


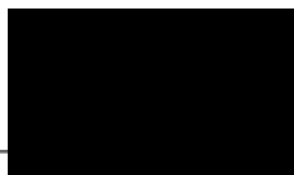
APPENDIX E

HPLC Methodology for Analysis of Steviol Glycoside Mixtures

 PureCircle PureCircle Sdn Bhd	Report#: KNN02102015001	Issue date: 02/10/15
		Revision date: -----
HPLC Assay of A95 samples in 5 commercial lots		Revision No: 00

HPLC Assay of A95 samples in 5 commercial lots

Prepared by:



(QC Chemist)

02/10/2015

Date


Approved by:



(QA/QC Manager)

2/10/2015

Date

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		Revision date: -----
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Objective


To determine rebaudioside D and others related steviol glycosides content in five commercial lots of *A95 samples* produced by *PureCircle*.

Samples

Five samples representing commercial lots of *A95* labeled as “PT180815”, “PT240815”, “PT280815”, “PT010915”, and “PT010915”.

Standards

1. Rebaudioside A Standard (USP Rockville, MD USA, Lot#F0I077), or equivalent;
2. Stevioside Standard (USP Rockville, MD USA, Lot#F0I080), or equivalent;
3. Rebaudioside B Standard (Chromadex Inc. Irvine, CA USA, Lot#00018237-001), or equivalent.
4. Rebaudioside C Standard (Chromadex Inc. Irvine, CA USA, Lot#00018228-3962), or equivalent;
5. Rebaudioside D Standard (PureCircle Sdn. Bhd., Malaysia, Lot#L03082015), or equivalent;
6. Rebaudioside F Standard (Chromadex Inc. Irvine, CA USA, Lot#00018305-103), or equivalent;
7. Dulcoside A Standard (Chromadex Inc. Irvine, CA USA, Lot#04949-002), or equivalent;
8. Rubusoside Standard, (PureCircle Sdn. Bhd., Malaysia, Lot#270308), or equivalent;

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9. Steviolbioside Standard (Chromadex Inc. Irvine, CA USA, Lot#00019349-1821), or equivalent.
10. Rebaudioside M Standard (PureCircle Sdn. Bhd., Malaysia, Lot#L11112013), or equivalent;
11. Rebaudioside E Standard (Chromadex Inc. Irvine, CA USA, Lot#00018235-131), or equivalent.
12. Rebaudioside O Standard (Chromadex Inc. Irvine, CA USA, Lot#00018233-134), or equivalent.
13. Rebaudioside N Standard (Chromadex Inc. Irvine, CA USA, Lot#0018232-121), or equivalent.

Solvents and Reagents

- Acetonitrile, HPLC grade (JT Baker, USA);
- Water, HPLC grade (JT Baker, USA);
- Sodium dihydrogen phosphate monohydrate, reagent grade (Merck, Germany);
- Ortho-phosphoric acid 85%, reagent grade (SYSTEM, USA).

Apparatus

1. Agilent 1200 HPLC system equipped with binary pump (G1312B), auto sampler (G1367D), thermostatted column compartment (G1316B) and DAD detector (G1315C), (Agilent Technologies, USA);
2. Analytical column, Poroshell 120 SB-C18, 4.6x150mm, 2.7µm (PN 683975-902), (Agilent Technologies, USA);
3. Analytical balance, XS205, (Mettler Toledo, USA);
4. Karl Fischer coulometer, Mettler Toledo DL-39, (Mettler Toledo, USA);
5. Volumetric (class A) and Laboratory glassware.

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Solution Preparation

1. **Phosphate buffer** : prepared 10 mmol/L sodium phosphate buffer with pH 2.6.
2. **Mobile phase** : Premix 1 - 75 % (v/v) Phosphate buffer in acetonitrile. Premix 2 - 68 % (v/v) Phosphate buffer in acetonitrile.
3. **Diluent** : 70 % (v/v) water in acetonitrile. [NOTE: Allow *Diluent* to come to room temperature before use.]

Solution Preparation

Rebaudioside D: approx. 700, 850, 1050, 1200 and 1400 mg/L Rebaudioside D in standard solution in *Diluent*.

Rebaudioside M: approx. 300, 400, 600, 700 and 800 mg/L Rebaudioside M in standard solution in *Diluent*.


Rebaudioside A: approx. 2, 10, 20, 40 and 100 mg/L USP Rebaudioside A in standard solution in *Diluent*.

Stevioside : approx. 0.5 (LOD), 2, 10, 20, 40 and 100 mg/L USP Stevioside solutions in *Diluent*.

Steviol Glycosides retention time marker solution (M13): approx. 100 mg/L (each) of Rebaudioside E, Rebaudioside O, Rebaudioside D, Rebaudioside M, Rebaudioside N, Rebaudioside A, Stevioside, Rebaudioside F, Rebaudioside C, Dulcoside A, Rubusoside, Rebaudioside B and Steviolbioside solution in *Diluent*.

Sample Solution

Sample solution was prepared at approx. 1600~2000 mg/L in *Diluent*. Water content was determined by Karl Fischer method.


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Chromatographic system

- Chromatograph – Agilent 1200 series HPLC equipped with binary pump, auto sampler, column oven and DAD detector.
- Column – Poroshell 120 SB-C18, 4.6x150mm, 2.7 µm at 40°C;
- Gradient setup:

Time (min)	Premix 1 (%)	Premix 2 (%)
0	100	0
12	100	0
12.5	50	50
13	0	100
40	0	100

- Flow rate – 0.5 mL/min;
- Injection volume – 5 µL;
- Detector – UV 210 nm.
- Run time – 45 minutes
- Post time – 10 minutes

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Calculation

Rebaudioside D : Prepare a standard curve for rebaudioside D by plotting rebaudioside D peak areas versus concentrations in mg/L, corrected for purity. From the standard curve, calculate the concentration (C_D) of rebaudioside D in the *Sample solution* in mg/L. Calculate the content (%) of rebaudioside D in the portion of the sample taken by the formula:

$$C_D / C_{SMP} \times 100 \%$$

C_D = Concentration of rebaudioside D in the *Sample solution* determined from the standard curve (mg/L)

C_{SMP} = Concentration of the sample in the *Sample solution* (mg/L)

Rebaudioside M : Prepare a standard curve for rebaudioside M by plotting rebaudioside M peak areas versus concentrations in mg/L, corrected for purity. From the standard curve, calculate the concentration (C_M) of rebaudioside M in the *Sample solution* in mg/L. Calculate the content (%) of rebaudioside M in the portion of the sample taken by the formula:

$$C_M / C_{SMP} \times 100 \%$$

C_M = Concentration of rebaudioside M in the *Sample solution* determined from the standard curve (mg/L)

C_{SMP} = Concentration of the sample in the *Sample solution* (mg/L)

Rebaudioside A : Prepare a standard curve for rebaudioside A by plotting rebaudioside A peak areas versus concentrations in mg/L, corrected for purity. From the standard curve, calculate the concentration (C_A) of rebaudioside A in the *Sample solution* in mg/L. Calculate the content (%) of rebaudioside A in the portion of the sample taken by the formula:

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$$C_A / C_{SMP} \times 100 \%$$

C_A = Concentration of rebaudioside A in the *Sample solution* determined from the standard curve (mg/L)

C_{SMP} = Concentration of the sample in the *Sample solution* (mg/L)

Stevioside : Prepare a standard curve for Stevioside by plotting Stevioside peak areas versus concentrations in mg/L, corrected for purity. From the standard curve, calculate the concentration (C_S) of Stevioside in the *Sample solution* in mg/L. Calculate the content (%) of Stevioside in the portion of the sample taken by the formula:

$$C_S / C_{SMP} \times 100 \%$$

C_S = Concentration of Stevioside in the *Sample solution* determined from the standard curve (mg/L)


C_{SMP} = Concentration of the sample in the *Sample solution* (mg/L)

Other steviol glycosides: Rebaudioside B, rebaudioside F, rebaudioside C, dulcoside A, rubusosodie and steviolbioside use the stevioside standard cuve prepared above to calculated the mg/L stevioside for each. Separately calculate the percents of each analyte (rebaudioside B, rebaudioside F, rebaudioside C, dulcoside A, rubusoside and steviolbioside) in the sample taken using the following formula which takes into account the difference in molecular weights between the analytes and stevioside:

$$(C_S \times f_s) / C_{SMP} \times 100 \%$$

C_S = Concentration of Stevioside equivalents in the *Sample solution* determined from the standard curve (mg/L)

f_s = ratio of the formula weight of others steviol glycosides to the formula weight of stevioside; 1.00 (rebaudioside B), 1.16 (rebaudioside F), 1.18 (rebaudioside C), 0.98 (dulcoside A), 0.80 (rubusoside), and 0.80 (steviolbioside)

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C_{SMP} = Concentration of the sample in the *Sample solution* (mg/L)

Rebaudioside E, rebaudioside O and rebaudioside N use the rebaudioside D standard curve prepared above to calculate the mg/L rebaudioside D for each. Separately calculate the percents of each analyte (Rebaudioside E, rebaudioside O and rebaudioside N) in the sample taken using the following formula which takes into account the difference in molecular weights between the analytes and stevioside:

$$(C_D \times f_D) / C_{SMP} \times 100 \%$$

C_D = Concentration of Stevioside equivalents in the *Sample solution* determined from the standard curve (mg/L)


f_D = ratio of the formula weight of others steviol glycosides to the formula weight of Rebaudioside D; 0.86 (rebaudioside E), 1.27 (rebaudioside O) and 1.13 (rebaudioside N)

C_{SMP} = Concentration of the sample in the *Sample solution* (mg/L)

System Suitability Requirements

System suitability was checked as below.

- Detector response: Peak to noise 0.5 mg/L Stevioside Standard solution – NLT 3 (see appendix A)
- Relative Standard Deviation : NMT 2.0 % for rebaudioside D peak area and retention time from the 1300 mg/L rebaudioside D standard solution (see appendix A)
- Tailing Factor : NMT 2.0 for the rebaudioside D peak from the 1300 mg/L rebaudioside D standard solution (see appendix A)

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Analysis

Analysis and calculation were performed as per instruction above.

The assay results are summarized in Table 1.

Table 1

A95 samples assay results

Lot#	Steviol Glycosides, % dry basis*													
	RebE	RebO	RebD	RebN	RebM	RebA	Stev	RebF	RebC	DulcA	Rubu	RebB	Sbio	TSG
PT180815	0.76	1.71	59.89	3.25	27.55	2.04	0.05	ND	ND	ND	ND	0.19	ND	95.44
PT240815	0.65	1.56	65.27	2.80	22.34	2.12	0.08	ND	ND	ND	ND	0.21	ND	95.03
PT280815	0.82	1.32	63.09	2.83	26.98	1.40	0.03	ND	ND	ND	ND	0.17	ND	96.66
PT010915	0.64	1.10	69.30	2.67	21.57	1.34	0.08	ND	ND	ND	ND	0.20	ND	96.90
PT040915	0.59	0.9	62.15	2.63	27.3	1.67	0.17	ND	0.07	ND	ND	0.37	ND	95.76

* Average of 3 duplicates is reported.

RebA – Rebaudioside A;

RebB – Rebaudioside B;

RebC – Rebaudioside C;

RebD – Rebaudioside D;

RebE – Rebaudioside E;

RebF – Rebaudioside F;

RebM – Rebaudioside M;

RebN – Rebaudioside N;

RebO – Rebaudioside O;

Rubu – Rubusoside;

Dulc.A– Dulcoside A;

Stev– Stevioside;

Sbio– Steviolbioside;


ND – Not detected.

<div><div><div></div><div>6</div></div><div>PureCircle</div></div> <div>PureCircle Sdn Bhd</div>	Report#: KNN02102015001	Issue date: 02/10/15
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APPENDIX A

System Suitability Check

- A1 Detector response
- A2 % RSD of Rebaudioside B peak area and retention time
- A3 Tailing factor

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APPENDIX A1

Detector response

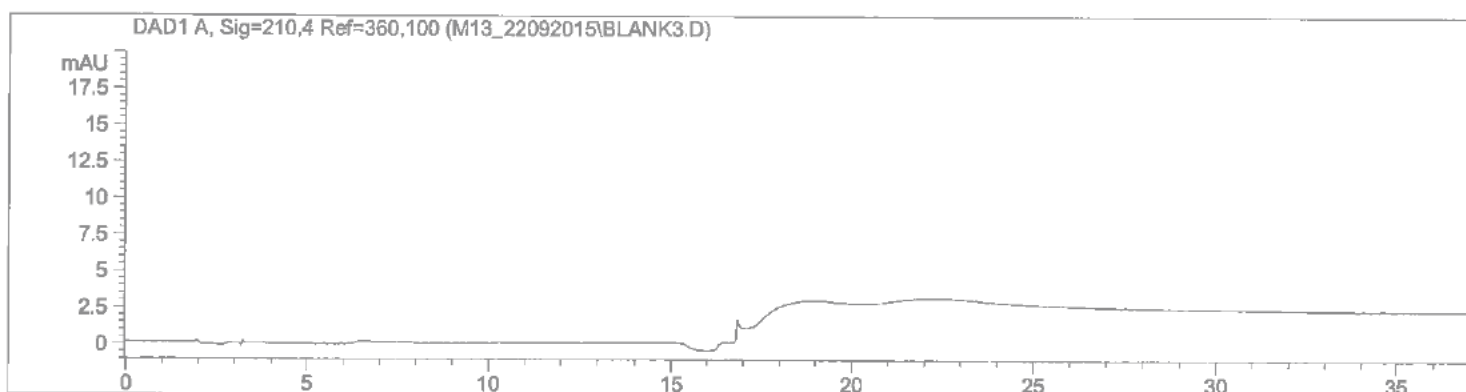
Sample Name: Blank

```
=====
Acq. Operator   : QC Chemist                      Seq. Line :    2
Acq. Instrument : HPLC 3                          Location  : Vial 1
Injection Date  : 9/23/2015 2:07:45 AM             Inj       :    3
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 11:33:49 AM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info      : Diluent (70% Water : 30% Acetonitrile)
=====
```



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

```
Sorted By           :      Signal
Calib. Data Modified :      Monday, September 28, 2015 10:31:55 AM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.920		0.0000	0.00000	0.0000	Reb E
2	9.643		0.0000	0.00000	0.0000	Reb O
3	10.288		0.0000	0.00000	0.0000	Reb D
4	11.160		0.0000	0.00000	0.0000	Reb N
5	13.020		0.0000	0.00000	0.0000	Reb M
6	20.477		0.0000	0.00000	0.0000	Reb A
7	20.630		0.0000	0.00000	0.0000	Stev
8	22.191		0.0000	0.00000	0.0000	Reb F
9	22.970		0.0000	0.00000	0.0000	Reb C
10	23.633		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Blank

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.501		0.0000	0.00000	0.0000	Rubu
12	32.271		0.0000	0.00000	0.0000	Reb B
13	33.279		0.0000	0.00000	0.0000	Sbio

Totals : 0.00000

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

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



Sample Name: Blank

```

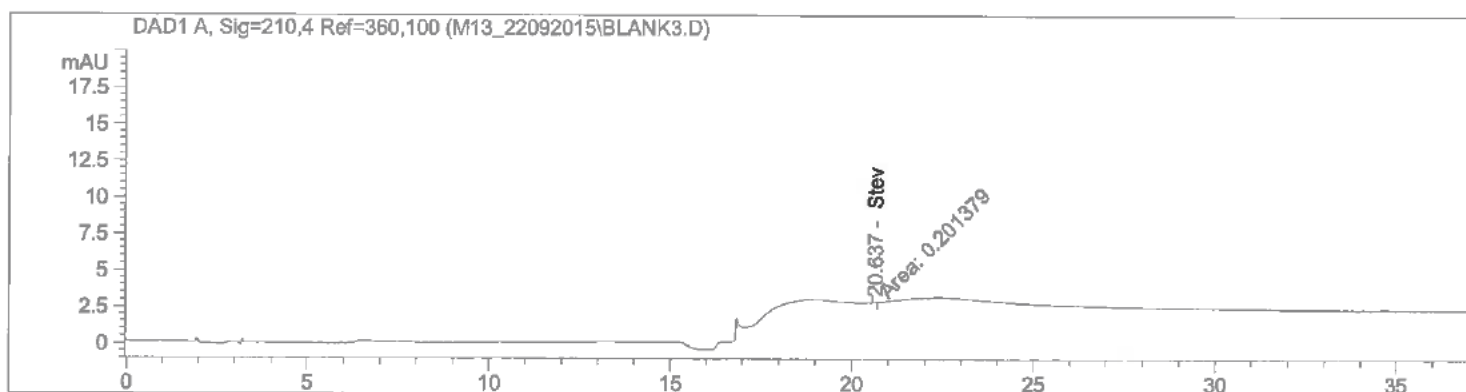
=====
Acq. Operator   : QC Chemist                      Seq. Line :    2
Acq. Instrument : HPLC 3                          Location  : Vial 1
Injection Date  : 9/23/2015 2:07:45 AM             Inj       :    3
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 11:35:05 AM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info      : Diluent (70% Water : 30% Acetonitrile)
=====

```



```

=====
Height Percent Report
=====

```

```

Sorted By           :      Signal
Calib. Data Modified :      Monday, September 28, 2015 11:34:20 AM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Height [mAU]	Height %	Name
1	8.920		0.0000	0.00000	0.0000	Reb E
2	9.643		0.0000	0.00000	0.0000	Reb O
3	10.288		0.0000	0.00000	0.0000	Reb D
4	11.160		0.0000	0.00000	0.0000	Reb N
5	13.020		0.0000	0.00000	0.0000	Reb M
6	20.477		0.0000	0.00000	0.0000	Reb A
7	20.637 MM		0.0974	3.44518e-2	100.0000	Stev
8	22.191		0.0000	0.00000	0.0000	Reb F
9	22.970		0.0000	0.00000	0.0000	Reb C
10	23.633		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Blank

Peak #	RetTime [min]	Type	Width [min]	Height [mAU]	Height %	Name
11	26.501		0.0000	0.00000	0.0000	Rubu
12	32.271		0.0000	0.00000	0.0000	Reb B
13	33.279		0.0000	0.00000	0.0000	Sbio

Totals : 3.44518e-2

3 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

Warning : Invalid calibration curve, (Stev)

*** End of Report ***



Sample Name: LOD

```

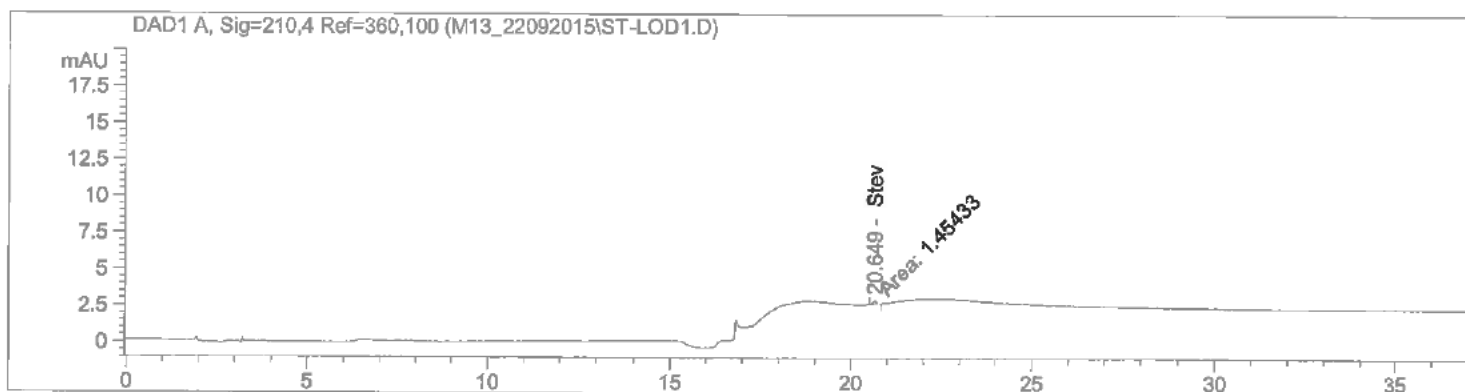
=====
Acq. Operator   : QC Chemist                      Seq. Line :    3
Acq. Instrument : HPLC 3                          Location  : Vial 2
Injection Date  : 9/23/2015 3:10:42 AM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 11:35:05 AM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : Limit of Detection, 0.4779 mg/L of Stevioside Std
=====

```



```

=====
                        Height Percent Report
=====

```

```

Sorted By           :      Signal
Calib. Data Modified :      Monday, September 28, 2015 11:34:20 AM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Height [mAU]	Height %	Name
1	8.920		0.0000	0.00000	0.0000	Reb E
2	9.643		0.0000	0.00000	0.0000	Reb O
3	10.288		0.0000	0.00000	0.0000	Reb D
4	11.160		0.0000	0.00000	0.0000	Reb N
5	13.020		0.0000	0.00000	0.0000	Reb M
6	20.477		0.0000	0.00000	0.0000	Reb A
7	20.649 MM		0.1011	2.39702e-1	100.0000	Stev
8	22.191		0.0000	0.00000	0.0000	Reb F
9	22.970		0.0000	0.00000	0.0000	Reb C
10	23.633		0.0000	0.00000	0.0000	Dulc.A



Sample Name: LOD

Peak #	RetTime [min]	Type	Width [min]	Height [mAU]	Height %	Name
11	26.501		0.0000	0.00000	0.0000	Rubu
12	32.271		0.0000	0.00000	0.0000	Reb B
13	33.279		0.0000	0.00000	0.0000	Sbio

Totals : 2.39702e-1

3 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

Warning : Invalid calibration curve, (Stev)

*** End of Report ***



'3D' Signal Overlay

*DAD1 A, Sig=210,4 Ref=360,100 (M13_22092015\ST-LOD1.D)
*DAD1 A, Sig=210,4 Ref=360,100 (M13_22092015\BLANK3.D)

mAU

3.4

3.3

3.2

3.1

3.0

2.9

2.8

20

20.5


21

21.5

22

22.5 min



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APPENDIX A2

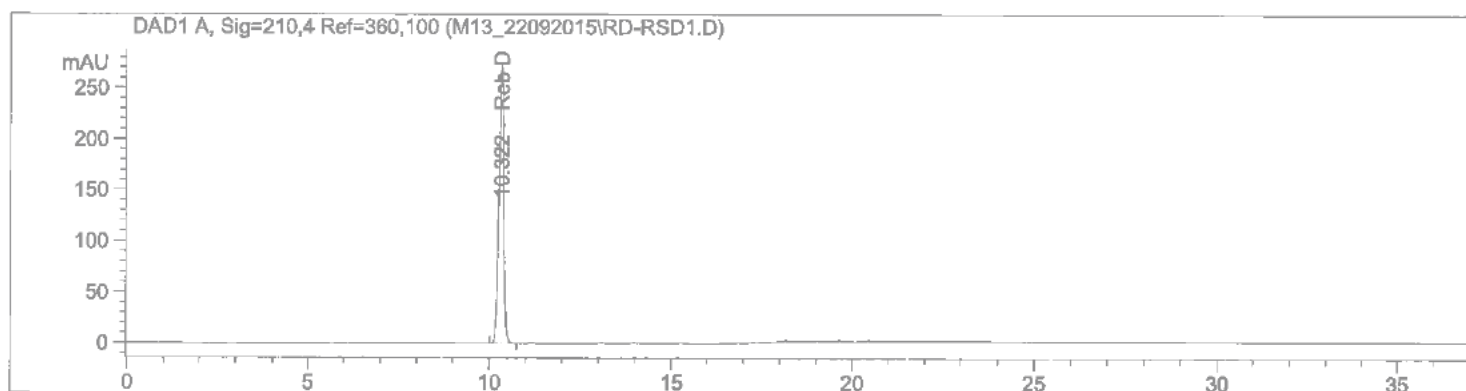
% RSD of rebaudioside D peak area and retention time

Sample Name: Reb D Standard

```
=====
Acq. Operator   : QC Chemist                      Seq. Line :    4
Acq. Instrument : HPLC 3                          Location  : Vial 3
Injection Date  : 9/23/2015 6:19:56 AM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 3:22:46 PM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info      : Reb D Std, 1301.5102 mg/L, Relative Standard Deviation
=====
```



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

```
Sorted By           :      Signal
Calib. Data Modified :      Thursday, October 01, 2015 3:22:46 PM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.970		0.0000	0.00000	0.0000	Reb E
2	9.710		0.0000	0.00000	0.0000	Reb O
3	10.322	BB	0.1415	2518.72827	100.0000	Reb D
4	11.232		0.0000	0.00000	0.0000	Reb N
5	13.138		0.0000	0.00000	0.0000	Reb M
6	20.400		0.0000	0.00000	0.0000	Reb A
7	20.600		0.0000	0.00000	0.0000	Stev
8	22.243		0.0000	0.00000	0.0000	Reb F
9	23.023		0.0000	0.00000	0.0000	Reb C
10	23.689		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb D Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.584		0.0000	0.00000	0.0000	Rubu
12	32.428		0.0000	0.00000	0.0000	Reb B
13	33.438		0.0000	0.00000	0.0000	Sbio

Totals : 2518.72827

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

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

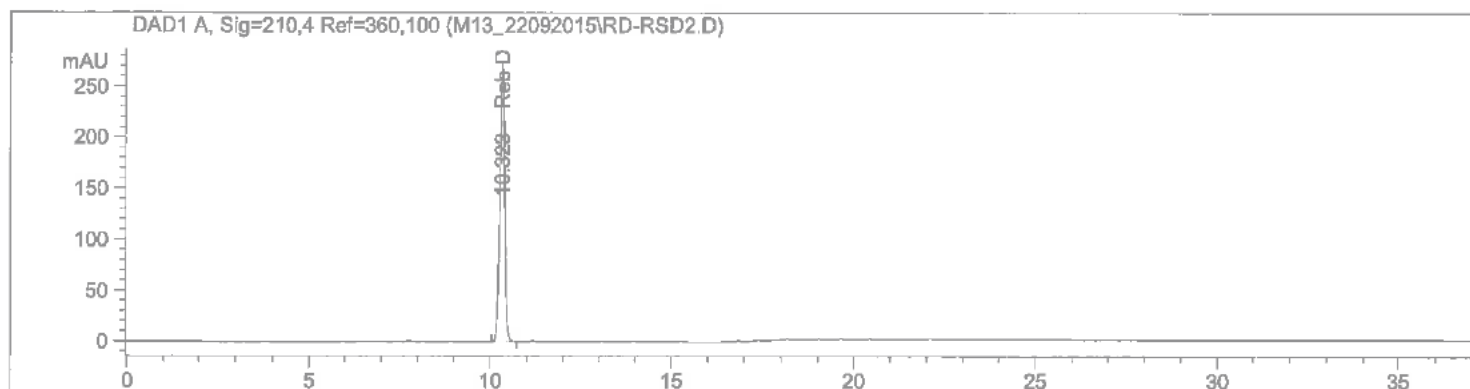
Sample Name: Reb D Standard

```

=====
Acq. Operator   : QC Chemist                      Seq. Line :    4
Acq. Instrument : HPLC 3                          Location  : Vial 3
Injection Date  : 9/23/2015 7:22:21 AM             Inj       :    2
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 3:22:46 PM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info      : Reb D Std, 1301.5102 mg/L, Relative Standard Deviation
  
```



```

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

```

Sorted By           : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:22:46 PM
Multiplier          : 1.0000
Dilution            : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.970		0.0000	0.00000	0.0000	Reb E
2	9.710		0.0000	0.00000	0.0000	Reb O
3	10.323	BB	0.1417	2517.77271	100.0000	Reb D
4	11.232		0.0000	0.00000	0.0000	Reb N
5	13.138		0.0000	0.00000	0.0000	Reb M
6	20.400		0.0000	0.00000	0.0000	Reb A
7	20.600		0.0000	0.00000	0.0000	Stev
8	22.243		0.0000	0.00000	0.0000	Reb F
9	23.023		0.0000	0.00000	0.0000	Reb C
10	23.689		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb D Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.584		0.0000	0.00000	0.0000	Rubu
12	32.428		0.0000	0.00000	0.0000	Reb B
13	33.438		0.0000	0.00000	0.0000	Sbio

Totals : 2517.77271

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

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

Sample Name: Reb D Standard

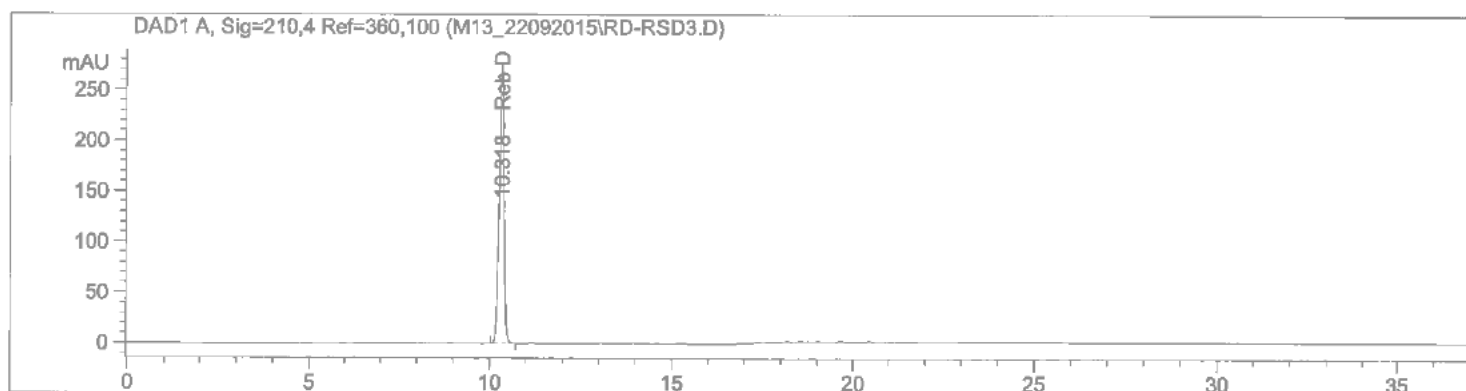
```

=====
Acq. Operator   : QC Chemist                      Seq. Line :    4
Acq. Instrument : HPLC 3                          Location  : Vial 3
Injection Date  : 9/23/2015 8:25:34 AM            Inj       :    3
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 3:22:46 PM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info      : Reb D Std, 1301.5102 mg/L, Relative Standard Deviation

```



```

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

```

```

Sorted By           :      Signal
Calib. Data Modified :      Thursday, October 01, 2015 3:22:46 PM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.970		0.0000	0.00000	0.0000	Reb E
2	9.710		0.0000	0.00000	0.0000	Reb O
3	10.318	BB	0.1436	2538.30591	100.0000	Reb D
4	11.232		0.0000	0.00000	0.0000	Reb N
5	13.138		0.0000	0.00000	0.0000	Reb M
6	20.400		0.0000	0.00000	0.0000	Reb A
7	20.600		0.0000	0.00000	0.0000	Stev
8	22.243		0.0000	0.00000	0.0000	Reb F
9	23.023		0.0000	0.00000	0.0000	Reb C
10	23.689		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb D Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.584		0.0000	0.00000	0.0000	Rubu
12	32.428		0.0000	0.00000	0.0000	Reb B
13	33.438		0.0000	0.00000	0.0000	Sbio

Totals : 2538.30591

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***

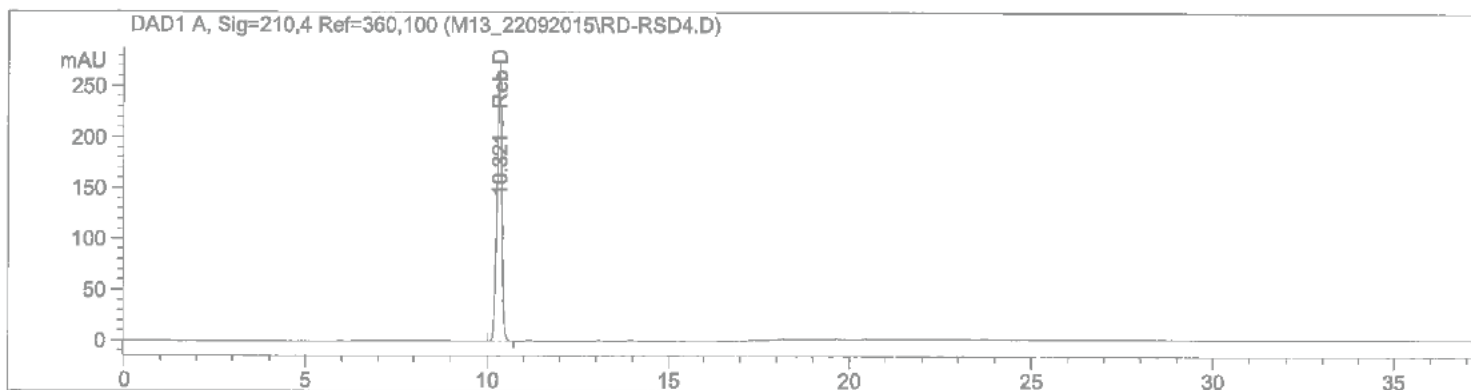


Sample Name: Reb D Standard

```
=====
Acq. Operator   : QC Chemist                      Seq. Line :    4
Acq. Instrument : HPLC 3                          Location  : Vial 3
Injection Date  : 9/23/2015 9:28:32 AM             Inj       :    4
                                                    Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 3:22:46 PM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info      : Reb D Std, 1301.5102 mg/L, Relative Standard Deviation
=====
```



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

```
Sorted By           : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:22:46 PM
Multiplier          : 1.0000
Dilution            : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.970		0.0000	0.00000	0.0000	Reb E
2	9.710		0.0000	0.00000	0.0000	Reb O
3	10.321	BB	0.1416	2524.35742	100.0000	Reb D
4	11.232		0.0000	0.00000	0.0000	Reb N
5	13.138		0.0000	0.00000	0.0000	Reb M
6	20.400		0.0000	0.00000	0.0000	Reb A
7	20.600		0.0000	0.00000	0.0000	Stev
8	22.243		0.0000	0.00000	0.0000	Reb F
9	23.023		0.0000	0.00000	0.0000	Reb C
10	23.689		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb D Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.584		0.0000	0.00000	0.0000	Rubu
12	32.428		0.0000	0.00000	0.0000	Reb B
13	33.438		0.0000	0.00000	0.0000	Sbio

Totals : 2524.35742

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



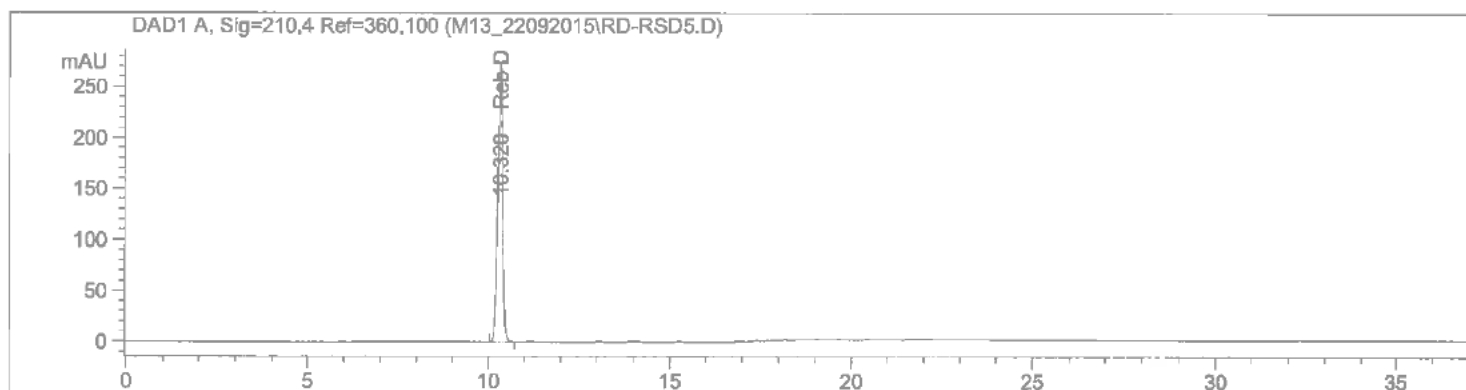
Sample Name: Reb D Standard

```

=====
Acq. Operator   : QC Chemist                      Seq. Line :    4
Acq. Instrument : HPLC 3                          Location  : Vial 3
Injection Date  : 9/23/2015 10:31:05 AM           Inj       :    5
                                                Inj Volume: 5 µl
Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 3:22:46 PM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info      : Reb D Std, 1301.5102 mg/L, Relative Standard Deviation

```



```

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

```

```

Sorted By           :      Signal
Calib. Data Modified :      Thursday, October 01, 2015 3:22:46 PM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.970		0.0000	0.00000	0.0000	Reb E
2	9.710		0.0000	0.00000	0.0000	Reb O
3	10.320	BB	0.1416	2515.63770	100.0000	Reb D
4	11.232		0.0000	0.00000	0.0000	Reb N
5	13.138		0.0000	0.00000	0.0000	Reb M
6	20.400		0.0000	0.00000	0.0000	Reb A
7	20.600		0.0000	0.00000	0.0000	Stev
8	22.243		0.0000	0.00000	0.0000	Reb F
9	23.023		0.0000	0.00000	0.0000	Reb C
10	23.689		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb D Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.584		0.0000	0.00000	0.0000	Rubu
12	32.428		0.0000	0.00000	0.0000	Reb B
13	33.438		0.0000	0.00000	0.0000	Sbio

Totals : 2515.63770

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Reb D Standard

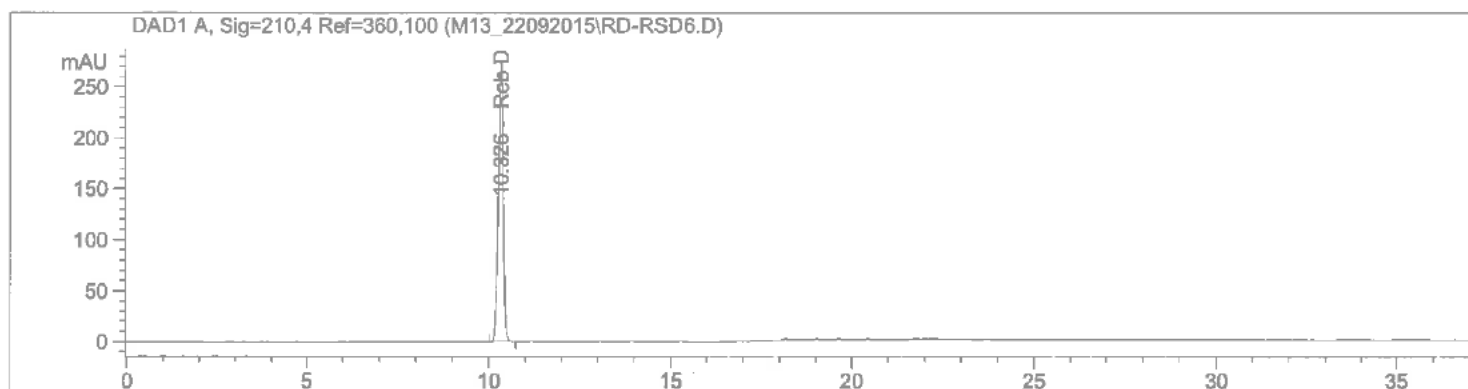
```

=====
Acq. Operator   : QC Chemist                      Seq. Line :    4
Acq. Instrument : HPLC 3                          Location  : Vial 3
Injection Date  : 9/23/2015 11:33:48 AM           Inj       :    6
                                                    Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 3:22:46 PM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info      : Reb D Std, 1301.5102 mg/L, Relative Standard Deviation
=====

```



```

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

```

```

Sorted By           :      Signal
Calib. Data Modified :      Thursday, October 01, 2015 3:22:46 PM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.970		0.0000	0.00000	0.0000	Reb E
2	9.710		0.0000	0.00000	0.0000	Reb O
3	10.326	BB	0.1416	2520.55591	100.0000	Reb D
4	11.232		0.0000	0.00000	0.0000	Reb N
5	13.138		0.0000	0.00000	0.0000	Reb M
6	20.400		0.0000	0.00000	0.0000	Reb A
7	20.600		0.0000	0.00000	0.0000	Stev
8	22.243		0.0000	0.00000	0.0000	Reb F
9	23.023		0.0000	0.00000	0.0000	Reb C
10	23.689		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb D Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.584		0.0000	0.00000	0.0000	Rubu
12	32.428		0.0000	0.00000	0.0000	Reb B
13	33.438		0.0000	0.00000	0.0000	Sbio


Totals : 2520.55591

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

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

 PureCircle	PureCircle Sdn Bhd	Report#: KNN02102015001	Issue date: 02/10/15
			Revision date: -----
HPLC Assay of A95 samples in 5 commercial lots			Revision No: 00

APPENDIX A3

Tailing Factor

Sample Name: Reb D Standard

Extended Performance Report

Instrument: HPLC 3

Module	Type	Firmware rev.	Serial number
1100/1200 Quaternary Pump	G1311A	A.06.10 [005]	DE62963952
1100/1200 Diode Array Detector	G1315B	A.06.10 [004]	DE63058785
1100/1200 Column Thermostat	G1316A	A.06.10 [004]	DE63067068
1100/1200 Thermostatted Autosampler	G1329A	A.06.10 [006]	DE64765541

Software Revision: Rev. B.03.02 [341] Copyright © Agilent Technologies

Column Description : Poroshell 120 SB-C18
Product# : 683975-902 Batch#: B10225
Serial# : USCEV01277
Diameter : 4.6 mm Length : 150.0 mm
Particle size : 2.7 µm Void volume : 60.0 %
Maximum Pressure : 600 bar Maximum pH : 9
Maximum Temperature: 60 °C
Comment : JECFA2010

Analysis method: C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M

Sample information for Location: Vial 3

Sample Name:	Reb D Standard	Multiplier:	1.00
Injection#:	6	Dilution:	1.00
Injection volume:	5 µl		

Acquisition information:

Operator: QC Chemist
Date/Time: 9/23/2015 11:33:48 AM
Data file name: C:\CHEM32\1\DATA\M13_22092015\RD-RSD6.D
Method file name: C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M

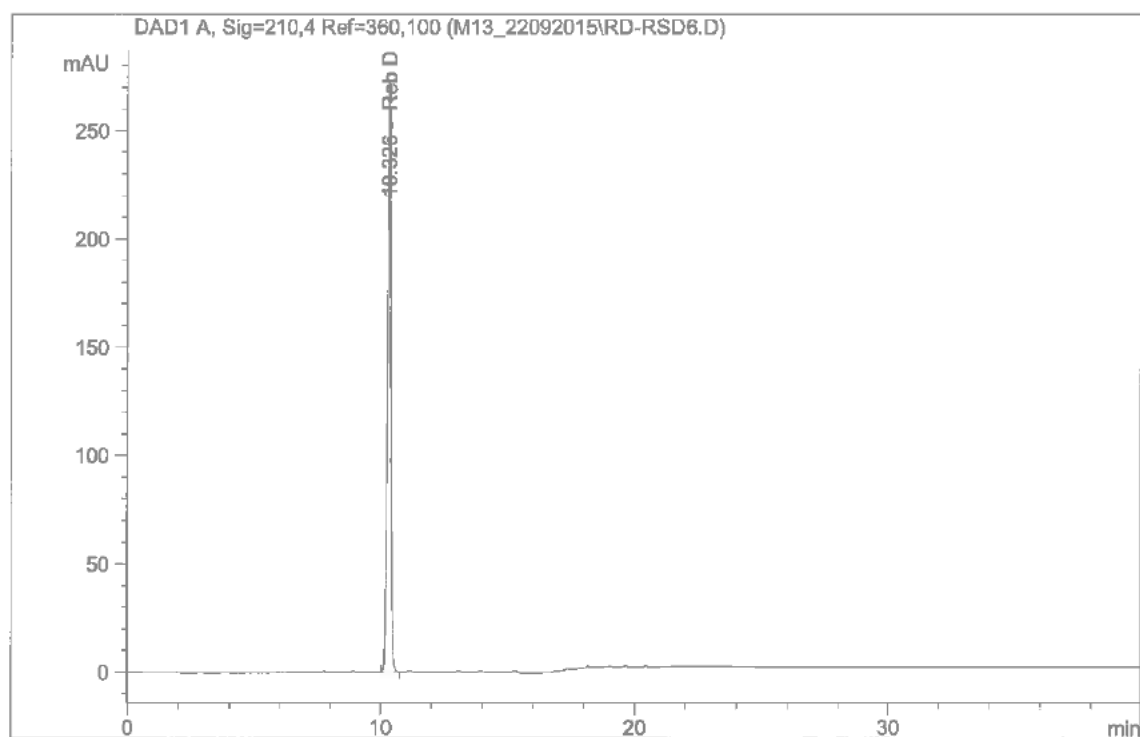
Flow:	0.500	ml/min	
Pressure at start:	132	bar	Pressure at end: 130 bar
Left Temp. at start:	40.0	°C	Left Temp. at end: 40.0 °C
Right Temp. at start:	40.0	°C	Right Temp. at end: 40.0 °C



Sample Name: Reb D Standard

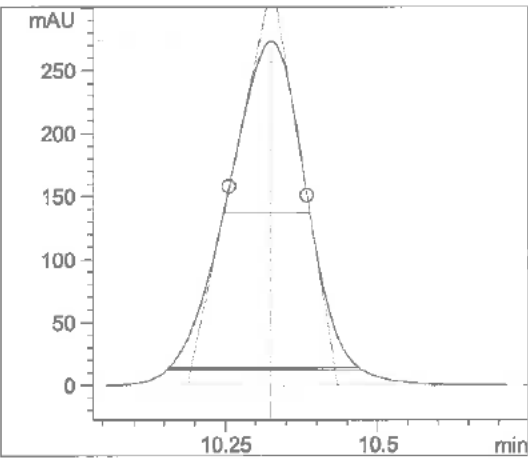
Solvents: PMP1 , Solvent 50:50 (buffer:ACN)
PMP1 , Solvent ACN
PMP1 , Solvent 68:32 (buffer:ACN)
PMP1 , Solvent 75:25 (buffer:ACN)

Signal description: DAD1 A, Sig=210,4 Ref=360,100



Compound# 3 : Reb D
Amount [ng/ul]: 1298.6220

Peak description [min]:
Signal: DAD1 A, Sig=210,4 Ref=360,100
RetTime: 10.326 k': 2.452
Height: 273.23 Area: 2520.6
Start: 10.040 End: 10.740
Skew: 0.123 Excess: 0.814
Width at half height: 0.140
5 sigma: 0.318
tangent: 0.245
tailing: 0.310
Symmetry: 1.199
USP Tailing: 0.928
Integration type: BB
Time increment [msec]: 400.0
Data points: 109



Statistical moments (BB peak detection):			Efficiency: Plates per ..	
M0: 2519.1			column	meter
M1: 10.317	Tangent method	28374		189160
M2: 0.004263	Halfwidth method	30136		200908
M3: 0.000034	5 sigma method	26303		175355
M4: 0.000069	Statistical	24969		166460

Relationship to preceeding peak:	Selectivity:	
Resolution Tangent method: -	5 sigma method	-
Halfwidth method -	Statistical method	-




Sample Name: Reb D Standard

#	Ret.Time [min]	Amount [ng/ul]	Name	Page #
1	10.326	1298.6220	Reb D	3
=====				
Total:		1298.6220		

*** End of Report ***




 PureCircle PureCircle Sdn Bhd	Report#: KNN02102015001	Issue date: 02/10/15
		Revision date: -----
HPLC Assay of A95 samples in 5 commercial lots		Revision No: 00

APPENDIX B


Standard Chromatograms

- B1 Rebaudioside D
- B2 Rebaudioside M
- B3 Rebaudioside A
- B4 Stevioside
- B5 M9 retention time marker
- B6 In-assay standard recovery check

 PureCircle	PureCircle Sdn Bhd	Report#: KNN02102015001	Issue date: 02/10/15
			Revision date: -----
HPLC Assay of A95 samples in 5 commercial lots			Revision No: 00

APPENDIX B1

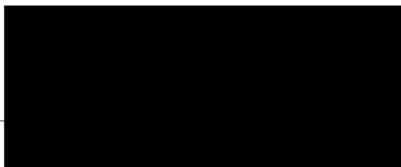
Rebaudioside D

 PureCircle Sdn Bhd	Document#: PCL-LAB-004	Issue date: 01/12/07
		Revision date: -----
HPLC reference standard preparation form		Revision No: 00

Standard Info	Rebaudioside D
Lot#	L03082015
Purity, %wt ("as is")	97.04
Purity, %wt ("dry basis")	N/A

Stock Solution				
Level#	Weight, mg	Volumetric flask volume, mL	Gross concentration, mg/L	Adjusted concentration, mg/L
1	17.45	25	698.0000	644.1110
2	21.42	25	856.8000	790.6508
3	26.24	25	1049.6000	968.5657
4	30.06	25	1202.4000	1109.5688
5	35.26	25	1410.4000	1301.5102
Remarks				

Done by:



 QC Chemist

22/09/15
 Date

Checked by:



 QA/QC Manager

22/9/15
 Date



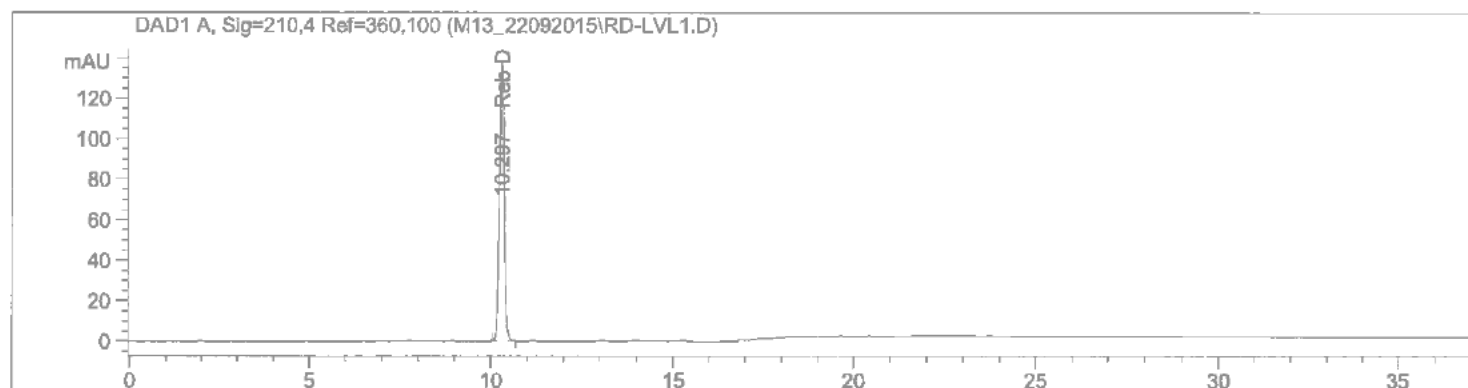
Sample Name: Reb D Standard

```

=====
Acq. Operator   : QC Chemist                      Seq. Line :    6
Acq. Instrument : HPLC 3                          Location  : Vial 4
Injection Date  : 9/23/2015 1:39:56 PM            Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 3:22:46 PM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : Reb D Std, 644.1110 mg/L
=====

```



```

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

```

```

Sorted By           :      Signal
Calib. Data Modified :      Thursday, October 01, 2015 3:22:46 PM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.970		0.0000	0.00000	0.0000	Reb E
2	9.710		0.0000	0.00000	0.0000	Reb O
3	10.297	BB	0.1425	1254.32336	100.0000	Reb D
4	11.232		0.0000	0.00000	0.0000	Reb N
5	13.138		0.0000	0.00000	0.0000	Reb M
6	20.400		0.0000	0.00000	0.0000	Reb A
7	20.600		0.0000	0.00000	0.0000	Stev
8	22.243		0.0000	0.00000	0.0000	Reb F
9	23.023		0.0000	0.00000	0.0000	Reb C
10	23.689		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb D Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.584		0.0000	0.00000	0.0000	Rubu
12	32.428		0.0000	0.00000	0.0000	Reb B
13	33.438		0.0000	0.00000	0.0000	Sbio

Totals : 1254.32336

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Reb D Standard

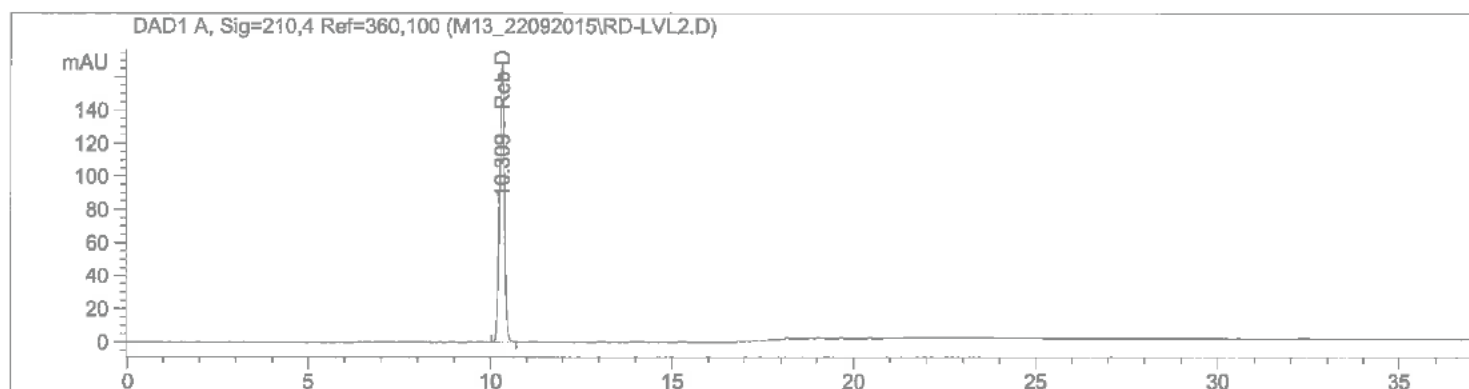
```

=====
Acq. Operator   : QC Chemist                      Seq. Line :    7
Acq. Instrument : HPLC 3                          Location  : Vial 5
Injection Date  : 9/23/2015 2:42:29 PM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 3:22:46 PM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info      : Reb D Std, 790.6508 mg/L
=====

```



```

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

```

```

Sorted By           : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:22:46 PM
Multiplier          : 1.0000
Dilution            : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.970		0.0000	0.00000	0.0000	Reb E
2	9.710		0.0000	0.00000	0.0000	Reb O
3	10.309	BB	0.1407	1536.87134	100.0000	Reb D
4	11.232		0.0000	0.00000	0.0000	Reb N
5	13.138		0.0000	0.00000	0.0000	Reb M
6	20.400		0.0000	0.00000	0.0000	Reb A
7	20.600		0.0000	0.00000	0.0000	Stev
8	22.243		0.0000	0.00000	0.0000	Reb F
9	23.023		0.0000	0.00000	0.0000	Reb C
10	23.689		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb D Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.584		0.0000	0.00000	0.0000	Rubu
12	32.428		0.0000	0.00000	0.0000	Reb B
13	33.438		0.0000	0.00000	0.0000	Sbio

Totals : 1536.87134

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

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

Sample Name: Reb D Standard

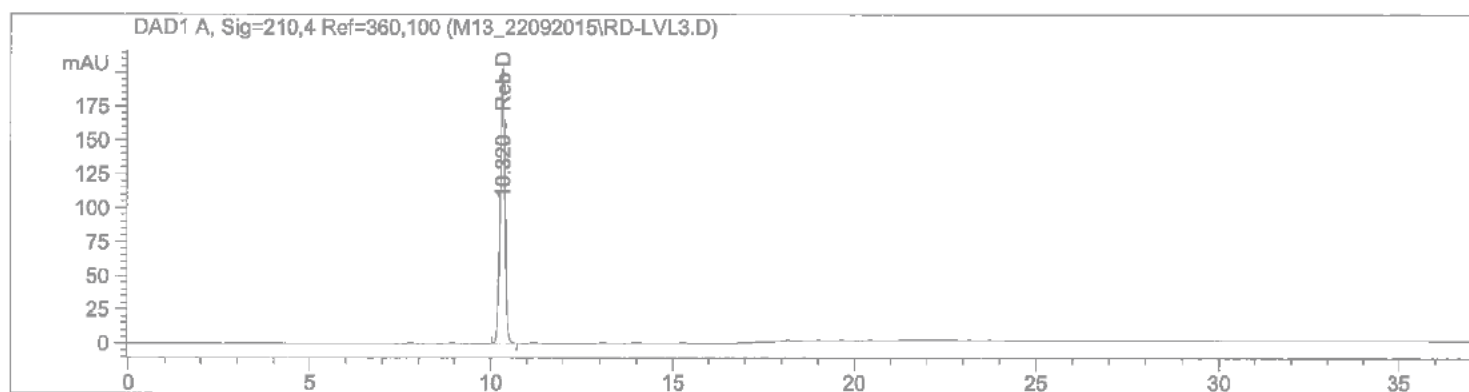
```

=====
Acq. Operator   : QC Chemist                      Seq. Line :    8
Acq. Instrument : HPLC 3                          Location  : Vial 6
Injection Date  : 9/23/2015 3:45:53 PM             Inj       :    1
                                                    Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 3:22:46 PM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : Reb D Std, 968.5657 mg/L
=====

```



```

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

```

```

Sorted By           :      Signal
Calib. Data Modified :      Thursday, October 01, 2015 3:22:46 PM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.970		0.0000	0.00000	0.0000	Reb E
2	9.710		0.0000	0.00000	0.0000	Reb O
3	10.320	BB	0.1430	1885.82812	100.0000	Reb D
4	11.232		0.0000	0.00000	0.0000	Reb N
5	13.138		0.0000	0.00000	0.0000	Reb M
6	20.400		0.0000	0.00000	0.0000	Reb A
7	20.600		0.0000	0.00000	0.0000	Stev
8	22.243		0.0000	0.00000	0.0000	Reb F
9	23.023		0.0000	0.00000	0.0000	Reb C
10	23.689		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb D Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.584		0.0000	0.00000	0.0000	Rubu
12	32.428		0.0000	0.00000	0.0000	Reb B
13	33.438		0.0000	0.00000	0.0000	Sbio

Totals : 1885.82812

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Reb D Standard

```

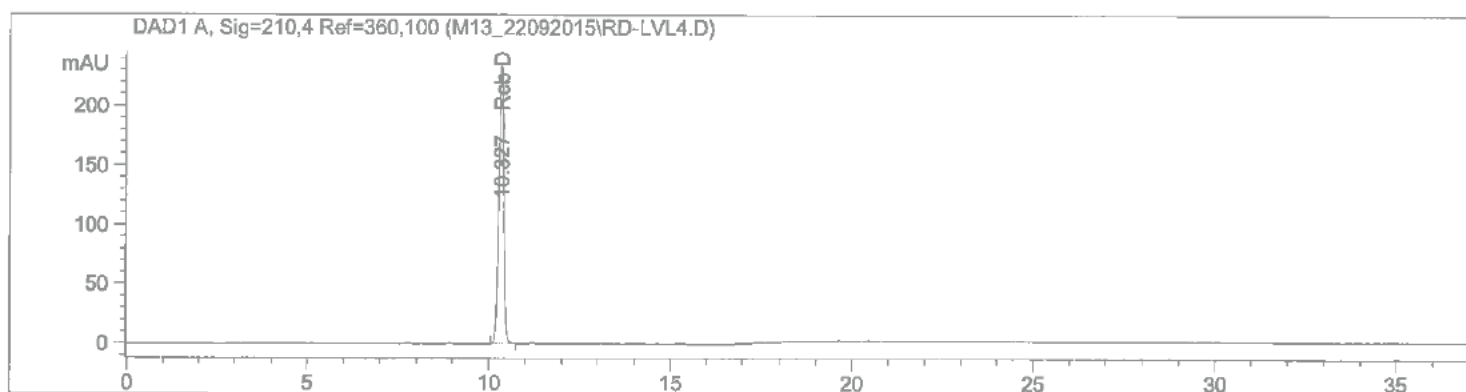
=====
Acq. Operator   : QC Chemist                      Seq. Line :    9
Acq. Instrument : HPLC 3                          Location  : Vial 7
Injection Date  : 9/23/2015 4:48:27 PM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 3:22:46 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : Reb D Std, 1109.5688 mg/L
=====

```



```

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

```

```

Sorted By           : Signal
Calib. Data Modified: Thursday, October 01, 2015 3:22:46 PM
Multiplier          : 1.0000
Dilution            : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.970		0.0000	0.00000	0.0000	Reb E
2	9.710		0.0000	0.00000	0.0000	Reb O
3	10.327	BB	0.1412	2154.63916	100.0000	Reb D
4	11.232		0.0000	0.00000	0.0000	Reb N
5	13.138		0.0000	0.00000	0.0000	Reb M
6	20.400		0.0000	0.00000	0.0000	Reb A
7	20.600		0.0000	0.00000	0.0000	Stev
8	22.243		0.0000	0.00000	0.0000	Reb F
9	23.023		0.0000	0.00000	0.0000	Reb C
10	23.689		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb D Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.584		0.0000	0.00000	0.0000	Rubu
12	32.428		0.0000	0.00000	0.0000	Reb B
13	33.438		0.0000	0.00000	0.0000	Sbio

Totals : 2154.63916

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Reb D Standard

```

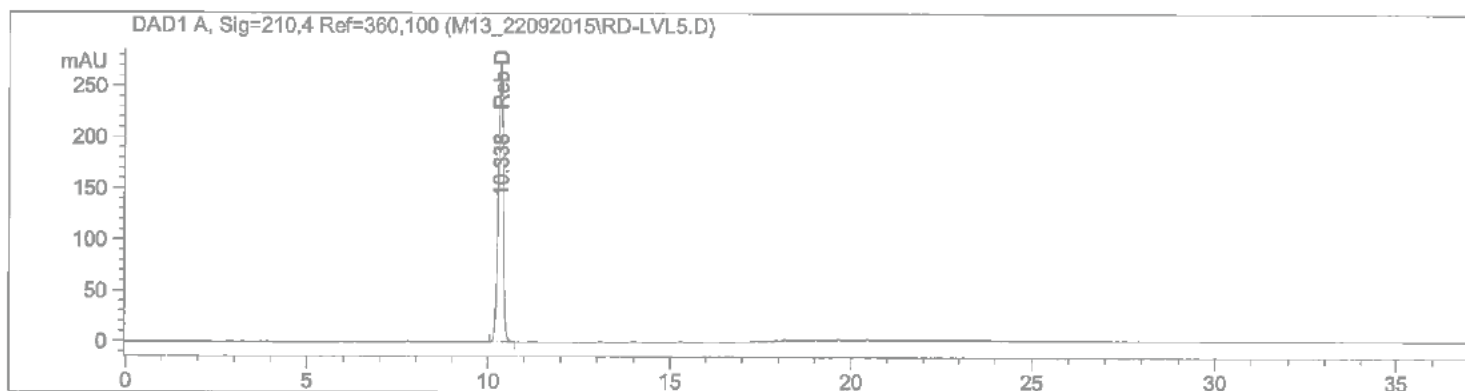
=====
Acq. Operator   : QC Chemist                      Seq. Line :   10
Acq. Instrument : HPLC 3                          Location  : Vial 8
Injection Date  : 9/23/2015 5:51:42 PM             Inj       :    1
                                                    Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 3:22:46 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : Reb D Std, 1301.5102 mg/L
=====

```



```

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

```

```

Sorted By           :      Signal
Calib. Data Modified :      Thursday, October 01, 2015 3:22:46 PM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.970		0.0000	0.00000	0.0000	Reb E
2	9.710		0.0000	0.00000	0.0000	Reb O
3	10.338	BB	0.1417	2517.48047	100.0000	Reb D
4	11.232		0.0000	0.00000	0.0000	Reb N
5	13.138		0.0000	0.00000	0.0000	Reb M
6	20.400		0.0000	0.00000	0.0000	Reb A
7	20.600		0.0000	0.00000	0.0000	Stev
8	22.243		0.0000	0.00000	0.0000	Reb F
9	23.023		0.0000	0.00000	0.0000	Reb C
10	23.689		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb D Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.584		0.0000	0.00000	0.0000	Rubu
12	32.428		0.0000	0.00000	0.0000	Reb B
13	33.438		0.0000	0.00000	0.0000	Sbio

Totals : 2517.48047

2 Warnings or Errors :

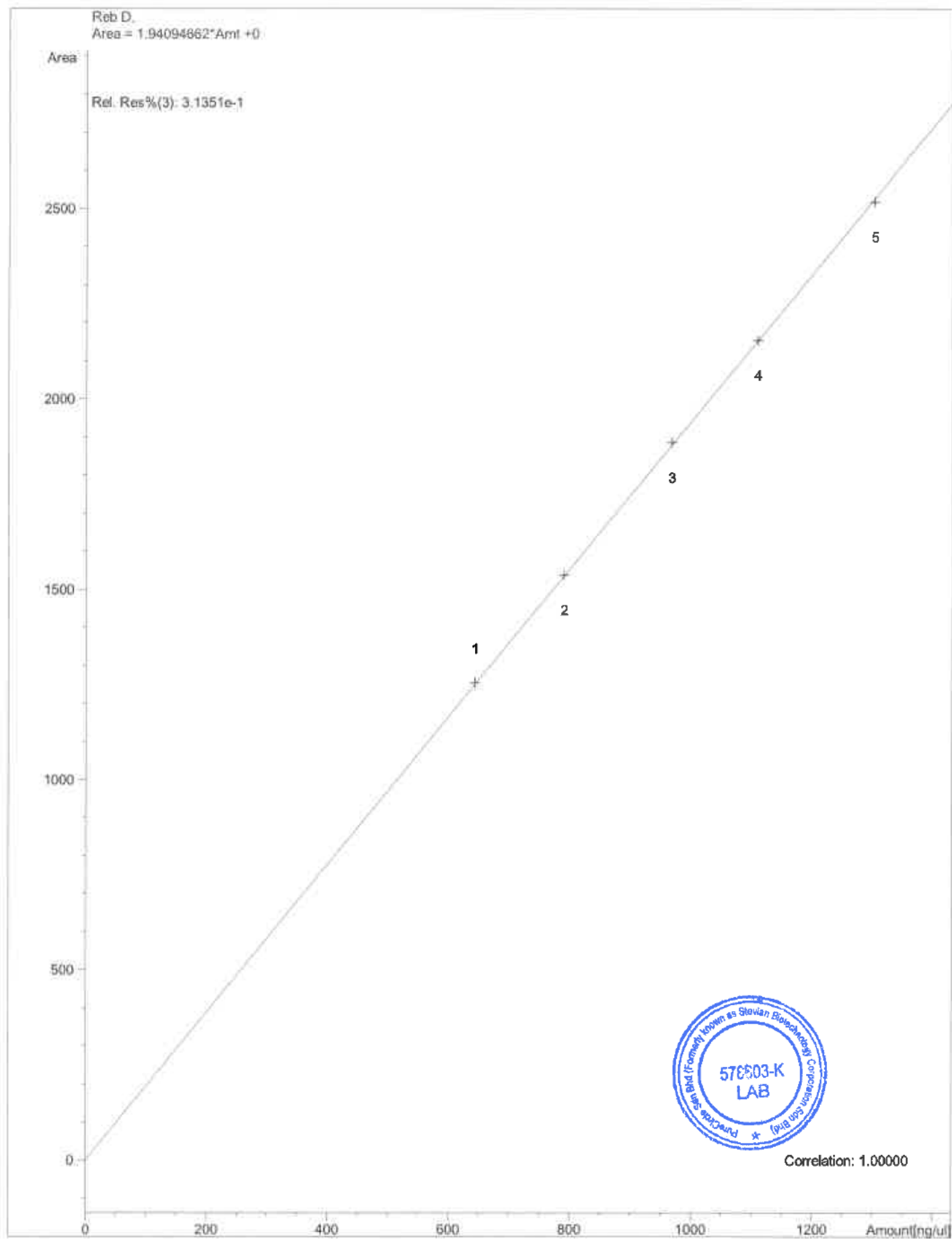
Warning : Calibration warnings (see calibration table listing)


Warning : Calibrated compound(s) not found

*** End of Report ***




Calibration Curve



 PureCircle	PureCircle Sdn Bhd	Report#: KNN02102015001	Issue date: 02/10/15
			Revision date: _____
HPLC Assay of A95 samples in 5 commercial lots			Revision No: 00

APPENDIX B2

Rebaudioside M

 PureCircle Sdn Bhd	Document#:	Issue date: 01/12/07
	PCL-LAB-004	Revision date: -----
	HPLC reference standard preparation form	
		Revision No: 00

Standard Info	Rebaudioside M
Lot#	L11112013
Purity, %wt ("as is")	97.05
Purity, %wt ("dry basis")	N/A

Stock Solution				
Level#	Weight, mg	Volumetric flask volume, mL	Gross concentration, mg/L	Adjusted concentration, mg/L
1	7.41	25	296.4000	245.8172
2	10.35	25	414.0000	343.3479
3	14.88	25	595.2000	493.6248
4	17.09	25	683.6000	566.9387
5	20.60	25	824.0000	683.3784
Remarks				

Done by:

 QC Chemist

22/09/15

 Date

Checked by:

 QA/QC Manager

22/09/15

 Date



Sample Name: Reb M Standard

```

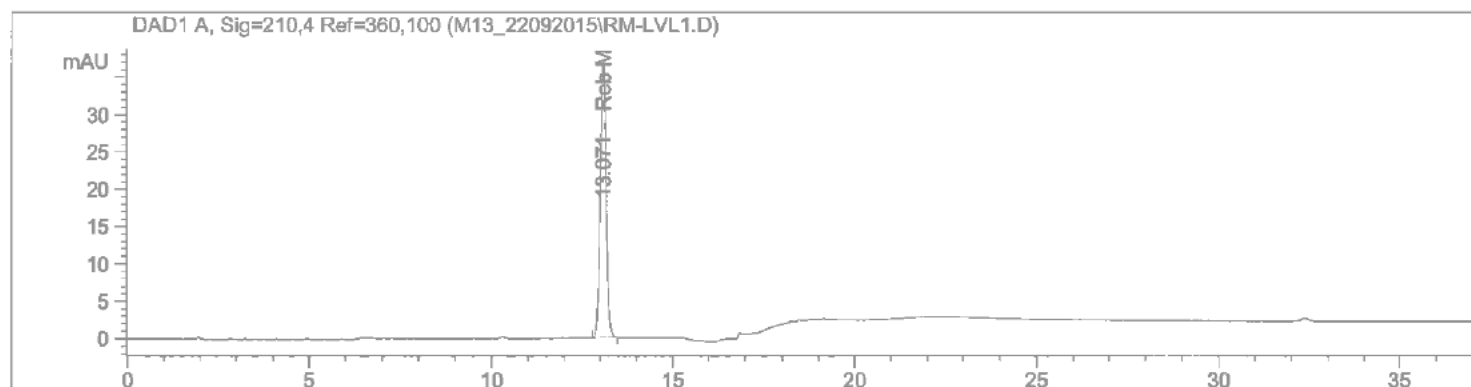
=====
Acq. Operator   : QC Chemist                      Seq. Line :   11
Acq. Instrument : HPLC 3                          Location  : Vial 9
Injection Date  : 9/23/2015 6:54:46 PM            Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 9:29:58 AM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : Reb M Std, 245.8172 mg/L
=====

```



```

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

```

```

Sorted By           :      Signal
Calib. Data Modified :      Monday, September 28, 2015 9:22:25 AM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.920		0.0000	0.00000	0.0000	Reb E
2	9.643		0.0000	0.00000	0.0000	Reb O
3	10.288		0.0000	0.00000	0.0000	Reb D
4	11.160		0.0000	0.00000	0.0000	Reb N
5	13.071	BB	0.1719	409.89001	100.0000	Reb M
6	20.477		0.0000	0.00000	0.0000	Reb A
7	20.630		0.0000	0.00000	0.0000	Stev
8	22.191		0.0000	0.00000	0.0000	Reb F
9	22.970		0.0000	0.00000	0.0000	Reb C
10	23.633		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb M Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.501		0.0000	0.00000	0.0000	Rubu
12	32.271		0.0000	0.00000	0.0000	Reb B
13	33.279		0.0000	0.00000	0.0000	Sbio

Totals : 409.89001

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

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

Sample Name: Reb M Standard

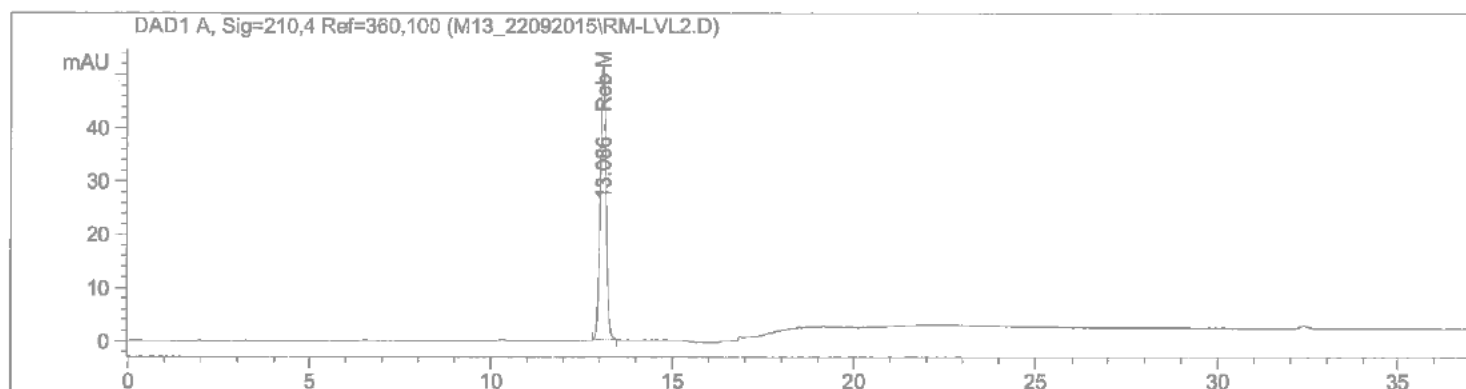
```

=====
Acq. Operator   : QC Chemist                      Seq. Line :   12
Acq. Instrument : HPLC 3                          Location  : Vial 10
Injection Date  : 9/23/2015 7:57:30 PM            Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 9:29:58 AM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : Reb M Std, 343.3479 mg/L
=====

```



```

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

```

```

Sorted By           :      Signal
Calib. Data Modified :      Monday, September 28, 2015 9:22:25 AM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.920		0.0000	0.00000	0.0000	Reb E
2	9.643		0.0000	0.00000	0.0000	Reb O
3	10.288		0.0000	0.00000	0.0000	Reb D
4	11.160		0.0000	0.00000	0.0000	Reb N
5	13.086	BB	0.1721	578.59448	100.0000	Reb M
6	20.477		0.0000	0.00000	0.0000	Reb A
7	20.630		0.0000	0.00000	0.0000	Stev
8	22.191		0.0000	0.00000	0.0000	Reb F
9	22.970		0.0000	0.00000	0.0000	Reb C
10	23.633		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb M Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.501		0.0000	0.00000	0.0000	Rubu
12	32.271		0.0000	0.00000	0.0000	Reb B
13	33.279		0.0000	0.00000	0.0000	Sbio

Totals : 578.59448

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

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

Sample Name: Reb M Standard

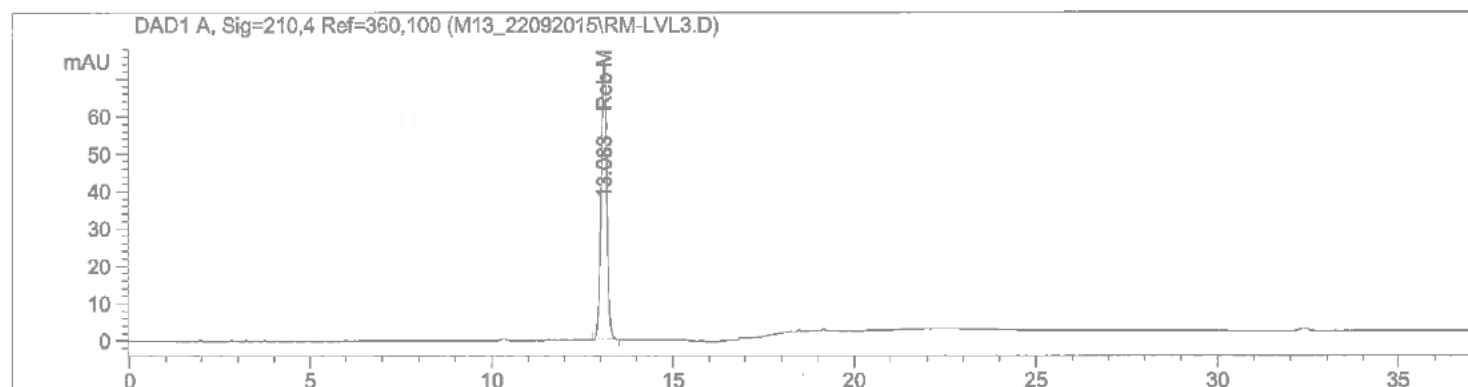
```

=====
Acq. Operator   : QC Chemist                      Seq. Line :   13
Acq. Instrument : HPLC 3                          Location  : Vial 11
Injection Date  : 9/23/2015 9:00:33 PM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 9:29:58 AM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info      : Reb M Std, 493.6248 mg/L
=====

```



```

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

```

```

Sorted By           :      Signal
Calib. Data Modified :      Monday, September 28, 2015 9:22:25 AM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.920		0.0000	0.00000	0.0000	Reb E
2	9.643		0.0000	0.00000	0.0000	Reb O
3	10.288		0.0000	0.00000	0.0000	Reb D
4	11.160		0.0000	0.00000	0.0000	Reb N
5	13.083	BB	0.1747	831.13428	100.0000	Reb M
6	20.477		0.0000	0.00000	0.0000	Reb A
7	20.630		0.0000	0.00000	0.0000	Stev
8	22.191		0.0000	0.00000	0.0000	Reb F
9	22.970		0.0000	0.00000	0.0000	Reb C
10	23.633		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb M Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.501		0.0000	0.00000	0.0000	Rubu
12	32.271		0.0000	0.00000	0.0000	Reb B
13	33.279		0.0000	0.00000	0.0000	Sbio

Totals : 831.13428

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***

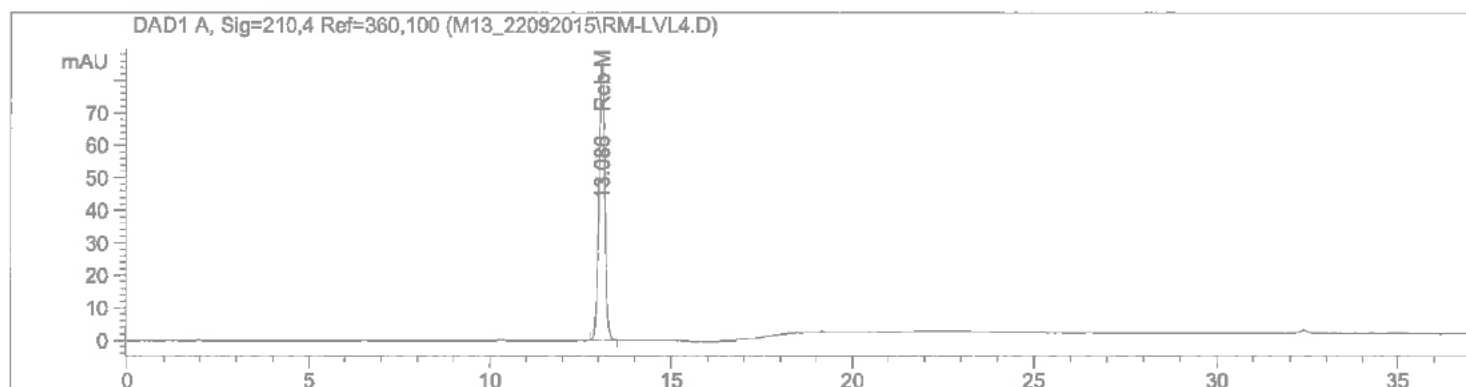


Sample Name: Reb M Standard

```
=====
Acq. Operator   : QC Chemist                      Seq. Line :   14
Acq. Instrument : HPLC 3                          Location  : Vial 12
Injection Date  : 9/23/2015 10:03:30 PM           Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 9:29:58 AM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info      : Reb M Std, 566.9387 mg/L
=====
```



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

```
Sorted By           :      Signal
Calib. Data Modified :      Monday, September 28, 2015 9:22:25 AM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.920		0.0000	0.00000	0.0000	Reb E
2	9.643		0.0000	0.00000	0.0000	Reb O
3	10.288		0.0000	0.00000	0.0000	Reb D
4	11.160		0.0000	0.00000	0.0000	Reb N
5	13.086	BB	0.1728	957.10413	100.0000	Reb M
6	20.477		0.0000	0.00000	0.0000	Reb A
7	20.630		0.0000	0.00000	0.0000	Stev
8	22.191		0.0000	0.00000	0.0000	Reb F
9	22.970		0.0000	0.00000	0.0000	Reb C
10	23.633		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb M Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.501		0.0000	0.00000	0.0000	Rubu
12	32.271		0.0000	0.00000	0.0000	Reb B
13	33.279		0.0000	0.00000	0.0000	Sbio

Totals : 957.10413

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

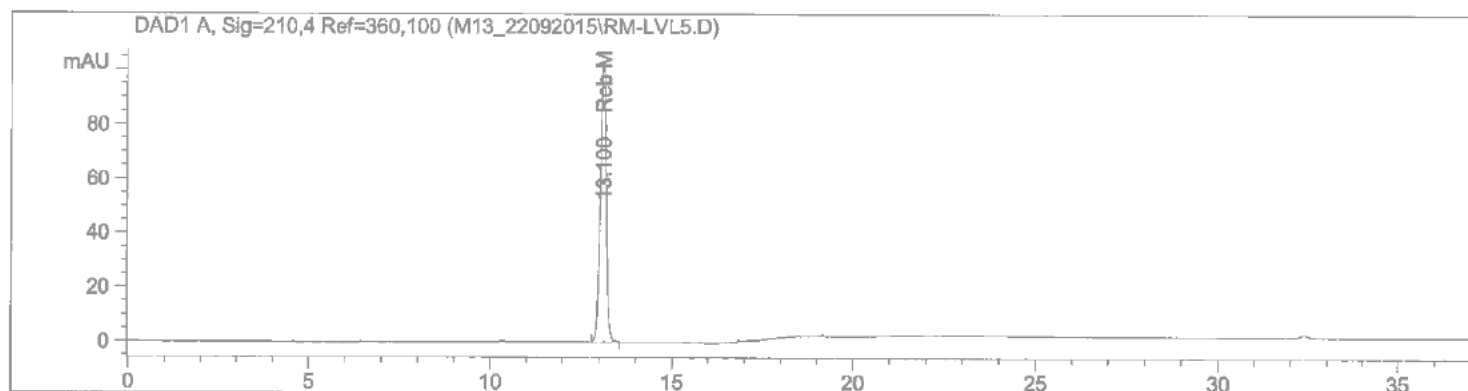
=====
*** End of Report ***

Sample Name: Reb M Standard

```
=====
Acq. Operator   : QC Chemist                      Seq. Line :   15
Acq. Instrument : HPLC 3                          Location  : Vial 13
Injection Date  : 9/23/2015 11:06:02 PM           Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 9:29:58 AM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : Reb M Std, 683.3784 mg/L
=====
```



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

```
Sorted By      : Signal
Calib. Data Modified : Monday, September 28, 2015 9:22:25 AM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.920		0.0000	0.00000	0.0000	Reb E
2	9.643		0.0000	0.00000	0.0000	Reb O
3	10.288		0.0000	0.00000	0.0000	Reb D
4	11.160		0.0000	0.00000	0.0000	Reb N
5	13.100	BB	0.1731	1147.17065	100.0000	Reb M
6	20.477		0.0000	0.00000	0.0000	Reb A
7	20.630		0.0000	0.00000	0.0000	Stev
8	22.191		0.0000	0.00000	0.0000	Reb F
9	22.970		0.0000	0.00000	0.0000	Reb C
10	23.633		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb M Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.501		0.0000	0.00000	0.0000	Rubu
12	32.271		0.0000	0.00000	0.0000	Reb B
13	33.279		0.0000	0.00000	0.0000	Sbio

Totals : 1147.17065

2 Warnings or Errors :

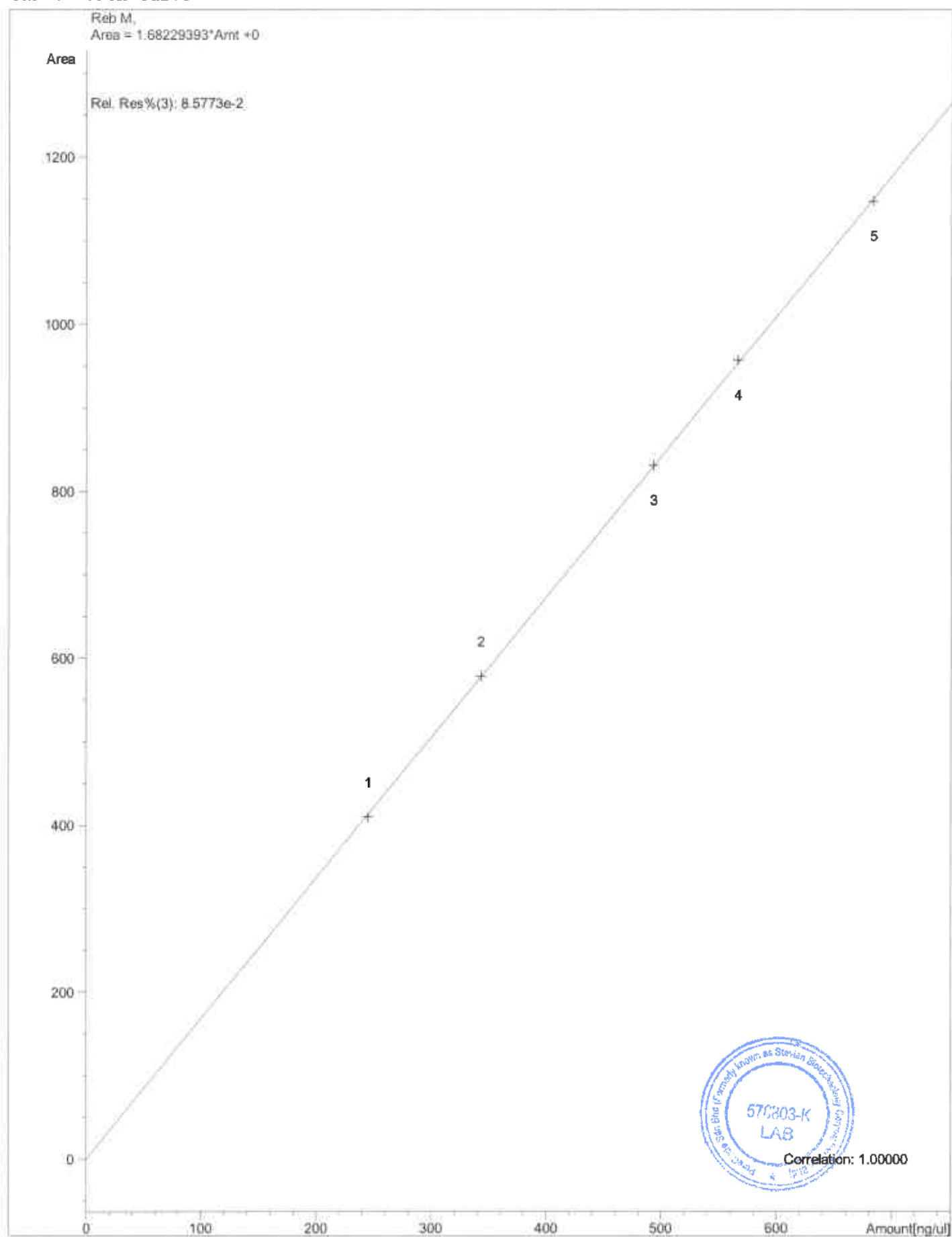
Warning : Calibration warnings (see calibration table listing)


Warning : Calibrated compound(s) not found

*** End of Report ***




Calibration Curve



 PureCircle	PureCircle Sdn Bhd	Report#: KNN02102015001	Issue date: 02/10/15
			Revision date: -----
HPLC Assay of A95 samples in 5 commercial lots			Revision No: 00

APPENDIX B3

Rebaudioside A

 PureCircle Sdn Bhd	Document#: PCL-LAB-004	Issue date: 01/12/07
		Revision date: -----
HPLC reference standard preparation form		Revision No: 00

Standard Info	Rebaudioside A (USP)
Lot#	FOI077
Purity, %wt ("as is")	96.9
Purity, %wt ("dry basis")	N/A

Stock Solution	
Weight, mg	5.58
Volumetric flask volume, mL	50
Gross concentration, mg/L	111.6000
Water, %wt (Karl Fischer)	7.3046
Residual Solvent, %wt	—
Adjusted concentration, mg/L	100.2412
Remarks	

Dilution Levels		
Level#	Dilution	Concentration, mg/L
1	X 50.0	2.0048
2	X 10.0	10.0241
3	X 5.0	20.0482
4	X 2.5	40.0965
5	X 1	100.2412
Remarks		

Done by:

_____  QC Chemist

22/09/15

Date

Checked by:

_____  QA/QC Manager

22/09/15

Date



Sample Name: Reb A Standard

```

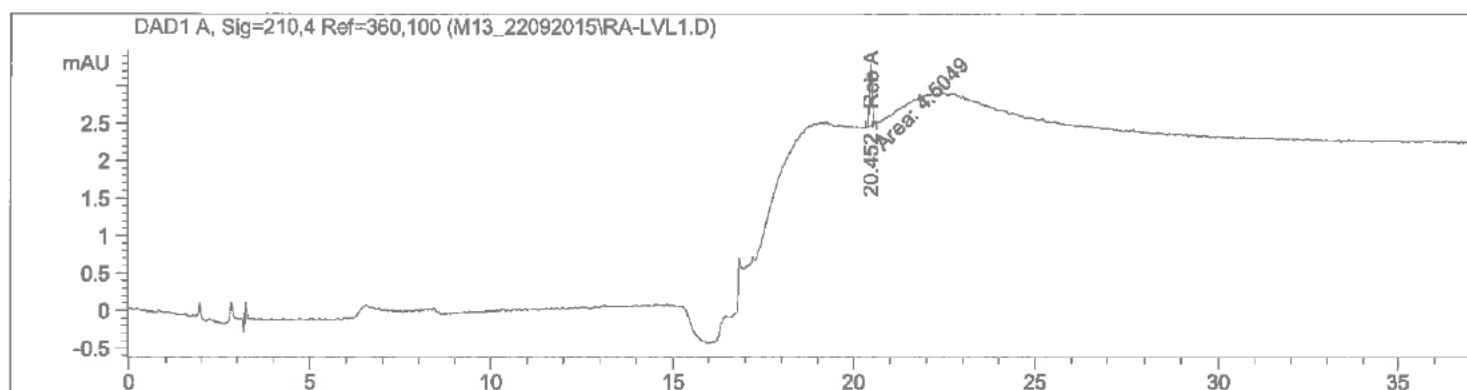
=====
Acq. Operator   : QC Chemist                      Seq. Line :   16
Acq. Instrument : HPLC 3                          Location  : Vial 14
Injection Date  : 9/24/2015 12:09:10 AM           Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 9:22:25 AM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : Reb A Std, 2.0048 mg/L
=====

```



```

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

```

```

Sorted By           :      Signal
Calib. Data Modified :      Monday, September 28, 2015 9:22:25 AM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.920		0.0000	0.00000	0.0000	Reb E
2	9.643		0.0000	0.00000	0.0000	Reb O
3	10.288		0.0000	0.00000	0.0000	Reb D
4	11.160		0.0000	0.00000	0.0000	Reb N
5	13.020		0.0000	0.00000	0.0000	Reb M
6	20.452	MM	0.0913	4.50490	100.0000	Reb A
7	20.630		0.0000	0.00000	0.0000	Stev
8	22.191		0.0000	0.00000	0.0000	Reb F
9	22.970		0.0000	0.00000	0.0000	Reb C
10	23.633		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb A Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.501		0.0000	0.00000	0.0000	Rubu
12	32.271		0.0000	0.00000	0.0000	Reb B
13	33.279		0.0000	0.00000	0.0000	Sbio

Totals : 4.50490

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***

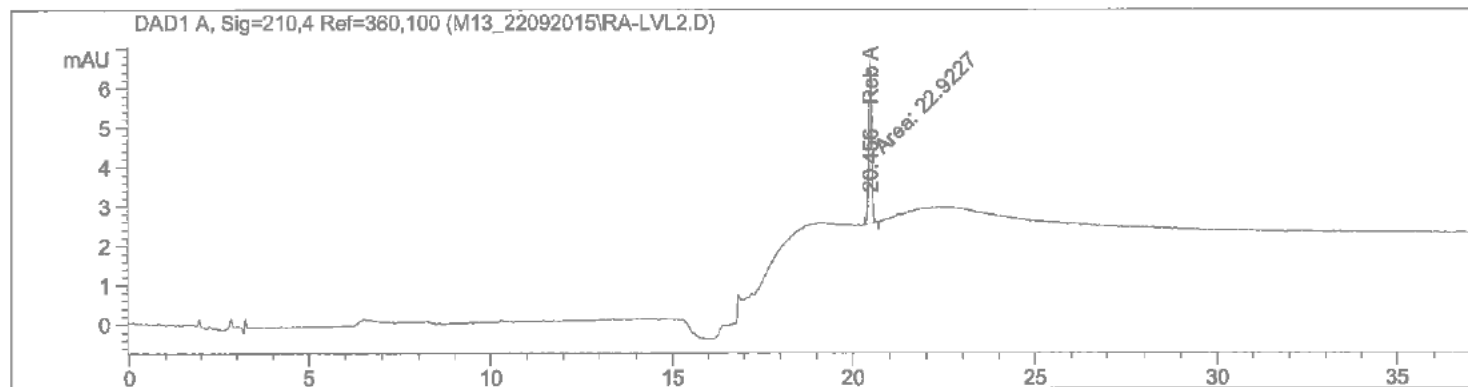



```
=====
Acq. Operator   : QC Chemist                      Seq. Line :   17
Acq. Instrument : HPLC 3                          Location  : Vial 15
Injection Date  : 9/24/2015 1:12:27 AM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 9:22:25 AM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : Reb A Std, 10.0241 mg/L
=====
```



Area Percent Report

```
=====
Sorted By      : Signal
Calib. Data Modified : Monday, September 28, 2015 9:22:25 AM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.920		0.0000	0.00000	0.0000	Reb E
2	9.643		0.0000	0.00000	0.0000	Reb O
3	10.288		0.0000	0.00000	0.0000	Reb D
4	11.160		0.0000	0.00000	0.0000	Reb N
5	13.020		0.0000	0.00000	0.0000	Reb M
6	20.456	MM	0.0915	22.92266	100.0000	Reb A
7	20.630		0.0000	0.00000	0.0000	Stev
8	22.191		0.0000	0.00000	0.0000	Reb F
9	22.970		0.0000	0.00000	0.0000	Reb C
10	23.633		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb A Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.501		0.0000	0.00000	0.0000	Rubu
12	32.271		0.0000	0.00000	0.0000	Reb B
13	33.279		0.0000	0.00000	0.0000	Sbio

Totals : 22.92266

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

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



Sample Name: Reb A Standard

```

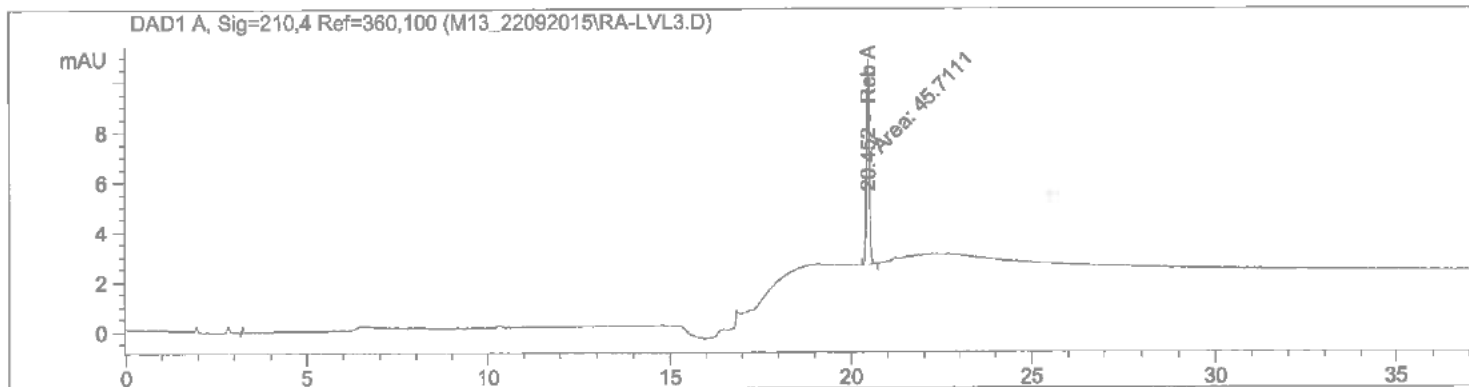
=====
Acq. Operator   : QC Chemist                      Seq. Line :   18
Acq. Instrument : HPLC 3                          Location  : Vial 16
Injection Date  : 9/24/2015 2:15:45 AM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 9:22:25 AM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info      : Reb A Std, 20.0482 mg/L
=====

```



```

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

```

```

Sorted By           : Signal
Calib. Data Modified : Monday, September 28, 2015 9:22:25 AM
Multiplier          : 1.0000
Dilution            : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.920		0.0000	0.00000	0.0000	Reb E
2	9.643		0.0000	0.00000	0.0000	Reb O
3	10.288		0.0000	0.00000	0.0000	Reb D
4	11.160		0.0000	0.00000	0.0000	Reb N
5	13.020		0.0000	0.00000	0.0000	Reb M
6	20.452 MM		0.0918	45.71113	100.0000	Reb A
7	20.630		0.0000	0.00000	0.0000	Stev
8	22.191		0.0000	0.00000	0.0000	Reb F
9	22.970		0.0000	0.00000	0.0000	Reb C
10	23.633		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb A Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.501		0.0000	0.00000	0.0000	Rubu
12	32.271		0.0000	0.00000	0.0000	Reb B
13	33.279		0.0000	0.00000	0.0000	Sbio

Totals : 45.71113

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

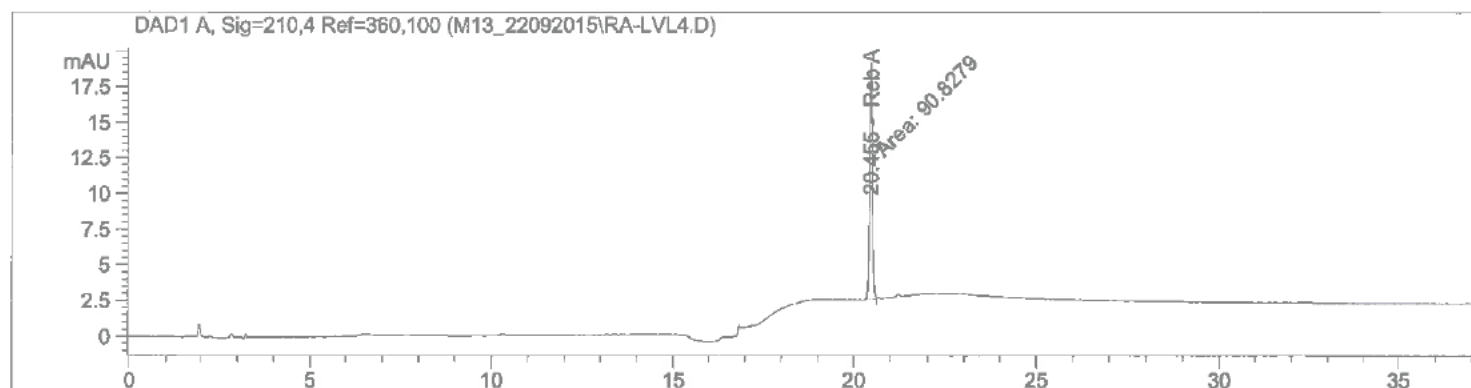
Warning : Calibrated compound(s) not found

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

Sample Name: Reb A Standard

```
=====
Acq. Operator   : QC Chemist                      Seq. Line :   19
Acq. Instrument : HPLC 3                          Location  : Vial 17
Injection Date  : 9/24/2015 3:19:13 AM             Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 9:22:25 AM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : Reb A Std, 40.0965 mg/L
=====
```



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

```
Sorted By           : Signal
Calib. Data Modified : Monday, September 28, 2015 9:22:25 AM
Multiplier          : 1.0000
Dilution            : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.920		0.0000	0.00000	0.0000	Reb E
2	9.643		0.0000	0.00000	0.0000	Reb O
3	10.288		0.0000	0.00000	0.0000	Reb D
4	11.160		0.0000	0.00000	0.0000	Reb N
5	13.020		0.0000	0.00000	0.0000	Reb M
6	20.455	MF	0.0910	90.82787	100.0000	Reb A
7	20.630		0.0000	0.00000	0.0000	Stev
8	22.191		0.0000	0.00000	0.0000	Reb F
9	22.970		0.0000	0.00000	0.0000	Reb C
10	23.633		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb A Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.501		0.0000	0.00000	0.0000	Rubu
12	32.271		0.0000	0.00000	0.0000	Reb B
13	33.279		0.0000	0.00000	0.0000	Sbio

Totals : 90.82787

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Reb A Standard

```

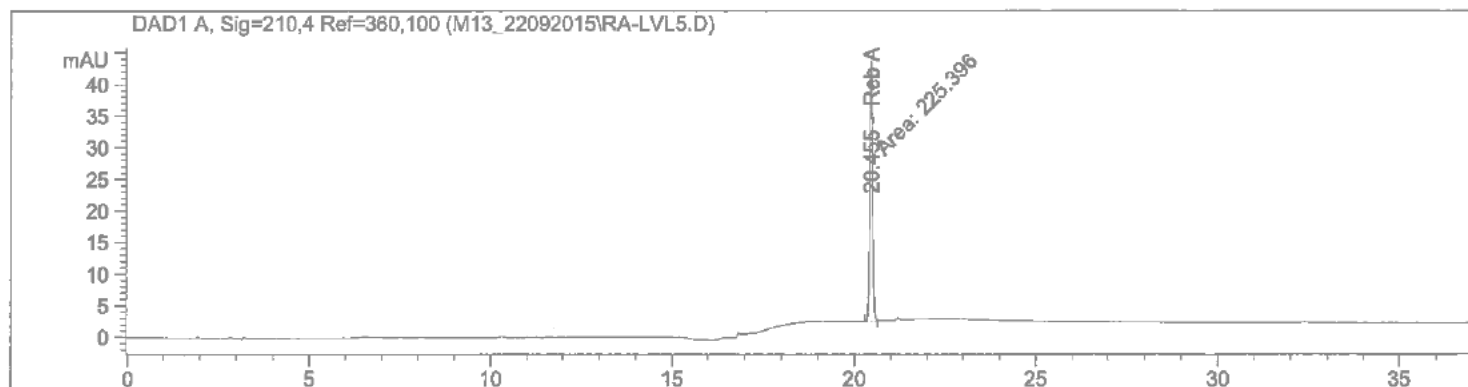
=====
Acq. Operator   : QC Chemist                      Seq. Line :   20
Acq. Instrument : HPLC 3                          Location  : Vial 18
Injection Date  : 9/24/2015 4:22:29 AM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 9:22:25 AM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info      : Reb A Std, 100.2412 mg/L
=====

```



```

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

```

```

Sorted By           :      Signal
Calib. Data Modified :      Monday, September 28, 2015 9:22:25 AM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.920		0.0000	0.00000	0.0000	Reb E
2	9.643		0.0000	0.00000	0.0000	Reb O
3	10.288		0.0000	0.00000	0.0000	Reb D
4	11.160		0.0000	0.00000	0.0000	Reb N
5	13.020		0.0000	0.00000	0.0000	Reb M
6	20.455	MF	0.0911	225.39577	100.0000	Reb A
7	20.630		0.0000	0.00000	0.0000	Stev
8	22.191		0.0000	0.00000	0.0000	Reb F
9	22.970		0.0000	0.00000	0.0000	Reb C
10	23.633		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Reb A Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.501		0.0000	0.00000	0.0000	Rubu
12	32.271		0.0000	0.00000	0.0000	Reb B
13	33.279		0.0000	0.00000	0.0000	Sbio

Totals : 225.39577

2 Warnings or Errors :

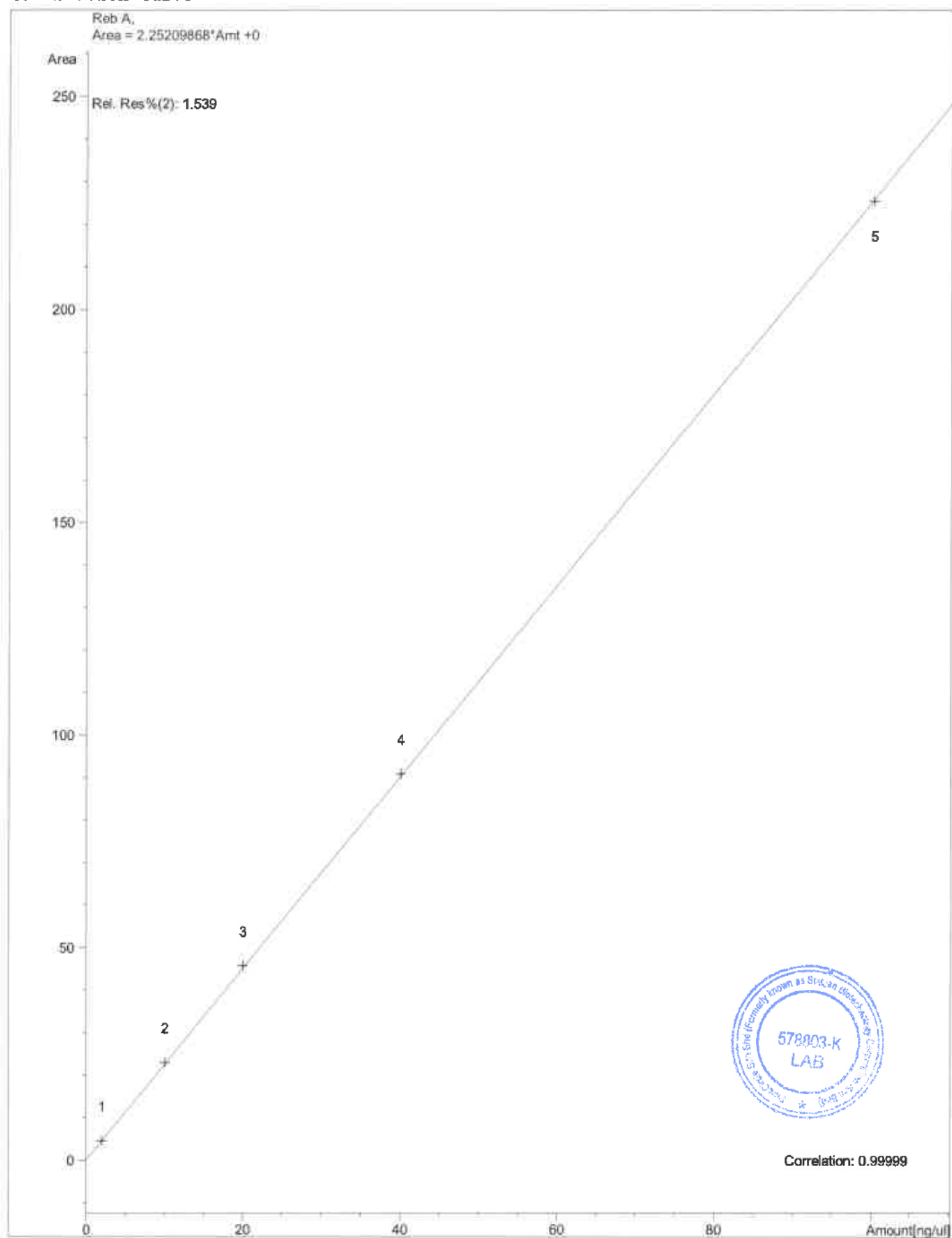
Warning : Calibration warnings (see calibration table listing)


Warning : Calibrated compound(s) not found

*** End of Report ***




Calibration Curve



 PureCircle PureCircle Sdn Bhd	Report#: KNN02102015001	Issue date: 02/10/15
		Revision date: -----
HPLC Assay of A95 samples in 5 commercial lots		Revision No: 00

APPENDIX B4

Stevioside

 PureCircle Sdn Bhd	Document#: PCL-LAB-004	Issue date: 01/12/07
		Revision date: -----
HPLC reference standard preparation form		Revision No: 00

Standard Info	Stevioside (USP)
Lot#	F0I080
Purity, %wt ("as is")	97.0
Purity, %wt ("dry basis")	N/A

Stock Solution	
Weight, mg	5.55
Volumetric flask volume, mL	50
Gross concentration, mg/L	111.0000
Water, %wt (Karl Fischer)	11.2316
Residual Solvent, %wt	—
Adjusted concentration, mg/L	95.5769
Remarks	

Dilution Levels		
Level#	Dilution	Concentration, mg/L
1	X 50.0	1.9115
2	X 10.0	9.5577
3	X 5.0	19.1154
4	X 2.5	38.2308
5 (Stock)	X 1	95.5769
LOD	X200	0.4779
Remarks		

Done by:

_____  QC Chemist

22/09/15
Date

Checked by:

_____  QA/QC Manager

22/09/15
Date

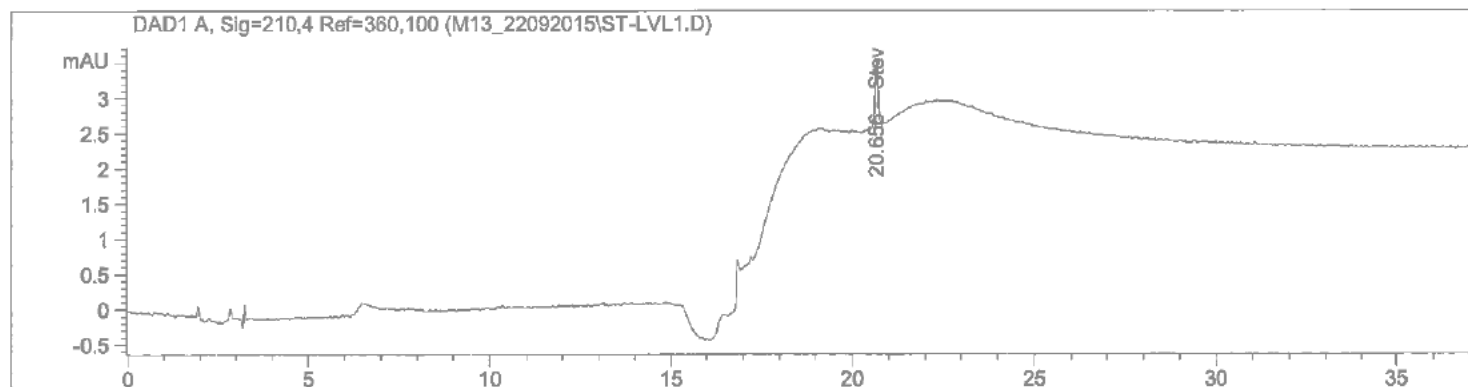


```
=====
Acq. Operator   : QC Chemist                      Seq. Line :   21
Acq. Instrument : HPLC 3                          Location  : Vial 19
Injection Date  : 9/24/2015 5:26:24 AM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 9:29:58 AM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : Stev Std, 1.9115 mg/L
=====
```



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

```
Sorted By      : Signal
Calib. Data Modified : Monday, September 28, 2015 9:22:25 AM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.920		0.0000	0.00000	0.0000	Reb E
2	9.643		0.0000	0.00000	0.0000	Reb O
3	10.288		0.0000	0.00000	0.0000	Reb D
4	11.160		0.0000	0.00000	0.0000	Reb N
5	13.020		0.0000	0.00000	0.0000	Reb M
6	20.477		0.0000	0.00000	0.0000	Reb A
7	20.656	BB	0.0870	5.12708	100.0000	Stev
8	22.191		0.0000	0.00000	0.0000	Reb F
9	22.970		0.0000	0.00000	0.0000	Reb C
10	23.633		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Stev Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.501		0.0000	0.00000	0.0000	Rubu
12	32.271		0.0000	0.00000	0.0000	Reb B
13	33.279		0.0000	0.00000	0.0000	Sbio

Totals : 5.12708

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

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

Sample Name: Stev Standard

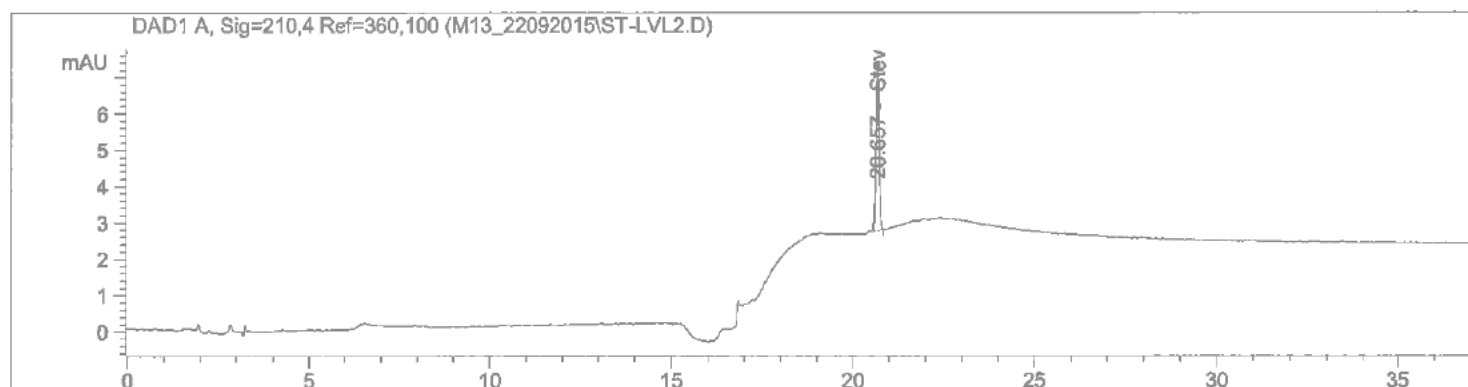
```

=====
Acq. Operator   : QC Chemist                      Seq. Line :   22
Acq. Instrument : HPLC 3                          Location  : Vial 20
Injection Date  : 9/24/2015 6:29:59 AM            Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 9:29:58 AM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info      : Stev Std, 9.5577 mg/L
=====

```



```

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

```

```

Sorted By           :      Signal
Calib. Data Modified :      Monday, September 28, 2015 9:22:25 AM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.920		0.0000	0.00000	0.0000	Reb E
2	9.643		0.0000	0.00000	0.0000	Reb O
3	10.288		0.0000	0.00000	0.0000	Reb D
4	11.160		0.0000	0.00000	0.0000	Reb N
5	13.020		0.0000	0.00000	0.0000	Reb M
6	20.477		0.0000	0.00000	0.0000	Reb A
7	20.657	BB	0.0885	26.34745	100.0000	Stev
8	22.191		0.0000	0.00000	0.0000	Reb F
9	22.970		0.0000	0.00000	0.0000	Reb C
10	23.633		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Stev Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.501		0.0000	0.00000	0.0000	Rubu
12	32.271		0.0000	0.00000	0.0000	Reb B
13	33.279		0.0000	0.00000	0.0000	Sbio

Totals : 26.34745

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Stev Standard

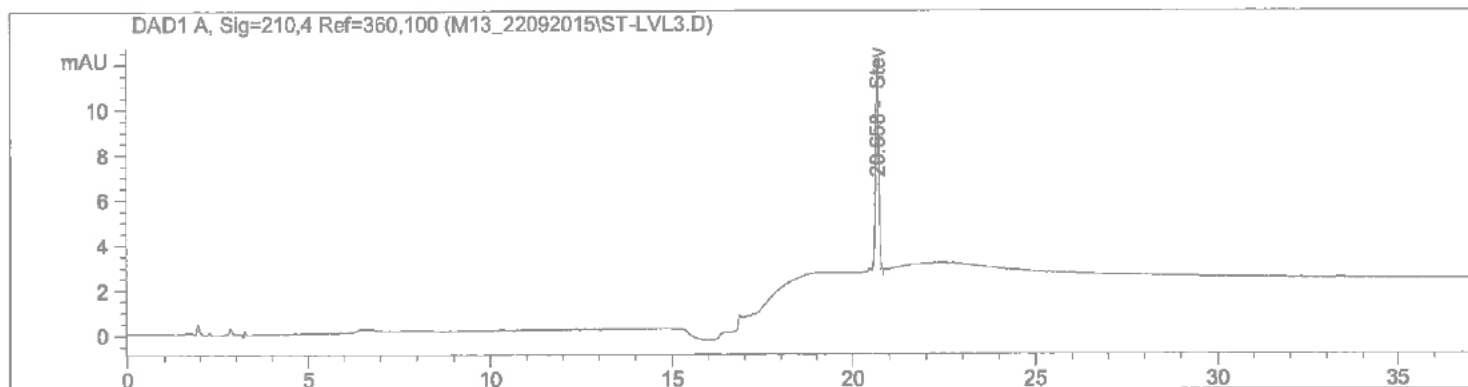
```

=====
Acq. Operator   : QC Chemist                      Seq. Line :   23
Acq. Instrument : HPLC 3                          Location  : Vial 21
Injection Date  : 9/24/2015 7:34:15 AM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 9:29:58 AM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info      : Stev Std, 19.1154 mg/L
=====

```



```

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

```

```

Sorted By           :      Signal
Calib. Data Modified :      Monday, September 28, 2015 9:22:25 AM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.920		0.0000	0.00000	0.0000	Reb E
2	9.643		0.0000	0.00000	0.0000	Reb O
3	10.288		0.0000	0.00000	0.0000	Reb D
4	11.160		0.0000	0.00000	0.0000	Reb N
5	13.020		0.0000	0.00000	0.0000	Reb M
6	20.477		0.0000	0.00000	0.0000	Reb A
7	20.658	BB	0.0882	52.57774	100.0000	Stev
8	22.191		0.0000	0.00000	0.0000	Reb F
9	22.970		0.0000	0.00000	0.0000	Reb C
10	23.633		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Stev Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.501		0.0000	0.00000	0.0000	Rubu
12	32.271		0.0000	0.00000	0.0000	Reb B
13	33.279		0.0000	0.00000	0.0000	Sbio

Totals : 52.57774

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

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

Sample Name: Stev Standard

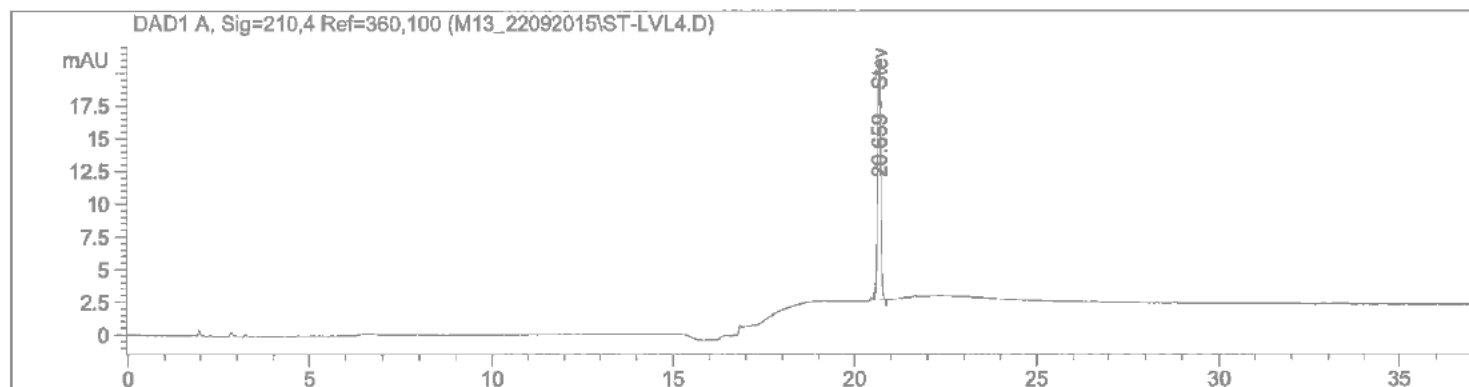
```

=====
Acq. Operator   : QC Chemist                      Seq. Line :   24
Acq. Instrument : HPLC 3                          Location  : Vial 22
Injection Date  : 9/24/2015 8:37:11 AM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 9:29:58 AM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : Stev Std, 38.2308 mg/L
=====

```



```

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

```

```

Sorted By           :      Signal
Calib. Data Modified :      Monday, September 28, 2015 9:22:25 AM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.920		0.0000	0.00000	0.0000	Reb E
2	9.643		0.0000	0.00000	0.0000	Reb O
3	10.288		0.0000	0.00000	0.0000	Reb D
4	11.160		0.0000	0.00000	0.0000	Reb N
5	13.020		0.0000	0.00000	0.0000	Reb M
6	20.477		0.0000	0.00000	0.0000	Reb A
7	20.659	VB	0.0887	104.56646	100.0000	Stev
8	22.191		0.0000	0.00000	0.0000	Reb F
9	22.970		0.0000	0.00000	0.0000	Reb C
10	23.633		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Stev Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.501		0.0000	0.00000	0.0000	Rubu
12	32.271		0.0000	0.00000	0.0000	Reb B
13	33.279		0.0000	0.00000	0.0000	Sbio

Totals : 104.56646

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Stev Standard

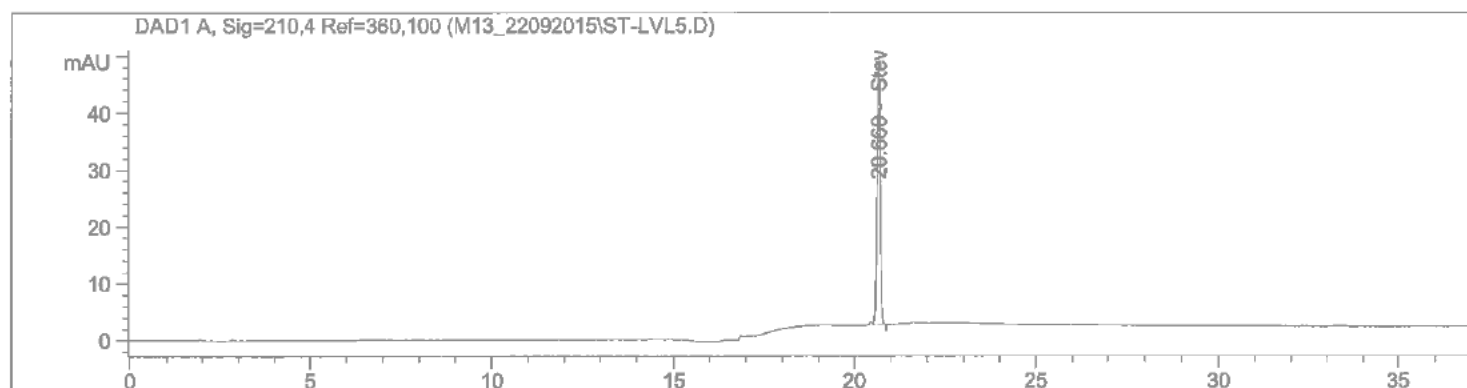
```

=====
Acq. Operator   : QC Chemist                      Seq. Line :   25
Acq. Instrument : HPLC 3                          Location  : Vial 23
Injection Date  : 9/24/2015 9:40:26 AM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 9:29:58 AM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info      : Stev Std, 95.5769 mg/L
=====

```



```

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

```

```

Sorted By           :      Signal
Calib. Data Modified :      Monday, September 28, 2015 9:22:25 AM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	8.920		0.0000	0.00000	0.0000	Reb E
2	9.643		0.0000	0.00000	0.0000	Reb O
3	10.288		0.0000	0.00000	0.0000	Reb D
4	11.160		0.0000	0.00000	0.0000	Reb N
5	13.020		0.0000	0.00000	0.0000	Reb M
6	20.477		0.0000	0.00000	0.0000	Reb A
7	20.660	VB	0.0888	263.25757	100.0000	Stev
8	22.191		0.0000	0.00000	0.0000	Reb F
9	22.970		0.0000	0.00000	0.0000	Reb C
10	23.633		0.0000	0.00000	0.0000	Dulc.A



Sample Name: Stev Standard

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
11	26.501		0.0000	0.00000	0.0000	Rubu
12	32.271		0.0000	0.00000	0.0000	Reb B
13	33.279		0.0000	0.00000	0.0000	Sbio

Totals : 263.25757

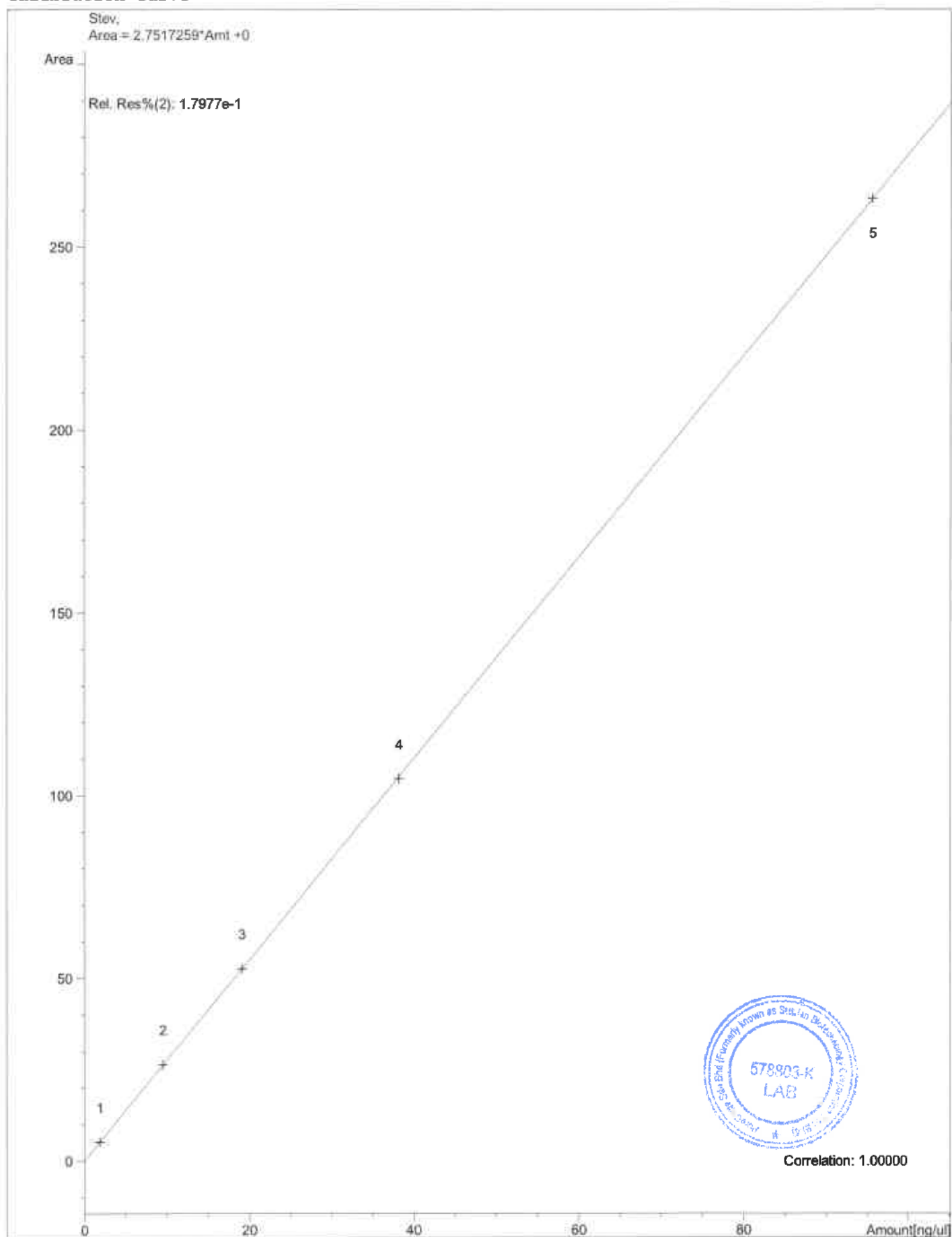
2 Warnings or Errors :


Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

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

Calibration Curve



 PureCircle PureCircle Sdn Bhd	Report#: KNN02102015001	Issue date: 02/10/15
		Revision date: -----
HPLC Assay of A95 samples in 5 commercial lots		Revision No: 00

APPENDIX B4

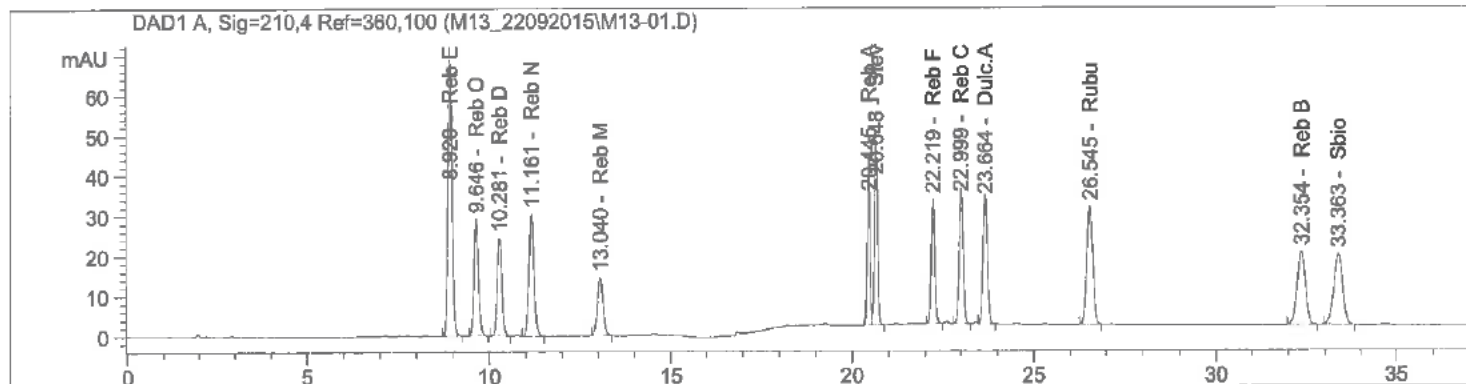
M9 retention time marker

=====

Acq. Operator	: QC Chemist	Seq. Line	: 5
Acq. Instrument	: HPLC 3	Location	: Vial 83
Injection Date	: 9/23/2015 12:37:07 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M		
Last changed	: 9/22/2015 9:46:31 PM by QC Chemist		
Analysis Method	: C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M		
Last changed	: 10/2/2015 10:52:10 AM by QC Chemist (modified after loading)		
Method Info	: New Method: RE,RO,RD,RN,RM		
Sample Info	: M13 - Beginning of Sequence		

=====

Column Description : Poroshell 120 SB-C18
Product# : 683975-902 Batch#: B10225
Serial# : USCEV01277
Diameter : 4.6 mm Length : 150.0 mm
Particle size : 2.7 µm Void volume : 60.0 %
Maximum Pressure : 600 bar Maximum pH : 9
Maximum Temperature: 60 °C
Comment : JECFA2010



=====
Area Percent Report with Performance
=====

Calib. Data Modified : Friday, October 02, 2015 10:52:05 AM
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=210,4 Ref=360,100



Sample Name: M13

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
8.920	1.98	551.08472	69.02546	0.93	0.1213	29940	-	-
9.646	2.22	256.61197	29.18185	0.91	0.1317	29734	3.37	1.12
10.281	2.44	217.80515	24.32576	0.93	0.1350	32130	2.80	1.10
11.161	2.73	296.59827	30.52435	0.94	0.1467	32079	3.67	1.12
13.040	3.36	156.26929	14.38162	0.94	0.1667	33914	7.05	1.23
20.445	5.83	227.03499	41.56662	0.97	0.0837	330529	34.75	1.74
20.648	5.90	257.18106	44.94873	0.98	0.0867	314467	1.40	1.01
22.219	6.43	214.88655	30.94191	0.99	0.1053	246504	9.61	1.09
22.999	6.69	255.96837	33.49796	1.01	0.1173	212854	4.12	1.04
23.664	6.91	269.05594	32.36592	1.02	0.1267	193355	3.20	1.03
26.545	7.87	331.94730	29.45314	1.06	0.1733	129928	11.28	1.14
32.354	9.82	291.00949	18.33842	1.10	0.2444	97054	16.34	1.25
33.363	10.15	302.30469	17.60313	1.08	0.2633	88925	2.33	1.03

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



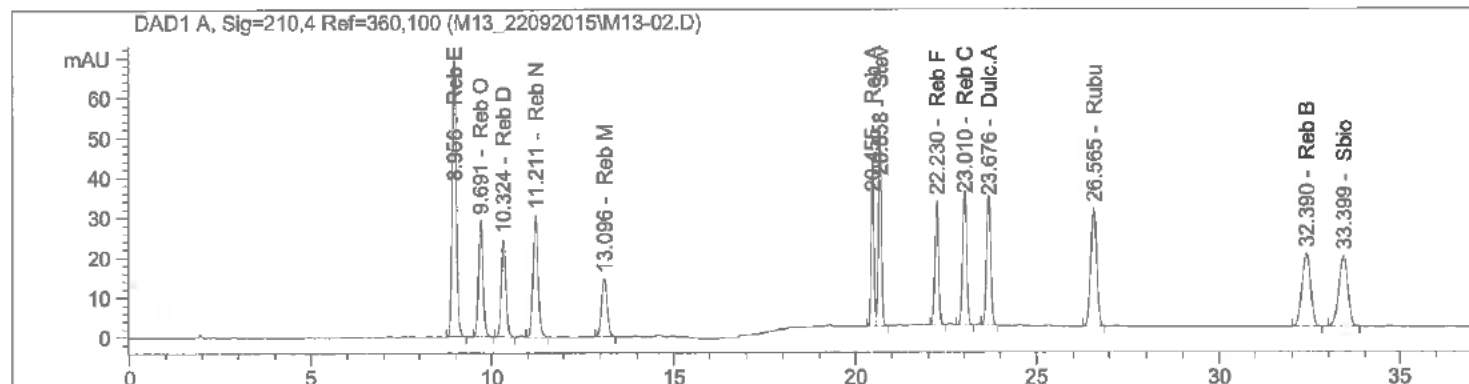
Sample Name: M13

```
=====
Acq. Operator   : QC Chemist                      Seq. Line :   26
Acq. Instrument : HPLC 3                          Location  : Vial 83
Injection Date  : 9/24/2015 10:43:43 AM           Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/2/2015 10:52:10 AM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info      : M13 - Middle of Sequence
=====
```

```
Column Description : Poroshell 120 SB-C18
Product#           : 683975-902                   Batch# : B10225
Serial#            : USCEV01277
Diameter           : 4.6 mm                       Length : 150.0 mm
Particle size      : 2.7 µm                      Void volume : 60.0 %
Maximum Pressure   : 600 bar                      Maximum pH :    9
Maximum Temperature: 60 °C
Comment            : JECFA2010
=====
```



```
=====
Area Percent Report with Performance
=====
```

```
Calib. Data Modified : Friday, October 02, 2015 10:52:05 AM
Multiplier           : 1.0000
Dilution             : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100



Sample Name: M13

RetTime [min]	k' [min]	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
8.956	1.99	552.78729	69.23502	0.93	0.1200	30858	-	-
9.691	2.24	257.70865	29.22470	0.92	0.1333	29264	3.41	1.12
10.324	2.45	218.86394	24.37483	0.93	0.1350	32398	2.77	1.09
11.211	2.75	297.52045	30.59371	0.95	0.1467	32370	3.70	1.12
13.096	3.38	156.49368	14.38658	0.95	0.1667	34204	7.07	1.23
20.455	5.84	227.69621	41.64502	0.97	0.0844	325061	34.43	1.73
20.658	5.91	258.04550	45.03617	0.98	0.0876	307965	1.39	1.01
22.230	6.43	216.72720	30.99574	0.98	0.1067	240617	9.50	1.09
23.010	6.69	257.14145	33.52480	1.02	0.1173	213061	4.09	1.04
23.676	6.91	270.00583	32.39898	1.02	0.1267	193549	3.21	1.03
26.565	7.88	332.94949	29.49408	1.06	0.1711	133526	11.40	1.14
32.390	9.83	291.67178	18.35014	1.11	0.2467	95522	16.38	1.25
33.399	10.17	302.89178	17.58617	1.09	0.2633	89120	2.33	1.03

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



Sample Name: M13

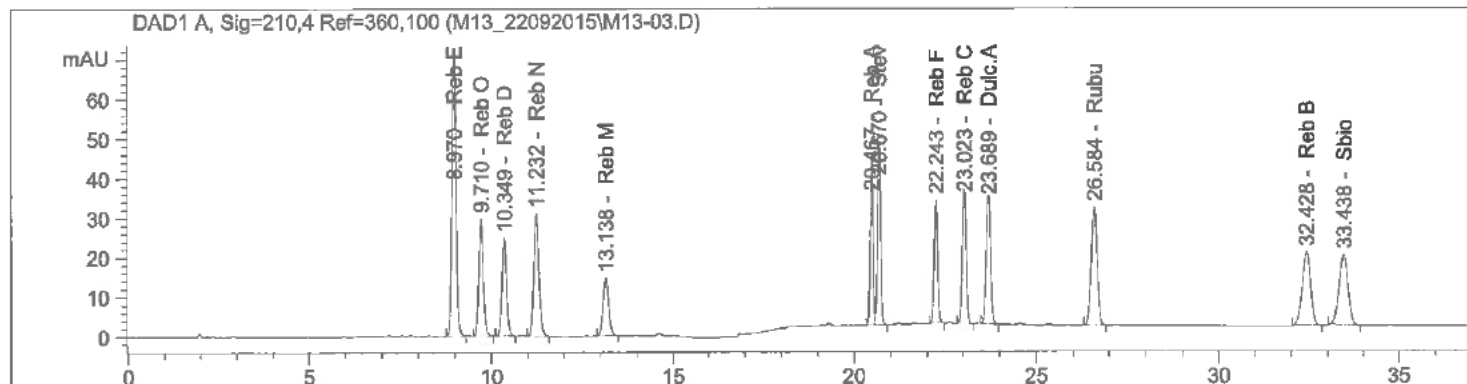
```
=====
Acq. Operator   : QC Chemist                      Seq. Line :   49
Acq. Instrument : HPLC 3                          Location  : Vial 83
Injection Date  : 9/25/2015 10:52:58 AM           Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/2/2015 10:52:10 AM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : M13 - End of sequence
=====
```

```
=====
Column Description : Poroshell 120 SB-C18
Product#          : 683975-902                    Batch# : B10225
Serial#           : USCEV01277
Diameter          : 4.6 mm                        Length : 150.0 mm
Particle size     : 2.7 µm                        Void volume : 60.0 %
Maximum Pressure  : 600 bar                        Maximum pH :    9
Maximum Temperature: 60 °C
Comment           : JECFA2010
=====
```



```
=====
Area Percent Report with Performance
=====
```

```
Calib. Data Modified : Friday, October 02, 2015 10:52:05 AM
Multiplier           : 1.0000
Dilution             : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```


Signal 1: DAD1 A, Sig=210,4 Ref=360,100



Sample Name: M13

RetTime [min]	k' [min]	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
8.970	2.00	558.59296	69.99323	0.93	0.1200	30957	-	-
9.710	2.25	259.74646	29.55478	0.91	0.1317	30127	3.45	1.12
10.349	2.46	221.44629	24.68795	0.93	0.1350	32556	2.82	1.10
11.232	2.75	300.29459	30.87823	0.95	0.1467	32493	3.69	1.12
13.138	3.39	159.56750	14.57910	0.93	0.1667	34424	7.15	1.23
20.467	5.84	230.32327	42.11120	0.97	0.0844	325432	34.29	1.72
20.670	5.91	261.12076	45.56599	0.98	0.0867	315130	1.40	1.01
22.243	6.44	217.67195	31.28289	0.99	0.1067	240901	9.56	1.09
23.023	6.70	259.11572	33.88632	1.02	0.1173	213291	4.09	1.04
23.689	6.92	272.93750	32.74316	1.01	0.1267	193758	3.21	1.03
26.584	7.89	336.13046	29.78913	1.06	0.1733	130316	11.34	1.14
32.428	9.84	295.12943	18.53638	1.11	0.2444	97495	16.43	1.25
33.438	10.18	306.36313	17.79404	1.09	0.2633	89326	2.34	1.03

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

 PureCircle PureCircle Sdn Bhd	Report#: KNN02102015001	Issue date: 02/10/15
		Revision date: -----
HPLC Assay of A95 samples in 5 commercial lots		Revision No: 00

APPENDIX B4

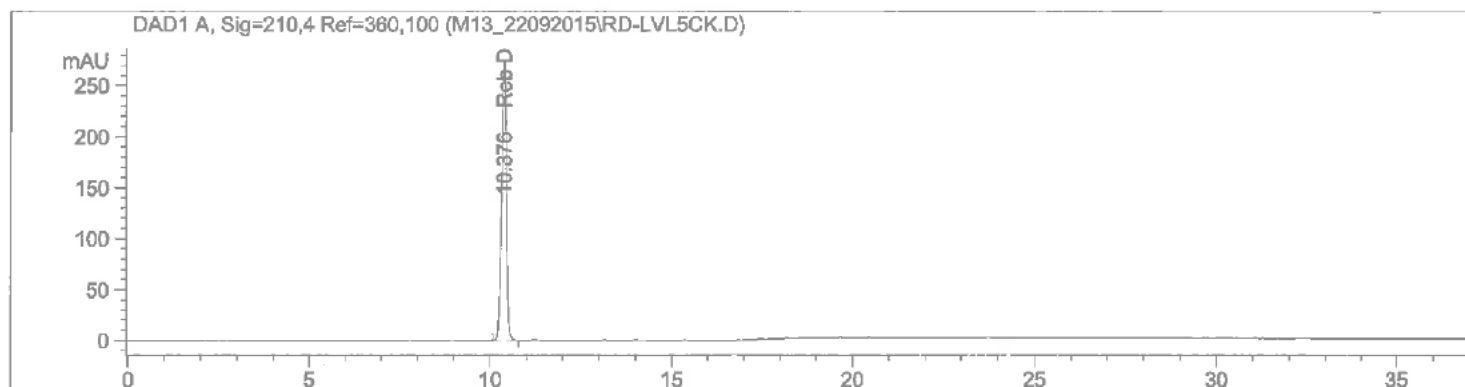
In-assay standard recovery check

Sample Name: Reb D Standard

```
=====
Acq. Operator   : QC Chemist                      Seq. Line :   45
Acq. Instrument : HPLC 3                          Location  : Vial 8
Injection Date  : 9/25/2015 6:41:58 AM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 2:11:06 PM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : Reb D Std, 1301.5102 mg/L
=====
```



```
=====
                        External Standard Report
=====
```

```
Sorted By           :      Signal
Calib. Data Modified :      9/30/2015 12:10:52 PM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.970		-	-	-		Reb E
9.710		-	-	-		Reb O
10.376	BB	2532.28760	5.15213e-1	1304.66627		Reb D
11.232		-	-	-		Reb N
13.138		-	-	-		Reb M
20.400		-	-	-		Reb A
20.600		-	-	-		Stev
22.243		-	-	-		Reb F
23.023		-	-	-		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Reb D Standard

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.428						Reb B
33.438						Sbio

Totals : 1304.66627

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Reb M Standard

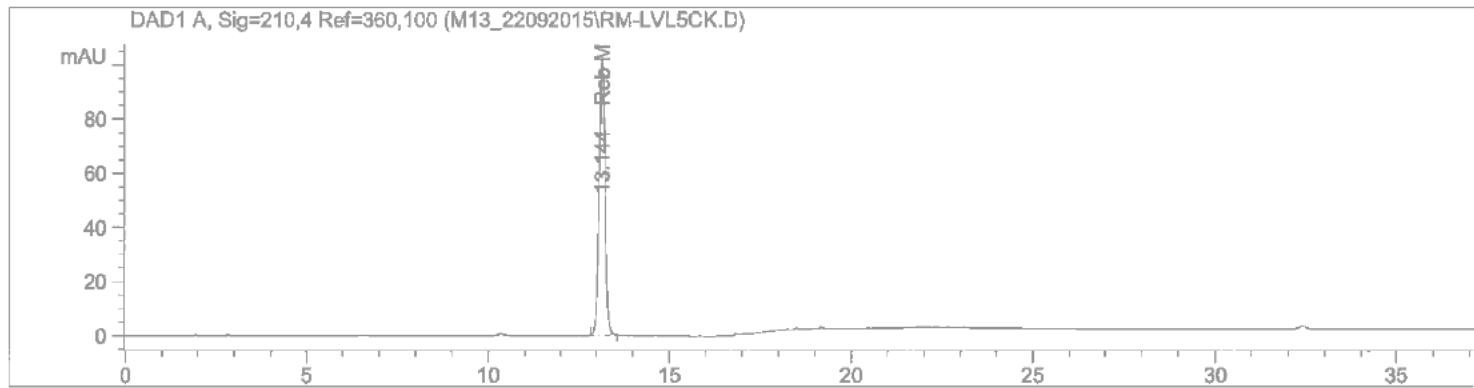
```

=====
Acq. Operator   : QC Chemist                      Seq. Line :   46
Acq. Instrument : HPLC 3                          Location  : Vial 13
Injection Date  : 9/25/2015 7:44:54 AM             Inj       :    1
                                                    Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 11:56:11 AM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : Reb M Std, 683.3784 mg/L
=====

```



```

=====
                        External Standard Report
=====

```

```

Sorted By           :      Signal
Calib. Data Modified :      Monday, September 28, 2015 11:56:04 AM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.970	-	-	-	-		Reb E
9.710	-	-	-	-		Reb O
10.349	-	-	-	-		Reb D
11.232	-	-	-	-		Reb N
13.144	BB	1155.47717	5.94426e-1	686.84619		Reb M
20.467	-	-	-	-		Reb A
20.670	-	-	-	-		Stev
22.243	-	-	-	-		Reb F
23.023	-	-	-	-		Reb C
23.689	-	-	-	-		Dulc.A
26.584	-	-	-	-		Rubu



Sample Name: Reb M Standard

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.428		-	-	-		Reb B
33.438		-	-	-		Sbio

Totals : 686.84619

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

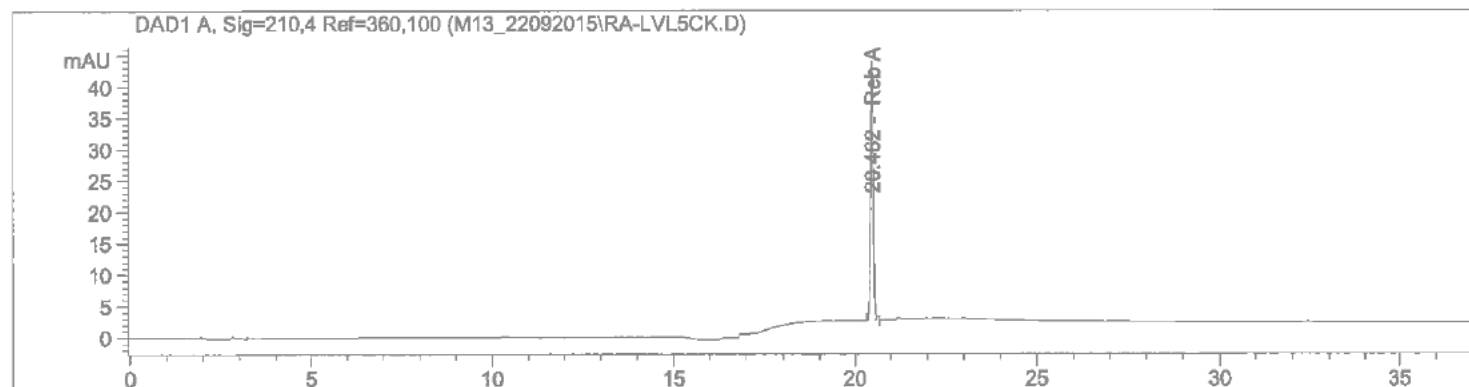
*** End of Report ***



```
=====
Acq. Operator   : QC Chemist                      Seq. Line :   47
Acq. Instrument : HPLC 3                          Location  : Vial 18
Injection Date  : 9/25/2015 8:47:42 AM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 11:56:11 AM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : Reb A Std, 100.2412 mg/L
=====
```



External Standard Report

```
=====
Sorted By      : Signal
Calib. Data Modified : Monday, September 28, 2015 11:56:04 AM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.970						Reb E
9.710						Reb O
10.349						Reb D
11.232						Reb N
13.138						Reb M
20.462	BB	225.26048	4.44030e-1	100.02247		Reb A
20.670						Stev
22.243						Reb F
23.023						Reb C
23.689						Dulc.A
26.584						Rubu



Sample Name: Reb A Standard

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.428						Reb B
33.438						Sbio

Totals : 100.02247

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

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

Sample Name: Stev Standard

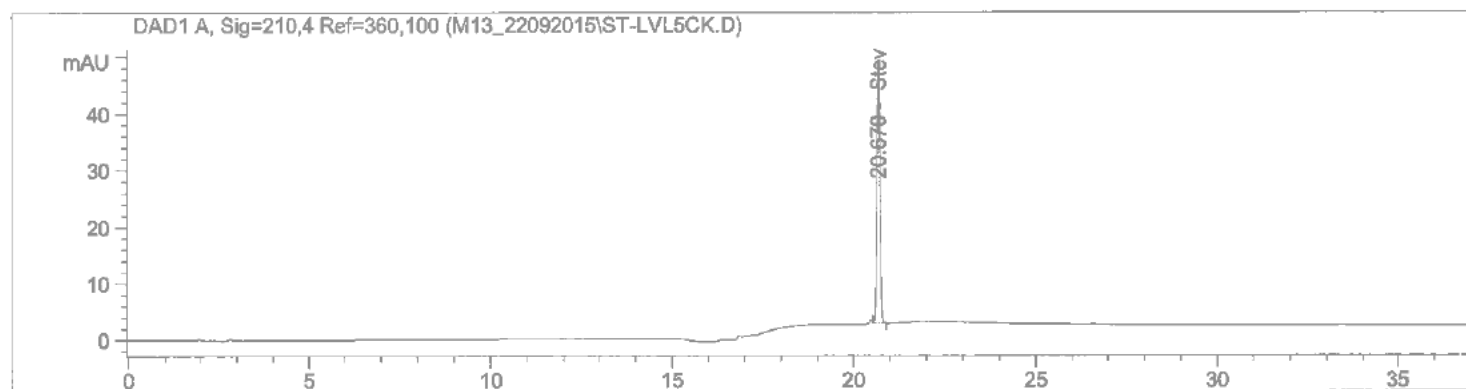
```

=====
Acq. Operator   : QC Chemist                      Seq. Line :   48
Acq. Instrument : HPLC 3                          Location  : Vial 23
Injection Date  : 9/25/2015 9:50:09 AM            Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 9/28/2015 11:56:11 AM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : Stev Std, 95.5769 mg/L
=====

```



```

=====
                        External Standard Report
=====

```

```

Sorted By           : Signal
Calib. Data Modified : Monday, September 28, 2015 11:56:04 AM
Multiplier          : 1.0000
Dilution            : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.970	-	-	-	-	-	Reb E
9.710	-	-	-	-	-	Reb O
10.349	-	-	-	-	-	Reb D
11.232	-	-	-	-	-	Reb N
13.138	-	-	-	-	-	Reb M
20.467	-	-	-	-	-	Reb A
20.670	VB	264.55365	3.63408e-1	96.14099	-	Stev
22.243	-	-	-	-	-	Reb F
23.023	-	-	-	-	-	Reb C
23.689	-	-	-	-	-	Dulc.A
26.584	-	-	-	-	-	Rubu



Sample Name: Stev Standard

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.428						Reb B
33.438						Sbio

Totals : 96.14099


2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found


*** End of Report ***



 PureCircle PureCircle Sdn Bhd	Report#: KNN02102015001	Issue date: 02/10/15
		Revision date: -----
HPLC Assay of A95 samples in 5 commercial lots		Revision No: 00

APPENDIX C

Sample Chromatograms


 PureCircle Sdn Bhd HPLC sample data record	Document#: PCL-LAB-003	Issue date: 01/12/07
		Revision date: -----
	Revision No: 00	

Sample Info	A95 lot #PT180815		
	Replicate I	Replicate II	Replicate III
Sample ID	PT180815#1	PT180815#2	PT180815#3
Weight, mg	50.38	50.14	50.86
Volumetric flask volume, mL	25	25	25
Concentration, mg/L	2015.24000	2005.6000	2034.4000
Water, %wt (Karl Fischer)	9.4162	9.4162	9.4162
Adjusted concentration, mg/L	1825.4447	1816.7487	1842.8368
Remarks			

Sample Info	A95 lot #PT240815		
	Replicate I	Replicate II	Replicate III
Sample ID	PT240815#1	PT240815#2	PT240815#3
Weight, mg	50.05	50.00	50.58
Volumetric flask volume, mL	100	100	100
Concentration, mg/L	2002.0000	2000.0000	2023.2000
Water, %wt (Karl Fischer)	9.4347	9.4347	9.4347
Adjusted concentration, mg/L	1813.1173	1811.3060	1832.3171
Remarks			

Sample Info	A95 lot #PT280815		
	Replicate I	Replicate II	Replicate III
Sample ID	PT280815#1	PT280815#2	PT280815#3
Weight, mg	50.78	50.71	50.64
Volumetric flask volume, mL	25	25	25
Concentration, mg/L	2031.2000	2028.4000	2025.6000
Water, %wt (Karl Fischer)	9.5547	9.5547	9.5547
Adjusted concentration, mg/L	1837.1249	1834.5925	1832.0600
Remarks			



 PureCircle	PureCircle Sdn Bhd	Document#: PCL-LAB-003	Issue date: 01/12/07
			Revision date: -----
HPLC sample data record			Revision No: 00

Sample Info	A95 lot #PT010915		
	Replicate I	Replicate II	Replicate III
Sample ID	PT010915#1	PT010915 #2	PT010915 #3
Weight, mg	50.39	50.02	49.95
Volumetric flask volume, mL	25	25	25
Concentration, mg/L	2015.6000	2000.8000	1998.0000
Water, %wt (Karl Fischer)	9.0090	9.0090	9.0090
Adjusted concentration, mg/L	1834.0146	1820.5479	1818.0002
Remarks			

Sample Info	A95 lot #PT040915		
	Replicate I	Replicate II	Replicate III
Sample ID	PT040915#1	PT040915 #2	PT040915#3
Weight, mg	50.27	51.07	51.00
Volumetric flask volume, mL	25	25	25
Concentration, mg/L	2010.8000	2042.8000	2040.0000
Water, %wt (Karl Fischer)	8.9945	8.9945	8.9945
Adjusted concentration, mg/L	1829.9386	1859.0604	1856.5122
Remarks			

Done by:

[Redacted Signature]

QC Chemist

22/09/15

Date

Checked by:

[Redacted Signature]

QA/QC Manager

22/09/15

Date



Sample Name: Sample

```

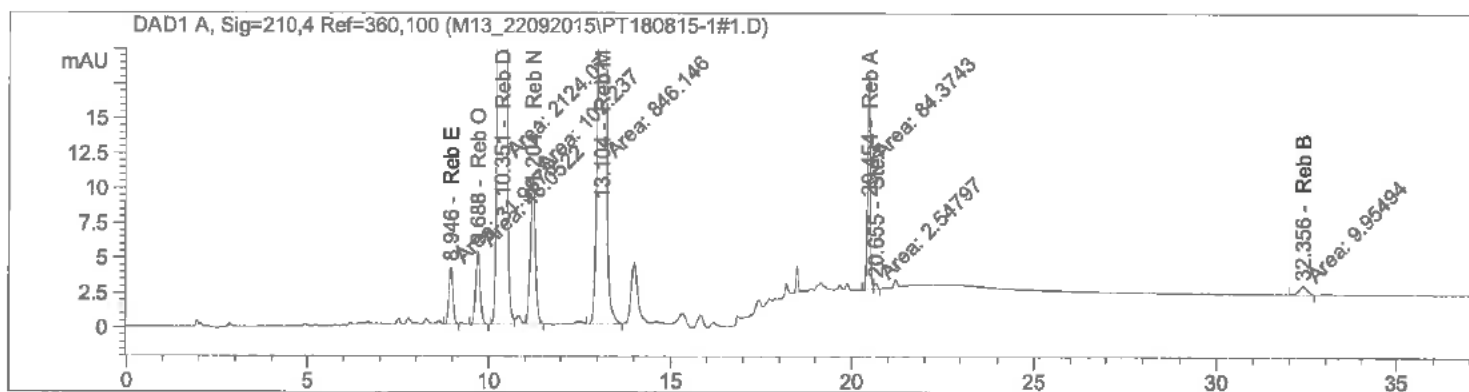
=====
Acq. Operator   : QC Chemist                      Seq. Line :   27
Acq. Instrument : HPLC 3                          Location  : Vial 24
Injection Date  : 9/24/2015 11:47:53 AM           Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 2:36:08 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT180815 1, 1825.4447 mg/L
=====

```



```

=====
External Standard Report
=====

```

```

Sorted By           : Signal
Calib. Data Modified : 9/30/2015 12:10:52 PM
Multiplier          : 1.0000
Dilution            : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.946	MM	31.95726	5.15213e-1	14.10011	Reb	E
9.688	MM	48.05222	5.15213e-1	31.51679	Reb	O
10.351	MM	2124.07007	5.15213e-1	1094.34749	Reb	D
11.204	MM	102.23683	5.15213e-1	59.48440	Reb	N
13.104	MM	846.14648	5.94426e-1	502.97185	Reb	M
20.454	MF	84.37432	4.44030e-1	37.46475	Reb	A
20.655	FM	2.54797	3.63408e-1	9.25952e-1	Stev	
22.243		-	-	-	Reb	F
23.023		-	-	-	Reb	C
23.689		-	-	-	Dulc.	A
26.584		-	-	-	Rubu	



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.356	MF	9.95494	3.63408e-1	3.61771		Reb B
33.438		-	-	-		Sbio

Totals : 1744.42905

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



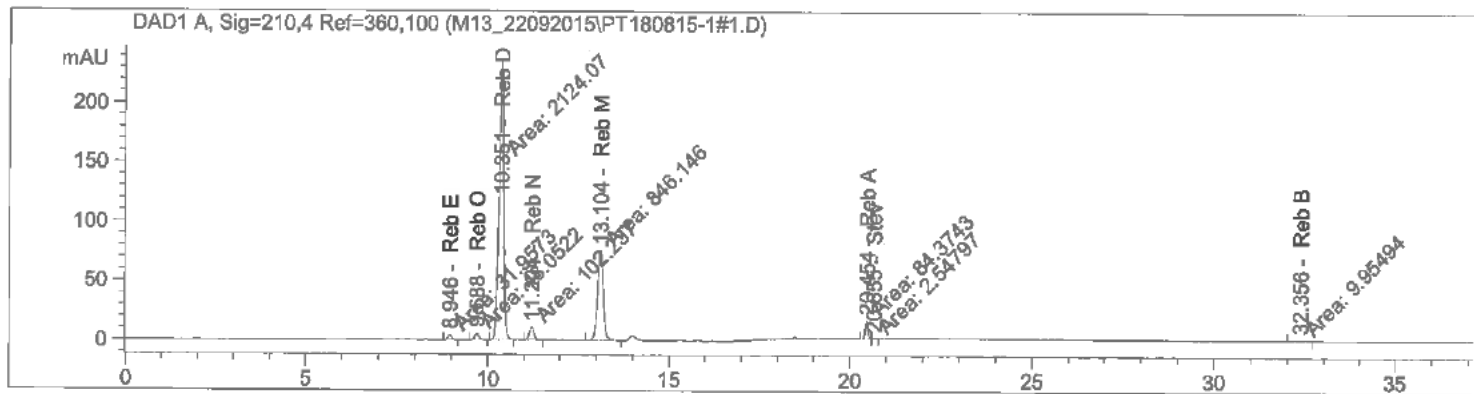
Sample Name: Sample

```
=====
Acq. Operator   : QC Chemist                      Seq. Line :   27
Acq. Instrument : HPLC 3                          Location  : Vial 24
Injection Date  : 9/24/2015 11:47:53 AM           Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 2:39:45 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT180815 1, 1825.4447 mg/L
=====
```



External Standard Report

```
=====
Sorted By      : Signal
Calib. Data Modified : 9/30/2015 12:10:52 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.946	MM	31.95726	5.15213e-1	14.10011		Reb E
9.688	MM	48.05222	5.15213e-1	31.51679		Reb O
10.351	MM	2124.07007	5.15213e-1	1094.34749		Reb D
11.204	MM	102.23683	5.15213e-1	59.48440		Reb N
13.104	MM	846.14648	5.94426e-1	502.97185		Reb M
20.454	MF	84.37432	4.44030e-1	37.46475		Reb A
20.655	FM	2.54797	3.63408e-1	9.25952e-1		Stev
22.243		-	-	-		Reb F
23.023		-	-	-		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.356	MF	9.95494	3.63408e-1	3.61771		Reb B
33.438		-	-	-		Sbio

Totals : 1744.42905

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

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



Sample Name: Sample

```

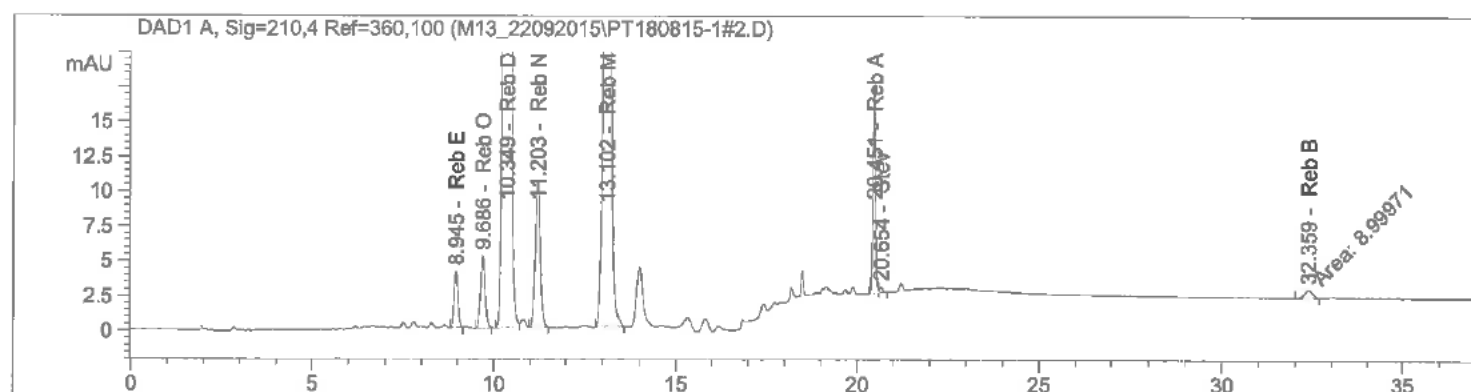
=====
Acq. Operator   : QC Chemist                      Seq. Line :   28
Acq. Instrument : HPLC 3                          Location  : Vial 25
Injection Date  : 9/24/2015 12:51:19 PM           Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 2:43:13 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT180815 1, 1816.7487 mg/L
=====

```



```

=====
External Standard Report
=====

```

```

Sorted By           : Signal
Calib. Data Modified : 9/30/2015 12:10:52 PM
Multiplier          : 1.0000
Dilution            : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.945	BB	30.67148	5.15213e-1	13.53280		Reb E
9.686	BB	46.78622	5.15213e-1	30.68643		Reb O
10.349	BV	2102.14795	5.15213e-1	1083.05294		Reb D
11.203	VB	101.13636	5.15213e-1	58.84412		Reb N
13.102	BB	841.09589	5.94426e-1	499.96964		Reb M
20.451	BV	83.05584	4.44030e-1	36.87931		Reb A
20.654	VB	2.07020	3.63408e-1	7.52328e-1		Stev
22.243		-	-	-		Reb F
23.023		-	-	-		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.359	MM	8.99971	3.63408e-1	3.27057		Reb B
33.438		-	-	-		Sbio

Totals : 1726.98813

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

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

Sample Name: Sample

```

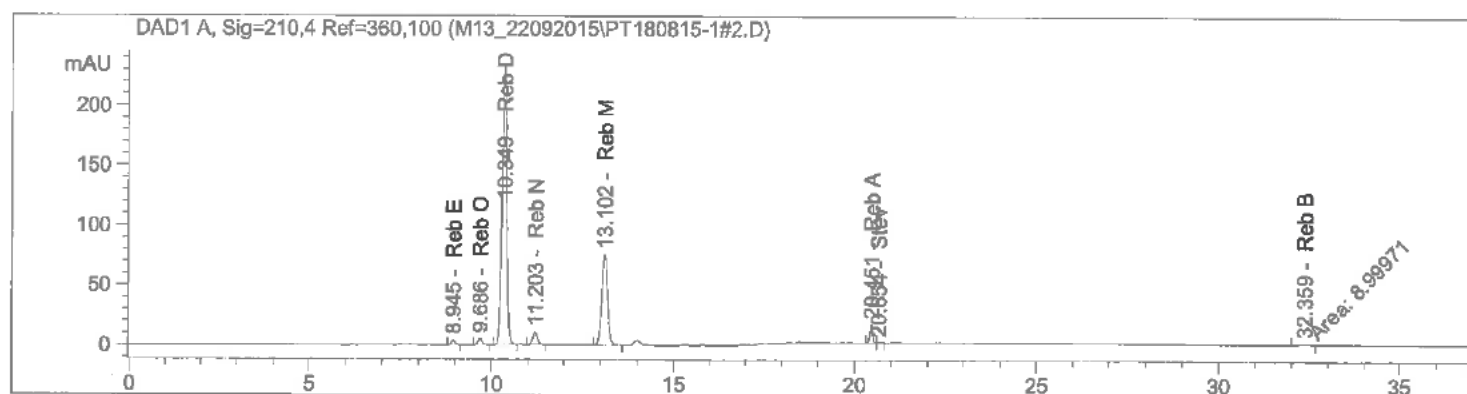
=====
Acq. Operator   : QC Chemist                      Seq. Line :   28
Acq. Instrument : HPLC 3                          Location  : Vial 25
Injection Date  : 9/24/2015 12:51:19 PM           Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 2:45:16 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT180815 1, 1816.7487 mg/L
=====

```



```

=====
External Standard Report
=====

```

```

Sorted By      : Signal
Calib. Data Modified : 9/30/2015 12:10:52 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.945	BB	30.67148	5.15213e-1	13.53280		Reb E
9.686	BB	46.78622	5.15213e-1	30.68643		Reb O
10.349	BV	2102.14795	5.15213e-1	1083.05294		Reb D
11.203	VB	101.13636	5.15213e-1	58.84412		Reb N
13.102	BB	841.09589	5.94426e-1	499.96964		Reb M
20.451	BV	83.05584	4.44030e-1	36.87931		Reb A
20.654	VB	2.07020	3.63408e-1	7.52328e-1		Stev
22.243		-	-	-		Reb F
23.023		-	-	-		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.359	MM	8.99971	3.63408e-1	3.27057		Reb B
33.438		-	-	-		Sbio

Totals : 1726.98813

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

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



Sample Name: Sample

```

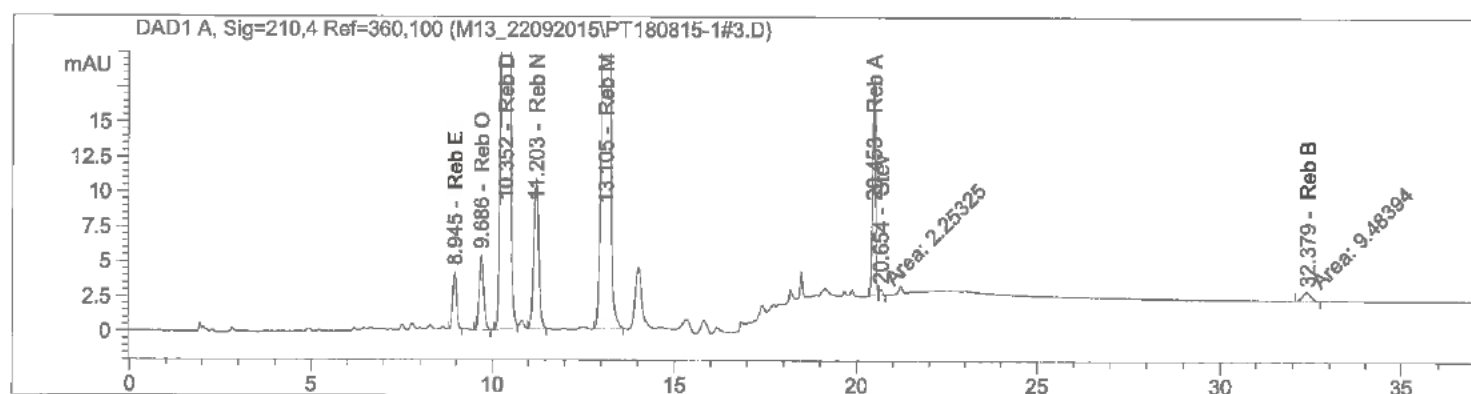
=====
Acq. Operator   : QC Chemist                      Seq. Line :   29
Acq. Instrument : HPLC 3                          Location  : Vial 26
Injection Date  : 9/24/2015 1:54:28 PM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 3:01:51 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT180815 1, 1842.8368 mg/L
=====

```



```

=====
External Standard Report
=====

```

```

Sorted By      : Signal
Calib. Data Modified : 9/30/2015 12:10:52 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.945	VB	31.65989	5.15213e-1	13.96890		Reb E
9.686	BB	48.11968	5.15213e-1	31.56103		Reb O
10.352	BV	2149.80347	5.15213e-1	1107.60566		Reb D
11.203	VB	103.36661	5.15213e-1	60.14174		Reb N
13.105	BB	854.99158	5.94426e-1	508.22960		Reb M
20.453	BV	84.78565	4.44030e-1	37.64740		Reb A
20.654	MM	2.25325	3.63408e-1	8.18849e-1		Stev
22.243		-				Reb F
23.023		-				Reb C
23.689		-				Dulc.A
26.584		-				Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.379	MM	9.48394	3.63408e-1	3.44654		Reb B
33.438						Sbio

Totals : 1763.41973

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

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



Sample Name: Sample

```

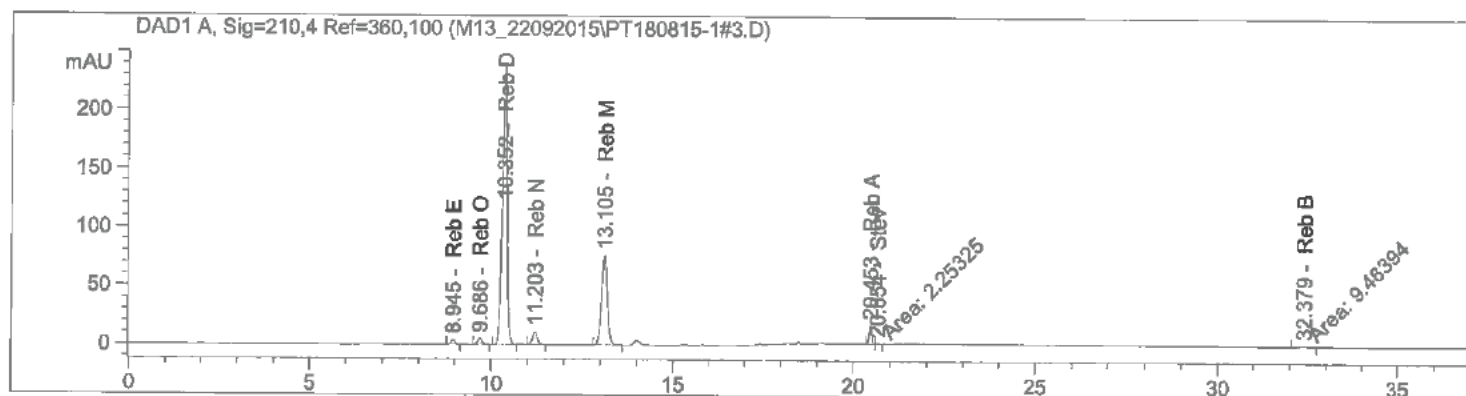
=====
Acq. Operator   : QC Chemist                      Seq. Line :   29
Acq. Instrument : HPLC 3                          Location  : Vial 26
Injection Date  : 9/24/2015 1:54:28 PM            Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 3:03:20 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT180815 1, 1842.8368 mg/L
=====

```



```

=====
External Standard Report
=====

```

```

Sorted By      : Signal
Calib. Data Modified : 9/30/2015 12:10:52 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.945	VB	31.65989	5.15213e-1	13.96890		Reb E
9.686	BB	48.11968	5.15213e-1	31.56103		Reb O
10.352	BV	2149.80347	5.15213e-1	1107.60566		Reb D
11.203	VB	103.36661	5.15213e-1	60.14174		Reb N
13.105	BB	854.99158	5.94426e-1	508.22960		Reb M
20.453	BV	84.78565	4.44030e-1	37.64740		Reb A
20.654	MM	2.25325	3.63408e-1	8.18849e-1		Stev
22.243		-	-	-		Reb F
23.023		-	-	-		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.379	MM	9.48394	3.63408e-1	3.44654		Reb B
33.438						Sbio

Totals : 1763.41973

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Sample

```

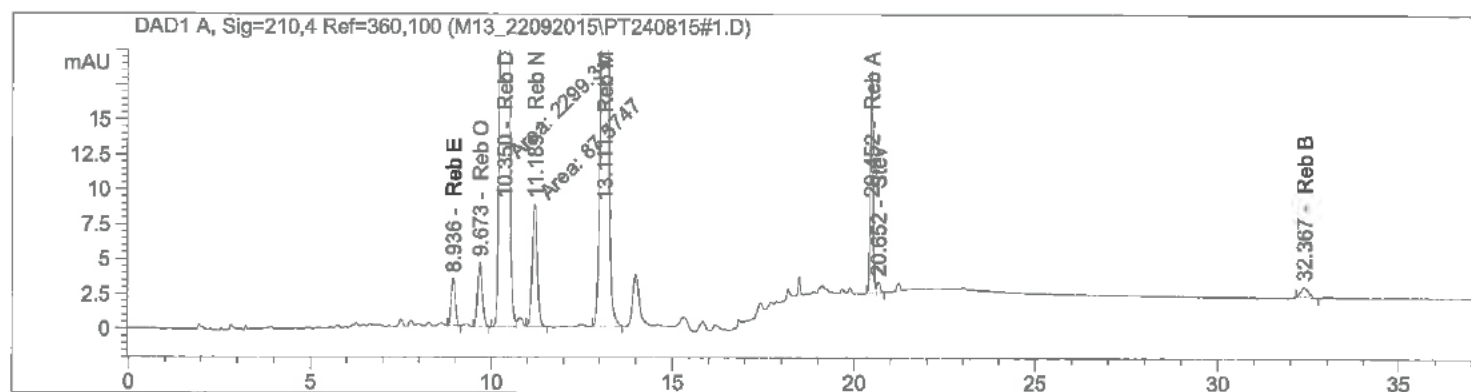
=====
Acq. Operator   : QC Chemist                      Seq. Line :   33
Acq. Instrument : HPLC 3                          Location  : Vial 30
Injection Date  : 9/24/2015 6:06:32 PM            Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 3:35:02 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT240815, 1813.1173 mg/L
=====

```



```

=====
External Standard Report
=====

```

```

Sorted By           : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:35:02 PM
Multiplier          : 1.0000
Dilution            : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.936	BB	26.81107	5.15213e-1	11.82952		Reb E
9.673	BB	43.08279	5.15213e-1	28.25741		Reb O
10.350	MF	2299.33545	5.15213e-1	1184.64641		Reb D
11.189	FM	87.57471	5.15213e-1	50.95355		Reb N
13.111	BB	679.84521	5.94426e-1	404.11798		Reb M
20.452	BV	86.54562	4.44030e-1	38.42888		Reb A
20.652	VB	4.15437	3.63408e-1	1.50973		Stev
22.243		-	-	-		Reb F
23.023		-	-	-		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.367	BB	10.23048	3.63408e-1	3.71784		Reb B
33.438		-	-	-		Sbio

Totals : 1723.46131

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Sample

```

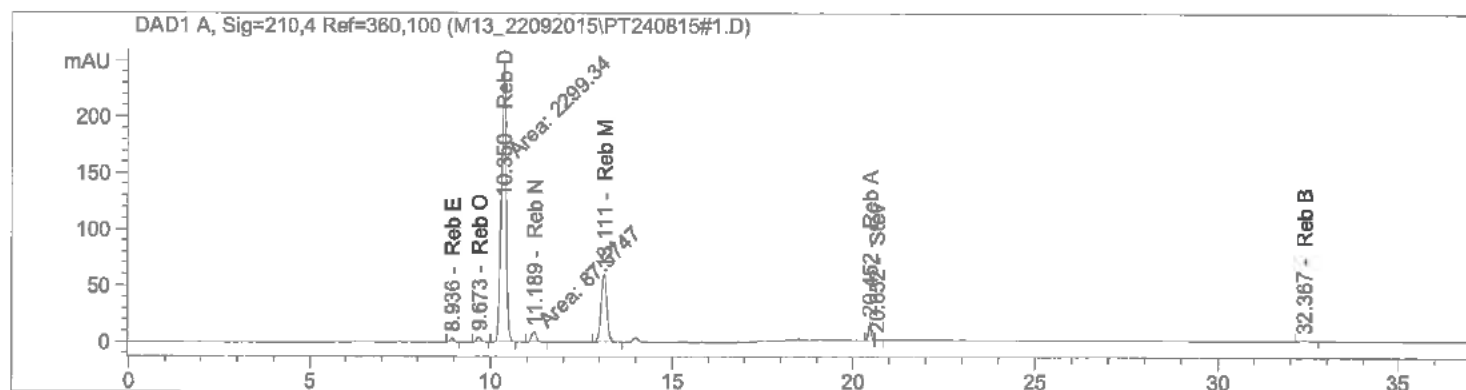
=====
Acq. Operator   : QC Chemist                      Seq. Line :   33
Acq. Instrument : HPLC 3                          Location  : Vial 30
Injection Date  : 9/24/2015 6:06:32 PM            Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 3:38:36 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT240815, 1813.1173 mg/L
=====

```



```

=====
External Standard Report
=====

```

```

Sorted By           : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:35:02 PM
Multiplier          : 1.0000
Dilution            : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.936	BB	26.81107	5.15213e-1	11.82952		Reb E
9.673	BB	43.08279	5.15213e-1	28.25741		Reb O
10.350	MF	2299.33545	5.15213e-1	1184.64641		Reb D
11.189	FM	87.57471	5.15213e-1	50.95355		Reb N
13.111	BB	679.84521	5.94426e-1	404.11798		Reb M
20.452	BV	86.54562	4.44030e-1	38.42888		Reb A
20.652	VB	4.15437	3.63408e-1	1.50973		Stev
22.243		-	-	-		Reb F
23.023		-	-	-		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.367	BB	10.23048	3.63408e-1	3.71784		Reb B
33.438		-	-	-		Sbio

Totals : 1723.46131

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Sample

```

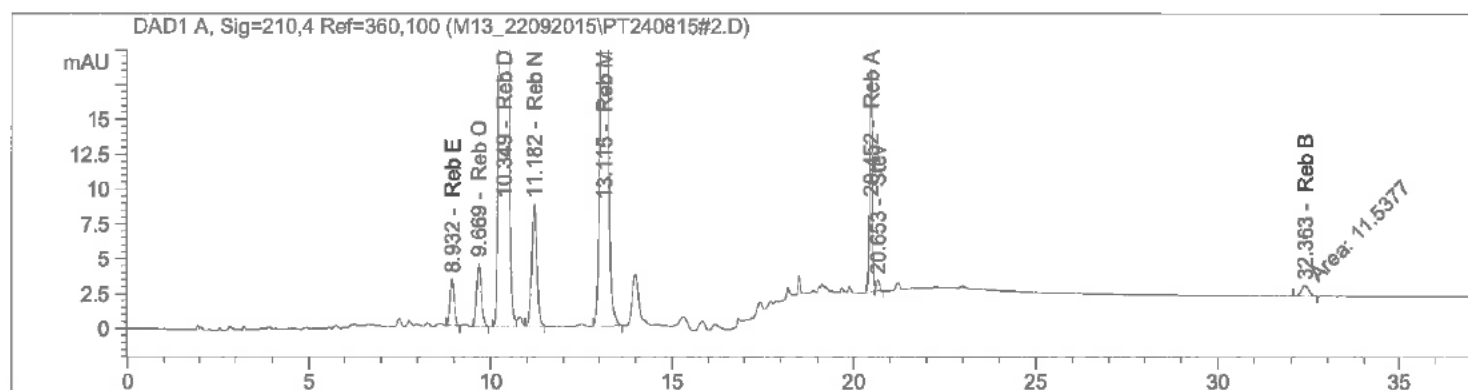
=====
Acq. Operator   : QC Chemist                      Seq. Line :   34
Acq. Instrument : HPLC 3                          Location  : Vial 31
Injection Date  : 9/24/2015 7:09:03 PM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 3:39:19 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT240815, 1811.3060 mg/L
=====

```



=====

External Standard Report

=====

```

Sorted By           :      Signal
Calib. Data Modified :      Thursday, October 01, 2015 3:35:02 PM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.932	BB	26.58440	5.15213e-1	11.72951		Reb E
9.669	BB	43.29434	5.15213e-1	28.39616		Reb O
10.349	BV	2289.37646	5.15213e-1	1179.51542		Reb D
11.182	VB	86.67501	5.15213e-1	50.43008		Reb N
13.115	BB	684.97986	5.94426e-1	407.17014		Reb M
20.452	BV	86.71150	4.44030e-1	38.50253		Reb A
20.653	VB	4.12291	3.63408e-1	1.49830		Stev
22.243		-	-	-		Reb F
23.023		-	-	-		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.363	MM	11.53772	3.63408e-1	4.19290		Reb B
33.438		-	-	-		Sbio

Totals : 1721.43503

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Sample

```

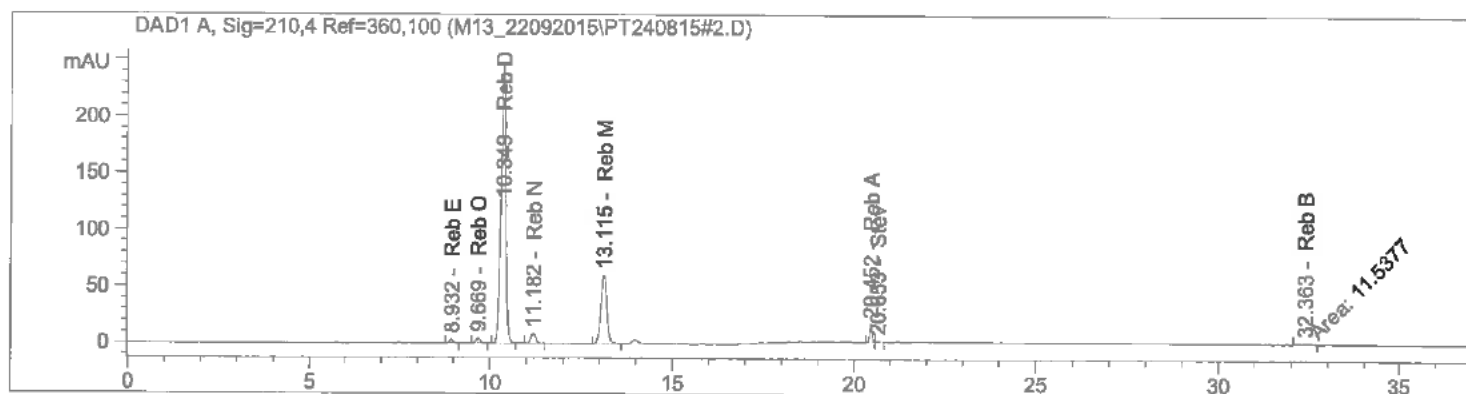
=====
Acq. Operator   : QC Chemist                      Seq. Line :   34
Acq. Instrument : HPLC 3                          Location  : Vial 31
Injection Date  : 9/24/2015 7:09:03 PM            Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 3:40:38 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT240815, 1811.3060 mg/L
=====

```



```

=====
External Standard Report
=====

```

```

Sorted By      : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:35:02 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.932	BB	26.58440	5.15213e-1	11.72951		Reb E
9.669	BB	43.29434	5.15213e-1	28.39616		Reb O
10.349	BV	2289.37646	5.15213e-1	1179.51542		Reb D
11.182	VB	86.67501	5.15213e-1	50.43008		Reb N
13.115	BB	684.97986	5.94426e-1	407.17014		Reb M
20.452	BV	86.71150	4.44030e-1	38.50253		Reb A
20.653	VB	4.12291	3.63408e-1	1.49830		Stev
22.243		-				Reb F
23.023		-				Reb C
23.689		-				Dulc.A
26.584		-				Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.363	MM	11.53772	3.63408e-1	4.19290		Reb B
33.438						Sbio

Totals : 1721.43503

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Sample

```

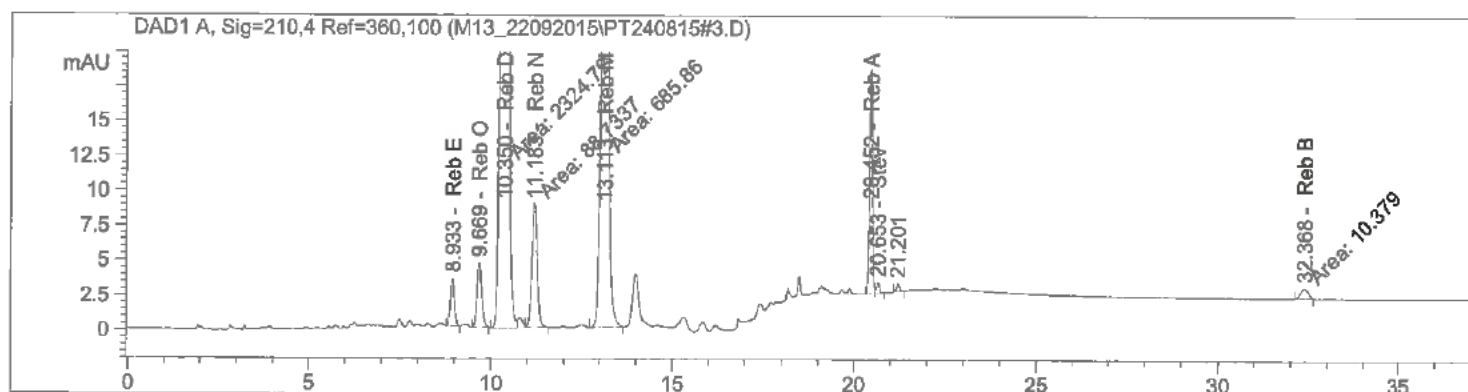
=====
Acq. Operator   : QC Chemist                      Seq. Line :   35
Acq. Instrument : HPLC 3                          Location  : Vial 32
Injection Date  : 9/24/2015 8:12:18 PM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 4:53:56 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT240815, 1832.3171 mg/L
=====

```



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External Standard Report

=====

```

Sorted By           :      Signal
Calib. Data Modified :      Thursday, October 01, 2015 3:35:02 PM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.933	BB	27.19131	5.15213e-1	11.99729		Reb E
9.669	BB	43.62359	5.15213e-1	28.61211		Reb O
10.350	MM	2324.72217	5.15213e-1	1197.72597		Reb D
11.183	MM	88.73373	5.15213e-1	51.62790		Reb N
13.113	MM	685.86035	5.94426e-1	407.69353		Reb M
20.452	BV	87.63667	4.44030e-1	38.91334		Reb A
20.653	VB	4.12977	3.63408e-1	1.50079		Stev
22.243		-	-	-		Reb F
23.023		-	-	-		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.368	MM	10.37904	3.63408e-1	3.77183		Reb B
33.438		-	-	-		Sbio

Totals : 1741.84275

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Sample

```

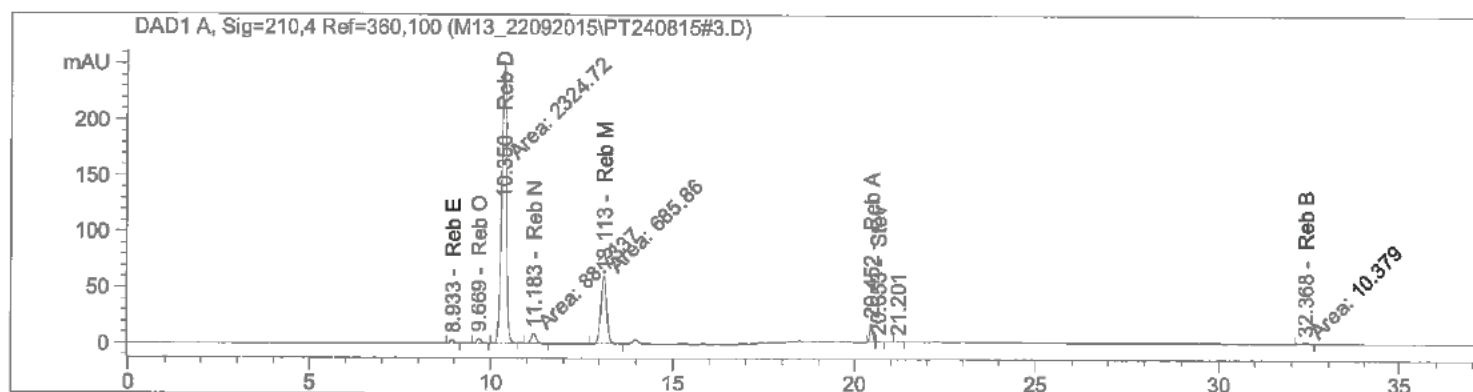
=====
Acq. Operator   : QC Chemist                      Seq. Line :   35
Acq. Instrument : HPLC 3                          Location  : Vial 32
Injection Date  : 9/24/2015 8:12:18 PM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 3:43:04 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info      : PT240815, 1832.3171 mg/L
=====

```



```

=====
External Standard Report
=====

```

```

Sorted By           : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:35:02 PM
Multiplier          : 1.0000
Dilution            : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.933	BB	27.19131	5.15213e-1	11.99729		Reb E
9.669	BB	43.62359	5.15213e-1	28.61211		Reb O
10.350	MM	2324.72217	5.15213e-1	1197.72597		Reb D
11.183	MM	88.73373	5.15213e-1	51.62790		Reb N
13.113	MM	685.86035	5.94426e-1	407.69353		Reb M
20.452	BV	87.63667	4.44030e-1	38.91334		Reb A
20.653	VB	4.12977	3.63408e-1	1.50079		Stev
22.243		-	-	-		Reb F
23.023		-	-	-		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.368	MM	10.37904	3.63408e-1	3.77183		Reb B
33.438						Sbio

Totals : 1741.84275

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

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



Sample Name: Sample

```

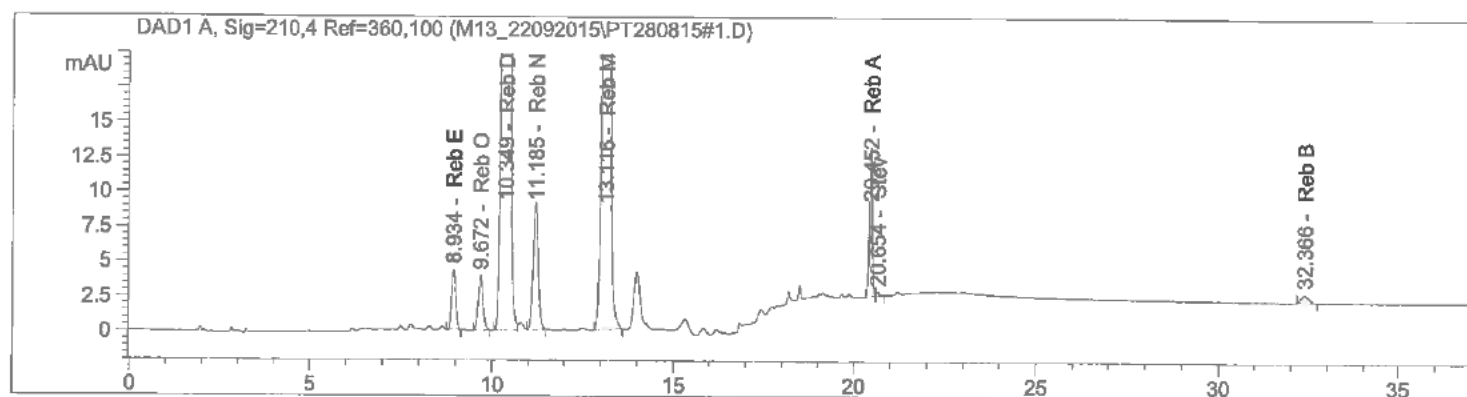
=====
Acq. Operator   : QC Chemist                      Seq. Line :   36
Acq. Instrument : HPLC 3                          Location  : Vial 33
Injection Date  : 9/24/2015 9:15:02 PM            Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 5:20:20 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT280815, 1837.1249 mg/L
=====

```



```

=====
External Standard Report
=====

```

```

Sorted By      : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:35:02 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.934	VB	34.40784	5.15213e-1	15.18135		Reb E
9.672	BB	37.18423	5.15213e-1	24.38862		Reb O
10.349	BV	2259.54810	5.15213e-1	1164.14747		Reb D
11.185	VB	90.14944	5.15213e-1	52.45160		Reb N
13.116	BB	834.12073	5.94426e-1	495.82342		Reb M
20.452	BV	58.11037	4.44030e-1	25.80276		Reb A
20.654	VB	1.72831	3.63408e-1	6.28081e-1		Stev
22.243		-	-	-		Reb F
23.023		-	-	-		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.366	BB	7.47657	3.63408e-1	2.71705		Reb B
33.438						Sbio

Totals : 1781.14035

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Sample

```

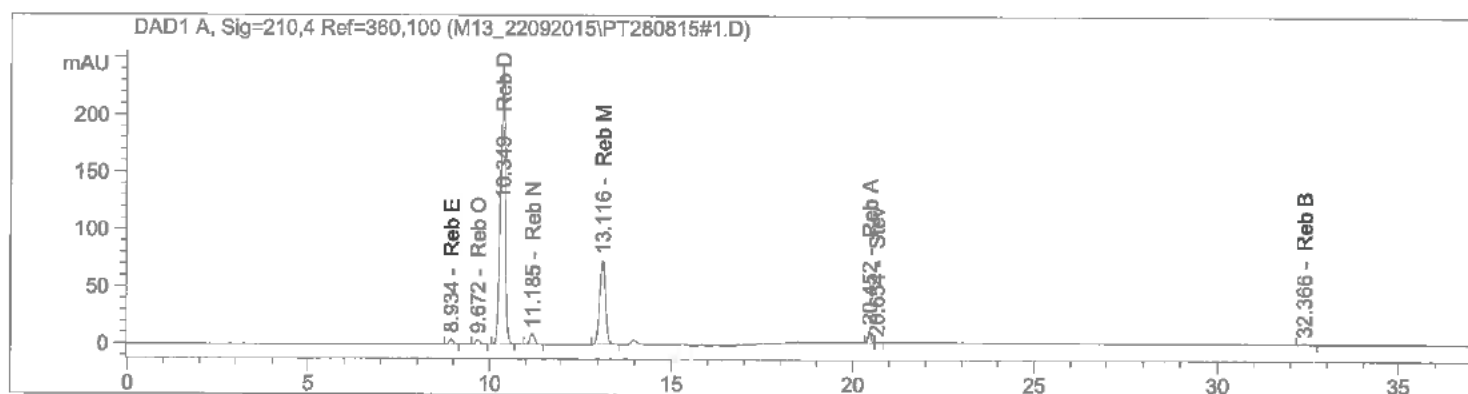
=====
Acq. Operator   : QC Chemist                      Seq. Line :   36
Acq. Instrument : HPLC 3                          Location  : Vial 33
Injection Date  : 9/24/2015 9:15:02 PM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 5:20:44 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT280815, 1837.1249 mg/L
=====

```



```

=====
External Standard Report
=====

```

```

Sorted By           : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:35:02 PM
Multiplier          : 1.0000
Dilution            : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.934	VB	34.40784	5.15213e-1	15.18135		Reb E
9.672	BB	37.18423	5.15213e-1	24.38862		Reb O
10.349	BV	2259.54810	5.15213e-1	1164.14747		Reb D
11.185	VB	90.14944	5.15213e-1	52.45160		Reb N
13.116	BB	834.12073	5.94426e-1	495.82342		Reb M
20.452	BV	58.11037	4.44030e-1	25.80276		Reb A
20.654	VB	1.72831	3.63408e-1	6.28081e-1		Stev
22.243		-	-	-		Reb F
23.023		-	-	-		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.366	BB	7.47657	3.63408e-1	2.71705		Reb B
33.438						Sbio

Totals : 1781.14035

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



DAD1 A, Sig=210,4 Ref=360,100 (M13_22092015\PT280815#2.D)

mAU

15

12.5

10

7.5

5

2.5

0

0 5 10 15 20 25 30 35

8.935 - Reb E

9.674 - Reb Q

10.349 - Reb D

11.188 - Reb N

13.115 - Reb M

20.652 - Reb A

20.651 - Reb A

32.379 - Reb B

Area: 8.92481

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.935	VB	33.96622	5.15213e-1	14.98650	Reb	E
9.674	BB	36.83965	5.15213e-1	24.16262	Reb	O
10.349	BV	2240.83838	5.15213e-1	1154.50799	Reb	D
11.188	VB	88.99573	5.15213e-1	51.78034	Reb	N
13.115	BB	829.31378	5.94426e-1	492.96604	Reb	M
20.451	BV	57.93146	4.44030e-1	25.72332	Reb	A
20.652	VB	1.86047	3.63408e-1	6.76110e-1	Stev	
22.243		-	-	-	Reb	F
23.023		-	-	-	Reb	C
23.689		-	-	-	Dulc.	A
26.584		-	-	-	Rubu	



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.379	MM	8.92481	3.63408e-1	3.24335		Reb B
33.438						Sbio

Totals : 1768.04627

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Sample

```

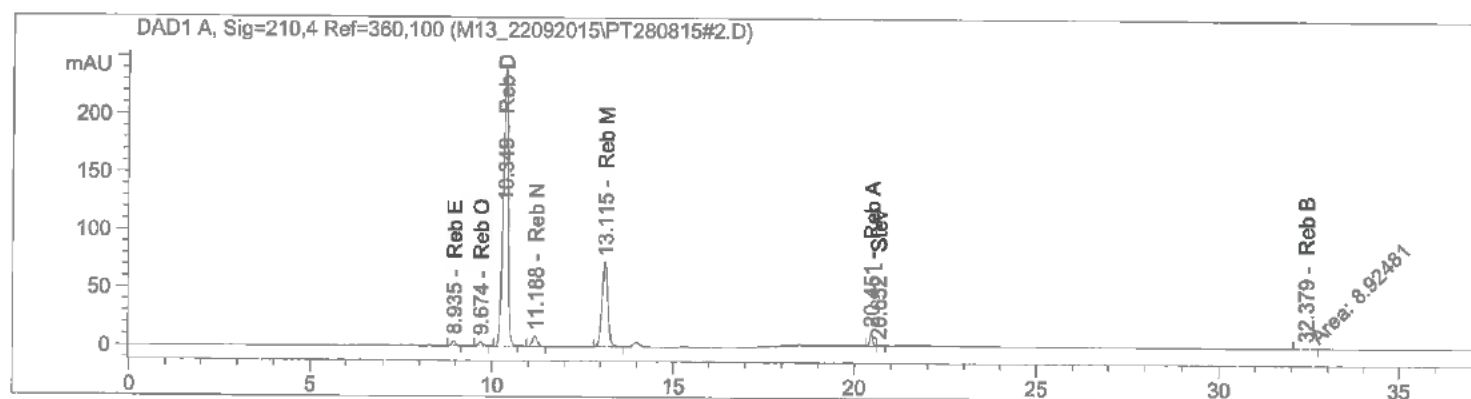
=====
Acq. Operator   : QC Chemist                      Seq. Line :   37
Acq. Instrument : HPLC 3                          Location  : Vial 34
Injection Date  : 9/24/2015 10:18:00 PM           Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 5:21:53 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT280815, 1837.1249 mg/L
=====

```



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External Standard Report

=====

```

Sorted By      : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:35:02 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.935	VB	33.96622	5.15213e-1	14.98650		Reb E
9.674	BB	36.83965	5.15213e-1	24.16262		Reb O
10.349	BV	2240.83838	5.15213e-1	1154.50799		Reb D
11.188	VB	88.99573	5.15213e-1	51.78034		Reb N
13.115	BB	829.31378	5.94426e-1	492.96604		Reb M
20.451	BV	57.93146	4.44030e-1	25.72332		Reb A
20.652	VB	1.86047	3.63408e-1	6.76110e-1		Stev
22.243		-	-	-		Reb F
23.023		-	-	-		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.379	MM	8.92481	3.63408e-1	3.24335		Reb B
33.438						Sbio

Totals : 1768.04627

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Sample

```

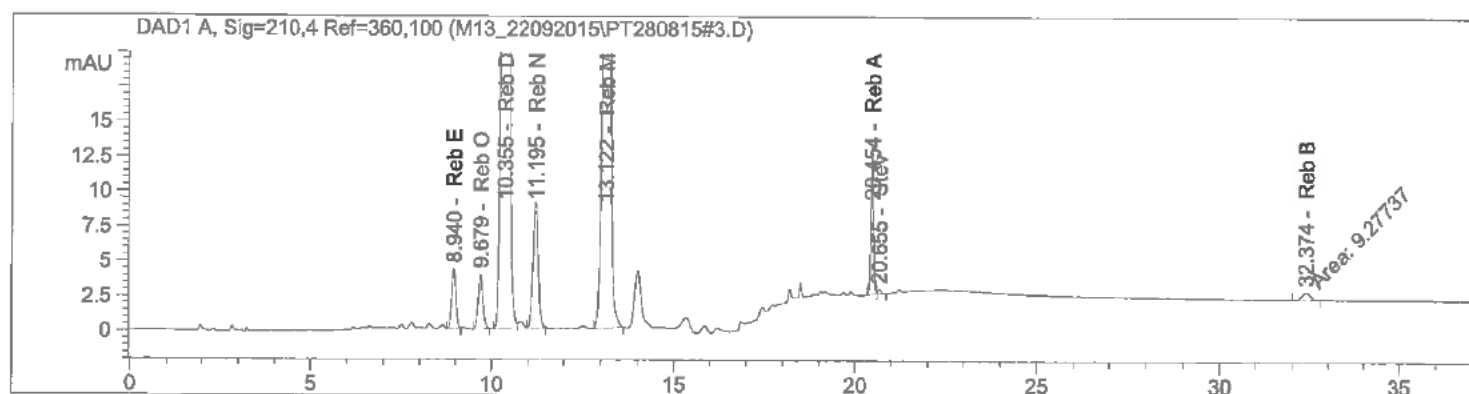
=====
Acq. Operator   : QC Chemist                      Seq. Line :   38
Acq. Instrument : HPLC 3                          Location  : Vial 35
Injection Date  : 9/24/2015 11:20:33 PM           Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 4:53:56 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT280815, 1832.0600 mg/L
=====

```



```

=====
External Standard Report
=====

```

```

Sorted By      : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:35:02 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.940	VB	34.12744	5.15213e-1	15.05763		Reb E
9.679	BB	36.67737	5.15213e-1	24.05618		Reb O
10.355	BB	2242.10645	5.15213e-1	1155.16131		Reb D
11.195	VB	88.97018	5.15213e-1	51.76548		Reb N
13.122	BB	836.05298	5.94426e-1	496.97200		Reb M
20.454	BV	58.00605	4.44030e-1	25.75644		Reb A
20.655	VB	1.69924	3.63408e-1	6.17517e-1		Stev
22.243		-	-	-		Reb F
23.023		-	-	-		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.374	MM	9.27737	3.63408e-1	3.37147		Reb B
33.438						Sbio

Totals : 1772.75803

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Sample

```

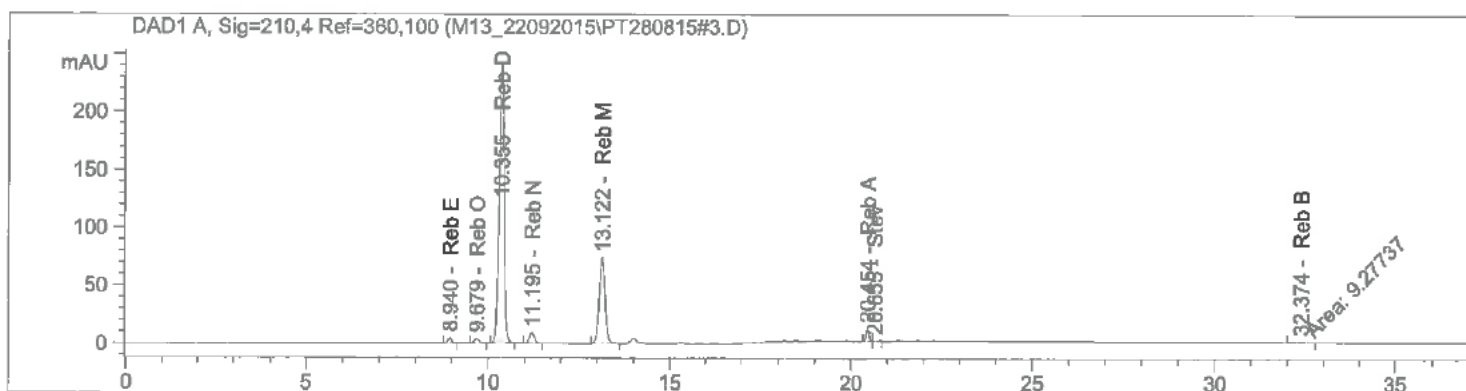
=====
Acq. Operator   : QC Chemist                      Seq. Line :   38
Acq. Instrument : HPLC 3                          Location  : Vial 35
Injection Date  : 9/24/2015 11:20:33 PM           Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 5:19:51 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT280815, 1832.0600 mg/L
=====

```



=====

External Standard Report

=====

```

Sorted By      : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:35:02 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.940	VB	34.12744	5.15213e-1	15.05763		Reb E
9.679	BB	36.67737	5.15213e-1	24.05618		Reb O
10.355	BB	2242.10645	5.15213e-1	1155.16131		Reb D
11.195	VB	88.97018	5.15213e-1	51.76548		Reb N
13.122	BB	836.05298	5.94426e-1	496.97200		Reb M
20.454	BV	58.00605	4.44030e-1	25.75644		Reb A
20.655	VB	1.69924	3.63408e-1	6.17517e-1		Stev
22.243						Reb F
23.023						Reb C
23.689						Dulc.A
26.584						Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.374	MM	9.27737	3.63408e-1	3.37147		Reb B
33.438						Sbio

Totals : 1772.75803

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Sample

```

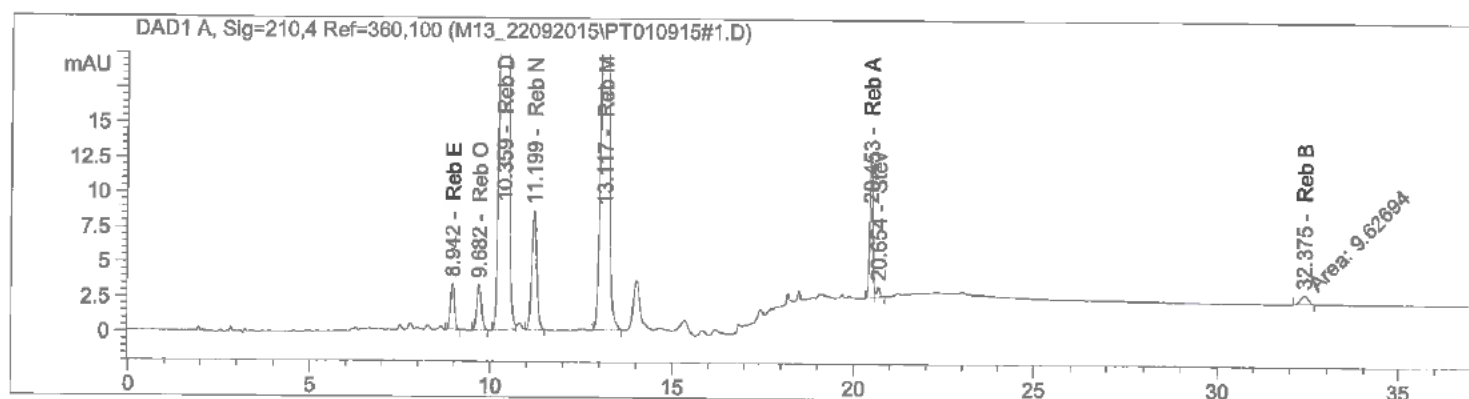
=====
Acq. Operator   : QC Chemist                      Seq. Line :   39
Acq. Instrument : HPLC 3                          Location  : Vial 36
Injection Date  : 9/25/2015 12:23:40 AM           Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 5:22:44 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT010915, 1834.0146 mg/L
=====

```



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External Standard Report

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```

Sorted By      : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:35:02 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.942	VB	26.76102	5.15213e-1	11.80744		Reb E
9.682	BB	30.68524	5.15213e-1	20.12603		Reb O
10.359	BV	2473.97388	5.15213e-1	1274.62231		Reb D
11.199	VB	84.09854	5.15213e-1	48.93101		Reb N
13.117	BB	670.92883	5.94426e-1	398.81784		Reb M
20.453	BV	55.41852	4.44030e-1	24.60750		Reb A
20.654	VB	4.20078	3.63408e-1	1.52660		Stev
22.243		-	-	-		Reb F
23.023		-	-	-		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.375	MM	9.62694	3.63408e-1	3.49851		Reb B
33.438						Sbio

Totals : 1783.93724

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Sample

```

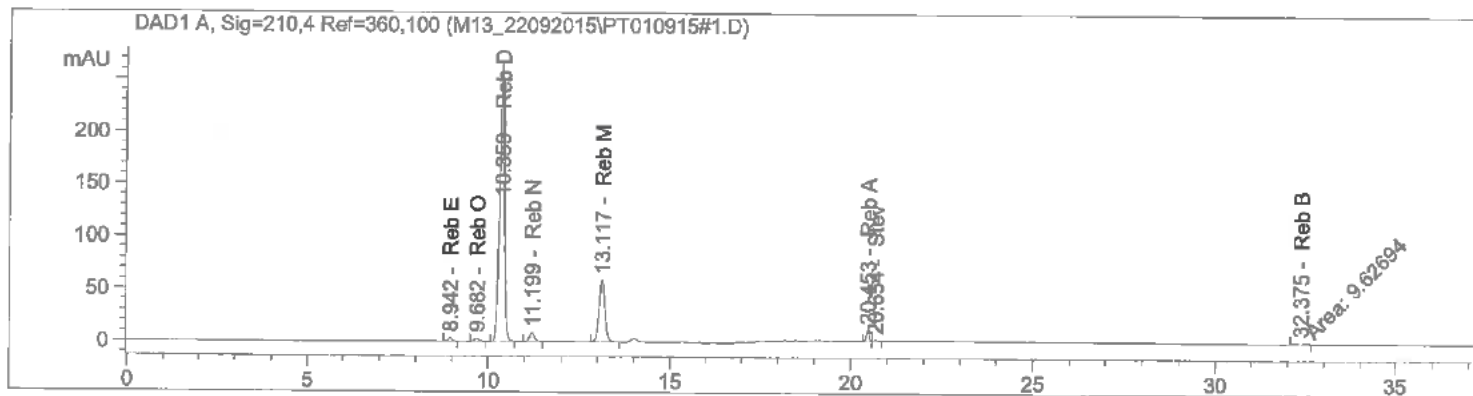
=====
Acq. Operator   : QC Chemist                      Seq. Line :   39
Acq. Instrument : HPLC 3                          Location  : Vial 36
Injection Date  : 9/25/2015 12:23:40 AM           Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 5:23:27 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT010915, 1834.0146 mg/L
=====

```



```

=====
External Standard Report
=====

```

```

Sorted By      : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:35:02 PM
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.942	VB	26.76102	5.15213e-1	11.80744		Reb E
9.682	BB	30.68524	5.15213e-1	20.12603		Reb O
10.359	BV	2473.97388	5.15213e-1	1274.62231		Reb D
11.199	VB	84.09854	5.15213e-1	48.93101		Reb N
13.117	BB	670.92883	5.94426e-1	398.81784		Reb M
20.453	BV	55.41852	4.44030e-1	24.60750		Reb A
20.654	VB	4.20078	3.63408e-1	1.52660		Stev
22.243		-	-	-		Reb F
23.023		-	-	-		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.375	MM	9.62694	3.63408e-1	3.49851		Reb B
33.438						Sbio

Totals : 1783.93724

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Sample

```

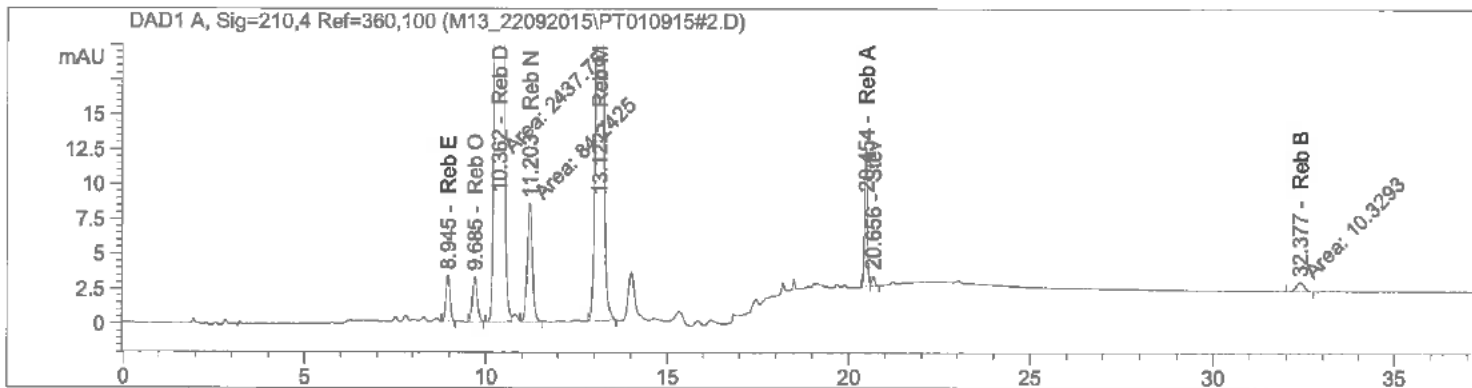
=====
Acq. Operator   : QC Chemist                      Seq. Line :   40
Acq. Instrument : HPLC 3                          Location  : Vial 37
Injection Date  : 9/25/2015 1:26:53 AM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 5:24:42 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT010915, 1820.5479 mg/L
=====

```



```

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External Standard Report
=====

```

```

Sorted By           : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:35:02 PM
Multiplier          : 1.0000
Dilution            : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.945	VB	25.97795	5.15213e-1	11.46193		Reb E
9.685	BB	30.74942	5.15213e-1	20.16812		Reb O
10.362	MM	2437.74878	5.15213e-1	1255.95869		Reb D
11.203	MM	84.24247	5.15213e-1	49.01475		Reb N
13.122	BB	652.04266	5.94426e-1	387.59140		Reb M
20.454	BV	54.56462	4.44030e-1	24.22835		Reb A
20.656	VB	3.94423	3.63408e-1	1.43337		Stev
22.243						Reb F
23.023						Reb C
23.689						Dulc.A
26.584						Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.377	MM	10.32930	3.63408e-1	3.75375		Reb B
33.438						Sbio

Totals : 1753.61036

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

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



Sample Name: Sample

```

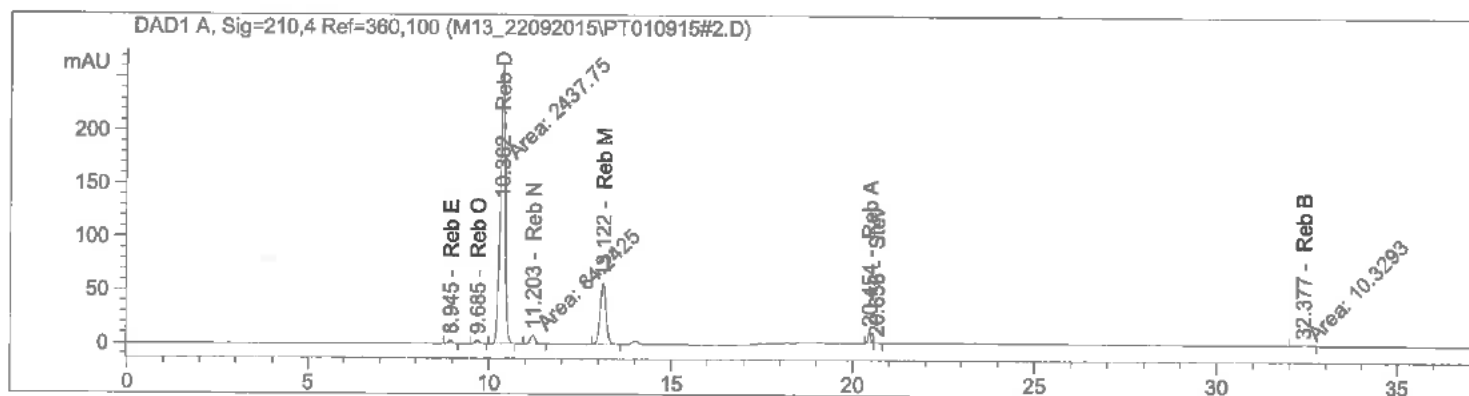
=====
Acq. Operator   : QC Chemist                      Seq. Line :   40
Acq. Instrument : HPLC 3                          Location  : Vial 37
Injection Date  : 9/25/2015 1:26:53 AM            Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 5:27:08 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT010915, 1820.5479 mg/L
=====

```



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External Standard Report

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```

Sorted By      : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:35:02 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.945	VB	25.97795	5.15213e-1	11.46193		Reb E
9.685	BB	30.74942	5.15213e-1	20.16812		Reb O
10.362	MM	2437.74878	5.15213e-1	1255.95869		Reb D
11.203	MM	84.24247	5.15213e-1	49.01475		Reb N
13.122	BB	652.04266	5.94426e-1	387.59140		Reb M
20.454	BV	54.56462	4.44030e-1	24.22835		Reb A
20.656	VB	3.94423	3.63408e-1	1.43337		Stev
22.243		-	-	-		Reb F
23.023		-	-	-		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.377	MM	10.32930	3.63408e-1	3.75375		Reb B
33.438						Sbio

Totals : 1753.61036

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***

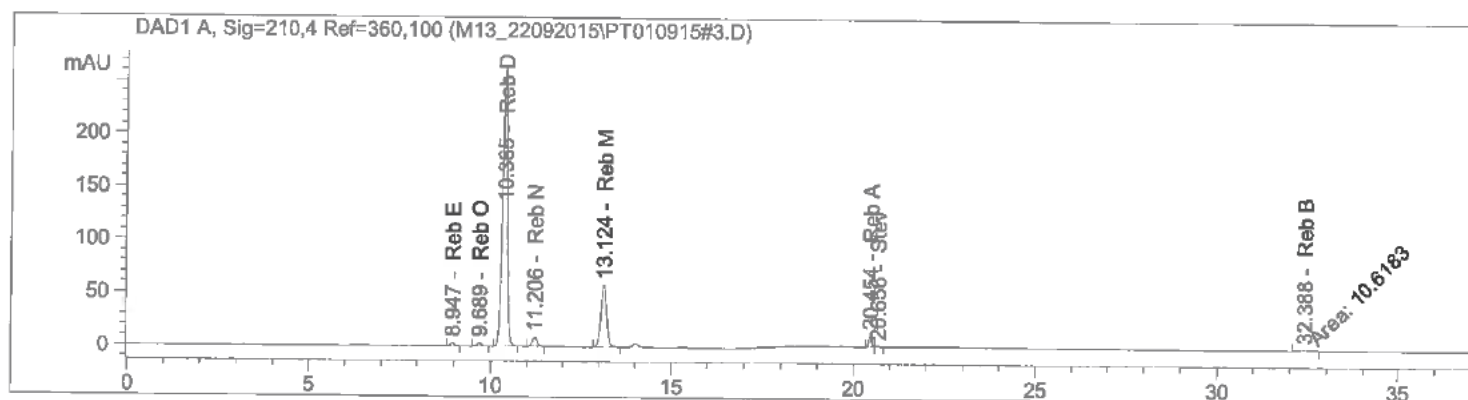


Sample Name: Sample

```

=====
Acq. Operator   : QC Chemist                      Seq. Line :   41
Acq. Instrument : HPLC 3                          Location  : Vial 38
Injection Date  : 9/25/2015 2:29:51 AM           Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 5:28:37 PM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM
Sample Info     : PT010915, 1818.0002 mg/L
=====

```



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External Standard Report

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```

Sorted By      : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:35:02 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.947	BB	26.28018	5.15213e-1	11.59528		Reb E
9.689	BB	30.54177	5.15213e-1	20.03193		Reb O
10.365	BB	2449.03540	5.15213e-1	1261.77370		Reb D
11.206	VB	82.58861	5.15213e-1	48.05249		Reb N
13.124	BB	663.09924	5.94426e-1	394.16373		Reb M
20.454	BV	54.93068	4.44030e-1	24.39088		Reb A
20.656	VB	3.95394	3.63408e-1	1.43690		Stev
22.243		-	-	-		Reb F
23.023		-	-	-		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.388	MM	10.61831	3.63408e-1	3.85878		Reb B
33.438						Sbio

Totals : 1765.30368

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Sample

```

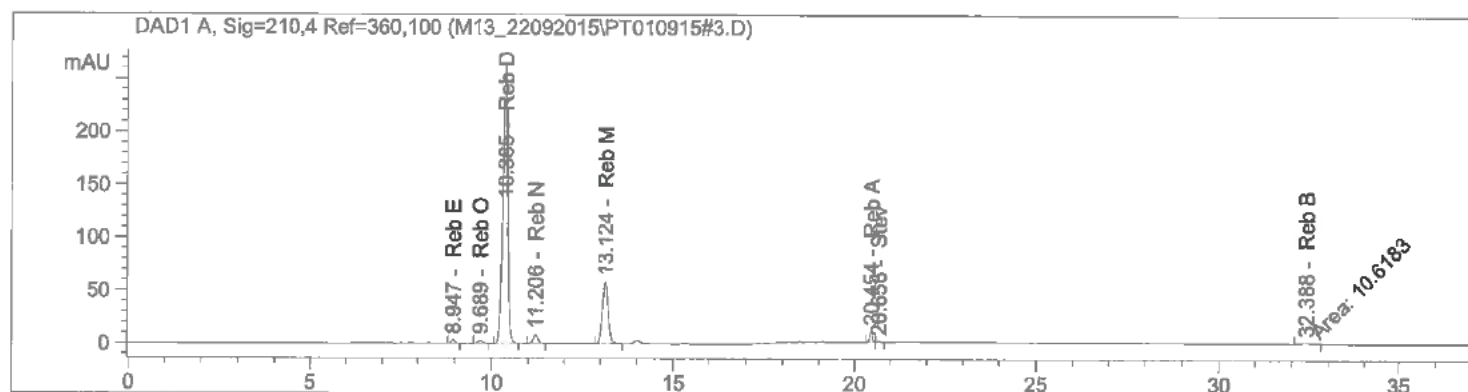
=====
Acq. Operator   : QC Chemist                      Seq. Line :   41
Acq. Instrument : HPLC 3                          Location  : Vial 38
Injection Date  : 9/25/2015 2:29:51 AM            Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 5:28:37 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT010915, 1818.0002 mg/L
=====

```



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External Standard Report

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```

Sorted By      : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:35:02 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.947	BB	26.28018	5.15213e-1	11.59528		Reb E
9.689	BB	30.54177	5.15213e-1	20.03193		Reb O
10.365	BB	2449.03540	5.15213e-1	1261.77370		Reb D
11.206	VB	82.58861	5.15213e-1	48.05249		Reb N
13.124	BB	663.09924	5.94426e-1	394.16373		Reb M
20.454	BV	54.93068	4.44030e-1	24.39088		Reb A
20.656	VB	3.95394	3.63408e-1	1.43690		Stev
22.243		-	-	-		Reb F
23.023		-	-	-		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.388	MM	10.61831	3.63408e-1	3.85878		Reb B
33.438		-	-	-		Sbio

Totals : 1765.30368

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

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

Sample Name: Sample

```

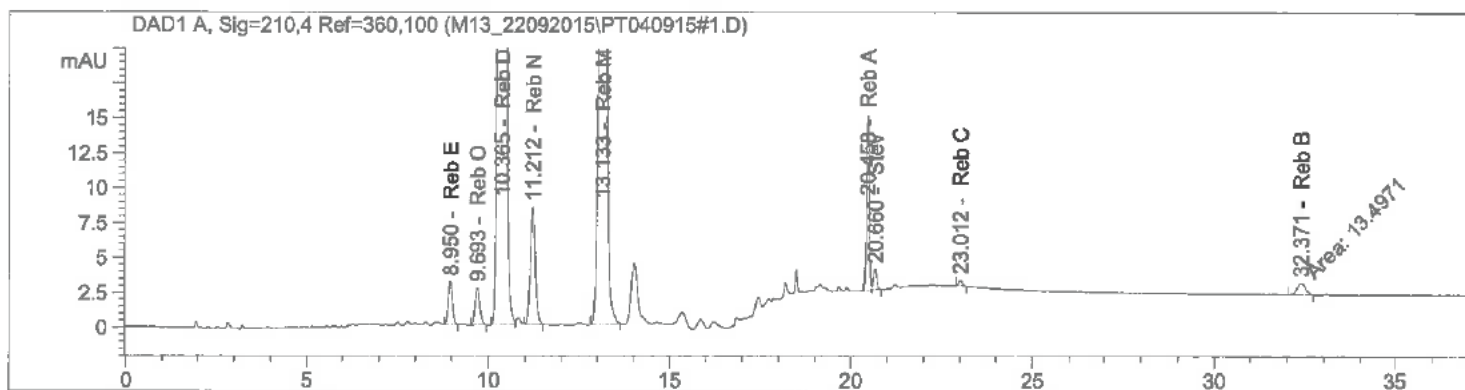
=====
Acq. Operator   : QC Chemist                      Seq. Line :   42
Acq. Instrument : HPLC 3                          Location  : Vial 39
Injection Date  : 9/25/2015 3:32:41 AM             Inj       :    1
                                                    Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 5:29:22 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT040915, 1829.9386 mg/L
=====

```



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External Standard Report
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```

Sorted By           : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:35:02 PM
Multiplier          : 1.0000
Dilution            : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.950	BB	24.61104	5.15213e-1	10.85882		Reb E
9.693	BB	25.17015	5.15213e-1	16.50875		Reb O
10.365	BB	2196.95605	5.15213e-1	1131.89926		Reb D
11.212	VB	82.52480	5.15213e-1	48.01536		Reb N
13.133	BB	846.73395	5.94426e-1	503.32105		Reb M
20.458	BV	68.70069	4.44030e-1	30.50519		Reb A
20.660	VB	8.62599	3.63408e-1	3.13476		Stev
22.243		-	-	-		Reb F
23.012	BB	2.96658	3.63408e-1	1.27385		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.371	MM	13.49709	3.63408e-1	4.90495		Reb B
33.438						Sbio

Totals : 1750.42200

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Sample

```

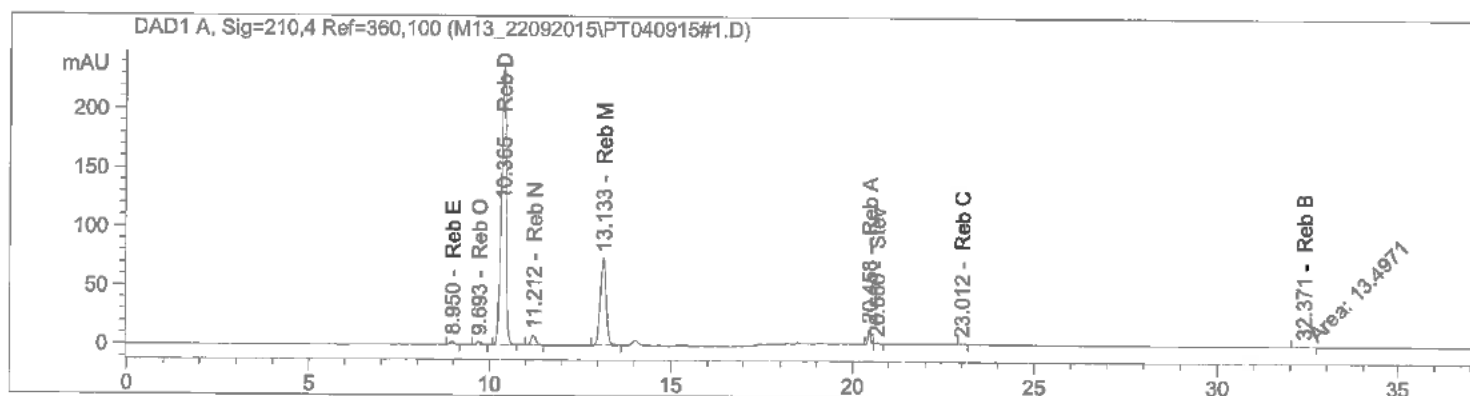
=====
Acq. Operator   : QC Chemist                      Seq. Line :   42
Acq. Instrument : HPLC 3                          Location  : Vial 39
Injection Date  : 9/25/2015 3:32:41 AM             Inj       :    1
                                                    Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMNS.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 5:30:12 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT040915, 1829.9386 mg/L
=====

```



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External Standard Report
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```

Sorted By           : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:35:02 PM
Multiplier          : 1.0000
Dilution            : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.950	BB	24.61104	5.15213e-1	10.85882		Reb E
9.693	BB	25.17015	5.15213e-1	16.50875		Reb O
10.365	BB	2196.95605	5.15213e-1	1131.89926		Reb D
11.212	VB	82.52480	5.15213e-1	48.01536		Reb N
13.133	BB	846.73395	5.94426e-1	503.32105		Reb M
20.458	BV	68.70069	4.44030e-1	30.50519		Reb A
20.660	VB	8.62599	3.63408e-1	3.13476		Stev
22.243						Reb F
23.012	BB	2.96658	3.63408e-1	1.27385		Reb C
23.689						Dulc.A
26.584						Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.371	MM	13.49709	3.63408e-1	4.90495		Reb B
33.438						Sbio

Totals : 1750.42200

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

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



Sample Name: Sample

```

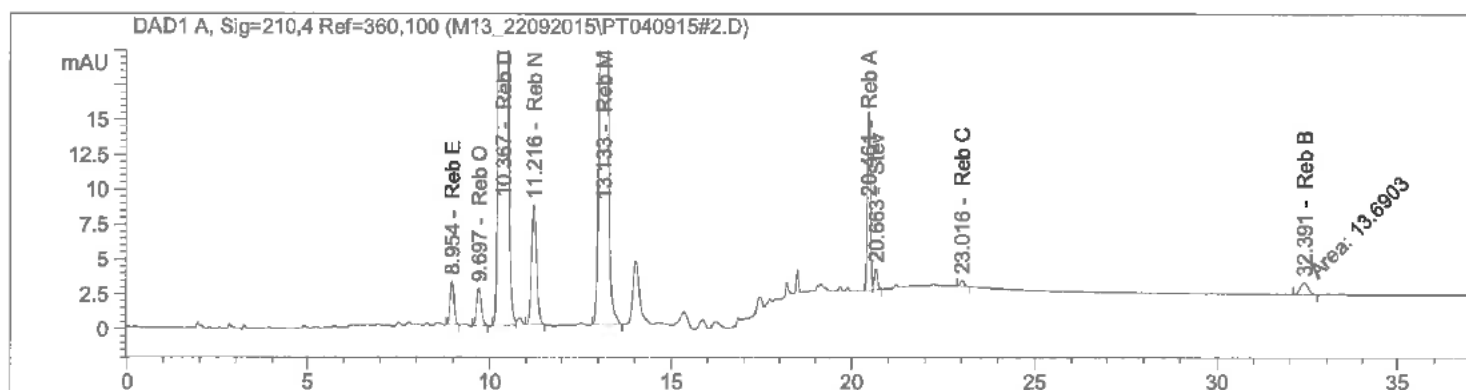
=====
Acq. Operator   : QC Chemist                      Seq. Line :   43
Acq. Instrument : HPLC 3                          Location  : Vial 40
Injection Date  : 9/25/2015 4:35:24 AM            Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 5:31:01 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT040915, 1859.0604 mg/L
=====

```



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External Standard Report
=====

```

```

Sorted By      : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:35:02 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.954	BB	25.26752	5.15213e-1	11.14847		Reb E
9.697	BB	25.77351	5.15213e-1	16.90449		Reb O
10.367	BV	2252.85669	5.15213e-1	1160.69997		Reb D
11.216	VB	84.45052	5.15213e-1	49.13581		Reb N
13.133	BB	856.82947	5.94426e-1	509.32210		Reb M
20.461	BV	70.14787	4.44030e-1	31.14778		Reb A
20.663	VB	8.72415	3.63408e-1	3.17043		Stev
22.243		-	-	-		Reb F
23.016	BB	3.10246	3.63408e-1	1.33220		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.391	MM	13.69034	3.63408e-1	4.97518		Reb B
33.438		-	-	-		Sbio

Totals : 1787.83642

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Sample

```

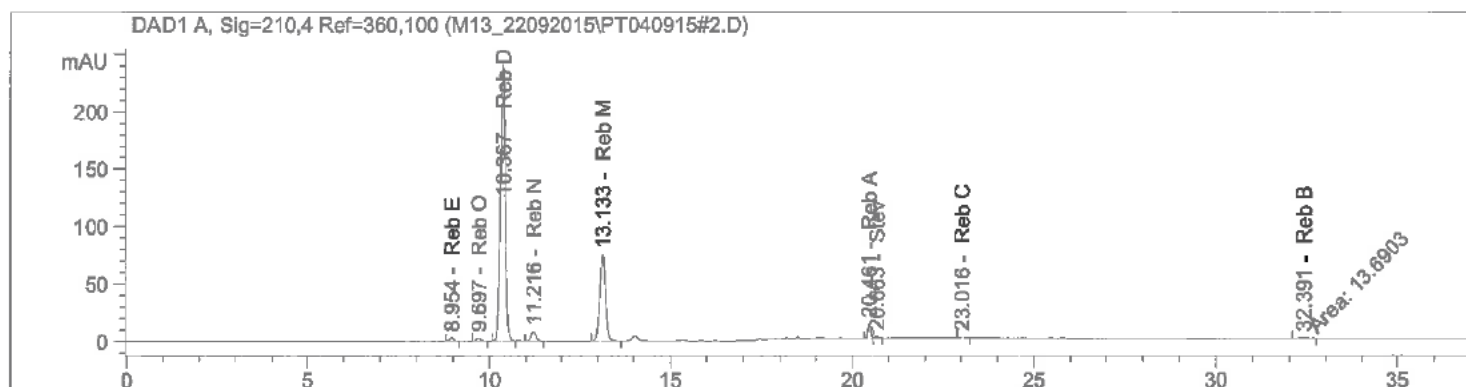
=====
Acq. Operator   : QC Chemist                      Seq. Line :   43
Acq. Instrument : HPLC 3                          Location  : Vial 40
Injection Date  : 9/25/2015 4:35:24 AM            Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 5:31:46 PM by QC Chemist
                  (modified after loading)

Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT040915, 1859.0604 mg/L
=====

```



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External Standard Report

=====

```

Sorted By      : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:35:02 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.954	BB	25.26752	5.15213e-1	11.14847		Reb E
9.697	BB	25.77351	5.15213e-1	16.90449		Reb O
10.367	BV	2252.85669	5.15213e-1	1160.69997		Reb D
11.216	VB	84.45052	5.15213e-1	49.13581		Reb N
13.133	BB	856.82947	5.94426e-1	509.32210		Reb M
20.461	BV	70.14787	4.44030e-1	31.14778		Reb A
20.663	VB	8.72415	3.63408e-1	3.17043		Stev
22.243		-	-	-		Reb F
23.016	BB	3.10246	3.63408e-1	1.33220		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.391	MM	13.69034	3.63408e-1	4.97518		Reb B
33.438						Sbio

Totals : 1787.83642

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Sample

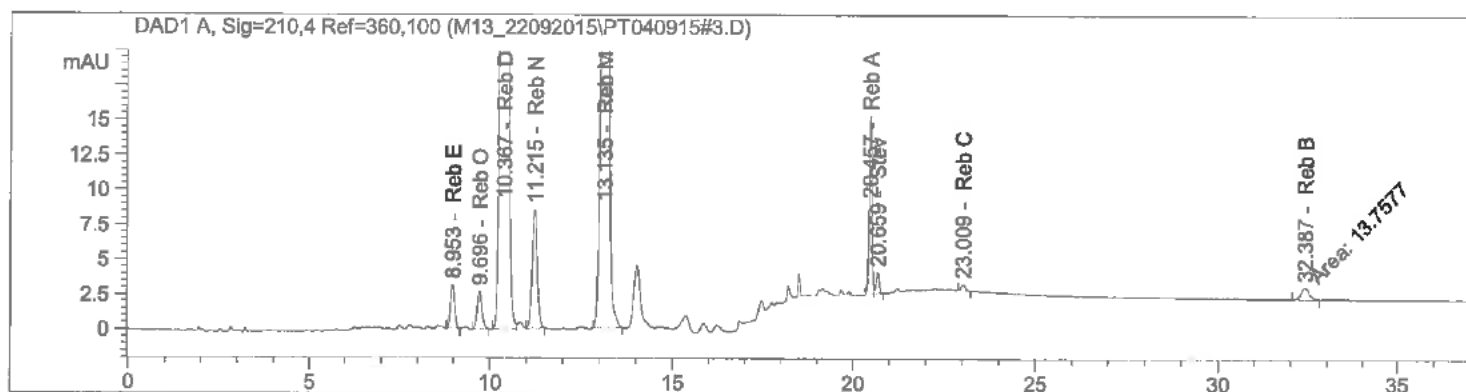
```

=====
Acq. Operator   : QC Chemist                      Seq. Line :   44
Acq. Instrument : HPLC 3                          Location  : Vial 41
Injection Date  : 9/25/2015 5:38:34 AM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 5:32:09 PM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT040915, 1856.5122 mg/L
=====

```



```

=====
External Standard Report
=====

```

```

Sorted By           :      Signal
Calib. Data Modified :      Thursday, October 01, 2015 3:35:02 PM
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.953	BB	24.79751	5.15213e-1	10.94110		Reb E
9.696	BB	25.45218	5.15213e-1	16.69373		Reb O
10.367	BB	2240.02271	5.15213e-1	1154.08774		Reb D
11.215	VB	83.87279	5.15213e-1	48.79966		Reb N
13.135	BB	843.31201	5.94426e-1	501.28696		Reb M
20.457	BV	69.80558	4.44030e-1	30.99579		Reb A
20.659	VB	8.61103	3.63408e-1	3.12932		Stev
22.243		-	-	-		Reb F
23.009	BB	3.03394	3.63408e-1	1.30277		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.387	MM	13.75774	3.63408e-1	4.99968		Reb B
33.438		-	-	-		Sbio

Totals : 1772.23676

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

Warning : Calibrated compound(s) not found

*** End of Report ***



Sample Name: Sample

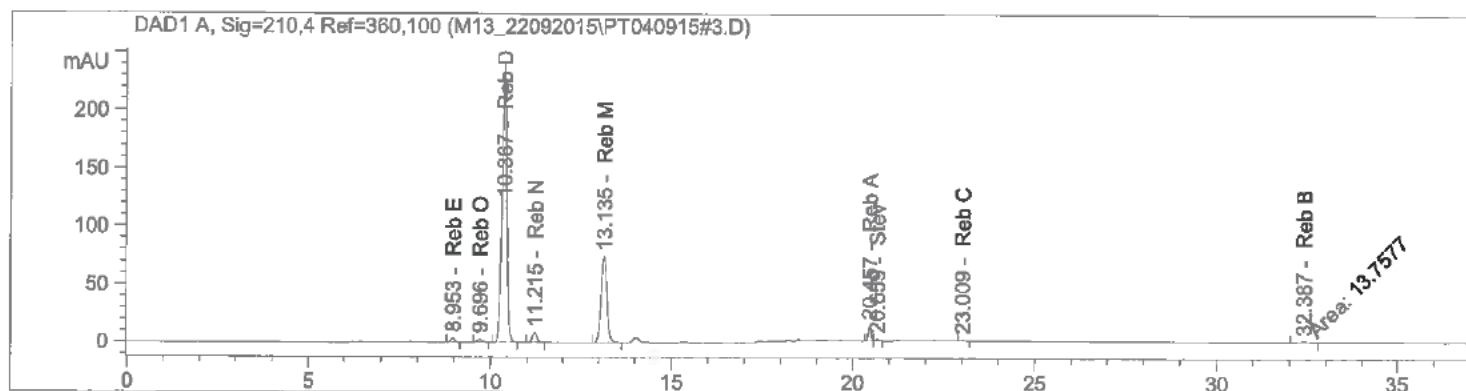
```

=====
Acq. Operator   : QC Chemist                      Seq. Line :   44
Acq. Instrument : HPLC 3                          Location  : Vial 41
Injection Date  : 9/25/2015 5:38:34 AM             Inj       :    1
                                                Inj Volume: 5 µl

Acq. Method     : C:\CHEM32\1\METHODS\C18_GRADIENT-SHORTCOLUMN.M
Last changed    : 9/22/2015 9:46:31 PM by QC Chemist
Analysis Method : C:\CHEM32\1\METHODS\C18_GRADIENT-28092015.M
Last changed    : 10/1/2015 5:33:12 PM by QC Chemist
                  (modified after loading)
Method Info     : New Method: RE,RO,RD,RN,RM

Sample Info     : PT040915, 1856.5122 mg/L
=====

```



```

=====
External Standard Report
=====

```

```

Sorted By      : Signal
Calib. Data Modified : Thursday, October 01, 2015 3:35:02 PM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: DAD1 A, Sig=210,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
8.953	BB	24.79751	5.15213e-1	10.94110		Reb E
9.696	BB	25.45218	5.15213e-1	16.69373		Reb O
10.367	BB	2240.02271	5.15213e-1	1154.08774		Reb D
11.215	VB	83.87279	5.15213e-1	48.79966		Reb N
13.135	BB	843.31201	5.94426e-1	501.28696		Reb M
20.457	EV	69.80558	4.44030e-1	30.99579		Reb A
20.659	VB	8.61103	3.63408e-1	3.12932		Stev
22.243		-	-	-		Reb F
23.009	BB	3.03394	3.63408e-1	1.30277		Reb C
23.689		-	-	-		Dulc.A
26.584		-	-	-		Rubu



Sample Name: Sample

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ng/ul]	Grp	Name
32.387	MM	13.75774	3.63408e-1	4.99968		Reb B
33.438						Sbio

Totals : 1772.23676

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)

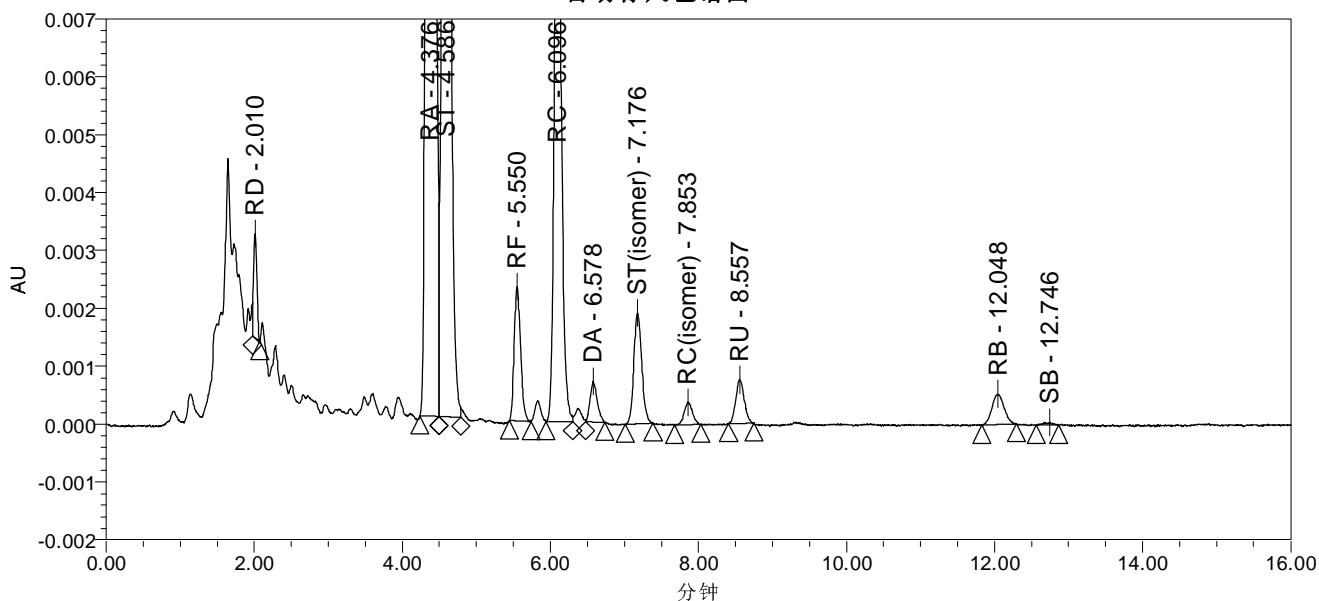
Warning : Calibrated compound(s) not found

*** End of Report ***

样品信息

样品名称:	C1-002-0115-0007-1	采集者:	System
样品类型:	未知	样品组名称:	20150106
瓶号:	92	采集方法组:	JECF_C
进样次数:	1	处理方法:	JECFA 11组份计算
进样体积:	5.00 ul	通道名称:	W2489 ChA
运行时间:	16.0 Minutes	处理通道注释:	W2489 ChA 210nm
采集时间:	2015-1-7 3:18:03 CST	净浓度	1088.00
处理时间:	2015-10-11 8:08:59 CST		

自动标尺色谱图



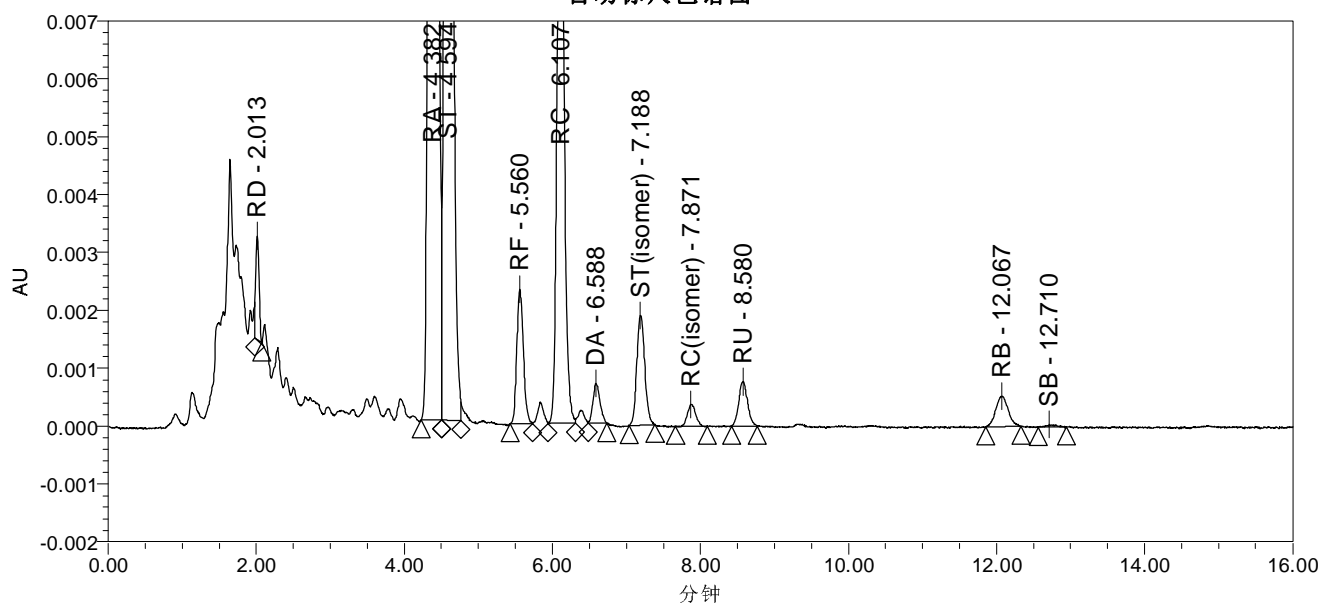
峰结果

	名称	保留时间 (分钟)	面积 (微伏*秒)	%面积	高度 (微伏)	含量	单位	percent
1	RD	2.010	5540	0.57	1803			
2	RA	4.376	561437	57.48	111016	629.341	ppm	57.84
3	ST	4.586	293920	30.09	57109	280.365	ppm	25.77
4	RF	5.550	13711	1.40	2315	15.171	ppm	1.39
5	RC	6.096	67990	6.96	10418	76.528	ppm	7.03
6	DA	6.578	4660	0.48	699	4.356	ppm	0.40
7	ST(isomer)	7.176	14040	1.44	1915	13.610	ppm	1.25
8	RC(isomer)	7.853	2994	0.31	390	3.735	ppm	0.34
9	RU	8.557	6050	0.62	754	4.617	ppm	0.42
10	RB	12.048	5910	0.61	527	5.638	ppm	0.52
11	SB	12.746	469	0.05	55	0.358	ppm	0.03

样品信息

样品名称:	C1-002-0115-0007-3	采集者:	System
样品类型:	未知	样品组名称:	20150106
瓶号:	94	采集方法组:	JECF_C
进样次数:	1	处理方法:	JECFA 11组份计算
进样体积:	5.00 ul	通道名称:	W2489 ChA
运行时间:	16.0 Minutes	处理通道注释:	W2489 ChA 210nm
采集时间:	2015-1-7 3:51:37 CST	净浓度	1091.41
处理时间:	2015-10-11 8:09:06 CST		

自动标尺色谱图



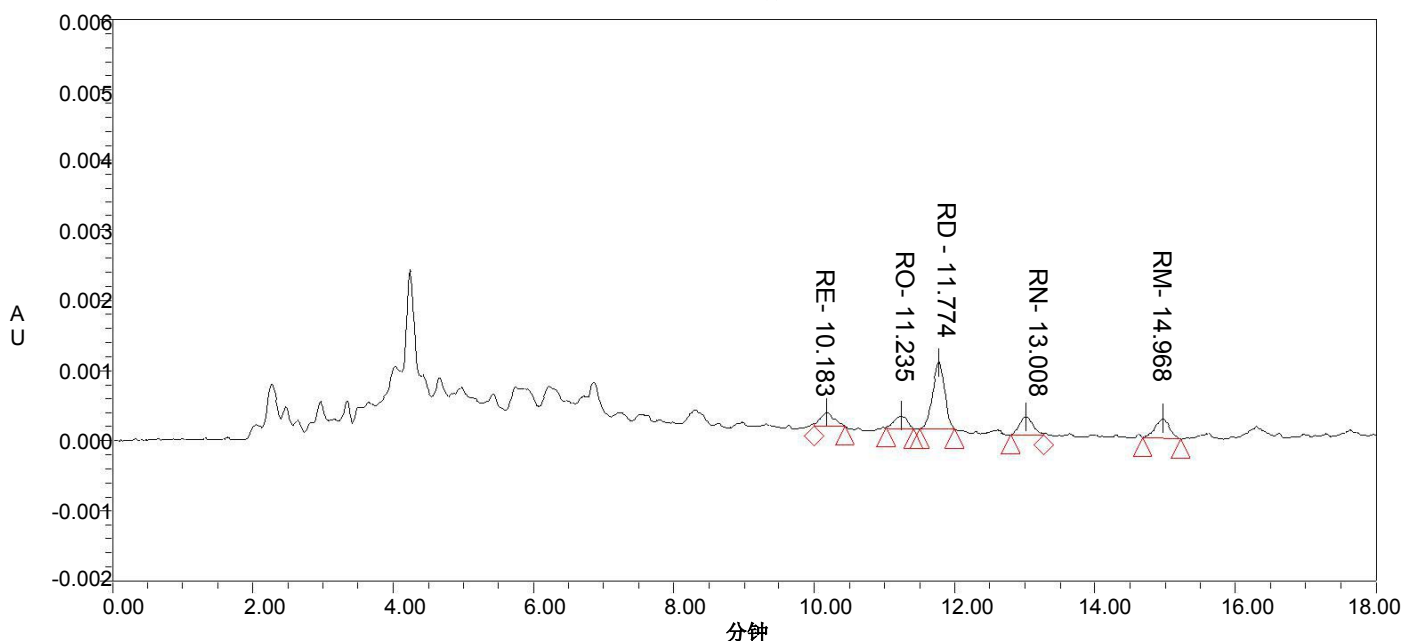
峰结果

	名称	保留时间 (分钟)	面积 (微伏*秒)	%面积	高度 (微伏)	含量	单位	percent
1	RD	2.013	5458	0.56	1794			
2	RA	4.382	563819	57.50	109623	632.012	ppm	57.91
3	ST	4.594	294793	30.06	56478	281.198	ppm	25.76
4	RF	5.560	13986	1.43	2322	15.476	ppm	1.42
5	RC	6.107	68105	6.95	10380	76.658	ppm	7.02
6	DA	6.588	4553	0.46	685	4.256	ppm	0.39
7	ST(isomer)	7.188	14019	1.43	1898	13.590	ppm	1.25
8	RC(isomer)	7.871	2939	0.30	376	3.666	ppm	0.34
9	RU	8.580	6371	0.65	763	4.861	ppm	0.45
10	RB	12.067	6097	0.62	529	5.816	ppm	0.53
11	SB	12.710	459	0.05	44	0.350	ppm	0.03

样品信息

样品名称:	C1-002-0115-0007-1	采集者:	System
样品类型:	未知	样品组名称:	20150617
瓶号:	11	采集方法组:	马来西亚五组分46min
进样次数:	1	处理方法:	POROSHELL 五组分2
进样体积:	5.00 ul	通道名称:	2487 通道 1
运行时间:	46.0 Minutes	处理通道注释:	
采集时间:	2015-6-17 18:21:54 CST	样品浓度:	1106.64 ppm
处理时间:	2015-8-6 14:45:09 CST		

自动标尺色谱图



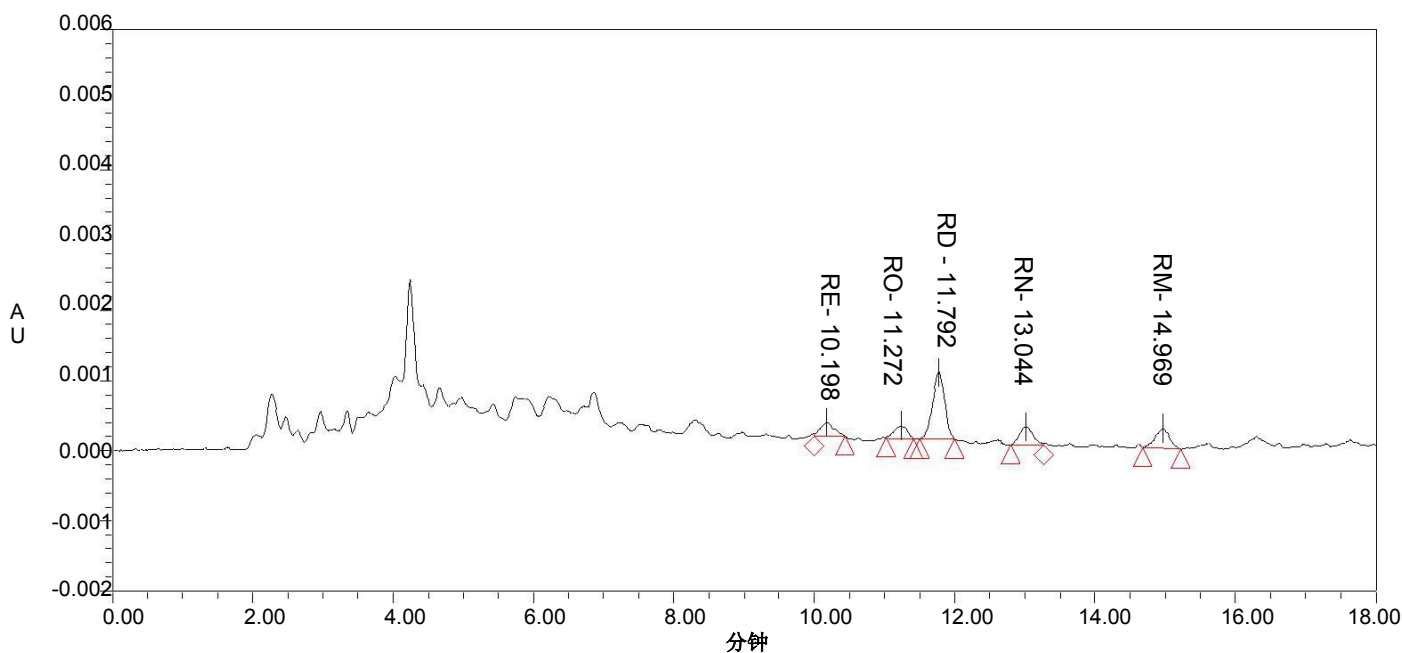
组份结果

	名称	RT (min)	Area (mV * s)	height (mV)	% Area	Con. c	单位	W/w %	峰代码	积分类型
1	RE	10.183	2587	190	11.05	1.27	ppm	0.11	Q20	bb
2	RO	11.235	2232	175	9.53	1.58	ppm	0.14	Q20	bb
3	RD	11.774	11244	947	48.02	6.27	ppm	0.57	Q20	bb
4	RN	13.008	3482	255	14.87	2.19	ppm	0.2	I37 Q20	vb
5	RM	14.968	3870	284	16.53	2.61	ppm	0.24	Q20	bb

样品信息

样品名称:	C1-002-0115-0007-2	采集者:	System
样品类型:	未知	样品组名称:	20150617
瓶号:	12	采集方法组:	马来西亚五组分46min
进样次数:	1	处理方法:	POROSHELL 五组分2
进样体积:	5.00 ul	通道名称:	2487 通道 1
运行时间:	46.0 Minutes	处理通道注释:	
采集时间:	2015-6-17 19:08:51 CST	样品浓度:	1115.93 ppm
处理时间:	2015-8-6 14:45:10 CST		

自动标尺色谱图



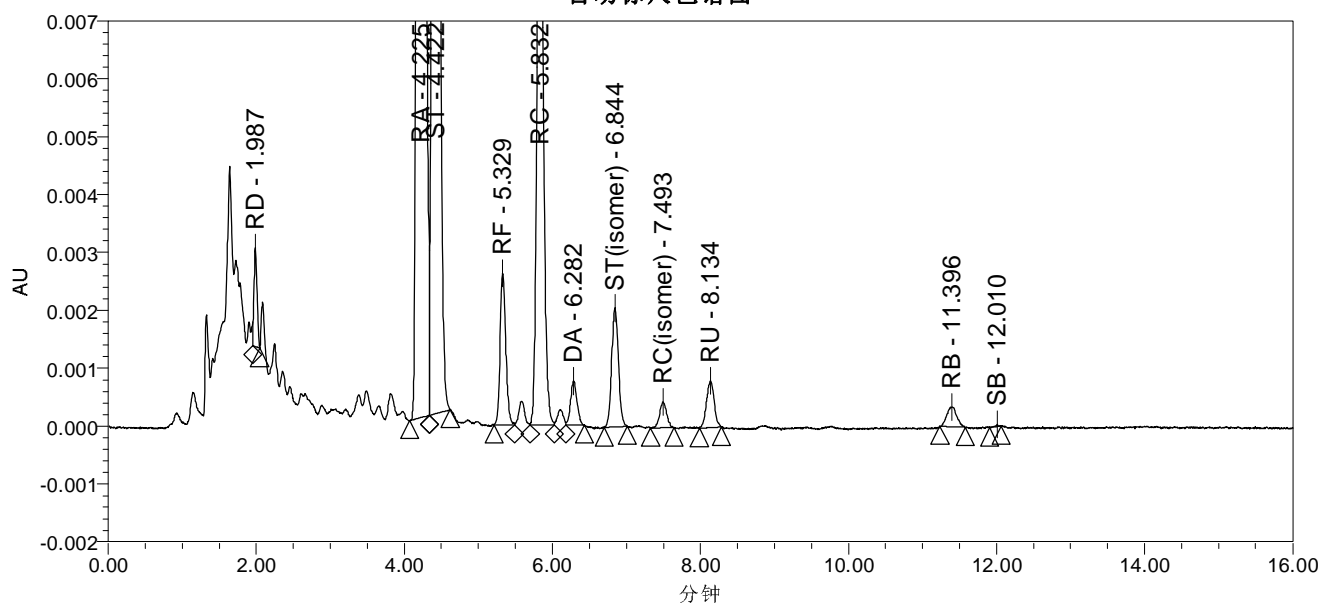
组份结果

	名称	RT (min)	Area (mV * s)	height (mV)	% Area	Con. c	单位	W/w %	峰代码	积分类型
1	RE	10.198	3307	210	13.22	1.62	ppm	0.15	Q20	bb
2	RO	11.272	2445	198	9.77	1.73	ppm	0.16	Q20	bb
3	RD	11.792	11708	968	46.81	6.53	ppm	0.59	Q20	bb
4	RN	13.044	3655	261	14.61	2.3	ppm	0.21	I37 Q20	vb
5	RM	14.969	3900	284	15.59	2.63	ppm	0.24	Q20	bb

样品信息

样品名称:	C1-002-0115-0011-2	采集者:	System
样品类型:	未知	样品组名称:	20150107
瓶号:	77	采集方法组:	JECF_C
进样次数:	1	处理方法:	JECFA 11组份计算
进样体积:	5.00 ul	通道名称:	W2489 ChA
运行时间:	16.0 Minutes	处理通道注释:	W2489 ChA 210nm
采集时间:	2015-1-8 6:08:54 CST	净浓度	1084.19
处理时间:	2015-10-11 8:27:09 CST		

自动标尺色谱图



峰结果

	名称	保留时间 (分钟)	面积 (微伏*秒)	%面积	高度 (微伏)	含量	单位	percent
1	RD	1.987	4871	0.50	1710			
2	RA	4.225	570293	58.97	125882	642.458	ppm	59.26
3	ST	4.422	280652	29.02	61803	269.248	ppm	24.83
4	RF	5.329	13877	1.43	2619	15.443	ppm	1.42
5	RC	5.832	67647	7.00	11801	76.580	ppm	7.06
6	DA	6.282	4531	0.47	764	4.260	ppm	0.39
7	ST(isomer)	6.844	13075	1.35	2053	12.213	ppm	1.13
8	RC(isomer)	7.493	2983	0.31	449	2.972	ppm	0.27
9	RU	8.134	5842	0.60	812	4.484	ppm	0.41
10	RB	11.396	3185	0.33	342	3.056	ppm	0.28
11	SB	12.010	98	0.01	36	0.075	ppm	0.01

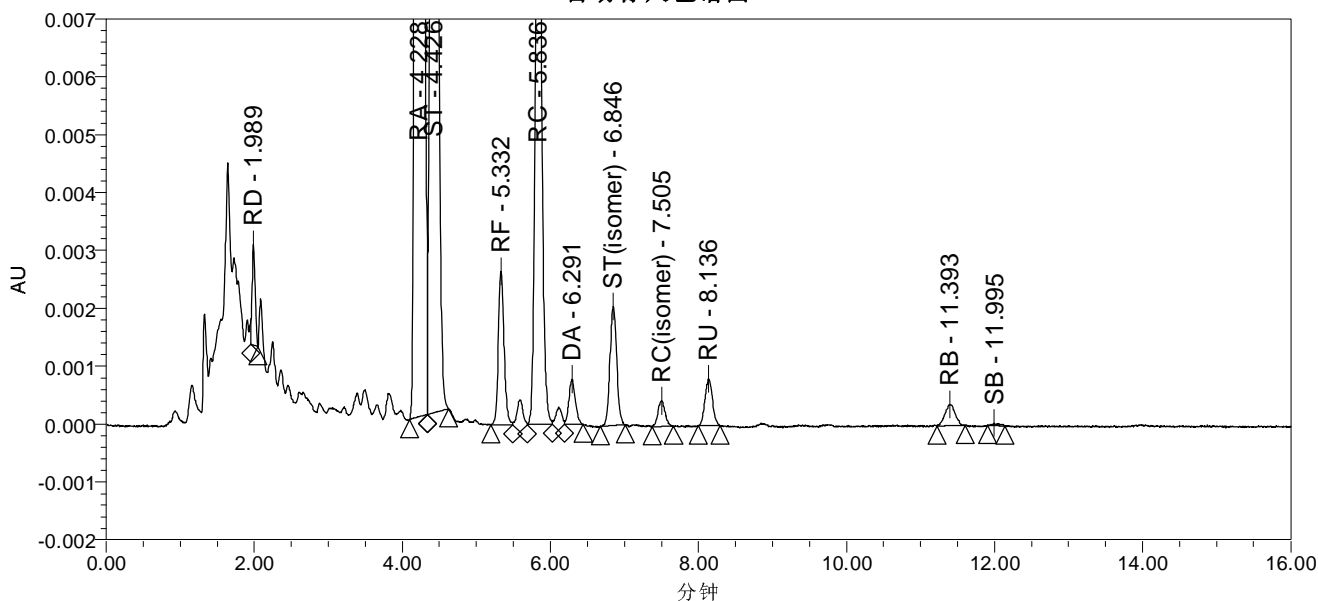
报告用户: System
报告方法: JECFA_S测试
报告方法 ID: 324580
页码: 1 (共计 1)

项目名称: PC2013
打印日期:
2015-10-11
8:41:22 PRC

样品信息

样品名称:	C1-002-0115-0011-3	采集者:	System
样品类型:	未知	样品组名称:	20150107
瓶号:	78	采集方法组:	JECF_C
进样次数:	1	处理方法:	JECFA 11组份计算
进样体积:	5.00 ul	通道名称:	W2489 ChA
运行时间:	16.0 Minutes	处理通道注释:	W2489 ChA 210nm
采集时间:	2015-1-8 6:25:42 CST	净浓度	1092.27
处理时间:	2015-10-11 8:27:19 CST		

自动标尺色谱图



峰结果

	名称	保留时间 (分钟)	面积 (微伏*秒)	%面积	高度 (微伏)	含量	单位	percent
1	RD	1.989	4897	0.50	1737			
2	RA	4.228	572896	58.91	127098	645.392	ppm	59.09
3	ST	4.426	282058	29.00	62394	270.597	ppm	24.77
4	RF	5.332	14106	1.45	2650	15.698	ppm	1.44
5	RC	5.836	68213	7.01	11916	77.221	ppm	7.07
6	DA	6.291	4664	0.48	782	4.385	ppm	0.40
7	ST(isomer)	6.846	13143	1.35	2060	12.276	ppm	1.12
8	RC(isomer)	7.505	2954	0.30	443	2.943	ppm	0.27
9	RU	8.136	5738	0.59	813	4.404	ppm	0.40
10	RB	11.393	3567	0.37	369	3.422	ppm	0.31
11	SB	11.995	324	0.03	44	0.249	ppm	0.02

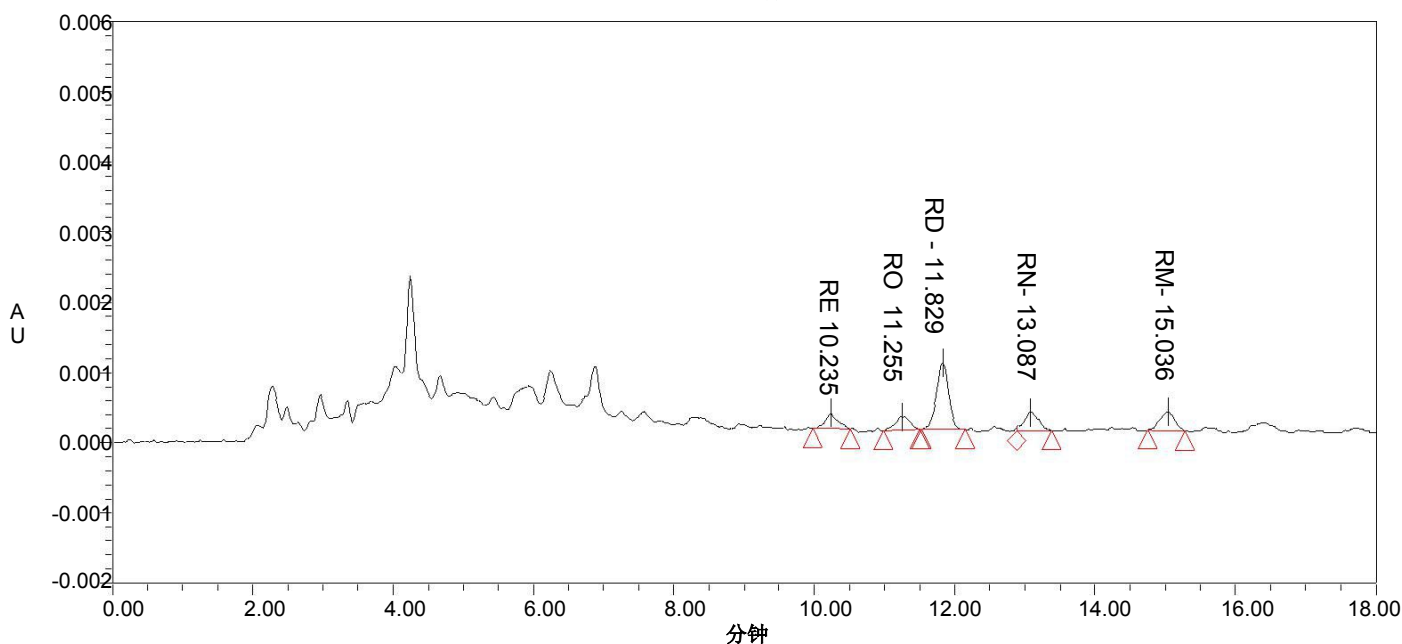
报告用户: System
 报告方法: JECFA_S测试
 报告方法 ID: 324580
 页码: 1 (共计 1)

项目名称: PC2013
 打印日期:
 2015-10-11
 8:41:23 PRC

样品信息

样品名称:	C1-002-0115-0011-1	采集者:	System
样品类型:	未知	样品组名称:	20150617
瓶号:	14	采集方法组:	马来西亚五组分46min
进样次数:	1	处理方法:	POROSHELL 五组分2
进样体积:	5.00 ul	通道名称:	2487 通道 1
运行时间:	46.0 Minutes	处理通道注释:	
采集时间:	2015-6-17 20:42:47 CST	样品浓度:	1115.37 ppm
处理时间:	2015-8-6 14:44:51 CST		

自动标尺色谱图



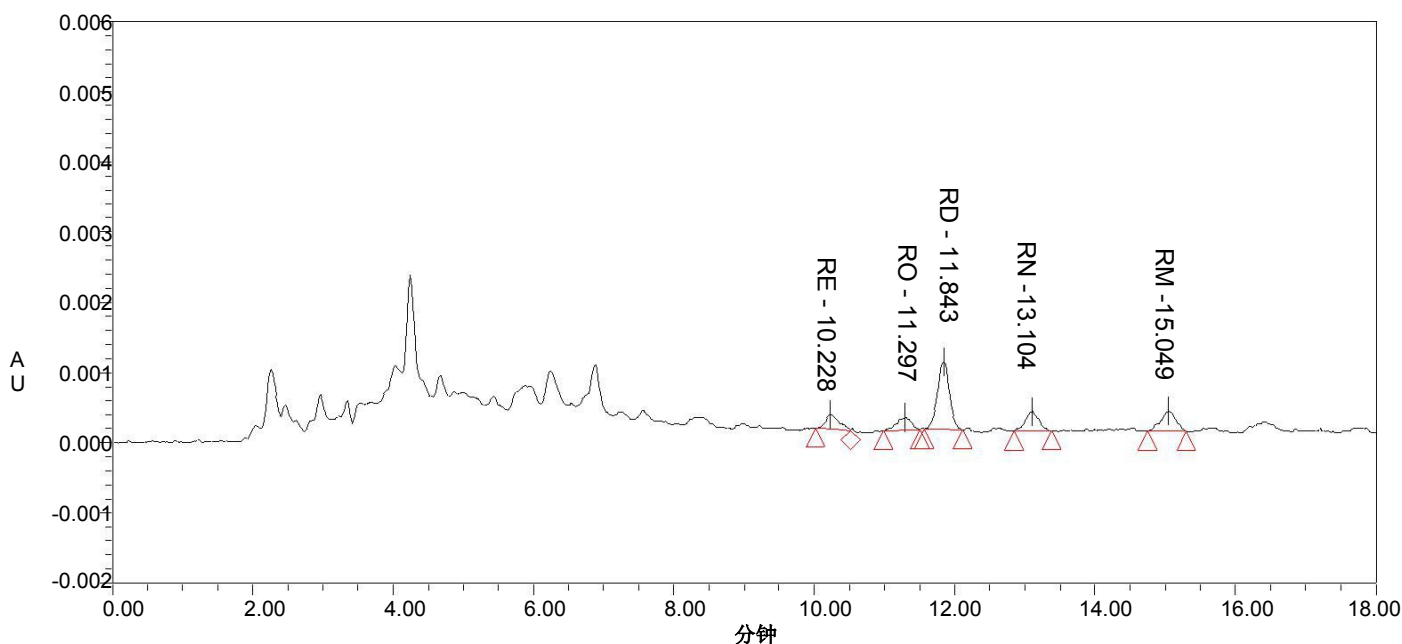
组份结果

	名称	RT (min)	Area (mV * s)	height (mV)	% Area	Con. c	单位	W/w %	峰代码	积分 类型
1	RE	10.235	2612	222	10.70	1.28	ppm	0.11	Q20	bb
2	RO	11.255	2646	183	10.84	1.87	ppm	0.17	Q20	bb
3	RD	11.829	11468	943	46.99	6.4	ppm	0.57	Q20	bb
4	RN	13.087	3781	264	15.49	2.38	ppm	0.21	I37 Q20	vb
5	RM	15.036	3899	266	15.98	2.63	ppm	0.24	Q20	bb

样品信息

样品名称:	C1-002-0115-0011-2	采集者:	System
样品类型:	未知	样品组名称:	20150617
瓶号:	15	采集方法组:	马来西亚五组分46min
进样次数:	1	处理方法:	POROSHELL 五组分2
进样体积:	5.00 ul	通道名称:	2487 通道 1
运行时间:	46.0 Minutes	处理通道注释:	
采集时间:	2015-6-17 21:29:45 CST	样品浓度:	1109.67 ppm
处理时间:	2015-8-6 14:45:04 CST		

自动标尺色谱图



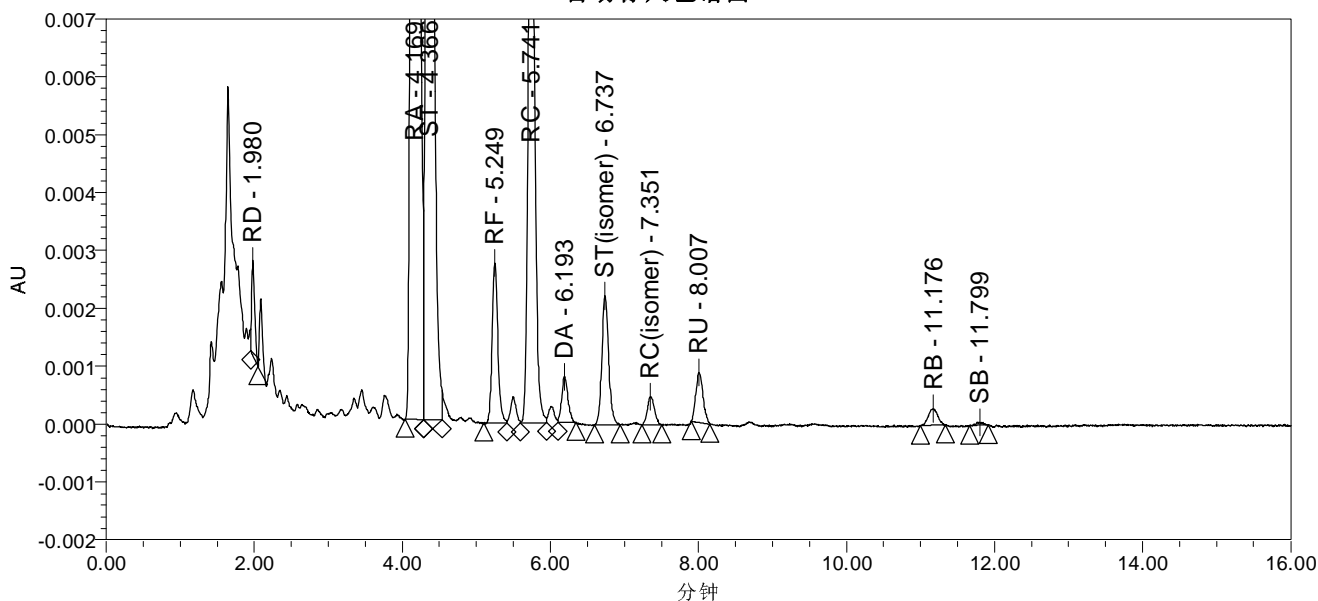
组份结果

	名称	RT (min)	Area (mV * s)	height (mV)	% Area	Con. c	单位	W/w %	峰代码	积分类型
1	RE	10.228	2737	208	11.15	1.34	ppm	0.12	Q20	bb
2	RO	11.297	2514	173	10.24	1.78	ppm	0.16	Q20	bb
3	RD	11.843	11438	961	46.60	6.38	ppm	0.57	Q20	bb
4	RN	13.104	3637	267	14.82	2.29	ppm	0.21	I37 Q20	vb
5	RM	15.049	4219	280	17.19	2.85	ppm	0.26	Q20	bb

样品信息

样品名称:	C1-002-0115-0026-1	采集者:	System
样品类型:	未知	样品组名称:	20150121
瓶号:	92	采集方法组:	JECF_C
进样次数:	1	处理方法:	JECFA 11组份计算
进样体积:	5.00 ul	通道名称:	W2489 ChA
运行时间:	16.0 Minutes	处理通道注释:	W2489 ChA 210nm
采集时间:	2015-1-22 6:34:36 CST	净浓度	1127.77
处理时间:	2015-10-11 8:29:20 CST		

自动标尺色谱图



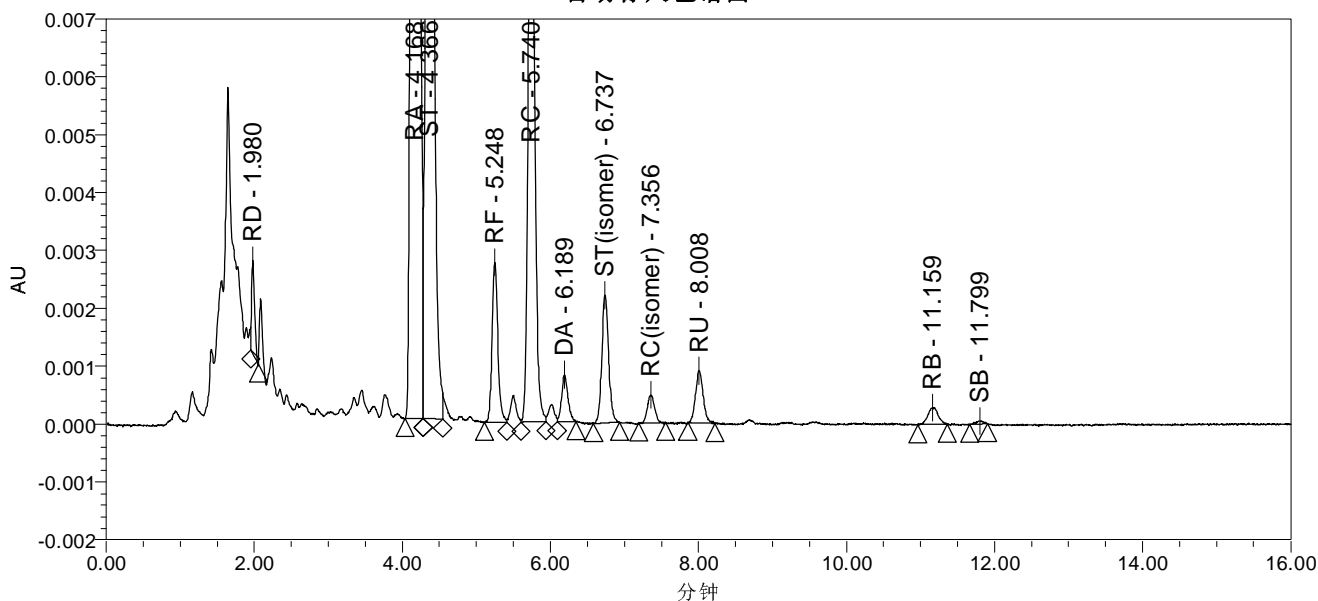
峰结果

	名称	保留时间 (分钟)	面积 (微伏*秒)	%面积	高度 (微伏)	含量	单位	percent
1	RD	1.980	4716	0.48	1641			
2	RA	4.169	577224	58.32	127967	663.410	PPM	58.82
3	ST	4.366	293019	29.61	63979	284.788	ppm	25.25
4	RF	5.249	14667	1.48	2767	16.535	ppm	1.47
5	RC	5.741	69191	6.99	12176	79.352	ppm	7.04
6	DA	6.193	4746	0.48	793	4.520	ppm	0.40
7	ST(isomer)	6.737	14177	1.43	2225	13.519	ppm	1.20
8	RC(isomer)	7.351	3165	0.32	478	3.539	ppm	0.31
9	RU	8.007	5908	0.60	867	4.593	ppm	0.41
10	RB	11.176	2526	0.26	290	2.455	ppm	0.22
11	SB	11.799	350	0.04	56	0.272	ppm	0.02

样品信息

样品名称:	C1-002-0115-0026-2	采集者:	System
样品类型:	未知	样品组名称:	20150121
瓶号:	93	采集方法组:	JECF_C
进样次数:	1	处理方法:	JECFA 11组份计算
进样体积:	5.00 ul	通道名称:	W2489 ChA
运行时间:	16.0 Minutes	处理通道注释:	W2489 ChA 210nm
采集时间:	2015-1-22 6:51:23 CST	净浓度	1122.76
处理时间:	2015-10-11 8:29:33 CST		

自动标尺色谱图



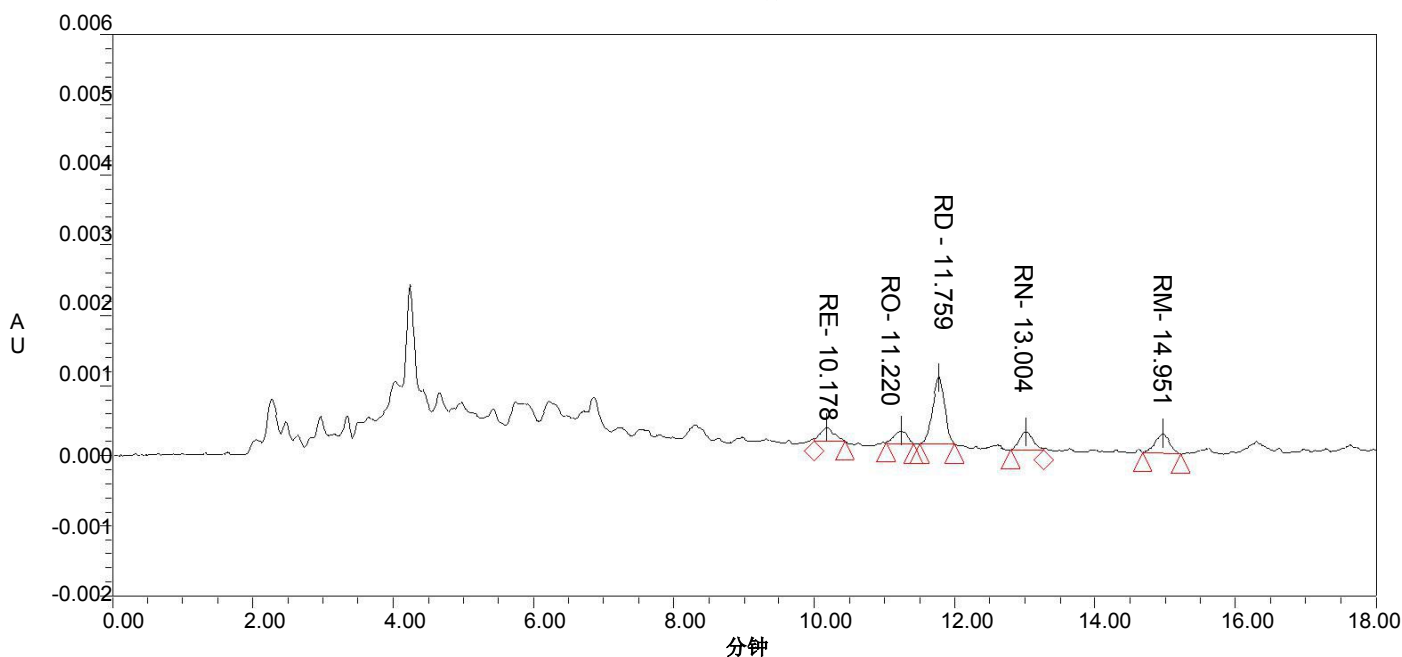
峰结果

	名称	保留时间 (分钟)	面积 (微伏*秒)	%面积	高度 (微伏)	含量	单位	percent
1	RD	1.980	4568	0.46	1614			
2	RA	4.168	572486	58.19	127039	657.964	PPM	58.60
3	ST	4.366	291454	29.62	63512	283.268	ppm	25.23
4	RF	5.248	14705	1.49	2764	16.579	ppm	1.48
5	RC	5.740	68803	6.99	12094	78.907	ppm	7.03
6	DA	6.189	4887	0.50	814	4.655	ppm	0.41
7	ST(isomer)	6.737	14067	1.43	2208	13.415	ppm	1.19
8	RC(isomer)	7.356	3237	0.33	484	3.620	ppm	0.32
9	RU	8.008	6527	0.66	900	5.075	ppm	0.45
10	RB	11.159	2805	0.29	290	2.726	ppm	0.24
11	SB	11.799	339	0.03	49	0.263	ppm	0.02

样品信息

样品名称:	C1-002-0115-0026-3	采集者:	System
样品类型:	未知	样品组名称:	20150617
瓶号:	10	采集方法组:	马来西亚五组分46min
进样次数:	1	处理方法:	POROSHELL 五组分2
进样体积:	5.00 ul	通道名称:	2487 通道 1
运行时间:	46.0 Minutes	处理通道注释:	
采集时间:	2015-6-17 17:34:56 CST	样品浓度:	1111.67 ppm
处理时间:	2015-8-6 14:45:14 CST		

自动标尺色谱图



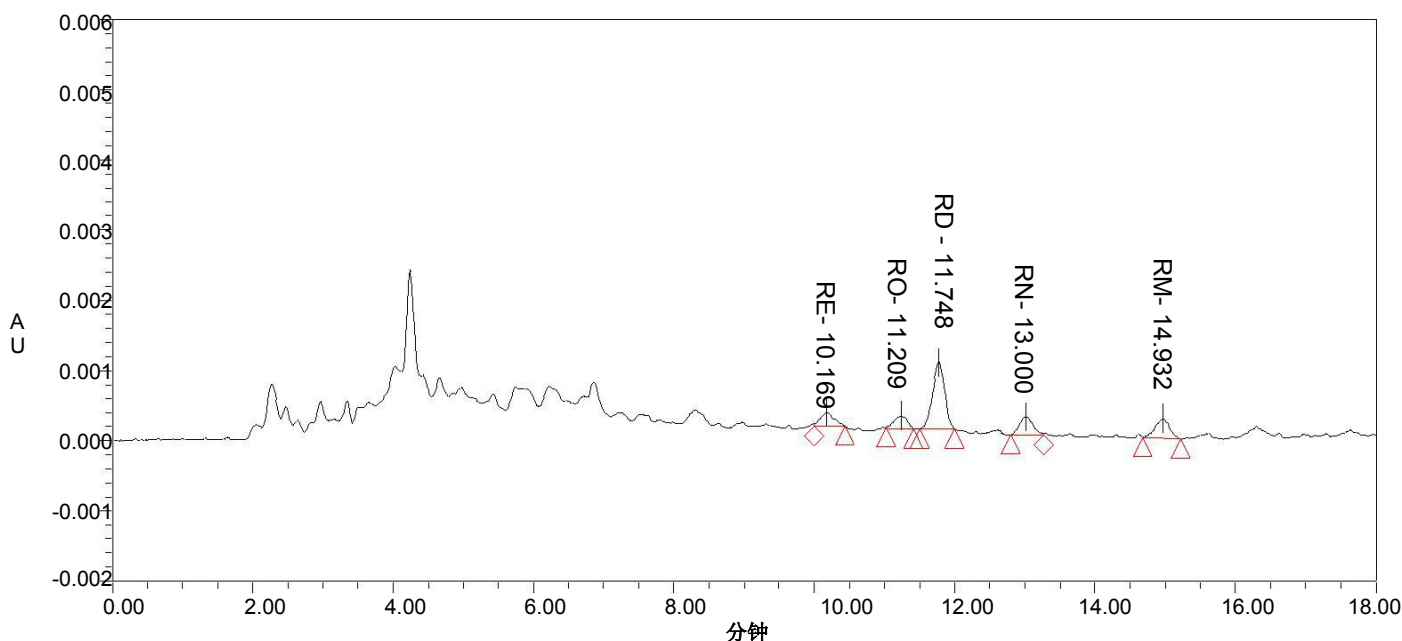
组份结果

	名称	RT (min)	Area (mV * s)	height (mV)	% Area	Con. c	单位	W/w %	峰代码	积分类型
1	RE	10.178	2194	202	9.56	1.08	ppm	0.10	Q20	bb
2	RO	11.220	2661	196	11.59	1.88	ppm	0.17	I37 Q20	bv
3	RD	11.759	11138	949	48.52	6.21	ppm	0.56	I37 Q20	vv
4	RN	13.004	3479	283	15.15	2.19	ppm	0.2	Q20	bb
5	RM	14.951	3485	233	15.18	2.35	ppm	0.21	Q20	bb

样品信息

样品名称:	C1-002-0115-0026-2	采集者:	System
样品类型:	未知	样品组名称:	20150617
瓶号:	9	采集方法组:	马来西亚五组分46min
进样次数:	1	处理方法:	POROSHELL 五组分2
进样体积:	5.00 ul	通道名称:	2487 通道 1
运行时间:	46.0 Minutes	处理通道注释:	
采集时间:	2015-6-17 16:47:56 CST	样品浓度:	1099.81 ppm
处理时间:	2015-8-6 14:45:14 CST		

自动标尺色谱图



组份结果

	名称	RT (min)	Area (mV * s)	height (mV)	% Area	Con. c	单位	W/w %	峰代码	积分类型
1	RE	10.169	2272	207	10.16	1.12	ppm	0.10	Q20	bb
2	RO	11.209	2650	199	11.85	1.88	ppm	0.17	I37 Q20	bv
3	RD	11.748	11194	939	50.06	6.24	ppm	0.57	I37 Q20	vv
4	RN	13.000	3256	258	14.56	2.05	ppm	0.19	Q20	bb
5	RM	14.932	2990	222	13.37	2.02	ppm	0.18	Q20	bb

HPLC methodology for major and minor components in stevia leaf extract

METHOD 1

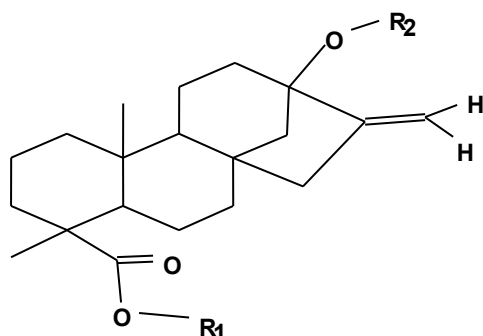
1. Method Summary

This HPLC method is applicable to the quantitation of Reb A & Stevioside and its major impurities, based on stevioside standards, for setting specifications and confirming adequate purity for bulk release.

The analytes are separated using an Agilent Poroshell 120 SB- C18, 4.6 mm x 150 mm, 2.7 μ m at 40 °C, as per JECFA 2010 method. The mobile phase consists of 68% 10mmol/L phosphate buffer (pH2.6) and 32% acetonitrile containing. Reb A, stevioside and the other steviol glycosides are detected by ultraviolet absorption (UV) at 210. Stevia extract samples are prepared in an acetonitrile-water solution at 1200~1300 mg/L. Quantitation of stevioside and the other steviol glycosides is performed using a Four-point linear regression curve fit of stevioside standards. The stevioside standards are prepared at concentrations of approximately 200, 300, 400 and 500 mg/L (corrected for moisture and purity). Quantitation of Rebaudioside A is performed using a four-point linear regression curve fit of standards prepared at approximately 400, 500, 600 and 700 mg/L (corrected for moisture and purity)

2. Structures

Structure of Stevia sweeteners hydrocarbon backbone.



Formula, and R-Groups in Backbone Structure of Identified Steviol Glycosides

Common Name	Symbol	Trivial Formula	R ₁	R ₂	Reference	Molecular Weight (g/mol)
Rubusoside	RUBU	SvG2	Glc β 1-	Glc β 1-	4a	642.74
Steviolbioside	SBIO	SvG2	H	Glc β (1-2)Glc β 1-	7	642.75
Stevioside	STEV	SvG3	Glc β 1-	Glc β (1-2)Glc β 1-	8	804.89
Stevioside A	STEVA	SvG3	Glc β (1-2)Glc β 1-	Glc β 1-	9	804.89
Rebaudioside B	REB B	SvG3	H	Glc β (1-2)[Glc β (1-3)]Glc β 1-	7	804.89
Rebaudioside A	REB A	SvG4	Glc β 1-	Glc β (1-2)[Glc β (1-3)]Glc β 1-	7	967.03
Rebaudioside D	REB D	SvG5	Glc β (1-2)Glc β 1-	Glc β (1-2)[Glc β (1-3)]Glc β 1-	11	1129.18
Rebaudioside F	REB F	SvX1G3	Glc β 1-	Xyl β (1-2)[Glc β (1-3)]Glc β 1-	17	936.00
Dulcoside A	DUL A	SvR1G2	Glc β 1-	Rha α (1-2)Glc β 1-	15	788.89
Rebaudioside C	REB C	SvR1G3	Glc β 1-	Rha α (1-2)[Glc β (1-3)]Glc β 1-	16	951.04
Rebaudioside C(isomer)	REB CI	SvR1G3	Rha α (1-2)Glc β 1-	Glc β (1-3)Glc β 1-	NA	951.04

*rha = rhamnose, glc = glucose, xyl = xylose

3. Experimental

3.1. Reagents and Chemicals

- 3.1.1. Rebaudioside A standard (Lot #D178001R).
- 3.1.2. Stevioside standard (Lot #ST0305002).
- 3.1.3. Acetonitrile (ACN), HPLC grade or equivalent
- 3.1.4. HPLC grade water or equivalent
- 3.1.5. Ortho-phosphoric acid 85%, reagent grade or equivalent
- 3.1.6. Monosodium dihydrogen phosphate anhydrous (MW:119.98 g/mol), reagent grade or equivalent

3.2. Apparatus

- 3.2.1. An HPLC system, Agilent HP 1100, or equivalent, comprised of a pump, a column thermostat, an autosampler, a UV detector capable of background correction and a data acquisition system.
- 3.2.2. Column: Agilent Poroshell 120 SB-C18, 4.6 mm x 150 mm, 2.7 μ m, (Part # 683975-902).
- 3.2.3. Analytical balance capable of weighing to 0.0001 g (0.1 mg).
- 3.2.4. Sonicator.
- 3.2.5. Volumetric flasks: 50 mL and 100 mL volumetric flasks to prepare all samples, and standard.

3.3. Mobile Phase Preparation

Mobile phase solutions may be scaled up or down as needed. Mobile phase may be used for up to 1 weeks.

- 3.3.1. Buffer Solution (10 mmol/L monosodium dihydrogen phosphate)

The buffer is prepared by dissolving 1.2 g monosodium dihydrogen phosphate in 1 liter of water. Adjust the pH to 2.6 by using ortho-phosphoric acid 85%.

- 3.3.2. Mobile Phase (acetonitrile : buffer)

Acetonitrile and buffer are mixed to be a mixture solution of 32% Acetonitrile and 68% buffer.

- 3.3.3. Diluent Solution (70% water in acetonitrile)

To prepare the diluent solution, combine 700 mL of Water and 300 mL of acetonitrile and mix thoroughly. Be sure that it is at room temperature before use because it cools on mixing.

3.3 Standard Preparation

- 3.4.1 Steviol Glycosides retention time marker solution (M11)

Place 10 mg (each) of Rebaudioside D, Rebaudioside A, Stevioside, Stevioside(isomer) , Rebaudioside F, Rebaudioside C, Rebaudioside C(isomer), Dulcoside A, Rubusoside, Rebaudioside B and Steviolbioside in 100ml volumetric flask and dilute to volume with diluent. This will make 11 component steviol glycosides that are approximately 100 mg/L (each).

3.4.2 Rebaudioside A standard

Place 40, 50.0, 60, and 70 ± 5 mg, of the Rebaudioside A standard in four separate 100 mL volumetric flasks and dilute to volume with the diluent solution (This is not allowed that standards are prepared with injection volume, and be sure that it is at room temperature, because it cools upon mixing). This will make standards that are approximately 400, 500, 600, and 700 mg/L. Measure the moisture content by Karl Fischer analysis every time a group of standards are prepared. Be sure to also correct for purity according to the certificate of analysis. Sonicate the solutions if necessary until dissolved. For all of the standards, the weights and volumes can be adjusted up proportionally as needed, but not down.

3.4.3 Stevioside standard

Place 20, 30.0, 40, and 50 ± 5 mg, of the Stevioside standard in four separate 100 mL volumetric flasks and dilute to volume with the diluent solution (This is not allowed that standards are prepared with injection volume, and be sure that it is at room temperature, because it cools upon mixing). This will make standards that are approximately 200, 300, 400, and 500 mg/L. Measure the moisture content by Karl Fischer analysis every time a group of standards are prepared. Be sure to also correct for purity according to the certificate of analysis. Sonicate the solutions if necessary until dissolved. For all of the standards, the weights and volumes can be adjusted up proportionally as needed, but not down.

3.5 Sample Preparation

Samples are prepared by placing 60 ± 5 mg, recorded to the nearest 0.01 mg, of sample in a 50 mL volumetric flask and diluting to volume with the diluent solution. Sonicate the solution if necessary until dissolved. This will make an approximately 1200 mg/L sample. The weight and volume can be adjusted up proportionally as needed, but not down. The sample weight can be adjusted as necessary for moisture. They are preparation in duplicate. If the sample will not be analyzed immediately, then it is be stored without headspace, under nitrogen and desiccated.

3.6 Instrumental Conditions

Column:	Poroshell 120 SB-C18, 150 x 4.6 mm, 2.7 μ m
Temperature:	40 °C
Mobile Phase:	68% Phosphate buffer, 32% acetonitrile
Flow Rate:	1.0 mL/min
Injection:	5 μ L
Detection:	UV at 210 nm (4 nm bandwidth), Reference: 260 nm (100 nm bandwidth)
Run Time:	20 min (initially, it will be longer, but should be no more than 30 min)
Autosampler Temp:	ambient
Sample Conc.:	1200 mg/L in diluent solution

4. Analysis Procedure

4.1 System Startup/Suitabi

Area Count and Retention Time Reproducibility

Make six injections of the rebaudioside standard 700 ppm (use Rebaudioside Standard Level 4 as for RSD test). The RSD of the peak area counts (mAU*s) and retention time (min.) for rebaudioside A should be 2.00% or less. If not, continue with up to three more injections until the peak area and retention time of the last five injections are reproducible. If the %RSD of peak areas and retention times are > 2.00% RSD, investigate the instrument for problems and take corrective action. Record any corrective actions taken. Repeat step 4.1.1.1 after correcting any problems with the instrument. Refer to section 4.5.2 for the equation to calculate % RSD.

4.2 Chromatographic Profile

Below is a guideline of the retention times (RT) for steviol glycoside component.

Compound	*R.T. (min.)
REBD	2.070
REBA	4.578
STEV	4.798
REBF	5.848
REBC	6.430
DULA	6.929
STEV A	7.563
REBC I	8.230
RUBU	8.926
REBB	12.669
SBIO	13.337

*Estimate

4.3 Assay Sequence

After performing the system suitability checks, inject all remaining standards followed by the samples. A sample or standard should not be followed by any other injections (blanks, impurity standards, etc.) without a sacrificial injection (mobile phase) in between to prevent carryover contamination.

4.5 Calculations

4.5.1 RSD Calculation for Peak Area

$$\%RSD = (\text{Std. Dev.}/\text{Mean}) * 100$$

Where:

$$\text{Std. Dev.} = ((\sum(\bar{X} - X)^2)/(N - 1))^{1/2}$$

$$\text{Mean} = \bar{X} = (X_1 + X_2 + X_3 + X_N)/N$$

X = Peak area

N = Total number in the sample

the analyte

4.6 Standard Curve Acceptance Criteria

4.6.1 Standard Curve for rebaudioside A

To be acceptable for use in the calibration curve, the standard recoveries must be within $100.0 \pm 3\%$ for all rebaudioside A concentration levels. No more than one standard may be omitted at each concentration level. The correlation coefficient for the standard curve is acceptable if it is greater than 0.9990. If the standard curve fails the acceptance criteria notify your supervisor and investigate the problem.

4.6.2 Standard Curve for Stevioside

To be acceptable for use in the calibration curve, the standard recoveries must be within $100.0 \pm 3\%$ for all stevioside concentration levels. No more than one standard may be omitted at each concentration level. The correlation coefficient for the standard curve is acceptable if it is greater than 0.9990. If the standard curve fails the acceptance criteria notify your supervisor and investigate the problem.

4.7 Analysis Calculation

4.7.1 Identify analytes of interest by retention time match with M9 solution.

4.7.2 Determine the area response of the analytes as well as any measurable peaks (except for solvent peaks) from the samples and standards.

4.7.3 Prepare full fit linear regression standard curve by plotting rebaudioside A or stevioside concentration in mg/L (corrected for moisture and purity) on the ordinate scale versus its respective area response on the abscissa scale. Alternatively, use the data acquisition software to prepare the calibration curve.

4.7.4 **From the slope and y-intercept (must be force to zero (0)) of the standard curve**, calculate the concentration in mg/L of the analytes (rebaudioside A using the rebaudioside A curve and all others use the stevioside curve) in the samples using the following equation. Alternatively, use the data acquisition software to calculate the concentrations of the analytes based on the calibration curves prepared using the software.

$$\text{Conc. (mg/L)} = \text{Area Response} \times \text{slope} + \text{y-intercept}$$

4.7.5 Calculate the % w/w of each analyte in the samples as follows:

4.7.5.1 Multiply the concentration of each known impurity (REB D, REB F, REB C, DUL.A, STEV A, REB CI, RUBU, REB B, SBIO) by its' correction factor to correct for the difference in molecular weight between it and stevioside. The correction factors are 1.40, 1.16, 1.18, 0.98, 1.00, 1.18, 0.80, 1.00 and 0.80, respectively. No correction is needed for REBB and STEV A because they have the same molecular weight with stevioside

4.7.5.2 Calculate the w/w% of rebaudioside A and other steviol glycosides in the samples as follows:

$$\text{w/w\%} = \text{Conc. of the Analyte (mg/L)} \times 100 / \text{Sample Conc. (mg/L)}$$

4.7.5.3 Correct rebaudioside A for moisture and solvents (if applicable) by multiplying the %wt/wt determined in section 4.7.5.2 by the following factor:

$$F = 100 / (100 - \% \text{ Moisture in Sample})$$

For example, the rebaudioside A value from the calculation in section 4.7.5.2 for the validation sample is 94.41% wt/wt and the % moisture is 5.78%. Therefore:

$$F = 100 / (100 - 5.78) = 1.061$$

and the CC-00201 in the validation sample on a dry basis is:

$$\% \text{wt/wt (dry basis)} = 94.41\% \text{ wt/wt} \times 1.061 = 100.2\%.$$

4.8 Quality Control Check

The % RSD for rebaudioside A and stevioside results between duplicate samples should be no greater than 2.00% and 50% for levels below 5 mg/L (0.1% in the sample) and no greater than 20% for levels above 5 mg/L. When the duplicate sample RSD does not fall within the limits described above, re-run fresh sample preparations. If the second sample set passes the QC check, report the second set of data. If the second set confirms the difference seen in the first set, inform your supervisor and investigate the problem.

METHOD 2

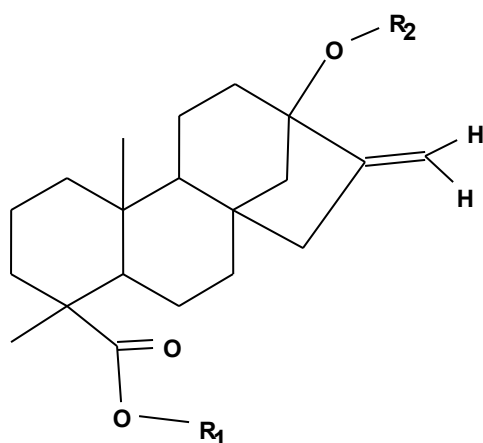
1. Method Summary

This HPLC method is applicable to the quantitation of 5 compounds of steviol glycosides, based on rebaudioside D, rebaudioside M standards, for setting specifications and confirming adequate purity for bulk release.

The analytes are separated using an Agilent Poroshell 120 SB- C18, 4.6 mm x 150 mm, 2.7 μm at 40 $^{\circ}\text{C}$. The mobile phase consists of two type of premix which is premix 1 containing 75% 10mmol/L phosphate buffer (pH2.6) and 25% acetonitrile, while premix 2 containing 50% 10mmol/L phosphate buffer (pH2.6) and 50% acetonitrile. All the 5 steviol glycosides compounds are detected by ultraviolet absorption (UV) at 210. Samples are prepared in an acetonitrile-water (30:70) solution at 1200 mg/L. Quantitation of 5 steviol glycosides is performed using a Four-point linear regression curve fit of rebaudioside D, rebaudioside M standards.

2. Structures

Structure of Stevia sweeteners hydrocarbon backbone.



Formula, and R-Groups in Backbone Structure of Identified Steviol Glycosides

Common Name	Symbol	Trivial Formula	R ₁	R ₂	Reference	Molecular Weight (g/mol)
Rebaudioside <i>N</i>	REBN	SvR1G5	Rha α (1-2)[Glc β (1-3)]Glc β 1-	Glc β (1-2)[Glc β (1-3)]Glc β 1-	4a	1275.29
Rebaudioside <i>O</i>	REBO	SvR1G6	Glc β (1-3)Rha α (1-2)[Glc β (1-3)]Glc β 1-	Glc β (1-2)[Glc β (1-3)]Glc β 1-	4a	1437.44
Rebaudioside <i>M</i>	REBM	SvG6	Glc β (1-2)[Glc β (1-3)]Glc β 1-	Glc β (1-2)[Glc β (1-3)]Glc β 1-	4a	1291.29
Rebaudioside <i>D</i>	REBD	SvG5	Glc β (1-2)Glc β 1-	Glc β (1-2)[Glc β (1-3)]Glc β 1-	11	1129.18
Rebaudioside <i>E</i>	REBE	SvG4	Glc β (1-2)Glc β 1-	Glc β (1-2)Glc β 1-	11	967.03

*rha = rhamnose, glc = glucose, xyl = xylose

3. Experimental

3.1. Reagents and Chemicals

- 3.1.1. Rebaudioside D standard
- 3.1.2. Rebaudioside M standard
- 3.1.3. Acetonitrile (ACN), HPLC grade or equivalent
- 3.1.4. HPLC grade water or equivalent
- 3.1.5. Ortho-phosphoric acid 85%, reagent grade or equivalent
- 3.1.6. Monosodium dihydrogen phosphate anhydrous (MW:119.98 g/mol), reagent grade or equivalent

3.2. Apparatus

- 3.2.1. An HPLC system, Agilent HP 1200, or equivalent, comprised of a pump, a column thermostat, an autosampler, a UV detector capable of background correction and a data acquisition system.
- 3.2.2. Column: Agilent Poroshell 120 SB-C18, 4.6 mm x 150 mm, 2.7 μ m, (Part# 683975-902).
- 3.2.3. Analytical balance capable of weighing to 0.0001 g (0.1 mg).
- 3.2.4. Sonicator.
- 3.2.5. Volumetric flasks: 50 mL volumetric flasks to prepare all samples, and standard.

3.3. Mobile Phase Preparation

Mobile phase solutions may be scaled up or down as needed. Mobile phase may be used for up to 1 weeks.

3.3.1 Buffer Solution (10 mmol/L monosodium dihydrogen phosphate)

The buffer is prepared by dissolving 1.4 g monosodium dihydrogen phosphate one (1) liter of water. Adjust the pH to 2.6 by using ortho-phosphoric acid 85%.

3.3.2 Mobile Phase (acetonitrile : buffer)

3.3.2.1 Premix 1: Acetonitrile and buffer are mixed to be a mixture solution of 25% Acetonitrile and 75% buffer.

3.3.2.2 Premix 2: Acetonitrile and buffer are mixed to be a mixture solution of 50% Acetonitrile and 50% buffer.

3.3.3 Diluents Solution (70% water in acetonitrile)

To prepare the diluent solution, combine 700 mL of Water and 300 mL of acetonitrile and mix thoroughly. Be sure that it is at room temperature before use because it cools on mixing.

3.4 Standard Preparation

3.4.1 Steviol Glycosides retention time marker solution (M5)

Place 10 mg (each) of Rebaudioside E, Rebaudioside O, Rebaudioside D, Rebaudioside N, Rebaudioside M in 100 ml volumetric flask and dilute to volume with diluent. This will make 5 component steviol glycosides that are approximately 100 mg/L (each).

3.4.2 Multilevel Standards Preparations

3.4.2.1 Rebaudioside D standards – Place 5.0, 10.0, 15.0 and 20.0 mg of the rebaudioside D standard in four separate 50 ml volumetric flask and dilute to volume with the diluents solution. This will make standards concentration are approximately 100, 200, 300 and 400 mg/L. Measure the moisture content by Karl Fischer analysis. Sonicate the solutions if necessary until dissolved. For all of the standards, the weights and volumes can be adjusted up proportionally as needed, but not down. And the concentration of standards should be corrected for moisture and purity.

3.4.2.2 Rebaudioside M standards – Place 5.0, 10.0, 15.0 and 20.0 mg of the rebaudioside M standard in four separate 50 ml volumetric flask and dilute to volume with the diluents solution. This will make standards concentration are approximately 100, 200, 300 and 400 mg/L. Measure the moisture content by Karl Fischer analysis. Sonicate the solutions if necessary until dissolved. For all of the standards, the weights and volumes can be adjusted up proportionally as needed, but not down. And the concentration of standards should be corrected for moisture and purity.

3.5 Instrumental Conditions

Column:	Poroshell 120 SB-C18, 150 x 4.6 mm, 2.7 mm		
Temperature:	40 °C		
Mobile Phase:	Premix 1 ; 75% Phosphate buffer, 25% acetonitrile		
	Premix 2 ; 50% Phosphate buffer, 50% acetonitrile		
Gradient setup:	Time (min)	Channel A: Premix 1	Channel B: Premix 2
	0	100%	0%
	16	100%	0%
	16.5	0%	100%
	25	0%	100%
	25.5	100%	0%
	40	100%	0%
Flow Rate:	0.5 mL/min		
Injection:	5 mL		
Detection:	UV at 210 nm		
Run Time:	45 min		
Autosampler Temp	ambient		
Sample Conc.:	1000-1200 mg/L in diluent solution		

4. Analysis Procedure

4.1 System Startup/Suitability

Area Count and Retention Time Reproducibility

Make six injections of the rebaudioside D standard 400 ppm (use standard level 4 as for RSD test). The RSD of the peak area counts (mAU*s) and retention time (min.) for rebaudioside D should be 1.00% or less. If not, continue with up to three more injections until the peak area and retention time of the last five injections are reproducible. If the %RSD of peak areas and retention times are > 2.00% RSD, investigate the instrument for problems and take corrective action. Record any corrective actions taken. Repeat after correcting any problems with the instrument. Refer to section 4.3.4 for the equation to calculate % RSD

4.2 Chromatographic Profile

Below is a guideline of the retention times (RT) for steviol glycoside component.

Compounds	*R.T. (min.)
REBE	10.23
REBO	11.41
REBD	12.03
REBN	13.26
REBM	15.23

*Estimate

After performing the system suitability checks, inject all remaining standards followed by the samples. A sample or standard should not be followed by any other injections (blanks, impurity standards, etc.) without a sacrificial injection (mobile phase) in between to prevent carryover contamination.

4.3 Calculations

4.3.1 Standard curve

Prepare full fit linear regression standard curve by plotting the Conc. of stds (corrected by moisture and purity) on the ordinate scale and the area of response on the abscissa scale. Use the data acquisition software to calculate the concentrations of the analytes based on the calibration curves prepared using the software.

$$\text{Conc. (mg/L)} = \text{Area Response} * \text{slope} + \text{y-intercept}$$

4.3.2 Standard Curve Acceptance Criteria

To be acceptable for use in the calibration curve, the standard recoveries and std checks must be within $100.0 \pm 2\%$ for all standards concentration levels. The correlation coefficient for the standard curve is acceptable if it is greater than 0.9990. If the standard curve fails the acceptance criteria notify your supervisor and investigate the problem.

4.3.3 Analysis Calculation

Identify analytes of interest by retention time match with M5 solution.

Determine the area response of the analytes as well as any measurable peaks (except for solvent peaks) from the samples and standards. Alternatively, use the data acquisition software to calculate the concentrations of the analytes based on the calibration curves prepared using the software. REB M is calculated by REB M curve. REB E, REB O, REB D, REB N are calculated by REB D curve. Because of the difference of Molecular between REB D and other compounds, correction factors are used to correct during the calculation. The correction factors are 0.86, 1.27, 1.00, 1.13

$$\text{Conc. (mg/L)} = \text{Peak area@210nm} * \text{Slope} * \text{Correct factors}$$

Calculate the w/w% of rebaudioside M and other steviol glycosides in the samples as follows:

$$\text{w/w\%} = \text{Conc. of the Analyte (mg/L)} * 100 / \text{Sample Conc. (mg/L)}$$

Correct rebaudioside M for moisture and solvents (if applicable).

For example, the rebaudioside M value from the calculation for the validation sample is 94.41% wt/wt and the % moisture is 5.78%. Therefore:

$$F = 100 / (100 - 5.78) = 1.061$$

and the validation sample on a dry basis is:

$$\% \text{wt/wt (dry basis)} = 94.41\% \text{ wt/wt} * 1.061 = 100.2\%.$$

4.3.4 RSD Calculation for Peak Area

$$\% \text{RSD} = (\text{Std. Dev.} / \text{Mean}) * 100$$

Where:

$$\text{Std. Dev.} = ((\sum (\bar{X} - X)^2) / (N - 1))^{1/2}$$

$$\text{Mean} = \bar{X} = (X_1 + X_2 + X_3 + X_N) / N$$

X = Peak area

N = Total number in the sample

the analyte

4.4 Quality Control Check

The % RSD between duplicate samples should be no greater than 2.00% for levels above 400 mg/L (20% in sample), 10% for levels above 200 mg/L (10% in sample) and 50% for levels below 2 mg/L (0.1% in the sample) and no greater than 20% for levels above 2 mg/L. When the duplicate sample RSD does not fall within the limits described above, re-run fresh sample preparations. If the second sample set passes the QC check, report the second set of data. If the second set confirms the difference seen in the first set, inform your supervisor and investigate the problem.