

Supporting Information

Identification of 3-acetyl-2-aminoquinolin-4-one as a novel, nonpeptidic scaffold for specific calpain inhibitory activity

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Result of optimization of fluorimetric calpain assay 1

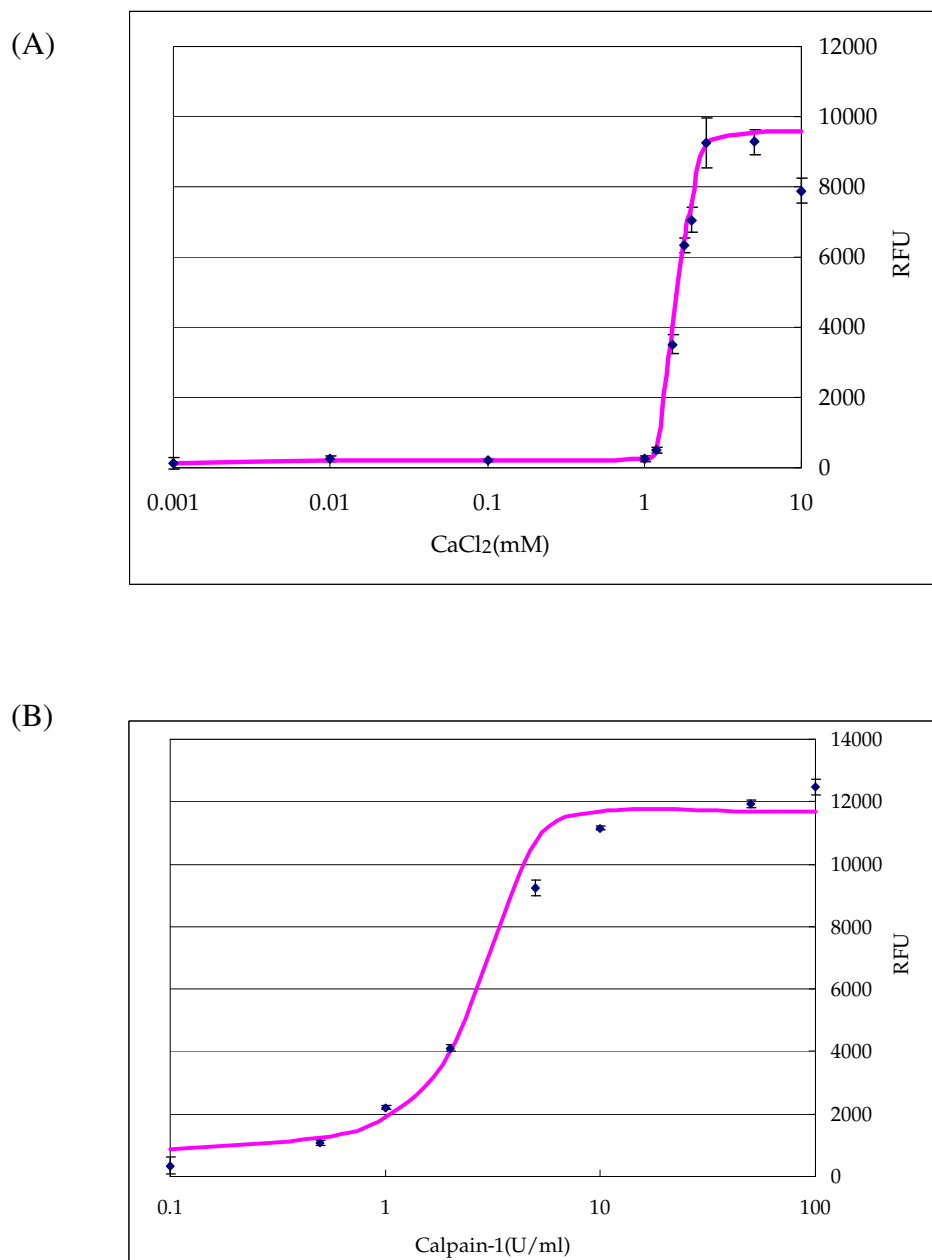


Figure S1. Effect of CaCl_2 concentration (A) and μ -calpain amount (B) on the proteolytic activity of μ -calpain.

General Procedure for LC/MS: LC/MS was employed to check the purity of the compounds. LC/MS was carried out with a Waters 2795(LC-MS) Micromass (ZQ 2000) equipped with XTerra® MS C18 column, 4.6×5mm, 5µm, HPLC with a linear gradient 5% to 80% 0.02% CF₃CO₂H/ACN into 0.02% CF₃CO₂H/H₂O over 2.5 to 3.5 minutes at flow rate 2 mL/min. UV detection (PDA Detector(2996)) was carried out at wavelength 254nm. Data are detailed in Table S1. Traces and MS data are presented in compound number order thereafter.

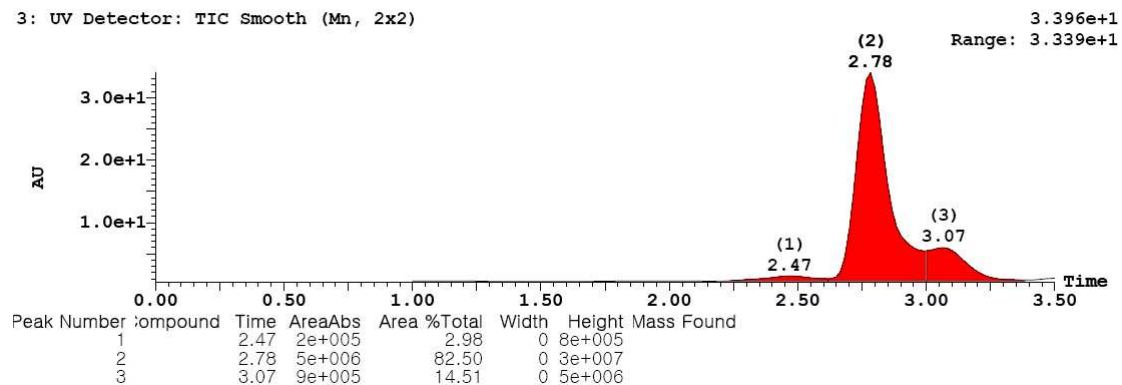
Table S1. LC/MS analyses for compounds 1-22.

Compound	Formula	M.W.	LC rt (min)	m/z	Purity (%)
1	C ₁₇ H ₁₁ Cl ₂ N ₃ O ₄	392.2	2.78	392.0	97
2	C ₂₁ H ₂₀ ClN ₃ O ₄	413.9	4.07	414.1	96
3	C ₁₈ H ₁₁ Cl ₂ F ₃ N ₂ O ₂	415.2	2.87	415.1	97
4	C ₁₉ H ₁₁ ClF ₆ N ₂ O ₂	448.7	3.08	449.2	94
5	C ₁₈ H ₁₃ Cl ₂ N ₃ O ₄	406.2	3.83	406.0	97
6	C ₁₇ H ₁₀ Cl ₃ N ₃ O ₄	426.6	3.85	427.9	94
7	C ₁₈ H ₁₁ Cl ₂ F ₃ N ₂ O ₂	415.2	2.95	415.0	98
8	C ₂₃ H ₁₇ FN ₂ O ₂	372.4	2.70	373.2	96
9	C ₁₇ H ₁₁ Br ₃ N ₂ O ₂	515.0	3.82	514.8	69
10	C ₁₇ H ₁₁ BrCl ₂ N ₂ O ₂	426.1	2.85	427.0	94
11	C ₁₇ H ₁₀ Cl ₄ N ₂ O ₂	416.1	3.82	416.9	94
12	C ₁₈ H ₁₄ ClN ₃ O ₄	371.8	2.73	372.1	98
13	C ₁₇ H ₁₂ ClFN ₂ O ₂	330.7	2.45	331.1	100
14	C ₂₁ H ₂₀ F ₂ N ₂ O ₂	370.4	2.83	371.2	83
15	C ₁₇ H ₁₁ ClF ₂ N ₂ O ₂	348.7	2.38	349.1	88
16	C ₁₉ H ₁₀ F ₈ N ₂ O ₂	450.3	2.72	451.1	98
17	C ₁₈ H ₁₃ Br ₂ ClN ₂ O ₂	484.6	4.17	484.8	88
18	C ₁₈ H ₁₃ Cl ₂ FN ₂ O ₂	379.2	2.98	379.1	98
19	C ₁₈ H ₁₂ ClF ₃ N ₂ O ₂	380.7	2.90	381.1	100
20	C ₁₈ H ₁₂ ClF ₃ N ₂ O ₂	380.7	2.75	381.1	98
21	C ₁₉ H ₁₃ ClF ₄ N ₂ O ₂	412.8	2.93	413.1	97
22	C ₁₇ H ₉ F ₅ N ₂ O ₂	368.3	2.50	369.1	99

Compound 1

Sample 53 Vial 1:53 ID 53 File ri845-53 Date 12-May-2006 Time 21:57:11 Description

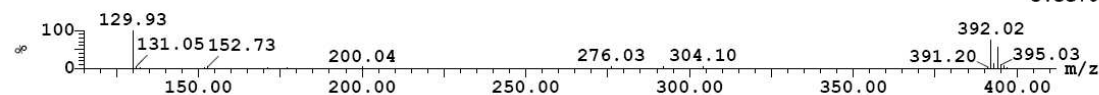
3: UV Detector: TIC Smooth (Mn, 2x2)



Peak ID :omponent Time Mass Found
2 2.78

Combine (117:121-88:91)

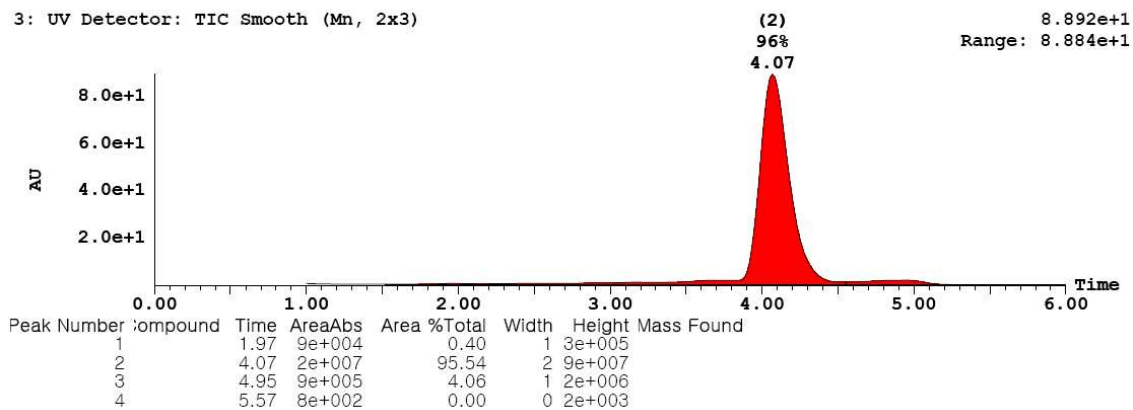
1:MS ES+
5.3e+005



Compound 2

Sample 1 Vial 3:1 ID A File ri847-1 Date 27-Nov-2008 Time 14:31:25 Description

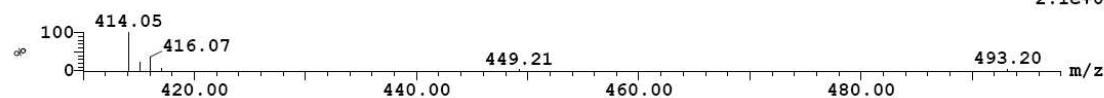
3: UV Detector: TIC Smooth (Mn, 2x3)



Peak ID :omponent Time Mass Found
2 4.07

Combine (200:204-(80:82+251:253))

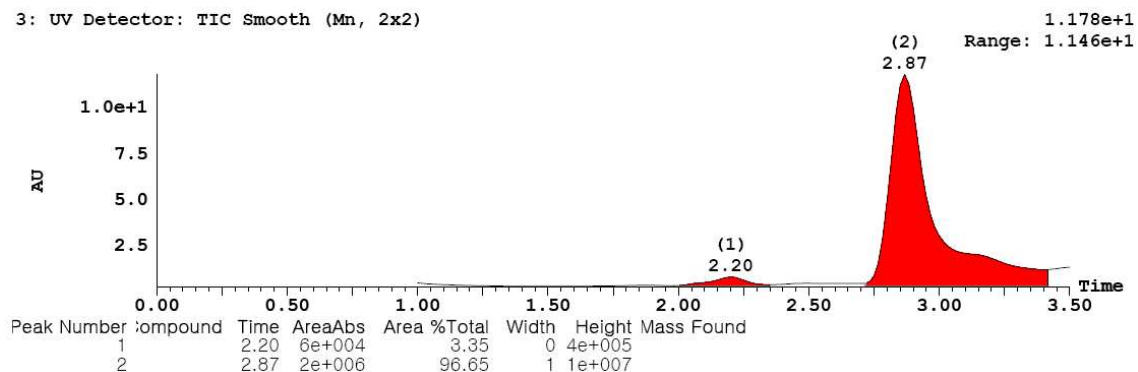
1:MS ES+
2.1e+007



Compound 3

Sample 6 Vial 3:6 ID 6 File ri915-06 Date 31-May-2006 Time 01:00:19 Description

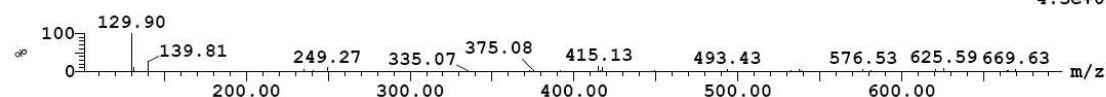
3: UV Detector: TIC Smooth (Mn, 2x2)



Peak ID :compound Time Mass Found
2 2.87

Combine (120:125-93:95)

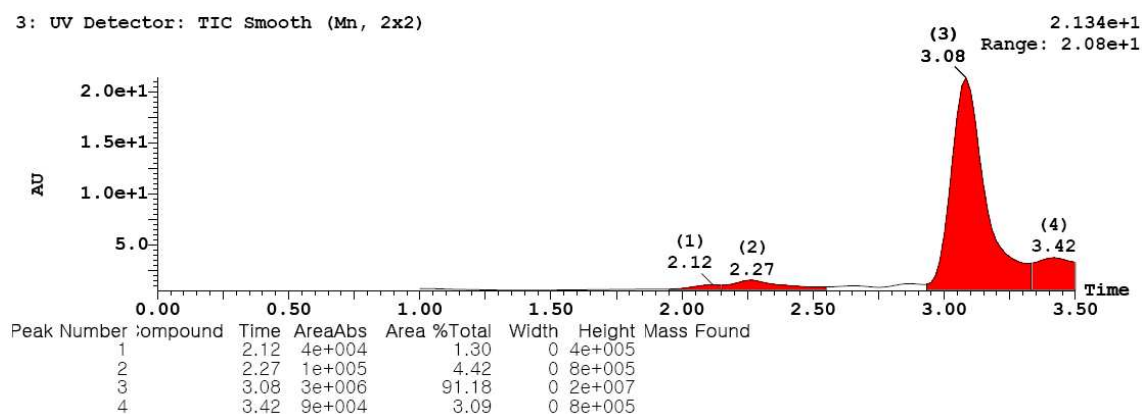
1:MS ES+
4.3e+005



Compound 4

Sample 88 Vial 1:88 ID 88 File ri845-88 Date 13-May-2006 Time 00:19:23 Description

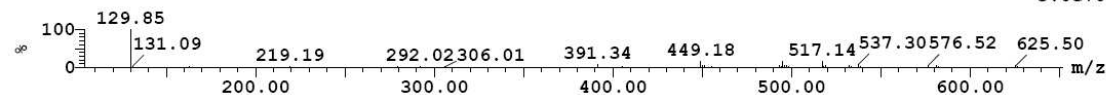
3: UV Detector: TIC Smooth (Mn, 2x2)



Peak ID :compound Time Mass Found
3 3.08

Combine (129:134-102:104)

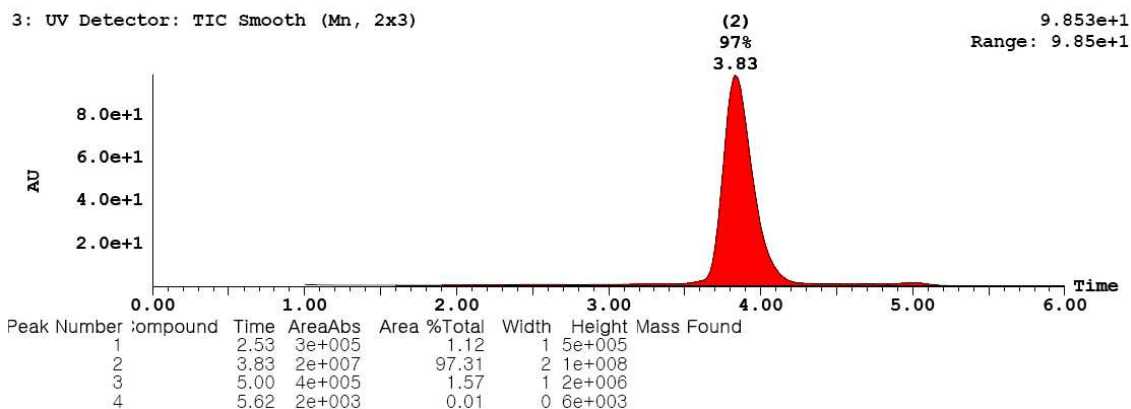
1:MS ES+
5.0e+005



Compound 5

Sample 2 Vial 3:2 ID B File ri847-2 Date 27-Nov-2008 Time 14:41:51 Description

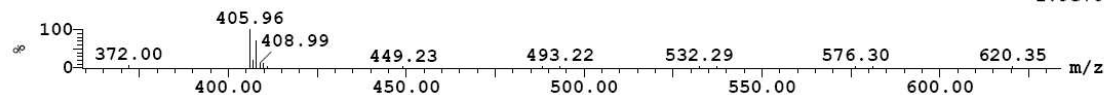
3: UV Detector: TIC Smooth (Mn, 2x3)



Peak ID :compound Time Mass Found
2 3.83

Combine (188:193-(111:114+265:267))

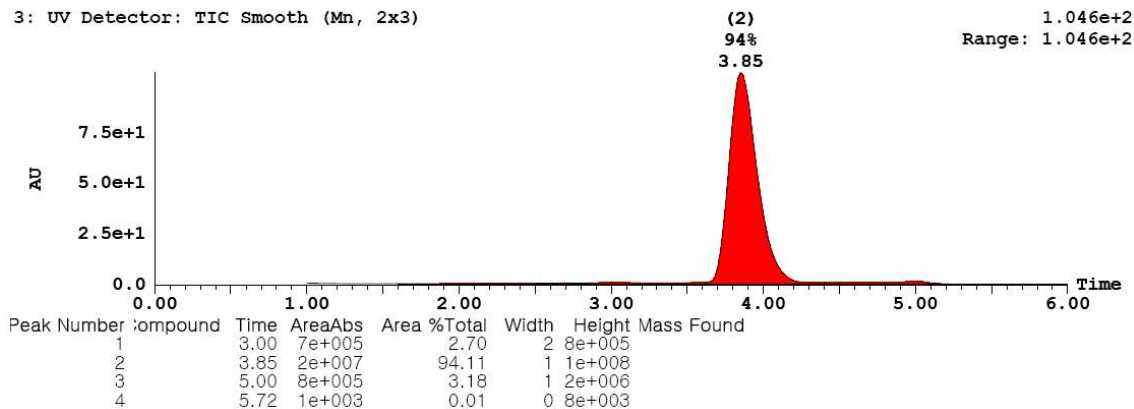
1:MS ES+
1.9e+007



Compound 6

Sample 3 Vial 3:3 ID C File ri847-3 Date 27-Nov-2008 Time 14:48:23 Description

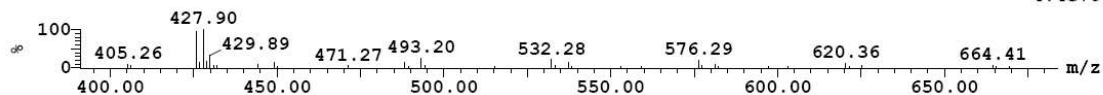
3: UV Detector: TIC Smooth (Mn, 2x3)



Peak ID :compound Time Mass Found
2 3.85

Combine (189:194-(143:146+243:246))

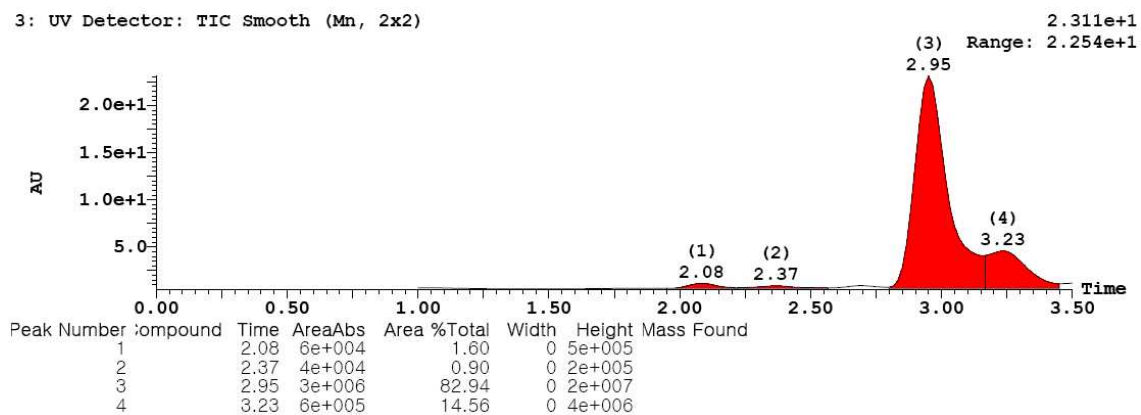
1:MS ES+
4.4e+006



Compound 7

Sample 31 Vial 1:31 ID 31 File ri845-31 Date 12-May-2006 Time 20:27:45 Description

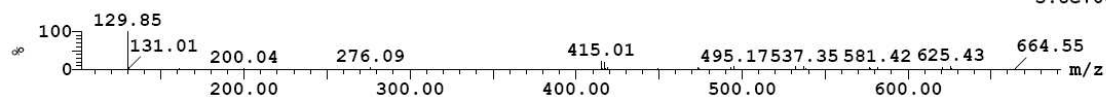
3: UV Detector: TIC Smooth (Mn, 2x2)



Peak ID :compound Time Mass Found
3 2.95

Combine (124:128-96:98)

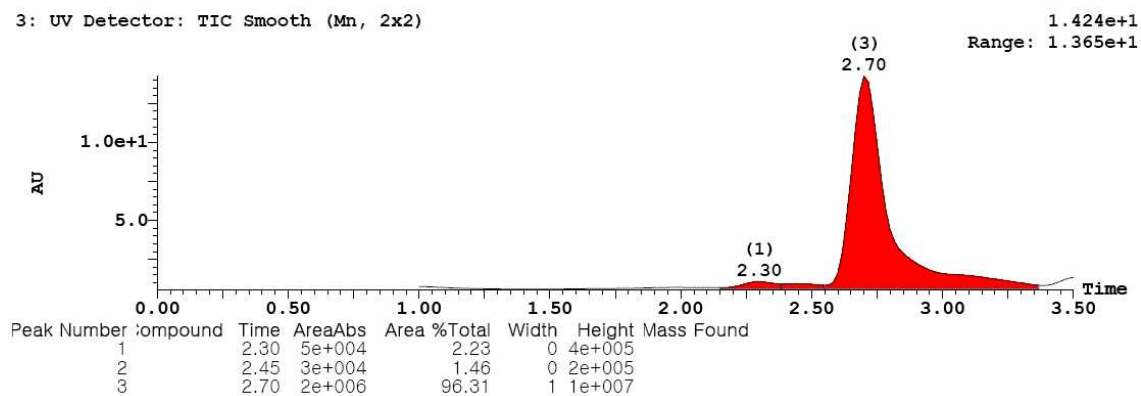
1:MS ES+
5.8e+005



Compound 8

Sample 76 Vial 3:76 ID 76 File ri806-76 Date 05-May-2006 Time 06:22:38 Description

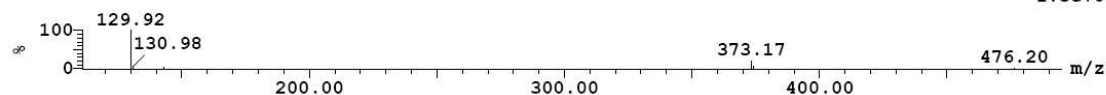
3: UV Detector: TIC Smooth (Mn, 2x2)



Peak ID :compound Time Mass Found
3 2.70

Combine (113:117-86:88)

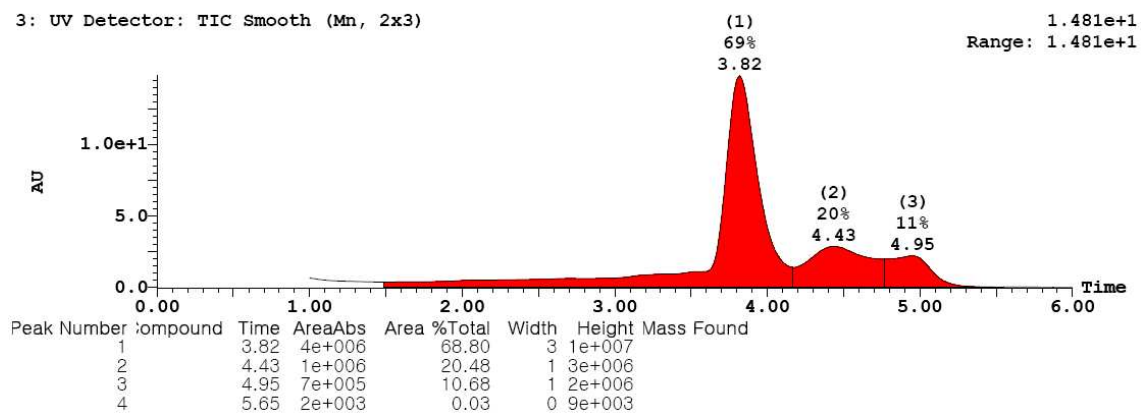
1:MS ES+
1.5e+006



Compound 9

Sample 4 Vial 3:4 ID D File r1847-4 Date 27-Nov-2008 Time 14:54:59 Description

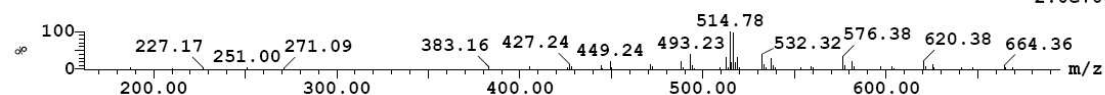
3: UV Detector: TIC Smooth (Mn, 2x3)



Peak ID :compound Time Mass Found
1 3.82

Combine (187:192-(47:49+232:234))

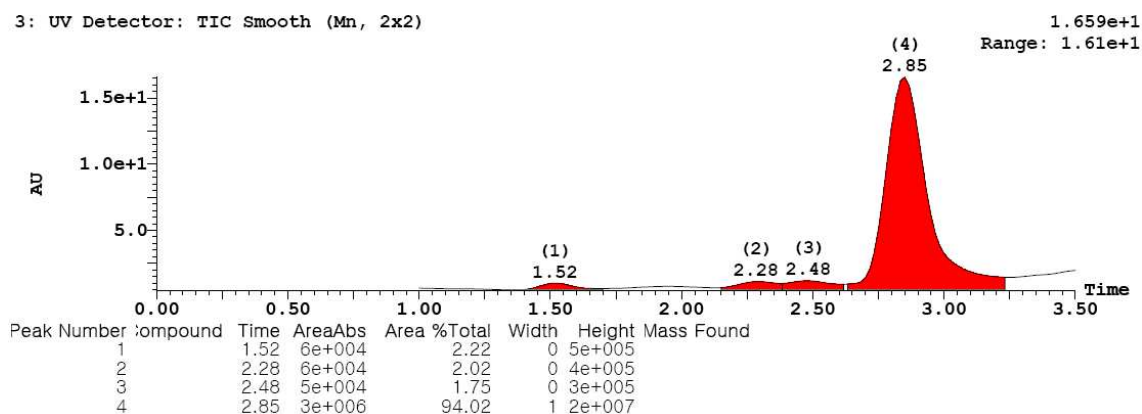
1:MS ES+
2.0e+006



Compound 10

Sample 10 Vial 1:10 ID 10 File r1863-10 Date 17-May-2006 Time 18:37:27 Description

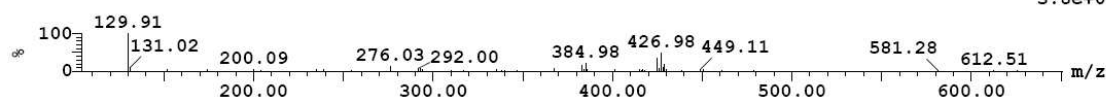
3: UV Detector: TIC Smooth (Mn, 2x2)



Peak ID :compound Time Mass Found
4 2.85

Combine (120:124-89:91)

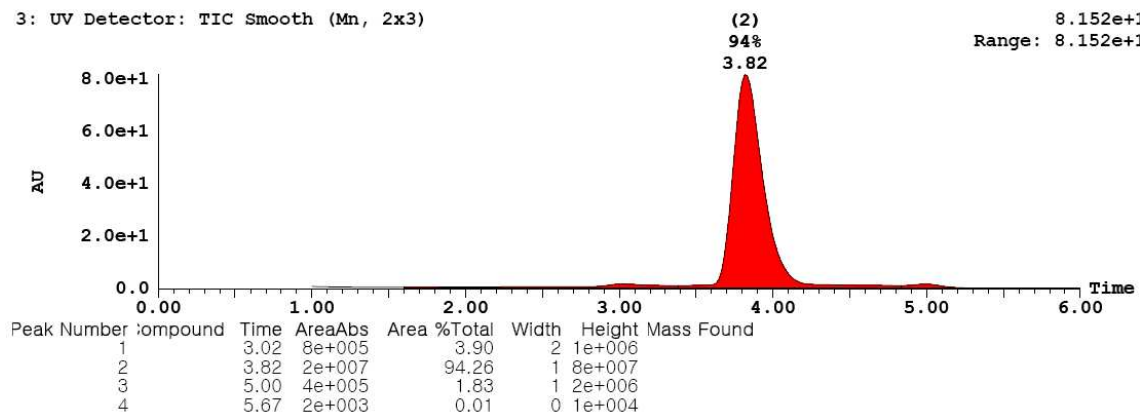
1:MS ES+
3.8e+005



Compound 11

Sample 5 Vial 3:5 ID E File ri847-5 Date 27-Nov-2008 Time 15:01:35 Description

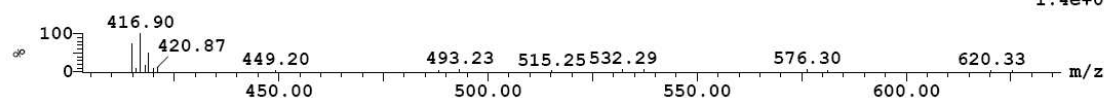
3: UV Detector: TIC Smooth (Mn, 2x3)



Peak ID :compound Time Mass Found
2 3.82

Combine (187:192-(141:143+265:267))

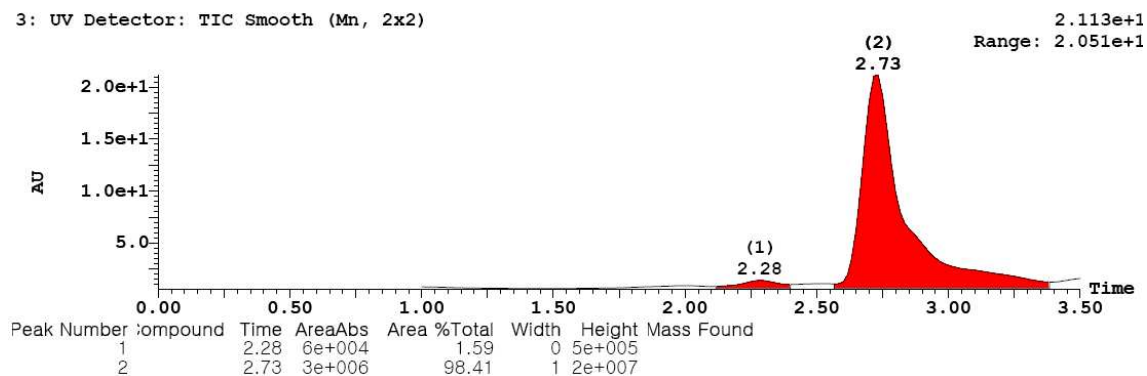
1:MS ES+
1.4e+007



Compound 12

Sample 92 Vial 3:92 ID 92 File ri806-92 Date 05-May-2006 Time 07:27:47 Description

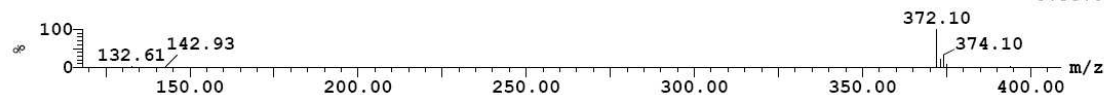
3: UV Detector: TIC Smooth (Mn, 2x2)



Peak ID :compound Time Mass Found
2 2.73

Combine (115:119-86:88)

1:MS ES+
8.5e+005

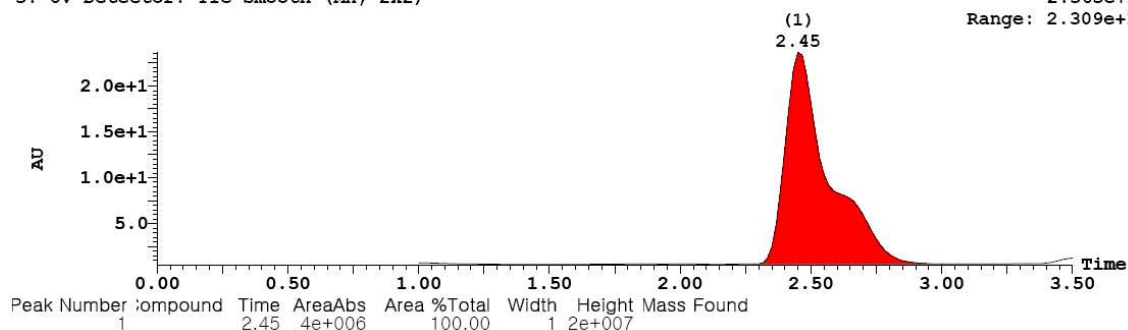


Compound 13

Sample 24 Vial 2:24 ID 24 File ri839-24 Date 12-May-2006 Time 03:23:09 Description

3: UV Detector: TIC Smooth (Mn, 2x2)

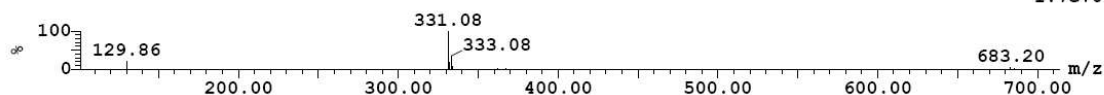
2.363e+1
Range: 2.309e+1



Peak ID :compound Time Mass Found
1 2.45

Combine (103:107-74:76)

1:MS ES+
1.7e+006

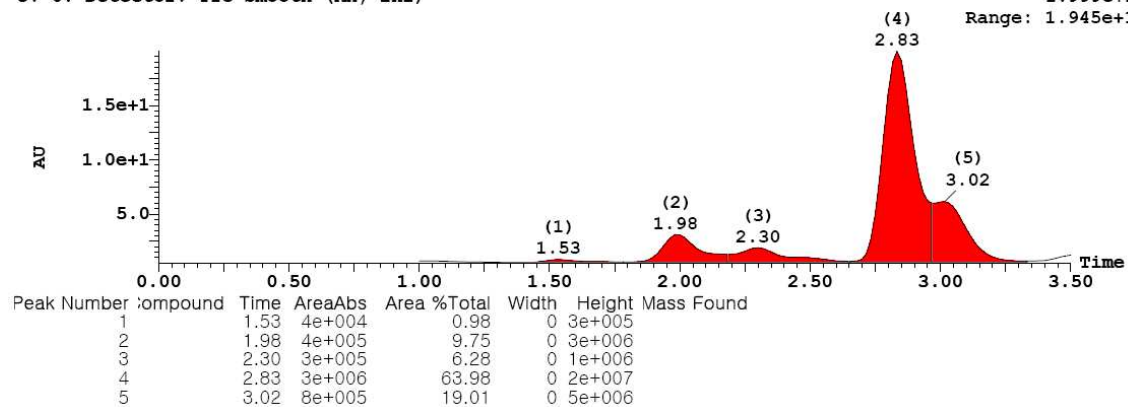


Compound 14

Sample 11 Vial 2:11 ID 11 File ri839-11 Date 12-May-2006 Time 02:30:15 Description

3: UV Detector: TIC Smooth (Mn, 2x2)

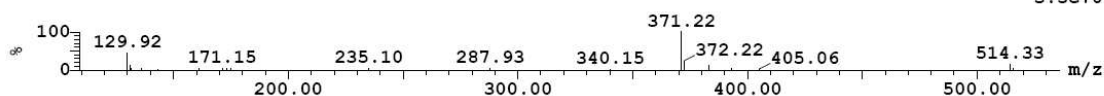
1.999e+1
Range: 1.945e+1



Peak ID :compound Time Mass Found
4 2.83

Combine (119:123-(90:92+148:149))

1:MS ES+
3.5e+005

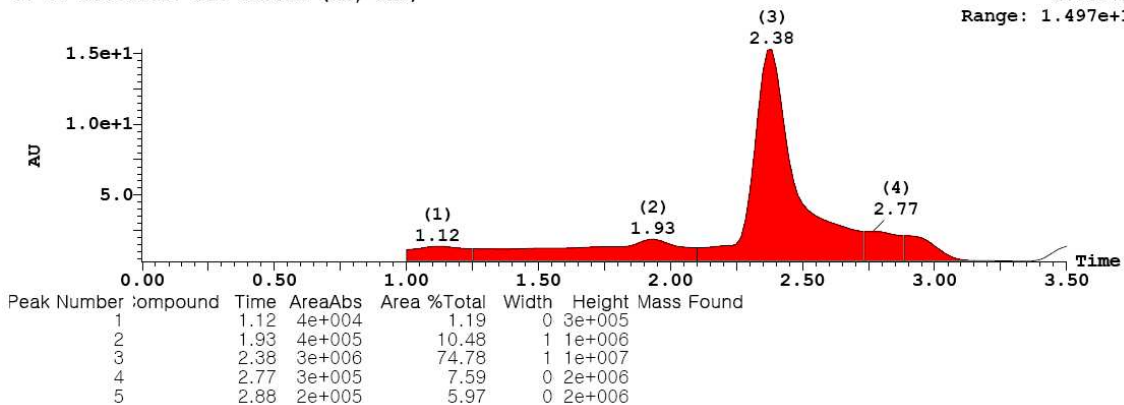


Compound 15

Sample 84 Vial 2:84 ID 84 File ri899-84 Date 27-May-2006 Time 06:23:25 Description

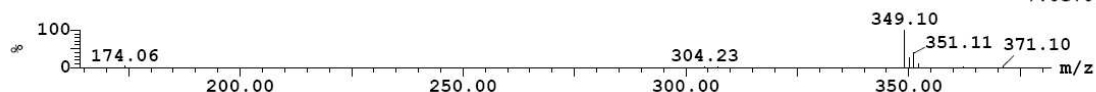
3: UV Detector: TIC Smooth (Mn, 2x2)

1.53e+1
Range: 1.497e+1



Peak ID :compound Time Mass Found
3 2.38
Combine (100:104-(66:69+138:140))

1:MS ES+
7.0e+005

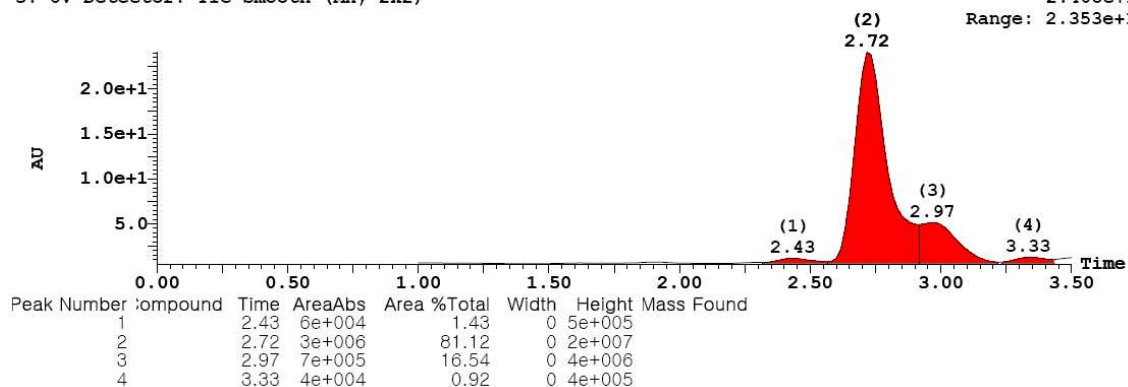


Compound 16

Sample 42 Vial 1:42 ID 42 File ri845-42 Date 12-May-2006 Time 21:12:28 Description

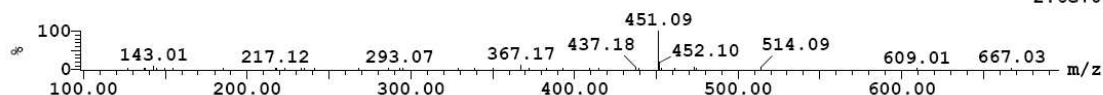
3: UV Detector: TIC Smooth (Mn, 2x2)

2.408e+1
Range: 2.353e+1



Peak ID :compound Time Mass Found
2 2.72
Combine (114:118-(86:88+146:148))

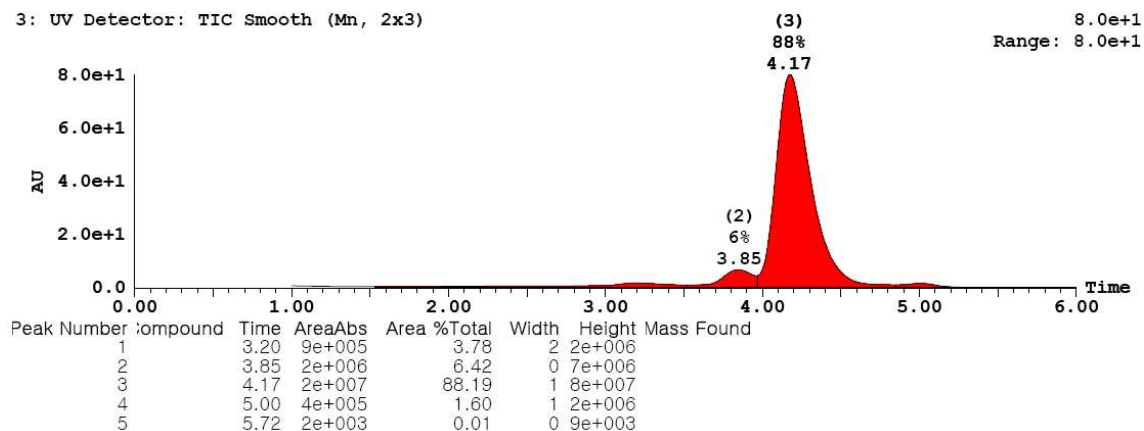
1:MS ES+
2.6e+005



Compound 17

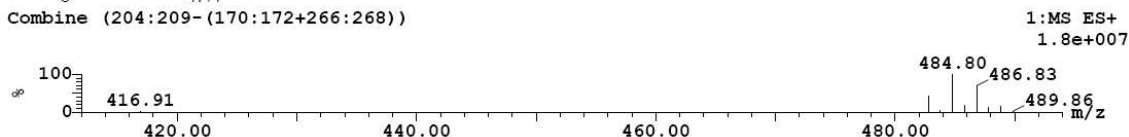
Sample 6 Vial 3:6 ID F File ri847-6 Date 27-Nov-2008 Time 15:08:07 Description

3: UV Detector: TIC Smooth (Mn, 2x3)



Peak ID :omponent Time Mass Found
3 4.17

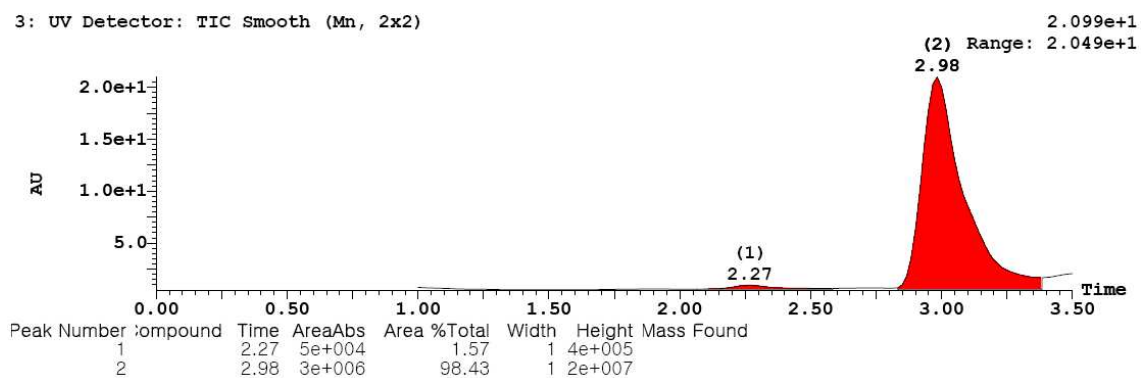
Combine (204:209-(170:172+266:268))



Compound 18

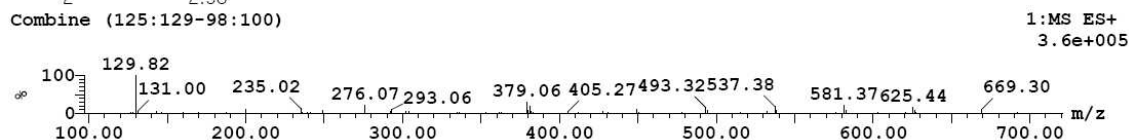
Sample 59 Vial 1:59 ID 59 File ri863-59 Date 17-May-2006 Time 21:56:33 Description

3: UV Detector: TIC Smooth (Mn, 2x2)



Peak ID :omponent Time Mass Found
2 2.98

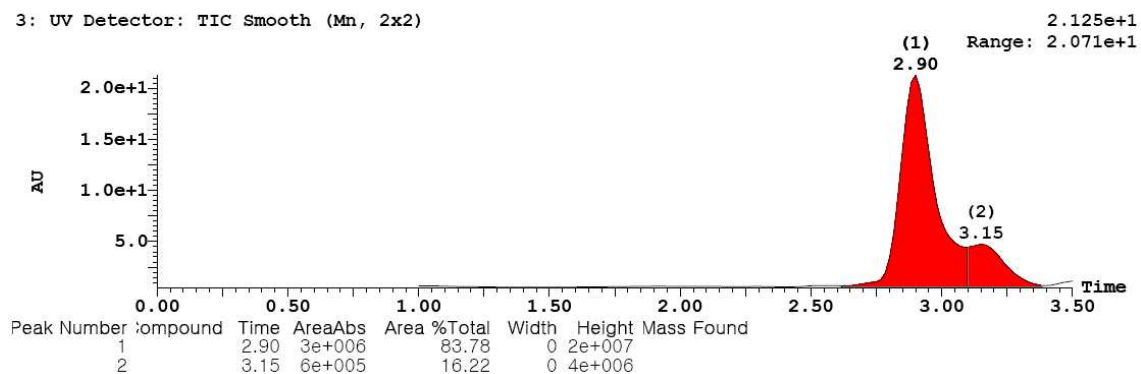
Combine (125:129-98:100)



Compound 19

Sample 5 Vial 1:5 ID 5 File ri845-05 Date 12-May-2006 Time 18:42:00 Description

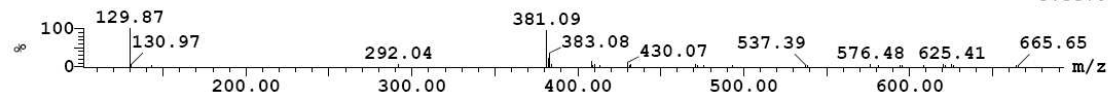
3: UV Detector: TIC Smooth (Mn, 2x2)



Peak ID :compound Time Mass Found
1 2.90

Combine (122:126-88:91)

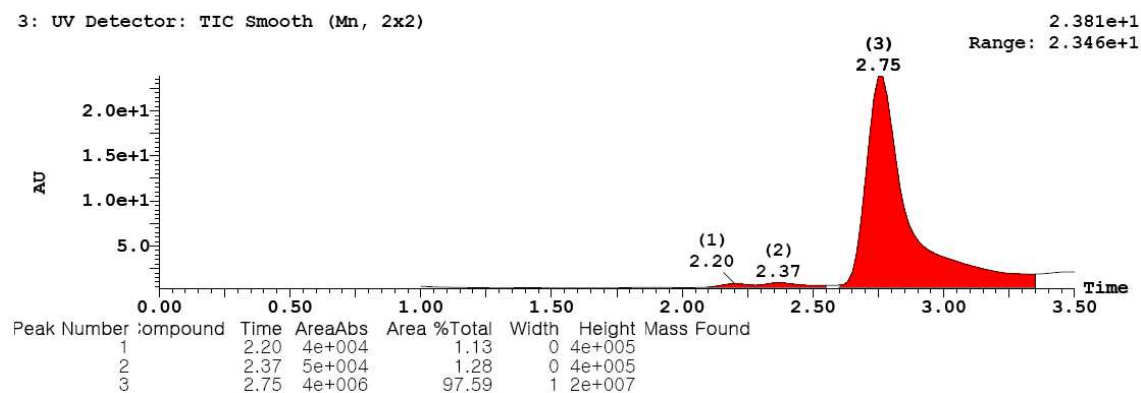
1:MS ES+
5.5e+005



Compound 20

Sample 7 Vial 3:7 ID 7 File ri915-07 Date 31-May-2006 Time 01:04:22 Description

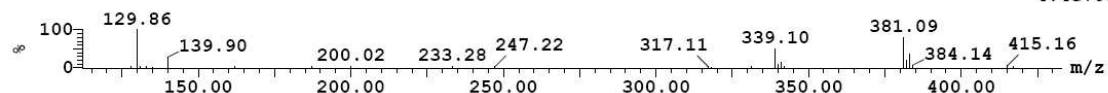
3: UV Detector: TIC Smooth (Mn, 2x2)



Peak ID :compound Time Mass Found
3 2.75

Combine (115:120-88:90)

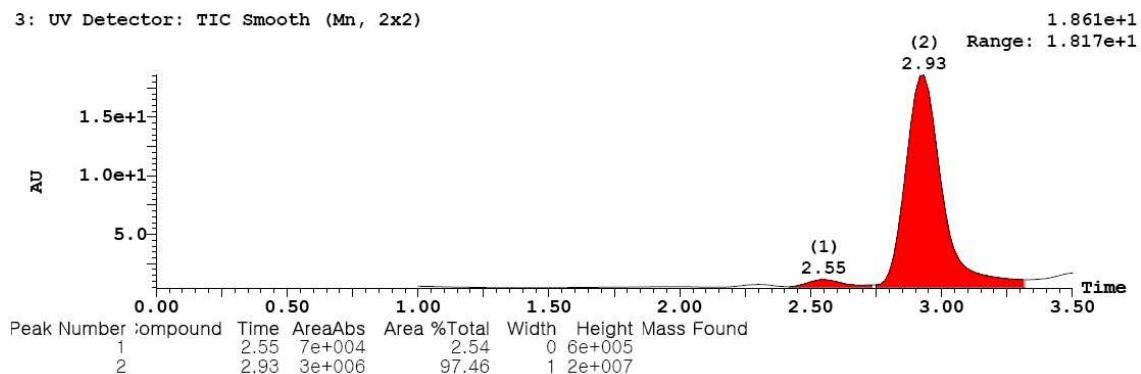
1:MS ES+
4.4e+005



Compound 21

Sample 4 Vial 1:4 ID 4 File ri863-04 Date 17-May-2006 Time 18:13:01 Description

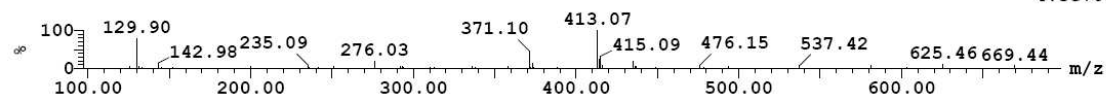
3: UV Detector: TIC Smooth (Mn, 2x2)



Peak ID :compound Time Mass Found
2 2.93

Combine (123:127-94:96)

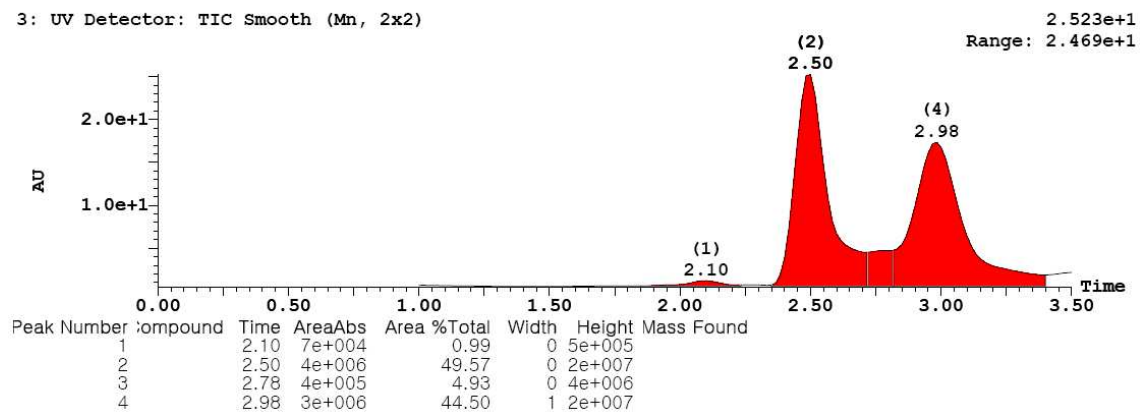
1:MS ES+
4.5e+005



Compound 22

Sample 65 Vial 1:65 ID 65 File ri845-65 Date 12-May-2006 Time 22:45:58 Description

3: UV Detector: TIC Smooth (Mn, 2x2)



Peak ID :compound Time Mass Found
2 2.50

Combine (105:109-(77:79+137:139))

1:MS ES+
2.3e+006

