Supporting Information

Cooperative Catalytic Reactions Using Organocatalysts and Transition Metal Catalysts: Enantioselective Propargylic Alkylation of Propargylic Esters with Aldehydes

Akiko Yoshida, Masahiro Ikeda, Gaku Hattori, Yoshihiro Miyake, and Yoshiaki Nishibayashi*

Institute of Engineering Innovation, School of Engineering, The University of Tokyo, Yayoi, Bunkyo-ku, Tokyo 113-8656, Japan

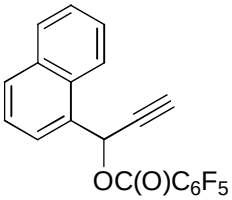
General Methods. ^{1}H NMR (270 MHz) and ^{13}C NMR (67.8 MHz) spectra were measured on a JEOL Excalibur 270 spectrometer using $CDCl_3$ as solvent. HPLC analyses were performed on Hitachi L-7100 and GL-7410 apparatuses equipped with a UV detector using 25 cm x 4.6 mm DAICEL Chiralpak AS-H column. Elemental analyses were performed at Microanalytical Center of The University of Tokyo. Mass spectra were measured on a JEOL JMS-700 mass spectrometer.

All reactions were carried out under a dry nitrogen atmosphere. Solvents were dried by the usual methods, then distilled under N_2 and degassed before use. Aldehydes (**2a** and **2d**), diphosphines, and optically pure secondary amines (**3**) are commercially available reagents. Aldehyde (**2c**) was prepared by PCC oxidation of the corresponding alcohol. Aldehydes (**2b**[S1] and **2e**[S2]) were synthesized according to literature.

General Procedure for the Preparation of Propargylic Esters. A typical experimental procedure for the preparation of 1-(1-naphthyl)-2-propynyl pentafluorobenzoate **(1a)** is described below. In a 100 mL Schlenk flask were placed 1-naphthaldehyde (2.34g, 15.0 mmol) and anhydrous diethyl ether (15 mL). After cooling the reaction flask to 0 ºC, ethynylmagnesium bromide (0.5 M in tetrahydrofuran; 33.0 mL, 16.5 mmol) was added to the solution. Then, the mixture was stirred at room temperature for 1.5 h. The reaction was quenched by saturated NH_4Cl solution (30 mL), and organic materials were extracted with diethyl ether (15 mL x 2). The combined extracts were washed with brine, and dried over anhydrous $MgSO_4$. After the concentration under reduced pressure, the resulting residue was purified by column chromatography (SiO_2, eluent: hexane/ethyl acetete, 90/10) to give 1-(1-naphthyl)-2-propyn-1-ol as a white solid (2.47 g, 13.6 mmol, 92 % isolated yield).

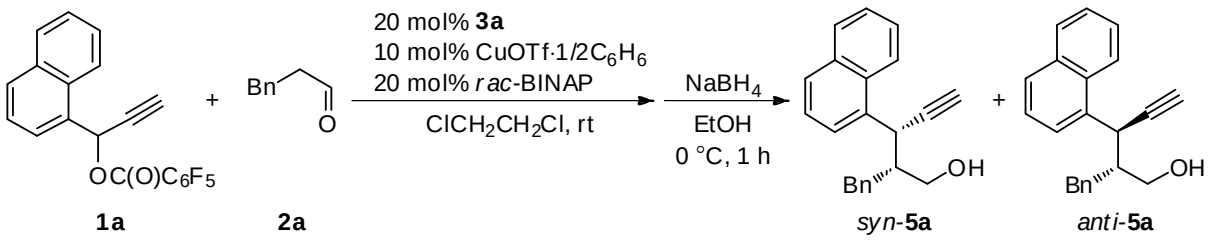
In a 200 mL round-bottomed flask were placed 1-(1-naphthyl)-2-propyn-1-ol (1.82 g, 10.0 mmol), triethylamine (1.21 g, 12.0mmol), and anhydrous dichloromethane (50 mL). After cooling the reaction flask to 0 ºC, pentafluorobenzoyl chloride (2.54 g, 11.0 mmol) was added to the solution. Then, the mixture was stirred at room temperature for 1 h. The reaction was quenched by water (30 mL), and organic materials were extracted with dichloromethane (15 mL x 3). The combined extracts were washed with brine, and dried over anhydrous Na_2SO_4. The solvent was concentrated under reduced pressure and the

resulting residue was purified by column chromatography (SiO_2, eluent: hexane/ethyl acetete, 90/10 to 70/30) to give 1-(1-naphthyl)-2-propynyl pentafluorobenzoate (**1a**) as a white solid (3.32 g, 8.83 mmol, 88 % isolated yield).



1-(1-naphthyl)-2-propynyl 2,3,4,5,6-pentafluorobenzoate (1a): A white solid, mp 102.4-102.9 ºC. ^{1}H NMR 8.23 (d, *J* = 8.4 Hz, 1H), 7.87-7.93 (m, 3H), 7.47-7.63 (m, 3H), 7.27 (d, *J* = 2.3 Hz, 1H), 2.82 (d, *J* = 2.3 Hz, 1H). ^{13}C NMR δ 158.0, 145.6 (md, *J* = 251Hz), 143.5 (md, *J* = 251Hz), 137.7 (md, *J* = 251Hz), 134.0, 130.6, 130.4, 130.3, 128.9, 127.1, 126.9, 126.2, 125.1, 123.5, 107.5 (m), 78.9, 77.5, 66.4. Anal. Calcd for: $C_{20}H_9F_5O_2$: C, 63.84; H, 2.41. Found: C, 63.75; H, 2.66.

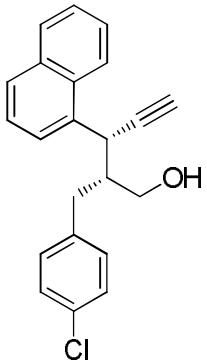
Enantioselective Propargylic Alkylations of Propargylic Esters with Aldehydes.



1a **2a** *syn*-**5a** *anti*-**5a**

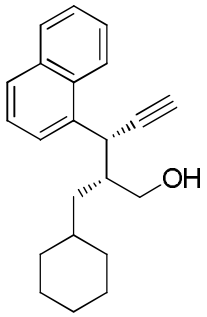
A typical experimental procedure for the reaction of 1-(1-naphthyl)-2-propynyl pentafluorobenzoate (**1a**) with 3-phenylpropanal (**2a**) is described below. In a 20 mL Schlenk flask were placed $CuOTf·1/2C_6H_6$ (5.0 mg, 0.020 mmol) and *rac*-BINAP (24.9 mg, 0.040 mmol) under N_2. After anhydrous 1,2-dichloroethane (2.0 mL) was added, the mixture was magnetically stirred at 60 ºC for 1 h. After cooling the reaction flask to room temperature, (*S*)-α,α-bis[3,5-bis(trifluoromethyl)phenyl]-2-pyrrolidinemethanol trimethylsilyl ether (**3a**) (23.9 mg, 0.040 mmol) and **2a** (53.7 mg, 0.400 mmol) were added to the reaction mixture, and then **1a** (75.3 mg, 0.200 mmol) and anhydrous 1,2-dichloroethane (3.0 mL) were added successively under N_2. The reaction flask was kept at room temperature for 1.5 h. After cooling the reaction flask to 0 ºC, ethanol (5.0 ml) and $NaBH_4$ (22.7 mg, 0.600 mmol) were added, and then the mixture was magnetically stirred at 0 ºC for 1 h. The reaction was quenched by water (15 mL), and the resulting mixture was extracted with dichloromethane (10 mL x 3). The combined organic layer was washed with brine, and was dried over anhydrous Na_2SO_4. After the concentration under reduced pressure, the resulting residue was purified by column chromatography (SiO_2, eluent: hexane/ethyl acetete, 95/5 to 85/15) to give 2-benzyl-3-(1-naphthyl)- 4-pentyn-1-ol[S3] (**5a**) as a pale yellow oil (31.9 mg, 0.106 mmol, 53% isolated yield, *syn*-**5a**/*anti*-**5a** = 3.2/1). The optical purity of **5a** was determined by HPLC analysis; Daicel Chiralpak AS-H, hexane/^{i}PrOH = 95/5, flow rate = 0.5 mL/min, λ = 254 nm, retention time; 23.6 min (*syn*-major) and 77.5 min (*syn*-minor), 98% *ee* (*syn*); 27.2 min (*anti*-minor) and 47.5 min (*anti*-major), 96% *ee* (*anti*).

Spectroscopic Data and Isolated Yield of Other Products.



syn-**5b**

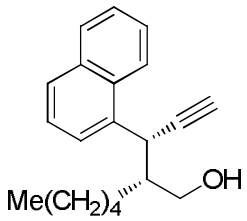
2-(4-Chlorobenzyl)-3-(1-naphtyl)-4-butyn-1-ol (5b): Isolated yield 54% (*syn*-**5b**/*anti*-**5b** = 3.8/1). A pale yellow oil. *syn*-isomer: ^{1}H NMR δ 8.16 (d, *J* = 8.1 Hz, 1H), 7.79-7.92 (m, 3H), 7.45-7.57 (m, 3H), 7.09 (d, *J* = 8.4 Hz, 2H), 6.80 (d, *J* = 8.4 Hz, 2H), 5.08 (dd, *J* = 4.3 and 2.6 Hz, 1H), 3.70 (dd, *J* = 10.8 and 8.4 Hz, 1H), 3.57 (dd, *J* = 10.0 and 3.8 Hz, 1H), 2.79 (dd, *J* = 13.8 and 3.8 Hz, 1H), 2.62 (dd, *J* = 13.8 and 10.8 Hz, 1H), 2.44 (d, *J* = 2.6 Hz, 1H), 2.30-2.42 (m, 1H). *anti*-isomer: ^{1}H NMR δ 7.75-7.92 (m, 4H), 7.28-7.57 (m, 7H), 4.50 (dd, *J* = 4.3 and 2.6 Hz, 1H), 3.80 (dd, *J* = 11.2 and 5.8 Hz, 1H), 3.63 (dd, *J* = 11.2 and 4.2 Hz, 1H), 3.08 (dd, *J* = 13.6 and 5.9 Hz, 1H), 2.80 (dd, *J* = 13.6 and 8.8 Hz, 1H), 2.48 (d, *J* = 2.6 Hz, 1H), 2.20-2.28 (m, 1H). *syn*-isomer: ^{13}C NMR δ 138.5, 134.93, 134.1, 131.7, 130.6, 130.3, 128.7, 128.3, 127.9, 126.6, 126.4, 125.64, 125.2, 123.0, 83.4, 73.3, 63.0, 47.0, 35.0, 32.6. *anti*-isomer: ^{13}C NMR δ 138.6, 134.86, 134.0, 132.3, 130.8, 130.4, 129.1, 128.1, 126.2, 126.1, 125.61, 122.4, 83.5, 73.7, 62.7, 47.4, 35.8, 34.6. HRMS (EI) Calcd for $C_{22}H_{19}ClO$ [M]: 334.1124. Found: 334.1132. The optical purity of *syn*-**5b** was determined by HPLC analysis; Daicel Chiralpak AS-H, hexane/^{i}PrOH = 95/5, flow rate = 0.5 mL/min, λ = 254 nm, retention time; 20.0 min (major) and 54.8 min (minor), 99% *ee*. The optical purity of *anti*-**5b** was determined by HPLC analysis; Daicel Chiralpak AS-H, hexane/^{i}PrOH = 95/5, flow rate = 0.5 mL/min, λ = 254 nm, retention time; 22.8 min (minor) and 36.0 min (major), 97% *ee*.



syn-**5c**

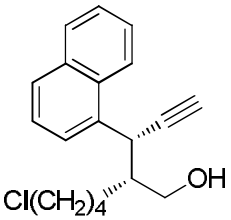
2-(Cyclohexylmethyl)-3-(1-naphtyl)-4-butyn-1-ol (5c): Isolated yield 52% (*syn*-**5c**/*anti*-**5c** = 3.2/1). A pale yellow oil. *syn*-isomer: ^{1}H NMR δ 8.16 (d, *J* = 8.4 Hz, 1H), 7.75-7.90 (m, 3H), 7.43-7.56 (m, 3H), 5.03 (dd, *J* = 3.5 and 2.6 Hz, 1H), 3.74-3.83 (m, 2H), 2.33

(d, *J* = 2.6 Hz, 1H), 2.12-2.23 (m, 1H), 0.66-1.79 (m, 12H), 0.17-0.32 (m, 1H). *anti*-isomer: ^{1}H NMR δ 7.99 (d, *J* = 8.1 Hz, 1H), 4.56 (dd, *J* = 5.1 and 2.6 Hz, 1H), 3.65 (dd, *J* = 11.3 and 4.6 Hz, 1H), 2.40 (d, *J* = 2.6 Hz, 1H). *syn*-isomer: ^{13}C NMR δ 135.3, 134.0, 130.6, 128.9, 127.6, 126.4, 126.11, 125.4, 125.1, 123.1, 83.8, 72.7, 64.7, 42.0, 36.7, 35.2, 34.4, 34.3, 32.1, 26.4, 26.2, 25.9. *anti*-isomer: ^{13}C NMR δ 135.5, 134.1, 130.7, 129.2, 127.9, 126.2, 126.07, 125.6, 125.3, 122.8, 84.6, 73.0, 63.3, 41.9, 38.0, 35.0, 34.1, 33.3, 26.6, 26.3. HRMS (EI) Calcd for $C_{22}H_{26}O$ [M]: 306.1984. Found: 306.1987. The optical purity of *syn*-**5c** was determined by HPLC analysis; Daicel Chiralpak AS-H, hexane/^{i}PrOH = 98/2, flow rate = 0.5 mL/min, λ = 254 nm, retention time; 20.1 min (major) and 32.7 min (minor), 97% *ee*. The optical purity of *anti*-**5c** was determined by HPLC analysis; Daicel Chiralpak AS-H, hexane/^{i}PrOH = 98/2, flow rate = 0.5 mL/min, λ = 254 nm, retention time; 18.6 min (minor) and 29.5 min (major), 98% *ee*.



syn-**5d**

2-[1-(1-naphtyl)-2-propynyl]heptan-1-ol (5d): Isolated yield 58% (*syn*-**5d**/*anti*-**5d** = 3.5/1). A pale yellow oil. *syn*-isomer: ^{1}H NMR δ 8.16 (d, *J* = 8.4 Hz, 1H), 7.76-7.90 (m, 3H), 7.44-7.56 (m, 3H), 5.01 (dd, *J* = 3.8 and 2.7 Hz, 1H), 3.64-3.81 (m, 2H), 2.34 (d, *J* = 3.0 Hz, 1H), 2.02-2.13 (m, 1H), 0.97-1.72 (m, 8H), 0.73 (t, *J* = 6.9 Hz, 3H). *anti*-isomer: ^{1}H NMR δ 8.01 (d, *J* = 8.1 Hz, 1H), 4.62 (dd, *J* = 5.1 and 2.7 Hz, 1H), 2.39 (d, *J* = 2.7 Hz, 1H), 0.89 (t, *J* = 6.6 Hz, 3H). *syn*-isomer: ^{13}C NMR δ 135.4, 134.0, 130.66, 129.0, 127.6, 126.5, 126.1, 125.5, 125.2, 123.1, 83.9, 72.7, 64.2, 45.00, 35.2, 31.9, 27.1, 26.6, 22.4, 13.9. *anti*-isomer: ^{13}C NMR δ 135.5, 134.1, 130.69, 129.2, 128.0, 126.2, 125.6, 125.3, 122.8, 84.4, 73.0, 63.2, 44.96, 36.1, 32.0, 30.1, 26.9, 22.6, 14.0. HRMS (EI) Calcd for $C_{20}H_{24}O$ [M]: 280.1827. Found: 280.1823. The optical purity of *syn*-**5d** was determined by HPLC analysis; Daicel Chiralpak AS-H, hexane/^{i}PrOH = 98/2, flow rate = 0.5 mL/min, λ = 254 nm, retention time; 21.1 min (minor) and 25.2 min (major), 83% *ee*. The optical purity of *anti*-**5d** was determined by HPLC analysis; Daicel Chiralpak AS-H, hexane/^{i}PrOH = 98/2, flow rate = 0.5 mL/min, λ = 254 nm, retention time; 18.8 min (minor) and 35.7 min (major), 94% *ee*.



syn-**5e**

6-Chloro-2-(1-(1-naphtyl)-2-propynyl)hexan-1-ol (5e): Isolated yield 64% (*syn*-**5e**/*anti*-**5e** = 3.5/1). A pale yellow oil. *syn*-isomer: ^{1}H NMR δ 8.15 (d, *J* = 8.4 Hz, 1H), 7.77-7.90 (m, 3H), 7.44-7.58 (m, 3H), 5.02 (dd, *J* = 3.8 and 2.7 Hz, 1H), 3.75-3.84 (m, 2H), 3.33 (t, *J* = 6.5 Hz, 2H), 2.35 (d, *J* = 2.7 Hz, 1H), 2.03-2.17 (m, 1H), 1.25-1.81 (m, 5H), 0.96-1.13 (m, 1H). *anti*-isomer: ^{1}H NMR δ 8.01 (d, *J* = 8.6 Hz, 1H), 4.62 (dd, *J* = 5.4 and 2.6 Hz, 1H), 3.68 (dd, *J* = 11.5 and 4.5 Hz, 1H), 3.54 (t, *J* = 6.5 Hz, 2H), 2.41 (d, *J* = 2.6 Hz, 1H). *syn*-isomer: ^{13}C NMR δ 135.2, 134.0, 130.6, 129.0, 127.8, 126.4, 126.24, 125.6, 125.2, 123.0, 83.6, 72.9, 64.1, 44.8, 35.1, 32.5, 25.9, 24.7. *anti*-isomer: ^{13}C NMR δ 134.1, 130.7, 129.2, 128.1, 126.3, 126.19, 125.7, 125.3, 122.7, 84.3, 73.1, 62.9, 44.7, 36.0, 32.7, 29.3, 24.5. HRMS (EI) Calcd for $C_{19}H_{21}ClO$ [M]: 300.1281. Found: 300.1268. The optical purity of *syn*-**5e** was determined by HPLC analysis; Daicel Chiralpak AS-H, hexane/^{i}PrOH = 95/5, flow rate = 0.5 mL/min, λ = 254 nm, retention time; 35.0 min (minor) and 39.1 min (major), 84% *ee*. The optical purity of *anti*-**5e** was determined by HPLC analysis; Daicel Chiralpak AS-H, hexane/^{i}PrOH = 95/5, flow rate = 0.5 mL/min, λ = 254 nm, retention time; 31.4 min (minor) and 62.4 min (major), 94% *ee*.

References

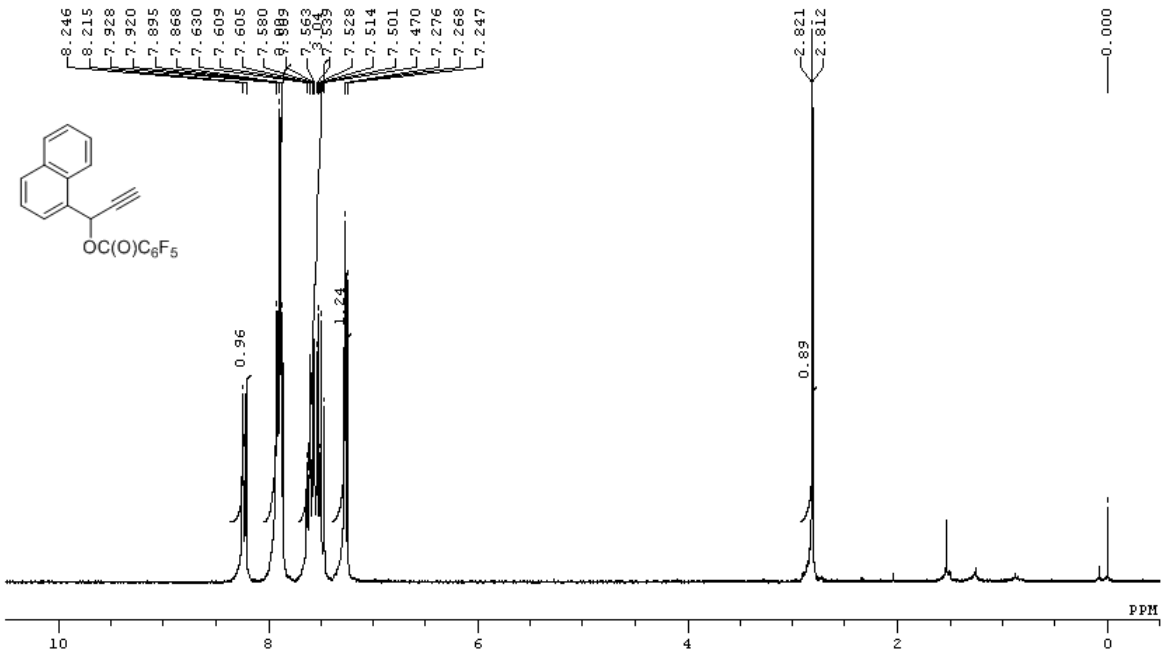
(S1) (a) Stauffer, S. R.; Hartwig, J. F. *J. Am. Chem. Soc.* **2003**, *125*, 6977. (b) Nestl, B. M.; Glueck, S. M.; Hall, M.; Kroutil, W.; Stuermer, R.; Hauer, B.; Faber, K. *J. Org. Chem.* **2006**, 4573.

(S2) Fox, R. J.; Lalic, G.; Bergman, R. G. *J. Am. Chem. Soc.* **2007**, *129*, 14144.

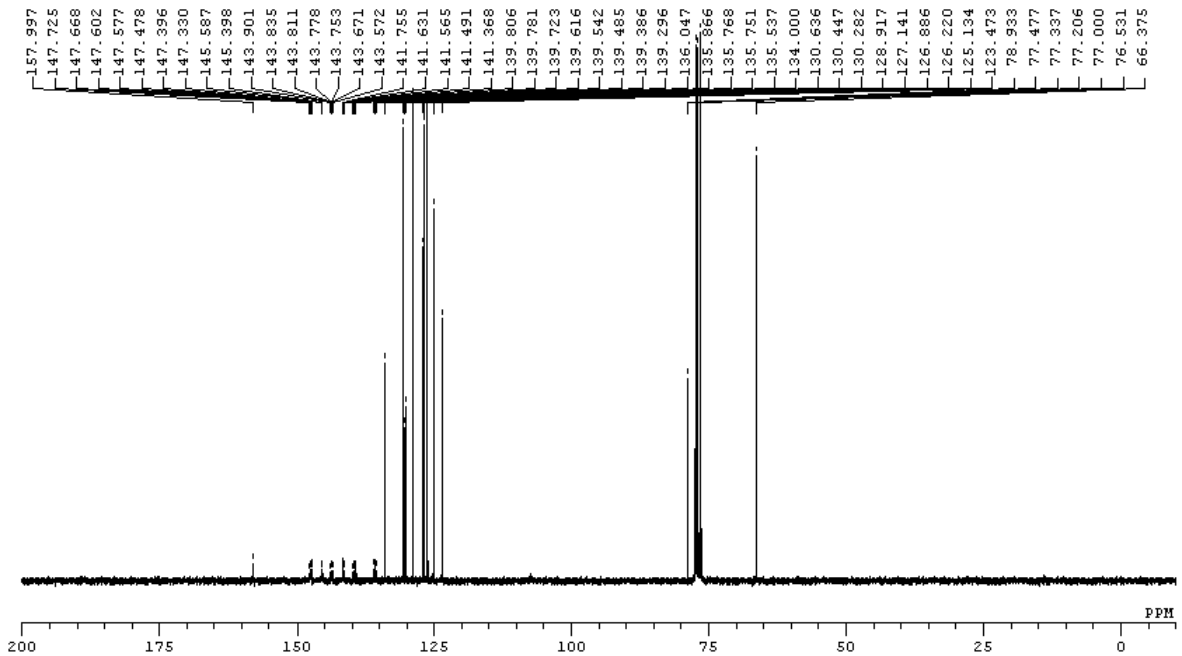
(S3) Ikeda, M.; Miyake, Y.; Nishibayashi, Y. *Angew. Chem., Int. Ed.* **2010**, *49*, 7289.

^{1}H and ^{13}C NMR Spectra.

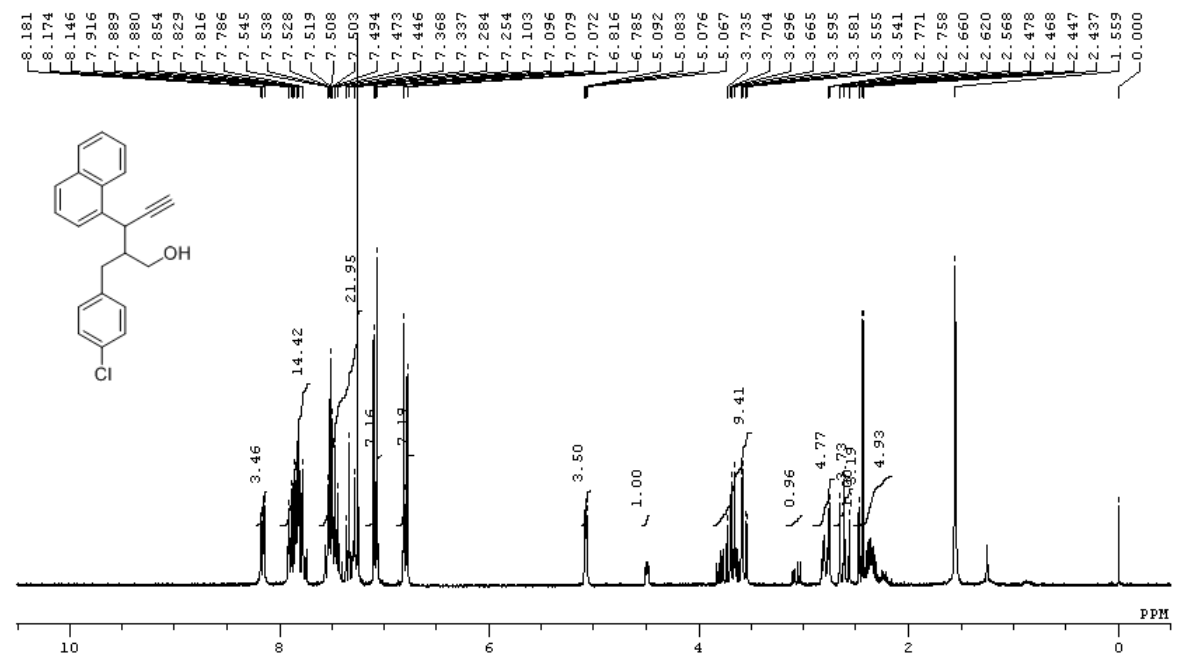
1a



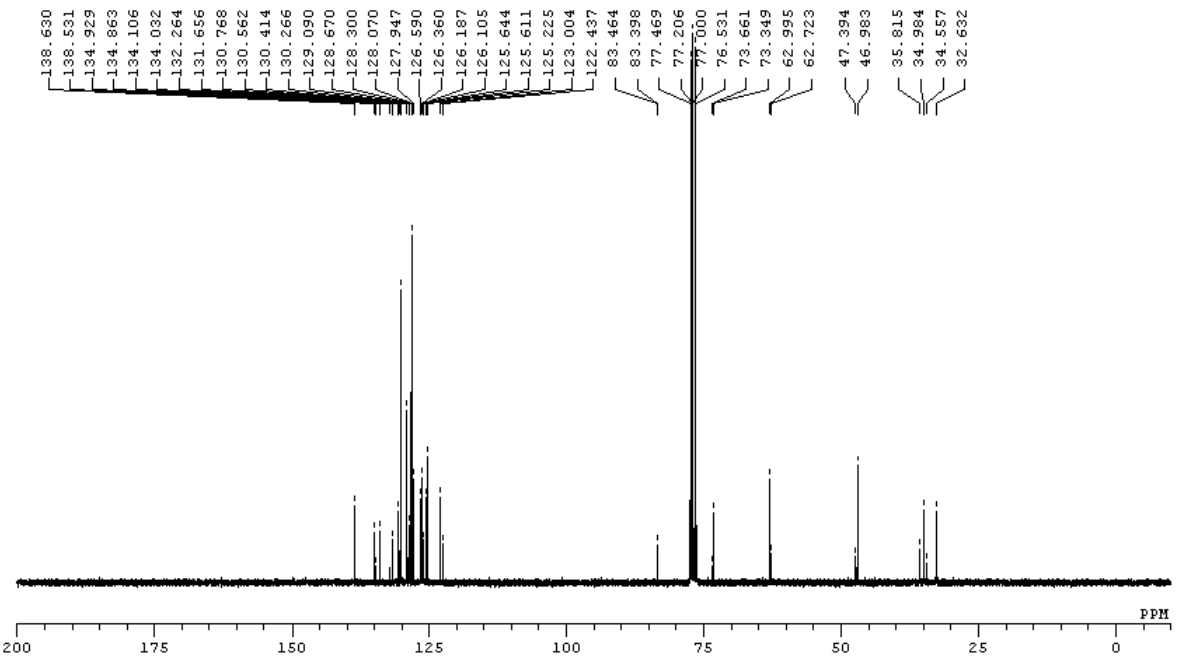
1a



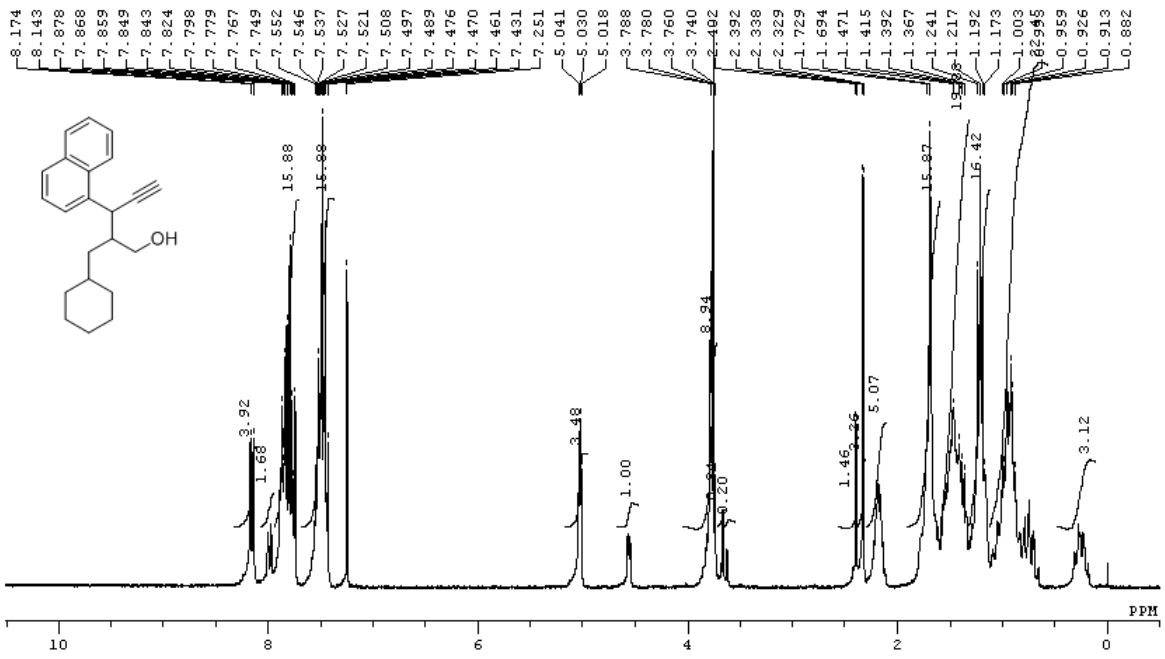
5b


8.181
8.174
8.146
7.916
7.889
7.880
7.854
7.829
7.816
7.786
7.545
7.538
7.528
7.519
7.508
7.503
7.494
7.473
7.446
7.368
7.337
7.284
7.254
7.103
7.096
7.079
7.072
6.816
6.785
5.092
5.083
5.076
5.067
3.735
3.704
3.696
3.665
3.595
3.581
3.555
3.541
2.771
2.758
2.660
2.620
2.568
2.478
2.468
2.447
2.437
1.559
0.000
OH
Cl
21.95
14.42
7.16
7.19
3.46
3.50
1.00
9.41
0.96
4.77
4.93
PPM
10
8
6
4
2
0

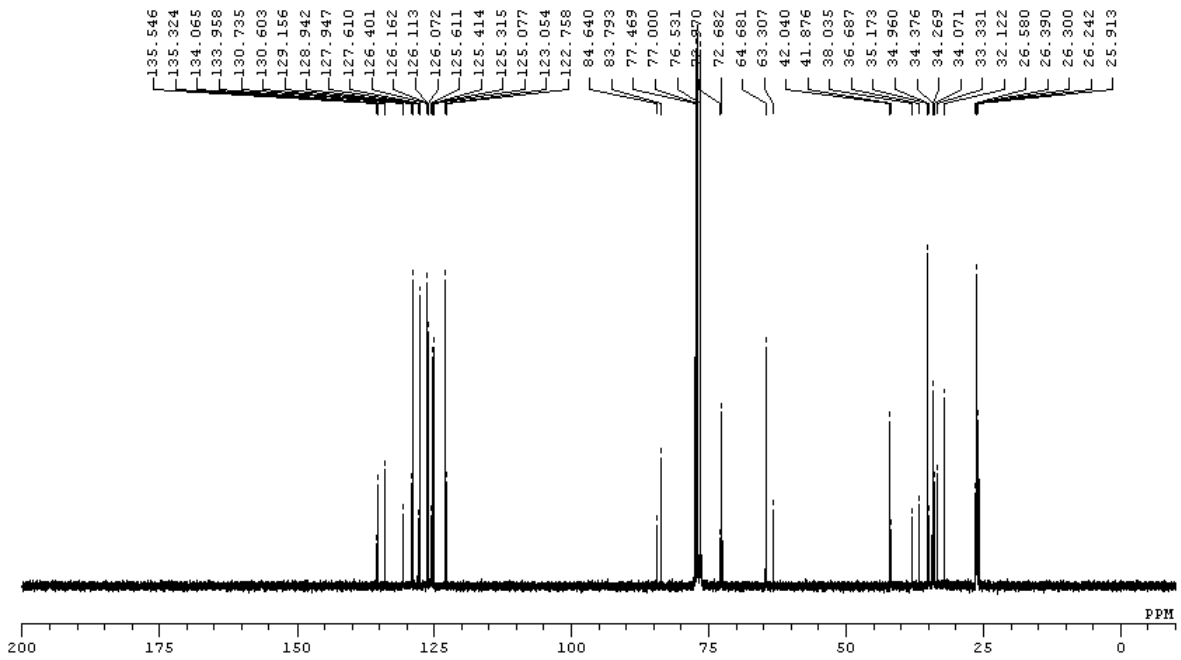
5b


138.630
138.531
134.929
134.863
134.106
134.032
132.264
131.656
130.768
130.562
130.414
130.266
129.090
128.670
128.300
128.070
127.947
126.590
126.360
126.187
126.105
125.644
125.611
125.225
123.004
122.437
83.464
83.398
77.469
77.206
77.000
76.531
73.661
73.349
62.995
62.723
47.394
46.983
35.815
34.984
34.557
32.632
PPM
200
175
150
125
100
75
50
25
0

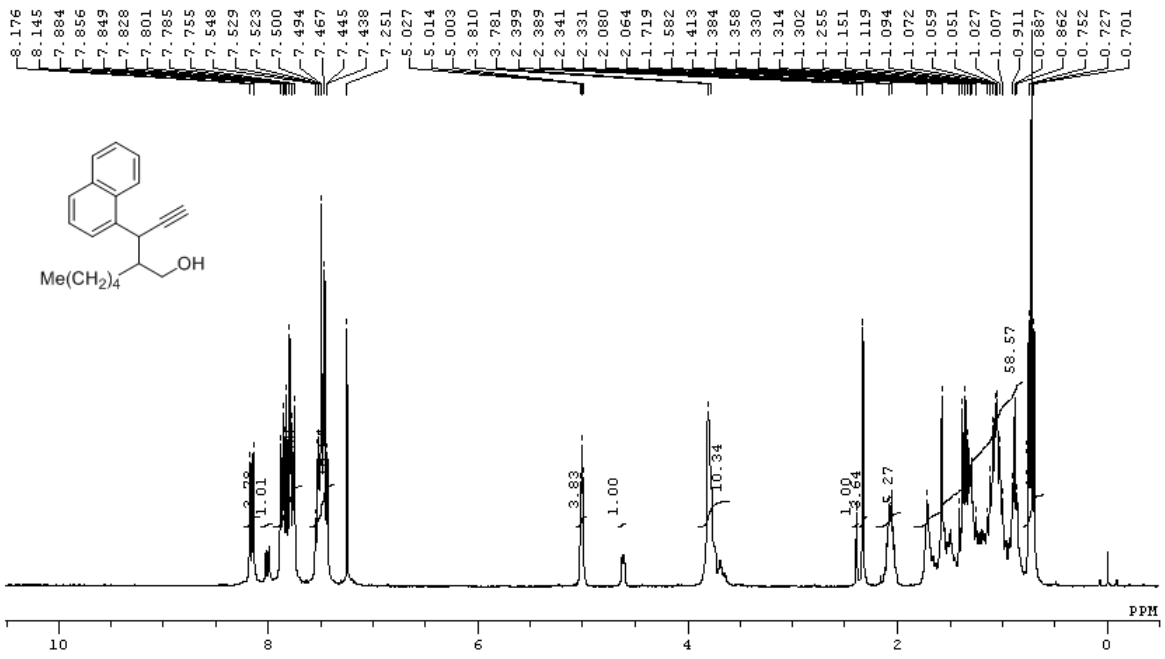
5c


8.174
8.143
7.878
7.868
7.859
7.849
7.843
7.824
7.798
7.779
7.767
7.749
7.552
7.546
7.537
7.527
7.521
7.508
7.497
7.489
7.476
7.470
7.461
7.431
7.251
5.041
5.030
5.018
3.788
3.780
3.760
3.740
2.492
2.392
2.338
2.329
1.729
1.694
1.471
1.415
1.392
1.367
1.241
1.217
1.192
1.173
1.003
0.959
0.926
0.913
0.882
OH
15.88
15.88
8.94
15.87
16.42
3.92
1.68
3.48
1.00
0.84
0.20
1.46
2.36
5.07
3.12
PPM
10
8
6
4
2
0

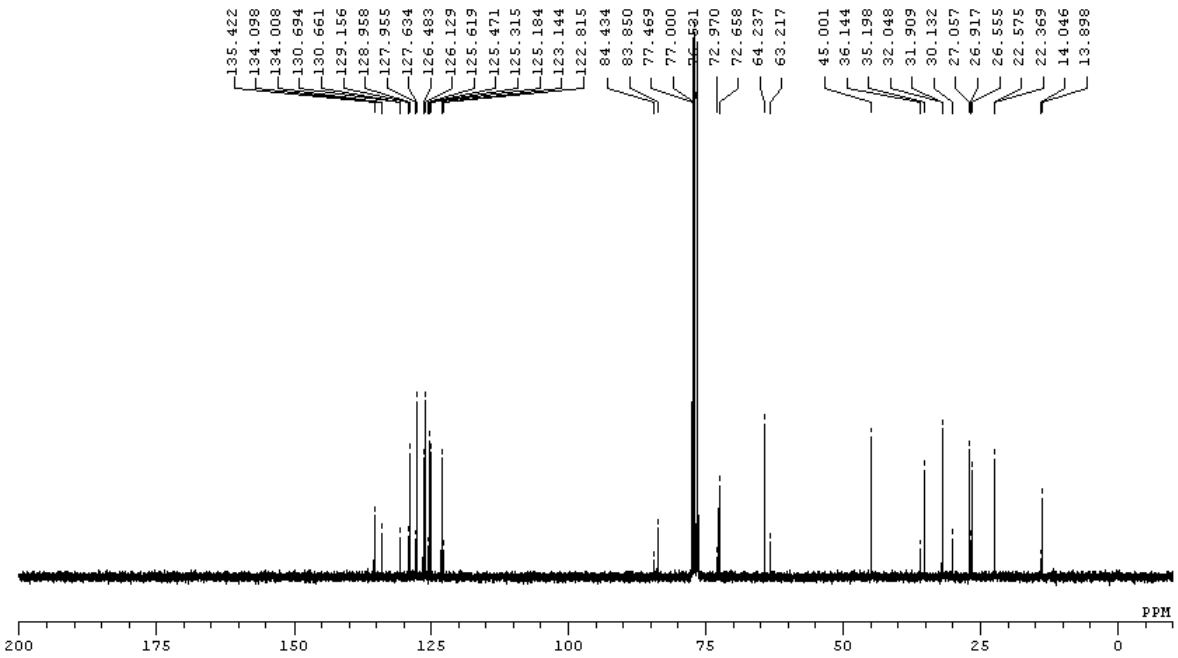
5c


135.546
135.324
134.065
133.958
130.735
130.603
129.156
128.942
127.947
127.610
126.401
126.162
126.113
126.072
125.611
125.414
125.315
125.077
123.054
122.758
84.640
83.793
77.469
77.000
76.531
72.682
64.681
63.307
42.040
41.876
38.035
36.687
35.173
34.960
34.376
34.269
34.071
33.331
32.122
26.580
26.390
26.300
26.242
25.913
PPM
200
175
150
125
100
75
50
25
0

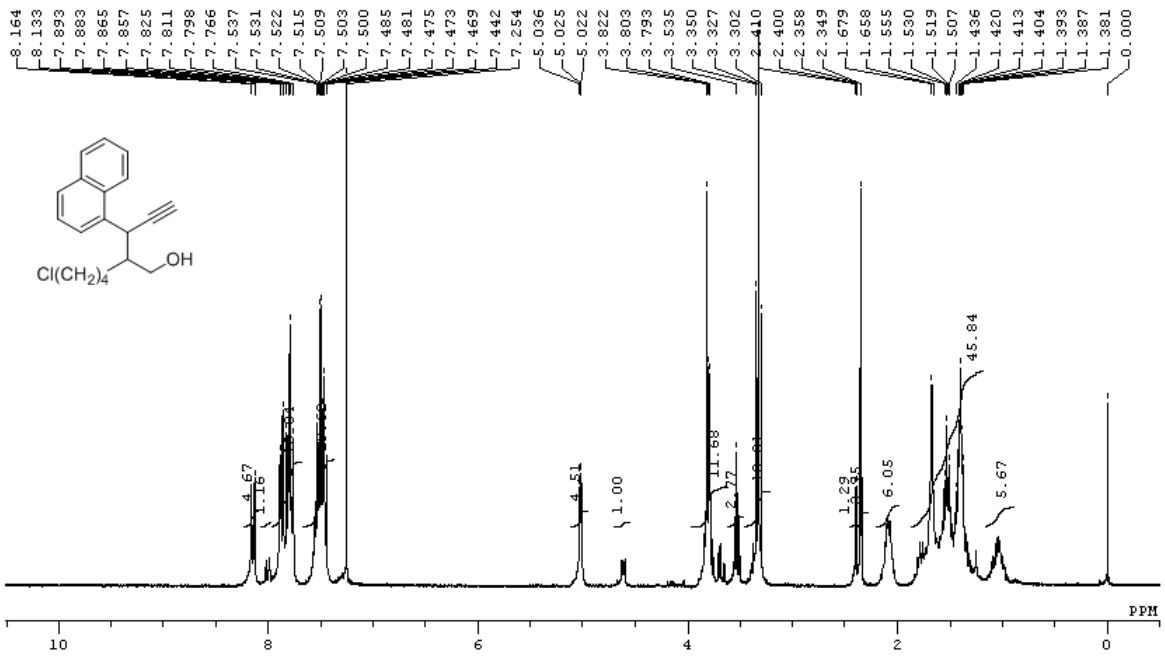
5d


8.176
8.145
7.884
7.856
7.849
7.828
7.801
7.785
7.755
7.548
7.529
7.523
7.500
7.494
7.467
7.445
7.438
7.251
5.027
5.014
5.003
3.810
3.781
2.399
2.389
2.341
2.331
2.080
2.064
1.719
1.582
1.413
1.384
1.358
1.330
1.314
1.302
1.255
1.151
1.119
1.094
1.072
1.059
1.051
1.027
1.007
0.911
0.887
0.862
0.752
0.727
0.701
OH
Me(CH2)4
2.78
1.01
3.83
1.00
10.34
1.00
3.64
5.27
58.57
PPM
10
8
6
4
2
0

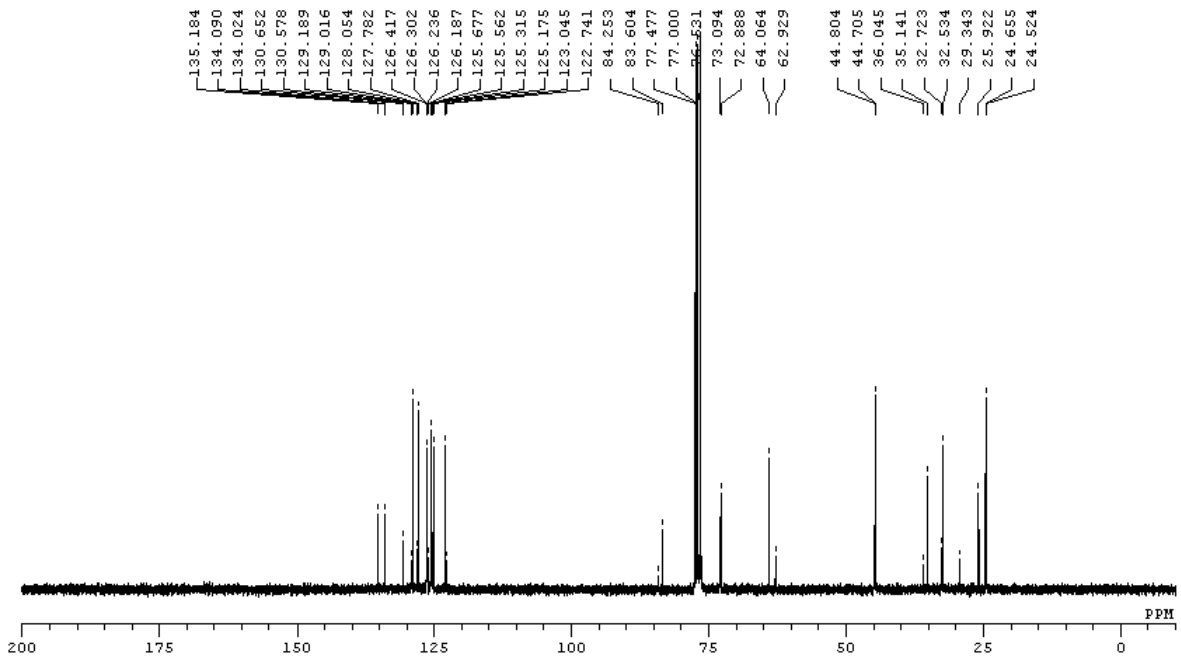
5d


135.422
134.098
134.008
130.694
130.661
129.156
128.958
127.955
127.634
126.483
126.129
125.619
125.471
125.315
125.184
123.144
122.815
84.434
83.850
77.469
77.000
72.970
72.658
64.237
63.217
45.001
36.144
35.198
32.048
31.909
30.132
27.057
26.917
26.555
22.575
22.369
14.046
13.898
PPM
200
175
150
125
100
75
50
25
0

5e

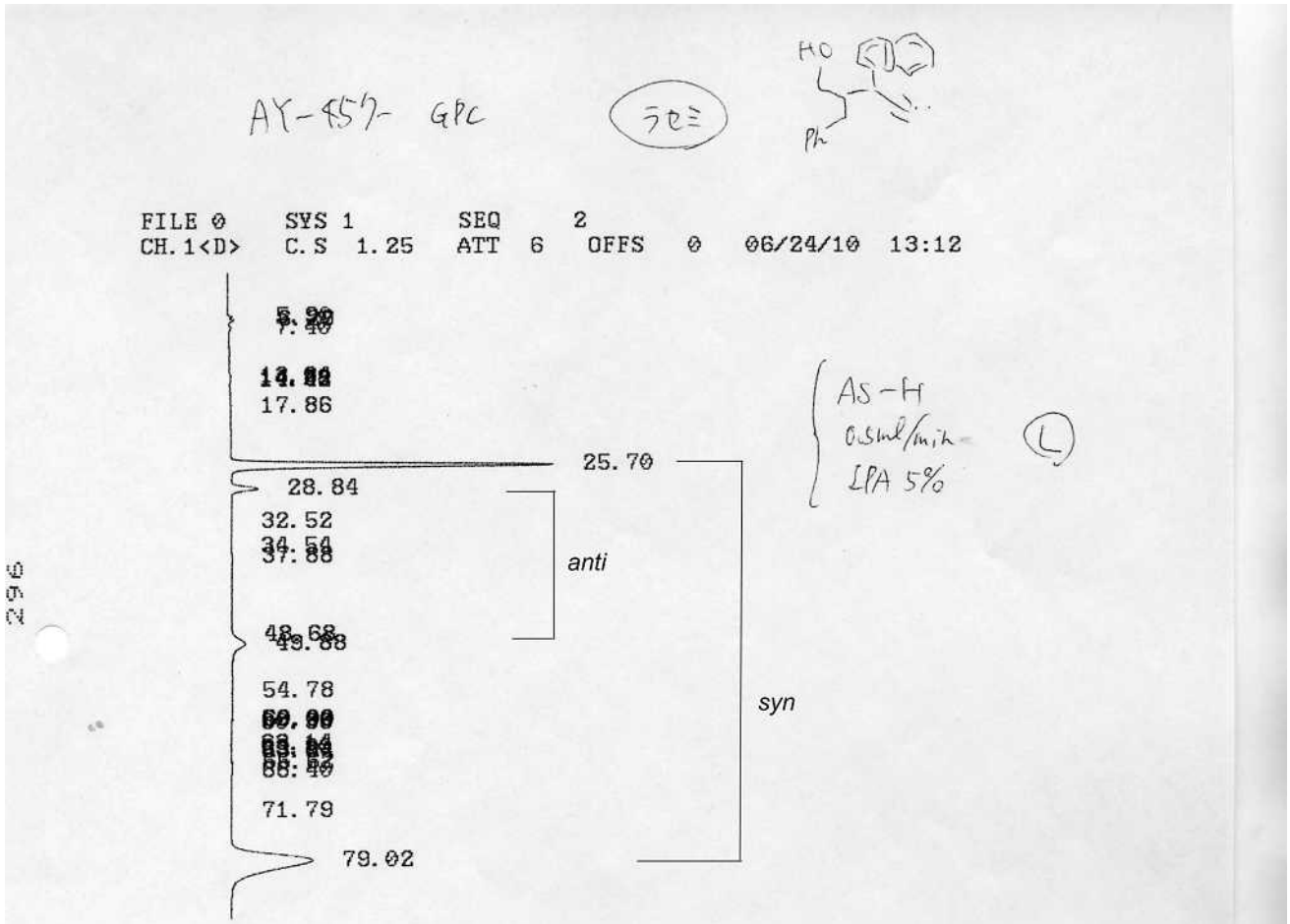

8.164
8.133
7.893
7.883
7.865
7.857
7.825
7.811
7.798
7.766
7.537
7.531
7.522
7.515
7.509
7.503
7.500
7.485
7.481
7.475
7.473
7.469
7.442
7.254
5.036
5.025
5.022
3.822
3.803
3.793
3.535
3.350
3.327
3.302
2.410
2.400
2.358
2.349
1.679
1.658
1.555
1.530
1.519
1.507
1.436
1.420
1.413
1.404
1.393
1.387
1.381
0.000
OH
Cl(CH2)4
4.67
1.16
4.51
1.00
11.68
2.77
10.01
1.29
1.35
6.05
45.84
5.67
PPM
10
8
6
4
2
0

5e


135.184
134.090
134.024
130.652
130.578
129.189
129.016
128.054
127.782
126.417
126.302
126.236
126.187
125.677
125.562
125.315
125.175
123.045
122.741
84.253
83.604
77.477
77.000
76.631
73.094
72.888
64.064
62.929
44.804
44.705
36.045
35.141
32.723
32.534
29.343
25.922
24.655
24.524
PPM
200
175
150
125
100
75
50
25
0

Charts of Propargylic Alkylated Products by HPLC Analysis.

5a (*rac*)



D-7500 INTEGRATOR REPORT

ANALYZED: 06/24/10 13:12 REPORTED: 06/24/10 14:47
SYSTEM : 1
METHOD : OPERATOR:
CHANNEL : 1 <DIGITAL> SEQ : 2

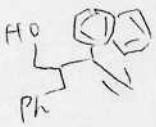
FILE : 0 MODULE T-PROG : DETECTOR= 1
CALC-METHOD: AR/HI% <AREA> COMPONENT TBL : 0

NO.	RT	AREA	CONC	BC
8	25.70	836518	45.844	BB
9	28.84	75721	4.150	BB
14	49.88	79007	4.330	VB
25	79.02	833442	45.676	BB
TOTAL		1824688	100.000	

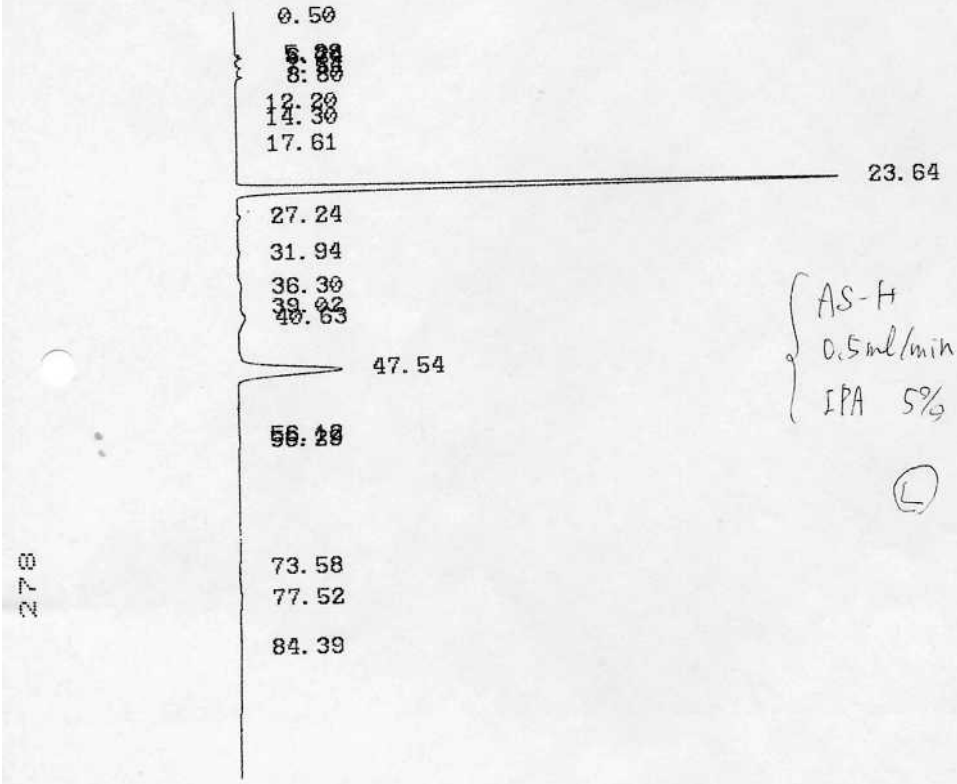
PEAK REJ : 70000

5a

AY-468



FILE 0 SYS 1 SEQ 1
CH. 1<D> C.S 1.25 ATT 7 OFFS 0 06/29/10 11:50



AS-H
0.5ml/min
IPA 5%

L

D-7500 INTEGRATOR REPORT

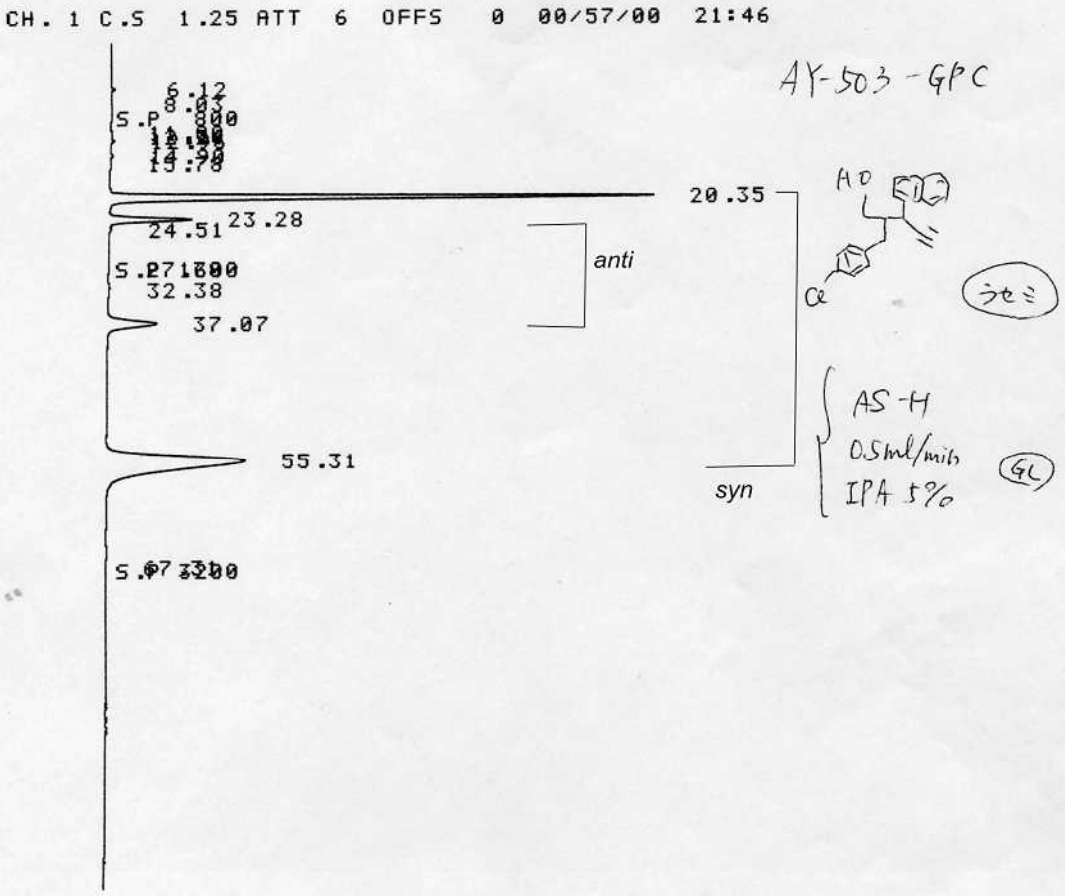
ANALYZED: 06/29/10 11:50 REPORTED: 06/29/10 13:34
SYSTEM : 1
METHOD :
CHANNEL : 1 <DIGITAL>
OPERATOR:
SEQ : 1

FILE : 0
CALC-METHOD: AR/HI% <AREA>
MODULE T-PROG : DETECTOR= 1
COMPONENT TBL : 0

NO.	RT	AREA	CONC	BC	
9	23.64	3022226	71.398	BB	syn 98.2%
10	27.24	20026	0.473	BB	
12	36.30	25304	0.598	BB	
14	40.63	73162	1.728	BB	
15	47.54	1065062	25.161	BB	anti 96.3%
19	77.52	27126	0.641	BB	
TOTAL		4232906	100.000		

PEAK REJ : 20000

5b (*rac*)



D-2500 00/57/00 21:46

METHOD: TAG: 23 CH: 1

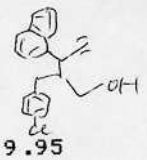
FILE: 0 CALC-METHOD: AREA% TABLE: 0 CONC: AREA

NO.	RT	AREA	CONC	BC
8	20.35	1177060	42.611	BV
9	23.28	203307	7.360	VV
13	37.07	207965	7.529	VB
14	55.31	1174003	42.500	BB
TOTAL		2762335	100.000	

PEAK REJ : 50000

5b

CH. 1 C.S 1.25 ATT 8 OFFS 0 00/11/00 00:40 AY-564-GPC2 -1.2



19.95

35.95

AS-H
0.5ml/min
IIA 5%

GL

D-2500 00/11/00 00:40

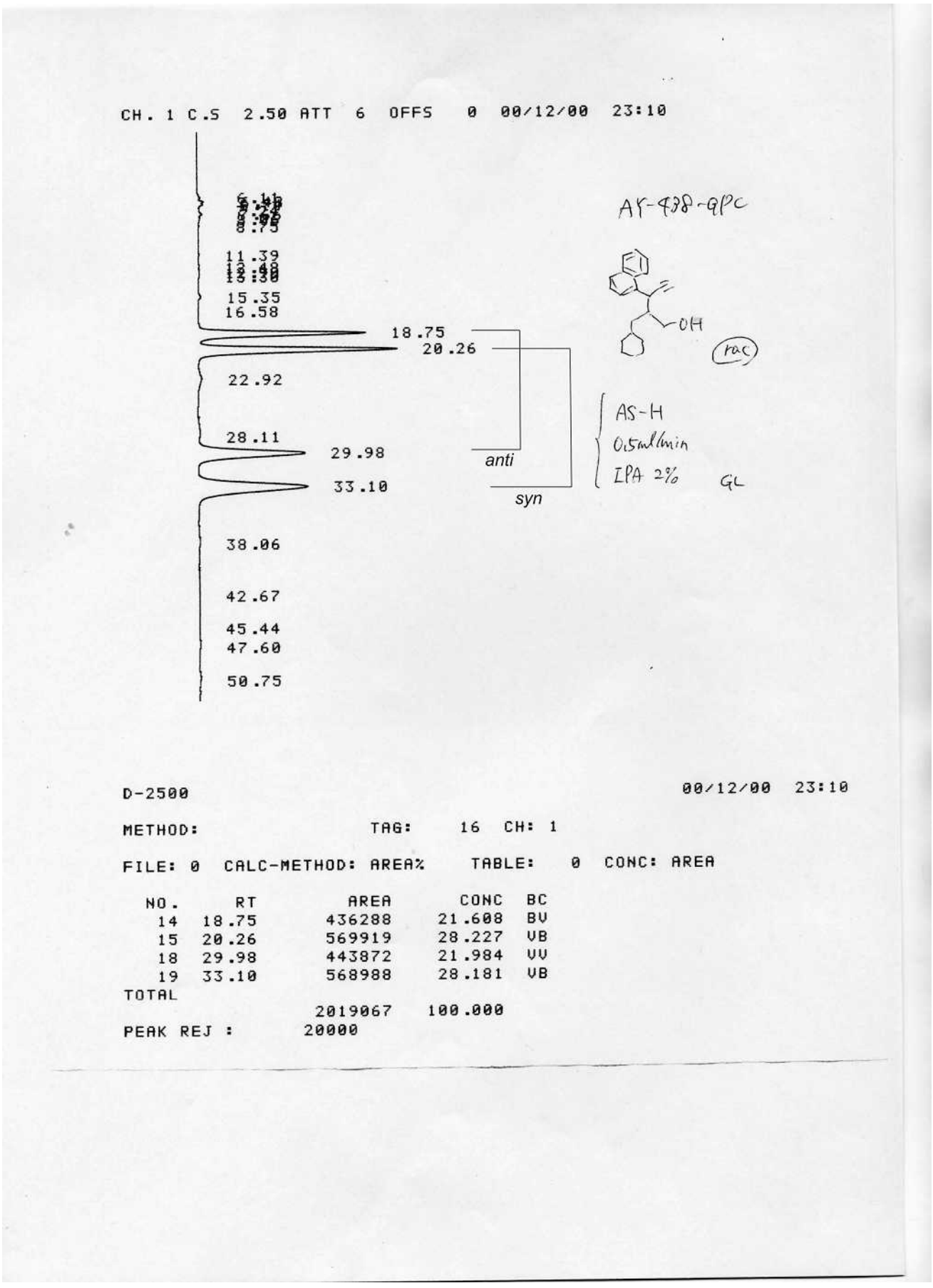
METHOD: TAG: 12 CH: 1

FILE: 0 CALC-METHOD: AREA% TABLE: 0 CONC: AREA

NO.	RT	AREA	CONC	BC	
5	9.57	58035	0.724	VV	
6	10.08	62619	0.781	VV	
14	19.95	6115145	76.245	VV	syn 98.8%
15	22.83	26240	0.327	TBB	
21	35.95	1703512	21.240	BB	anti 97.0%
23	50.06	17912	0.223	BB	
24	54.80	36976	0.461	BB	
TOTAL		8020439	100.000		

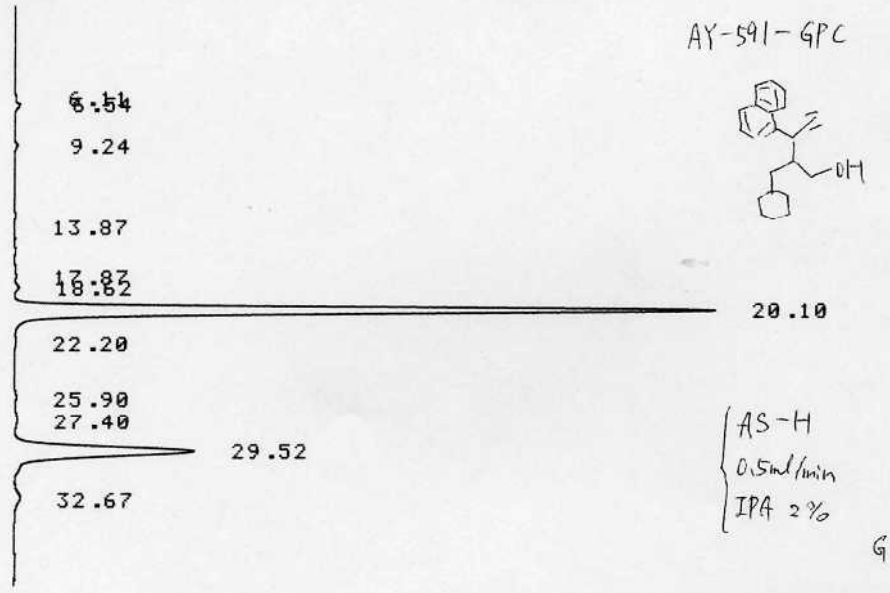
PEAK REJ : 17000

5c (*rac*)


CH. 1 C.S 2.50 ATT 6 OFFS 0 00/12/00 23:10
11.39
15.35
16.58
18.75
20.26
22.92
28.11
29.98
33.10
38.06
42.67
45.44
47.60
50.75
anti
syn
AY-H38-QPC
OH
rac
AS-H
0.5ml/min
IPA 2%
GL
D-2500
00/12/00 23:10
METHOD:
TAG: 16 CH: 1
FILE: 0 CALC-METHOD: AREA% TABLE: 0 CONC: AREA
NO. RT AREA CONC BC
14 18.75 436288 21.608 BV
15 20.26 569919 28.227 VB
18 29.98 443872 21.984 VV
19 33.10 568988 28.181 VB
TOTAL
2019067 100.000
PEAK REJ : 20000

5c

CH. 1 C.S 2.50 ATT 6 OFFS 0 00/14/00 18:17



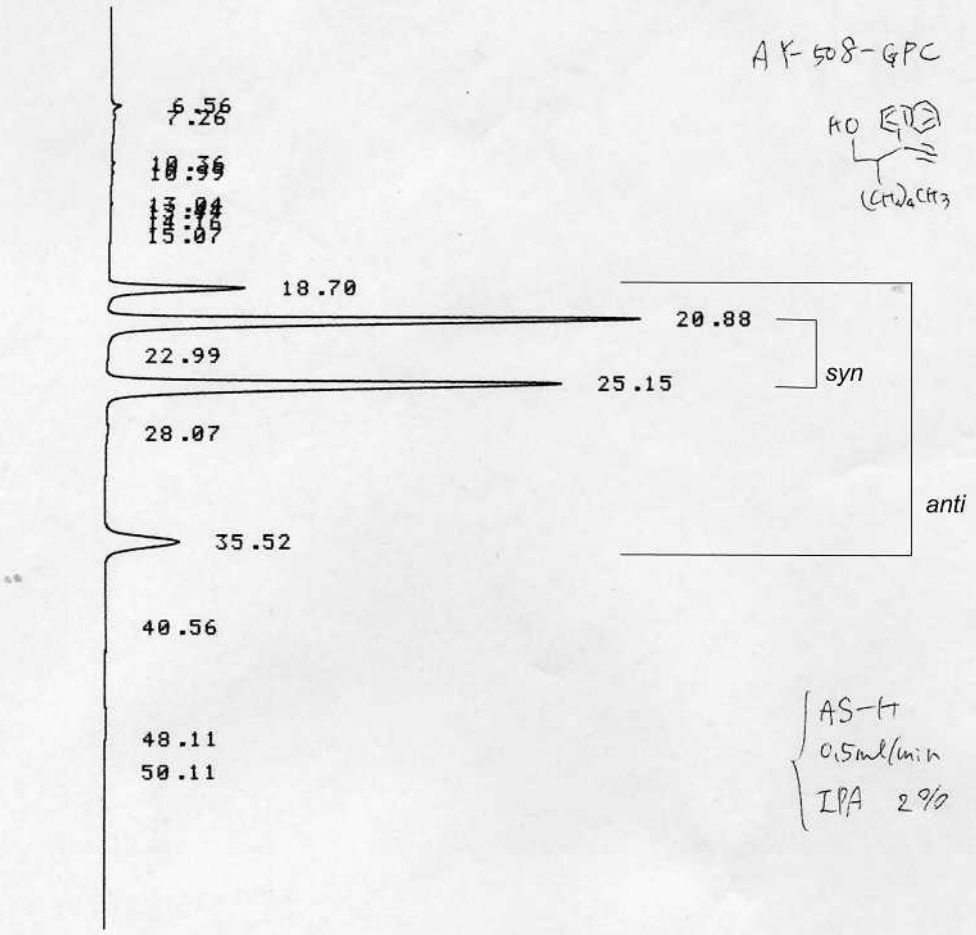
D-2500 00/14/00 18:17

METHOD: TAG: 18 CH: 1

FILE: 0 CALC-METHOD: AREA% TABLE: 0 CONC: AREA

NO.	RT	AREA	CONC	BC		
6	18.62	6946	0.346	VB	anti	97.5%
7	20.10	1427082	71.100	BV		
10	27.40	11605	0.578	VV		
11	29.52	540397	26.924	VV		
12	32.67	21116	1.052	TBB	syn	97.1%
TOTAL		2007146	100.000			
PEAK REJ :		6500				

5d (*rac*)



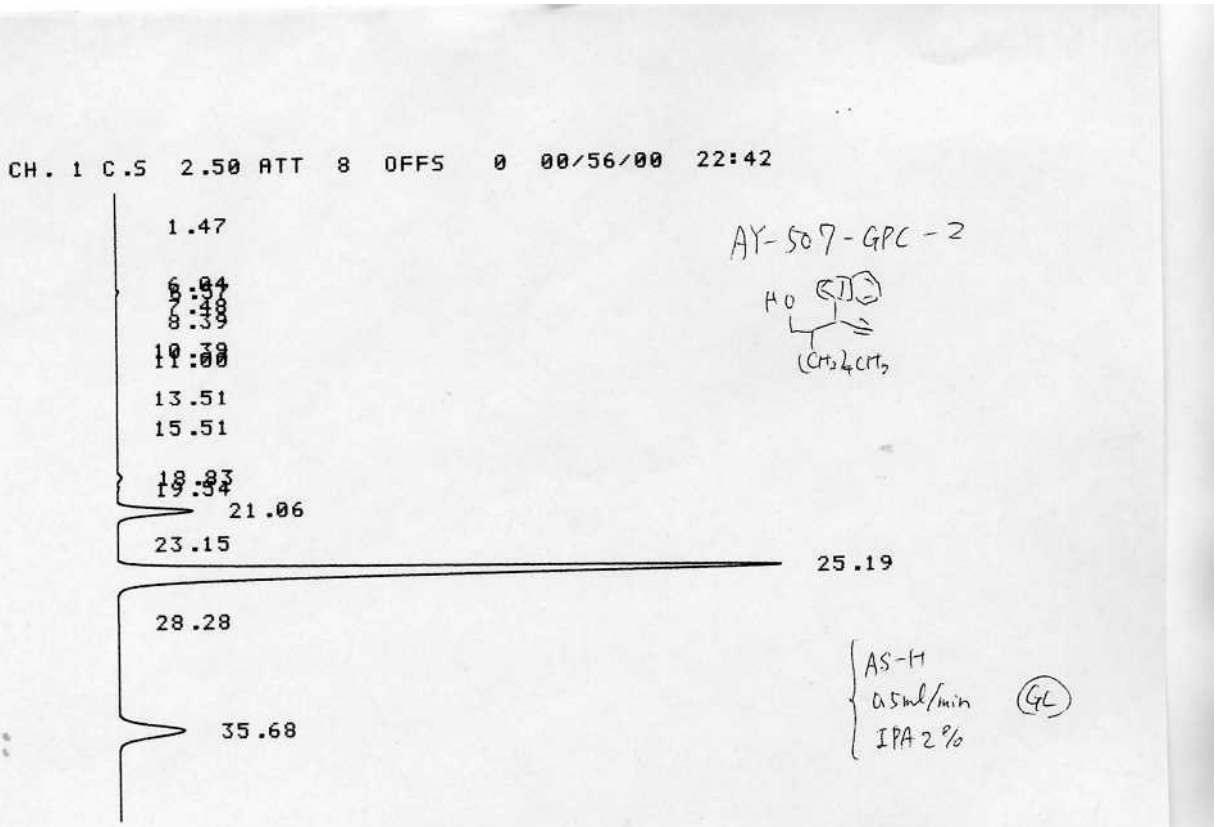
D-2500 00/56/00 21:33

METHOD: TAG: 19 CH: 1

FILE: 0 CALC-METHOD: AREA% TABLE: 0 CONC: AREA

NO.	RT	AREA	CONC	BC	
9	18.70	507216	9.250	BB	
10	20.88	2237147	40.800	BV	syn
12	25.15	2237507	40.807	BB	
14	35.52	501332	9.143	BB	anti
TOTAL		5483202	100.000		
PEAK REJ :		500000			

5d



D-2500 00/56/00 22:42

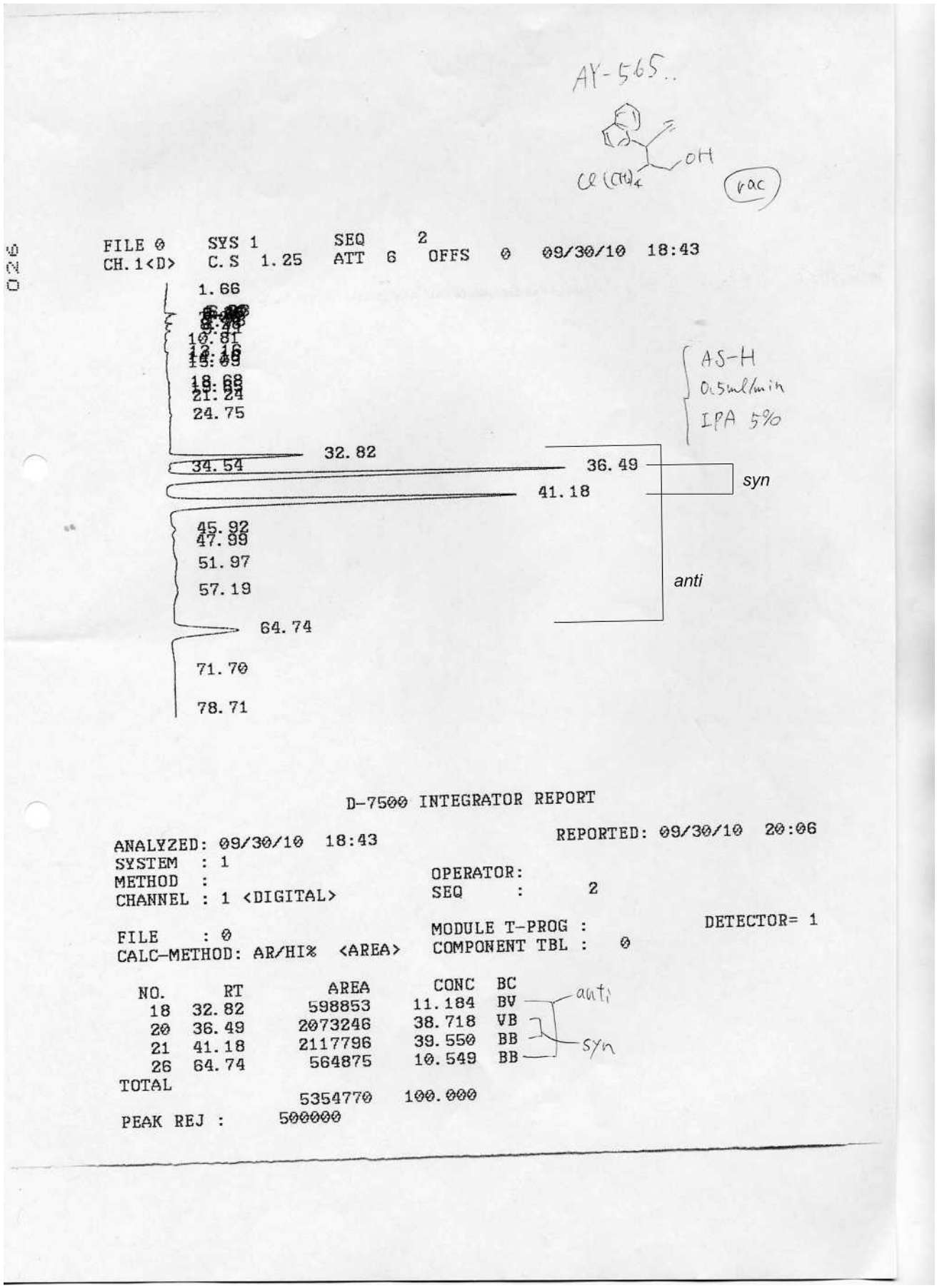
METHOD: TAG: 20 CH: 1

FILE: 0 CALC-METHOD: AREA% TABLE: 0 CONC: AREA

NO.	RT	AREA	CONC	BC	
10	18.83	30528	0.364	BV	syn 83.4%
12	21.06	619677	7.391	BB	
14	25.19	6827663	81.432	BB	anti 93.5%
16	35.68	906587	10.813	BB	
TOTAL		8384455	100.000		

PEAK REJ : 30000

5e (*rac*)



D-7500 INTEGRATOR REPORT

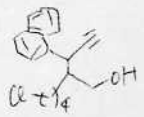
ANALYZED: 09/30/10 18:43 REPORTED: 09/30/10 20:06
SYSTEM : 1
METHOD :
CHANNEL : 1 <DIGITAL>
OPERATOR:
SEQ : 2
FILE : 0
CALC-METHOD: AR/HI% <AREA>
MODULE T-PROG : DETECTOR= 1
COMPONENT TBL : 0

NO.	RT	AREA	CONC	BC	
18	32.82	598853	11.184	BV	anti
20	36.49	2073246	38.718	VB	
21	41.18	2117796	39.550	BB	syn
26	64.74	564875	10.549	BB	
TOTAL		5354770	100.000		

PEAK REJ : 500000

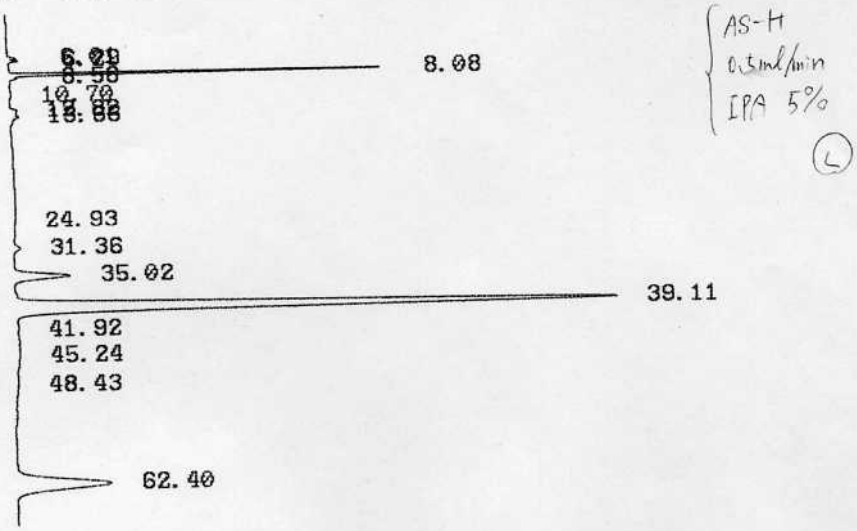
5e

AY-566-GPC



FILE 0 SYS 1 SEQ 3
CH.1<D> C.S 1.25 ATT 5 OFFS 0 11/04/10 15:19

AS-H
0.5ml/min
IPA 5%



D-7500 INTEGRATOR REPORT

ANALYZED: 11/04/10 15:19 REPORTED: 11/04/10 16:27
SYSTEM : 1
METHOD :
CHANNEL : 1 <DIGITAL>
OPERATOR:
SEQ : 3
FILE : 0
CALC-METHOD: AR/HI% <AREA>
MODULE T-PROG : DETECTOR= 1
COMPONENT TBL : 0

NO.	RT	AREA	CONC	BC	
3	8.08	157508	8.914	BV	
6	12.82	11016	0.623	BV	
7	13.66	11360	0.643	VB	
8	24.93	14978	0.848	BB	anti 94.3%
9	31.36	8455	0.478	BB	
10	35.02	98517	5.575	BB	
11	39.11	1165308	65.948	BB	syn 84.4%
13	45.24	10304	0.583	BB	
15	62.40	289553	16.387	BB	
TOTAL		1766999	100.000		
PEAK REJ :		6000			