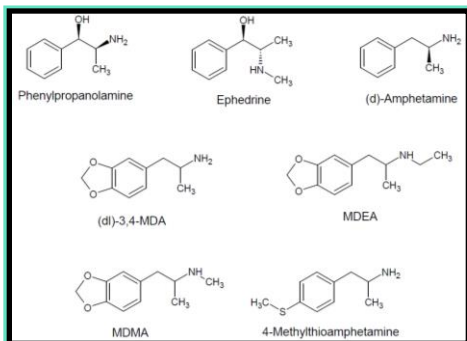
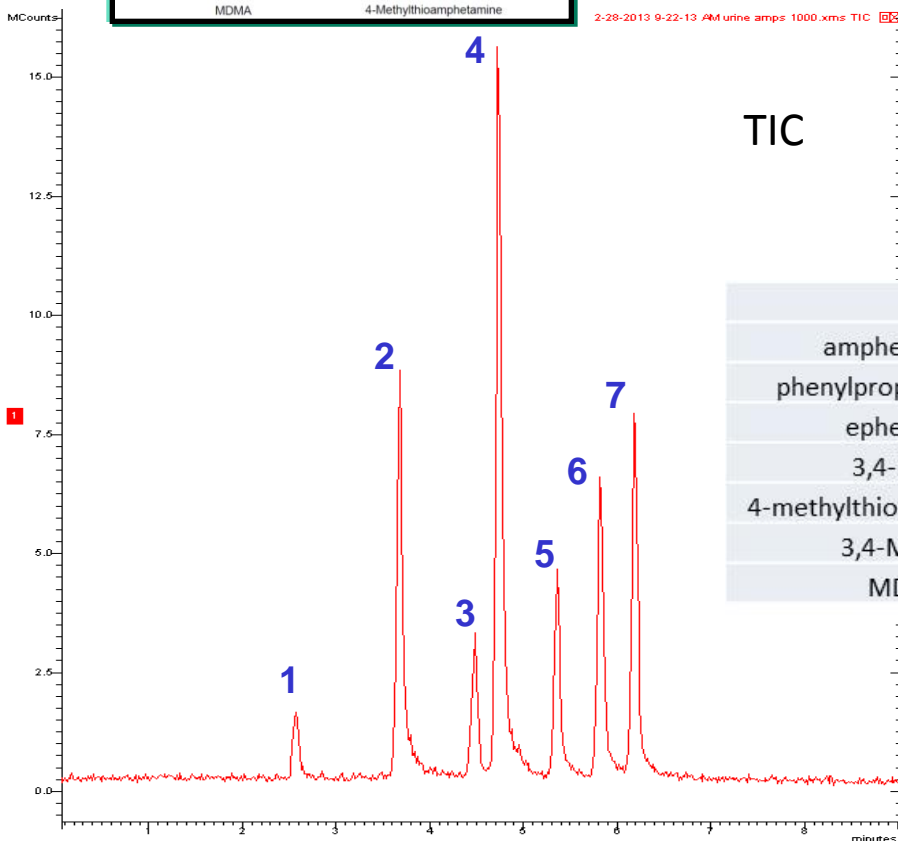




# Amphetamines In Urine by LC-MS/MS



1. Phenylpropanolamine
2. (l)-Ephedrine
3. (dl)-3,4-MDA
4. (d)-Amphetamine
5. (dl)-3,4-MDMA
6. 4-methylthioamphetamine
7. (±)-MDEA



ACE Excel SuperC18, 3um, 75 x 2.1 mm  
Gradient analysis

MP A: 5mM Ammonium Hydroxide, pH 10.8.  
MP B: 5mM Ammonium Hydroxide, pH 10.8  
in 1:9 v/v H<sub>2</sub>O:MeOH.

0.6mL/min

T	%B
0	30
8	95

60C, 2uL.

Varian 320 Triple Quadrupole MS

Electrospray voltage: +5 kV

Inlet capillary voltage: 30 V

CID with argon at 1.5 mTorr; Collision cell  
potential ranges from 5 to 17 V

Drying gas (nitrogen) temperature: 325 C

Nebulizing gas (nitrogen) pressure: 35 psi

Extended Dynamic Range

Compound	Q1 Mass	Q3 Mass
(dl)-3,4-MDMA	193.7	163.0
Phenylpropanolamine	151.6	134.0
(d)-Amphetamine	135.8	90.9
(l)-Ephedrine	166.2	148.0
(dl)-3,4-MDA	179.7	163.0
(±)-MDEA	207.7	163.0
4-methylthioamphetamine	182.2	165.0



# Opiates In Urine by LC-MS/MS

1. Morphine 3-β-D-glucuronide
2. Normorphine
3. Morphine 6-β-D-glucuronide
4. Morphine
5. 6-Acetylmorphine

	LOD (est)
Normorphine	100 ppb
Morphine	20 ppb
6-acetylmorphine	10 ppb
Morphine 3-β-DG	100ppb
Morphine 6-β-DG	100ppb

ACE Excel SuperC18, 3μm, 75 x 2.1 mm + guard  
Gradient analysis

MP A: 5mM Ammonium Hydroxide, pH 10.8.  
MP B: 5mM Ammonium Hydroxide, pH 10.8  
in 1:9 v/v H<sub>2</sub>O:MeOH.

0.6mL/min

T	%B
0	5
5	95

60C, 2μL.

Varian 320 Triple Quadrupole MS

Electrospray voltage: +5 kV

Inlet capillary voltage: 30 V

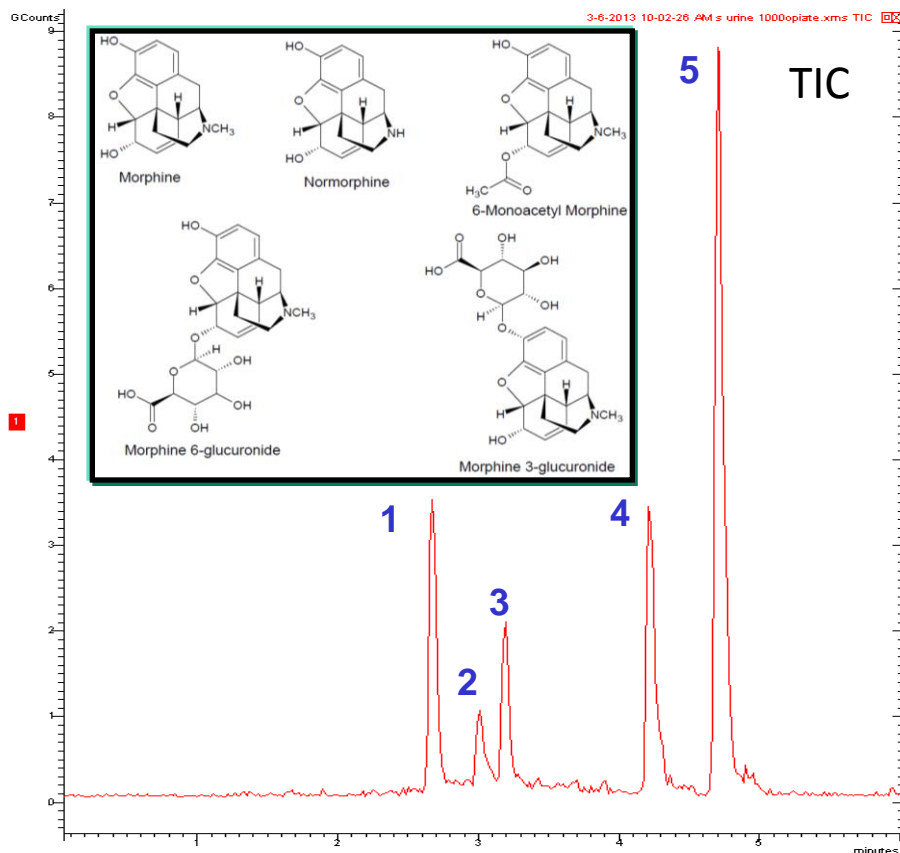
CID with argon at 1.5 mTorr; Collision cell  
potential ranges from 5 to 17 V

Drying gas (nitrogen) temperature: 325 C

Nebulizing gas (nitrogen) pressure: 35 psi

Extended Dynamic Range

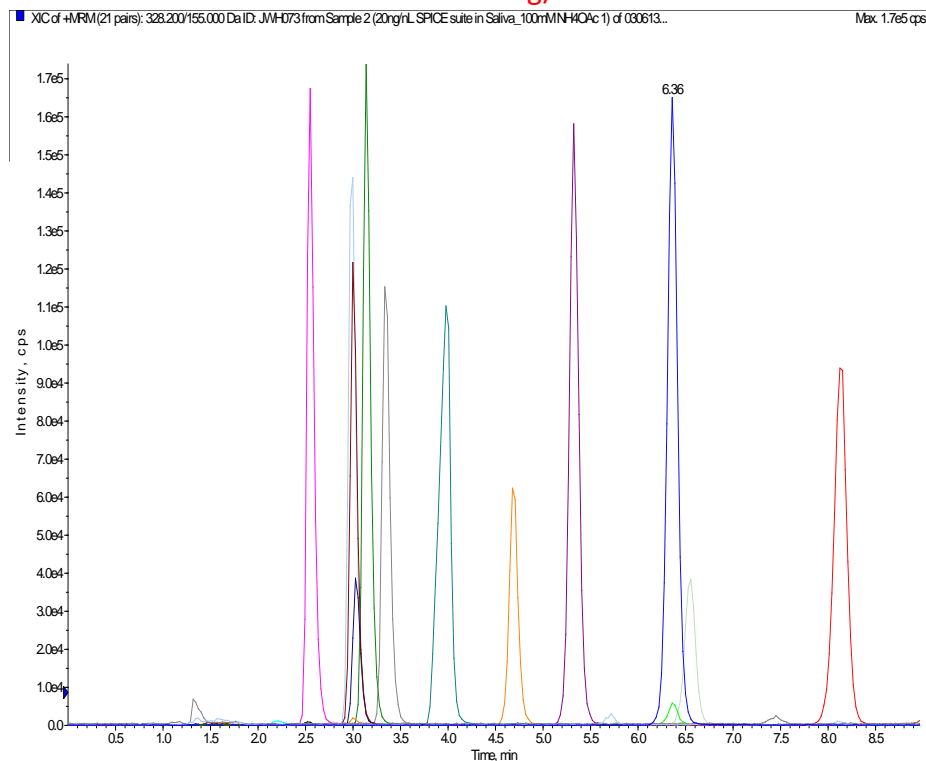
Compound	Q1 Mass	Q3 Mass
morphine 3-β-D glucuronide	462.0	285.9
Normorphine	272.0	165.0
morphine 6-β-D glucuronide	462.0	285.9
6-acetylmorphine	328.0	164.9
morphine	286.0	200.9





# Synthetic Cannabinoids (SPICE) From Oral Fluid

Extracted ion chromatogram for SPICE analytes fortified in neat oral fluid at 20ng/mL



**ACE Excel C18-AR 100x2.1mm, 2 $\mu$ m**

**Isocratic analysis**

**15:85 v/v A:B**

**A = 0.1% v/v formic acid (aq)**

**B = 0.1% v/v formic acid in MeOH**

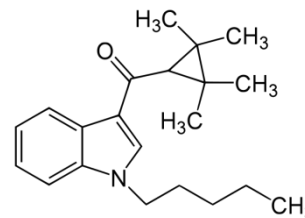
**Ambient**

**0.3mL/min**

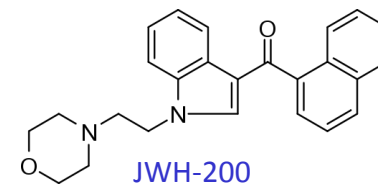
**Applied Biosystems / MDS Sciex 4000 Q-Trap**

**Positive mode Turbo Ionspray®**

Retention Time (minutes)	Analyte	MRM Transition	Declustering Potential (DP)	Collision Energy (CE)	Cell Exit Potential (CXP)
2.55	JWH-250 N-(5-hydroxypentyl)	352>120.9	40	30	16
2.99	JWH-073 N-(3-hydroxybutyl)	344>155	40	30	16
3.00	UR-144 5-Hydroxy-pentyl	328.5>125	30	35	16
3.03	UR-144 Pentanoic Acid	342.5>125	30	35	16
3.14	d5-JWH-018 N-(4-hydroxypentyl)	363.5> 155	40	35	16
3.14	JWH-018 N-(4-hydroxypentyl)	358> 155	40	30	16
3.34	JWH-018 5-pentanoic acid	372>155	40	30	16
3.98	JWH-200	385>155	40	30	16
4.69	XLR-11	330>125	30	35	16
5.32	JWH-250	336>121	40	30	16
6.36	JWH-073	328>155	40	30	16
6.37	UR-144 5-Chloro-pentyl	346.9>125	30	35	16
6.55	UR-144	312.5>125	30	35	16
8.14	JWH-018	342>155	40	30	16

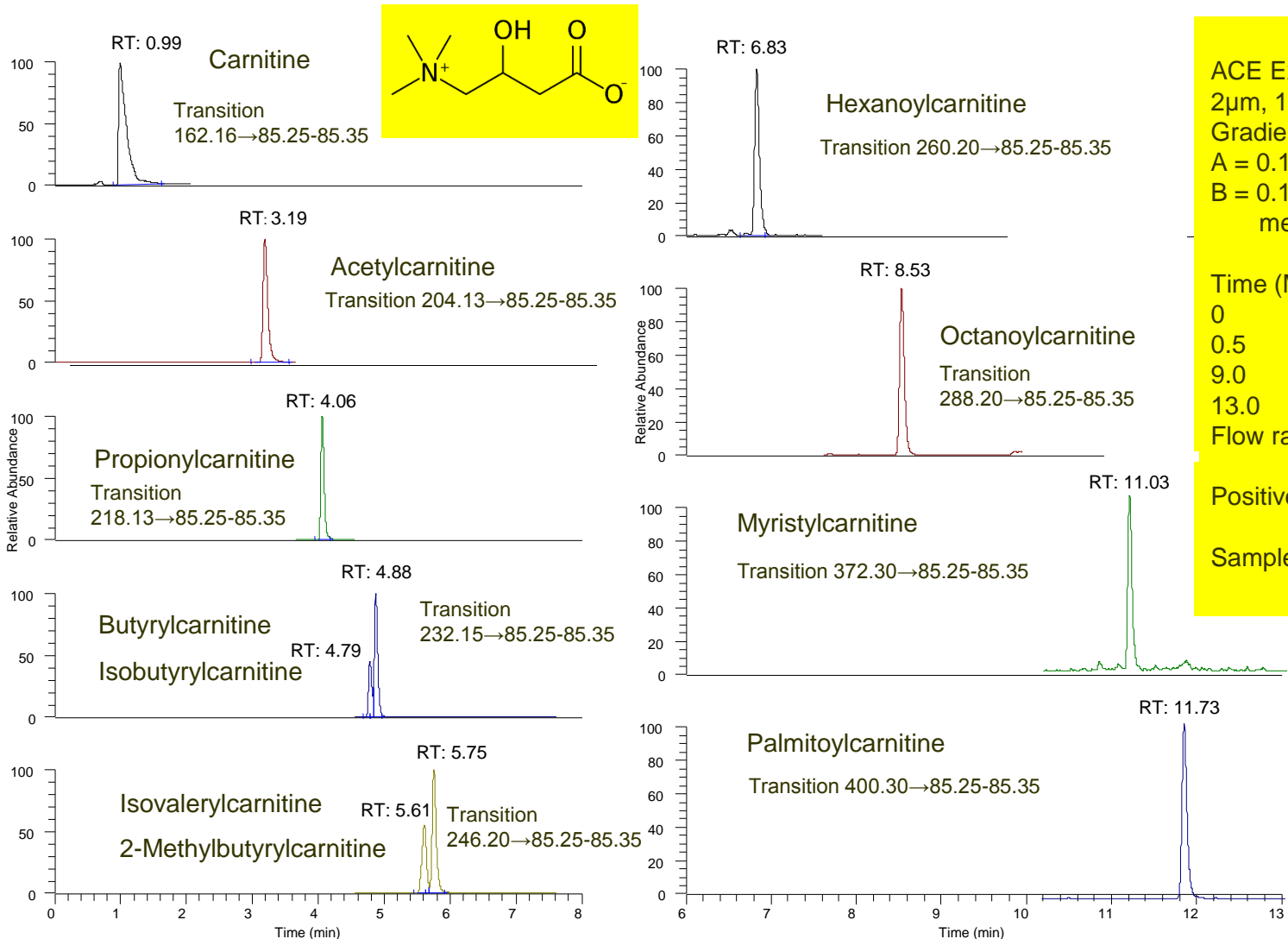


UR-144



JWH-200

# LC-MS/MS of Acylcarnitines



ACE Excel C18-PFP  
2µm, 100 x 2.1mm  
Gradient analysis  
A = 0.1% formic acid in water  
B = 0.1% formic acid in methanol

Time (Mins)	%B
0	0.5
0.5	0.5
9.0	90
13.0	90

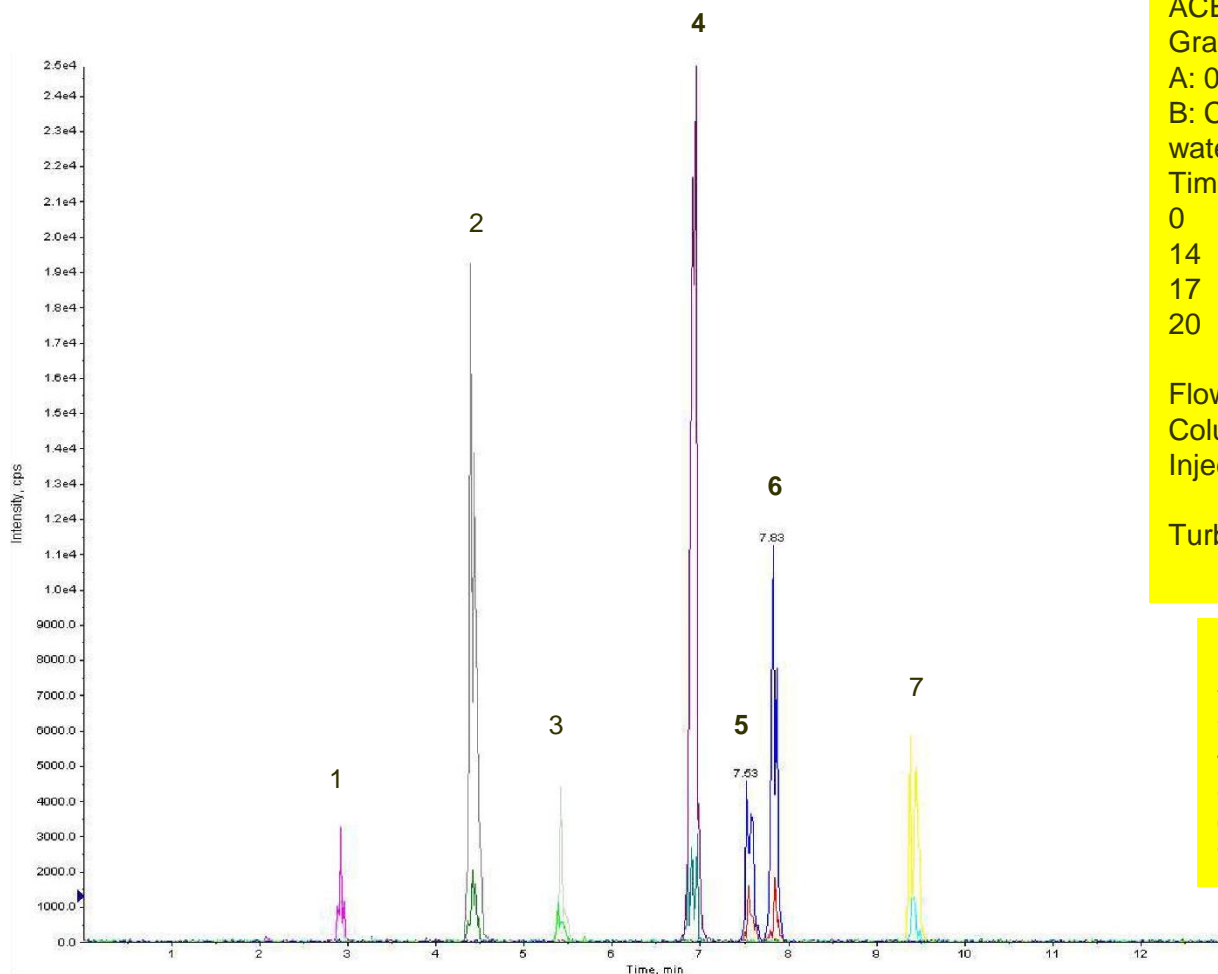
Flow rate: 0.3ml/min

Positive mode ESI

Sample: Dried serum extract



# Corticosteroids by LC-MS/MS



ACE C18-PFP, 3 $\mu$ m, 150 x 2.1mm  
Gradient analysis

A: 0.1% formic acid in water

B: CH<sub>3</sub>CN – 0.1% formic acid in water

Time (mins)	%B
0	30
14	50
17	95
20	30

Flow rate: 0.3ml/min

Column temperature: 15°C

Injection volume: 25 $\mu$ l

Turbospray, MRM

1. Triamcinolone
2. Prednisolone
3. Fluoroprednisolone
4. Methylprednisolone
5. Betamethasone
6. Dexamethasone
7. Flumethasone

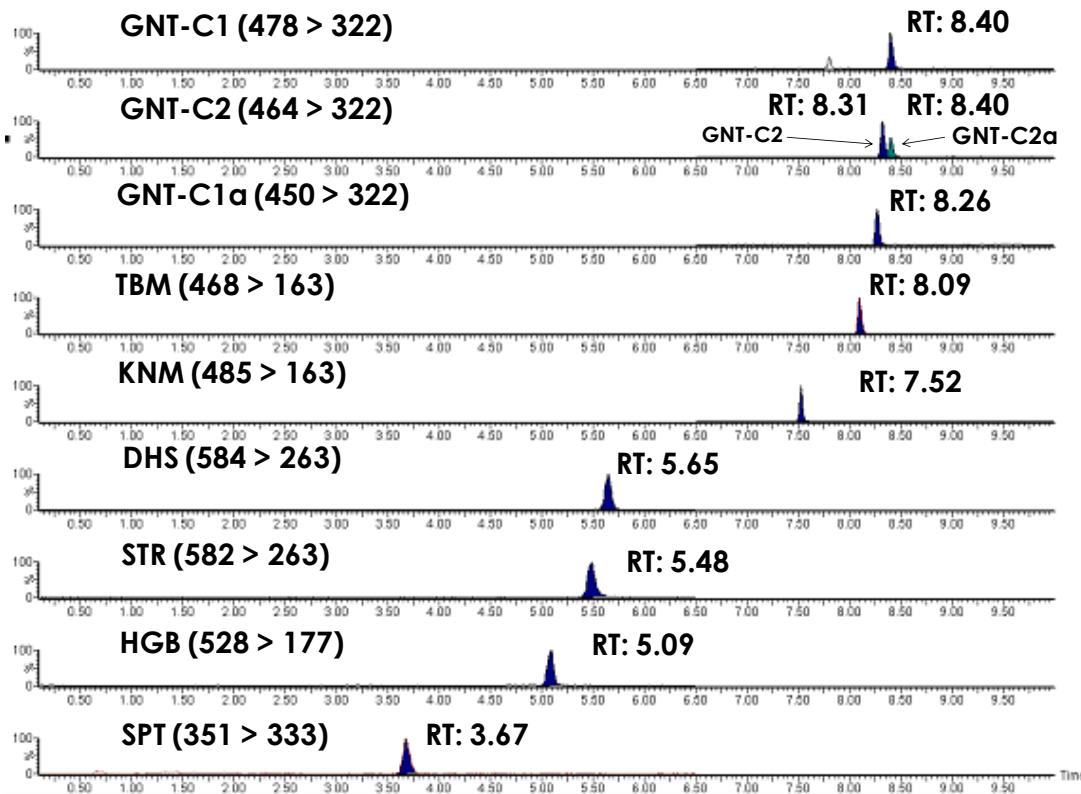


# Aminoglycosides in Eggs

## 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

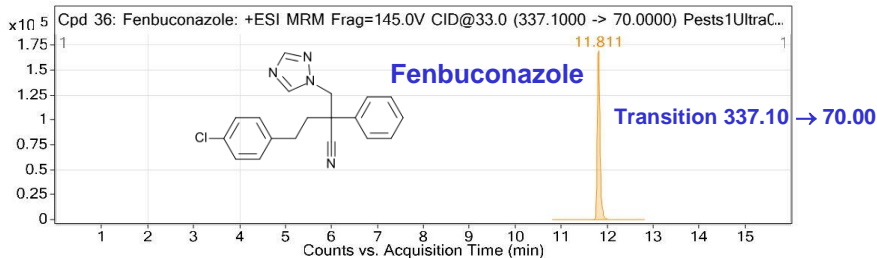
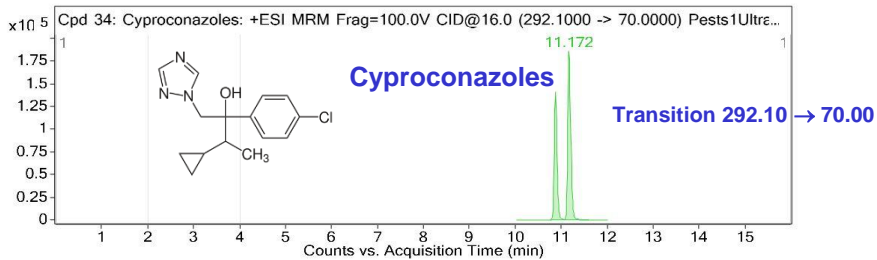
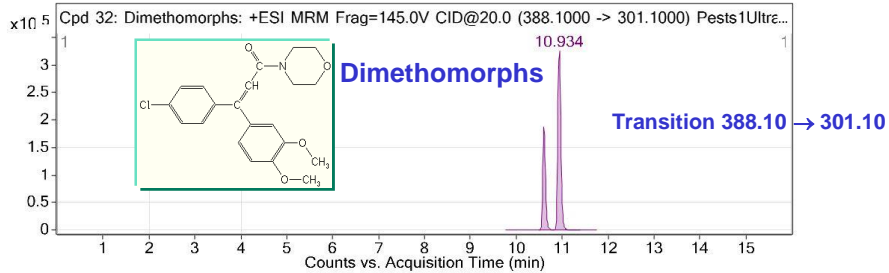
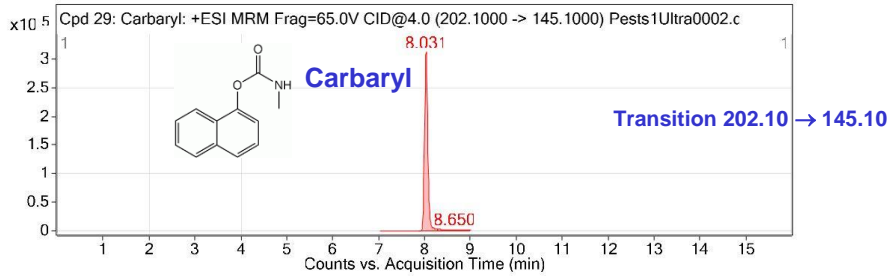
0	5	-
2	15	6
4.5	19	6
5.5	19.5	8
6	22	6
7	35	6
9	48	8
9.5	5	6

Flow rate: 0.4ml/min

Column temperature: 40°C

Positive ESI MRM (transitions as shown)

# Pesticides by LC-MS/MS



Reproduced with permission of Barry Whatmore, Kent Scientific Services

ACE UltraCore SuperC18, 2.5µm, 50 x 2.1mm  
Gradient analysis

A = 0.1% HCOOH + 5mM NH<sub>4</sub>CO<sub>2</sub>H in 9:1 v/v H<sub>2</sub>O: MeOH  
B = 0.1% HCOOH + 5mM NH<sub>4</sub>CO<sub>2</sub>H in 1:9 v/v H<sub>2</sub>O: MeOH

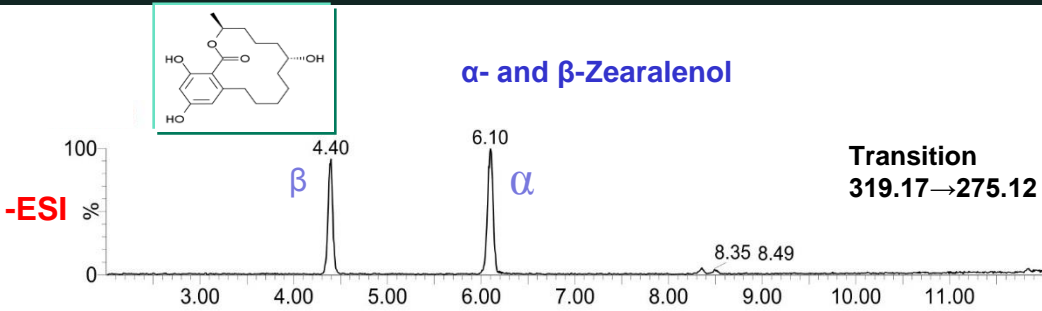
Flow Rate: 0.4ml/min      Gradient conditions  
Temperature: 40°C      Time (mins) 0 1 15 18 18.05 20  
Injection volume: 20µl      %B 0 0 100 100 0 0

Agilent 6420 Triple Quadrupole MS, +ve mode ESI  
Dynamic MRM

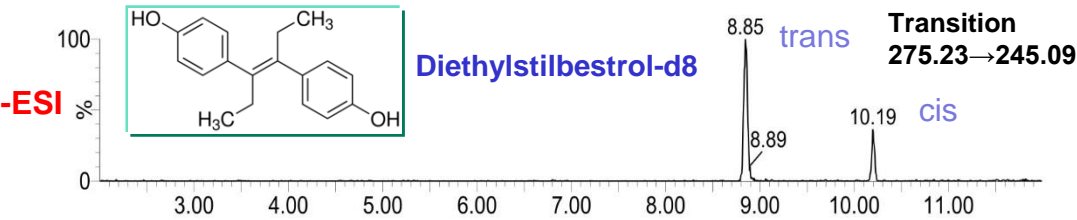
Also analysed under same conditions:

- |                            |                    |
|----------------------------|--------------------|
| Acephate                   | Hexaconazole       |
| Acetamiprid                | Hexaflumuron       |
| Aldicarb                   | Imidacloprid       |
| Aldicarb sulphone          | Indoxacarb         |
| Aldicarb sulphoxide        | Mandipropamid      |
| Benomyl                    | Methamidophos      |
| Carbendazim                | Methomyl           |
| Carbofuran                 | Monocrotophos      |
| Clofentezine               | Nicotine           |
| Clothianidin               | Omethoate          |
| Cyfluthrin                 | Oxamyl             |
| Demeton S-methylsulphone   | Pencycuron         |
| Demeton S-methylsulphoxide | Prochloraz         |
| Dicrotophos                | Propargite         |
| Dimethoate                 | Thiabendazole      |
| Dinotefuran                | Thiacloprid        |
| DMA                        | Thiamethoxam       |
| DMPF                       | Thiodicarb         |
| Flubendiamide              | Thiophanate methyl |
| Folpet                     | Triforine          |
| Formetanate                |                    |

# Veterinary Steroids by LC-MS/MS



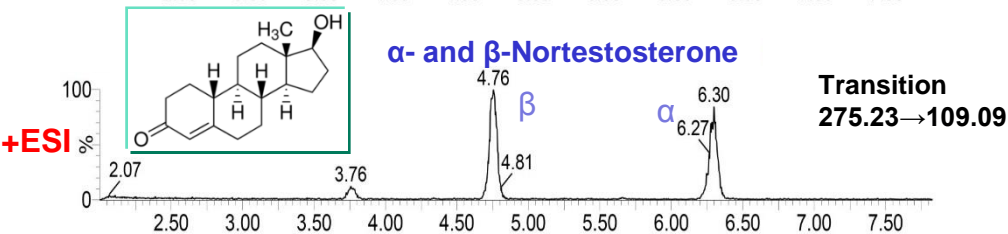
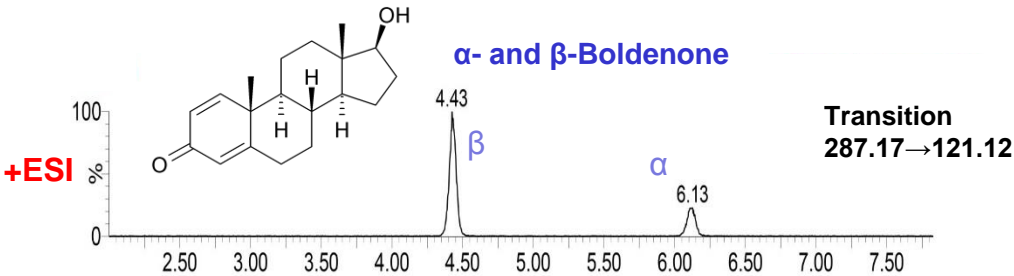
Also analysed in the same run (-ESI):  
Talaranol and zeranol-d4  
Talaranol and zeranol  
Zearalenone  
Hexestrol  
Diethylstilbestrol  
Dienestrol



ACE UltraCore SuperC18, 2.5μm, 100 x 2.1mm  
Waters Acquity SDS system  
Gradient analysis  
A = 0.01mM amm. fluoride + 0.001% formic acid  
B = Acetonitrile

Time (mins)	%B	Time (mins)	%B
0	25	7.5	35
0.5	25	10.5	60
7.0	35		

Flow rate: 0.5ml/min  
Column temperature: 45°C  
Positive or negative ESI MRM data

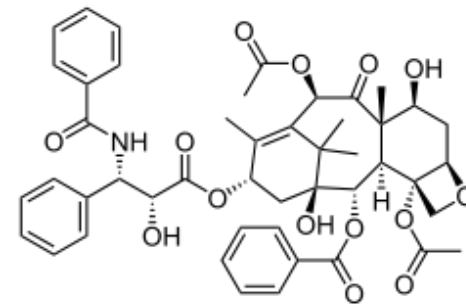
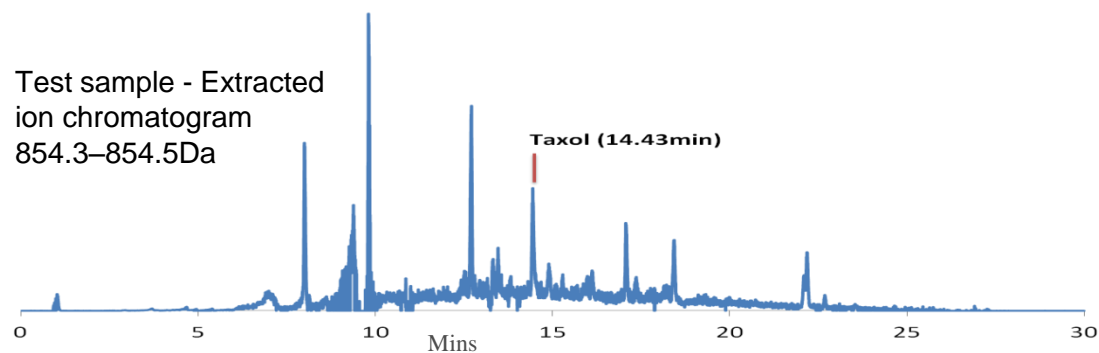
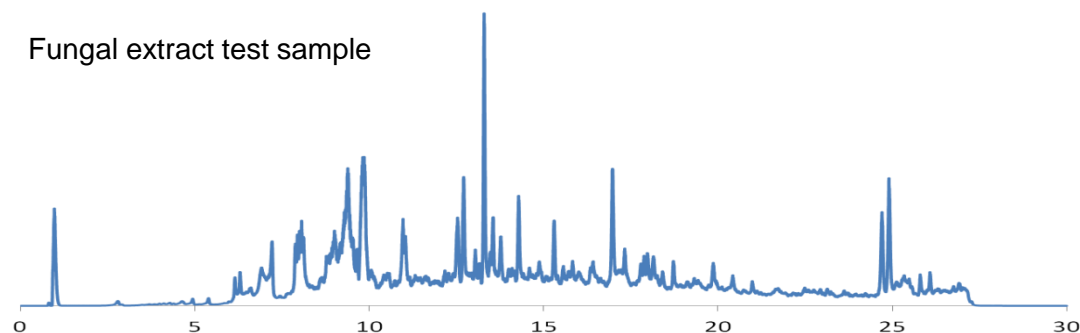
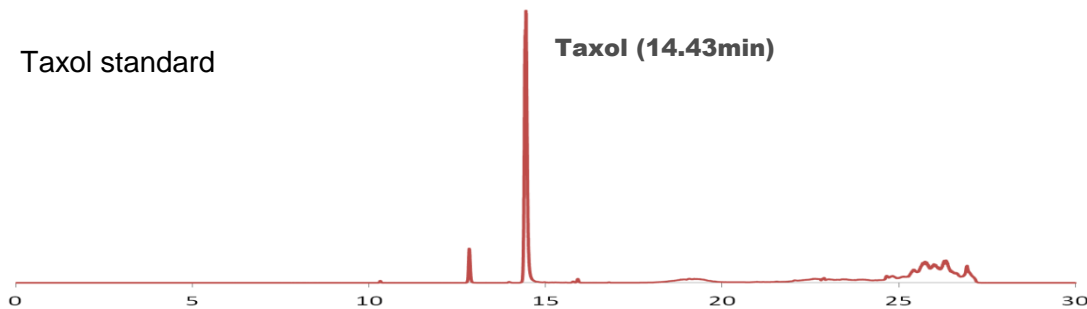


Also analysed in the same run (+ESI):  
Hydroxystanazolol  
Hydroxystanazolol-d3  
Methyltestosterone  
Methyltestosterone-d3  
β-Nortestosterone-d3  
β-Trenbolone  
α-Trenbolone





# Taxol in Fungal Extract by LC-MS/MS



Taxol (Paclitaxel)

**ACE UltraCore SuperC18,  
2.5 $\mu$ m, 150 x 2.1 mm**

**Gradient analysis**

**A = 0.5% formic acid in water**

**B = 0.5% formic acid in acetonitrile**

Time (mins)	%B
0	10
1	10
3	40
22	60
25	95

**Flow rate: 0.35ml/min**

**Orbitrap Elite MS**

**FT positive ion mode**

**Collision induced dissociation –  
Isolation width 5Da**

**Normalised collision energy 32eV**

**Activation Q 0.25**

**Activation time 10ms**



# Microbial Extract by LC-MS

Dionex 3000RS UHPLC system coupled with  
Bruker MaXis Q-TOF MS

UltraCore SuperC18 2.5 $\mu$ m, 150 x 2.1mm

Gradient analysis

A = 0.1% formic acid in water

B = 0.1% formic acid in acetonitrile

Time (mins)	%B	Time (mins)	%B
0	5	20	100
5	5	25	100

Flow rate: 0.2ml/min

Electrospray MS (positive mode)

Source: End plate offset: -500V,

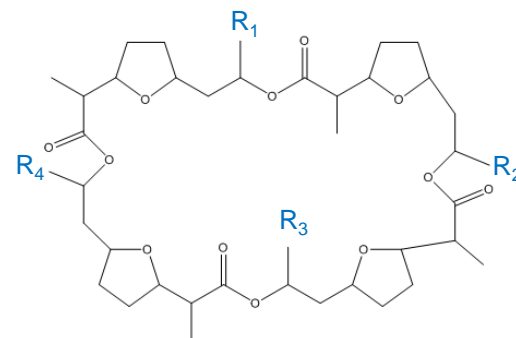
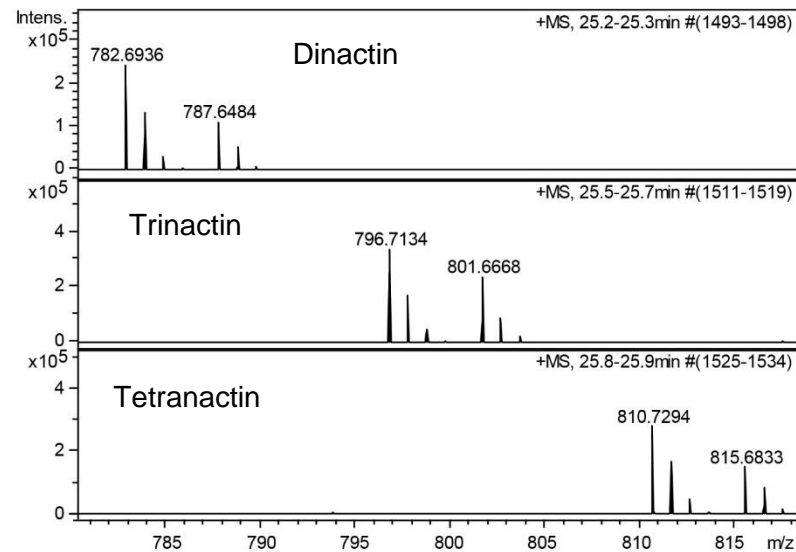
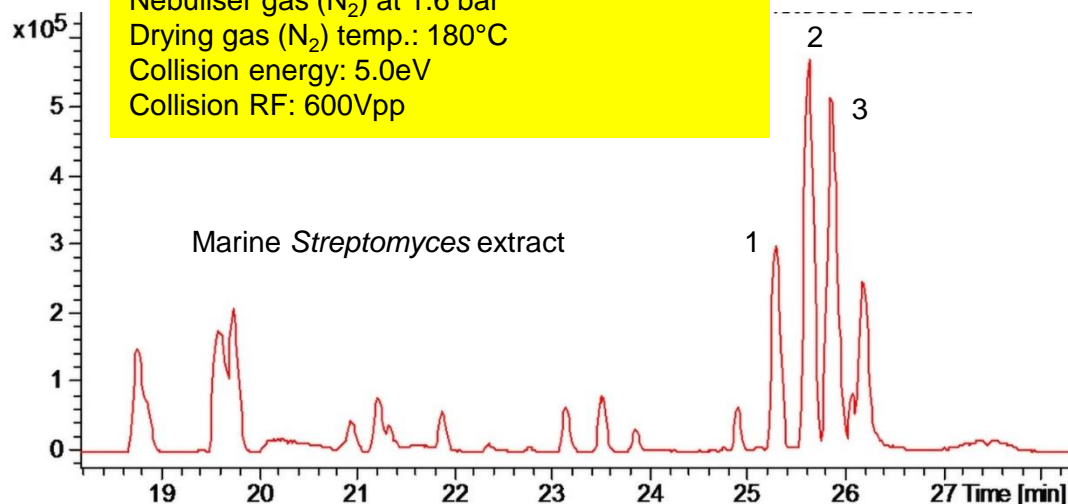
Capillary: -4500V

Nebuliser gas (N<sub>2</sub>) at 1.6 bar

Drying gas (N<sub>2</sub>) temp.: 180°C

Collision energy: 5.0eV

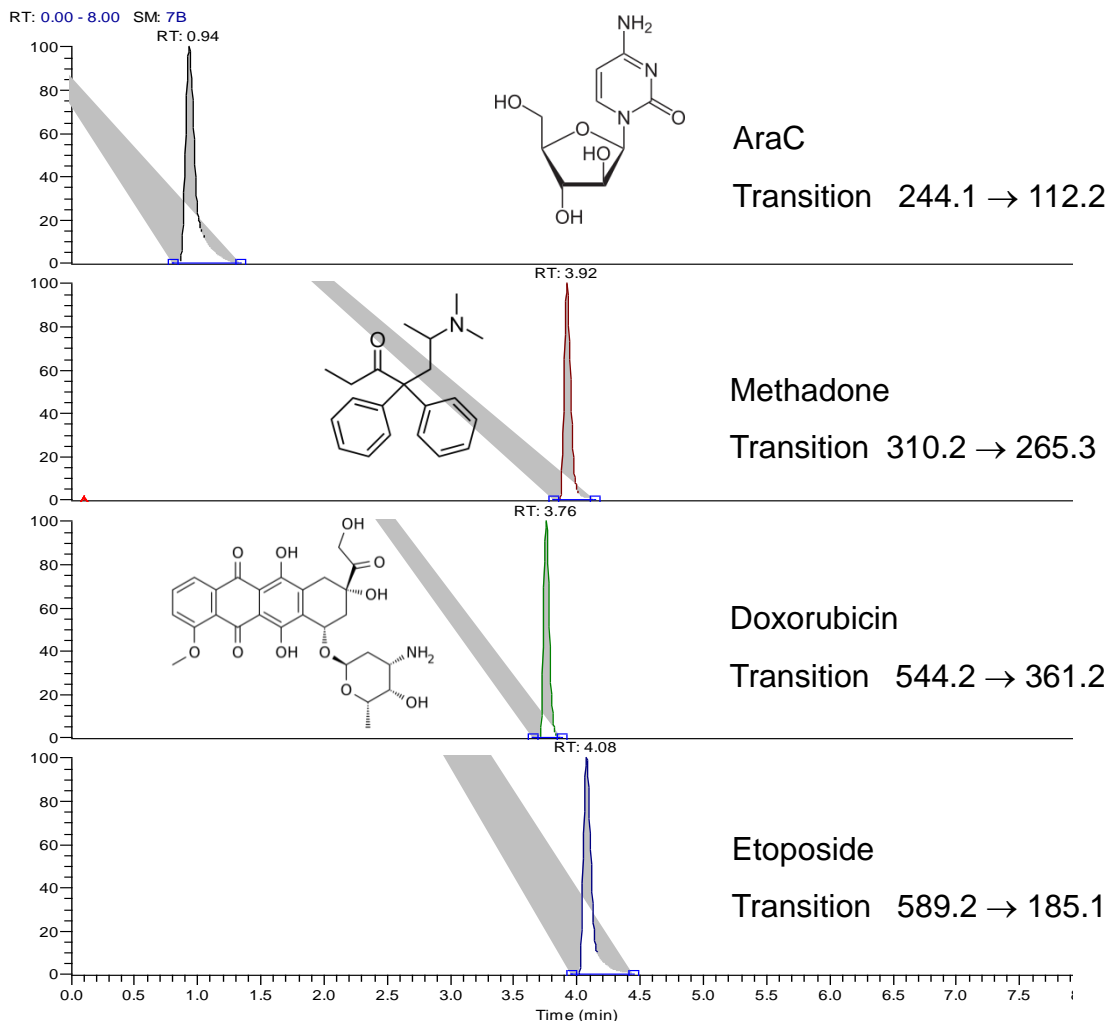
Collision RF: 600Vpp



Macrotetralides

1. Dinactin  $R_1 = R_3 = \text{CH}_2\text{CH}_3$ ,  $R_2 = R_4 = \text{CH}_3$
2. Trinactin  $R_1 = R_2 = R_3 = \text{CH}_2\text{CH}_3$ ,  $R_4 = \text{CH}_3$
3. Tetractin  $R_1 = R_2 = R_3 = R_4 = \text{CH}_2\text{CH}_3$

# Cytotoxic Agents by UHPLC-MS/MS



## Thermo Scientific Accela UHPLC

ACE UltraCore SuperC18, 2.5 $\mu$ m, 100 x 2.1mm

### Gradient analysis

A = 0.1% formic acid in water

B = 0.1% formic acid in acetonitrile

Time (mins)	%B
0	2
1	2
3	80
5	80
5.1	2
8	2

Flow rate: 0.25ml/min

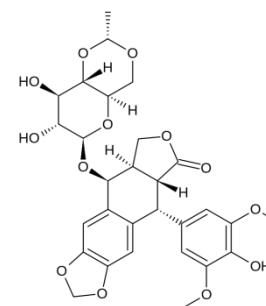
## Thermo Vantage triple quadrupole MS

MRM +ve ESI mode

Spray voltage: 3500V

Nitrogen sheath and auxiliary gas

CID with argon at 1.5 mTorr



# Peptides in saliva

Sample preparation: SPE on C18

ACE UltraCore SuperC18  
2.5µm, 50 x 3mm

Gradient analysis  
A: 0.1% formic acid in water  
B: 0.1% formic acid in CH<sub>3</sub>CN

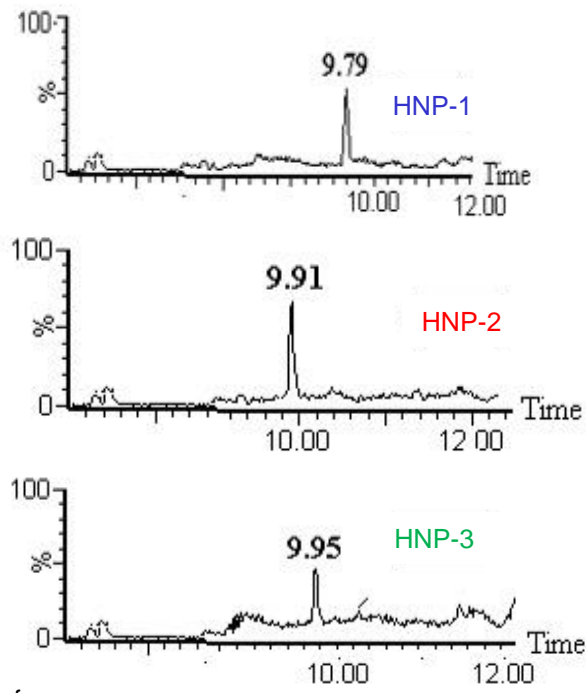
Time (mins)	%B
0	2
2	2
17	50
19	95
20	95

Flow rate: 0.6ml/min

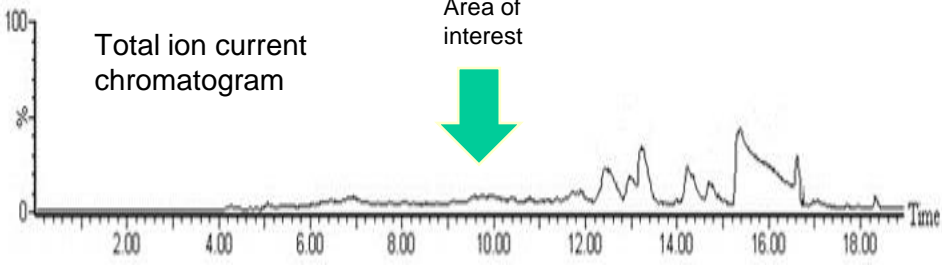
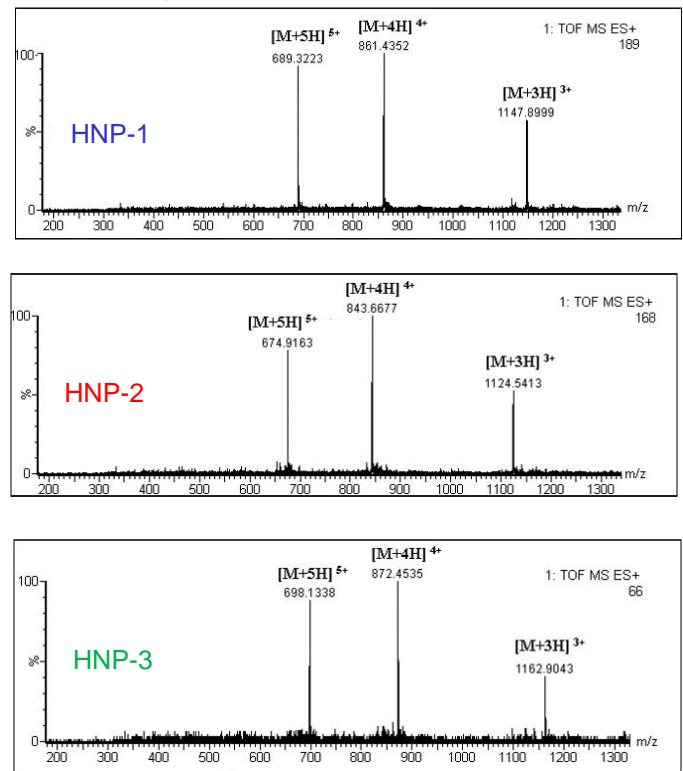
Synapt G1 QToF +ESI MS  
Sampling cone voltage: 40V  
Source temperature: 150°C  
Capillary voltages: 4.8kV  
Extraction cone voltages: 41kV  
Desolvation temperature: 500°C  
Acquisition: 100-2000 m/z

Extracted ion current chromatograms

(sum of multiply protonated ions [M+3H]<sup>3+</sup>, [M+4H]<sup>4+</sup> and [M+5H]<sup>5+</sup>)



Mass spectra

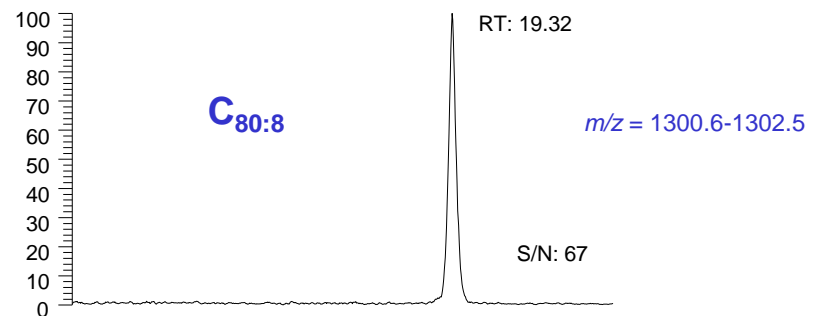


Defensin Human Neutrophil Peptides

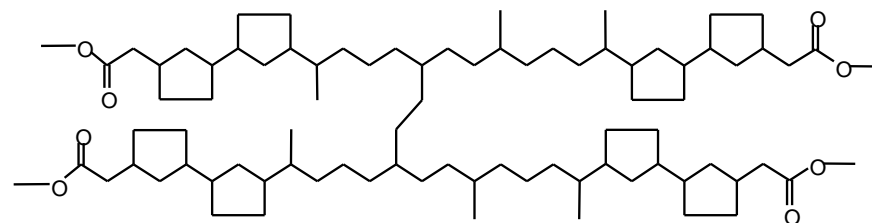
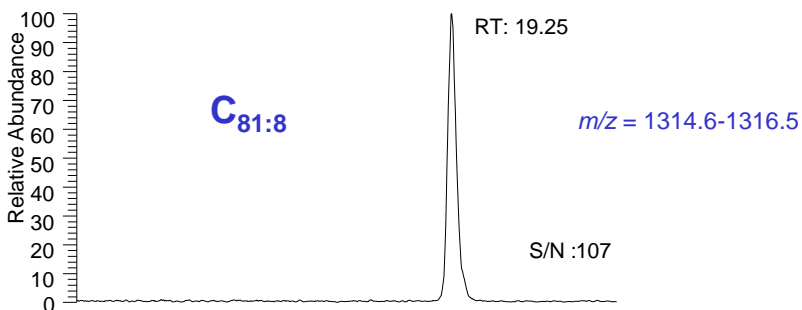
- HNP-1 30 amino acid residues
- HNP-2 29 amino acid residues
- HNP-3 30 amino acid residues

Detection limit ~ 2.1ng/µl

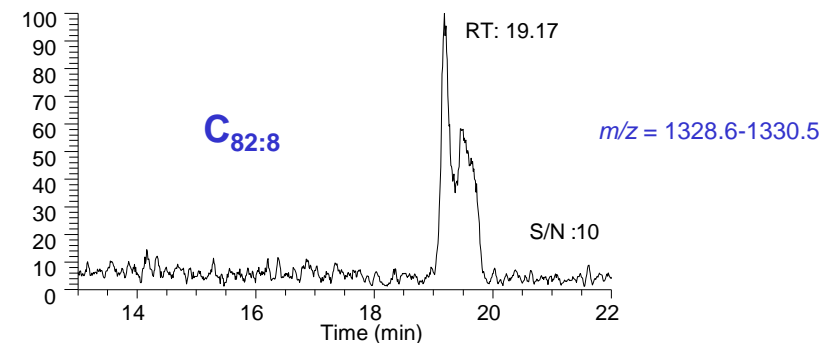
# Polycyclic Tetracarboxylic Acids



C80-82 polycyclic tetracarboxylic acids isolated from oilfield deposits



**Tetramethyl ester of C<sub>80:8</sub> ring acid**



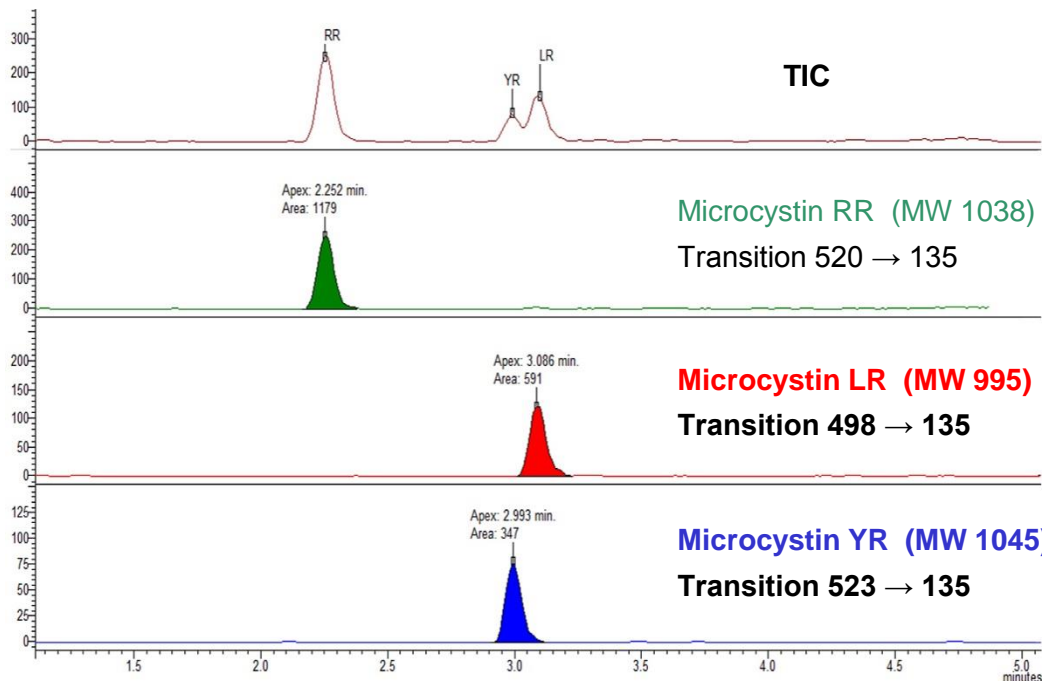
ACE UltraCore SuperPhenylHexyl 2.5µm, 100 x 2.1mm  
Gradient analysis  
A: CH<sub>3</sub>OH – H<sub>2</sub>O (98:2) containing 10mM ammonium acetate  
B: IPA-H<sub>2</sub>O (98:2) containing 10mM ammonium acetate

T (mins)	%B	T (mins)	%B
0	0	15	100
1	0	25	100

Flow rate: 0.15ml/min  
Column temperature: Ambient  
Injection volume: 5µl  
LCQ Ion trap MS  
LC-ESI-MS extracted ion chromatograms  
Compounds detected as ammoniated quasimolecular ions  
[M+NH<sub>4</sub>]<sup>+</sup>  
Detection limit ~ 0.1ppm

# Microcystins From Blue/Green Algae In Drinking Water

ACE Excel 2  $\mu\text{m}$  C18, 100 x 2.1 mm



0.05 ppb each

Variants	R	L
MC-LR	Leucine	Arginine
MC-RR	Arginine	Arginine
MC-YR	Tyrosine	Arginine

Bruker Advance UHPLC system  
ACE Excel 2  $\mu\text{m}$  C18, 100 x 2.1mm  
Gradient elution  
A = 0.1% formic acid in water  
B = Acetonitrile

T (mins)	%B	T (mins)	%B
0	30	7.1	30
1	30	10	30
7	95		

Flow rate: 0.4mL/min

Column temperature: 40°C

Injection volume: 50  $\mu\text{L}$

Concentration each microcystin: 0.05ppb

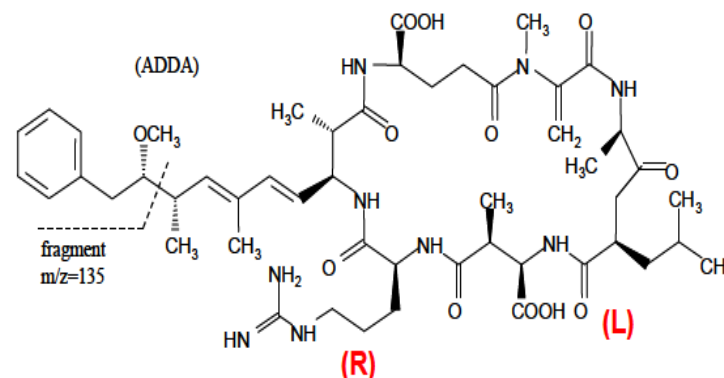
Bruker EVOQ Elite triple quad MS

VIP heated-ESI temperature: 350°C

Cone gas temperature: 200°C

Spray voltage: 4500V (+)

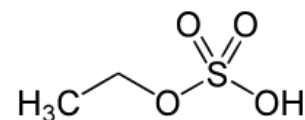
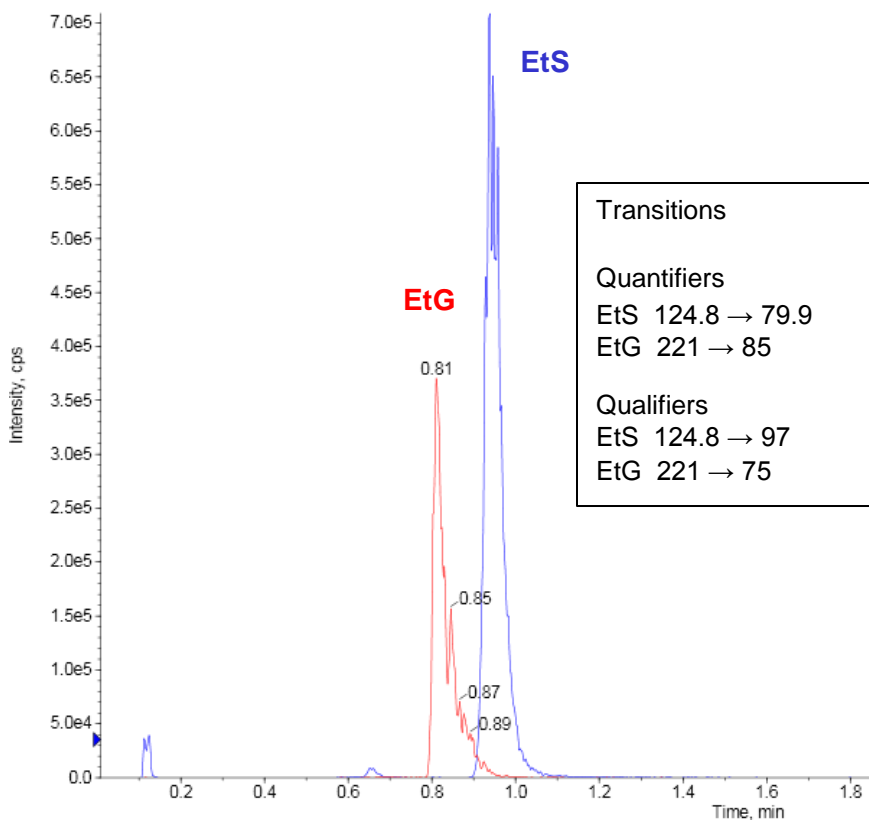
Collision gas: argon 1.5mTorr



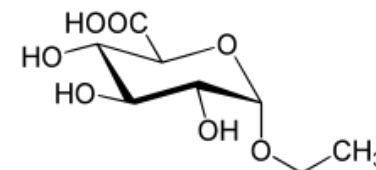


# Alcohol Biomarkers by LC-MS/MS

## Extracted ion chromatogram



Ethyl sulphate (EtS)



Ethyl glucuronide (EtG)

ACE Excel 1.7 C18 1.7µm, 100 x 2.1mm

Gradient analysis

A = 1mM ammonium fluoride

B = Acetonitrile

Time (mins)	%B	Time (mins)	%B
0	0	2.0	100
0.5	20	4.0	100
1.5	20	4.5	0

Flow rate: 0.4ml/min

Column temperature: 40°C

Injection volume: 1µl

AB SCIEX triple quad 5500

Negative ESI MRM

Source temperature: 750°C

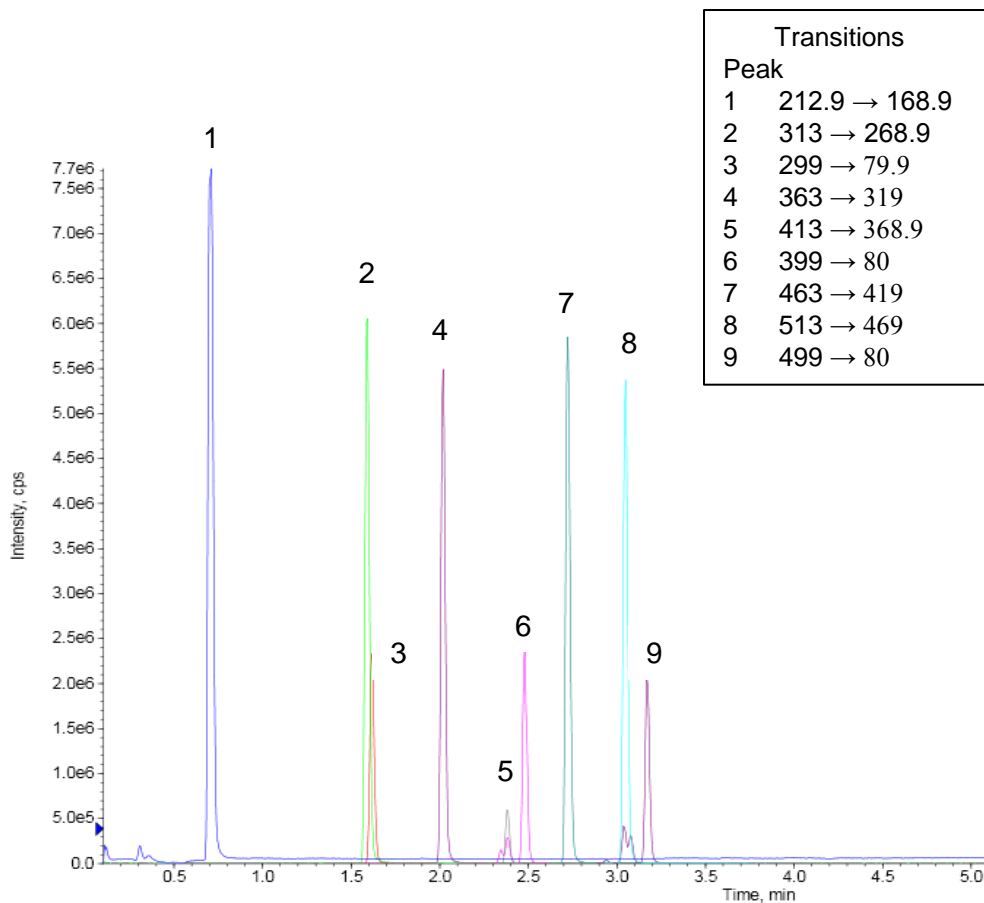
IonSpray voltage: -4500V

Fluoride counter-ion thought to enhance negative ESI response

Detection limit ~ 1ng/ml in oral fluid



# Perfluoro acids by LC-MS/MS



Peak ID	Analyte
1	Heptafluorobutyric acid
2	Perfluorohexanoic acid
3	Perfluorobutylsulphonic acid
4	Perfluoroheptanoic acid
5	Perfluorooctanoic acid
6	Perfluorohexylsulphonic acid
7	Perfluorononanoic acid
8	Perfluorodecanoic acid
9	Perfluorooctanesulphonic acid

ACE Excel 2 C18 2 $\mu$ m, 50 x 2.1mm

Gradient analysis

A = 2mM NH<sub>4</sub>OAc, 0.1% acetic acid/CH<sub>3</sub>CN (95:5)

B = 2mM NH<sub>4</sub>OAc, 0.1% acetic acid/CH<sub>3</sub>CN (5:95)

T (mins)	% B	T (mins)	% B
0	25	7.5	95
0.5	25	8.0	25
5.5	95	10.0	25

Flow rate: 0.5ml/min

Column temperature: 40°C

Injection volume: 20 $\mu$ l

AB SCIEX triple quad 5500

Negative ESI MRM

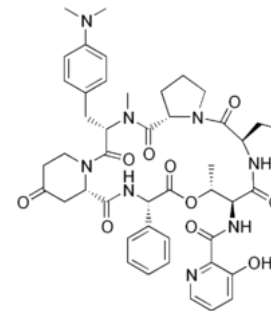
Source temperature: 450°C

IonSpray voltage: -2400V

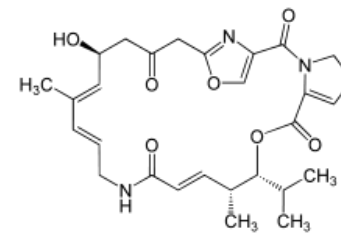


# Pristinamycin components in plasma by LC-MS/MS

Pristinamycin antibiotic is a mixture of 2 components –  
pristinamycin IA and IIA  
Virginiamycin used as internal standard

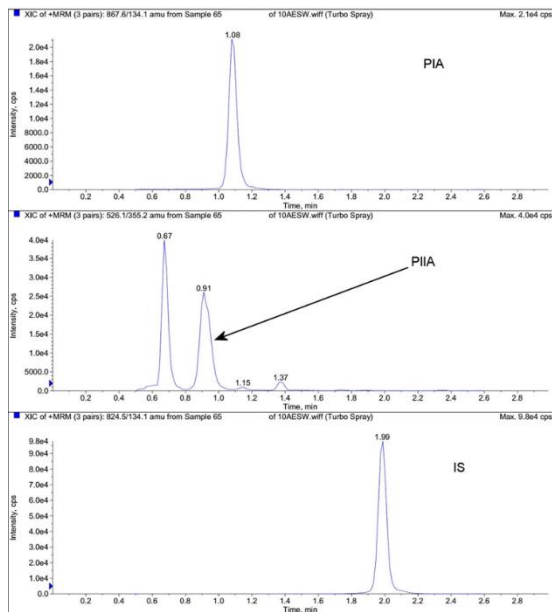


Pristinamycin IA

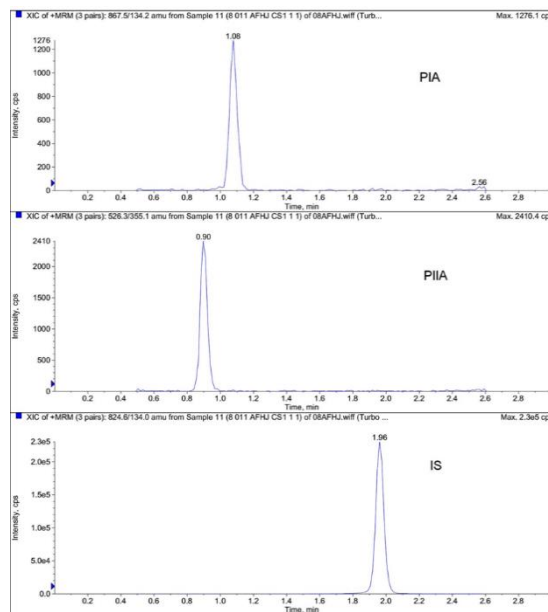


Pristinamycin IIA

Processed study sample containing  
pristinamycin IA and IIA



Low calibration standard containing  
2.5ng/ml each of pristinamycin IA and IIA  
in human NaF/K<sub>2</sub>C<sub>2</sub>O<sub>4</sub> plasma



ACE 3 C18 3µm, 30 x 3.0mm  
Gradient analysis  
A = 1mM NH<sub>4</sub>CO<sub>2</sub>H + 0.1%  
HCO<sub>2</sub>H in 65:35 H<sub>2</sub>O:CH<sub>3</sub>CN  
B = CH<sub>3</sub>CN

T (mins)	%B	T (mins)	%B
0	0	1.61	100
0.3	0	2.6	100
0.31	10	2.61	0
1.6	10	4	0

Flow rate: 1ml/min

Column temperature: 25°C

Injection volume: 10µl

MDS Sciex API 4000

TurboIonSpray positive mode

Transitions monitored:

Pristinamycin IA 867.5 → 134.2

Pristinamycin IIA 526.3 → 355.1

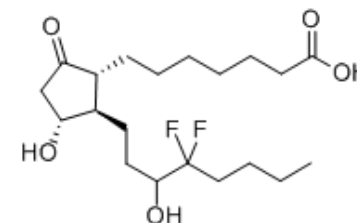
I.S. (Virginiamycin) 824.6 → 134.0



# 15-Hydroxy Lubiprostone in Human Plasma

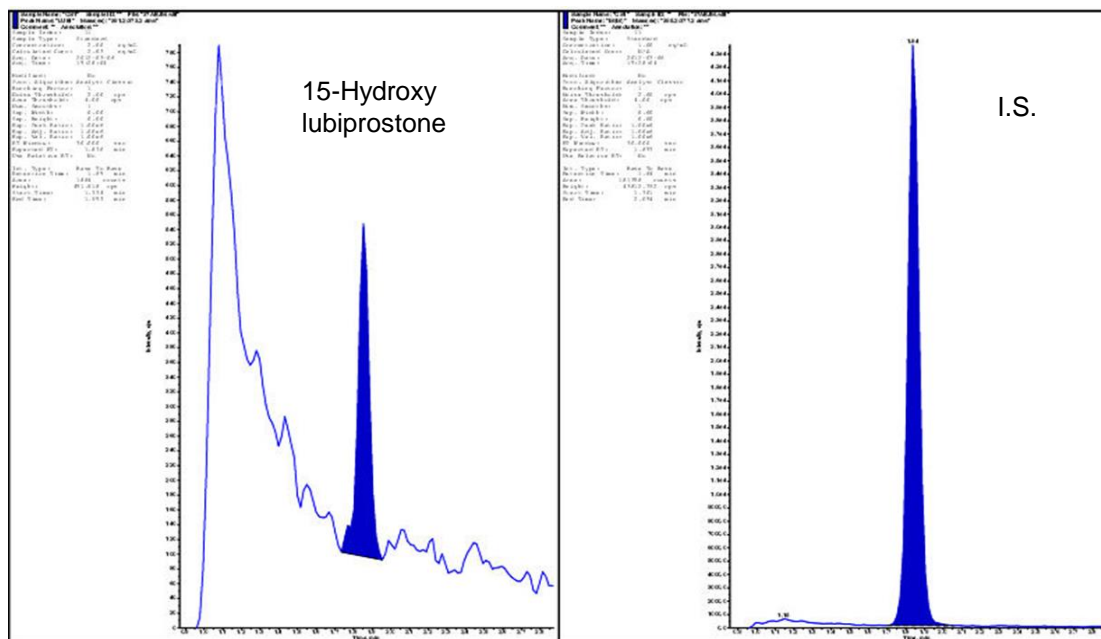
Lubiprostone, a fatty acid derived from prostaglandin E1, is rapidly metabolised to 15-hydroxy lubiprostone.

Quantitation is based on 15-hydroxy lubiprostone, with the d4 analogue as internal standard



15-Hydroxy lubiprostone  
MW 392.5

Lowest calibration standard sample containing 2.0pg/ml in human EDTA K3 plasma



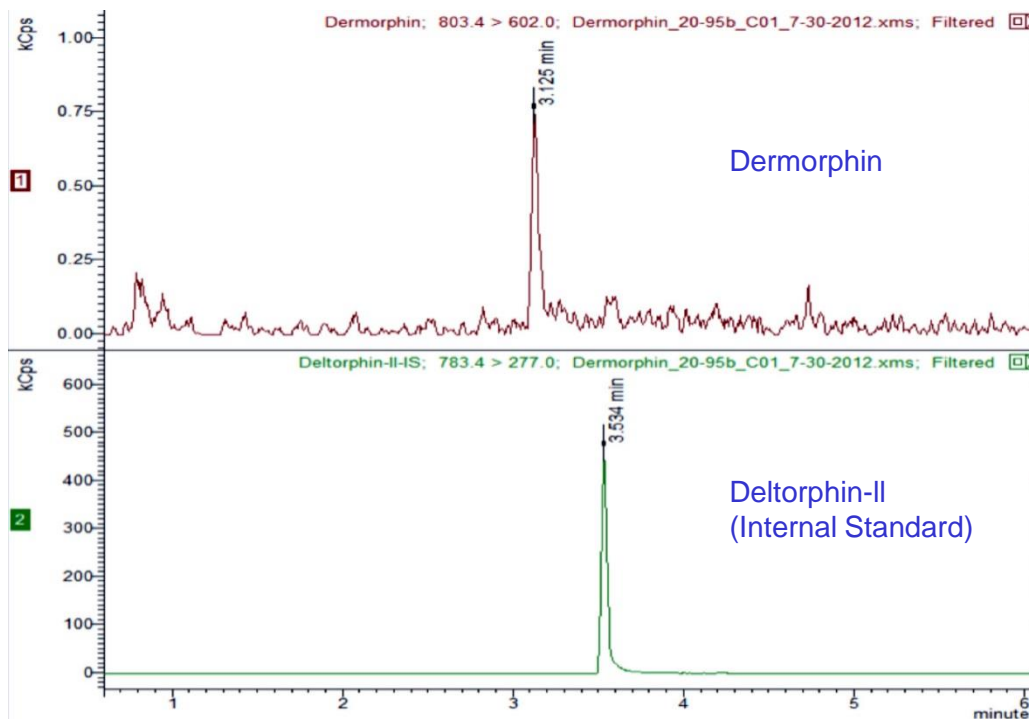
ACE Excel 2 C18 2 $\mu$ m, 50 x 3.0mm  
Isocratic analysis  
A = 0.1% formic acid in water  
B = Acetonitrile  
Flow rate: 0.65ml/min  
Column temperature: 35°C  
Injection volume: 15 $\mu$ l

MDS Sciex API 5000  
TurbolonSpray negative mode  
IonSpray voltage: -4500V  
Source temperature: 450°C

Transitions monitored:  
15-Hydroxy lubiprostone  
391.2  $\rightarrow$  373.2  
I.S. (15-Hydroxy lubiprostone-d4)  
395.2  $\rightarrow$  377.2

# Dermorphin in Equine Urine by LC-MS/MS

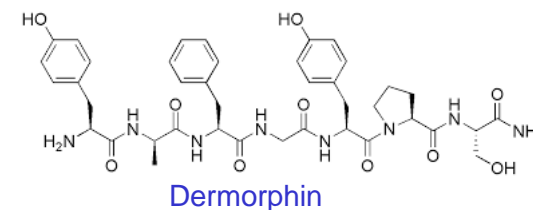
MRM chromatogram of 0.05ng/ml dermorphin in equine urine



Accurate quantification of dermorphin in equine urine in range 0.05 – 100ng/ml

LLOQ = 0.05ng/ml

Transitions  
 Dermorphin:  
 m/z 803.4 → 602 (quantifier ion)  
 m/z 803.4 → 202 (qualifier ion)  
 Deltorphin:  
 m/z 783 → 277



ACE 3 C18 (3 $\mu$ m, 100 x 2.1mm)  
 Gradient analysis  
 A = 0.2% formic acid in water  
 B = 0.2% formic acid in acetonitrile

T (mins)	%B	T (mins)	%B
0	5	8.5	95
0.2	5	8.51	5
8	95	12.5	5

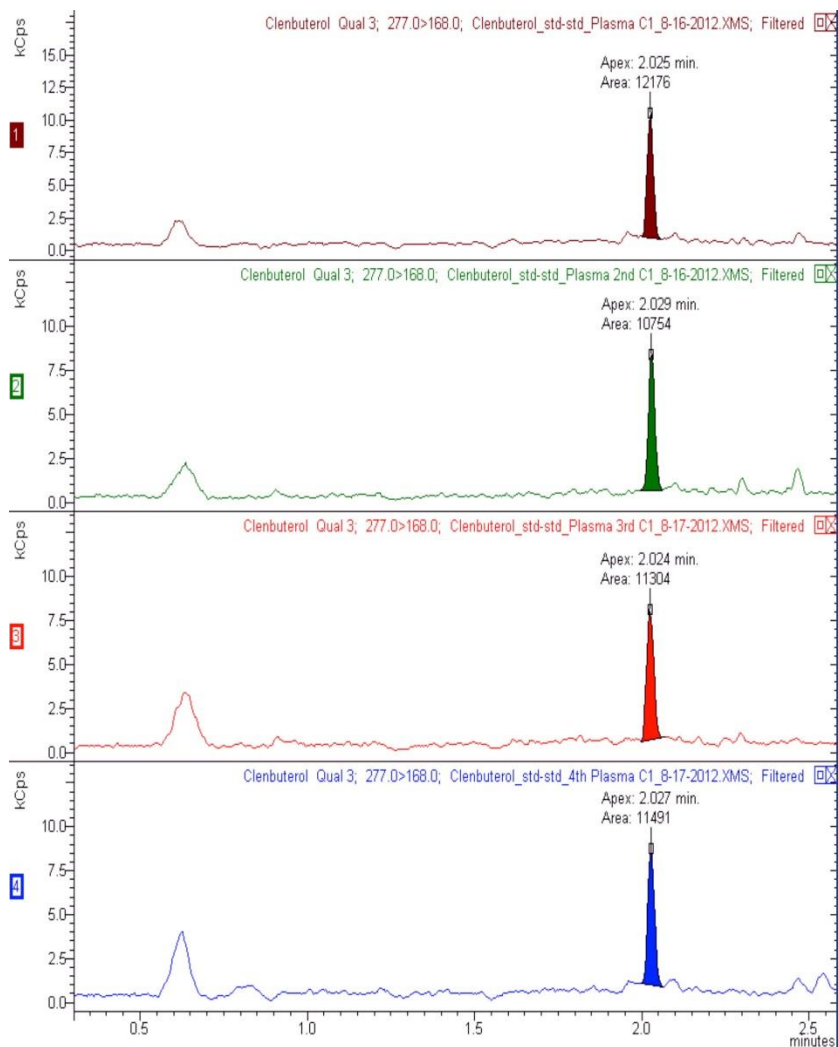
Flow rate: 0.4ml/min  
 Injection volume: 40 $\mu$ l

Bruker EVOQ Elite triple quad MS  
 VIP heated-ESI temperature: 350°C  
 Cone gas temperature: 250°C  
 Spray voltage: +4000V

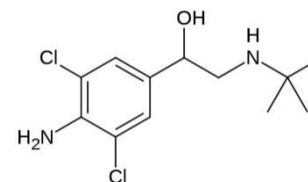


# Clenbuterol in Equine Plasma by LC-MS/MS

Representative MRM chromatograms of 5 ppt clenbuterol (150 fg on-column)



Sustained high sensitivity performance under repeated exposure to horse plasma samples. Clenbuterol in crashed horse plasma injected by dilute and shoot method.



Clenbuterol

ACE 3 C18 (3 $\mu$ m, 100 x 2.1mm)  
Gradient analysis

A = 0.2% formic acid in water

B = 0.2% formic acid in acetonitrile

T (mins)	%B	T (mins)	%B
0	10	2.8	10
0.3	10	4.5	10
2.5	95		

Flow rate: 0.45ml/min

Injection volume: 30 $\mu$ l

Bruker EVOQ Elite triple quad MS

VIP heated-ESI temperature: 300°C

Cone gas temperature: 300°C

Spray voltage: +3500V

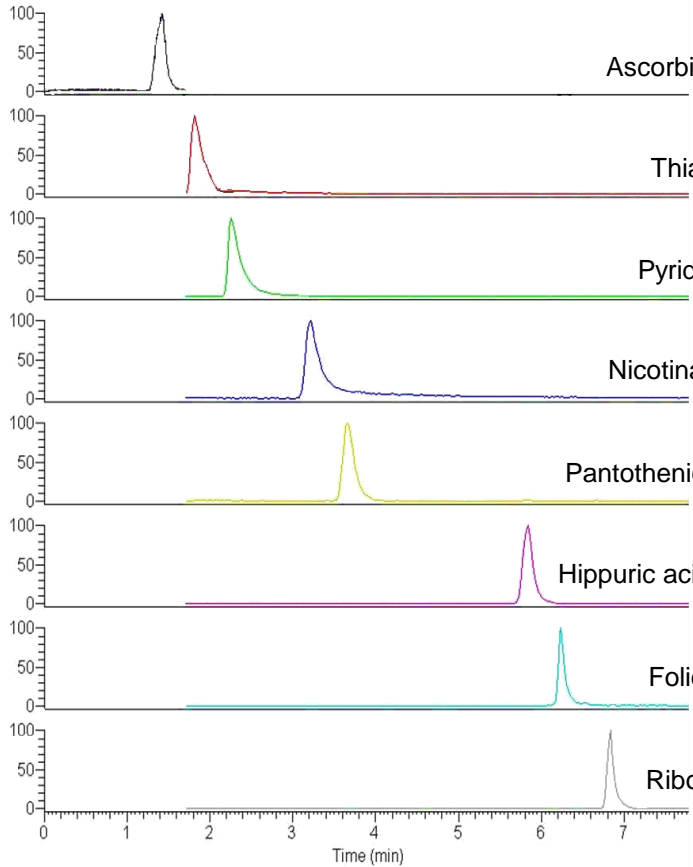
Transitions:

Clenbuterol m/z 277.1  $\rightarrow$  168

d9-Clenbuterol m/z 286.1  $\rightarrow$  204

(Internal Standard)

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



Ascorbic acid

Thiamine

Pyridoxine

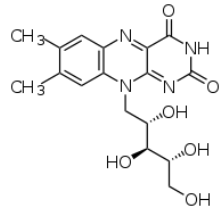
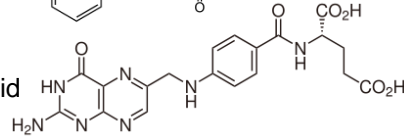
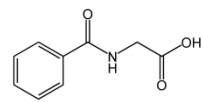
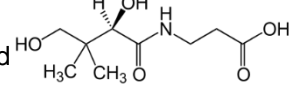
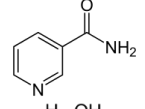
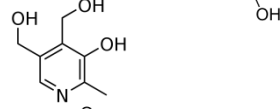
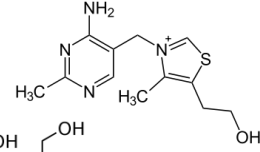
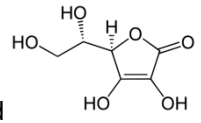
Nicotinamide

Pantothenic acid

Hippuric acid (IS)

Folic acid

Riboflavin



ACE C18, 3µm, 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

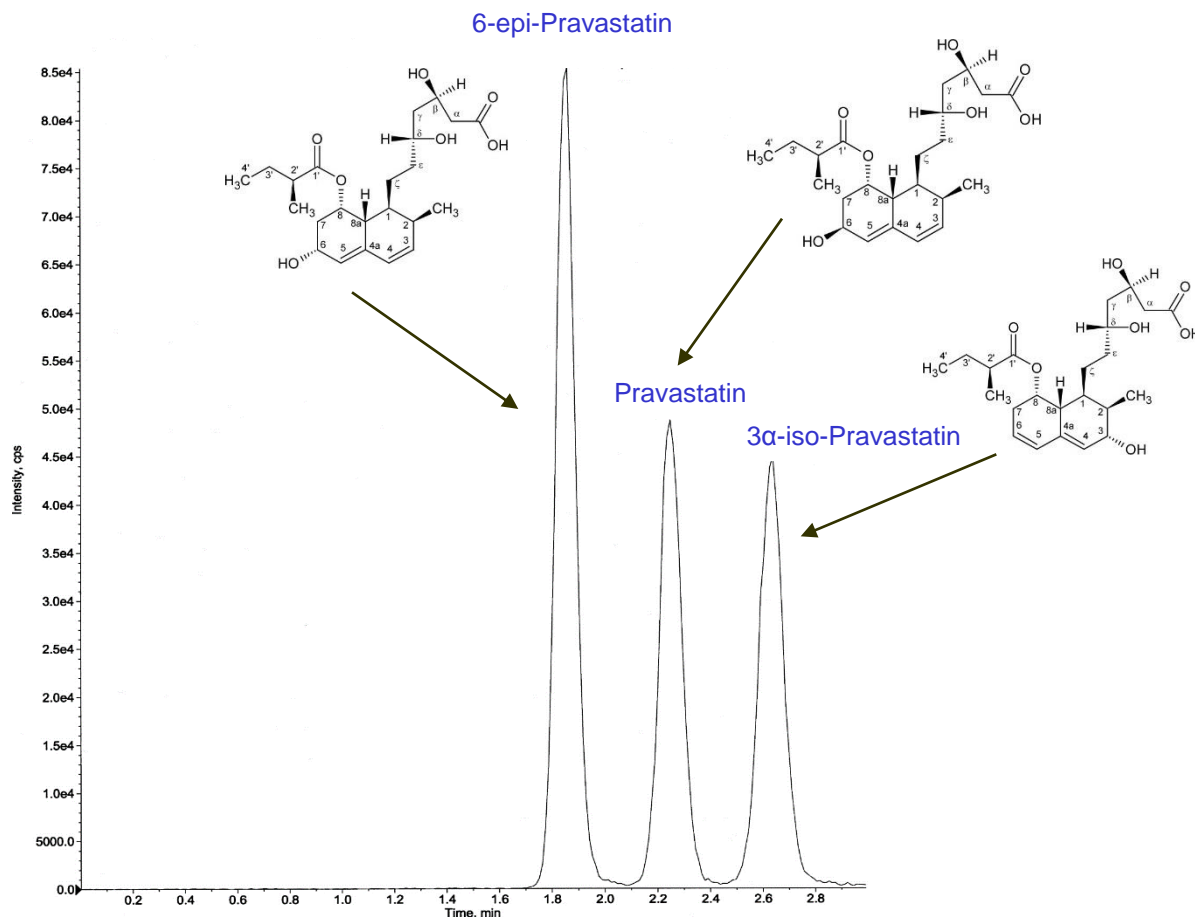
Time (mins)	%A	%B	%C
0	90	10	0
3	90	10	0
4	50	0	50
7	50	0	50
10	0	100	0

Flow rate: 0.2ml/min  
Injection volume: 10µl  
Column temperature: 20°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 → 115.2	128.3
Thiamine (Vit B1)	265.1 → 122.1	2.4
Pyridoxine (Vit B6)	169.9 → 152.1	0.6
Nicotinamide (Vit B3)	123.0 → 80.3	13.2
Pantothenic acid (Vit B5)	220.0 → 202.1	23.3
Folic acid (Vit B9)	442.0 → 294.9	1.9
Riboflavin (Vit B2)	377.1 → 243.0	0.2
Hippuric acid (IS)	180.1 → 105.2	14.9



# Pravastatin and Isomers by LC-MS/MS



All 3 compounds have MW 424

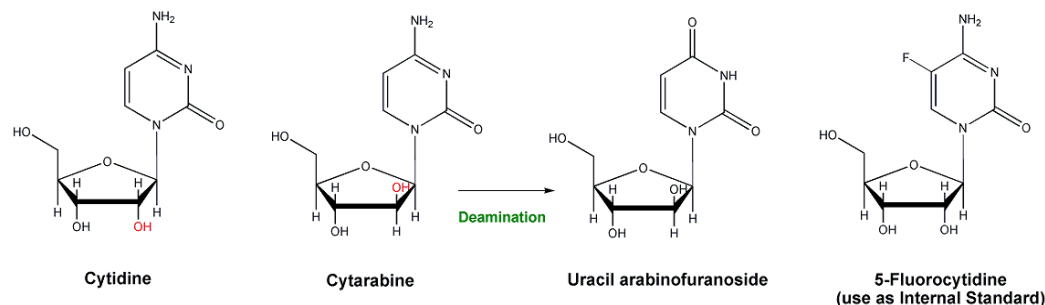
MS/MS conditions alone  
insufficient for selective  
quantitation

Baseline separation important

**ACE C18 3 μm, 50 x 3.0 mm**  
**Isocratic analysis**  
**Acetonitrile-Methanol-THF-Water-Acetic acid (15:20:5:60:0.1)**  
**Flow rate: 0.6 ml/min**  
**Column temperature: Ambient**  
**Injection volume: 2 μl**  
**Sample: 1 μg/ml each isomer**

**API 3000 triple quad MS**  
**TurbolonSpray – negative mode**  
**Extracted ion chromatogram of**  
**MRM m/z 423.3 → 321.1**

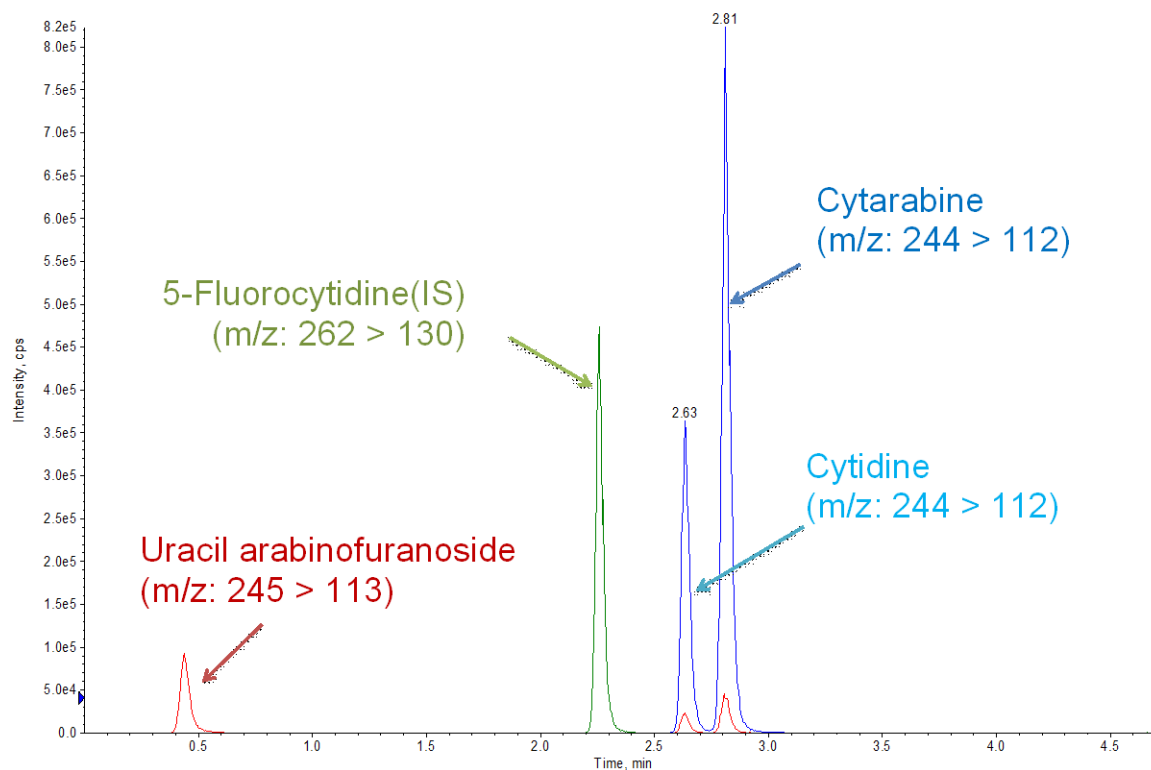
# Cytarabine Analogues by Ion-Pairing LC-MS/MS



Cytarabine and cytidine are isobaric.

Robust method with good separation achieved.

LLOQ = 1 ng/ml human plasma



ACE 3 C18 3 $\mu$ m, 50 x 2.1 mm  
 Gradient analysis  
 A = 0.1% perfluoropentanoic acid + 0.1% formic acid in water  
 B = 0.1% perfluoropentanoic acid + 0.1% formic acid in acetonitrile

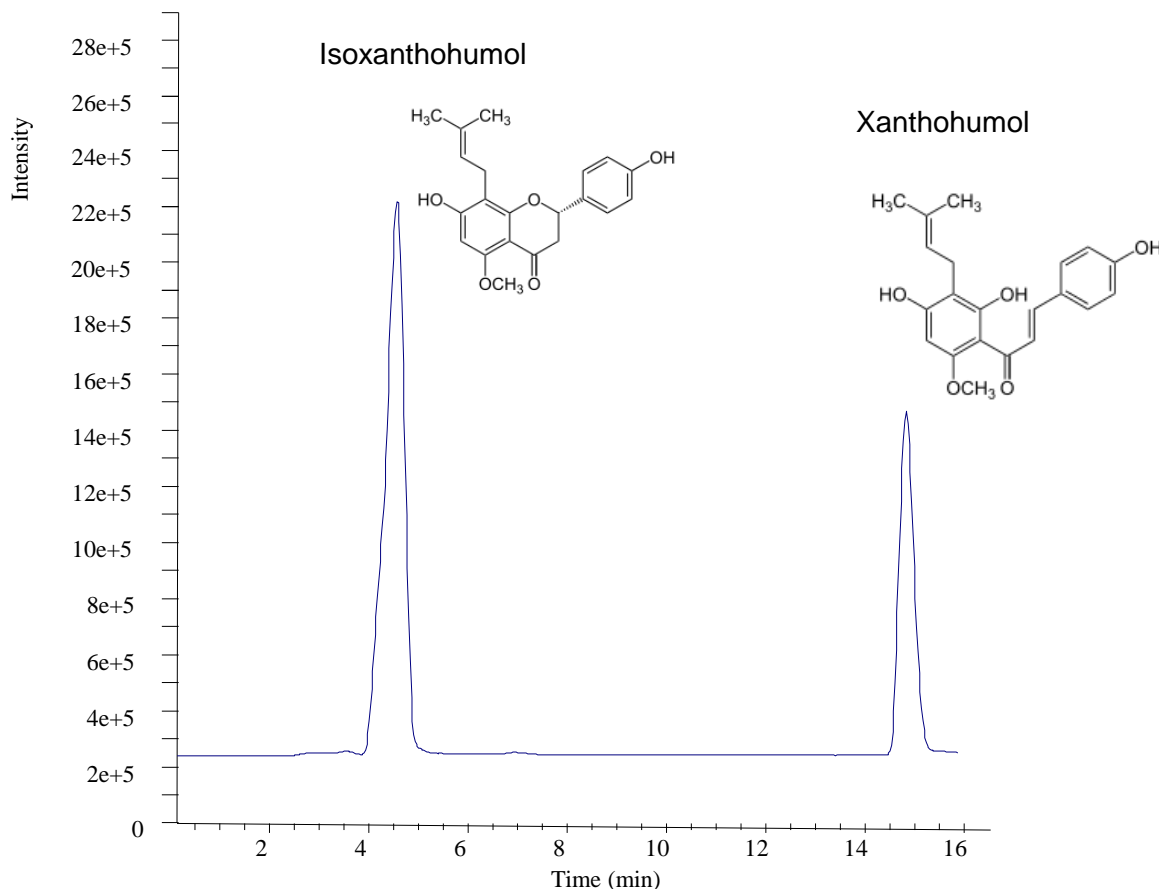
T (mins)	%B
0	0
0.5	0
3.0	13
4.0	90
5.0	0

Flow rate: 0.7 ml/min

API 4000 MS  
 TurbolonSpray, positive mode  
 Source temperature: 550°C



# 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

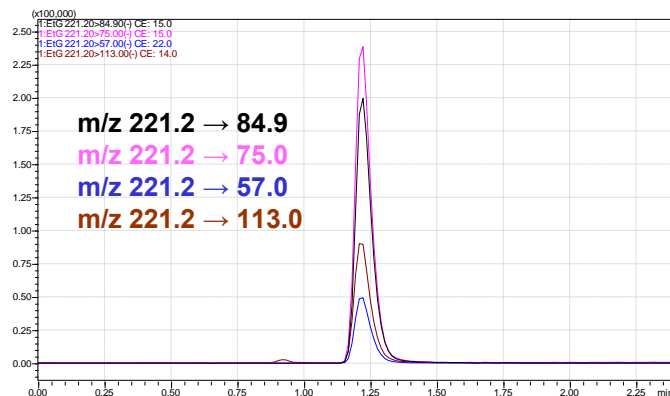
LOQ: xanthohumol: 0.01 $\mu$ g/ml



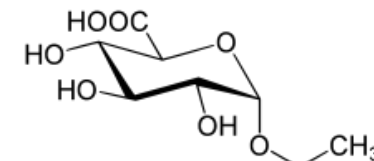
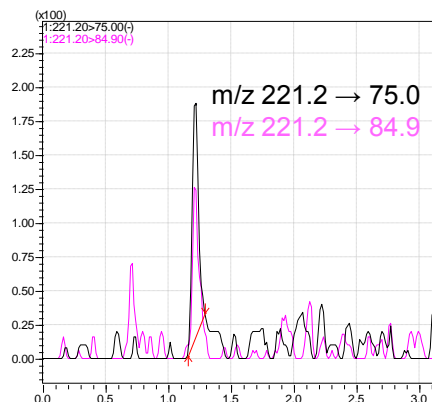


# Ethyl Glucuronide in Water by LC-MS/MS

## Ethyl glucuronide (EtG)



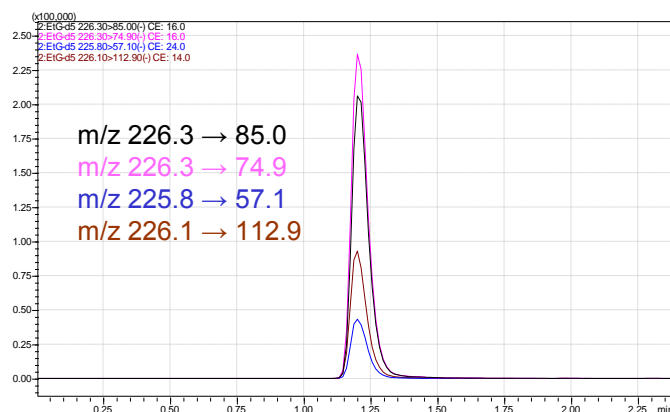
## EtG 100pg/ml



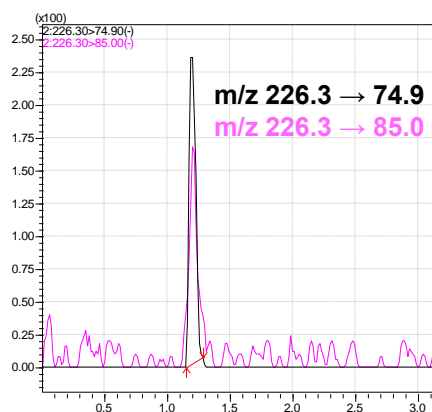
Ethyl glucuronide

**LOQ for EtG in water  
= 300pg on-column**

## Ethyl glucuronide-d5 (EtG-d5)



## EtG-d5 100pg/ml



ACE Excel 2 C18-PFP

2µm, 100 x 2.1 mm

Gradient analysis

A = 0.05% formic acid in water

B = Methanol

T (mins)	%B	T (mins)	%B
4	70	7	95
6	95	7.01	5

Flow rate: 0.4 ml/min

Injection volume: 3 µl

Column temperature: 40°C

Shimadzu LCMS-8050

ESI voltage: -3kV

Desolvation line: 250°C

Interface heater: 380°C

Nebulizing gas: 3 l/min

Heat block: 400°C



# 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:

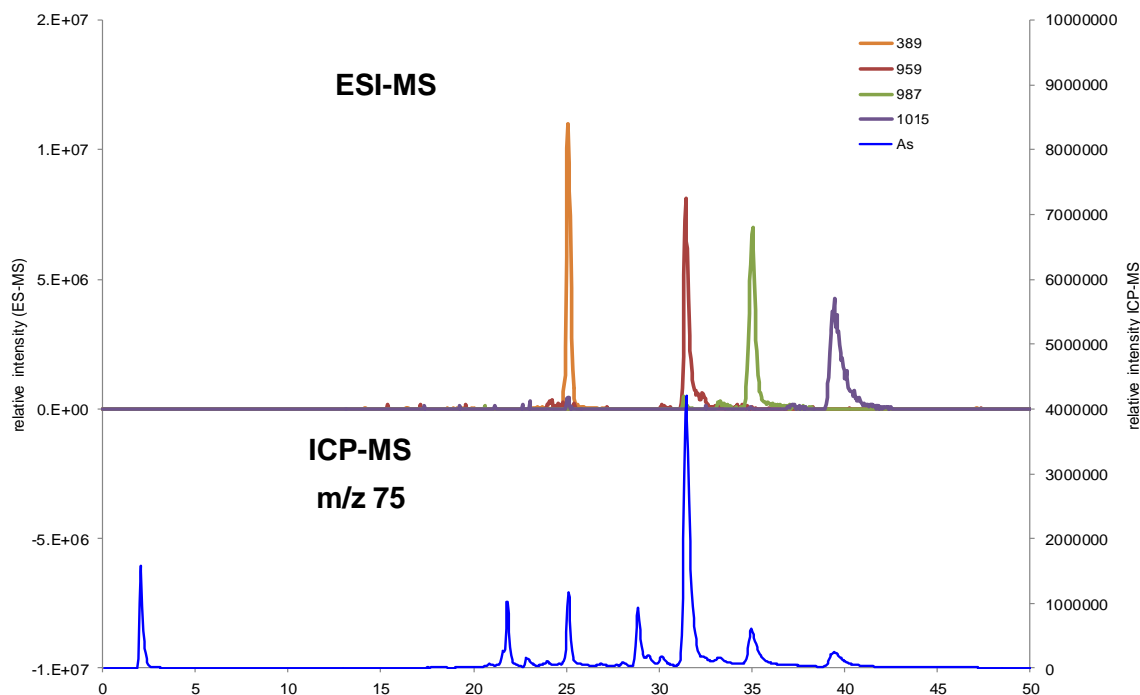
$m/z$  389 [M + H]<sup>+</sup> for C<sub>21</sub>H<sub>46</sub>AsO

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: 1ml/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

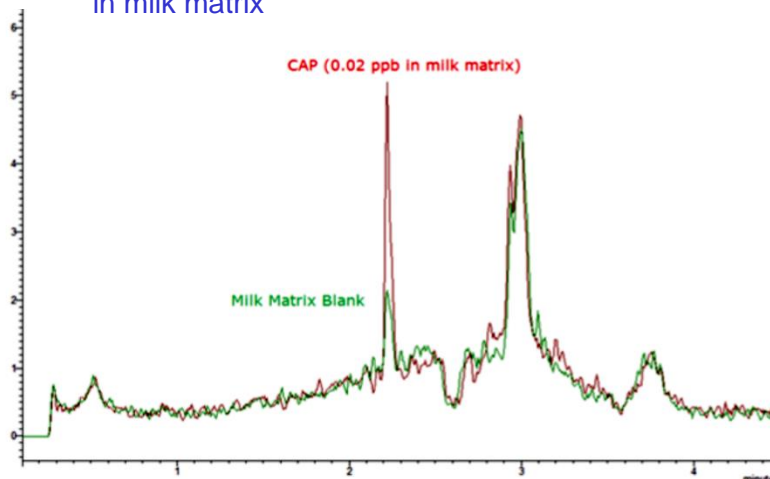
Capillary temperature: 320°C

Capillary voltage: 42V

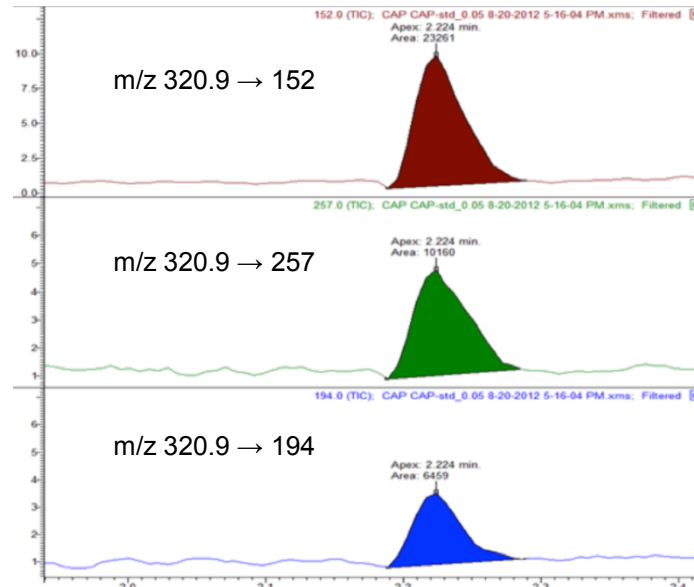


# Chloramphenicol in Milk by LC-MS/MS

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

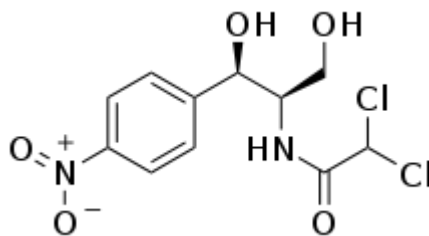
Injection volume: 10 $\mu$ l

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



# Organophosphorus Flame Retardants in Water by LC-MS/MS

Symbol	Compound Name	Q1 Mass	Q3 Mass
TMP	tri-methyl phosphate	141	109
TEP	tri-ethyl phosphate	183	127
TiPP	tri-iso-propyl phosphate	225	99
TPrP	tri-n-propyl phosphate	225	99
TBP	tri-n-butyl phosphate	267	211
TCEP	tris(2-chloroethyl) phosphate	285	223
TCPP	tris((2R)-1-chloro-2-propyl) phosphate	327	99
TDCPP	tris(1,3-dichloro-2-propyl) phosphate	431	99
BDCP	bis(1,3-dichloro-2-propyl) phosphate	321	99
TPP	triphenyl phosphate	327	215
TBEP	tris(2-butoxyethyl) phosphate	399	299
BEHP	bis(2-ethylhexyl) phosphate	323	99

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

Gradient analysis

A = 0.05mM NH<sub>4</sub>CO<sub>2</sub>H + 0.005%  
HCO<sub>2</sub>H in water

B = CH<sub>3</sub>OH/CH<sub>3</sub>CN (95:5)

Time (mins) %B Curve

0.1 50 -3

12.0

90

13.0

100

15.0

100

15.1

50

20.0

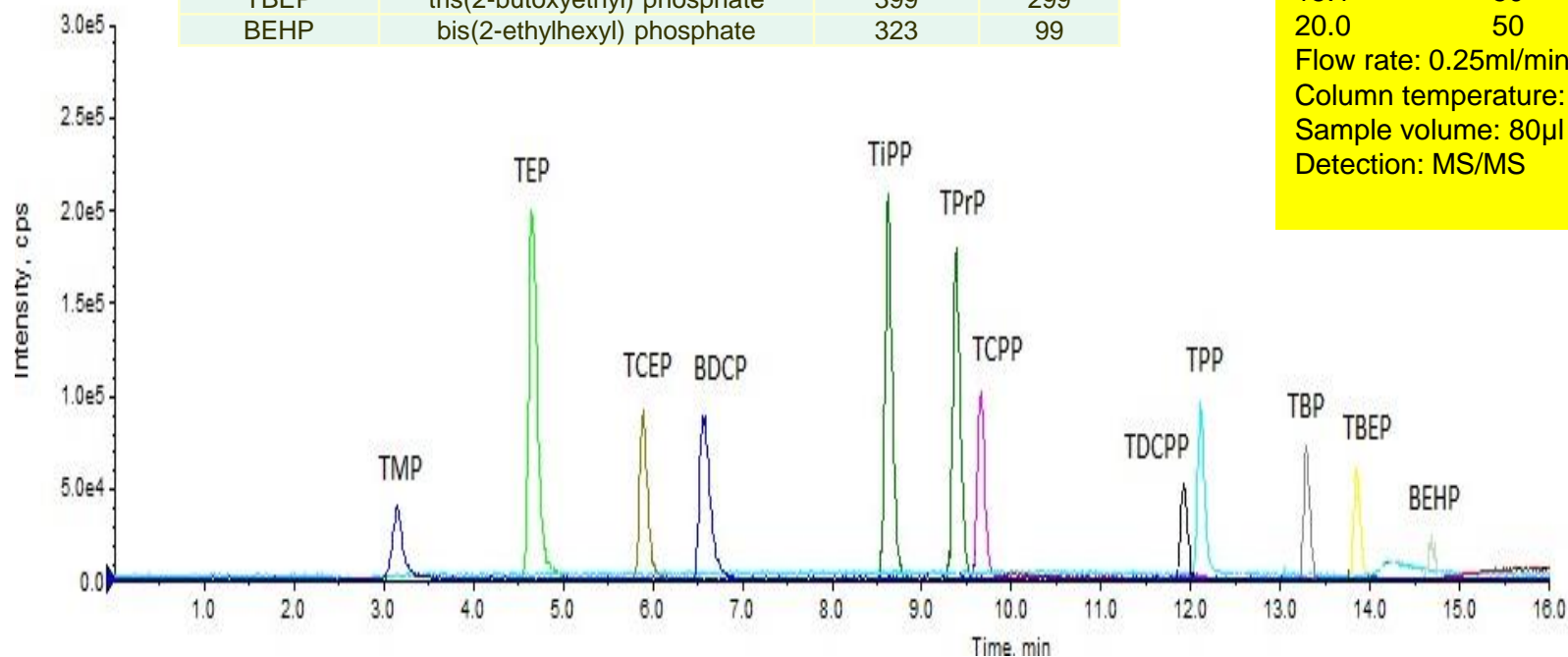
50

Flow rate: 0.25ml/min

Column temperature: 25°C

Sample volume: 80 $\mu$ l

Detection: MS/MS





# ACE UltraCore SuperC18: Impurity Profile of a Herbicide

ACE UltraCore SuperC18, 2.5µm, 150 x 4.6mm

Gradient analysis

A = CH<sub>3</sub>CN – H<sub>2</sub>O – TFA (5:95:0.05 v/v/v)

B = CH<sub>3</sub>CN – TFA (99.9:0.05 v/v/v)

Time (mins)	%B	Time (mins)	%B
0	10	55	100
3	10	56	10
35	100	60	10

Flow rate: 0.60ml/min

Column temperature: 25°C

Injection volume: 10µl

Detection: UV, 240nm

Sample: Technical Grade Herbicide

