

Supplementary Material

Asymmetric amination of cyclic β -keto esters catalyzed by amine-thiourea bearing multiple hydrogen bonding donors

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Table of Contents

1. General procedure for the asymmetric amination	1
2. HPLC spectra	2

General procedure for asymmetric amination of cyclic β -keto esters with dialkyl azodicarboxylates:

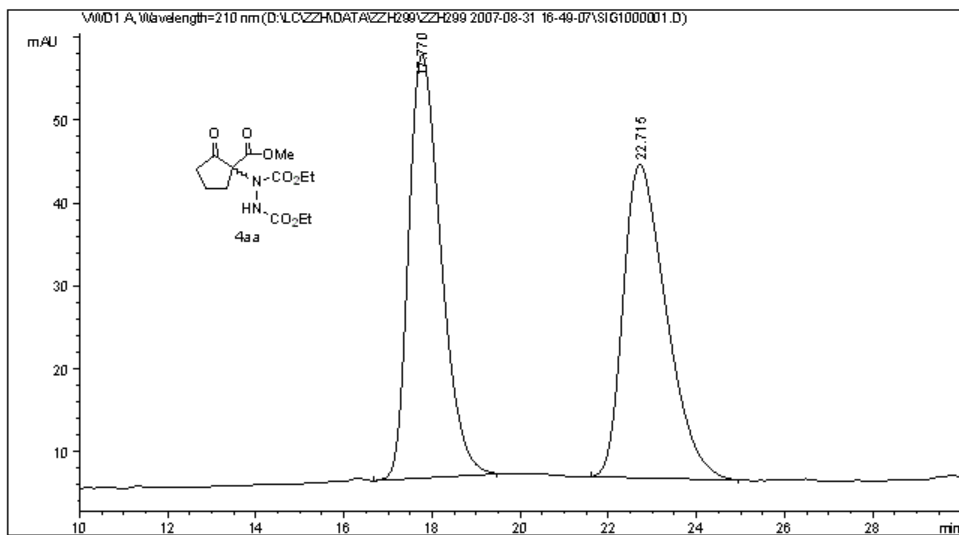
The dialkyl azodicarboxylates **3** (0.104 mmol) in ethyl acetate (0.2 mL) was added to a vial containing cyclic β -keto esters **2** (0.125 mmol) and catalyst **1d** (7 mg, 0.0104 mmol) in ethyl acetate (0.3 mL) at -78°C . TLC analysis indicated completion of the reaction after about 1-8 h. Then the reaction mixture was concentrated in vacuo to obtain the crude product. The crude product was purified by flash silica gel chromatography to afford the product. **4aa** ^1H NMR (300 MHz, CDCl_3) δ 6.88 (br, 1H), 4.22-4.28 (m, 4H), 3.86 (s, 3H), 2.75 (m, 2H), 2.40 (m, 2H), 2.11 (m, 1H), 1.78 (m, 1H), 1.30-1.37 (m, 6H); HPLC analysis (Chiralcel AS-H, *i*-PrOH /Hexane = 10/90, flow rate: 1 mL/min, $\lambda = 210$ nm, $t_{\text{minor}} = 24.8$ min, $t_{\text{major}} = 19.3$ min, 95% ee.

Data File D:\LC\ZZH\DATA\ZZH299\ZZH299 2007-08-31 16-49-07\SIG1000001.D
 Sample Name: ZZH299-R

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                                           Inj Volume: 5 µl

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Method Info     : ASH-10-90-210NM-LML-100MIN
  
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 Area Percent Report
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Sorted By : Signal
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 Dilution : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: WMD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	17.770	VB	0.7905	2606.84595	51.20568	50.3346
2	22.715	BB	1.0383	2572.18359	37.88078	49.6654

Totals : 5179.02954 89.08647

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Instrument 1 11/19/2008 1:53:23 PM xzy

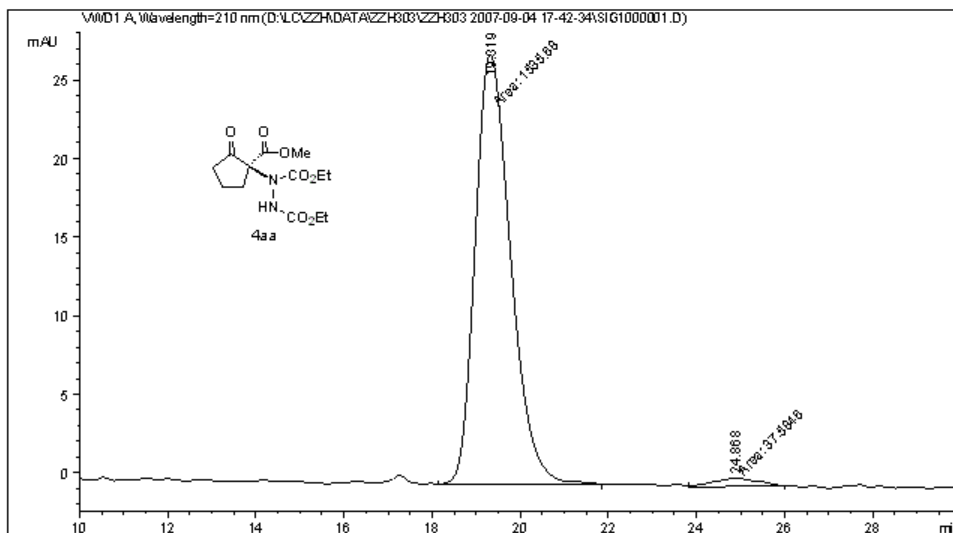
Page 1 of 1

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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: WVD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	19.319	MM	0.9347	1535.68347	97.6110	27.38264
2	24.868	MM	1.1650	37.58461	2.3890	5.37674e-1

Totals : 1573.26808 27.92031

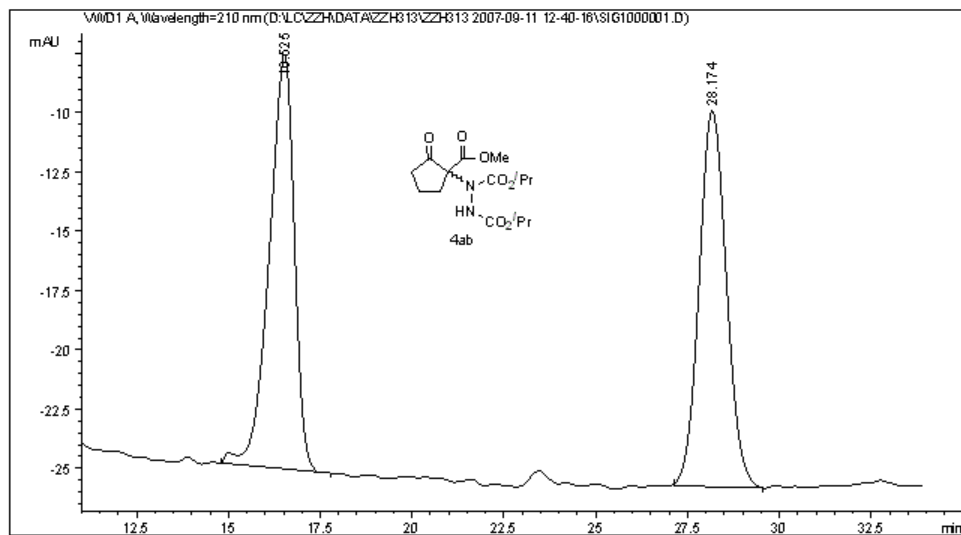
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 Area Percent Report
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 Dilution : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: WVD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	16.525	BB	0.7134	830.56647	50.3890	17.46838
2	28.174	BB	0.7954	817.74158	49.6110	15.85479

Totals : 1648.30804 33.32317

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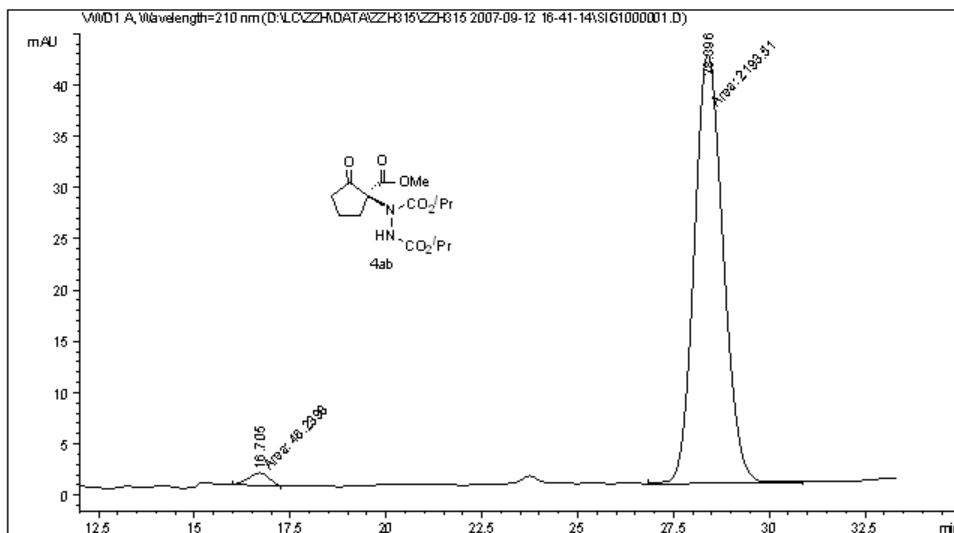
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 Area Percent Report
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Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
    
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Signal 1: WVD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	16.705	MM	0.6319	46.23982	2.0645	1.21968
2	28.396	MM	0.8744	2193.51270	97.9355	41.80984

Totals : 2239.75251 43.02952

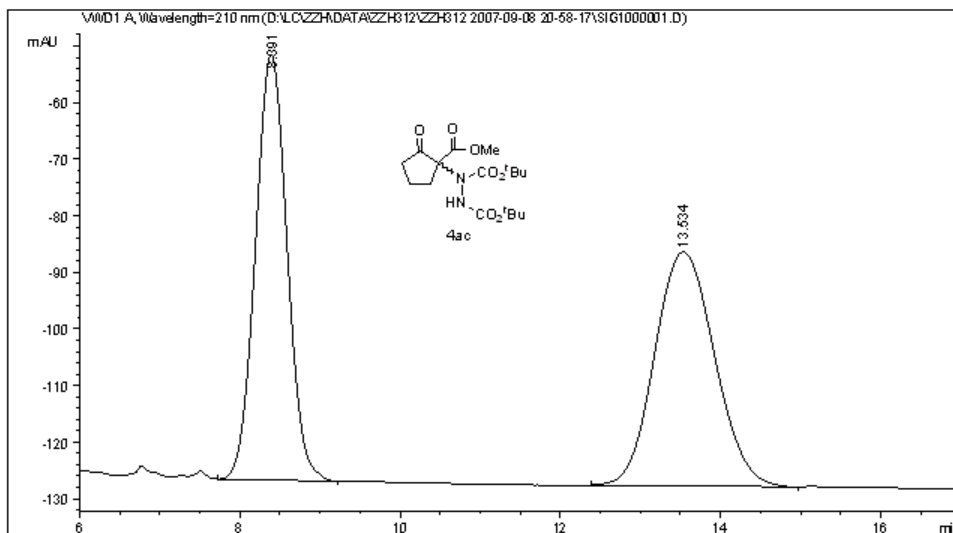
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Injection Date  : 9/8/2007 8:59:58 PM      Inj       :    1
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Acq. Method     : D:\LC\ZZH\data\ZZH312\ZZH312 2007-09-08 20-58-17\ADH-10-90-210NM-LML-100MIN.M
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 Area Percent Report
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Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: WMD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	8.391	VB	0.4288	2061.41211	48.9817	75.25227
2	13.534	BB	0.8195	2147.12549	51.0183	41.37218

Totals : 4208.53760 116.62445

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 *** End of Report ***

Instrument 1 11/19/2008 2:16:32 PM xzy

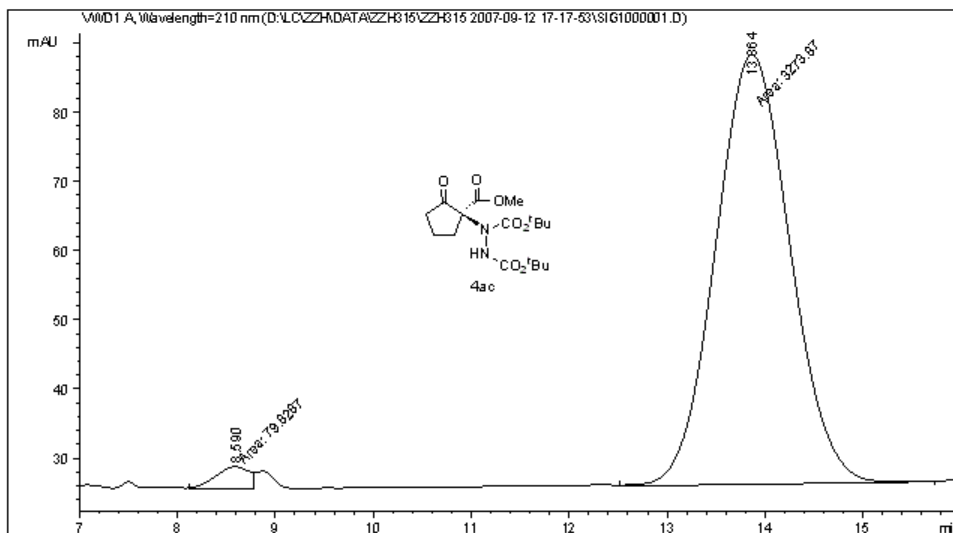
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Acq. Instrument : Instrument 1              Location  : Vial 66
Injection Date  : 9/12/2007 5:19:41 PM    Inj       :    1
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Acq. Method     : D:\LC\ZZH\data\ZZH315\ZZH315 2007-09-12 17-17-53\ADH-10-90-210NM-LML-100MIN.M
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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: WVD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	8.590	MF	0.4062	79.82874	2.3803	3.27518
2	13.864	MM	0.8794	3273.87231	97.6197	62.04833

Totals : 3353.70105 65.32351

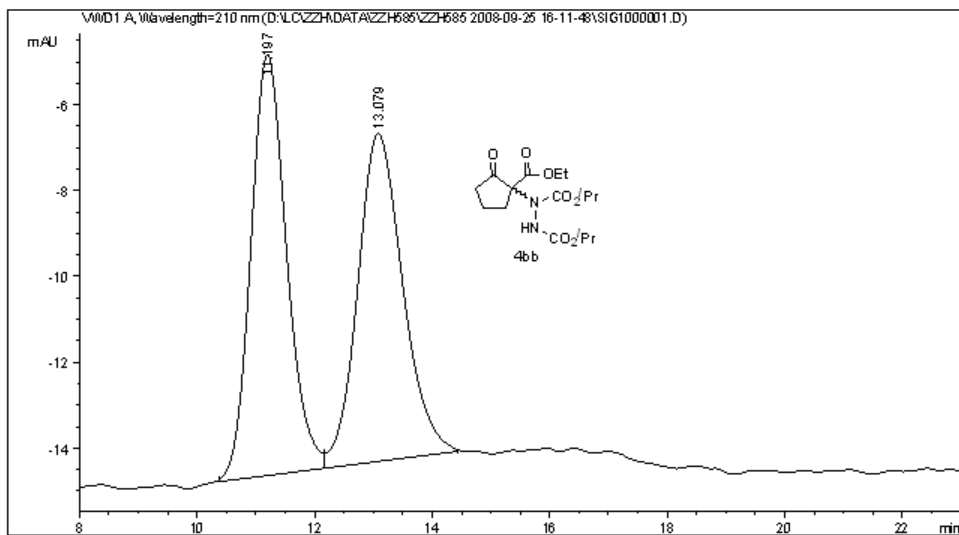
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Injection Date  : 9/25/2008 4:13:45 PM Inj       :    1
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Method Info    : ASH-5-95-210nm-60min
  
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 Area Percent Report
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Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: WMD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	11.197	BV	0.6421	412.69406	50.3415	9.81260
2	13.079	VB	0.7980	407.09503	49.6585	7.63825

Totals : 819.78909 17.45085

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Instrument 1 11/19/2008 2:04:52 PM xzy

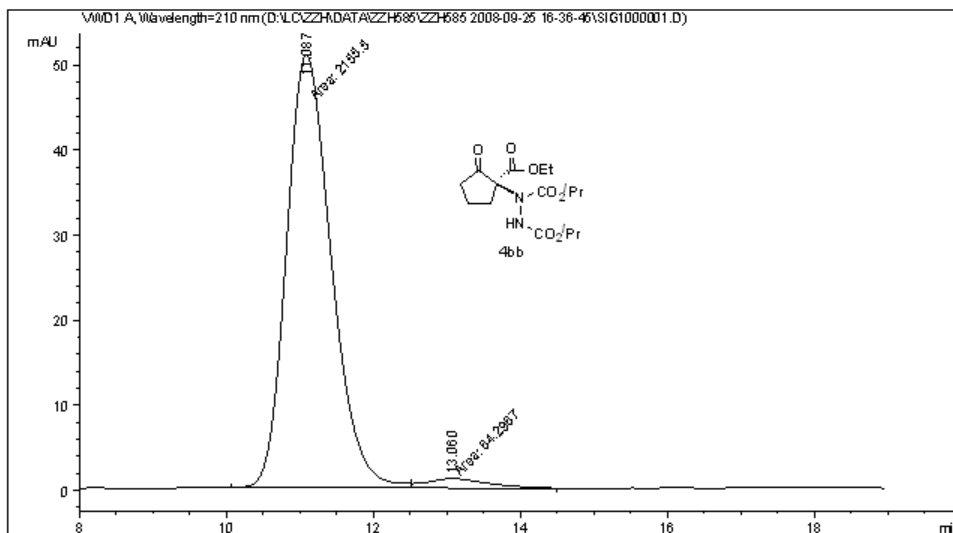
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 Sample Name: zzh585b

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Injection Date  : 9/25/2008 4:38:15 PM   Inj       :    1
                                           Inj Volume: 5 µl

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Method Info     : ASH-5-95-210nm-60min
    
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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: WVD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	11.087	MF	0.7052	2155.50342	97.1035	50.94019
2	13.060	FM	0.9603	64.29674	2.8965	1.11596

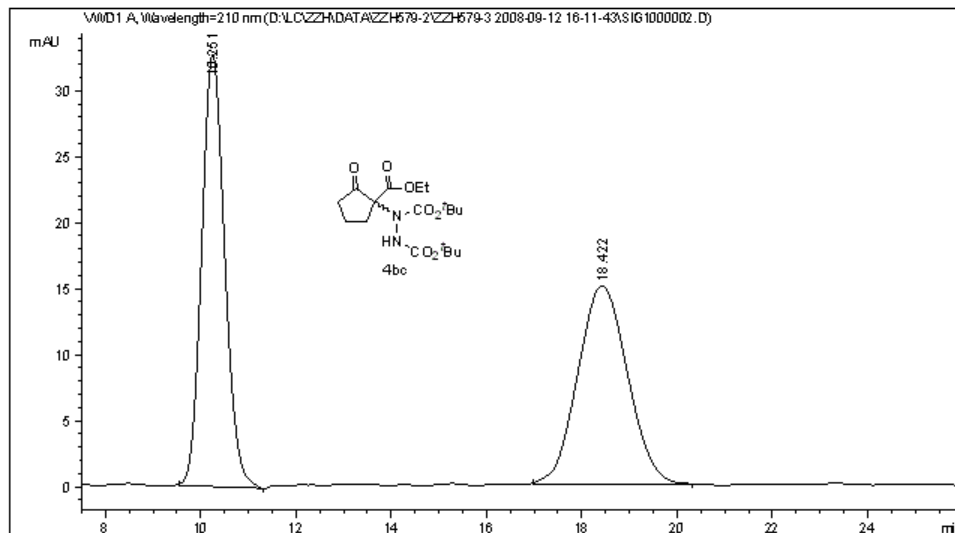
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 Area Percent Report
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Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: WWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.251	BB	0.5197	1099.93127	32.76785	50.2349
2	18.422	BB	1.1085	1089.64502	15.01972	49.7651

Totals : 2189.57629 47.78757

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 *** End of Report ***

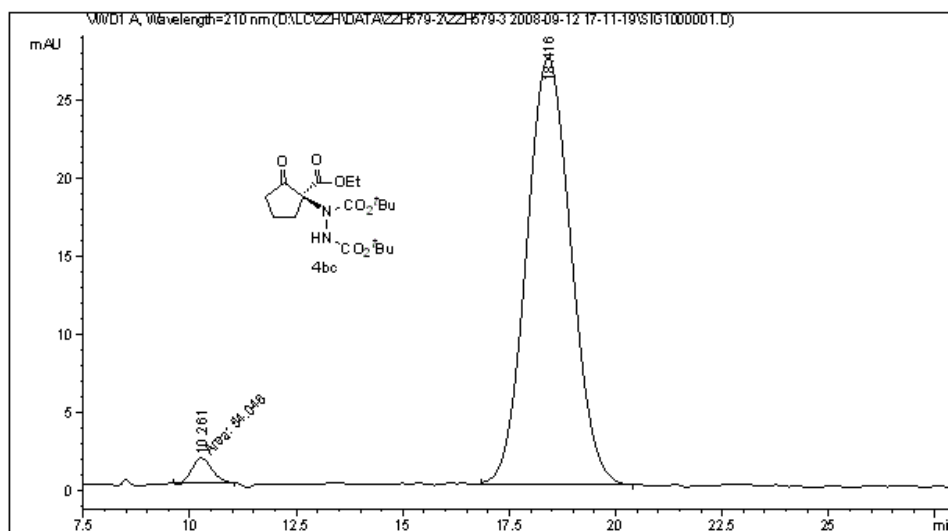
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 Area Percent Report
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Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: WVD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.261	MM	0.5609	54.04604	1.60601	2.6498
2	18.416	BB	1.1419	1985.59888	27.37238	97.3502

Totals : 2039.64492 28.97839

Instrument 1 3/3/2010 8:54:32 AM dxq

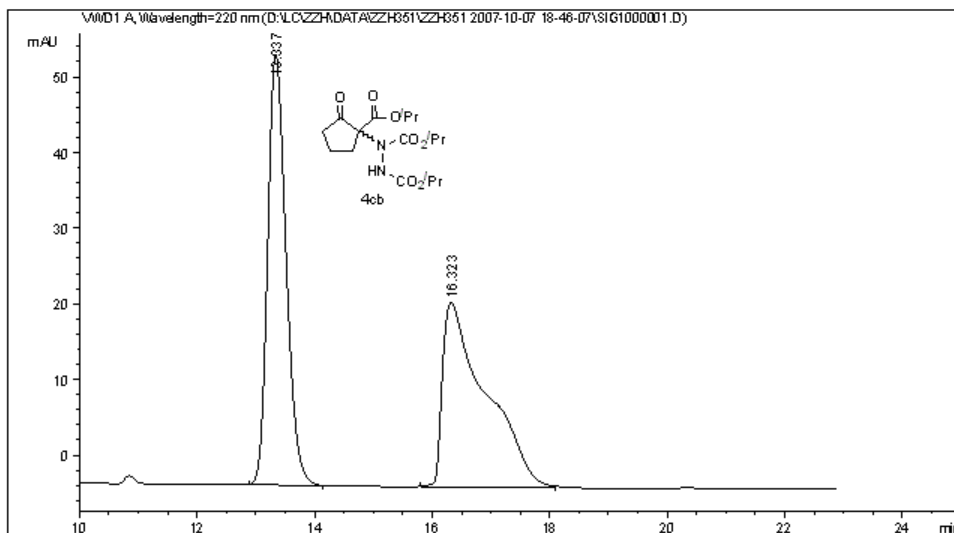
Page 1 of 2

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Injection Date  : 10/7/2007 6:47:32 PM     Inj       :    1
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Method Info     : ADH-5-95-220NM-08ML-100MIN
    
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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: WVD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	13.337	BB	0.3348	1235.49744	49.9414	56.91808
2	16.323	BB	0.6914	1238.39526	50.0586	24.32895

Totals : 2473.89270 81.24703

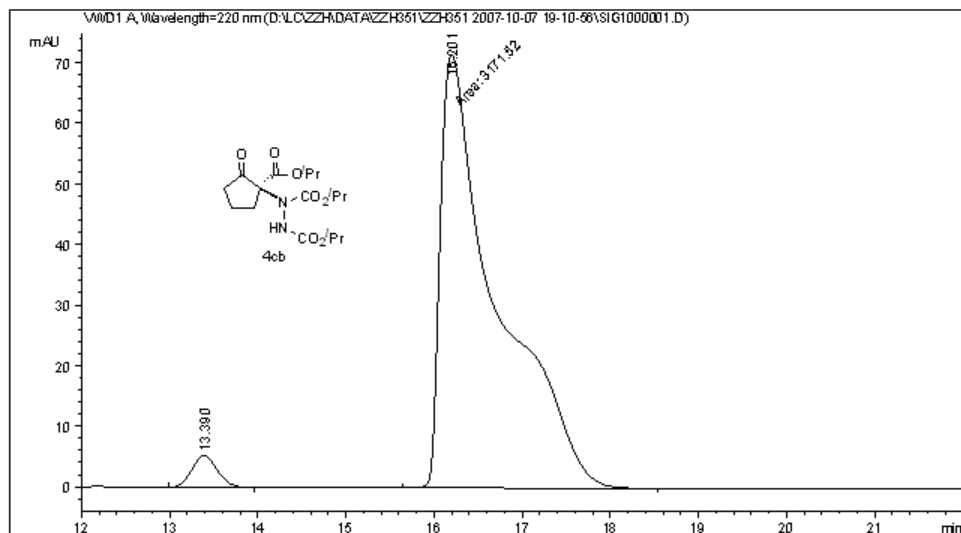
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Injection Date  : 10/7/2007 7:12:23 PM    Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method    : D:\LC\ZZH\data\ZZH351\ZZH351 2007-10-07 19-10-56\ADH-5-95-220NM-08ML-100MIN.M
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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: WMD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	13.390	BB	0.3195	109.22492	3.3293	5.28986
2	16.201	MM	0.7386	3171.52002	96.6707	71.57050

Totals : 3280.74494 76.86035

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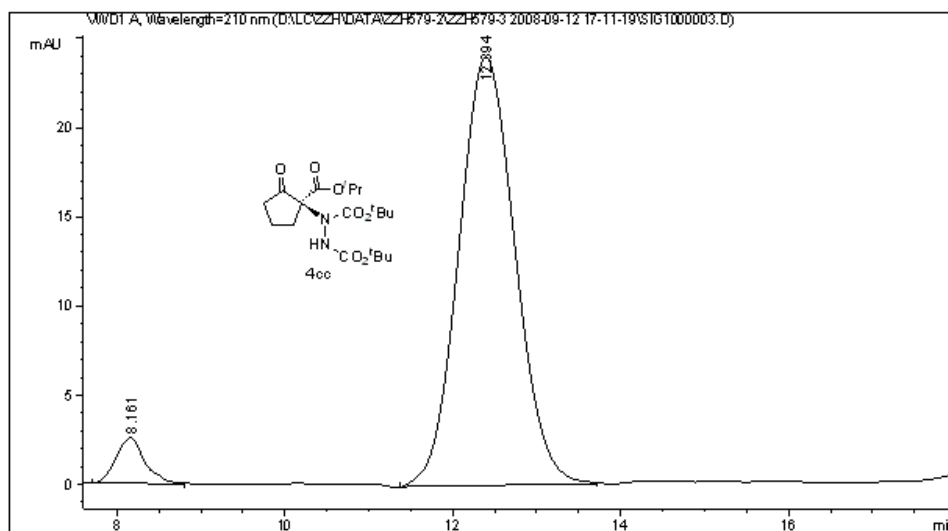
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Page 1 of 1

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                                                    Inj Volume: 5 µl
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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: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.161	BB	0.3712	61.28892	2.55862	5.2000
2	12.394	BB	0.7299	1117.33972	23.98139	94.8000

Totals : 1178.62864 26.54001

Instrument 1 3/3/2010 8:57:24 AM dxq

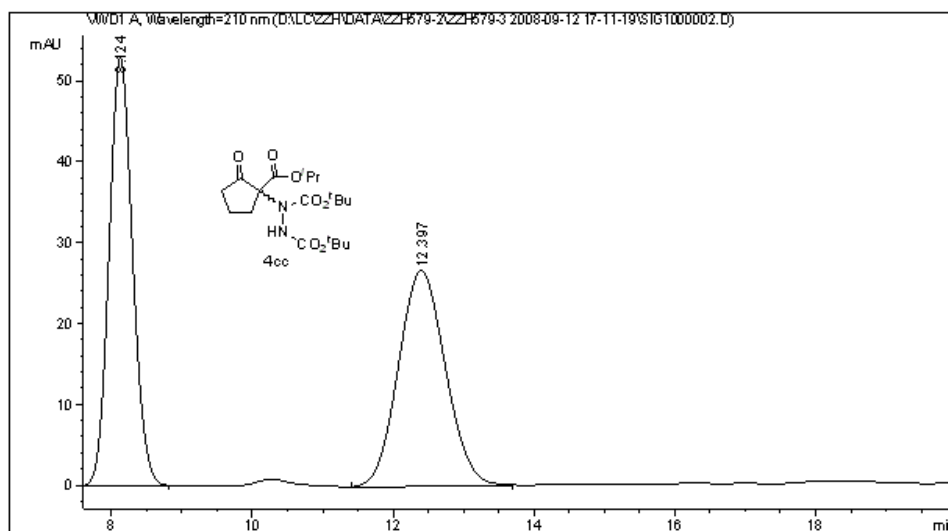
Page 1 of 2

Data File D:\LC\ZZH\DATA\ZZH579-2\ZZH579-3 2008-09-12 17-11-19\SIG1000002.D
 Sample Name: ZZH597BR

```

=====
Acq. Operator   : ZHANG ZHIHAI                      Seq. Line :    2
Acq. Instrument : Instrument 1                      Location  : Vial 65
Injection Date  : 9/12/2008 5:44:28 PM             Inj       :    1
                                                    Inj Volume: 5 µl

Acq. Method     : D:\LC\ZZH\data\ZZH579-2\ZZH579-3 2008-09-12 17-11-19\ADH-95-5-210NM-1ML-
                 30MIN.M
Last changed    : 9/12/2008 2:30:54 PM by dxq
Analysis Method : D:\LC\ZZH\DATA\ZZH579-2\ZZH579-3 2008-09-12 17-11-19\SIG1000002.D\DA.M (
                 ADH-95-5-210NM-1ML-30MIN.M)
Last changed    : 3/3/2010 8:55:55 AM by dxq
                 (modified after loading)
Method Info     : ADH-95-5-210NM-1ML/MIN-30MIN
  
```



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

```

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

Signal 1: WVD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.124	BB	0.3603	1226.34497	52.98310	49.8045
2	12.397	BB	0.7258	1235.97400	26.66160	50.1955

Totals : 2462.31897 79.64470

Instrument 1 3/3/2010 8:56:01 AM dxq

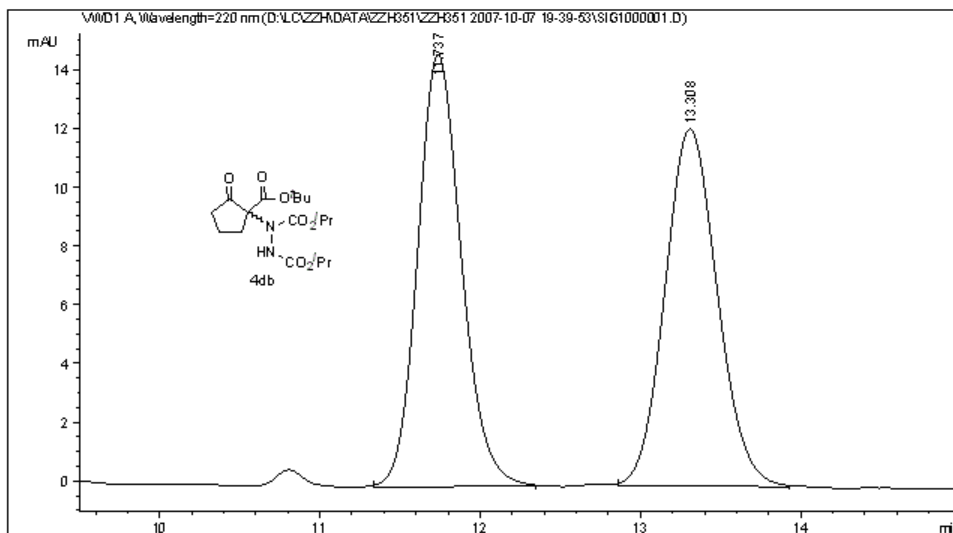
Page 1 of 2

Data File D:\LC\ZZH\DATA\ZZH351\ZZH351 2007-10-07 19-39-53\SIG1000001.D
 Sample Name: zzh351B-R

```

=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 6
Injection Date  : 10/7/2007 7:41:20 PM    Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method     : D:\LC\ZZH\data\ZZH351\ZZH351 2007-10-07 19-39-53\ADH-5-95-220NM-08ML-100MIN.M
Last changed    : 9/13/2007 4:47:00 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH351\ZZH351 2007-10-07 19-39-53\SIG1000001.D\DA.M (ADH-5-95-
                220NM-08ML-100MIN.M)
Last changed    : 11/19/2008 2:12:13 PM by xzy
                (modified after loading)
Method Info     : ADH-5-95-220NM-08ML-100MIN
    
```



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

```

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

Signal 1: WVD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	11.737	BB	0.2680	278.54340	50.2561	14.70236
2	13.308	BB	0.3536	275.70404	49.7439	12.14711

Totals : 554.24744 26.84947

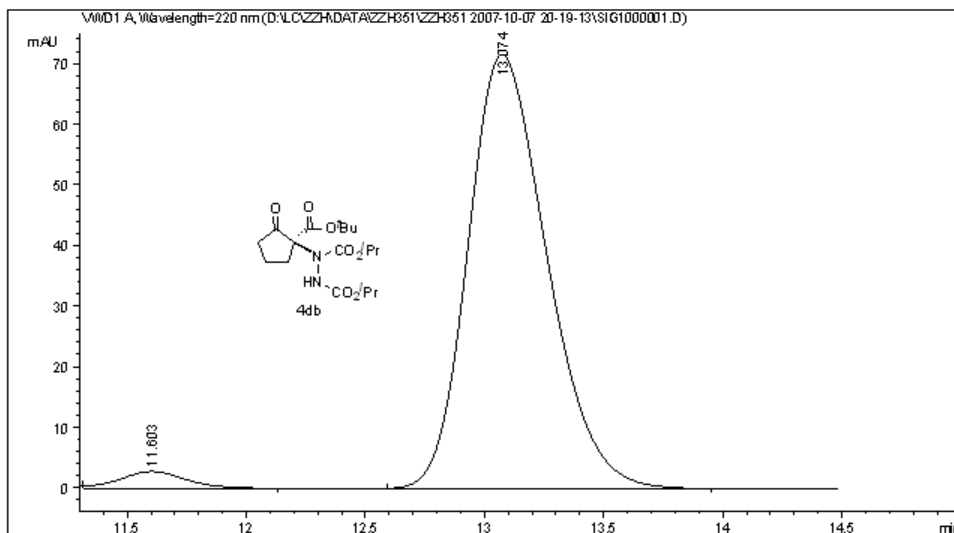
=====
 *** End of Report ***

Data File D:\LC\ZZH\DATA\ZZH351\ZZH351 2007-10-07 20-19-13\SIG1000001.D
 Sample Name: zzh351B

```

=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 7
Injection Date  : 10/7/2007 8:20:41 PM     Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method     : D:\LC\ZZH\data\ZZH351\ZZH351 2007-10-07 20-19-13\ADH-5-95-220NM-08ML-100MIN.M
Last changed    : 9/13/2007 4:47:00 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH351\ZZH351 2007-10-07 20-19-13\SIG1000001.D\DA.M (ADH-5-95-
                220NM-08ML-100MIN.M)
Last changed    : 11/19/2008 2:13:46 PM by xzy
                (modified after loading)
Method Info     : ADH-5-95-220NM-08ML-100MIN
    
```



Area Percent Report

```

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

Signal 1: WVD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	11.603	VB	0.2989	55.61470	3.2633	2.81577
2	13.074	BB	0.3558	1648.62378	96.7367	71.65225

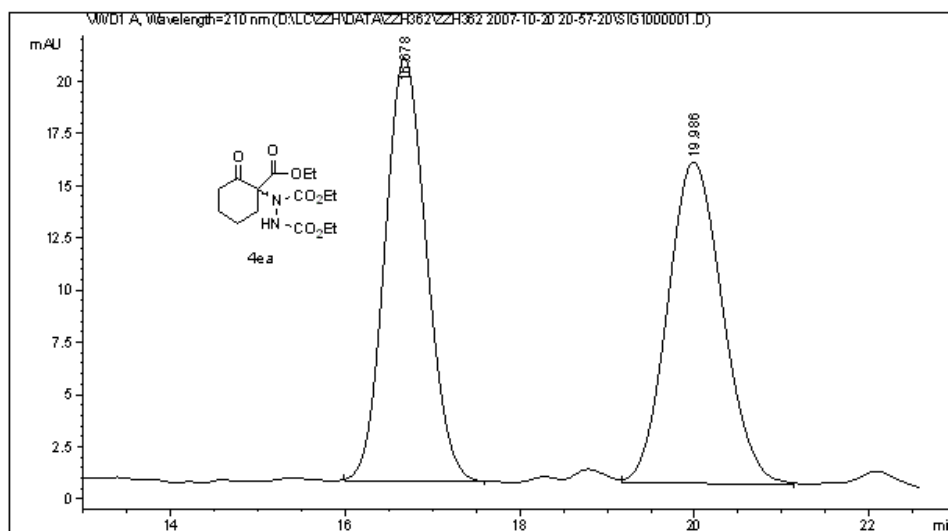
Totals : 1704.23848 74.46802

*** End of Report ***

Data File D:\LC\ZZH\DATA\ZZH362\ZZH362 2007-10-20 20-57-20\SIG1000001.D
 Sample Name: zzh362a-r

```

=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 35
Injection Date  : 10/20/2007 8:58:47 PM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH362\ZZH362 2007-10-20 20-57-20\ADH-10-90-210NM-1ML-
                  100MIN.M
Last changed    : 9/1/2007 11:37:00 AM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH362\ZZH362 2007-10-20 20-57-20\SIG1000001.D\DA.M (ADH-
                  10-90-210NM-1ML-100MIN.M)
Last changed    : 3/4/2010 8:36:38 PM by zhihai zhang
                  (modified after loading)
Method Info     : ADH-10-90-210NM-1ML-100MIN
  
```



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

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

Signal 1: WVD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	16.678	EB	0.5147	669.70599	20.28958	49.9038
2	19.986	VB	0.6825	672.28711	15.40185	50.0962

Totals : 1341.99310 35.69144

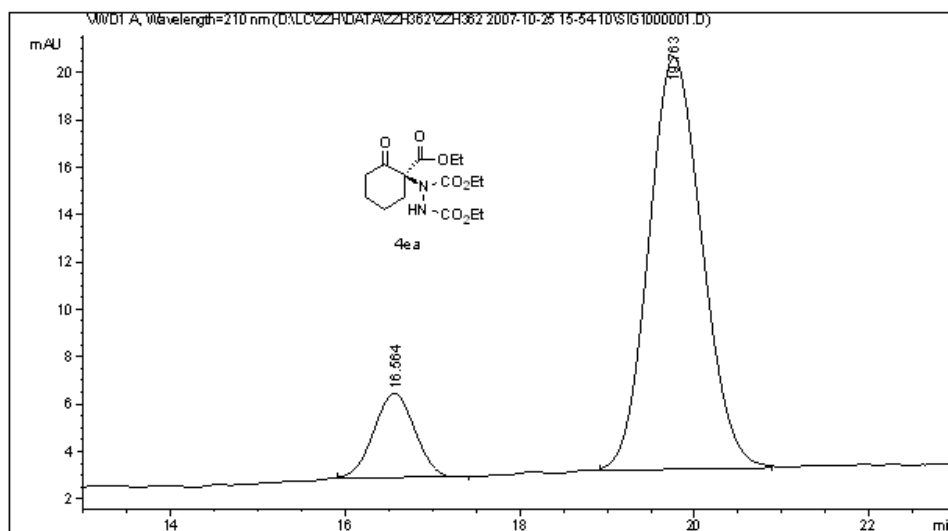
Instrument 1 3/4/2010 8:36:49 PM zhihai zhang

Page 1 of 2

Data File D:\LC\ZZH\DATA\ZZH362\ZZH362 2007-10-25 15-54-10\SIG1000001.D
 Sample Name: zzh362A

```

=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1             Location  : Vial 49
Injection Date  : 10/25/2007 3:55:34 PM   Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH362\ZZH362 2007-10-25 15-54-10\ADH-10-90-210NM-1ML-
                  100MIN.M
Last changed    : 9/1/2007 11:37:00 AM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH362\ZZH362 2007-10-25 15-54-10\SIG1000001.D\DA.M (ADH-
                  10-90-210NM-1ML-100MIN.M)
Last changed    : 3/4/2010 8:39:35 PM by zhihai zhang
                  (modified after loading)
Method Info     : ADH-10-90-210NM-1ML-100MIN
  
```



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

```

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

Signal 1: WVD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	16.564	BB	0.5034	115.44807	3.53428	13.5488
2	19.763	BB	0.6559	736.64532	17.38641	86.4512

Totals : 852.09340 20.92069

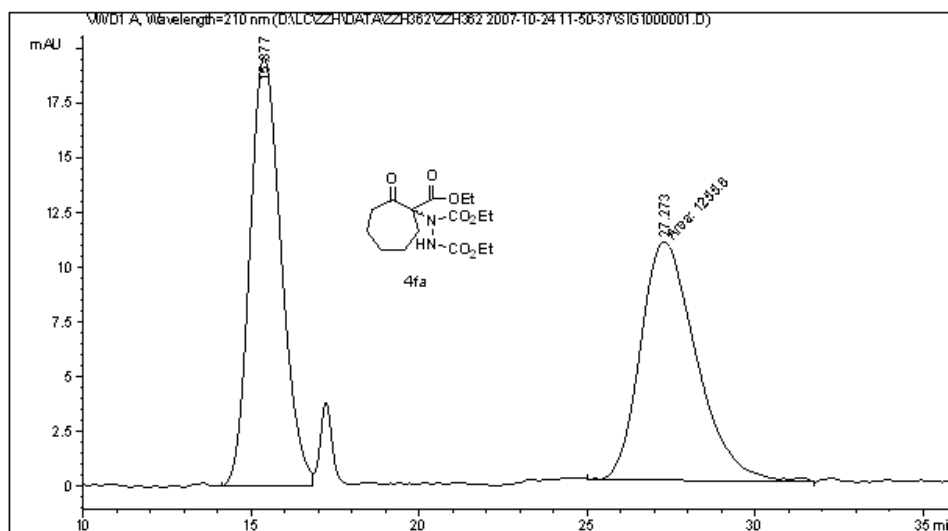
Instrument 1 3/4/2010 8:39:42 PM zhihai zhang

Page 1 of 2

Data File D:\LC\ZZH\DATA\ZZH362\ZZH362 2007-10-24 11-50-37\SIG1000001.D
 Sample Name: zzh362B-R

```

=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 45
Injection Date  : 10/24/2007 11:52:06 AM   Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH362\ZZH362 2007-10-24 11-50-37\ASH-10-90-210NM-1ML-
                  100MIN.M
Last changed    : 8/28/2007 4:06:26 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH362\ZZH362 2007-10-24 11-50-37\SIG1000001.D\DA.M (ASH-
                  10-90-210NM-1ML-100MIN.M)
Last changed    : 3/3/2010 9:27:33 AM by dxq
                  (modified after loading)
Method Info     : ASH-10-90-210NM-1ML-100MIN
  
```



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

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

Signal 1: WVD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	15.377	BV	1.0030	1266.76050	19.52554	50.2213
2	27.273	MM	1.9259	1255.59680	10.86591	49.7787

Totals : 2522.35730 30.39145

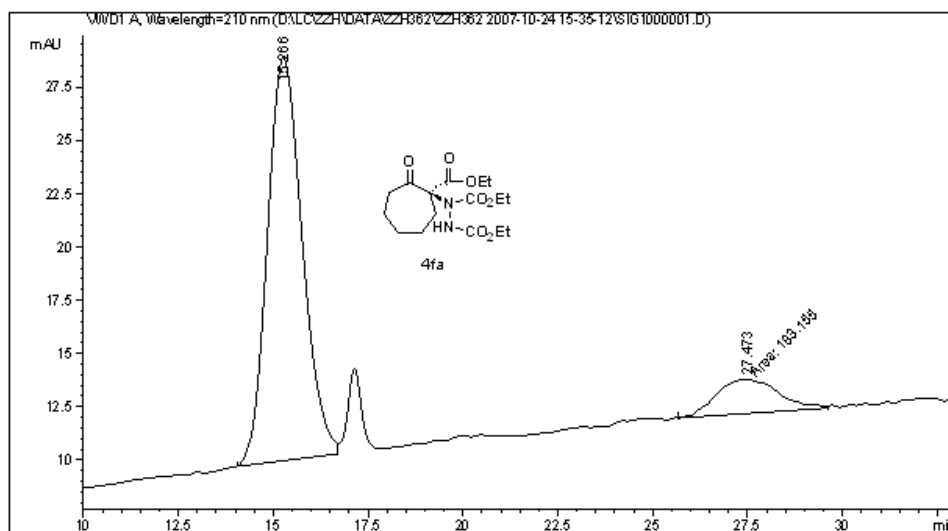
Instrument 1 3/3/2010 9:27:36 AM dxq

Page 1 of 2

Data File D:\LC\ZZH\DATA\ZZH362\ZZH362 2007-10-24 15-35-12\SIG1000001.D
 Sample Name: zzh362B

```

=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 46
Injection Date  : 10/24/2007 3:36:37 PM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH362\ZZH362 2007-10-24 15-35-12\ASH-10-90-210NM-1ML-
                  100MIN.M
Last changed    : 8/28/2007 4:06:26 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH362\ZZH362 2007-10-24 15-35-12\SIG1000001.D\DA.M (ASH-
                  10-90-210NM-1ML-100MIN.M)
Last changed    : 3/3/2010 9:29:07 AM by dxq
                  (modified after loading)
Method Info     : ASH-10-90-210NM-1ML-100MIN
  
```



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

```

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

Signal 1: WVD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	15.266	BV	0.9781	1206.17407	18.92095	86.8170
2	27.473	MM	1.9219	183.15512	1.58831	13.1830

Totals : 1389.32919 20.50926

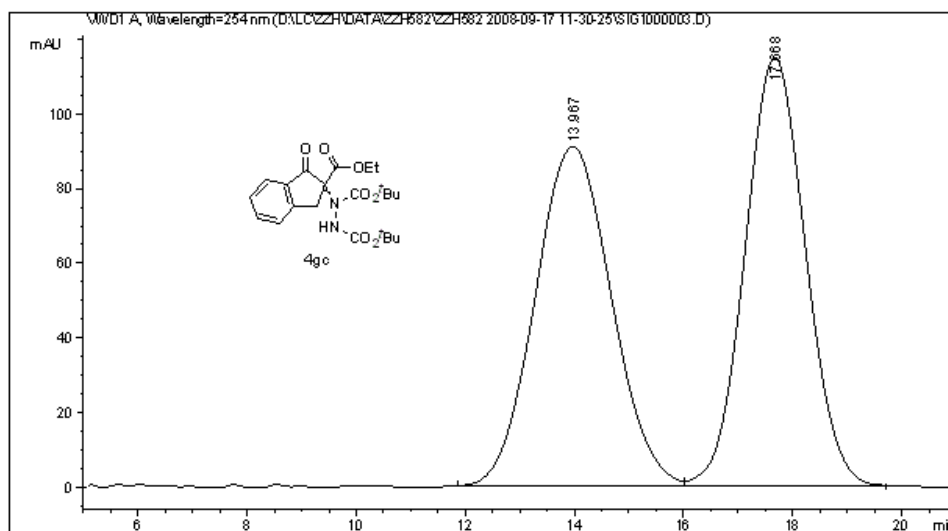
Instrument 1 3/3/2010 9:29:14 AM dxq

Page 1 of 2

Data File D:\LC\ZZH\DATA\ZZH582\ZZH582 2008-09-17 11-30-25\SIG1000003.D
 Sample Name: zzh582dr

```

=====
Acq. Operator   : zhang zhihai           Seq. Line :    3
Acq. Instrument : Instrument 1           Location  : Vial 63
Injection Date  : 9/17/2008 1:54:54 PM   Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH582\ZZH582 2008-09-17 11-30-25\ADH-10-90-254NM-1ML-40MIN.M
Last changed    : 12/4/2007 10:30:01 AM by zzh
Analysis Method : D:\LC\ZZH\DATA\ZZH582\ZZH582 2008-09-17 11-30-25\SIG1000003.D\DA.M (ADH-10-90-254NM-1ML-40MIN.M)
Last changed    : 3/3/2010 9:01:50 AM by dxq
                 (modified after loading)
Method Info     : ADH-10-90-254NM-1ML-40MIN
  
```



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

```

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

Signal 1: WVD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.967	BV	1.4975	8804.40625	90.97343	50.0429
2	17.668	VB	1.2003	8789.29688	114.62961	49.9571

Totals : 1.75937e4 205.60304

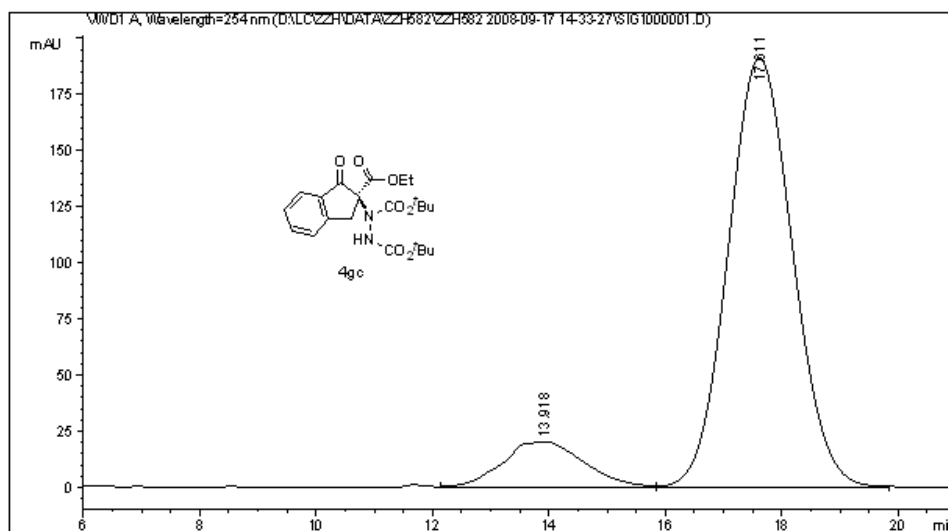
Instrument 1 3/3/2010 9:01:55 AM dxq

Page 1 of 2

Data File D:\LC\ZHZH\DATA\ZHZH582\ZHZH582 2008-09-17 14-33-27\SIG1000001.D
 Sample Name: zzh582d

```

=====
Acq. Operator   : zhang zhihai           Seq. Line :    1
Acq. Instrument : Instrument 1           Location  : Vial 64
Injection Date  : 9/17/2008 2:34:57 PM   Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZHZH\data\ZHZH582\ZHZH582 2008-09-17 14-33-27\ADH-10-90-254NM-1ML-
40MIN.M
Last changed    : 12/4/2007 10:30:01 AM by zzh
Analysis Method : D:\LC\ZHZH\DATA\ZHZH582\ZHZH582 2008-09-17 14-33-27\SIG1000001.D\DA.M (ADH-
10-90-254NM-1ML-40MIN.M)
Last changed    : 3/3/2010 9:03:20 AM by dxq
                 (modified after loading)
Method Info     : ADH-10-90-254NM-1ML-40MIN
  
```



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

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

Signal 1: WVD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.918	BV	1.3296	1957.66638	20.23481	11.7747
2	17.611	VB	1.2081	1.46684e4	191.19147	88.2253

Totals : 1.66261e4 211.42628

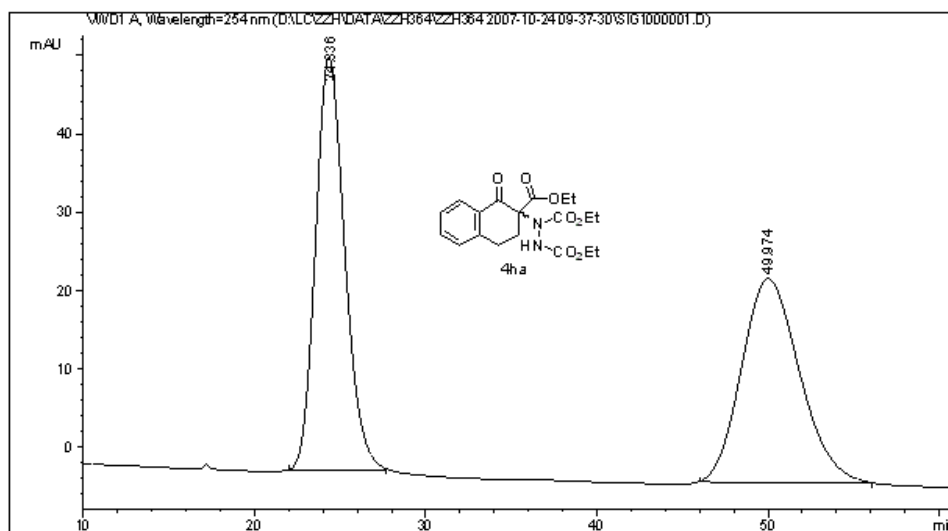
Instrument 1 3/3/2010 9:03:25 AM dxq

Page 1 of 2

Data File D:\LC\ZZH\DATA\ZZH364\ZZH364 2007-10-24 09-37-30\SIG1000001.D
 Sample Name: ZZH364B-R

```

=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1             Location  : Vial 43
Injection Date  : 10/24/2007 9:38:51 AM   Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH364\ZZH364 2007-10-24 09-37-30\ASH-10-90-1ML-254NM-100MIN.M
Last changed    : 9/28/2007 3:40:51 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH364\ZZH364 2007-10-24 09-37-30\SIG1000001.D\DA.M (ASH-10-90-1ML-254NM-100MIN.M)
Last changed    : 3/3/2010 9:14:31 AM by dxq
                 (modified after loading)
Method Info     : ASH-10-90-1ML-254NM-100MIN
=====
  
```



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

```

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

Signal 1: WVD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	24.336	BB	1.8087	6248.0764	52.74216	50.4794
2	49.974	BB	2.9391	6129.41260	26.04666	49.5206

Totals : 1.23775e4 78.78881

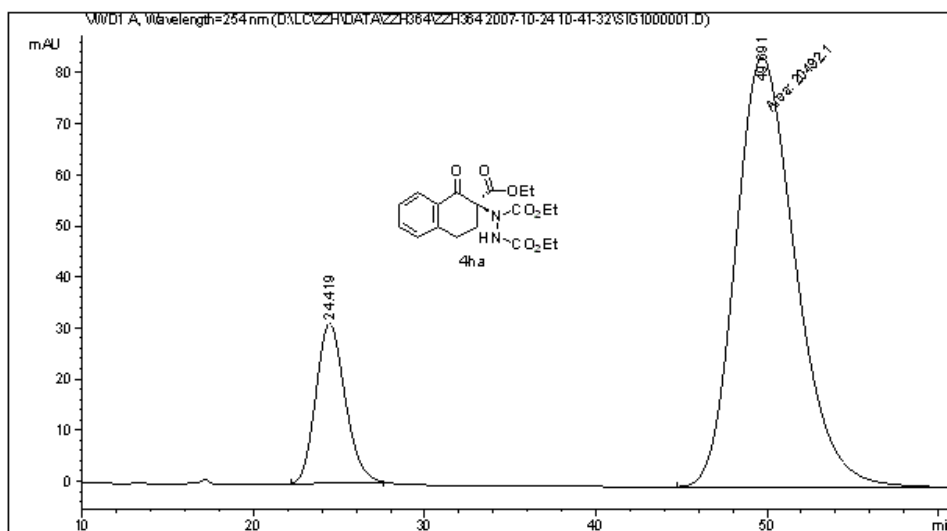
Instrument 1 3/3/2010 9:14:36 AM dxq

Page 1 of 2

Data File D:\LC\ZZH\DATA\ZZH364\ZZH364 2007-10-24 10-41-32\SIG1000001.D
 Sample Name: ZZH364B

```

=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 44
Injection Date  : 10/24/2007 10:43:04 AM   Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH364\ZZH364 2007-10-24 10-41-32\ASH-10-90-1ML-254NM-100MIN.M
Last changed    : 9/28/2007 3:40:51 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH364\ZZH364 2007-10-24 10-41-32\SIG1000001.D\DA.M (ASH-10-90-1ML-254NM-100MIN.M)
Last changed    : 3/3/2010 9:19:18 AM by dxq
                 (modified after loading)
Method Info     : ASH-10-90-1ML-254NM-100MIN
    
```



Area Percent Report

```

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

Signal 1: VWD1 A, Wavelength=254 nm

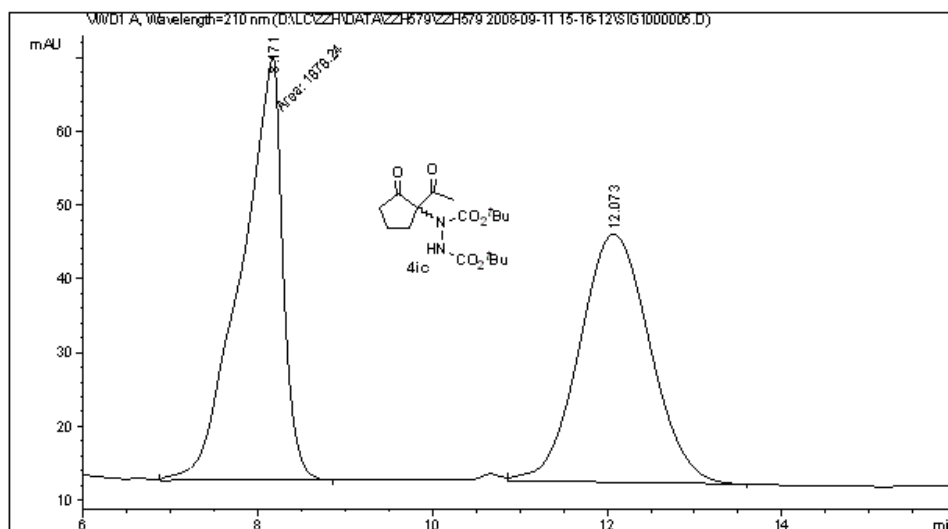
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	24.419	BB	1.7754	3713.09595	31.20837	15.3401
2	49.691	MM	4.0566	2.04921e4	84.19297	84.6599

Totals : 2.42052e4 115.40134

Data File D:\LC\ZZH\DATA\ZZH579\ZZH579 2008-09-11 15-16-12\SIG1000005.D
 Sample Name: ZZH579GR

```

=====
Acq. Operator   : zhang zhihai           Seq. Line :    5
Acq. Instrument : Instrument 1           Location  : Vial 67
Injection Date  : 9/11/2008 5:21:32 PM   Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH579\ZZH579 2008-09-11 15-16-12\ADH-HEX-ETOH-90-10-1ML-
30MIN-210NM.M
Last changed    : 9/11/2008 3:03:09 PM by ZHANG ZHIHAI
Analysis Method : D:\LC\ZZH\DATA\ZZH579\ZZH579 2008-09-11 15-16-12\SIG1000005.D\DA.M (ADH-
HEX-ETOH-90-10-1ML-30MIN-210NM.M)
Last changed    : 3/3/2010 4:13:09 PM by dxq
                 (modified after loading)
Method Info     : ADH-HEX/ETOH-90/10-1ML-30MIN-210NM
  
```



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

```

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

Signal 1: WVD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.171	MM	0.5472	1878.23633	57.21196	49.9760
2	12.073	VB	0.8810	1880.03857	33.61860	50.0240

Totals : 3758.27490 90.83056

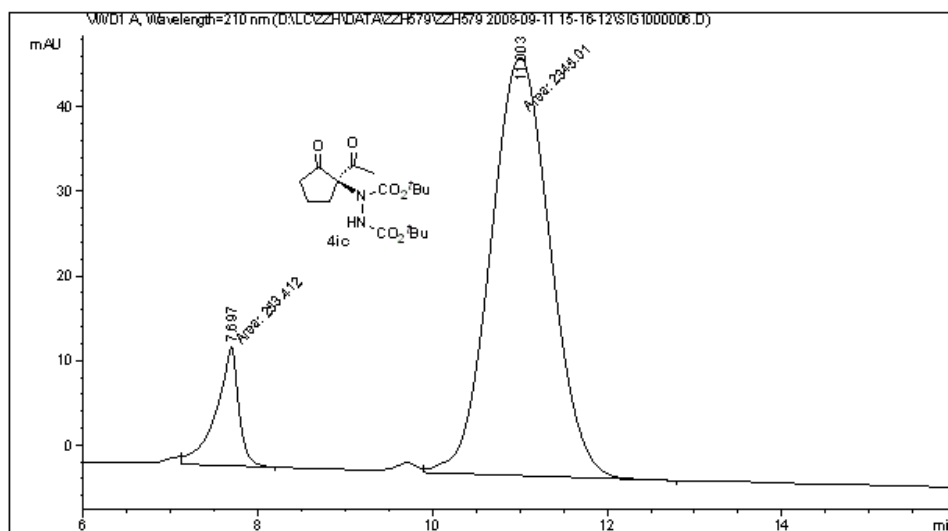
Instrument 1 3/3/2010 4:13:13 PM dxq

Page 1 of 2

Data File D:\LC\ZHZH\DATA\ZZH579\ZZH579 2008-09-11 15-16-12\SIG1000006.D
 Sample Name: ZZH579G

```

=====
Acq. Operator   : zhang zhihai           Seq. Line :    6
Acq. Instrument : Instrument 1           Location  : Vial 68
Injection Date  : 9/11/2008 5:52:37 PM Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZHZH\data\ZZH579\ZZH579 2008-09-11 15-16-12\ADH-HEX-ETOH-90-10-1ML-
30MIN-210NM.M
Last changed    : 9/11/2008 3:03:09 PM by ZHANG ZHIHAI
Analysis Method : D:\LC\ZHZH\DATA\ZZH579\ZZH579 2008-09-11 15-16-12\SIG1000006.D\DA.M (ADH-
HEX-ETOH-90-10-1ML-30MIN-210NM.M)
Last changed    : 3/3/2010 4:15:57 PM by dxq
(modified after loading)
Method Info     : ADH-HEX/ETOH-90/10-1ML-30MIN-210NM
=====
  
```



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

```

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

Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.697	FM	0.2996	253.41159	14.09611	9.7525
2	11.003	FM	0.7890	2345.01025	49.53565	90.2475

Totals : 2598.42184 63.63176

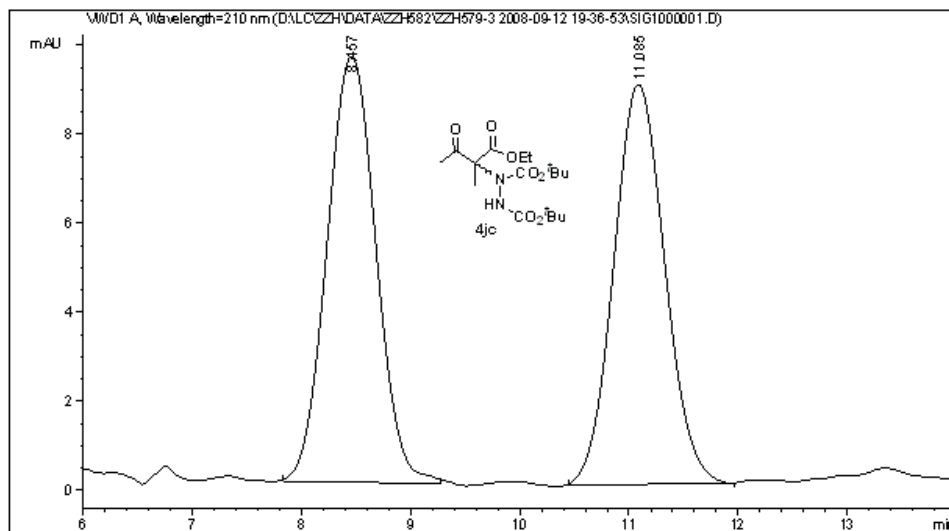
Instrument 1 3/3/2010 4:16:02 PM dxq

Page 1 of 2

Data File D:\LC\ZHZH\DATA\ZZH582\ZZH579-3 2008-09-12 19-36-53\SIG1000001.D
 Sample Name: ZZH582CR

```

=====
Acq. Operator   : ZHANG ZHIHAI                Seq. Line :    1
Acq. Instrument : Instrument 1                 Location  : Vial 75
Injection Date  : 9/12/2008 7:38:25 PM        Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZHZH\data\ZZH579-3 2008-09-12 19-36-53\ADH-10-90-210NM-1ML-60MIN.M
Last changed    : 9/11/2008 8:12:40 PM by ZHANG ZHIHAI
Analysis Method : D:\LC\ZHZH\DATA\ZZH582\ZZH579-3 2008-09-12 19-36-53\SIG1000001.D\DA.M (ADH-
                  10-90-210NM-1ML-60MIN.M)
Last changed    : 3/3/2010 4:17:03 PM by dxq
                  (modified after loading)
Method Info     : ADH-10-90-210NM-1ML-60MIN
  
```



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

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

Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.457	EB	0.4839	297.96051	9.57608	50.5278
2	11.085	EB	0.5036	291.73624	8.99589	49.4722

Totals : 589.69675 18.57196

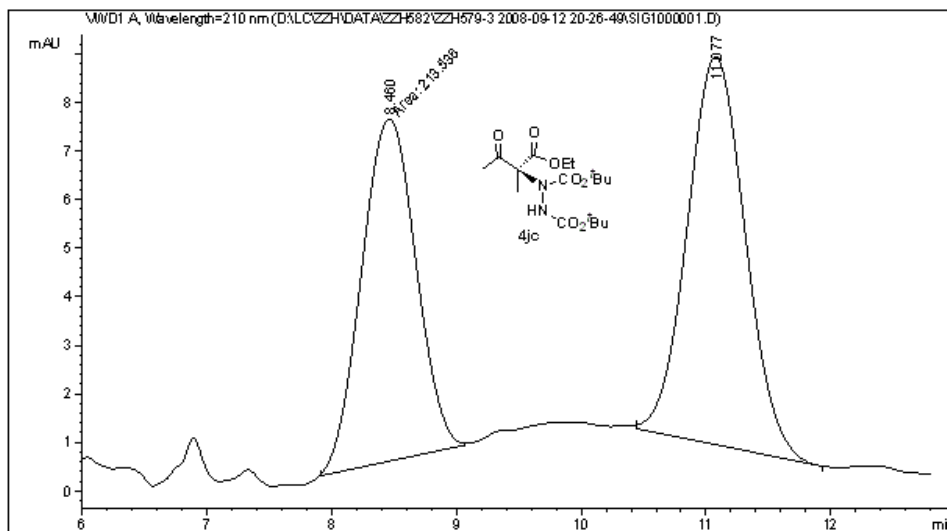
Instrument 1 3/3/2010 4:17:07 PM dxq

Page 1 of 2

Data File D:\LC\ZZH\DATA\ZZH582\ZZH579-3 2008-09-12 20-26-49\SIG1000001.D
 Sample Name: ZZH582C

```

=====
Acq. Operator   : ZHANG ZHIHAI           Seq. Line :    1
Acq. Instrument : Instrument 1           Location  : Vial 76
Injection Date  : 9/12/2008 8:28:21 PM   Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH579-3 2008-09-12 20-26-49\ADH-10-90-210NM-1ML-60MIN.M
Last changed    : 9/11/2008 8:12:40 PM by ZHANG ZHIHAI
Analysis Method : D:\LC\ZZH\DATA\ZZH582\ZZH579-3 2008-09-12 20-26-49\SIG1000001.D\DA.M (ADH-
                  10-90-210NM-1ML-60MIN.M)
Last changed    : 3/3/2010 4:20:09 PM by dxq
                  (modified after loading)
Method Info     : ADH-10-90-210NM-1ML-60MIN
    
```



Area Percent Report

```

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

Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	8.460	FM	0.5048	213.53587	7.04952	45.2509
2	11.077	EB	0.5034	258.35684	8.00025	54.7491

Totals : 471.89272 15.04976