Electronic Supplementary Information

Direct Catalytic Asymmetric Aldol Reaction of β-Keto Esters with Formaldehyde Promoted by a Dinuclear Ni_2-Schiff Base Complex

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Experimental Section

General:

Infrared (IR) spectra were recorded on a JASCO FT/IR 410 Fourier transform infrared spectrophotometer. NMR spectra were recorded on JEOL JNM-LA500, ECX500, or ECA500 spectrometers, operating at 500 MHz for ^{1}H NMR and 125.65 MHz for ^{13}C NMR. Chemical shifts in $CDCl_3$ were reported in the scale relative to tetramethylsilane (0 ppm) for ^{1}H NMR. For ^{13}C NMR, chemical shifts were reported in the scale relative to $CHCl_3$ (77.0 ppm) as an internal reference. Column chromatography was performed with silica gel Merck 60 (230-400 mesh ASTM). Optical rotations were measured on a JASCO P-1010 polarimeter. ESI mass spectra were measured on Waters micromass ZQ (for LRMS) and FAB mass spectra (for HRMS) were measured on a JEOL JMS-700 spectrometer. The enantiomeric excess (ee) was determined by HPLC analysis. HPLC was performed on JASCO HPLC systems consisting of the following: pump, PU-2080 plus; detector, UV-2075 plus, measured at 280 nm; column, DAICEL CHIRALPAK AS-H or AD-H; mobile phase, hexane-2-propanol. Diisopropylether ($i$$Pr_2O$) was distilled from sodium benzophenone ketyl. Other reagents were purified by the usual methods. Formaldehyde solution (37% w/w in H_2O) and paraformaldehyde were purchased from Aldich. β-Keto esters[S1, S2] were prepared by following the same procedures as described in literatures.

References

[S1] C. Palomo, M. Oiarbide, J. M. García, P. Bañuelos, J. M. Odriozola, J. Razkin, A. Linden, *Org. Lett.* **2008**, *10*, 2637.

[S2] T. B. Poulsen, L. Bernardi, M. Bell, K. A. Jørgensen, *Angew. Chem., Int. Ed.* **2006**, *45*, 6551.

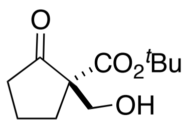
Preparation of Ni_2-Schiff Base 1 Complex:

To a solution of (*R,R*)-Schiff base ligand **1** (400 mg, 0.76 mmol) in EtOH (7.6 mL), was added $Ni(OAc)_2$•$4H_2O$ (378 mg, 1.52 mmol), and the mixture was stirred for 12 h under reflux. After cooling down to room temperature, H_2O (1.0 mL) was added to the mixture. The precipitate (Ni_2/Schiff base **1** complex) was collected by filtration. Then, the solid was washed with hexane and EtOH. The solid was dried under reduced pressure at 50 ºC to afford the Ni_2-Schiff base **1** complex (417 mg) as a dark yellow solid. The complex was used for the asymmetric reaction without further purification, and was stored under Ar at room temperature. Catalytic activity did not change for 5 months.

General Procedure for Catalytic Asymmetric Hydroxymethylation of β-Keto Esters Using a Ni_2-Schiff Base 1 Catalyst:

To a stirred solution of the Ni_2/Schiff base **1** catalyst (0.096 mg, 0.15 μmol) in $i$$Pr_2O$ (7.5 mL) was added β-keto ester **2a** (26.2 mg, 0.15 mmol). To the mixture at 40 °C was added 37% aqueous formaldehyde solution (21.7 mg, 0.165 mmol), and the resulting suspension was stirred for 1 h. The mixture was diluted with diethyl ether, and the precipitate was removed by filtration through a pad of Celite. After the filtrate was concentrated under reduced pressure, the residue was purified by silica gel flash column chromatography (hexane/ethyl acetate = 3/1) to give the desired product **3a** (30.2 mg, 94% yield) as a colorless oil.

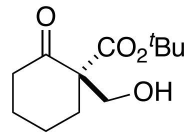
(*S*)-*tert*-butyl 1-(hydroxymethyl)-2-oxocyclopentanecarboxylate (3a)



colorless oil; IR (neat) ν 3460, 2973, 1748, 1721 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.45 (s, 9H), 1.93-2.11 (m, 2H), 2.14-2.20 (m, 1H), 2.25-2.36 (m, 2H), 2.42-2.50 (m, 1H), 2.74 (dd, *J* = 4.6, 9.0, 1H), 3.75 (dd, *J* = 9.0, 12.0, 1H), 3.85 (dd, *J* = 4.6, 12.0, 1H); ^{13}C NMR ($CDCl_3$) δ 19.7, 27.9, 31.3, 38.4, 62.2, 63.8, 82.6, 170.8, 215.4; LRMS(ESI): *m/z* 237 $[M+Na]^+$; HRMS (FAB): *m/z* calculated for $C_{11}H_{18}O_4Cs^+$ $[M+Cs]^+$: 347.0260, found: 347.0272, $[\alpha]_D^{16.0}$ +6.3 (*c* 1.00, $CHCl_3$); HPLC (DAICEL CHIRALPAK AS-H, hexane/2-propanol

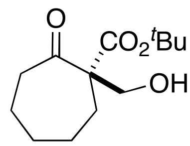
= 95/5, flow 0.5 mL/min, detection at 280 nm) t_R 20.4 min (major) and 25.2 min (minor).

(*S*)-*tert*-butyl 1-(hydroxymethyl)-2-oxocyclohexanecarboxylate (3b)



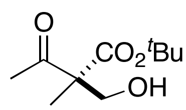
colorless oil; IR (neat) ν 3463, 2978, 1709 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.49 (s, 9H), 1.49-1.63 (m, 3H), 1.78-1.82 (m, 1H), 2.02-2.08 (m, 1H), 2.27-2.32 (m, 1H), 2.41-2.47 (m, 1H), 2.60-2.68 (m, 1H), 2.85 (dd, *J* = 5.3, 9.7 Hz, 1H), 3.68 (dd, *J* = 9.7, 11.5 Hz, 1H), 3.78 (dd, *J* = 5.3, 11.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 22.0, 26.9, 27.9, 32.9, 41.0, 63.1, 66.7, 82.7, 170.3, 211.3; LRMS(ESI): *m/z* 251 $[M+Na]^+$; HRMS (FAB): *m/z* calculated for $C_{12}H_{20}O_4Cs^+$ $[M+Cs]^+$: 361.0416, found: 361.0406; $[\alpha]_D^{19.0}$ +5.2 (*c* 1.04, $CHCl_3$), HPLC (DAICEL CHIRALPAK AS-H, hexane/2-propanol = 95/5, flow 0.5 mL/min, detection at 280 nm) t_R 14.3 min (major) and 17.2 min (minor).

(*S*)-*tert*-butyl 1-(hydroxymethyl)-2-oxocycloheptanecarboxylate (3c)



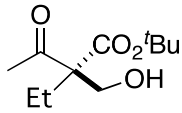
colorless oil; IR (neat) ν3535, 2976, 2932, 2862, 1701 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.48 (s, 9H), 1.58-1.64 (m, 4H), 1.74-1.84 (m, 3H), 1.91-1.96 (m, 1H), 2.49- 2.54 (m, 1H), 2.71-2.76 (m, 1H), 3.04 (dd, *J* = 4.9, 9.7 Hz ,1H), 3.68 (dd, *J* = 9.7, 11.4 Hz, 1H), 3.94 (dd, *J* = 4.9, 11.4 Hz 1H); ^{13}C NMR ($CDCl_3$) δ 25.2, 25.6, 27.9, 30.1, 30.9, 43.0, 64.4, 67.3, 82.6, 171.3, 212.6 ; ESI-MS *m/z* 265 $[M+Na]^+$; HRMS calcd. for $C_{13}H_{22}O_4Cs$ $[M+Cs]^+$:375.0573, found 375.0561; $[\alpha]_D^{18.0}$ +4.6 (*c* 1.23, $CHCl_3$); HPLC (DAICEL CHIRALPAK AD-H, hexane/2-propanol = 90/10, flow 0.5 mL/min, detection at 280 nm) t_R 13.1 min (major) and 16.1 min (minor).

(*S*)-*tert*-butyl 2-(hydroxymethyl)-2-methyl-3-oxobutanoate (3d)



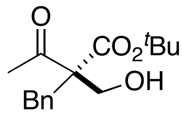
colorless oil; IR (neat) ν 3419, 2979, 1735, 1715 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.35 (s, 3H), 1.48 (s, 9H), 2.23 (s, 3H), 2.73 (dd, *J* = 6.9, 8.0 Hz), 3.76 (dd, *J* = 8.0, 11.5 Hz), 3.85 (dd, *J* = 6.9, 11.5); ^{13}C NMR ($CDCl_3$) δ 17.2, 27.0, 27.9, 61.7, 66.6, 82.6, 171.5, 207.0; ESI-MS *m/z* 225 $[M+Na]^+$; HRMS calcd. for $C_{10}H_{18}O_4Cs$ $[M+Cs]^+$: 335.0260, found335.0250; $[\alpha]_D^{19.0}$ +4.6 (*c* 1.03, $CHCl_3$); HPLC (DAICEL CHIRALPAK AD-H, hexane/2-propanol = 90/10, flow 0.5 mL/min, detection at 280 nm) t_R 13.2 min (major) and 15.1 min (minor).

(*S*)-*tert*-butyl 2-ethyl-2-(hydroxymethyl)-3-oxobutanoate (3e)



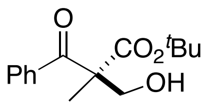
colorless oil; [1]H NMR ($CDCl_3$) δ 0.90 (t, *J* = 8.0 Hz, 3H), 1.49 (s, 9H), 1.81-1.89 (m, 1H), 1.92-2.00 (m, 1H), 2.23 (s, 3H), 2.47 (dd, *J* = 6.0, 8.0 Hz ,1H), 3.85 (dd, *J* = 8.0, 12.0 Hz, 1H), 3.96 (dd, *J* = 6.0, 12.0 Hz, 1H); [13]C NMR ($CDCl_3$) δ 8.8, 24.3, 27.5, 27.9, 63.9, 65.9, 82.5, 171.1, 206.8; ESI-MS *m/z* 239 $[M+Na]^+$; HRMS calcd. for $C_{11}H_{20}O_4Cs$ $[M+Cs]^+$: 349.0416, found 349.0408; $[\alpha]_D^{26.0}$ +5.3 (*c* 1.11, $CHCl_3$); HPLC (DAICEL CHIRALPAK AS-H, hexane/2-propanol = 90/10, flow 0.5 mL/min, detection at 280 nm) t_R 12.3 min (major) and 13.1 min (minor).

(*S*)-*tert*-butyl 2-benzyl-2-(hydroxymethyl)-3-oxobutanoate (3f)



colorless oil; IR (neat) ν 3463, 2978, 2938, 1709 cm^{-1}; [1]H NMR ($CDCl_3$) δ 1.46 (s, 9H), 2.24 (dd, *J* = 5.5, 7.5, 1H), 2.27 (s, 3H), 3.18 (d, *J* = 14.0 Hz, 1H), 3.25 (d, *J* = 14.0, 1H), 3.79 (dd, *J* = 7.5, 12.0 Hz, 1H), 3.93 (dd, *J* = 5.5, 12.0 Hz, 1H), 7.18-7.29 (m, 5H); [13]C NMR ($CDCl_3$) δ 27.87, 27.92, 36.5, 63.7, 66.7, 82.9, 127.0, 128.3, 130.1, 135.9, 170.2, 205.6; ESI-MS *m/z* 301 $[M+Na]^+$; HRMS calcd. for $C_{16}H_{22}O_4Cs$ $[M+Cs]^+$: 411.0573, found 411.0580; $[\alpha]_D^{26.0}$ +2.6 (*c* 1.23, $CHCl_3$); HPLC (DAICEL CHIRALPAK AD-H, hexane/2-propanol = 9/1, flow 0.5 mL/min, detection at 254 nm) t_R 15.0 min (major) and 18.6 min (minor).

(*S*)-*tert*-butyl 2-benzyl-2-(hydroxymethyl)-3-oxo-3-phenylpropanoate (3g)



colorless solid; mp 66-70 °C; IR (neat) ν 3487, 2976, 2932, 1718, 1679 cm^{-1}; [1]H NMR ($CDCl_3$) δ 1.32 (s, 9H), 1.58 (s, 3H), 2.79 (dd, *J* = 6.9, 6.9, 1H), 3.93 (d, *J* = 6.9, 11.5 Hz, 1H), 4.01 (dd, *J* = 6.9, 11.5 Hz, 1H), 7.42-7.46 (m, 2H), 7.53-7.57 (m, 1H), 7.86-7.89 (m, 2H); [13]C NMR ($CDCl_3$) δ 18.6, 27.6, 59.6, 68.0, 82.6, 128.4, 128.8, 133.0, 135.4, 171.8, 199.3; ESI-MS *m/z* 287 $[M+Na]^+$; HRMS calcd. for $C_{15}H_{20}O_4Cs$ $[M+Cs]^+$: 397.0416, found 397.0414; $[\alpha]_D^{13.0}$ +2.6 (*c* 1.00, $CHCl_3$); HPLC (DAICEL CHIRALPAK AD-H, hexane/2-propanol = 9/1, flow 0.5 mL/min, detection at 254 nm) t_R 13.9 min (major) and 15.3 min (minor).

5

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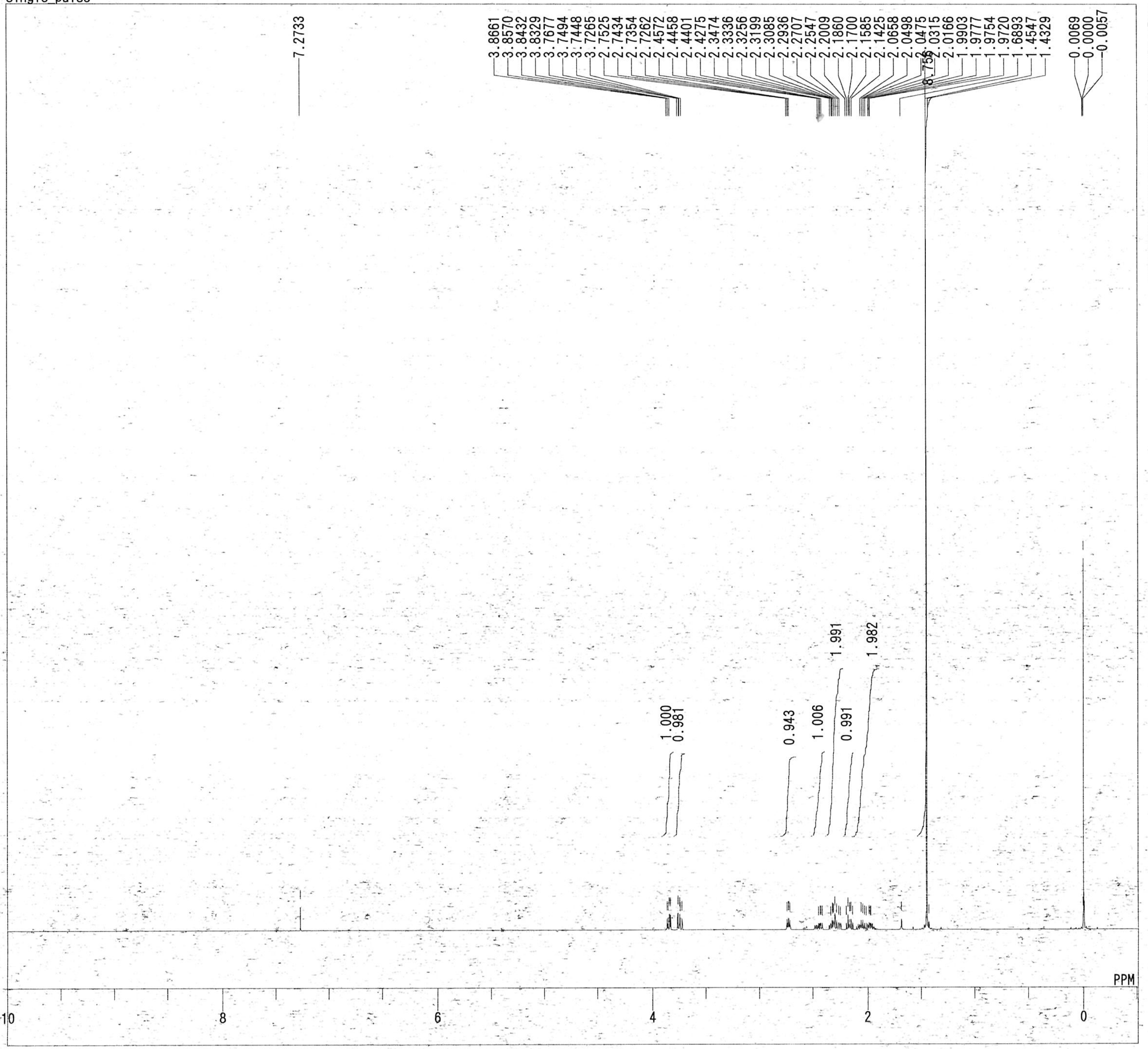

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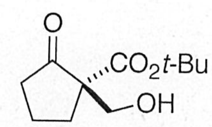

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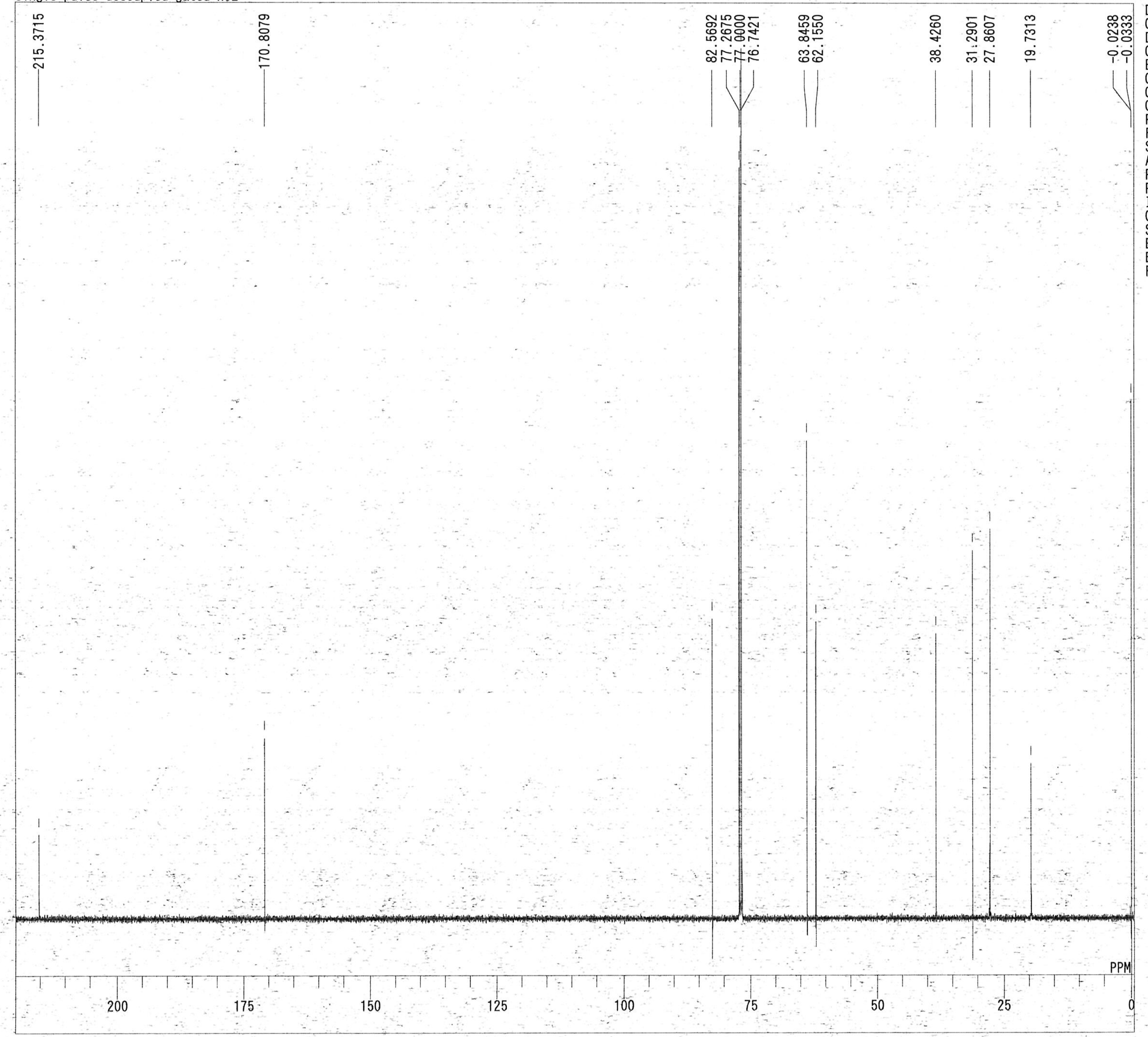


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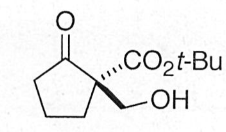

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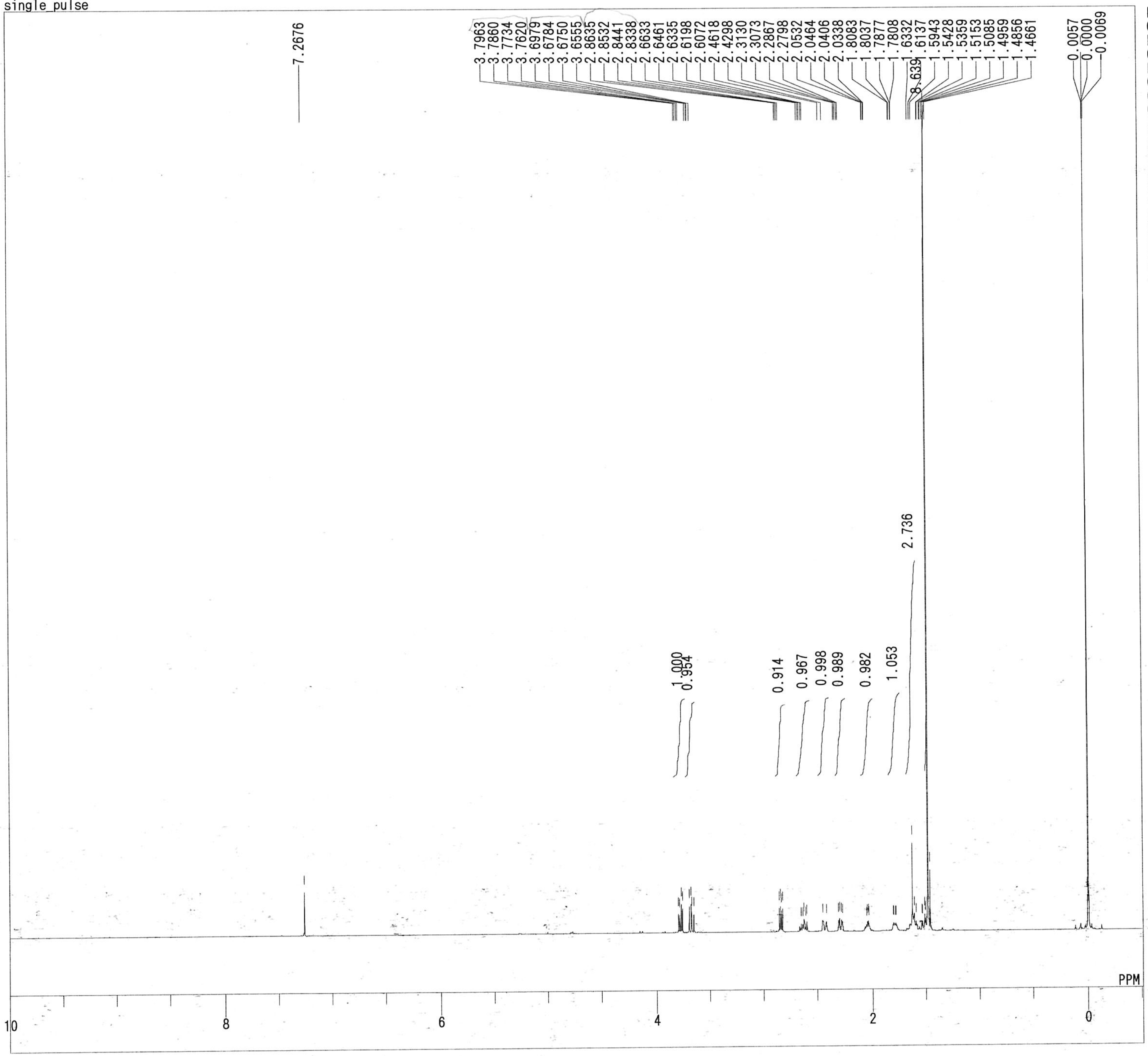

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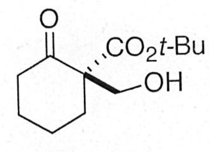

3b

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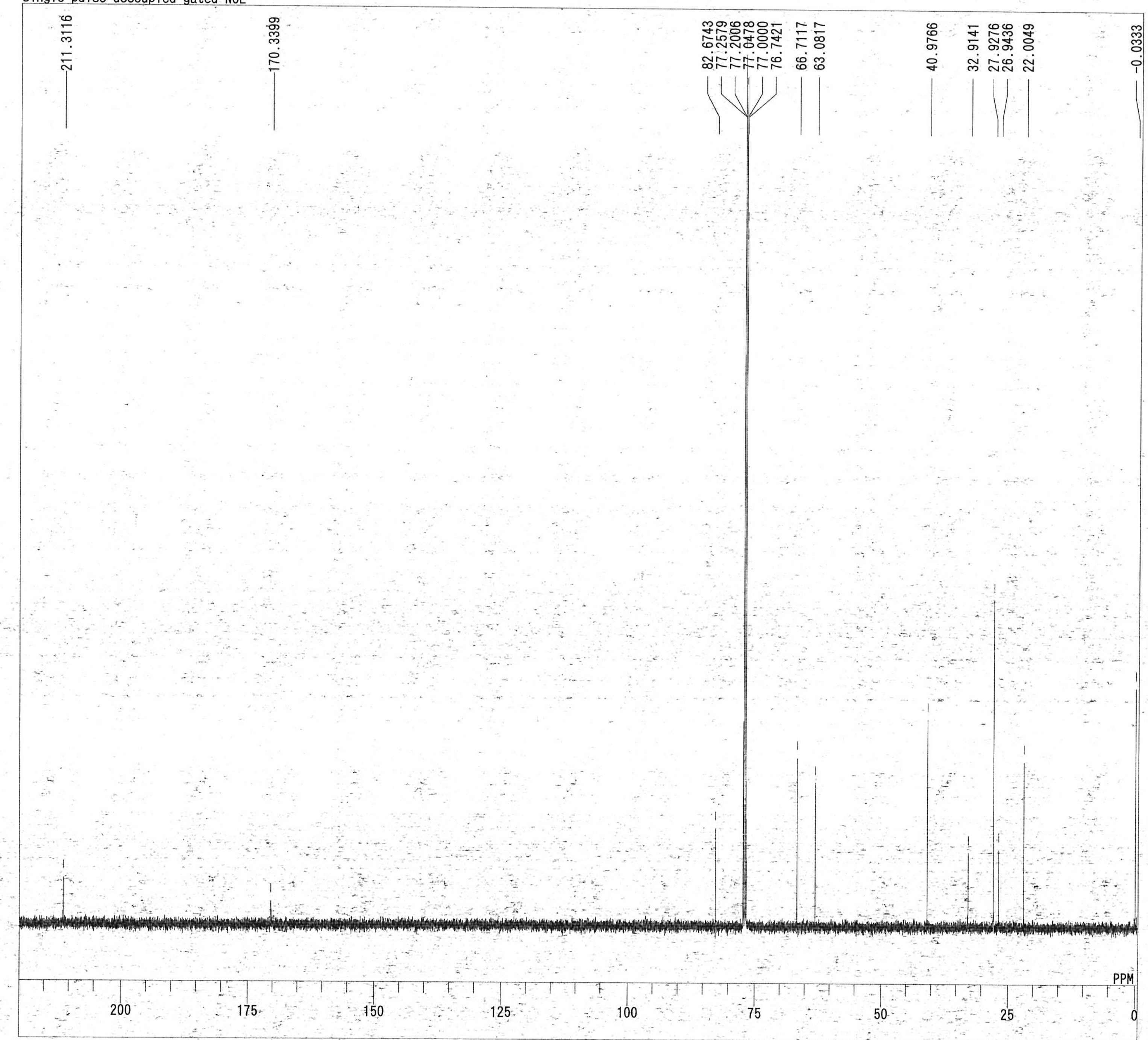

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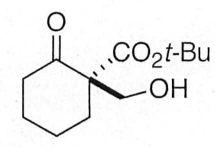

3b

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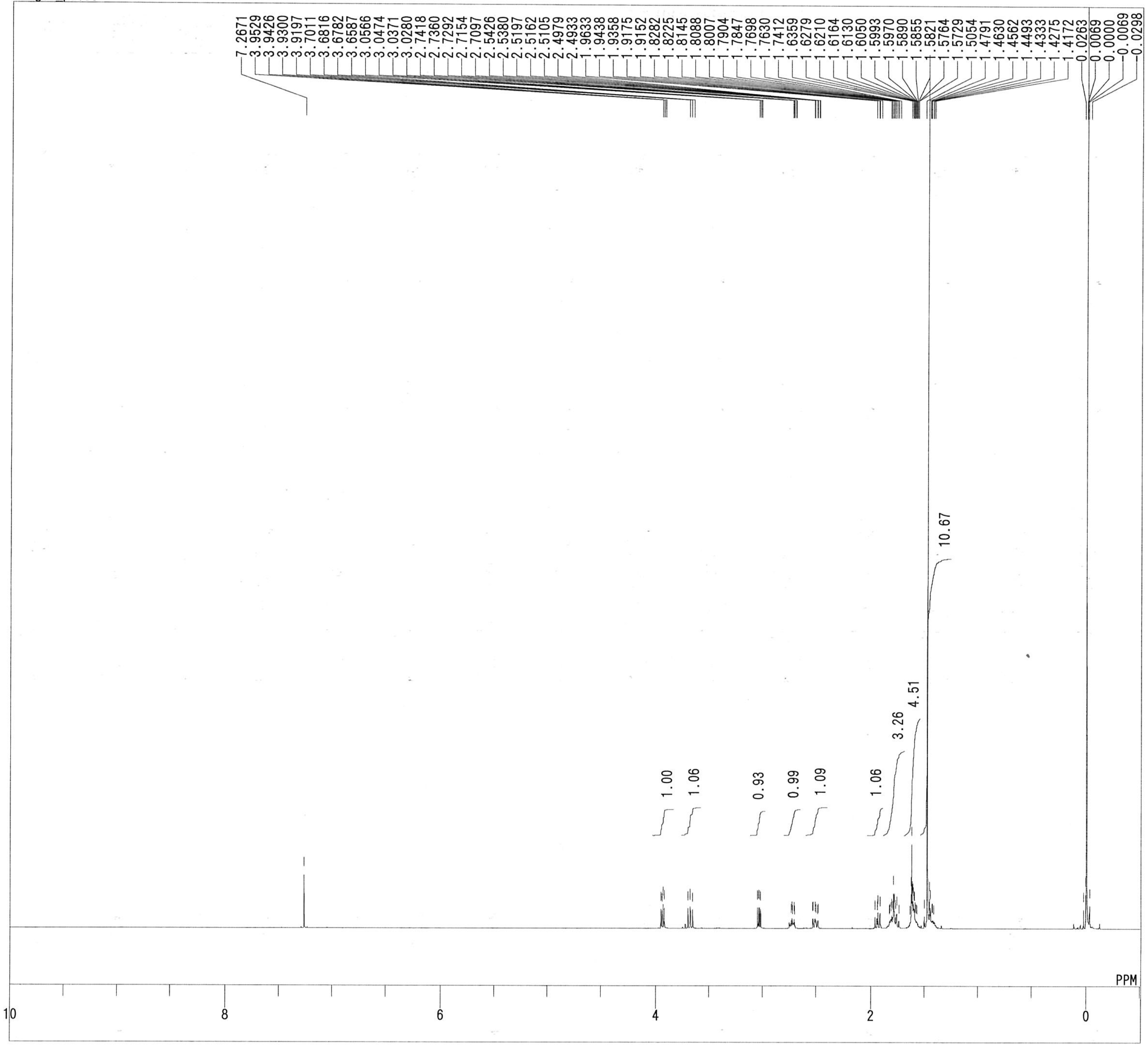

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PD	5.0000	sec
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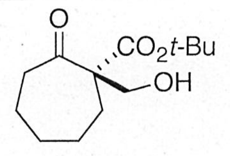
RGAIN	46	



3c

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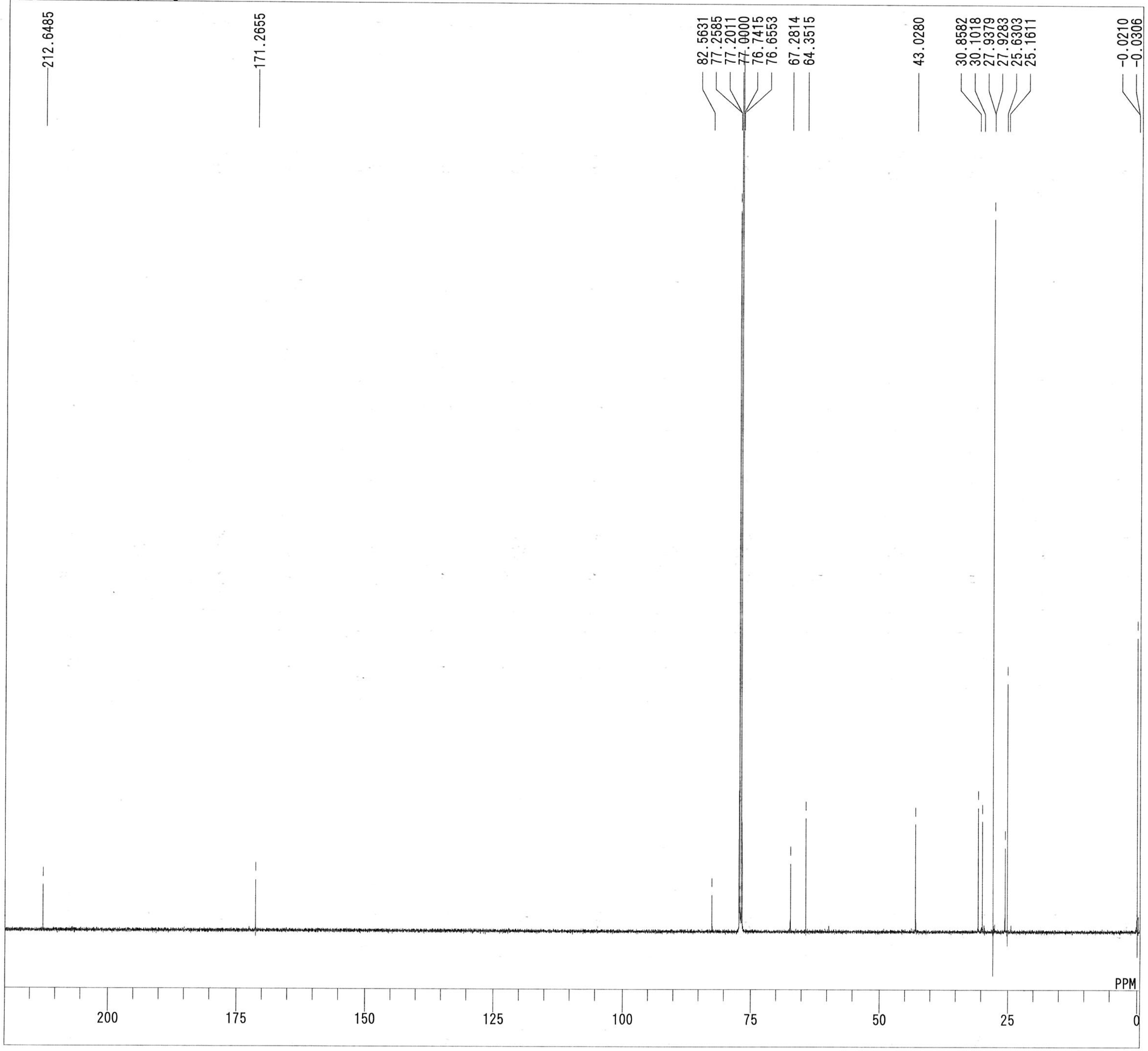

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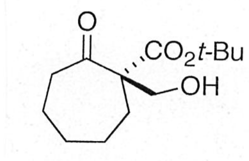
RGAIN	60



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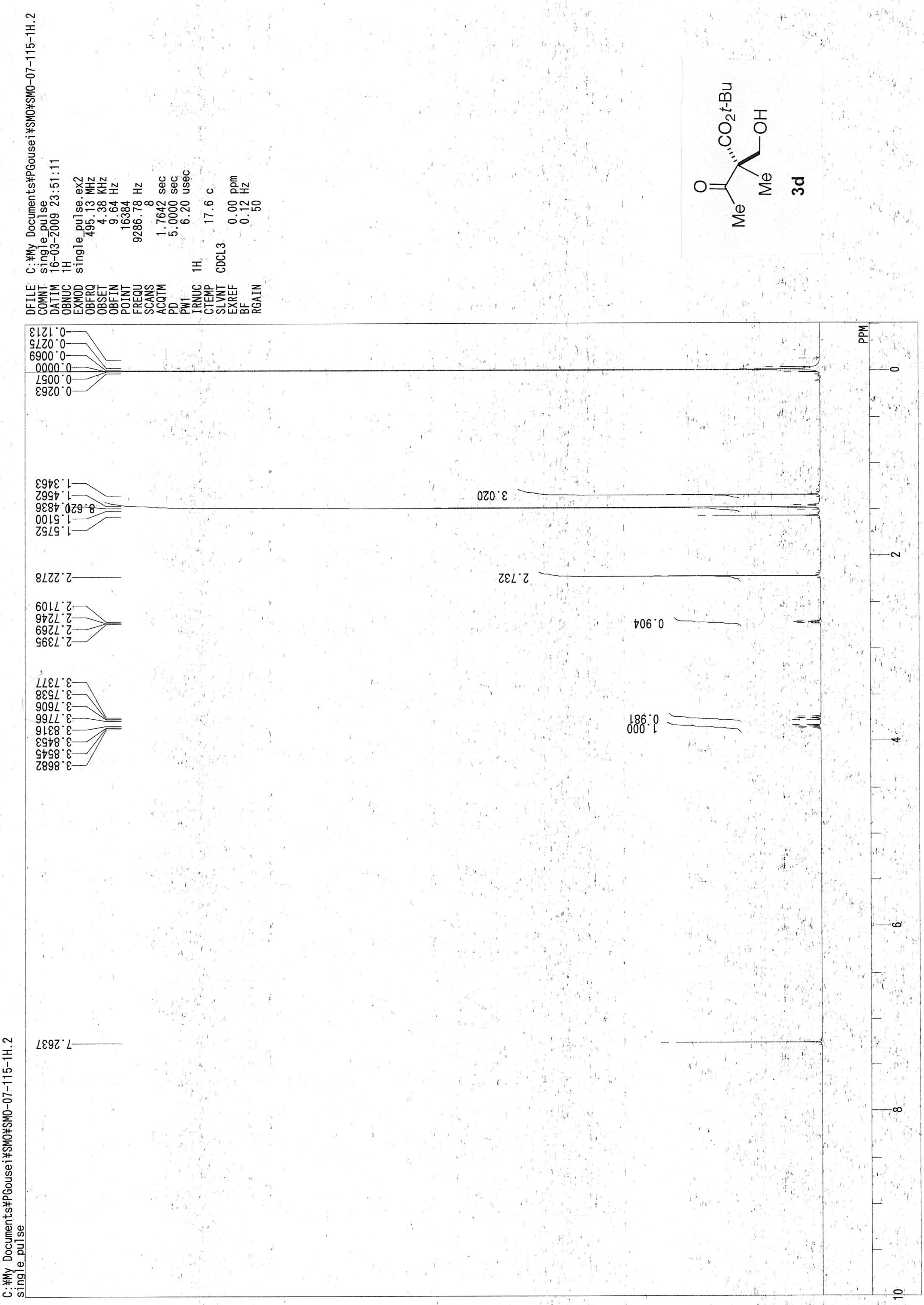
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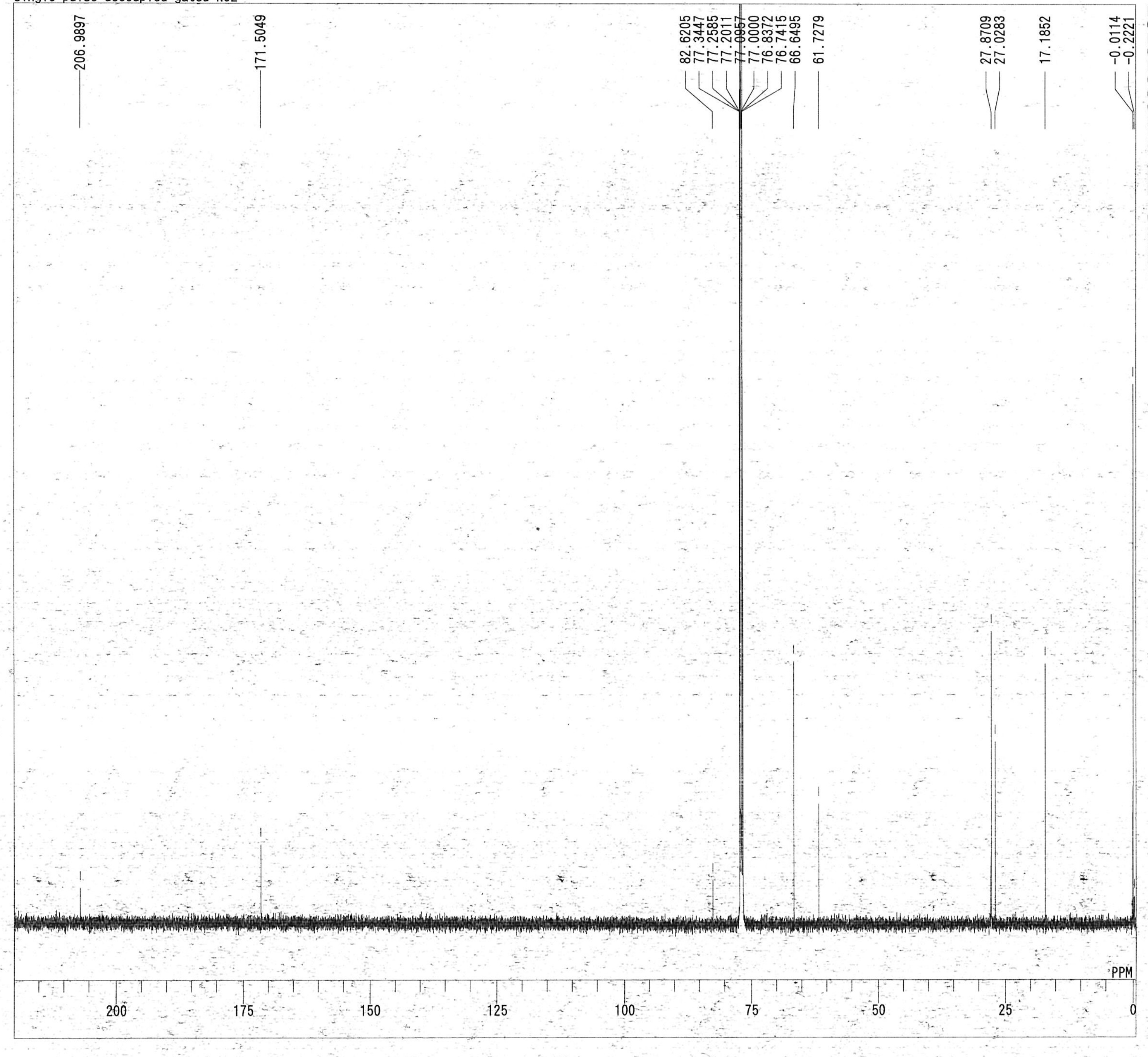


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RGAIN           60
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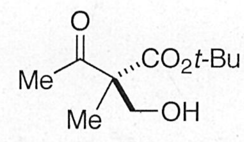
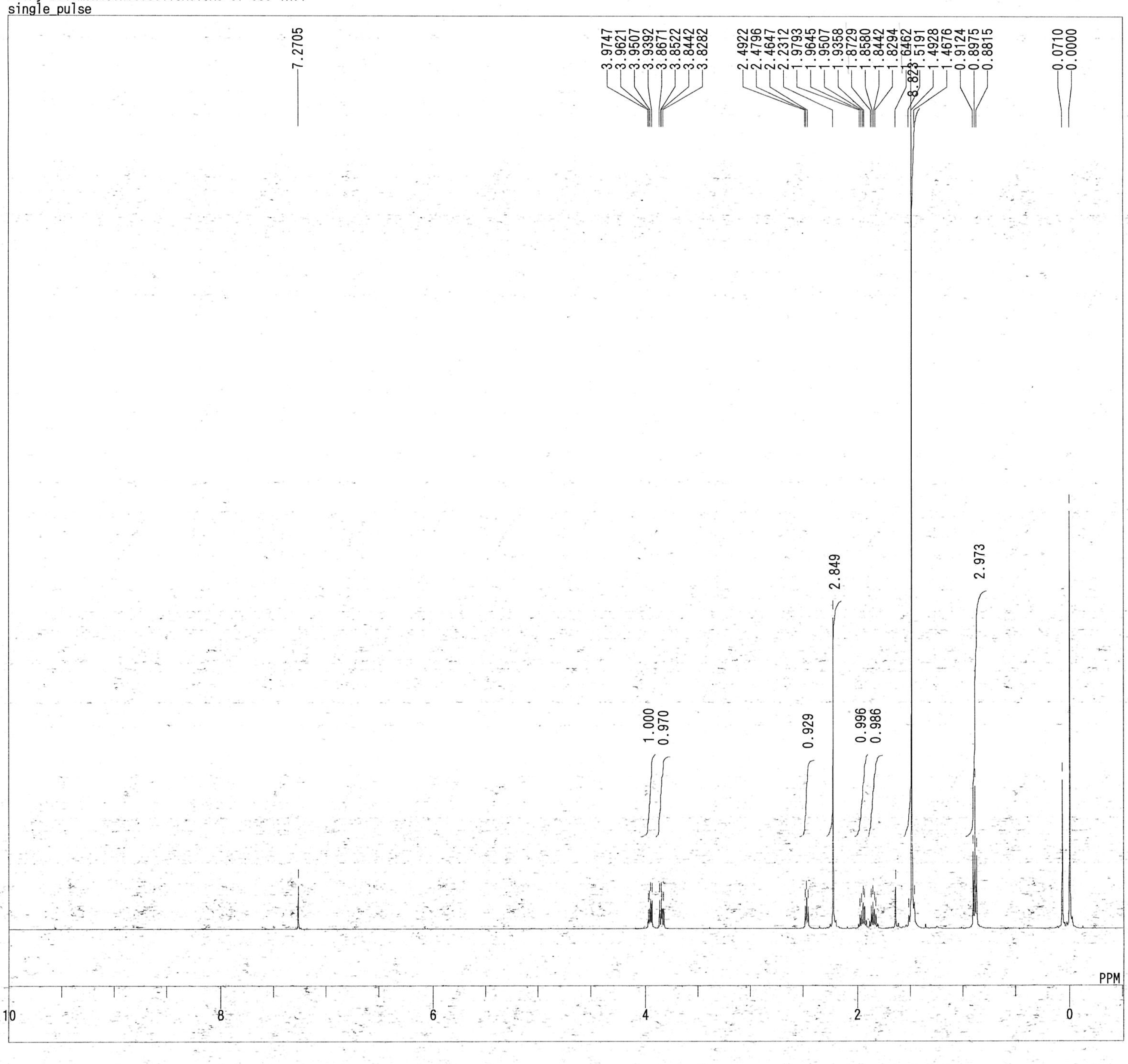
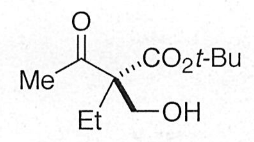


3d

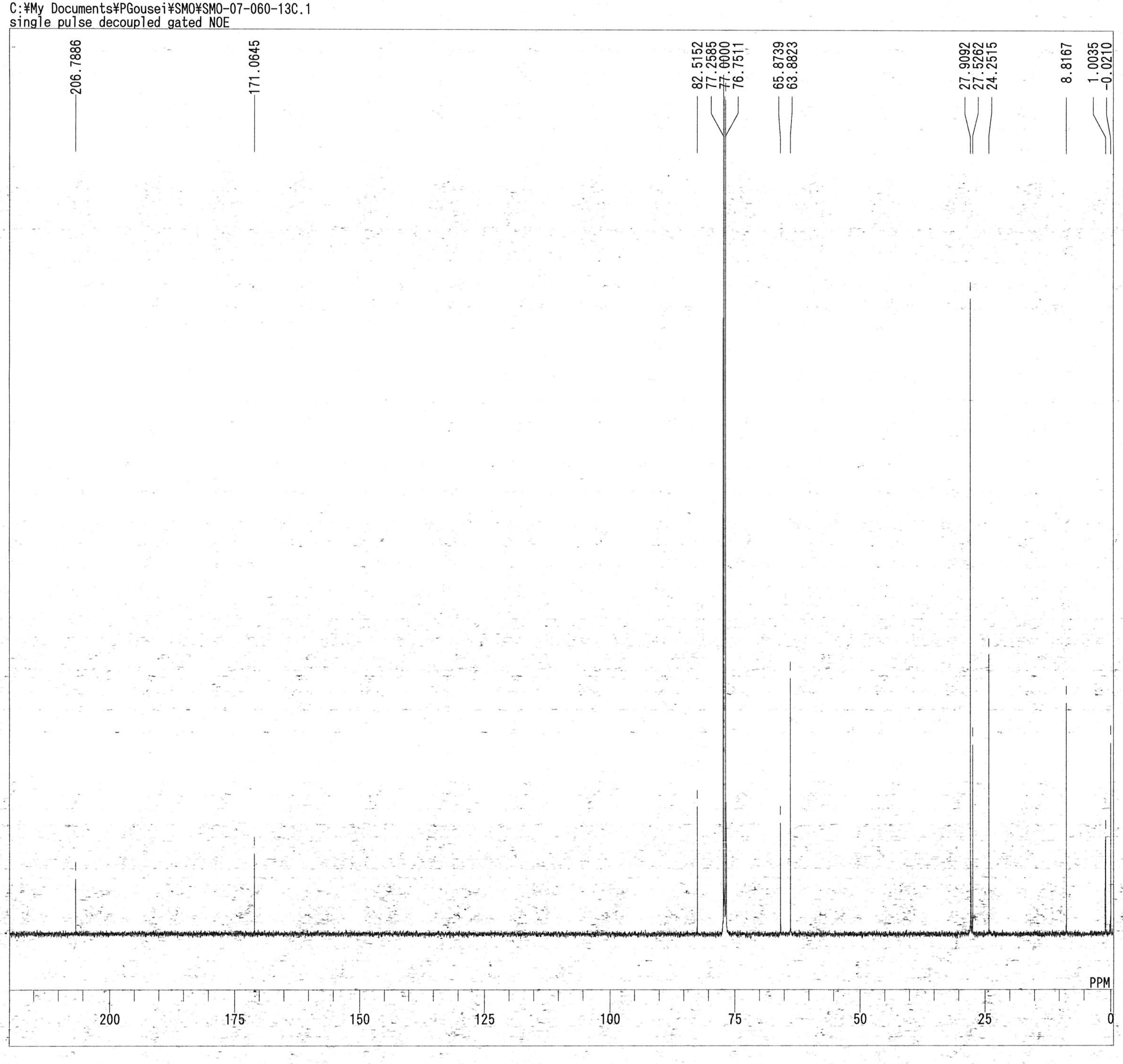
C:¥My Documents¥PGousei¥SMO¥SMO-07-060-1H.1
single_pulse



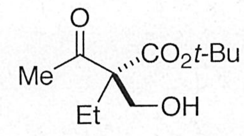
DFILE	C:¥My Documents¥PGousei¥SMO¥SMO-07-060-1H.1
COMNT	single_pulse
DATIM	05-03-2009 15:13:43
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	495.13 MHz
OBSET	4.38 KHz
OBFIN	9.64 Hz
POINT	16384
FREQU	9286.78 Hz
SCANS	8
ACQTM	1.7642 sec
PD	5.0000 sec
PW1	6.20 usec
IRNUC	1H
CTEMP	18.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	1.00 Hz
RGAIN	44



3e

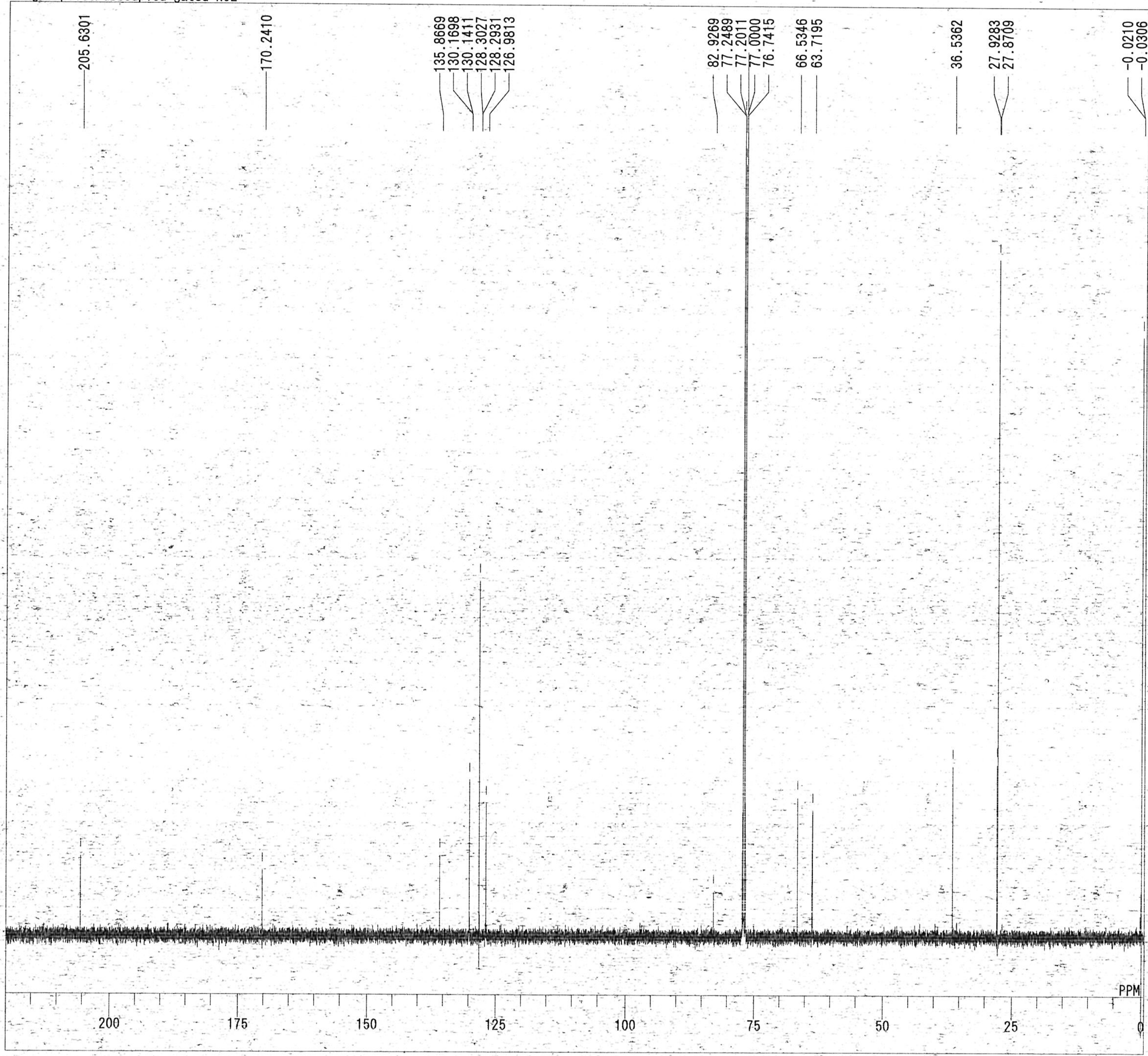


DFILE	C:¥My Documents¥PGousei¥SMO¥SMO-07-060-13C.1
COMNT	single pulse decoupled gated NOE
DATIM	05-03-2009 17:21:48
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	124.51 MHz
OBSET	3.45 KHz
OBFIN	6.00 Hz
POINT	32768
FREQU	39062.50 Hz
SCANS	2650
ACQTM	0.8389 sec
PD	2.0000 sec
PW1	3.57 usec
IRNUC	1H
CTEMP	19.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	60

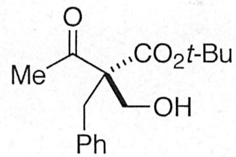


3e

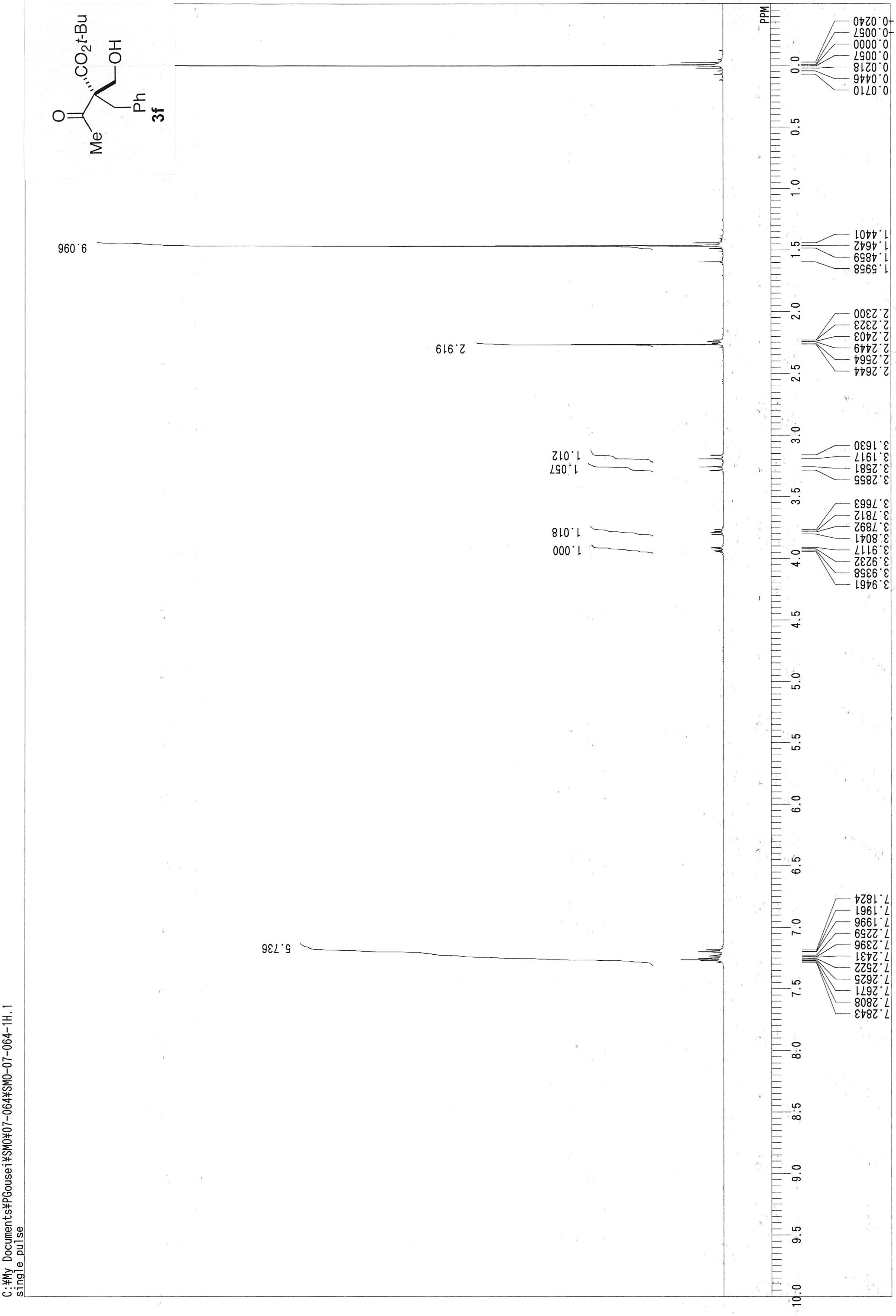
C:¥My Documents¥PGousei¥SMO¥07-064¥SMO-07-064-13C.1
single pulse decoupled gated NOE

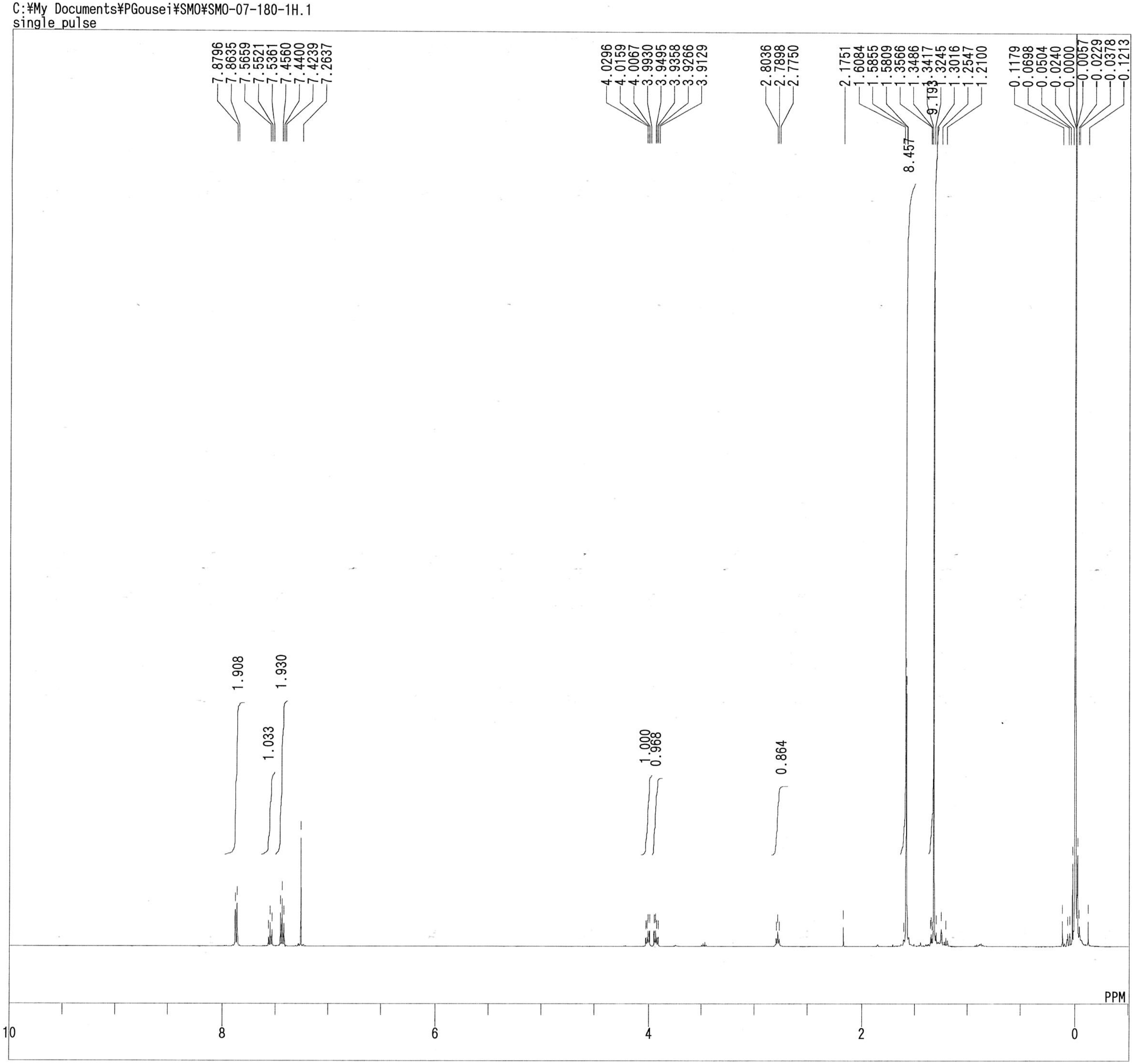


DFILE C:¥My Documents¥PGousei¥SMO¥07-064¥SMO-07-064-13C.1
COMNT single pulse decoupled gated NOE
DATIM 16-02-2009 21:34:10
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 124.51 MHz
OBSET 3.45 KHz
OBFIN 6.00 Hz
POINT 32768
FREQU 39062.50 Hz
SCANS 800
ACQTM 0.8389 sec
PD 2.0000 sec
PW1 3.57 usec
IRNUC 1H
CTEMP 19.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.01 Hz
RGAIN 60

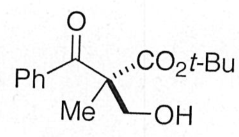


3f

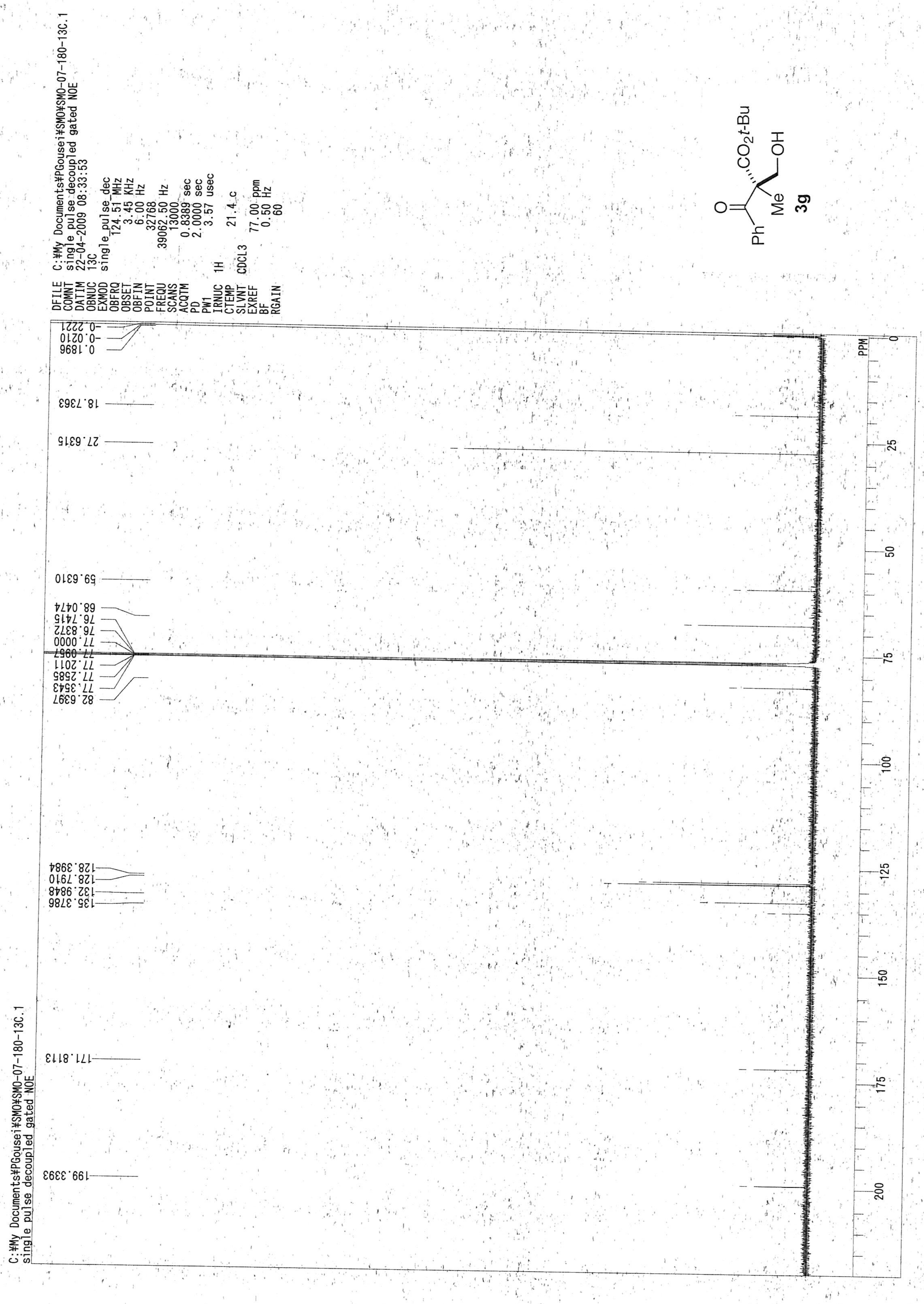

Me
O
CO2t-Bu
OH
Ph
3f
PPM
-0.0240
-0.0057
0.0000
0.0057
0.0218
0.0446
0.0710
1.4401
1.4642
1.4859
1.5958
2.2300
2.2323
2.2403
2.2449
2.2564
2.2644
3.1630
3.1917
3.2581
3.2855
3.7663
3.7812
3.7892
3.8041
3.9117
3.9232
3.9358
3.9461
7.1824
7.1961
7.1996
7.2259
7.2396
7.2431
7.2522
7.2625
7.2671
7.2808
7.2843
9.096
2.919
1.012
1.057
1.018
1.000
5.736
C:¥My Documents¥PGousei¥SMO¥07-064¥SMO-07-064-1H.1
single_pulse



DFILE C:¥My Documents¥PGousei¥SMO¥SMO-07-180-1H.1
COMNT single_pulse
DATIM 21-04-2009 22:16:19
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 495.13 MHz
OBSET 4.38 KHz
OBFIN 9.64 Hz
POINT 16384
FREQU 9286.78 Hz
SCANS 32
ACQTM 1.7642 sec
PD 5.0000 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 50



3g


DFILE C:¥My Documents¥PGousei¥SMO¥SMO-07-180-13C.1
COMNT single pulse decoupled gated NOE
DATIM 22-04-2009 08:33:53
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 124.51 MHz
OBSET 3.45 KHz
OBFIN 6.00 Hz
POINT 32768
FREQU 39062.50 Hz
SCANS 13000
ACQTM 0.8389 sec
PD 2.0000 sec
PW1 3.57 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.50 Hz
RGAIN 60
-0.2221
-0.0210
0.1896
18.7363
27.6315
59.6310
68.0474
76.7415
76.8372
77.0000
77.0957
77.2011
77.2585
77.3543
82.6397
128.3984
128.7910
132.9848
135.3786
171.8113
199.3393
PPM
0
25
50
75
100
125
150
175
200
C:¥My Documents¥PGousei¥SMO¥SMO-07-180-13C.1
single pulse decoupled gated NOE
O
Ph
CO2t-Bu
Me
OH
3g