Catalytic Asymmetric Rearrangement of Allylic Trichloroacetimidates. A Practical Method for Preparing Allylic Amines and Congeners of High Enantiomeric Purity

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Supporting Information

Representative experimental procedures, copies of HPLC and GC traces used to determine enantiopurity, and copies of ^1H and ^{13}C NMR spectra for new compounds.

(38 Pages)

General experimental details: The procedure employed to purify THF and CH_2Cl_2 has been described;¹ other general experimental details also have been described.² All HPLC and GC analyses used to determine enantiomeric purity were calibrated with samples of the racemate.

General procedure for the synthesis of trichloroacetimidates. Preparation of (*E*)-2-

hexen-6-yl acid trichloroacetimidate methyl ester (**1k**).

Trichloroacetoneitrile (0.36 mL, 3.6 mmol) was added to 6-hydroxyhex-4-enoic acid methyl ester³ (310 mg, 2.4 mmol), DBU (70 μL , 0.48 mmol), and CH_2Cl_2 (15 mL) at 0 $^\circ\text{C}$. The reaction was warmed slowly to room temperature. After 2 h, the reaction was concentrated. Purification of the residue by chromatography (Davisil grade SiO_2 , 95:5 hexanes/EtOAc) afforded 622 mg (95%) of **1k** as a colorless oil: ^1H NMR (500 MHz, CDCl_3) δ 8.28 (broad s, 1H), 5.83–5.92 (m, 1H), 5.70–5.77 (m, 1H), 4.74 (d, J = 6.1 Hz, 2H), 3.67 (s, 3H), 2.38–2.47 (m, 4H); ^{13}C NMR (125 MHz, CDCl_3) δ 173.1, 162.4, 134.2, 124.3, 91.4, 69.4, 51.5, 33.2, 27.4; IR (neat) 3344, 2958, 1738, 1661, 1444, 1290 cm^{-1} ; HRMS (CI- NH_3) m/z 286.9881 [286.9883 calcd for $\text{C}_9\text{H}_{12}\text{Cl}_3\text{NO}_3$ (M)]. Anal. Calcd for $\text{C}_9\text{H}_{12}\text{Cl}_3\text{NO}_3$: C, 37.46; H, 4.19; N, 4.85. Found: C, 37.61; H, 4.18; N, 4.90.

¹ Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics*, **1996**, 15, 1518.

² Deng, W.; Overman, L. E. *J. Am. Chem. Soc.* **1994**, 116, 11241.

³ Analogous to 6-hydroxyhex-4-enoic acid ethyl ester: Casara, P. *Tetrahedron Lett.* **1994**, 35, 3049–3050.

General procedure for allylic imide rearrangement using 5 mol % COP-Cl (5).

Preparation of (S)-2,2,2-trichloro-N-((9-(N,N-dibenzyl)-1-nonanylamino)allyl)acetamide (2q). A solution of COP-Cl (**5**, 12 mg, 0.008 mmol) in CH_2Cl_2 (0.26 mL) was added to imide **1q** (81 mg, 0.16 mmol). The reaction flask was then sealed, protected from light and maintained at room temperature. After 18 h, the reaction was concentrated. Purification of the residue by chromatography (Davisil grade SiO_2 , 98:2 hexanes/EtOAc) afforded 67 mg (82%) of **2q** as a pale yellow oil.

Chiral HPLC (Chiracel OD-H, 99.6:0.4 *n*-hexane/IPA, 0.8 mL/min, 230 nm) showed that **2q** had been formed in 97% ee: $[\alpha]_D^{27} = +7.1$, $[\alpha]_{577}^{27} = +6.2$, $[\alpha]_{546}^{27} = +7.1$, $[\alpha]_{435}^{27} = +6.9$, $[\alpha]_{405}^{27} = +10.2$ (c 0.29, $CHCl_3$); 1H NMR (500 MHz, $CDCl_3$) δ 7.19–7.39 (m, 10H), 6.50 (d, $J = 8.1$ Hz, 1H), 5.79 (ddd, $J = 17.0, 10.4, 5.6$ Hz, 1H), 5.23 (d, $J = 17.2$ Hz, 1H), 5.19 (d, $J = 10.5$ Hz, 1H), 4.37–4.44 (m, 1H), 3.54 (s, 4H), 2.39 (t, $J = 6.9$ Hz, 2H) 1.45–1.71 (m, 4H), 1.15–1.40 (m, 12H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.1, 139.8, 136.7, 129.7, 128.8, 128.1, 126.8, 116.0, 92.8, 58.2, 53.5, 53.3, 34.4, 29.41, 29.35, 29.18, 29.15, 26.8, 25.5; IR (neat) 3329, 3028, 2927, 2858, 2796, 1699, 1514, 1452, 1244 cm^{-1} ; HRMS (CI- NH_3) m/z 523.2039 [523.2050 calcd for $C_{28}H_{38}Cl_3N_2O$ (M+H)]. Anal. Calcd for $C_{28}H_{37}Cl_3N_2O$: C, 64.18; H, 7.12; N, 5.35. Found: C, 63.89; H, 7.06; N, 5.28.

General procedure for allylic imide rearrangement using 5 mol % COP-Cl (5) at

38 °C. Preparation of (S)-2,2,2-trichloro-N-(1-cyclohexylallyl)acetamide (2f). A solution of COP-Cl (**5**, 129 mg, 0.088 mmol) in CH_2Cl_2 (1.76 mL) was added to imide **1f** (500 mg, 1.76 mmol). The reaction flask was then sealed, protected from light and maintained at 38 °C. After 18 h, the reaction was concentrated.

Purification of the residue by chromatography (Davisil grade SiO_2 , 99.5:0.5 hexanes/EtOAc) afforded 412 mg (82%) of **2f** as a colorless solid. Chiral HPLC (Chiracel OD-H, 99:1 *n*-hexane/IPA, 0.8 mL/min, 230 nm) showed that **2f** had been formed in 95% ee: mp: 69–72 °C; $[\alpha]_D^{27} = +30.7$, $[\alpha]_{577}^{27} = +30.5$, $[\alpha]_{546}^{27} = +34.8$, $[\alpha]_{435}^{27} = +60.1$, $[\alpha]_{405}^{27} = +71.4$ (c 0.42, $CHCl_3$); 1H NMR (500 MHz, $CDCl_3$) δ 6.58 (broad s, 1H), 5.79 (ddd, $J = 17.1, 10.5, 6.0$ Hz, 1H), 5.19–5.25 (m, 2H), 4.27 (dd, $J = 14.8, 6.2$ Hz, 1H), 1.65–1.81 (m, 5H), 1.51–1.60 (m, 1H), 0.95–1.30 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.2, 135.3, 116.7, 93.0, 58.3, 41.9, 29.4, 28.5, 26.2, 25.9; IR (neat) 3298, 2927, 2858, 1692, 1522, 1452, 1251 cm^{-1} ; HRMS (CI- NH_3) m/z 284.0370 [284.0376 calcd for $C_{11}H_{17}Cl_3NO$ (M+H)]. Anal. Calcd for $C_{11}H_{16}Cl_3NO$: C, 46.42; H, 5.67; N, 4.92. Found: C, 46.51; H, 5.66; N, 5.02.

General procedure for allylic imidate rearrangement using 1 mol % COP-Cl (5**).** **Preparation of (S)-2,2,2-Trichloro-N-(1-iso-butylallyl)acetamide (**2c**).**

A solution of COP-Cl (**5**, 0.13, 0.12 M in CH_2Cl_2) was added to imidate **1c** (41 mg, 0.16 mmol). The reaction flask was then sealed, protected from light and maintained at 38 °C. After 18 h, the reaction was cooled to room temperature. Purification of the residue directly by chromatography (Davisil grade SiO_2 , 99.5:0.5 hexanes/EtOAc) afforded 38 mg (92%) of **2c** as a colorless solid. Chiral HPLC (Chiracel OD-H, 99.5:0.5 *n*-hexane/IPA, 0.8 mL/min, 230 nm) showed that **2c** had been formed in 98% ee (*S*): $[\alpha]_D^{27} = +9.0$, $[\alpha]_{577}^{27} = +7.1$, $[\alpha]_{546}^{27} = +4.6$, $[\alpha]_{435}^{27} = +8.8$, $[\alpha]_{405}^{27} = +12.5$ (c 0.22, CHCl_3); mp: 69–71 °C; ^1H NMR (500 MHz, CDCl_3) δ 6.46 (broad s, 1H), 5.79 (ddd, $J = 16.2, 10.4, 5.7$ Hz, 1H), 5.24 (td, $J = 17.2, 0.9$ Hz, 1H), 5.18 (td, $J = 10.4, 0.9$ Hz, 1H), 4.45–4.53 (m, 1H), 1.64–1.73 (m, 1H), 1.47–1.52 (m, 2H), 0.96 (d, $J = 6.6$ Hz, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ 161.1, 136.9, 115.8, 92.8, 51.9, 43.6, 24.8, 22.7, 22.2; IR (neat) 3306, 2958, 1692, 1522, 1468, 1259 cm^{-1} ; HRMS (CI- NH_3) m/z 199.9443 [199.9437 calcd for $\text{C}_9\text{H}_5\text{Cl}_3\text{NO}$ ($\text{M}^+\text{-}i\text{Bu}$)]. Anal. Calcd for $\text{C}_9\text{H}_{14}\text{Cl}_3\text{NO}$: C, 41.81; H, 5.46; N, 5.42. Found: C, 41.99; H, 5.46; N, 5.39.

¹H spectrum ¹³C spectrum with ¹H decoupling

1H spectrum

13C spectrum with 1H decoupling

¹H spectrum ¹³C spectrum with ¹H decoupling

¹H spectrum ¹³C spectrum with ¹H decoupling

¹H spectrum

1H spectrum

13C spectrum with 1H decoupling

¹H spectrum ¹³C spectrum with ¹H decoupling

¹H spectrum

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Current Data Parameters
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PROCID        : 1

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10 Non proton parameters
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¹³C spectrum with ¹H decoupling

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PROCNAME      1

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  UG         0.400000 sec
  D1         0.39000000 sec
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  D12        0.000000 sec

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===== CHANNEL 12 =====
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F2 - Processing parameters
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13C spectrum with 1H decoupling

¹H spectrum ¹³C spectrum with ¹H decoupling

¹H spectrum ¹³C spectrum with ¹H decoupling

¹H spectrum

¹³C spectrum with ¹H decoupling

1H spectrum

13C spectrum with 1H decoupling

¹H spectrum ¹³C spectrum with ¹H decoupling

1H spectrum

^{13}C spectrum with ^1H decoupling

Data File D:\DATA\CEO\01070303.D

Sample Name: ceo-V-180A

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Injection Date : 1/7/03 5:11:22 PM Seq. Line : 3
Sample Name : ceo-V-180A Location : Vial 63
Acq. Operator : ceo Inj : 1
Inj Volume : 5 µl

Acq. Method : D:\METHODS\CEOODH2.M
Last changed : 1/7/03 3:25:40 PM by ceo
Analysis Method : D:\METHODS\CEOODH2.M
Last changed : 5/28/03 12:20:42 PM by ceo
(modified after loading)

ODH
99.5:0.5 nHEX:IPA
0.8 mL/MIN
no guard

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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.542	MM	0.2436	415.03391	28.40023	96.7990
2	10.766	MM	0.2919	13.72468	7.83625e-1	3.2010

Totals : 428.75859 29.18386

Results obtained with enhanced integrator!

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*** End of Report ***

Data File C:\HPCHEM\1\DATA\CEO\06040305.D

Sample Name: ceo-VI-102A

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Injection Date   : 6/4/03 2:06:50 PM          Seq. Line :    5
Sample Name      : ceo-VI-102A                Location  : Vial 61
Acq. Operator    : ceo                        Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method      : D:\METHODS\CEOODH2.M
Last changed     : 6/4/03 11:51:01 AM by ceo
Analysis Method  : D:\METHODS\CEOODH2.M
Last changed     : 5/28/03 12:20:42 PM by ceo
                  (modified after loading)
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ODH
99.5:0.5 nHEX:IPA
0.8 mL/MIN
no guard

=====
Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.596	BB	0.2177	47.37278	3.29632	14.2743
2	10.823	BB	0.2475	284.50217	17.89297	85.7257

Totals : 331.87494 21.18929

Results obtained with enhanced integrator!

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*** End of Report ***

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		Inj Volume	: 5 µl
Acq. Method	: D:\METHODS\CEODH2.M		
Last changed	: 1/15/03 10:44:41 AM by mmp		
Analysis Method	: D:\METHODS\CEODH2.M		
Last changed	: 5/28/03 12:20:42 PM by ceo		
	(modified after loading)		
ODH			
99.5:0.5 nHEX:IPA			
0.8 mL/MIN			
no guard			

=====

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

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.740	PB	0.2195	720.63904	50.80690	98.1092
2	8.918	PB	0.1806	13.88880	1.09065	1.8908

Totals : 734.52784 51.89754

Results obtained with enhanced integrator!

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*** End of Report ***

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Acq. Operator    : ceo                        Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method      : D:\METHODS\CEOODH2.M
Last changed     : 6/4/03 11:51:01 AM by ceo
Analysis Method  : D:\METHODS\CEOODH2.M
Last changed     : 5/28/03 12:20:42 PM by ceo
                  (modified after loading)

ODH
99.5:0.5 nHEX:IPA
0.8 mL/MIN
no guard
=====
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                          Area Percent Report
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Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
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Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.861	BB	0.1753	40.20476	3.51793	12.4930
2	9.285	BB	0.2188	281.61255	19.94164	87.5070

Totals : 321.81731 23.45957

Results obtained with enhanced integrator!

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

Data File D:\DATA\CEO\01170310.D

Sample Name: ceo-V-200A

```
=====
Injection Date   : 1/17/03 5:36:03 PM      Seq. Line :    5
Sample Name      : ceo-V-200A              Location  : Vial 63
Acq. Operator    : ceo                     Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method      : D:\METHODS\CEOODH3.M
Last changed     : 1/17/03 5:36:43 PM by ceo
                  (modified after loading)
Analysis Method  : D:\METHODS\CEOODH2.M
Last changed     : 5/28/03 12:20:42 PM by ceo
                  (modified after loading)

ODH
99.5:0.5 nHEX:IPA
0.8 mL/MIN
no guard
```

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

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.428	BV	0.1991	63.65575	4.91658	3.7988
2	10.032	VB	0.2249	1612.00513	108.78682	96.2012

Totals : 1675.66088 113.70340

Results obtained with enhanced integrator!

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

Data File D:\DATA\CEO\02150302.D

Sample Name: ceo-V-243

```
=====
Injection Date   : 2/15/03 12:32:22 PM      Seq. Line :    2
Sample Name      : ceo-V-243                Location  : Vial 62
Acq. Operator    : ceo                      Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method      : D:\METHODS\CEODH3.M
Last changed     : 1/29/03 5:54:48 PM by mmp
Analysis Method  : D:\METHODS\CEODH3.M
Last changed     : 6/28/03 8:08:19 PM by MMP
ODH
99:1 nHEX:IPA
0.8 mL/MIN
=====
```

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

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.993	BB	0.1532	731.01862	72.75700	97.5202
2	8.190	BB	0.1875	18.58839	1.53310	2.4798

Totals : 749.60701 74.29011

Results obtained with enhanced integrator!

=====
*** End of Report ***

Data File D:\DATA\CEO\01150311.D

Sample Name: ceo-V-197B

=====

Injection Date	: 1/15/03 3:59:44 PM	Seq. Line	: 11
Sample Name	: ceo-V-197B	Location	: Vial 69
Acq. Operator	: ceo	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\METHODS\CEOODH2.M		
Last changed	: 1/15/03 3:59:17 PM by ceo		
	(modified after loading)		
Analysis Method	: D:\METHODS\CEOODH3.M		
Last changed	: 6/28/03 8:08:19 PM by MMP		
ODH			
99:1 nHEX:IPA			
0.8 mL/MIN			

=====

=====

Area Percent Report

=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	37.862	MM	1.3318	3106.81641	38.88011	97.7907
2	41.364	MM	1.4177	70.19093	8.25196e-1	2.2093

Totals : 3177.00734 39.70530

Results obtained with enhanced integrator!

=====

*** End of Report ***

Data File D:\DATA\CEO\02270307.D

Sample Name: ceo-V-234A

=====

Injection Date	: 2/27/03 4:19:55 PM	Seq. Line	: 4
Sample Name	: ceo-V-234A	Location	: Vial 71
Acq. Operator	: ceo	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\METHODS\CEODH6.M		
Last changed	: 2/27/03 4:18:47 PM by ceo		
	(modified after loading)		
Analysis Method	: D:\METHODS\CEODH6.M		
Last changed	: 7/1/03 9:22:22 PM by ceo		
	(modified after loading)		

OBH
9:1 nHEX:IPA
0.8 mL/MIN

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

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	35.371	MM	1.7371	4274.37891	41.01061	94.9317
2	44.195	MM	2.7607	228.20667	1.37773	5.0683

Totals : 4502.58557 42.38834

Results obtained with enhanced integrator!

=====
*** End of Report ***

Data File C:\HPCHEM\1\DATA\CEO\04240302.D

Sample Name: ceo-VI-48A

=====

Injection Date	: 4/24/03 10:49:50 AM	Seq. Line	: 2
Sample Name	: ceo-VI-48A	Location	: Vial 62
Acq. Operator	: ceo	Inj	: 1
		Inj Volume	: 5 µl

Acq. Method : D:\METHODS\CEOODH6.M
Last changed : 4/24/03 11:04:38 AM by ceo
(modified after loading)
Analysis Method : D:\METHODS\CEOODH2.M
Last changed : 7/1/03 9:25:55 PM by ceo
(modified after loading)

ODH
95:5 nHEX:IPA
0.8 mL/MIN
no guard

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

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.628	BP	0.2842	29.76179	1.47016	2.2886
2	14.007	BB	0.3749	1270.70056	52.76957	97.7114

Totals : 1300.46235 54.23974

Results obtained with enhanced integrator!

=====
*** End of Report ***

Data File D:\DATA\CEO\02200302.D

Sample Name: ceo-V-259B

```
=====
Injection Date   : 2/20/03 12:34:35 PM      Seq. Line :    2
Sample Name      : ceo-V-259B              Location  : Vial 62
Acq. Operator    : ceo                     Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method      : D:\METHODS\CEOODH5.M
Last changed     : 2/20/03 11:10:31 AM by ceo
Analysis Method  : D:\METHODS\CEOODH6.M
Last changed     : 7/1/03 8:59:28 PM by ceo
                  (modified after loading)

ODH
96:4 nHEX:IPA
0.8 mL/MIN
no guard
=====
```

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

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.538	MM	0.5198	9.76844	3.13214e-1	2.2590
2	17.850	MM	0.5322	422.64951	13.23678	97.7410

Totals : 432.41795 13.54999

Results obtained with enhanced integrator!

=====
*** End of Report ***

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

Data File Name : C:\HPCHEM\2\DATA\ANDERSON\CEO6-25A.D
 Operator : ANDERSON Page Number : 1
 Instrument : INSTRUMEN Vial Number :
 Sample Name : Injection Number :
 Run Time Bar Code: Sequence Line :
 Acquired on : 02 Jul 03 10:30 AM Instrument Method: LONGRUN.MTH
 Report Created on: 02 Jul 03 11:47 AM Analysis Method : LONGRUN.MTH

Sig. 2 in C:\HPCHEM\2\DATA\ANDERSON\CEO6-25A.D

Pk#	Ret Time	Area	Height	Type	Width	Area %
1	71.722	32729	1982	BB	0.219	2.8187
2	73.593	1128411	27220	BB	0.500	97.1813

Total area = 1161141

=====

Data File C:\HPCHEM\1\DATA\CEO\02110302.D

Sample Name: ceo-V-300A

=====

Injection Date : 3/13/03 12:23:18 PM Seq. Line : 2
Sample Name : ceo-V-300A Location : Vial 62
Acq. Operator : ceo Inj : 1
Inj Volume : 5 µl

Acq. Method : D:\METHODS\CEODH3.M
Last changed : 3/13/03 12:22:17 PM by ceo
(modified after loading)

Analysis Method : D:\METHODS\CEODH3.M
Last changed : 6/28/03 8:08:19 PM by MMP
ODH
99:1 nHEX:IPA
0.8 mL/MIN

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

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.956	BB	0.1233	549.13025	68.64648	98.7982
2	6.834	MM	0.1367	6.67988	8.14557e-1	1.2018

Totals : 555.81013 69.46103

Results obtained with enhanced integrator!

=====
*** End of Report ***

Data File C:\HPCHEM\1\DATA\CEO\03150303.D

Sample Name: ceo-VI-18B

```
=====
Injection Date   : 3/15/03 2:14:33 PM      Seq. Line :    3
Sample Name      : ceo-VI-18B              Location  : Vial 63
Acq. Operator    : ceo                     Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method      : D:\METHODS\CEOODH5.M
Last changed     : 3/15/03 2:13:29 PM by ceo
                  (modified after loading)
Analysis Method  : D:\METHODS\CEOODH6.M
Last changed     : 7/1/03 8:59:28 PM by ceo
                  (modified after loading)
=====
```

ODH
96:4 nHEX:IPA
0.8 mL/MIN
no guard

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

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.013	BB	0.3921	197.80394	7.79420	9.9407
2	16.104	BB	0.4601	1792.04370	60.06741	90.0593

Totals : 1989.84764 67.86161

Results obtained with enhanced integrator!

=====
*** End of Report ***

Data File C:\HPCHEM\1\DATA\CEO\03190308.D

Sample Name: ceo-V-290A

=====

Injection Date : 3/19/03 3:50:44 PM Seq. Line : 8
Sample Name : ceo-V-290A Location : Vial 62
Acq. Operator : ceo Inj : 1
 Inj Volume : 5 µl

Acq. Method : D:\METHODS\CEOODH2.M
Last changed : 3/19/03 3:49:21 PM by ceo
 (modified after loading)

Analysis Method : D:\METHODS\CEOODH2.M
Last changed : 5/28/03 12:20:42 PM by ceo
 (modified after loading)

ODH
99.5:0.5 nHEX:IPA
0.8 mL/MIN
no guard

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

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	47.288	MM	2.4364	4421.76025	30.24774	96.8815
2	53.856	MM	2.2791	142.33278	1.04086	3.1185

Totals : 4564.09303 31.28861

Results obtained with enhanced integrator!

=====
*** End of Report ***

Data File C:\HPCHEM\1\DATA\CEO\03190302.D

Sample Name: ceo-VI-22A

=====

Injection Date : 3/19/03 9:20:42 AM Seq. Line : 2
Sample Name : ceo-VI-22A Location : Vial 62
Acq. Operator : ceo Inj : 1
Inj Volume : 5 µl

Acq. Method : D:\METHODS\CEODH5.M
Last changed : 3/19/03 9:19:23 AM by ceo
(modified after loading)
Analysis Method : D:\METHODS\CEODH5.M
Last changed : 6/27/03 4:16:20 PM by MMP
ODH
99.6:0.4 nHEX:IPA
0.8 mL/MIN
no guard

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

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.361	MM	1.1123	4536.80762	67.98120	98.4901
2	29.588	MM	1.2200	69.55017	9.50138e-1	1.5099

Totals : 4606.35779 68.93134

Results obtained with enhanced integrator!

=====
*** End of Report ***