**Supporting Information**

# Practical Rh(I)-Catalyzed Asymmetric Hydrogenation of β-(Acylamino)acrylates Using a New Unsymmetrical Hybrid Ferrocenylphosphine-Phosphoramidite Ligand: Crucial Influence of a N-H Proton in the Ligand

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**General Procedures**: All synthetic reactions and manipulations were performed in a nitrogen or argon atmosphere using standard Schlenk techniques. Solvents were reagent grade, dried and distilled before use following standard procedures. ($S_c$,$R_p$,$S_a$)-**1**,[1] Bophoz-Me and Bophoz-H[2] were prepared according the literature procedure. $\beta$-aryl-$\beta$-(acylamino)acrylates **3**[3] and $\beta$-alkyl-$\beta$-(acylamino)acrylates **4**[4] were known compounds which were synthesized according to the literature procedure. All other chemicals obtained commercially. Optical rotations were measured on a JASCO P-1020 high sensitive polarimeter. $^{1}$H, $^{13}$C and $^{31}$P NMR spectra were recorded at room temperature on a BRUKER DEX 400 (400 MHz) spectrometers. Chemical shifts were determined relative to the residual solvent peaks (e.g. $CH_2Cl_2$, $\delta$ = 5.30 ppm for proton atoms, $\delta$ = 54.2 ppm for carbon atoms; $H_3PO_4$, $\delta$ = 0 ppm for phosphorus atoms). Enantiomeric excesses were determined by capillary GC analysis with a Chiral Select 1000 column (0.25mm x 30m) for **5a-h, 6a-b** and **6d**, with a CP-Chiralsil-L-Val capillary column (0.25mm x 25m) for **6e**, and HPLC analysis with a chiralcel OD column for **6c.**

**Synthesis of ferrocenylphosphine-phosphoramidite ligand** ($S_c$,$R_p$,$S_a$)-**2**

($S_a$)-Chlorophosphite **8** (350.5 mg, 1.0 mmol) was dissolved in 4.0 mL of dried dichloromethane, which was cooled to 0$^{o}$C. A solution of ($S_c$,$R_p$)-PPFNH$_2$ **7** (413 mg,

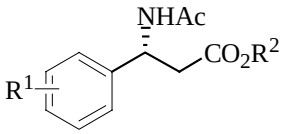
1.0 mmol) and $Et_3N$ (303 mg, 3.0 mmol) in 4.0 mL of dichloromethane was added to above-solution during 30 minutes. The resulting mixture was standing at room temperature overnight. The precipitation was filtrated. The filtrate was collected, and concentrated under reduced pressure to c.a. 2 mL. Adding the *n*-hexane to the filtrate gave the yellow power, which was sufficient pure for further use. An analytic sample was obtained by column chromatography purification (silica gel, hexanes / ethyl acetate = 1 / 1) to give yellow power ($S_c$,$R_p$,$S_a$)-**2**, which can be crystallized from hexane/dichloromethane. $^1$H NMR (400 MHz, $CD_2Cl_2$): δ 1.69-1.70 (d, $J$ = 6.8 Hz, 3 H), 3.35-3.36 (m, 1 H), 3.85 (s, 1 H), 3.97 (s, 5 H), 4.28-4.29 (t, $J$ = 2.4 Hz, 1 H), 4.41 (s, 1 H), 4.75-4.84 (m, 1 H), 6.95-6.98 (m, 1 H), 7.11-7.59 (m, 17 H), 7.84-7.92 (m, 4 H) ppm; $^1$C NMR (400 MHz, $CD_2Cl_2$): 26.1, 46.5, 69.5, 69.9, 70.5, 72.5, 122.6, 123.5, 125.4, 126.7, 127.4, 128.3, 128.6, 128.8, 129.0, 129.9, 130.1, 130.6, 132.0, 133.1, 133.3, 136.0, 136.2 ppm; $^{31}$P NMR (400 MHz, $CD_2Cl_2$): δ -24.2, 152.7 (d, $J$ = 58.0 Hz) ppm. HRMS calcd for $C_{45}H_{35}FeNO_2P_2$: 727.1492, found: 727.1480.

**General procedure for asymmetric hydrogenation and determination of enantiomeric excesses.**

In a nitrogen-filled glovebox, a stainless steel autoclave was charged with $Rh(COD)_2BF_4$ (2.0 mg, 0.5 x $10^{-2}$ mmol) and ferrocenylphosphine-phosphoramidite ligand ($S_c$,$R_p$,$S_a$)-**2** (4.0 mg, 0.55 x $10^{-2}$ mmol) in 1.5 mL of a degassed solvent. After stirring for 10 min at room temperature. A substrate (0.5 mmol) in 1.5 mL of same solvents was added to the reaction mixture, and then the hydrogenation was performed under 10 bar of $H_2$ pressure for 12 hour at the indicated temperature. The reaction mixture was passed through a short silica gel column to remove the catalyst. After evaporation of the solvent, the crude reaction mixture was subjected for GC to determine the conversion and enantiomeric excesses.

**Determination of Enantiomeric Excesses for β-Aryl-β-(Acetylamino)propanoate 5: Chiral Capillary GC Column.** Chiral Select-1000 column (dimensions 30 m x 0.25 mm (i.d.)). Carrier gas: $N_2$. The racemic products were obtained by hydrogenation of substrates with an achiral catalyst prepared from $PPh_3$ and

$Rh(COD)_2BF_4$. The following are the retention times for the racemic products.




**5a**: $R^1$ = H, $R^2$ = Et; **5b**: $R^1$ = 4-Me, $R^2$ = Et;
**5c**: $R^1$ = 4-Me, $R^2$ = Me; **5d**: $R^1$ = 4-OMe, $R^2$ = Et;
**5e**: $R^1$ = 4-OMe, $R^2$ = Me; **5f**: $R^1$ = 4-Cl, $R^2$ = Et;
**5g**: $R^1$ = 4-Cl, $R^2$ = Me; **5h**: $R^1$ = 4-F, $R^2$ = Me;
**5i**: $R^1$ = 3-OMe, $R^2$ = Me

**Ethyl 3-Acetamido-3-phenylpropanoate (5a)**: (capillary GC, Chiral Select-1000 column, 155°C, 15 psi) (*S*) $t_1$ = 29.96, (*R*) $t_2$ = 31.86; (capillary GC, Chiral Select-1000 column, 160°C, 15 psi) (*S*) $t_1$ = 22.96, (*R*) $t_2$ = 24.86.

**Ethyl 3-Acetamido-3-(4-methylphenyl)propanoate (5b)**: (capillary GC, Chiral Select-1000 column, 160°C, 10 psi) (*S*) $t_1$ = 58.19, (*R*) $t_2$ = 60.76.

**Methyl 3-Acetamido-3-(4-methylphenyl)propanoate (5c)**: (capillary GC, Chiral Select-1000 column, 160°C, 10 psi) (*S*) $t_1$ = 44.22, (*R*) $t_2$ = 46.78.

**Ethyl 3-Acetamido-3-(4-methoxyphenyl)propanoate (5d)**: (capillary GC, Chiral Select-1000 column, 160°C, 10 psi) (*S*) $t_1$ = 130.12, (*R*) $t_2$ = 134.49.

**Methyl 3-Acetamido-3-(4-methoxyphenyl)propanoate (5e)**: (capillary GC, Chiral Select-1000 column, 160°C, 10 psi) (*S*) $t_1$ = 103.6, (*R*) $t_2$ = 108.8.

**Ethyl 3-Acetamido-3-(4-chlorophenyl)propanoate (5f)**: (capillary GC, Chiral Select-1000 column, 160°C, 15 psi) (*S*) $t_1$ = 72.15, (*R*) $t_2$ = 76.93.

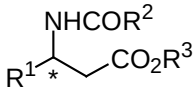
**Methyl 3-Acetamido-3-(4-chlorophenyl)propanoate (5g)**: (capillary GC, Chiral Select-1000 column, 160°C, 15 psi) (*S*) $t_1$ = 58.40, (*R*) $t_2$ = 63.19.

**Methyl 3-Acetamido-3-(4-fluorophenyl)propanoate (5h)**: (capillary GC, Chiral Select-1000 column, 160°C, 15 psi) (*S*) $t_1$ = 20.86, (*R*) $t_2$ = 22.32.

**Methyl 3-Acetamido-3-(3-methoxyphenyl)propanoate (5i)**: (capillary GC, Chiral Select-1000 column, 160°C, 10 psi) (*S*) $t_1$ = 80.31, (*R*) $t_2$ = 85.17.

**Determination of Enantiomeric Excesses for β-Alkyl-β-(Acylamino)propanoate 6:** Chiral Select-1000 column (dimensions 30 m x 0.25 mm (i.d.)), carrier gas: $N_2$, or CP-Chiralsil-L-Val column (dimensions 25 m x 0.25 mm (i.d.)), carrier gas: $H_2$. The

racemic products were obtained by hydrogenation of substrates with an achiral catalyst prepared from $PPh_3$ and $Rh(COD)_2BF_4$. The following are the retention times for the racemic products.




**6a**: $R^1$ = Me, $R^2$ = Me, $R^3$ = Me;
**6b**: $R^1$ = Me, $R^2$ = Me, $R^3$ = Et;
**6c**: $R^1$ = Me, $R^2$ = Ph, $R^3$ = Me;
**6d**: $R^1$ = Et, $R^2$ = Me, $R^3$ = Me;
**6e**: $R^1$ = *i*-Pr, $R^2$ = Me, $R^3$ = Me

**Methyl 3-Acetamidobutanoate (6a)**: (capillary GC, Chiral Select-1000 column, 130$^o$C, 15 psi) (*S*) $t_1$ = 4.54, (*R*) $t_2$ = 5.22.

**Ethyl 3-Acetamidobutanoate (6b)**: (capillary GC, Chiral Select-1000 column, 130$^o$C, 15 psi) (*S*) $t_1$ = 6.30, (*R*) $t_2$ = 7.20.

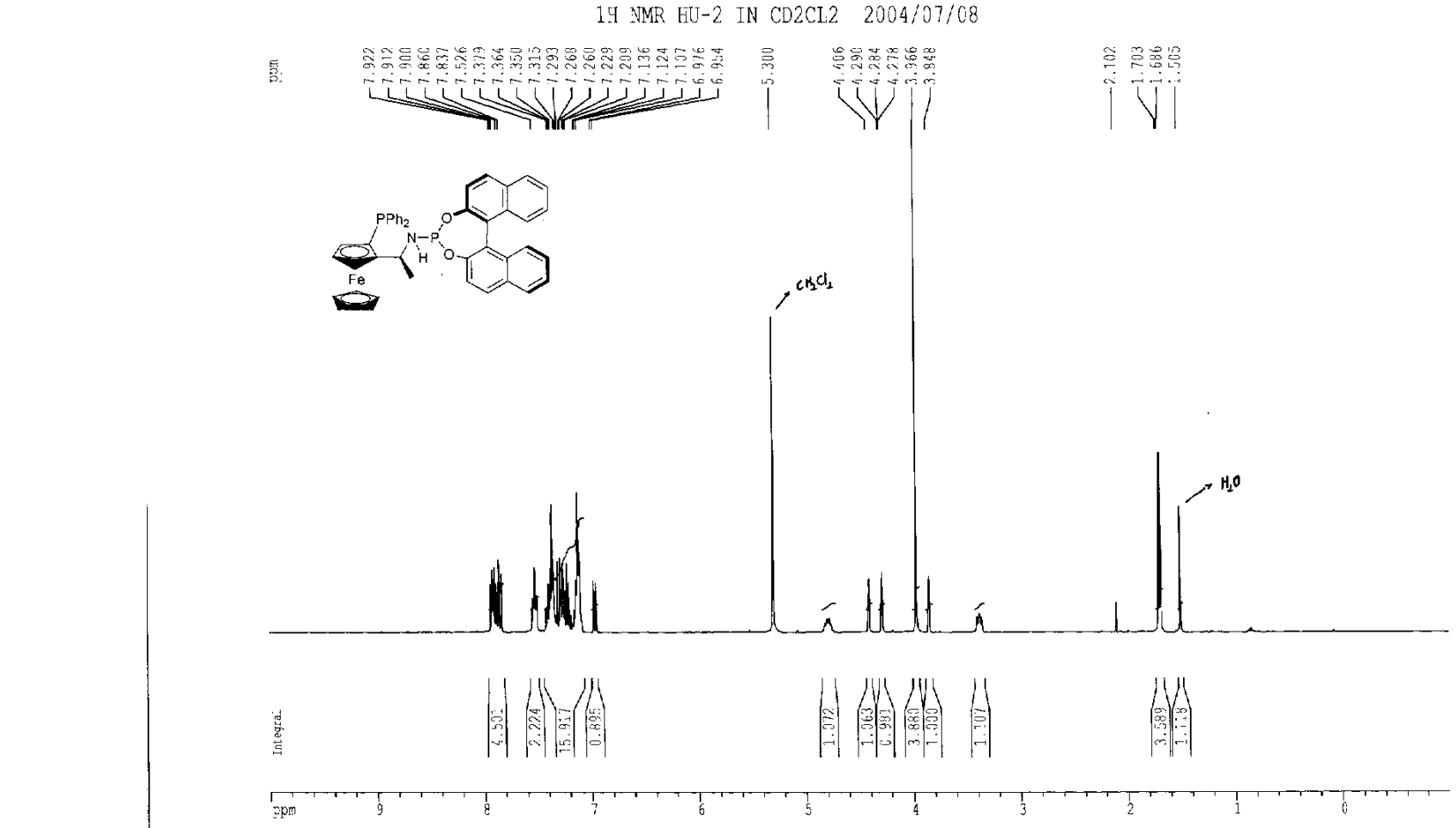
**Methyl 3-Benzamidobutanoate (6c)**: (HPLC, Chiralcel OD column, hexane/*i*-propanol = 95: 5, 1 ml/min, 254nm ) (*S*) $t_1$ = 40.41, (*R*) $t_2$ = 45.44.

**Methyl 3-Acetamidopentanoate (6d)**: (capillary GC, Chiral Select-1000 column, 110$^o$C, 15 psi) (*S*) $t_1$ = 14.82, (*R*) $t_2$ = 16.52. (capillary GC, Chiral Select-1000 column, 110$^o$C, 17 psi) (*S*) $t_1$ = 13.03, (*R*) $t_2$ = 14.52.

**Methyl 4-methyl-3-Acetamidopentanoate (6e)**: (capillary GC, CP-Chiralsil-L-Val column, 125$^o$C, 20 psi) (*S*) $t_1$ = 5.40, (*R*) $t_2$ = 5.67.

## References:


1. Hu, X.-P.; Zheng, Z. *Org. Lett.* **2002**, *4*, 2421.
2. (a) Boaz, N. W.; Debenham, S. D.; Mackenzie, E. B.; Large, S. E. *Org. Lett.* **2002**, *4*, 2421.
3. (a) Zhou, Y.-G.; Tang, W.; Wang, W.; Li, W.; Zhang, X. *J. Am. Chem. Soc.* **2002**, *124*, 4952. (b) Tang, W.; Wang, W.; Chi, Y.; Zhang, X. *Angew. Chem. Int. Ed.* **2003**, *42*, 3509.
4. (a) Zhu, G.; Chen, Z.; Zhang, X. *J. Org. Chem.* **1999**, *64*, 6907. (b) Heller, D.; Holz, J.; Drexler, H. J.; Lang, J.; Drauz, K.; Krimmer, H.-P.; Börner, A. *J. Org. Chem.* **2001**, *66*, 6816.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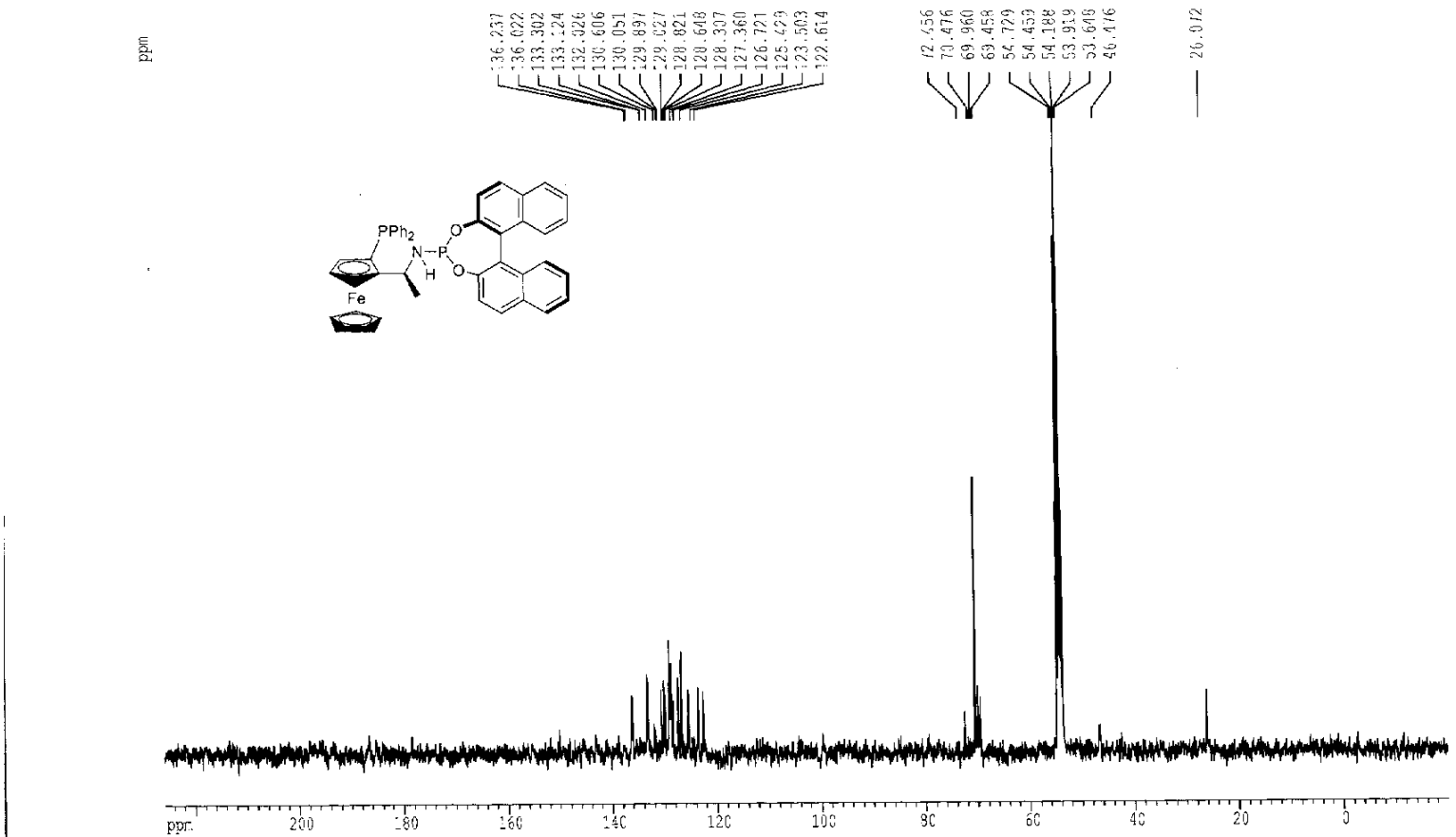

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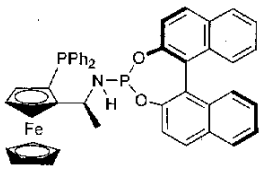

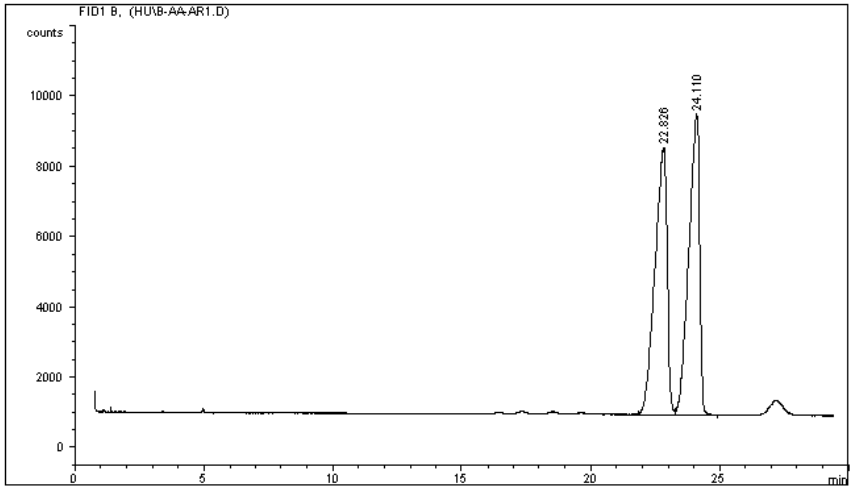


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 Results obtained with enhanced integrator!
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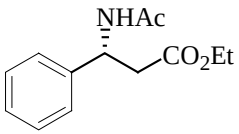
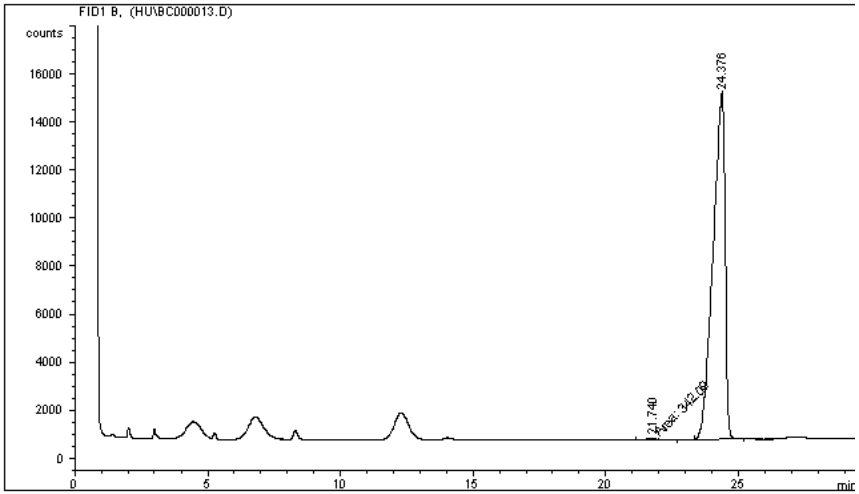



*Rac*-**5a**

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 Results obtained with enhanced integrator!
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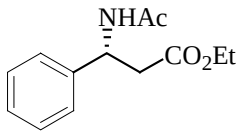
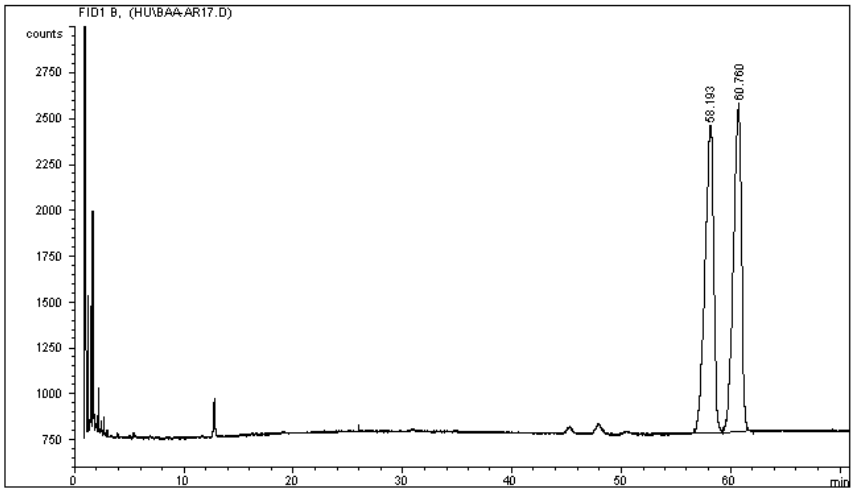



**5a**

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 Results obtained with enhanced integrator!
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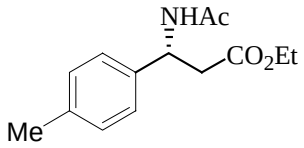
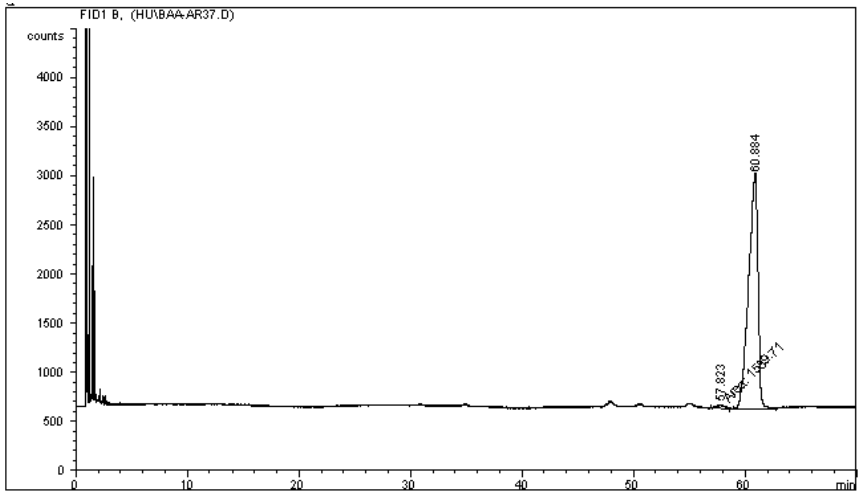



*Rac*-**5b**

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   2  60.884 VV    0.9129 1.39882e5  2368.80884 98.87631

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 Results obtained with enhanced integrator!
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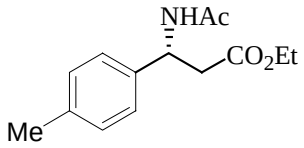
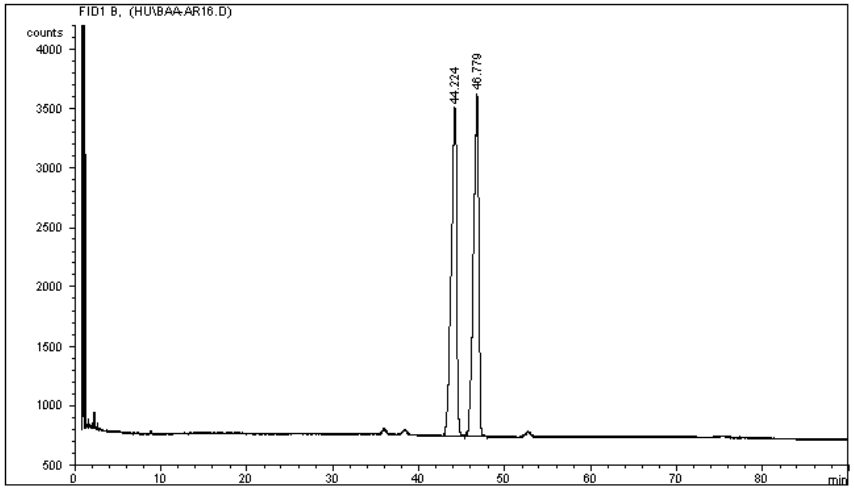



**5b**

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 Results obtained with enhanced integrator!
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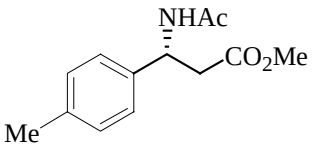
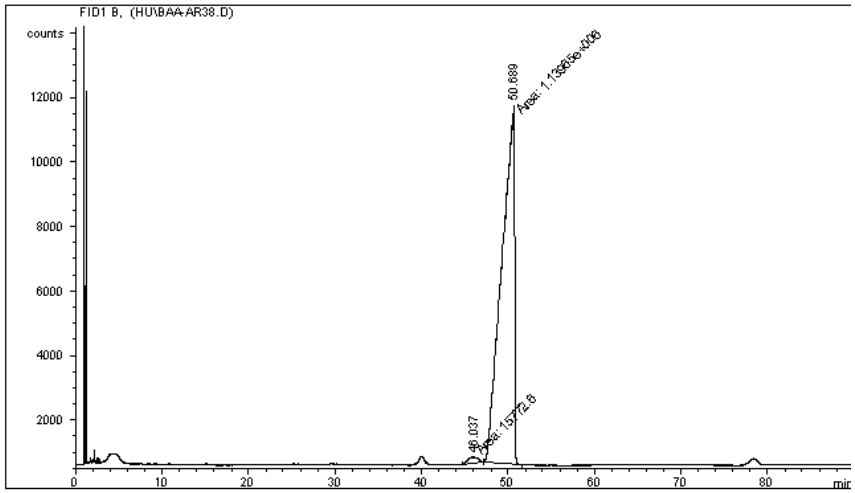



*Rac*-**5c**

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   2  50.689 MM    1.7061 1.13965e6  1.11328e4  98.63491

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 Results obtained with enhanced integrator!
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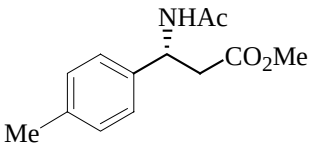
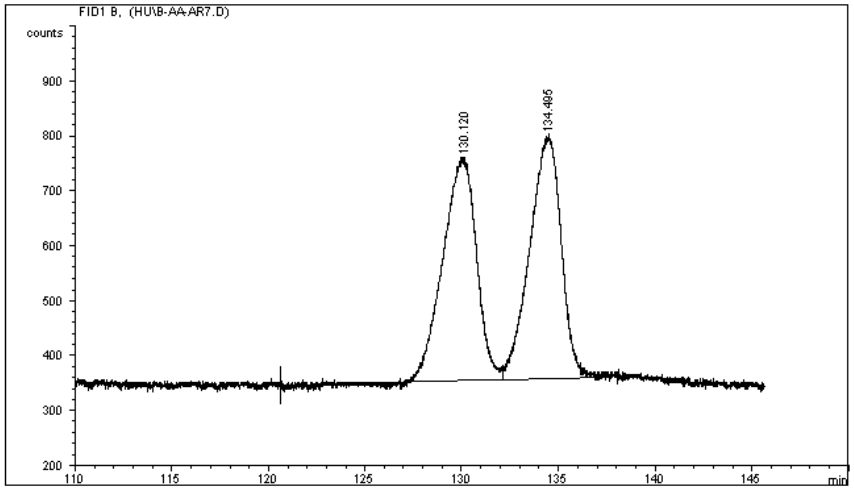



**5c**

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 Results obtained with enhanced integrator!
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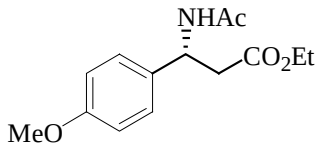
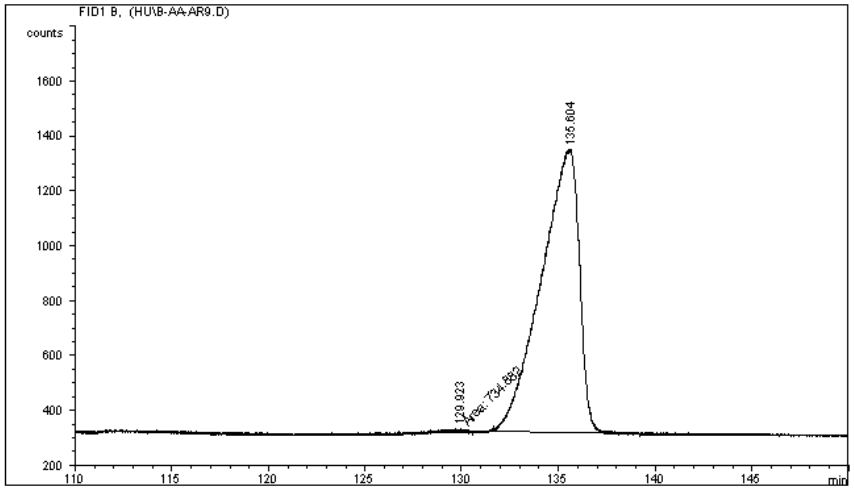



*Rac*-**5d**

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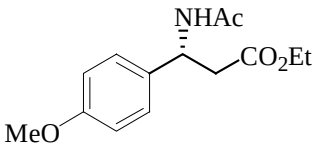
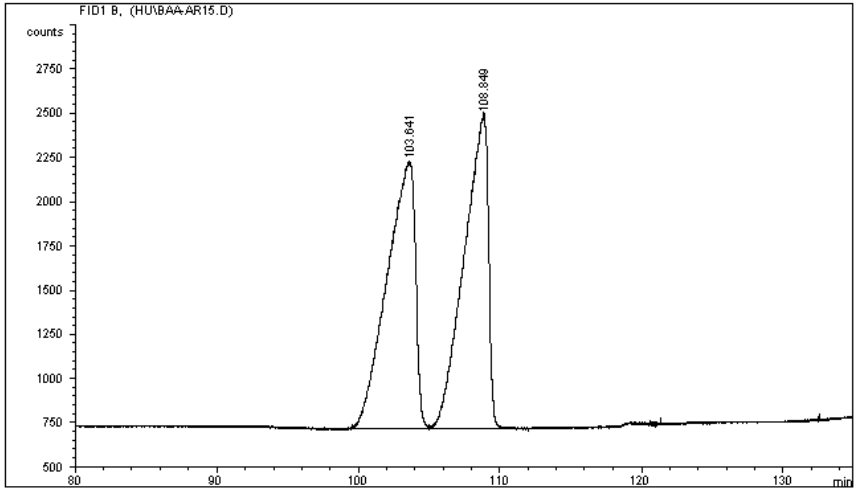
----|-------|----|-------|----------|----------|--------|
   1 129.923 MM    0.9587  734.88165   12.77611  0.51977
   2 135.604 BB    1.6318 1.40652e5  1028.26294 99.48023

Totals :                  1.41387e5  1041.03905

 Results obtained with enhanced integrator!
================================================================================
```




**5d**




```
=====================================================================
                      Area Percent Report
=====================================================================

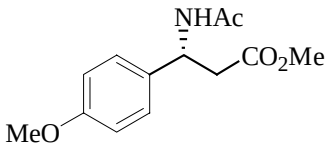
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)


Signal 1: FID1 B,

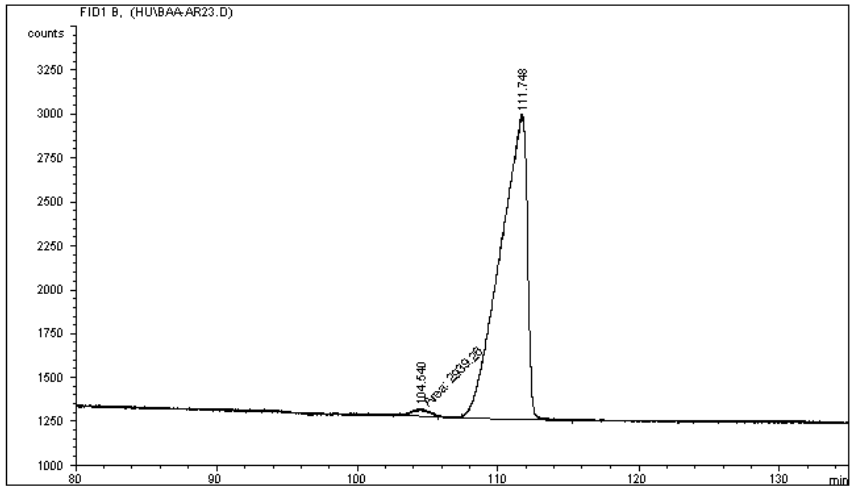
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  counts*s   [counts]      %
----|-------|----|-------|----------|----------|--------|
   1 103.641 BV    1.6578 2.10745e5  1508.57861 49.83825
   2 108.849 VP    1.4408 2.12113e5  1766.60559 50.16175

Totals :                  4.22857e5  3275.18420

 Results obtained with enhanced integrator!
=====================================================================
```




*Rac*-**5e**




```
=====================================================================
                      Area Percent Report
=====================================================================

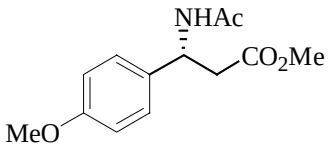
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)


Signal 1: FID1 B,

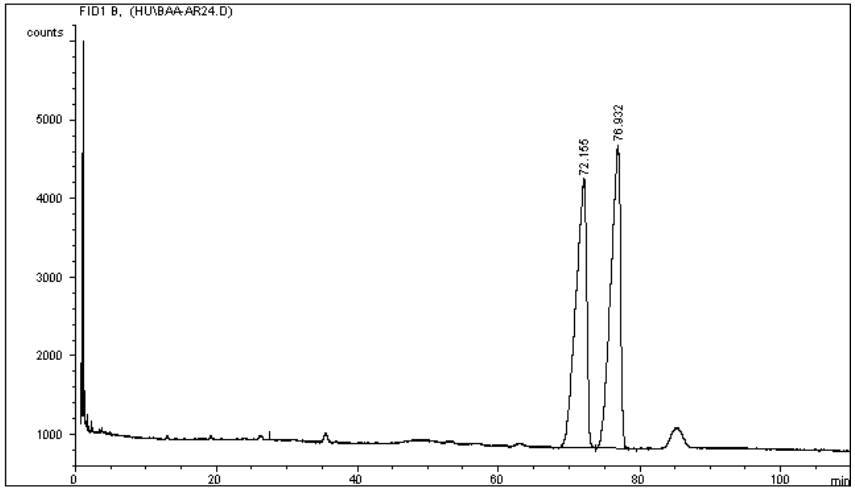
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  counts*s   [counts]      %
----|-------|----|-------|----------|----------|--------|
   1 104.540 MM    1.3355 2939.26367   36.68153  1.26118
   2 111.748 BB    1.5805 2.30118e5  1729.28931 98.73882

Totals :                  2.33057e5  1765.97084

 Results obtained with enhanced integrator!
=====================================================================
```




**5e**




```
=====================================================================
                        Area Percent Report
=====================================================================

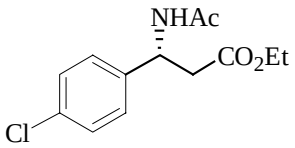
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)

Signal 1: FID1 B,

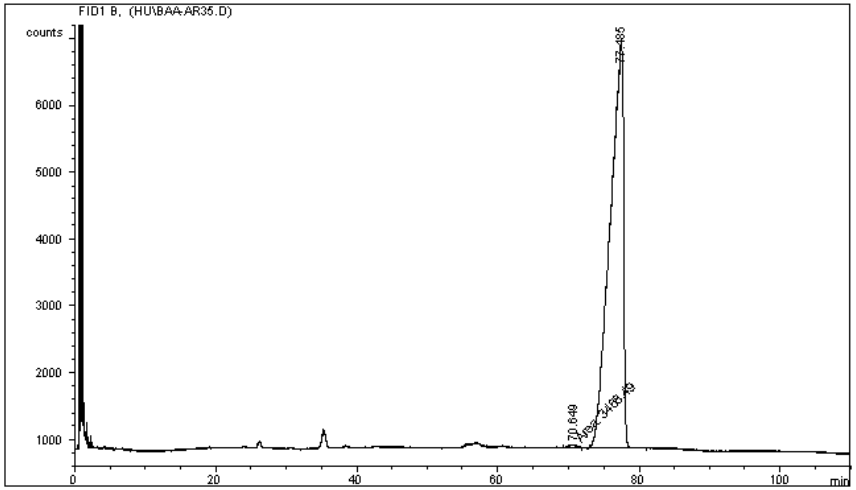
Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]  counts*s   [counts]      %
----|-------|----|-------|----------|----------|--------|
   1  72.155 BV    1.3965 3.69866e5  3412.37964 49.97074
   2  76.932 VP    1.1539 3.70299e5  3850.62427 50.02926

Totals :                  7.40164e5  7263.00391

 Results obtained with enhanced integrator!
=====================================================================
```




*Rac*-**5f**




```
=====================================================================
                        Area Percent Report
=====================================================================

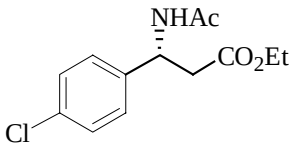
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)

Signal 1: FID1 B,

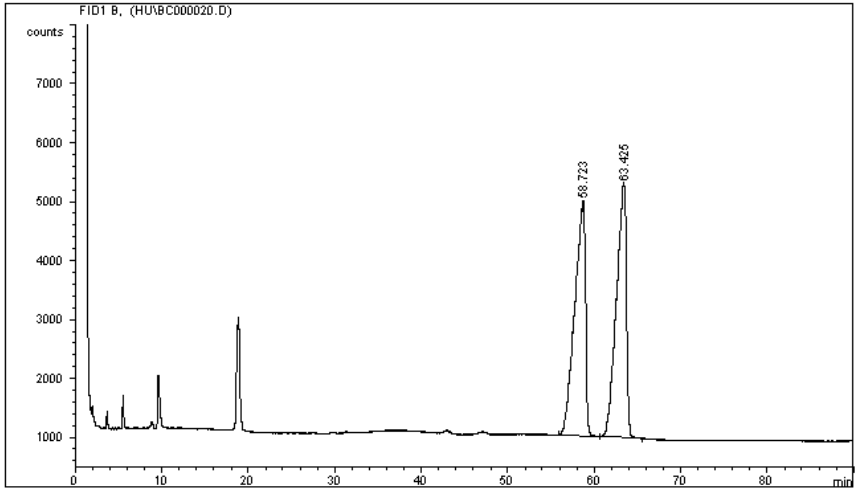
Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]  counts*s   [counts]      %
----|-------|----|-------|----------|----------|--------|
   1  70.649 MM    1.4006 3468.49268   41.27474  0.42730
   2  77.485 BB    1.5967 8.08257e5  6041.79639 99.57270

Totals :                  8.11725e5  6083.07113

 Results obtained with enhanced integrator!
=====================================================================
```




**5f**




```
=====================================================================
                      Area Percent Report
=====================================================================

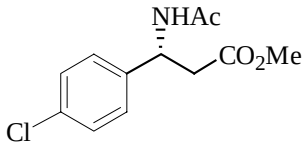
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)


Signal 1: FID1 B,

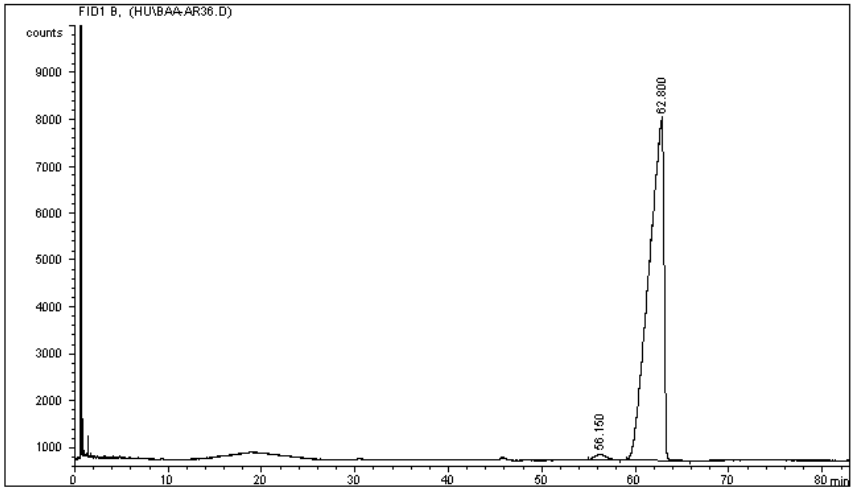
Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]  counts*s   [counts]      %
----|-------|----|-------|----------|----------|--------|
   1  58.723 BV    1.1328 3.49108e5  3974.31128 50.16878
   2  63.425 VB    1.1002 3.46759e5  4284.45605 49.83122

Totals :                  6.95867e5  8258.76733

 Results obtained with enhanced integrator!
=====================================================================
```




*Rac*-**5g**




```
=====================================================================
                      Area Percent Report
=====================================================================

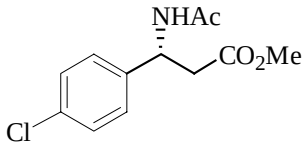
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)


Signal 1: FID1 B,

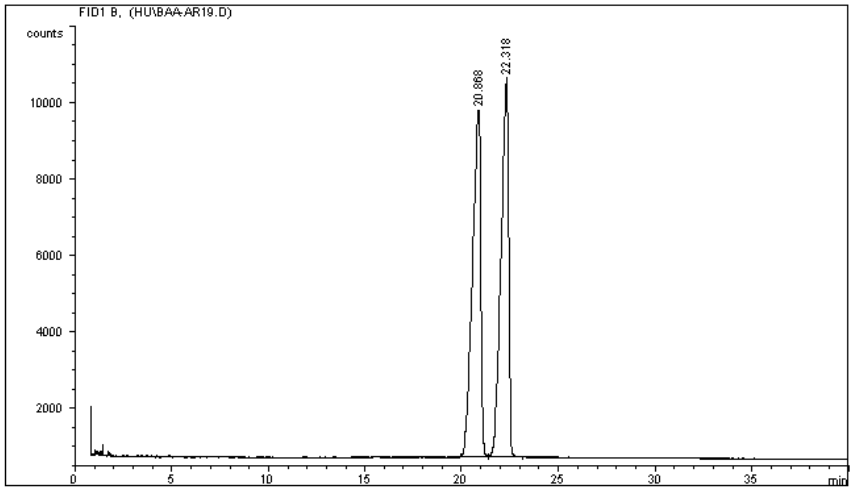
Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]  counts*s   [counts]      %
----|-------|----|-------|----------|----------|--------|
   1  56.150 BB    1.0410 1.05539e4   120.12556  1.28178
   2  62.800 BP    1.3906 8.12826e5  7302.35791 98.71822

Totals :                  8.23380e5  7422.48347

 Results obtained with enhanced integrator!
=====================================================================
```




**5g**




```
=====================================================================
                        Area Percent Report
=====================================================================

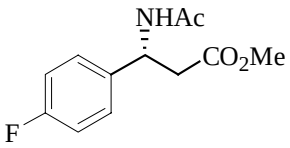
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)

Signal 1: FID1 B,

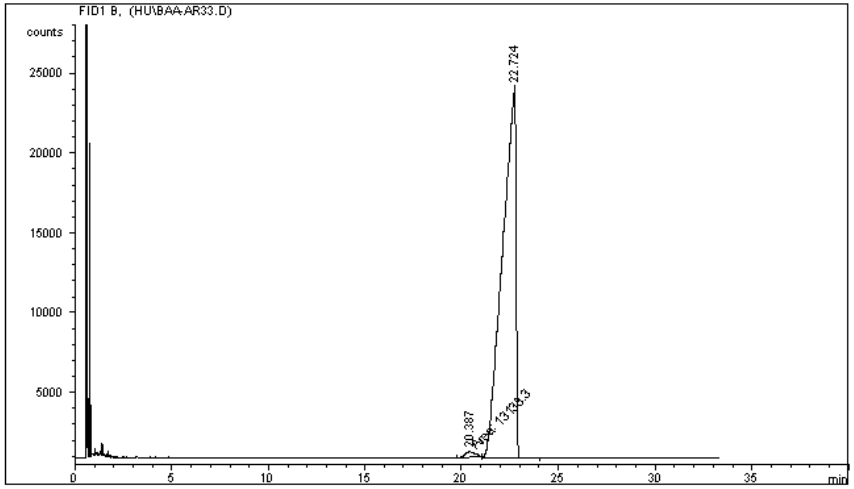
Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]  counts*s   [counts]      %
----|-------|----|-------|----------|----------|--------|
   1  20.868 BV    0.4728 2.70702e5  9053.41211 50.00410
   2  22.318 VB    0.3907 2.70658e5  9909.35840 49.99590

Totals :                  5.41360e5  1.89628e4

 Results obtained with enhanced integrator!
=====================================================================
```




*Rac*-**5h**




```
=====================================================================
                        Area Percent Report
=====================================================================

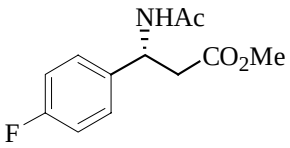
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)

Signal 1: FID1 B,

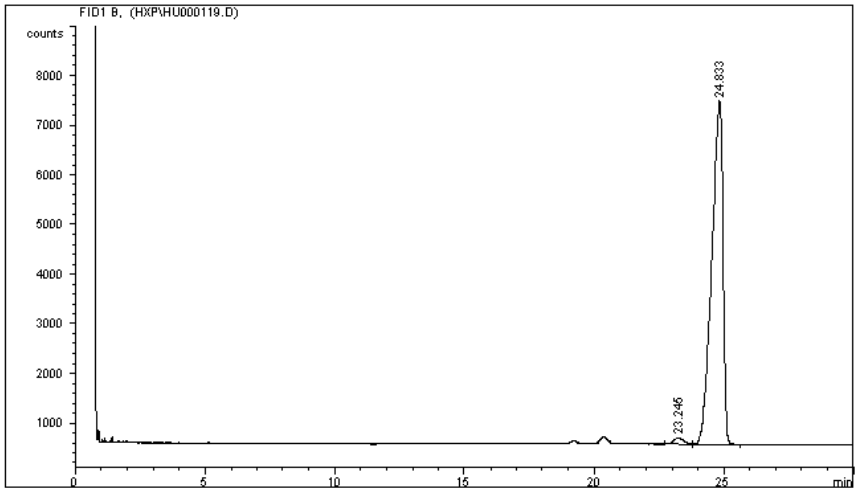
Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]  counts*s   [counts]      %
----|-------|----|-------|----------|----------|--------|
   1  20.387 MM    0.6337 1.31383e4   345.54306  1.15795
   2  22.724 VB    0.6109 1.12148e6  2.32451e4  98.84205

Totals :                  1.13462e6  2.35906e4

 Results obtained with enhanced integrator!
=====================================================================
```




**5h**




```
=====================================================================
                        Area Percent Report
=====================================================================

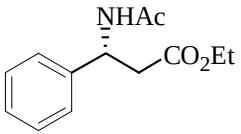
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)

Signal 1: FID1 B,

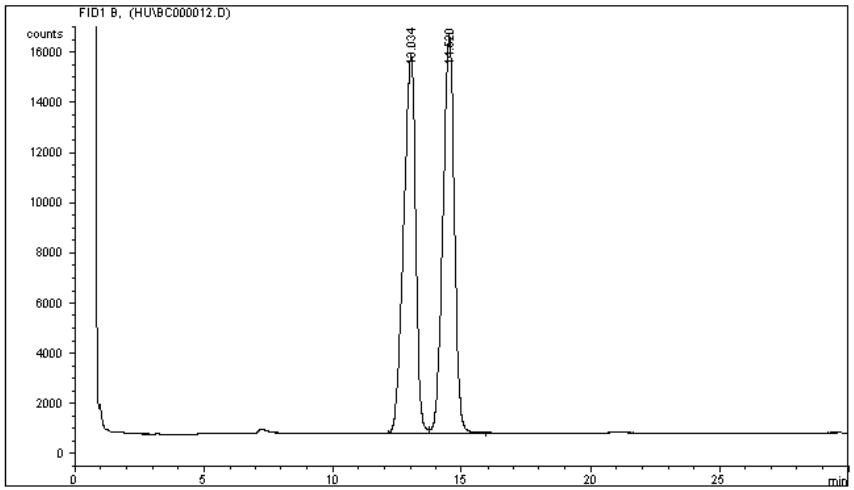
Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]  counts*s   [counts]      %
----|-------|----|-------|----------|----------|--------|
   1  23.245 BV    0.4197 3666.40552  133.78531  1.74379
   2  24.833 VB    0.4495 2.06589e5  6896.34082 98.25621

Totals :                  2.10255e5  7030.12613

 Results obtained with enhanced integrator!
=====================================================================
```




**5a** (0.02 mol% of Rh)




```
=====================================================================
                        Area Percent Report
=====================================================================

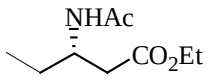
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)

Signal 1: FID1 B,

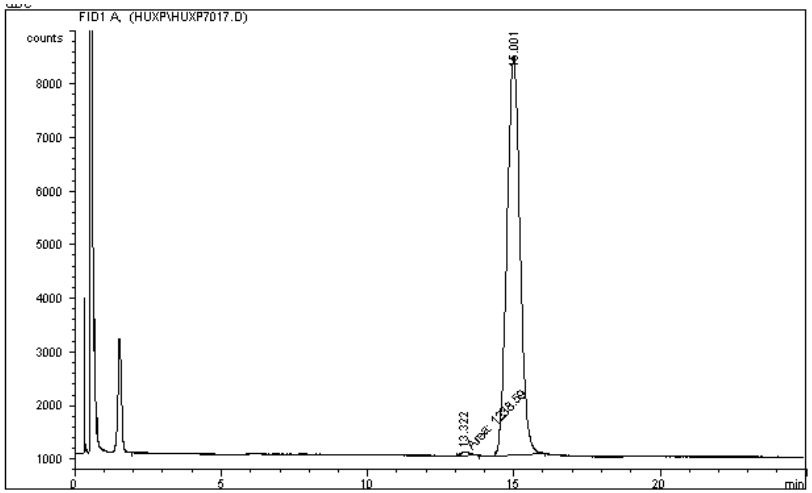
Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]  counts*s   [counts]      %
----|-------|----|-------|----------|----------|--------|
   1  13.034 BV    0.5032 4.85549e5  1.50988e4  49.91984
   2  14.520 VB    0.4794 4.87108e5  1.58133e4  50.08016

Totals :                  9.72657e5  3.09122e4

 Results obtained with enhanced integrator!
=====================================================================
```




*Rac*-**6d**




```
=====================================================================
                        Area Percent Report
=====================================================================

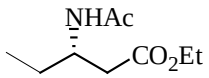
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

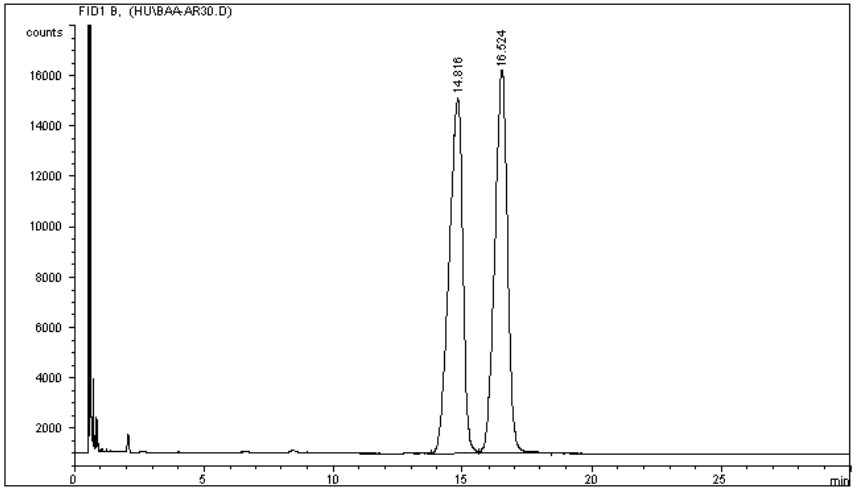
Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]  counts*s   [counts]      %
----|-------|----|-------|----------|----------|--------|
   1  13.322 MM    0.3398 1238.59106   60.74482  0.53540
   2  15.001 BB    0.4661 2.30102e5  7413.61719 99.46460

Totals :                  2.31341e5  7474.36200

 Results obtained with enhanced integrator!
=====================================================================
```




**6d**




```
=====================================================================
                        Area Percent Report
=====================================================================

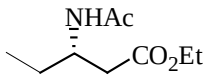
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)

Signal 1: FID1 B,

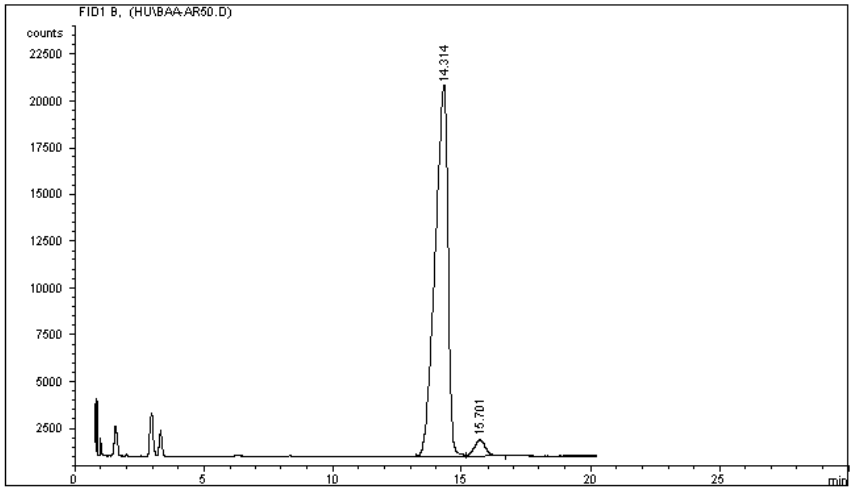
Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]  counts*s   [counts]      %
----|-------|----|-------|----------|----------|--------|
   1  14.816 BV    0.5590 5.16301e5  1.41071e4  49.80261
   2  16.524 VB    0.5241 5.20394e5  1.51758e4  50.19739

Totals :                  1.03669e6  2.92829e4

 Results obtained with enhanced integrator!
=====================================================================
```




***Rac*-6d**




```
=====================================================================
                        Area Percent Report
=====================================================================

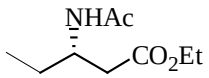
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)

Signal 1: FID1 B,

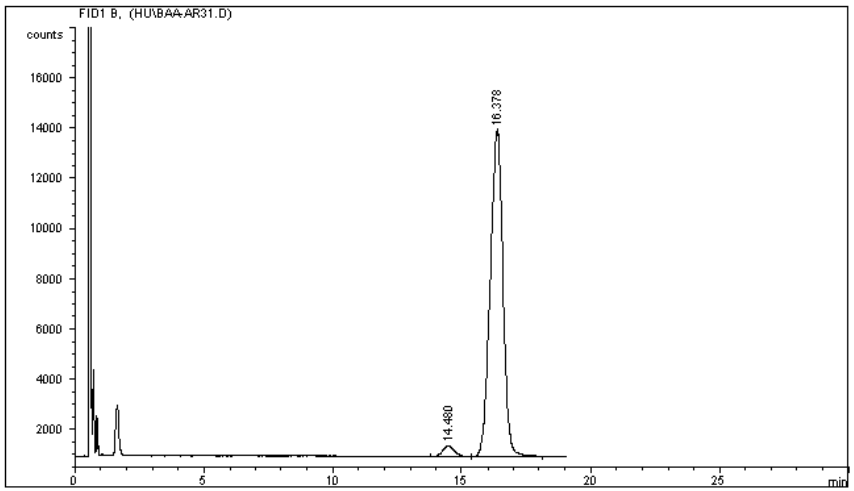
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  counts*s   [counts]      %
----|-------|----|-------|---------|---------|--------|
   1  14.314 BV    0.5109 6.90265e5  1.97917e4  96.30860
   2  15.701 VP    0.4846 2.64570e4   846.60614  3.69140

Totals :                  7.16722e5  2.06383e4

 Results obtained with enhanced integrator!
=====================================================================
```




**6d**




```
=====================================================================
                        Area Percent Report
=====================================================================

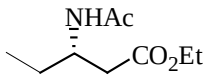
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)

Signal 1: FID1 B,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  counts*s   [counts]      %
----|-------|----|-------|---------|---------|--------|
   1  14.480 PP    0.4953 1.27390e4   409.05493  2.79027
   2  16.378 VB    0.5313 4.43810e5  1.29670e4  97.20973

Totals :                  4.56549e5  1.33761e4

 Results obtained with enhanced integrator!
=====================================================================
```




**6d**