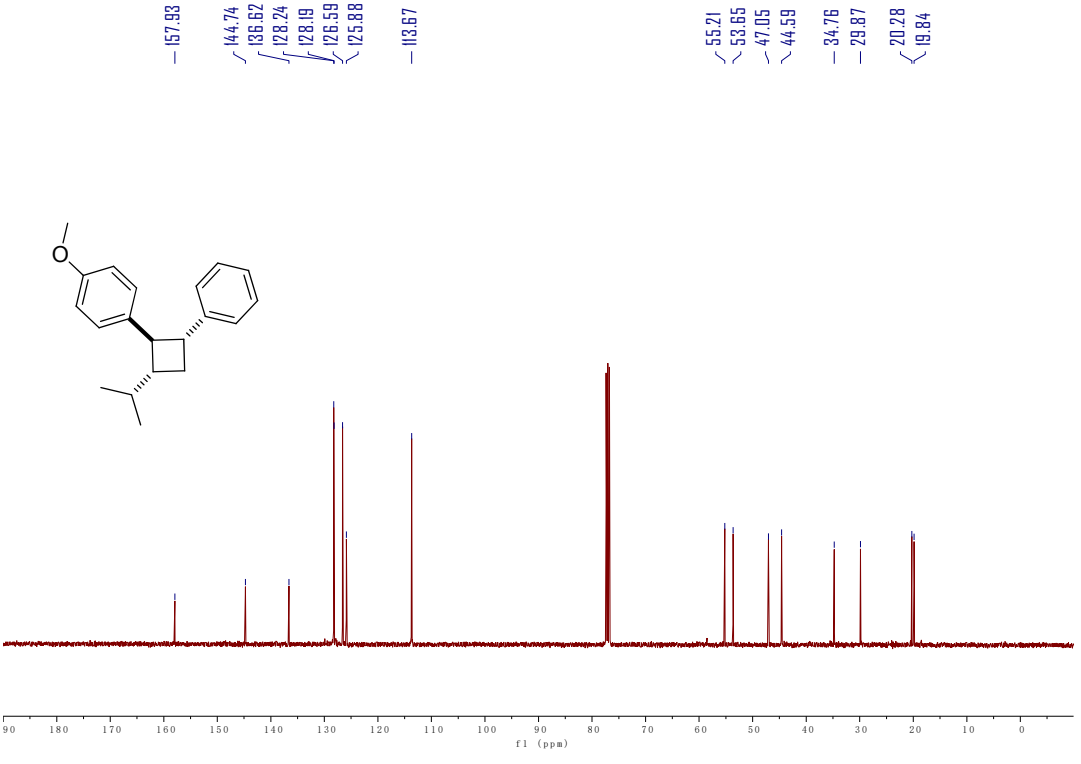
2.99
0.88
0.92
0.94
0.97
1.78
2.84
2.48
f1 (ppm)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126.59
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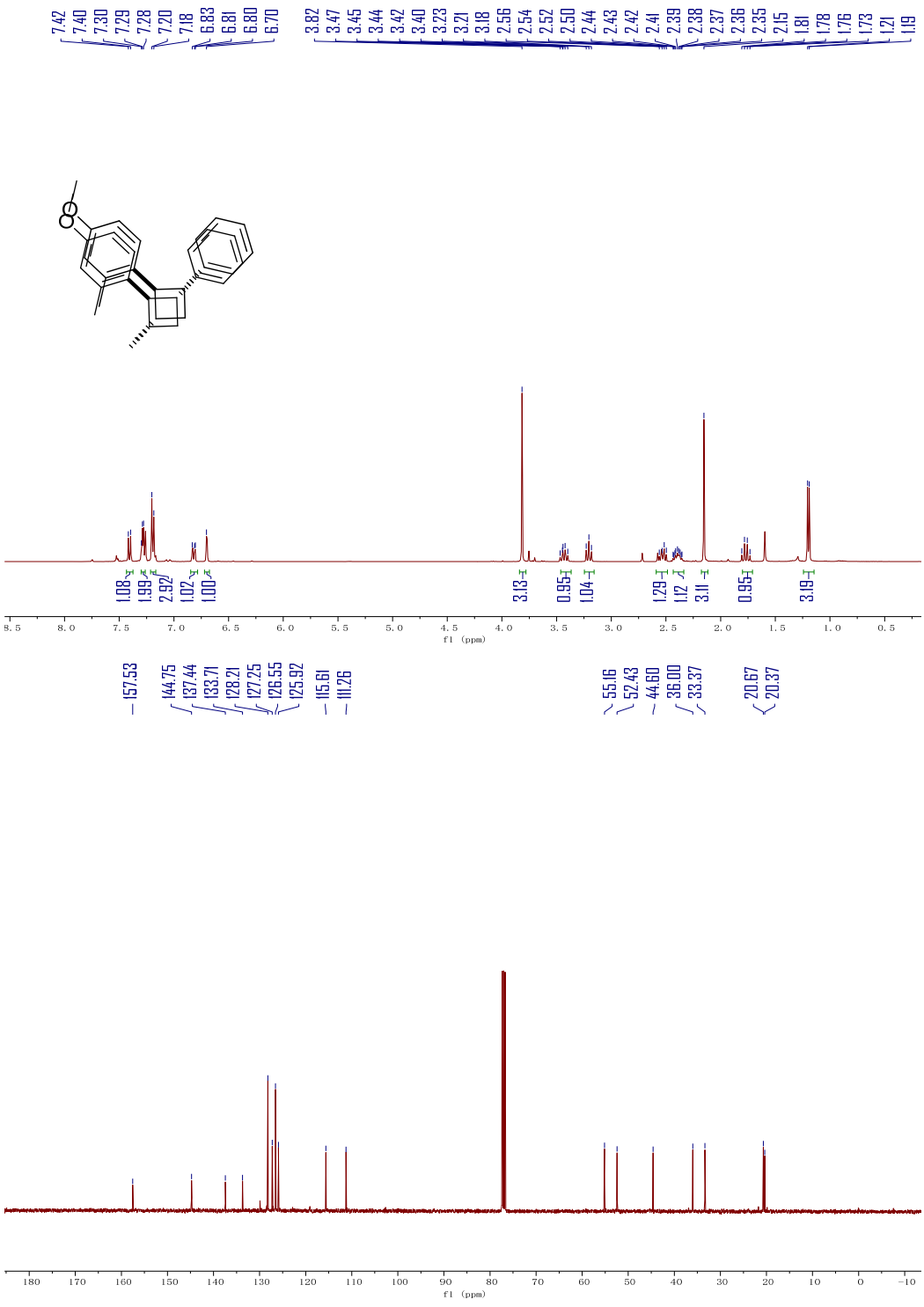
53.65
47.05
44.59
34.76
29.87
20.28
19.84
O
f1 (ppm)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7.18
6.83
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6.80
6.70
3.82
3.47
3.45
3.44
3.42
3.40
3.23
3.21
3.18
2.56
2.54
2.52
2.50
2.44
2.43
2.42
2.41
2.39
2.38
2.37
2.36
2.35
2.15
1.81
1.78
1.76
1.73
1.21
1.19
1.08
1.99
2.92
1.02
1.00
3.13
0.95
1.04
1.29
1.12
3.11
0.95
3.19
f1 (ppm)
157.53
144.75
137.44
133.71
128.21
127.25
126.55
125.92
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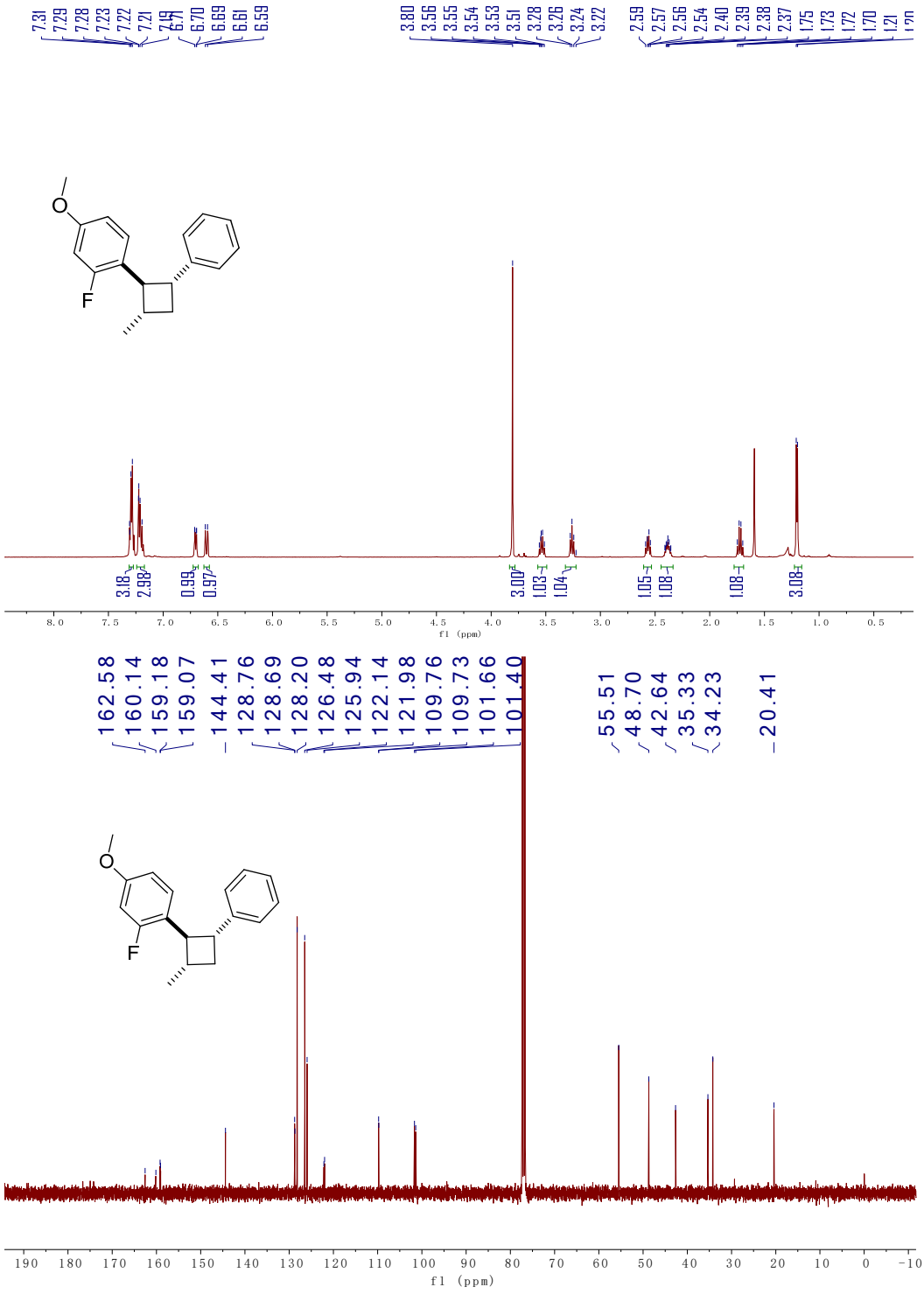
111.26
55.16
52.43
44.60
36.00
33.37
20.67
20.37
f1 (ppm)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6.59
3.80
3.56
3.55
3.54
3.53
3.51
3.28
3.26
3.24
3.22
2.59
2.57
2.56
2.54
2.40
2.39
2.38
2.37
1.75
1.73
1.72
1.70
1.21
O
F
3.18
2.98
0.99
0.97
3.00
1.03
1.04
1.05
1.08
1.08
3.08
f1 (ppm)
162.58
160.14
159.18
159.07
144.41
128.76
128.69
128.20
126.48
125.94
122.14
121.98
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55.51
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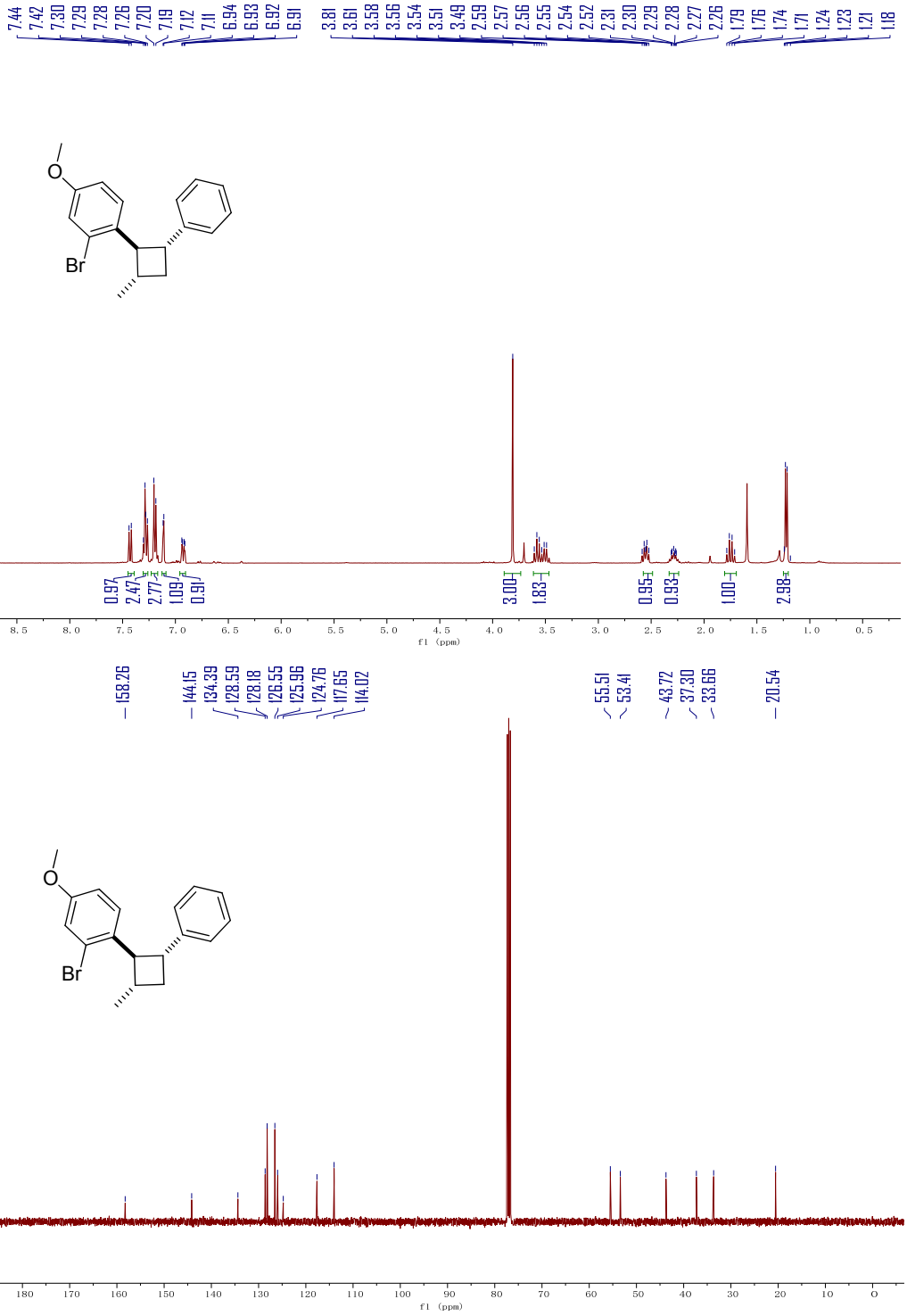
34.23
20.41
O
F
f1 (ppm)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7.29
7.28
7.26
7.20
7.19
7.12
7.11
6.94
6.93
6.92
6.91
3.81
3.61
3.58
3.56
3.54
3.51
3.49
2.59
2.57
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2.54
2.52
2.31
2.30
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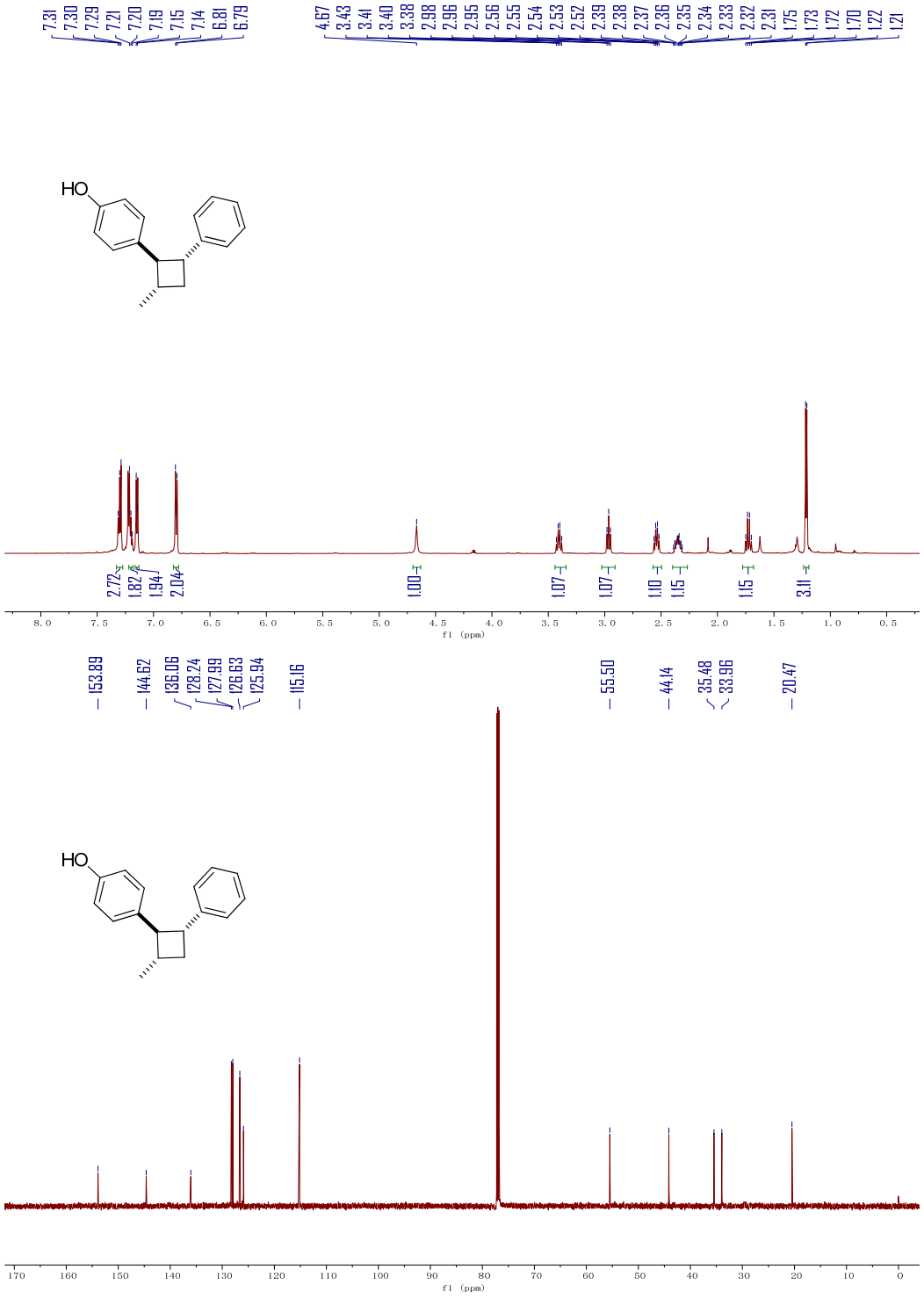
2.28
2.27
2.26
1.79
1.76
1.74
1.71
1.24
1.23
1.21
1.18
O
Br
0.97
2.47
2.77
1.09
0.91
3.00
1.83
0.95
0.93
1.00
2.98
f1 (ppm)
158.26
144.15
134.39
128.59
128.18
126.55
125.96
124.76
117.65
114.02
55.51
53.41
43.72
37.30
33.66
20.54
O
Br
f1 (ppm)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6.79
4.67
3.43
3.41
3.40
3.38
2.98
2.96
2.95
2.56
2.55
2.54
2.53
2.52
2.39
2.38
2.37
2.36
2.35
2.34
2.33
2.32
2.31
1.75
1.73
1.72
1.70
1.22
1.21
HO
2.72
1.82
1.94
2.04
1.00
1.07
1.07
1.10
1.15
1.15
3.11
f1 (ppm)
153.89
144.62
136.06
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125.94
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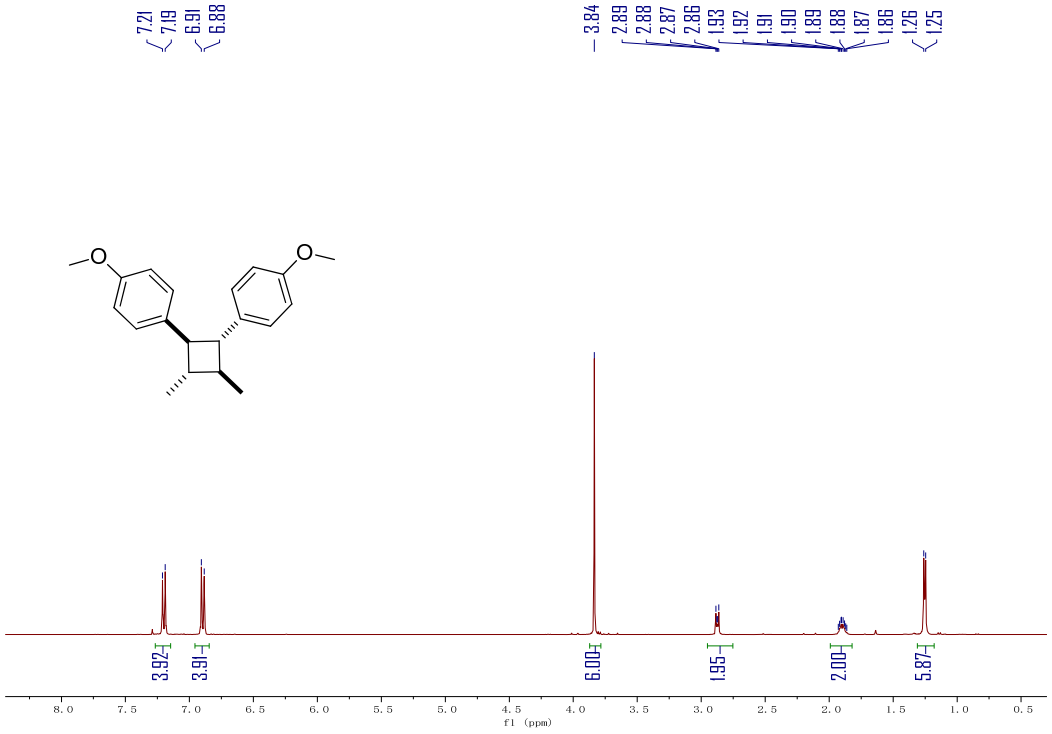
55.50
44.14
35.48
33.96
20.47
HO
f1 (ppm)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1.92
1.91
1.90
1.89
1.88
1.87
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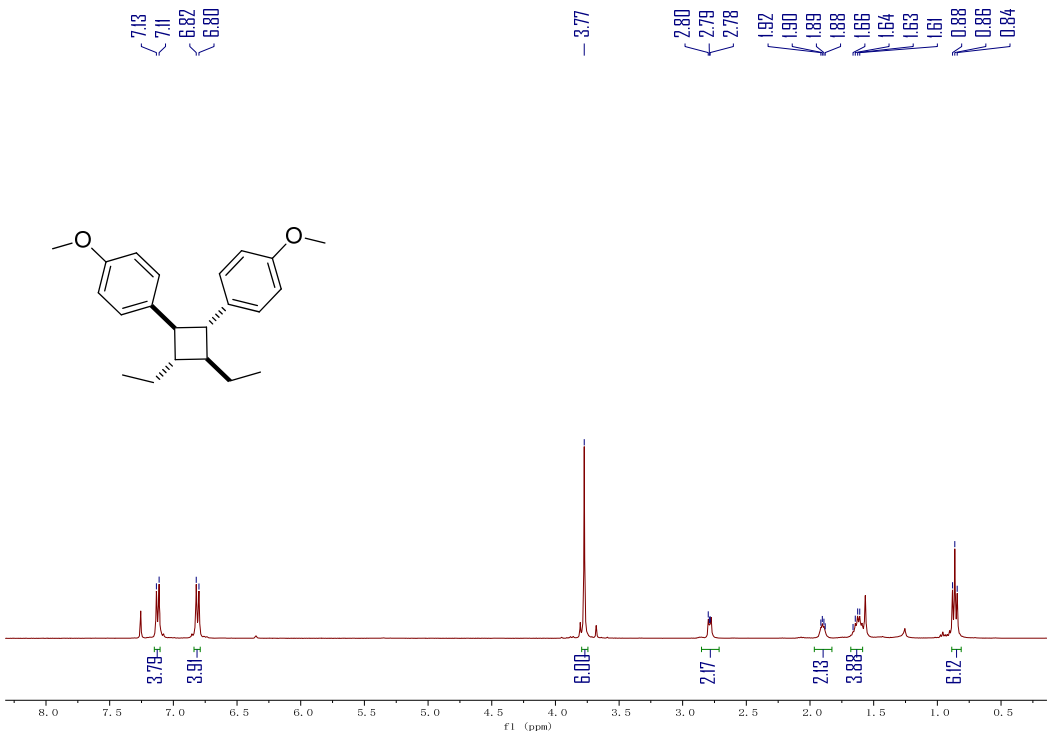
1.26
1.25
3.92
3.91
6.00
1.95
2.00
5.87
f1 (ppm)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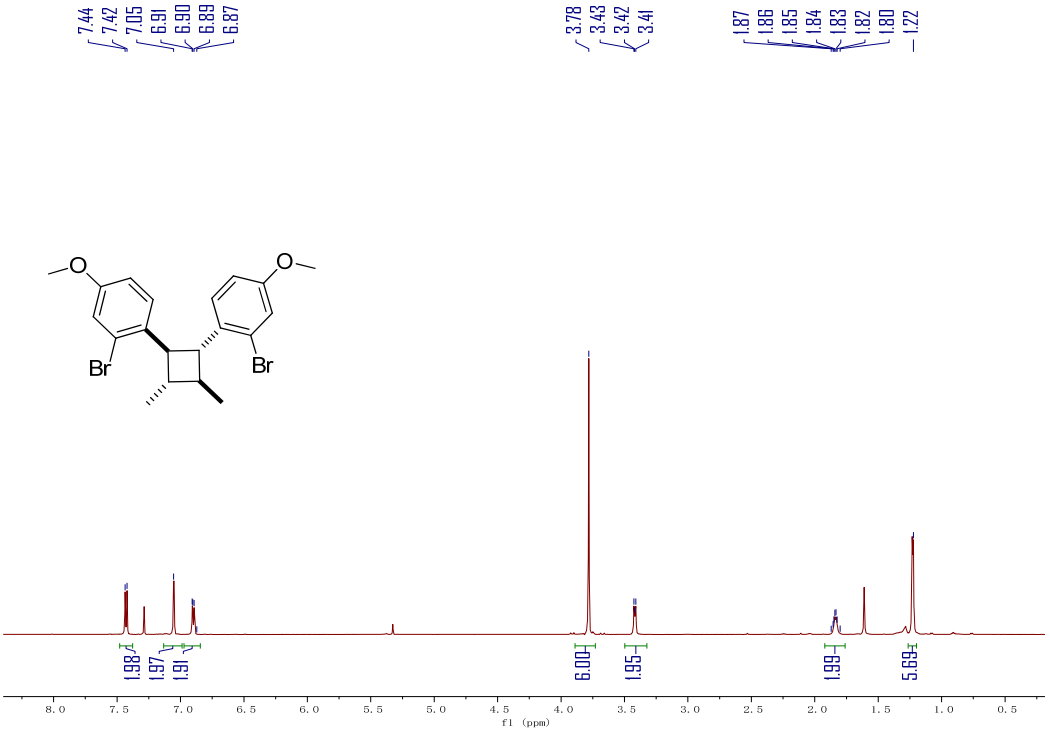
2.78
1.92
1.90
1.89
1.88
1.66
1.64
1.63
1.61
0.88
0.86
0.84
3.79
3.91
6.00
2.17
2.13
3.88
6.12
f1 (ppm)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7.42
7.05
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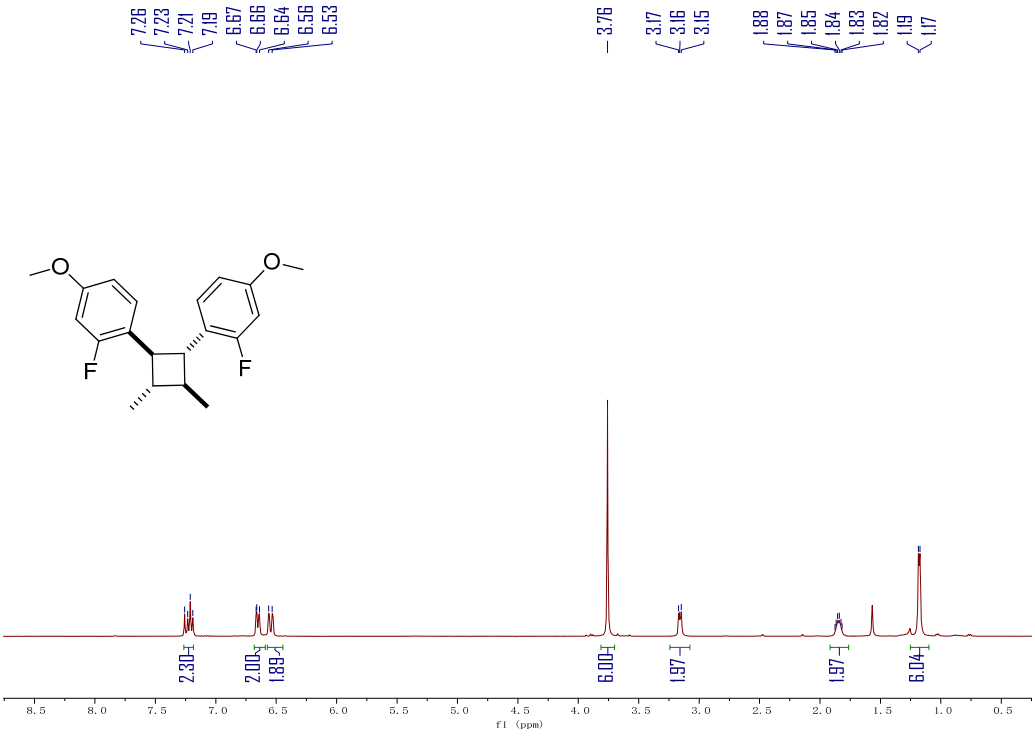
6.90
6.89
6.87
3.78
3.43
3.42
3.41
1.87
1.86
1.85
1.84
1.83
1.82
1.80
1.22
O
O
Br
Br
1.98
1.97
1.91
6.00
1.95
1.99
5.69
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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7.23
7.21
7.19
6.67
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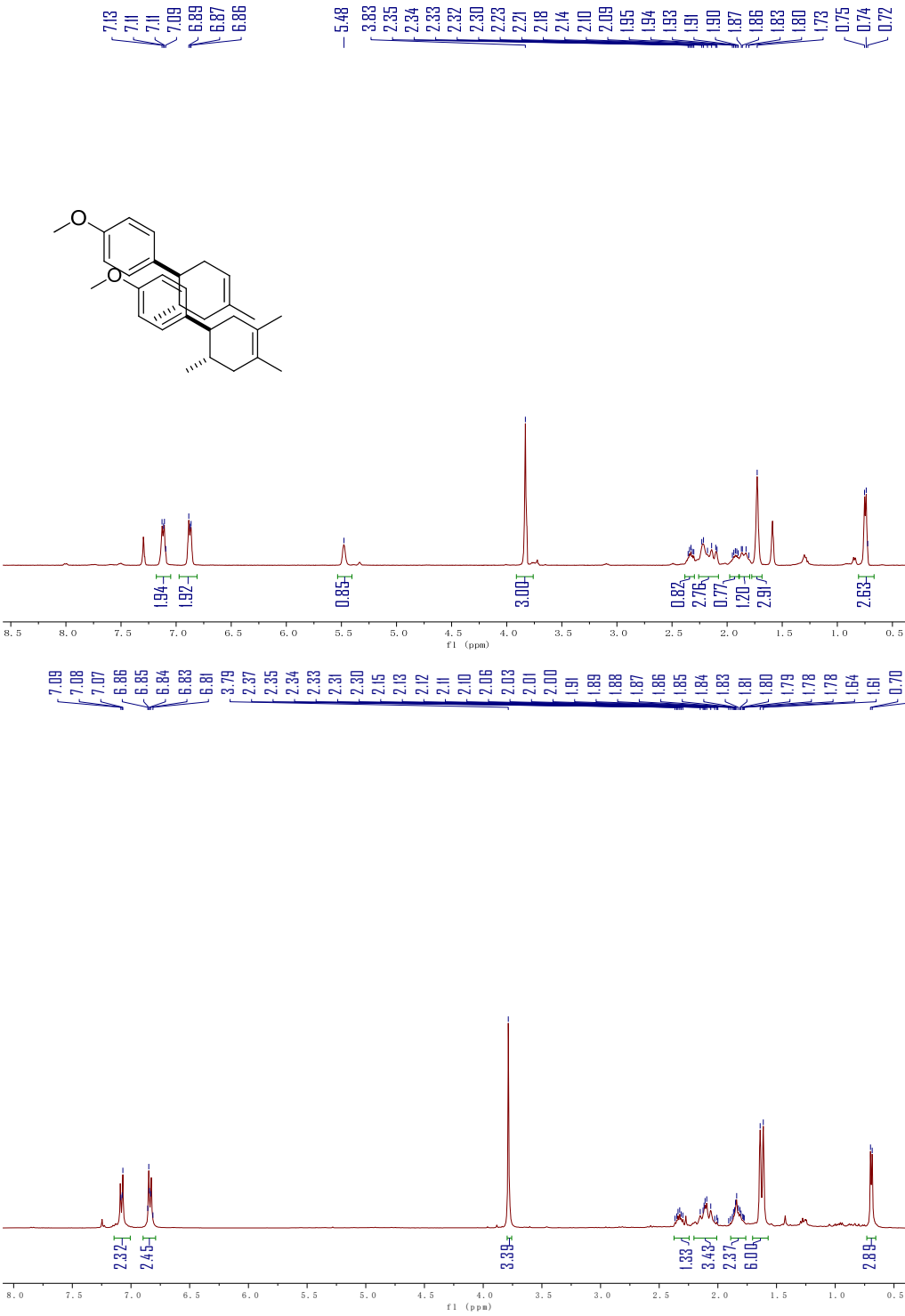
6.64
6.56
6.53
3.76
3.17
3.16
3.15
1.88
1.87
1.85
1.84
1.83
1.82
1.19
1.17
O
O
F
F
2.30
2.00
1.89
6.00
1.97
1.97
6.04
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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7.11
7.11
7.09
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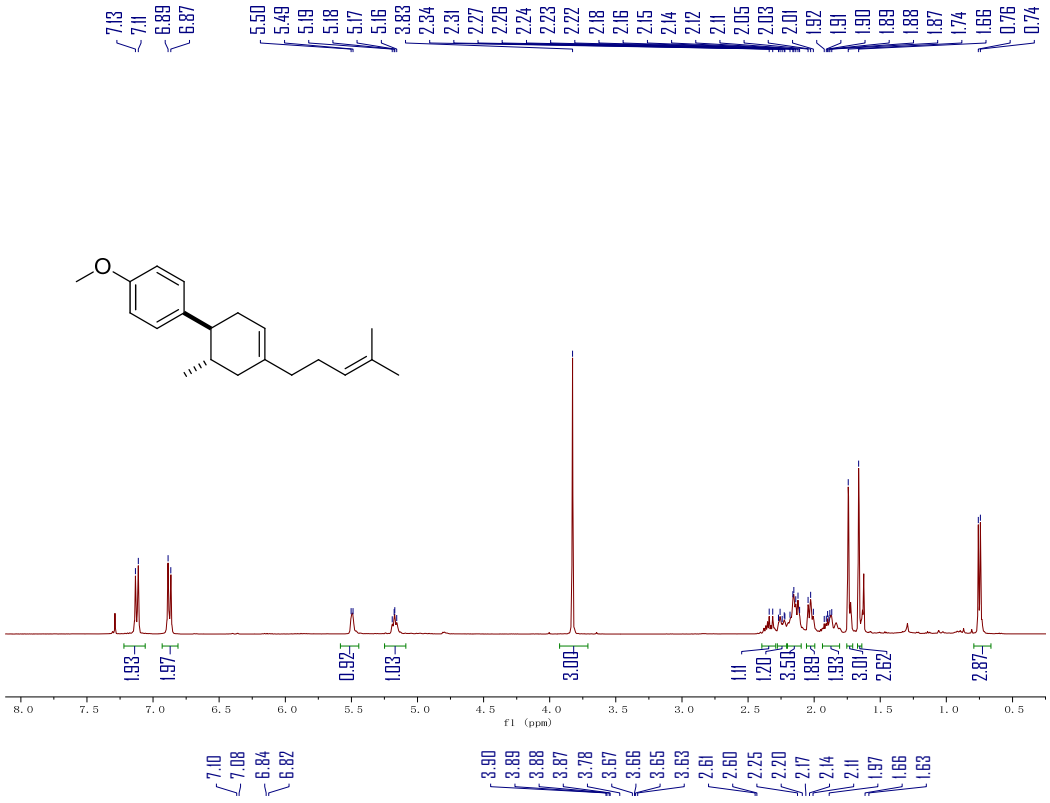
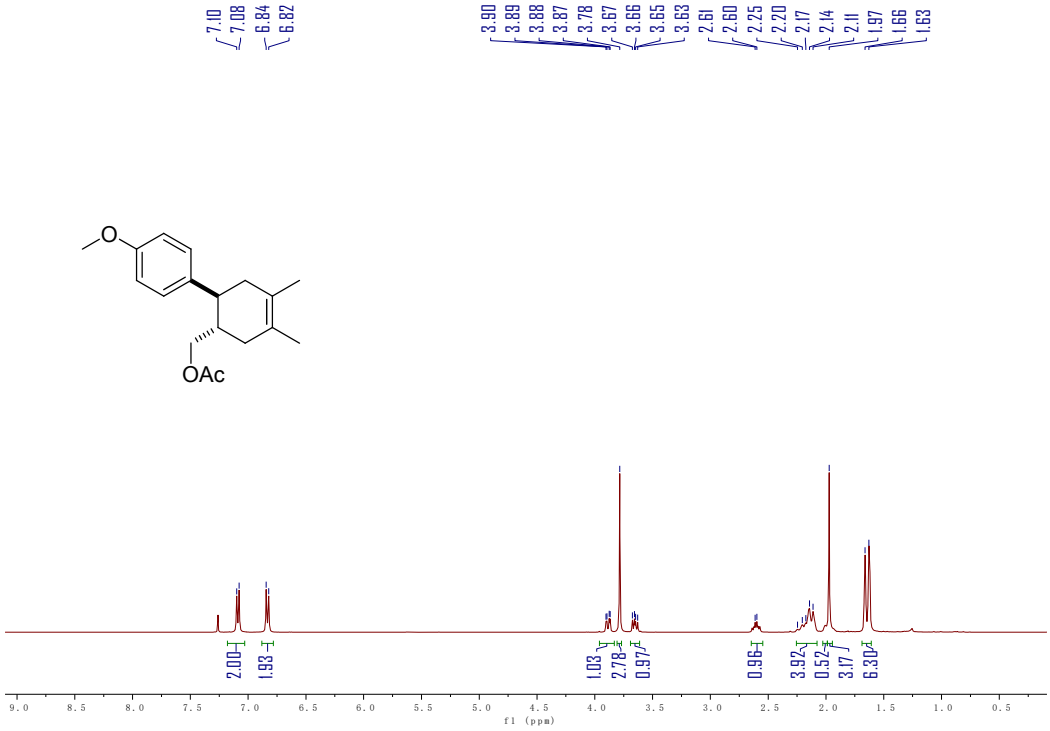
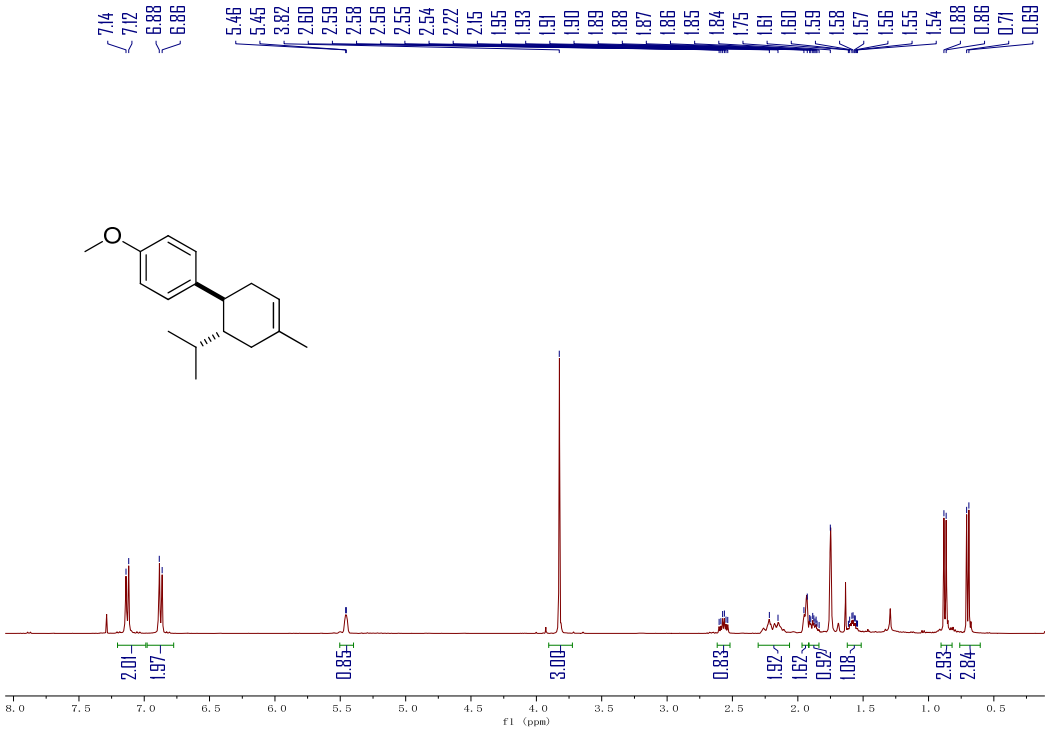
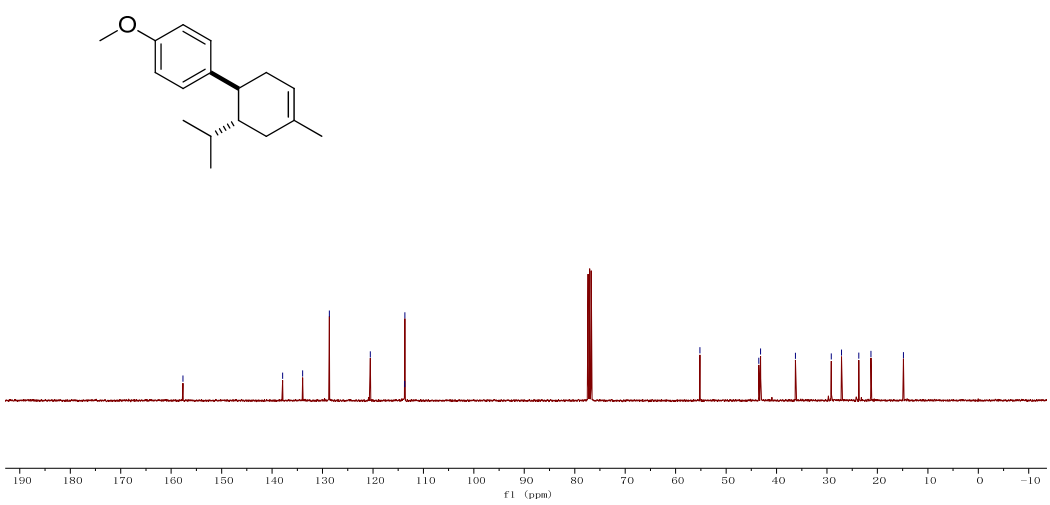
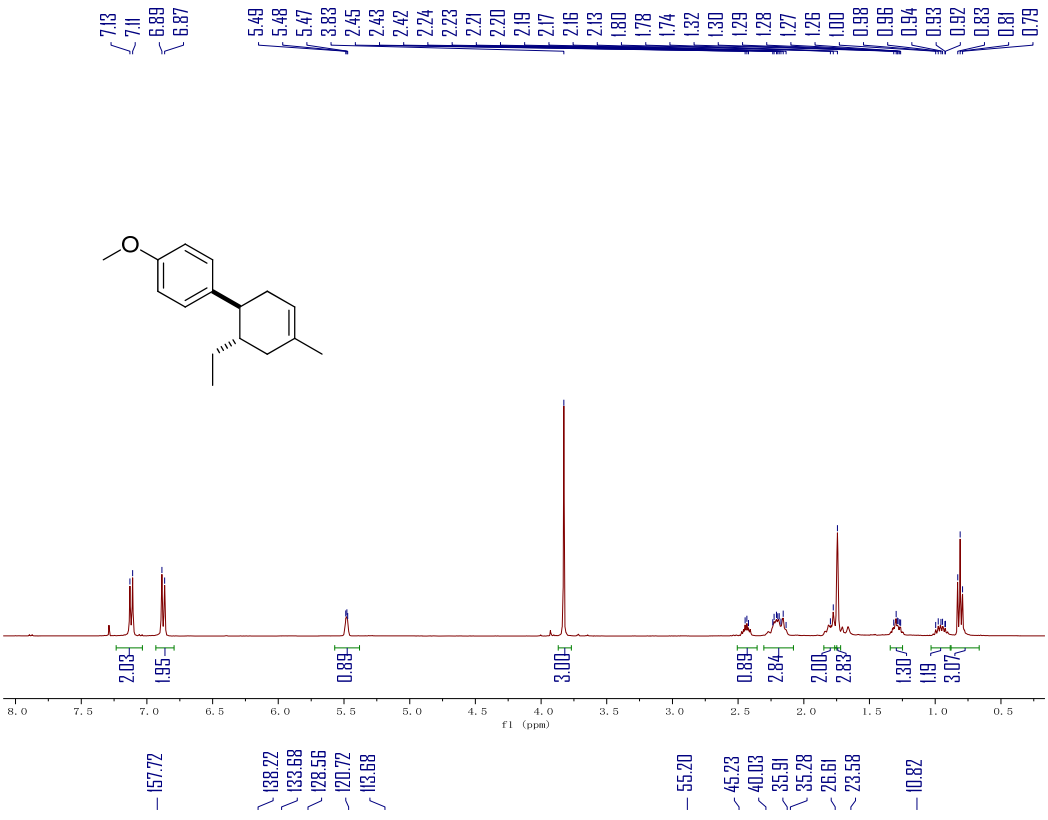
6.87
6.86
5.48
3.83
2.35
2.34
2.33
2.32
2.30
2.23
2.21
2.18
2.14
2.10
2.09
1.95
1.94
1.93
1.91
1.90
1.87
1.86
1.83
1.80
1.73
0.75
0.74
0.72
O
O
1.94
1.92
0.85
3.00
0.82
2.76
0.77
1.20
2.91
2.63
f1 (ppm)
7.09
7.08
7.07
6.86
6.85
6.84
6.83
6.81
3.79
2.37
2.35
2.34
2.33
2.31
2.30
2.15
2.13
2.12
2.11
2.10
2.06
2.03
2.01
2.00
1.91
1.89
1.88
1.87
1.86
1.85
1.84
1.83
1.81
1.80
1.79
1.78
1.78
1.64
1.61
0.70
0.69
2.32
2.45
3.39
1.33
3.43
2.37
6.00
2.89
f1 (ppm)

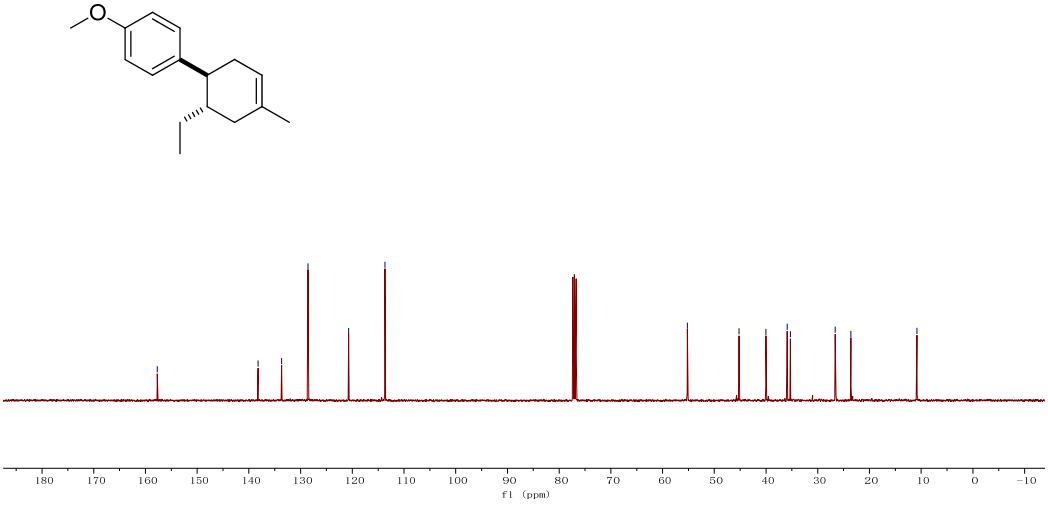

7.13
7.11
6.89
6.87
5.50
5.49
5.19
5.18
5.17
5.16
3.83
2.34
2.31
2.27
2.26
2.24
2.23
2.22
2.18
2.16
2.15
2.14
2.12
2.11
2.05
2.03
2.01
1.92
1.91
1.90
1.89
1.88
1.87
1.74
1.66
0.76
0.74
O
1.93
1.97
0.92
1.03
3.00
1.11
1.20
3.50
1.89
1.93
3.01
2.62
2.87
f1 (ppm)

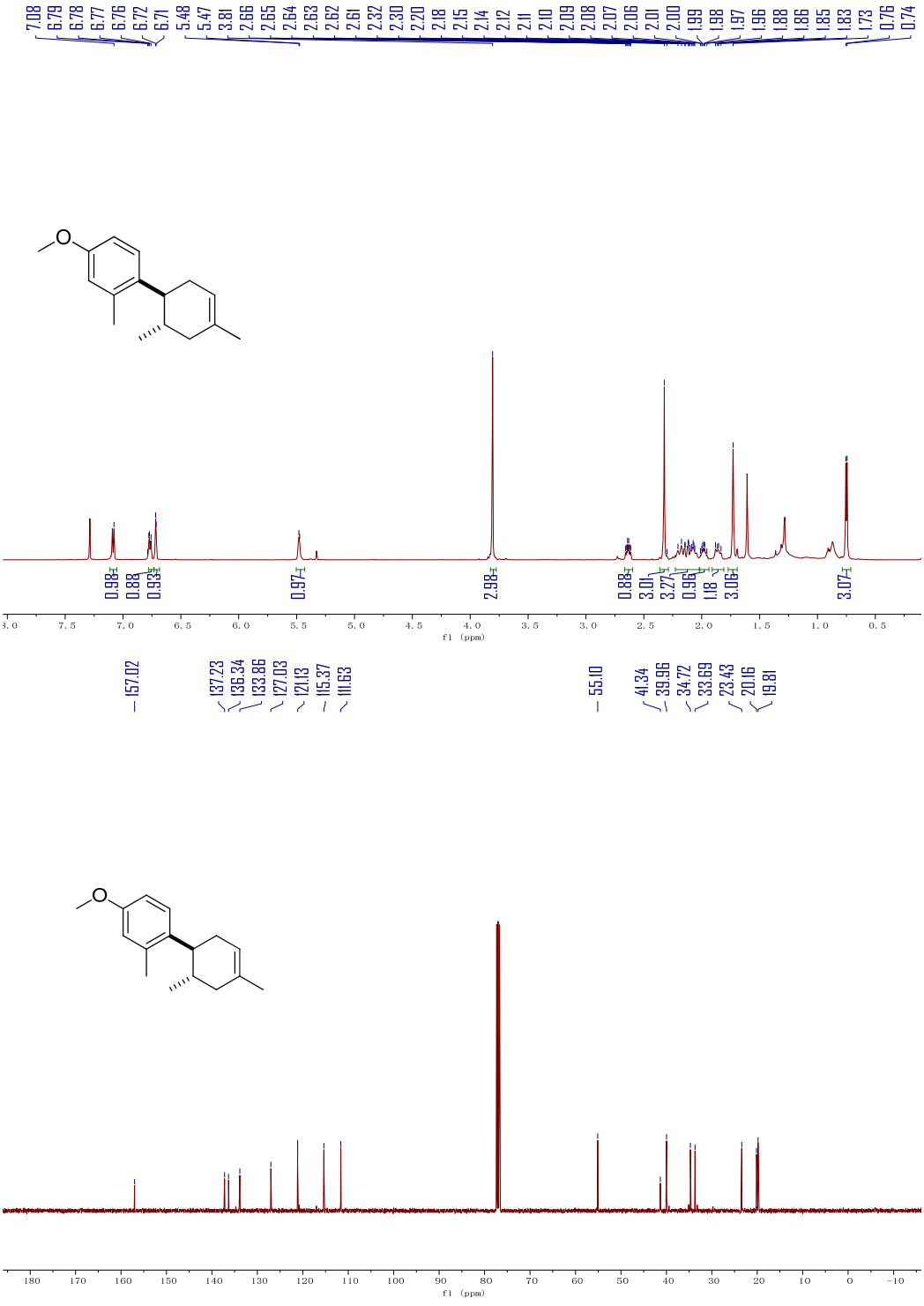

7.10
7.08
6.84
6.82
3.90
3.89
3.88
3.87
3.78
3.67
3.66
3.65
3.63
2.61
2.60
2.25
2.20
2.17
2.14
2.11
1.97
1.66
1.63
O
OAc
2.00
1.93
1.03
2.78
0.97
0.96
3.92
0.52
3.17
6.30
f1 (ppm)

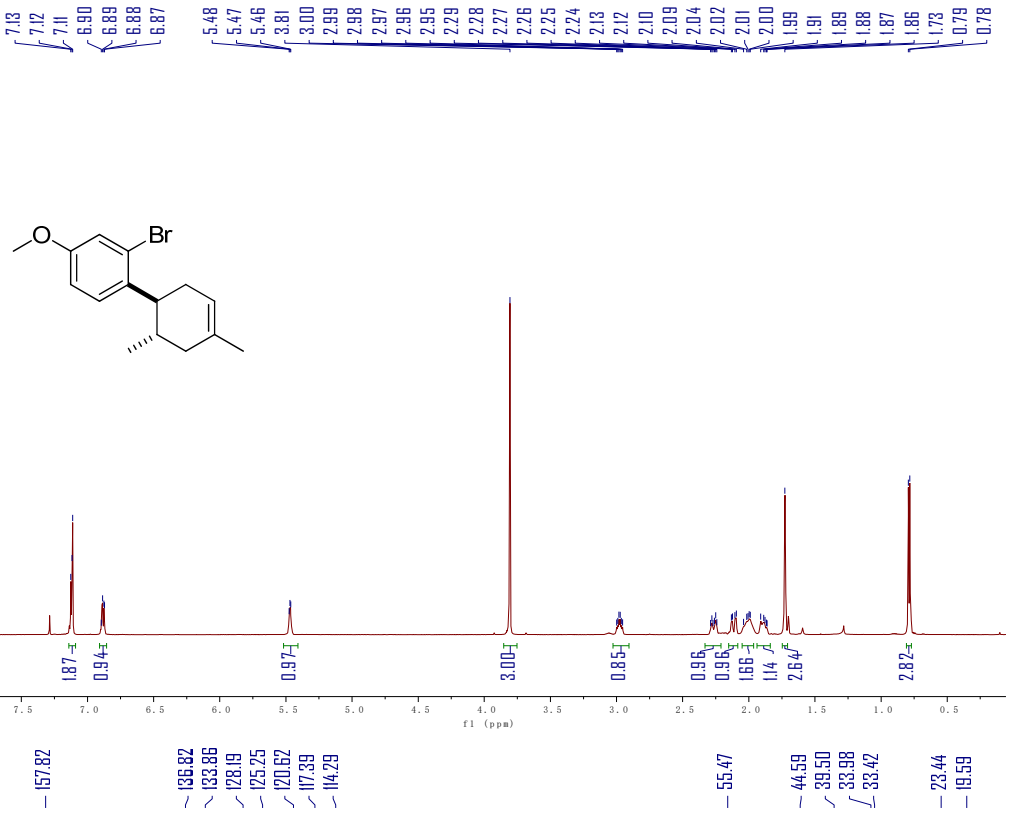

7.14
7.12
6.88
6.86
5.46
5.45
3.82
2.60
2.59
2.58
2.56
2.55
2.54
2.22
2.15
1.95
1.93
1.91
1.90
1.89
1.88
1.87
1.86
1.85
1.84
1.75
1.61
1.60
1.59
1.58
1.57
1.56
1.55
1.54
0.88
0.86
0.71
0.69
2.01
1.97
0.85
3.00
0.83
1.92
1.62
0.92
1.08
2.93
2.84
f1 (ppm)

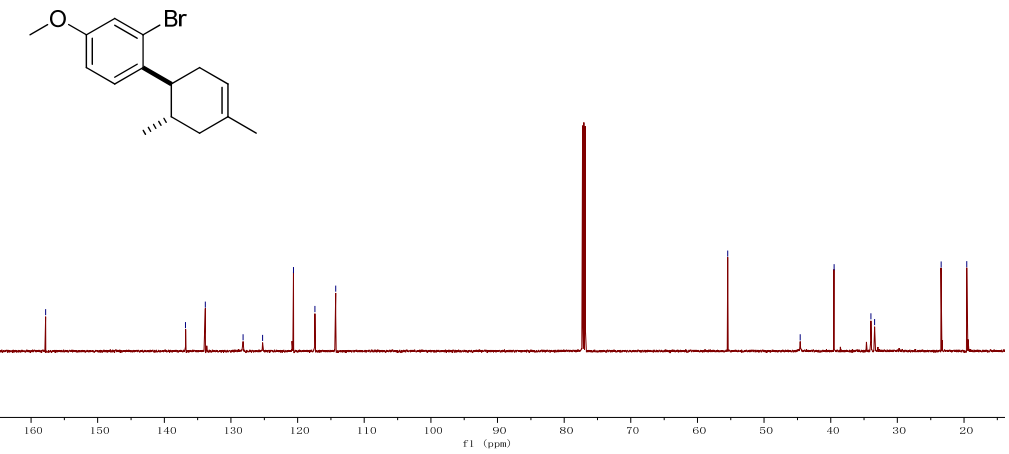

157.65
137.91
133.94
128.65
120.53
113.72
113.68
55.18
43.51
43.16
36.25
29.13
27.12
23.70
21.28
14.85
f1 (ppm)

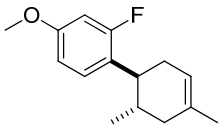

7.13
7.11
6.89
6.87
5.49
5.48
5.47
3.83
2.45
2.43
2.42
2.24
2.23
2.21
2.20
2.19
2.17
2.16
2.13
1.80
1.78
1.74
1.32
1.30
1.29
1.28
1.27
1.26
1.00
0.98
0.96
0.94
0.93
0.92
0.83
0.81
0.79
O
2.03
1.95
0.89
3.00
0.89
2.84
2.00
2.83
1.30
1.19
3.07
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

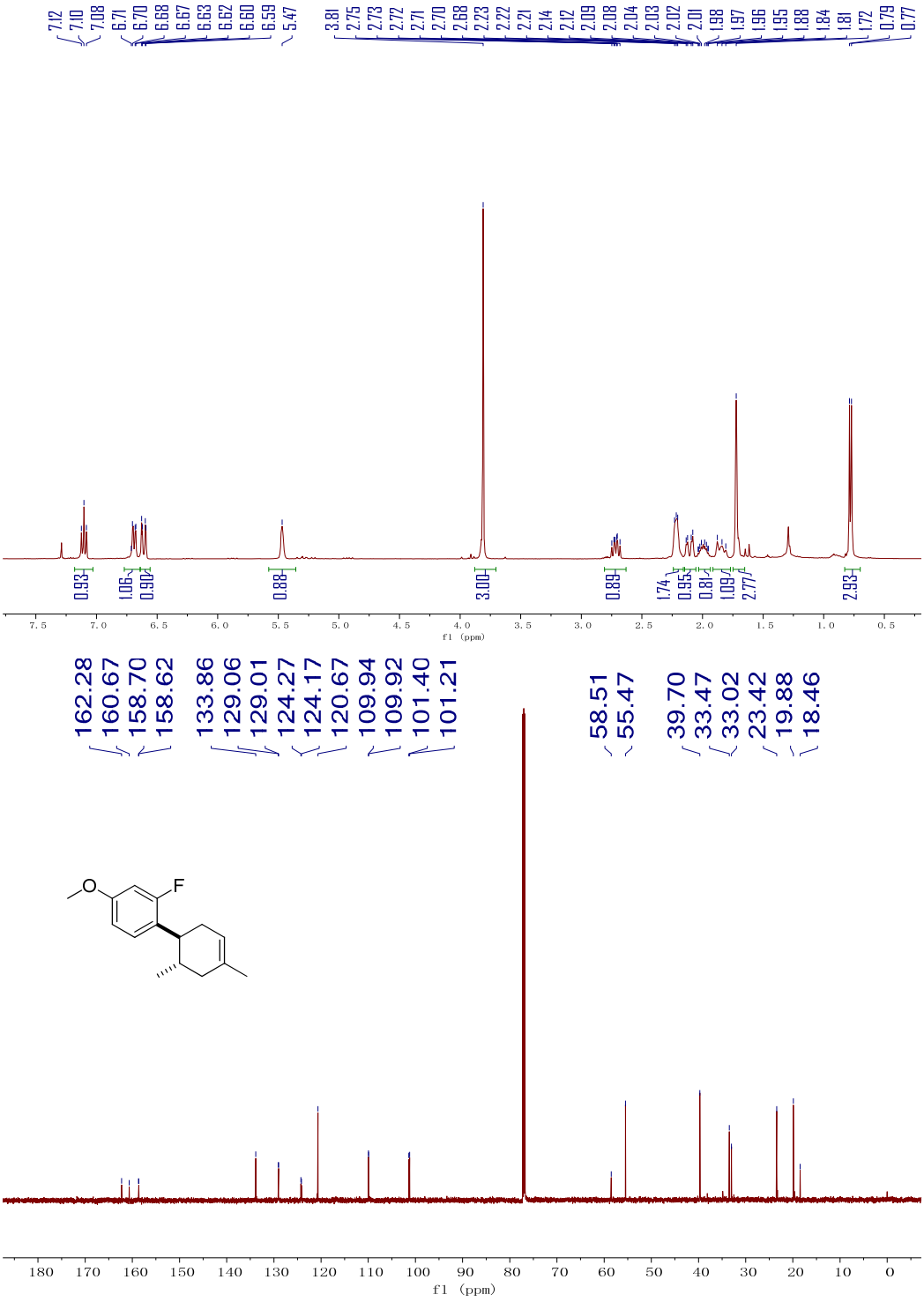

157.72
138.22
133.68
128.56
120.72
113.68
55.20
45.23
40.03
35.91
35.28
26.61
23.58
10.82
O
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

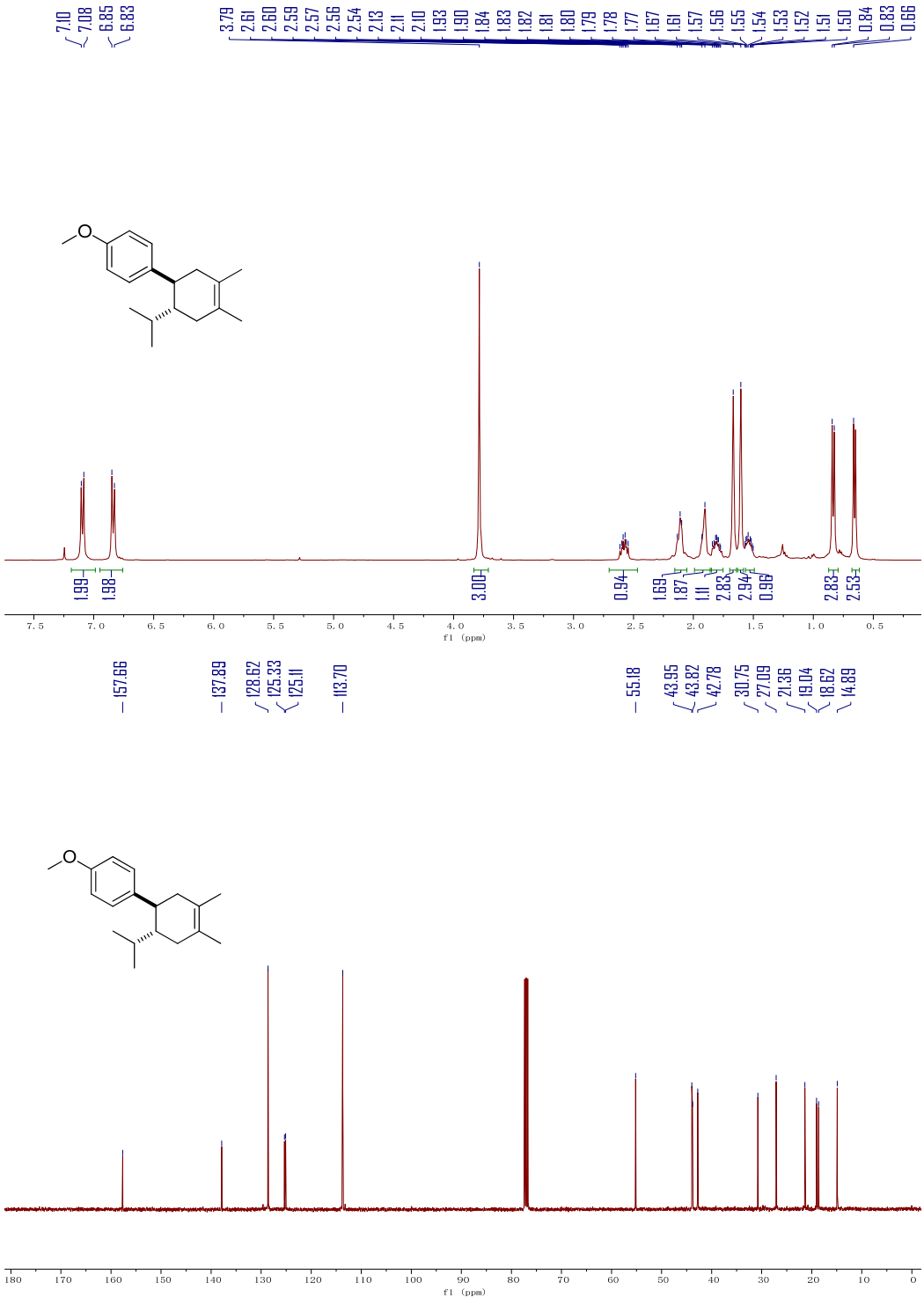

7.08
6.79
6.78
6.77
6.76
6.72
6.71
5.48
5.47
3.81
2.66
2.65
2.64
2.63
2.62
2.61
2.32
2.30
2.20
2.18
2.15
2.14
2.12
2.11
2.10
2.09
2.08
2.07
2.06
2.01
2.00
1.99
1.98
1.97
1.96
1.88
1.86
1.85
1.83
1.73
0.76
0.74
O
0.98
0.88
0.93
0.97
2.98
0.88
3.01
3.27
0.96
1.18
3.06
3.07
f1 (ppm)
157.02
137.23
136.34
133.86
127.03
121.13
115.37
111.63
55.10
41.34
39.96
34.72
33.69
23.43
20.16
19.81
O
f1 (ppm)


7.13
7.12
7.11
6.90
6.89
6.88
6.87
5.48
5.47
5.46
3.81
3.00
2.99
2.98
2.97
2.96
2.95
2.29
2.28
2.27
2.26
2.25
2.24
2.13
2.12
2.10
2.09
2.04
2.02
2.01
2.00
1.99
1.91
1.89
1.88
1.87
1.86
1.73
0.79
0.78
O
Br
1.87
0.94
0.97
3.00
0.85
0.96
0.96
1.66
1.14
2.64
2.82
f1 (ppm)
157.82
136.82
133.86
128.19
125.25
120.62
117.39
114.29
55.47
44.59
39.50
33.98
33.42
23.44
19.59

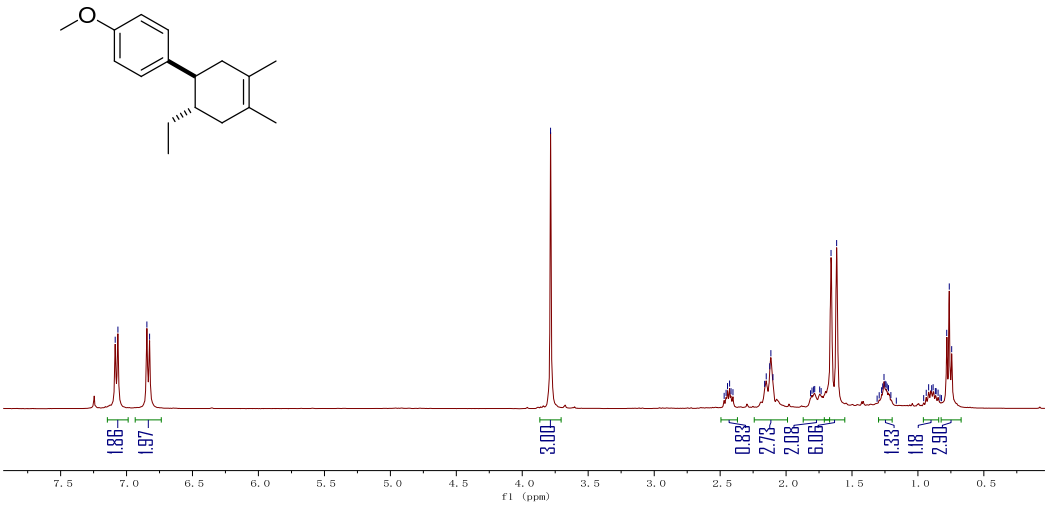

O
Br
f1 (ppm)


O
F

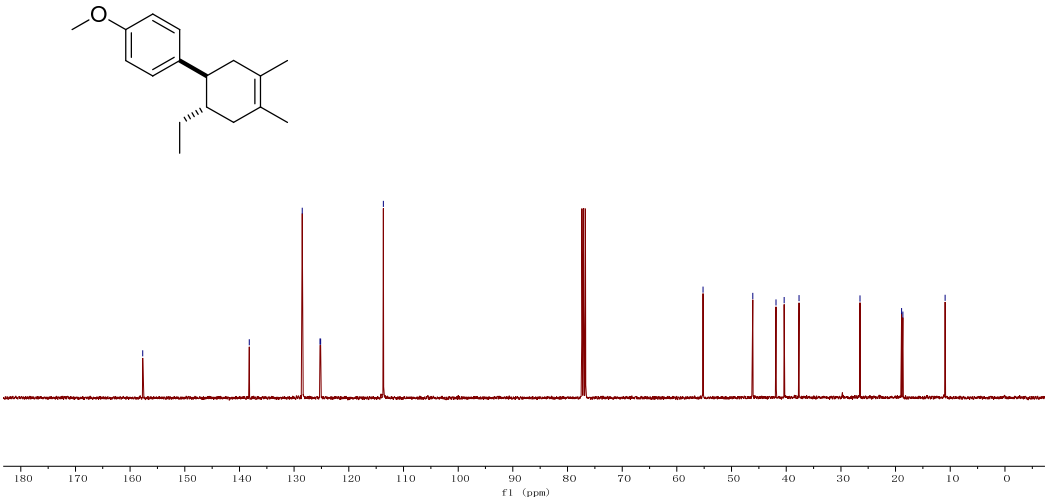

7.12
7.10
7.08
6.71
6.70
6.68
6.67
6.63
6.62
6.60
6.59
5.47
3.81
2.75
2.73
2.72
2.71
2.70
2.68
2.23
2.22
2.21
2.14
2.12
2.09
2.08
2.04
2.03
2.02
2.01
1.98
1.97
1.96
1.95
1.88
1.84
1.81
1.72
0.79
0.77
0.93
1.06
0.90
0.88
3.00
0.89
1.74
0.95
0.81
1.09
2.77
2.93
f1 (ppm)
162.28
160.67
158.70
158.62
133.86
129.06
129.01
124.27
124.17
120.67
109.94
109.92
101.40
101.21
58.51
55.47
39.70
33.47
33.02
23.42
19.88
18.46
O
F
f1 (ppm)

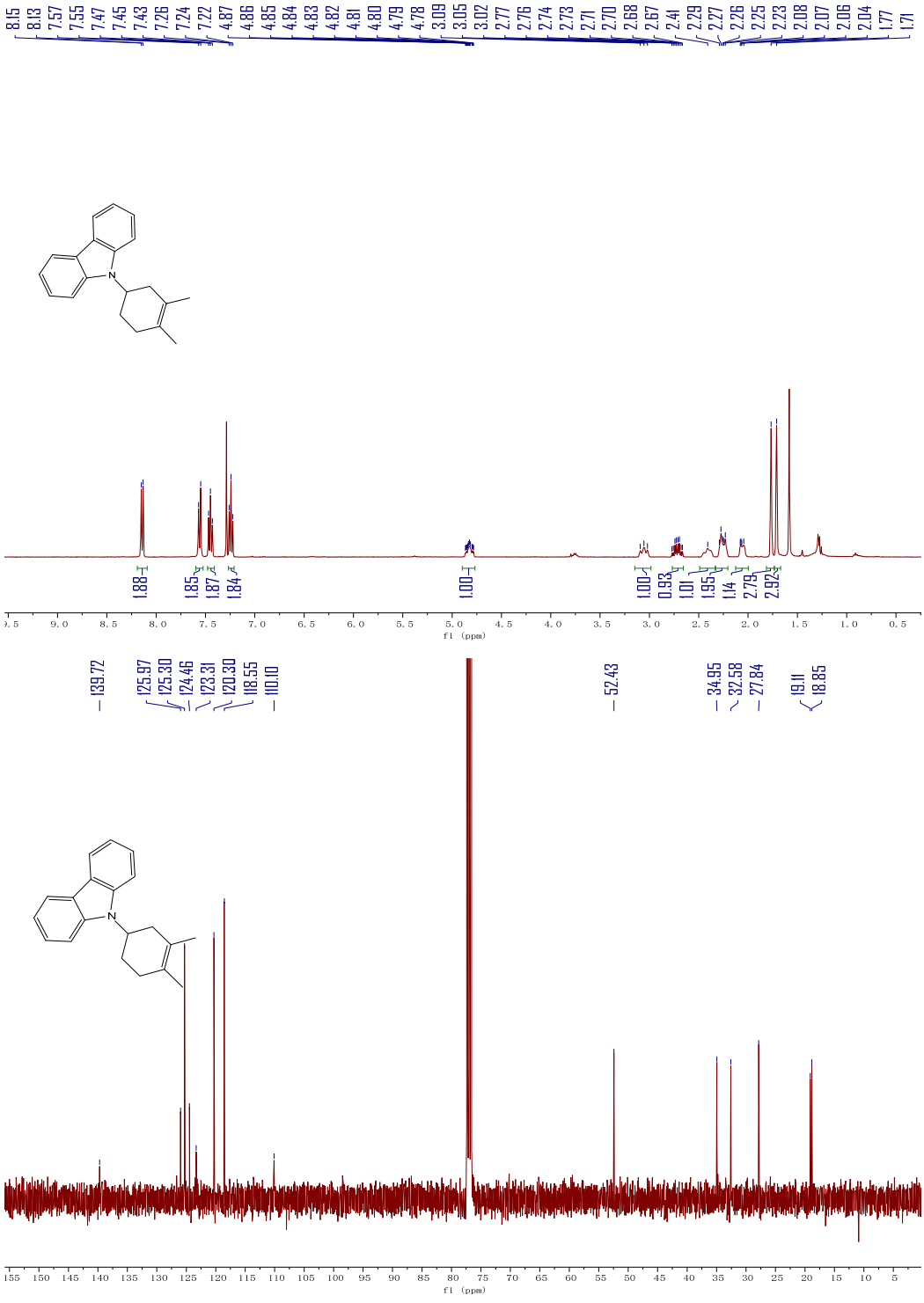

7.10
7.08
6.85
6.83
3.79
2.61
2.60
2.59
2.57
2.56
2.54
2.13
2.11
2.10
1.93
1.90
1.84
1.83
1.82
1.81
1.80
1.79
1.78
1.77
1.67
1.61
1.57
1.56
1.55
1.54
1.53
1.52
1.51
1.50
0.84
0.83
0.66
O
1.99
1.98
3.00
0.94
1.69
1.87
1.11
2.83
2.94
0.99
2.83
2.53
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)
157.66
137.89
128.62
125.33
125.11
113.70
55.18
43.95
43.82
42.78
30.75
27.09
21.36
19.04
18.62
14.89
O
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

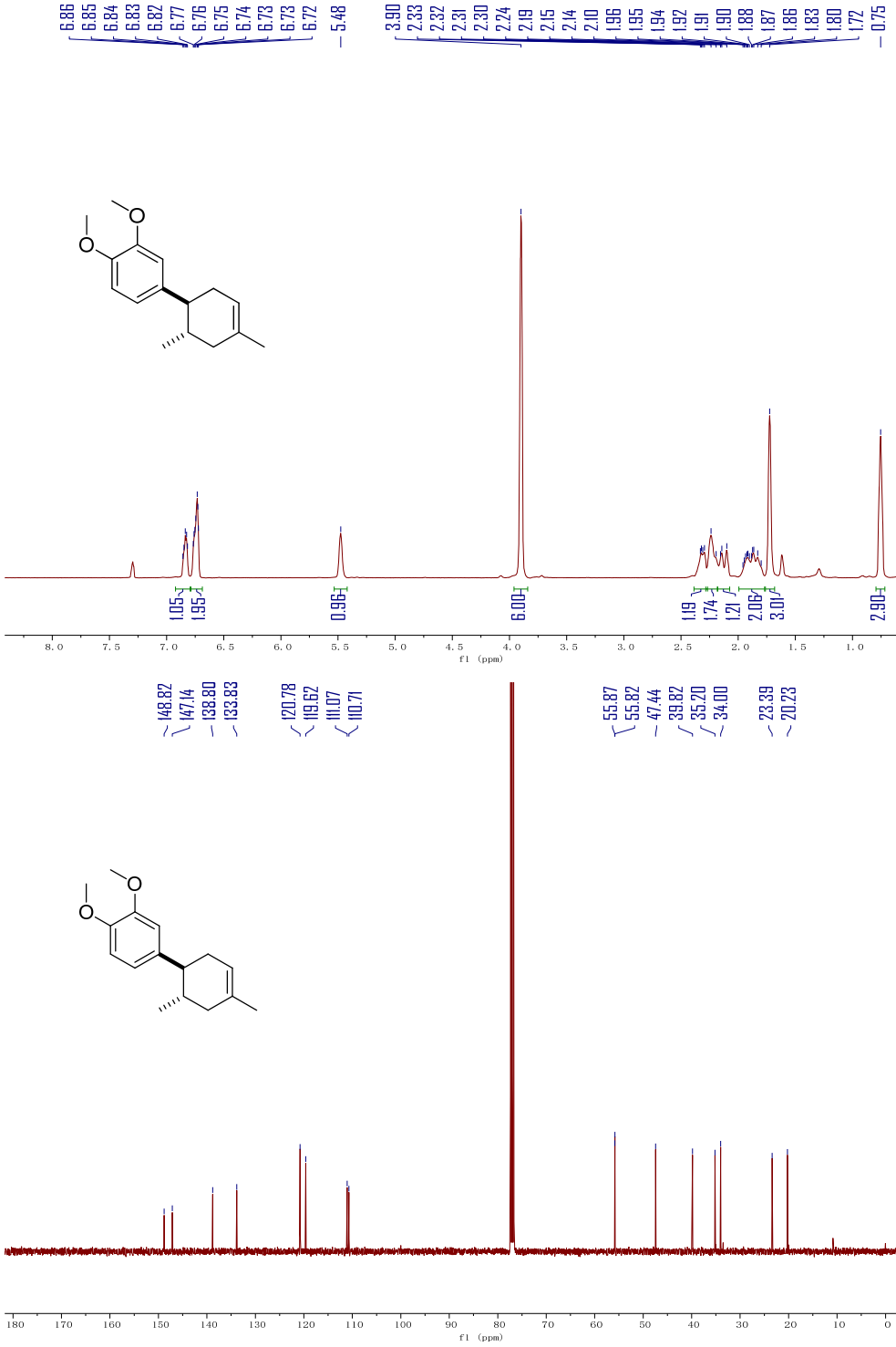

7.09
7.07
6.85
6.83
3.79
2.47
2.45
2.44
2.43
2.42
2.40
2.16
2.15
2.12
2.11
2.10
1.81
1.80
1.79
1.78
1.75
1.73
1.66
1.62
1.29
1.28
1.27
1.26
1.25
1.24
1.23
1.22
1.20
0.94
0.92
0.90
0.88
0.87
0.86
0.85
0.78
0.76
0.74


O
1.86
1.97
3.00
0.83
2.73
2.08
6.06
1.33
1.18
2.90
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

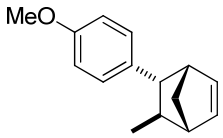

157.71
138.21
128.52
125.28
125.17
113.69
55.20
46.12
41.86
40.38
37.67
26.51
18.91
18.66
10.94


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

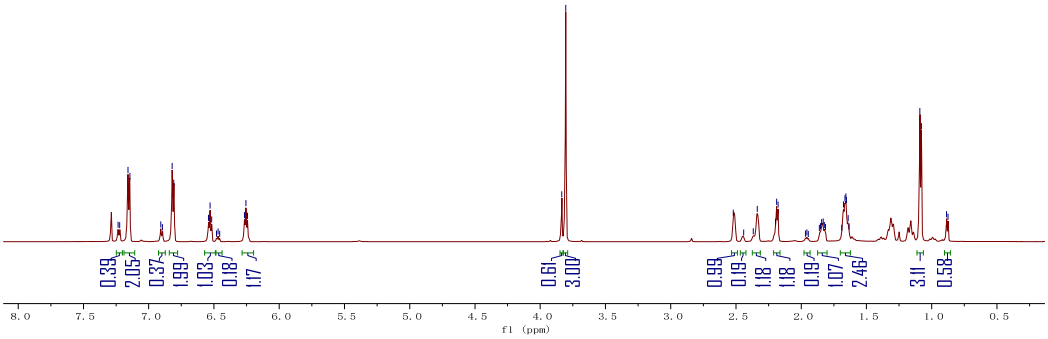

8.15
8.13
7.57
7.55
7.47
7.45
7.43
7.26
7.24
7.22
4.87
4.86
4.85
4.84
4.83
4.82
4.81
4.80
4.79
4.78
3.09
3.05
3.02
2.77
2.76
2.74
2.73
2.71
2.70
2.68
2.67
2.41
2.29
2.27
2.26
2.25
2.23
2.08
2.07
2.06
2.04
1.77
1.71
N
1.88
1.85
1.87
1.84
1.00
1.00
0.93
1.01
1.95
1.14
2.79
2.92
f1 (ppm)
139.72
125.97
125.30
124.46
123.31
120.30
118.55
110.10
52.43
34.95
32.58
27.84
19.11
18.85
N
f1 (ppm)


6.86
6.85
6.84
6.83
6.82
6.77
6.76
6.75
6.74
6.73
6.73
6.72
5.48
3.90
2.33
2.32
2.31
2.30
2.24
2.19
2.15
2.14
2.10
1.96
1.95
1.94
1.92
1.91
1.90
1.88
1.87
1.86
1.83
1.80
1.72
0.75
1.05
1.95
0.96
6.00
1.19
1.74
1.21
2.06
3.01
2.90
f1 (ppm)
148.82
147.14
138.80
133.83
120.78
119.62
111.07
110.71
55.87
55.82
47.44
39.82
35.20
34.00
23.38
20.23
f1 (ppm)


7.24
7.22
7.16
7.14
6.91
6.90
6.82
6.81
6.54
6.53
6.52
6.48
6.47
6.27
6.25
6.24
3.83
3.80
2.52
2.44
2.37
2.34
2.20
2.19
2.18
1.96
1.86
1.85
1.84
1.83
1.82
1.81
1.69
1.68
1.67
1.66
1.65
1.64
1.63
1.09
1.08
0.89
0.87


MeO

5:1 *endo*:*exo*


0.39
2.05
0.37
1.99
1.03
0.18
1.17
0.61
3.00
0.99
0.19
1.18
1.18
0.19
1.07
2.46
3.11
0.58
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)