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

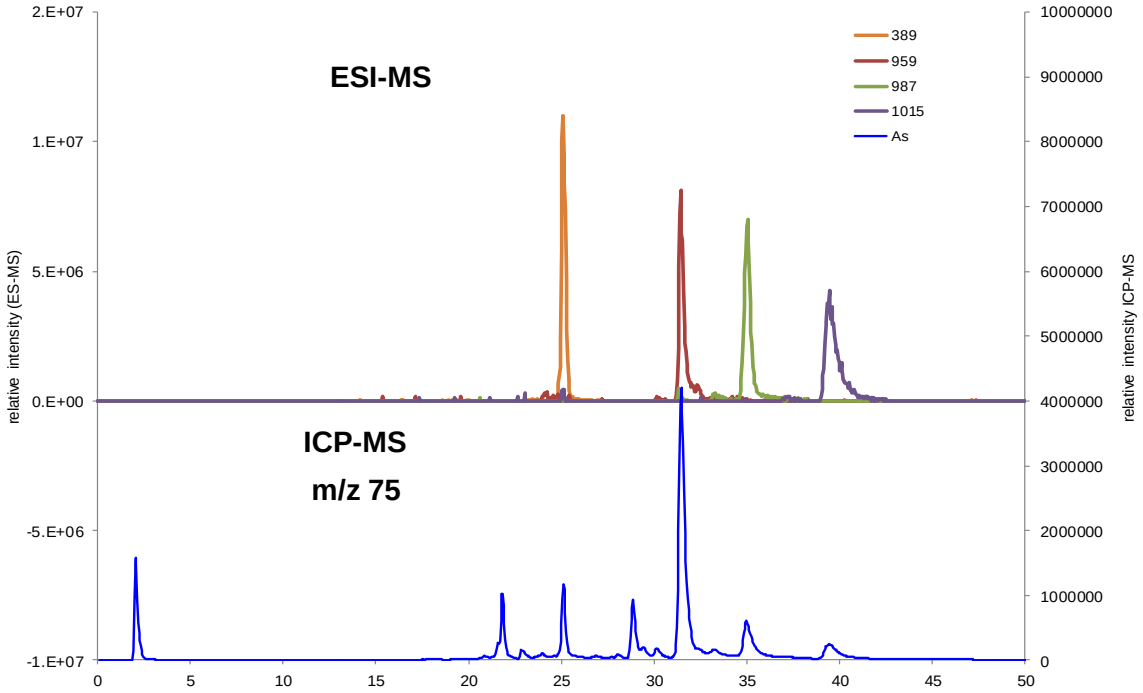
Separation of arsenic species from methanolic extract of the edible seaweed *Alaria esculenta*

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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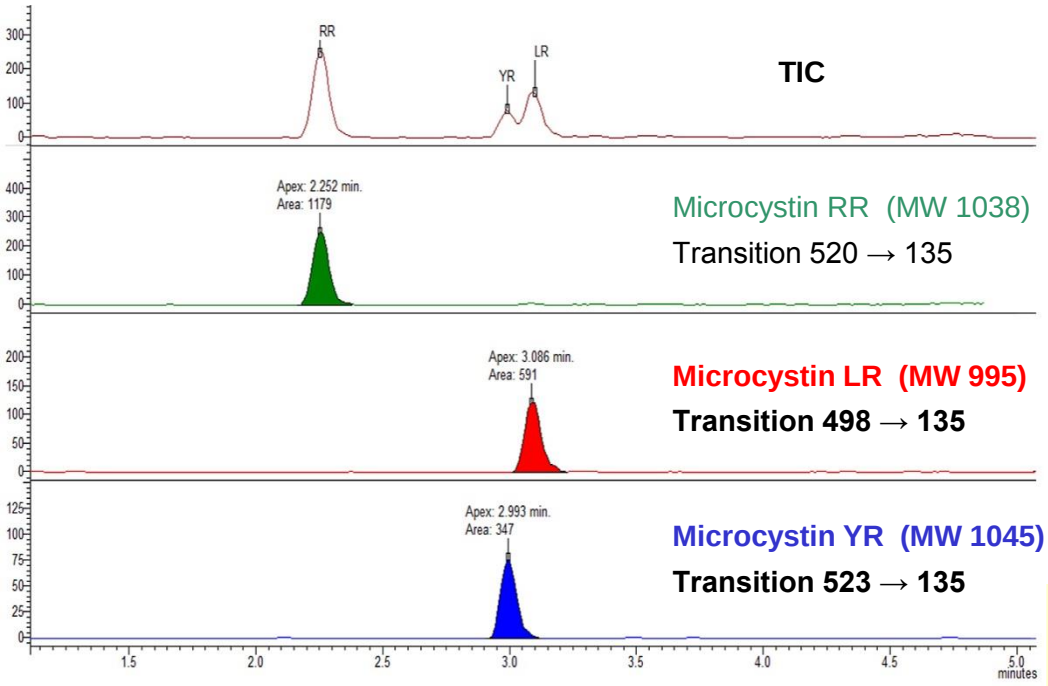
Microcystins From Blue/Green Algae In Drinking Water

ACE Excel 2 μm C18, 100 x 2.1 mm



0.05 ppb each

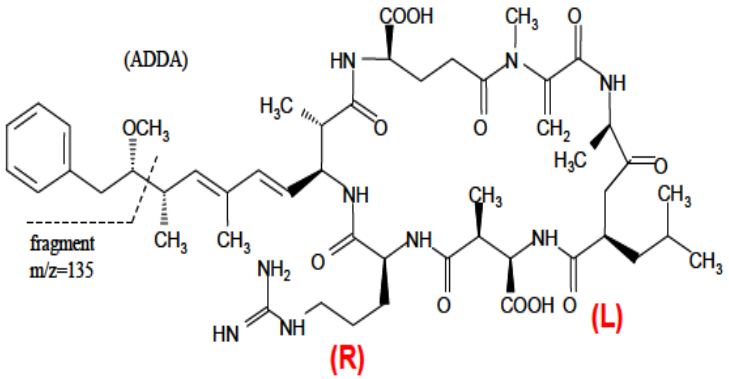
Variants	R	L
MC-LR	Leucine	Arginine
MC-RR	Arginine	Arginine
MC-YR	Tyrosine	Arginine

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

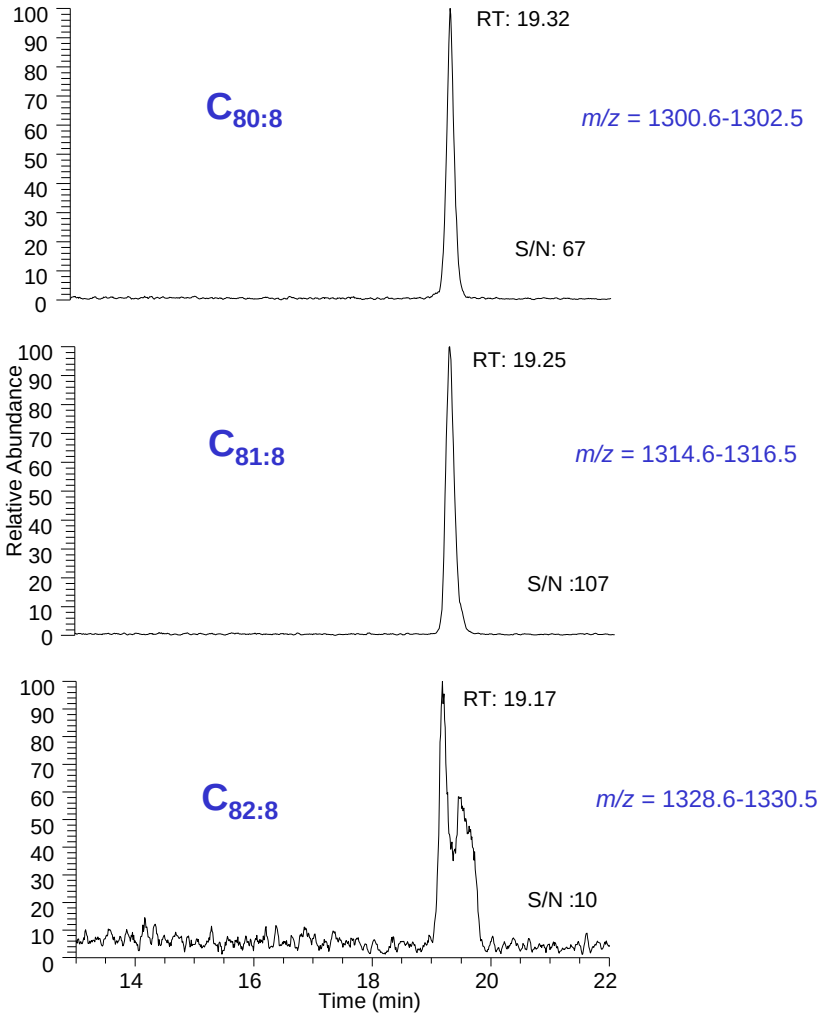
T (mins)	%B	T (mins)	%B
0	30	7.1	30
1	30	10	30
7	95		

Flow rate: 0.4mL/min
Column temperature: 40°C
Injection volume: 50μL
Concentration each microcystin: 0.05ppb

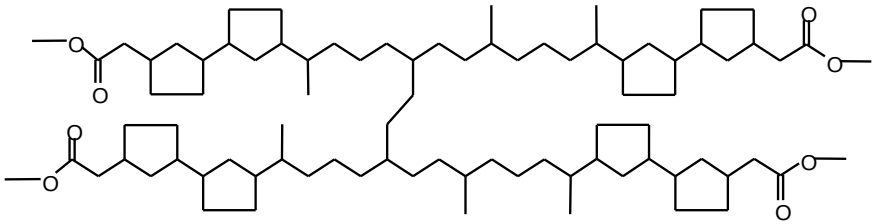
Bruker EVOQ Elite triple quad MS
VIP heated-ESI temperature: 350°C
Cone gas temperature: 200°C
Spray voltage: 4500V (+)
Collision gas: argon 1.5mTorr



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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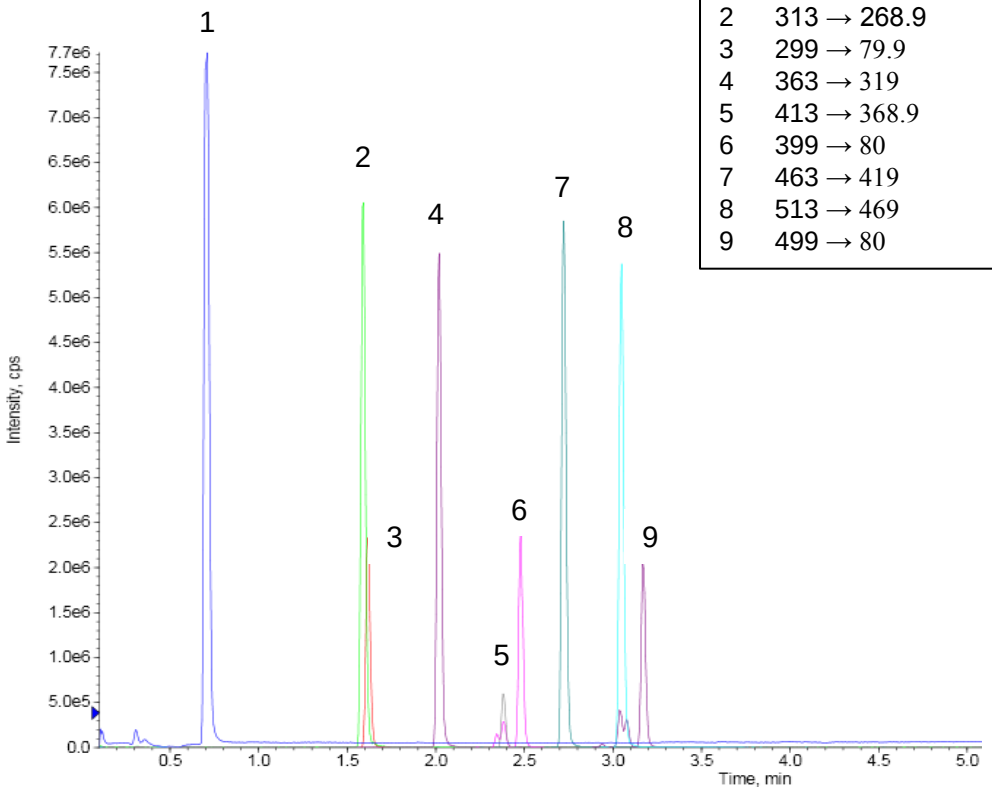
Perfluoro acids by LC-MS/MS



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

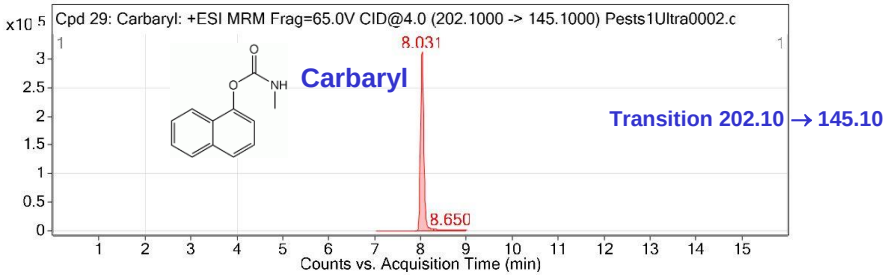
ACE Excel 2 C18 2µm, 50 x 2.1mm
Gradient analysis
A = 2mM NH_4OAc, 0.1% acetic acid/CH_3CN (95:5)
B = 2mM NH_4OAc, 0.1% acetic acid/CH_3CN (5:95)

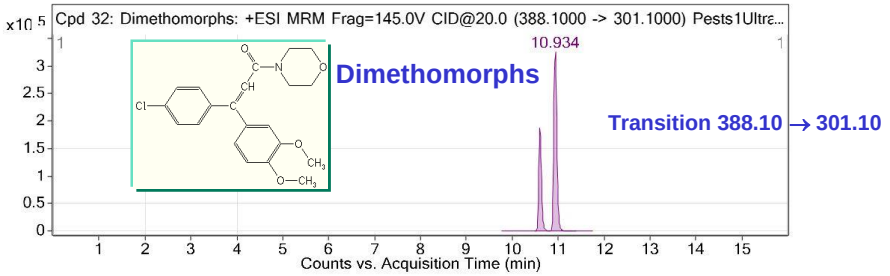
T (mins)	% B	T (mins)	%B
0	25	7.5	95
0.5	25	8.0	25
5.5	95	10.0	25

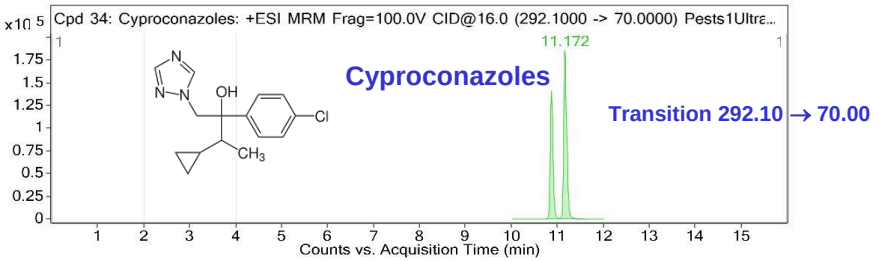
Flow rate: 0.5ml/min
Column temperature: 40°C
Injection volume: 20µl

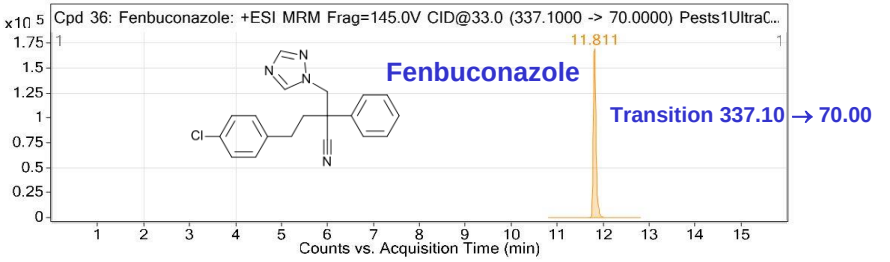
AB SCIEX triple quad 5500
Negative ESI MRM
Source temperature: 450°C
IonSpray voltage: -2400V

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

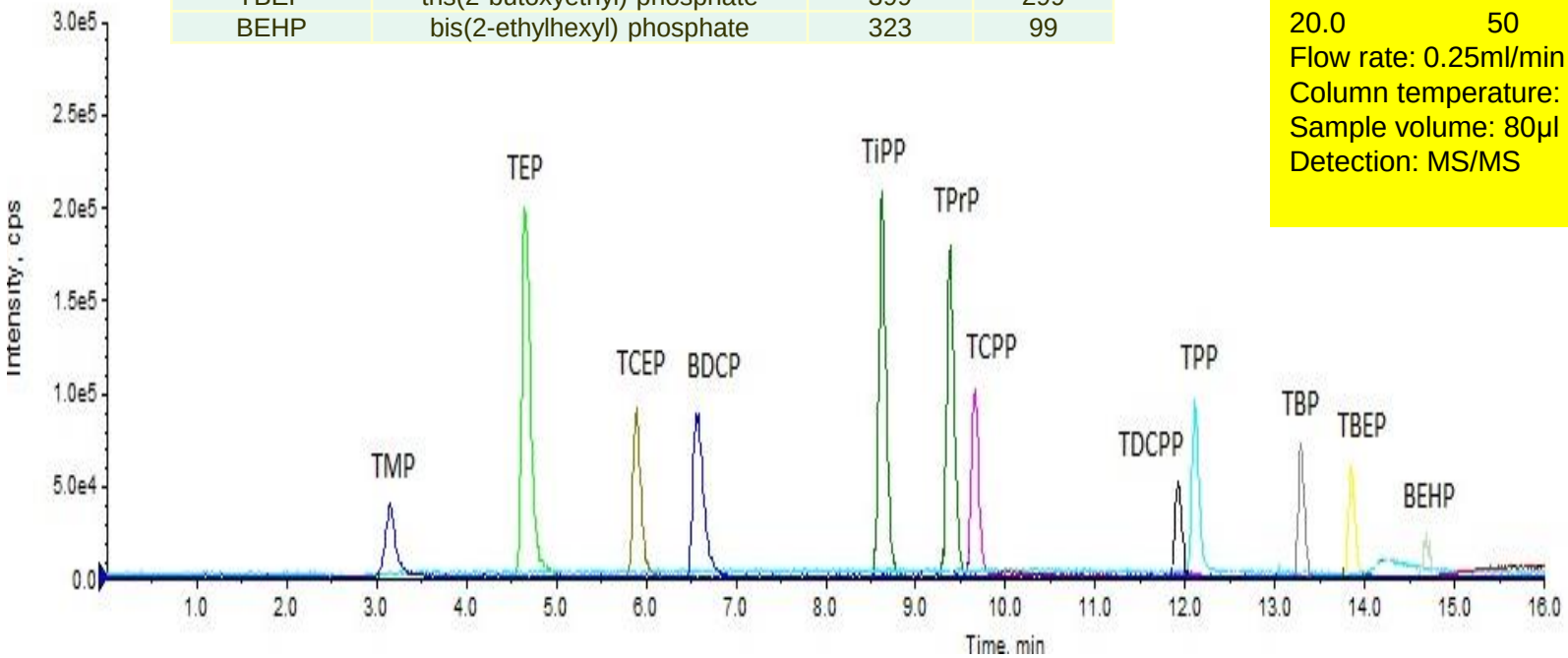
Organophosphorus Flame Retardants in Water by LC-MS/MS

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 3µm, 100 x 2.1mm
Gradient analysis
A = 0.05mM NH_4CO_2H + 0.005% HCO_2H in water
B = CH_3OH/CH_3CN (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: 80µl
Detection: MS/MS



ACE UltraCore SuperC18: Impurity Profile of a Herbicide

ACE UltraCore SuperC18, 2.5µm, 150 x 4.6mm
Gradient analysis
A = CH_3CN – H_2O – TFA (5:95:0.05 v/v/v)
B = CH_3CN – 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