Formation of a Quaternary Carbon Center through the Pd(0)/PhCOOH Catalyzed Allylation of Cyclic β-Keto Esters and 1,3-Diketones with Alkynes

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Contents of Supporting Information

(15 Pages)

General Procedure. A representative procedure for the palladium/benzoic acid catalyzed allylation is as follows. To a solution of **1a** (0.053 g, 0.3443 mmol) in toluene (0.07 mL) were added 1-phenyl-1-propyne **2a** (0.040 g, 0.3443 mmol), $Pd_2dba_3.CHCl_3$ (0.018 g, 0.0172 mmol), PPh_3 (0.022 g, 0.1377 mmol) and PhCOOH (0.004 g, 0.0344 mmol). The mixture was stirred at 100 °C for 8-15 h in a screw capped vial. The reaction progress was monitored by TLC. The solvent was removed under reduced pressure, and the residue was purified by short silica gel column with hexane:ethyl acetate, 9:1 to give **3a** (0.088 g) in 95% yield.

Structure of **3a**,[1] **3d**,[2] **3e**,[3] **3h**,[4] **3l**,[3] **3m**,[5] **3n**[3] are known in literature and their structure is confirmed by comparison with the published spectral data. The characterization data for the newly synthesized compounds **3b**, **3c**, **3f**, **3g**, **3i**, **3j**, **3k**, **3o** and the copies of their ^{1}H NMR spectra given below.

1-[3-(4-Chloro-phenyl)-allyl]-2-oxo-cyclopentanecarboxylic acid ethyl ester (3b). Colorless oil, IR (neat) 1749, 1721, 1619 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.26-7.24 (m, 4H), 6.38 (d, *J* = 15.6 Hz, 1H), 6.07 (dt, *J* = 15.6, 7.6 Hz, 1H), 4.16 (q, *J* = 7.6 Hz, 2H), 2.79 (dd, *J* = 14.4, 7.2 Hz, 1H), 2.53-2.40 (m, 3H), 2.30-2.24 (m, 1H), 2.07-1.88 (m, 3H), 1.26 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 214.1, 170.6, 135.3, 132.5, 128.7, 128.5, 128.2, 127.2, 125.3, 125.2, 61.4, 60.1, 37.9, 36.8, 32.3, 19.5, 14.1; HRMS calcd for $C_{17}H_{19}ClO_3$ (M^+) 306.1023, found 306.1024.

1-[3-(4-Methoxy-phenyl)-allyl]-2-oxo-cyclopentanecarboxylic acid ethyl ester (3c). Colorless oil; IR (neat) 1745, 1721, 1620 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.24 (d, *J* = 8.8 Hz, 2H), 6.81 (d, J = 8.8 Hz, 2H), 6.36 (d, *J* = 15.6 Hz, 1H), 5.91 (dt, *J* = 15.6, 7.6 Hz, 1H), 4.15 (2q, *J* = 7.2 Hz, 2H), 3.78 (s, 3H), 2.76 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 2.52 (m, 3H), 2.38-2.18 (m, 1H), 2.10-1.84 (m, 3H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 214.3, 170.7, 158.8, 133.2, 129.6, 127.1, 122.0, 113.7, 61.3, 60.2, 55.1, 37.9, 36.9, 32.1, 19.5, 14.0; HRMS calcd for $C_{18}H_{22}O_4$ (M^+) 302.1518, found 302.1514.

1-Oxo-2-(3-phenyl-allyl)-indan-2-carboxylic acid methyl ester (3f). red oil; IR (neat) 1744, 1720, 1622 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.70 (d, *J* = 7.6 Hz, 1H), 7.51 (dt, *J* = 8.0, 1.2 Hz, 1H),

7.38 (d, J = 8.0 Hz, 1H), 7.30 (t, J = 7.6 Hz, 1H), 7.10-7.19 (m, 5H), 6.39 (d, J = 16 Hz, 1H), 5.95 (dt, J = 16.0, 7.6 Hz, 1H), 3.61 (s, 3H), 3.58 (d, J = 17.4 Hz, 1H), 3.32 (d, J = 17.4 Hz, 1H), 2.90 (ddd, J = 8.4, 7.2, 1.2 Hz, 1H), 2.65 (ddd, J = 8.4, 7.2, 1.2 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 201.8, 171.0, 136.8, 135.6, 135.3, 134.9, 127.3, 126.4, 126.3, 126.0, 124.7, 124.6, 124.2, 60.3, 52.8, 38.3, 36.0; HRMS calcd for $C_{28}H_{18}O_3$ (M^+) 306.1256, found 306.1255.

2-[3-(4-Methoxy-phenyl)-allyl]-1-oxo-indan-2-carboxylic acid methyl ester (3g). yellow oil; IR (neat) 1744, 1720, 1622 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.76 (d, J = 7.6 Hz, 1H), 7.59 (dt, J = 8.0, 1.2 Hz, 1H), 7.43 (d, J = 8.0 Hz, 1H), 7.37 (t, J = 7.6 Hz, 1H), 7.14 (d, J = 9.6 Hz, 2H), 6.76 (d, J = 9.6 Hz, 2H), 6.39 (d, J = 16 Hz, 1H), 5.87 (dt, J = 16.0, 7.6 Hz, 1H), 3.76 (s, 3H), 3.69 (s, 3H), 3.64 (d, J = 17.6 Hz, 1H), 3.19 (d, J = 17.6 Hz, 1H), 3.01 (ddd, J = 8.4, 7.2, 1.2 Hz, 1H), 2.69 (ddd, J = 8.4, 7.2, 1.2 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 201.9, 171.0, 158.9, 135.3, 134.8, 129.5, 129.2, 127.2, 124.6, 121.8, 113.7, 60.4, 55.1, 52.7, 38.3, 35.9; HRMS calcd for $C_{21}H_{20}O_4$ (M^+) 336.1362, found 336.1364.

2-Acetyl-2-[3-(4-chloro-phenyl)-allyl]-3,4-dihydro-2H-naphthalen-1-one (3i). colorless oil; IR (neat) 1720, 1699, 1615 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.01 (d, J = 8.0 Hz, 1H), 7.56-7.54 (m, 1H), 7.43-7.10 (m, 6H), 6.32 (d, J = 15.6 Hz, 1H), 6.02 (dt, J = 15.6, 7.6 Hz, 1H), 3.01 (ddd, J = 15.6, 10.8, 4.8 Hz, 1H), 2.86-2.77 (m, 2H), 2.68 (ddd, J = 8.8, 7.2, 1.2 Hz, 1H), 2.52 (dt, J = 18.4, 4.8 Hz, 1H), 2.03 (s, 3H), 2.00-1.97 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 205.2, 196.9, 143.5, 143.1, 135.3, 133.8, 132.8, 131.8, 130.3, 128.8, 128.5, 128.3, 127.8, 127.3, 126.7, 125.3, 63.9, 38.2, 29.5, 27.1, 25.7; HRMS calcd for $C_{21}H_{19}ClO_2$ (M^+) 338.1074, found 338.1075.

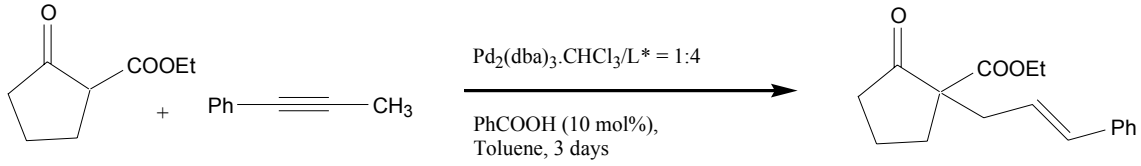
2-Acetyl-2-[3-(4-methoxy-phenyl)-allyl]-3,4-dihydro-2H-naphthalen-1-one (3j). Colorless oil, IR (neat) 1720, 1700, 1617 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.00 (dd, J = 8.4, 1.2 Hz, 1H), 7.40 (dt, J = 7.6, 1.2 Hz, 1H), 7.24 (t, J = 7.6 Hz, 1H), 7.18-7.17 (m, 1H), 7.13 (d, J = 7.6 Hz, 2H), 6.74 (d, J = 7.6 Hz, 2H), 6.62 (d, J = 15.6 Hz, 1H), 5.88 (dt, J = 15.6, 7.2 Hz, 1H), 3.71 (s, 3H), 3.05-2.96 (ddd, J = 15.6, 10.8, 4.8 Hz, 1H), 2.85-2.76 (m, 3H), 2.52 (dt, J = 18.4, 4.8 Hz, 1H), 2.08 (s, 3H), 2.07-1.95 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 205.4, 197.1, 158.9, 143.7, 133.8, 133.2, 131.9, 129.7, 128.8, 127.8, 127.3, 126.6, 122.1, 113.8, 64.1, 55.3, 38.4, 29.4, 27.1, 25.8; HRMS calcd for $C_{22}H_{22}O_3$ (M^+) 334.1569, found 334.1570.

4-(2-Acetyl-1-oxo-1,2,3,4-tetrahydro-naphthalen-2-yl)-but-2-enoic acid ethyl ester (3k). Yellow oil; IR (neat) 1723, 1699, 1618 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 8.10 (d, J = 7.6 Hz, 1H), 7.61-6.84 (m, 3H), 6.80 (dt J = 15.2, 7.6 Hz, 1H), 5.87 (d, J = 15.2 Hz, 1H), 4.14 (q, J = 7.2 Hz, 2H), 3.06 (ddd, J = 16.8, 10.4, 4.8 Hz, 1H), 2.91-2.82 (m, 2H), 2.71 (dd, J = 14.4, 7.6 Hz, 1H), 2.55 (dt, J = 9.6, 4.8 Hz, 1H), 2.11 (s, 3H), 2.05-1.95 (m, 1H), 1.25 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 204.6, 196.4, 171.5, 165.7, 143.4, 142.8, 134.0, 133.6, 131.6, 130.0, 128.3, 63.3, 60.4, 36.9, 29.5, 27.1, 25.6, 14.2; HRMS calcd for $C_{18}H_{20}O_4$ (M^+) 300.1362, found 300.1364.

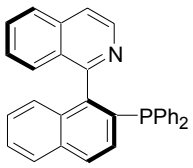
4-(1-Acetyl-2-oxo-cyclohexyl)-but-2-enoic acid ethyl ester (3o). Colorless oil; IR (neat) 1725, 1717, 1698, 1625 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 6.69 (dt, *J* = 16.0, 8.0 Hz, 1H), 5.79 (d, *J* = 16.0 Hz, 1H), 4.12 (q, *J* = 6.8 Hz, 2H), 2.63 (dd, *J* = 15.4, 8.4 Hz, 1H), 2.55 (dd. *J* = 15.4, 7.6 Hz, 1H), 2.50-2.43 (m, 2H), 2.30-2.19 (m, 1H), 2.07 (s, 3H), 1.97-1.91 (m, 1H), 1.73-1.62 (m, 3H), 1.48-1.40 (m, 1H), 1.24 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 208.6, 205.0, 165.6, 142.6, 124.8, 67.2, 60.4, 41.6, 36.8, 34.1, 27.0, 26.3, 22.1, 14.2; HRMS calcd for $C_{14}H_{20}O_4$ (M^+) 252.1362, found 252.1362.

Typical procedure for the enantioselective allylation of 1a with 2a. To a solution of **1a** (15 mg, 0.096 mmol) in toluene (0.02 mL) were added 1-phenyl-1-propyne **2a** (11 mg, 0.096 mmol), $Pd_2dba_3.CHCl_3$ (5 mg, 0.005 mmol), (S,S)-CHIRAPHOS (8 mg, 0.0192 mmol) and PhCOOH (1 mg, 0.009 mmol). The mixture was stirred at 100 °C for 3 days in screw capped vial. The solvent was removed under reduced pressure, and the residue was purified by short silica gel column with hexane:ethyl acetate, 9:1 to give **3a** (16 mg) in 63 % yield. The ratio of enantiomers was determined to be 63.73/36.27 by HPLC analysis (hexane/*i*-PrOH = 100/1, flow rate = 1.0 mL/min): t_R = 14.79 min (major enantiomer), t_R = 16.78 min (minor enantiomer); 27% ee.
Column Specification: Daicel CHIRALCEL OD column (4.6 mm x 250 mm).

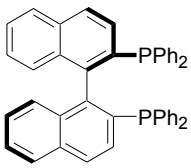
TABLE 1. Assymetric allylation of cyclic β-ketoester **1a** with alkyne **2a**.



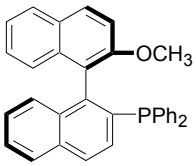
Entry	L*	Yield (%)	Ee (%)
1	(S)-QUINAP (**1**)	71	3
2	(R)-BINAP (**2**)	58	11
3	(R)-MOP (**3**)	Quant.	0
4	Trost Ligand **(4)**	95	0
5	(R, R)-Me-BPE (**5**)	No Reaction	-
6	(R, R)-Me-DUPHOS (**6**)	No Reaction	-
7	(S, S)-Me-FerroTane (**7**)	48	0
8	(S)-PHANEPHOS (**8**)	88	6
9	(R, R)-NORPHOS (**9**)	No Reaction	-
10	(R, R)-RENORPHOS (**10**)	67	7
11	(S, S)-CHIRAPHOS (**11**)	63	27
12	(S, S)-BDPP (**12**)	67	20
13	(R)-PROPHOS (**13**)	51	3
14	(S, S)-DIOP (**14**)	Quant.	4
15	(S, S)-BPPM (**15**)	98	9
16	(+) NMDPP (**16**)	57	2
17	(R)-(S)-JOSIPHOS (**17**)	80	0
18	(R)-(S)-BPPFOAc (**18**)	78	4
19	(**19**)	89	0
20	(**20**)	86	0
21	(**21**)	Trace	nd



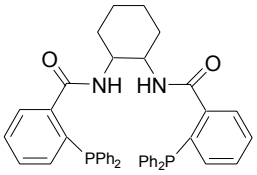
(S)-QUINAP (**1**)



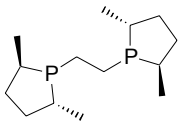
(R)-BINAP (**2**)



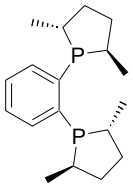
(R)-MOP (**3**)



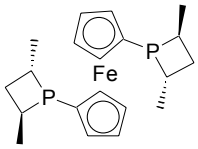
Trost Ligand (**4**)



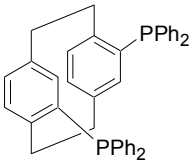
(R, R)-Me-BPE (**5**)



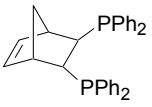
(R, R)-Me-DUPHOS (**6**)



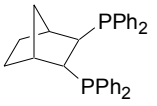
(S, S)-Me-FerroTane (**7**)



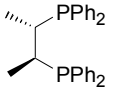
(S)-PHANEPHOS (**8**)



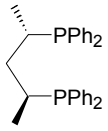
(R, R)-NORPHOS (**9**)



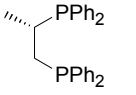
(R, R)-RENORPHOS (**10**)



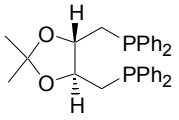
(S, S)-CHIRAPHOS (**11**)



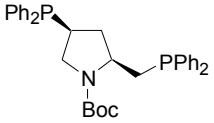
(S, S)-BDPP (**12**)



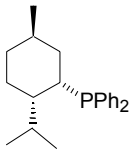
(R)-PROPHOS (**13**)



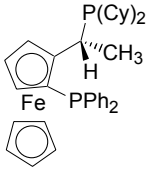
(S, S)-DIOP (**14**)



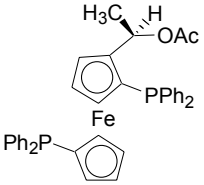
(S, S)-BPPM (**15**)



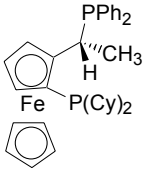
(+) NMDPP (**16**)



(R)-(S)-JOSIPHOS (**17**)



(R)-(S)-BPPFOAc (**18**)



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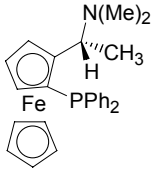

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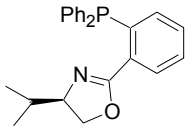

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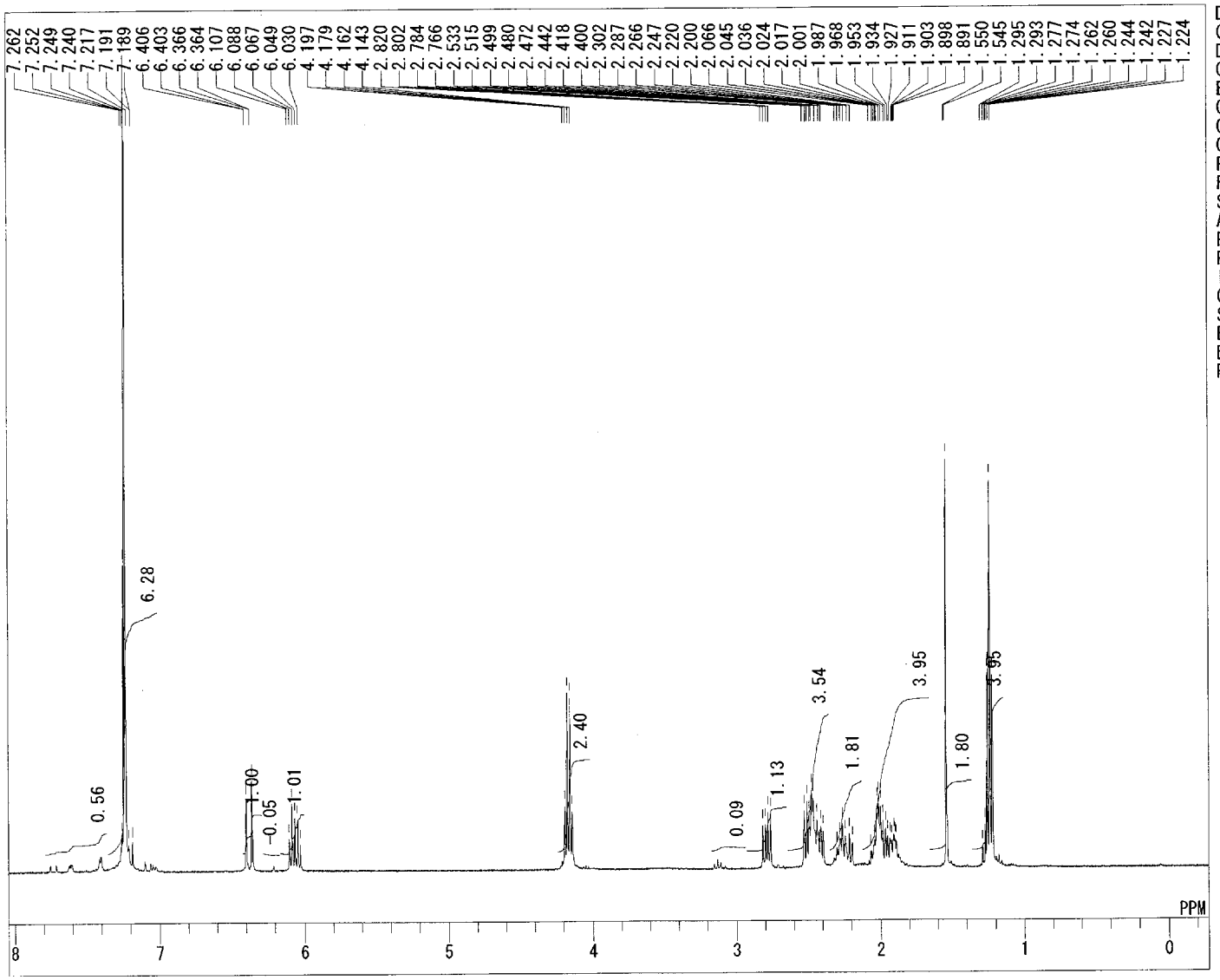

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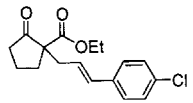

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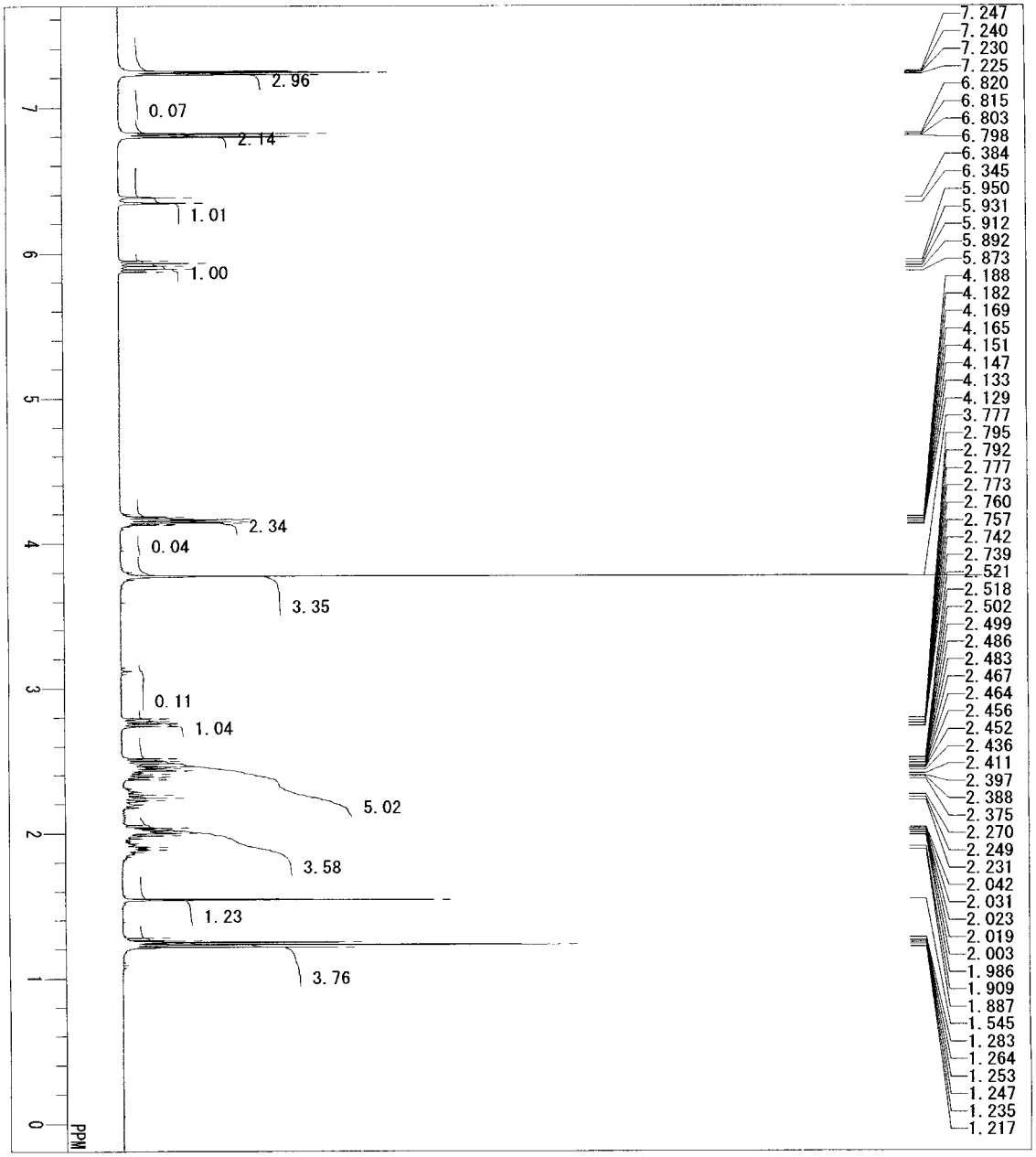
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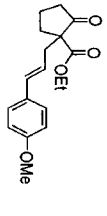
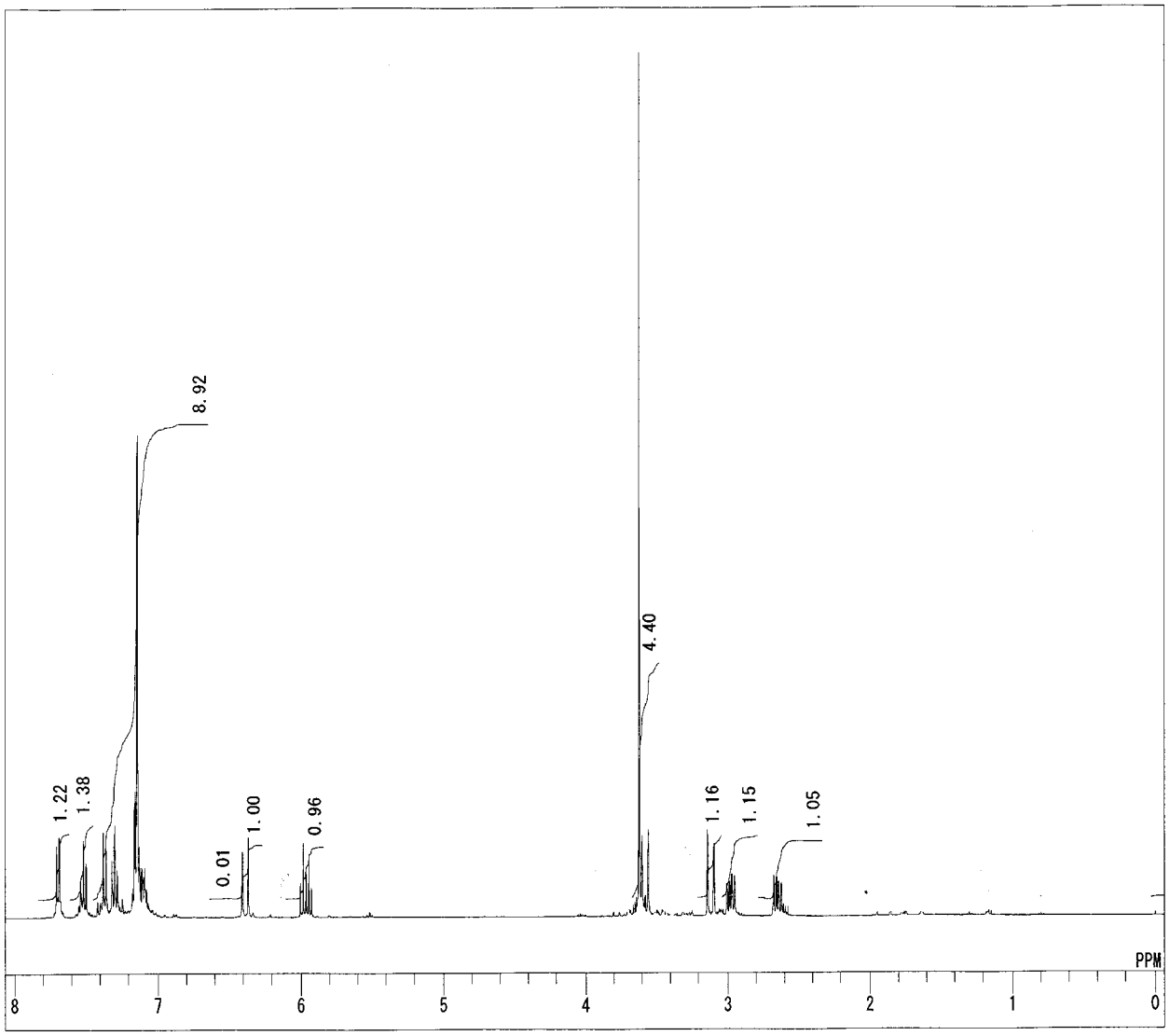
5.912
5.892
5.873
4.188
4.182
4.169
4.165
4.151
4.147
4.133
4.129
3.777
2.795
2.792
2.777
2.773
2.760
2.757
2.742
2.739
2.521
2.518
2.502
2.499
2.486
2.483
2.467
2.464
2.456
2.452
2.436
2.411
2.397
2.388
2.375
2.270
2.249
2.231
2.042
2.031
2.023
2.019
2.003
1.986
1.909
1.887
1.545
1.283
1.264
1.253
1.247
1.235
1.217
2.96
0.07
2.14
1.01
1.00
2.34
0.04
3.35
0.11
1.04
5.02
3.58
1.23
3.76
PPM
DFILE C:¥WINNMR98¥COMMON¥
COMNT
DATIM Tue Apr 27 17:12:28
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 8000.0 Hz
SCANS 8
ACQTM 4.096 sec
PD 2.904 sec
PW1 5.8 us
IRNUC 1H
CTEMP 25.9 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.12 Hz
RGAIN 16

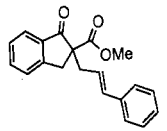

O
O
OEt
OMe

3c

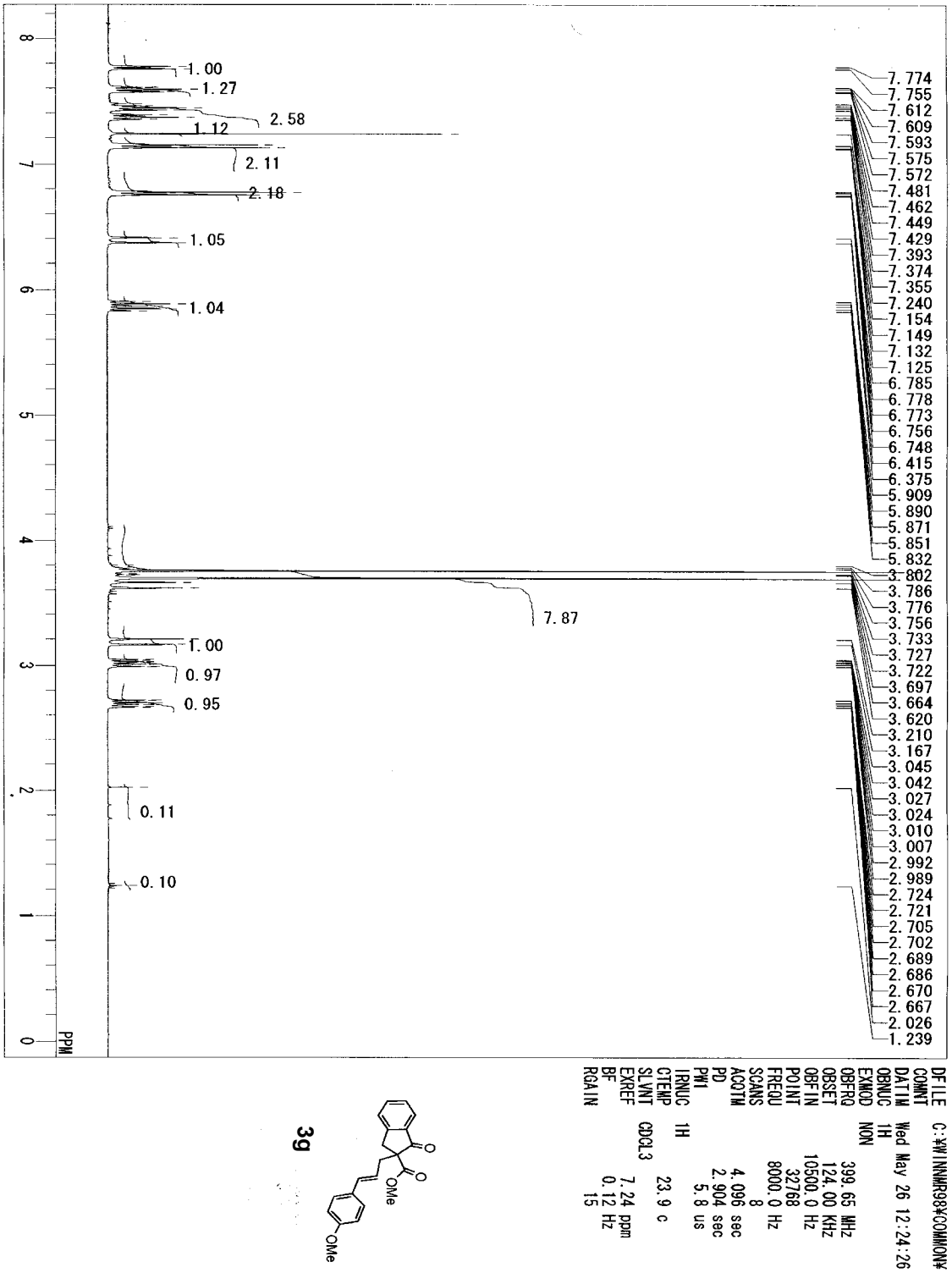
C:¥共有フォルダ¥yukin¥Nitin¥extension¥NTP 300 (conc) PMR.als



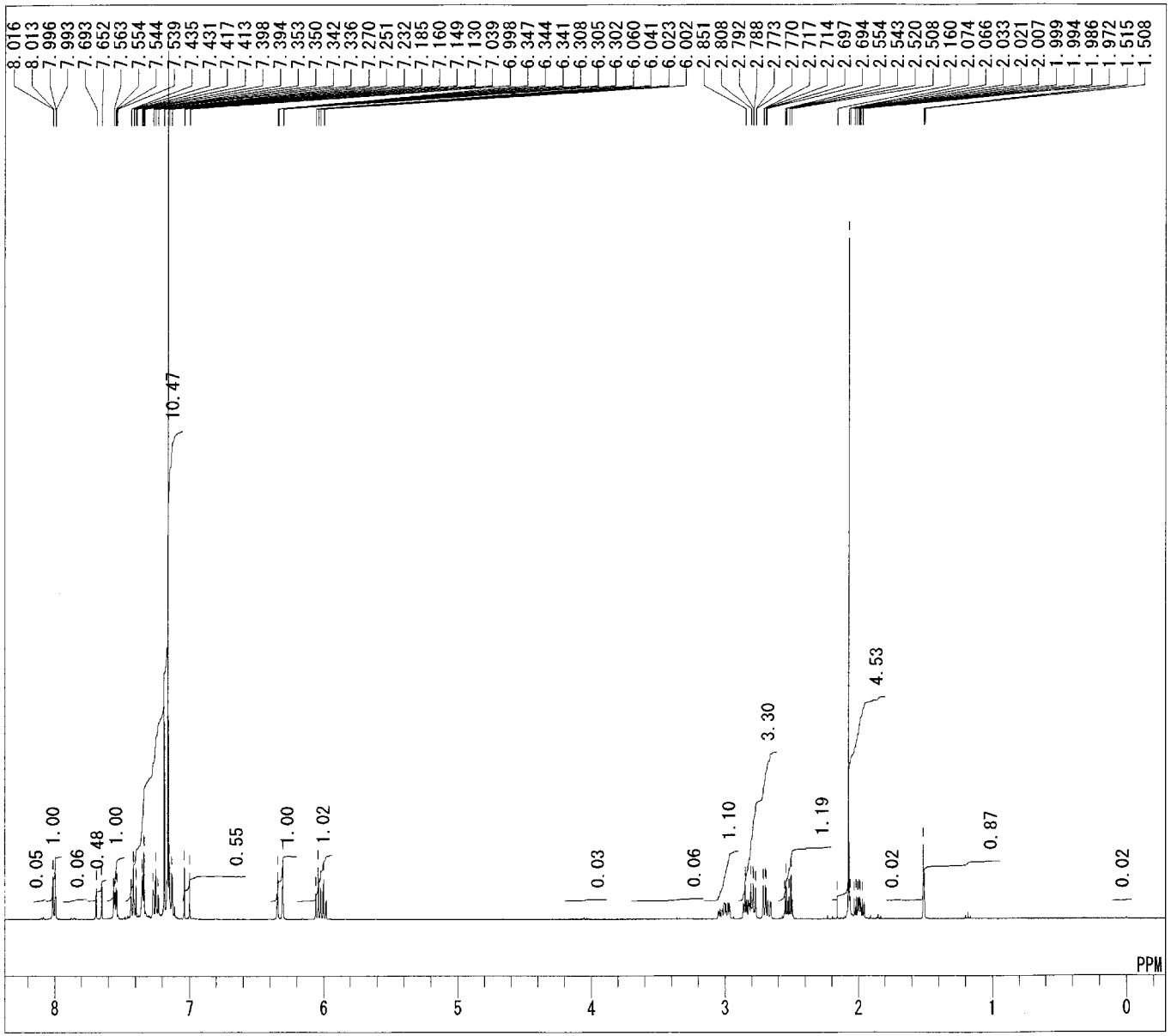
DFILE	C:¥共有フォルダ¥yuk
COMNT	
DATIM	Tue May 25 14:08:01
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.0 Hz
POINT	32768
FREQU	8000.0 Hz
SCANS	8
ACQTM	4.096 sec
PD	2.904 sec
PW1	5.8 us
IRNUC	1H
CTEMP	23.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	9



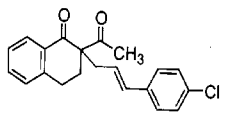
3f


C:¥WINNMR98¥COMMON¥_DEFAULT.ALS
PPM
1.00
1.27
2.58
1.12
2.11
2.18
1.05
1.04
7.87
1.00
0.97
0.95
0.11
0.10
7.774
7.755
7.612
7.609
7.593
7.575
7.572
7.481
7.462
7.449
7.429
7.393
7.374
7.355
7.240
7.154
7.149
7.132
7.125
6.785
6.778
6.773
6.756
6.748
6.415
6.375
5.909
5.890
5.871
5.851
5.832
3.802
3.786
3.776
3.756
3.733
3.727
3.722
3.697
3.664
3.620
3.210
3.167
3.045
3.042
3.027
3.024
3.010
3.007
2.992
2.989
2.724
2.721
2.705
2.702
2.689
2.686
2.670
2.667
2.026
1.239
DFILE C:¥WINNMR98¥COMMON¥
COMNT 1H
DATIM Wed May 26 12:24:26
OBNUC
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 8000.0 Hz
SCANS 8
ACQTM 4.096 sec
PD 2.904 sec
PW1 5.8 us
IRNUC 1H
CTEMP 23.9 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.12 Hz
RGAIN 15
3g
OMe
OMe

C:¥WINNMR98¥COMMON¥_DEFAULT.ALS

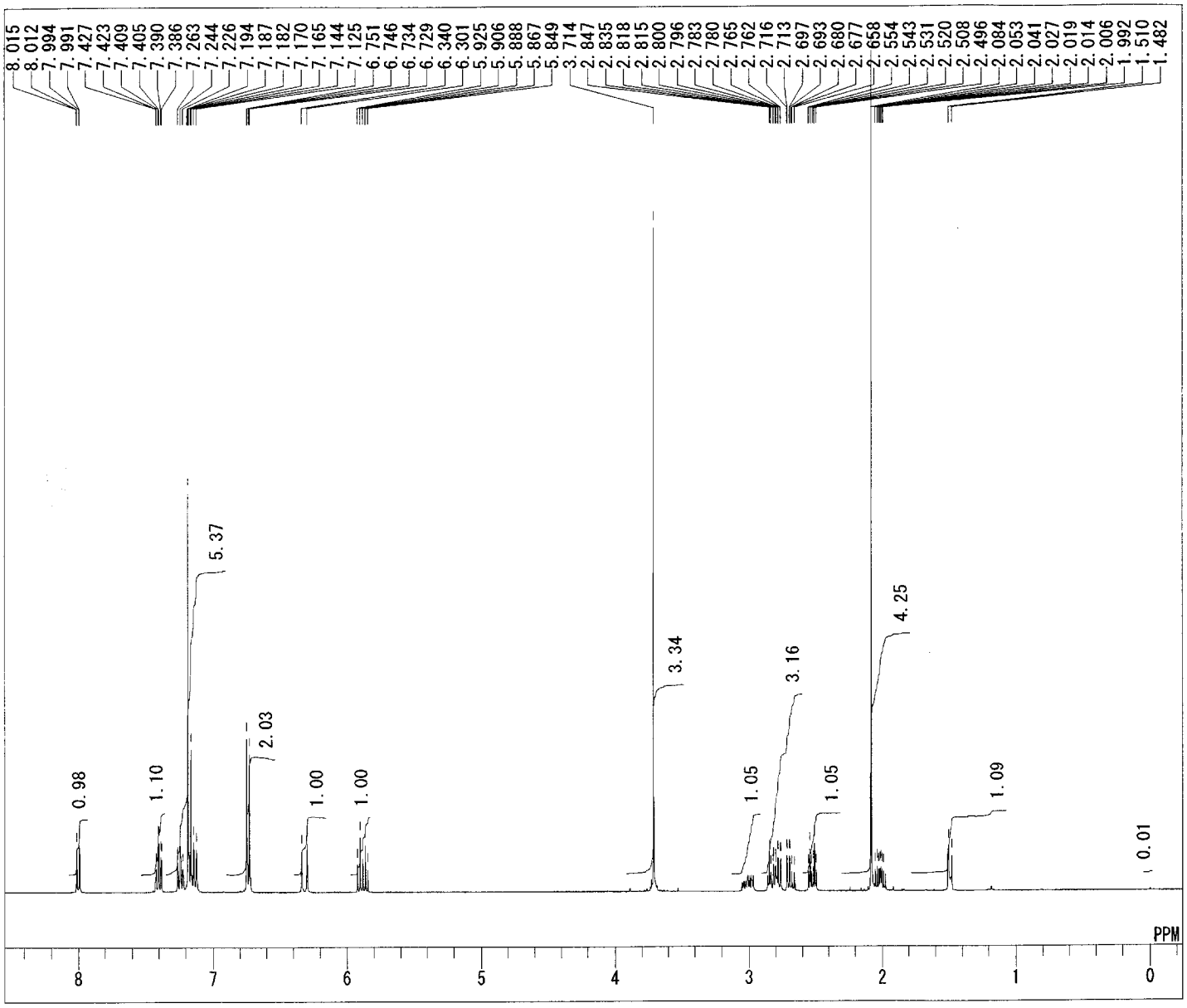


DFILE C:¥WINNMR98¥COMMON¥
COMNT
DATIM Wed Apr 28 13:22:02
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 8000.0 Hz
SCANS 8
ACQTM 4.096 sec
PD 2.904 sec
PW1 5.4 us
IRNUC 1H
CTEMP 25.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 13

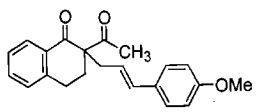


3i

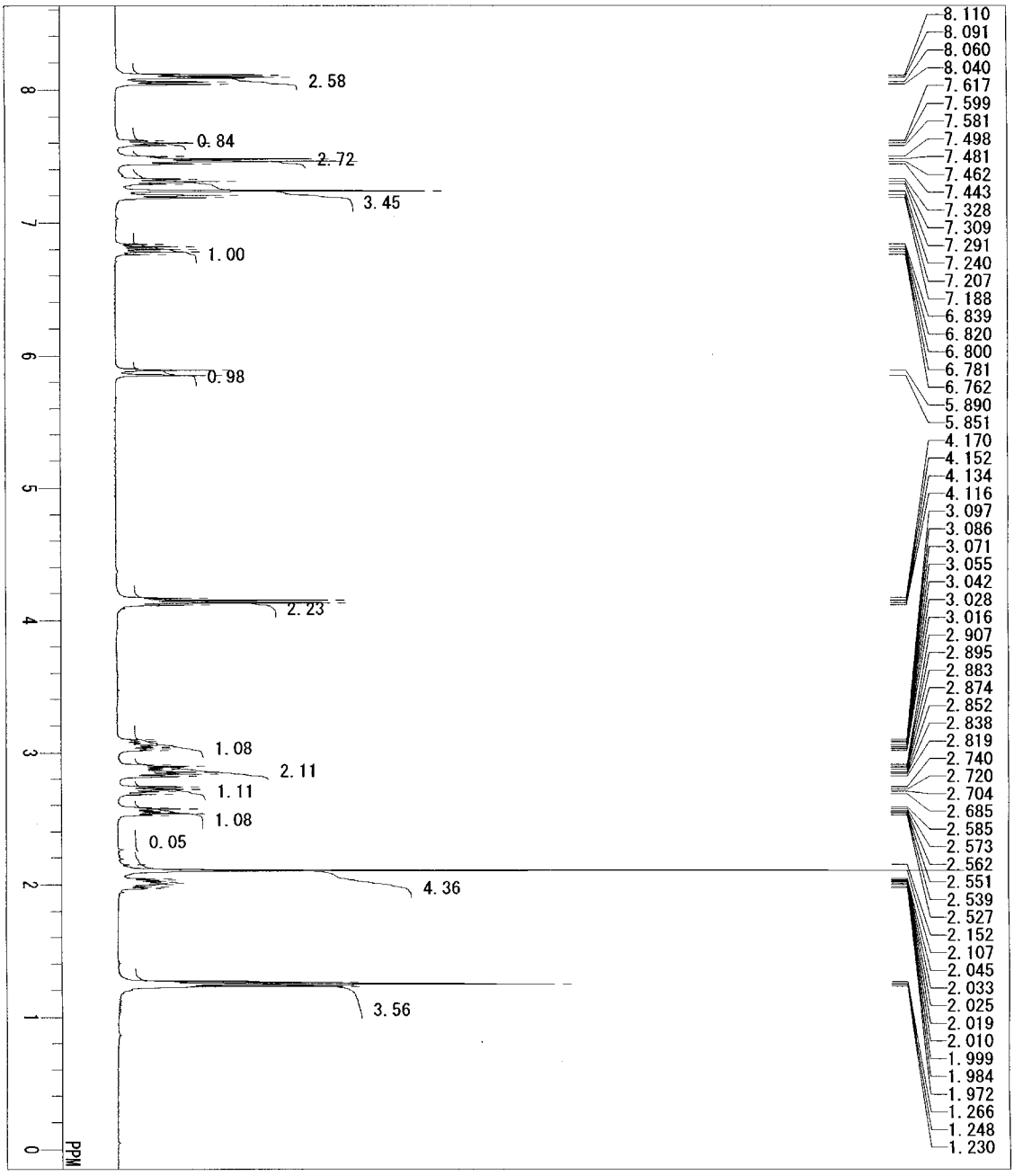
C:¥WINNMR98¥COMMON¥_DEFAULT.ALS

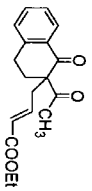


DFILE C:¥WINNMR98¥COMMON¥
COMNT
DATIM Wed Apr 28 17:03:21
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 8000.0 Hz
SCANS 8
ACQTM 4.096 sec
PD 2.904 sec
PW1 5.8 us
IRNUC 1H
CTEMP 26.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 15

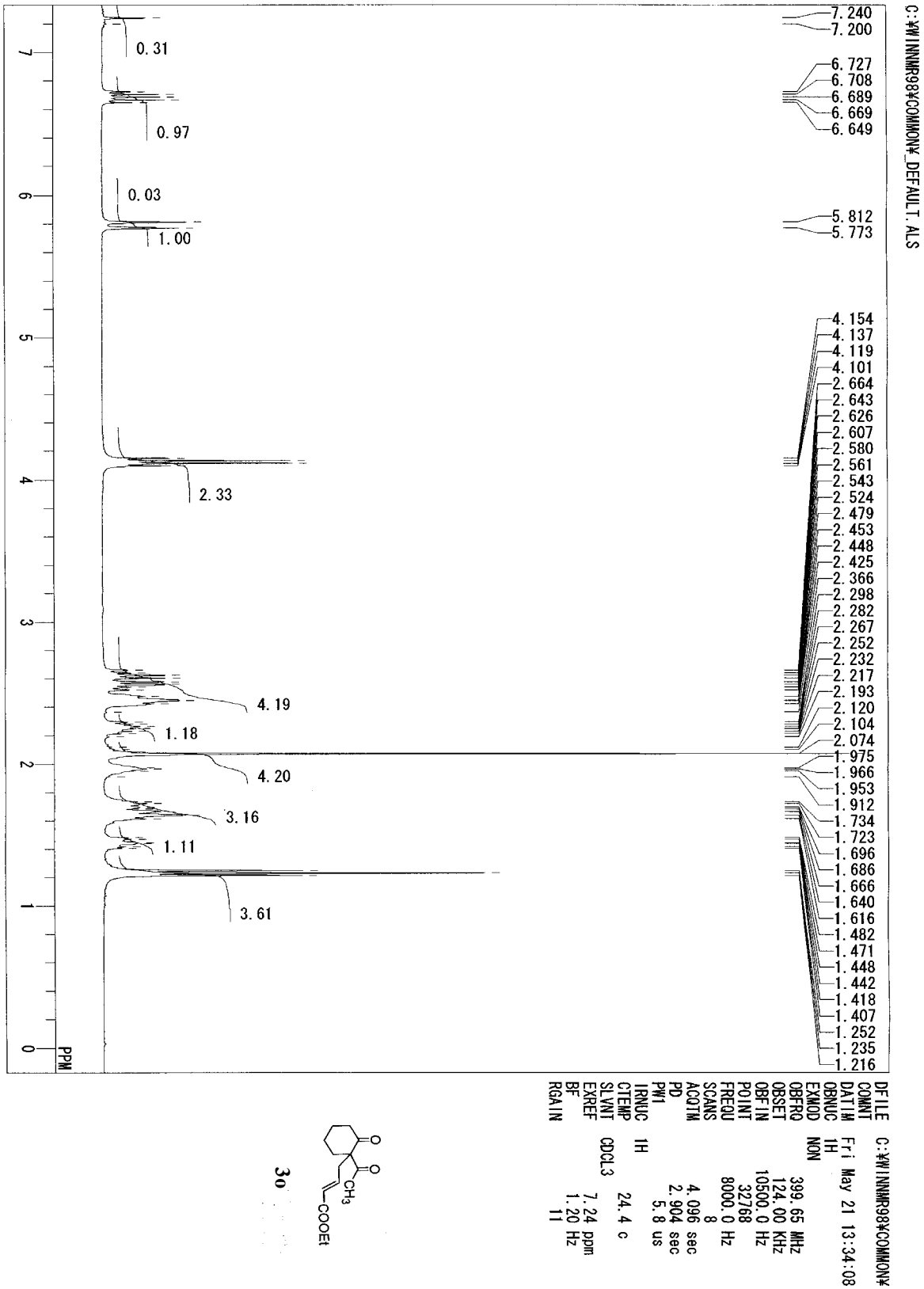


3j


C:¥WINNMR98¥COMMON¥_DEFAULT.ALS
8.110
8.091
8.060
8.040
7.617
7.599
7.581
7.498
7.481
7.462
7.443
7.328
7.309
7.291
7.240
7.207
7.188
6.839
6.820
6.800
6.781
6.762
5.890
5.851
4.170
4.152
4.134
4.116
3.097
3.086
3.071
3.055
3.042
3.028
3.016
2.907
2.895
2.883
2.874
2.852
2.838
2.819
2.740
2.720
2.704
2.685
2.585
2.573
2.562
2.551
2.539
2.527
2.152
2.107
2.045
2.033
2.025
2.019
2.010
1.999
1.984
1.972
1.266
1.248
1.230
2.58
0.84
2.72
3.45
1.00
0.98
2.23
1.08
2.11
1.11
1.08
0.05
4.36
3.56
PPM
DFILE C:¥WINNMR98¥COMMON¥
COMNT
DATIM Fri May 21 12:05:59
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 8000.0 Hz
SCANS 8
ACQTM 4.096 sec
PD 2.904 sec
PW1 5.8 us
IRNUC 1H
CTEMP 24.9 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 1.20 Hz
RGAIN 16


O
O
CH3
COOEt

3k


C:¥WINNMR98¥COMMON¥_DEFAULT.ALS
7.240
7.200
6.727
6.708
6.689
6.669
6.649
5.812
5.773
4.154
4.137
4.119
4.101
2.664
2.643
2.626
2.607
2.580
2.561
2.543
2.524
2.479
2.453
2.448
2.425
2.366
2.298
2.282
2.267
2.252
2.232
2.217
2.193
2.120
2.104
2.074
1.975
1.966
1.953
1.912
1.734
1.723
1.696
1.686
1.666
1.640
1.616
1.482
1.471
1.448
1.442
1.418
1.407
1.252
1.235
1.216
0.31
0.97
0.03
1.00
2.33
4.19
1.18
4.20
3.16
1.11
3.61
PPM
0
1
2
3
4
5
6
7
DFILE C:¥WINNMR98¥COMMON¥
COMNT
DATIM Fri May 21 13:34:08
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 8000.0 Hz
SCANS 8
ACQTM 4.096 sec
PD 2.904 sec
PW1 5.8 us
IRNUC 1H
CTEMP 24.4 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 1.20 Hz
RGAIN 11
O
O
CH3
COOEt

3o