Supplemental Materials for:

The Palladium-Catalyzed Oxidative Kinetic Resolution of Secondary Alcohols with Molecular Oxygen

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Additional References:

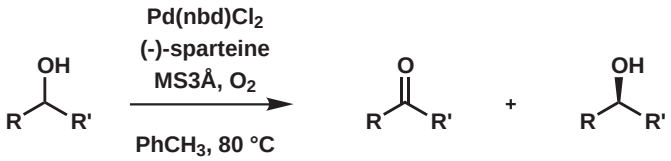
Footnote 16: Sparteine has been used as a ligand for palladium-catalyzed allylic substitution reactions and olefin polymerization reactions. (a) Trost, B. M.; Dietsch, T. J. *J. Amer. Chem. Soc.* **1973**, *95*, 8200. (b) Togni, A.; Rihs, G.; Pregosin, P. S.; Ammann, C. *Helv. Chim. Acta* **1990**, *73*, 723. (c) Togni, A. *Tetrahedron: Asymmetry* **1991**, *2*, 683. (d) Pregosin, P. S.; Ruegger, H. *Magn. Reson. Chem.* **1994**, *32*, 297. (e) Dani, P.; Dupont, J.; Monteiro, A. L. *J. Braz. Chem. Soc.* **1996**, *7*, 15. (f) Rush, S.; Reinmuth, A.; Risse, W. *Macromolecules* **1997**, *30*, 7375. (g) Mathews, N.; Hager, H.; Rush, S.; Risse, W. *Polym. Mater. Sci. Eng.* **1999**, *80*, 435.

For a recent account describing the use of (–)-sparteine for dynamic thermodynamic resolutions, see: Beak, P.; Anderson, D. R.; Curtis, M. D.; Laumer, J. M.; Pippel, D. J.; Weisenburger, G. A. *Acc. Chem. Res.* **2000**, *33*, 715.

Material and Methods. Unless stated otherwise, reactions were performed in flame-dried glassware under a nitrogen or an argon atmosphere, using freshly distilled solvents. All other commercially obtained reagents were used as received. Reaction temperatures were controlled by an IKAmag temperature modulator. Thin-layer chromatography (TLC) was performed using E. Merck silica gel 60 F254 precoated plates (0.25 mm). ICN Silica gel (particle size 0.032-0.063 mm) was used for flash chromatography. Analytical chiral HPLC was performed on a Chiralcel OJ, AS, or OD-H column (each is 4.6 mm x 25 cm) obtained from Daicel Chemical Industries, Ltd. Analytical achiral GC was performed using an Agilent DB-WAX (30.0 m x 0.25 m) column. Analytical chiral GC was carried out using a Chiraldex B-DM column (30.0 m x 0.25 mm) purchased from Bodman Industries. Commercially available racemic alcohols in Table 3 (entries 1, 2, 3, 5, 7, 8, and 9) were purchased from the Sigma-Aldrich Chemical Company, Milwaukee, WI. Non-commercially available racemic alcohols used in Table 3 (corresponding to entries 4, 6, and 10) were prepared as previously described.[1] Commercially available samples of enantiopure alcohols for analytical comparison purposes (entries 1, 4, 7, 8, and 9) were purchased from the Sigma-Aldrich Chemical Company, Milwaukee, WI. Non-commercially available enantiopure alcohols prepared

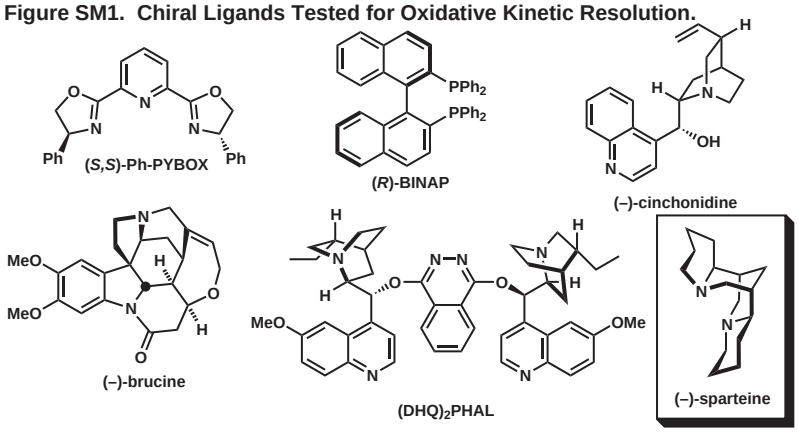
[1] (a) Ruble, J. C.; Latham, H. A.; Fu, G. C. *J. Am. Chem. Soc.* **1997**, *119*, 1492. (b) Ruble, J. C., Tweddell, J.; Fu, G. C. *J. Org. Chem.* **1998**, *63*, 2794.

by palladium-catalyzed oxidative kinetic resolution (Table 3 entries 2,[2] 3,[3] 5,[4] 6,[5] and 10[6,1]) were compared by optical rotation to known values.

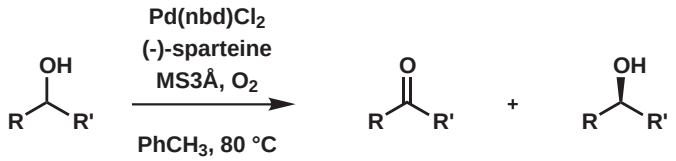


General Procedure for the Oxidative Kinetic Resolution of Secondary Alcohols. Ligand and Palladium Source Screening Trials. A 25 mL Schlenk flask equipped with a magnetic stir bar was charged with powdered molecular sieves (MS3Å, 0.25 g) and flame-dried under vacuum. After cooling under dry N_2, Pd complex (0.025 mmol, 0.05 equiv)[7] was added followed by toluene (5.0 mL), and an appropriate ligand (0.10 mmol, 0.20 equiv). [8] The flask was vacuum evacuated and filled with O_2 (3x, balloon), and the reaction mixture was heated to 80 °C for 10 min. The alcohol (0.50 mmol, 1.0 equiv) was introduced and the reaction monitored by standard analytical techniques (TLC, GC, ^{1}H-NMR, and HPLC) for % conversion and enantiomeric excess values. Aliquots of the reaction mixture (0.2 mL) were collected after 24 h, 40 h, 72 h, 96 h, 120 h, and 144 h depending on the course of the reaction (typically three aliquots per run). Each aliquot was filtered through a small plug of silica gel (EtOAc eluent), evaporated and analyzed.[9]

Figure SM1. Chiral Ligands Tested for Oxidative Kinetic Resolution.

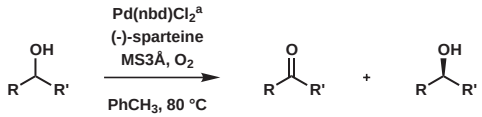


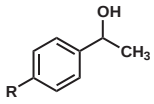
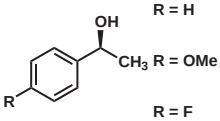
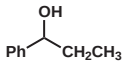
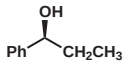
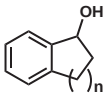
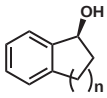
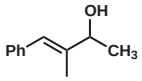
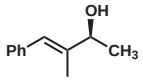
[2] Nakamura, K.; Inoue, Y.; Matsuda, T.; Misawa, I. *J. Chem. Soc., Perkin. Trans. 1* **1999**, 2397.
[3] Nieduzak, T. R.; Margolin, A. L. *Tetrahedron: Asymmetry* **1991**, *2*, 113.
[4] Bakker, M.; Spruijt, A. S.; van Rantwijk, F.; Sheldon, R. A. *Tetrahedron: Asymmetry* **2000**, *11*, 1801.
[5] Nakamura, K.; Matsuda, T. *J. Org. Chem.* **1998**, *63*, 8957.
[6] Argus, C. L.; Cort, L. A.; Howard, T. J.; Loc, L. B. *J. Chem. Soc.* **1960**, 1195.
[7] For experiments that probed the effect of palladium source, the appropriate Pd complex was used in the same general procedure.
[8] For experiments which probed the effect of chiral ligand, the appropriate ligand was used in the same general procedure with $Pd(OAc)_2$. The structures of all chiral ligands tested are provided in Figure SM1.
[9] Percent conversions were measured by GC integration of the alcohol and the ketone peaks, correcting for response factors (for conditions see SM Table 2).



General Procedure for the Oxidative Kinetic Resolution of Secondary Alcohols. Preparative Runs (6.0 mmol) in Table 3. A 200 mL flask equipped with a magnetic stir bar was charged with powdered molecular sieves (MS3Å, 3.0 g) and flame-dried under vacuum. After cooling under dry N_2, $Pd(nbd)Cl_2$ (80.8 mg, 0.30 mmol, 0.05 equiv) was added followed by toluene (60.0 mL), and (–)-sparteine (276 μL, 1.20 mmol, 0.20 equiv). The flask was vacuum evacuated and filled with O_2 (3x, balloon), and the reaction mixture was heated to 80 °C for 10 min. The racemic alcohol (6.00 mmol, 1.0 equiv) was introduced and the reaction monitored by standard analytical techniques (TLC, GC, ^{1}H-NMR, and HPLC) for % conversion and enantiomeric excess values. Aliquots of the reaction mixture (0.2 mL) were collected after 24 h, 40 h, 72 h, 96 h, 120 h, and 144 h depending on the course of the reaction (typically three aliquots per run). Each aliquot was filtered through a small plug of silica gel (EtOAc eluent), evaporated and analyzed. Upon completion of the reaction, the reaction mixture was filtered through a pad of SiO_2 (EtOAc eluent) and purified by column chromatography on SiO_2 (see below for details).

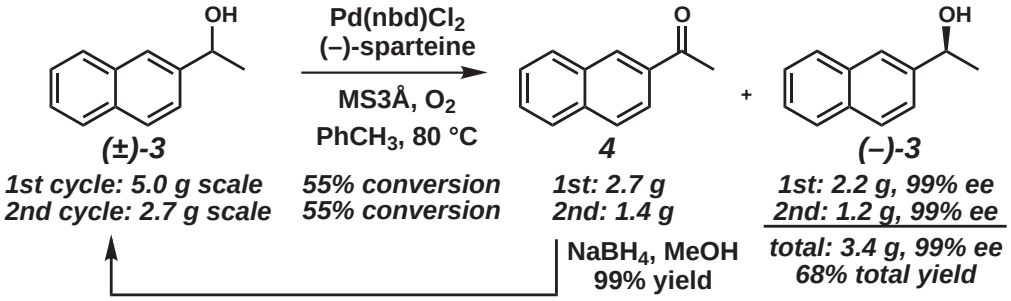
General Procedure for the Oxidative Kinetic Resolution of Secondary Alcohols. Preparative Runs (8.0 mmol) in Table 3. A 200 mL flask equipped with a magnetic stir bar was charged with powdered molecular sieves (MS3Å, 4.0 g) and flame-dried under vacuum. After cooling under dry N_2, $Pd(nbd)Cl_2$ (108 mg, 0.40 mmol, 0.05 equiv) was added followed by toluene (80.0 mL), and (–)-sparteine (368 μL, 1.60 mmol, 0.20 equiv). The flask was vacuum evacuated and filled with O_2 (3x, balloon), and the reaction mixture was heated to 80 °C for 10 min. The alcohol (8.00 mmol, 1.0 equiv) was introduced and the reaction monitored by standard analytical techniques (TLC, GC, ^{1}H-NMR, and HPLC) for % conversion and enantiomeric excess values. Aliquots of the reaction mixture (0.2 mL) were collected after 24 h, 40 h, 72 h, 96 h, 120 h, and 144 h depending on the course of the reaction (typically three aliquots per run). Each aliquot was filtered through a small plug of silica gel (EtOAc eluent), evaporated and analyzed. Upon completion of the reaction, the reaction mixture was filtered through a pad of SiO_2 (EtOAc eluent) and purified by column chromatography on SiO_2 (see below for details).



entry	racemic alcohol	amount	time	conversion	chromatography eluent	isolated yield of ketone	unreacted alcohol, major enantiomer	isolated yield ROH	ee ROH[b]	s[c,d]
1.	 R = H	0.977 g (8.00 mmol)	96 h	59.9%	6:1→3:1 hexane/EtOAc	0.535 g (56%)	 R = H	0.366 g (37%)	98.7%	23.1
2.	R = OMe	1.22 g (8.00 mmol)	96 h	66.6%	6:1→3:1 hexane/EtOAc	0.773 g (64%)	R = OMe	0.392 g (32%)	98.1%	12.3
3.	R = F	1.12 g (8.00 mmol)	54 h	63.3%	6:1→3:1 hexane/EtOAc	0.623 g (56%)	R = F	0.361 g (32%)	97.4%	14.4
4.	Ar = 1-Naphthyl	1.03 g (6.00 mmol)	192 h	55.9%	6:1→3:1 hexane/EtOAc	0.555 g (54%)	Ar = 1-Naphthyl	0.443 g (43%)	78.4%	9.8
5.	 Ar = 2-Naphthyl	5.00 g (29.00 mmol)	112 h	55.2%	6:1→3:1 hexane/EtOAc	2.75 g (55%)	 Ar = 2-Naphthyl	2.20 g (44%)	99.0%	47.1
6.	Ar = *o*-tolyl	1.09 g (8.00 mmol)	144 h	48.4%	6:1→3:1 hexane/EtOAc	0.492 g (46%)	Ar = *o*-tolyl	0.533 g (49%)	68.7%	13.1
7.		1.09 g (8.00 mmol)	192 h	59.3%	6:1→4:1 hexane/EtOAc	0.625 g (58%)		0.435 g (40%)	93.1%	14.8
8.	 n = 1	1.07 g (8.00 mmol)	54 h[e]	67.5%	6:1→3:1 hexane/EtOAc	0.662 g (63%)	 n = 1	0.323 g (30%)	93.4%	8.3
9.	n = 2	1.19 g (8.00 mmol)	40 h	68.6%	9:1→4:1 hexane/EtOAc	0.796 g (68%)	n = 2	0.370 g (31%)	99.8%	15.8
10.		0.973 g (6.00 mmol)	120 h	70.4%	6:1→3:1 hexane/EtOAc	0.671 g (70%)		0.286 g (29%)	91.8%	6.6

[a]5 mol% Pd(nbd)Cl_2, 20 mol% (–)-sparteine, 1 atm O_2. [b]The degree of enantioselectivity was measured directly by chiral HPLC or GC of the recovered alcohols.[10] [c]Selectivity (s) values represent an average of at least two experiments, while conversion and ee values are for specific cases. [d]For each entry, comparable selectivities are observed throughout the course of the run. [e]Experiment performed at 60 °C.

[10] Enantiomeric excess was measured by chiral HPLC analysis using either a Chiralcel OJ, AS or OD-H column or by chiral GC using a Bodman Chiraldex B-DM column. Conversion was measured by GC using a DB-WAX column.

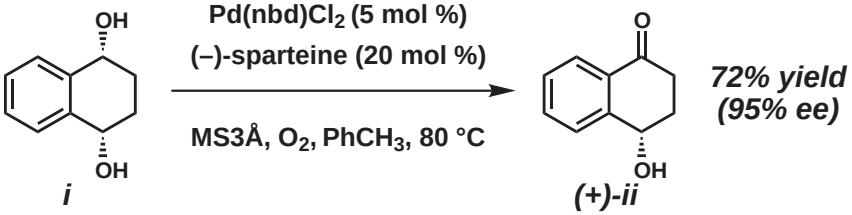


Scale-up Procedure for the Two Cycle Oxidative Kinetic Resolution of α-methyl-2-naphthalenemethanol 3. 1st cycle: A 500 mL round bottom flask was charged with powdered molecular sieves (MS3Å, 14.5 g) and a magnetic stir bar and flame-dried under vacuum. After cooling under dry N_2, Pd(nbd)Cl_2 (0.391 g, 1.45 mmol, 0.05 equiv) was added followed by toluene (290 mL), and (–)-sparteine (1.34 mL, 5.81 mmol, 0.20 equiv). The flask was vacuum evacuated and filled with O_2 (3x, balloon), and the reaction mixture was heated to 80 °C for 10 min. Alcohol **(±)-3** (5.00 g, 29.0 mmol, 1.0 equiv) was introduced and the reaction mixture heated at 80 °C for 112 h. Progress of the reaction was monitored by standard analytical techniques (TLC, GC, ^{1}H-NMR, and HPLC) for % conversion and enantiomeric excess values by the removal of small aliquots of the reaction mixture (0.2 mL) which were filtered through silica gel (EtOAc eluent), evaporated and analyzed. After the reaction rate had significantly slowed (112 h, 55% conversion), and aliquot analysis showed high levels of enantiocontrol for the remaining alcohol **(–)-3** (99.0% ee), the entire reaction mixture was filtered through a small column of silica gel (5 x 6 cm, EtOAc eluent). The filtrate was evaporated and purified by flash chromatography on silica gel (6:1→3:1 hexanes/EtOAc eluent) to provide ketone **4** (R_F = 0.56, 2.75 g, 55% yield) and alcohol **(–)-3** (R_F = 0.44, 2.20 g, 44% yield, 99.0% ee) as white solids.

Regeneration of alcohol (±)-3. A cooled (0 °C) solution of ketone **4** (2.75 g, 16.2 mmol, 1.0 equiv) in 1:1 CH_2Cl_2/MeOH (16.2 mL) was treated with $NaBH_4$ (733 mg, 19.4 mmol, 1.2 equiv) in four portions over 10 min. The reaction was stirred at 0 °C for 15 min, and treated with 1 N HCl solution (30 mL) slowly over 15 min. After the evolution of gas was complete, the layers were separated, and the aqueous layer extracted with CH_2Cl_2 (3 x 30 mL). The combined organic layers were dried over $MgSO_4$, evaporated, and purified by flash chromatography on silica gel (3:1 hexanes/EtOAc eluent) to provide alcohol **(±)-3** (2.76 g, 99% yield) as a white solid, which was used in cycle two.

2nd cycle: A 500 mL round bottom flask was charged with Molecular Sieves (MS3Å, 8.0 g) and flame-dried under vacuum. After cooling under dry N_2, Pd(nbd)Cl_2 (0.216 g, 0.800 mmol, 0.05 equiv) was added followed by toluene (160 mL), and (–)-sparteine (0.735 mL, 3.20 mmol, 0.20 equiv). The flask was vacuum evacuated and filled with O_2 (3x, balloon), and the reaction mixture was heated to 80 °C for 10 min. Alcohol **(±)-3** (2.76 g, 16.0 mmol, 1.0 equiv) prepared above was introduced and the reaction mixture heated at 80 °C for 96 h. Progress of the reaction was monitored by standard analytical techniques (TLC, GC, ^{1}H-NMR, and HPLC) for % conversion and enantiomeric excess values by the removal of small

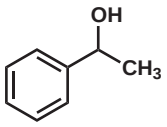
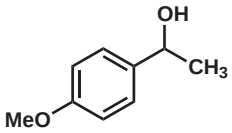
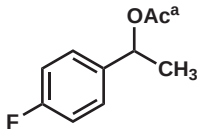
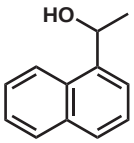
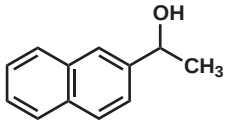
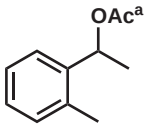
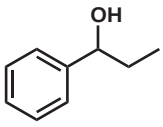
aliquots (0.2 mL) which were filtered through silica gel (EtOAc eluent), evaporated and analyzed. After the reaction rate had significantly slowed (81 h, 55% conversion), and aliquot analysis showed high levels of enantiocontrol for the remaining alcohol **(–)-3** (99.0% ee), the entire reaction mixture was filtered through a small column of silica gel (5 x 6 cm, EtOAc eluent). The filtrate was evaporated and purified by flash chromatography on silica gel (6:1→3:1 hexanes/EtOAc eluent) to provide ketone **4** (1.43 g, 54% yield) and alcohol **(–)-3** (1.20 g, 44% yield, 99.0% ee) as white solids. The combination of both cycles provided alcohol **(–)-3** (3.39 g, 68% yield, 99.0% ee).



Oxidative Desymmetrization of Meso Diol i. A 50 mL Schlenk flask equipped with a magnetic stir bar was charged with Molecular Sieves (MS3Å, 625 mg) and flame-dried under vacuum. After cooling under dry N_2, Pd(nbd)Cl_2 (16.8 mg, 0.0625 mmol, 0.05 equiv) was added followed by toluene (12.5 mL), and (–)-sparteine (57 μL, 0.25 mmol, 0.20 equiv). The flask was vacuum evacuated and filled with O_2 (3x, balloon), and the reaction mixture was heated to 80 °C for 10 min. Diol **i**[11] (205 mg, 1.25 mmol, 1.0 equiv) was introduced and the reaction monitored by standard analytical techniques (TLC, GC, ^{1}H-NMR, and HPLC) for % conversion and enantiomeric excess values. Upon completion of the reaction, the reaction mixture was filtered through a pad of SiO_2 (EtOAc eluent) and purified by column chromatography on SiO_2 (3:1→1:1 hexane/EtOAc eluent) to provide hydroxyketone **(+)-ii** as an oil (145 mg, 72% yield, 95% ee); $[\alpha]_D^{23}$ +19.6 (*c* 1.0, MeOH).[12] See Table SM1 for details regarding the ee assay.

[11] Diol **i** was prepared according to the procedure of Yamada, see: Yamada, S.; Katsumata, H. *J. Org. Chem.* **1999**, *64*, 9365.
[12] The assignment of absolute stereochemistry is based on analogy to the results in Table 3.

Table SM 1. Methods utilized for the determination of enantiomeric excess.

entry	Substrate	ee Assay	Conditions	Retention Time of (*R*) isomer (min)	Retention Time of (*S*) isomer (min)
1.		HPLC Chiralcel OD-H	3% EtOH/hexane 1.0 mL/min	10.69	13.37
2.		HPLC Chiralcel OD-H	3% EtOH/hexane 1.0 mL/min	14.60	16.52
3.		GC Chiraldex B-DM	50 °C, 0 min 5 °C/min to 200 °C 1.0 mL/min carrier gas flow	16.41	15.78
4.		HPLC Chiralcel OD-H	3% EtOH/hexane 1.0 mL/min	31.99	18.96
5.		HPLC Chiralcel OJ	4% 2-propanol/hexane 1.0 mL/min	38.69	31.32
6.		GC Chiraldex B-DM	85 °C, 45 min 1.0 mL/min carrier gas flow	42.17	40.71
7.		HPLC Chiralcel OD-H	3% EtOH/hexane 1.0 mL/min	11.15	13.23

a. Prepared by reaction of the alcohol with Ac_2O and pyridine.

Table SM 1 (continued).

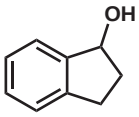
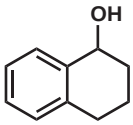
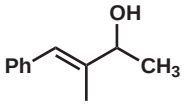
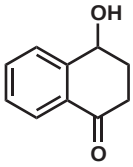
entry	Substrate	ee Assay	Conditions	Retention Time of (*R*) isomer (min)	Retention Time of (*S*) isomer (min)
8.		HPLC Chiralcel OJ	3% EtOH/hexane 1.0 mL/min	17.35	14.76
9.		HPLC Chiralcel AS	2% EtOH/hexane 1.0 mL/min	15.55	12.68
10.		HPLC Chiralcel OD-H	4% 2-propanol/hexane 1.0 mL/min	13.44	15.44
ii.		HPLC Chiralcel AS	6% 2-propanol/hexane 1.0 mL/min	37.97	30.44

Table SM 2. Selected Experimental Data for the Determination of Conversion, Enantiomeric Excess, and Selectivity (s).

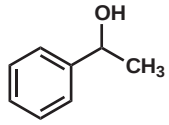
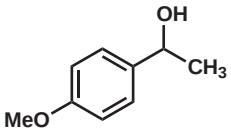
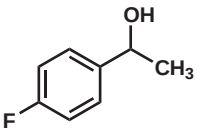
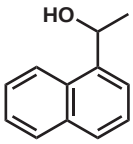
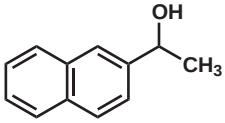
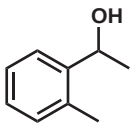
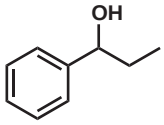
entry	Substrate	time (h)	% Conversion	Measured %ee, unreacted ROH	s
1.		19 40 96 96	35.7 47.4 59.9 57.1	48.6 75.7 98.7 96.6	24.3 26.1 23.1 24.8
2.		40 96 96 96 120	50.8 64.8 66.6 65.8 66.0	72.5 97.6 98.1 98.3 98.9	12.2 13.1 12.3 13.3 14.3
3.		48 54 60 72	63.9 63.3 65.7 65.2	96.1 97.4 96.9 97.9	12.3 14.4 11.6 13.2
4.		40 144 144 168 192	26.5 47.4 47.4 54.5 55.9	27.3 62.2 61.8 76.6 78.4	9.4 10.2 10.0 10.2 9.8
5.		81 112	55.1 55.2	99.0 99.0	48.0 47.1
6.		96 96 144 144	34.2 40.5 39.5 48.4	41.6 52.0 48.7 68.7	13.5 12.5 11.1 13.1
7.		40 48 96 96 192	30.3 41.6 57.2 55.7 59.3	34.4 55.0 89.0 86.8 93.1	12.0 13.4 14.4 15.0 14.8

Table SM 2 (continued).

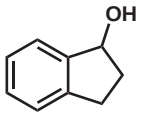
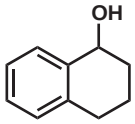
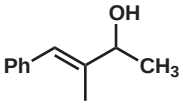
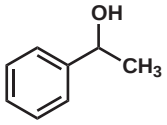
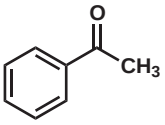
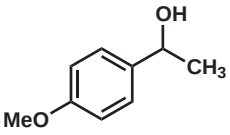
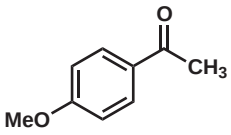
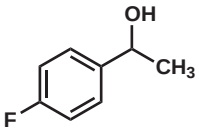
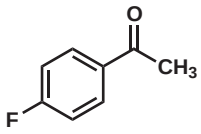
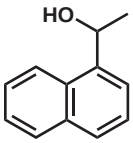
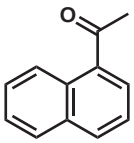
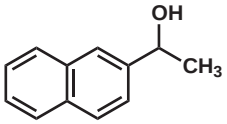
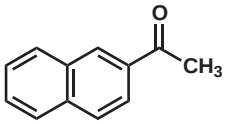
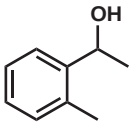
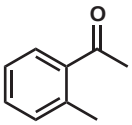
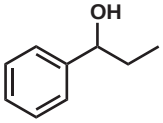
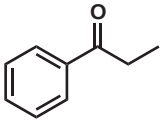
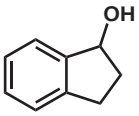
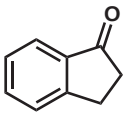
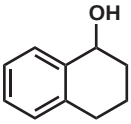
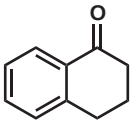
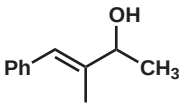
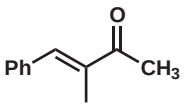
entry	Substrate	time (h)	% Conversion	Measured %ee, unreacted ROH	s
8.		48 54 96	65.2 67.5 68.0	92.5 93.4 90.0	9.1 8.3 6.9
9.		40 40 48 96 96	68.6 59.9 67.6 68.7 69.3	99.8 95.2 99.7 99.9 99.9	15.8 16.1 15.9 17.2 16.6
10.		40 96 120 144	46.0 66.2 70.4 68.4	54.5 85.9 91.8 90.7	7.7 6.6 6.6 7.0

Table SM 3. Methods utilized for the determination of % conversion.

entry	alcohol	ketone	GC Conditions[a]	Retention Time of alcohol (min)	Retention Time of ketone (min)
1.			70 °C, 15 min; 7.0 °C/min to 220 °C 1.0 mL/min carrier gas flow	29.03	26.02
2.			70 °C, 15 min; 7.0 °C/min to 220 °C 1.0 mL/min carrier gas flow	34.82	33.90
3.			70 °C, 15 min; 7.0 °C/min to 220 °C 1.0 mL/min carrier gas flow	29.82	25.93
4.			70 °C, 0 min; 3.0 °C/min to 270 °C 1.0 mL/min carrier gas flow	50.74	44.91
5.			70 °C, 0 min; 3.0 °C/min to 270 °C 1.0 mL/min carrier gas flow	36.17	35.96
6.			70 °C, 15 min; 7.0 °C/min to 220 °C 1.0 mL/min carrier gas flow	31.01	26.68
7.			70 °C, 15 min; 7.0 °C/min to 220 °C 1.0 mL/min carrier gas flow	30.06	27.43

[a]All assays performed on Agilent DB-WAX column.

Table SM 3 (continued).

entry	alcohol	ketone	GC Conditions[a]	Retention Time of alcohol	Retention Time of ketone
8.			70 °C, 15 min; 7.0 °C/min to 220 °C 1.0 mL/min carrier gas flow	33.12	32.20
9.			70 °C, 15 min; 7.0 °C/min to 220 °C 1.0 mL/min carrier gas flow	34.90	33.39
10.			70 °C, 15 min; 5.0 °C/min to 220 °C 1.0 mL/min carrier gas flow	25.37	23.04

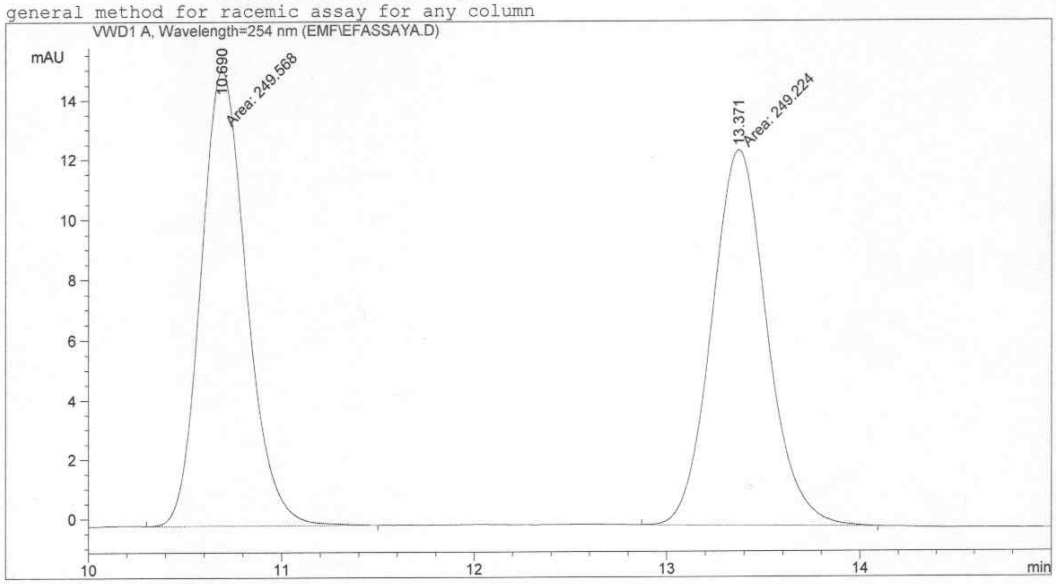
[a]All assays performed on Agilent DB-WAX column.

Selected HPLC traces for the data in Table 3 using the conditions outlined in Table SM3.

Table 3, entry 1 (racemic):

Data File D:\HPCHEM\1\DATA\EMF\EFASSAYA.D Sample Name: emf-assay-A

```
==========================================================================
Injection Date  : 5/28/01 01:49:50 PM          Seq. Line :   2
Sample Name     : emf-assay-A                       Vial :  61
Acq. Operator   : eric                               Inj :   1
                                                Inj Volume : 5 µl

Acq. Method     : D:\HPCHEM\2\METHODS\3-EOH30.M
Last changed    : 9/5/00 16:36:56 PM by sean
Analysis Method : D:\HPCHEM\2\METHODS\4-IPA100.M
Last changed    : 6/10/01 08:41:30 PM by Joel
                  (modified after loading)
general method for racemic assay for any column
```

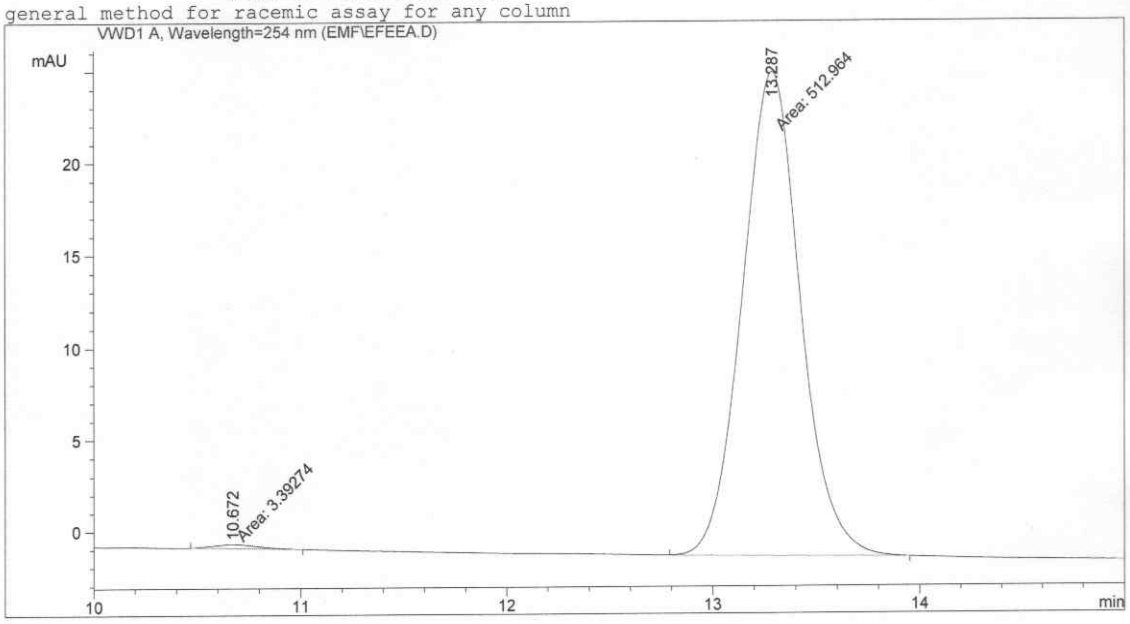



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  10.690 MM    0.2731  249.56844   15.23012  50.0345
   2  13.371 MM    0.3311  249.22391   12.54574  49.9655

Totals :                  498.79234   27.77586

 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```

Table 3, entry 1 (resolved):

Data File D:\HPCHEM\1\DATA\EMF\EFEEA.D Sample Name: emf-ee-A

```
==========================================================================
Injection Date  : 5/29/01 14:39:31 PM          Seq. Line :  14
Sample Name     : emf-ee-A                          Vial :  31
Acq. Operator   : eric                               Inj :   1
                                                Inj Volume : 5 µl
Acq. Method     : D:\HPCHEM\2\METHODS\3-EOH30.M
Last changed    : 9/5/00 16:36:56 PM by sean
Analysis Method : D:\HPCHEM\2\METHODS\4-IPA100.M
Last changed    : 6/10/01 08:42:35 PM by Joel
                  (modified after loading)
general method for racemic assay for any column
```



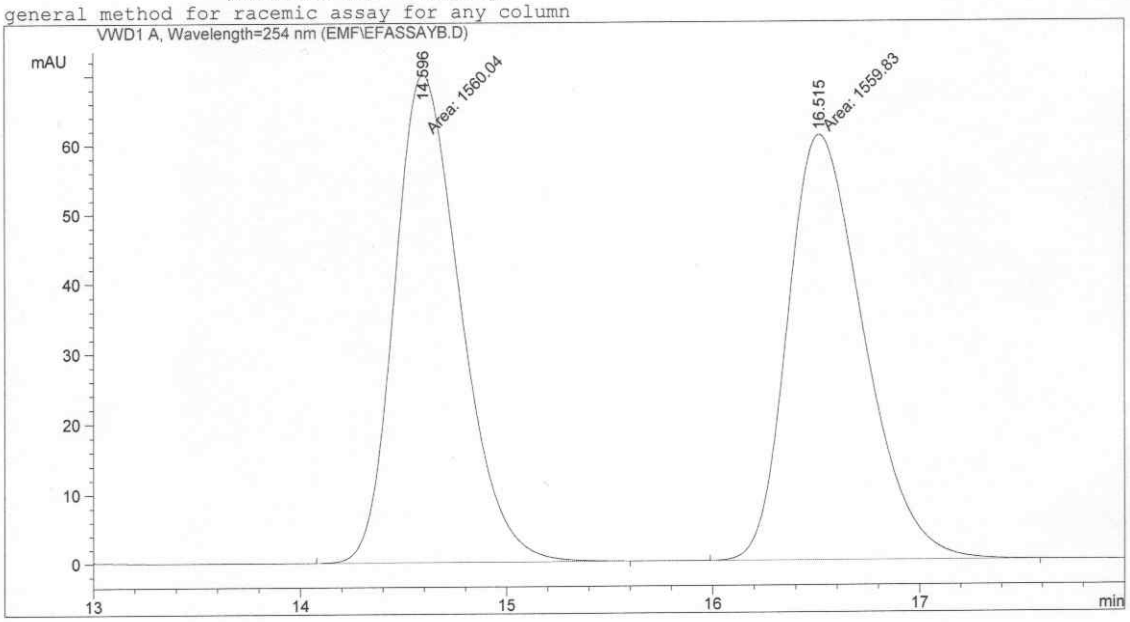

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  10.672 MM    0.2552    3.39274 2.21545e-1   0.6571
   2  13.287 MM    0.3247  512.96387   26.33393  99.3429

Totals :                   516.35661   26.55547

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Instrument 1 6/10/01 08:43:08 PM Joel Page 1 of 1

Table 3, entry 2 (racemic):

Data File D:\HPCHEM\1\DATA\EMF\EFASSAYB.D Sample Name: emf-assay-B

```
Injection Date  : 5/28/01 02:21:25 PM          Seq. Line :   3
Sample Name     : emf-assay-B                      Vial :  62
Acq. Operator   : eric                              Inj :   1
                                                Inj Volume : 5 µl

Acq. Method     : D:\HPCHEM\2\METHODS\3-EOH40.M
Last changed    : 9/25/00 23:26:38 PM by BORTHS
Analysis Method : D:\HPCHEM\2\METHODS\4-IPA100.M
Last changed    : 6/10/01 08:43:37 PM by Joel
                  (modified after loading)
general method for racemic assay for any column
```

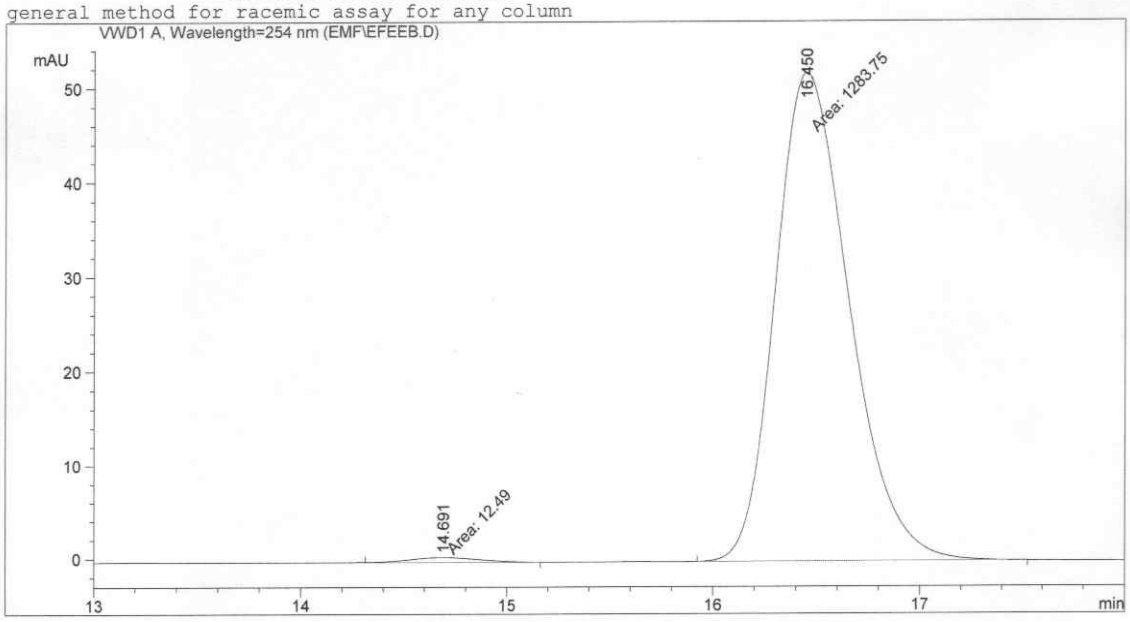



```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  14.596 MM    0.3710 1560.04199   70.09108  50.0034
   2  16.515 MM    0.4257 1559.83069   61.07388  49.9966

Totals :                 3119.87268  131.16496

 Results obtained with enhanced integrator!

                        *** End of Report ***
```

Table 3, entry 2 (resolved):

Data File D:\HPCHEM\1\DATA\EMF\EFEEB.D Sample Name: emf-ee-B

```
==========================================================================
Injection Date  : 5/29/01 15:11:00 PM          Seq. Line :  15
Sample Name     : emf-ee-B                          Vial :  32
Acq. Operator   : eric                               Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\HPCHEM\2\METHODS\3-EOH30.M
Last changed    : 9/5/00 16:36:56 PM by sean
Analysis Method : D:\HPCHEM\2\METHODS\4-IPA100.M
Last changed    : 6/10/01 08:44:45 PM by Joel
                  (modified after loading)
general method for racemic assay for any column
```

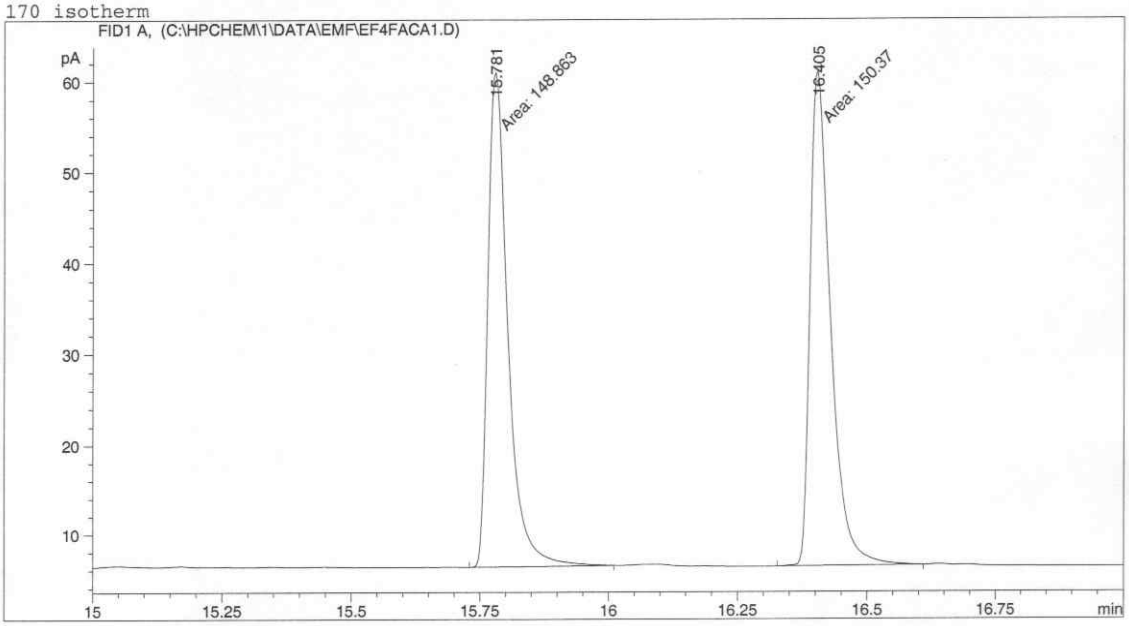



```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  14.691 MM    0.3923   12.48999 5.30598e-1   0.9636
   2  16.450 MM    0.4125 1283.75464   51.86497  99.0364

Totals :                  1296.24463   52.39557

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Table 3, entry 3 (racemic):

Data File C:\HPCHEM\1\DATA\EMF\EF4FACA1.D Sample Name: emf-4f-ac-a1

```
==========================================================================
Injection Date  : 5/30/01 4:46:59 PM            Seq. Line :  14
Sample Name     : emf-4f-ac-a1                       Vial : 104
Acq. Operator   : eric                               Inj :   1
Acq. Instrument : Prof. Plum (6890)           Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\50R5F.M
Last changed    : 5/30/01 9:29:15 AM by Alan
Analysis Method : C:\HPCHEM\4\METHODS\JP170ISO.M
Last changed    : 6/10/01 9:02:37 AM by ALAN
                  (modified after loading)
170 isotherm
```



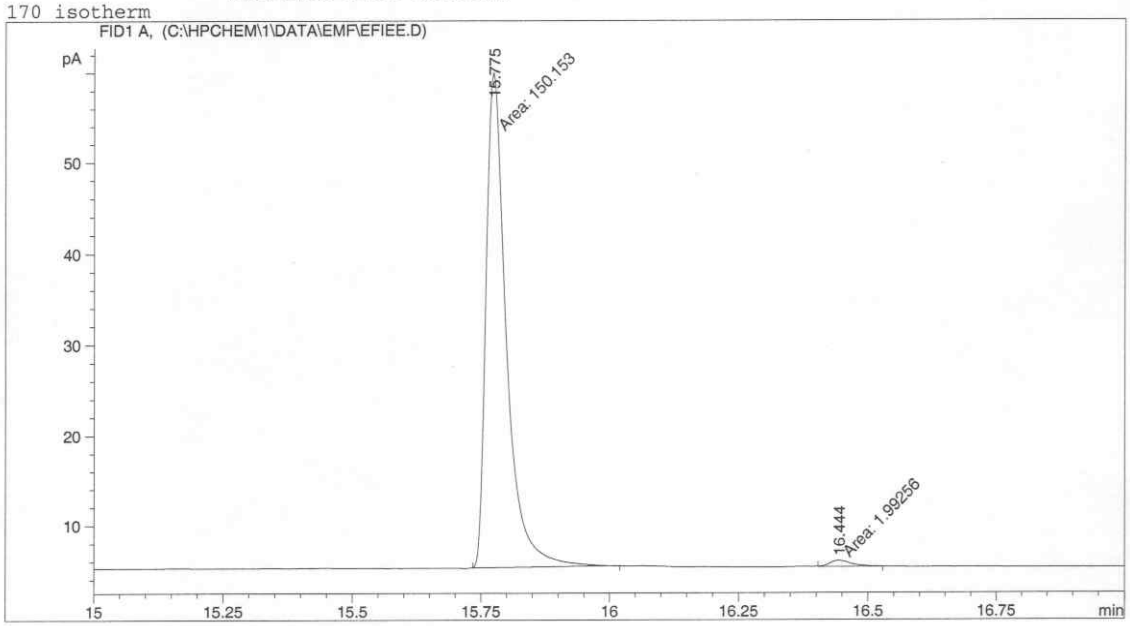

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  15.781 MM    0.0453  148.86282   54.76731 49.74814
   2  16.405 MM    0.0461  150.37013   54.35407 50.25186

Totals :                  299.23296  109.12139

 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```

Miss Scarlet 6/10/01 9:03:11 AM ALAN Page 1 of 1

Table 3, entry 3 (resolved):

Data File C:\HPCHEM\1\DATA\EMF\EFIEE.D Sample Name: emf-I-ee

```
==========================================================================
Injection Date  : 6/2/01 12:46:45 AM            Seq. Line :   1
Sample Name     : emf-I-ee                        Vial :  104
Acq. Operator   : eric                             Inj :   1
Acq. Instrument : Prof. Plum (6890)            Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\50R5F.M
Last changed    : 5/30/01 9:29:15 AM by Alan
Analysis Method : C:\HPCHEM\4\METHODS\JP170ISO.M
Last changed    : 6/10/01 8:15:58 AM by ALAN
                  (modified after loading)
170 isotherm
```

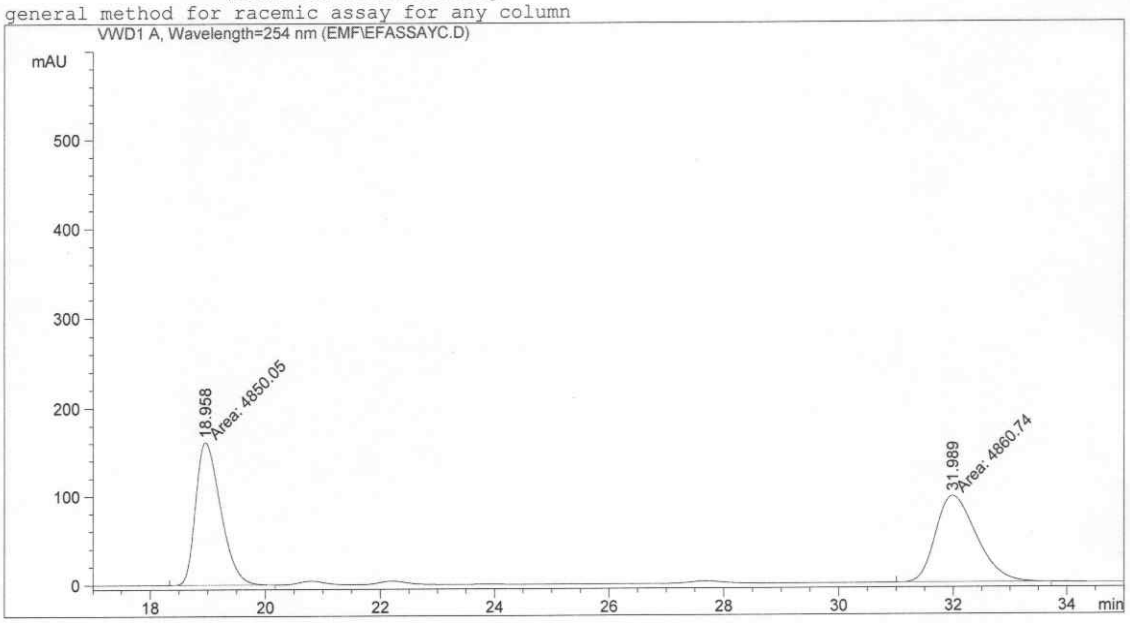



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  15.775 MM    0.0455  150.15273   55.01765 98.69036
   2  16.444 MM    0.0485    1.99256 6.84783e-1  1.30964

Totals :                   152.14528   55.70243

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Table 3, entry 4 (racemic):

Data File D:\HPCHEM\1\DATA\EMF\EFASSAYC.D Sample Name: emf-assay-C

```
==========================================================================
Injection Date  : 5/28/01 03:03:01 PM          Seq. Line :   4
Sample Name     : emf-assay-C                      Vial :  63
Acq. Operator   : eric                             Inj :   1
                                              Inj Volume : 5 µl
Acq. Method     : D:\HPCHEM\2\METHODS\3-EOH60.M
Last changed    : 1/27/01 10:08:12 PM by julie
Analysis Method : D:\HPCHEM\2\METHODS\4-IPA100.M
Last changed    : 6/10/01 08:46:03 PM by Joel
                  (modified after loading)
general method for racemic assay for any column
```



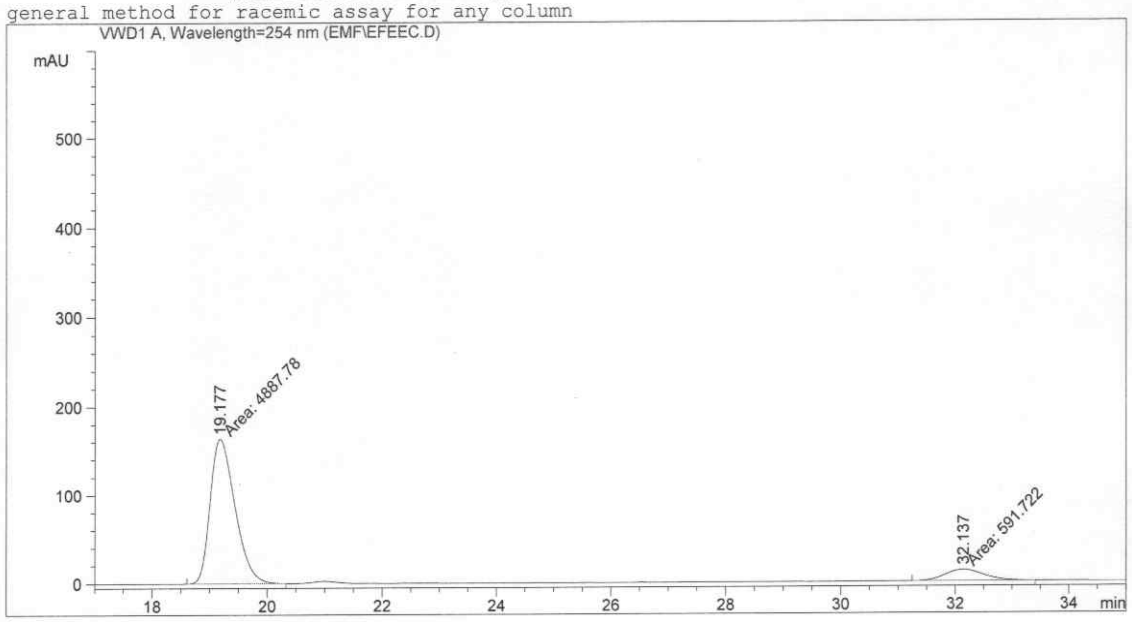

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  18.958 MM    0.5003 4850.04541  161.55646  49.9449
   2  31.989 MM    0.8300 4860.73975   97.60128  50.0551

Totals :                  9710.78516  259.15774

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Instrument 1 6/10/01 08:46:06 PM Joel Page 1 of 1

Table 3, entry 4 (resolved):

Data File D:\HPCHEM\1\DATA\EMF\EFEEC.D Sample Name: emf-ee-C

```
Injection Date  : 5/29/01 15:42:34 PM          Seq. Line :  16
Sample Name     : emf-ee-C                        Vial :  33
Acq. Operator   : eric                             Inj :   1
                                                Inj Volume : 5 µl
Acq. Method     : D:\HPCHEM\2\METHODS\3-EOH60.M
Last changed    : 1/27/01 10:08:12 PM by julie
Analysis Method : D:\HPCHEM\2\METHODS\4-IPA100.M
Last changed    : 6/10/01 08:46:30 PM by Joel
                  (modified after loading)
general method for racemic assay for any column
```

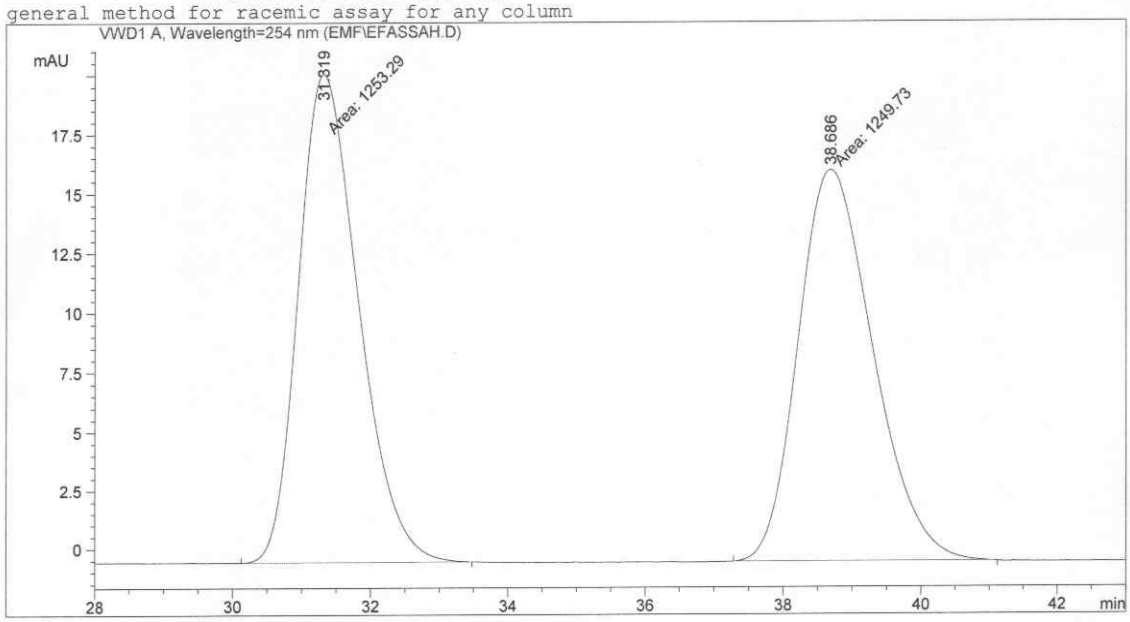



```
                          Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  19.177 MM    0.4981 4887.77881  163.53767  89.2012
   2  32.137 MM    0.7803  591.72174   12.63872  10.7988

Totals :                  5479.50055  176.17640

 Results obtained with enhanced integrator!

                          *** End of Report ***
```

Table 3, entry 5 (racemic):

Data File D:\HPCHEM\1\DATA\EMF\EFASSAH.D Sample Name: emf-H-assay

```
==========================================================================
Injection Date  : 6/2/01 23:18:47 PM            Seq. Line :  15
Sample Name     : emf-H-assay                        Vial :  37
Acq. Operator   : eric                               Inj :   1
                                                Inj Volume : 5 µl
Acq. Method     : D:\HPCHEM\2\METHODS\4-IPA50.M
Last changed    : 4/23/01 13:13:49 PM by julie
Analysis Method : D:\HPCHEM\2\METHODS\4-IPA100.M
Last changed    : 6/10/01 08:57:02 PM by Joel
                  (modified after loading)
general method for racemic assay for any column
```

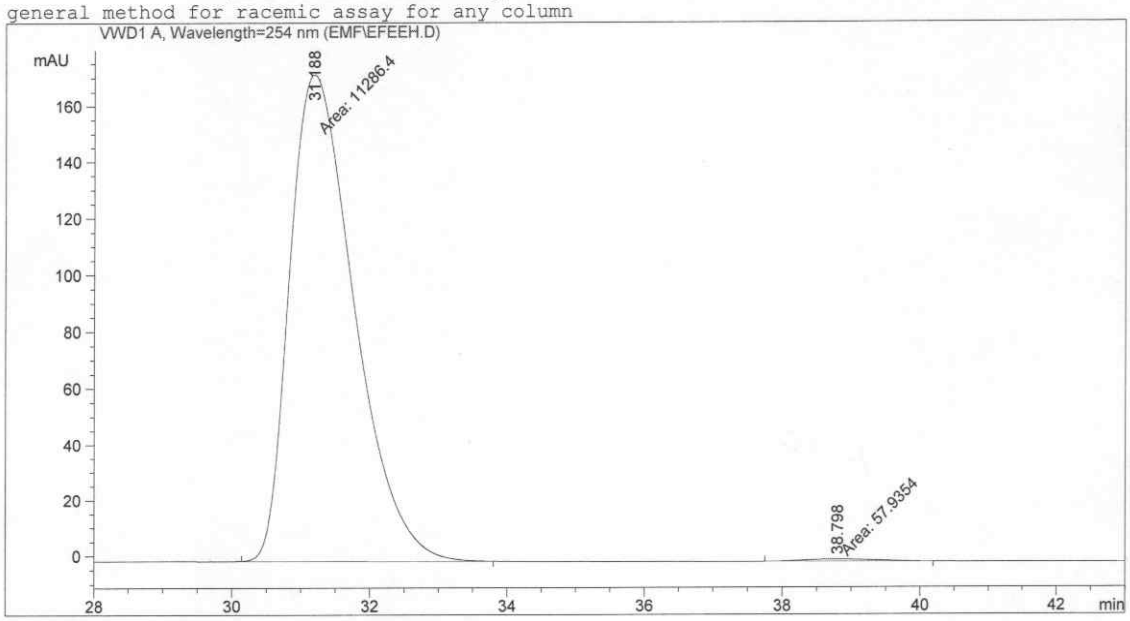



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  31.319 MM    1.0131 1253.29260   20.61803  50.0713
   2  38.686 MM    1.2616 1249.72559   16.50980  49.9287

Totals :                  2503.01819   37.12783

 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```

Table 3, entry 5 (resolved):

Data File D:\HPCHEM\1\DATA\EMF\EFEEH.D Sample Name: emf-H-ee

```
==========================================================================
Injection Date  : 6/2/01 22:27:14 PM            Seq. Line :  14
Sample Name     : emf-H-ee                         Vial :  38
Acq. Operator   : eric                              Inj :   1
                                                Inj Volume : 5 µl
Acq. Method     : D:\HPCHEM\2\METHODS\4-IPA50.M
Last changed    : 4/23/01 13:13:49 PM by julie
Analysis Method : D:\HPCHEM\2\METHODS\4-IPA100.M
Last changed    : 6/10/01 08:58:00 PM by Joel
                  (modified after loading)
general method for racemic assay for any column
```

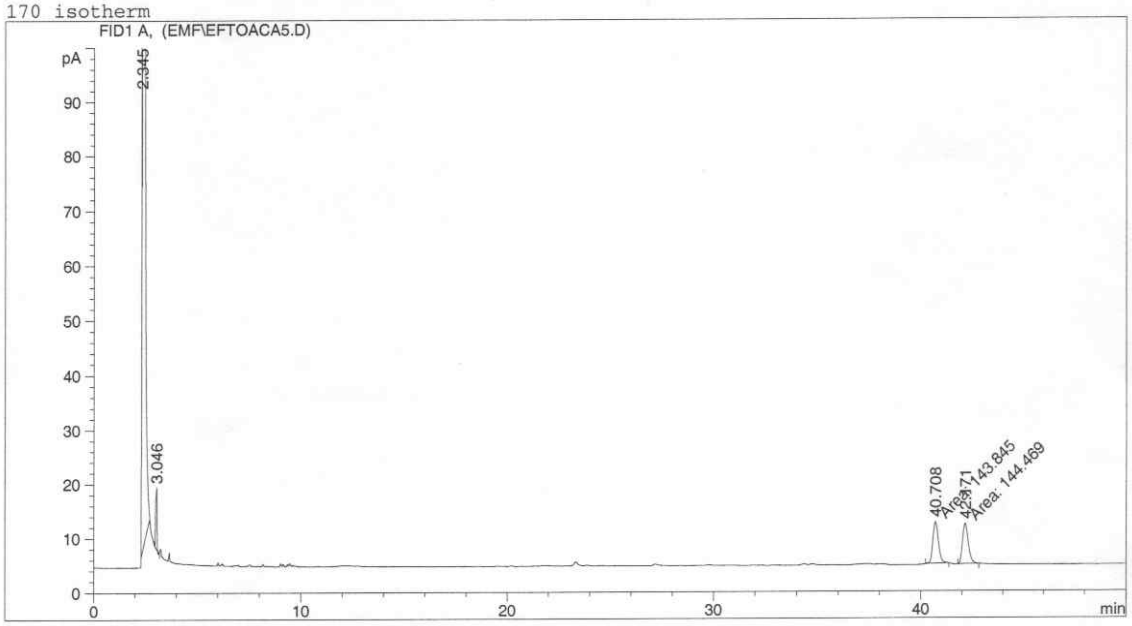



```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|---------|----------|--------|
   1  31.188 MM    1.0864 1.12864e4   173.14220  99.4893
   2  38.798 MM    1.2390   57.93544 7.79314e-1   0.5107

Totals :                  1.13443e4   173.92151

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Table 3, entry 6 (racemic):

Data File C:\HPCHEM\4\DATA\EMF\EFTOACA5.D Sample Name: emf-otol-ac-a5

```
==========================================================================
Injection Date  : 6/1/01 1:21:17 AM              Seq. Line :   9
Sample Name     : emf-otol-ac-a5                      Vial :   3
Acq. Operator   : eric                                Inj :   1
                                                 Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\4\METHODS\EF85ISO.M
Last changed    : 5/31/01 6:56:50 PM by eric
Analysis Method : C:\HPCHEM\4\METHODS\100ISO60.M
Last changed    : 6/2/01 11:00:10 PM by ALAN
                  (modified after loading)
170 isotherm
```

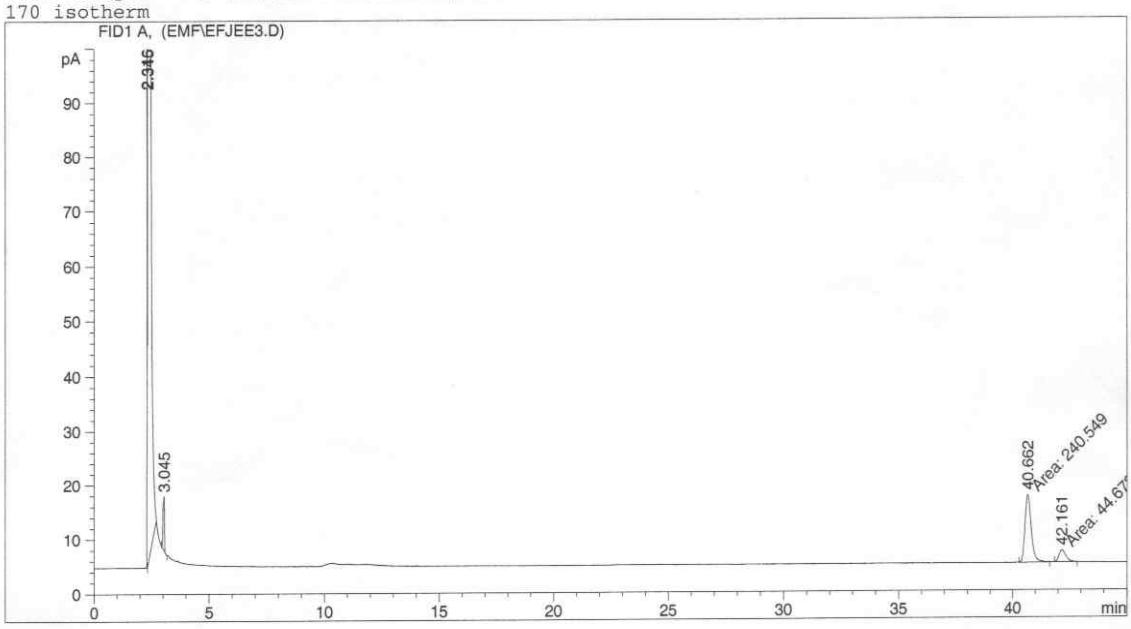



```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1   2.345 BB S  0.0451 7.62928e4  2.27571e4  99.55844
   2   3.046 PP    0.0627   50.06284   11.29815  0.06533
   3  40.708 MM    0.3115  143.84453    7.69671  0.18771
   4  42.171 MM    0.3245  144.46886    7.42024  0.18852

Totals :                  7.66312e4  2.27836e4

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Table 3, entry 6 (resolved):

Data File C:\HPCHEM\4\DATA\EMF\EFJEE3.D Sample Name: emf-J-ee3

```
==========================================================================
Injection Date  : 6/2/01 2:56:58 AM              Seq. Line :   3
Sample Name     : emf-J-ee3                         Vial :   1
Acq. Operator   : eric                               Inj :   1
                                                 Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\4\METHODS\EF85ISO.M
Last changed    : 5/31/01 6:56:50 PM by eric
Analysis Method : C:\HPCHEM\4\METHODS\100ISO60.M
Last changed    : 6/2/01 7:05:55 PM by ALAN
170 isotherm
```

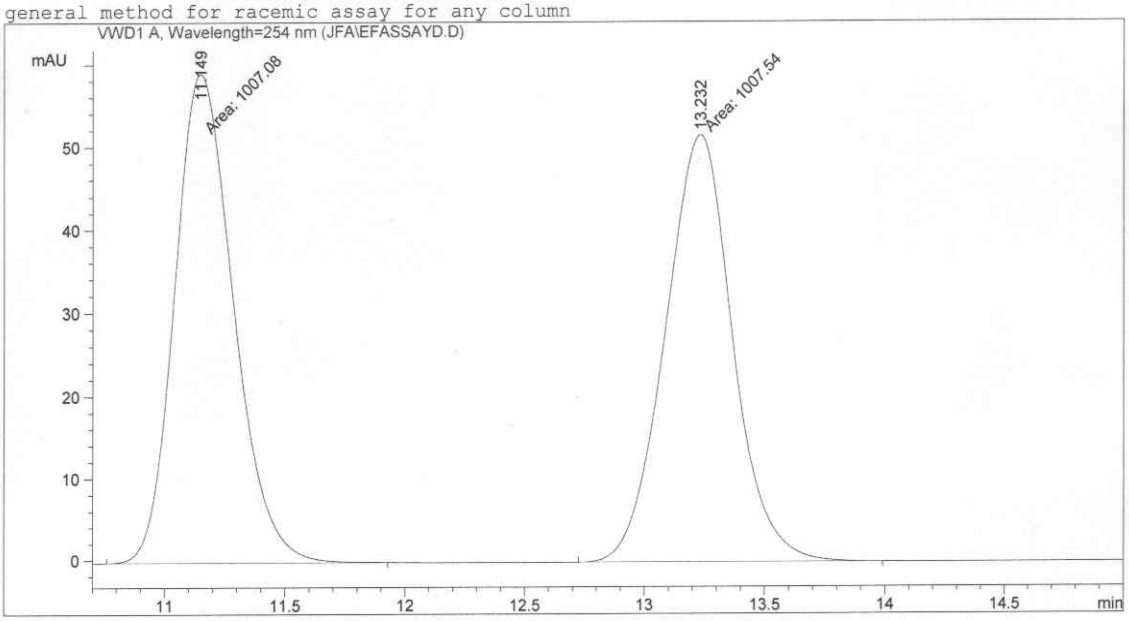



```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]    [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1   2.316 BP    0.0156  128.08118  135.49774  0.16719
   2   2.345 VB S  0.0568 7.61505e4  2.23518e4  99.40374
   3   3.045 PP    0.0612   43.47715    9.90621  0.05675
   4  40.662 MM    0.3218  240.54904   12.46029  0.31400
   5  42.161 MM    0.3412   44.67281    2.18190  0.05831

Totals :                  7.66072e4  2.25119e4

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Table 3, entry 7 (racemic):

Data File D:\HPCHEM\1\DATA\JFA\EFASSAYD.D Sample Name: emf-assay-D

```
==========================================================================
Injection Date  : 5/31/01 03:39:52 PM          Seq. Line :  12
Sample Name     : emf-assay-D                       Vial :  31
Acq. Operator   : Joel                               Inj :   1
                                                Inj Volume : 5 µl
Acq. Method     : D:\HPCHEM\2\METHODS\3-EOH30.M
Last changed    : 9/5/00 16:36:56 PM by sean
Analysis Method : D:\HPCHEM\2\METHODS\4-IPA100.M
Last changed    : 6/10/01 08:37:50 PM by Joel
                  (modified after loading)
general method for racemic assay for any column
```

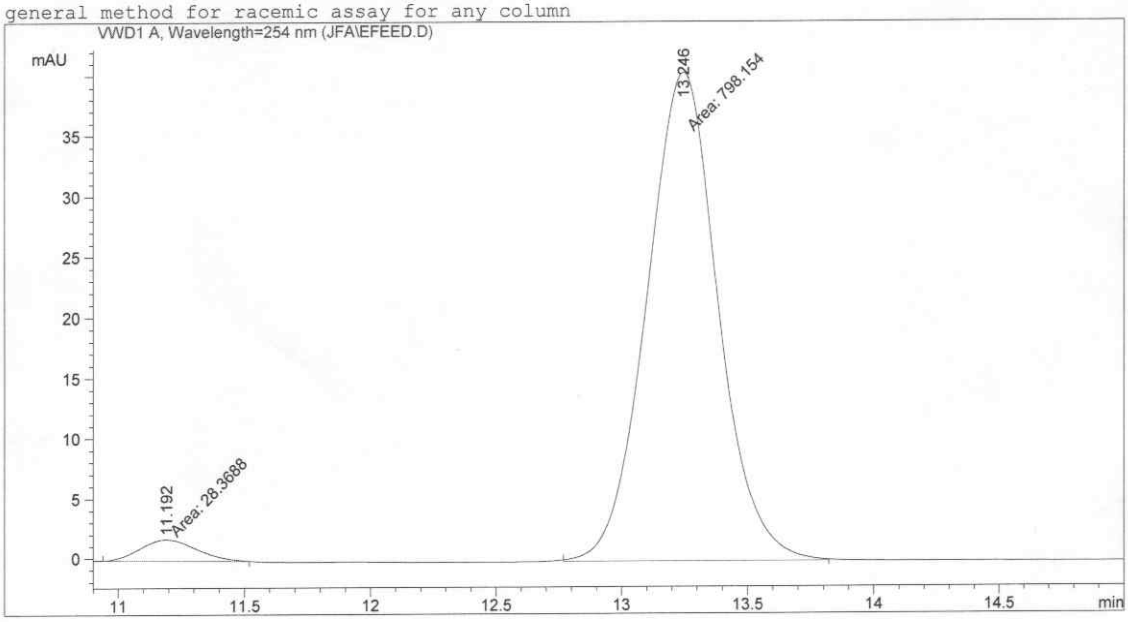



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  11.149 MM    0.2836 1007.07867   59.18278  49.9886
   2  13.232 MM    0.3251 1007.53864   51.65504  50.0114

Totals :                  2014.61731  110.83782

 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```

Table 3, entry 7 (resolved):

Data File D:\HPCHEM\1\DATA\JFA\EFEED.D Sample Name: emf-ee-D

```
==========================================================================
Injection Date  : 5/31/01 04:11:20 PM          Seq. Line :  13
Sample Name     : emf-ee-D                        Vial :  41
Acq. Operator   : Joel                            Inj :   1
                                              Inj Volume : 5 µl

Acq. Method     : D:\HPCHEM\2\METHODS\3-EOH30.M
Last changed    : 9/5/00 16:36:56 PM by sean
Analysis Method : D:\HPCHEM\2\METHODS\4-IPA100.M
Last changed    : 6/10/01 08:36:33 PM by Joel
                  (modified after loading)
general method for racemic assay for any column
```

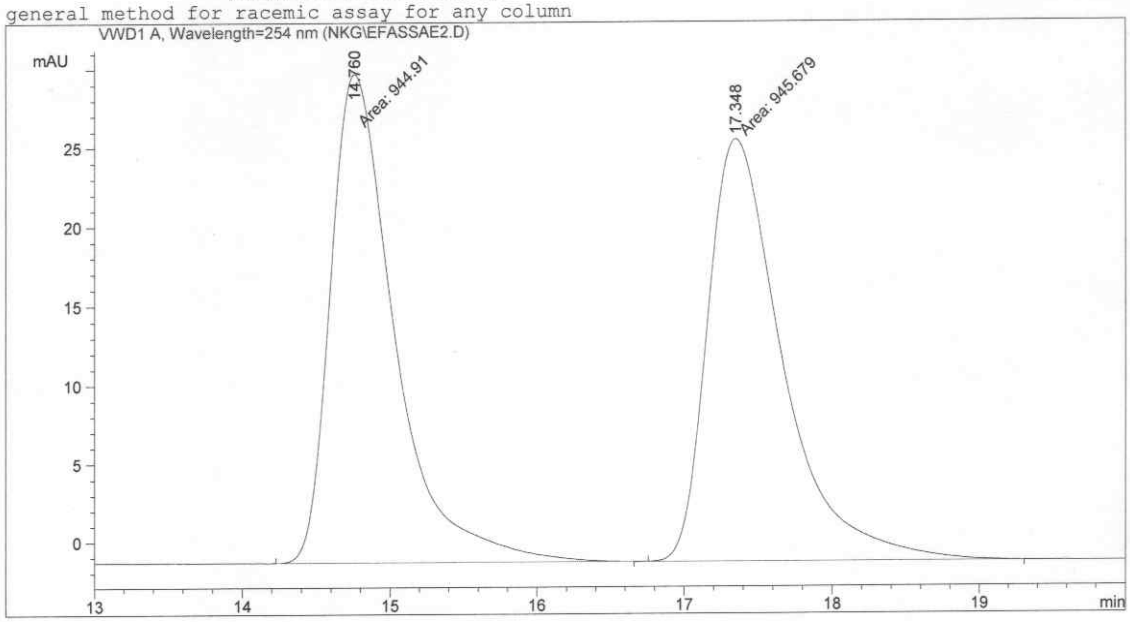



```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  11.192 MM    0.2607   28.36876    1.81343   3.4323
   2  13.246 MM    0.3277  798.15424   40.58965  96.5677

Totals :                   826.52300   42.40308

 Results obtained with enhanced integrator!
==========================================================================
                         *** End of Report ***
```

Table 3, entry 8 (racemic):

Data File D:\HPCHEM\1\DATA\NKG\EFASSAE2.D Sample Name: emf-E-assay2

```
==========================================================================
Injection Date  : 6/5/01 20:05:07 PM            Seq. Line :  13
Sample Name     : emf-E-assay2                       Vial :  33
Acq. Operator   : garg                               Inj :   1
                                                Inj Volume : 5 µl

Acq. Method     : D:\HPCHEM\2\METHODS\3-EOH60.M
Last changed    : 1/27/01 10:08:12 PM by julie
Analysis Method : D:\HPCHEM\2\METHODS\4-IPA100.M
Last changed    : 6/10/01 08:48:50 PM by Joel
                  (modified after loading)
general method for racemic assay for any column
```

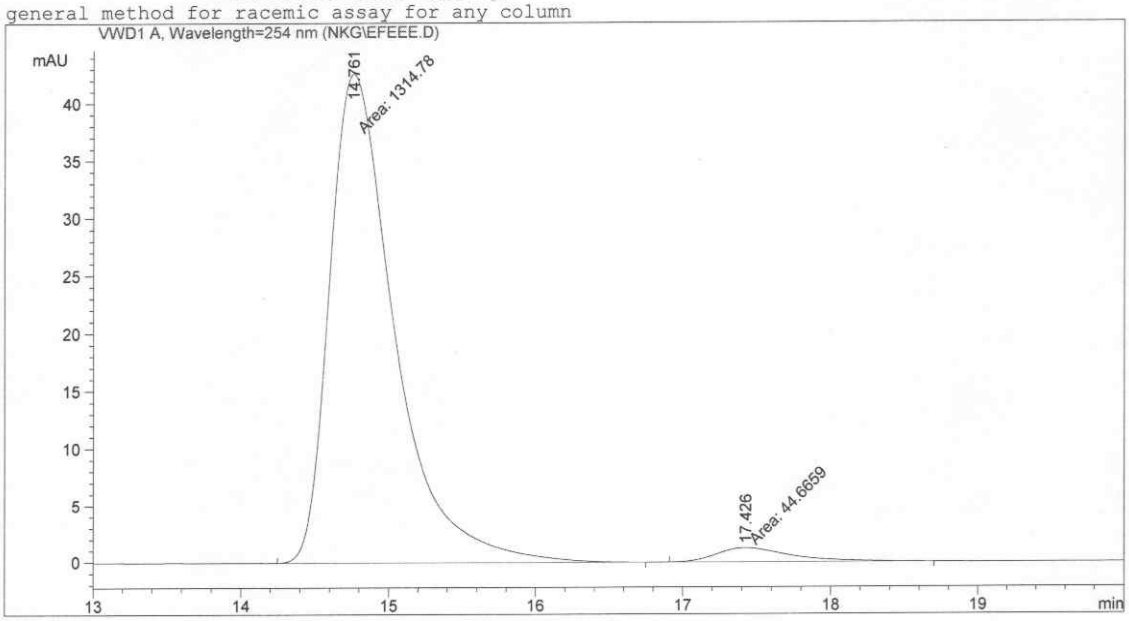



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  14.760 MM    0.5075  944.90955   31.03063  49.9797
   2  17.348 MM    0.5882  945.67865   26.79563  50.0203

Totals :                  1890.58820   57.82626

 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```

Table 3, entry 8 (resolved):

Data File D:\HPCHEM\1\DATA\NKG\EFEEE.D Sample Name: emf-E-ee

```
==========================================================================
Injection Date  : 6/5/01 21:06:41 PM            Seq. Line :  14
Sample Name     : emf-E-ee                          Vial :  34
Acq. Operator   : garg                              Inj :   1
                                                Inj Volume : 5 µl
Acq. Method     : D:\HPCHEM\2\METHODS\3-EOH30.M
Last changed    : 9/5/00 16:36:56 PM by sean
Analysis Method : D:\HPCHEM\2\METHODS\4-IPA100.M
Last changed    : 6/10/01 08:49:31 PM by Joel
                  (modified after loading)
general method for racemic assay for any column
```



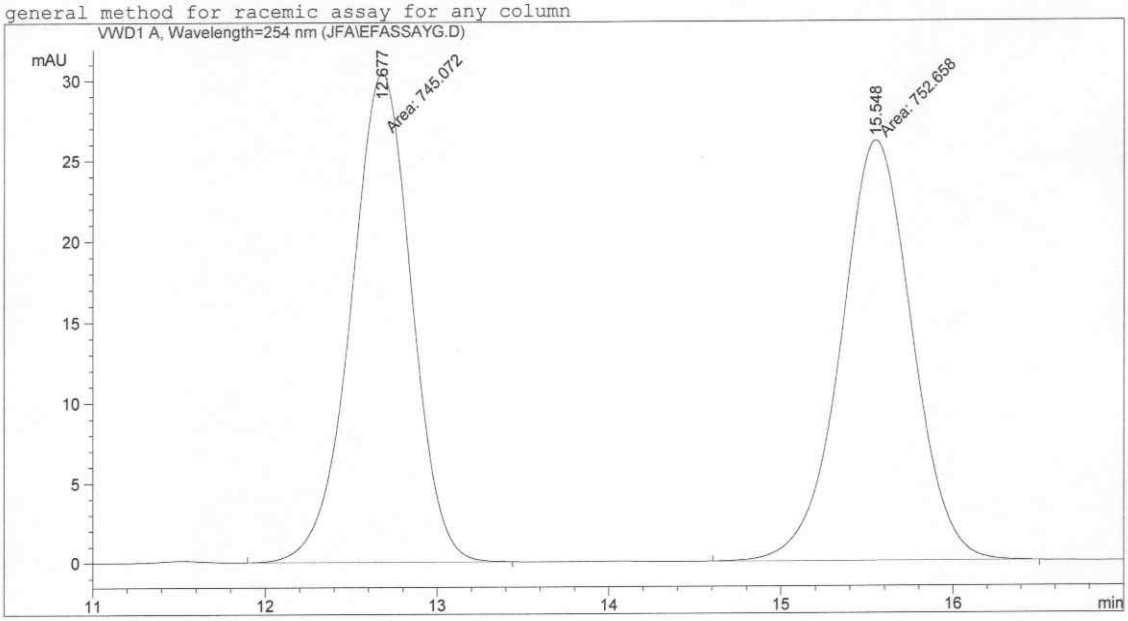

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  14.761 MM    0.5139 1314.77576   42.64273  96.7144
   2  17.426 MM    0.5905   44.66591    1.26071   3.2856

Totals :                  1359.44167   43.90344

 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```

Instrument 1 6/10/01 08:49:54 PM Joel Page 1 of 1

Table 3, entry 9 (racemic):

Data File D:\HPCHEM\1\DATA\JFA\EFASSAYG.D Sample Name: emf-assay-G

```
==========================================================================
Injection Date  : 5/31/01 08:00:42 PM          Seq. Line :  22
Sample Name     : emf-assay-G                      Vial :  34
Acq. Operator   : Joel                              Inj :   1
                                                Inj Volume : 5 µl
Acq. Method     : D:\HPCHEM\2\METHODS\2-EOH20.M
Last changed    : 12/15/00 14:45:04 PM by JFA
Analysis Method : D:\HPCHEM\2\METHODS\4-IPA100.M
Last changed    : 6/10/01 08:54:14 PM by Joel
                  (modified after loading)
general method for racemic assay for any column
```

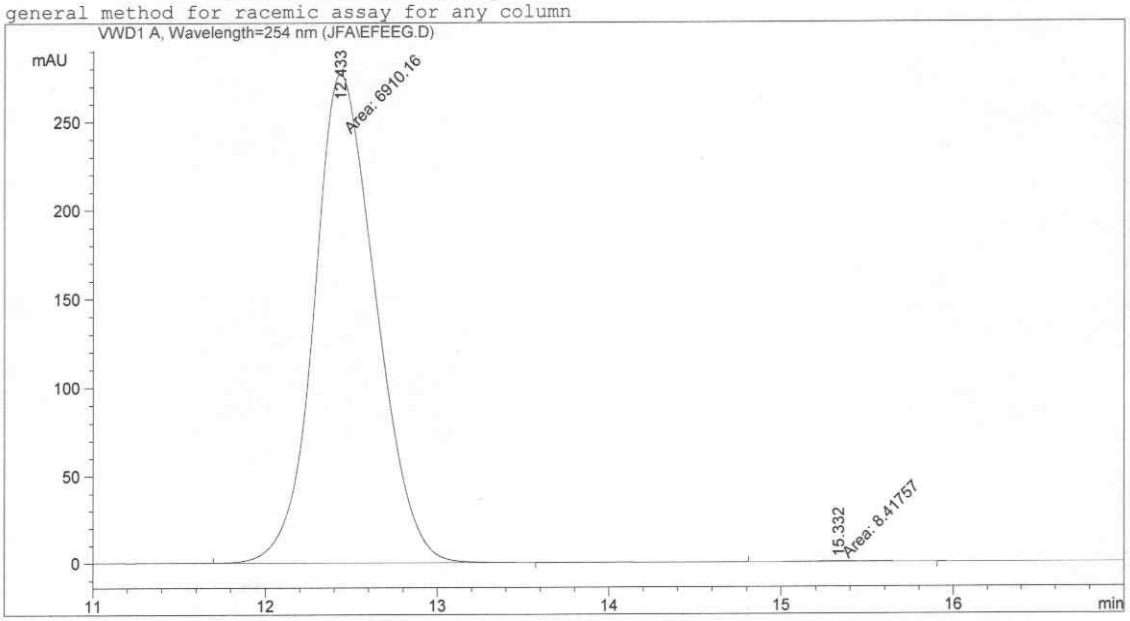



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  12.677 MM    0.4086  745.07184   30.39280  49.7467
   2  15.548 MM    0.4806  752.65845   26.10356  50.2533

Totals :                  1497.73029   56.49635

 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```

Table 3, entry 9 (resolved):

Data File D:\HPCHEM\1\DATA\JFA\EFEEG.D Sample Name: emf-ee-G

```
==========================================================================
Injection Date  : 5/31/01 08:22:14 PM          Seq. Line :  23
Sample Name     : emf-ee-G                        Vial :  44
Acq. Operator   : Joel                            Inj :   1
                                              Inj Volume : 5 µl

Acq. Method     : D:\HPCHEM\2\METHODS\2-EOH20.M
Last changed    : 12/15/00 14:45:04 PM by JFA
Analysis Method : D:\HPCHEM\2\METHODS\4-IPA100.M
Last changed    : 6/10/01 08:54:14 PM by Joel
                  (modified after loading)
general method for racemic assay for any column
```



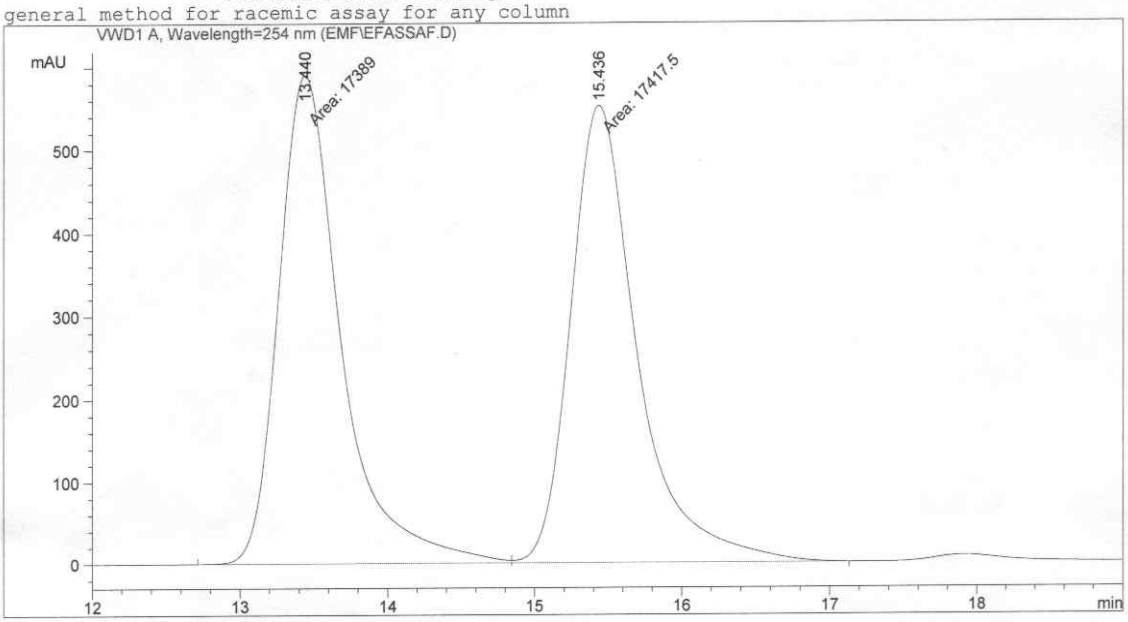

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  12.433 MM    0.4158 6910.15527  276.99277  99.8783
   2  15.332 MM    0.5579    8.41757 2.51479e-1   0.1217

Totals :                  6918.57285  277.24425

 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```

Table 3, entry 10 (racemic):

Data File D:\HPCHEM\1\DATA\EMF\EFASSAF.D Sample Name: emf-F-assay

Injection Date	: 6/10/01 23:01:42 PM	Seq. Line	: 2
Sample Name	: emf-F-assay	Vial	: 25
Acq. Operator	: eric	Inj	: 1
		Inj Volume	: 5 µl

Acq. Method : D:\HPCHEM\2\METHODS\4-IPA30.M
Last changed : 9/11/00 22:12:44 PM by Larsen
Analysis Method : D:\HPCHEM\2\METHODS\100HEX20.M
Last changed : 6/11/01 08:16:07 PM by Joel
(modified after loading)
general method for racemic assay for any column



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.440	MF	0.4912	1.73890e4	590.02283	49.9590
2	15.436	FM	0.5260	1.74175e4	551.86200	50.0410
Totals :				3.48065e4	1141.88483	

Results obtained with enhanced integrator!

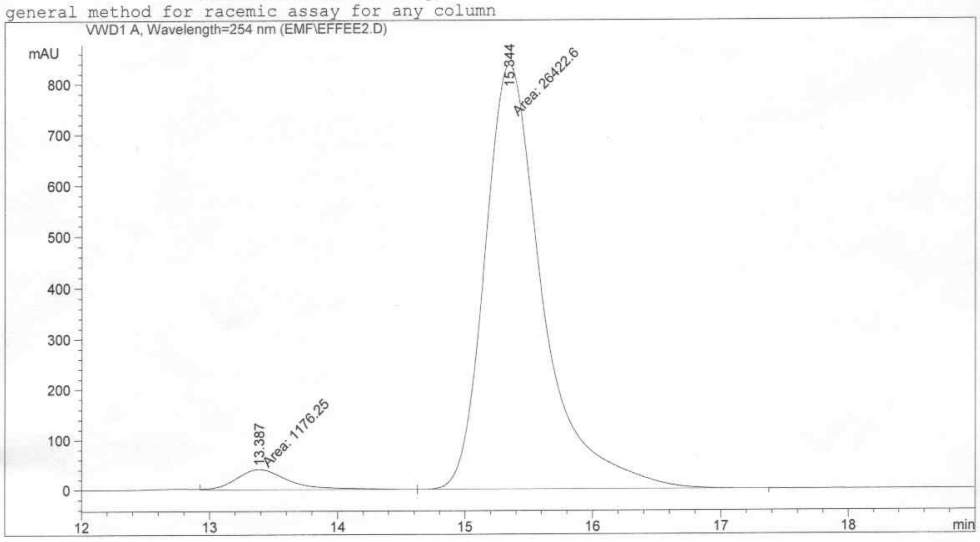
*** End of Report ***

Instrument 1 6/11/01 08:16:39 PM Joel Page 1 of 1

Table 3, entry 10 (resolved):

Data File D:\HPCHEM\1\DATA\EMF\EFFEE2.D Sample Name: emf-F-ee-2

```
Injection Date  : 6/10/01 20:27:24 PM          Seq. Line :   2
Sample Name     : emf-F-ee-2                      Vial :  24
Acq. Operator   : eric                             Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\HPCHEM\2\METHODS\4-IPA30.M
Last changed    : 9/11/00 22:12:44 PM by Larsen
Analysis Method : D:\HPCHEM\2\METHODS\100HEX20.M
Last changed    : 6/10/01 22:34:49 PM by Joel
                  (modified after loading)
general method for racemic assay for any column
```



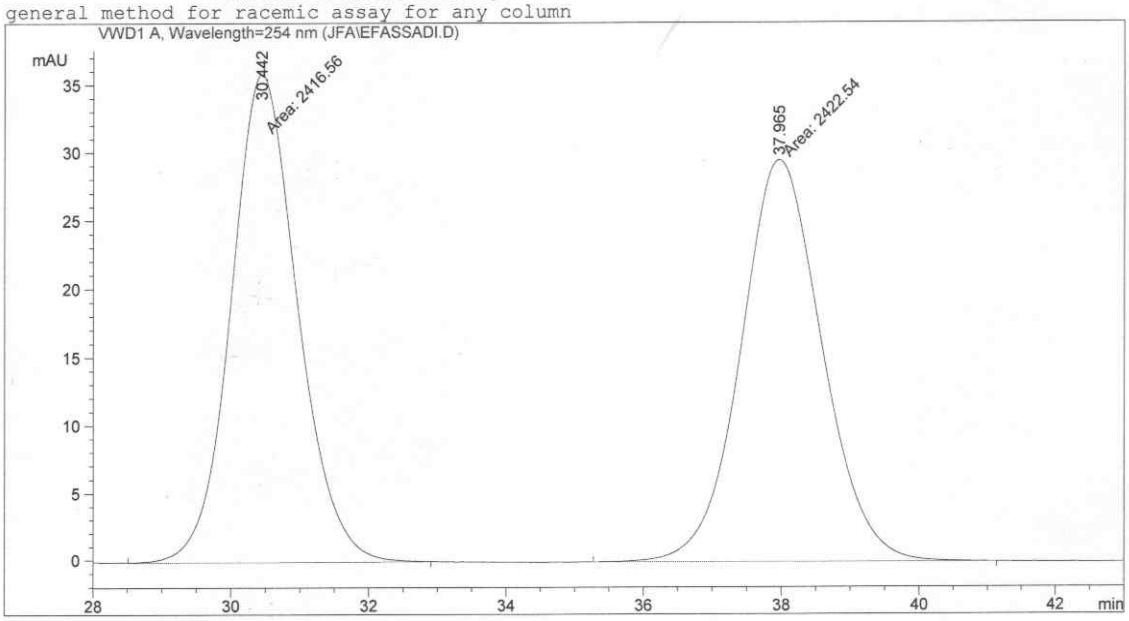

```
                          Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  13.387 MF    0.4800 1176.25110   40.83941   4.2620
   2  15.344 FM    0.5264 2.64226e4   836.53857  95.7380

Totals :                  2.75988e4   877.37798

 Results obtained with enhanced integrator!

                          *** End of Report ***
```

Instrument 1 6/10/01 22:36:08 PM Joel Page 1 of 1

Ketoalcohol **(±)-ii** :

Data File D:\HPCHEM\1\DATA\JFA\EFASSADI.D Sample Name: emf-assay-diol

```
======================================================================
Injection Date  : 5/31/01 13:59:40 PM          Seq. Line :   8
Sample Name     : emf-assay-diol                    Vial :  46
Acq. Operator   : Joel                               Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\HPCHEM\2\METHODS\6-IPA60.M
Last changed    : 1/8/01 18:23:47 PM by JFA
Analysis Method : D:\HPCHEM\2\METHODS\4-IPA100.M
Last changed    : 6/10/01 08:59:35 PM by Joel
                  (modified after loading)
general method for racemic assay for any column
```



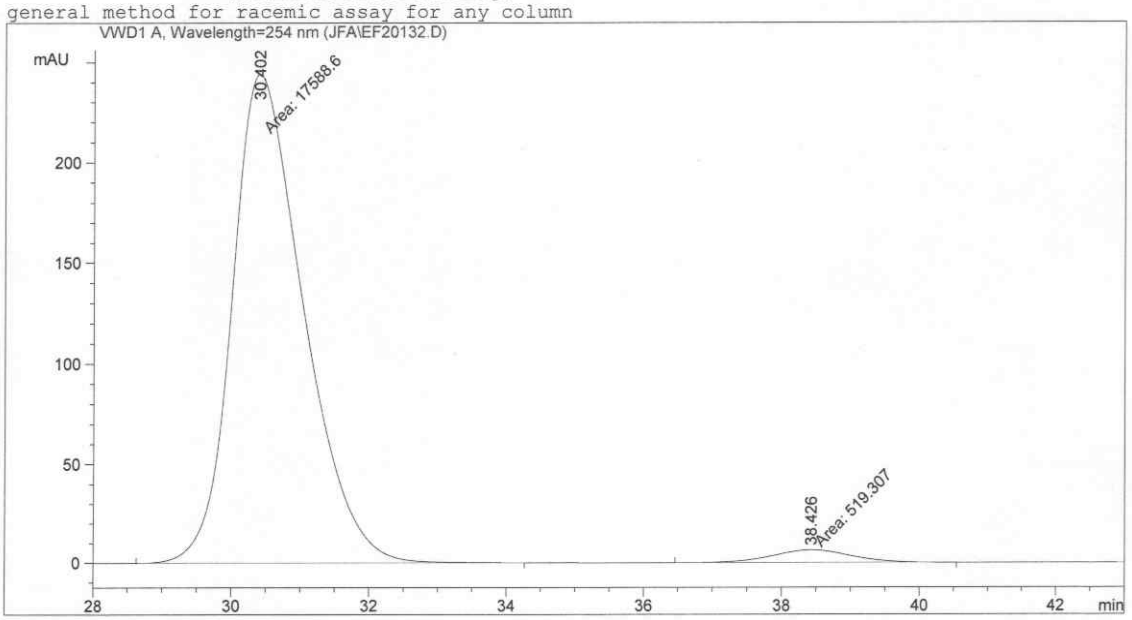

```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  30.442 MM    1.1200 2416.55933   35.95950  49.9382
   2  37.965 MM    1.3666 2422.54297   29.54392  50.0618

Totals :                  4839.10229   65.50342

 Results obtained with enhanced integrator!
======================================================================
                        *** End of Report ***
```

Instrument 1 6/10/01 09:00:30 PM Joel Page 1 of 1

Ketoalcohol **(+)-ii** :

Data File D:\HPCHEM\1\DATA\JFA\EF20132.D Sample Name: emf-iv-201-32h

```
==========================================================================
Injection Date  : 5/31/01 12:58:07 PM          Seq. Line :   7
Sample Name     : emf-iv-201-32h                    Vial :  45
Acq. Operator   : Joel                              Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\HPCHEM\2\METHODS\6-IPA60.M
Last changed    : 1/8/01 18:23:47 PM by JFA
Analysis Method : D:\HPCHEM\2\METHODS\4-IPA100.M
Last changed    : 6/10/01 09:01:37 PM by Joel
                  (modified after loading)
general method for racemic assay for any column
```




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

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  30.402 MM    1.1999 1.75886e4   244.31598  97.1321
   2  38.426 MM    1.3321  519.30701    6.49755   2.8679

Totals :                  1.81079e4   250.81353

 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```