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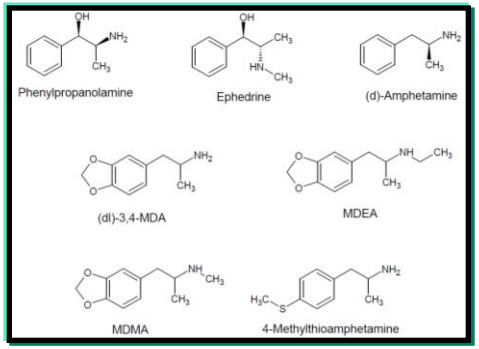
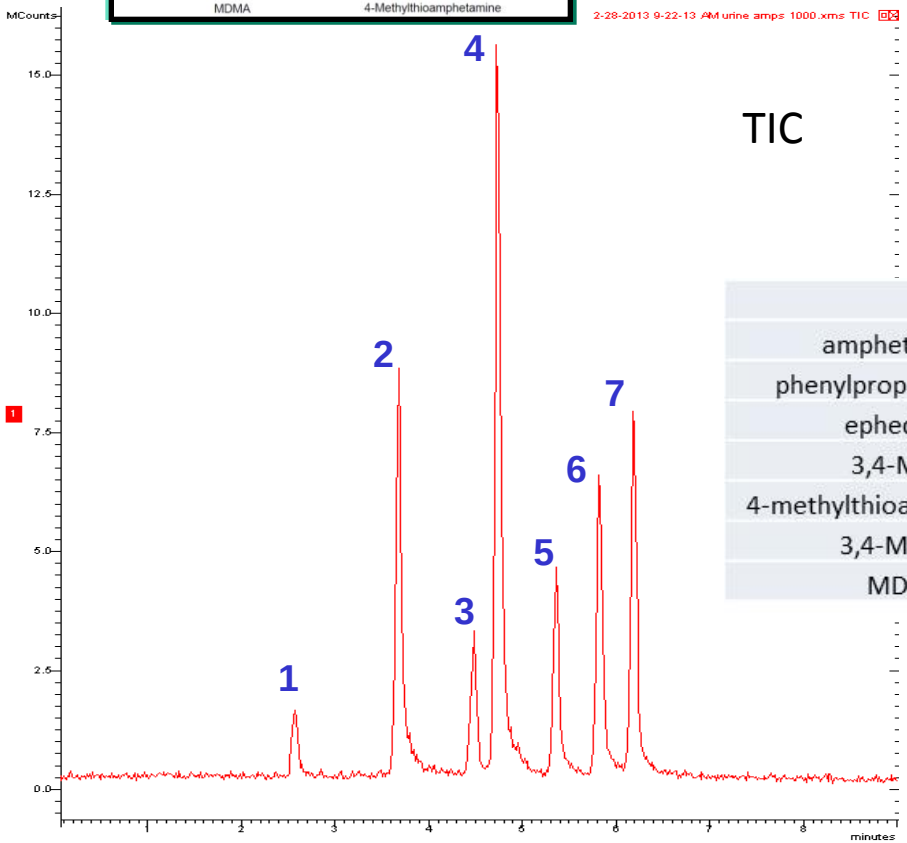
Amphetamines In Urine by LC-MS/MS



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



	LOD (est)
amphetamine	4 ppb
phenylpropanolamine	4 ppb
ephedrine	2 ppb
3,4-MDA	30 ppb
4-methylthioamphetamine	10 ppb
3,4-MDMA	2 ppb
MDEA	1 ppb

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

MP A: 5mM Ammonium Hydroxide, pH 10.8.
MP B: 5mM Ammomnium Hydroxide, pH 10.8 in 1:9 v/v H2O: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

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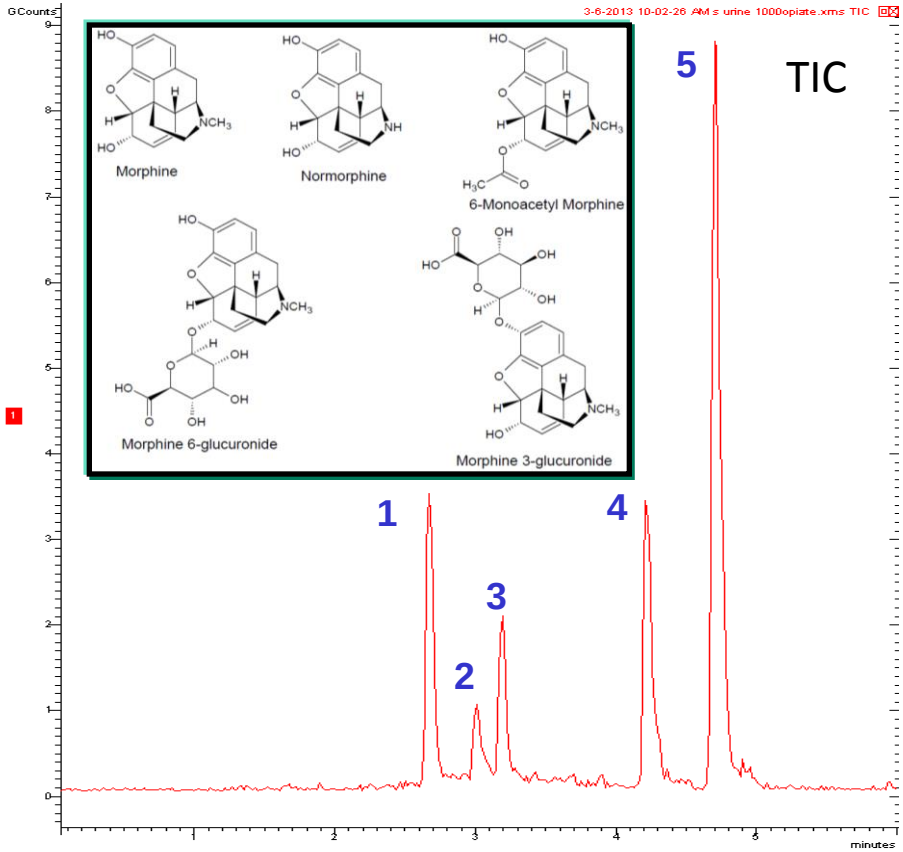
Opiates In Urine by LC-MS/MS

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-acetylmorphone	10 ppb
Morphine 3-β-DG	100ppb
Morphine 6-β-DG	100ppb



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

MP A: 5mM Ammonium Hydroxide, pH 10.8.
MP B: 5mM Ammomnium Hydroxide, pH 10.8 in 1:9 v/v H2O:MeOH.
0.6mL/min

T	%B
0	5
5	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
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

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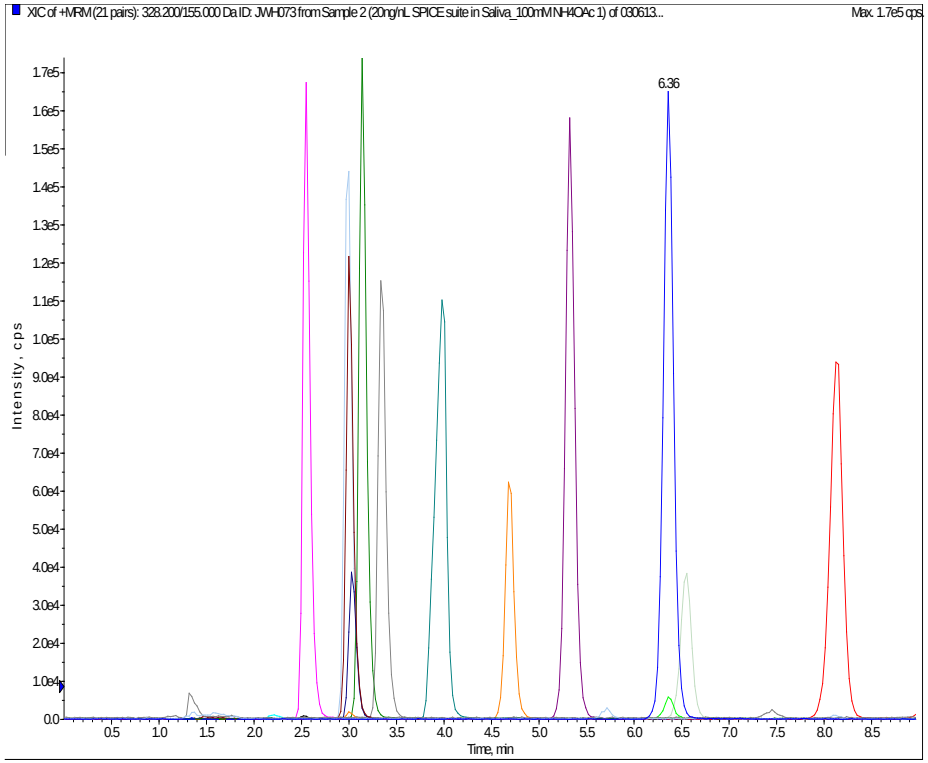
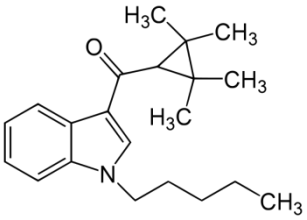
Synthetic Cannabinoids (SPICE) From Oral Fluid

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

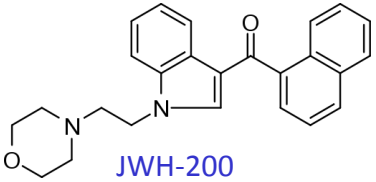


Retention Time (minutes)	Analyte	MRM Transition	Declustering Potential (DP)	Collision Energy (CE)	Cell Exit Potential (CXP)
2.55	JWH-250 N-(5-hydroxypentyl)	352>120.9	40	30	16
2.99	JWH-073 N-(3-hydroxybutyl)	344>155	40	30	16
3.00	UR-144 5-Hydroxy-pentyl	328.5>125	30	35	16
3.03	UR-144 Pentanoic Acid	342.5>125	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

ACE Excel C18-AR 100x2.1mm, 2µm
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®



UR-144



JWH-200

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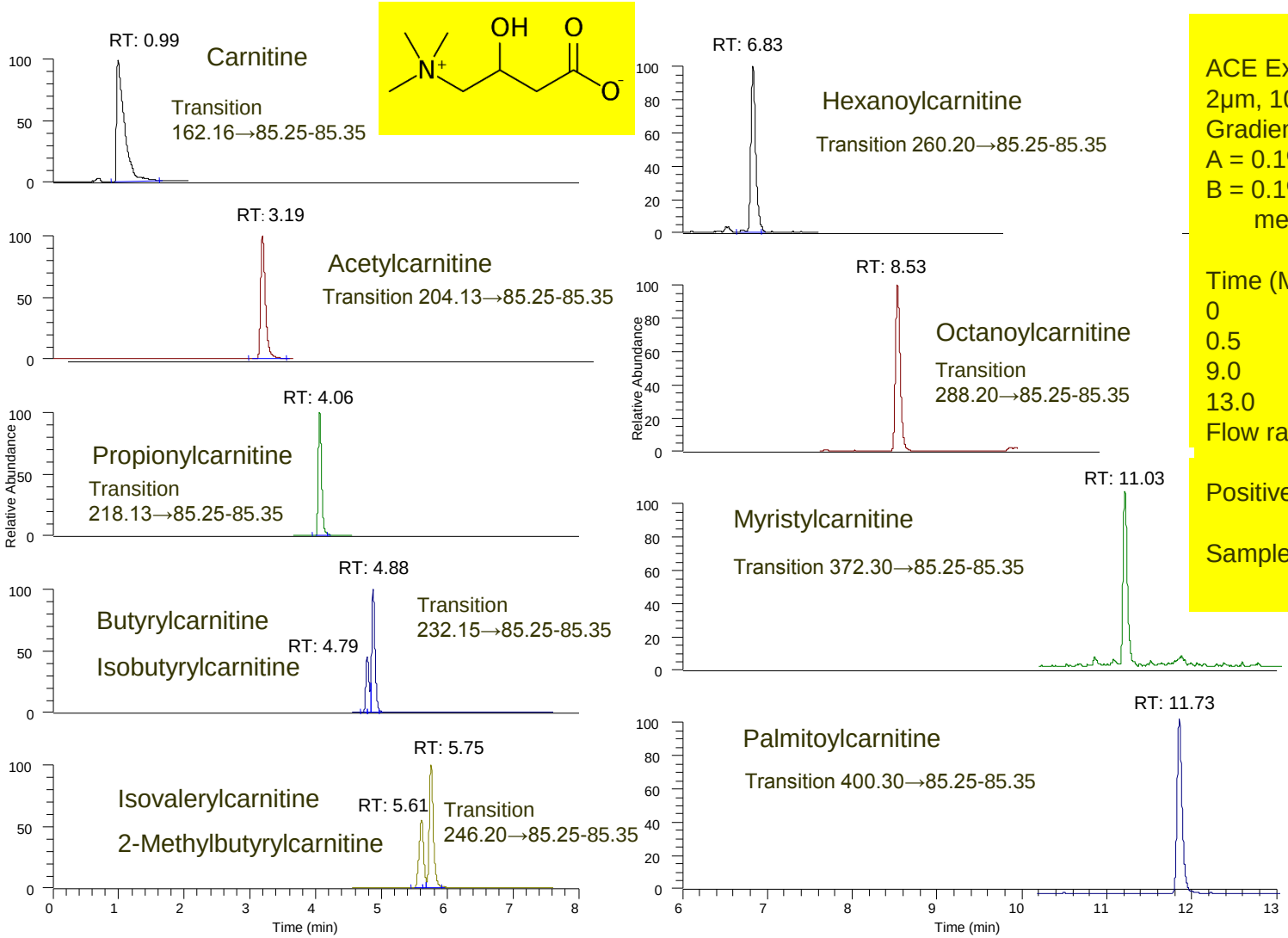
LC-MS/MS of Acylcarnitines



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

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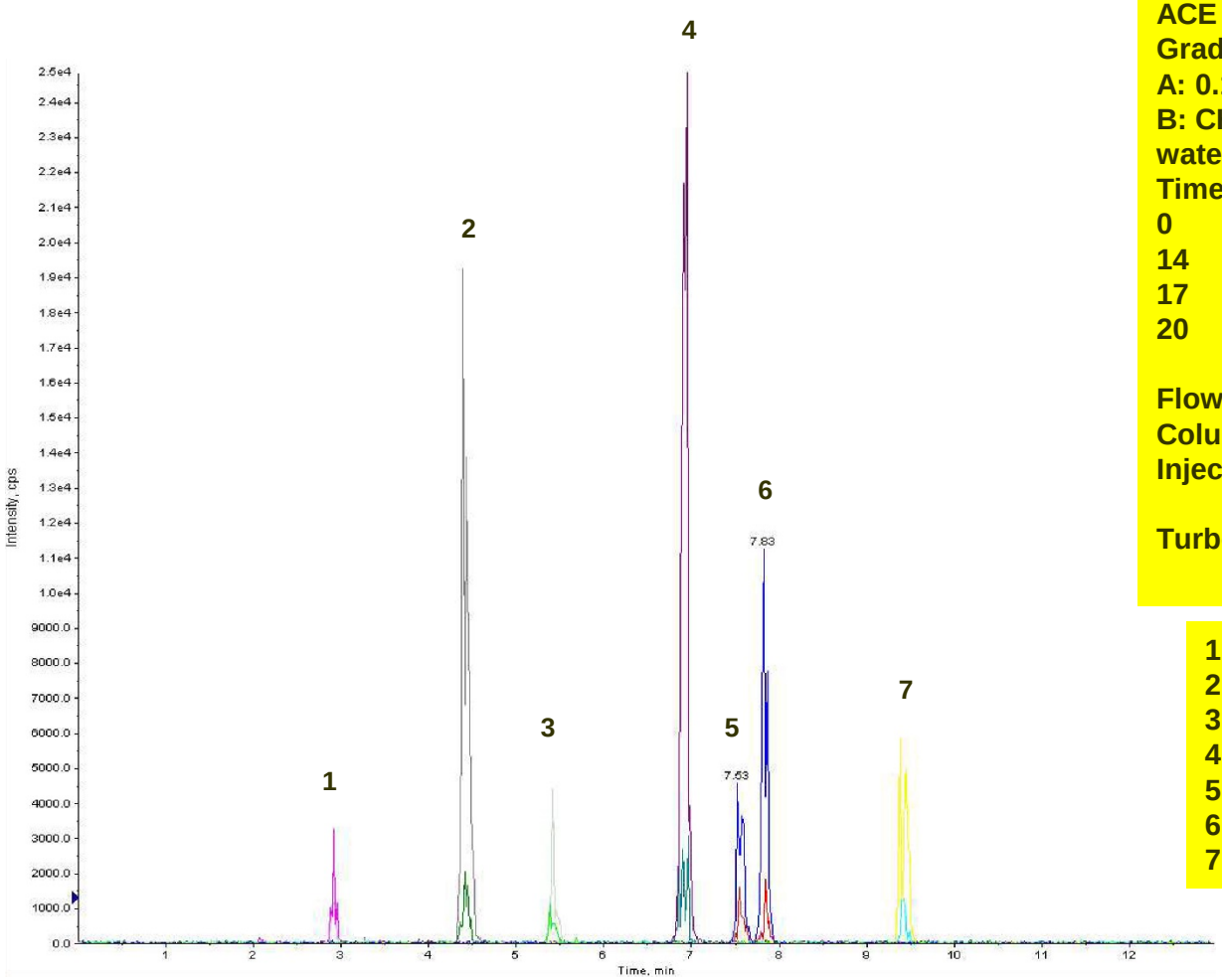
ACE 3 C18-PFP: Corticosteroids by LC-MS/MS



ACE C18-PFP, 3µm, 150 x 2.1mm
Gradient analysis
A: 0.1% formic acid in water
B: CH_3CN – 0.1% formic acid in water (90:10)

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

Flow rate: 0.3ml/min
Column temperature: 15°C
Injection volume: 25µl

Turbospray, MRM

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

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Aminoglycosides in Eggs

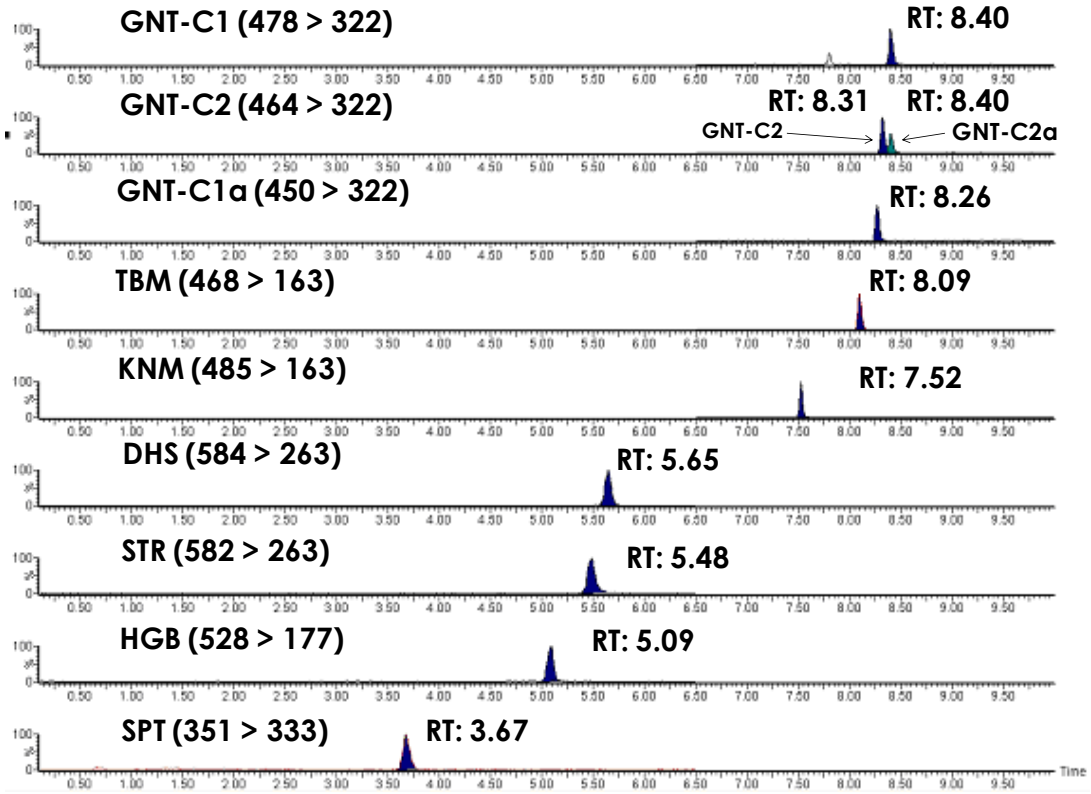
UHPLC-MS/MS method for aminoglycosides in eggs

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

Key

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

Egg sample spiked at 100µg/kg (CCα)



ACE Excel C18-PFP, 2µm, 100 x 2.1mm
Gradient analysis
A = 20mM HFBA in H_2O/CH_3CN (98:2)
B = 20mM HFBA in CH_3CN/H_2O (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)

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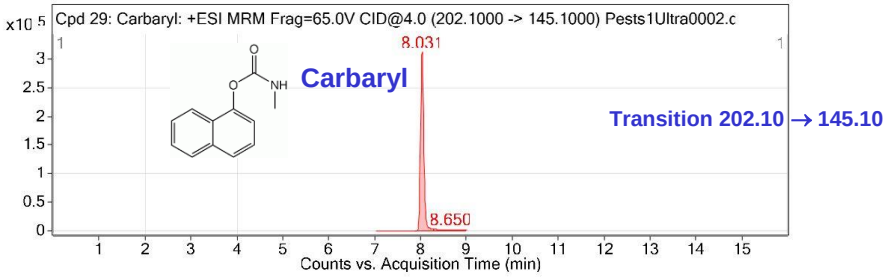
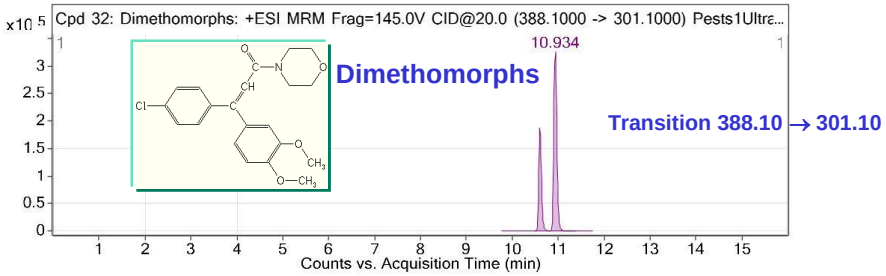
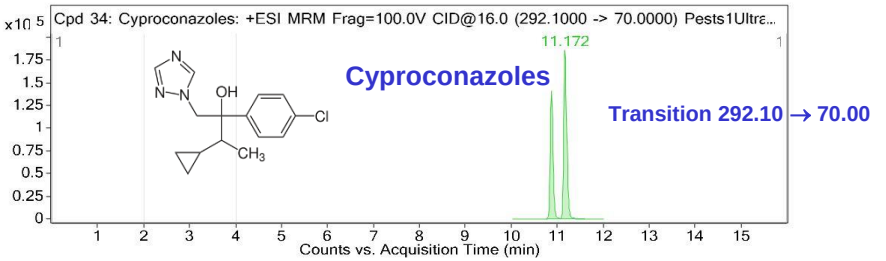
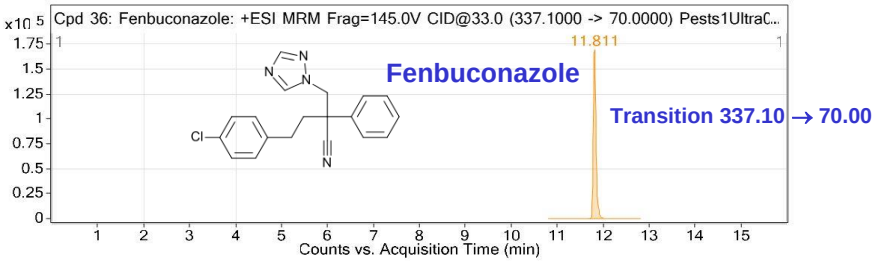
Pesticides by LC-MS/MS









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

A = 0.1% HCOOH + 5mM NH_4CO_2H in 9:1 v/v H_2O: MeOH
B = 0.1% HCOOH + 5mM NH_4CO_2H in 1:9 v/v H_2O: MeOH

Flow Rate: 0.4ml/min
Temperature: 40°C
Injection volume: 20µl

Gradient conditions

Time (mins)	0	1	15	18	18.05	20
%B	0	0	100	100	0	0

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

Also analysed under same conditions:

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

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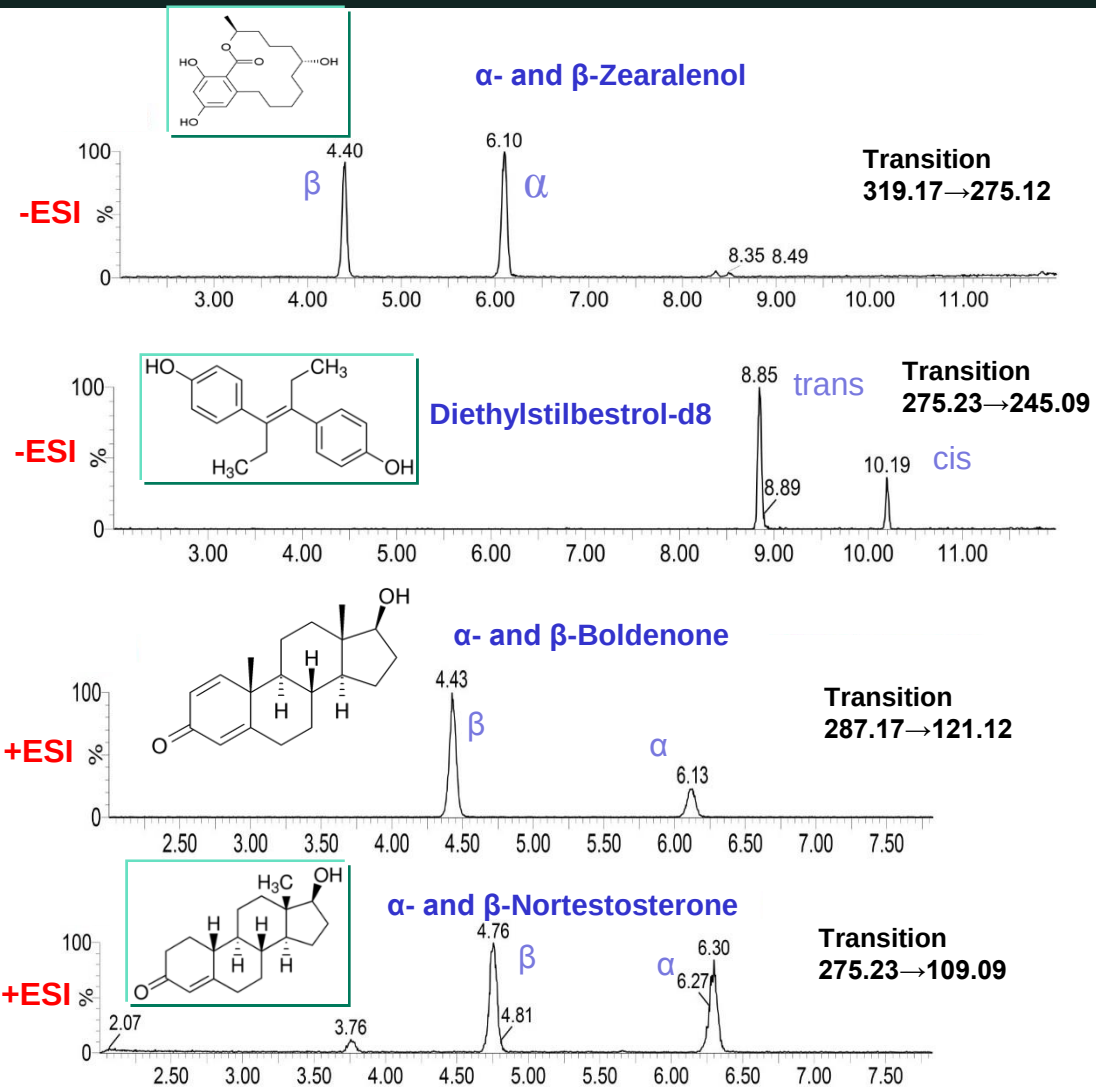
Veterinary Steroids by LC-MS/MS



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

ACE UltraCore SuperC18, 2.5um, 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

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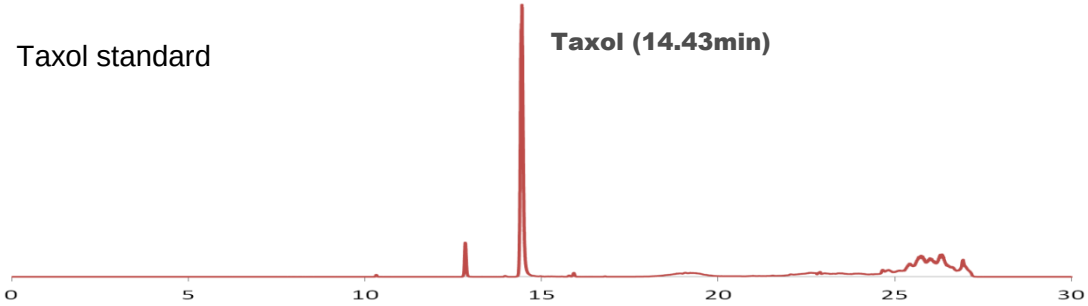
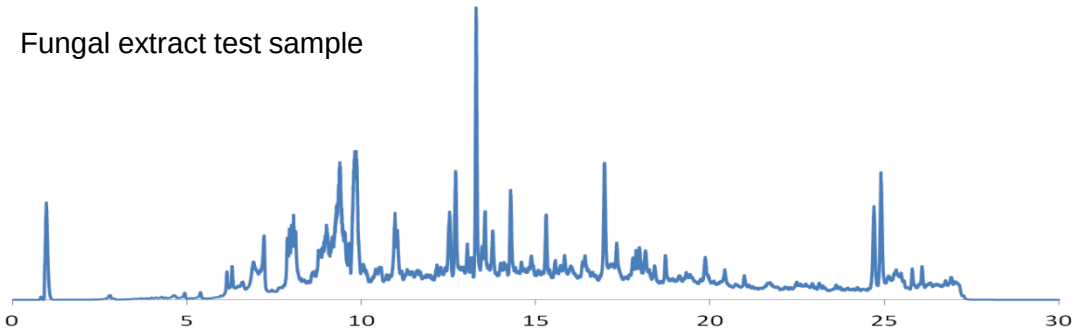
Taxol in Fungal Extract by LC-MS/MS

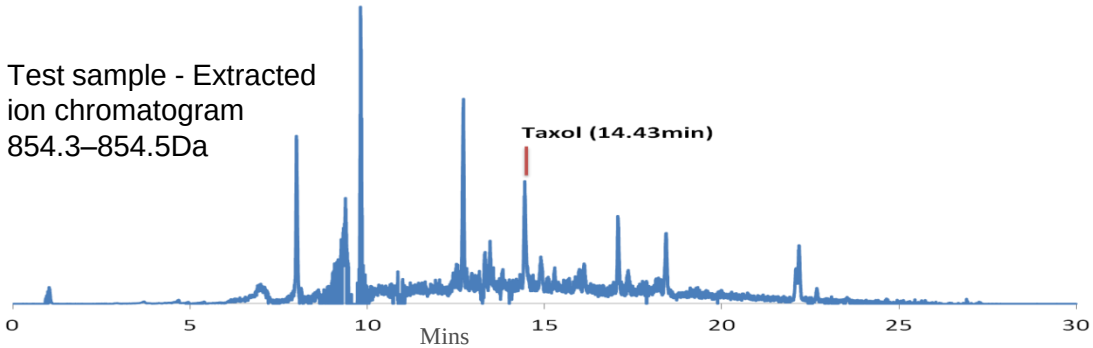
Taxol standard

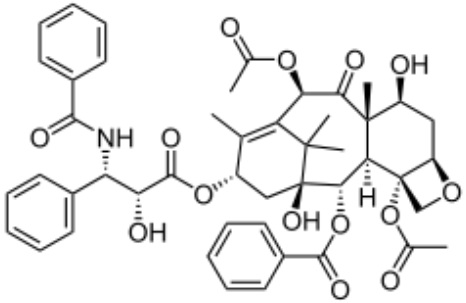


Fungal extract test sample



Test sample - Extracted ion chromatogram 854.3–854.5Da





Taxol (Paclitaxel)

ACE UltraCore SuperC18, 2.5µm, 150 x 2.1mm

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

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Microbial Extract by LC-MS

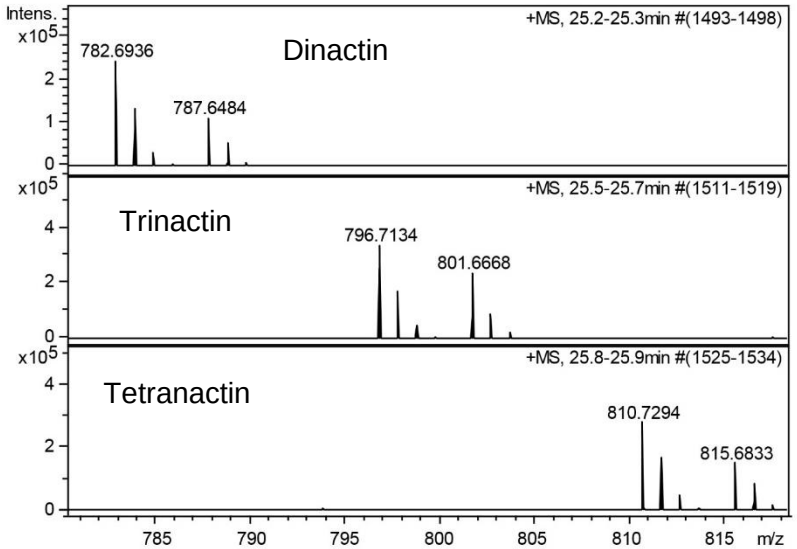
Dionex 3000RS UHPLC system coupled with Bruker MaXis Q-TOF MS

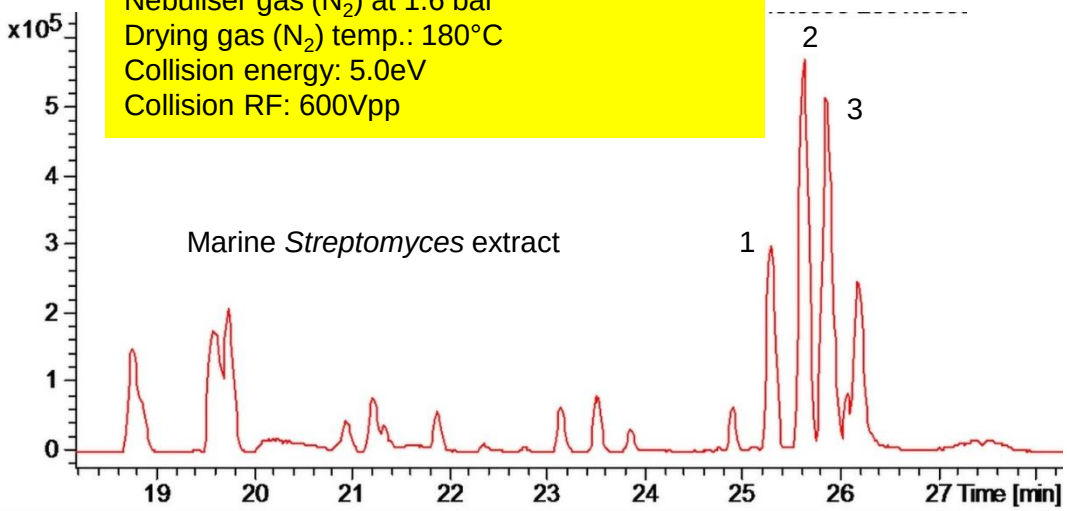
UltraCore SuperC18 2.5um, 150 x 2.1mm
Gradient analysis
A = 0.1% formic acid in water
B = 0.1% formic acid in acetonitrile

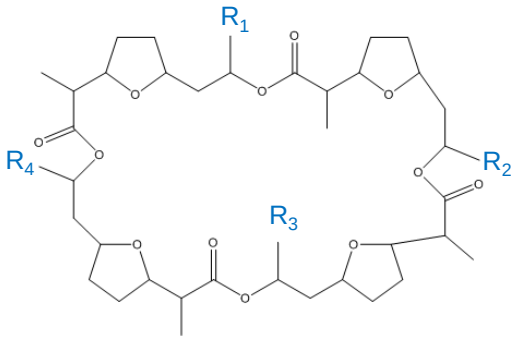
Time (mins)	%B	Time (mins)	%B
0	5	20	100
5	5	25	100

Flow rate: 0.2ml/min

Electrospray MS (positive mode)
Source: End plate offset: -500V,
Capillary: -4500V
Nebuliser gas (N_2) at 1.6 bar
Drying gas (N_2) temp.: 180°C
Collision energy: 5.0eV
Collision RF: 600Vpp







Macrotetralides

1. Dinactin $R_1 = R_3 = CH_2CH_3$, $R_2 = R_4 = CH_3$
2. Trinactin $R_1 = R_2 = R_3 = CH_2CH_3$, $R_4 = CH_3$
3. Tetranactin $R_1 = R_2 = R_3 = R_4 = CH_2CH_3$

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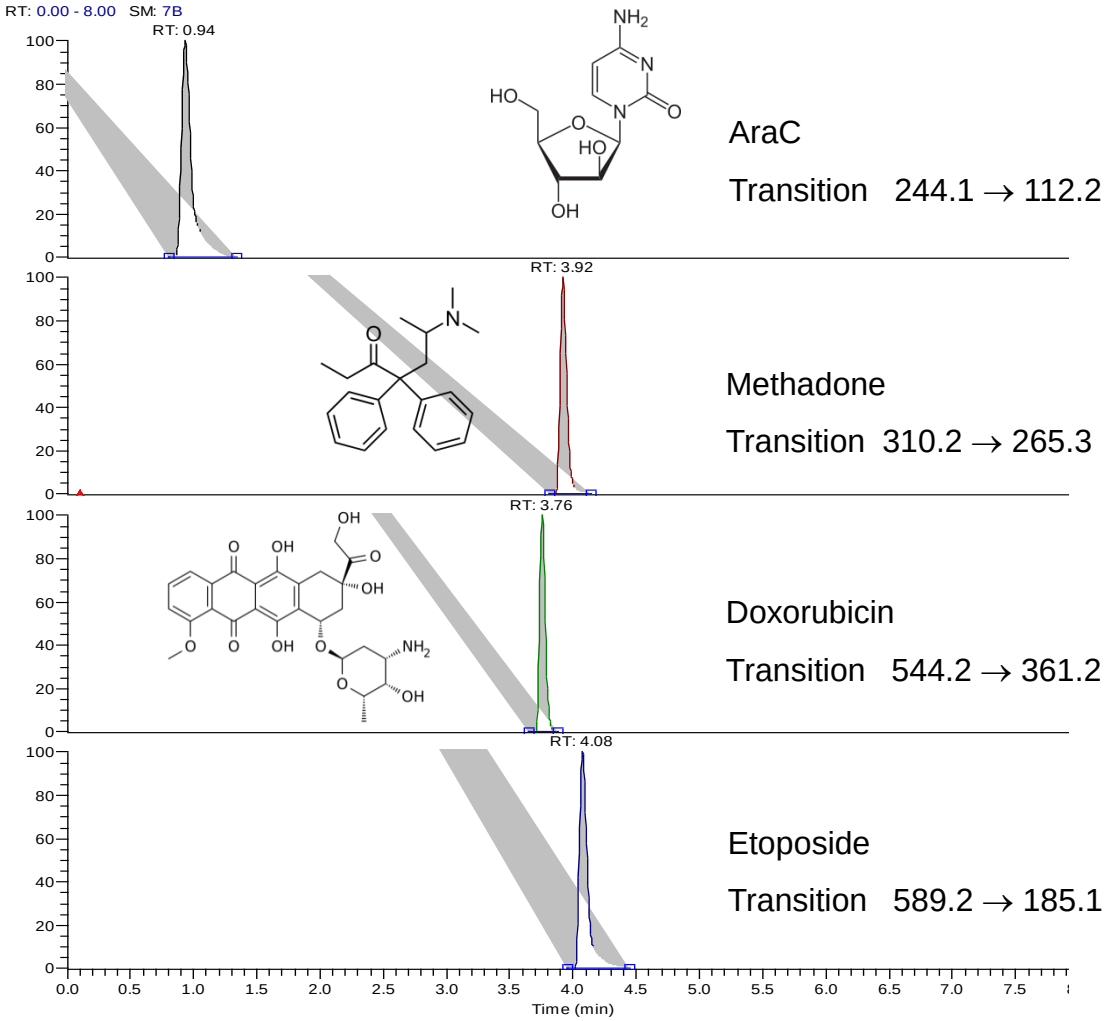
Cytotoxic Agents by UHPLC-MS/MS



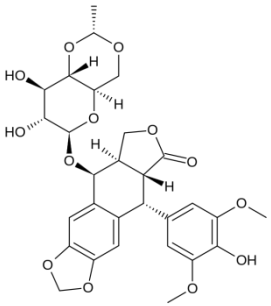
Thermo Scientific Accela UHPLC

ACE UltraCore SuperC18, 2.5µm, 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



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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_3CN

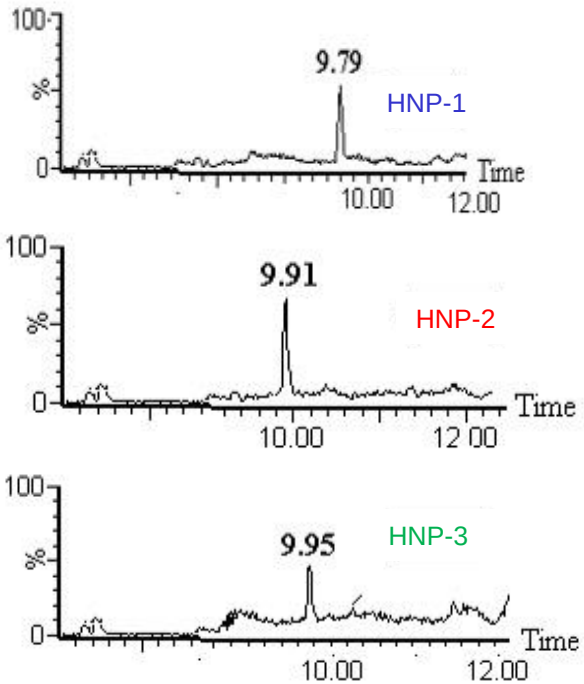
Time (mins)	%B
0	2
2	2
17	50
19	95
20	95

Flow rate: 0.6ml/min

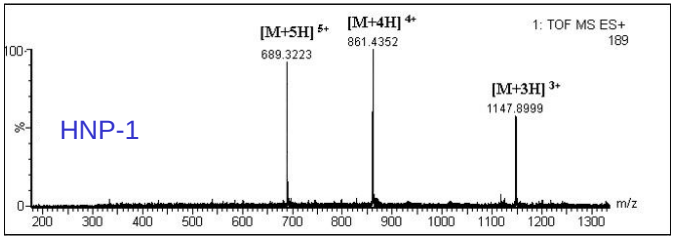
Synapt G1 QToF +ESI MS
Sampling cone voltage: 40V
Source temperature: 150°C
Capillary voltages: 4.8kV
Extraction cone voltages: 41kV
Desolvation temperature: 500°C
Acquisition: 100-2000 m/z

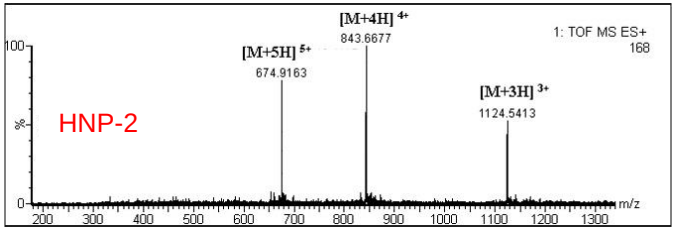
Extracted ion current chromatograms

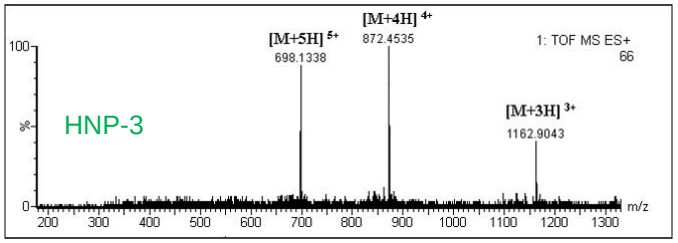
(sum of multiply protonated ions $[M+3H]^{3+}$, $[M+4H]^{4+}$ and $[M+5H]^{5+}$)

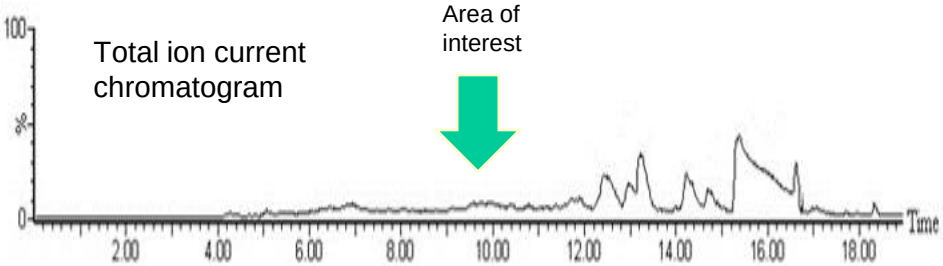


Mass spectra









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

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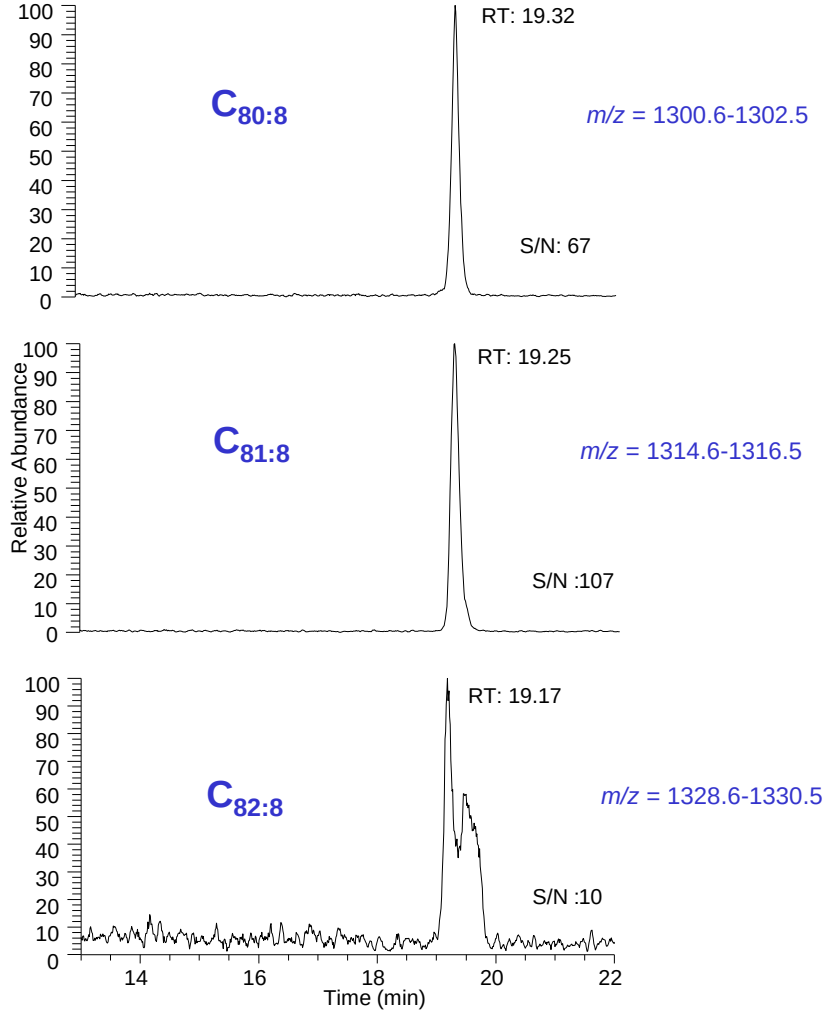
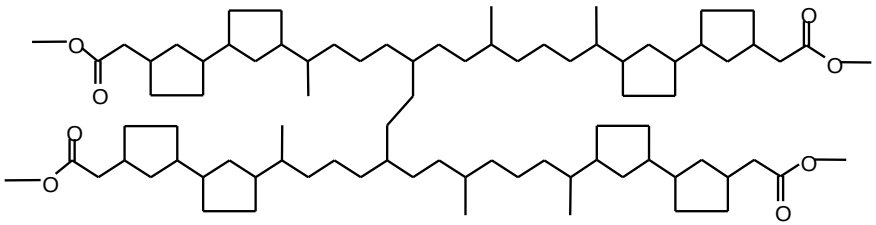
Polycyclic Tetracarboxylic Acids



C80-82 polycyclic tetracarboxylic acids isolated from oilfield deposits



Tetramethyl ester of $C_{80:8}$ ring acid

ACE UltraCore SuperPhenylHexyl 2.5µm, 100 x 2.1mm
Gradient analysis
A: CH_3OH – H_2O (98:2) containing 10mM ammonium acetate
B: IPA-H_2O (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 $[M+NH_4]+$
Detection limit ~ 0.1ppm

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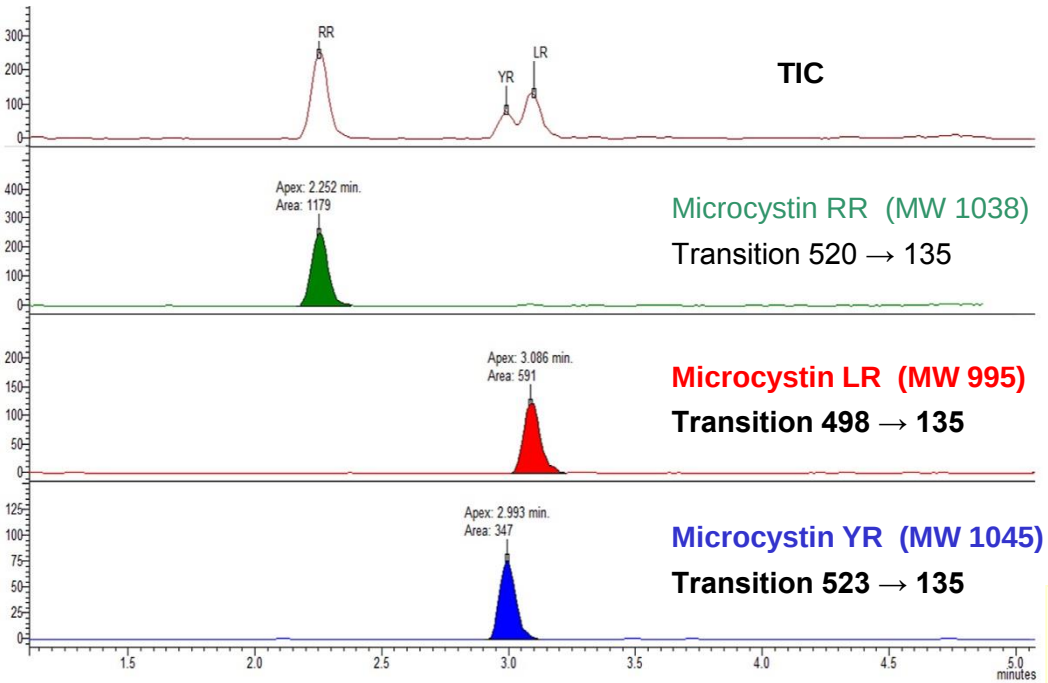
Microcystins From Blue/Green Algae In Drinking Water

ACE Excel 2 µm C18, 100 x 2.1 mm



Bruker Advance UHPLC system
ACE Excel 2µm C18, 100 x 2.1mm
Gradient elution
A = 0.1% formic acid in water
B = Acetonitrile

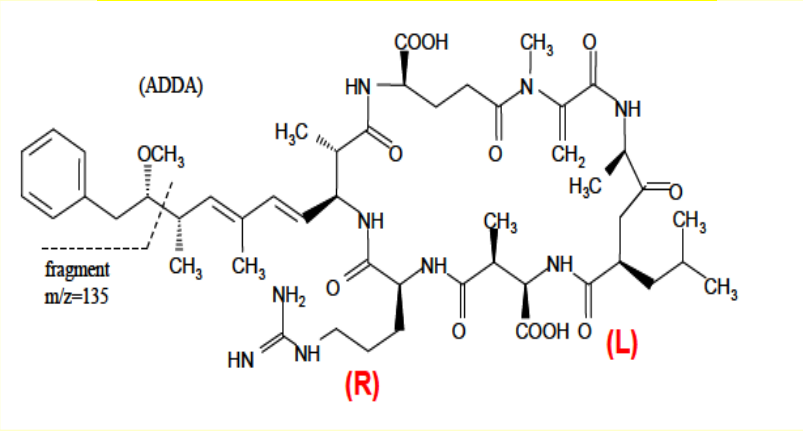
T (mins)	%B	T (mins)	%B
0	30	7.1	30
1	30	10	30
7	95		

Flow rate: 0.4mL/min
Column temperature: 40°C
Injection volume: 50µL
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

0.05 ppb each

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



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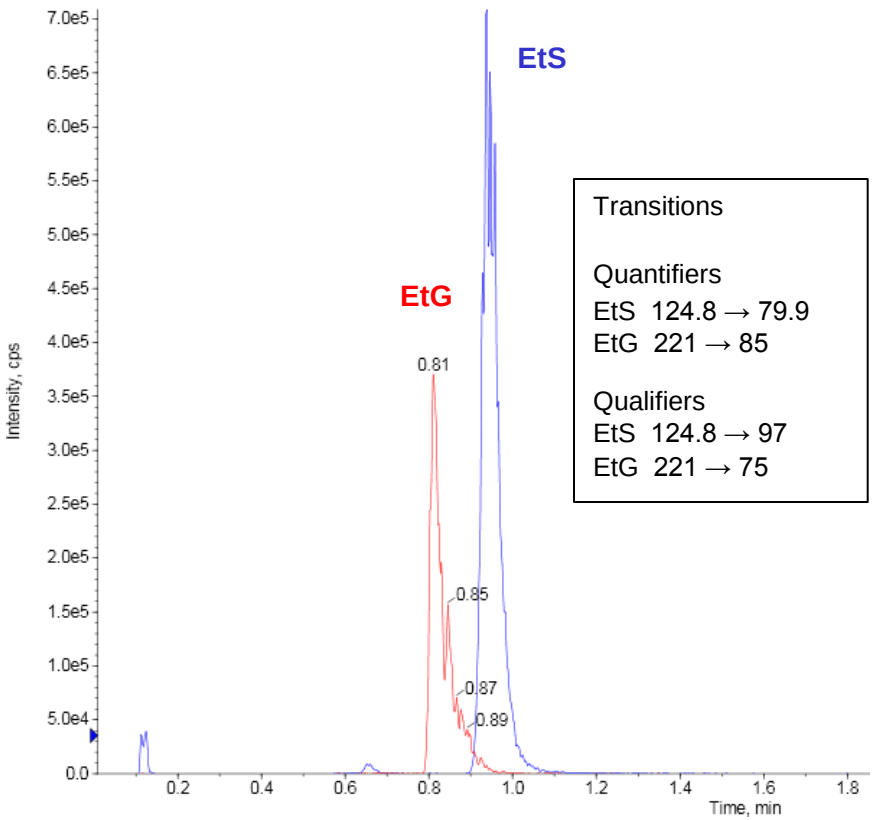
Alcohol Biomarkers by LC-MS/MS

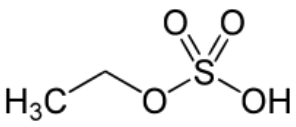
Extracted ion chromatogram



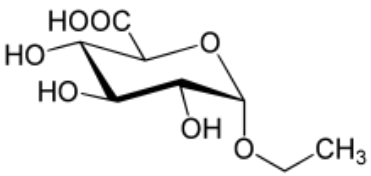
Transitions

Quantifiers
EtS 124.8 → 79.9
EtG 221 → 85

Qualifiers
EtS 124.8 → 97
EtG 221 → 75



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

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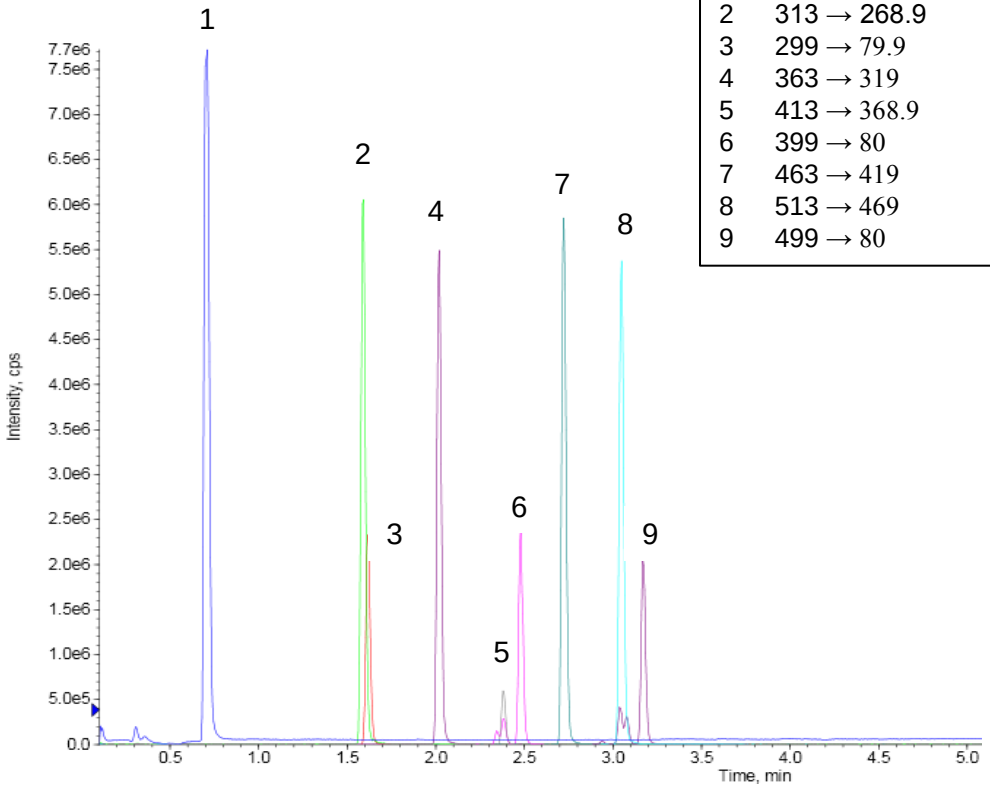
Perfluoro acids by LC-MS/MS



Transitions

Peak	
1	212.9 → 168.9
2	313 → 268.9
3	299 → 79.9
4	363 → 319
5	413 → 368.9
6	399 → 80
7	463 → 419
8	513 → 469
9	499 → 80

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 2µm, 50 x 2.1mm
Gradient analysis
A = 2mM NH_4OAc, 0.1% acetic acid/CH_3CN (95:5)
B = 2mM NH_4OAc, 0.1% acetic acid/CH_3CN (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: 20µl

AB SCIEX triple quad 5500
Negative ESI MRM
Source temperature: 450°C
IonSpray voltage: -2400V

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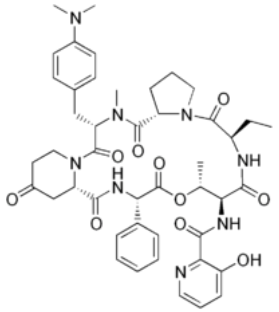
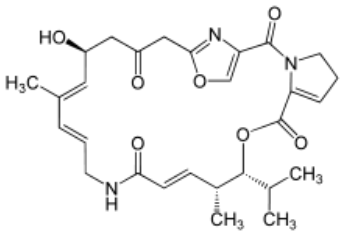
Pristinamycin components in plasma by LC-MS/MS

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

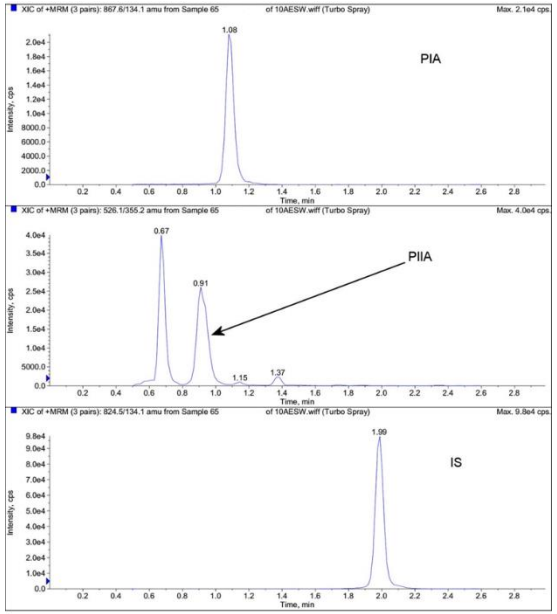


Pristinamycin IA

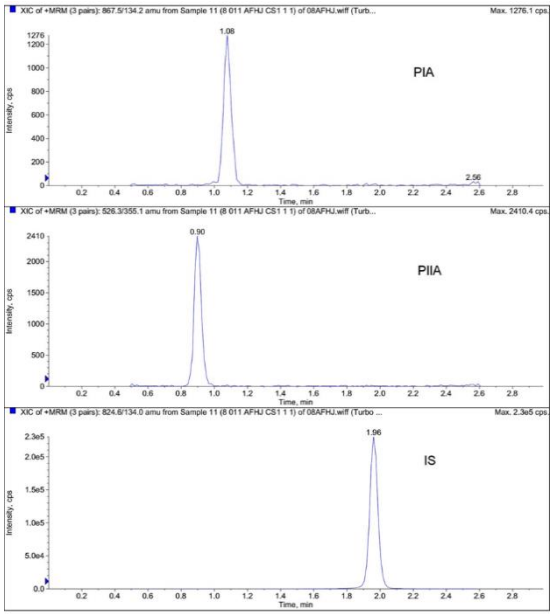


Pristinamycin IIA

Processed study sample containing pristinamycin IA and IIA



Low calibration standard containing 2.5ng/ml each of pristinamycin IA and IIA in human NaF/$K_2C_2O_4$ plasma



ACE 3 C18 3µm, 30 x 3.0mm
Gradient analysis
A = 1mM NH_4CO_2H + 0.1% HCO_2H in 65:35 H_2O:CH_3CN
B = CH_3CN

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

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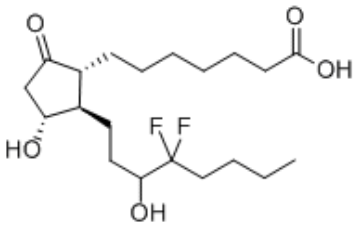
15-Hydroxy Lubiprostone in Human Plasma

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

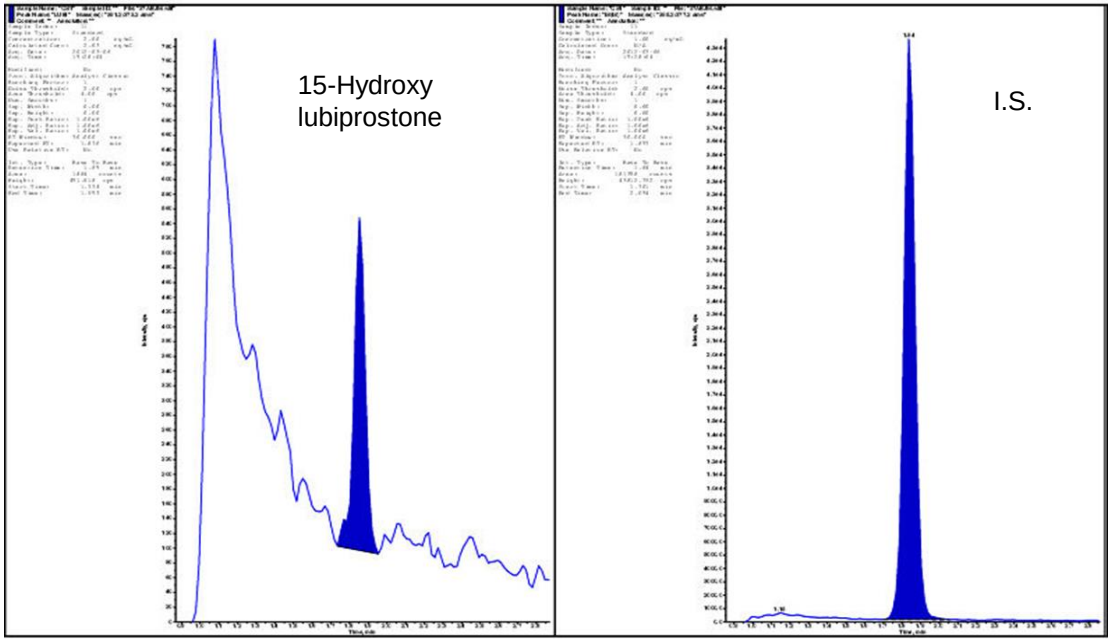
Quantitation is based on 15-hydroxy lubiprostone, with the d4 analogue as internal standard



15-Hydroxy lubiprostone

MW 392.5

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



ACE Excel 2 C18 2µm, 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: 15µl

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

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

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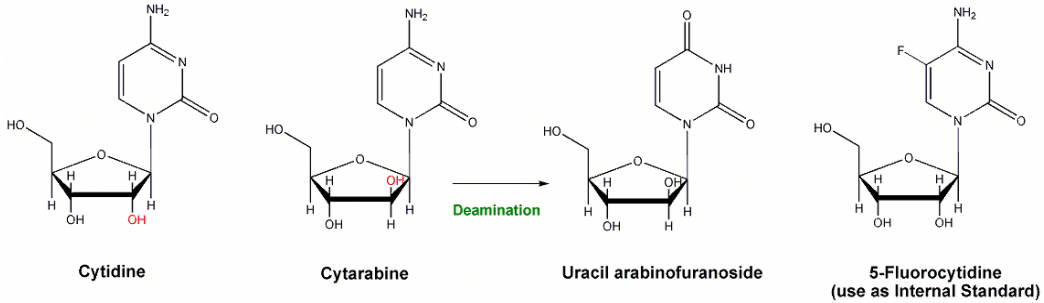
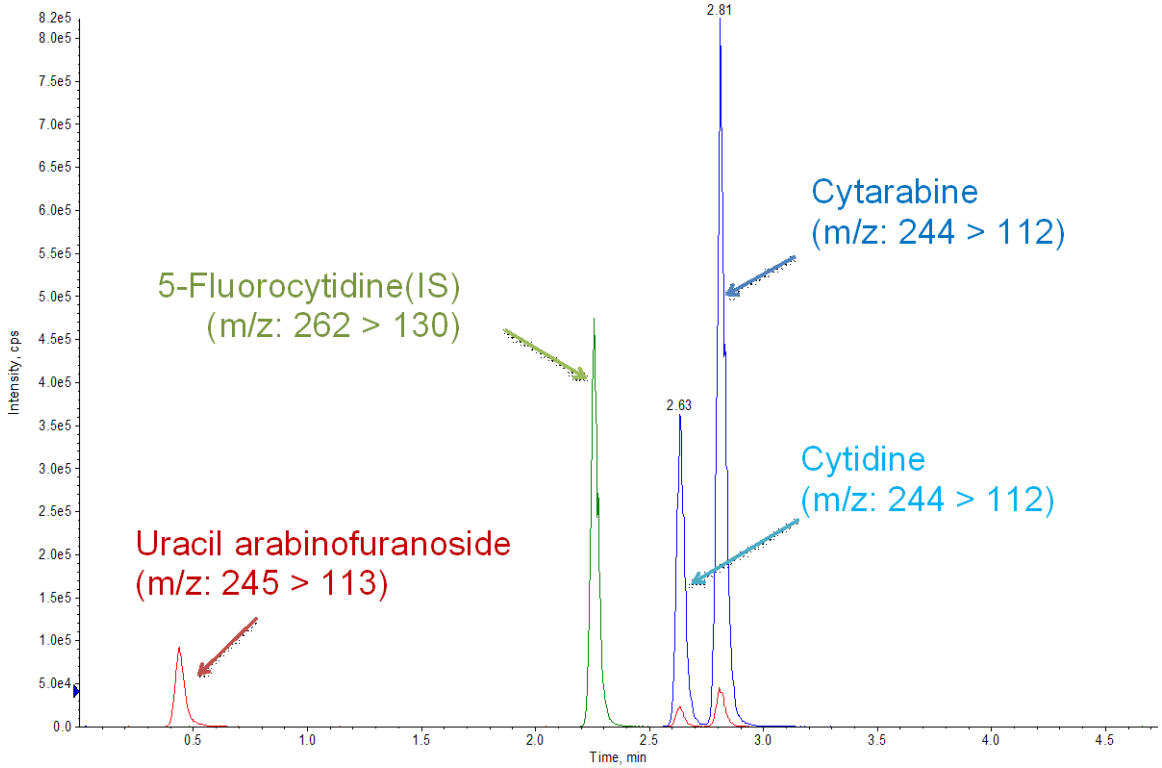
Cytarabine Analogues by Ion-Pairing LC-MS/MS



Cytarabine and cytidine are isobaric.

Robust method with good separation achieved.

LLOQ = 1 ng/ml human plasma



ACE 3 C18 3µm, 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
TurboIonSpray, positive mode
Source temperature: 550°C

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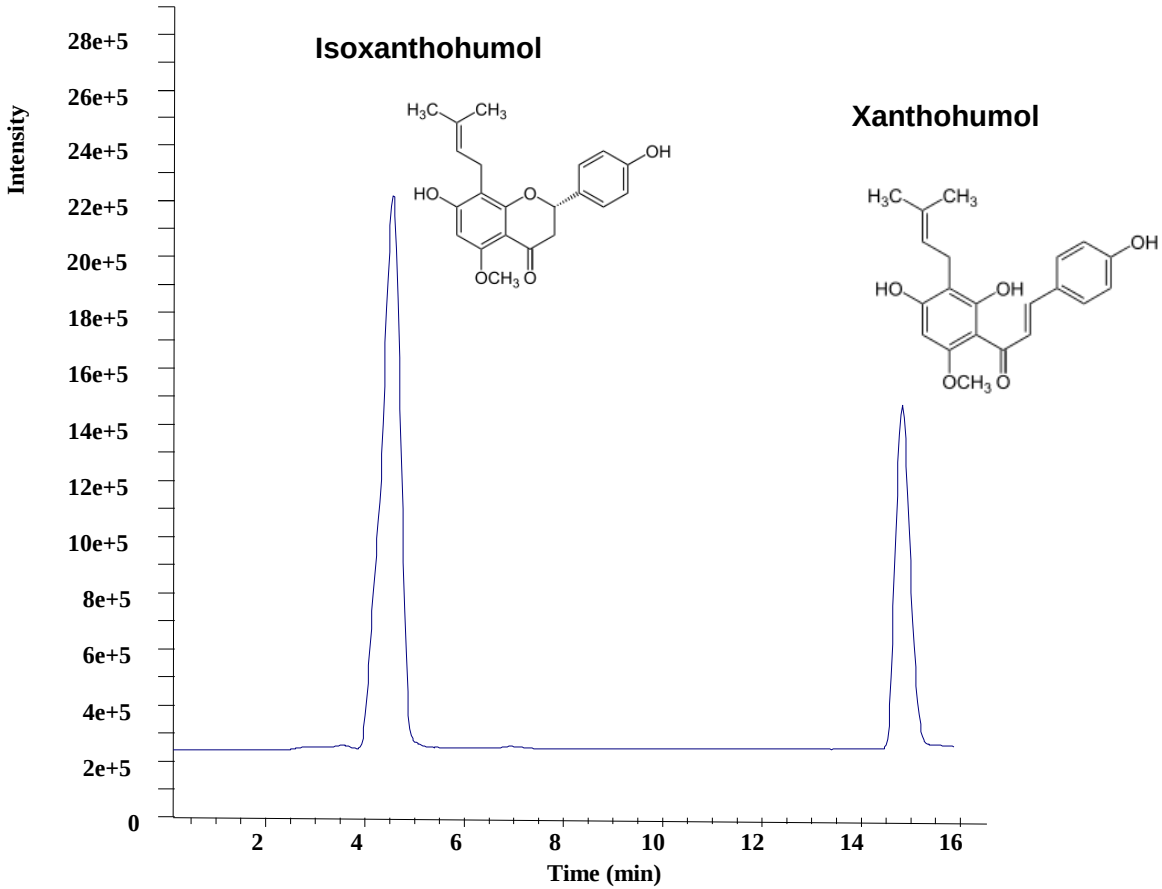
LC-MS/MS of phytoestrogens from hop extracts



Hop extracts obtained by pressurised liquid extraction using ASE 200 system

ACE C18-AR, 3µm, 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 $[M+H]^+$
MRM transition ions: 179 and 299
Collision energy: 28 and 16V

LOQ isoxanthohumol : 0.07µg/ml
LOQ: xanthohumol: 0.01µg/ml

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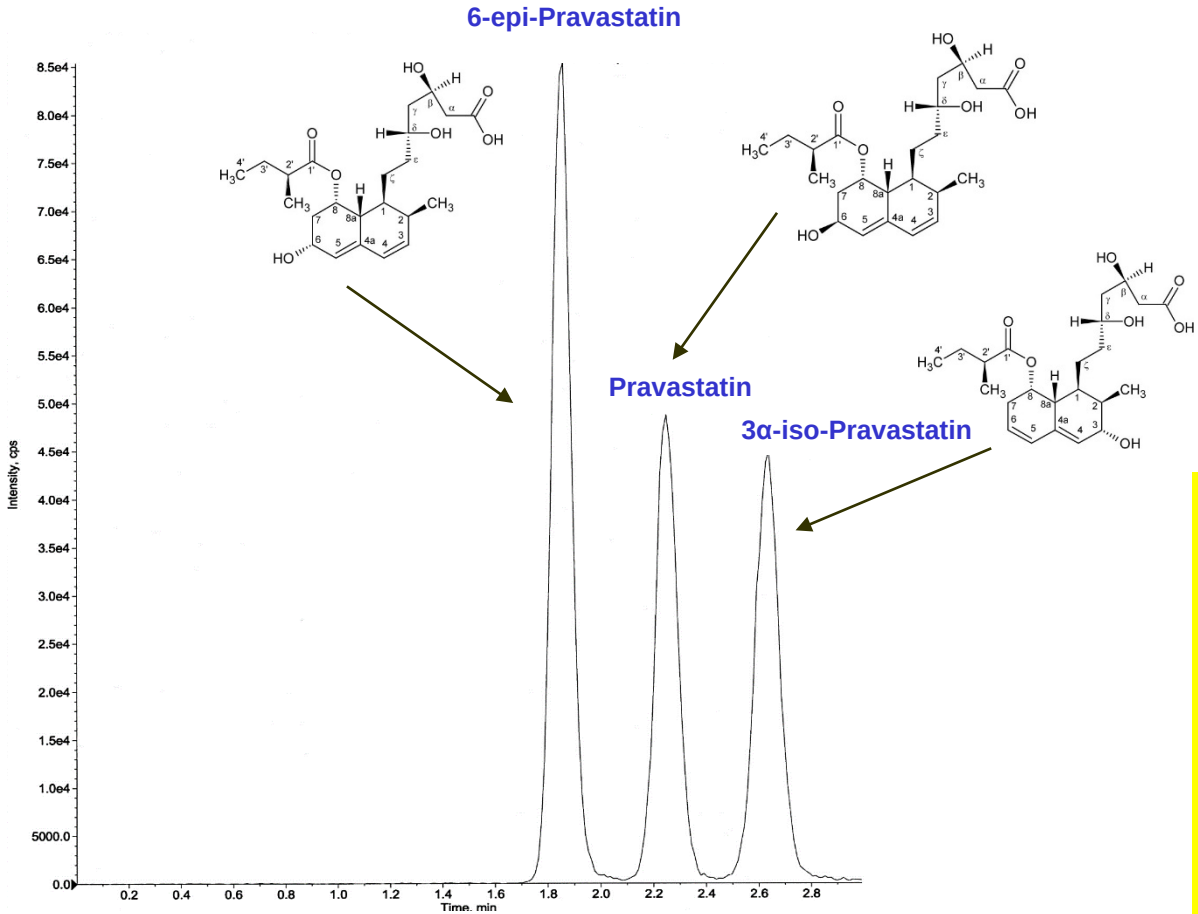
Pravastatin and Isomers by LC-MS/MS



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
TurboIonSpray – negative mode
Extracted ion chromatogram of MRM m/z 423.3 → 321.1

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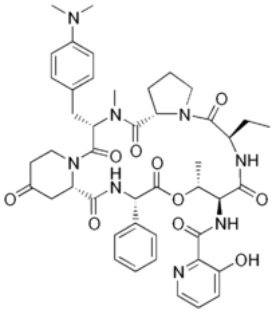
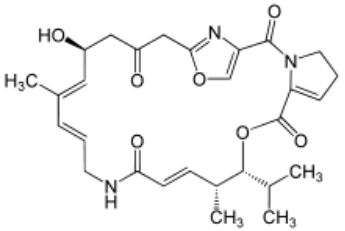
Pristinamycin components in plasma by LC-MS/MS

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

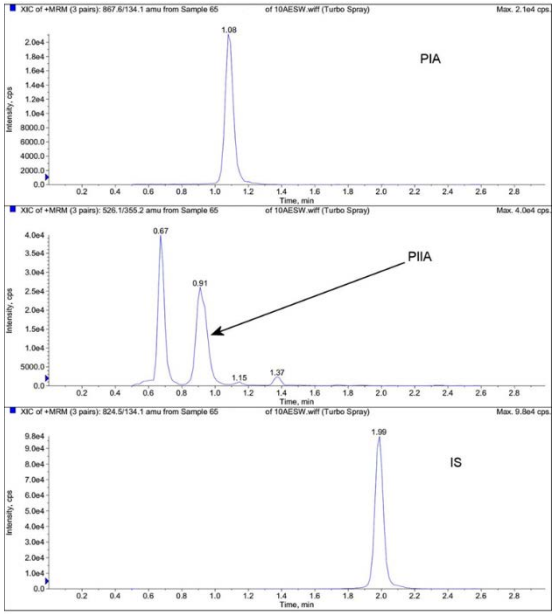


Pristinamycin IA

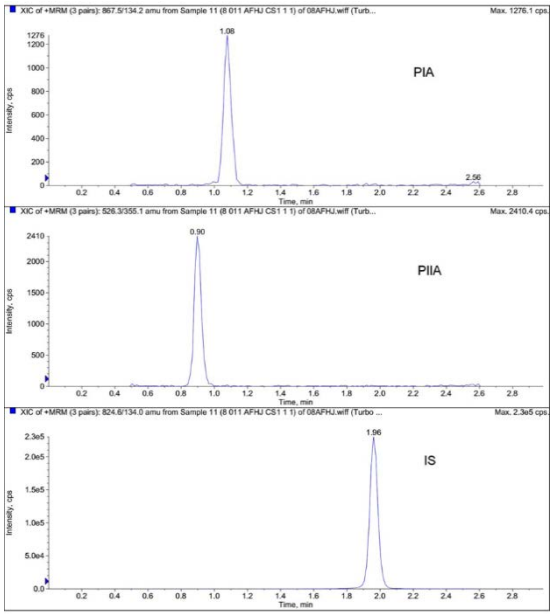


Pristinamycin IIA

Processed study sample containing pristinamycin IA and IIA



Low calibration standard containing 2.5ng/ml each of pristinamycin IA and IIA in human NaF/$K_2C_2O_4$ plasma



ACE 3 C18 3µm, 30 x 3.0mm
Gradient analysis
A = 1mM NH_4CO_2H + 0.1% HCO_2H in 65:35 H_2O:CH_3CN
B = CH_3CN

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

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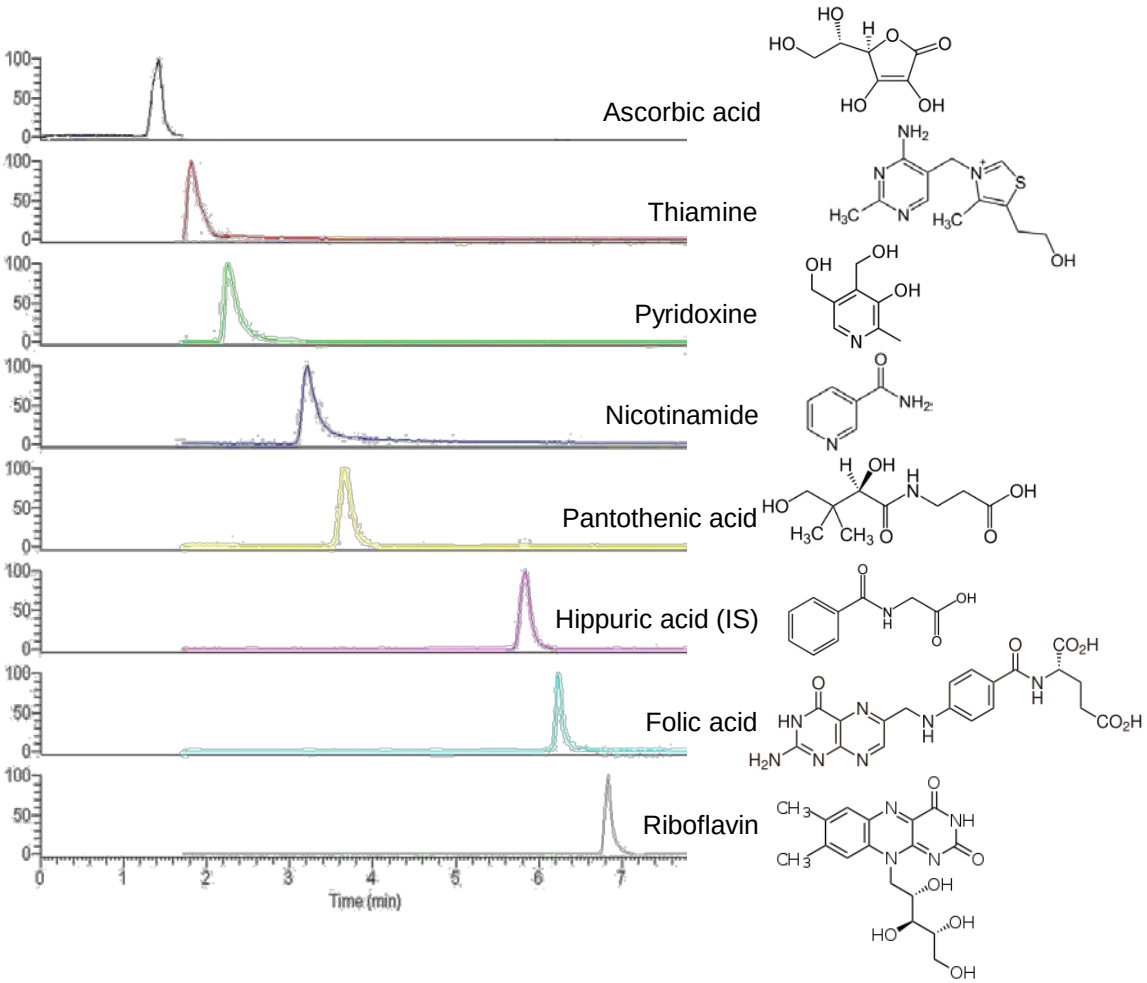
Water Soluble Vitamins In Green Vegetables By LC-MS/MS



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

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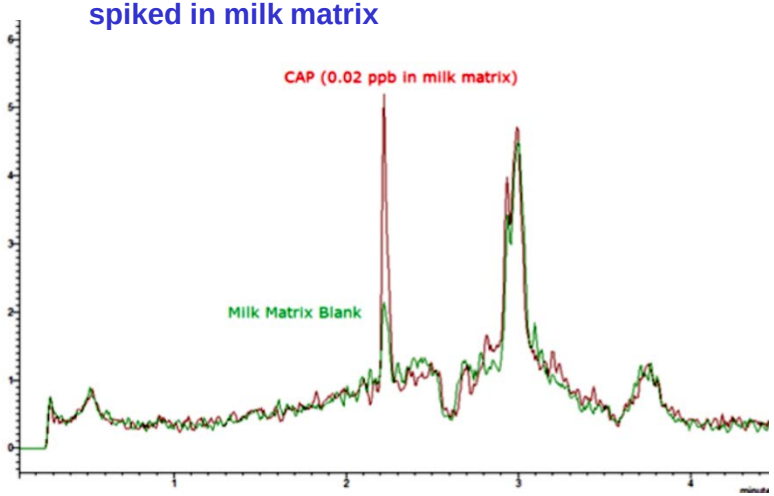
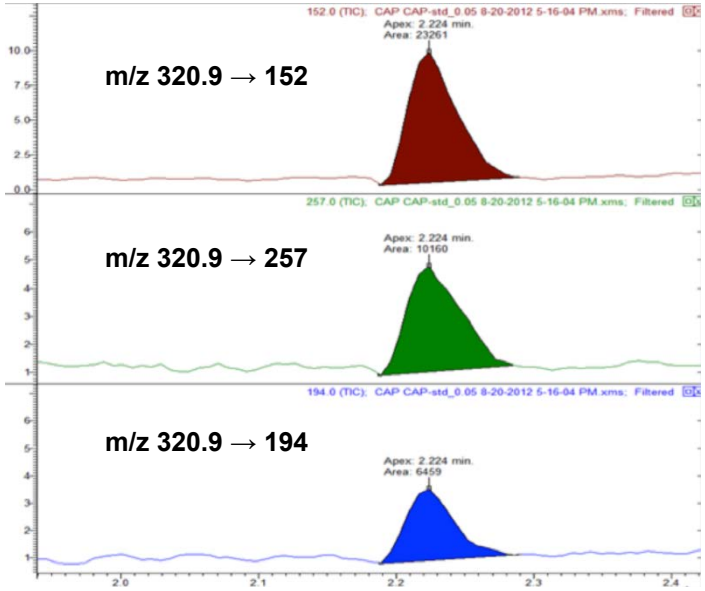
Chloramphenicol in Milk by LC-MS/MS

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



MRM chromatograms of 0.05 ppb chloramphenicol in milk

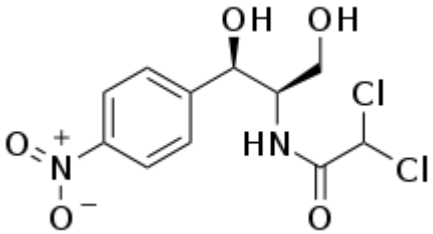


ACE C18 3µm, 50 x 2.1mm
Gradient analysis
A = Water
B = Methanol

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: 10µl

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

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

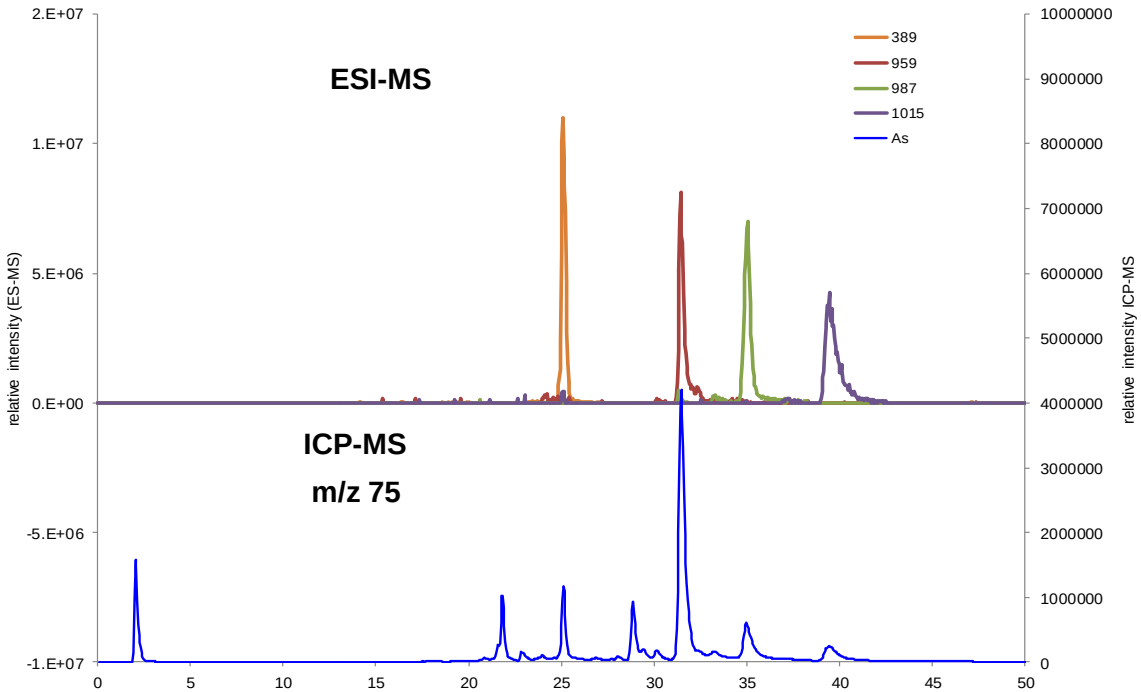
Separation of arsenic species from methanolic extract of the edible seaweed *Alaria esculenta*

Arsenic-containing hydrocarbon:

m/z 389 [M + H]+ for $C_{21}H_{46}AsO$

Arsenic-containing phospholipids:

m/z 959 [M + H]+ for $C_{45}H_{89}AsO_{14}P$ (C16:0/C16:0)
m/z 987 [M + H]+ for $C_{47}H_{93}AsO_{14}P$ (C18:0/C16:0)
m/z 1015 [M + H]+ for $C_{49}H_{97}AsO_{14}P$ (C20:0/C16:0)



ACE C18, 3µm 150 x 4.6mm
Gradient analysis
A = 0.1% formic acid in H_2O
B = 0.1% formic acid in CH_3OH

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

Flow rate: 1ml/min
Injection volume: 100µl
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

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