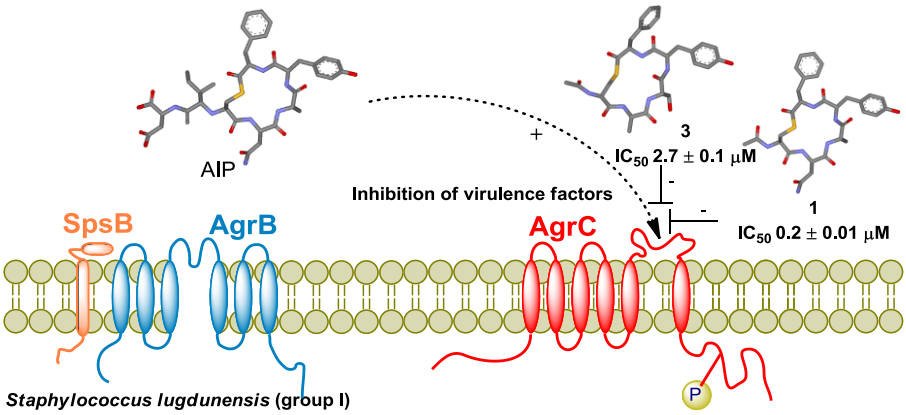
Supplementary Information

Truncated auto-inducing peptides as antagonists of *Staphylococcus lugdunensis* quorum sensing

Christopher P. Gordon,[a]* Shondra D. Olson,[b] Jessica L. Lister,[b] Jeffrey S. Kavanaugh,[b] and Alexander R. Horswill[b]*

Competitive quorum sensing antagonisim (QS) offers a novel strategy for attenuating current multi-drug resistant staphylococcal infections. To this end, a series of 10 truncated analogues based on the parent auto-inducing peptides (AIPs) of *Staphylococcus lugdunensis* (group I & II) and *Staphylococcus epidermidis* (groups I – III) were sequentially assessed against a newly developed Staphylococcus *lugdunensis* group I QS reporter strain. The truncated analogues based upon *Staphylococcus lugdunensis* AIP-1 (**1**) and AIP-2 (**2**) displayed respective IC_{50} values of 0.2 ± 0.01 μM and 0.3 ± 0.01 μM whilst the truncated analogue of the *S Staphylococcus. epidermidis* AIP-1 (**3**) elicited an IC_{50} value of 2.7± 0.1 μM. These findings demonstrate the potential of cognate and 'cross-talk' competitive quorum sensing inhibition using truncated AIPs as a means of attenuating staphylococcal infections in species beyond *Staphylococcus aureus*.



Biology, Quorum-sensing inhibition assay

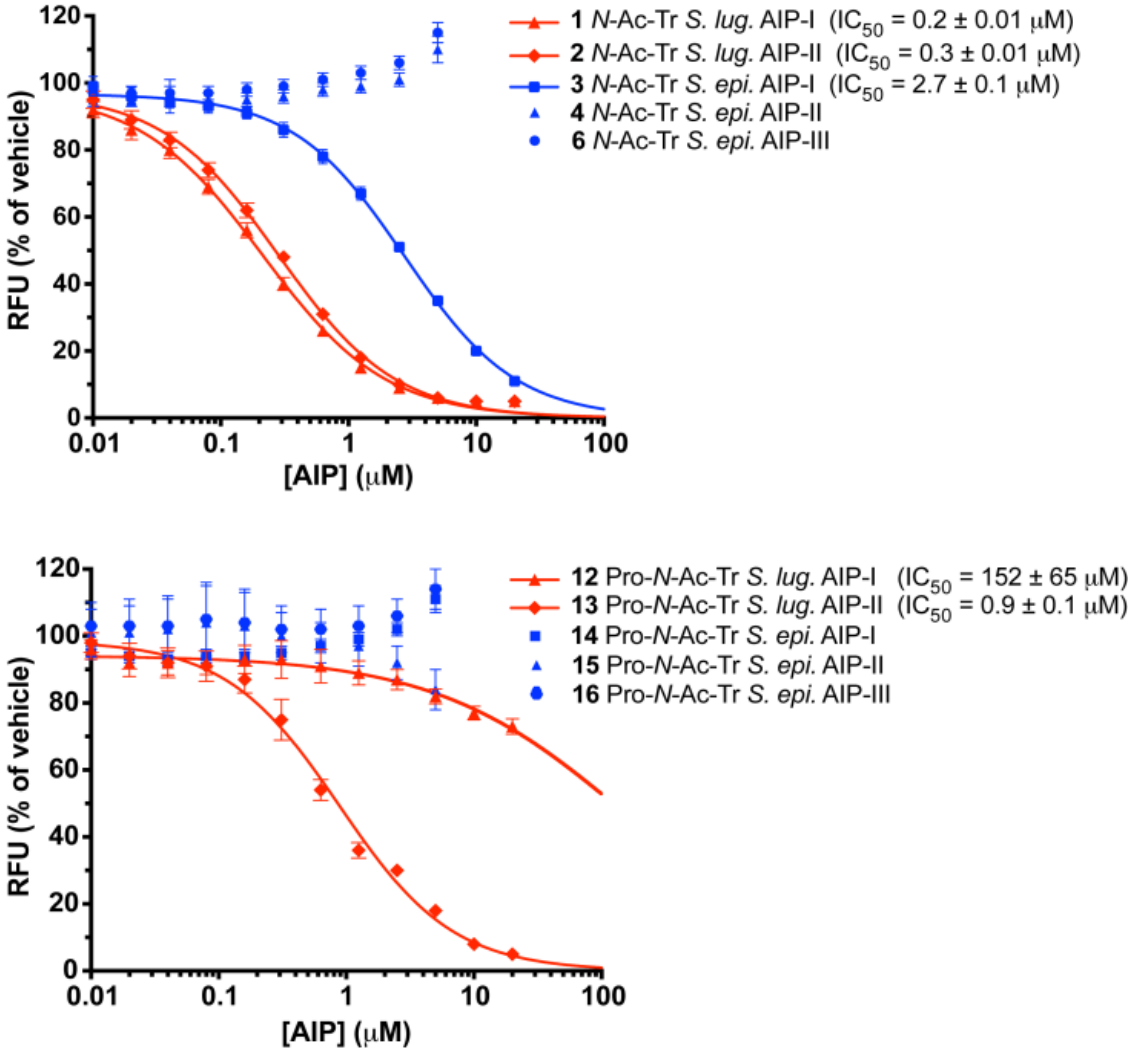


Figure 1. Dose-response curves and tabulated IC_{50} values for *N*-acetylated truncated analogues (A) and protected *N*-acetylated truncated analogues (B). Analogues based on *S. lugdunensis* and *S. epidermidis* AIP macrocycles are shown in red and blue, respectively. In cases where the data could be fit, the IC_{50} values are listed in the Figure key.

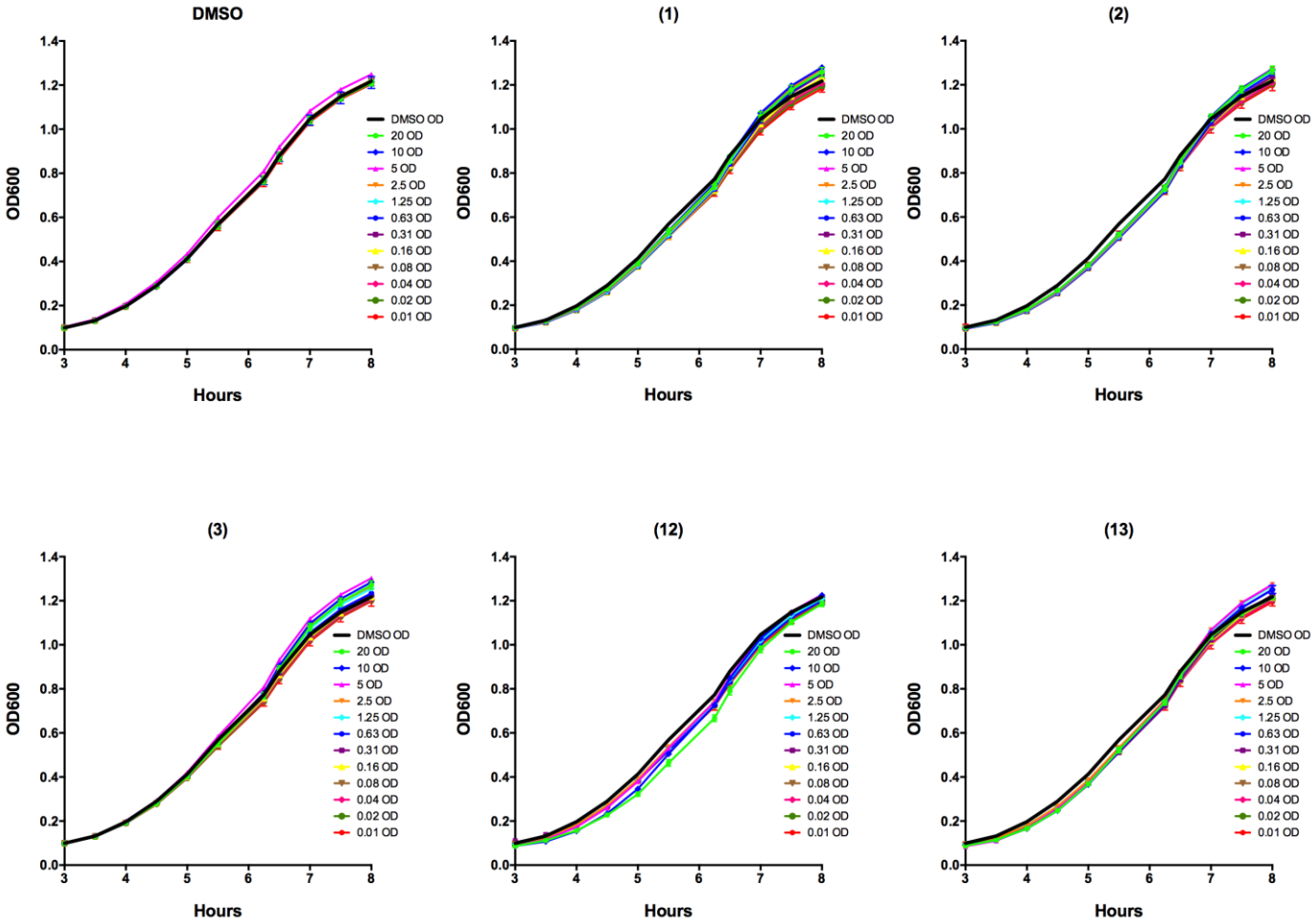


Figure 2. OD600 curve for DMSO control and curves for active compound **1** - **3**, **12** and **13** which demonstrate that the compounds do not inhibit bacterial growth over an 8 hour period.

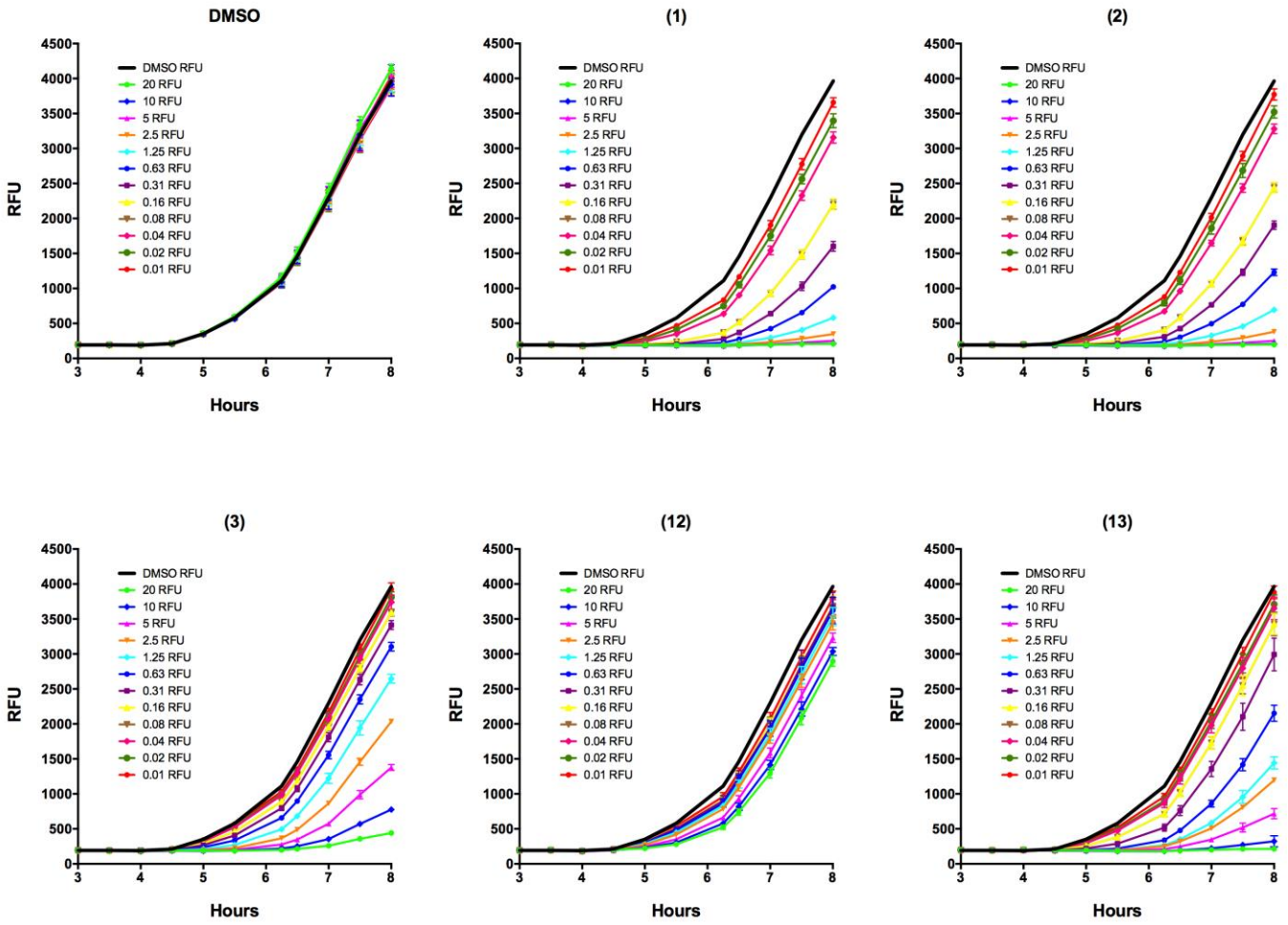


Figure 3: Relative fluorescence units (RFU) curve for a DMSO control and curves for active compound **1** - **3**, **12** and **13** which demonstrate that the compounds do not inhibit bacterial growth over an 8 hour period.

In order to assess the ArgC inhibitory activity of the AIP analogues a *S. lugdunensis* I *agr* reporter strain, AH4031, was constructed by moving *agr*P3sGFP reporter plasmid pCM40[1] into *S. lugdunensis* strain N920143[2] using the electroporation protocol described by Heilbronner and colleagues.[3] Prior to electroporation into *S. lugdunensis* N920143, pCM40 was passaged through *E. coli* strain SL01B,[3] which is engineered to express the specificity (HsdS) and methylation (HsdM) subunits of the *S. lugdunensis* N920143 type I restriction system.

Overnight cultures of AH4031, that were grown in Tryptic Soy Broth (TSB) supplemented with Cam at a concentration of 10 μg/mL, were inoculated at a dilution of 1:500 into fresh TSB containing Cam. 100 μL aliquots were added to 96-well microtiter plates (Costar 3603) and combined with 100 μL aliquots of TSB containing Cam and 2-fold serial dilutions (either 10 μM to 0.004 μM or 40 μM to 0.2 μM) of the AIP analogs (dissolved in DMSO). After mixing, the effective inoculum dilution was 1:1000 and the final AIP concentrations ranged from 5 μM to 0.002 μM or 20 μM to 0.1 μM, with a final DMSO concentration of 2% (v/v) in all wells. Four dilution series were prepared for each AIP, and 4 mock vehicle (DMSO) dilution series were also prepared. Microtiter plates were incubated at 37°C with shaking (1000 rpm) in a Stuart SI505 incubator (Bibby Scientific, Burlington, NJ) with a humidified chamber. Fluorescence (top reading, 493 nm excitation, 535 nm emission, gain 60) and

optical density (OD) readings at 600 nm were recorded at 30 min increments using a Tecan Systems (San Jose, CA) Infinite M200 plate reader. Data was transformed into units of percent of vehicle, and IC_{50}'s were obtained by subjecting the transformed fluorescent data from 8 hours of growth to four-parameter-logistic fits (4PL) using GraphPad Prism version 6.0g. In cases where the fluorescence at the highest AIP concentration tested was insufficient to fully inhibit quorum-sensing the bottom baseline was fixed at 0%.

Chemistry. General Methods. Chemicals and solvents were purchased from standard suppliers and used without further purification. Fmoc-protected amino acids were purchase for Auspep as was 2-chlorotrityl chloride resin (loading 1.5 mmol/g) and (2-(6-Chloro-1H-benzotriazole-1-yl)-1,1,3,3-tetramethylaminium hexafluorophosphate) (HCTU). Polymer-bound 1-(3-Dimethylaminopropyl)-3-ethylcarbodiimide and N-benzyl-N′-cyclohexylcarbodiimide were purchased from Sigma-Aldrich with respective loadings of ≈ 1 – 2 mmol/g). The PS-carbodiimde was obtained from biotage (loading 1.27 mmol/g). All solvents were used as supplied (analytical, HPLC or peptide grade), without prior purification. Milli-Q water was used for chemical reactions. Deuterated DMSO-d_6 was purchased from Sigma Aldrich. Reactions were monitored by using analytical RP-HPLC and MS. Final compound purity was assessed *via* analytical HPLC will all biologically evaluated peptides of > 95 % purity.

Mass spectra (ES-TOF) were recorded on a Waters 2795 separation module/Micromass LCTTM platform. ^{1}H and ^{13}C spectra were recorded at 25 °C on a Varian mercury 300 operating at 299.8 MHz and 75.4 MHz respectively. Chemical shifts (δ) are reported in parts per million (ppm), referenced to TMS (1H, 0.0 ppm). Coupling constants (*J*) are recorded in Hz and significant multiplicities described by singlet (s), doublet (d), doublet of doublets (dd), doublet of triplets (dt), triplet (t), quadruplet (q), broad (br), multiplet (m). Spectra were assigned using appropriate gCOSY sequences.

Analytical RP-HPLC was performed using a Waters instrument comprised of two 510 pumps, a 486 detector and Millenium^{TM} software. The systems outlined below were used for purification and to confirm purity. Analytical RP-HPLC was performed using Phenomenex Onyx Monolithic reversed-phase C18 column (4.6 x 100 mm). Solvent A: 0.06% TFA in water and solvent B: 0.06% TFA in CH_3CN:H_2O (90:10), flow rate of 1.0 mL/min, gradient 10-100 (%B), curve = 6, over 15.0 mins, and detection at 216 nm and 254 nm (System 1).

Semi-preparative RP-HPLC was performed using a Waters 2525 binary gradient pump equipped with a water 2487 dual λ absorbance detector and a Chromolith®SemiPrep RP-18e 100-10 mm column. A flow rate of 10 mL/min was used with solvent A: 0.06% TFA in water and solvent B: 0.06% TFA in CH_3CN:H_2O (90:10). Gradient 10-75 (%B) over 15 mins, curve = 6, with UV detection at 216 nm and 254 nm.

General procedure 1: Fmoc-Phe-2-ClTrt resin; 2-Chlorotritylchloide resin (100-200 mesh), 1% DVB (5.00 g, loading = 1.5 mmol/g) (Merck Chemicals Ltd. # 8.55017) was swelled in DCM (50.0 mL) for 0.5 h prior to the addition of 4.0 eq. of Fmoc-Phe-OH (11.62 g, 0.03 mol), and 8.0 eq. of DIPEA (10.45 mL, 0.06 mmol). The resulting suspension was gently stirred at rt for 2 h before MeOH (5.0 mL) was added. After an additional 20 mins of gentle stirring the resin was filtered and sequentially washed with DMF (2 × 50 mL), acetonitrile (2 × 50 mL), hexanes (2 × 50 mL), and DCM (2 × 50 mL). The resin was dried overnight under high vacuum and standard UV Fmoc quantification revealed a resin loading of 0.83 mmol/g (loading = 86 %).

Fmoc Loading Determination; Two samples of pre-loaded dried resin (~10.0 mg each) were each added to a vial containing a freshly prepared solution of 20% piperidine in DMF (3.00 mL). The resulting suspensions were gently agitated at rt for 2 h after which 300 μL of each resin suspension was transferred to a quartz UV cuvette and an additional 2.70 mL of 20% piperidine in DMF was added. A reference cell containing 3.00 mL of 20% piperidine was placed into a UV spectrophotometer and reference was set at 290 nm. The absorbance of the cuvettes containing the settled resin was then obtained (290 nm). The loading of the resin was then determined using the following equation (final loading = average of the two resin samples):

$$\text{Loading (mmol/g)} = [(\text{Abs}_{\text{sample}})/(\text{mg of sample} \times 1.75)] \times 10$$

Theoretical loading was determined using the following equation:

$$A = B \times 1000/[1000 + (B \times (M - X))]$$

A = theoretical substitution (mmol/g)

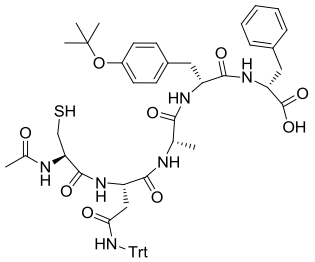
B = substitution of resin (mmol/g)

M = molecular weight of target peptide (with all protecting groups)

X = 36 (loss of Cl on amino acid loading)

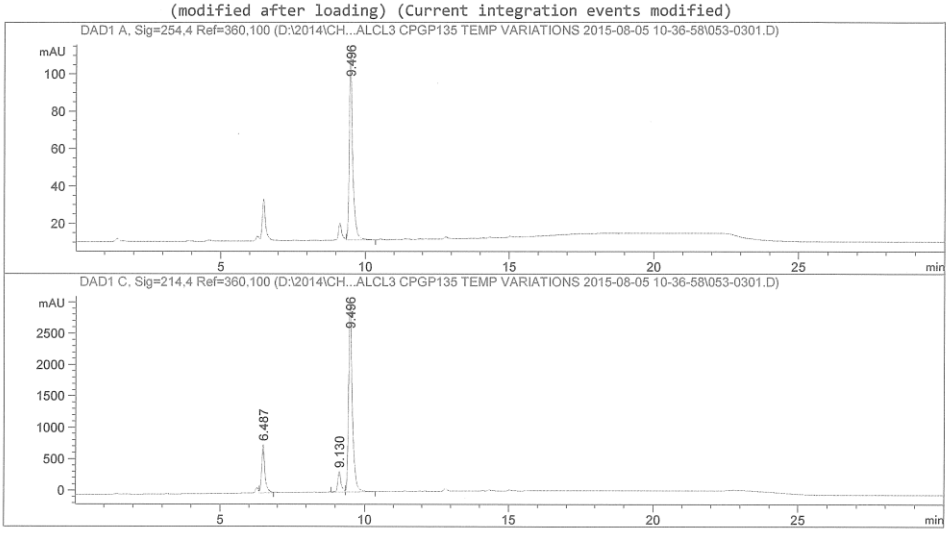
Fmoc-Leu-2-ClTrt resin; prepared utilising general procedure 1 with 2-Chlorotritylchloide resin (100-200 mesh), 1% DVB (5.00 g, loading = 1.5 mmol/g), 4.0 eq. of Fmoc-Leu-OH (10.60 g, 0.03 mol), and 8.0 eq. of DIPEA (10.45 mL, 0.06 mmol). Fmoc quantification revealed a resin loading of 1.07 mmol/g (loading = 94 %).

General Procedure 2, Compound 6. Fmoc-Phe-2-Chlorotritylchloide resin (0.14 g, 0.12 mmol, loading = 0.83 mmol/g) was placed in an Omnifit™ BenchMark™ column assembly and swelled with DMF (2.5 mL) for 0.5 h. Utilising a NovaSyn® manual peptide synthesizer Fmoc-depotection and washing was achieved using 20% piperidine in DMF and DMF (2.8 mL/min) respectively. The column was drained and a solution of 4.0 eq. Fmoc-(tBu)Try-OH (0.22 g, 0.48 mmol), 4.0 eq. HCTU (0.19 g, 0.48 mmol), and 8.0 eq DIPEA (0.16 mL, 3.84 mmol) in DMF (1.5 mL) was added. The column was gently agitated at ~40 °C for 1 hour after which the resin was washed (DMF 2.8 mL/min), Fmoc-deprotected (20% piperidine in DMF 2.8 mL/min), and washed (DMF 2.8 mL/min). The remaining linear peptide sequence was prepared *via* subsequent rounds of acylation, washing (DMF 2.8 mL/min), Fmoc-deprotection (20% piperidine in DMF, 2.8 mL/min), and washing (DMF 2.8 mL/min). Each acylation was achieved using a solution of 4.0 eq. HCTU (0.19 g, 0.48 mmol), 8.0 eq DIPEA (0.16 mL, 3.84 mmol), DMF (1.5 mL) at ~40 °C over 1 hour and 4.0 eq. Fmoc-Ala-OH (0.14 g, 0.48 mmol), and 4.0 eq. Fmoc-(Trt)Asn-OH (0.28 g, 0.48 mmol), and Fmoc-(Mmt)Cys-OH (0.29 g, 0.48), respectively. The *N*-terminal Cys-amine was acetyaled *via* addition of an acetic anhydride (0.27 mL, 2.94 mmol) DIPEA (0.51 mL, 2.94 mmol) solution. After 0.5 h the resin was washed with MeOH (2 × 5.0 mL), acetonitrile (2 × 5.0 mL), hexanes (2 × 5.0 mL), and DCM (2 × 5.0 mL) and dried *in vacuo*. Concurrent cleavage of the linear peptide from the resin and deprotection of the Cys(Mmt) protecting group was achieved *via* flushing the column with a TFA cocktail (10 × 2 mL, DCM:TFA:TIPS 96:2:2). The resulting solution was concerted *in vacuo* and the residual crude material was triturated with ice-cold ether (× 3) to furnish an off-white solid. MS (ESI^+) m/z 957 (M + 1, 100 %) HRMS (ESI^+) for $C_{53}H_{61}N_6O_9S$; calculated 957.4142, found, 957.4146; RP-HPLC Onyx Monolithic C18 100 × 4.6 mm, 40-100% B in 15 min, t_R 9.5 min.

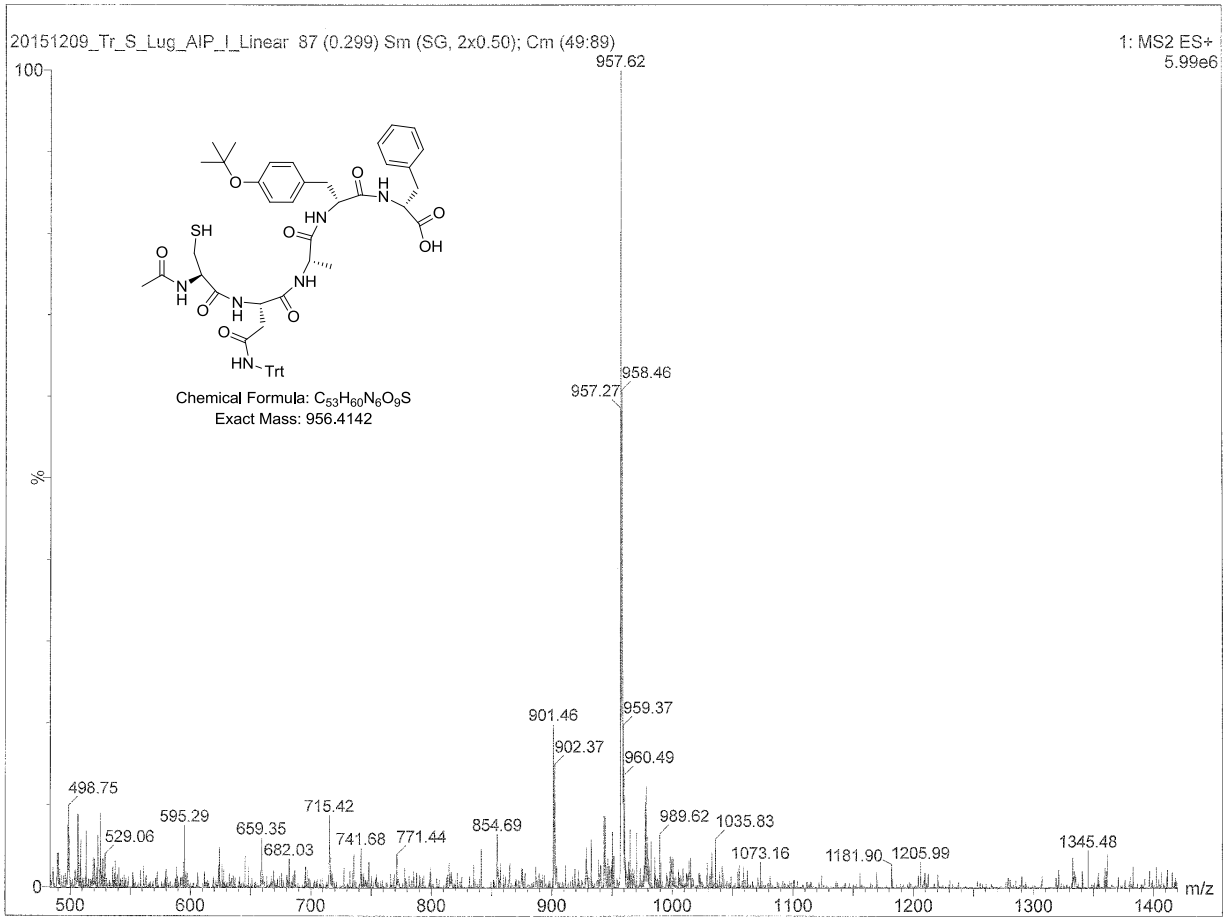


Data File D:\2014\CH...POUNDS\ALCL3 CPGP135 TEMP VARIATIONS 2015-08-05 10-36-58\053-0301.D
Sample Name: Ac-CNAYF-OH uncyclised

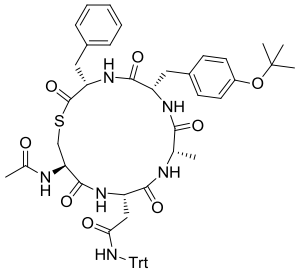
```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : LC1260                        Location : Vial 53
Injection Date  : 8/5/2015 11:41:17 AM               Inj :   1
                                               Inj Volume : 10.000 µl
Acq. Method     : D:\2014\CHRIS\DATA\DDCOPPERCOMPOUNDS\ALCL3 CPGP135 TEMP VARIATIONS 2015-08-
                  05 10-36-58\40 TO 100 OVER 15 MINS 10UL.M
Last changed    : 8/5/2015 10:36:58 AM by SYSTEM
Analysis Method : D:\2014\CHRIS\DATA\DDCOPPERCOMPOUNDS\ALCL3 CPGP135 TEMP VARIATIONS 2015-08-
                  05 10-36-58\40 TO 100 OVER 15 MINS 10UL.M (Sequence Method)
Last changed    : 8/5/2015 12:34:49 PM by SYSTEM
                  (modified after loading) (Current integration events modified)
```




```
==========================================================================
                          Fraction Information
==========================================================================
==========================================================================
No Fractions found.
==========================================================================
==========================================================================
                        Area Percent Report
==========================================================================

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


20151209_Tr_S_Lug_AIP_I_Linear 87 (0.299) Sm (SG, 2x0.50); Cm (49:89)
1: MS2 ES+
5.99e6
Chemical Formula: C53H60N6O9S
Exact Mass: 956.4142
957.62
958.46
957.27
959.37
901.46
902.37
960.49
498.75
595.29
715.42
529.06
659.35
682.03
741.68
771.44
854.69
989.62
1035.83
1073.16
1181.90
1205.99
1345.48
%
m/z
100
0
500
600
700
800
900
1000
1100
1200
1300
1400

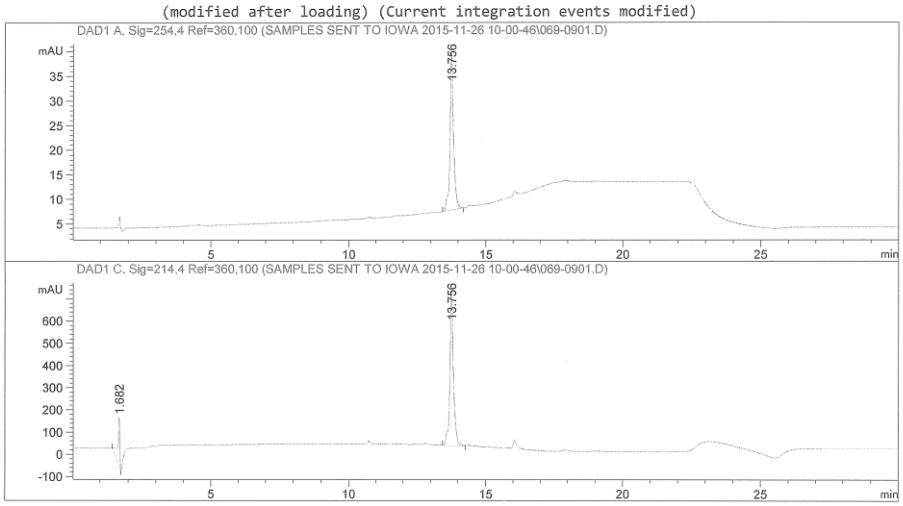
General Procedure 3 Compound 7; A suspension of the crude linear peptide and 3.0 eq. of PS-carbodiimide (0.28g, 0.36 mmol) and CH_3Cl (120 mL, 1.0 mM) was then stirred under an atmosphere of nitrogen for 5 h. After this period the solution was filtered, concentrated *in vacuo*, purified *via* semi-preparative RP-HPLC, and lyophilised to afford **7** (12 mg, 10.6 %). (*Note; 3 mg of this sample was collect for biological analysis*) MS (ESI^+) for $C_{53}H_{59}N_6O_8S$; *m/z* 939.29 (M + 1, 100 %); HRMS (ESI^+) for $C_{53}H_{59}N_6O_8S$; calculated 939.4037, found, 939.4042; RP-HPLC Onyx Monolithic C18 100 × 4.6 mm, 10-100% B in 15 min, t_R 13.75 min.



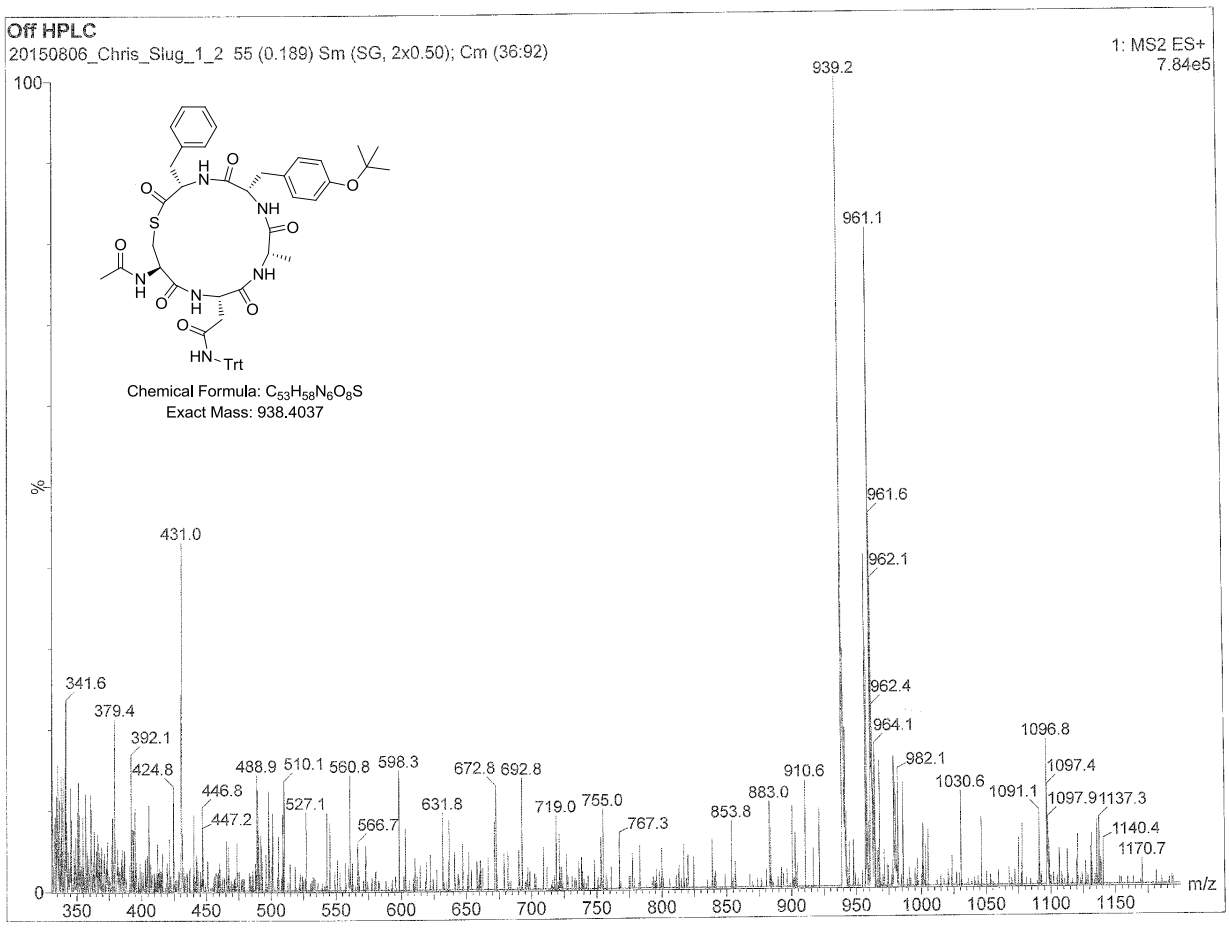
Chemical Formula: $C_{53}H_{58}N_6O_8S$
Exact Mass: 938.4037

Data File C:\CHEM32\1\DATA\SAMPLES SENT TO IOWA 2015-11-26 10-00-46\069-0901.D
Sample Name: Pro S lug AIP I cyclised

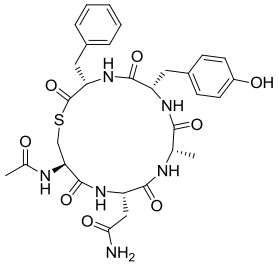
```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   9
Acq. Instrument : LC1260                        Location : Vial 69
Injection Date  : 11/26/2015 2:14:33 PM              Inj :   1
                                               Inj Volume : 20.000 µl
Acq. Method     : C:\CHEM32\1\DATA\SAMPLES SENT TO IOWA 2015-11-26 10-00-46\10 TO 100 OVER 15
                  MINS 20UL.M
Last changed    : 11/26/2015 10:00:46 AM by SYSTEM
Analysis Method : C:\CHEM32\1\DATA\SAMPLES SENT TO IOWA 2015-11-26 10-00-46\10 TO 100 OVER 15
                  MINS 20UL.M (Sequence Method)
Last changed    : 12/1/2015 10:39:28 AM by SYSTEM
                  (modified after loading) (Current integration events modified)
```




```
================================================================================
                          Fraction Information
================================================================================
================================================================================
No Fractions found.
================================================================================
======================================================================
                       Area Percent Report
======================================================================

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

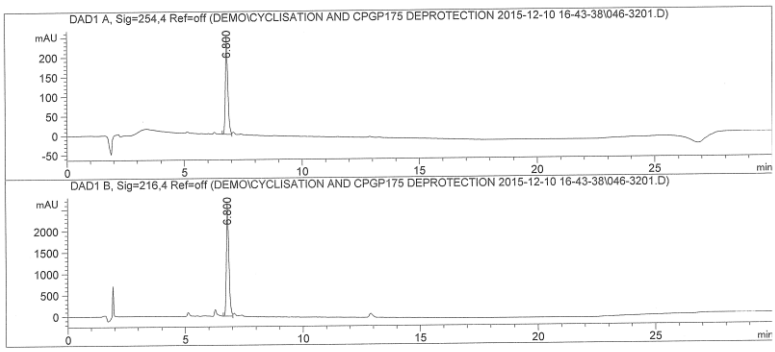

Off HPLC
20150806_Chris_Slug_1_2 55 (0.189) Sm (SG, 2x0.50); Cm (36:92)
1: MS2 ES+
7.84e5
Chemical Formula: C53H58N6O8S
Exact Mass: 938.4037
939.2
961.1
961.6
962.1
962.4
964.1
431.0
341.6
379.4
392.1
424.8
446.8
447.2
488.9
510.1
527.1
560.8
566.7
598.3
631.8
672.8
692.8
719.0
755.0
767.3
853.8
883.0
910.6
982.1
1030.6
1091.1
1096.8
1097.4
1097.9
1137.3
1140.4
1170.7
%
m/z

General Procedure 4; Compound 1; A solution of a TFA (10.0 mL), TIPS (0.25 mL), H_2O (0.25 mL), and **7** (9 mg, 0.009 mmol) was stirred at rt for 6 h. The resulting mixture was concentrated *in vacuo* and the residual crude material was triturated with ice-cold ether (× 3) to furnish an off-white solid. The crude material was purified by *via* semi-preparative RP-HPLC to afford compound **1** (5.2 mg, 85%) as a white solid. MS (ESI^+) for $C_{30}H_{37}N_6O_8S$ *m/z* 641.56 (M + 1, 100%); HRMS (ESI^+); calculated 641.2315, found 641.2309. RP-HPLC Onyx Monolithic C18 100 × 4.6 mm, 10-100% B in 15 min, t_R 6.08 min. ^1H NMR (300 MHz, DMSO) δ 9.13 (s, 1H), 8.27 (d, *J* = 7.6 Hz, 1H), 8.10 (d, *J* = 7.7 Hz, 1H), 7.95 (d, *J* = 7.8 Hz, 1H), 7.93 – 7.88 (m, 2H), 7.43 (s, 1H), 7.30 – 7.14 (m, 5H), 6.98 (d, *J* = 8.4 Hz, 3H), 6.60 (d, *J* = 8.4 Hz, 2H), 4.49 (q, *J* = 6.7 Hz, 1H), 4.42 – 4.27 (m, 3H), 4.15 – 4.02 (m, 1H), 3.05 (d, *J* = 5.9 Hz, 1H), 3.00 (d, *J* = 5.8 Hz, 1H), 2.95 – 2.79 (m, 3H), 2.79 – 2.50 (m, 5H), 2.46 – 2.38 (m, 1H), 2.30 (t, *J* = 8.4 Hz, 1H), 1.84 (d, *J* = 3.4 Hz, 3H), 1.07 (d, *J* = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, DMSO) δ 172.98, 172.35, 172.23, 172.04, 171.38, 171.09, 170.95, 170.32, 170.12, 156.09, 137.77, 130.52, 129.55, 128.64, 128.35, 126.87, 115.27, 55.51, 54.63, 53.97, 50.26, 49.14, 37.29, 37.19, 36.72, 26.57, 22.92, 18.23.



Data File C:\CHEM32\...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\046-3201.D
Sample Name: Tr S lug AIP I

Acq. Operator : Simi120102015 Seq. Line : 32
Acq. Instrument : LC1260 Location : Vial 46
Injection Date : 12/11/2015 9:11:57 AM Inj : 1
Inj Volume : 100.000 µl
Acq. Method : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\10 TO 100 OV 15MIN 100UL.M
Last changed : 12/10/2015 4:12:03 PM by Simi120102015
Analysis Method : C:\CHEM32\1\DATA\CPG170S AND PS CYCLISATION 2015-12-14 09-26-38\10 TO 100 OV 15MIN 5UL.M
Last changed : 12/14/2015 11:20:50 AM by Simi120102015 (modified after loading) (Current integration events modified)



Area Percent Report

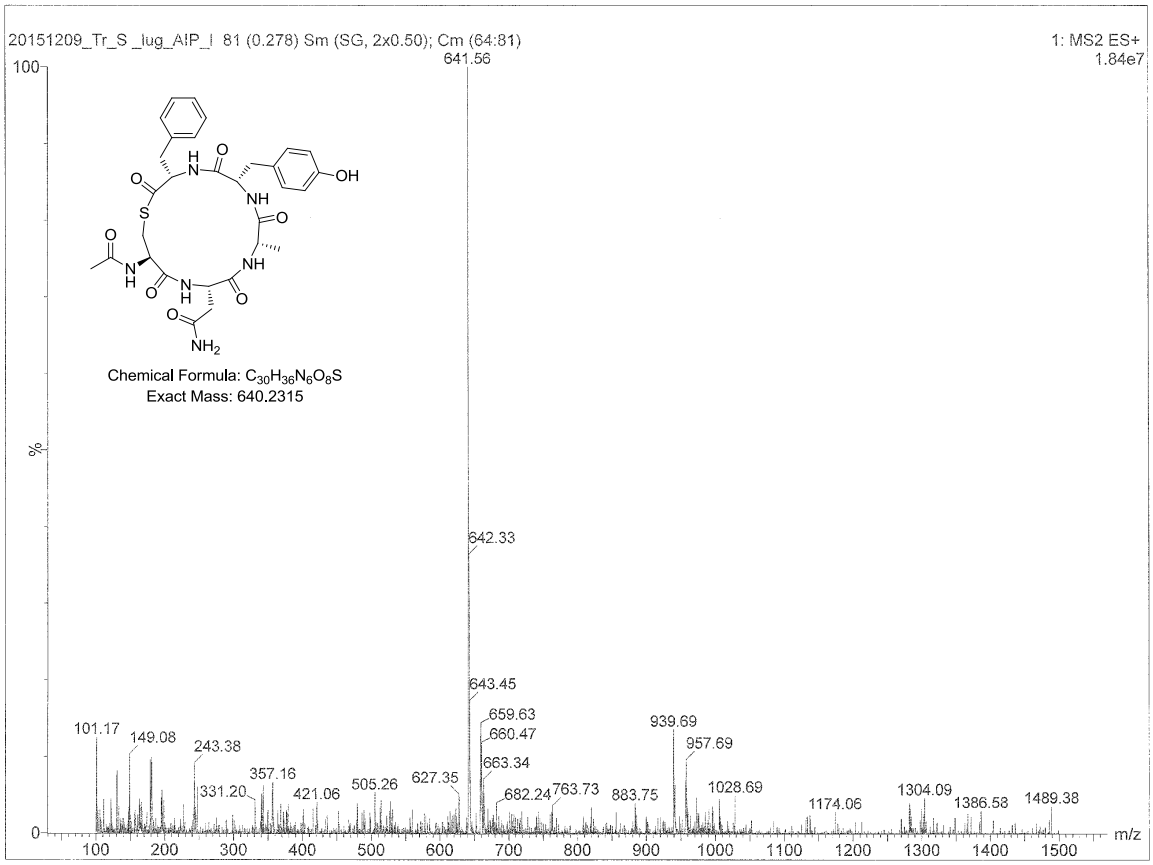
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.800	BV	0.1049	1785.58350	251.35371	100.0000
Totals :				1785.58350	251.35371	

Signal 2: DAD1 B, Sig=216,4 Ref=off

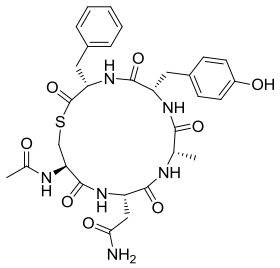
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.800	BV	0.1102	1.96317e4	2657.20728	100.0000


20151209_Tr_S _lug_AIP_I 81 (0.278) Sm (SG, 2x0.50); Cm (64:81)
1: MS2 ES+
1.84e7
641.56
100
%
0
642.33
643.45
659.63
660.47
663.34
939.69
957.69
101.17
149.08
243.38
357.16
331.20
421.06
505.26
627.35
682.24
763.73
883.75
1028.69
1174.06
1304.09
1386.58
1489.38
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
1500
m/z
Chemical Formula: $C_{30}H_{36}N_6O_8S$
Exact Mass: 640.2315

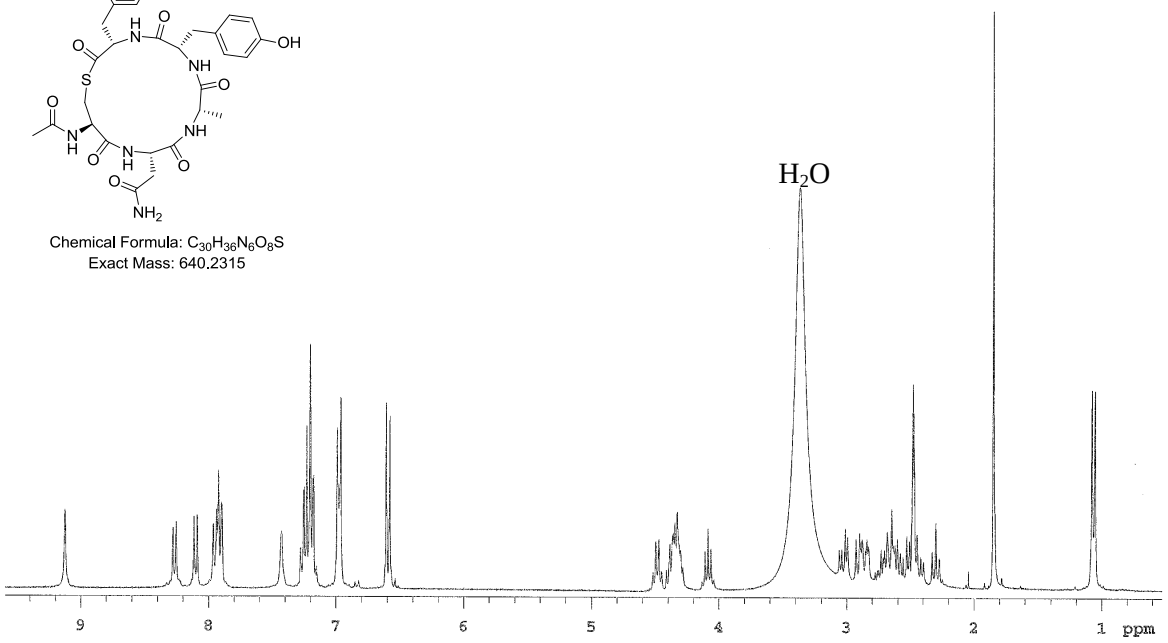
tr_S_Lug_AIPI_H1

Agilent Technologies

Sample Name	tr_S_Lug_AIPI_H1	Pulse sequence	PROTON	Temperature	25	Study owner	nmr300
Date collected	2016-03-10	Solvent	dmso	Spectrometer	uwsm300.uws.edu.au-mercury300	Operator	nmr300

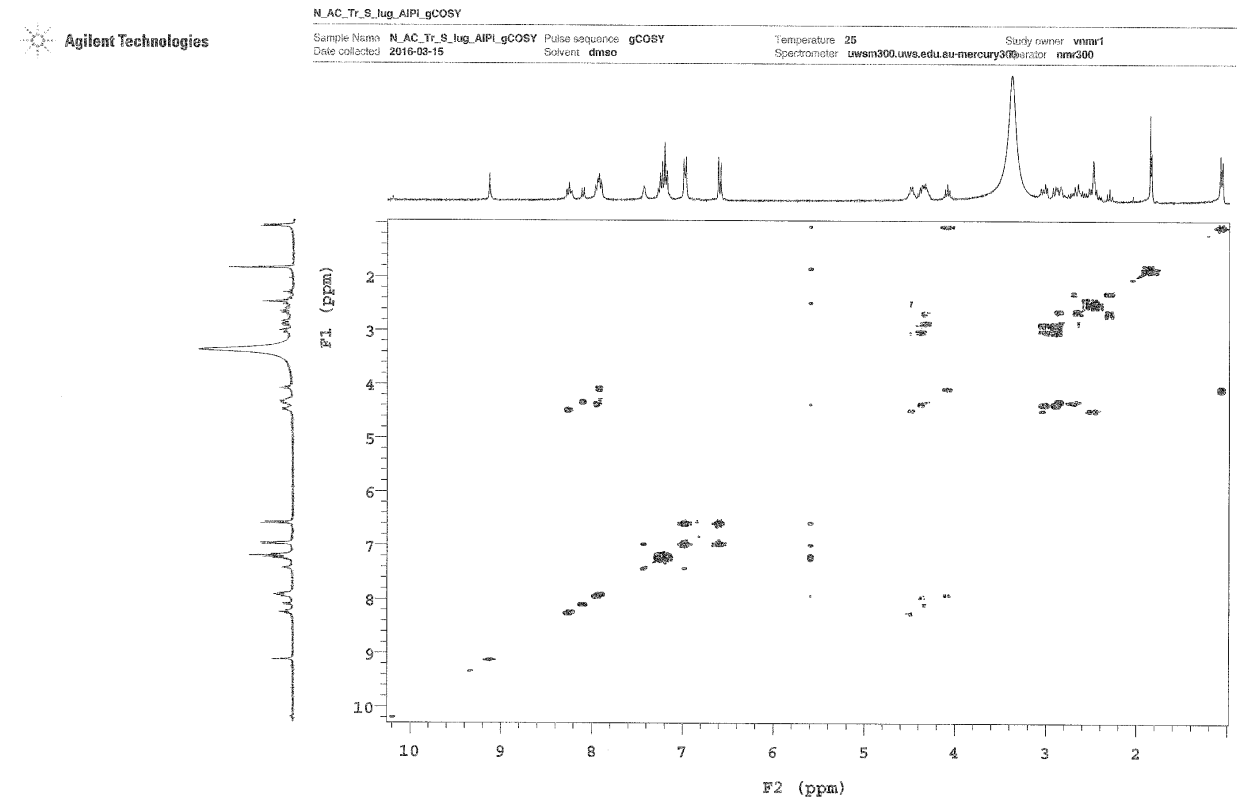


Chemical Formula: $C_{30}H_{36}N_6O_8S$
Exact Mass: 640.2315



Data file /home/nmr300/vnmrsys/data/CpgData/H_Ac_tr_S_lug_AIPI_H1.fid

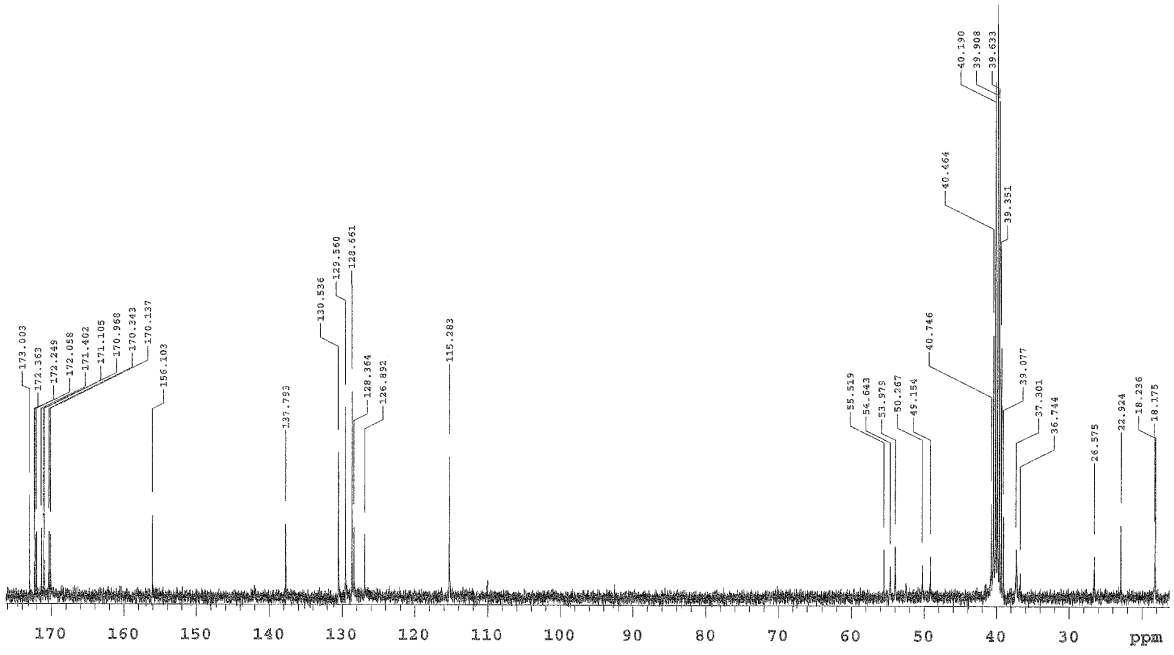
Plot date 2016-04-05


Agilent Technologies
N_AC_Tr_S_lug_AIPI_gCOSY
Sample Name N_AC_Tr_S_lug_AIPI_gCOSY Pulse sequence gCOSY
Date collected 2016-03-15 Solvent dmso
Temperature 25
Spectrometer uwsm300.uws.edu.au-mercury300
Study owner vnmr1
Operator nmr300
F1 (ppm)
F2 (ppm)
Data file /home/nmr300/vnmrsys/data/N_AC_Tr_S_lug_AIPI_gCOSY/N_AC_Tr_S_lug_AIPI_gCOSY_gCOSY_dmso_20160315_01.fid
Plot date 2016-03-15

Agilent Technologies

N_AC_Tr_S_Iug_AIPI_H1

Sample Name N_AC_Tr_S_Iug_AIPI_H1 | Pulse sequence CARBON | Temperature 25 | Study owner vnmr1
Date collected 2016-03-15 | Solvent dmso | Spectrometer uwsm300.uws.edu.au-mercury300 | Operator nmr300



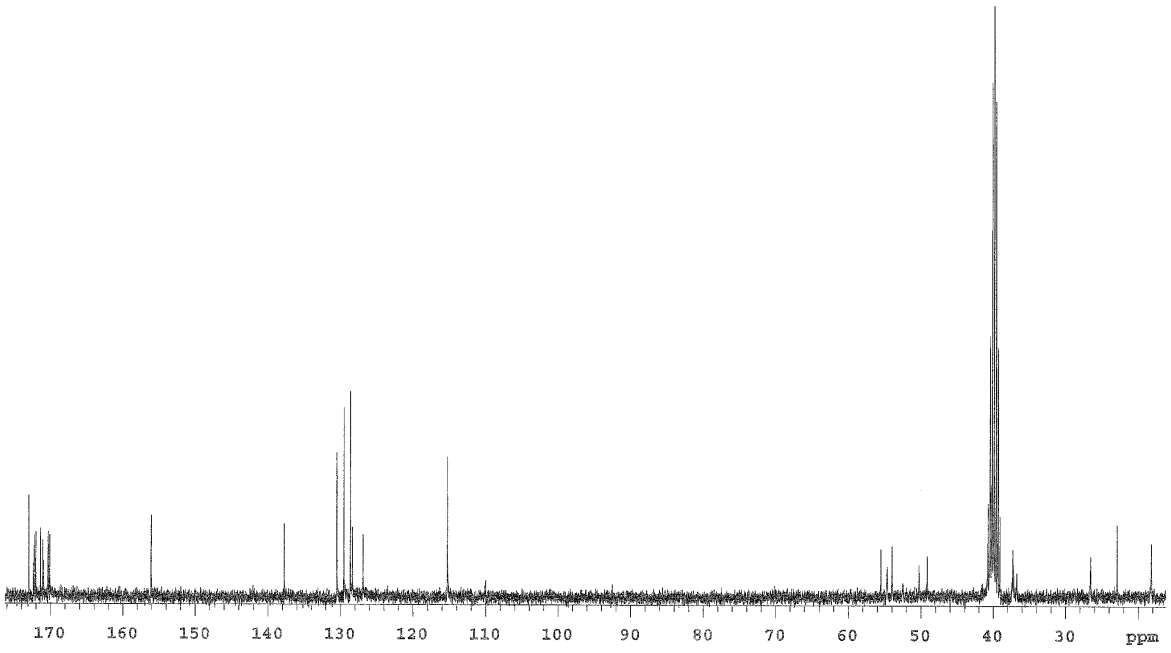
Data file /home/nmr300/vnmrsys/data/OpgData/N_AC_Tr_S_Iug_AIPI_C13.fid

Plot date 2016-04-05

Agilent Technologies

N_AC_Tr_S_lug_AIPI_H1

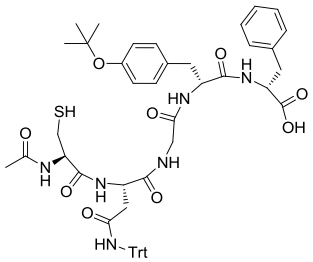
Sample Name N_AC_Tr_S_lug_AIPI_H1	Pulse sequence CARBON	Temperature 25	Study owner vnmr1
Date collected 2016-03-15	Solvent dmso	Spectrometer uwsm300.uws.edu.au-mercury300	Operator nmr300



Data file /home/nmr300/vnmrsys/data/CpgData/N_AC_Tr_S_lug_AIPI_C13.fid

Plot date 2016-04-05

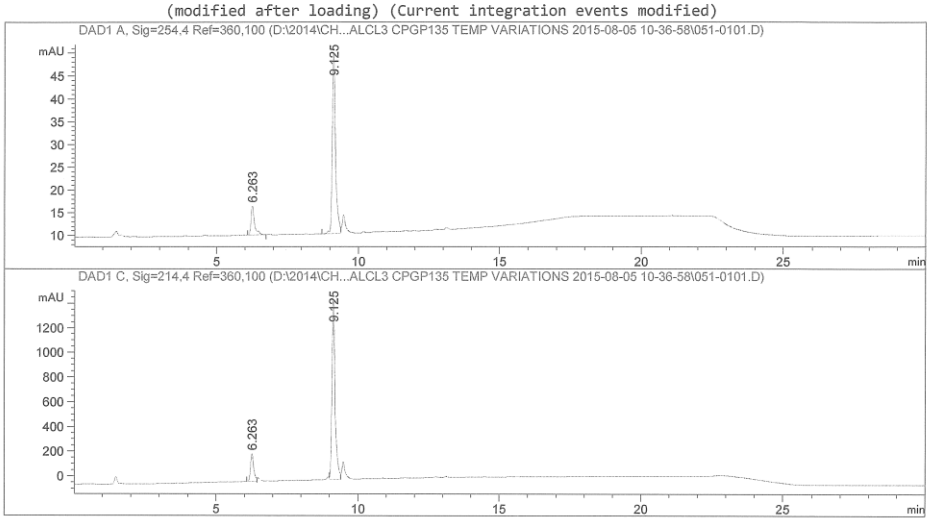
Compounds 11 & 2; The linear sequence required for **compound 11** was prepared using general procedure 2. The residual crude material was triturated with ice-cold ether (× 3) to furnish an off-white solid. MS (ESI^+) for $C_{52}H_{59}N_6O_9S$; calculated 943.40, found, 943.51. RP-HPLC Onyx Monolithic C18 100 × 4.6 mm, 40-100% B in 15 min, t_R 9.1 min.



Chemical Formula: $C_{52}H_{58}N_6O_9S$
Exact Mass: 942.3986

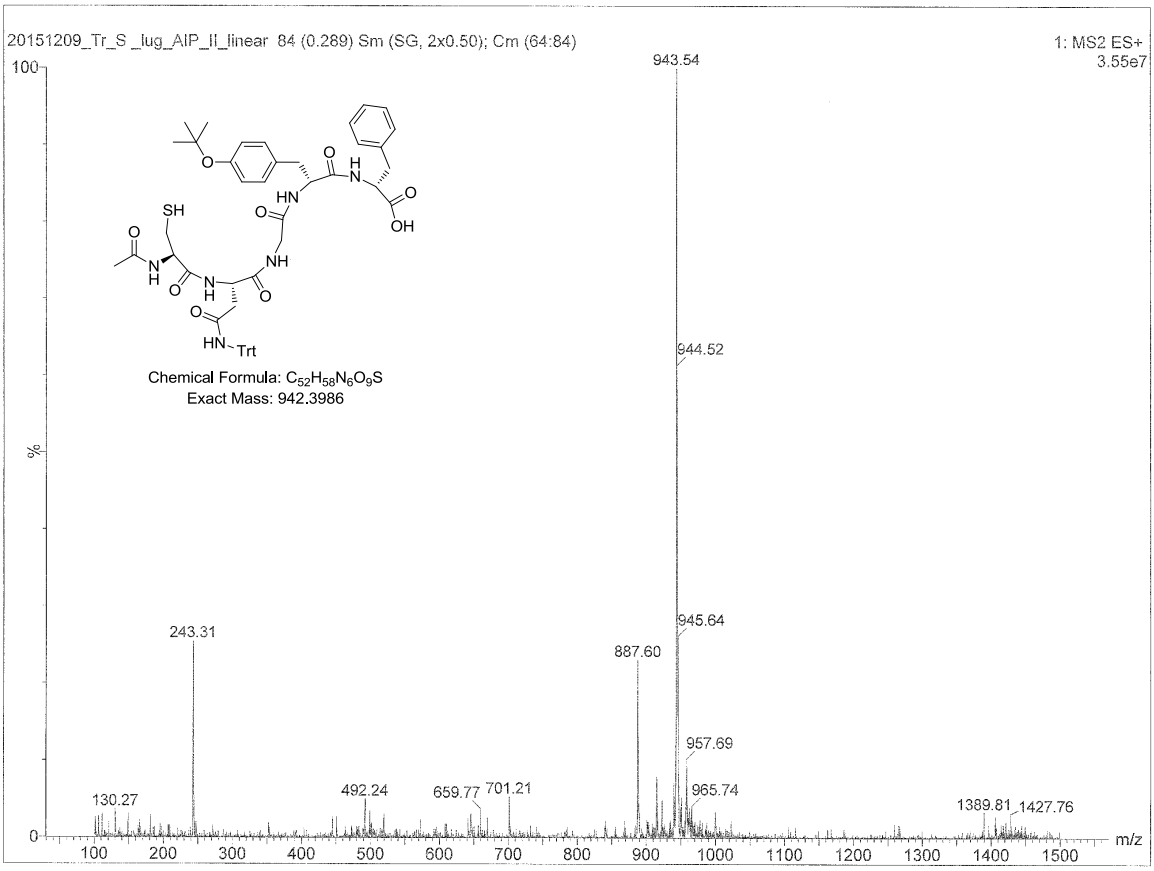
Data File D:\2014\CH...POUNDS\ALCL3 CPGP135 TEMP VARIATIONS 2015-08-05 10-36-58\051-0101.D
Sample Name: Ac-CNGYF-OH uncyclised

```
======================================================================
Acq. Operator   : SYSTEM                       Seq. Line :   1
Acq. Instrument : LC1260                       Location : Vial 51
Injection Date  : 8/5/2015 10:38:23 AM             Inj :   1
                                              Inj Volume : 10.000 µl
Method          : D:\2014\CHRIS\DATA\DDCOPPERCOMPOUNDS\ALCL3 CPGP135 TEMP VARIATIONS 2015-08-
                  05 10-36-58\40 TO 100 OVER 15 MINS 10UL.M (Sequence Method)
Last changed    : 8/5/2015 10:36:58 AM by SYSTEM
                  (modified after loading) (Current integration events modified)
```

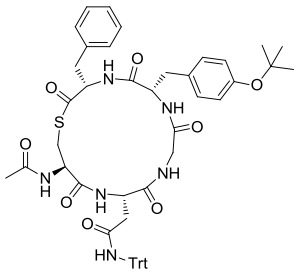



```
================================================================================
                          Fraction Information
================================================================================
================================================================================
No Fractions found.
================================================================================
======================================================================
                       Area Percent Report
======================================================================

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


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.263 BB    0.1276   55.50295    6.37214  15.1200
   2   9.125 BV    0.1160  311.57983   39.52258  84.8800
```

LC1260 8/5/2015 12:33:41 PM SYSTEM Page 1 of 2


20151209_Tr_S _lug_AIP_II_linear 84 (0.289) Sm (SG, 2x0.50); Cm (64:84)
1: MS2 ES+
3.55e7
100
%
0
943.54
944.52
945.64
887.60
243.31
957.69
965.74
130.27
492.24
659.77
701.21
1389.81
1427.76
m/z
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
1500
Chemical Formula: C52H58N6O9S
Exact Mass: 942.3986

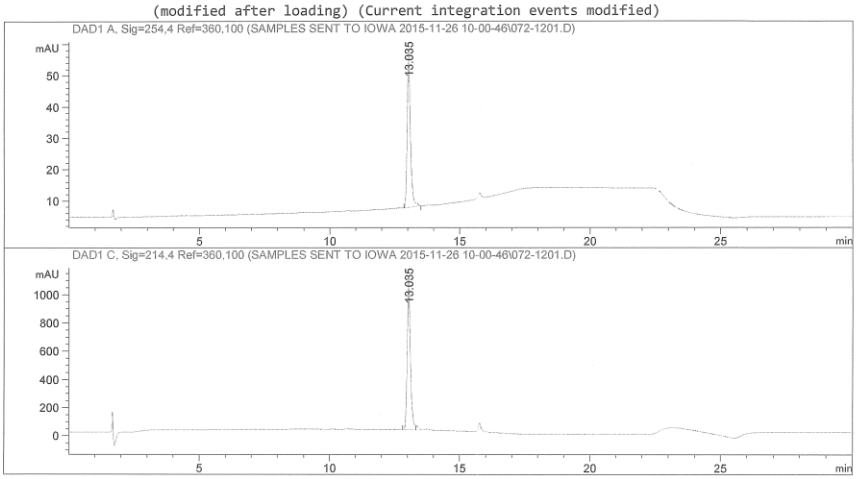
Compound 11; Compound **11** was synthesised utilizing general produce 3. The crude material was purified *via* semi-preparative RP-HPLC, and lyophilised to afford **11** (15 mg, 13.5 %). (*Note; 3 mg of this sample was collect for biological analysis*) MS (ESI^+) for $C_{52}H_{57}N_6O_8S$ m/z 925.2 (M + 1, 100%); HRMS (ESI^+); calculated 925.3880, found, 925.3497. RP-HPLC Onyx Monolithic C18 100 × 4.6 mm, 10-100% B in 15 min, t_R 13.03 min.



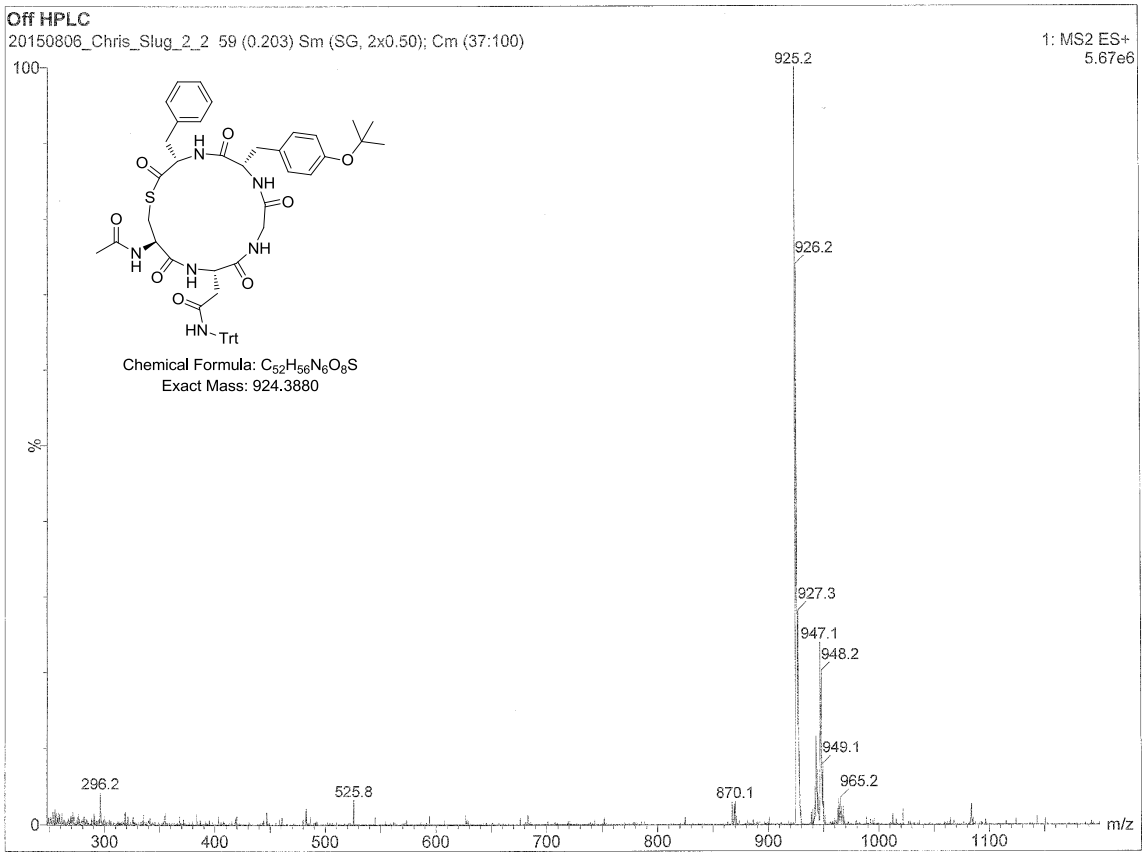
Chemical Formula: $C_{52}H_{56}N_6O_8S$
Exact Mass: 924.3880

Data File C:\CHEM32\1\DATA\SAMPLES SENT TO IOWA 2015-11-26 10-00-46\072-1201.D
Sample Name: Pro S lug AIP II cyclised

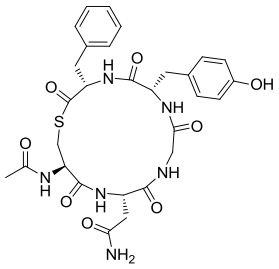
```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :  12
Acq. Instrument : LC1260                        Location : Vial 72
Injection Date  : 11/26/2015 3:49:03 PM              Inj :   1
                                               Inj Volume : 20.000 µl
Acq. Method     : C:\CHEM32\1\DATA\SAMPLES SENT TO IOWA 2015-11-26 10-00-46\10 TO 100 OVER 15
                  MINS 20UL.M
Last changed    : 11/26/2015 10:00:46 AM by SYSTEM
Analysis Method : C:\CHEM32\1\DATA\SAMPLES SENT TO IOWA 2015-11-26 10-00-46\10 TO 100 OVER 15
                  MINS 20UL.M (Sequence Method)
Last changed    : 12/1/2015 10:41:51 AM by SYSTEM
                  (modified after loading) (Current integration events modified)
```



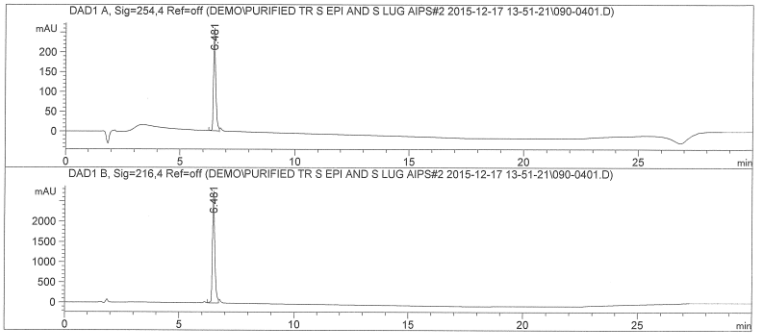

```
================================================================================
                          Fraction Information
================================================================================
================================================================================
No Fractions found.
================================================================================
======================================================================
                        Area Percent Report
======================================================================

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


Off HPLC
20150806_Chris_Slug_2_2 59 (0.203) Sm (SG, 2x0.50); Cm (37:100)
1: MS2 ES+
5.67e6
Chemical Formula: C52H56N6O8S
Exact Mass: 924.3880
925.2
926.2
927.3
947.1
948.2
949.1
965.2
296.2
525.8
870.1
100
%
0
300
400
500
600
700
800
900
1000
1100
m/z

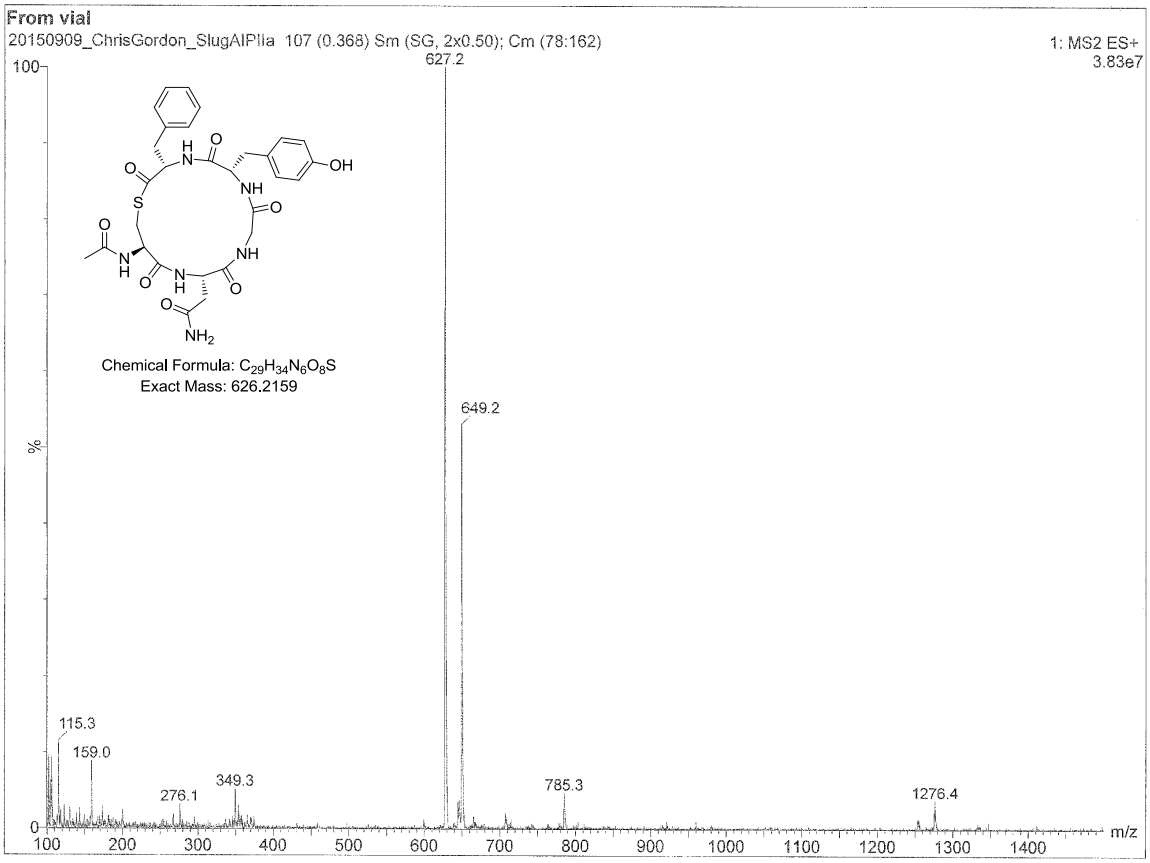
Compound 2; Compound **2** was prepared utilizing general procedure 4 with compound **11**. The crude material was purified *via* semi-preparative RP-HPLC, and lyophilised to afford **2** (7.2 mg, 88.5 %). MS (ESI^+) for $C_{52}H_{57}N_6O_8S$, *m/z* 627.2 (M + 1, 100%); HRMS (ESI^+); calculated 627.2159, found, 627.2204. RP-HPLC Onyx Monolithic C18 100 × 4.6 mm, 10-100% B in 15 min, t_R 6.48 min. ^1H NMR (300 MHz, DMSO) δ 9.12 (s, 1H), 8.28 (d, *J* = 7.5 Hz, 1H), 8.17 (d, *J* = 7.8 Hz, 1H), 8.09 (d, *J* = 7.8 Hz, 1H), 7.97 (t, *J* = 5.5 Hz, 1H), 7.89 (d, *J* = 8.6 Hz, 1H), 7.39 (s, 1H), 7.28 – 7.16 (m, 5H), 6.98 (d, *J* = 8.5 Hz, 2H), 6.59 (d, *J* = 8.5 Hz, 2H), 4.47 (dd, *J* = 13.6, 7.0 Hz, 1H), 4.42 – 4.30 (m, 3H), 3.58 (t, *J* = 5.9 Hz, 2H), 3.06 (d, *J* = 5.5 Hz, 1H), 3.01 (d, *J* = 5.4 Hz, 1H), 2.92 (d, *J* = 8.7 Hz, 1H), 2.89 – 2.52 (m, 7H), 2.43 (d, *J* = 7.0 Hz, 1H), 2.29 (t, *J* = 8.5 Hz, 1H), 1.85 (s, 3H). ^{13}C NMR (75 MHz, DMSO) δ 173.08, 172.07, 171.57, 171.46, 170.44, 170.15, 168.63, 156.09, 137.84, 130.55, 129.54, 128.65, 128.35, 126.89, 115.28, 55.50, 54.54, 54.51, 54.00, 50.39, 42.41, 37.26, 37.14, 37.04, 26.49, 22.92.

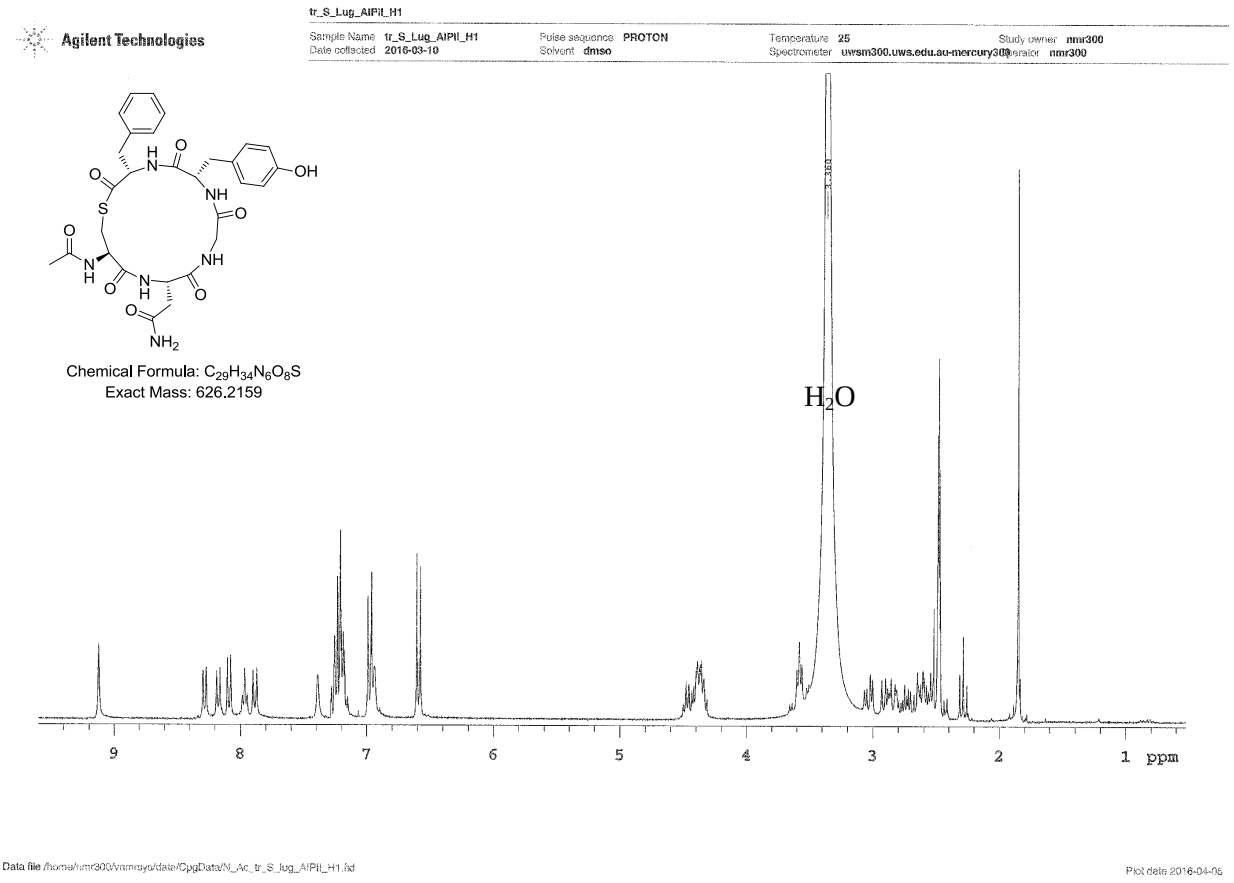


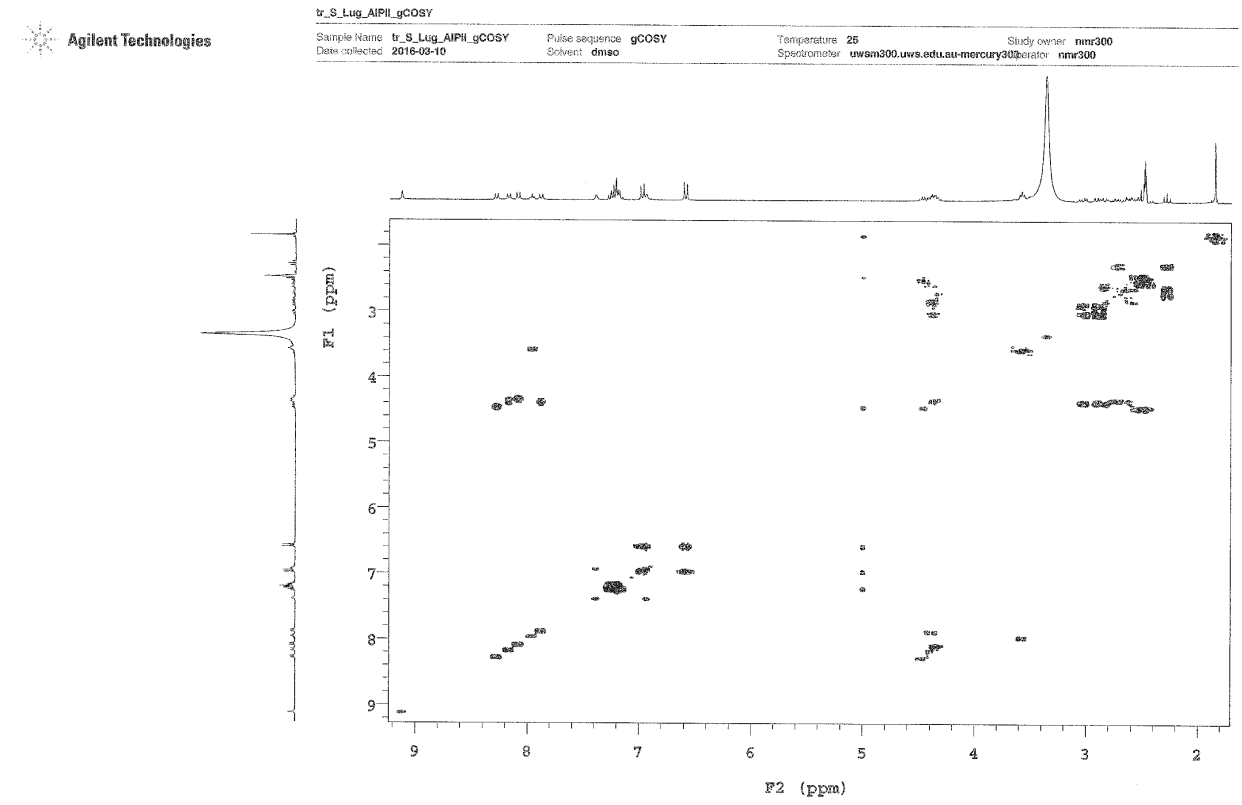

```
Data File C:\CHEM32\...O\PURIFIED TR S EPI AND S LUG AIPS#2 2015-12-17 13-51-21\090-0401.D
Sample Name: Tr S lug AIP II 6.4 min
    ======================================================================
    Acq. Operator   : Simi120102015                  Seq. Line :   4
    Acq. Instrument : LC1260                        Location : Vial 90
    Injection Date  : 12/17/2015 3:28:30 PM              Inj :   1
                                                   Inj Volume : 100.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\PURIFIED TR S EPI AND S LUG AIPS#2 2015-12-17 13-
                      51-21\10 TO 100 OV 15MIN 100UL.M
    Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
    Analysis Method : C:\CHEM32\1\DATA\DEMO\PURIFIED TR S EPI AND S LUG AIPS 2016-01-05 09-
                      33-14\10 TO 100 OV 15MIN 20UL.M
    Last changed    : 1/6/2016 1:51:31 PM by Simi120102015
                      (modified after loading) (Current integration events modified)
    ======================================================================
```

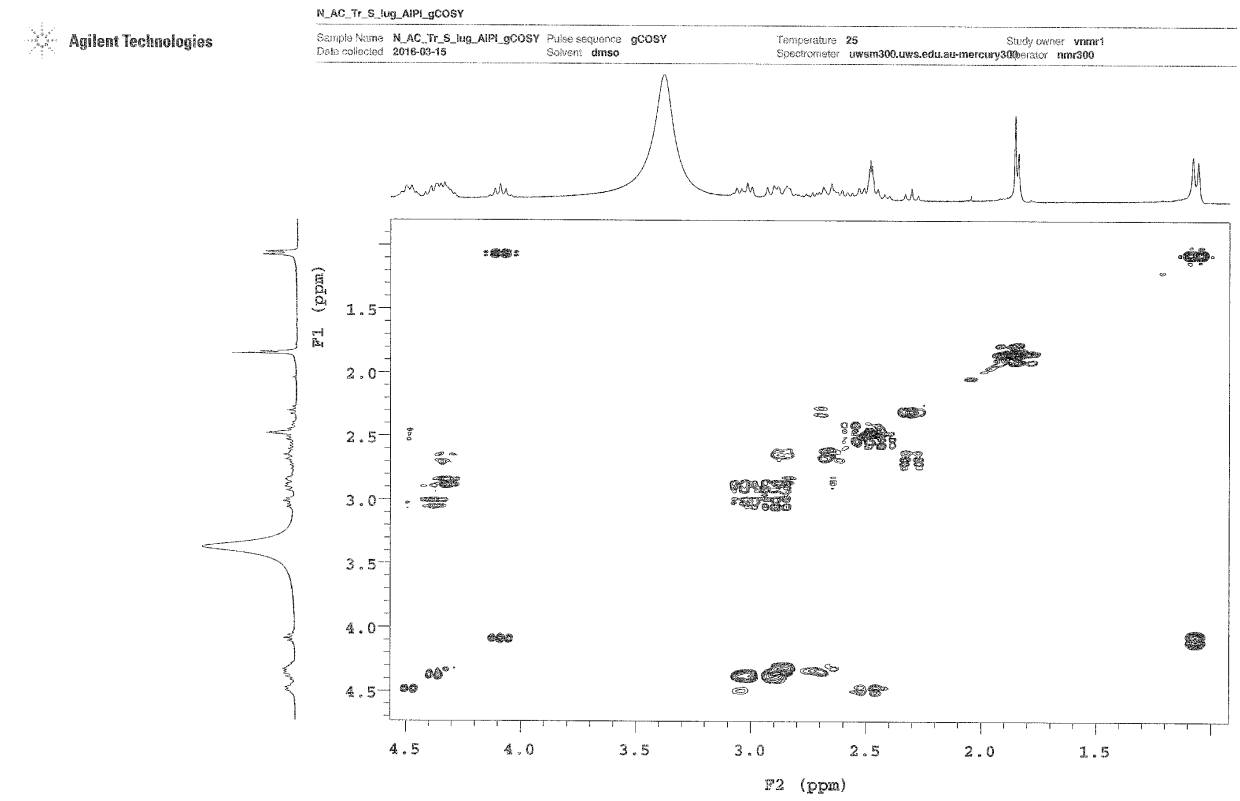



```
    ======================================================================
                              Area Percent Report
    ======================================================================

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]    [mAU*s]     [mAU]      %
    ----|-------|----|-------|----------|----------|--------|
       1   6.481 BV    0.1004 1733.96338  257.95151 100.0000

    Totals :                   1733.96338  257.95151


    Signal 2: DAD1 B, Sig=216,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]    [mAU*s]     [mAU]      %
    ----|-------|----|-------|----------|----------|--------|
       1   6.481 VV    0.1084 1.99227e4  2753.27295 100.0000

LC1260 1/6/2016 3:03:38 PM Simi120102015                    Page   1 of 2
```


From vial
20150909_ChrisGordon_SlugAIPIia 107 (0.368) Sm (SG, 2x0.50); Cm (78:162)
1: MS2 ES+
3.83e7
627.2
649.2
115.3
159.0
276.1
349.3
785.3
1276.4
Chemical Formula: C29H34N6O8S
Exact Mass: 626.2159
%
m/z

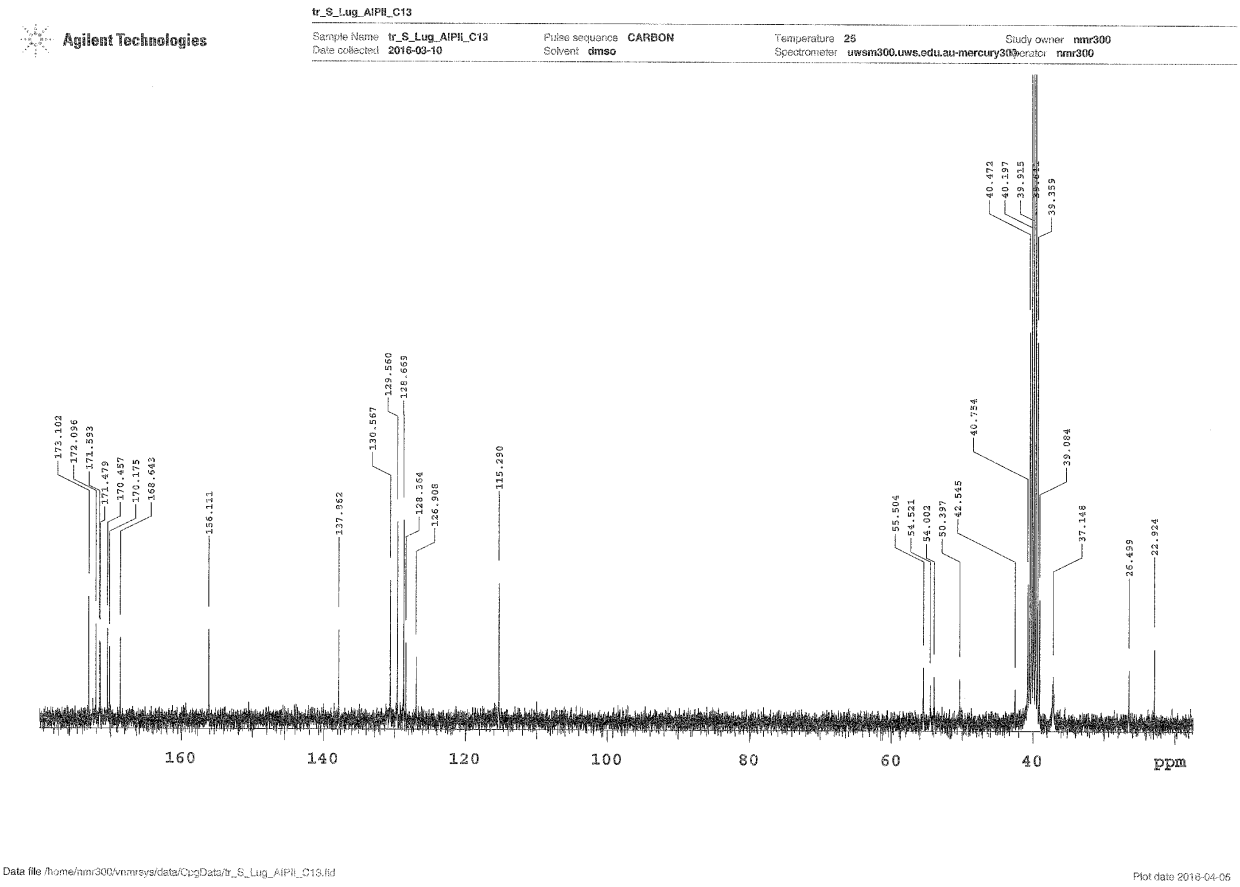

tr_S_Lug_AlPil_H1
Agilent Technologies
Sample Name tr_S_Lug_AlPil_H1
Date collected 2016-03-10
Pulse sequence PROTON
Solvent dmso
Temperature 25
Spectrometer uwsm300.uws.edu.au-mercury300
Study owner nmr300
Operator nmr300
Chemical Formula: C29H34N6O8S
Exact Mass: 626.2159
H2O
3.360
9
8
7
6
5
4
3
2
1 ppm
Data file /home/nmr300/vnmrsys/data/CpgData/N_Ac_tr_S_lug_AlPil_H1.fid
Plot date 2016-04-05


tr_S_Lug_AlPII_gCOSY
Agilent Technologies
Sample Name tr_S_Lug_AlPII_gCOSY
Date collected 2016-03-10
Pulse sequence gCOSY
Solvent dmso
Temperature 25
Spectrometer uwsm300.uws.edu.au-mercury300
Study owner nmr300
Operator nmr300
F1 (ppm)
F2 (ppm)
Data file /home/nmr300/vnmrsys/data/tr_S_Lug_AlPII_gCOSY/tr_S_Lug_AlPII_gCOSY_gCOSY_dmso_20160310_01.fid
Plot date 2016-03-10



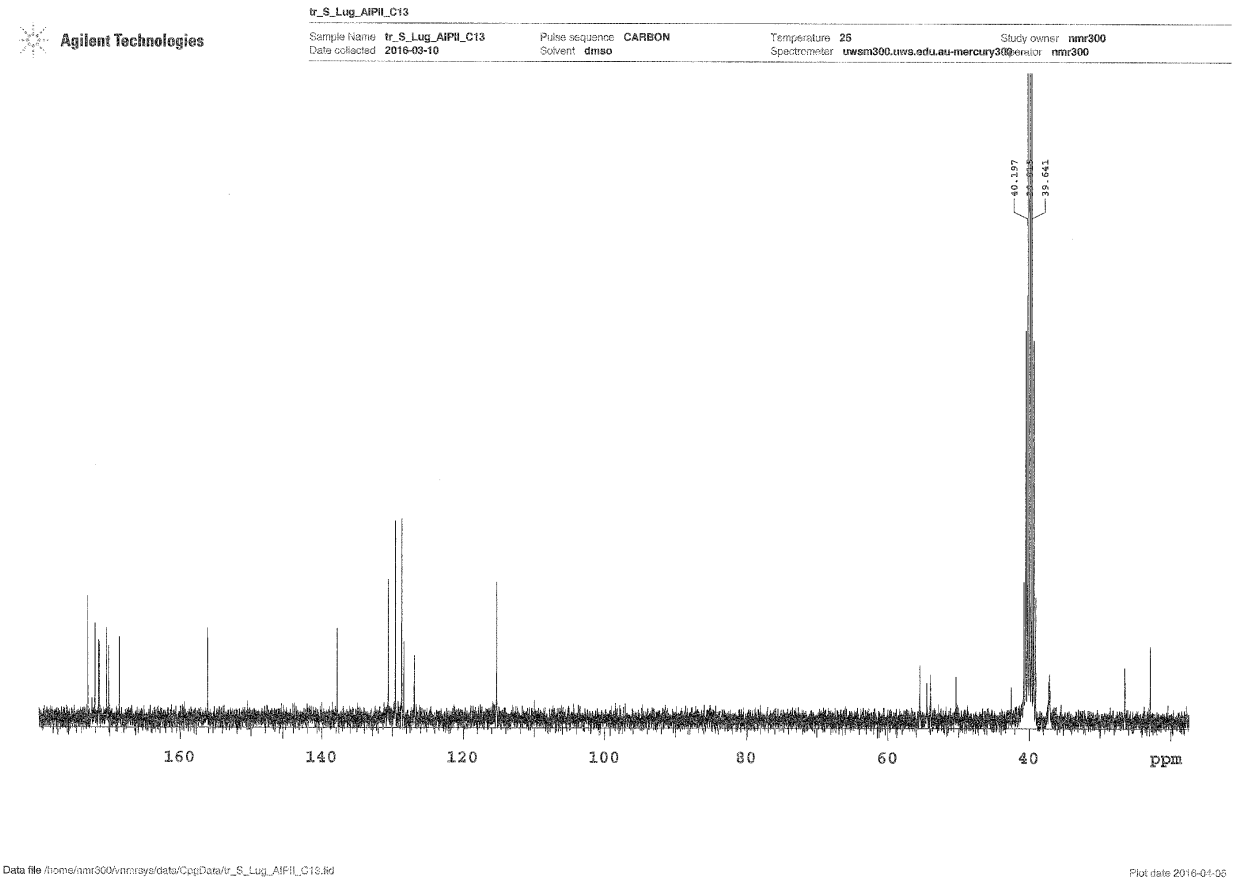
Data file /home/nmr300/vnmrsys/data/N_AC_Tr_S_lug_AIPI_gCOSY/N_AC_Tr_S_lug_AIPI_gCOSY_gCOSY_dmso_20160315_01.fid

Plot date 2016-03-15

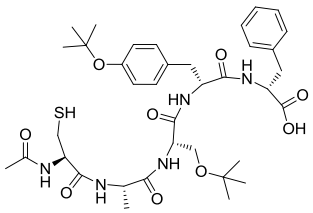


Data file /home/nmr300/vnmrsys/data/CpgData/tr_S_Lug_AlPli_C13.fid

Plot date 2016-04-06


tr_S_Lug_AlPII_C13
Agilent Technologies
Sample Name tr_S_Lug_AlPII_C13
Date collected 2016-03-10
Pulse sequence CARBON
Solvent dmso
Temperature 26
Spectrometer uwsm300.uws.edu.au-mercury300
Study owner nmr300
Operator nmr300
40.197
39.919
39.641
160
140
120
100
80
60
40
ppm
Data file /home/nmr300/vnmrsys/data/CpgData/tr_S_Lug_AlPII_C13.fid
Plot date 2016-04-05

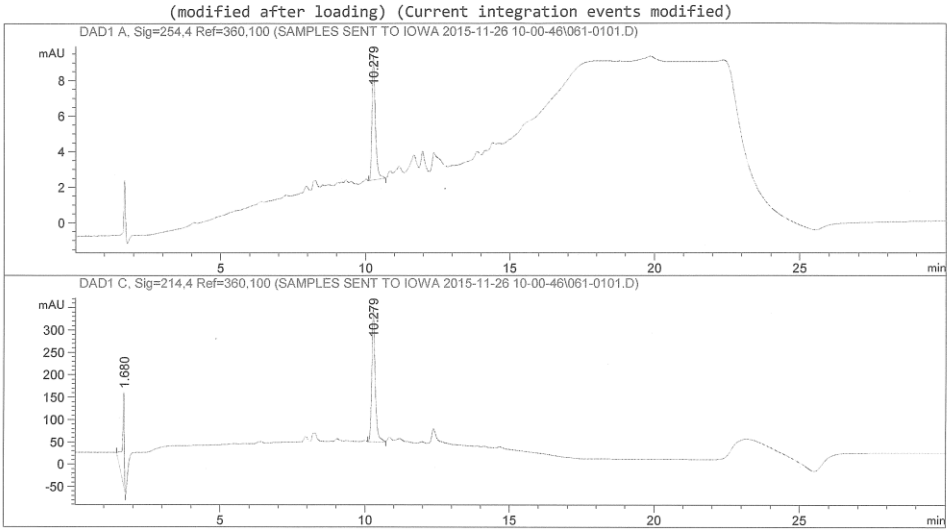
Compounds 12 & 3; The linear sequence required for **12** was prepared using general procedure 2. The residual crude material was triturated with ice-cold ether (× 3) to furnish an off-white solid. MS (ESI^+) for $C_{37}H_{54}N_5O_9S$; calculated 744.35, found, 744.55. RP-HPLC Onyx Monolithic C18 100 × 4.6 mm, 10-100% B in 15 min, t_R 10.28 min.



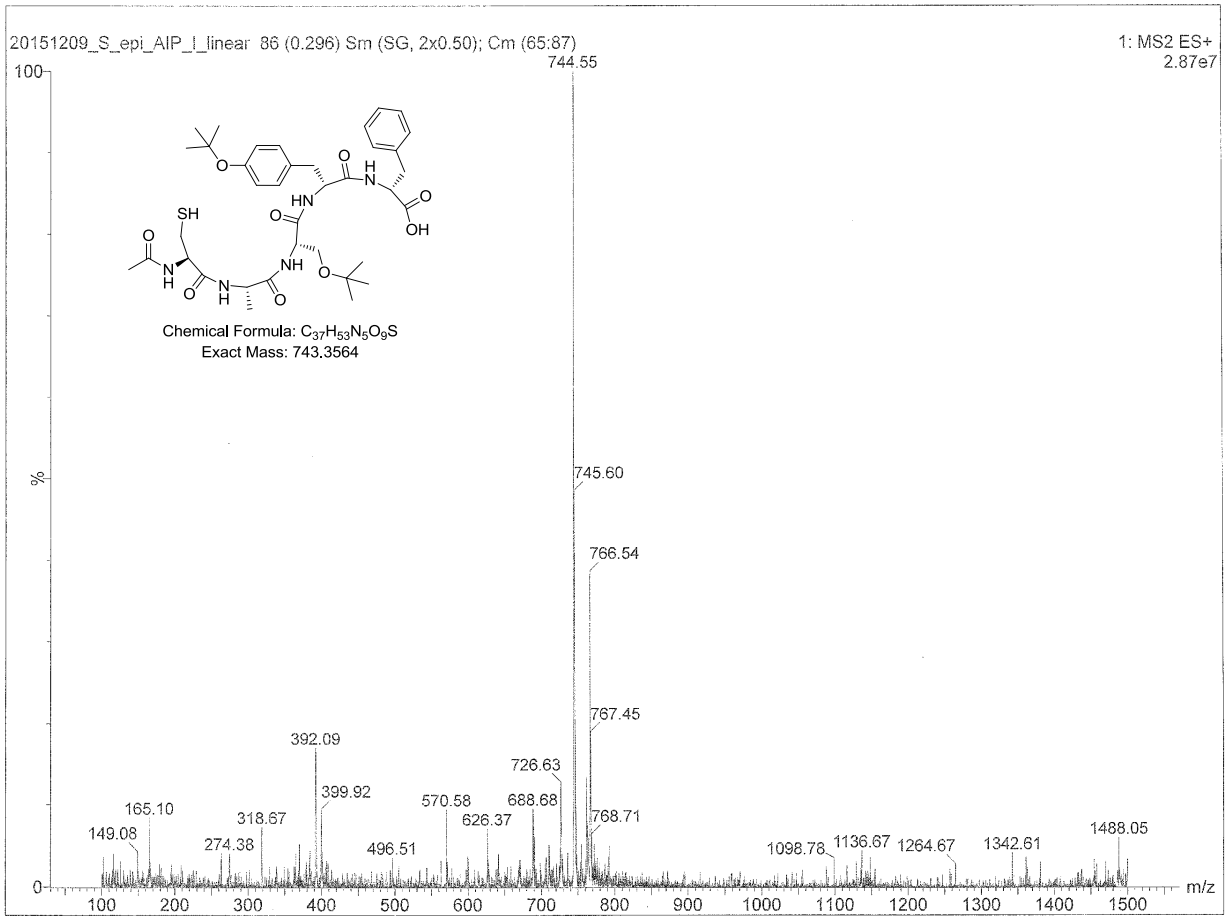
Chemical Formula: $C_{37}H_{53}N_5O_9S$
Exact Mass: 743.3564

Data File C:\CHEM32\1\DATA\SAMPLES SENT TO IOWA 2015-11-26 10-00-46\061-0101.D
Sample Name: S epi AIP I linear

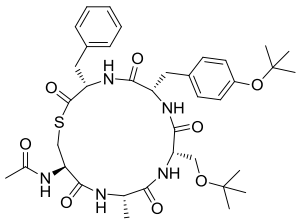
```
==========================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   1
Acq. Instrument : LC1260                        Location : Vial 61
Injection Date  : 11/26/2015 10:02:13 AM             Inj :   1
                                                Inj Volume : 20.000 µl
Method          : C:\CHEM32\1\DATA\SAMPLES SENT TO IOWA 2015-11-26 10-00-46\10 TO 100 OVER 15
                  MINS 20UL.M (Sequence Method)
Last changed    : 11/26/2015 10:00:46 AM by SYSTEM
                  (modified after loading) (Current integration events modified)
```




```
==========================================================================
                        Fraction Information
==========================================================================
==========================================================================
No Fractions found.
==========================================================================
==========================================================================
                        Area Percent Report
==========================================================================

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


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.279 BB    0.1253   58.45141    7.00503 100.0000

Totals :                    58.45141    7.00503
```


20151209_S_epi_AIP_I_linear 86 (0.296) Sm (SG, 2x0.50); Cm (65:87)
1: MS2 ES+
2.87e7
744.55
745.60
766.54
767.45
392.09
726.63
399.92
570.58
688.68
165.10
626.37
768.71
318.67
149.08
274.38
496.51
1098.78
1136.67
1264.67
1342.61
1488.05
Chemical Formula: C37H53N5O9S
Exact Mass: 743.3564
%
m/z

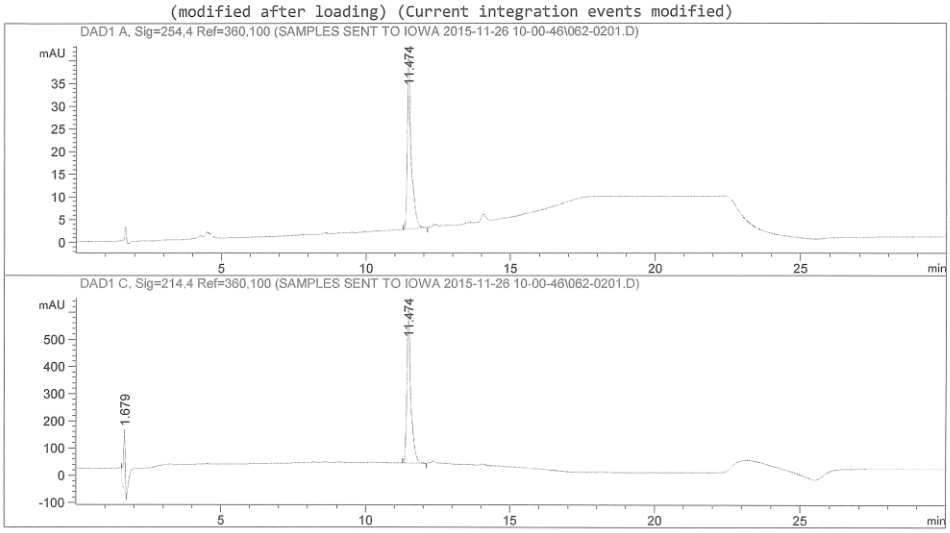
Compound 12; Compound **12** was synthesised utilizing general produce 3. The crude material was purified *via* semi-preparative RP-HPLC, and lyophilised to afford **12** (12 mg, 13.8 %). (*Note; 3 mg of this sample was collect for biological analysis*) MS (ESI^+) for $C_{37}H_{52}N_5O_8S$, *m/z* 726.63 (M + 1, 100%); HRMS (ESI^+) calculated 726.3458, found, 726.3455. RP-HPLC Onyx Monolithic C18 100 × 4.6 mm, 10-100% B in 15 min, t_R 11.47 min.



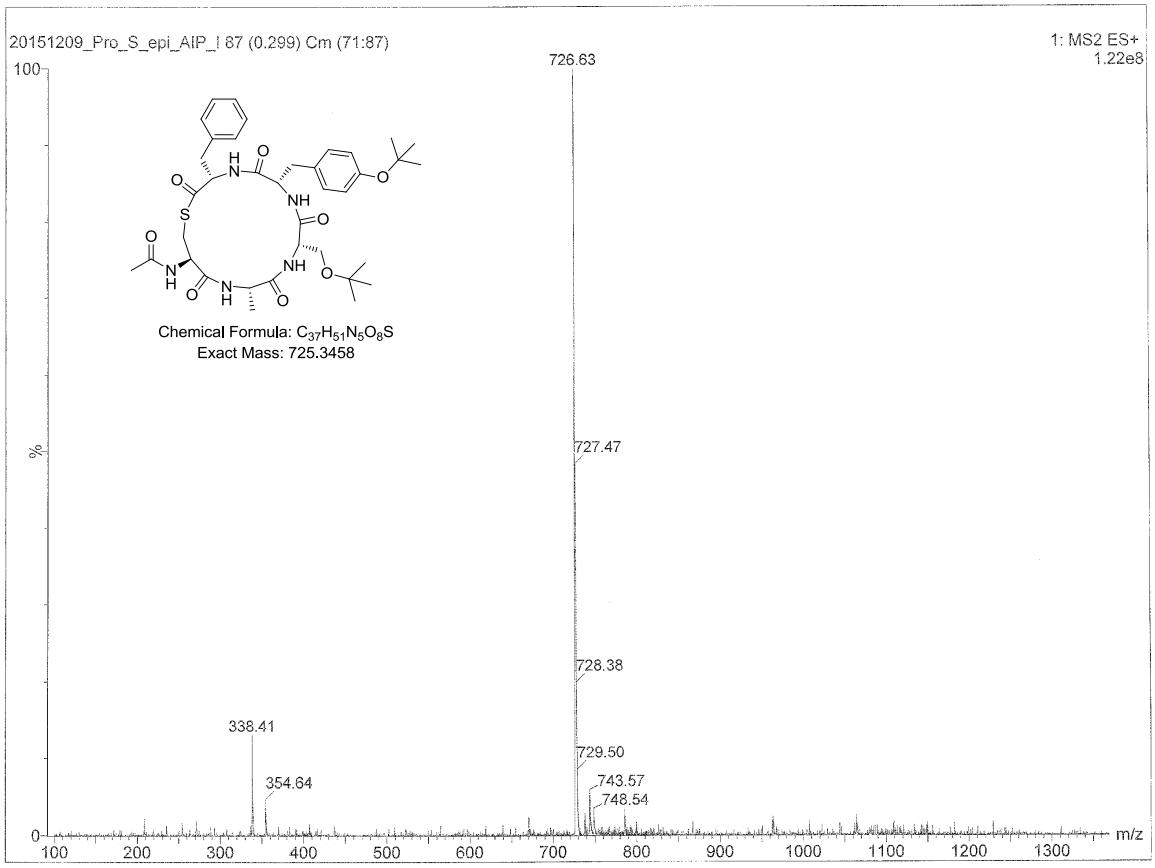
Chemical Formula: $C_{37}H_{51}N_5O_8S$
Exact Mass: 725.3458

Data File C:\CHEM32\1\DATA\SAMPLES SENT TO IOWA 2015-11-26 10-00-46\062-0201.D
Sample Name: Pro S epi AIP I cyclised

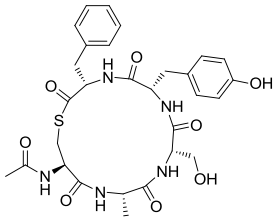
```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : LC1260                        Location : Vial 62
Injection Date  : 11/26/2015 10:33:47 AM             Inj :   1
                                               Inj Volume : 20.000 µl
Acq. Method     : C:\CHEM32\1\DATA\SAMPLES SENT TO IOWA 2015-11-26 10-00-46\10 TO 100 OVER 15
                  MINS 20UL.M
Last changed    : 11/26/2015 10:00:46 AM by SYSTEM
Analysis Method : C:\CHEM32\1\DATA\SAMPLES SENT TO IOWA 2015-11-26 10-00-46\10 TO 100 OVER 15
                  MINS 20UL.M (Sequence Method)
Last changed    : 12/1/2015 10:33:30 AM by SYSTEM
                  (modified after loading) (Current integration events modified)
```



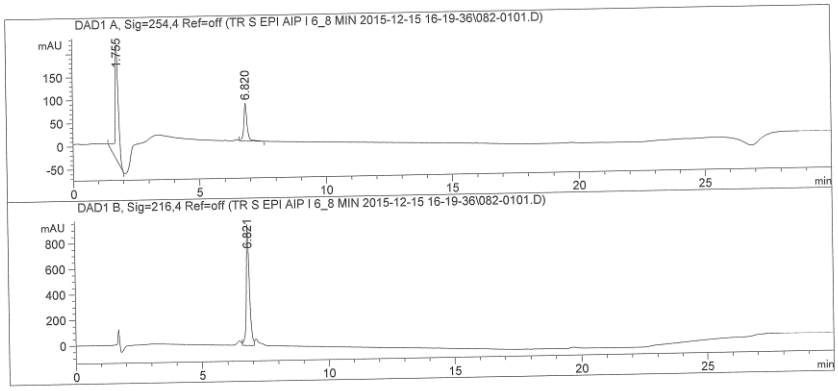

```
==========================================================================
                        Fraction Information
==========================================================================
==========================================================================
No Fractions found.
==========================================================================
======================================================================
                        Area Percent Report
======================================================================

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


20151209_Pro_S_epi_AIP_I 87 (0.299) Cm (71:87)
1: MS2 ES+
1.22e8
100
%
0
726.63
727.47
728.38
729.50
338.41
354.64
743.57
748.54
m/z
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
Chemical Formula: C37H51N5O8S
Exact Mass: 725.3458

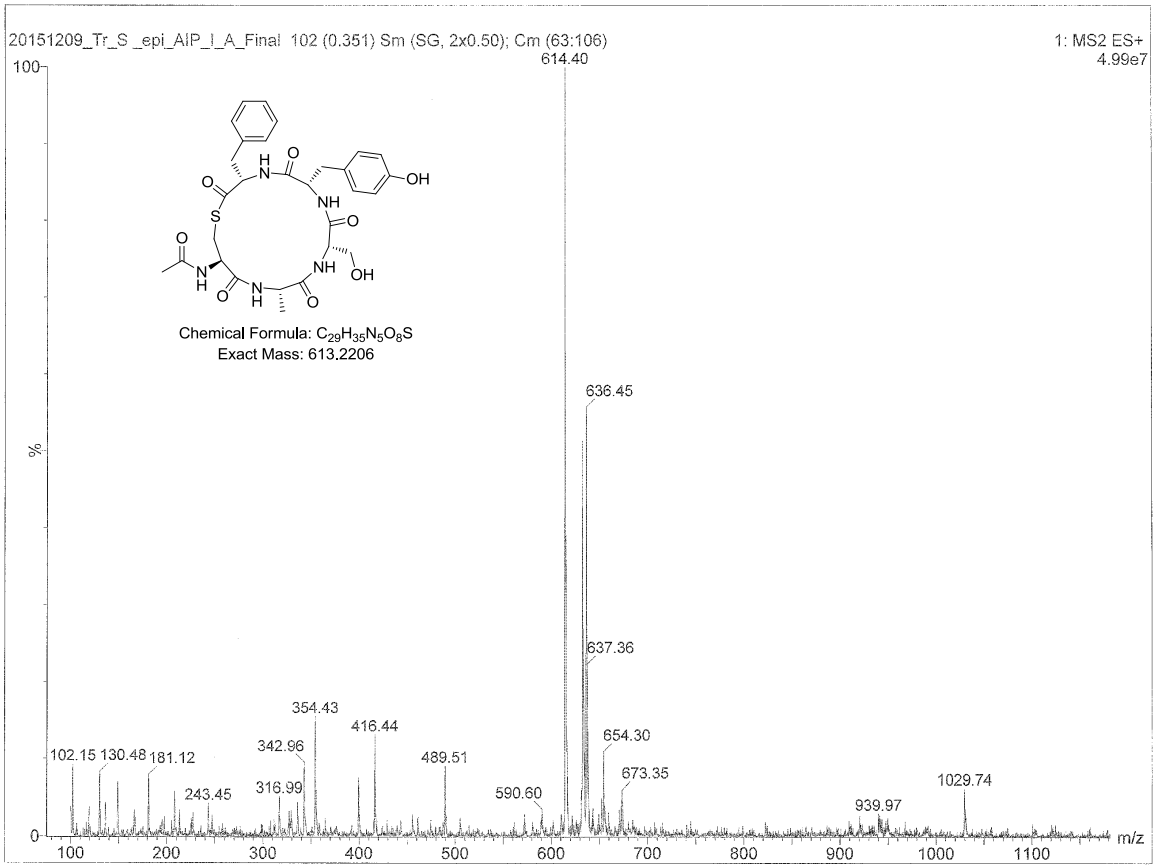
Compound 3; Compound **3** was prepared utilising general procedure 4 with **12**. The crude material was purified *via* semi-preparative RP-HPLC, and lyophilised to afford **3** (6.2 mg, 81.5 %). MS (ESI$^+$) for $C_{29}H_{36}N_5O_8S$ *m/z* 614.40; HRMS (ESI$^+$) calculated 614.2206, found, 614.2231. RP-HPLC Onyx Monolithic C18 100 × 4.6 mm, 10-100% B in 15 min, t_R 6.82 min. ^{1}H NMR (300 MHz, DMSO) δ 9.13 (s, 1H), 8.23 (d, *J* = 7.8 Hz, 1H), 8.15 (d, *J* = 7.1 Hz, 1H), 8.07 (d, *J* = 7.9 Hz, 1H), 7.82 (d, *J* = 7.7 Hz, 1H), 7.73 (d, *J* = 8.2 Hz, 1H), 7.29 – 7.17 (m, 5H), 6.95 (d, *J* = 8.5 Hz, 2H), 6.58 (d, *J* = 8.5 Hz, 2H), 4.45 – 4.30 (m, 3H), 4.30 – 4.16 (m, 2H), 3.05 (d, *J* = 5.5 Hz, 1H), 3.00 (d, *J* = 5.4 Hz, 1H), 2.93 – 2.81 (m, 3H), 2.80 – 2.53 (m, 4H), 2.32 (t, *J* = 8.4 Hz, 1H), 1.85 (s, 3H), 1.16 (d, *J* = 7.1 Hz, 3H). 13C NMR (75 MHz, DMSO) δ 173.05, 172.52, 171.26, 170.12, 170.00, 169.88, 156.14, 137.79, 130.57, 129.53, 128.66, 127.93, 126.90, 115.23, 62.12, 55.40, 54.31, 54.03, 48.77, 37.20, 36.89, 26.62, 22.92, 18.35.

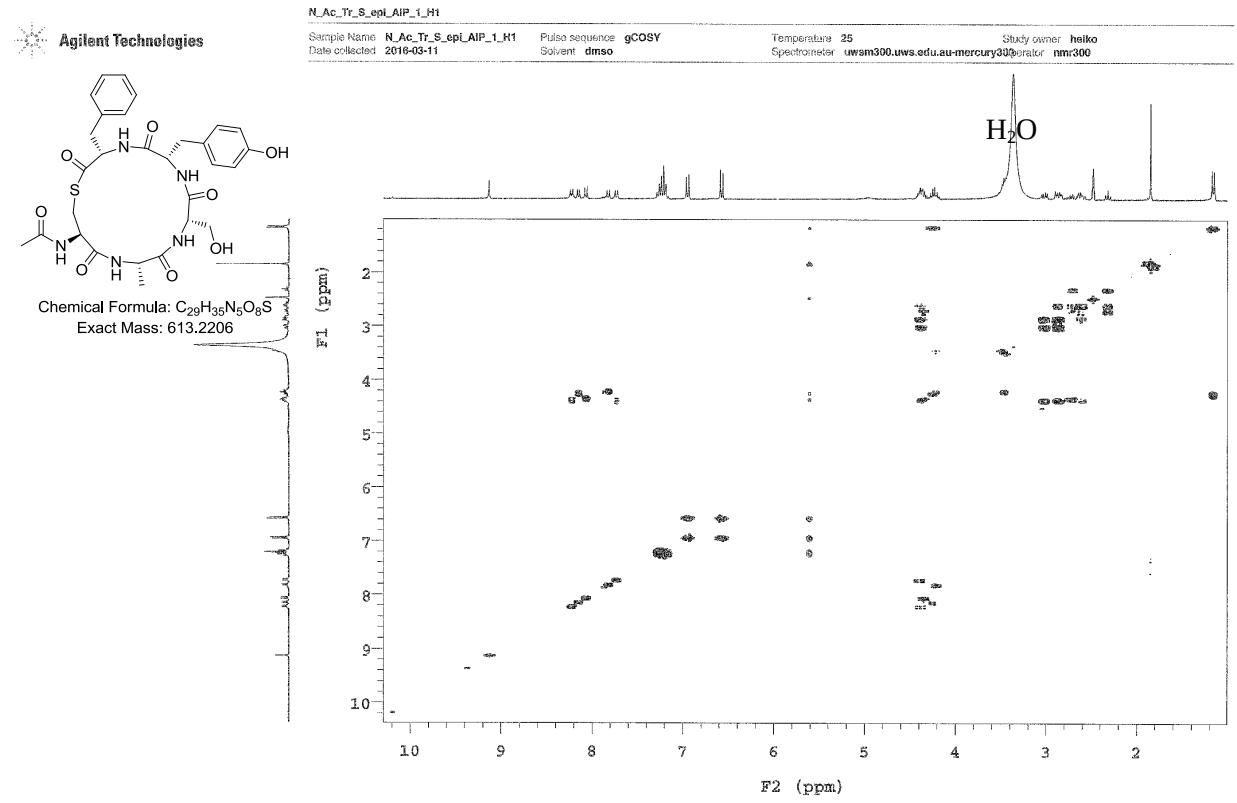


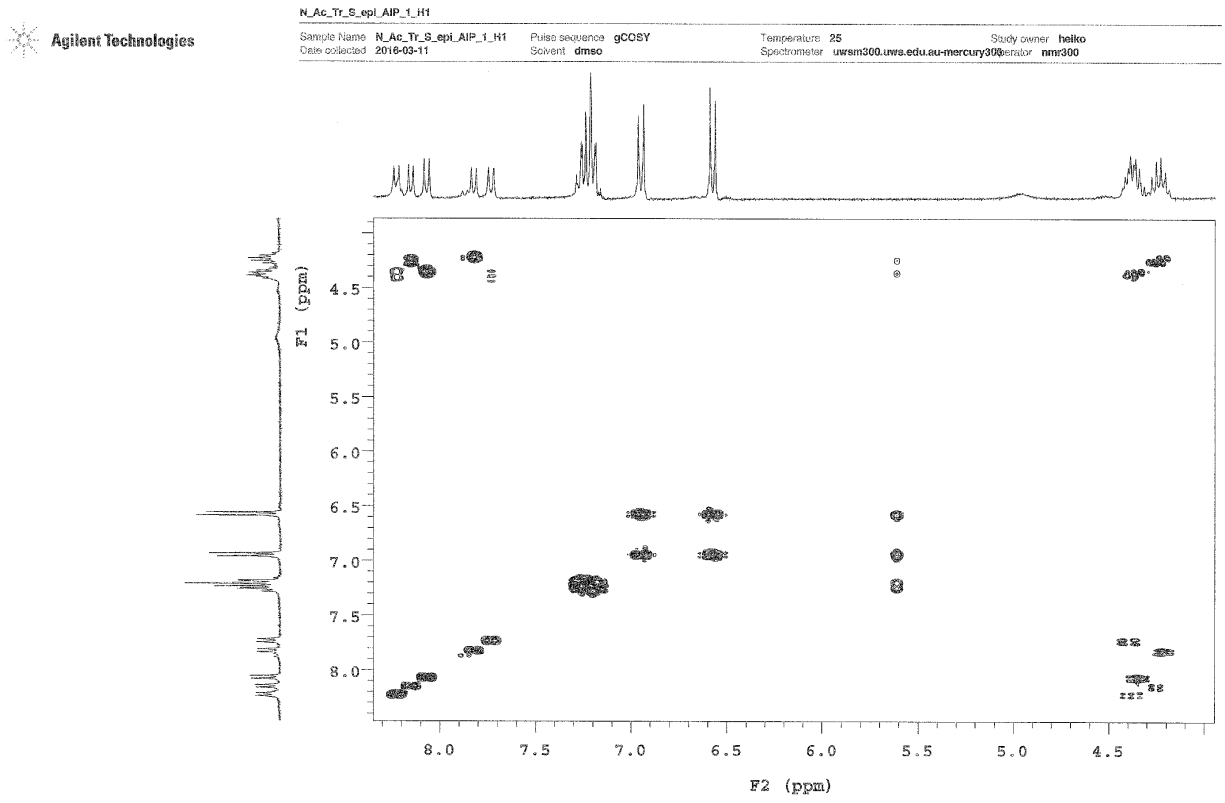

```
Data File C:\CHEM32\1\DATA\TR S EPI AIP I 6_8 MIN 2015-12-15 16-19-36\082-0101.D
Sample Name: Tr S epi AIP I 6.8 mins
    ======================================================================
    Acq. Operator   : Simi120102015                  Seq. Line :   1
    Acq. Instrument : LC1260                        Location : Vial 82
    Injection Date  : 12/15/2015 4:21:23 PM             Inj :   1
                                                   Inj Volume : 100.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\TR S EPI AIP I 6_8 MIN 2015-12-15 16-19-36\10 TO 100
                      OV 15MIN 100UL.M
    Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
    Analysis Method : C:\CHEM32\1\METHODS\10 TO 100 OV 15MIN 100UL.M
    Last changed    : 12/15/2015 5:24:52 PM by Simi120102015
                      (modified after loading) (Current integration events modified)
    ======================================================================
```

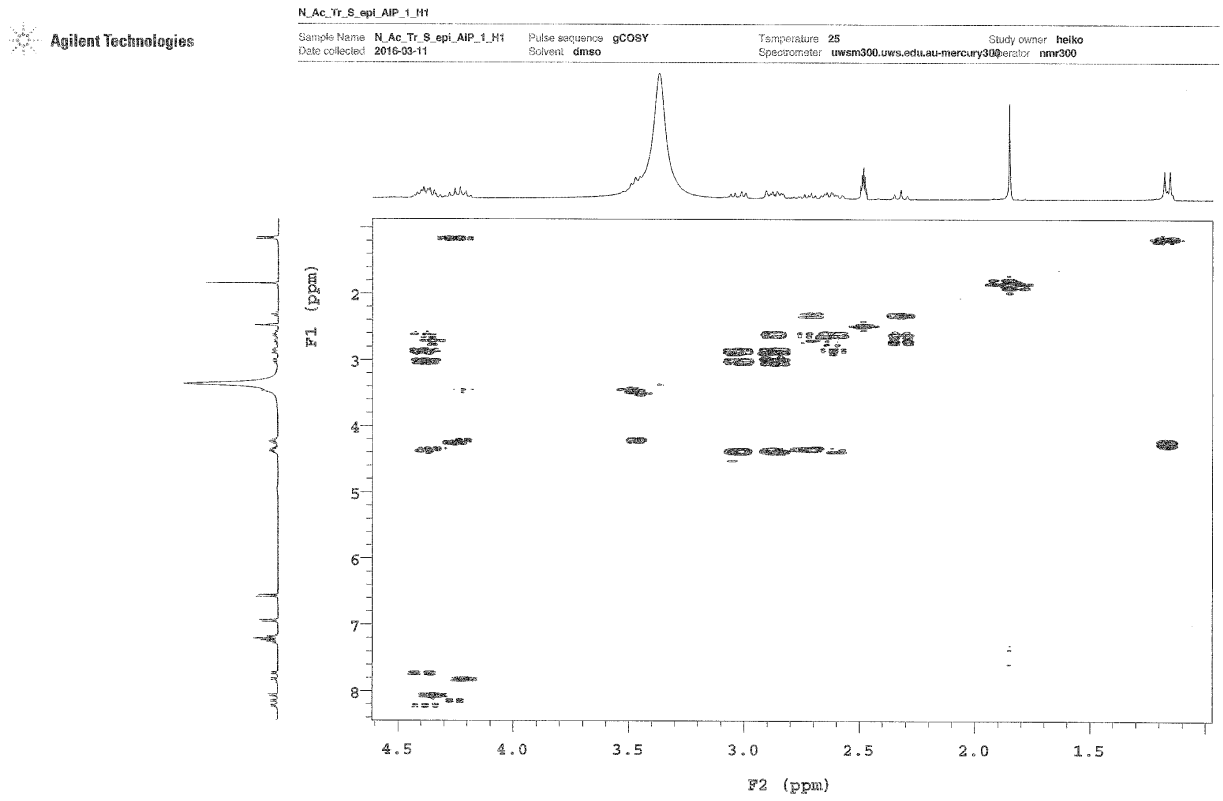


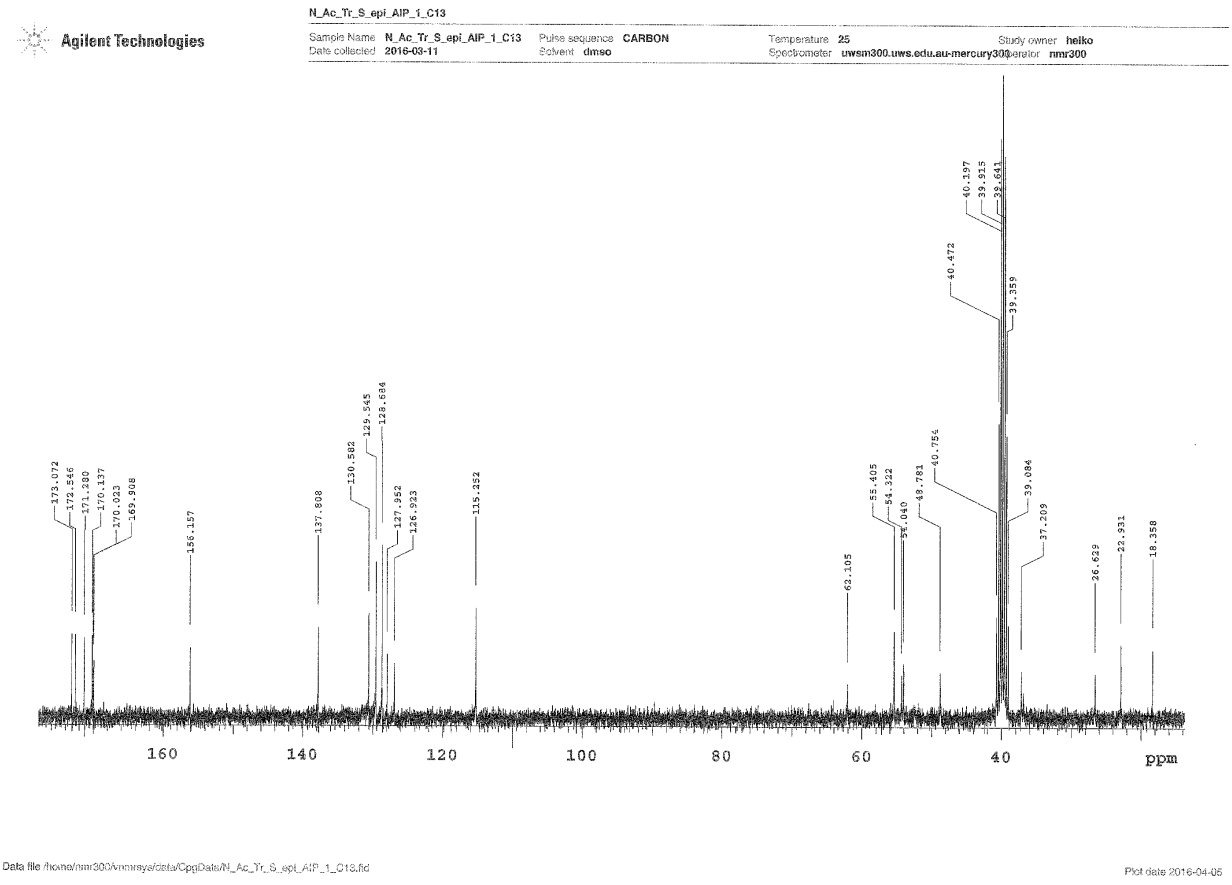

```
    ======================================================================
                               Area Percent Report
    ======================================================================

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]    [mAU*s]    [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.755 BB    0.1350 2235.54028  253.18295  75.1678
       2   6.820 VB    0.1317  738.52789   81.55083  24.8322

    Totals :                   2974.06818  334.73378


    Signal 2: DAD1 B, Sig=216,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]    [mAU*s]    [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   6.821 VV    0.1260 8081.33545  943.21979 100.0000

LC1260 12/15/2015 5:25:08 PM Simi120102015                    Page   1 of 2
```


20151209_Tr_S _epi_AIP_I_A_Final 102 (0.351) Sm (SG, 2x0.50); Cm (63:106)
1: MS2 ES+
4.99e7
614.40
Chemical Formula: C29H35N5O8S
Exact Mass: 613.2206
636.45
637.36
354.43
416.44
654.30
342.96
489.51
673.35
102.15
130.48
181.12
316.99
590.60
1029.74
243.45
939.97
%
m/z


Agilent Technologies
N_Ac_Tr_S_epi_AIP_1_H1
Sample Name N_Ac_Tr_S_epi_AIP_1_H1
Date collected 2016-03-11
Pulse sequence gCOSY
Solvent dmso
Temperature 25
Spectrometer uwsm300.uws.edu.au-mercury300
Study owner heiko
Operator nmr300
Chemical Formula: C29H35N5O8S
Exact Mass: 613.2206
H2O
F1 (ppm)
F2 (ppm)
Data file /home/nmr300/vnmrsys/data/N_Ac_Tr_S_epi_AIP_1_H1/N_Ac_Tr_S_epi_AIP_1_H1_gCOSY_01.fid
Plot date 2016-03-11


Agilent Technologies
N_Ac_Tr_S_epi_AIP_1_H1
Sample Name N_Ac_Tr_S_epi_AIP_1_H1
Date collected 2016-03-11
Pulse sequence gCOSY
Solvent dmso
Temperature 25
Spectrometer uwsm300.uwrs.edu.au-mercury300
Study owner heiko
Operator nmr300
F1 (ppm)
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
F2 (ppm)
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
Data file /home/nmr300/vnmrsys/data/N_Ac_Tr_S_epi_AIP_1_H1/N_Ac_Tr_S_epi_AIP_1_H1_gCOSY_01.fid
Plot date 2016-03-11

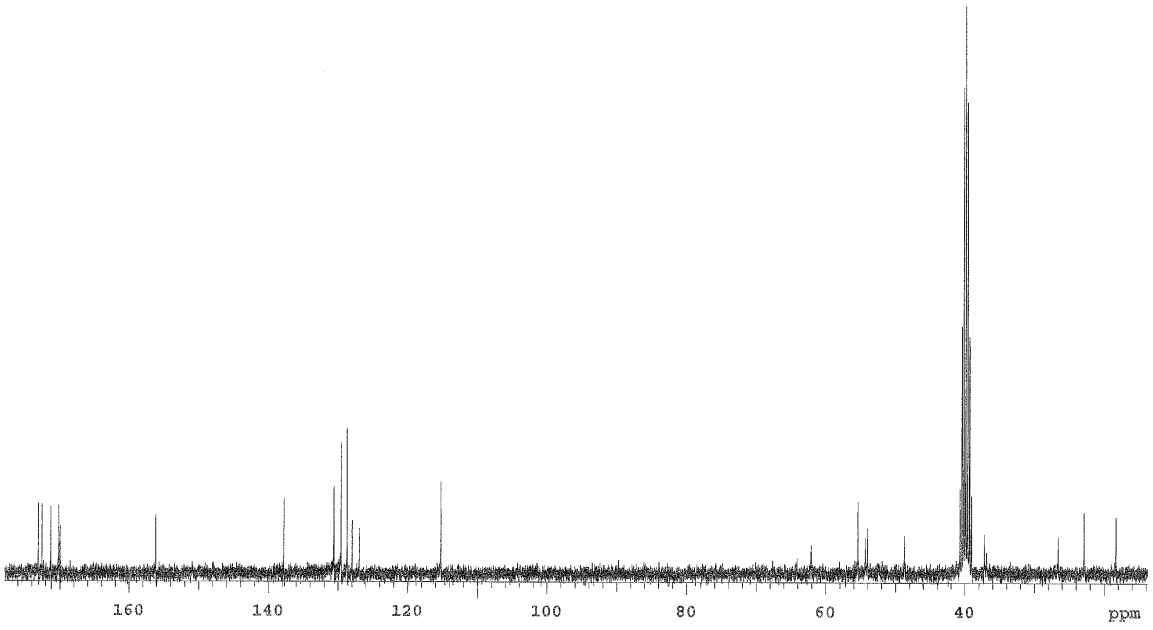

Agilent Technologies
N_Ac_Tr_S_epi_AIP_1_H1
Sample Name N_Ac_Tr_S_epi_AIP_1_H1
Date collected 2016-03-11
Pulse sequence gCOSY
Solvent dmso
Temperature 25
Spectrometer uwsm300.uws.edu.au-mercury300
Study owner heiko
Operator nmr300
F1 (ppm)
F2 (ppm)
Data file /home/nmr300/vnmrsys/data/N_Ac_Tr_S_epi_AIP_1_H1/N_Ac_Tr_S_epi_AIP_1_H1_gCOSY_01.fid
Plot data 2016-03-11


Agilent Technologies
N_Ac_Tr_S_epi_AIP_1_C13
Sample Name N_Ac_Tr_S_epi_AIP_1_C13
Date collected 2016-03-11
Pulse sequence CARBON
Solvent dmso
Temperature 25
Spectrometer unwsm300.uws.edu.au-mercury300
Study owner heiko
Operator nmr300
173.072
172.546
171.280
170.137
170.023
169.908
156.157
137.808
130.582
129.545
128.684
127.952
126.923
115.352
63.105
55.405
54.322
54.040
48.781
40.754
40.472
40.197
39.915
39.641
39.359
39.084
37.209
26.639
22.931
18.350
160
140
120
100
80
60
40
ppm
Data file /home/nmr300/vnmrsys/data/CpgiData/N_Ac_Tr_S_epi_AIP_1_C13.fid
Plot date 2016-04-05

Agilent Technologies

N_Ac_Tr_S_epi_AIP_1_C13

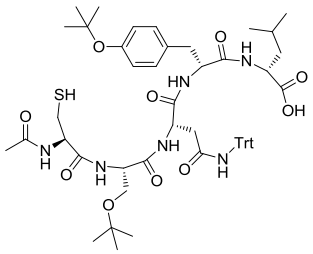
Sample Name **N_Ac_Tr_S_epi_AIP_1_C13** Pulse sequence **CARBON** Temperature **25** Study owner **helko**
Date collected **2016-03-11** Solvent **dmso** Spectrometer **uwsm300.uws.edu.au-mercury300** Operator **nmr300**



Data file /home/nmr300/vnmrsys/data/CpgData/N_Ac_Tr_S_epi_AIP_1_C13.fid

Plot date 2016-04-05

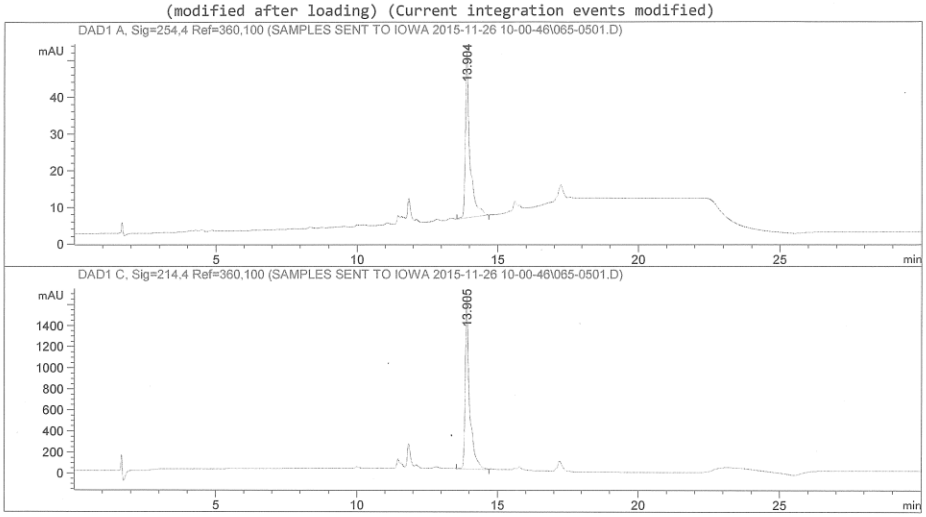
Compounds 13 & 4; The linear sequence required for **13** was prepared using general procedure 2. The residual crude material was triturated with ice-cold ether (× 3) to furnish an off-white solid. MS (ESI^+) for $C_{54}H_{71}N_6O_{10}S$; calculated 995.49, found, 995.71. RP-HPLC Onyx Monolithic C18 100 × 4.6 mm, 10-100% B in 15 min, t_R 13.91 min.



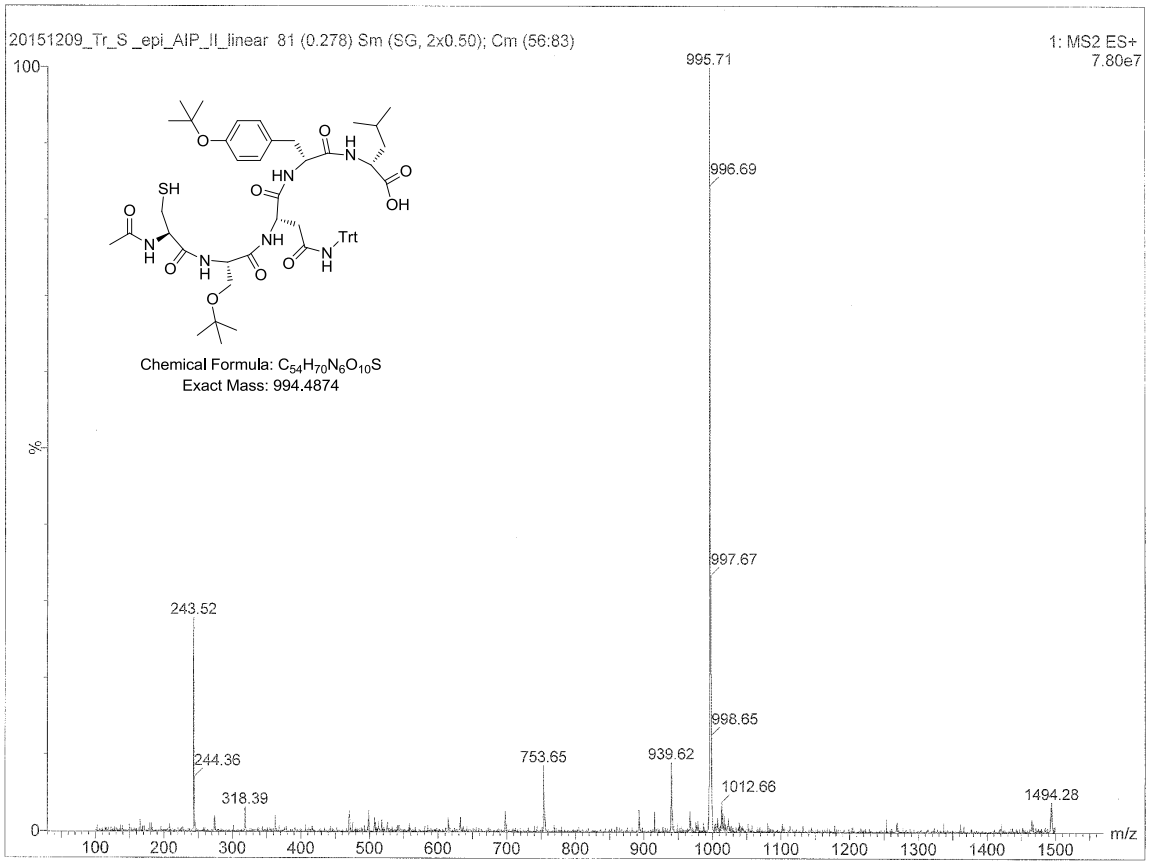
Chemical Formula: $C_{54}H_{70}N_6O_{10}S$
Exact Mass: 994.4874

Data File C:\CHEM32\1\DATA\SAMPLES SENT TO IOWA 2015-11-26 10-00-46\065-0501.D
Sample Name: S epi AIP II linear

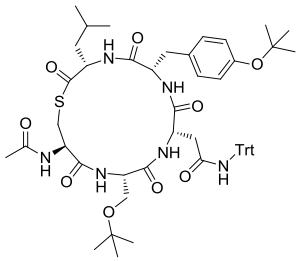
```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   5
Acq. Instrument : LC1260                        Location : Vial 65
Injection Date  : 11/26/2015 12:08:28 PM             Inj :   1
                                               Inj Volume : 20.000 µl
Acq. Method     : C:\CHEM32\1\DATA\SAMPLES SENT TO IOWA 2015-11-26 10-00-46\10 TO 100 OVER 15
                  MINS 20UL.M
Last changed    : 11/26/2015 10:00:46 AM by SYSTEM
Analysis Method : C:\CHEM32\1\DATA\SAMPLES SENT TO IOWA 2015-11-26 10-00-46\10 TO 100 OVER 15
                  MINS 20UL.M (Sequence Method)
Last changed    : 12/1/2015 10:35:17 AM by SYSTEM
                  (modified after loading) (Current integration events modified)
```




```
======================================================================================
                              Fraction Information
======================================================================================
======================================================================================
No Fractions found.
======================================================================================
======================================================================
                            Area Percent Report
======================================================================

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


20151209_Tr_S _epi_AIP_II_linear 81 (0.278) Sm (SG, 2x0.50); Cm (56:83)
1: MS2 ES+
7.80e7
100
%
0
995.71
996.69
997.67
998.65
1012.66
243.52
244.36
318.39
753.65
939.62
1494.28
m/z
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
1500
Chemical Formula: C54H70N6O10S
Exact Mass: 994.4874

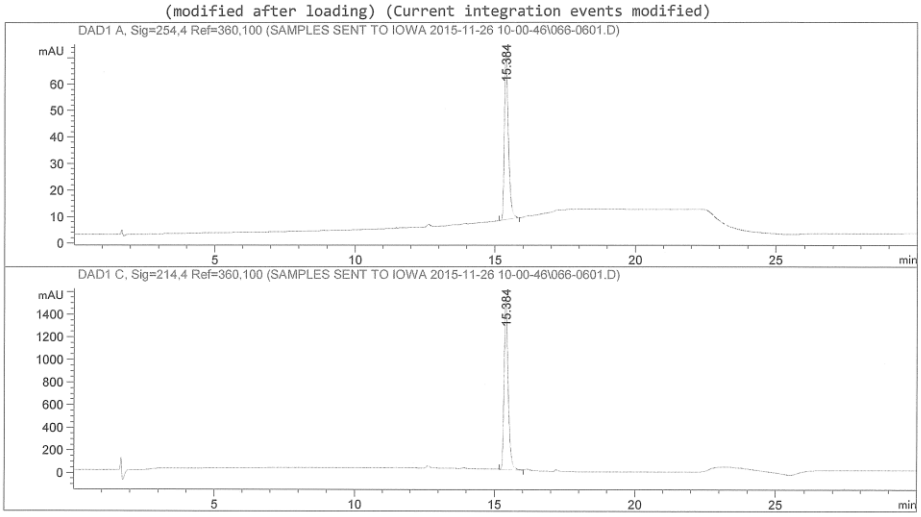
Compound 13; Compound **13** was synthesized utilizing general produce 3. The crude material was purified *via* semi-preparative RP-HPLC, and lyophilised to afford **13** (13 mg, 10.9 %). (*Note; 3 mg of this sample was collect for biological analysis*) MS (ESI^+) for $C_{54}H_{69}N_6O_9S$, *m/z* 977.71 (M + 1, 100%); HRMS (ESI^+) calculated 977.4768, found, 977.4719. RP-HPLC Onyx Monolithic C18 100 × 4.6 mm, 10-100% B in 15 min, t_R 15.38 min.



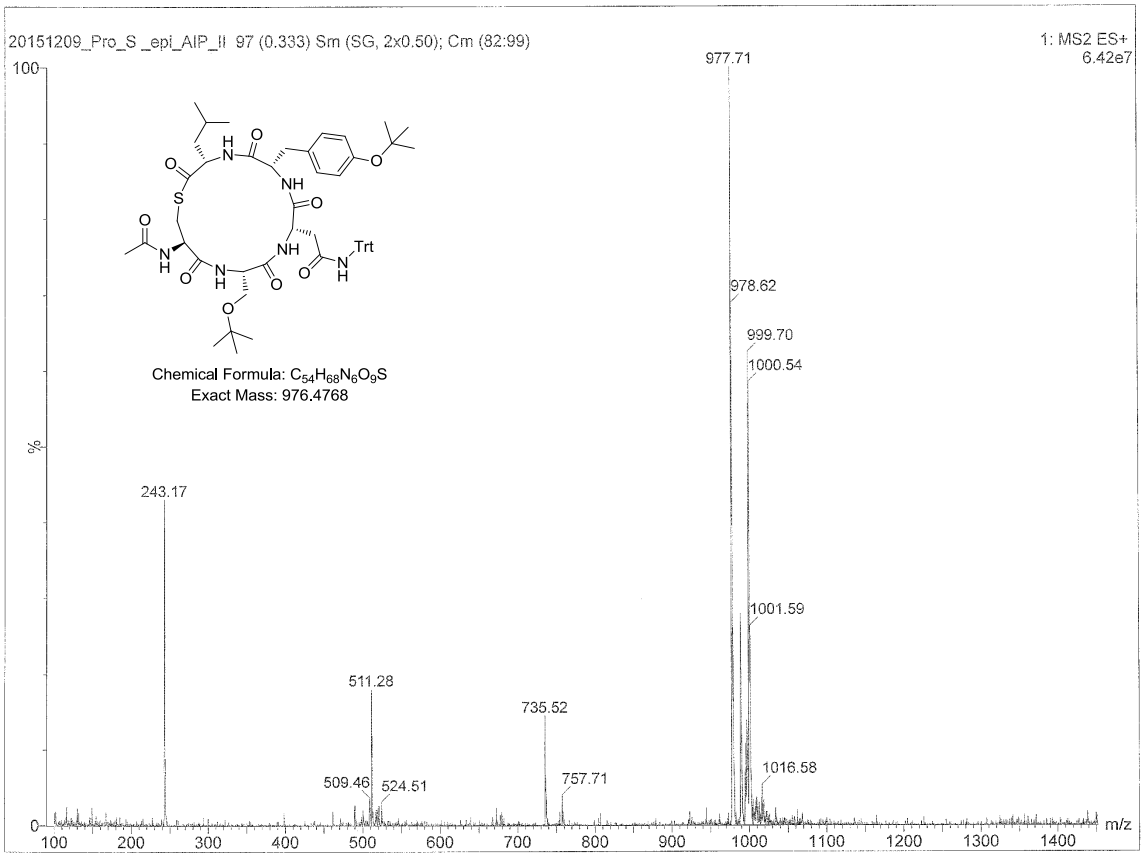
Chemical Formula: $C_{54}H_{68}N_6O_9S$
Exact Mass: 976.4768

Data File C:\CHEM32\1\DATA\SAMPLES SENT TO IOWA 2015-11-26 10-00-46\066-0601.D
Sample Name: Pro S epi AIP II cyclised

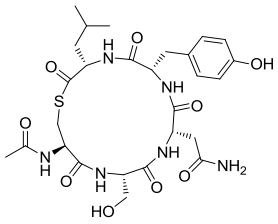
```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   6
Acq. Instrument : LC1260                         Location : Vial 66
Injection Date  : 11/26/2015 12:39:59 PM              Inj :   1
                                                Inj Volume : 20.000 µl
Acq. Method     : C:\CHEM32\1\DATA\SAMPLES SENT TO IOWA 2015-11-26 10-00-46\10 TO 100 OVER 15
                  MINS 20UL.M
Last changed    : 11/26/2015 10:00:46 AM by SYSTEM
Analysis Method : C:\CHEM32\1\DATA\SAMPLES SENT TO IOWA 2015-11-26 10-00-46\10 TO 100 OVER 15
                  MINS 20UL.M (Sequence Method)
Last changed    : 12/1/2015 10:36:05 AM by SYSTEM
                  (modified after loading) (Current integration events modified)
```




```
================================================================================
                          Fraction Information
================================================================================
================================================================================
No Fractions found.
================================================================================
==================================================================
                       Area Percent Report
==================================================================

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

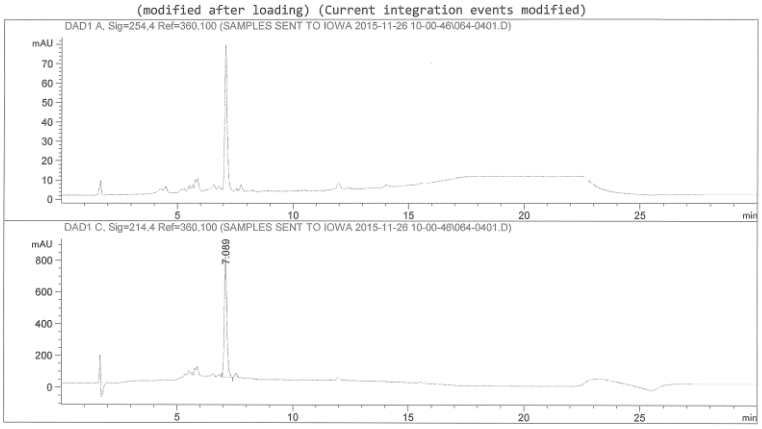

20151209_Pro_S _epi_AIP_II 97 (0.333) Sm (SG, 2x0.50); Cm (82:99)
1: MS2 ES+
6.42e7
100
%
0
977.71
978.62
999.70
1000.54
1001.59
1016.58
243.17
511.28
735.52
509.46
524.51
757.71
Trt
Chemical Formula: C54H68N6O9S
Exact Mass: 976.4768
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
m/z

Compound 4; Compound **4** was prepared utilising general procedure 4 with **13**. The crude material was purified *via* semi-preparative RP-HPLC, and lyophilised to afford **4** (4.7 mg, 75.1 %). MS (ESI^+) for $C_{27}H_{39}N_6O_9S$, *m/z* 623.3 (M + 1, 100%); HRMS (ESI^+) calculated 623.2421, found, 623.2425. RP-HPLC Onyx Monolithic C18 100 × 4.6 mm, 10-100% B in 15 min, t_R 7.08 min. ^{1}H NMR (300 MHz, DMSO) δ 9.16 (s, 1H), 8.10 (d, *J* = 8.0 Hz, 2H), 8.06 (d, *J* = 7.4 Hz, 1H), 8.01 (d, *J* = 7.8 Hz, 1H), 7.81 (d, *J* = 8.3 Hz, 1H), 7.33 (s, 1H), 6.98 (d, *J* = 8.5 Hz, 2H), 6.89 (s, 1H), 6.61 (d, *J* = 8.5 Hz, 2H), 4.51 – 4.30 (m, 3H), 4.26 (dd, *J* = 13.2, 6.0 Hz, 1H), 4.20 – 4.09 (m, 1H), 3.65 – 3.50 (m, 4H), 2.92 (dd, *J* = 14.2, 3.9 Hz, 1H), 2.79 (d, *J* = 5.3 Hz, 1H), 2.75 (d, *J* = 5.2 Hz, 1H), 2.69 – 2.56 (m, 2H), 2.44 (d, *J* = 5.4 Hz, 1H), 2.35 (d, *J* = 7.5 Hz, 1H), 2.30 (d, *J* = 7.3 Hz, 1H), 1.85 (d, *J* = 7.0 Hz, 3H), 1.67 – 1.40 (m, 3H), 0.87 (d, *J* = 6.3 Hz, 2H), 0.82 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (75 MHz, DMSO) δ 174.29, 172.96, 172.88, 172.03, 171.29, 170.85, 170.56, 170.26, 170.09, 156.13, 130.51, 128.24, 128.20, 115.34, 115.34, 62.04, 55.49, 53.24, 53.02, 50.84, 50.34, 32.85, 26.62, 26.61, 24.64, 23.30, 22.92, 21.82.



Data File C:\CHEM32\1\DATA\SAMPLES SENT TO IOWA 2015-11-26 10-00-46\064-0401.D
Sample Name: Tr S epi AIP I 8.5 min

Acq. Operator : SYSTEM Seq. Line : 4
Acq. Instrument : LC1260 Location : Vial 64
Injection Date : 11/26/2015 11:36:54 AM Inj : 1
Inj Volume : 20.000 µl
Acq. Method : C:\CHEM32\1\DATA\SAMPLES SENT TO IOWA 2015-11-26 10-00-46\10 TO 100 OVER 15 MINS 20UL.M
Last changed : 11/26/2015 10:00:46 AM by SYSTEM
Analysis Method : C:\CHEM32\1\DATA\SAMPLES SENT TO IOWA 2015-11-26 10-00-46\10 TO 100 OVER 15 MINS 20UL.M (Sequence Method)
Last changed : 12/1/2015 10:34:30 AM by SYSTEM (modified after loading) (Current integration events modified)



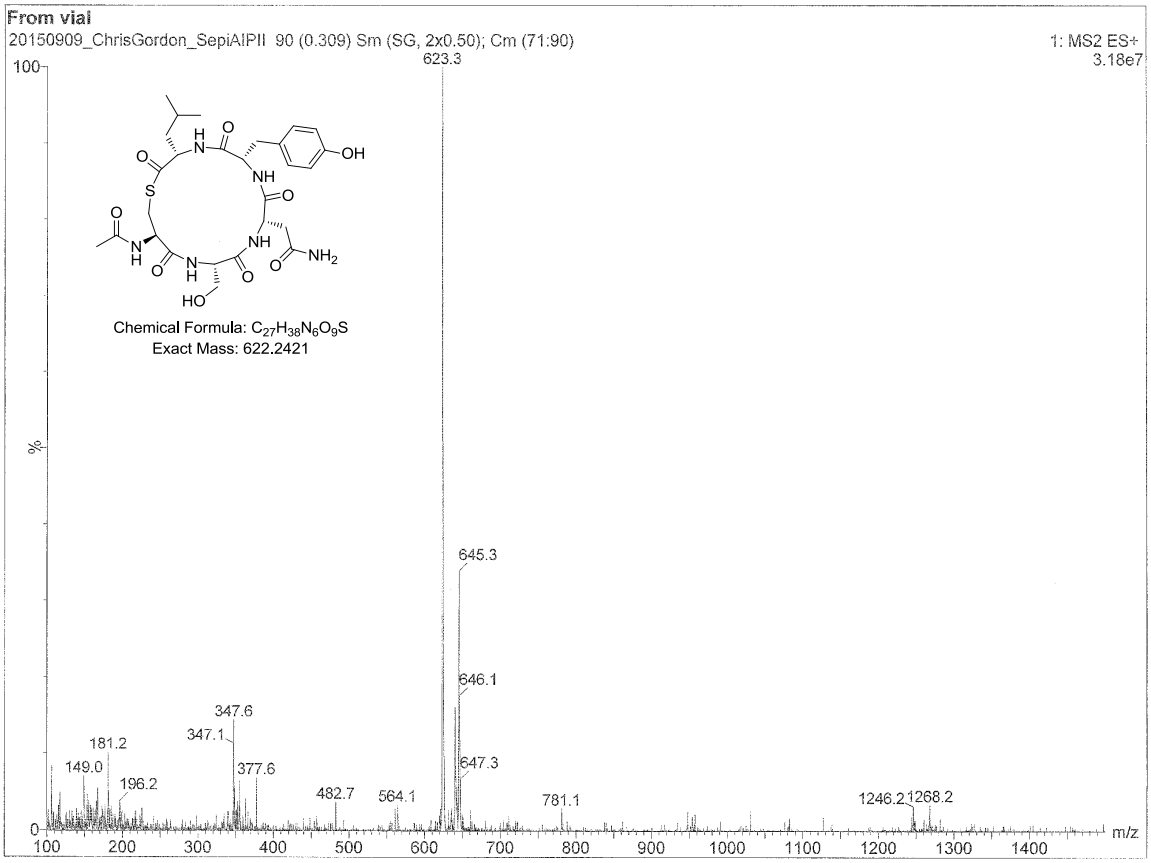
Fraction Information

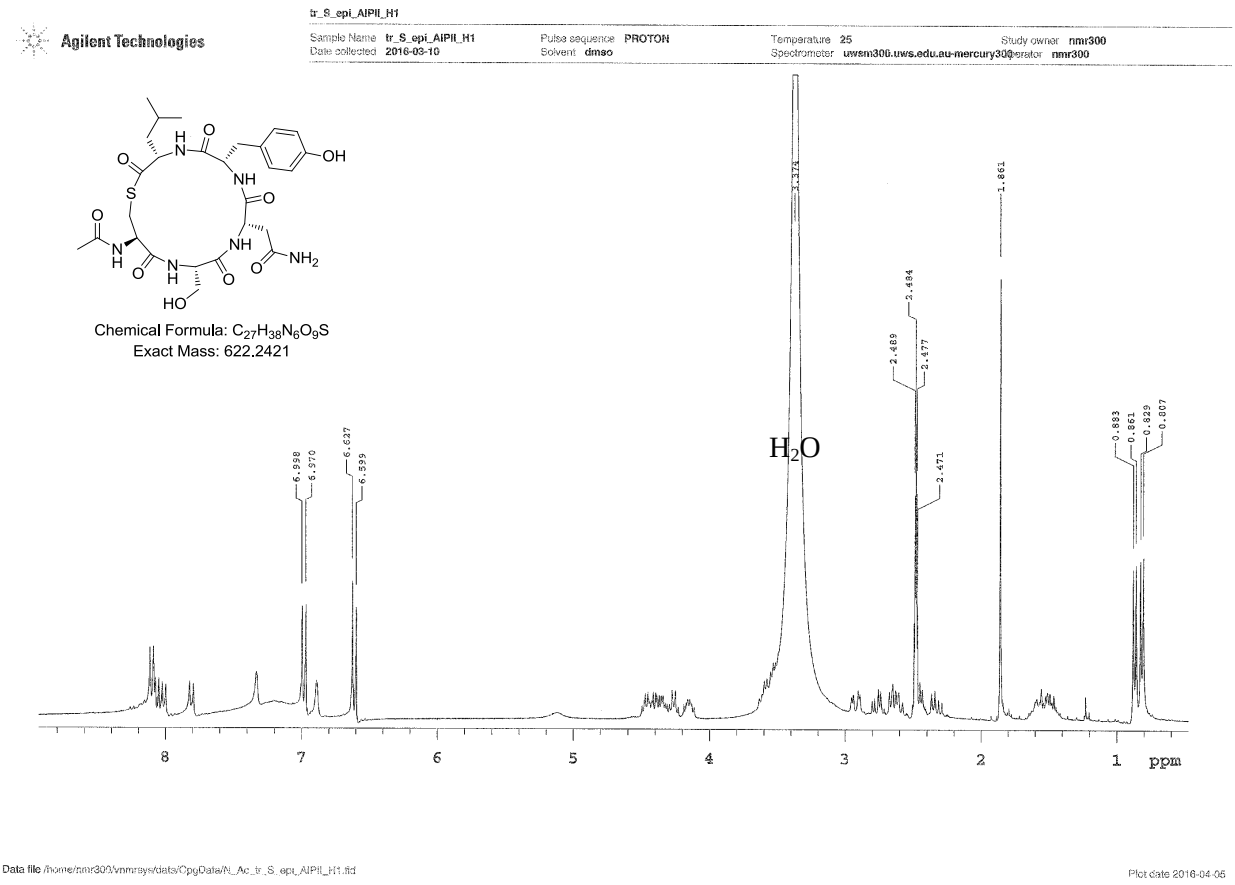
No Fractions found.

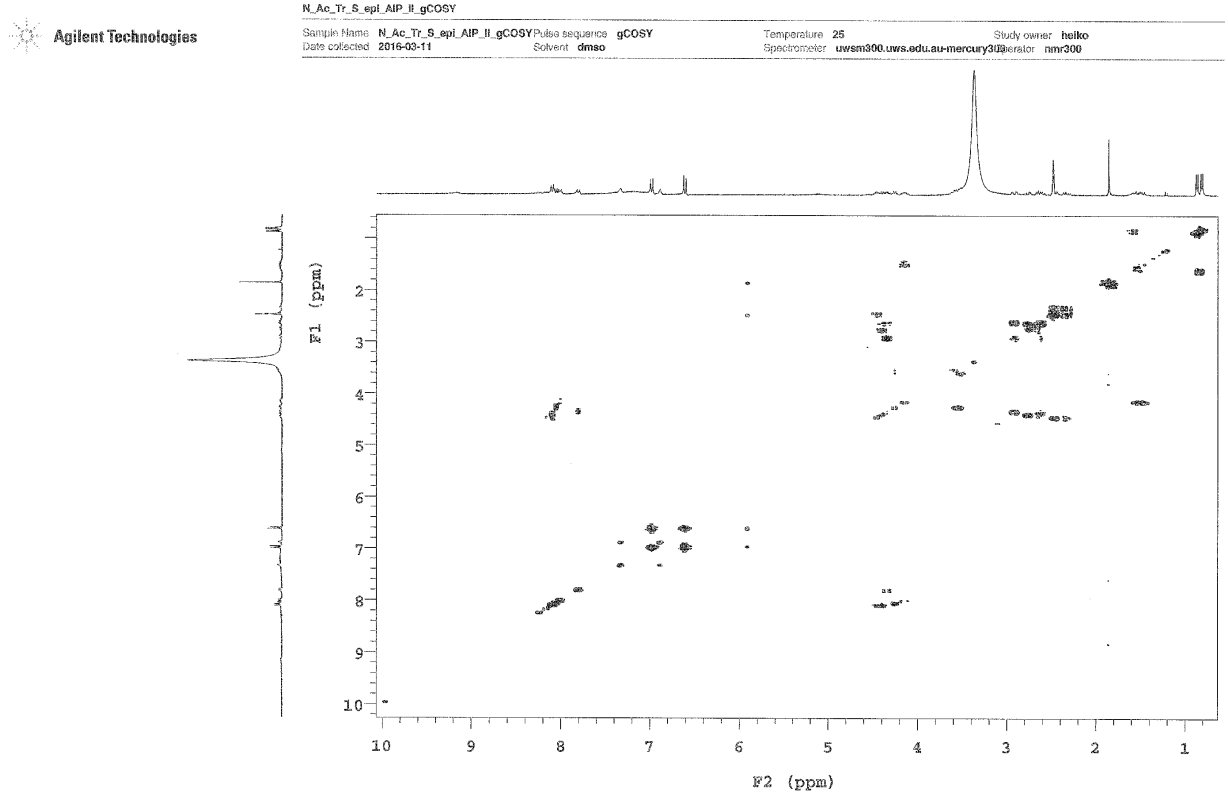
Area Percent Report

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

LC1260 12/1/2015 10:35:23 AM SYSTEM Page 1 of 2

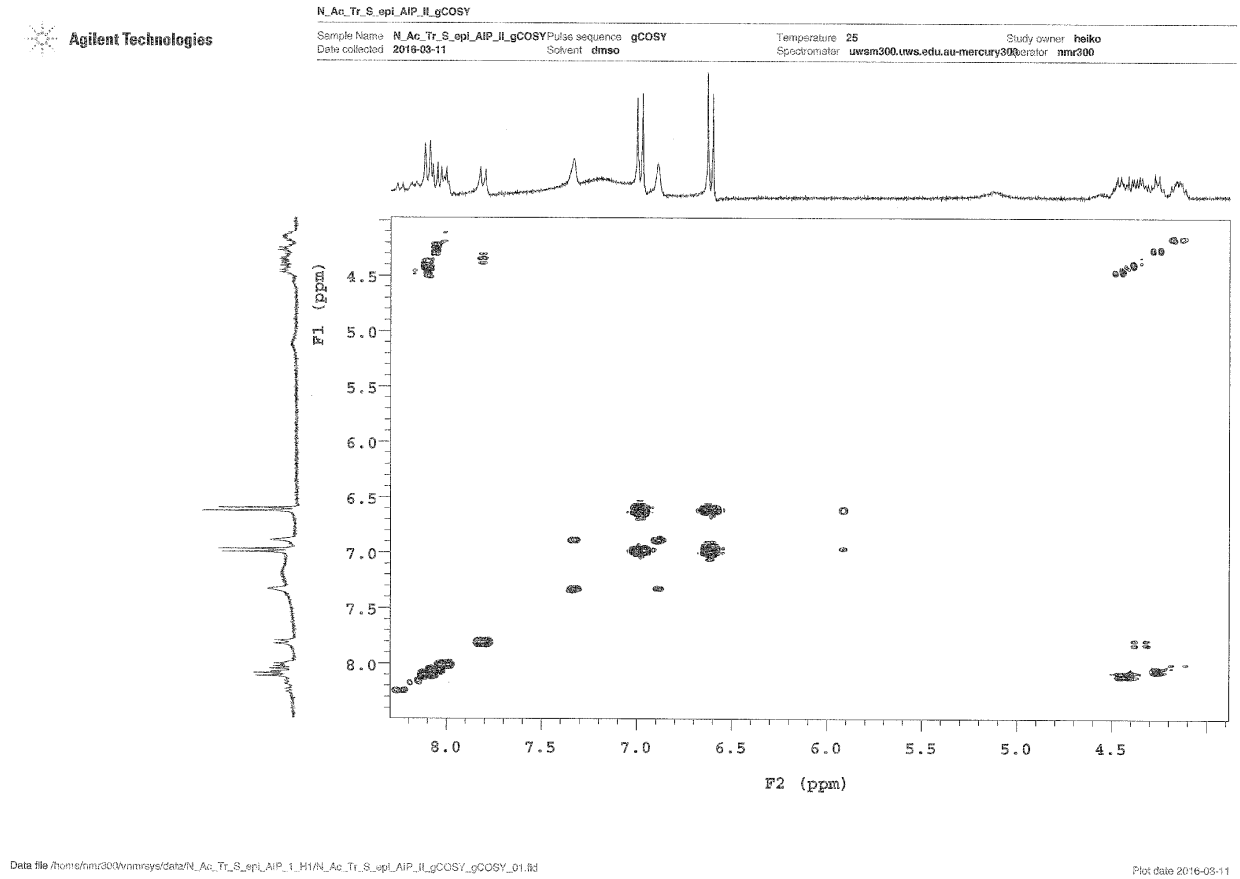

From vial
20150909_ChrisGordon_SepiAIPII 90 (0.309) Sm (SG, 2x0.50); Cm (71:90)
1: MS2 ES+
3.18e7
623.3
Chemical Formula: C27H38N6O9S
Exact Mass: 622.2421
645.3
646.1
647.3
347.6
347.1
181.2
149.0
196.2
377.6
482.7
564.1
781.1
1246.2
1268.2
%
m/z

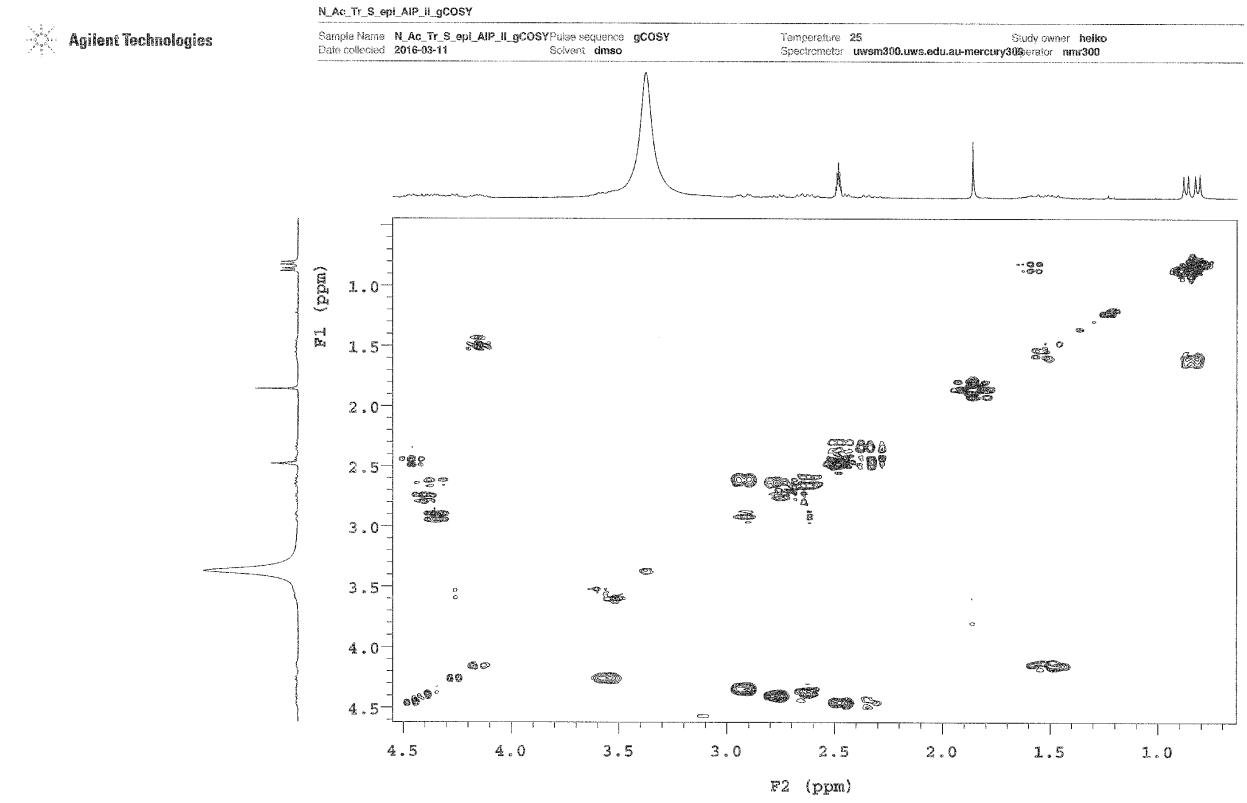

tr_S_epi_AIPII_H1
Agilent Technologies
Sample Name tr_S_epi_AIPII_H1
Date collected 2016-03-10
Pulse sequence PROTON
Solvent dmso
Temperature 25
Spectrometer uwsm300.uws.edu.au-mercury300
Study owner nmr300
Operator nmr300
Chemical Formula: C27H38N6O9S
Exact Mass: 622.2421
6.958
6.970
6.627
6.599
3.374
H2O
2.489
2.484
2.477
2.471
1.861
0.883
0.861
0.829
0.807
8
7
6
5
4
3
2
1
ppm
Data file /home/nmr300/vnmrsys/data/CpgData/N_Ac_tr_S_epi_AIPII_H1.fid
Plot date 2016-04-05

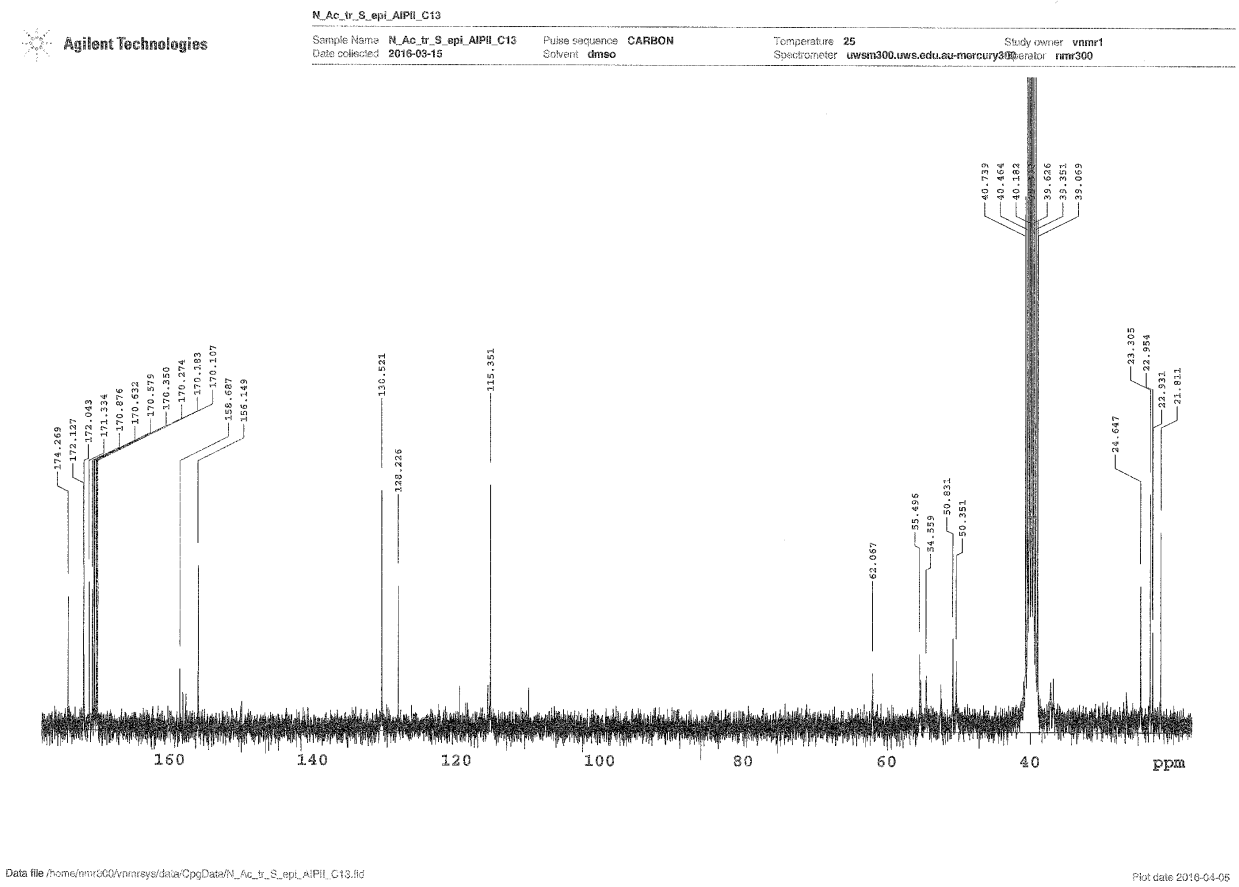


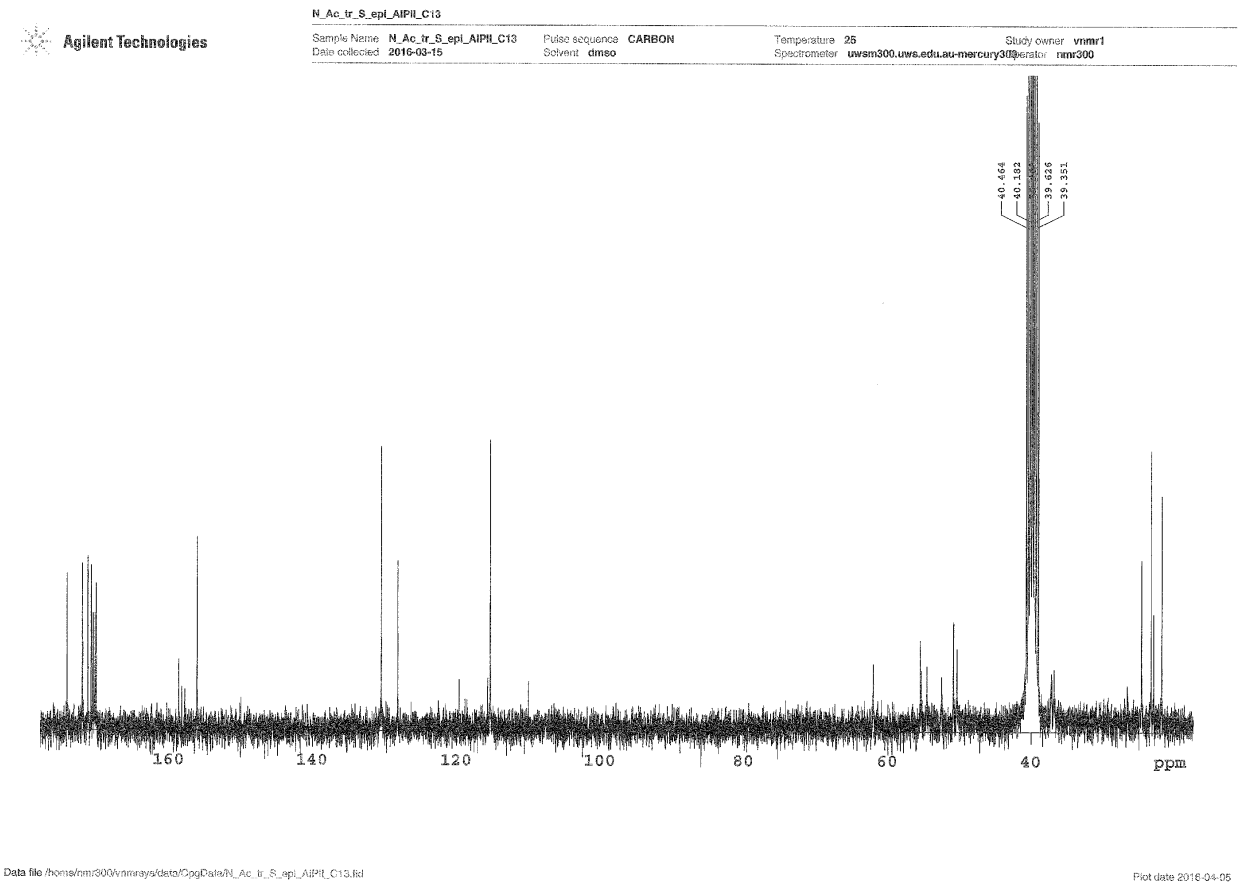
Data file /home/nmr300/vnmrsys/data/N_Ac_Tr_S_epi_AIP_1_H1/N_Ac_Tr_S_epi_AIP_II_gCOSY_gCOSY_01.fid

Plot date 2016-03-11

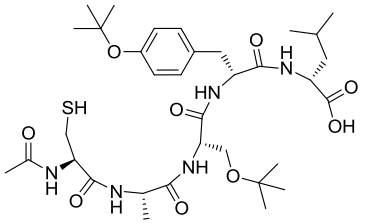

Agilent Technologies
N_Ac_Tr_S_epi_AIP_II_gCOSY
Sample Name N_Ac_Tr_S_epi_AIP_II_gCOSY Pulse sequence gCOSY Temperature 25 Study owner heiko
Date collected 2016-03-11 Solvent dmso Spectrometer uwsm300.uws.edu.au-mercury300 Operator nmr300
F1 (ppm)
F2 (ppm)
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
Data file /home/nmr300/vnmrsys/data/N_Ac_Tr_S_epi_AIP_1_H1/N_Ac_Tr_S_epi_AIP_II_gCOSY_gCOSY_01.fid
Plot date 2016-03-11


Agilent Technologies
N_Ac_Tr_S_epi_AIP_II_gCOSY
Sample Name N_Ac_Tr_S_epi_AIP_II_gCOSY Pulse sequence gCOSY Temperature 25 Study owner heiko
Date collected 2016-03-11 Solvent dmso Spectrometer uwsm300.uws.edu.au-mercury300 Operator nmr300
F1 (ppm)
F2 (ppm)
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
Data file /home/nmr300/vnmrsys/data/N_Ac_Tr_S_epi_AIP_1_H1/N_Ac_Tr_S_epi_AIP_II_gCOSY_gCOSY_01.fid
Plot date 2016-03-11


Agilent Technologies
N_Ac_tr_S_epi_AlPII_C13
Sample Name N_Ac_tr_S_epi_AlPII_C13
Date collected 2016-03-15
Pulse sequence CARBON
Solvent dmso
Temperature 25
Spectrometer uwsm300.uws.edu.au-mercury300
Study owner vnmr1
Operator nmr300
174.269
172.127
172.043
171.334
170.876
170.632
170.579
170.350
170.274
170.183
170.107
156.687
156.449
130.521
130.236
115.351
62.067
55.496
54.359
50.831
50.351
40.739
40.464
40.182
39.626
39.351
39.069
24.647
23.305
22.954
22.931
21.811
160
140
120
100
80
60
40
ppm
Data file /home/nmr300/vnmrsys/data/CpgData/N_Ac_tr_S_epi_AlPII_C13.fid
Plot date 2016-04-05


N_Ac_tr_S_epi_AlPil_C13
Agilent Technologies
Sample Name N_Ac_tr_S_epi_AlPil_C13
Date collected 2016-03-15
Pulse sequence CARBON
Solvent dmso
Temperature 25
Spectrometer uwsm300.uws.edu.au-mercury300
Study owner vnmr1
Operator nmr300
40.464
40.182
39.626
39.351
160
140
120
100
80
60
40
ppm
Data file /home/nmr300/vnmrsys/data/OpgData/N_Ac_tr_S_epi_AlPil_C13.fid
Plot date 2016-04-05

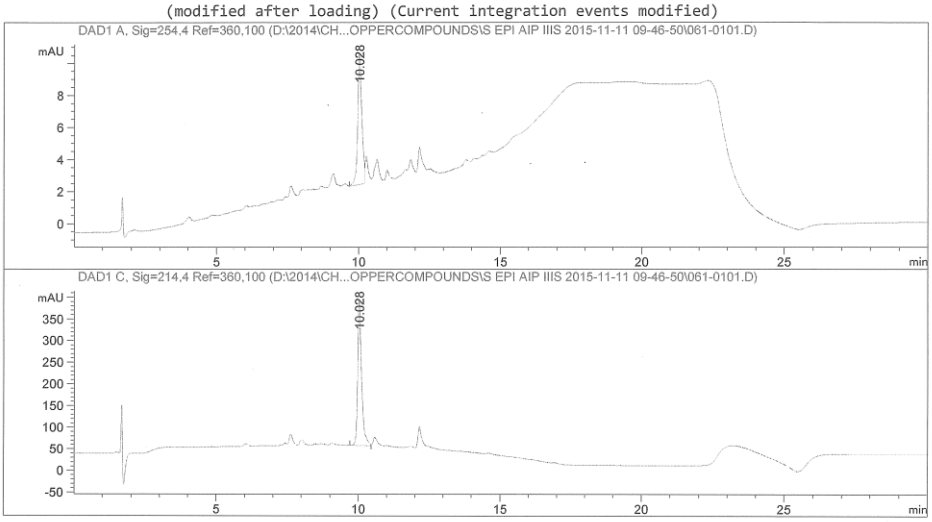
Compounds 14 & 5; The linear sequence required for **14** was prepared using general procedure 2. The residual crude material was triturated with ice-cold ether (× 3) to furnish an off-white solid. MS (ESI^+) for $C_{34}H_{56}N_5O_9S$; calculated 710.37, found, 710.52. RP-HPLC Onyx Monolithic C18 100 × 4.6 mm, 10-100% B in 15 min, t_R 10.03 min.



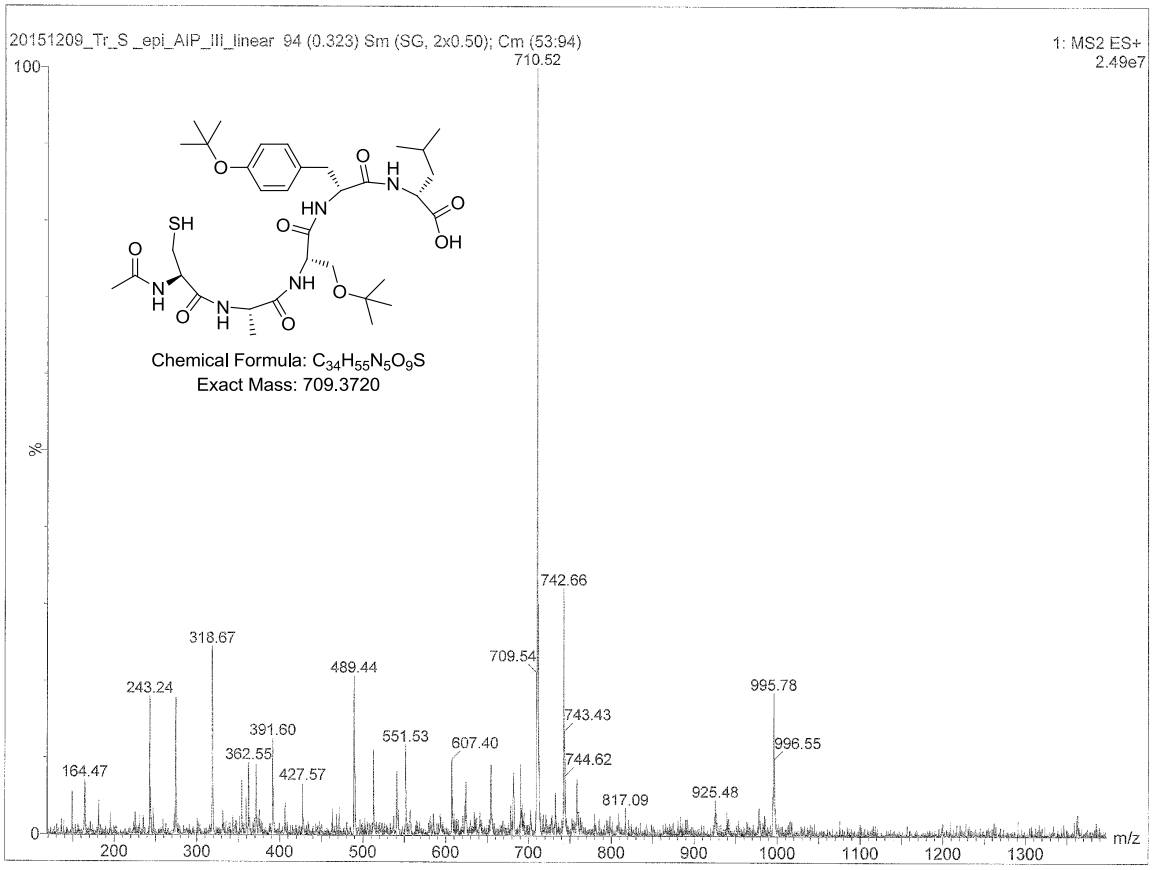
Chemical Formula: $C_{34}H_{55}N_5O_9S$
Exact Mass: 709.3720

Data File D:\2014\CH...ATA\DDCOPPERCOMPOUNDS\S EPI AIP IIIS 2015-11-11 09-46-50\061-0101.D
Sample Name: S epi AIP III linear

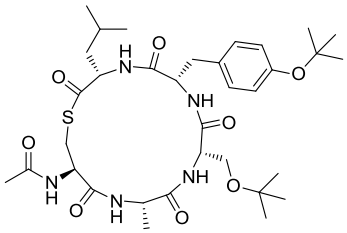
```
======================================================================
Acq. Operator   : SYSTEM                       Seq. Line :   1
Acq. Instrument : LC1260                       Location : Vial 61
Injection Date  : 11/11/2015 9:48:10 AM             Inj :   1
                                               Inj Volume : 10.000 µl
Method          : D:\2014\CHRIS\DATA\DDCOPPERCOMPOUNDS\S EPI AIP IIIS 2015-11-11 09-46-50\10
                  TO 100 OVER 15 MINS 10UL.M (Sequence Method)
Last changed    : 11/11/2015 9:46:50 AM by SYSTEM
                  (modified after loading) (Current integration events modified)
```




```
================================================================================
                          Fraction Information
================================================================================
================================================================================
No Fractions found.
================================================================================
======================================================================
                        Area Percent Report
======================================================================

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


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.028 BV    0.1450   83.26463    8.16124 100.0000

Totals :                   83.26463    8.16124
```


20151209_Tr_S _epi_AIP_III_linear 94 (0.323) Sm (SG, 2x0.50); Cm (53:94)
1: MS2 ES+
2.49e7
Chemical Formula: C34H55N5O9S
Exact Mass: 709.3720
710.52
742.66
318.67
709.54
489.44
995.78
243.24
743.43
391.60
551.53
607.40
996.55
362.55
744.62
164.47
427.57
817.09
925.48
m/z

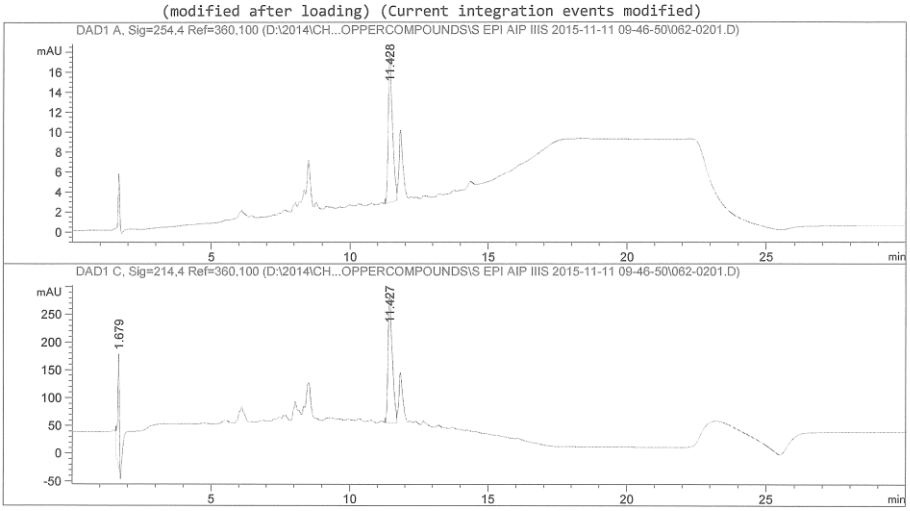
Compound 14; Compound **14** was synthesised utilising general produce 3. The crude material was purified *via* semi-preparative RP-HPLC, and lyophilised to afford **14** (16 mg, 18.8 %). (*Note; 3 mg of this sample was collect for biological analysis*) MS (ESI^+) for $C_{34}H_{54}N_5O_8S$ *m/z* 692.46 (M + 1, 100%); HRMS (ESI^+) calculated 692.3615, found, 692.3620. RP-HPLC Onyx Monolithic C18 100 × 4.6 mm, 10-100% B in 15 min, t_R 11.43 min.



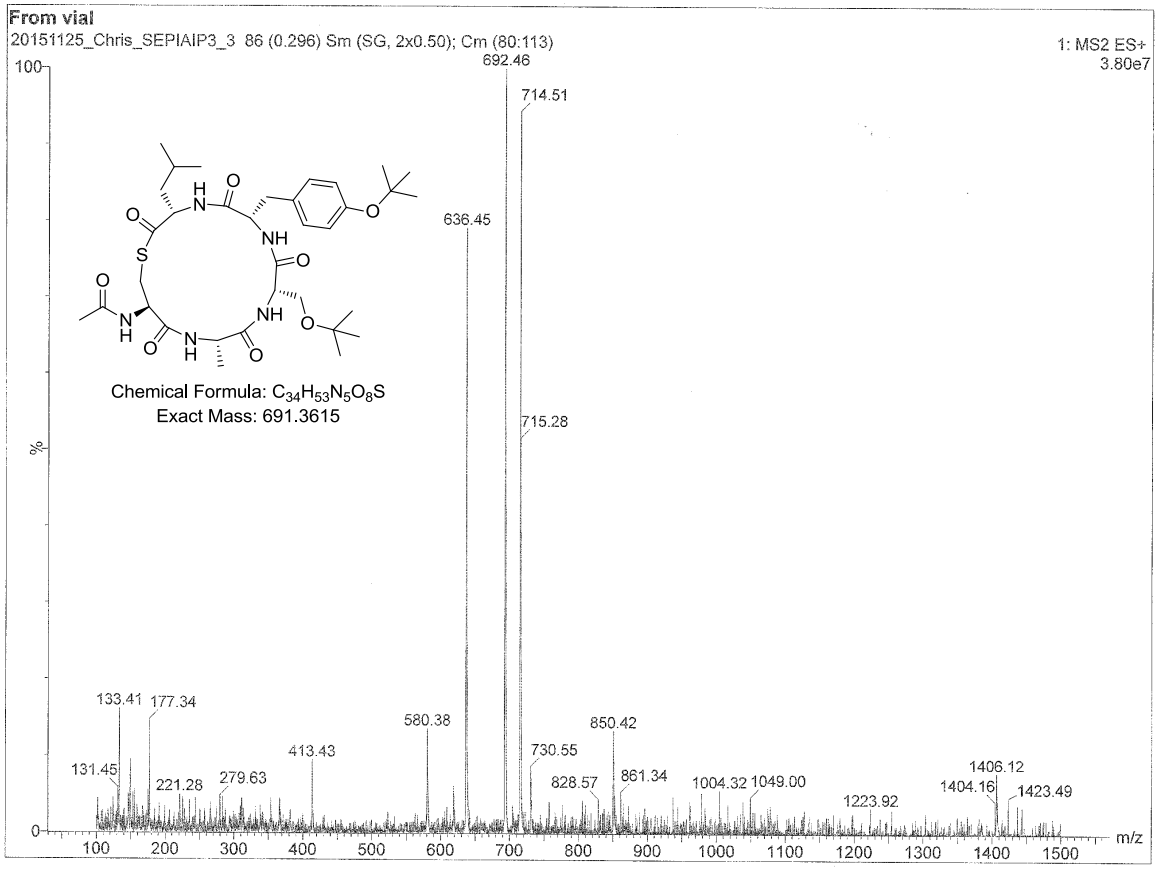
Chemical Formula: $C_{34}H_{53}N_5O_8S$
Exact Mass: 691.3615

Data File D:\2014\CH...ATA\DDCOPPERCOMPOUNDS\S EPI AIP IIIS 2015-11-11 09-46-50\062-0201.D
Sample Name: S epi AIP III cyclised crude

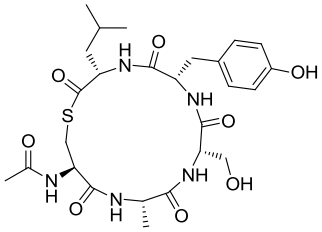
```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : LC1260                        Location : Vial 62
Injection Date  : 11/11/2015 10:19:37 AM             Inj :   1
                                               Inj Volume : 10.000 µl
Acq. Method     : D:\2014\CHRIS\DATA\DDCOPPERCOMPOUNDS\S EPI AIP IIIS 2015-11-11 09-46-50\10
                  TO 100 OVER 15 MINS 10UL.M
Last changed    : 11/11/2015 9:46:50 AM by SYSTEM
Analysis Method : D:\2014\CHRIS\DATA\DDCOPPERCOMPOUNDS\S EPI AIP IIIS 2015-11-11 09-46-50\10
                  TO 100 OVER 15 MINS 10UL.M (Sequence Method)
Last changed    : 11/11/2015 4:29:12 PM by SYSTEM
                  (modified after loading) (Current integration events modified)
```




```
================================================================================
                          Fraction Information
================================================================================
================================================================================
No Fractions found.
================================================================================
==================================================================
                        Area Percent Report
==================================================================

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


From vial
20151125_Chris_SEPIAIP3_3 86 (0.296) Sm (SG, 2x0.50); Cm (80:113)
1: MS2 ES+
3.80e7
Chemical Formula: C34H53N5O8S
Exact Mass: 691.3615
692.46
714.51
636.45
715.28
133.41
177.34
580.38
850.42
413.43
730.55
131.45
221.28
279.63
828.57
861.34
1004.32
1049.00
1406.12
1404.16
1423.49
1223.92
%
m/z

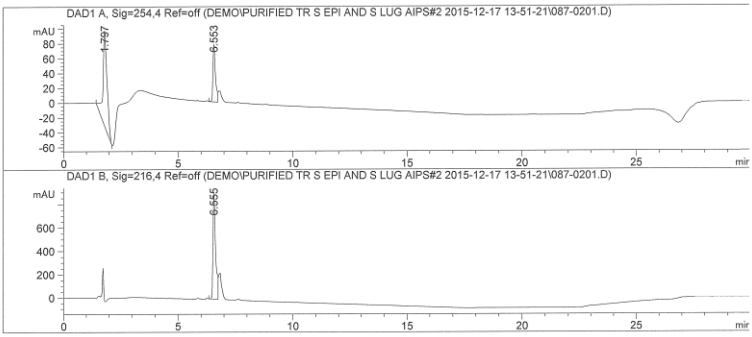
Compound 5; Compound **5** was prepared utilising general procedure 4 with **14**. The crude material was purified *via* semi-preparative RP-HPLC, and lyophilised to afford **5** (9.1 mg, 71.8 %). MS (ESI^+) for $C_{27}H_{38}N_5O_8S$, *m/z* 580.16; HRMS (ESI^+) calculated 580.2363, found, 580.2367. RP-HPLC Onyx Monolithic C18 100 × 4.6 mm, 10-100% B in 15 min, t_R 6.55 min. ^{1}H NMR (300 MHz, DMSO) δ 9.16 (s, 1H), 8.10 (d, *J* = 8.0 Hz, 2H), 8.06 (d, *J* = 7.4 Hz, 1H), 8.01 (d, *J* = 7.8 Hz, 1H), 7.81 (d, *J* = 8.3 Hz, 1H), 7.33 (s, 1H), 6.98 (d, *J* = 8.5 Hz, 2H), 6.89 (s, 1H), 6.61 (d, *J* = 8.5 Hz, 2H), 4.51 – 4.30 (m, 3H), 4.26 (dd, *J* = 13.2, 6.0 Hz, 1H), 4.20 – 4.10 (m, 1H), 3.65 – 3.49 (m, 6H), 2.92 (dd, *J* = 14.2, 3.9 Hz, 2H), 2.79 (d, *J* = 5.3 Hz, 1H), 2.75 (d, *J* = 5.2 Hz, 1H), 2.70 – 2.56 (m, 2H), 2.44 (d, *J* = 5.4 Hz, 1H), 2.35 (d, *J* = 7.5 Hz, 1H), 2.30 (d, *J* = 7.3 Hz, 1H), 1.86 (s, 3H), 1.67 – 1.39 (m, 3H), 0.87 (d, *J* = 6.3 Hz, 3H), 0.82 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (75 MHz, DMSO) δ 176.39, 174.74, 174.59, 173.46, 172.42, 172.30, 172.18, 172.11, 158.33, 132.76, 130.16, 117.41, 64.30, 57.70, 57.57, 56.42, 52.91, 50.95, 42.51, 38.98, 28.79, 26.80, 25.46, 25.10, 23.93, 20.65, 20.49.



Chemical Formula: $C_{26}H_{37}N_5O_8S$
Exact Mass: 579.2363

Data File C:\CHEM32\...O\PURIFIED TR S EPI AND S LUG AIPS#2 2015-12-17 13-51-21\087-0201.D
Sample Name: Tr S epi AIP II 6.5 min

Acq. Operator	: Simi120102015	Seq. Line	: 2
Acq. Instrument	: LC1260	Location	: Vial 87
Injection Date	: 12/17/2015 2:24:49 PM	Inj	: 1
		Inj Volume	: 100.000 µl
Acq. Method	: C:\CHEM32\1\DATA\DEMO\PURIFIED TR S EPI AND S LUG AIPS#2 2015-12-17 13-51-21\10 TO 100 OV 15MIN 100UL.M		
Last changed	: 12/10/2015 4:12:03 PM by Simi120102015		
Analysis Method	: C:\CHEM32\1\DATA\DEMO\PURIFIED TR S EPI AND S LUG AIPS 2016-01-05 09-33-14\10 TO 100 OV 15MIN 20UL.M		
Last changed	: 1/6/2016 3:19:20 PM by Simi120102015 (modified after loading)		



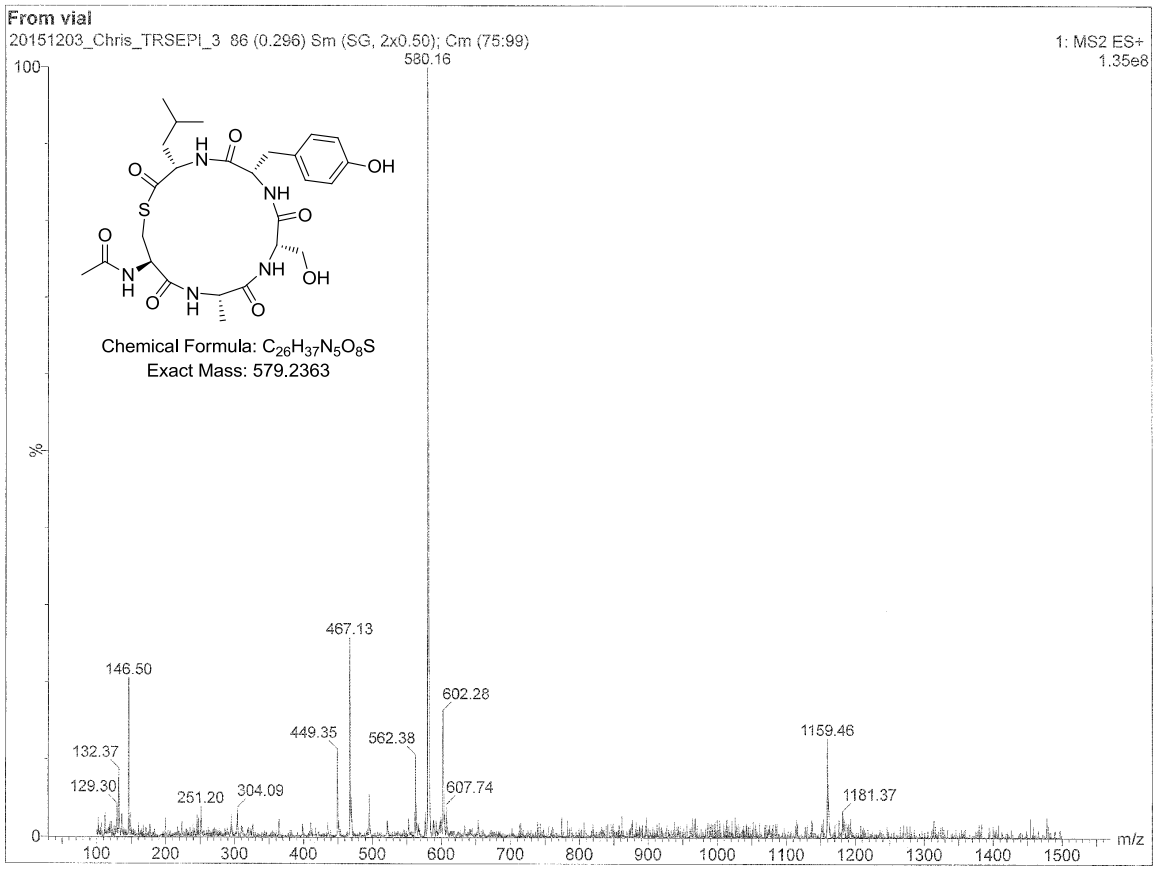
Area Percent Report

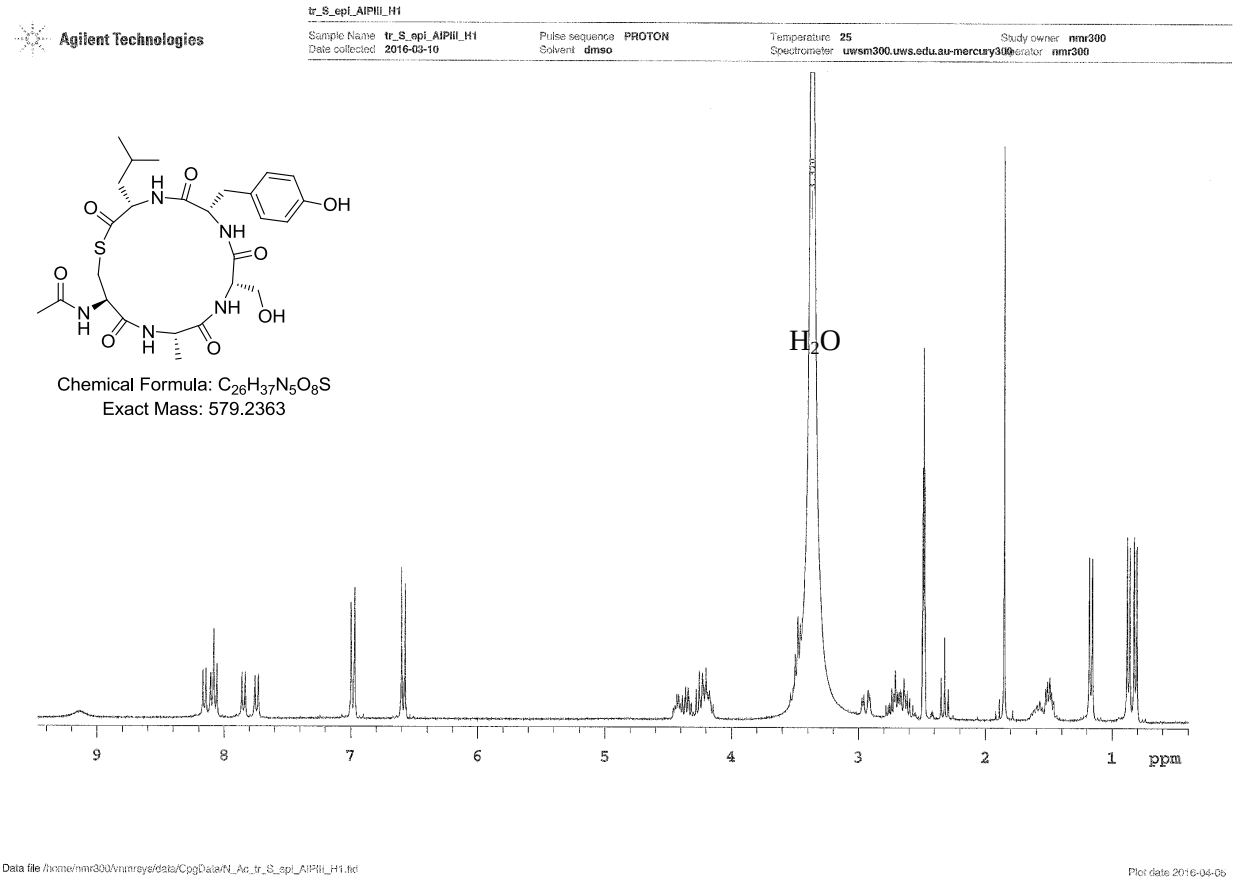
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

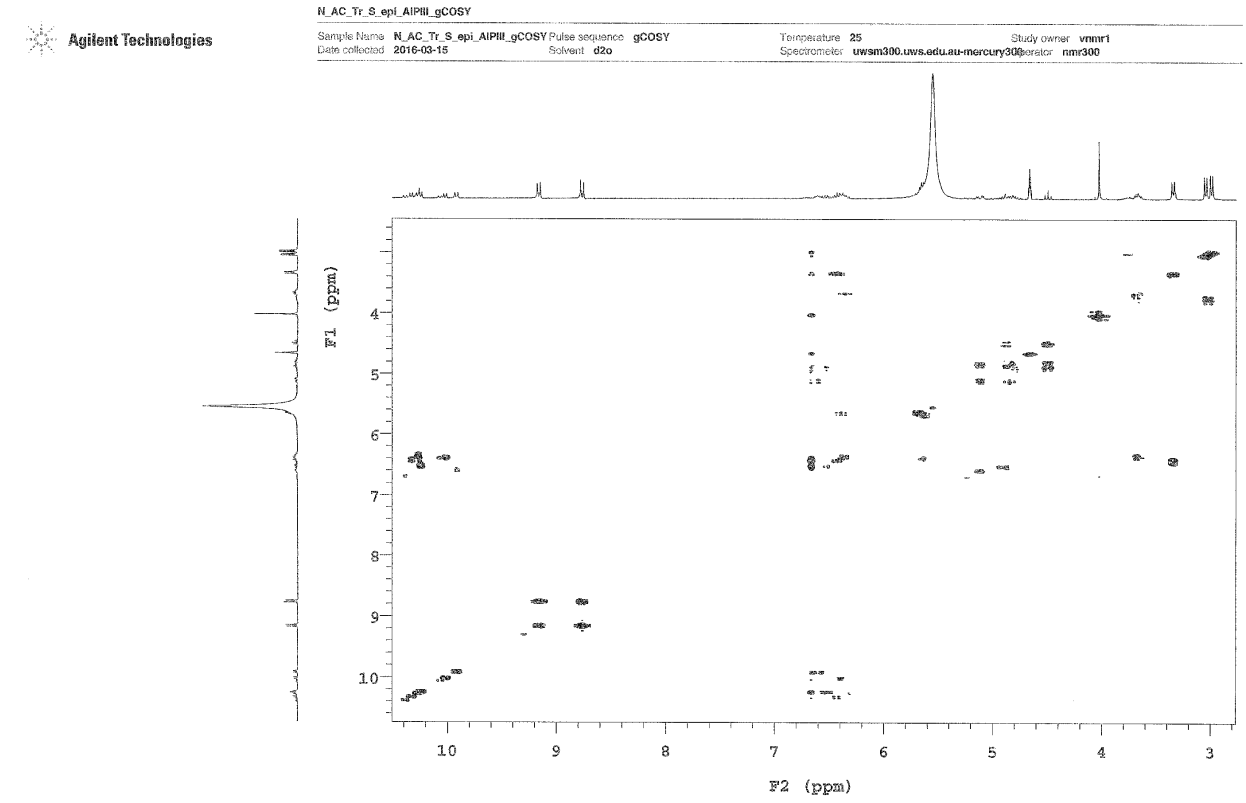
Signal 1: DAD1 A, Sig=254,4 Ref=off

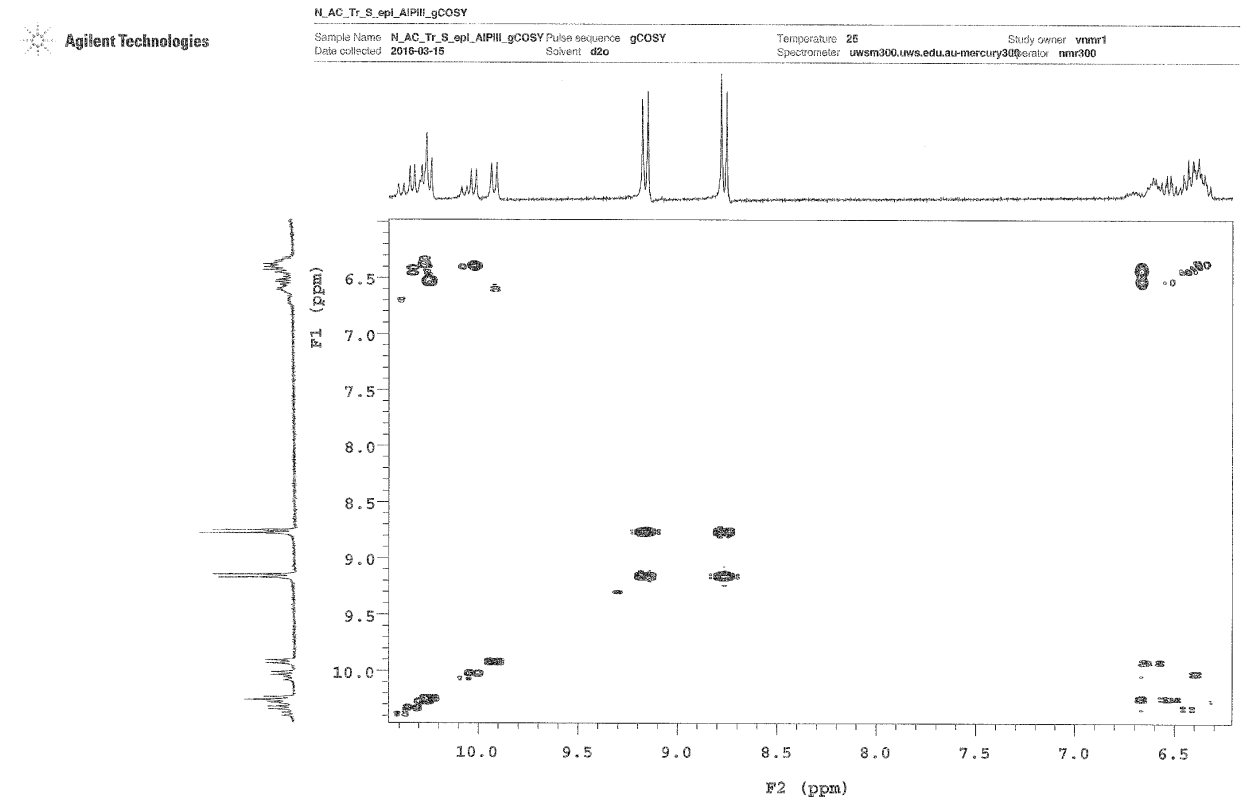
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.797	BB	0.1759	1600.58044	127.94724	73.1662
2	6.553	VV	0.1109	587.01422	77.06809	26.8338
Totals :				2187.59467	205.01534	

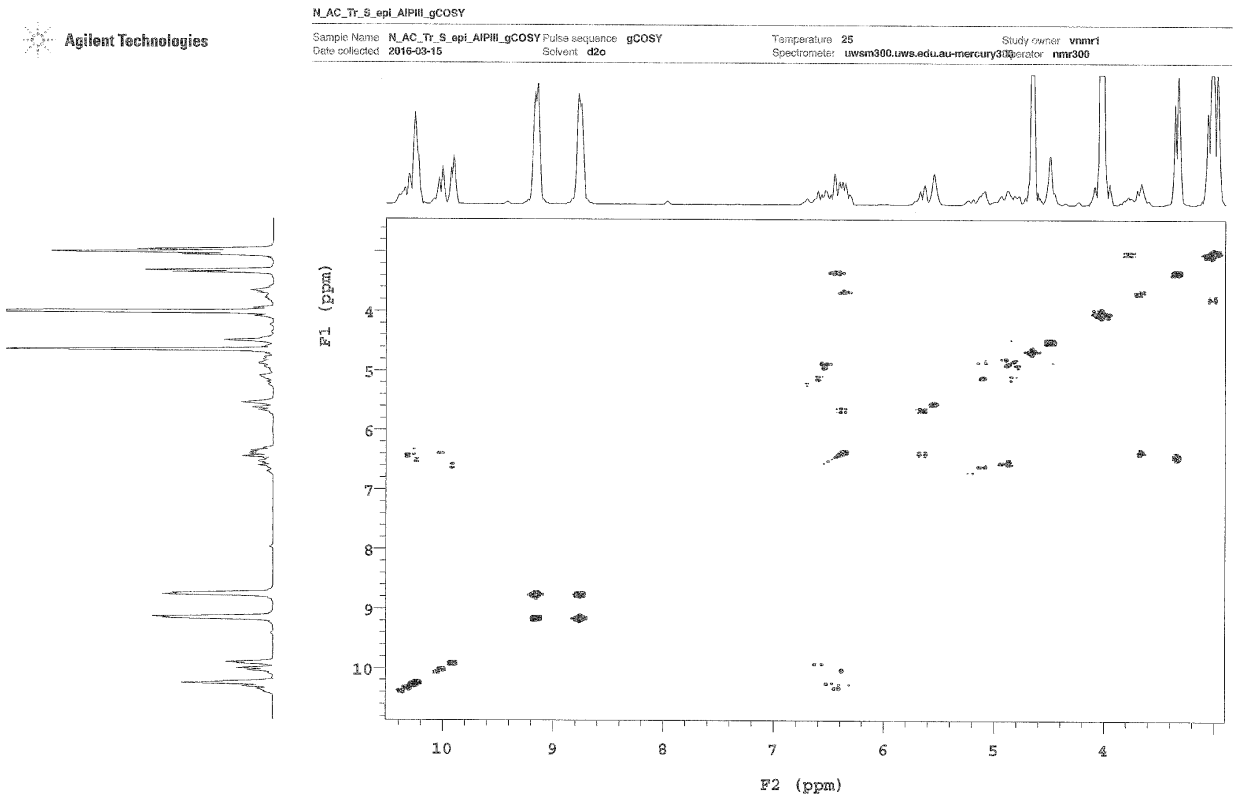
Signal 2: DAD1 B, Sig=216,4 Ref=off

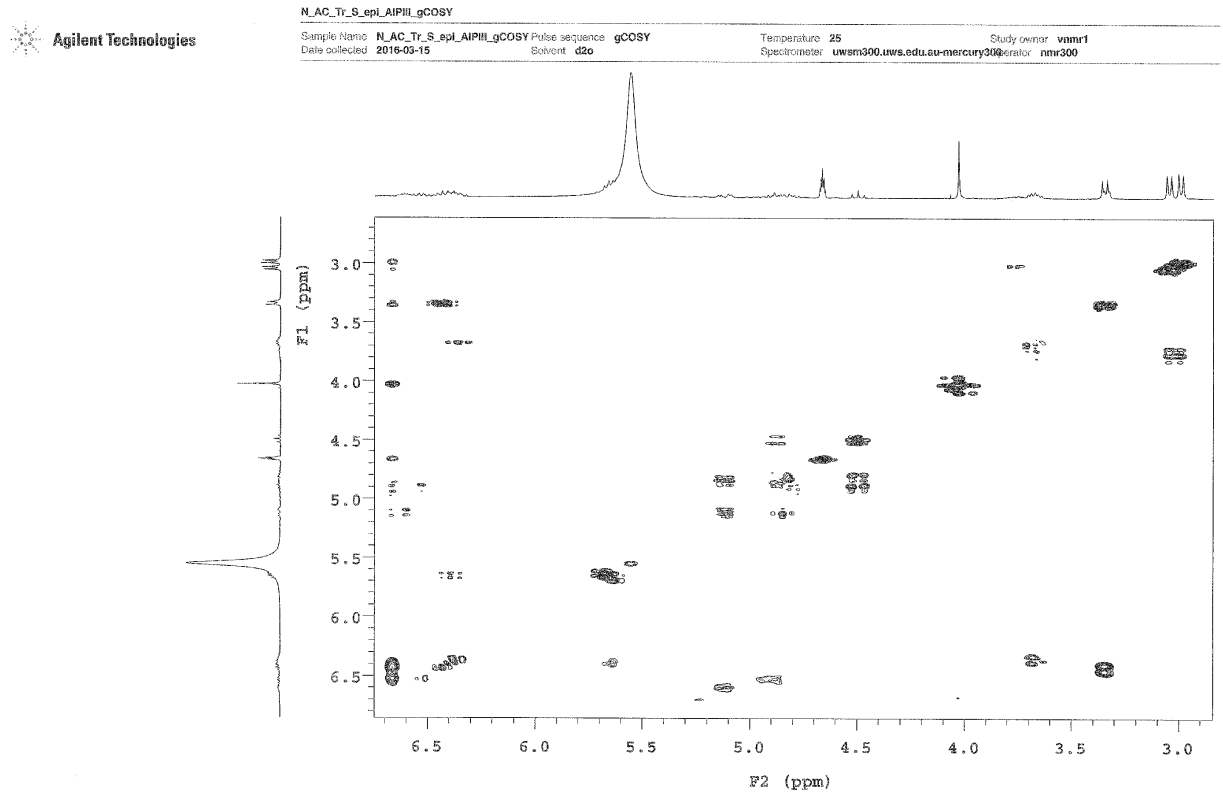

From vial
20151203_Chris_TRSEPI_3 86 (0.296) Sm (SG, 2x0.50); Cm (75:99)
1: MS2 ES+
1.35e8
580.16
Chemical Formula: C26H37N5O8S
Exact Mass: 579.2363
467.13
146.50
602.28
449.35
562.38
1159.46
132.37
129.30
251.20
304.09
607.74
1181.37
%
m/z

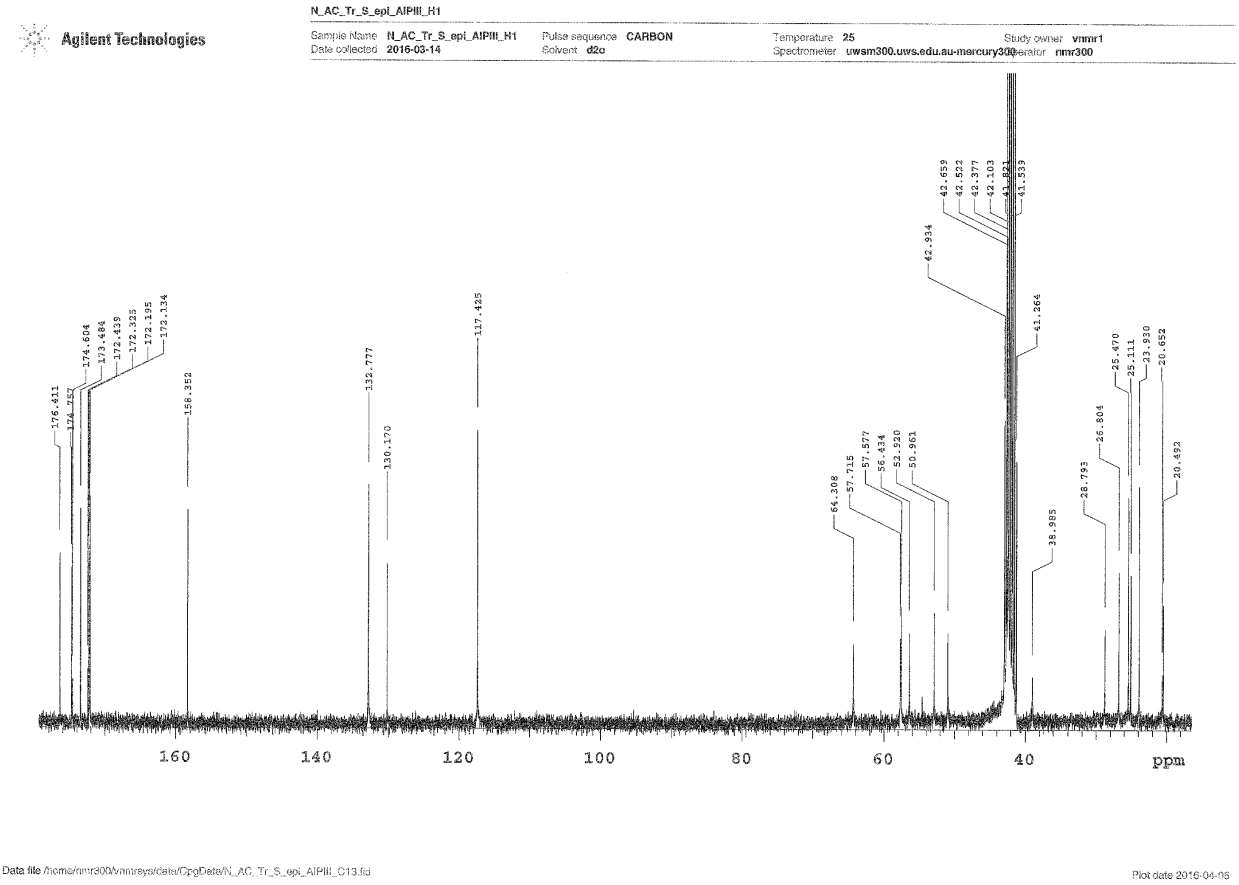

tr_S_epi_AlPlil_H1
Agilent Technologies
Sample Name tr_S_epi_AlPlil_H1
Date collected 2016-03-10
Pulse sequence PROTON
Solvent dmso
Temperature 25
Spectrometer uwsm300.uws.edu.au-mercury300
Study owner nmr300
Operator nmr300
Chemical Formula: C26H37N5O8S
Exact Mass: 579.2363
H2O
9
8
7
6
5
4
3
2
1
ppm
Data file /home/nmr300/vnmrsys/data/CpgData/N_Ac_tr_S_epi_AlPlil_H1.fid
Plot date 2016-04-06

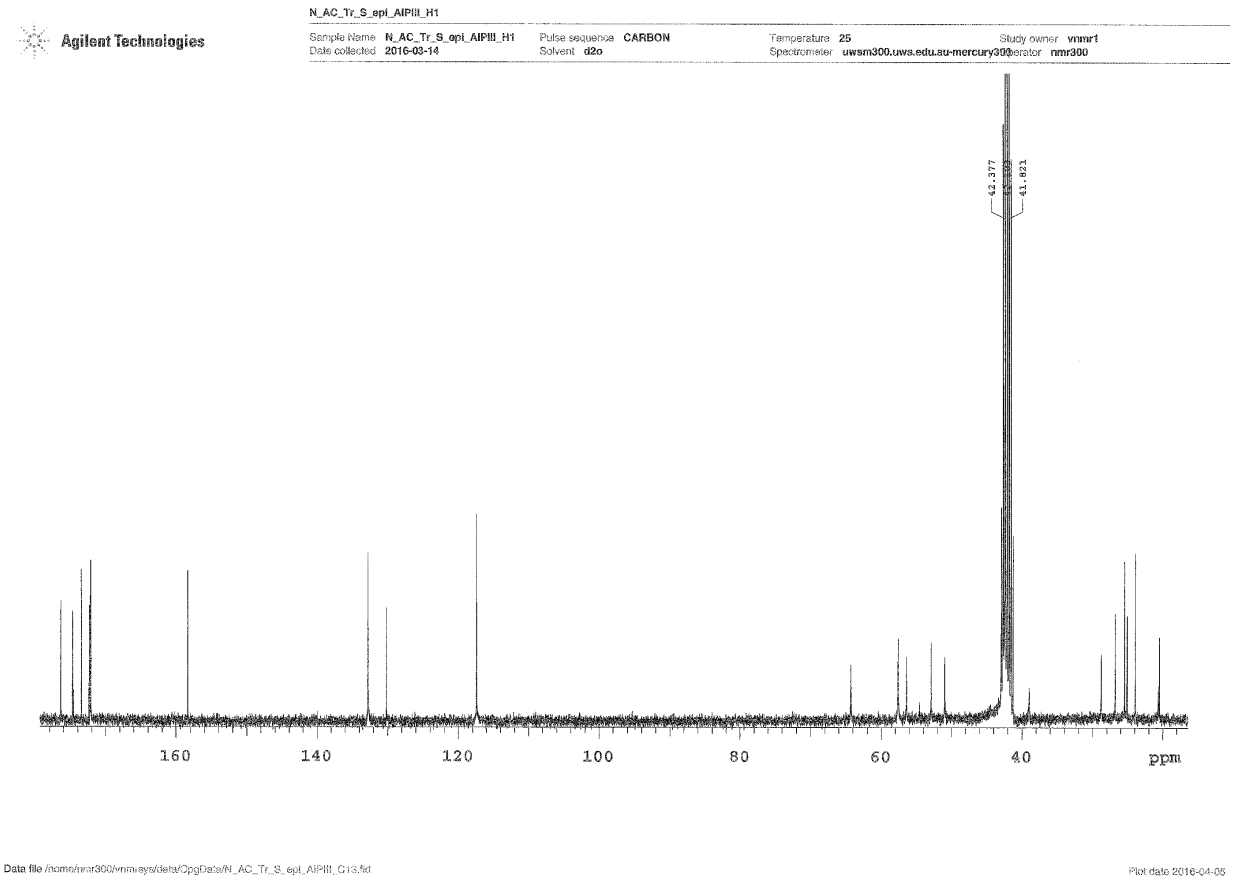

Agilent Technologies
N_AC_Tr_S_epi_AlPIII_gCOSY
Sample Name N_AC_Tr_S_epi_AlPIII_gCOSY Pulse sequence gCOSY
Date collected 2016-03-15 Solvent d2o
Temperature 25
Spectrometer uwsm300.uws.edu.au-mercury300
Study owner vnmr1
Operator nmr300
F1 (ppm)
F2 (ppm)
Data file /home/nmr300/vnmrsys/data/N_AC_Tr_S_epi_AlPIII_gCOSY/N_AC_Tr_S_epi_AlPIII_gCOSY_gCOSY_d2o_20160315_01.fid
Plot date 2016-03-15


Agilent Technologies
N_AC_Tr_S_epi_AlPIII_gCOSY
Sample Name N_AC_Tr_S_epi_AlPIII_gCOSY Pulse sequence gCOSY
Date collected 2016-03-15 Solvent d2o
Temperature 25
Spectrometer uwsm300.uws.edu.au-mercury300
Study owner vnmr1
Operator nmr300
F1 (ppm)
6.5
7.0
7.5
8.0
8.5
9.0
9.5
10.0
F2 (ppm)
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
Data file /home/nmr300/vnmrsys/data/N_AC_Tr_S_epi_AlPIII_gCOSY/N_AC_Tr_S_epi_AlPIII_gCOSY_gCOSY_d2o_20160315_01.fid
Plot date 2016-03-15

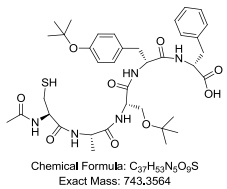

Agilent Technologies
N_AC_Tr_S_epi_AlPIII_gCOSY
Sample Name N_AC_Tr_S_epi_AlPIII_gCOSY Pulse sequence gCOSY
Temperature 25
Study owner vnmrj
Date collected 2016-03-15
Solvent d2o
Spectrometer uwsm300.uws.edu.au-mercury300
Operator nmr300
F1 (ppm)
F2 (ppm)
Data file exp
Plot date 2016-03-15


Agilent Technologies
N_AC_Tr_S_epi_AlPIII_gCOSY
Sample Name N_AC_Tr_S_epi_AlPIII_gCOSY Pulse sequence gCOSY Temperature 25 Study owner vnmr1
Date collected 2016-03-15 Solvent d2o Spectrometer uwsm300.uws.edu.au-mercury300 Operator nmr300
F1 (ppm)
F2 (ppm)
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
Data file /home/nmr300/vnmrsys/data/N_AC_Tr_S_epi_AlPIII_gCOSY/N_AC_Tr_S_epi_AlPIII_gCOSY_gCOSY_d2o_20160315_01.fid
Plot date 2016-03-15

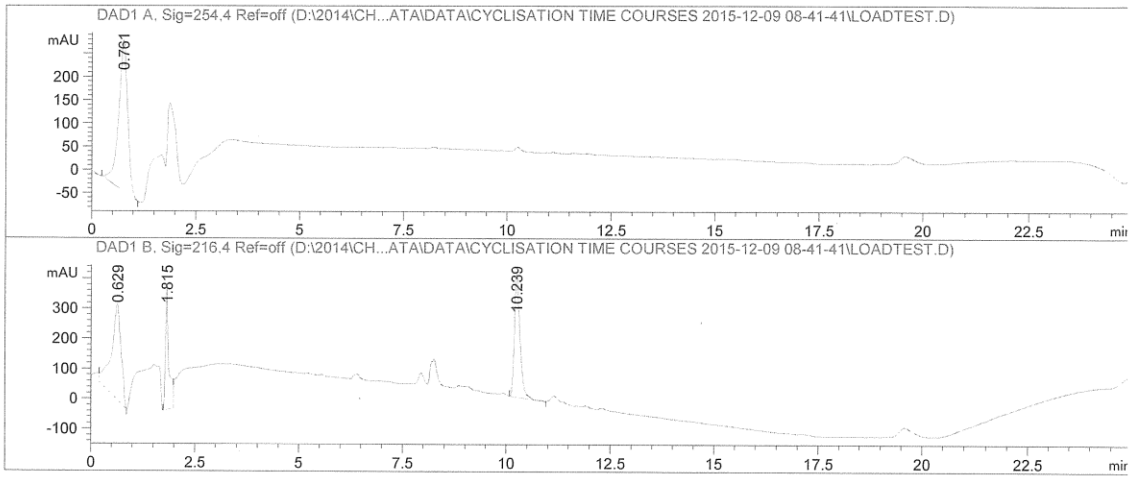

Agilent Technologies
N_AC_Tr_S_epi_AlPIII_H1
Sample Name N_AC_Tr_S_epi_AlPIII_H1
Date collected 2016-03-14
Pulse sequence CARBON
Solvent d2o
Temperature 25
Spectrometer uwsm300.uws.edu.au-mercury300
Study owner vnmr1
Operator nmr300
176.411
174.252
174.604
173.484
172.439
172.325
172.195
172.134
158.352
132.777
130.170
117.425
64.308
57.715
57.577
56.434
52.920
50.961
42.934
42.659
42.522
42.377
42.103
41.821
41.539
41.264
38.985
28.793
26.804
25.470
25.111
23.930
20.652
20.492
160
140
120
100
80
60
40
ppm
Data file /home/nmr300/vnmrsys/data/CpgData/N_AC_Tr_S_epi_AlPIII_C13.fid
Plot date 2016-04-05


N_AC_Tr_S_epi_AlPIII_H1
Agilent Technologies
Sample Name N_AC_Tr_S_epi_AlPIII_H1
Date collected 2016-03-14
Pulse sequence CARBON
Solvent d2o
Temperature 25
Spectrometer uwsm300.uws.edu.au-mercury300
Study owner vnmr1
Operator nmr300
42.377
41.821
160
140
120
100
80
60
40
ppm
Data file /home/nmr300/vnmrsys/data/OpgData/N_AC_Tr_S_epi_AlPIII_C13.fid
Plot date 2016-04-05

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\LOADTEST.D
Sample Name: S epi AIP I linear

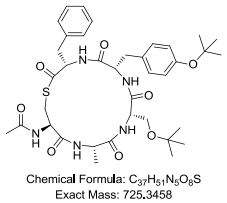



```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :   1
    Acq. Instrument : LC1260                        Location : Vial 1
    Injection Date  : 12/9/2015 8:43:37 AM               Inj :   1
                                                  Inj Volume : 40.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                      100 OV 15MIN 40UL.M
    Last changed    : 12/9/2015 8:34:25 AM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                      \10 TO 100 OV 15MIN 40UL.M (Sequence Method)
    Last changed    : 3/11/2016 3:03:37 PM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```

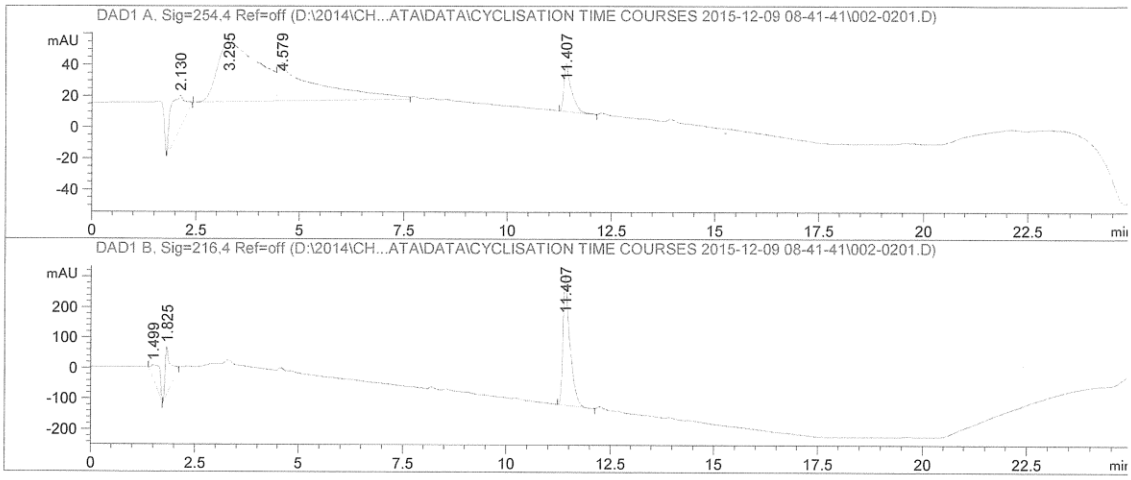



```
    ======================================================================
                            Area Percent Report
    ======================================================================

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   0.761 BB    0.2168 4873.58838  325.23016 100.0000

    Totals :                   4873.58838  325.23016
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\002-0201.D
Sample Name: Pro S epi AIP I



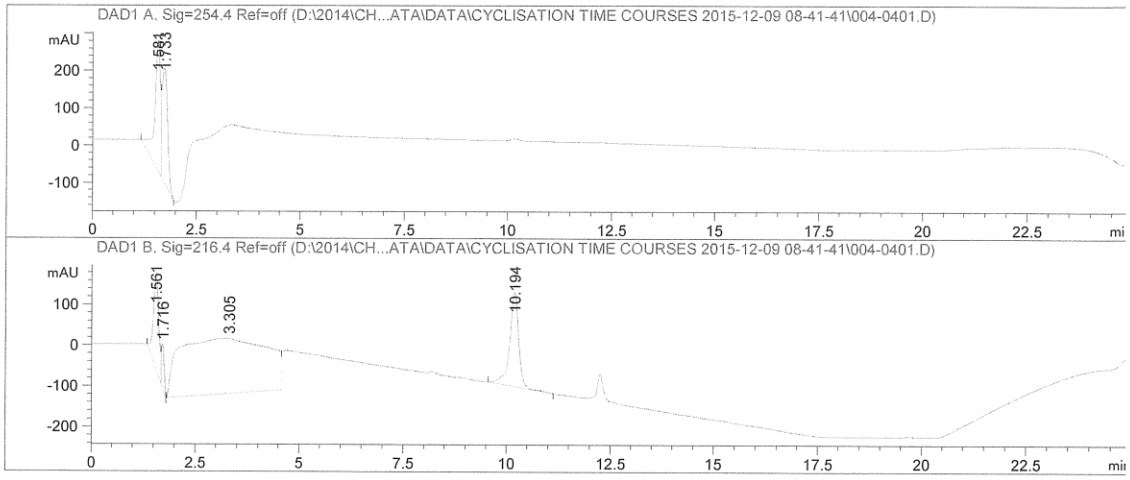

```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :   2
    Acq. Instrument : LC1260                        Location : Vial 2
    Injection Date  : 12/9/2015 9:09:51 AM               Inj :   1
                                                  Inj Volume : 20.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                      100 OV 15MIN 20UL.M
    Last changed    : 12/9/2015 8:37:43 AM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                      \10 TO 100 OV 15MIN 20UL.M (Sequence Method)
    Last changed    : 3/11/2016 3:05:03 PM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   2.130 BB    0.3460  586.05396   20.71649  11.2059
       2   3.295 BV    0.9111 2659.98999   39.12846  50.8616
       3   4.579 VV    0.7974 1601.84705   24.41543  30.6289
       4  11.407 BV    0.1722  381.96857   30.90078   7.3036

    Totals :                  5229.85956  115.16116
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\004-0401.D
Sample Name: Pro S epi AIP I EDCI T=1

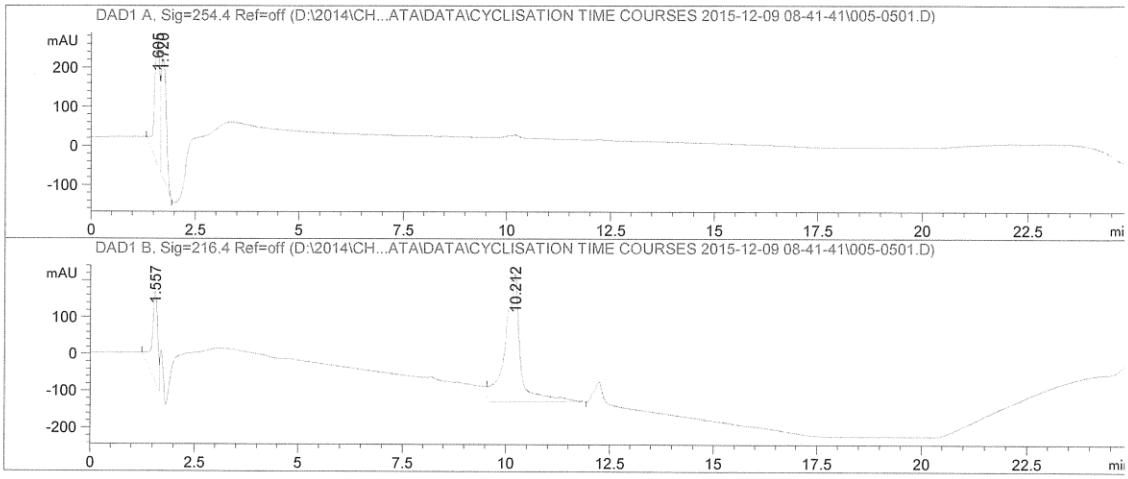
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :   4
    Acq. Instrument : LC1260                         Location : Vial 4
    Injection Date  : 12/9/2015 10:02:04 AM               Inj :   1
                                                   Inj Volume : 50.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                      100 OV 15MIN 50UL.M
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                      \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
    Last changed    : 3/11/2016 3:07:01 PM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.581 BV    0.1396 3367.25024  352.04962  54.4977
       2   1.733 VB    0.1370 2811.45020  306.56219  45.5023

    Totals :                   6178.70044  658.61182
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\005-0501.D
Sample Name: Pro S epi AIP I EDCI T=2

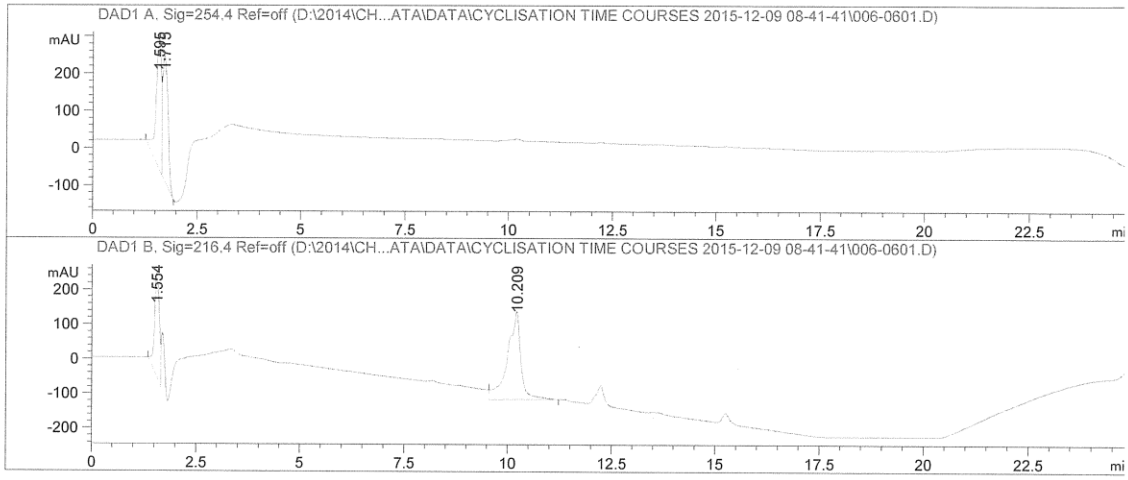
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :   5
    Acq. Instrument : LC1260                         Location : Vial 5
    Injection Date  : 12/9/2015 10:28:28 AM               Inj :   1
                                                   Inj Volume : 50.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                      100 OV 15MIN 50UL.M
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                      \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
    Last changed    : 3/11/2016 3:08:14 PM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.605 BV    0.1429 2877.90430  320.21597  52.3450
       2   1.720 VB    0.1291 2620.05444  321.40698  47.6550

    Totals :                   5497.95874  641.62296
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\006-0601.D
Sample Name: Pro S epi AIP I EDCI T=3

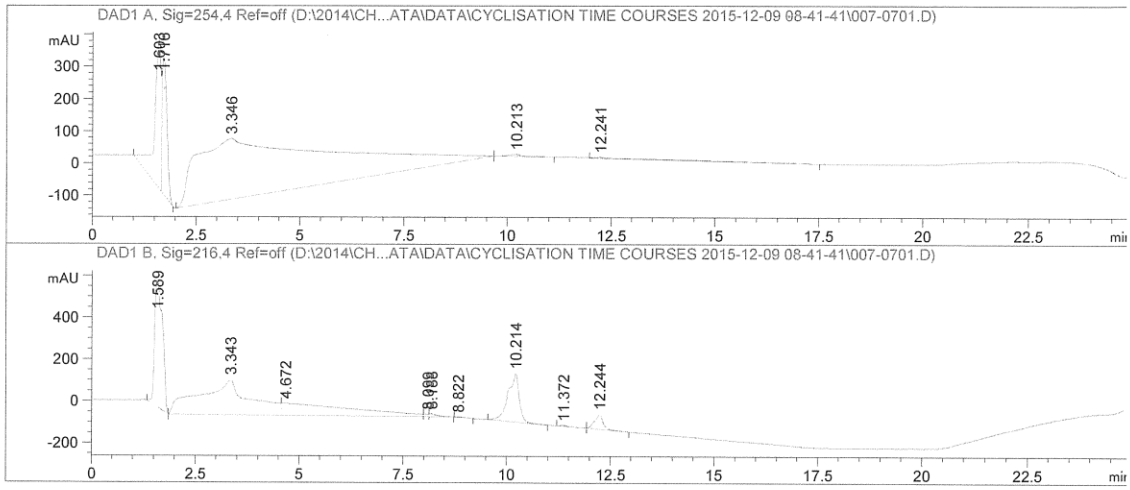
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :   6
    Acq. Instrument : LC1260                         Location : Vial 6
    Injection Date  : 12/9/2015 10:54:51 AM               Inj :   1
                                                  Inj Volume : 50.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                      100 OV 15MIN 50UL.M
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                      \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
    Last changed    : 3/11/2016 3:08:17 PM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.595 BV    0.1423 3228.24658  347.73294  54.0304
       2   1.715 VB    0.1271 2746.62378  337.04803  45.9696

    Totals :                   5974.87036  684.78098
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\007-0701.D
Sample Name: Pro S epi AIP I EDCI T=4

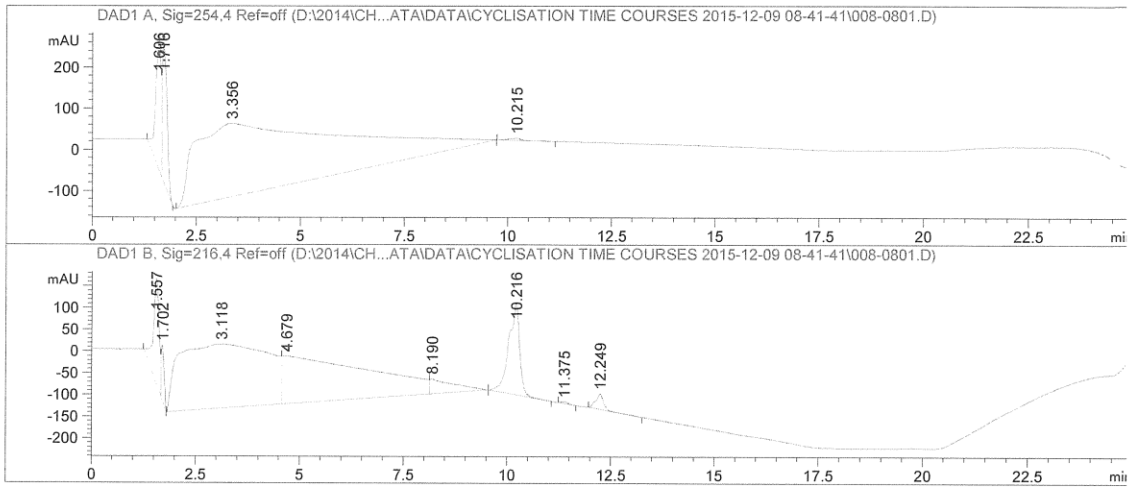
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :   7
    Acq. Instrument : LC1260                         Location : Vial 7
    Injection Date  : 12/9/2015 11:21:12 AM               Inj :   1
                                                  Inj Volume : 50.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                      100 OV 15MIN 50UL.M
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                      \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.603 BV    0.1725 5201.67871  458.07654  10.4150
       2   1.716 VB    0.1248 3381.00098  425.11902   6.7696
       3   3.346 BB    2.6305 4.09253e4   188.53644  81.9426
       4  10.213 BB    0.2607  127.26412    6.43710   0.2548
       5  12.241 BB    1.4487  308.64209    2.53808   0.6180

    Totals :                  4.99439e4  1080.70718
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\008-0801.D
Sample Name: Pro S epi AIP I EDCI T=5

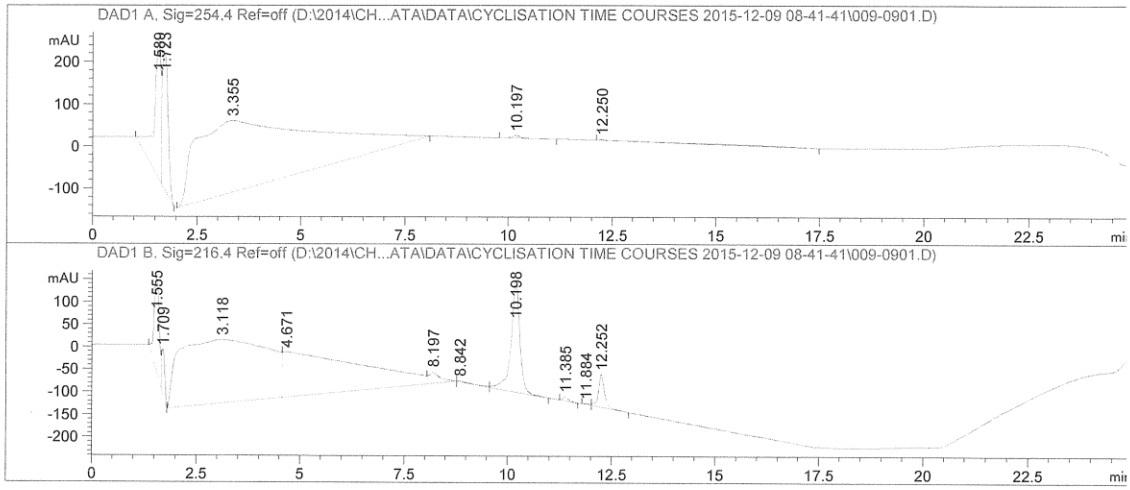
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :   8
    Acq. Instrument : LC1260                         Location : Vial 8
    Injection Date  : 12/9/2015 11:47:34 AM               Inj :   1
                                                  Inj Volume : 50.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                      100 OV 15MIN 50UL.M
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                      \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.606 BV    0.1476 2910.32202  315.53625   6.1846
       2   1.716 VB    0.1274 2650.71338  330.95630   5.6329
       3   3.356 BB    2.8918 4.13931e4   177.55893  87.9624
       4  10.215 BB    0.2443  103.56112    5.58528   0.2201

    Totals :                  4.70577e4   829.63676
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\009-0901.D
Sample Name: Pro S epi AIP I EDCI T=6

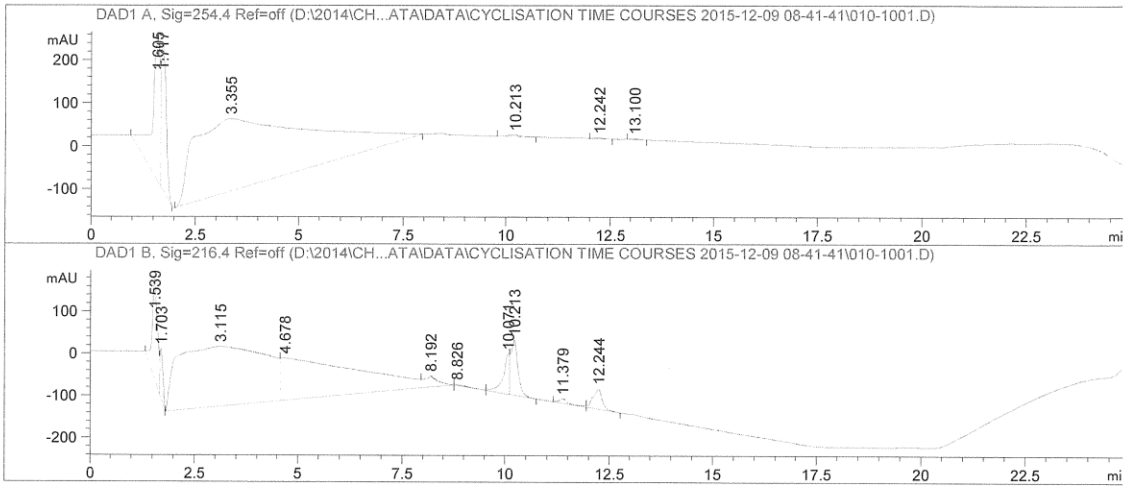
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :   9
    Acq. Instrument : LC1260                         Location : Vial 9
    Injection Date  : 12/9/2015 12:13:57 PM               Inj :   1
                                                   Inj Volume : 50.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                      100 OV 15MIN 50UL.M
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                      \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.589 BV    0.1806 4022.81079  329.05768  10.1141
       2   1.723 VB    0.1354 2870.10156  323.77527   7.2160
       3   3.355 BB    2.4141 3.25097e4   169.00912  81.7353
       4  10.197 BB    0.2156   93.89954    6.45743   0.2361
       5  12.250 BB    1.3976  277.87198    2.37042   0.6986

    Totals :                  3.97743e4   830.66993
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\010-1001.D
Sample Name: Pro S epi AIP I EDCI T=7

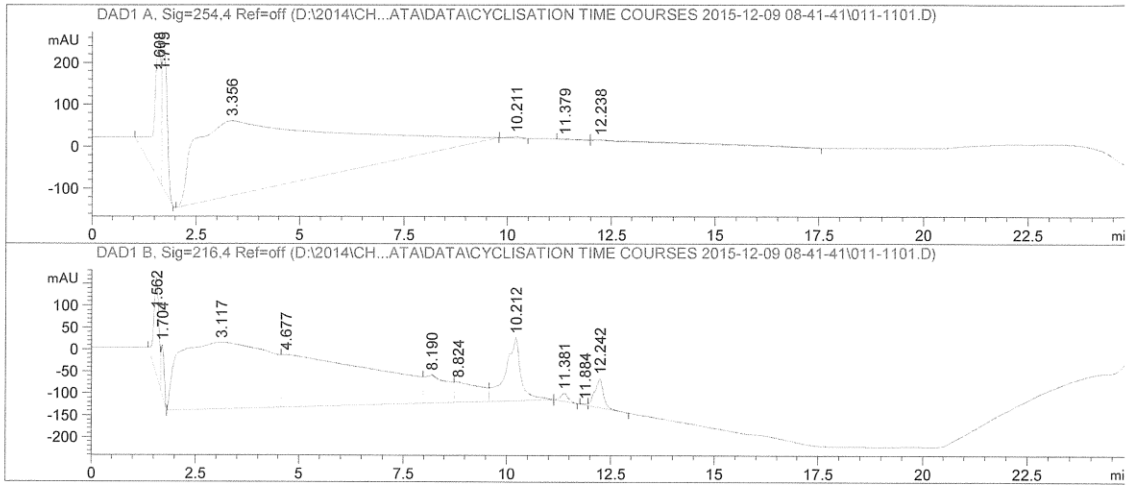
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :  10
    Acq. Instrument : LC1260                         Location : Vial 10
    Injection Date  : 12/9/2015 12:40:21 PM               Inj :   1
                                                   Inj Volume : 50.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                      100 OV 15MIN 50UL.M
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                      \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.605 BV    0.1772 4489.14160  329.06613  11.4893
       2   1.717 VB    0.1265 2783.65601  343.53024   7.1244
       3   3.355 BB    2.3539 3.16887e4   168.51677  81.1029
       4  10.213 BB    0.2389   64.03980    3.57667   0.1639
       5  12.242 BB    0.1880   22.24874    1.60204   0.0569
       6  13.100 BB    0.2078   24.43504    1.87783   0.0625

    Totals :                  3.90722e4   848.16968
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\011-1101.D
Sample Name: Pro S epi AIP I EDCI T=8

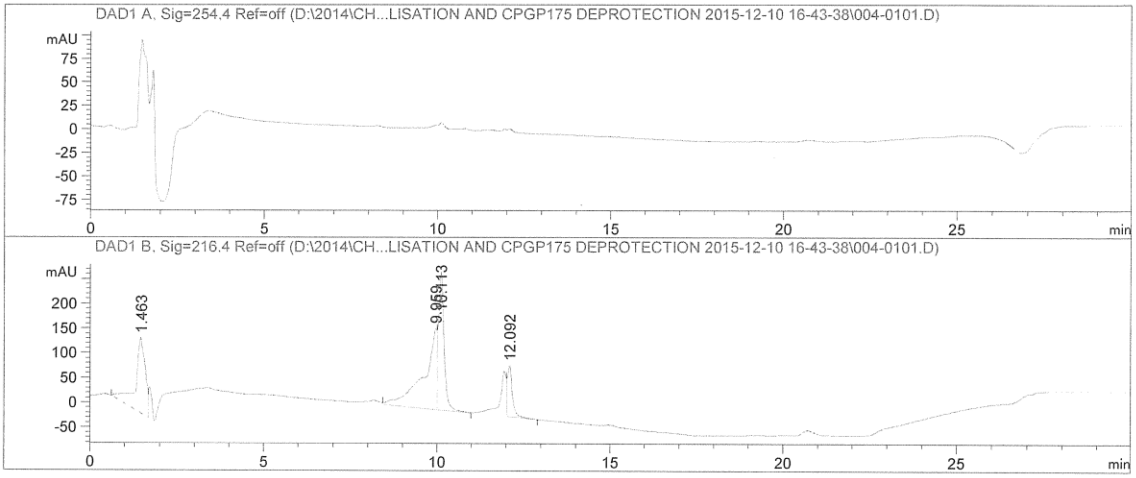
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :  11
    Acq. Instrument : LC1260                         Location : Vial 11
    Injection Date  : 12/9/2015 1:06:46 PM                Inj :   1
                                                   Inj Volume : 50.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                      100 OV 15MIN 50UL.M
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                      \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.608 BV    0.1869 4166.40869  335.40826   8.4552
       2   1.715 VB    0.1096 2818.24805  343.94464   5.7193
       3   3.356 BB    2.9109 4.17501e4   177.70731  84.7267
       4  10.211 BB    0.2299   58.64925    3.42352   0.1190
       5  11.379 BV    0.3491   37.02816    1.39740   0.0751
       6  12.238 VB    1.7572  445.76926    3.01096   0.9046

    Totals :                  4.92762e4   864.89209
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\004-0101.D
Sample Name: Pro S epi AIP I EDCI T=1

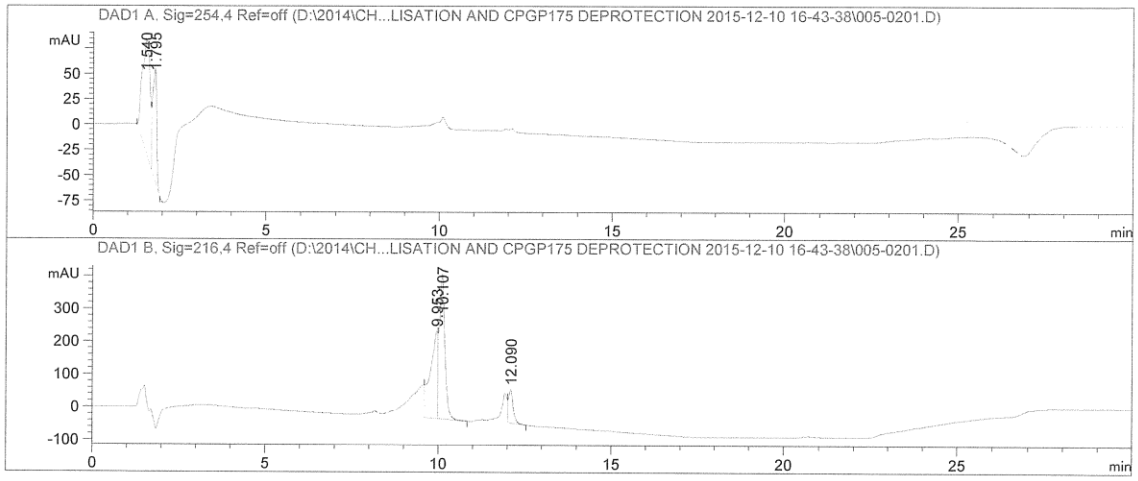
```
    ======================================================================
    Acq. Operator    : Simi120102015                 Seq. Line :   1
    Acq. Instrument : LC1260                          Location : Vial 4
    Injection Date   : 12/10/2015 4:45:50 PM               Inj :   1
                                                    Inj Volume : 100.000 µl
    Acq. Method      : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                       -38\10 TO 100 OV 15MIN 100UL.M
    Last changed     : 12/10/2015 4:12:03 PM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                       10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
    Last changed     : 12/14/2015 1:10:24 PM by Simi120102015
                       (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off


    Signal 2: DAD1 B, Sig=216,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.463 BV    0.2446 3036.20068  153.58002  26.0546
       2   9.959 VV    0.3315 4390.29346  164.40900  37.6746
       3  10.113 VB    0.1585 3154.84546  277.88388  27.0728
       4  12.092 VB    0.1419 1071.86414  104.28788   9.1980

    Totals :                  1.16532e4   700.16077
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\005-0201.D
Sample Name: Pro S epi AIP I EDCI T=2

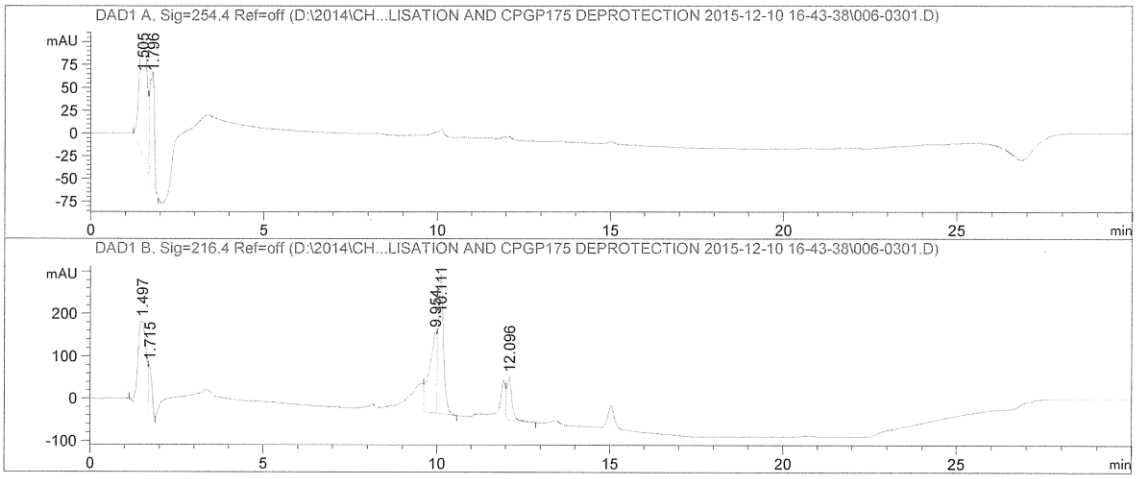
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :   2
    Acq. Instrument : LC1260                         Location : Vial 5
    Injection Date  : 12/10/2015 5:17:40 PM               Inj :   1
                                                   Inj Volume : 100.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                      -38\10 TO 100 OV 15MIN 100UL.M
    Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                      10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
    Last changed    : 3/15/2016 8:54:32 AM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.540 BV    0.1986 1781.25818  112.59834  67.5538
       2   1.795 VB    0.1072  855.54169  111.93997  32.4462

    Totals :                   2636.79987  224.53831
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\006-0301.D
Sample Name: Pro S epi AIP I EDCI T=3

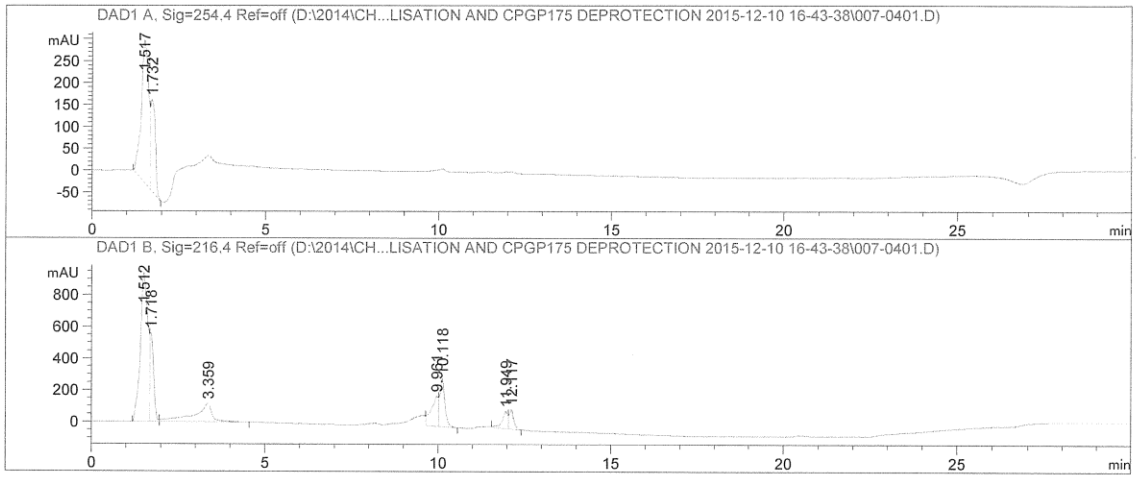
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :   3
    Acq. Instrument : LC1260                         Location : Vial 6
    Injection Date  : 12/10/2015 5:49:29 PM               Inj :   1
                                                   Inj Volume : 100.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                      -38\10 TO 100 OV 15MIN 100UL.M
    Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                      10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
    Last changed    : 3/15/2016 8:54:32 AM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.505 BV    0.2453 2191.87305  128.33954  68.0926
       2   1.796 VB    0.1123 1027.08752  124.38364  31.9074

    Totals :                   3218.96057  252.72318
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\007-0401.D
Sample Name: Pro S epi AIP I EDCI T=4

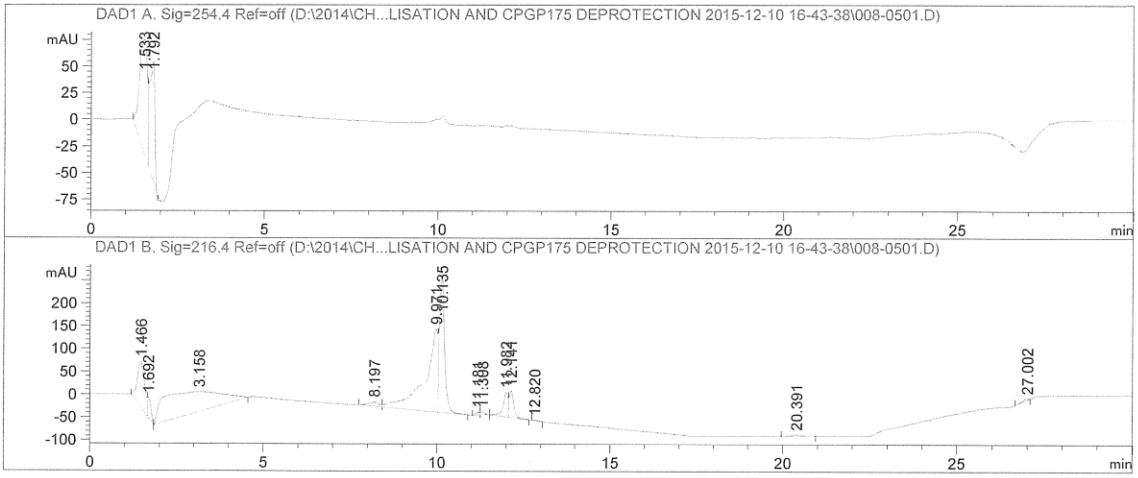
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :   4
    Acq. Instrument : LC1260                         Location : Vial 7
    Injection Date  : 12/10/2015 6:21:19 PM               Inj :   1
                                                  Inj Volume : 100.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                      -38\10 TO 100 OV 15MIN 100UL.M
    Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                      10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
    Last changed    : 3/15/2016 8:55:17 AM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.517 BV    0.1917 4909.50439  326.01376  72.3912
       2   1.732 VB    0.1158 1872.40393  210.45964  27.6088

    Totals :                   6781.90833  536.47340
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\008-0501.D
Sample Name: Pro S epi AIP I EDCI T=5

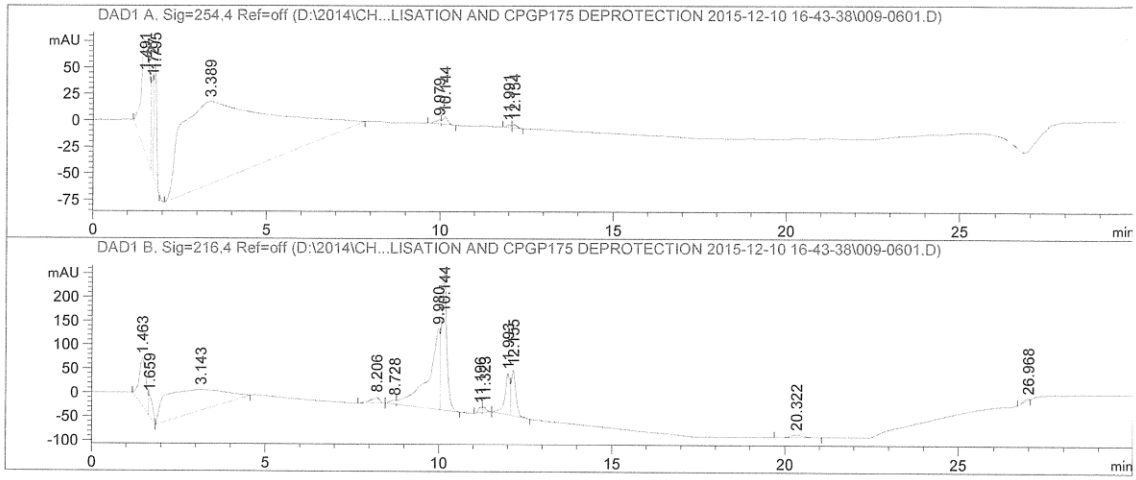
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :   5
    Acq. Instrument : LC1260                         Location : Vial 8
    Injection Date  : 12/10/2015 6:53:08 PM               Inj :   1
                                                  Inj Volume : 100.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                      -38\10 TO 100 OV 15MIN 100UL.M
    Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                      10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
    Last changed    : 3/15/2016 8:56:26 AM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.533 BV    0.2171 1665.01831  107.28703  60.6916
       2   1.792 VB    0.1301 1078.38867  112.23992  39.3084

    Totals :                   2743.40698  219.52695
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\009-0601.D
Sample Name: Pro S epi AIP I EDCI T=6

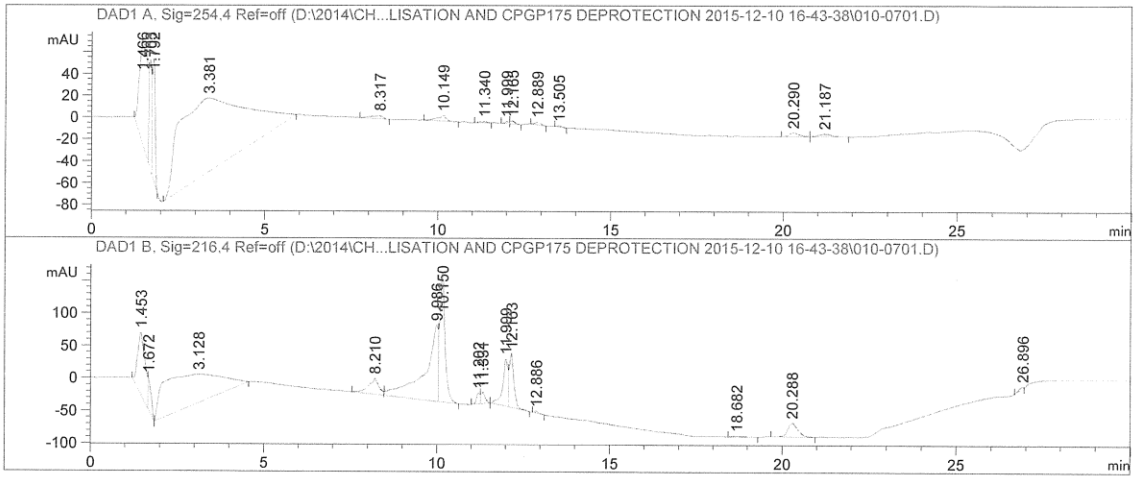
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :   6
    Acq. Instrument : LC1260                         Location : Vial 9
    Injection Date  : 12/10/2015 7:24:58 PM               Inj :   1
                                                  Inj Volume : 100.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                      -38\10 TO 100 OV 15MIN 100UL.M
    Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                      10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
    Last changed    : 3/15/2016 8:56:35 AM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.491 BV    0.2361 1843.86450  105.40421  10.8162
       2   1.727 VV    0.0568  341.28214   89.78984   2.0020
       3   1.795 VB    0.0843  635.22333  111.85163   3.7263
       4   3.389 BB    2.2422 1.40668e4    77.69736  82.5165
       5   9.979 BV    0.1553   42.23698    3.69582   0.2478
       6  10.144 VB    0.1404   69.88574    7.12447   0.4100
       7  11.991 BV    0.1196   21.28784    2.54482   0.1249
       8  12.154 VB    0.1232   26.67034    3.07549   0.1564

    Totals :                  1.70472e4   401.18364
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\010-0701.D
Sample Name: Pro S epi AIP I EDCI T=7

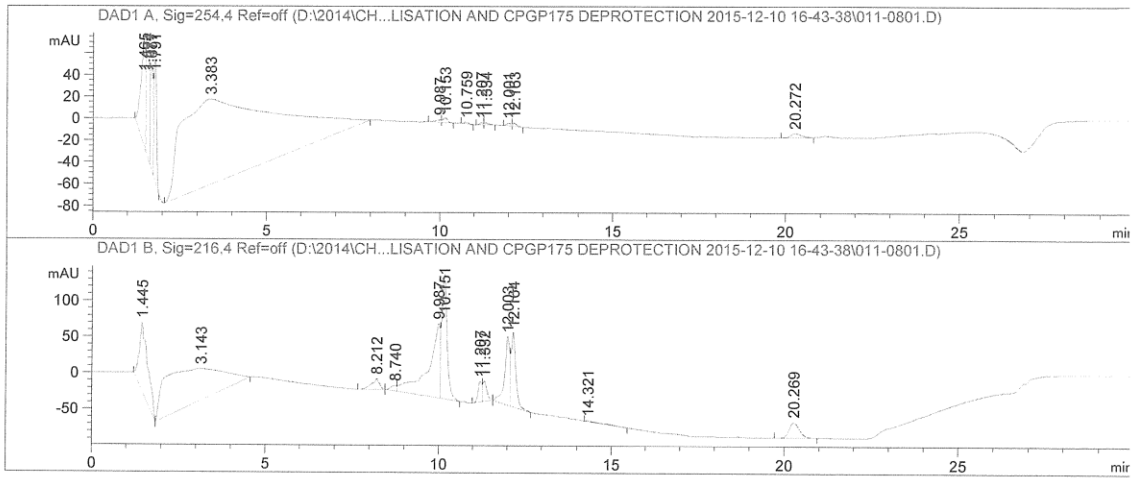
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :   7
    Acq. Instrument : LC1260                         Location : Vial 10
    Injection Date  : 12/10/2015 7:56:50 PM               Inj :   1
                                                   Inj Volume : 100.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                      -38\10 TO 100 OV 15MIN 100UL.M
    Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                      10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
    Last changed    : 3/15/2016 8:56:55 AM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.466 BV    0.2237 1583.25232   94.39129  13.0924
       2   1.703 VV    0.0786  556.03668  100.40852   4.5980
       3   1.792 VB    0.0808  579.42816  111.17802   4.7915
       4   3.381 BB    1.6932 8997.04297   67.75424  74.3994
       5   8.317 BB    0.2922   67.80720    3.03977   0.5607
       6  10.149 BB    0.2199   86.47871    5.20270   0.7151
       7  11.340 BB    0.2114   18.46382    1.17442   0.1527
       8  11.999 BV    0.1154   16.60037    2.07312   0.1373
       9  12.165 VB    0.1302   26.37010    2.89770   0.2181
      10  12.889 BB    0.1486   27.80781    2.64722   0.2300
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\011-0801.D
Sample Name: Pro S epi AIP I EDCI T=8

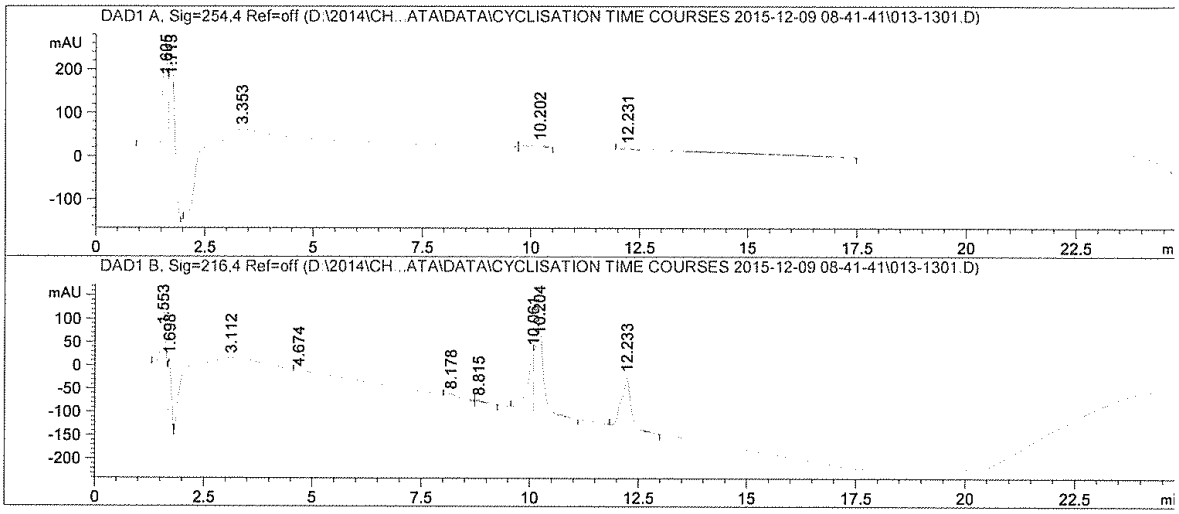
```
    ======================================================================
    Acq. Operator   : Simi120102015                  Seq. Line :   8
    Acq. Instrument : LC1260                          Location : Vial 11
    Injection Date  : 12/10/2015 8:28:43 PM               Inj :   1
                                                    Inj Volume : 100.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                      -38\10 TO 100 OV 15MIN 100UL.M
    Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                      10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
    Last changed    : 3/15/2016 8:57:13 AM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.465 BV    0.1316  937.44293   96.23300   5.3705
       2   1.574 VV    0.0891  667.00934  103.61787   3.8212
       3   1.677 VV    0.0874  542.16125   93.82185   3.1060
       4   1.791 VB    0.0814  608.92010  112.03243   3.4884
       5   3.383 BB    2.2716 1.44600e4    78.48016  82.8390
       6   9.987 BV    0.1528   25.63412    2.25257   0.1469
       7  10.153 VB    0.1351   41.03677    4.38950   0.2351
       8  10.759 BB    0.1353   13.90667    1.51162   0.0797
       9  11.207 BV    0.1033   13.99164    2.00774   0.0802
      10  11.334 VB    0.1380   20.33339    2.08010   0.1165
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\013-1301.D
Sample Name: Pro S epi AIP I DCC T=1

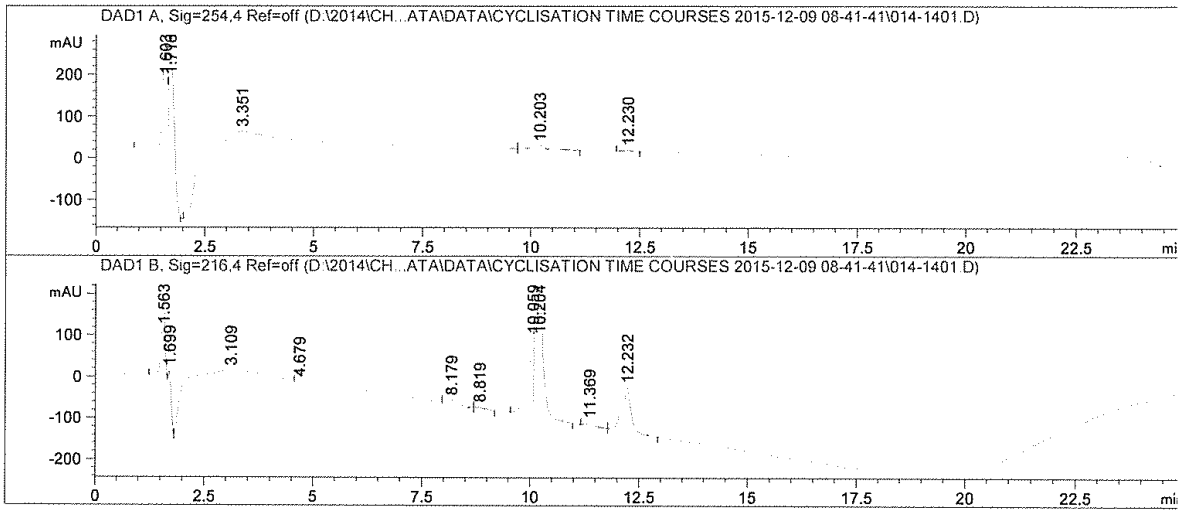
```
==========================================================================
Acq. Operator   : Simi120102015                 Seq. Line :  13
Acq. Instrument : LC1260                       Location : Vial 13
Injection Date  : 12/9/2015 1:59:28 PM              Inj :   1
                                               Inj Volume : 50.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                  100 OV 15MIN 50UL.M
Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                  \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
==========================================================================
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   1.605 BV    0.1915 4572.15869  347.44028   9.2912
   2   1.713 VB    0.1272 2888.10840  353.98679   5.8690
   3   3.353 BB    2.8775 4.13539e4   177.40935  84.0364
   4  10.202 BB    0.2351   90.66362    5.15845   0.1842
   5  12.231 BB    0.9588  304.67273    3.82686   0.6191

Totals :                  4.92095e4   887.82172
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\014-1401.D
Sample Name: Pro S epi AIP I DCC T=2

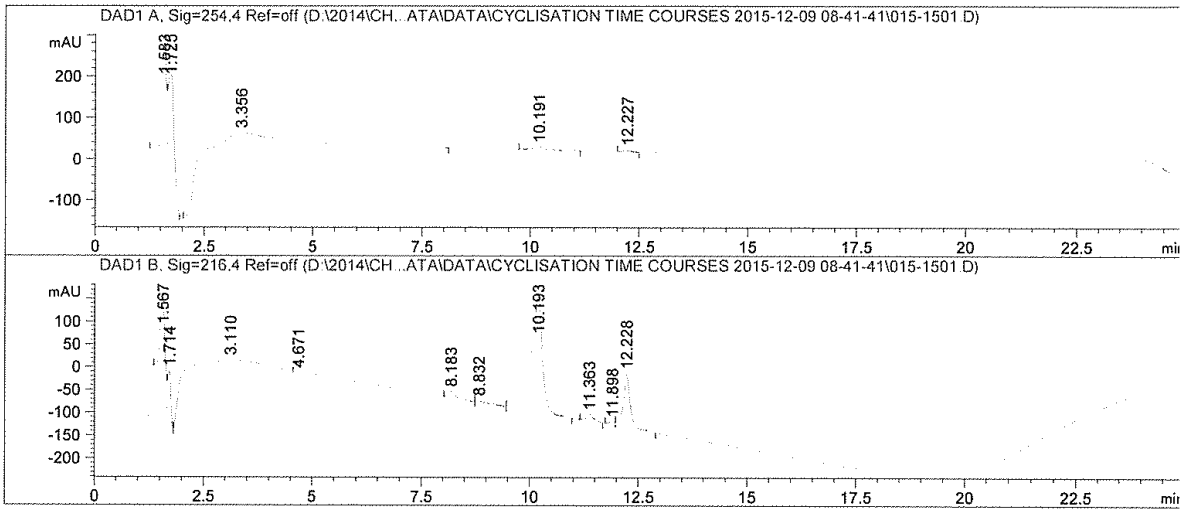
```
==========================================================================
Acq. Operator   : Simi120102015                 Seq. Line :  14
Acq. Instrument : LC1260                       Location : Vial 14
Injection Date  : 12/9/2015 2:25:50 PM              Inj :   1
                                              Inj Volume : 50.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                  100 OV 15MIN 50UL.M
Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                  \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
==========================================================================
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   1.603 BV    0.1872 4779.18799  363.93860   9.7463
   2   1.716 VB    0.1259 2873.19458  357.02588   5.8593
   3   3.351 BB    2.8653 4.11772e4   177.31512  83.9732
   4  10.203 BB    0.2592  161.51819    8.15060   0.3294
   5  12.230 BB    0.1792   44.99989    3.51958   0.0918

Totals :                  4.90361e4   909.94977
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\015-1501.D
Sample Name: Pro S epi AIP I DCC T=3

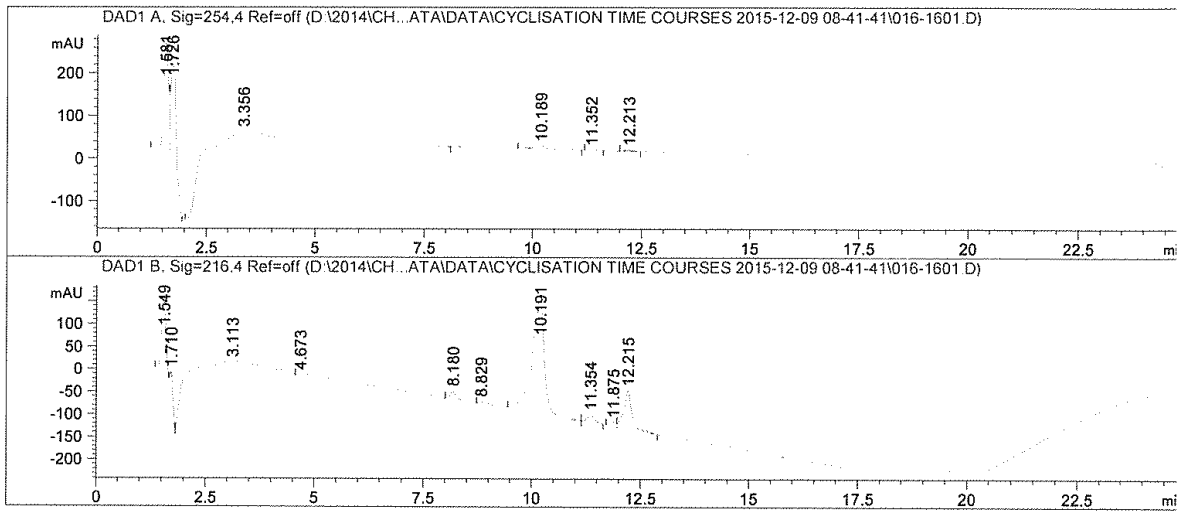
```
==========================================================================
Acq. Operator   : Simi120102015                 Seq. Line :  15
Acq. Instrument : LC1260                       Location : Vial 15
Injection Date  : 12/9/2015 2:52:11 PM              Inj :   1
                                              Inj Volume : 50.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                  100 OV 15MIN 50UL.M
Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                  \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
==========================================================================
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   1.583 BV    0.1199 2985.13208  335.22275   7.7443
   2   1.725 VB    0.1354 2756.44702  311.15768   7.1510
   3   3.356 BB    2.4180 3.26423e4   169.10605  84.6840
   4  10.191 BB    0.2544  122.40668    6.72286   0.3176
   5  12.227 BB    0.1463   39.73694    3.98539   0.1031

Totals :                  3.85461e4   826.19473
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\016-1601.D
Sample Name: Pro S epi AIP I DCC T=4

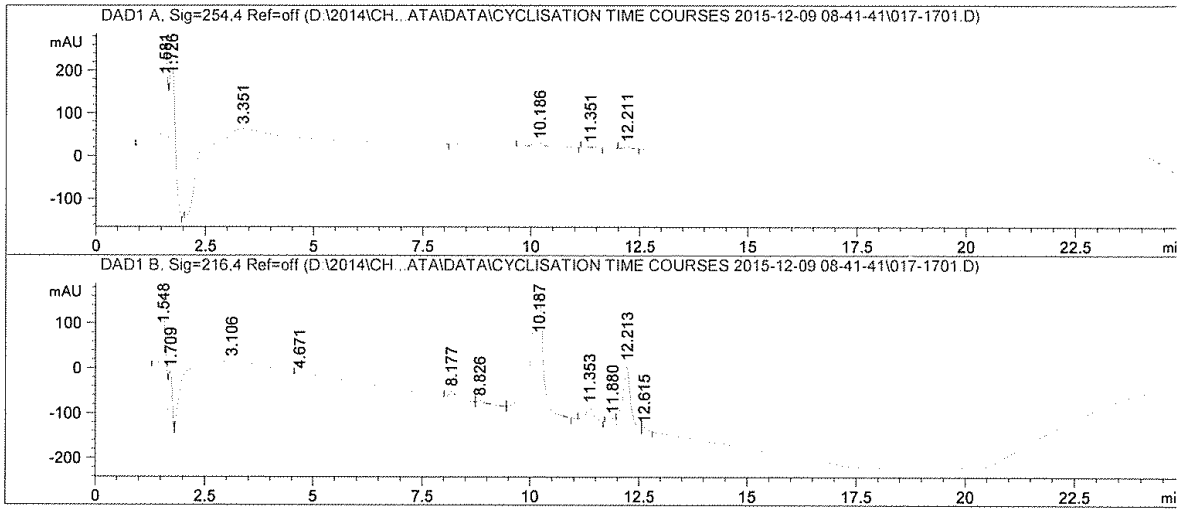
```
==========================================================================
Acq. Operator   : Simi120102015                 Seq. Line :  16
Acq. Instrument : LC1260                       Location : Vial 16
Injection Date  : 12/9/2015 3:18:32 PM              Inj :   1
                                              Inj Volume : 50.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                  100 OV 15MIN 50UL.M
Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                  \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
==========================================================================
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   1.581 BV    0.1490 3199.37695  324.70987   8.2637
   2   1.726 VB    0.1294 2664.96948  312.72717   6.8833
   3   3.356 BB    2.4054 3.26748e4   169.76886  84.3956
   4  10.189 BB    0.2567  137.00867    7.04991   0.3539
   5  11.352 BB    0.1482   10.37941    1.04253   0.0268
   6  12.213 BB    0.1620   29.70581    2.75120   0.0767

Totals :                  3.87163e4   818.04955
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\017-1701.D
Sample Name: Pro S epi AIP I DCC T=5

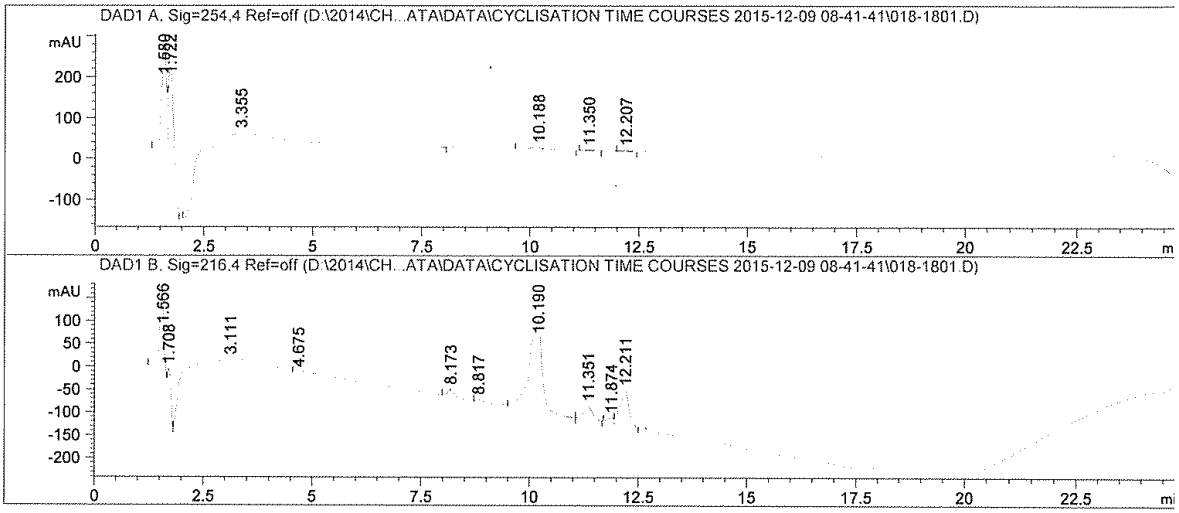
```
==========================================================================
Acq. Operator   : Simi120102015                 Seq. Line :  17
Acq. Instrument : LC1260                       Location : Vial 17
Injection Date  : 12/9/2015 3:44:54 PM              Inj :   1
                                               Inj Volume : 50.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                  100 OV 15MIN 50UL.M
Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                  \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
==========================================================================
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   1.581 BV    0.1910 4623.04297  347.97897  11.4510
   2   1.726 VB    0.1327 2822.01587  326.90796   6.9899
   3   3.351 BB    2.4078 3.27222e4   169.99074  81.0506
   4  10.186 BB    0.2514  135.57300    7.07972   0.3358
   5  11.351 BB    0.1643   21.82674    1.95492   0.0541
   6  12.211 BB    0.1620   47.89104    4.43370   0.1186

Totals :                  4.03726e4   858.34601
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\018-1801.D
Sample Name: Pro S epi AIP I DCC T=6

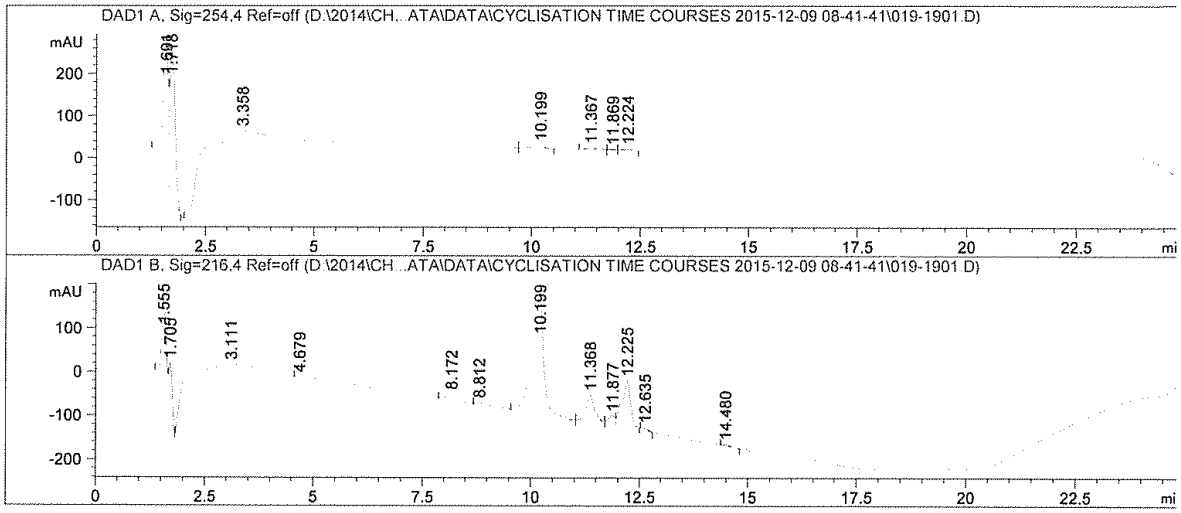
```
==========================================================================
Acq. Operator   : Simi120102015                  Seq. Line :  18
Acq. Instrument : LC1260                        Location : Vial 18
Injection Date  : 12/9/2015 4:11:15 PM               Inj :   1
                                               Inj Volume : 50.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                  100 OV 15MIN 50UL.M
Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                  \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
==========================================================================
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   1.580 BV    0.1184 2893.84497  329.67761   7.5922
   2   1.722 VB    0.1303 2587.79492  307.17178   6.7892
   3   3.355 BB    2.4080 3.24771e4   169.45660  85.2058
   4  10.188 BB    0.2570  105.20487    5.35966   0.2760
   5  11.350 BB    0.1753   24.47615    2.04994   0.0642
   6  12.207 BB    0.1749   27.63758    2.35579   0.0725

Totals :                  3.81161e4   816.07138
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\019-1901.D
Sample Name: Pro S epi AIP I DCC T=7

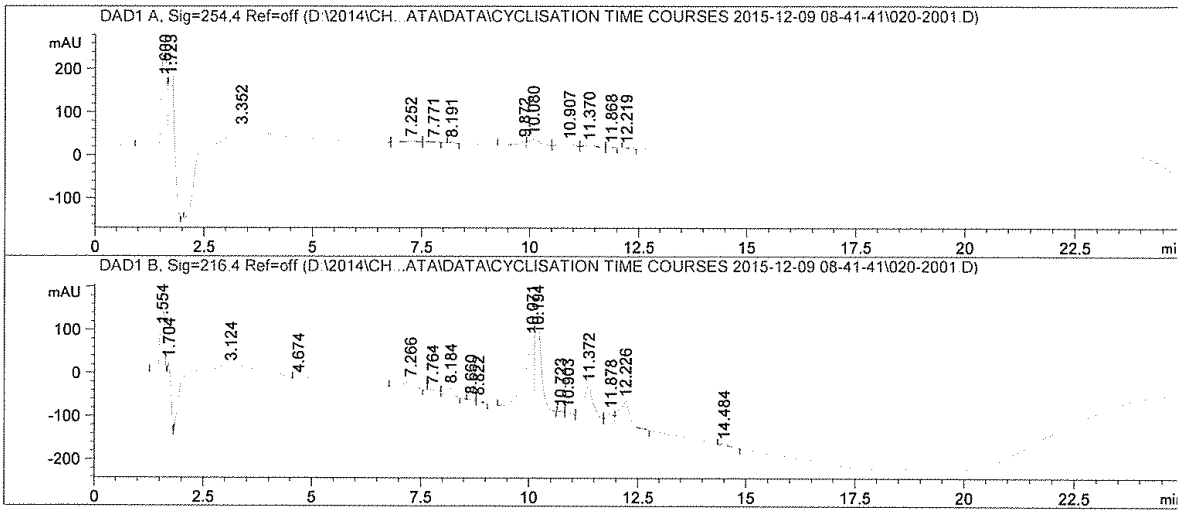
```
==========================================================================
Acq. Operator   : Simi120102015                 Seq. Line :  19
Acq. Instrument : LC1260                       Location : Vial 19
Injection Date  : 12/9/2015 4:37:37 PM              Inj :   1
                                               Inj Volume : 50.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                  100 OV 15MIN 50UL.M
Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                  \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
==========================================================================
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   1.601 BV    0.1236 3045.34766  330.10397   6.4429
   2   1.718 VB    0.1098 2739.89038  340.63965   5.7967
   3   3.358 BB    2.8858 4.12533e4   177.08905  87.2783
   4  10.199 BB    0.2294  125.25597    7.25652   0.2650
   5  11.367 BB    0.1847   49.42875    3.92963   0.1046
   6  11.869 BV    0.1399   10.39240    1.16696   0.0220
   7  12.224 VB    0.1741   42.75997    3.41457   0.0905

Totals :                  4.72664e4   863.60035
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\020-2001.D
Sample Name: Pro S epi AIP I DCC T=8

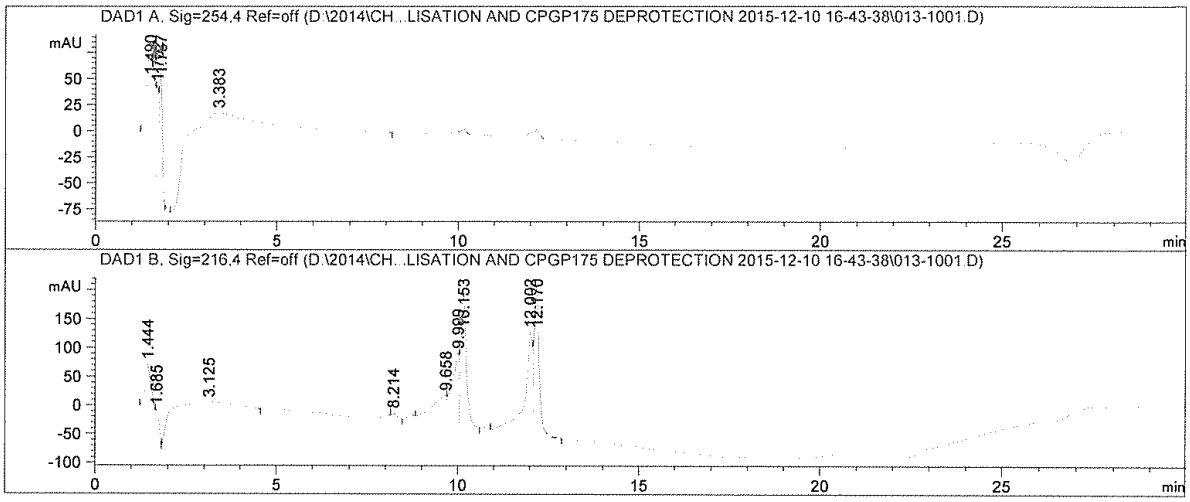
```
==========================================================================
Acq. Operator   : Simi120102015                 Seq. Line :  20
Acq. Instrument : LC1260                       Location : Vial 20
Injection Date  : 12/9/2015 5:04:02 PM              Inj :   1
                                               Inj Volume : 50.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                  100 OV 15MIN 50UL.M
Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                  \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
==========================================================================
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   1.600 BV    0.1977 4705.84521  348.08038  14.1124
   2   1.723 VB    0.1287 2841.90308  342.74042   8.5226
   3   3.352 BB    1.9856 2.50345e4   158.24918  75.0765
   4   7.252 BV    0.2906   59.79783    2.67723   0.1793
   5   7.771 VB    0.1862   25.25790    1.86263   0.0757
   6   8.191 BB    0.1210   12.33597    1.61699   0.0370
   7   9.872 BV    0.1921  122.03799    8.67838   0.3660
   8  10.080 VV    0.2573  269.69159   14.61151   0.8088
   9  10.907 VV    0.3375  161.59647    6.51323   0.4846
  10  11.370 VV    0.2279   92.23831    5.72746   0.2766
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\013-1001.D
Sample Name: Pro S epi AIP I DCC T=1

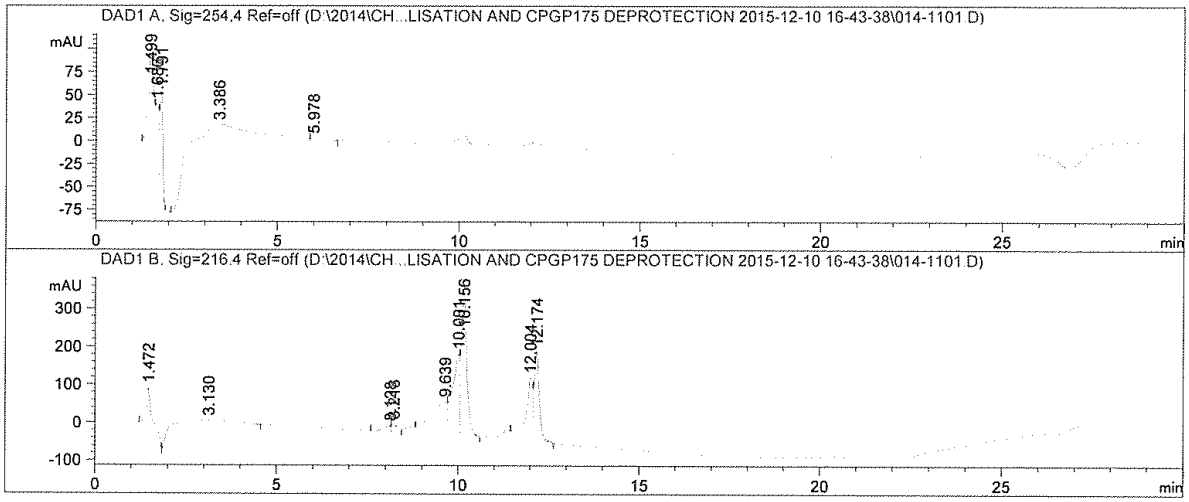
```
Acq. Operator   : Simi120102015                 Seq. Line :  10
Acq. Instrument : LC1260                        Location : Vial 13
Injection Date  : 12/10/2015 9:32:25 PM              Inj :   1
                                               Inj Volume : 100.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                  -38\10 TO 100 OV 15MIN 100UL.M
Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                  10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
Last changed    : 3/15/2016 8:58:04 AM by SYSTEM
                  (modified after loading) (Current integration events modified)
```




```
                          Area Percent Report

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   1.490 BV    0.2120 1773.21631  111.20480  10.0263
   2   1.702 VV    0.0544  349.67706   91.83134   1.9772
   3   1.787 VB    0.0826  603.62054  112.52320   3.4130
   4   3.383 BB    2.3448 1.49592e4    79.08295  84.5835

Totals :                  1.76857e4   394.64230
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\014-1101.D
Sample Name: Pro S epi AIP I DCC T=2

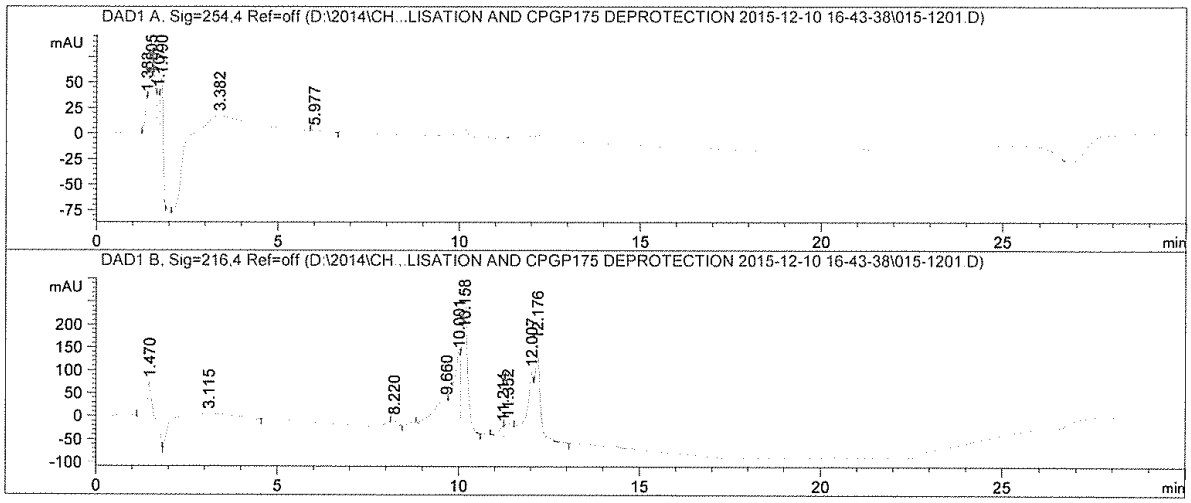
```
==========================================================================
Acq. Operator   : Simi120102015                 Seq. Line :  11
Acq. Instrument : LC1260                        Location : Vial 14
Injection Date  : 12/10/2015 10:04:14 PM             Inj :   1
                                               Inj Volume : 100.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                  -38\10 TO 100 OV 15MIN 100UL.M
Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                  10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
Last changed    : 3/15/2016 8:58:30 AM by SYSTEM
                  (modified after loading) (Current integration events modified)
==========================================================================
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   1.499 BV    0.1532 1512.02771  132.39766  11.0680
   2   1.680 VV    0.0858  499.43127   85.90598   3.6558
   3   1.791 VB    0.0851  619.86835  111.04037   4.5374
   4   3.386 BV    1.8387 1.06876e4    72.97361  78.2331
   5   5.978 VB    0.3121  342.30881   13.86132   2.5057

Totals :                  1.36613e4   416.17895
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\015-1201.D
Sample Name: Pro S epi AIP I DCC T=3

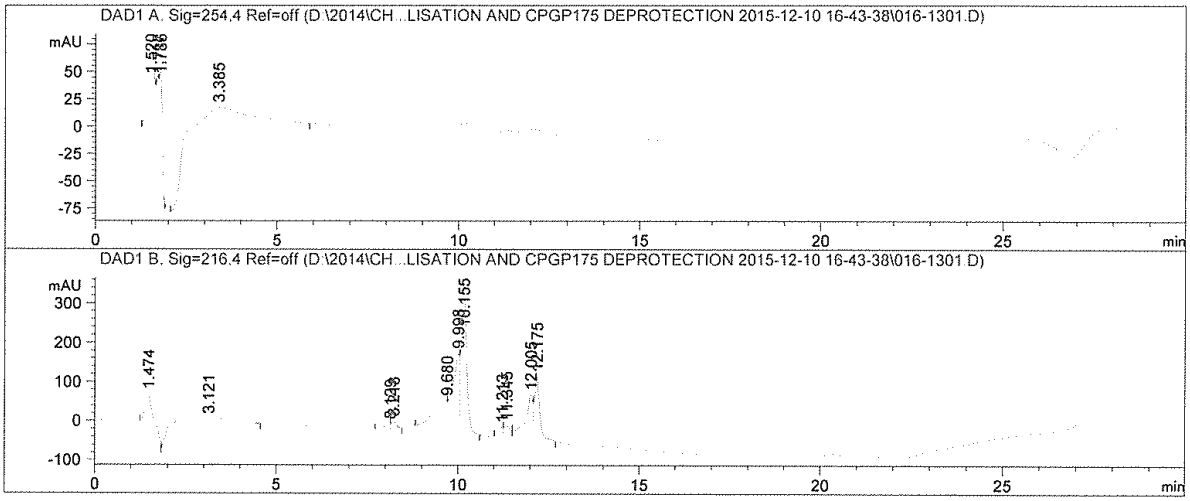
```
==========================================================================
Acq. Operator   : Simi120102015                  Seq. Line :  12
Acq. Instrument : LC1260                        Location : Vial 15
Injection Date  : 12/10/2015 10:36:04 PM             Inj :   1
                                               Inj Volume : 100.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                  -38\10 TO 100 OV 15MIN 100UL.M
Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                  10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
Last changed    : 3/15/2016 8:58:48 AM by SYSTEM
                  (modified after loading) (Current integration events modified)
==========================================================================
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   1.383 BV    0.0709  244.92012   50.25099   1.7865
   2   1.505 VV    0.1665 1396.61377  115.94518  10.1869
   3   1.707 VV    0.0675  426.61719   89.64386   3.1118
   4   1.790 VB    0.0810  602.15082  111.55718   4.3921
   5   3.382 BV    1.8693 1.07003e4    72.69448  78.0483
   6   5.977 VB    0.3114  339.23999   13.76942   2.4744

Totals :                  1.37099e4   453.86111
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\016-1301.D
Sample Name: Pro S epi AIP I DCC T=4

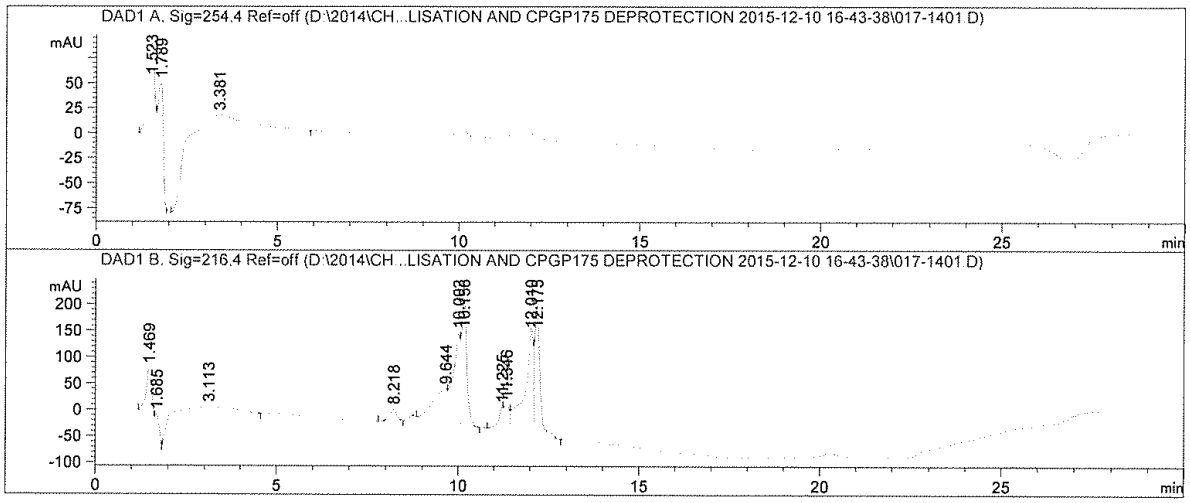
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :  13
    Acq. Instrument : LC1260                         Location : Vial 16
    Injection Date  : 12/10/2015 11:07:53 PM              Inj :   1
                                                   Inj Volume : 100.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                      -38\10 TO 100 OV 15MIN 100UL.M
    Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                      10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
    Last changed    : 3/15/2016 8:59:10 AM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.520 BV    0.2333 1617.18909  105.21475  13.8630
       2   1.717 VV    0.0656  458.88681   96.35241   3.9337
       3   1.786 VB    0.0777  563.38470  110.04817   4.8295
       4   3.385 BB    1.6803 9026.07324   67.83665  77.3739

    Totals :                  1.16655e4   379.45199
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\017-1401.D
Sample Name: Pro S epi AIP I DCC T=5

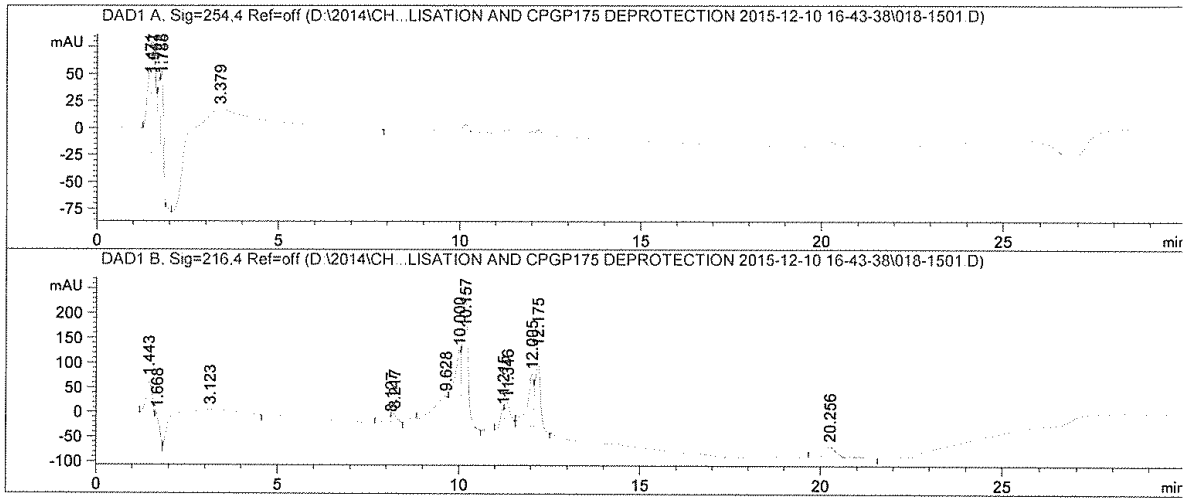
```
==========================================================================
Acq. Operator   : Simi120102015                 Seq. Line :  14
Acq. Instrument : LC1260                       Location : Vial 17
Injection Date  : 12/10/2015 11:39:43 PM            Inj :   1
                                              Inj Volume : 100.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                  -38\10 TO 100 OV 15MIN 100UL.M
Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                  10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
Last changed    : 3/15/2016 8:59:30 AM by SYSTEM
                  (modified after loading) (Current integration events modified)
==========================================================================
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   1.523 BV    0.1989 1762.90051  123.22841  14.6140
   2   1.789 VB    0.1296 1054.14795  110.16790   8.7386
   3   3.381 BB    1.6919 9246.08301   69.15031  76.6475

Totals :                  1.20631e4   302.54662
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\018-1501.D
Sample Name: Pro S epi AIP I DCC T=6

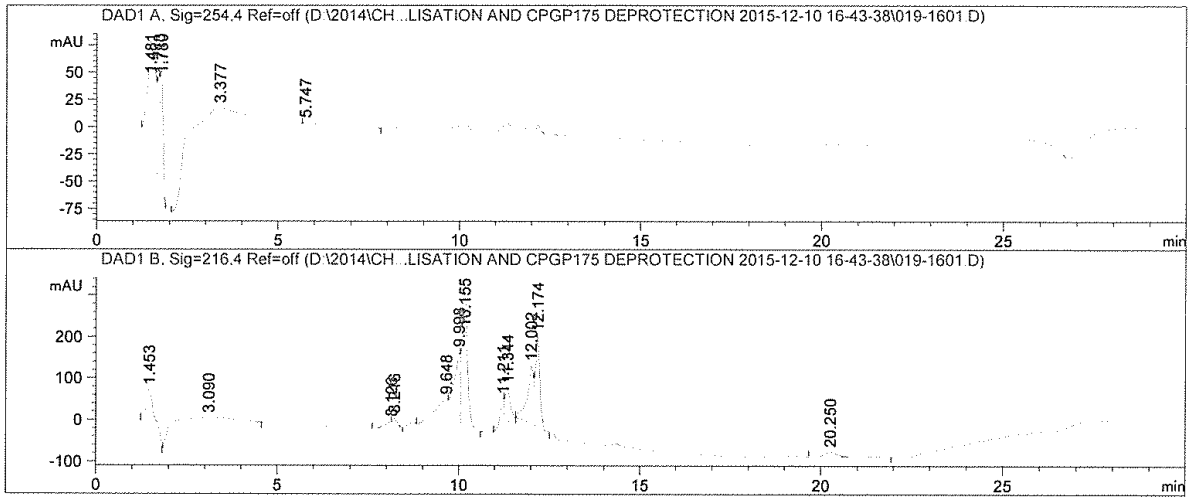
```
==========================================================================
Acq. Operator   : Simi120102015                 Seq. Line :  15
Acq. Instrument : LC1260                       Location : Vial 18
Injection Date  : 12/11/2015 12:11:32 AM            Inj :   1
                                               Inj Volume : 100.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                  -38\10 TO 100 OV 15MIN 100UL.M
Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                  10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
Last changed    : 3/15/2016 8:59:49 AM by SYSTEM
                  (modified after loading) (Current integration events modified)
==========================================================================
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   1.471 BV    0.0993  681.69727   97.83884   4.0274
   2   1.547 VV    0.1129  899.30505  110.54456   5.3130
   3   1.722 VV    0.0726  485.67072   96.73171   2.8693
   4   1.785 VB    0.0773  558.99750  109.97053   3.3025
   5   3.379 BB    2.2864 1.43009e4   78.24427  84.4879

Totals :                  1.69266e4   493.32991
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\019-1601.D
Sample Name: Pro S epi AIP I DCC T=7

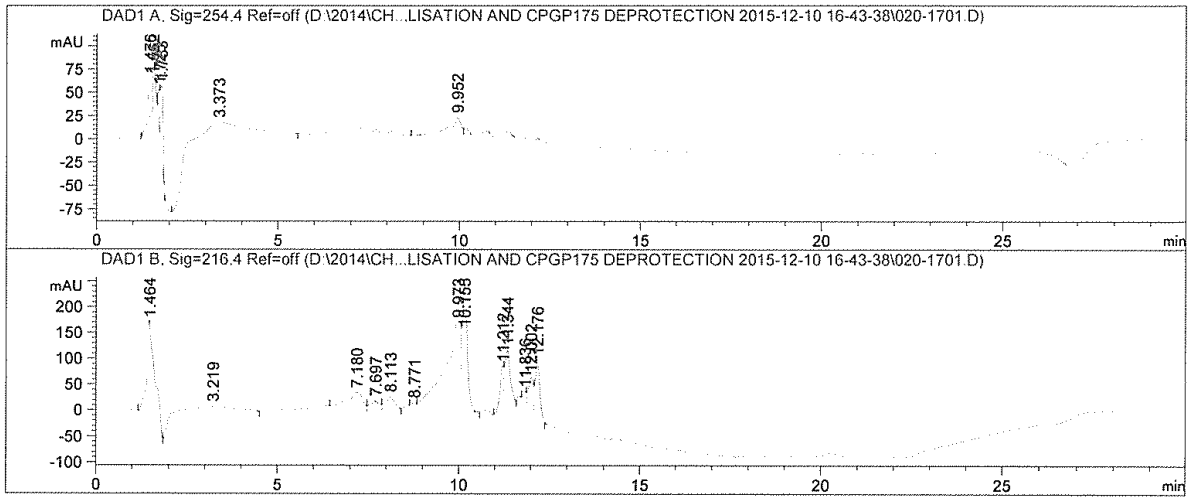
```
==========================================================================
Acq. Operator   : Simi120102015                 Seq. Line :  16
Acq. Instrument : LC1260                       Location : Vial 19
Injection Date  : 12/11/2015 12:43:22 AM            Inj :   1
                                               Inj Volume : 100.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                  -38\10 TO 100 OV 15MIN 100UL.M
Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                  10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
Last changed    : 3/15/2016 9:00:07 AM by SYSTEM
                  (modified after loading) (Current integration events modified)
==========================================================================
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   1.481 BV    0.2182 1690.13171  103.69606  10.0486
   2   1.713 VV    0.0649  446.71085   98.53292   2.6559
   3   1.780 VB    0.0781  570.95282  110.74967   3.3946
   4   3.377 BV    1.9284 1.19976e4    78.00579  71.3311
   5   5.747 VB    0.7814 2114.18994   32.47945  12.5698

Totals :                  1.68196e4   423.46389
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\020-1701.D
Sample Name: Pro S epi AIP I DCC T=8

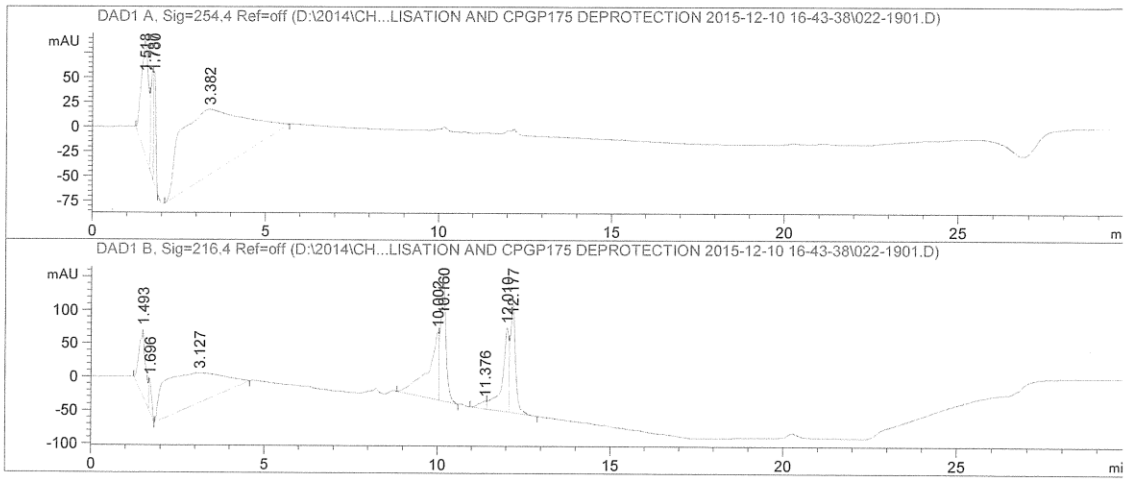
```
==========================================================================
Acq. Operator   : Simi120102015                  Seq. Line :  17
Acq. Instrument : LC1260                        Location : Vial 20
Injection Date  : 12/11/2015 1:15:13 AM              Inj :   1
                                               Inj Volume : 100.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                  -38\10 TO 100 OV 15MIN 100UL.M
Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                  10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
Last changed    : 3/15/2016 9:00:24 AM by SYSTEM
                  (modified after loading) (Current integration events modified)
==========================================================================
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   1.476 BV    0.0982  542.02014   78.81866   5.5760
   2   1.562 VB    0.0616  213.40388   52.32769   2.1954
   3   1.725 BV    0.0496  125.30008   38.83519   1.2890
   4   1.783 VB    0.0742  346.05554   71.82365   3.5600
   5   3.373 BB    1.5965 8039.54834   64.57506  82.7063
   6   9.952 BV    0.3204  454.26746   18.37459   4.6732

Totals :                  9720.59544  324.75485
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\022-1901.D
Sample Name: Pro S epi AIP I PS CD T=1

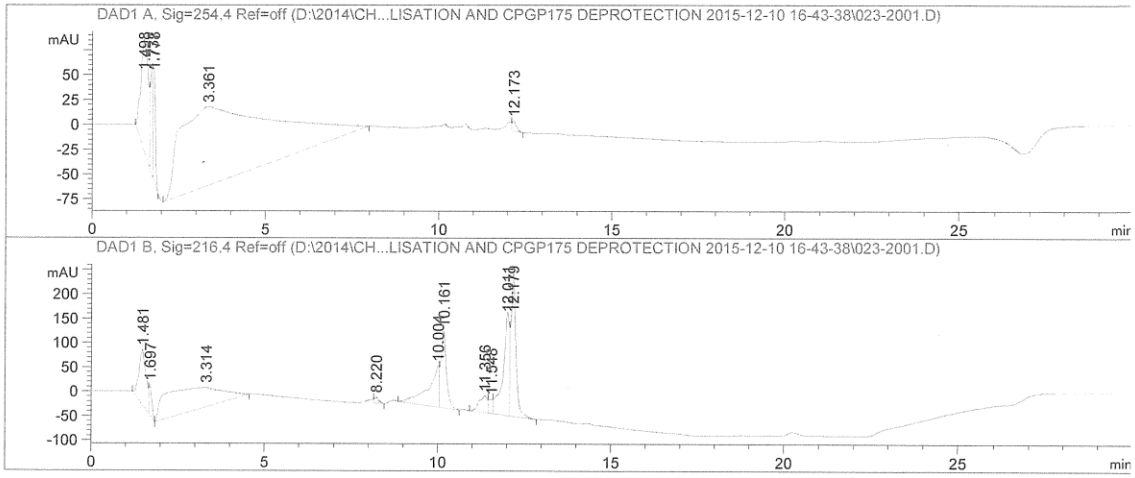
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :  19
    Acq. Instrument : LC1260                         Location : Vial 22
    Injection Date  : 12/11/2015 2:18:53 AM               Inj :   1
                                                   Inj Volume : 100.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                      -38\10 TO 100 OV 15MIN 100UL.M
    Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                      10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
    Last changed    : 3/15/2016 9:01:02 AM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.518 BV    0.2002 1660.21387  115.15921  14.8509
       2   1.737 VV    0.0725  563.58258  108.64600   5.0414
       3   1.780 VB    0.0681  481.81845  112.27672   4.3100
       4   3.382 BB    1.6321 8473.57813   66.16603  75.7978

    Totals :                   1.11792e4   402.24796
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\023-2001.D
Sample Name: Pro S epi AIP I PS CD T=2

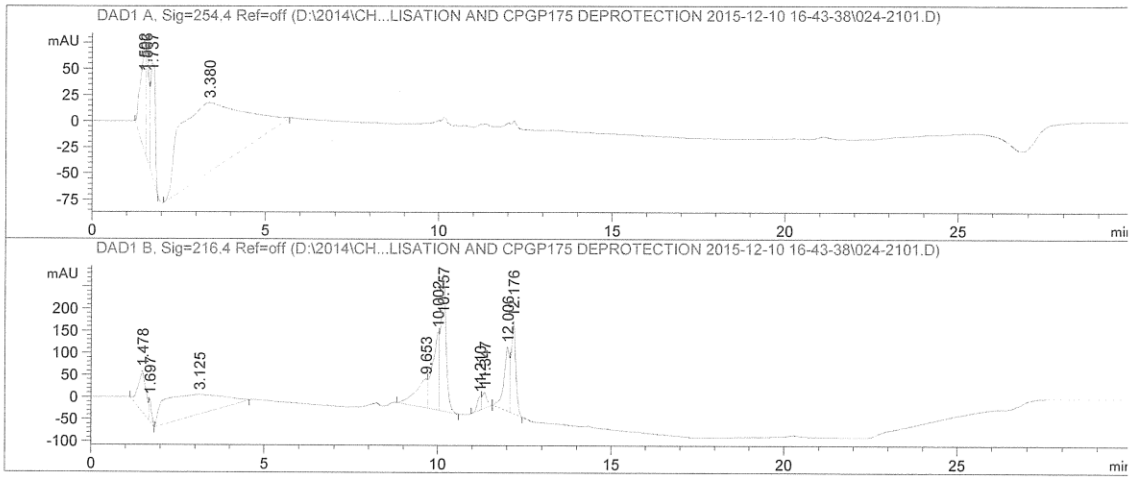
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :  20
    Acq. Instrument : LC1260                         Location : Vial 23
    Injection Date  : 12/11/2015 2:50:43 AM              Inj :   1
                                                   Inj Volume : 100.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                      -38\10 TO 100 OV 15MIN 100UL.M
    Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                      10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
    Last changed    : 3/15/2016 9:01:22 AM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.498 BV    0.1840 1647.77673  111.78018   9.5032
       2   1.737 VV    0.0730  566.62054  108.45362   3.2679
       3   1.778 VB    0.0722  527.20453  113.49354   3.0405
       4   3.361 BB    2.2568 1.45051e4    79.12041  83.6548
       5  12.173 VB    0.1251   92.52731   10.68017   0.5336

    Totals :                  1.73392e4   423.52793
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\024-2101.D
Sample Name: Pro S epi AIP I PS CD T=3

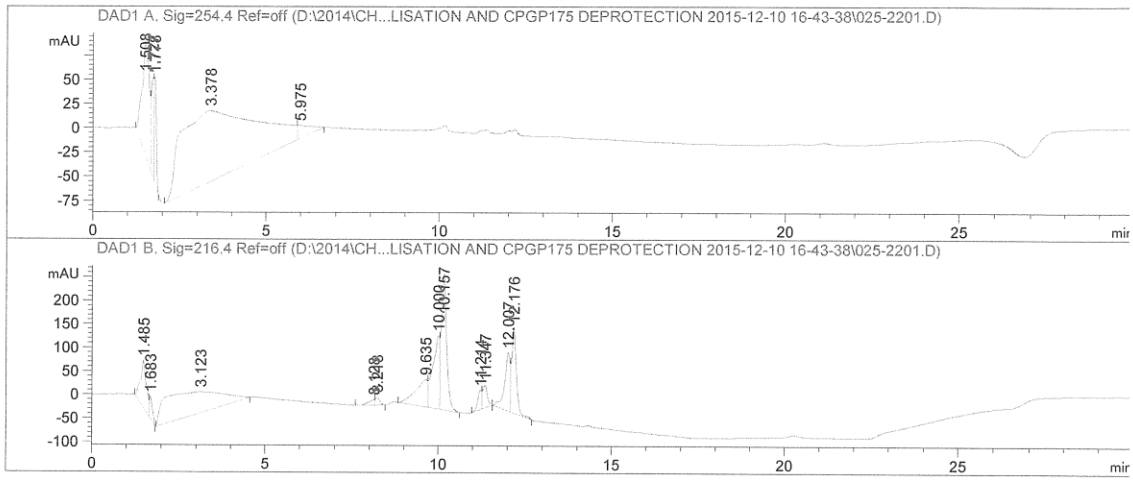
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :  21
    Acq. Instrument : LC1260                         Location : Vial 24
    Injection Date  : 12/11/2015 3:22:32 AM               Inj :   1
                                                   Inj Volume : 100.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                      -38\10 TO 100 OV 15MIN 100UL.M
    Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                      10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
    Last changed    : 3/15/2016 9:01:42 AM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.502 BV    0.1311 1050.07764  104.61230   9.3340
       2   1.566 VV    0.0897  635.97784  100.64491   5.6531
       3   1.737 VB    0.1224 1004.64984  108.08659   8.9302
       4   3.380 BB    1.6486 8559.33203   66.45619  76.0827

    Totals :                  1.12500e4   379.79998
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\025-2201.D
Sample Name: Pro S epi AIP I PS CD T=4

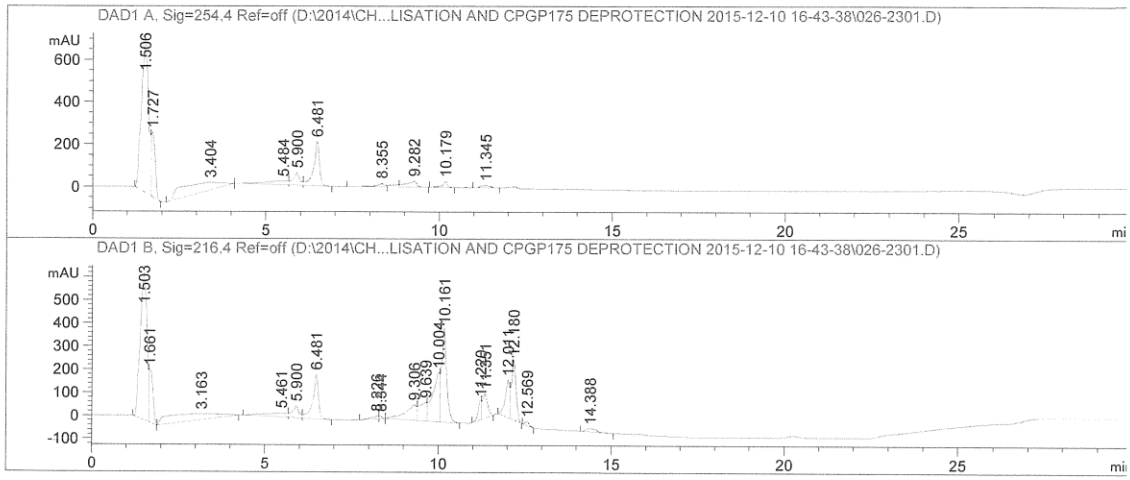
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :  22
    Acq. Instrument : LC1260                         Location : Vial 25
    Injection Date  : 12/11/2015 3:54:22 AM               Inj :   1
                                                  Inj Volume : 100.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                      -38\10 TO 100 OV 15MIN 100UL.M
    Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                      10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
    Last changed    : 3/15/2016 9:02:00 AM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.508 BV    0.1835 1661.88477  118.54943  12.0587
       2   1.727 VV    0.0705  520.62689  103.85775   3.7777
       3   1.778 VB    0.0699  504.38531  109.18156   3.6598
       4   3.378 BV    1.8522 1.07563e4    73.11949  78.0479
       5   5.975 VB    0.3093  338.46811   13.74439   2.4559

    Totals :                  1.37816e4   418.45262
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\026-2301.D
Sample Name: Pro S epi AIP I PS CD T=5

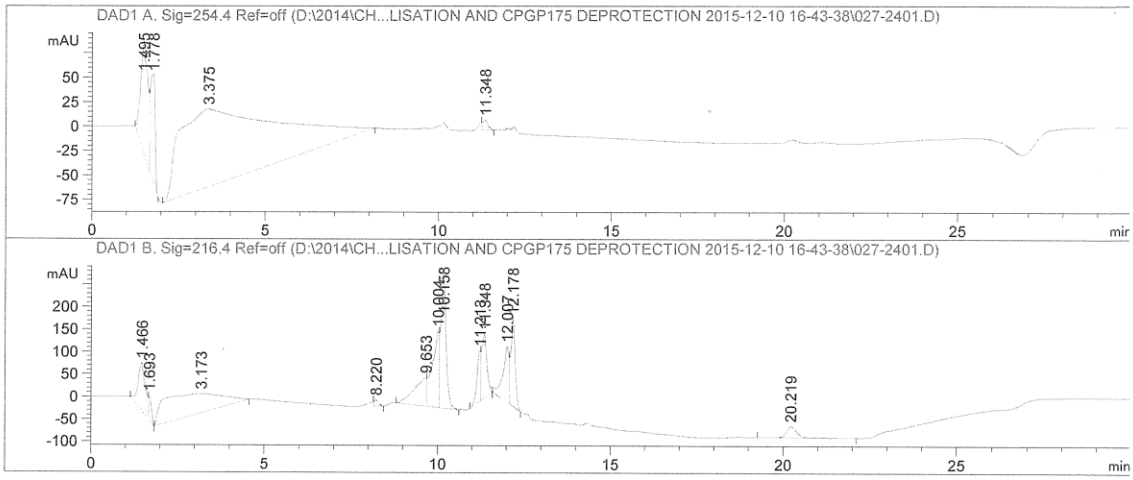
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :  23
    Acq. Instrument : LC1260                         Location : Vial 26
    Injection Date  : 12/11/2015 4:26:12 AM               Inj :   1
                                                   Inj Volume : 100.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                      -38\10 TO 100 OV 15MIN 100UL.M
    Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                      10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
    Last changed    : 3/15/2016 9:02:17 AM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.506 BV    0.1808 1.02451e4   716.78851  47.5757
       2   1.727 VB    0.1219 2299.40771  305.07468  10.6779
       3   3.404 BB    1.3687 3721.28955   35.33350  17.2807
       4   5.484 BV    0.5729  796.08685   17.83087   3.6968
       5   5.900 VV    0.1798  776.93054   58.92851   3.6079
       6   6.481 VB    0.1603 2397.76270  208.35289  11.1346
       7   8.355 BV    0.2192  260.92456   15.43838   1.2117
       8   9.282 VB    0.2590  549.01959   27.49500   2.5495
       9  10.179 BB    0.1373  273.00604   27.62776   1.2678
      10  11.345 BB    0.2537  214.80388   11.10468   0.9975
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\027-2401.D
Sample Name: Pro S epi AIP I PS CD T=6

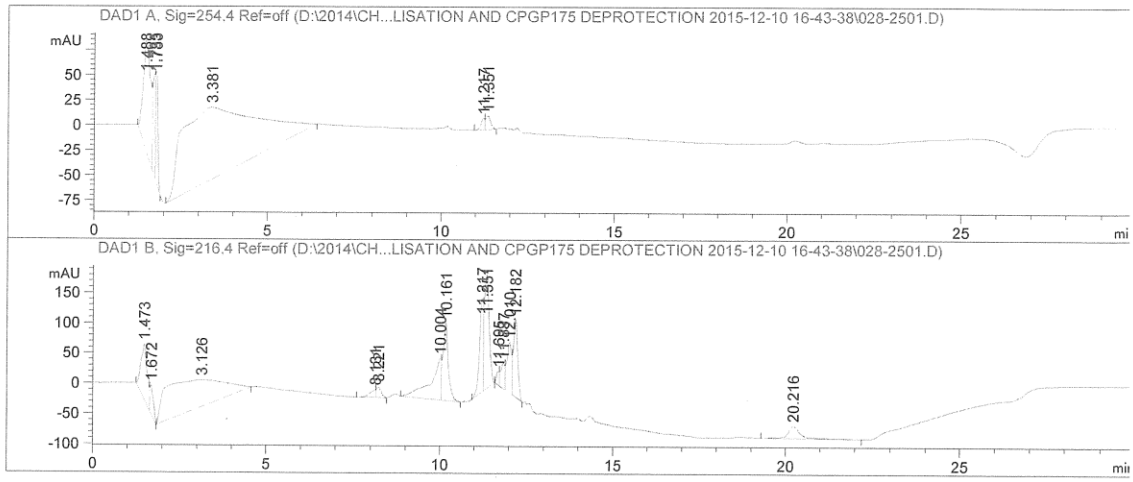
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :  24
    Acq. Instrument : LC1260                         Location : Vial 27
    Injection Date  : 12/11/2015 4:58:02 AM               Inj :   1
                                                  Inj Volume : 100.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                      -38\10 TO 100 OV 15MIN 100UL.M
    Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                      10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
    Last changed    : 3/15/2016 9:02:33 AM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.495 BV    0.1857 1662.95837  114.31179   9.3171
       2   1.778 VB    0.1266 1034.69458  111.17526   5.7971
       3   3.375 BB    2.3414 1.50364e4    79.54213  84.2445
       4  11.348 VB    0.1491  114.46119   10.84628   0.6413

    Totals :                  1.78485e4   315.87546
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\028-2501.D
Sample Name: Pro S epi AIP I PS CD T=7

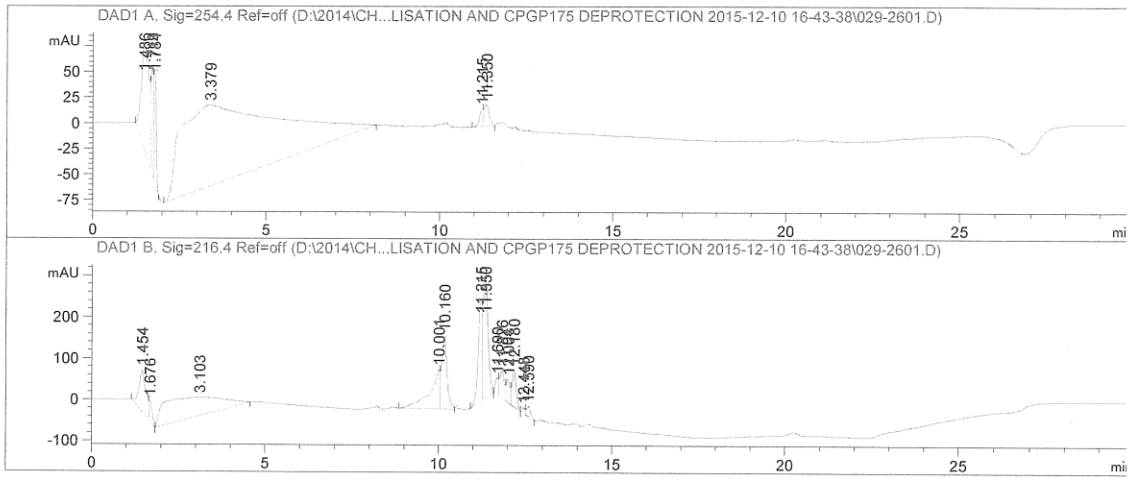
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :  25
    Acq. Instrument : LC1260                        Location : Vial 28
    Injection Date  : 12/11/2015 5:29:51 AM              Inj :   1
                                                  Inj Volume : 100.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                      -38\10 TO 100 OV 15MIN 100UL.M
    Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                      10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
    Last changed    : 3/15/2016 9:02:52 AM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.488 BV    0.2075 1636.97119  108.82732  12.2968
       2   1.735 VV    0.0668  479.67804  102.20838   3.6033
       3   1.783 VB    0.0751  540.25592  110.29816   4.0584
       4   3.381 BB    1.8424 1.04387e4    71.62056  78.4150
       5  11.217 BV    0.1113   83.07583   11.10474   0.6241
       6  11.351 VB    0.1407  133.44598   13.57142   1.0024

    Totals :                  1.33122e4   417.63059
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\029-2601.D
Sample Name: Pro S epi AIP I PS CD T=8

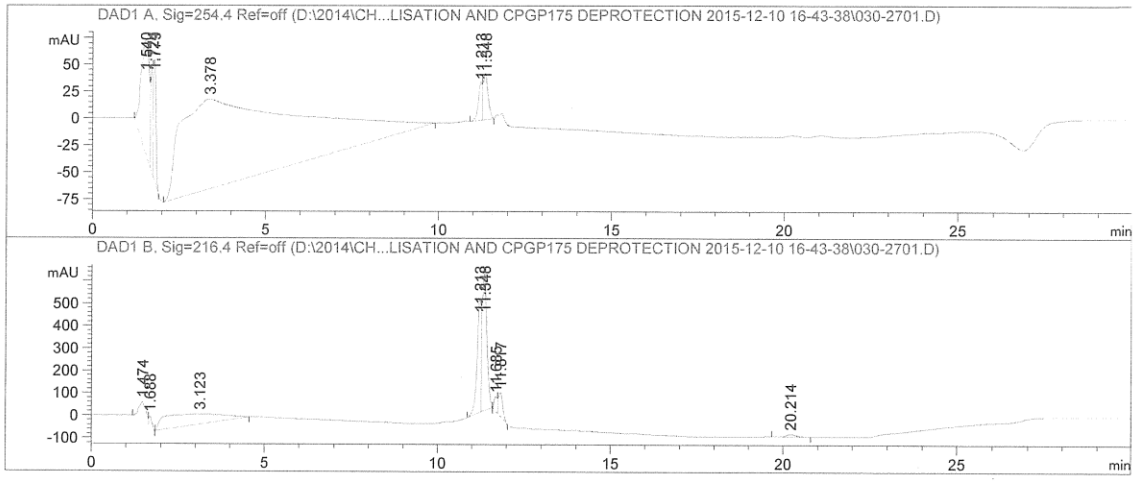
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :  26
    Acq. Instrument : LC1260                         Location : Vial 29
    Injection Date  : 12/11/2015 6:01:45 AM               Inj :   1
                                                   Inj Volume : 100.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                      -38\10 TO 100 OV 15MIN 100UL.M
    Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                      10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
    Last changed    : 3/15/2016 9:03:09 AM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.486 BV    0.2108 1617.18762  106.70644   8.9874
       2   1.719 VV    0.0746  552.19513  102.85656   3.0688
       3   1.784 VB    0.0738  525.49359  109.88778   2.9204
       4   3.379 BB    2.3545 1.49545e4    78.99718  83.1089
       5  11.215 BV    0.1124  133.09993   17.57053   0.7397
       6  11.350 VB    0.1385  211.38432   21.54458   1.1748

    Totals :                  1.79939e4   437.56308
```

Data File D:\2014\CH...CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43-38\030-2701.D
Sample Name: Pro S epi AIP I PS CD 2 days

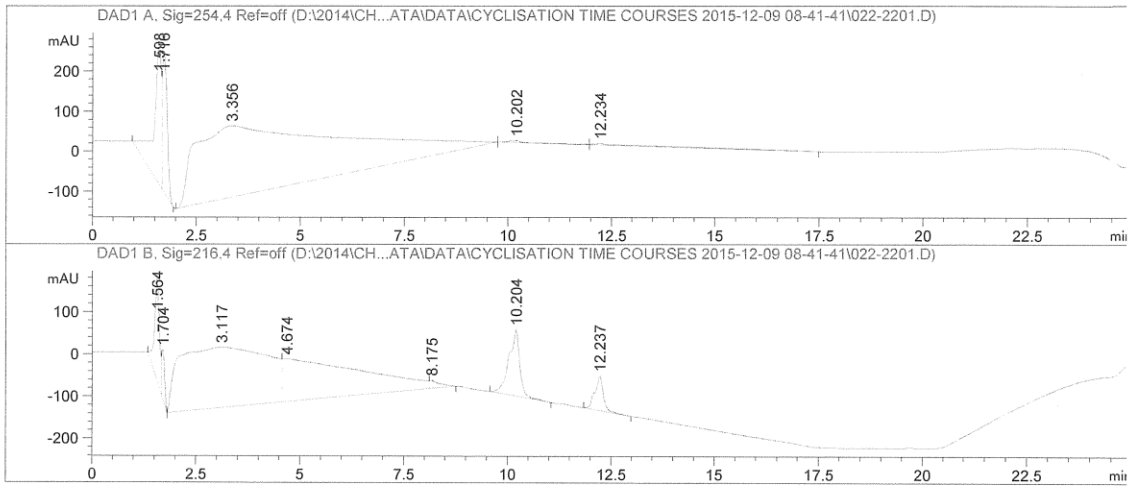
```
    ======================================================================
    Acq. Operator   : Simi120102015                  Seq. Line :  27
    Acq. Instrument : LC1260                          Location : Vial 30
    Injection Date  : 12/11/2015 6:33:35 AM               Inj :   1
                                                    Inj Volume : 100.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-10 16-43
                      -38\10 TO 100 OV 15MIN 100UL.M
    Last changed    : 12/10/2015 4:12:03 PM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION AND CPGP175 DEPROTECTION 2015-12-
                      10 16-43-38\10 TO 100 OV 15MIN 100UL.M (Sequence Method)
    Last changed    : 3/15/2016 9:03:26 AM by SYSTEM
                      (modified after loading) (Current integration events modified)
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.540 BV    0.2209 1739.51306  106.29937   7.6243
       2   1.724 VV    0.0623  440.05896  102.06106   1.9288
       3   1.779 VB    0.0763  559.52161  111.87844   2.4524
       4   3.378 BB    2.8608 1.93695e4    83.17658  84.8968
       5  11.213 BV    0.1146  276.97739   35.68401   1.2140
       6  11.348 VB    0.1390  429.76346   43.58271   1.8837

    Totals :                  2.28153e4   482.68216
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\022-2201.D
Sample Name: Pro S epi AIP I PS CD T=1

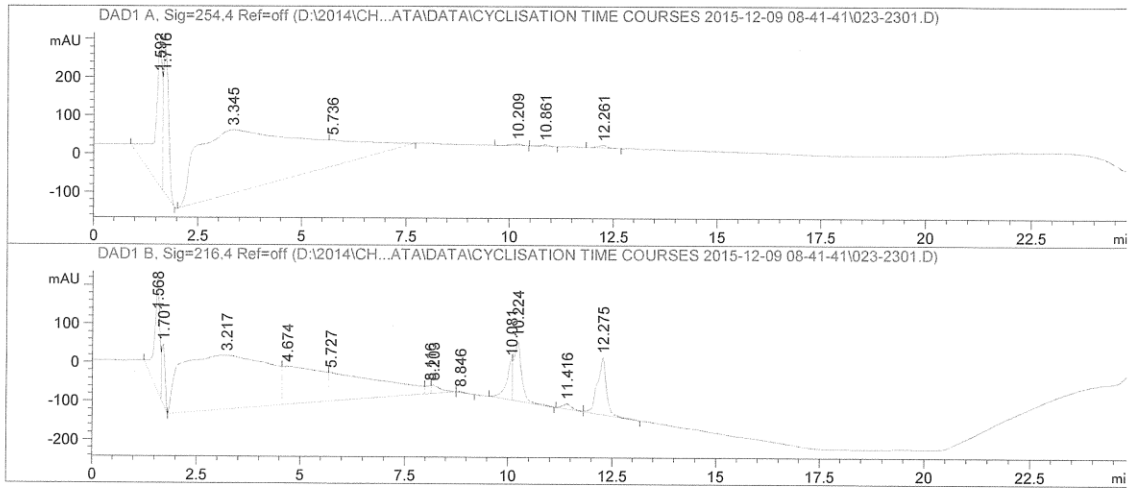
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :  22
    Acq. Instrument : LC1260                        Location : Vial 22
    Injection Date  : 12/9/2015 5:56:48 PM               Inj :   1
                                                  Inj Volume : 50.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                      100 OV 15MIN 50UL.M
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                      \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.598 BV    0.1721 4475.40674  357.39114   9.0291
       2   1.716 VB    0.1296 2861.49512  349.05670   5.7731
       3   3.356 BB    2.8882 4.14808e4   177.38484  83.6873
       4  10.202 BV    0.4864  177.58722    4.52004   0.3583
       5  12.234 VB    1.5600  571.15106    4.34961   1.1523

    Totals :                  4.95664e4   892.70234
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\023-2301.D
Sample Name: Pro S epi AIP I PS CD T=2

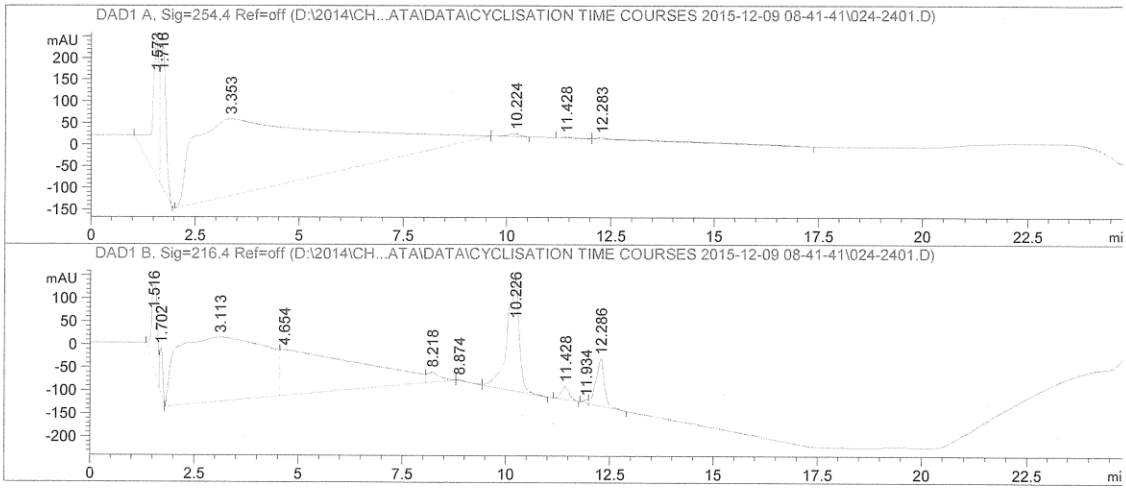
```
======================================================================
Acq. Operator   : Simi120102015                 Seq. Line :  23
Acq. Instrument : LC1260                        Location : Vial 23
Injection Date  : 12/9/2015 6:23:10 PM               Inj :   1
                                               Inj Volume : 50.000 µl
Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                  100 OV 15MIN 50UL.M
Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                  \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
======================================================================
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   1.592 BV    0.1710 4785.58105  379.84222  12.4461
   2   1.716 VB    0.1278 3116.48901  379.48575   8.1052
   3   3.345 BV    1.9716 2.60660e4   166.57294  67.7914
   4   5.736 VB    0.7355 4221.63525   68.64129  10.9795
   5  10.209 BB    0.2575   86.80096    4.65526   0.2257
   6  10.861 BB    0.2216   62.66041    3.98249   0.1630
   7  12.261 BB    0.2353  111.13319    6.70899   0.2890

Totals :                 3.84503e4  1009.88894
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\024-2401.D
Sample Name: Pro S epi AIP I PS CD T=3

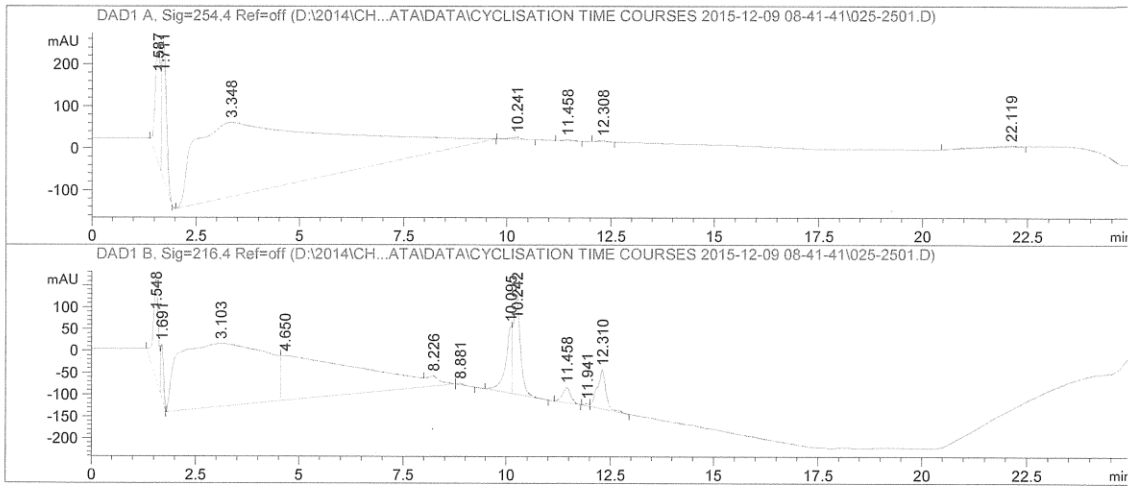
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :  24
    Acq. Instrument : LC1260                        Location : Vial 24
    Injection Date  : 12/9/2015 6:49:33 PM               Inj :   1
                                                   Inj Volume : 50.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                      100 OV 15MIN 50UL.M
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                      \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.573 BV    0.1631 3866.66553  315.27979   7.9711
       2   1.716 VB    0.1345 2936.91284  328.01505   6.0544
       3   3.353 BB    2.8744 4.10430e4   176.79466  84.6097
       4  10.224 BB    0.2467  117.43279    6.26312   0.2421
       5  11.428 BV    0.3077   56.32907    2.43674   0.1161
       6  12.283 VB    1.3310  488.26379    4.40569   1.0066

    Totals :                  4.85086e4   833.19503
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\025-2501.D
Sample Name: Pro S epi AIP I PS CD T=4

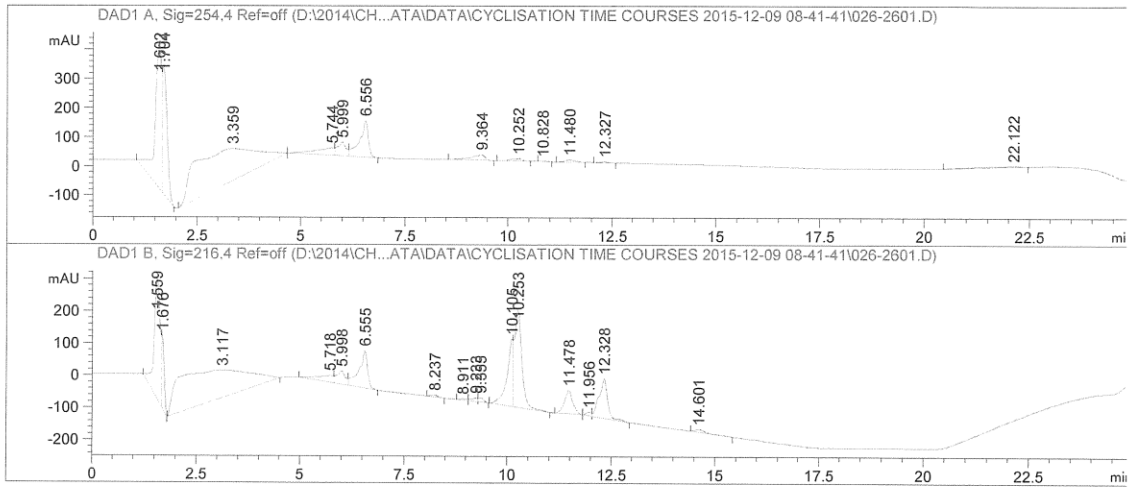
```
    ======================================================================
    Acq. Operator   : Simi120102015                  Seq. Line :  25
    Acq. Instrument : LC1260                          Location : Vial 25
    Injection Date  : 12/9/2015 7:15:55 PM                 Inj :   1
                                                    Inj Volume : 50.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                      100 OV 15MIN 50UL.M
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                      \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.587 BV    0.1370 2517.76270  290.86017   5.3746
       2   1.711 VB    0.1231 2619.98633  328.32071   5.5928
       3   3.348 BB    2.8989 4.13049e4   176.33109  88.1717
       4  10.241 BB    0.2320  101.90182    5.88484   0.2175
       5  11.458 BB    0.2007   37.15019    2.69713   0.0793
       6  12.308 BB    0.1748   37.44059    2.97651   0.0799
       7  22.119 BB    1.1297  226.86057    2.48345   0.4843

    Totals :                  4.68460e4   809.55389
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\026-2601.D
Sample Name: Pro S epi AIP I PS CD T=5

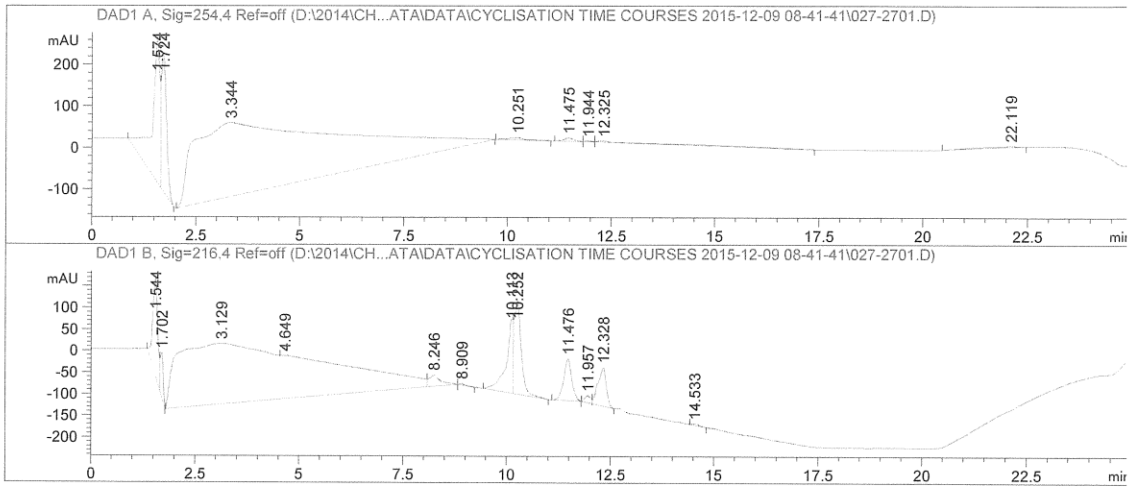
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :  26
    Acq. Instrument : LC1260                        Location : Vial 26
    Injection Date  : 12/9/2015 7:42:17 PM               Inj :   1
                                                  Inj Volume : 50.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                      100 OV 15MIN 50UL.M
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                      \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.602 BV    0.1680 5580.91895  508.85904  21.9606
       2   1.704 VB    0.1156 3330.00220  443.56009  13.1034
       3   3.359 BB    1.4560 1.22159e4   109.29186  48.0690
       4   5.744 BV    0.3984  766.57751   25.52615   3.0164
       5   5.999 VV    0.1941  690.55743   47.92941   2.7173
       6   6.556 VB    0.1979 1854.15955  127.28959   7.2960
       7   9.364 BB    0.2931  421.56686   18.69113   1.6588
       8  10.252 BB    0.2312  139.49071    8.08670   0.5489
       9  10.828 BB    0.1420    9.70850    1.02928   0.0382
      10  11.480 BB    0.2287  122.68991    7.35035   0.4828
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\027-2701.D
Sample Name: Pro S epi AIP I PS CD T=6

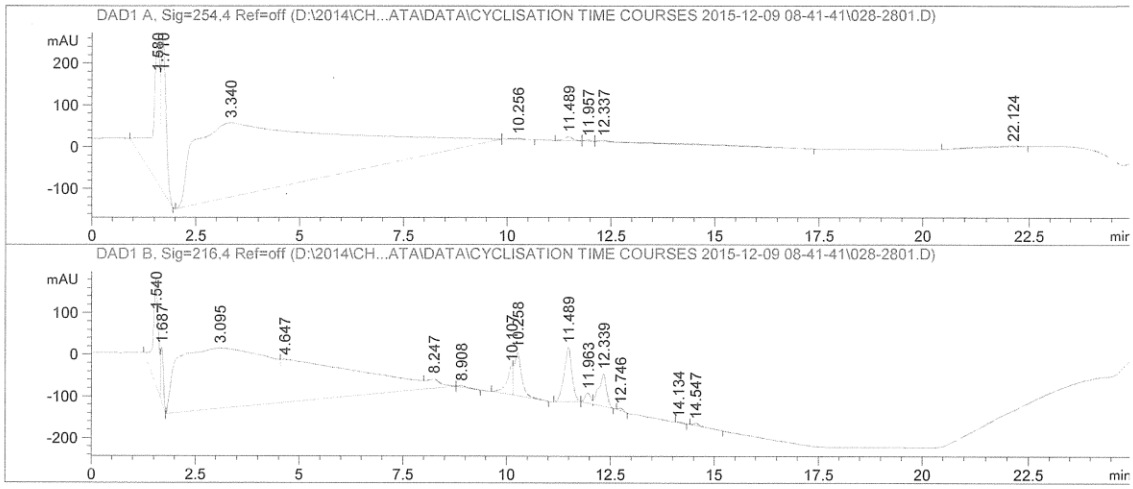
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :  27
    Acq. Instrument : LC1260                         Location : Vial 27
    Injection Date  : 12/9/2015 8:08:39 PM               Inj :   1
                                                   Inj Volume : 50.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                      100 OV 15MIN 50UL.M
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                      \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.574 BV    0.1851 4707.99365  340.84787   9.4353
       2   1.724 VB    0.1276 2849.03564  333.68732   5.7097
       3   3.344 BB    2.8834 4.13704e4   177.22363  82.9101
       4  10.251 BB    0.2719  118.68468    5.67604   0.2379
       5  11.475 BV    0.2220  123.13974    7.89394   0.2468
       6  11.944 VV    0.1930   29.98242    2.22746   0.0601
       7  12.325 VB    1.3417  460.10406    4.08597   0.9221
       8  22.119 BB    1.1310  238.57440    2.63836   0.4781

    Totals :                  4.98979e4   874.28059
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\028-2801.D
Sample Name: Pro S epi AIP I PS CD T=7

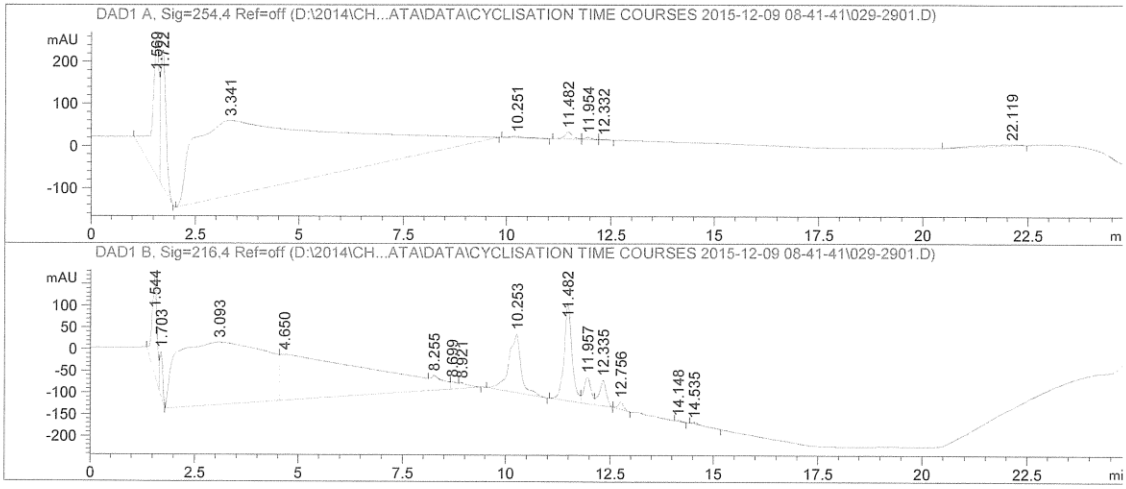
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :  28
    Acq. Instrument : LC1260                         Location : Vial 28
    Injection Date  : 12/9/2015 8:35:02 PM               Inj :   1
                                                   Inj Volume : 50.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                      100 OV 15MIN 50UL.M
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                      \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.580 BV    0.1941 4443.66699  332.10226   8.8077
       2   1.710 VB    0.1245 2983.06885  360.52243   5.9127
       3   3.340 BB    2.9327 4.20051e4   177.00153  83.2577
       4  10.256 BB    0.2146   41.02726    2.56509   0.0813
       5  11.489 BV    0.2192  159.67226   10.51505   0.3165
       6  11.957 VV    0.1934   46.38908    3.43854   0.0919
       7  12.337 VB    1.5655  535.06836    4.06021   1.0606
       8  22.124 BB    1.1321  237.91399    2.64833   0.4716

    Totals :                  5.04519e4   892.85345
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\029-2901.D
Sample Name: Pro S epi AIP I PS CD T=8

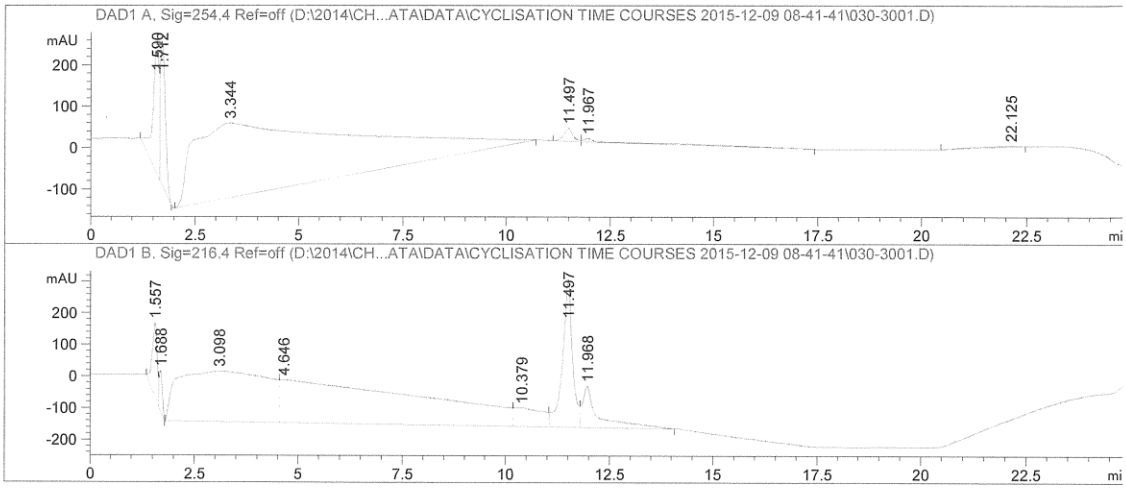
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :  29
    Acq. Instrument : LC1260                         Location : Vial 29
    Injection Date  : 12/9/2015 9:01:23 PM               Inj :   1
                                                   Inj Volume : 50.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                      100 OV 15MIN 50UL.M
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                      \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.569 BV    0.1660 3945.06543  324.15161   8.0010
       2   1.722 VB    0.1244 2753.34375  326.33218   5.5841
       3   3.341 BB    2.9324 4.19959e4   177.36577  85.1726
       4  10.251 BB    0.2612   67.30489    3.36711   0.1365
       5  11.482 BV    0.1974  223.53157   16.35609   0.4533
       6  11.954 VV    0.1812   62.37595    5.01051   0.1265
       7  12.332 VB    0.1537   21.76268    2.08670   0.0441
       8  22.119 BB    1.1242  237.54454    2.64390   0.4818

    Totals :                  4.93069e4   857.31387
```

Data File D:\2014\CH...C DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41\030-3001.D
Sample Name: Pro S epi AIP I PS CD 2 days

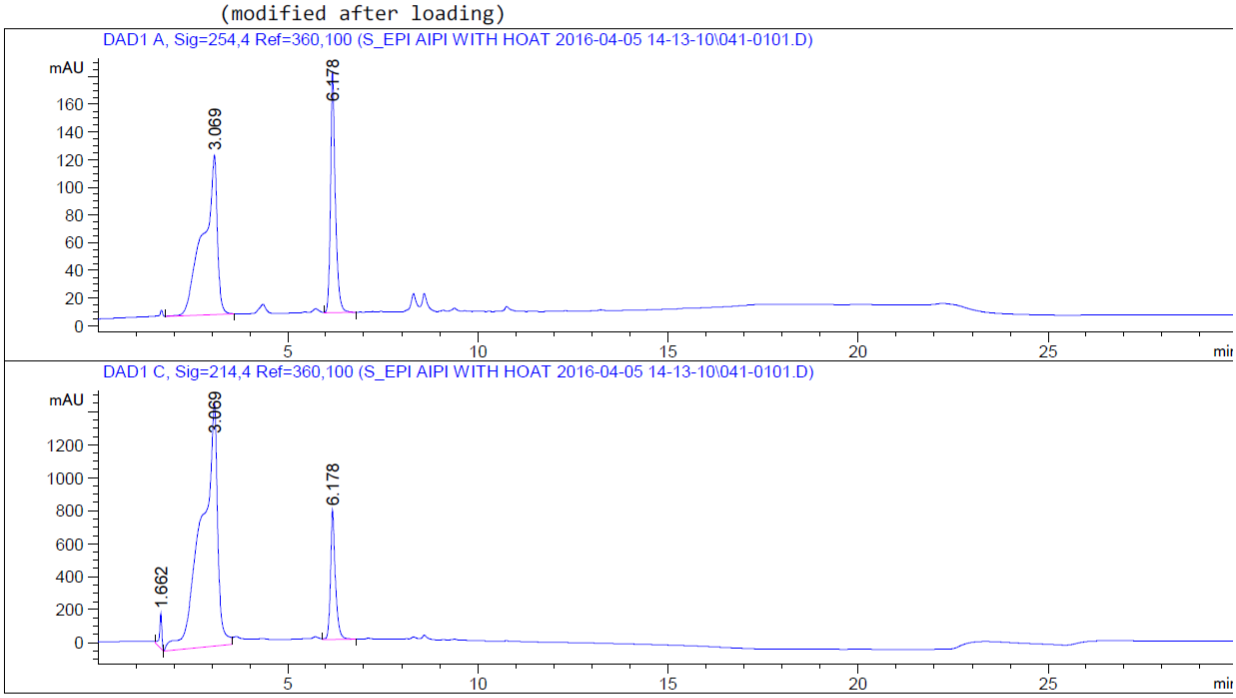
```
    ======================================================================
    Acq. Operator   : Simi120102015                 Seq. Line :  30
    Acq. Instrument : LC1260                         Location : Vial 30
    Injection Date  : 12/9/2015 9:27:46 PM               Inj :   1
                                                   Inj Volume : 50.000 µl
    Acq. Method     : C:\CHEM32\1\DATA\DEMO\CYCLISATION TIME COURSES 2015-12-09 08-41-41\10 TO
                      100 OV 15MIN 50UL.M
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    Analysis Method : D:\2014\CHRIS HPLC DATA\DATA\CYCLISATION TIME COURSES 2015-12-09 08-41-41
                      \10 TO 100 OV 15MIN 50UL.M (Sequence Method)
    Last changed    : 12/9/2015 8:34:52 AM by Simi120102015
    ======================================================================
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   1.590 BV    0.1557 3266.97339  323.89368   6.0611
       2   1.712 VB    0.1223 2821.94189  348.96078   5.2354
       3   3.344 BB    3.1929 4.65812e4   179.53839  86.4203
       4  11.497 BV    0.2117  458.28003   31.50893   0.8502
       5  11.967 VB    0.7869  533.45526    8.33246   0.9897
       6  22.125 BB    1.1335  238.91240    2.66091   0.4432

    Totals :                  5.39007e4   894.89515
```

Data File C:\CHEM32\1\DATA\S_EPI AIPI WITH HOAT 2016-04-05 14-13-10\041-0101.D
Sample Name: S epi AIP I cyclised with HOAt t = 0

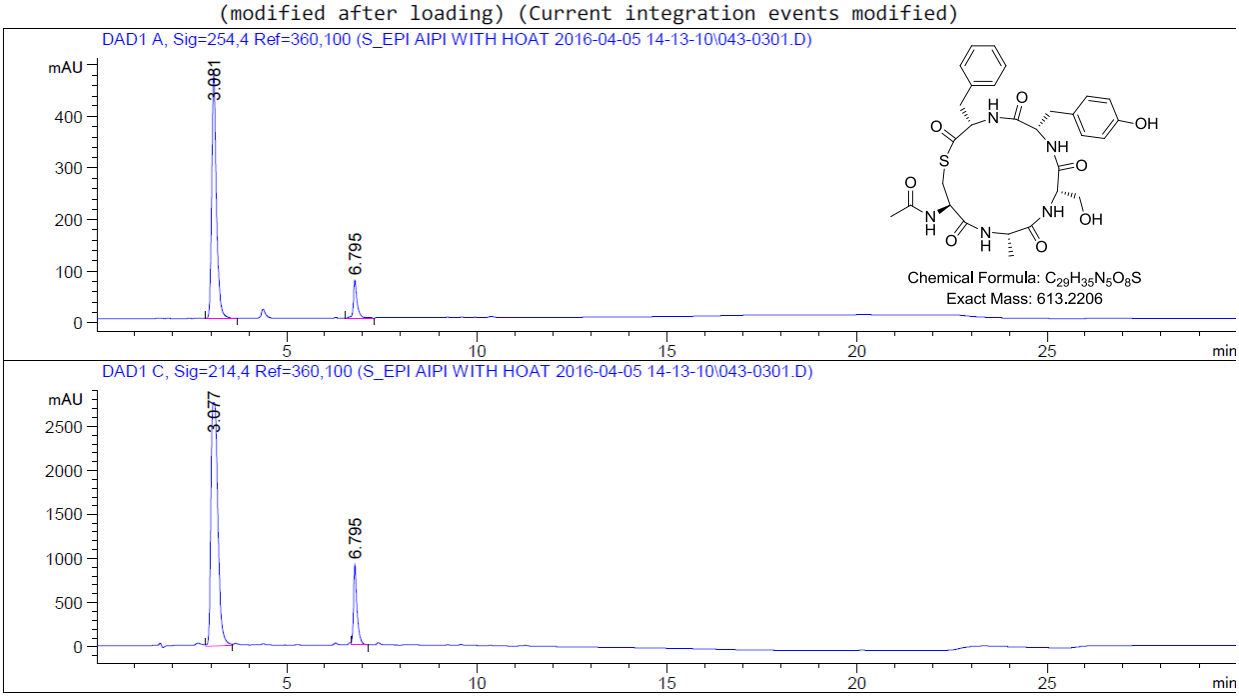
```
==================================================================
Acq. Operator    : SYSTEM                        Seq. Line :   1
Acq. Instrument : LC1260                          Location : Vial 41
Injection Date   : 4/5/2016 2:14:35 PM                 Inj :   1
                                                 Inj Volume : 20.000 µl
Acq. Method      : C:\CHEM32\1\DATA\S_EPI AIPI WITH HOAT 2016-04-05 14-13-10\10 TO 100 OVER 15
                   MINS 20UL.M
Last changed     : 4/5/2016 2:13:10 PM by SYSTEM
Analysis Method : C:\CHEM32\1\DATA\S_EPI AIPI WITH HOAT 2016-04-05 14-13-10\10 TO 100 OVER 15
                   MINS 20UL.M (Sequence Method)
Last changed     : 4/5/2016 4:09:36 PM by SYSTEM
                   (modified after loading)
```



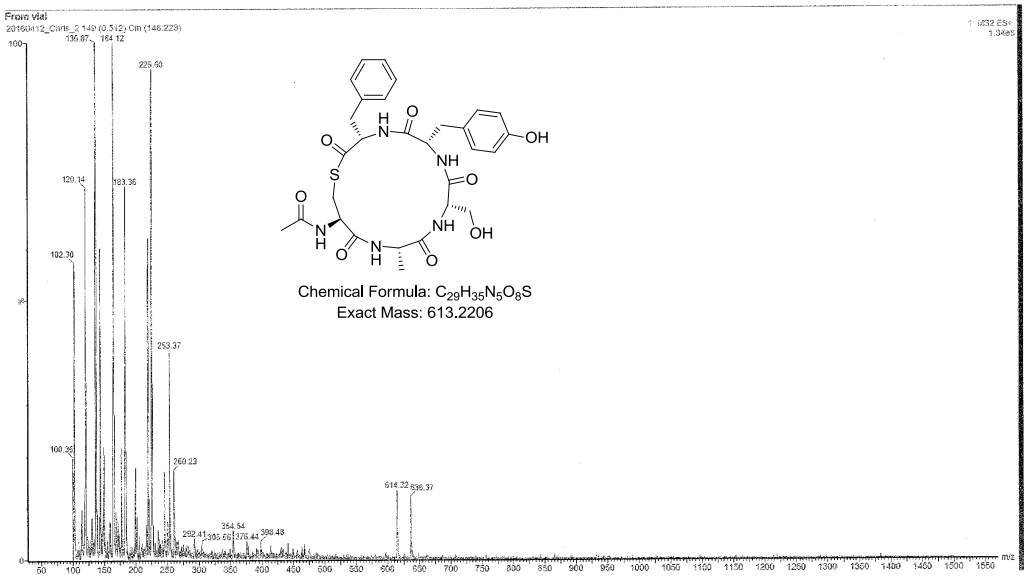

```
==========================================================================================
                          Fraction Information
==========================================================================================
==========================================================================================
No Fractions found.
==========================================================================================
==================================================================
                       Area Percent Report
==================================================================

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

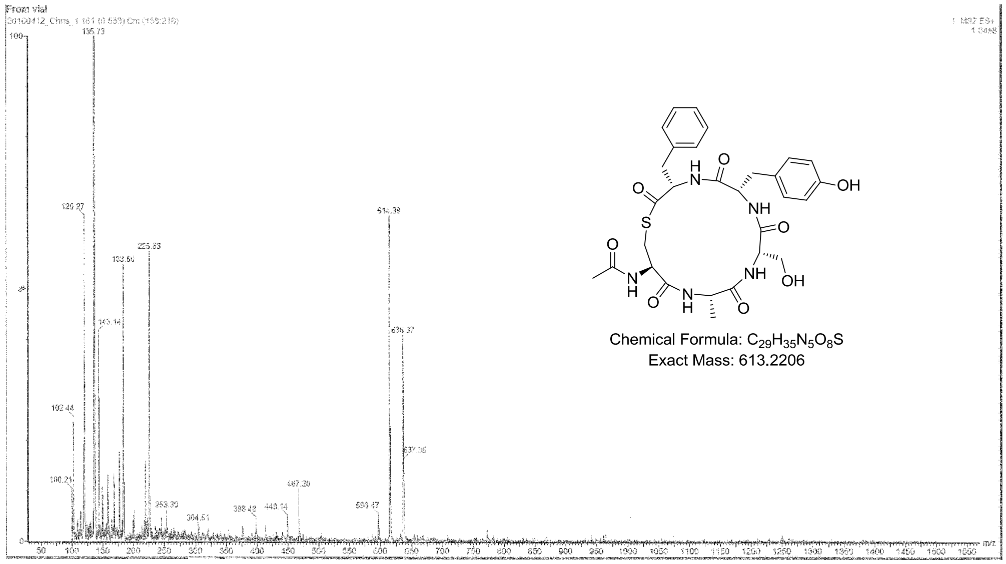
Data File C:\CHEM32\1\DATA\S_EPI AIPI WITH HOAT 2016-04-05 14-13-10\043-0301.D
Sample Name: S epi AIP I cyclised with HOAt t = 2

```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   3
Acq. Instrument : LC1260                         Location : Vial 43
Injection Date  : 4/5/2016 3:17:39 PM                 Inj :   1
                                                Inj Volume : 20.000 µl
Acq. Method     : C:\CHEM32\1\DATA\S_EPI AIPI WITH HOAT 2016-04-05 14-13-10\10 TO 100 OVER 15
                  MINS 20UL.M
Last changed    : 4/5/2016 2:13:10 PM by SYSTEM
Analysis Method : C:\CHEM32\1\DATA\S_EPI AIPI WITH HOAT 2016-04-05 14-13-10\10 TO 100 OVER 15
                  MINS 20UL.M (Sequence Method)
Last changed    : 4/5/2016 4:09:36 PM by SYSTEM
                  (modified after loading) (Current integration events modified)
```




```
==========================================================================================
                          Fraction Information
==========================================================================================
==========================================================================================
No Fractions found.
==========================================================================================
==========================================================================
                        Area Percent Report
==========================================================================

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


From vial
135.07
164.12
225.90
120.14
183.36
102.36
253.37
100.36
260.23
614.22
636.37
292.41
354.54
376.44
398.43
Chemical Formula: C29H35N5O8S
Exact Mass: 613.2206


From vial
135.73
120.27
225.53
183.50
143.14
102.44
100.21
253.39
304.51
467.30
556.47
614.39
636.37
637.36
Chemical Formula: $C_{29}H_{35}N_5O_8S$
Exact Mass: 613.2206

References

1. M. E. Olson, D. A. Todd, C. R. Schaeffer, A. E. Paharik, M. J. Van Dyke, H. Buettner, P. M. Dunman, H. Rohde, N. B. Cech, P. D. Fey and A. R. Horswill, *J. Bacteriol.*, 2014, **196**, 3482-3493, 3413 pp.
2. S. Heilbronner, M. T. G. Holden, A. van Tonder, J. A. Geoghegan, T. J. Foster, J. Parkhill and S. D. Bentley, *FEMS Microbiol. Lett.*, 2011, **322**, 60-67.
3. S. Heilbronner, F. Hanses, I. R. Monk, P. Speziale and T. J. Foster, *Microbiology*, 2013, **159**, 2141-2152.