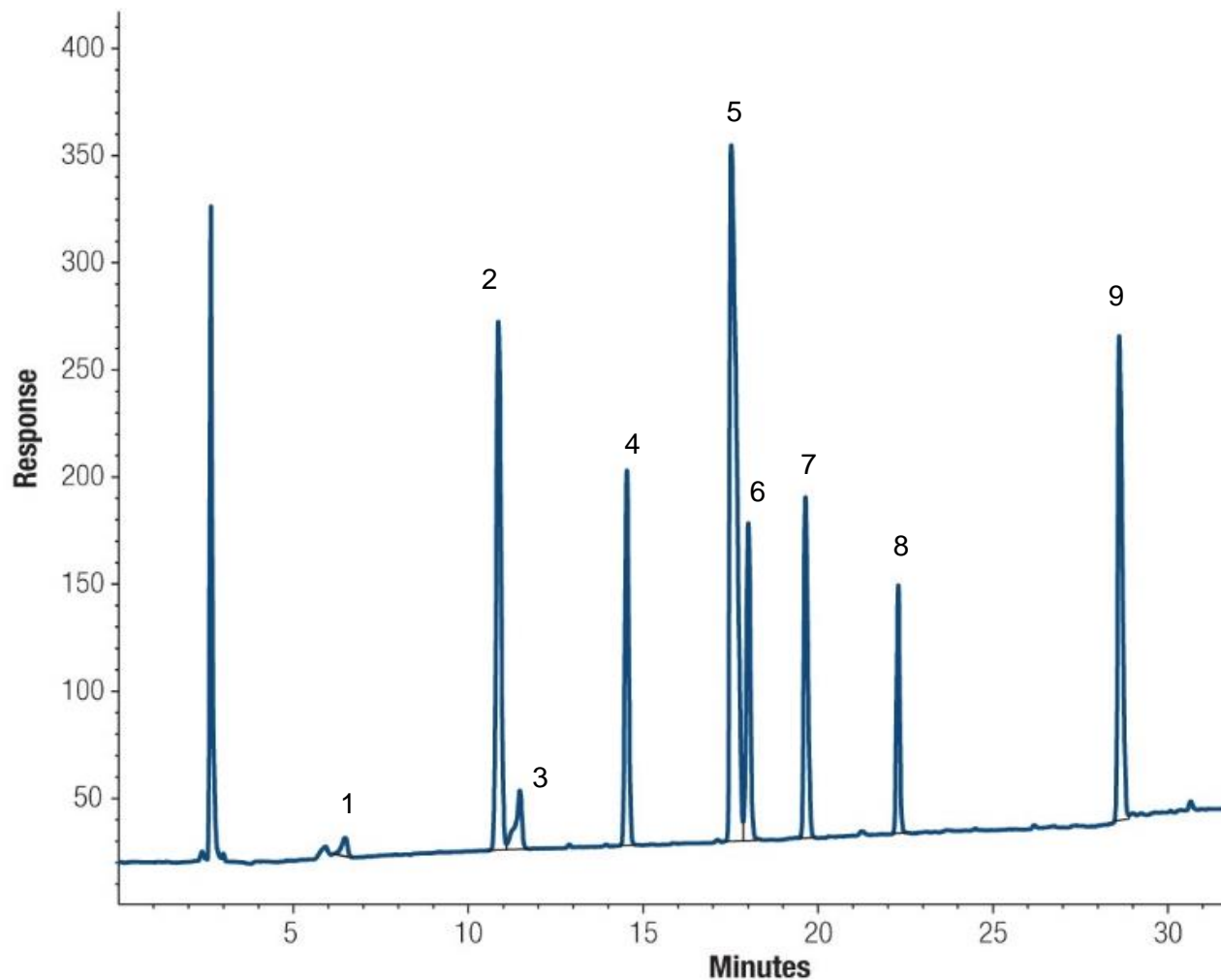




# Artificial Sweeteners Global Method



1. Acesulfame K
2. Cyclamate
3. Saccharin
4. Sucralose
5. Aspartame
6. Neotame
7. Alitame
8. Neohesperidin dihydrochalcone
9. Dulcin

ACE C18, 5 $\mu$ m, 250 x 4.6mm

Gradient analysis

A: Water

B: Acetonitrile

C: 0.1% TFA

Time (mins)	%A	%B	%C
0	88	2	10
25	50	40	10
30	30	60	10
35	88	2	10

Flow rate: 1 ml/min

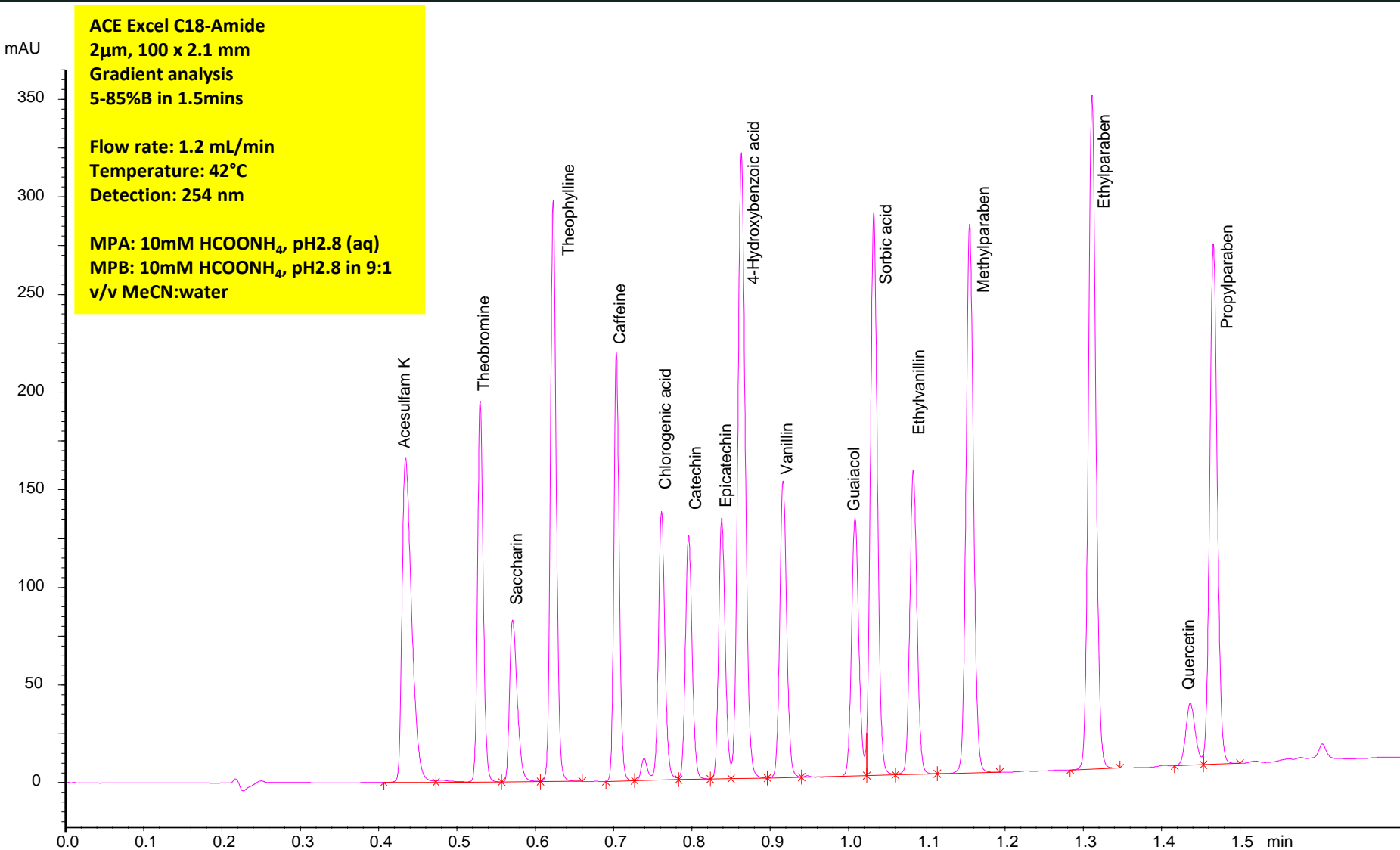
Column temperature: 30°C

Injection volume: 50 $\mu$ l

Detection: Corona CAD



# Chocolate Analysis





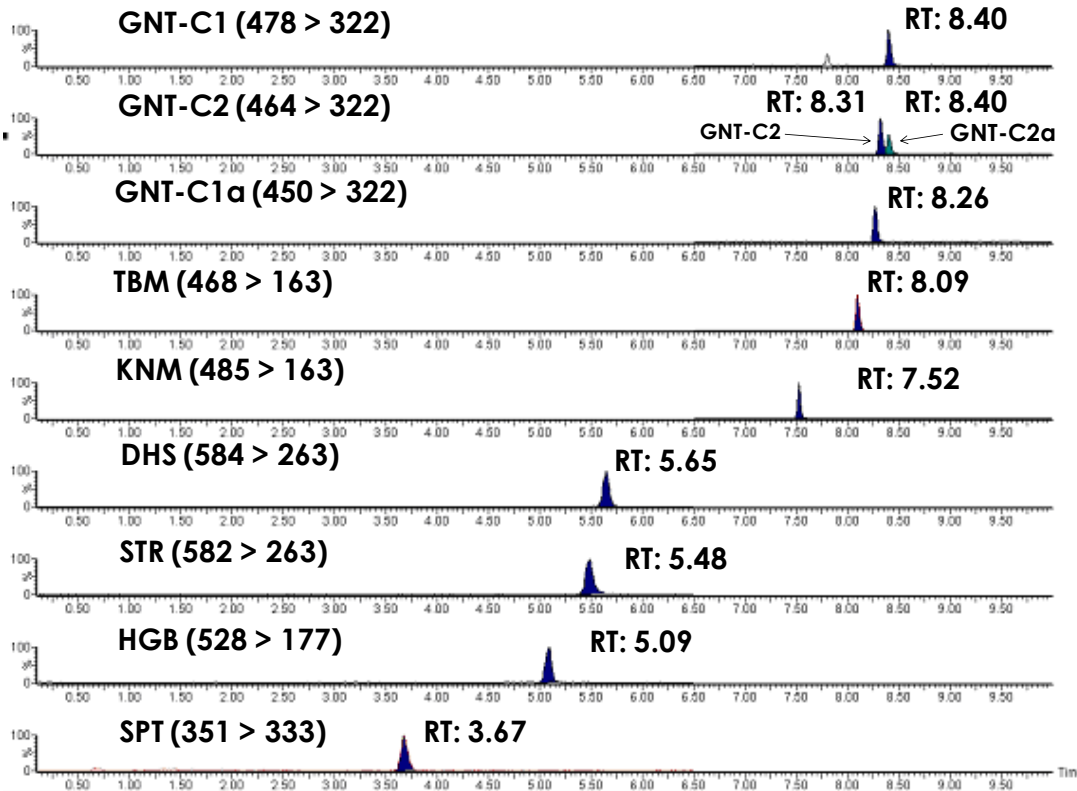
# Aminoglycosides in Eggs

UHPLC-MS/MS method for aminoglycosides in eggs

Extraction at low pH, clean-up with WCX SPE cartridge

Egg sample spiked at 100µg/kg (CCα)

- Key
- GNT Gentamicin
  - TBM Tobramycin
  - KNM Kanamycin
  - DHS Dihydrostreptomycin
  - STR Streptomycin
  - HGB Higromycin-B
  - SPT Spectinomycin



ACE Excel C18-PFP, 2µm, 100 x 2.1mm  
Gradient analysis

A = 20mM HFBA in H<sub>2</sub>O/CH<sub>3</sub>CN (98:2)

B = 20mM HFBA in CH<sub>3</sub>CN/H<sub>2</sub>O (98:2)

Time (mins) %B Curve

0	5	-
2	15	6
4.5	19	6
5.5	19.5	8
6	22	6
7	35	6
9	48	8
9.5	5	6

Flow rate: 0.4ml/min

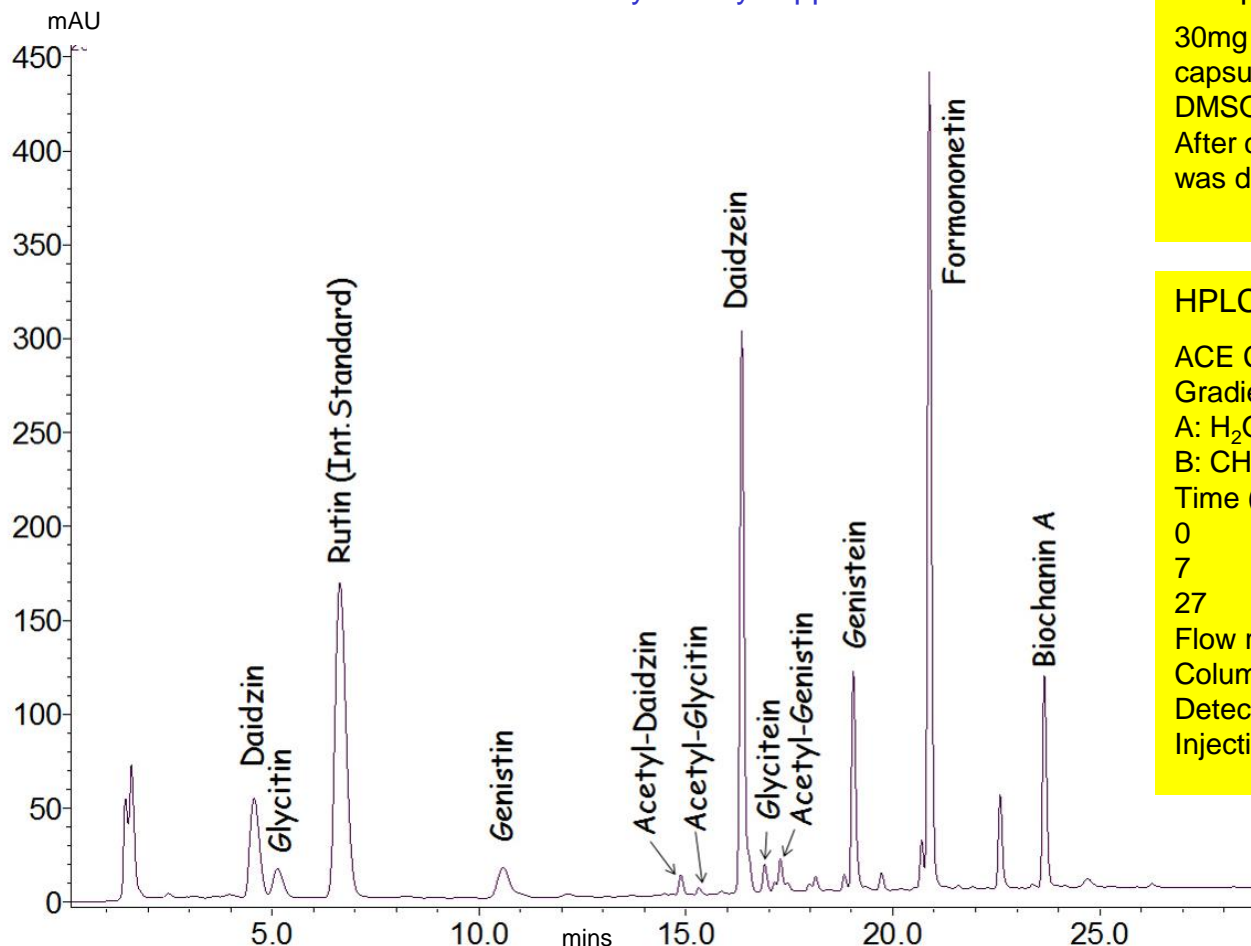
Column temperature: 40°C

Positive ESI MRM (transitions as shown)



# Isoflavones in Red Clover and Soy Extract

Extract from red clover and soy dietary supplement



## Sample preparation

30mg of homogenised capsules/tablets extracted into 3ml DMSO-H<sub>2</sub>O (3:1) by ultrasonication. After centrifuging, 1ml of supernatant was diluted with 4ml DMSO-H<sub>2</sub>O (3:1)

## HPLC Analysis

ACE C18-AR, 3 $\mu$ m, 150 x 2.1mm

Gradient analysis

A: H<sub>2</sub>O – CH<sub>3</sub>COOH, pH 2.8

B: CH<sub>3</sub>CN + 0.6% CH<sub>3</sub>OOH

Time (mins)	%B
0	15
7	15
27	75

Flow rate: 0.35ml/min

Column temperature: 25°C

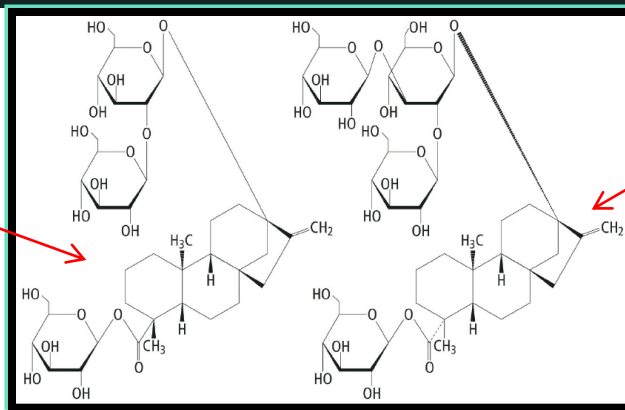
Detection: UV, 254nm

Injection volume: 3 $\mu$ l

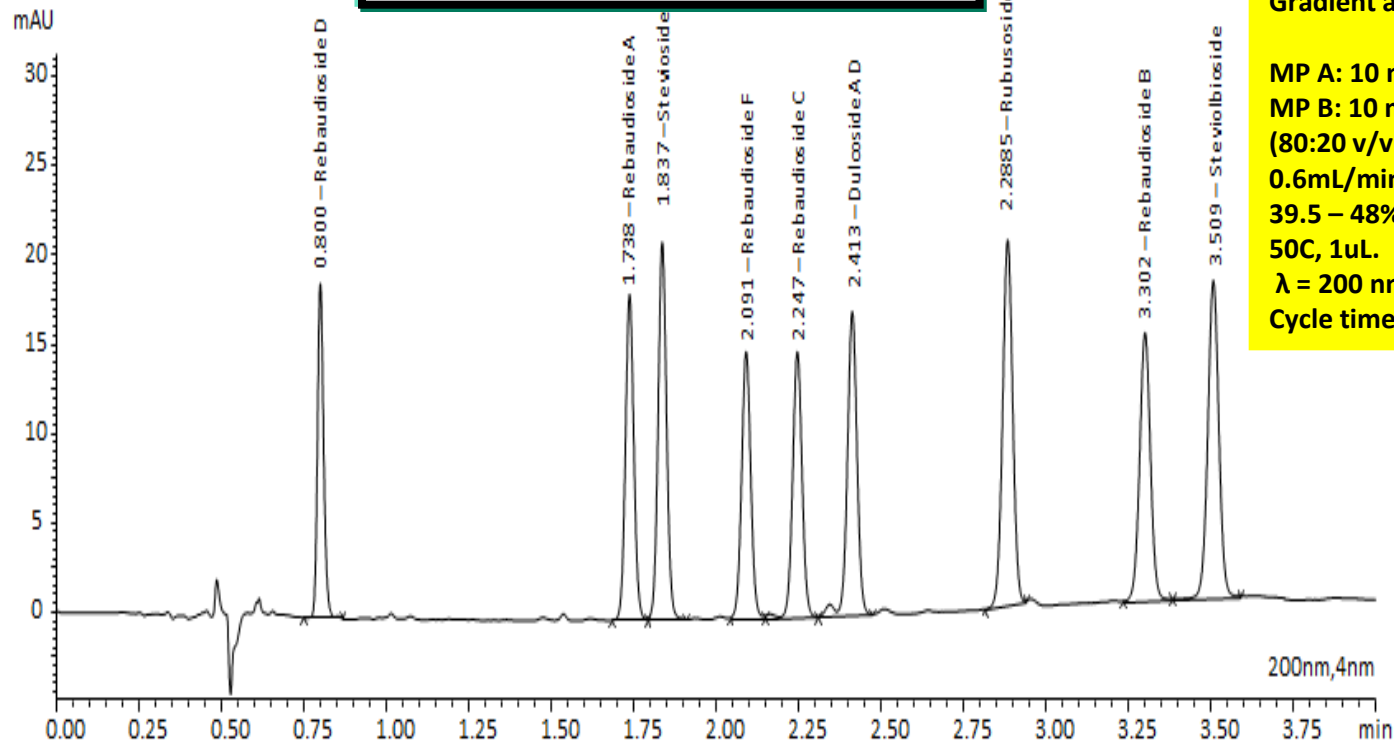


# Artificial Sweeteners (Stevia Glycosides)

Stevioside  
MW = 804.87



Rebaudioside  
MW = 967.01

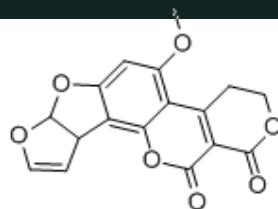


ACE Excel SuperC18, 2 $\mu$ m, 150 x 2.1 mm  
Gradient analysis

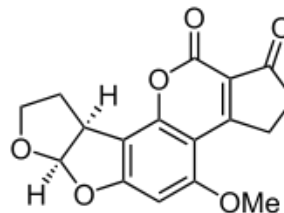
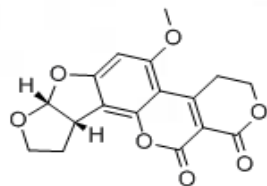
MP A: 10 mM NaH<sub>2</sub>PO<sub>4</sub>, pH 2.8 in H<sub>2</sub>O.  
MP B: 10 mM NaH<sub>2</sub>PO<sub>4</sub>, pH 2.8 in MeCN/H<sub>2</sub>O (80:20 v/v).  
0.6mL/min  
39.5 – 48%B in 4 mins.  
50C, 1 $\mu$ L.  
 $\lambda$  = 200 nm.  
Cycle time = 7 mins.



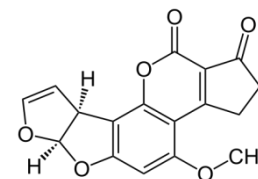
# Mycotoxins / Aflatoxins From Peppers



Aflatoxin G1

Aflatoxin B<sub>2</sub>

Aflatoxin G2

Aflatoxin B<sub>1</sub>

ACE C18-PFP, 3um, 150 x 4.6 mm

Isocratic analysis

Flow rate: 1 mL / min

Temperature: 25°C

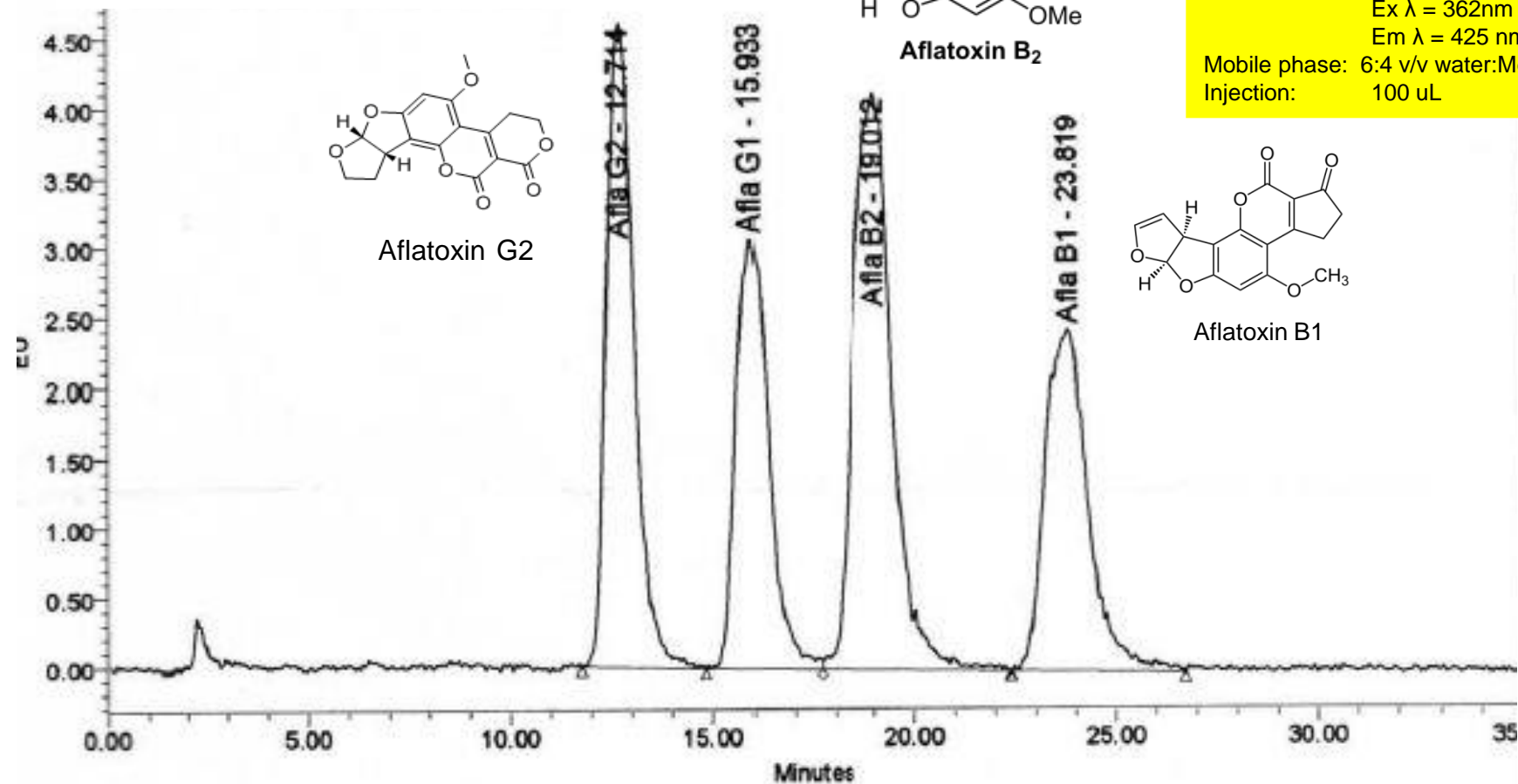
Detection: Fluorescence

Ex λ = 362nm

Em λ = 425 nm

Mobile phase: 6:4 v/v water:MeOH

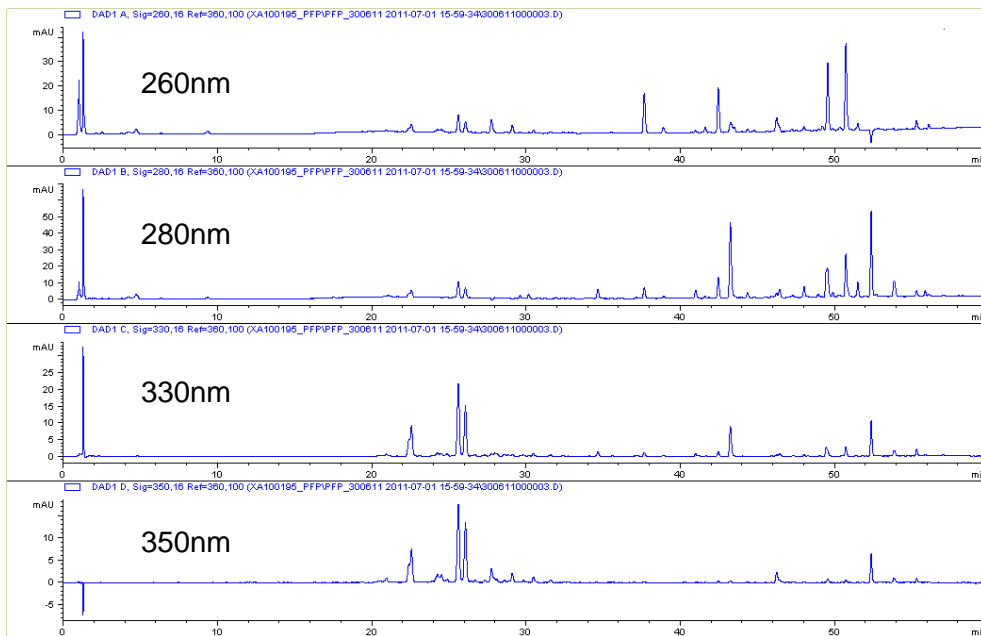
Injection: 100 uL



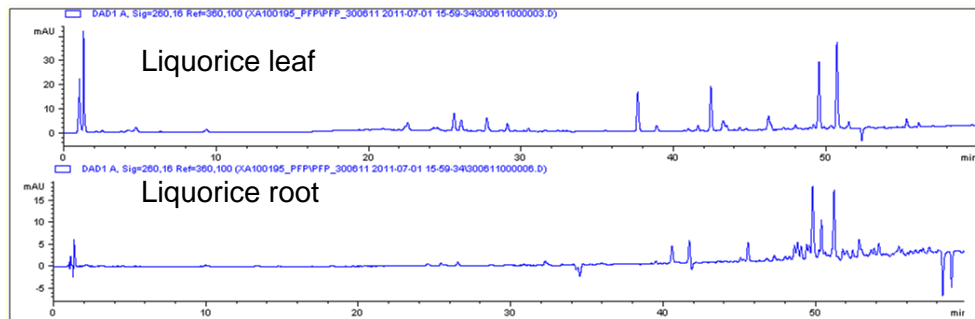


# Fingerprinting of Liquorice Extracts

## Methanolic liquorice leaf extract at different wavelengths



## Comparison of methanolic extracts at 260nm



### Sample preparation

Plant material ground to fine powder in pestle and mortar.  
Powdered material extracted into methanol by ultrasonication for 30 minutes, followed by centrifugal filtration.

### HPLC analysis

ACE 3 C18-PFP, 3µm, 150 x 2.1mm

Gradient analysis

A: Ammonium acetate in water, pH 4

B: Methanol

Time (mins)	%B
0	10
1	10
11	15
55	90
60	100

0

1

11

55

60

Flow rate: 0.4ml/min

Column temperature: 40°C

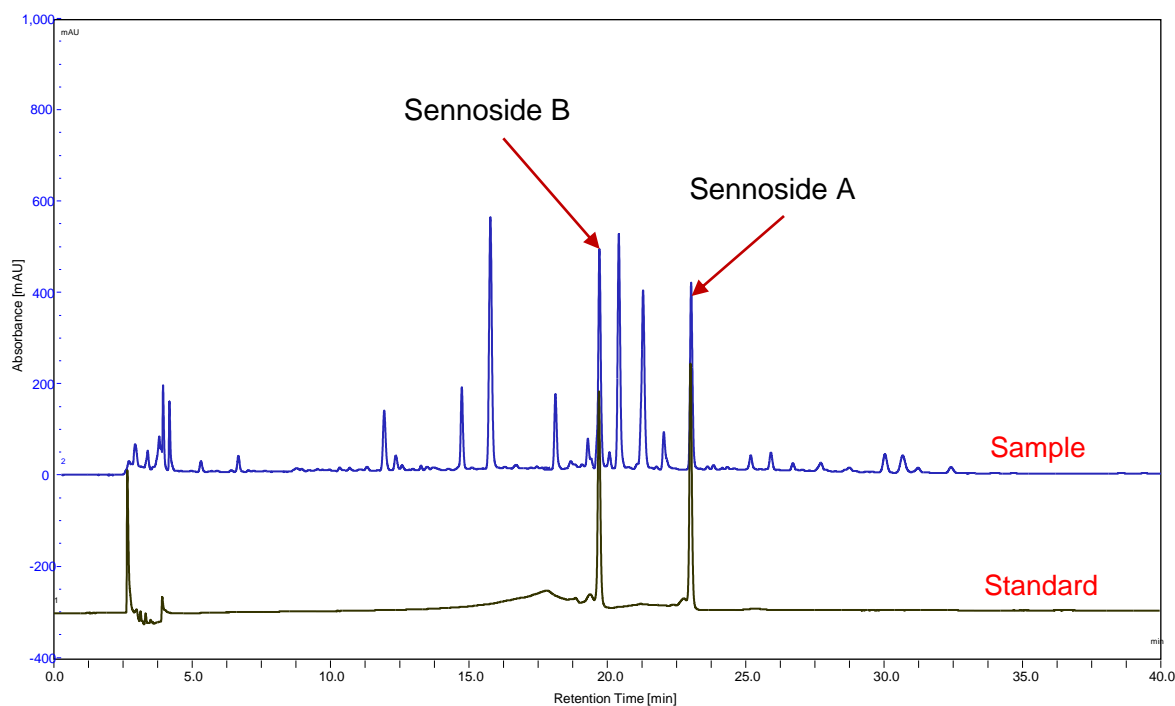
Injection volume: 2µl

Detection: UV at 260, 280, 330 and 350nm



# Sennosides in Traditional Chinese Medicine

Herbal tea bag containing Folium Sennae, Peppermint, Folium Mori, Folium Nelumbinis, Glycyrrhiza Uralensis and Lalang Grass Rhizome



ACE C18-PFP 3 $\mu$ m, 150 x 4.6mm

Gradient analysis

A = 0.75% CH<sub>3</sub>COOH in H<sub>2</sub>O

B = CH<sub>3</sub>CN/CH<sub>3</sub>OH (90:10)

Time (mins) %B

0 9

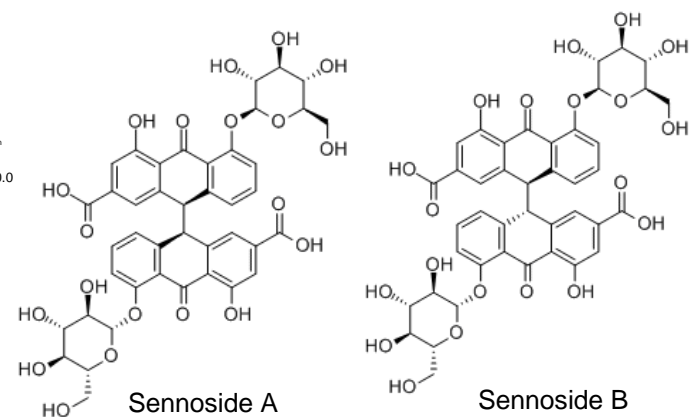
23 28

40 28

Flow rate: 0.6ml/min

Column temperature: 35°C

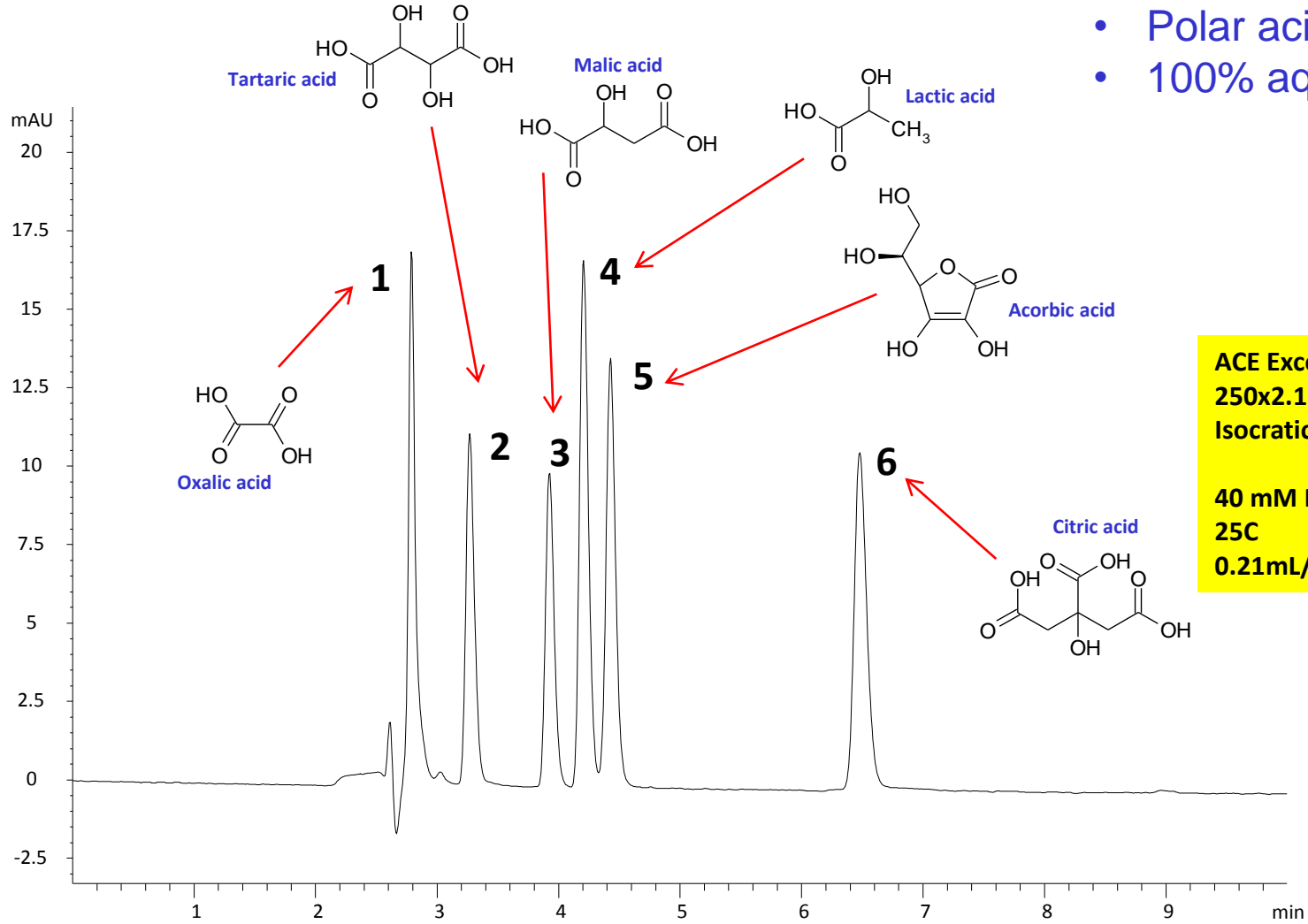
Detection: UV, 271nm





# Beverage Analysis: Wine Acids

- Polar acid resolution
- 100% aqueous eluent



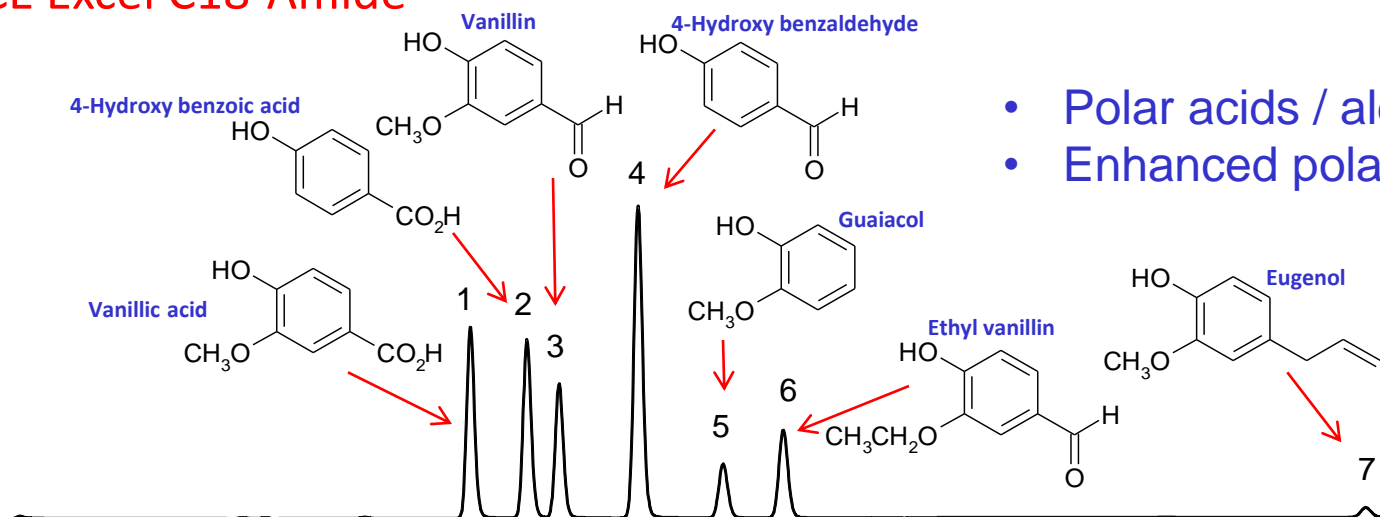
**ACE Excel C18-Amide**  
250x2.1mm, 3µm  
Isocratic analysis

**40 mM NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub>, pH 2.5 (aq)**  
**25C**  
**0.21mL/min**



# Food & Beverage Additives: Vanillins

## ACE Excel C18-Amide

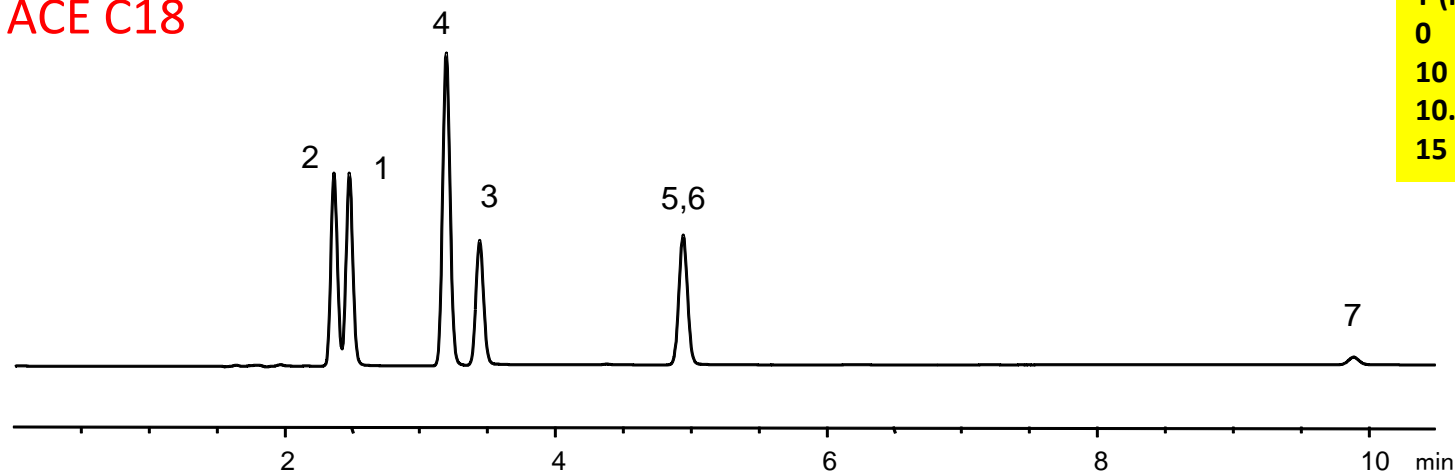


- Polar acids / aldehydes resolution
- Enhanced polar retention

150x4.6mm, 3 $\mu$ m  
Gradient analysis

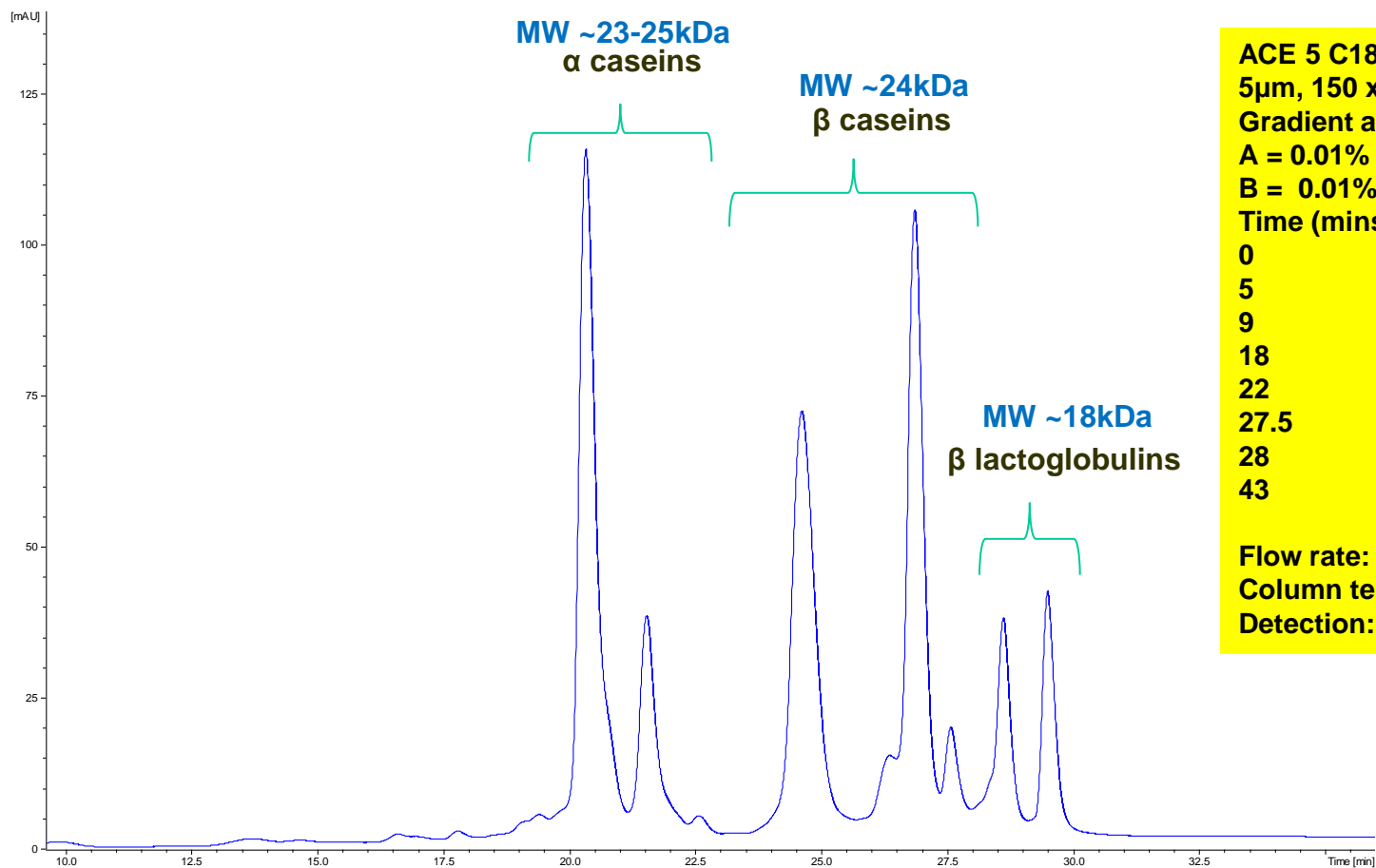
A = 0.1% v/ formic acid (aq)  
B = 0.1% v/v formic acid in MeCN  
40C  
1.0mL/min

## ACE C18



T (min)	%B
0	30
10	55
10.5	30
15	30

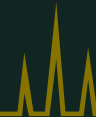
# Separation of Milk Proteins



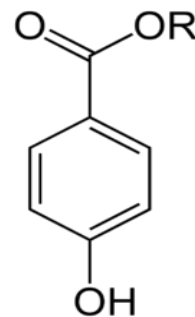
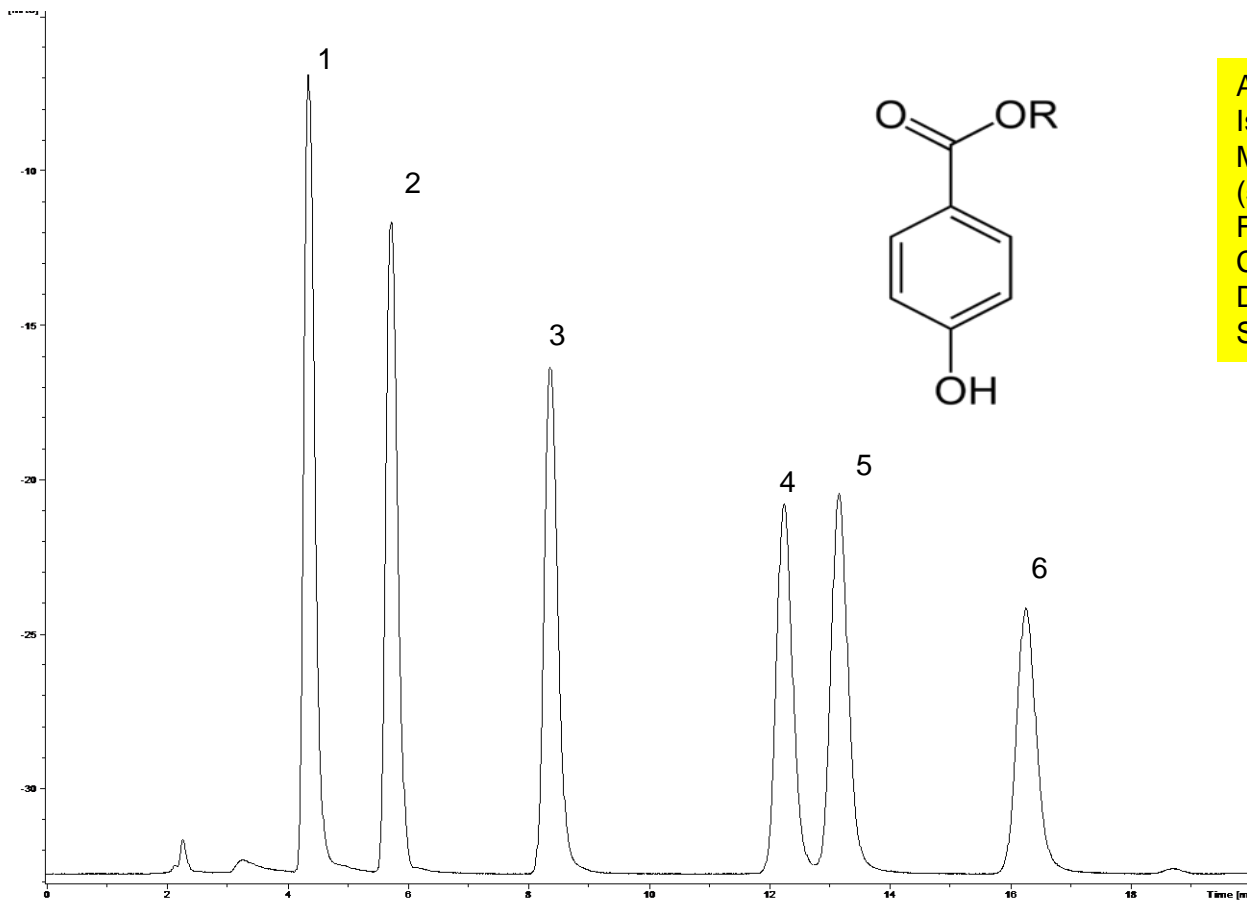
**ACE 5 C18-300**  
**5 $\mu$ m, 150 x 2.1mm**  
**Gradient analysis**  
**A = 0.01% TFA in water**  
**B = 0.01% TFA in acetonitrile**

Time (mins)	%B
0	33
5	33
9	35
18	37
22	40
27.5	41
28	41
43	43

**Flow rate: 0.2ml/min**  
**Column temperature: 45°C**  
**Detection: UV, 214nm**



# Paraben Preservatives



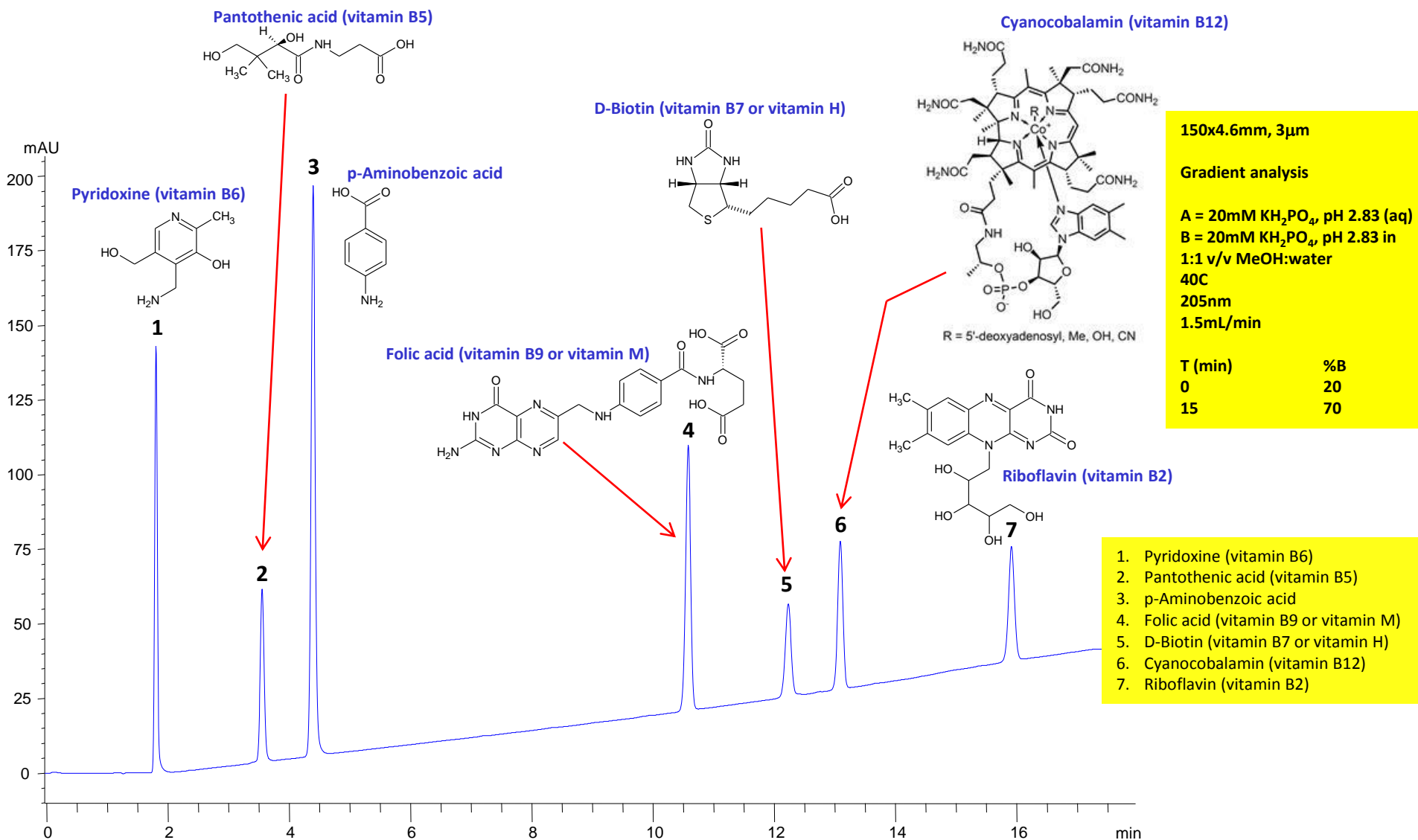
ACE 3 Phenyl, 150 x 2.1mm  
Isocratic  
Methanol – 25mM NH<sub>4</sub>OAc  
(50:50), pH 6.8  
Flow rate: 0.2ml/min  
Column temperature: 40°C  
Detection: UV at 240nm  
Sample volume: 2µl

1. Methylparaben
2. Ethylparaben
3. n-Propylparaben
4. i-Butylparaben
5. n-Butylparaben
6. Benzylparaben

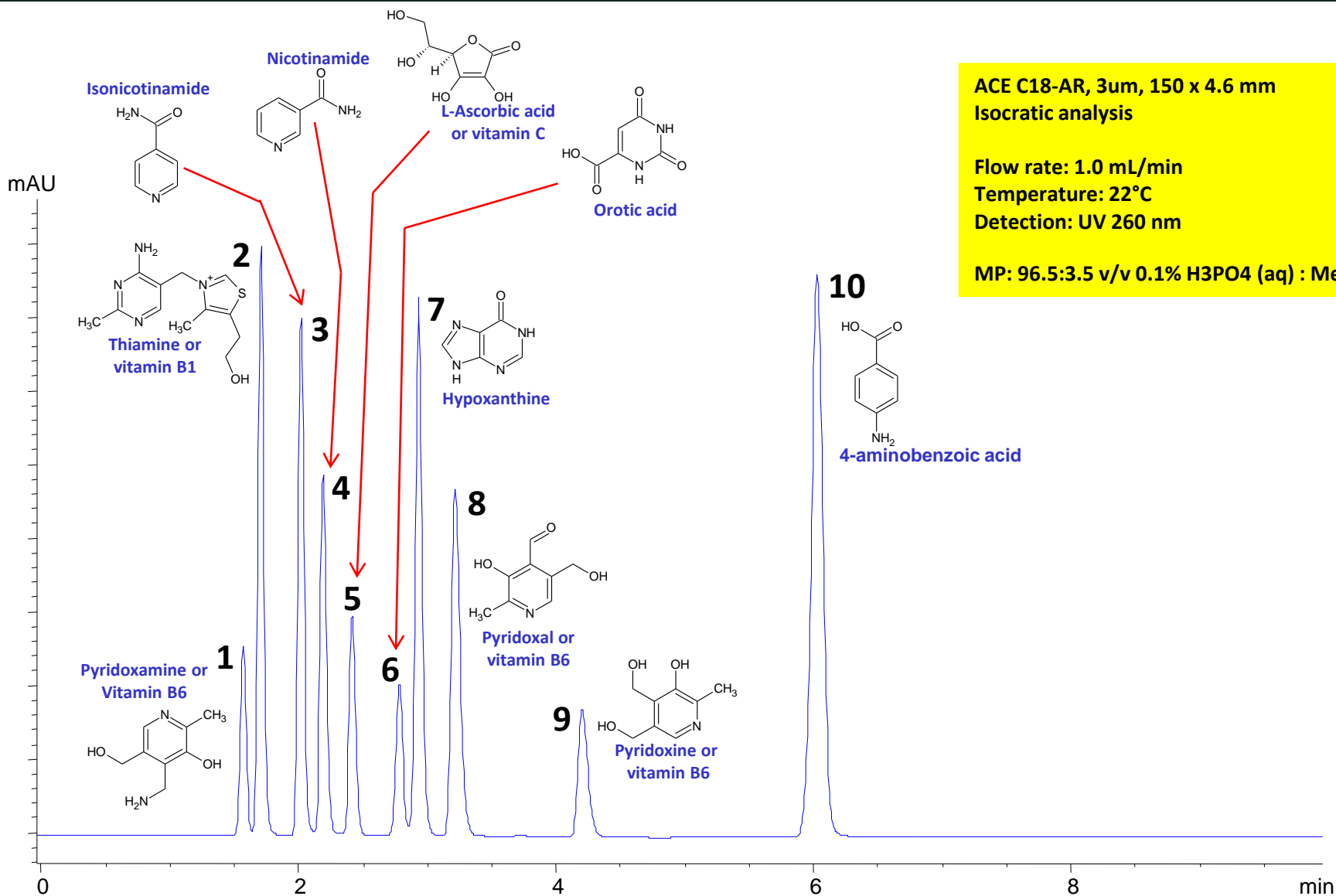


# Water Soluble Vitamins

## ACE 3 C18-AR



# Water Soluble Vitamins / Polar Molecules





# Arsenolipids from Edible Seaweed by LC-ICP-MS and LC-ESI-MS

## Separation of arsenic species from methanolic extract of the edible seaweed *Alaria esculenta*

Arsenic-containing hydrocarbon:

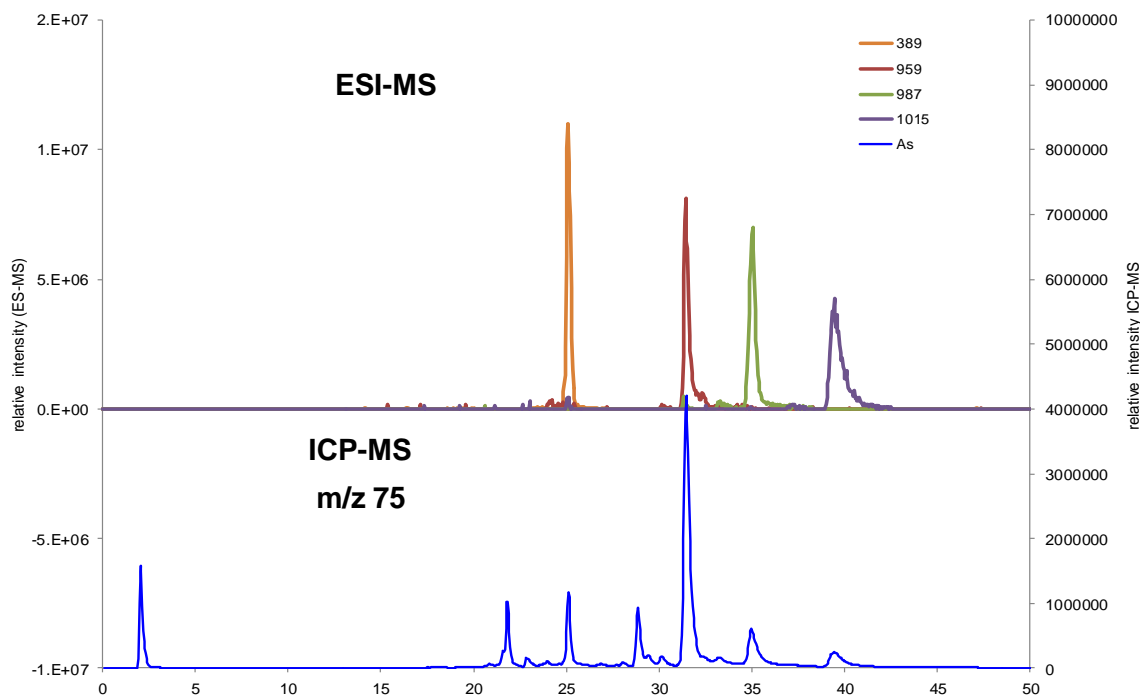
$m/z$  389 [M + H]<sup>+</sup> for C<sub>21</sub>H<sub>46</sub>AsO

Arsenic-containing phospholipids:

$m/z$  959 [M + H]<sup>+</sup> for C<sub>45</sub>H<sub>89</sub>AsO<sub>14</sub>P (C16:0/C16:0)

$m/z$  987 [M + H]<sup>+</sup> for C<sub>47</sub>H<sub>93</sub>AsO<sub>14</sub>P (C18:0/C16:0)

$m/z$  1015 [M + H]<sup>+</sup> for C<sub>49</sub>H<sub>97</sub>AsO<sub>14</sub>P (C20:0/C16:0)



ACE C18, 3 $\mu$ m 150 x 4.6mm

Gradient analysis

A = 0.1% formic acid in H<sub>2</sub>O

B = 0.1% formic acid in CH<sub>3</sub>OH

Time (mins)	%B
0	0
20	100
45	100

Flow rate: 1 ml/min

Injection volume: 100 $\mu$ l

Split ratio: 75% ESI-MS: 25% ICP-MS

Thermo Scientific Element 2 ICP-MS

Mode: Organic mode

Medium resolution

Thermo Scientific Orbitrap Discovery

Positive ESI mode

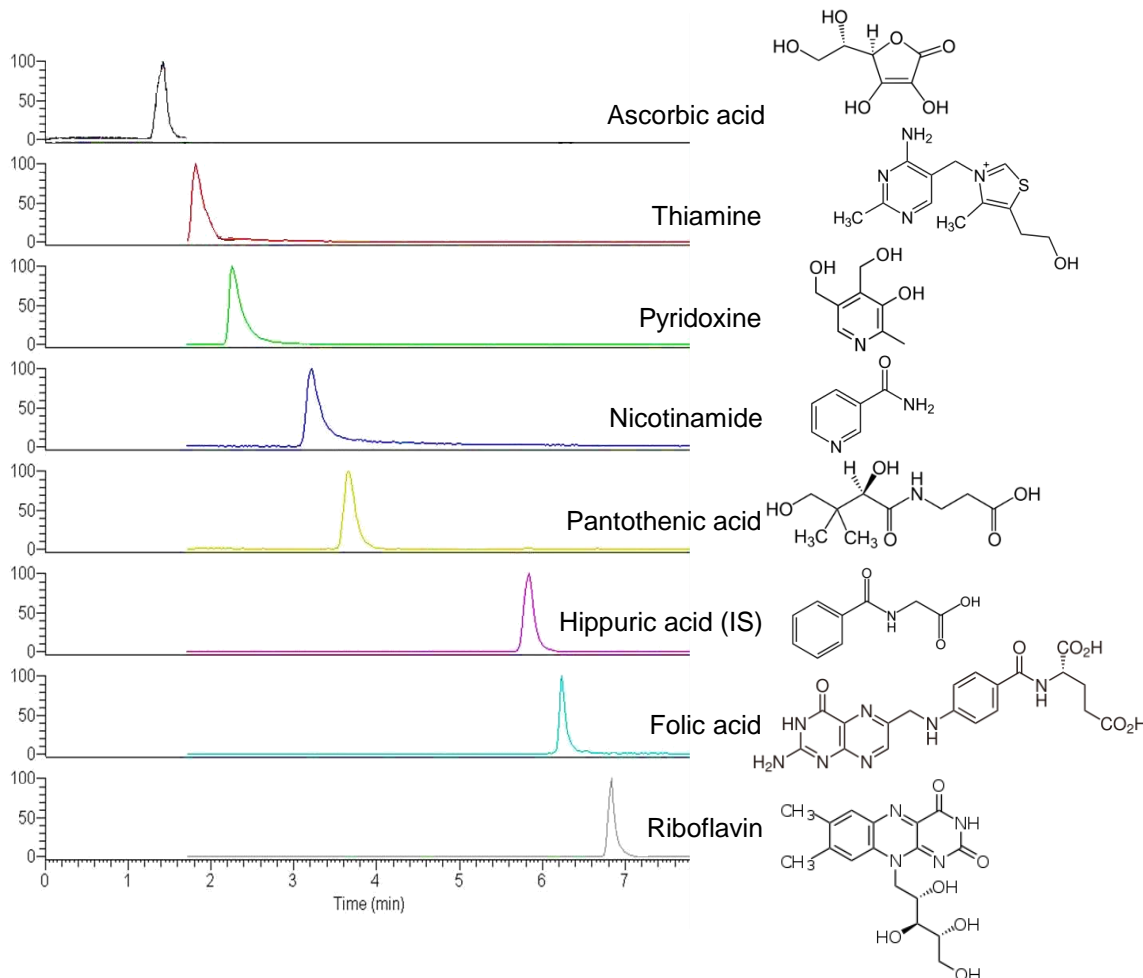
Spray voltage: 4.5kV

Capillary temperature: 320°C

Capillary voltage: 42V



# Water Soluble Vitamins In Green Vegetables By LC-MS/MS



ACE C18, 3 $\mu$ m, 100 x 2.1mm

Gradient analysis

A: 10mM ammonium acetate (aq), pH 4.5

B: 0.1% acetic acid in methanol

C: 0.3% acetic acid in methanol

Time (mins) %A %B %C

0 90 10 0

3 90 10 0

4 50 0 50

7 50 0 50

10 0 100 0

Flow rate: 0.2ml/min

Injection volume: 10 $\mu$ l

Column temperature: 20 $^{\circ}$ C

TSQ triple quad MS; SRM mode

Detection: -ESI for vitamin C

+ESI for B vitamins

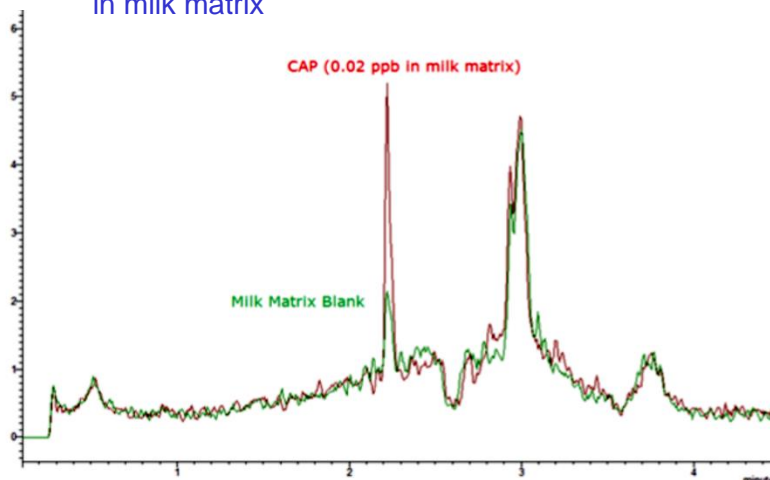
Analyte	Transition m/z	LOQ (ng/mL)
Ascorbic acid (Vit C)	174.9 $\rightarrow$ 115.2	128.3
Thiamine (Vit B1)	265.1 $\rightarrow$ 122.1	2.4
Pyridoxine (Vit B6)	169.9 $\rightarrow$ 152.1	0.6
Nicotinamide (Vit B3)	123.0 $\rightarrow$ 80.3	13.2
Pantothenic acid (Vit B5)	220.0 $\rightarrow$ 202.1	23.3
Folic acid (Vit B9)	442.0 $\rightarrow$ 294.9	1.9
Riboflavin (Vit B2)	377.1 $\rightarrow$ 243.0	0.2
Hippuric acid (IS)	180.1 $\rightarrow$ 105.2	14.9



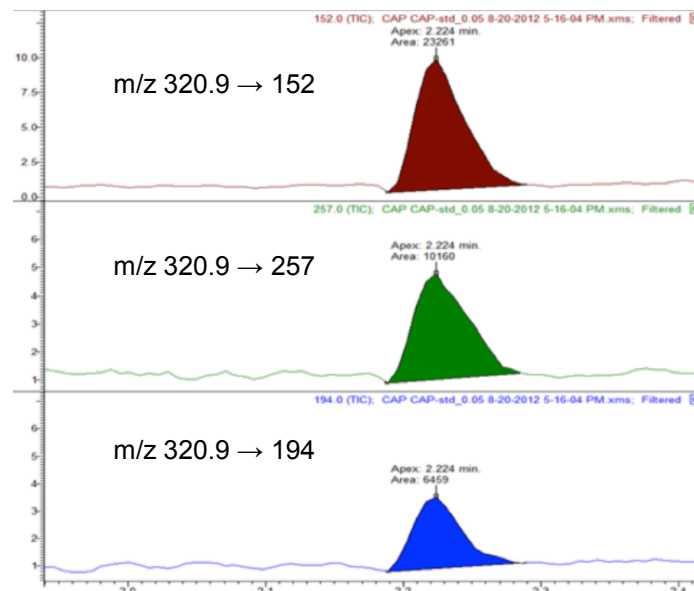


# Chloramphenicol in Milk by LC-MS/MS

TIC of 3 MRMs of 0.02 ppb chloramphenicol spiked in milk matrix



MRM chromatograms of 0.05 ppb chloramphenicol in milk



ACE C18 3 $\mu$ m, 50 x 2.1mm

Gradient analysis

A = Water

B = Methanol

T (mins)	%B	T (mins)	%B
0	10	3.0	95
0.05	10	3.1	95
2.5	95	4.5	10

Flow rate: 0.5ml/min

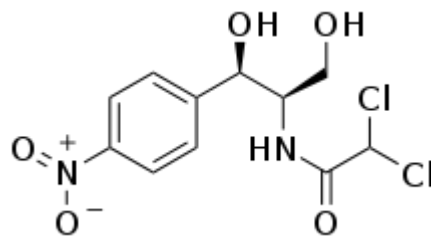
Injection volume: 10 $\mu$ l

Bruker EVOQ Elite triple quad MS

VIP heated-ESI temperature: 400°C

Cone gas temperature: 350°C

Spray voltage: -4500V



Chloramphenicol

LOQ Chloramphenicol = 0.02 ppb

Transitions:

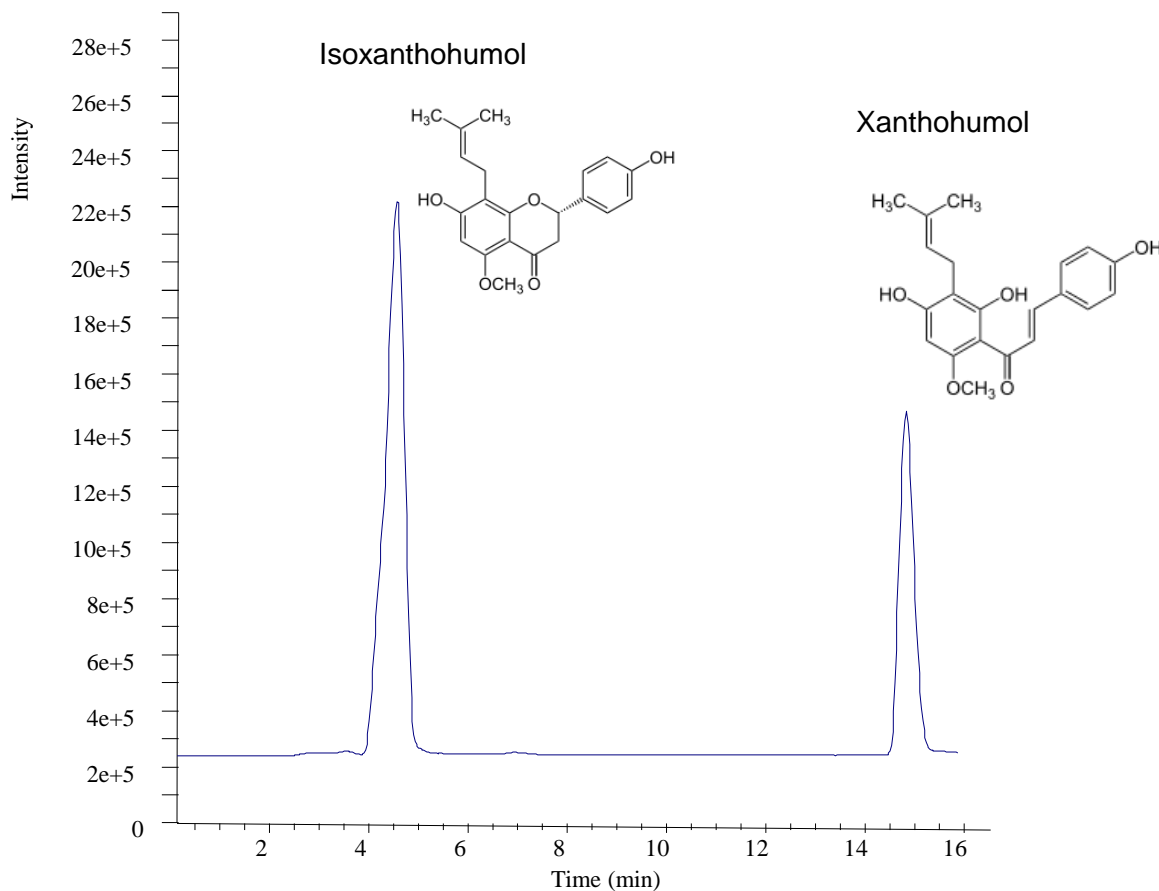
m/z 320.9 → 152 Quantification

m/z 320.9 → 257 Qualification

m/z 320.9 → 194 Qualification



# LC-MS/MS of phytoestrogens from hop extracts



Hop extracts obtained by pressurised liquid extraction using ASE 200 system

ACE C18-AR, 3 $\mu$ m, 150 x 4.6mm  
Gradient analysis

A: 1% formic acid in acetonitrile

B: 1% formic acid in methanol

C: 1% formic acid in water

D: Methanol

Time (mins)	%A	%B	%C	%D
0	56	0	44	0
8	51	5	44	0
10	51	5	44	0
17	95	5	0	0
22	95	0	0	5

Flow rate: 0.6ml/min

TSQ-Quantum triple quad ESI

Spray voltage: -4500V

Precursor ion: 355.4 [M+H]<sup>+</sup>

MRM transition ions: 179 and 299

Collision energy: 28 and 16V

LOQ isoxanthohumol : 0.07 $\mu$ g/ml

LOQ: xanthohumol: 0.01 $\mu$ g/ml