GOLD(III)-CATALYZED SYNTHESIS OF 2,3,4-TRISUBSTITUTED DIHYDROPYRANS FROM PROPARGYLIC ALCOHOLS WITH 1,3-DICARBONYL COMPOUNDS

Supporting Information

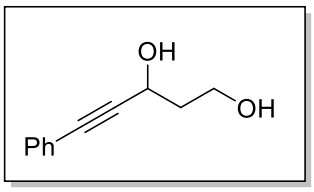
Nobuyoshi Morita*, Kazuki Oguro, Saori Takahashi, Midori, Kawahara, Shintaro Ban, Yoshimitsu Hashimoto, and Osamu Tamura*

Showa Pharmaceutical University, 3-3165 Higashi-Tamagawagakuen, Machida, Tokyo 194-8543, Japan

Contents

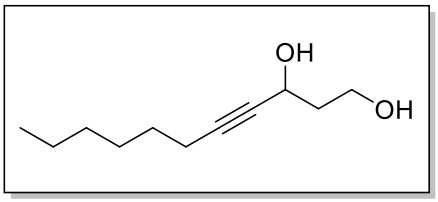
General procedure ^{1}H and ^{13}C NMR spectra were recorded with JEOL JNM-AL300 or BRUKER AV-300 spectrometer at room temperature, with tetramethylsilane as an internal standard ($CDCl_3$ solution). Chemical shifts were recorded in ppm, and coupling constants (*J*) in Hz. Infrared (IR) spectra were recorded with a Shimadzu FTIR-8200A spectrometer. Mass spectra were recorded on JEOL JMS-D300 and HX110 spectrometers. Merck silica gel 60 (1.09385) and Merck silica gel 60 F254 were used for column chromatography and thin layer chromatography (TLC), respectively.

5-Phenylpent-4-yne-1,3-diol (1a)



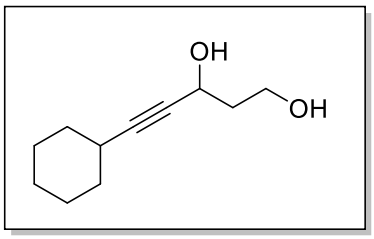
IR (KBr) 3363, 2953, 2887, 1489, 1443, 1070, 1051, 756, 692 cm^{-1};
^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.45-7.42 (2H, m), 7.33-7.29 (3H, m), 4.89 (1H, td, *J* = 5.1, 6.6 Hz), 4.10-4.04 (1H, m), 3.95-3.89 (1H, m), 2.84 (1H, d, *J* = 5.1 Hz), 2.20 (1H, br s), 2.17-2.03 (2H, m);
^{13}C-NMR (75 MHz, $CDCl_3$) δ 131.7, 128.5, 128.3, 122.4, 89.2, 85.3, 62.3, 60.6, 38.9;
HRMS (EI) *m*/*z* calcd for $C_{11}H_{12}O_2$ 176.0837, found 176.0841.

Undec-4-yne-1,3-diol (1b)



IR (KBr) 3355, 2957, 2932, 2858, 1431, 1138, 1105, 1018, 962, 887, 766 cm^{-1};
^{1}H-NMR (300 MHz, $CDCl_3$) δ 4.63 (1H, td, *J* = 4.5, 6.1 Hz), 4.02-3.94 (1H, m), 3.88-3.81 (1H, m), 2.21 (2H, dt, *J* = 1.8, 6.9 Hz), 1.98-1.87 (3H, m), 1.85 (1H, br s), 1.53-1.45 (2H, m), 1.38-1.24 (6H, m), 0.89 (1H, t, *J* = 6.9 Hz);
^{13}C-NMR (75 MHz, $CDCl_3$) δ 86.4, 80.5, 62.1, 60.6, 39.3, 31.3, 28.57, 28.54, 22.5, 18.6, 14.1;
HRMS (EI) *m*/*z* calcd for $C_{11}H_{20}O_2$ 184.1463, found 184.1453.

5-Cyclohexylpent-4-yne-1,3-diol (1c)



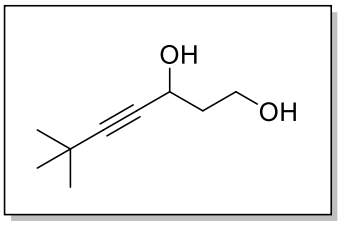
IR (KBr) 3350, 2930, 2855, 1448, 1053, 889, 575 cm^{-1};

^{1}H-NMR (300 MHz, $CDCl_3$) δ 4.64 (1H, td, *J* = 4.8, 5.1 Hz), 4.02-3.94 (1H, m), 3.89-3.82 (1H, m), 2.45-2.35 (1H, m), 2.39 (1H, br s), 2.10 (1H, br s), 2.00-1.88 (2H, m), 1.80-1.65 (4H, m), 1.51-1.29 (6H, m);

^{13}C-NMR (75 MHz, $CDCl_3$) δ 90.3, 80.4, 62.0, 60.5, 39.3, 32.6, 28.9, 25.8, 24.8;

HRMS (FAB) *m/z* calcd for $C_{11}H_{19}O_2$ $[M + H]^+$ 183.1380, found 183.1395.

6,6-Dimethylhept-4-yne-1,3-diol (1d)



IR (KBr) 3345, 2970, 2365, 1364, 1263, 1055, 750 cm^{-1};

^{1}H-NMR (300 MHz, $CDCl_3$) δ 4.61 (1H, br t, *J* = 6.0 Hz), 4.00-3.92 (1H, m), 3.89-3.80 (1H, m), 2.78 (1H, br s), 2.51 (1H, br s), 1.97-1.88 (2H, m), 1.20 (9H, s);

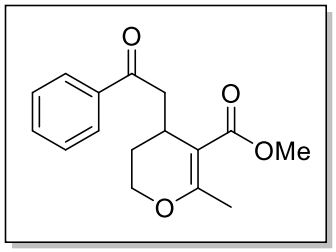
^{13}C-NMR (75 MHz, $CDCl_3$) δ 94.4, 78.9, 61.8, 60.5, 39.3, 30.9, 27.3;

HRMS (FAB) *m/z* calcd for $C_9H_{17}O_2$ $[M + H]^+$ 157.1223, found 157.1233.

Gold(III)-catalyzed synthesis of 2,3,4-trisubstituted dihydropyrans 3 from propargylic alcohols 1 with 1,3-dicarbonyl compounds 2

Gold catalyst **A** (dichloro [2-pyridinecarboxylato] gold) (5 mol%) and silver catalyst ($AgNTf_2$; 10 mol%) were added at room temperature to a solution of propargylic alcohol **1** and 1,3-dicarbonyl compound **2** in $ClCH_2CH_2Cl$. After complete consumption of propargylic alcohol **1** (the reaction was monitored by thin layer chromatography; usually within 1-3 h), the solvent was removed in vacuo and the crude product was subjected to SiO_2 column chromatography (Hexane:AcOEt = 6:1) to give the corresponding 2,3,4-trisubstituted dihydropyrans **3**.

Methyl 6-methyl-4-(2-oxo-2-phenylethyl)-3,4-dihydro-2*H*-pyran-5-carboxylate (3aa)



Propargylic alcohol **1a** (50 mg, 0.28 mmol) and methylacetoacetate (**2a**) (0.36 mL, 3.0 mmol) in $ClCH_2CH_2Cl$ (5 mL) and gold catalyst (5.5 mg, 0.014 mmol, 5 mol%) and $AgNTf_2$ (11 mg, 0.028 mmol, 10 mol%) furnished dihydropyran **3aa** (46 mg, 59%) as colorless oil.

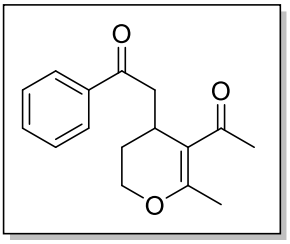
IR (KBr) 2953, 1705, 1683, 1612, 1265, 1186, 1091, 1062, 883, 754, 690 cm^{-1};

^{1}H-NMR (300 MHz, $CDCl_3$) δ 8.04-8.01 (2H, m), 7.60-7.54 (1H, m), 7.50-7.45 (2H, m), 4.19-4.13 (1H, m), 3.97 (1H, dt, *J* = 3.3, 11.7 Hz), 3.70 (3H, s), 3.38 (1H, ddd, *J* = 16.2, 2.4, 0.9 Hz), 3.36-3.31 (1H, m), 2.77 (1H, dd, *J* = 11.1, 16.2 Hz), 2.25 (3H, s), 1.94-1.74 (2H, m);

^{13}C-NMR (75 MHz, $CDCl_3$) δ 199.1, 168.6, 166.0, 136.8, 133.1, 128.6, 128.2, 104.1, 62.8, 51.0, 43.9, 26.9, 25.2, 20.8;

HRMS (EI) *m*/*z* calcd for $C_{16}H_{18}O_4$ 274.1205, found 274.1194.

2-(5-Acetyl-6-methyl-3,4-dihydro-2*H*-pyran-4-yl)-1-phenylethan-1-one (3ab)



Propargylic alcohol **1a** (50 mg, 0.28 mmol) and acetylacetone (**2b**) (0.30 mL, 2.9 mmol) in $ClCH_2CH_2Cl$ (5 mL) and gold catalyst (5.5 mg, 0.014 mmol, 5 mol%) and $AgNTf_2$ (11 mg, 0.028 mmol, 10 mol%) furnished dihydropyran **3da** (24 mg, 33%) as colorless oil.

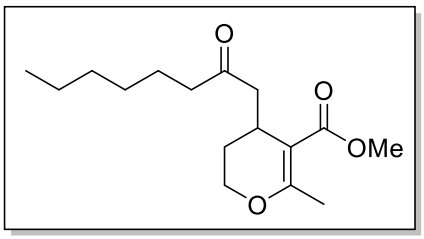
IR (KBr) 2953, 1670, 1597, 1448, 1377, 1358, 1267, 1055, 1005, 972, 880, 756, 691, 634 cm^{-1};

^{1}H-NMR (300 MHz, $CDCl_3$) δ 8.05-8.03 (2H, m), 7.64-7.55 (1H, m), 7.50-7.45 (2H, m), 4.19-4.13 (1H, m), 3.94 (1H, dt, *J* = 3.0, 12.3 Hz), 3.50-3.44 (1H, m), 3.14 (1H, ddd, *J* = 0.9, 2.4, 16.5 Hz), 2.86 (1H, dd, *J* = 11.1, 16.5 Hz), 2.29 (3H, s), 2.18 (3H, s), 1.89-1.82 (1H, m), 1.79-1.72 (1H, m);

^{13}C-NMR (75 MHz, $CDCl_3$) δ 199.3, 198.8, 164.4, 136.7, 133.2, 128.6, 128.2, 115.3, 62.7, 43.7, 30.3, 26.9, 25.2, 21.4;

HRMS (EI) *m*/*z* calcd for $C_{16}H_{18}O_3$ 258.1256, found 258.1247.

Methyl 6-methyl-4-(2-oxooctyl)-3,4-dihydro-2*H*-pyran-5-carboxylate (3ba)



Propargylic alcohol **3b** (50 mg, 0.27 mmol) and methylacetoacetate (**2a**) (0.31 mL, 2.7 mmol) in $ClCH_2CH_2Cl$ (5 mL) and gold catalyst (5.5 mg, 0.014 mmol, 5 mol%) and $AgNTf_2$ (11 mg, 0.028 mmol, 10 mol%) furnished dihydropyran **3ba** (13 mg, 17%) as colorless oil.

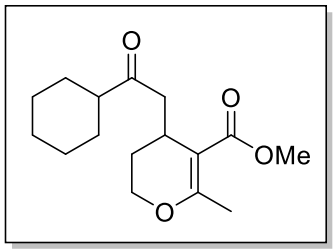
IR (KBr) 2953, 1717, 1653, 1558, 1541, 1508, 1265, 1091, 534 cm^{-1};

^{1}H-NMR (300 MHz, $CDCl_3$) δ 4.17-4.11 (1H, m), 3.87 (1H, dt, *J* = 2.7,11.1 Hz), 3.69 (3H, s), 3.18-3.13 (1H, m), 2.7 (1H, ddd, *J* = 16.2, 3.0, 1.2 Hz), 2.49-2.36 (2H, m), 2.33-2.24 (1H, m), 2.21 (3H, s), 1.92-1.79 (1H, m), 1.68 (1H, dq, *J* = 14.4, 2.1 Hz), 1.31-1.25 (8H, m), 0.88 (3H, t, *J* = 6.9 Hz);

^{13}C-NMR (75 MHz, $CDCl_3$) δ 210.2, 168.6, 165.7, 104.1, 62.7, 51.0, 47.9, 42.9, 31.6, 28.9, 26.1, 25.6, 23.8, 22.5, 20.7, 14.0;

HRMS (EI) *m*/*z* calcd for $C_{16}H_{26}O_4$ 282.1831, found 282.1835.

Methyl 4-(2-cyclohexyl-2-oxoethyl)-6-methyl-3,4-dihydro-2*H*-pyran-5-carboxylate (3ca)



Propargylic alcohol **1c** (50 mg, 0.27 mmol) and methylacetoacetate (**2a**) (0.31 mL, 2.8 mmol) in $ClCH_2CH_2Cl$ (5 mL) and gold catalyst (5.5 mg, 0.014 mmol, 5 mol%) and $AgNTf_2$ (11 mg, 0.028 mmol, 10 mol%) furnished dihydropyran **3ca** (13 mg, 17%) as colorless oil.

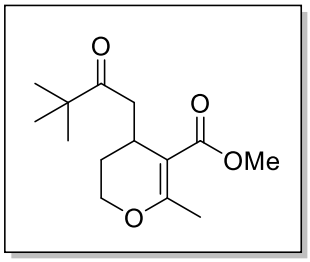
IR (KBr) 2929, 2854, 1705, 1612, 1377, 1265, 1188, 1092, 775 cm^{-1};

^{1}H-NMR (300 MHz, $CDCl_3$) δ 4.13 (1H, m), 3.86 (1H, dt, *J* = 2.4, 10.8 Hz), 3.68 (3H, s), 3.17-3.14 (1H, m), 2.73 (1H, br d, *J* = 16.5 Hz), 2.31 (2H, dd, *J* = 10.5, 16.5 Hz), 2.20 (3H, s), 1.89-1.79 (5H, m), 1.67-1.62 (2H, m), 1.41-1.25 (5H, m);

^{13}C-NMR (75 MHz, $CDCl_3$) δ 212.9, 168.6, 165.6, 104.2, 62.8, 51.0, 50.8, 45.8, 28.5, 28.2, 25.9, 25.8, 25.7, 25.7, 25.5, 20.6;

HRMS (EI) *m*/*z* calcd for $C_{16}H_{24}O_4$ 280.1675, found 280.1671.

Methyl 4-(3,3-dimethyl-2-oxobutyl)-6-methyl-3,4-dihydro-2*H*-pyran-5-carboxylate (3da)



Propargylic alcohol **1d** (50 mg, 0.32 mmol) and methylacetoacetate (**2a**) (0.34 mL, 3.2 mmol) in $ClCH_2CH_2Cl$ (5 mL) and gold catalyst (5.5 mg, 0.014 mmol, 5 mol%) and $AgNTf_2$ (11 mg, 0.028 mmol, 10 mol%) furnished dihydropyran **3da** (16 mg, 19%) as colorless oil.

IR (KBr) 2953, 1705, 1610, 1435, 1265, 1094, 914, 743 cm^{-1};

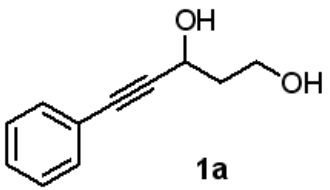
^{1}H-NMR (300 MHz, $CDCl_3$) δ 4.17-4.11 (1H, m), 3.85 (1H, dt, *J* = 2.4, 11.1 Hz), 3.67 (3H, s), 3.21-3.15 (1H, m), 2.73 (1H, ddd, *J* = 0.9, 2.1, 17.4 Hz), 2.42 (1H, dd, *J* = 10.8, 17.4 Hz), 2.22 (3H, s), 1.92-1.80 (1H, m), 1.68-1.65 (1H, m), 1.12 (9H, s);

^{13}C-NMR (75 MHz, $CDCl_3$) δ 214.5, 168.6, 165.6, 104.3, 62.8, 51.0, 44.1, 41.6, 26.2, 25.8, 25.7, 20.6;

HRMS (EI) *m*/*z* calcd for $C_{14}H_{22}O_4$ 254.1518, found 254.1523.

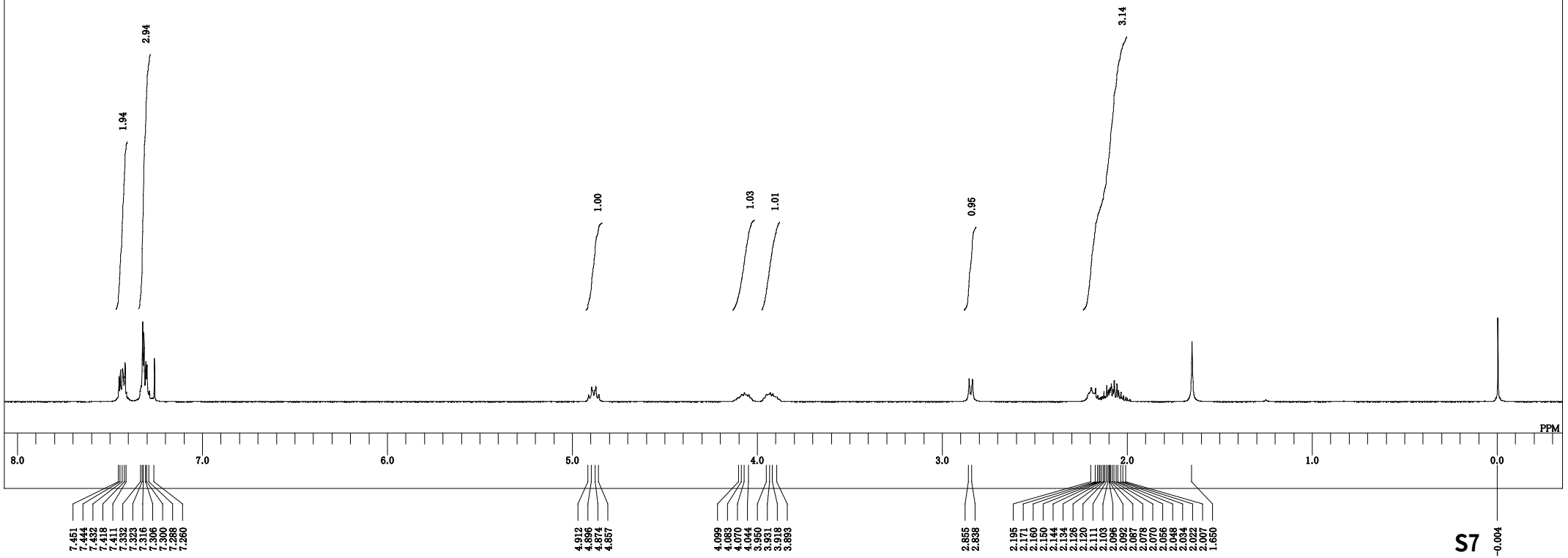
20160518TM_propal

¥¥192.168.226.211¥anal¥6年生¥村井¥2015年3月～¥原料合成¥20160518TM_propal1NON_E63.als



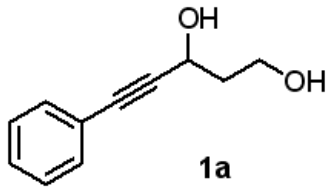
1a

DFILE	20160518TM_propal1NON_E63.als
COMNT	20160518TM_propal
DATIM	Wed May 18 11:43:18 2016
OBNUC	1H
EXMOD	NON
OBFRQ	300.40 MHz
OBSET	130.00 KHz
OBFIN	1150.00 Hz
POINT	16384
FREQU	6006.01 Hz
SCANS	8
ACQTM	2.7279 sec
PD	4.2720 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	20.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	22



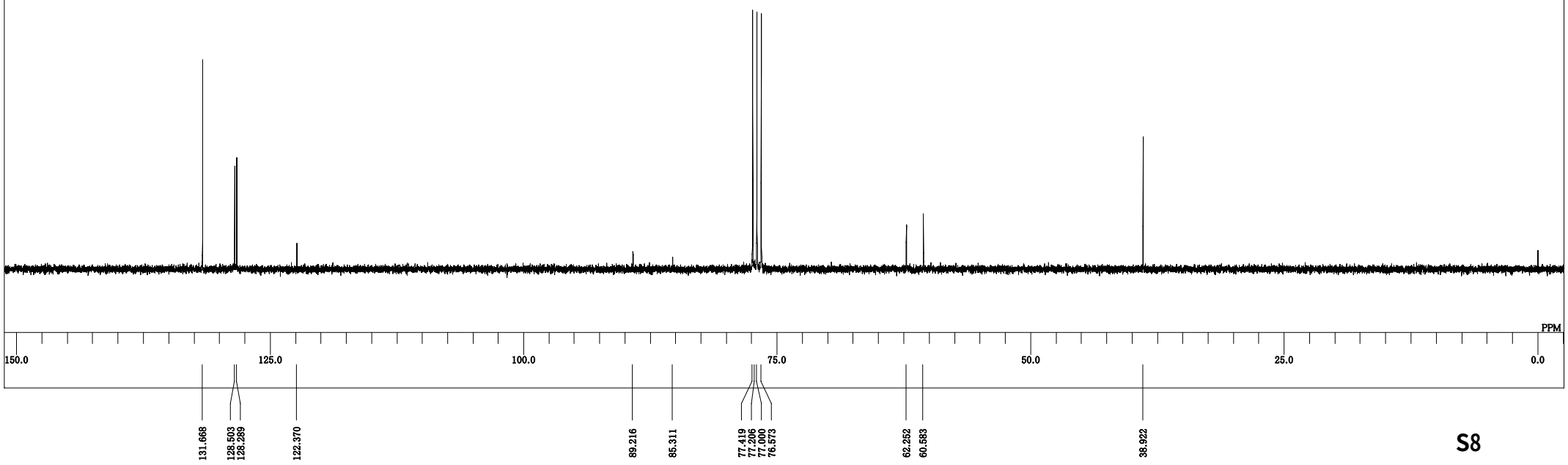
nHex_c

¥¥192.168.226.211¥anal¥6年生¥河原¥プロパルギルアルコールNMR¥Ph¥nHex_c1BCM_E64.als



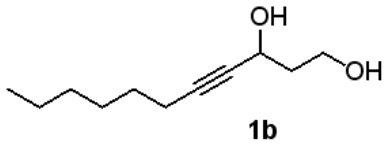
1a

DFILE	nHex_c1BCM_E64.als
COMNT	nHex_c
DATIM	Sat May 07 23:04:13 2016
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.00 Hz
POINT	32768
FREQU	20356.23 Hz
SCANS	1680
ACQTM	1.6097 sec
PD	1.3900 sec
PW1	5.10 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	26



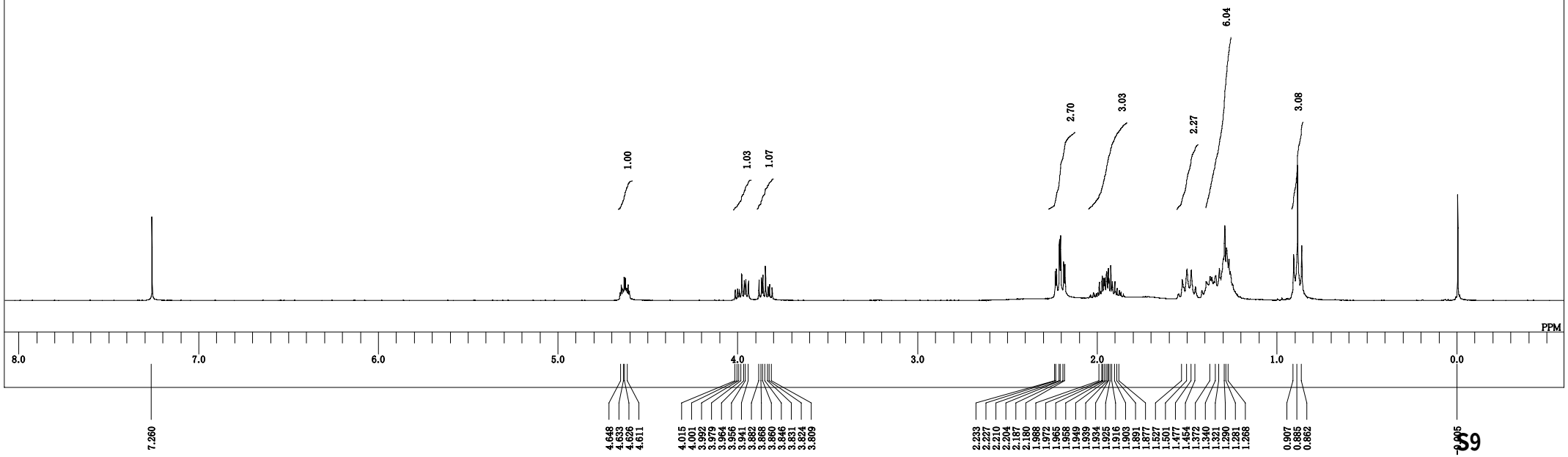
nHex

¥¥192.168.226.211¥anal¥6年生¥河原¥プロパルギルアルコールNMR¥nHex¥nHex1NON_E30.als



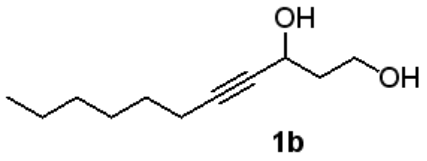
1b

```
DFILE     nHex1NON_E30.als
COMNT     nHex
DATIM     Tue Aug 04 14:39:50 2015
OBNUC     1H
EXMOD     NON
OBFRQ       300.40 MHz
OBSET       130.00 KHz
OBFIN      1150.00 Hz
POINT       16384
FREQU      6006.01 Hz
SCANS          8
ACQTM       2.7279 sec
PD          4.2720 sec
PW1          6.10 usec
IRNUC     1H
CTEMP         23.0 c
SLVNT     CDCL3
EXREF         0.00 ppm
BF            0.12 Hz
RGAIN           17
```



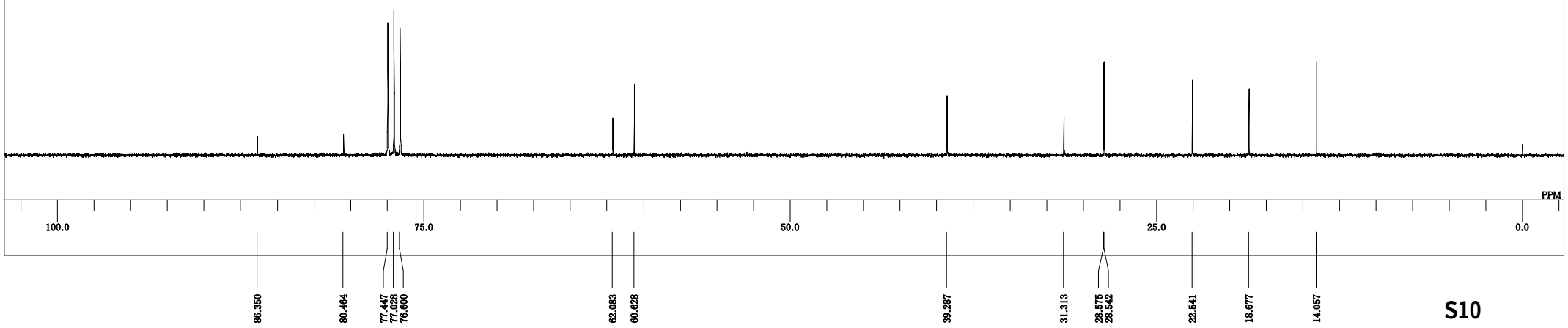
Ph_c

¥¥192.168.226.211¥anal¥6年生¥河原¥プロパルギルアルコールNMR¥nHex¥Ph_c1BCM_E68.als



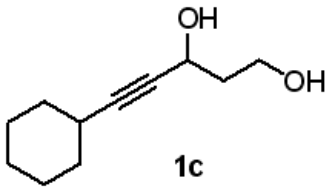
1b

DFILE	Ph_c1BCM_E68.als
COMNT	Ph_c
DATIM	Sun May 08 04:49:06 2016
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.00 Hz
POINT	32768
FREQU	20356.23 Hz
SCANS	3240
ACQTM	1.6097 sec
PD	1.3900 sec
PW1	5.10 usec
IRNUC	1H
CTEMP	20.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	25



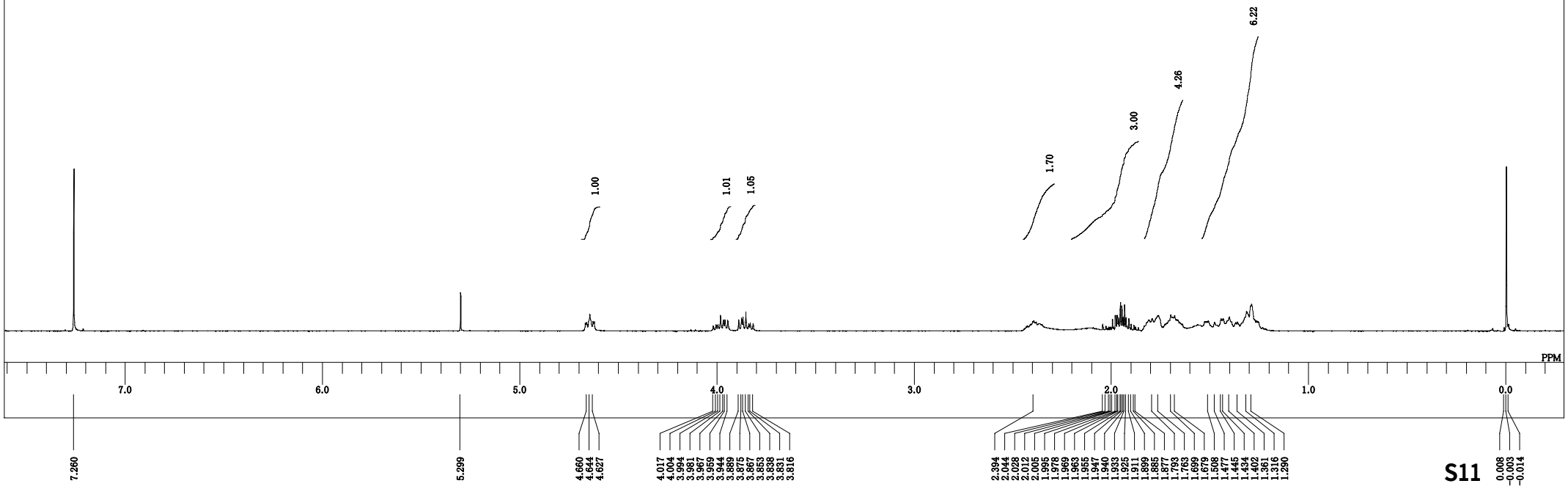
140527-04

¥¥192.168.226.211¥anal¥2015 3月卒業¥江口¥140527-041NON_E4.als



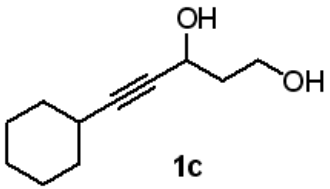
1c

DFILE	140527-041NON_E4.als
COMNT	140527-04
DATIM	Tue May 27 13:22:04 2014
OBNUC	1H
EXMOD	NON
OBFRQ	300.40 MHz
OBSET	130.00 KHz
OBFIN	1150.00 Hz
POINT	16384
FREQU	6006.01 Hz
SCANS	8
ACQTM	2.7279 sec
PD	5.6360 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	21



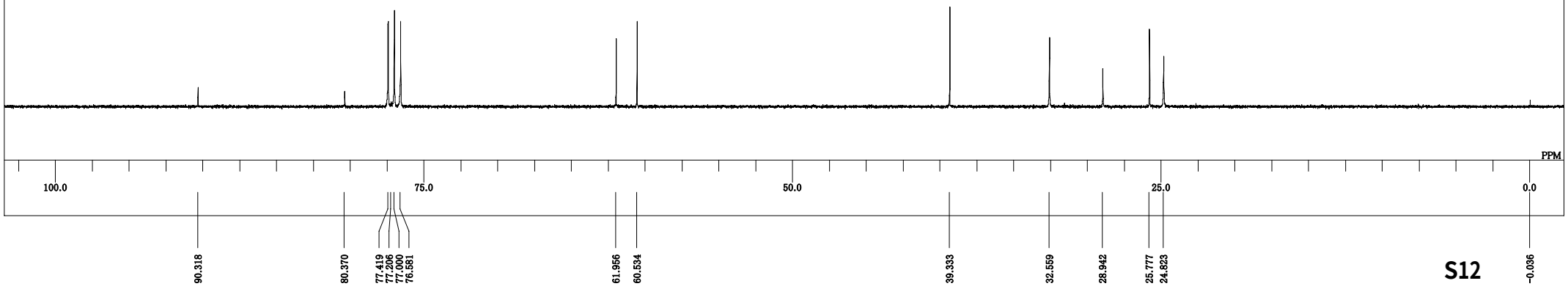
cHex_c

¥¥192.168.226.211¥anal¥6年生¥河原¥プロパルギルアルコールNMR¥cHex¥cHex_c1BCM_E66.als



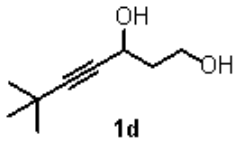
1c

DFILE	cHex_c1BCM_E66.als
COMNT	cHex_c
DATIM	Sun May 08 01:56:09 2016
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.00 Hz
POINT	32768
FREQU	20356.23 Hz
SCANS	3240
ACQTM	1.6097 sec
PD	1.3900 sec
PW1	5.10 usec
IRNUC	1H
CTEMP	20.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	26



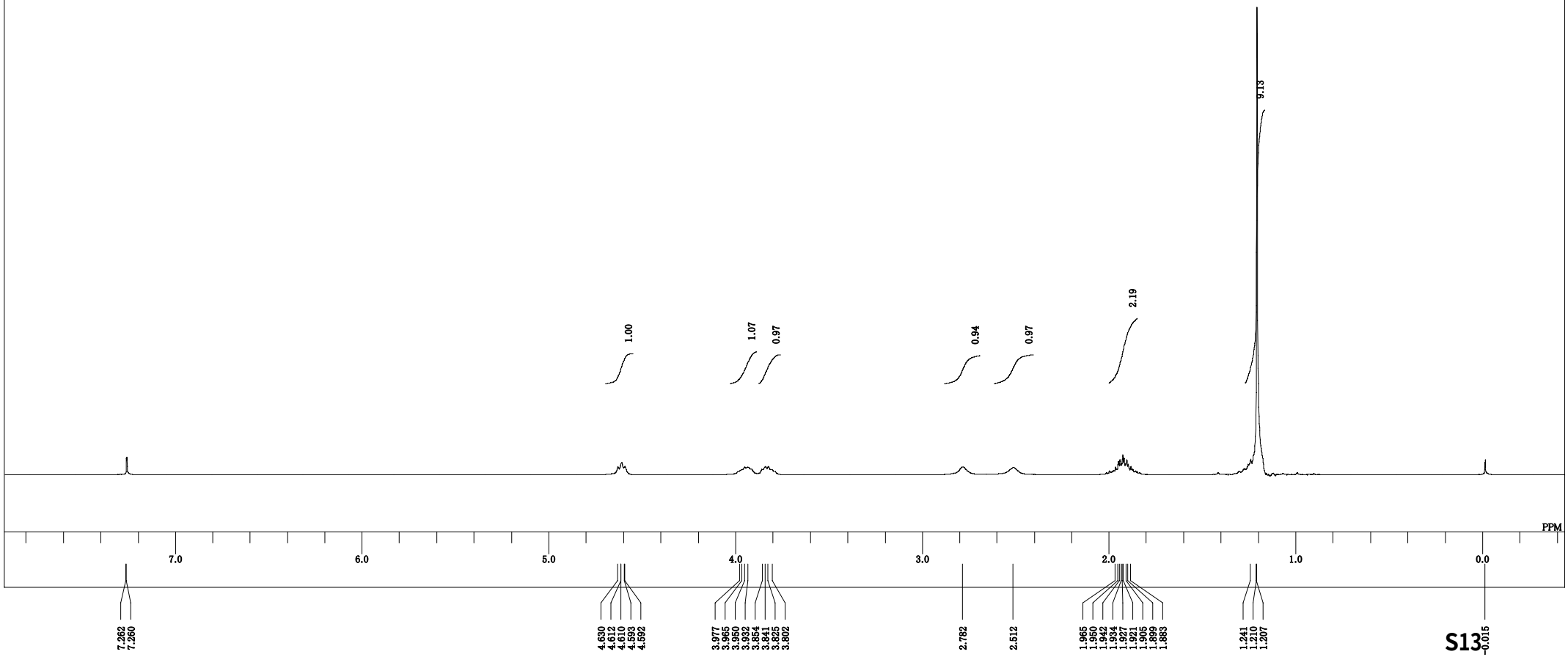
tBu_H2

D:¥data¥6年生¥河原¥原料合成¥tBu_H21NON_E32.als



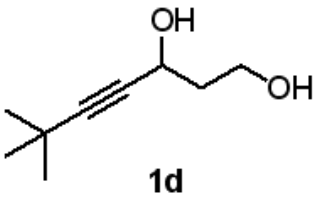
1d

DFILE	tBu_H21NON_E32.als
COMNT	tBu_H2
DATIM	Wed May 11 15:33:43 2016
OBNUC	1H
EXMOD	NON
OBFRQ	300.40 MHz
OBSET	130.00 KHz
OBFIN	1150.00 Hz
POINT	16384
FREQU	6006.01 Hz
SCANS	8
ACQTM	2.7279 sec
PD	4.2720 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	19.9 c
SLVNT	CDCL3
EXREF	-0.01 ppm
BF	0.12 Hz
RGAIN	16



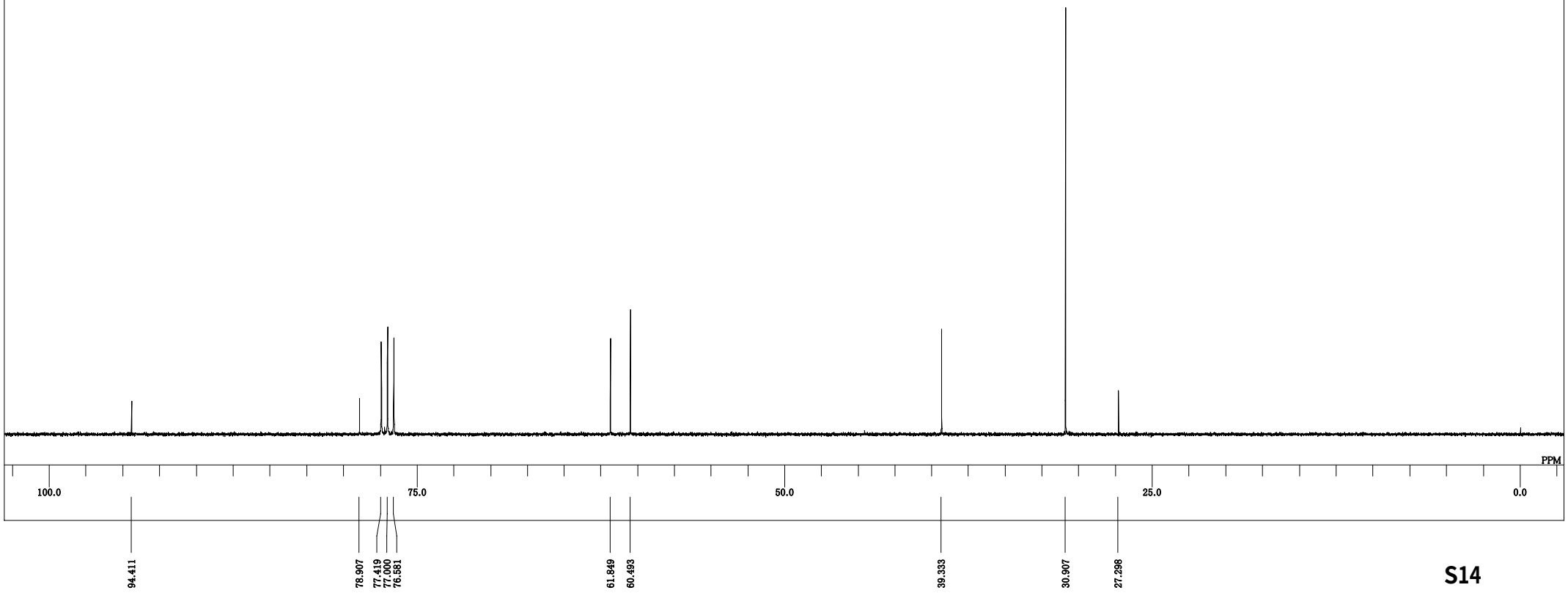
tBu

¥¥192.168.226.211¥anal¥6年生¥河原¥プロパルギルアルコールNMR¥tBu¥tBu1BCM_E3.als



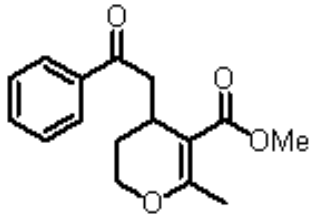
1d

DFILE	tBu1BCM_E3.als
COMNT	tBu
DATIM	Mon May 02 12:51:05 2016
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.00 Hz
POINT	32768
FREQU	20356.23 Hz
SCANS	1680
ACQTM	1.6097 sec
PD	1.3900 sec
PW1	5.10 usec
IRNUC	1H
CTEMP	20.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	26



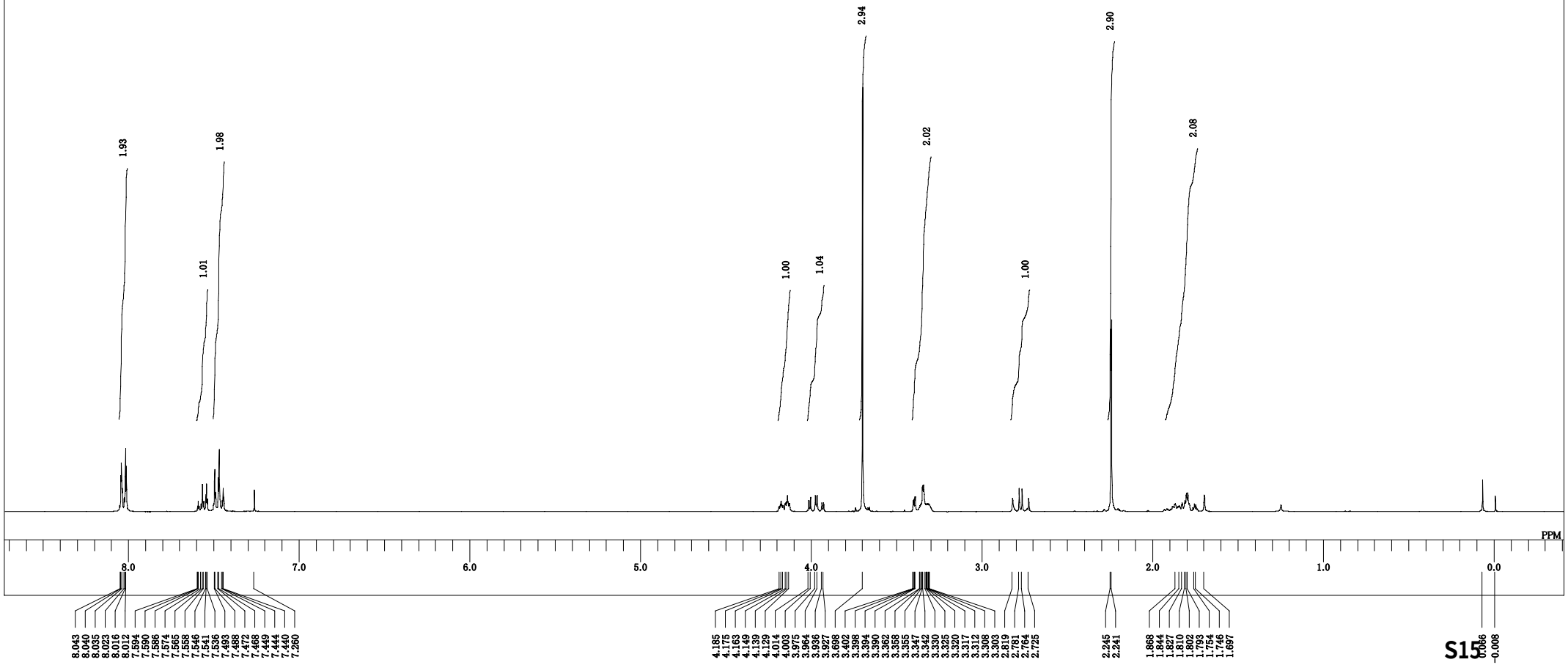
20140927 AuCl_1

D:¥data¥卒業生 2016年3月卒業¥小黒¥20140927 AuCl_11NON_E17.als



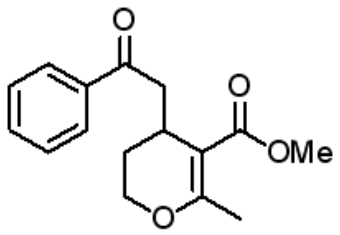
3aa

DFILE	20140927 AuCl_11NON_E17.als
COMNT	20140927 AuCl_1
DATIM	Sat Sep 27 14:11:18 2014
OBNUC	1H
EXMOD	NON
OBFRQ	300.40 MHz
OBSET	130.00 KHz
OBFIN	1150.00 Hz
POINT	16384
FREQU	6006.01 Hz
SCANS	8
ACQTM	2.7279 sec
PD	5.6360 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	22.8 c
SLVNT	CDCL3
EXREF	0.07 ppm
BF	0.12 Hz
RGAIN	15



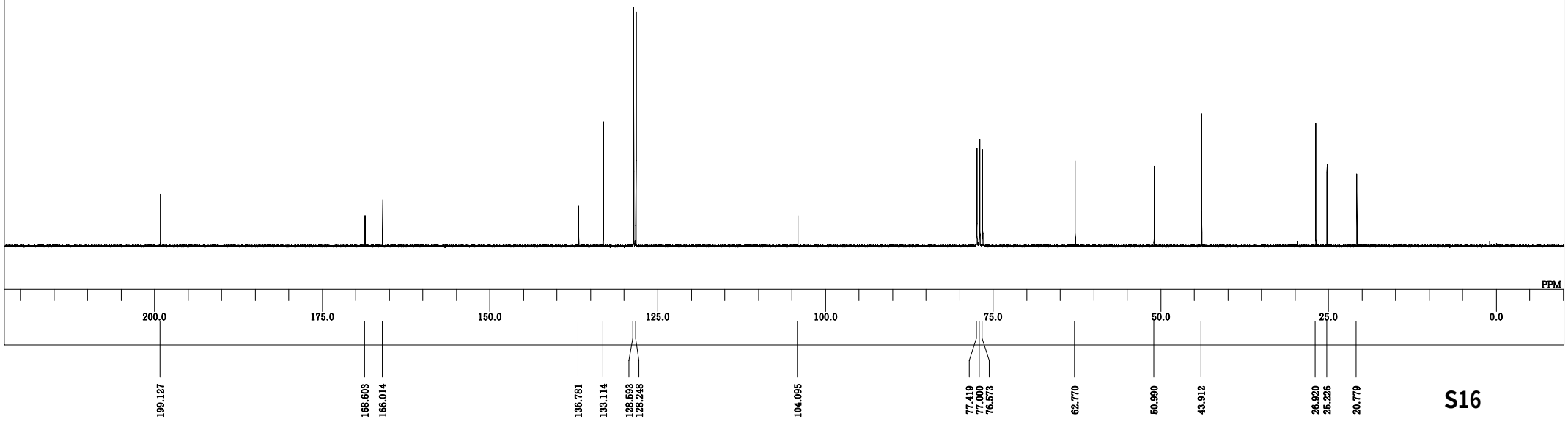
20140930 AuCl_1

¥¥192.168.226.211¥anal¥6年生¥小黒¥20140930 AuCl_12BCM_E7.als



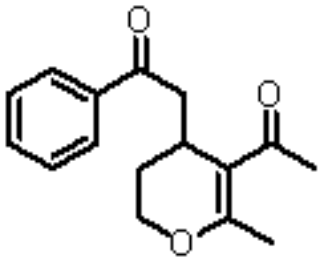
3aa

DFILE	20140930 AuCl_12BCM_E7.als
COMNT	20140930 AuCl_1
DATIM	Wed Oct 01 02:52:20 2014
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.00 Hz
POINT	32768
FREQU	20356.23 Hz
SCANS	7000
ACQTM	1.6097 sec
PD	1.3900 sec
PW1	4.00 usec
IRNUC	1H
CTEMP	23.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	26



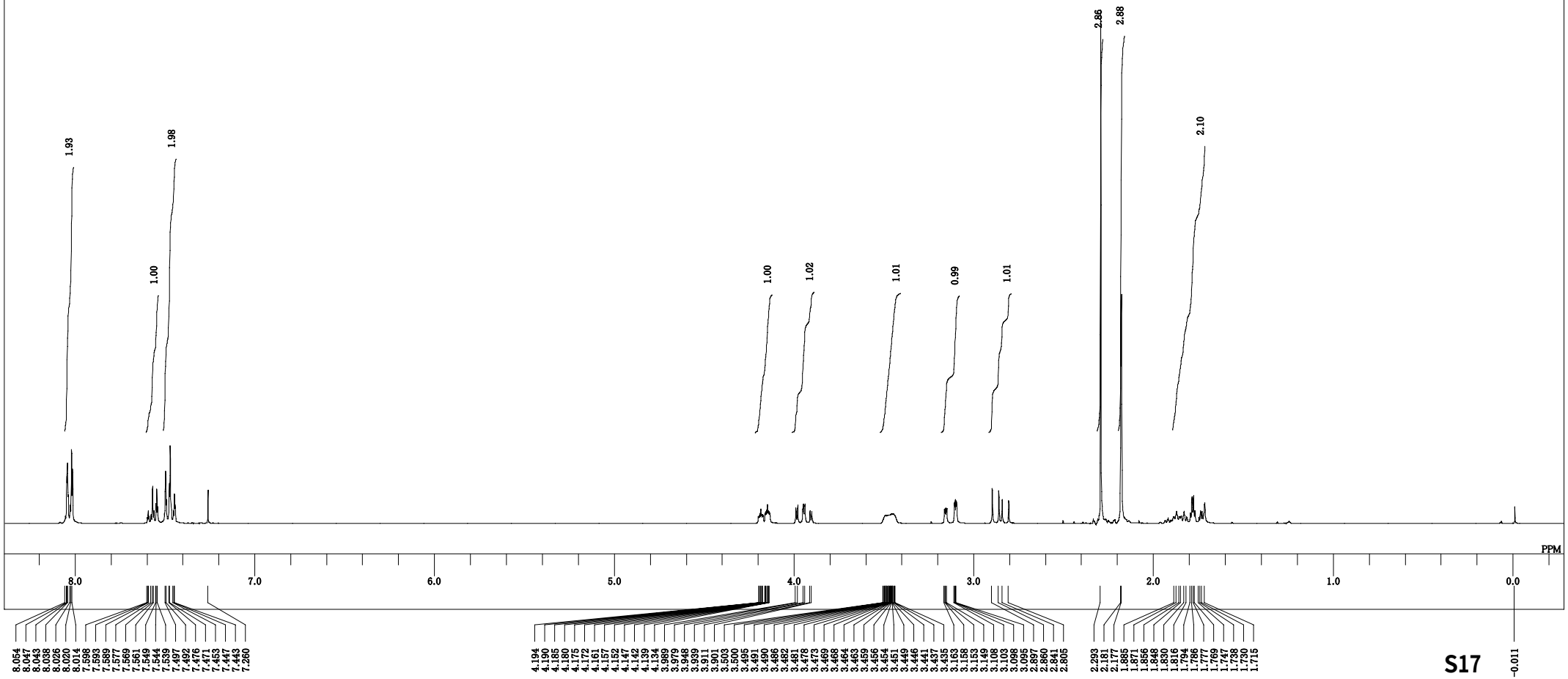
20141210_AA_1

¥¥192.168.226.211¥anal¥2016年 3月卒業¥小黒¥20141210_AA_11NON_E1.als



3ab

DFILE	20141210_AA_11NON_E1.als
COMNT	20141210_AA_1
DATIM	Wed Dec 10 17:30:41 2014
OBNUC	1H
EXMOD	NON
OBFRQ	300.40 MHz
OBSET	130.00 KHz
OBFIN	1150.00 Hz
POINT	16384
FREQU	6006.01 Hz
SCANS	8
ACQTM	2.7279 sec
PD	5.6360 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	-0.01 ppm
BF	0.12 Hz
RGAIN	16



8.054 8.047 8.043 8.038 8.026 8.020 8.014 7.598 7.593 7.589 7.577 7.569 7.561 7.549 7.544 7.539 7.497 7.492 7.476 7.471 7.453 7.447 7.443 7.260

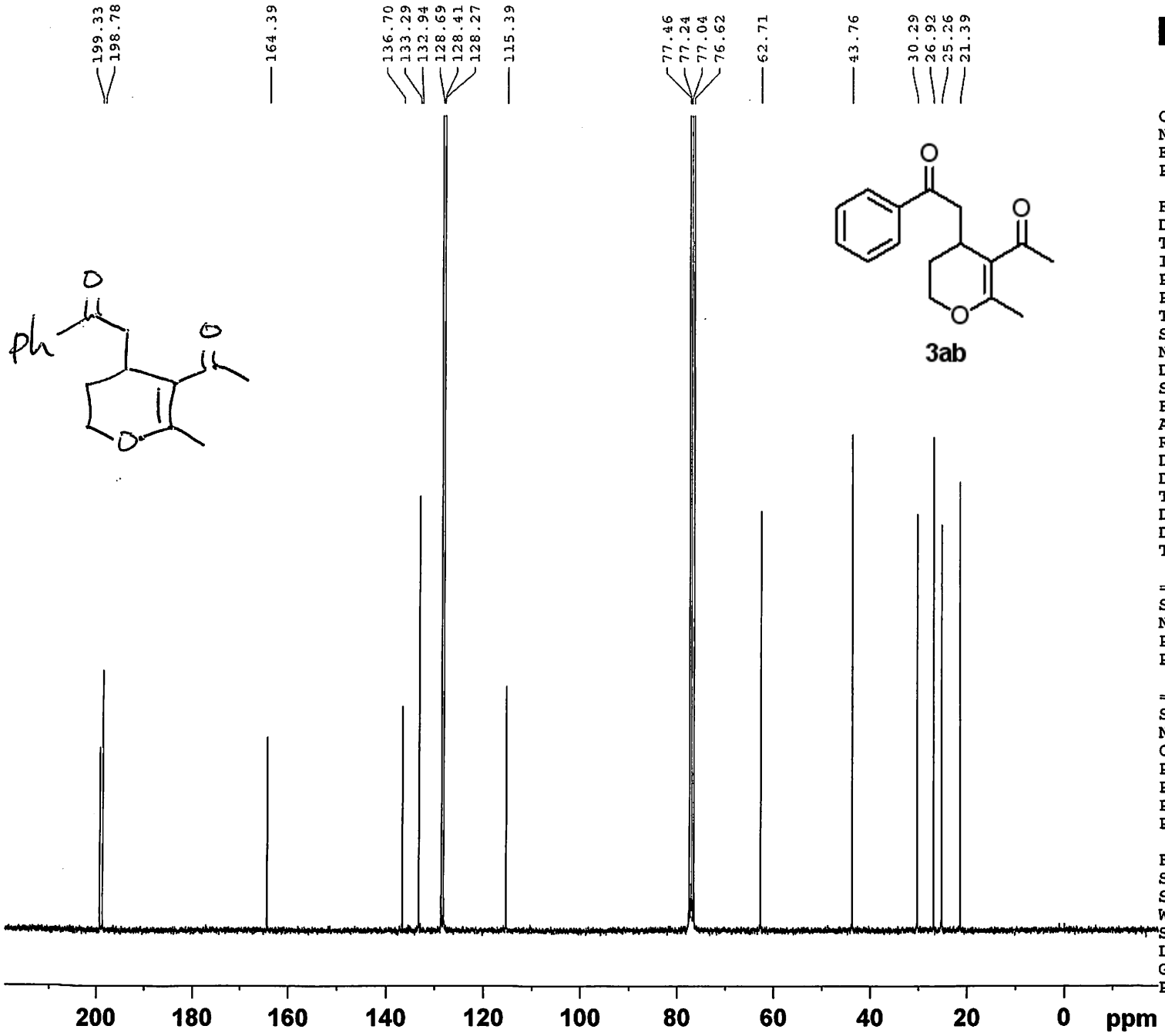
4.194 4.190 4.185 4.180 4.175 4.172 4.161 4.157 4.152 4.147 4.142 4.139 4.134 3.989 3.979 3.948 3.939 3.911 3.901 3.503 3.496 3.491 3.490 3.486 3.482 3.481 3.478 3.473 3.469 3.468 3.464 3.463 3.459 3.456 3.454 3.451 3.449 3.446 3.441 3.437 3.435 3.163 3.158 3.153 3.149 3.108 3.103 3.098 3.095 2.897 2.860 2.841 2.805

2.293 2.181 2.171 1.885 1.871 1.856 1.848 1.830 1.816 1.794 1.786 1.777 1.769 1.747 1.738 1.730 1.715

-0.011

20141215_AA_1

3ab



BRUKER

Current Data Parameters
NAME Yakka-5
EXPNO 171
PROCNO 1

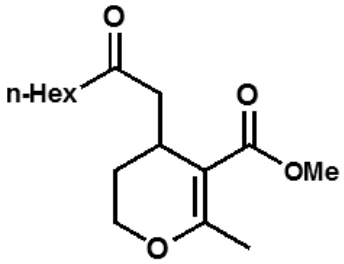
F2 - Acquisition Parameters
Date_ 20141216
Time 1.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4096
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 2050
DW 27.733 usec
DE 6.50 usec
TE 296.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 75.4752953 MHz
NUC1 13C
P1 10.00 usec
PLW1 32.00000000 W

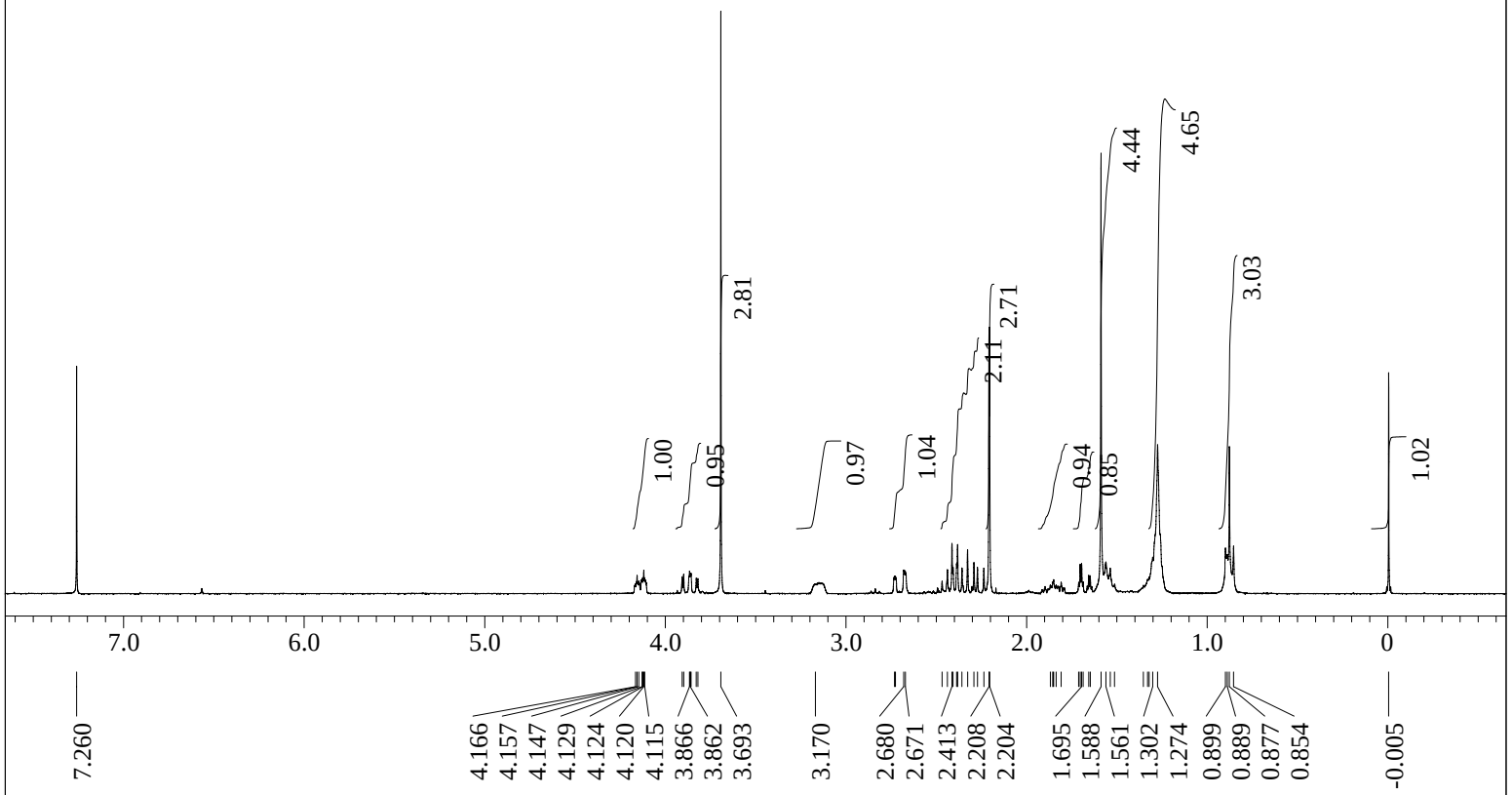
======== CHANNEL f2 ========
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 7.00000000 W
PLW12 0.24608999 W
PLW13 0.15750000 W

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

20150821_nHex_A



3ba



X : ppm : 1H

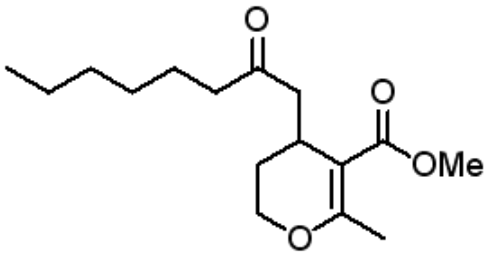
JEOL Solutions for Innovation

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2, 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
ppm
machinephase

以下に由来:: Yakka-6_1803-1.jdf
```

```
Filename       = Yakka-6_1803-8.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = 20150821_nHex_A
Solvent        = CHLOROFORM-D
Creation_Time  = 30-JUN-2016 16:28:43
Revision_Time  = 30-JUN-2016 16:39:40
Current_Time   = 30-JUN-2016 16:45:08

Comment        = 20150821_nHex_A
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 295.26[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```

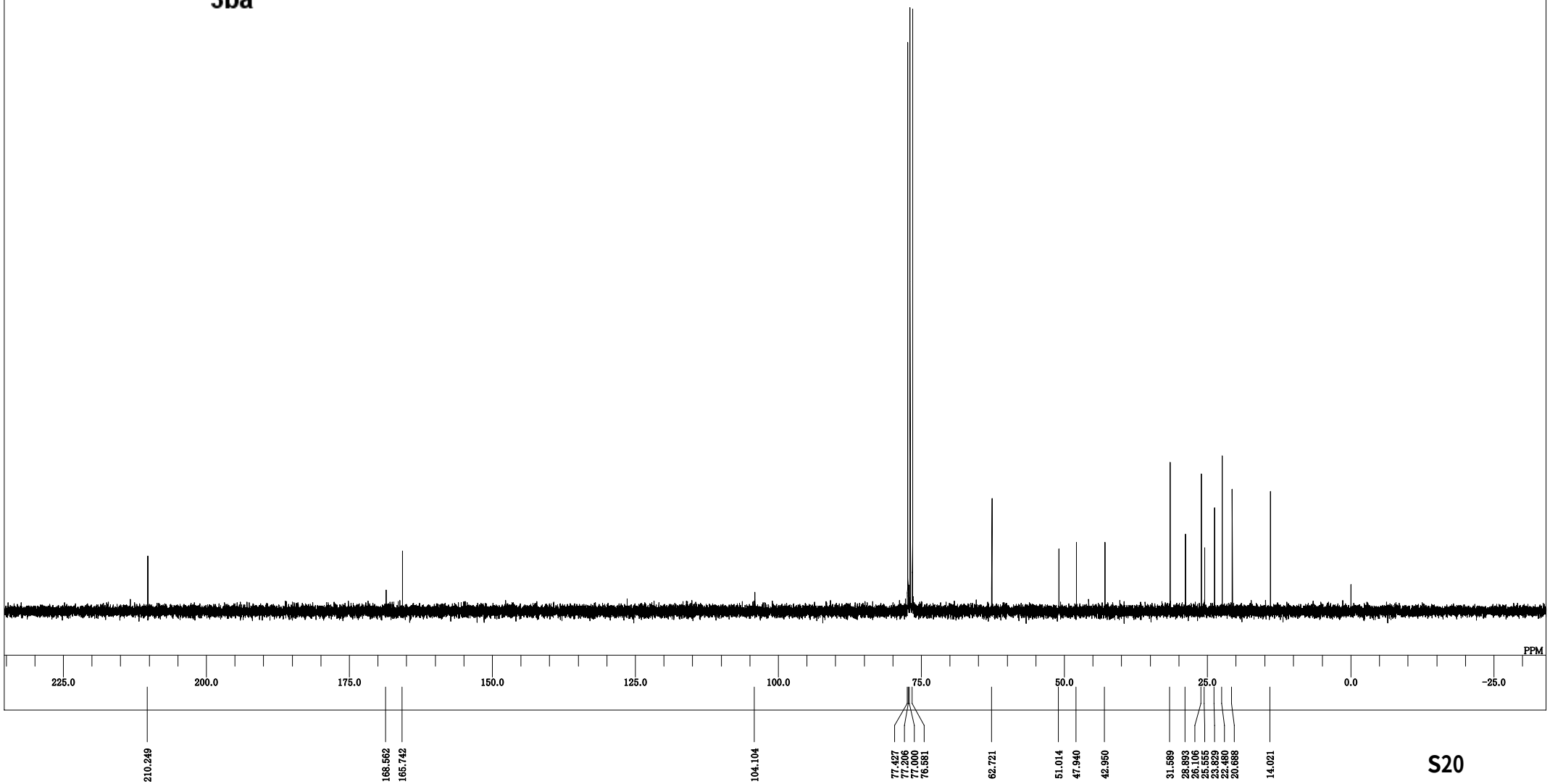
20150822_nHex_A

¥¥192.168.226.211¥anal¥6年生¥小黒¥20150822_nHex_A1BCM_E25.als



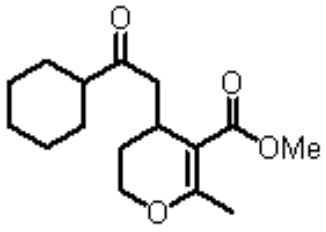
3ba

DFILE	20150822_nHex_A1BCM_E25.als
COMNT	20150822_nHex_A
DATIM	Sat Aug 22 13:45:31 2015
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.00 Hz
POINT	32768
FREQU	20356.23 Hz
SCANS	2500
ACQTM	1.6097 sec
PD	1.3900 sec
PW1	5.00 usec
IRNUC	1H
CTEMP	21.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	26



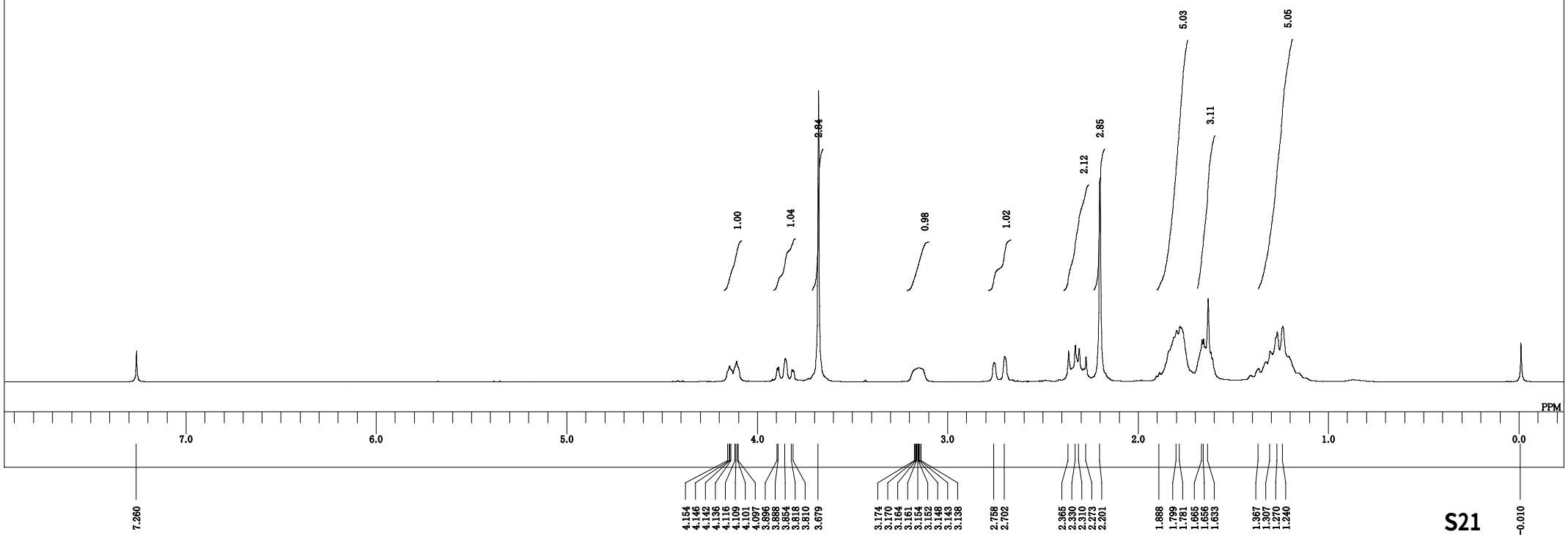
20150828_cHex

¥¥192.168.226.211¥anal¥2016年 3月卒業¥小黒¥20150828_cHex1NON_E48.als



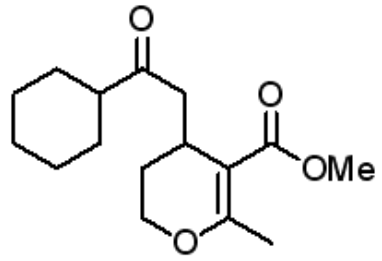
3ca

DFILE 20150828_cHex1NON_E48.als
COMNT 20150828_cHex
DATIM Fri Aug 28 16:30:30 2015
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.00 Hz
POINT 16384
FREQU 6006.01 Hz
SCANS 8
ACQTM 2.7279 sec
PD 4.2720 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF -0.01 ppm
BF 1.20 Hz
RGAIN 16



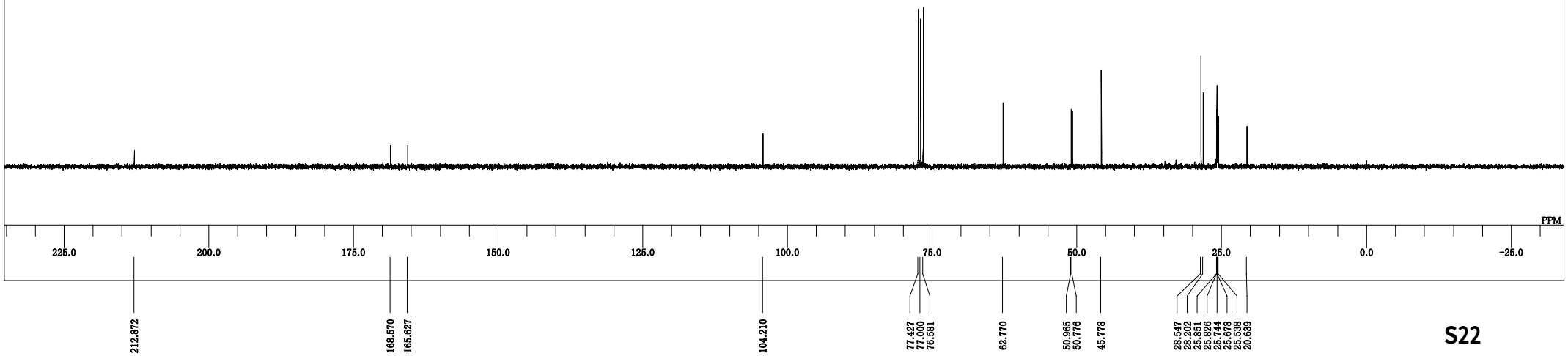
20150514cHex_A

¥¥192.168.226.211¥anal¥6年生¥小黒¥20150514cHex_A1BCM_E2.als



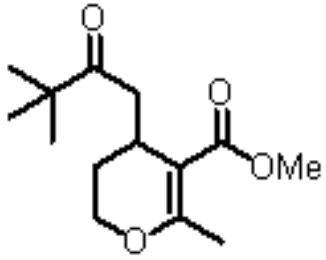
3ca

DFILE 20150514cHex_A1BCM_E2.als
COMNT 20150514cHex_A
DATIM Thu May 14 13:07:41 2015
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.00 Hz
POINT 32768
FREQU 20356.23 Hz
SCANS 2300
ACQTM 1.6097 sec
PD 1.3900 sec
PW1 5.10 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 25



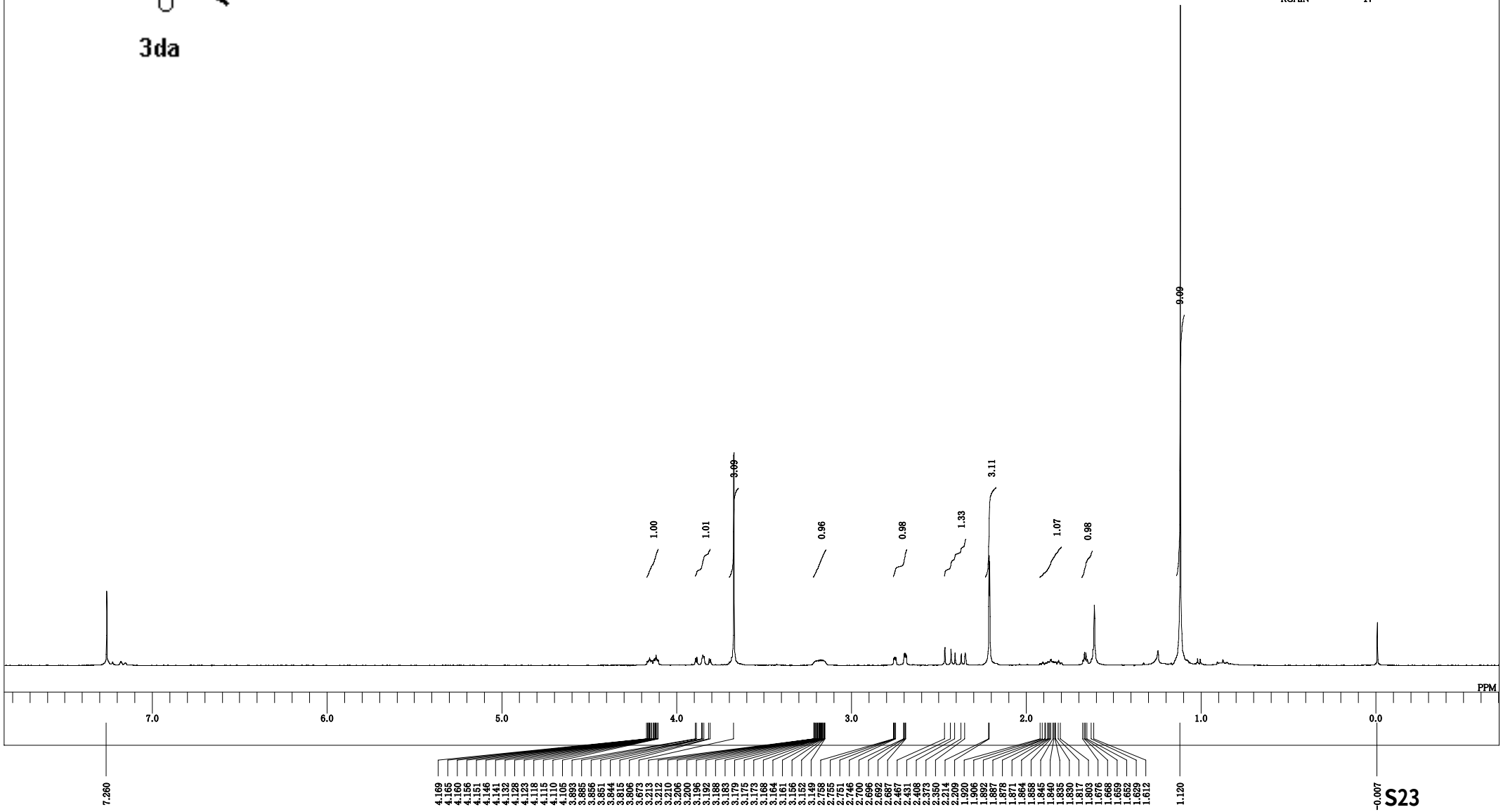
20150831_tBu_B

D:¥data¥卒業生 2016年3月卒業¥小黒¥20150831_tBu_B1NON_E2.als



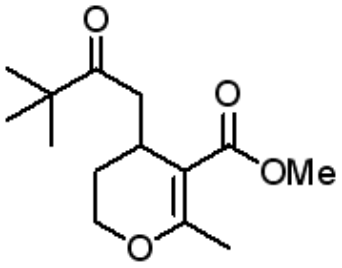
3da

DFILE 20150831_tBu_B1NON_E2.als
COMNT 20150831_tBu_B
DATIM Mon Aug 31 16:43:02 2015
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.00 Hz
POINT 16384
FREQU 6006.01 Hz
SCANS 8
ACQTM 2.7279 sec
PD 4.2720 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF -0.01 ppm
BF 0.12 Hz
RGAIN 17



20150519_tBu

¥¥192.168.226.211¥anal¥6年生¥小黒¥20150519_tBu1BCM_E22.als



3da

DFILE 20150519_tBu1BCM_E22.als
COMNT 20150519_tBu
DATIM Wed May 20 08:34:29 2015
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.00 Hz
POINT 32768
FREQU 20356.23 Hz
SCANS 4500
ACQTM 1.6097 sec
PD 1.3900 sec
PW1 5.10 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 26

PPM

225.0 200.0 175.0 150.0 125.0 100.0 75.0 50.0 25.0 0.0 -25.0

214.516 168.628 165.594 104.350 77.419 77.206 77.000 76.573 62.803 50.957 44.084 41.626 26.229 25.834 25.703 20.623