Electronic Supplementary Material (ESI) for ChemComm.

Decarboxylation-Triggered Homo-Nazarov Cyclization of Cyclic Enol Carbonates Catalyzed by Rhenium Complex

Natsuki Kimaru, Keiichi Komatsuki, Kodai Saito and Tohru Yamada*

Department of Chemistry, Keio University,

3-14-1 Hiyoshi, Kohoku-ku, Yokohama 223-8522, Japan

Table of Contents

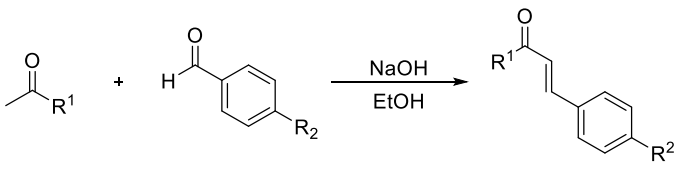
1. General information

The ^{1}H and ^{13}C NMR spectra were recorded with a JEOL model AL-400 or ECS-400 spectrometer using $CDCl_3$ and benzene-d_6 as the solvent. The IR spectra were measured with a Thermo Electron Corporation model NICOLET 6700 FT-IR spectrometer. The melting points were measured with a Stanford Research Systems MPA100. The ESI high resolution mass spectra were obtained using a Waters LCT Premier XE mass spectrometer. Column chromatography was conducted on silica gel (CHROMATOREX PSQ 100B, Fuji Silysia Chemical, Ltd.). Rhenium pentacarbonyl bromide was purchased from Sigma-Aldrich. The dehydrated toluene, 1,8-Diazabicyclo[5.4.0]undec-7-ene (DBU), 4-phenyl-1-butyne were purchased from Wako Pure Chemical Industries, Ltd., and used without further purification. Ethynylbenzene was purchased from Tokyo Chemical Industry Co., Ltd., and used without further purification. The dehydrated CH_2Cl_2, dehydrated tetrahydrofuran (THF), the solution of *n*-BuLi (1.6 M in hexane) were purchased from Kanto Chemical Co., Inc.

2. Method for the preparation of substrates and characterization data

2.1 General procedure for the synthesis of propargyl alcohols

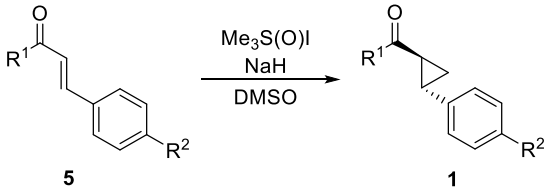
2.1.1 Preparation of enone derivatives (**5a**, **5b**, **5c**, **5d**, **5e**, **5f**, **5g**)



5a: R^1 = *i*-Pr, R^2 = *p*-MeO **5e**: R^1 = *t*-Bu, R^2 = *p*-MeO
5b: R^1 = *i*-Pr, R^2 = *p*-MeS **5f** : R^1 = *t*-Bu, R^2 = *m*-MeO
5c: R^1 = *i*-Pr, R^2 = *p*-NMe_2 **5g**: R^1 = *t*-Bu, R^2 = *p*-Br
5d: R^1 = *i*-Pr, R^2 = H

The enones **5a-5g** were synthesized according to the literature.[1] The corresponding ketone (40 mmol) was dissolved to EtOH (40 mL) and the solution was cooled to 0 ºC. Benzaldehyde derivative (44 mmol) was added to the solution, then 10wt% aq. NaOH (5 mmol) was added dropwise over 5 min. After being stirred overnight, the reaction mixture was refluxed for 1-2 h. The reaction mixture was neutralized with 1 M aq. HCl, then diluted with water (100 mL) and extracted three times with Et_2O. The combined organic layer was washed with sat. aq. $NaHCO_3$, and dried over Na_2SO_4. The solvent was removed under reduced pressure. The residue was purified by silica-gel flash column chromatography using *n*-hexane/EtOAc = 5/1 to obtain the desired enone **5a-5g**.

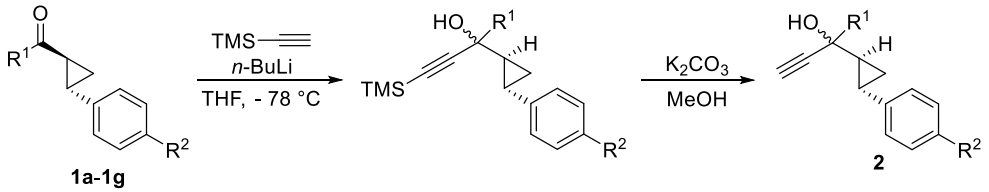
2.1.2 Procedure for the preparation of propargyl alcohols (**2a**, **2b**, **2c**, **2d**, **2e**, **2f**, **2g**)



5 1

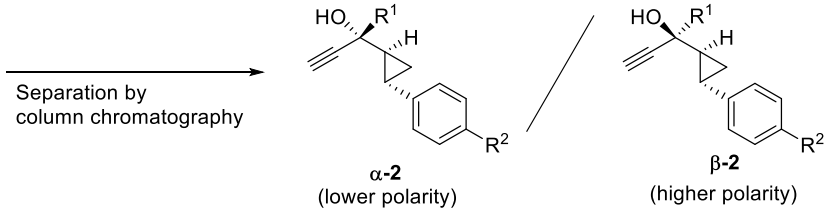
1a: R^1 = *i*-Pr, R^2 = *p*-MeO **1e**: R^1 = *t*-Bu, R^2 = *p*-MeO
1b: R^1 = *i*-Pr, R^2 = *p*-MeS **1f** : R^1 = *t*-Bu, R^2 = *m*-MeO
1c: R^1 = *i*-Pr, R^2 = *p*-NMe_2 **1g**: R^1 = *t*-Bu, R^2 = *p*-Br
1d: R^1 = *i*-Pr, R^2 = H

Trans-cyclopropylketones **1a-1g** were synthesized according to the literature.[2] To dehydrated DMSO solution (15 mL) of trimethylsulfoxonium iodide (6 mmol) was added a suspension of sodium hydride (6 mmol) in dehydrated DMSO (3 mL). The mixture was stirred at the same temperature for 30 min, then cooled to 0 °C and a solution of corresponding enone **5** (6 mmol) in dehydrated DMSO (10 mL) was slowly added. The reaction mixture was stirred at room temperature for 3 h. The reaction was quenched by water, then, extracted three times with Et_2O. The combined organic layer was washed three times with water, and dried over Na_2SO_4. The solvent was removed under reduced pressure. The residue was purified by silica-gel flash column chromatography using *n*-hexane/EtOAc = 6/1 to afford the *trans*-cyclopropylketones **1a-1g**. The formation of **1d** was confirmed according to the literature.[3]



1a-1g 2

2a: R^1 = *i*-Pr, R^2 = *p*-MeO **2e**: R^1 = *t*-Bu, R^2 = *p*-MeO
2b: R^1 = *i*-Pr, R^2 = *p*-MeS **2f**: R^1 = *t*-Bu, R^2 = *m*-MeO
2c: R^1 = *i*-Pr, R^2 = *p*-NMe_2 **2g**: R^1 = *t*-Bu, R^2 = *p*-Br
2d: R^1 = *i*-Pr, R^2 = H

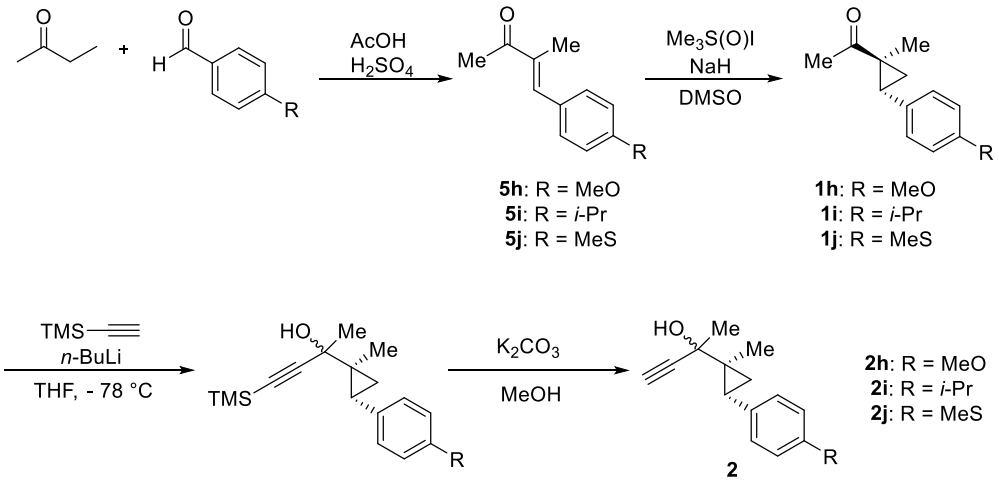


α-**2** (lower polarity) β-**2** (higher polarity)

Trimethylsilylacetylene (0.71 mL, 5.50 mmol) was dissolved in dehydrated THF (20 mL), and the solution was cooled to -78 °C. To the solution, *n*-BuLi (3.6 mL, 5.50 mmol, 1.55 M in *n*-hexane) was added dropwise. After being stirred for 1 h at -78 °C, **1** (5.00 mmol) was added dropwise. The reaction mixture was then allowed to stir overnight at room temperature. The reaction was quenched by sat. aq. NH_4Cl, and the mixture was extracted three times with Et_2O and dried over Na_2SO_4. The

solvent was removed under reduced pressure and the residue was used for the next step without further purification. The residue was dissolved in MeOH (10 mL) with K_2CO_3 (0.207 g, 1.50 mmol) and the mixture was stirred for 2 h. MeOH was removed under reduced pressure, and extracted three times with Et_2O. The combined organic layer was washed with brine, and dried over Na_2SO_4. The solvent was removed under reduced pressure. The residue was purified by silica-gel flash column chromatography using *n*-hexane/EtOAc = 6/1 to afford **α-2a-2g** and **β-2a-2g**.

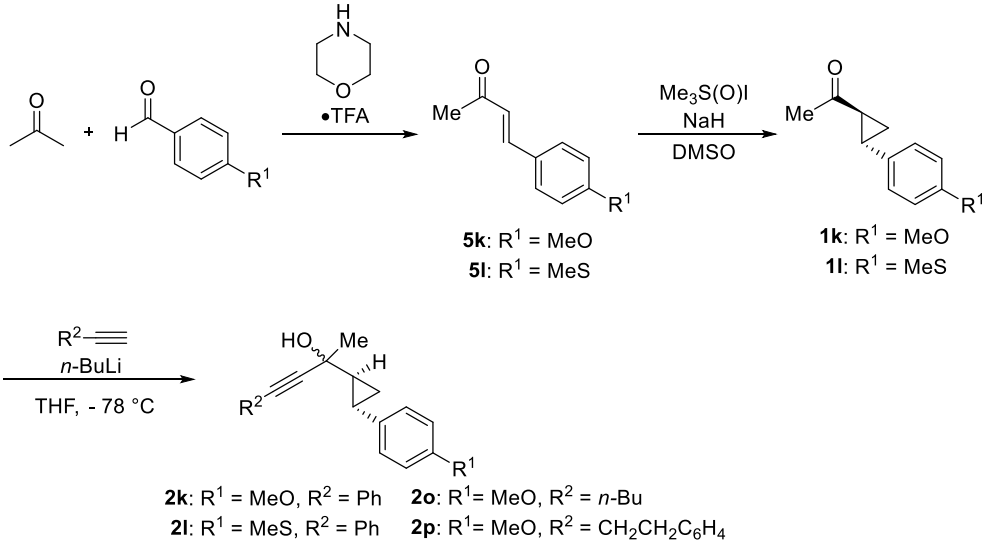
2.1.3 Procedure for the preparation of propargyl alcohols **2h**, **2i**, **2j**



Enones **5h-5j** were synthesized according to the literature.[4] Benzaldehyde derivative (20.0 mmol) was added to the solution of 2-butanone (40.0 mmol) in AcOH (16 mL). The solution was stirred in ice bath, and concentrated H_2SO_4 (1.92 g, 19.6 mmol) was slowly added. The reaction mixture was then allowed to stir at room temperature overnight. The reaction was quenched by pouring reaction solution into water (100 mL), and neutralized with 25wt% aq. NaOH (ca. 30 mL), then, extracted three times with EtOAc. The combined organic layer was washed with sat. aq. $NaHCO_3$, and dried over Na_2SO_4. The solvent was removed under reduced pressure. The residue was purified by silica-gel flash column chromatography using *n*-hexane/EtOAc = 6/1 to obtain the enone **5h-5j**.

The procedure for the preparation of **1h-1j** from **5h-5j** is similar to the above procedure **2.1.2**. Propargyl alcohols **2h-2j** were obtained as mixture of diastereomers.

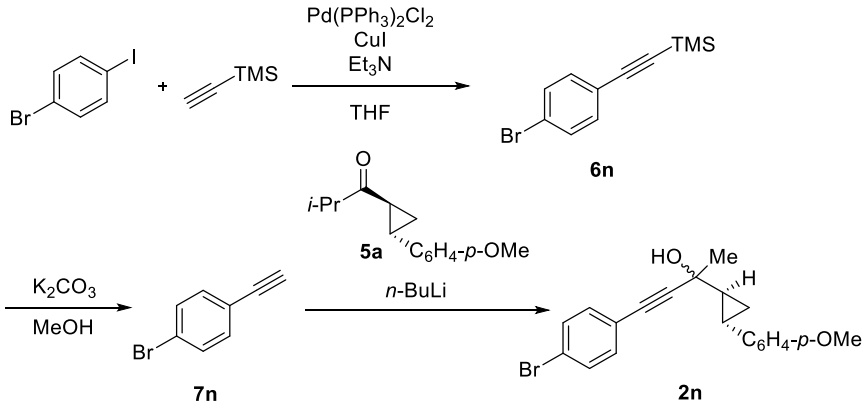
2.1.4 Procedure for the preparation of propargyl alcohols **2k**, **2l**, **2o**, **2q**



Enones **5k** and **5l** were synthesized according to the literature.[5] Morpholine·TFA (1.20 mmol) was added to a solution of corresponding benzaldehyde derivative (12.0 mmol) in acetone (30 mL). The solution was refluxed for 3 days. The reaction was quenched by sat. aq. $NaHCO_3$, and extracted three times with EtOAc. The combined organic layer was dried over Na_2SO_4, and the solvent was removed under a reduced pressure. The residue was purified by silica-gel flash column chromatography to afford the desired enones **5k** and **5l**.

The procedure for the preparation of **2k**, **2l**, **2o** and **2p** from **5k** and **5l** are similar to the above procedure **2.1.2**. The formation of **1k** was confirmed according to the literature.[6]

2.1.5 Procedure for the preparation of propargyl alcohol **2n**

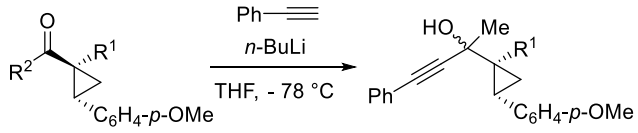


The ethynylbenzene derivative **7n** was synthesized by the following procedure. To the solution of $Pd(PPh_3)_2Cl_2$ (526 mg, 0.750 mmol, 5 mol%), CuI(I) (85.5 mg, 0.450 mmol, 3 mol%) and 4-iodobenzene (4.24 g, 15.0 mmol) in THF (25 mL), Triethylamine (4.16 mL, 30.0 mmol, 2 equiv)

was added dropwise. The reaction mixture was stirred in ice bath, and a solution of trimethylsilylacetylene (2.08 mL, 15.0 mmol, 1.0 equiv) in THF (13 mL) was slowly added and the mixture was stirred at room temperature for 12 h. Then, the reaction mixture was filtered through a Celite® pad, and the filtrate was evaporated under reduced pressure. After the solvent was removed, the residue was purified by silica-gel flash column chromatography using *n*-hexane to obtain the compounds **6n** (3.32 g, 13.1 mmol, 87%).

The compound **6n** was dissolved in MeOH (70 mL) with K_2CO_3 (3.62 g, 2.62mmol, 2 equiv) and stirred for 2 h. The solvent was removed under reduced pressure, and extracted three times with Et_2O. The combined organic layer was washed with brine, and dried over Na_2SO_4. The solvent was removed under reduced pressure. The residue was purified by silica-gel flash column chromatography using *n*-hexane to obtain the desired ethynylbenzene **7n** (775 mg, 4.28 mmol, 33%). The procedure for the preparation of **2n** using **7n** is similar to the above procedure **2.1.2**.

2.1.6 Procedure for the preparation of propargyl alcohols **2m**, **2q**

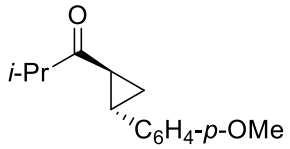


1e: R^1 = H, R^2 = *t*-Bu
1h: R^1 = Me, R^2 = Me

2m: R^1 = H, R^2 = *t*-Bu
2q: R^1 = Me, R^2 = Me

Ethynylbenzene (5.50 mmol) was dissolved in dehydrated THF (20 mL), and the solution was cooled to -78 ºC. To the solution, *n*-BuLi (5.50 mmol, 1.55 M in n-hexane) was added dropwise. After being stirred for 1 h at -78 ºC, **1e** or **1h** (5.00 mmol) was added dropwise. The reaction mixture was then allowed to stir at room temperature overnight. The reaction was quenched by sat. aq. NH_4Cl, and extracted three times with EtOAc and dried over Na_2SO_4. The solvent was removed under reduced pressure. The residue was purified by silica-gel flash column chromatography or PTLC to obtain the desired propargyl alcohol **2m** or **2q**. In particular, propargyl alcohol **2q** was obtained as mixture of diastereomers.

2.2 Characterization data of cyclopropyl ketones



1-[(1*R, 2*R**)-2-(4-methoxyphenyl)cyclopropyl]-2-methyl-1-propanone**

(1a)

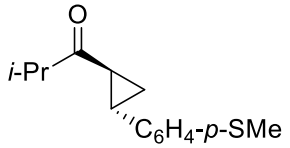
Purified by silica-gel flash column chromatography (5.35 mmol scale. eluent: *n*-hexane/EtOAc = 6/1, 372 mg, 32%); R_f = 0.54 (*n*-hexane/EtOAc = 3/1). Yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.14 (d, *J* = 6.8 Hz, 3 H), 1.16 (d, *J* = 6.8 Hz, 3 H), 1.28-1.33 (m, 1 H), 1.58-1.63 (m, 1 H), 2.12-2.17 (m, 1 H), 2.39-2.44 (m, 1 H), 2.76 (sept, *J* = 6.8 Hz, 1 H), 3.76 (s, 3 H), 6.81 (d, *J* = 8.8 Hz, 2 H), 7.02 (d, *J* = 8.8 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 17.9, 17.9, 18.2, 28.3, 30.3, 41.5, 55.1, 113.7, 127.1, 132.2, 158.1, 212.3;

IR (KBr): 2969, 1696, 1613, 1517, 1466, 1440, 1396, 1250, 1179, 1062, 1038, 821 cm^{-1};

HRMS (ESI): $[M+K]^+$ calcd for $C_{14}H_{18}KO_2^+$, 257.0938; found, m/z 257.0936.



1-[(1*R, 2*R**)-2-(4-methylthiophenyl)cyclopropyl]-2-methyl-1-propanone**

(1b)

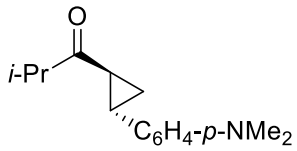
Purified by silica-gel flash column chromatography (2.33 mmol scale. eluent: *n*-hexane/EtOAc = 6/1, 87.9 mg, 16%); R_f = 0.37 (*n*-hexane/EtOAc = 3/1). Yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.14 (d, *J* = 6.8 Hz, 3 H), 1.15 (d, *J* = 6.8 Hz, 3 H), 1.31-1.35 (m, 1 H), 1.61-1.66 (m, 1 H), 2.16-2.21 (m, 1 H), 2.39-2.44 (m, 1 H), 2.46 (s, 3 H), 2.77 (sept, *J* = 6.8 Hz, 1 H), 7.02 (d, *J* = 8.0 Hz, 2 H), 7.18 (d, *J* = 8.0 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 16.1, 17.9, 18.0, 18.4, 28.5, 30.5, 41.7, 126.6, 126.9, 136.2, 137.4, 212.3;

IR (KBr): 2970, 1695, 1499, 1466, 1439, 1392, 1177, 1062, 1041, 810 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{14}H_{19}OS^+$, 235.1151; found, m/z 235.1151.



1-[(1*R, 2*R**)-2-(4-dimethylaminophenyl)cyclopropyl]-2-methyl-1-propanone**

(1c)

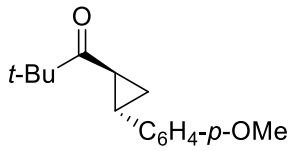
Purified by silica-gel flash column chromatography (2.36 mmol scale. eluent: *n*-hexane/EtOAc = 5/1, 172 mg, 31%); R_f = 0.54 (*n*-hexane/EtOAc = 3/1). Yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ =1.14 (d, *J* = 6.8 Hz, 3 H), 1.14 (d, *J* = 6.8 Hz, 3 H), 1.28-1.33 (m, 1 H), 1.59-1.62 (m, 1 H), 2.10-2.15 (m, 1 H), 2.75 (sept, *J* = 6.8 Hz, 1 H), 2.90 (s, 6 H), 6.66 (d, *J* = 8.8 Hz, 2 H), 6.99 (s, *J* = 8,8 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 17.9, 18.0, 18.0, 28.8, 30.4, 40.5, 40.6, 41.6, 112.6, 126.9, 128.0, 149.3, 212.7;

IR (KBr): 2969, 1692, 1616, 1525, 1466, 1439, 1395, 1347, 1061, 810 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{15}H_{22}NO^+$, 232.1696; found, m/z 232.1701.



1-[(1*R, 2*R**)-2-(4-methoxyphenyl)cyclopropyl]-2,2-dimethyl-1-Propanone**

(1e)

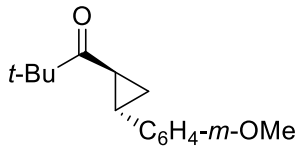
Purified by silica-gel flash column chromatography (3.29 mmol scale. eluent: *n*-hexane/EtOAc = 12/1, 726 mg, 95%); R_f = 0.60 (*n*-hexane/EtOAc = 3/1). Colorless solid; mp 61-62 °C;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.20 (s, 9 H), 1.28-1.33 (m, 1 H), 1.57-1.62 (m, 1 H), 2.27-2.31 (m, 1 H), 2.35-2.40 (m, 1 H), 3.79 (s, 3 H), 6.83 (d, *J* = 8.8 Hz, 2 H), 7.04 (d, *J* = 8.8 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 18.2, 26.2, 27.6, 28.7, 44.0, 55.3, 113.9, 127.4, 132.6, 158.3, 213.6;

IR (KBr): 2962, 1689, 1516, 1481, 1463, 1391, 1294, 1251, 1175, 1075, 1034, 814 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{15}H_{21}O_2^+$, 233.1536; found, m/z 233.1541.



1-[(1*R, 2*R**)-2-(3-methoxyphenyl)cyclopropyl]-2,2-dimethyl-1-propanone**

(1f)

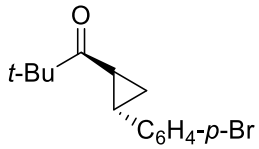
Purified by silica-gel flash column chromatography (5.86 mmol scale. eluent: *n*-hexane/EtOAc = 19/1, 1.21 g, 89%); R_f = 0.61 (*n*-hexane/EtOAc = 3/1). Colorless oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ =1.20 (s, 9 H), 1.32-1.36 (m, 1 H), 1.59-1.63 (m, 1 H), 2.35-2.40 (m, 2 H), 3.80 (s, 3 H), 6.65-6.66 (m, 1 H), 6.70 (d, *J* = 8.0 Hz, 1 H), 6.74-6.77 (m, 1 H), 7.20 (dd, *J* = 8.0, 8.0 Hz, 1 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 18.5, 26.2, 27.7, 29.1, 44.1, 55.2, 111.4, 112.3, 118.6, 129.5, 142.3, 159.7, 213.5;

IR (KBr): 2963, 1692, 1584, 1465, 1396, 1291, 1260, 1091, 1044, 803 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{15}H_{21}O_2^+$, 233.1536; found, m/z 233.1534.



1-[(1*R, 2*R**)-2-(4-bromophenyl)cyclopropyl]-2,2-dimethyl-1-propanone**

(1g)

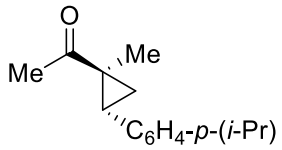
Purified by silica-gel flash column chromatography (20.4 mmol scale. eluent: *n*-hexane/EtOAc = 6/1, 3.38 g, 59%); R_f = 0.63 (*n*-hexane/EtOAc = 3/1). Colorless solid; mp 54-55 ºC;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.19 (s, 9 H), 1.30-1.34 (m, 1 H), 1.60-1.65 (m, 1 H), 2.30-2.39 (m, 2 H), 6.98 (d, *J* = 8.0 Hz, 2 H), 7.40 (d, *J* = 8.0 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 18.4 26.1, 27.8, 28.4, 44.0, 120.1, 127.9, 131.5, 139.6, 213.2;

IR (KBr): 2975, 1685, 1490, 1389, 1336, 1175, 1081, 1008, 806, 692 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{14}H_{18}BrO^+$, 281.0536; found, m/z 281.0534.



1-((1*R,2*R**)-2-(4-isopropylphenyl)-1-methylcyclopropyl)ethan-1-one**

(1i)

Purified by silica-gel flash column chromatography (11.5 mmol scale. eluent: *n*-hexane/EtOAc = 6/1,

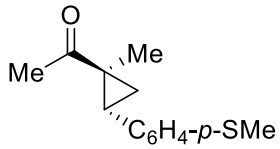
155 mg, 6%); R_f = 0.50 (*n*-hexane/EtOAc = 3/1). Yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ =1.07 (s, 3 H), 1.24 (d, J = 7.2 Hz, 6 H), 1.17-2.00 (m, 1 H), 1.72-1.75 (m, 1 H), 2.23 (s, 3 H), 2.48 (s, 3 H), 2.72 (dd, *J* = 9.2, 7.2 Hz, 1 H), 2.89 (sept, *J* = 7.2 Hz, 1 H), 7.08 (d, *J* = 8.0 Hz, 2 H), 7.20 (d, *J* = 8.0 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 15.3, 21.4, 23.9, 26.9, 33.3, 33.5, 33.7, 126.2, 129.1, 134.1, 147.3, 209.7;

IR (KBr): 2961, 1688, 1515, 1460, 1374, 1354, 1269, 1144, 954, 839 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{15}H_{21}O^+$, 217.1587; found, m/z 217.1581.



1-((1*R,2*R**)-1-methyl-2-(4-(methylthio)phenyl)cyclopropyl)ethan-1-one**

(1j)

Purified by silica-gel flash column chromatography (13.2 mmol scale. eluent: *n*-hexane/EtOAc = 6/1, 150 mg, 5%); R_f = 0.31 (*n*-hexane/EtOAc = 3/1). Yellow oil;

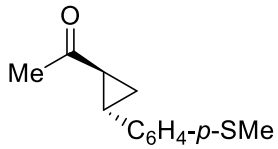
^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.06 (s, 3 H), 1.16-1.219 (m, 1 H), 1.72-1.75 (m, 1 H), 2.32 (s, 3 H), 2.48 (s, 3 H), 2.70-2.74 (m, 1 H), 2.89 (sept, *J* = 7.2 Hz, 1 H), 7.08 (d, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 15.3, 15.9, 21.4, 27.0, 33.3, 33.3, 126.4, 129.7, 133.8, 136.7, 209.5;

IR (KBr): 2964, 1686, 1592, 1496, 1436, 1373, 1278, 1145, 1094, 955, 828 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{13}H_{17}OS^+$, 221.0995;

found, m/z 221.1002.



1-((1*R,2*S**)-2-(4-(methylthio)phenyl)cyclopropyl)ethan-1-one**

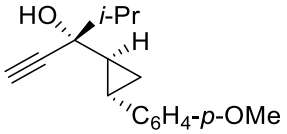
(1l)

Purified by silica-gel flash column chromatography (5.51 mmol scale. eluent: *n*-hexane/EtOAc = 4/1, 378 mg, 33%); R_f = 0.37 (*n*-hexane/EtOAc = 3/1). Pale yellow solid; mp 60-62 °C;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.33-1.38 (m, 1 H), 1.64-1.69 (m, 1 H), 2.15-2.20 (m, 1 H), 2.30 (s, 3 H), 2.45-2.51 (m, 1 H), 2.47 (s, 3 H), 7.01 (d, *J* = 8.4 Hz, 2 H), 7.18 (d, *J* = 8.4 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 16.2, 19.0, 28.6, 30.9, 32.8, 126.5, 127.0, 136.4, 137.3, 206.8;
IR (KBr): 3007, 1693, 1499, 1424, 1392, 1359, 1181, 966, 904, 811cm^{-1};
HRMS (ESI): $[M+H]^+$ calcd for $C_{12}H_{15}OS^+$, 207.0838; found, m/z 207.0833.

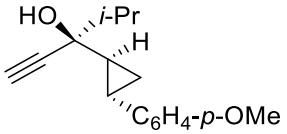
2.3 Characterization data of propargyl alcohols



(3*R)-3-((2*S**)-2-(4-methoxyphenyl)cyclopropyl)-4-methylpent-1-yn-3-ol**

(α-2a)

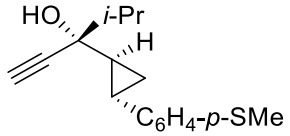
Purified by silica-gel flash column chromatography (9.91 mmol scale. eluent: *n*-hexane/EtOAc = 5/1, 827 mg, 34%); R_f = 0.34 (*n*-hexane/EtOAc = 3/1). Colorless oil;
^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.92-0.97 (m, 1 H), 1.08 (dd, *J* = 11.6, 7.2 Hz, 6 H), 1.16-1.21 (m, 1 H), 1.33-1.38 (m, 1 H), 1.97-2.02 (m, 2 H), 2.08-2.13 (m, 1 H), 2.44 (s, 1 H), 3.78 (s, 3 H), 6.81 (d, *J* = 9.2 Hz, 2 H), 7.03 (d, *J* = 9.2 Hz, 2 H);
^{13}C NMR (100 MHz, $CDCl_3$) δ = 13.6, 17.5, 17.9, 18.0, 29.9, 39.0, 55.3, 73.7, 75.9, 83.1, 113.8, 127.2, 134.2, 157.7;
IR (KBr): 3477, 3290, 2964, 1613, 1516, 1467, 1385, 1366, 1247, 1178, 1036, 825, 652 cm^{-1};
HRMS (ESI): $[M+H-H_2O]^+$ calcd for $C_{16}H_{19}O^+$, 227.1430;
found, m/z 227.1436.



(3*S)-3-((2*S**)-2-(4-methoxyphenyl)cyclopropyl)-4-methylpent-1-yn-3-ol**

(β-2a)

Purified by silica-gel flash column chromatography (9.91 mmol scale. eluent: *n*-hexane/EtOAc = 5/1, 735 mg, 30%); R_f = 0.26 (*n*-hexane/EtOAc = 3/1). Yellow oil;
^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.81-0.86 (m, 1 H), 1.01 (d, *J* = 6.8 Hz, 3 H), 1.09 (d, *J* = 6.8 Hz, 3 H), 1.16-1.20 (m, 1 H), 1.27-1.32 (m, 1 H), 2.00 (sept, *J* = 6.8 Hz, 1 H), 2.03-2.08 (m, 1 H), 2.44 (s, 1 H), 3.75 (s, 3 H), 6.81 (d, *J* = 8.8 Hz, 2 H), 7.00 (d, *J* = 8.8 Hz, 2 H);
^{13}C NMR (100 MHz, $CDCl_3$) δ = 10.5, 17.5, 17.8, 21.0, 30.0, 38.9, 55.1, 74.0, 76.2, 82.6, 113.7, 126.8, 133.8, 157.6;
IR (KBr): 3469, 3295, 2965, 1613, 1516, 1464, 1386, 1366, 1247, 1178, 1036, 826, 652 cm^{-1};
HRMS (ESI): $[M+H-H_2O]^+$ calcd for $C_{16}H_{19}O^+$, 227.1430; found, m/z 227.1429.



(3*R)-4-methyl-3-((2*S**)-2-(4-(methylthio)phenyl)cyclopropyl)pent-1-yn-3-ol**

(α-2b)

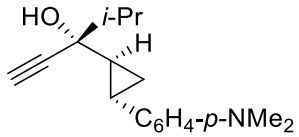
Purified by silica-gel flash column chromatography (2.20 mmol scale. eluent: *n*-hexane/EtOAc = 7/1, 164 mg, 29%); R_f = 0.43 (*n*-hexane/EtOAc = 3/1). Colorless oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.97-1.01 (m, 1 H), 1.08 (dd, *J* = 12.0, 6.8 Hz, 6 H), 1.21-1.26 (m, 1 H), 1.37-1.42 (m, 1 H), 1.99 (sept, *J* = 6.8 Hz, 1 H), 2.09-2.14 (m, 1 H), 2.48 (s, 3 H), 7.03 (d, *J* = 8.0 Hz, 2 H), 7.18 (d, *J* = 8.0 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 13.9, 16.4, 17.5, 17.8, 18.4, 30.3, 39.0, 73.7. 75.7, 83.1, 126.6, 127.2, 135.1, 139.5;

IR (KBr): 3457, 3289, 2968, 1498, 1385, 1094, 1015, 814, 650cm^{-1};

HRMS (ESI): $[M+H-H_2O]^+$ calcd for $C_{16}H_{19}S^+$, 243.1202; found, m/z 243.1203.



(3*R)-3-((2*S**)-2-(4-(dimethylamino)phenyl)cyclopropyl)-4-methylpent-1-yn-3-ol**

(α-2c)

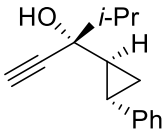
Purified by silica-gel flash column chromatography (0.74 mmol scale. eluent: *n*-hexane/EtOAc = 5/1, 18.1 mg, 9%); R_f = 0.36 (*n*-hexane/EtOAc = 3/1). Yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.90-0.95 (m, 1 H), 1.08 (dd, *J* = 12.4, 6.8 Hz, 6 H), 1.13-1.17 (m, 1 H), 1.32-1.36 (m, 1 H), 1.98 (sept, *J* = 6.8 Hz, 1 H), 2.04-2.11 (m, 2 H), 2.43 (s, 1 H), 2.89 (s, 6 H), 6.68 (d, *J* = 8.8 Hz, 2 H), 7.01 (d, *J* = 8.8 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 13.4, 17.5, 17.9, 29.5, 39.0, 40.9, 73.6, 75.9, 83.3, 113.1, 126.9, 130.2, 149.0;

IR (KBr): 3454, 3287, 2964, 1616, 1523, 1471, 1344, 1163, 1019, 815, 650cm^{-1};

HRMS (ESI): $[M+H-H_2O]^+$ calcd for $C_{17}H_{22}N^+$, 240.1747; found, m/z 240.1753.



(3*R)-4-methyl-3-((2*S**)-2-phenylcyclopropyl)pent-1-yn-3-ol**

(α-2d)

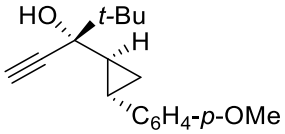
Purified by silica-gel flash column chromatography (3.00 mmol scale. eluent: *n*-hexane/EtOAc = 10/1, 141 mg, 22%); R_f = 0.51 (*n*-hexane/EtOAc = 3/1). Colorless oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.00-1.05 (m, 1 H), 1.08 (dd, J = 12.0, 6.8 Hz, 6 H), 1.22-1.27 (m, 1 H), 1.41-1.46 (m 1 H), 1.69-2.03 (m, 1 H), 2.13-2.18 (m, 1 H), 2.46 (s, 1 H), 7.10 (d, *J* = 7.2 Hz, 2 H), 7.15 (t, *J* = 7.6 Hz, 1 H), 7.26 (dd, *J* = 7.6, 7.2 Hz, 2 H), ;

^{13}C NMR (100 MHz, $CDCl_3$) δ = 14.0, 17.5, 17.9, 18.8, 30.4, 39.1, 73.8, 75.8, 83.1, 125.7, 126.0, 128.3, 142.3;

IR (KBr): 3455, 3300, 2966, 1604, 1499, 1462, 1385, 1087, 1018, 754, 698, cm^{-1};

HRMS (ESI): $[M+H-H_2O]^+$ calcd for $C_{15}H_{17}^+$, 197.1325; found, m/z 197.1324.



(3*S)-3-((2*S**)-2-(4-methoxyphenyl)cyclopropyl)-4,4-dimethylpent-1-yn-3-ol**

(α-2e)

Purified by silica-gel flash column chromatography (5.00 mmol scale. eluent: *n*-hexane/EtOAc = 6/1, 659 mg, 57%); R_f = 0.53 (*n*-hexane/EtOAc = 3/1). Colorless solid; mp 50-52 ºC;

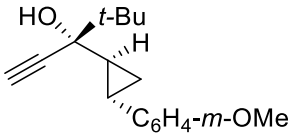
^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.94-0.98 (m, 1 H), 1.10 (s, 9 H), 1.16-1.21 (m, 1 H), 1.41-1.46 (m, 1 H), 1.89 (s, 1 H), 2.11-2.16 (m, 1 H), 2.45 (s, 1 H), 3.78 (s, 3 H), 6.81 (d, *J* = 8.8 Hz, 2 H), 7.03 (d, *J* = 8.8 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 13.8, 18.6, 25.4, 27.4, 39.3, 55.3, 73.6, 77.9, 84.0, 113.8, 127.1, 134.3, 157.5;

IR (KBr): 3490, 3258, 2977, 1516, 1464, 1389, 1239, 1178, 1063, 822 cm^{-1};

HRMS (ESI): $[M+H-H_2O]^+$ calcd for $C_{17}H_{21}O^+$, 241.1587;

found, m/z 241.1585.



(3*S)-3-((2*S**)-2-(3-methoxyphenyl)cyclopropyl)-4,4-dimethylpent-1-yn-3-ol**

(α-2f)

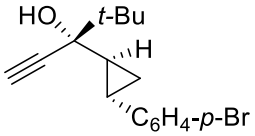
Purified by silica-gel flash column chromatography (4.00 mmol scale. eluent: *n*-hexane/EtOAc = 5/1, 548 mg, 52%); R_f = 0.51 (*n*-hexane/EtOAc = 3/1). Colorless oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.00-1.05 (m, 1 H), 1.14 (s, 9 H), 1.21-1.26 (m, 1 H), 1.48-1.52 (m, 1 H), 1.88 (s, 1 H), 2.13-2.18 (m, 1 H), 2.46 (s, 1 H), 3.80 (s, 3 H), 6.64-6.65 (m, 1 H), 6.69-6.72 (m, 2 H), 7.16-7.20 (m, 1 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 14.3, 19.4, 25.4, 28.0, 39.3, 55.1, 73.6, 77.8, 83.9, 110.8, 111.9, 118.4, 129.3, 144.2, 159.7;

IR (KBr): 3495, 3290, 2961, 1584, 1494, 1364, 1253, 1157, 1051, 995, 777, 696 cm^{-1};

HRMS (ESI): $[M+H-H_2O]^+$ calcd for $C_{17}H_{21}O^+$, 241.1587; found, m/z 241.1589.



(3*S)-3-((2*S**)-2-(4-bromophenyl)cyclopropyl)-4,4-dimethylpent-1-yn-3-ol**

(α-2g)

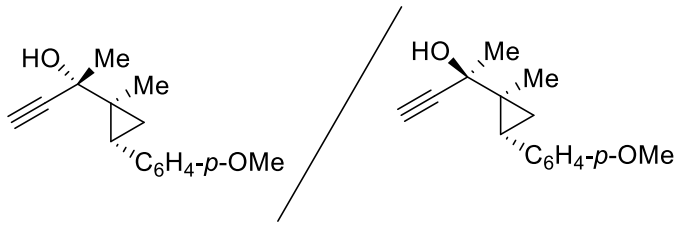
Purified by silica-gel flash column chromatography (2.00 mmol scale. eluent: *n*-hexane/EtOAc = 10/1, 275 mg, 53%); R_f = 0.43 (*n*-hexane/EtOAc = 3/1). Colorless solid; mp 57-59 °C;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.97-1.02 (m, 1 H), 1.09 (s, 9 H), 1.21-1.27 (m, 1 H), 1.43-1.48 (m, 1 H), 1.84 (s, 1 H), 2.11-2.16 (m, 1 H), 2.46 (s, 1 H), 6.96 (d, *J* = 8.4 Hz, 2 H), 7.36 (d, *J* = 8.4 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 14.1, 19.0, 25.4, 28.1, 39.3, 73.8, 77.5, 83.8, 119.2, 127.7, 131.3, 141.5;

IR (KBr): 3549, 3271, 2966, 1492, 1193, 1092, 1010, 816, 861, cm^{-1};

HRMS (ESI): $[M+H-H_2O]^+$ calcd for $C_{16}H_{18}Br^+$, 289.0586; found, m/z 289.9590.



2-((1*R,2*R**)-2-(4-methoxyphenyl)-1-methylcyclopropyl)but-3-yn-2-ol**

mixture of diastereomers-(2h)

Purified by silica-gel flash column chromatography (0.530 mmol scale. eluent: *n*-hexane/EtOAc = 5/1, 18.0 mg, 15%); R_f = 0.33 (*n*-hexane/EtOAc = 3/1). Yellow oil;

(2h)-major diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.70-0.73 (m, 1 H), 0.85 (s, 3 H), 1.30-1.33 (m, 1 H), 1.61 (s, 3 H), 1.95 (s, 1 H), 2.36-2.40 (m, 1 H), 3.80 (s, 3 H), 6.83 (d, *J* = 8.4 Hz, 2 H), 7.10 (d, *J* = 8.4 Hz, 2 H);

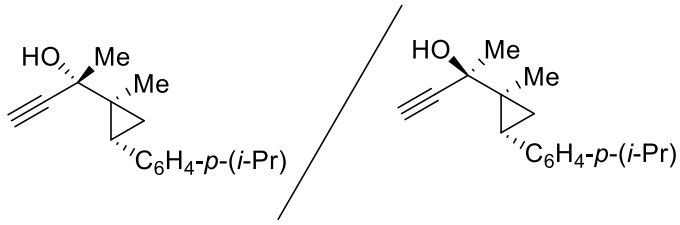
^{13}C NMR (100 MHz, $CDCl_3$) δ =14.6, 14.8, 25.0, 27.0, 29.2, 55.2, 71.5, 71.8, 86.4, 113.4, 130.1, 130.9, 157.8;

(2h)-minor diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.72-0.75 (m, 1 H), 0.88 (s, 3 H), 1.22-1.25 (m, 1 H), 1.60 (s, 3 H), 1.90 (s, 1 H), 2.43-2.46 (m, 1 H), 3.80 (s, 3 H), 6.83 (d, *J* = 8.4 Hz, 2 H), 7.10 (d, *J* = 8.4 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 14.9, 15.1, 24.5, 27.1, 29.5, 55.2, 71.1, 71.7, 86.4, 113.4, 130.1, 131.0, 157.8;

IR (KBr): 3454, 3293, 2986, 1611, 1514, 1443, 1247, 1177, 1117, 1036, 916, 836, 656 cm^{-1};

HRMS (ESI): $[M+H-H_2O]^+$ calcd for $C_{15}H_{17}O^+$, 213.1274; found, m/z 213.1275.



2-((1*R,2*R**)-2-(4-isopropylphenyl)-1-methylcyclopropyl)but-3-yn-2-ol**

mixture of diastereomers-(2i)

Purified by preparative thin layer chromatography (0.486 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 26.7 mg, 23%); R_f = 0.50 (*n*-hexane/EtOAc = 3/1). Colorless oil;

(2i)-major diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.67-0.70 (m, 1 H), 0.72-0.76 (m, 1 H), 0.80 (s, 3 H), 1.17 (d, *J* = 7.2 Hz, 6 H), 1.23-1.26 (m, 1 H), 1.60 (s, 3 H), 1.86 (s, 1 H), 2.31-2.35 (m, 1 H), 2.81 (sept, *J* = 7.2 Hz, 1 H), 7.02-7.08 (m, 4 H);

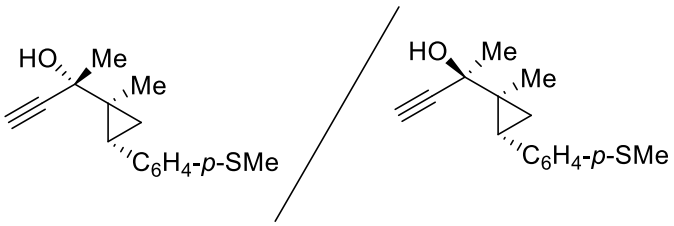
^{13}C NMR (100 MHz, $CDCl_3$) δ =14.5, 14.8, 24.0, 25.5, 27.0, 29.3, 33.6, 71.6, 71.8, 86.4, 126.0, 129.0, 126.0, 129.0, 136.1, 146.4;

(2i)-minor diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.72-0.76 (m, 1 H), 0.83 (s, 3 H), 1.16-1.18 (d, *J* = 7.2 Hz, 6 H), 1.28-1.32 (m, 1 H), 1.64 (s, 3 H), 1.81 (s, 1 H), 2.38-2.42 (m, 1 H), 2.81 (sept, *J* = 7.2 Hz, 1 H), 7.02-7.08 (m, 4 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ =14.8, 15.2, 23.9, 25.0, 27.2, 29.8, 33.7, 71.1, 71.7, 86.6, 125.9, 128.8, 125.9, 128.8, 136.2, 146.4;

IR (KBr): 3440, 3307, 3050, 2960, 1515, 1458, 1383, 1249, 1118, 1057, 915, 837, 652 cm^{-1};

HRMS (ESI): $[M+H-H_2O]^+$ calcd for $C_{17}H_{21}^+$, 225.1638; found, m/z 225.1636.



2-((1*R,2*R**)-1-methyl-2-(4-(methylthio)phenyl)cyclopropyl)but-3-yn-2-ol**

mixture of diastereomers-(2j)

Purified by preparative thin layer chromatography (0.426 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 32.9 mg, 31%); R_f = 0.30 (*n*-hexane/EtOAc = 3/1). Colorless oil;

(2j)-major diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.74-0.76 (m, 1 H), 0.85 (s, 3 H), 1.30-1.35 (m, 1 H), 2.38-2.42 (m, 1 H), 2.48 (s, 3 H), 7.10-7.12 (m, 2 H), 7.18-7.20 (m, 2 H)

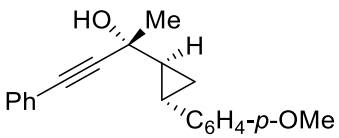
^{13}C NMR (100 MHz, $CDCl_3$) δ = 14.5, 14.8, 16.1, 25.3, 27.0, 29.5, 71.3, 71.9, 86.5, 126.6, 129.7, 135.4, 136.1;

(2j)-minor diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.76-0.79 (m, 1 H), 0.88 (s, 3 H), 1.25-1.27 (m, 1 H), 1.60 (s, 3 H), 2.47 (s, 3 H), 2.48-2.51 (m, 1 H), 7.10-7.12 (m, 2 H), 7.18-7.20 (m, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 14.8, 15.1, 16.1, 24.9, 27.0, 29.5, 71.3, 71.8, 86.3, 126.6, 129.7, 135.4, 136.0;

IR (KBr): 3440, 3292, 3073, 2985, 1496, 1442, 1370, 1117, 1014, 914, 829, 647 cm^{-1};

HRMS (ESI): $[M+H-H_2O]^+$ calcd for $C_{15}H_{17}S^+$, 229.1045; found, m/z 229.1054.



(2*R)-2-((2*S**)-2-(4-methoxyphenyl)cyclopropyl)-4-phenylbut-3-yn-2-ol**

(α-2k)

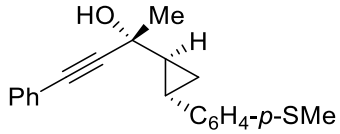
Purified by silica-gel flash column chromatography (4.48 mmol scale. eluent: *n*-hexane/EtOAc = 6/1, 211 mg, 16%); R_f = 0.54 (*n*-hexane/EtOAc = 3/1). Pale yellow solid; mp 49-51 °C;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.91-0.96 (m, 1 H), 1.16-1.21 (m, 1 H), 1.47-1.52 (m, 1 H), 1.70 (s, 3 H), 2.14 (s, 1 H), 2.20-2.25 (m, 1 H), 3.78 (s, 3 H), 6.82 (d, *J* = 8.8 Hz, 2 H), 7.06 (d, *J* = 8.8 Hz, 2 H), 7.30-7.33 (m, 3 H), 7.42-7.45 (m, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 12.8, 19.3, 29.8, 32.8, 55.3, 69.9, 84.1, 90.2, 113.8, 122.4, 127.3, 128.3, 128.4, 131.7, 134.2, 157.8;

IR (KBr): 3452, 3064, 2984, 1518, 1361, 1240, 1130, 1031, 822, 754, 693 cm^{-1};

HRMS (ESI): $[M+H-H_2O]^+$ calcd for $C_{20}H_{19}O^+$, 275.1430; found, m/z 275.1436.



(2*R)-2-((2*S**)-2-(4-(methylthio)phenyl)cyclopropyl)-4-phenylbut-3-yn-2-ol**

(α-2l)

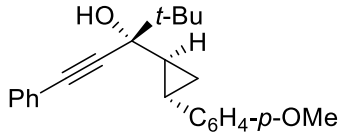
Purified by silica-gel flash column chromatography (0.838 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 89.6 mg, 37%). Yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.96-1.00 (m, 1 H), 1.20-1.25 (m, 1 H), 1.51-1.56 (m, 1 H), 1.70 (s, 3 H), 2.17 (s, 1 H), 2.20-2.25 (m, 1 H), 2.46 (s, 3 H), 7.05 (d, *J* = 8.8 Hz, 2 H), 7.18 (d, *J* = 8.8 Hz, 2 H), 7.31-7.33 (m, 3 H), 7.42-7.45 (m, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 13.1, 16.4, 19.6, 29.9, 33.2, 69.8, 84.2, 90.1, 122.3, 126.7, 127.2, 128.3, 128.5, 131.7, 135.1, 139.5;

IR (KBr): 3425, 3076, 2982, 1598, 1498, 1443, 1273, 1127, 926, 817, 757, 692 cm^{-1};

HRMS (ESI): $[M+H-H_2O]^+$ calcd for $C_{20}H_{19}S^+$, 291.1202; found, m/z 291.1198.



(3*S)-3-((2*S**)-2-(4-methoxyphenyl)cyclopropyl)-4,4-dimethyl-1-phenylpent-1-yn-3-ol**

(α-2m)

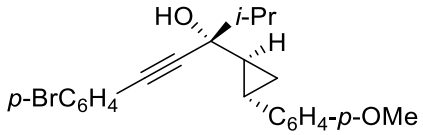
Purified by silica-gel flash column chromatography (4.00 mmol scale. eluent: *n*-hexane/EtOAc = 6/1, 535 mg, 40%); R_f = 0.47 (*n*-hexane/EtOAc = 3/1). Yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.97-1.03 (m, 1 H), 1.17 (s, 9 H), 1.25-1.28 (m, 1 H), 1.49-1.54 (m, 1 H), 1.96 (s, 1 H), 1.96-2.21 (m, 1 H), 3.79 (s, 3 H), 6.82 (d, *J* = 8.8 Hz, 2 H), 7.06 (d, *J* = 8.8 Hz, 2 H), 7.32-7.33 (m, 3 H), 7.44-7.46 (m, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 14.0, 18.7, 25.6, 27.8, 39.8, 55.2, 78.4, 85.6, 89.4, 113.8, 122.7, 127.2, 128.3, 131.7, 134.4, 157.7;

IR (KBr): 3493, 3032, 2959, 1515, 1489, 1363, 1247, 1177, 1037, 995, 828, 757, 692 cm^{-1};

HRMS (ESI): $[M+H-H_2O]^+$ calcd for $C_{23}H_{25}O^+$, 317.1900; found, m/z 317.1900.



(3*R)-1-(4-bromophenyl)-3-((2*S**)-2-(4-methoxyphenyl)cyclopropyl)-4-methylpent-1-yn-3-ol (α-2n)**

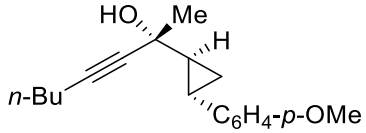
Purified by silica-gel flash column chromatography (4.00 mmol scale. eluent: *n*-hexane/EtOAc = 5/1, 641 mg, 38%); R_f = 0.48 (*n*-hexane/EtOAc = 3/1). Yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.94-1.01 (m, 1 H), 1.13 (dd, *J* = 12.4, 6.8 Hz, 6 H), 1.21-1.25 (m, 1 H), 1.40-1.45 (m, 1 H), 2.03-2.09 (m, 2 H), 2.10-2.17 (m, 1 H), 3.78 (s, 3 H), 6.81 (d, *J* = 8.8 Hz, 2 H), 7.05 (d, *J* = 8.8 Hz, 2 H), 7.29 (d, *J* = 8.8 Hz, 2 H), 7.45 (d, *J* = 8.8 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 13.6, 17.2, 18.2, 30.1, 39.4, 55.3, 76.4, 84.7, 89.7, 113.8, 121.5, 122.6, 127.2, 131.5, 133.2, 134.2, 157.8;

IR (KBr): 3466, 2963, 1612, 1515, 1392, 1247, 1177, 1037, 1011, 824, 732 cm^{-1};

HRMS (ESI): $[M+H-H_2O]^+$ calcd for $C_{22}H_{22}BrO+$, 381.0849; found, m/z 381.0851.



(2*R)-2-((2*S**)-2-(4-methoxyphenyl)cyclopropyl)oct-3-yn-2-ol (α-2o)**

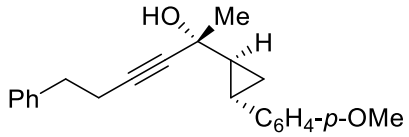
Purified by silica-gel flash column chromatography (4.40 mmol scale. eluent: *n*-hexane/EtOAc = 6/1, 114 mg, 42%); R_f = 0.29 (*n*-hexane/EtOAc = 3/1). Yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.83-0.87 (m, 1 H), 0.92 (t, *J* = 7.6 Hz, 3 H), 1.03-1.08 (m, 1 H), 1.36-1.51 (m, 5H), 2.11-2.13 (m, 1 H), 2.21 (t, *J* = 6.8 Hz, 2 H), 3.77 (s, 3 H), 6.90 (d, *J* = 8.8 Hz, 2 H), 7.03 (d, *J* = 8.8 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 12.7, 13.6, 18.2, 19.2, 21.9, 30.0, 30.7, 32.8, 55.3, 69.7, 81.2, 84.7, 113.7, 127.2, 134.4, 157.6;

IR (KBr): 3445, 3063, 2931, 1612, 1517, 1454, 1290, 1248, 1178, 1078, 1035, 938, 828, 749, 700 cm^{-1};

HRMS (ESI): $[M+H-H_2O]^+$ calcd for $C_{18}H_{23}O^+$, 255.1743; found, m/z 255.1744.



(2*R)-2-((2*S**)-2-(4-methoxyphenyl)cyclopropyl)-6-phenylhex-3-yn-2-ol**

(α-2p)

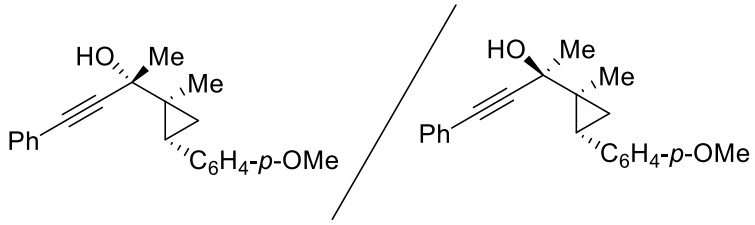
Purified by silica-gel flash column chromatography (2.29 mmol scale. eluent: *n*-hexane/EtOAc = 5/1, 248 mg, 30%); R_f = 0.37 (*n*-hexane/EtOAc = 3/1). Colorless oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.78-0.82 (m, 1 H), 0.93-0.98 (m, 1 H), 1.32-1.37 (m, 1 H), 1.55 (s, 3 H), 1.94 (s, 1 H), 1.98-2.04 (m, 1 H), 2.51 (t, *J* = 7.2 Hz, 2 H), 2.82 (t, *J* = 7.2 Hz, 2 H), 3.77 (s, 3 H), 6.80 (d, *J* = 8.8 Hz, 2 H), 7.00 (t, *J* = 8.8 Hz, 2 H), 7.22-7.25 (m, 3 H), 7.28-7.32 (m, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 12.7, 19.1, 20.7, 30.0, 32.7, 34.9, 55.3, 69.6, 82.1, 83.9, 112.7, 126.3, 127.2, 128.4, 128.5, 134.4, 140.4, 157.7;

IR (KBr): 3445, 2931, 1612, 1515, 1455, 1289, 1247, 1178, 1078, 1036, 938, 828, 700 cm^{-1};

HRMS (ESI): $[M+H-H_2O]^+$ calcd for $C_{22}H_{23}O^+$, 303.1743; found, m/z 303.1737.



2-((1*R,2*R**)-2-(4-methoxyphenyl)-1-methylcyclopropyl)-4-phenylbut-3-yn-2-ol**

mixture of diastereomers-(2p)

Purified by preparative thin layer chromatography (0.500 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 96.5 mg, 84%); R_f = 0.37 (*n*-hexane/EtOAc = 3/1). Pale yellow oil;

(2p)-major diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.73-0.77 (m, 1 H), 0.90 (s, 3 H), 1.37-1.41 (m, 1 H), 1.69 (s, 3 H), 2.20 (s, 1 H), 2.43-2.47 (m, 1 H), 3.78 (s, 3 H), 6.83 (d, *J* = 8.8 Hz, 2 H), 7.12 (d, *J* = 8.8 Hz, 2 H), 7.29-7.31 (m, 3 H), 7.42-7.44 (m, 2 H);

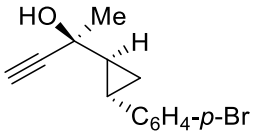
^{13}C NMR (100 MHz, $CDCl_3$) δ = 14.9, 15.2, 25.1, 27.0, 30.0, 55.2, 72.0, 83.7, 91.7, 113.4, 122.6, 128.2, 128.3, 130.1, 131.1, 131.2, 157.8;

(2p)-minor diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.74-0.78 (m, 1 H), 0.93 (s, 3 H), 1.28-1.31 (m, 1 H), 1.68 (s, 3 H), 2.16 (s, 1 H), 2.43-2.47 (m, 1 H), 3.78 (s, 3 H), 6.83 (d, *J* = 8.8 Hz, 2 H), 7.12 (d, *J* = 8.8 Hz, 2 H), 7.29-7.31 (m, 3 H), 7.42-7.44 (m, 2 H)

^{13}C NMR (100 MHz, $CDCl_3$) δ =14.9, 15.15.1, 24.7, 27.1, 29.6, 55.2, 71.7, 83.6, 91.8, 113.4, 122.6, 128.2, 128.3, 130.1, 131.2, 131.6, 157.7;

IR (KBr): 3453, 3062, 3033, 2993, 1611, 1514, 1442, 1299, 1245, 1177, 1037, 916, 831, 757, 692 cm^{-1};

HRMS (ESI): $[M+H-H_2O]^+$ calcd for $C_{21}H_{21}O$+, 289.1587; found, m/z 289.1584.



(2*S)-2-((2*S**)-2-(4-bromophenyl)cyclopropyl)but-3-yn-2-ol**

(β-2q)

Purified by silica-gel flash column chromatography (2.05 mmol scale. eluent: *n*-hexane/EtOAc = 5/1, 159 mg, 29%); R_f = 0.29 (*n*-hexane/EtOAc = 3/1). Colorless solid; mp 49-51 °C;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.89-0.93 (m, 1 H), 1.27-1.32 (m, 1 H), 1.39-1.44 (m, 1 H), 1.63 (s, 3 H), 2.02-2.05 (m, 1 H), 2.46 (s, 1 H), 6.97 (d, *J* = 8.8 Hz, 2 H), 7.37 (d, *J* = 8.8 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 12.0, 20.3, 29.8, 32.7, 69.0, 72.5, 84.8, 119.4, 127.9, 131.3, 141.1;

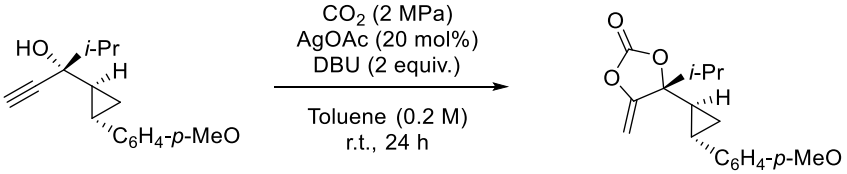
IR (KBr): 3289, 2988, 1491, 1344, 1224, 1126, 1074, 1009, 958, 919, 817, 656 cm^{-1};

HRMS (ESI): $[M+H-H_2O]^+$ calcd for $C_{13}H_{12}Br^+$, 247.0117; found, m/z 247.0119.

3. Method for decarboxylative homo-Nazarov cyclization

3.1 General procedure for decarboxylative homo-Nazarov cyclization

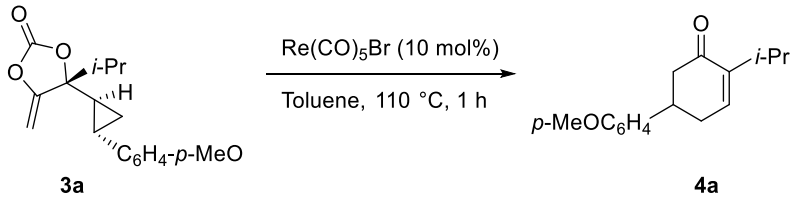
3.1.1 CO_2 incorporation on propargyl alcohols



2a **3a**

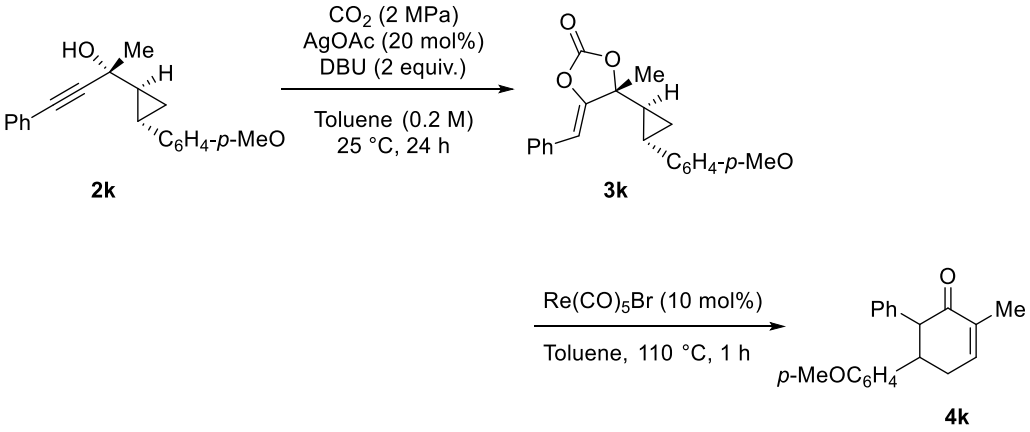
The reaction was performed using a pressure-resistant test-tube equipped with stirring bar in a 50 mL autoclave. To a mixture of AgOAc (113 mg, 0.68 mmol) and **2a** (827 mg, 3.39 mmol) in dehydrated toluene (17 mL) in the test-tube was added DBU (1.01 mL, 6.78 mmol). The test-tube containing the reaction mixture was placed in the autoclave. The autoclave was charged with CO_2 (2 MPa) and the reaction mixture was stirred at room temperature for 24 h. After CO_2 was carefully released, the reaction mixture was diluted with EtOAc (30 mL), and washed with water (30 mL), then brine (30 mL). The organic phase was dried over Na_2SO_4, and the solvent was removed under reduced pressure and dried *in vacuo*. The residue was purified by silica-gel flash column chromatography using *n*-hexane/EtOAc = 6/1 to give the desired cyclic carbonate **3a** (544 mg, 2.23 mmol, 66% yield).

3.1.2 Lewis acid-catalyzed decarboxylative homo-Nazarov cyclization



A typical procedure for the reaction of **3a** is described. A magnetic stirrer bar was placed in a Schlenk tube under argon atmosphere. $Re(CO)_5Br$ (3.0 mg, 0.0075 mmol) was added to the tube and the tube was refilled with argon. The solution of cyclic carbonate **3a** (21.6 mg, 0.0750 mmol) in dehydrated toluene (1.5 mL) was added to the Schkenk tube and the Schlenk tube was warmed up to 110 °C. After 1 h, $Re(CO)_5Br$ was removed through a short silica-gel column using EtOAc as eluent. After evaporation, the residue was purified by preparative thin layer chromatography (*n*-hexane/EtOAc = 3/1) to give cyclohexenone **4a** (16.7 mg, 0.0685 mmol, 91% yield).

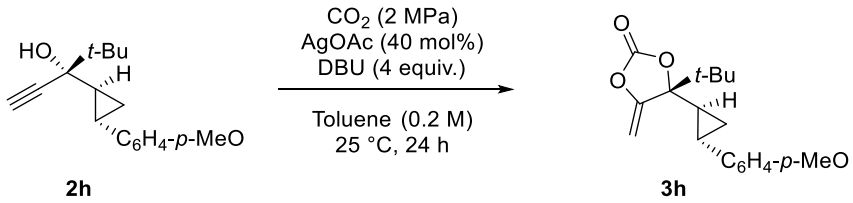
3.1.3 Decarboxylative homo-Nazarov cyclization using cyclic enol carbonates derived from internal alkynes



The reaction was performed using a pressure-resistant test-tube equipped with a stirring bar in a 30 mL autoclave. To a mixture of AgOAc (2.60 mg, 0.0150 mmol) and **2k** (21.9 mg, 0.0750 mmol) in dehydrated toluene (1.5 mL) in the test-tube was added DBU (24 μL, 0.150 mmol). The test-tube was placed in the autoclave. The autoclave was charged with CO_2 (2 MPa) and the reaction mixture was stirred at 25 °C for 4 h. After CO_2 was carefully released, the reaction mixture was diluted with EtOAc (10 mL), and washed with water (10 mL), then brine (10 mL). The organic phase was dried over Na_2SO_4, and the solvent was removed under reduced pressure and dried *in vacuo* to obtain the corresponding cyclic carbonate **3k**. The cyclic carbonate **3k** was immediately used for the next step without further purification. The procedure for the preparation of **4k** from **3k** is similar to the above procedure **3.1.3** (16.6 mg, 0.0567 mmol, 76% yield (*trans*:*cis* = 63:37)).

3.2 Procedure for preparative scale of decarboxylative homo-Nazarov cyclization

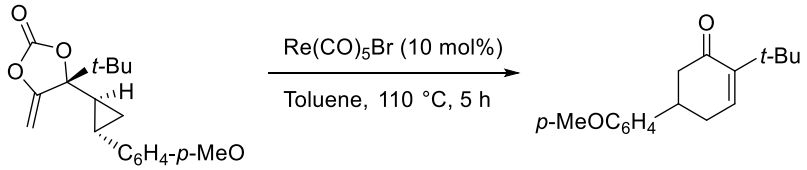
3.2.1 CO_2 incorporation on propargyl alcohol



2h **3h**

The reaction was performed using a pressure-resistant test-tube equipped with stirring bar in a 30 mL autoclave. To a mixture of AgOAc (113 mg, 0.676 mmol) and **2h** (436 mg, 1.69 mmol) in dehydrated toluene (8.4 mL) in the test-tube was added DBU (1.01 mL, 6.76 mmol). The test-tube containing the reaction mixture was placed in the autoclave. The autoclave was charged with CO_2 (2 MPa) and the reaction mixture was stirred at 25 °C for 24 h. After CO_2 was carefully released, the reaction mixture was diluted with EtOAc (15 mL), and washed with water (15 mL), then brine (15 mL). The organic phase was dried over Na_2SO_4, and the solvent was removed under reduced pressure and dried *in vacuo*. The residue was purified by silica-gel flash column chromatography using *n*-hexane/EtOAc = 5/1 to give the desired cyclic carbonate **3h** (335 mg, 1.11 mmol, 67% yield).

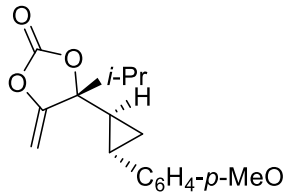
3.2.2 Lewis acid-catalyzed decarboxylative homo-Nazarov cyclization



3h **4h**

A magnetic stirrer bar was placed in a 100 mL round bottom flask under argon atmosphere. $Re(CO)_5Br$ (410 mg, 0.101 mmol) was added to the flask and the flask was refilled with argon. The solution of cyclic carbonate **3h** (302 mg, 1.01 mmol) in dehydrated toluene (20 mL) was added to the flask and the flask was warmed up to 110 °C. After 5 h, $Re(CO)_5Br$ was removed through a short silica-gel column using EtOAc as eluent. After evaporation, the residue was purified by silica-gel flash column chromatography (*n*-hexane/EtOAc = 5/1) to give cyclohexenone **4h** (256 mg, 0.993 mmol, 98% yield).

3.3 Characterization data of cyclic carbonates



(4*R)-4-isopropyl-4-((2*S**)-2-(4-methoxyphenyl)cyclopropyl)-5-methylene-1,3-dioxolan-2-one (α-3a)**

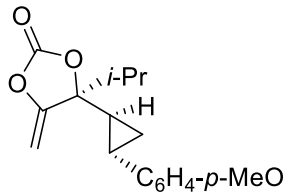
Purified by silica-gel flash column chromatography (3.39 mmol scale. eluent: *n*-hexane/EtOAc = 6/1, 544 mg, 66%); R_f = 0.46 (*n*-hexane/EtOAc = 3/1). Colorless oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.94-0.98 (m, 2 H), 1.03 (d, *J* = 6.8 Hz, 3 H), 1.07 (d, *J* = 6.8 Hz, 3 H), 13.5-13.9 (m, 1 H), 2.05-2.10 (m, 2 H), 3.78 (s, 3 H), 4.32 (d, *J* = 3.6 Hz, 1 H), 4.89 (d, *J* = 3.6 Hz, 1 H), 6.82 (d, *J* = 8.4 Hz, 2 H), 7.00 (d, *J* = 8.4 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 9.7, 16.4, 16.5, 19.5, 27.9, 37.1, 55.2, 86.7, 91.0, 113.9, 127.0, 132.5, 151.6, 155.3, 158.0;

IR (KBr): 2971, 1824, 1682, 1517, 1466, 1292, 1248, 1115, 1036, 826, 767 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{17}H_{21}O_4^+$, 289.1434; found, m/z 289.1429.



(4*S)-4-isopropyl-4-((2*S**)-2-(4-methoxyphenyl)cyclopropyl)-5-methylene-1,3-dioxolan-2-one (β-3a)**

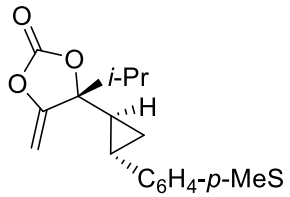
Purified by silica-gel flash column chromatography (3.00 mmol scale. eluent: *n*-hexane/EtOAc = 5/1, 418 m g, 48%); R_f = 0.46 (*n*-hexane/EtOAc = 3/1). Colorless oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.00-1.05 (m, 1 H), 1.04 (d, *J* = 6.8 Hz, 3 H), 1.09 (d, *J* = 6.8 Hz, 3 H), 1.10-1.15 (m, 1 H), 1.38-1.43 (m, 1 H), 1.87-1.90 (m, 1 H), 2.12 (sept, *J* = 6.8 Hz, 1 H), 3.75 (s, 3 H), 4.34 (d, *J* = 4.0 Hz, 2 H), 4.89 (d, *J* = 4.0 Hz, 2 H), 6.80 (d, *J* = 8.4 Hz, 2 H), 7.01 (d, *J* = 8.4 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 11.2, 16.2, 16.3, 17.6, 26.9, 36.9, 55.0, 87.0, 91.1, 113.7, 127.3, 132.3, 151.5, 154.8, 158.0;

IR (KBr): 2973, 1824, 1681, 1517, 1467, 1292, 1249, 1116, 1039, 828, 767 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{17}H_{21}O_4^+$, 289.1434; found, m/z 289.1425.



(4*R)-4-isopropyl-5-methylene-4-((2*S**)-2-(4-(methylthio)phenyl)cyclopropyl)-1,3-dioxolan-2-one**

(α-3b)

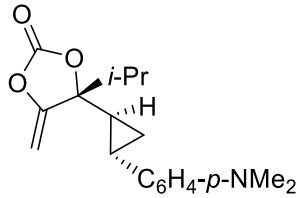
Purified by silica-gel flash column chromatography (4.83 mmol scale. eluent: *n*-hexane/EtOAc = 5/1, 691 mg, 47%); R_f = 0.49 (*n*-hexane/EtOAc = 3/1). Colorless oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.00 (t, *J* = 6.8 Hz, 2 H), 1.03 (d, *J* = 6.8 Hz, 3 H), 1.06 (d, *J* = 6.8 Hz, 3 H), 1.38-1.43 (m, 1 H), 2.05-2.11(m, 2 H), 4.34 (d, *J* = 3.6 Hz), 2.46 (s, 3 H), 4.90 (d, *J* = 3.6 Hz), 6.99 (d, *J* = 8.8 Hz, 2 H), 7.18 (d, *J* = 8.8 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 10.0, 16.1, 16.4, 16.5, 19.8, 28.3, 37.1, 86.8, 90.7, 126.3, 126.5, 127.1, 127.2, 135.9, 137.7, 151.5, 155.3;

IR (KBr): 2973, 1825, 1681, 1499, 1292, 1210, 1040, 814, 766, cm^{-1};

HRMS (ESI): $[M]^+$ calcd for $C_{17}H_{20}O_3S$, 304.1133; found, m/z 304.1135.



(4*R)-4-((2*S**)-2-(4-(dimethylamino)phenyl)cyclopropyl)-4-isopropyl-5-methylene-1,3-dioxolan-2-one**

(α-3c)

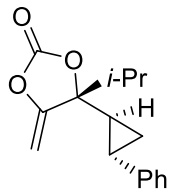
Purified by silica-gel flash column chromatography (0.534 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 105 mg, 65%); R_f = 0.54 (*n*-hexane/EtOAc = 3/1). Orange oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.91-0.95 (m, 2 H), 1.04 (d, *J* = 6.8 Hz, 3 H), 1.08 (d, *J* = 6.8 Hz, 3 H), 1.32-1.37 (m, 1 H), 2.03-2.18 (m, 2 H), 2.92 (s, 6 H), 4.32 (d, *J* = 4.0 Hz, 1 H), 4.89 (d, *J* = 4.0 Hz), 6.69 (d, *J* = 8.8 Hz, 2 H), 6.97 (d, *J* = 8.8 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 9.5, 16.5, 16.6, 19.4, 27.6, 37.1, 40.8, 86.6, 91.1, 112.9, 126.9, 151.8, 155.6;

IR (KBr): 2971, 1824, 1682, 1523, 1471, 1341, 1292, 1208, 1024, 816, 766, 731cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{18}H_{24}NO_3^+$, 302.1751; found, m/z 302.1749.



(4*R)-4-isopropyl-5-methylene-4-((2*S**)-2-phenylcyclopropyl)-1,3-dioxolan-2-one (α-3d)**

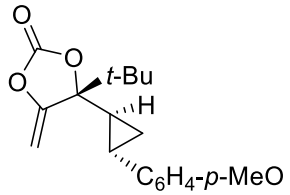
Purified by preparative thin layer chromatography (0.244 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 32.1 mg, 51%); R_f = 0.61 (*n*-hexane/EtOAc = 3/1). Colorless oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.01-1.09 (m, 8 H), 1.41-1.46 (m, 1 H), 2.06-2.18 (m, 2 H), 4.34 (d, *J* = 4.0 Hz, 1 H), 4.90 (d, *J* = 4.0 Hz, 1 H), 7.07-7.09 (m, 2 H), 7.18-7.21 (m, 1 H), 7.27-7.30 (m, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 10.1, 16.5, 16.5, 20.2, 28.4, 37.1, 86.8, 90.8, 126.0, 126.2, 128.5, 140.6, 151.6, 155.5;

IR (KBr): 3028, 2972, 1824, 1682, 1605, 1467, 1293, 1209, 1024, 854, 757, 698 cm^{-1};

HRMS (ESI): $[M]^+$ calcd for $C_{16}H_{18}O_3$, 258.1256; found, m/z 258.1265.



(4*S)-4-(tert-butyl)-4-((2*S**)-2-(4-methoxyphenyl)cyclopropyl)-5-methylene-1,3-dioxolan-2-one (α-3e)**

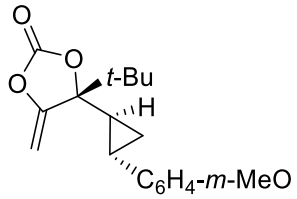
Purified by silica-gel flash column chromatography (2.17 mmol scale. eluent: *n*-hexane/EtOAc = 6/1, 293 mg, 48%); R_f = 0.60 (*n*-hexane/EtOAc = 3/1). Colorless oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.88-0.96 (m, 2 H), 1.09 (s, 1 H), 1.46-1.51 (m, 1 H), 2.10-2.15 (m, 1 H), 3.79 (s, 3 H), 4.42 (d, *J* = 4.4 Hz, 1 H), 4.95 (d, *J* = 4.4 Hz), 6.83 (d, *J* = 8.8 Hz, 2 H), 7.01 (d, *J* = 8.8 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 10.1, 20.5, 24.6, 25.1, 26.3, 39.0, 55.3, 88.7, 93.0, 114.0, 127.0, 132.6, 151.6, 155.1, 158.1;

IR (KBr): 2983, 1825, 1801, 1683, 1518, 1299, 1246, 1191, 1147, 1036, 829 cm^{-1};

HRMS (ESI): $[M]^+$ calcd for $C_{18}H_{22}O_4$, 302.1518; found, m/z 302.1509.



(4*S)-4-(tert-butyl)-4-((2*S**)-2-(3-methoxyphenyl)cyclopropyl)-5-methylene-1,3-dioxolan-2-one (α-3f)**

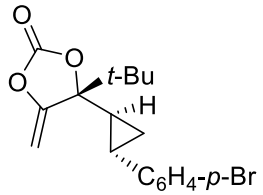
Purified by silica-gel flash column chromatography (1.40 mmol scale. eluent: *n*-hexane/EtOAc = 5/1, 231 mg, 55%); R_f = 0.51 (*n*-hexane/EtOAc = 3/1). Colorless oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.96-1.00 (m, 2 H), 1.08 (s, 9 H), 1.53-1.57 (m, 1 H), 2.13-2.18 (m, 1 H), 3.80 (s, 3 H), 4.43 (d, *J* = 4.0 Hz, 1 H), 4.96 (d, *J* = 4.0 Hz, 1 H), 6.61-6.62 (m, 1 H), 6.65-6.76 (m, 2 H), 7.19-7.23 (m, 1 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 10.5, 21.3, 24.6, 25.6, 39.0, 25.6, 55.1, 88.7, 92.8, 111.1, 112.1, 118.3, 129.5, 142.4, 151.6, 155.1, 159.8;

IR (KBr): 2963, 1832, 1979, 1605, 1469, 1294, 1186, 1036, 857, 767, 695 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{18}H_{23}O_4^+$, 303.1591; found, m/z 303.1593.



(4*S)-4-((2*S**)-2-(4-bromophenyl)cyclopropyl)-4-(tert-butyl)-5-methylene-1,3-dioxolan-2-one (α-3g)**

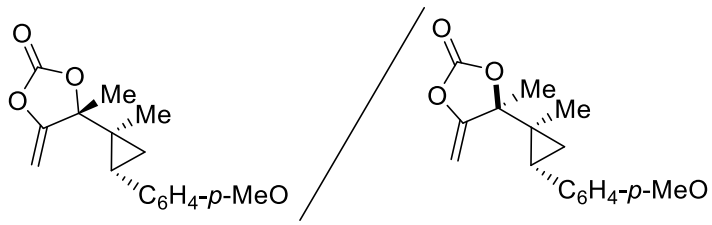
Purified by silica-gel flash column chromatography (0.145 mmol scale. eluent: *n*-hexane/EtOAc = 5/1, 27.0 mg, 53%); R_f = 0.51 (*n*-hexane/EtOAc = 3/1). Colorless oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.10 (s, 9 H), 1.13-1.18 (m, 1 H), 1.26-1.31 (m, 1 H), 1.46-1.50 (m, 1 H), 1.84-1.89 (m, 1 H), 4.36 (d, *J* = 4.0 Hz, 1 H), 4.95 (d, *J* = 4.0 Hz, 1 H), 6.95 (d, *J* = 8.4 Hz, 2 H), 7.38 (d, *J* = 8.4 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 12.6, 18.1, 24.5, 25.3, 39.1, 89.0, 92.7, 119.9, 128.0, 131.5, 139.8, 151.5, 154.7;

IR (KBr): 2974, 1828, 1678, 1492, 1368, 1294, 1187, 1036, 856, 820, 765, 654 cm^{-1};

HRMS (ESI): $[M]^+$ calcd for $C_{17}H_{19}BrO_3$, 350.0518; found, m/z 350.0517.



4-((1*S,2*R**)-2-(4-methoxyphenyl)-1-methylcyclopropyl)-4-methyl-5-methylene-1,3-dioxolan-2-one**

mixture of diastereomers-(3h)

Purified by silica-gel flash column chromatography (0.0782 mmol scale. eluent: *n*-hexane/EtOAc = 5/1, 14.3 mg, 67%); R_f = 0.40 (*n*-hexane/EtOAc = 3/1).Colorless oil;

(3h)-major diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.83-0.87 (m, 1 H), 0.86 (s, 3 H), 1.10-1.13 (m, 1 H), 1.63 (s, 3 H), 2.25-2.29 (m, 1 H), 3.79 (s, 3 H), 4.39 (d, *J* = 4.0 Hz, 1 H), 4.90 (d, *J* = 4.0 Hz, 1 H), 6.83 (d, *J* = 8.8 Hz, 2 H), 7.06 (d, *J* = 8.8 Hz, 2 H);

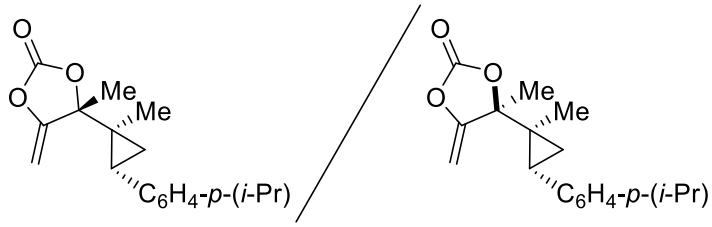
^{13}C NMR (100 MHz, $CDCl_3$) δ = 14.8, 15.1, 23.0, 23.6, 27.0, 55.2, 87.5, 89.8, 113.6, 129.1, 130.2, 151.5, 156.3, 158.2;

(3h)-minor diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.83-0.87 (m, 1 H), 0.87 (s, 3 H), 1.13-1.17 (m, 1 H), 1.65 (s, 3 H), 2.22-2.25 (m, 1 H), 3.80 (s, 3 H), 4.41 (d, *J* = 4.0 Hz, 1 H), 4.91 (d, *J* = 4.0 Hz, 1 H), 6.83 (d, *J* = 8.8 Hz, 2 H), 7.06 (d, *J* = 8.8 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 13.9, 14.7, 23.0, 24.8, 27.4, 55.2, 87.6, 89.9, 113.7, 129.1, 130.2, 151.5, 156.3, 158.2;

IR (KBr): 2989, 1812, 1684, 1612, 1515, 1457, 1379, 1245, 1021, 835, 767, 698 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{16}H_{19}O_4^+$, 275.1278; found, m/z 275.1277.



4-((1*S,2*R**)-2-(4-isopropylphenyl)-1-methylcyclopropyl)-4-methyl-5-methylene-1,3-dioxolan-2-one**

mixture of diastereomers-(3i)

Purified by preparative thin layer chromatography (0.298 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 40.3 mg, 47%); R_f = 0.57 (*n*-hexane/EtOAc = 3/1). Pale yellow amorphous solid;

(3i)-major diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.86-0.91 (m, 1 H), 0.88 (s, 3 H), 1.10-1.13 (m, 1 H), 1.23 (s, 3 H), 2.27-2.31 (m, 1 H), 2.89 (sept, 6.8 Hz, 1 H), 4.39 (d, *J* = 3.6 Hz, 1 H), 4.90 (d, *J* = 3.6 Hz, 1 H), 7.06 (d, *J* = 8.0 Hz, 2 H), 7.14 (d, *J* = 8.0 Hz, 2 H);

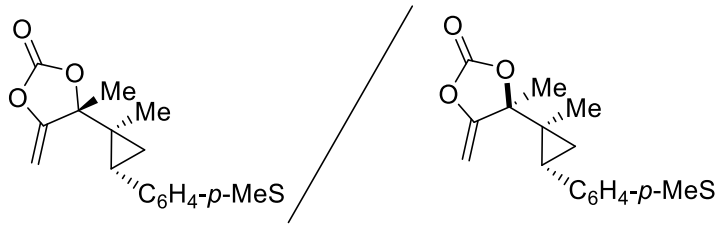
^{13}C NMR (100 MHz, $CDCl_3$) δ =14.7, 15.1, 23.0, 24.0, 24.1, 27.0, 33.7, 87.6, 89.8, 126.3, 129.1, 134.4, 147.1, 151.5, 156.3;

(3i)-minor diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.86-0.91 (m, 1 H), 0.89 (s, 3 H), 1.17-1.21 (m, 1 H), 2.24-2.28 (m, 1 H), 2.89 (sept, 6.8 Hz, 1 H), 4.42 (d, *J* = 4.0 Hz, 1 H), 4.91 (d, *J* = 4.0 Hz, 1 H), 7.06 (d, *J* = 8.0 Hz, 2 H), 7.14 (d, *J* = 8.0 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ =13.7, 14.5, 23.0, 25.2, 27.5, 33.7, 87.6, 89.8, 126.3, 129.1, 134.4, 147.1, 151.5, 156.2;

IR (KBr): 2960, 1804, 1683, 1517, 1289, 1238, 1121, 1024, 883, 838, 769, 649 cm^{-1};

HRMS (ESI): $[M]^+$ calcd for $C_{18}H_{22}O_3$, 286.1569; found, m/z 286.1565.



4-methyl-4-((1*S,2*R**)-1-methyl-2-(4-(methylthio)phenyl)cyclopropyl)-5-methylene-1,3-dioxolan-2-one**

mixture of diastereomers-(3j)

Purified by preparative thin layer chromatography (0.172 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 29.1 mg, 85%); R_f = 0.46 (*n*-hexane/EtOAc = 3/1). Colorless amorphous solid;

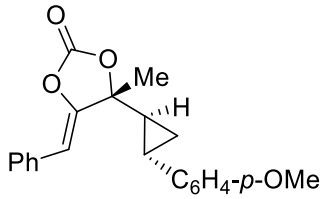
(3j)-major diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.85-0.90 (m, 1 H), 0.86 (s, 3 H), 1.12-1.16 (m, 1 H), 1.64 (s, 3 H), 2.26-2.30 (m, 1 H), 2.47 (s, 3 H), 4.40 (d, *J* = 4.0 Hz, 1 H), 4.91 (d, *J* = 4.0 Hz, 1 H), 7.06 (d, *J* = 8.4 Hz, 2 H), 7.19 (d, *J* = 8.4 Hz, 2 H);

(3j)-minor diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.85-0.90 (m, 1 H), 0.88 (s, 3 H), 1.15-1.19 (m, 1 H), 1.66 (s, 3 H), 2.24-2.30 (m, 1 H), 2.47 (s, 3 H), 4.41 (d, *J* = 4.0 Hz, 1 H), 4.92 (d, *J* = 4.0 Hz, 1 H), 7.06 (d, *J* = 8.4 Hz, 2 H), 7.19 (d, *J* = 8.4 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 13.8, 14.7, 15.1, 15.3, 15.6, 15.9, 22.9, 23.0, 23.9, 25.0, 27.2, 27.6, 87.6, 87.7, 89.6, 89.6, 126.4, 126.5, 129.5, 129.7, 134.1, 134.1, 136.4, 136.4, 151.4, 151.4, 156.0, 156.2;

IR (KBr): 2974, 1806, 1684, 1497, 1450, 1294, 1240, 1118, 1027, 766, 649 cm^{-1};

HRMS (ESI): $[M]^+$ calcd for $C_{16}H_{18}O_3S$, 290.0977; found, m/z 290.0977.



(4*R)-5-((*Z*)-benzylidene)-4-((2*S**)-2-(4-methoxyphenyl)cyclopropyl)-4-methyl-1,3-dioxolan-2-one**

(α-3k)

Purified by silica-gel flash column chromatography (2.21 mmol scale. eluent: *n*-hexane/EtOAc = 6/1, 380 mg, 51%); R_f = 0.60 (*n*-hexane/EtOAc = 3/1). Colorless solid; 79-81 °C;

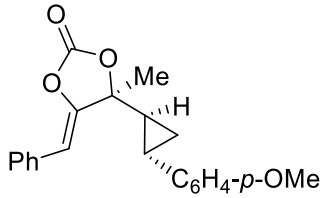
^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.98-1.03 (m, 1 H), 1.10-1.15 (m, 1 H), 1.49-1.55 (m, 1 H), 1.76 (s, 3 H), 2.04-2.09 (m, 1 H), 3.78 (s, 3 H), 5.54 (s, 1 H), 6.82 (d, *J* = 8.8 Hz, 2 H), 7.02 (d, *J* = 8.8 Hz, 2 H), 7.28 (d, *J* = 7.6 Hz, 1 H), 7.37 (t, *J* = 7.6 Hz, 2 H), 7.55 (d, *J* = 7.6 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 10.5, 19.3, 25.5, 30.1, 55.3, 87.1, 102.9, 113.9, 127.5, 127.7, 128.5, 128.7, 132.2, 132.4, 148.9, 151.3, 158.2;

IR (KBr): 3026, 2934, 1820, 1701, 1516, 1292, 1180, 1049, 1012, 691 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{21}H_{21}O_4^+$, 337.1434;

found, m/z 337.1433.



(4*S)-5-((*Z*)-benzylidene)-4-((2*S**)-2-(4-methoxyphenyl)cyclopropyl)-4-methyl-1,3-dioxolan-2-one**

(β-3k)

Purified by silica-gel flash column chromatography (0.491 mmol scale. eluent: *n*-hexane/EtOAc = 6/1, 129 mg, 78%); R_f = 0.60 (*n*-hexane/EtOAc = 3/1). Yellow oil;

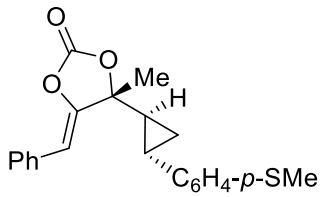
^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.02-1.13 (m, 2 H), 1.51-1.56 (m, 1 H), 1.75 (s, 3 H), 2.05-2.11 (m, 1 H), 3.77 (s, 3 H), 5.56 (s, 1 H), 6.80 (d, *J* = 8.8 Hz, 2 H), 7.02 (d, *J* = 8.8 Hz, 2 H), 7.28 (d, *J* = 7.6 Hz, 1 H), 7.37 (t, *J* = 7.6 Hz, 2 H), 7.56 (d, *J* = 7.6 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 11.2, 18.8, 25.2, 29.7, 55.3, 87.4, 103.1, 113.9, 127.7, 127.8, 128.6, 128.7, 132.2, 132.2, 148.8, 151.4, 158.2;

IR (KBr): 3061, 3003, 1824, 1700, 1612, 1452, 1248, 1040, 828, 765, 696 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{21}H_{21}O_4^+$, 337.1434;

found, m/z 337.1425.



(4*R)-5-((*Z*)-benzylidene)-4-methyl-4-((*2*S*)-2-(4-(methylthio)phenyl)cyclopropyl)-1,3-dioxolan-2-one**

(α-3l)

Purified by preparative thin layer chromatography (0.075 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 17.8 mg, 67%); R_f = 0.37 (*n*-hexane/EtOAc = 3/1). Pale yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.02-1.05 (m, 1 H), 1.14-1.19 (m, 1 H), 1.53-1.58 (m, 1 H), 1.77 (s, 3 H), 2.05-2.10 (m, 1 H), 2.46 (s, 3 H), 7.03 (d, *J* = 8.0 Hz, 2 H), 7.19 (d, *J* = 8.0 Hz, 2 H), 7.29 (d, *J* = 7.6 Hz, 1 H), 7.38 (t, *J* = 7.6 Hz, 2 H), 7.56 (d, *J* = 7.6 Hz, 2 H);

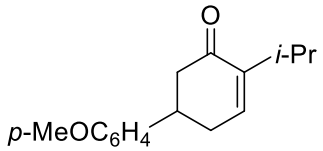
^{13}C NMR (100 MHz, $CDCl_3$) δ = 10.9, 16.2, 19.6, 25.5, 30.5, 89.8, 103.0, 126.8, 127.1, 127.8, 128.6, 128.7, 132.2, 136.1, 137.6, 148.8, 151.2;

IR (KBr): 3059, 2922, 1820, 1699, 1600, 1498, 1450, 1353, 1224, 1044, 1009, 817, 765, 694 cm^{-1};

HRMS (ESI): $[M]^+$ calcd for $C_{21}H_{20}O_3S$, 352.1133;

found, m/z 352.1135.

3.4 Characterization data of products



2-isopropyl-5-(4-methoxyphenyl)cyclohex-2-en-1-one

(4a)

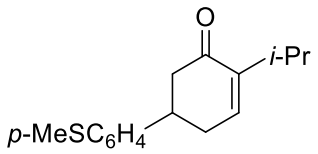
Purified by preparative thin layer chromatography (0.0753 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 16.8 mg, 91%); R_f = 0.51 (*n*-hexane/EtOAc = 3/1). Pale yellow solid; mp 55-57 ºC;

^{1}H NMR (400 MHz, $CDCl_3$): δ =1.04 (d, *J* = 6.8 Hz, 3 H), 1.05 (d, *J* = 6.8 Hz, 3 H), 2.44-2.71 (m, 4 H), 2.91 (sept, *J* = 7.2 Hz, 1 H), 3.20-3.26 (m, 1 H), 3.79 (s, 3 H), 6.72-6.73 (m, 1 H), 6.72 (d, *J* = 8.0 Hz, 2 H), 7.15 (d, *J* = 8.0 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 21.6, 22.0, 26.2, 34.1, 40.2, 45.7, 55.2, 114.0, 127.5, 135.6, 141.2, 145.4, 158.3, 198.5;

IR (KBr): 2959, 1671, 1514, 1464, 1384, 1251, 1180, 1035, 828, 531cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{16}H_{21}O_2^+$, 245.1536; found, m/z 245.1531.



2-isopropyl-5-(4-methylthiophenyl)cyclohex-2-en-1-one

(4b)

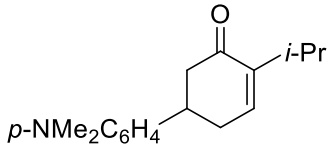
Purified by preparative thin layer chromatography (0.0750 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 14.4 mg, 74%); R_f = 0.60 (*n*-hexane/EtOAc = 3/1). Yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.04 (d, *J* = 6.8 Hz, 3 H), 1.05 (d, *J* =6.8 Hz, 3 H), 2.48 (s, 3 H), 2.51-2.70 (m, 4 H), 2.91 (sept, *J* = 6.8 Hz, 1 H), 3.21-3.26 (m, 1 H), 6.72 (m, 1 H), 7.17 (d, *J* = 8.8 Hz, 2 H), 7.24 (d, *J* = 8.8 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ =16.0, 21.7, 22.1, 26.3, 33.9, 40.5, 45.4, 127.0, 127.2, 136.7, 140.5, 141.1, 145.6, 198.3;

IR (KBr): 2959, 1672, 1495, 1383, 1241, 1095, 1002, 906, 817, 523 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{16}H_{21}OS^+$, 261.1308; found, m/z 261.1307.



2-isopropyl-5-(4-dimethylaminophenyl)cyclohex-2-en-1-one

(4c)

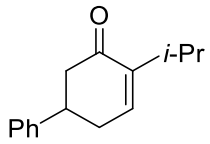
Purified by preparative thin layer chromatography (0.0760 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 16.9 mg, 86%); R_f = 0.60 (*n*-hexane/EtOAc = 3/1). Yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ =1.03 (d, *J* = 6.8 Hz, 3 H), 1.05 (d, *J* = 6.8 Hz, 3 H), 2.44-2.73 (m, 4 H), 2.93 (s, 6 H), 2.88-2.93 (m, 1 H), 3.15-3.24 (m, 1 H), 6.72 (m, 3 H), 7.12 (d, *J* = 9.2 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 21.7, 22.1, 26.2, 34.3, 40.2, 40.7, 45.9, 112.8, 127.2, 131.6, 141.5, 145.4, 149.5, 199.0;

IR (KBr): 2973, 1673, 1614, 1523, 1378, 1354, 1078, 1050, 800, 524cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{17}H_{24}NO^+$, 258.1852; found, m/z 258.1859.



2-isopropyl-5-phenylcyclohex-2-en-1-one

(4d)

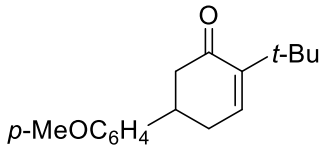
Purified by preparative thin layer chromatography (0.0694 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 13.2 mg, 71%); R_f = 0.64 (*n*-hexane/EtOAc = 3/1). Yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ =1.04 (d, *J* = 6.8 Hz, 3 H), 1.06 (d, *J* = 6.8 Hz, 3 H), 2.49-2.76 (m, 4 H), 2.92 (sept, *J* = 6.8 Hz, 1 H), 3.25-3.33 (m, 1 H), 6.73-6.75 (m, 1 H), 7.24-7.27 (m, 3 H), 7.33-7.46 (m, 2 H) ;

^{13}C NMR (100 MHz, $CDCl_3$) δ = 21.7, 22.1, 26.2, 33.9, 41.0, 45.4, 126.7, 126.8, 128.7, 141.2, 143.5, 145.6, 198.5;

IR (KBr): 3030, 2960, 1672, 1495, 1454, 1384, 1001, 907, 759, 700, 513 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{15}H_{19}O^+$, 215.1430; found, m/z 215.1437.



2-(*tert*-butyl)-5-(4-methoxyphenyl)cyclohex-2-en-1-one

(4e)

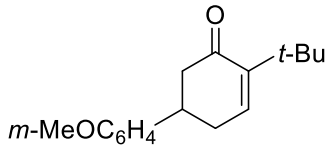
Purified by preparative thin layer chromatography (0.0754 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 17.3 mg, 89%); R_f = 0.51 (*n*-hexane/EtOAc = 3/1). Colorless solid; mp 73-74 °C;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.13 (s, 9 H), 2.40-2.59 (m, 4 H), 3.10-3.18 (m, 1 H), 3.72 (s, 3 H), 6.70-6.73 (m, 1 H), 6.79 (d, *J* = 8.8 Hz, 2 H), 7.07 (d, *J* = 8.8 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 29.3, 34.4, 34.5, 40.0, 47.3, 55.2, 114.0, 127.5, 135.7, 142.0, 146.7, 158.3, 198.8;

IR (KBr): 2962, 1672, 1514, 1366, 1250, 1181, 1033, 994, 831, 533 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{17}H_{23}O_2^+$, 259.1693; found, m/z 259.1691.



2-(*tert*-butyl)-5-(3-methoxyphenyl)cyclohex-2-en-1-one

(4f)

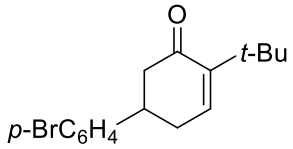
Purified by preparative thin layer chromatography (0.0747 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 18.2 mg, 94%); R_f = 0.68 (*n*-hexane/EtOAc = 3/1). Colorless oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.20 (s, 9 H), 2.47-2.57 (m, 1 H), 2.61-2.69 (m, 3 H), 3.20-3.29 (m, 1 H), 3.80 (s, 3 H), 6.78-6.83 (m, 4 H), 7.24-7.28 (m, 1 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 29.3, 34.2, 34.5, 40.8, 47.0, 55.2, 111.8, 112.7, 118.9, 129.7, 141.9, 145.2, 146.7, 159.8, 198.6;

IR (KBr): 2956, 1673, 1602, 1455, 1390, 1288, 1157, 1046, 992, 776, 699 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{17}H_{23}O_2^+$, 259.1693; found, m/z 259.1693.



5-(4-bromophenyl)-2-(*tert*-butyl)-cyclohex-2-en-1-one

(4g)

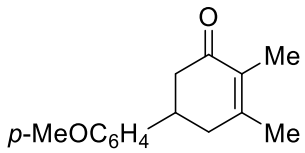
Purified by preparative thin layer chromatography (0.0336 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 9.8 mg, 95%); R_f = 0.62 (*n*-hexane/EtOAc = 3/1). Colorless solid; mp 95-96 °C;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.20 (s, 9 H), 2.44-2.68 (m, 4 H), 3.20-3.28 (m, 1 H), 6.78-6.80 (m, 1 H), 7.10 (d, *J* = 8.4 Hz, 2 H), 7.45 (d, *J* = 8.4 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 29.3, 34.1, 34.5, 40.3, 46.8, 120.5, 128.4, 131.7, 141.5, 142.4, 146.9, 198.2;

IR (KBr): 2962, 1671, 1488, 1365, 1260, 1072, 1008, 991, 914, 830 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{16}H_{20}BrO^+$, 307.0692; found, m/z 307.0695.



5-(4-methoxyphenyl)-2,3-dimethylcyclohex-2-en-1-one

(4h)

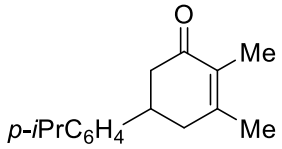
Purified by preparative thin layer chromatography (0.0521 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 10.8 mg, 90%); R_f = 0.34 (*n*-hexane/EtOAc = 3/1). Yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ =; 1.82 (s, 3 H), 1.97 (s, 3 H), 2.53-2.71 (m, 4 H), 3.19-3.25 (m, 1 H), 3.80 (s, 3 H), 6.87 (d, *J* = 8.8 Hz, 2 H), 7.15 (d, *J* = 8.8 Hz, 2 H)

^{13}C NMR (100 MHz, $CDCl_3$) δ = 10.8, 21.5, 39.4, 41.0, 44.5, 55.3, 114.0, 127.6, 130.9, 135.8, 154.2, 158.3, 198.7

IR (KBr): 2912, 1709, 1661, 1514, 1463, 1380, 1249, 1180, 1035, 828, 689 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{15}H_{19}O_2^+$, 231.1380; found, m/z 231.1381.



5-(4-isopropylphenyl)-2,3-dimethylcyclohex-2-en-1-one

(4i)

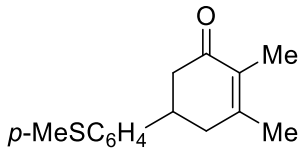
Purified by preparative thin layer chromatography (0.0443 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 9.0 mg, 71%); R_f = 0.50 (*n*-hexane/EtOAc = 3/1). Colorless oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.24 (d, *J* = 7.2 Hz, 6 H), 1.82 (s, 3 H), 1.97 (s, 3 H), 2.54-2.73 (m, 4 H), 2.90 (sept, *J* = 7.2 Hz, 1 H), 3.21-3.29 (m, 1 H), 7.15-7.21 (m, 4 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 10.8, 21.5, 24.0, 33.7, 39.7, 40.9, 44.2, 126.5, 126.7, 130.9, 140.9, 147.4, 154.2, 198.8;

IR (KBr): 2960, 1666, 1514, 1459, 1380, 1307, 1181, 1118, 825, 545 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{17}H_{23}O^+$, 243.1743; found, m/z 243.1752.



2,3-dimethyl-5-(4-methylthiophenyl)cyclohex-2-en-1-one

(4j)

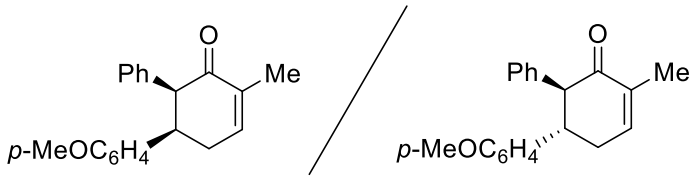
Purified by preparative thin layer chromatography (0.0489 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 8.1 mg, 67%); R_f = 0.46 (*n*-hexane/EtOAc = 3/1). Colorless oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.82 (s, 3 H), 1.97 (s, 3 H), 2.48 (s, 3 H), 2.52-2.71 (m, 4 H), 3.20-3.28 (m, 1 H), 7.15 (d, *J* = 8.4 Hz, 2 H), 7.23 (d, *J* = 8.4 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 10.8, 16.0 21.5, 39.6, 40.7, 44.1, 127.0, 127.2, 131.0, 136.7, 140.5, 154.0, 198.5;

IR (KBr): 2920, 1661, 1495, 1378, 1318, 1094, 968, 815, 520 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{15}H_{19}OS^+$, 247.1151; found, m/z 247.1155.



5-(4-methoxyphenyl)-2-methyl-6-phenylcyclohex-2-en-1-one

(4k)

Purified by preparative thin layer chromatography (0.0774 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 17.1 mg, 76%, *trans*:*cis* = 61:39).

(*cis*-4k): R_f = 0.54 (*n*-hexane/EtOAc = 3/1). Pale yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.97 (s, 3 H). 2.40-2.48 (m, 1 H), 2.68-2.76 (m, 1 H), 3.64-3.72 (m, 1 H), 3.76 (s, 3 H), 3.82 (d, *J* = 4.8 Hz, 1 H), 6.69-6.74 (m, 6 H), 7.02-7.03 (m, 1 H), 7.09-7.18 (m, 3 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 16.3, 28.0, 45.2, 55.2, 59.4, 113,4, 126.9, 128.0, 128.9, 129.2, 132.9, 135.1, 136.8, 146.1, 158.3, 199.9;

IR (KBr): 3028, 2921, 1666, 1514, 1452, 1367, 1180, 1034, 829, 747, 702 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{20}H_{21}O_2^+$, 293.1536; found, m/z 293.1541;

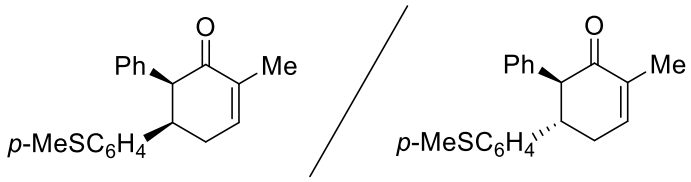
(*trans*-4k): R_f = 0.49 (*n*-hexane/EtOAc = 3/1). Pale yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.80 (s, 3 H), 2.58-2.60 (m, 2 H), 3.41-3,45 (m, 1 H), 3.61 (s, 3 H), 3.72 (d, *J* =12.4 Hz, 1 H), 6.60 (d, *J* = 6.4 Hz, 2 H), 6.75 (s, 1 H), 6.86-6.91 (m, 4 H), 7.00-7.09 (m, 3 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 16.2, 34.6, 47.4, 55.0, 60.0, 113.6, 126.4, 128.0, 128.4, 129.1, 134.5, 135.9, 138.4, 143.5, 157.9, 199.3;

IR (KBr): 3028, 2921, 1667, 1510, 1452, 1248, 1179, 1033, 827, 742, 699 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{20}H_{21}O_2^+$, 293.1536; found, m/z 293.1544;



5-(4-methoxyphenyl)-2-methyl-6-phenylcyclohex-2-en-1-one

(4l)

Purified by preparative thin layer chromatography (0.0505 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 12.3 mg, 79%, *trans*:*cis* = 65:35).

(*cis*-4l): R_f = 0.49 (*n*-hexane/EtOAc = 3/1). Yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.97 (s, 3 H), 2.45-2.48 (m, 4 H), 2.69-2.78 (m, 1 H), 3.65-3.70 (m, 1 H), 3.83 (d, *J* = 5.2 Hz, 1 H), 6.70-6.74 (m, 4 H), 7.02-7.08 (m, 3 H), 7.10-7.19 (m, 3 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 15.8, 16.3, 27.7, 45.5, 59.1, 126.2, 127.0, 128.1, 128.4, 129.2, 134.9, 136.7, 136.9, 137.6, 145.9, 199.6;

IR (KBr): 3028, 2923, 1667, 1495, 1453, 1095, 818, 701 cm^{-1} ;

HRMS (ESI): $[M+H]^+$ calcd for $C_{20}H_{21}OS^+$, 309.1308;

found, m/z 309.1317;

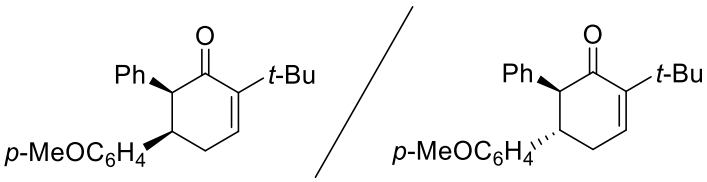
(*trans*-4l): R_f = 0.40 (*n*-hexane/EtOAc = 3/1). Pale yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.88 (s, 3 H), 2.40 (s, 3 H), 2.65-2.70 (m, 1 H), 3.52-3.59 (m, 1 H), 3.81 (d, *J* = 12.4 Hz, 1 H), 6.82-6.85 (m, 1 H), 6.94-7.19 (m, 9 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 15.7, 16.3, 34.4, 47.7, 59.6, 126.5, 126.5, 128.0, 129.1, 136.0, 136.2, 138.2, 139.3, 143.3, 199.0;

IR (KBr): 3029, 1682, 1597, 1495, 1433, 1363, 1093, 809, 743, 699 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{20}H_{21}OS^+$, 309.1308; found, m/z 309.1300;



2-(*tert*-butyl)-5-(4-methoxyphenyl)-6-phenylcyclohex-2-en-1-one

(4m)

Purified by preparative thin layer chromatography (0.0680 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 22.9 mg, quant, *trans*:*cis* = 74:26).

(*cis*-4m): R_f = 0.49 (*n*-hexane/EtOAc = 3/1). Colorless solid; mp 116-118 °C;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.29 (s, 9 H), 2.46-2.54 (m, 1 H), 2.67-2.75 (m, 1 H), 3.59-3.64 (m, 1 H), 3.76 (s, 3 H), 3.78 (d, *J* = 5.2 Hz, 1 H), 6.70-6.73 (m, 6 H), 7.01-7.04 (m, 1 H),7.09-7.18 (m, 3 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 28.2, 29.3, 34.9, 44.5, 55.2, 61.0, 113.3, 126.8, 127.9, 128.9, 129.3, 132.9, 135.2, 143.6, 147.9, 158.3, 198.9;

IR (KBr): 3061, 2957, 1663, 1512, 1454, 1280, 1182, 1040, 829, 745, 705 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{23}H_{27}O_2^+$, 335.2006; found, m/z 335.2005;

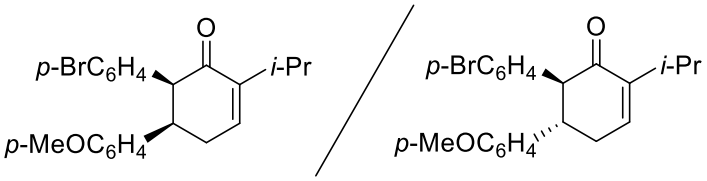
(*trans*-4m): R_f = 0.46 (*n*-hexane/EtOAc = 3/1). Colorless solid; mp 150-152 °C;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.22 (s, 9 H), 2.68-2.71 (m, 2H), 3.44-3.51 (m, 1 H), 3.71 (s, 3 H), 3.77 (d, *J* = 12.0 Hz, 1 H) 6.70 (d, *J* = 8.8 Hz, 2 H), 6.84 (t, *J* = 3.6 Hz, 1 H), 6.95-7.00 (m, 4 H), 7.08-7.11 (m, 1 H), 7.15-7.19 (m, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 29.4, 34.7, 34.7, 46.8, 55.1, 61.4, 113.6, 126.4, 128.1, 128.4, 129.2, 134.7, 138.8, 140.9, 147.3, 157.9, 198.7;

IR (KBr): 2942, 1663, 1515, 1454, 1364, 1255, 1177, 1031, 837, 742, 699 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{23}H_{27}O_2^+$, 335.2006; found, m/z 335.2010;



6-(4-bromophenyl)-2-isopropyl-5-(4-methoxyphenyl)cyclohex-2-en-1-one

(4n)

Purified by preparative thin layer chromatography (0.0632 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 20.9 mg, 83%, *trans*:*cis* = 72:28).

(*cis*-4n): R_f = 0.49 (*n*-hexane/EtOAc = 3/1). Colorless solid; mp 118-120 °C;

^{1}H NMR (400 MHz, $CDCl_3$): δ =1.13 (d, *J* = 6.8 Hz, 3 H), 1.08 (d, *J* = 6.8 Hz, 3 H), 2.52 (dt, *J* =

19.2, 2.8 Hz, 1 H), 2.67 (dd, *J* = 18.8, 11.6 Hz, 1 H), 3.03 (sept, *J* = 6.8 Hz, 1 H), 3.60-3.65 (m, 1 H), 3.77 (s, 3 H), 3.78 (d, *J* = 5.2 Hz, 1 H), 6.56 (d, *J* = 8.8 Hz, 2 H), 6.70-6.79 (m, 4 H), 6.95 (dd, *J* = 6.0, 2.4 Hz, 1 H), 7.24 (d, *J* = 8.8 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 21.8, 22.0, 26.7, 27.7, 44.6, 55.2, 59.1, 113.5, 121.0, 128.8, 130.9, 131.1, 132.4, 142.8, 146.7, 158.4, 198.2;

IR (KBr): 3012, 2961, 1665, 1611, 1515, 1383, 1254, 1179, 1028, 820 cm^{-1} ;

HRMS (ESI): $[M+H]^+$ calcd for $C_{22}H_{24}BrO_2^+$, 399.0954; found, m/z 399.0945;

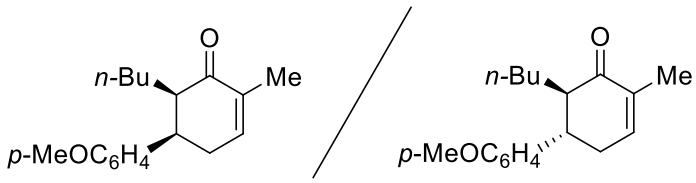
(*trans*-4n): R_f = 0.49 (*n*-hexane/EtOAc = 3/1). Yellow solid; mp 180-182 °C;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.06 (d, *J* = 6.8 Hz, 3 H), 1.08 (d, *J* = 6.8 Hz, 3 H), 2.68-2.71 (m, 2 H), 2.92 (sept, *J* = 6.8 Hz, 1 H), 3.40-3.47 (m, 1 H), 3.72 (s, 3 H), 3.74 (d, *J* = 12.8 Hz, 1 H), 6.70 (d, *J* = 8.4 Hz, 2 H), 6.77-6.79 (m, 1 H), 6.81 (d, *J* = 8.4 Hz, 2 H), 6.96 (d, *J* = 8.4 Hz, 2 H), 7.27 (d, *J* = 8.4 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 21.7, 22.1, 26.8, 34.8, 47.1, 55.1, 60.0, 113.8, 120.3, 128.4, 131.0, 131.2, 134.2, 137.6, 140.5, 145.8, 158.1, 197.9;

IR (KBr):2958, 1665, 1514, 1384, 1255, 1177, 1031, 1008, 832 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{22}H_{24}BrO_2^+$, 399.0954; found, m/z 399.0950;



6-dibutyl-5-(4-methoxyphenyl)-2-methylcyclohex-2-en-1-one

(4o)

Purified by preparative thin layer chromatography (0.0645 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 13.3 mg, 76%, *trans*:*cis* = 62:38).

(*cis*-4o): R_f = 0.49 (*n*-hexane/EtOAc = 3/1). Pale yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.76 (t, *J* = 7.2 Hz, 3 H), 1.12-1.25 (m, 6 H), 1.83(s, 3 H), 2.52-2.55 (m, 2 H), 2.70-2.78 (m, 1 H), 3.46-3.51 (m, 1 H), 3.80 (s, 3 H), 3.82 (d, *J* = 16.0 Hz, 1 H), 6.71-6.74 (m, 1 H), 6.85 (d, *J* = 8.8 Hz, 2 H), 7.11 (d, *J* = 8.8 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 13.9, 16.1, 22.6, 24.4, 29.4, 43.0, 52.5, 55.2, 113.8, 128.6, 133.6, 134.2, 143.0, 158.2, 203.1;

IR (KBr): 2956, 2931, 1669, 1610, 1514, 1464, 1370, 1251, 1182, 1037, 830 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{18}H_{25}O_2^+$, : 273.1849; found, m/z 273.1843;

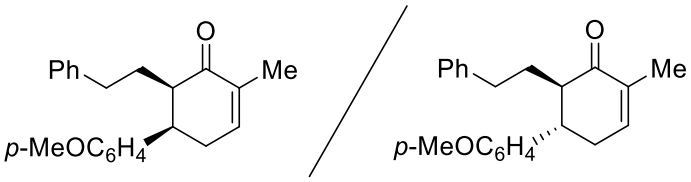
(*trans*-4o): R_f = 0.49 (*n*-hexane/EtOAc = 3/1). Pale yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.78 (t, *J* = 6.8 Hz, 3 H), 1.08-1.45 (m, 6 H), 1.80-1.82 (m, 3 H), 2.49-2.62 (m, 3 H), 3.08-3.14 (m, 1 H), 3.80 (s, 3 H), 6.65-6.68 (m, 1 H), 6.85 (d, *J* = 6.4 Hz, 2 H), 7.13 (d, *J* = 6.4 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 13.9, 16.2, 23.0, 26.9, 28.5, 34.4, 44.7, 51.8, 55.2, 113.9, 128.4, 135.5, 135.5, 142.7, 158.2, 201.3;

IR (KBr): 2955, 2929, 1668, 1612, 1513, 1464, 1368, 1251, 1035, 829 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{18}H_{25}O_2^+$, 273.1849; found, m/z 273.1854;



5-(4-methoxyphenyl)-2-methyl-6-phenethylcyclohex-2-en-1-one

(4p)

Purified by preparative thin layer chromatography (0.0719 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 20.9 mg, 91%, *trans*:*cis* = 62:38).

(*cis*-4p): R_f = 0.49 (*n*-hexane/EtOAc = 3/1). Colorless oil,

^{1}H NMR (400 MHz, $CDCl_3$): δ = ; 1.52-1.61 (m, 1 H), 1.77-1.85 (m, 1 H), 1.85 (s, 3 H), 2.42-2.75 (m, 5 H), 3.48-3.52 (m, 1 H), 3.80 (s, 3 H), 6.72-6.74 (m, 1 H), 6.83 (d, *J* = 8.8 Hz, 2 H), 7.03-7.06 (m, 4 H), 7.12-7.23 (m, 3 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 16.1, 26.7, 28.7, 33.3, 42.9, 51.7, 55.3, 113.8, 125.7, 128.2, 128.4, 128.6, 133.3, 134.3, 141.8, 143.1, 158.2, 202.6;

IR (KBr): 2923, 1667, 1610, 1513, 1454, 1370, 1253, 1182, 1035, 828, 751, 700 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{22}H_{25}O_2^+$, 321.1849; found, m/z 321.1844;

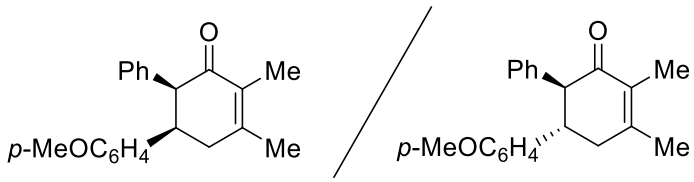
(*trans*-4p): R_f = 0.49 (*n*-hexane/EtOAc = 3/1). Colorless solid;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.67-1.76 (m, 1 H), 1.80-1.89 (m, 4 H), 2.45-2.66 (m, 5 H), 3.12-3.18 (m, 1 H), 3.82 (s, 3 H), 6.67-6.72 (m, 1 H), 6.85 (d, *J* = 8.8 Hz, 2 H), 7.01 (d, *J* = 8. Hz, 2 H), 7.09-7.12 (m, 3 H), 7.18-7.22 (m, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 16.2, 29.2, 32.6, 34.9, 45.3, 51.0, 55.3, 114.0, 125.6, 128.1, 128.4, 128.5, 135.0, 135.5, 142.5, 142.9, 158.3, 201.0;

IR (KBr): 2924, 1668, 1612, 1514, 14554, 1368, 1252, 1179, 1035, 829, 749, 701cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{22}H_{25}O_2^+$, 321.1849; found, m/z 321.1852;



5-(4-methoxyphenyl)-2, 3-dimethyl-6-phenylcyclohex-2-en-1-one

(4q)

Purified by preparative thin layer chromatography (0.0468 mmol scale. eluent: *n*-hexane/EtOAc = 3/1, 10.1 mg, 71%, *trans*:*cis* = 49:51).

(*cis*-4q): R_f = 0.40 (*n*-hexane/EtOAc = 3/1). Pale yellow solid; mp 124-126 °C;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.96 (s, 3 H), 2.10 (s, 3 H), 2.32-2.40 (m, 1 H), 2.75-2.83 (m, 1 H), 3.60-3.66 (m, 1 H), 3.76 (s, 3 H), 3.80 (d, *J* = 5.2 Hz, 1 H), 6.65 (d, *J* = 6.8 Hz, 2 H), 6.69-6.74 (m, 4 H), 7.08-7.17 (m, 3 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = 11.2, 21.8, 34.5, 44.0, 55.2, 58.5, 113.4, 126.8, 127.9, 128.9, 129.3, 132.1, 133.0, 135.5, 155.7, 158.3, 199.2;

IR (KBr): 2921, 1654, 1629, 1514, 1459, 1382, 1248, 1178, 1037, 829, 703 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{21}H_{23}O_2^+$, 307.1693; found, m/z 307.1702;

(*trans*-4q): R_f = 0.37 (*n*-hexane/EtOAc = 3/1). Pale yellow solid; mp 96-98 °C;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.86 (s, 3 H), 2.00 (s, 3 H), 2.56-2.61 (m, 1 H), 2.72-2.79 (m, 1 H), 3.45-3.52 (m, 1 H), 3.71 (s, 3 H), 3.73 (d, *J* = 12.8 Hz, 1 H), 6.69 (d, *J* = 8.8 Hz, 2 H), 6.94-7.00 (m, 4 H), 7.05-7.11 (m, 1 H), 7.14-7.18 (m, 2 H);

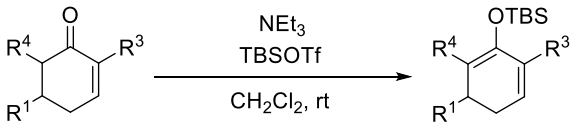
^{13}C NMR (100 MHz, $CDCl_3$) δ = 11.3, 21.4, 41.2, 46.3, 55.1, 59.1, 113.6, 126.3, 128.0, 128.4, 129.1, 131.3, 134.7, 139.0, 153.0, 158.0, 198.5;

IR (KBr): 2925, 1660, 1646, 1513, 1454, 1378, 1247, 1177, 1038, 837, 698 cm^{-1};

HRMS (ESI): $[M+H]^+$ calcd for $C_{21}H_{23}O_2^+$, 307.1693; found, m/z 307.1692;

4. Method for the derivatization of 2-cyclohexenones[7]

4.1 Procedure for the aromatization of 2-cyclohexenones **4e** and *cis*-**4k**



4

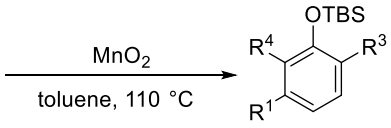
4e R^1 = *p*-$MeOC_6H_4$, R^3 = *t*-Bu, R^4 = H

***cis*-4k** R^1 = *p*-$MeOC_6H_4$, R^3 = Me, R^4 = Ph

7

7e R^1 = *p*-$MeOC_6H_4$, R^3 = *t*-Bu, R^4 = H

7k R^1 = *p*-$MeOC_6H_4$, R^3 = Me, R^4 = Ph



8

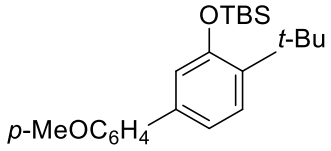
8e R^1 = *p*-$MeOC_6H_4$, R^3 = *t*-Bu, R^4 = H

8k R^1 = *p*-$MeOC_6H_4$, R^3 = Me, R^4 = Ph

Cyclohexenone **4e** (0.0762 mmol) was dissolved in dehydrated CH_2Cl_2 (2.0 mL), and the solution was cooled to 0 °C. To the solution, TBSOTf (5 equiv., 88 μL) was added dropwise. Then, NEt_3 (7 equiv., 73 μL) was added dropwise. The reaction mixture was then allowed to stir at room temperature overnight. The reaction was quenched by pouring the reaction solution into water (10 mL), the reaction mixture was diluted with EtOAc (15 mL), and washed with brine (10 mL). The organic phase was dried over Na_2SO_4, and the solvent was removed under reduced pressure and dried *in vacuo* to obtain the corresponding silyl enol ether **7e**. The silyl enol ether **7e** was immediately used for the next step without further purification.

A magnetic stirrer bar and MnO_2 (40 equiv., 0.264 g) were placed in a Schlenk tube. MnO_2 was then dried with a heat gun under reduced pressure and the tube was filled with argon. The solution of silyl enol ether **7e** in dehydrated toluene (2.0 mL) was added to the Schkenk tube and the Schlenk tube was warmed up to 110 °C. After 22 h, MnO_2 was removed through a Celite® pad using EtOAc as eluent. After evaporation, the residue was purified by preparative thin layer chromatography (*n*-hexane/EtOAc = 5/1) to give the phenol derivative **8e** (24.2 mg, 0.0653 mmol, 86% yield).

4.2 Characterization data of products



***tert*-butyl((4-(*tert*-butyl)-4'-methoxy-[1,1'-biphenyl]-3-yl)oxy)dimethylsilane**

(8e)

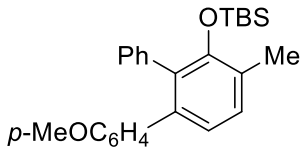
Purified by preparative thin layer chromatography (0.0762 mmol scale. eluent: *n*-hexane/EtOAc = 5/1, 24.2 mg, 86%); R_f = 0.79 (*n*-hexane/EtOAc = 3/1). Colorless solid; mp 92-94 °C;

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.37 (s, 6 H), 1.06 (s, 9 H), 1.41 (s, 9 H), 3.84 (s, 3 H), 6.96 (d, *J* = 8.8 Hz, 2 H), 7.02 (s, 1 H), 7.05 (d, *J* = 8.4 Hz, 1 H), 7.32 (d, *J* = 8.4 Hz, 1 H), 7.47 (d, *J* = 8.8 Hz, 2 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = -3.4, 18.8, 26.3, 29.8, 34.5, 55.3, 114.2, 117.1, 118.5, 127.4, 127.8, 133.4, 137.8, 139.4, 154.7, 159.0;

IR (KBr): 3010, 2962, 2859, 1610, 1554, 1490, 1256, 1221, 1180, 936, 780 cm^{-1};

HRMS (ESI): $[M]^+$ calcd for $C_{23}H_{34}O_2Si$, 370.2328; found, m/z 370.2331.



***tert*-butyl((4-methoxy-4'-methyl-[1,1':2',1''-terphenyl]-3'-yl)oxy)dimethylsilane**

(8k)

Purified by preparative thin layer chromatography (0.0434 mmol scale. eluent: *n*-hexane/EtOAc = 5/1, 9.50 mg, 54%); R_f = 0.79 (*n*-hexane/EtOAc = 3/1). Colorless solid; mp 73-74 °C;

^{1}H NMR (400 MHz, $CDCl_3$): δ = -0.40 (s, 6 H), 0.87 (s, 9 H), 2.32 (s, 3 H), 3.74 (s, 3 H), 6.66 (d, *J* = 8.8 Hz, 2 H), 6.89 (d, *J* = 8.8 Hz, 2 H), 6.95 (d, *J* = 7.6 Hz, 1 H) 7.08-7.17 (m, 6 H);

^{13}C NMR (100 MHz, $CDCl_3$) δ = -4.1, 17.9, 18.6, 26.1, 55.1, 112.9, 123.7, 126.2, 127.3, 128.1, 130.8, 132.1, 132.5, 134.5, 137.9, 140.2, 151.0, 157.8;

IR (KBr): 2957, 2930, 2857, 1615, 1472, 1256, 1181, 1109, 1037, 834 cm^{-1};

HRMS (ESI): $[M]^+$ calcd for $C_{26}H_{32}O_2Si$, 404.2172; found, m/z 404.2173.

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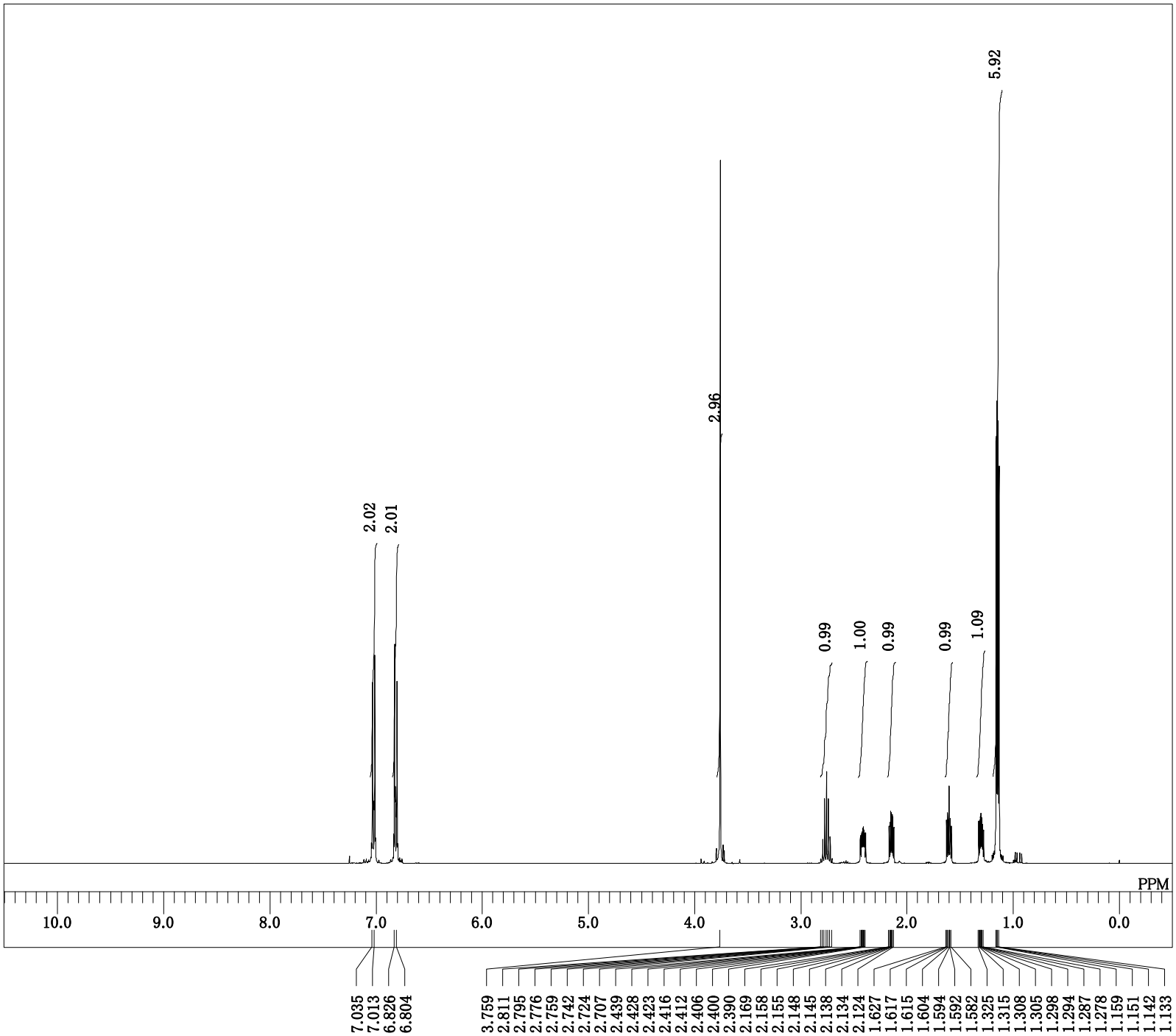

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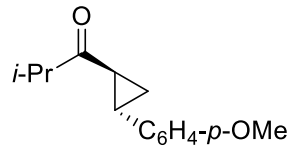

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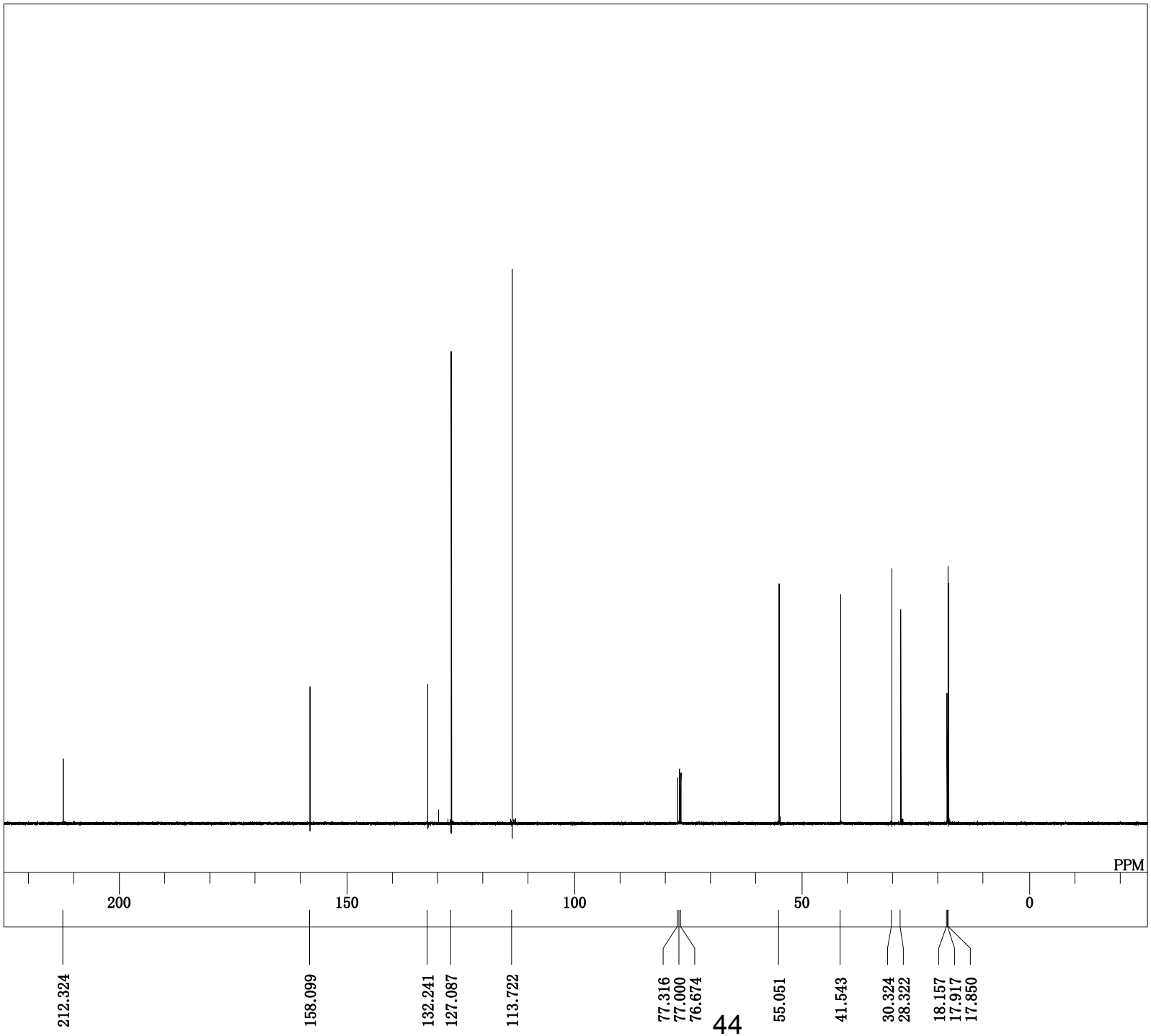

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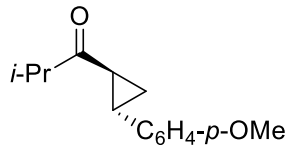
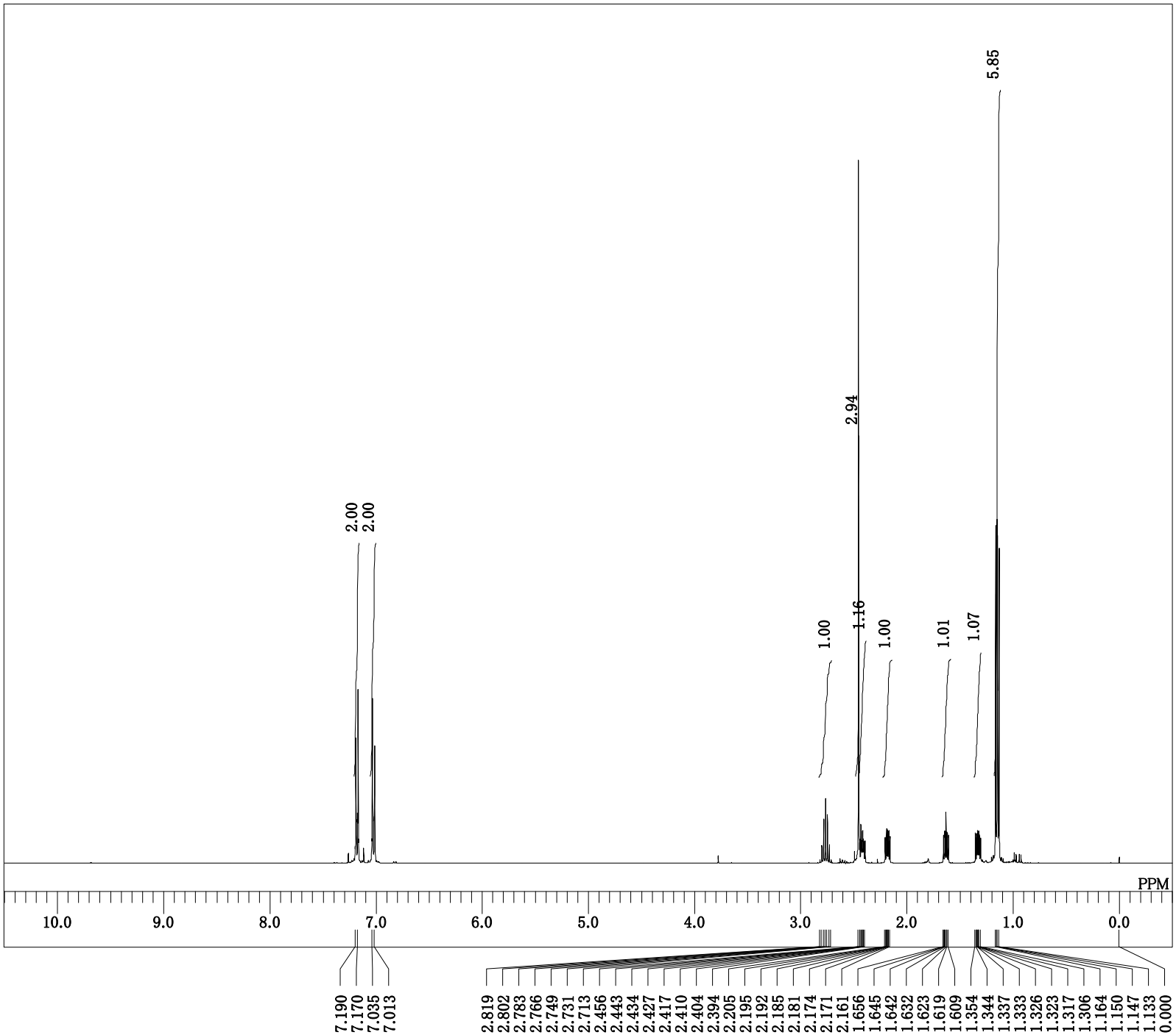


1a



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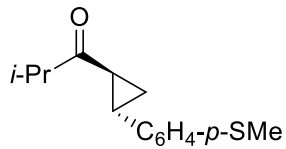

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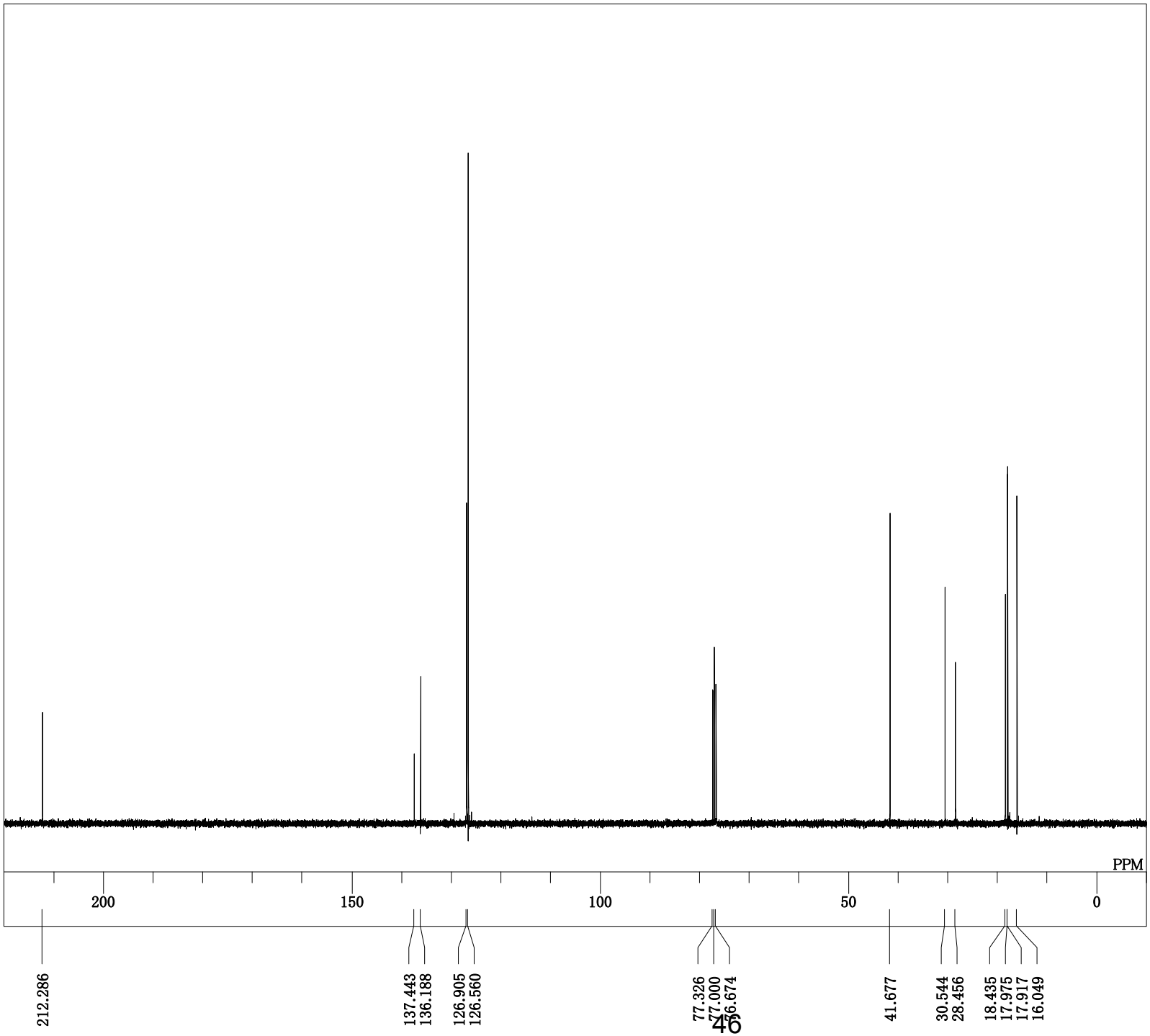

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OBFIN	0.98 Hz
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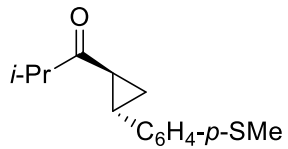
RGAIN	50



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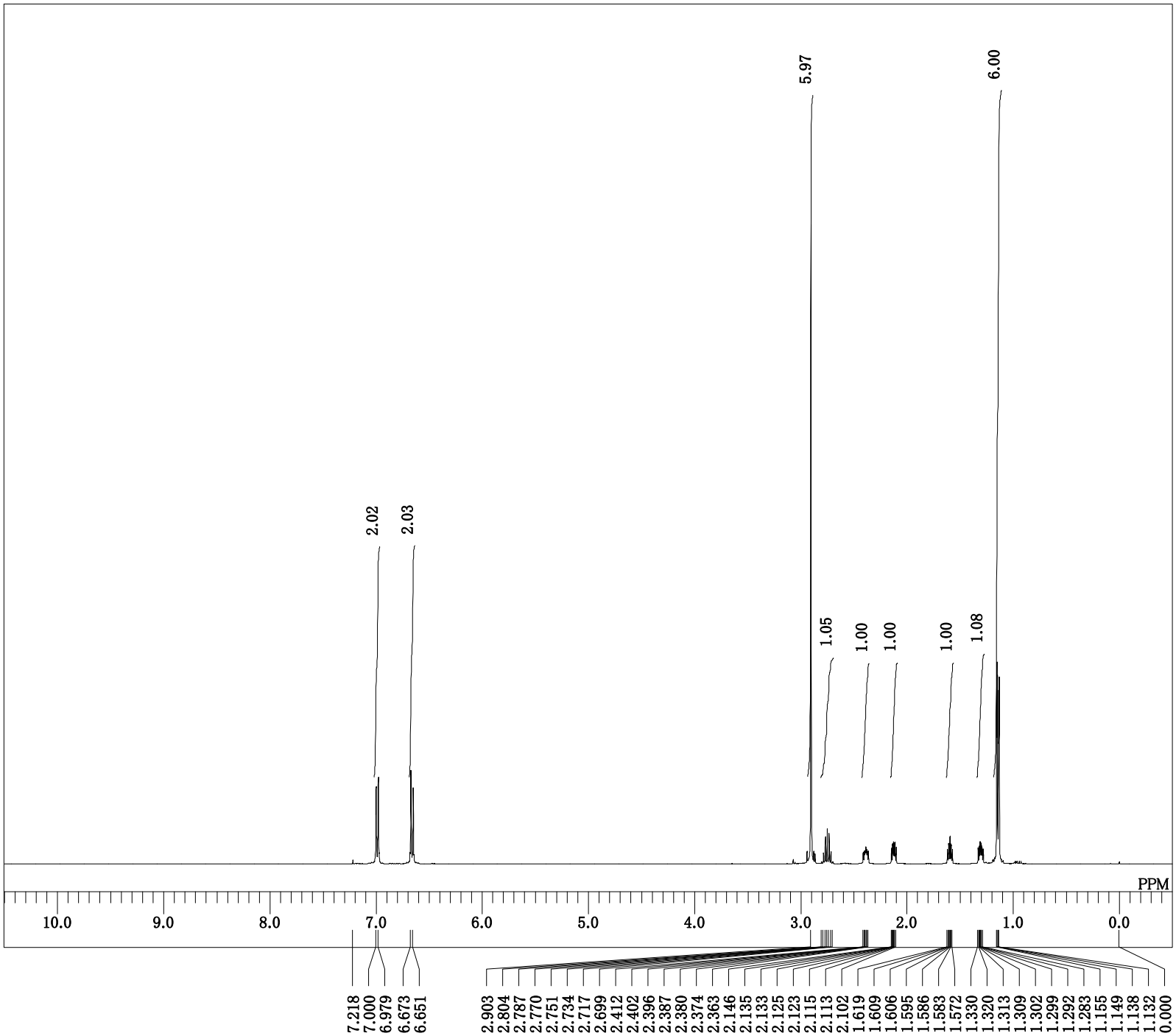

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OBFIN	0.87 Hz
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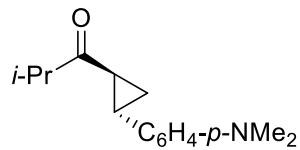
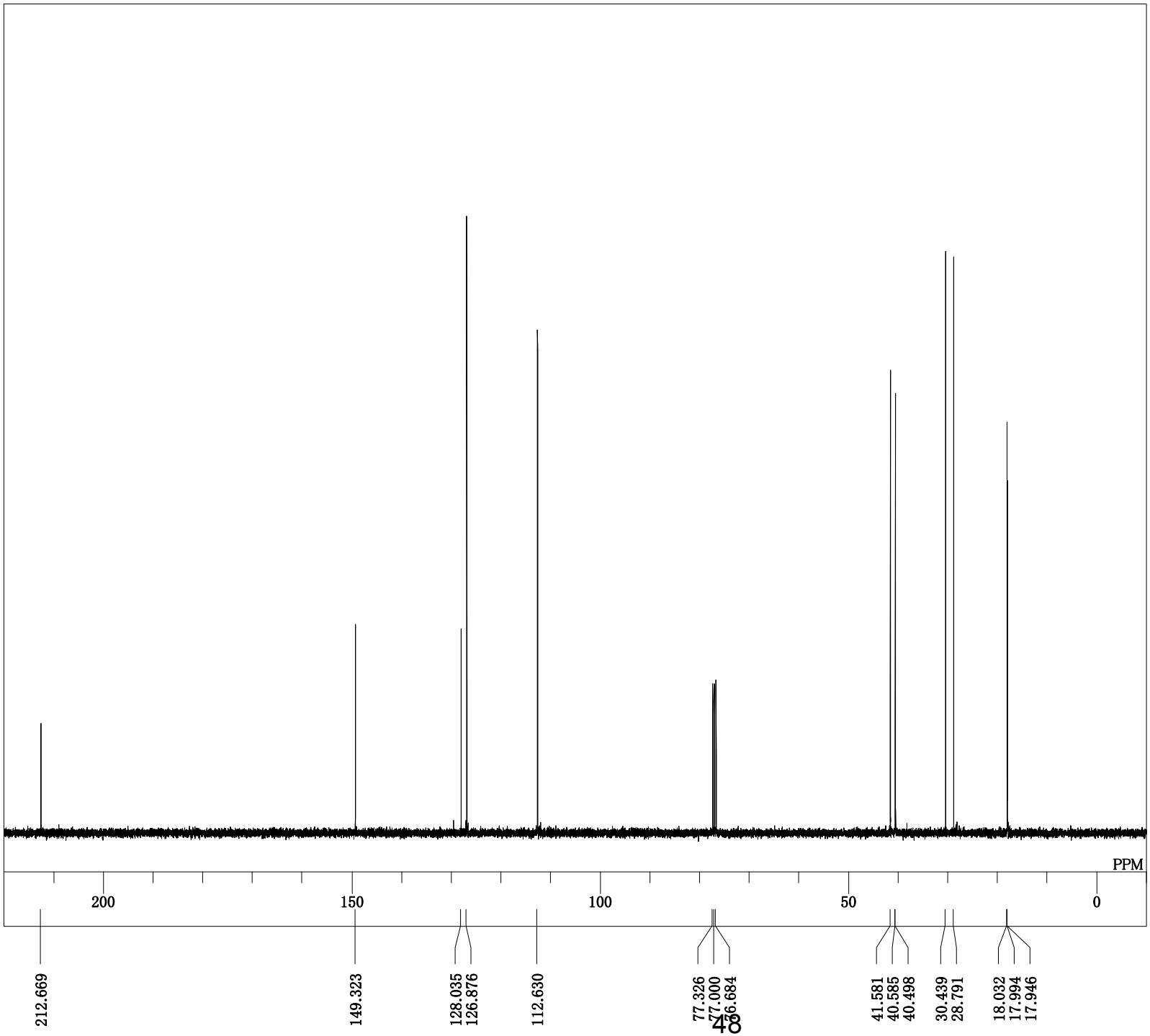
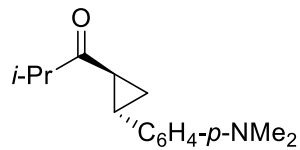
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	18.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	14



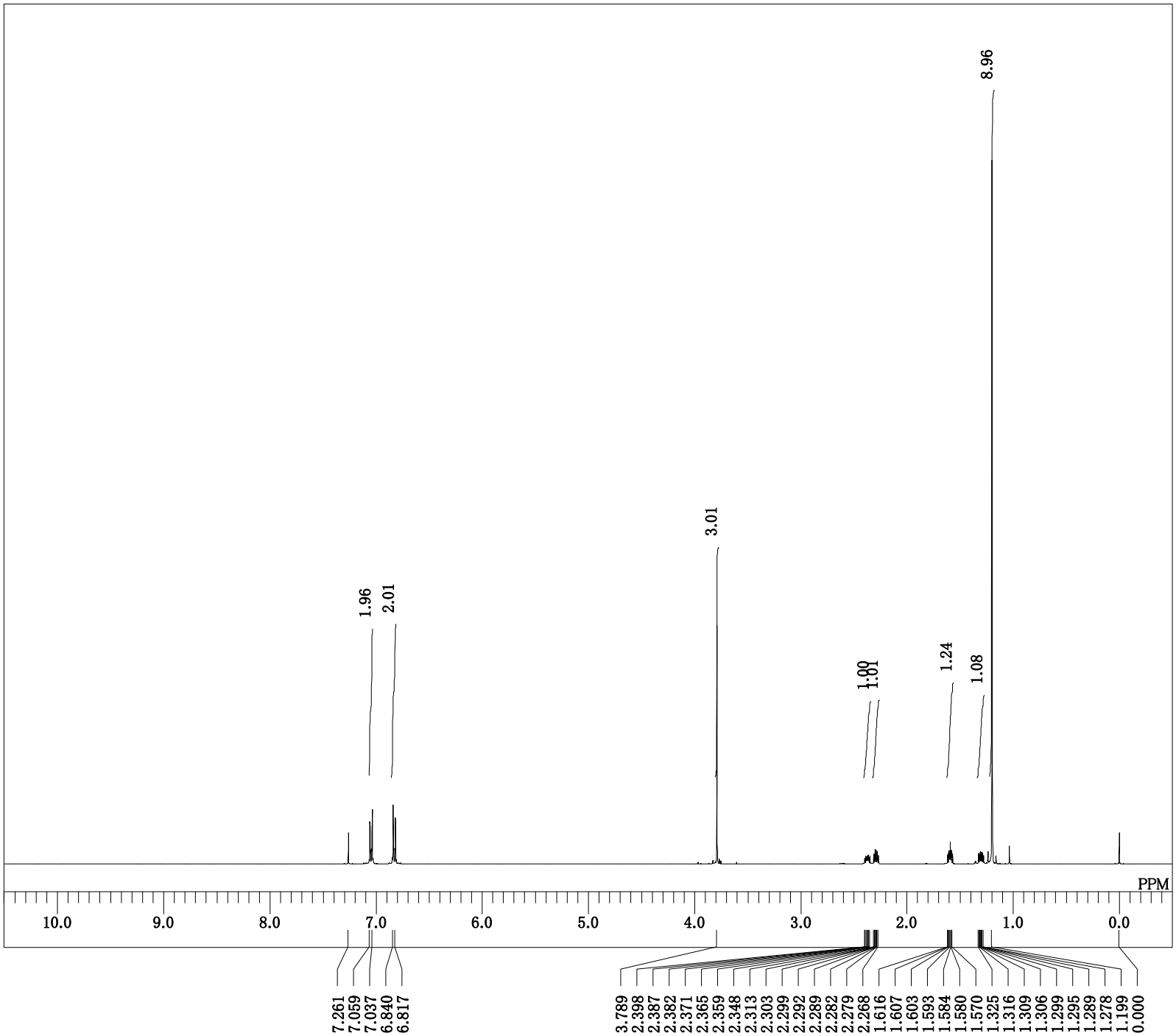
1c



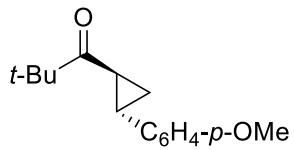
DFILE	1c_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-12-29 18:19:26
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	128
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	18.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



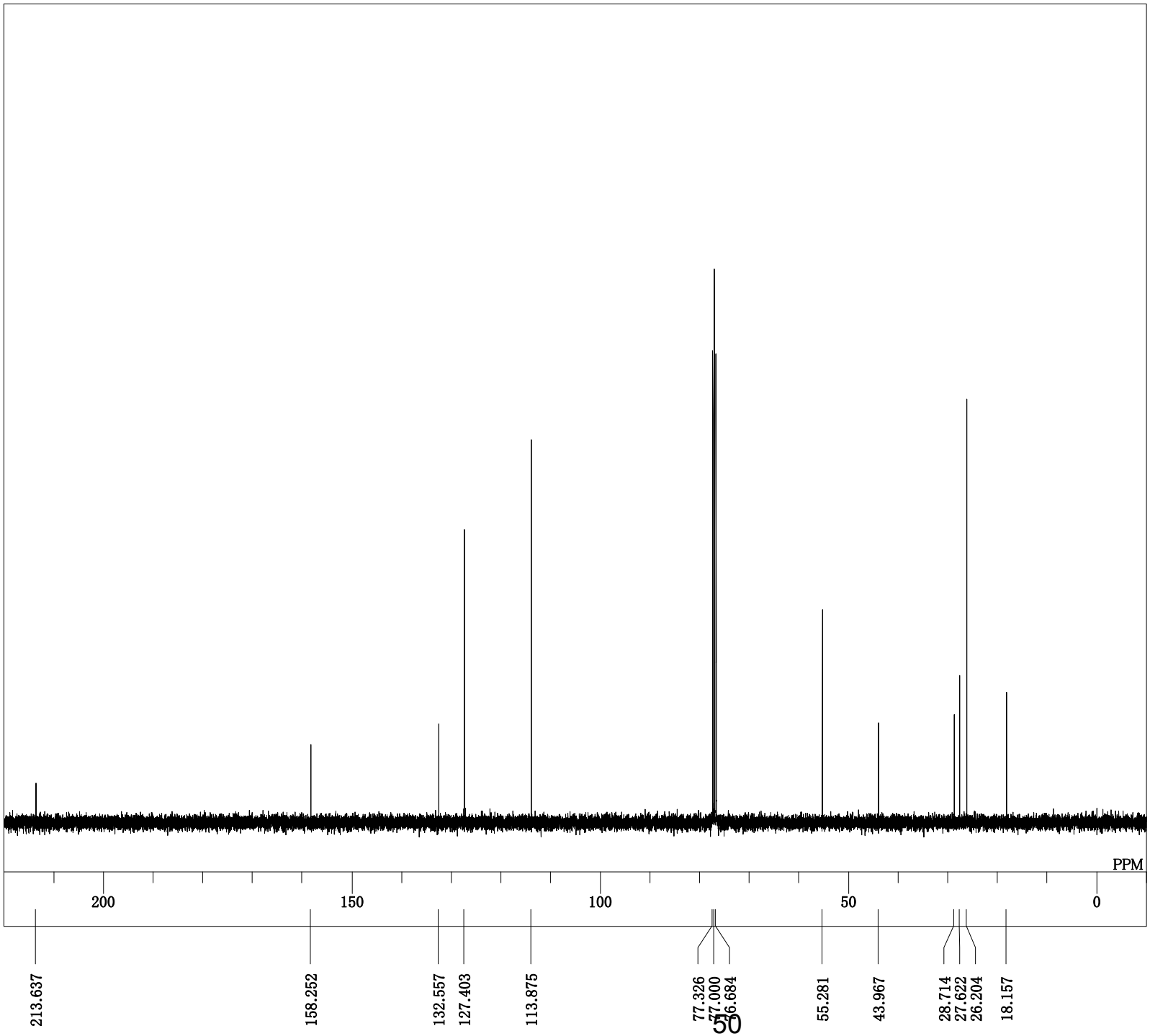
1c



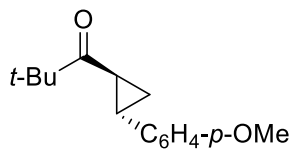
DFILE	1e_1H.als
COMNT	single_pulse
DATIM	2020-12-17 18:57:09
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	16384
FREQU	7422.80 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	18.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	34



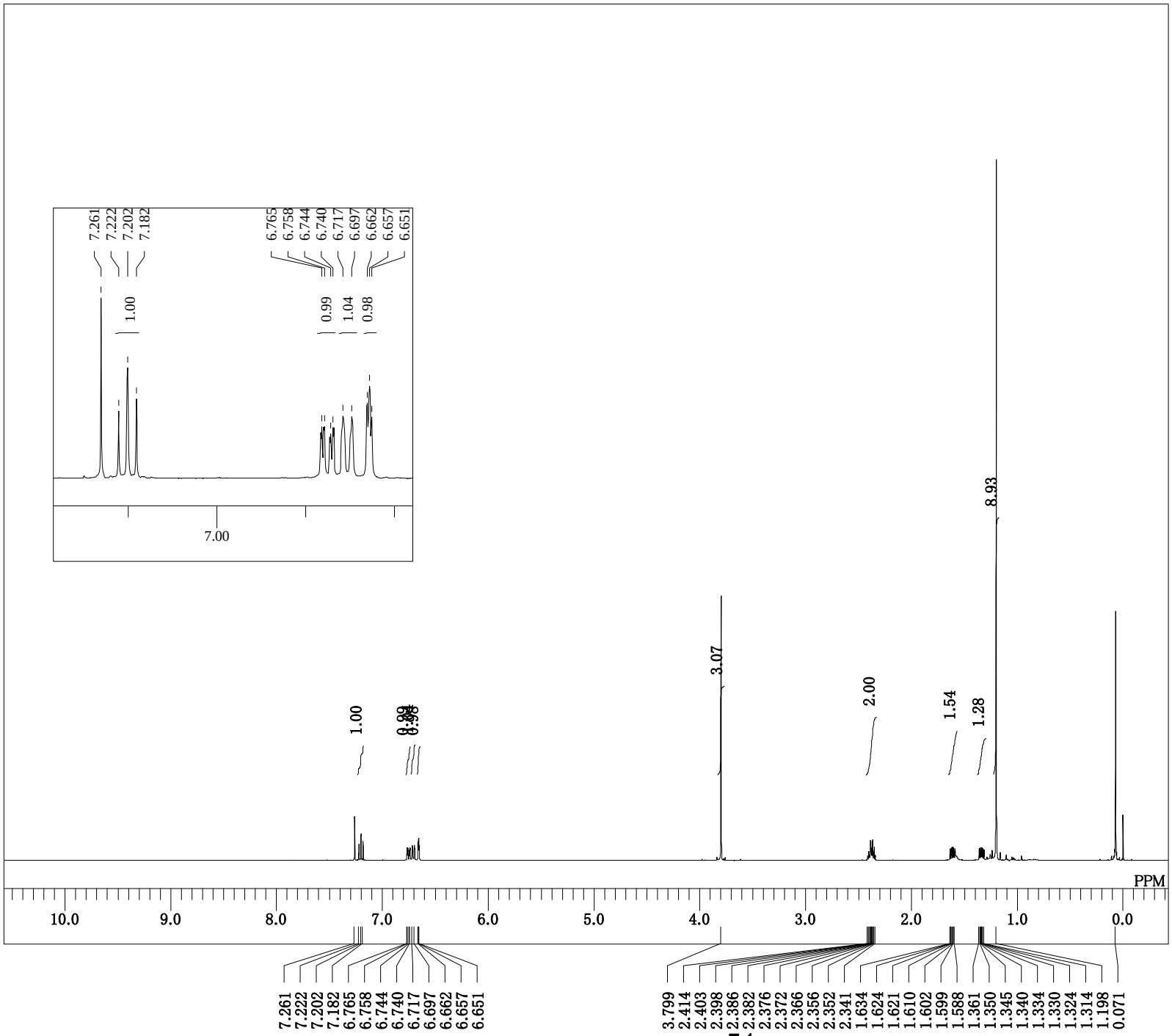
1e



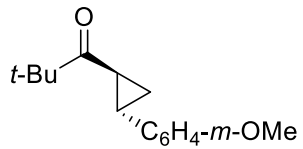
DFILE	1e_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-12-17 18:58:41
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	256
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



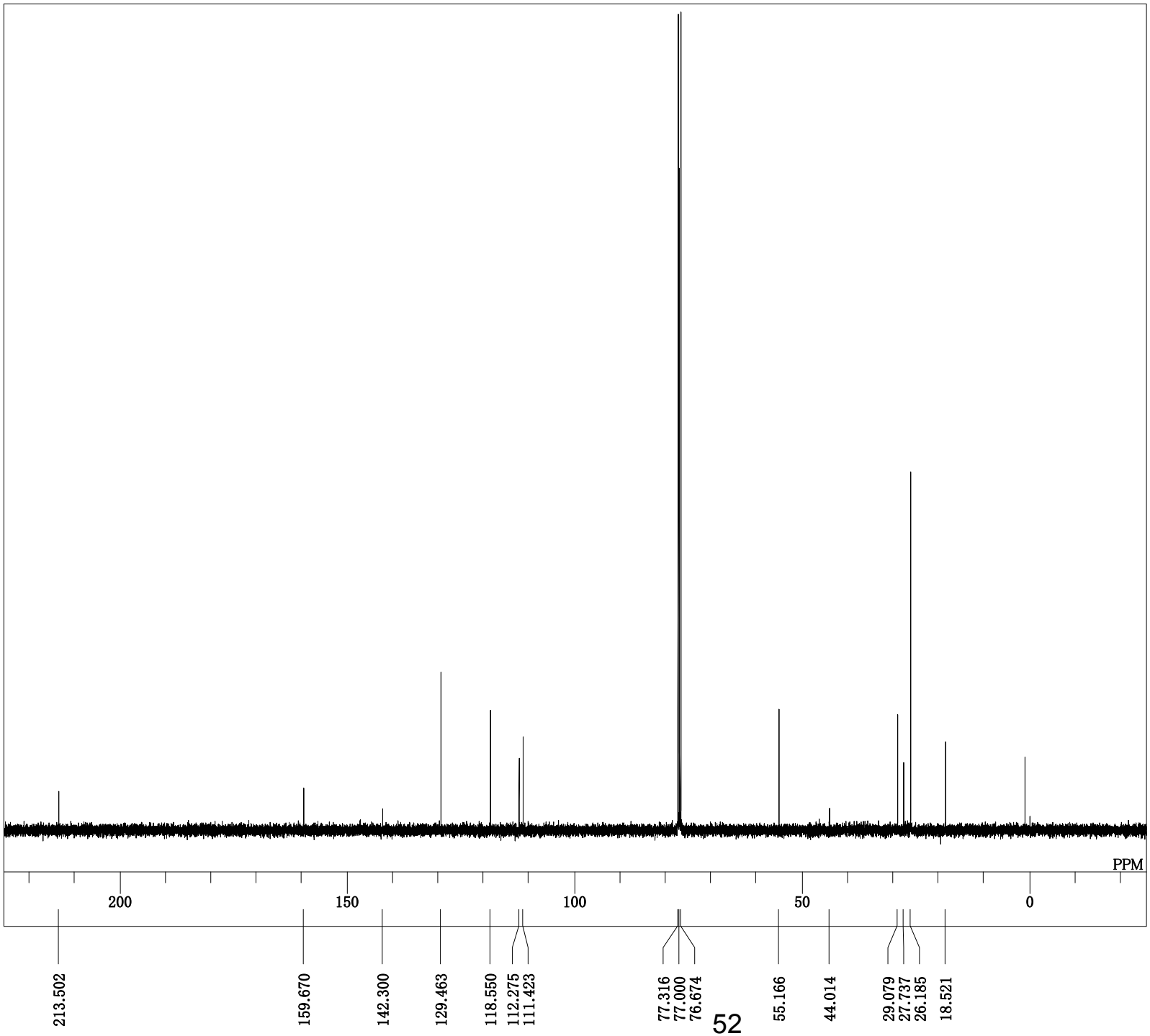
1e



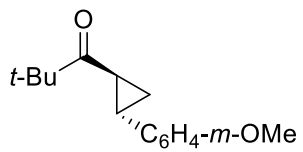
DFILE	1f_1H.als
COMNT	single_pulse
DATIM	2020-12-23 21:13:46
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	19.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	36



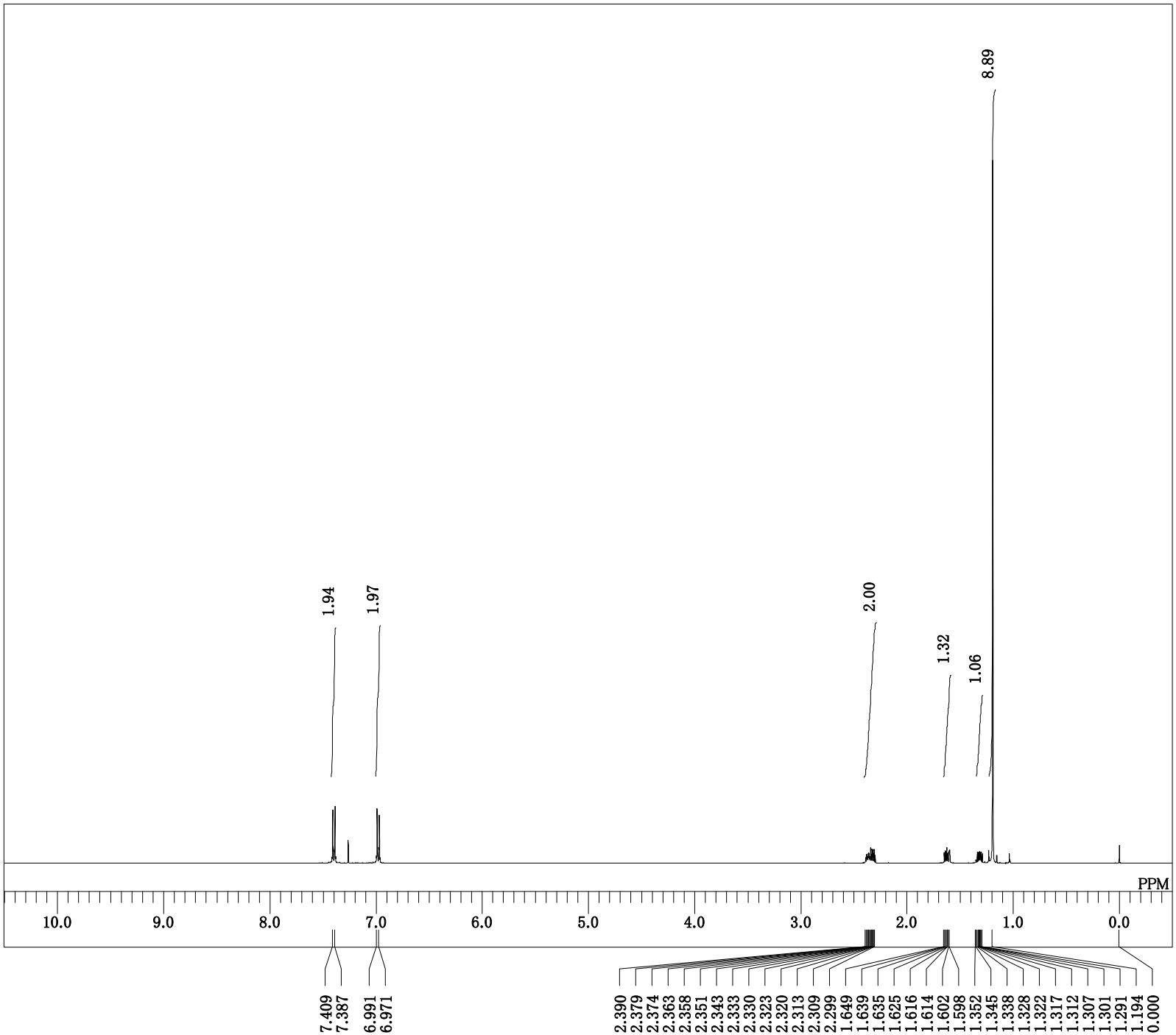
1f



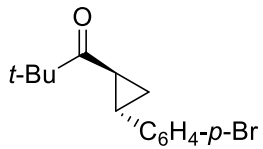
DFILE	1f_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-12-23 21:14:59
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	1024
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	18.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



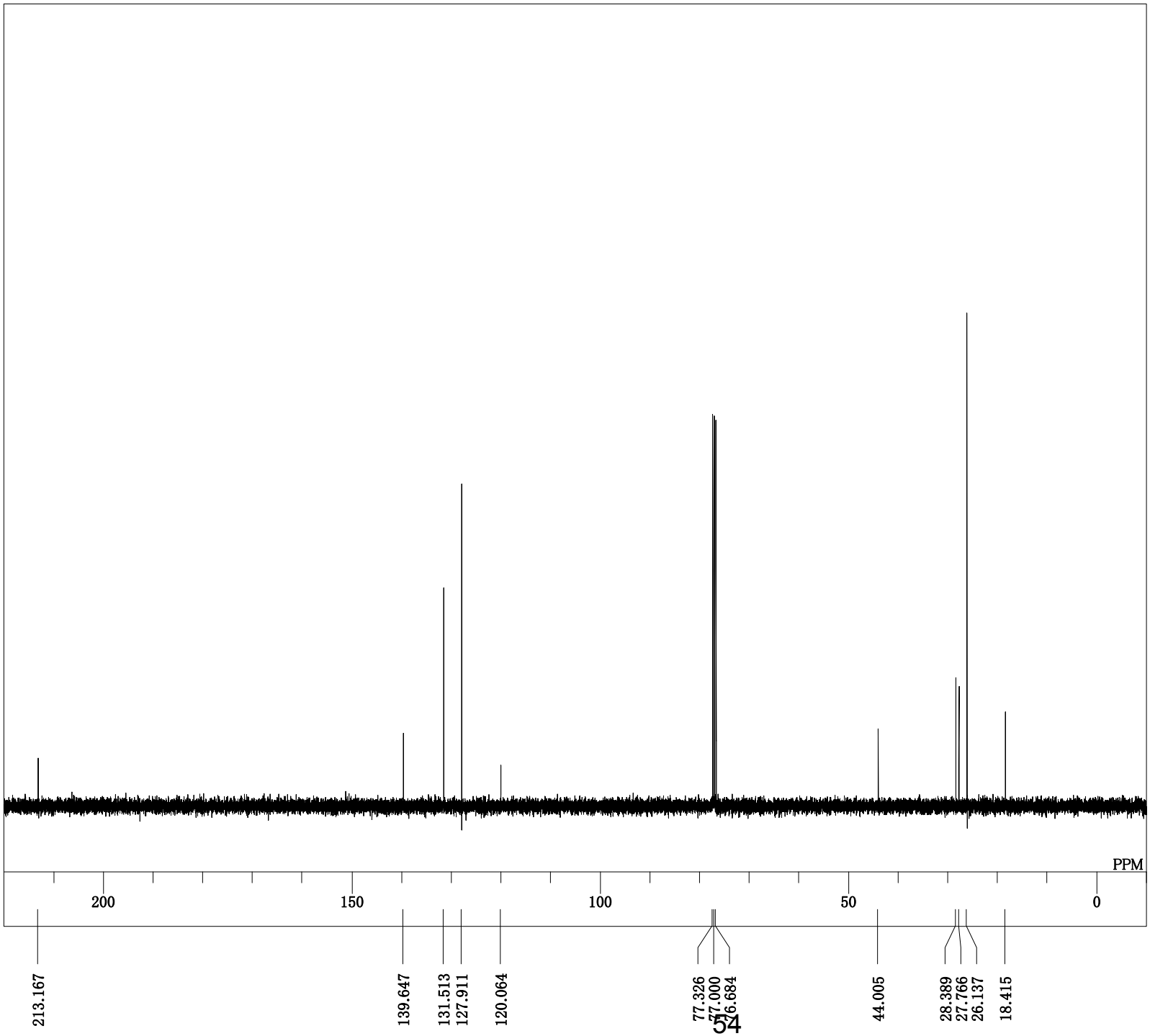
1f



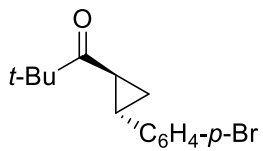
DFILE	1g_1H.als
COMNT	single_pulse
DATIM	2020-12-28 14:37:06
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	18.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	34



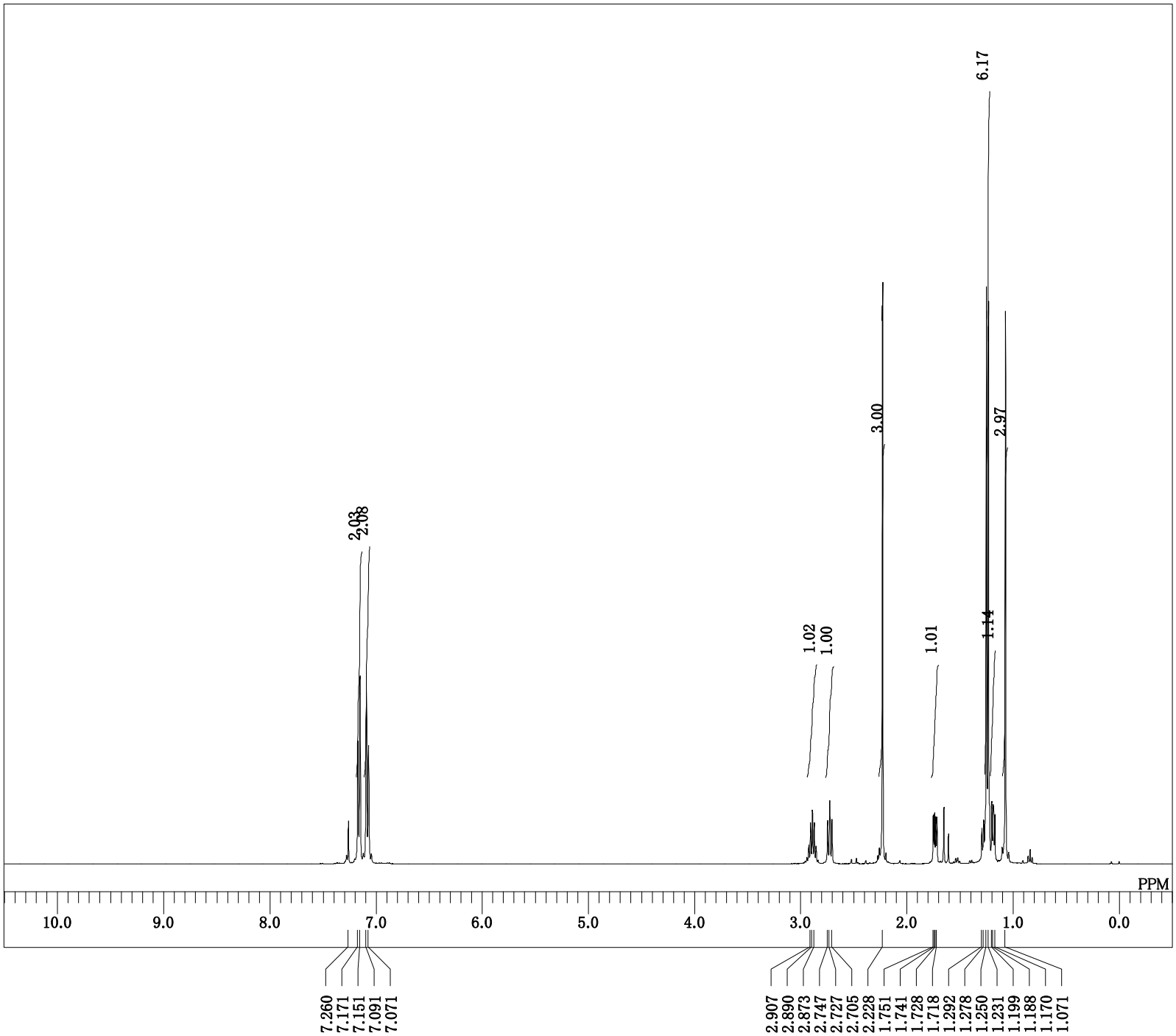
1g



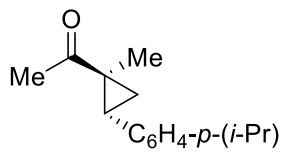
DFILE	1g_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-12-28 14:38:18
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	128
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



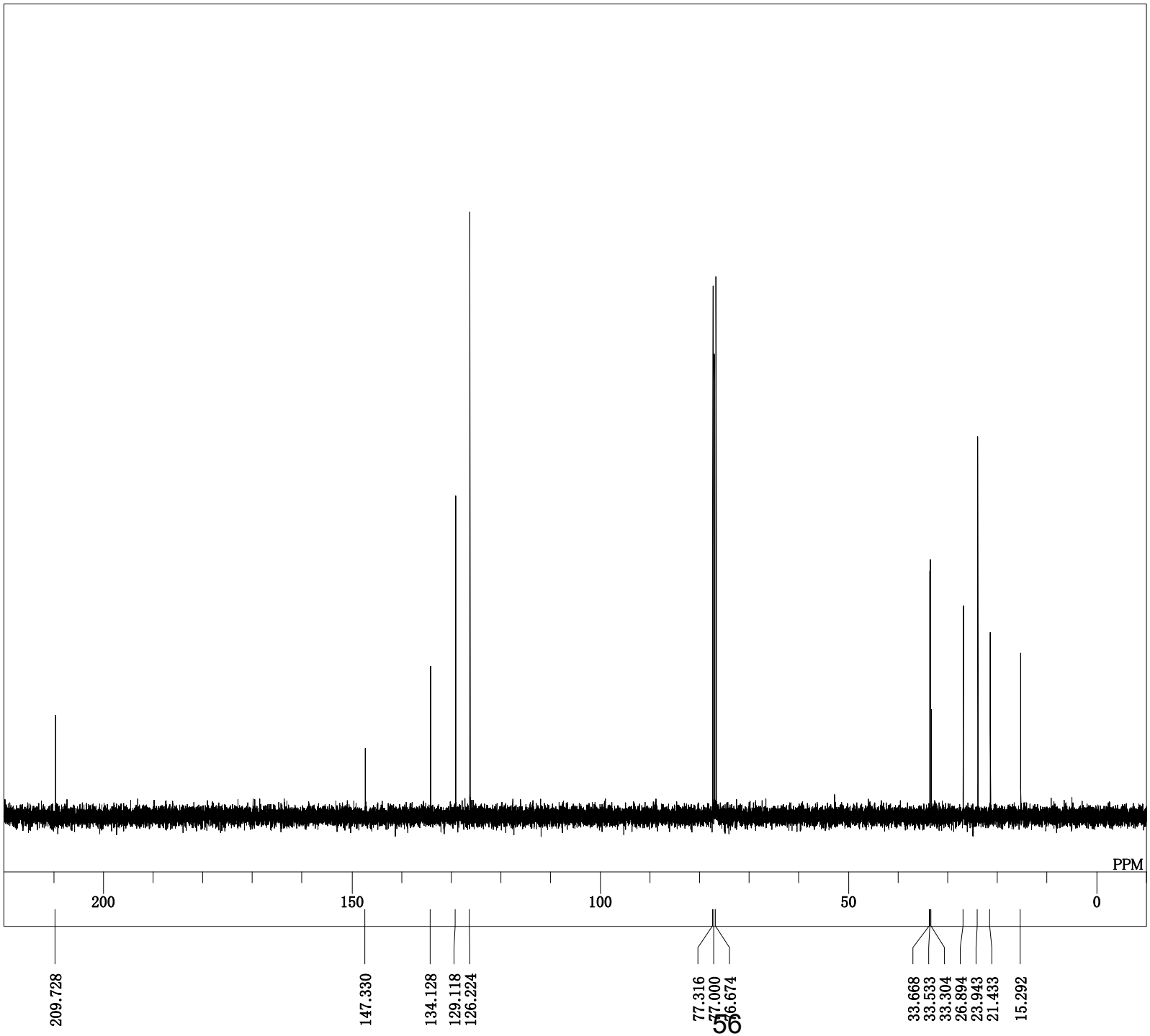
1g



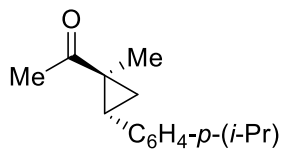
DFILE	1i_1H.als
COMNT	single_pulse
DATIM	2020-12-06 13:47:53
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	18.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	28



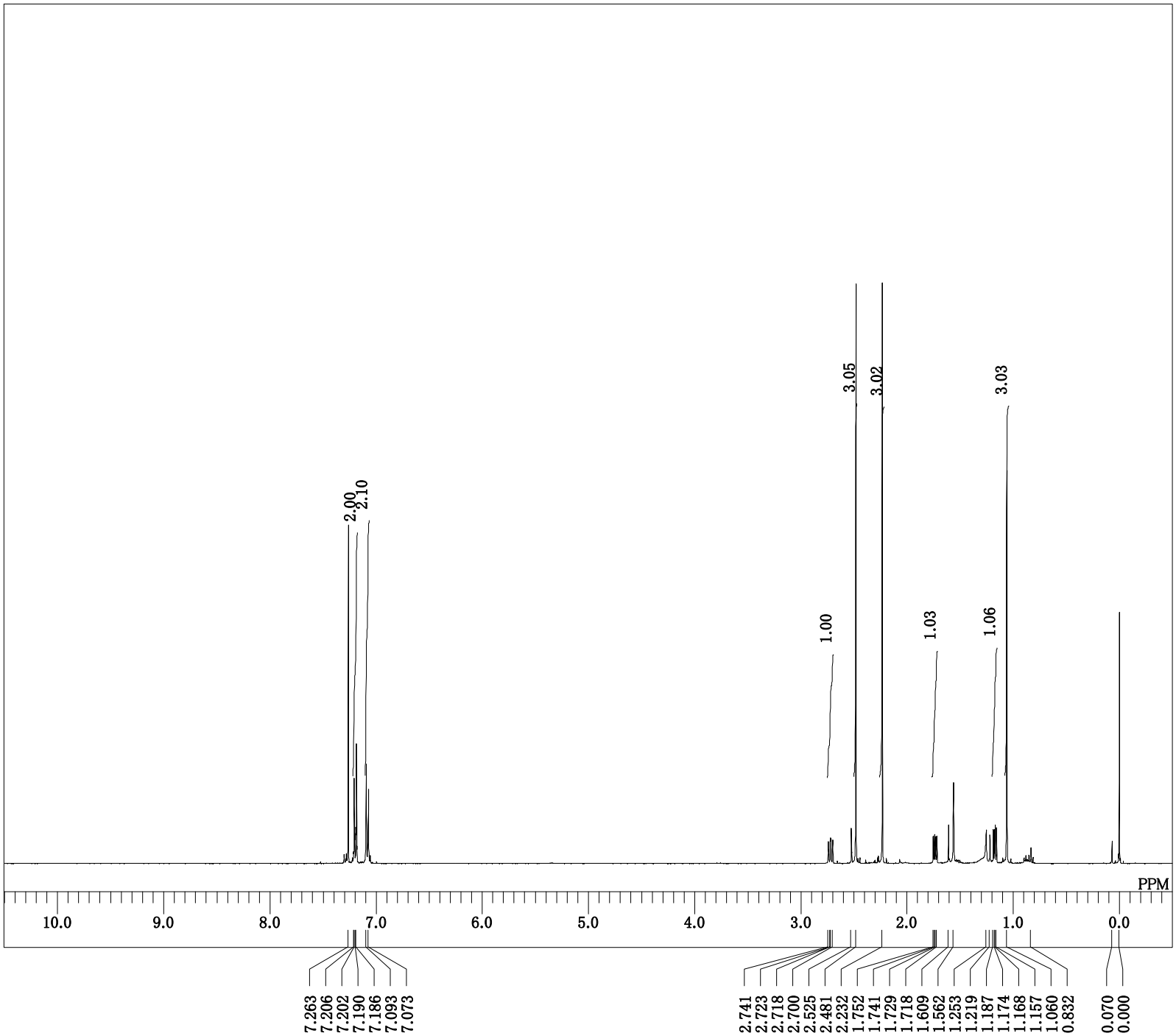
1i



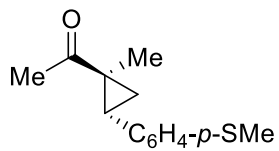
DFILE	1i_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-12-06 13:49:05
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	128
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



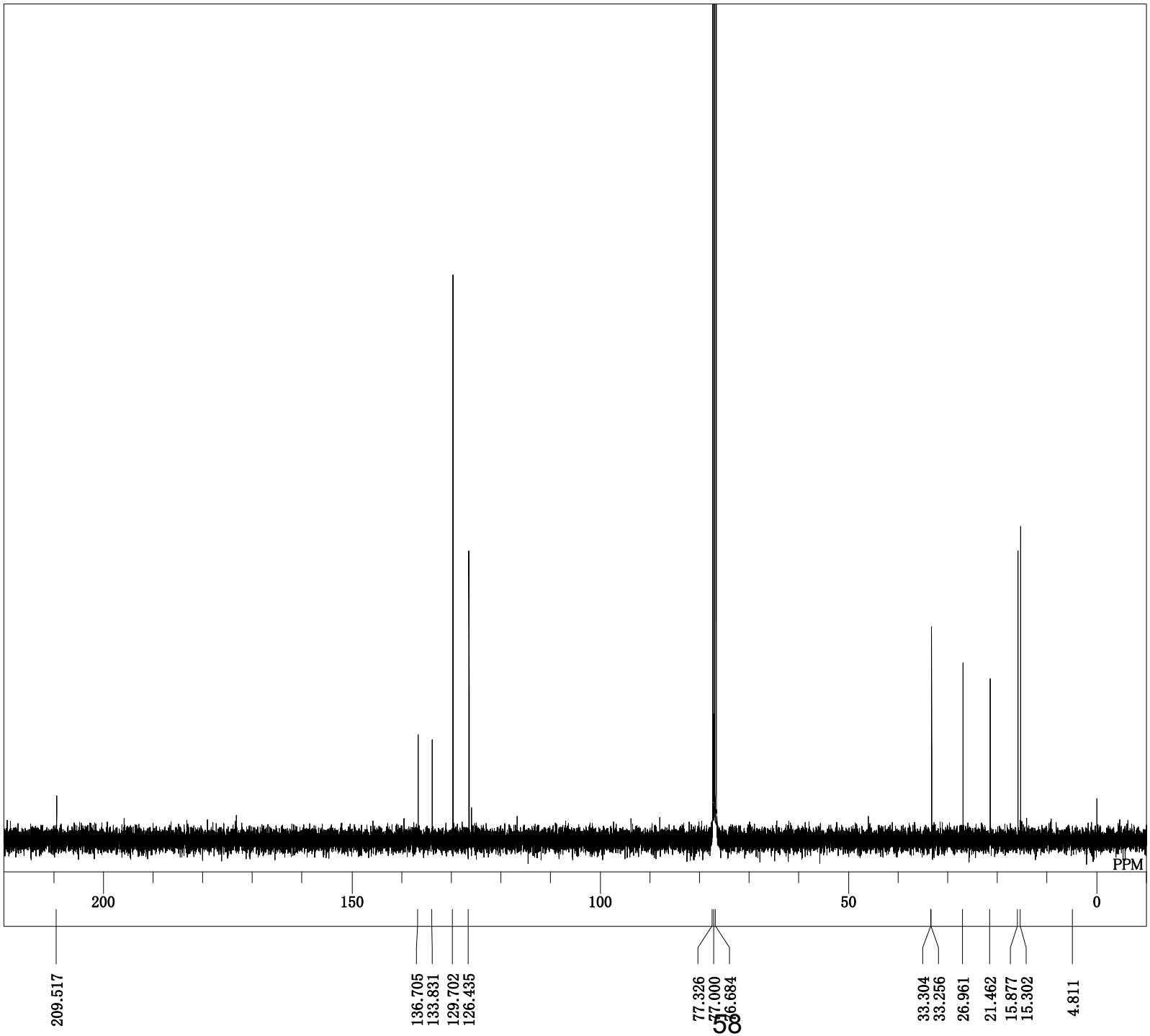
1i



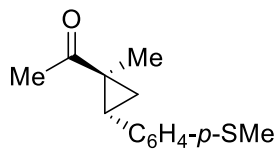
DFILE	1j_1H.als
COMNT	single_pulse
DATIM	2020-12-16 22:18:24
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	16
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	18.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	44



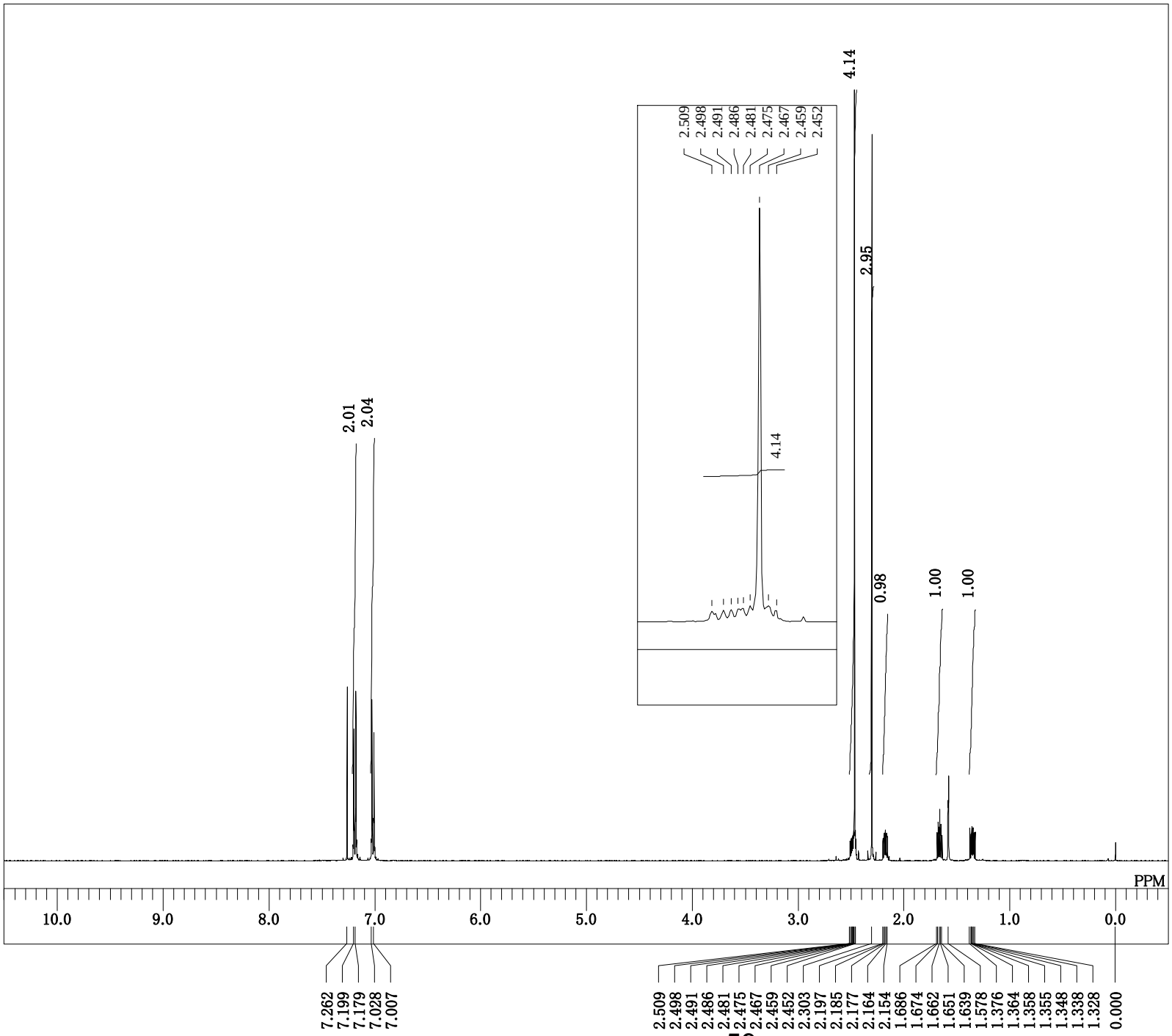
1j



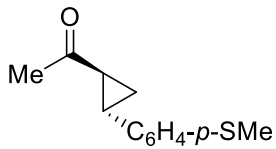
DFILE	1j_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-12-16 22:20:33
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	8192
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	18.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



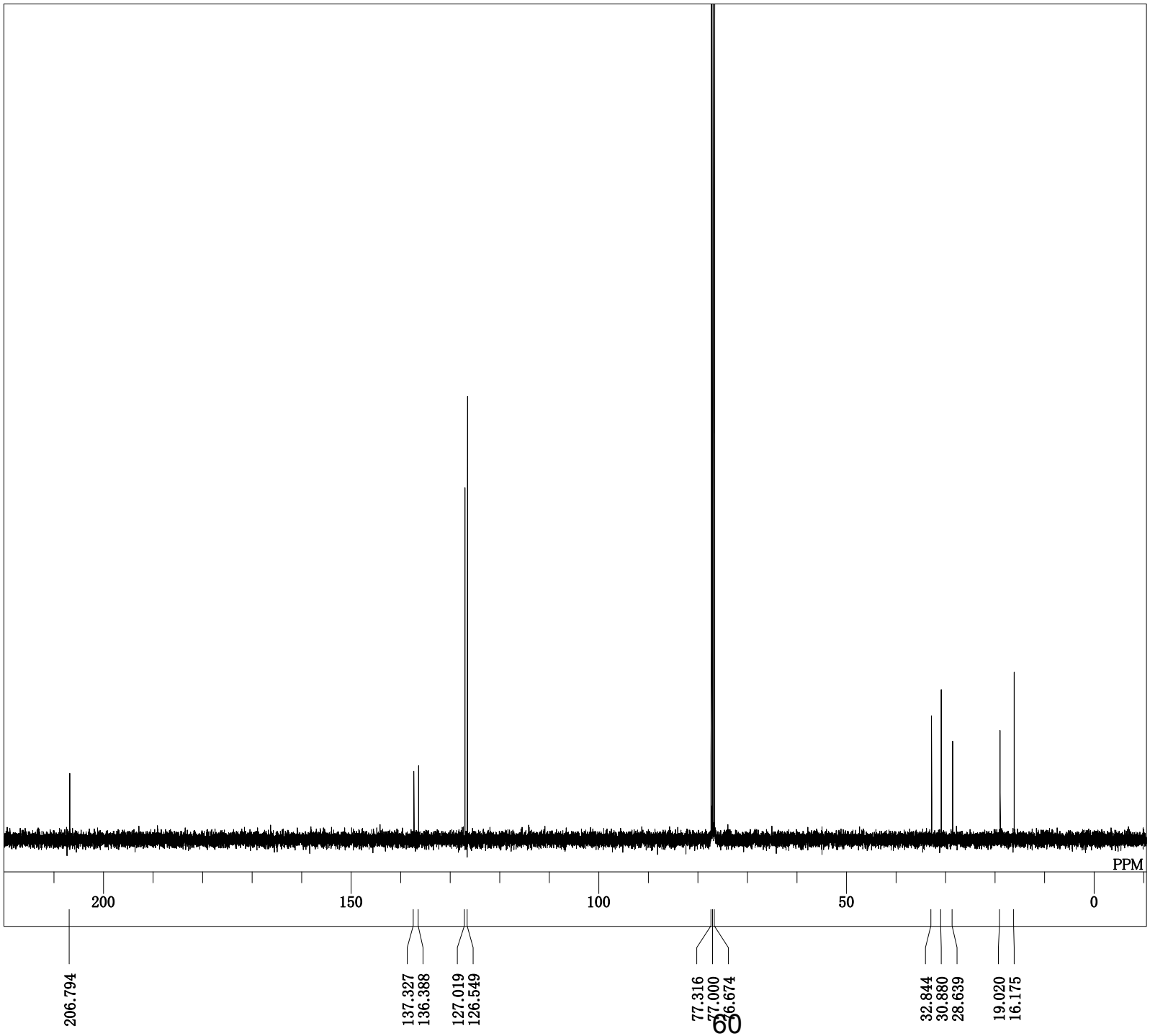
1j



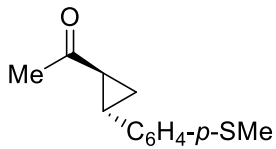
DFILE	1l_1H.als
COMNT	single_pulse
DATIM	2021-01-24 11:22:26
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	16384
FREQU	7422.80 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	19.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	42



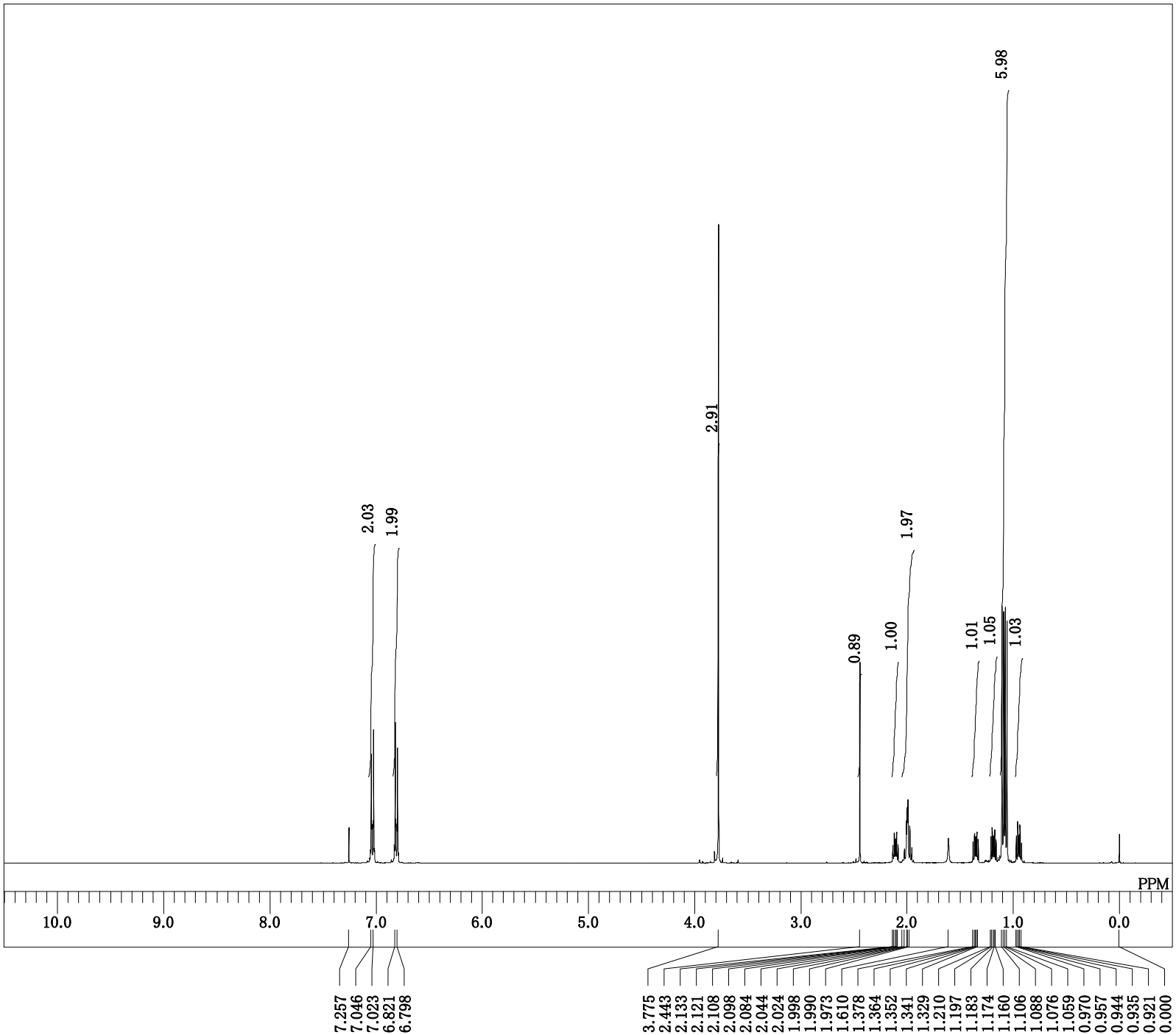
1l



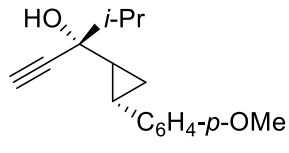
DFILE	1l_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2021-01-24 18:12:42
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	32767
FREQU	31250.00 Hz
SCANS	1024
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



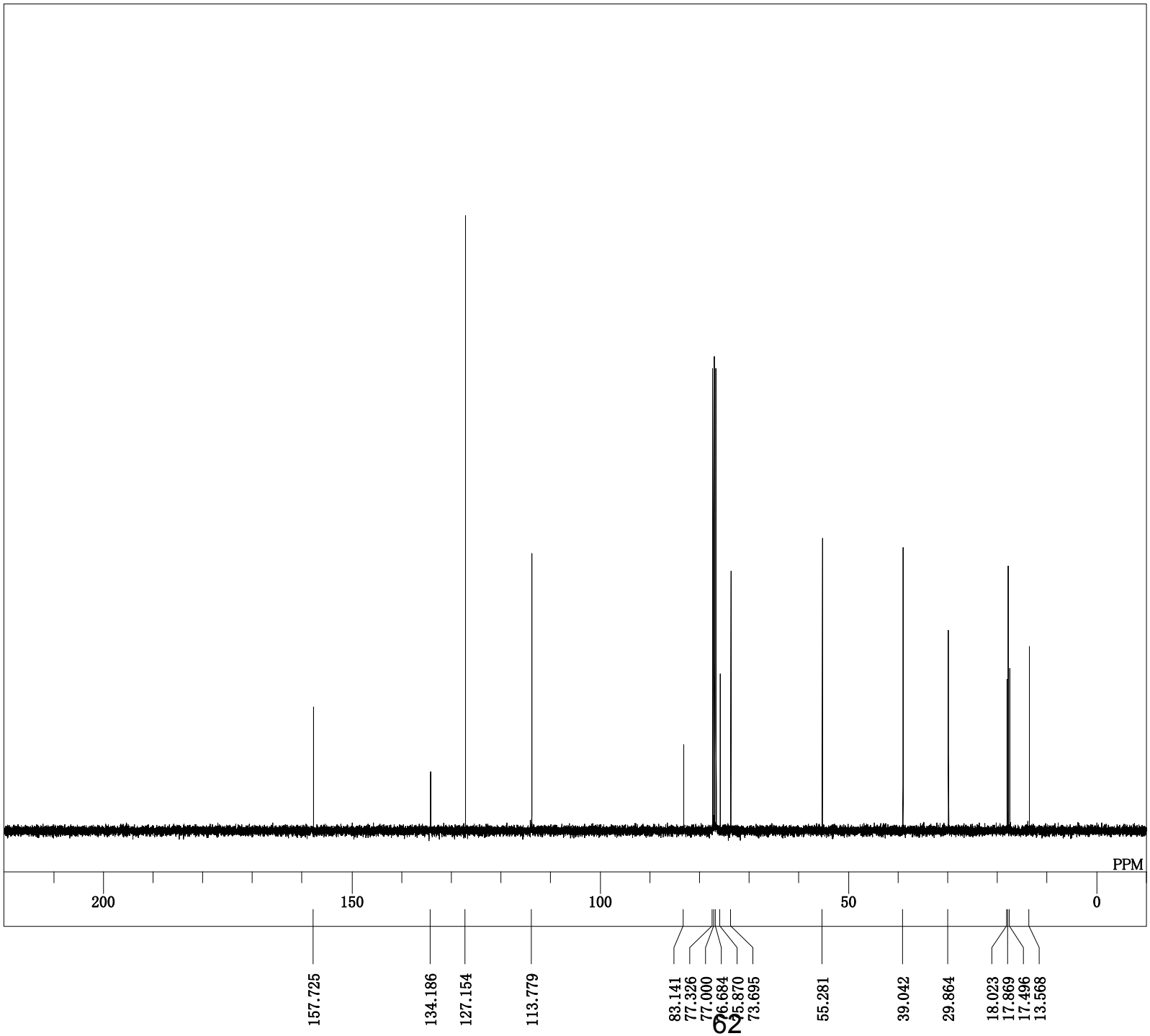
1l



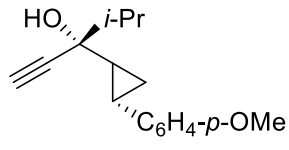
DFILE	alpha_2a_1H.als
COMNT	single_pulse
DATIM	2020-10-28 12:58:53
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	30



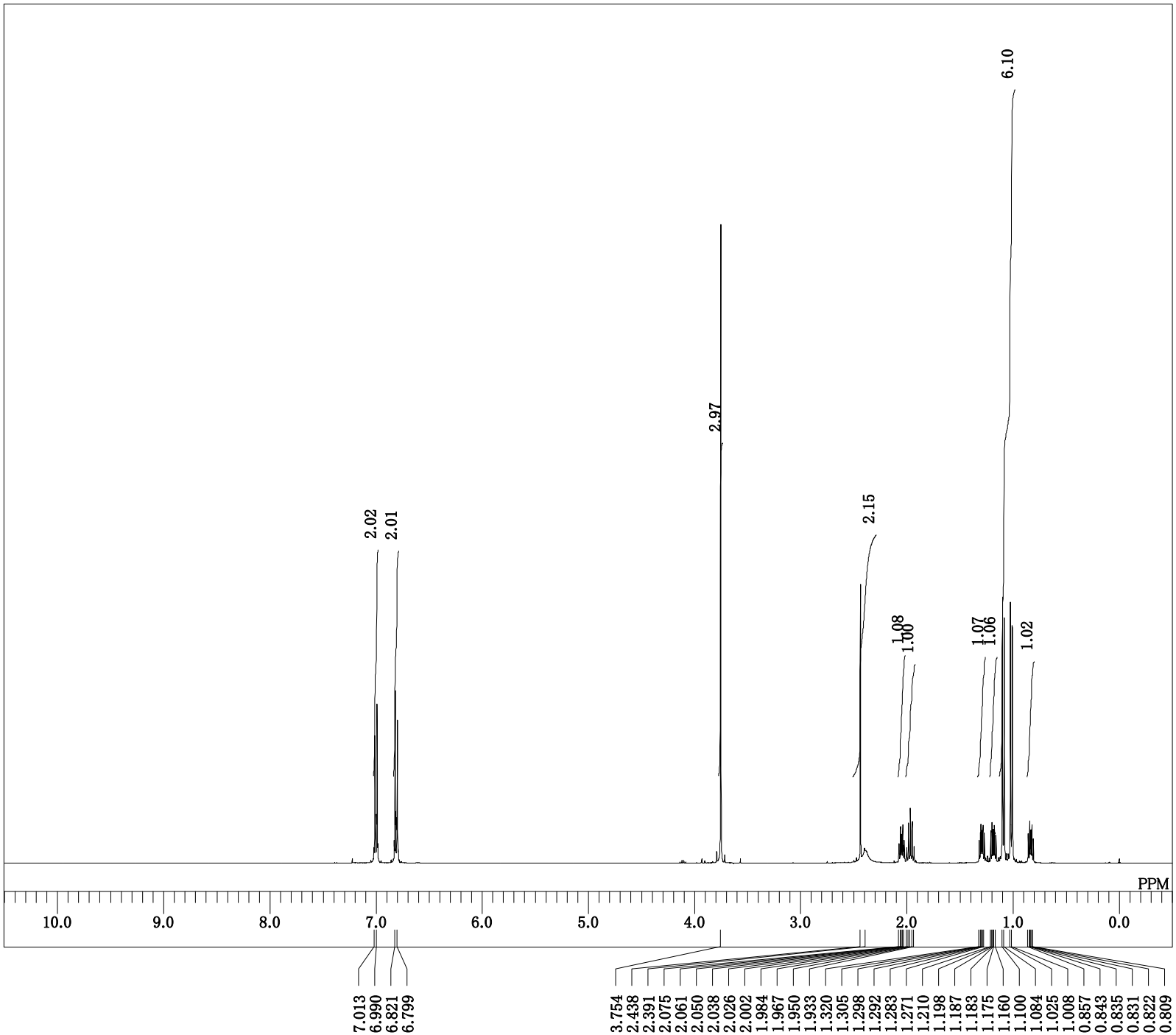
α-2a



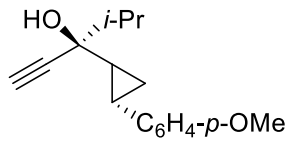
DFILE	alpha_2a_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-12-24 21:29:44
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	512
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	18.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



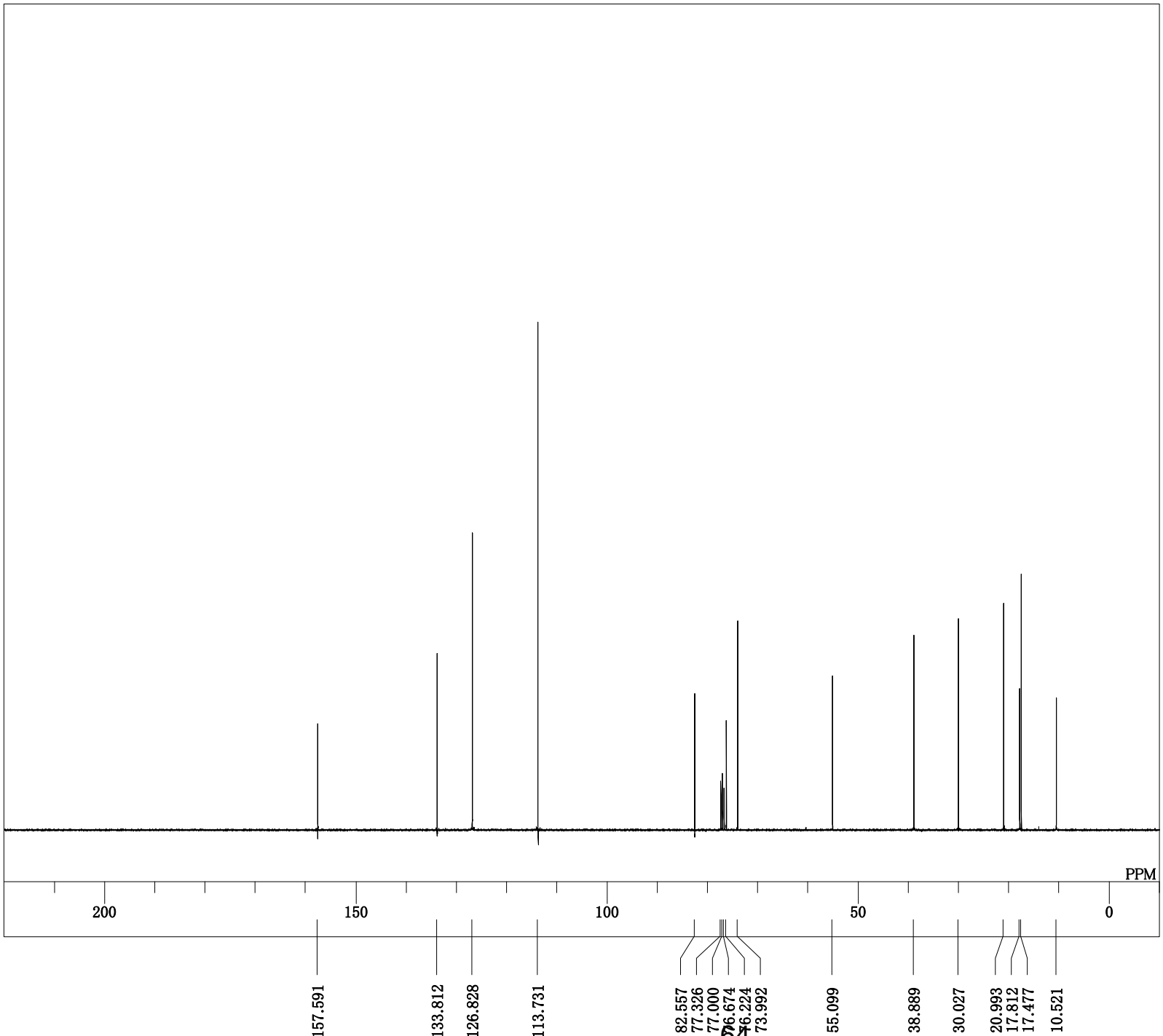
α-2a



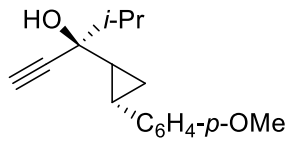
DFILE	beta_2a_1H.als
COMNT	single_pulse
DATIM	2020-11-18 21:45:05
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	10



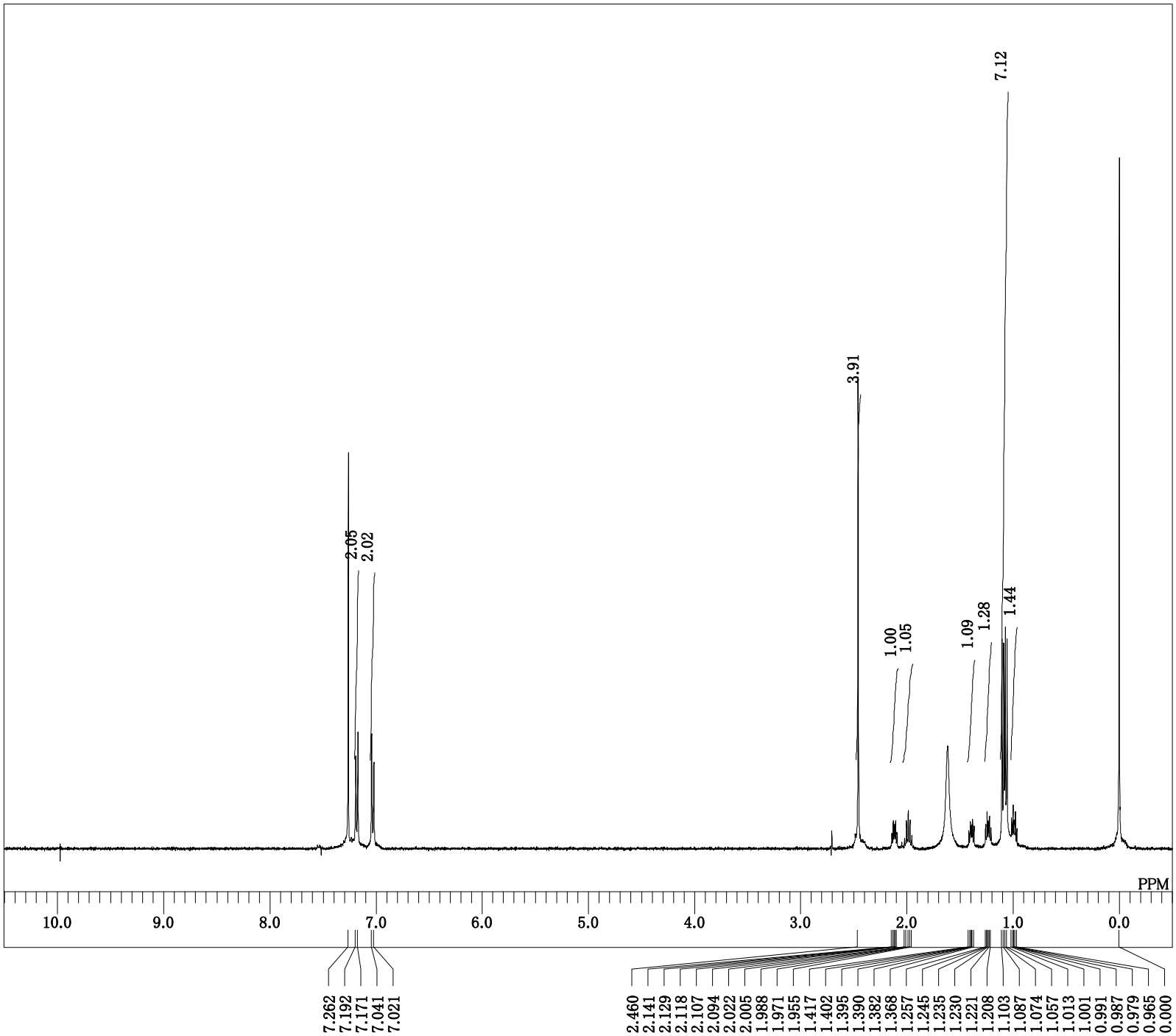
β-2a



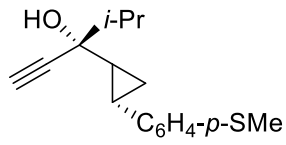
DFILE	beta_2a_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-18 21:47:20
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	256
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



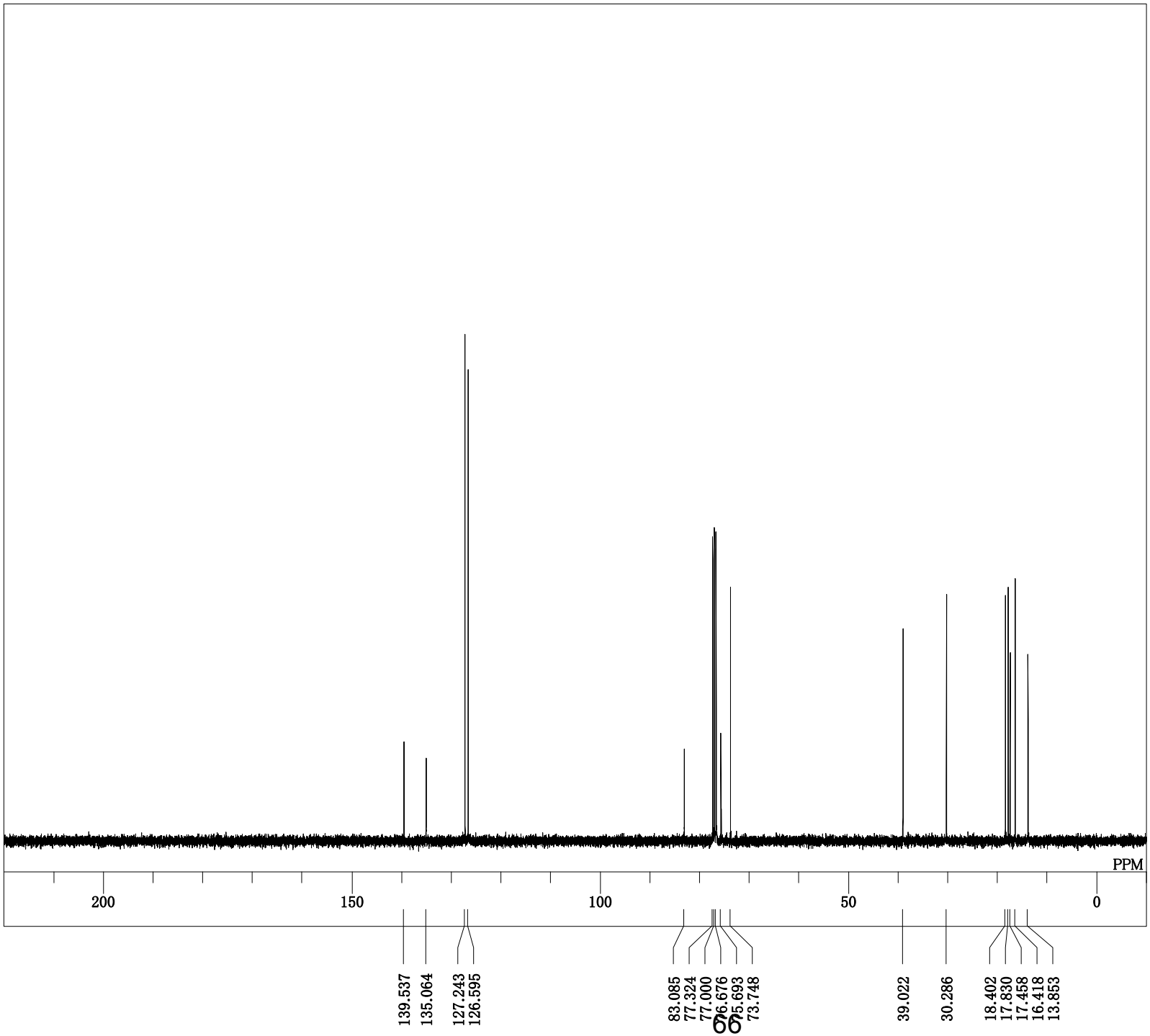
β-2a



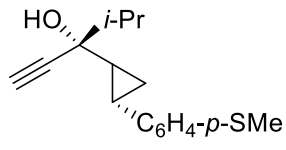
DFILE	alpha_2b_1H.als
COMNT	
DATIM	Fri Aug 30 20:36:04 2019
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	2.0000 sec
PW1	6.60 usec
IRNUC	1H
CTEMP	6348.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	24



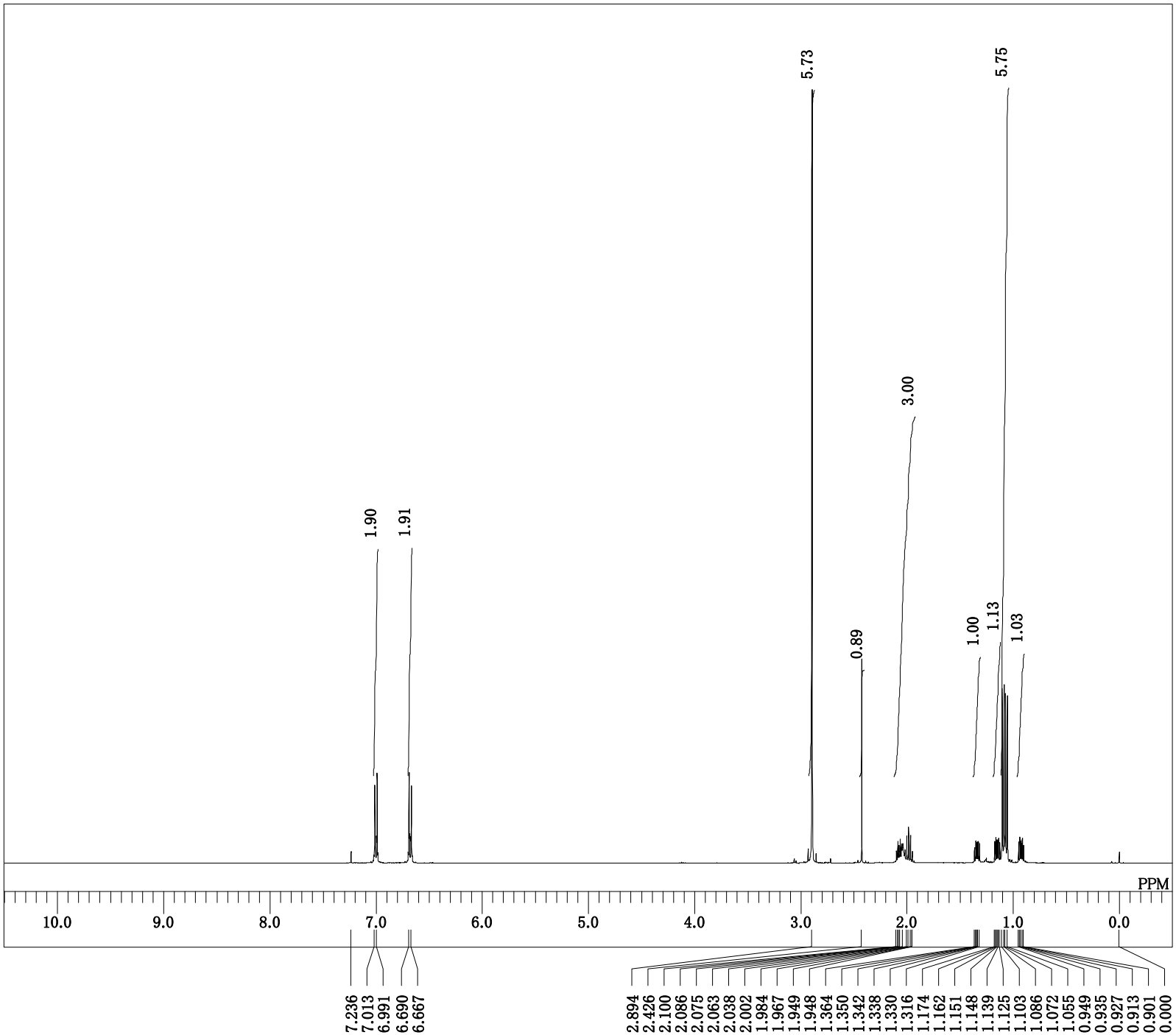
α-2b



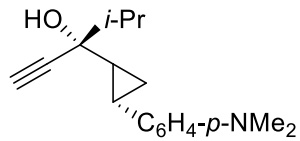
DFILE	alpha_2b_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-01-31 18:09:09
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.17 Hz
SCANS	500
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.17 usec
IRNUC	1H
CTEMP	20.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	44



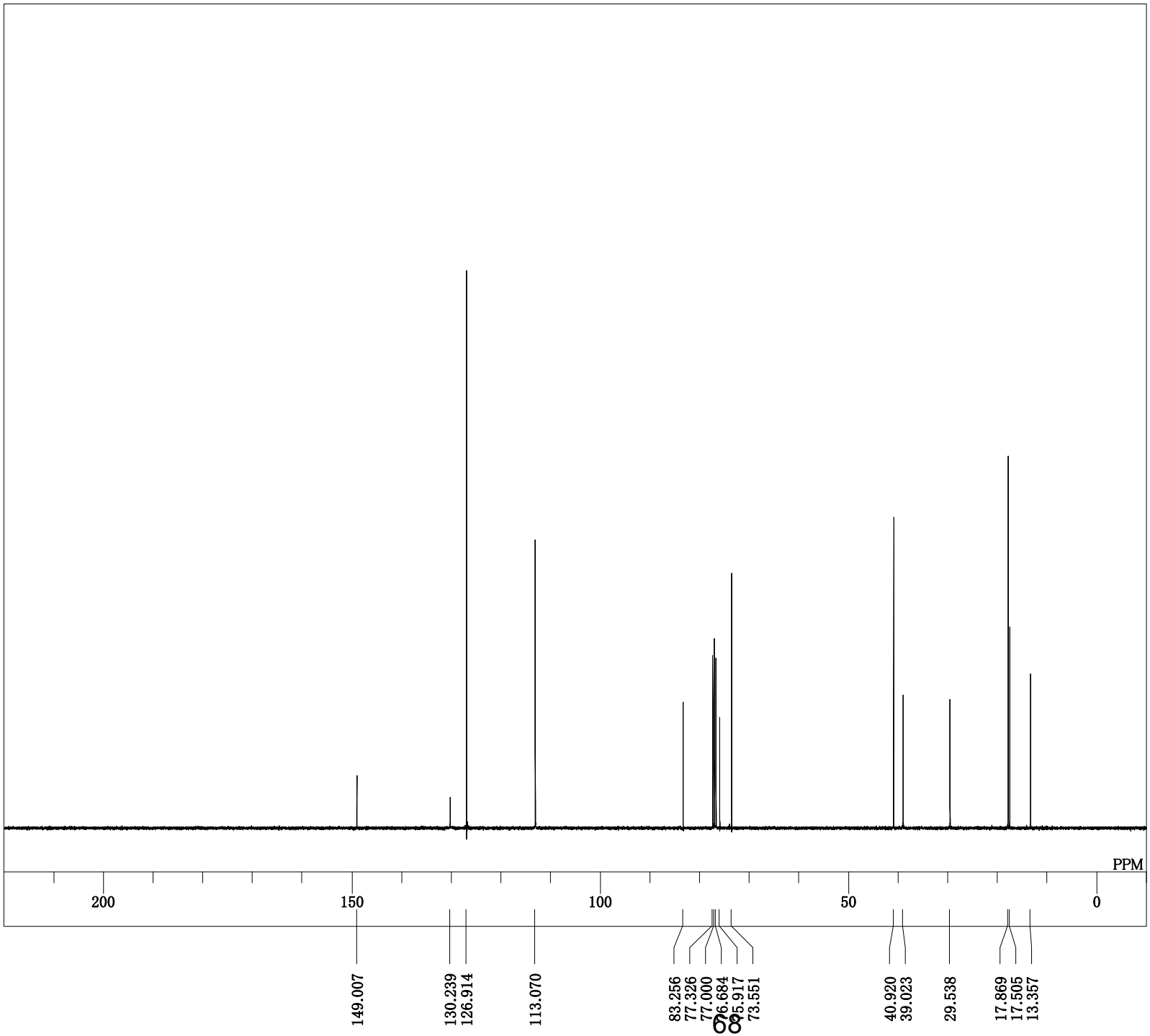
α-2b



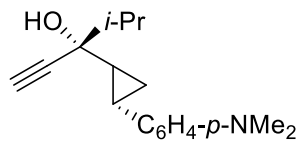
DFILE	alpha_2c_1H.als
COMNT	single_pulse
DATIM	2020-11-08 20:06:57
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	22



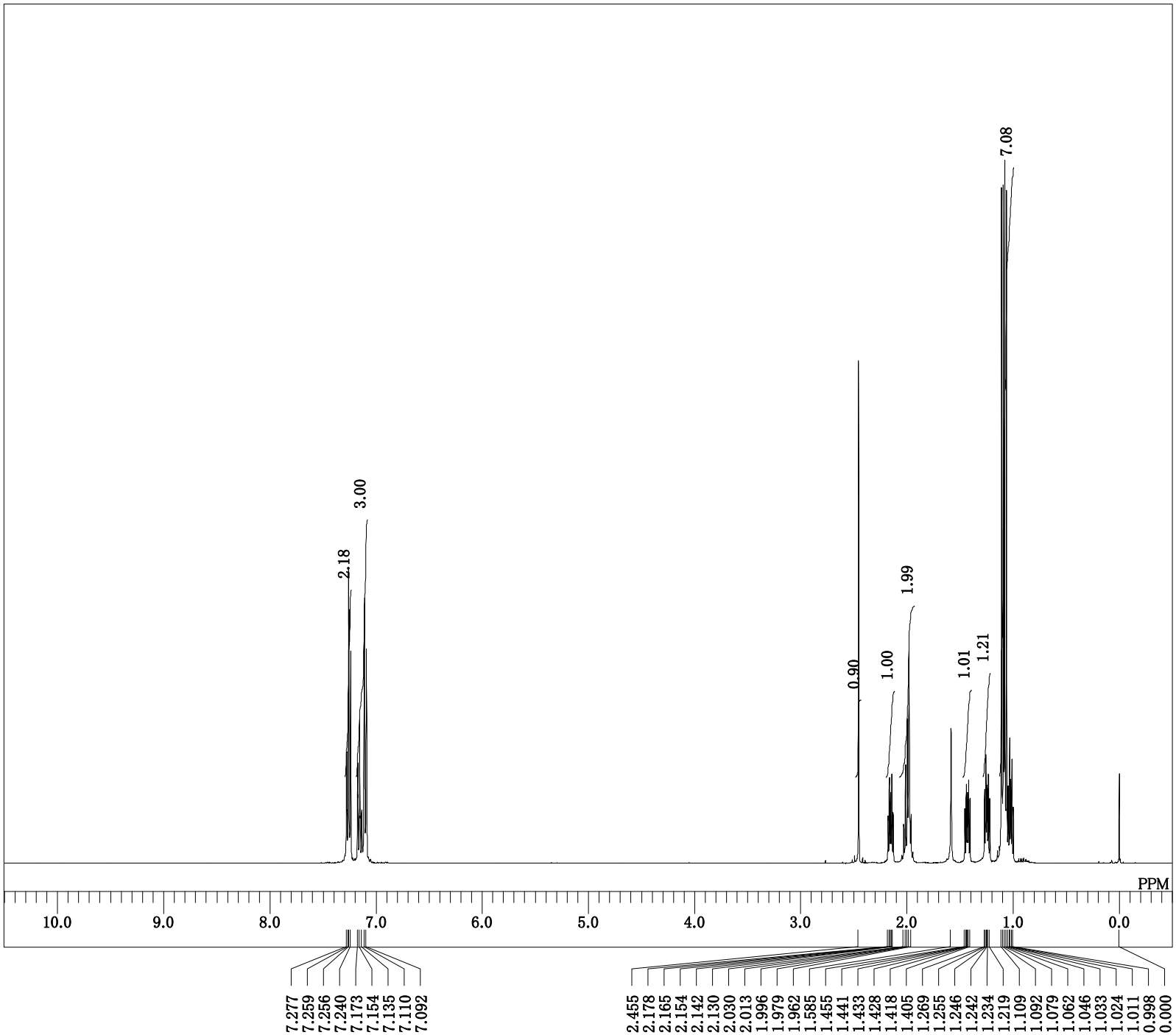
α-2c



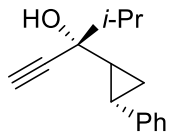
DFILE	alpha_2c_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-08 20:08:08
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	1024
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



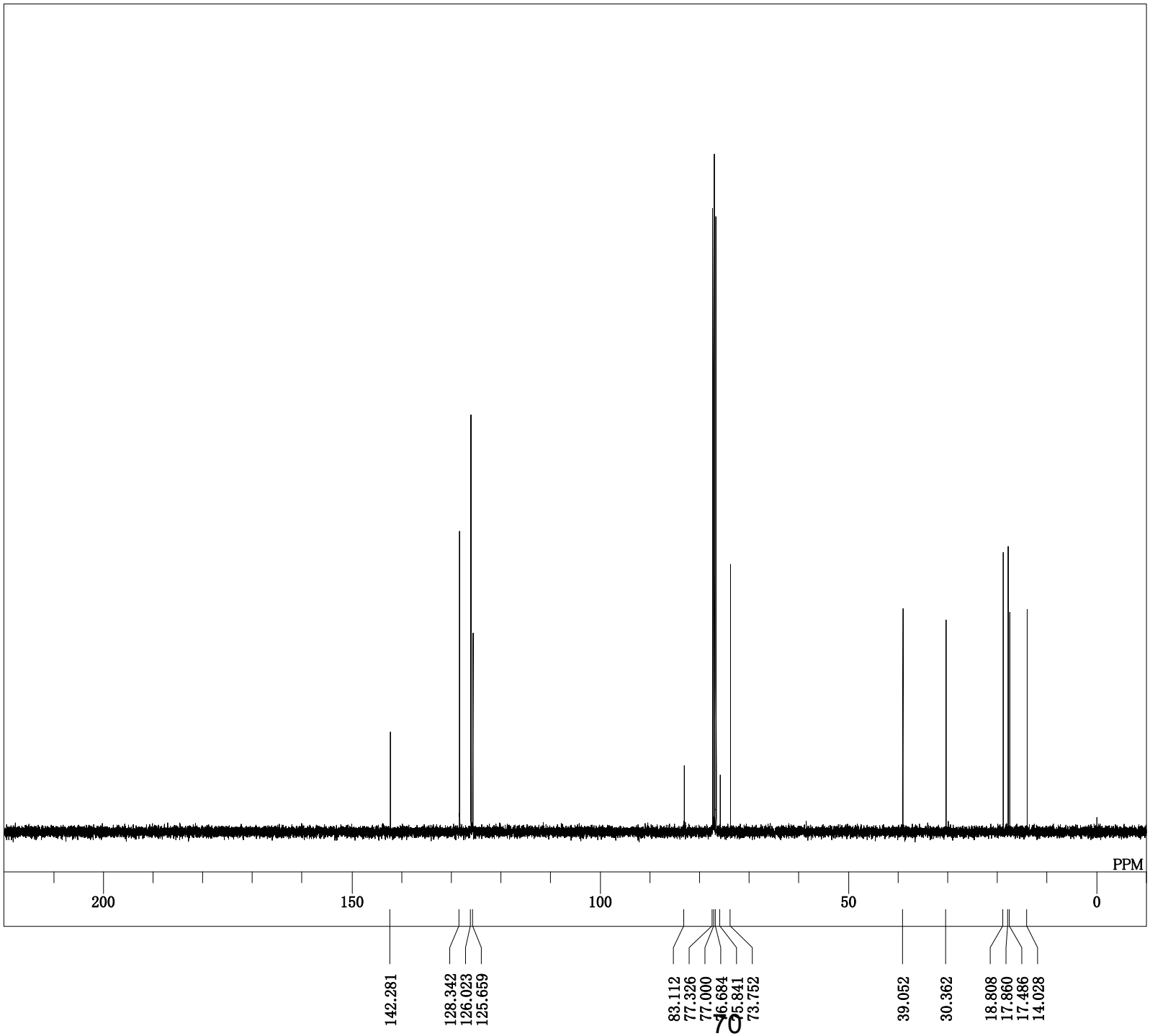
α-2c



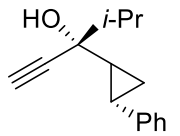
DFILE	alpha_2d_1H.als
COMNT	single_pulse
DATIM	2020-10-28 19:55:00
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	32



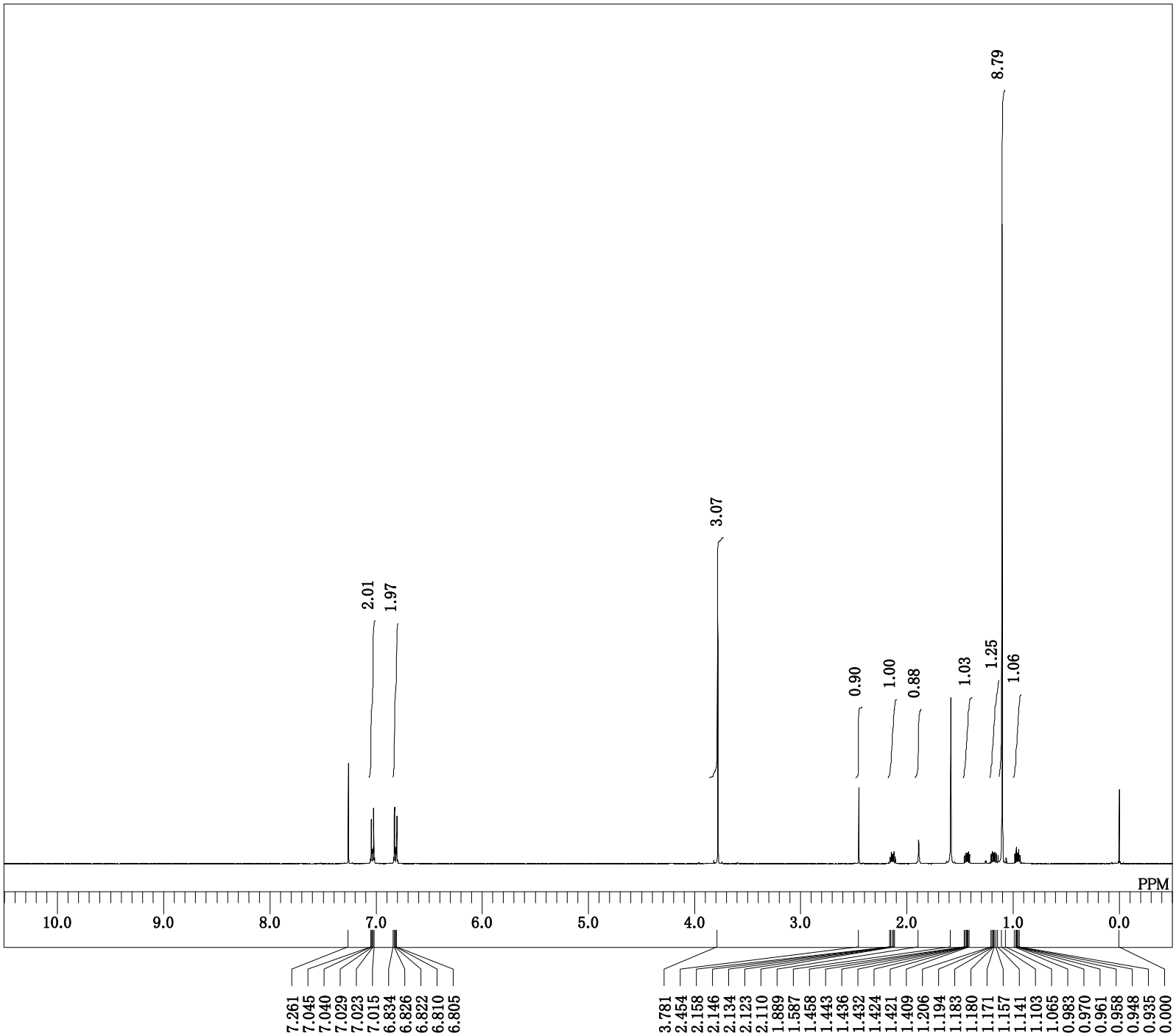
α−2**d**



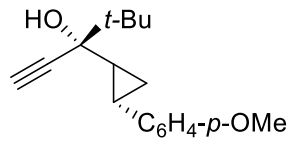
DFILE	alpha_2d_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-10-28 19:56:14
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	802
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



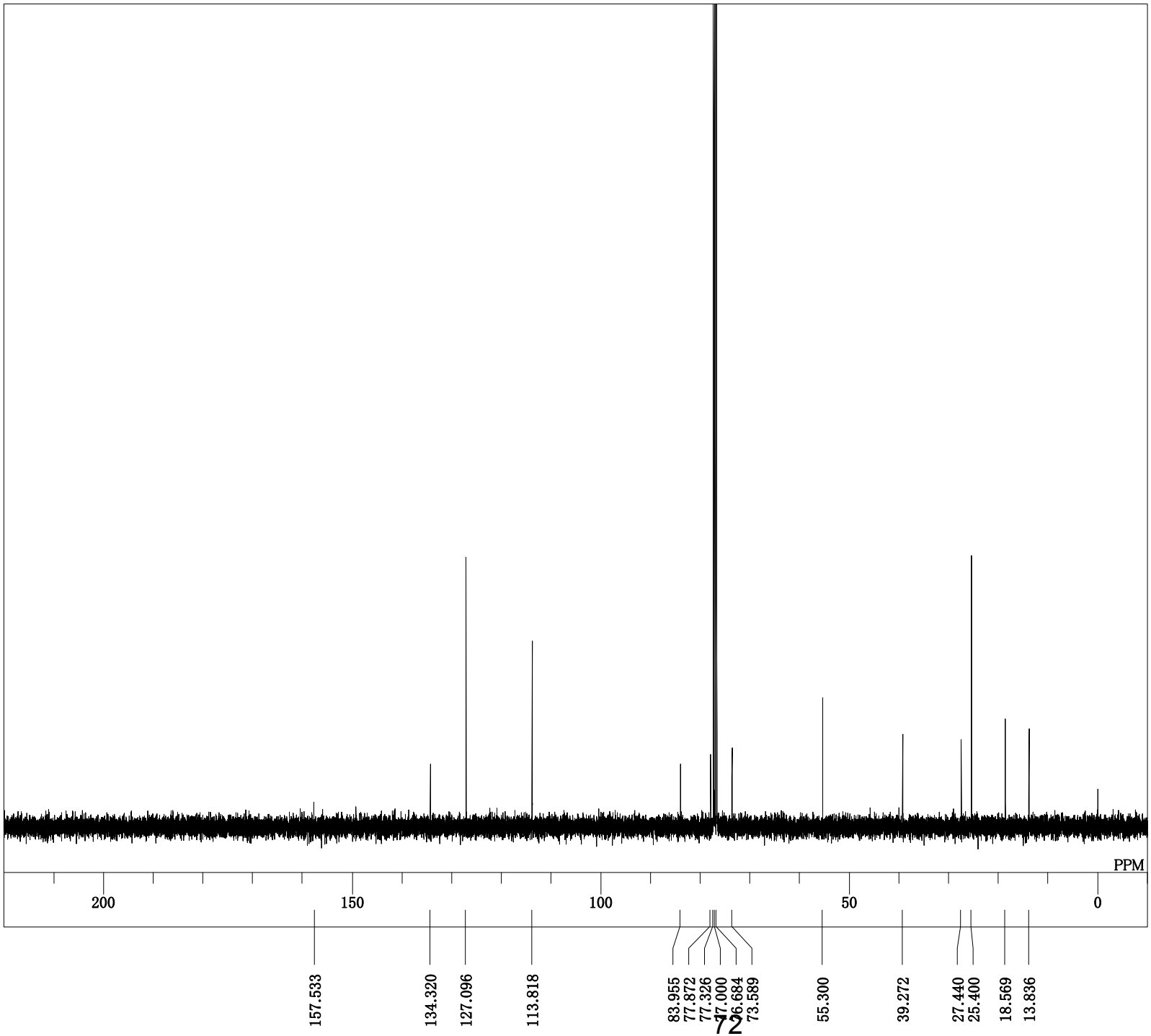
α−2**d**



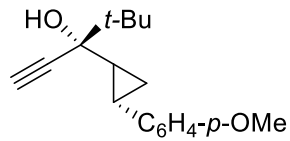
DFILE	alpha_2e_1H.als
COMNT	single_pulse
DATIM	2020-09-03 09:45:40
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40



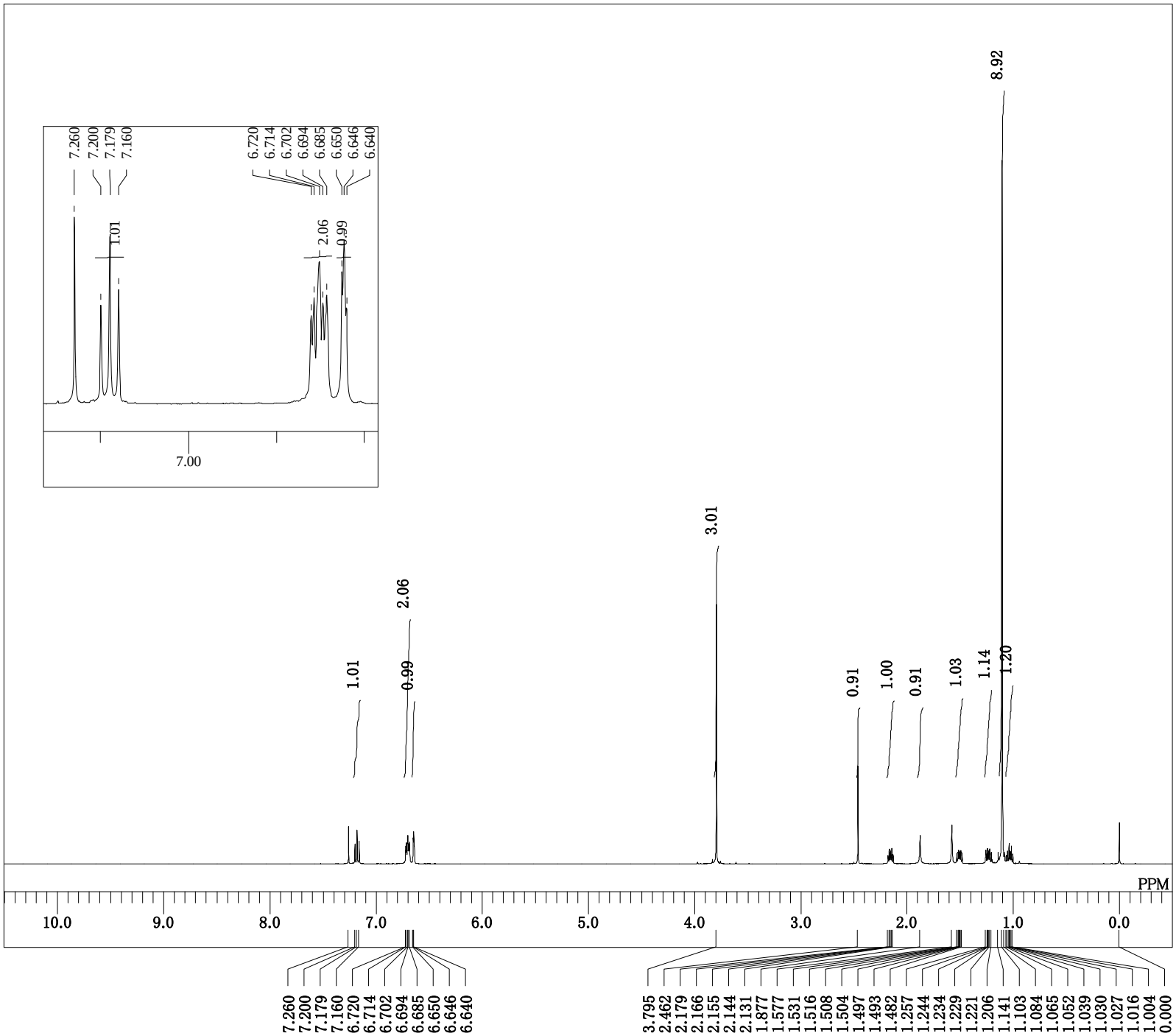
α–**2e**



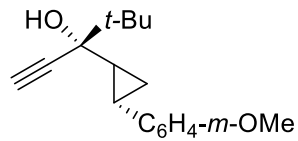
DFILE	alpha_2e_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-09-03 09:46:53
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	512
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



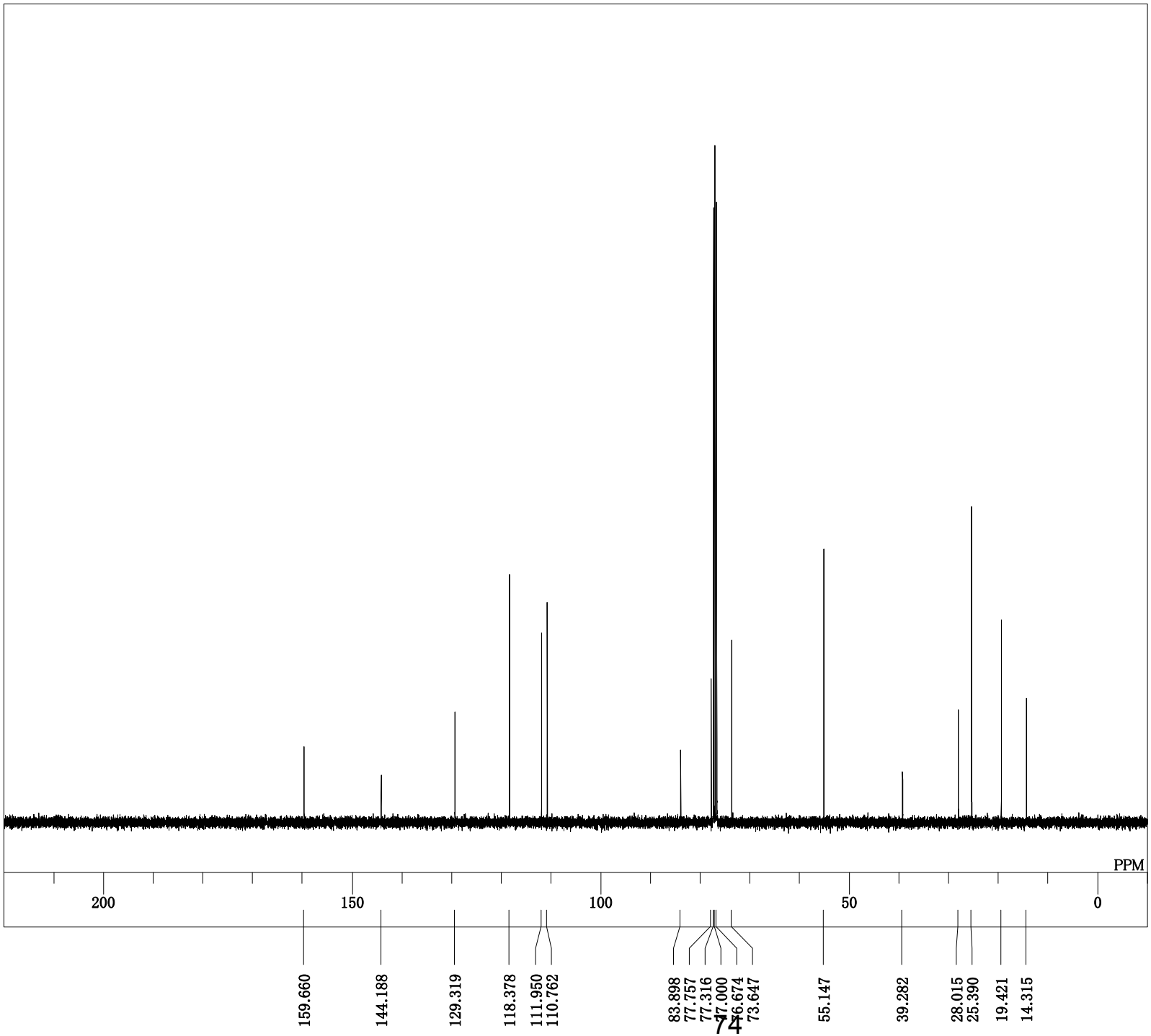
α–**2e**



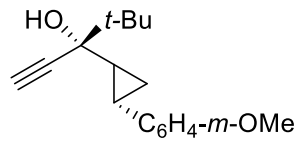
DFILE	alpha_2f_1H.als
COMNT	single_pulse
DATIM	2020-11-08 14:26:45
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	36



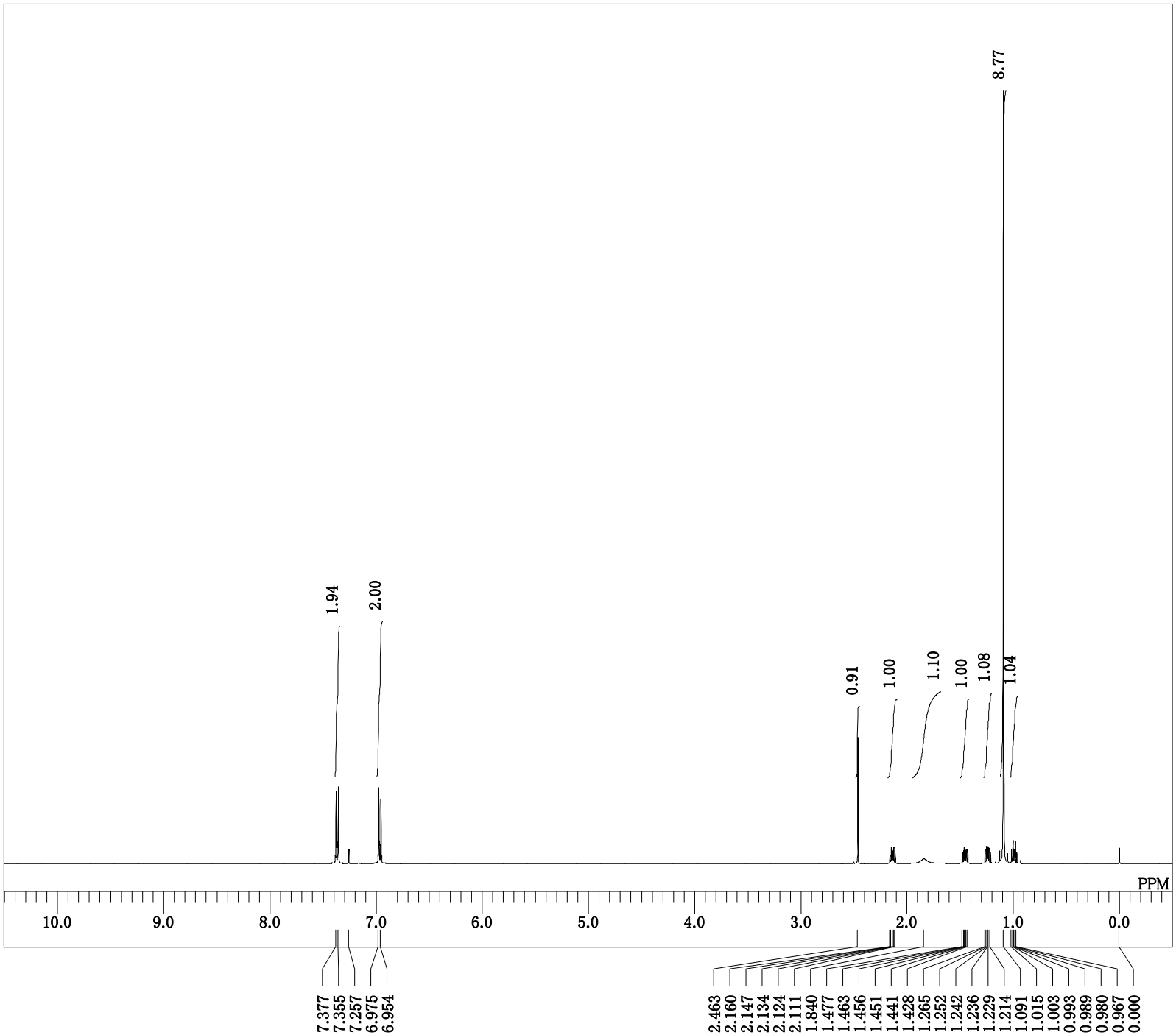
α–**2f**



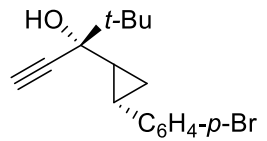
DFILE	alpha_2f_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-08 15:44:28
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	962
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



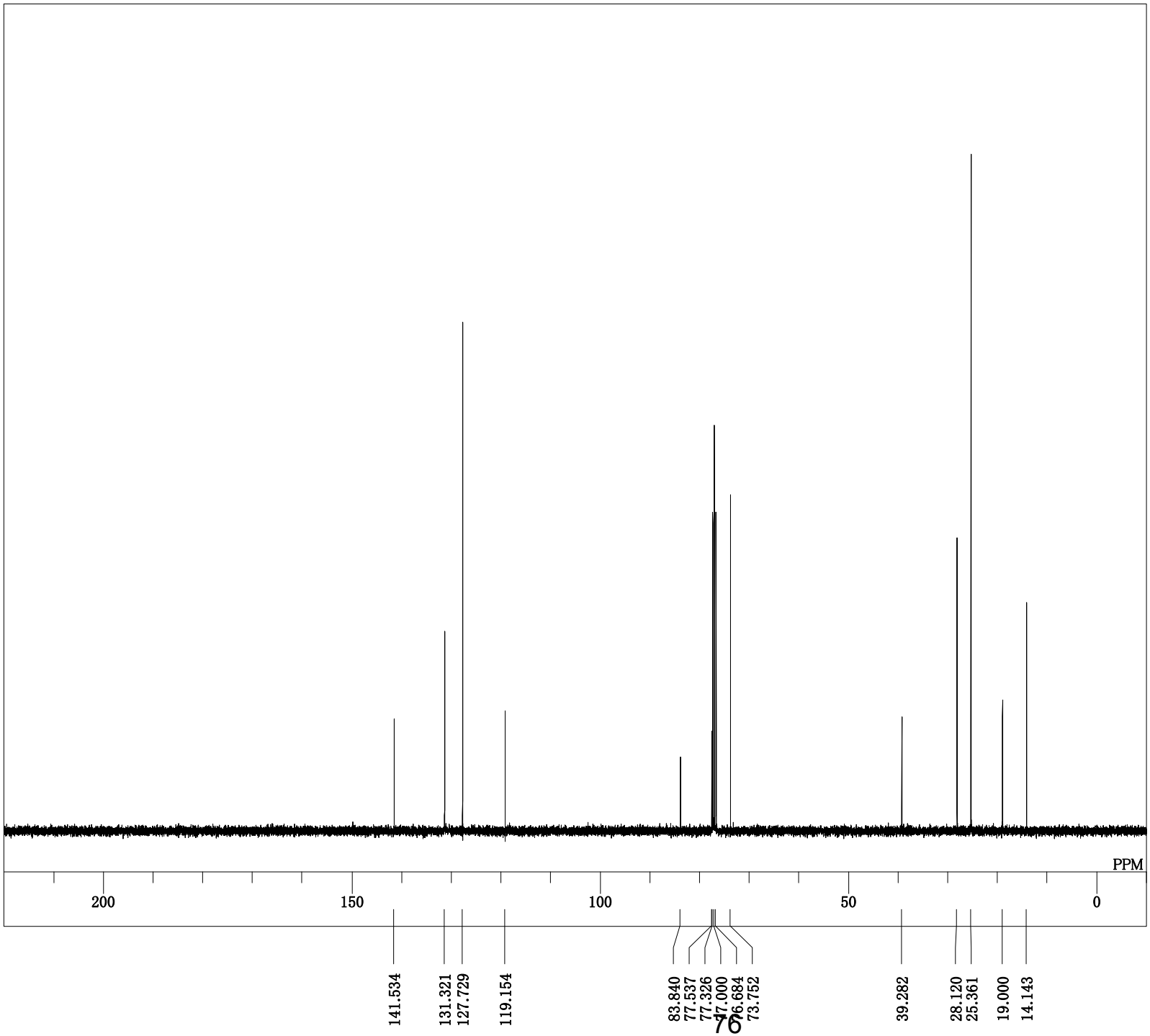
α–**2f**



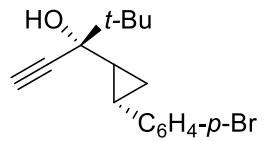
DFILE	alpha_2g_1H.als
COMNT	single_pulse
DATIM	2020-11-09 20:02:32
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	19.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	28



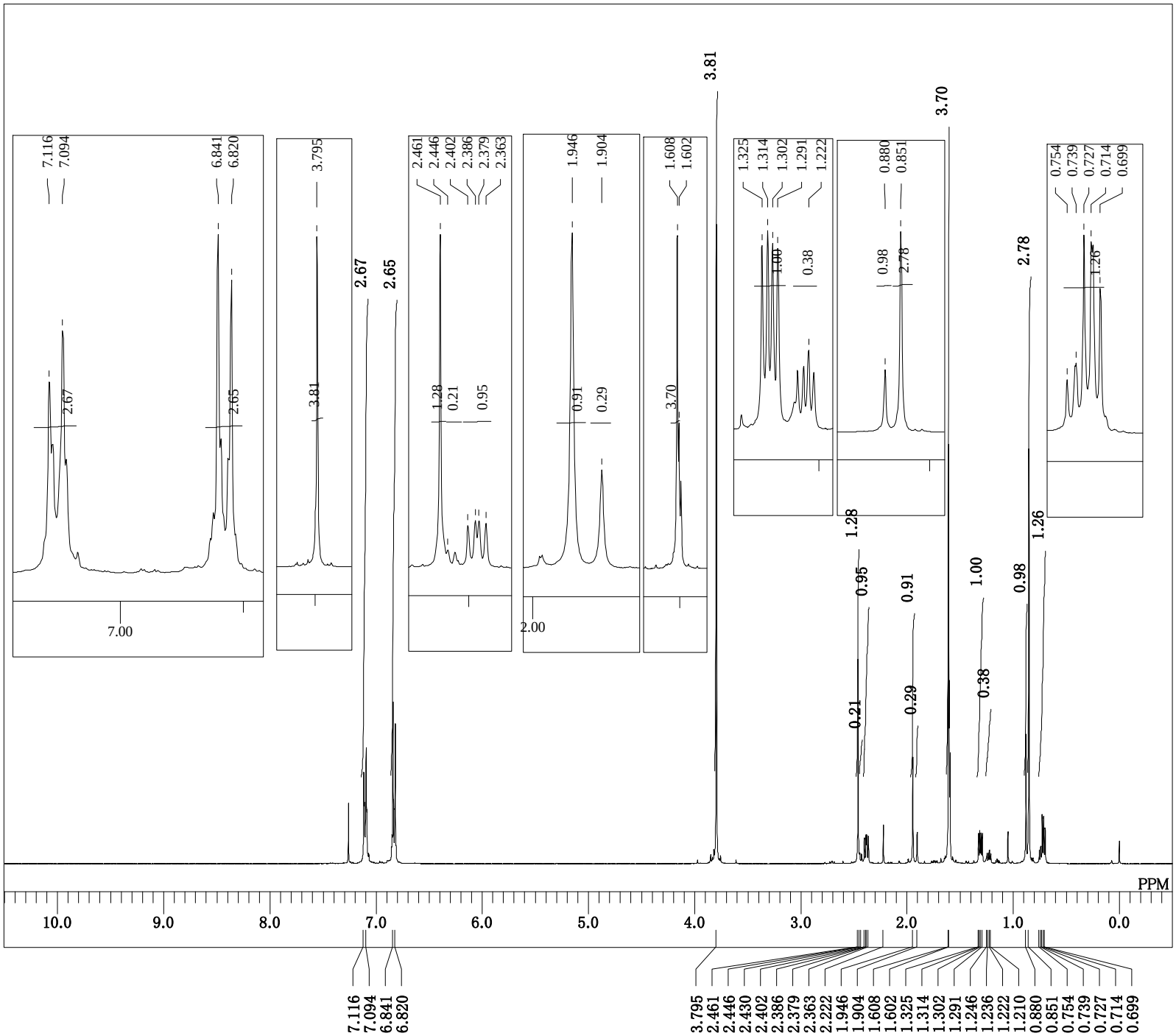
α-2g

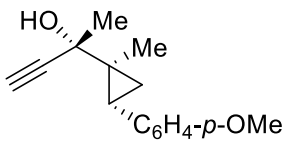


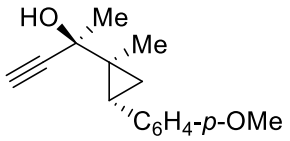
DFILE	alpha_2g_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-09 20:04:48
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	389
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



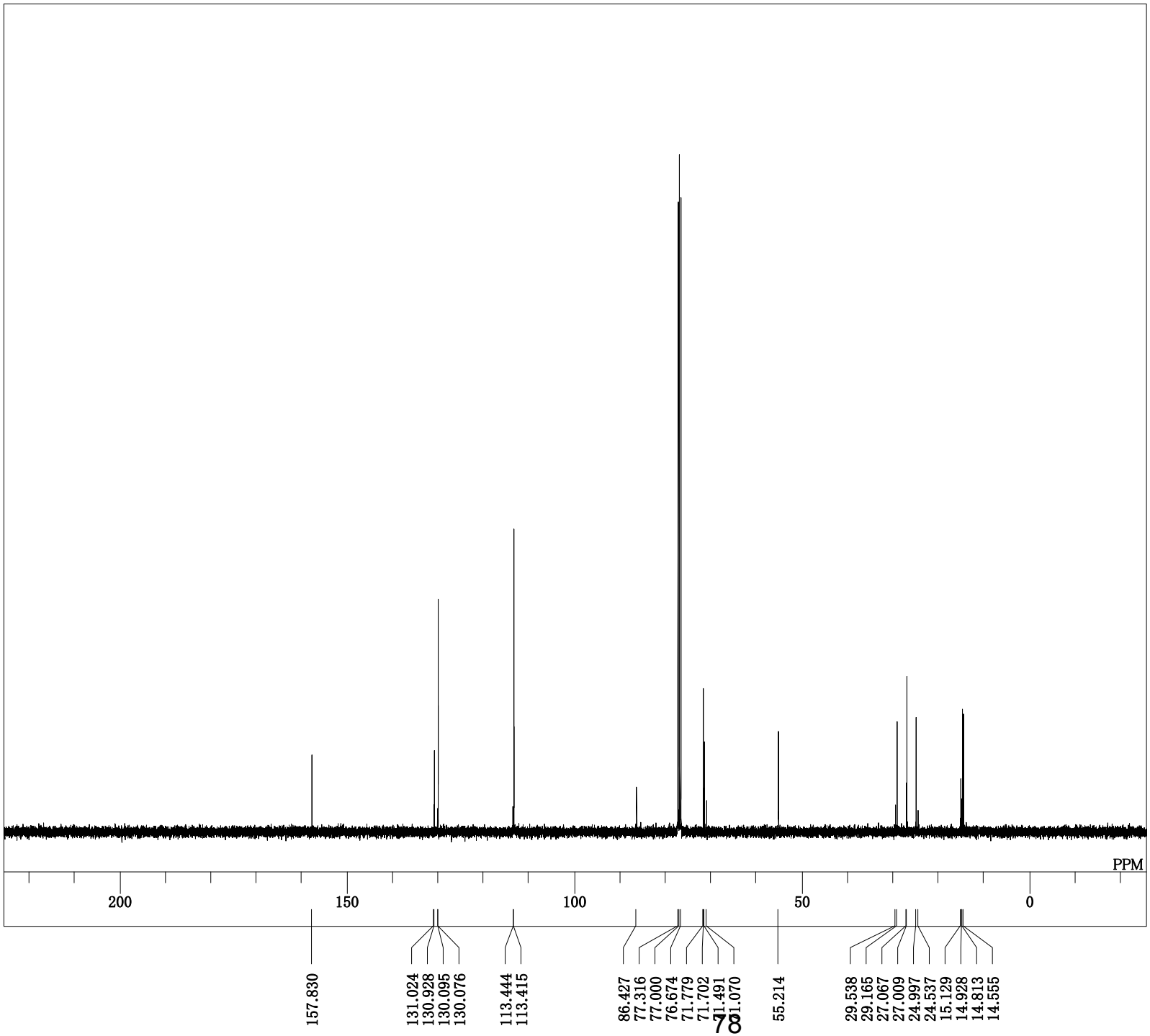
α-2g



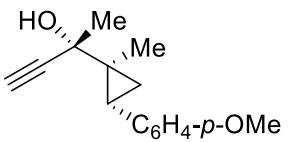


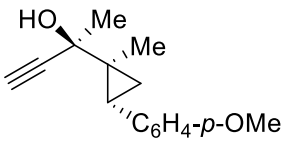


mixture of diastereomers 2h

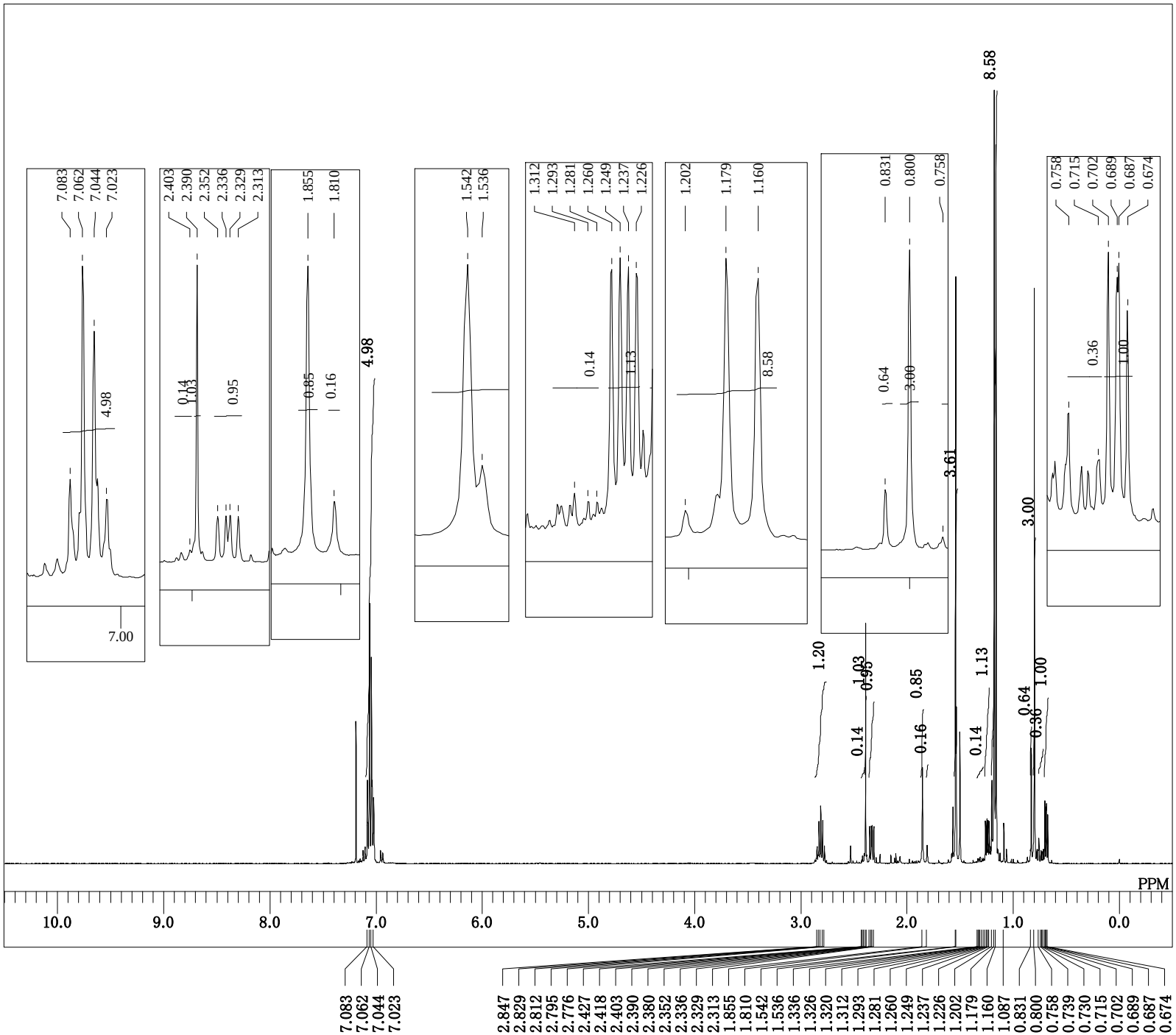


DFILE	2h_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-23 15:44:40
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	1024
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50

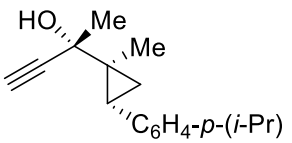


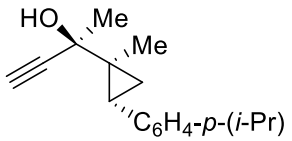


mixture of diastereomers 2h

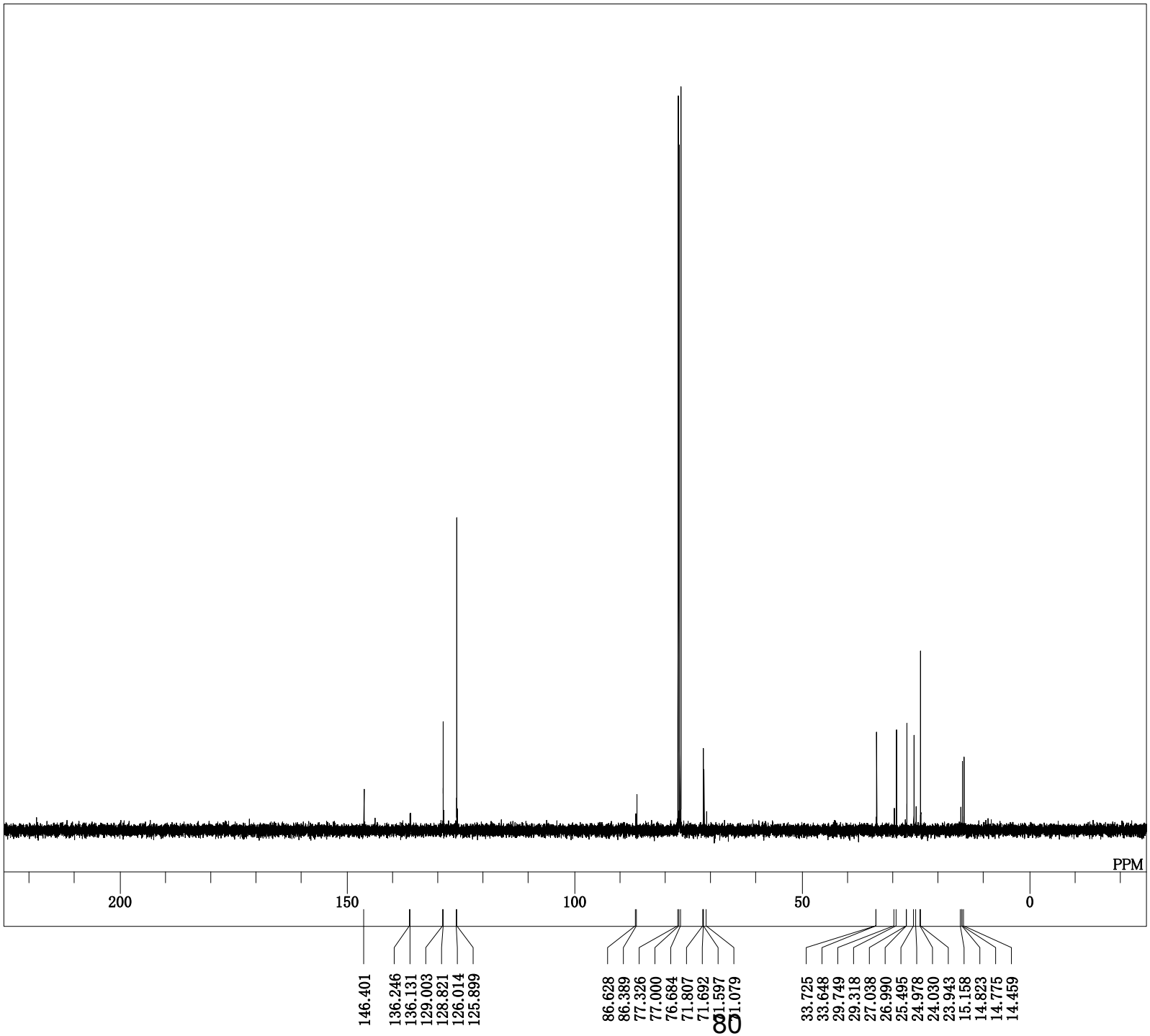


DFILE	2i_1H.als
COMNT	single_pulse
DATIM	2020-12-13 10:11:21
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	18.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	38

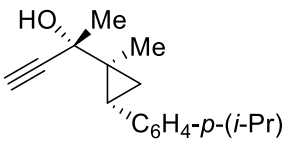


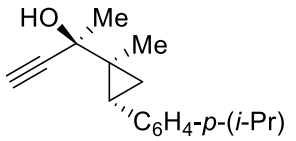


mixture of diastereomers 2i

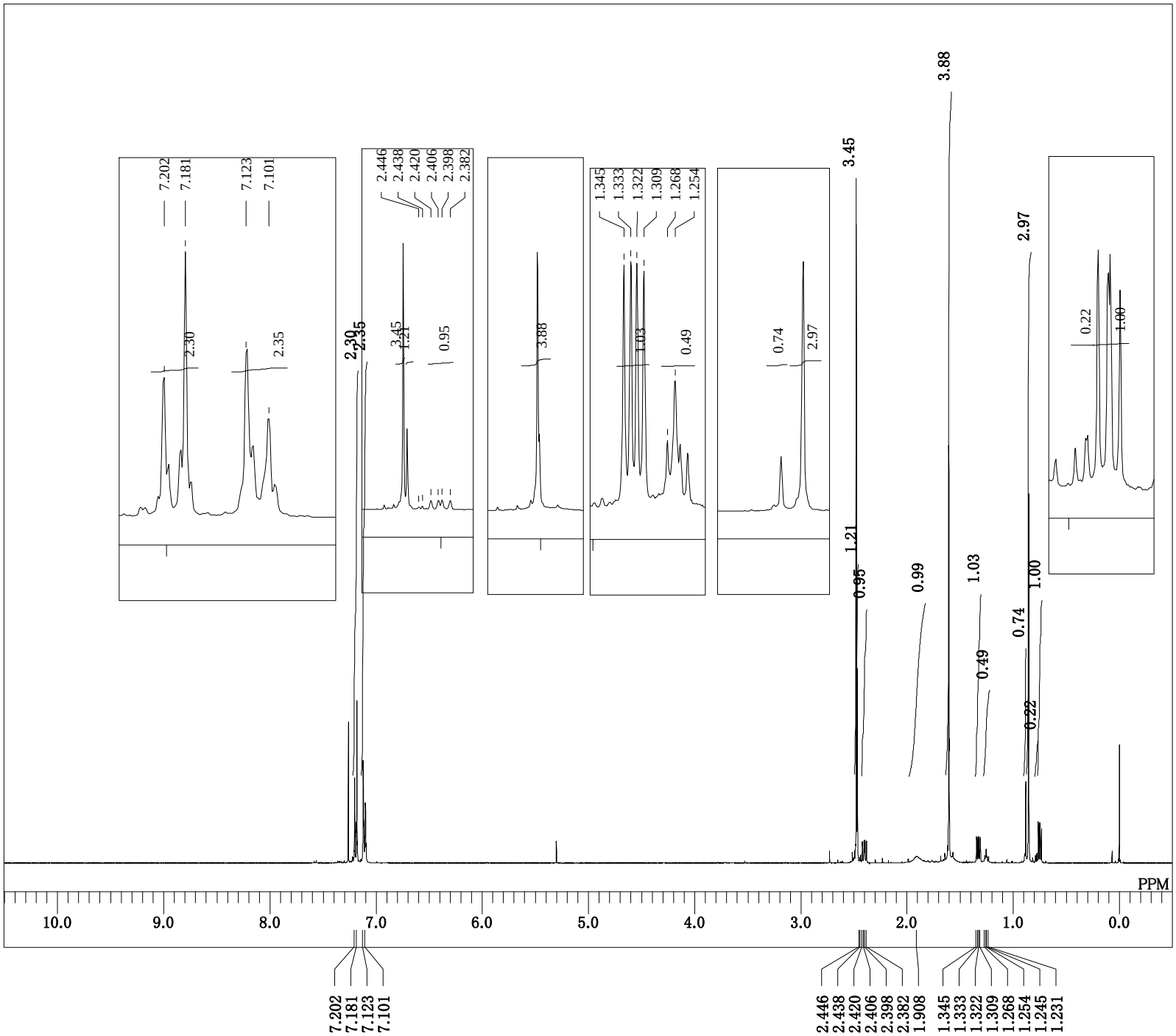


DFILE	2i_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-12-13 10:12:34
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	1024
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	18.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50

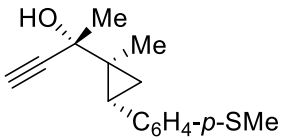


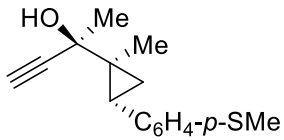


mixture of diastereomers 2i

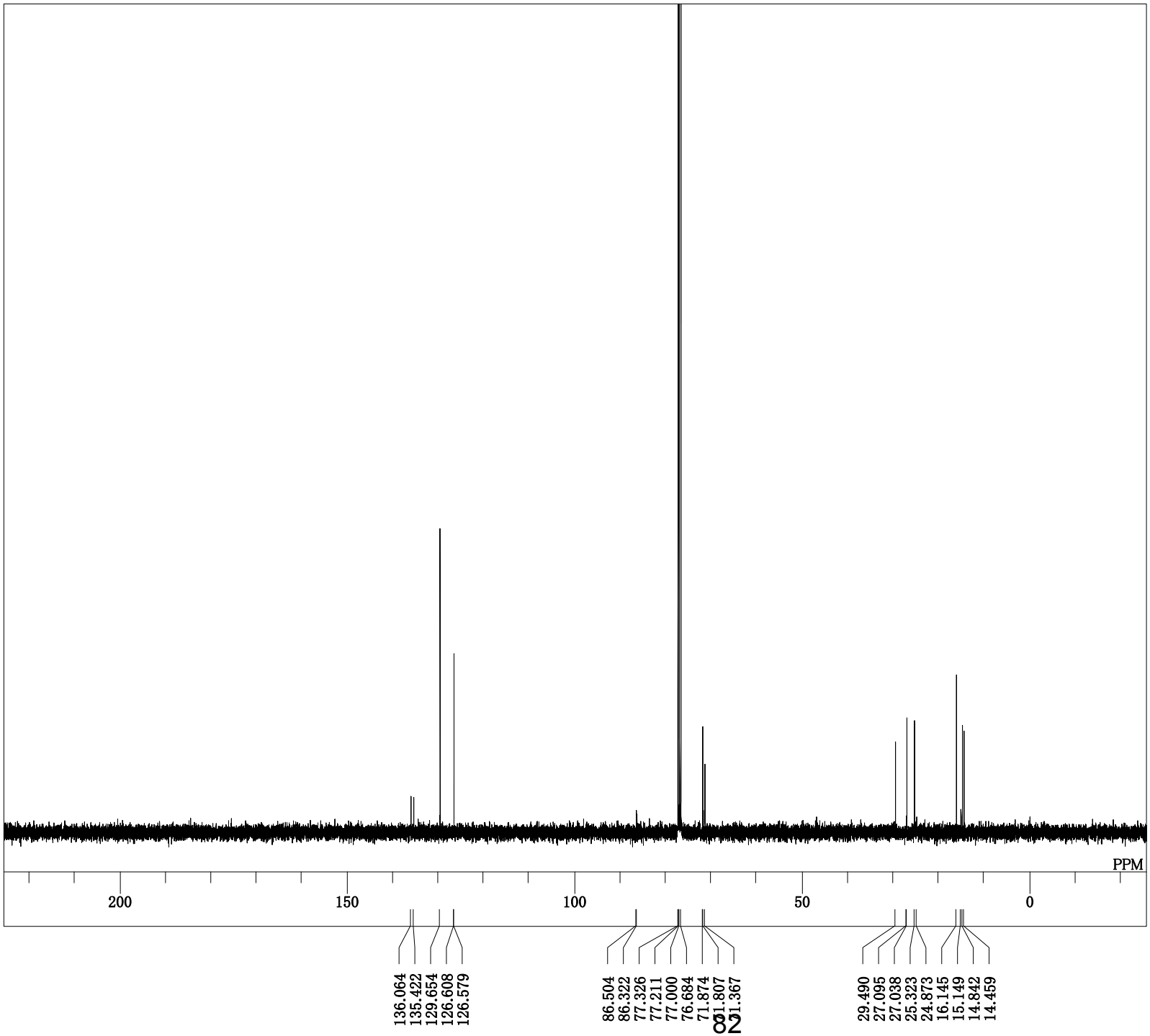


DFILE	2j_1H.als
COMNT	single_pulse
DATIM	2020-12-13 12:20:19
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	19.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40

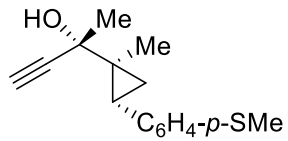


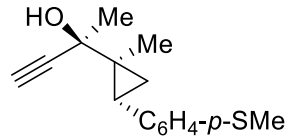


mixture of diastereomers 2j

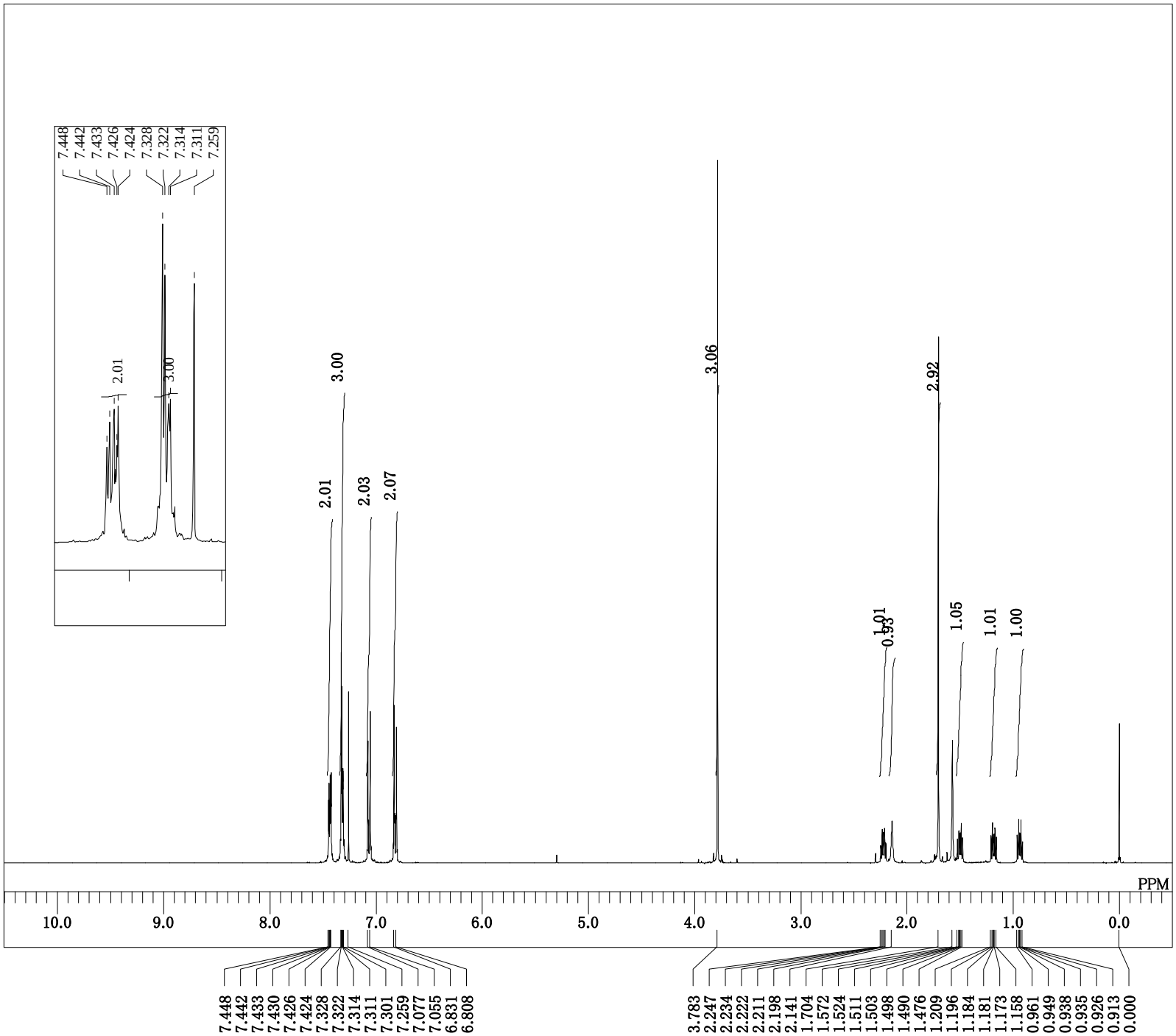


DFILE 2j_13C.als
COMNT single pulse decoupled gated NOE
DATIM 2020-12-13 12:21:31
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 25000.00 Hz
SCANS 1024
ACQTM 1.0486 sec
PD 2.0000 sec
PW1 3.59 usec
IRNUC 1H
CTEMP 18.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50

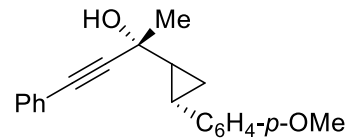




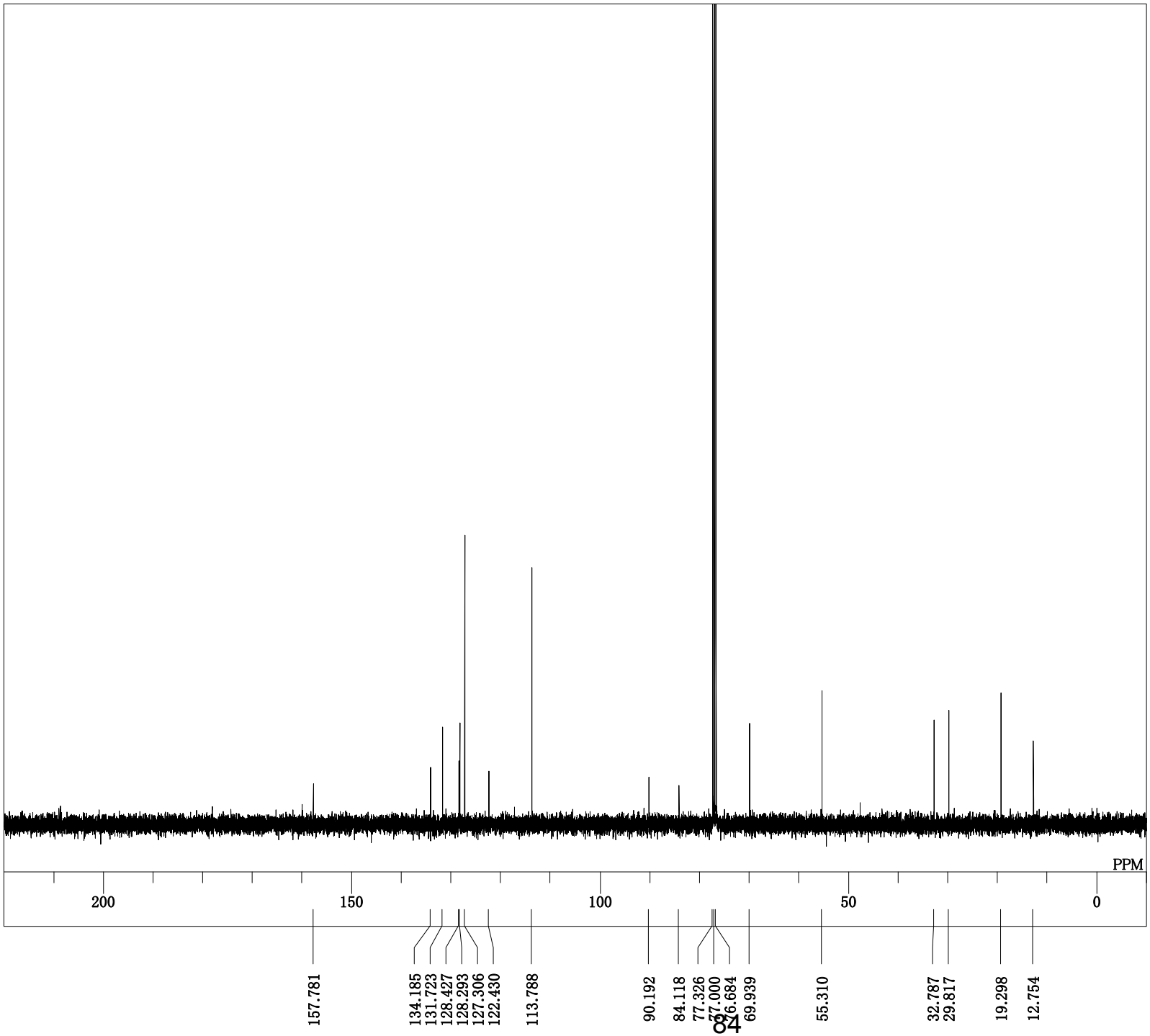
mixture of diastereomers 2j



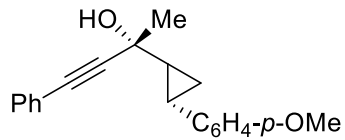
DFILE	alpha_2k_1H.als
COMNT	single_pulse
DATIM	2020-10-27 15:44:05
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40



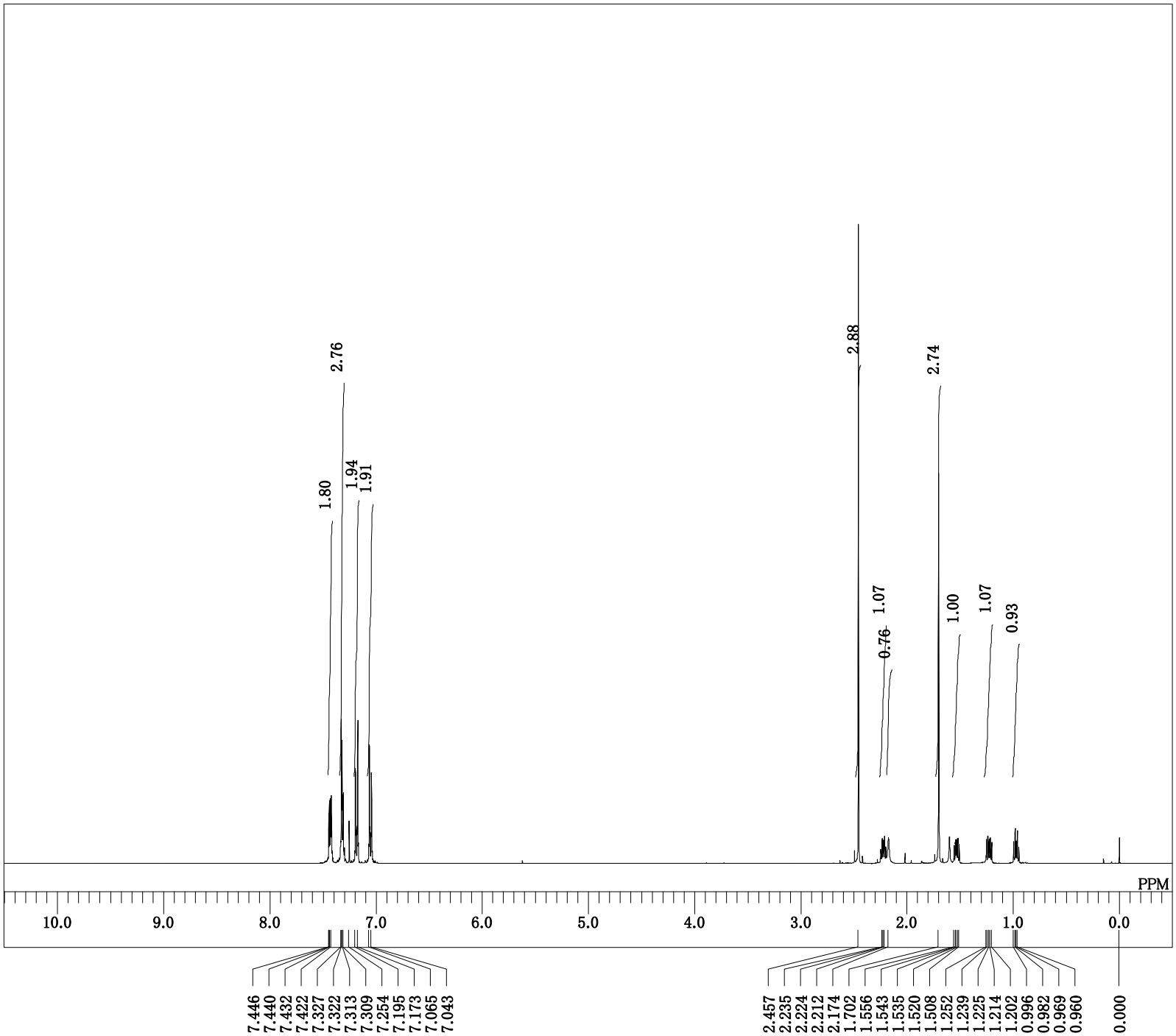
α–**2k**



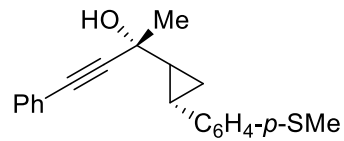
DFILE	alpha_2k_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-10-27 15:45:18
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	32767
FREQU	31250.00 Hz
SCANS	484
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



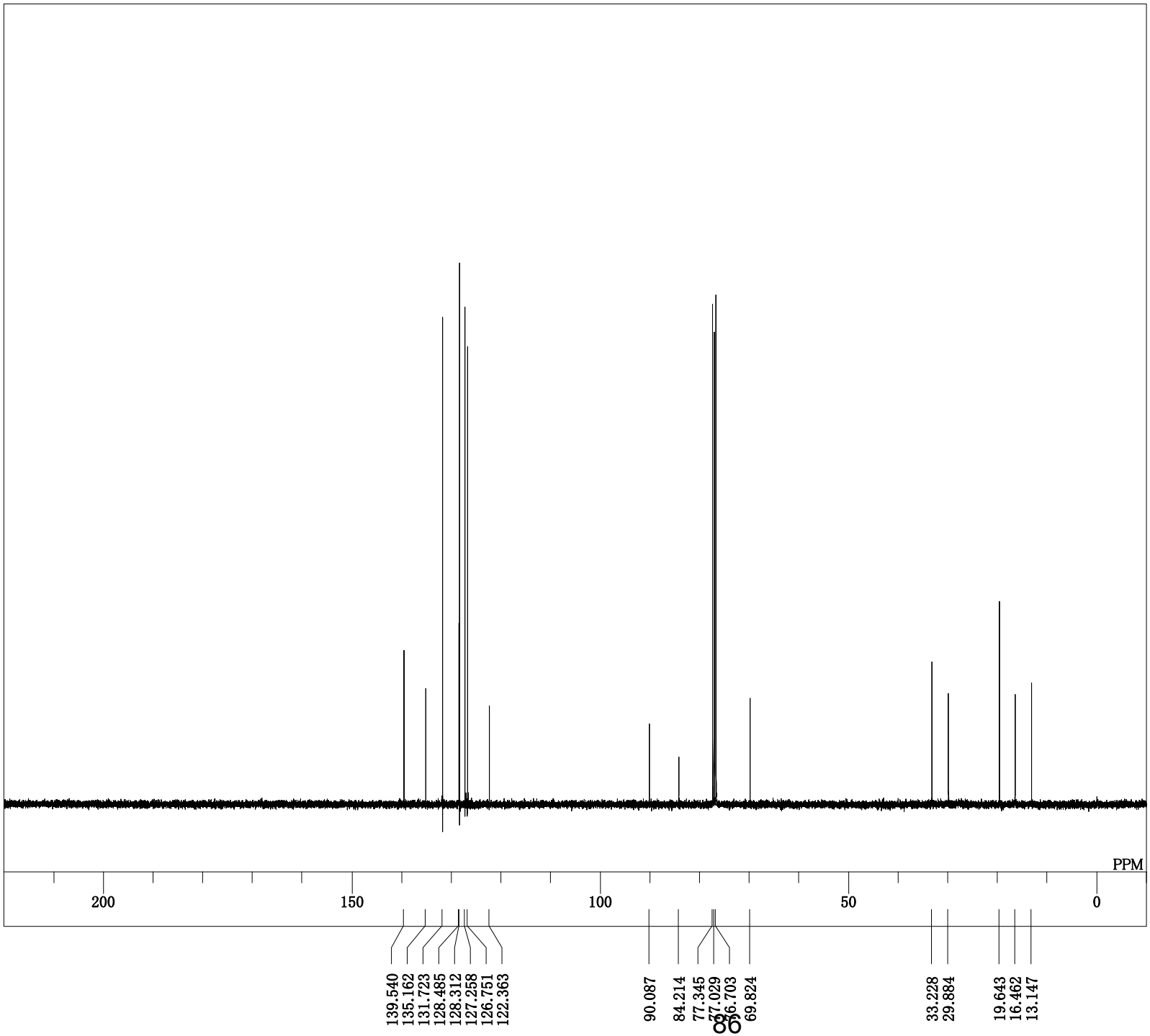
α–**2k**



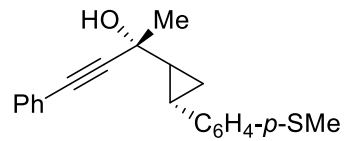
DFILE	alpha_2l_1H.als
COMNT	single_pulse
DATIM	2020-11-23 14:23:10
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	30



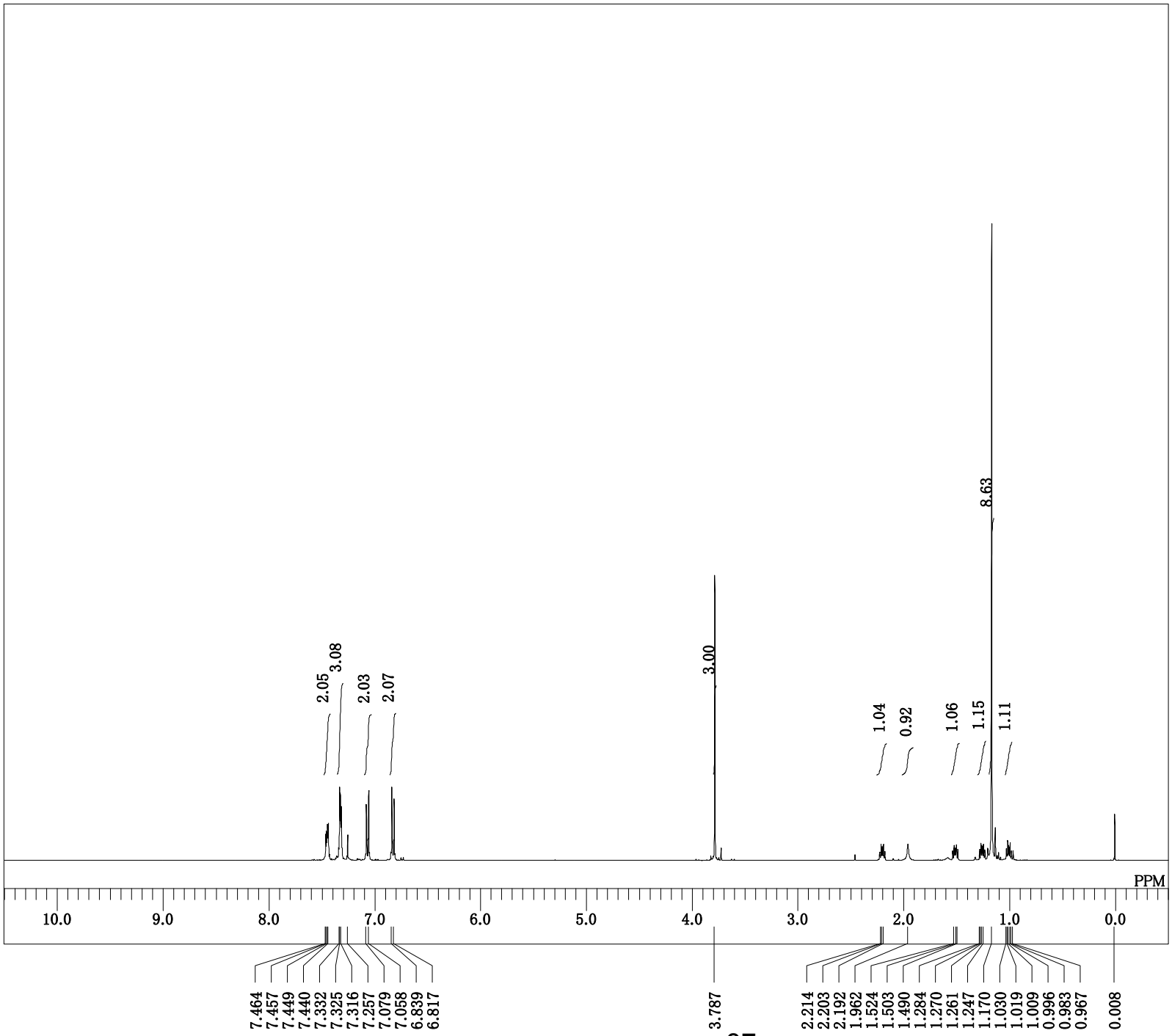
α–**2l**



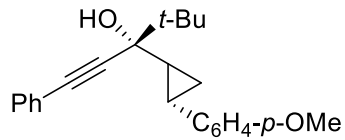
DFILE	alpha_2l_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-23 14:24:22
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	32767
FREQU	31250.00 Hz
SCANS	1024
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



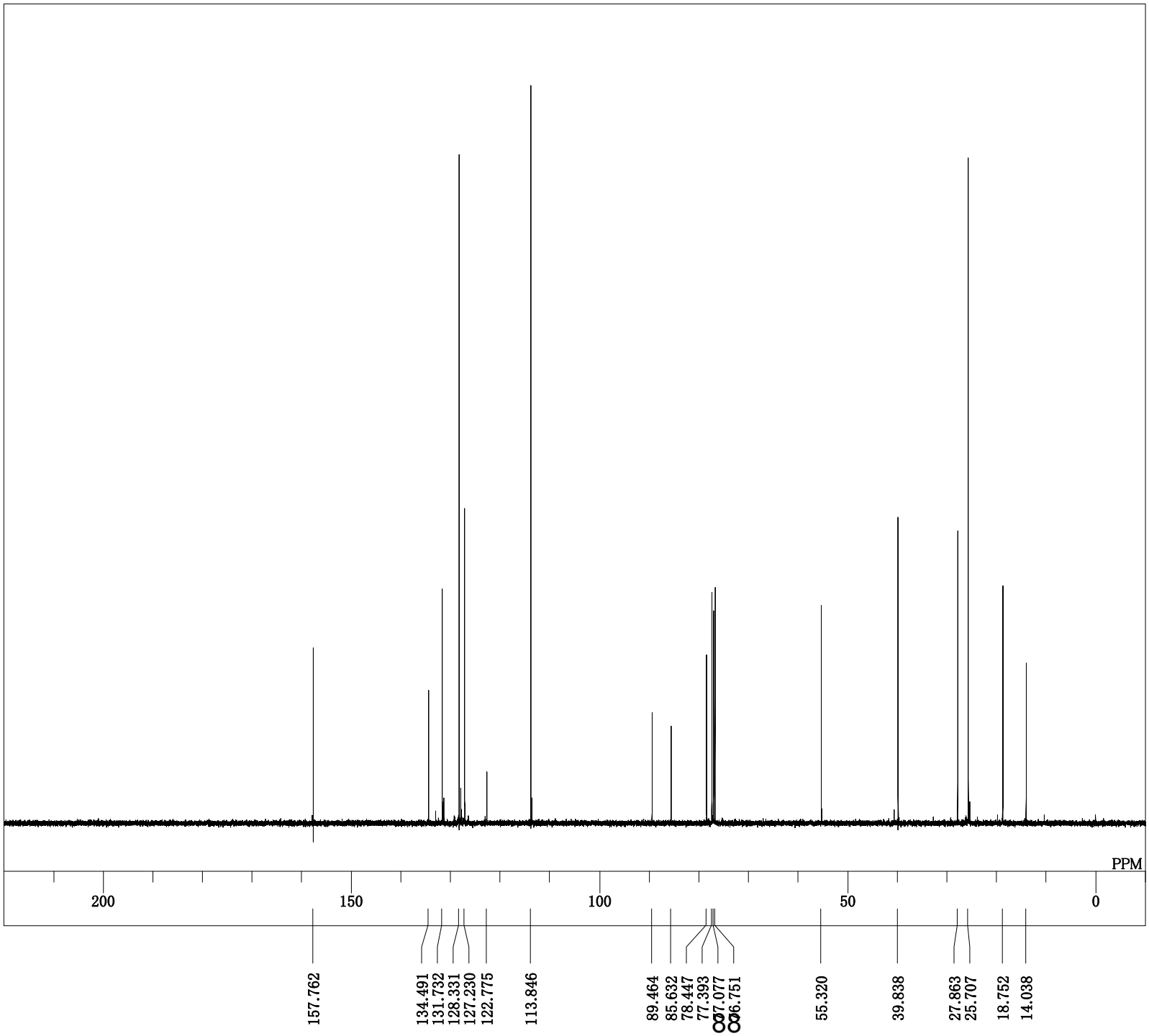
α–**2l**



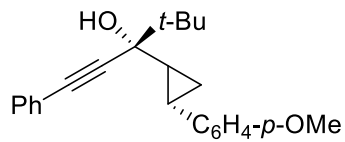
DFILE	alpha_2m_1H.als
COMNT	single_pulse
DATIM	2020-10-13 17:38:59
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	30



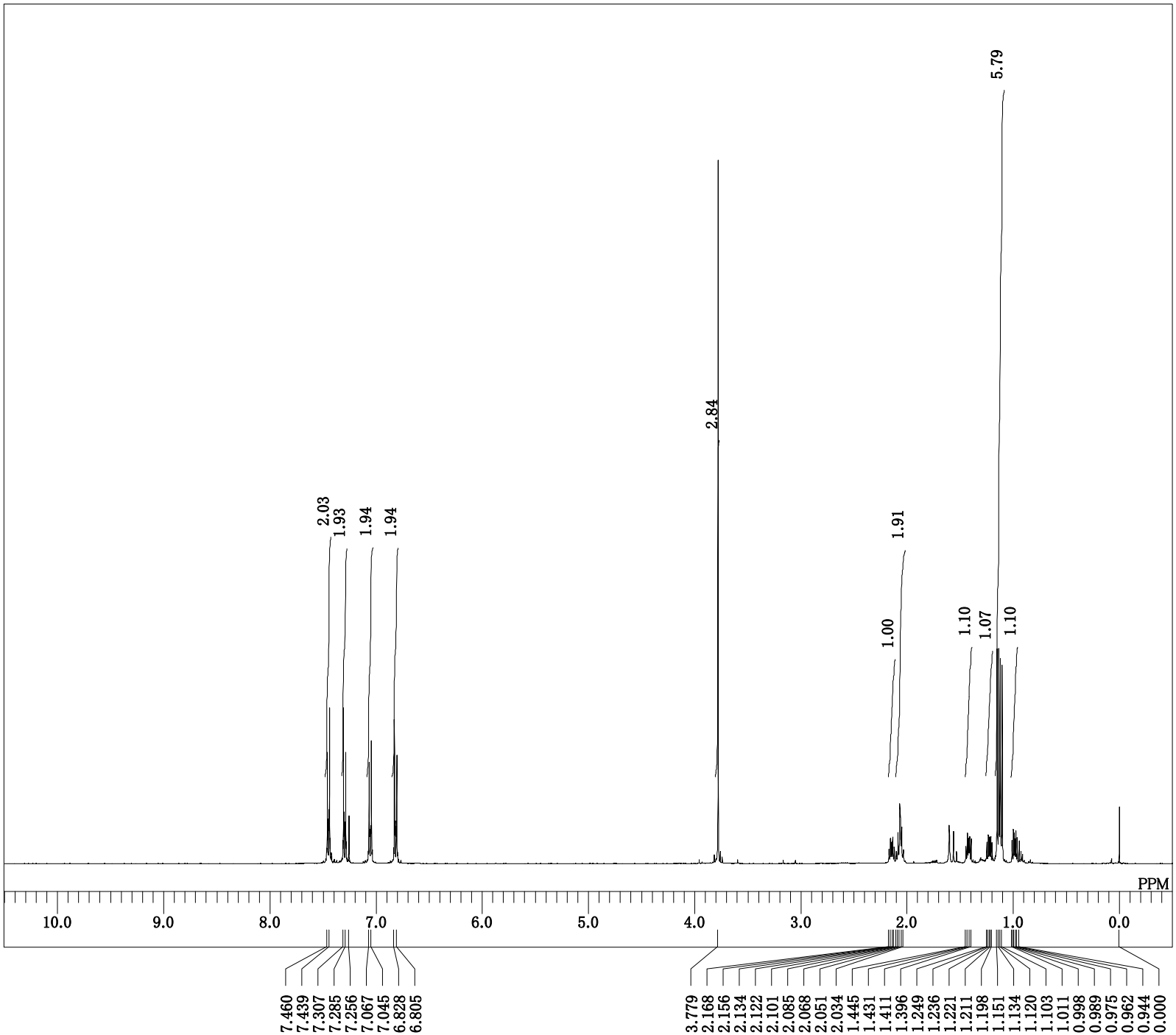
α-2m



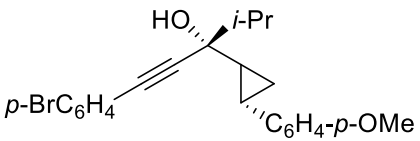
DFILE	alpha_2m_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-08 17:05:59
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	32767
FREQU	31250.00 Hz
SCANS	512
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



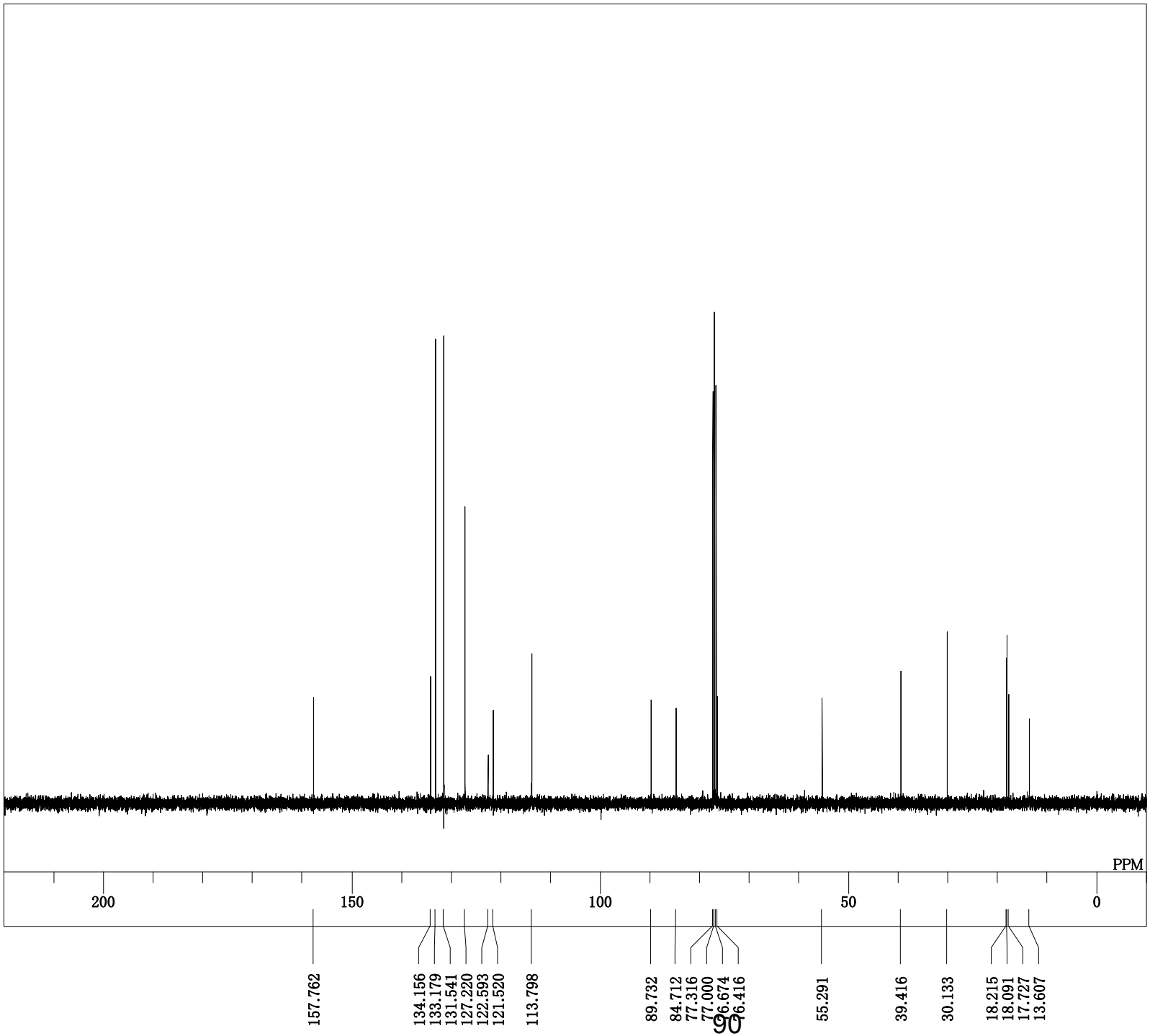
α-2m



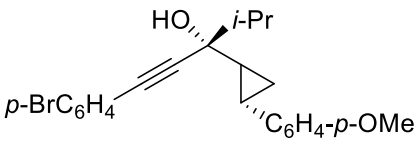
DFILE	alpha_2n_1H.als
COMNT	single_pulse
DATIM	2020-10-27 16:17:11
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	30



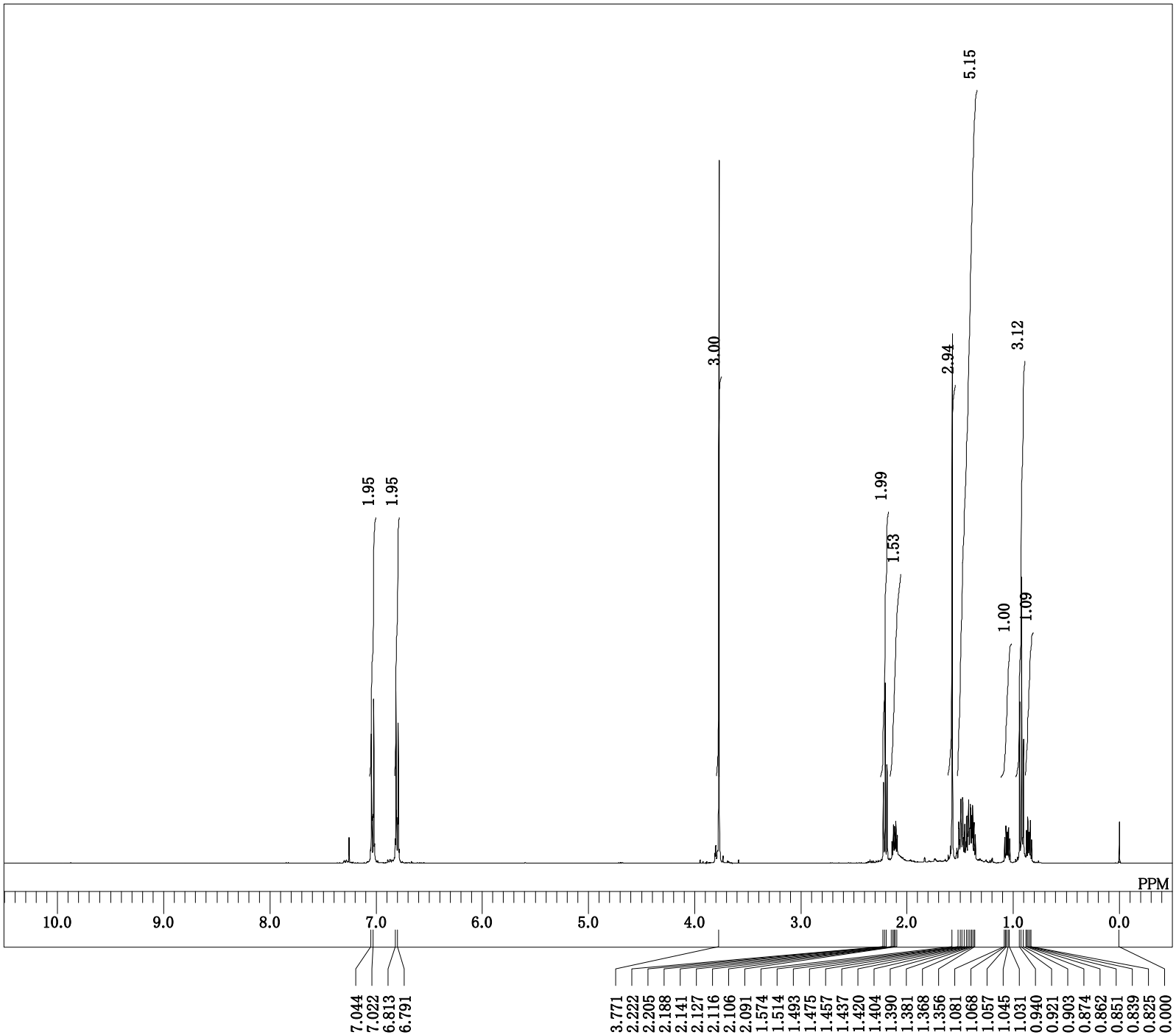
2n



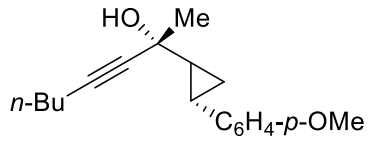
DFILE	alpha_2n_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-10-27 16:18:23
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	32767
FREQU	31250.00 Hz
SCANS	259
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



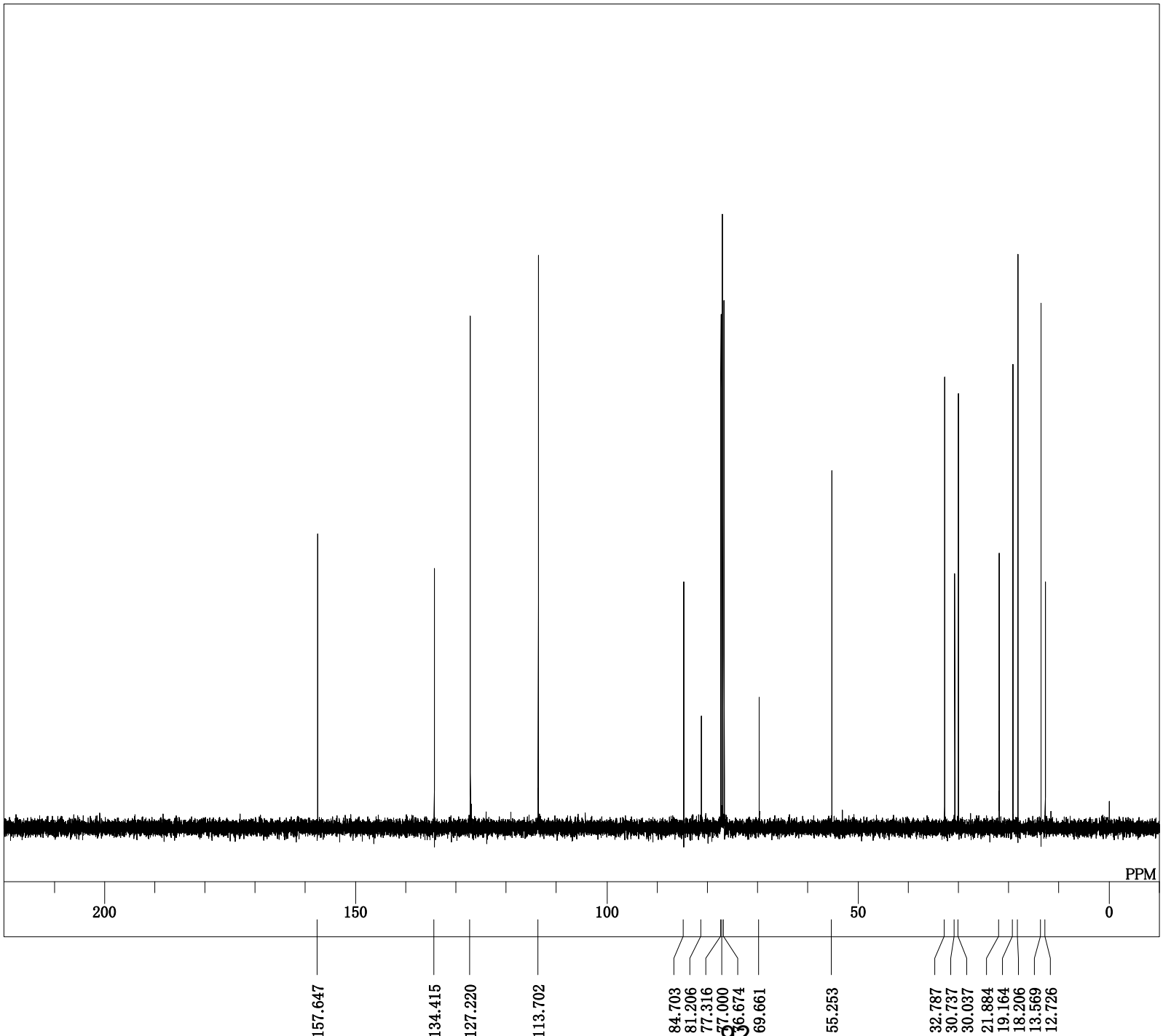
2n



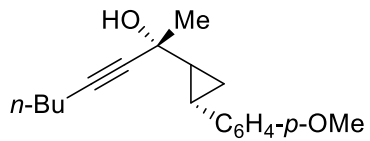
DFILE alpha_2o_1H.als
COMNT single_pulse
DATIM 2020-10-18 15:22:57
OBNUC 1H
EXMOD proton.jxp
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.24 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 3.14 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 24



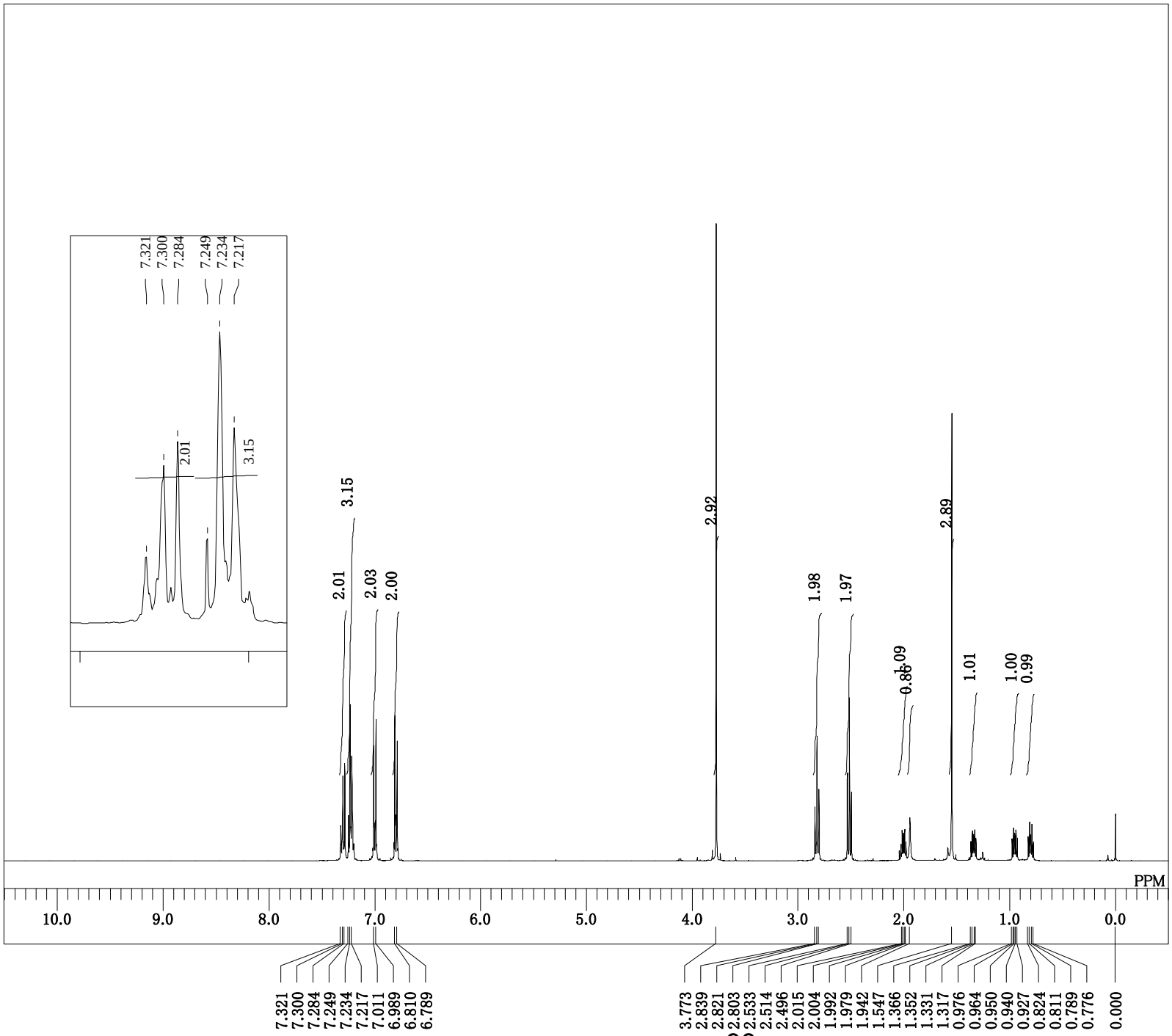
α–**2o**



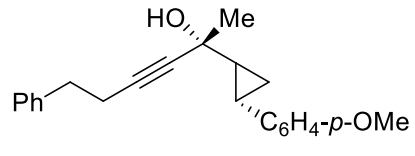
DFILE	alpha_2o_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-10-18 15:24:10
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	32767
FREQU	31250.00 Hz
SCANS	256
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



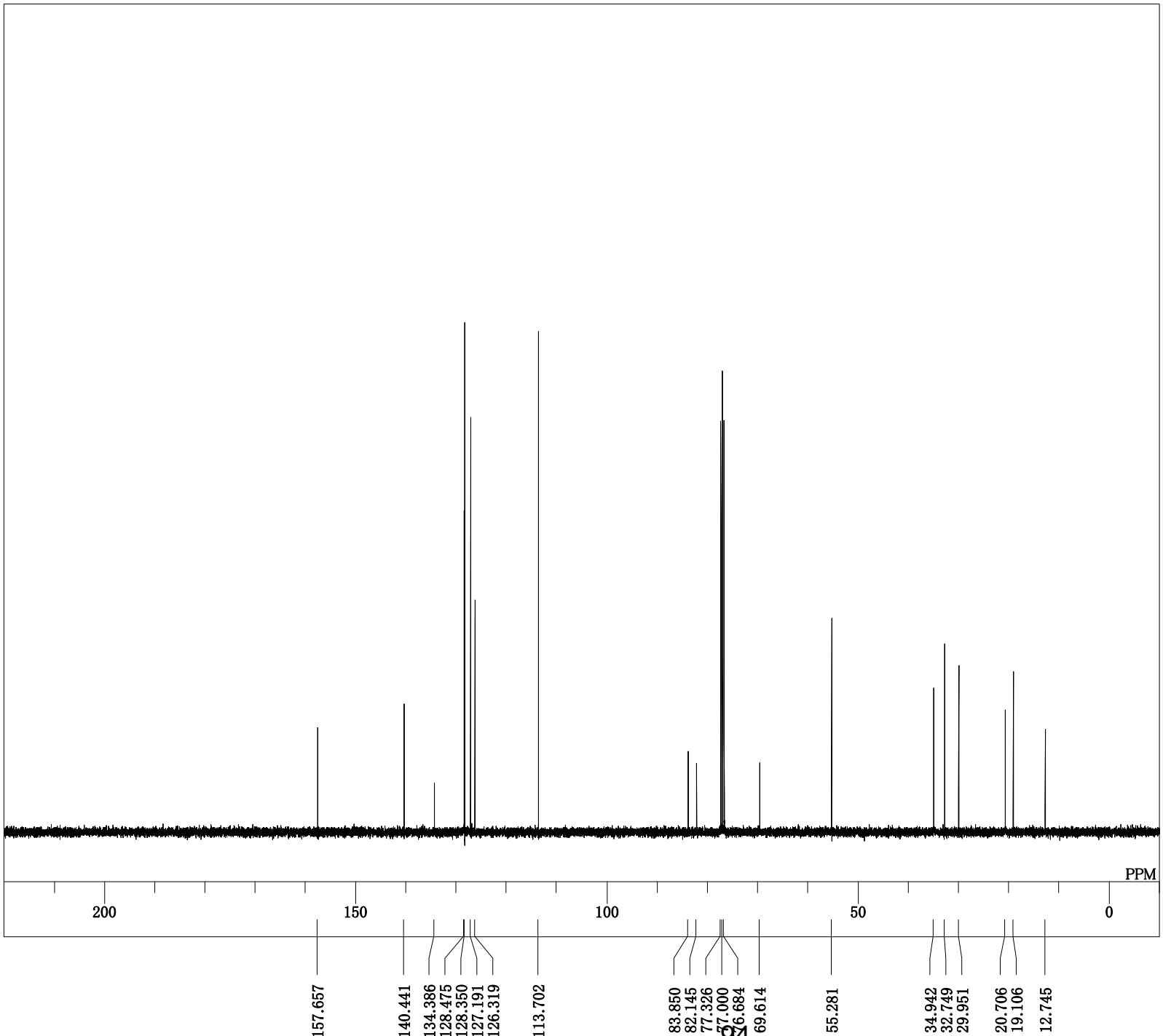
α–**2o**



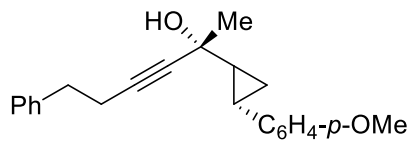
DFILE	alpha_2p_1H.als
COMNT	single_pulse
DATIM	2020-12-16 18:32:31
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	18.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	30



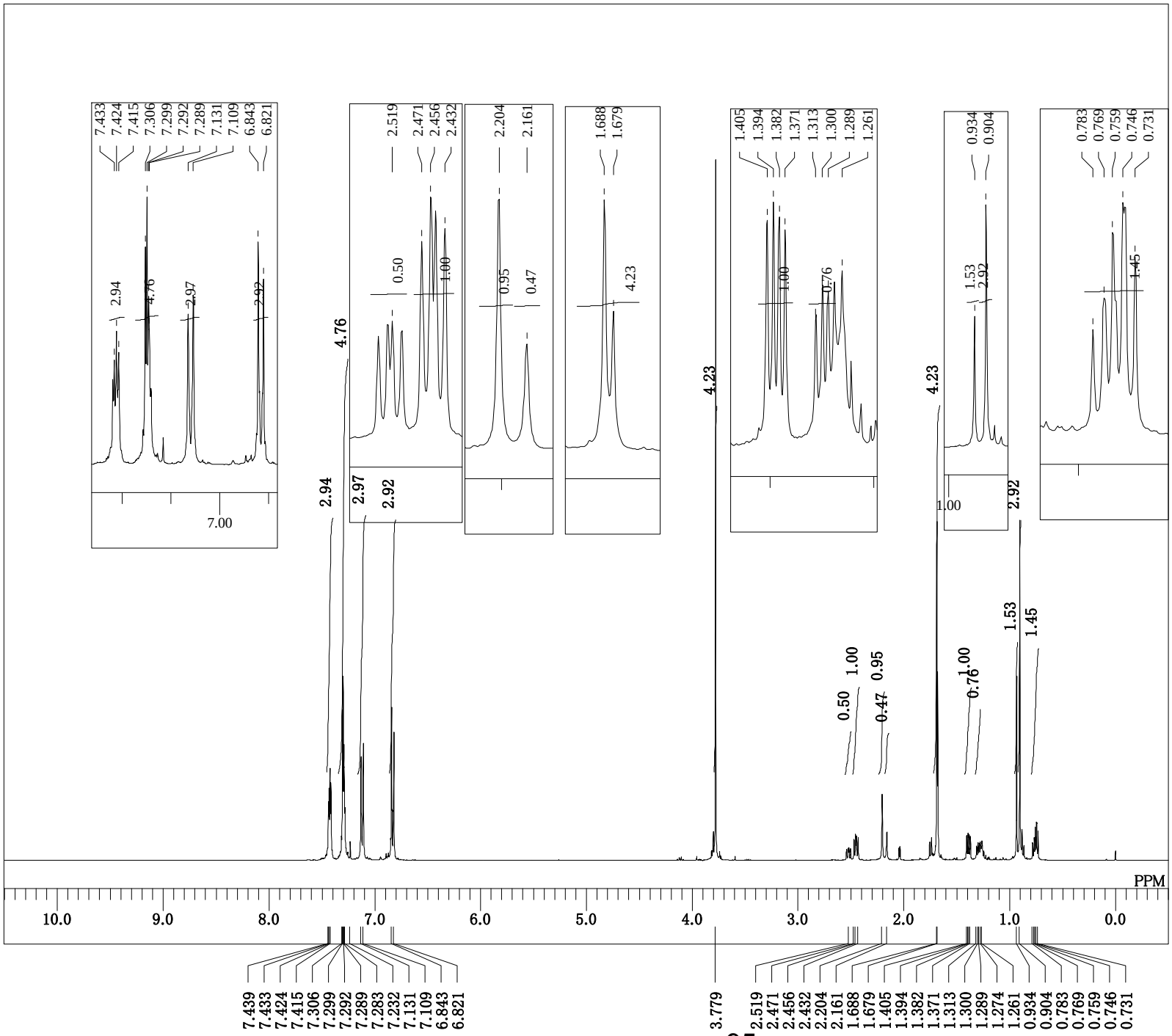
α-2p



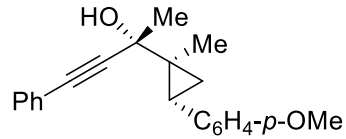
DFILE	alpha_2p_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-12-16 18:33:43
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	32767
FREQU	31250.00 Hz
SCANS	512
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50

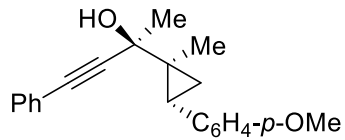


α-2p

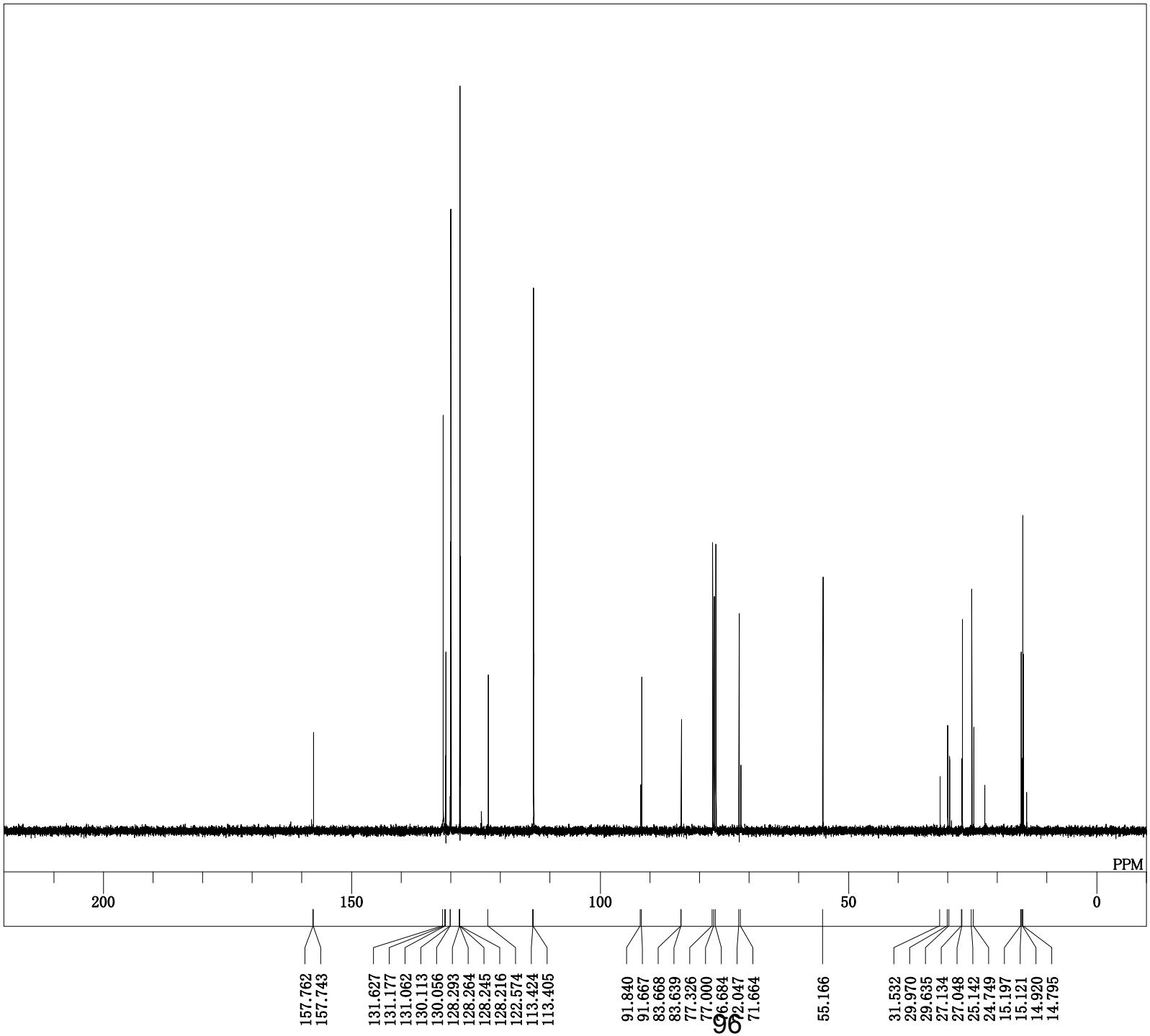


DFILE	alpha_2q_1H.als
COMNT	single_pulse
DATIM	2020-11-20 19:44:44
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	20

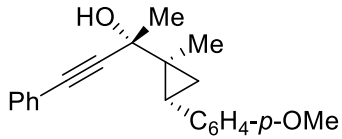


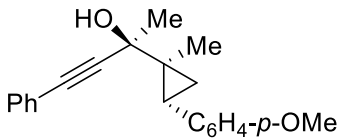


mixture of diastereomers 2q

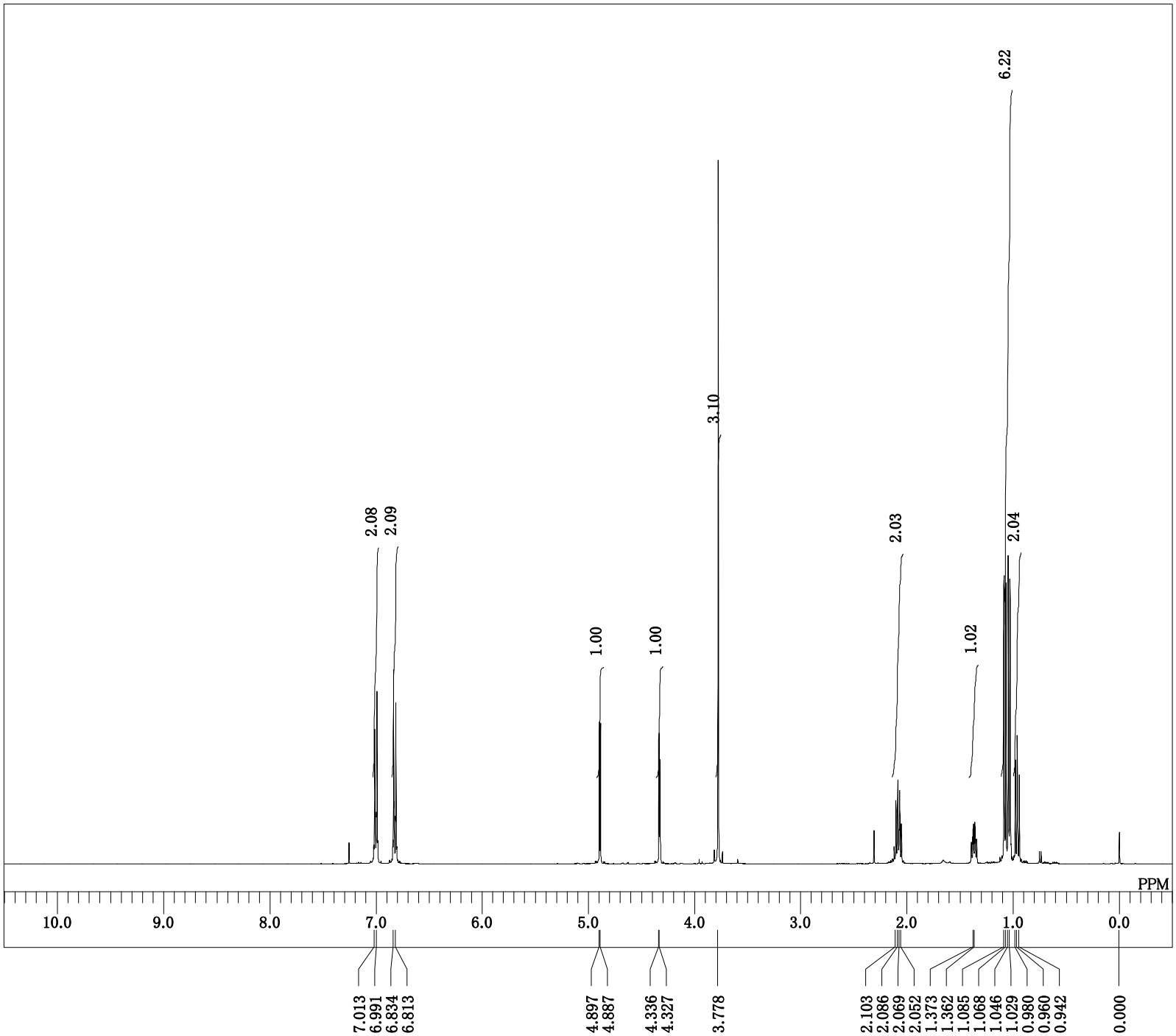


DFILE	alpha_2q_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-20 19:45:58
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	32767
FREQU	31250.00 Hz
SCANS	256
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50

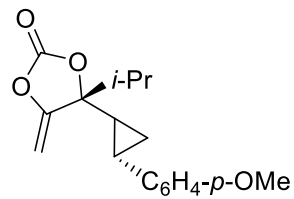




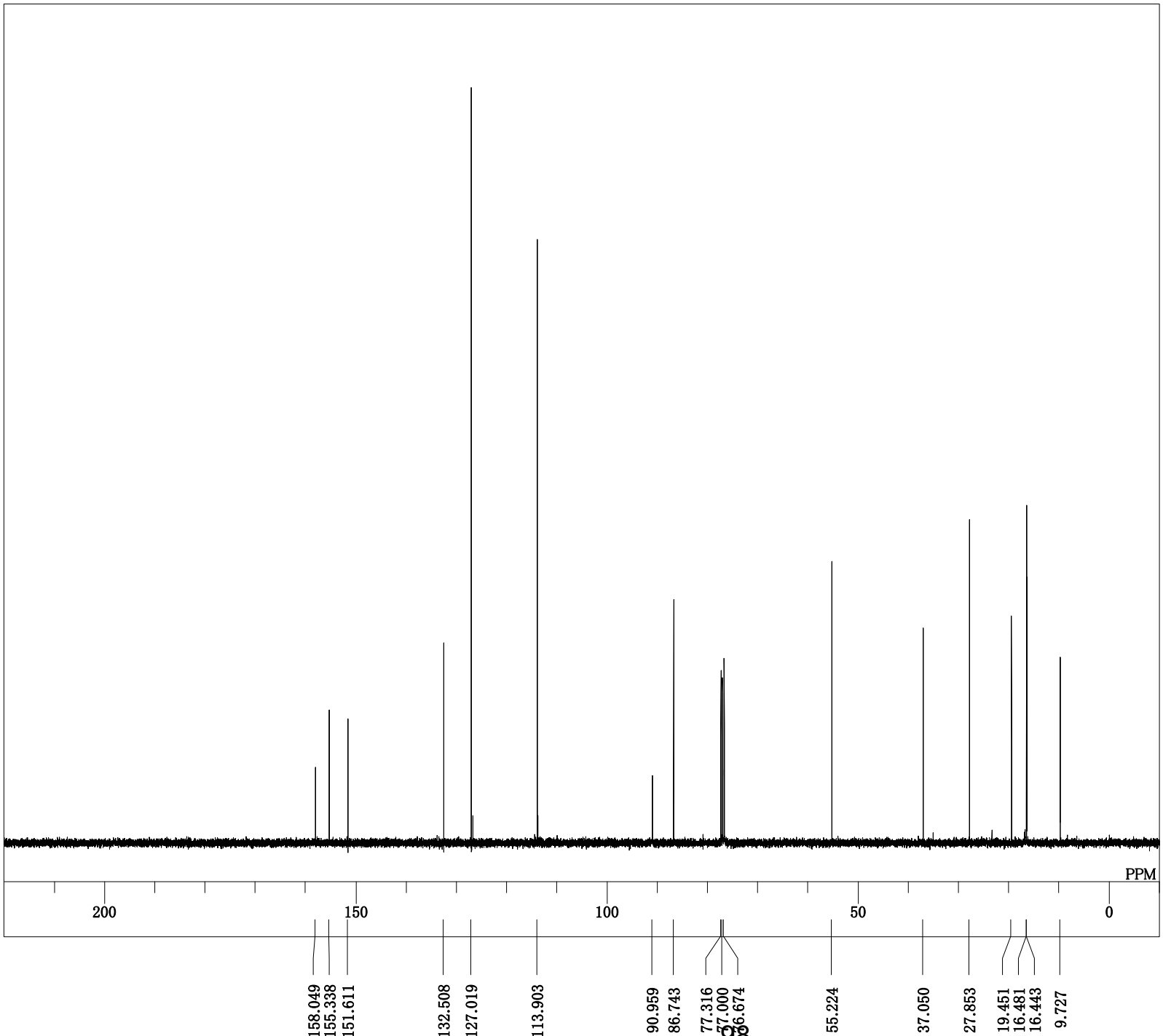
mixture of diastereomers 2q



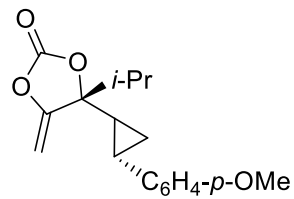
DFILE	alpha_3a_1H.als
COMNT	single_pulse
DATIM	2020-10-18 14:18:29
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	19.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	22



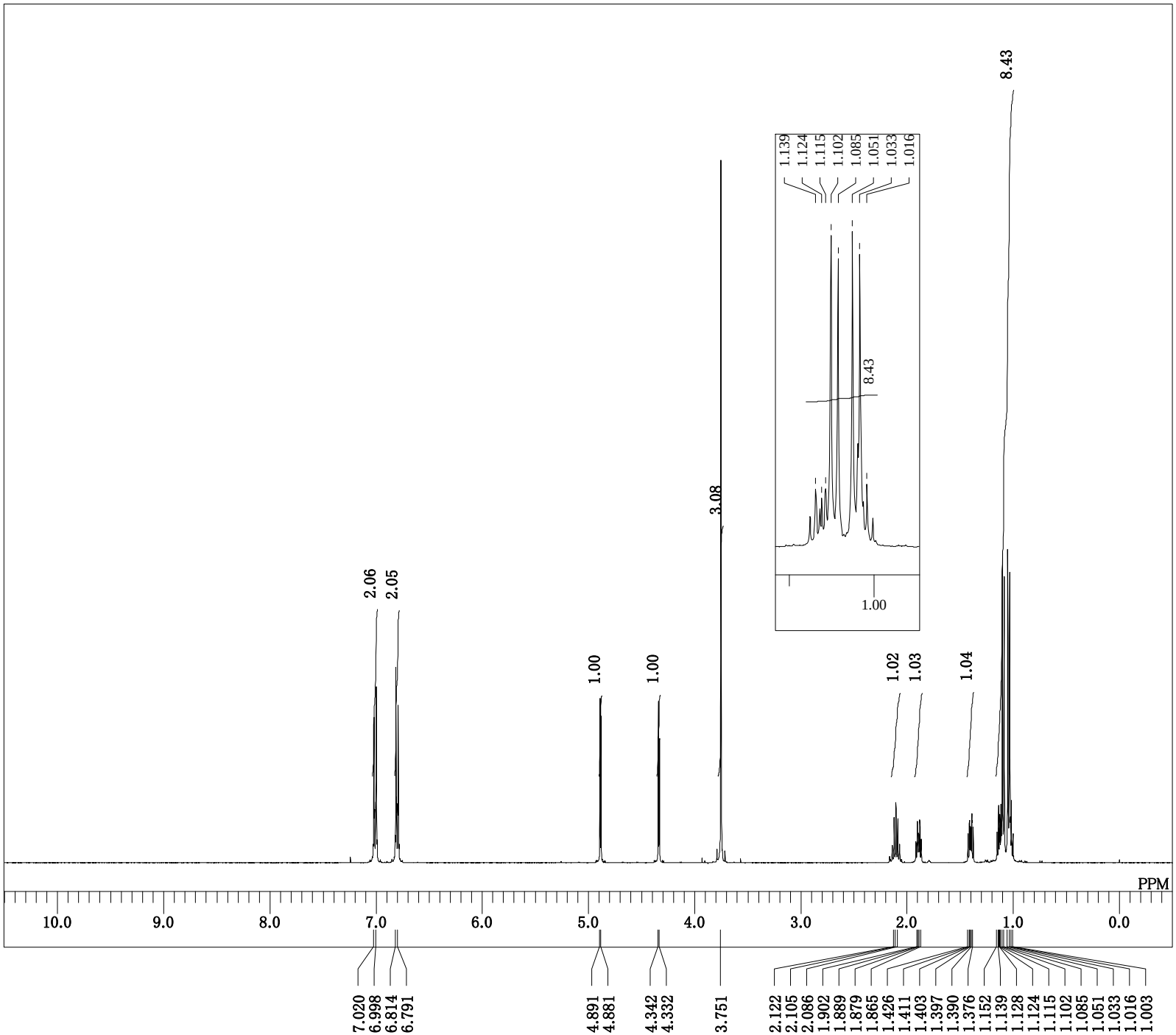
α-3a



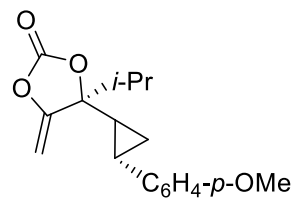
DFILE	alpha_3a_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-10-18 14:19:42
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	32767
FREQU	31250.00 Hz
SCANS	257
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



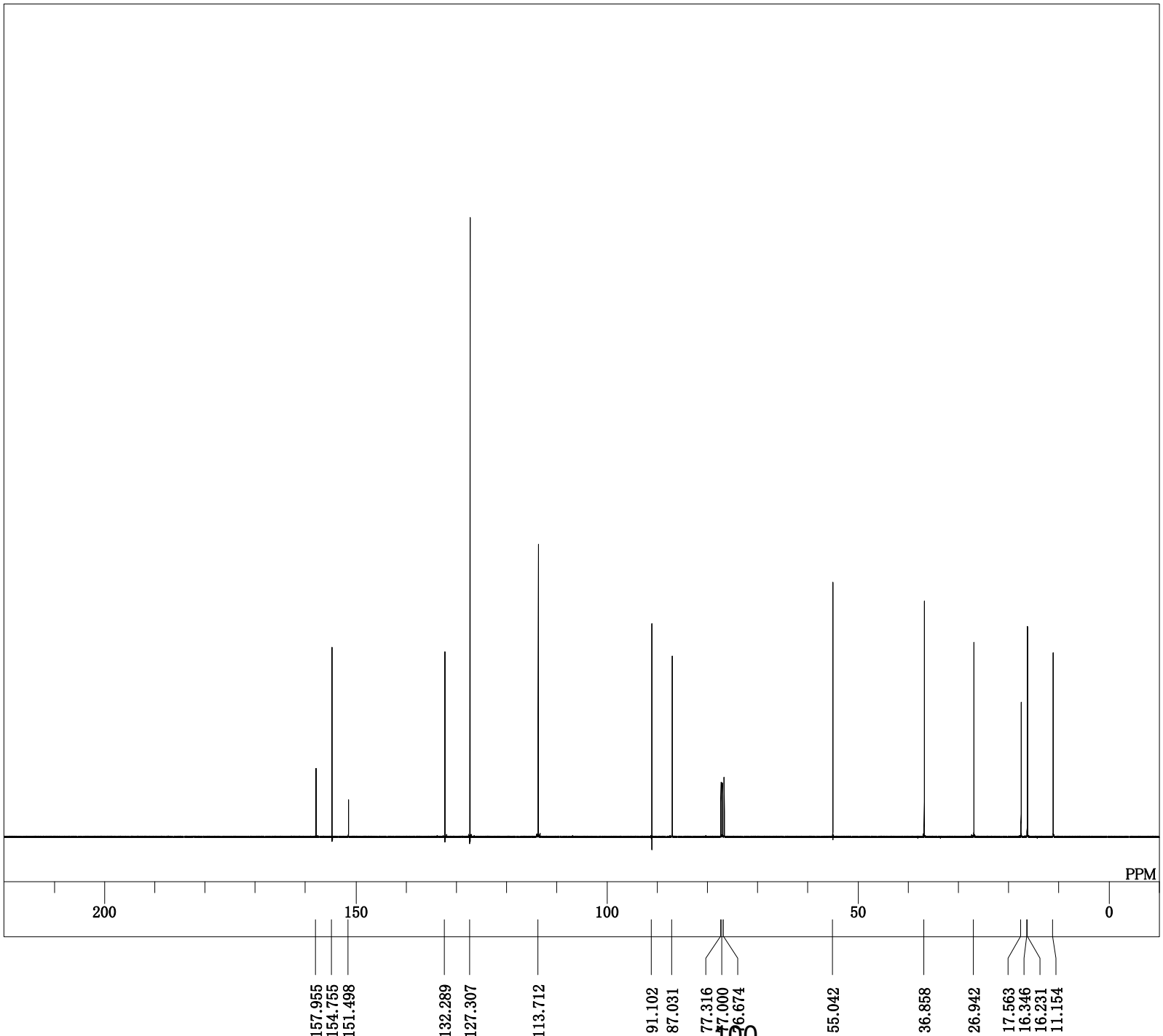
α-3a



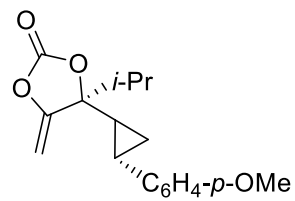
DFILE	beta_3a_1H.als
COMNT	single_pulse
DATIM	2020-11-25 14:08:49
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	19.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	12



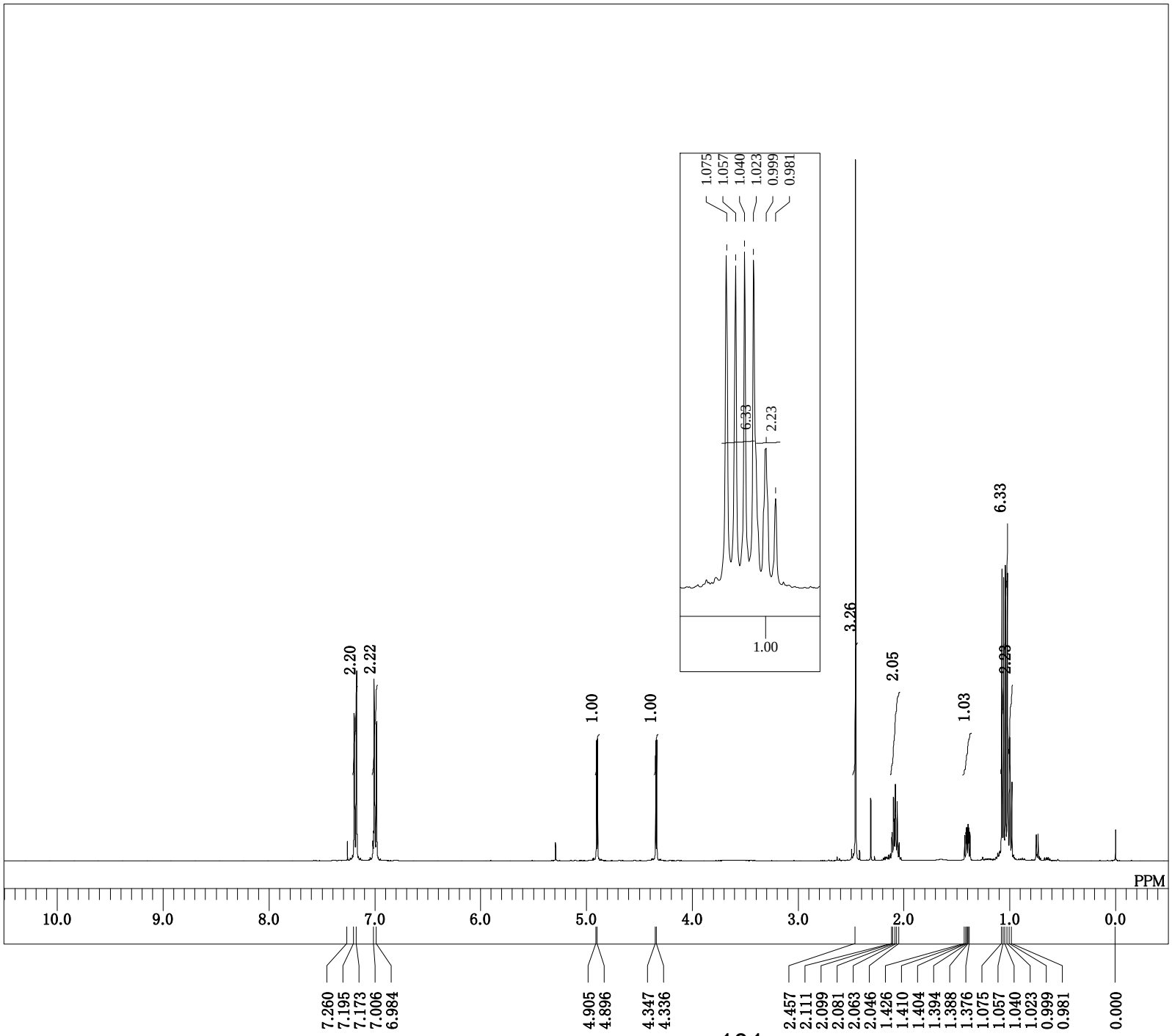
β-3a

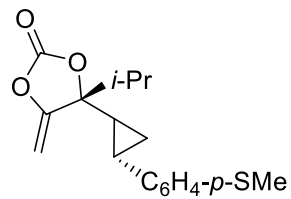


DFILE	beta_3a_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-25 14:10:01
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	512
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	18.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50

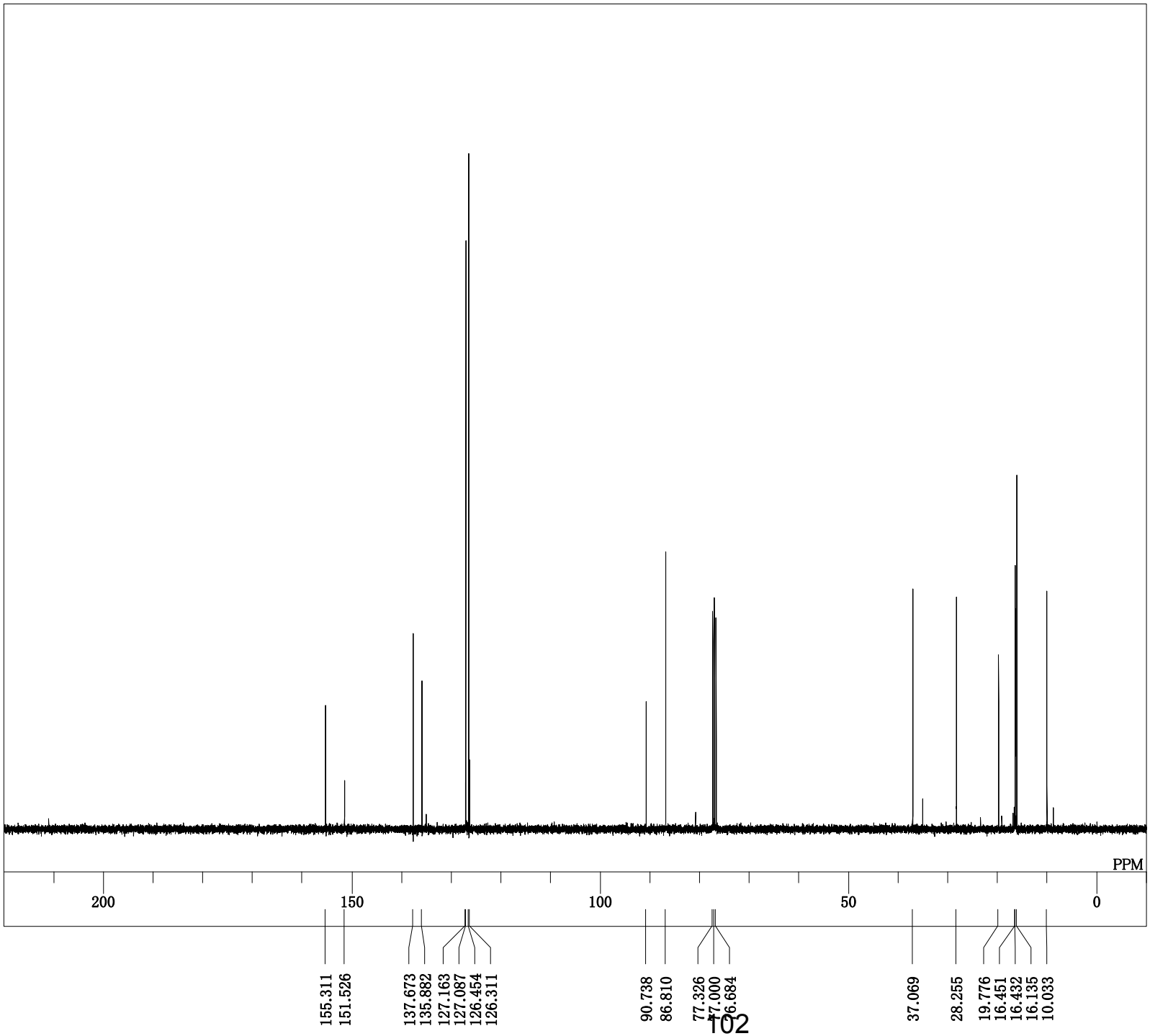


β-3a

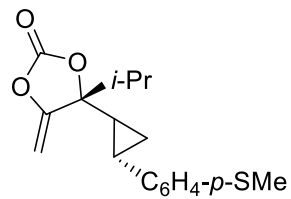

DFILE alpha_3b_1H.als
COMNT single_pulse
DATIM 2020-10-18 15:42:24
OBNUC 1H
EXMOD proton.jxp
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.24 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 3.14 usec
IRNUC 1H
CTEMP 19.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 22
1.075
1.057
1.040
1.023
0.999
0.981
6.33
2.23
1.00
2.20
2.22
1.00
1.00
3.26
2.05
1.03
6.33
PPM
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
7.260
7.195
7.173
7.006
6.984
4.905
4.896
4.347
4.336
2.457
2.111
2.099
2.081
2.063
2.046
1.426
1.410
1.404
1.394
1.388
1.376
1.075
1.057
1.040
1.023
0.999
0.981
0.000


O
O
O
i-Pr
C6H4-p-SMe

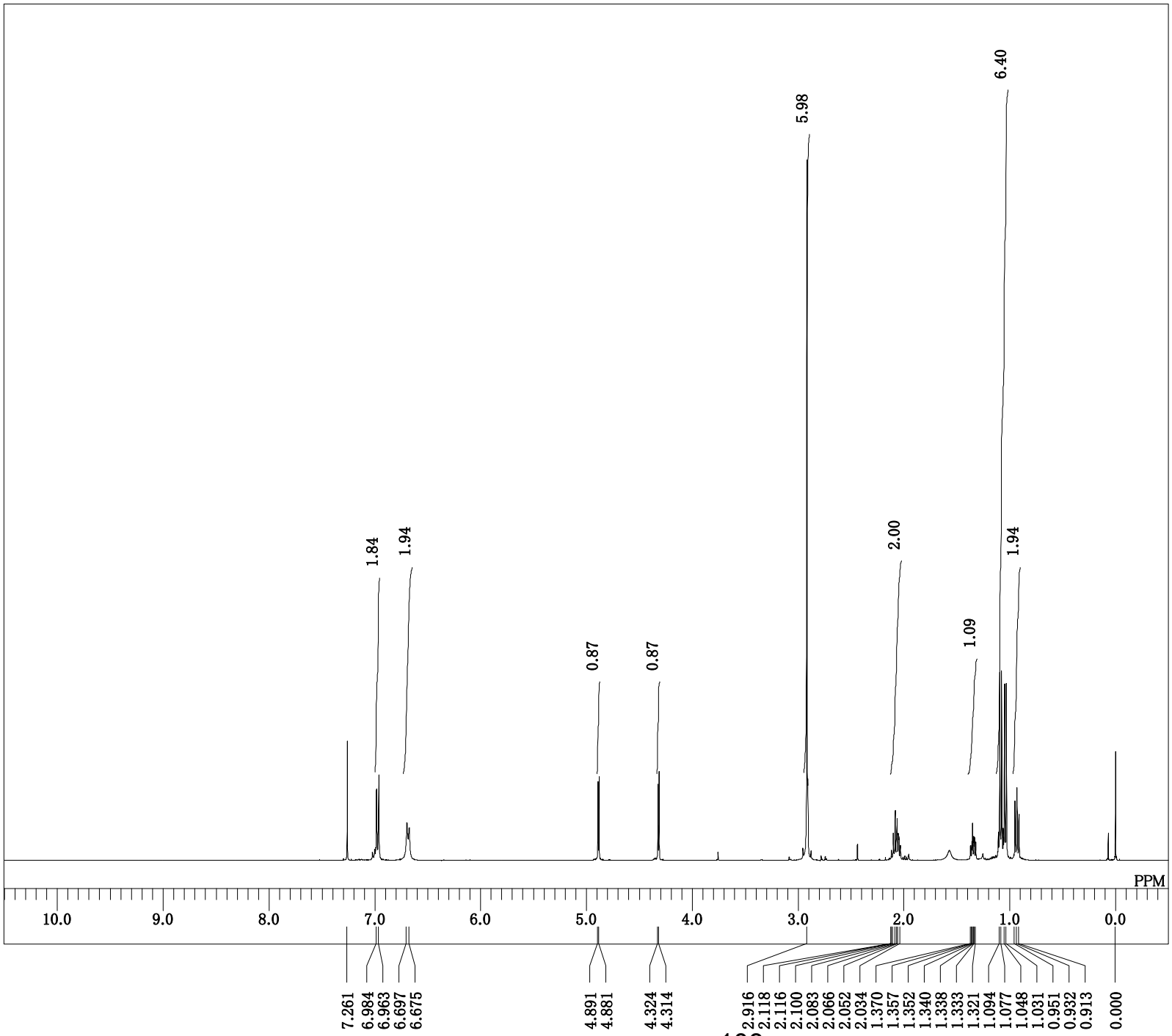
α–**3b**



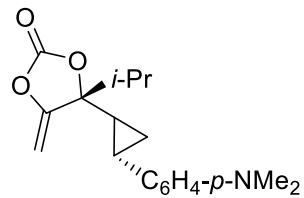
DFILE	alpha_3b_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-10-18 15:43:36
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	256
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



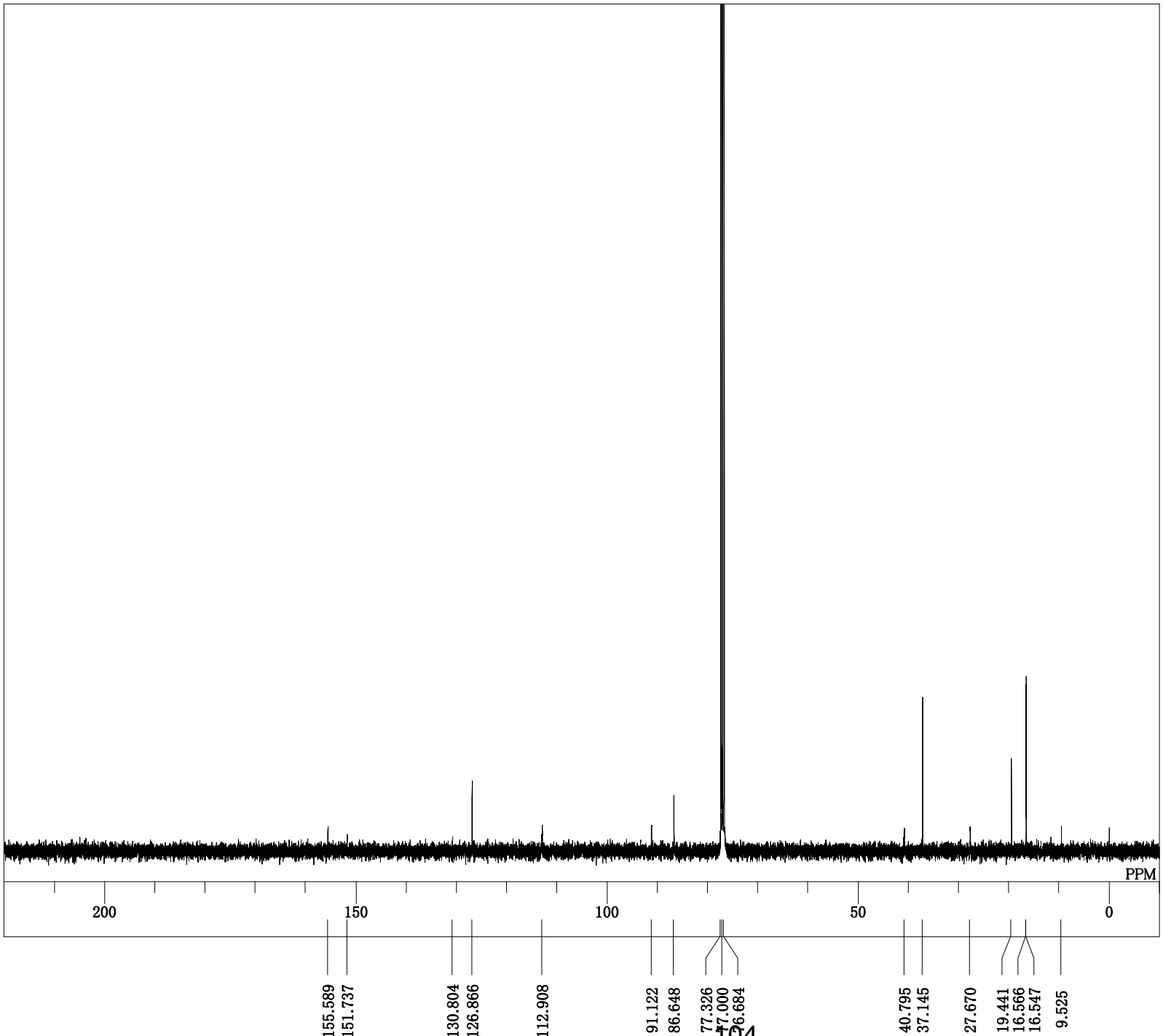
α–**3b**



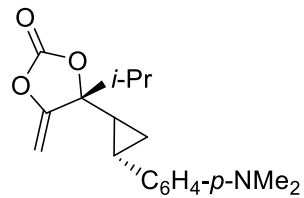
DFILE	alpha_3c_1H.als
COMNT	single_pulse
DATIM	2020-11-10 19:39:02
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	16
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	18.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	38



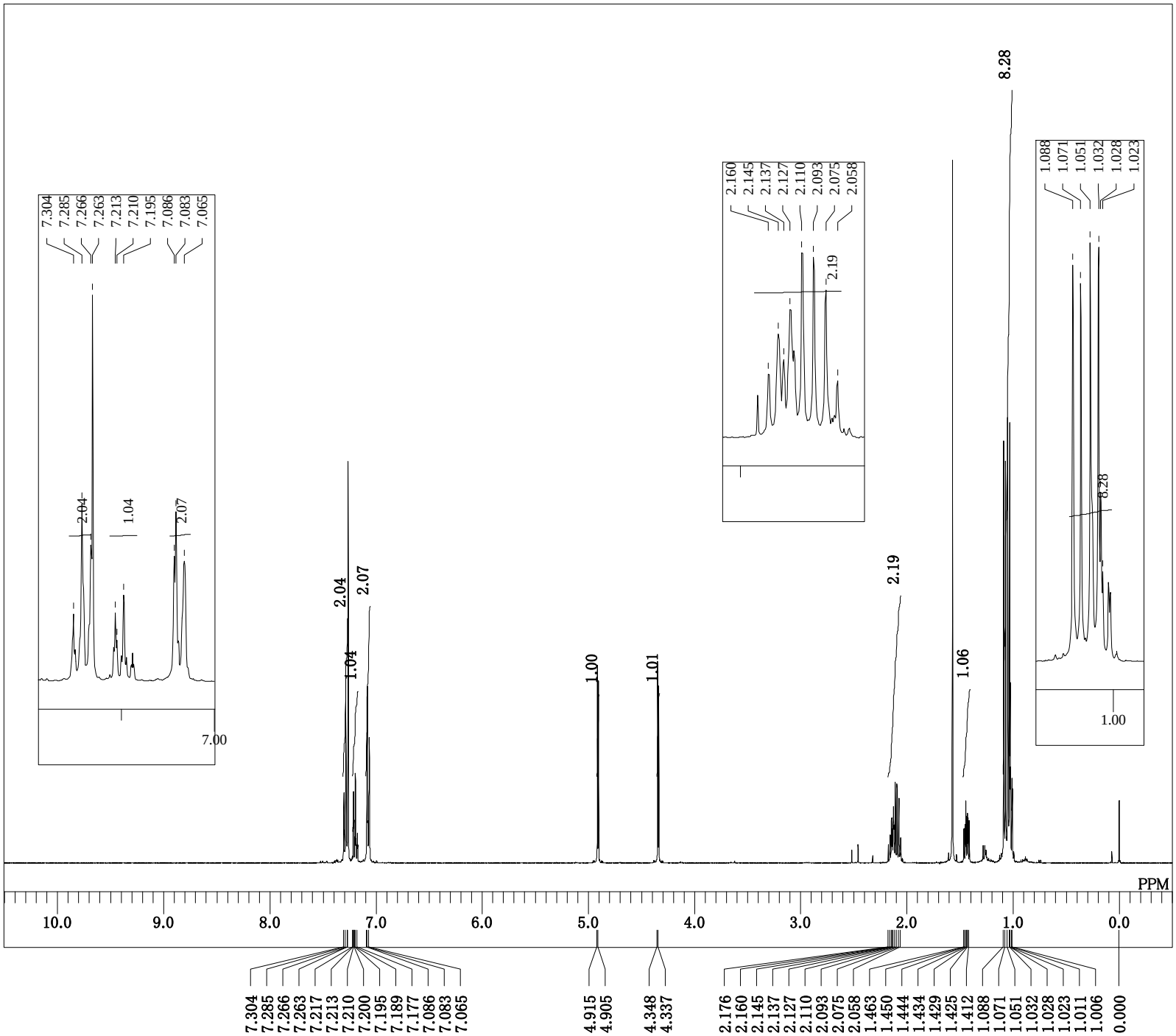
α–**3c**



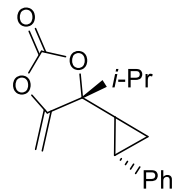
DFILE	alpha_3c_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-12-27 20:15:29
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	15963
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	18.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



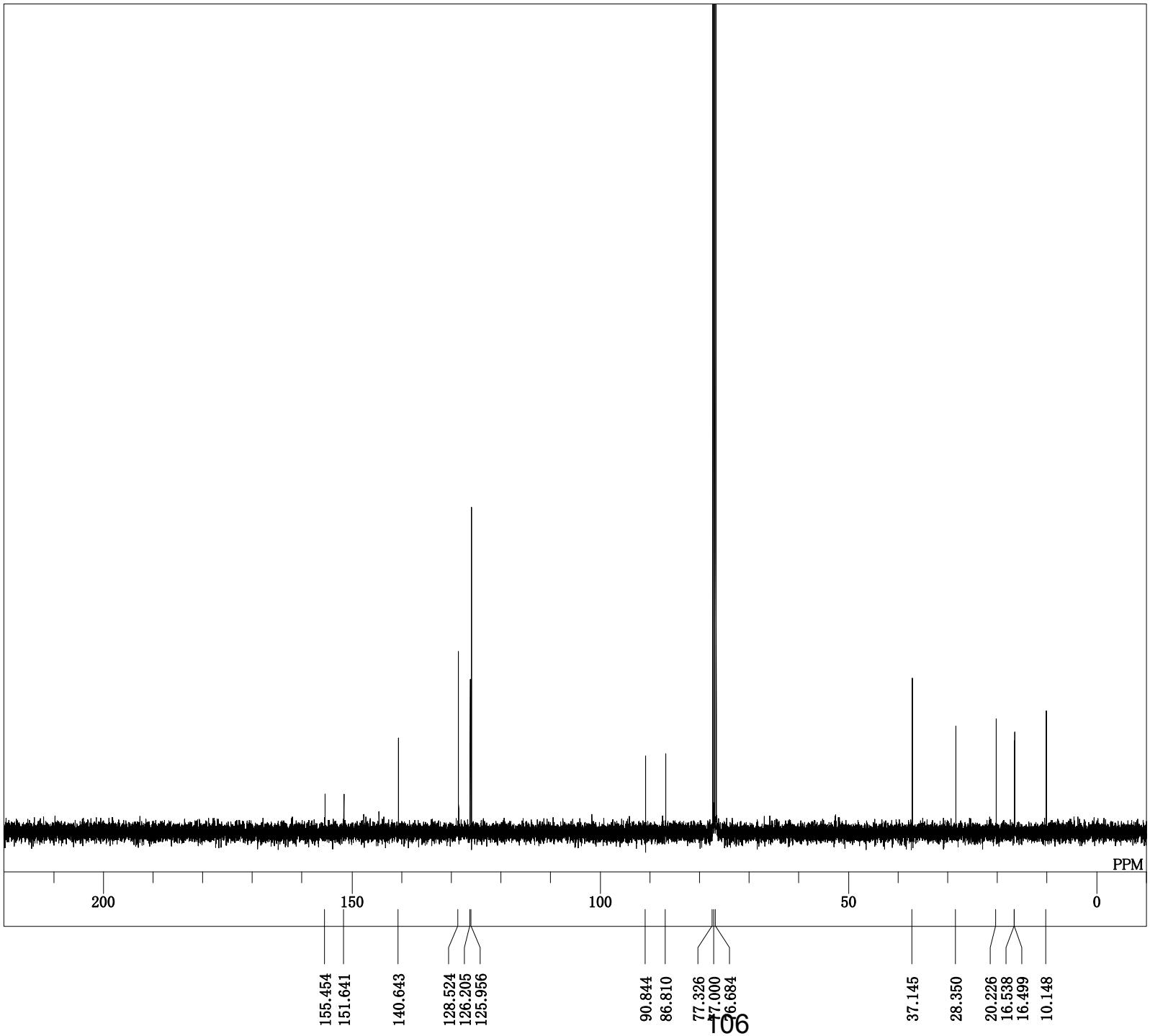
α–**3c**



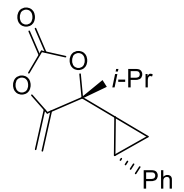
DFILE	alpha_3d_1H.als
COMNT	single_pulse
DATIM	2020-11-02 19:11:22
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	42



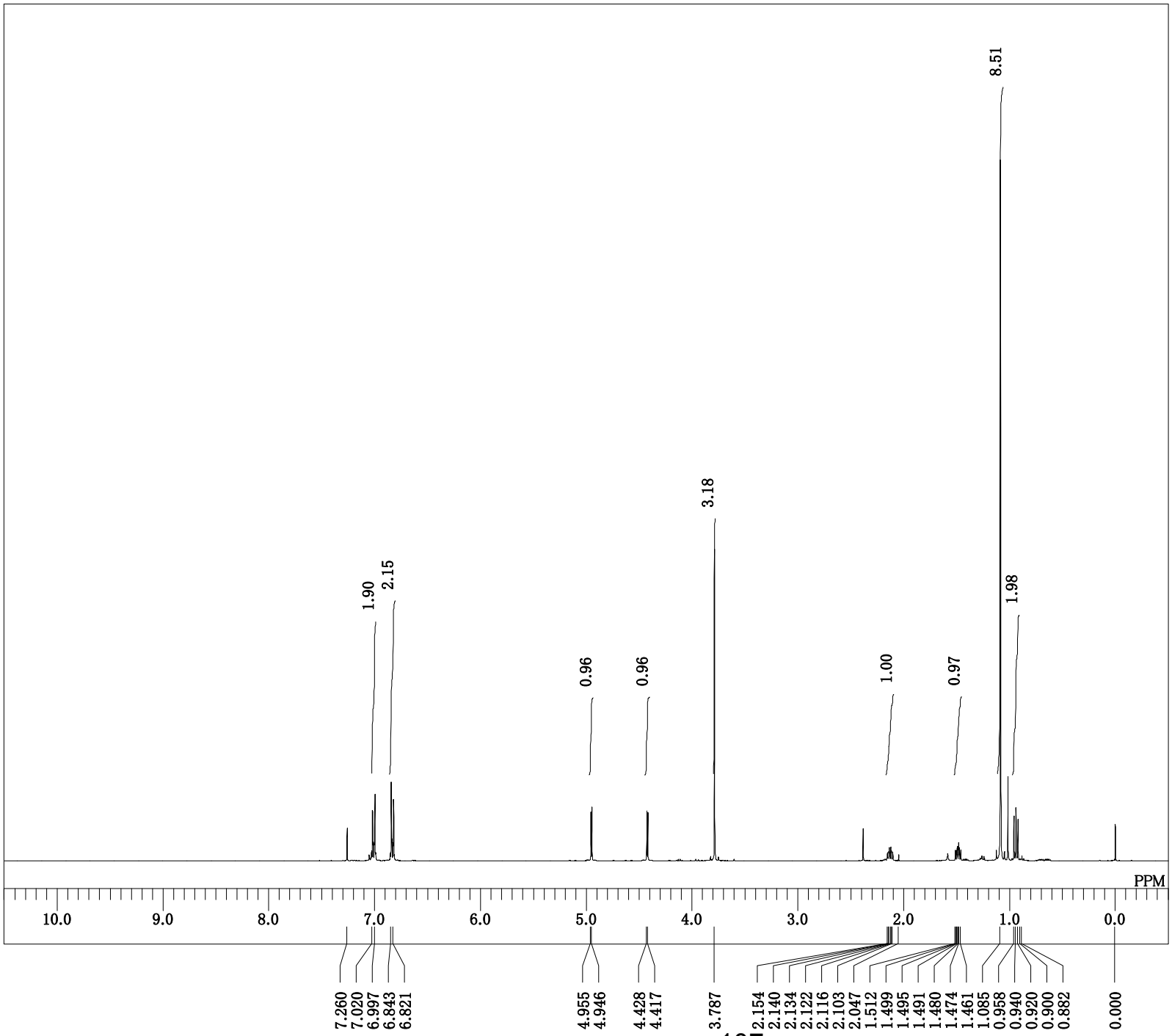
α–**3d**



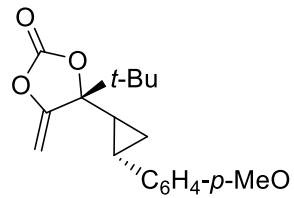
DFILE	alpha_3d_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-02 19:12:34
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	995
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



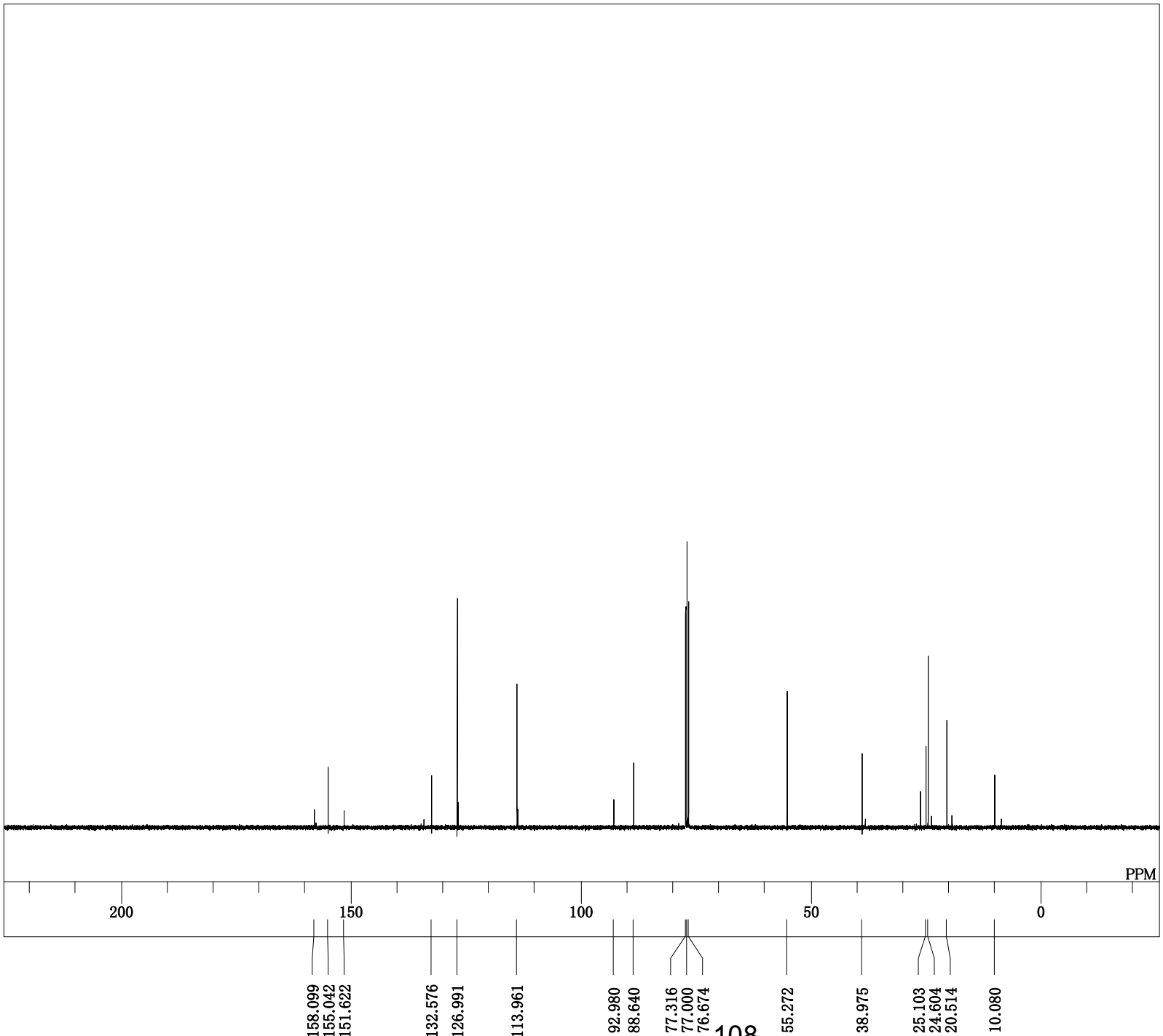
α–**3d**



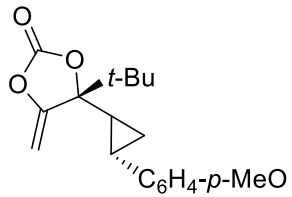
DFILE alpha_3e_1H.als
COMNT single_pulse
DATIM 2020-12-14 20:49:06
OBNUC 1H
EXMOD proton.jxp
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.24 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 3.14 usec
IRNUC 1H
CTEMP 18.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 30



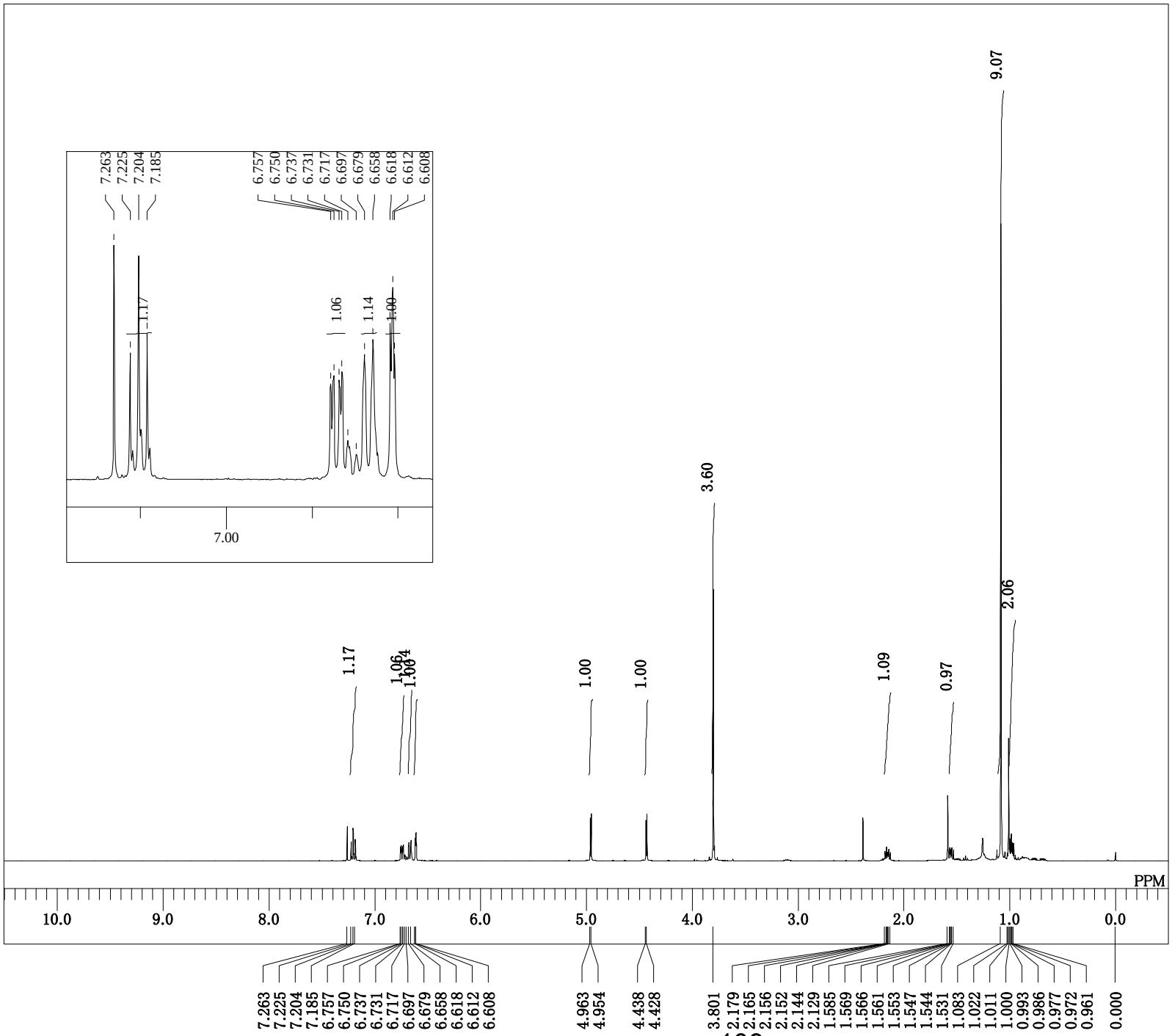
α–**3e**

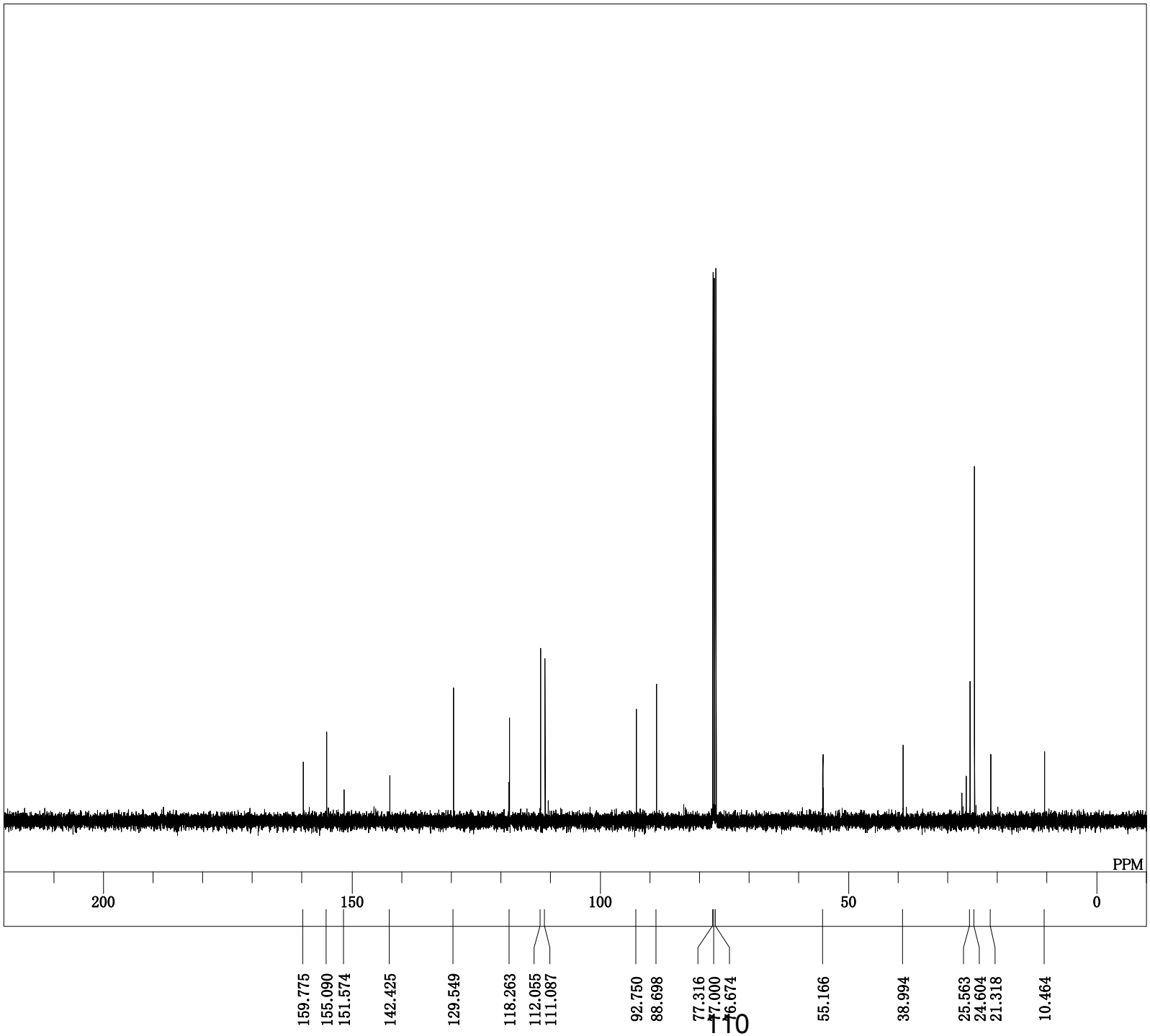


DFILE	alpha_3e_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-12-28 10:08:34
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	1024
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	18.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50

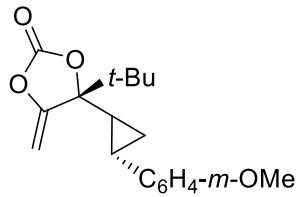


α–**3e**

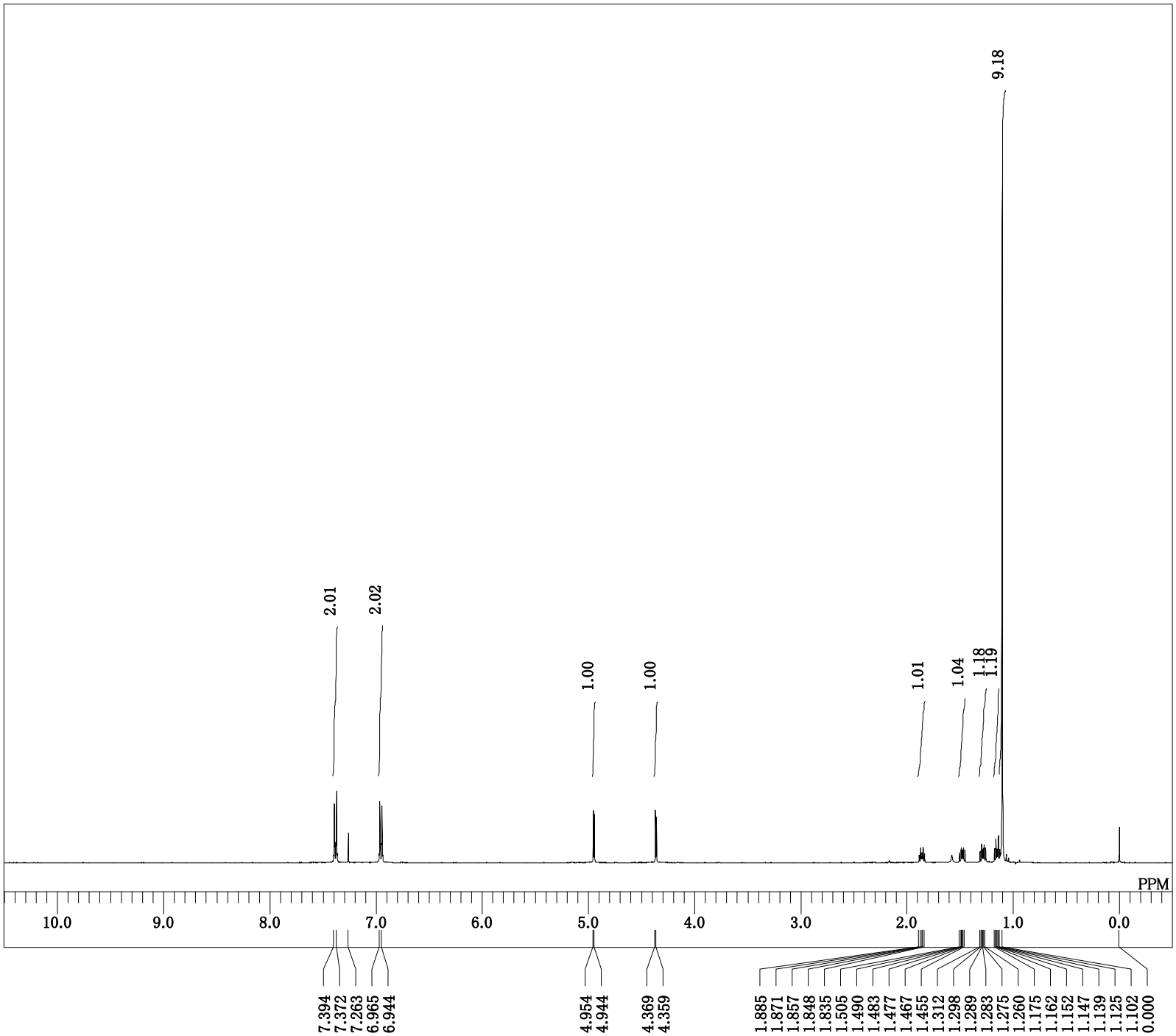

DFILE alpha_3f_1H.als
COMNT single_pulse
DATIM 2020-11-03 16:04:25
OBNUC 1H
EXMOD proton.jxp
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.24 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 3.14 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 32
PPM
t-Bu
C6H4-m-OMe
α–3f



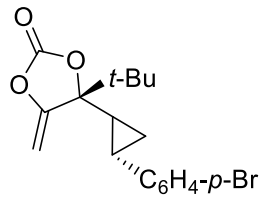
DFILE	alpha_3f_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-03 16:06:41
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	319
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



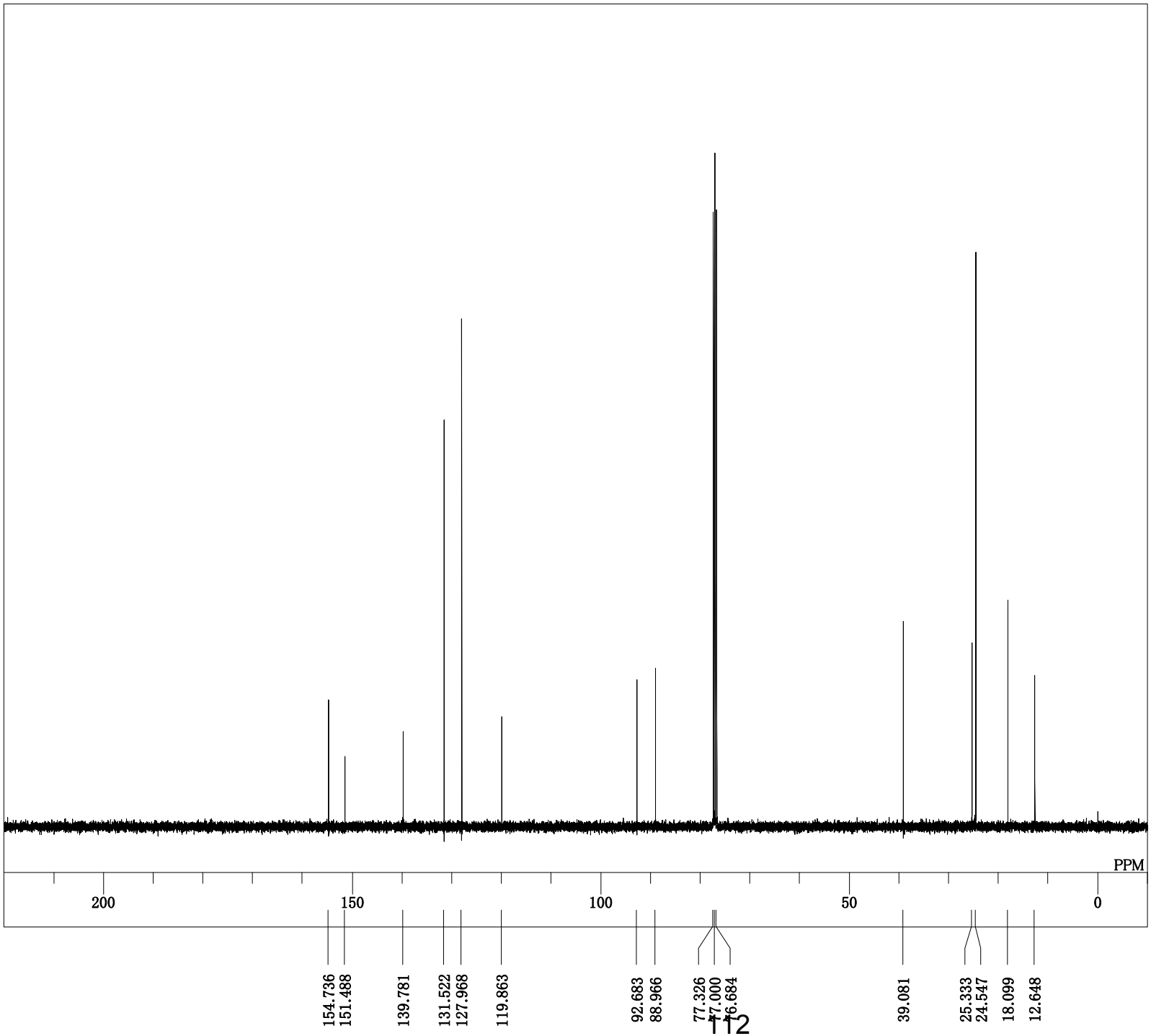
α–**3f**



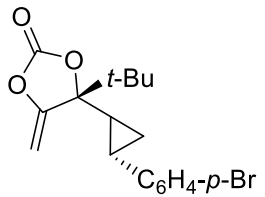
DFILE	alpha_3g_1H.als
COMNT	single_pulse
DATIM	2020-11-05 18:40:43
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	19.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	34



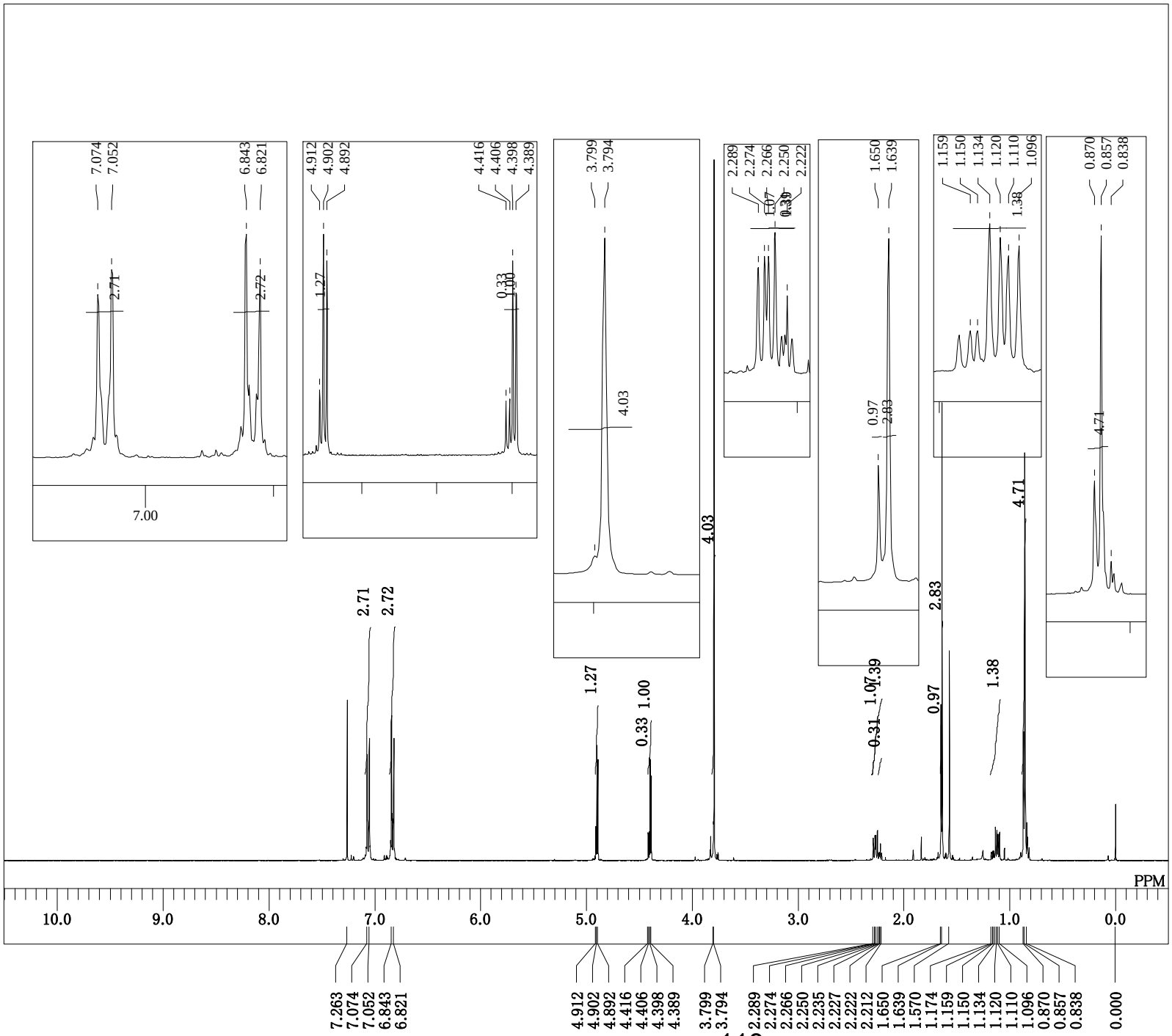
α-3g



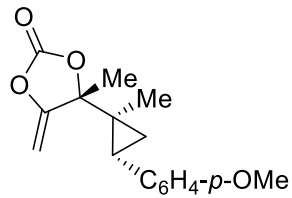
DFILE	alpha_3g_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-05 18:42:57
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	1009
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50

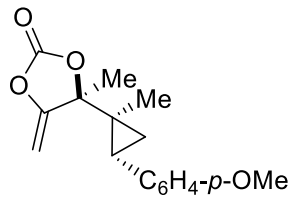


α-3g

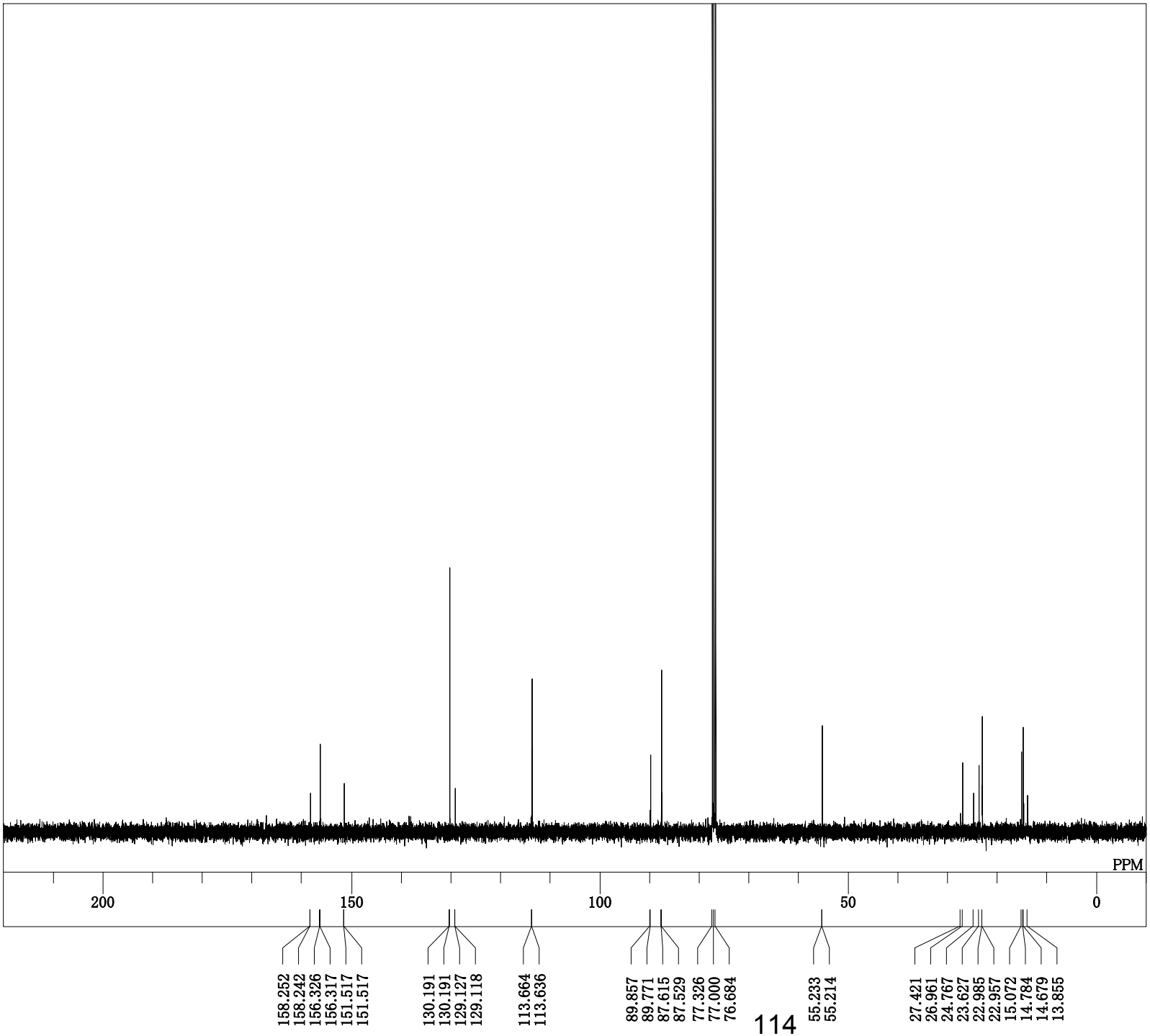


DFILE	3h_1H.als
COMNT	single_pulse
DATIM	2020-11-23 20:37:25
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	38

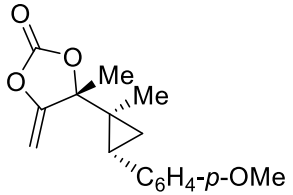


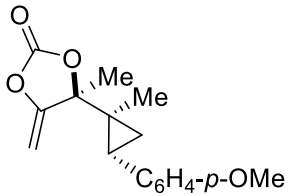


mixture of diastereomers 3h

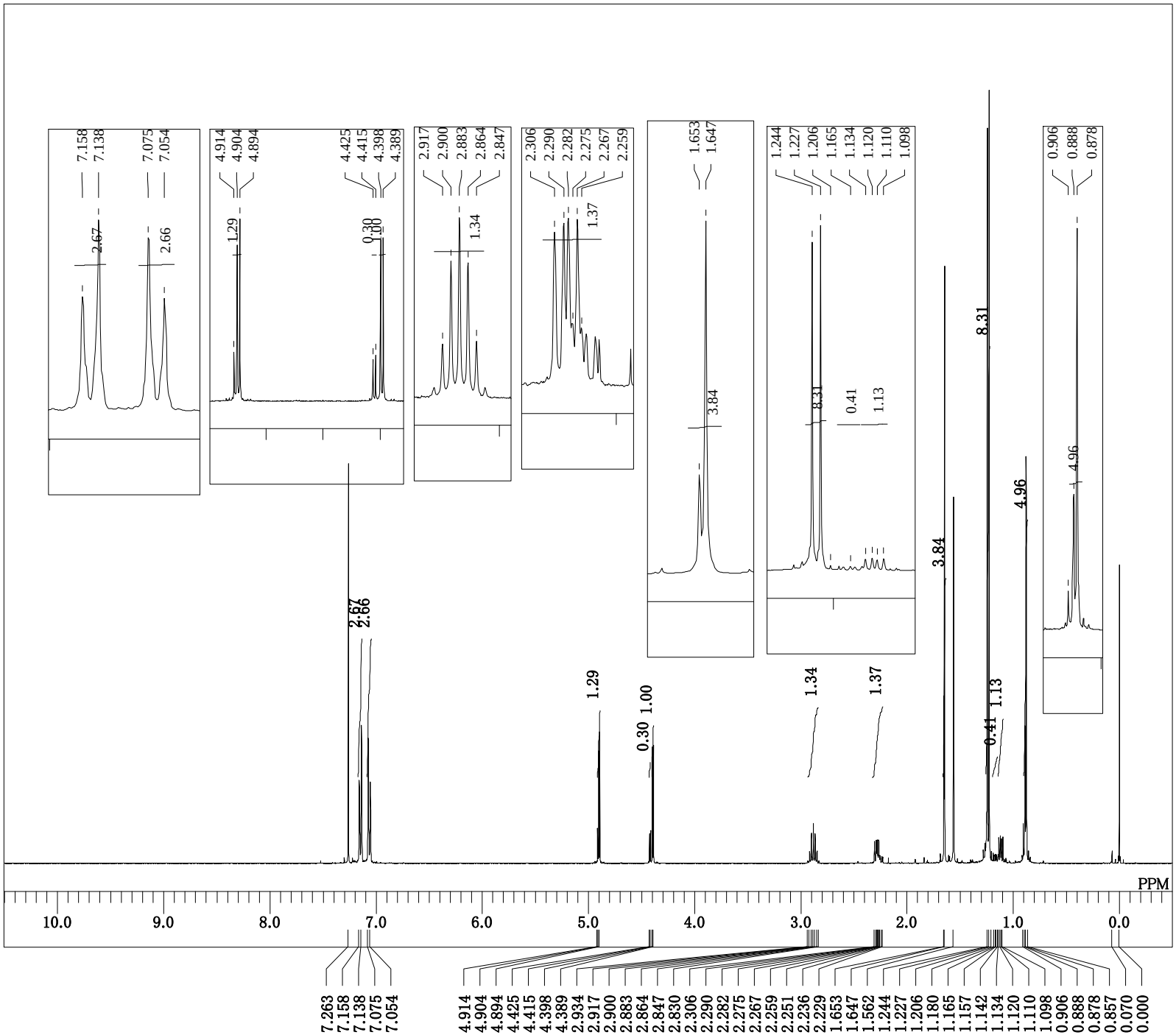


DFILE	alpha_3h_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-23 20:38:37
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	1024
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50

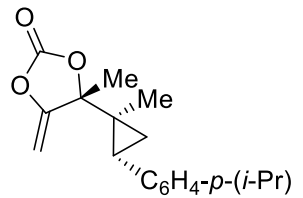


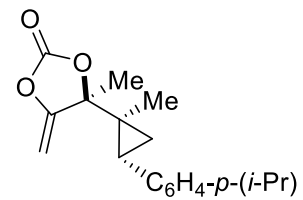


mixture of diastereomers 3h

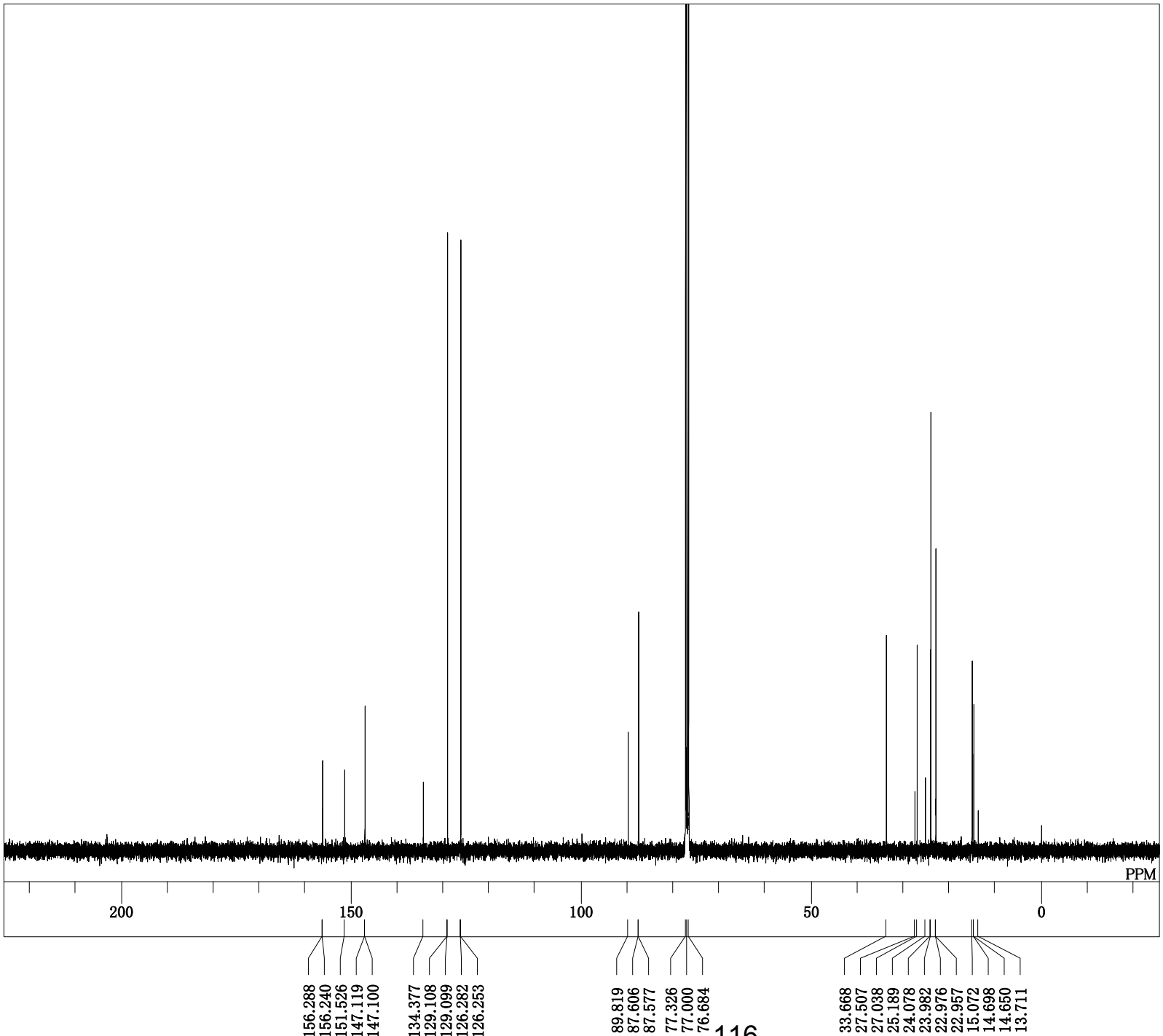


DFILE	3i_1H.als
COMNT	single_pulse
DATIM	2020-11-15 21:15:33
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	19.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	42

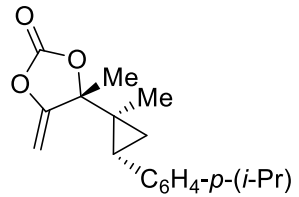


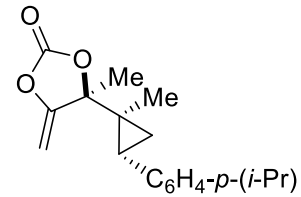


mixture of diastereomers 3i

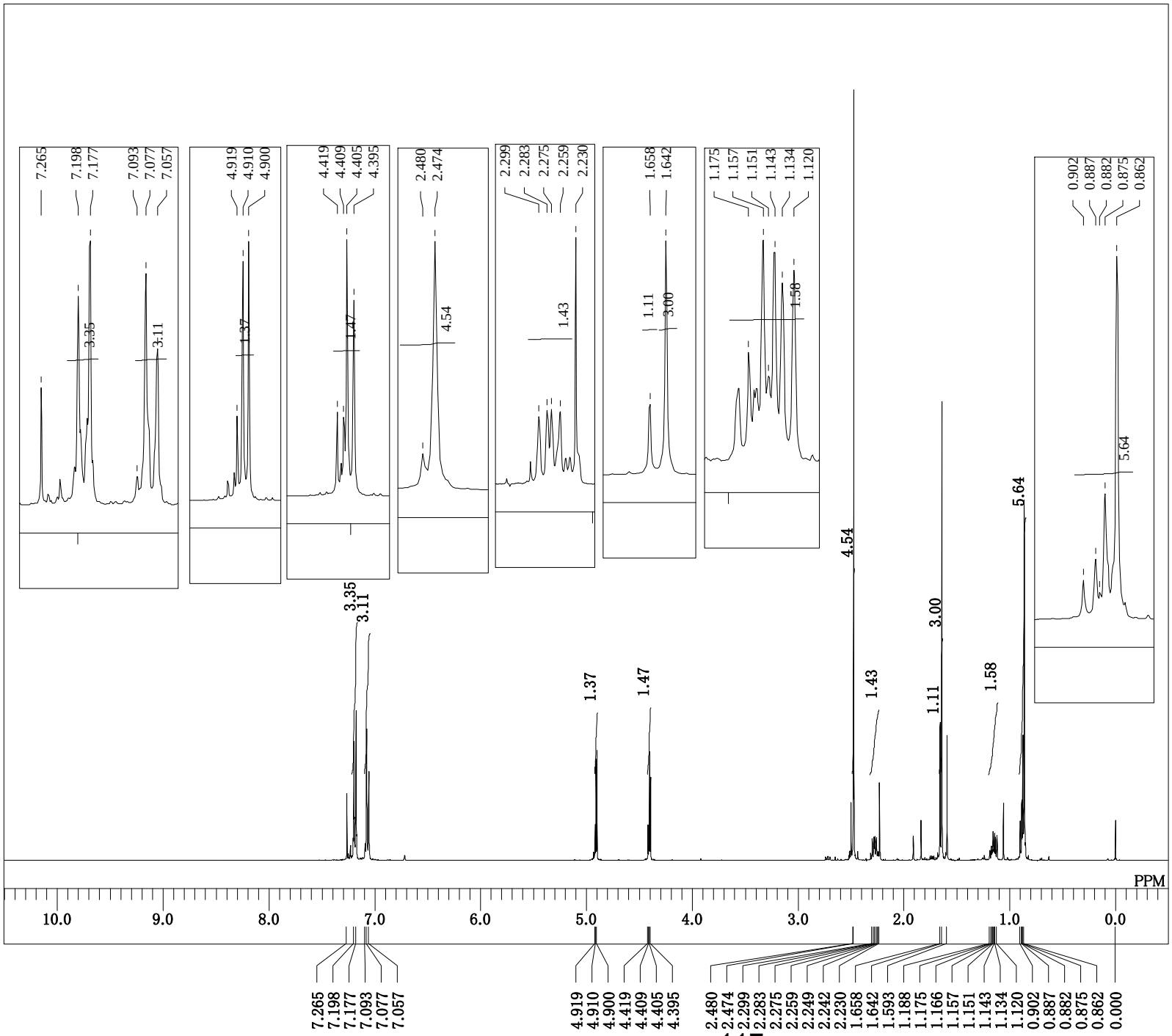


DFILE	alpha_3i_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-15 21:16:46
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	14449
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50

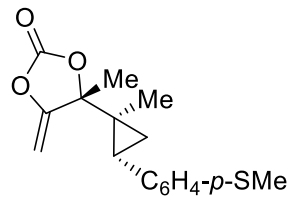


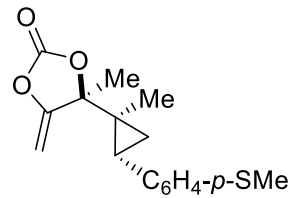


mixture of diastereomers 3i

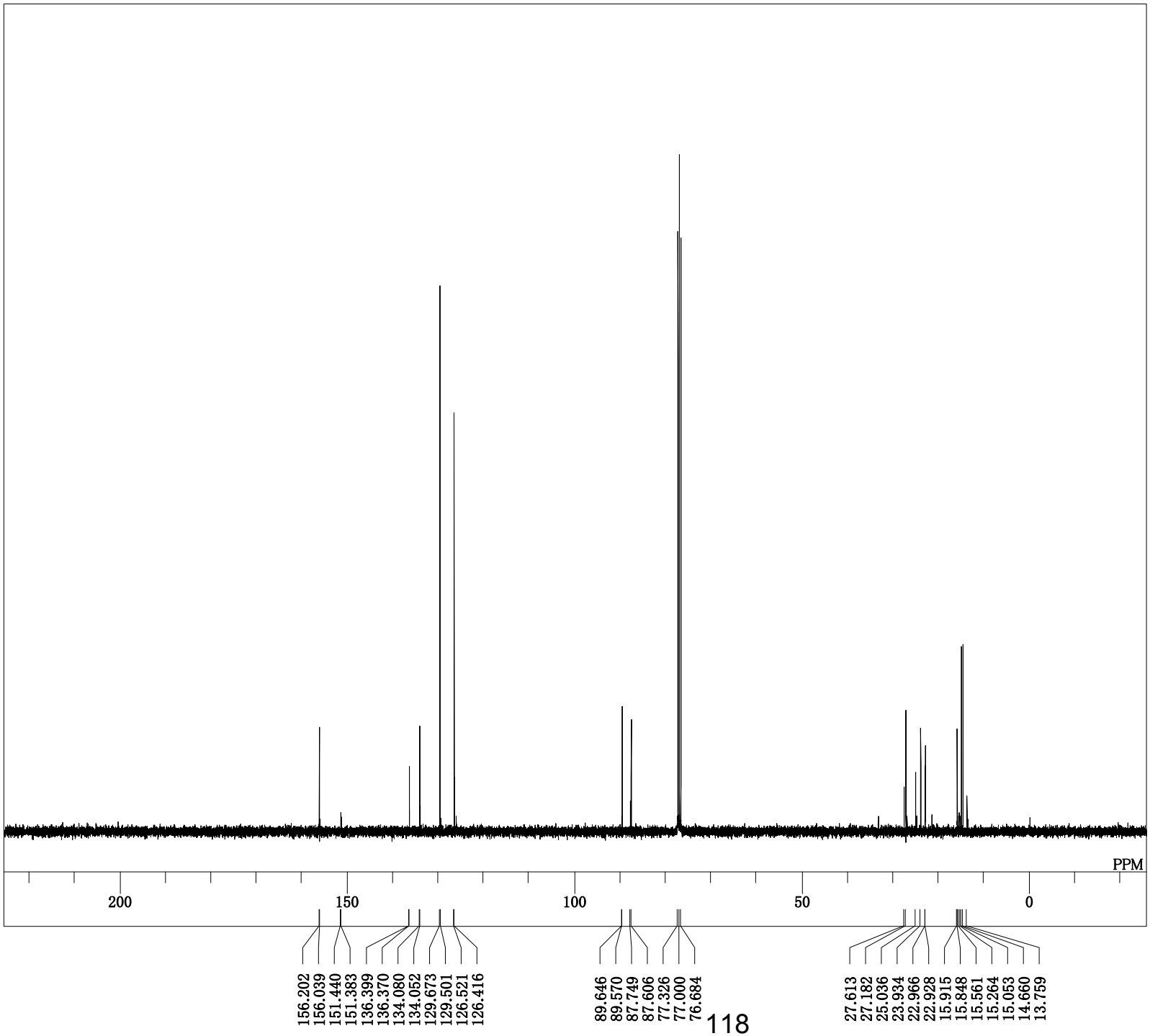


DFILE	3j_1H.als
COMNT	single_pulse
DATIM	2020-11-18 09:51:23
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	19.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	32

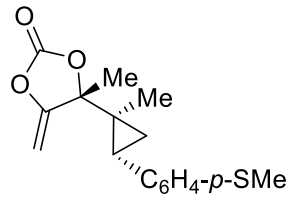


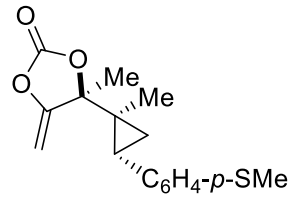


mixture of diastereomers 3j

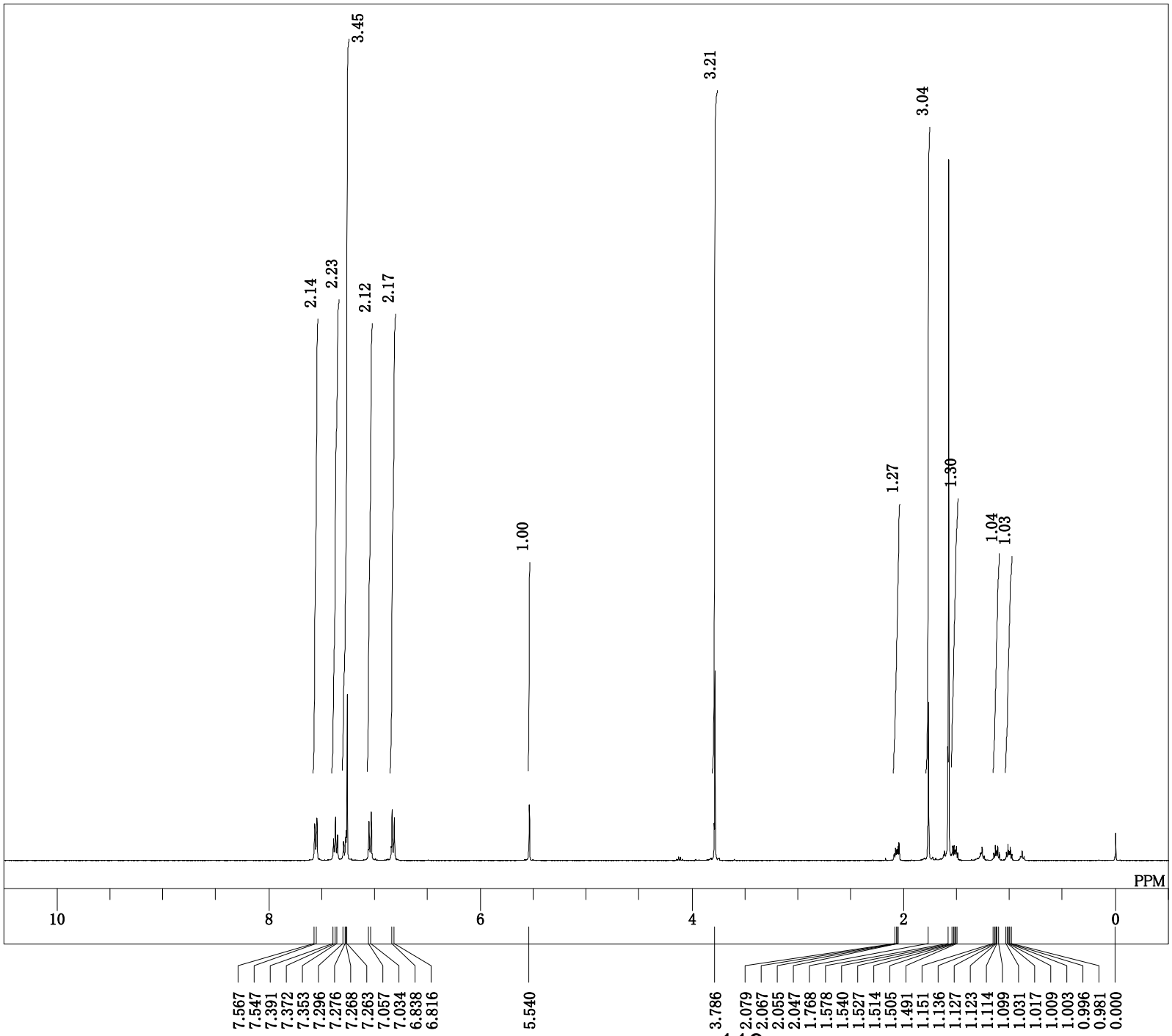


DFILE	alpha_3j_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-18 09:52:35
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	1024
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50

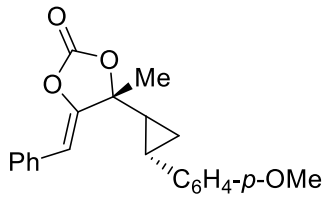




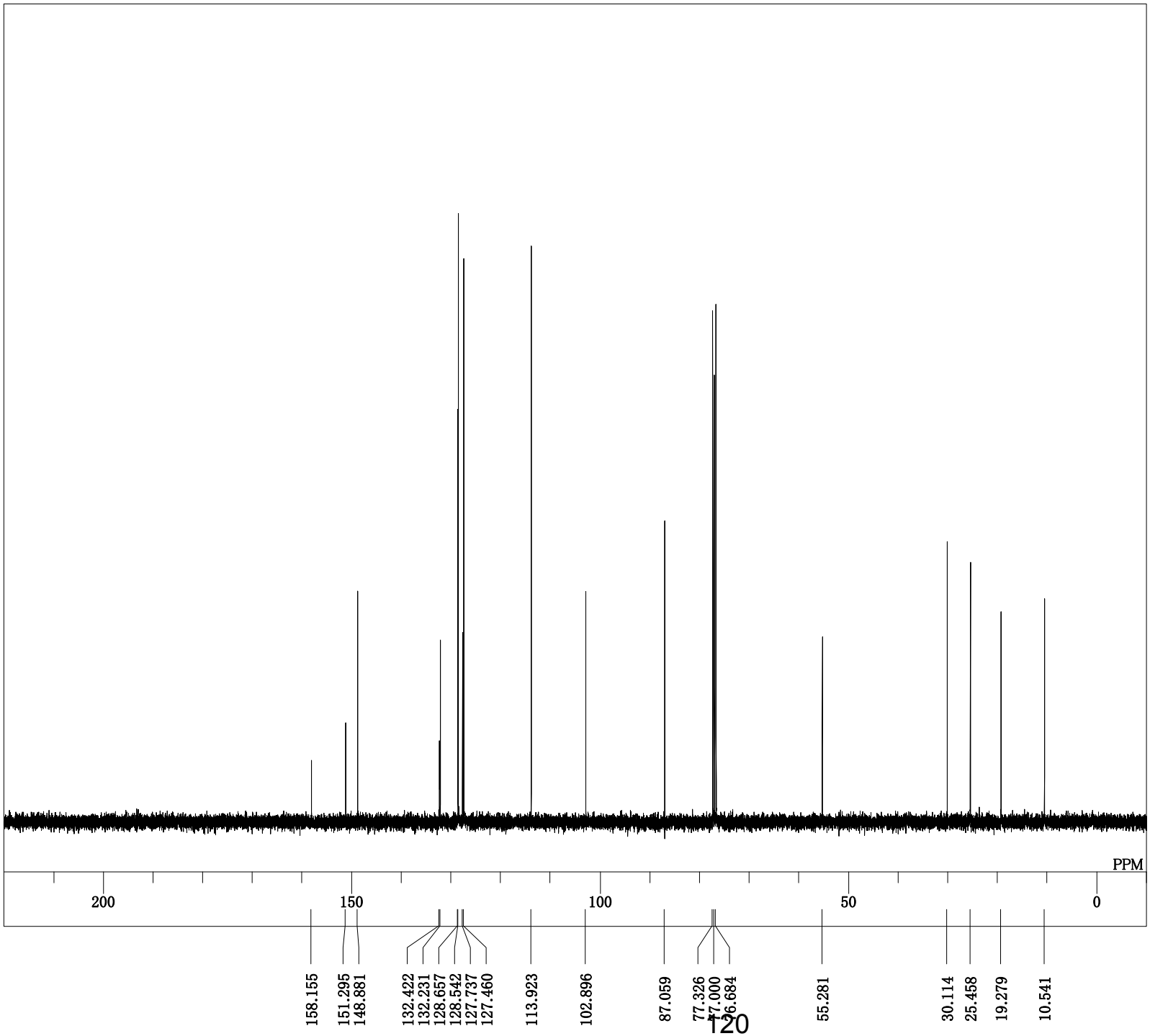
mixture of diastereomers 3j



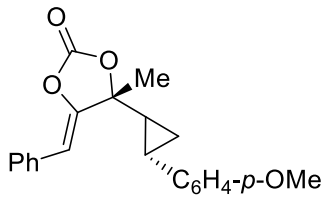
DFILE	alpha_3k_1H.als
COMNT	single_pulse
DATIM	2020-08-14 15:31:17
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	21.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	44



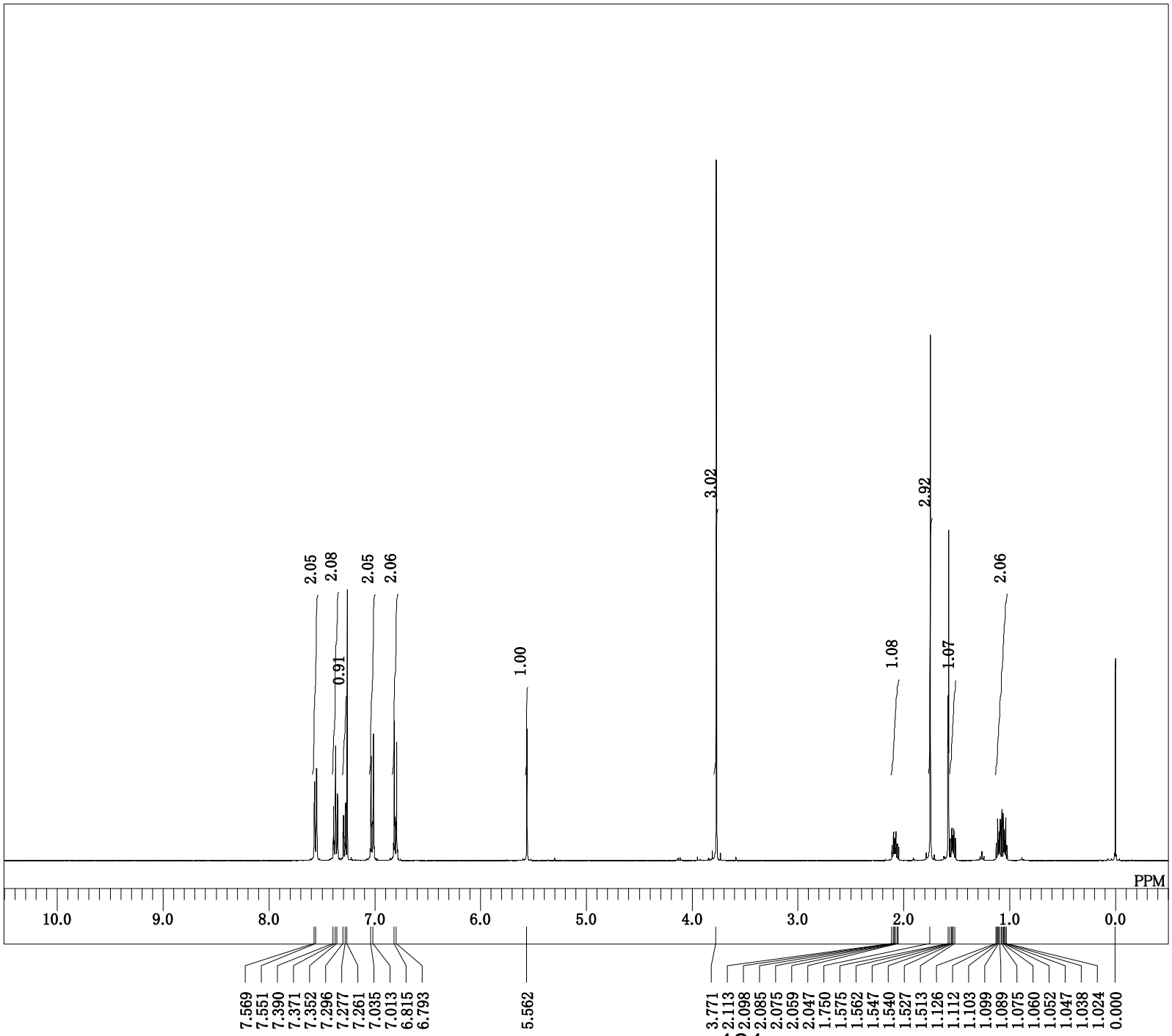
α-3k



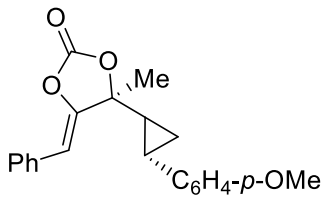
DFILE	alpha_3k_13Cals.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-18 12:24:39
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	32767
FREQU	31250.00 Hz
SCANS	256
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



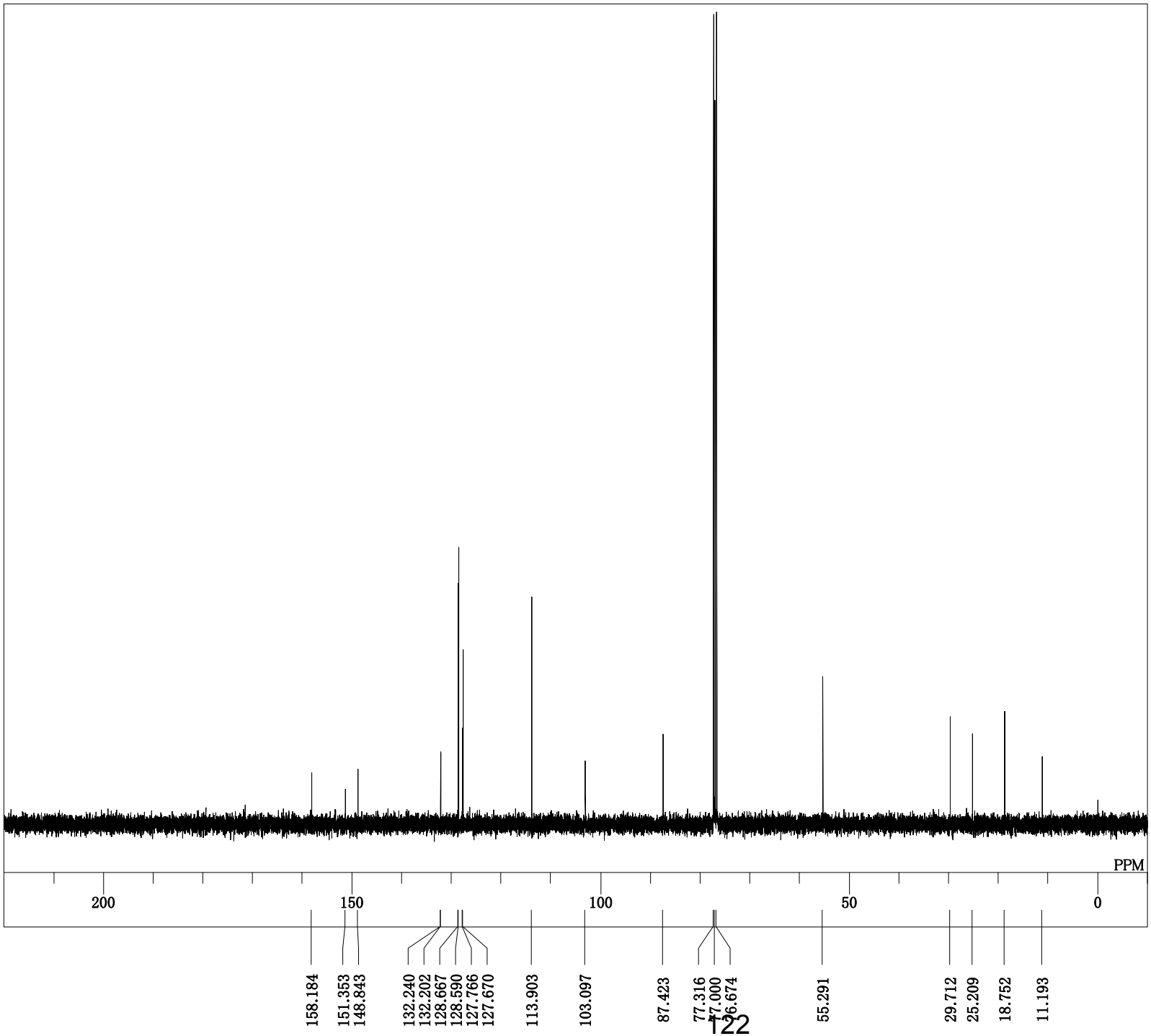
α-3k



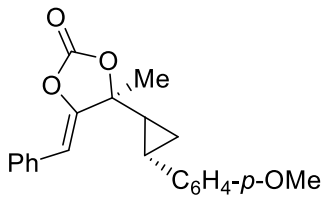
DFILE	beta_3k_1H.als
COMNT	single_pulse
DATIM	2020-08-27 10:37:49
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40



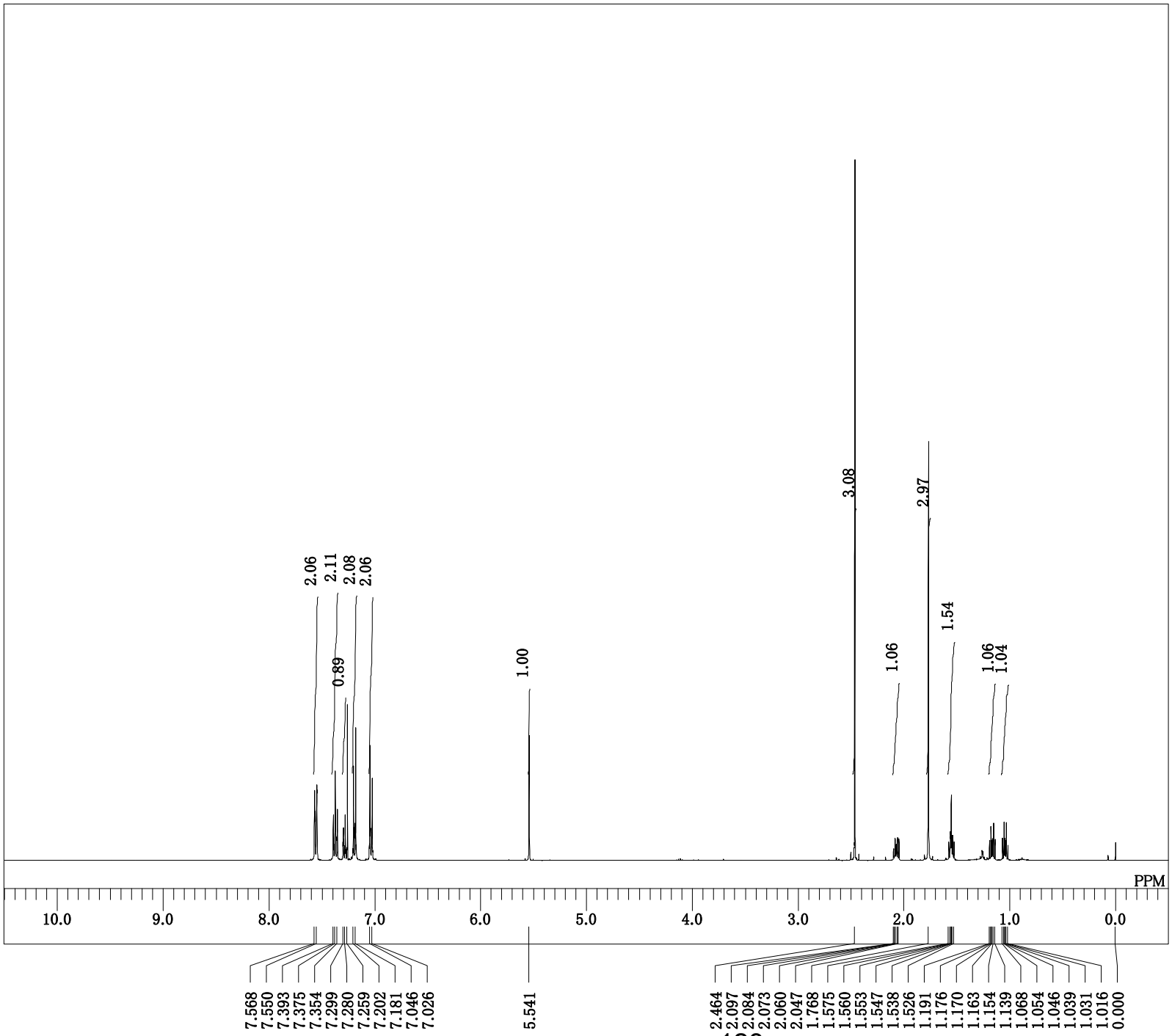
β-3k



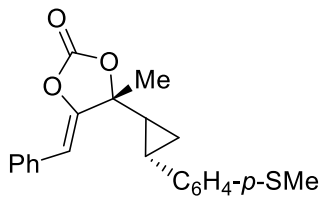
DFILE	beta_3k_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-08-27 10:39:01
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	32767
FREQU	31250.00 Hz
SCANS	512
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



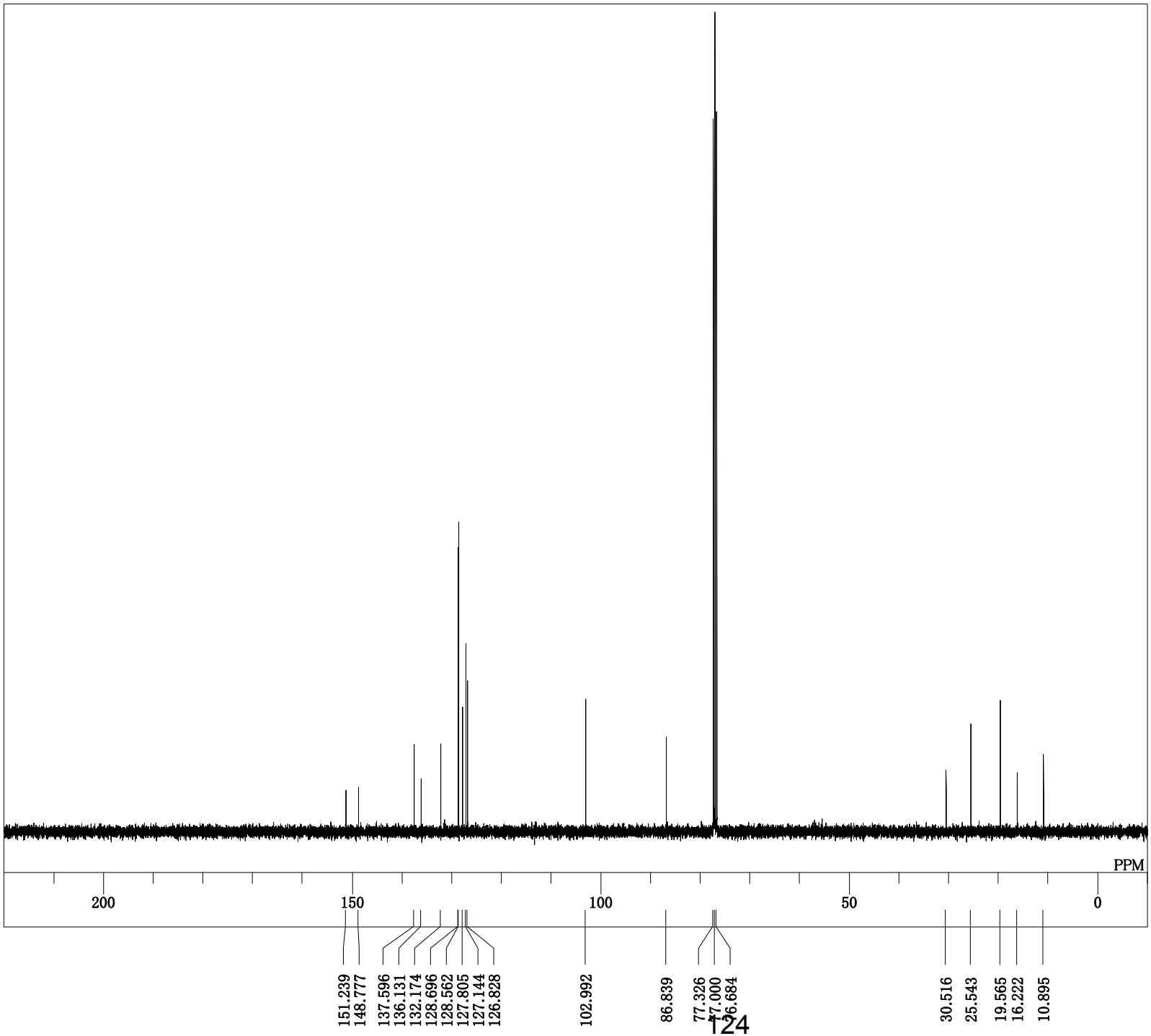
β-3k



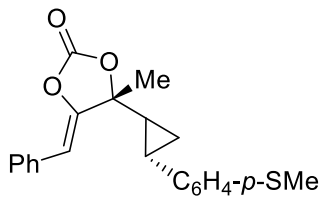
DFILE	alpha_3l_1H.als
COMNT	single_pulse
DATIM	2021-01-20 18:11:33
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	19.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40



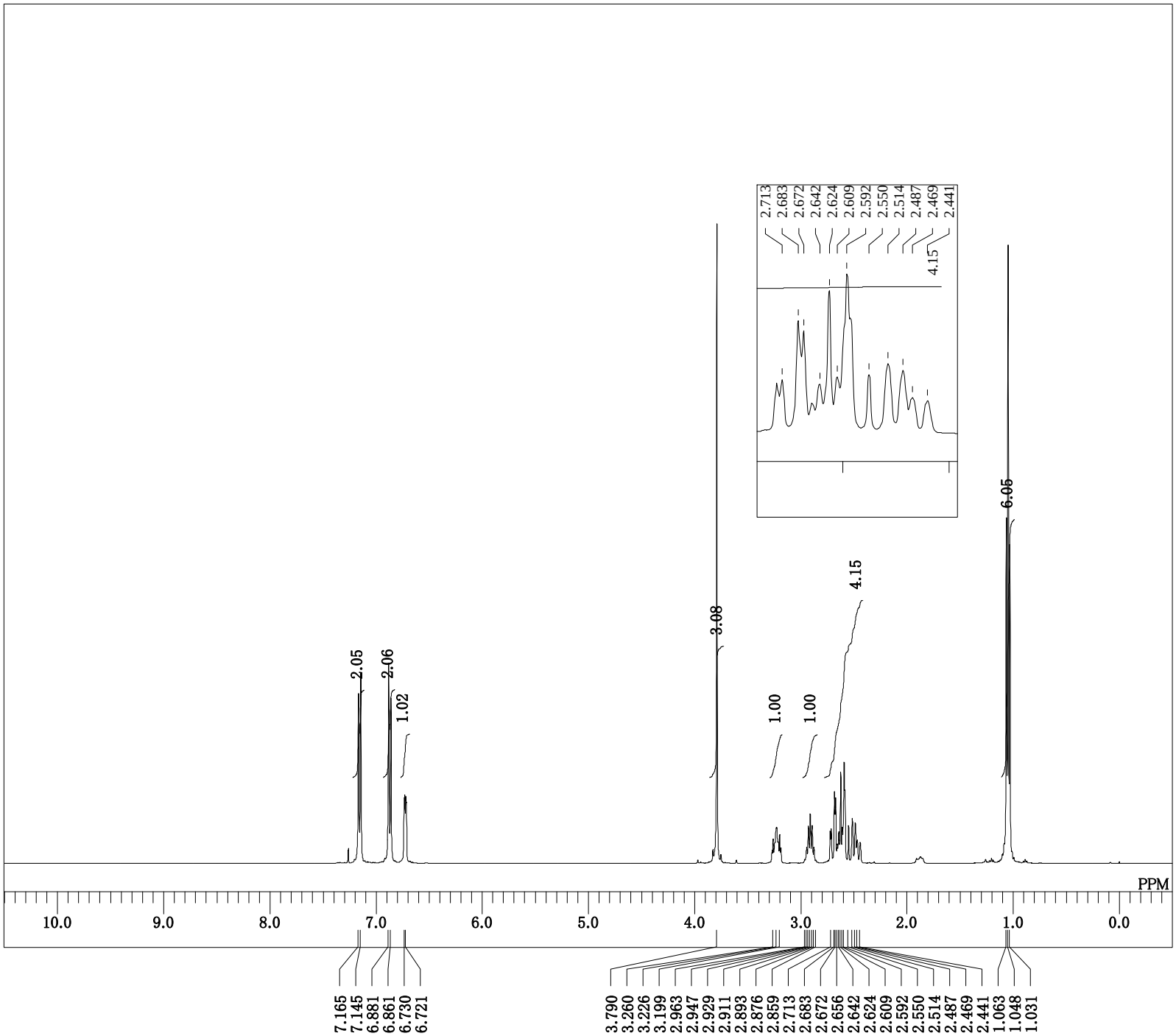
α-3l



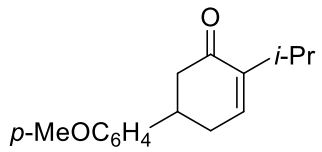
DFILE alpha_3l_13C.als
COMNT single pulse decoupled gated NOE
DATIM 2021-01-20 18:12:46
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 25000.00 Hz
SCANS 1024
ACQTM 1.0486 sec
PD 2.0000 sec
PW1 3.59 usec
IRNUC 1H
CTEMP 19.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50



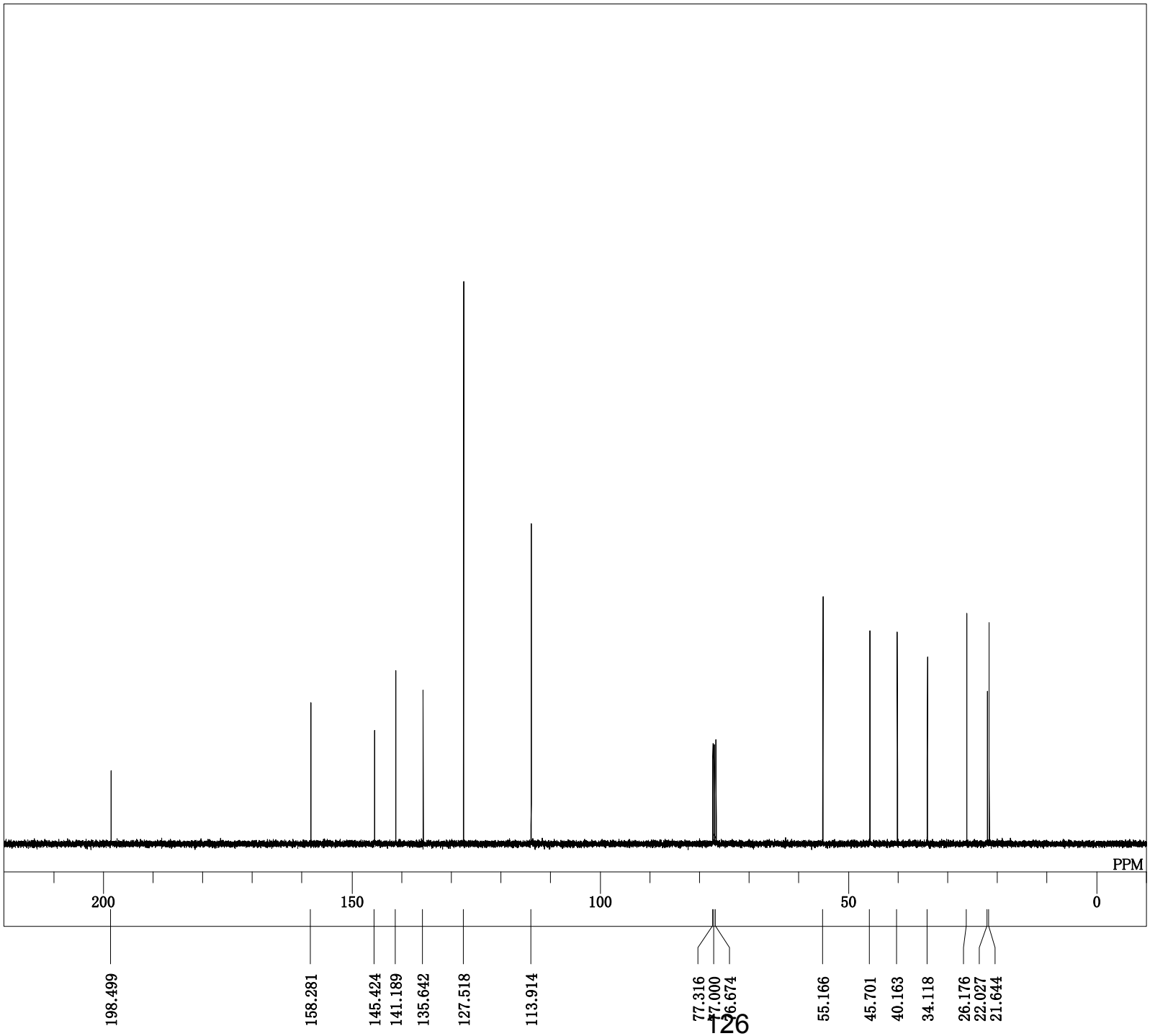
α-3l



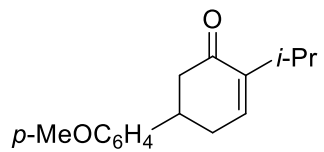
DFILE	4a_1H.als
COMNT	single_pulse
DATIM	2020-10-12 12:50:30
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	21.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	18



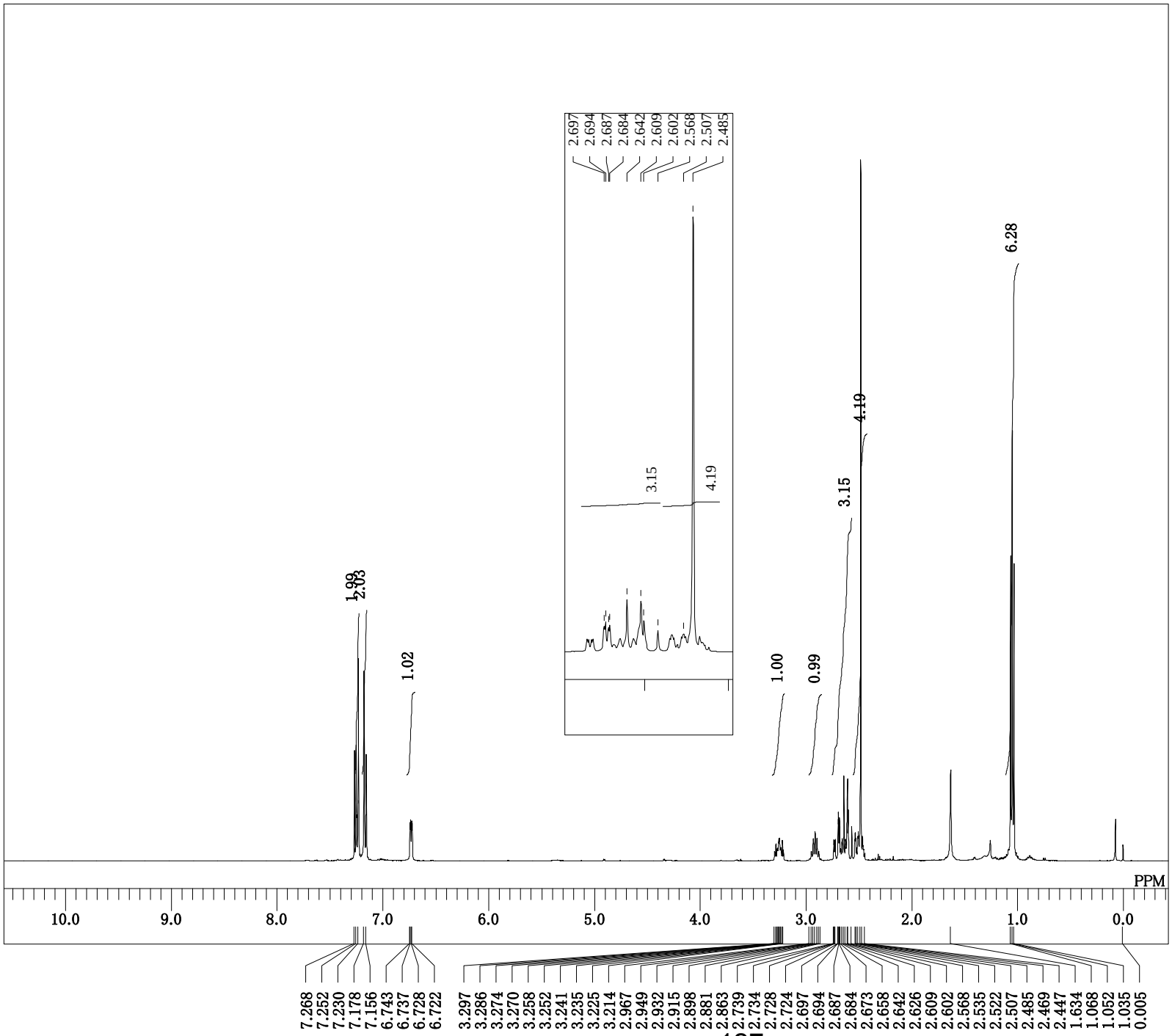
4a



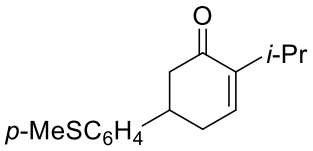
DFILE	4a_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-10-12 12:51:43
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	129
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



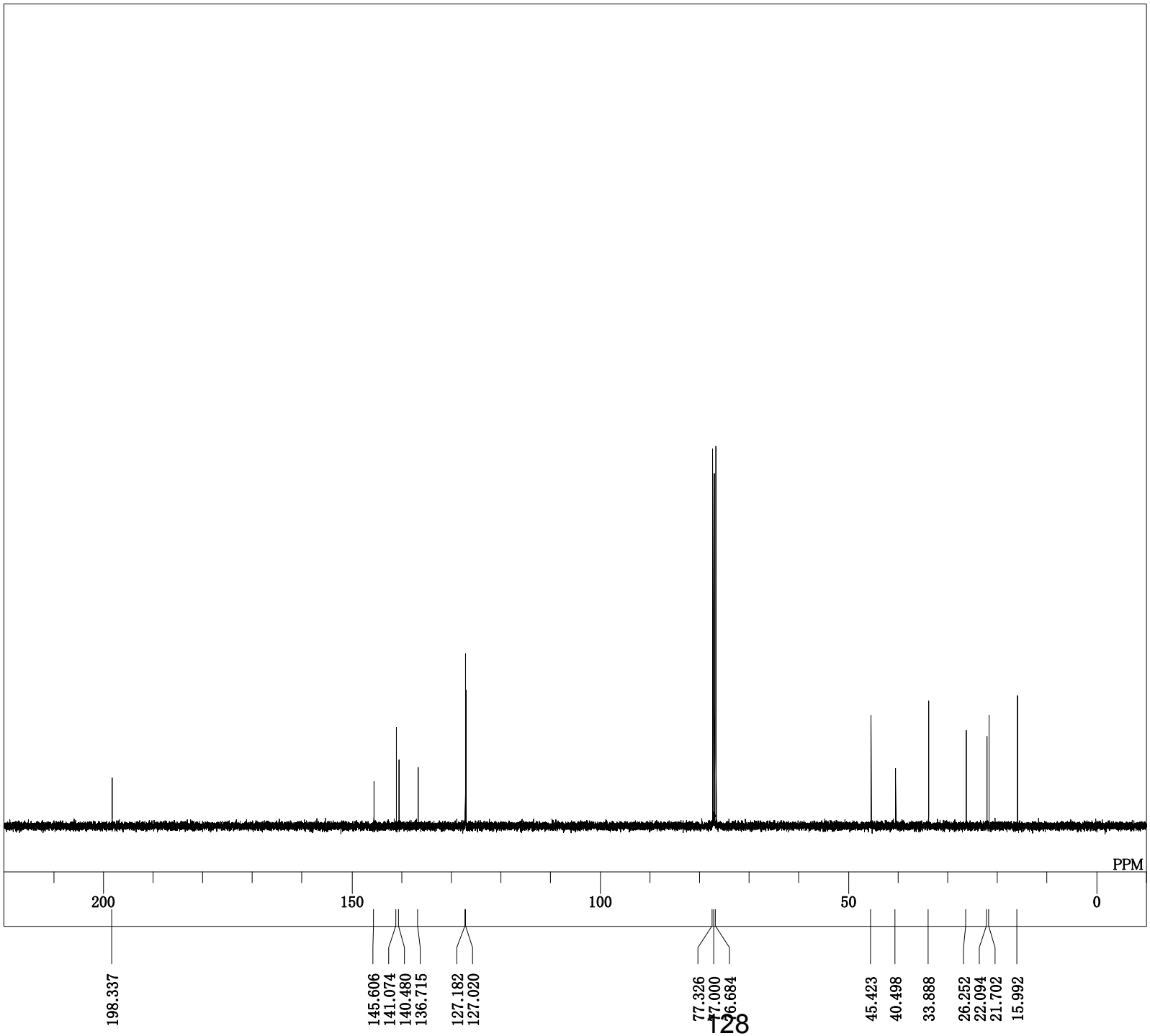
4a



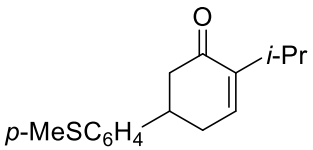
DFILE	4b_1H.als
COMNT	single_pulse
DATIM	2020-10-11 18:11:03
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	32



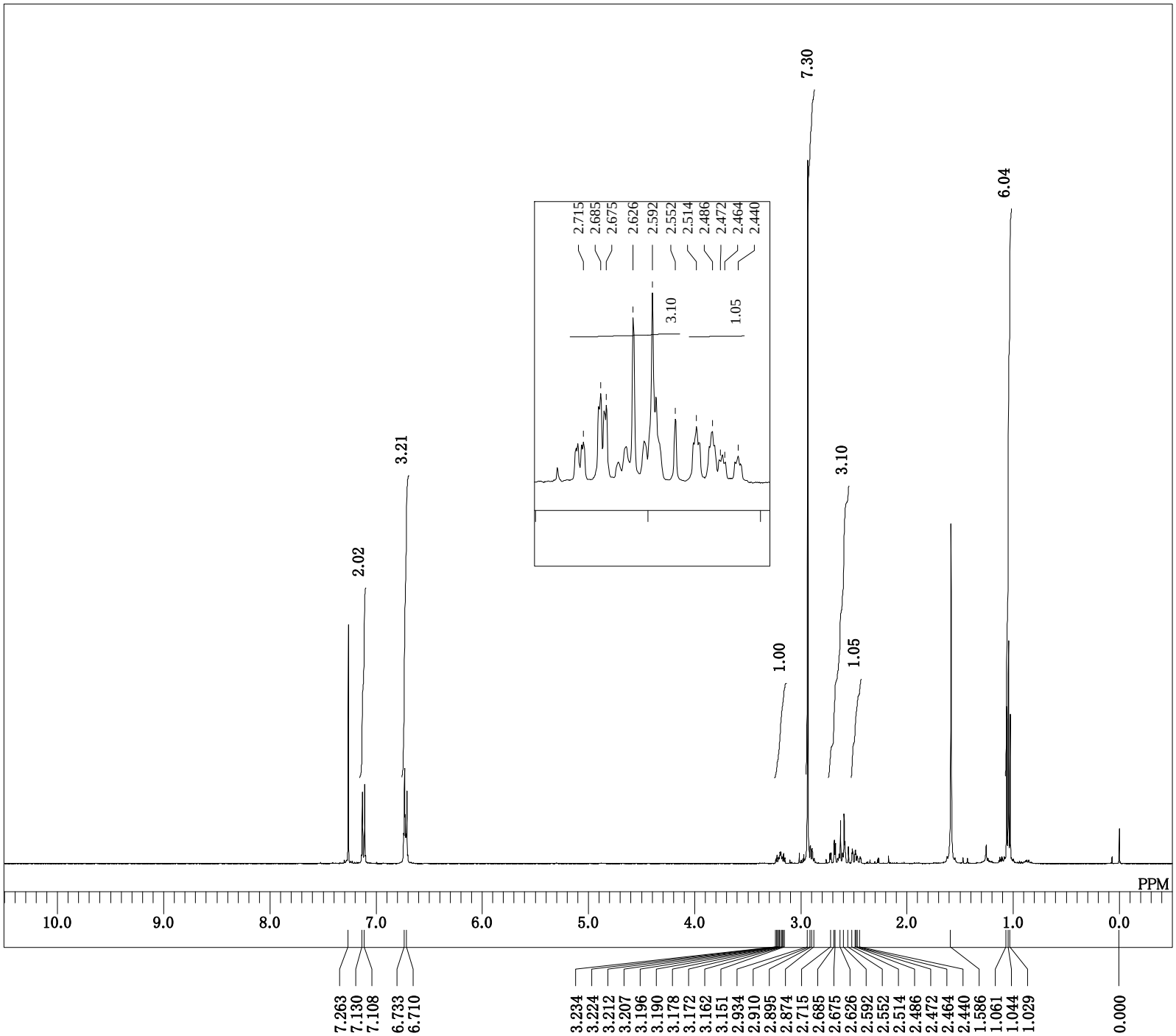
4b



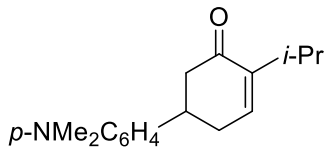
DFILE	4b_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-10-11 18:12:15
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	482
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



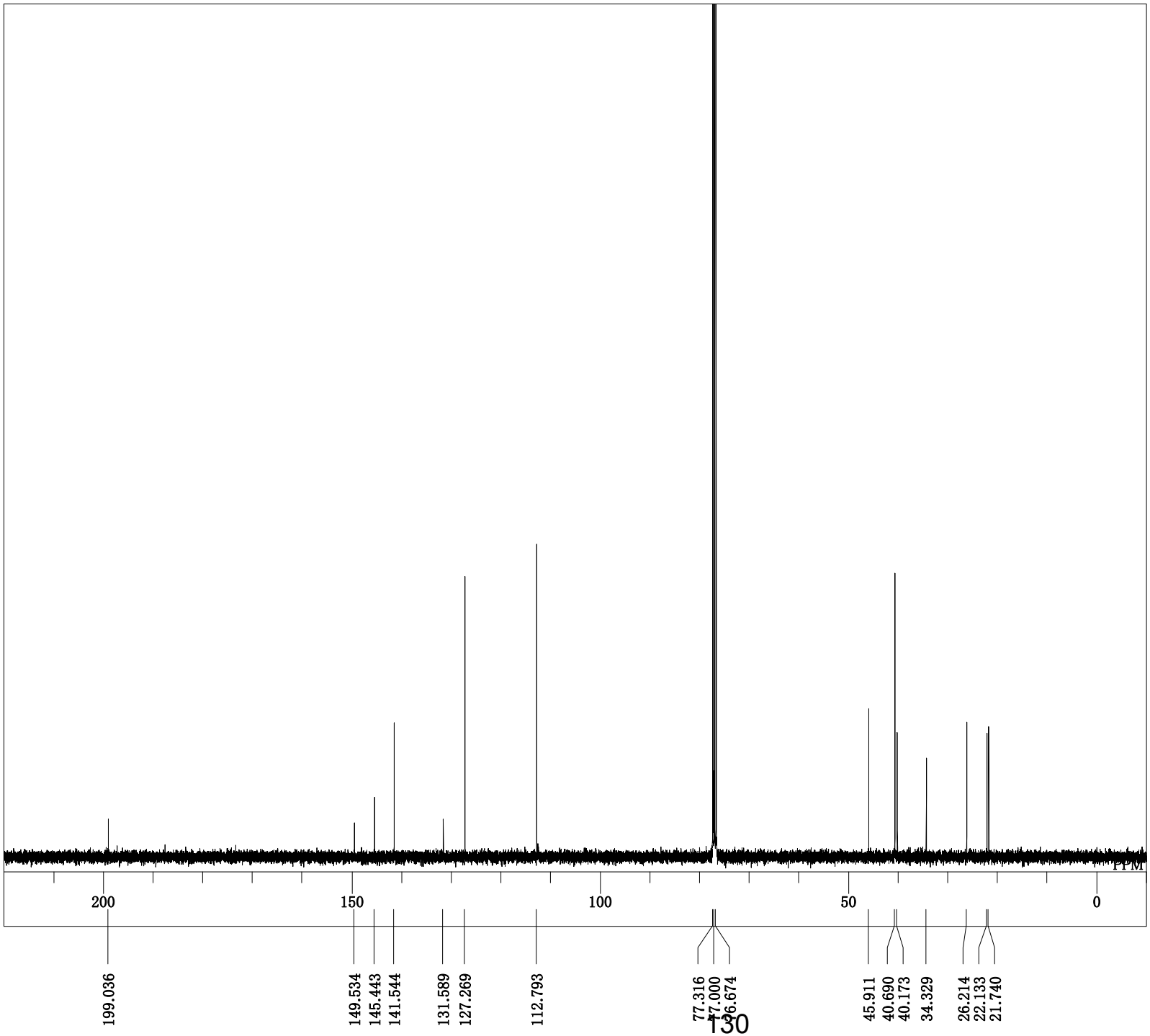
4b



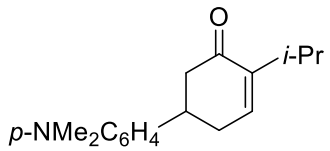
DFILE	4c_1H.als
COMNT	single_pulse
DATIM	2020-10-11 18:54:09
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	21.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	42



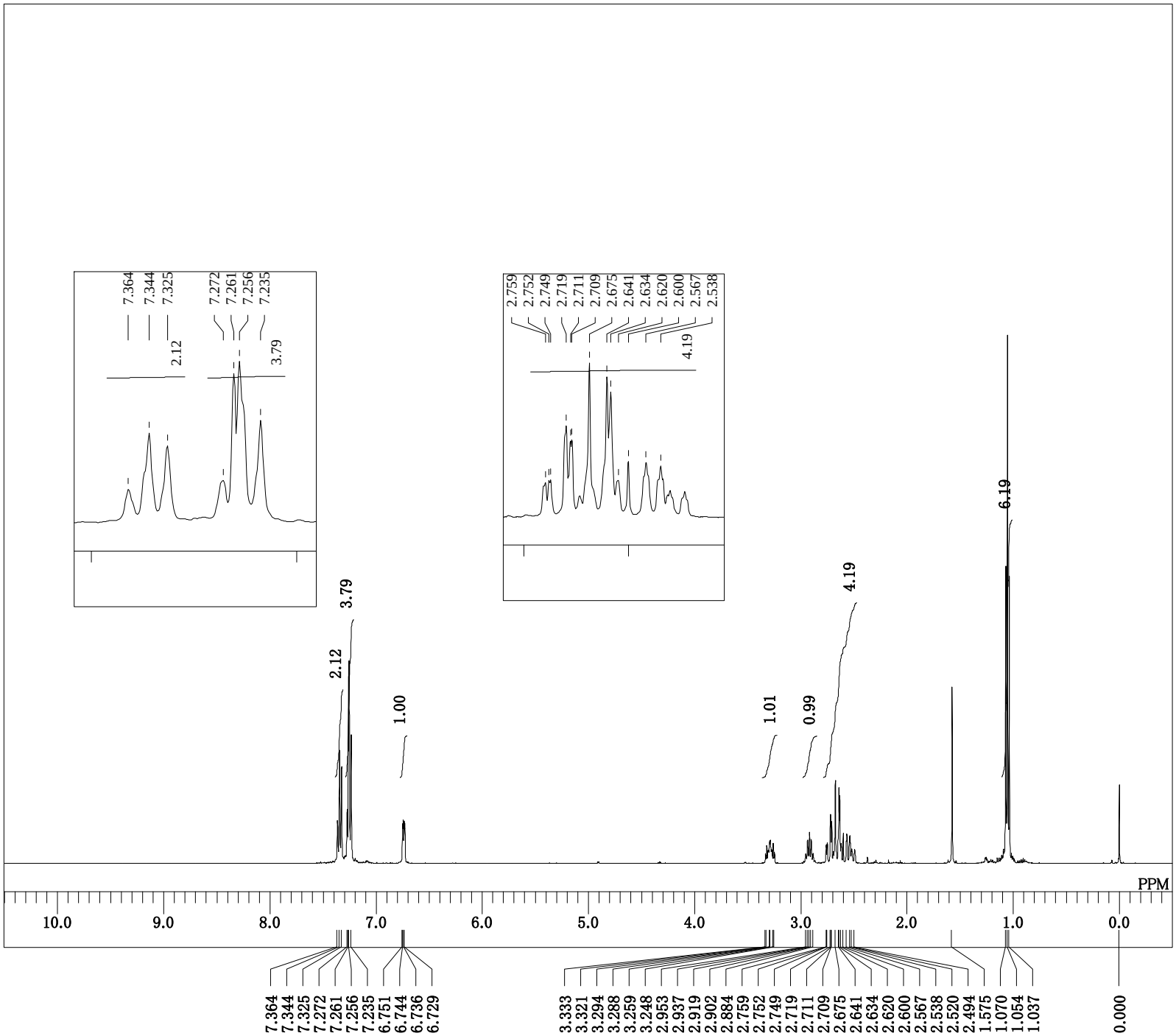
4c



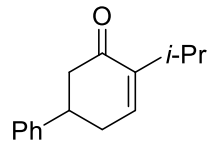
DFILE	4c_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-10-11 21:23:32
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	8192
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



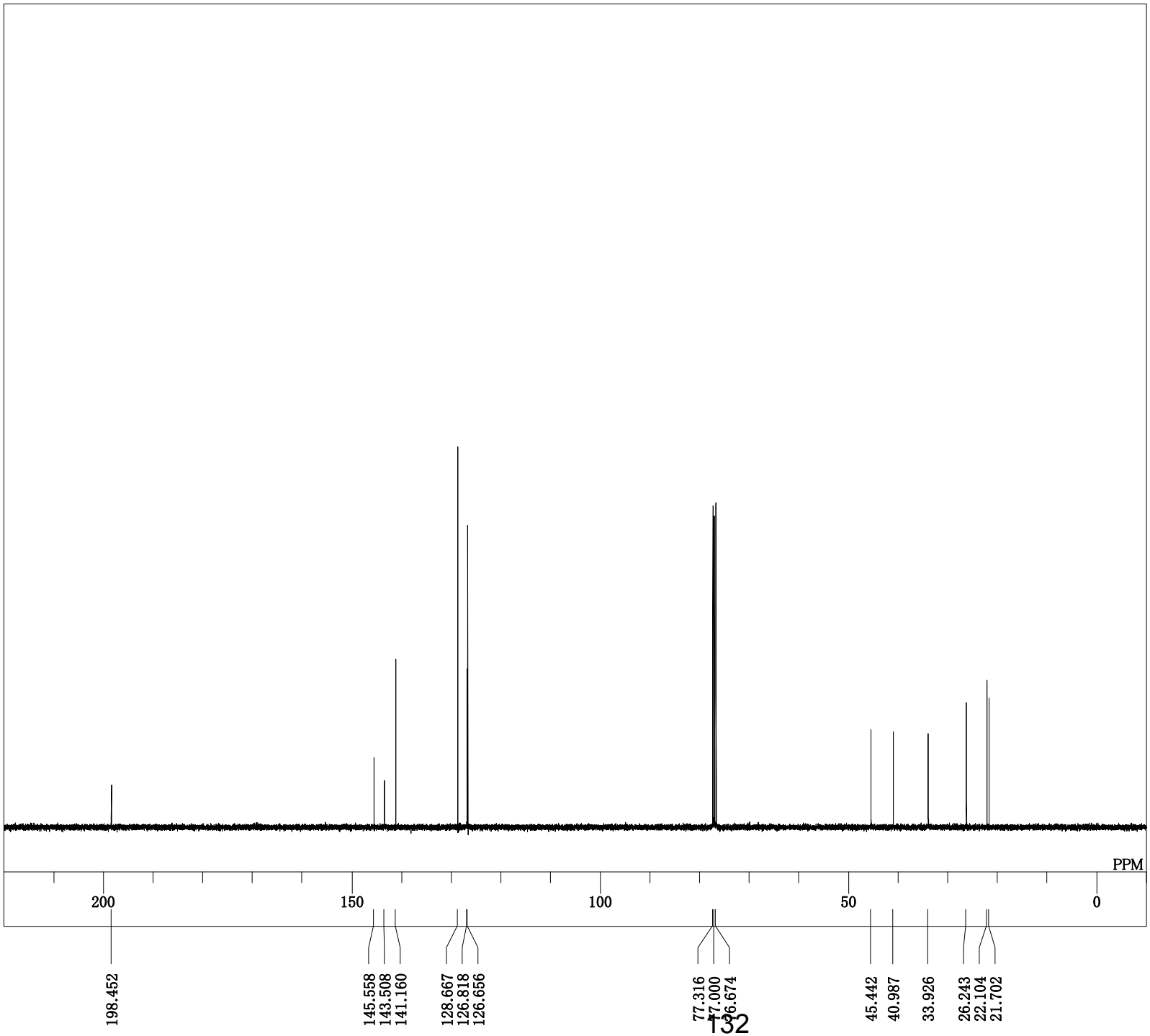
4c



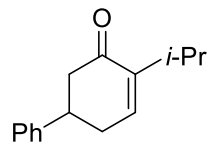
DFILE	4d_1H.als
COMNT	single_pulse
DATIM	2020-01-20 21:42:45
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	19.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40



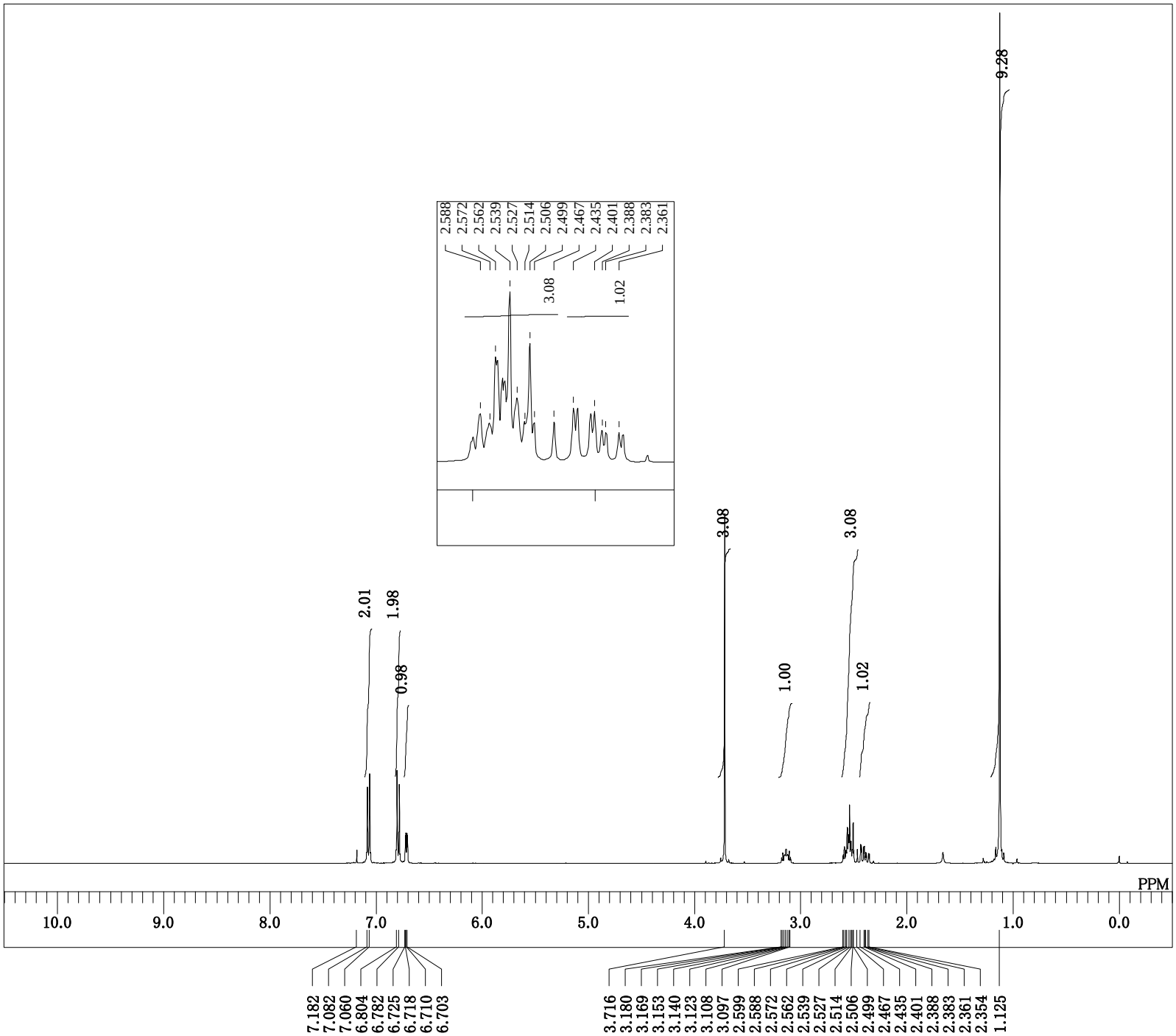
4d



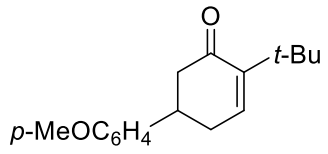
DFILE	4d_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-01 19:33:08
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	971
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



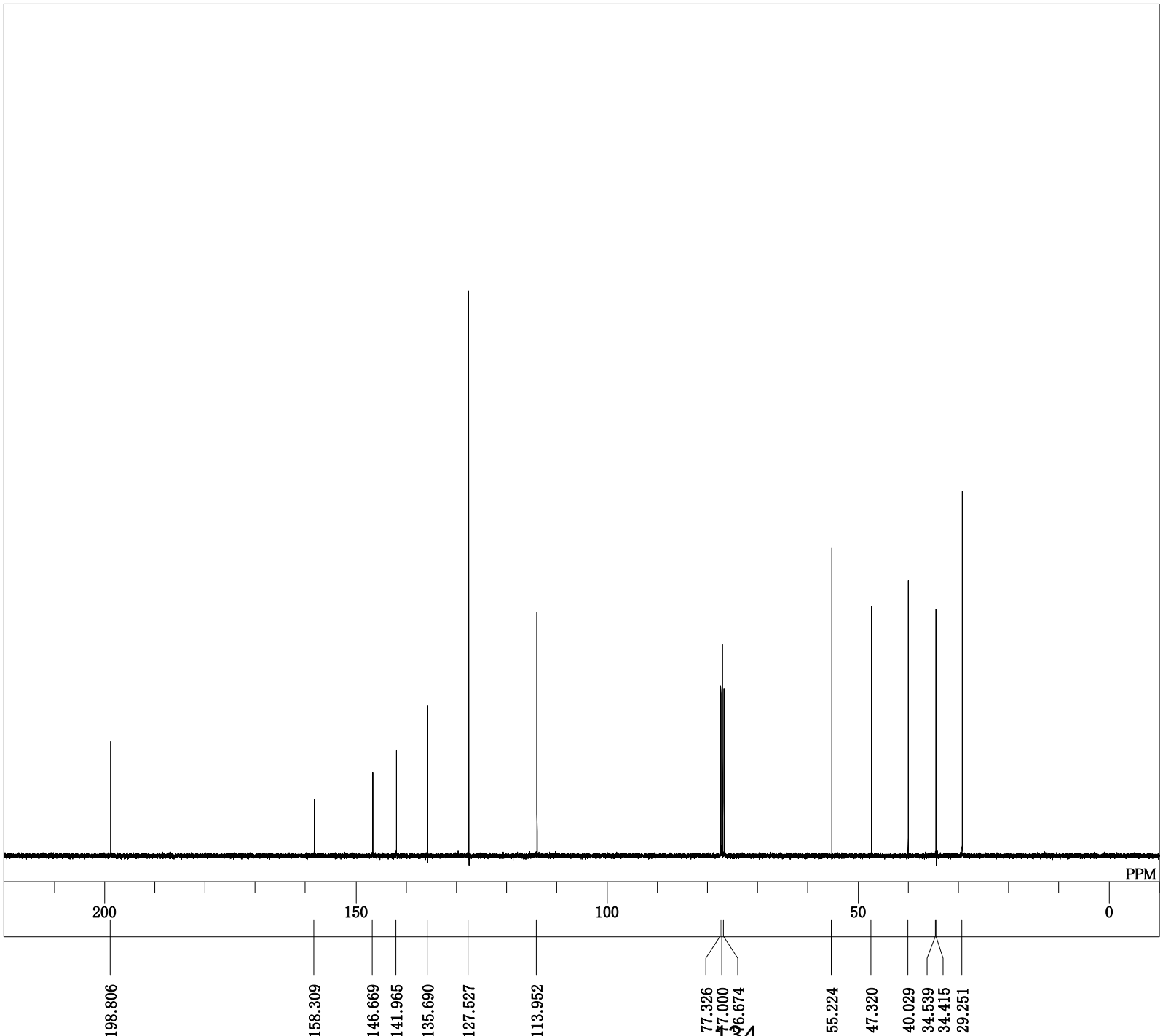
4d



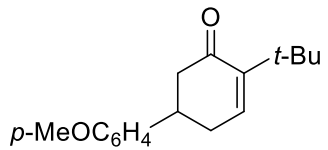
DFILE	4e_1H.als
COMNT	single_pulse
DATIM	2020-10-12 13:03:35
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	22



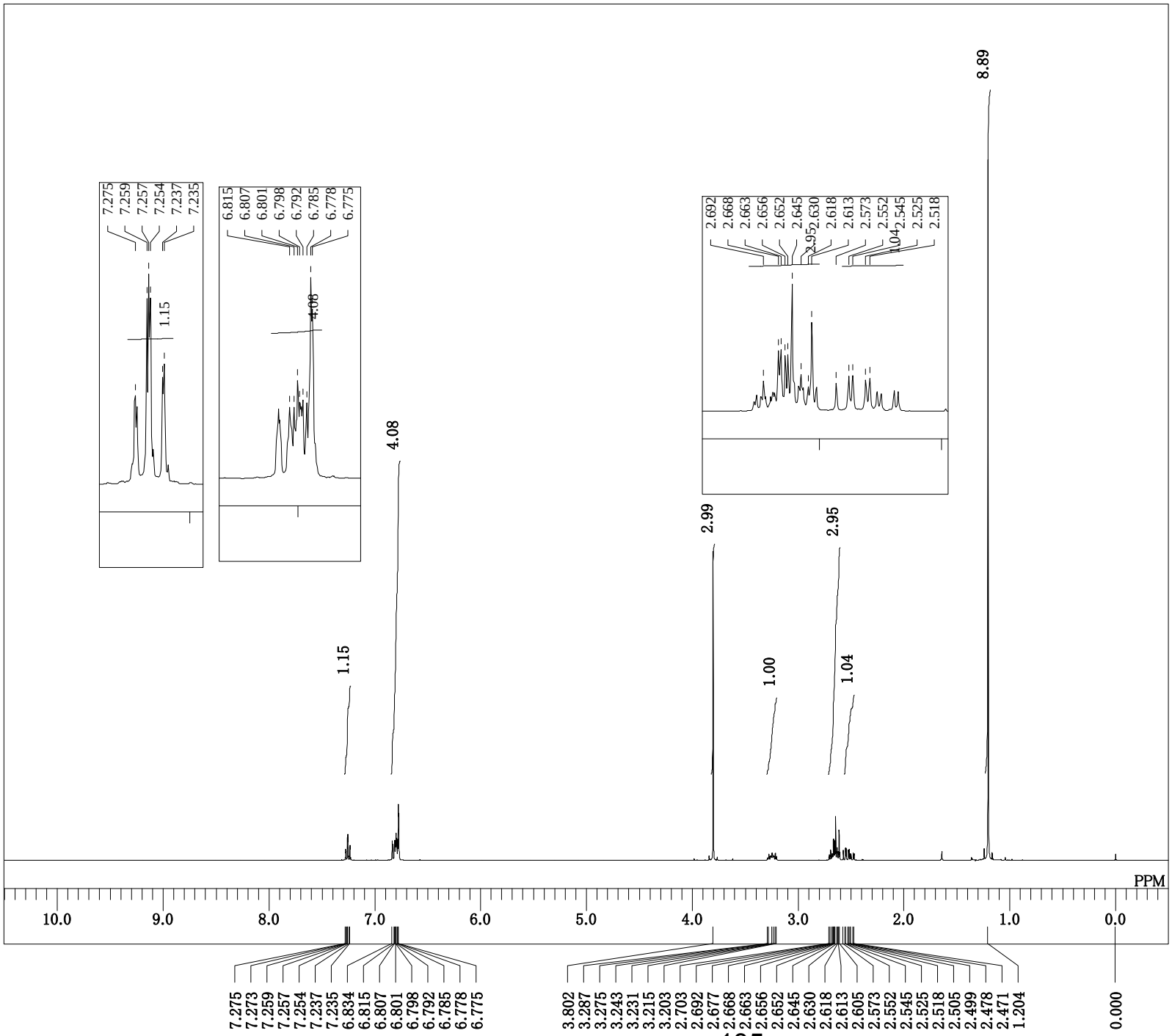
4e



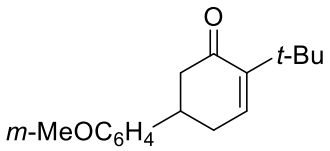
DFILE	4e_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-10-12 13:04:47
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	512
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



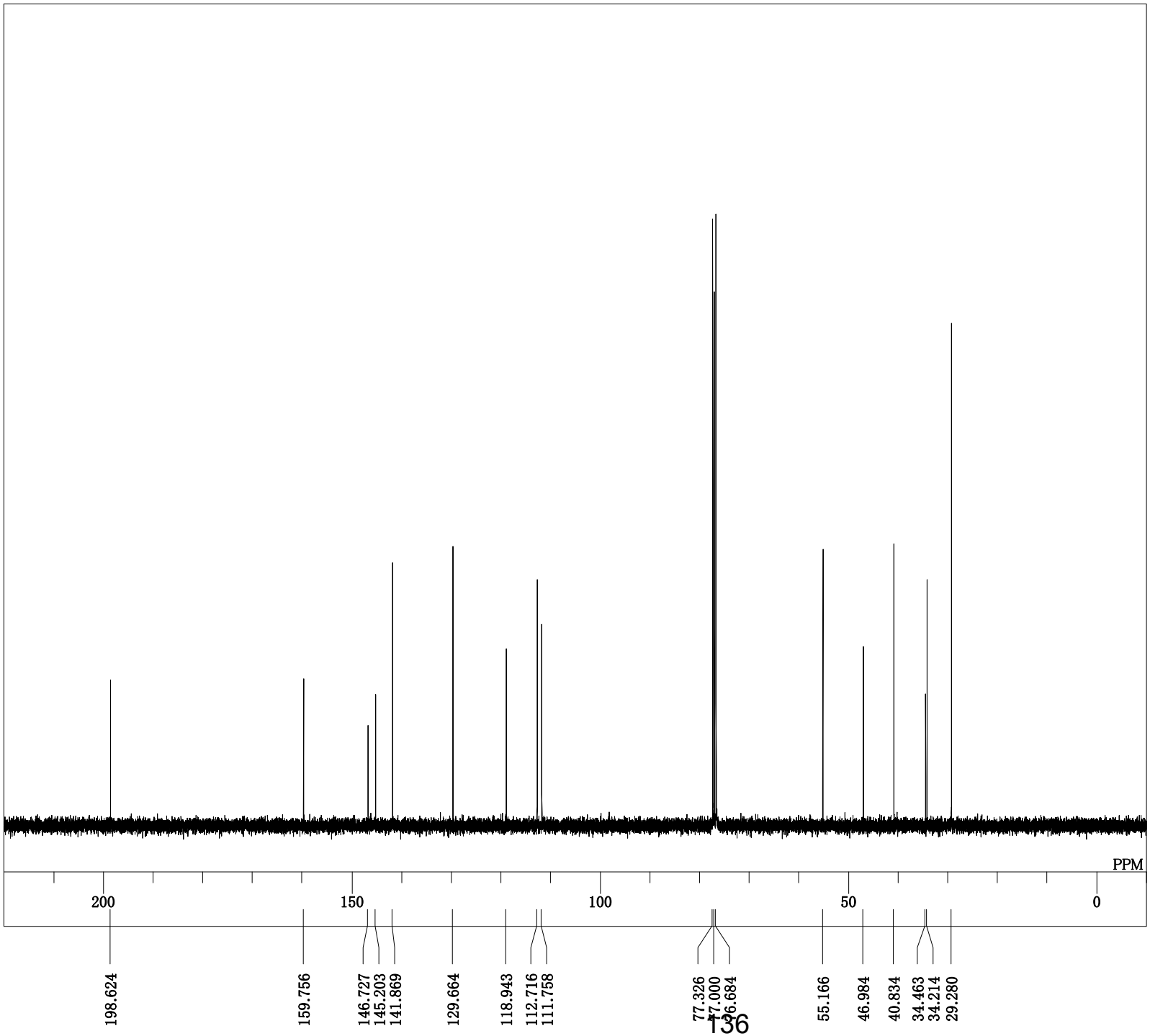
4e



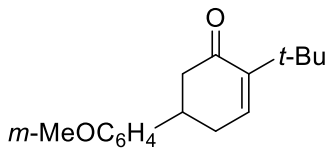
DFILE	4f_1H.als
COMNT	single_pulse
DATIM	2021-01-08 14:33:45
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	19.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	28



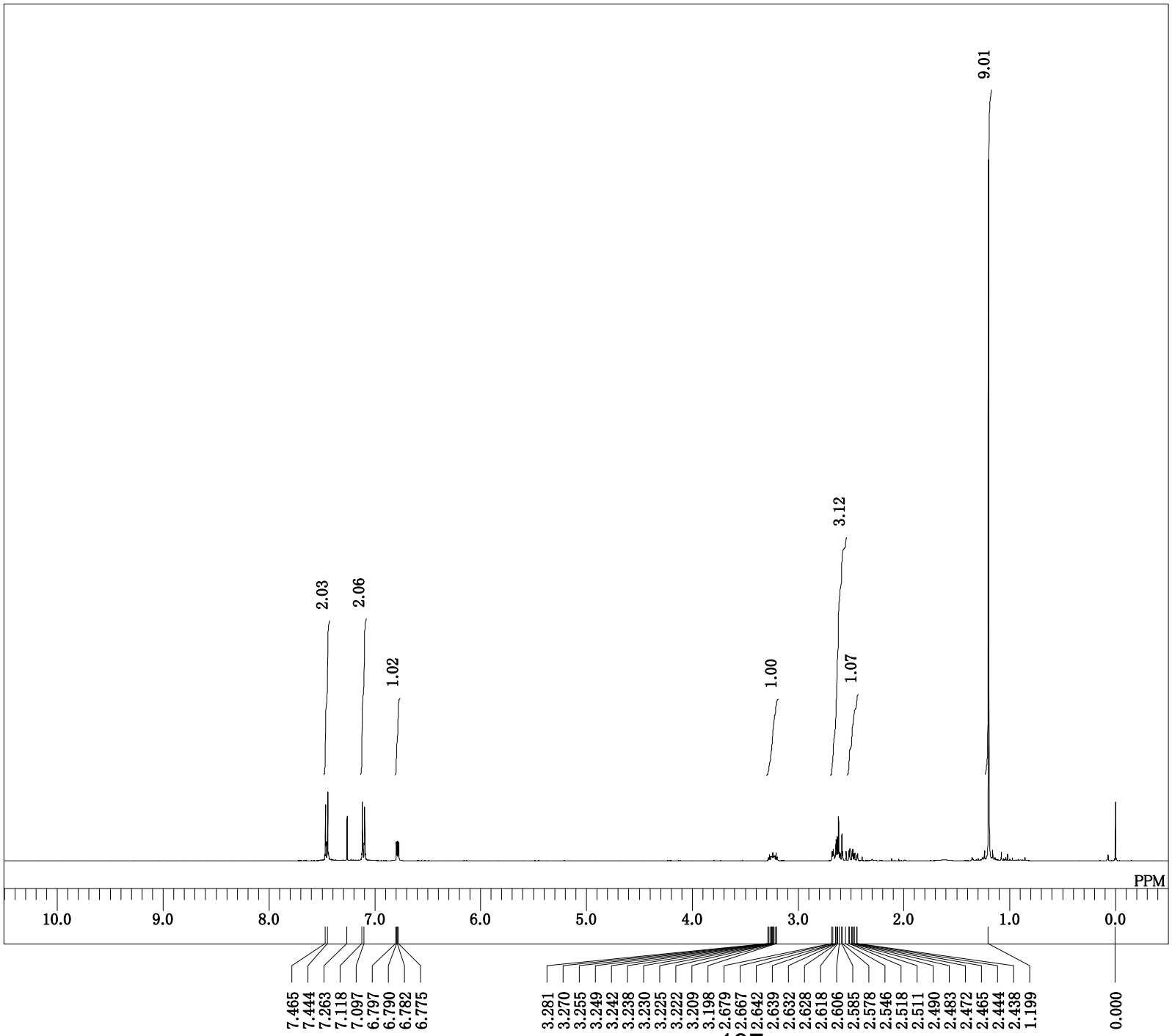
4f



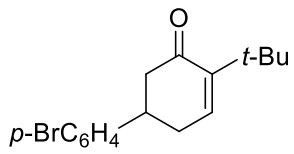
DFILE	4f_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-10-30 20:41:35
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	403
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



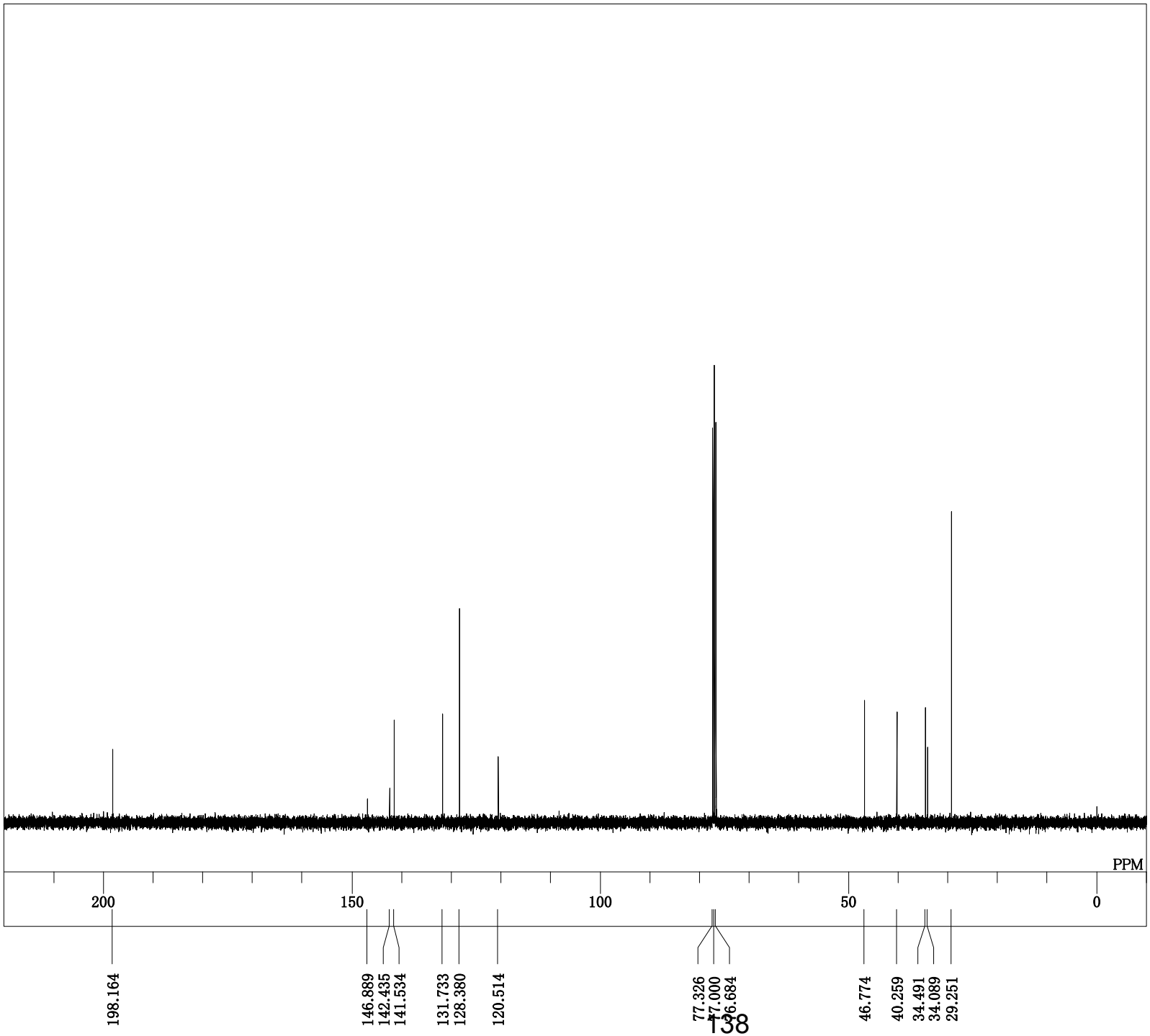
4f



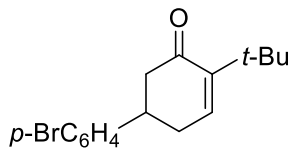
DFILE	4g_1H.als
COMNT	single_pulse
DATIM	2020-12-14 20:43:03
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	18.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	36



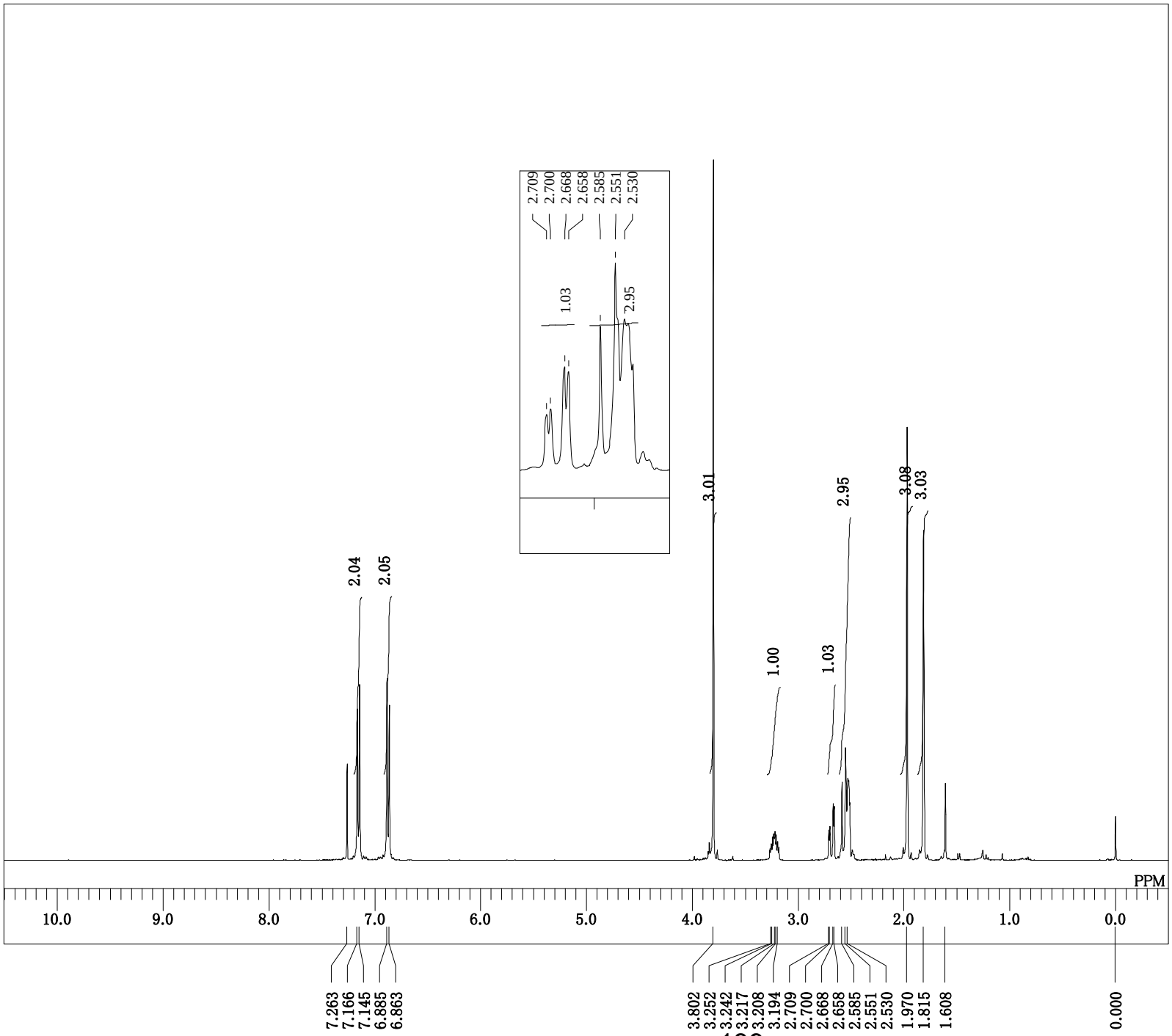
4g



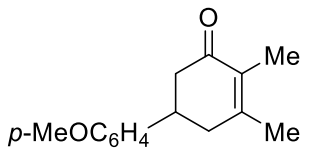
DFILE	4g_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-12-13 16:10:53
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	256
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



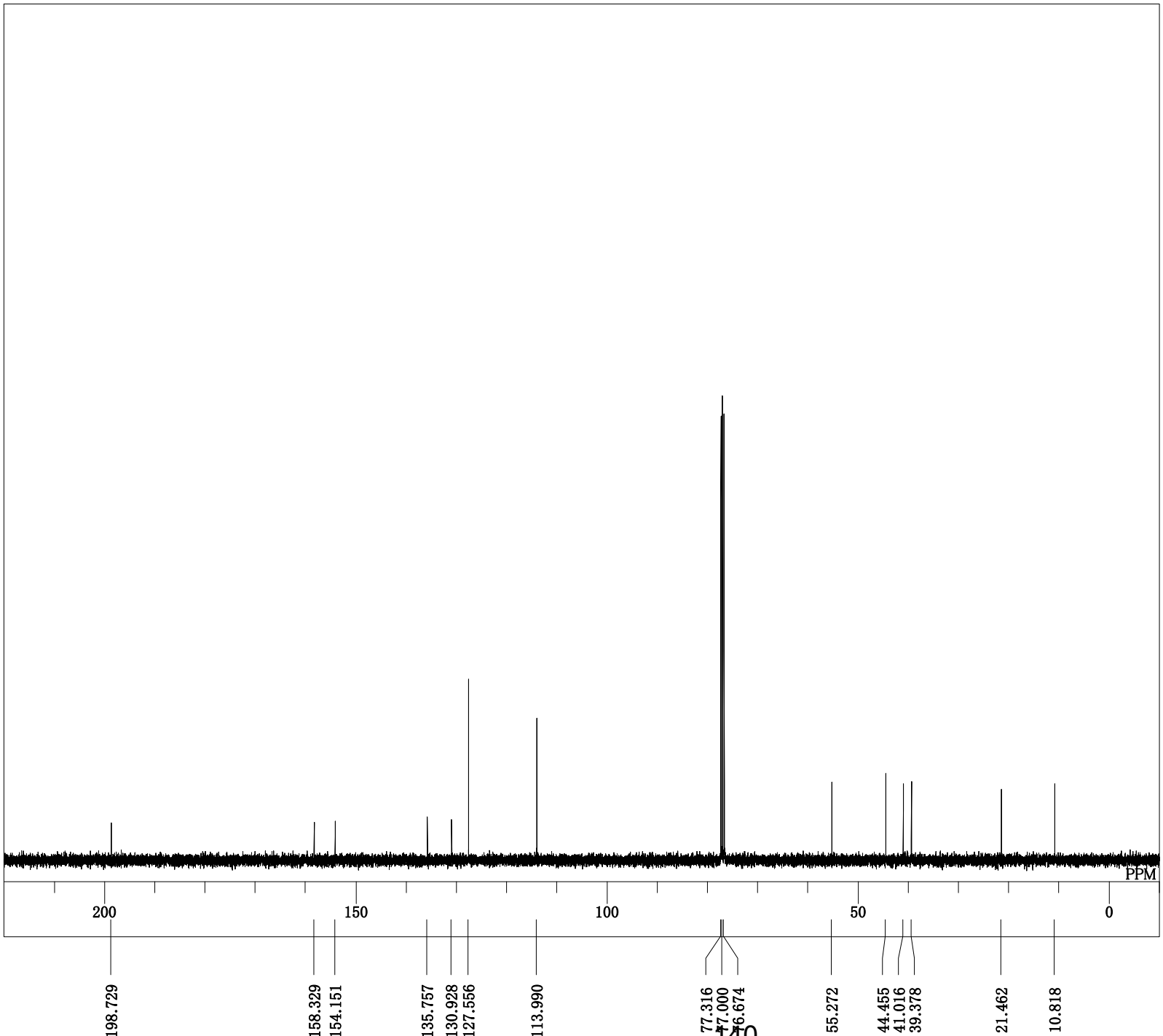
4g



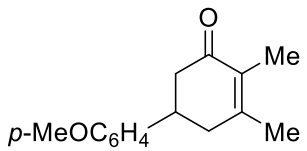
DFILE	4h_1H.als
COMNT	single_pulse
DATIM	2020-01-23 11:30:18
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.1 c
SLVNT	NONE
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	36



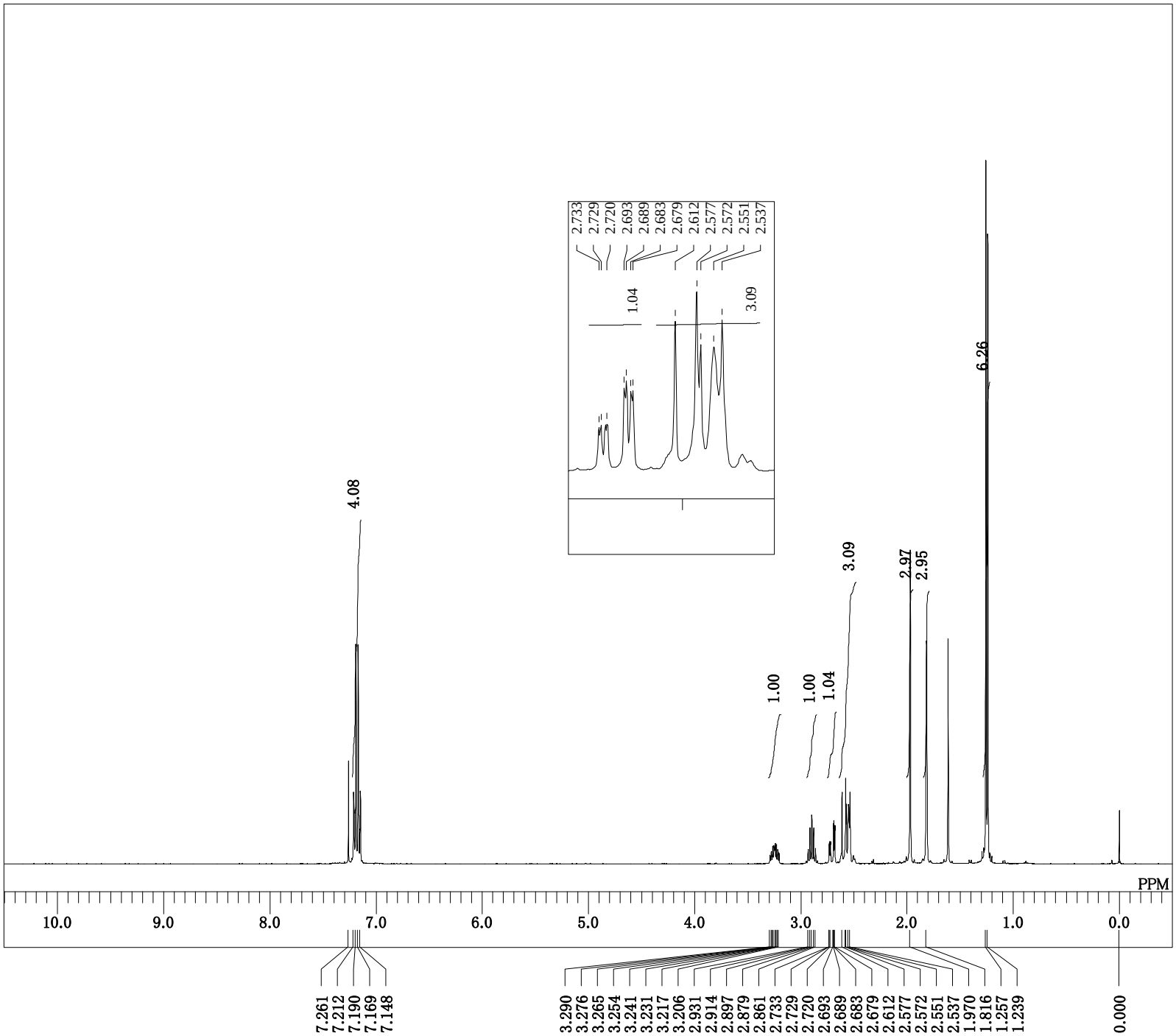
4h



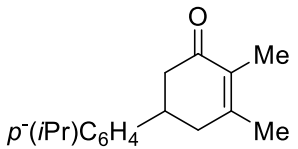
DFILE	4h_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-01-23 11:36:09
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	500
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	NONE
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



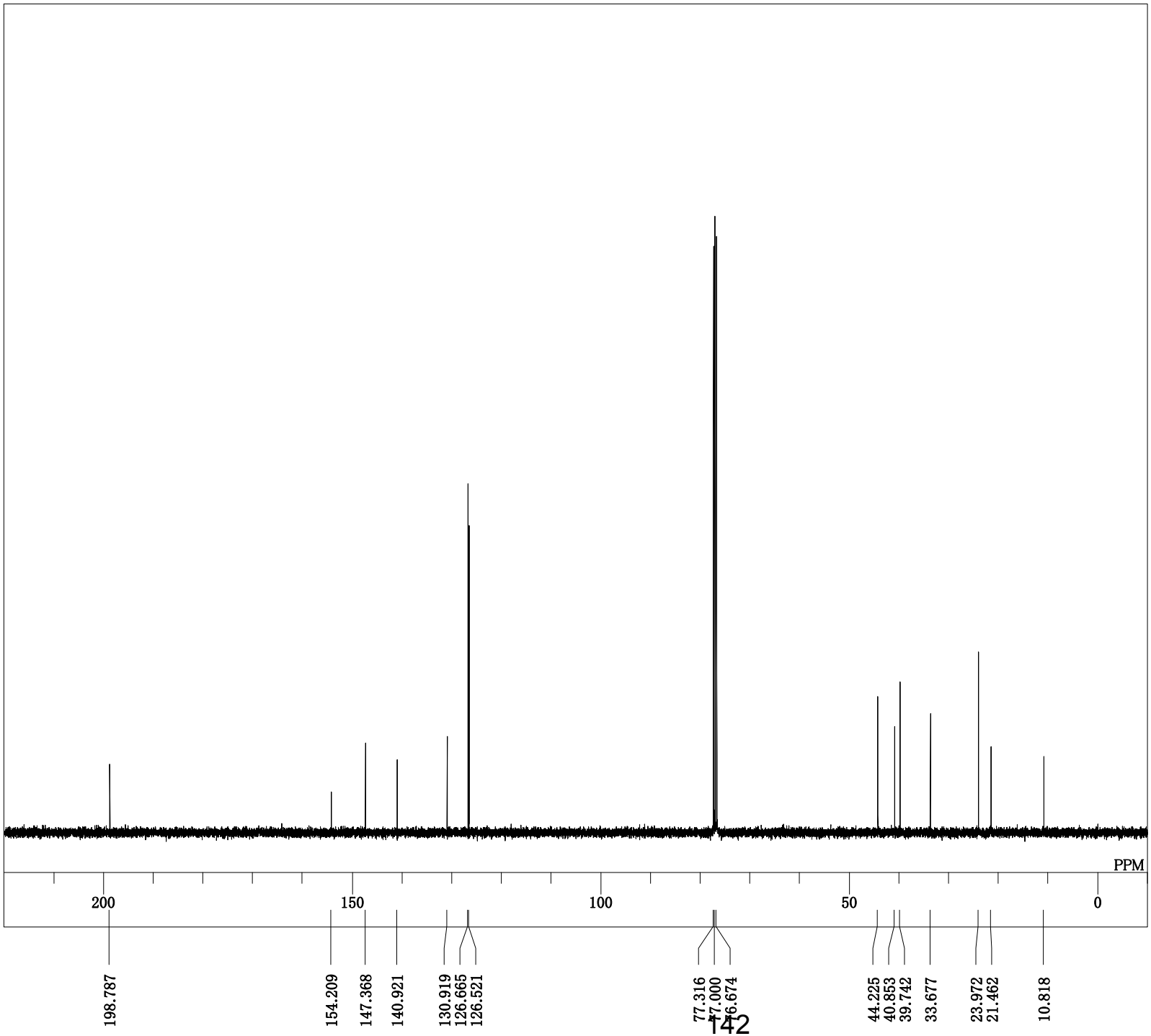
4h



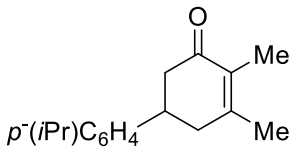
DFILE	4i_1H.als
COMNT	single_pulse
DATIM	2020-11-19 19:40:25
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	36



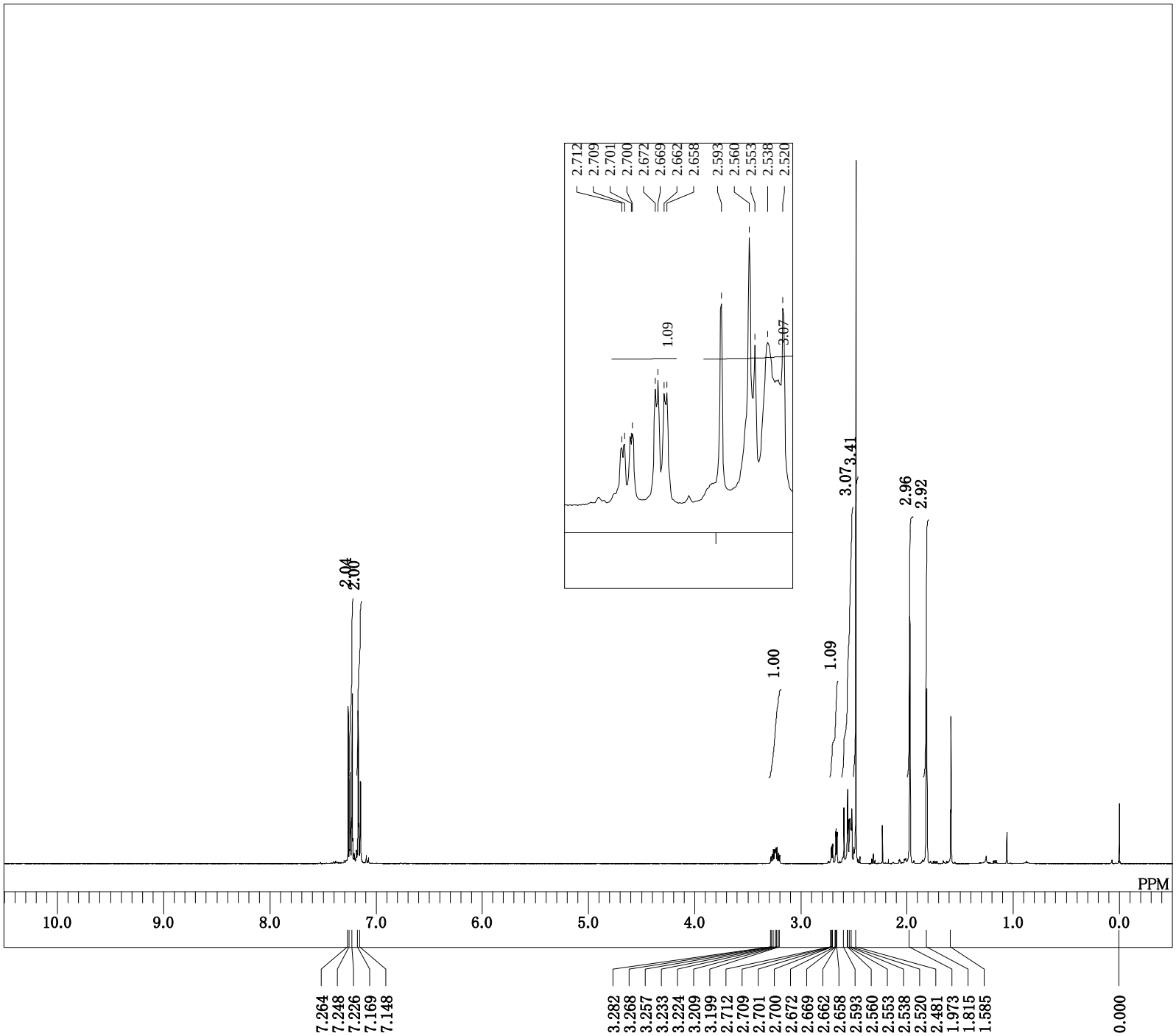
4i



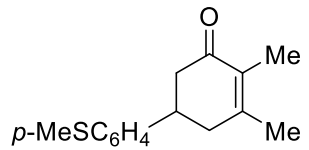
DFILE	4i_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-19 19:41:36
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	1024
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



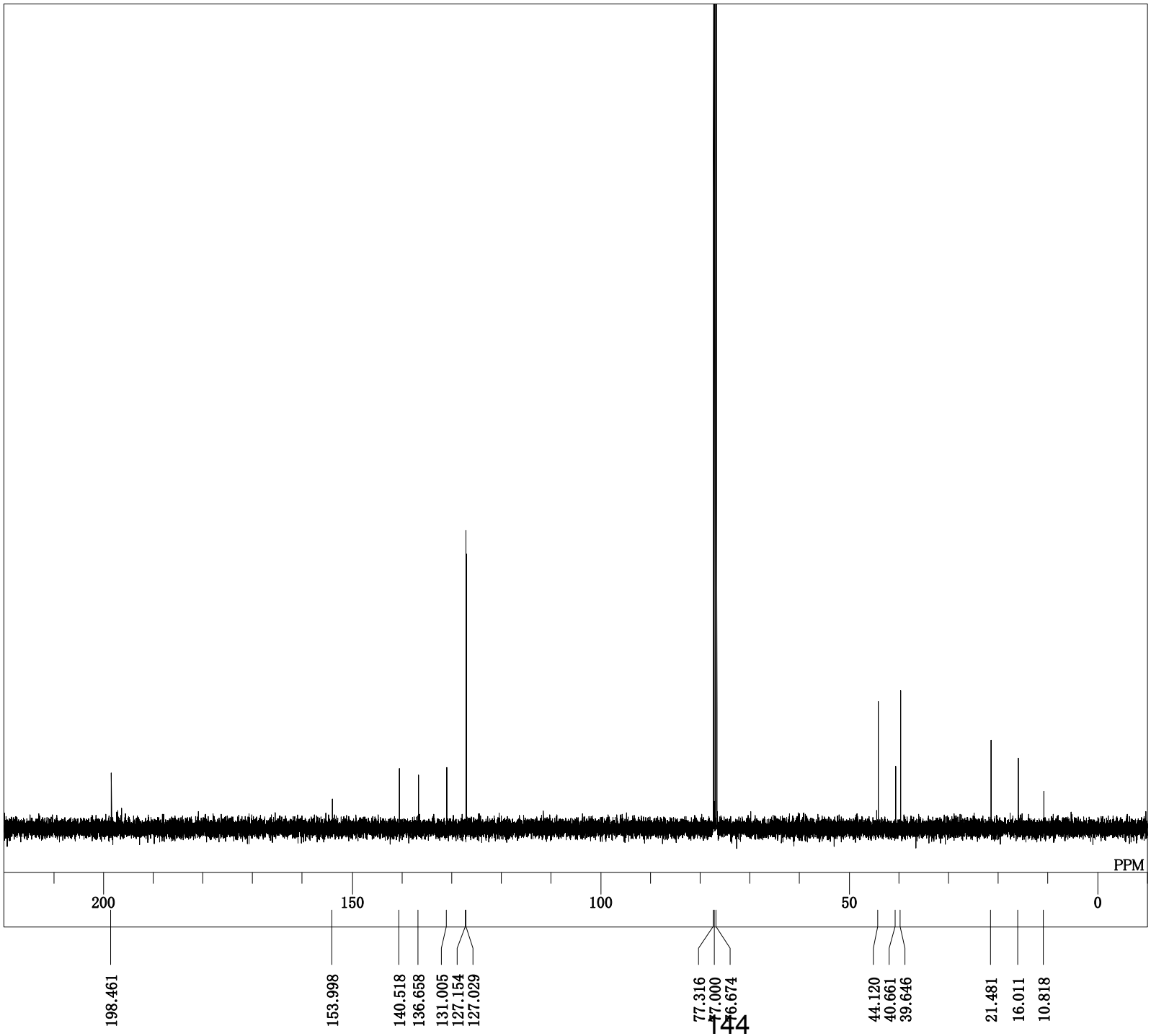
4i



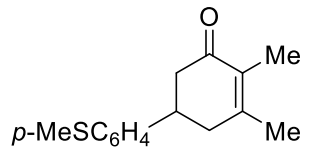
DFILE	4j_1H.als
COMNT	single_pulse
DATIM	2020-11-24 14:56:09
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	19.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40



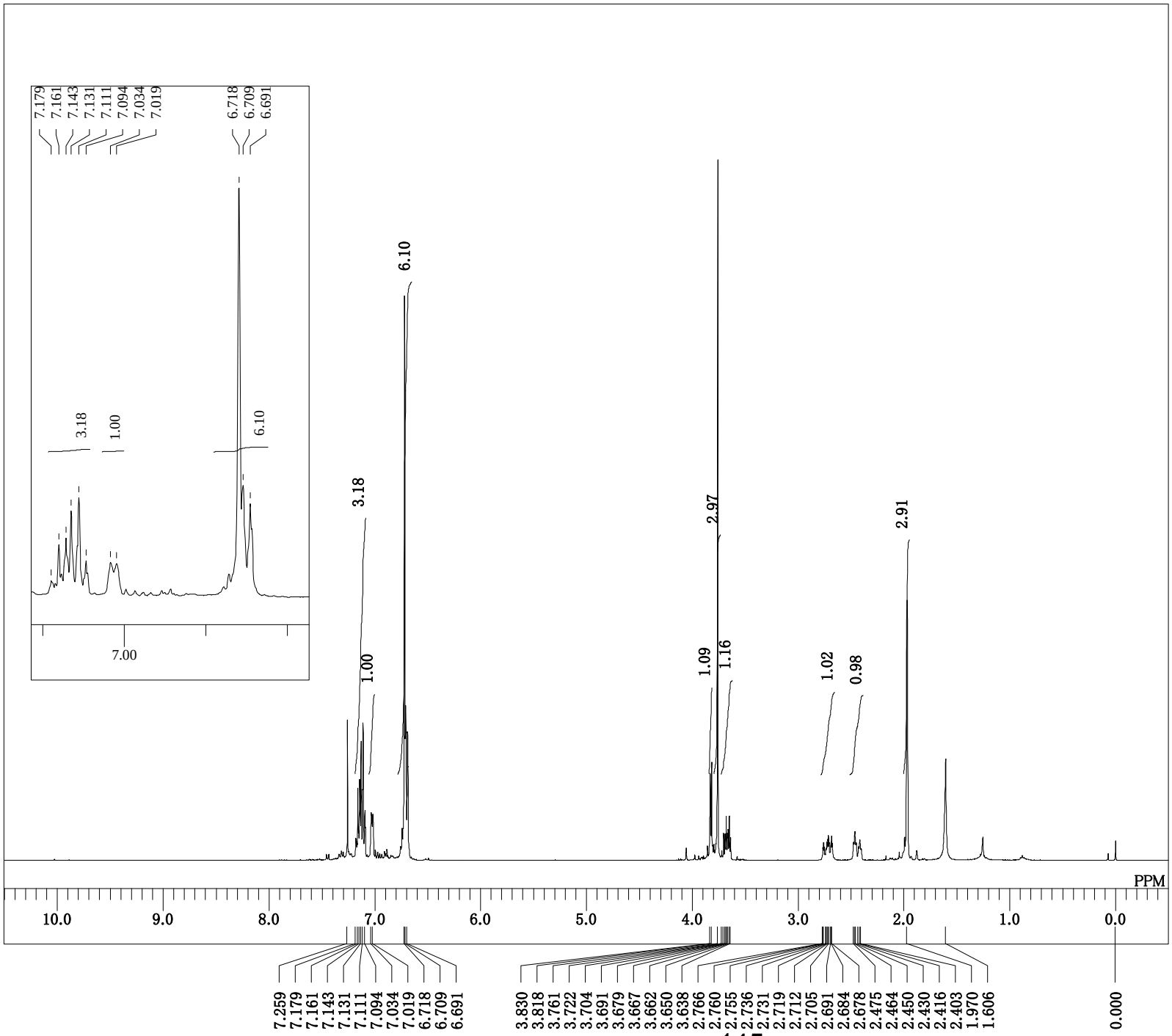
4j



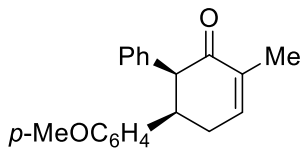
DFILE	4j_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-24 14:57:21
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	512
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



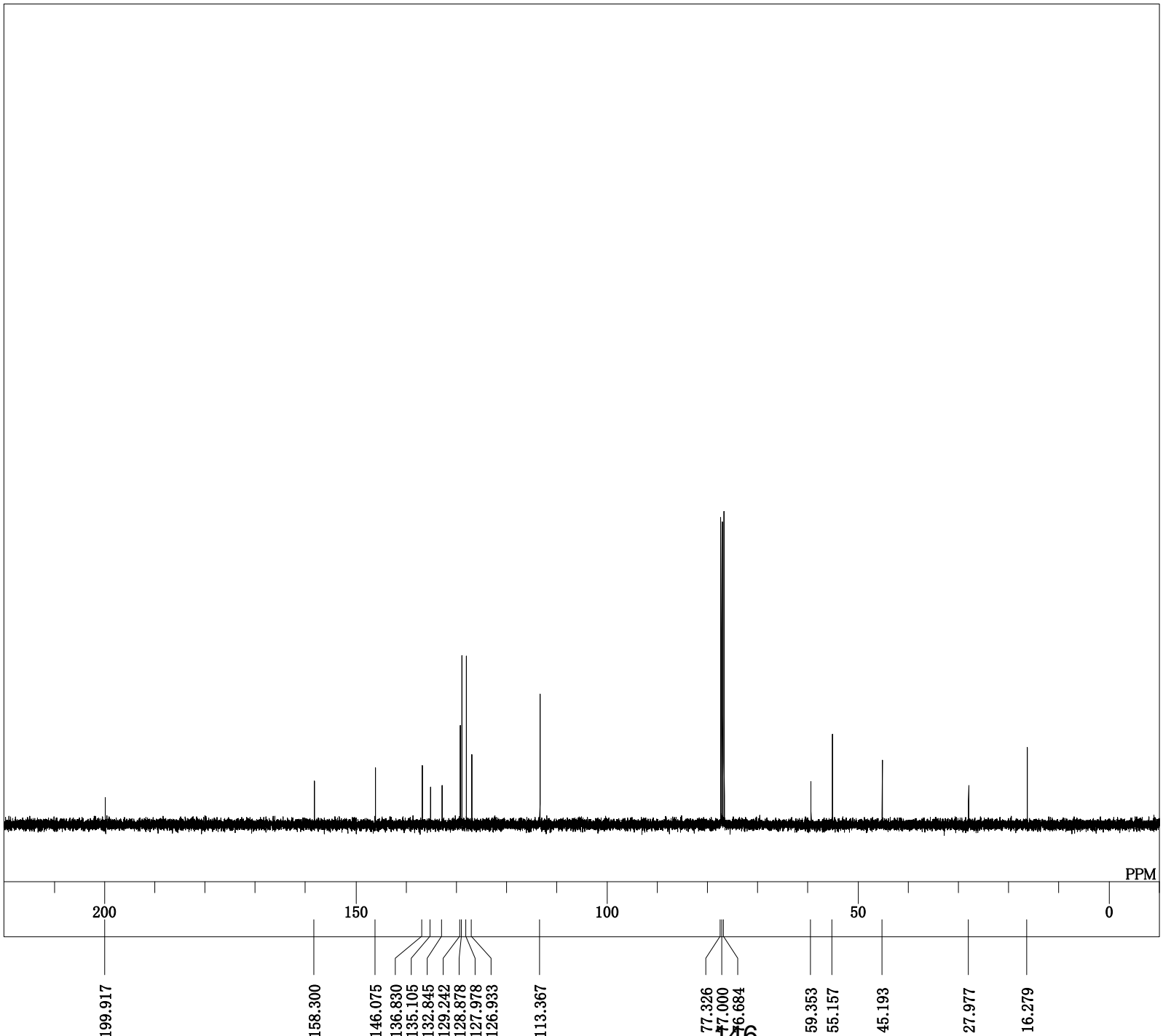
4j



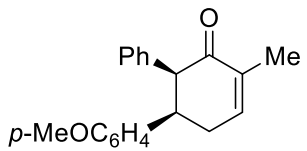
DFILE	cis_4k_1H.als
COMNT	single_pulse
DATIM	2020-10-11 16:37:20
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	34



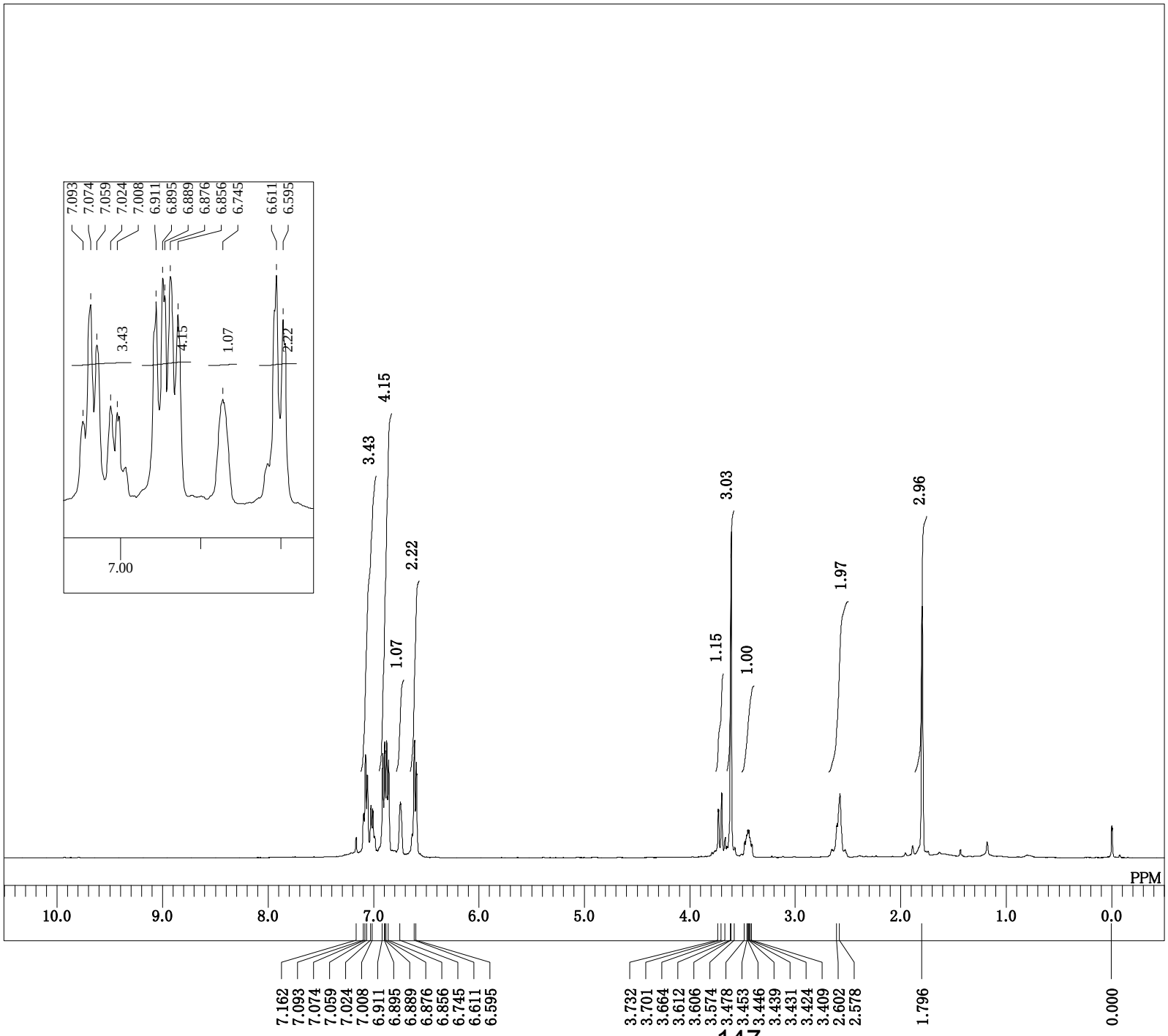
***cis*-4k**



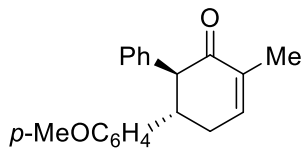
DFILE	cis_4k_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-10-11 16:38:32
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	193
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



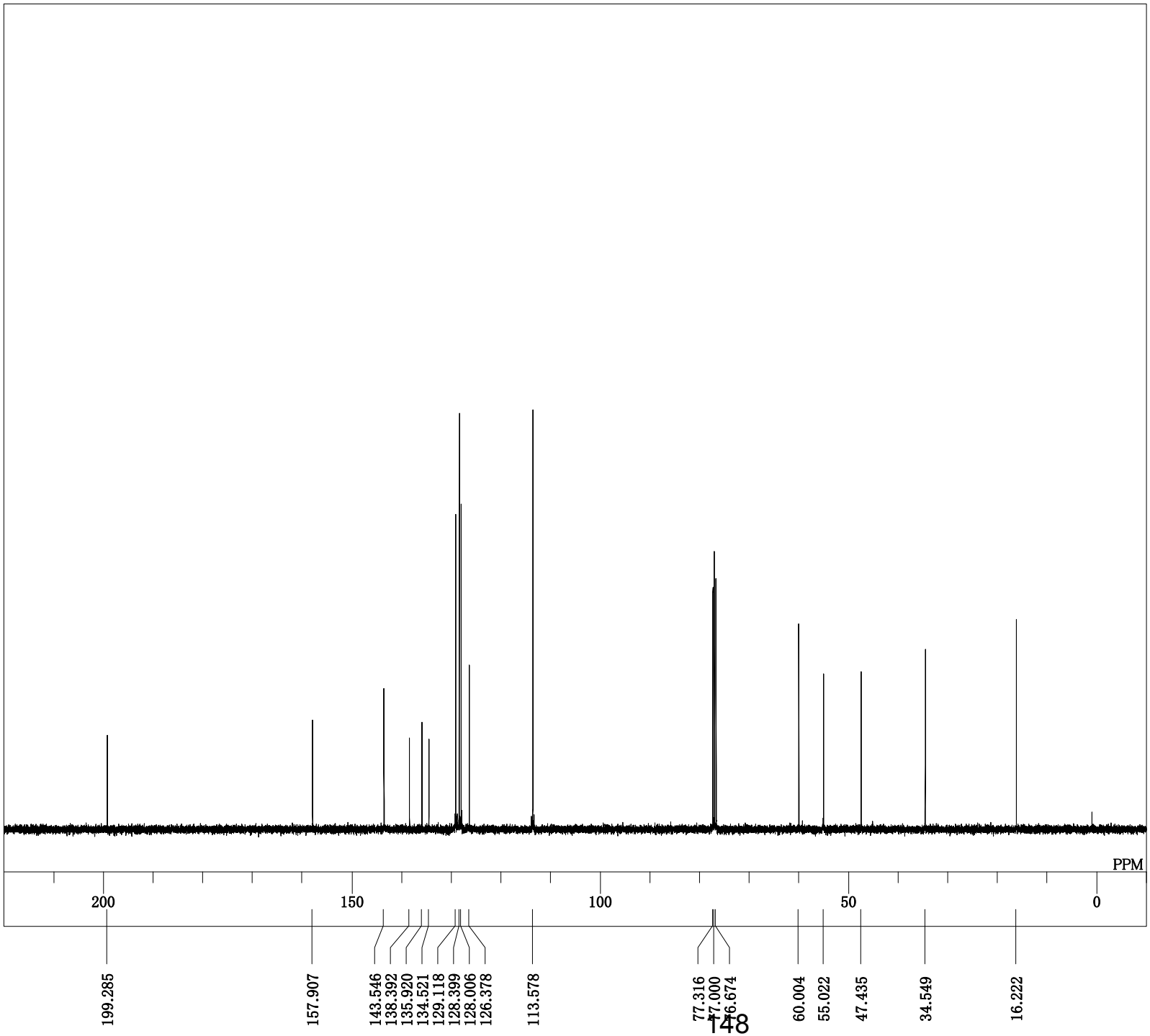
***cis*-4k**



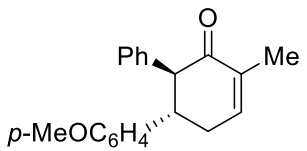
DFILE	trans_4k_1H.als
COMNT	single_pulse
DATIM	2020-10-11 16:54:19
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	24



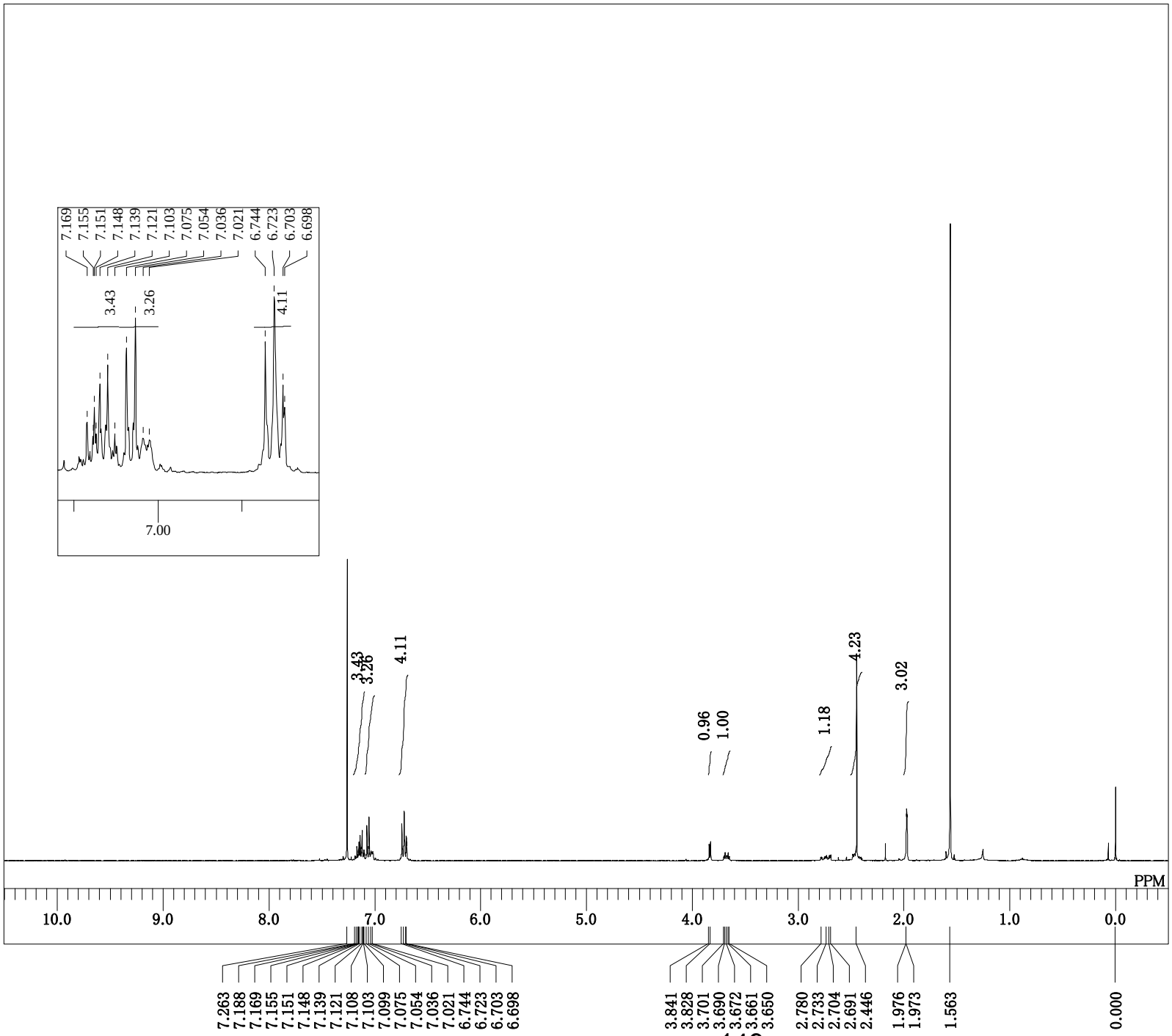
***trans*-4k**



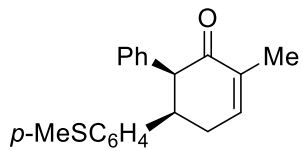
DFILE	trans_4k_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-10-11 16:55:32
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	508
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



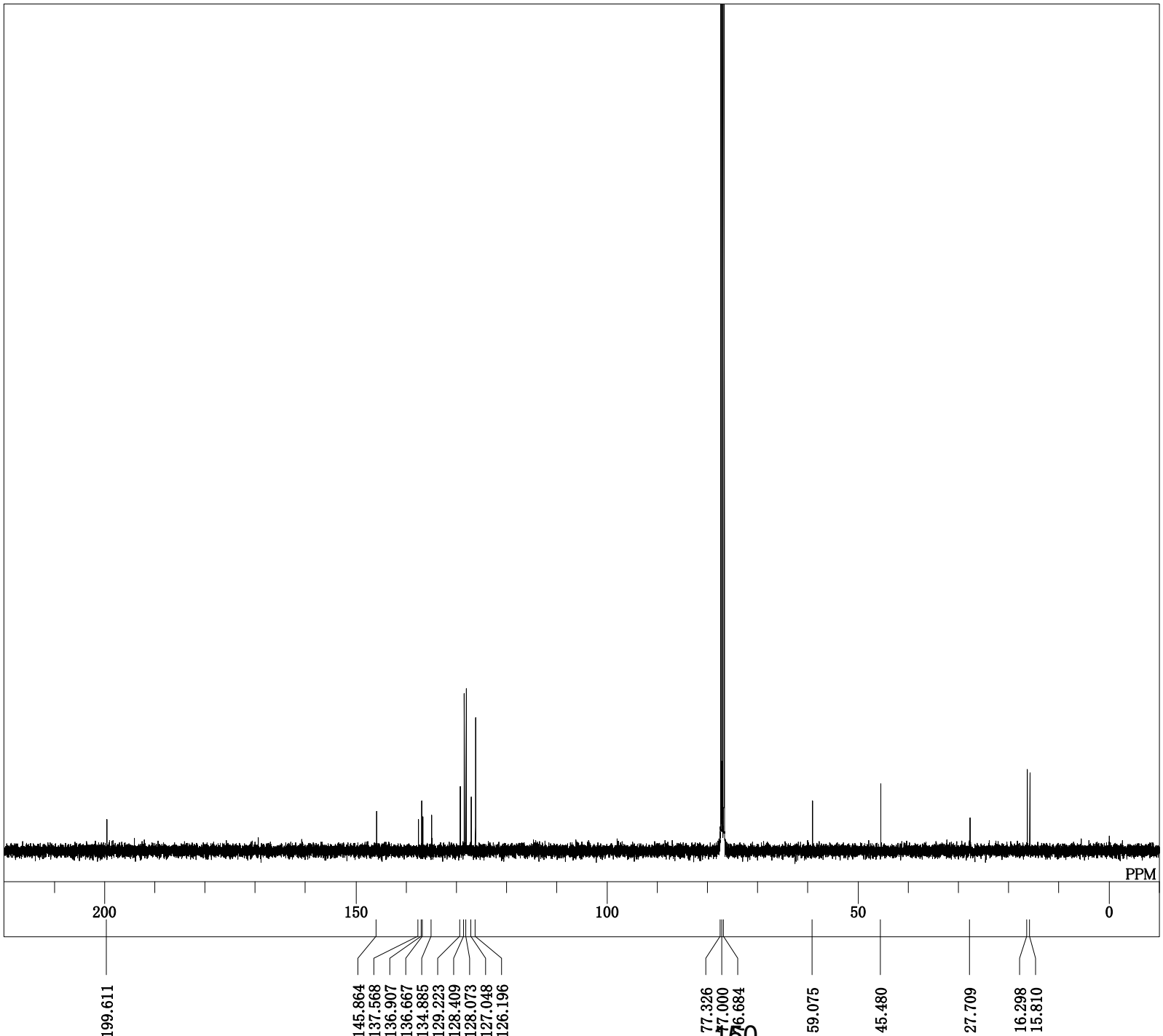
***trans*-4k**



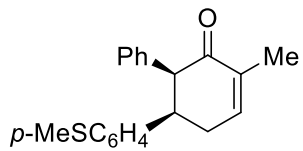
DFILE	cis_4l_1H.als
COMNT	single_pulse
DATIM	2020-11-01 21:10:12
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	32
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	19.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	48



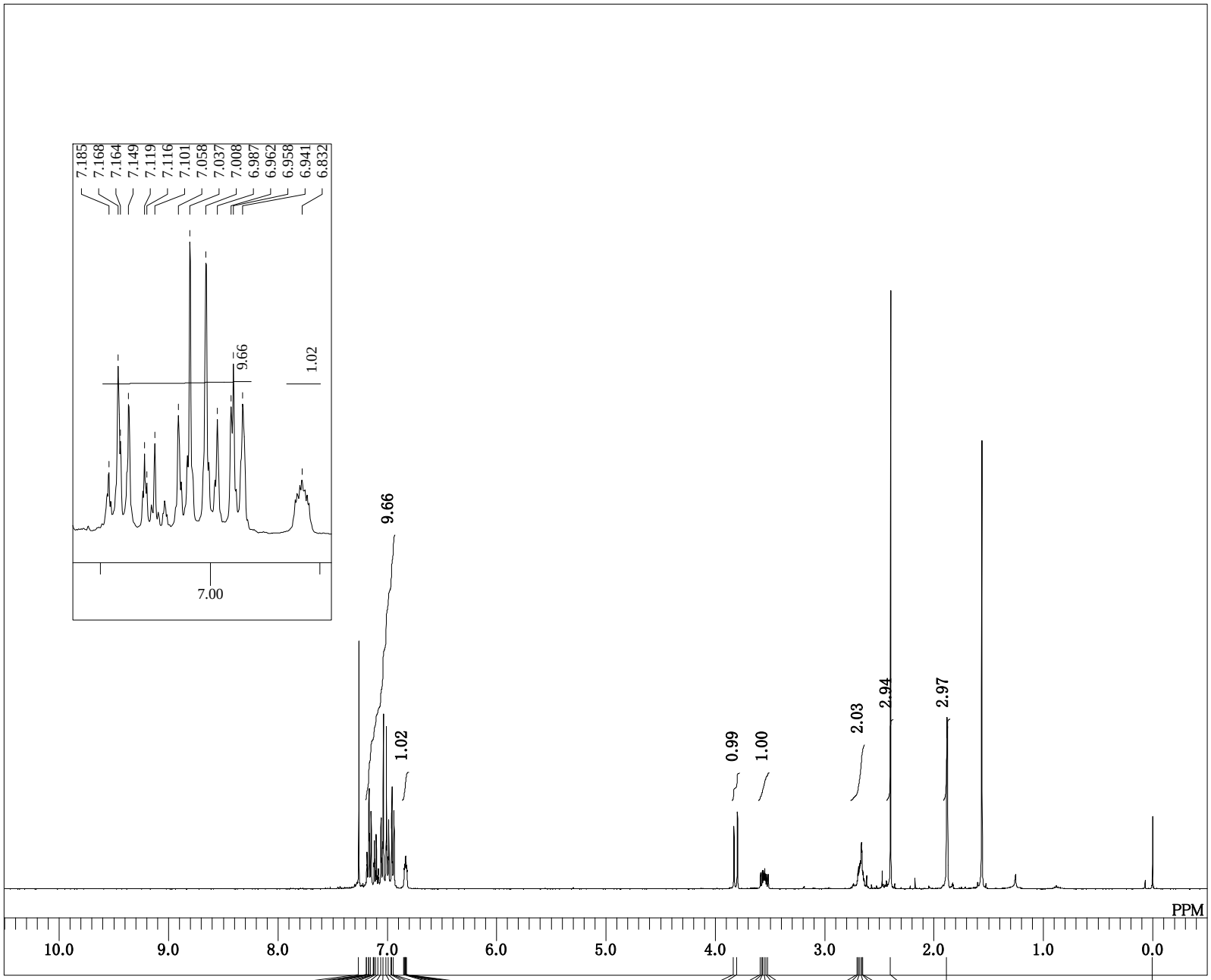
***cis*-4l**



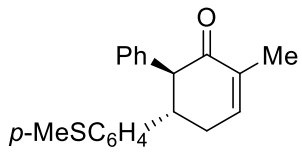
DFILE	cis_4l_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-01 21:14:18
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	14364
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



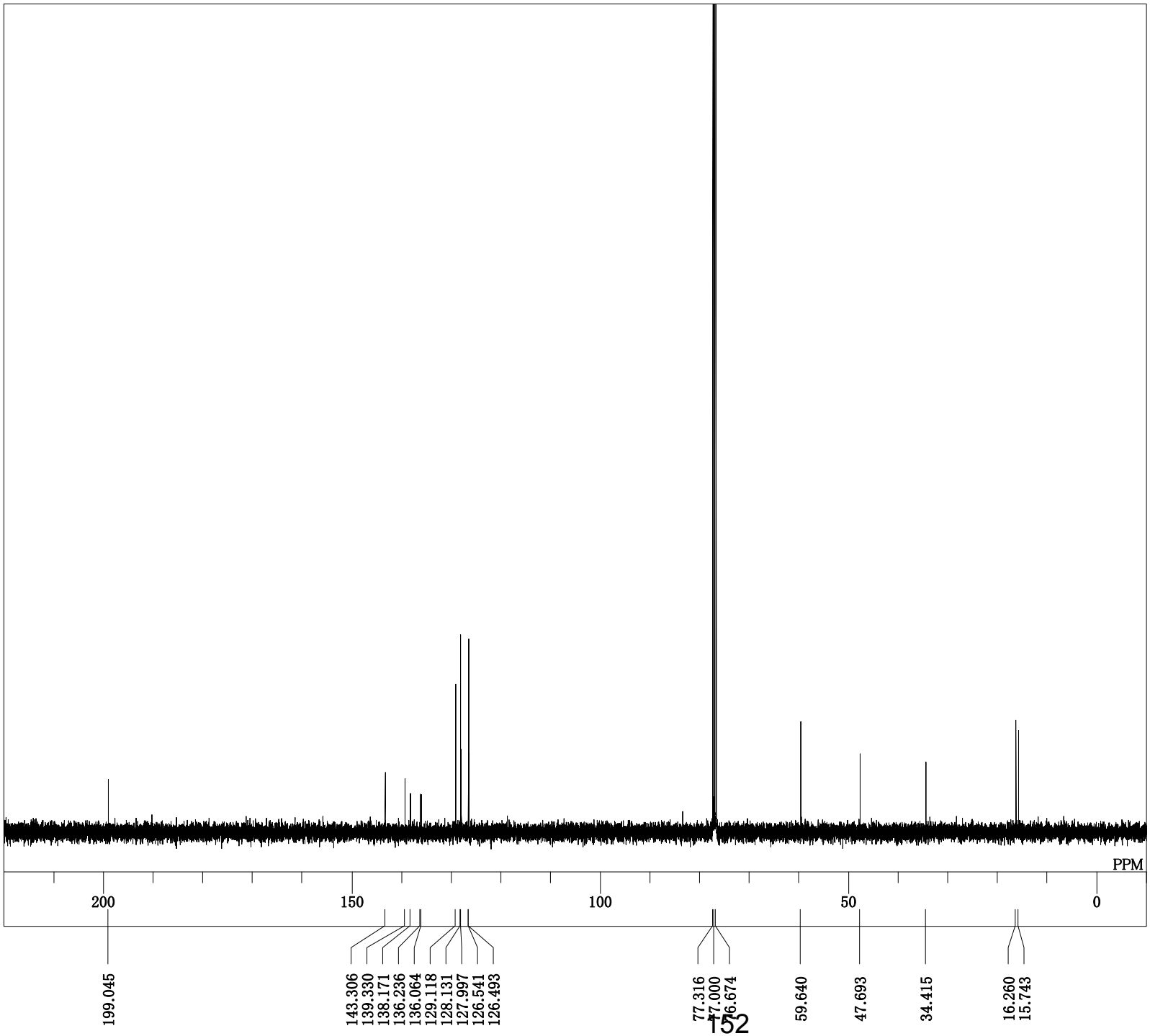
***cis*-4l**



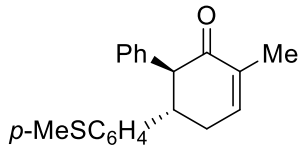
DFILE	trans_4l_1H.als
COMNT	single_pulse
DATIM	2020-11-01 18:18:47
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	19.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	42



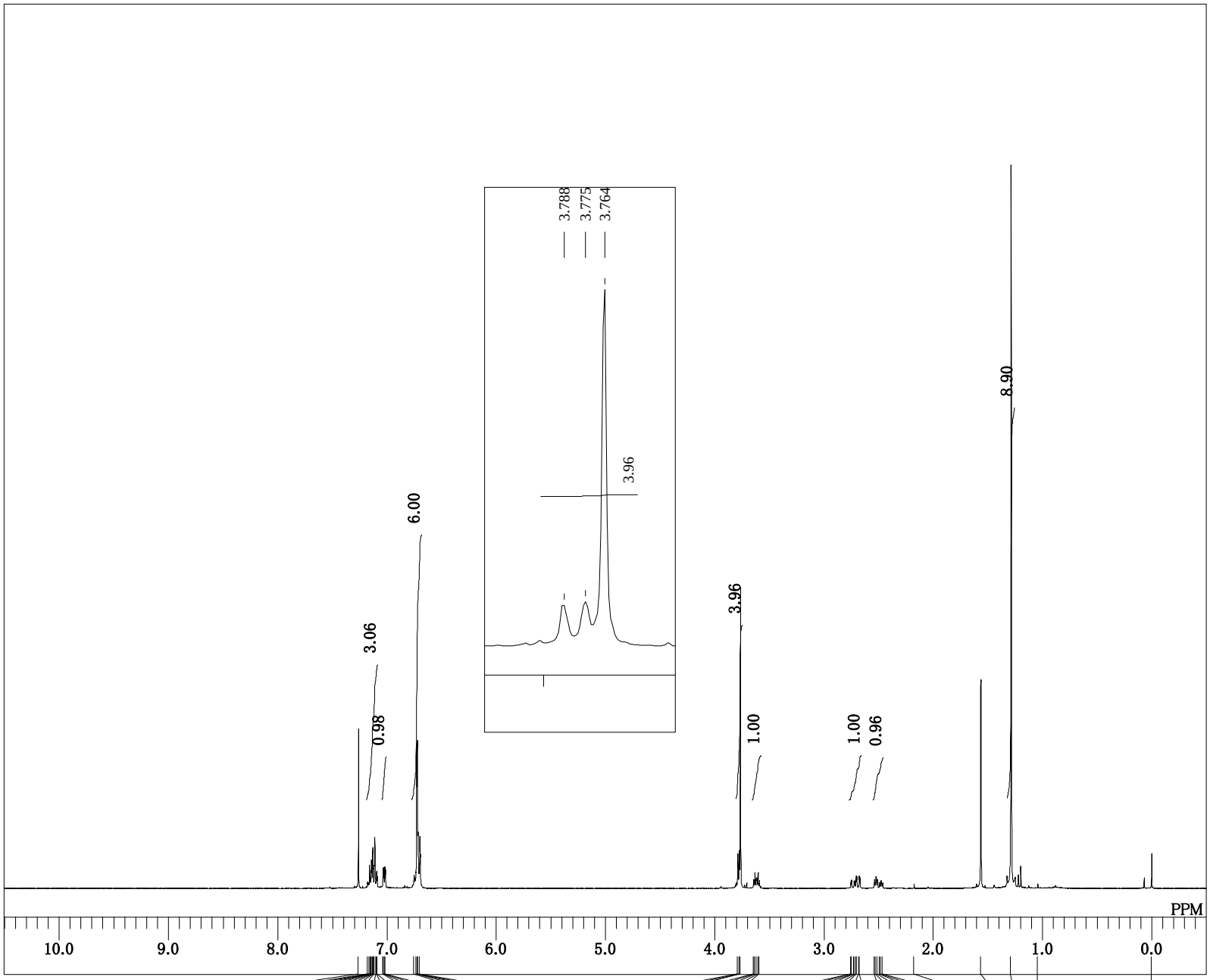
***trans*-4l**



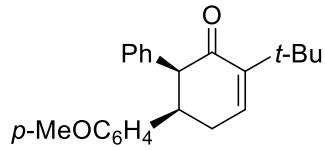
DFILE	trans_4l_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-01 18:19:59
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	916
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



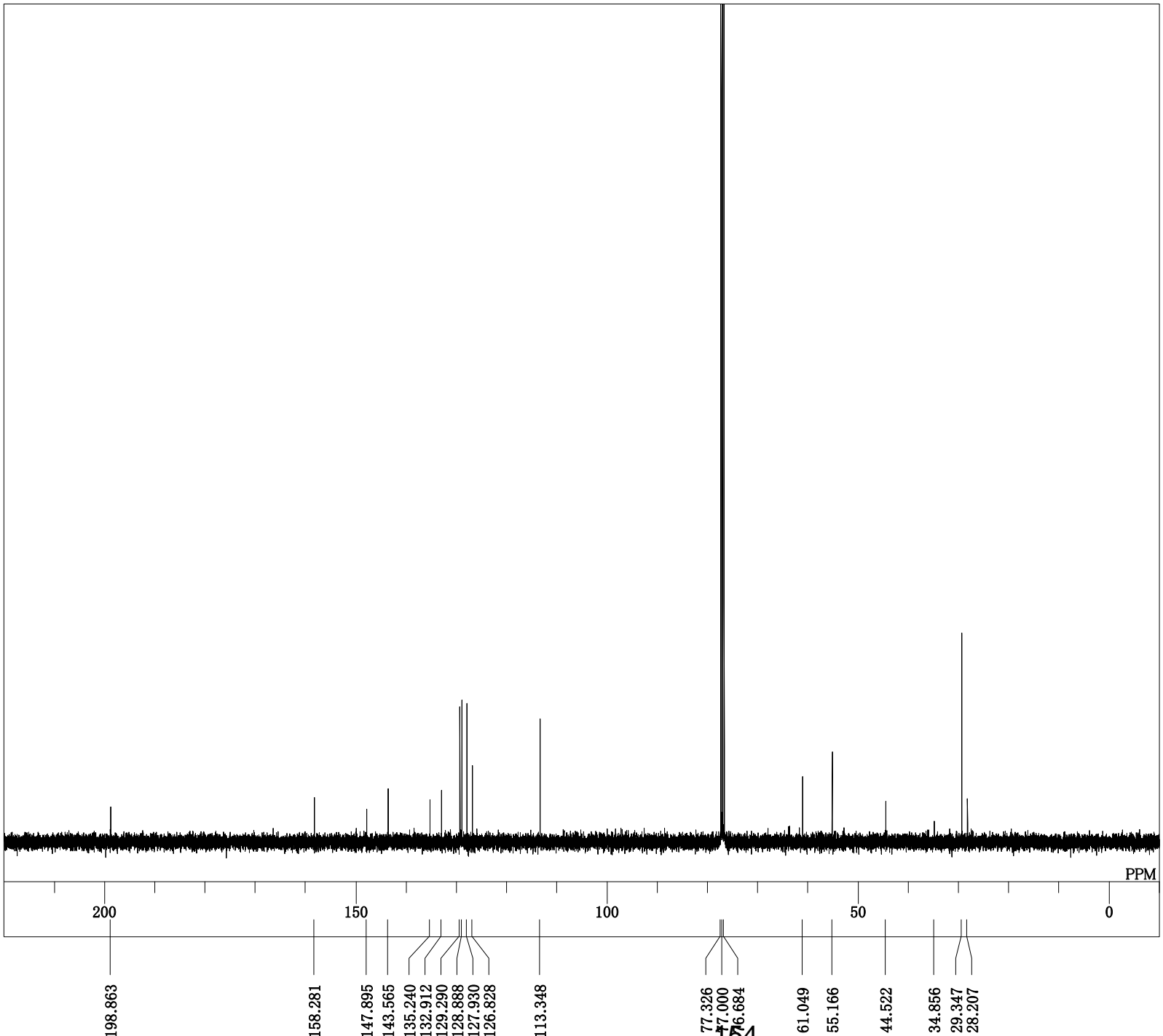
***trans*-4l**



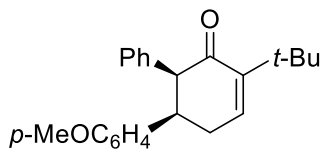
DFILE	cis_4m_1H.als
COMNT	single_pulse
DATIM	2020-11-01 16:40:26
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	19.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	42



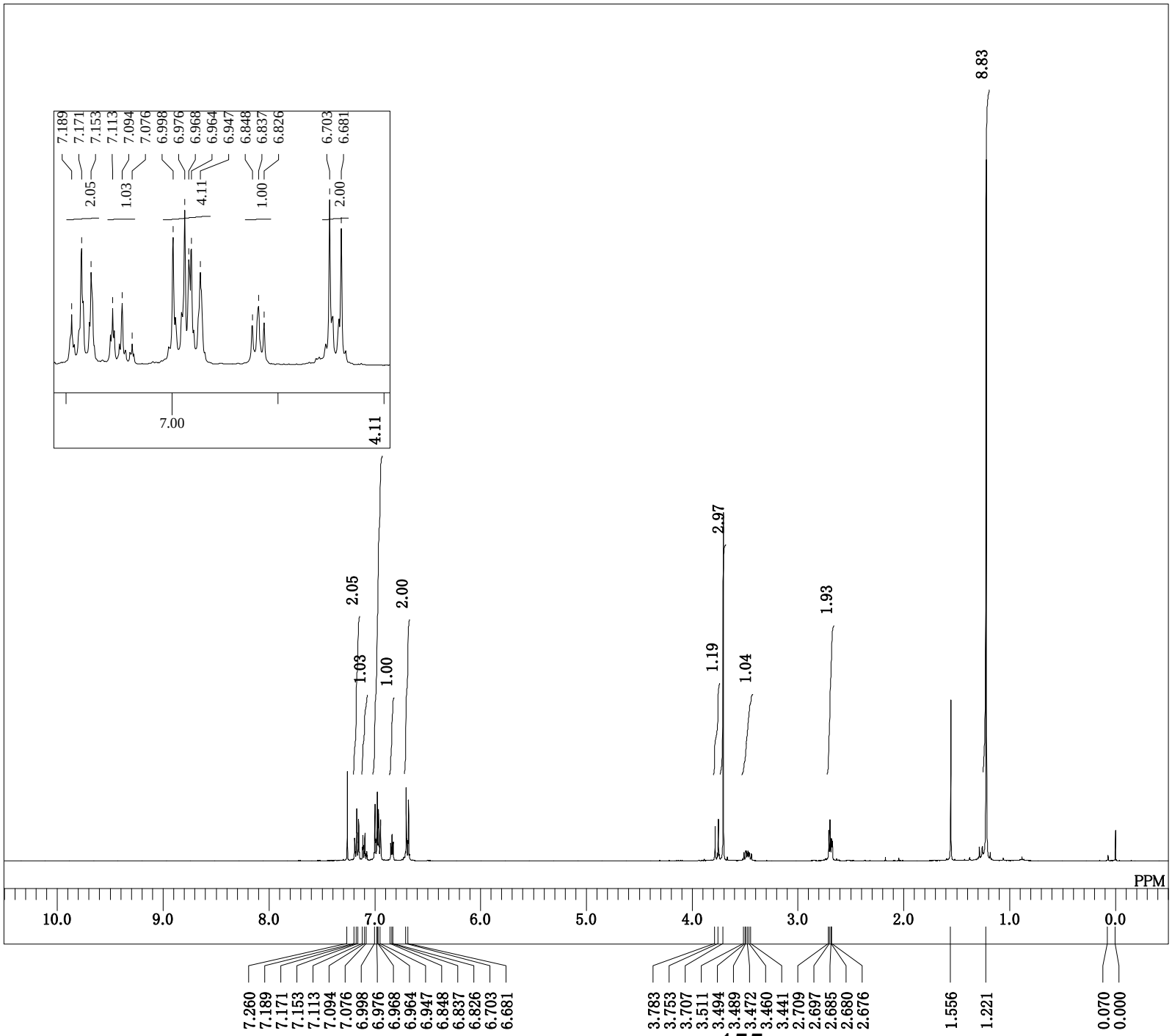
***cis*-4m**



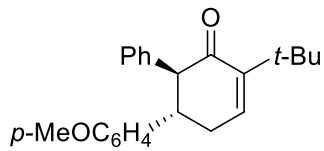
DFILE cis_4m_13C.als
COMNT single pulse decoupled gated NOE
DATIM 2020-11-01 16:41:38
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 25000.00 Hz
SCANS 1024
ACQTM 1.0486 sec
PD 2.0000 sec
PW1 3.59 usec
IRNUC 1H
CTEMP 19.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50



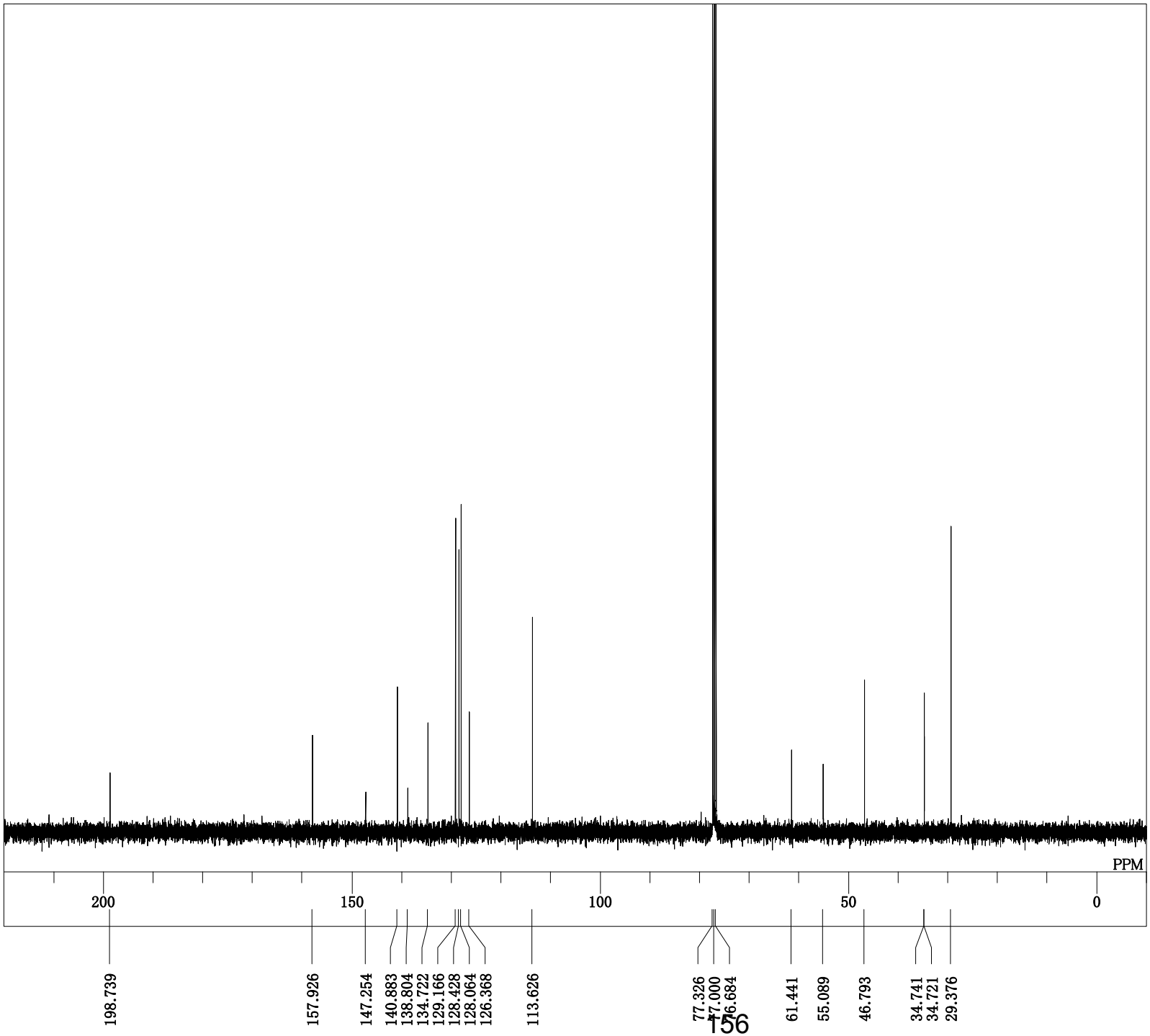
***cis*-4m**



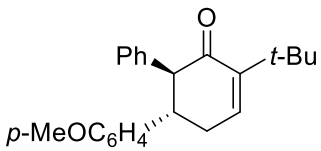
DFILE	trans_4m_1H.als
COMNT	single_pulse
DATIM	2020-11-01 15:12:09
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	19.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	38



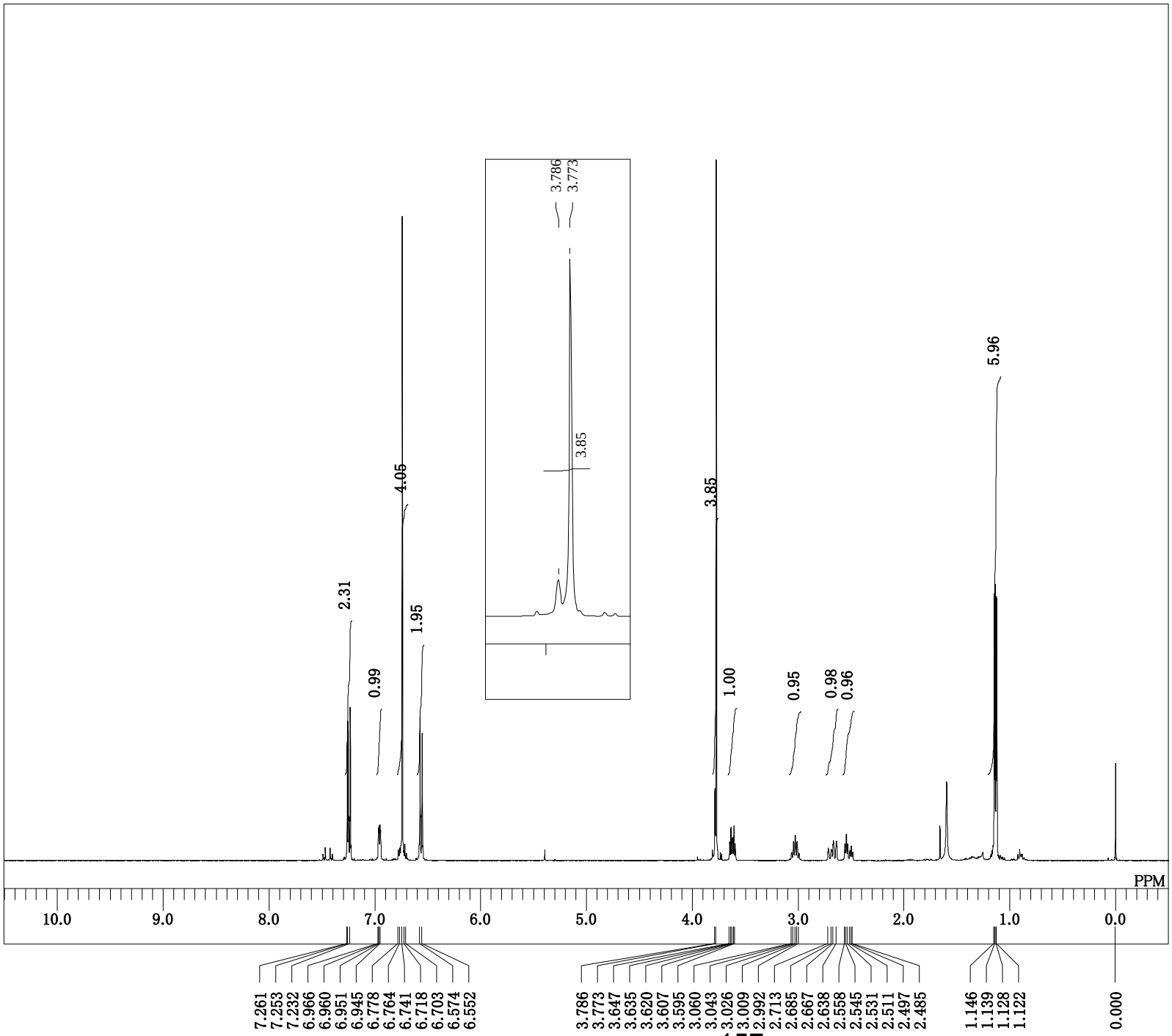
***trans*-4m**



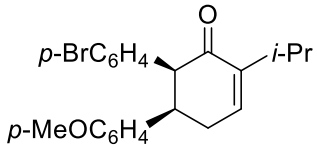
DFILE	trans_4m_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-01 15:13:21
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	1024
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



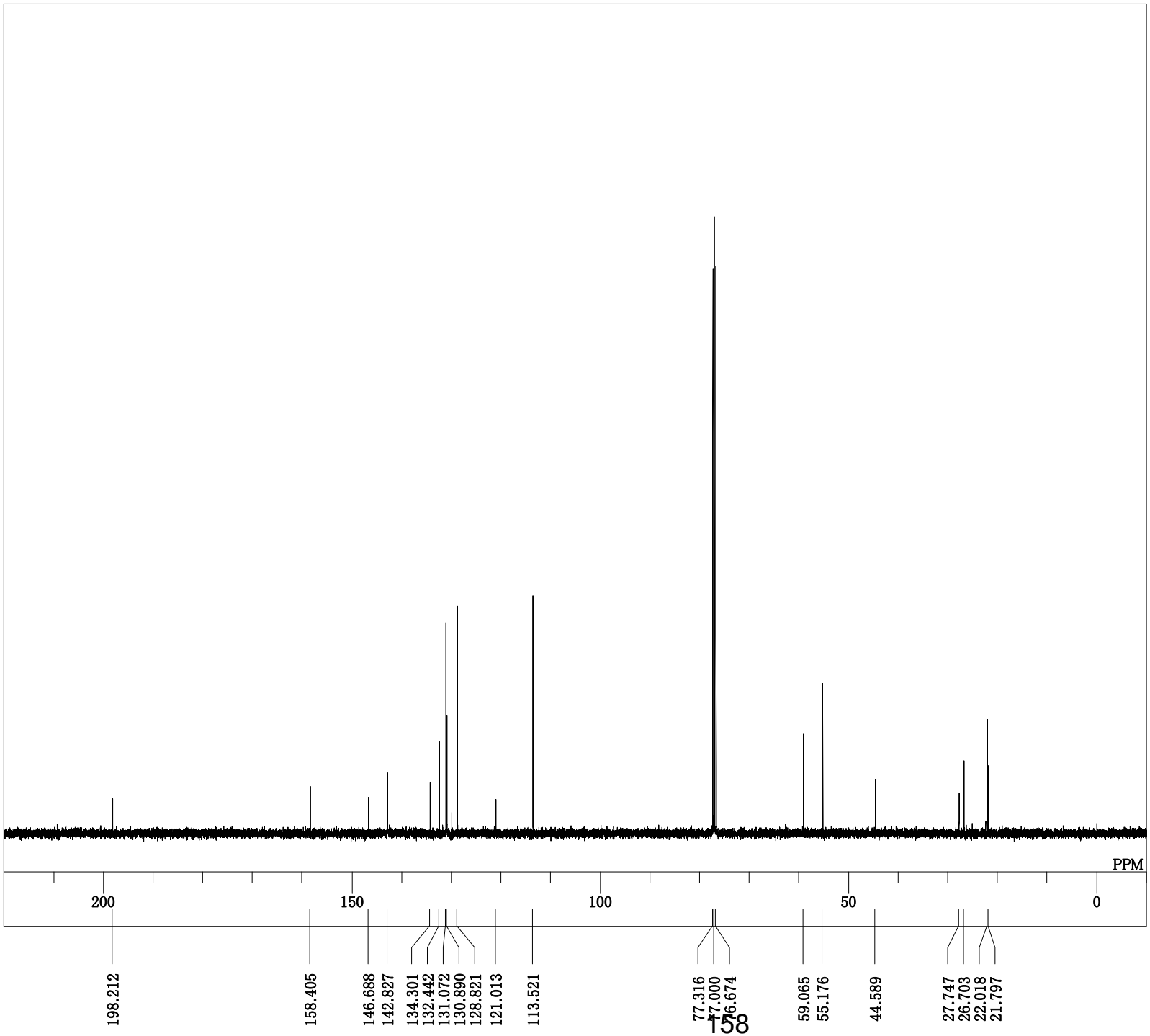
***trans*-4m**



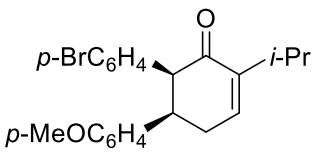
DFILE	cis_4n_1H.als
COMNT	single_pulse
DATIM	2020-10-24 10:24:35
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	36



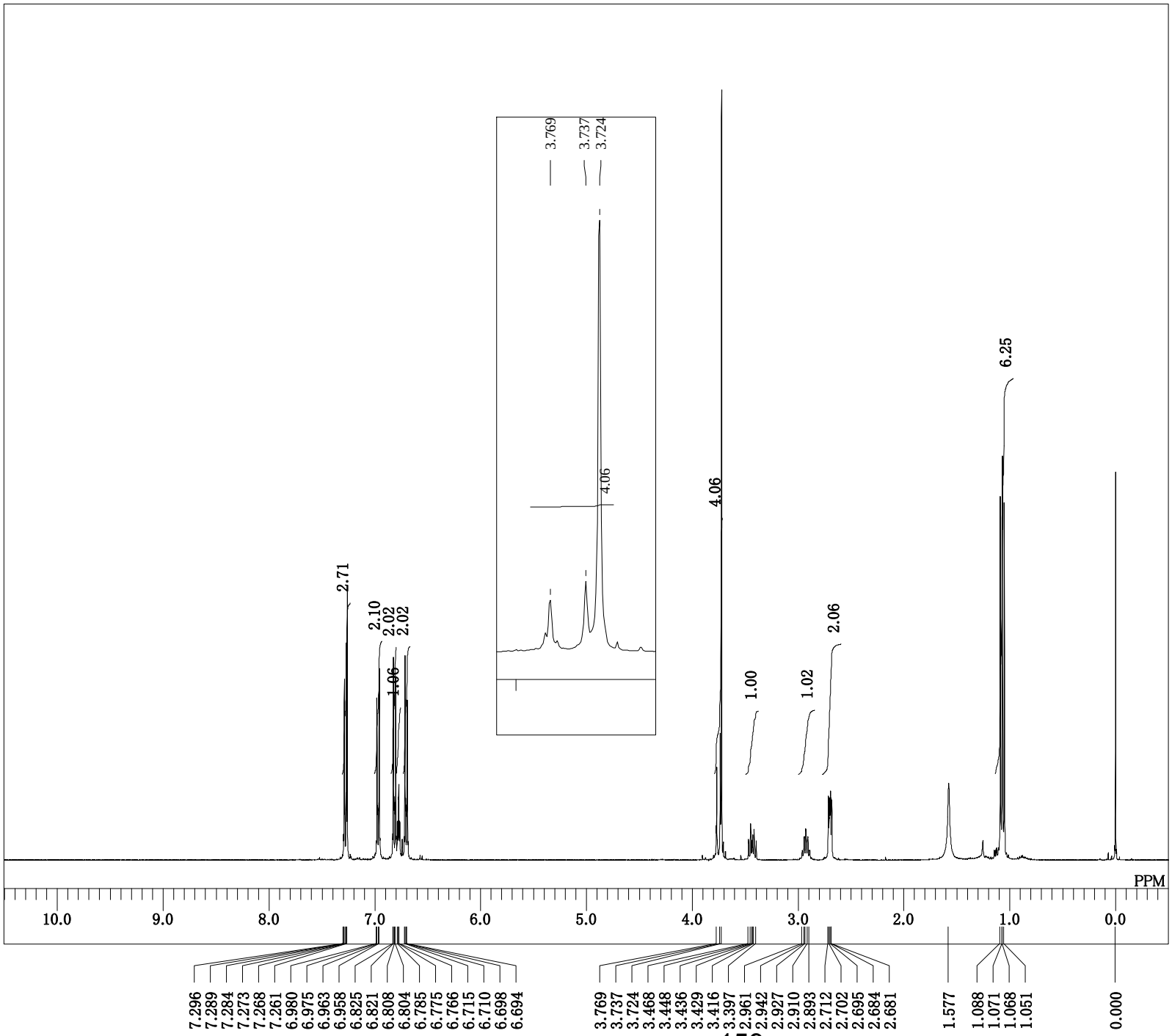
***cis*-4n**



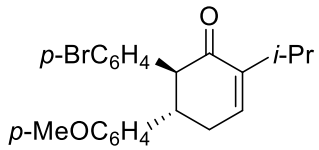
DFILE	cis_4n_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-10-24 10:25:46
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	930
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



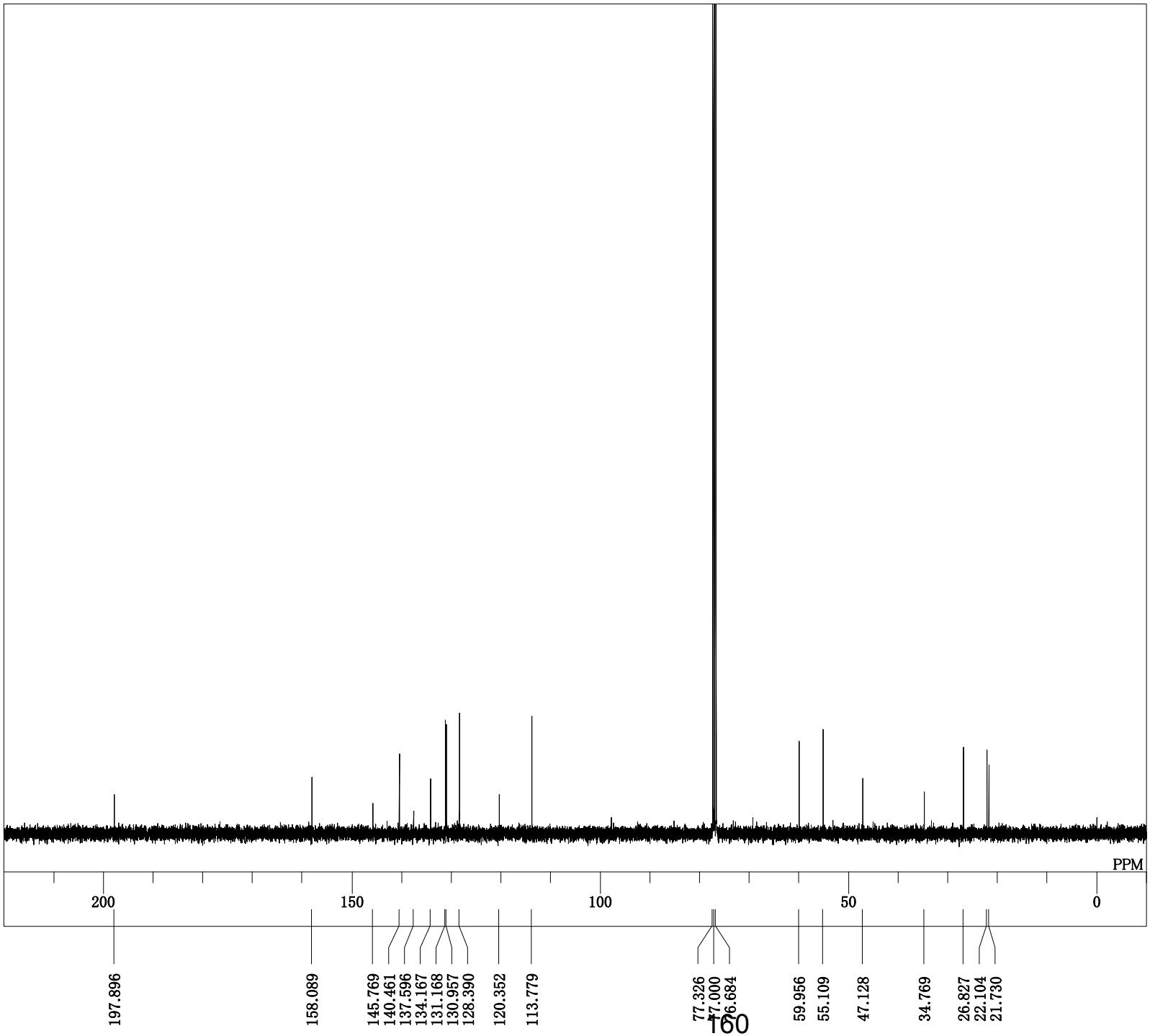
***cis*-4n**



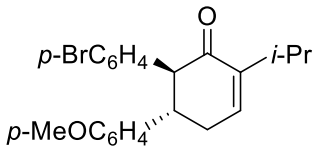
DFILE	trans_4n_1H.als
COMNT	single_pulse
DATIM	2020-10-14 20:00:10
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	38



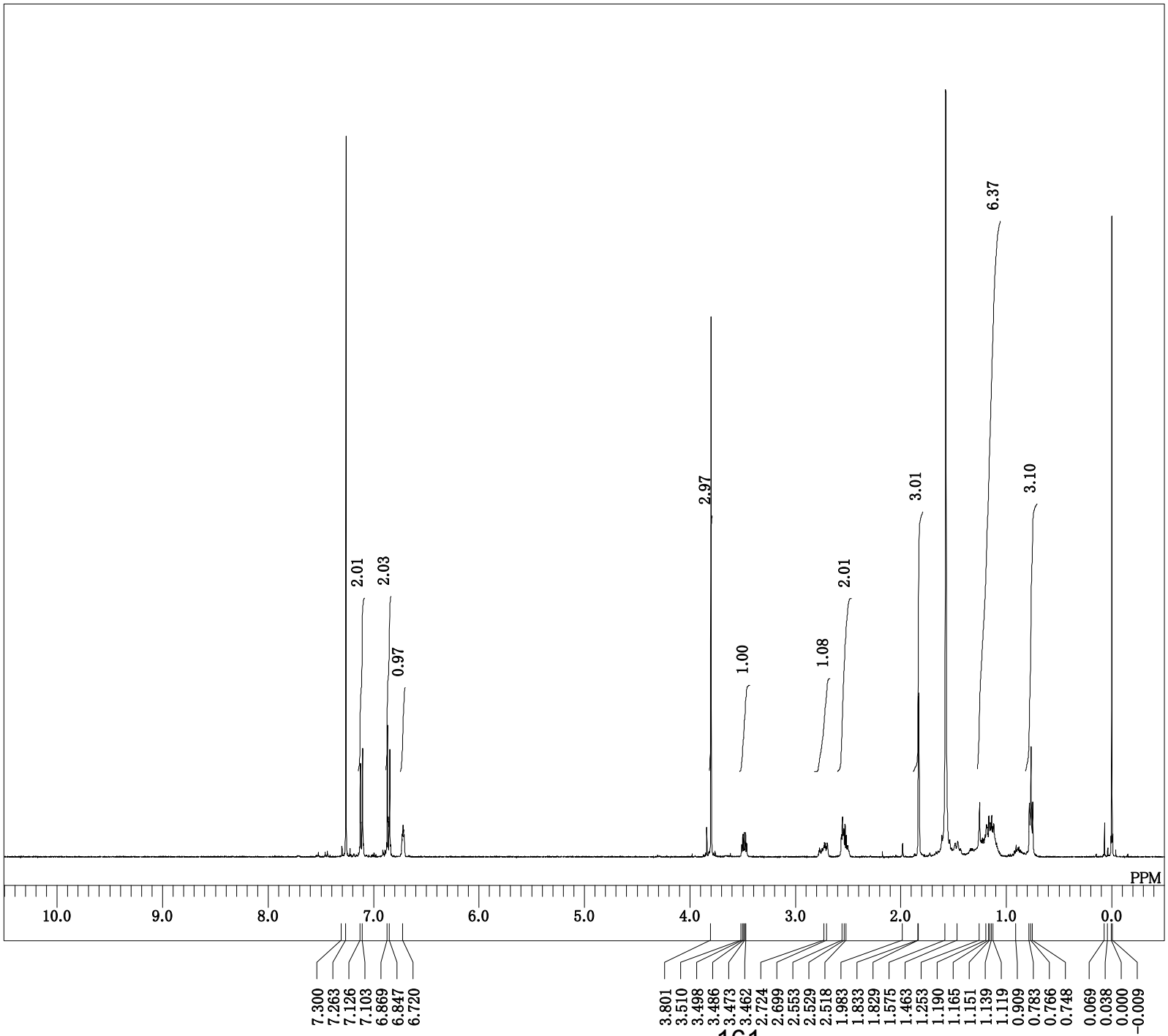
***trans*-4n**



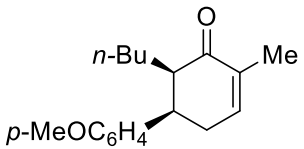
DFILE	trans_4n_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-10-29 20:13:26
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	999
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



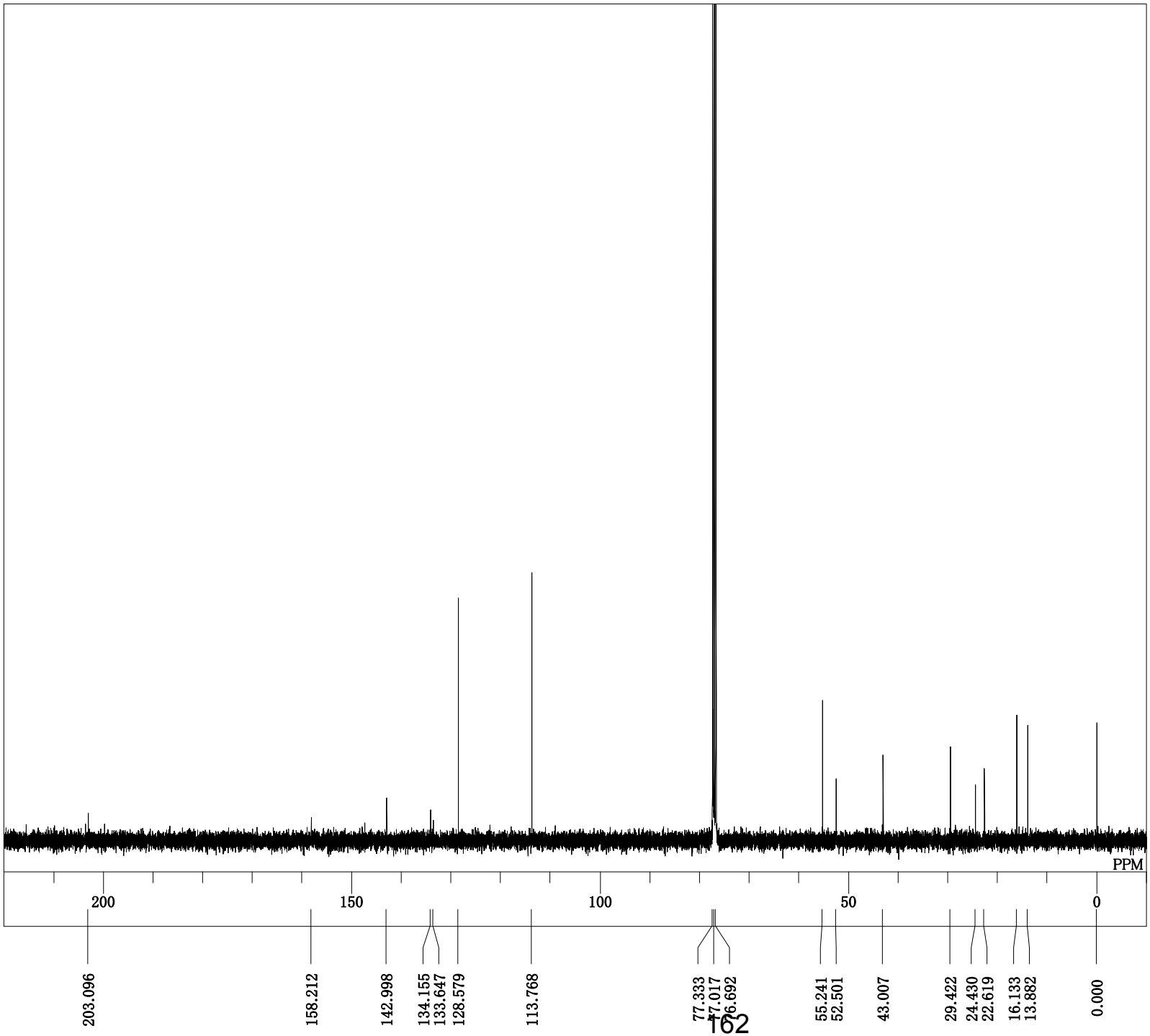
***trans*-4n**



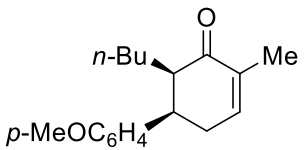
DFILE	cis_4o_1H.als
COMNT	single_pulse
DATIM	2020-10-23 20:30:54
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	48



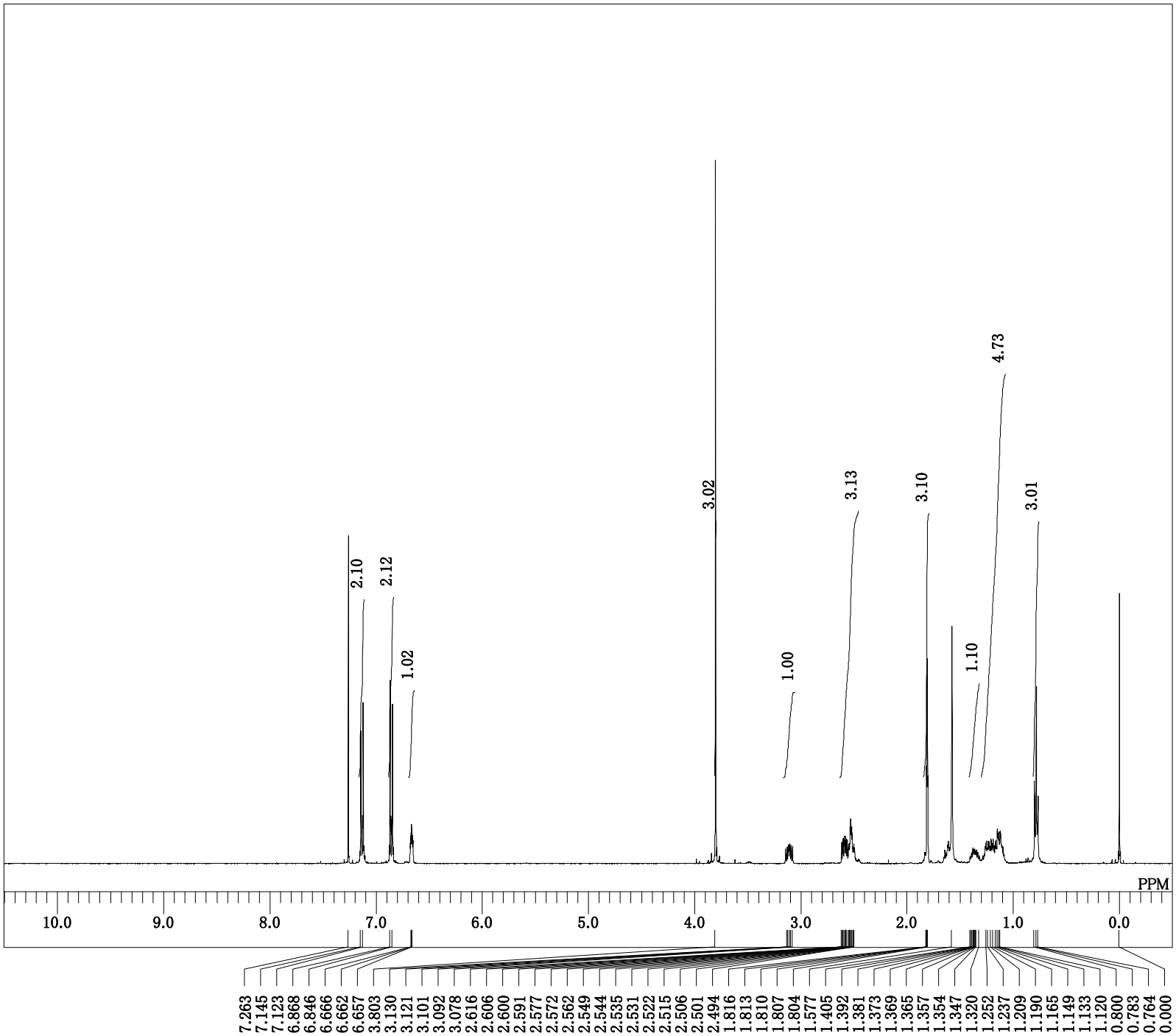
***cis*-4o**



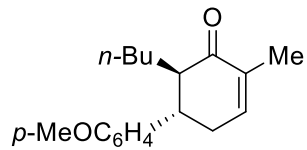
DFILE	cis_4o_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-10-25 22:07:21
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	13553
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	50



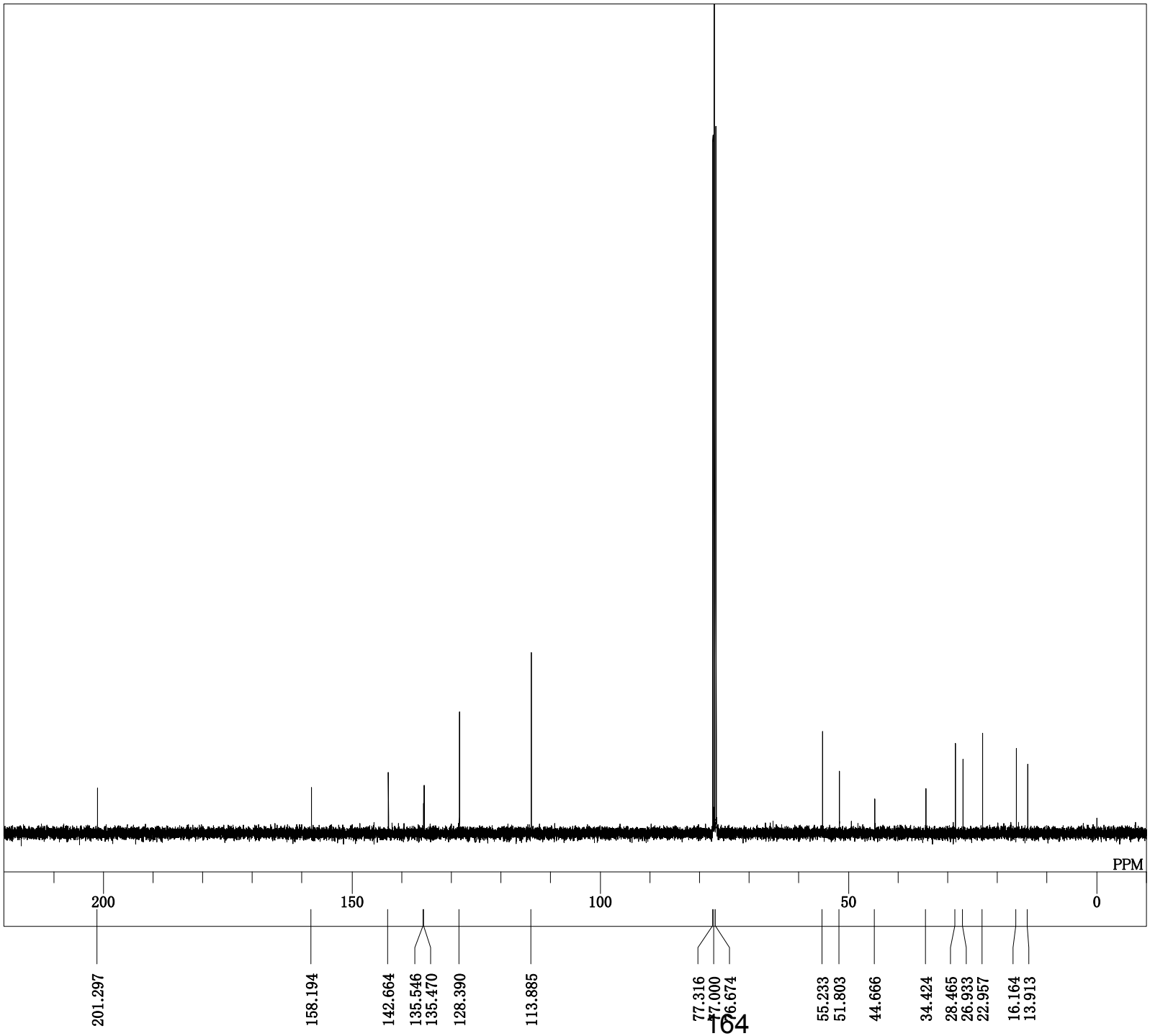
***cis*-4o**



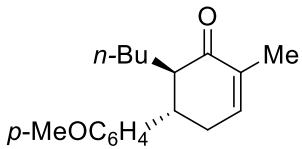
DFILE	trans_4o_1H.als
COMNT	single_pulse
DATIM	2020-10-22 20:06:03
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40



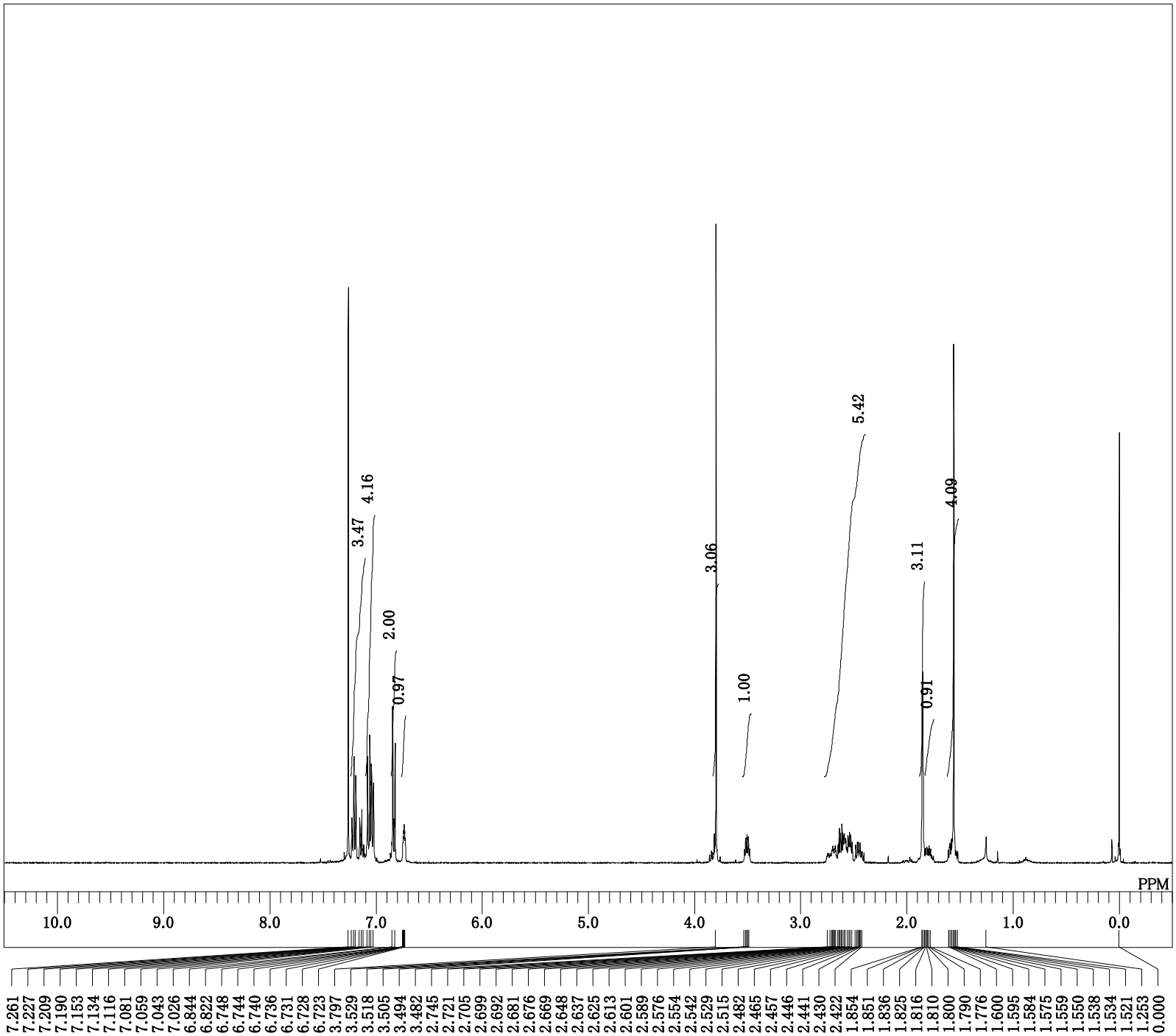
***trans*-4o**



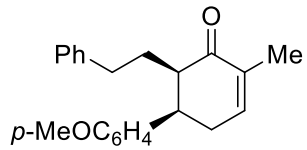
DFILE	trans_4o_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-18 22:19:31
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	1024
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



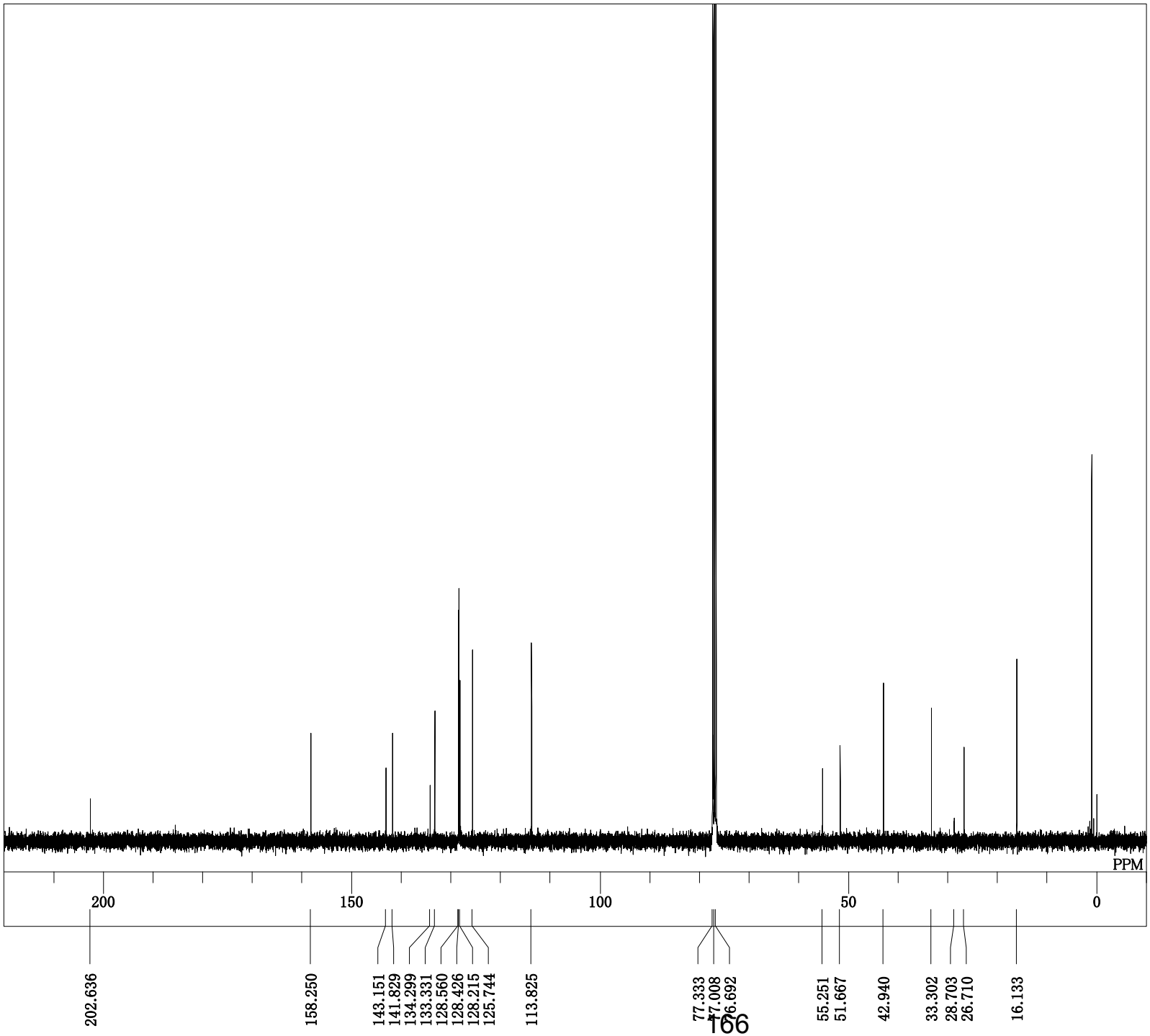
***trans*-4o**



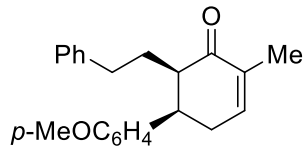
DFILE	cis_4p_1H.als
COMNT	single_pulse
DATIM	2020-12-22 21:14:11
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	18.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	44



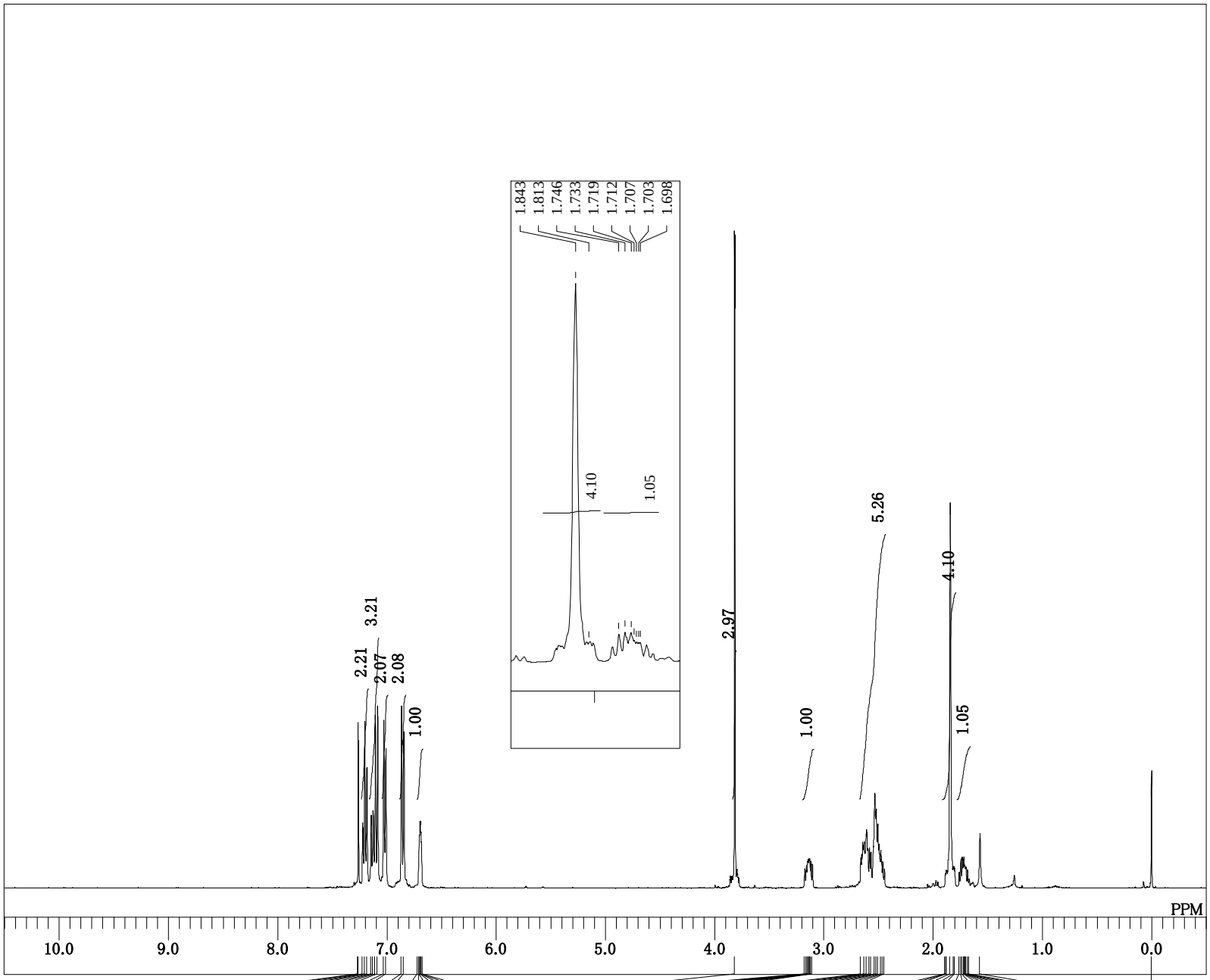
***cis*-4p**



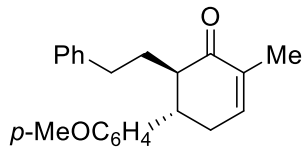
DFILE	cis_4p_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-12-23 22:23:56
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	13227
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	50



***cis*-4p**

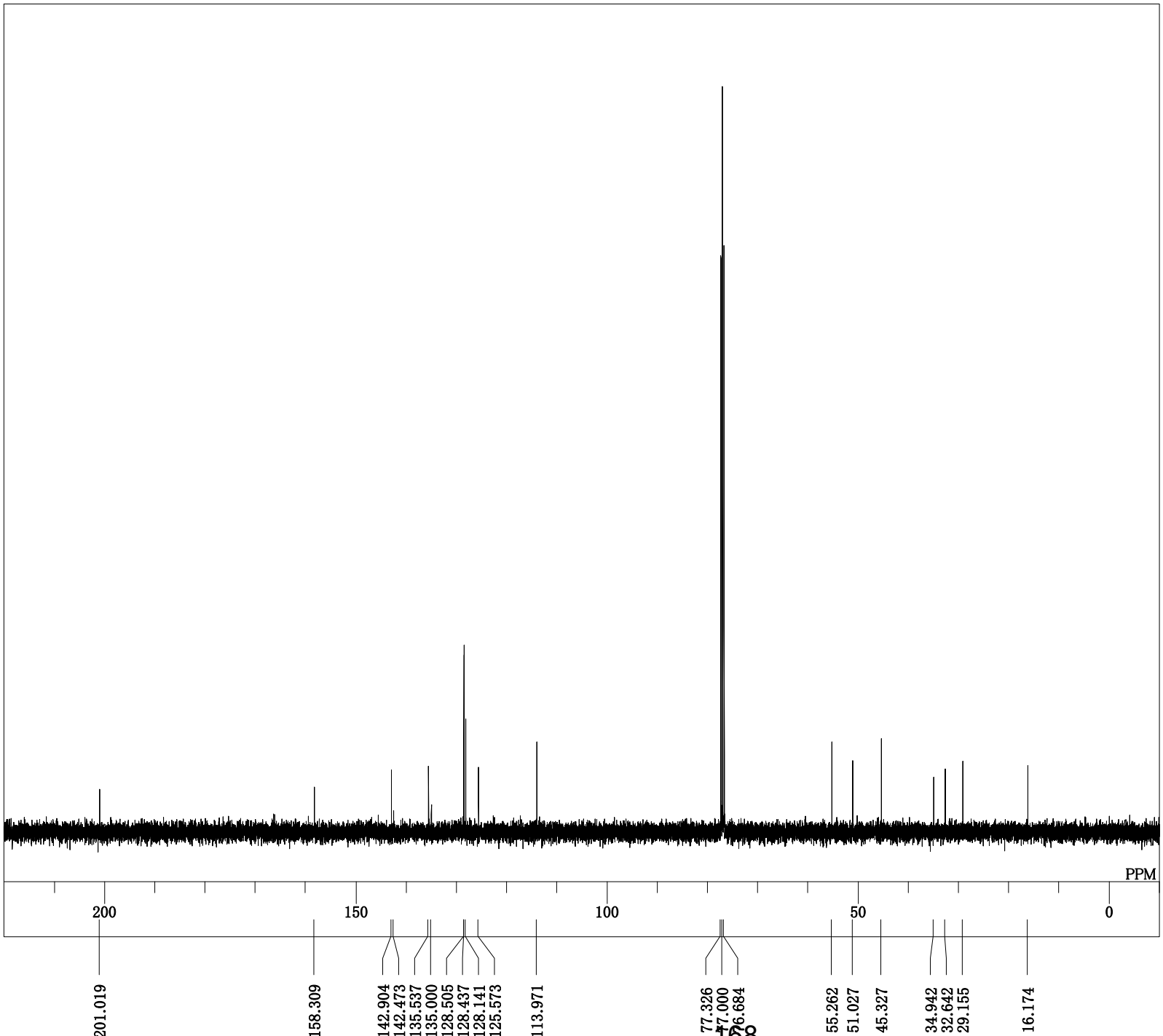


DFILE	trans_4p_1H.als
COMNT	single_pulse
DATIM	2020-12-21 16:12:37
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	18.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	38

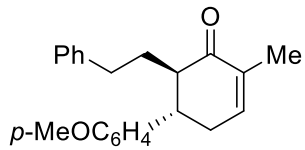


trans-4p

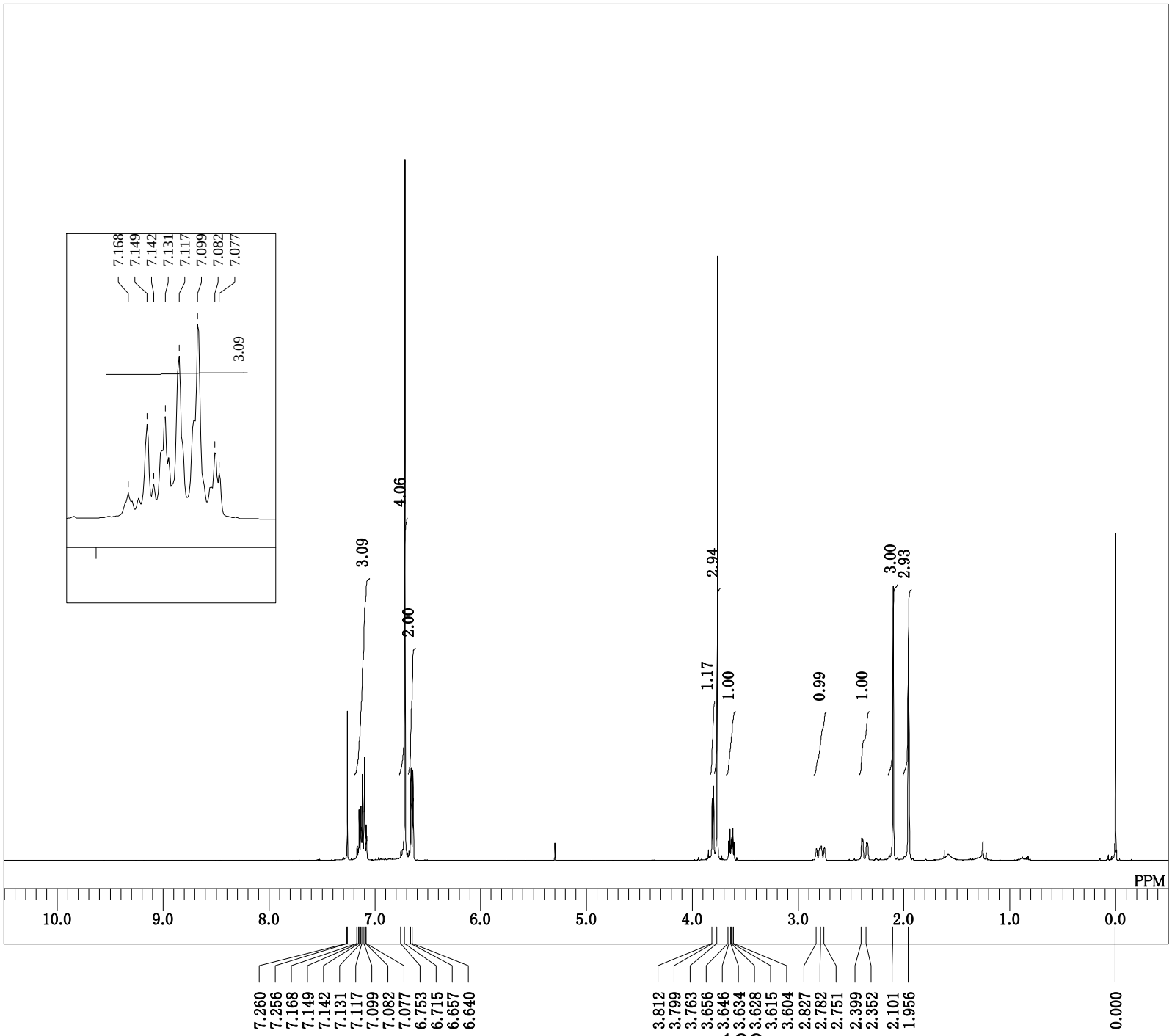
7.264 7.260 7.222 7.202 7.184 7.143 7.127 7.106 7.086 7.029 7.010 6.865 6.846 6.719 6.706 6.702 6.694 6.689 6.685 6.674 3.816 3.175 3.159 3.148 3.143 3.136 3.130 3.120 3.106 2.662 2.630 2.608 2.582 2.569 2.534 2.521 2.506 2.479 2.462 2.446 1.888 1.880 1.875 1.843 1.813 1.802 1.760 1.746 1.733 1.719 1.712 1.707 1.703 1.698 1.683 1.669 1.571 0.000



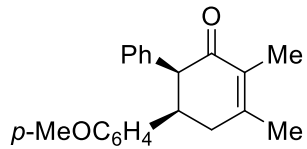
DFILE	trans_4p_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-12-21 16:24:13
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	256
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	18.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



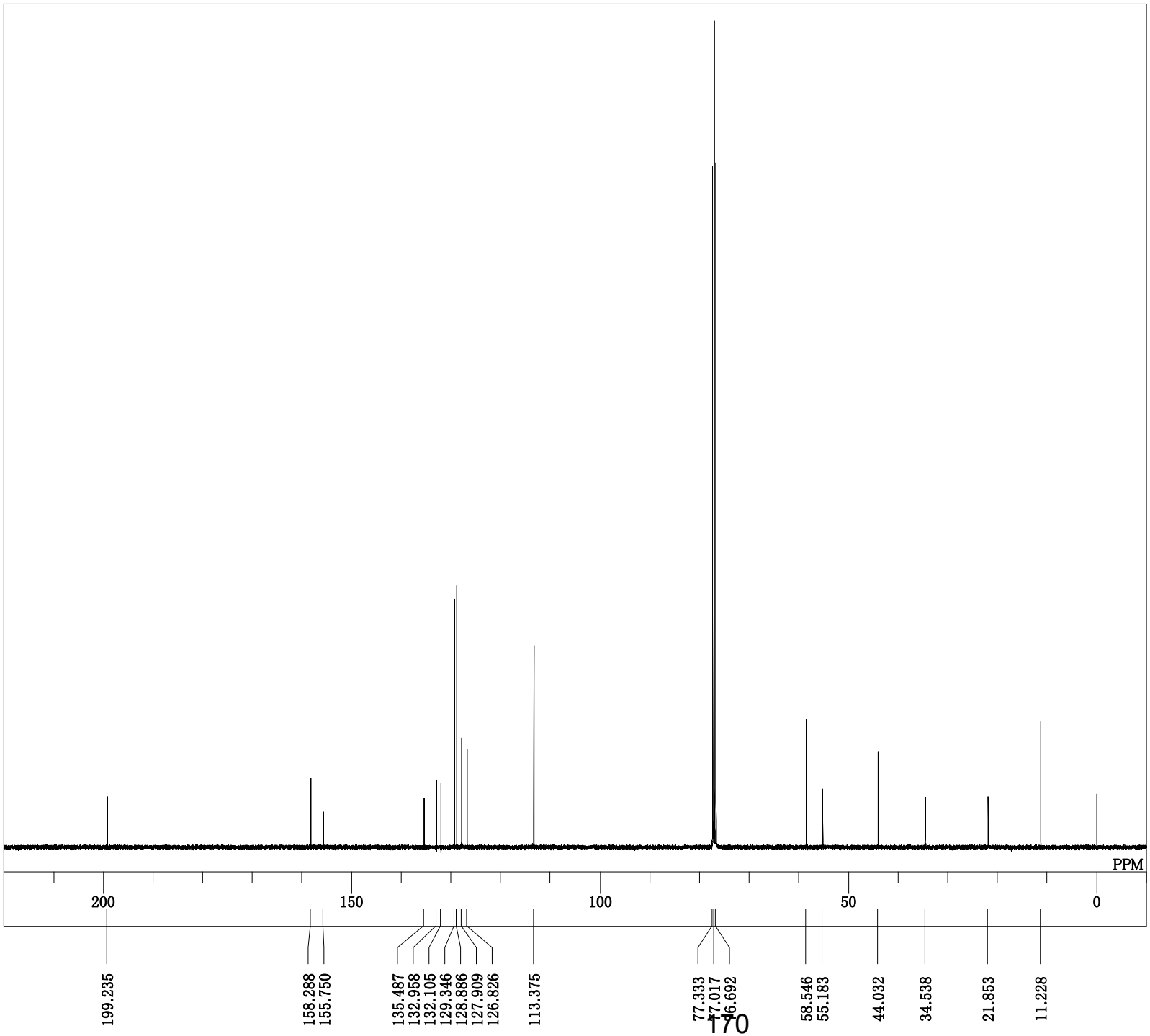
trans-4p



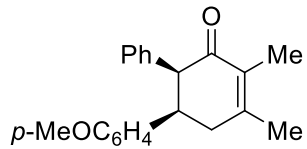
DFILE	cis_4q_1H.als
COMNT	single_pulse
DATIM	2020-10-13 21:14:06
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	32
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40



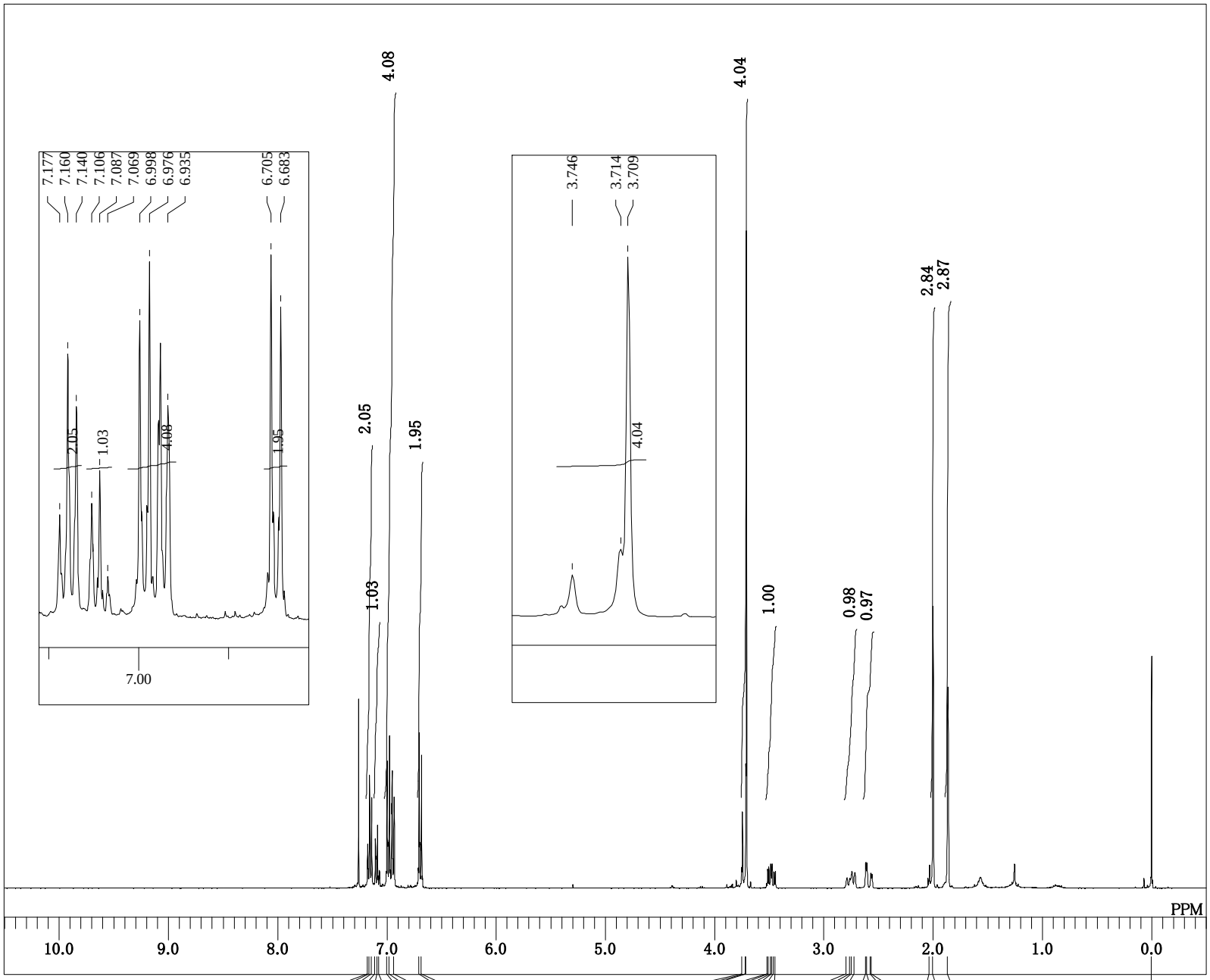
***cis*-4q**



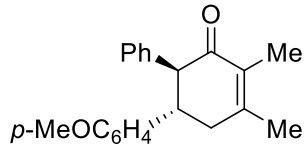
DFILE	cis_4q_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-10-13 21:18:11
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	14304
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	50



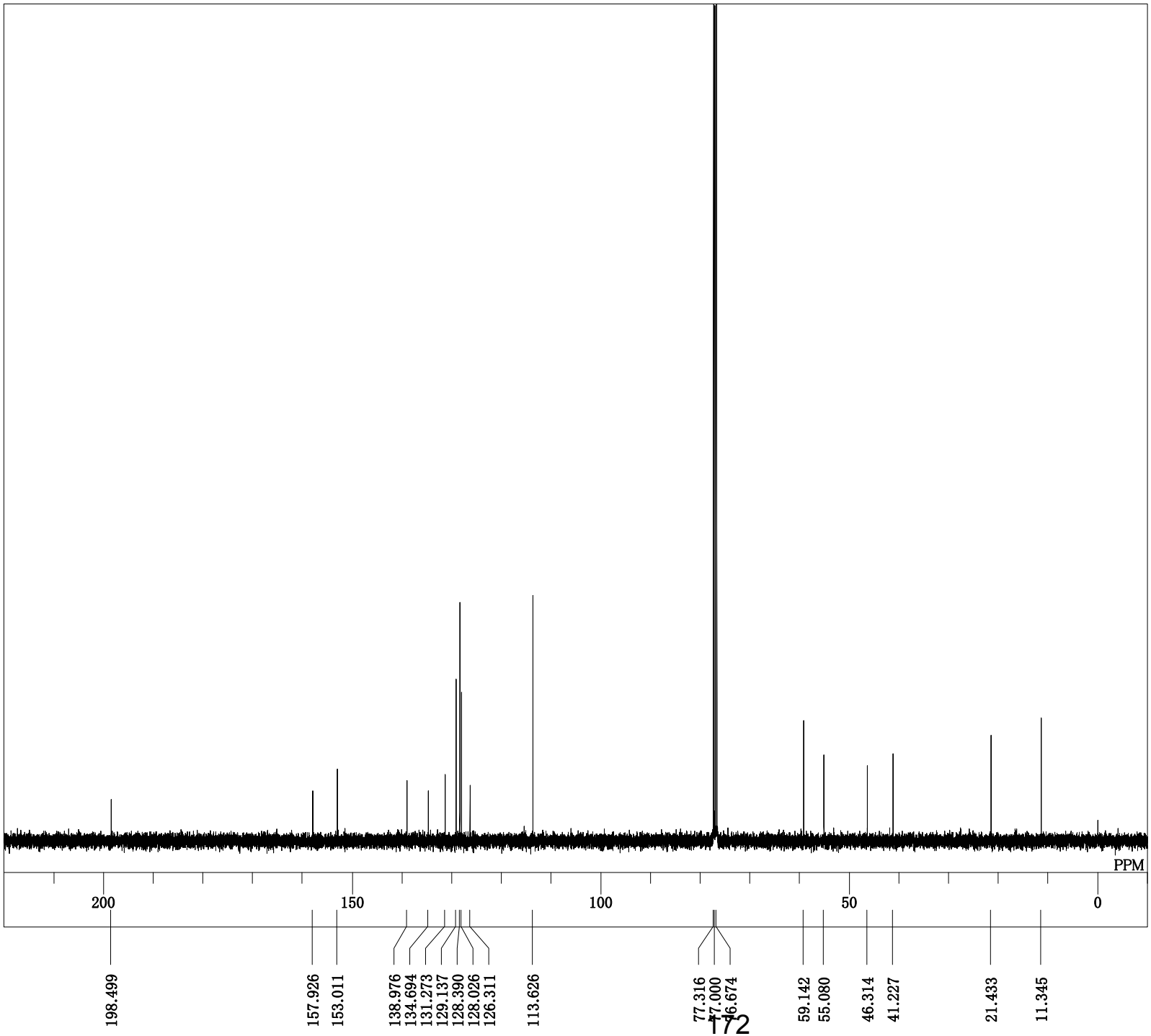
***cis*-4q**



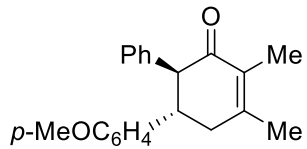
DFILE	trans_4q_1H.als
COMNT	single_pulse
DATIM	2020-11-04 19:38:56
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	19.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40



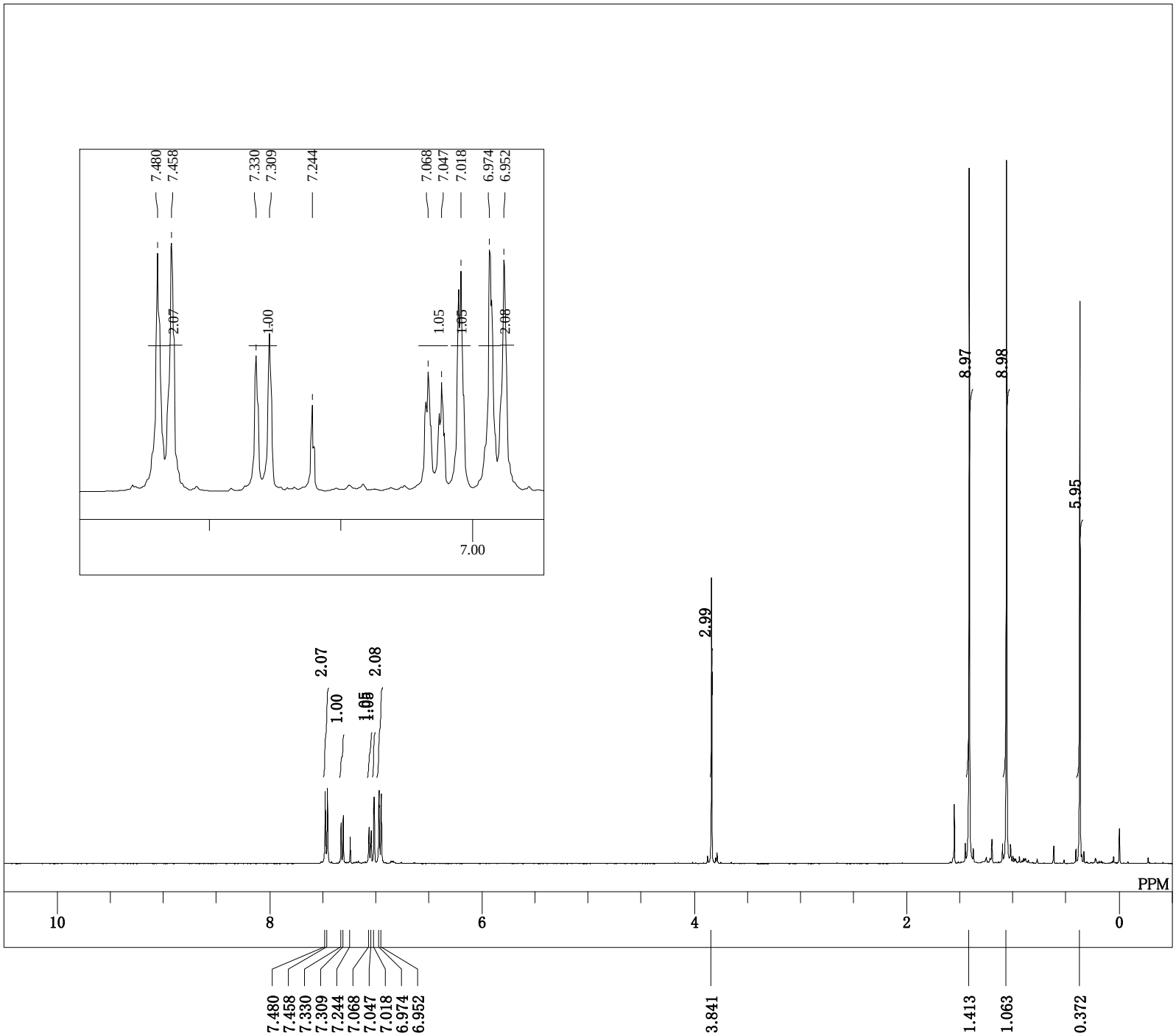
***trans*-4q**



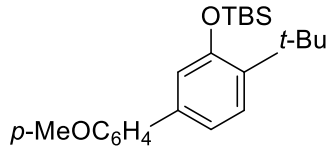
DFILE	trans_4q_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2020-11-04 19:40:09
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	1024
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	19.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



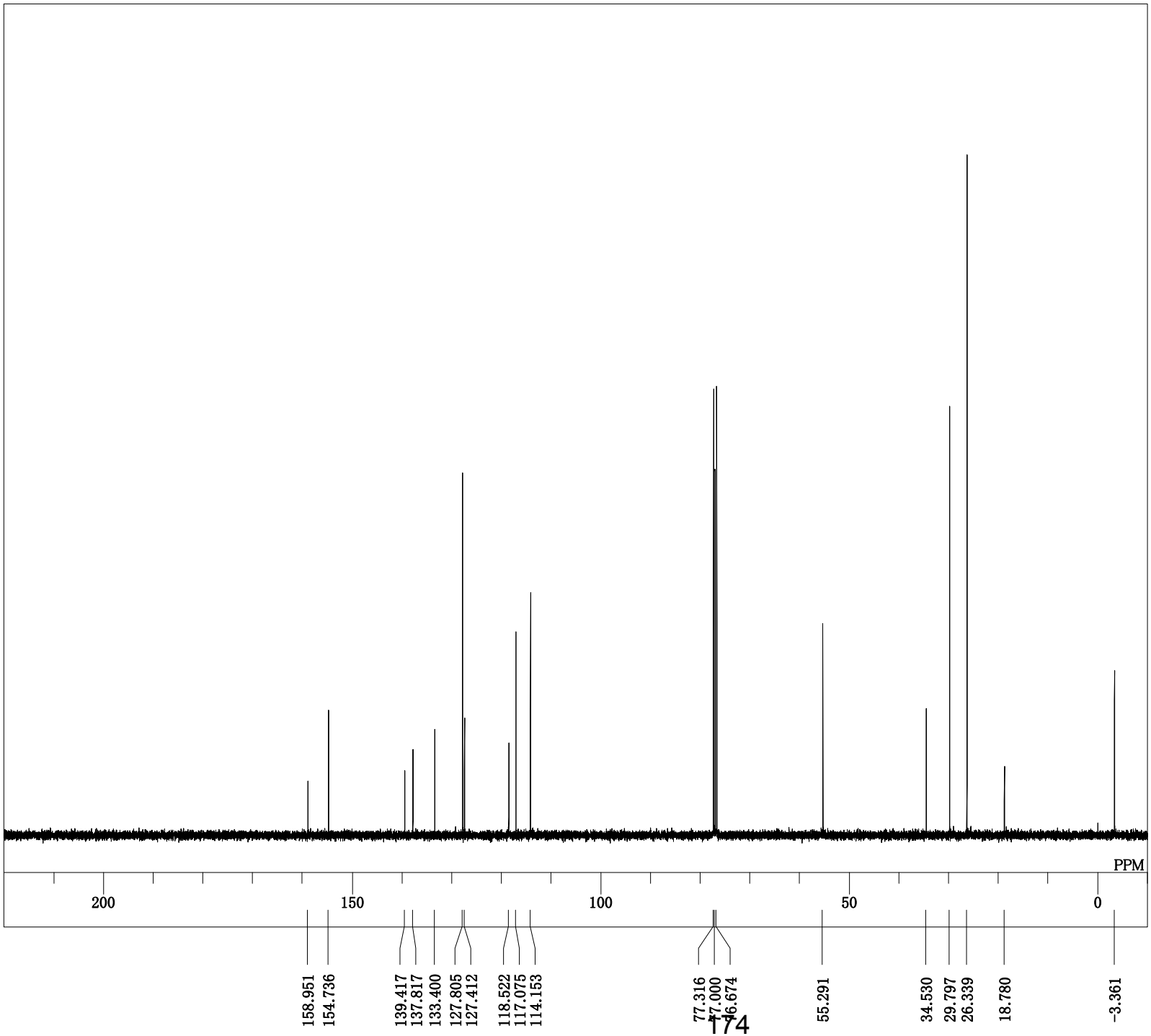
***trans*-4q**



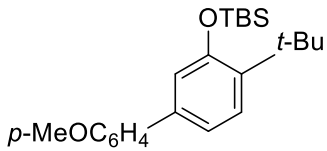
DFILE 8e_1H.als
COMNT single_pulse
DATIM 2021-05-08 16:28:23
OBNUC 1H
EXMOD proton.jxp
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.24 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 3.14 usec
IRNUC 1H
CTEMP 20.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 26



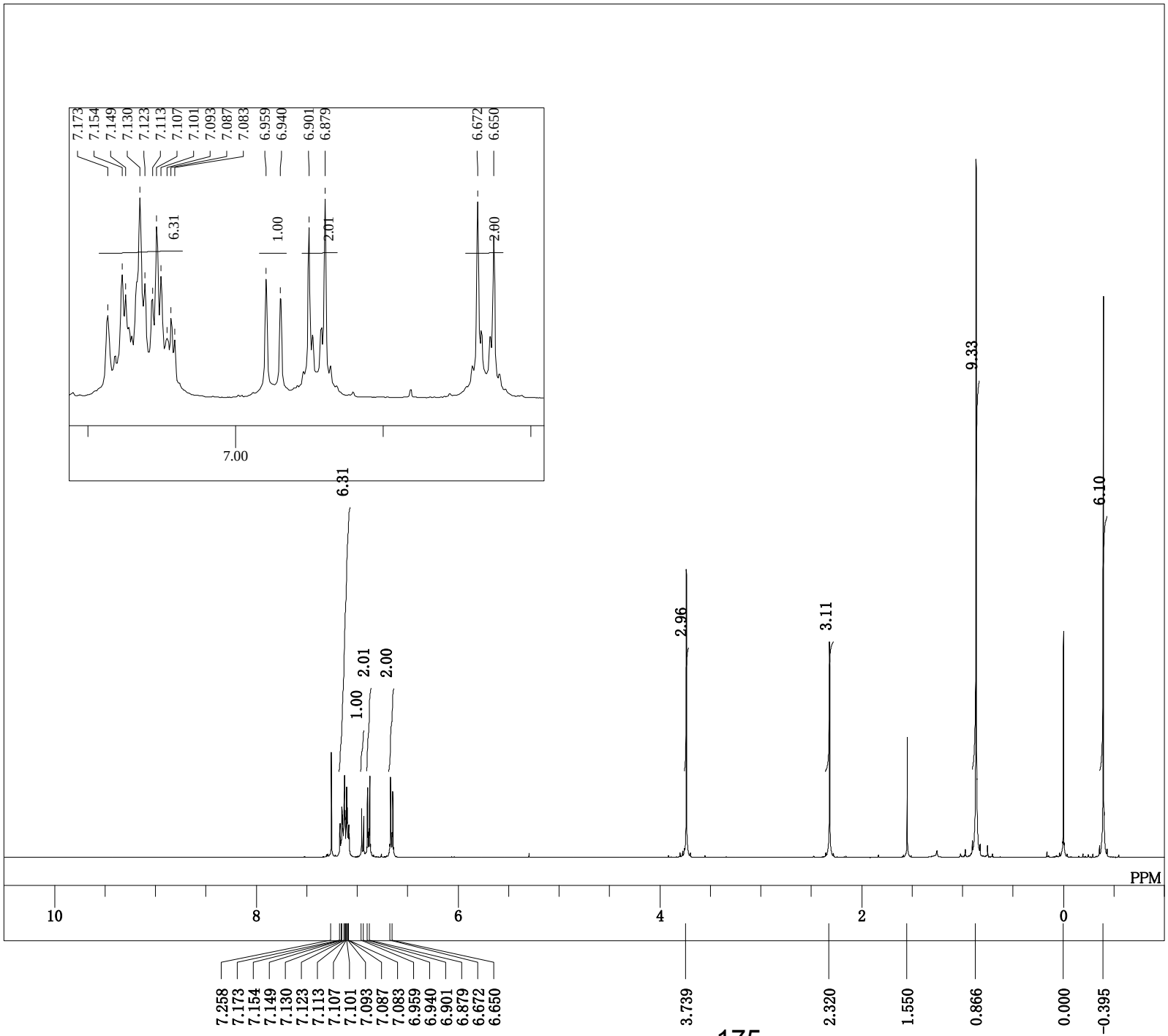
8e



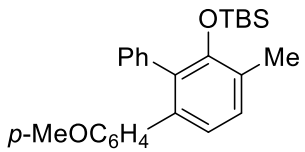
DFILE	8e_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2021-05-08 17:35:60
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	512
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50



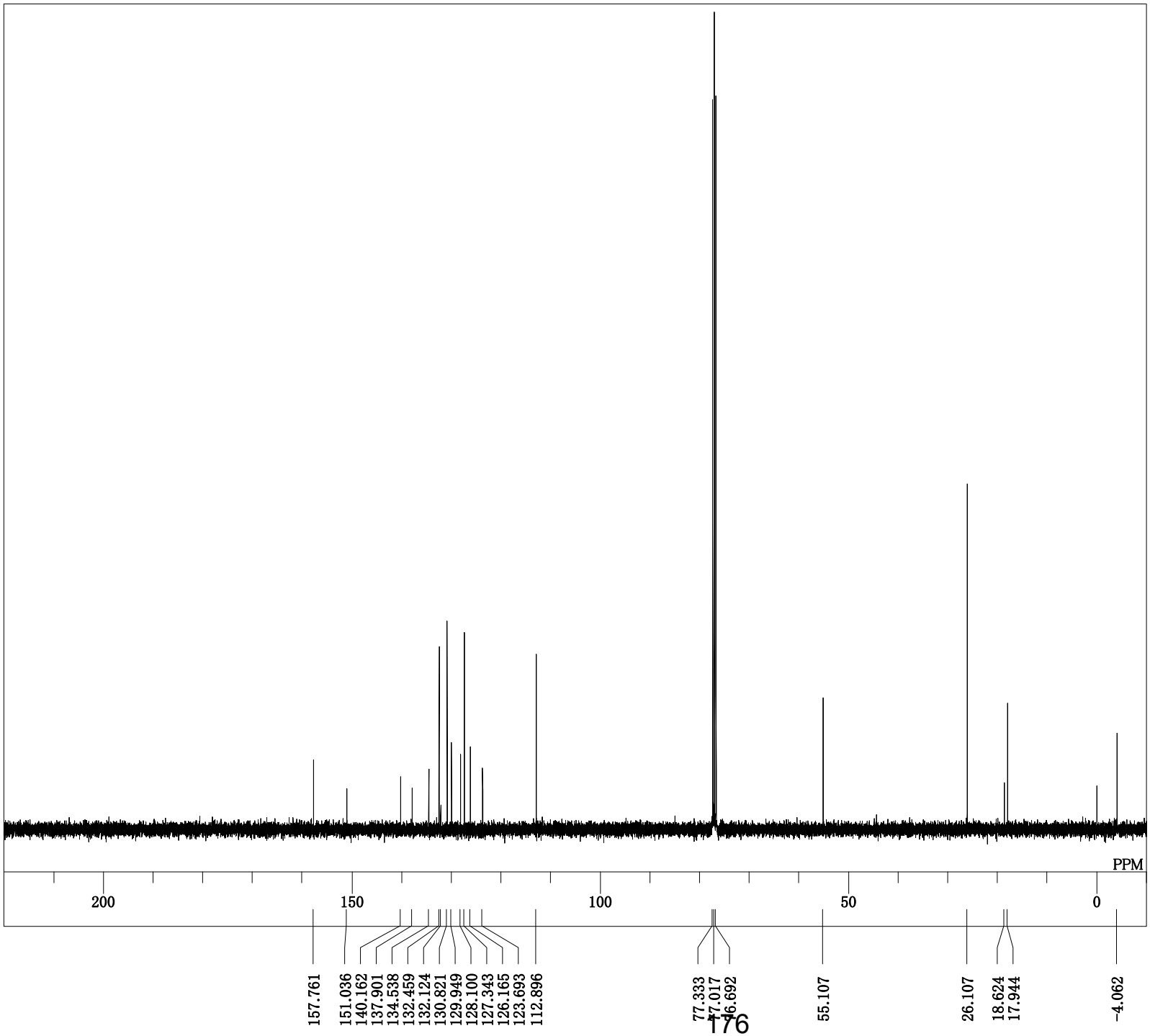
8e



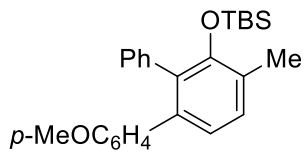
DFILE	8k_1H.als
COMNT	single_pulse
DATIM	2021-05-12 14:05:48
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.24 Hz
SCANS	8
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	3.14 usec
IRNUC	1H
CTEMP	20.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	36



8k



DFILE	8k_13C.als
COMNT	single pulse decoupled gated NOE
DATIM	2021-05-12 14:07:01
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	25000.00 Hz
SCANS	967
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.59 usec
IRNUC	1H
CTEMP	20.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	50



8k