

Supporting Information

Decarboxylative 1,2-Rearrangement of Cyclic Carbonates Promoted by Lewis Acid

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Contents

1. General Information	S1
2. Supporting Results and Discussion	
2.1 Screening of Solvents	S2
2.2 The Comparison of 1,2-Rearrangement	S3
2.3 Limitation of Decarboxylative 1,2-Rearrangement	S5
3. Preparation of Substrates	
3.1 General Procedure for the Synthesis of 1,2-Diols	S5
3.2 General Procedure for the Synthesis of Cyclic Carbonates	S15
4. General Procedure & Characterization Data	
4.1 Procedure for Conventional Pinacol Rearrangement	S27
4.2 Procedure for Decarboxylative 1,2-Rearrangement	S34
4.3 Procedure for House-Meinwald Rearrangement	S41
5. NMR Spectra	S46

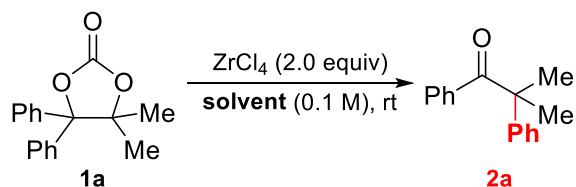
1. General Information

The ^1H and $^{13}\text{C}\{^1\text{H}\}$, and ^{19}F NMR spectra were recorded with a JEOL model AL-400 or ECS-400 spectrometer using CDCl_3 as the solvent. All chemical shifts are reported in ppm and coupling constants are in Hz. The ^1H and ^{13}C NMR chemical shifts are relative to tetramethylsilane; the resonance of residual protons of chloroform were used as an internal standard for ^1H ($\delta 7.26$ ppm) and all-d solvent peaks for ^{13}C ($\delta 77.0$ ppm). ^{19}F NMR chemical shifts are relative to hexafluorobenzene in CDCl_3 at $\delta = -163.0$ ppm (external reference). 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. The HPLC analysis were performed by Shimadzu SCL-10A chromatograph using Daicel Chiralpak[®] IF; the peak areas were obtained using Shimadzu SPD-M10A VP diode array detector/Shimadzu Class-VP. Column chromatography was conducted on silica gel (CHROMATREX PSQ 100B, Fuji Silysia Chemical, Ltd.). ZrCl_4 was purchased from Aldrich. The dehydrated dichloromethane (DCM) and dehydrated tetrahydrofuran (THF) were purchased from Kanto Chemical Co., Inc. Bis(trichloromethyl) carbonate was purchased from Tokyo Chemical Industry Co., Ltd., and used without further purification. AlCl_3 and HfCl_4 were purchased from FUJIFILM Wako Pure Chemical Co. Pyridine was distilled from KOH.

2. Supporting Result and Discussion

2.1 Screening of Solvents

Table S1. Screening of Solvents



Entry	solvent	Time (h)	Yield of 2a (%) ^[a]	RSM (%) ^[a,b]
1	CH_2Cl_2	45 min	94	0
2	1,2-DCE	45 min	99	0
3	chlorobenzene	45 min	99	0
4	toluene	6	16	83
5	Et_2O	12	0	98
6	THF	12	0	>99
7	CH_3CN	12	0	>99

[a] Isolated yield. [b] Recovery of starting material.
1,2-DCE = 1,2-dichloroethane

The use of halogen solvents such as 1,2-dichloroethane and chlorobenzene gave as the good results as dichloromethane (entries 2 and 3). When the reaction was carried out in toluene, desired product was obtained in only 16% yield (entry 3). No reaction was observed in the case of diethyl ether, tetrahydrofuran, and acetonitrile because these solvents coordinate to Lewis acid (entries 5, 6, and 7). Therefore, we chose dichloromethane as the best solvent.

2.2 The Comparison of 1,2-Rearrangement

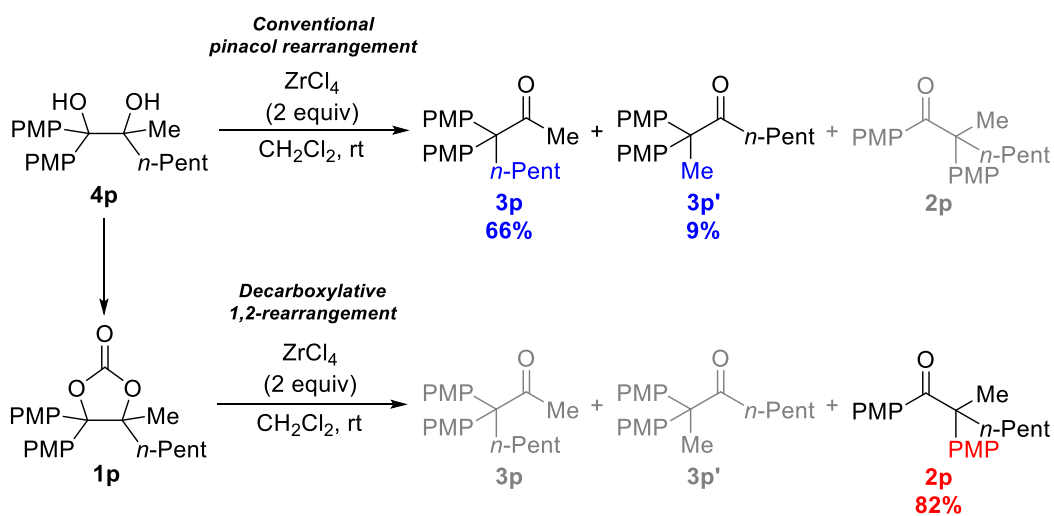
Table S2. The Comparison of each 1,2-Rearrangement

Conventional pinacol rearrangement

Decarboxylative 1,2-rearrangement

Entry	Ar ¹	Ar ²	Substrate (4 or 1)	Yield [%]		
				3	2	2'
1	<i>p</i> -ClC ₆ H ₄		4n	79	—	0
			1n	0	—	98
2	<i>p</i> -BrC ₆ H ₄		4o	94	—	0
			1o	0	—	84
3	Ph	PMP	4q	95	0	0
			1q	0	48	35

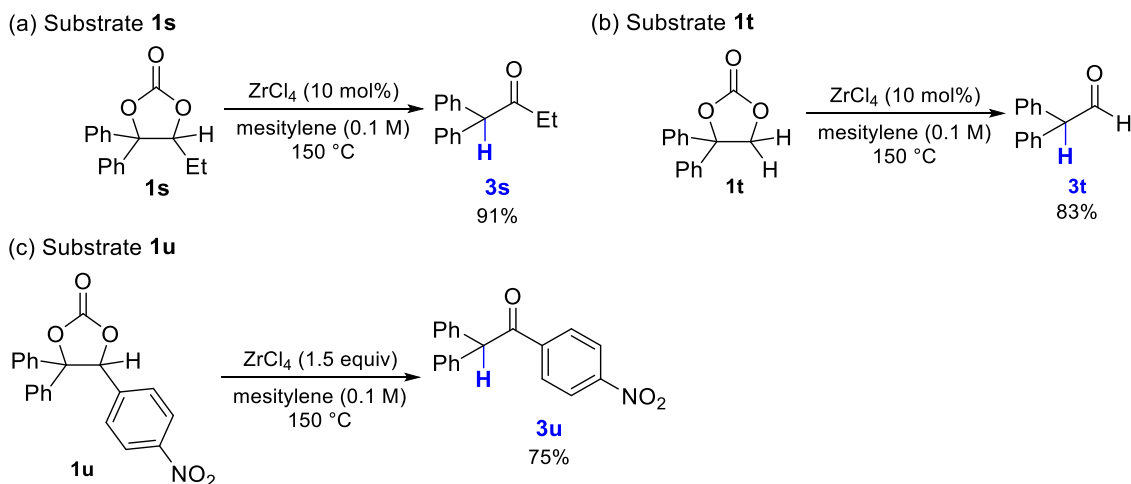
Scheme S1. The Comparison of 1,2-Rearrangements of 4p and 1p



The comparison of each 1,2-rearrangement of 1,2-diols **4** and corresponding cyclic carbonates **1** was investigated (Table S1, S2 and Scheme S1). When 1,2-diols **4** treated with zirconium (IV) chloride in dichloromethane at room temperature, the alkyl or hydride migration depending on the stability of carbocation occurred to afford **3** and/or **3'**. 1,2-rearrangement of diols containing a secondary alcohol moiety (**4i-m**) were resulted in the low selectivity of 1,2-migration. On the other hand, cyclic carbonates **1** were converted into 1,2-aryl migrated products **2** under the same reaction conditions.

2.3 Limitation of Decarboxylative 1,2-Rearrangement

Scheme S2. Limitation of Selectivity on Decarboxylative 1,2-Rearrangement

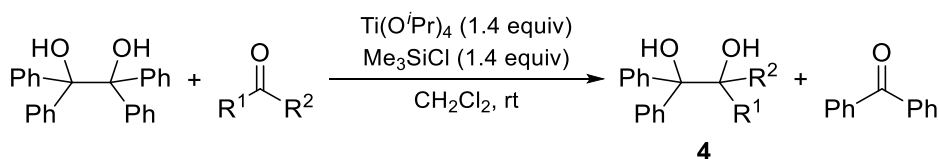


A few cyclic carbonates showed the similar selectivity with the corresponding 1,2-diols. The decarboxylation of substrates **1s**, **1t** and **1u** did not proceed under typical conditions (scheme 3 in manuscript). Although the reaction was carried out using zirconium chloride in mesitylene at 150 °C, only hydride-migrated products (**3s-3u**) were obtained.

3. Preparation of Substrates

3.1 General Procedure for the Synthesis of 1,2-Diols

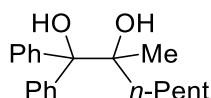
3.1.1 Procedure for the Preparation of 1,2-Diols (**4a-4m**, **4r**, **4s**, **4u**)



Diols **4a-4m**, **4r**, **4s**, **4u** were prepared according to the literature¹. Under N₂ atmosphere, benzopinacol (1.83 g, 5.00 mmol) and the corresponding aldehyde or ketone (10.0 mmol) were dissolved into dehydrate CH₂Cl₂ (25 mL). To the solution, Ti(O^{*i*}Pr)₄ (2.0 mL, 6.76 mmol) and Me₃SiCl (0.9 mL, 7.09 mmol) was added. The resulting solution was stirred at room temperature. The reaction was continuously monitored by thin layer chromatography. When no more benzopinacol was detectable, the reaction was quenched by sat. NH₄Cl aq. and sat. NaHCO₃ aq., then, filtered through a celite[®] pad. The resulting mixture was extracted three times with CH₂Cl₂. The combined organic layer was dried

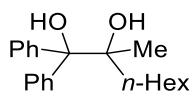
over Na₂SO₄, and the solvent was removed under reduced pressure. The residue was purified by column chromatography to afford the desired 1,2-diols (**4**).

The synthesis and characterization data for 1,2-diols **4a-4c**, **4f-4i**, **4m**, **4u** were previously reported¹.



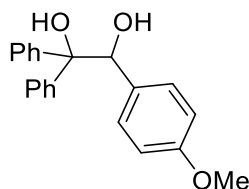
2-methyl-1,1-diphenylheptane-1,2-diol (**4d**)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 30:1 as an eluent; Colorless oil (1.34 g, 90%); ¹H NMR (400 MHz, CDCl₃): δ = 0.85 (t, *J* = 7.6 Hz, 3H), 1.15-1.37 (m, 8H), 1.43-1.48 (m, 2H), 1.59-1.72 (m, 1H), 2.08 (brs, 1H), 2.83 (brs, 1H), 7.24-7.32 (m, 6H), 7.61-7.64 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ = 14.0, 22.7, 23.0, 23.2, 32.5, 37.4, 78.1, 83.1, 126.9, 126.9, 127.59, 127.61, 128.3, 128.4, 144.5, 144.6; IR (neat): 3494, 2954, 2870, 1492, 1447, 1375, 1158, 1035, 754, 705 cm⁻¹; HRMS (ESI): [M+H-H₂O]⁺ calcd for C₂₀H₂₅O⁺, 281.1900; found, *m/z* 281.1898.



2-methyl-1,1-diphenyloctane-1,2-diol (**4e**)²

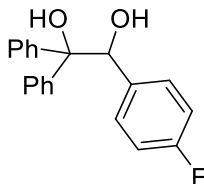
Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 30:1 as an eluent; White solid (953.0 mg, 61%); mp 68 °C (lit.², 65-68 °C); ¹H NMR (400 MHz, CDCl₃): δ = 0.85 (t, *J* = 7.6 Hz, 3H), 1.23-1.72 (m, 13H), 2.03 (brs, 1H), 2.79 (brs, 1H), 7.22-7.32 (m, 6H), 7.61-7.65 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ = 14.1, 22.6, 23.0, 23.5, 29.9, 31.9, 37.4, 78.1, 83.1, 126.8, 126.9, 127.5, 127.6, 128.3, 128.4, 144.5, 144.6; IR (KBr): 3557, 3446, 2994, 1445, 1372, 1151, 1037, 753, 705, 640 cm⁻¹; HRMS (ESI): [M+H-H₂O]⁺ calcd for C₂₁H₂₇O⁺, 295.2057; found, *m/z* 295.2056.



2-(4-methoxyphenyl)-1,1-diphenylethane-1,2-diol (**4j**)³

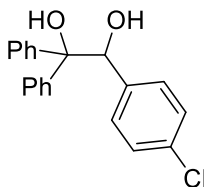
Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 10:1 as an eluent; White solid (1.36 g, 85%); mp 188-195 °C (lit.³, 179-180 °C); ¹H NMR (400 MHz,

CDCl₃): δ = 2.33 (brs, 1H), 3.11 (brs, 1H), 3.74 (s, 3H), 5.60 (s, 1H), 6.68 (d, J = 9.2 Hz, 2H), 7.00 (d, J = 9.2 Hz, 2H), 7.06-7.17 (m, 5H), 7.30 (t, J = 7.2 Hz, 1H), 7.40 (t, J = 7.2 Hz, 2H), 7.69 (d, J = 7.2 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 55.1, 77.6, 80.7, 112.9, 126.2, 126.7, 126.9, 127.3, 127.7, 128.4, 129.2, 130.9, 143.5, 145.1, 159.1.



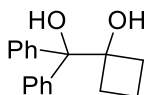
2-(4-fluorophenyl)-1,1-diphenylethane-1,2-diol (**4k**)³

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 10:1 as an eluent; White solid (1.39 g, 90%); mp 157-162 °C (lit.³, 148-150 °C); ¹H NMR (400 MHz, CDCl₃): δ = 2.40 (brs, 1H), 3.09 (brs, 1H), 5.62 (s, 1H), 6.82 (t, J = 7.6 Hz, 2H), 7.00-7.17 (m, 7H), 7.31 (t, J = 7.6 Hz, 1H), 7.41 (t, J = 7.6 Hz, 2H), 7.68 (t, J = 7.6 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 77.3, 80.7, 114.2, 114.3 (d, J = 21.1 Hz), 126.1, 126.9, 126.9, 127.5, 127.7, 128.6, 129.7 (d, J = 8.6 Hz), 134.5 (d, J = 2.9 Hz), 143.1, 144.9, 162.3 (d, J = 244.3 Hz); ¹⁹F NMR (373 MHz, CDCl₃): δ = -116.0.



2-(4-chlorophenyl)-1,1-diphenylethane-1,2-diol (**4l**)³

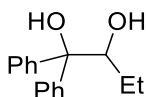
Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 5:1 as an eluent; White solid (1.48 g, 91%); mp 180-182 °C (lit.³, 182-183 °C); ¹H NMR (400 MHz, CDCl₃): δ = 2.45 (brs, 1H), 3.08 (brs, 1H), 5.60 (s, 1H), 6.97 (d, J = 8.4 Hz, 2H), 7.02-7.15 (m, 7H), 7.29-7.33 (m, 1H), 7.39-7.43 (m, 2H), 7.67 (d, J = 7.6 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 77.4, 80.7, 126.1, 126.9, 126.9, 127.5 (\times 2C), 127.8, 128.6, 129.4, 133.4, 137.3, 143.0, 144.8.



1-(hydroxydiphenylmethyl)cyclobutan-1-ol (**4r**)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 10:1 as an

eluent; White solid (quant.); mp 145-146 °C (lit.⁴, 144.5-145.5 °C); ¹H NMR (400 MHz, CDCl₃): δ = 1.05 (brs, 1H), 1.74-1.85 (m, 1H), 2.04-2.17 (m, 3H), 2.63-2.70 (m, 2H), 2.85 (brs, 1H), 7.24-7.34 (m, 6H), 7.47-7.50 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ = 13.1, 32.9, 80.6, 81.2, 127.2, 127.8, 127.9, 144.1; IR (KBr): 3544, 3434, 2999, 1447, 1371, 1258, 1121, 1048, 753, 709 cm⁻¹; HRMS (ESI): [M+H-H₂O]⁺ calcd for C₁₇H₁₇O⁺, 237.1274; found, m/z 237.1274.

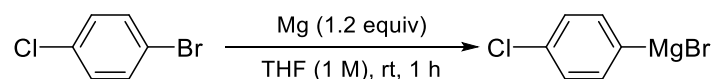


1,1-diphenylbutane-1,2-diol (4s)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 15:1 as an eluent; White solid (860.2 mg, 71%); mp 114 °C; ¹H NMR (400 MHz, CDCl₃): δ = 0.97 (t, *J* = 7.2 Hz, 3H), 1.32-1.52 (m, 2H), 1.82 (brs, 1H), 2.98 (brs, 1H), 4.48 (dd, *J* = 2.4, 10.0 Hz, 1H), 7.17-7.37 (m, 6H), 7.42-7.44 (m, 2H), 7.59-7.62 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 11.0, 23.3, 77.3, 80.1, 125.6, 126.1, 126.7, 127.2, 128.2, 128.6, 143.8, 145.8; IR (KBr): 3513, 2968, 1492, 1447, 1170, 1096, 969, 894, 751, 695 cm⁻¹; HRMS (ESI): [M+H-H₂O]⁺ calcd for C₁₆H₁₇O⁺, 225.1274; found, m/z 225.1275.

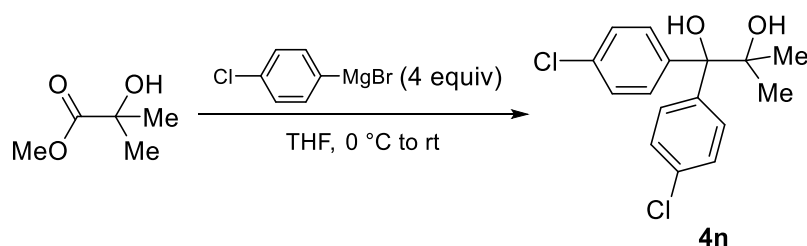
3.1.2 Procedure for the Preparation of 1,2-Diol (4n)

Preparation of *p*-chlorophenylmagnesium bromide

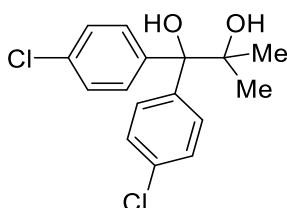


A 100 mL three-neckround bottom flask equipped with a reflux condenser and dropping funnel was charged with magnesium turnings (1.46 g, 60.0 mmol). The system was evacuated, flame dried, cooled, and backfilled with Ar. The magnesium turnings were stirred vigorously at room temperature for 30 min. Then, the turnings were suspended in THF (10 mL) and a small crystal of I₂ was added. The dropping funnel was charged with solution of 1-chloro-4-bromobenzene (50.0 mmol) in THF (40 mL), and the solution was added to the magnesium turnings dropwise over 30 min. The reaction mixture was maintained at room temperature for additional 1 h. The reaction mixture was transferred *via* cannula into a flame dried Schlenk flask.

Grignard Reaction



Under N₂ atmosphere, methyl-2-hydroxyisobutyrate (0.57 mL, 5.0 mmol) was dissolved into THF (5.0 mL). After cooling at 0 °C, a solution of *p*-chlorophenylmagnesium bromide (20 mL, 20 mmol, 1 M solution in THF) was added dropwise, and the reaction mixture was warmed to room temperature. The reaction progress was continuously monitored by thin layer chromatography. Upon completion, the reaction was quenched by sat. NH₄Cl aq., and the mixture was extracted three times with EtOAc. The combined organic layer was washed with brine, dried over Na₂SO₄, and the solvent was removed under reduced pressure. The residue was purified by silica gel flash column chromatography as indicated below to afford the 1,2-diol (**4n**).



1,1-bis(4-chlorophenyl)-2-methylpropane-1,2-diol (**4n**)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 5:1 as an eluent; Colorless oil (1.27 g, 82%); ¹H NMR (400 MHz, CDCl₃): δ = 1.31 (s, 6H), 2.04 (brs, 1H), 2.71 (brs, 1H), 7.27 (d, *J* = 8.6 Hz, 4H), 7.57 (d, *J* = 8.6 Hz, 4H); ¹³C NMR (100 MHz, CDCl₃): δ = 26.6, 76.4, 81.8, 127.8, 129.7, 133.2, 142.6; IR (neat): 3473, 2985, 2941, 1490, 1401, 1094, 1014, 901, 814, 574 cm⁻¹; HRMS (ESI): [M+H-H₂O]⁺ calcd for C₁₆H₁₅Cl₂O⁺, 293.0495; found, *m/z* 293.0496.

3.1.3 Procedure for the Preparation of 1,2-Diol (**4o**)

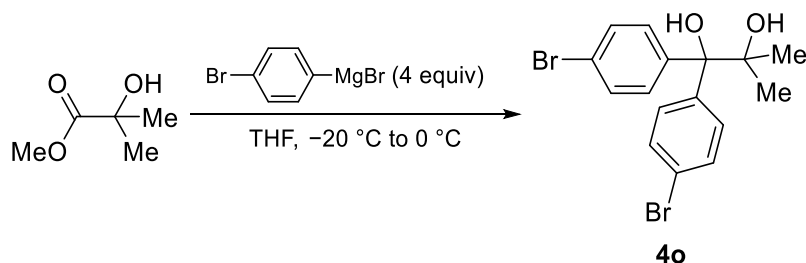
Preparation of *p*-bromophenylmagnesium bromide



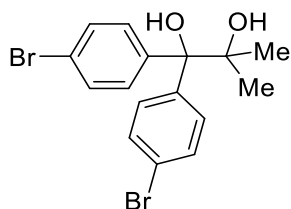
A solution of *p*-bromophenylmagnesium bromide was prepared with reference to the

literature procedure⁵. A dried 30 mL two-neckround bottom flask equipped with a septum was charged with magnesium turnings (486 mg, 20.0 mmol). The system was evacuated, flame dried, cooled, and backfilled with Ar. The magnesium turnings were stirred vigorously at room temperature for 30 min. Then, the turnings were suspended in THF (45 mL) and LiCl⁶ (971 mg, 22.9 mmol) was added. After LiCl was completely dissolved, the magnesium was activated with DIBAL-H (0.09 mL, 0.09 mmol, 1 M in toluene). After 5 min of stirring, the solution was cooled to -20 °C and 1,4-dibromobenzene (6.75 g, 28.6 mmol) was added in one portion. The reaction mixture was maintained at -20 °C until the magnesium turnings disappeared. The reaction mixture was used up at once without storage in the next reaction.

Grignard Reaction



Under N₂ atmosphere, methyl-2-hydroxyisobutyrate (0.57 mL, 5.0 mmol) was dissolved into THF (5.0 mL). After cooling at -20 °C, a solution of the Grignard reagent (45 mL, 20 mmol, 0.44 M solution in THF) was added dropwise, and the reaction mixture was warmed to 0 °C. The reaction progress was continuously monitored by thin layer chromatography. Upon completion, the reaction was quenched by sat. NH₄Cl aq., and the mixture was extracted three times with EtOAc. The combined organic layer was washed with brine, dried over Na₂SO₄, and the solvent was removed under reduced pressure. The residue was purified by silica gel flash column chromatography as indicated below to afford 1,1-bis(4-bromophenyl)-2-methylpropane-1,2-diol **4o**.

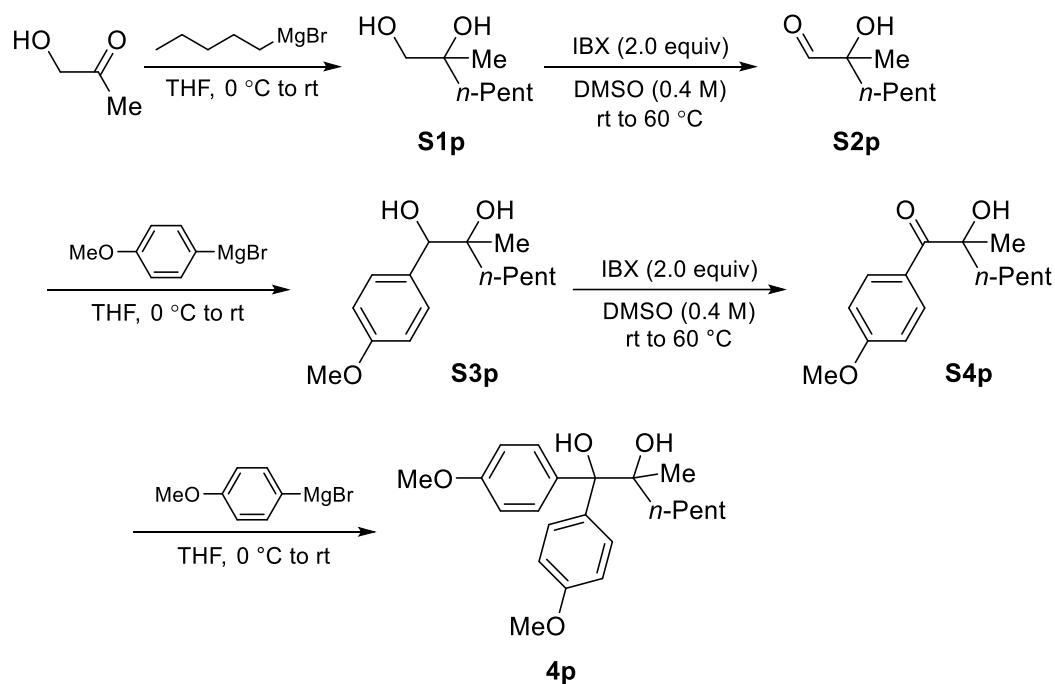


1,1-bis(4-bromophenyl)-2-methylpropane-1,2-diol (**4o**)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 5:1 as an eluent; White solid (780.0 mg, 39%); mp 129-131 °C; ¹H NMR (400 MHz, CDCl₃): δ = 1.31 (s, 6H), 2.01 (brs, 1H), 2.69 (brs, 1H), 7.42 (d, *J* = 9.0 Hz, 4H), 7.51 (d, *J* = 9.0 Hz,

4H); ^{13}C NMR (100 MHz, CDCl_3): δ = 26.6, 76.3, 81.9, 121.5, 130.0, 130.8, 143.1; IR (KBr): 3486, 2966, 1485, 1397, 1154, 1010, 808, 581 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}-\text{H}_2\text{O}]^+$ calcd for $\text{C}_{16}\text{H}_{15}\text{Br}_2\text{O}^+$, 380.9484; found, m/z 380.9482.

3.1.4 Procedure for the Preparation of 1,2-Diol (4p)



Step 1: Under N_2 atmosphere, hydroxyacetone (1.4 mL, 20 mmol) was dissolved into THF (20 mL). After cooling at $0\text{ }^\circ\text{C}$, a solution of pentylmagnesium bromide⁷ (50 mL, 50 mmol, 1 M solution in THF) was added dropwise, and the reaction mixture was warmed to room temperature. The reaction progress was continuously monitored by thin layer chromatography. Upon completion, the reaction was quenched by sat. NH_4Cl aq., and the mixture was extracted several times with EtOAc. The combined organic layer was washed with brine, dried over Na_2SO_4 , and the solvent was removed under reduced pressure. The residue was purified by silica gel flash column chromatography using $n\text{-hexane}/\text{EtOAc} = 1:1$ as an eluent to afford **S1p** as a colorless oil (1.61 g, 55%). ^1H NMR (400 MHz, CDCl_3): δ = 0.89 (t, $J = 7.2$ Hz, 3H), 1.15 (s, 3H), 1.26-1.37 (m, 6H), 1.45-1.49 (m, 2H), 2.72 (brs, 1H), 3.08 (brs, 1H), 3.39 (d, $J = 11.2$ Hz, 1H), 3.45 (d, $J = 11.2$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.0, 22.6, 23.0, 23.4, 32.4, 38.6, 69.6, 73.1; IR (neat): 3381, 2934, 1466, 1139, 1044, 609 cm^{-1} ; HRMS (ESI): $[\text{M}]^+$ calcd for $\text{C}_8\text{H}_{18}\text{O}_2^+$, 147.1380; found, m/z 147.1371.

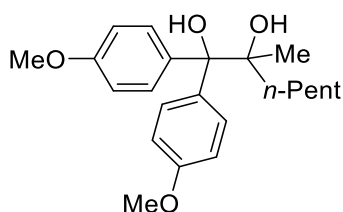
Step 2: IBX⁸ (2-iodoxybenzoic acid) solution (0.8 M in DMSO) was prepared by IBX (6.2 g, 22 mmol) dissolved in DMSO (28 mL). The dissolution of IBX in DMSO

completely requires about 5-20 min. To the solution of IBX in DMSO, 1,2-diol **S1p** (1.61 g, 11.0 mmol) was added and the reaction mixture was stirred at room temperature. After 10 min of stirring, the solution was heated to 60 °C. The reaction was monitored by thin layer chromatography until consumption of starting material was observed. The reaction mixture was cooled to room temperature and quenched by water. The mixture was filtered through a celite[®] pad. The filter cake was washed with Et₂O, and the combined filtrates was extracted three times with Et₂O. The combined organic layer was washed with sat. NaHCO₃ aq. (3×), brine (3×), dried over Na₂SO₄, and the solvent was removed under reduced pressure. The resulting compound **S2p** was pure enough (colorless oil, 1.01 g, 64%), and so used in the next reaction without purification.

Step 3: Under N₂ atmosphere, the aldehyde **S2p** (1.0 g, 7.0 mmol) was dissolved into THF (7.0 mL). After cooling at 0 °C, a solution of *p*-methoxyphenylmagnesium bromide (14 mL, 14 mmol, 1 M solution in THF) was added dropwise, and the reaction mixture was warmed to room temperature. The reaction progress was continuously monitored by thin layer chromatography. Upon completion, the reaction was quenched by sat. NH₄Cl aq., and the mixture was extracted three times with EtOAc. The combined organic layer was washed with brine, dried over Na₂SO₄, and the solvent was removed under reduced pressure. The residue was purified by silica gel flash column chromatography using *n*-hexane /EtOAc = 3:1 as an eluent to afford **S3p** as a colorless oil (1.39 g, 79%). ¹H NMR (400 MHz, CDCl₃; Mixture of diastereoisomers in a 55:45 ratio. The structure of major isomer is not clear.): δ = 0.84-0.91 (m, 3H), 1.00 (s, 1.65H), 1.19-1.47 (m, 9.35H), 1.91 (s, 0.44H), 2.01 (s, 0.56H), 2.44 (brs, 0.45H), 2.54 (brs, 0.55H), 3.81 (s, 3H), 4.49 (s, 1H), 6.86-6.88 (m, 2H), 7.26-7.31 (m, 2H); ¹³C NMR (100 MHz, CDCl₃; Mixture of diastereoisomers.): δ = 14.0, 22.7, 23.0, 23.2, 32.5, 37.4, 78.1, 83.1, 126.9, 126.9, 127.6, 127.6, 128.3, 128.4, 144.5, 144.6; IR (neat): 3494, 2954, 2870, 1492, 1447, 1375, 1158, 1035, 754, 705 cm⁻¹; HRMS (ESI): [M+H-H₂O]⁺ calcd for C₂₀H₂₅O⁺, 281.1900; found, m/z 281.1898.

Step 4: The hydroxyketone **S3p** was prepared as a colorless oil from IBX (3.1 g, 11 mmol) and the aldehyde **S4p** (1.4 g, 5.5 mmol) in DMSO (14 mL) according to the same as procedure **Step 2** (1.28 g, 93%, without purification).

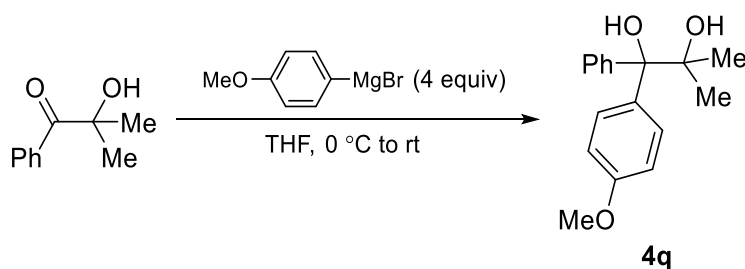
Step 5: 1,1-bis(4-methoxyphenyl)-2-methylheptane-1,2-diol (**4p**) was prepared as a colorless oil from *p*-methoxyphenylmagnesium bromide (10 mL, 10 mmol, 1 M solution in THF) and the hydroxyketone **S4p** (1.3 g, 5.0 mmol) in THF (5.0 mL) according to the same as procedure **Step 3** (purified by flash column chromatography as indicated below, 1.5 g, 86 %).



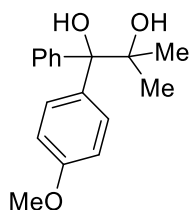
1,1-bis(4-methoxyphenyl)-2-methylheptane-1,2-diol (**4p**)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 5:1 as an eluent; Colorless oil (1.54 g, 86%); ^1H NMR (400 MHz, CDCl_3): δ = 0.86 (t, J = 7.2 Hz, 3H), 1.20-1.50 (m, 10H), 1.69 (m, 1H), 2.15 (brs, 1H), 2.71 (brs, 1H), 3.80 (s, 6H), 6.82 (d, J = 8.8 Hz, 4H), 7.52 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.1, 22.7, 22.9, 23.2, 32.5, 37.4, 55.1, 78.2, 82.7, 112.8 (\times 2C), 129.6, 129.7, 136.9, 137.0, 158.3, 158.3; IR (neat): 3507, 2953, 2836, 1608, 1509, 1248, 1179, 1036, 831, 734, 584 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}-\text{H}_2\text{O}]^+$ calcd for $\text{C}_{22}\text{H}_{29}\text{O}_3^+$, 341.2111; found, m/z 341.2113.

3.1.5 Procedure for the Preparation of 1,2-Diol (**4q**)



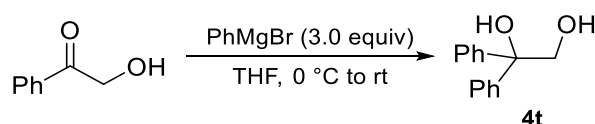
Under N_2 atmosphere, 2-hydroxy-2-methylpropiophenone (0.76 mL, 5.0 mmol) was dissolved into THF (5.0 mL). After cooling at 0 $^\circ\text{C}$, a solution of *p*-methoxyphenylmagnesium bromide (20 mL, 20 mmol, 1 M solution in THF) was added dropwise, and the reaction mixture was warmed to room temperature. The reaction progress was continuously monitored by thin layer chromatography. Upon completion, the reaction was quenched by sat. NH_4Cl aq., and the mixture was extracted three times with EtOAc. The combined organic layer was washed with brine, dried over Na_2SO_4 , and the solvent was removed under reduced pressure. The residue was purified by silica gel flash column chromatography as indicated below to afford 1-(4-methoxyphenyl)-2-methyl-1-phenylpropane-1,2-diol **4q**.



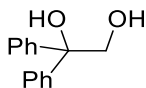
1-(4-methoxyphenyl)-2-methyl-1-phenylpropane-1,2-diol (**4q**)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 4:1 as an eluent; Colorless oil (953.0 mg, 70%); ^1H NMR (400 MHz, CDCl_3): δ = 1.30 (s, 3H), 1.33 (s, 3H), 2.28 (brs, 1H), 2.74 (brs, 1H), 6.82 (d, J = 9.2 Hz, 2H), 7.24-7.31 (m, 3H), 7.56 (d, J = 9.2 Hz, 2H), 7.61 (d, J = 6.8 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 26.4, 26.7, 55.1, 76.5, 82.3, 112.9, 126.9, 127.5, 128.2, 129.6, 136.8, 144.6, 158.4; IR (neat): 3502, 2995, 2836, 1608, 1509, 1251, 1166, 1035, 838, 753, 706 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}-\text{H}_2\text{O}]^+$ calcd for $\text{C}_{17}\text{H}_{19}\text{O}_2^+$, 255.1379; found, m/z 255.1375.

3.1.6 Procedure for the Preparation of 1,2-Diol (**4t**)



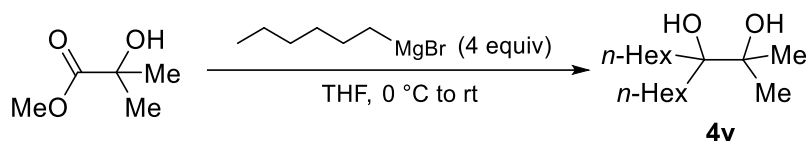
Under N_2 atmosphere, 2-hydroxyacetophenone (0.62 mL, 5.0 mmol) was dissolved into THF (5.0 mL). After cooling at 0 $^\circ\text{C}$, a solution of phenylmagnesium bromide (15 mL, 15 mmol, 1 M solution in THF) was added dropwise, and the reaction mixture was warmed to room temperature. The reaction progress was continuously monitored by thin layer chromatography. Upon completion, the reaction was quenched by sat. NH_4Cl aq., and the mixture was extracted several times with EtOAc. The combined organic layer was washed with brine, dried over Na_2SO_4 , and the solvent was removed under reduced pressure. The residue was purified by silica gel flash column chromatography as indicated below to afford 1,1-diphenylethane-1,2-diol **4t**.



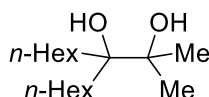
1,1-diphenylethane-1,2-diol (**4t**)⁹

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 3:1 as an eluent; White solid (857.0 mg, 80%); mp 117-119 $^\circ\text{C}$ (lit.⁹, 120-121 $^\circ\text{C}$); ^1H NMR (400 MHz, CDCl_3): δ = 1.88 (brs, 1H), 3.19 (brs, 1H), 4.17 (s, 2H), 7.26-7.45 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3): δ = 69.4, 78.5, 126.37, 127.46, 128.42, 143.73.

3.1.7 Procedure for the Preparation of 1,2-Diol (**4v**)



Under N₂ atmosphere, 2-hydroxy-2-methylpropiophenone (0.76 mL, 5.0 mmol) was dissolved into THF (5.0 mL). After cooling at 0 °C, a solution of hexylmagnesium bromide⁷ (20 mL, 20 mmol, 1 M solution in THF) was added dropwise, and the reaction mixture was warmed to room temperature. The reaction progress was continuously monitored by thin layer chromatography. Upon completion, the reaction was quenched by sat. NH₄Cl aq., and the mixture was extracted several times with EtOAc. The combined organic layer was washed with brine, dried over Na₂SO₄, and the solvent was removed under reduced pressure. The residue was purified by silica gel flash column chromatography as indicated below to afford 3-hexyl-2-methylnonane-2,3-diol **4v**.

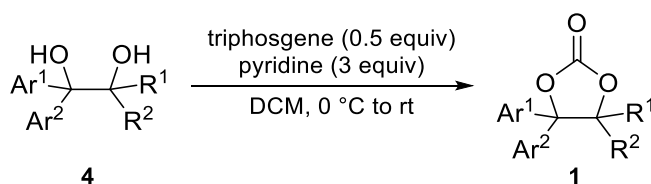


3-hexyl-2-methylnonane-2,3-diol (**4v**)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 5:1 as an eluent; Colorless oil (294.9 mg, 23%); ¹H NMR (400 MHz, CDCl₃): δ = 0.89 (t, *J* = 7.2 Hz, 6H), 1.23-1.38 (m, 22H), 1.53-1.55 (m, 4H), 1.91 (s, 1H), 2.07 (s, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 14.1, 22.7, 24.5, 25.3, 30.3, 31.8, 35.0, 75.7, 77.7; IR (neat): 3445, 2955, 2858, 1466, 1379, 1135, 953 cm⁻¹; HRMS (ESI): [M+H-H₂O]⁺ calcd for C₁₆H₃₃O⁺, 242.2604; found, *m/z* 242.2613.

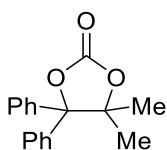
3.2 General Procedure for the Synthesis of Cyclic Carbonate

3.2.1 Procedure for the Preparation of Cyclic Carbonates (**1a-1v**)



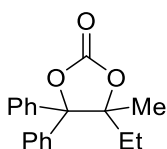
Cyclic carbonate **1a-1v** were prepared according to the literature¹⁰. Under N₂ atmosphere, the corresponding 1,2-diol (2.00 mmol) and pyridine (6.00 mmol, 3.0 equiv)

were dissolved in dehydrate CH_2Cl_2 (5.0 mL), and the solution was cooled to 0 °C. To the solution, triphosgene (1.00 mmol, 0.5 equiv) was added in batches. The reaction mixture was then allowed to stir at room temperature, and the reaction progress was monitored by thin layer chromatography. Upon completion, the reaction was quenched by sat. NH_4Cl aq., and extracted three times with CH_2Cl_2 . The combined organic layer was dried over Na_2SO_4 , and the solvent was removed under reduced pressure. The residue was purified by column chromatography to obtain the cyclic carbonate (**1a-1v**).



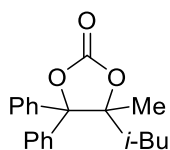
4,4-dimethyl-5,5-diphenyl-1,3-dioxolan-2-one (**1a**)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 10:1 as an eluent; White solid (quant.); mp 82 °C; ^1H NMR (400 MHz, CDCl_3): δ = 1.42 (s, 6H), 7.35-7.44 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3): δ = 25.6, 89.0, 92.2, 126.4, 128.4, 128.5, 137.8, 153.8; IR (KBr): 2980, 1793, 1493, 1446, 1389, 1267, 1044, 757, 708 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{17}\text{H}_{17}\text{O}_3^+$, 269.1172; found, m/z 269.1176.



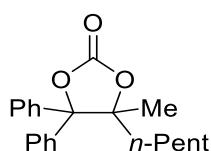
4-ethyl-4-methyl-5,5-diphenyl-1,3-dioxolan-2-one (**1b**)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 10:1 as an eluent; White solid (519.5 mg, 92%); mp 67-68 °C; ^1H NMR (400 MHz, CDCl_3): δ = 0.97 (t, J = 7.2 Hz, 3H), 1.36 (s, 3H), 1.66 (q, J = 7.2 Hz, 2H), 7.33-7.44 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3): δ = 8.1, 22.0, 31.1, 91.1, 92.7, 126.5, 126.6, 128.3 (\times 2C), 128.4, 128.4, 137.5, 138.0, 154.0; IR (KBr): 2987, 1801, 1450, 1255, 1026, 779, 762, 709, 669 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{18}\text{H}_{19}\text{O}_3^+$, 283.1329; found, m/z 283.1331.



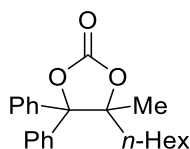
4-isobutyl-4-methyl-5,5-diphenyl-1,3-dioxolan-2-one (1c)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 20:1 as an eluent; White Solid (478.0 mg, 77%); mp 102 °C; ¹H NMR (400 MHz, CDCl₃): δ = 0.91 (d, *J* = 6.4 Hz, 6H), 1.38 (m, 4H), 1.58-1.63 (m, 1H), 1.85-1.95 (m, 1H), 7.31-7.45 (m, 10H); ¹³C NMR (100 MHz, CDCl₃): δ = 23.1, 23.3, 24.5, 25.0, 46.0, 91.1, 93.0, 126.5, 126.9, 128.4, 128.4, 128.4, 128.5, 137.6, 137.7, 154.1; IR (KBr): 2992, 2962, 2910, 1797, 1450, 1253, 1224, 1045, 1034, 756, 704 cm⁻¹; HRMS (ESI): [M+H]⁺ calcd for C₂₀H₂₃O₃⁺, 311.1642; found, m/z 311.1639.



4-methyl-4-pentyl-5,5-diphenyl-1,3-dioxolan-2-one (1d)

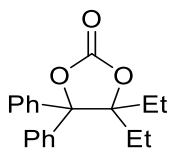
Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 10:1 as an eluent; White solid (524.8 mg, 81%); mp 93 °C; ¹H NMR (400 MHz, CDCl₃): δ = 0.81 (t, *J* = 7.2 Hz, 3H), 1.12-1.24 (m, 4H), 1.36-1.67 (m, 7H), 7.32-7.43 (m, 10H); ¹³C NMR (100 MHz, CDCl₃): δ = 13.9, 22.4, 22.8, 23.3, 31.9, 38.1, 91.0, 92.8, 126.56, 126.59, 128.36, 128.38 (× 2C), 128.4, 137.5, 138.0, 154.0; IR (KBr): 2953, 1798, 1452, 1384, 1261, 1135, 1050, 766, 704 cm⁻¹; HRMS (ESI): [M+H]⁺ calcd for C₂₁H₂₅O₃⁺, 325.1798; found, m/z 325.1797.



4-hexyl-4-methyl-5,5-diphenyl-1,3-dioxolan-2-one (1e)

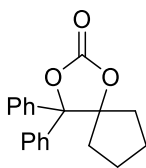
Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 20:1 as an eluent; Colorless oil (609.2 mg, 90%); ¹H NMR (400 MHz, CDCl₃): δ = 0.83 (t, *J* = 7.2 Hz, 3H), 1.14-1.22 (m, 6H), 1.36-1.44 (m, 5H), 1.51-1.67 (m, 2H), 7.32-7.43 (m, 10H); ¹³C NMR (100 MHz, CDCl₃): δ = 14.0, 22.4, 22.8, 23.6, 29.4, 31.5, 38.2, 91.0, 92.8, 126.6, 126.6, 128.3, 128.4, 128.4, 128.4, 137.6, 138.0, 154.0; IR (neat): 3062, 2931, 2857,

1804, 1450, 1386, 1251, 1028, 761, 706 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{22}\text{H}_{26}\text{O}_3\text{Na}^+$, 361.1774; found, m/z 361.1779.



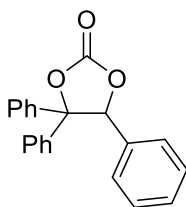
4,4-diethyl-5,5-diphenyl-1,3-dioxolan-2-one (1f)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 10:1 as an eluent; White solid (515.7 mg, 87%); mp 64-66 °C; ^1H NMR (400 MHz, CDCl_3): δ = 0.86 (t, J = 7.2 Hz, 6H), 1.67-1.86 (m, 4H), 7.33-7.42 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3): δ = 8.2, 29.2, 92.9, 93.0, 126.6, 128.3 (\times 2C), 138.1, 154.3; IR (KBr): 2983, 2945, 1794, 1450, 1248, 1055, 1033, 780, 757, 709 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{19}\text{H}_{21}\text{O}_3^+$, 297.1485; found, m/z 297.1484.



4,4-diphenyl-1,3-dioxaspiro[4.4]nonan-2-one (1g)

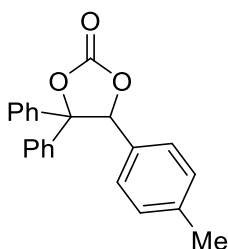
Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 20:1 as an eluent; White solid (518.1 mg, 88%); mp 77-78 °C; ^1H NMR (400 MHz, CDCl_3): δ = 1.65-1.82 (m, 8H), 7.34-7.43 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3): δ = 23.8, 36.4, 91.1, 100.1, 126.6, 128.5, 128.6, 138.4, 153.8; IR (KBr): 3063, 2958, 2878, 1793, 1494, 1450, 1340, 1246, 1029, 996, 764, 707 cm^{-1} ; HRMS (ESI): $[\text{M}]^+$ calcd for $\text{C}_{19}\text{H}_{18}\text{O}_3^+$, 294.1251; found, m/z 294.1252.



4,4,5-triphenyl-1,3-dioxolan-2-one (1h)

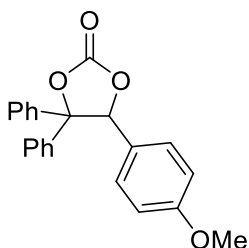
Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 10:1 as an eluent; White solid (398.6 mg, 63%); mp 97-98 °C; ^1H NMR (400 MHz, CDCl_3): δ = 6.33 (s, 1H), 6.95-7.21 (m, 10H), 7.34-7.48 (m, 3H), 7.61-7.63 (m, 2H); ^{13}C NMR (100

MHz, CDCl₃): δ = 86.6, 91.0, 126.3, 126.5, 127.3, 127.8, 128.0, 128.2, 128.9, 129.1, 129.2, 133.6, 137.2, 140.6, 153.9; IR (KBr): 3062, 3033, 1786, 1449, 1208, 1178, 1043, 990, 775, 696 cm⁻¹; HRMS (ESI): [M+H]⁺ calcd for C₂₁H₁₇O₃⁺, 317.1172; found, m/z 317.1179.



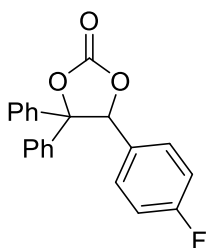
4,4-diphenyl-5-(*p*-tolyl)-1,3-dioxolan-2-one (1i)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 5:1 as an eluent; White solid (422.9 mg, 64%); mp 126-127 °C; ¹H NMR (400 MHz, CDCl₃): δ = 2.26 (s, 3H), 6.28 (s, 1H), 6.90-6.99 (m, 6H), 7.08-7.15 (m, 3H), 7.38-7.47 (m, 3H), 7.59-7.61 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 21.2, 86.9, 90.9, 126.2, 126.5, 127.5, 127.8, 128.0, 128.9, 128.9, 129.0, 130.4, 137.3, 139.2, 140.8, 154.0; IR (KBr): 3064, 3025, 1784, 1448, 1208, 1181, 1048, 994, 746, 694 cm⁻¹; HRMS (ESI): [M+H]⁺ calcd for C₂₂H₁₉O₃⁺, 331.1329; found, m/z 331.1329.



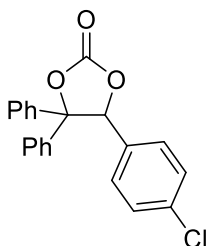
5-(4-methoxyphenyl)-4,4-diphenyl-1,3-dioxolan-2-one (1j)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 5:1 as an eluent; White solid (360.2 mg, 52%); mp 104 °C; ¹H NMR (400 MHz, CDCl₃): δ = 3.74 (s, 3H), 6.26 (s, 1H), 6.68-6.71 (m, 2H), 6.93-7.00 (m, 4H), 7.09-7.15 (m, 3H), 7.37-7.47 (m, 3H), 7.56-7.60 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 55.2, 86.9, 90.8, 113.6, 125.3, 126.1, 126.5, 127.9, 128.0, 128.7, 128.9, 129.0, 129.1, 137.4, 153.9, 160.2; IR (KBr): 3032, 1783, 1518, 1447, 1260, 1209, 1177, 1042, 697 cm⁻¹; HRMS (ESI): [M+H]⁺ calcd for C₂₂H₁₉O₄⁺, 347.1278; found, m/z 347.1286.



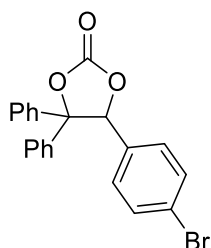
5-(4-fluorophenyl)-4,4-diphenyl-1,3-dioxolan-2-one (**1k**)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 10:1 as an eluent; White solid (635.3 mg, 95%); mp 82 °C; ¹H NMR (400 MHz, CDCl₃): δ = 6.32 (s, 1H), 6.84-6.89 (m, 2H), 6.94-6.97 (m, 4H), 6.99-7.14 (m, 3H), 7.39-7.48 (m, 3H), 7.57-7.60 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 85.9, 90.9, 115.4 (d, *J* = 22.1 Hz), 126.2, 126.4, 128.0, 128.2, 129.0, 129.2, 129.3 (d, *J* = 8.6 Hz), 129.5 (d, *J* = 2.9 Hz), 137.1, 140.4, 153.6, 162.9 (d, *J* = 251.0 Hz); ¹⁹F NMR (373 MHz, CDCl₃): δ = -112.8; IR (KBr): 3068, 3025, 1798, 1513, 1450, 1236, 1209, 1050, 765, 701 cm⁻¹; HRMS (ESI): [M+H]⁺ calcd for C₂₁H₁₆FO₃⁺, 335.1078; found, *m/z* 335.1080.



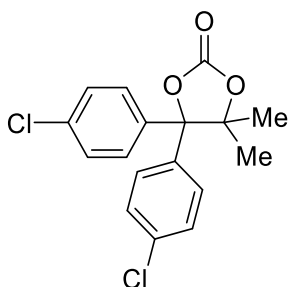
5-(4-chlorophenyl)-4,4-diphenyl-1,3-dioxolan-2-one (**1l**)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 5:1 as an eluent; White solid (609.5 mg, 87%); mp 144 °C; ¹H NMR (400 MHz, CDCl₃): δ = 6.23 (s, 1H), 6.95-6.98 (m, 4H), 7.12-7.17 (m, 5H), 7.42-7.48 (m, 3H), 7.57-7.60 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 85.8, 91.0, 126.3, 126.4, 128.0, 128.3, 128.5, 128.7, 129.0, 129.2, 132.1, 135.2, 136.9, 140.3, 153.6; IR (KBr): 3064, 1798, 1494, 1449, 1211, 1181, 1050, 808, 749, 692 cm⁻¹; HRMS (ESI): [M+H]⁺ calcd for C₂₁H₁₆ClO₃⁺, 351.0783; found, *m/z* 351.0782.



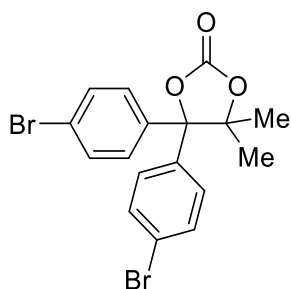
5-(4-bromophenyl)-4,4-diphenyl-1,3-dioxolan-2-one (1m)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 5:1 as an eluent; White solid (695.6 mg, 88%); mp 144 °C; ¹H NMR (400 MHz, CDCl₃): δ = 6.28 (s, 1H), 6.89-6.91 (m, 2H), 6.94-6.97 (m, 2H), 7.10-7.16 (m, 3H), 7.30-7.32 (m, 2H), 7.42-7.49 (m, 3H), 7.57-7.59 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 85.8, 90.9, 123.4, 126.3, 126.4, 128.1, 128.3, 128.9, 129.0, 129.2, 131.5, 132.6, 136.9, 140.2, 153.6; IR (KBr): 3060, 3026, 1798, 1489, 1449, 1210, 1181, 1074, 1010, 805, 748, 702 cm⁻¹; HRMS (ESI): [M+H]⁺ calcd for C₂₁H₁₆BrO₃⁺, 395.0278; found, m/z 395.0274.



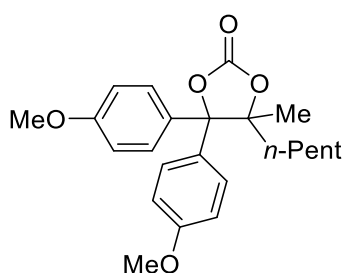
4,4-bis(4-chlorophenyl)-5,5-dimethyl-1,3-dioxolan-2-one (1n)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 5:1 as an eluent; Colorless oil (640.7 mg, 95%); ¹H NMR (400 MHz, CDCl₃): δ = 1.41 (s, 6H), 7.33 (d, *J* = 8.4 Hz, 4H), 7.38 (d, *J* = 8.8 Hz, 4H); ¹³C NMR (100 MHz, CDCl₃): δ = 25.5, 88.7, 91.3, 127.8, 128.8, 134.9, 135.9, 153.2; IR (neat): 2985, 1797, 1495, 1405, 1249, 1097, 1037, 820, 776, 536 cm⁻¹; HRMS (ESI): [M+H]⁺ calcd for C₁₇H₁₅Cl₂O₃⁺, 337.0393; found, m/z 337.0384.



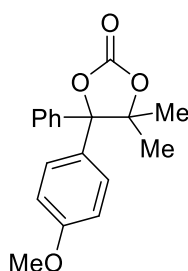
4,4-bis(4-bromophenyl)-5,5-dimethyl-1,3-dioxolan-2-one (**1o**)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 5:1 as an eluent; White solid (724.4 mg, 85%); mp 146-148 °C; ¹H NMR (400 MHz, CDCl₃): δ = 1.41 (s, 6H), 7.26 (d, *J* = 8.8 Hz, 4H), 7.54 (d, *J* = 9.2 Hz, 4H); ¹³C NMR (100 MHz, CDCl₃): δ = 25.5, 88.6, 91.4, 123.1, 128.0, 131.8, 136.4, 153.1; IR (KBr): 2979, 1798, 1488, 1400, 1251, 1039, 1007, 826, 774, 533 cm⁻¹; HRMS (ESI): [M+H]⁺ calcd for C₁₇H₁₅Br₂O₃⁺, 429.9383; found, *m/z* 429.9391.



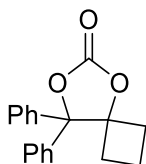
4,4-bis(4-methoxyphenyl)-5-methyl-5-pentyl-1,3-dioxolan-2-one (**1p**)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 5:1 as an eluent; Colorless oil (515.2 mg, 67%); ¹H NMR (400 MHz, CDCl₃): δ = 0.82 (t, *J* = 7.6 Hz, 3H), 1.12-1.25 (m, 4H), 1.34 (s, 3H), 1.39-1.42 (m, 2H), 1.54-1.58 (m, 2H), 6.87-6.90 (m, 4H), 7.26-7.31 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ = 13.9, 22.4, 22.6, 23.3, 31.9, 37.9, 55.2, 91.1, 92.8, 113.6 (× 2C), 127.8, 127.9, 129.9, 130.4, 154.2, 159.3 (× 2C); IR (neat): 2956, 1800, 1610, 1514, 1256, 1181, 1033, 829, 774, 582 cm⁻¹; HRMS (ESI): [M+H]⁺ calcd for C₂₃H₂₉O₅⁺, 385.2010; found, *m/z* 385.2009.



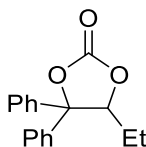
4-(4-methoxyphenyl)-5,5-dimethyl-4-phenyl-1,3-dioxolan-2-one (**1q**)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 5:1 as an eluent; Colorless oil (533.2 mg, 89%); ^1H NMR (400 MHz, CDCl_3): δ = 1.38 (s, 3H), 1.43 (s, 3H), 3.80 (s, 3H), 7.89 (d, J = 9.2 Hz, 2H), 7.30-7.40 (m, 7H); ^{13}C NMR (100 MHz, CDCl_3): δ = 25.3, 25.8, 55.3, 89.0, 92.2, 113.7, 126.3, 127.9, 128.4 (\times 2C), 130.0, 137.9, 153.9, 159.4; IR (neat): 2983, 1781, 1611, 1513, 1304, 1176, 1031, 758, 586 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{18}\text{H}_{19}\text{O}_4^+$, 299.1278; found, m/z 299.1280.



8,8-diphenyl-5,7-dioxaspiro[3.4]octan-6-one (**1r**)

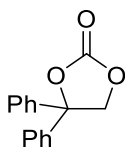
Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 10:1 as an eluent; White solid (521.4 mg, 93%); mp 99-100 $^\circ\text{C}$; ^1H NMR (400 MHz, CDCl_3): δ = 1.47-1.58 (m, 1H), 1.95-2.06 (m, 1H), 2.30-2.38 (m, 2H), 2.50-2.58 (m, 2H), 7.36-7.41 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3): δ = 13.4, 31.9, 91.3, 92.5, 127.1, 128.6, 129.0, 137.8, 153.2; IR (KBr): 3055, 3003, 2953, 1794, 1448, 1292, 1216, 1116, 1012, 756, 702 cm^{-1} ; HRMS (ESI): $[\text{M}]^+$ calcd for $\text{C}_{18}\text{H}_{16}\text{O}_3^+$, 280.1094; found, m/z 280.1103.



5-ethyl-4,4-diphenyl-1,3-dioxolan-2-one (**1s**)

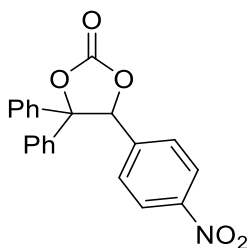
Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 5:1 as an eluent; White solid (413.2 mg, 77%); mp 86-87 $^\circ\text{C}$; ^1H NMR (400 MHz, CDCl_3): δ = 1.04 (t, J = 7.2 Hz, 3H), 1.21-1.33 (m, 1H), 1.39-1.49 (m, 1H), 5.18 (dd, J = 2.8, 11.2 Hz, 1H), 7.19-7.23 (m, 2H), 7.30-7.44 (m, 8H); ^{13}C NMR (100 MHz, CDCl_3): δ = 10.5, 25.9, 86.2, 89.5, 126.0, 126.2, 128.4, 128.5, 128.8, 129.0, 137.9, 140.4, 153.9; IR (KBr): 2974,

2934, 1791, 1447, 1224, 1032, 1017, 799, 763, 702 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{17}\text{H}_{17}\text{O}_3^+$, 269.1172; found, m/z 269.1178.



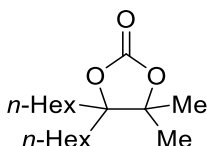
4,4-diphenyl-1,3-dioxolan-2-one (1t)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 5:1 as an eluent; White solid (395.7 mg, 82%); mp 109-110 $^{\circ}\text{C}$; ^1H NMR (400 MHz, CDCl_3): δ = 4.69 (s, 2H), 7.36-7.40 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3): δ = 75.8, 87.2, 125.5, 128.9 (\times 2C), 140.0, 154.0; IR (KBr): 3029, 2933, 1801, 1488, 1374, 1220, 1058, 980, 878, 757, 696 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{15}\text{H}_{13}\text{O}_3^+$, 241.0859; found, m/z 241.0856.



5-(4-nitrophenyl)-4,4-diphenyl-1,3-dioxolan-2-one (1u)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 5:1 as an eluent; White solid (361.4 mg, 50%); mp 156-157 $^{\circ}\text{C}$; ^1H NMR (400 MHz, CDCl_3): δ = 6.44 (s, 1H), 6.93-6.95 (m, 2H), 7.08-7.14 (m, 3H), 7.22-7.26 (m, 2H), 7.46-7.51 (m, 3H), 7.52-7.62 (m, 2H), 8.02-8.04 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 84.7, 91.2, 123.3, 126.4, 126.4, 128.0, 128.2, 128.7, 129.2, 129.6, 136.5, 139.6, 140.9, 148.0, 153.2; IR (KBr): 3114, 3086, 1789, 1521, 1350, 1207, 1177, 1048, 761, 699 cm^{-1} ; HRMS (ESI): $[\text{M}]^+$ calcd for $\text{C}_{21}\text{H}_{15}\text{O}_5\text{N}^+$, 361.0945; found, m/z 361.0951.



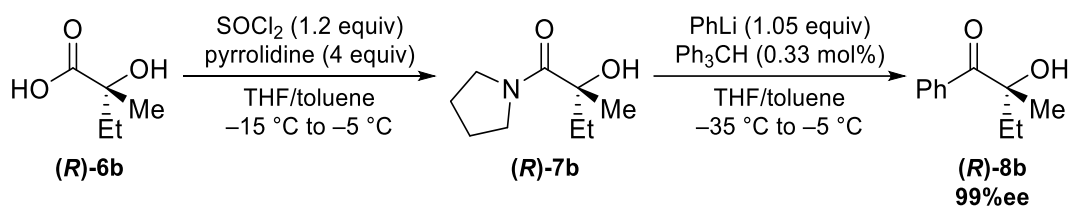
4,4-dihexyl-5,5-dimethyl-1,3-dioxolan-2-one (1v)

Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 10:1 as an

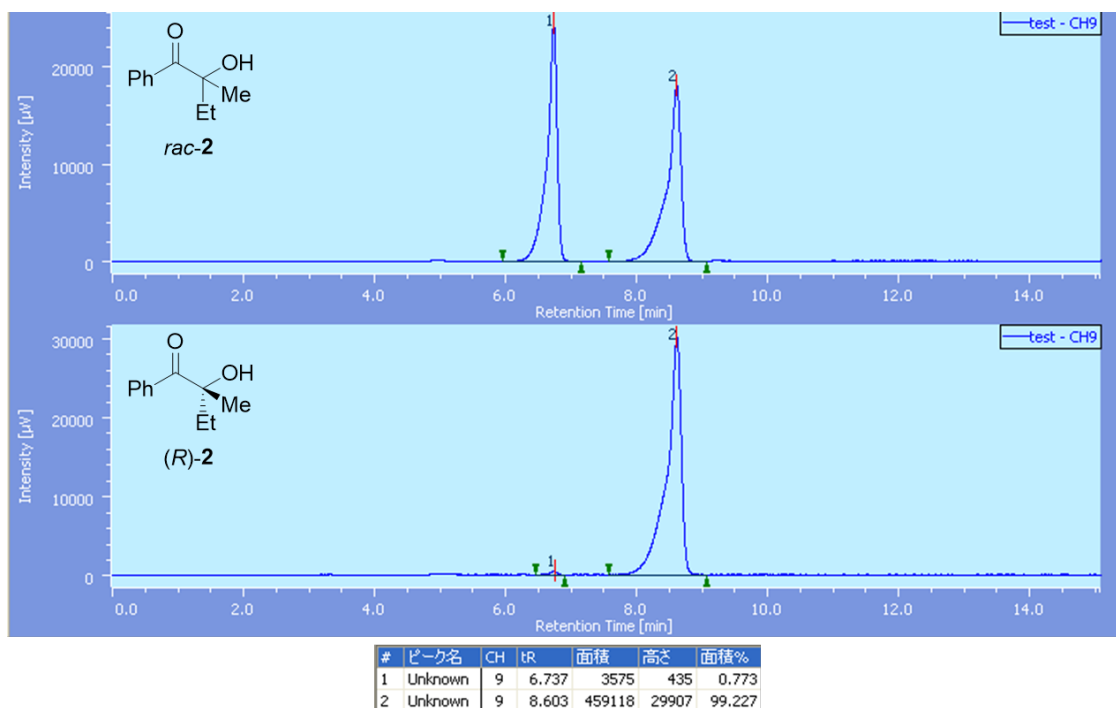
eluent; Colorless oil (551.8 mg, 97%); ^1H NMR (400 MHz, CDCl_3): δ = 0.90 (t, J = 6.8 Hz, 6H), 1.18-1.43 (m, 22H), 1.64-1.80 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.0, 22.5, 23.0, 23.4, 29.6, 31.5, 31.8, 86.4, 89.7, 154.0; IR (neat): 2930, 2858, 1798, 1466, 1378, 1271, 1109, 1025, 779 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{17}\text{H}_{33}\text{O}_3^+$, 285.2424; found, m/z 285.2419.

3.2.2 Preparation of Cyclic Carbonate ((*R*)-1b)

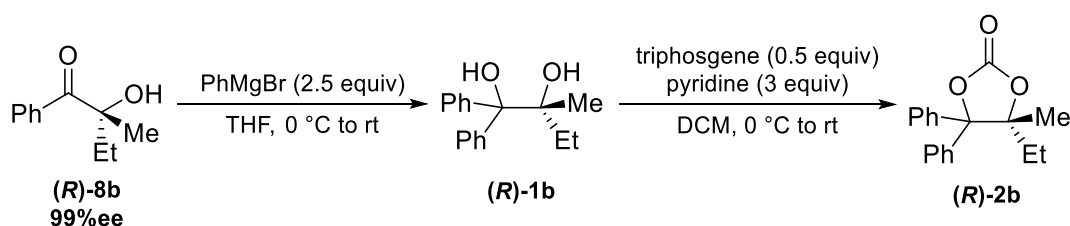
Synthesis of (*R*)-2-hydroxy-2-methyl-1-phenylbutan-1-one ((*R*)-8b)



To afford (*R*)-6b, the acid (\pm)-6b (6.00 g, 50.8 mmol) was neutralized with brucine (20.0 g, 50.8 mmol) in acetone (80 mL)¹¹, and the two brucine salts were recrystallized from methanol. Furthermore, the less-soluble salt was recrystallized from methanol five times. The diastereomeric pure salt (4.0 g, 7.8 mmol) was acidified with hydrochloric acid solution (6 M, 40 mL) and extracted nine times with diethyl ether. The ether extracts were washed with saturated sodium chloride solution and dried over sodium sulfite. The solvent was removed *in vacuo* and the white solid (700 mg, 5.93 mmol) was used directly in the following step. Hydroxyketone (*R*)-8b was prepared from 5.00 mmol of (*R*)-6b according to the literature.¹² This afforded 597 mg (67%; 2 steps) of (*R*)-8b as a colorless oil, 99% ee (determined by chiral HPLC: column, Daicel CHIRALPAK[®] IF; eluent, *n*-hexane /2-propanol = 90/10 (v/v); temperature, 20 $^\circ\text{C}$; flow rate, 1.0 mL/min; retention time, 6.7 min (minor) and 8.6 min (major)). $[\alpha]_{\text{D}}^{21} -39.0^\circ$ (c 0.132, CCl_4 , 99%ee); [lit¹³. $[\alpha]_{\text{D}}^{27} -34.7^\circ$ (c 0.150, CCl_4)].



Preparation of cyclic carbonate ((*R*)-2b)

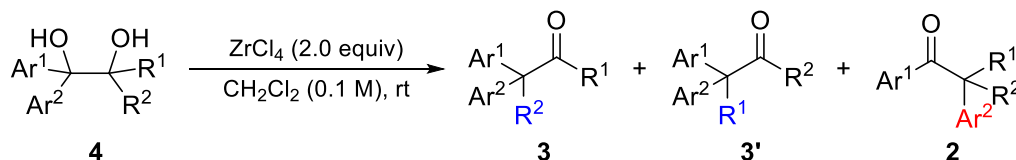


Step 1: Under N₂ atmosphere, (*R*)-2-hydroxy-2-methyl-1-phenylbutan-1-one ((*R*)-8b) (446 mg, 2.50 mmol) was dissolved into THF (3.0 mL). After cooling at 0 °C, a solution of pentylmagnesium bromide⁷ (6.25 mL, 6.25 mmol, 1 M solution in THF) was added dropwise, and the reaction mixture was warmed to room temperature. The reaction progress was continuously monitored by thin layer chromatography. Upon completion, the reaction was quenched by sat. NH₄Cl aq., and the mixture was extracted three times with Et₂O. The combined organic layer was washed with brine, dried over Na₂SO₄, and the solvent was removed under reduced pressure. The residue was purified by silica gel flash column chromatography using *n*-hexane /EtOAc = 7:1 as an eluent to afford (*R*)-1b as a colorless oil (600 mg, 94%).

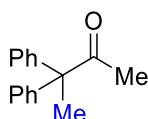
Step 2: Cyclic carbonate (*R*)-2b was prepared by **Procedure 3.2.1** using 1.5 mmol of diol (*R*)-1b. This afforded 380 mg (90%) of (*R*)-2b as a white solid.

4. General Procedure & Characterization Data

4.1 Procedure for Conventional Pinacol Rearrangement

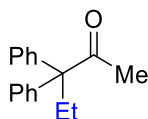


The 1,2-diol **4** (0.1 mmol) was dissolved in dehydrated CH₂Cl₂ (1.0 mL) and ZrCl₄ (46.6 mg, 0.2 mmol) was added. The reaction mixture was stirred at room temperature under N₂, and the reaction progress was monitored by thin layer chromatography. Upon completion, the reaction was quenched by sat. NaHCO₃ aq., and extracted three times with CH₂Cl₂. The combined organic layer was dried over Na₂SO₄, and the solvent was removed under reduced pressure. The residue was purified by preparative thin layer chromatography (*n*-Hexane : EtOAc = 5 : 1) to give ketones (**3** and/or **2**).



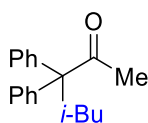
3,3-diphenylbutan-2-one (**3a**)¹⁴

Colorless oil (lit.⁴⁸, White solid) (22.2 mg, 99%); (lit.¹⁴, 41°C); ¹H NMR (400 MHz, CDCl₃): δ = 1.87 (s, 3H), 2.11 (s, 3H), 7.18-7.21 (m, 4H), 7.25-7.35 (m, 6H); ¹³C NMR (100 MHz, CDCl₃): δ = 26.4, 27.6, 62.3, 126.9, 128.3, 128.3, 143.5, 209.2; IR (neat): 3058, 2985, 1708, 1494, 1445, 1352, 1195, 1028, 762, 700, 577 cm⁻¹; HRMS (ESI): [M+H]⁺ calcd for C₁₆H₁₇O⁺, 225.1274; found, m/z 225.1277.



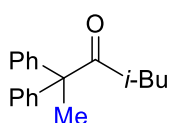
3,3-diphenylpentan-2-one (**3b**)

Colorless oil (quant.); ¹H NMR (400 MHz, CDCl₃): δ = 0.69 (t, *J* = 7.6 Hz, 3H), 2.03 (s, 3H), 2.35 (q, *J* = 7.6 Hz, 2H), 7.25-7.35 (m, 10H); ¹³C NMR (100 MHz, CDCl₃): δ = 9.4, 27.2, 30.0, 66.7, 126.8, 128.1, 129.4, 141.2, 208.2; IR (neat): 3058, 2974, 2937, 1705, 1599, 1494, 1448, 1352, 1188, 756, 702, 570 cm⁻¹; HRMS (ESI): [M]⁺ calcd for C₁₇H₁₈O⁺, 238.1352; found, m/z 238.1360.



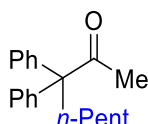
5-methyl-3,3-diphenylhexan-2-one (3c)

Colorless oil (14.5 mg, 54%); ^1H NMR (400 MHz, CDCl_3): δ = 0.63 (d, J = 6.8 Hz, 6H), 1.35 (m, 1H), 2.02 (s, 3H), 2.26 (d, J = 6.8 Hz, 2H), 7.24-7.33 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3): δ = 24.3, 24.9, 27.2, 45.9, 66.5, 126.8, 128.1, 129.5, 141.8, 208.1; IR (neat): 2955, 2868, 1706, 1494, 1444, 1352, 1187, 1159, 762, 701 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{19}\text{H}_{23}\text{O}^+$, 267.1743; found, m/z 267.1740.



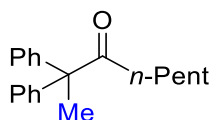
5-methyl-2,2-diphenylhexane-3-one (3c')

Colorless oil (9.3 mg, 35%); ^1H NMR (400 MHz, CDCl_3): δ = 0.77 (d, J = 6.8 Hz, 6H), 1.87 (s, 3H), 2.05 (m, 1H), 2.30 (d, J = 6.8 Hz, 2H), 7.17-7.20 (m, 4H), 7.25-7.35 (m, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ = 22.4, 24.3, 26.4, 47.9, 62.1, 126.8, 128.2, 128.5, 143.6, 210.4; IR (KBr): 2956, 2870, 1708, 1494, 1444, 1153, 1028, 1011, 761, 728, 700 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{19}\text{H}_{23}\text{O}^+$, 267.1743; found, m/z 267.1732.



3,3-diphenyloctan-2-one (3d)

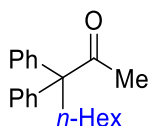
Colorless oil (23.3 mg, 83%); ^1H NMR (400 MHz, CDCl_3): δ = 0.79 (t, J = 6.8 Hz, 3H), 0.94-1.02 (m, 2H), 1.19-1.27 (m, 4H), 2.03 (s, 3H), 2.25-2.30 (m, 2H), 7.24-7.35 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.0, 22.4, 24.5, 27.2, 32.4, 37.3, 66.4, 126.8, 128.1, 129.3, 141.6, 208.3; IR (neat): 2954, 1707, 1495, 1352, 1188, 702 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{20}\text{H}_{25}\text{O}^+$, 281.1900; found, m/z 281.1901.



2,2-diphenyloctan-3-one (3d')

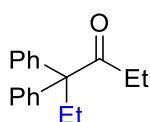
Colorless oil (3.1 mg, 11%); ^1H NMR (400 MHz, CDCl_3): δ = 0.81 (t, J = 7.6 Hz, 3H), 1.09-1.21 (m, 4H), 1.46 (quin, J = 7.6 Hz, 2H), 1.86 (s, 3H), 2.38 (t, J = 7.6 Hz, 2H),

7.17-7.35 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 13.9, 22.4, 24.2, 26.4, 31.2, 39.5, 62.2, 126.8, 128.3, 128.4, 143.8, 211.5$; IR (neat): 2931, 1709, 1494, 1445, 1028, 762, 700 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{20}\text{H}_{25}\text{O}^+$, 281.1900; found, m/z 281.1905.



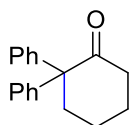
3,3-diphenylnonan-2-one (2e)

Colorless oil (26.8 mg, 91%); ^1H NMR (400 MHz, CDCl_3): $\delta = 0.82$ (t, $J = 6.8$ Hz, 3H), 0.94-1.01 (m, 2H), 1.18-1.27 (m, 6H), 2.03 (s, 3H), 2.26-2.30 (m, 2H), 7.25-7.35 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 14.0, 22.6, 24.9, 27.2, 29.9, 31.6, 37.3, 66.4, 126.8, 128.1, 129.3, 141.6, 208.3$; IR (neat): 2953, 2930, 2857, 1707, 1599, 1495, 1440, 1351, 1160, 752, 701, 580 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{21}\text{H}_{27}\text{O}^+$, 295.2056; found, m/z 295.2057.



4,4-diphenylhexan-3-one (3f)

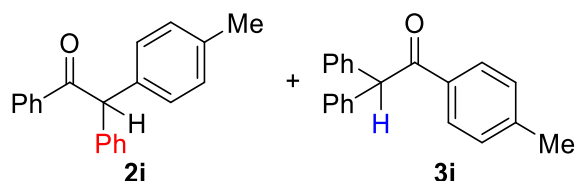
Colorless oil (quant.); ^1H NMR (400 MHz, CDCl_3): $\delta = 0.68$ (t, $J = 7.6$ Hz, 3H), 0.88 (t, $J = 7.2$ Hz, 3H), 2.34 (m, $J = 7.2, 7.6$ Hz, 4H), 7.24-7.35 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 9.1, 9.5, 30.0, 32.5, 66.6, 126.8, 128.1, 129.4, 141.5, 211.5$; IR (neat): 3058, 2973, 2937, 2878, 1707, 1494, 1449, 1128, 1033, 756, 701 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{18}\text{H}_{21}\text{O}^+$, 253.1587; found, m/z 253.1588.



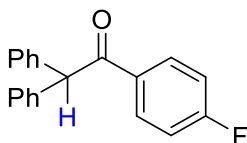
2,2-diphenylcyclohexan-1-one (3g)

White solid (23.3 mg, 80%); mp 100-101 $^{\circ}\text{C}$; ^1H NMR (400 MHz, CDCl_3): $\delta = 1.81$ -1.98 (m, 4H), 2.49-2.62 (m, 4H), 7.05-7.07 (m, 4H), 7.23-7.34 (m, 6H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 22.1, 27.8, 39.1, 40.7, 63.9, 126.8, 128.3, 128.5, 142.3, 211.3$; IR (KBr): 3052, 2940, 2866, 1706, 1495, 1445, 1118, 758, 748, 702, 571 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{18}\text{H}_{19}\text{O}^+$, 251.1430; found, m/z 251.1428.

2,2-diphenyl-1-(*p*-tolyl)ethan-1-one (**3i**)¹⁵

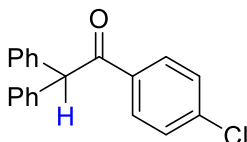


Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 5:1 as an eluent; White solid (22.5 mg, 95% combined yield, **2i/3i** = 79:21); ¹H NMR (400 MHz, CDCl₃, signals for **2i** where distinguishable are marked*, signals for **3i** where distinguishable are marked**, signal where the two overlap are marked***): δ = 2.30* (s, 3H), 2.35** (s, 3H), 6.00* (s, 1H), 6.02** (s, 1H), 7.11-7.32*** (m, 9H), 7.38* (t, *J* = 7.2 Hz, 2H), 7.48* (t, *J* = 7.2 Hz, 1H), 7.91** (d, *J* = 8.4 Hz, 2H), 7.99* (d, *J* = 8.8 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃, signals for **2i** where distinguishable are marked*, signals for **3i** where distinguishable are marked**): δ = 21.0*, 21.6**, 59.0*, 59.2**, 127.0***, 128.53*, 128.6***, 128.91*, 128.9*, 129.0**, 129.06*, 129.09**, 129.3**, 129.4*, 132.9*, 134.2**, 136.0*, 136.8*, 136.8*, 139.2**, 139.3*, 143.8**, 197.7**, 198.3*.



1-(4-fluorophenyl)-2,2-diphenylethan-1-one (**3k**)

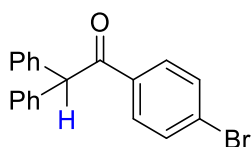
White solid (10.7 mg, 37%); mp 131-133 °C; ¹H NMR (400 MHz, CDCl₃): δ = 5.98 (s, 1H), 7.04-7.09 (m, 2H), 7.25-7.34 (m, 10H), 8.00-8.04 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 59.5, 115.7 (d, *J* = 22.0 Hz), 127.2, 128.8, 129.1, 131.6 (d, *J* = 8.6 Hz), 133.1 (d, *J* = 2.9 Hz), 138.8, 165.6 (d, *J* = 255.8 Hz), 196.6; ¹⁹F NMR (373 MHz, CDCl₃): δ = -106.3; IR (KBr): 3062, 3027, 1680, 1594, 1503, 1237, 1203, 1157, 821, 745, 697, 597 cm⁻¹; HRMS (ESI): [M+H]⁺ calcd for C₂₀H₁₆FO⁺, 291.1180; found, *m/z* 291.1178.



1-(4-chlorophenyl)-2,2-diphenylethan-1-one (**3l**)

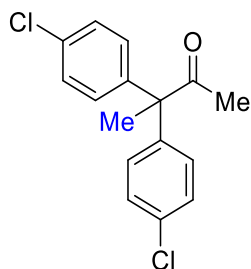
White solid (14.4 mg, 47%); mp 106-108 °C; ¹H NMR (400 MHz, CDCl₃): δ = 5.96 (s,

1H), 7.24-7.38 (m, 12H), 7.93 (d, $J=9.2$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 59.5, 127.3, 128.8, 128.9, 129.1, 130.4, 135.0, 138.7, 139.5, 197.0$; IR (KBr): 3025, 1678, 1588, 1400, 1208, 1092, 996, 700, 623 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{20}\text{H}_{16}\text{ClO}^+$, 307.0884; found, m/z 307.0884.



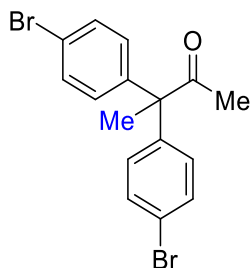
1-(4-bromophenyl)-2,2-diphenylethan-1-one (3m)¹⁵

White solid (14.5 mg, 41%); mp 105-106 °C; ^1H NMR (400 MHz, CDCl_3): $\delta = 5.95$ (s, 1H), 7.24-7.35 (m, 10H), 7.54 (d, $J = 8.8$ Hz, 2H), 7.85 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 59.5, 127.3, 128.2, 128.8, 129.1, 130.5, 131.9, 135.4, 138.6, 197.2$.



3,3-bis(4-chlorophenyl)butan-2-one (3n)

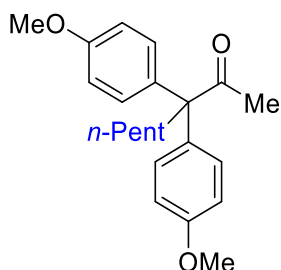
Colorless oil (23.2 mg, 79%); ^1H NMR (400 MHz, CDCl_3): $\delta = 1.85$ (s, 3H), 2.11 (s, 3H), 7.09 (d, $J = 8.8$ Hz, 4H), 7.31 (d, $J = 8.8$ Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 26.2, 27.3, 61.3, 128.6, 129.6, 133.1, 141.5, 208.1$; IR (neat): 2985, 1711, 1490, 1353, 1173, 1096, 1012, 824, 735, 557 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{16}\text{H}_{15}\text{Cl}_2\text{O}^+$, 293.0495; found, m/z 293.0490.



3,3-bis(4-bromophenyl)butan-2-one (3o)

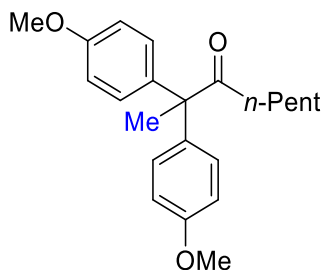
White solid (35.9 mg, 94%); mp 84-86 °C; ^1H NMR (400 MHz, CDCl_3): $\delta = 1.84$ (s, 3H), 2.11 (s, 3H), 7.03 (d, $J = 8.8$ Hz, 4H), 7.46 (d, $J = 8.8$ Hz, 4H); ^{13}C NMR (100 MHz,

CDCl₃): δ = 26.0, 27.3, 61.5, 121.3, 129.9, 131.6, 142.0, 207.9; IR (KBr): 2968, 1712, 1487, 1353, 1192, 1079, 1007, 821, 554 cm⁻¹; HRMS (ESI): [M+H]⁺ calcd for C₁₆H₁₅Br₂O⁺, 380.9484; found, m/z 380.9487.



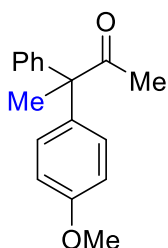
3,3-bis(4-methoxyphenyl)octan-2-one (3p)

Colorless oil (22.5 mg, 66%); ¹H NMR (400 MHz, CDCl₃): δ = 0.80 (t, *J* = 7.2 Hz, 3H), 0.94-1.24 (m, 2H), 1.21-1.24 (m, 4H), 2.01 (s, 3H), 2.17-2.23 (m, 2H), 3.81 (s, 6H), 6.86 (d, *J* = 9.2 Hz, 4H), 7.16 (d, *J* = 9.2 Hz, 4H); ¹³C NMR (100 MHz, CDCl₃): δ = 14.1, 22.5, 24.6, 27.0, 32.4, 37.5, 55.2, 65.0, 113.5, 130.4, 133.7, 158.2, 208.7; IR (neat): 2954, 1704, 1608, 1509, 1251, 1182, 1036, 827, 569 cm⁻¹; HRMS (ESI): [M+H]⁺ calcd for C₂₂H₂₉O₃⁺, 341.2111; found, m/z 341.2115.



2,2-bis(4-methoxyphenyl)octan-3-one (3p')

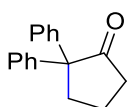
Colorless oil (3.1 mg, 9%); ¹H NMR (400 MHz, CDCl₃): δ = 0.81 (t, *J* = 7.6 Hz, 3H), 1.09-1.25 (m, 4H), 1.45 (quin, *J* = 7.6 Hz, 2H), 1.81 (s, 3H), 2.36 (t, *J* = 7.6 Hz, 2H), 3.81 (s, 3H), 6.85 (d, *J* = 9.2 Hz, 4H), 7.08 (d, *J* = 9.2 Hz, 4H); ¹³C NMR (100 MHz, CDCl₃): δ = 13.9, 22.4, 24.3, 26.5, 31.3, 39.2, 55.3, 60.8, 113.6, 129.4, 136.0, 158.2, 211.9; IR (neat): 2955, 1705, 1607, 1509, 1250, 1180, 1032, 828, 571 cm⁻¹; HRMS (ESI): [M+H]⁺ calcd for C₂₂H₂₉O₃⁺, 341.2111; found, m/z 341.2115.



3-(4-methoxyphenyl)-3-phenylbutan-2-one (**3q**)

Colorless oil (24.2 mg, 95%); $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 1.84 (s, 3H), 2.10 (s, 3H), 3.81 (s, 3H), 6.87 (d, J = 9.2 Hz, 2H), 7.11-7.19 (m, 4H), 7.24-7.35 (m, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 26.4, 27.5, 55.2, 61.6, 113.6, 126.8, 128.2, 128.3, 129.4, 135.2, 143.9, 158.3, 209.4; IR (neat): 2983, 1708, 1608, 1510, 1352, 1252, 1184, 1031, 703 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{17}\text{H}_{19}\text{O}_2^+$, 255.1380; found, m/z 255.1381.

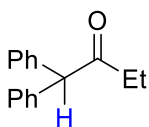
Similarly, Cyclic carbonate **2q** was treated with 2.0 equivalent of zirconium(IV) chloride in dichloromethane at room temperature to give 1.0 mg (4% yield) of **3q**.



2,2-diphenylcyclopentan-1-one (**3r**)¹⁶

White solid (20.8 mg, 88%); mp 68 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 1.94 (quin, J = 7.6 Hz, 2H), 2.45 (t, J = 8.0 Hz, 2H), 2.72 (t, J = 6.4 Hz, 2H), 7.20-7.31 (m, 10H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 18.8, 38.1, 38.2, 62.5, 126.7, 128.0, 128.4, 142.1, 217.8.

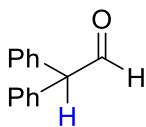
Similarly, cyclic carbonate **1r** was treated with 2.0 equivalent of zirconium(IV) chloride in dichloromethane at room temperature to give 22.9 mg (97% yield) of **3r**.



1,1-diphenylbutan-2-one (**3u**)

Colorless oil (20.2 mg, 90%); $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 1.06 (t, J = 7.6 Hz, 3H), 2.58 (q, J = 7.6 Hz, 2H), 5.14 (s, 1H), 7.22-7.34 (m, 10H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 8.1, 36.1, 63.9, 127.1, 128.6, 128.9, 138.6, 209.2; IR (neat): 3061, 3027, 2977, 2937, 1716, 1599, 1495, 1452, 1348, 1107, 1033, 750, 697, 619 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{16}\text{H}_{17}\text{O}^+$, 225.1274; found, m/z 225.1275.

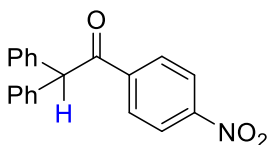
Similarly, cyclic carbonate **1u** was treated with 10 mol% of zirconium(IV) chloride in mesitylene at 150 °C to give 20.4 mg (91% yield) of **3u**.



2,2-diphenylacetaldehyde (**3t**)¹⁷

Colorless oil (11.4 mg, 58%); ¹H NMR (400 MHz, CDCl₃): δ = 4.88 (d, *J* = 2.0 Hz, 1H), 7.20-7.39 (m, 10H), 9.93 (d, *J* = 2.0 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 64.0, 127.6, 128.9, 129.1, 136.2, 198.5; HRMS (ESI): [M+H]⁺ calcd for C₁₄H₁₃O⁺, 197.0961; found, *m/z* 197.0965.

Similarly, cyclic carbonate **1t** was treated with 10 mol% of zirconium(IV) chloride in mesitylene at 150 °C to give 16.3 mg (83% yield) of **3t**.

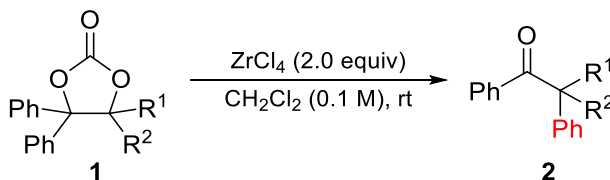


1-(4-nitrophenyl)-2,2-diphenylethan-1-one (**3u**)

Paleyellow solid (25.1 mg, 79%); mp 130-131 °C; ¹H NMR (400 MHz, CDCl₃): δ = 5.99 (s, 1H), 7.24-7.36 (m, 10H), 8.10-8.12 (m, 2H), 8.21-8.23 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 60.2, 123.8, 127.5, 128.9, 129.0, 129.9, 137.9, 141.2, 150.0, 196.7; IR (KBr): 3073, 3024, 1687, 1601, 1520, 1346, 1202, 835, 747, 703 cm⁻¹; HRMS (ESI): [M+H]⁺ calcd for C₂₀H₁₆O₃N⁺, 318.1125; found, *m/z* 318.1118.

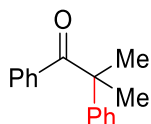
Similarly, cyclic carbonate **1u** was treated with 1.0 equivalent of zirconium(IV) chloride in mesitylene at 150 °C to give 23.8 mg (75% yield) of **3u**.

4.2 Procedure for Decarboxylative 1,2-Rearrangement



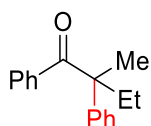
The cyclic carbonate **1** (0.1 mmol) was dissolved in dehydrated CH₂Cl₂ (1.0 mL) and ZrCl₄ (46.6 mg, 0.2 mmol) was added. The reaction mixture was stirred at room temperature under N₂, and the reaction progress was monitored by thin layer chromatography. Upon completion, the reaction was quenched by sat. NaHCO₃ aq., and extracted three times with CH₂Cl₂. The combined organic layer was dried over Na₂SO₄,

and the solvent was removed under reduced pressure. The residue was purified by preparative thin layer chromatography (*n*-Hexane : EtOAc = 5 : 1) to give ketone **2**.



2-methyl-1,2-diphenylpropan-1-one (**2a**)¹⁸

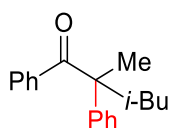
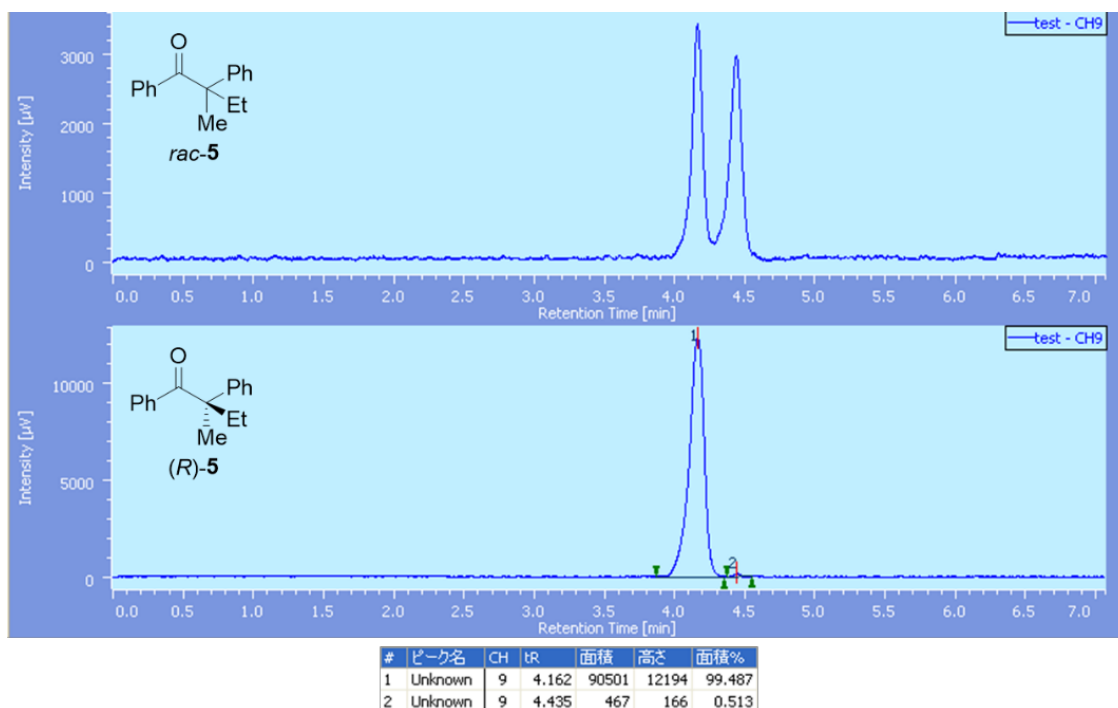
White solid (21.1 mg, 94%); mp 38 °C (lit.¹⁸, 41°C); ¹H NMR (400 MHz, CDCl₃): δ = 1.60 (s, 6H), 7.19-7.37 (m, 8H), 7.47-7.50 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 27.8, 51.4, 125.7, 126.7, 127.9, 129.0, 129.7, 131.6, 136.2, 145.2, 203.7.



2-methyl-1,2-diphenylbutan-1-one (**2b**)

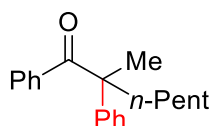
Colorless oil (19.1 mg, 80%); ¹H NMR (400 MHz, CDCl₃): δ = 0.75 (t, *J* = 7.6 Hz, 3H), 1.55 (s, 3H), 2.02-2.20 (m, 2H), 7.18-7.37 (m, 8H), 7.43-7.45 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 8.6, 23.7, 32.0, 54.9, 126.2, 126.7, 127.9, 128.8, 129.4, 131.5, 136.9, 144.3, 203.8; IR (neat): 3059, 2971, 2937, 1677, 1597, 1446, 1234, 963, 762, 701 cm⁻¹; HRMS (ESI): [M]⁺ calcd for C₁₇H₁₈O⁺, 238.1352; found, *m/z* 238.1345.

HPLC conditions: Daicel CHIRALPAK[®] IF column, *n*-hexane /2-propanol = 85/15 (v/v), 20 °C, flow rate = 1.0 mL/min, *t_R* = 4.16 min (major enantiomer), 4.44 (minor enantiomer). [*a*]_D²² -59.3° (*c* 0.150, C₆H₆, 99%ee), [cf. (**S**)-**2b**; lit¹⁹. [*a*]_D²⁰ +37° (*c* 1.0, C₆H₆)]



2,4-dimethyl-1,2-diphenylpentan-1-one (2c)

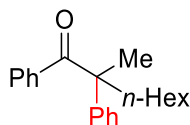
Colorless oil (26.1 mg, 98%); ^1H NMR (400 MHz, CDCl_3): δ = 0.73 (dd, J = 4.8, 6.8 Hz, 6H), 1.56 (m, 1H), 1.61 (s, 3H), 1.98-2.12 (m, 2H), 7.18-7.36 (m, 8H), 7.41-7.43 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 24.5, 24.7, 25.0, 48.0, 54.9, 126.3, 126.7, 127.9, 128.8, 129.4, 131.4, 137.1, 144.5, 204.1; IR (neat): 3059, 2955, 2868, 1676, 1597, 1445, 1238, 964, 761, 701 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{19}\text{H}_{22}\text{ONa}^+$, 289.1563; found, m/z 289.1563.



2-methyl-1,2-diphenylheptan-1-one (2d)

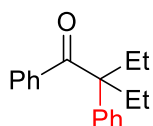
Colorless oil (26.6 mg, 95%); ^1H NMR (400 MHz, CDCl_3): δ = 0.78 (t, J = 7.2 Hz, 3H), 1.00-1.20 (m, 6H), 1.56 (s, 3H), 1.96-2.15 (m, 2H), 7.18-7.36 (m, 8H), 7.42-7.44 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 13.9, 22.3, 23.7, 24.5, 32.3, 39.1, 54.7, 126.2, 126.7, 127.9, 128.9, 129.4, 131.5, 136.9, 144.6, 203.8; IR (neat): 2932, 1677, 1597, 1446, 1376, 1240, 970, 701 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{20}\text{H}_{25}\text{O}^+$, 281.1900; found, m/z

281.1902.



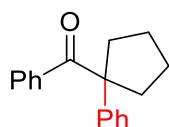
2-methyl-1,2-diphenyloctan-1-one (**2e**)

Colorless oil (29.1 mg, 99%); ^1H NMR (400 MHz, CDCl_3): δ = 0.80 (t, J = 6.4 Hz, 3H), 1.17 (m, 8H), 1.56 (s, 3H), 1.96-2.15 (m, 2H), 7.16-7.35 (m, 8H), 7.43-7.45 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 14.0, 22.5, 24.0, 24.5, 29.8, 31.4, 39.3, 54.6, 126.1, 126.7, 127.8, 128.8, 129.3, 131.4, 136.9, 144.6, 203.7; IR (neat): 3059, 2929, 2858, 1677, 1597, 1445, 1242, 964, 762, 701 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{21}\text{H}_{27}\text{O}^+$, 295.2056; found, m/z 295.2054.



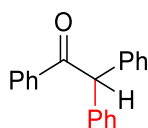
2-ethyl-1,2-diphenylbutan-1-one (**2f**)¹⁸

Colorless oil (22.7 mg, 90%); ^1H NMR (400 MHz, CDCl_3): δ = 0.66 (t, J = 7.6 Hz, 6H), 2.08-2.18 (m, 4H), 7.17-7.21 (m, 2H), 7.26-7.41 (m, 8H); ^{13}C NMR (100 MHz, CDCl_3): δ = 7.8, 26.3, 58.5, 126.8, 127.0, 127.9, 128.7, 129.3, 131.4, 137.4, 143.0, 204.1.



phenyl(1-phenylcyclopentyl)methanone (**2g**)

Colorless oil (21.0 mg, 84%); ^1H NMR (400 MHz, CDCl_3): δ = 1.66-1.78 (m, 4H), 2.06-2.11 (m, 2H), 2.49-2.54 (m, 2H), 7.19-7.38 (m, 8H), 7.61-7.63 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 24.6, 37.4, 63.3, 126.0, 126.5, 127.9, 128.9, 129.8, 131.7, 136.1, 144.5, 202.1; IR (neat): 3058, 2957, 2871, 1675, 1597, 1494, 1446, 1236, 1178, 1010, 757, 700 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{18}\text{H}_{19}\text{O}^+$, 251.1430; found, m/z 251.1424.

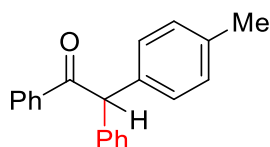


1,2,2-triphenylethan-1-one (**2h**)¹⁵

White solid (23.1 mg, 85%); mp 134-135 $^{\circ}\text{C}$ (lit.²⁰, 131-132 $^{\circ}\text{C}$); ^1H NMR (400 MHz,

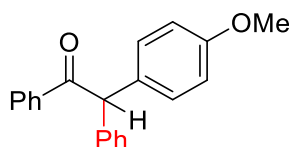
CDCl₃): δ = 6.04 (s, 1H), 7.23-7.50 (m, 13H), 7.99-8.01 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 59.4, 127.1, 128.6, 128.7, 128.9, 129.1, 133.0, 136.8, 139.0, 198.2.

Similarly, 1,2-diol **4h** was treated with 2.0 equivalent of zirconium(IV) chloride in dichloromethane at room temperature to give 27.0 mg (99% yield) of **2h**.



1,2-diphenyl-2-(*p*-tolyl)ethan-1-one (**2i**)

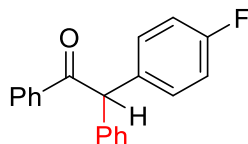
White solid (27.2 mg, 95%); mp 95-96 °C; ¹H NMR (400 MHz, CDCl₃): δ = 2.29 (s, 3H), 6.00 (s, 1H), 7.11-7.50 (m, 12H), 7.98-8.00 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 21.0, 59.0, 127.0, 128.5, 128.6, 128.9, 128.9, 129.1, 129.4, 132.9, 136.0, 136.8, 136.8, 139.3, 198.3; IR (KBr): 3058, 3027, 2916, 1681, 1447, 1204, 791, 755, 698, 684, 604 cm⁻¹; HRMS (ESI): [M+H]⁺ calcd for C₂₁H₁₉O⁺, 287.1430; found, m/z 287.1430.



2-(4-methoxyphenyl)-1,2-diphenylethan-1-one (**2j**)²¹

Colorless oil (25.1 mg, 83%); ¹H NMR (400 MHz, CDCl₃): δ = 3.76 (s, 3H), 5.99 (s, 1H), 6.84-6.87 (m, 2H), 7.18-7.52 (m, 10H), 7.98-8.01 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 55.2, 58.5, 114.1, 127.0, 128.6, 128.6, 128.9, 129.0, 130.1, 131.1, 132.9, 136.8, 139.4, 158.6, 198.4.

Similarly, 1,2-diol **4j** was treated with 2.0 equivalent of zirconium(IV) chloride in dichloromethane at room temperature to give 29.9 mg (99% yield) of **2j**.

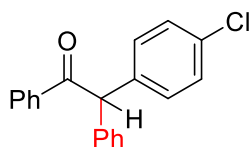


2-(4-fluorophenyl)-1,2-diphenylethan-1-one (**2k**)

White solid (25.3 mg, 87%); mp 106-107 °C; ¹H NMR (400 MHz, CDCl₃): δ = 6.02 (s, 1H), 6.98-7.00 (m, 2H), 7.02-7.53 (m, 10H), 7.98-8.00 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 58.5, 115.5 (d, *J* = 21.1 Hz), 127.3, 128.6, 128.9, 128.9, 128.9, 130.7 (d, *J* =

7.7 Hz), 133.3, 134.8 (d, $J = 2.8$ Hz), 136.5, 138.9, 161.9 (d, $J = 247.2$ Hz), 198.0; ^{19}F NMR (373 MHz, CDCl_3): $\delta = -116.9$; IR (KBr): 3057, 3029, 1682, 1597, 1507, 1448, 1218, 759, 740, 694 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{20}\text{H}_{15}\text{FNaO}^+$, 313.0999; found, m/z 313.1000.

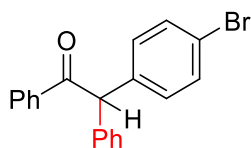
Similarly, 1,2-diol **4k** was treated with 2.0 equivalent of zirconium(IV) chloride in dichloromethane at room temperature to give 18.3 mg (63% yield) of **2k**.



2-(4-chlorophenyl)-1,2-diphenylethane-1-one (**2l**)

Colorless oil (29.1 mg, 95%); ^1H NMR (400 MHz, CDCl_3): $\delta = 6.00$ (s, 1H), 7.19 (d, $J = 8.4$ Hz, 2H), 7.24-7.32 (m, 7H), 7.39-7.42 (m, 2H), 7.50-7.51 (m, 1H), 7.98 (d, $J = 7.2$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 58.6, 127.4, 128.7, 128.8, 128.9, 128.9, 128.9, 130.5, 133.1, 133.2, 136.5, 137.6, 138.6, 197.8$; IR (neat): 3062, 1685, 1596, 1490, 1208, 1091, 1015, 798, 697 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{20}\text{H}_{16}\text{ClO}^+$, 307.0884; found, m/z 307.0889.

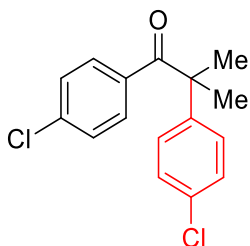
Similarly, 1,2-diol **4l** was treated with 2.0 equivalent of zirconium(IV) chloride in dichloromethane at room temperature to give 14.4 mg (47% yield) of **2l**.



2-(4-bromophenyl)-1,2-diphenylethane-1-one (**2m**)

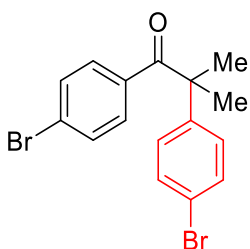
Colorless oil (33.7 mg, 96%); ^1H NMR (400 MHz, CDCl_3): $\delta = 5.99$ (s, 1H), 7.14 (d, $J = 8.8$ Hz, 2H), 7.24-7.53 (m, 10H), 7.98 (d, $J = 7.6$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 58.7, 121.3, 127.4, 128.7, 128.91, 128.93, 128.94, 130.9, 131.7, 133.2, 136.5, 138.2, 138.5, 197.7$; IR (neat): 3061, 3027, 1685, 1596, 1487, 1447, 1292, 1208, 1073, 1010, 795, 749, 697, 636 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{20}\text{H}_{16}\text{BrO}^+$, 351.0379; found, m/z 351.0374.

Similarly, 1,2-diol **4m** was treated with 2.0 equivalent of zirconium(IV) chloride in dichloromethane at room temperature to give 17.2 mg (49% yield) of **2m**.



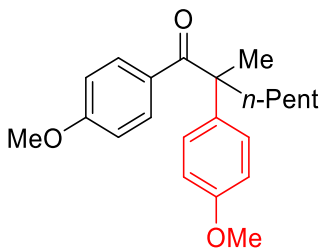
1,2-bis(4-chlorophenyl)-2-methylpropan-1-one (2n)

Colorless oil (28.7 mg, 98%); $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 1.58 (s, 6H), 7.21-7.23 (m, 4H), 7.33 (d, J = 8.8 Hz, 2H), 7.44 (d, J = 8.8 Hz, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 27.7, 51.0, 127.0, 128.4, 129.3, 131.2, 132.8, 133.9, 138.3, 143.6, 201.8; IR (neat): 2978, 1680, 1586, 1488, 1251, 1092, 976, 840, 757, 572 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{16}\text{H}_{15}\text{Cl}_2\text{O}^+$, 293.0495; found, m/z 293.0487.



1,2-bis(4-bromophenyl)-2-methylpropan-1-one (2o)

White solid (32.1 mg, 84%); mp 105-107 $^\circ\text{C}$; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 1.57 (s, 6H), 7.16 (d, J = 8.8 Hz, 2H), 7.35-7.40 (m, 4H), 7.47 (d, J = 8.8 Hz, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 27.7, 51.1, 120.9, 127.0, 127.4, 131.3, 131.3, 132.2, 134.3, 144.1, 201.9; IR (KBr): 2973, 1673, 1583, 1488, 1248, 978, 825, 752, 568 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{16}\text{H}_{15}\text{Br}_2\text{O}^+$, 380.9475; found, m/z 380.9475.



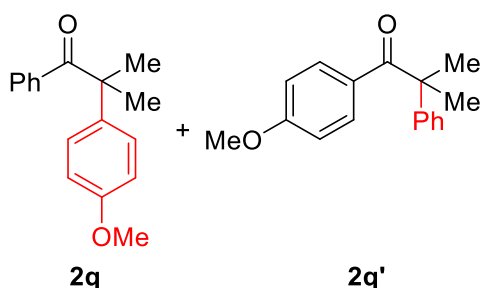
1,2-bis(4-methoxyphenyl)-2-methylheptan-1-one (2p)

Colorless oil (27.9 mg, 82%); $^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 0.79 (t, J = 7.2 Hz, 3H), 0.99-1.27 (m, 6H), 1.52 (s, 3H), 1.91-2.11 (m, 2H), 3.76 (s, 3H), 3.79 (s, 3H), 6.70 (d, J = 9.2 Hz, 2H), 6.87 (d, J = 9.2 Hz, 2H), 7.18 (d, J = 8.4 Hz, 2H), 7.51 (d, J = 9.2 Hz, 2H);

^{13}C NMR (100 MHz, CDCl_3): $\delta = 13.9, 22.4, 23.8, 25.0, 32.4, 39.6, 53.7, 55.1, 55.2, 113.0, 114.1, 127.1, 129.3, 131.9, 137.3, 158.1, 162.0, 202.3$; IR (neat): 2933, 1667, 1600, 1509, 1251, 1032, 832, 588 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{22}\text{H}_{29}\text{O}_3^+$, 341.2111; found, m/z 341.2109.

2-(4-methoxyphenyl)-2-methyl-1-phenylpropan-1-one (**2p**)²²

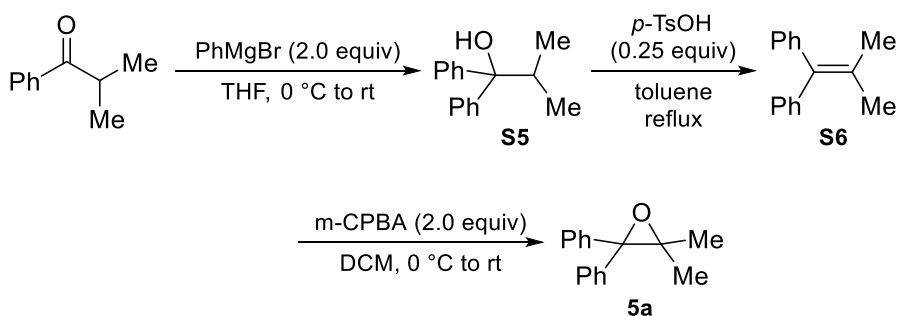
1-(4-methoxyphenyl)-2-methyl-2-phenylpropan-1-one (**2p'**)



Purified by silica-gel flash column chromatography using *n*-hexane /EtOAc = 5:1 as an eluent; Colorless oil (21.1 mg, 83% combined yield, **2q/2q'** = 58:42); ^1H NMR (400 MHz, CDCl_3 , signals for **2q** where distinguishable are marked*, signals for **2q'** where distinguishable are marked**, signal where the two overlap are marked***): $\delta = 1.58^*$ (s, 6H), 1.60^{**} (s, 6H), 3.76^{**} (s, 3H), 3.80^* (s, 3H), 6.70^{**} (d, $J = 9.2$ Hz, 2H), 6.89^* (d, $J = 9.2$ Hz, 2H), $7.23\text{-}7.36^{***}$ (m, 5H), 7.48^* (d, $J = 9.2$ Hz, 2H), 7.54^{**} (d, $J = 9.2$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3 , signals for **2q** where distinguishable are marked*, signals for **2q'** where distinguishable are marked**): $\delta = 27.9^{**}, 28.1^*, 50.7^*, 51.2^{**}, 55.2^{**}, 55.3^*, 113.1^{**}, 114.3^*, 125.6^{**}, 126.6^{**}, 126.8^*, 127.9^*, 128.4^{**}, 128.9^{**}, 129.6^*, 131.5^*, 132.3^{**}, 136.4^*, 137.2^*, 146.0^{**}, 158.3^*, 162.2^{**}, 202.0^{**}, 204.0^*$.

4.3 Procedure for House-Meinwald Rearrangement

4.3.1 Procedure for the Preparation of Oxirane (**5a**)

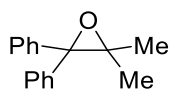


Step 1: Under N_2 atmosphere, 2-Hydroxy-2-methyl-1-phenylpropan-1-one (3.0 mL, 20 mmol) was dissolved into THF (20 mL). After cooling at 0 °C, a solution of

phenylmagnesium bromide (40 mL, 40 mmol, 1 M solution in THF) was added dropwise, and the reaction mixture was warmed to room temperature. The reaction progress was continuously monitored by thin layer chromatography. Upon completion, the reaction was quenched by sat. NH_4Cl aq., and the mixture was extracted several times with EtOAc. The combined organic layer was washed with brine, dried over Na_2SO_4 , and the solvent was removed under reduced pressure. The resulting compound **S5** was pure enough (yellow oil, 4.25 g, 94%), and so used in the next reaction without purification.

Step 2: Under N_2 atmosphere, alcohol **S5** (4.25 g, 18.8 mmol) was dissolved into toluene (20 mL). To the solution, *p*-TsOH (809.3 mg, 4.7 mmol) was added, and the reaction mixture was stirred under reflux. The reaction was monitored by thin layer chromatography until consumption of starting material was observed. The reaction mixture was cooled to room temperature and quenched by sat. NaHCO_3 aq., and the mixture was extracted three times with Et_2O . The combined organic layer was washed with brine, dried over Na_2SO_4 , and the solvent was removed under reduced pressure. The resulting compound **S6** was pure enough (yellow oil, 2.54 g, 65%), and so used in the next reaction without purification.

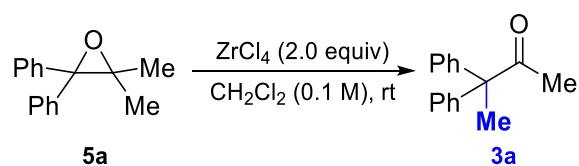
Step 2: Alkene **S6** (2.54 g, 12.2 mmol) was dissolved in dehydrate CH_2Cl_2 (10.0 mL), and the solution was cooled to 0 °C. To the solution, *m*-CPBA (70% purity; 6.0 g, 24 mmol) was added in batches. The reaction mixture was then allowed to stir at room temperature, and the reaction progress was monitored by thin layer chromatography. Upon completion, the reaction was quenched by sat. $\text{Na}_2\text{S}_2\text{O}_3$ aq., and extracted three times with CH_2Cl_2 . The combined organic layer was dried over Na_2SO_4 , and the solvent was removed under reduced pressure. The residue was purified by column chromatography to obtain epoxide **5a**.



2,2-dimethyl-3,3-diphenyloxirane (**5a**)

White solid (2.2 g, 49% (three steps)); mp 62-63 °C; ^1H NMR (400 MHz, CDCl_3): δ = 1.21 (s, 6H), 7.22 (t, J = 7.6 Hz, 2H), 7.31 (t, J = 7.6 Hz, 4H), 7.46 (d, J = 7.6 Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ = 21.8, 65.2, 71.2, 127.0, 127.1, 128.1, 140.4; IR (KBr): 2957, 1447, 1198, 1076, 925, 765, 703 cm^{-1} ; HRMS (ESI): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{16}\text{H}_{17}\text{O}^+$, 225.1274; found, m/z 225.1278.

4.3.2 House-Meinwald Rearrangement of Oxirane (**5a**)

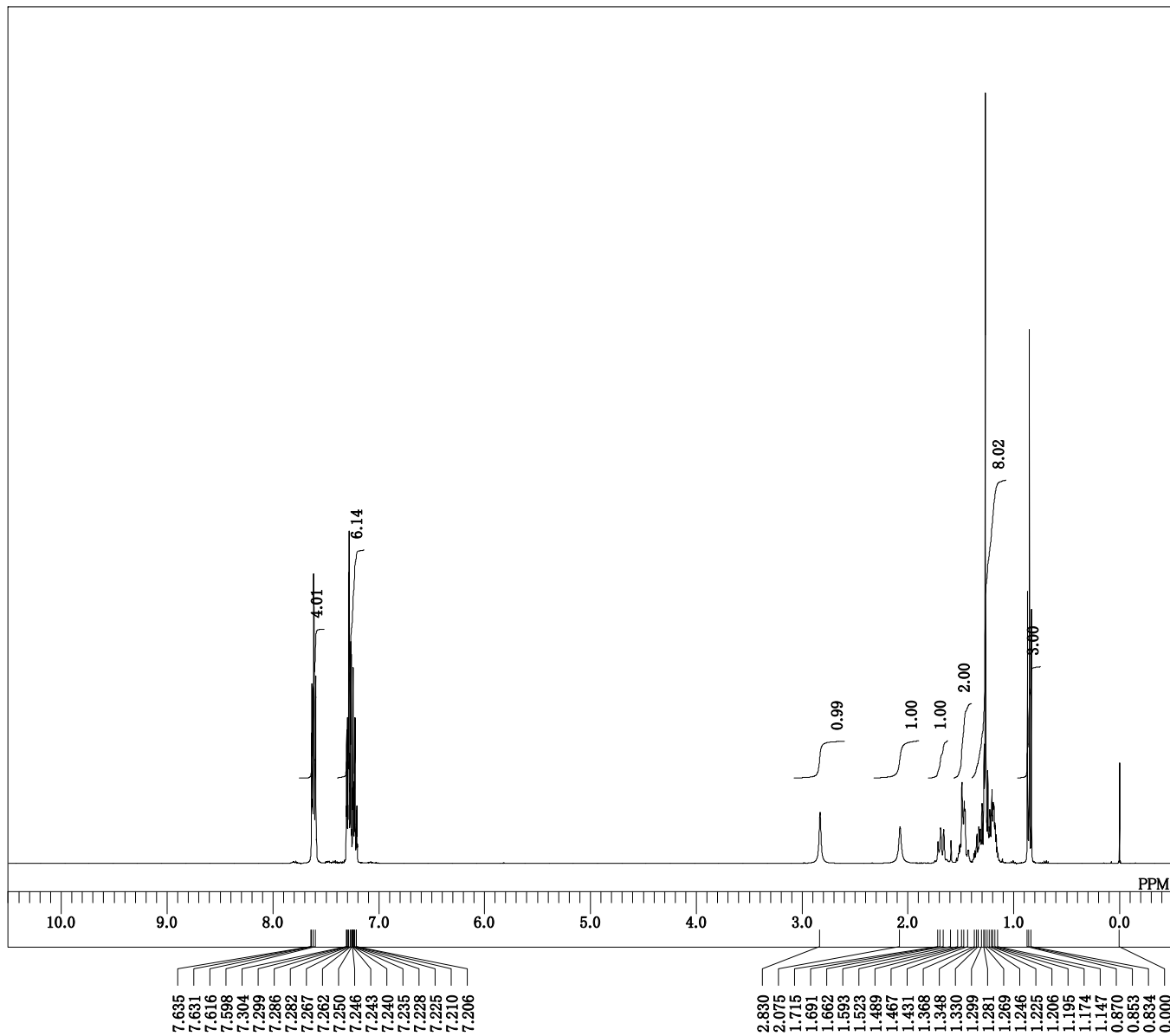


The epoxide **5a** (0.1 mmol) was dissolved in dehydrated CH₂Cl₂ (1.0 mL) and ZrCl₄ (46.6 mg, 0.2 mmol) was added. The reaction mixture was stirred at room temperature under N₂, and the reaction progress was monitored by thin layer chromatography. Upon completion, the reaction was quenched by sat. NaHCO₃ aq., and extracted three times with CH₂Cl₂. The combined organic layer was dried over Na₂SO₄, and the solvent was removed under reduced pressure. The residue was purified by preparative thin layer chromatography (*n*-Hexane : EtOAc = 5 : 1) to afford 3,3-diphenylbutan-2-one **3a** (19.1 mg, 85%) as a colorless oil.

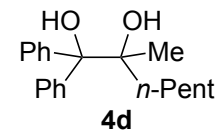
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- 6) LiCl which was dried under vacuum at 150 °C for 5h was used.
- 7) Grignard reagents were prepared according to the following method. A 100 mL three-neckround bottom flask equipped with a reflux condenser and dropping funnel was charged with magnesium turnings (1.46 g, 60.0 mmol). The system was evacuated, flame dried, cooled, and backfilled with Ar. The magnesium turnings were stirred vigorously at room temperature for 30 min. Then, the turnings were suspended in THF (10 mL) and a small crystal of I₂ was added. The dropping funnel was charged with solution of alkyl bromide (50.0 mmol) in THF (40 mL), and the solution was added to the magnesium turnings dropwise over 30 min. The reaction mixture was maintained at room temperature for additional 1 h. The reaction mixture was transferred *via* cannula into a flame dried Schlenk flask.
- 8) IBX was prepared by the following procedure.
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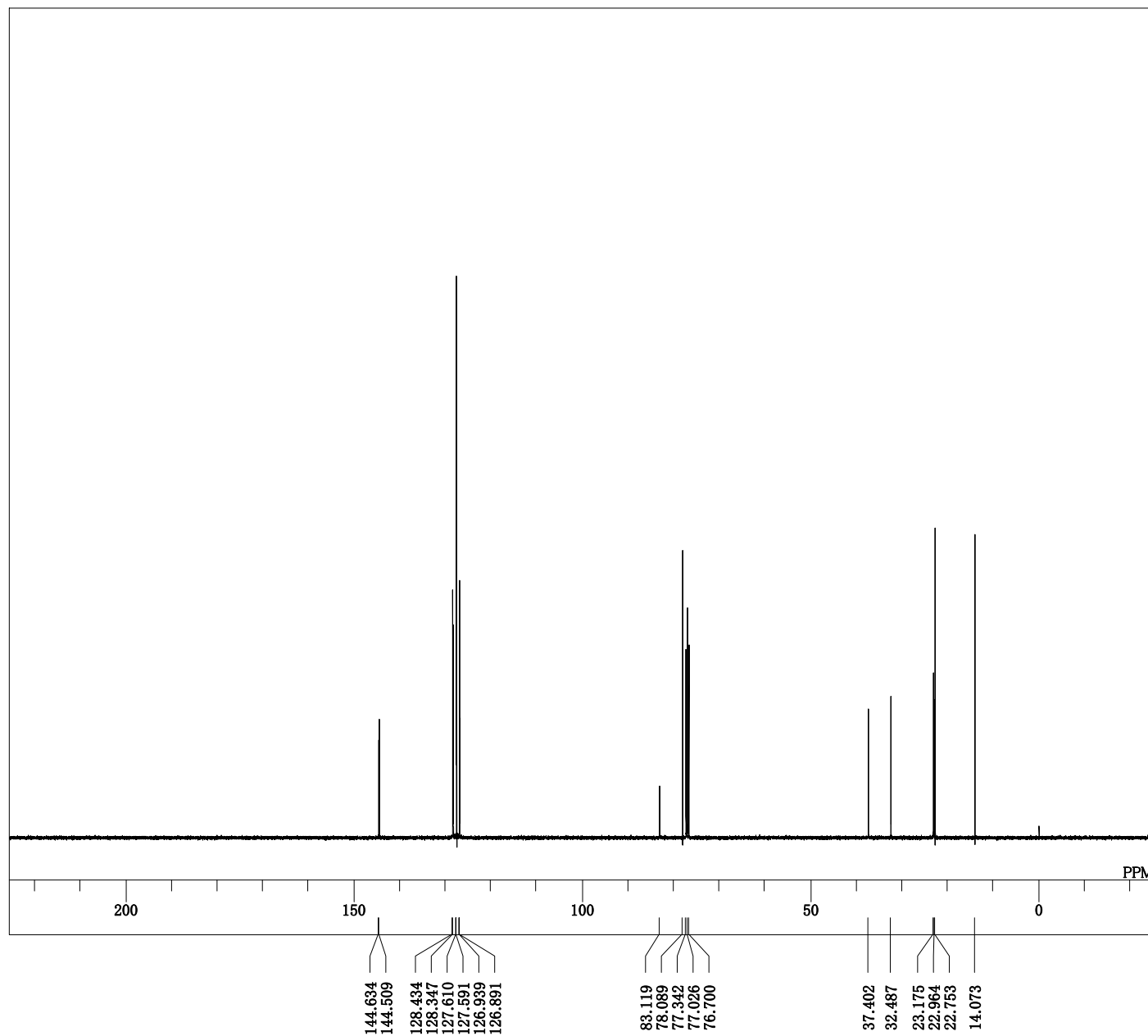
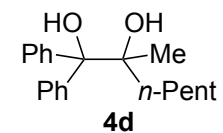
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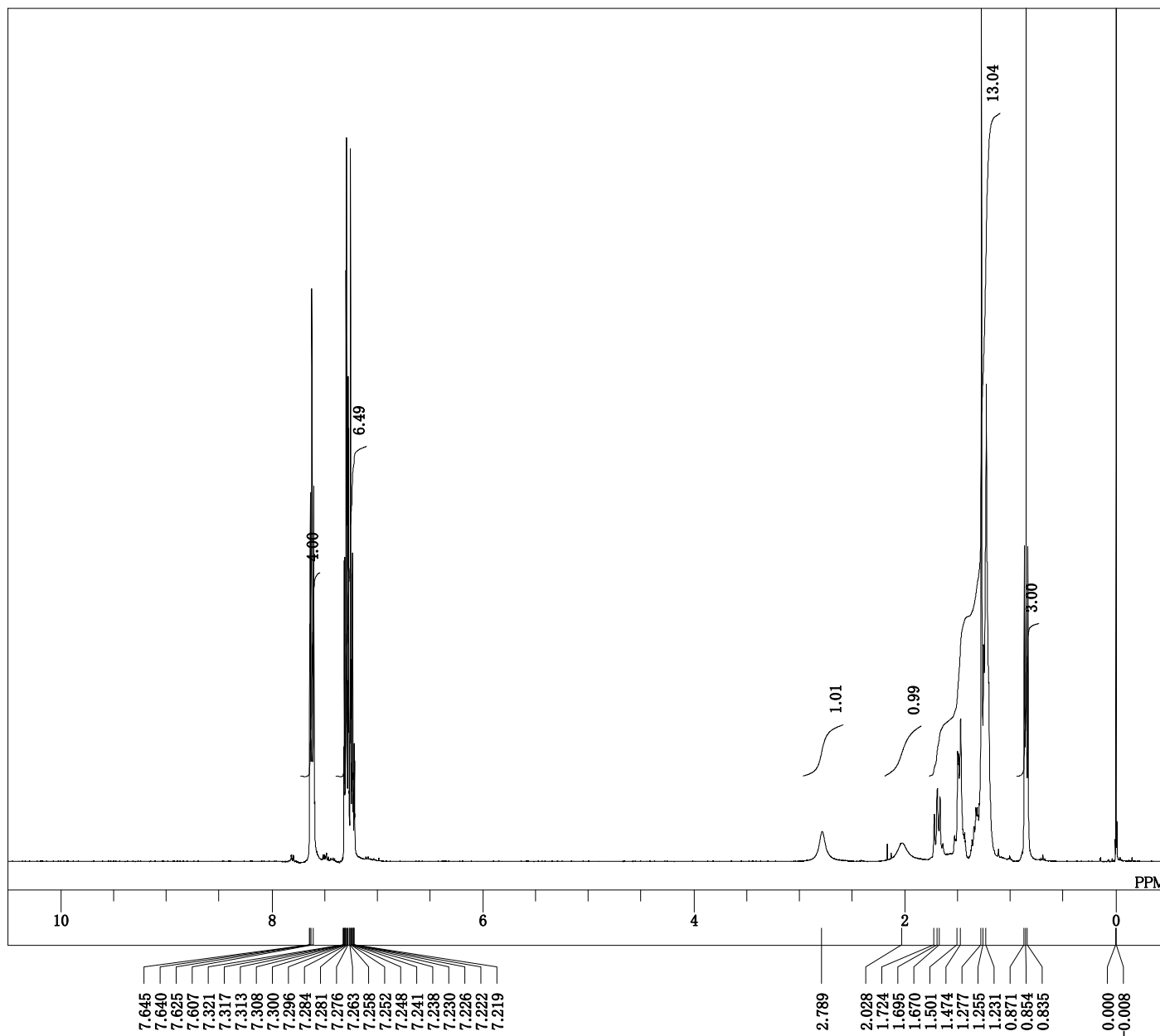


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 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 20

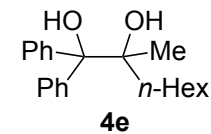


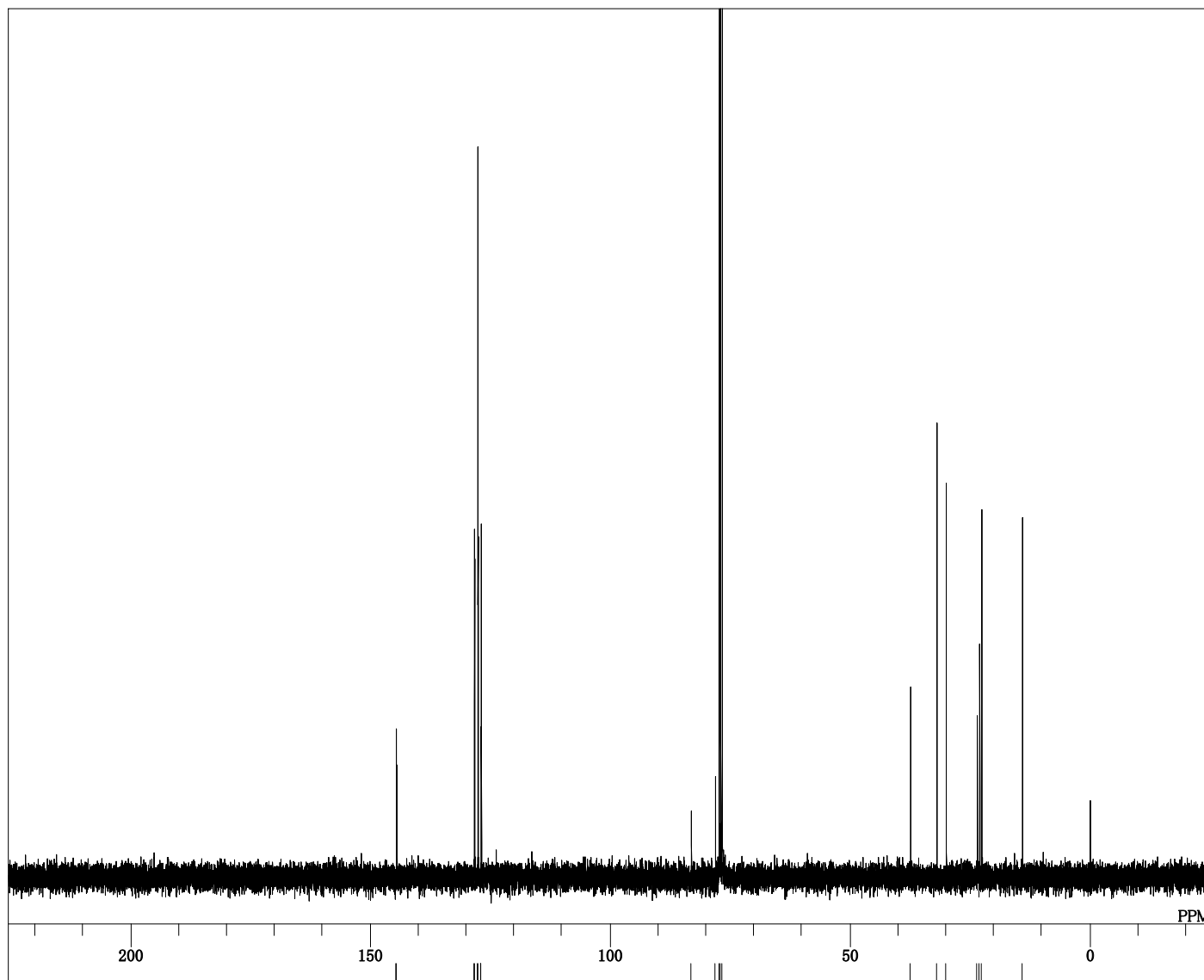
DFILE diol_PhPhMenPentyl_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2022-04-25 23:22:03
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 1024
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.67 usec
 IRNUC 1H
 CTEMP 20.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50



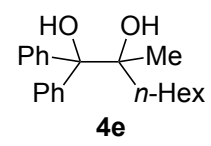


DFILE diol_Me_nHex_Proton.als
 COMNT single_pulse
 DATIM 2020-02-24 08:45:15
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.1 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 34





DFILE diol_Me_nHex_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-02-24 08:46:27
 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

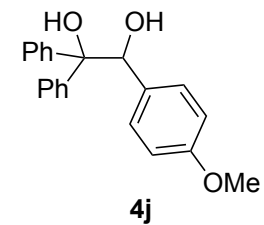
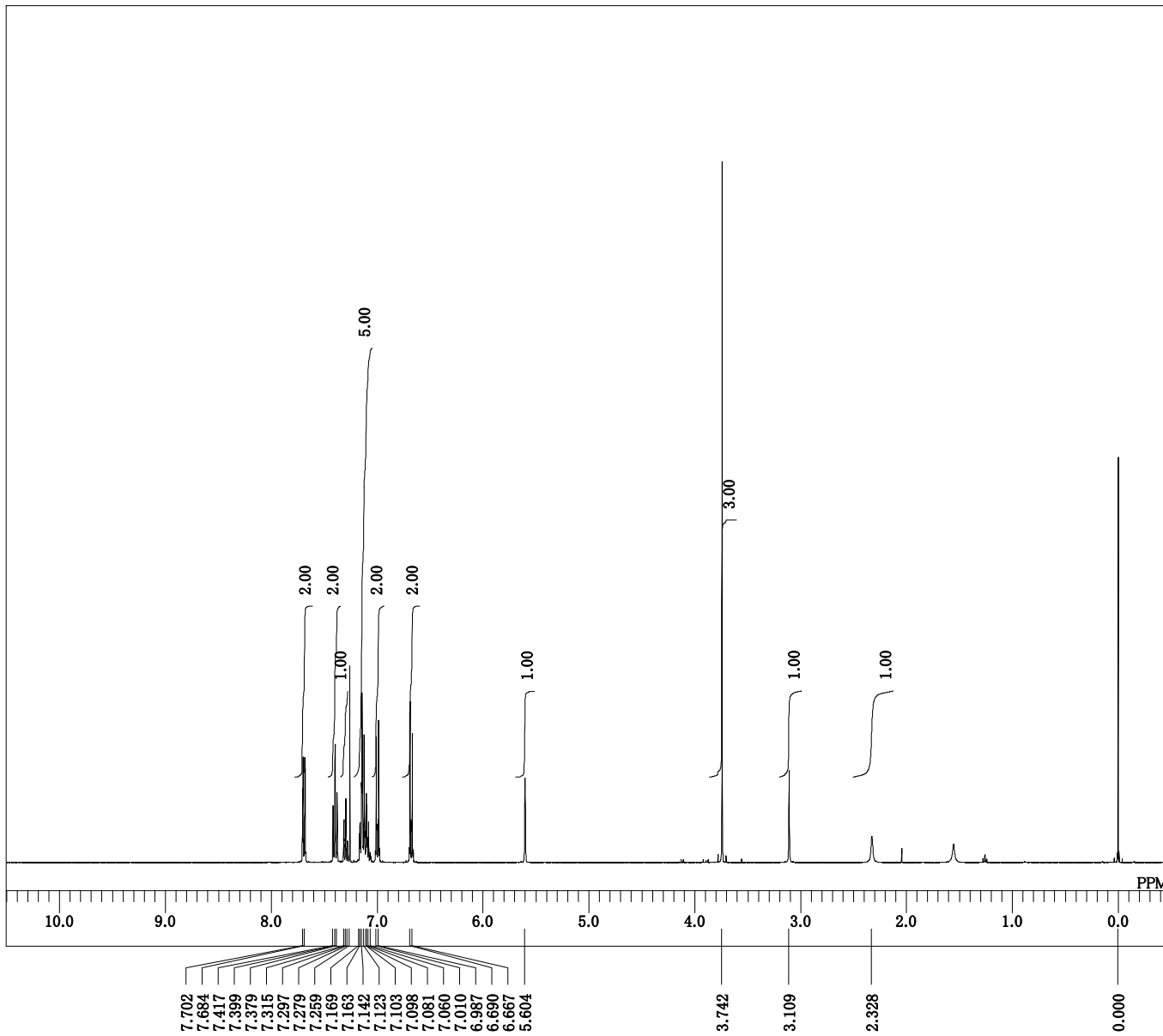


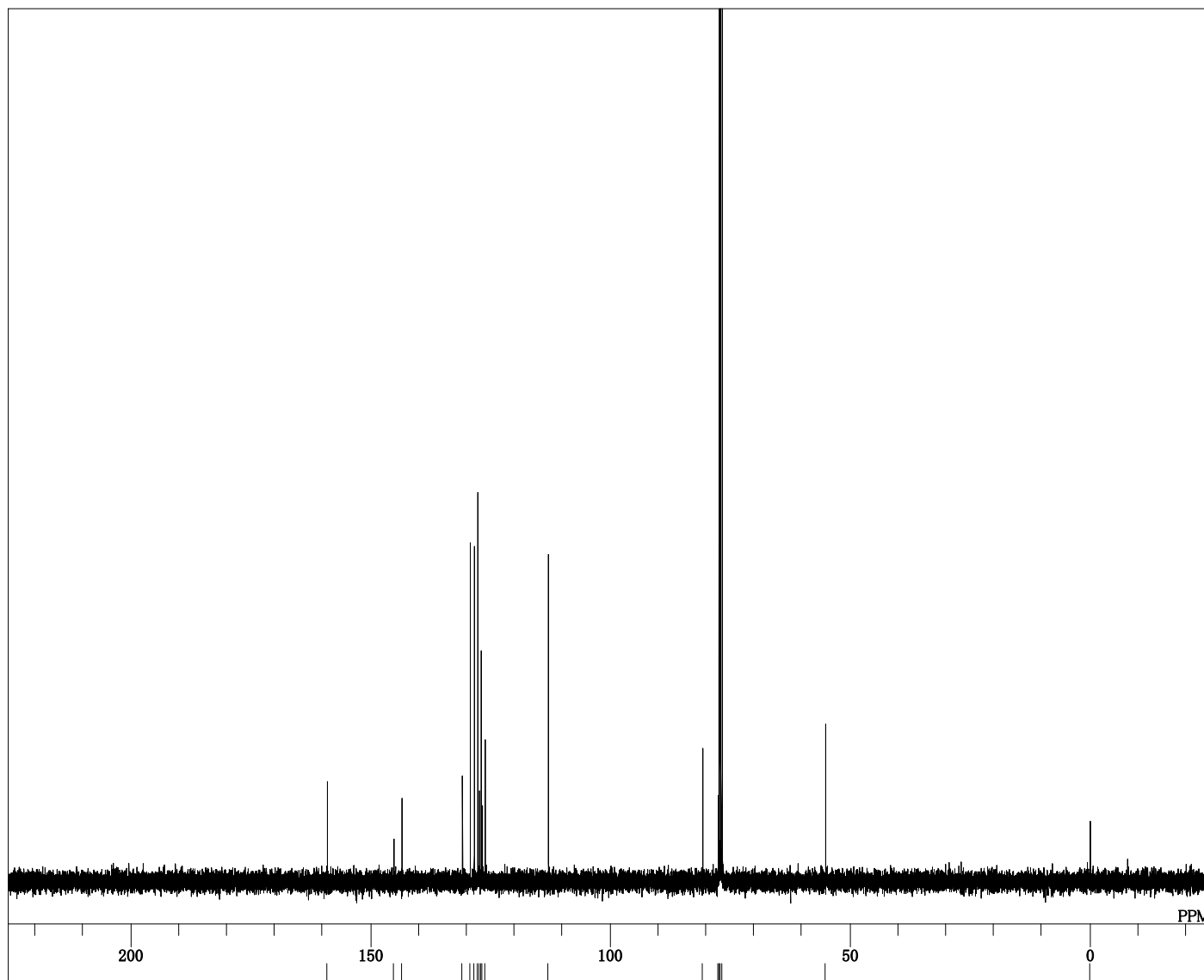
144.619
 144.494
 128.399
 128.313
 127.614
 127.594
 126.933
 126.895

83.093
 78.073
 77.316
 77.000
 76.674

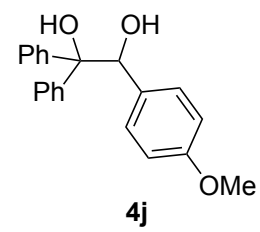
37.433
 31.934
 29.941
 23.464
 22.985
 22.602
 14.066

DFILE diol_H_pOMe_Proton-1-1.als
 COMNT single_pulse
 DATIM 2021-01-12 12:12:35
 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 18.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 40





DFILE diol_H_pOMe_Carbon-1-1.als
 COMNT single pulse decoupled gated NOE
 DATIM 2021-01-12 12:13:47
 OBNUC ¹³C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 925
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 19.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50



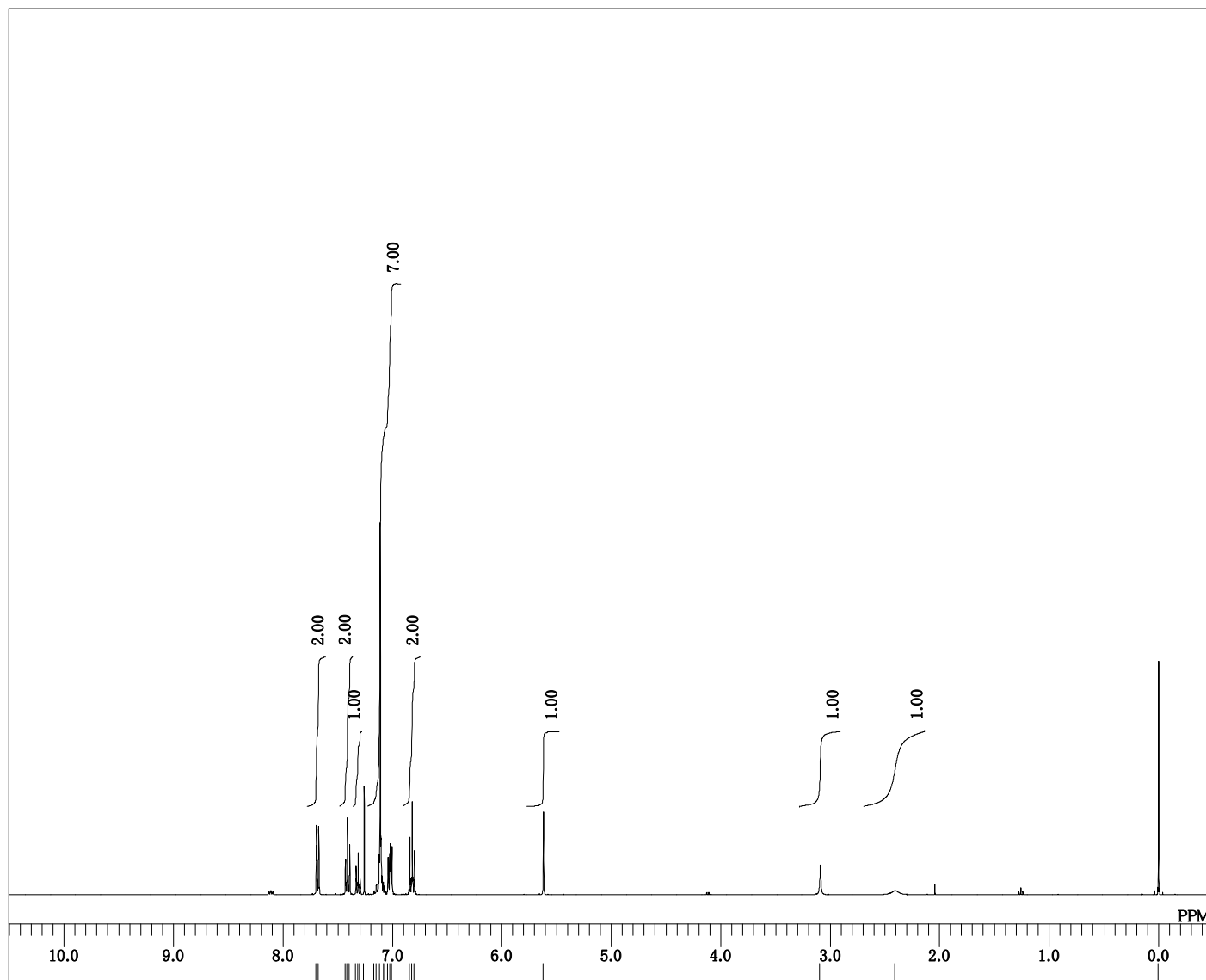
159.055
 145.125
 143.487
 130.860
 129.193
 128.445
 127.660
 127.343
 126.941
 126.673
 126.156
 112.915

80.744
 77.573
 77.333
 77.017
 76.692

55.145

0.000

DFILE dioLH_pF_Proton-1-1.als
 COMNT single_pulse
 DATIM 2021-01-07 13:18:48
 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 19.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 40

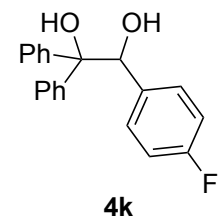


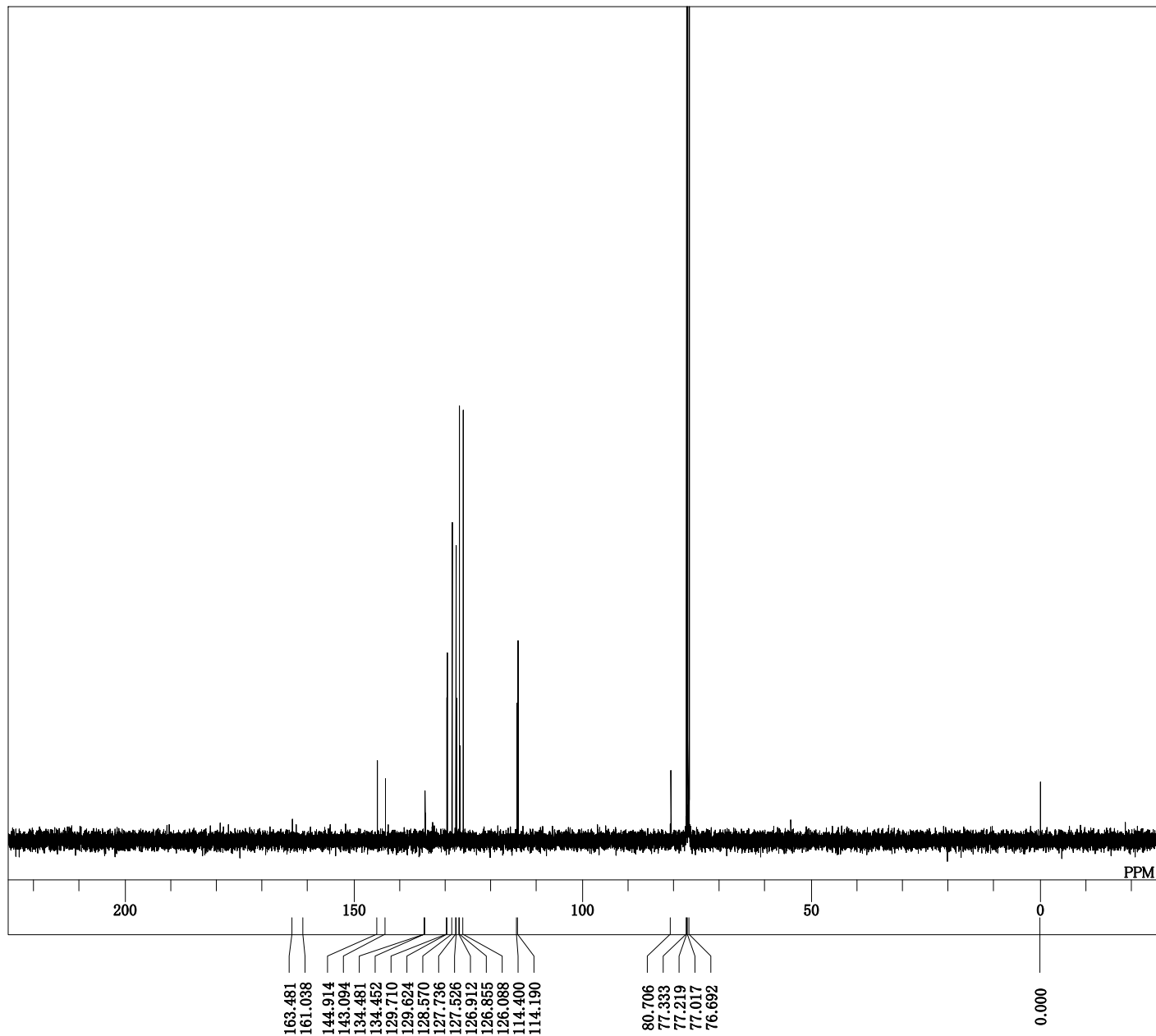
7.693
 7.674
 7.428
 7.410
 7.391
 7.331
 7.313
 7.295
 7.258
 7.168
 7.147
 7.111
 7.075
 7.065
 7.040
 7.018
 7.004
 6.841
 6.820
 6.797
 5.619

3.090

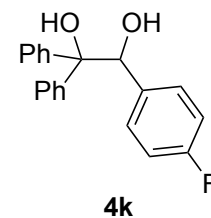
2.404

0.000

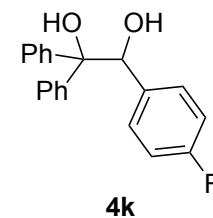
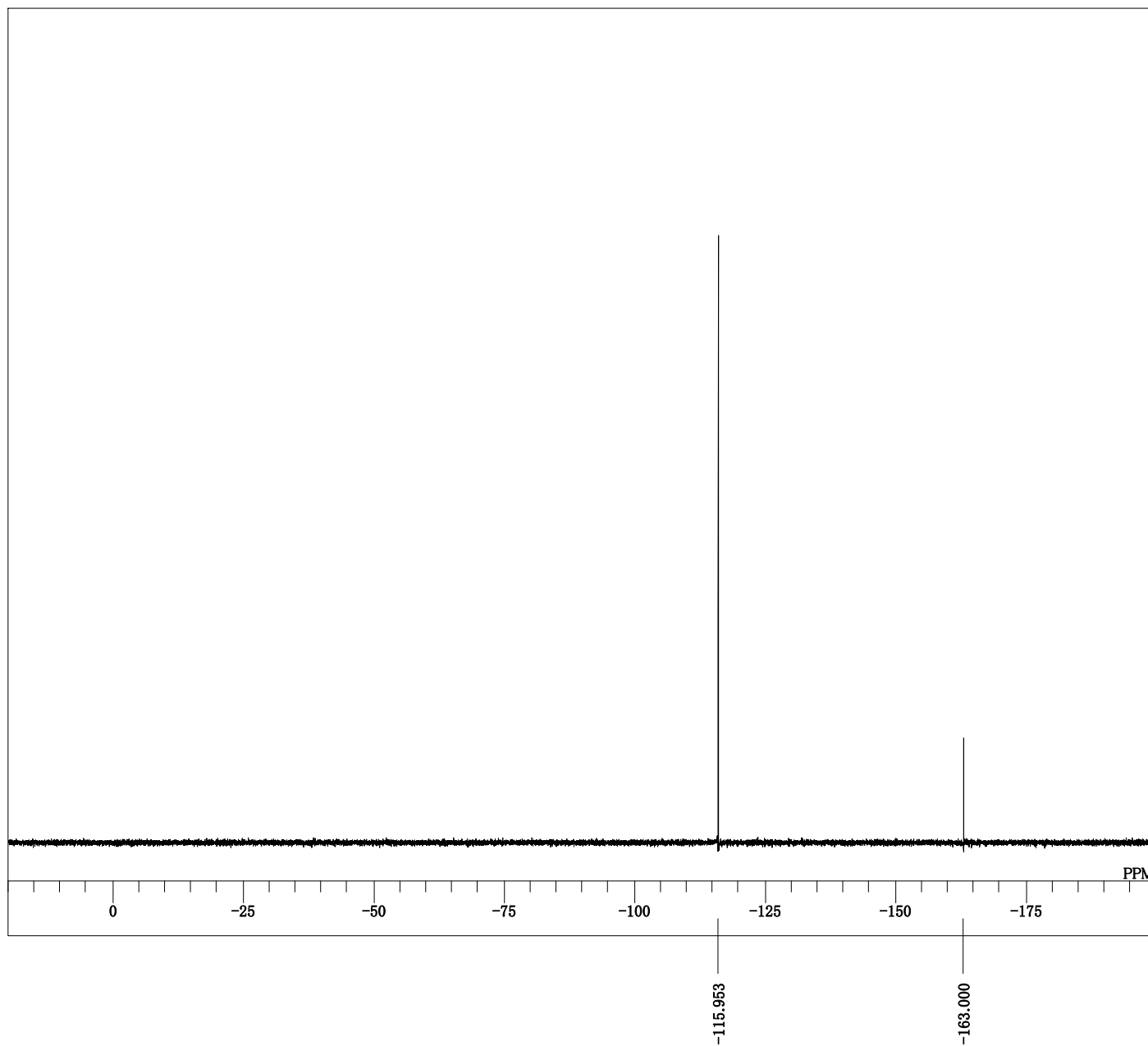


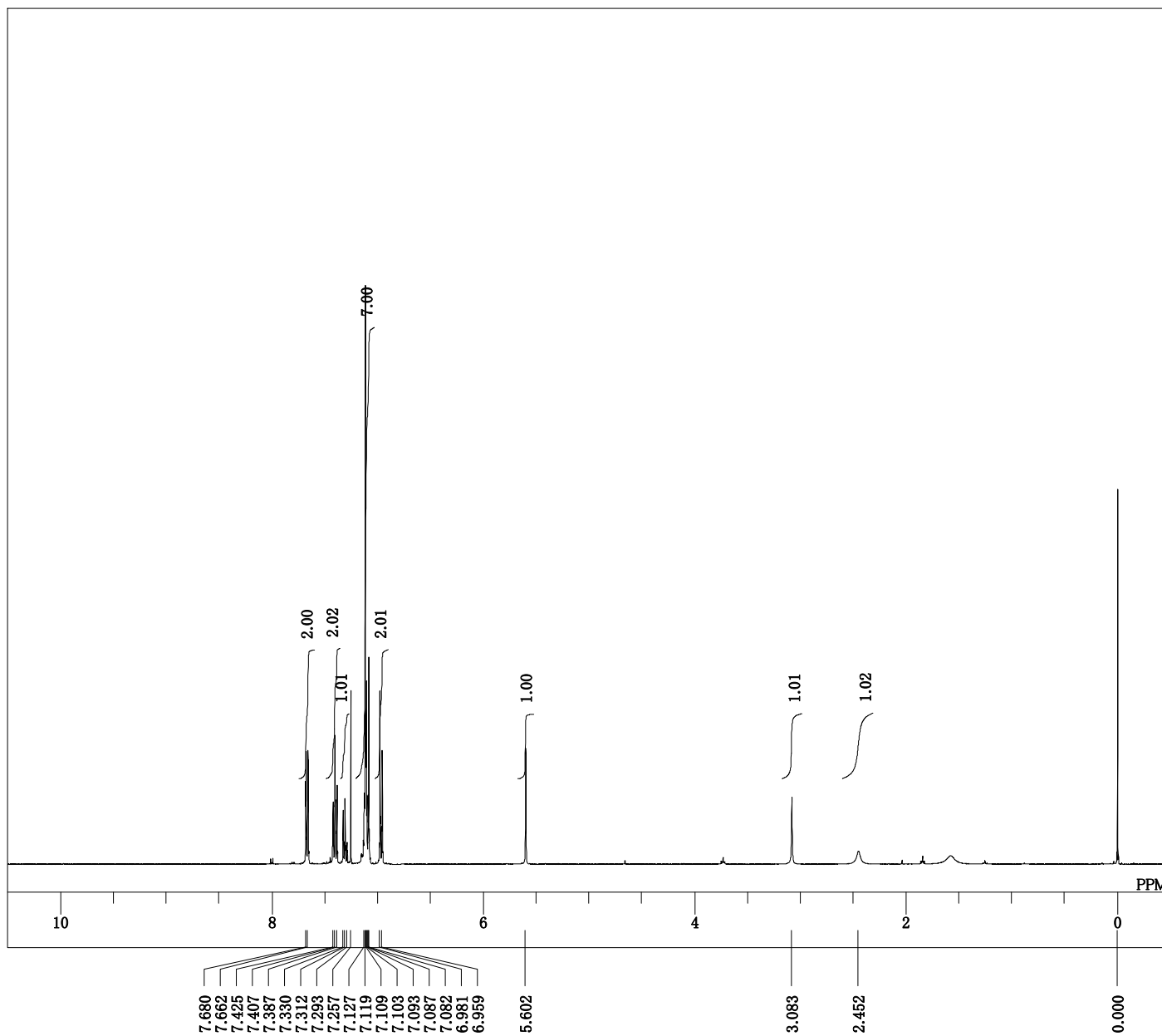


DFILE diol_H_pF_Carbon-1-1.als
 COMNT single pulse decoupled gated NOE
 DATIM 2021-01-07 13:20:01
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 1024
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 18.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50

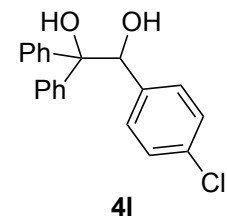


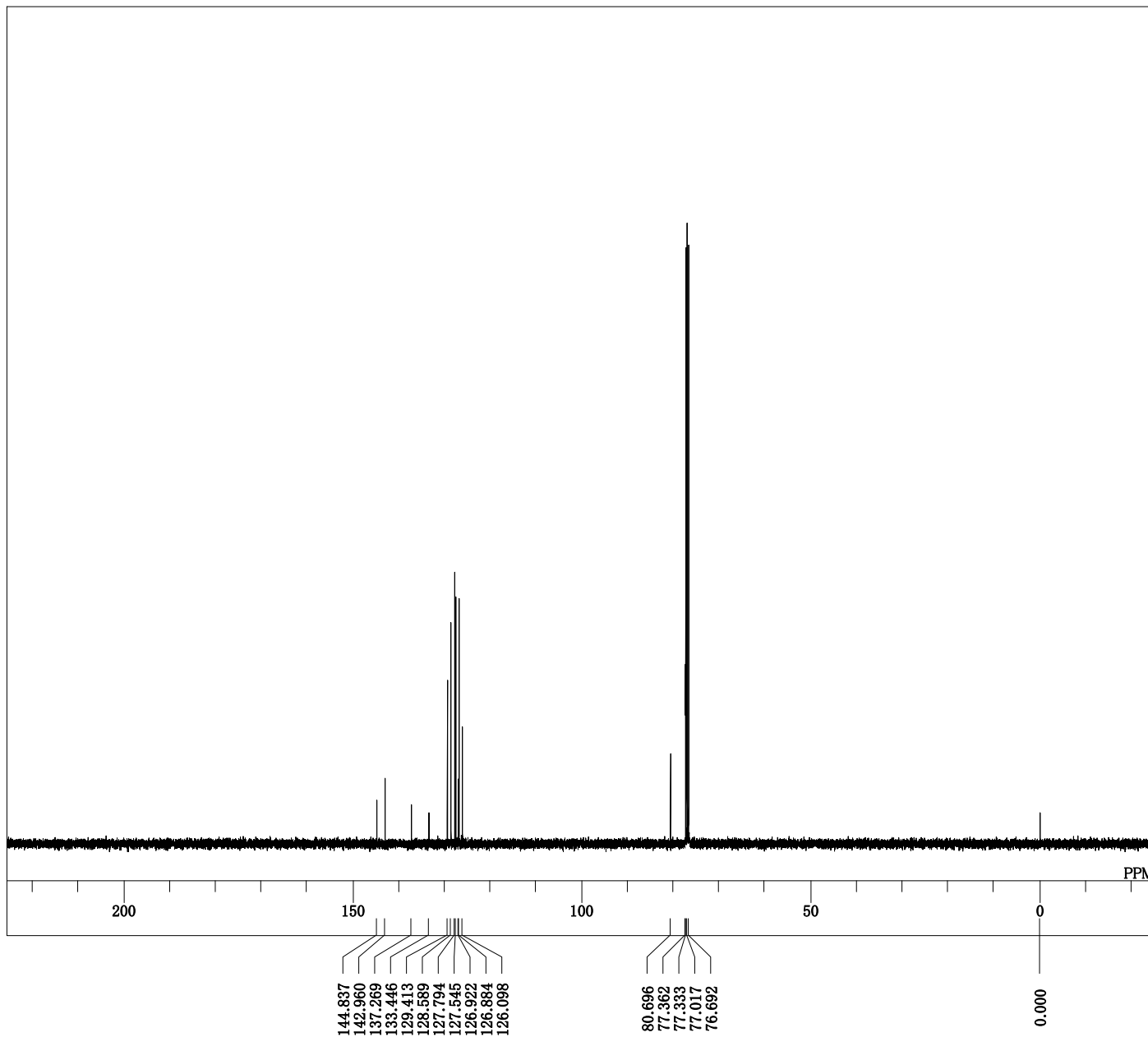
DFILE dio_L_H_pF_Fluorine.als
COMNT single_pulse
DATIM 2022-07-04 21:20:03
OBNUC 19F
EXMOD single_pulse.jxp
OBFRQ 372.50 MHz
OBSET 3.36 KHz
OBFIN 6.86 Hz
POINT 26214
FREQU 149253.73 Hz
SCANS 8
ACQTM 0.1756 sec
PD 5.0000 sec
PW1 3.98 usec
IRNUC 19F
CTEMP 20.4 c
SLVNT CDCL3
EXREF -163.00 ppm
BF 0.12 Hz
RGAIN 42



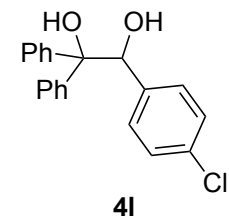


DFILE Ex638_column_Proton-1-1.als
 COMNT single_pulse
 DATIM 2020-06-03 16:18:50
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 38

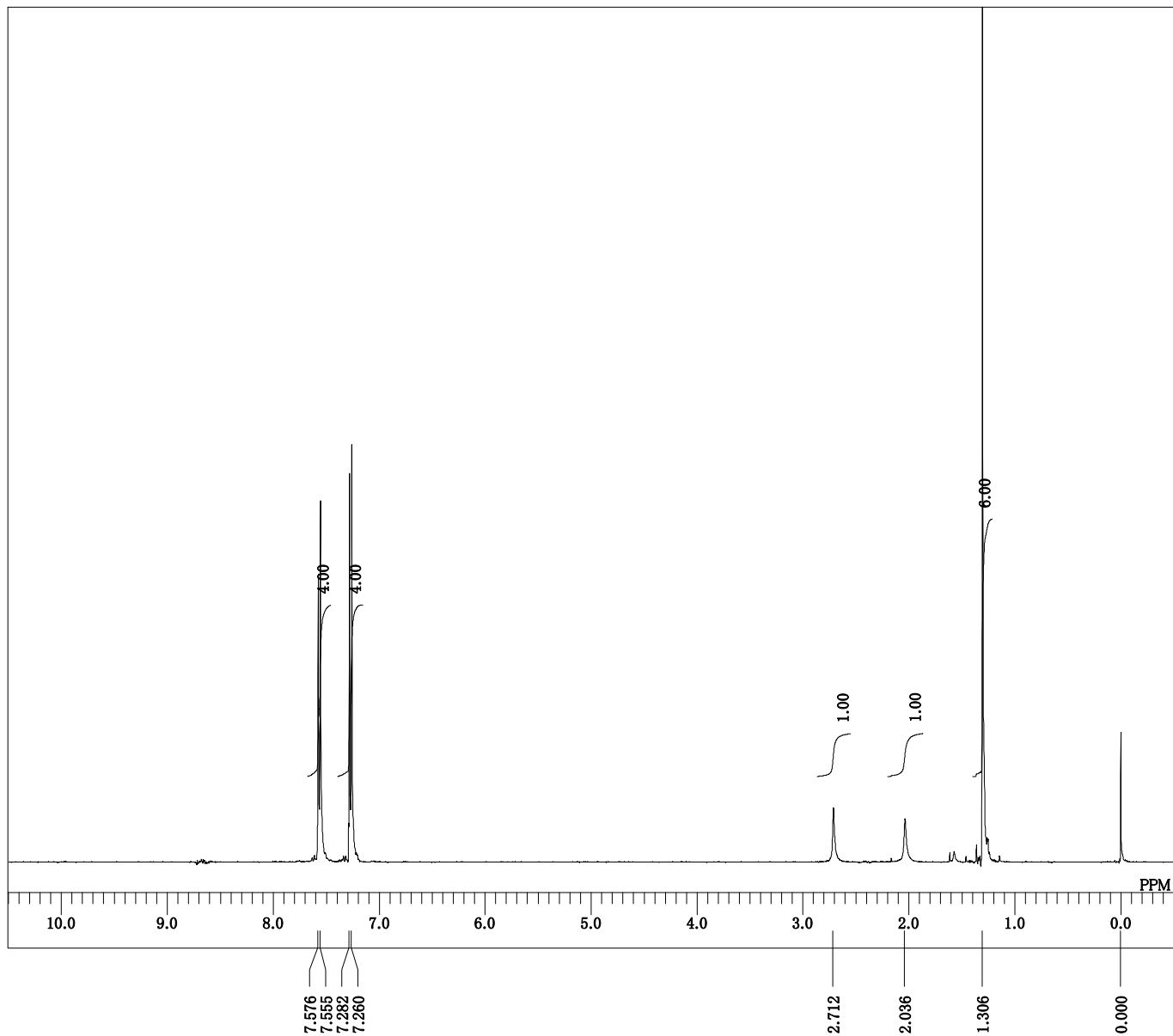
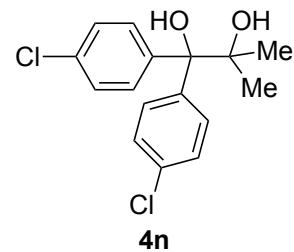


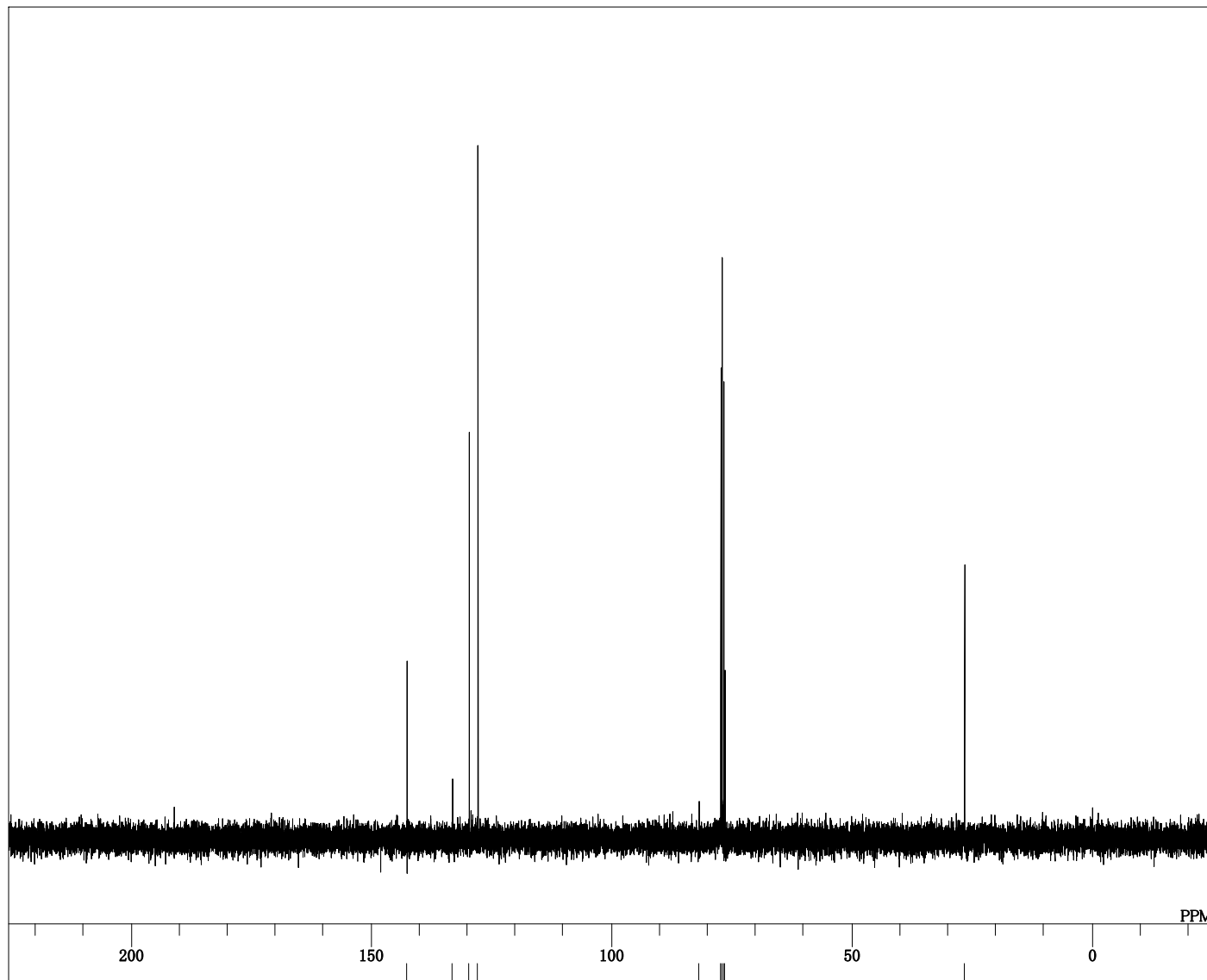


DFILE Ex638_column_Carbon-1-1.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-06-03 16:20:02
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 1024
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 20.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50

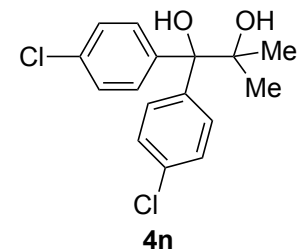


DFILE dioLpClpCl_MeMe_Proton.als
COMNT
DATIM Sun Nov 22 02:28:11 2020
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 2.0000 sec
PW1 6.60 usec
IRNUC 1H
CTEMP 6348.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 18





DFILE diol_pClpClMeMe_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-11-22 00:44:47
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 44
 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



142.606

133.169

129.701

127.843

81.848

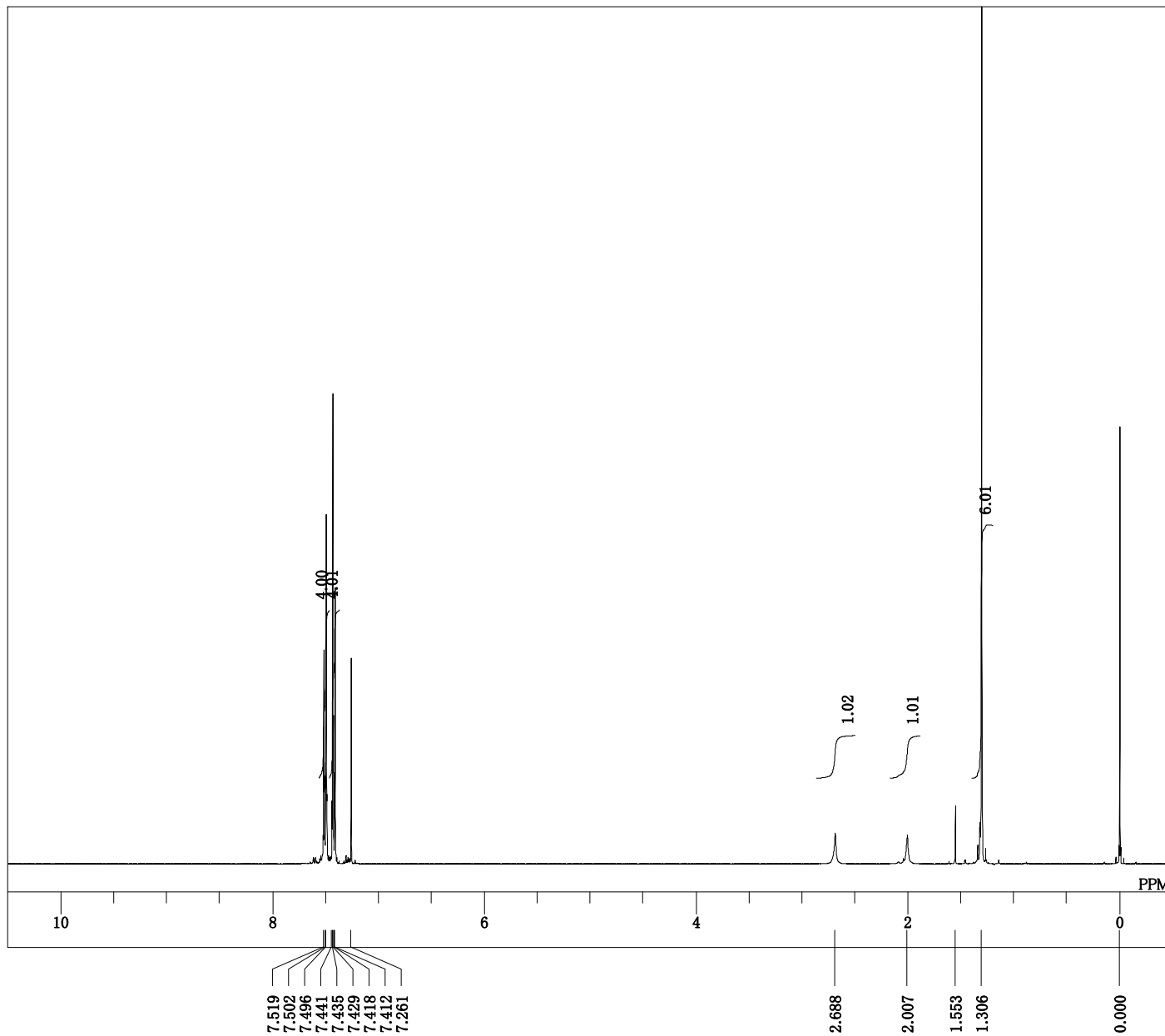
77.316

77.000

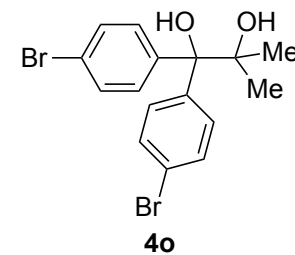
76.674

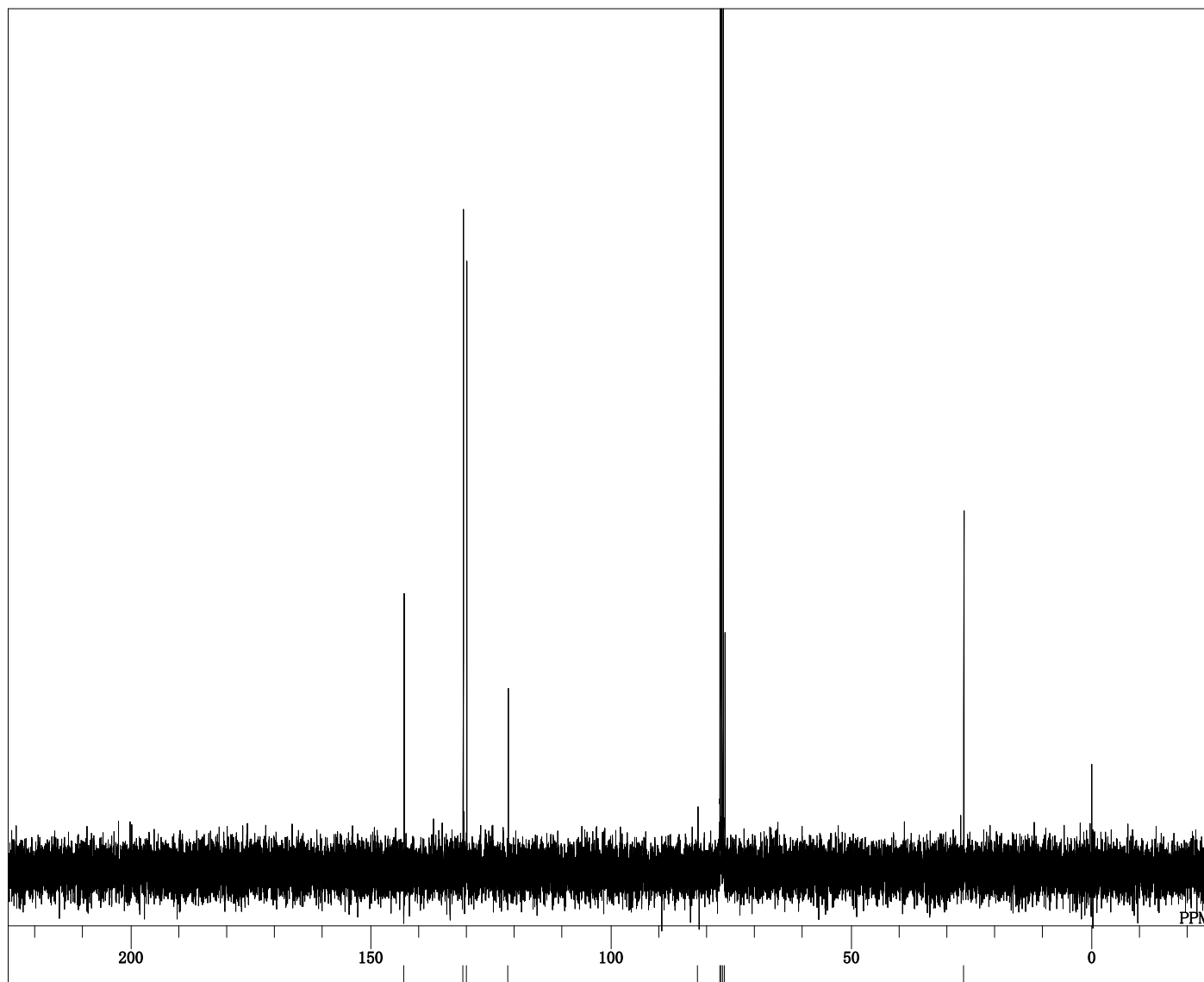
76.387

26.569

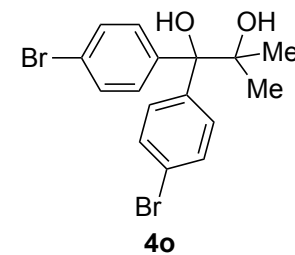


DFILE diol_pBrpBr_MeMe_Proton.als
 COMNT single_pulse
 DATIM 2020-12-02 09:02:55
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 18.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 40





DFILE diol_pBrpBr_MeMe_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-12-02 09:04:07
 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 19.1 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



143.066

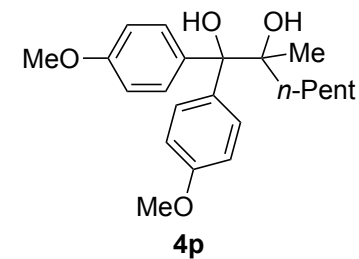
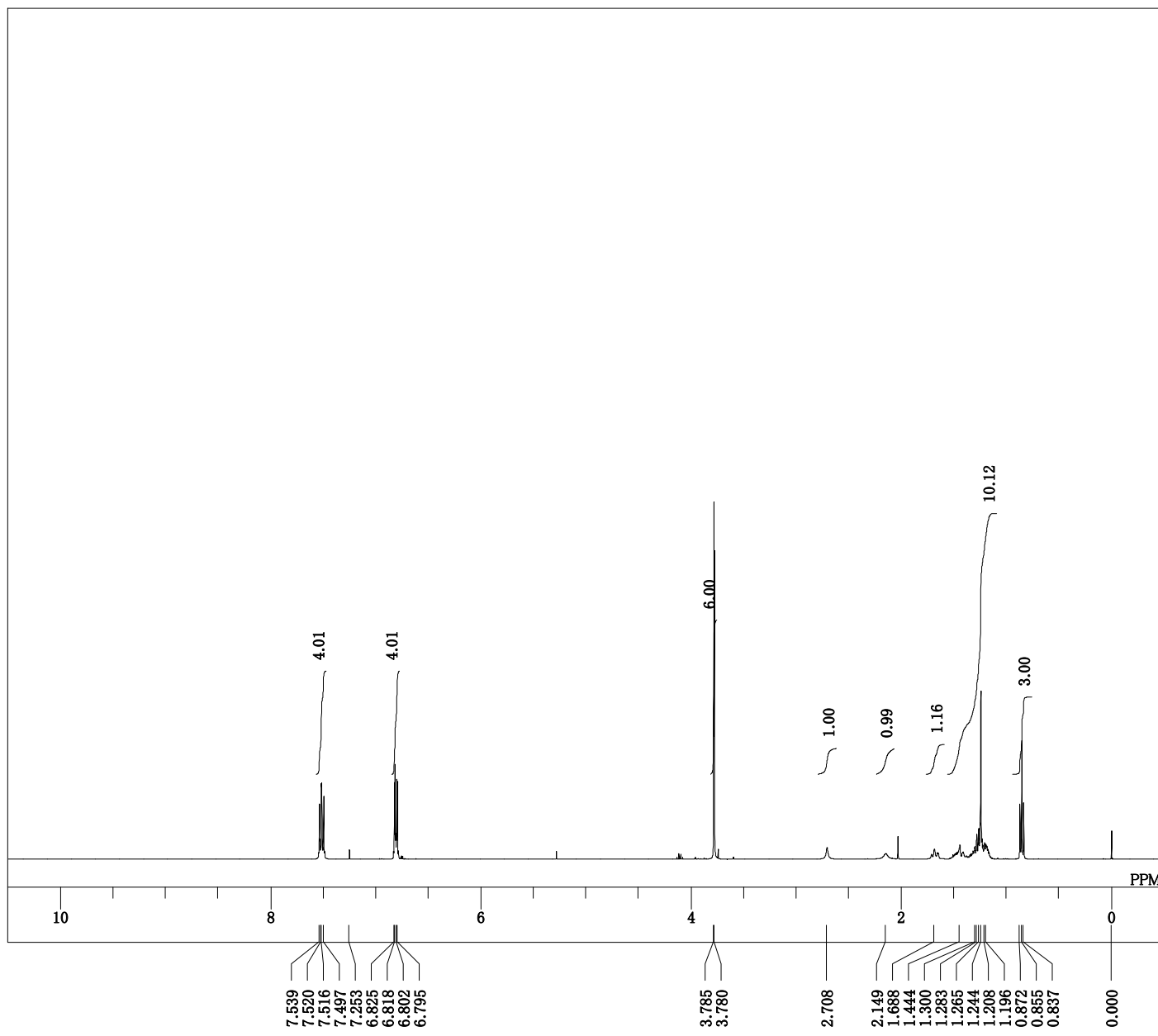
130.832
130.046

121.472

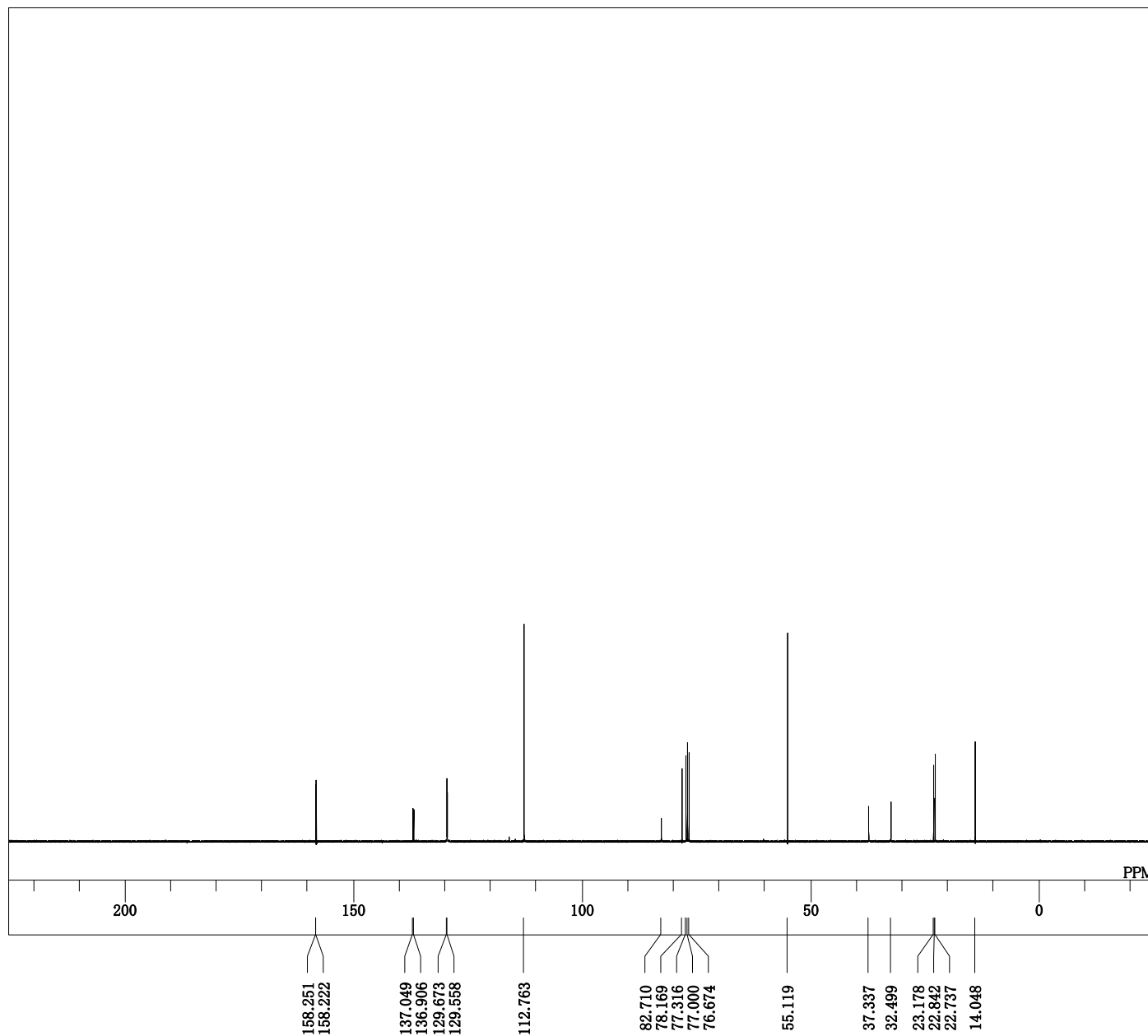
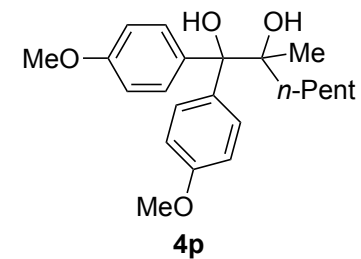
81.943
77.326
77.000
76.684
76.320

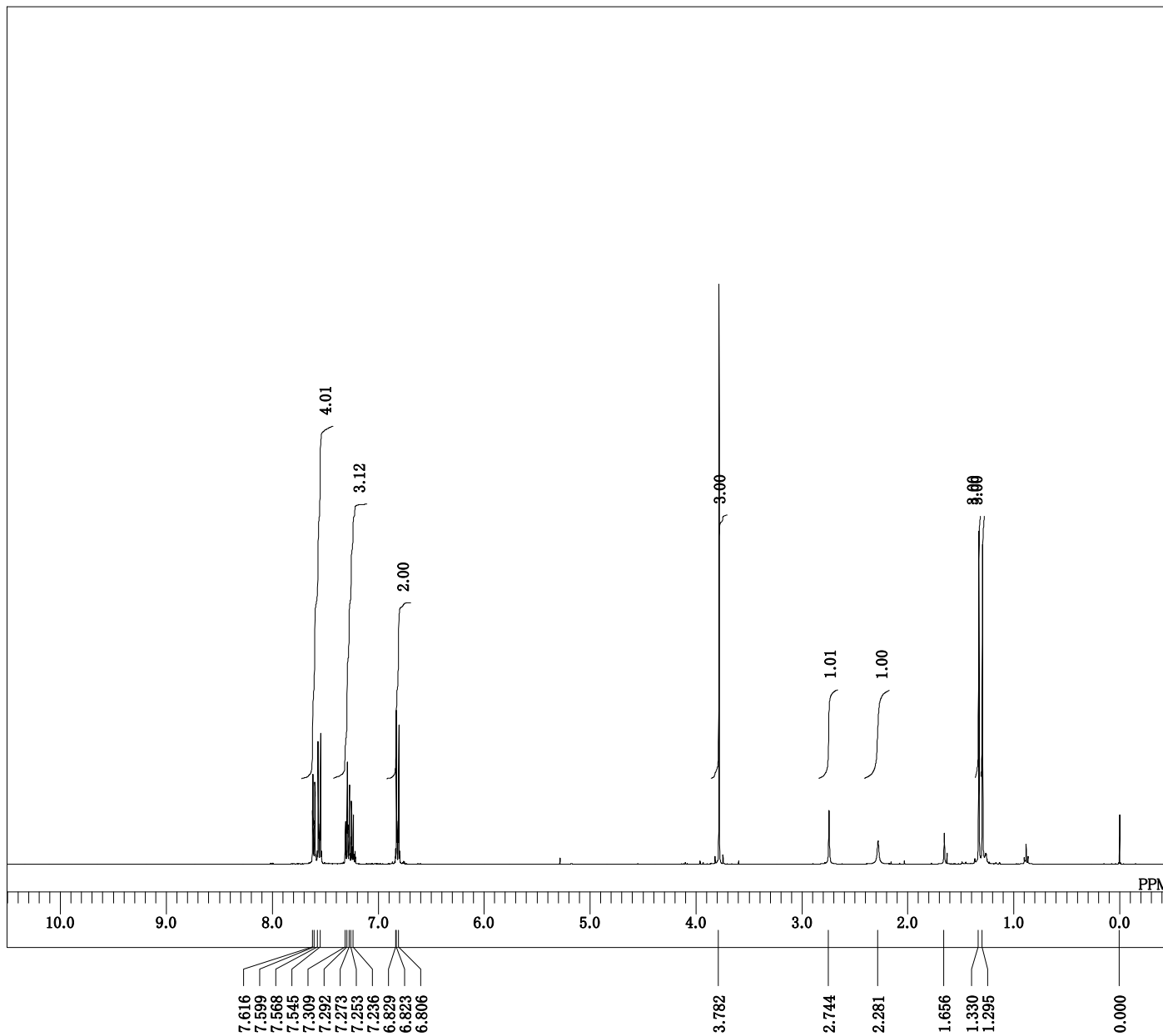
26.579

DFILE Ex834_20220426_Proton-1-1.als
 COMNT single_pulse
 DATIM 2022-04-26 00:24:26
 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 3.25 usec
 IRNUC 1H
 CTEMP 20.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 22

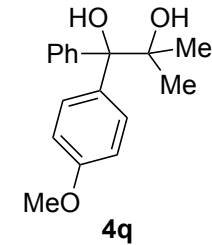


DFILE diol_PMPPMPhMenPentyl_Carbon.a
 COMNT single pulse decoupled gated NOE
 DATIM 2022-04-26 00:27:20
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 1024
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.67 usec
 IRNUC 1H
 CTEMP 20.3 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50

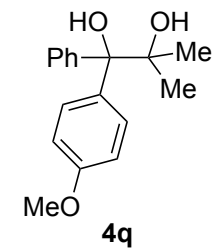
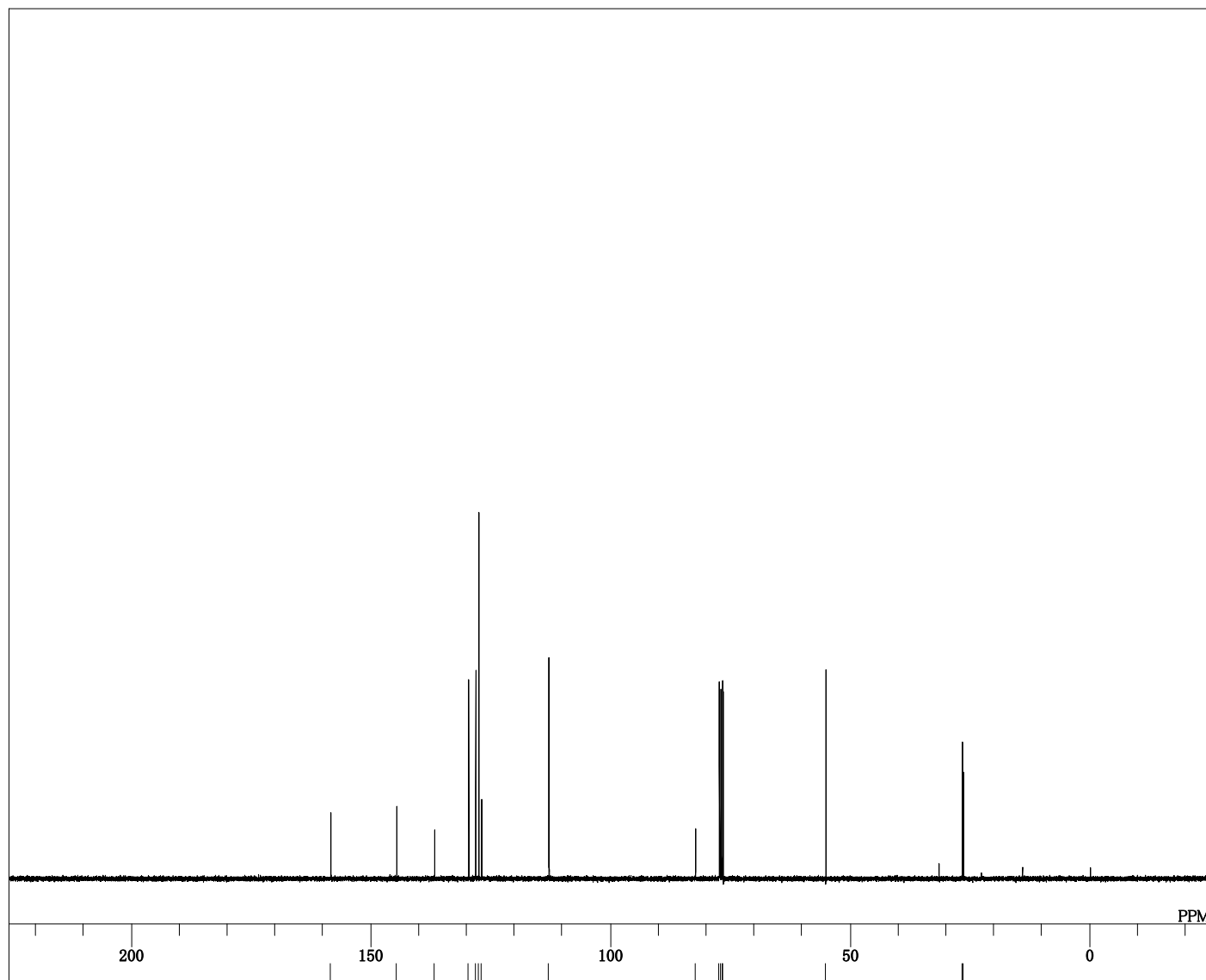




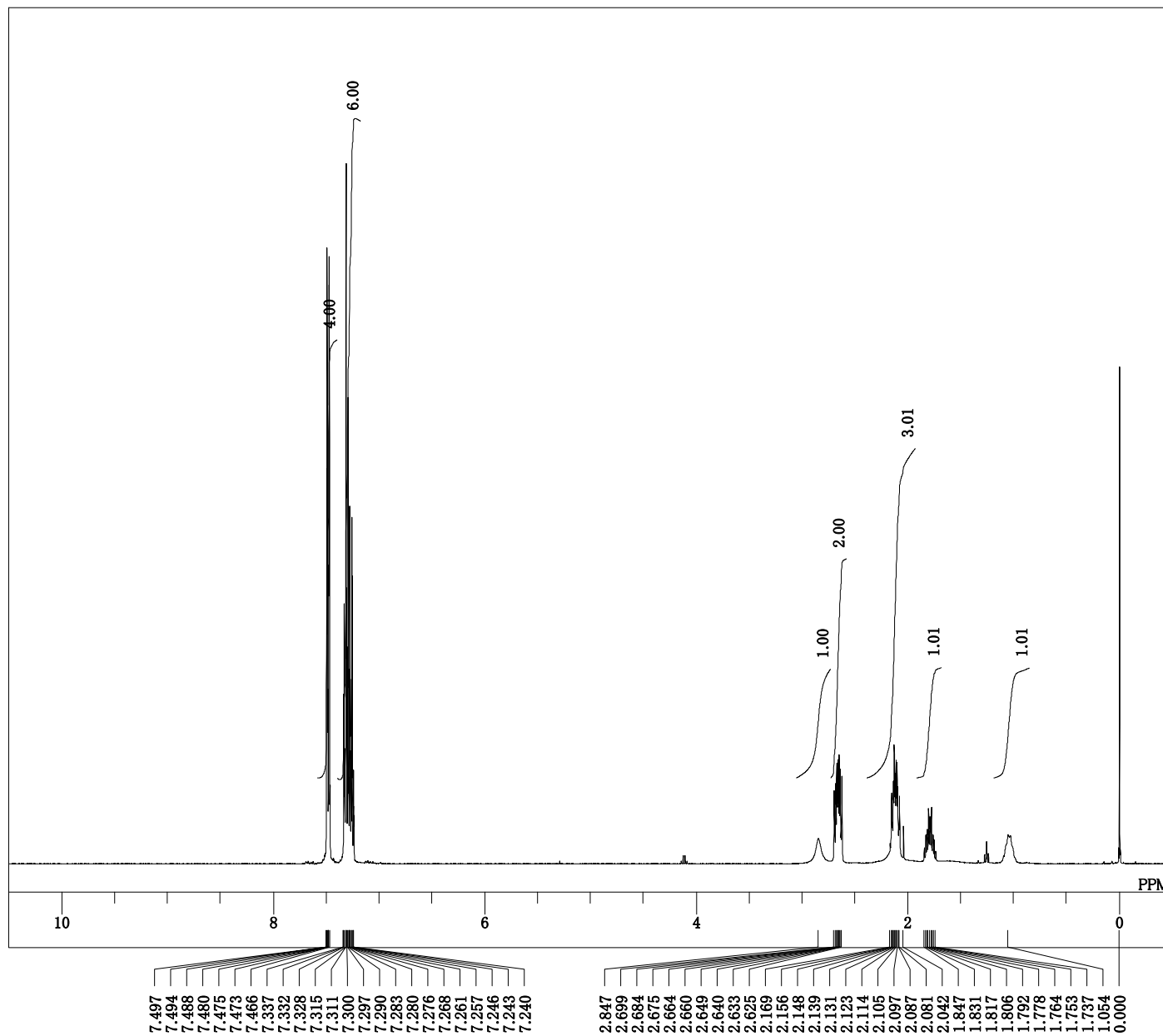
DFILE dio_PhPMP_MeMe_Proton.als
 COMNT single_pulse
 DATIM 2020-09-12 14:02:27
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 26



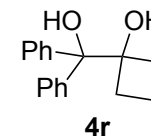
DFILE diol_PhPMP_MeMe_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-09-12 14:03:39
 OBNUC ¹³C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 508
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC ¹H
 CTEMP 20.3 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50

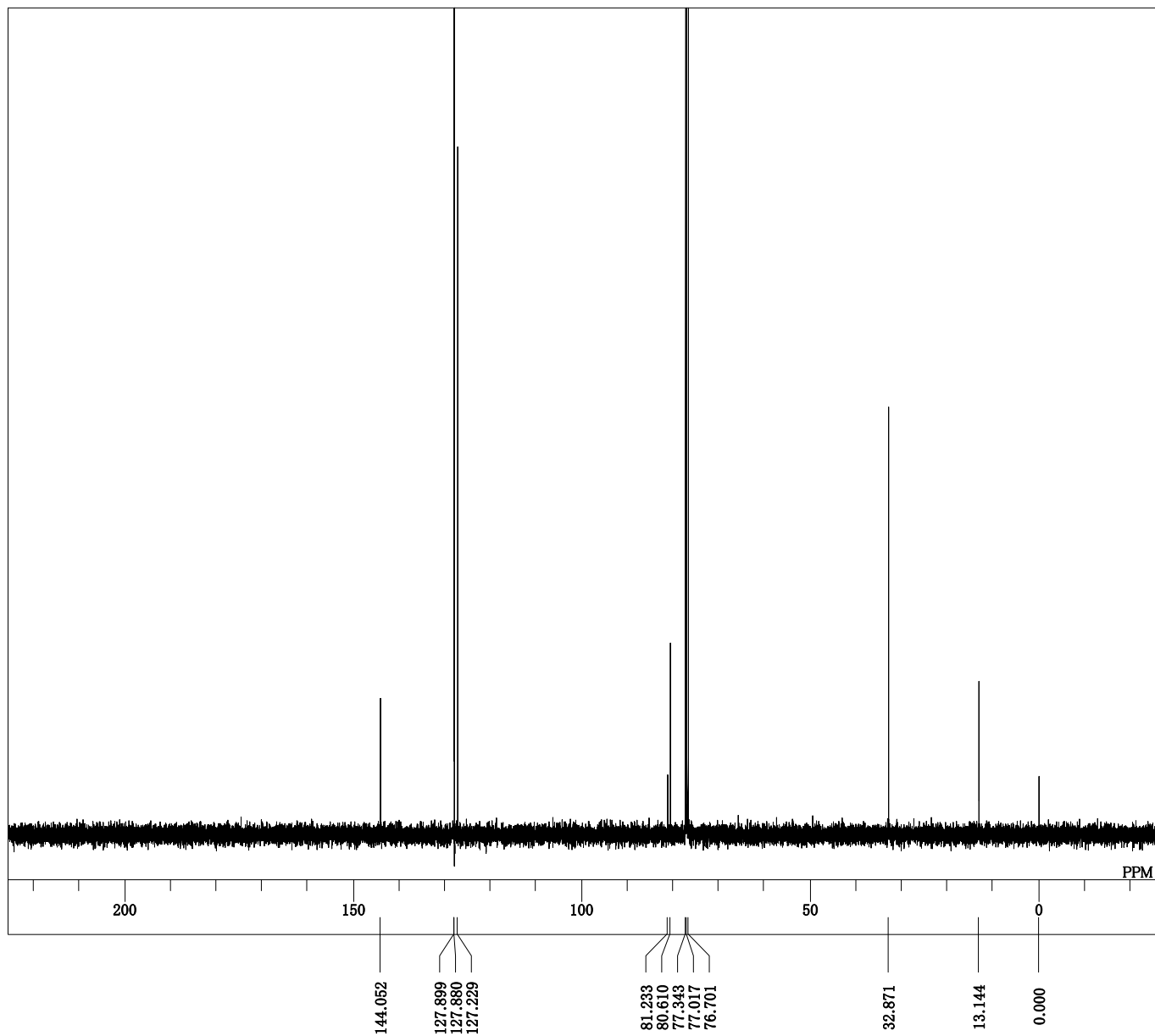


158.367
 144.648
 136.763
 129.625
 128.179
 127.537
 126.905
 112.888
 82.317
 77.326
 77.000
 76.684
 76.492
 55.147
 26.655
 26.425

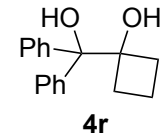


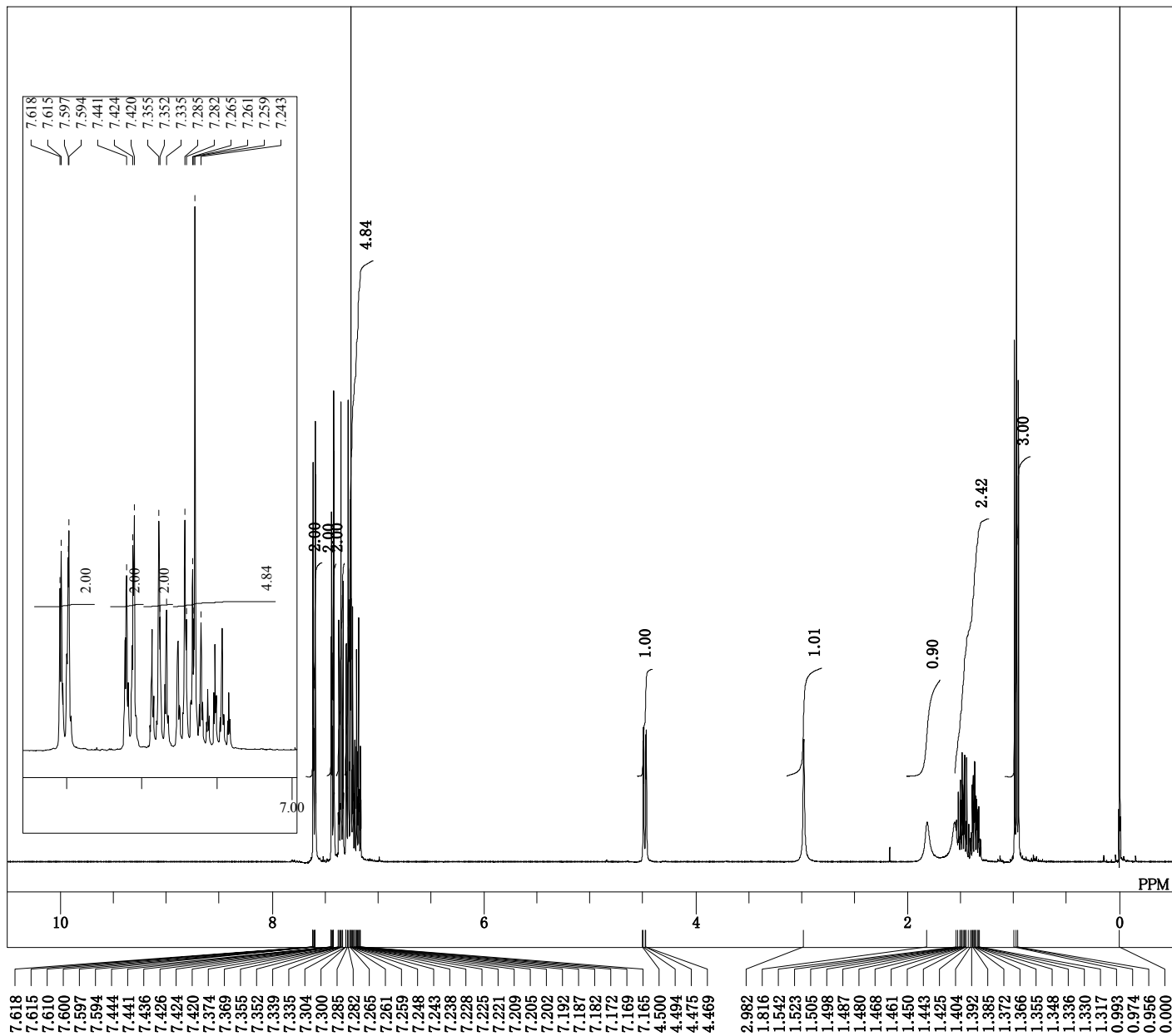
DFILE diol_cyc.butylProton.als
 COMNT single_pulse
 DATIM 2020-02-24 09:45:14
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.0 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 36



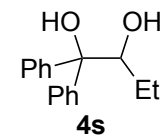


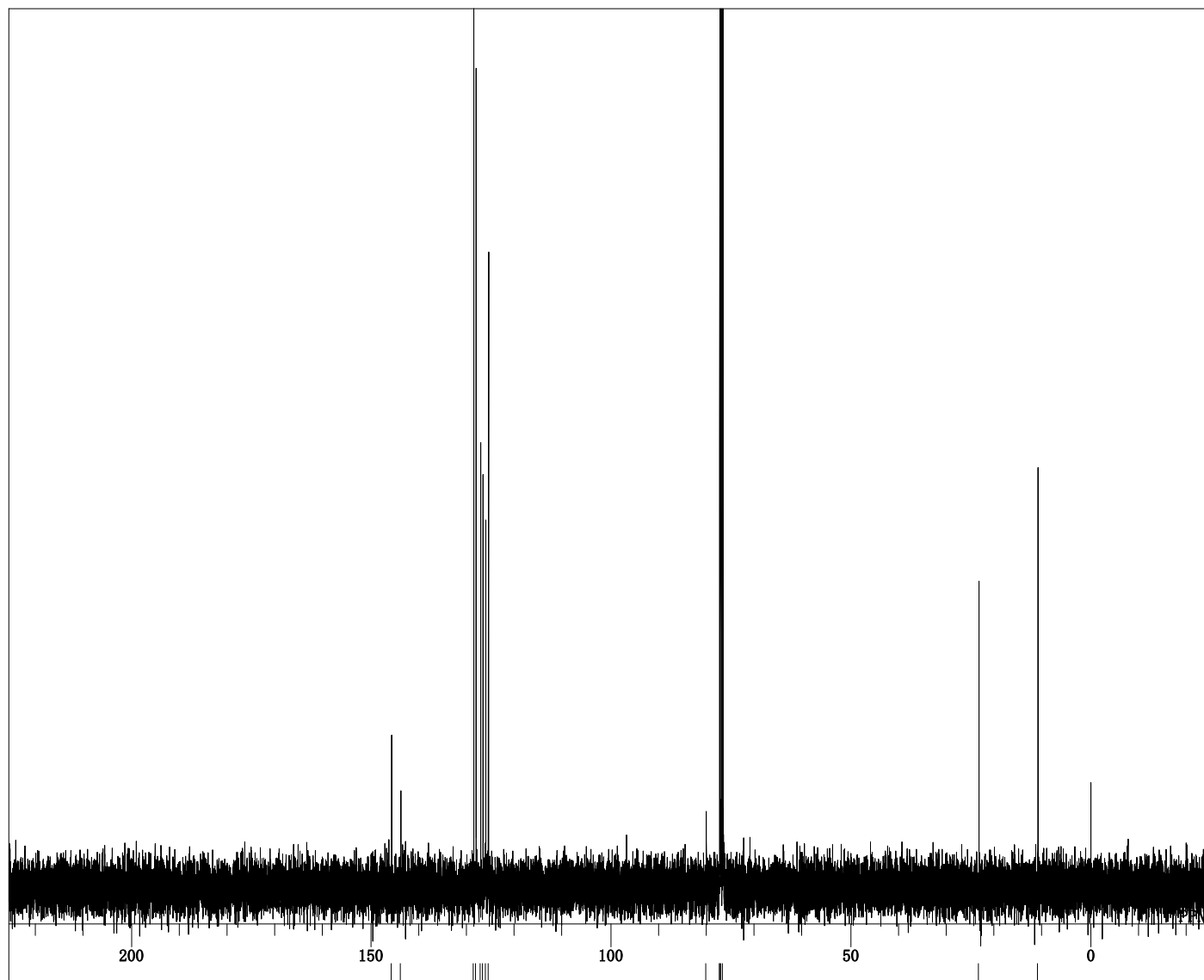
DFILE diol_cyc.butyl_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-02-24 09:46:26
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 1024
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 20.0 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50





DFILE diol_H_Et_Proton.als
 COMNT single_pulse
 DATIM 2020-02-24 10:44:39
 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



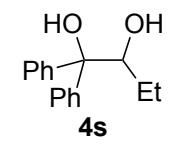


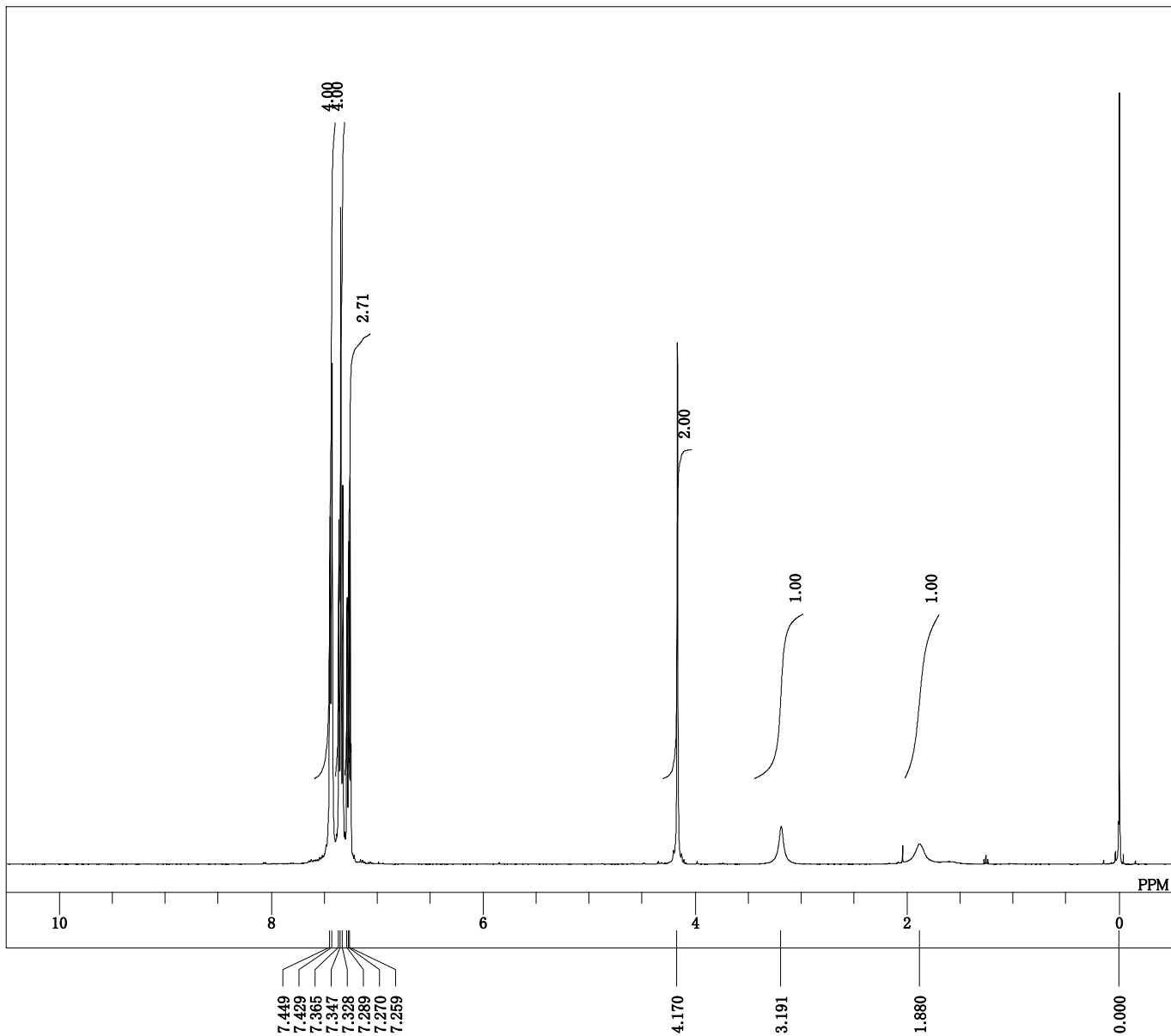
145.788
143.814
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128.160
127.202
126.732
126.138
125.573

80.104
77.326
77.287
77.000
76.684

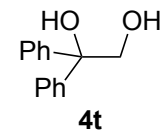
23.302
11.010

DFILE dioLH_Et_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-02-24 10:45:51
 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 19.4 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50

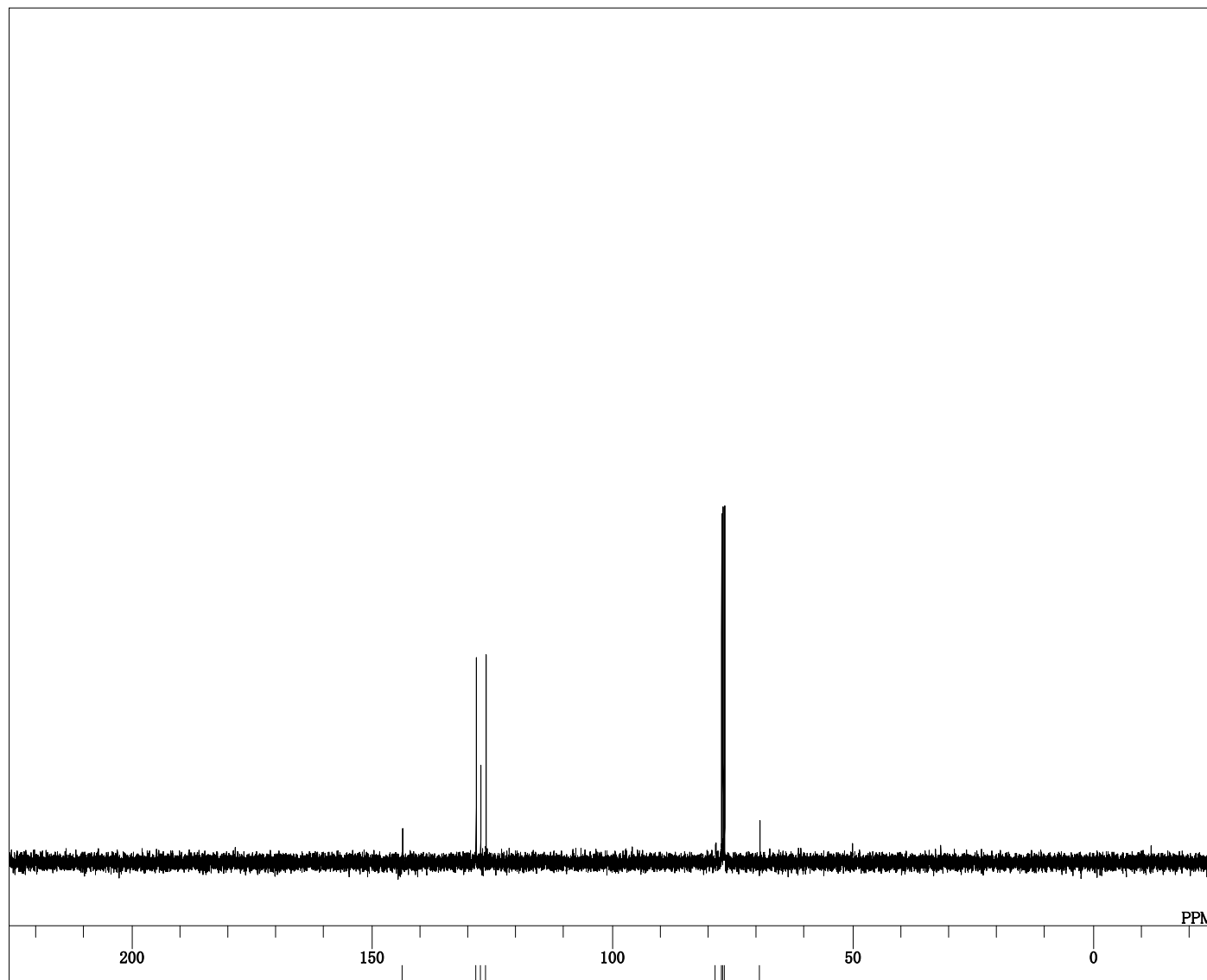




DFILE dio_L_H_Proton.als
 COMNT single_pulse
 DATIM 2020-12-22 23:59:11
 OBNUC 1H
 EXMOD proton_jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 18.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.72 Hz
 RGAIN 40



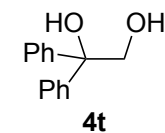
DFILE diol_H,H_Carbon.als
COMNT single pulse decoupled gated NOE
DATIM 2020-12-21 18:44:31
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 32767
FREQU 31250.00 Hz
SCANS 84
ACQTM 1.0486 sec
PD 2.0000 sec
PW1 3.59 usec
IRNUC 1H
CTEMP 19.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.72 Hz
RGAIN 50

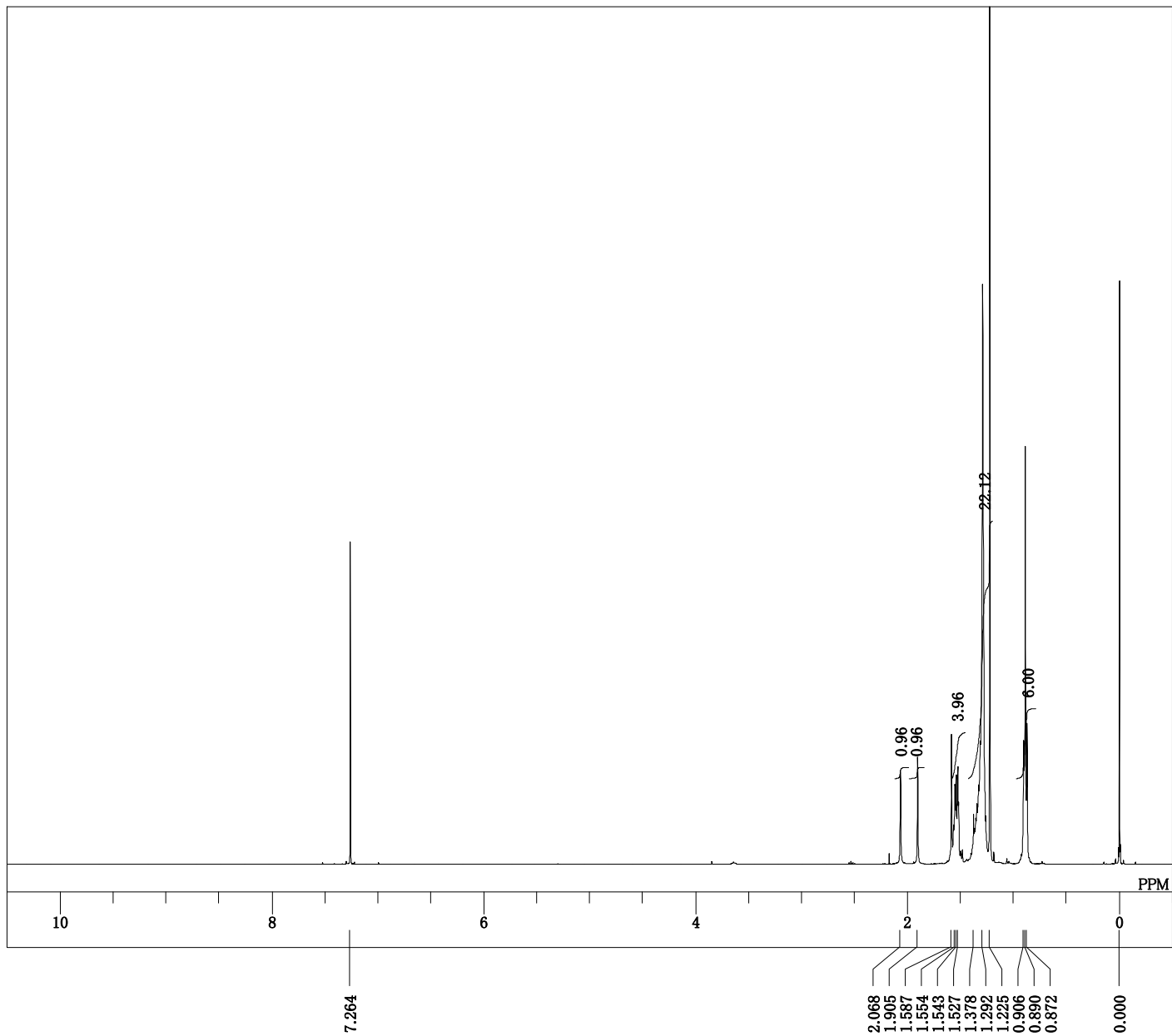


143.727

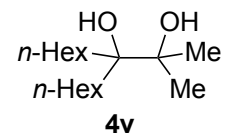
128.418
127.460
126.367

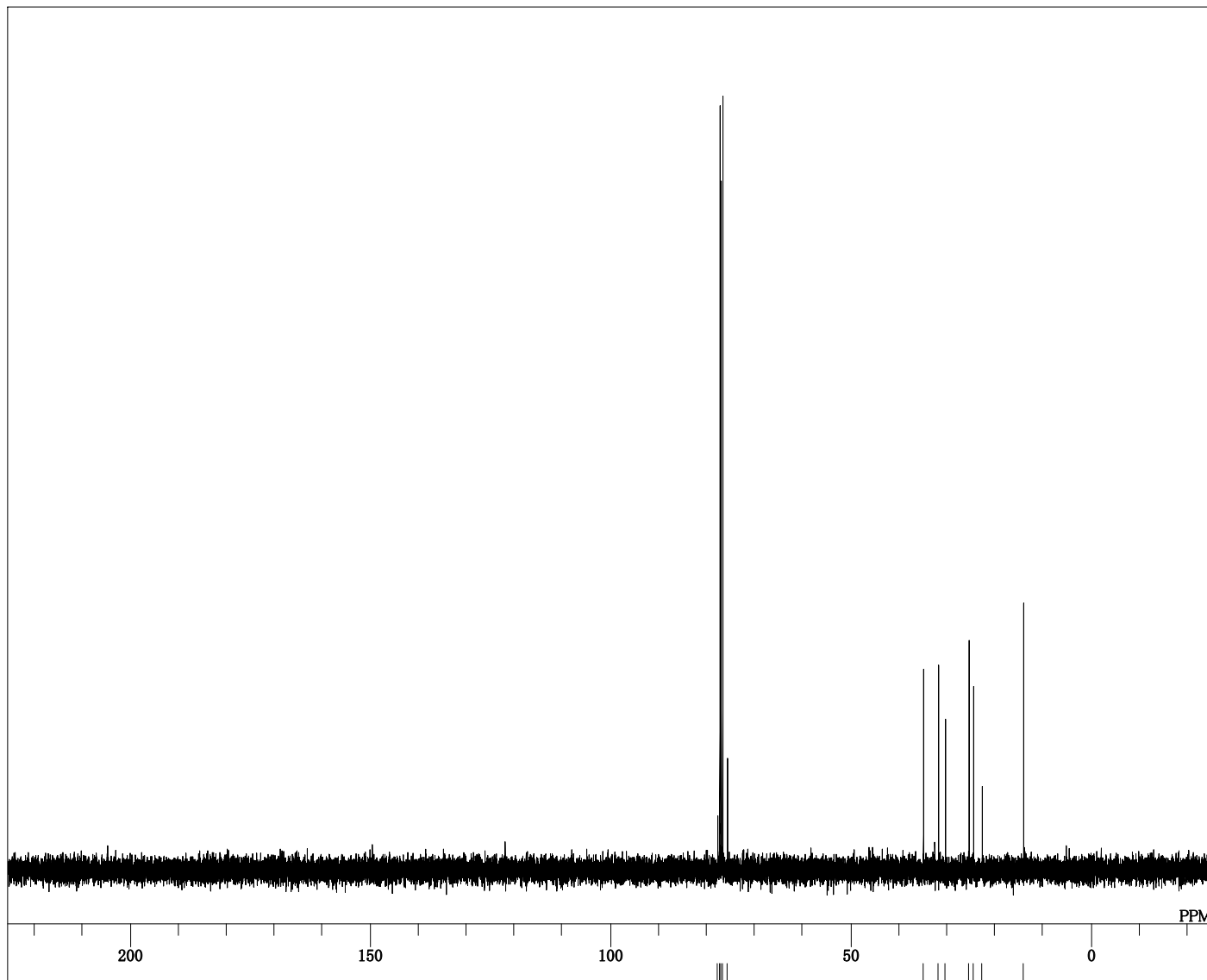
78.533
77.326
77.000
76.684
69.364



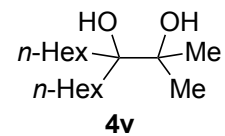


DFILE diol_Me_Me_nHex_nHex_Proton.als
 COMNT single_pulse
 DATIM 2020-07-14 14:39:12
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 34

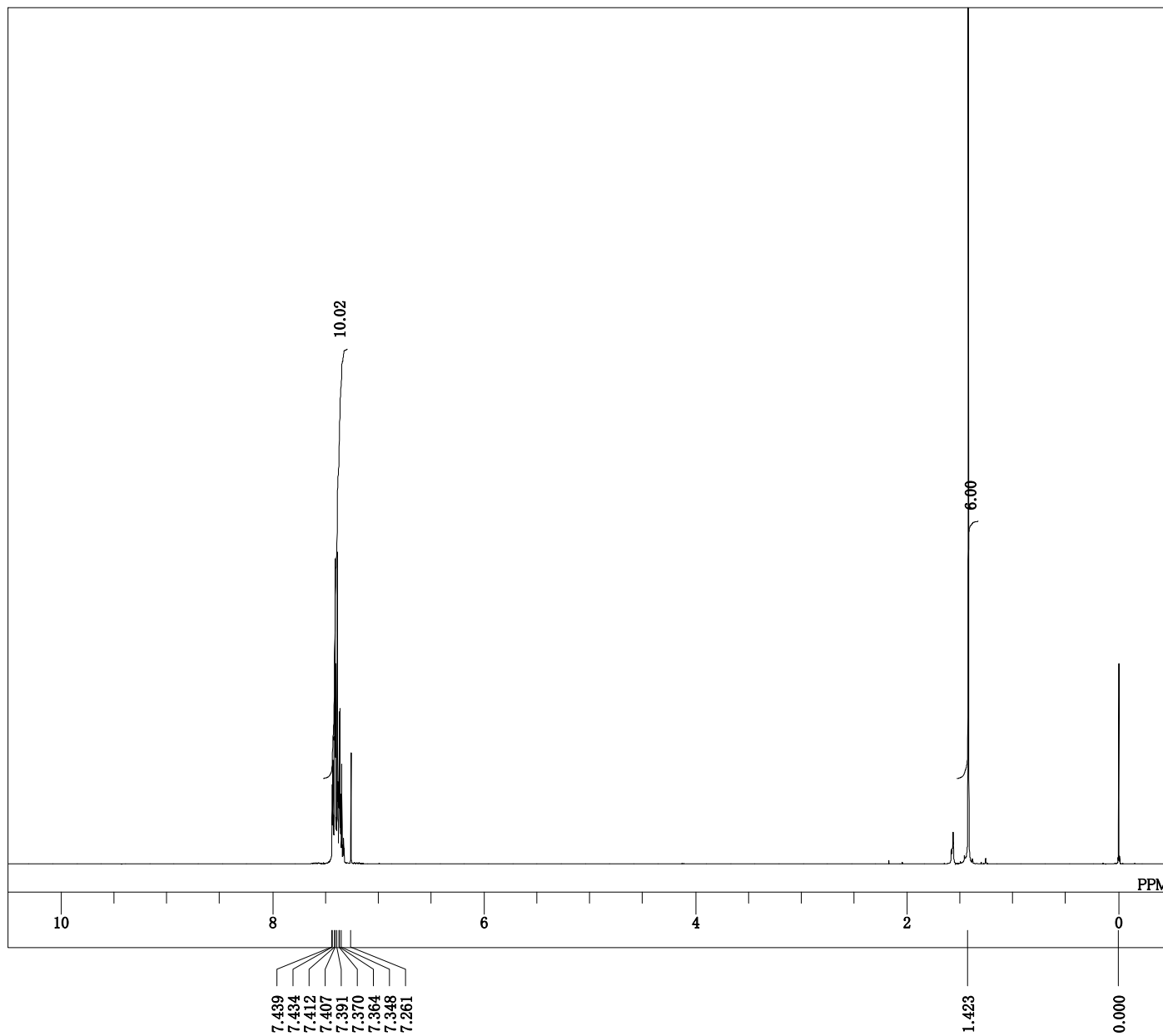




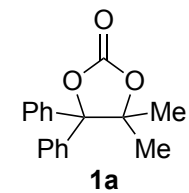
DFILE diol_Me_Me_nHex_nHex_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-07-14 14:40:25
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 150
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 20.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50

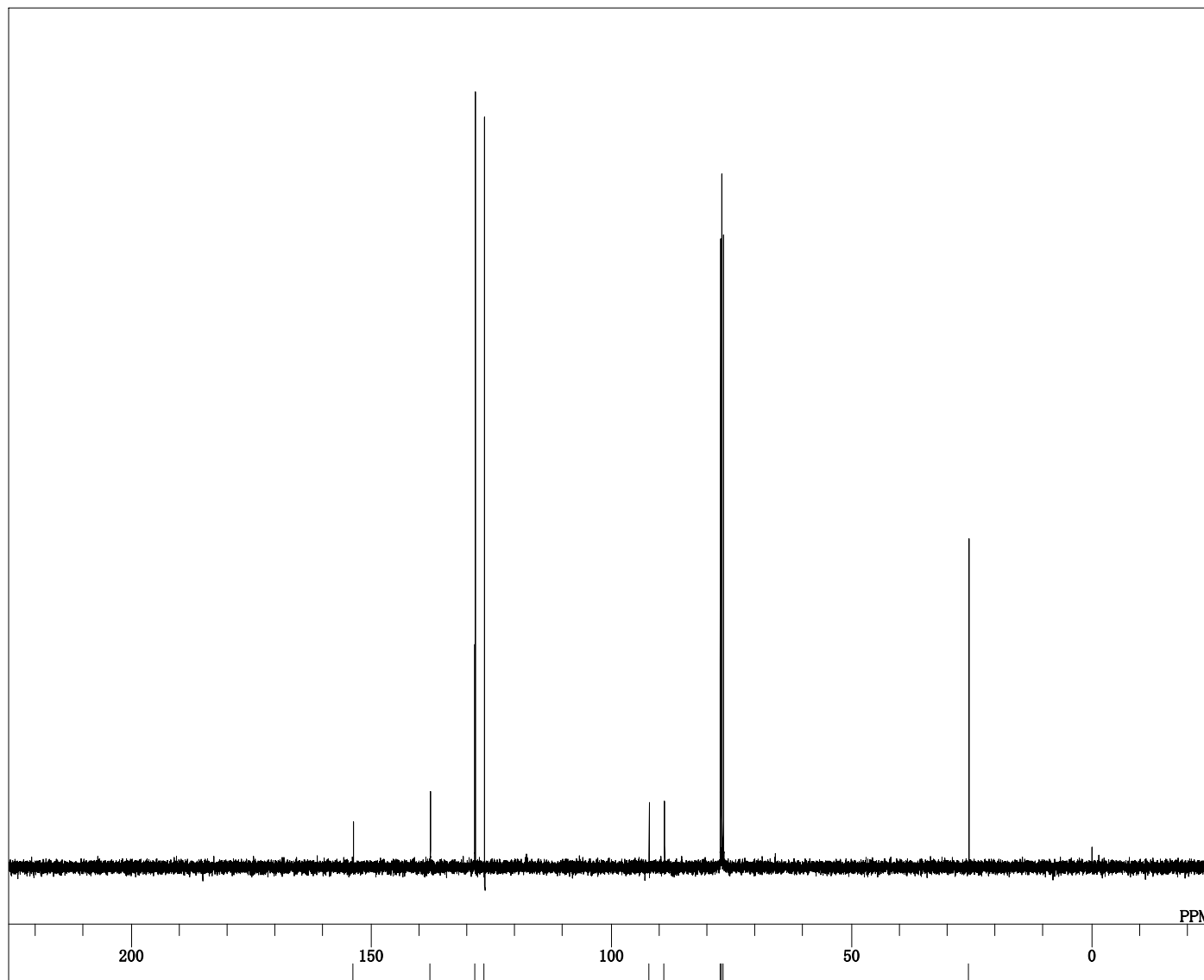


77.738
 77.316
 77.000
 76.674
 75.735
 34.951
 31.799
 30.334
 25.438
 24.499
 22.650
 14.076

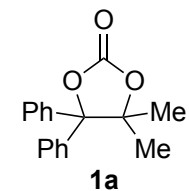


DFILE cyc_Me_Me_Proton.als
 COMNT single_pulse
 DATIM 2020-02-24 12:45:14
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OFFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 19.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 38





DFILE cyc_Me_Me_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-02-24 12:46:26
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 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



153.797

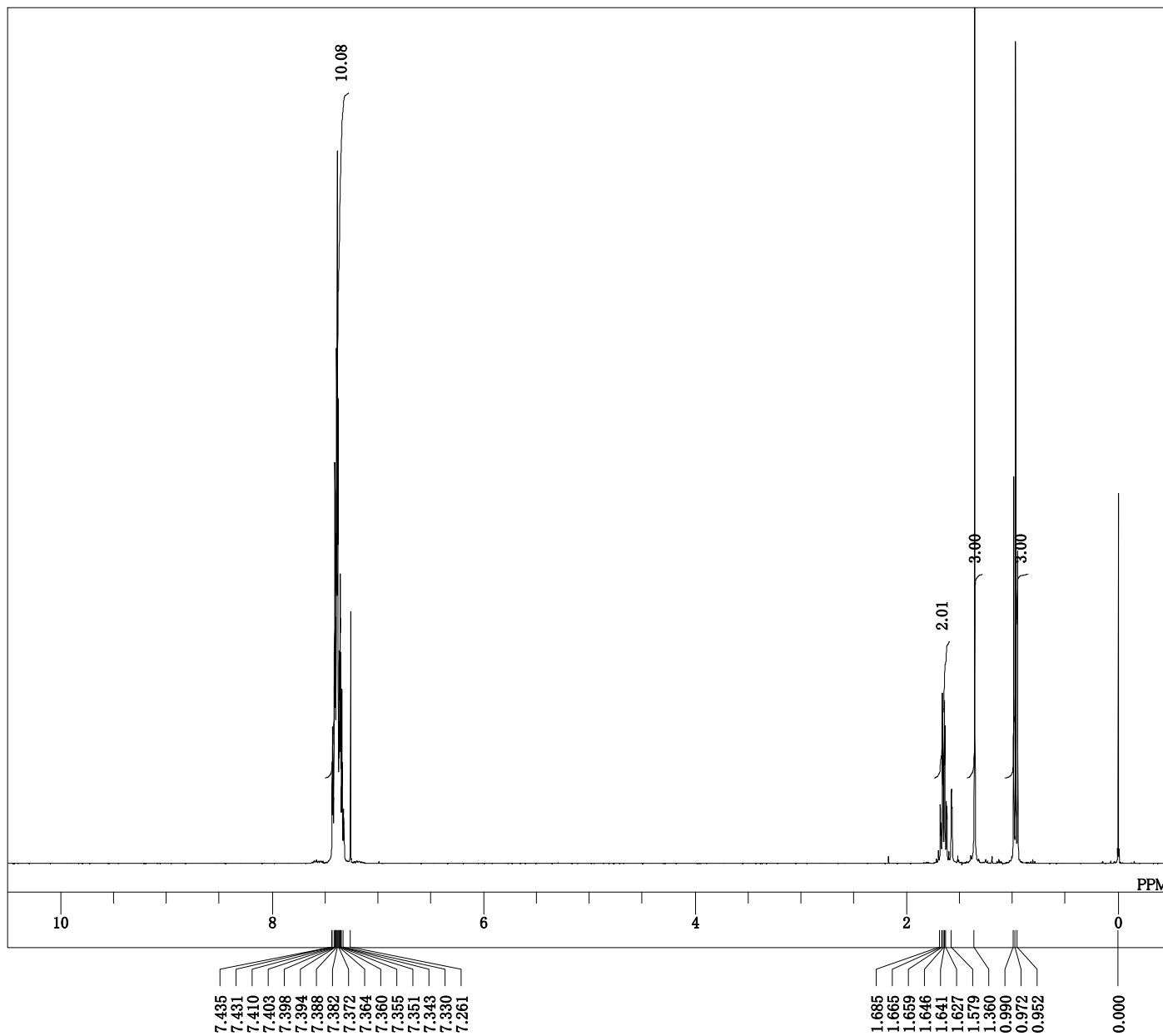
137.759

128.428
126.416

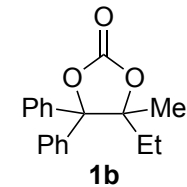
92.166
88.956

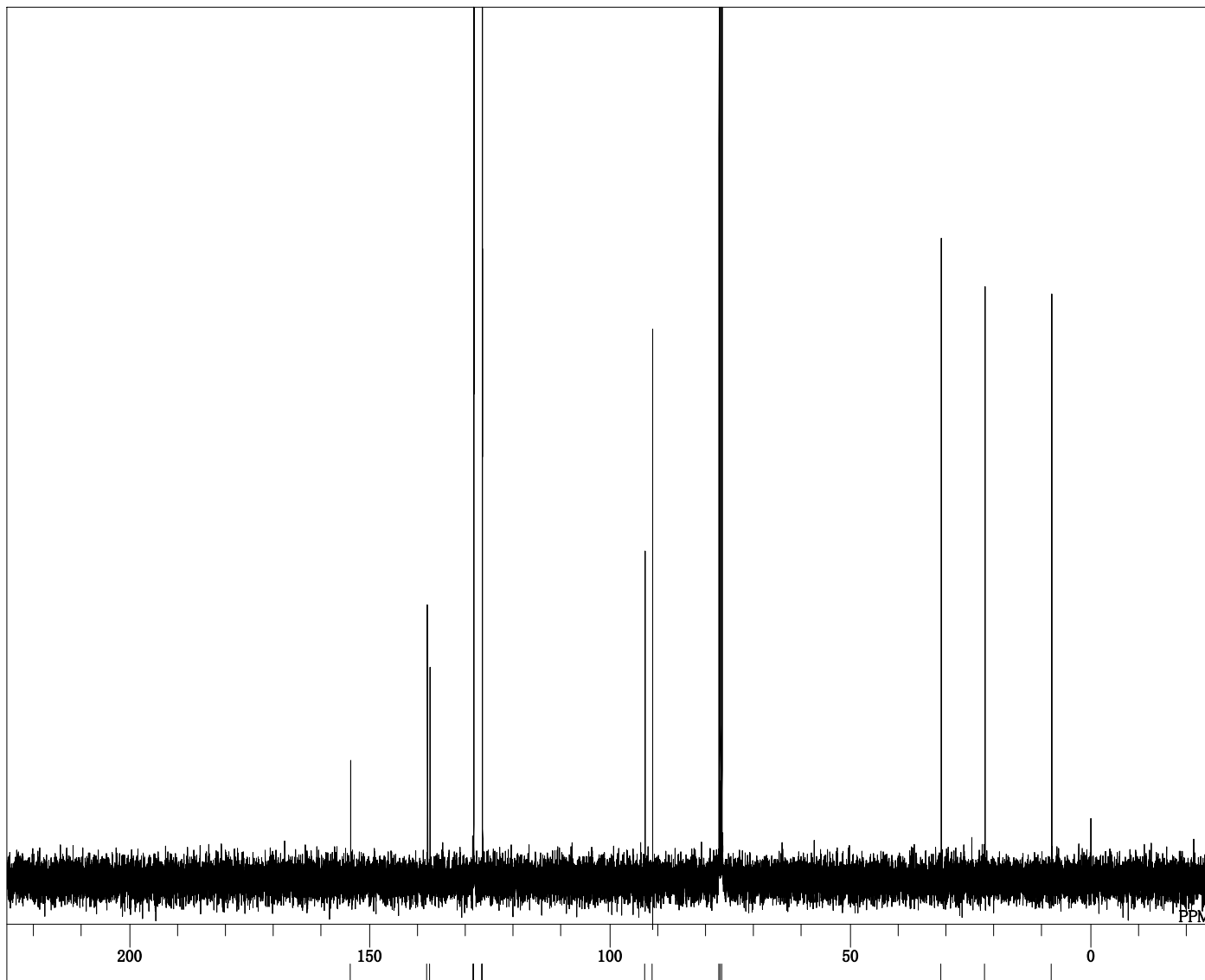
77.316
77.000
76.674

25.582



DFILE cyc_Me_Et_Proton.als
 COMNT single_pulse
 DATIM 2020-02-24 13:45:39
 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 36





153.960

138.018

137.520

128.409

128.390

128.370

126.579

126.560

92.722

91.131

77.326

77.000

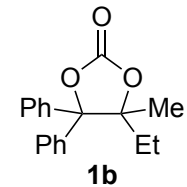
76.684

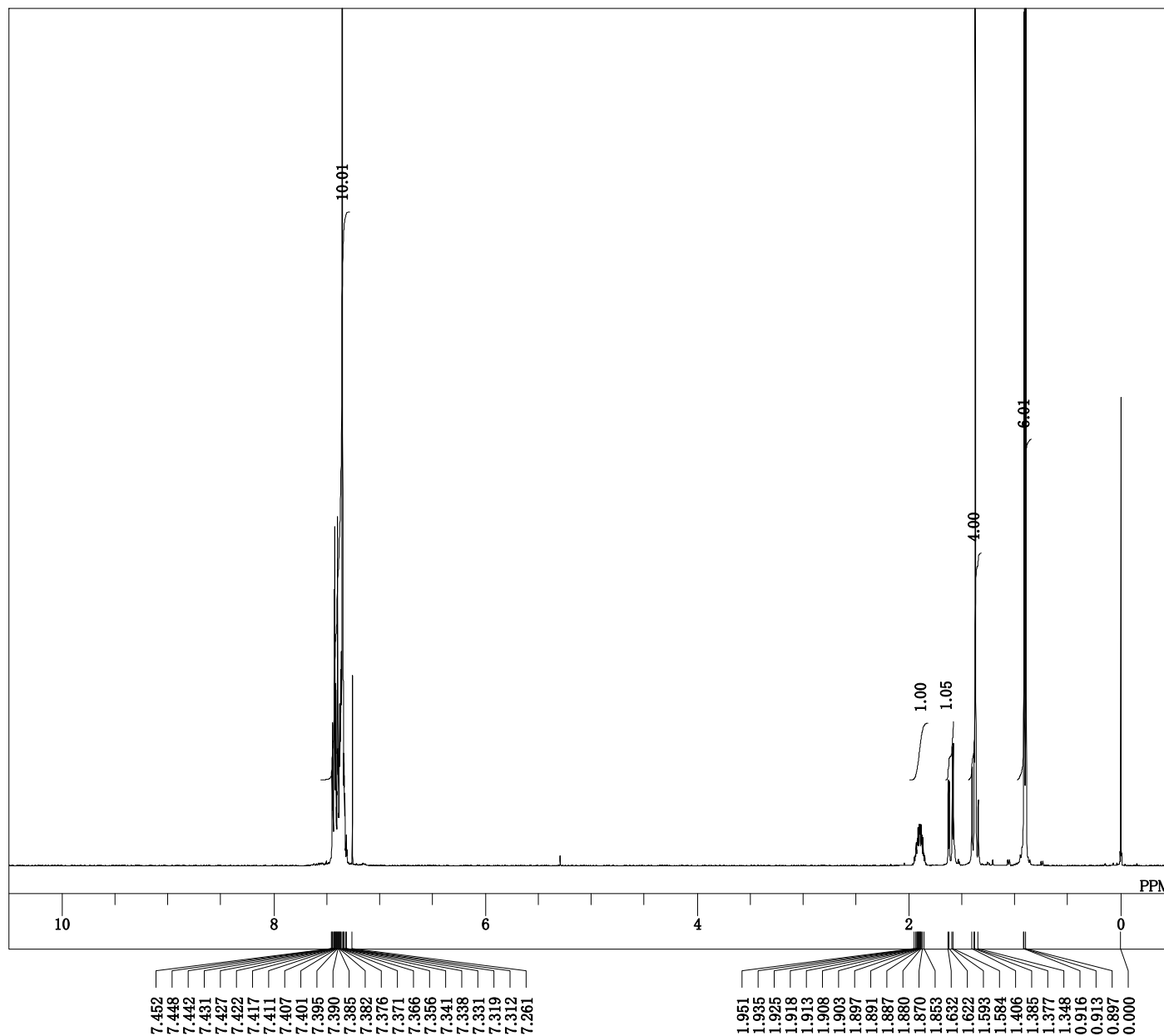
31.100

21.999

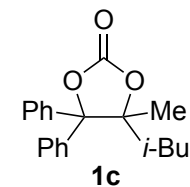
8.088

DFILE cyc_Me_Et_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-02-24 13:46:52
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 986
 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

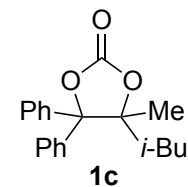
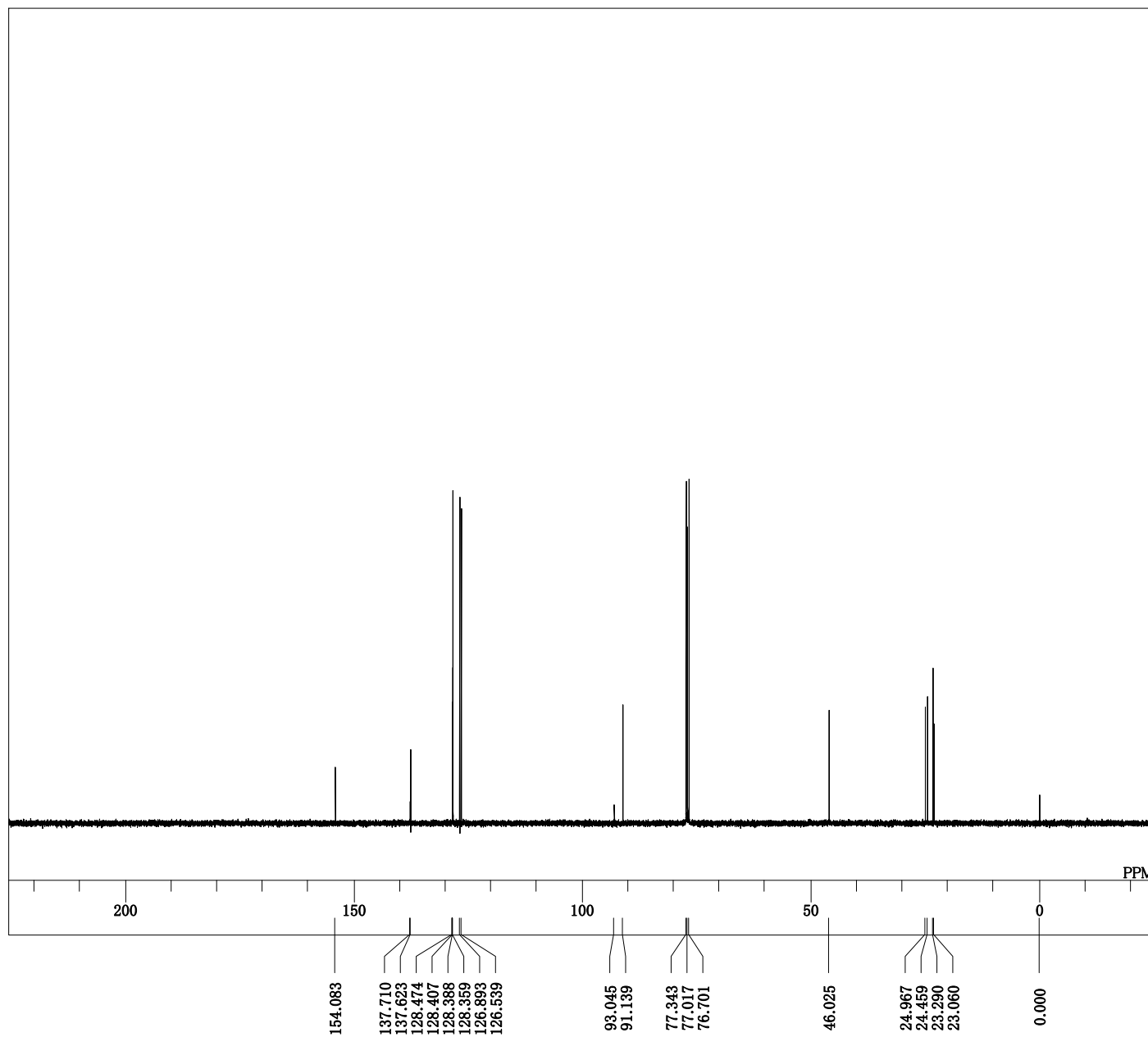


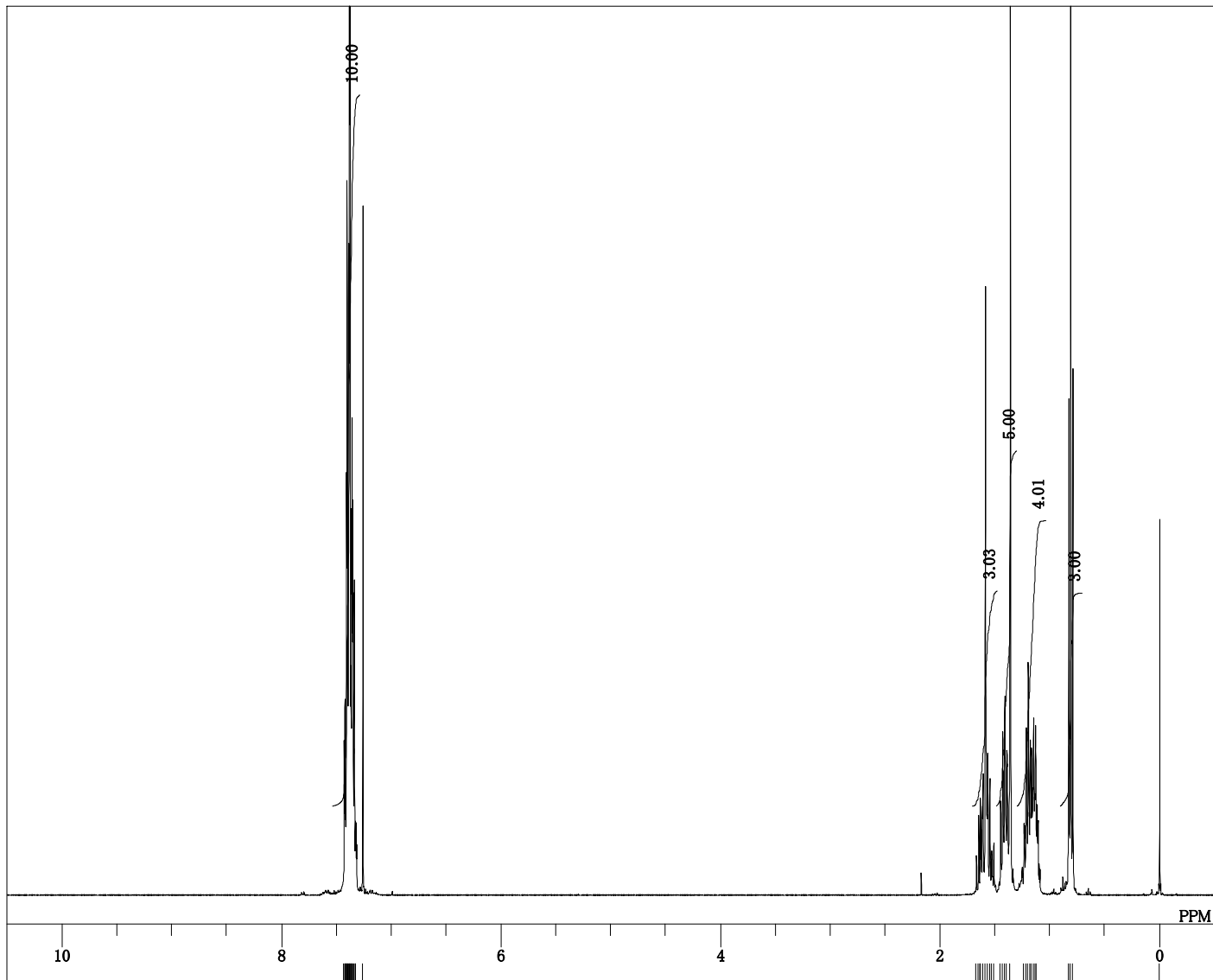


DFILE cyc_Me_iBu_Proton.als
 COMNT single_pulse
 DATIM 2020-03-26 23:25:26
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 19.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 30



DFILE cyc_Me_iBu_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-03-26 23:26:39
 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 0.00 ppm
 BF 0.12 Hz
 RGAIN 50

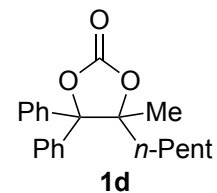




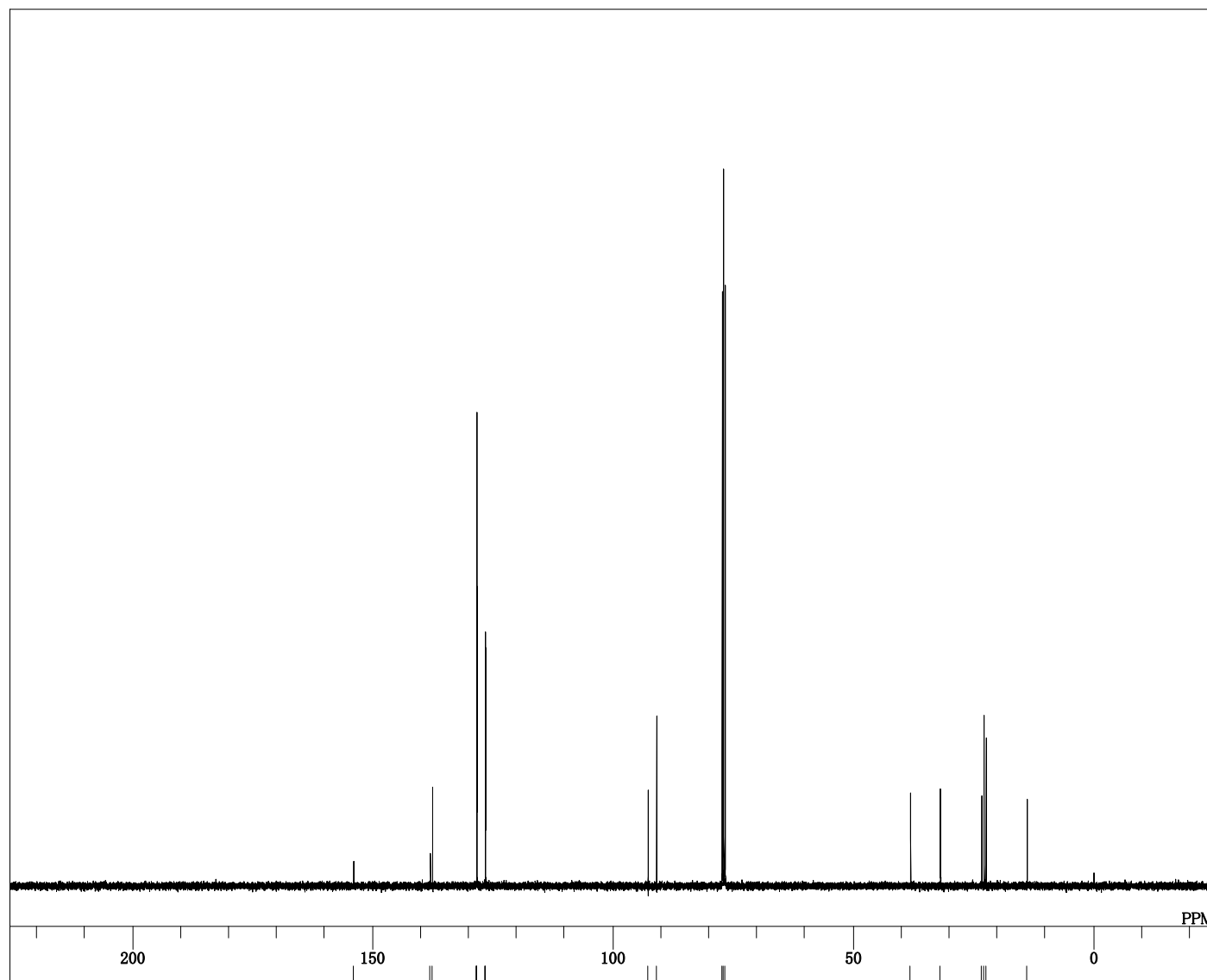
7.431
7.425
7.419
7.410
7.404
7.400
7.393
7.390
7.383
7.378
7.369
7.364
7.361
7.356
7.352
7.339
7.330
7.323
7.261

1.670
1.648
1.633
1.627
1.609
1.588
1.567
1.547
1.529
1.511
1.449
1.431
1.410
1.392
1.361
1.236
1.214
1.197
1.180
1.167
1.148
1.132
1.119
0.829
0.810
0.792
0.000

DFILE cyc_Me_nPentan_Proton.als
COMNT single_pulse
DATIM 2020-07-05 17:52:37
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 20.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 34



DFILE cyc_PhPhMenPentyl_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2022-04-26 06:37:06
 OBNUC ¹³C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 1650
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.67 usec
 IRNUC ¹H
 CTEMP 20.2 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



153.987

138.017

137.548

128.398

128.379

128.360

126.588

126.559

92.760

90.968

77.316

77.000

76.674

38.085

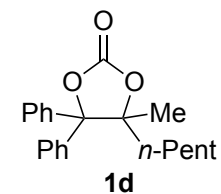
31.867

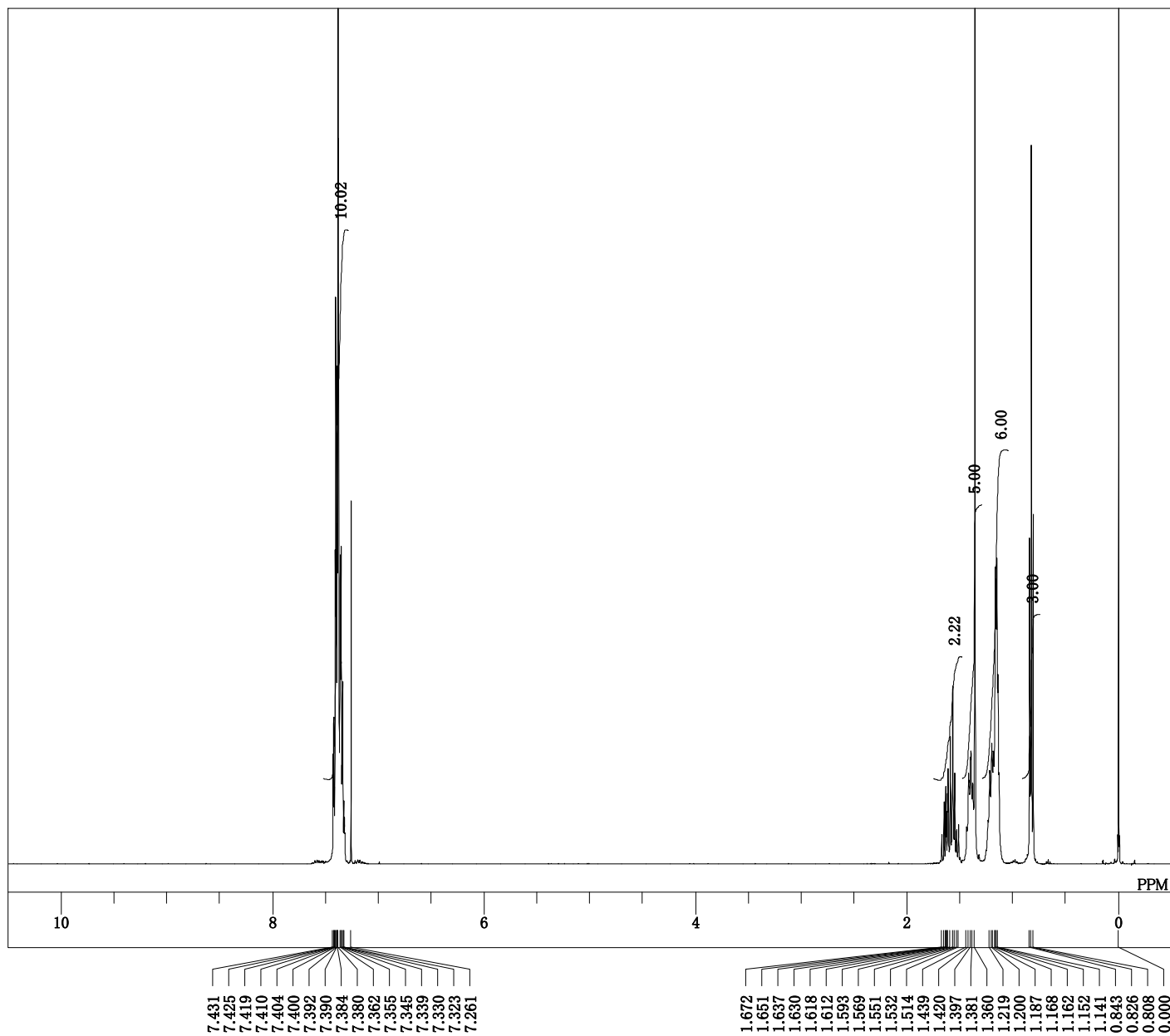
23.293

22.756

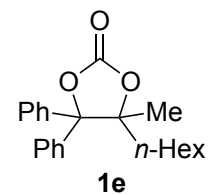
22.383

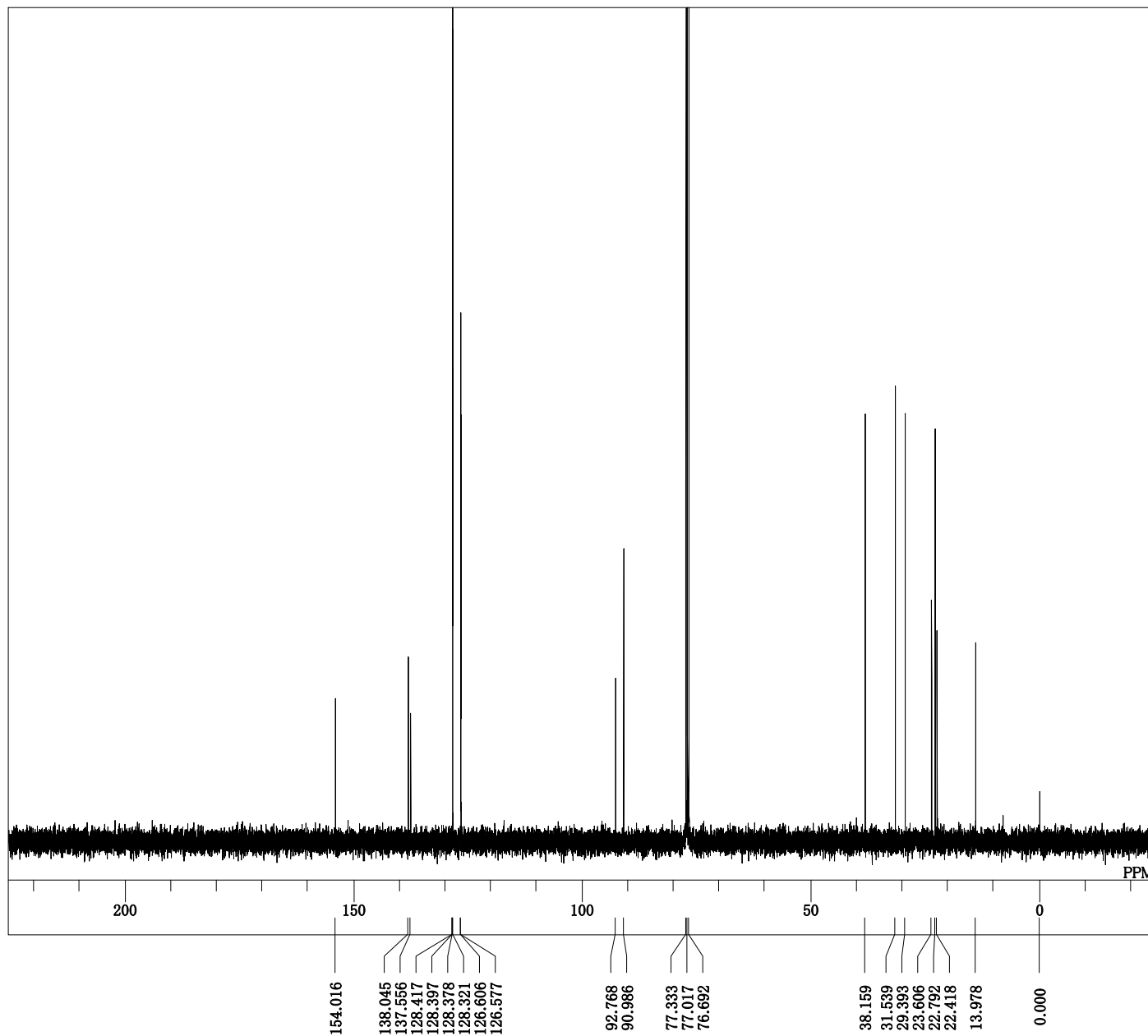
13.866



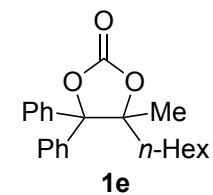


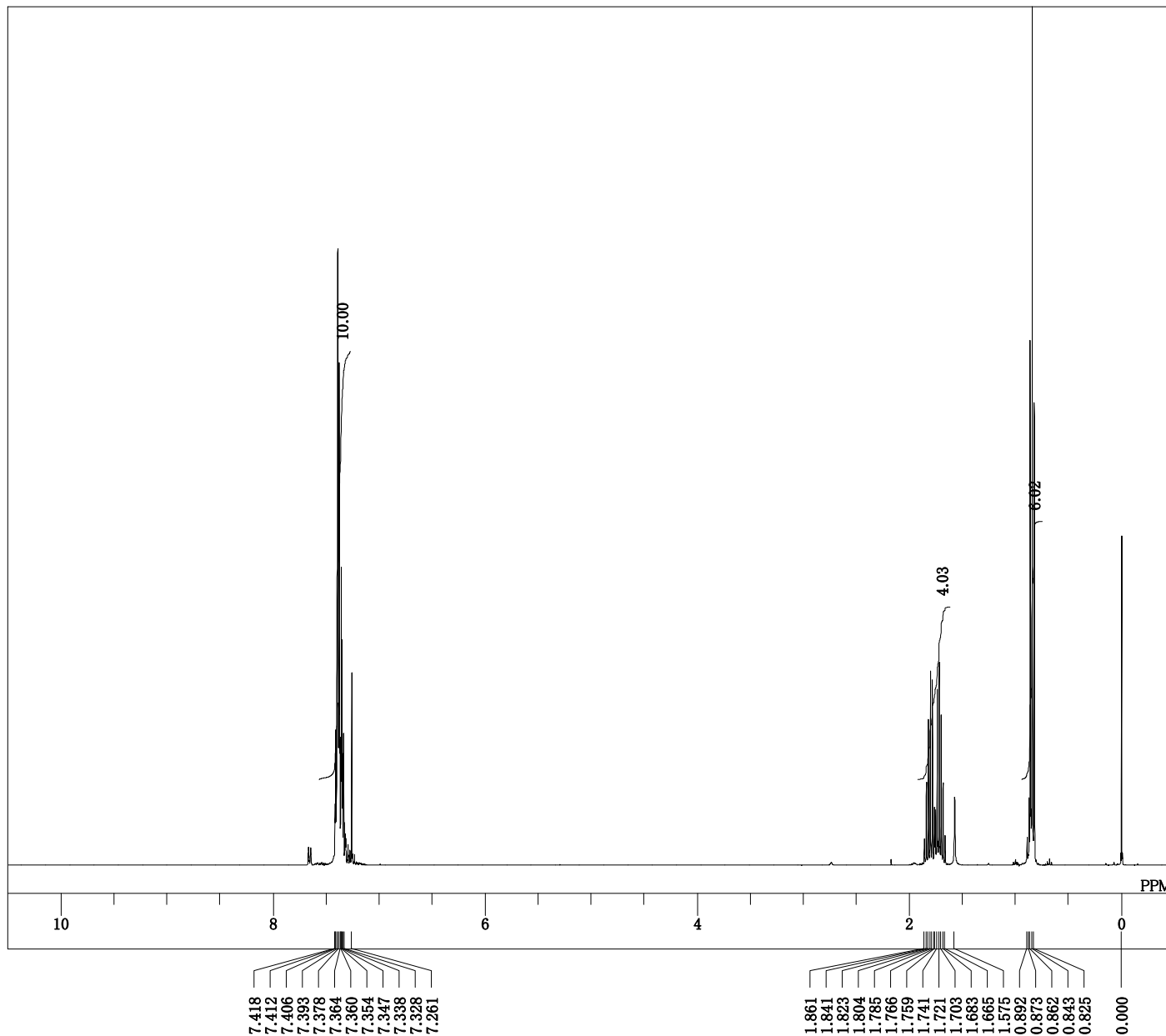
DFILE cyc_Me_nHex_Proton.als
 COMNT single_pulse
 DATIM 2020-03-27 00:47:07
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 19.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 34



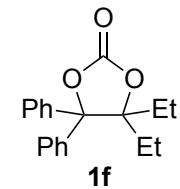


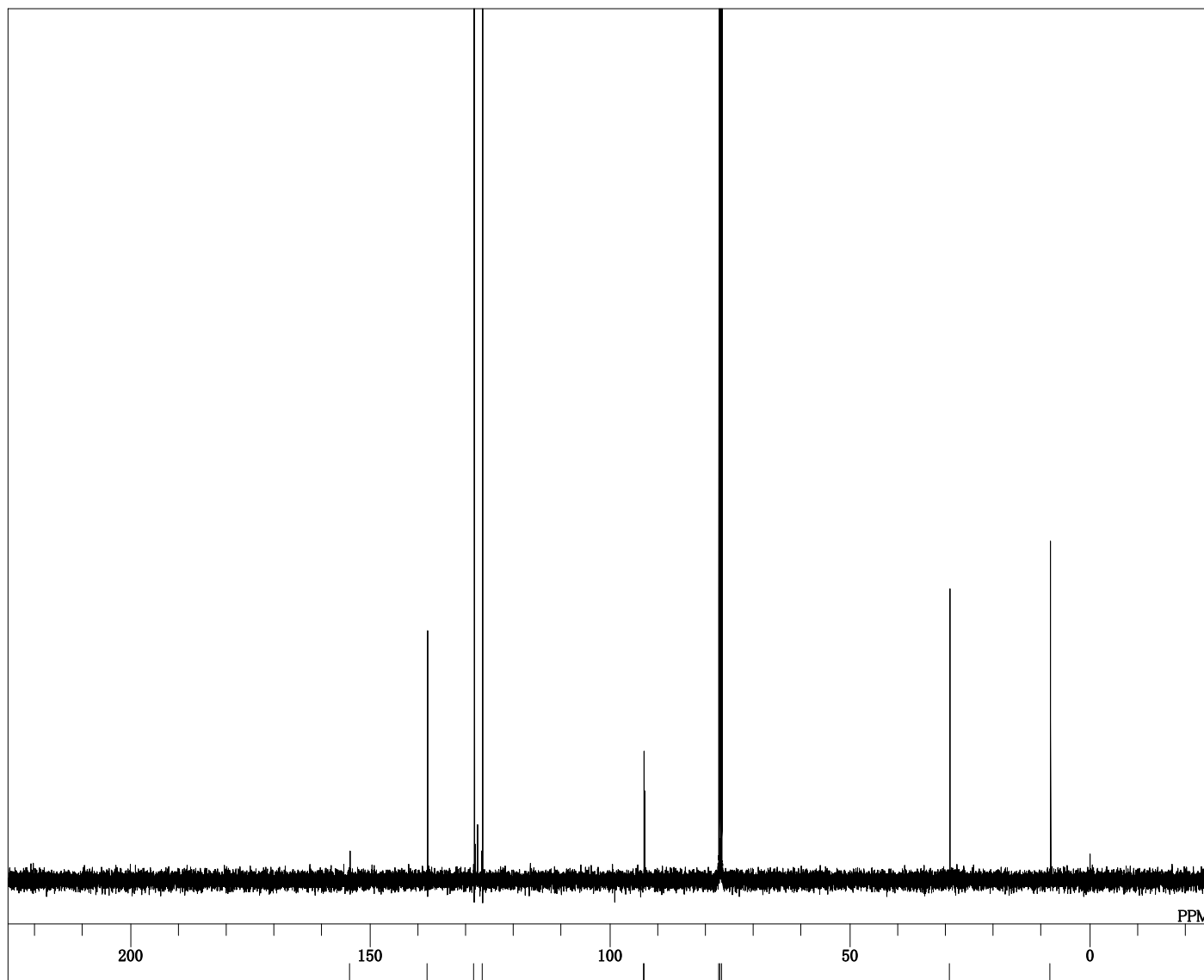
DFILE cyc_Me_nHex_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-03-27 00:48:19
 OBNUC ¹³C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 1024
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC ¹H
 CTEMP 19.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50





DFILE cyc_Et_Et_Proton.als
 COMNT single_pulse
 DATIM 2020-02-24 14:42:55
 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 36





154.257

138.056

128.332
126.598

92.971
92.865

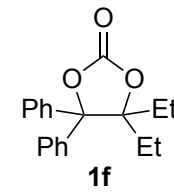
77.316
77.000
76.674

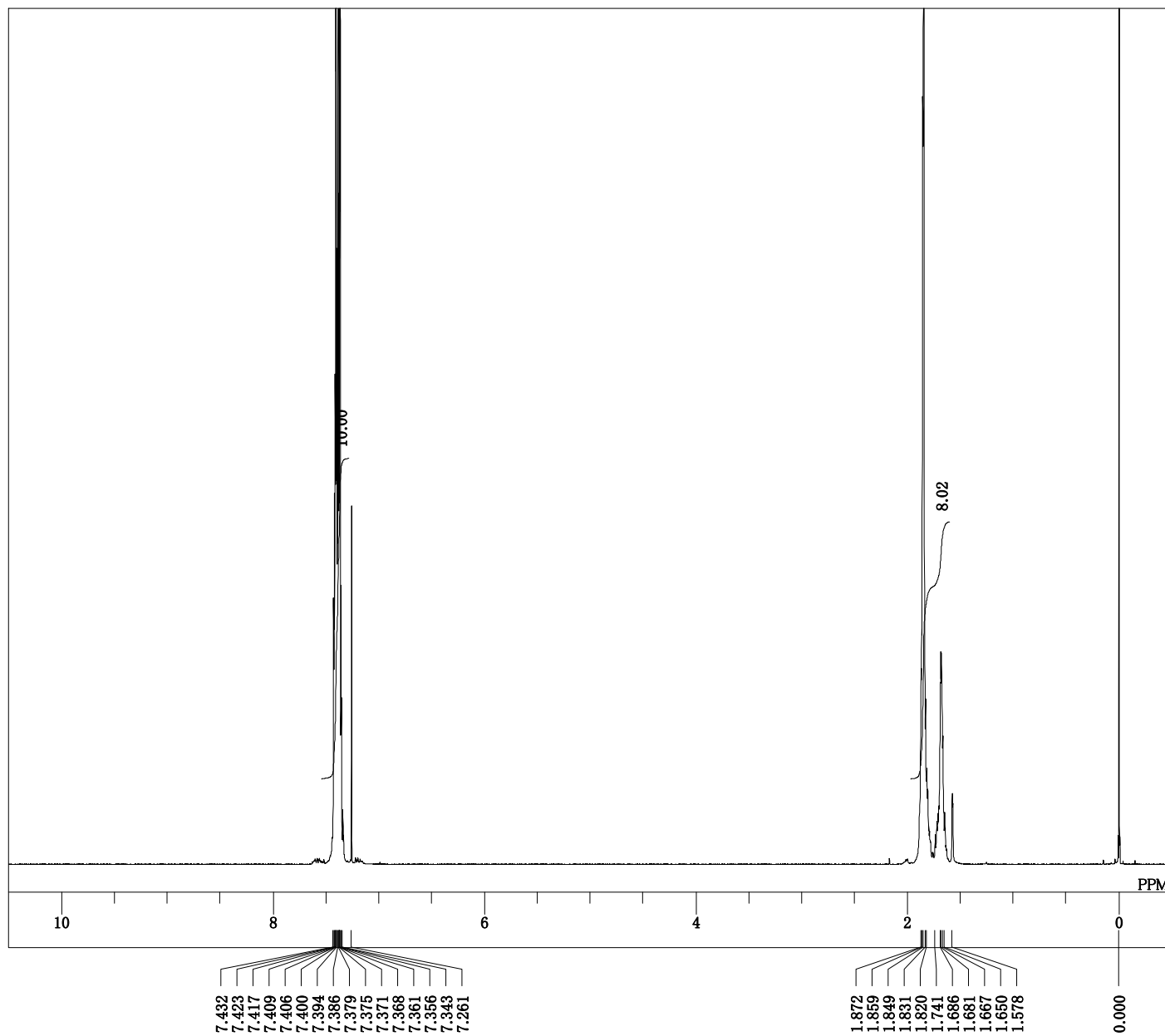
29.155

8.155

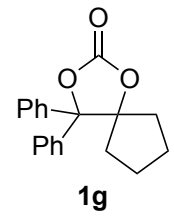
PPM

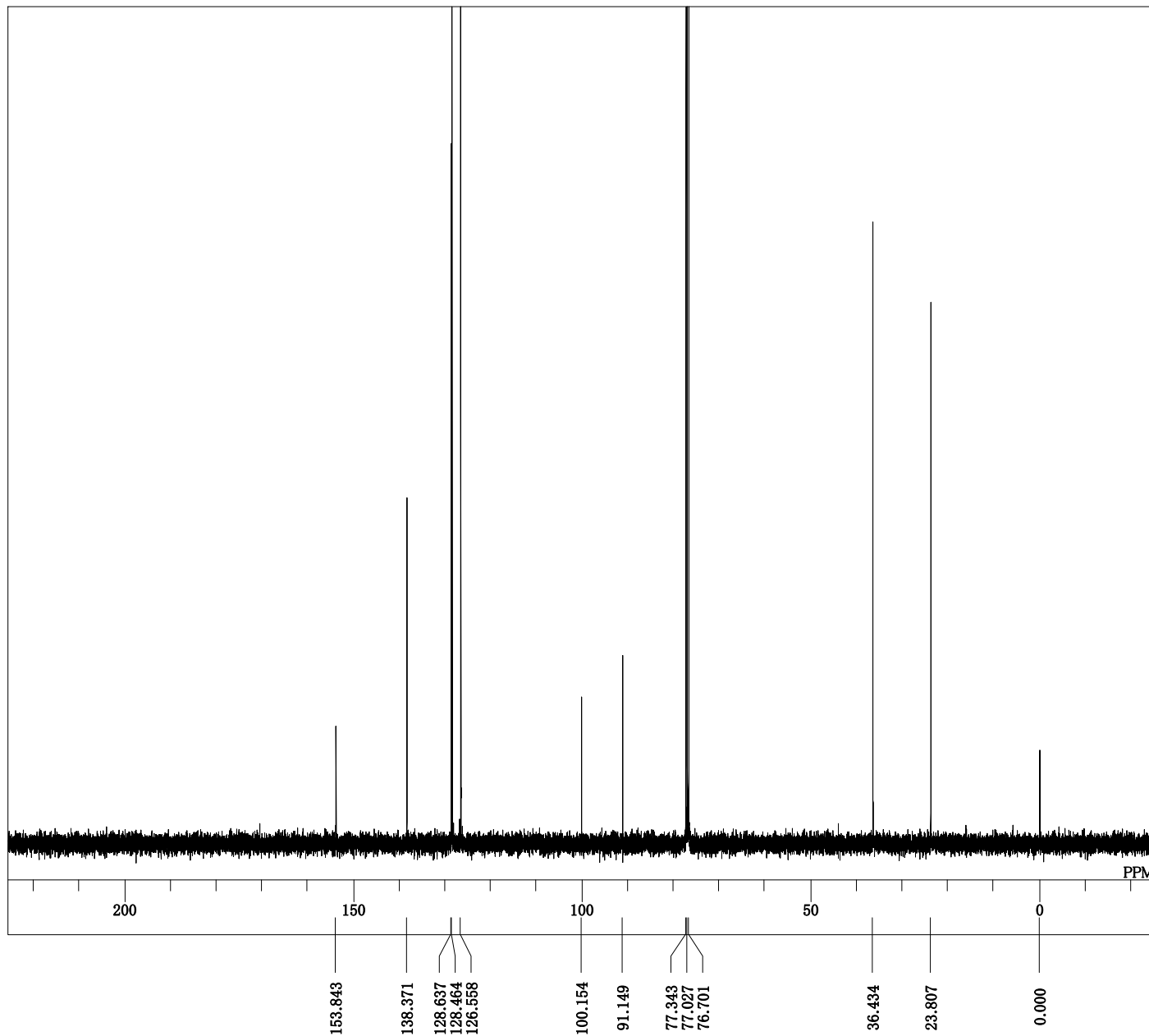
DFILE cyc_Et_Et_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-02-24 14:44:08
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 1024
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 19.6 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



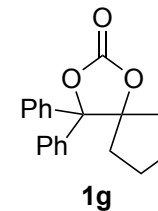


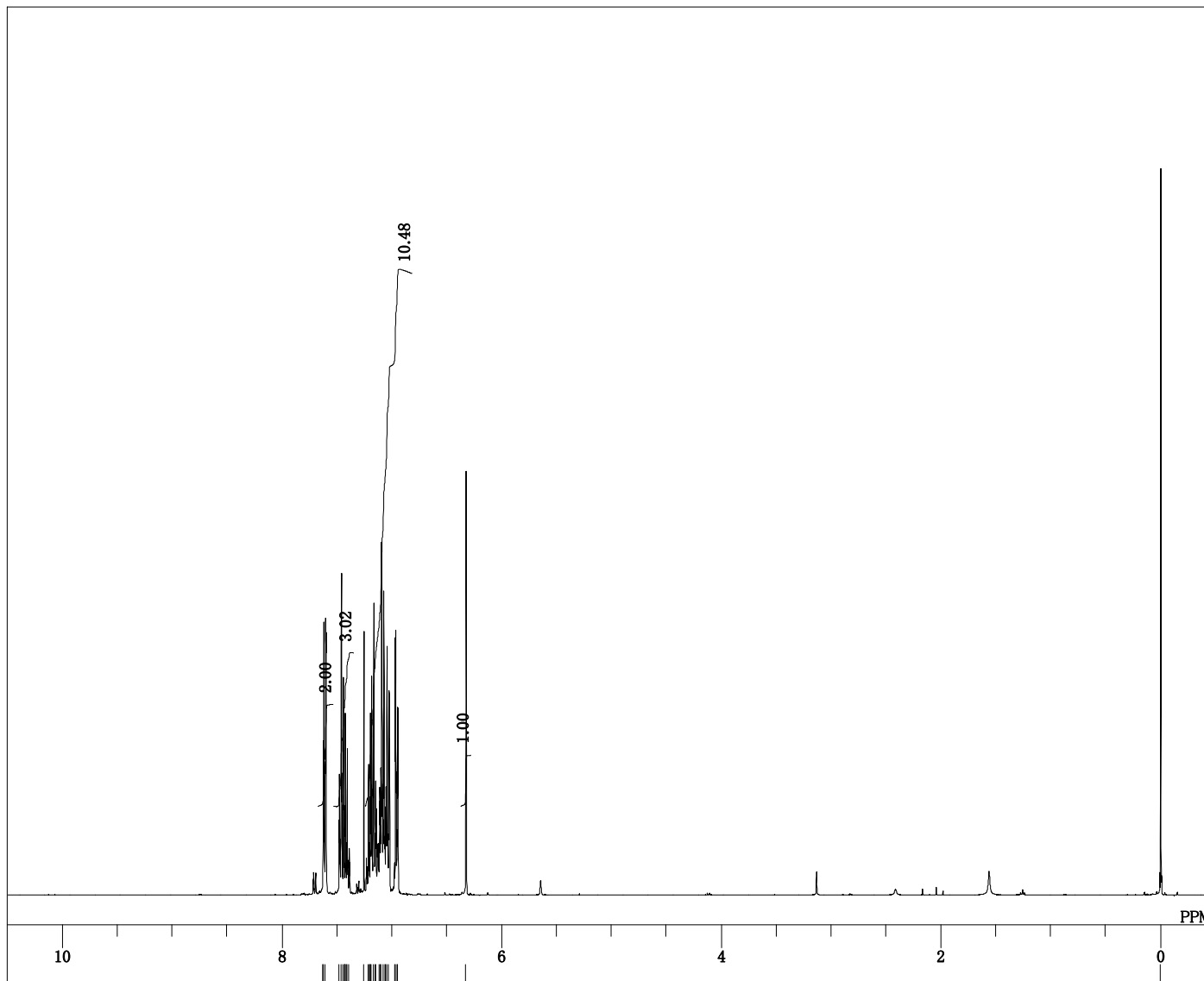
DFILE cyc_cyc_pentyl_Proton.als
 COMNT single_pulse
 DATIM 2020-03-27 04:44:33
 OBNUC 1H
 EXMOD proton_jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 19.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 34



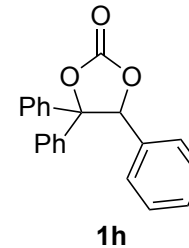


DFILE cyc_cyc_pentyl_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-03-27 04:45:46
 OBNUC ¹³C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 1024
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC ¹H
 CTEMP 19.9 c
 SLVNT CDCL₃
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50

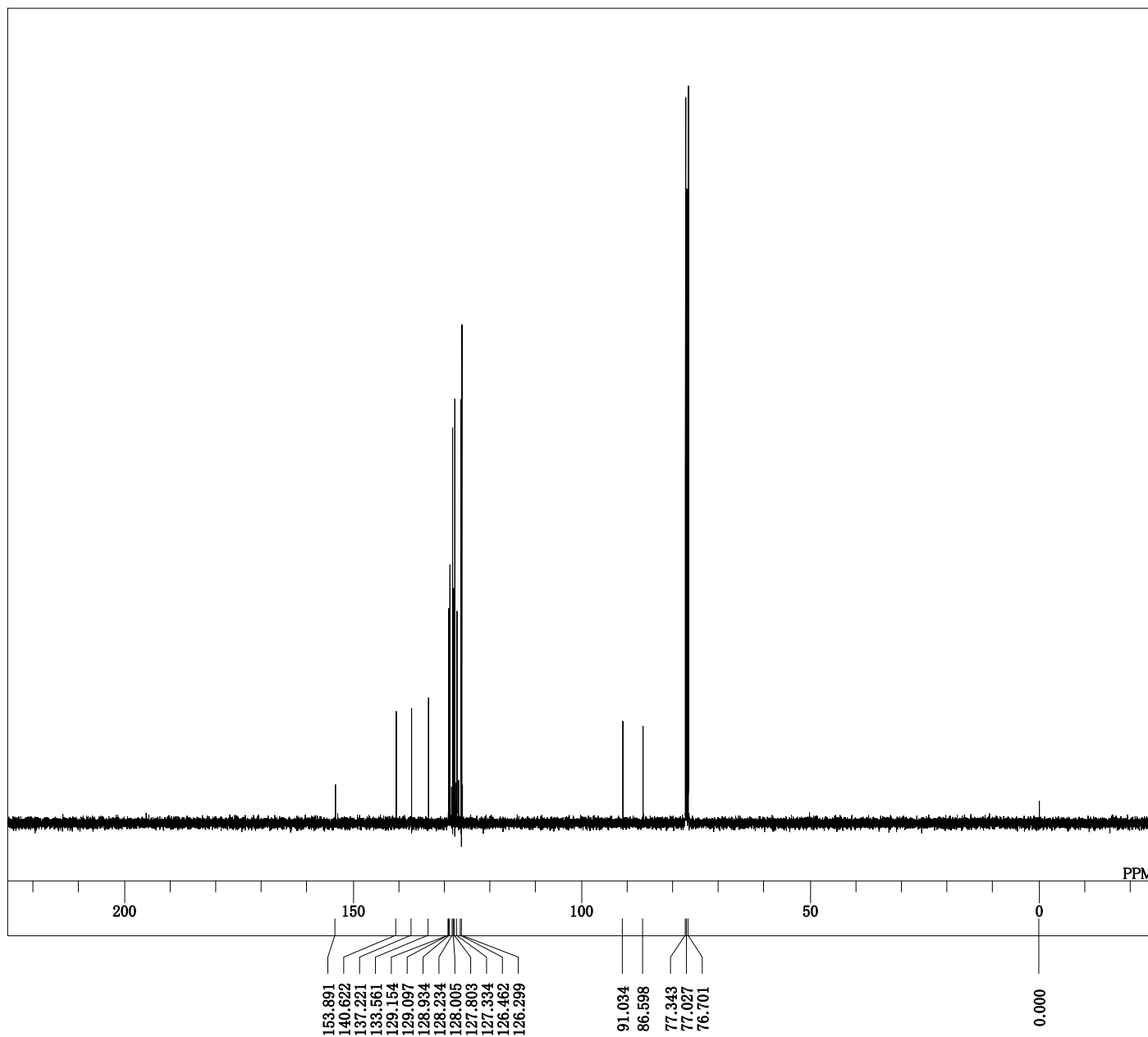




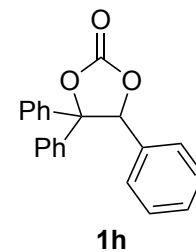
DFILE cyc_H_Ph_Proton.als
 COMNT single_pulse
 DATIM 2020-03-27 06:43:32
 OBNUC 1H
 EXMOD proton_jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 19.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 36

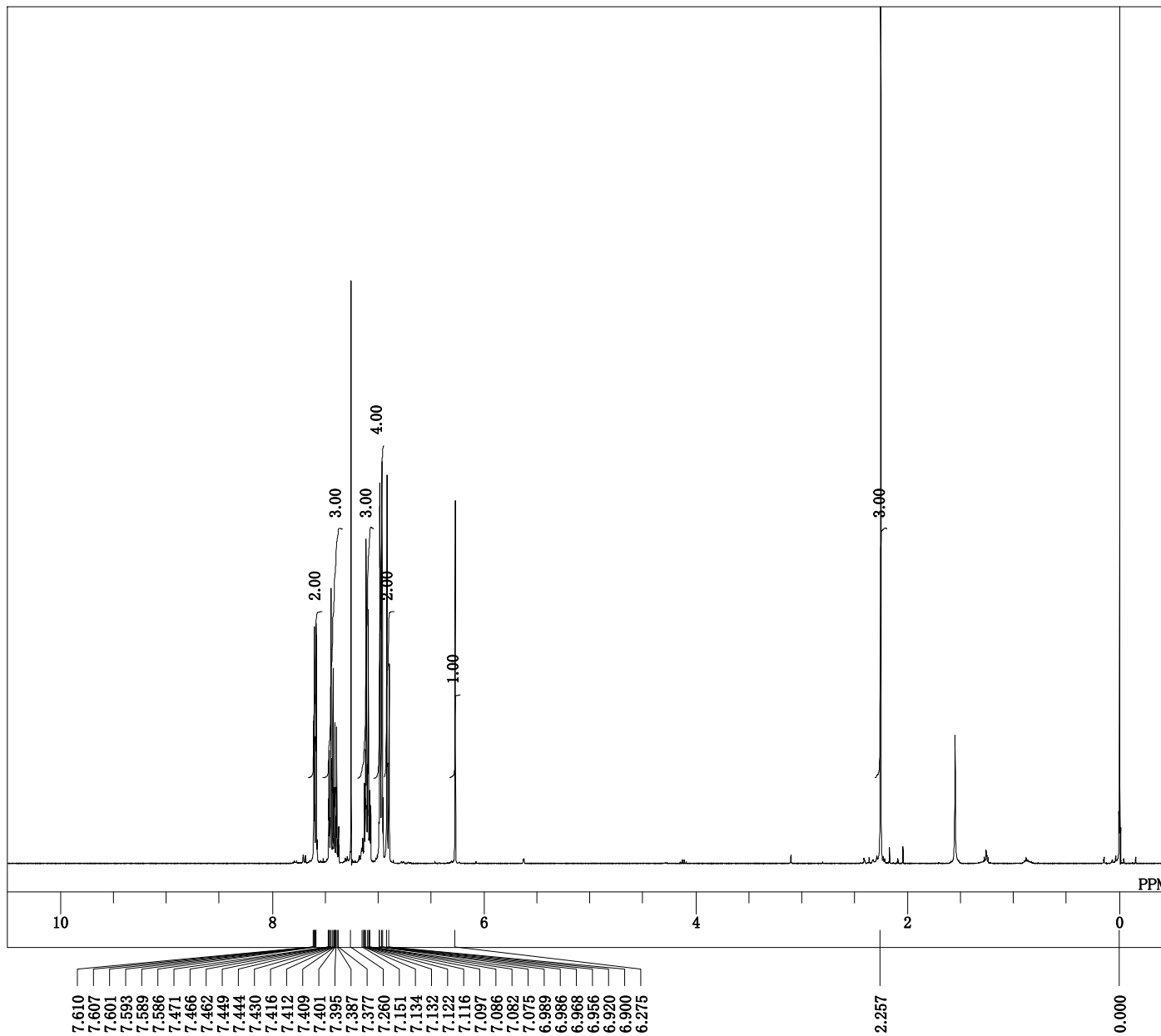


7.626
 7.622
 7.605
 7.479
 7.462
 7.442
 7.430
 7.426
 7.423
 7.415
 7.408
 7.390
 7.257
 7.214
 7.201
 7.197
 7.193
 7.186
 7.166
 7.149
 7.145
 7.111
 7.101
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 7.060
 7.053
 7.045
 7.028
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 6.967
 6.951
 6.947
 6.326

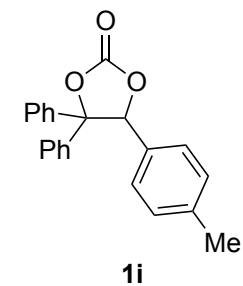


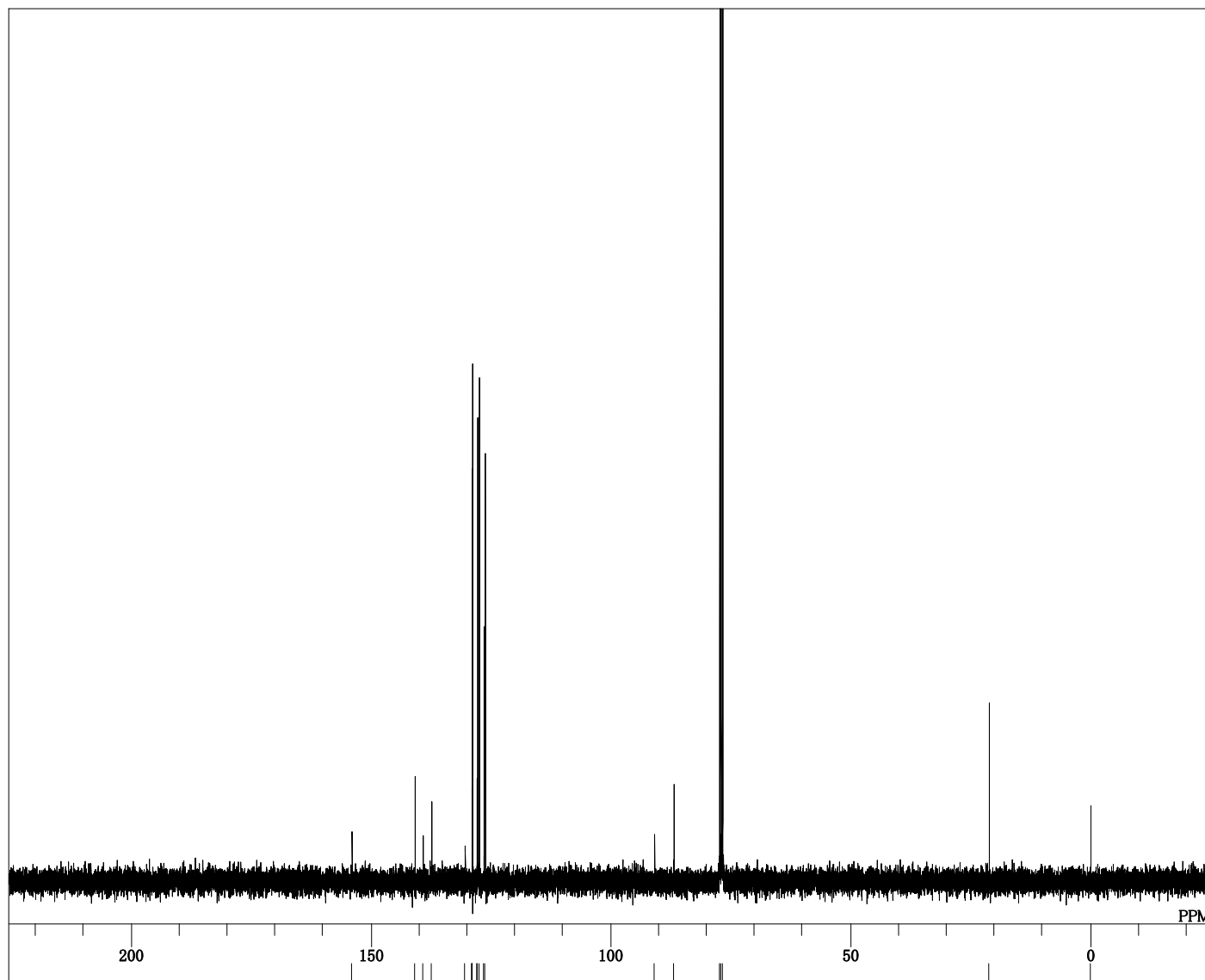
DFILE cyc_H_Ph_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-03-27 06:44:45
 OBNUC ¹³C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 1024
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC ¹H
 CTEMP 19.3 c
 SLVNT CDCL₃
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50





DFILE cyc_H_pMe_Proton.als
 COMNT single pulse
 DATIM 2020-03-27 07:42:07
 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 19.3 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 42





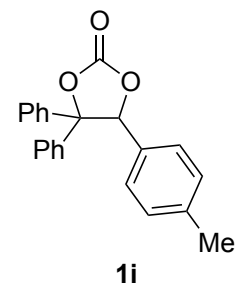
153.958
140.833
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137.345
130.390
128.962
128.915
128.886
127.966
127.803
127.498
126.472
126.184

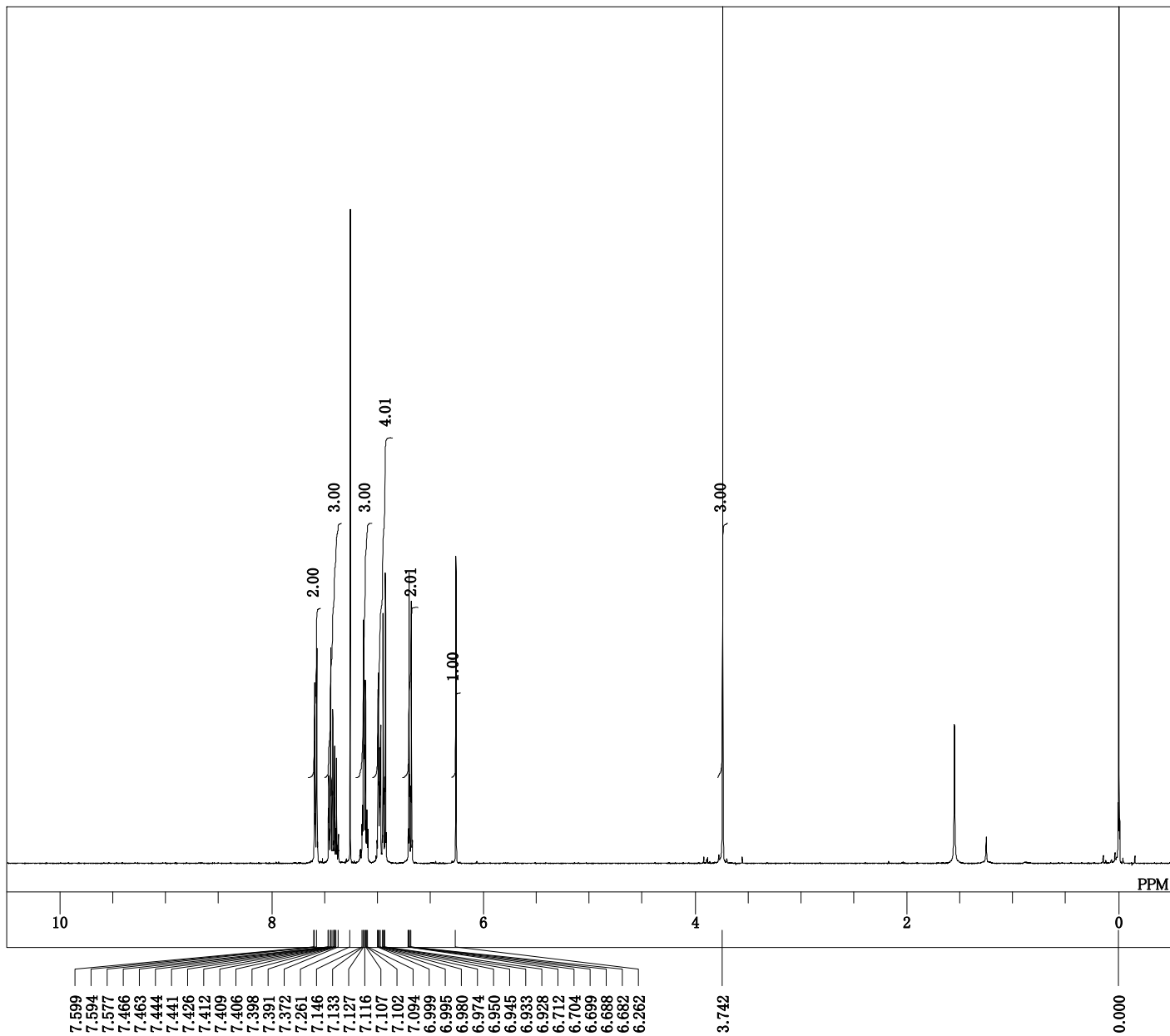
90.880
86.876
77.333
77.008
76.692

21.173

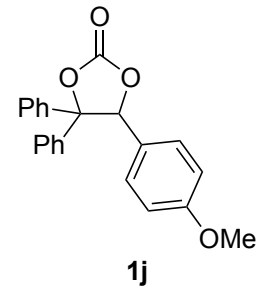
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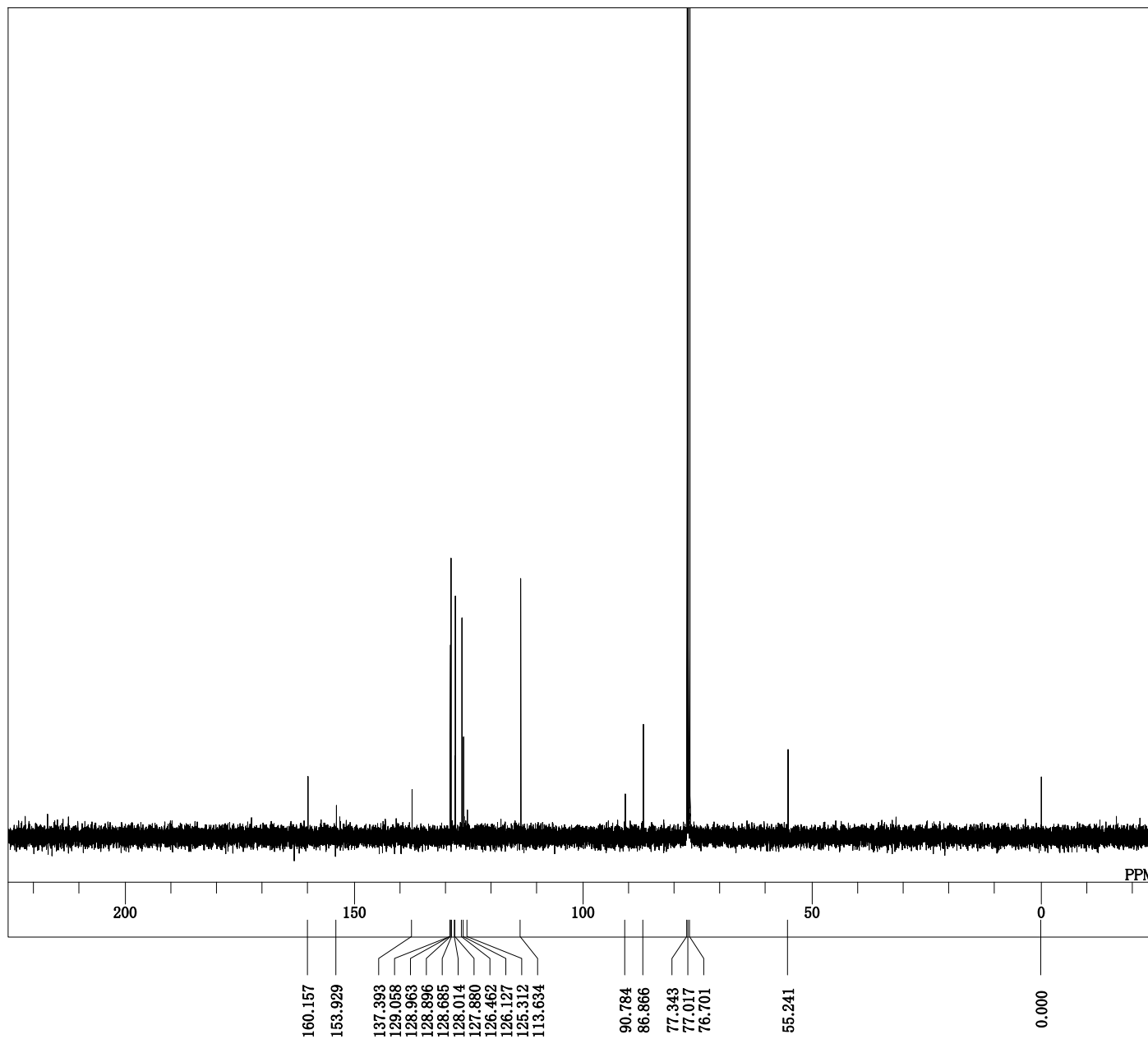
DFILE cyc_H_pMe_Carbon.als
COMNT single pulse decoupled gated NOE
DATIM 2020-03-27 07:43:19
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 25000.00 Hz
SCANS 1024
ACQTM 1.0486 sec
PD 2.0000 sec
PW1 3.59 usec
IRNUC 1H
CTEMP 19.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50



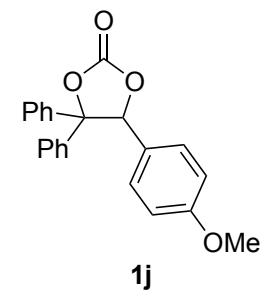


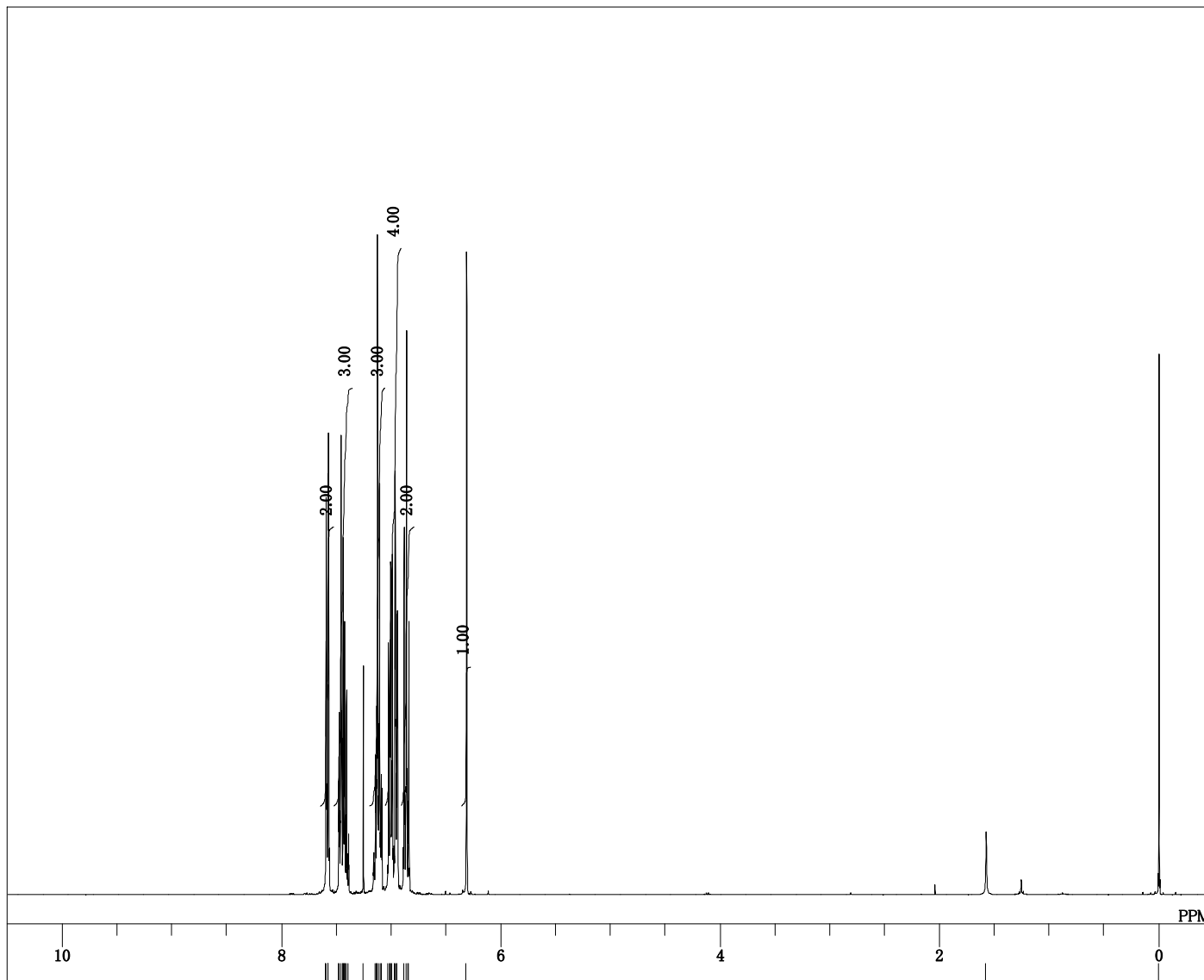
DFILE cyc_HpOMe_Proton.als
 COMNT single_pulse
 DATIM 2020-03-27 09:13:10
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 19.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 44



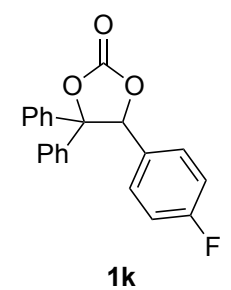


DFILE cyc_HpOMe_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-03-27 09:14:23
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 1024
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 19.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50





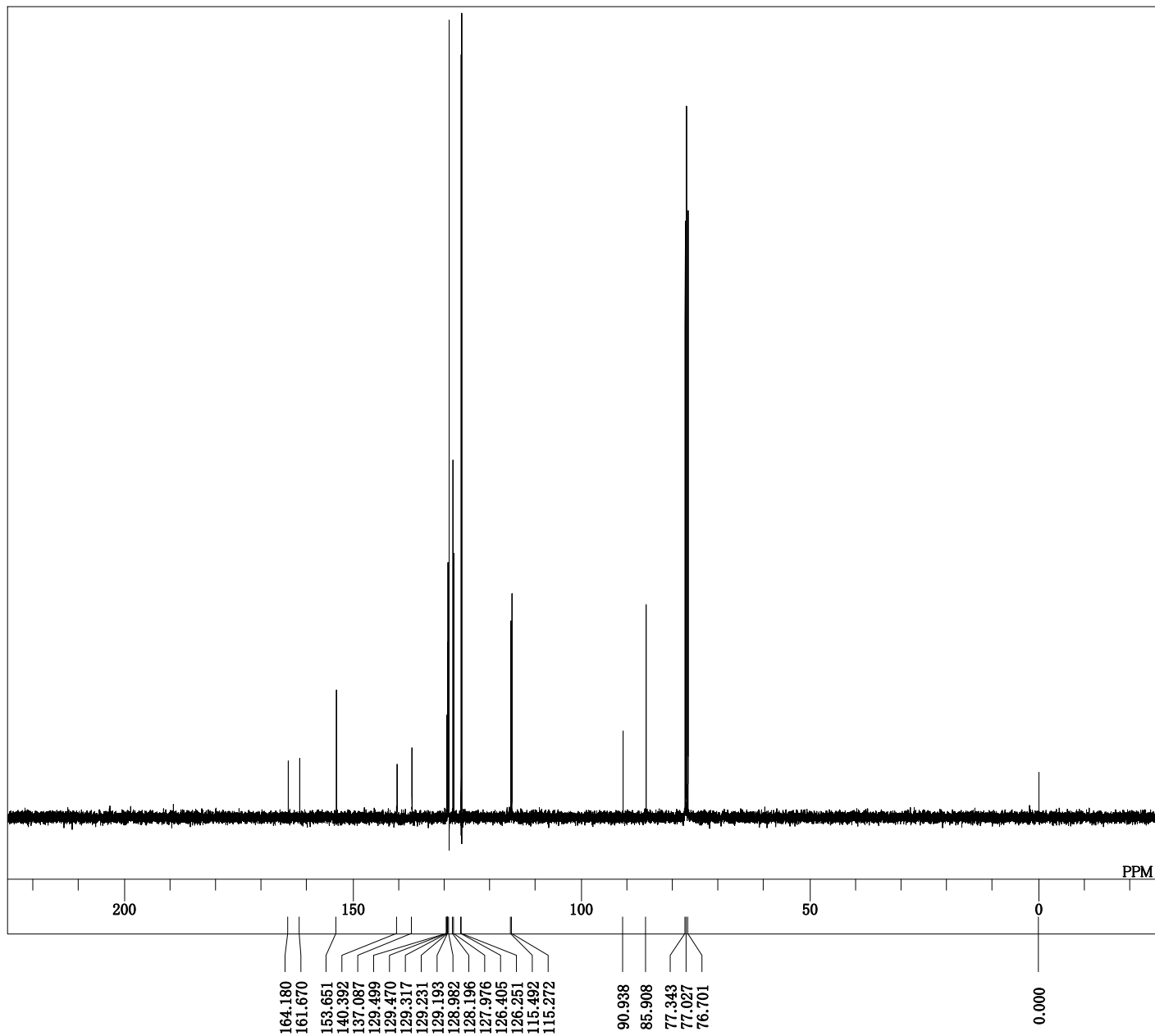
DFILE cyc_H_pF_Proton.als
 COMNT single_pulse
 DATIM 2020-03-28 13:41:50
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 32



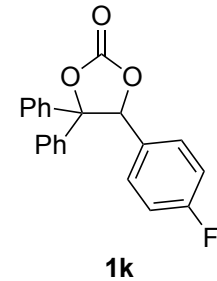
7.598
 7.593
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 7.477
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 7.416
 7.409
 7.391
 7.257
 7.142
 7.133
 7.129
 7.123
 7.109
 7.094
 7.086
 7.028
 7.015
 7.006
 6.998
 6.992
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 6.965
 6.949
 6.943
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 6.863
 6.847
 6.841
 6.315

1.576

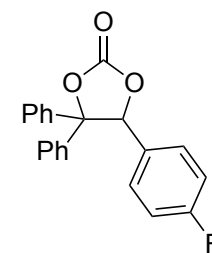
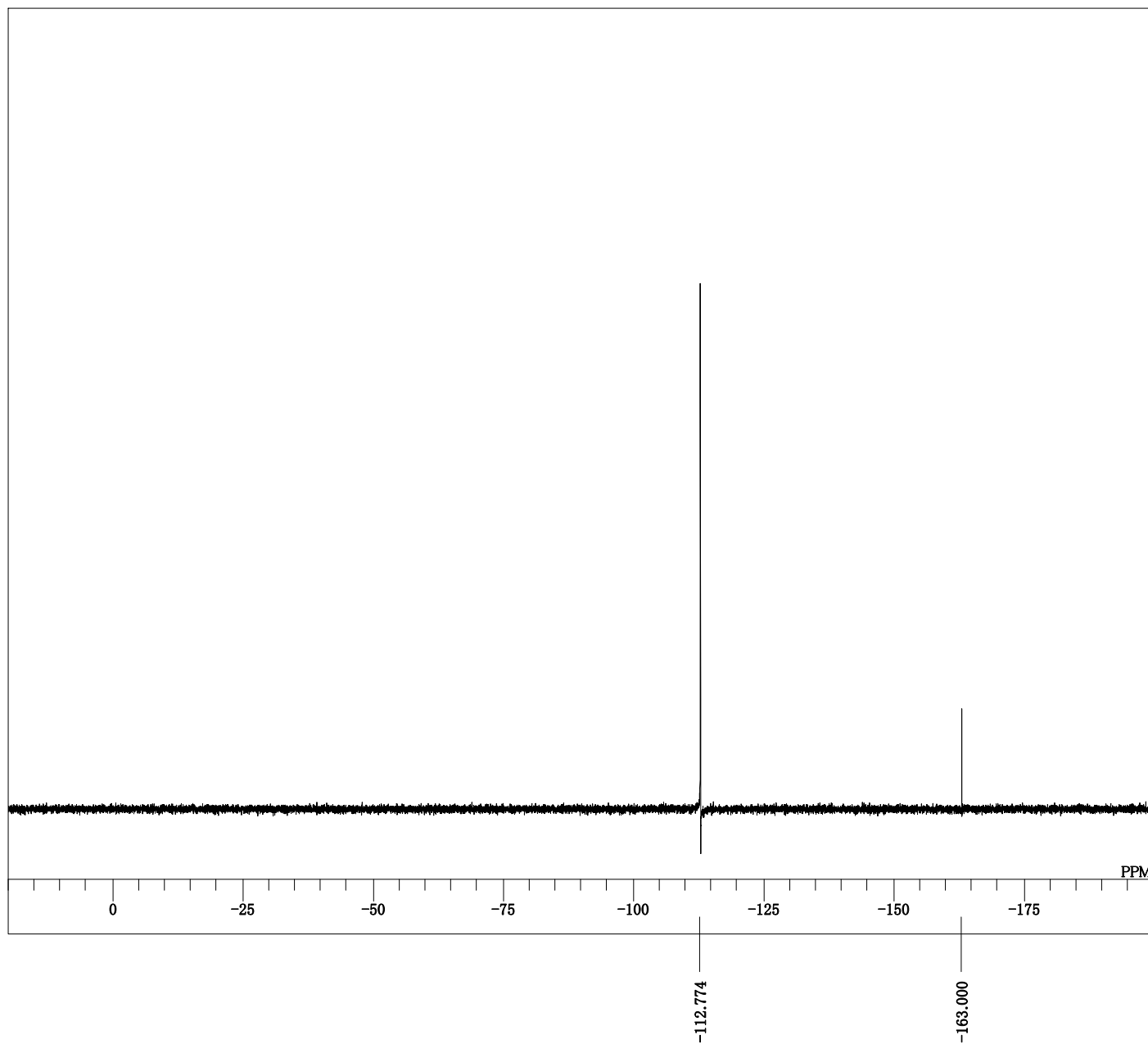
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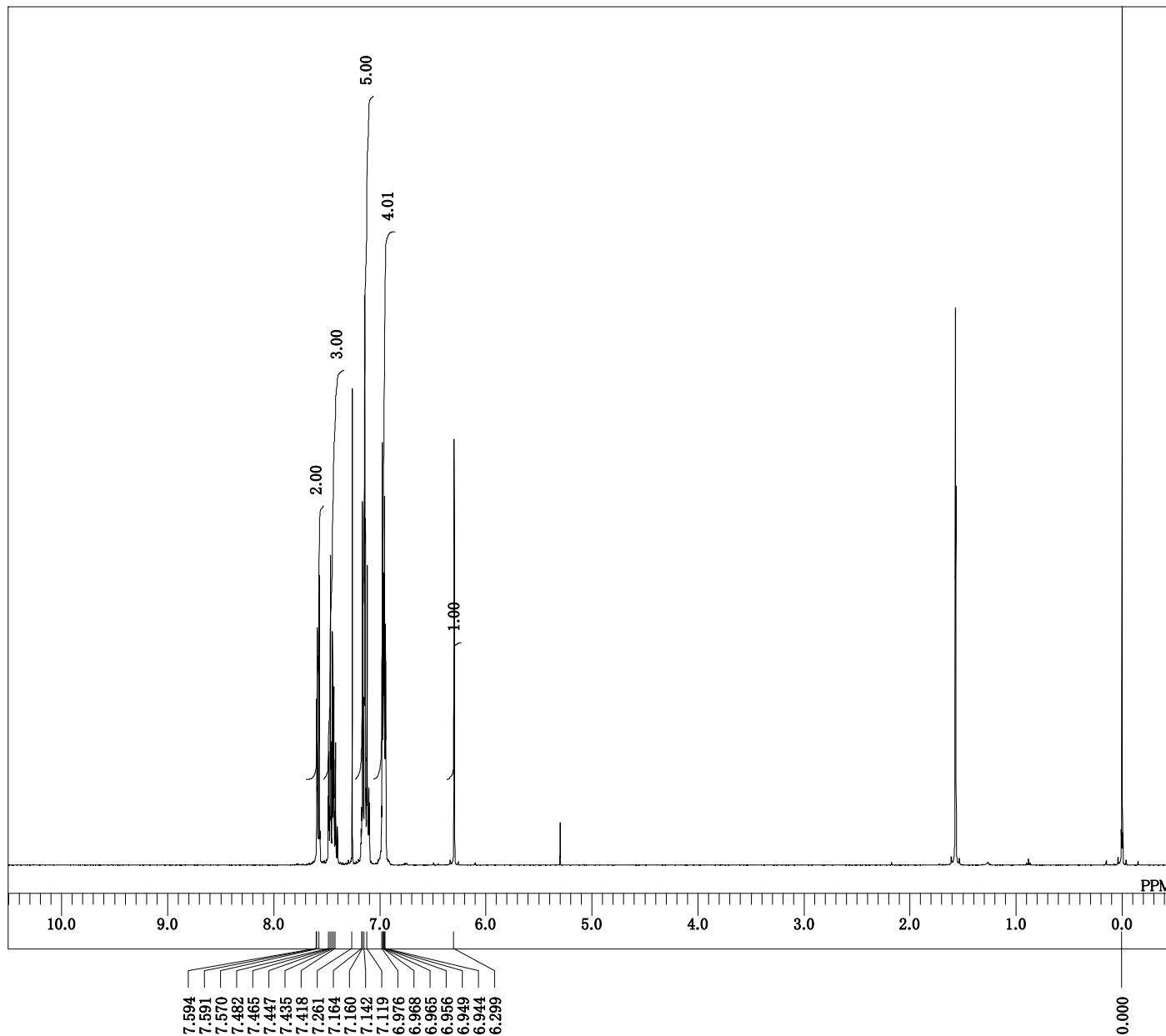
DFILE cyc_H_pF_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-03-28 13:43:03
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 1024
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 20.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50



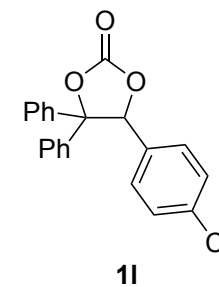
DFILE cyc_H_pF_Fluorine.als
COMNT single_pulse
DATIM 2022-07-04 21:30:06
OBNUC 19F
EXMOD single_pulse.jxp
OBFRQ 372.50 MHz
OBSET 3.36 KHz
OBFIN 6.86 Hz
POINT 26214
FREQU 149253.73 Hz
SCANS 4
ACQTM 0.1756 sec
PD 10.0000 sec
PW1 3.98 usec
IRNUC 19F
CTEMP 20.3 c
SLVNT CDCL3
EXREF -163.00 ppm
BF 0.12 Hz
RGAIN 42

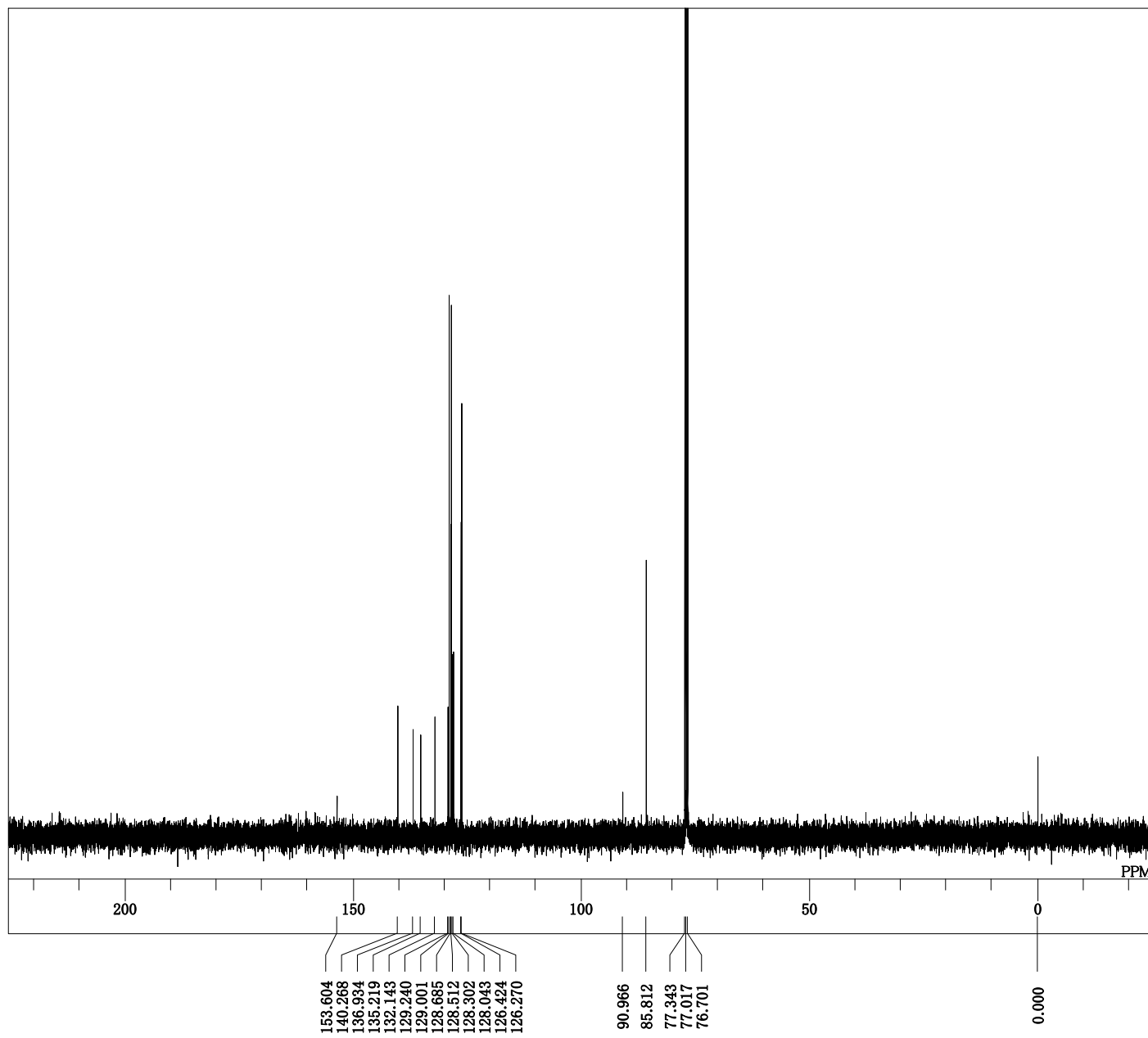


1k

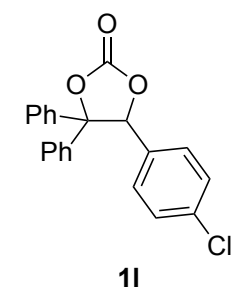


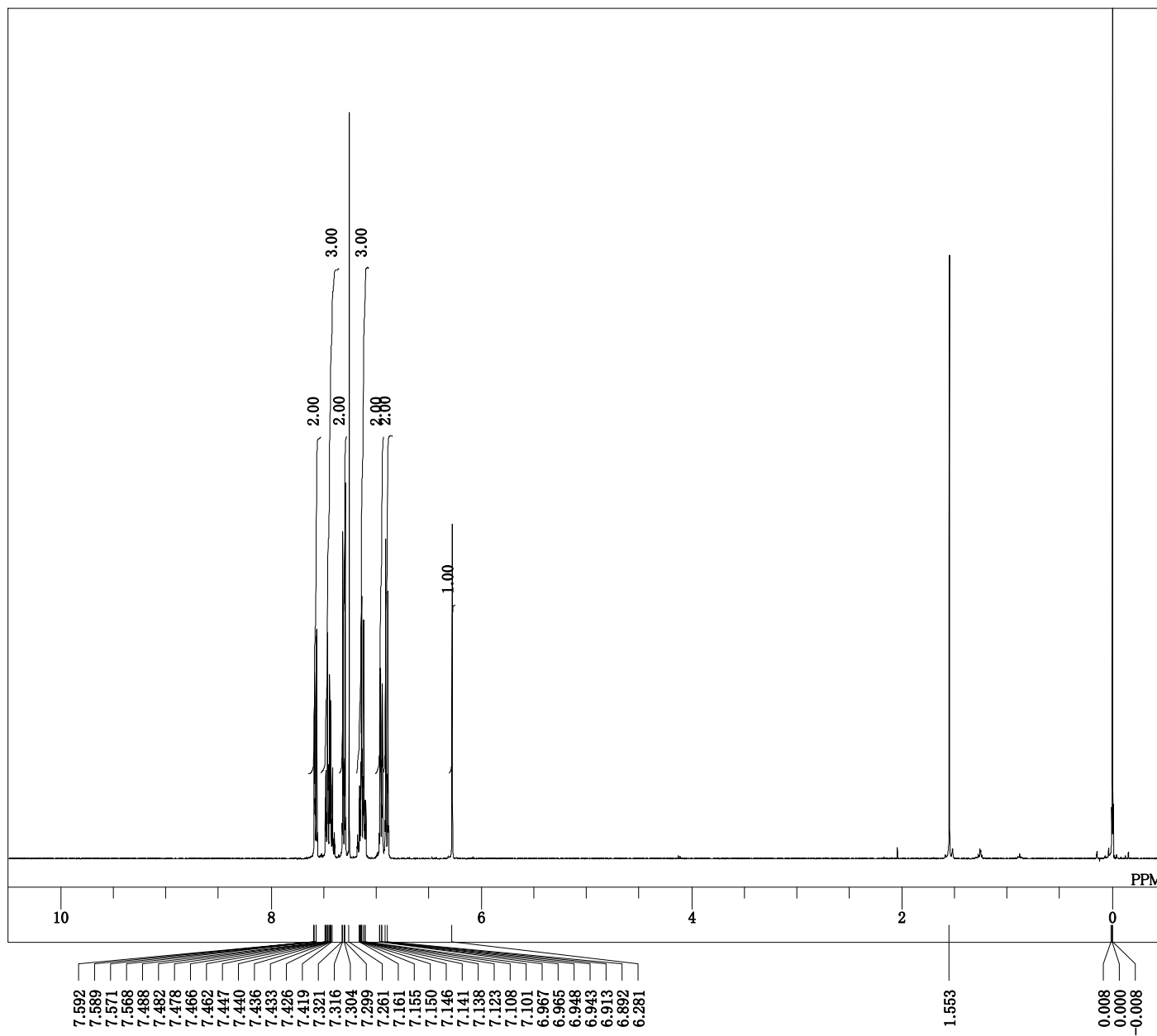
DFILE cyc_H_pClProton.als
 COMNT single_pulse
 DATIM 2020-06-08 18:55:55
 OBNUC 1H
 EXMOD proton_jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 42



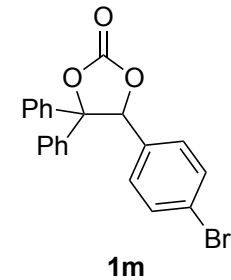


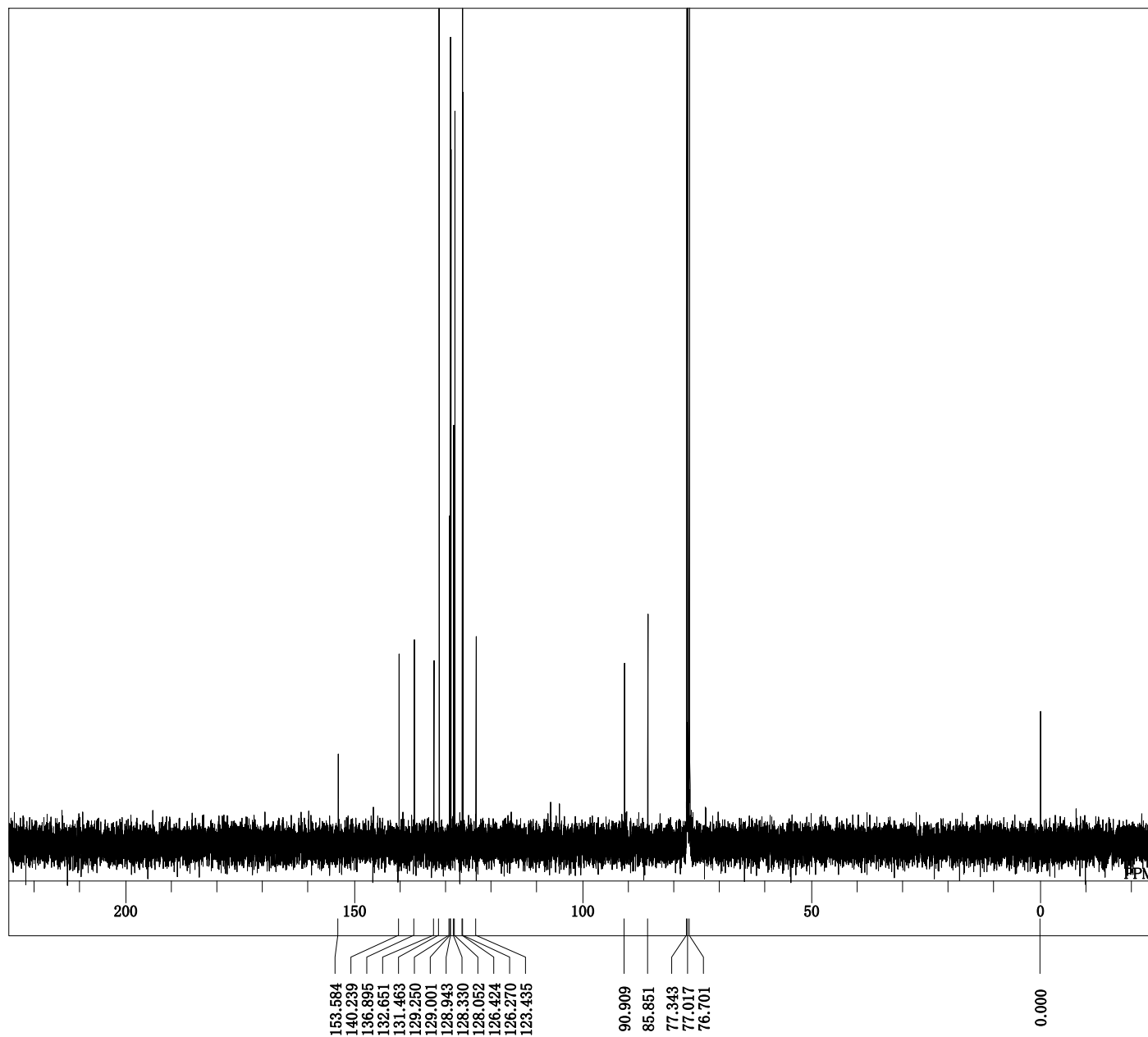
DFILE cyc_H_pClCarbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-06-09 10:25:13
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 648
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 20.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50



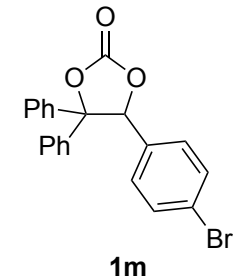


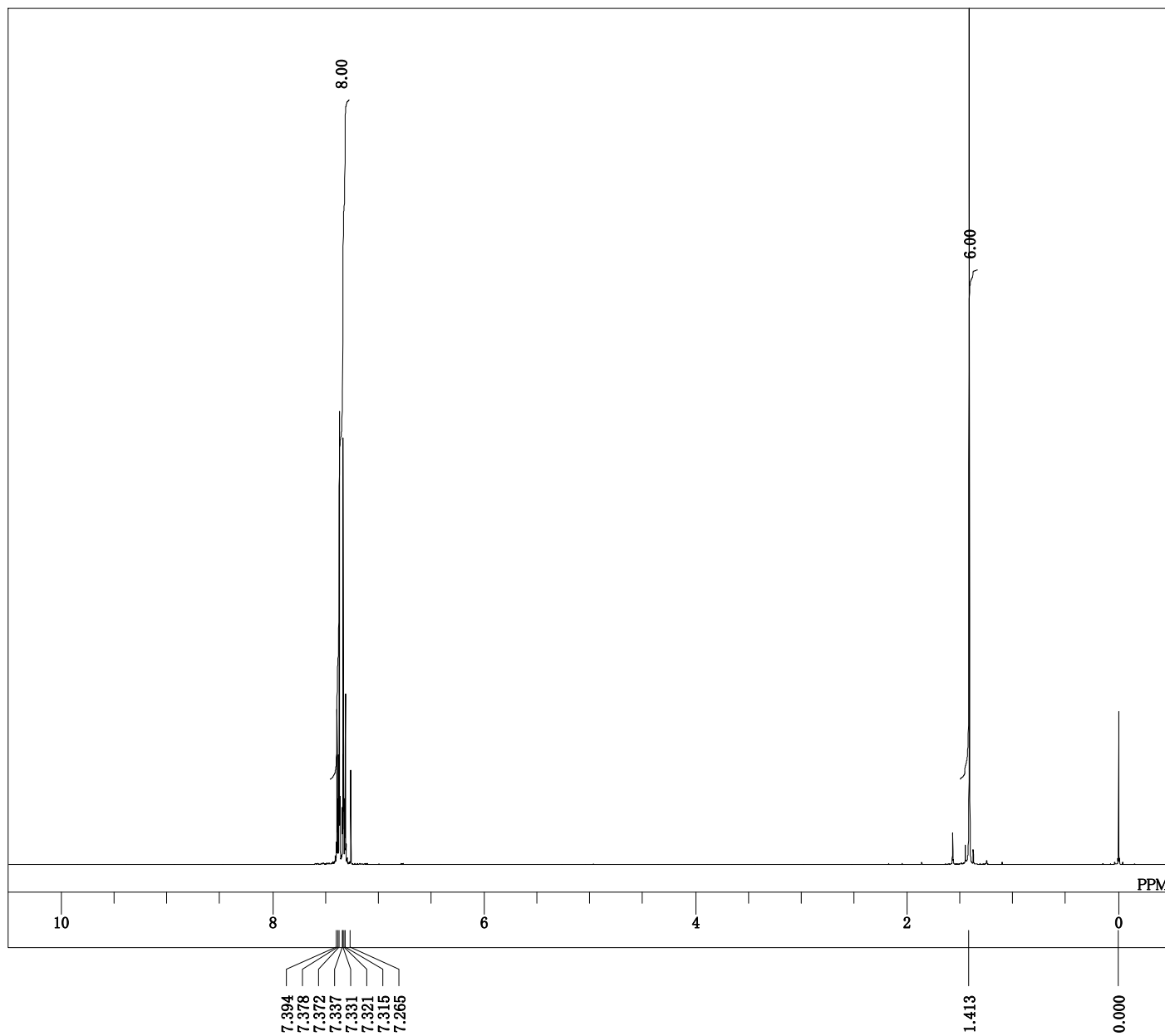
DFILE cyc_H_pBr_Proton.als
 COMNT single_pulse
 DATIM 2020-03-28 14:46:35
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 44



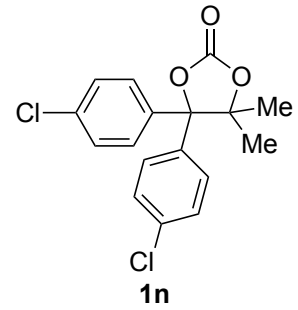


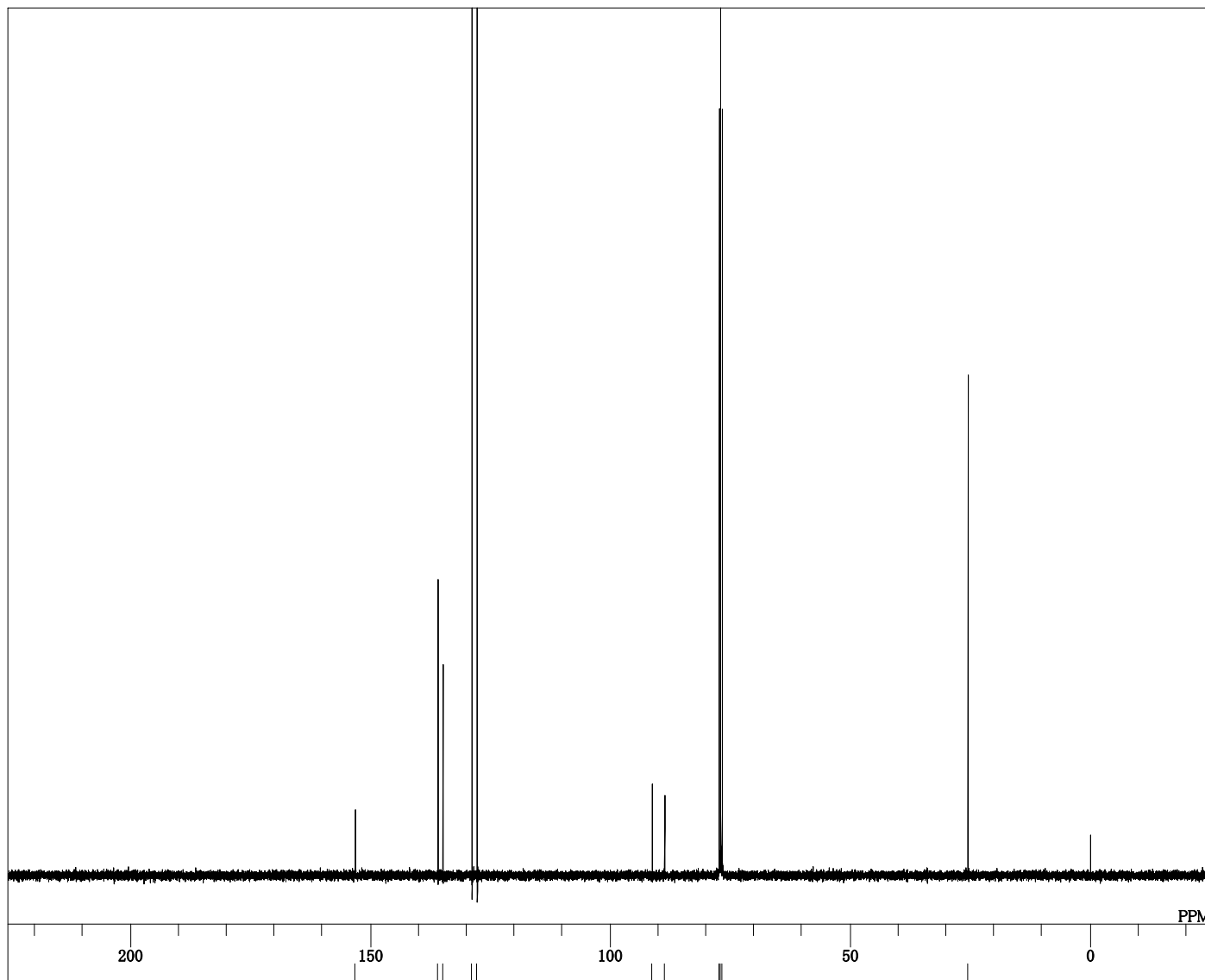
DFILE cyc_H_pBr_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-03-30 08:34:47
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 2048
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 20.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50



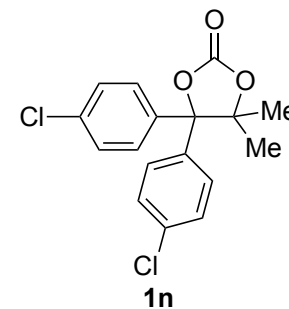


DFILE cyc_pClpClMeMe_Proton.als
 COMNT single_pulse
 DATIM 2020-11-25 00:32:07
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 18.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 38

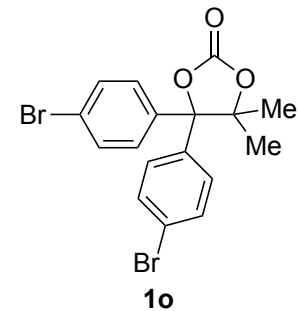
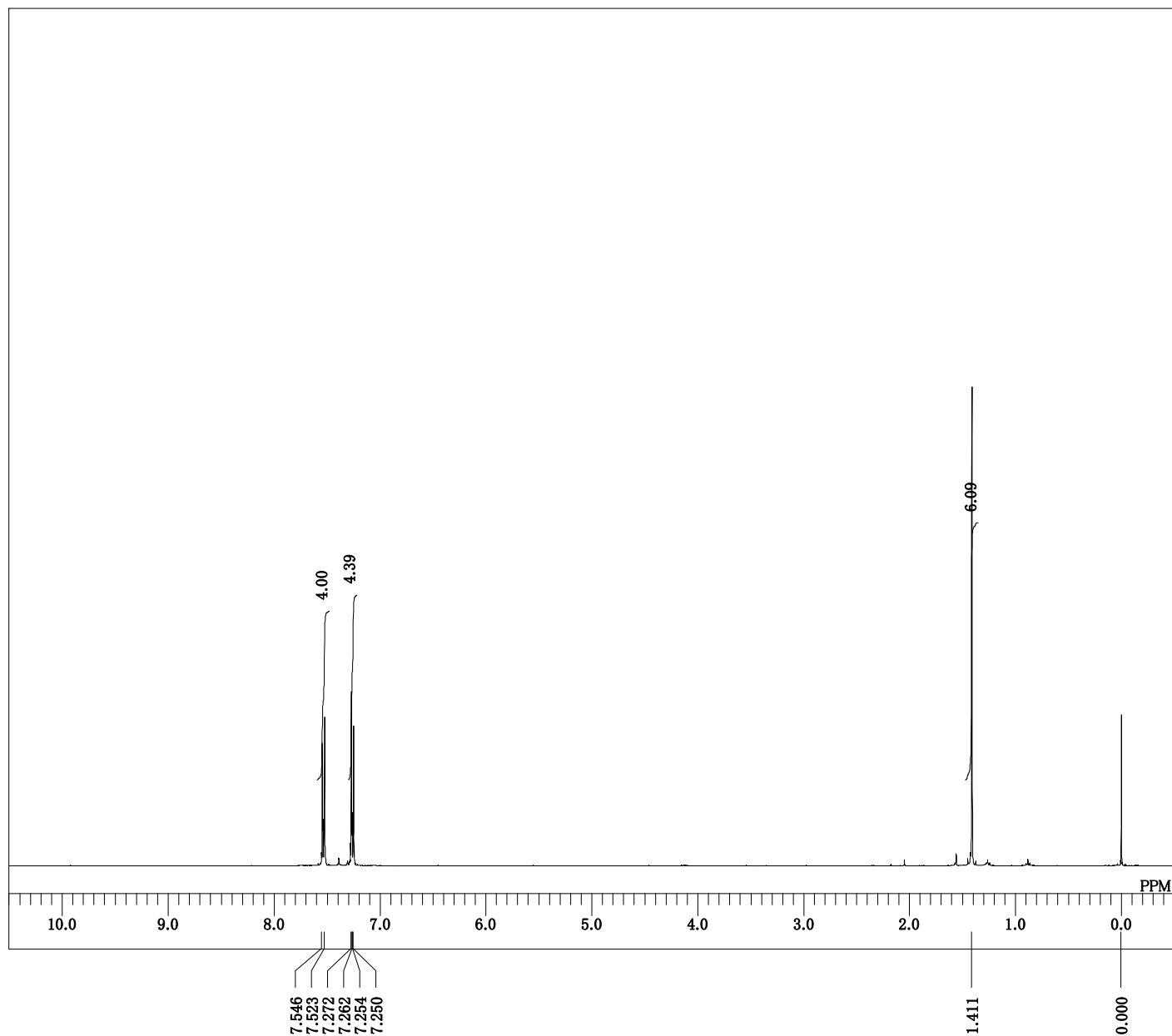


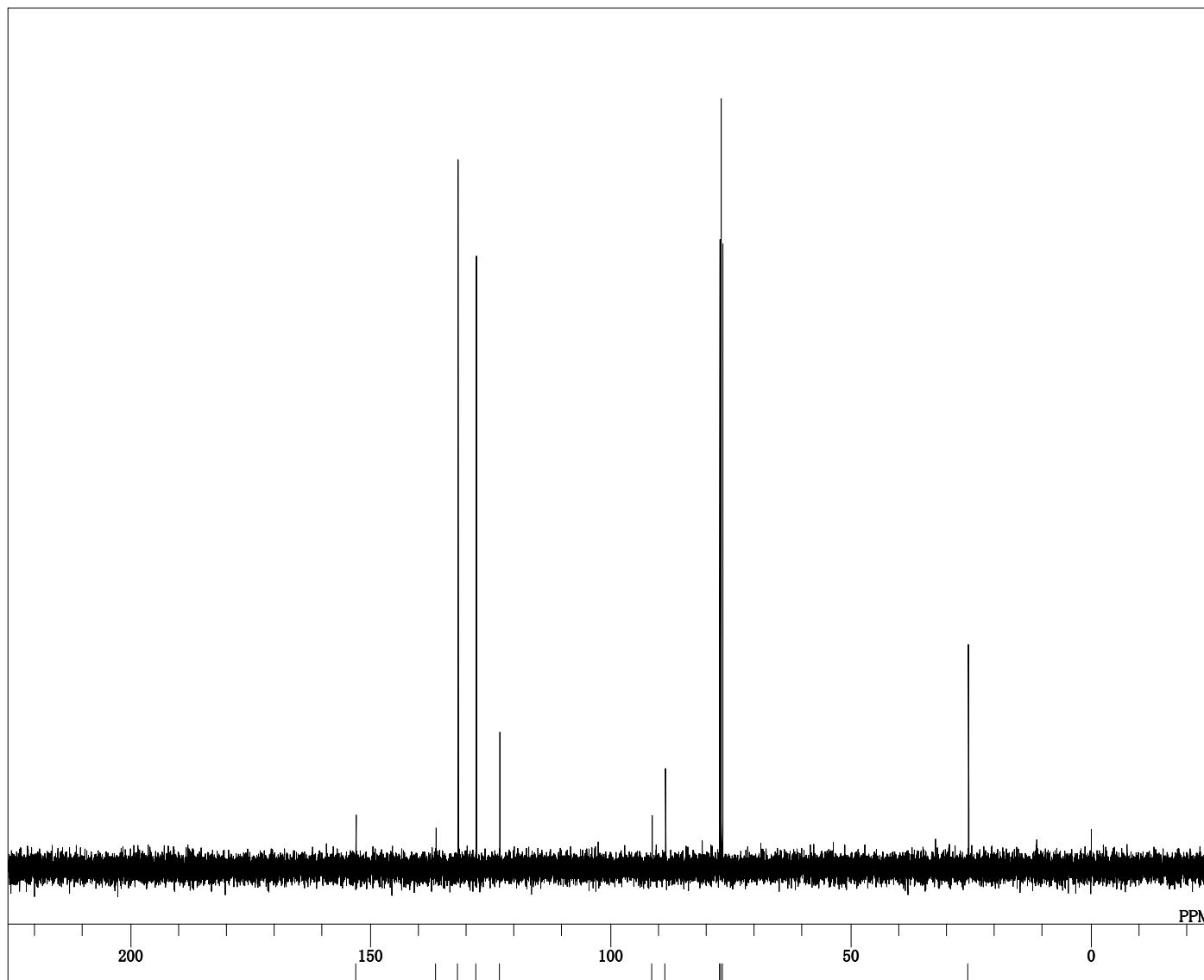


DFILE cyc_pClpCl_MeMe_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-11-25 00:33:20
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 2144
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 18.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50

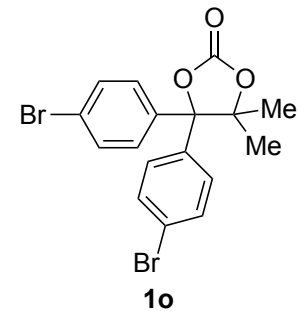


DFILE cyc_pBrpBr_MeMe_Proton.als
 COMNT single_pulse
 DATIM 2020-12-02 09:22:38
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 18.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 40





DFILE cyc_pBrpBr_MeMe_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-12-02 09:23:51
 OBNUC ¹³C
 EXMOD carbon.jxp
 OBFREQ 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 18.6 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



153.098

136.380

131.800

128.006

123.101

91.361

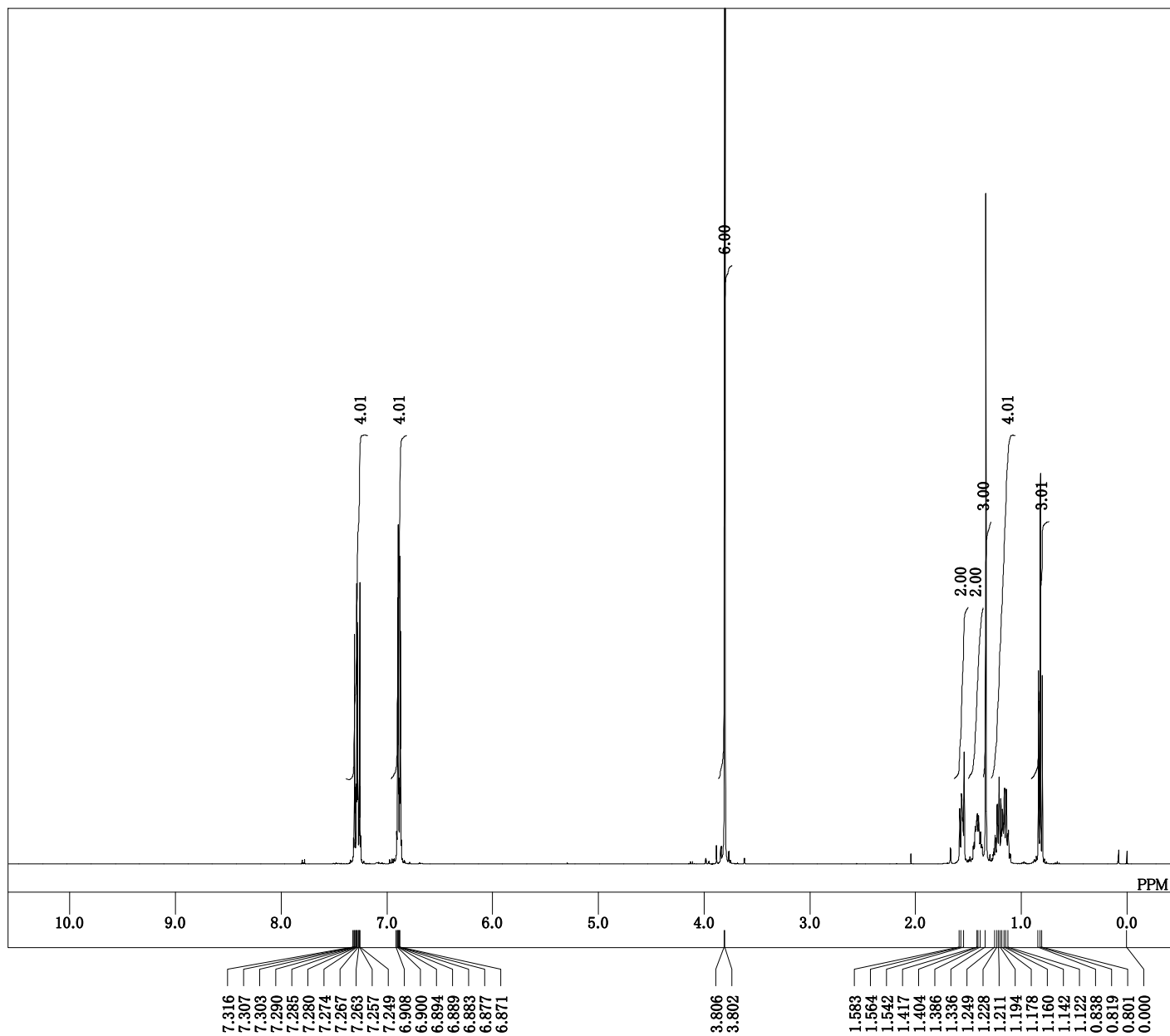
88.583

77.326

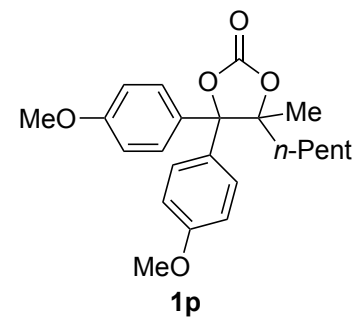
77.000

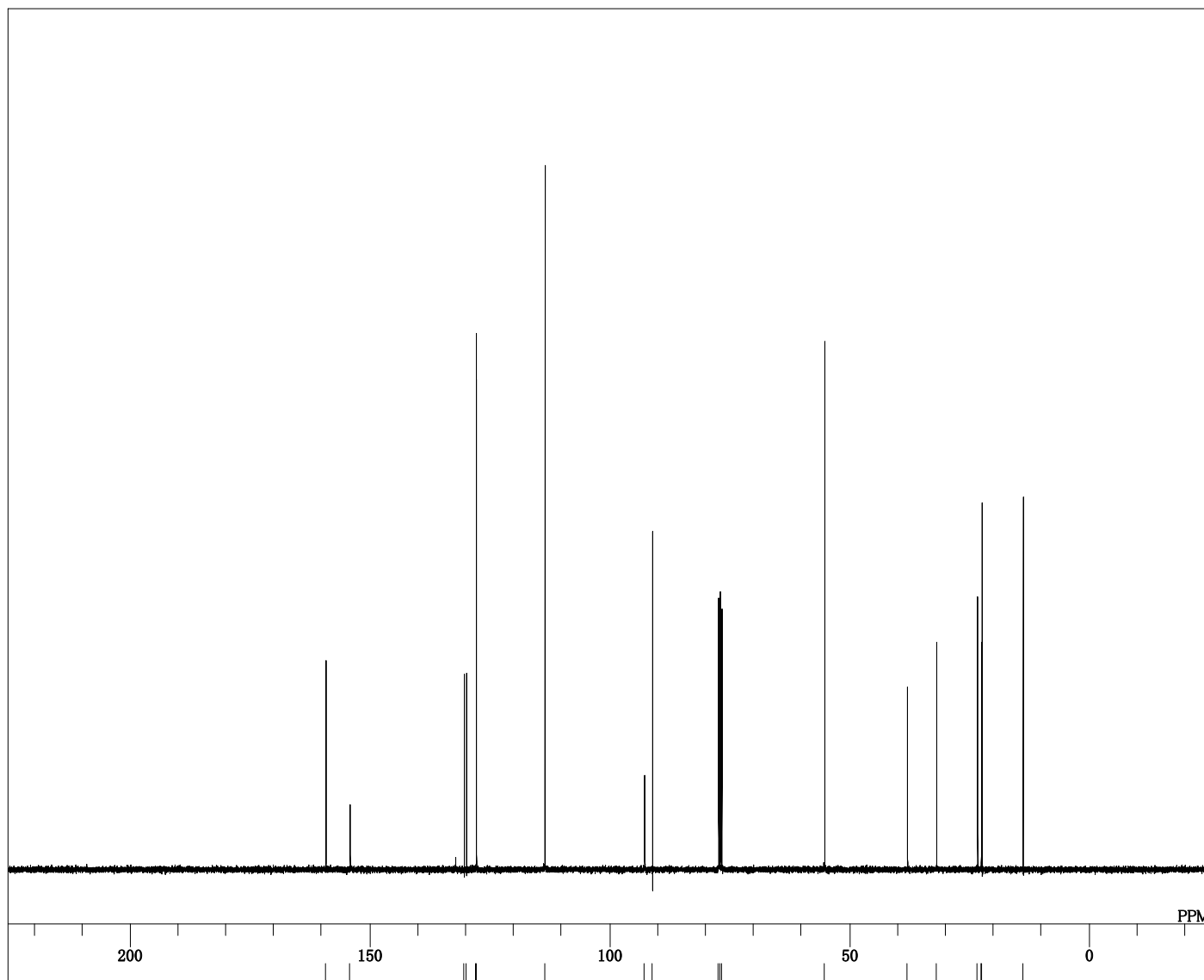
76.684

25.505

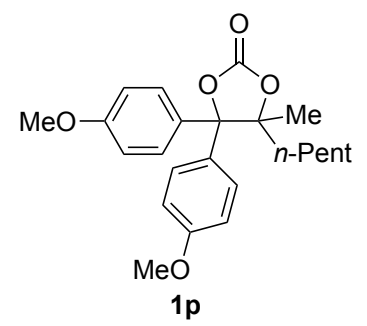


DFILE cyc_PMPPMP_MenPentan_Proton.al
 COMNT single_pulse
 DATIM 2020-10-09 09:47:57
 OBNUC 1H
 EXMOD proton.jp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 22

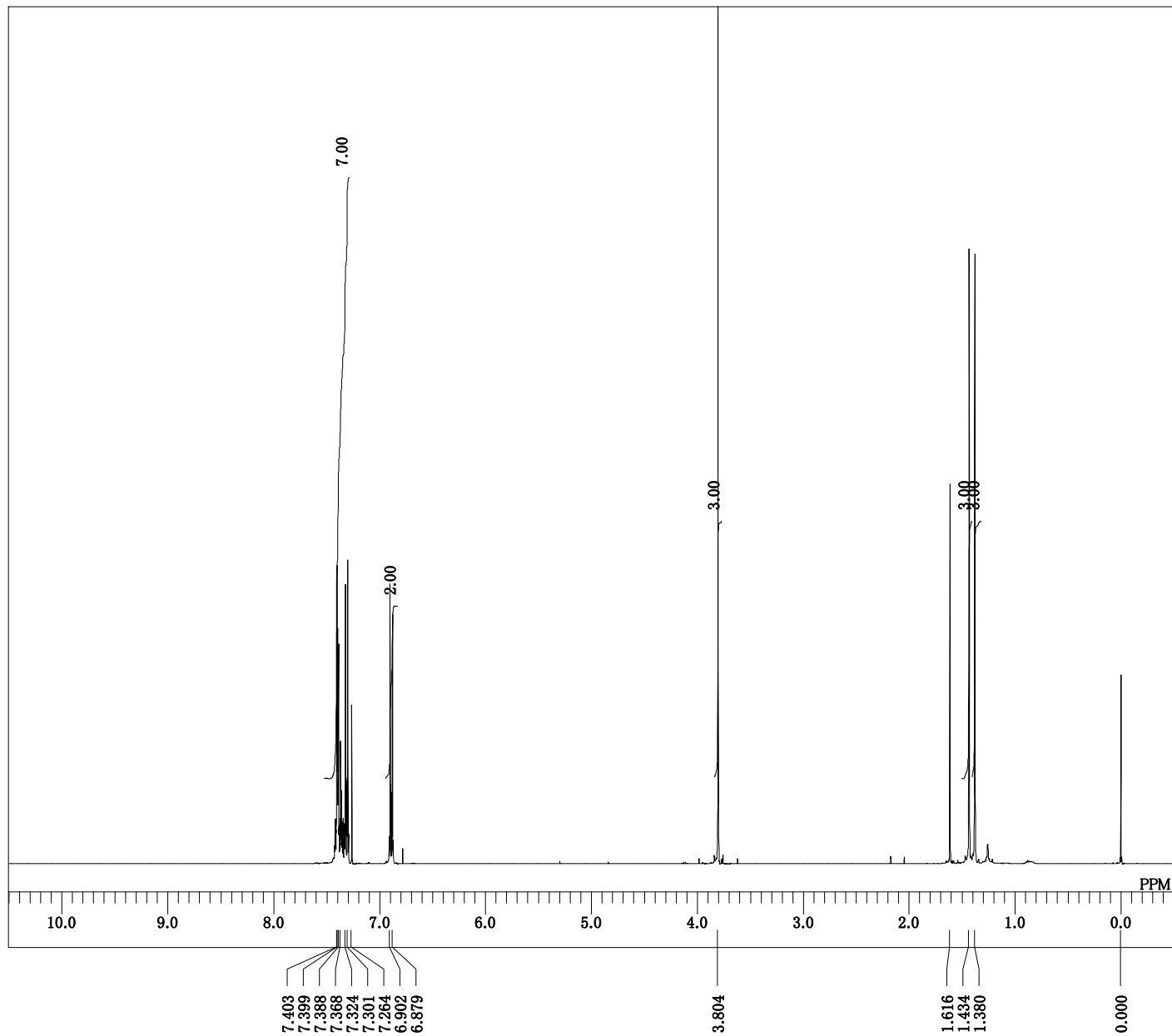




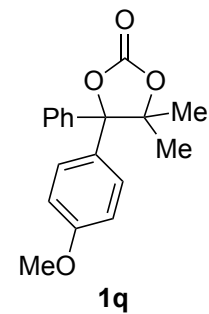
DFILE cyc_PMPMP_MenPentan_Carbon.
 COMNT single pulse decoupled gated NOE
 DATIM 2020-10-09 13:24:21
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 637
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 20.4 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50

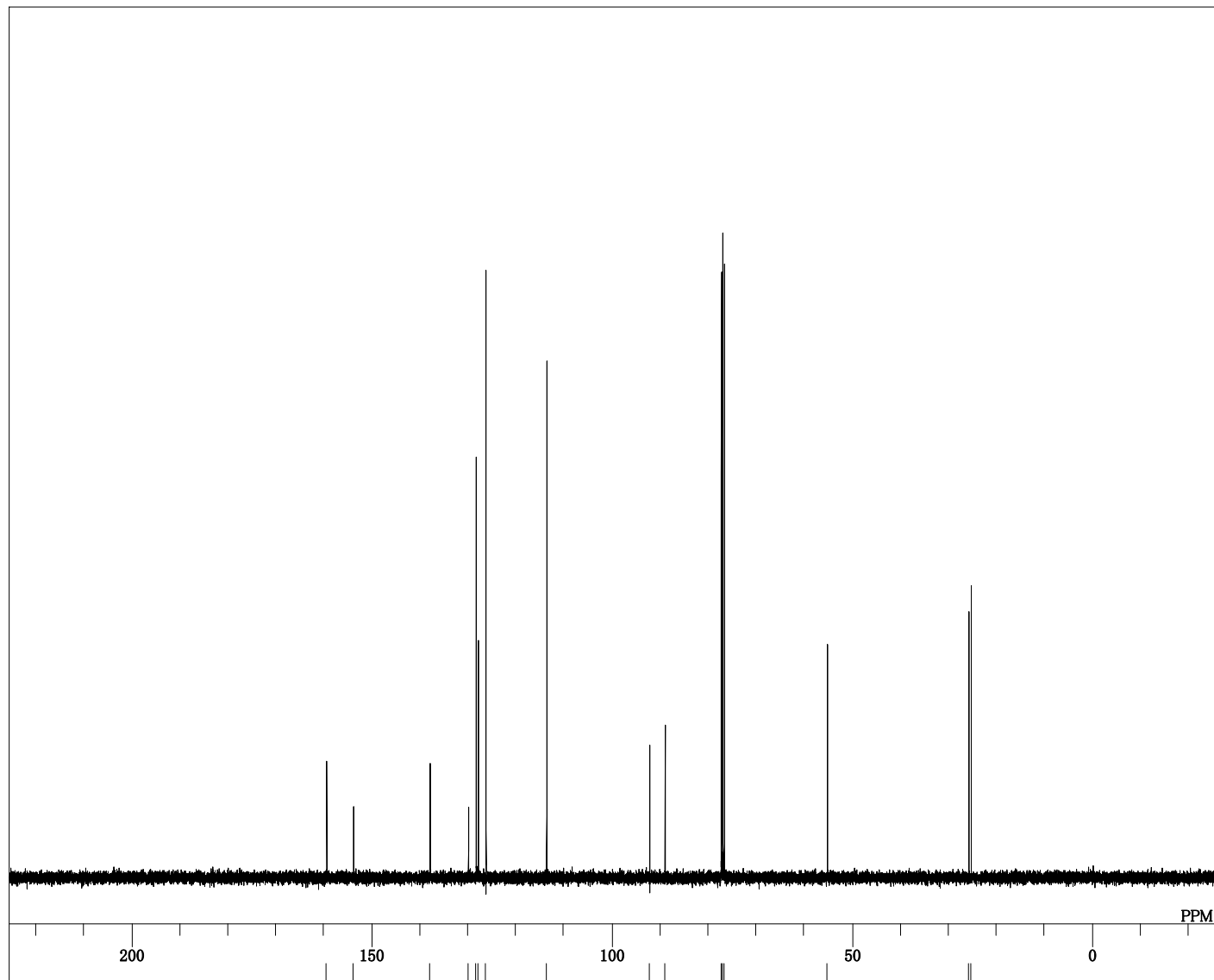


159.277
 154.161
 130.392
 129.922
 127.891
 127.834
 113.569
 92.769
 91.131
 77.326
 77.000
 76.684
 55.214
 37.940
 31.886
 23.330
 22.573
 22.382
 13.855

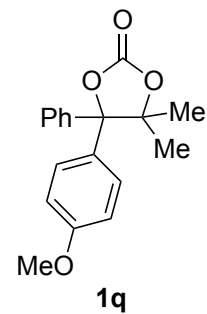


DFILE cyc_PhPMP_MeMe_Proton.als
 COMNT single_pulse
 DATIM 2020-09-13 11:10:51
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 32





DFILE cyc.PhPMP_MeMe_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-09-13 11:12:04
 OBNUC ¹³C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 611
 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



159.440
153.902

137.903
129.970
128.370
127.882
126.291

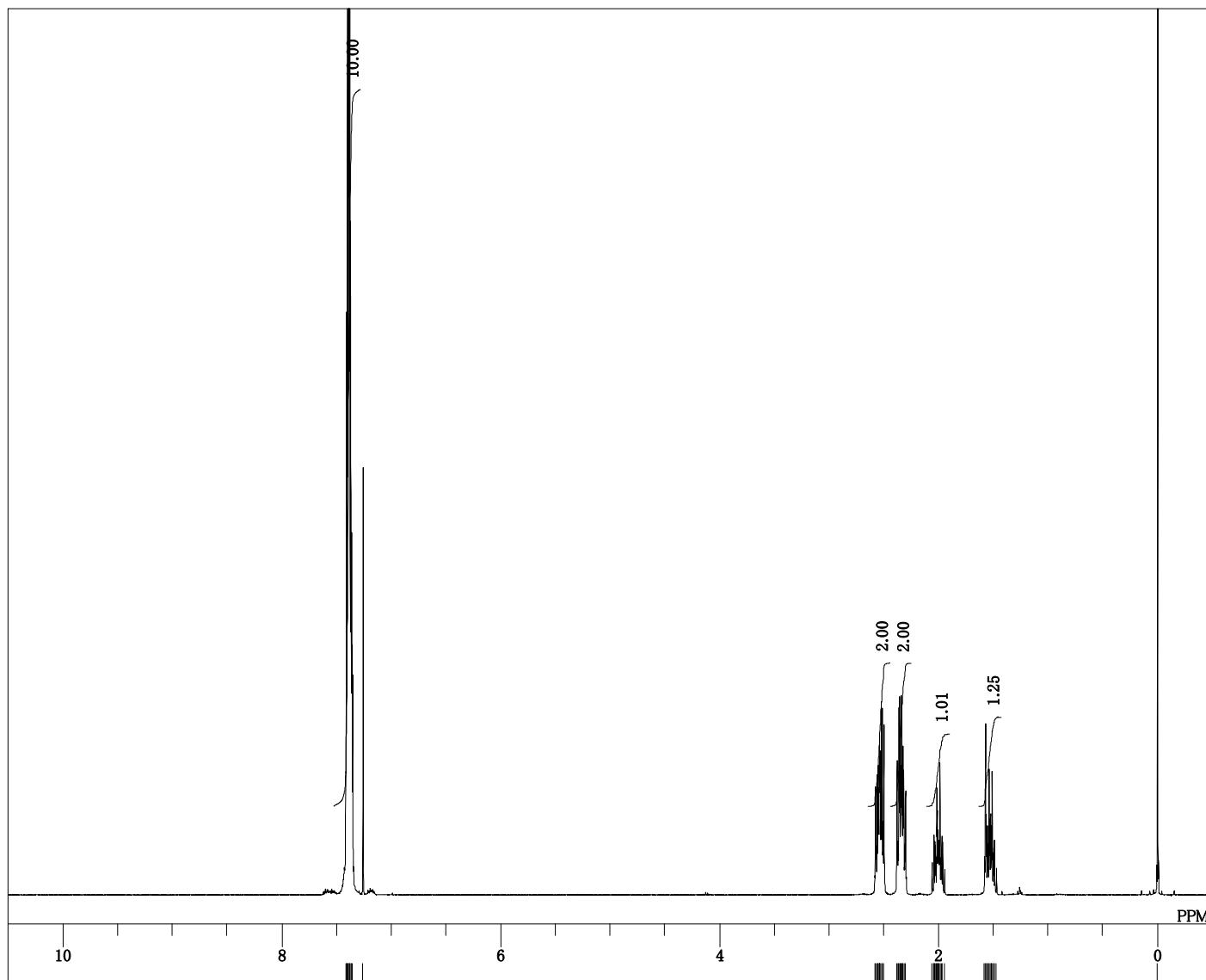
113.693

92.185
89.043

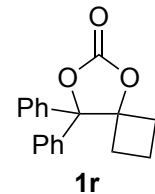
77.316
77.000
76.674

55.262

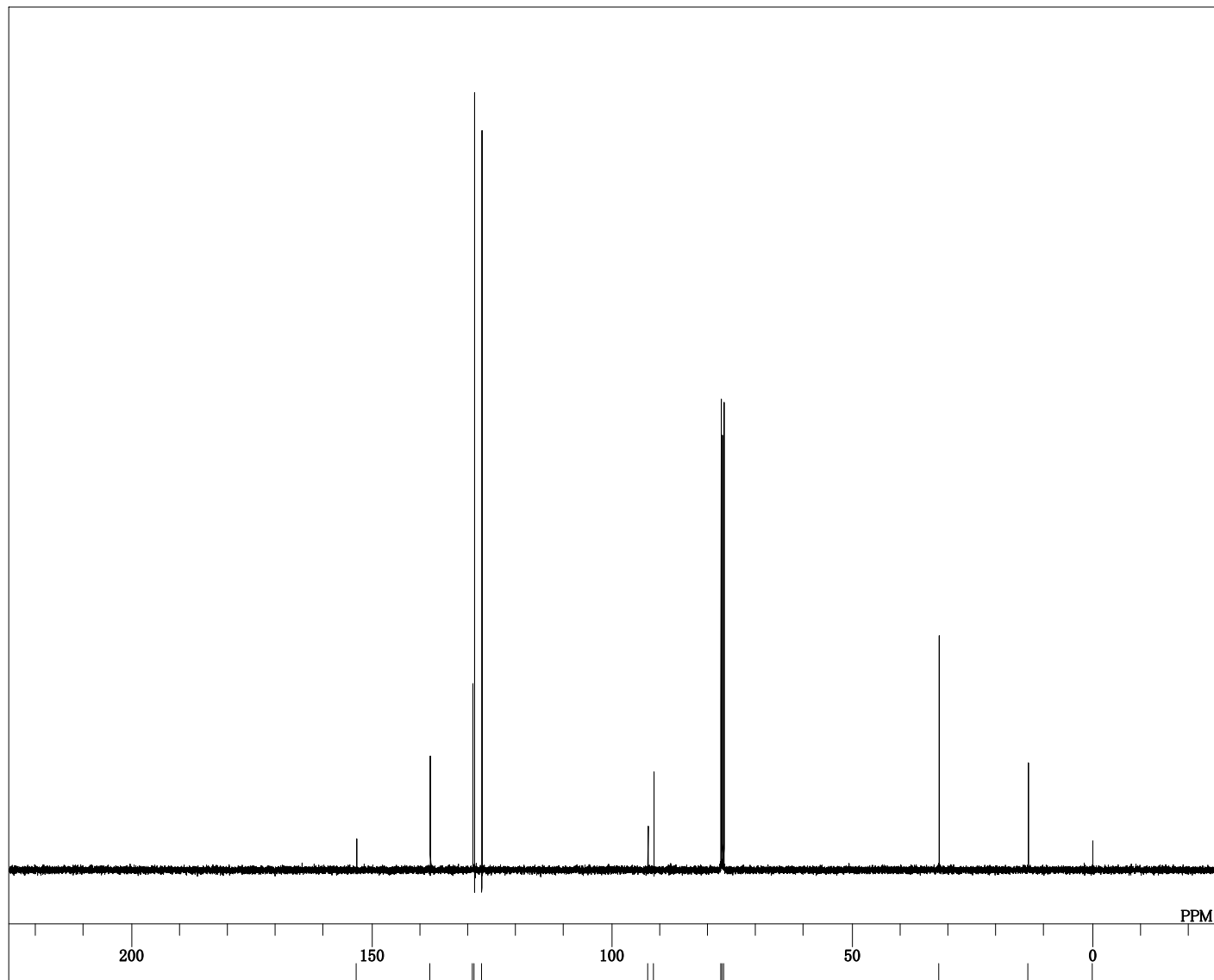
25.754
25.304



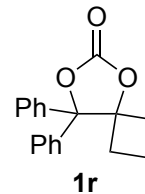
DFILE cyc_cyc_butyl_Proton.als
 COMNT single_pulse
 DATIM 2020-03-27 03:07:07
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 19.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 36



7.414
 7.408
 7.403
 7.395
 7.391
 7.383
 7.379
 7.375
 7.364
 7.362
 7.356
 7.261
 2.680
 2.572
 2.564
 2.554
 2.551
 2.542
 2.538
 2.535
 2.526
 2.517
 2.510
 2.501
 2.382
 2.374
 2.364
 2.358
 2.348
 2.344
 2.340
 2.331
 2.328
 2.320
 2.311
 2.303
 2.061
 2.044
 2.036
 2.031
 2.027
 2.018
 2.014
 2.006
 2.002
 1.997
 1.989
 1.981
 1.972
 1.964
 1.947
 1.582
 1.572
 1.566
 1.556
 1.550
 1.542
 1.536
 1.527
 1.516
 1.512
 1.501
 1.496
 1.488
 1.472
 0.000



DFILE cyc_cyc_butyl_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-03-27 03:08:20
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 1024
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 19.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50



153.192

137.805

128.982

128.599

127.123

92.547

91.321

77.343

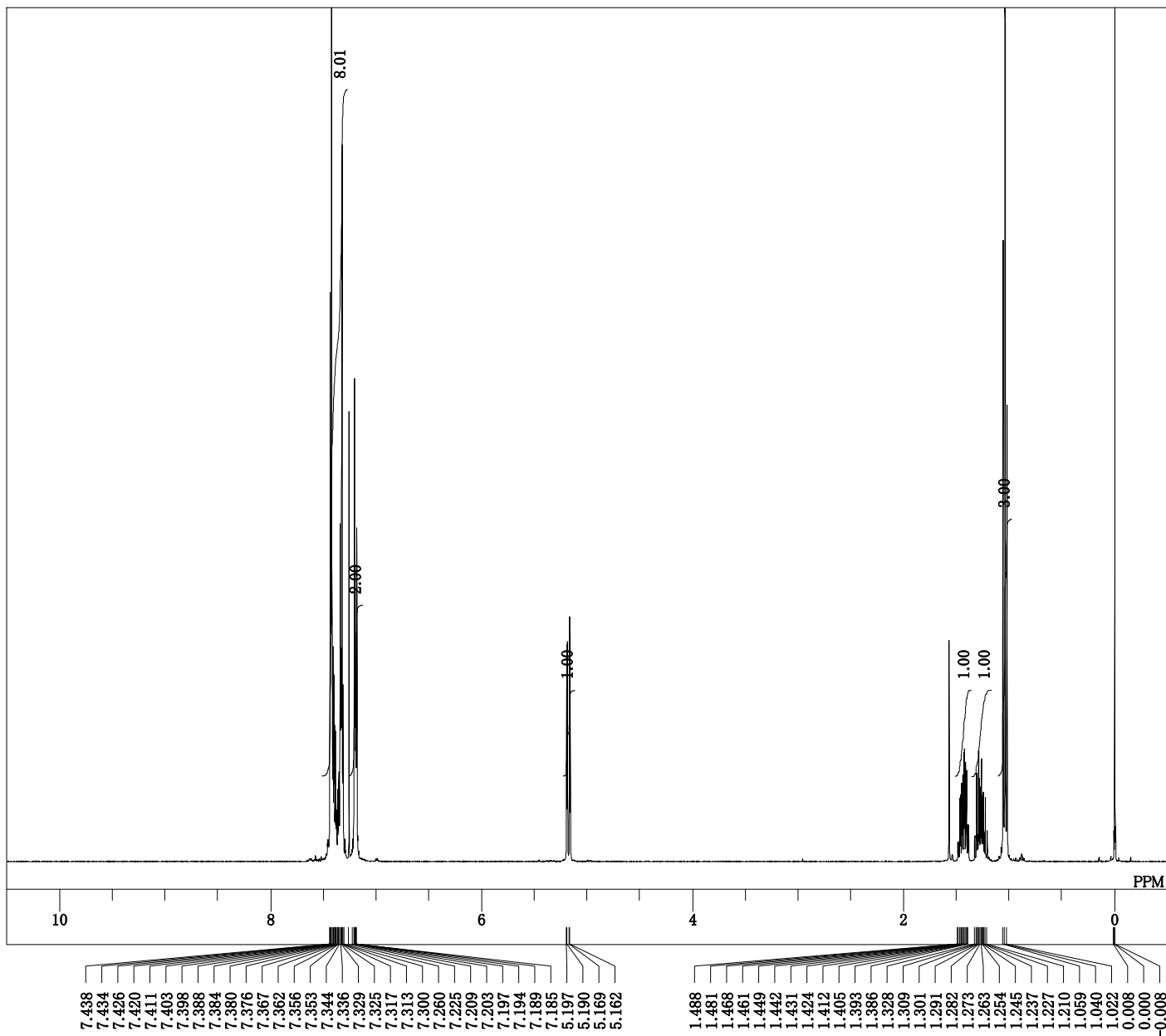
77.017

76.701

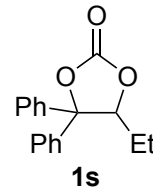
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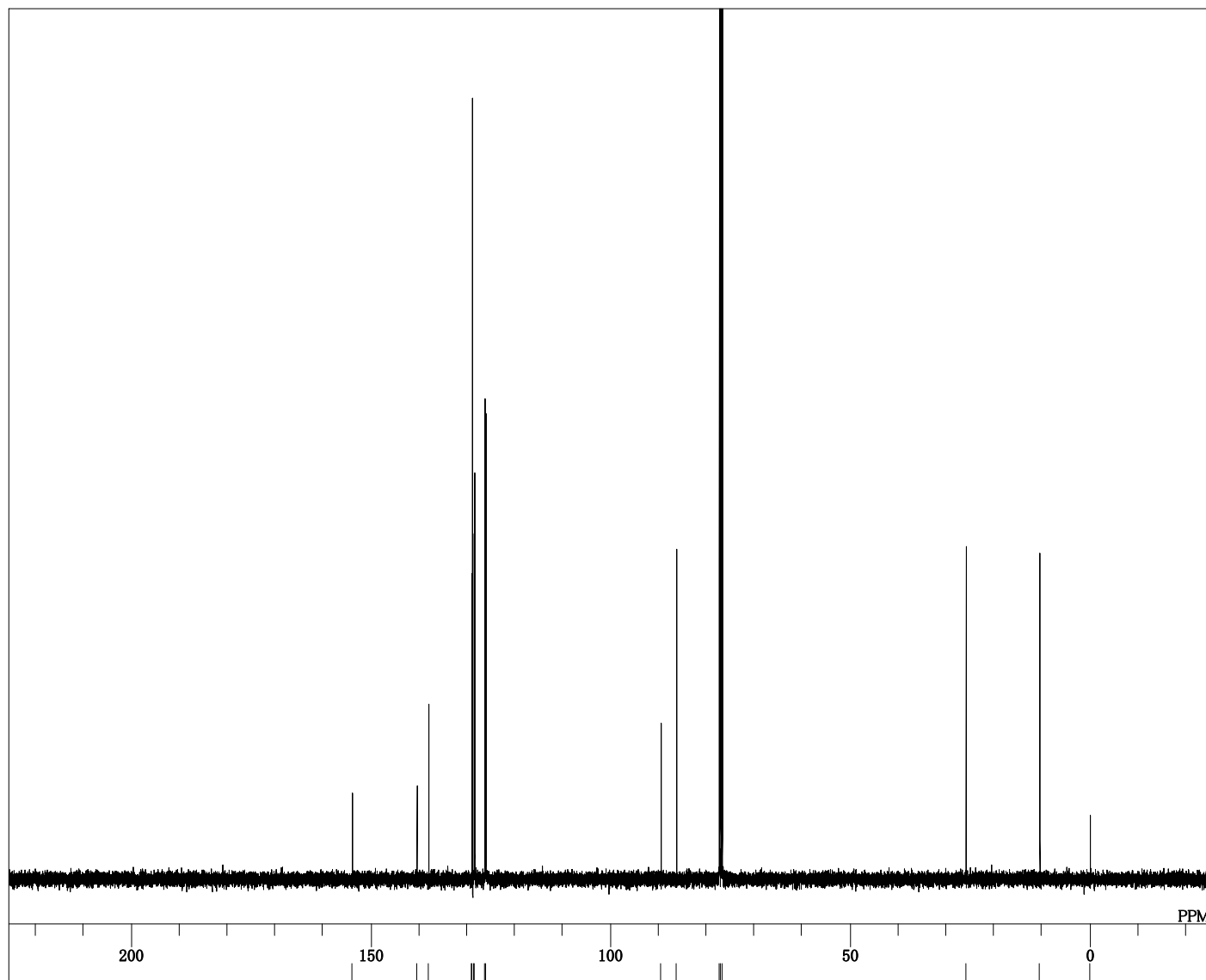
13.393

0.000

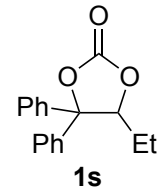


DFILE cyc_H_Et_Proton.als
 COMNT single_pulse
 DATIM 2020-03-27 05:44:45
 OBNUC 1H
 EXMOD proton_jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 19.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 36





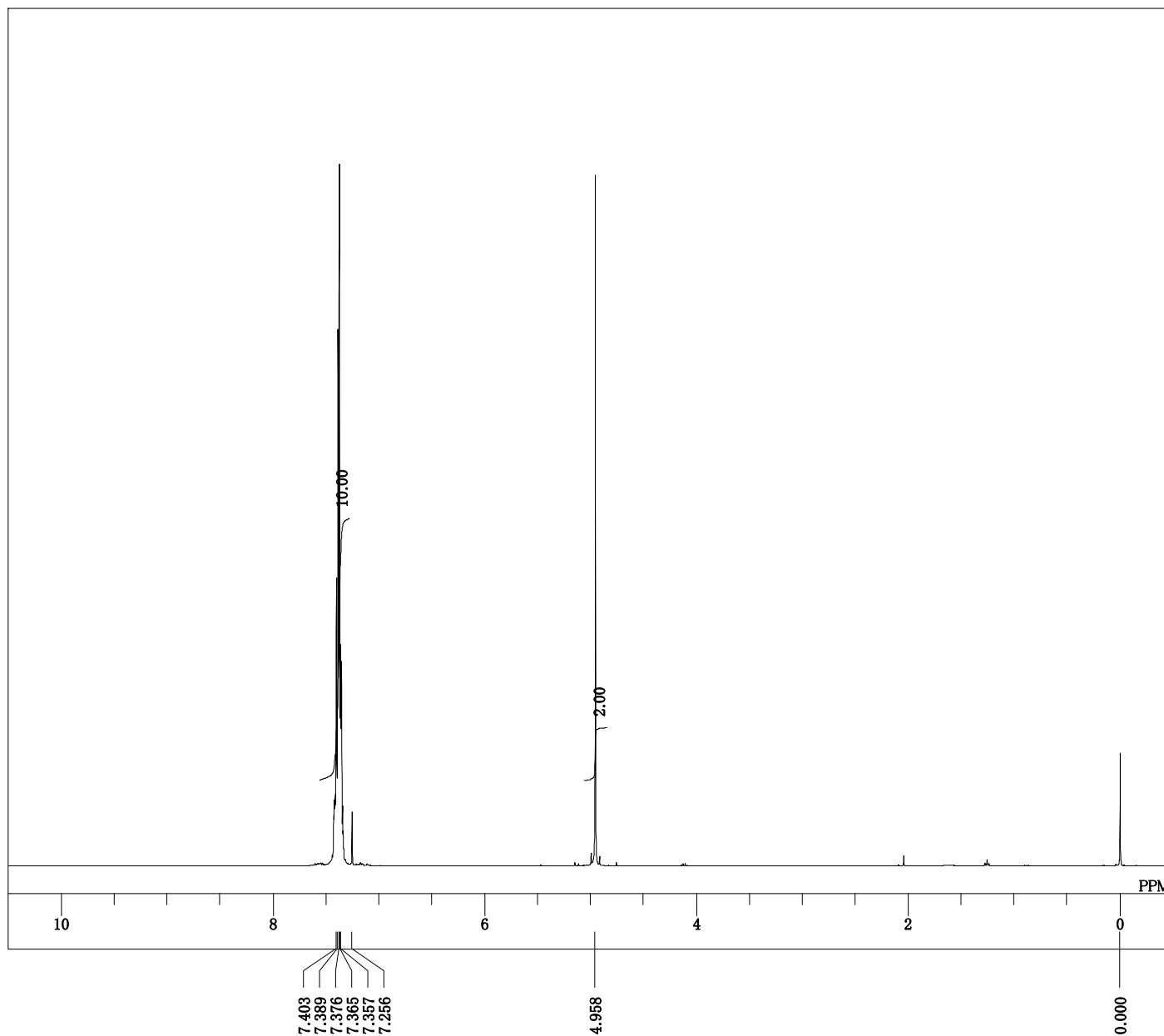
DFILE cyc_H_Et_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-03-27 05:45:58
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 1024
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 19.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50



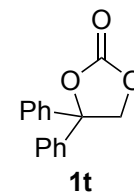
153.929
 140.421
 137.930
 128.982
 128.838
 128.493
 128.397
 126.242
 126.050

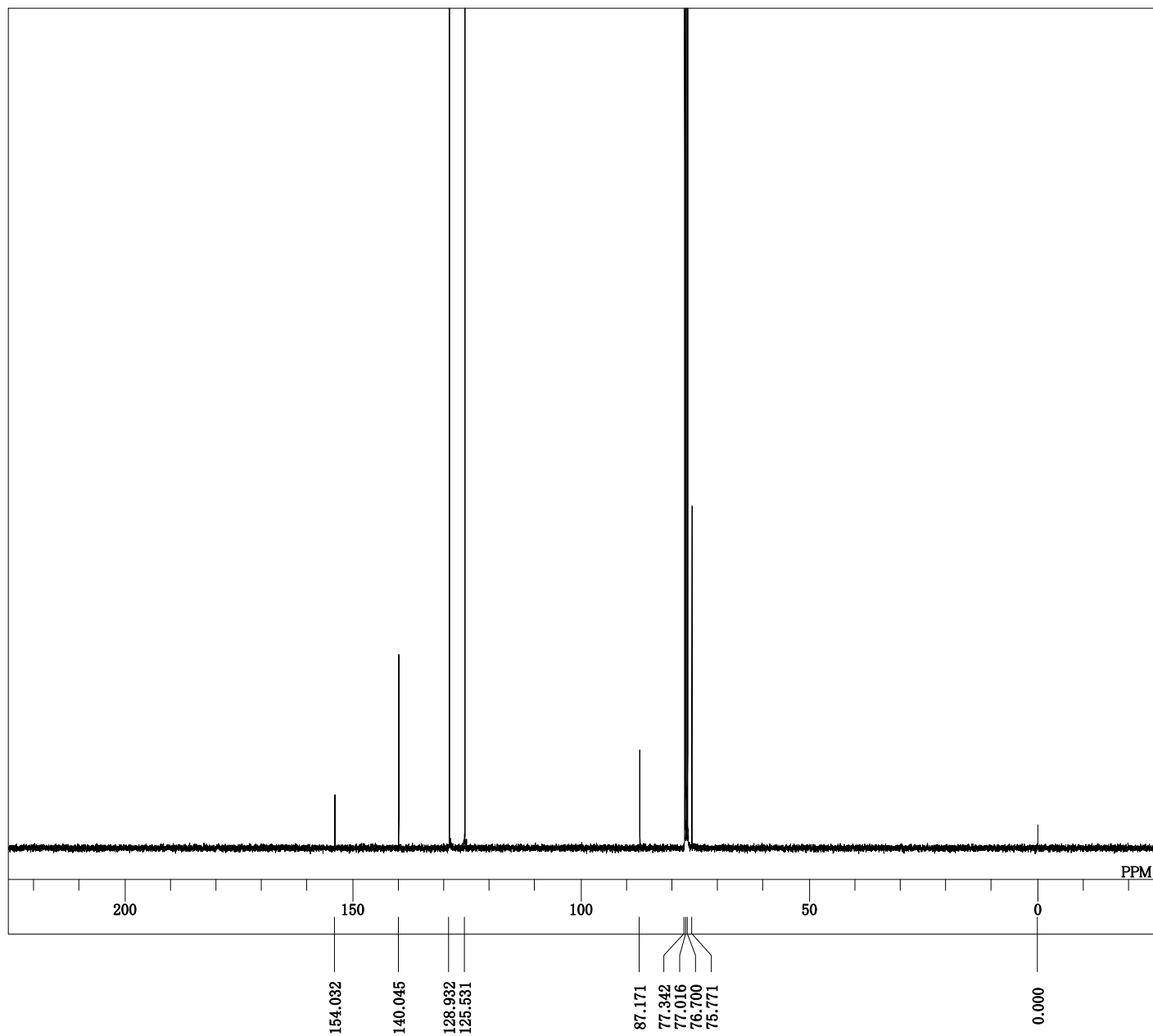
89.529
 86.234
 77.343
 77.017
 76.701

25.867
 10.452
 0.000

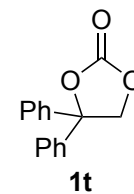


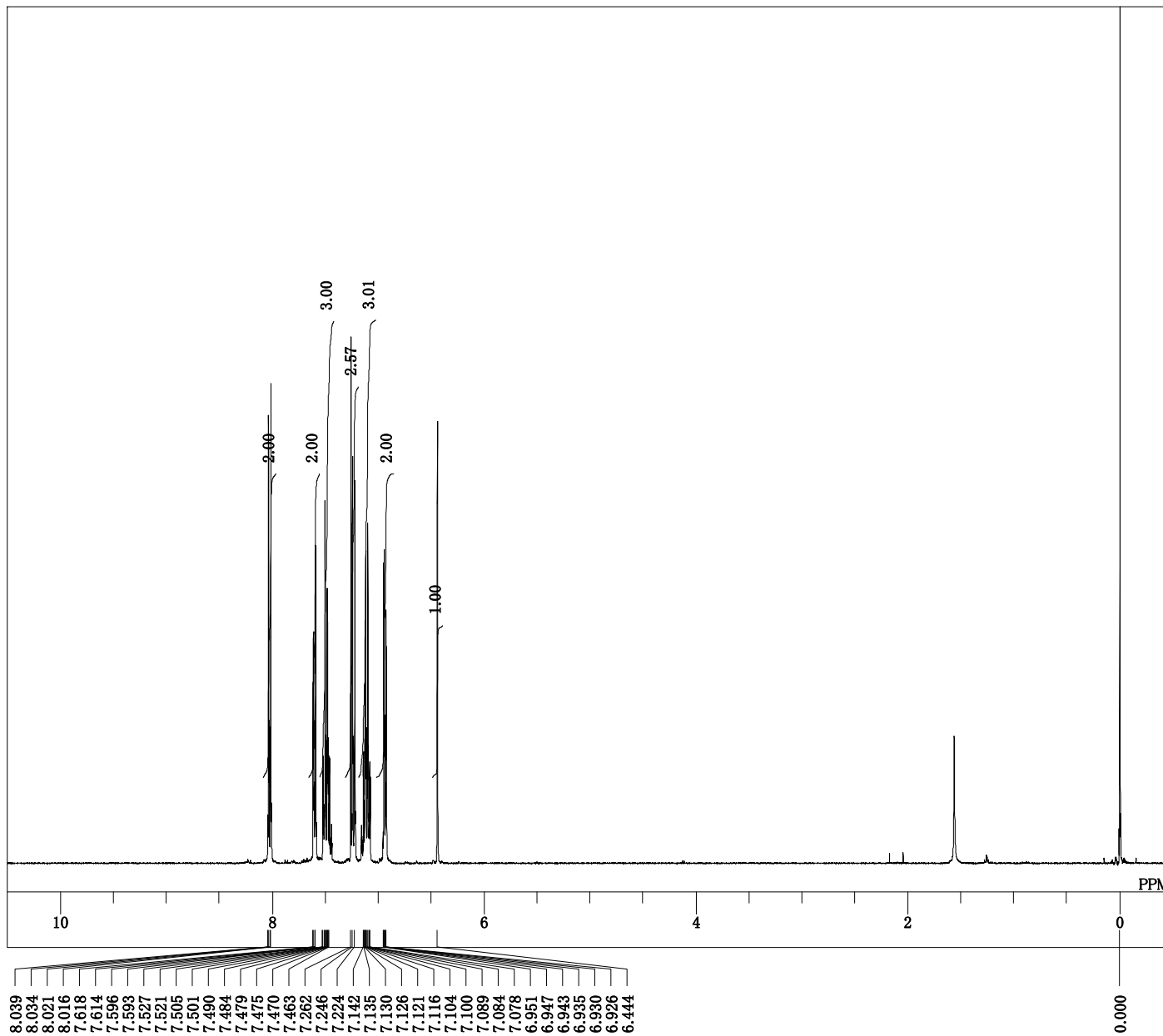
DFILE cyc_H_HProton.als
 COMNT single_pulse
 DATIM 2020-12-21 18:23:17
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 18.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.72 Hz
 RGAIN 32



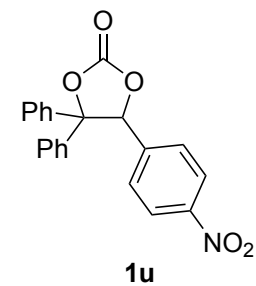


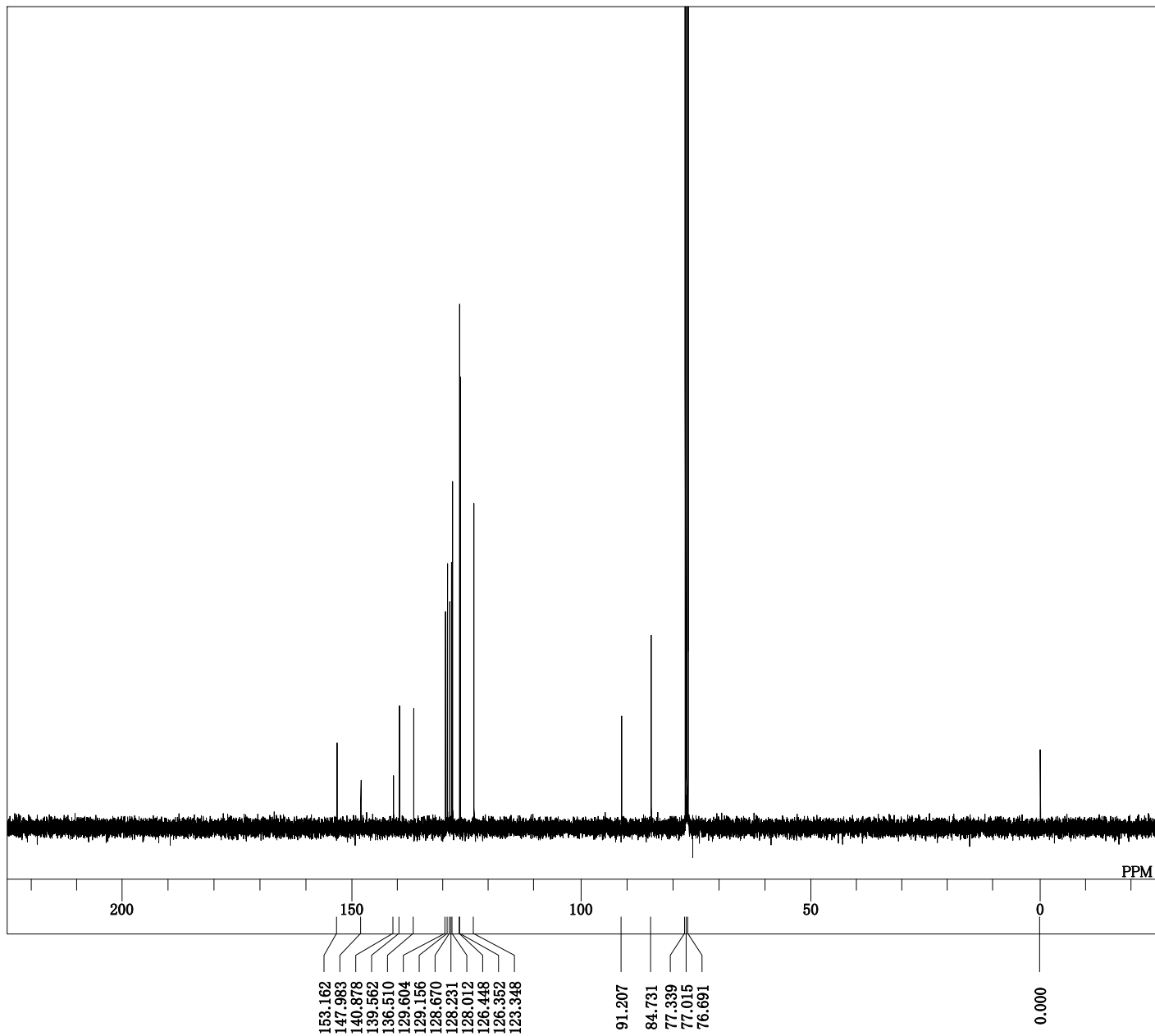
DFILE cyc_HH_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-12-23 00:13:16
 OBNUC ¹³C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 2319
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC ¹H
 CTEMP 19.6 c
 SLVNT CDCL₃
 EXREF 0.00 ppm
 BF 0.72 Hz
 RGAIN 50



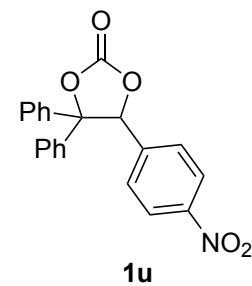


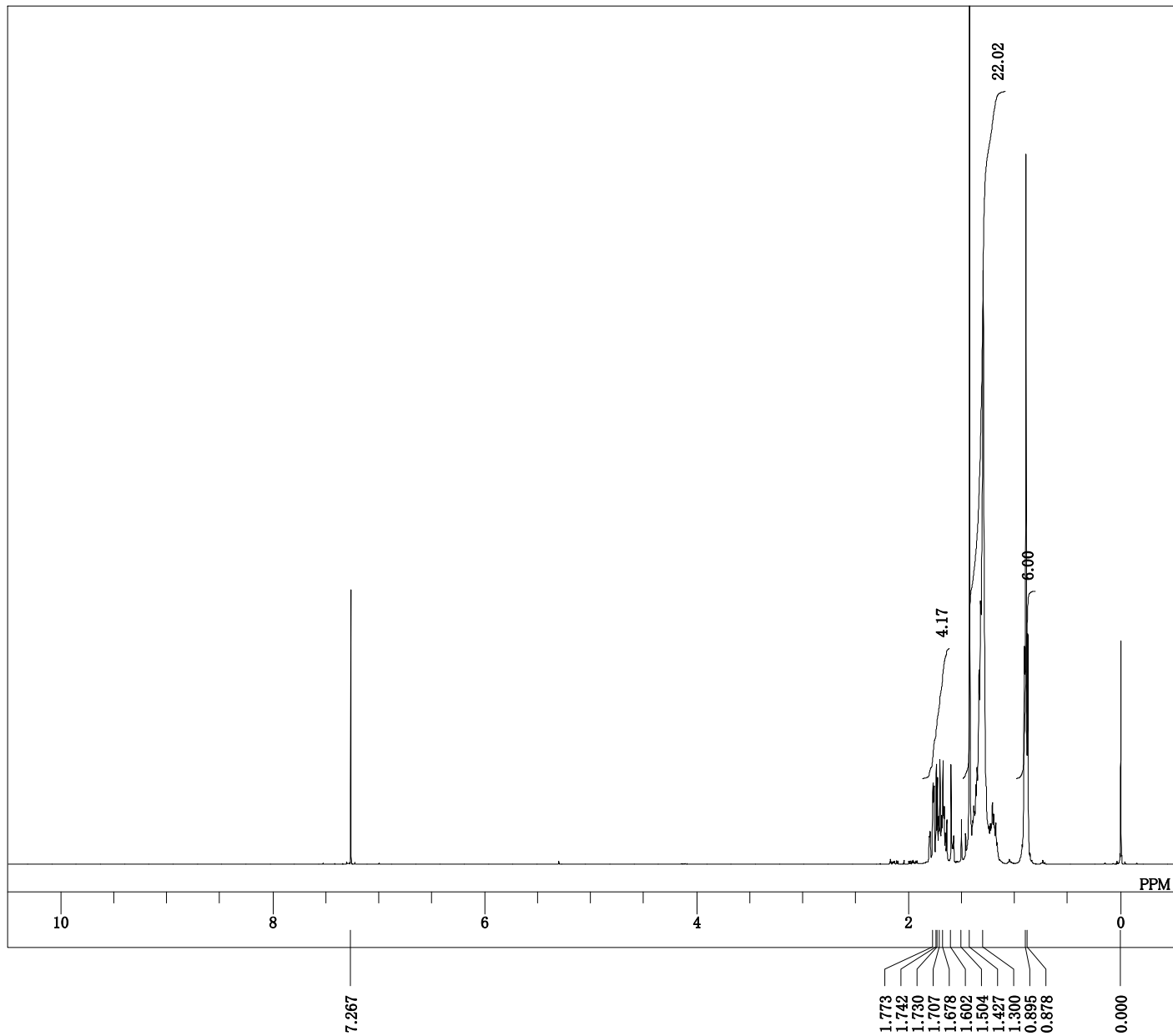
DFILE cyc_H_pNO2_Proton.als
 COMNT single_pulse
 DATIM 2020-03-28 15:33:31
 OBNUC 1H
 EXMOD single_pulse.ex2
 OBFRQ 391.78 MHz
 OBSET 8.51 KHz
 OBFIN 3.34 Hz
 POINT 26214
 FREQU 5882.26 Hz
 SCANS 16
 ACQTM 4.4564 sec
 PD 3.0000 sec
 PW1 5.90 usec
 IRNUC 1H
 CTEMP 20.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50



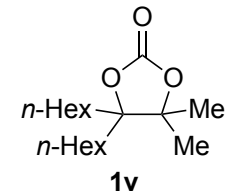


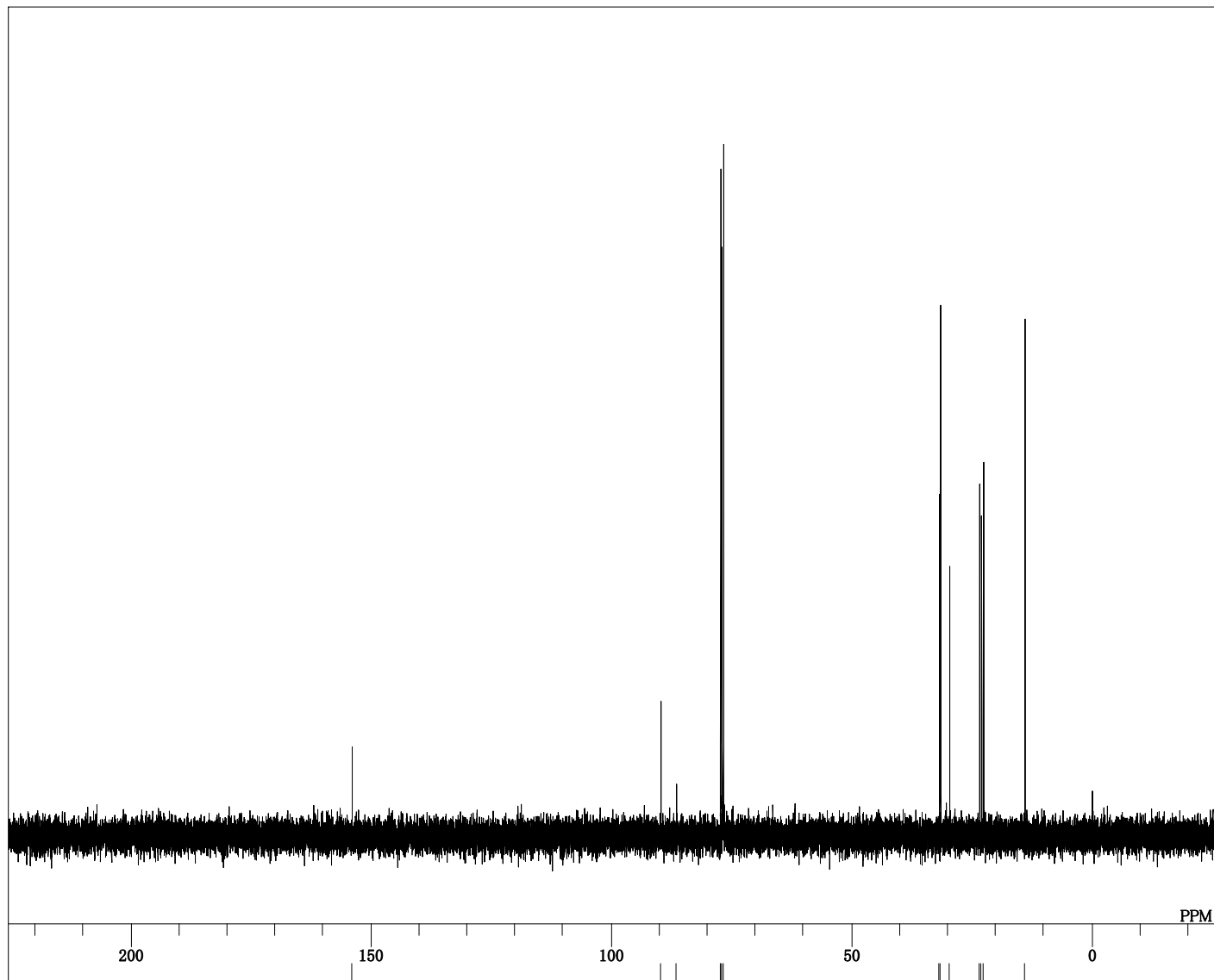
DFILE cyc_H_pNO2_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-03-28 16:28:44
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 98.52 MHz
 OBSET 4.64 KHz
 OBFIN 8.74 Hz
 POINT 26214
 FREQU 24630.17 Hz
 SCANS 1024
 ACQTM 1.0643 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 20.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50



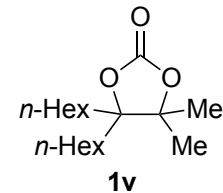


DFILE cyc_Me_Me_nHex_nHex_Proton.als
 COMNT single_pulse
 DATIM 2020-07-15 16:29:15
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 30





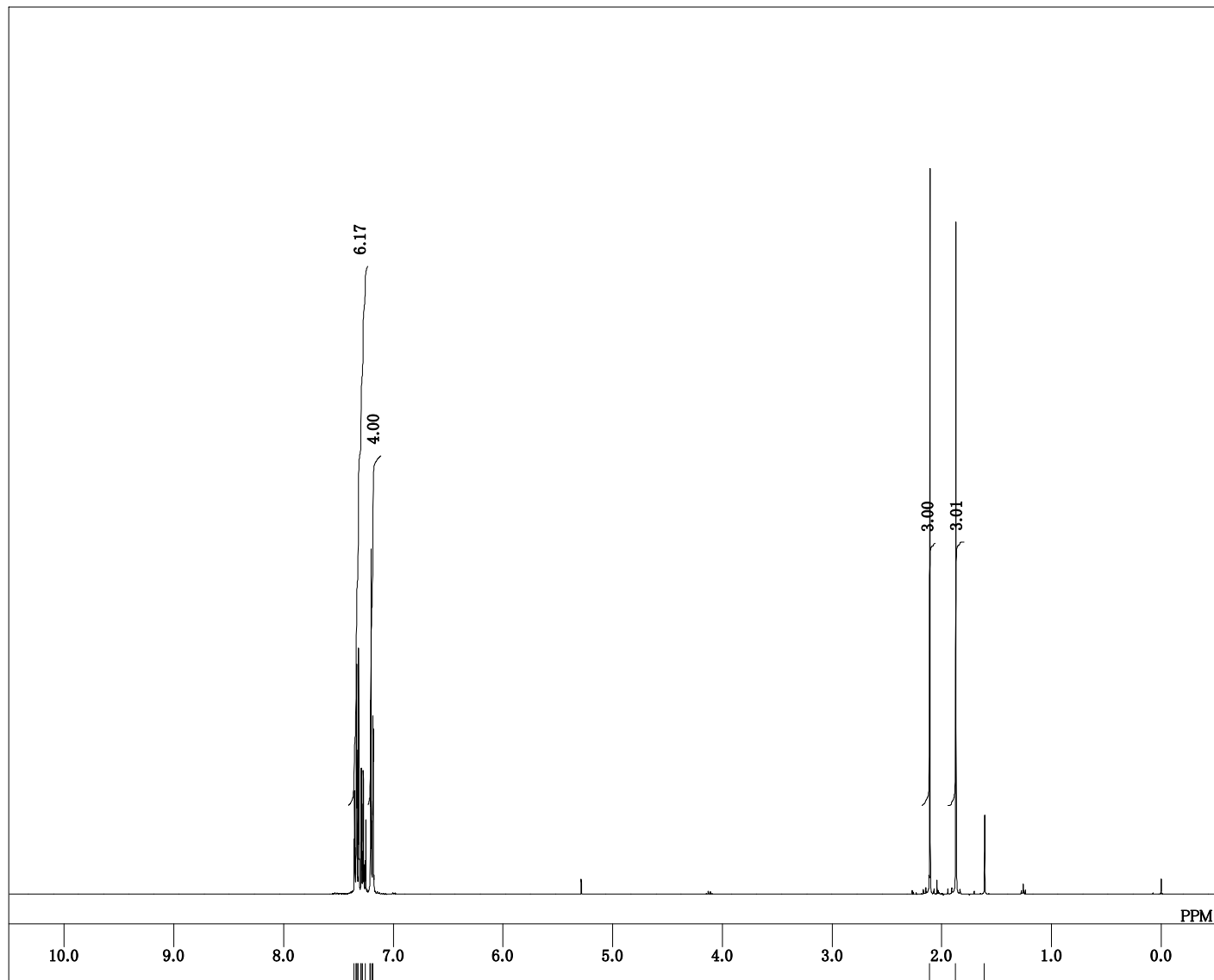
DFILE cyc_Me_Me_nHex_nHex_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-07-15 16:30:27
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 64
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 20.9 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



153.998

89.675
86.456
77.316
77.000
76.674

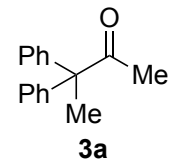
31.771
31.502
29.615
23.378
22.985
22.535
13.989

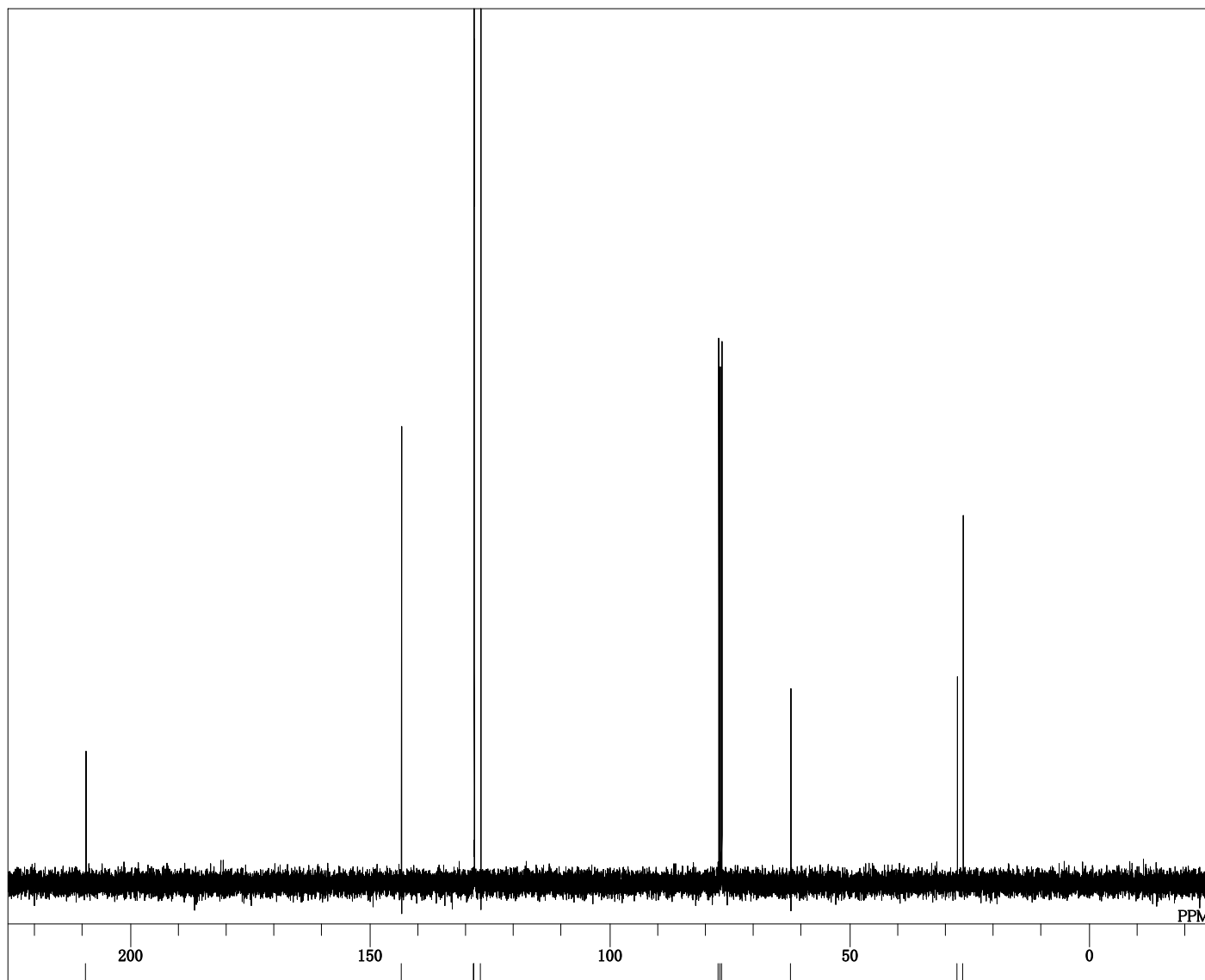


7.353
7.336
7.331
7.320
7.316
7.291
7.288
7.274
7.251
7.206
7.203
7.186
7.182

2.109
1.871
1.609

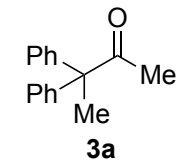
DFILE pro_diol_Me_Me_Proton.als
COMNT single_pulse
DATIM 2020-07-08 12:44:18
OBNUC 1H
EXMOD proton.jp
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.24 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 3.14 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 28





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DFILE      pro_diol_Me_Me_Carbon.als
COMNT      single pulse decoupled gated NOE
DATIM      2020-07-08 12:45:31
OBNUC      13C
EXMOD      carbon.jxp
OBFRQ      99.55 MHz
OBSET      5.13 KHz
OBFIN      0.98 Hz
POINT      26214
FREQU      25000.00 Hz
SCANS      106
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
  
```



209.239

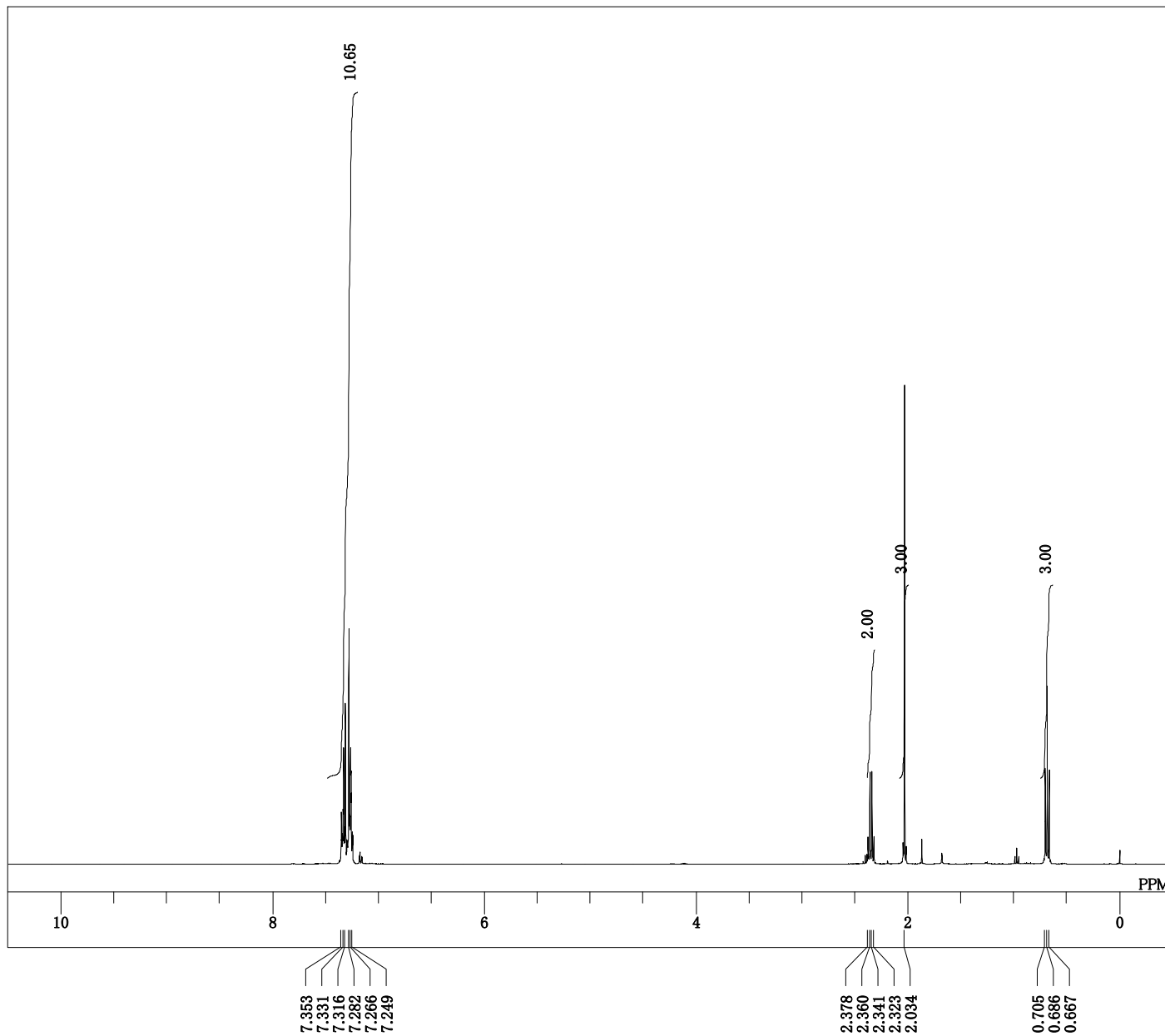
143.498

128.332
128.313
126.885

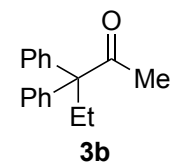
77.316
77.000
76.674

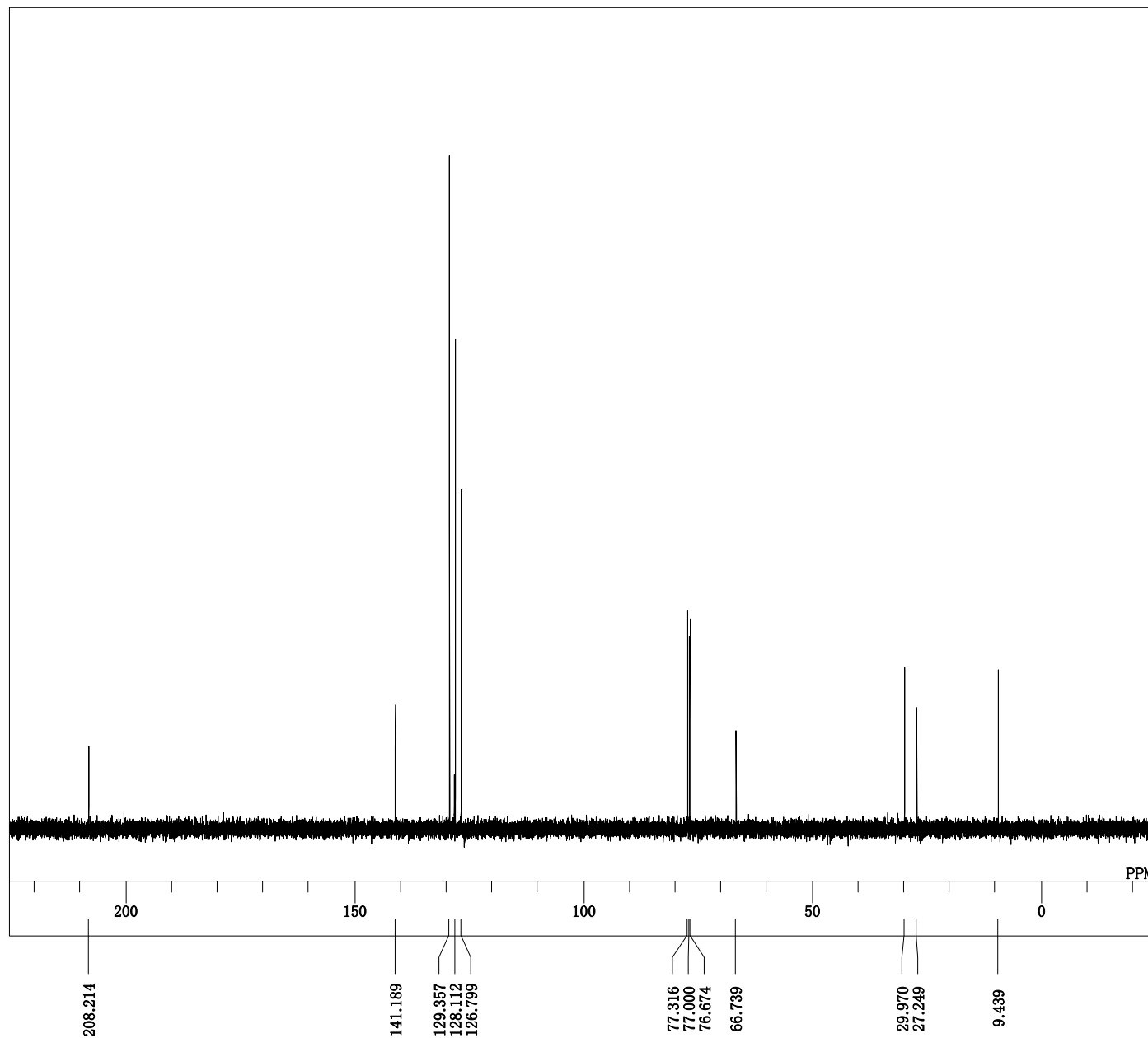
62.265

27.594
26.367

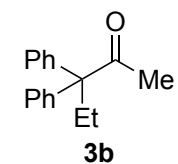


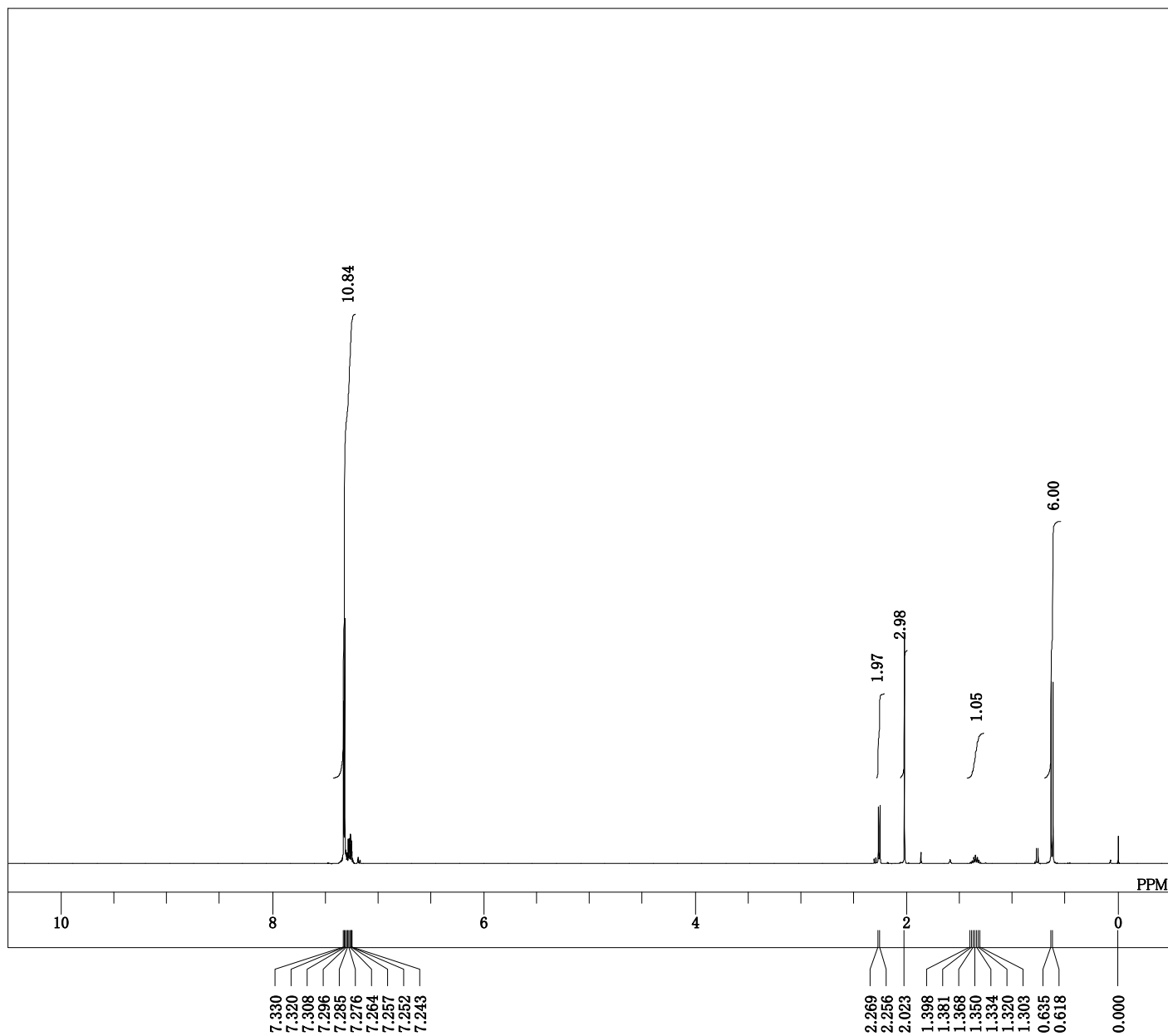
DFILE pro_diol_Me_Et_fr1_Proton.als
 COMNT single_pulse
 DATIM 2020-07-13 20:26:25
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 20



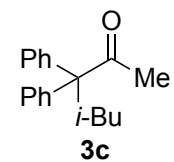


DFILE pro_diol_Me_Et_fr1_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-07-13 20:27:38
 OBNUC 13C
 EXMOD carbon_jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 17
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 20.6 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50

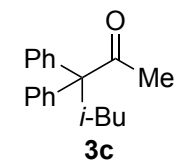
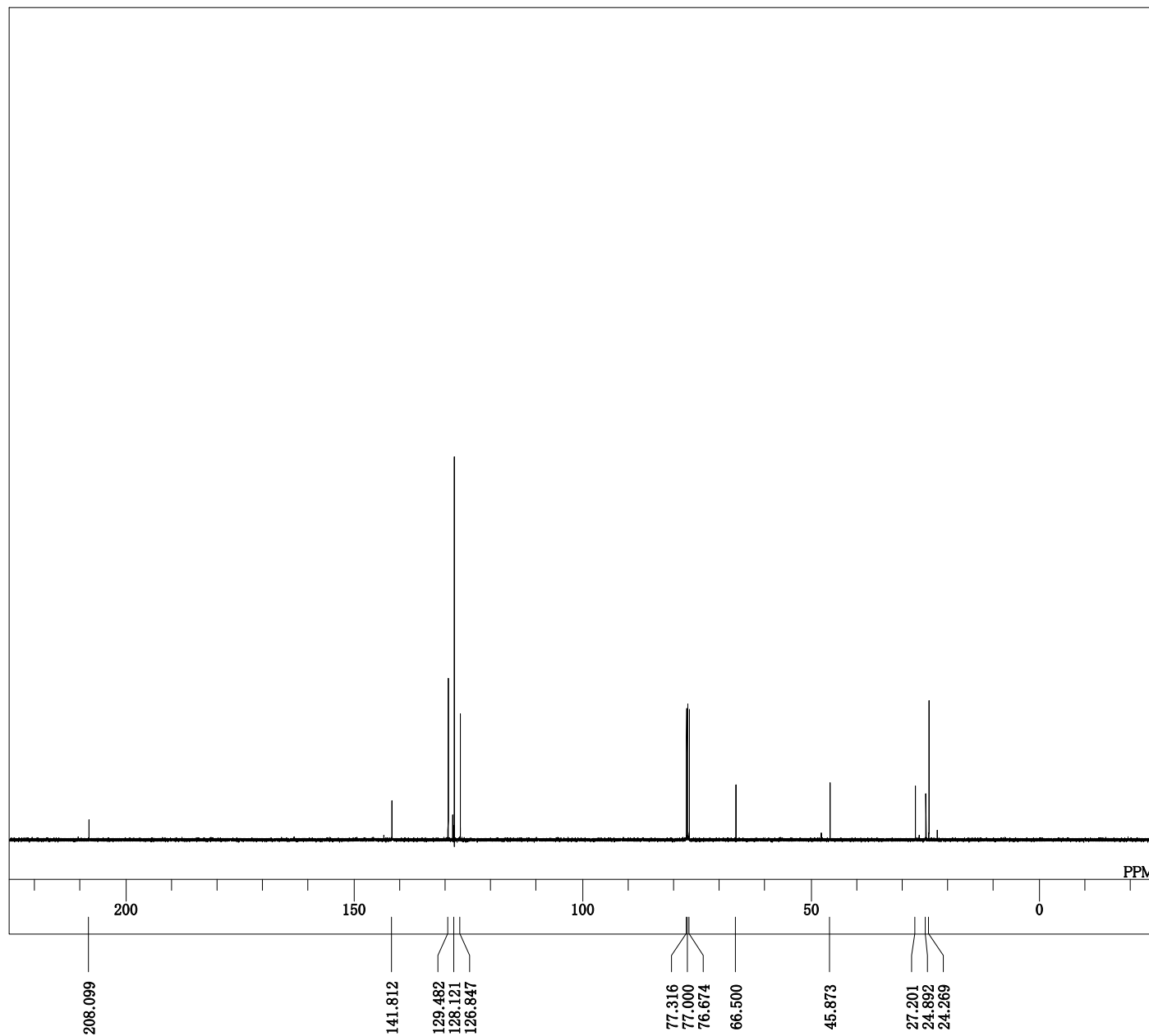




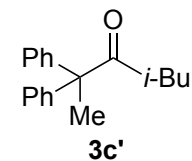
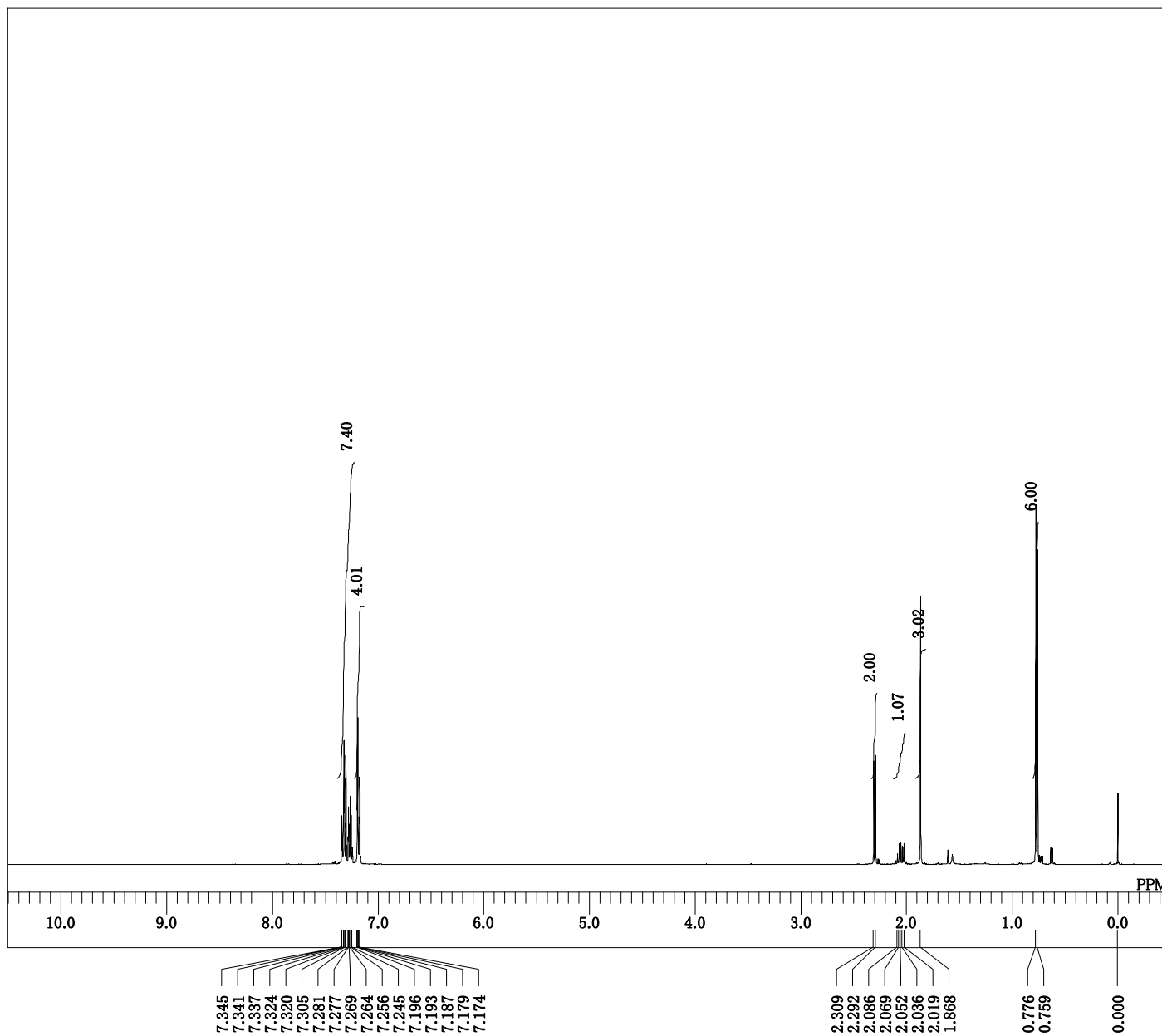
DFILE pro_diol_Me_iBu_fr2.2_Proton.als
 COMNT single_pulse
 DATIM 2020-03-31 13:17:20
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 19.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 28

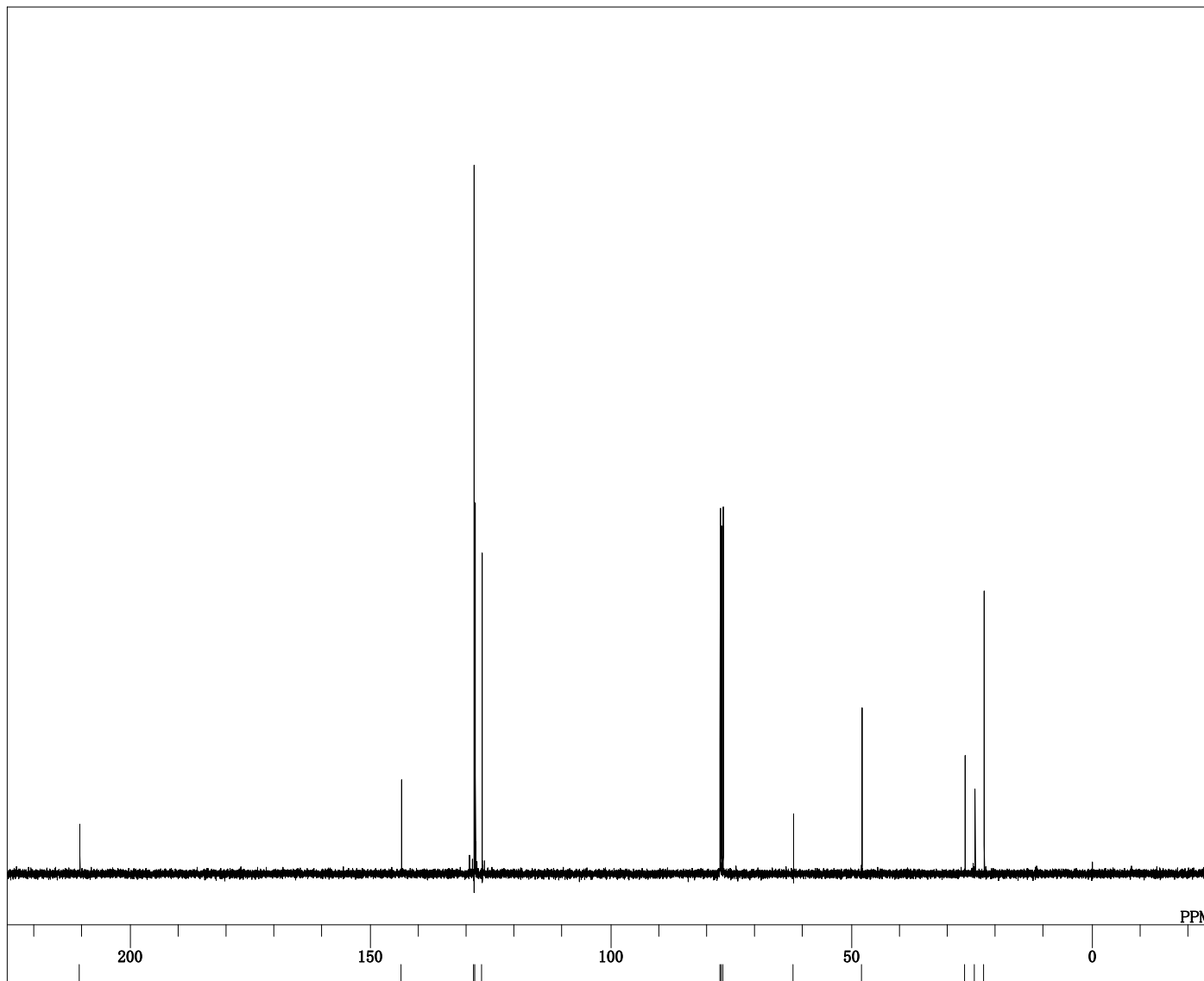


DFILE pro_diol_Me_iBu_fr2_2_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-03-31 13:18:33
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 512
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 20.2 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50

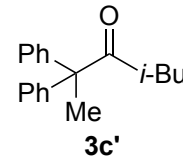


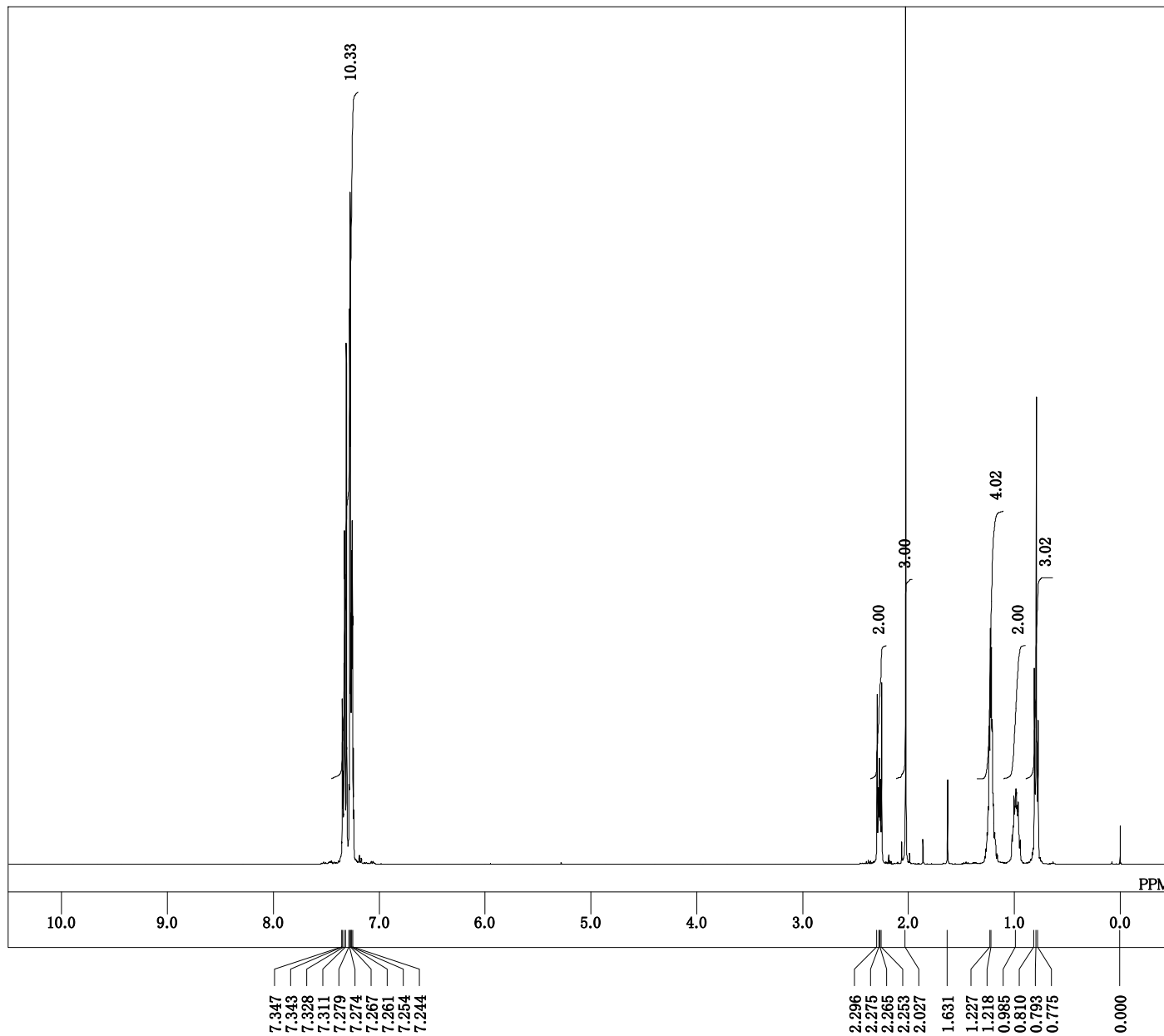
DFILE Ex616_fr2-1-Proton-1-1.als
 COMNT single_pulse
 DATIM 2020-03-31 12:45:15
 OBNUC 1H
 EXMOD proton_jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 30



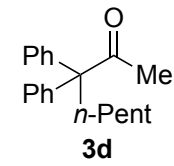


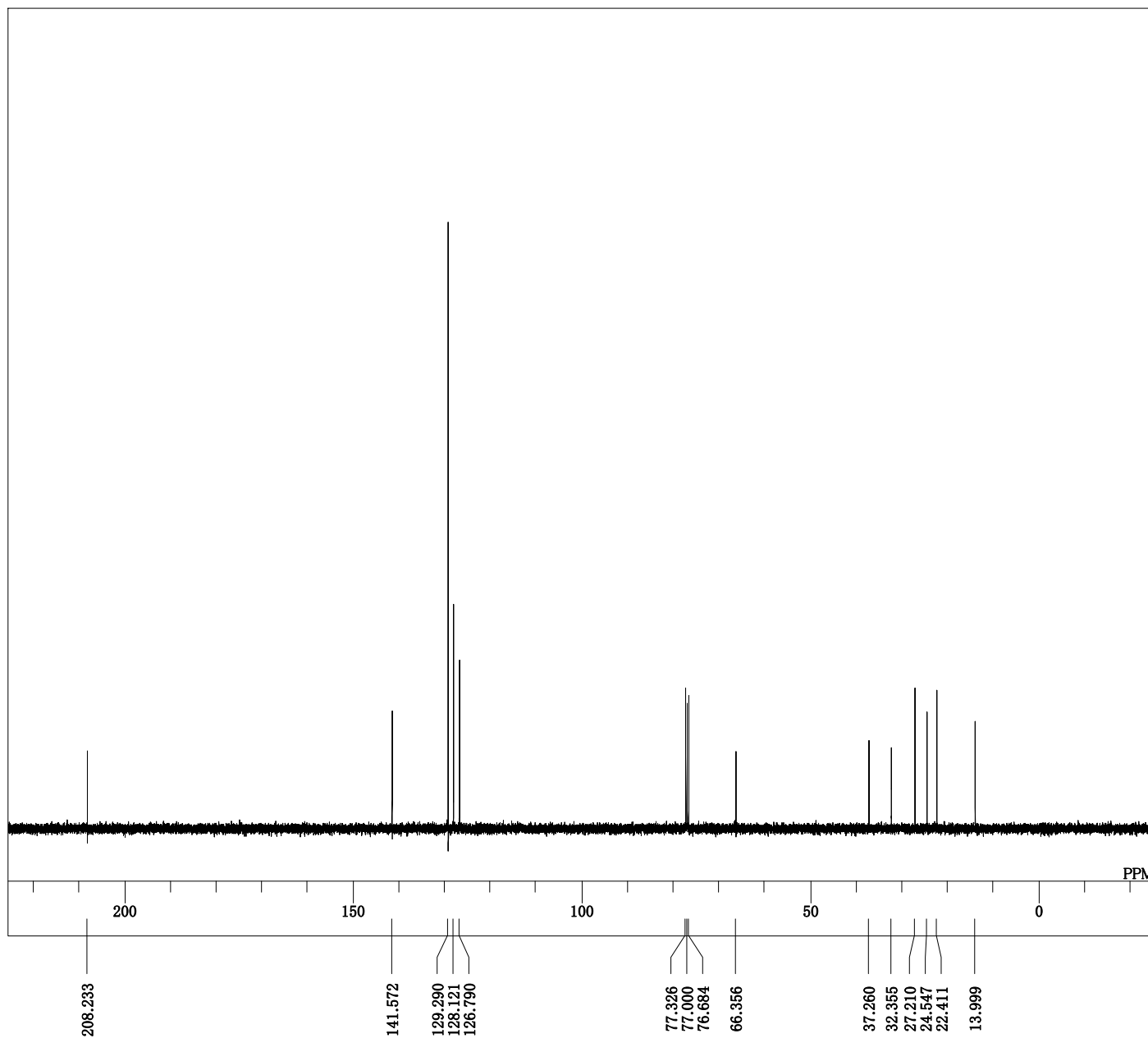
DFILE pro_diol_Me_iBu_fr2_1_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-03-31 12:46:28
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 512
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 20.0 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



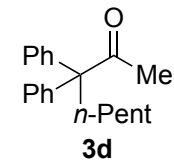


DFILE pro_diol_Me_nPentyl_fr2_Proton.als
 COMNT single_pulse
 DATIM 2020-07-05 14:23:09
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 22

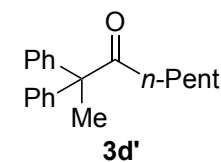
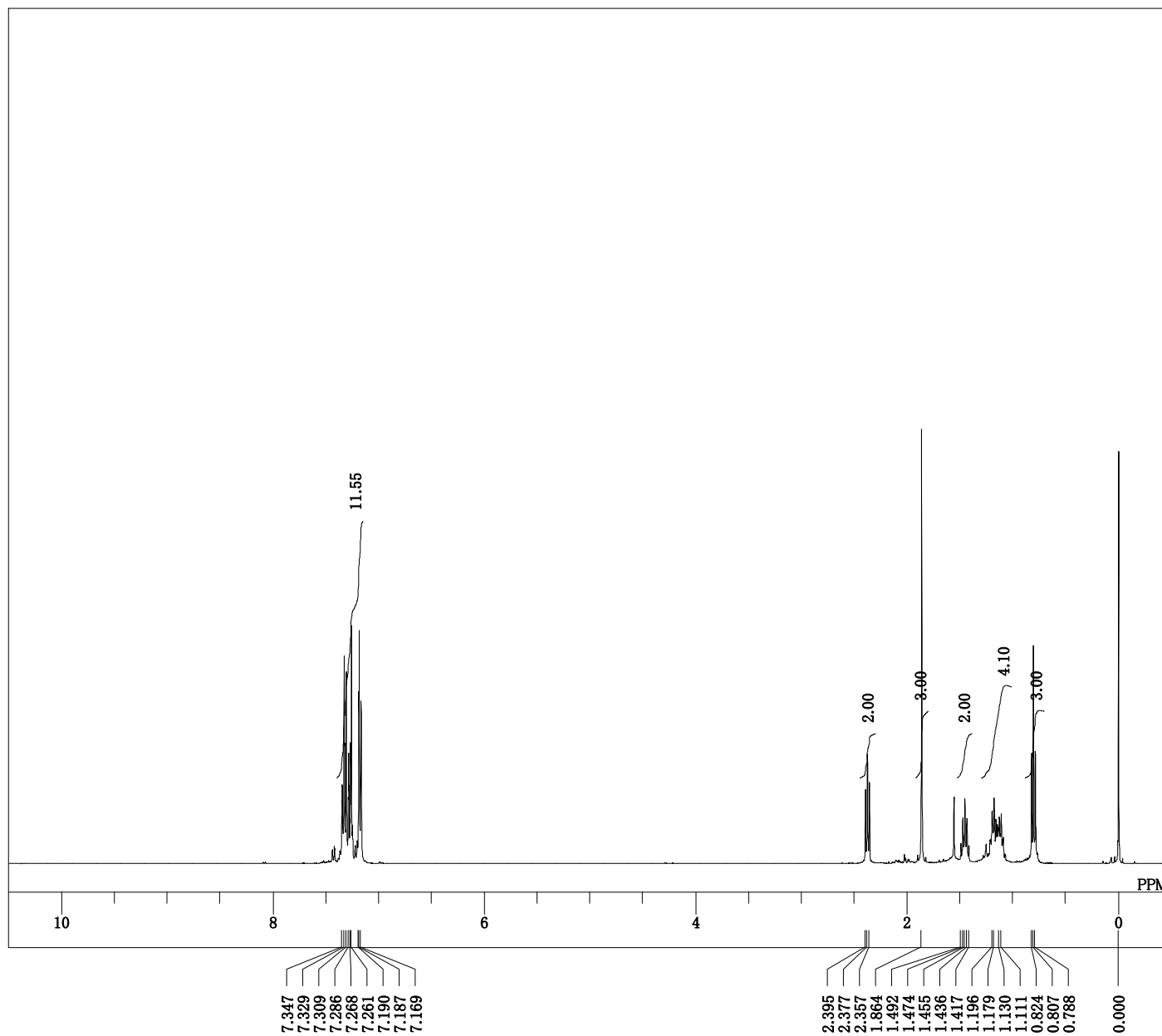


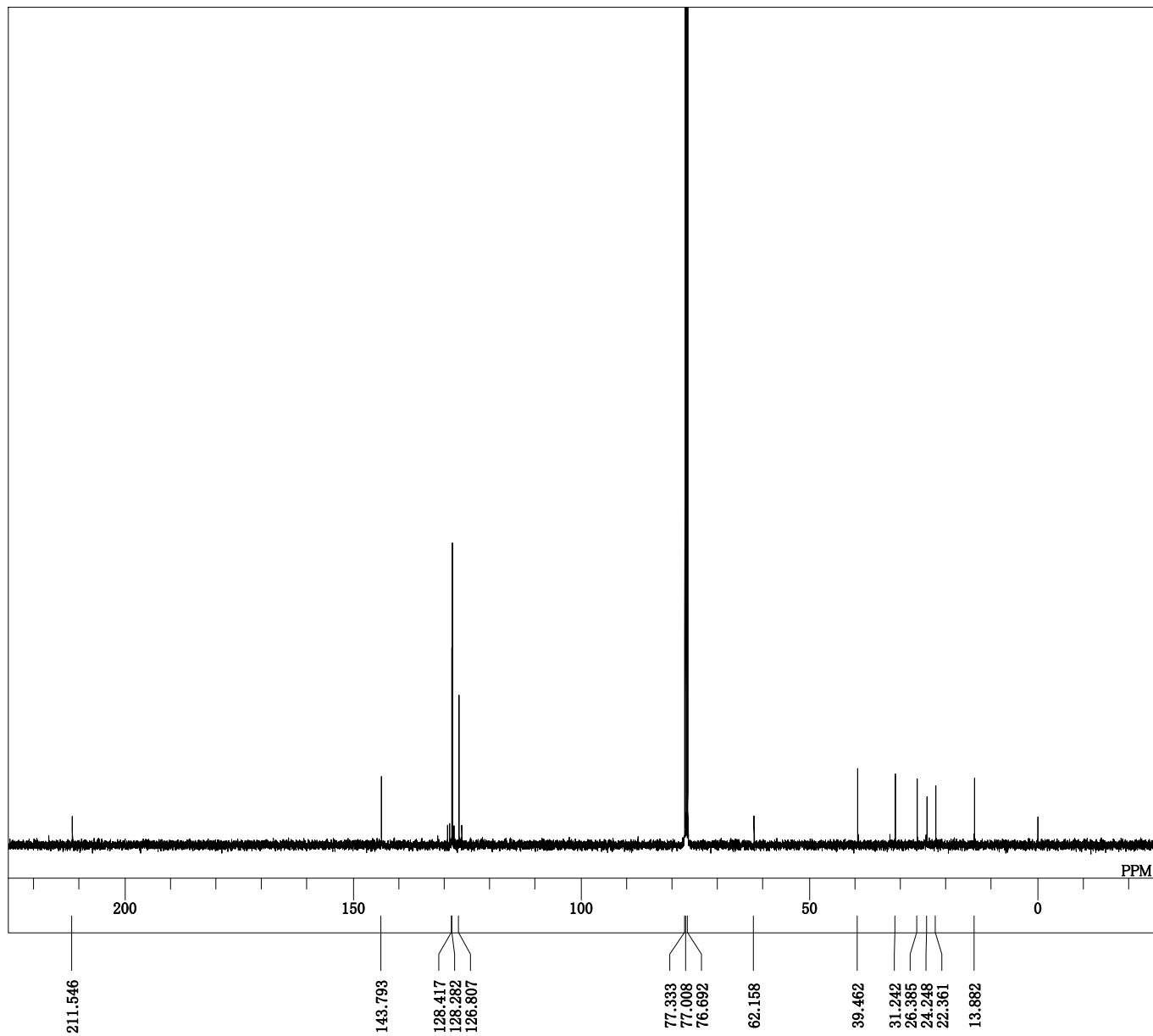


DFILE pro_diol_Me_nPentyl_fr2_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-07-05 14:24:21
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 59
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 20.4 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50

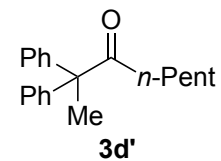


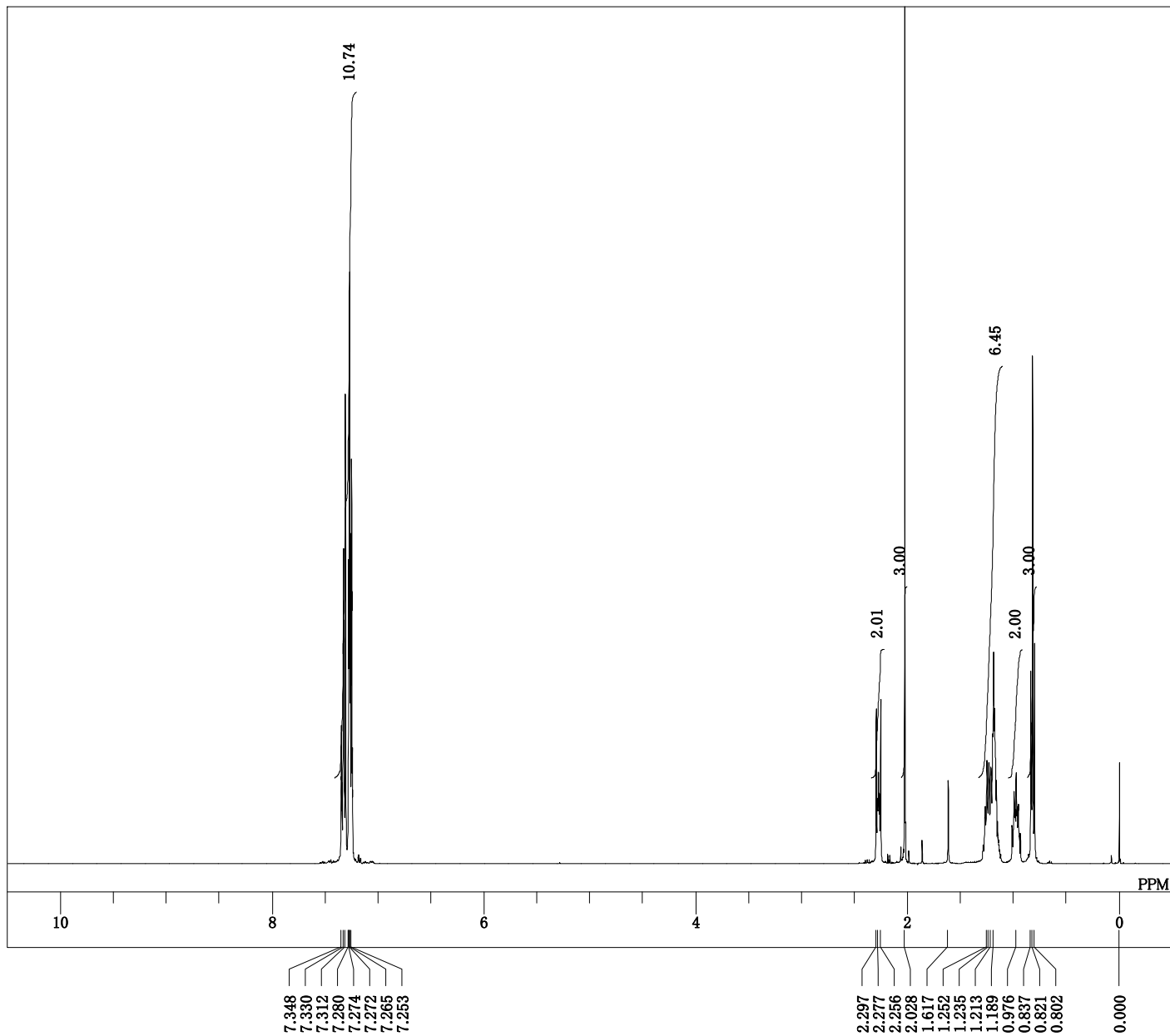
DFILE pro_diol_Me_nPentan_fr1_Proton.als
 COMNT single_pulse
 DATIM 2020-12-22 22:24:14
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 19.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.72 Hz
 RGAIN 40



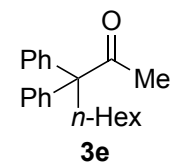


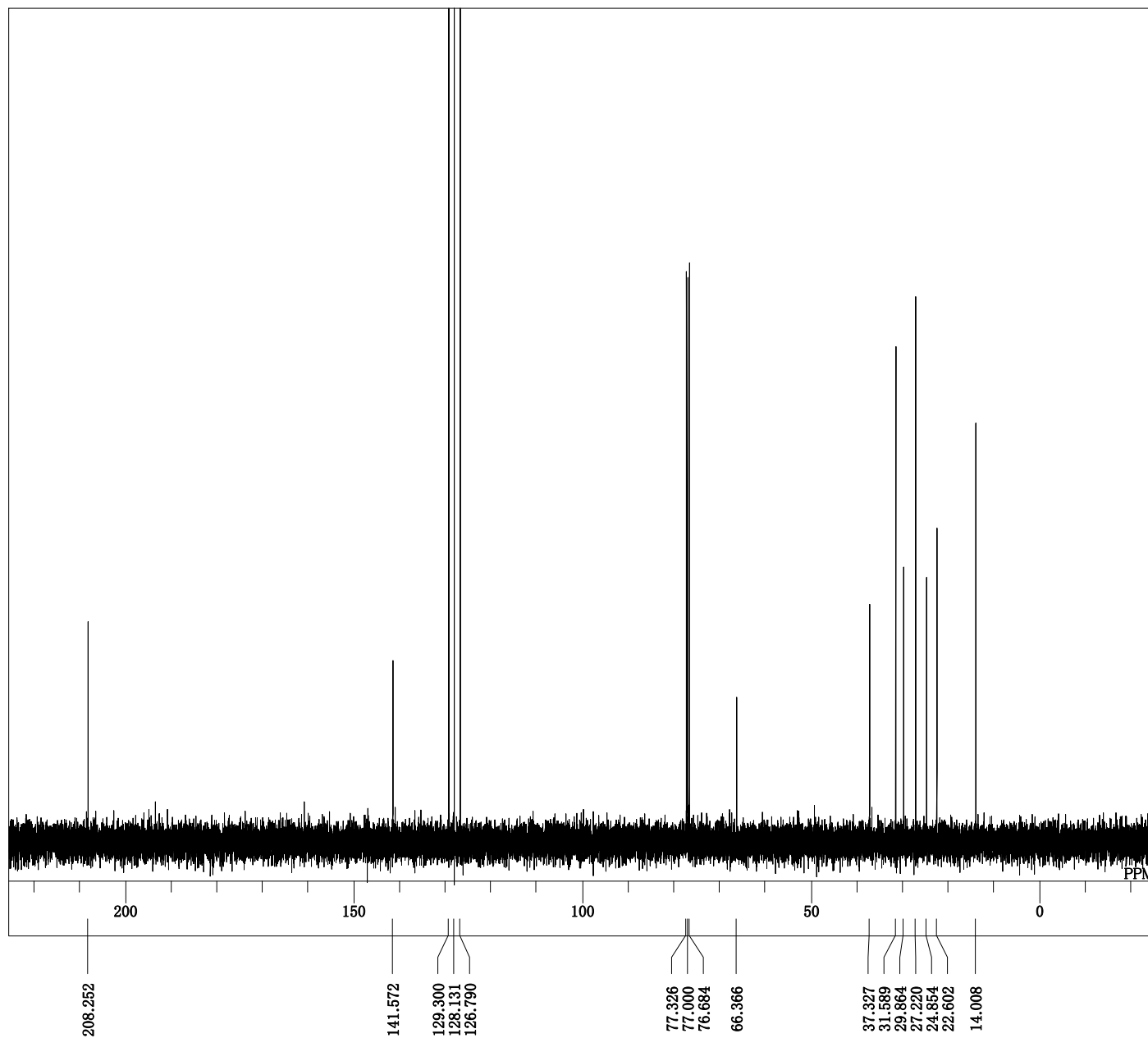
DFILE pro_diol_Me_nPentylfr1_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-12-22 22:25:27
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 1735
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 18.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.72 Hz
 RGAIN 50



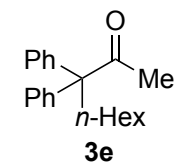


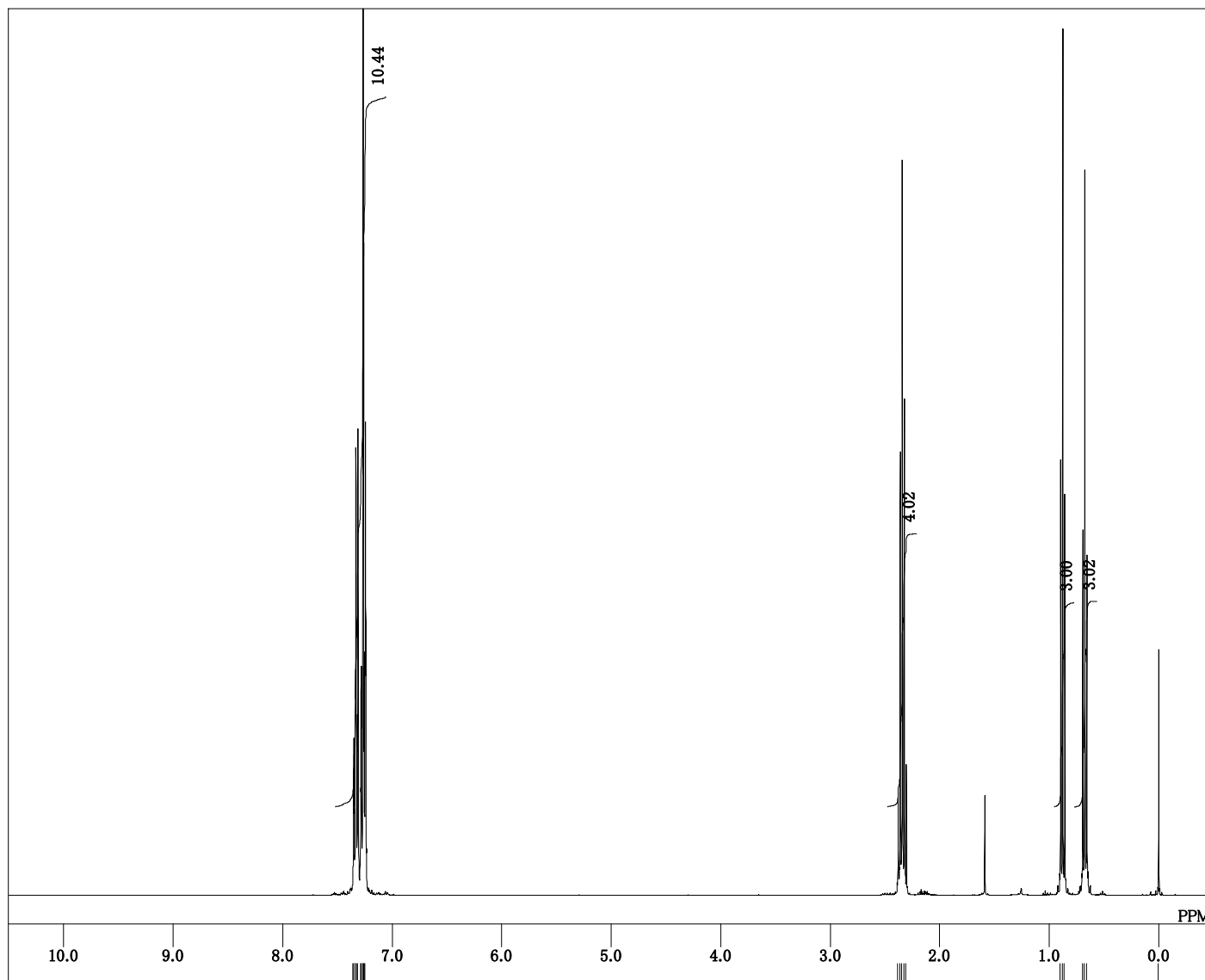
DFILE pro_diol_Me_nHexyl_fr2_Proton.als
 COMNT single_pulse
 DATIM 2020-07-15 16:38:56
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 24





DFILE pro_diol_Me_nHexyl_fr2_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-07-15 16:40:09
 OBNUC ¹³C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 31
 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





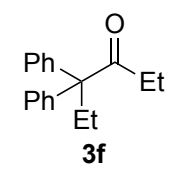
7.553
7.349
7.336
7.331
7.317
7.313
7.283
7.280
7.266
7.261
7.256
7.249
7.244

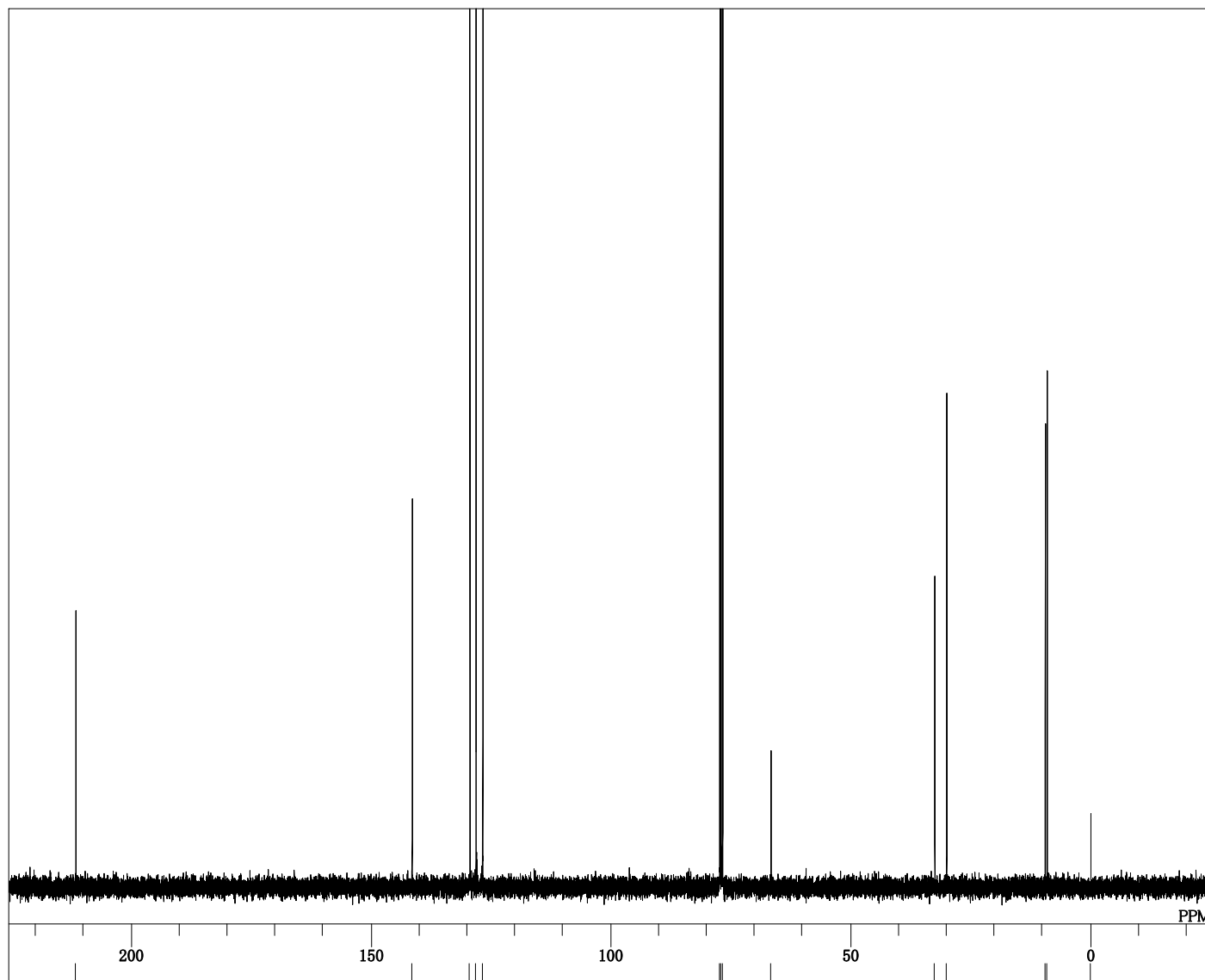
2.379
2.360
2.341
2.323
2.304

0.895
0.877
0.858
0.694
0.675
0.656

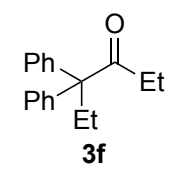
0.000

DFILE pro_diol_Et_Et_Proton.als
 COMNT single_pulse
 DATIM 2020-07-13 13:09:46
 OBNUC 1H
 EXMOD proton.jp
 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 28

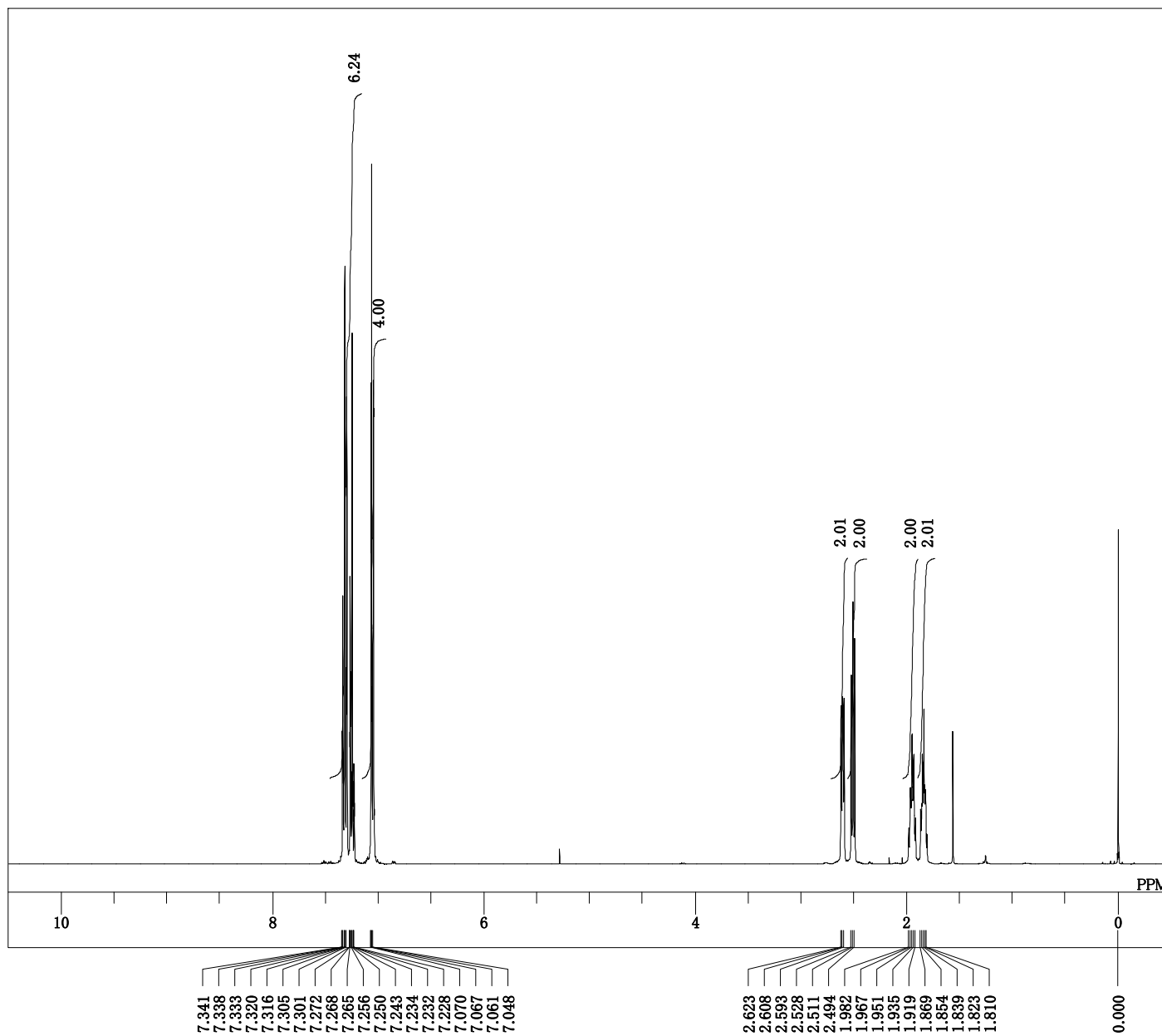




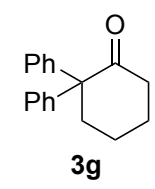
DFILE pro_diol_Et_Et_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-07-13 13:10:59
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 523
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 20.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50

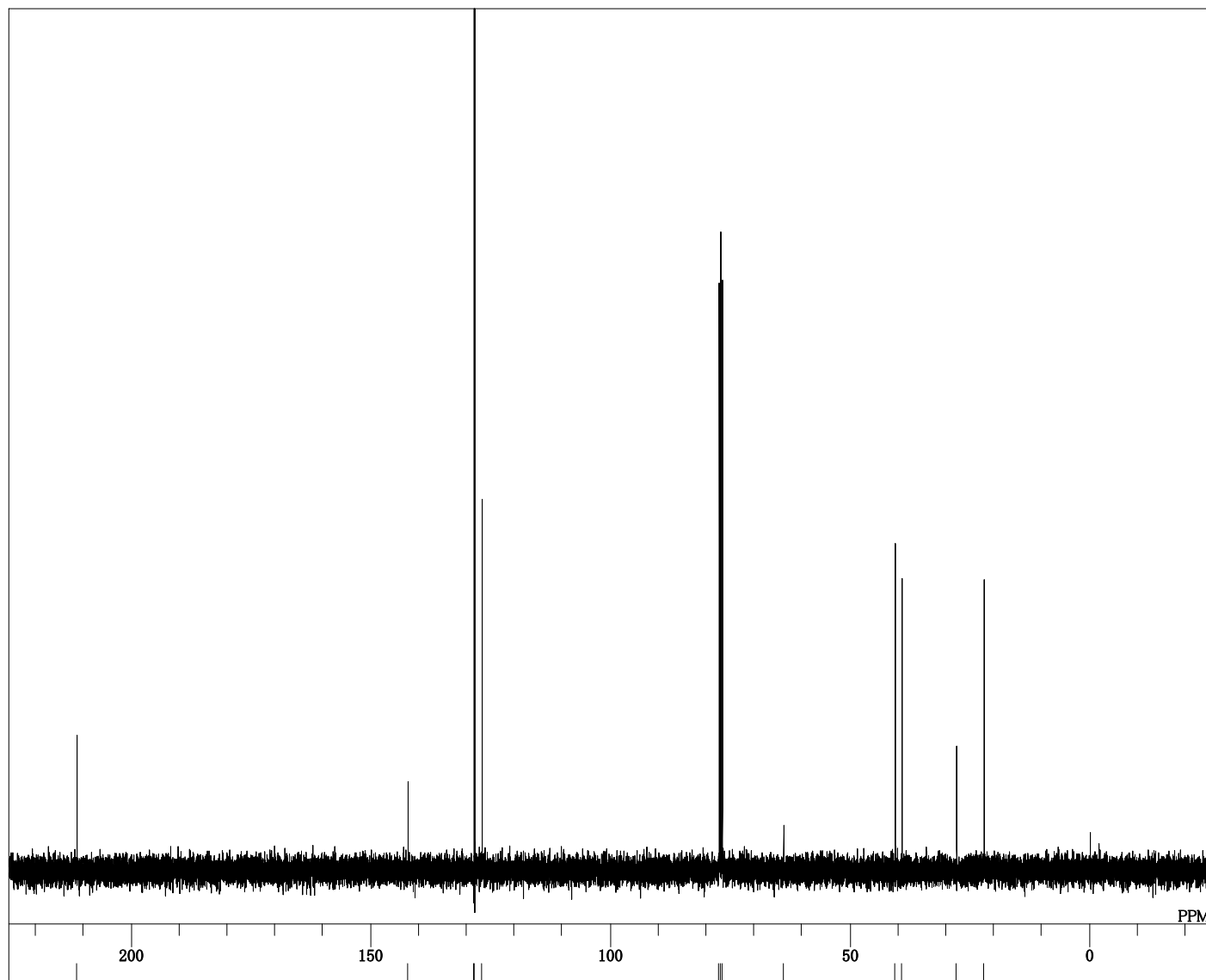


211.546
 141.494
 129.422
 128.139
 126.769
 77.343
 77.027
 76.701
 66.632
 32.478
 30.044
 9.475
 9.082
 0.000

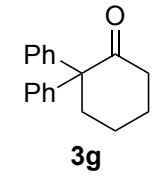


DFILE pro_diol_cyc_pentyl_Proton.als
 COMNT single_pulse
 DATIM 2020-07-13 13:42:34
 OBNUC 1H
 EXMOD proton_jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 30





DFILE pro_diol_cyc_pentyl_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-07-13 13:43:46
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 64
 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



211.337

142.262

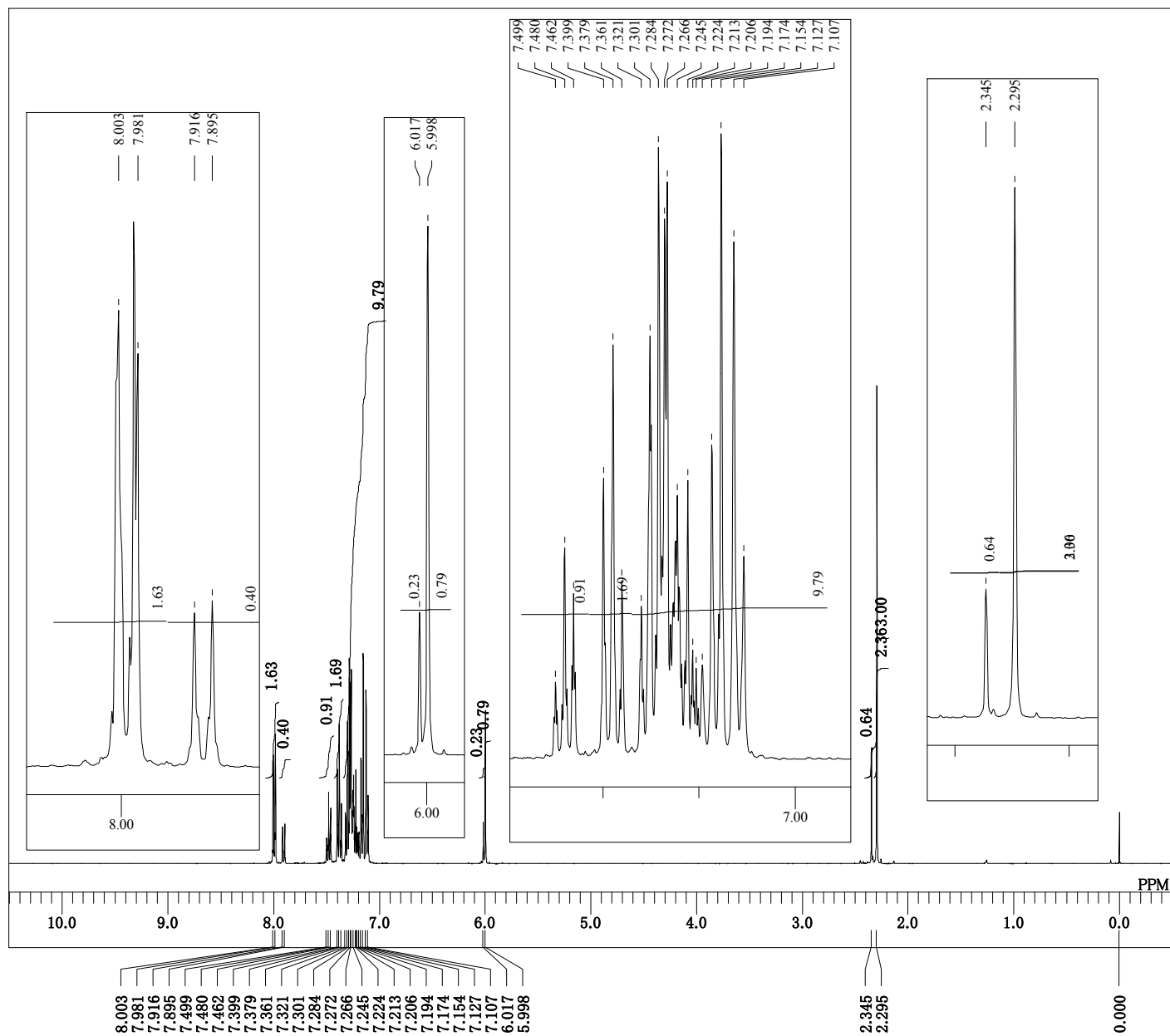
128.524
128.323
126.818

77.316
77.000
76.674

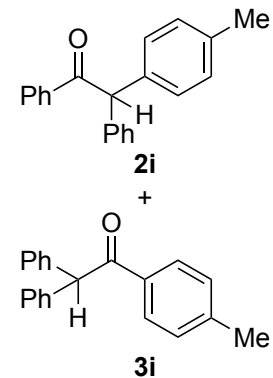
63.884

40.661
39.148

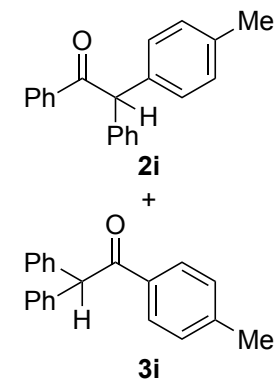
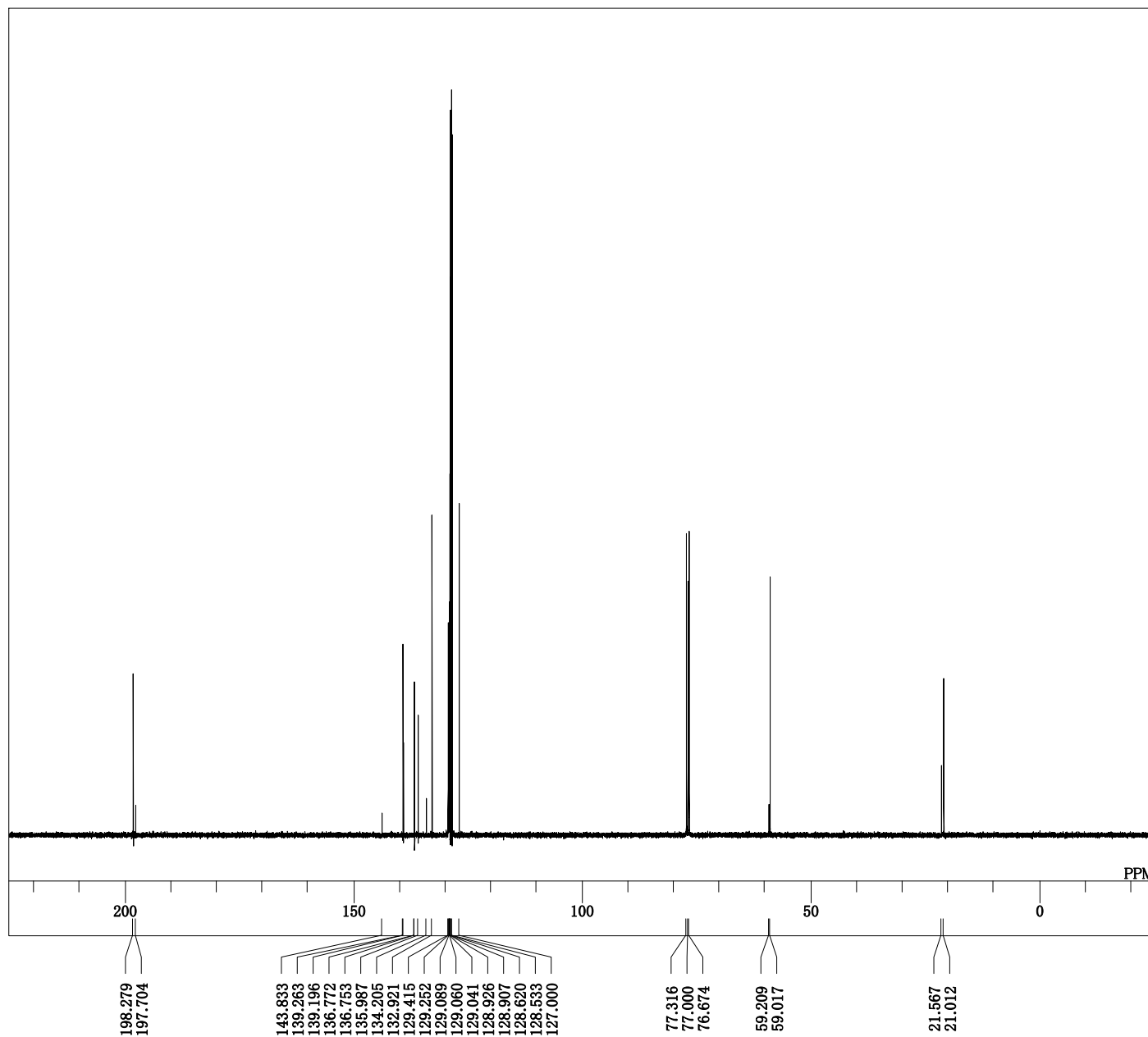
27.766
22.085

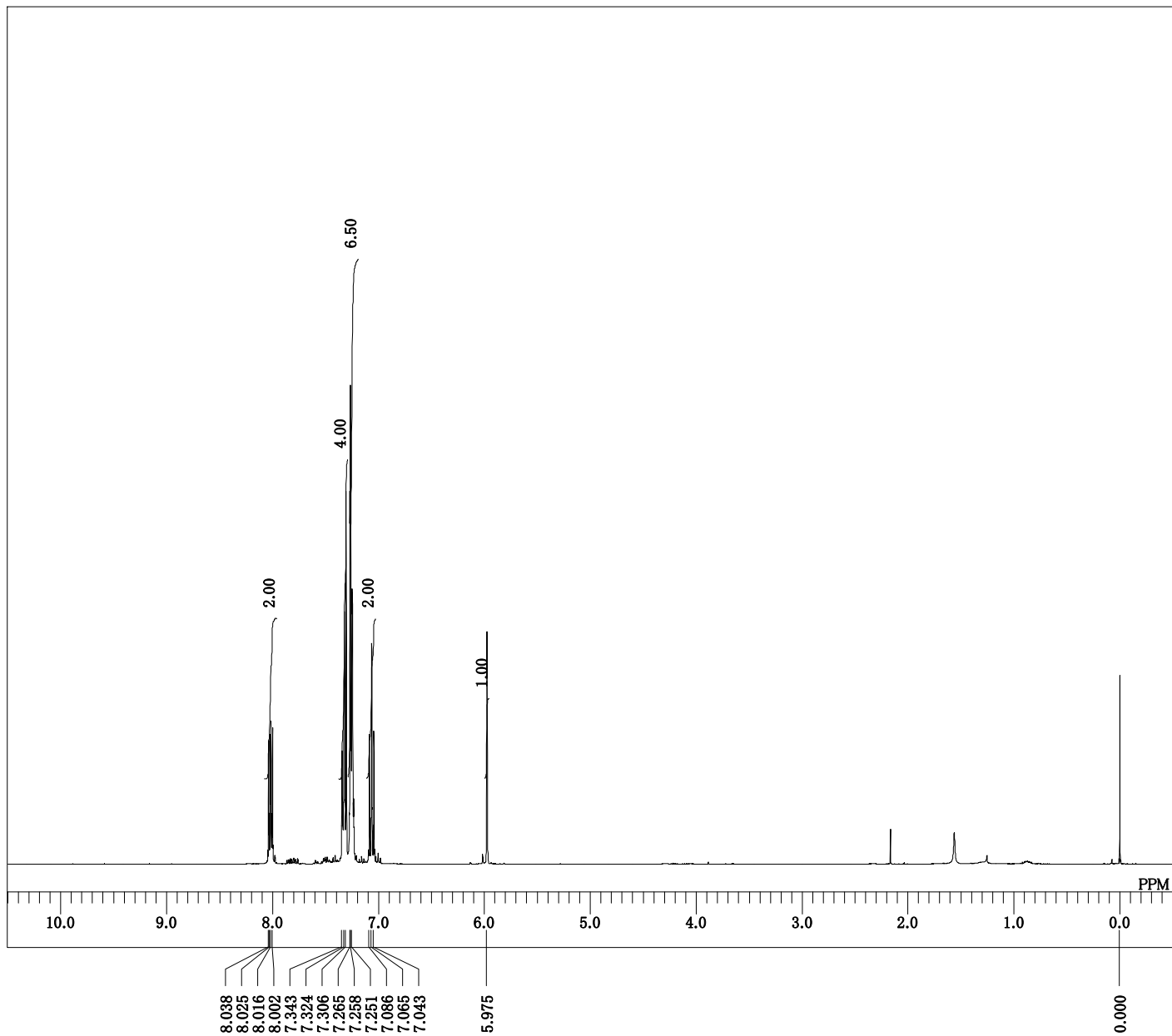


DFILE pro_diol_H_pMe.proton.als
 COMNT single_pulse
 DATIM 2021-01-12 15:15:30
 OBNUC 1H
 EXMOD proton.jp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 19.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 24

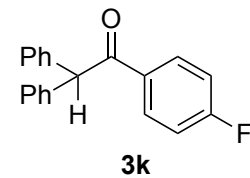


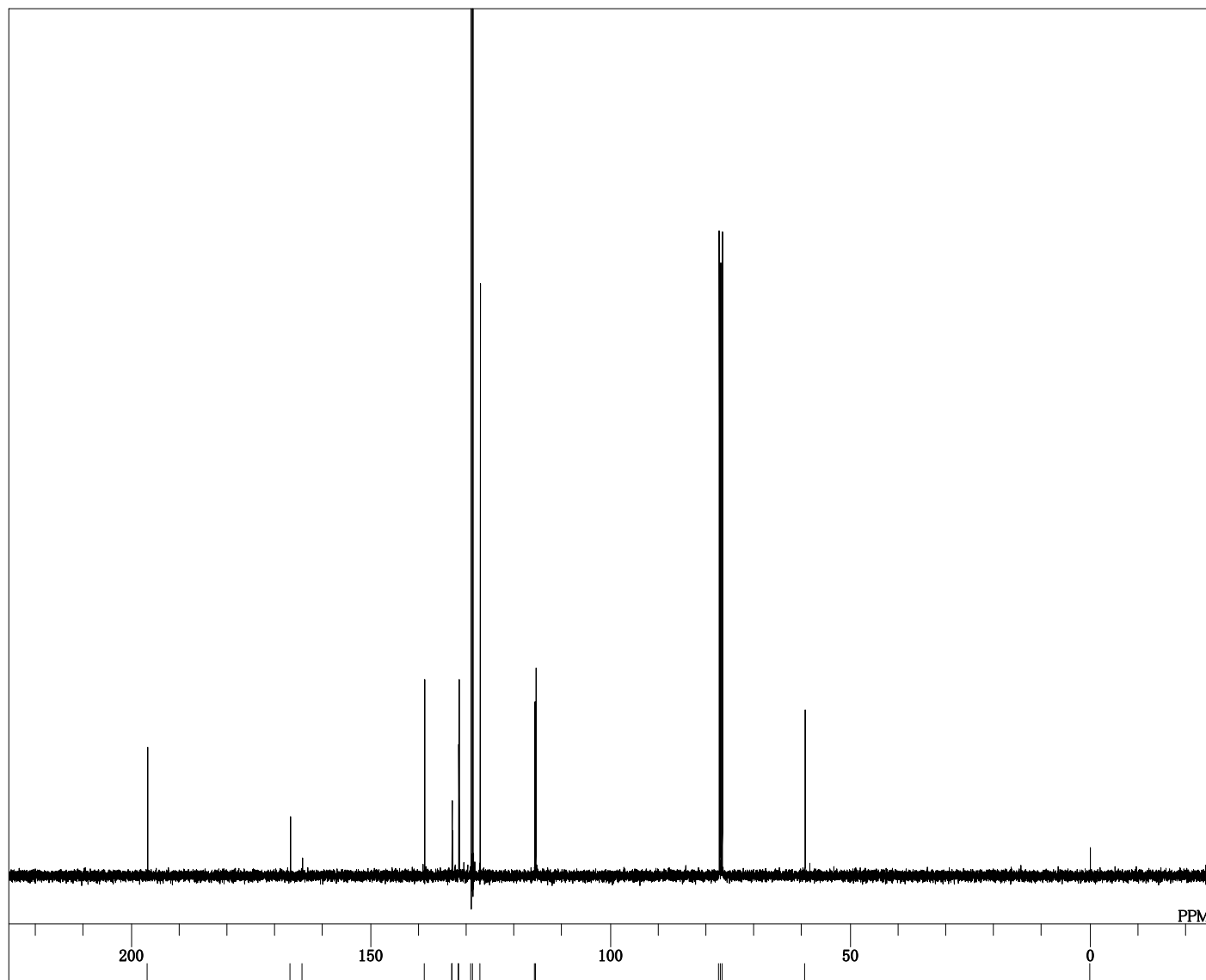
DFILE pro_diol_HpMe_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2021-01-12 15:16:43
 OBNUC ¹³C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 851
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC ¹H
 CTEMP 20.1 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50





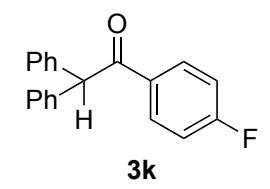
DFILE pro_diol_H_pF.fr1_Proton.als
 COMNT single_pulse
 DATIM 2020-03-28 10:44:23
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 32



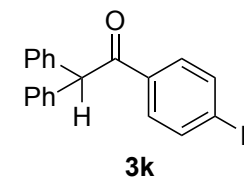
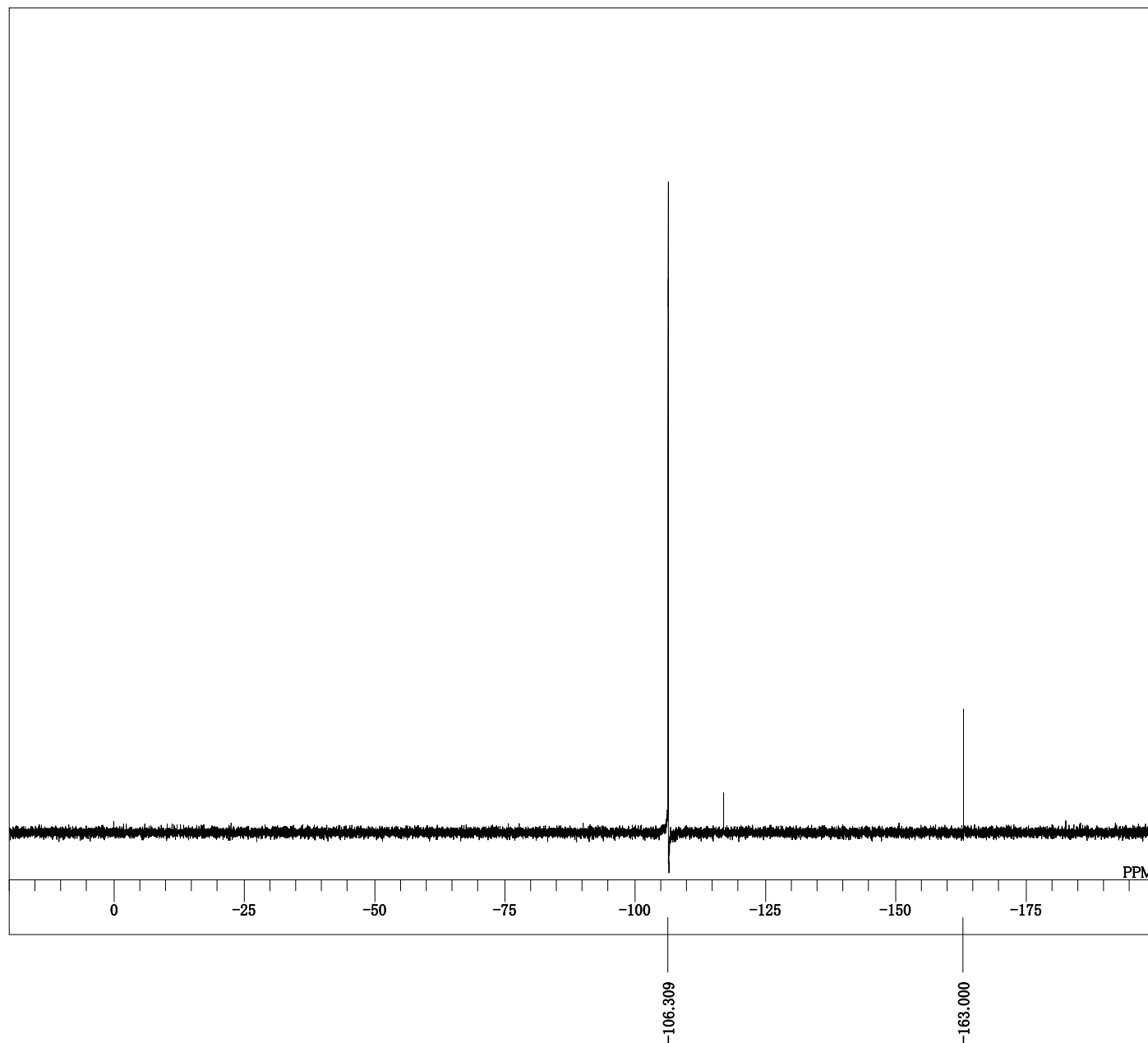


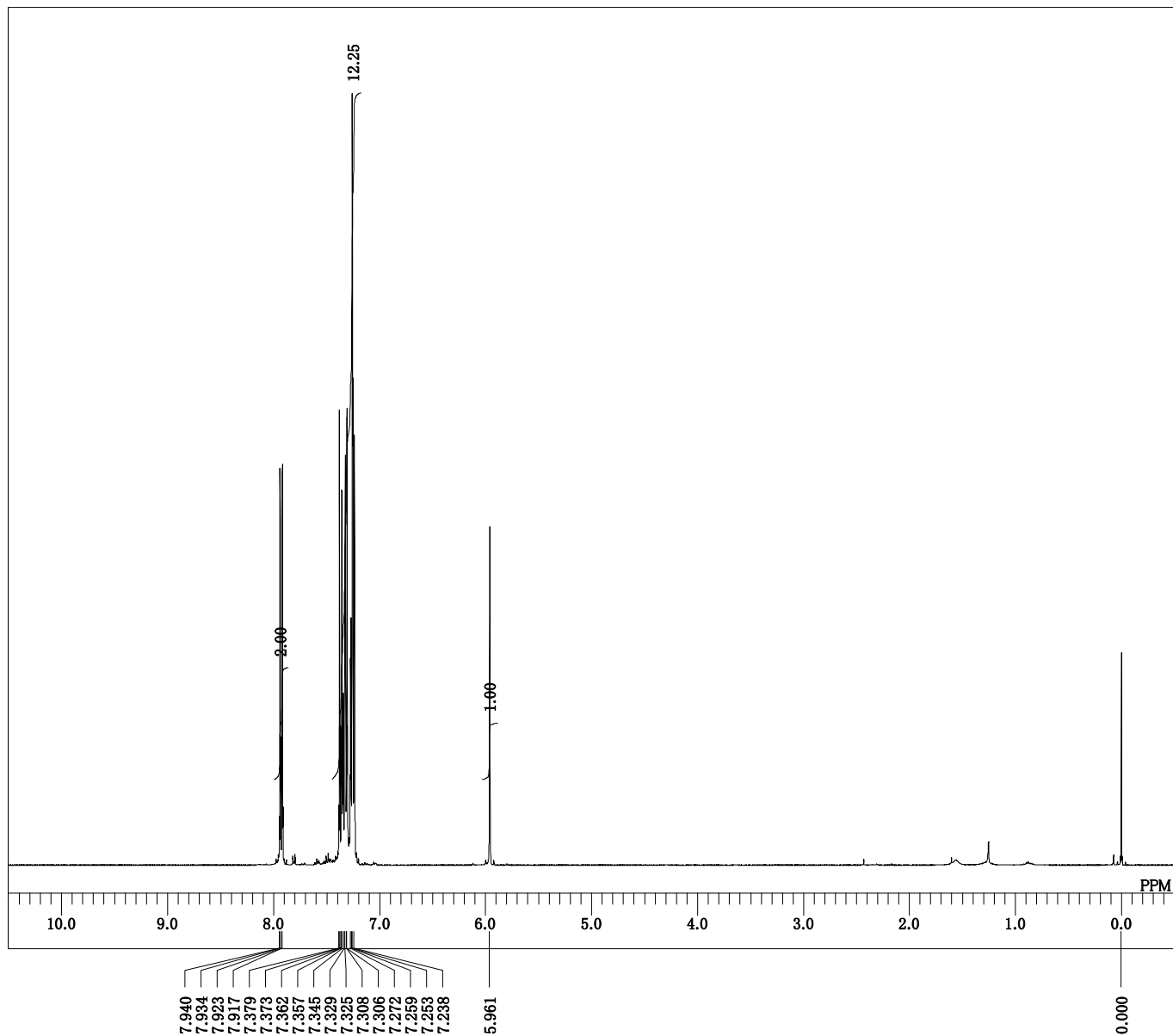
196.610
 166.863
 164.305
 138.840
 133.111
 133.082
 131.683
 131.597
 129.087
 128.781
 127.248
 115.847
 115.627
 77.343
 77.017
 76.701
 59.466
 0.000

DFILE pro_diol_H_pF_fr1_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-03-28 10:45:35
 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 20.3 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50

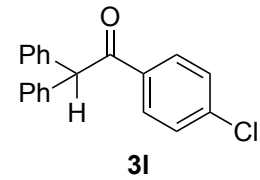


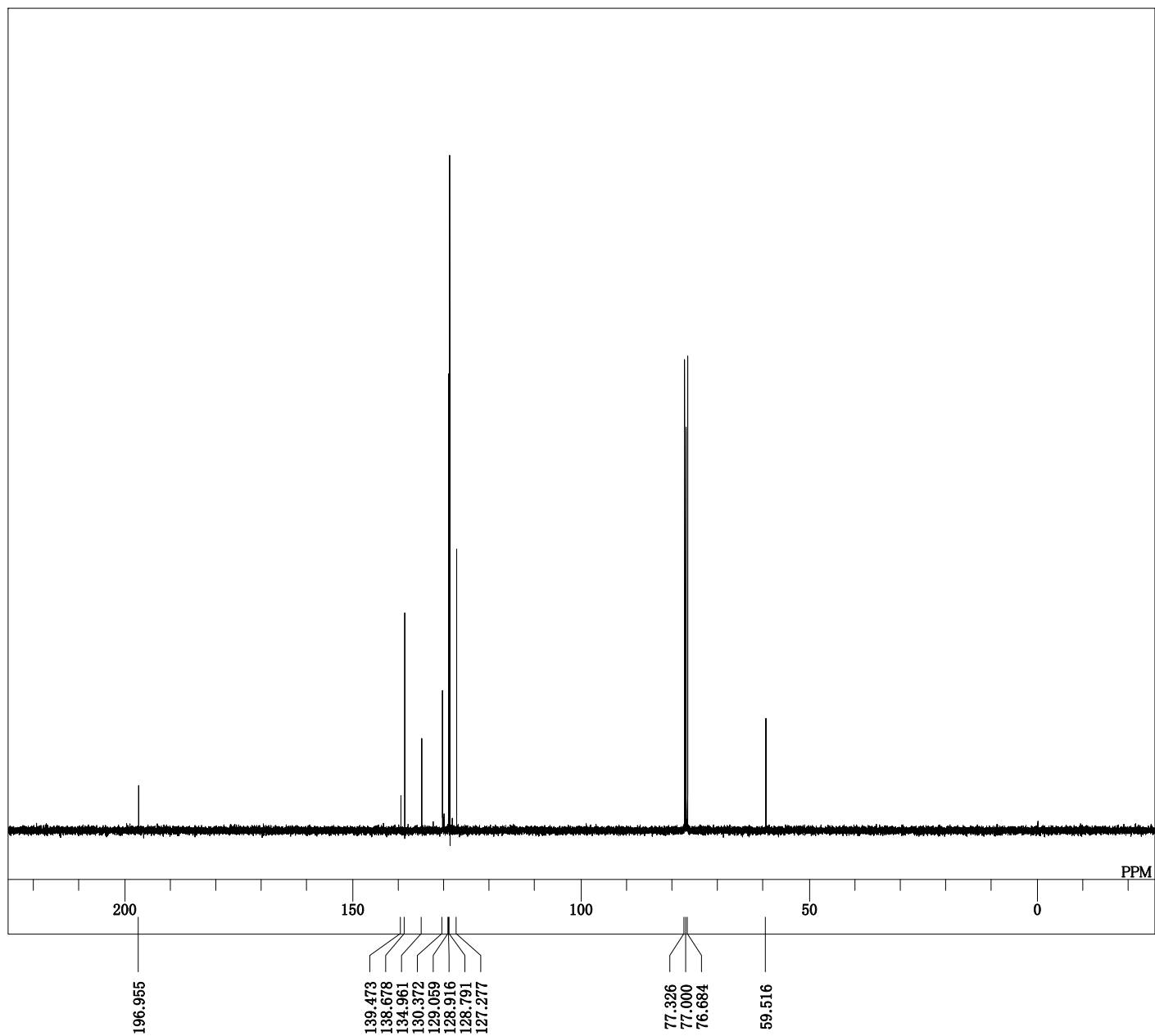
DFILE pro_diol_H_pF_Fluorine.als
COMNT single_pulse
DATIM 2022-07-04 20:52:34
OBNUC 19F
EXMOD single_pulse.jxp
OBFRQ 372.50 MHz
OBSET 3.36 KHz
OBFIN 6.86 Hz
POINT 26214
FREQU 149253.73 Hz
SCANS 8
ACQTM 0.1756 sec
PD 5.0000 sec
PW1 3.98 usec
IRNUC 19F
CTEMP 20.7 c
SLVNT CDCL3
EXREF -163.00 ppm
BF 0.12 Hz
RGAIN 44



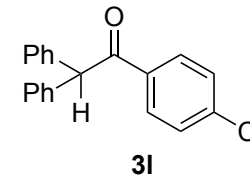


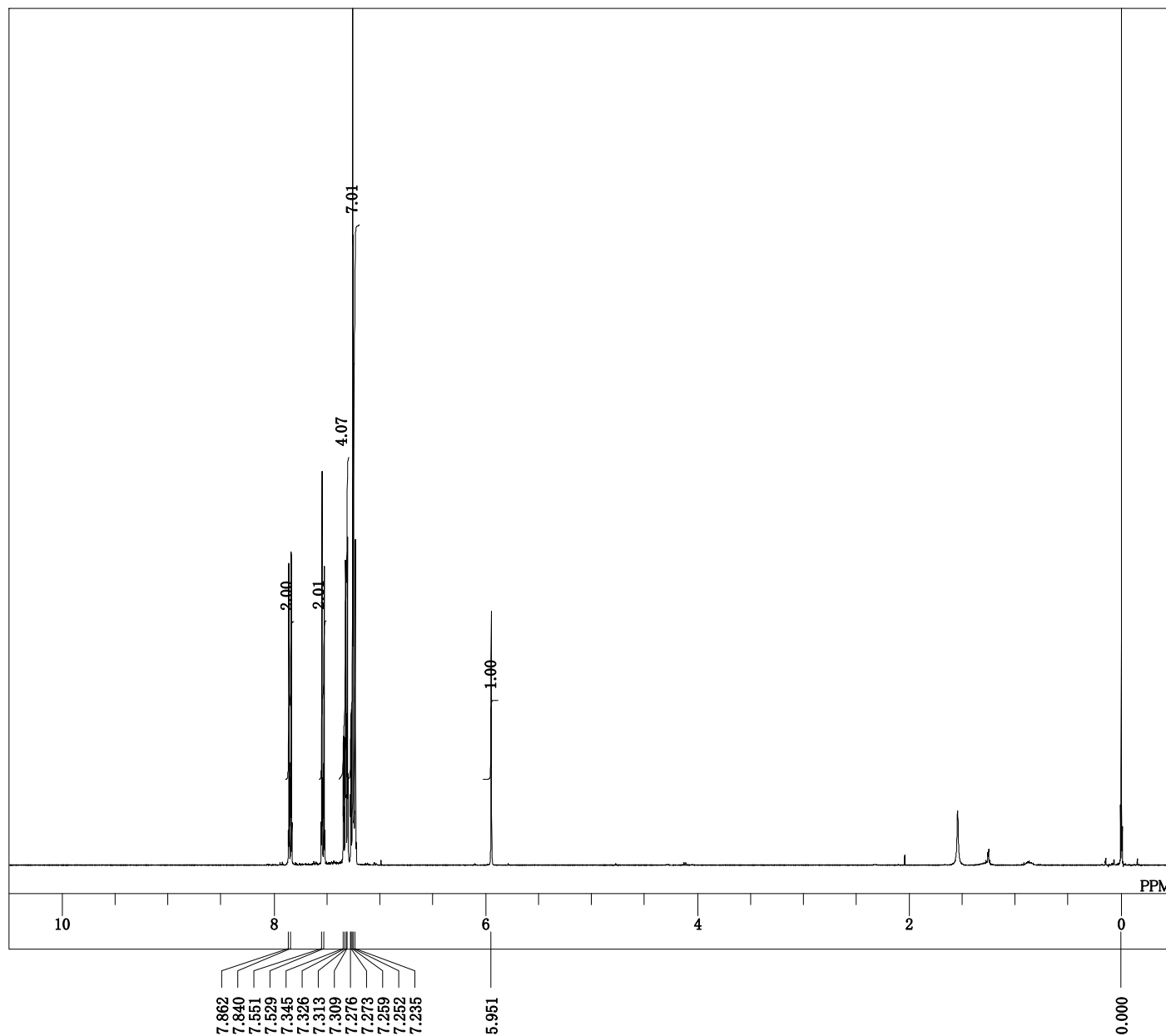
DFILE pro_dioLH_pCL2_Proton.als
 COMNT single_pulse
 DATIM 2020-12-27 15:11:14
 OBNUC 1H
 EXMOD proton_jxp
 OBFREQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 18.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 34



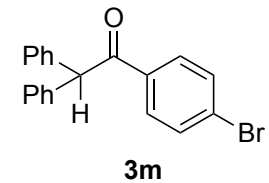


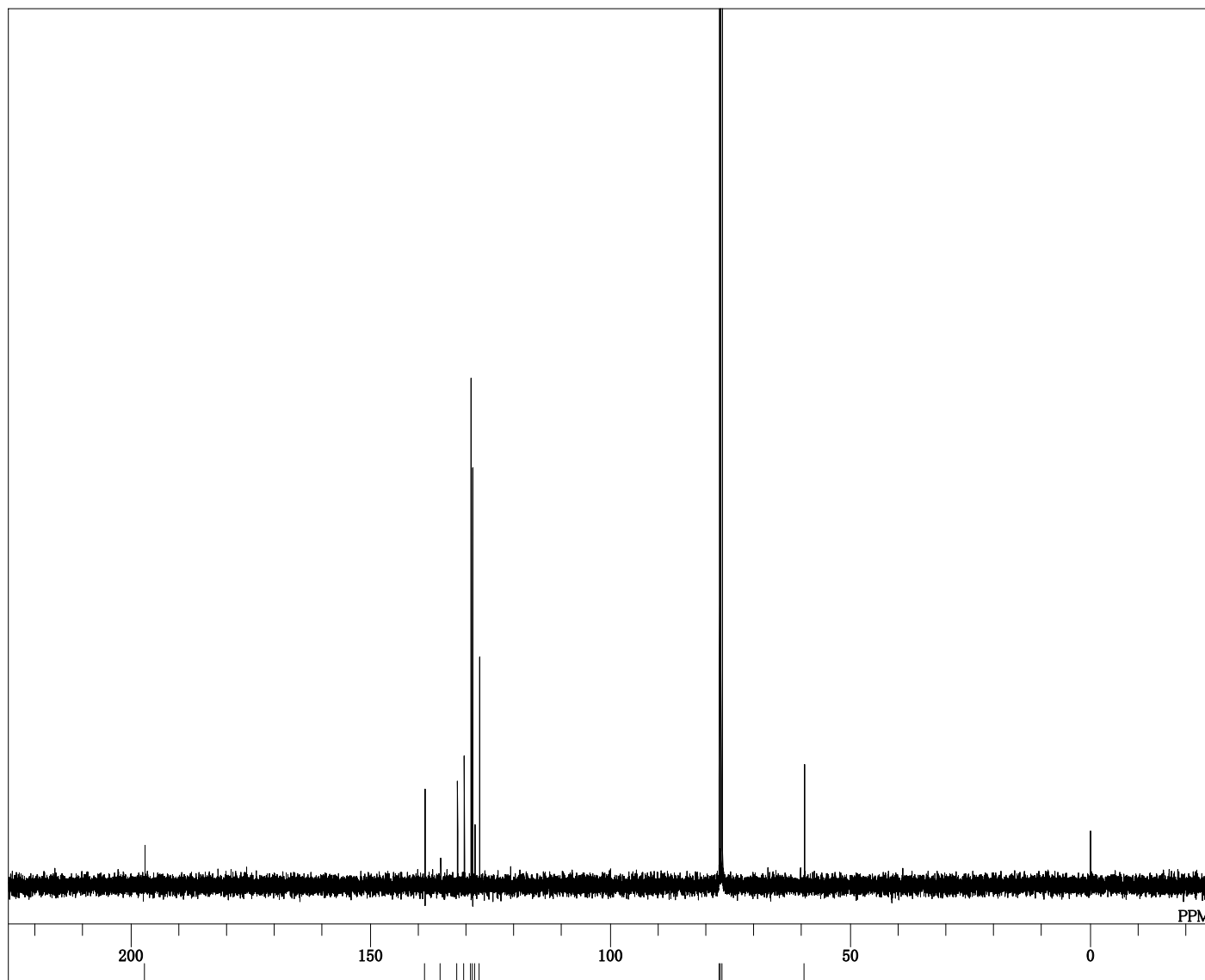
DFILE pro_dioLH_pClEx914_fr1_Carbon.al
 COMNT single pulse decoupled gated NOE
 DATIM 2020-12-27 15:12:27
 OBNUC 13C
 EXMOD carbon.jxp
 OBFREQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 720
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 19.4 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



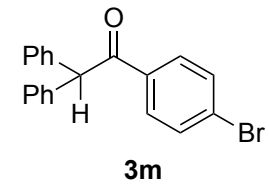


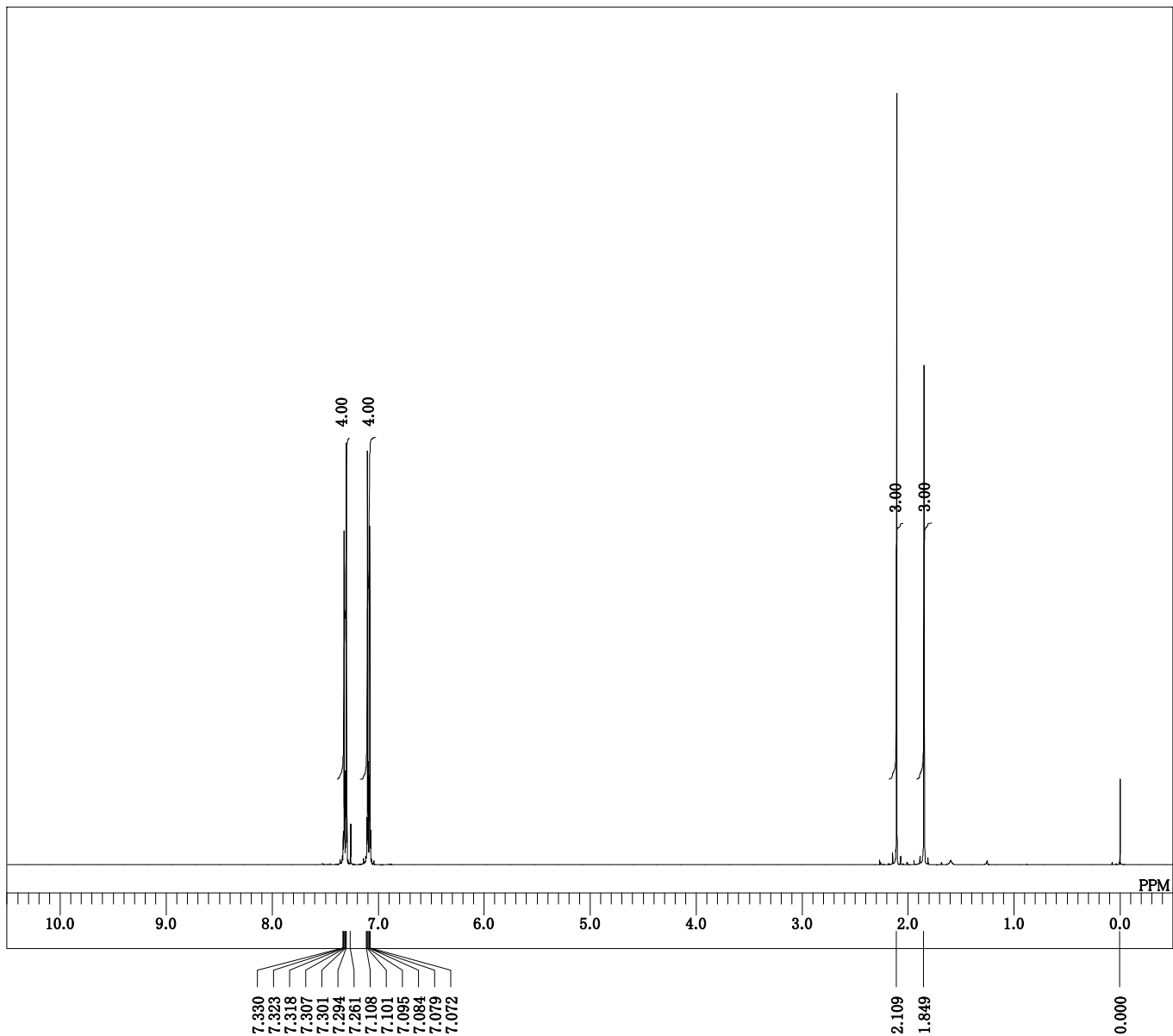
DFILE pro_diolH_pBr_Proton.als
 COMNT single_pulse
 DATIM 2022-04-09 20:16:25
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.25 usec
 IRNUC 1H
 CTEMP 20.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 46



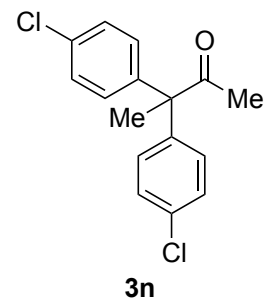


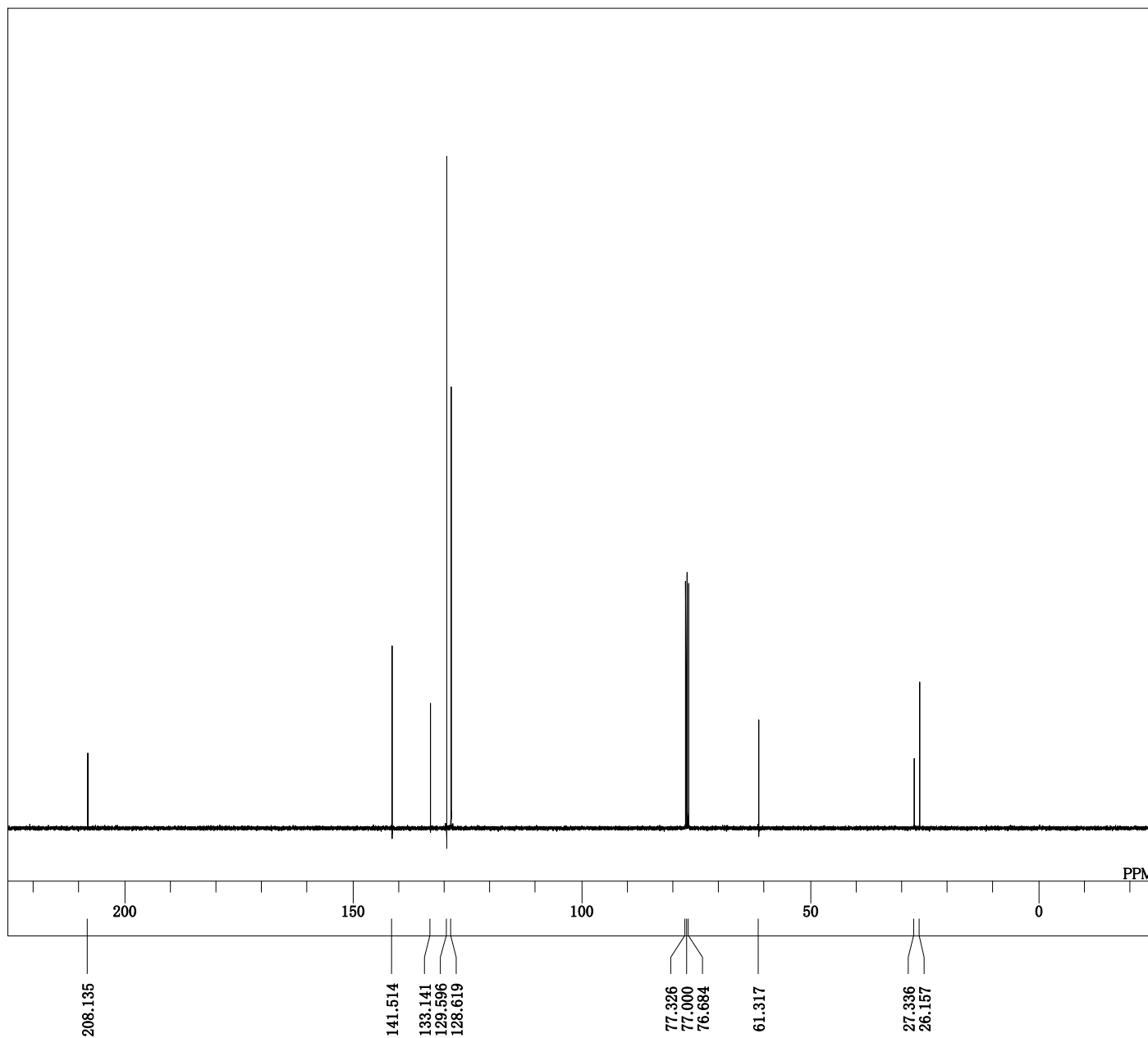
DFILE pro_diol_H_pBr_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2022-04-09 20:19:13
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 1024
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.67 usec
 IRNUC 1H
 CTEMP 20.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



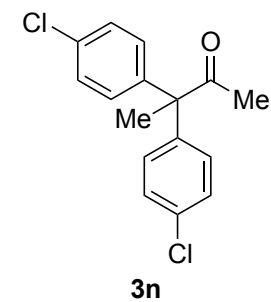


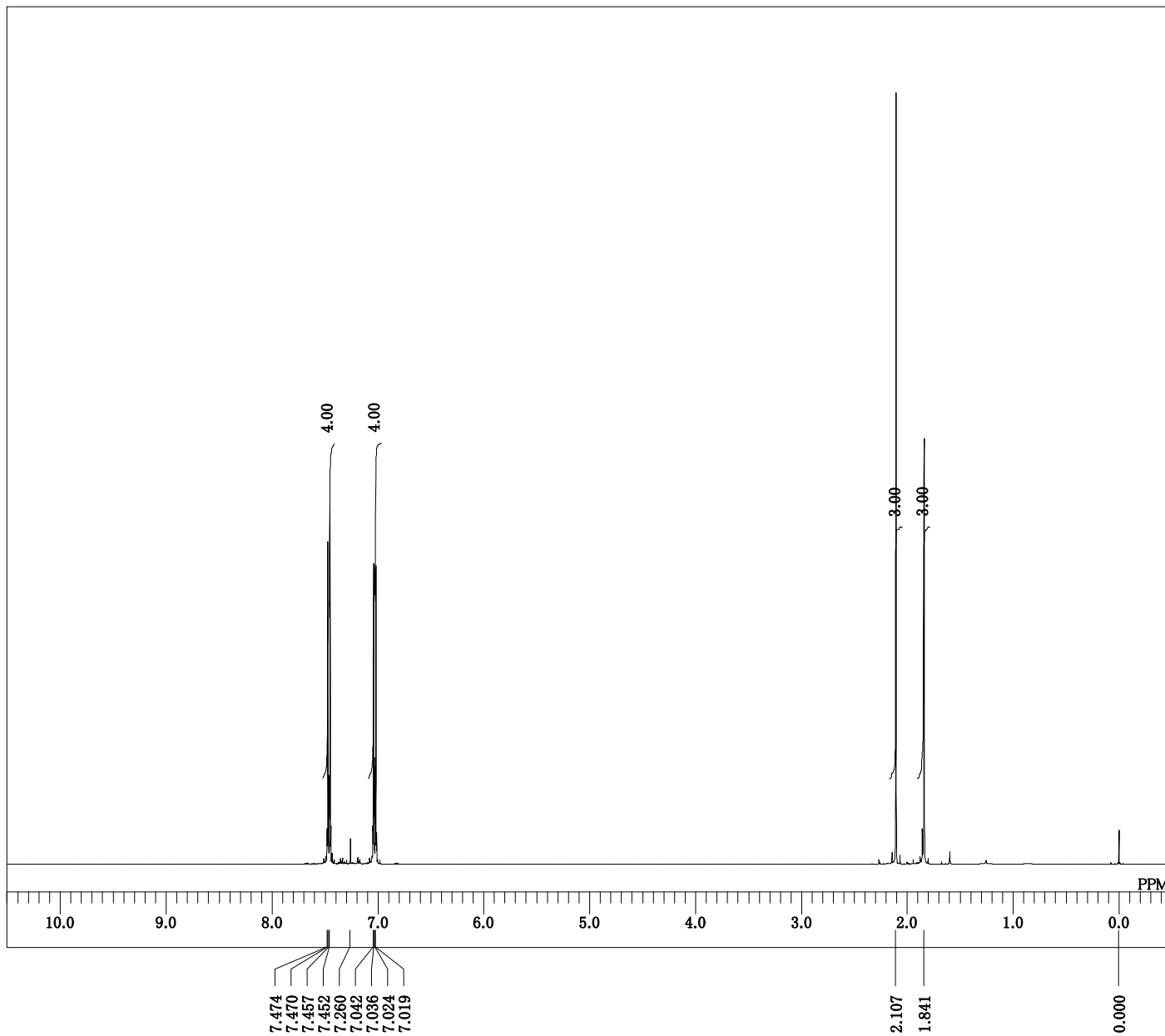
DFILE pro_dioLpClpClMeMe-Proton-1-1.als
 COMNT single_pulse
 DATIM 2020-11-24 23:27:51
 OBNUC 1H
 EXMOD proton.jpg
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 19.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 30



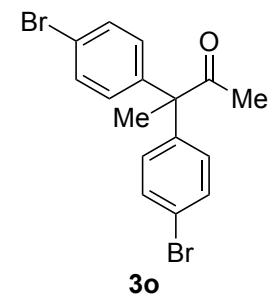


DFILE pro_diol_pClpClMeMe_Carbon-1-1
 COMNT single pulse decoupled gated NOE
 DATIM 2020-11-24 23:29:04
 OBNUC ¹³C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 1144
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC ¹H
 CTEMP 18.8 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50

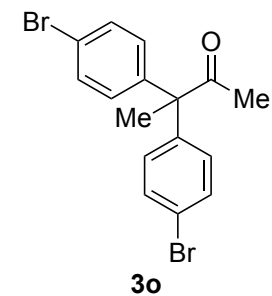
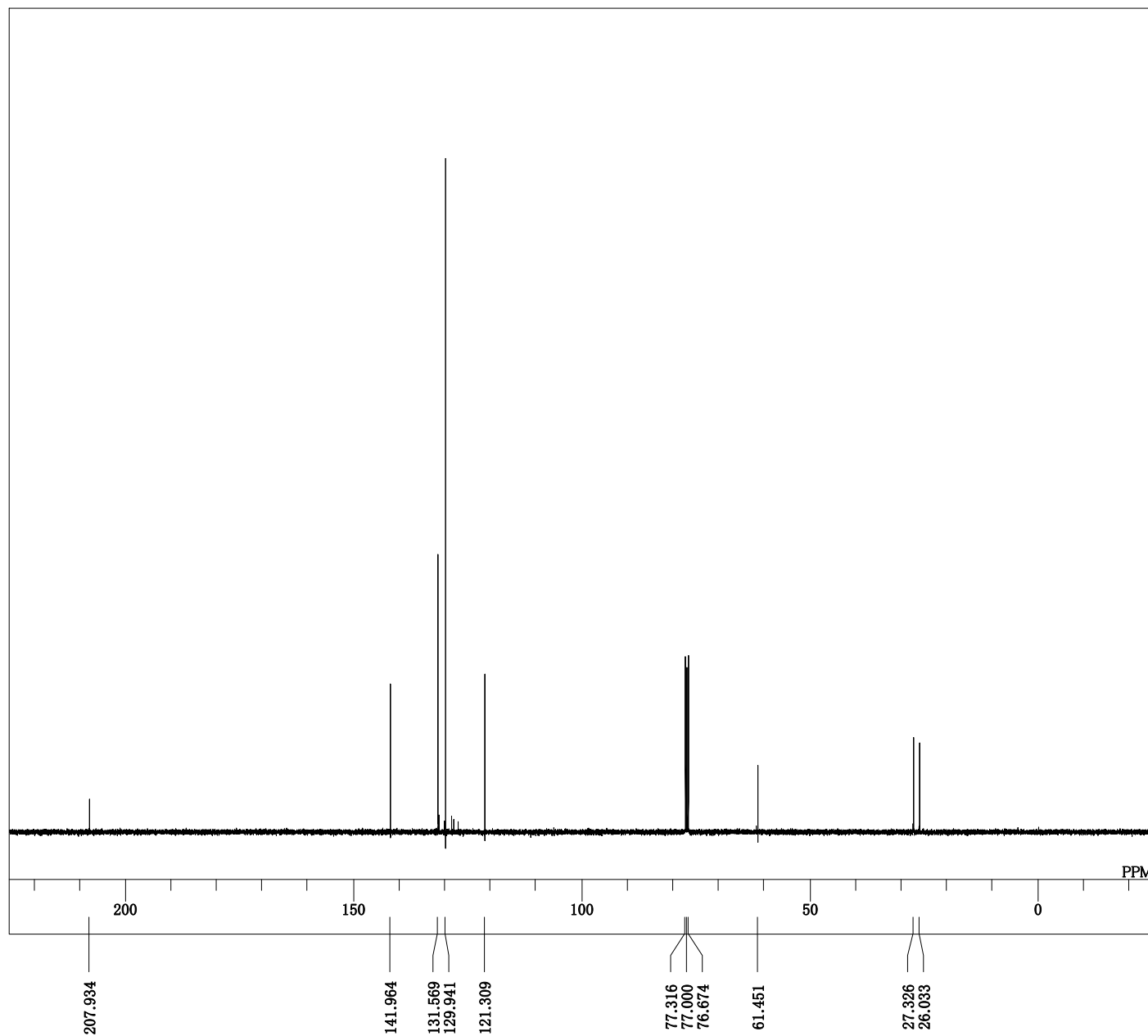


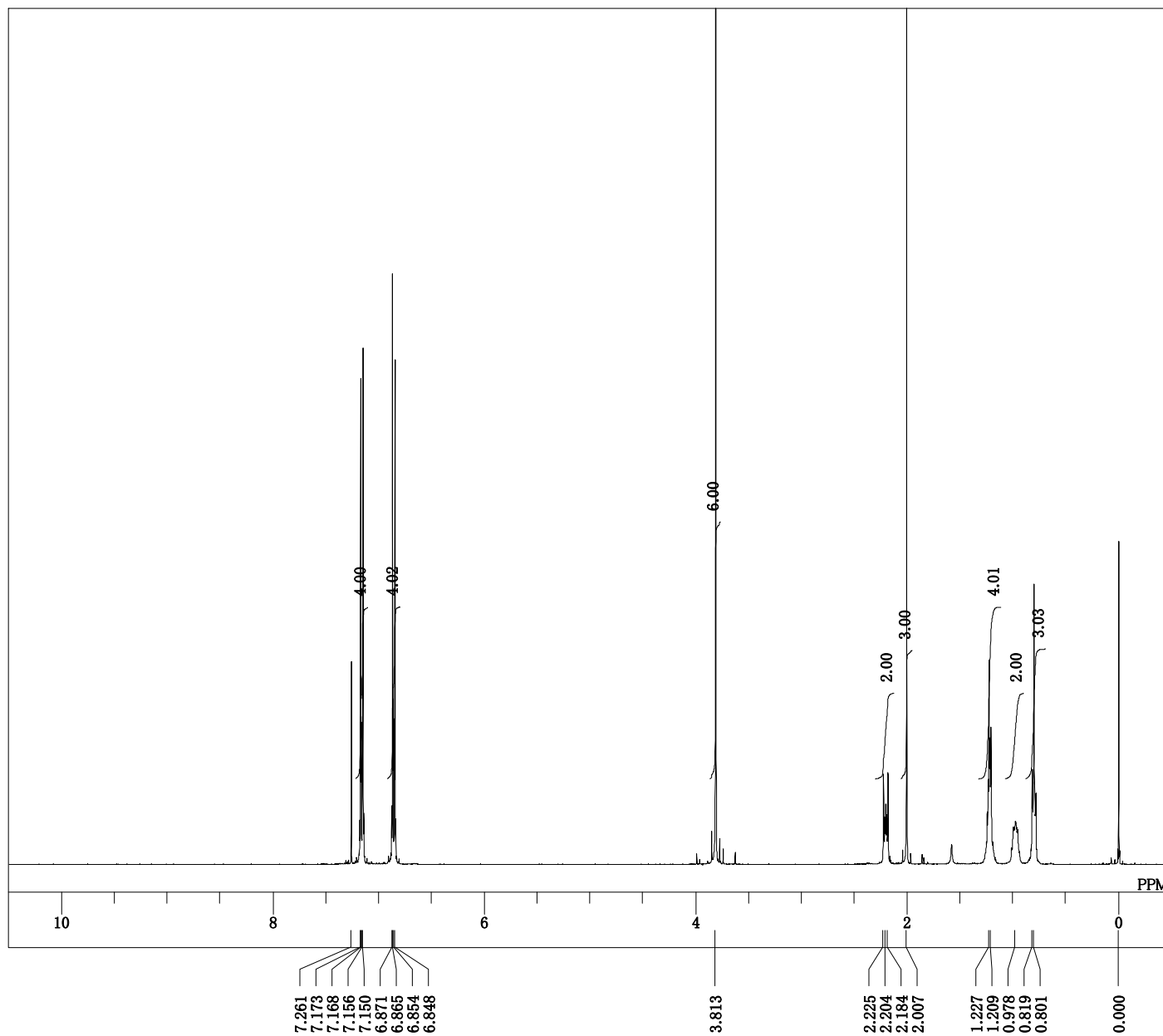


DFILE pro_diol_pBrpBr_MeMe_Proton.als
 COMNT single_pulse
 DATIM 2020-12-04 17:43:44
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 18.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 28

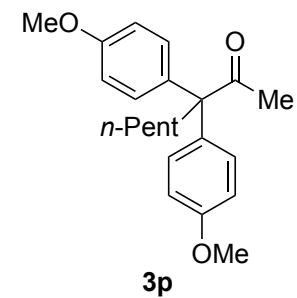


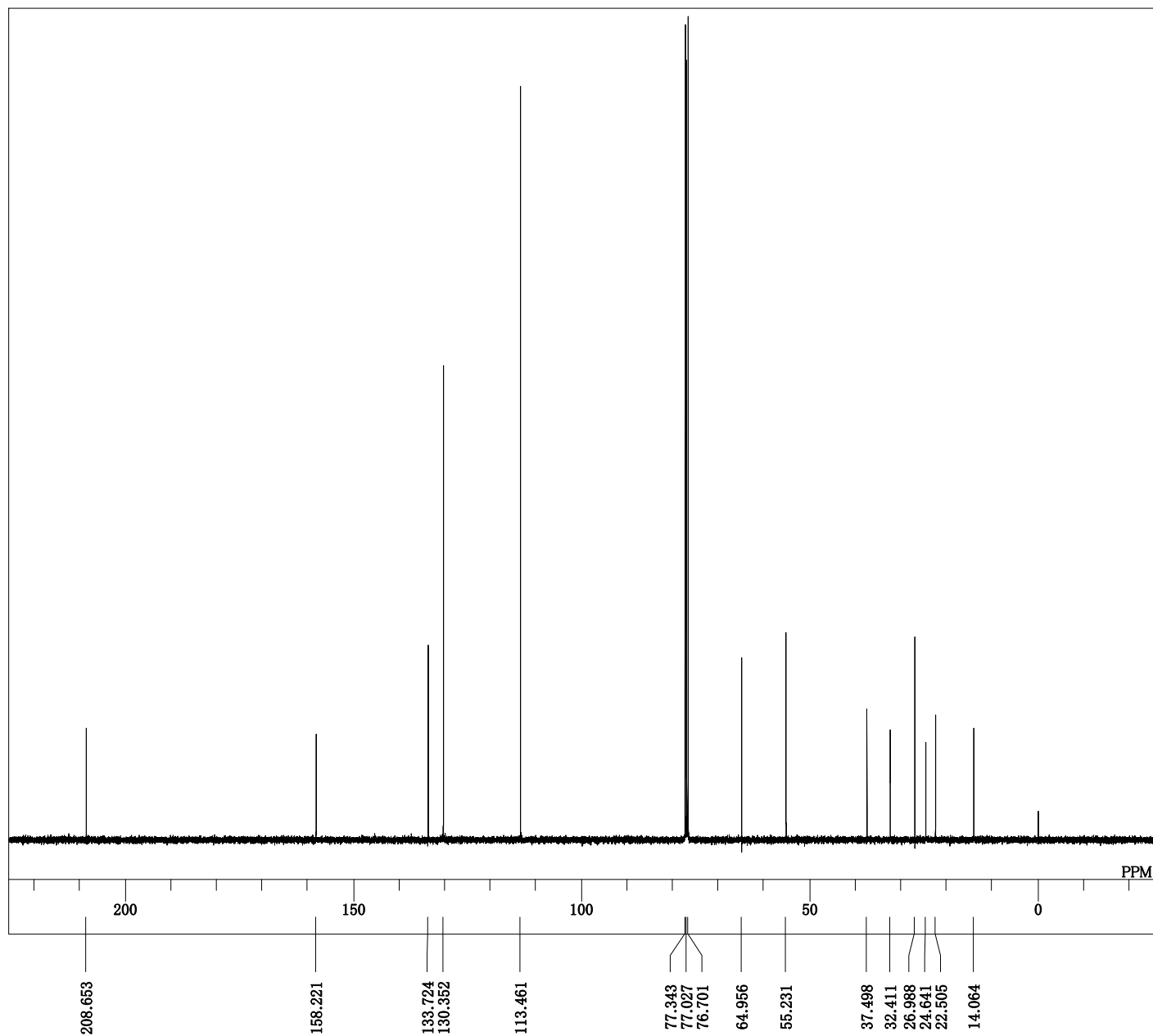
DFILE pro_diol_pBrpBr_MeMe_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-12-04 17:45:59
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 299
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 18.9 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



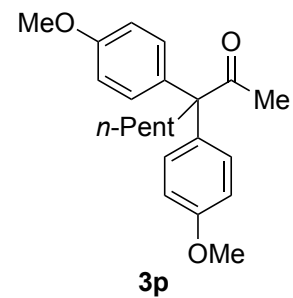


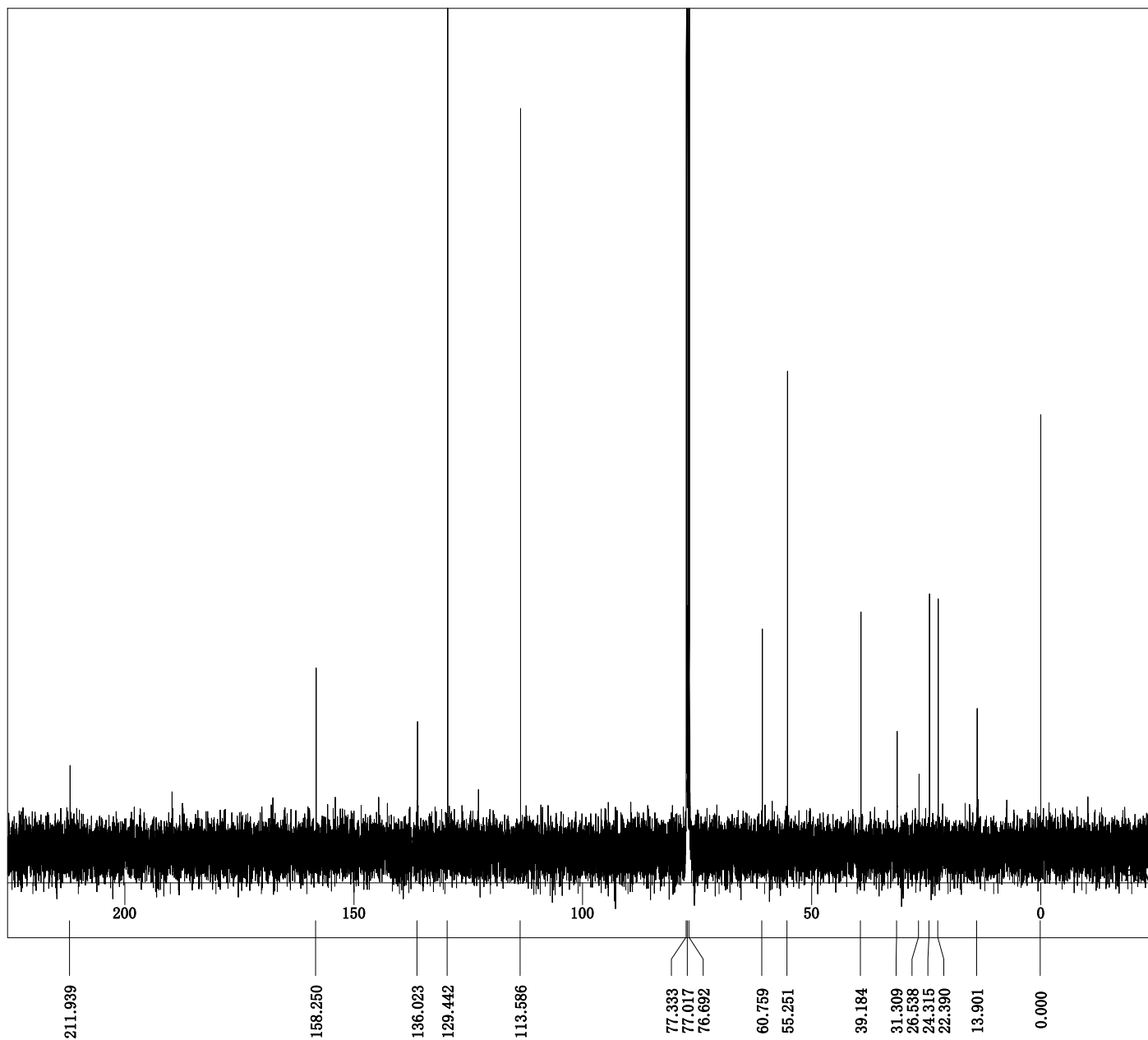
DFILE pro_diol_PMPPMP_MenPenty_lfr4_Pt
 COMNT single_pulse
 DATIM 2020-10-18 04:52:22
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 19.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 34



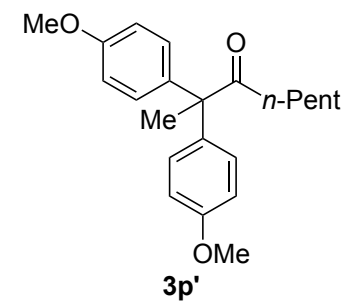


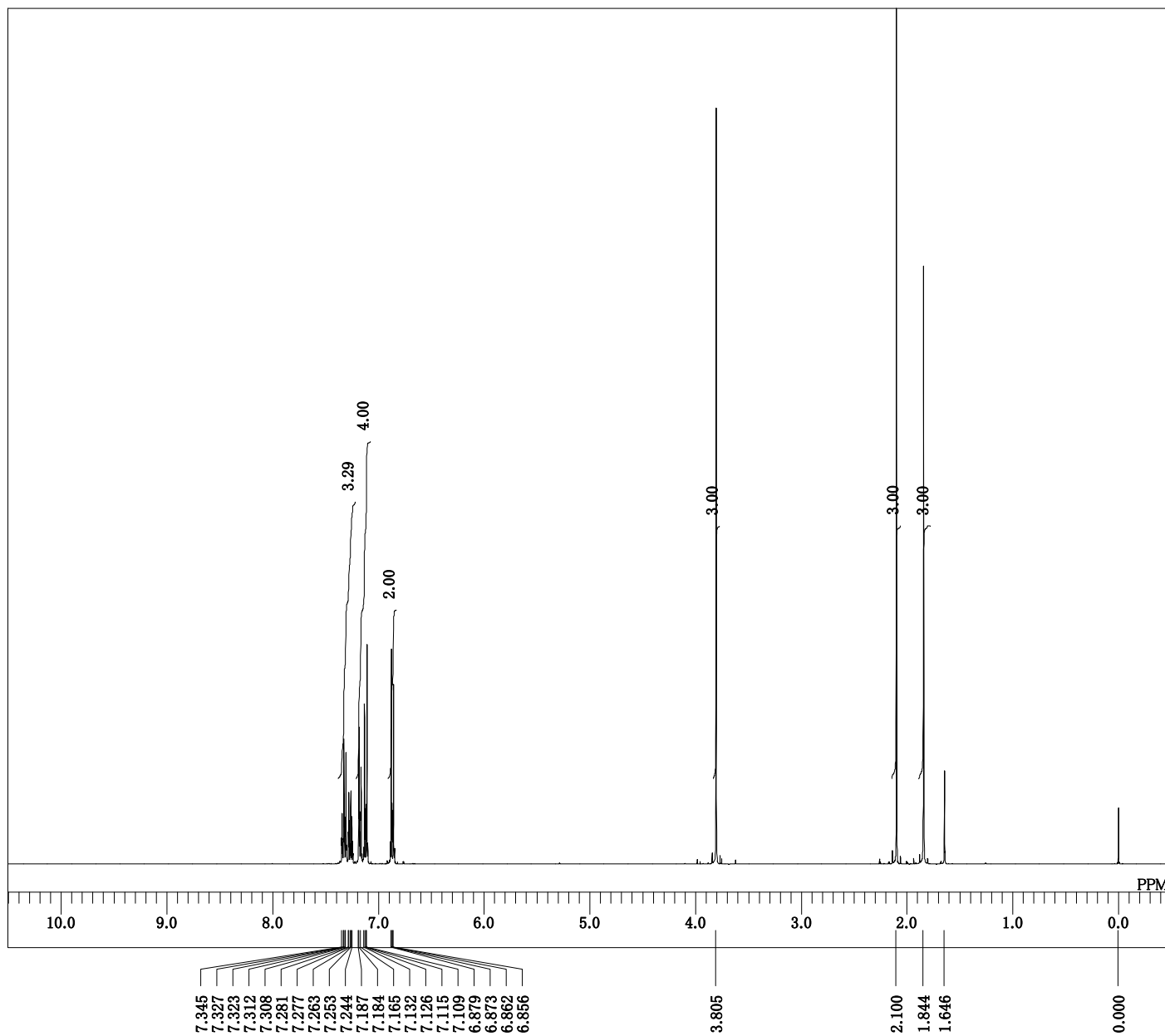
DFILE pro_diol_PMPPMP_MenPentyLfr4_C
 COMNT single pulse decoupled gated NOE
 DATIM 2020-10-18 04:53:35
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 3409
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 19.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50



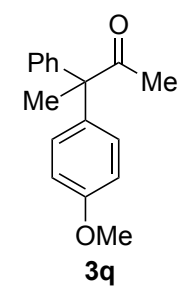


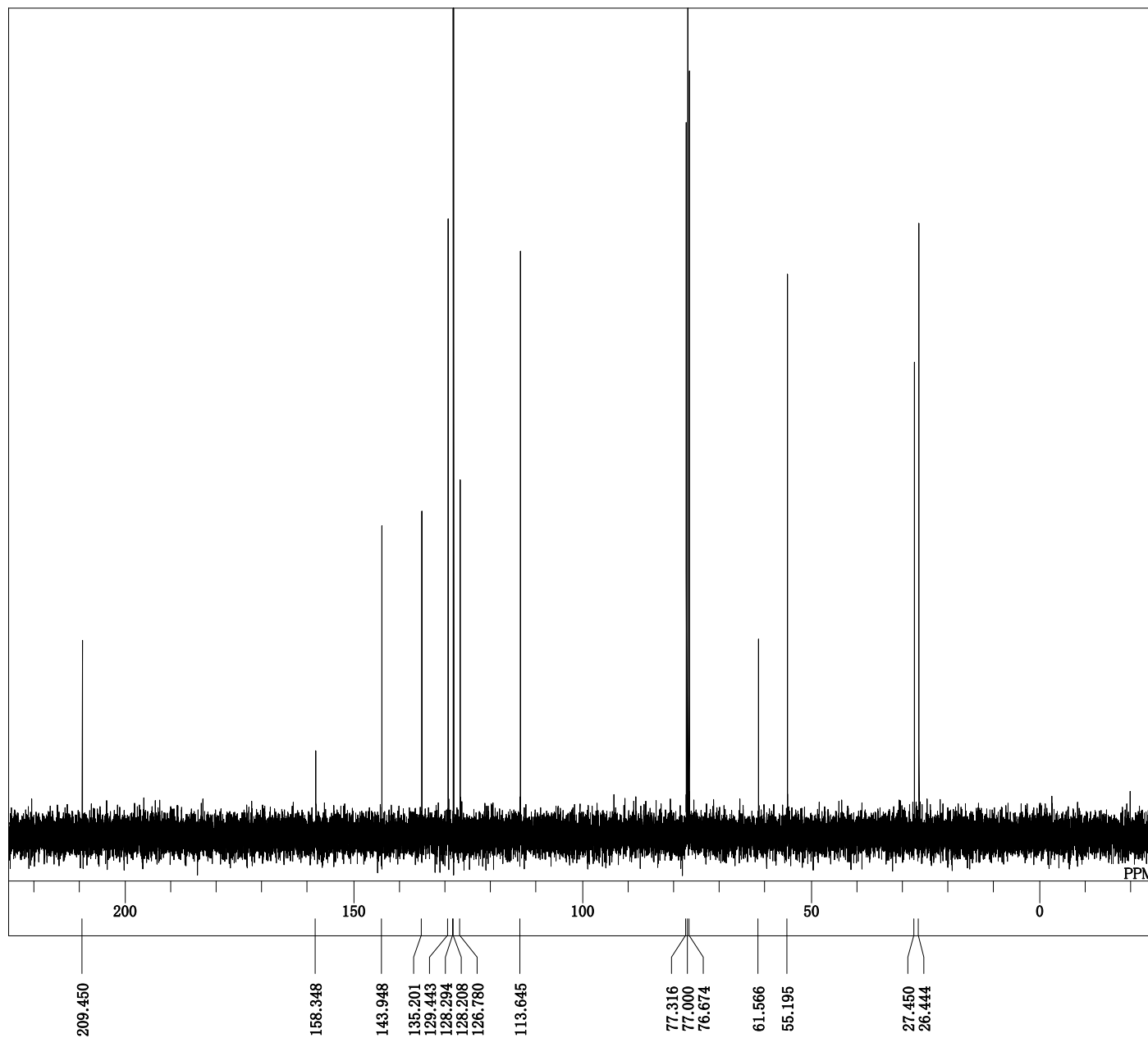
DFILE pro_diol_PMPPMP_MenPentyl_fr3_C
 COMNT single pulse decoupled gated NOE
 DATIM 2020-10-18 01:17:35
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 4096
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 20.1 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50



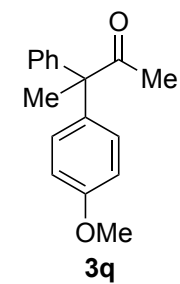


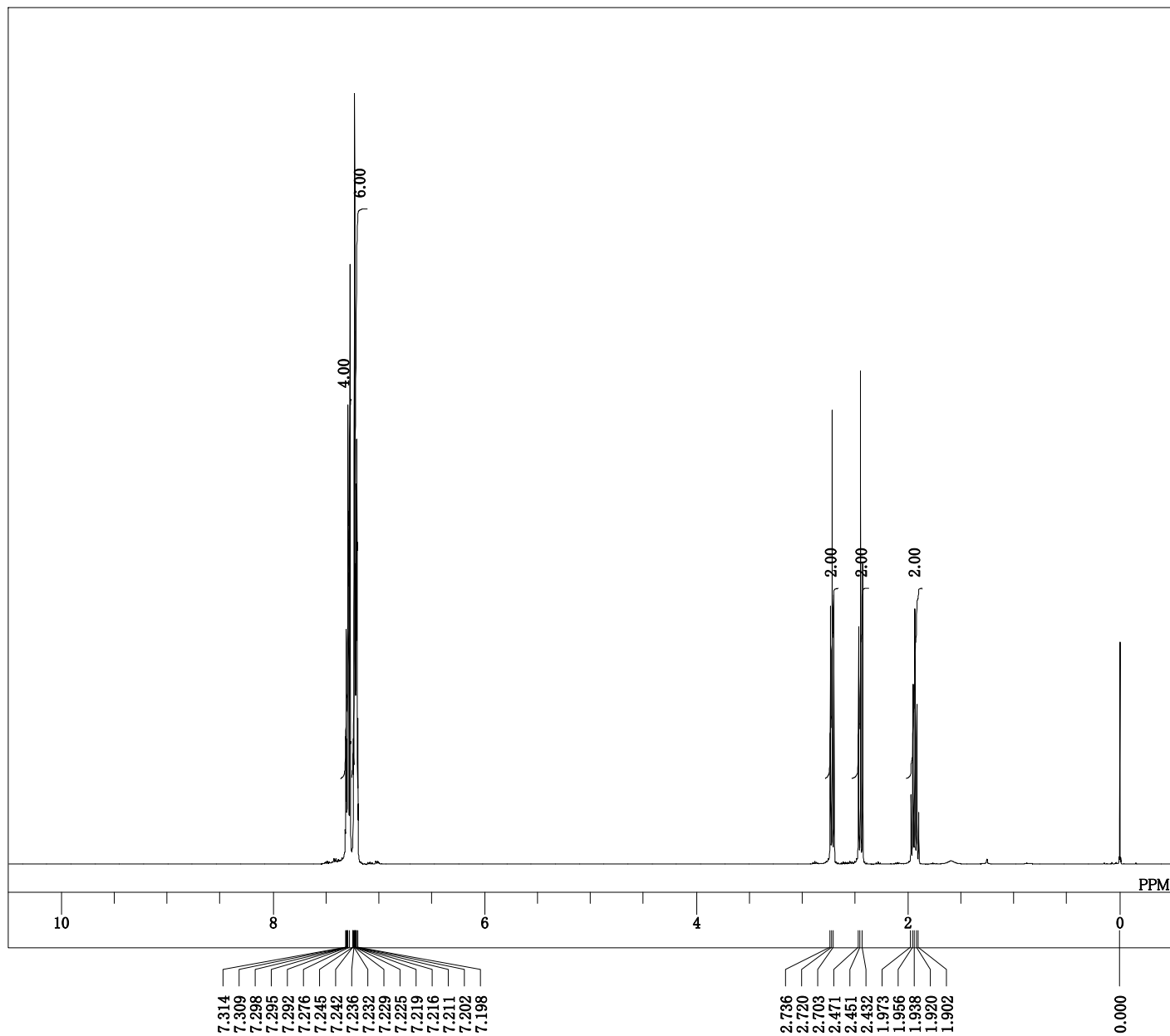
DFILE pro_diol_PhPMP.MeMe_Proton.als
 COMNT single_pulse
 DATIM 2020-09-12 09:54:12
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 26



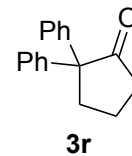


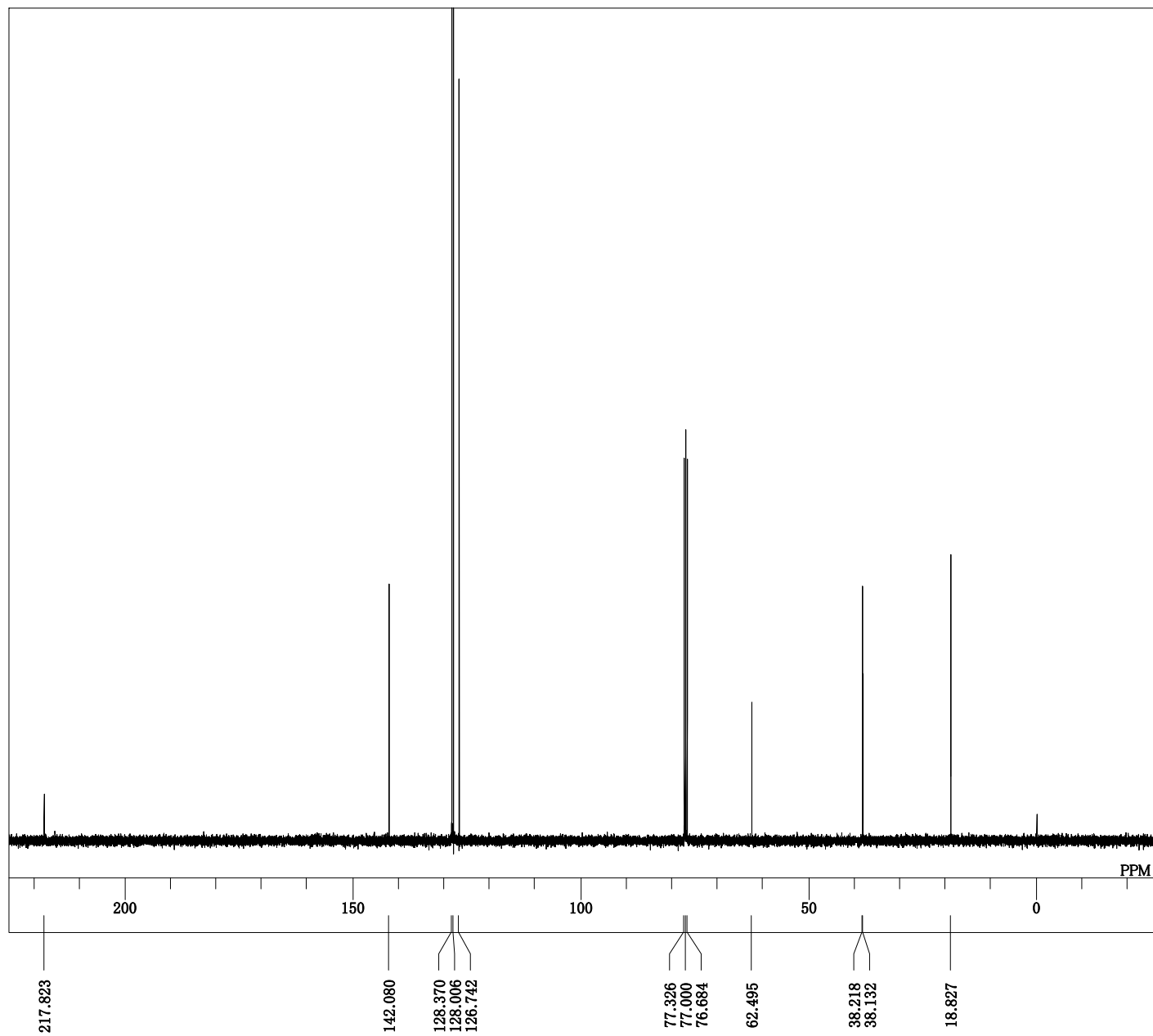
DFILE pro_diol_PhPMP_MeMe_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-09-12 10:05:40
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 64
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 21.0 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



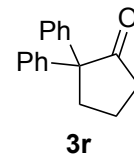


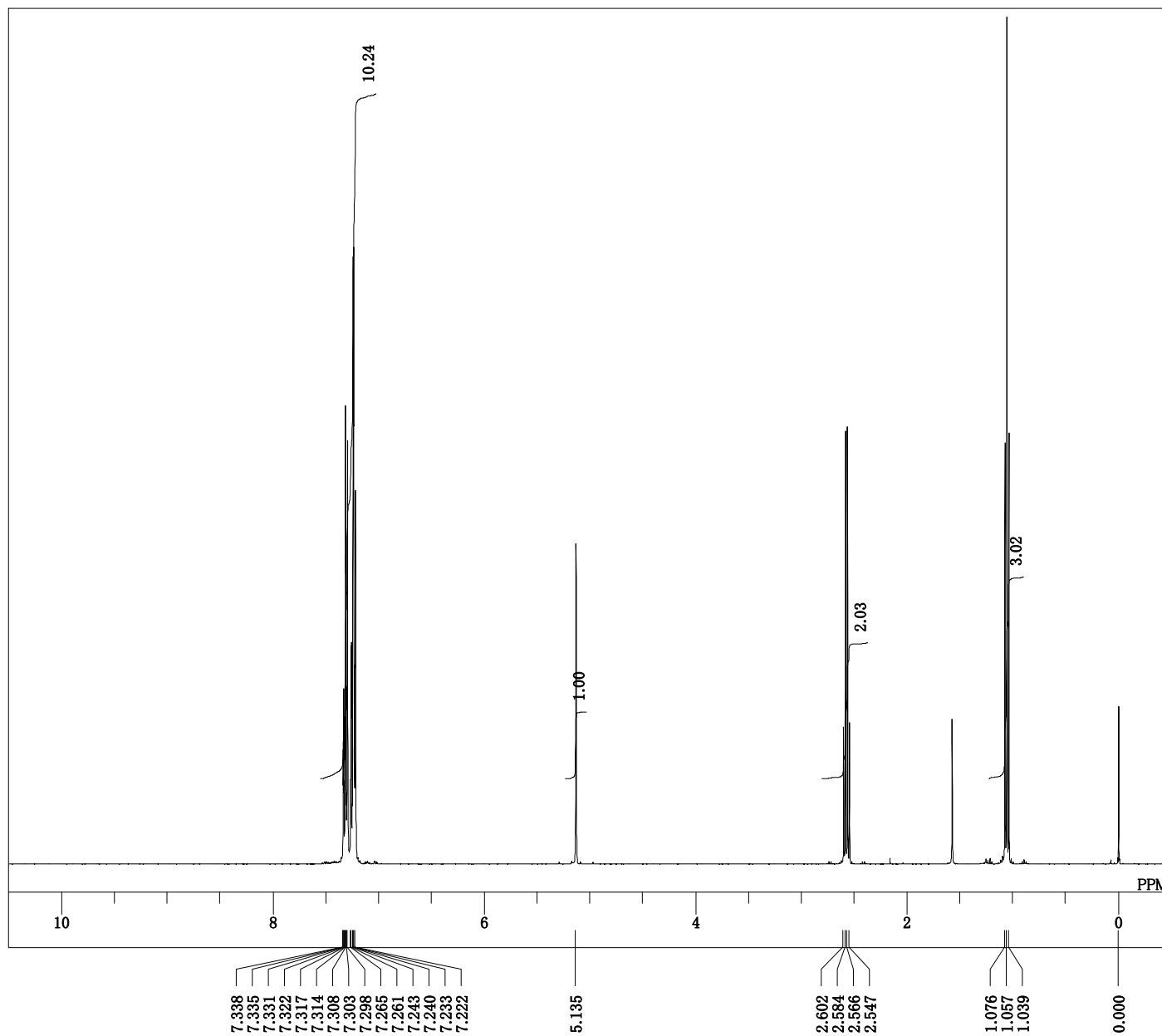
DFILE pro_cyc_cyc_butyl_Proton.als
 COMNT single_pulse
 DATIM 2020-04-01 15:47:11
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 19.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 28



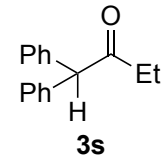


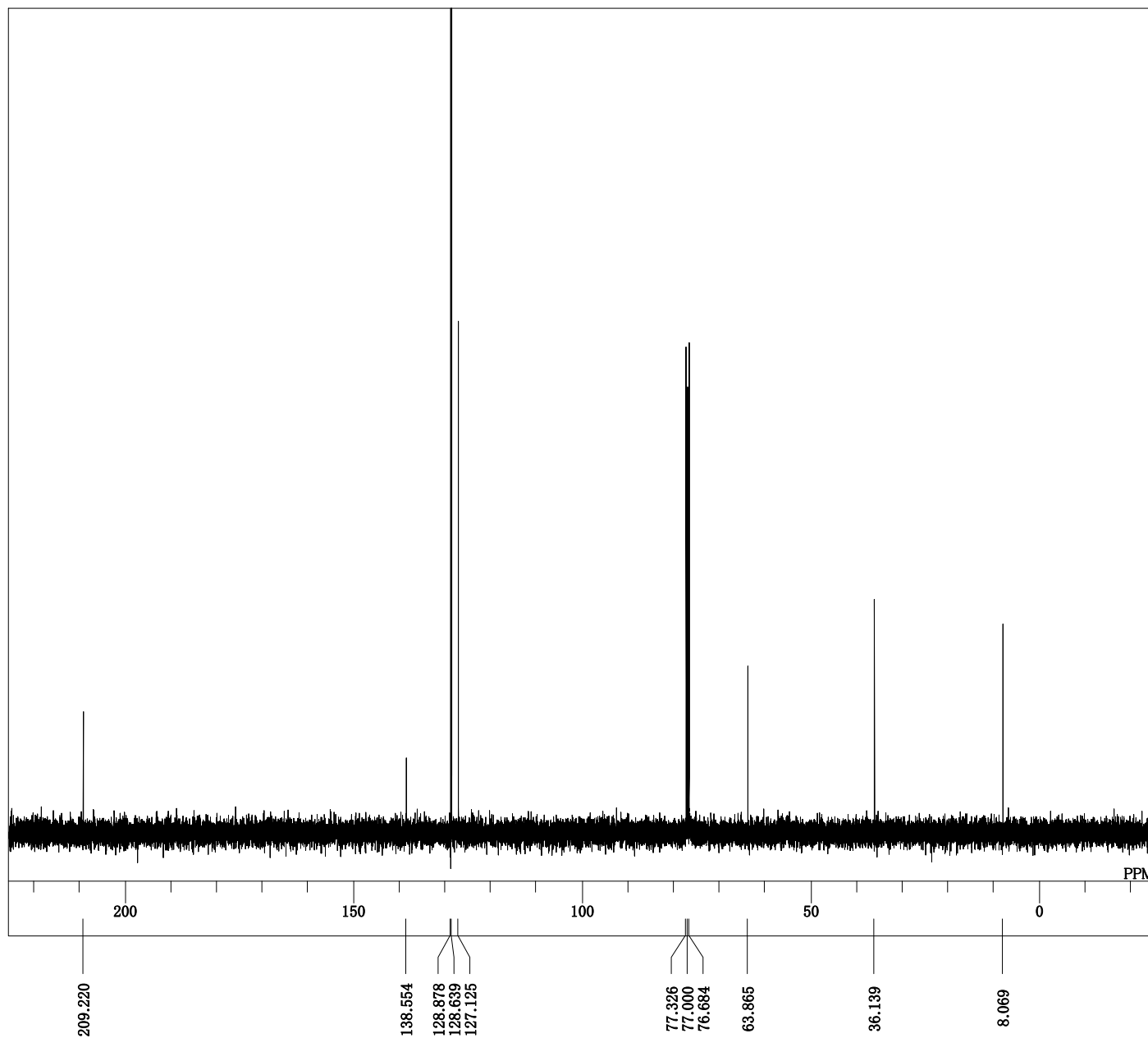
DFILE pro_cyc_cyc_butyl_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-04-01 15:48:24
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 372
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 19.8 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



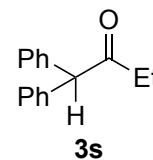


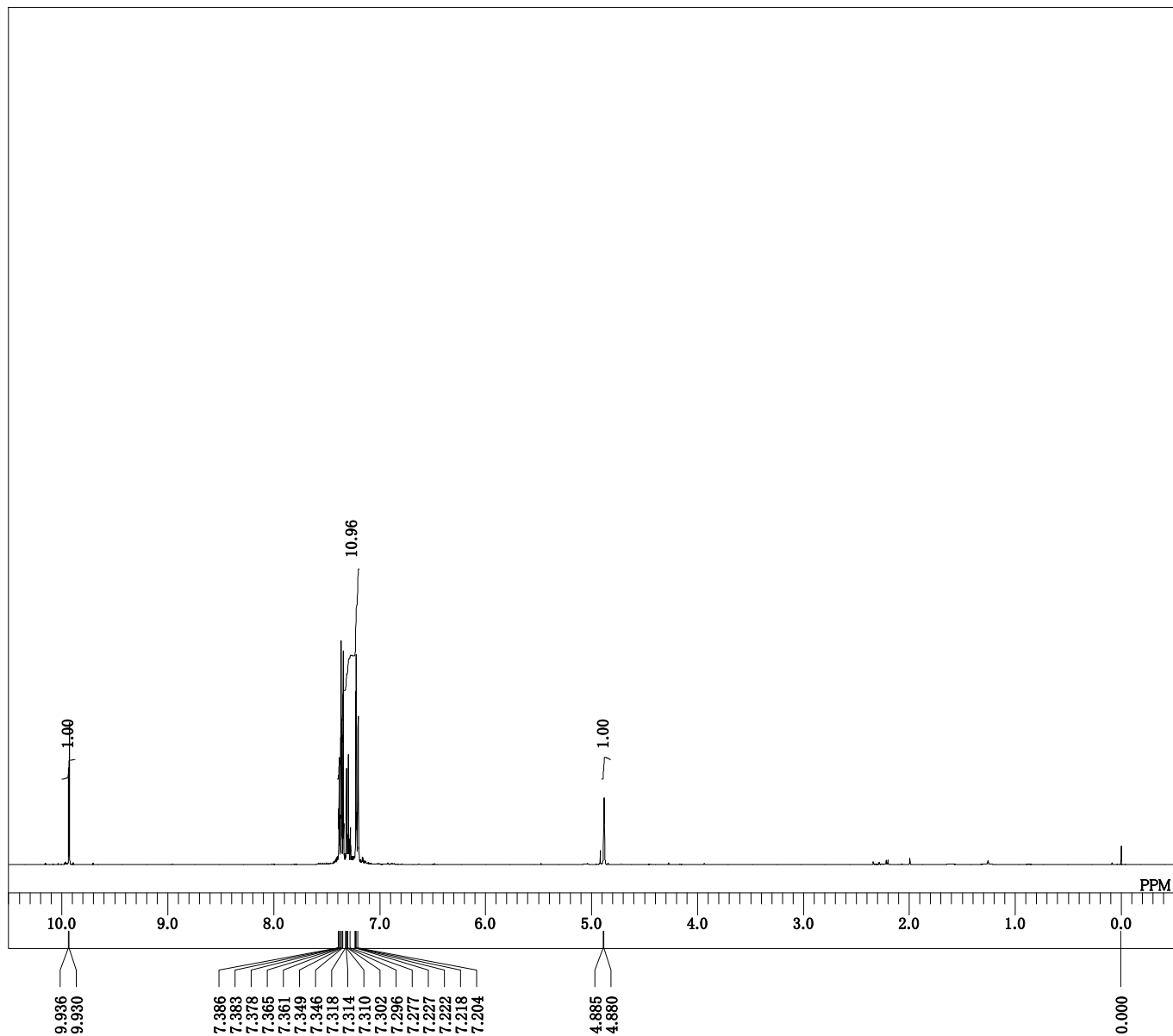
DFILE proton.jxp
 COMNT single_pulse
 DATIM 2020-07-13 13:54:60
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 30



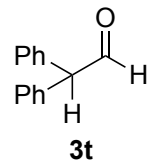


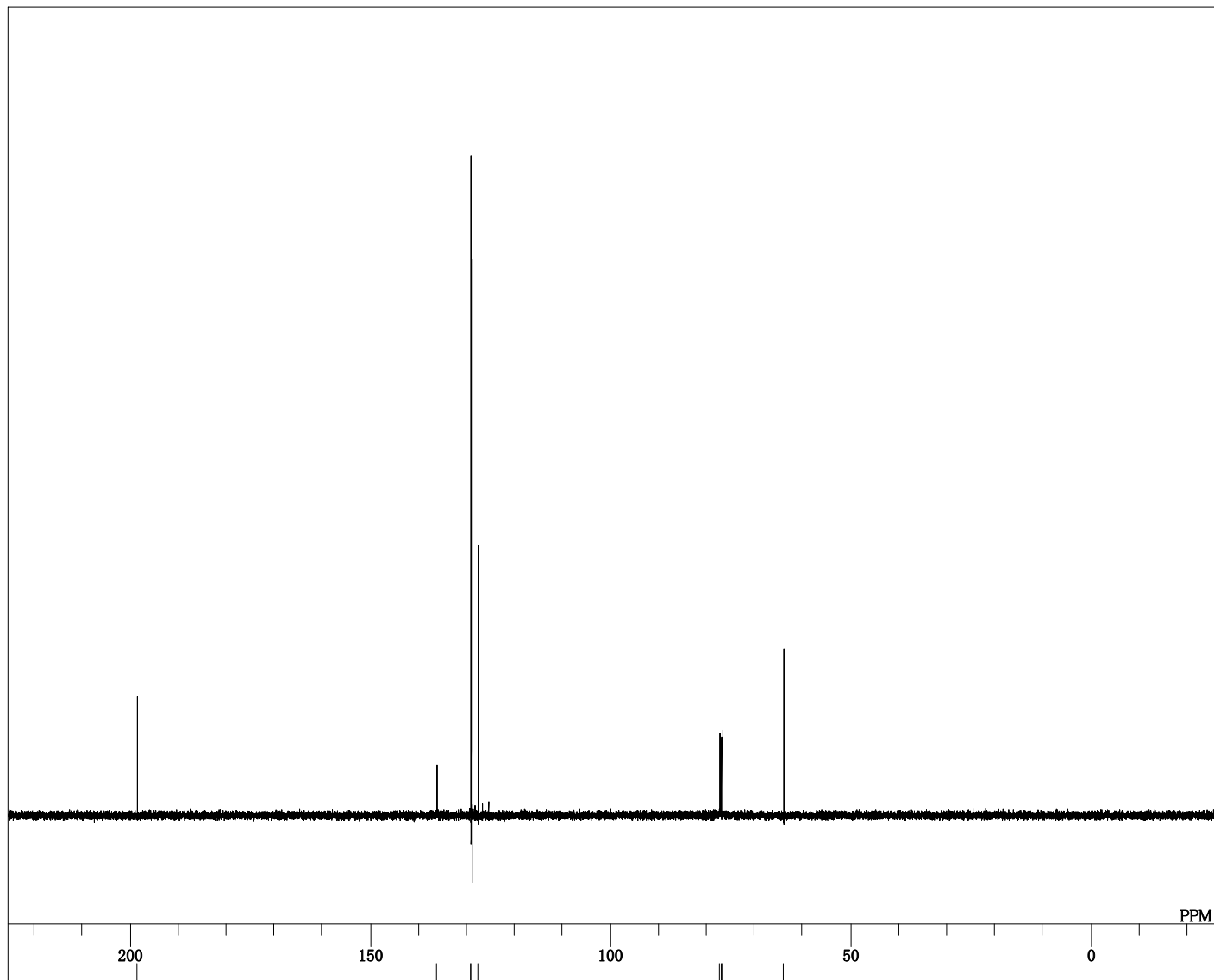
DFILE pro_diolH_Et_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-07-13 13:56:12
 OBNUC ¹³C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 40
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC ¹H
 CTEMP 20.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



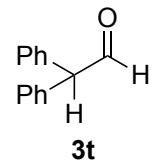


DFILE pro_diol_H_HProton.als
 COMNT single_pulse
 DATIM 2020-12-29 06:43:36
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 18.3 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 24





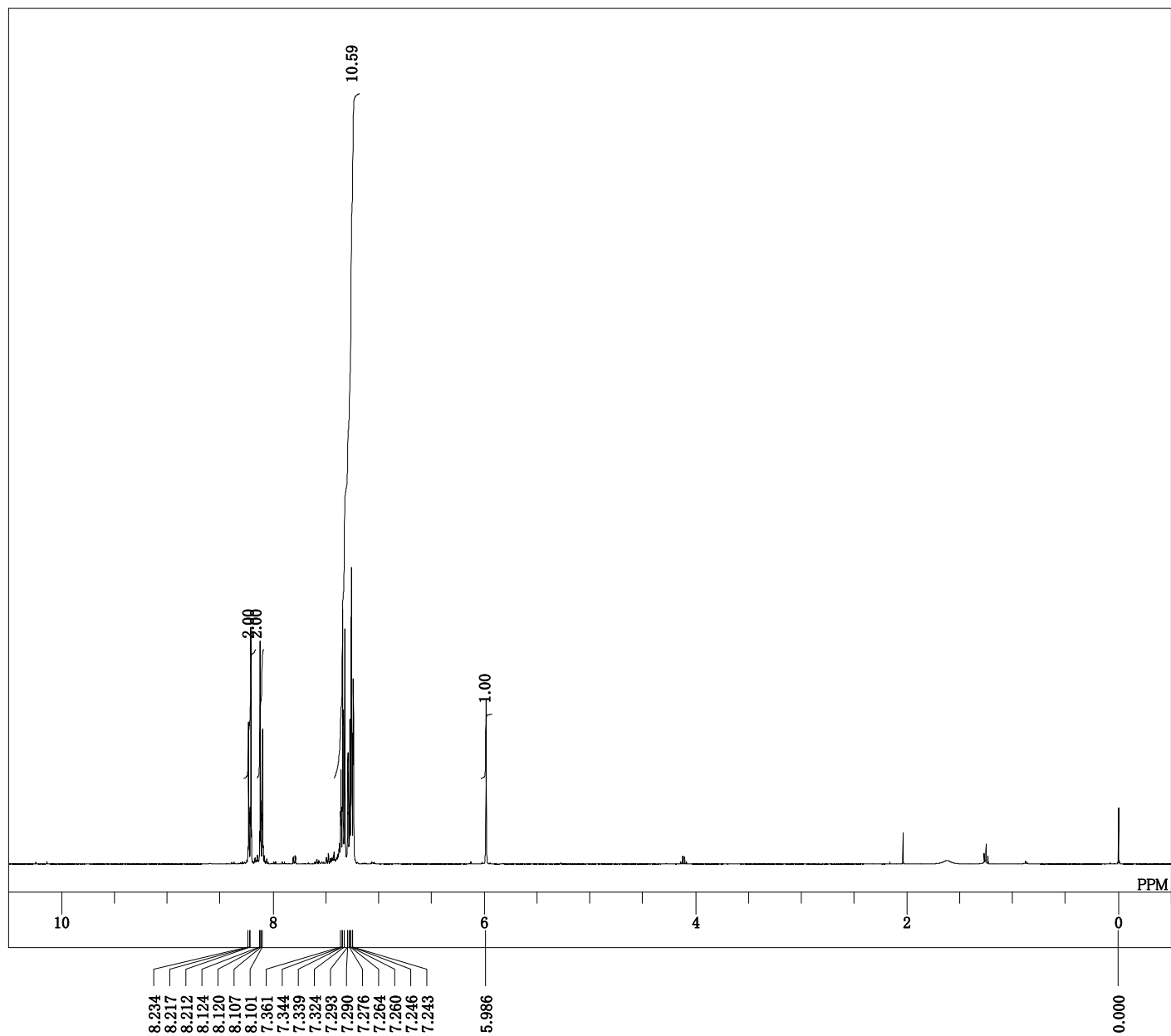
DFILE pro_diolH_H_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-12-29 06:44:48
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 MHz
 SCANS 16
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 18.4 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



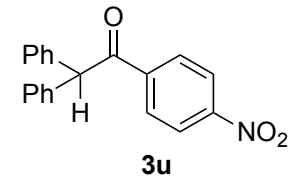
198.536

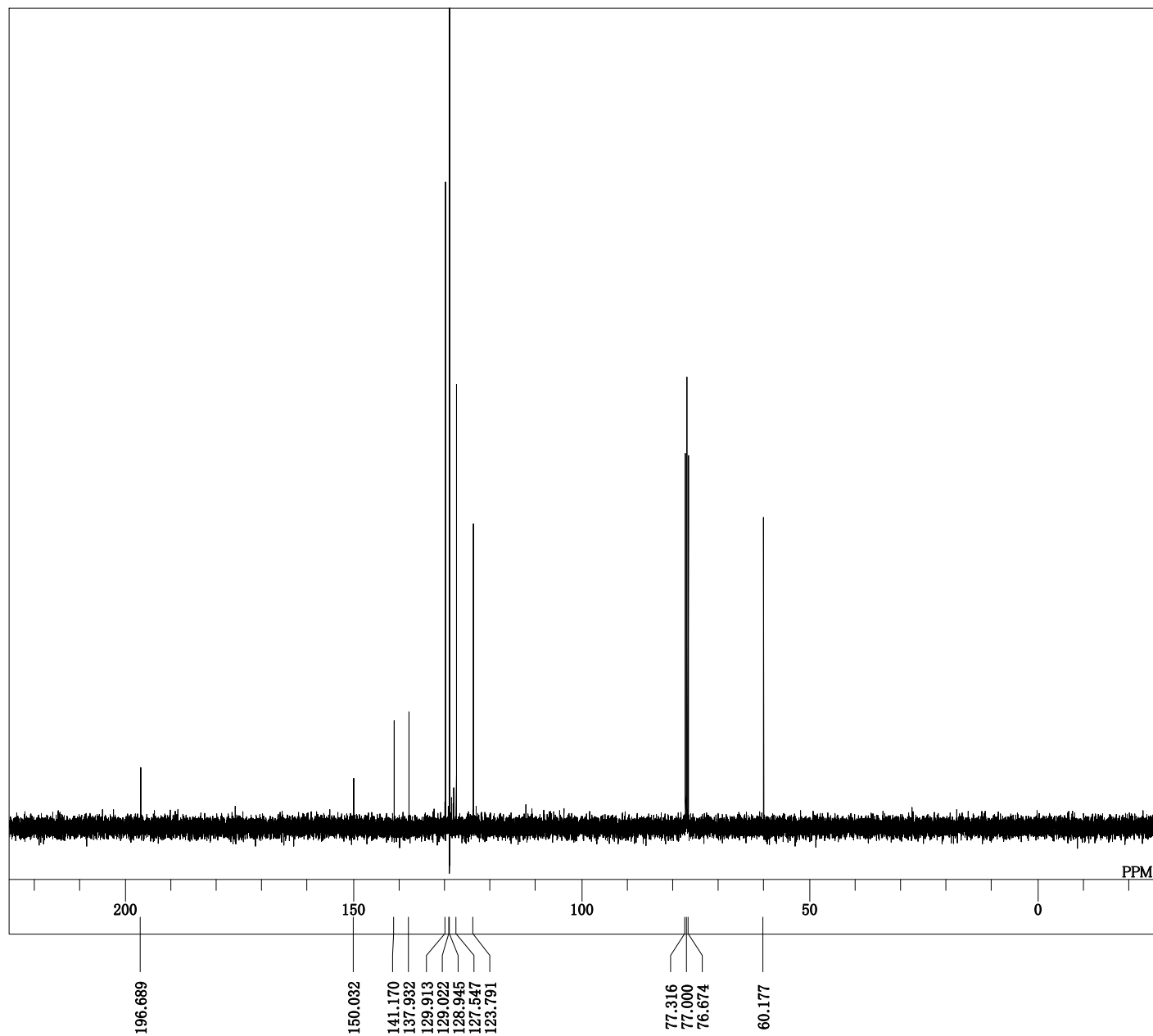
136.216
129.098
128.935
127.555

77.316
77.000
76.674
64.019

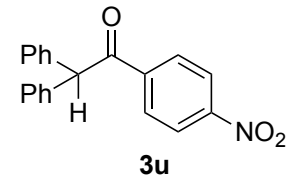


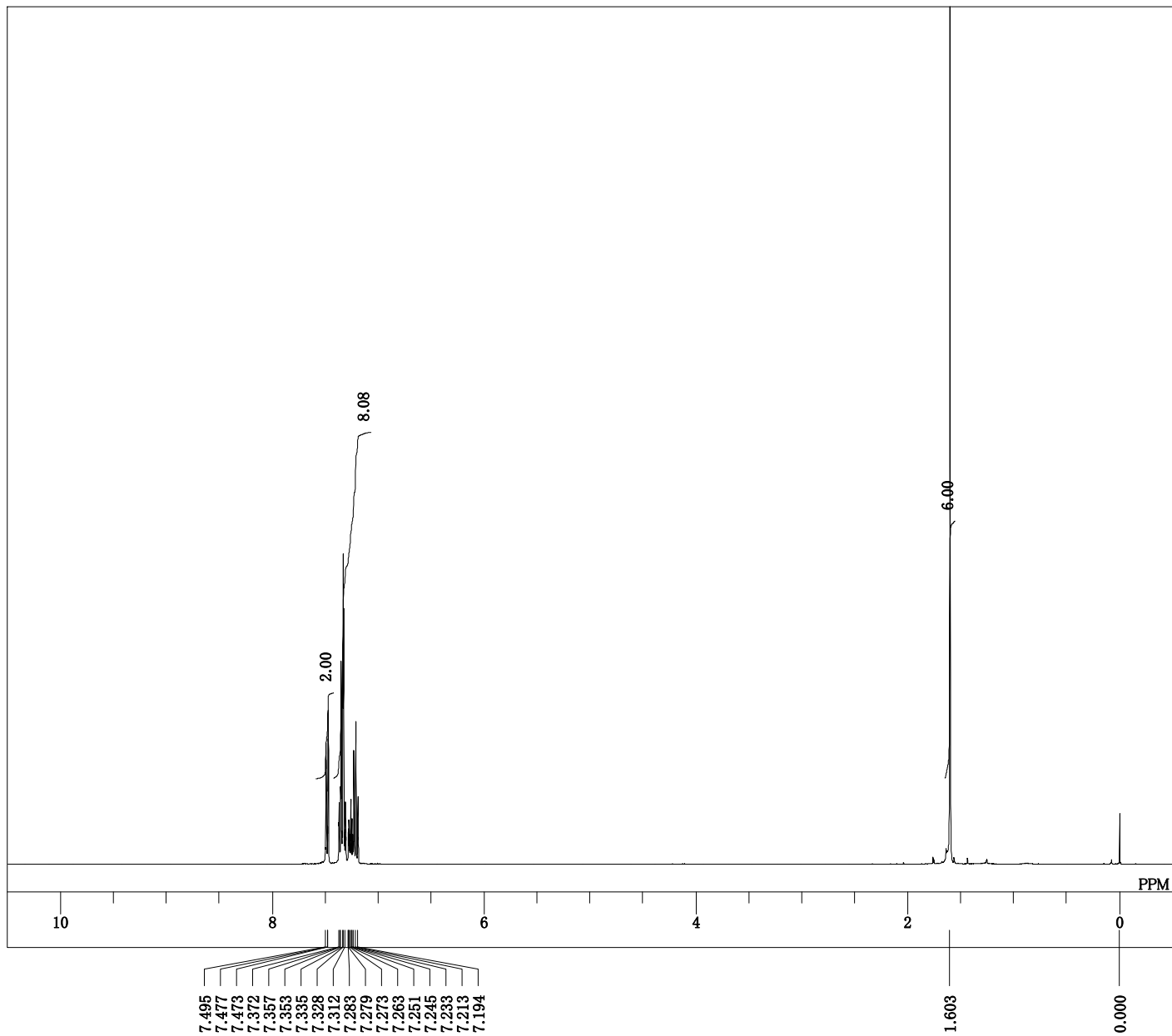
DFILE pro_diolH_pNO2_fr1_Proton.als
 COMNT single_pulse
 DATIM 2020-07-14 14:29:35
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 28



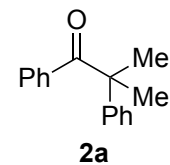


DFILE pro_diol_H_pNO2_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-07-14 14:30:47
 OBNUC ¹³C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 64
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC ¹H
 CTEMP 20.7 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50

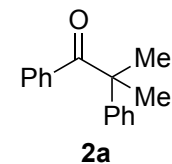
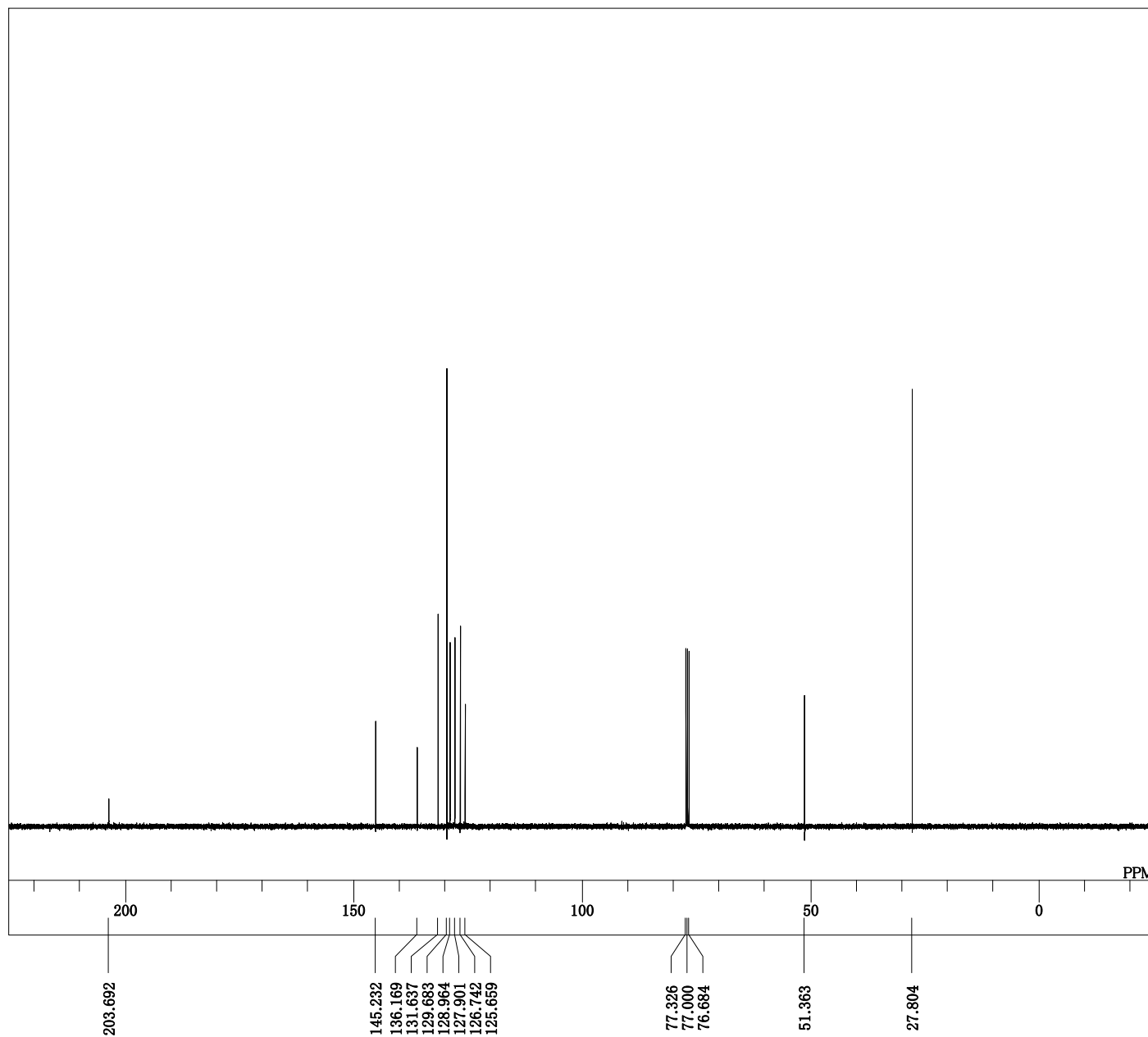


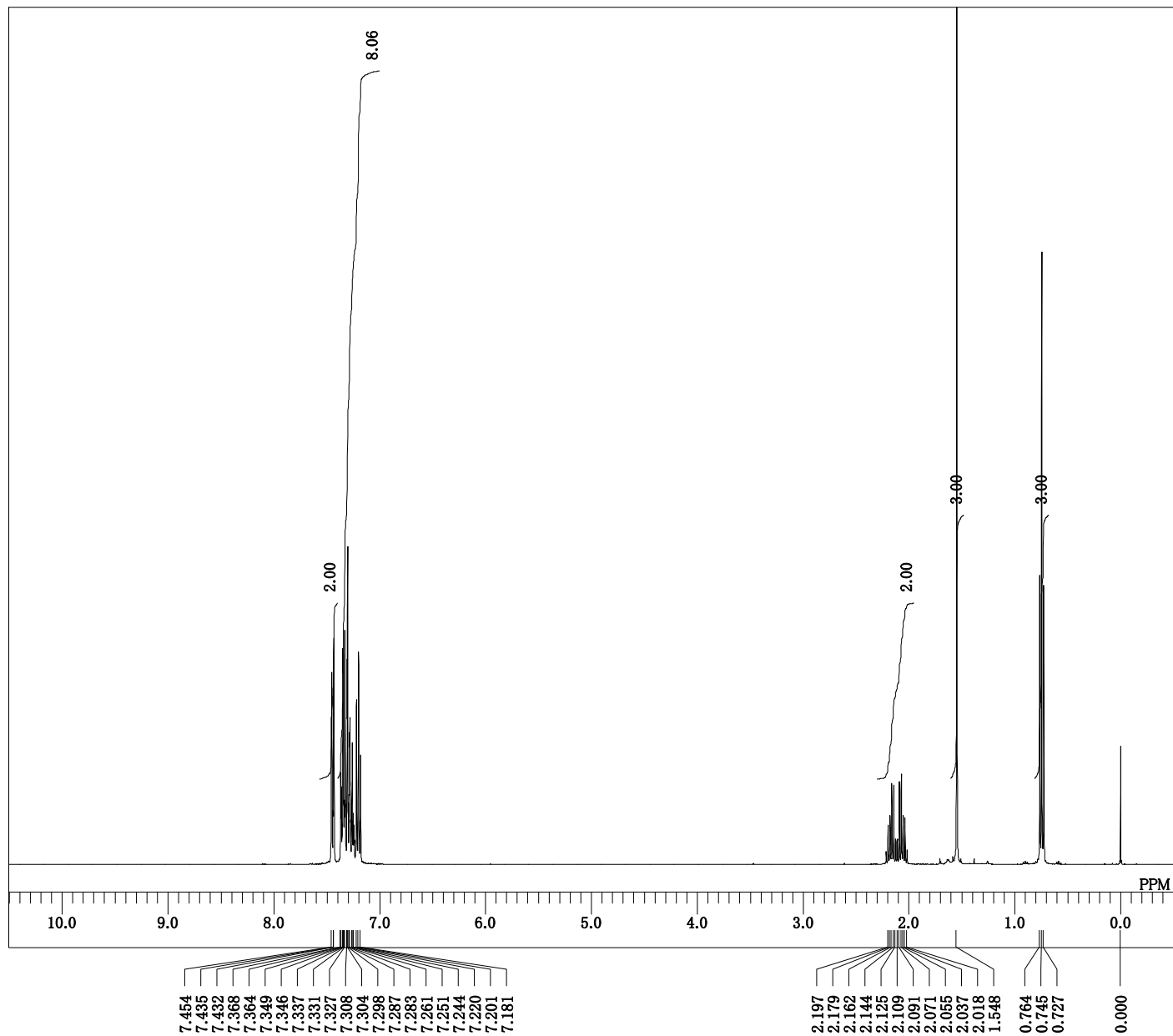


DFILE pro_cyc.Me_Me_Proton.als
 COMNT single_pulse
 DATIM 2020-04-01 14:29:32
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 19.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 26

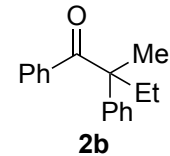


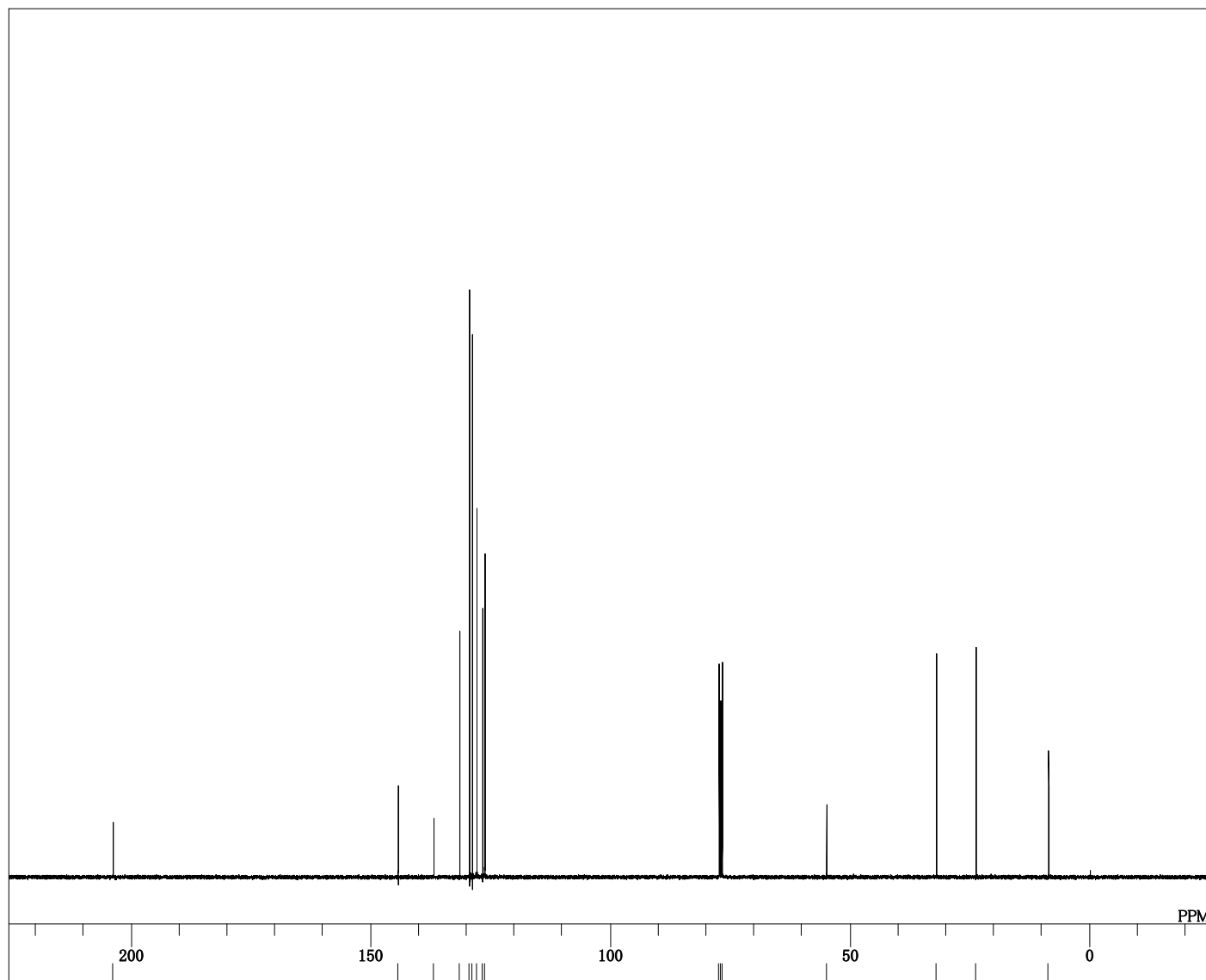
DFILE pro_cyc_Me_Me_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-04-01 14:30:45
 OBNUC ¹³C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 352
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC ¹H
 CTEMP 20.5 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



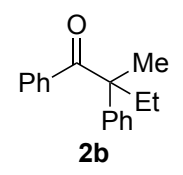


DFILE pro_cyc_Me_Et_Proton.als
 COMNT single pulse
 DATIM 2020-03-31 18:29:32
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 19.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 24





DFILE pro_cyc.Me_Et_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-03-31 18:30:44
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 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



203.769

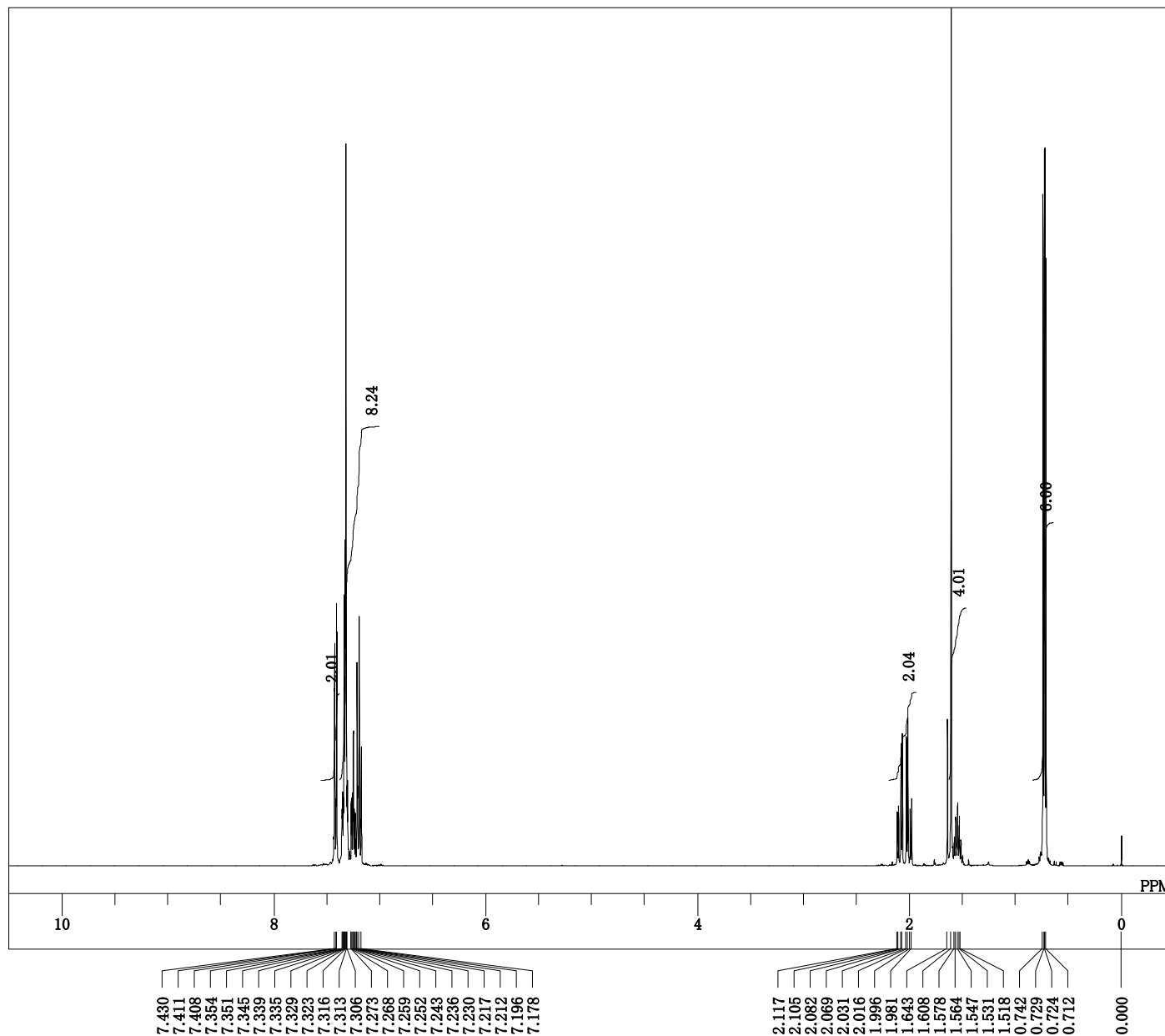
144.322
136.878
131.513
129.405
128.849
127.882
126.723
126.234

77.326
77.000
76.684

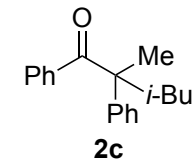
54.917

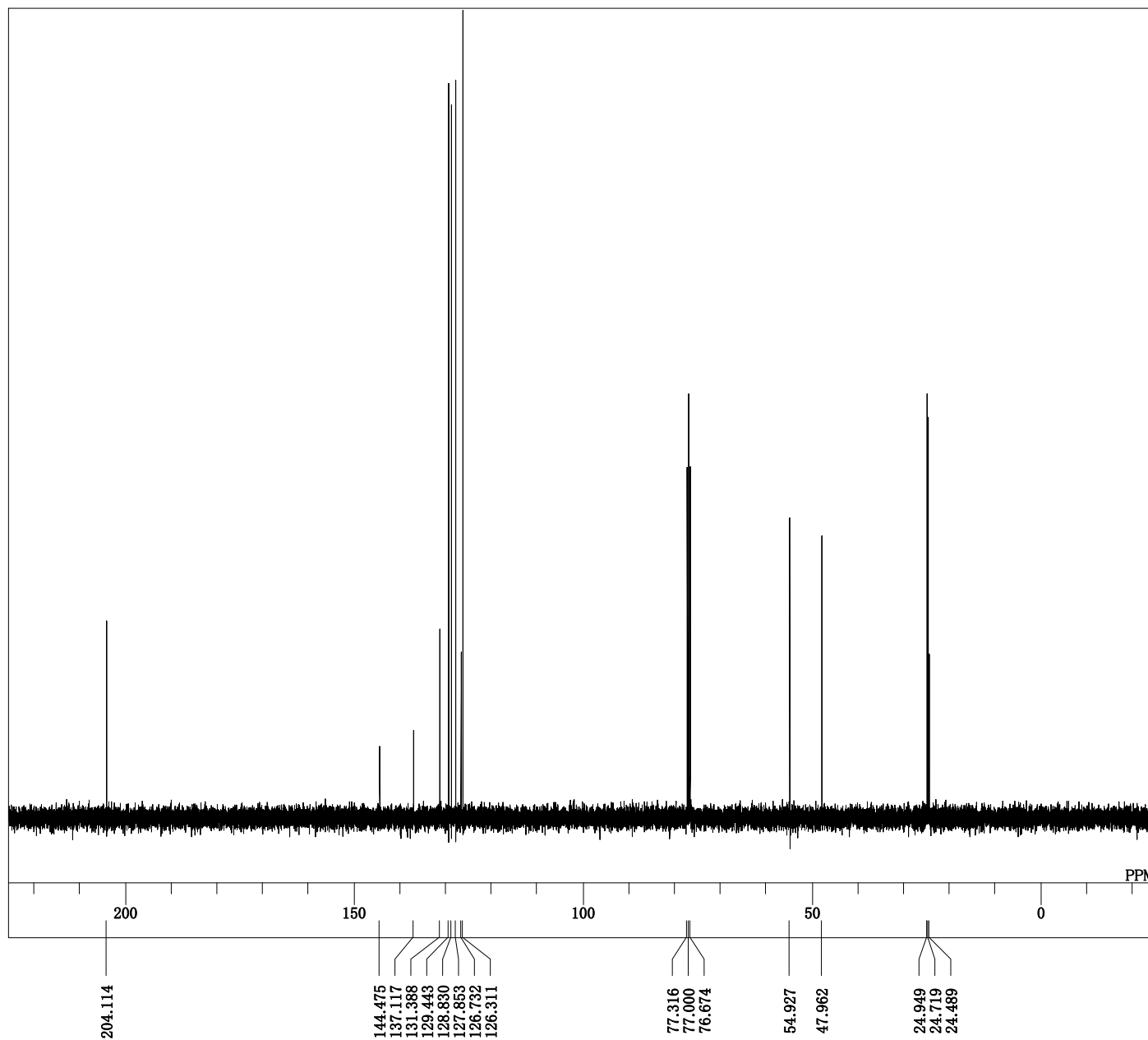
32.020
23.704

8.605

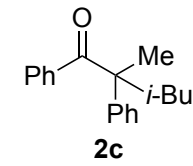


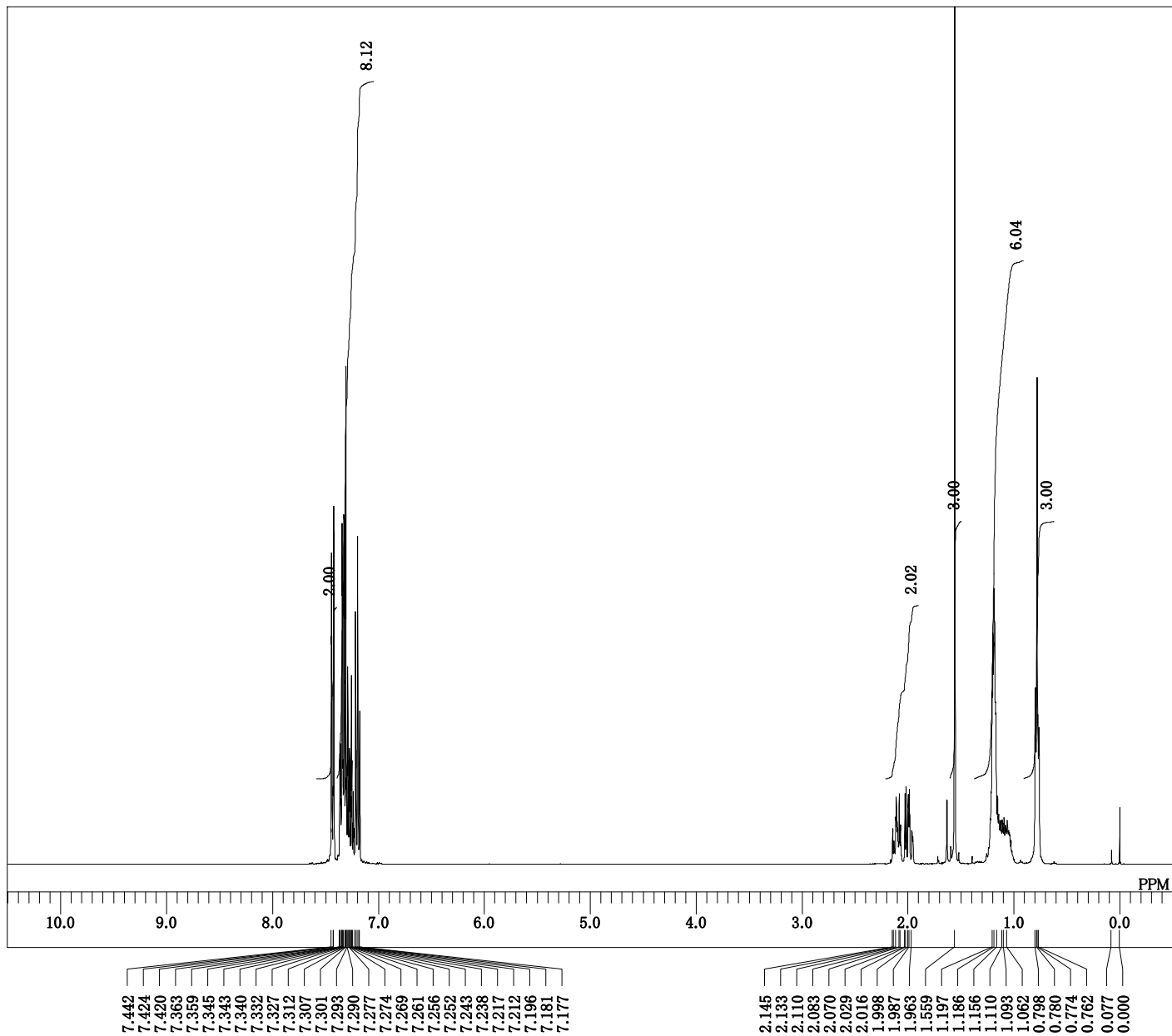
DFILE pro_cyc_Me_iBu_Proton.als
 COMNT single_pulse
 DATIM 2020-07-06 16:45:46
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 24



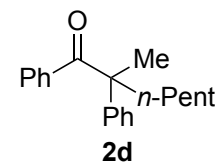


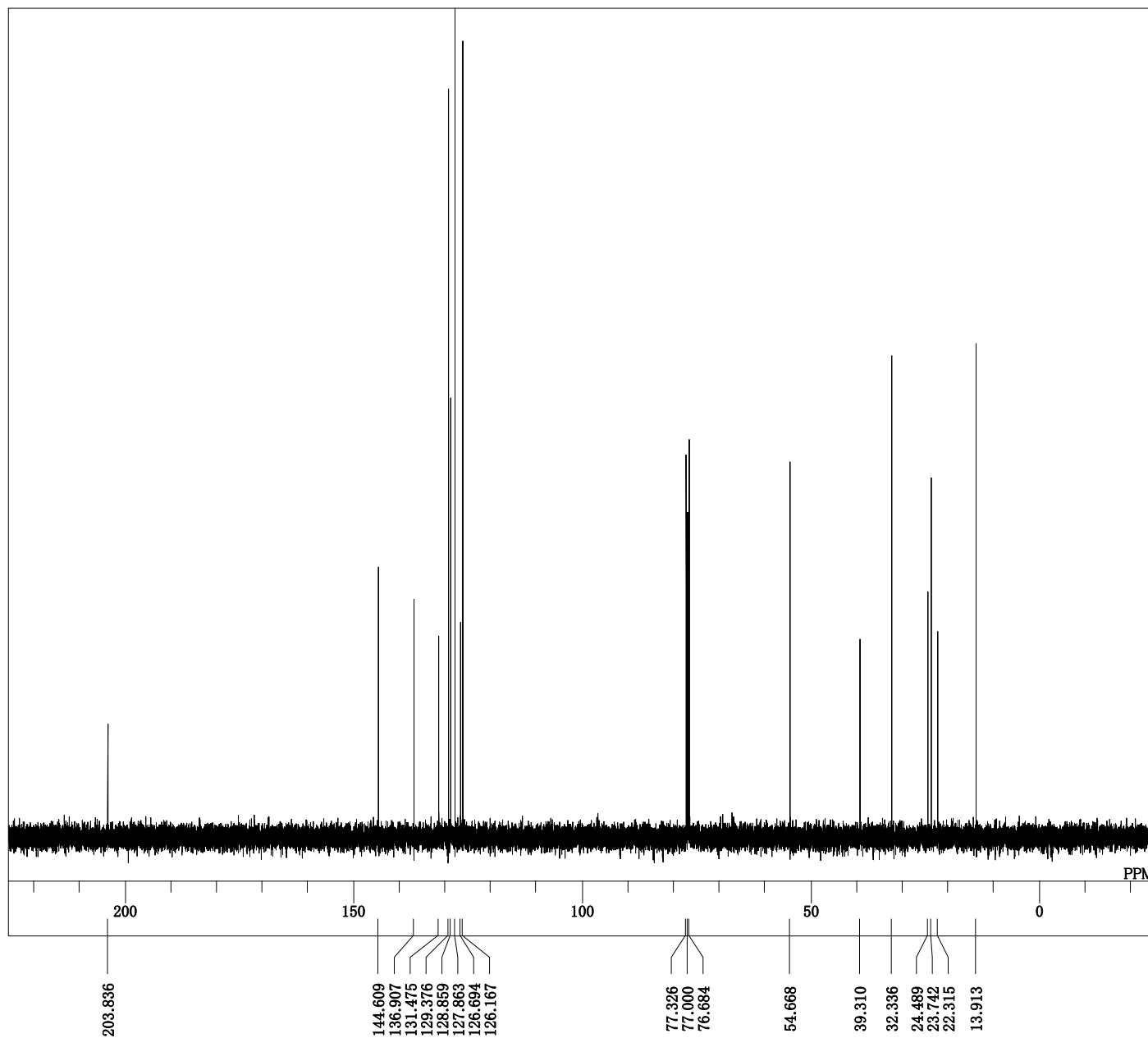
DFILE pro_cyc_Me_iBu_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-07-06 16:46:59
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 88
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 20.9 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



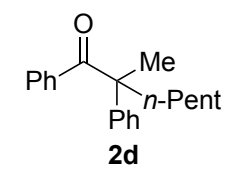


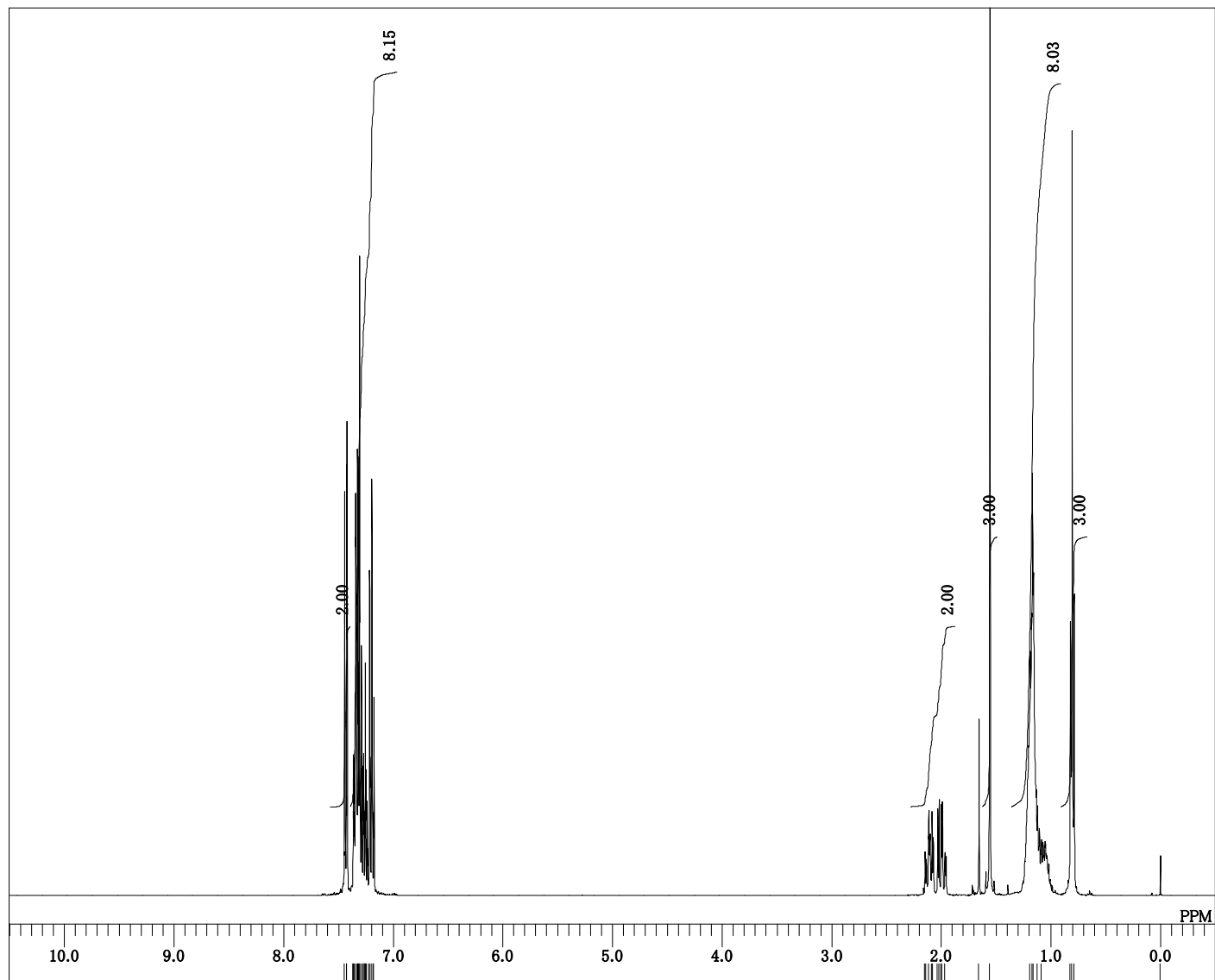
DFILE pro_cyc.Me_nPentylProton.als
 COMNT single_pulse
 DATIM 2020-07-05 16:55:24
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 22





DFILE pro_cyc.Me_nPentyl_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-07-05 16:56:36
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 64
 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

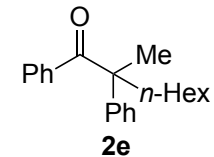


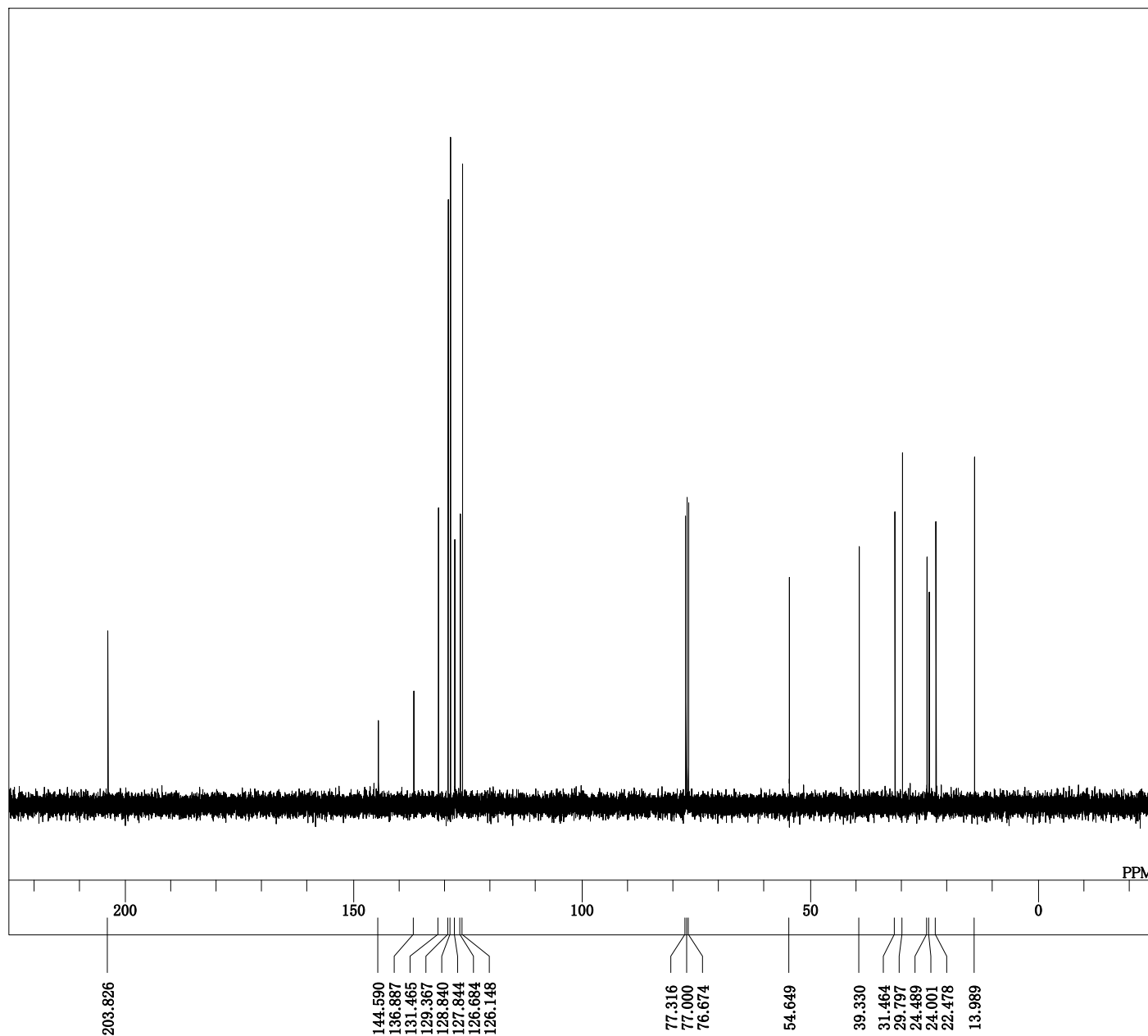


7.442
7.424
7.420
7.363
7.357
7.354
7.344
7.327
7.320
7.316
7.312
7.307
7.300
7.290
7.276
7.273
7.260
7.254
7.251
7.242
7.237
7.216
7.211
7.195
7.180
7.176

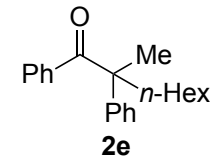
2.148
2.135
2.114
2.085
2.073
2.032
2.020
2.002
1.990
1.967
1.656
1.556
1.188
1.167
1.159
1.122
1.085
0.823
0.806
0.787
0.000

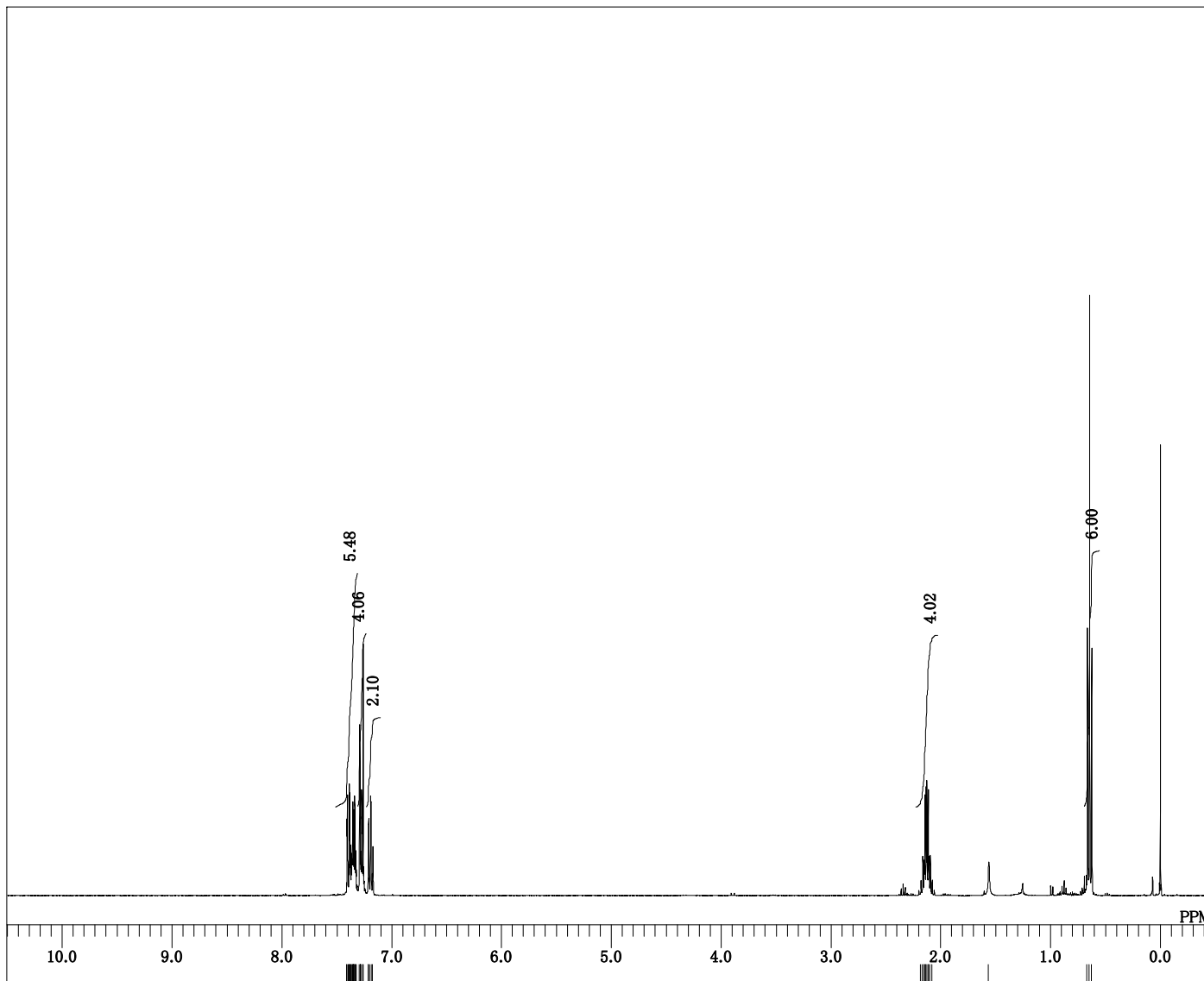
DFILE pro_cyc_Me_nHexyl_Proton.als
COMNT single_pulse
DATIM 2020-07-06 16:37:37
OBNUC 1H
EXMOD proton_jxp
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.24 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 3.14 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 20



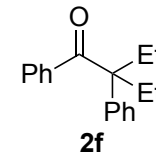


DFILE pro_cyc_Me_nHexyl_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-07-06 16:38:50
 OBNUC ¹³C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 33
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC ¹H
 CTEMP 20.2 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50





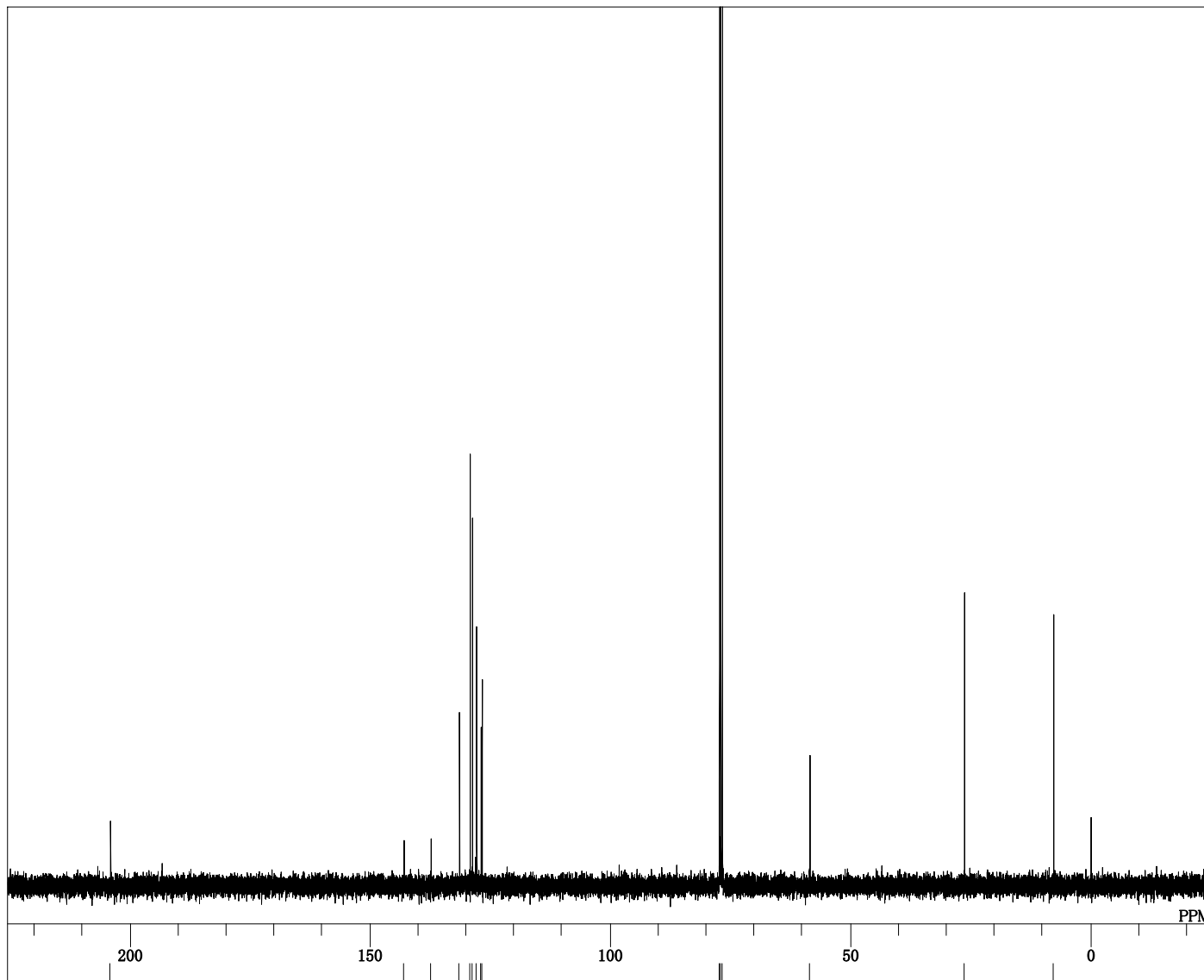
DFILE pro_cyc_Et_Et_Proton.als
 COMNT single_pulse
 DATIM 2020-04-02 09:15:34
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.1 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 40



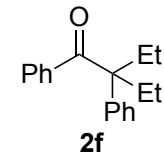
7.407
 7.403
 7.391
 7.385
 7.382
 7.375
 7.368
 7.364
 7.355
 7.353
 7.346
 7.341
 7.338
 7.331
 7.328
 7.324
 7.316
 7.290
 7.283
 7.279
 7.274
 7.261
 7.256
 7.210
 7.206
 7.192
 7.176
 7.171

2.180
 2.163
 2.150
 2.145
 2.132
 2.125
 2.113
 2.107
 2.094
 2.077
 1.561

0.663
 0.644
 0.625



DFILE pro_cyc.Et_Et_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-04-02 09:16:46
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 928
 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



204.104

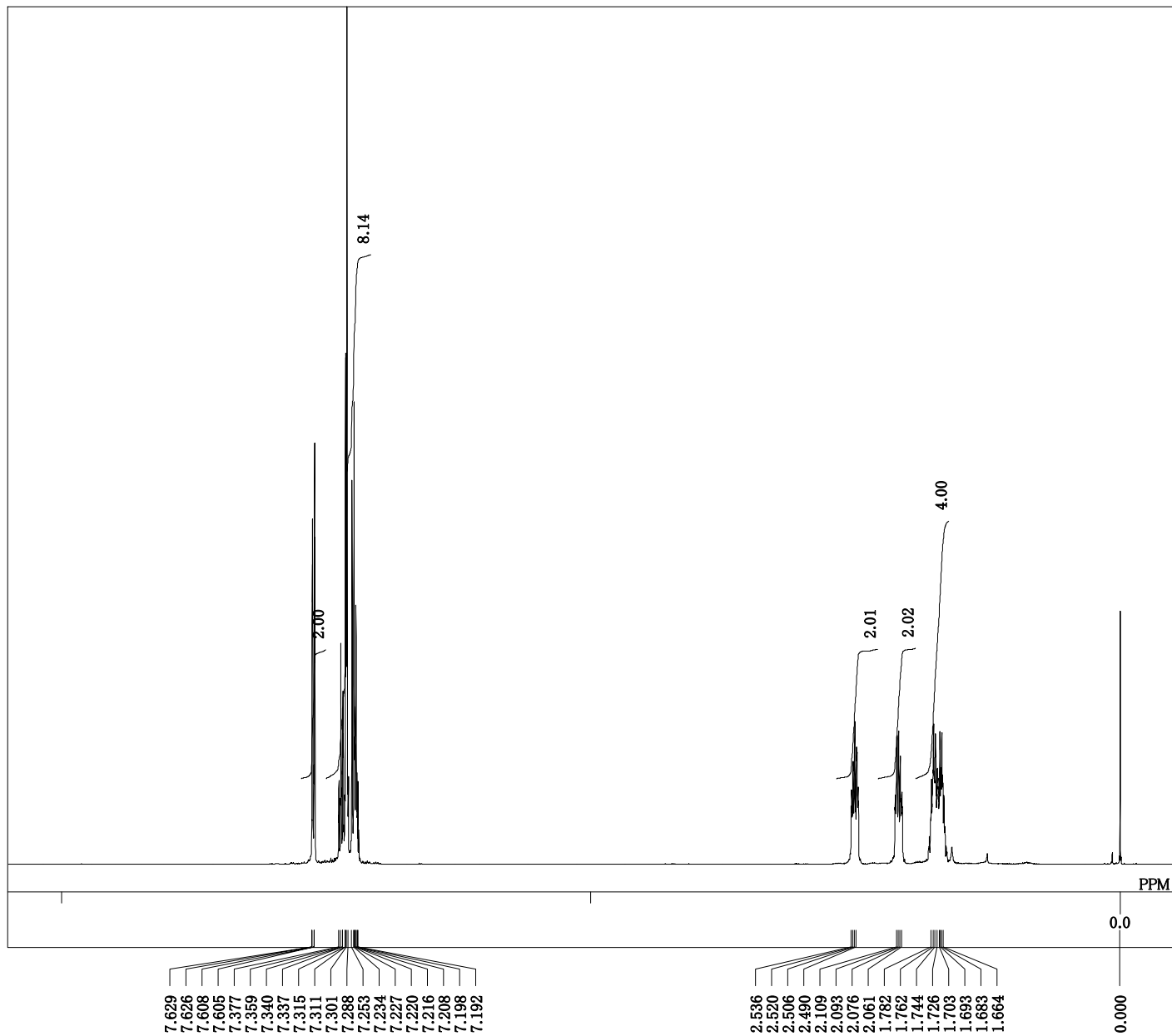
142.990
137.357
131.446
129.261
128.725
127.863
126.972
126.770

77.316
77.000
76.674

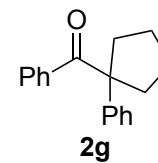
58.529

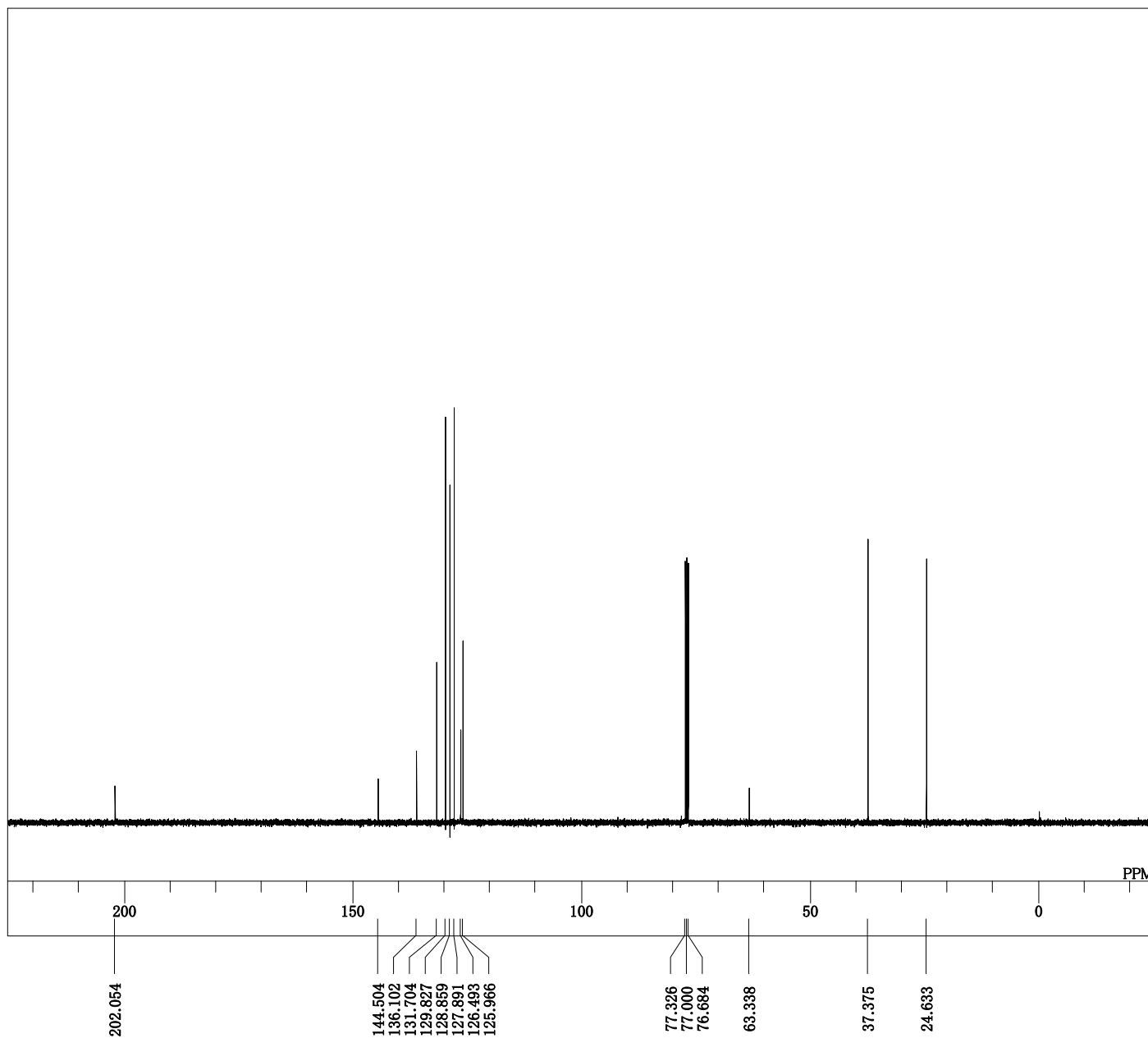
26.300

7.772

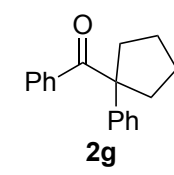


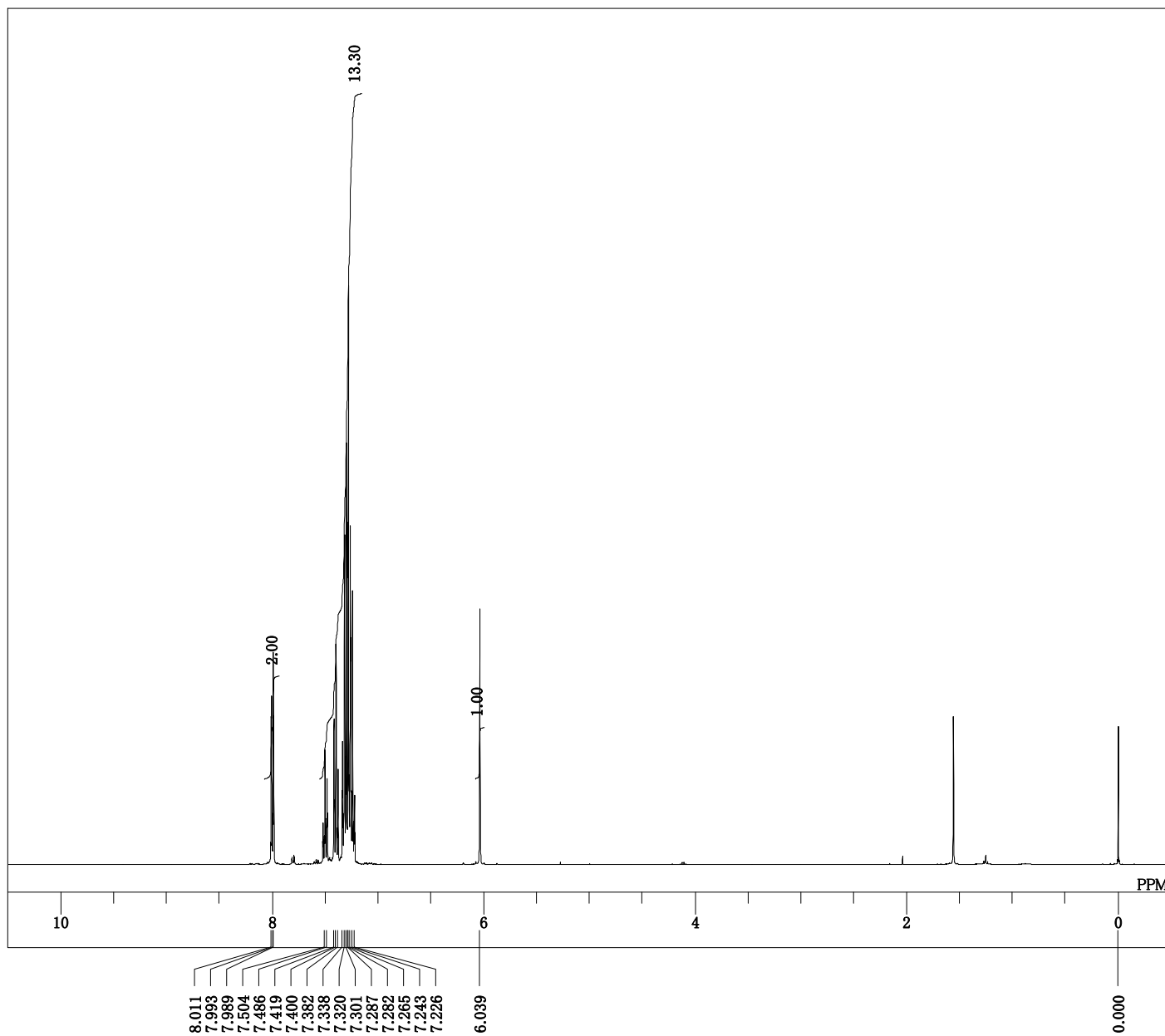
DFILE pro_cyc_cyc_pentylProton.als
 COMNT single_pulse
 DATIM 2020-04-02 10:15:58
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 28



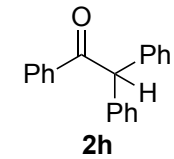


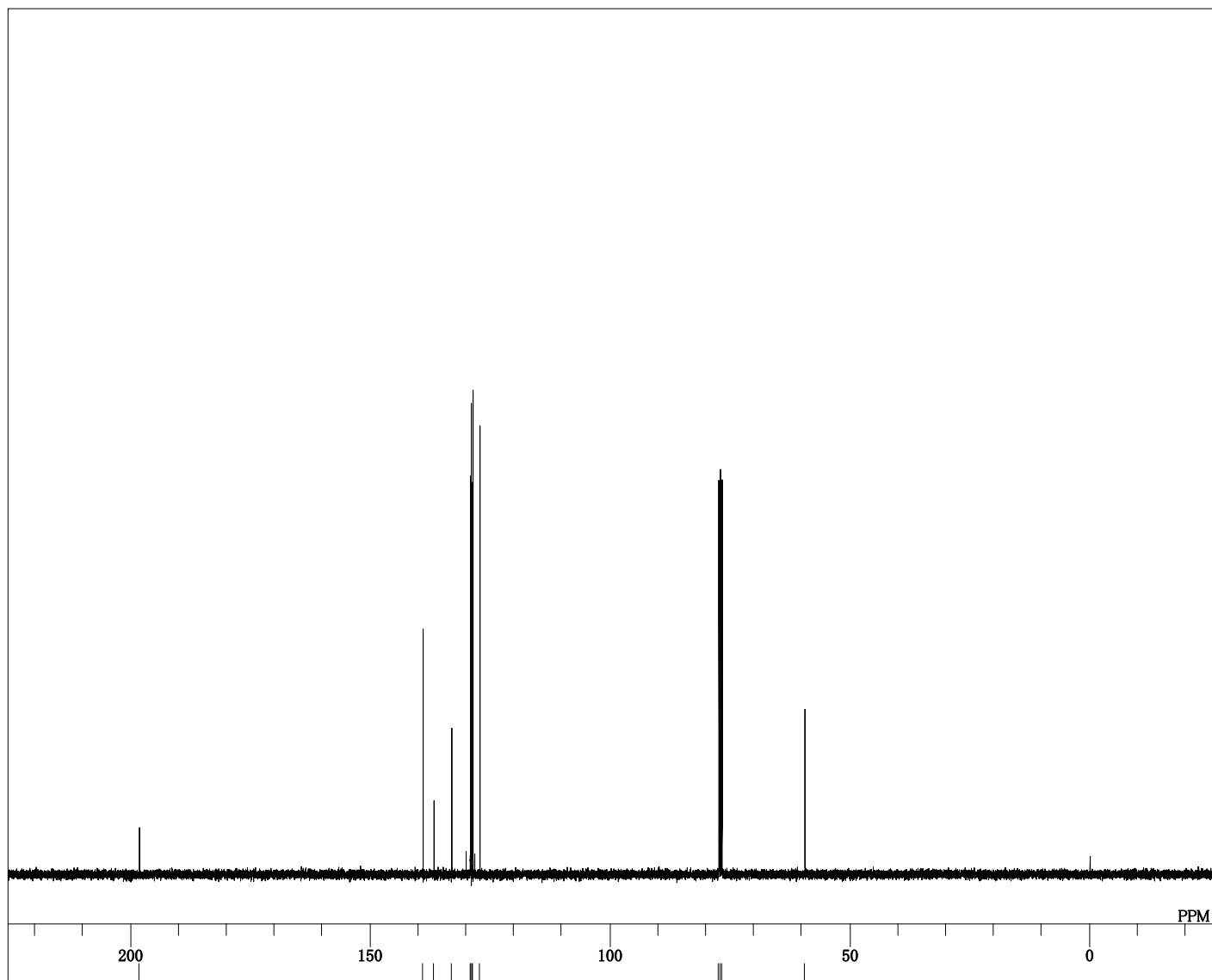
DFILE pro_cyc_cyc.pentyl_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-04-02 10:17:11
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 452
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 20.3 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



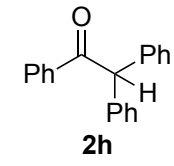


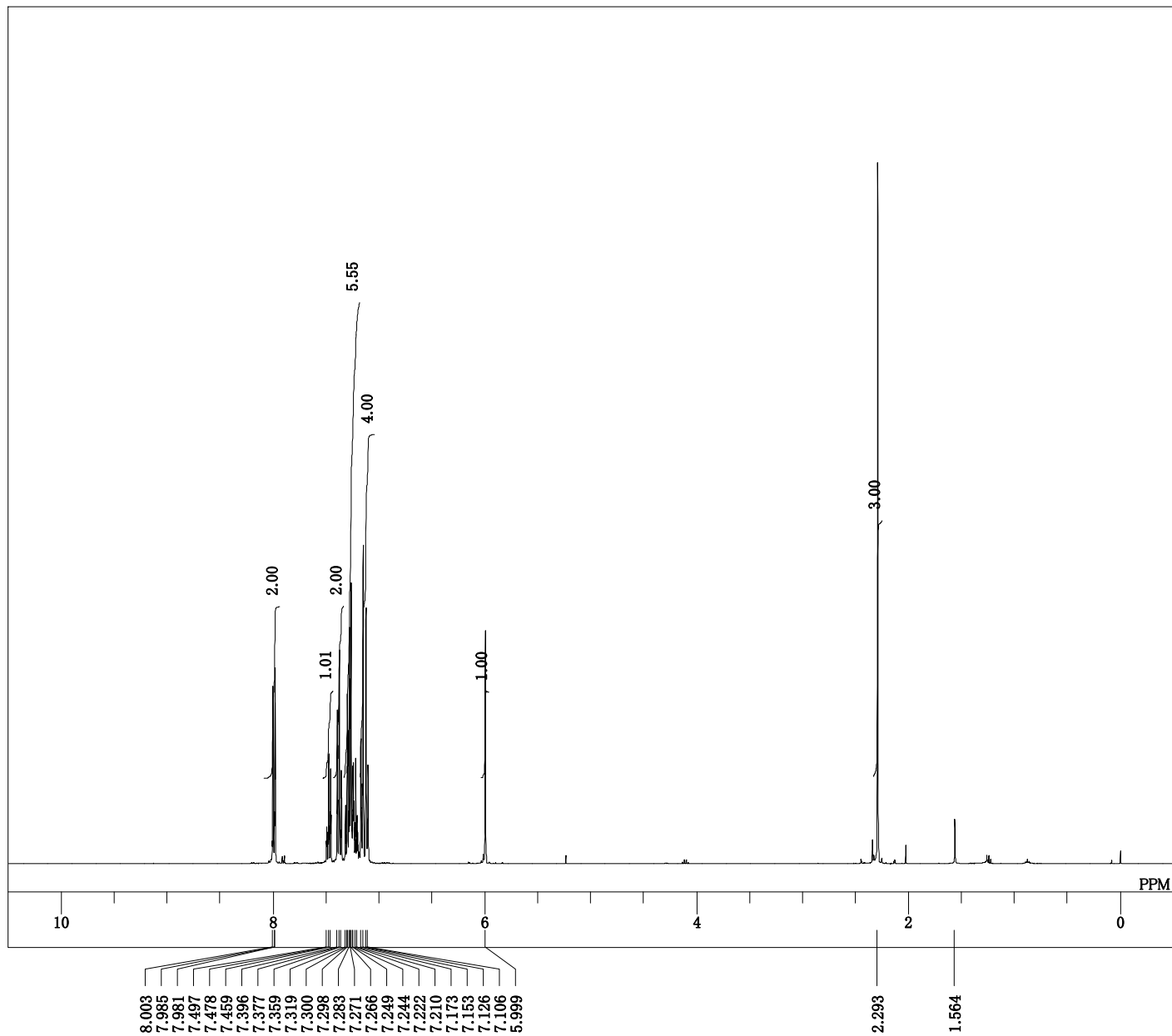
DFILE pro_cyc_H_Ph_Proton.als
 COMNT single_pulse
 DATIM 2020-06-10 15:59:34
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 32



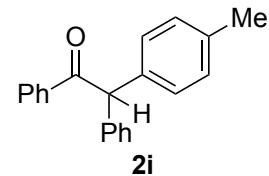


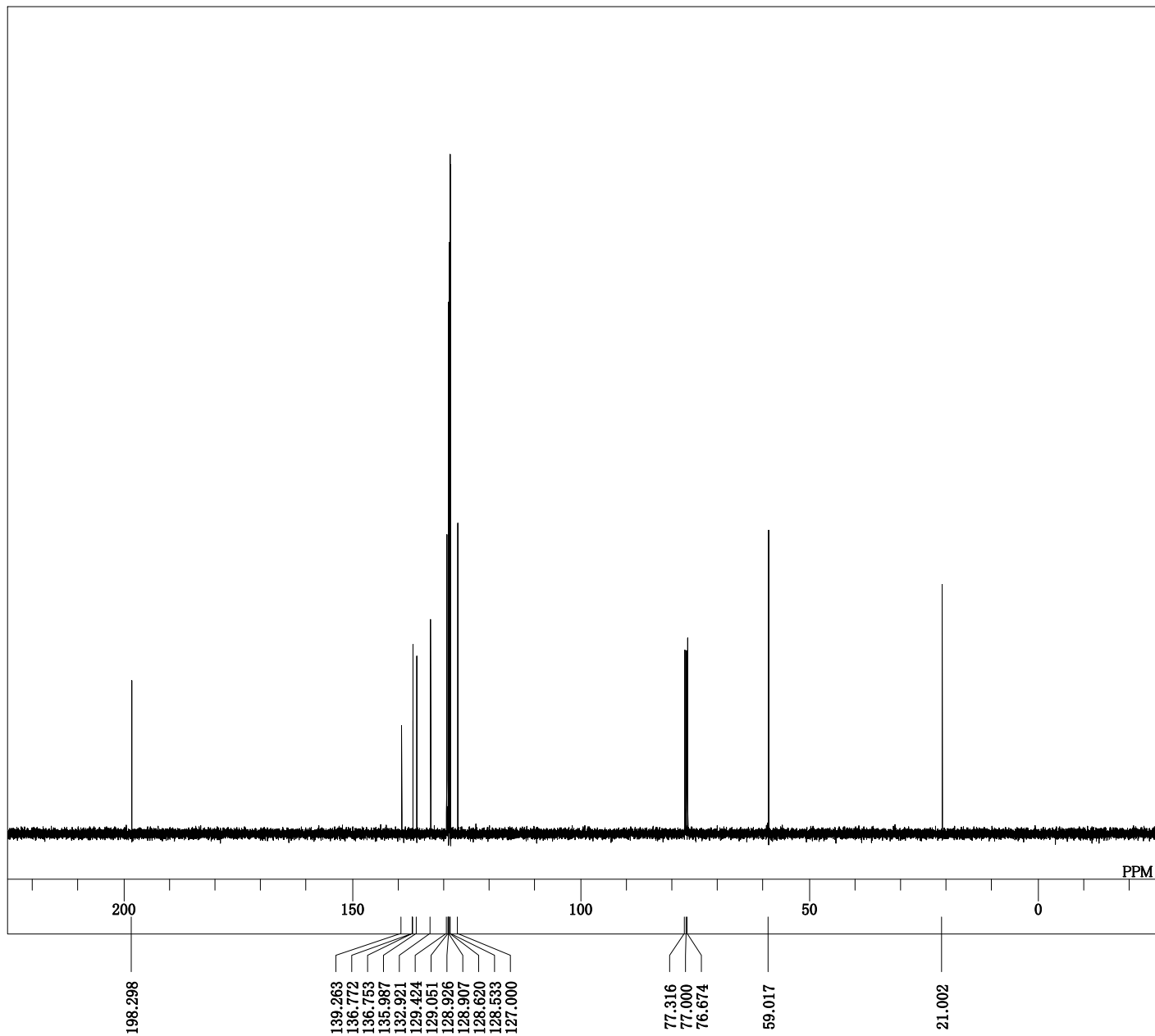
DFILE pro_cyc_H_Ph_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-06-10 16:00:46
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 426
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 20.0 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



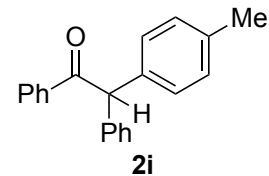


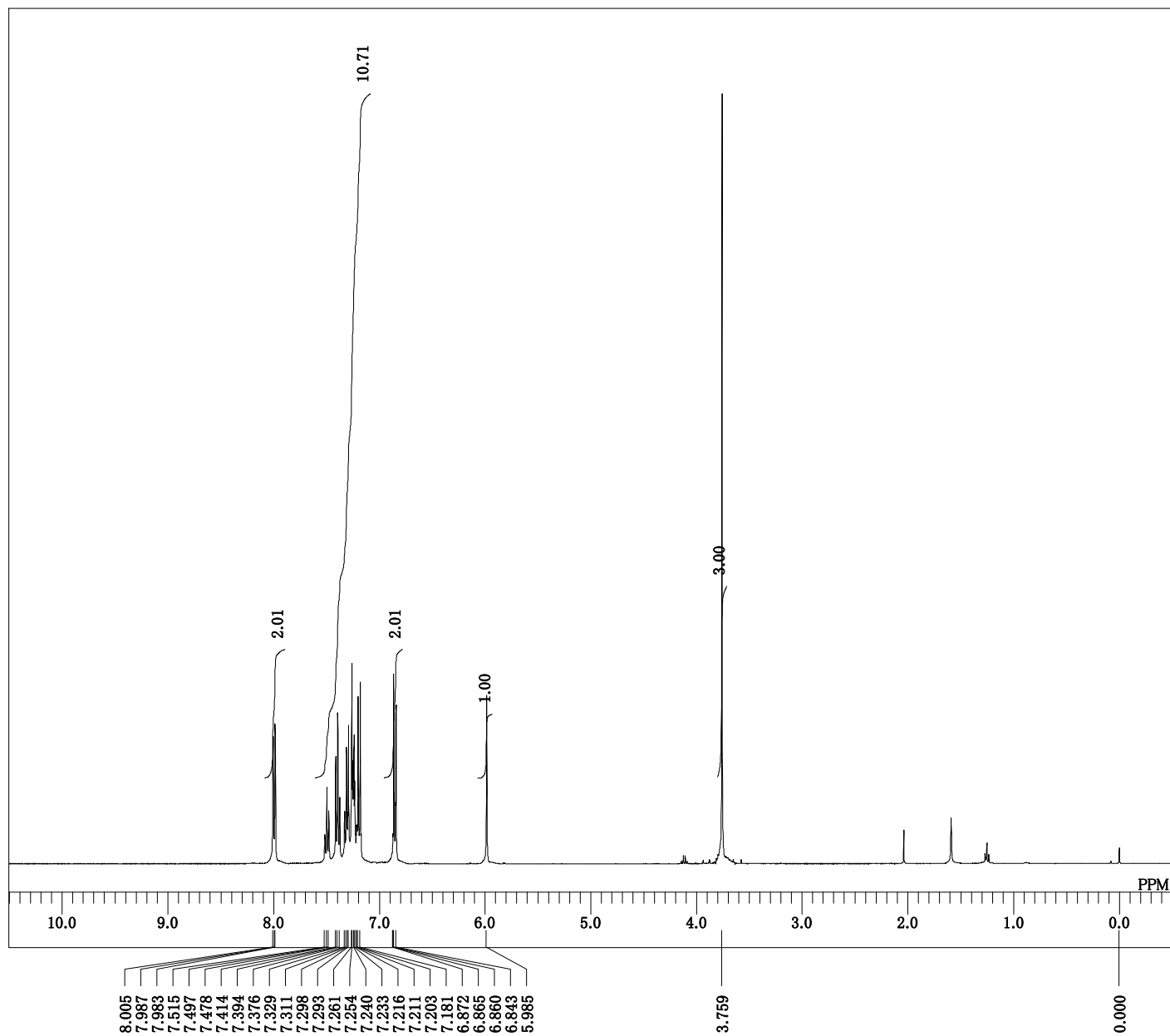
DFILE pro_cyc_HpMe_Proton.als
 COMNT single_pulse
 DATIM 2020-07-05 17:05:02
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.1 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 24



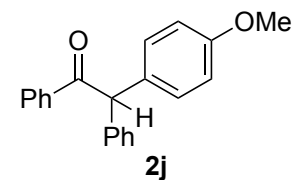


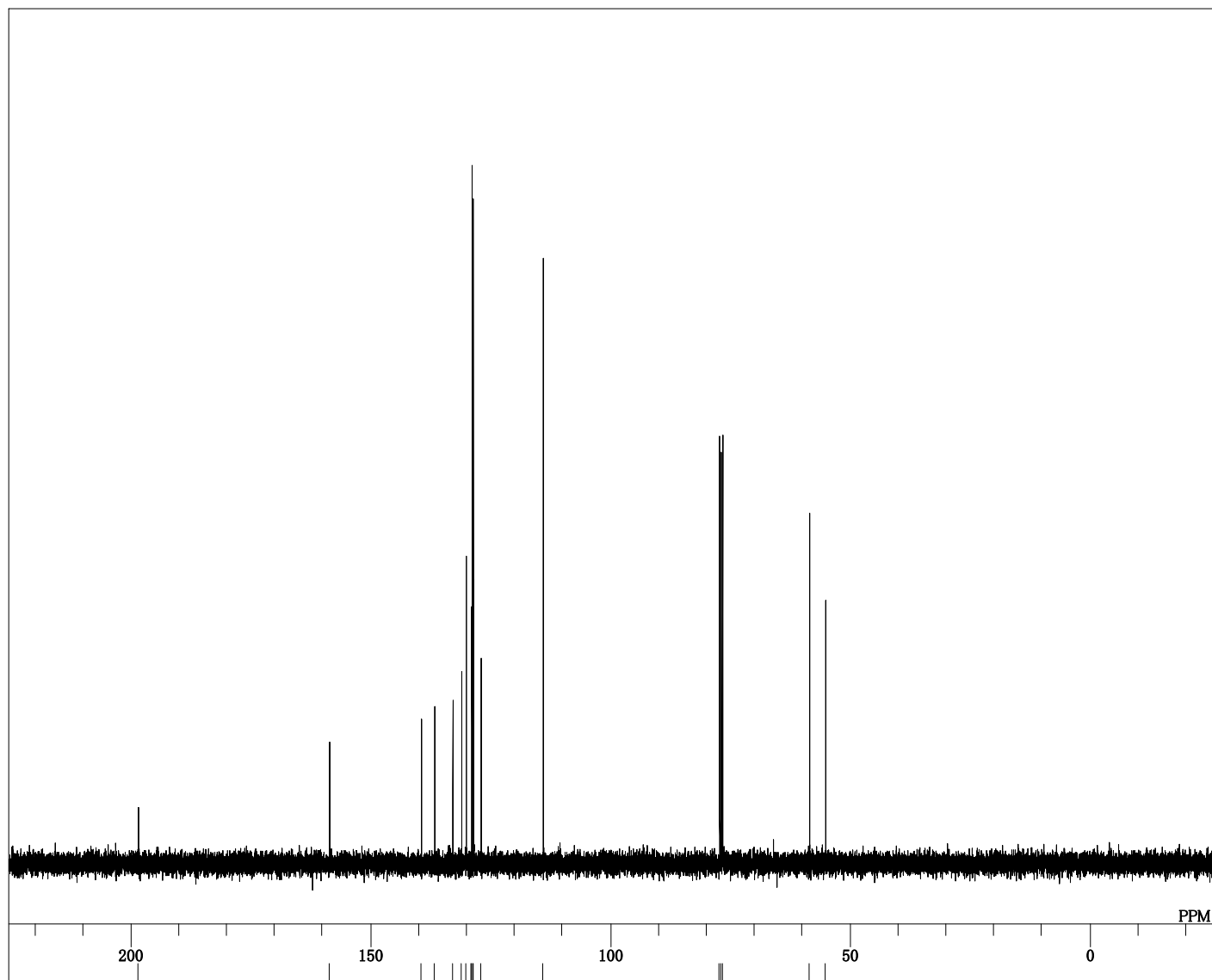
DFILE pro_cyc_H_pMe_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-07-05 17:06:15
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 149
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 20.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50





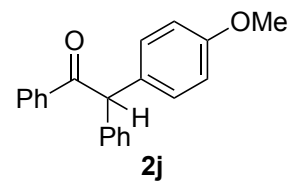
DFILE pro_cyc_HpOMe_Proton.als
 COMNT auto
 DATIM Sat Sep 01 12:39:05 2018
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 16384
 FREQU 7992.01 Hz
 SCANS 8
 ACQTM 2.0500 sec
 PD 2.0000 sec
 PW1 6.60 usec
 IRNUC 1H
 CTEMP 24.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 13





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DFILE      pro_cyc_HpOMe_Carbon.als
COMNT      single pulse decoupled gated NOE
DATIM      2020-07-06 16:57:41
OBNUC      13C
EXMOD      carbon.jxp
OBFRQ      99.55 MHz
OBSET      5.13 KHz
OBFIN      0.98 Hz
POINT      26214
FREQU      25000.00 Hz
SCANS      90
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
  
```



198.442

158.616

139.426

136.763

132.950

131.072

130.133

129.003

128.917

128.648

128.562

127.020

114.115

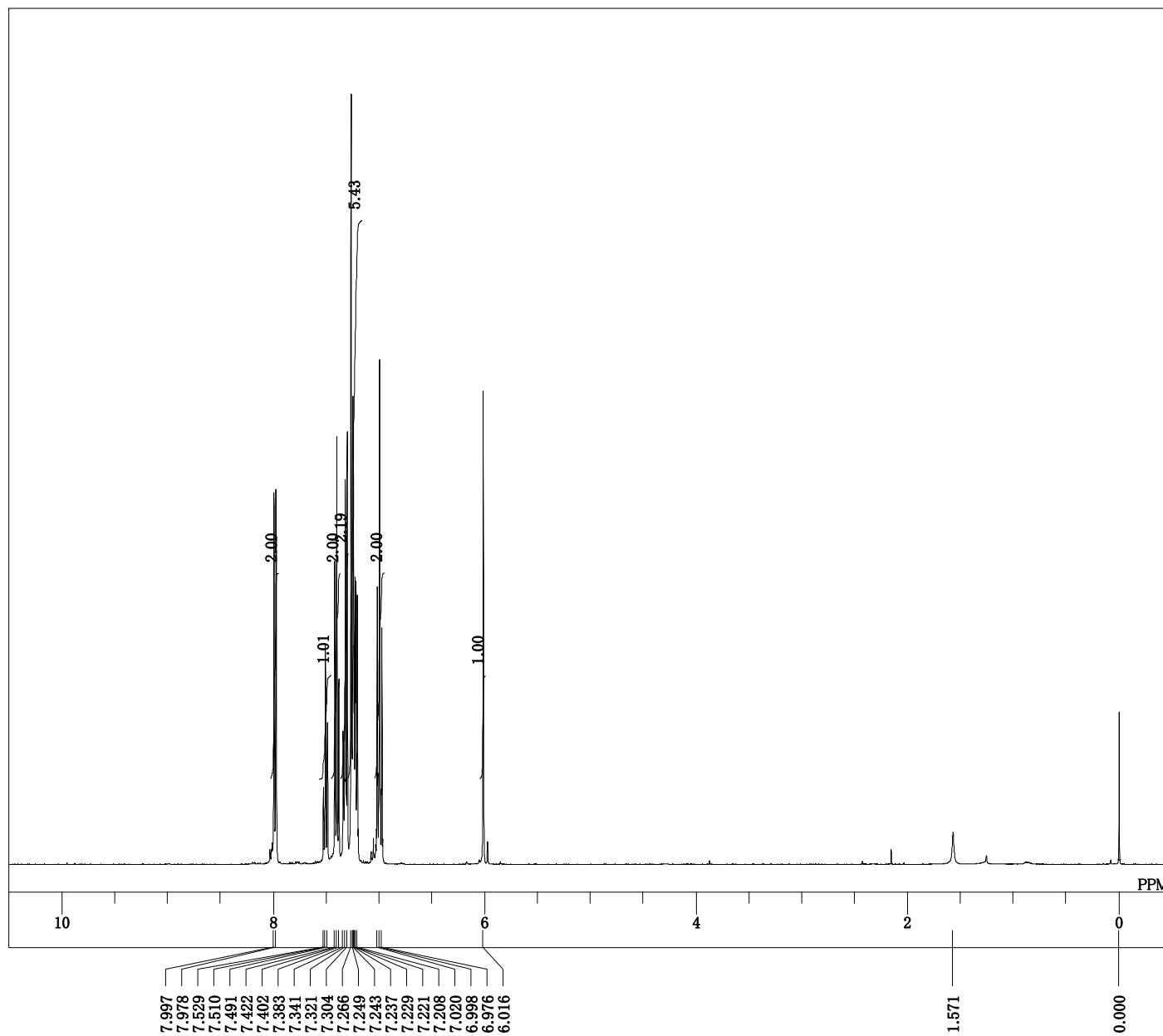
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77.000

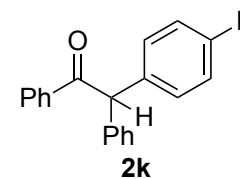
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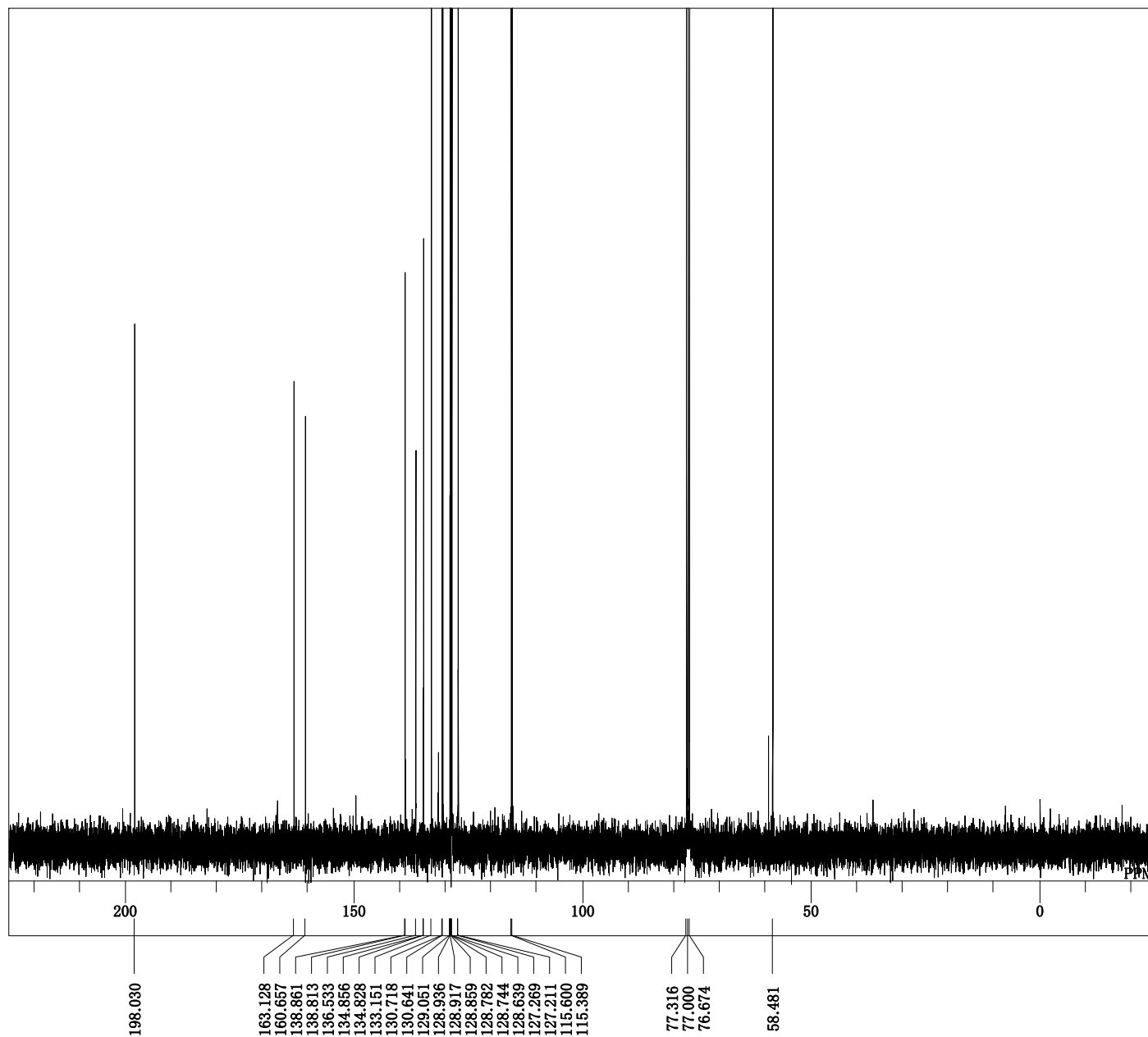
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55.176

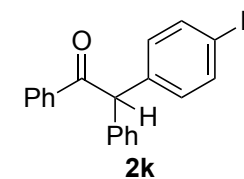


DFILE pro_diol_H_pF_fr2_Proton.als
 COMNT single_pulse
 DATIM 2020-03-28 11:45:02
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 28

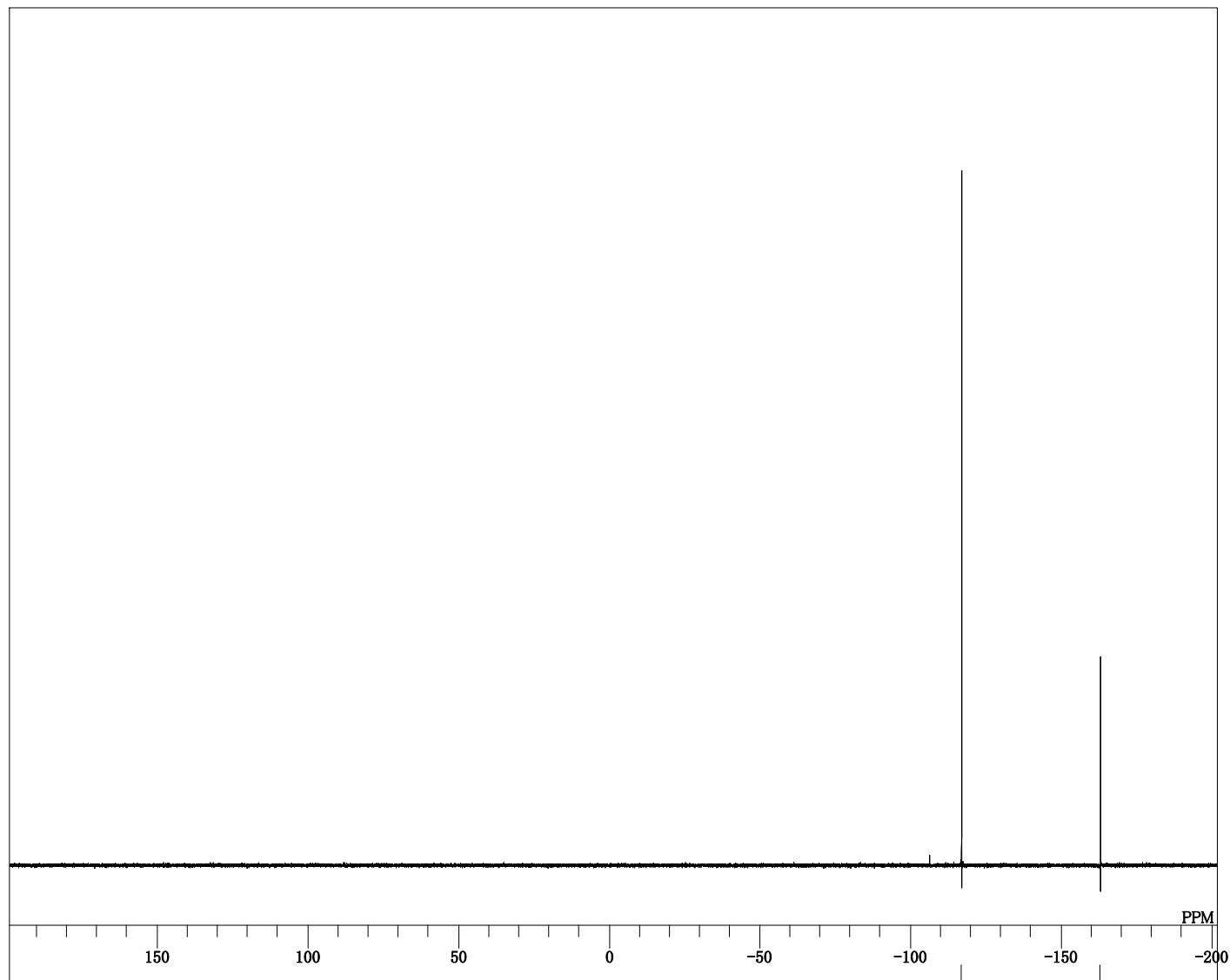
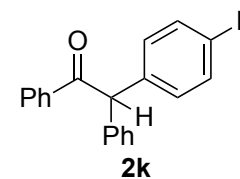


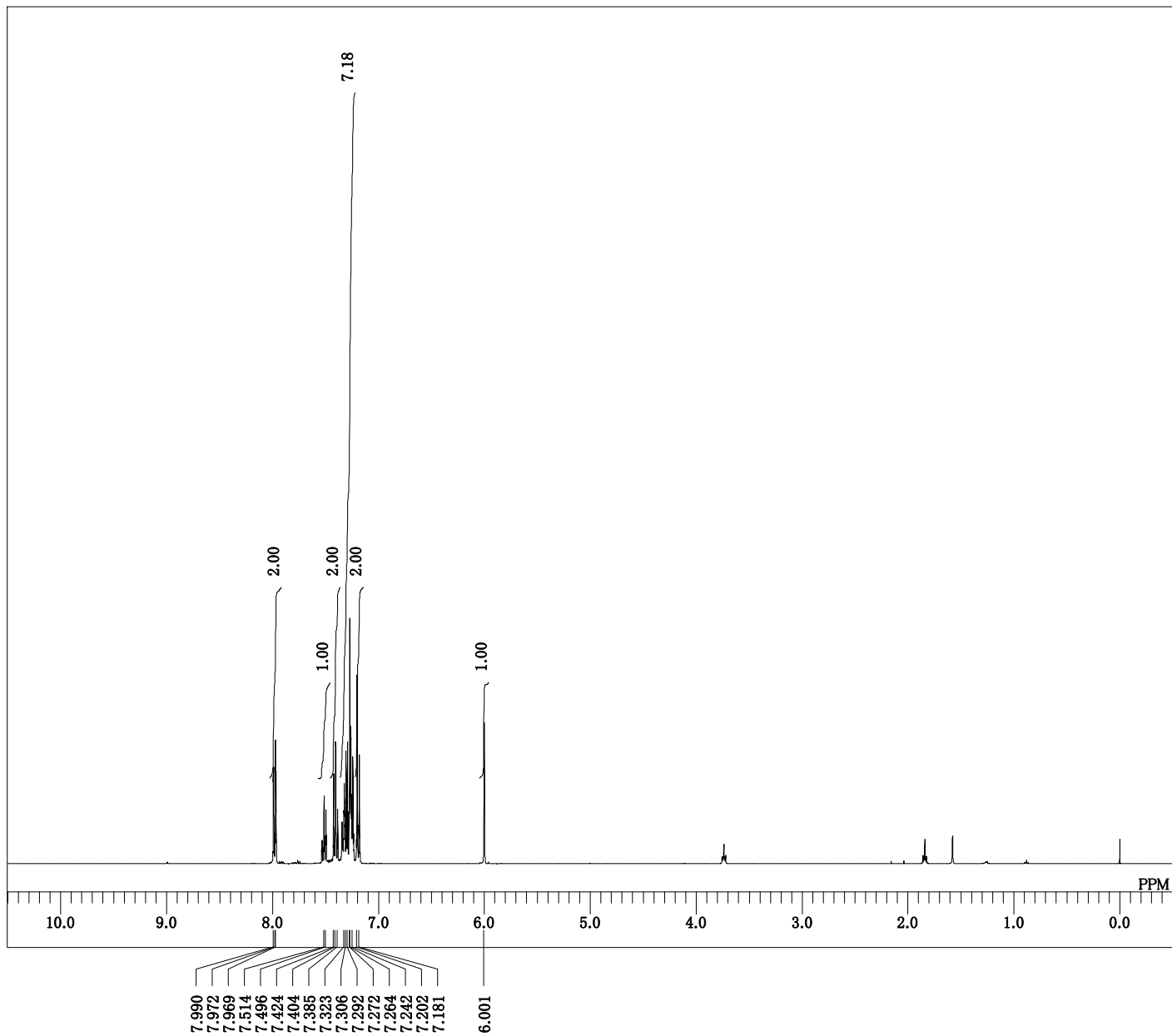


DFILE pro_diol_H_pF_fr2_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-03-28 11:46:14
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 1024
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 20.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50

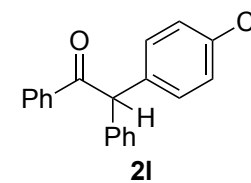


DFILE pro_cyc_H_pF_Fluorine.als
COMNT single_pulse
DATIM 2022-07-04 13:48:27
OBNUC 19F
EXMOD single_pulse.ixp
OBFRQ 372.50 MHz
OBSET 3.36 KHz
OBFIN 6.86 Hz
POINT 26214
FREQU 149253.73 Hz
SCANS 8
ACQTM 0.1756 sec
PD 5.0000 sec
PW1 3.98 usec
IRNUC 19F
CTEMP 20.7 c
SLVNT CDCL3
EXREF -163.00 ppm
BF 0.12 Hz
RGAIN 42

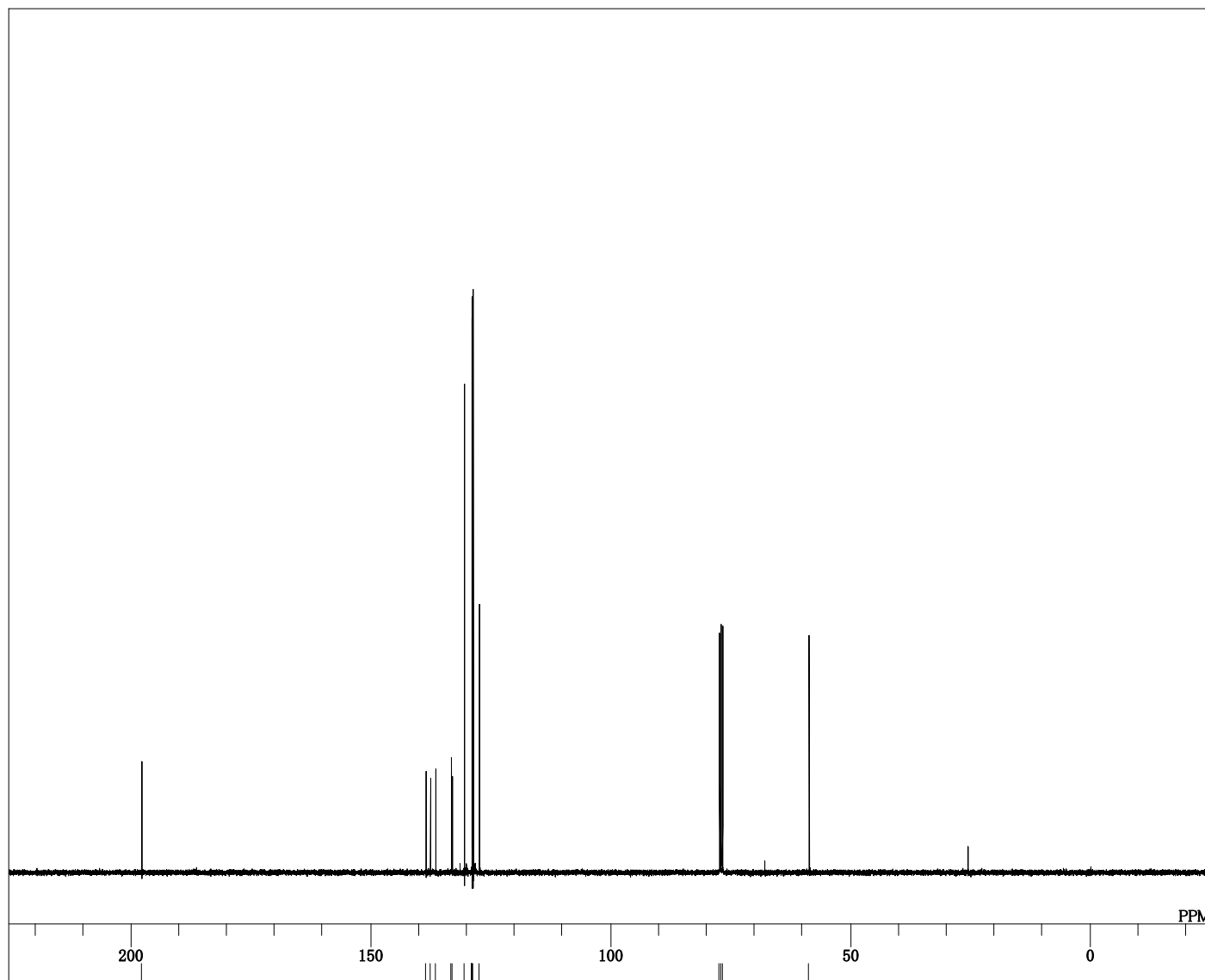




DFILE pro_cyc_H_pCLProton.als
 COMNT single_pulse
 DATIM 2020-06-25 09:51:57
 OBNUC ¹H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC ¹H
 CTEMP 20.6 c
 SLVNT CDCL₃
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 28



DFILE pro_cyc_HpClCarbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-06-25 09:53:10
 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 957
 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

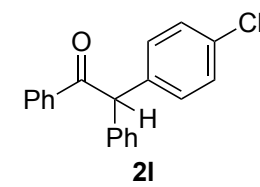


197.781

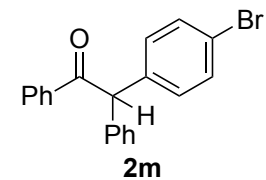
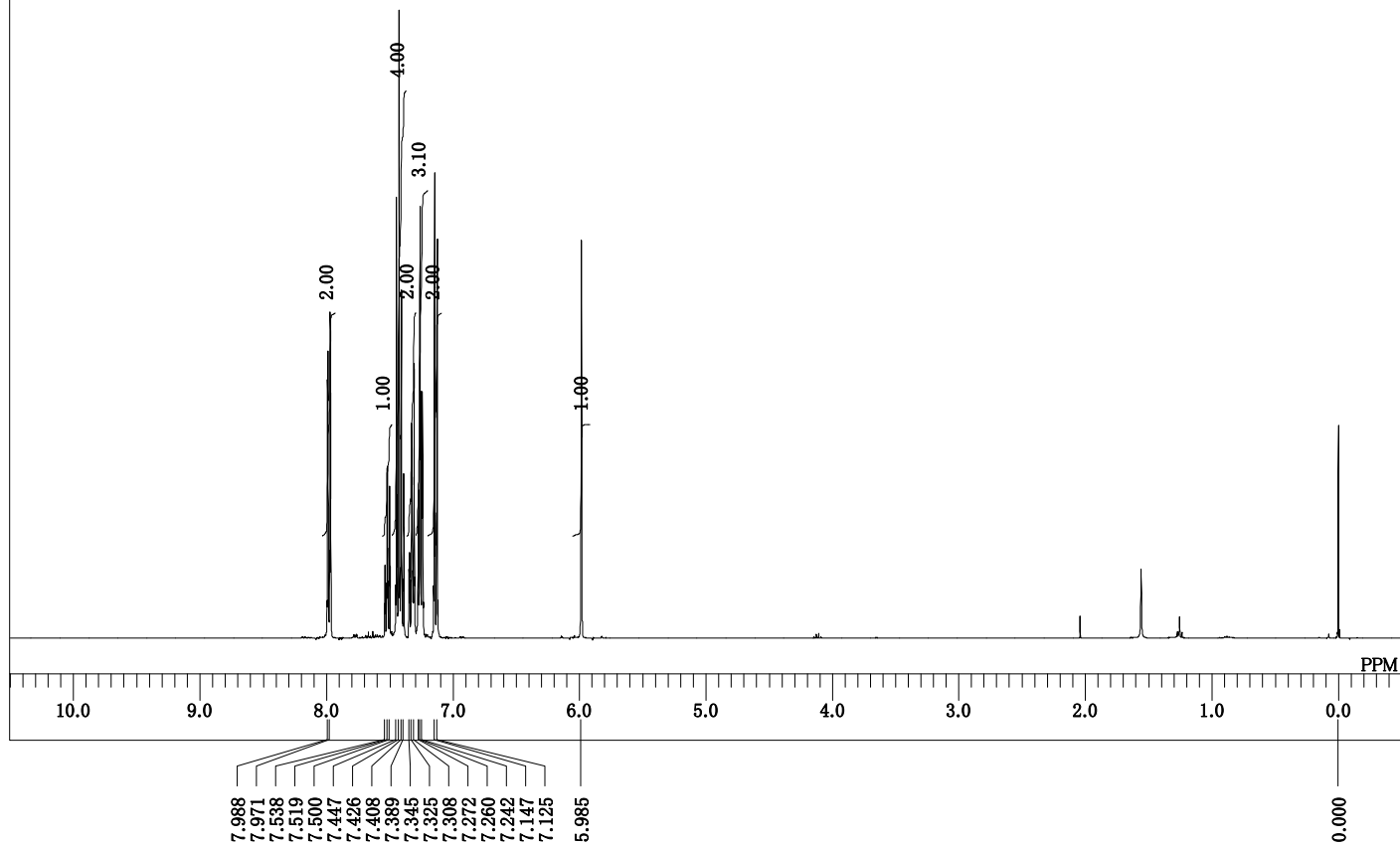
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 133.209
 133.065
 130.488
 128.945
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 128.773
 128.658
 127.355

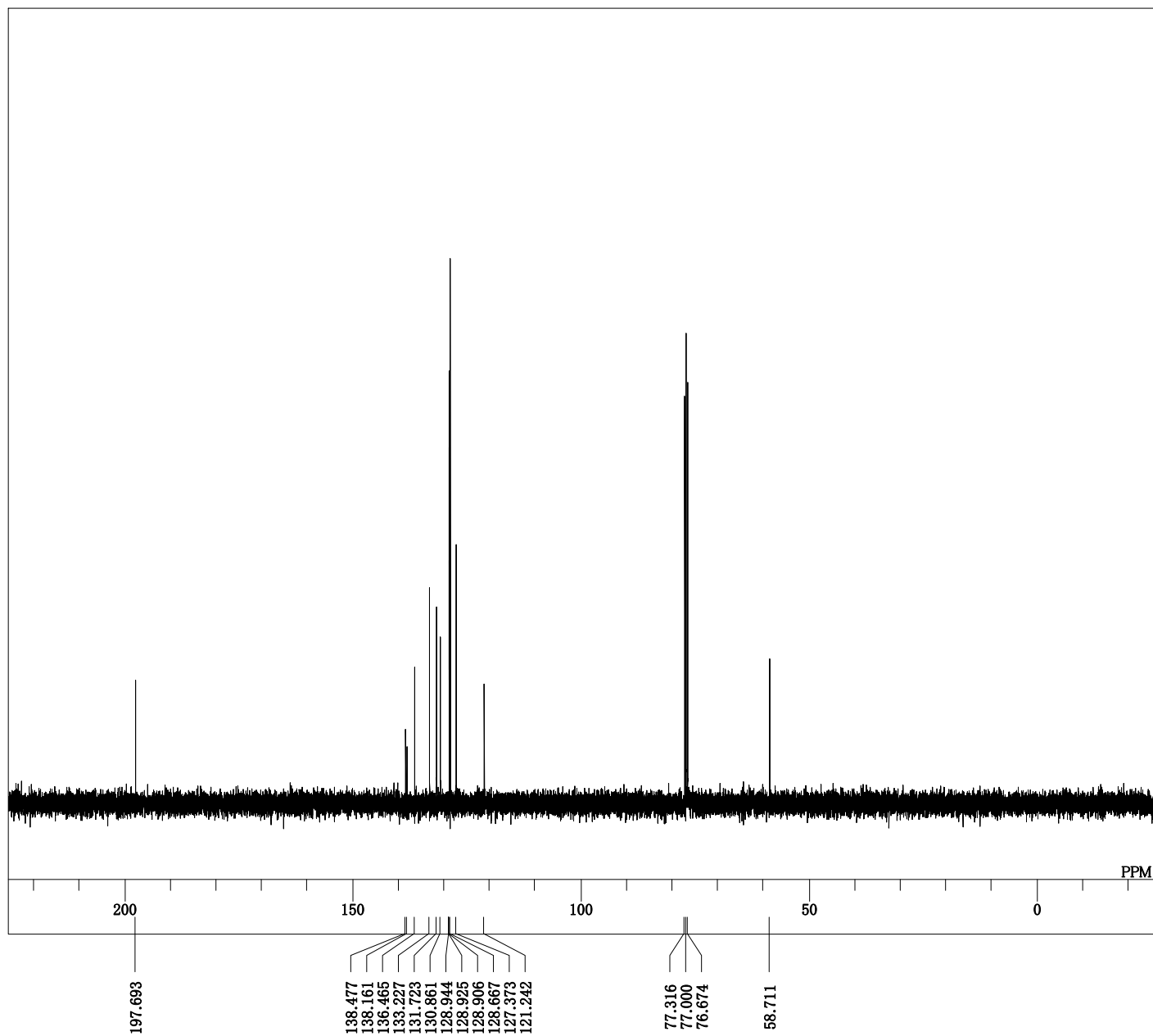
77.316
 77.000
 76.674

58.634

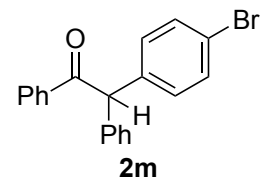


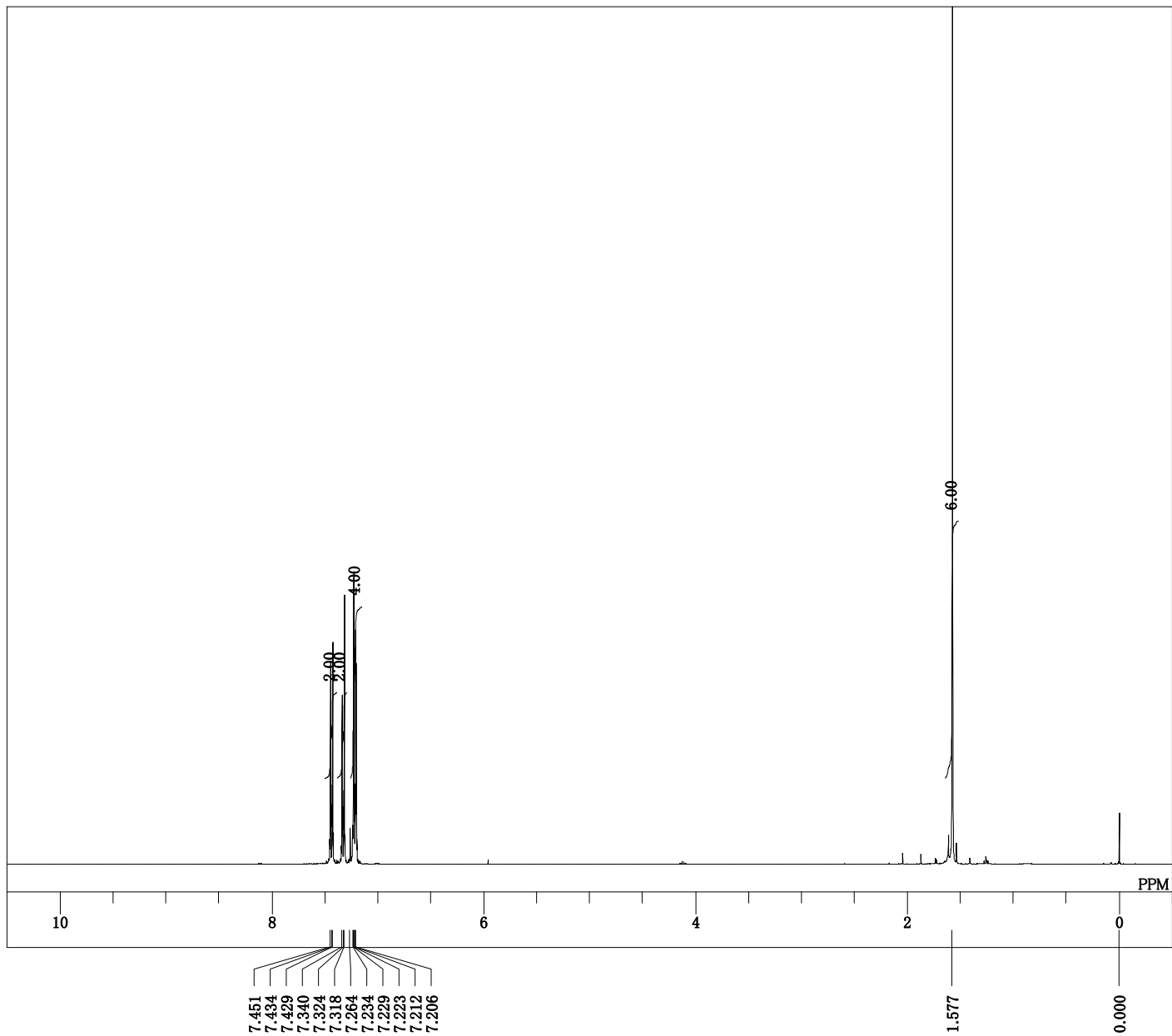
DFILE Ex655_fr1_20220411_Proton-1-1.a
 COMNT single_pulse
 DATIM 2022-04-11 14:21:34
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.25 usec
 IRNUC 1H
 CTEMP 21.0 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 32



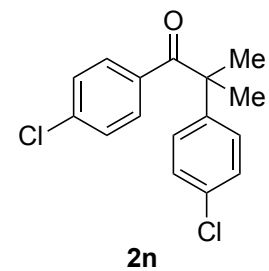


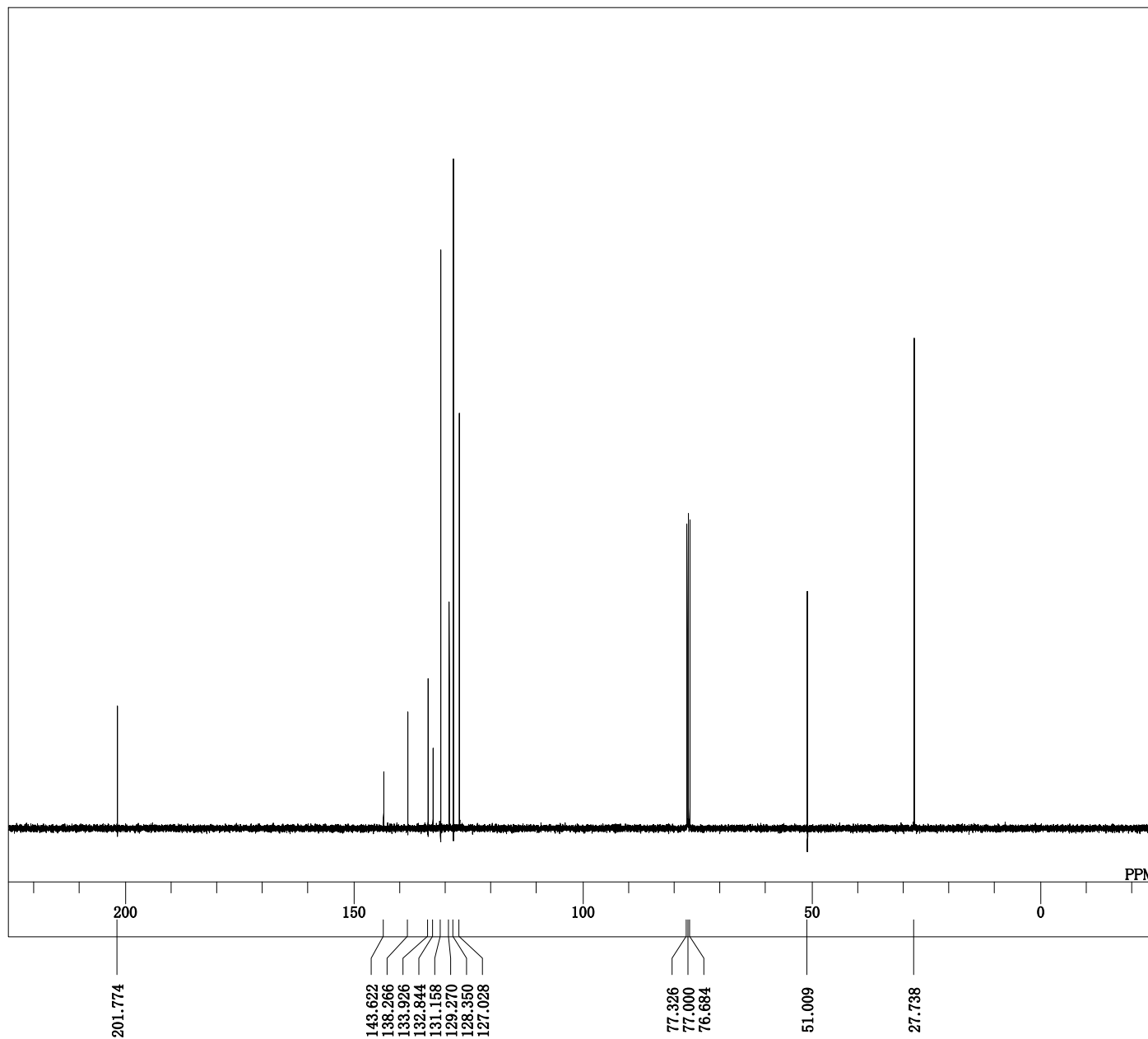
DFILE Ex655_fr1_20220411_Carbon_copy1
 COMNT single pulse decoupled gated NOE
 DATIM 2022-04-11 14:24:29
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 64
 ACQTM 0.0000 sec
 PD 2.0000 sec
 PW1 3.67 usec
 IRNUC 1H
 CTEMP 20.9 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



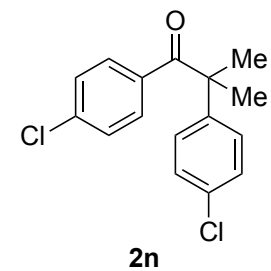


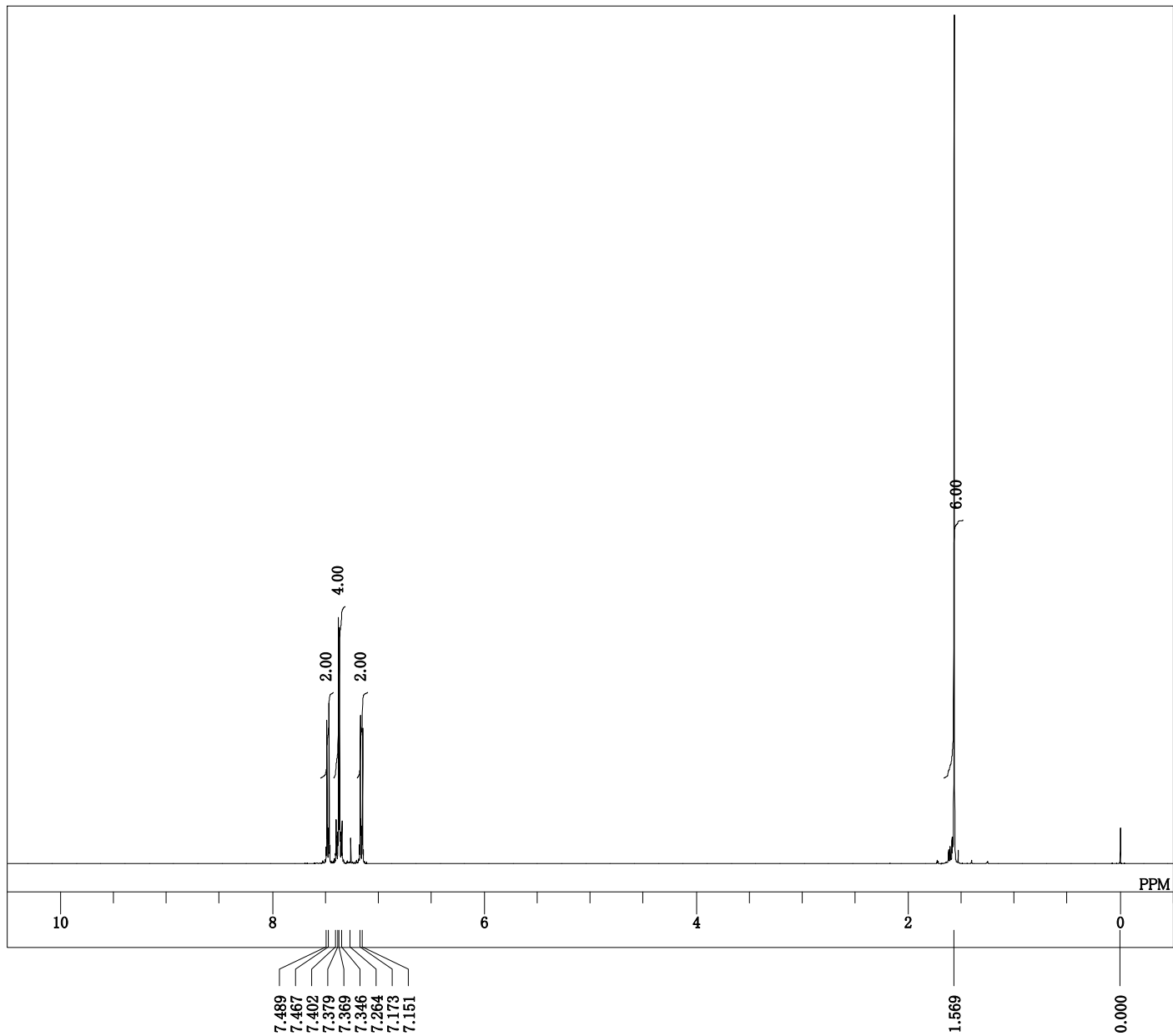
DFILE pro_cyc_pClpCl_MeMe_Proton.als
 COMNT single_pulse
 DATIM 2020-11-25 08:55:43
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 19.0 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 30



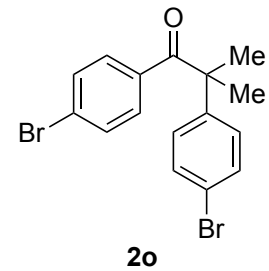


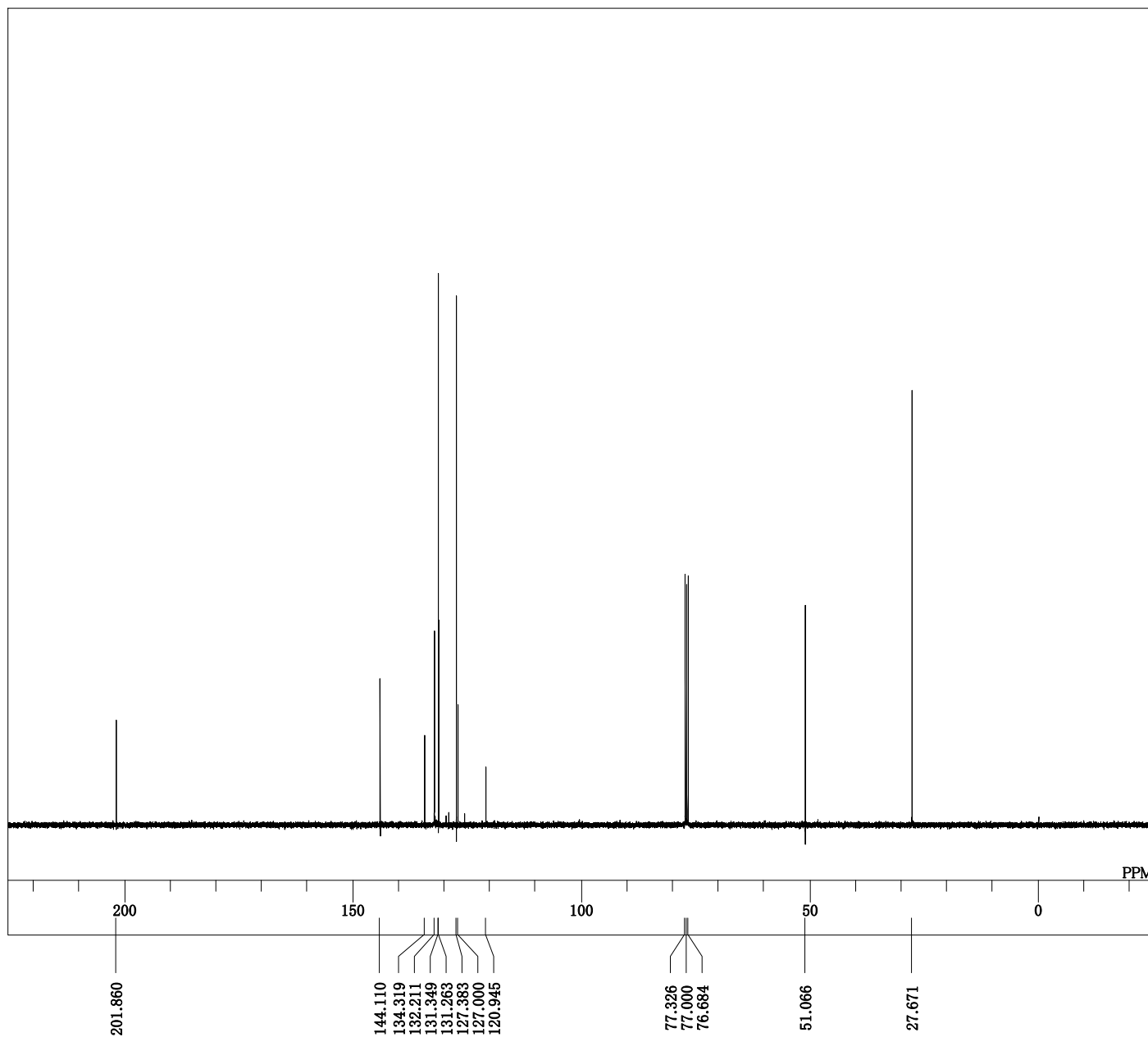
DFILE pro_cyc_pClpClMeMe_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-11-25 08:56:56
 OBNUC 13C
 EXMOD carbon.jxp
 OBFREQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 503
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 18.9 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



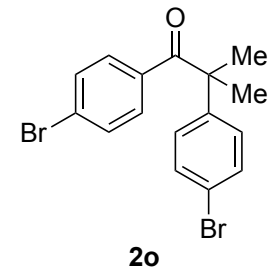


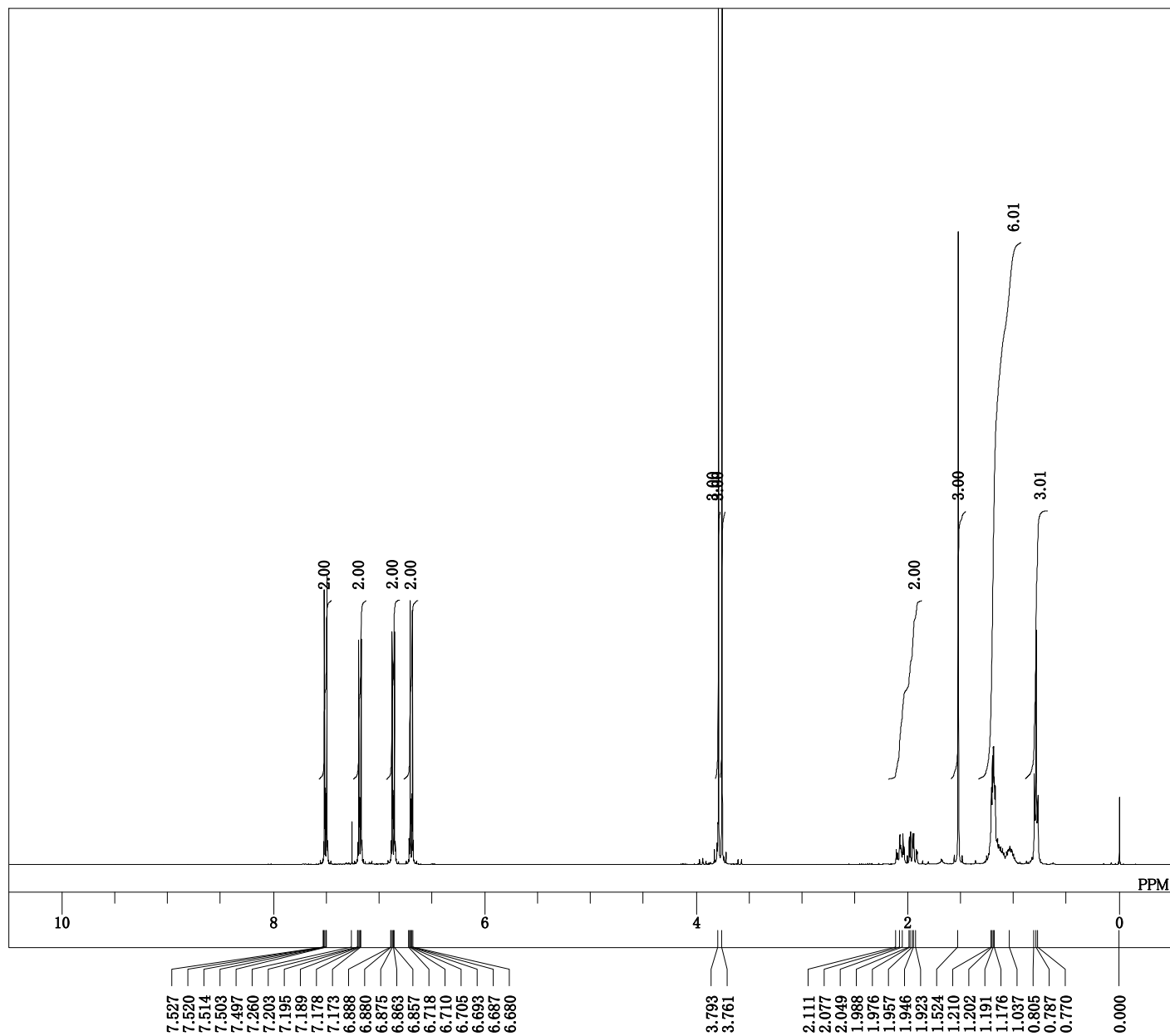
DFILE proton_xjp
 COMNT single pulse
 DATIM 2020-12-05 18:47:25
 OBNUC 1H
 EXMOD proton_xjp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 19.1 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 28



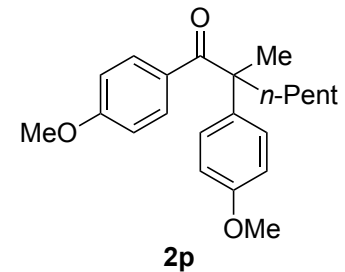


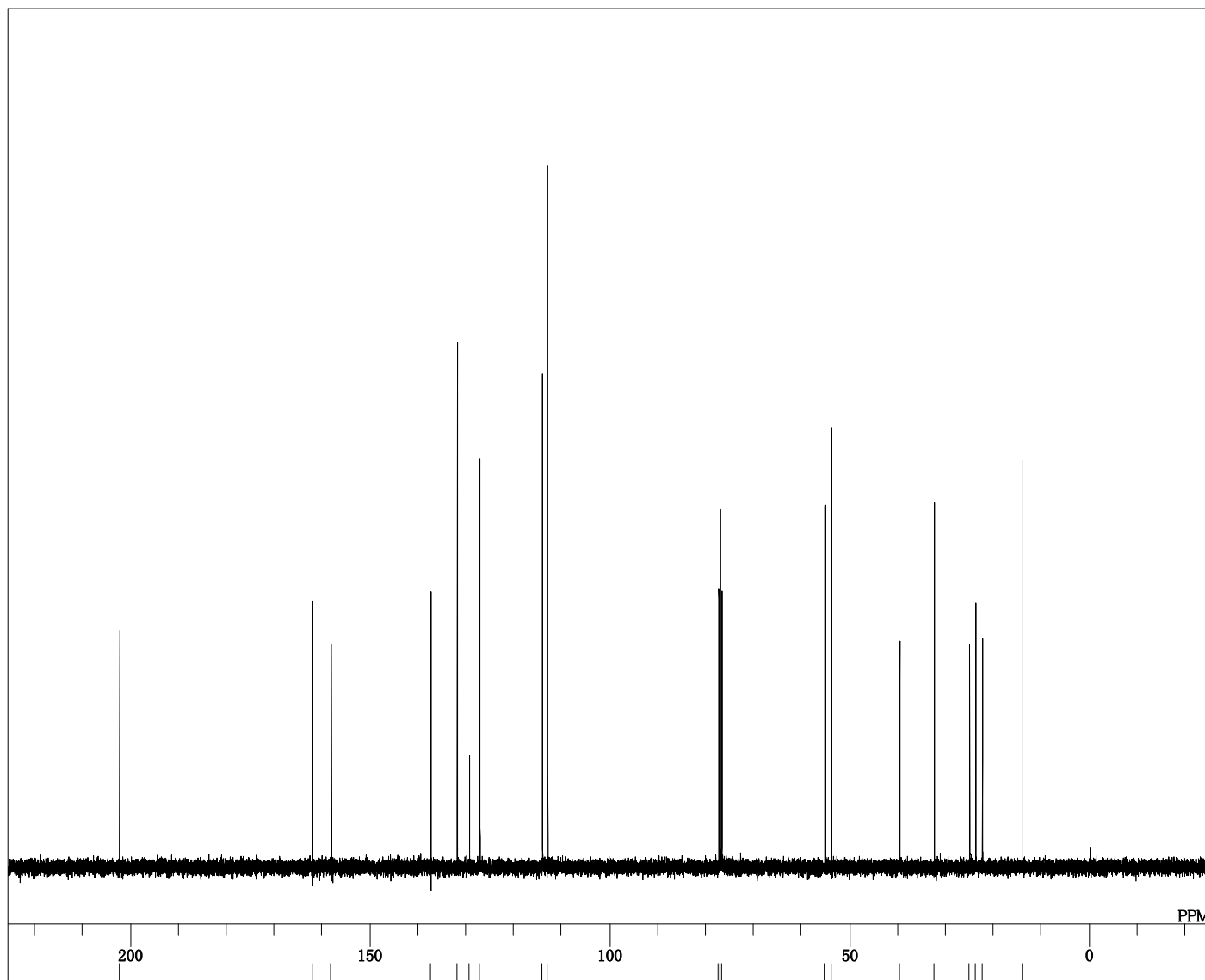
DFILE pro_cyc.pBrpBr_MeMe_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-12-05 18:48:37
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 515
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 18.9 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



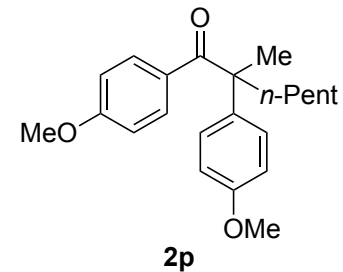


DFILE pro_cyc_PMPPMP_MenPentyl_Proto
 COMNT single_pulse
 DATIM 2020-10-13 09:42:50
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 20.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 22





DFILE pro_cyc.PMPPMP_MenPentylCarbo
 COMNT single pulse decoupled gated NOE
 DATIM 2020-10-13 09:44:03
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 26214
 FREQU 25000.00 Hz
 SCANS 152
 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



202.284

162.007
158.118

137.309
131.896
129.328
127.135

114.115
113.003

77.326
77.000
76.674

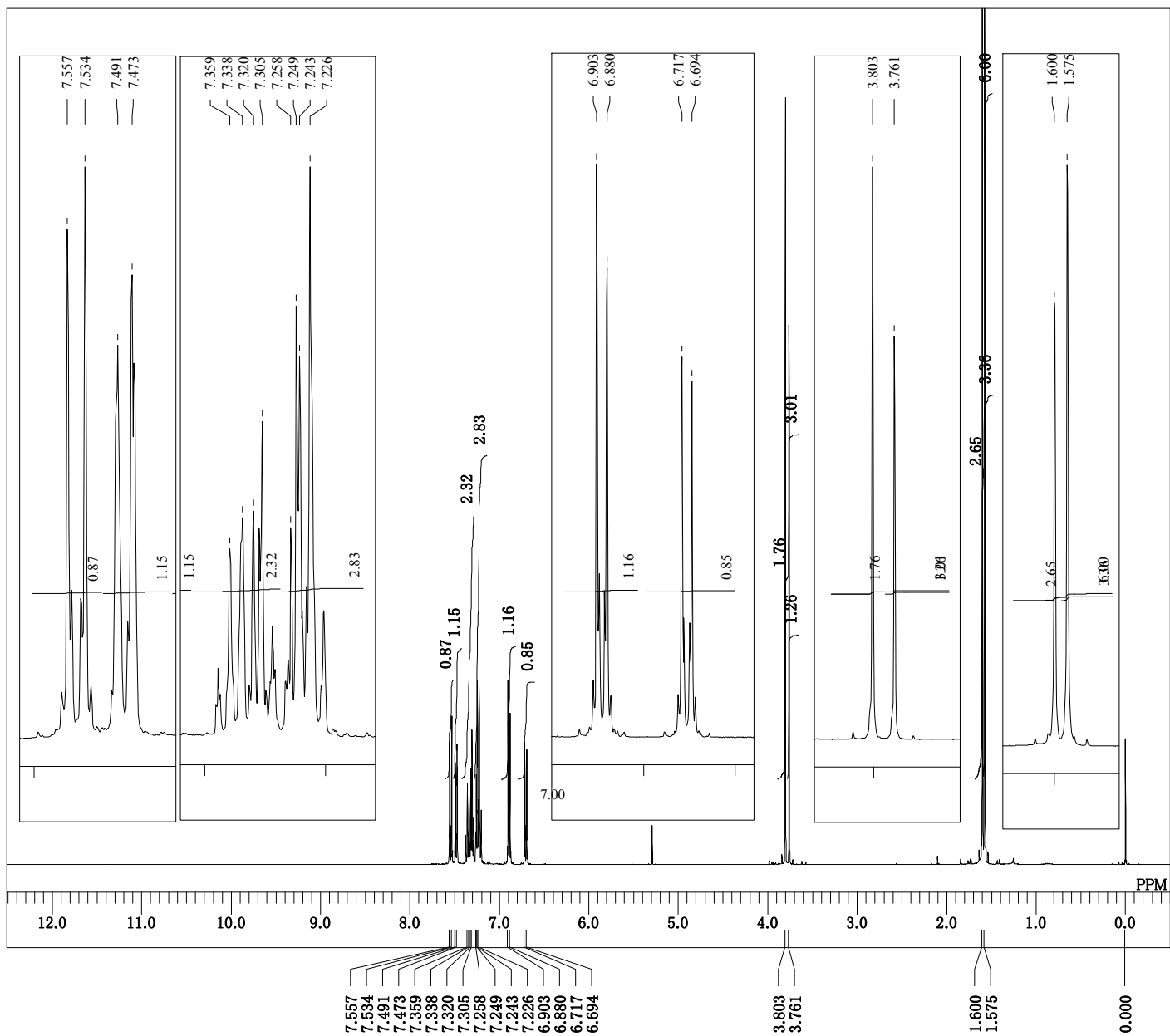
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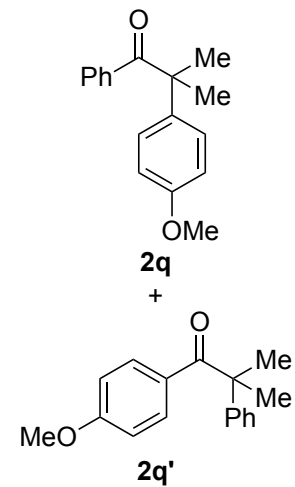
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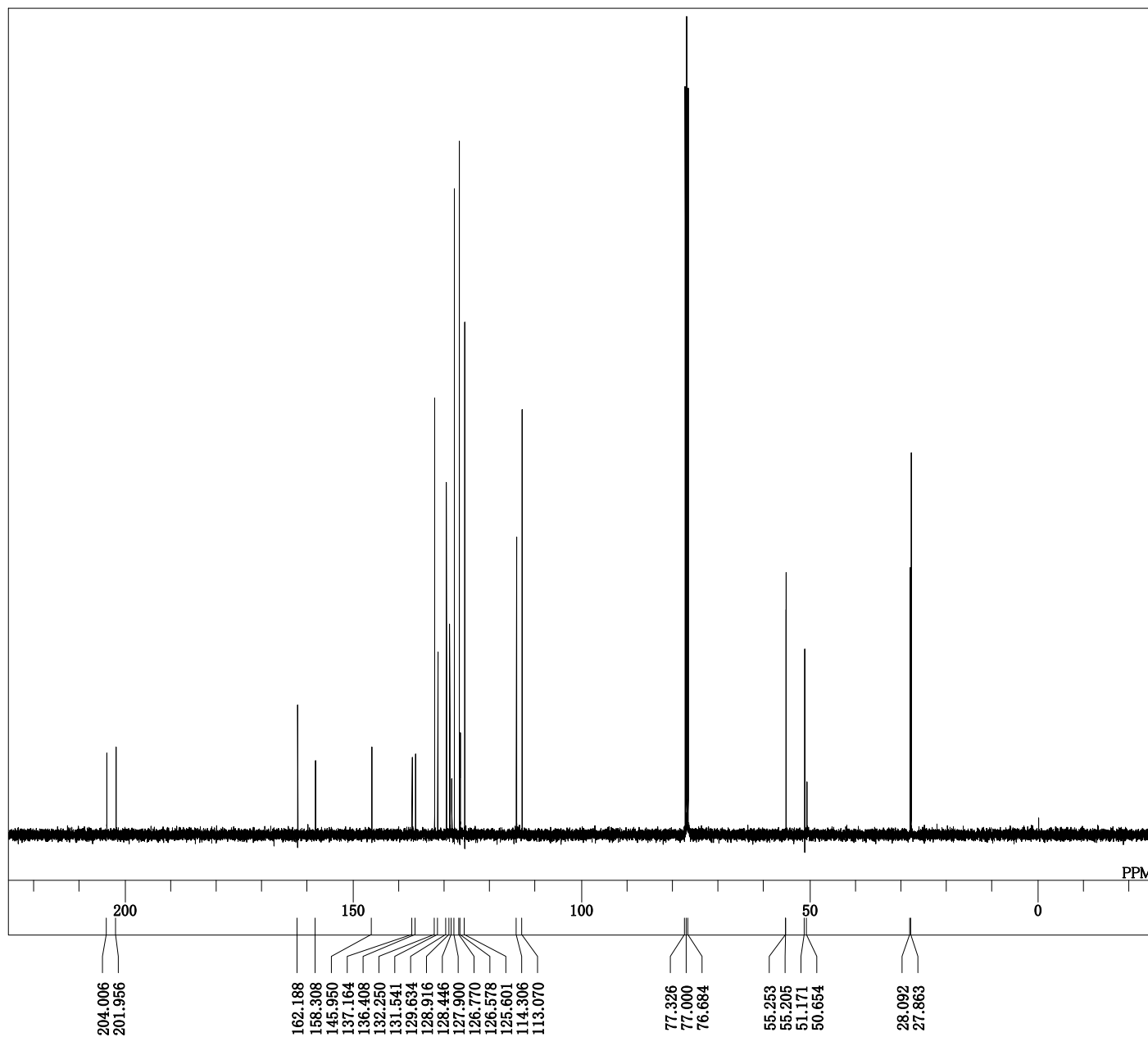
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13.941

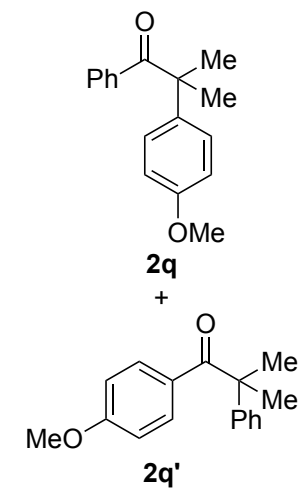


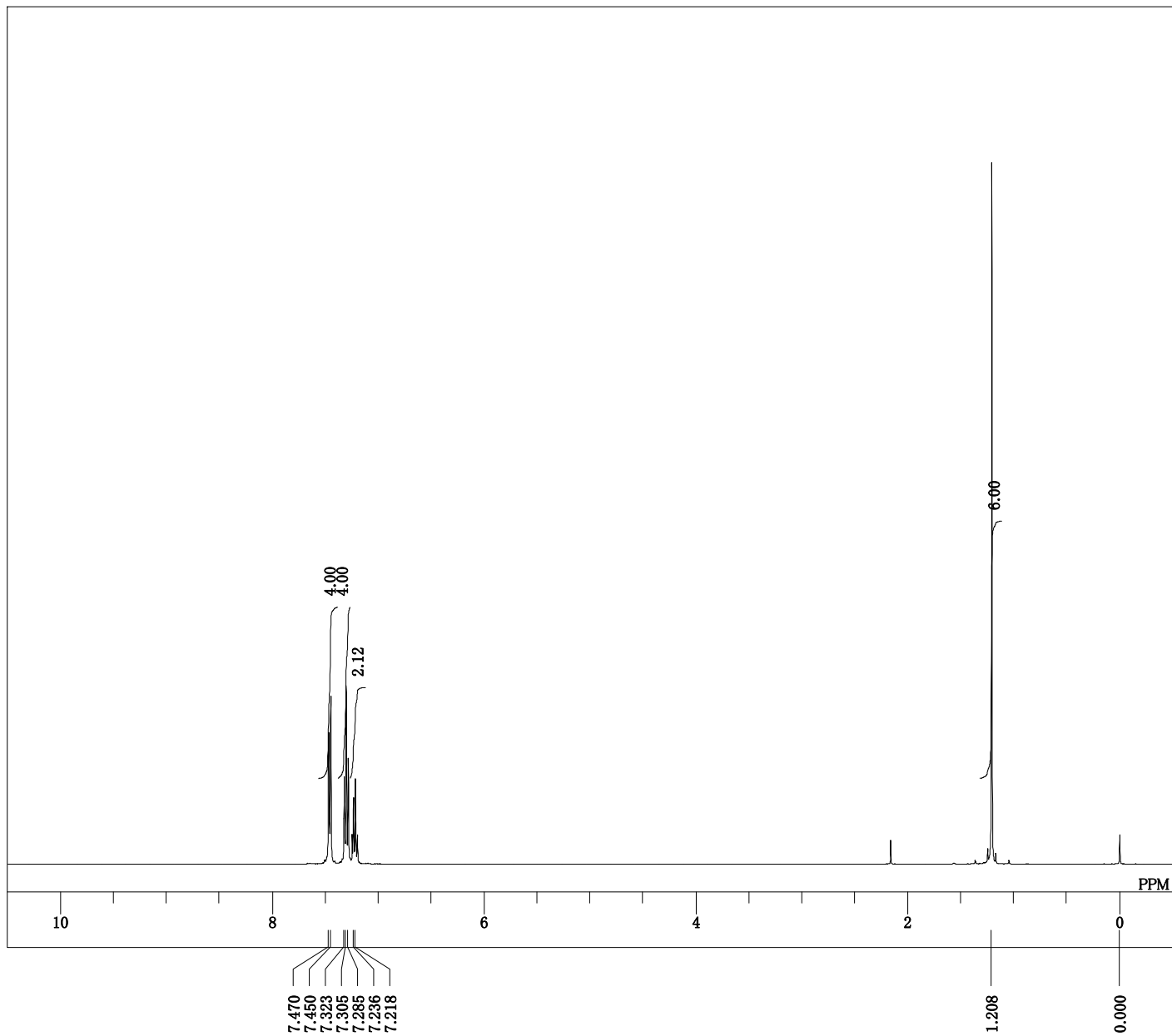
DFILE proton_cyc_PhPMP_MeMe_proton.als
 COMNT single_pulse
 DATIM 2021-01-07 17:11:54
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 18.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 30



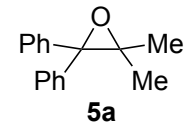


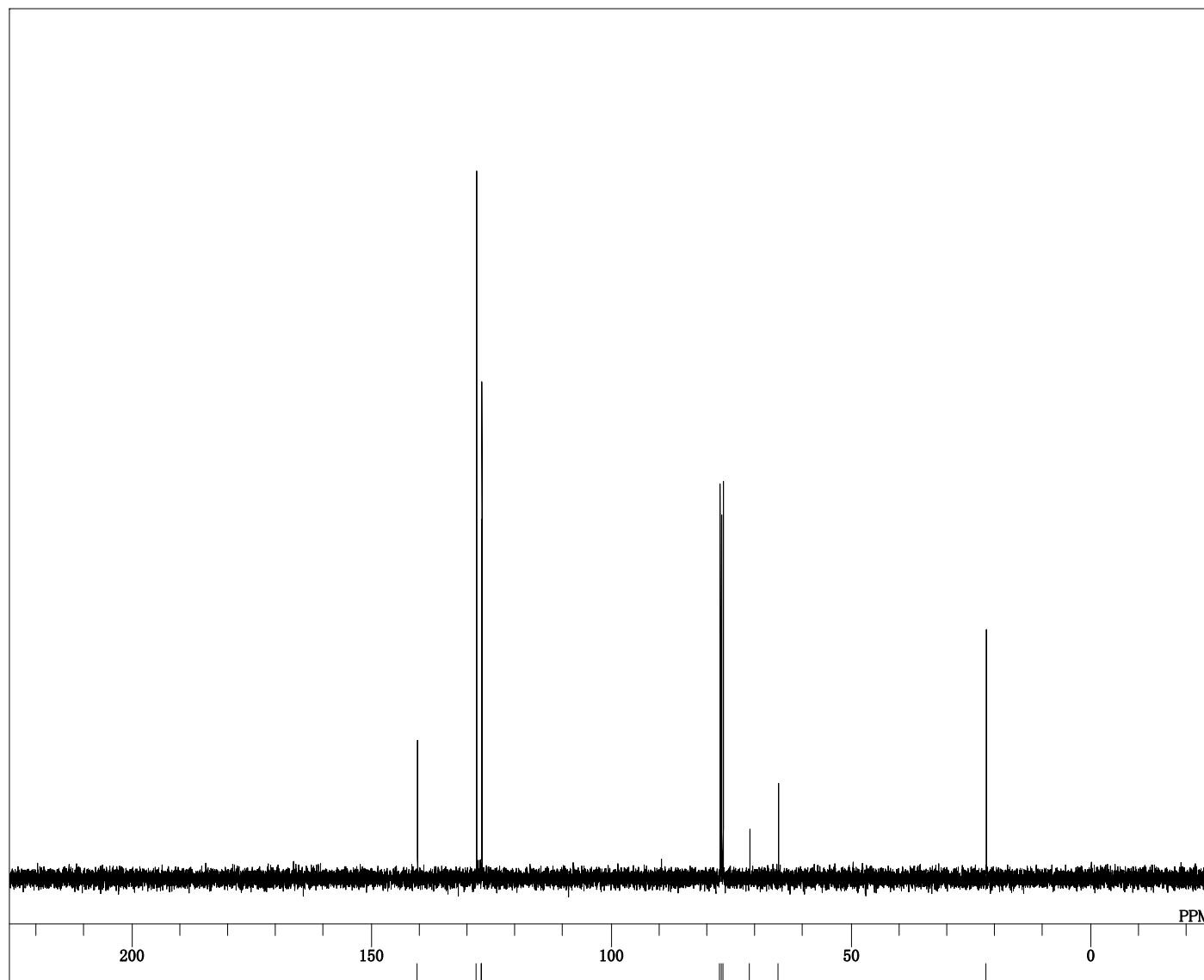
DFILE pro_cyc.PhPMP.MeMe_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2021-01-07 17:13:06
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 1089
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.59 usec
 IRNUC 1H
 CTEMP 18.6 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50



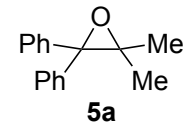


DFILE epoxy_PhPh_MeMe_Proton.als
 COMNT single_pulse
 DATIM 2020-12-21 18:57:55
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.14 usec
 IRNUC 1H
 CTEMP 18.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.72 Hz
 RGAIN 30





DFILE epoxy_PhPh_MeMe_Carbon.als
 COMNT single pulse decoupled gated NOE
 DATIM 2020-12-21 18:59:07
 OBNUC 13C
 EXMOD carbon.jxp
 OBFREQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 32
 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 0.72 Hz
 RGAIN 50



140.403
 128.111
 127.067
 126.990

77.316
 77.000
 76.674
 71.175
 65.159

21.846