

# OASIS SAMPLE PREPARATION

Application Notebook

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## WATERS OASIS FAMILY OF SAMPLE EXTRACTION PRODUCTS



The Oasis® family of sample extraction products is designed to simplify and improve your sample preparation by combining the right sorbent chemistry, device format and methodology. Achieve robust, selective, and sensitive solid-phase extraction [SPE] methods without worrying about low recoveries caused by breakthrough, sorbent drying, pH limitations, and undesirable silanol activity.

Oasis SPE sorbents—covered by nine U.S. patents\*—are unique in their purity, reproducibility, stability, and retention characteristics. Today, they are the most widely used polymeric SPE products in bioanalytical laboratories. Our ongoing goal for nearly three decades has been to provide innovative and flexible sample preparation tools for the separation scientist. In 1996, we introduced the first hydrophobic, yet water-wettable, polymeric SPE sorbent, Oasis HLB, designed for fast, generic, reversed-phase SPE. A year later, we introduced advanced, novel, 2-D methodologies using Oasis HLB to achieve cleaner, more selective extracts. In 1999, we introduced two mixed-mode strong ion-exchange chemistries—Oasis MCX and MAX—enabling highly selective and sensitive generic protocols for basic and

acidic compounds, respectively. In 2004, we introduced mixed-mode weak ion exchangers—Oasis WCX and WAX—to extract selectively strongly basic and strongly acidic compounds, respectively.

In 2005, we introduced the Oasis 2x4 Method, a simplified strategy for method development that provides a straightforward approach to the selection of the SPE sorbent and protocol. The Oasis 2x4 Method produces the cleanest extracts for analytic confidence by eliminating effects due to matrix interferences and sample variability.

We are committed to developing products and procedures that improve sample preparation performance. When it comes to device design and development, our goals are innovation and flexibility. Witness our award winning Oasis 96-well plate design\*\*, on-line Oasis SPE columns, and our innovative, patented Oasis μElution plate\*\*\* that, for the first time, enables SPE elution volumes as low as 25 μL.

\* U.S. patents 5,882,521; 5,976,367; 6,106,721; 6,254,780; 6,322,695; 6,468,422; 6,726,842; 6,723,236; 6,773,583

\*\* R&D 100 Award, 1999

\*\*\* U.S. Patent No. 6,723,236

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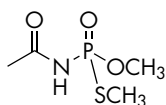
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### LC Conditions

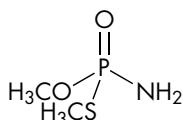
Column: Atlantis® dC<sub>18</sub>, 2.1 x 100 mm, 3 µm  
 Part Number: 186000295  
 Mobile Phase: 15% CH<sub>3</sub>OH in H<sub>2</sub>O  
 Flow Rate: 0.2 mL/min  
 Injection Volume: 20 µL  
 Temperature: 25 °C  
 Instrument: Waters Alliance® 2795

### MS Conditions

Instrument: Waters Quattro Ultima®  
 Ion Source: Positive Electrospray (ESI+)  
 Source Temperature: 150 °C  
 Desolvation Temperature: 450 °C  
 Cone Gas: 50 L/hr  
 Desolvation Gas: 500 L/hr  
 Collision Gas: Argon



Acephate



Methamidophos

Compound	MRM Transition (m/z)	Cone (V)	Coll Energy (eV)
Methamidophos	142 → 112	27	15
Acephate	184 → 143	20	10

### Acephate/Methamidophos Recovery Data

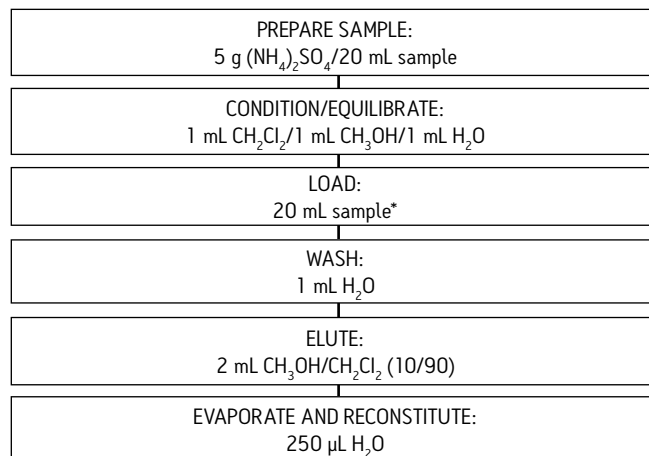
External standard calculation (n=5)

Methamidophos			Acephate		
Spike Level (ng/L)	Recovery* (%)	RSD (%)	Spike Level (ng/L)	Recovery* (%)	RSD (%)
50	50	25	50	92	12
200	62	9.8	200	91	5.4
400	66	12	400	90	15
800	60	6.8	800	105	12

\* Calculated against standard prepared in matrix. Matrix suppression was approximately 40%. Without Oasis HLB cleanup, matrix suppression was approximately 80%.

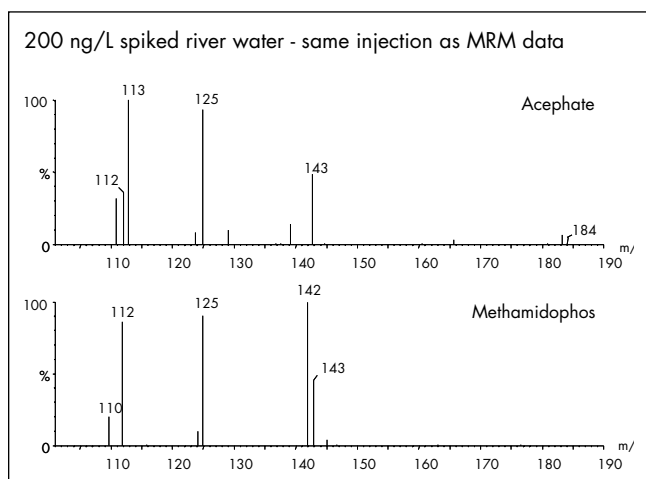
### Oasis HLB Extraction Method

Oasis® HLB Extraction Cartridge, 6 cc/200 mg  
 Part Number WAT106202

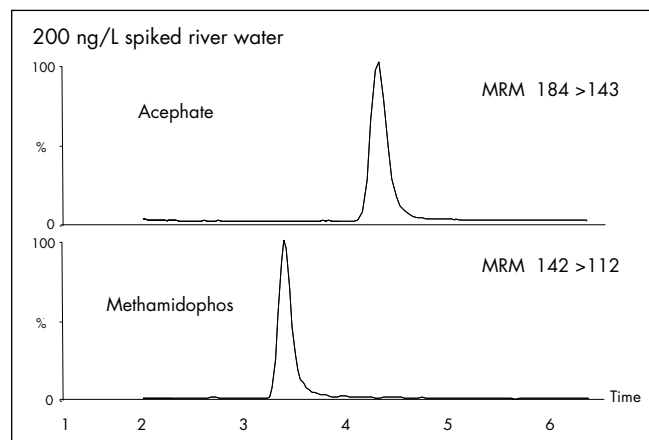


\* 25 mL maximum sample size

### Acephate/Methamidophos Daughter Ion Spectra



### LC/MS Chromatogram of Acephate/Methamidophos in River Water



## LC Conditions

Column: XTerra® MS C<sub>18</sub>, 2.1 x 100 mm, 3.5 µm  
 Part Number: 186000404  
 Mobile Phase A: 15 mM HCOONH<sub>4</sub>, pH 3.4  
 Mobile Phase B: CH<sub>3</sub>CN

Time (min)	Profile	
	A (%)	B (%)
Initial	75	25
9	40	60
14	40	60
30	10	90

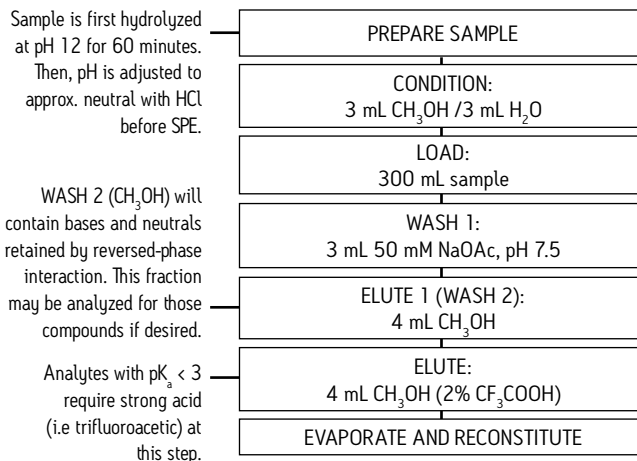
Flow Rate: 0.2 mL/min to MS  
 Injection Volume: 20 µL  
 Detection: Positive Electrospray (ESI)  
 Instrument: Waters Alliance® 2695  
 Detection: Waters ZQ™

Compound	RF (slope)	r <sup>2</sup>	LOQ	RSD (%)*
1. Picloram	3.7	0.999	300	16
2. Chloramben	1.8	0.989	200	9.3
3. 4-Nitrophenol	474	0.990	400	5.6
4. Bentazon	181	—	<100	6.1
5. 2,4-D	51	0.999	100	7.2
6. MCPA	43	0.980	200	9.2
7. 2,4,5-T	105	0.999	100	6.3
8. Dichlorprop	105	0.999	100	5.0
9. MCPP	136	0.992	100	7.0
10. Dichlorobenzoic acid	64	0.988	100	5.6
11. Acifluorfen	50	—	100	11
12. 2,4,5-TP	91	0.997	100	6.6
13. 2,4-DB	77	0.999	100	6.3
14. Dinoseb	>500	—	<100	10.7
15. Pentachlorophenol	67	0.998	100	11.7

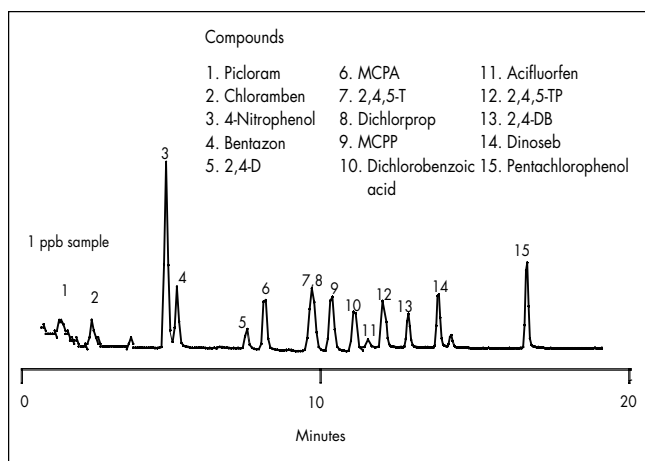
\*average of RSD from 4 levels

## Oasis MAX Extraction Method

Oasis® MAX Extraction Cartridge, 6 cc/150 mg  
 Part Number 186000369



## LC/MS Chromatogram of Acidic Herbicides in Drinking Water



### LC Conditions

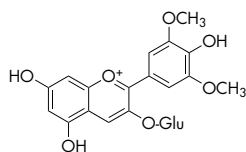
Column: XTerra® MS C<sub>18</sub>, 2.1 x 100 mm, 3.5 µm  
 Part Number: 186000404  
 Mobile Phase A: 20 mM KH<sub>2</sub>PO<sub>4</sub>/H<sub>3</sub>PO<sub>4</sub>, pH 3  
 Mobile Phase B: CH<sub>3</sub>OH  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	75	25
6	40	60

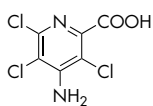
Flow Rate: 0.2 mL/min  
 Injection Volume: 10 µL  
 Detection: UV @ 221 nm

Compound	Recovery (%)
1. Picloram	95 (1)
2. Chloramben	98
3. Dicamba	100
4. Bentazon	102
5. 2,4-D	85
6. MCPA	88

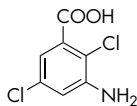
1 = pigment interference



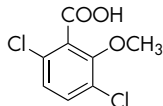
Typical Grape Pigment (malvidin-3-glucoside)



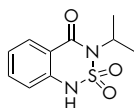
Picloram



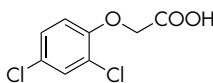
Chloramben



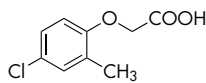
Dicamba



Bentazon

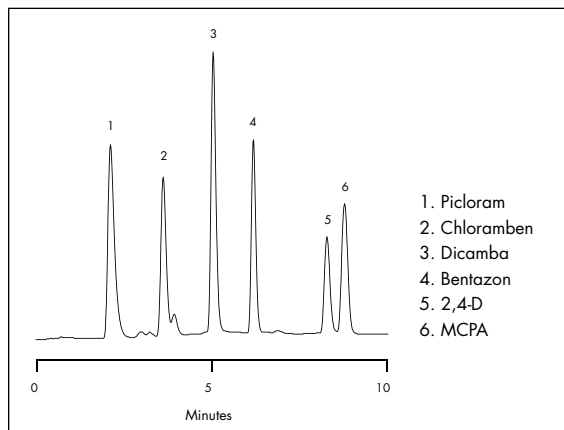


2,4-D



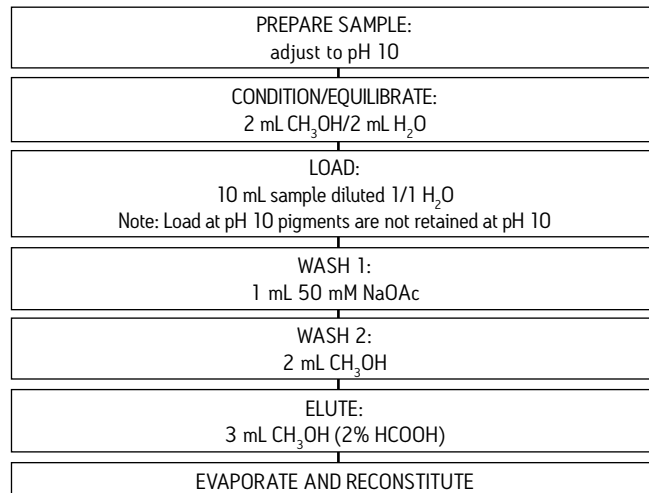
MCPA

### HPLC Chromatogram for Non Extracted Standard

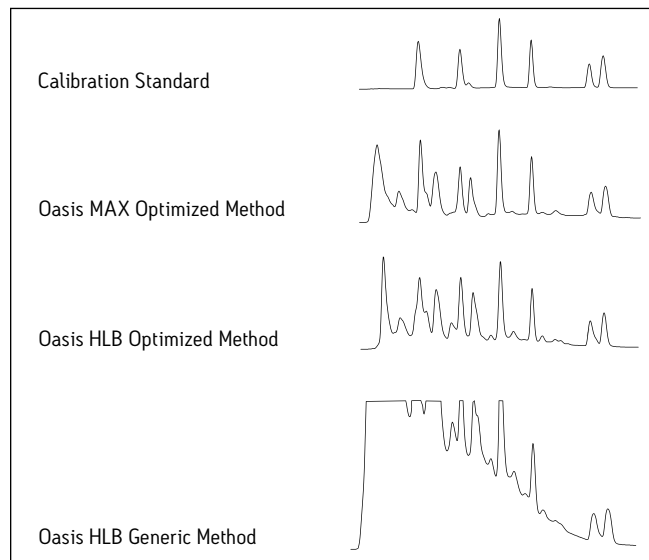


### Oasis MAX Extraction Method

Oasis® MAX Extraction Cartridge, 6 cc/150 mg  
 Part Number 186000369



### Comparison of SPE Cleanup Protocols



## LC/MS Conditions

Column: XTerra<sup>®</sup> MS C<sub>18</sub>, 2.1 x 100 mm, 3.5 μm  
 Part Number: 186000404  
 Mobile Phase A: 20 mM CH<sub>3</sub>COONH<sub>4</sub>, pH 4  
 Mobile Phase B: CH<sub>3</sub>OH  
 Gradient: 50% A initial to 90% CH<sub>3</sub>OH in 10 min  
 Flow Rate: 175 μL/min  
 Injection Volume: 10 μL  
 Instrument: Waters Alliance<sup>®</sup> Separations Module with 2996 PDA. Results obtained using negative electrospray MS, 37 V cone voltage, Waters ZQ<sup>™</sup>

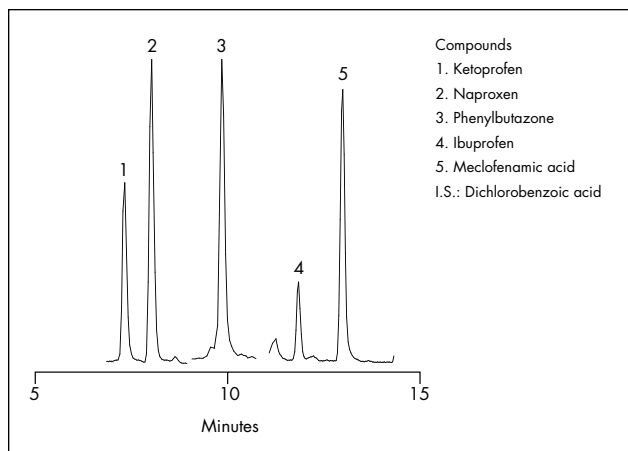
## LC/UV Conditions

Column: XTerra MS C<sub>18</sub>, 4.6 x 100 mm, 3.5 μm  
 Part Number: 186000436  
 Mobile Phase A: 0.1% CH<sub>3</sub>COOH  
 Mobile Phase B: CH<sub>3</sub>OH  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	50	50
2	50	50
20	40	60
30	10	90

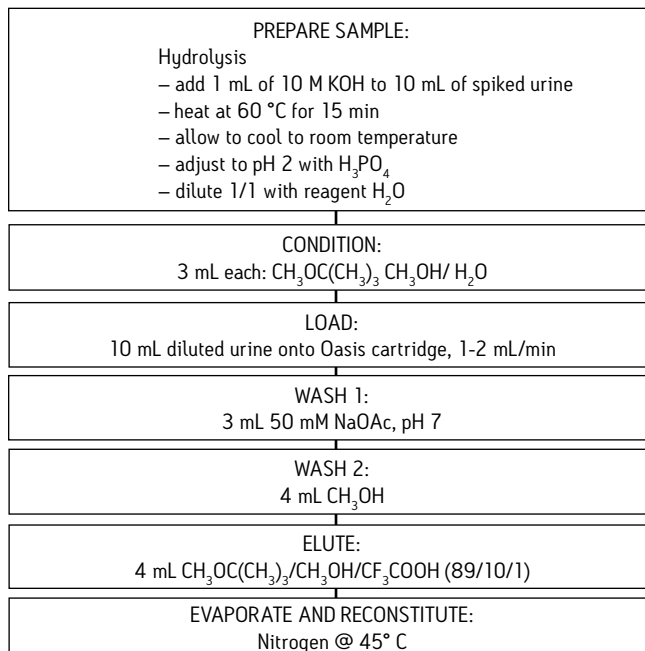
Flow Rate: 1 mL/min  
 Injection Volume: 40 μL  
 Detection: UV @ 221nm  
 Instrument: Waters Alliance 2695  
 Detection: 2996 PDA

## Reconstructed TIC Chromatogram of Acidic Veterinary Drugs in Horse Urine



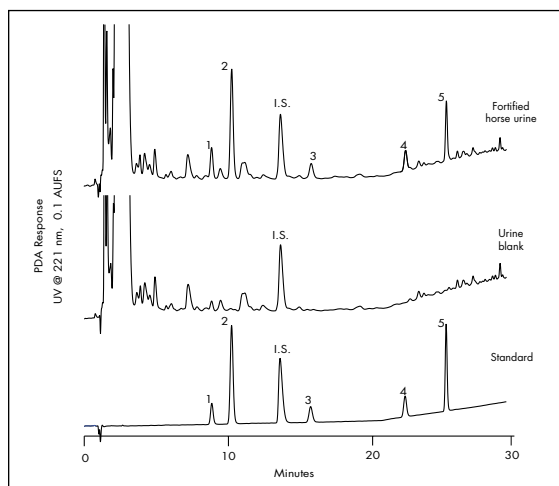
## Oasis MAX Extraction Method

Oasis<sup>®</sup> MAX Extraction Cartridge, 6 cc/150 mg  
 Part Number 186000368



Compound	0.1 ppm	0.4 ppm	2.0 ppm	10.0 ppm	r <sup>2</sup>
Ketoprofen	4804(4.7)	26389(11)	129366(9.6)	525903(8.1)	0.998
Naproxen	7621(8.8)	40234(23)	231980(12)	875595(7.3)	0.995
Phenylbutazone	778(7.8)	4252(37)	39387(15)	207163(5.6)	0.999
Ibuprofen	820(6.1)	3739(14)	23489(7.9)	127731(5.7)	0.999
Meclofenamic Acid	2070(11)	9531(23)	38822(11)	—	0.998

## LC/PDA Chromatogram of Acidic Veterinary Drugs, 2 ppm



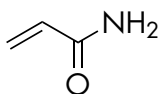


**LC Conditions**

Column: Atlantis® dC<sub>18</sub>, 2.1 x 150 mm, 5 µm  
 Part Number: 186001297  
 Mobile Phase: 0.1% HCOOH in H<sub>2</sub>O  
 Flow Rate: 0.2 mL/min  
 Injection Volume: 20 µL  
 Temperature: 30 °C  
 Instrument: Waters Alliance® 2695

**MS Conditions**

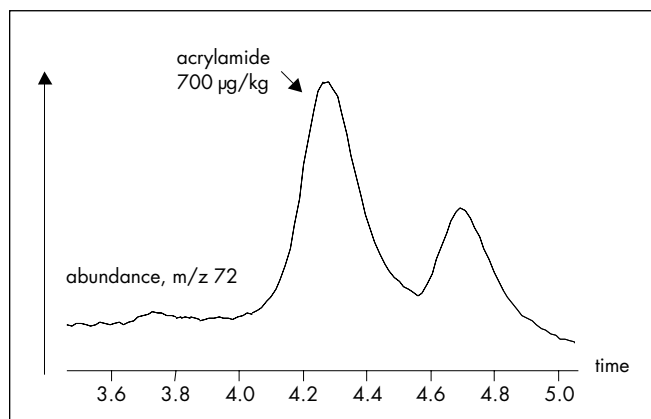
Instrument: Waters ZQ™  
 Ion Source: Positive Electrospray (ESI+)  
 Mode: Selected-ion recording  
 Source Temperature: 150 °C  
 Desolvation Temperature: 350 °C  
 Desolvation Gas Flow: 500 L/hr



Acrylamide

Internal standard calculation (n=5)		
Fortification Level (µg/kg)	Amount Found (µg/kg)	RSD (%)
100	96	12
200	211	8.7
500	488	5.8
1000	1010	8.0
2000	2000	6.5

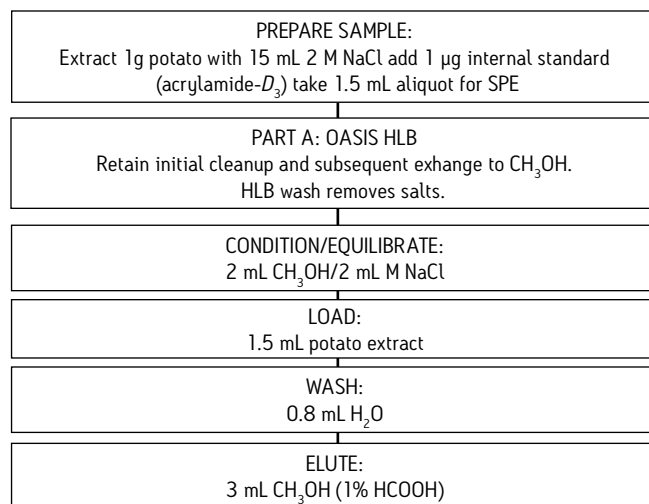
**LC/MS Chromatogram of Acrylamide in Actual Potato Chip Sample**



**Oasis Extraction Method**

Oasis® HLB Extraction Cartridge, 6 cc/200 mg  
 Part Number WAT106202  
 Oasis MCX Extraction Cartridge, 3 cc/60 mg  
 Part Number 186000254

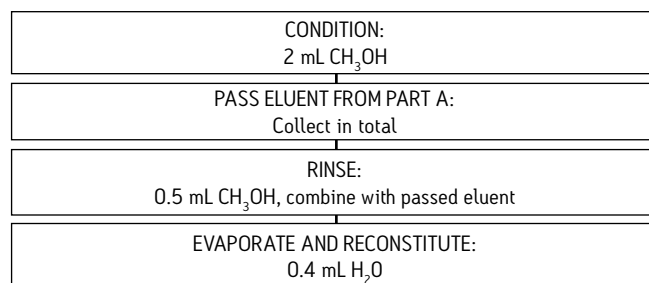
**PART A: Oasis HLB**



Proceed to Part B

**PART B: Oasis MCX**

Ion-exchange cleanup of methanol extract. MCX  
 cleanup removes food matrix



### LC Conditions

Column: ACQUITY UPLC<sup>®</sup> BEH C<sub>18</sub>,  
2.1 x 50 mm, 1.7 μm  
Mobile Phase A: 0.1% HCOOH in H<sub>2</sub>O  
Mobile Phase B: 0.1% HCOOH in CH<sub>3</sub>OH  
Flow Rate: 0.3 mL/min  
Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	95	5
1	95	5
3	20	80
4	20	80
4.5	95	5
6	95	5

Injection Volume: 10.0 μL  
Column Temperature: 4 °C  
Sample Temperature: 10 °C  
Instrument: Waters ACQUITY UPLC System

### MS Conditions

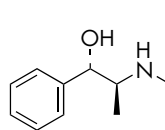
Instrument: Waters Quattro Premier™  
Ion Source: Positive and Negative Electrospray  
(ESI<sup>+</sup> and ESI<sup>-</sup>)  
Capillary: 3 kV  
Source Temperature: 100 °C  
Desolvation  
Temperature: 350 °C  
Cone Gas Flow: 0 L/hr  
Desolvation Gas Flow: 700 L/hr  
Collision Cell Pressure: 2.59 e-3 mbar

Compound	Precursor Ion (m/z)	Product Ion (m/z)	Cone Voltage (V)	Collision Energy (eV)
Ibuprofen (ESI <sup>-</sup> )	205.1	161.1	20	12
Pseudoephedrine (ESI <sup>+</sup> )	166.2	117	30	20
Chlorpheniramine (ESI <sup>+</sup> )	275.2	232.2	30	20

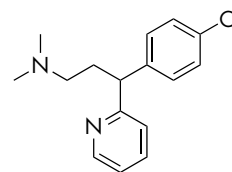
### Oasis MCX Extraction Method

Oasis<sup>®</sup> MCX Extraction Plate, 10 mg/96 wells  
Part Number 186000259

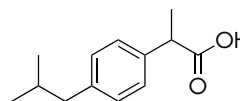
CONDITION: 500 μL CH <sub>3</sub> OH
EQUILIBRATE: 500 μL H <sub>2</sub> O
LOAD: 500 μL sample (250 μL plasma diluted 1/1 with 4% H <sub>3</sub> PO <sub>4</sub> in H <sub>2</sub> O)
WASH: 500 μL 2% HCOOH
ELUTE 1: 2 x 125 μL CH <sub>3</sub> OH (Ibuprofen)
ELUTE 2: 2 x 125 μL 5% NH <sub>4</sub> OH in CH <sub>3</sub> OH (Bases)
OPTIONS: 1. Dilute Elute 2 with 250 μL 2% HCOOH in water and Elute 1 with 250 μL 100% water and analyze separately. 2. Combine the two elutions and evaporate/reconstitute in 250 μL CH <sub>3</sub> OH/H <sub>2</sub> O (50/50)  Pseudoephedrine HCl (Base): 1.5 μg/mL Chlorpheniramine Maleate (Base): 0.1 μg/mL Ibuprofen (Acid): 10 μg/mL



Pseudoephedrine

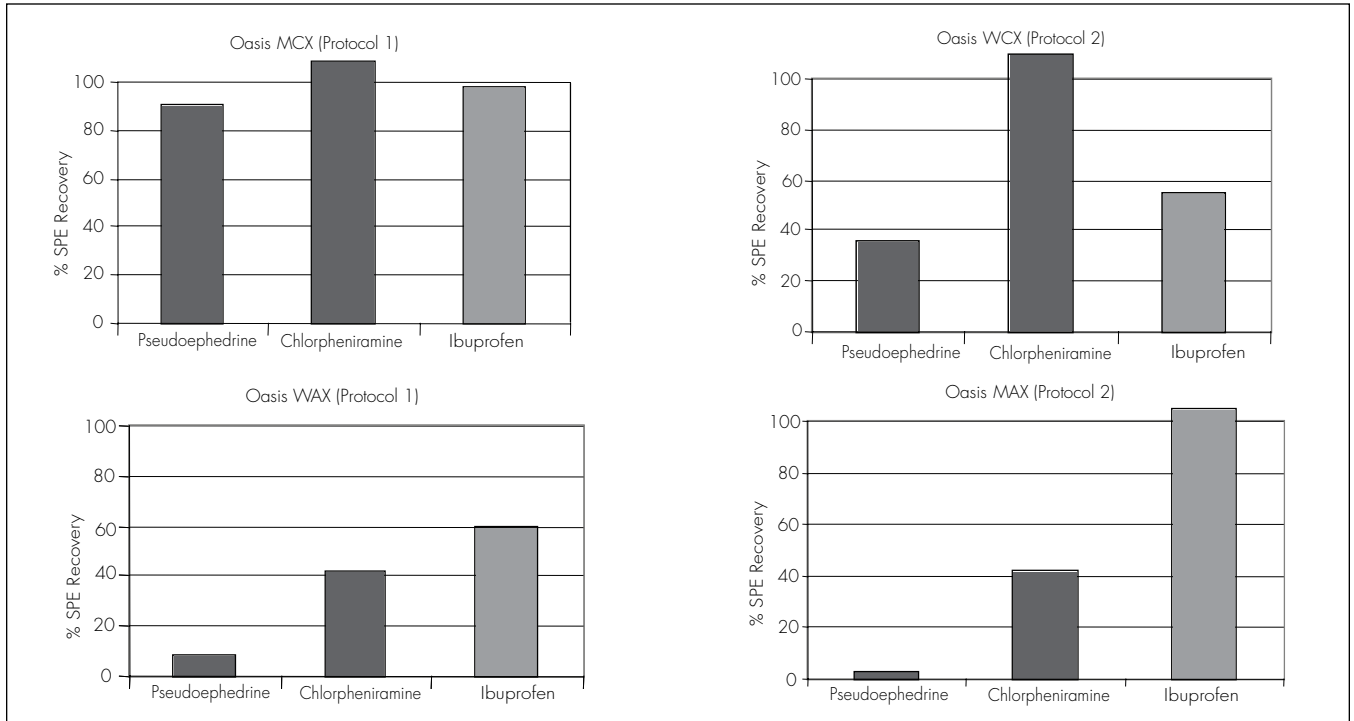


Chlorpheniramine



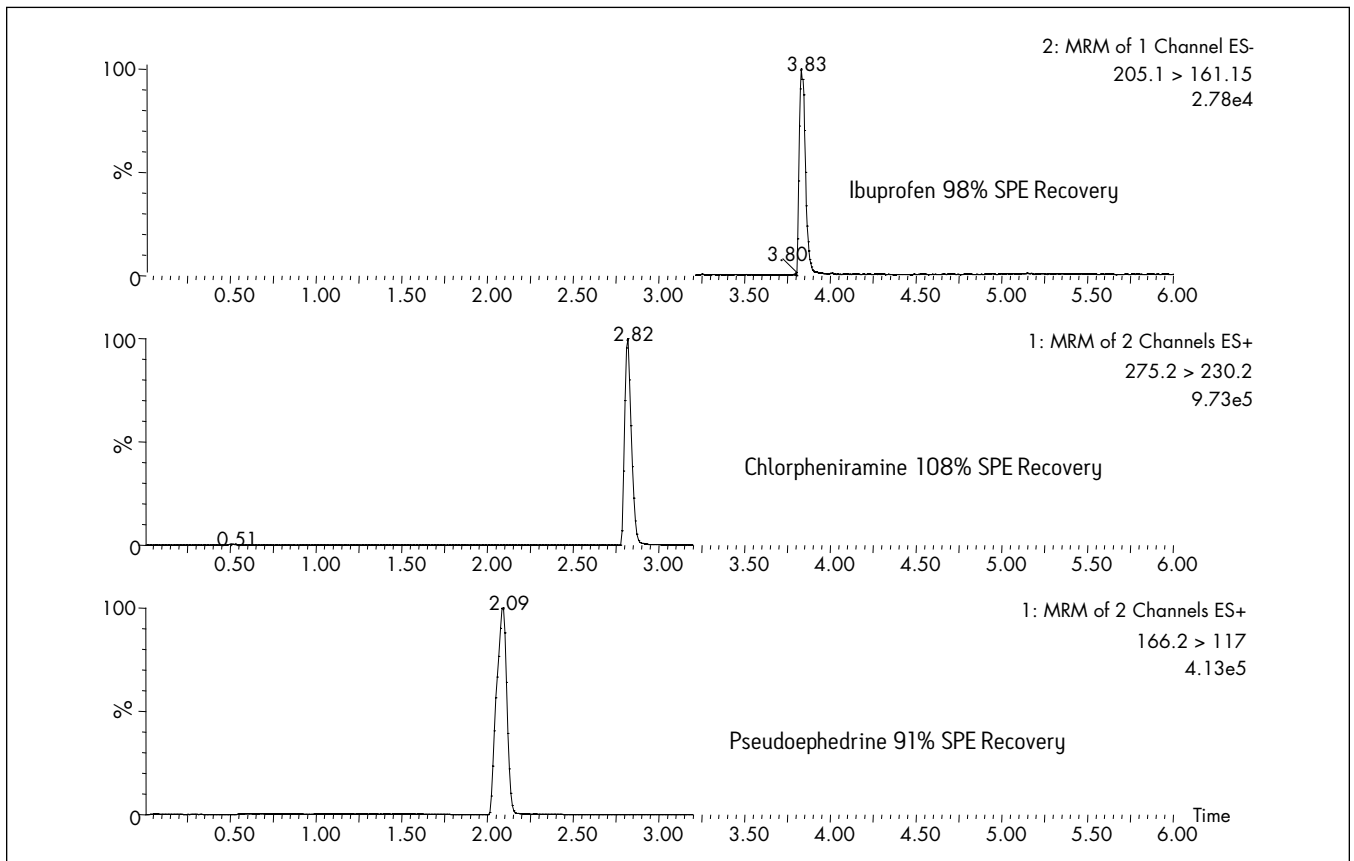
Ibuprofen

Recovery SPE Data Using Oasis 2x4 Method for the Selection of the Optimum Sorbent and Protocol\*



\*For more information on the Oasis 2x4 Method, go to [www.waters.com/oasis](http://www.waters.com/oasis)

UPLC/MS Chromatogram Oasis MCX Recovery (Protocol 1)



# ALKALOIDS IN GOLDENSEAL, COMMERCIAL PRODUCTS OR WHOLE LEAF BY LC/UV

## LC Conditions

Column: Symmetry<sup>®</sup> C<sub>18</sub>, 4.6 x 250 mm, 5 µm

Part Number: WAT054275

Mobile Phase A: 0.1% H<sub>3</sub>PO<sub>4</sub>

Mobile Phase B: CH<sub>3</sub>CN

Gradient:

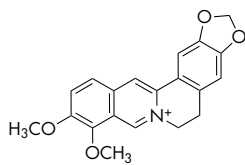
Time (min)	Profile	
	A (%)	B (%)
Initial	90	10
13	78	22
40	60	40

Flow Rate: 1.5 mL/min

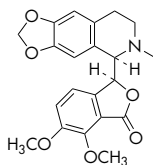
Injection Volume: 10 µL

Temperature: 35 °C

Detection: UV @ 330 nm

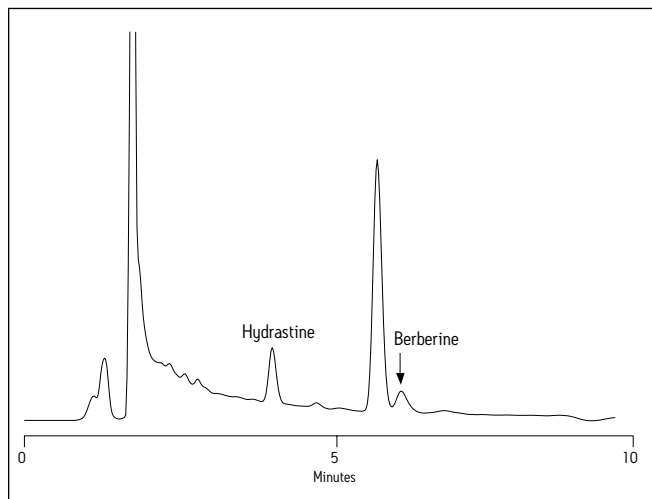


Berberine



Hydrastine

## HPLC Chromatogram of Goldenseal Alkaloids

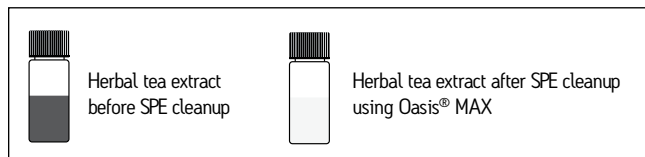


## Oasis HLB Extraction Method for Alkaloids in Goldenseal

Oasis<sup>®</sup> HLB Extraction Cartridge, 3 cc/60 mg

Part Number WAT094226

CONDITION/EQUILIBRATE: 1 mL CH <sub>3</sub> OH/1 mL 150 mM K <sub>2</sub> HPO <sub>4</sub>
LOAD: 3 mL of diluted sample
WASH: 1 mL 30% CH <sub>3</sub> OH/150 mM K <sub>2</sub> HPO <sub>4</sub>
ELUTE: 3 mL CH <sub>3</sub> OH



## Sample Pre-Preparation

100 mg sample is extracted with 25 mL of ethanol/ water (70/30)

1 mL of the ethanolic extract is diluted 1/3 with water (~ pH 7)

- Recovery, measured with certified standards at 100 ppm in reagent water, was greater than 85% for all compounds.
- All analyses gave results within ± 35% of the expected values with the exception of the liquid supplement (see goldenseal analysis).
- The selective SPE extraction and cleanup procedure provided a convenient analysis of echinacea phenolics in complex matrix such as in herbal tea.

### LC Conditions

Column: Waters Spherisorb® Silica,  
2 x 100 mm, 3 µm

Part Number: PSS832022

Mobile Phase A: CH<sub>3</sub>CN

Mobile Phase B: 0.05% H<sub>3</sub>PO<sub>4</sub>

Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	100	0
5	0	100

Flow Rate: 200 µL/min

Instrument: Waters Alliance® 2690 Separations Module

### MS Conditions

Instrument: Waters ZQ™

Ion Source: Positive Electrospray (ESI<sup>+</sup>)

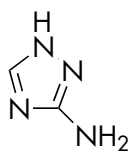
Mode: Selected-ion recording

Source Temperature: 150 °C

Desolvation Temperature: 450 °C

Desolvation Gas Flow: 500 L/hr

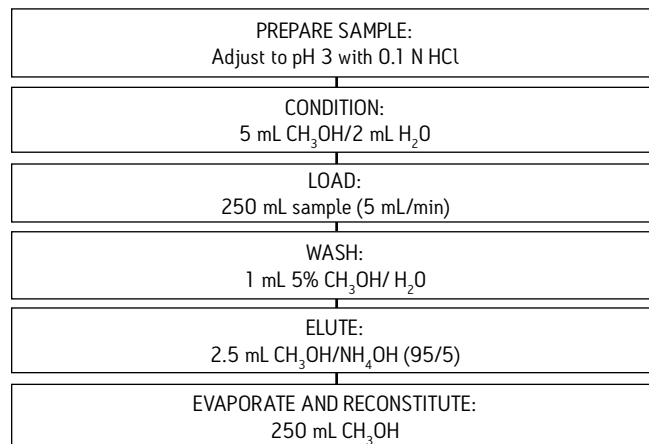
Cone: 50 V



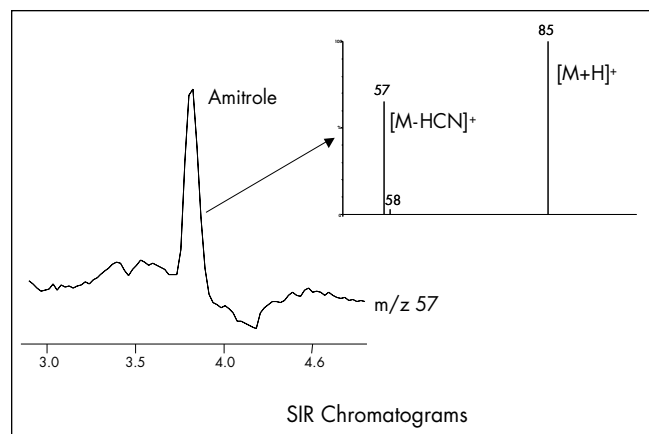
Amitrole

### Oasis MCX Extraction Method

Oasis® MCX Extraction Cartridge, 6 cc/150 mg (LP)  
Part Number 186000255



### LC/MS Chromatogram of Amitrole in River Water, 50 ng/L Spiked Level



### Amitrole Results (n=6)

Amitrole Spike Level (ng/L)	Recovery (%)	% RSD (n=6)*
50	95	9.4
100	85	7.6
400	84	5.0

\* Calculated against standard prepared in matrix. Matrix suppression was approximately 10%.

## LC Conditions

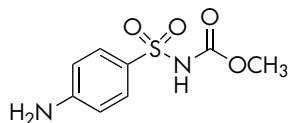
Column: Atlantis® dC<sub>18</sub>, 2.1 x 100 mm, 3 µm  
 Part Number: 1860001295  
 Mobile Phase A: 15 mM HCOONH<sub>4</sub>, pH 4.5  
 Mobile Phase B: CH<sub>3</sub>CN  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	90	10
19	10	90

Flow Rate: 200 µL/min  
 Injection Volume: 20 µL  
 Instrument: Waters Alliance® 2695 Separations Module

## MS Conditions

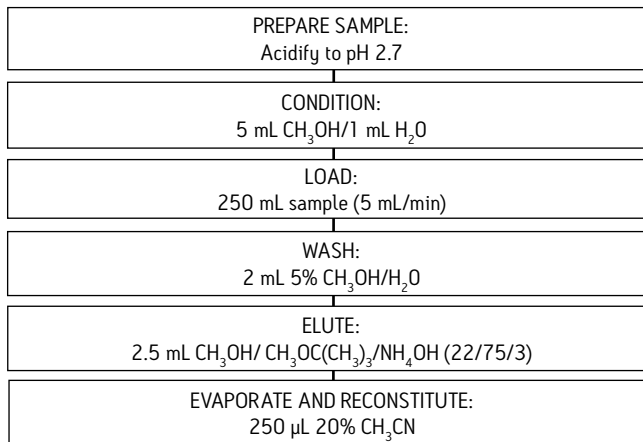
Instrument: Waters Quattro Ultima®  
 Ion Source: Electrospray Positive and Negative (ESI<sup>+</sup> and ESI<sup>-</sup>)  
 Mode: Multiple Reaction Monitoring  
 Source Temperature: 150 °C  
 Desolvation Temperature: 450 °C  
 Cone Gas Flow: 50 L/hr  
 Desolvation Gas Flow: 500 L/hr  
 Collision Gas: Argon



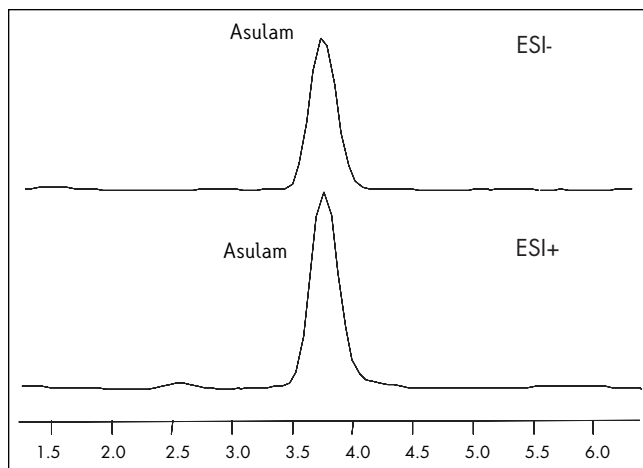
Asulam

## Oasis MCX Extraction Method

Oasis® MCX Extraction Cartridge, 6 cc/150 mg (LP)  
 Part Number 186000255



## LC/MS Chromatogram of Asulam in River Water, 250 ng/L Spiked Level



Asulam Spike Level (ng/L)	% Recovery (% RSD), (n=4)
50	81 (14)
250	78 (7.6)
1000	71 (12)

MRM	Cone (V)	Coll Energy (eV)
231-->156	30	10
229 -->197	25	15

### LC Method

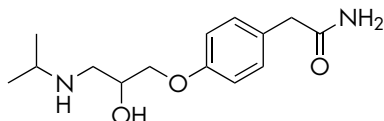
Column: XTerra® MS C<sub>18</sub>, 2.1 x 20 mm /S<sup>TM</sup>, 3.5 μm  
 Part Number: 186001923  
 Mobile Phase A: 10 mM NH<sub>4</sub>HCO<sub>3</sub>, pH 10  
 Mobile Phase b: CH<sub>3</sub>OH with 10 mM NH<sub>4</sub>HCO<sub>3</sub>, pH 10  
 Flow Rate: 0.4 mL/min  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	95	5
3.0	5	95
4.0	5	95
4.1	95	5
5.0	95	5

Injection Volume: 10 μL  
 Column Temperature: Ambient  
 Instruments: Waters 2777 Sample Manager and  
 Waters 1525μ Binary HPLC Pump

### MS Conditions

Instrument: Waters Quattro Ultima®  
 Ion Source: Electrospray Positive (ESI+)  
 Source Temperature: 150 °C  
 Desolvation Temperature: 350 °C  
 Cone Gas Flow: 50 L/hr  
 Desolvation Gas Flow: 550 L/hr  
 Collision Cell Pressure: 2.2e3 Bar (Argon Gas)  
 Cone Voltage: 45 V  
 CID: 25 eV  
 MRM Transition: m/z 266.9 → 144.9



Atenolol

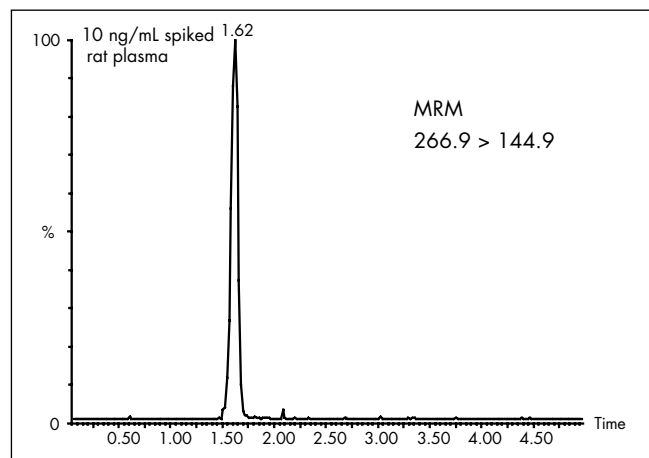
### Oasis WCX Extraction Method

Oasis® WCX 96-well μElution Plate  
 Part Number 186002499

CONDITION: 200 μL CH <sub>3</sub> OH
EQUILIBRATE: 200 μL H <sub>2</sub> O
LOAD: 150 μL of rat plasma diluted with 150 μL of 4% H <sub>3</sub> PO <sub>4</sub> in H <sub>2</sub> O
WASH 1: 200 μL 25mM KH <sub>2</sub> PO <sub>4</sub> /K <sub>2</sub> HPO <sub>4</sub> in H <sub>2</sub> O, pH 7
WASH 2: 200 μL CH <sub>3</sub> OH
ELUTE: 50 μL (25 μL x 2) 2% HCOOH in CH <sub>3</sub> OH
DILUTE: 100 μL 2% NH <sub>4</sub> OH in H <sub>2</sub> O
INJECT: 10 μL

**101% SPE Recovery**

### LC/MS Chromatogram of Atenolol in Rat Plasma



# ATENOLOL IN URINE BY MIXED-MODE WEAK CATION EXCHANGE BY LC/MS/MS

## LC Conditions

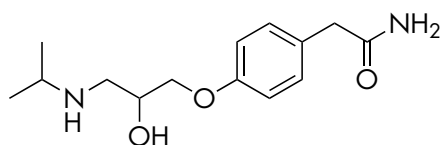
Column: XTerra® MS C<sub>18</sub>, 2.1 x 20 mm *IS*<sup>TM</sup>, 3.5 µm  
 Part Number: 186001923  
 Mobile Phase A: 10 mM NH<sub>4</sub>HCO<sub>3</sub>, pH 10  
 Mobile Phase B: CH<sub>3</sub>OH with 10 mM NH<sub>4</sub>HCO<sub>3</sub>, pH 10  
 Flow Rate: 0.4 mL/min  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	95	5
3.0	5	95
4.0	5	95
4.1	95	5
5.0	95	5

Injection Volume: 10 µL  
 Column Temperature: Ambient  
 Instruments: Waters 2777 Sample Manager and  
 Waters 1525µ Binary HPLC Pump

## MS Conditions

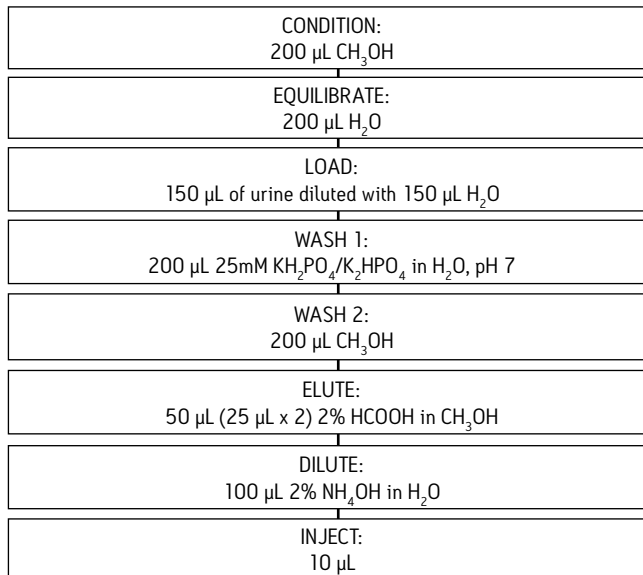
Instrument: Waters Quattro Ultima®  
 Ion Source: Electrospray Positive (ESI)  
 Source Temperature: 150 °C  
 Desolvation  
 Temperature: 350 °C  
 Cone Gas Flow: 50 L/hr  
 Desolvation Gas Flow: 550 L/hr  
 Collision Cell Pressure: 2.2e3 Bar (Argon Gas)  
 Cone Voltage: 45 V  
 CID: 25 eV  
 MRM Transition: m/z 266.9 → 144.9



Atenolol

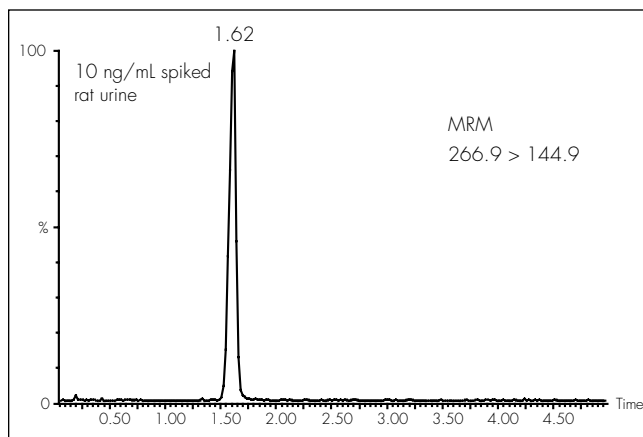
## Oasis WCX Extraction Method

Oasis® WCX 96-well µElution Plate  
 Part Number 186002499



**106% SPE Recovery**

## LC/MS Chromatogram of Atenolol in Urine



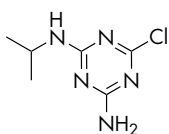


### LC Conditions

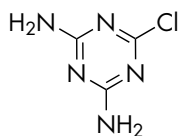
Column: SymmetryShield™ RP18, 3.9 x 150 mm, 5 µm  
 Part Number: 186000108  
 Mobile Phase A: 20 mM KH<sub>2</sub>PO<sub>4</sub>/ K<sub>2</sub>HPO<sub>4</sub>, pH 6.8  
 Mobile Phase B: CH<sub>3</sub>CN  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	95	5
2	95	5
20	25	75

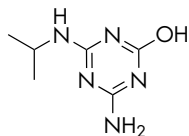
Flow Rate: 0.8 mL/min  
 Injection Volume: 80 µL  
 Detection: PDA (215 nm)



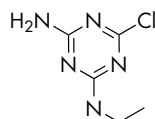
Desethylatrazine



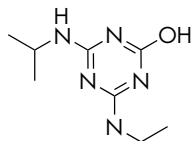
Desethyl-desisopropylatrazine



Hydroxydesethylatrazine



Desisopropylatrazine

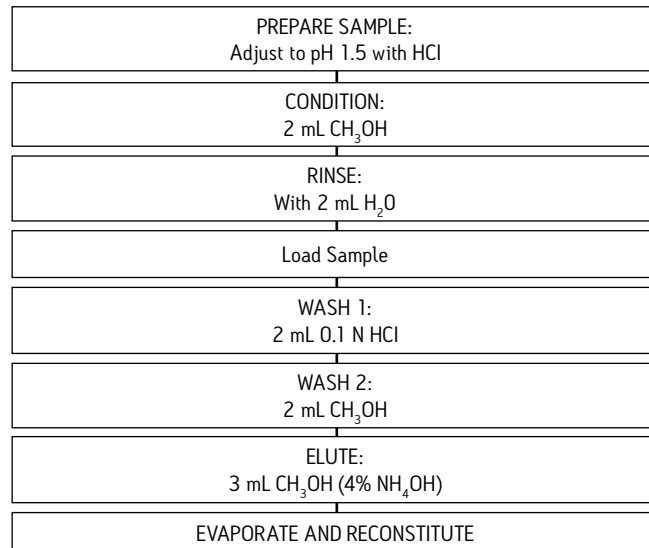


Hydroxyatrazine

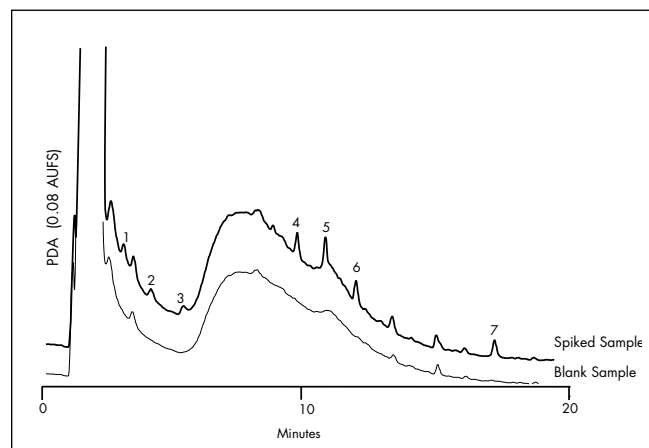
Compounds	% Recovery (% RSD), n=5	
	0.2 µg/L	1 µg/L
1. Hydroxydesisopropylatrazine	94 (3)	85 (3)
2. Desethyl-desisopropylatrazine	75 (8)	76 (5)
3. Hydroxydesethylatrazine	89 (6)	76 (7)
4. Desisopropylatrazine	79 (4)	83 (2)
5. Hydroxyatrazine	107 (7)	101 (2)
6. Desethylatrazine	79 (5)	83 (3)
7. Atrazine	89 (5)	77 (3)

### Oasis MCX Extraction Method

Oasis® MCX Extraction Cartridge, 6 cc/150 mg  
 Part Number 186000256



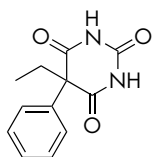
### HPLC Chromatogram of Atrazine and Metabolites in Drinking Water



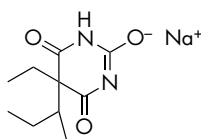
Drinking water samples (100 mL) were spiked with the herbicides and adjusted to pH 1.5. The samples were then analyzed using Oasis MCX 6 cc cartridges using the protocol for basic compounds.

**LC Conditions**

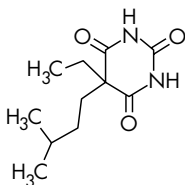
Column: SymmetryShield™ RP18, 2.1 x 150 mm, 5 µm  
 Guard Column: SymmetryShield RP18, 3.9 x 20 mm, 5 µm  
 Part Numbers: Column - 186000111, Guard - 186000107  
 Mobile Phase: (50 mM K<sub>2</sub>PO<sub>4</sub>/KH<sub>2</sub>PO<sub>4</sub>, pH 7)/CH<sub>3</sub>CN (71/29)  
 Flow Rate: 1 mL/min  
 Injection Volume: 80 µL urine extract  
 Temperature: 30 °C  
 Detection: UV @ 214 nm (0.350 AUFS)



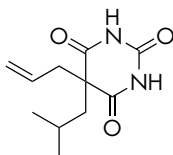
Phenobarbital



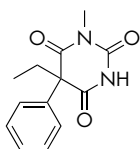
Butabarbital



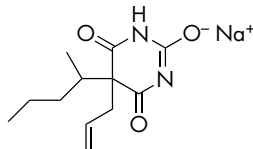
Amobarbital (I.S.)



Butalbital



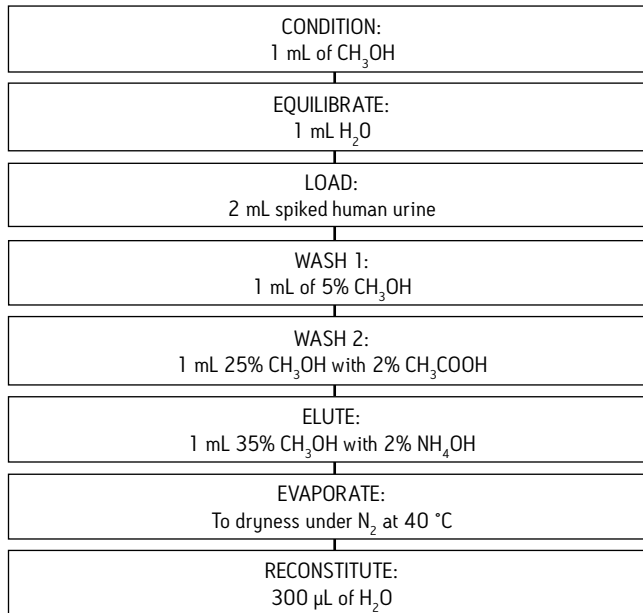
Mephobarbital



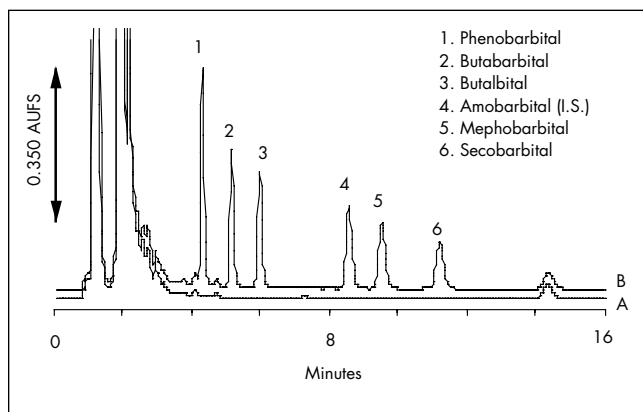
Secobarbital

**Oasis HLB Extraction Method**

Oasis® HLB Extraction Plate, 30 mg/96 wells  
 Part Number WAT058951



**HPLC Chromatogram of Barbiturates in Human Urine, A) Blank Urine, B) Spiked Urine**



Spiked Levels	% Recovery (% RSD), n=5	
	0.2 µg/L	1.0 µg/L
Phenobarbital	114.3 (1.7)	106.5 (0.5)
Butabarbital	95.7 (1.3)	105.5 (0.7)
Butalbital	109.5 (0.9)	104.2 (0.9)
Amobarbital (I.S.)		86.3 (1.7)
Mephobarbital	92.5 (3.6)	92.4 (1.7)
Secobarbital	111.5 (5.2)	94.8 (2.2)

### LC Conditions

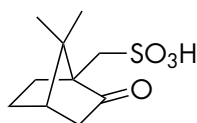
Column: SunFire™ C<sub>18</sub>, 2.1 x 20 mm /S™, 3.5 μm  
 Mobile Phase A: 10 mM CH<sub>3</sub>COONH<sub>4</sub>, pH 5.5  
 Mobile Phase B: CH<sub>3</sub>OH with 10 mM CH<sub>3</sub>COONH<sub>4</sub>, pH 5.5  
 Flow Rate: 0.4 mL/min  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	95	5
3.0	5	95
4.0	5	95
4.1	95	5
5.0	95	5

Injection Volume: 10 μL  
 Column Temperature: Ambient  
 Instrument: 2777 Sample Manager, 1525μ Binary HPLC Pump

### MS Conditions

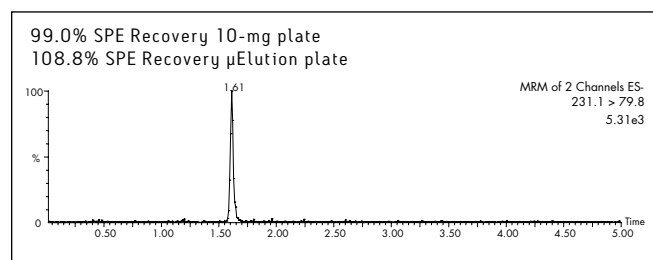
Instrument: Waters Quattro Premier™  
 Ion Source: Electrospray Negative (ESI)  
 Source Temperature: 150 °C  
 Desolvation Temperature: 350 °C  
 Cone Gas Flow: 50 L/hr  
 Desolvation Gas Flow: 600 L/hr  
 Collision Cell: 2.2e-3 bar (Argon gas)



10-Camphorsulfonic Acid

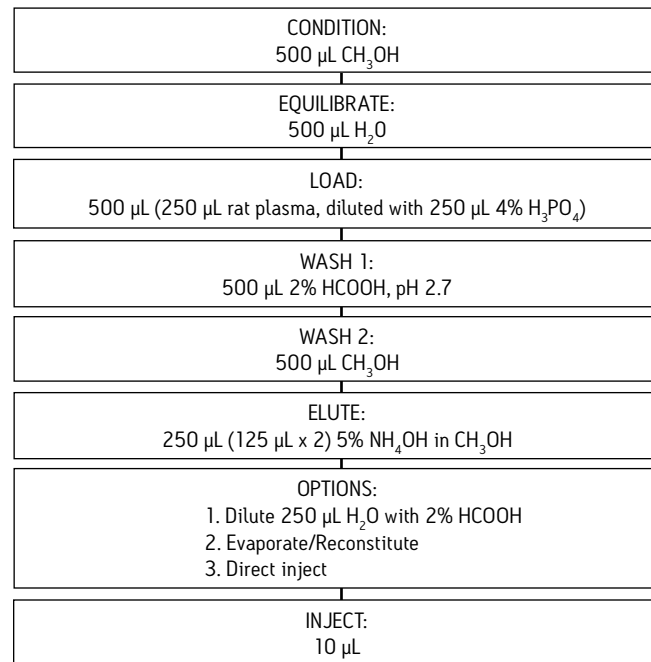
	MRM Transition (m/z)	Cone (V)	CID (eV)
Camphorsulfonic Acid	m/z 231.1 → 79.8	60	30

### LC/MS Chromatogram of Camphorsulfonic Acid in Rat Plasma, 10 ng/mL

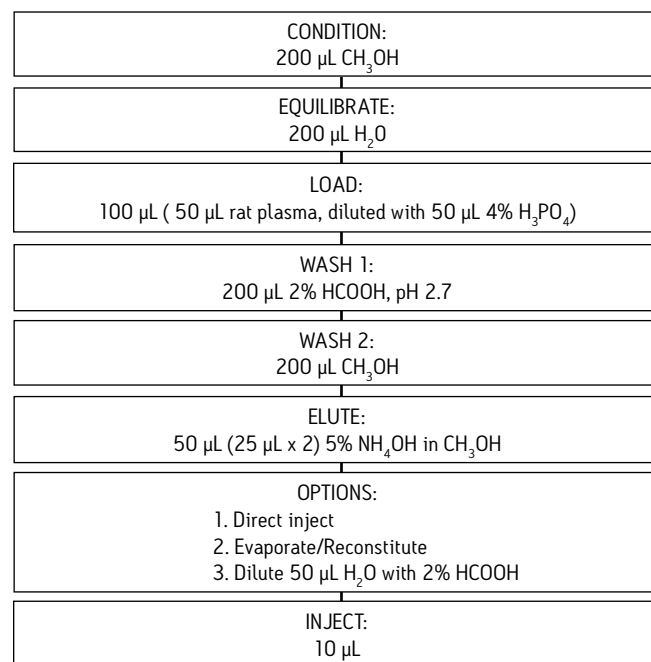


### Oasis MAX Extraction Method

Oasis® WAX Extraction Plate, 10 mg/96 wells  
 Part Number 186002502



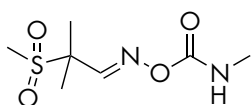
Conditions for Oasis WAX 96-well μElution Plate  
 Part Number 186002500



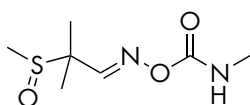
# CARBAMATES IN DRINKING WATER (ENDOCRINE DISRUPTORS) BY POST COLUMN DERIVATIZATION FLUORESCENCE

## LC Conditions

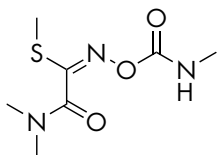
Column: Waters Carbamate Analysis, 3.9 x 150 mm  
 Mobile Phase A: H<sub>2</sub>O  
 Mobile Phase B: CH<sub>3</sub>OH  
 Mobile Phase C: CH<sub>3</sub>CN  
 Flow Rate: 1.5 mL/min  
 Injection Volume: 75 µL  
 Sample: 200 mL of drinking water spiked @ 50 ng/L  
 Detection: Post Column Derivatization, fluorescence



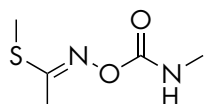
Aldicarb Sulfoxide



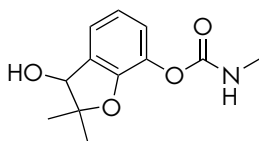
Aldicarb Sulfone



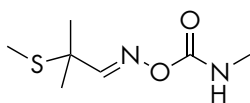
Oxamyl



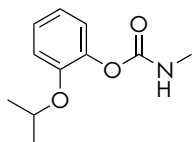
Methomyl



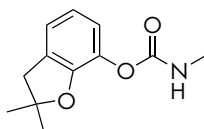
3-Hydroxycarbofuran



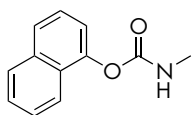
Aldicarb



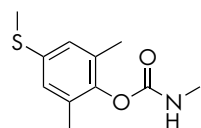
Propoxur



Carbofuran



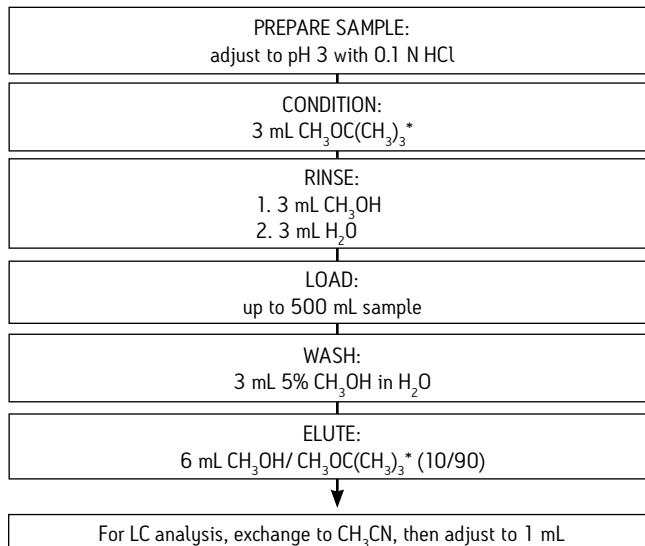
Carbaryl



Methiocarb

## Oasis HLB Extraction Method

Oasis® HLB Extraction Cartridge, 6 cc/200 mg  
 Part Number WAT106202

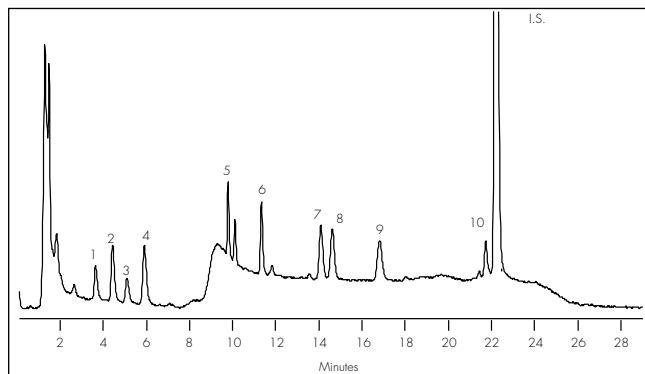


\* (C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>O can be used as an alternative to CH<sub>3</sub>OC(CH<sub>3</sub>)<sub>3</sub>

Compounds	LC/PCDF* % Recovery (%RSD)	
	500 ng/L	50 ng/L
1. Aldicarb Sulfoxide	54.7 (0.5)	45.7 (5.1)
2. Aldicarb Sulfone	98.7 (4.0)	101 (3.6)
3. Oxamyl	90.8 (7.0)	122 (18)
4. Methomyl	99.9 (6.4)	100 (3.2)
5. 3-Hydroxycarbofuran	98.7 (2.3)	111 (6.5)
6. Aldicarb	90.7 (9.3)	104 (5.8)
7. Propoxur	97.5 (5.6)	99.9 (5.8)
8. Carbofuran	97.2 (4.7)	104 (7.9)
9. Carbaryl	89.6 (2.2)	122 (11)
10. Methiocarb	91.6 (2.2)	120 (14)

\* The 500 ng/L sample SPE extracts were split and analyzed by each method LC/PCDF - LC with post column derivitization and fluorescence detection

## Chromatogram of Carbamates in Drinking Water, 50 ng/L Spiked Level

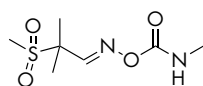


### LC Conditions

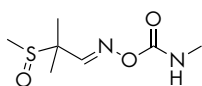
LC Column: Symmetry® C<sub>18</sub>, 1 x 150 mm, 3.5 µm  
 Part Number: WAT248059  
 Mobile Phase A: CH<sub>3</sub>OH/ 10 mM CH<sub>3</sub>COONH<sub>4</sub> (10/90)  
 Mobile Phase B: CH<sub>3</sub>OH/ 10 mM CH<sub>3</sub>COONH<sub>4</sub> (90/10)  
 Gradient: 90% A initial, linear gradient to 90% B  
 in 10 minutes  
 Injection Volume: 10 µL  
 Flow Rate: 75 µL/min  
 Temperature: 35 °C  
 Instrument: Waters Alliance® 2695

### MS Conditions

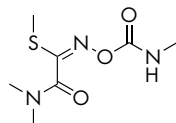
Instrument: Waters ZQ™  
 Ion Source: Positive Electrospray (ESI+)  
 Mode: Multiple Selected-Ion Recording (SIR)



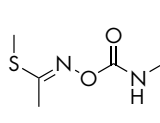
Aldicarb Sulfoxide



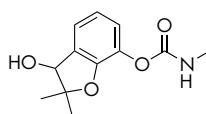
Aldicarb Sulfone



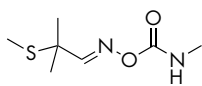
Oxamyl



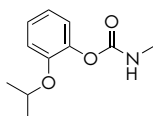
Methomyl



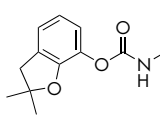
3-Hydroxycarbofuran



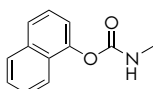
Aldicarb



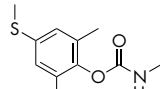
Propoxur



Carbofuran



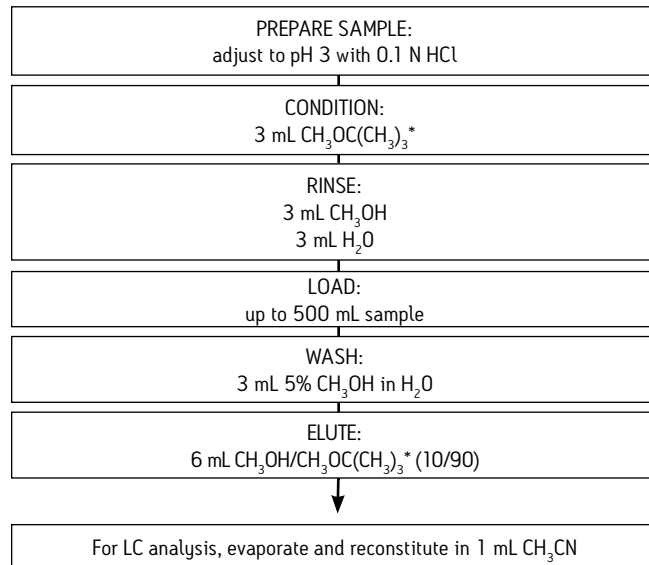
Carbaryl



Methiocarb

### Oasis HLB Extraction Method

Oasis® HLB Extraction Cartridge, 6 cc/200 mg  
 Part Number WAT106202



\* (C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>O can be used as an alternative to CH<sub>3</sub>OC(CH<sub>3</sub>)<sub>3</sub>

SIR Group	Time (min)	Compound	Mass	Cone Voltage (V)	Dwell Time (sec)
1	0-9	Aldicarb Sulfoxide	207.1	18	0.5
		Aldicarb Sulfone	223.2	25	0.5
		Oxamyl	237.2	10	0.5
		Methomyl	163.2	15	0.5
2	9-11	3-OH-Carbofuran	238.2	15	1.5
		Aldicarb	208.2	8	1.5
4	11.5-14	Propoxur	210.2	18	0.4
		Carbofuran	222.2	22	0.4
		Carbaryl	202.2	18	0.4
5	14-20	Methiocarb	226.2	19	0.6

Compounds	Recovery (%) LC/MS* 500 ng/L
1. Aldicarb Sulfoxide	74.8 (19)
2. Aldicarb Sulfone	88.7 (16)
3. Oxamyl	83.2 (18)
4. Methomyl	92.3 (8.0)
5. 3-Hydroxycarbofuran	101 (8.6)
6. Aldicarb	79.4 (9.3)
7. Propoxur	103 (13)
8. Carbofuran	95.6 (7.5)
9. Carbaryl	97.7 (14)
10. Methiocarb	81.2 (14)

## LC Conditions

Column: ACQUITY UPLC® BEH C<sub>18</sub>, 2.1 x 50 mm, 1.7 μm

Mobile Phase A: 0.1% HCOOH in H<sub>2</sub>O

Mobile Phase B: 0.1% HCOOH in CH<sub>3</sub>OH

Flow Rate: 0.4 mL/min

Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	98	2
0.5	98	2
2.5	0	100
3.0	0	100
3.1	98	2
4.0	98	2

Injection Volume: 10 μL

Column Temperature: 45 °C

Sample Temperature: 15 °C

Sample Diluent: 50/50 H<sub>2</sub>O/CH<sub>3</sub>OH

Instrument: Waters ACQUITY UPLC System

## MS Conditions

Instrument: Waters Quattro Premier™

Ion Source: Electrospray Positive (ESI<sup>+</sup>)

Capillary: 3.0 kV

Source Temperature: 120 °C

Desolvation Temperature: 350 °C

Cone Gas Flow: 50 L/hr

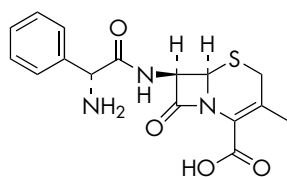
Desolvation Gas Flow: 700 L/hr

Collision Cell Pressure: 2.59 e-3 mbar

MRM Transition: 348 → 157.9

Cone Voltage: 20 V

Collision Energy: 18 eV



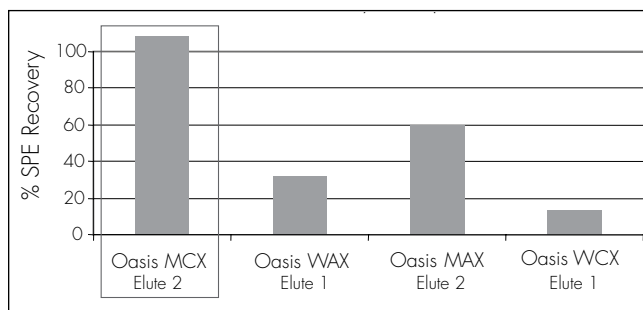
Cephalexin

## Oasis MCX Extraction Method

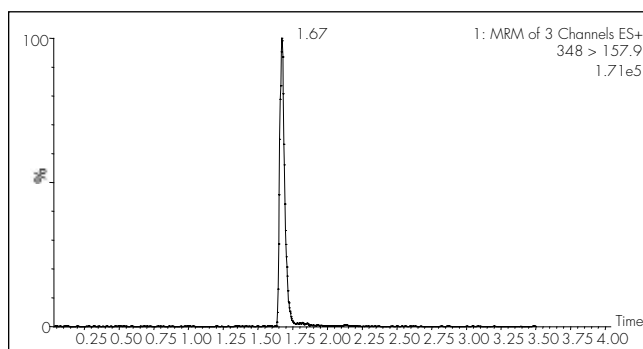
Oasis® MCX Extraction, 10 mg/96 wells  
Part Number 186000259

CONDITION: 500 μL CH <sub>3</sub> OH
EQUILIBRATE: 500 μL H <sub>2</sub> O
LOAD: 500 μL (250 μL rat plasma, diluted with 250 μL 4% H <sub>3</sub> PO <sub>4</sub> in H <sub>2</sub> O)
WASH 1: 500 μL 2% HCOOH
WASH 2: 500 μL CH <sub>3</sub> OH
ELUTE: 250 μL (125 μL x 2) 5% NH <sub>4</sub> OH in CH <sub>3</sub> OH
OPTIONS: 1. Dilute with 250 μL H <sub>2</sub> O and 2% HCOOH 2. Evaporate/Reconstitute 3. Direct inject
INJECT: 10 μL

## % SPE Recovery of Cephalexin



## LC/MS Chromatogram of Cephalexin in Rat Plasma, 200 ng/mL SPE Recovery: 105%

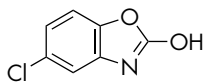


**LC Conditions**

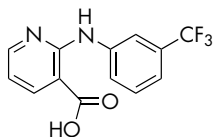
Column: XTerra® MS C<sub>18</sub>, 2.1 x 30 mm, 3.5 µm  
 Part Number: 186000398  
 Mobile Phase A: 0.2% HCOOH  
 Mobile Phase B: CH<sub>3</sub>CN  
 Isocratic Mobile  
 Phase Composition: 45% A; 55% B  
 Flow Rate: 0.2 mL/min  
 Injection Volume: 20 µL  
 Instrument: Waters Alliance® 2790

**MS Conditions**

Instrument: Waters Quattro Ultima®  
 Ion Source: Electrospray Negative (ESI)  
 Source Temperature: 150 °C  
 Gas Cell: 1.5e-3 mbar, 20 eV  
 Desolvation Temperature: 350 °C  
 Cone Gas Flow: 150 L/hr  
 Drying Gas Flow: 600 L/hr  
 Cone Voltage: 30 V



Chlorzoxazone

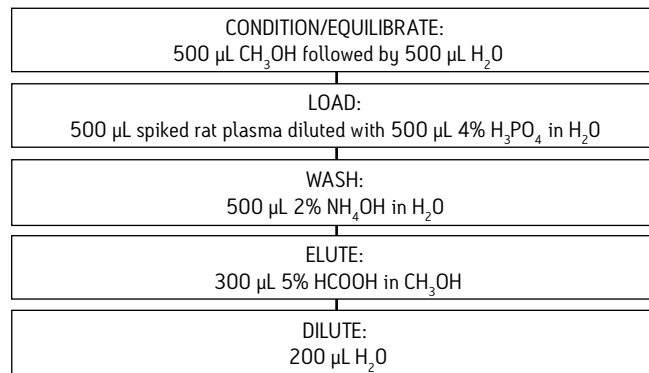


Niflumic Acid (I.S.)

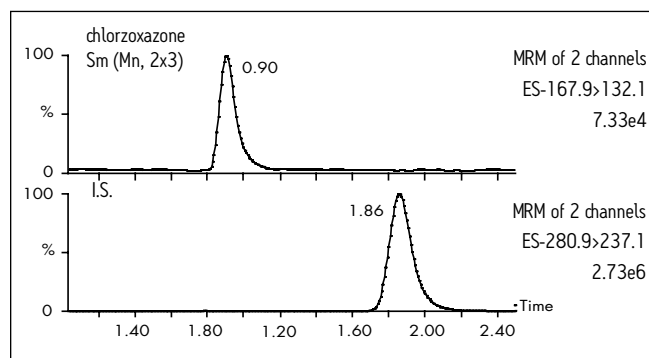
Cholorzoxazone (ng/ mL)	Mean	Standard Deviation	Coefficient of Variation (%)	Recovery (%)
5	4.93	0.076	1.5	99
10	10.4	0.38	3.7	104
25	24.96	1.43	5.7	99
100	99.85 (5)	3.53	3.5	99
250	245.91	8.31	3.3	98

**Oasis MAX Extraction Method**

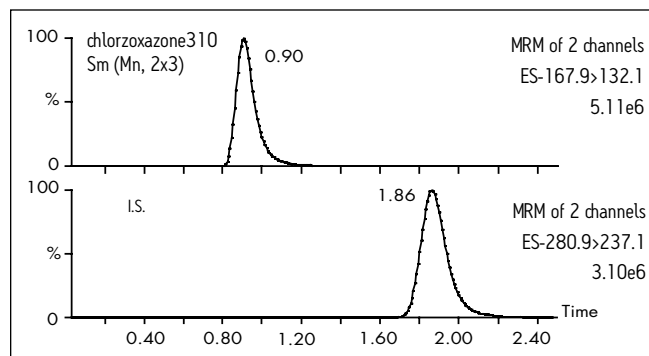
Oasis® MAX Extraction Plate, 10 mg/96 wells  
 Part Number 186000375



**LC/MS Chromatogram of Chlorzoxazone in Rat Plasma, 5 ng/mL Level**



**LC/MS Chromatogram of Chlorzoxazone in Rat Plasma, 250 ng/mL Level**



# CLEMASTINE IN RAT PLASMA - OASIS ON-LINE SPE: ONE EXTRACTION COLUMN

## LC Conditions

LC Instrument 1:	Waters Alliance® 2795 - 0.4 mL/min
LC Instrument 2:	Waters 515 - 4.0 mL/min
Loading Mobile Phase:	100% H <sub>2</sub> O
Eluting Mobile Phase:	1 min gradient 5% CH <sub>3</sub> CN to 95% CH <sub>3</sub> CN
Eluting Mobile Phase Additive:	0.5% HCOOH
Extraction Column	
Temperature:	40 °C
Switching Valve:	Waters Selector Valve

## MS Conditions

Instrument:	Waters Quattro Ultima®
Ion Source:	Electrospray Positive (ESI <sup>+</sup> )
Source Temperature:	150 °C
Desolvation Gas Flow:	600 L/hr
Collision Cell Pressure:	1.5e-3 mbar
Cone Voltage:	20 V
Collision Energy:	20 eV

Time (min)	HPLC Gradient Flow 0.4 mL/minute		Valve Position
	% A	% B	
Initial	5	95	
0.5			switch position 2 to 1
1.0	95	5	
2.60	95	5	
2.90			switch position 1 to 2
3.0	5	95	

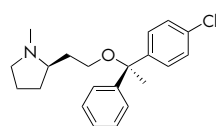
A- CH<sub>3</sub>CN + 0.5% HCOOH  
B- H<sub>2</sub>O + 0.5% HCOOH

Conc. ng/ mL	Average n=6	Standard Deviation	RSD (%)
1.0	0.98	0.02	2.1
2.5	2.56	0.11	4.4
5.0	5.25	0.12	2.3
10.0	9.50	0.25	2.7
100.0	101.43	2.69	2.6
200.0	201.14	3.20	1.6
250.0	247.10	1.58	0.6

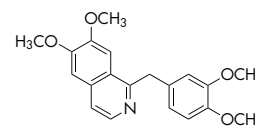
## On-Line SPE Method

Oasis® HLB Extraction Column, 2.1 x 20 mm, 25 µm  
Part Number 186000706

<b>SAMPLE PREPARATION:</b> Dilute 100 µL of rat plasma with 100 µL of I.S. solution containing 4% NH <sub>4</sub> OH in H <sub>2</sub> O
<b>LOAD:</b> 200 µL at 4 mL/min in 100% H <sub>2</sub> O
<b>ELUTE:</b> 0.4 mL/min gradient 5% CH <sub>3</sub> CN to 95% CH <sub>3</sub> CN in 1 min

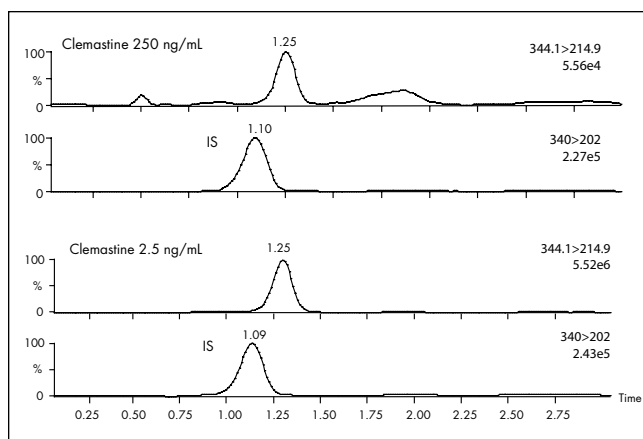


Clemastine

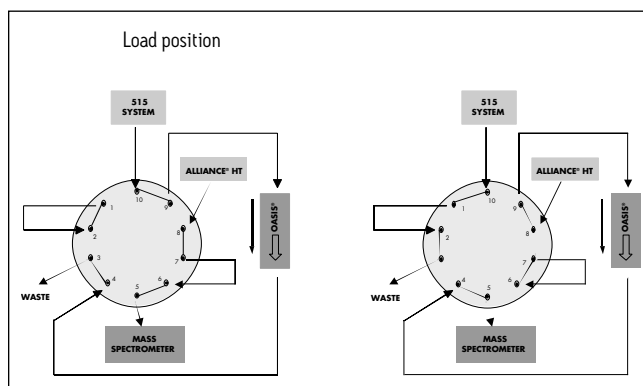


Papaverine I.S.

## Oasis HLB LC/MS/MS Analysis of Clemastine at 2.5 ng/mL and 250 ng/mL



## Selector Valve Position Diagram



Reference: Mallet, C.R.; Mazzeo, J.; Neue U. *Rapid Commun. Mass Spectrom.* **15**:1075-1083. (2001)



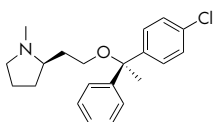
# CLEMASTINE IN RAT PLASMA - OASIS ON-LINE SPE: ONE EXTRACTION COLUMN AND ONE ANALYTICAL COLUMN

## LC Conditions

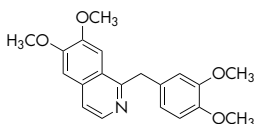
Column: XTerra® MS C<sub>18</sub>, 2.1 x 30 mm, 3.5 µm  
 LC Instrument 1: Waters Alliance® 2795 - 0.4 mL/min  
 LC Instrument 2: Waters 515 - 4 mL/min  
 Loading Mobile Phase: 100% H<sub>2</sub>O  
 Eluting Mobile Phase: 1 min gradient 5% CH<sub>3</sub>CN to 95% CH<sub>3</sub>CN  
 Eluting Mobile  
 Phase Additive: 0.5% HCOOH  
 Extraction Column  
 Temperature: 40 °C  
 Switching Valve: Waters Selector Valve

## MS Conditions

Instrument : Waters Quattro Ultima®  
 Ion Source: Electrospray Positive (ESI\*)  
 Source Temperature: 150 °C  
 Desolvation Gas Flow: 600 L/hr  
 Collision Cell Pressure: 1.5e-3 mbar  
 Cone Voltage: 20 V  
 Collision Energy: 20 eV



Clemastine



Papaverine (I.S.)

Time (min)	HPLC Gradient Flow 0.4 mL/min		Valve Position
	% A	% B	
Initial	5	95	
0.5			switch position 2 to 1
1.0	95	5	
2.60	95	5	
2.90			switch position 1 to 2
3.0	5	95	

A- CH<sub>3</sub>CN + 0.5% HCOOH  
 B- H<sub>2</sub>O + 0.5% HCOOH

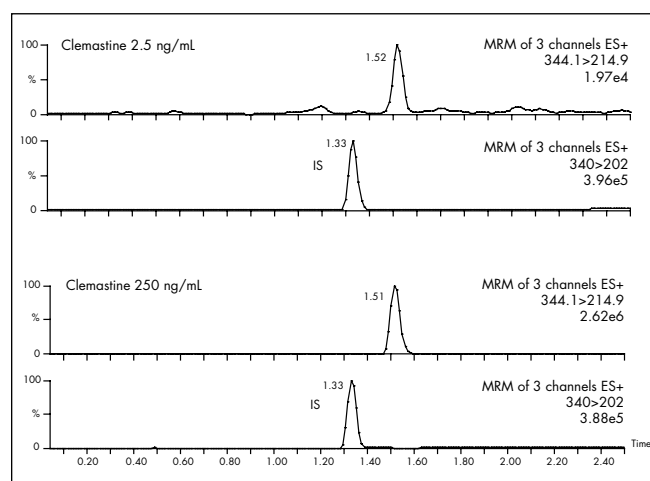
Conc. ng/ mL	Average n=6	Standard Deviation	RSD (%)
1	1.01	0.01	1.3
5	4.8	0.1	2.0
10	9.7	0.2	1.7
20	20.3	0.9	4.4
25	24.3	0.9	3.5
200	206.9	4.2	2.0
250	243.1	3.1	1.3

## On-Line Method

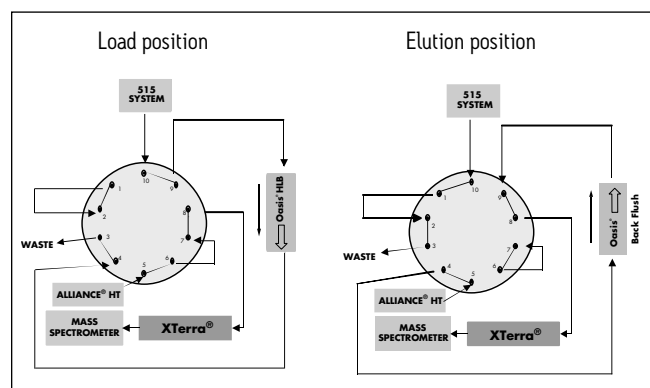
Oasis® HLB Extraction Column, 2.1 x 20 mm, 25 µm  
 Part Number 186000706

SAMPLE PREPARATION: Dilute 100 µL of rat plasma with 100 µL of I.S. solution containing 4% NH <sub>4</sub> OH in H <sub>2</sub> O
LOAD: 200 µL at 4 mL/min in 100% H <sub>2</sub> O
ELUTE: 0.4 mL/min gradient 5% CH <sub>3</sub> CN to 95% CH <sub>3</sub> CN in 1 min

## LC/MS Chromatogram of Clemastine Analysis, 2.5 ng/mL and 250 ng/mL Level



## Selector Valve Position Diagram



Reference: Mallet, C.R.; Mazzeo, J.; Neue U. Rapid Commun. Mass Spectrom. **15**:1075-1083. (2001)

# CLEMASTINE IN RAT PLASMA - OASIS ON-LINE SPE: TWO EXTRACTION COLUMNS, ONE ANALYTICAL COLUMN

## LC Conditions

Analytical Column: XTerra® MS C<sub>18</sub>, 2.1 x 30 mm, 3.5 µm  
 LC Instrument 1: Waters Alliance® 2795 - 0.4 mL/min  
 LC Instrument 2: Waters 515 - 5.0 mL/min  
 Loading Mobile phase: 100% H<sub>2</sub>O  
 Eluting Mobile phase: 1 min gradient 5% CH<sub>3</sub>CN to 95% CH<sub>3</sub>CN  
 Loading Mobile  
 Phase Additive: 0.5% HCOOH  
 Extraction Column  
 Temperature: 40 °C  
 Switching Valve: Waters Selector Valve

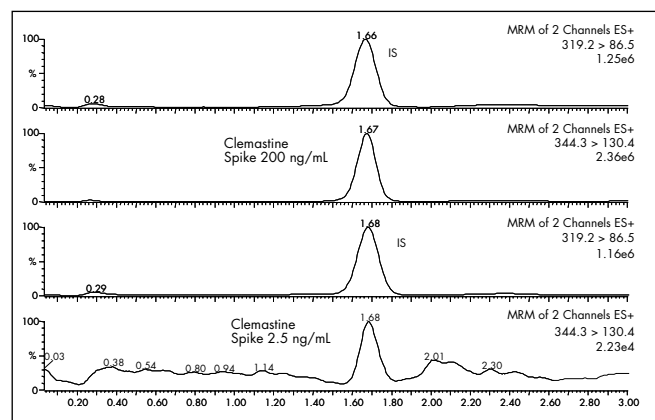
## MS Conditions

Instrument: Waters Quattro Ultima®  
 Ion Source: Electrospray Positive (ESI+)  
 Source Temperature: 150 °C  
 Desolvation Gas Flow: 600 L/hr  
 Collision Cell Pressure: 1.5 e-3 mbar  
 Cone Voltage: 20 V  
 Collision Energy: 20 eV

Time	HPLC Gradient Flow 0.4 mL/min		Valve Position
	% A	% B	Function
0.0	5	95	switch 2-position 1 to 2 (elution)
0.5			switch 3-position 2 to 1 (loading)
1.0	95	5	
1.7	95	5	
2.0	5	95	switch 2-position 2 to 1 switch 3- position 1 to 2 switch 1- position 1 to 2

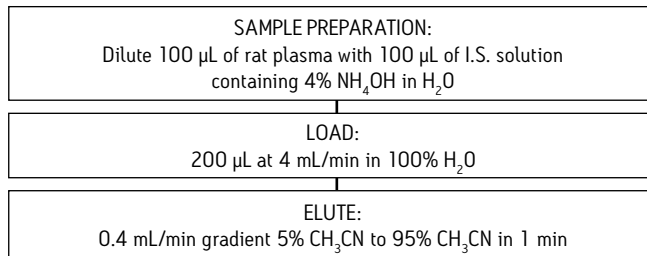
A- CH<sub>3</sub>CN + 0.5% HCOOH  
 B- H<sub>2</sub>O + 0.5% HCOOH

## LC/MS Chromatogram of Clemastine in Rat Plasma

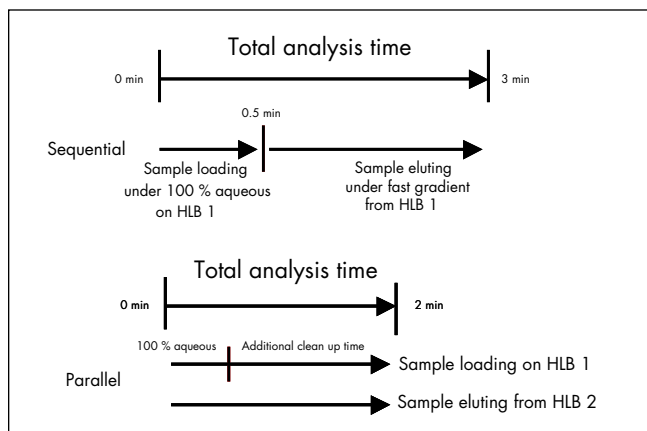


## On-Line Method

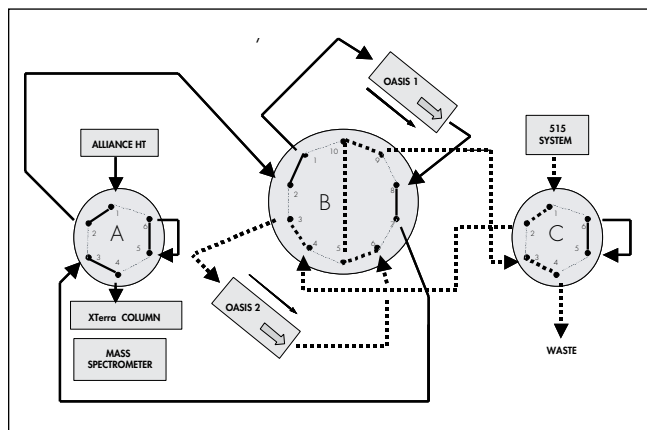
Oasis® HLB Extraction Column, 2.1 x 20 mm, 25 µm  
 Part Number 186000706



## Sequential Vs. Parallel



## Parallel On-Line Analysis



Reference: Mallet, C.R.; Mazzeo, J.; Neue U. Rapid Commun. Mass Spectrom. **15**:1075-1083. (2001)

### LC Conditions

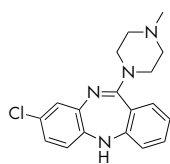
Column: SunFire™ C<sub>18</sub>, 2.1 x 50 mm, 3.5 μm  
 Mobile Phase A: 0.1% HCOOH in H<sub>2</sub>O  
 Mobile Phase B: 0.1% HCOOH in CH<sub>3</sub>OH  
 Flow Rate: 0.4 mL/min  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	95	5
5.0	5	95
7.0	5	95
7.5	95	5
10	95	5

Injection Volume: 50 μL  
 Instrument: 2777 Sample Manager and 1525μ Binary HPLC Pump

### MS Conditions

Instrument: Waters Quattro Premier™  
 Ion Source: Electrospray Positive (ESI+)  
 Source Temperature: 150 °C  
 Desolvation Temperature: 350 °C  
 Cone Gas Flow: 50 L/hr  
 Desolvation Gas Flow: 600 L/hr  
 Collision Cell Pressure: 2.2e-3 bar (Argon)

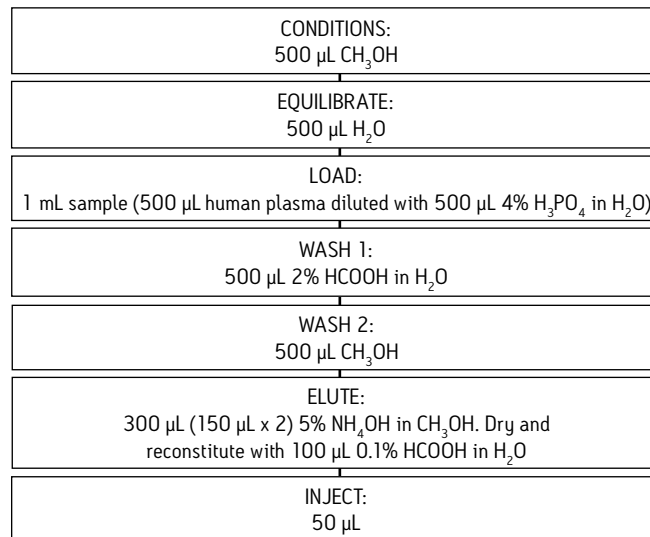


Clozapine

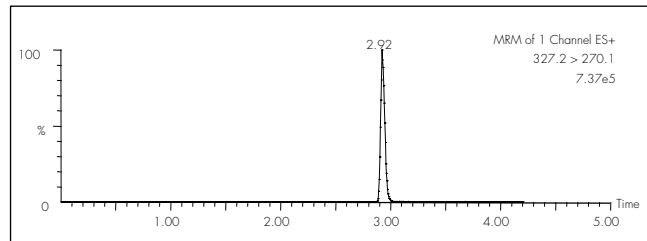
	MRM Transition (m/z)	Cone (V)	CID (eV)
Clozapine	327.2 → 270.1	30	20

### Oasis MCX Extraction Method

Oasis® MCX Plate, 10 mg/96 wells  
 Part Number 186000259

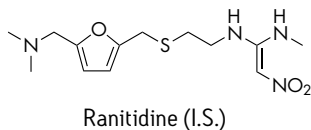
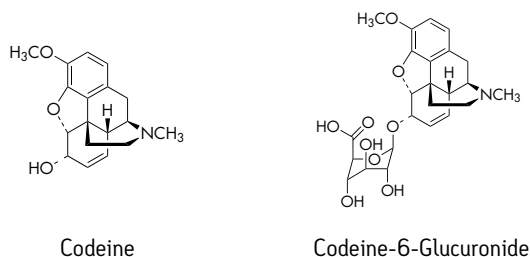


### LC/MS Chromatogram of Clozapine in Human Plasma



## LC Conditions

Column: Symmetry® C<sub>18</sub>, 2.1 x 150 mm, 3.5 µm  
 Guard Column: Symmetry C<sub>18</sub>, 2.1 x 10 mm, 3.5 µm  
 Part Numbers: Column - 186000174, Guard - 186000169  
 Mobile Phase: CH<sub>3</sub>OH/CH<sub>3</sub>CN/0.05% CF<sub>3</sub>COOH in H<sub>2</sub>O (5/5/90)  
 Flow Rate: 0.3 mL/min  
 Injection Volume: 80 µL urine extract  
 Temperature: 30° C  
 Detection: UV @ 220 nm (0.04 AUFS)

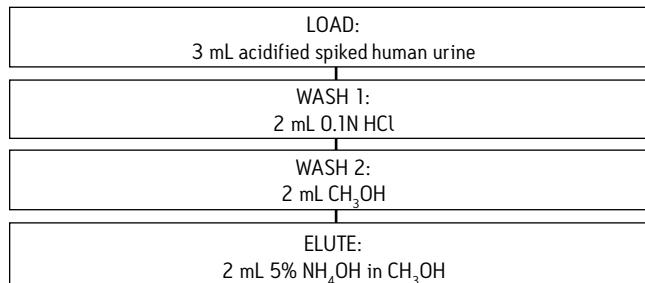


## Recovery Data

Spike Level	% Recovery (% RSD)	
	0.12 µg/mL	0.6 µg/mL
Codeine (n=3)	88.5 (2.7)	99.5 (1.2)
Codeine-6-Glucuronide (n=3)	99.3 (5.4)	98.7 (0.4)
Codeine Interday (n=6)	102.5 (3.5)	105.4 (4.0)
Codeine Interperson (n=9)	91.6 (7.1)	104.1 (5.4)
C-6-G Interday (n=6)	116.1 (9.4)	111.5 (3.3)
C-6-G Interperson (n=9)	102.4 (7.7)	107.4 (7.3)

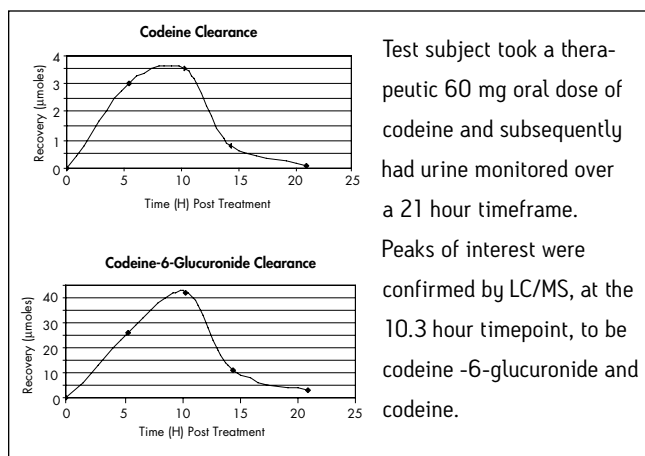
## Oasis MCX Extraction Method

Oasis® MCX Extraction Cartridge, 3 cc/60 mg  
 Part Number 186000254

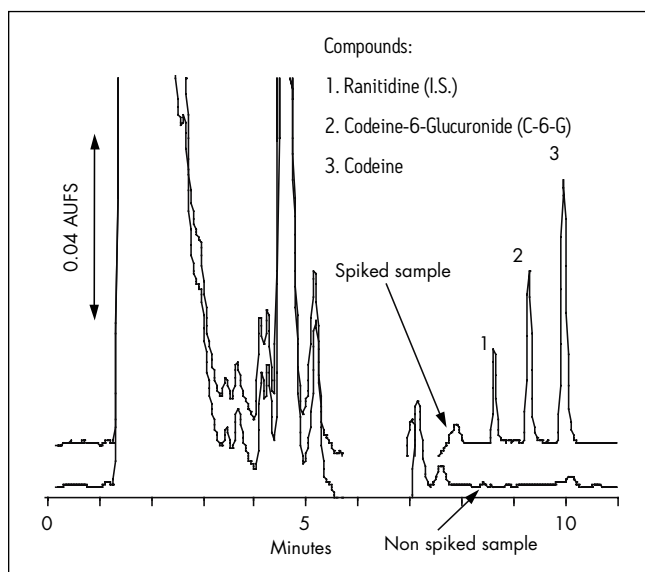


After elution, the extract is diluted 1/3 with H<sub>2</sub>O for HPLC analysis

## Clearance Study From Human Urine

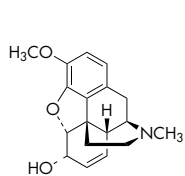


## HPLC Chromatogram of Codeine and its Glucuronide Metabolite in Human Urine

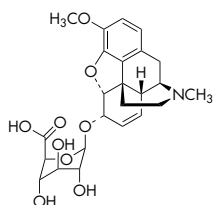


**LC Conditions**

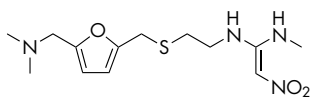
Column: Symmetry® C<sub>18</sub>, 2.1 x 150 mm, 3.5 µm  
 Guard Column: Symmetry C<sub>18</sub>, 2.1 x 10 mm, 3.5 µm  
 Part Numbers: Column - 186000174, Guard - 186000169  
 Mobile Phase: CH<sub>3</sub>OH/ CH<sub>3</sub>CN/ 0.05% CF<sub>3</sub>COOH in H<sub>2</sub>O (5/5/90)  
 Flow Rate: 0.3 mL/min  
 Injection Volume: 80 µL urine extract  
 Temperature: 30° C  
 Detection: UV @ 220 nm (0.04 AUFS)



Codeine



Codeine-6-Glucuronide



Ranitidine (I.S.)

Spike Level	% Recovery (% RSD) n=3	
	0.12 µg/ mL	0.6 µg/mL
Codeine	95.4 (5.0)	103.4 (0.9)
Codeine-6-Glucuronide	106.8 (1.9)	104.6 (1.3)

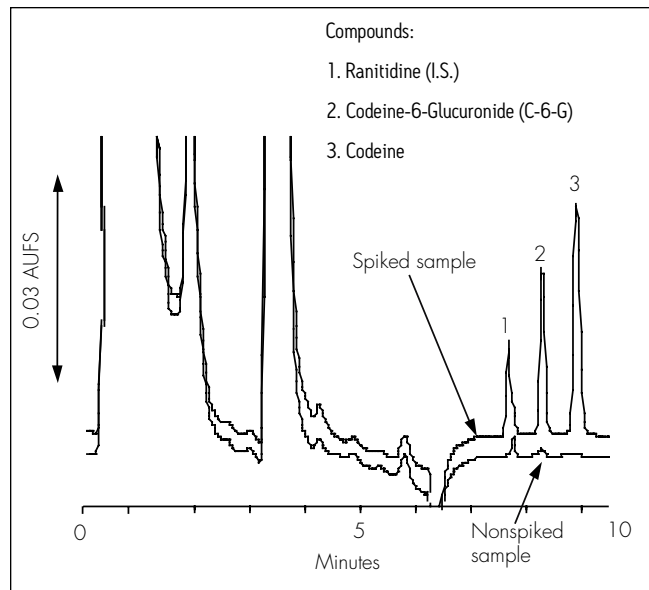
**Oasis MCX Extraction Method**

Oasis® MCX Extraction Cartridge, 3 cc/60 mg  
 Part Number 186000254

NO CONDITIONING
LOAD: 3 mL acidified plasma sample (1.5 mL plasma diluted 1/1 with 4% H <sub>3</sub> PO <sub>4</sub> in H <sub>2</sub> O)
WASH 1: 2 mL 0.1 N HCl
WASH 2: 2 mL CH <sub>3</sub> OH
ELUTE: 2 mL 5% NH <sub>4</sub> OH in CH <sub>3</sub> OH

After elution, the extract is diluted 1/3 with H<sub>2</sub>O for HPLC analysis

**LC/MS Chromatogram of Codeine and its Glucuronide Metabolite in Human Plasma**



## Principles of Desalting

DNA oligonucleotides are retained on the Oasis® HLB sorbent by ion-pair reversed-phase mechanism. A volatile ion-pair agent is used as loading buffer\* (triethylammonium acetate, pH ~ 7).

The Oasis HLB µElution 96-well plate has sufficient capacity for desalting from 1 pmol up to 5000 pmol of oligonucleotides sample.

## Oasis HLB Desalting Method

Oasis HLB 96-well µElution Plate

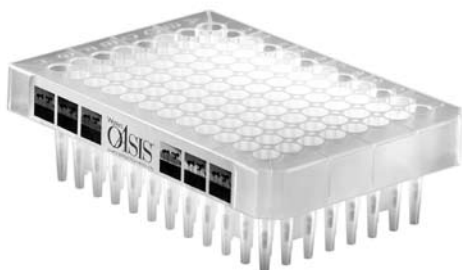
Part Number 186001828BA

<p>CONDITION: 200 µL CH<sub>3</sub>CN/ H<sub>2</sub>O (70/30)</p>
<p>EQUILIBRATE: 200 µL H<sub>2</sub>O</p>
<p>LOAD: Load solution onto plate at 1 mL/min or less (Low loading speed prevents breakthrough of oligonucleotides)</p>
<p>WASH 1: 800 µL of 0.1 M TEAAc* buffer (to remove salts)</p>
<p>WASH 2: 200 µL of H<sub>2</sub>O (to remove excess buffer and salts)</p>
<p>ELUTE: 25 µL of 70% CH<sub>3</sub>CN, using a vacuum manifold. Alternatively, centrifuge plate with 10 µL of 70% CH<sub>3</sub>CN</p>
<p>Lyophilize eluent to complete dryness using SpeedVac Dissolve sample in MALDI matrix solution</p>

\*0.1 M (C<sub>2</sub>H<sub>5</sub>)<sub>3</sub>NHOCOCH<sub>3</sub>, pH 7

# DESALTING OF PEPTIDE MIXTURE PRIOR TO ESI-MS ANALYSIS (CYTOCHROME C TRYPSIN DIGEST)

## Oasis $\mu$ Elution Plate



## MS Conditions

Instrument : Waters LCT System  
Capillary Voltage: 2.8 kV  
Sample Cone Voltage: 35 V  
Extraction Cone Voltage: 5 V  
Gas Flow: 520 and 310 L/h

Peptide mixture prepared by digestion of cytochrome c by trypsin in 20 mM trisglycine buffer.

## Oasis HLB Desalting Method

Oasis<sup>®</sup> HLB 96-well  $\mu$ Elution plate  
Part Number 186001828BA

CONDITION: 200 $\mu$ L CH <sub>3</sub> CN
EQUILIBRATE: 200 $\mu$ L 0.1% CF <sub>3</sub> COOH
LOAD: 0.1-0.5 mL of sample in 0.1% CF <sub>3</sub> COOH solution (at 1 mL/min or less; low loading speed prevents breakthrough of peptides)
WASH 1: 800 $\mu$ L of 0.1% CF <sub>3</sub> COOH solution (to remove salts)
WASH 2: 200 $\mu$ L of H <sub>2</sub> O (to remove excess buffer and salts)
ELUTE: 50 $\mu$ L of 70% CH <sub>3</sub> CN using a vacuum manifold (Alternatively, centrifuge plate with 25 $\mu$ L of 70% CH <sub>3</sub> CN)
Analyze by ESI-MS

Reference: M. Gilar, A. Belenky, B. H. Wang, *J. Chromatog. A*, **921** 3-13 (2001)

# DETERMINATION OF CLOPYRALID AND TRICLOPYR IN RIVER WATER BY LC/MS

## LC/MS Conditions

Column: XTerra® MS C<sub>18</sub>, 2.1 x 100 mm, 3.5 µm  
 Part Number: 186000404  
 Mobile Phase A: 10 mM CF<sub>3</sub>COOH, pH 2.1  
 Mobile Phase B: CH<sub>3</sub>CN  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	75	25
6.0	10	90

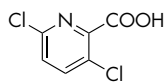
Flow Rate: 0.2 mL/min  
 Injection Volume: 20 µL  
 Ion Source: Electrospray Positive (ESI<sup>+</sup>)  
 Mode: Multiple Selected-Ion Recording (SIR)  
 Instrument: Waters Alliance® 2695  
 Detector: Waters ZQ™

## LC/UV Method

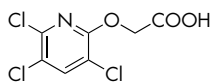
Column: XTerra® RP18, 4.6 x 100 mm, 3.5 µm  
 Part Number: 186000438  
 Mobile Phase A: 10 mM CF<sub>3</sub>COOH, pH 2.1  
 Mobile Phase B: CH<sub>3</sub>CN  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	80	20
20.0	20	80

Flow Rate: 1 mL/min  
 Injection Volume: 50 µL  
 Detection: UV @ 290 nm  
 Instrument: Waters Alliance 2695



Clopyralid

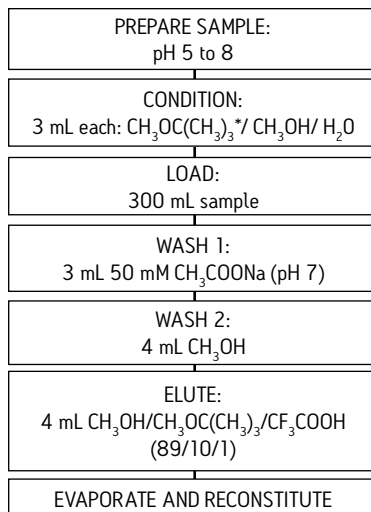


Triclopyr

	% Recovery (% RSD) n=5			
	Drinking Water		River Water	
	0.4 µg/L	2.0 µg/L	0.4 µg/L	2.0 µg/L
1. Clopyralid	100 (8)	110 (4)	94 (5)	110 (2)
2. Triclopyr	85 (3)	87 (2)	82 (11)	81 (8)

## Oasis MAX Extraction Method

Oasis® MAX Extraction Cartridge, 6 cc/500 mg  
 Part Number 186000865

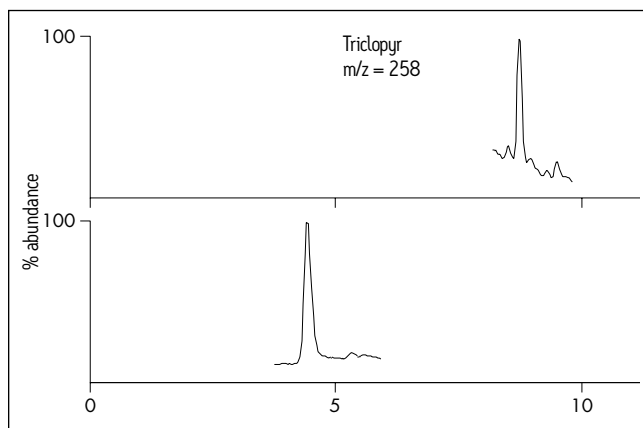


MTBE is employed as elution solvent to minimize humic interference from surface H<sub>2</sub>O. Therefore precondition with this solvent.

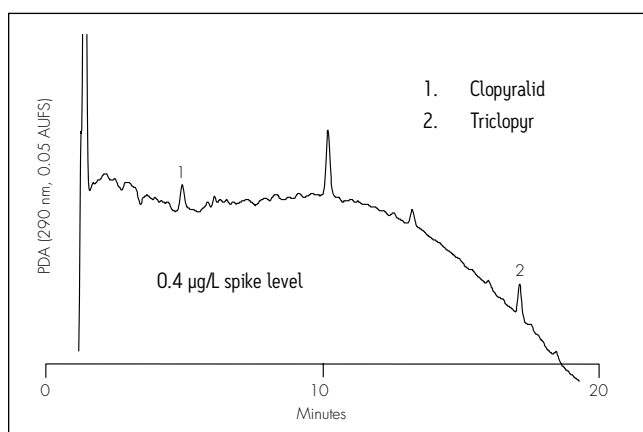
Clopyralid is a stronger acid than HCOOH. Therefore, HCOOH cannot be utilized to elute this compound from Oasis MAX sorbent. TFA was employed for elution of clopyralid.

\*(C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>O can be used as an alternative to CH<sub>3</sub>OC(CH<sub>3</sub>)<sub>3</sub>

## LC/MS Chromatogram of Clopyralid and Triclopyr in River Water, 0.4 µg/L Spike Level



## LC/UV Chromatogram of Clopyralid and Triclopyr in River Water, 0.4 µg/L Spike Level



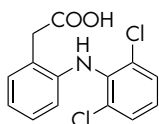


**LC Conditions**

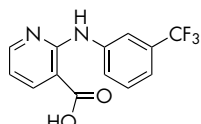
Column: XTerra® MS C<sub>18</sub>, 2.1 x 30 mm, 3.5 µm  
 Part Number: 186000398  
 Mobile Phase A: 0.05% HCOOH  
 Mobile Phase B: CH<sub>3</sub>CN  
 Isocratic Mobile  
 Phase Composition: 60% A; 40% B  
 Flow Rate: 0.2 mL/min  
 Injection Volume: 50 µL  
 Detection: Electrospray Negative (ESI)  
 Instrument: Waters Alliance® 2790

**MS Conditions**

Instrument: Waters Quattro Ultima®  
 Ion Source: Electrospray Negative (ESI)  
 Source Temperature: 150 °C  
 Collision Cell Pressure: 1.5 e-3 mbar, 12 eV  
 Desolvation Temperature: 350 °C  
 Cone Gas Flow: 150 L/hr  
 Drying Gas Flow: 600 L/hr  
 Cone Voltage: 30 V



Diclofenac

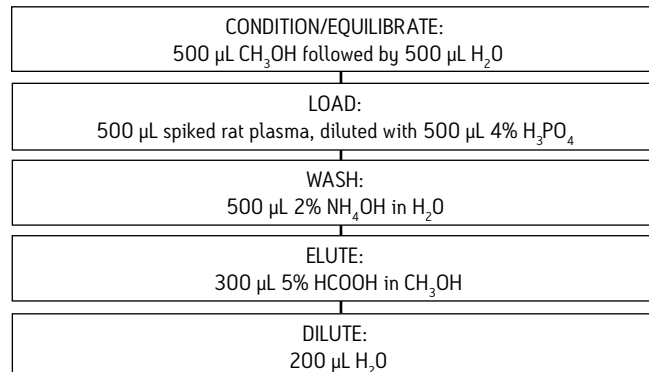


Niflumic Acid (I.S.)

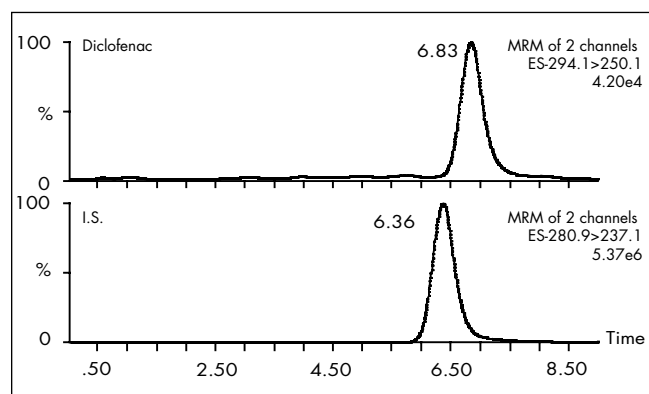
Diclofenac (ng/ mL)	Mean	Recovery (%)	Standard Deviation	Coefficient of Variation (%)
10	10.26	102	0.3	3
25	24.06	96	0.83	3.5
100	101.68	101	2.69	2.7
250	250.05	100	8.13	3.3
500	496.41	99	27.09	5.5
1000	997.8	99	28.1	2.8
1500	1497.25	99	27.98	1.9

**Oasis MAX Extraction Method**

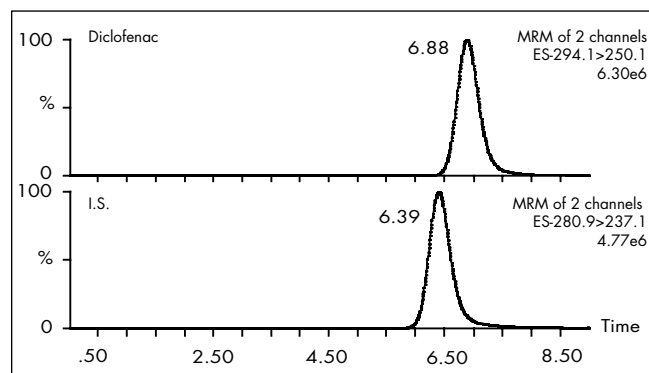
Oasis® MAX Extraction Plate, 10 mg/96 wells  
 Part Number 186000375



**LC/MS Chromatogram of Diclofenac in Rat Plasma, 10 ng/mL Spiked Level**



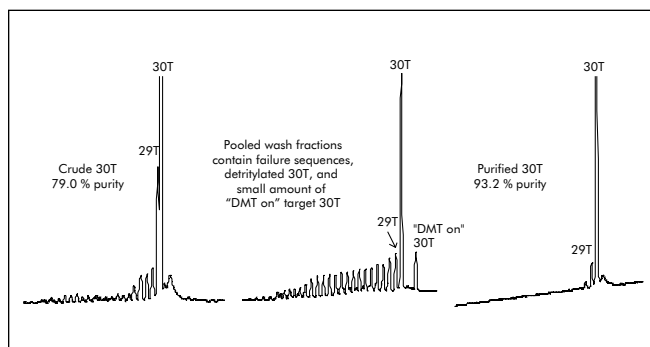
**LC/MS Chromatogram of Diclofenac in Rat Plasma, 1500 ng/mL Spiked Level**



# “DMT ON” PURIFICATION OF DNA OLIGONUCLEOTIDES < 35 MER USING OASIS HLB SPE PRODUCTS

Oasis® HLB EXTRACTION METHOD			96-well plate 30 mg WAT058951	3 cc cartridge 60 mg WAT094226	6 cc cartridge 200 mg WAT106202	
STEP		SYNTHESIS SCALE	0.1-0.2 µmol	0.2 µmol	0.1-1 µmol	
1	CONDITION: CH <sub>3</sub> CN	Organic solvent wets (conditions) the sorbent and frits	1 mL	2 mL	2 mL	GRAVITY FLOW
2	EQUILIBRATION: 0.1 M TEAAc*, pH 7	Removes CH <sub>3</sub> CN and equilibrates sorbent with TEAAc*	1 mL	2 mL	2 mL	
3	SAMPLE LOAD: in 0.1 M TEAAc*, pH 7	Retains target oligonucleotide and failure sequences	1 mL	2 mL	3 mL	
4	WASH 1: 8% CH <sub>3</sub> CN in 0.1 M TEAAc*, pH 7 (v/v)	Remove weakly retained failure sequences	1 mL	2 mL	3 mL	VACUUM FLOW 1-2 mL/minute
5	WASH 2: 12% CH <sub>3</sub> CN in 0.1 M TEAAc*, pH 7 (v/v)	Remove strongly retained failure sequences	1 mL	2 mL	3 mL	
6	DETRITYLATE: 2% CF <sub>3</sub> COOH Apply half of the volume by vacuum, release vacuum, wait 1 minute, then resume vacuum	On cartridge cleavage of DMT*** group from target oligonucleotide	1 mL	2 mL	3 mL	
7	ELUTE: 20% CH <sub>3</sub> CN in 0.36 M TEAAc**, pH 11.3	Neutralizes CF <sub>3</sub> COOH, dissolves and elutes target oligonucleotide	1 mL	2 mL	2 mL	

## Capillary gel electrophoresis analysis of fractions from oligodeoxythymine (30 mer) SPE purification



\*0.1 M (C<sub>2</sub>H<sub>5</sub>)<sub>3</sub> NHOCOH<sub>3</sub>, pH 7 Buffer – commercially available

\*\* For 100 mL of 0.36 M (C<sub>2</sub>H<sub>5</sub>)<sub>3</sub> NHOCOH<sub>3</sub> pH 11.3 buffer:

Mix 94.5 mL of MilliQ™ water and 0.5 mL of glacial acetic acid.

While mixing slowly add 5 mL of (C<sub>2</sub>H<sub>5</sub>)<sub>3</sub> N; mix until it dissolves.

pH of final 0.36 M solution is approximately 11.3 (desirable values are between 10.8-11.5)\*

\* Keep in closed polypropylene bottle. Handle in hood, (C<sub>2</sub>H<sub>5</sub>)<sub>3</sub>N; has a strong odor.

\*\*\* DMT= dimethoxytrityl

## Troubleshooting

Flow rates of > 0.5 mL/min in the load step (step 3) will cause sample breakthrough which reduces oligonucleotide recovery in final elution (step 7).

## Recovery Calculation

Recovery of target oligonucleotide is determined by analysis with a UV absorbance spectrometer.

Take 10 µL of sample solution (prior to loading), dilute to 1 mL and measure Absorbance A<sub>260</sub>(L).

Take 10 µL of final eluate (step 7), dilute to 1 mL and measure absorbance A<sub>260</sub>(E).

V<sub>E</sub> = elution volume from step 7

V<sub>L</sub> = elution volume from step 3

$$\text{Recovery (\%)} = \left[ \frac{A_{260}(E)}{A_{260}(L)} \times 100 \right] \times \frac{V_E}{V_L}$$

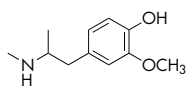
Source: M. Gilar, E.S.P. Bouvier, J. Chromatography A **890** (1), 167-177. (2001)

**LC Conditions**

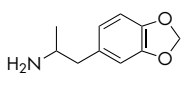
Column: XTerra® MS C<sub>18</sub>, 2.1 x 150 mm, 3.5 µm  
 Part Number: 186000408  
 Mobile Phase A: 20 mM NH<sub>4</sub>HCO<sub>3</sub>, pH 9.0  
 Mobile Phase B: CH<sub>3</sub>OH  
 Flow Rate: 0.2 mL/min  
 Isocratic Mobile  
 Phase Composition: 70% A; 30% B  
 Injection Volume: 15 µL  
 Temperature: 30 °C  
 Instrument: Waters Alliance® 2695

**MS Conditions**

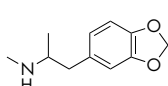
Instrument: Waters ZQ™  
 Ion Source: Electrospray Positive (ESI+)  
 Source Temperature: 150 °C  
 Desolvation Temperature: 350 °C



HMMA



MDA

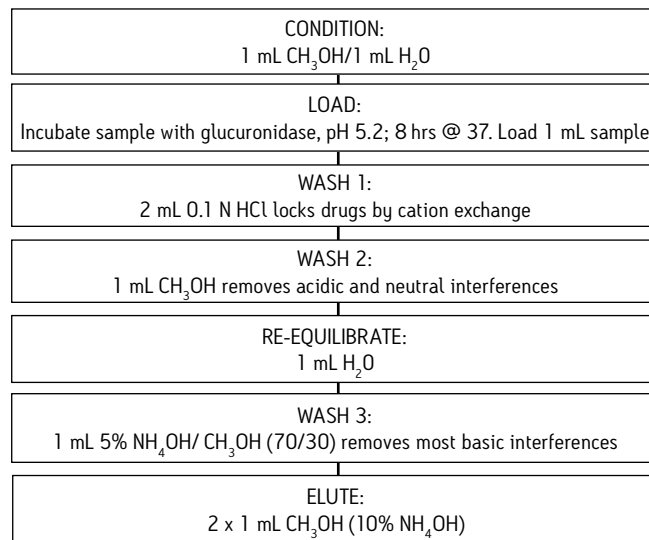


MDMA

Analytes	Recovery (%)	Concentration (µg/mL)	RSD (%)
MDMA	108.0	0.10	9.8
	89.3	0.50	4.9
	88.1	1.25	4.6
	98.8	2.50	3.7
	99.9	5.00	5.7
MDA	103.0	0.10	8.8
	84.2	0.50	13.9
	83.8	1.25	9.8
	95.4	2.50	9.0
	104.5	5.00	13.4
HMMA	93.	20.00	13.1
	90.5	0.04	8.2
	88.1	0.25	4.5
	84.8	0.50	5.4
	94.8	1.00	4.0
100.0	2.00	5.3	
97.9	8.00	11.4	

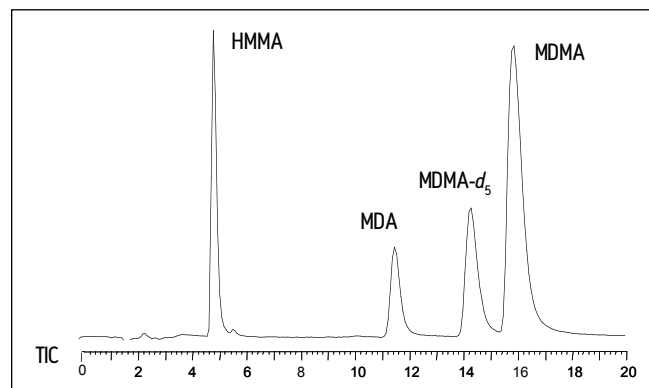
**Oasis MCX Extraction Method**

Oasis® MCX Extraction Cartridge, 1 cc/60 mg  
 Part Number 186000252



Ions Monitored					
MDMA		HMMA		MDA	
Ion (m/z)	Core (v)	Ion (m/z)	Cone (v)	Ion (m/z)	Cone (v)
194.11	25.0	196.16	20.0	180.0	20.0
163.08	37.5	165.08	37.5	163.08	37.5
135.00	55.0	137.00	55.0	135.00	55.0

**LC/MS Chromatogram MDMA and Metabolites**



Compounds:

- 4-Hydroxy-3-methoxymetamphetamine (HMMA)
- 3,4-Methylenedioxyamphetamine (MDA)
- MDMA-d<sub>5</sub> (I.S.)
- 3,4-Methylenedioxymethamphetamine (MDMA)

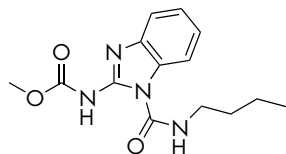
Analysis at pH 9 allows good peak shape and maximum retention for basic compounds with no modifiers that can interfere with LC/MS analysis. Do not try this with traditional silica based columns!

**LC Conditions**

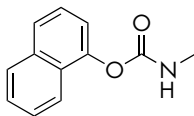
Column: Symmetry® C<sub>18</sub>, 3.9 x 150 mm, 5 µm  
 Part Number: WAT046970  
 Mobile Phase A: 10 mM K<sub>2</sub>HPO<sub>4</sub>/KH<sub>2</sub>PO<sub>4</sub>, pH 6.8  
 Mobile Phase B: CH<sub>3</sub>OH  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	60	40
20	0	100

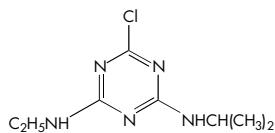
Flow Rate: 1 mL/min  
 Injection Volume: 100 µL  
 Sample: 10 g potting soil extracted with 25 mL CH<sub>3</sub>CN; then SPE on Oasis® HLB  
 Detection: PDA (225 nm, 0.04 AUFS)



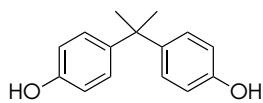
Benomyl



Carbaryl



Atrazine



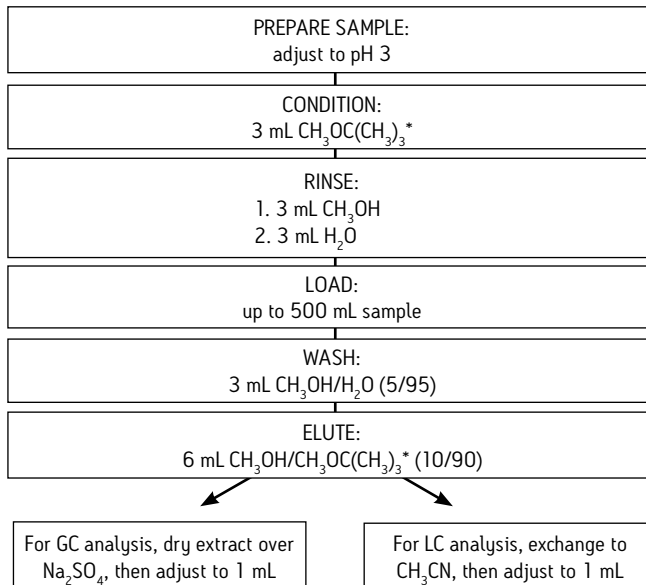
Bisphenol A

Compounds	% Recovery (% RSD)
1. Benomyl	62 (± 6)
2. Carbaryl	91 (± 4)
3. Atrazine	84 (± 5)
4. Bisphenol A	78 (± 6)

Soil samples (5 g) were spiked with the appropriate compounds and extracted with 25 mL of acetonitrile (30 minutes on shaker). A 5 mL aliquot of the acetonitrile extract was diluted to 100 mL with reagent water (MilliQ™) and then processed by SPE.

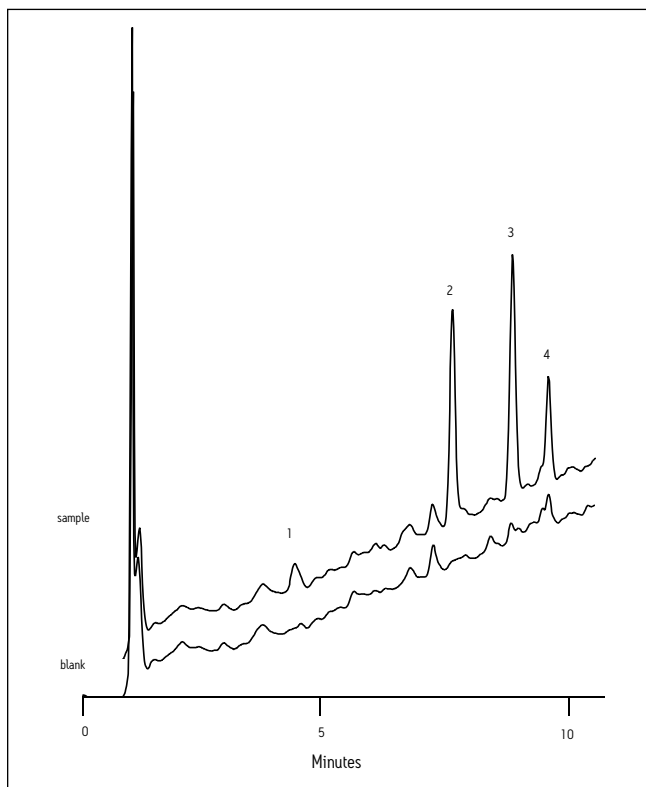
**Oasis HLB Extraction Method**

Oasis HLB Extraction Cartridge, 6 cc/200 mg  
 Part Number WAT106202



\* (C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>O can be used as an alternative to CH<sub>3</sub>OC(CH<sub>3</sub>)<sub>3</sub>

**HPLC Chromatogram of Endocrine Disruptors in Soil, 50 ppb Spiked Level**



Sample Pretreatment before SPE

Aqueous Samples

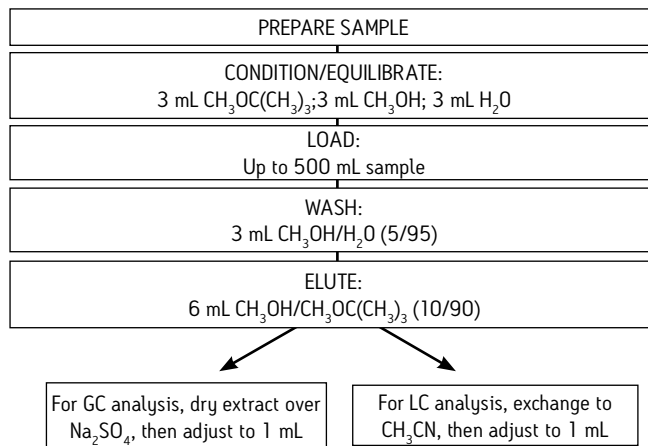
- Use 250 - 1000 mL of sample for H<sub>2</sub>O analysis
- Use 40 - 200 mL of sample for beverage analysis
- Centrifuge prior to analysis if necessary

Soil and Food Samples

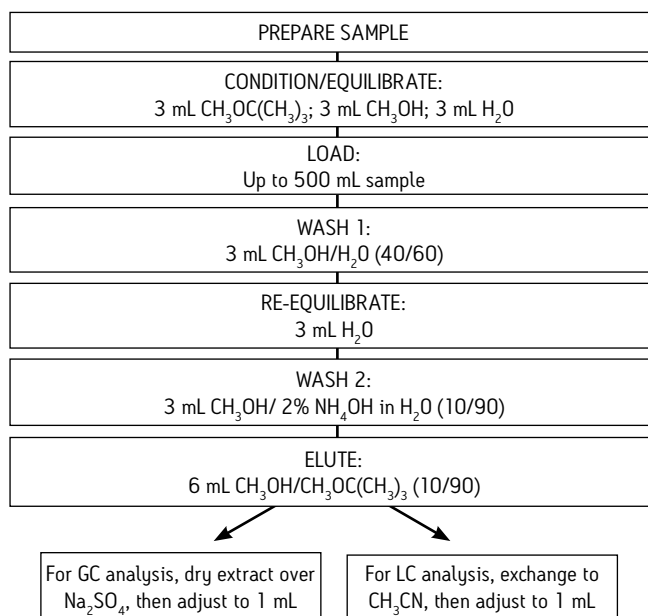
- Extract the homogenized sample (5 - 10 gm) with 20 - 40 mL CH<sub>3</sub>CN or other water miscible solvent
- If desired, concentrate the organic extract by evaporation
- Dilute the organic extract with water (85 - 95% water)

Oasis HLB Extraction Method

Oasis® HLB Extraction Cartridge, 200 mg; 1-D (LOQ = 50 ppt)  
Part Number 186000683 (Glass Recommended)



Oasis HLB Extraction Cartridge 200 mg; 2-D (LOQ < 5 ppt)  
Part Number 186000683 (Glass Recommended)



# ENDOTHALL IN DRINKING WATER OR SOIL BY LC/MS AND GC/MS

## LC/MS Conditions

Column: SymmetryShield™ RP8, 2.1 x 100 mm, 3.5 µm  
 Part Number: WAT058969  
 Mobile Phase: 5% CH<sub>3</sub>CN in 1% HCOOH/ H<sub>2</sub>O  
 Flow Rate: 200 µL/min  
 Injection Volume: 75 µL  
 Ion Source: Electrospray Negative (ESI<sup>-</sup>)  
 Mode: Multiple Selected-Ion Recording (SIR) m/z 185  
 Instrument: Waters Alliance® LC/MS with Waters Platform LC™ Mass Detector

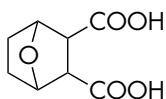
*For LC/MS: No derivatization required. The CH<sub>3</sub>OC(CH<sub>3</sub>)<sub>3</sub> in the eluent is removed by evaporation and the extract is adjusted to a final volume of 1 mL with 10% methanol in water.*

## GC/MS or GC/FID Conditions

Column: RTX 5 capillary, 30 meters, 0.25 mm ID, 0.25 µm film thickness  
 Carrier Gas: Helium @ 30 cm/sec  
 Temperature Program: 40 °C initial, 8 °C/min to 300 °C  
 Injection Volume: 1 µL  
 Detection: HP 5972 MSD, (EI, SIM mode, m/z=123)

For GC: The eluent is heated for 40 min @ 60 °C to convert endothall to the dimethyl ester. The ester is then extracted with CH<sub>2</sub>Cl<sub>2</sub>. After removal of water by treatment with ammonium sulfate, the CH<sub>2</sub>Cl<sub>2</sub> extract is evaporated to a final volume of 0.5 mL.

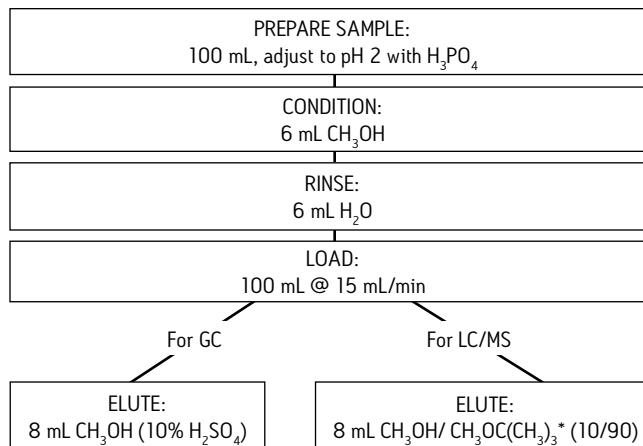
Soil Samples: The sample (10 g) is extracted with 35 mL pH 10 carbonate buffer (0.1 M) followed by 20 mL of water. The combined extracts are adjusted to pH 2 with phosphoric acid and centrifuged. SPE is then performed using the same protocol as water samples.



Endothall

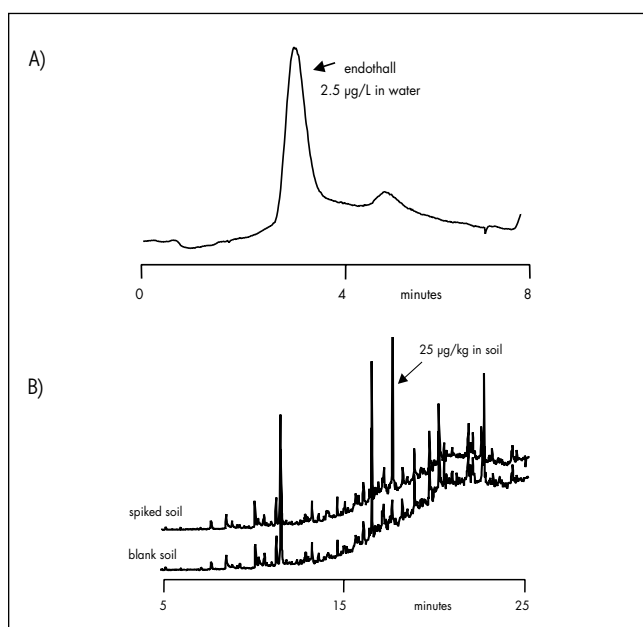
## Oasis HLB Extraction Method

Oasis® Extraction Cartridge, 6 cc/500 mg LP  
 Part Number 186000115



\* (C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>O can be used as an alternative to CH<sub>3</sub>OC(CH<sub>3</sub>)<sub>3</sub>

## LC/MS Chromatogram of Endothall in Drinking Water and Soil



	LC/MS (n=4)		GC (n=4)	
	Tap Water	Tap Water	Soil (GC/FID)	Soil (GC/MS)
Spike Level	2.5 µg/L	10 µg/L	100 µg/L	25 µg/L
Recovery (%)	81.1	99.6	81.8	76.2
RSD (%)	18	3.1	20	9.5

**LC Conditions**

Column: XTerra® Phenyl, 2.1 x 150 mm, 3.5 µm  
 Part Number: 186001181  
 Mobile Phase A: 10 mM NH<sub>4</sub>HCO<sub>3</sub>, pH 9.5  
 Mobile Phase B: CH<sub>3</sub>CN  
 Flow Rate: 0.23 mL/min  
 Isocratic Mobile  
 Phase Composition: 90% A; 10% B  
 Injection Volume: 5 µL  
 Temperature: 40 °C  
 Detection: UV @ 254 nm  
 Instrument: Waters Alliance® 2695, Waters 2996 PDA

**Sample Preparation**

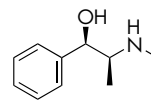
- Accurately weigh 1 gram of ephedra sample into a 100 mL volumetric flask, add 20 mL water and mix. Add 50 mL of methanol and 1 mL of Internal Standard.
  - Use 0.5 g sample for ephedra extracts
  - Use 10 g sample weight for high protein powdered drinks and other functional foods
- Sonicate for 1 hour at ambient temperature.
- Cool and bring to volume with methanol.
- Allow the suspended solids to settle; preferably centrifuge.
- Filter a 3 mL aliquot through a 0.45 mm filter before a sample preparation.
- This step is critical to good SPE recovery.
- For SPE, dilute 2 mL of filtered sample extract to 10 mL with 0.1% formic acid (aqueous).

	50% Level % Recovery RSD (%)	100% Level % Recovery RSD (%)	150% Level % Recovery RSD (%)
1 NE	81.9 ± 6.7	74.7 ± 5.9	66.7 ± 3.7
2 NPE	77.6 ± 1.8	66.2 ± 0.8	63.9 ± 0.73
3 E	101.9 ± 2.5	102.3 ± 3.7	98.0 ± 5.5
4 PE	89.0 ± 1.2	93.2 ± 3.4	92.8 ± 5.3
5 ME	94.7 ± 4.2	98.7 ± 3.3	81.9 ± 1.8
6 MPE	99.8 ± 16.3	85.4 ± 13.3	88.0 ± 7.7

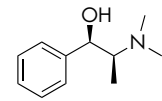
**Oasis MCX Extraction Method**

Oasis® MCX Extraction Cartridge, 3 cc/60 mg  
 Part Number 186000254

CONDITION: 1 mL CH <sub>3</sub> OH/1 mL H <sub>2</sub> O
LOAD: 10 mL sample
WASH 1: 2 mL 0.1 N HCl
WASH 2: 1 mL CH <sub>3</sub> OH
RE-EQUILIBRATE: 1 mL H <sub>2</sub> O
WASH 3: 1 mL 5% NH <sub>4</sub> OH/CH <sub>3</sub> OH (75/25)
ELUTE: 1.5 mL 95% CH <sub>3</sub> OH/5% NH <sub>4</sub> OH (95/5)
Adjust to 5.0 mL final volume: 10 mM NH <sub>4</sub> HCO <sub>3</sub> , pH 9.45

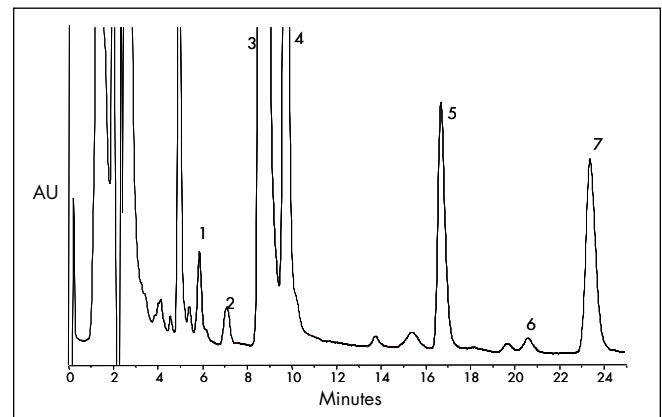


Ephedrine



N-Methylephedrine

**HPLC Chromatogram of Multi-Herb Dietary Supplement Capsule**



- |                                      |   |
|--------------------------------------|---|
| 1. Norephedrine (NE) 0.24mg/g        | 4. Pseudoephedrine (PE) 5.0mg/g           |
| 2. Norpseudoephedrine (NPE) 0.40mg/g | 5. N-Methylephedrine (ME) 0.70mg/g        |
| 3. Ephedrine (E) 20.0mg/g            | 6. N-Methylpseudoephedrine (MPE) 0.17mg/g |

# ESTROGENS IN RIVER WATER AT 5 NG/L (ENDOCRINE DISRUPTORS) BY LC/MS

## LC Conditions

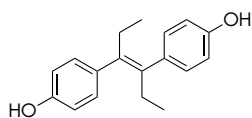
Column: XTerra® MS C<sub>18</sub>, 2.1 x 100 mm, 3.5 μm  
 Part Number: 186000404  
 Mobile Phase A: NH<sub>4</sub>OH in H<sub>2</sub>O, pH 10.5  
 Mobile Phase B: CH<sub>3</sub>CN  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	70	30
8	35	65
9	10	90

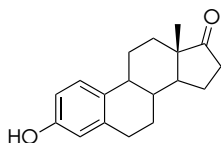
Injection Volume: 20 μL  
 Flow Rate: 200 μL/min, plumbed directly to detector  
 Instrument: Waters Alliance® Separations Module

## MS Conditions

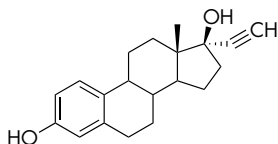
Instrument: Waters Platform LC™  
 Ion Source: Electrospray Negative (ESI-)  
 Mode: Multiple Selected-Ion Recording (SIR)  
 Cone Voltage: 27 V



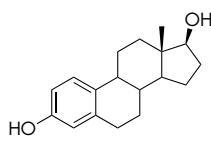
Diethylstilbestrol



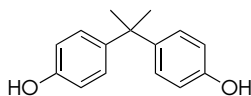
Estrone



Ethynylestradiol



Estradiol



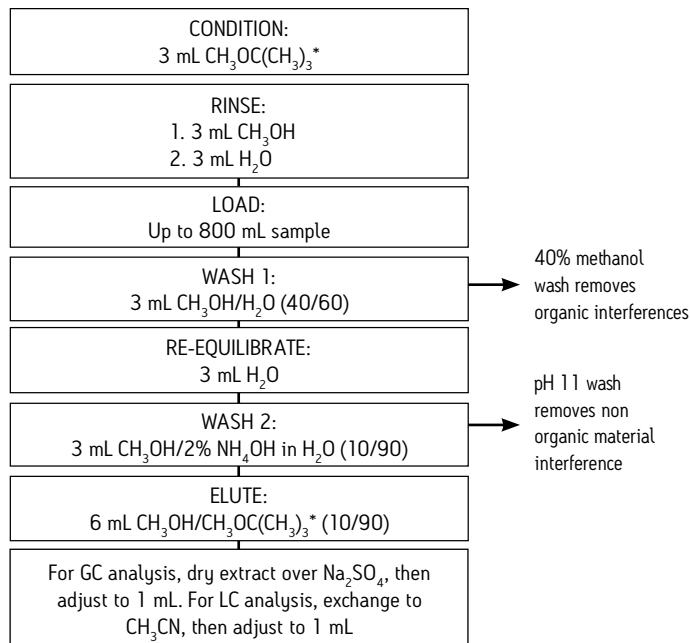
Bisphenol A

Compounds	% Recovery (% RSD), n=4
1. Diethylstilbestrol	75 ± 5
2. Estrone	87 ± 5
3. Ethynylestradiol	94 ± 12
4. Estradiol	93 ± 15
5. Bisphenol A	113 ± 11

\*5 ng/L spike level

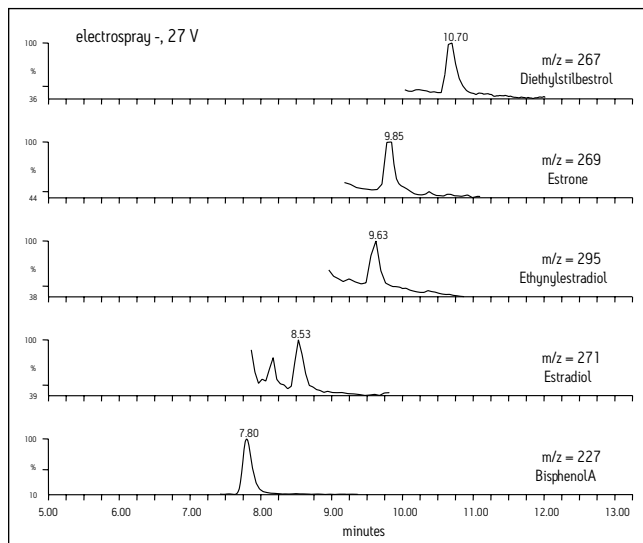
## Oasis HLB Extraction Method

Oasis® HLB Glass Cartridge, 5 cc/200 mg  
 Part Number 186000683  
 Modifications for low ppt analysis by LC/MS and GC/MS



\* (C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>O can be used as an alternative to CH<sub>3</sub>OC(CH<sub>3</sub>)<sub>3</sub>

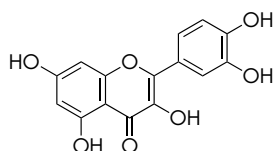
## LC/MS Chromatogram of Estrogens Compounds in River Water



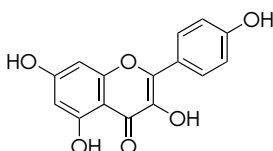


### LC Conditions

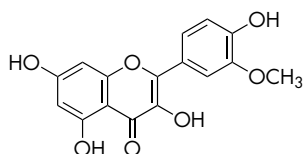
Column: Symmetry® C<sub>18</sub>, 4.6 x 250 mm, 5 µm  
 Part Number: WAT054275  
 Mobile Phase: 0.5% H<sub>3</sub>PO<sub>4</sub>/CH<sub>3</sub>OH (50/50)  
 Flow Rate: 1.5 mL/min  
 Injection Volume: 10 µL  
 Temperature: 25 °C  
 Detection: UV @ 270 nm (0.02 AUFS)



Quercetin



Kaempferol



Isorhamnetin

Compounds	Recovery (%), n=4
1. Quercetin	82
2. Kaempferol	> 90
3. Isorhamnetin	> 90

total: 11 mg / tablet

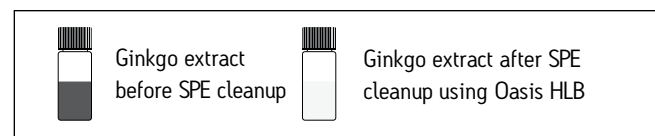
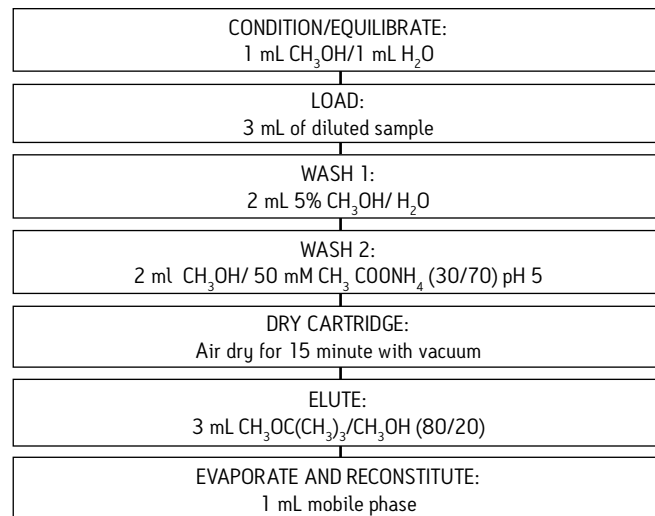
### Sample Pre-preparation

1 g sample is refluxed in 50 mL of ethano/3 M HCl (70/30) for 2.5 hr  
 The cooled sample is adjusted to exactly 100 mL  
 0.3 mL of the ethanolic extract is diluted 1/10 with water

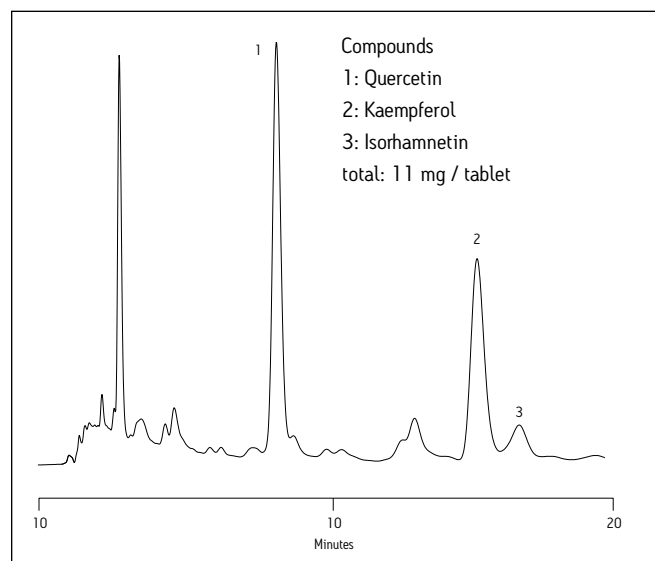
- Recovery, measured with certified standards at 100 ppm in reagent water, was 82% for quercetin and > 90% for the other compounds
- All analyses gave results within ± 40% of the expected values with the exception of the capsule (+ 60%)
- The selective SPE extraction and cleanup procedure provided a convenient analysis of ginkgo flavonoids in a complex matrix (Herbal One with 16 herbal ingredients)

### Oasis HLB Extraction Method

Oasis® HLB Extraction Cartridge, 3 cc/60 mg  
 Part Number WAT094226



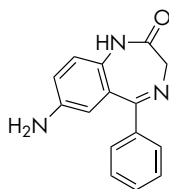
### HPLC/UV Chromatogram of Ginkgo Flavonoids in Commercial Products



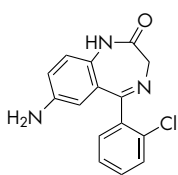
# FLUNITRAZEPAM (ROHYPNOL™) BY LC/UV

## LC Conditions

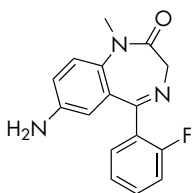
Column: SymmetryShield™ RP8, 3.9 x 150 mm, 5 µm  
 Part Number: WAT200655  
 Mobile Phase: H<sub>2</sub>O/CH<sub>3</sub>CN (70/30)  
 Flow Rate: 1.5 mL/min  
 Injection Volume: 100 µL  
 Temperature: Ambient  
 Detection: UV @ 220 nm



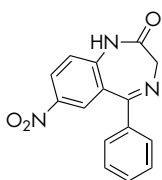
7-Amino-nitrazepam



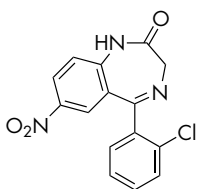
7-Amino-clonazepam



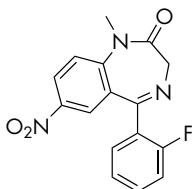
7-Amino-flunitrazepam



Nitrazepam



Clonazepam



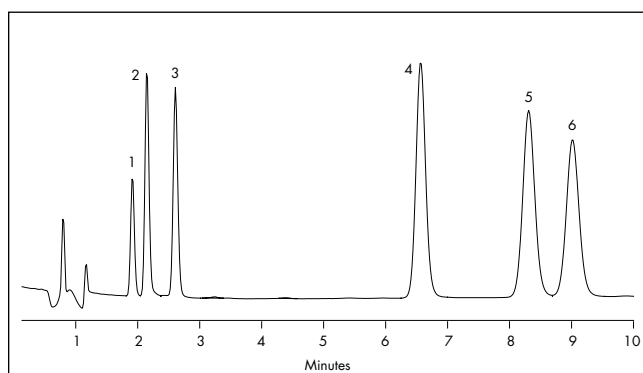
Flunitrazepam

## Oasis HLB Extraction Method

Oasis® HLB Extraction Plate, 30 mg/96 wells  
 Part Number WAT058951

CONDITION: 1 mL CH <sub>3</sub> OH
EQUILIBRATE: 1 mL H <sub>2</sub> O
LOAD: 250 µL
WASH: 1 mL CH <sub>3</sub> OH/5% CH <sub>3</sub> COOH (10/90)
ELUTE: 1 mL CH <sub>3</sub> OH/5% NH <sub>4</sub> OH (40/60)

## LC/UV Chromatogram Flunitrazepam and Related Compounds



Compounds:

- |                          |                  |
|--------------------------|------------------|
| 1. 7-Amino-nitrazepam    | 4. Nitrazepam    |
| 2. 7-Amino-clonazepam    | 5. Clonazepam    |
| 3. 7-Amino-flunitrazepam | 6. Flunitrazepam |

Compounds	Results (%)
7-Amino-nitrazepam	118
7-Amino-clonazepam	100
7-Amino-flunitrazepam	95.9
Nitrazepam	93.7
Clonazepam	101
Flunitrazepam	85.9

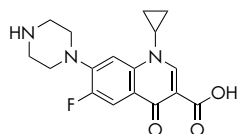
### LC Conditions

Column: Atlantis® dC18, 4.6 x 150 mm, 5 µm  
 Part Number: 1860001344  
 Mobile Phase A: 0.2% NFPA\* in H<sub>2</sub>O  
 Mobile Phase B: CH<sub>3</sub>OH  
 Gradient: Linear, 40% B to 80% B in 10 min  
 Flow Rate: 0.8 mL/min  
 Injection Volume: 50 µL  
 Temperature: 30 °C  
 Instrument: Waters Alliance® 2695 Separations Module

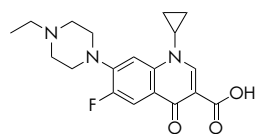
\*NFPA= nonafluoropentanoic acid - C<sub>4</sub>F<sub>9</sub>COOH

### MS Conditions

Instrument: Waters Quattro Ultima®  
 Ion Source: APCI+  
 Mode: Multiple Reaction Monitoring  
 Corona: 0.8 V  
 Source Temperature: 150 °C  
 Desolvation Temperature: 625 °C  
 Cone Gas Flow (N<sub>2</sub>): 175 L/hr  
 Desolvation Gas Flow (N<sub>2</sub>): 250 L/hr  
 Collision Gas: Argon



Ciprofloxacin



Enrofloxacin

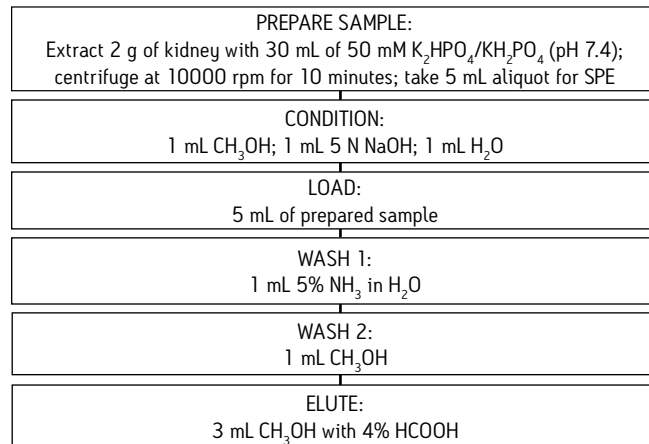
Compound	10 µg/kg Recovery (%) (± % RSD)	50 µg/kg Recovery (%) (± % RSD)
Flumequin	74 (± 9.1)	70 (± 17)
Enoxacin	63 (± 5.7)	65 (± 13)
Norfloxacin	64 (± 8.1)	65 (± 9.9)
Sarafloxacin	68 (± 9.5)	71 (± 9.8)
Ofloxacin	72 (± 7.0)	80 (± 8.6)
Enrofloxacin	73 (± 5.3)	76 (± 8.9)
Danofloxacin	64 (± 8.8)	68 (± 8.2)
Lomefloxacin	76 (± 6.9)	76 (± 7.9)
Ciprofloxacin	70 (± 8.7)	62 (± 6.1)

External standard calculation

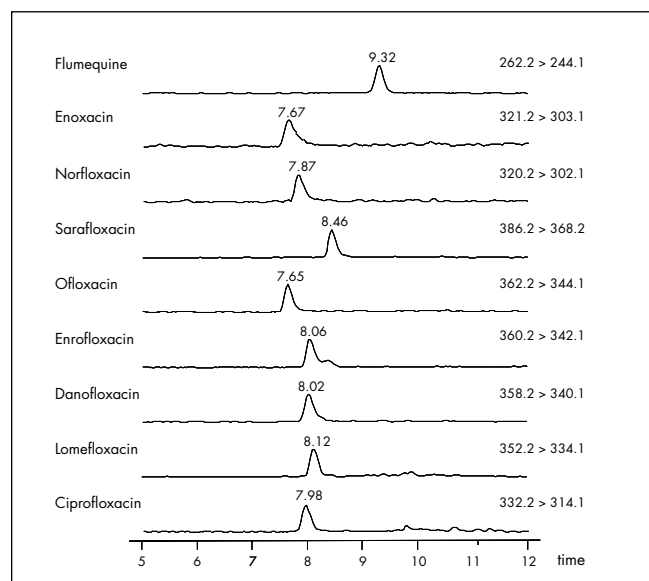
Results calculated against standards in matrix (n=5)

### Oasis MAX Extraction Method

Oasis® MAX Extraction Cartridge, 6 cc/150 mg, 30 µm  
 Part Number 186000369



### LC/MS Chromatogram of Fluoroquinolone Antibiotics in Beef Kidneys

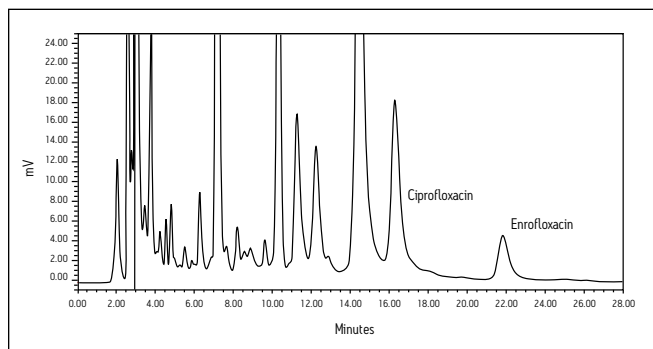


Compound	MW	MRM	Cone (V)	Coll. Energy (eV)
Flumequin	261	262 → 244	50	20
Enoxacin	320	321 → 303	50	20
Norfloxacin	319	320 → 302	50	23
Sarafloxacin	385	386 → 368	50	25
Ofloxacin	361	362 → 344	50	20
Enrofloxacin	359	360 → 342	50	20
Danofloxacin	357	358 → 340	50	25
Lomefloxacin	351	352 → 334	50	20
Ciprofloxacin	331	332 → 214	50	20

# FLUOROQUINOLONE ANTIBIOTICS IN BEEF KIDNEY USING TANDEM OASIS MAX-MCX METHOD BY LC/UV

## Comparison of Results Tandem Oasis MAX-MCX vs. Single Cartridge

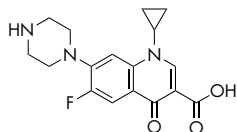
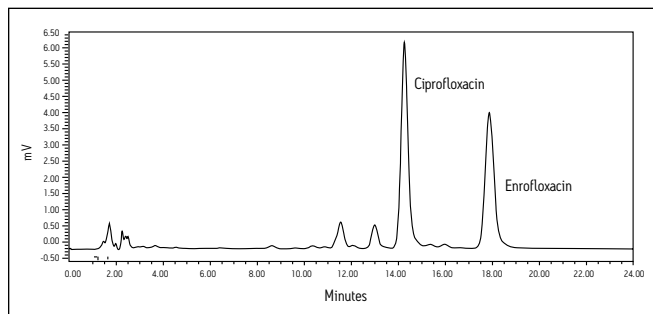
### HPLC Chromatogram of Fluoroquinolone Antibiotics in Beef Kidney by Single Cartridge Extraction



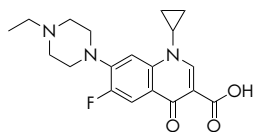
#### Mobile Phase Conditions

Column: Atlantis® dC<sub>18</sub>, 4.6 x 150, 5 µm  
 Mobile Phase: 73% 0.2% C<sub>4</sub>F<sub>9</sub>COOH  
 4% CH<sub>3</sub>OH  
 23% CH<sub>3</sub>CN

### HPLC Chromatogram of Fluoroquinolone Antibiotics in Beef Kidney by Tandem Oasis MAX/MCX Extraction



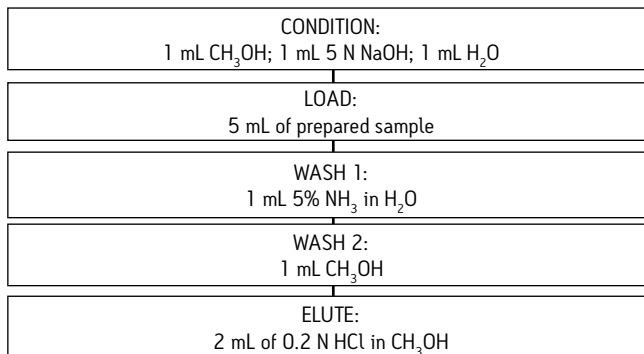
Ciprofloxacin



Enrofloxacin

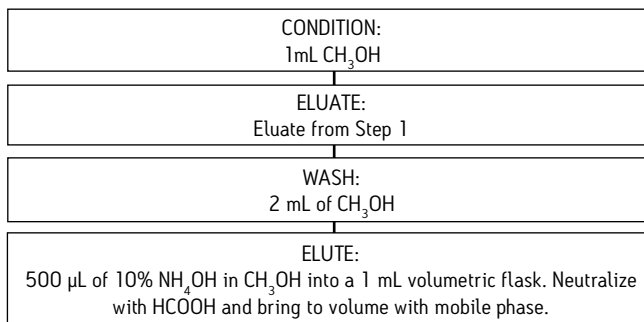
## Step 1: Oasis MAX

Oasis® MAX Extraction Cartridge, 6 cc/150 mg  
 Part Number 186000369  
 Removes basic and neutral interferences



## Step 2: Oasis MCX

Oasis MCX Extraction Cartridge, 1 cc/30 mg  
 Part Number 186000252  
 Removes acidic interferences

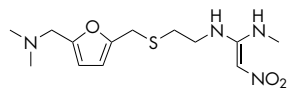


### LC Conditions

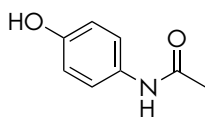
Column: SymmetryShield™ RP8, 4.6 x 75 mm, 3.5 μm  
 Guard Column: SymmetryShield RP8, 3.9 x 20 mm, 3.5 μm  
 Part Numbers: Column WAT094272, Guard 186000704  
 Mobile Phase: 5% CH<sub>3</sub>OH/95% 20 mM K<sub>2</sub>HPO<sub>4</sub>/KH<sub>2</sub>PO<sub>4</sub>, pH 7  
 Flow Rate: 2 mL/min  
 Temperature: 36 °C  
 Injection: 10 μL plasma extract  
 Detection: UV @ 214 nm (0.010 AUFS)

### Compounds:

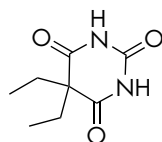
1. Acetaminophen
2. Barbital
3. *p*-Toluamide
4. Amphetamine
5. Methamphetamine
6. *m*-Toluidine
7. Ranitidine (I.S.)



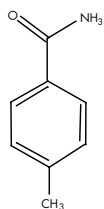
Ranitidine (I.S.)



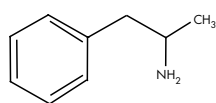
Acetaminophen



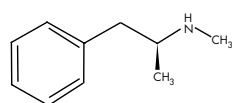
Barbital



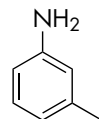
*p*-Toluamide



Amphetamine



Methamphetamine



*m*-Toluidine

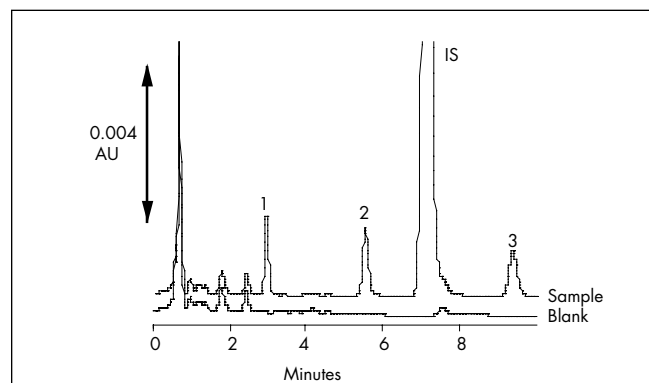
Compound	Recovery (%) n=6	RSD (%) n=6	Conc. (μg/mL)
Acetaminophen	103.4	0.55	2
Barbital	99.4	1.05	2
<i>p</i> -Toluamide	102.3	1.26	2
Amphetamine	96.8	1.35	4
Methamphetamine	90.4	4.01	4
Ranitidine	IS	-	50
<i>m</i> -Toluidine	96.4	1.32	4

### Oasis MCX Extraction Method

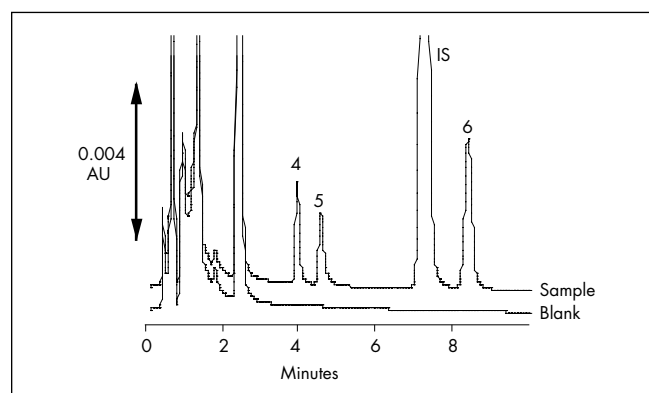
Oasis® MCX Extraction Cartridge, 1 cc/30 mg  
 Part Number 186000252

CONDITION: 1 mL CH <sub>3</sub> OH
EQUILIBRATE: 1 mL H <sub>2</sub> O
LOAD: 1 mL of spiked porcine plasma
WASH: 1 mL 2% HCOOH
ELUTE 1: 1 mL CH <sub>3</sub> OH (Eluate 1 contains acidic and neutral analytes.)
ELUTE 2: 1 mL 95% CH <sub>3</sub> OH with 5% NH <sub>4</sub> OH (Eluate 2 contains basic analytes)
ADD: 50 μL of 1 mg/mL I.S. ranitidine to Eluate 1 and Eluate 2

### HPLC Chromatogram of Eluate 1 Fraction



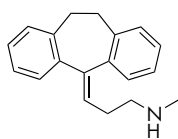
### HPLC Chromatogram of Eluate 2 Fraction



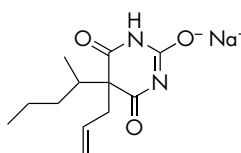
# FRACTIONATION OF ACIDIC FROM BASE AND NEUTRAL DRUGS IN URINE BY LC/UV

## LC Conditions

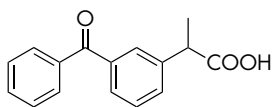
Column: SymmetryShield™, RP18 4.6 x 100 mm, 3.5 µm  
 Guard Column: Sentry™, 3.9 x 20 mm, 5 µm  
 Part Numbers: Column- 186000179, Guard- 186000701  
 Mobile Phase: 20 mM KH<sub>2</sub>PO<sub>4</sub>/H<sub>3</sub>PO<sub>4</sub>, pH 2.7/ CH<sub>3</sub>OH, 52/48 v/v  
 Flow Rate: 2 mL/min  
 Injection Volume: 10 µL of extract with internal standard  
 Temperature: 30 °C  
 Detection: UV @ 214 nm



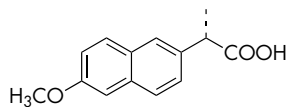
Nortriptyline



Secobarbital



Ketoprofen

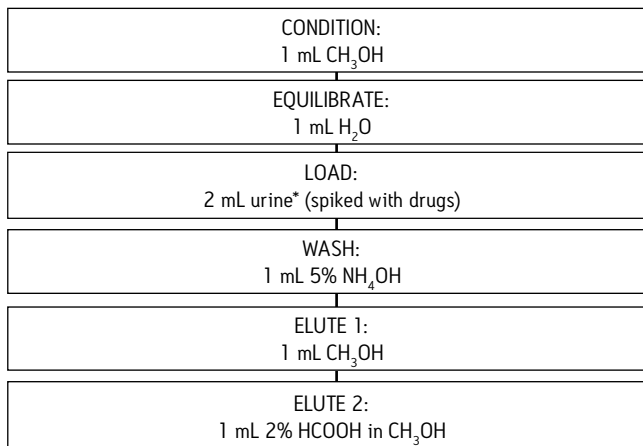


Naproxen

Drugs	Spike Level (µg/ mL)	Type of Drug	Recovery (%)	RSD (n=6)
Nortriptyline	5	Basic Elute 1	94.34	0.78
Secobarbital	10	Neutral Elute 1	88.53	1.03
Ketoprofen	5	Acidic Elute 2	91.64	2.35
Naproxen	2.5	Acidic Elute 2	103.43	3.04

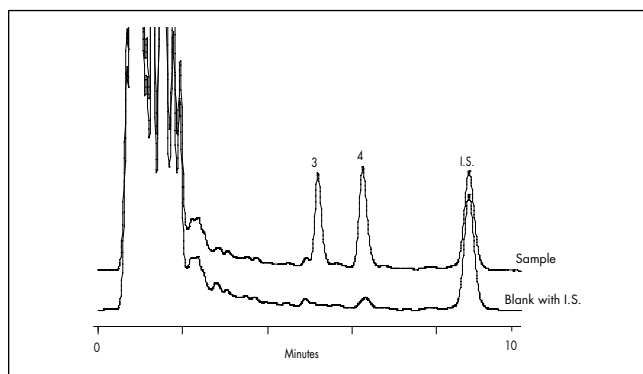
## Oasis MAX Extraction Method

Oasis® MAX Extraction Cartridge, 1 cc/30 mg  
 Part Number 186000366

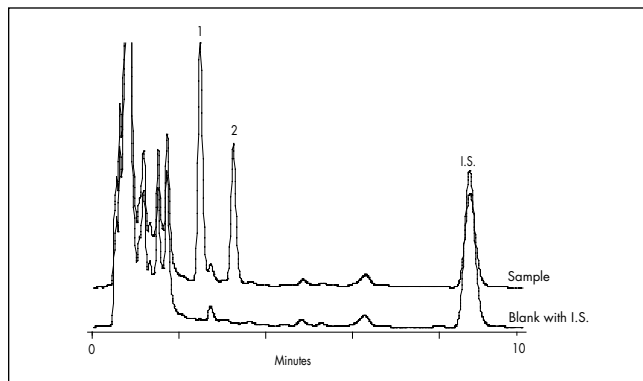


\* 1 mL urine hydrolyzed (1 M KOH 60 °C, 15 minutes) and adjusted to pH 2 with phosphoric acid; diluted 1/1 with 10 mM CH<sub>3</sub>COONa, pH 2.

## HPLC Chromatogram of Elute 2 (Acidic Drugs)

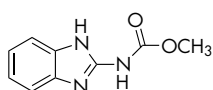


## HPLC Chromatogram of Elute 1 (Basic and Neutral Drugs)

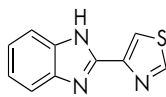


**LC Conditions**

Column: XTerra® RP18, 4.6 x 100 mm, 3.5 µm  
 Part Number: 186000438  
 Mobile Phase: 72.5% KH<sub>2</sub>PO<sub>4</sub>/K<sub>2</sub>HPO<sub>4</sub> (20 mM pH 6.8)  
 27.5% CH<sub>3</sub>CN  
 Flow Rate: 1 mL/min  
 Injection Volume: 20 µL  
 Detection: PDA (288 nm)

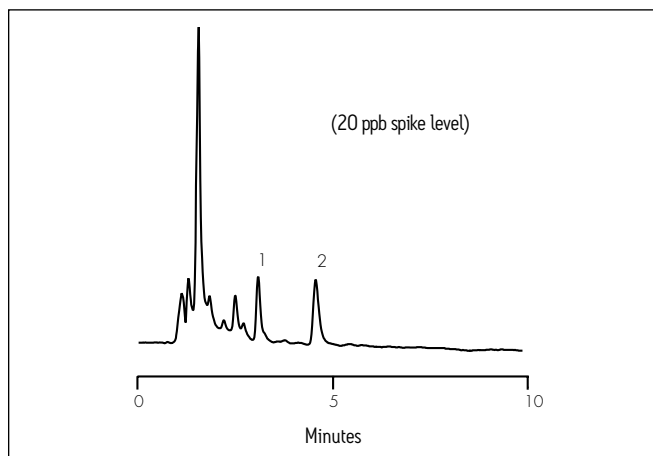


Carbendazim



Thiabendazole

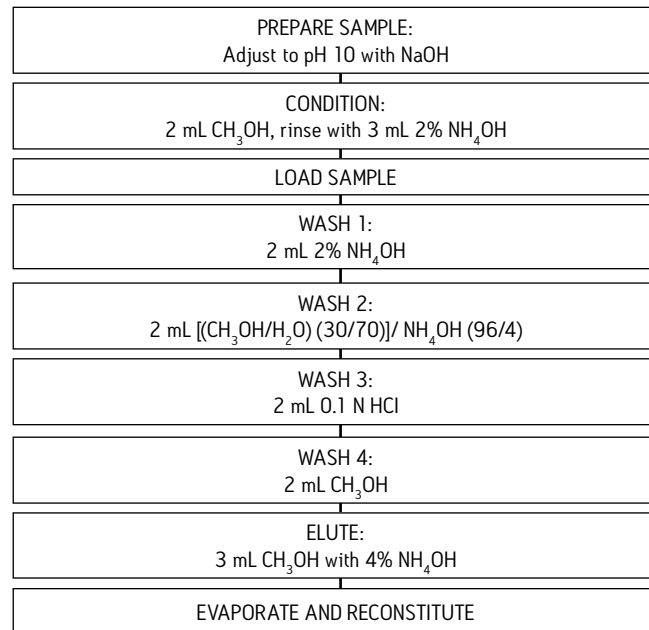
**LC/PDA Chromatogram of Fungicides in Apple Juice**



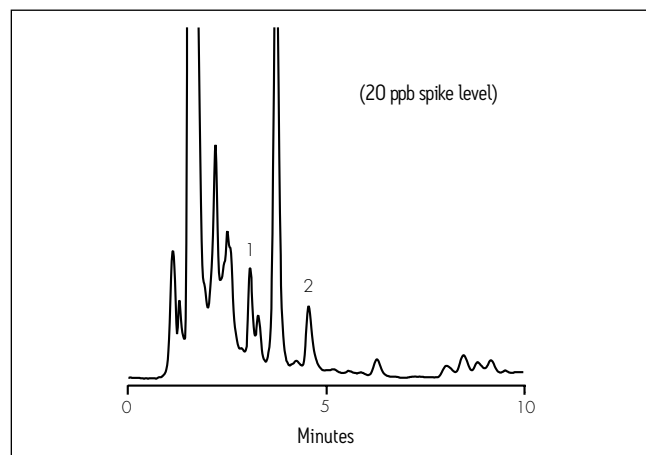
Compounds	% Recovery (% RSD), n=4
1. Carbendazim	82 (2)
2. Thiabendazole	96 (2)

**Oasis MCX Extraction Method**

Oasis® MCX Extraction Cartridge, 6 cc/150 mg  
 Part Number 186000256



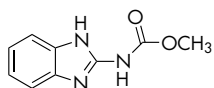
**LC/PDA Chromatogram of Fungicides in Grape Juice**



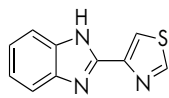
Compounds	% Recovery (% RSD), n=4
1. Carbendazim	81 (8)
2. Thiabendazole	94 (10)

## LC Conditions

Column: XTerra® RP18, 4.6 x 100 mm, 3.5 µm  
 Part Number: 186000438  
 Mobile Phase: 72.5% KH<sub>2</sub>PO<sub>4</sub>/K<sub>2</sub>HPO<sub>4</sub> (20 mM pH 6.8)  
 27.5% CH<sub>3</sub>CN  
 Flow Rate: 1 mL/min  
 Injection volume: 20 µL  
 Detection: PDA (288 nm)

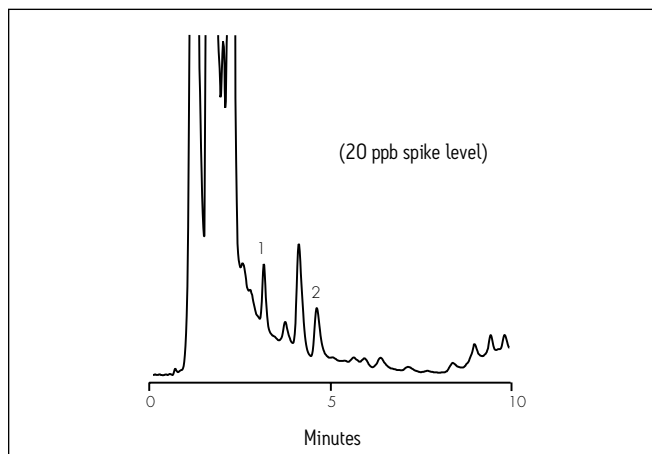


Carbendazim



Thiabendazole

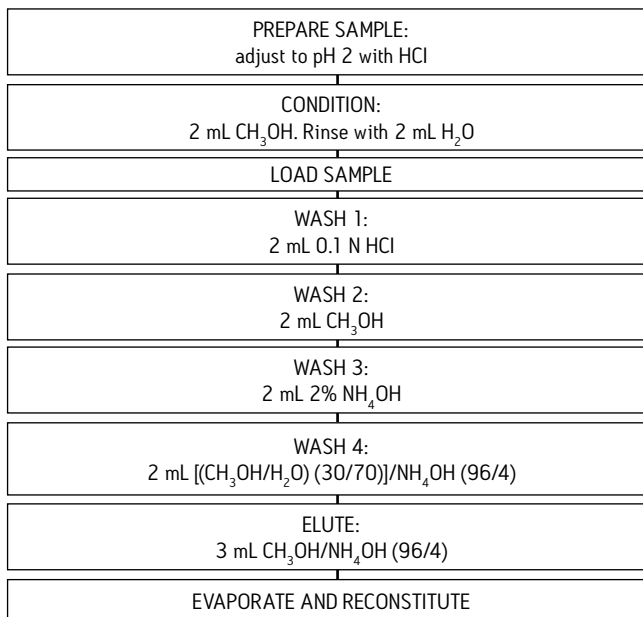
## LC/PDA Chromatogram of Fungicides in Orange Juice



Compounds	% Recovery (RSD %), n=4
1. Carbendazim	91 (2)
2. Thiabendazole	94 (3)

## Oasis MCX Extraction Method

Oasis® MCX Extraction Cartridge, 6 cc/150 mg  
 Part Number 186000256





### LC Conditions

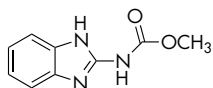
Column: XTerra® MS C<sub>18</sub>, 2.1 x 100 mm, 5 µm  
 Part Number: 186000450  
 Mobile Phase: CH<sub>3</sub>CN/10 mM NH<sub>4</sub>HCO<sub>3</sub>, pH 8.3 (20/80)  
 Flow Rate: 200 µL/min, split 1/1 through each detector  
 Instrument: Waters Alliance® Separations Module with 996 PDA

### MS Conditions

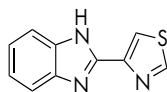
Instrument: Waters ZQ™  
 Source: Positive Electrospray (ESI+)  
 Mode: Multiple Selected-Ion Recording (SIR)

### Compounds

1. Carbendazim 70 µg/L
2. Thiabendazole 170 µg/L



Carbendazim

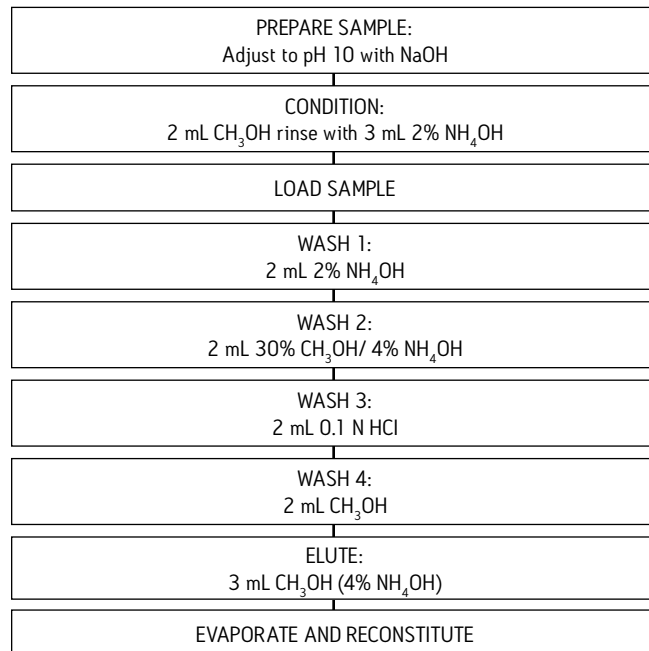


Thiabendazole

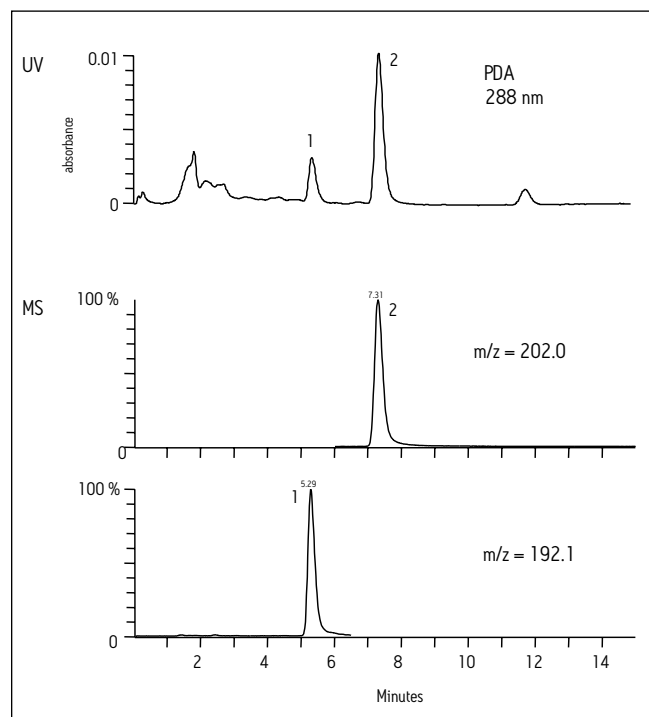
SIR Group	Time (mins)	Compound	Mass	Cone Voltage (V)
1	0-6.5	Carbendazim	192.1	25
2	6-15	Thiabendazole	202.0	35

### Oasis MCX Extraction Method

Oasis® MCX Extraction Cartridge, 6 cc/150 mg  
 Part Number 186000256



### LC/MS and LC/PDA Chromatograms of Incurred Fungicides in Apple Cider



# INVESTIGATION OF THE BIODEGRADATION OF NAPHTHOIC ACIDS IN GROUND WATER BY LC/MS/MS

## LC Conditions

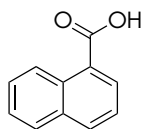
Column: XTerra® MS C<sub>18</sub>, 2.1 x 100 mm  
 Mobile Phase A: 15 mM HCOONH<sub>4</sub> in H<sub>2</sub>O  
 Mobile Phase B: CH<sub>3</sub>CN  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	75	25
9	40	60
14	40	60
16	10	90

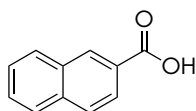
Flow Rate: 200 µL/min  
 Injection Volume: 20 µL  
 Instrument: Waters Alliance® 2695 Separations Module

## MS Conditions

Instrument: Waters Quattro Ultima®  
 Ion Source: Electrospray Negative (ESI<sup>-</sup>)  
 Mode: Multiple Reaction Monitoring (MRM)  
 Source Temperature: 150 °C  
 Desolvation Temperature: 450 °C  
 Cone Gas Flow: 50 L/hr  
 Desolvation Gas Flow: 500 L/hr  
 Collision Gas: Argon



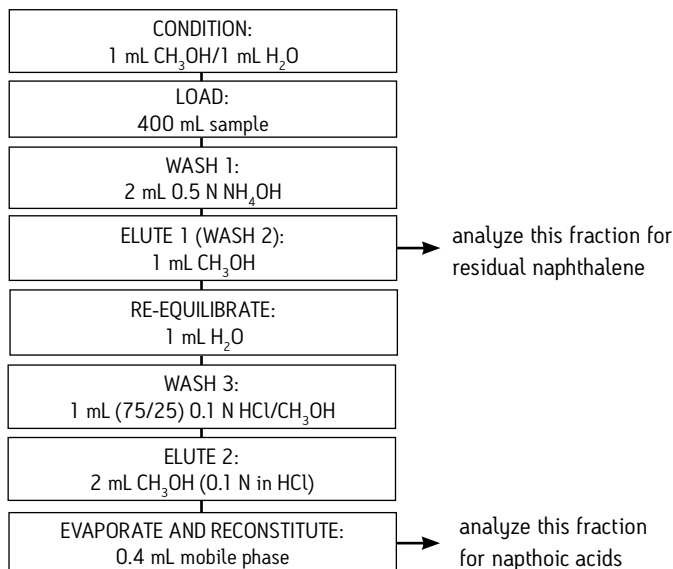
1-Naphthoic Acid



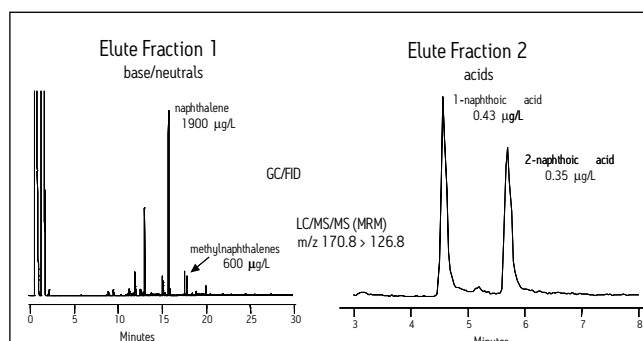
2-Naphthoic Acid

## Oasis MAX Extraction Method

Oasis® MAX Extraction Cartridge, 6 cc/150 mg, 30 µm  
 Part Number 196000369



## LC/MS Chromatogram of Naphthoic Acids and Naphthalenes in Contaminated Ground Water



### Matrix Spike Recoveries (from site H<sub>2</sub>O blank)

1. Naphthoic Acid: 69% (spike level 0.5 µg/L)
2. Naphthoic Acid: 75% (spike level 0.5 µg/L)
3. Naphthalene: 85% (spike level 10 µg/L)

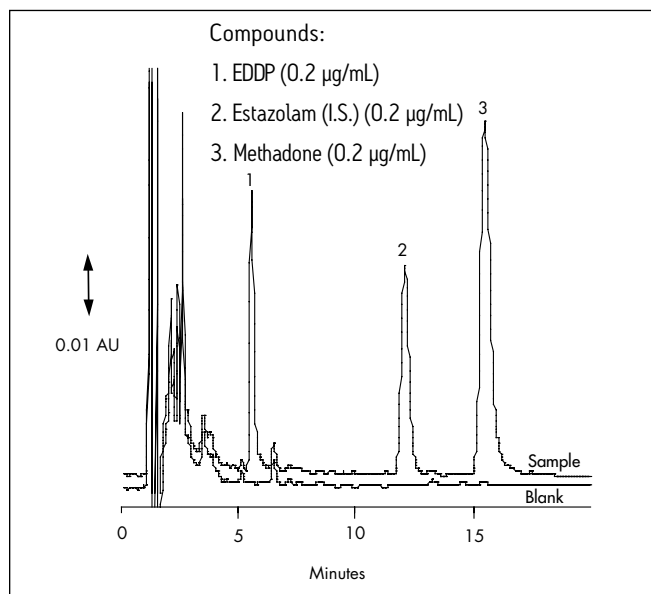
### LC/UV Conditions

Column: Symmetry® C<sub>18</sub>, 3.9 x 150 mm, 5 µm  
 Guard Column: Symmetry C<sub>18</sub>, 3.9 x 20 mm, 5 µm  
 Part Numbers: Column - WAT046980, Guard - WAT054225  
 Mobile Phase: 0.1% CF<sub>3</sub>COOH in H<sub>2</sub>O/ CH<sub>3</sub>OH (60/40)  
 Flow Rate: 1 mL/min  
 Injection Volume: 100 µL urine extract  
 Temperature: 30 °C  
 Detection: UV@ 210 nm

### LC/MS Conditions

Column: Symmetry C<sub>18</sub>, 2.1 x 100 mm, 3.5 µm  
 Part Number: WAT058965  
 Flow Rate: 200 µL/min  
 Mobile Phase: 2 mM CH<sub>3</sub>COONH<sub>4</sub>, 65% aqueous  
 CH<sub>3</sub>CN, 0.1% HCOOH  
 Injection Volume: 10 µL  
 MS Instrument: Waters Quattro Ultima®  
 Ion Source: Electrospray Positive (ESI+)  
 Cone Voltage: 25 V  
 Collision Energy: 15 eV

### LC/UV Chromatogram of Methadone and Metabolite in Human Urine

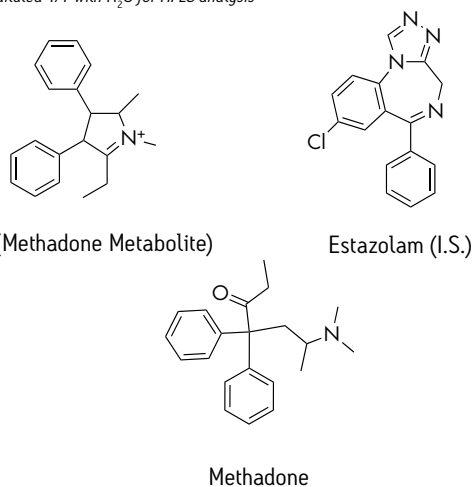


### Oasis MCX Extraction Method

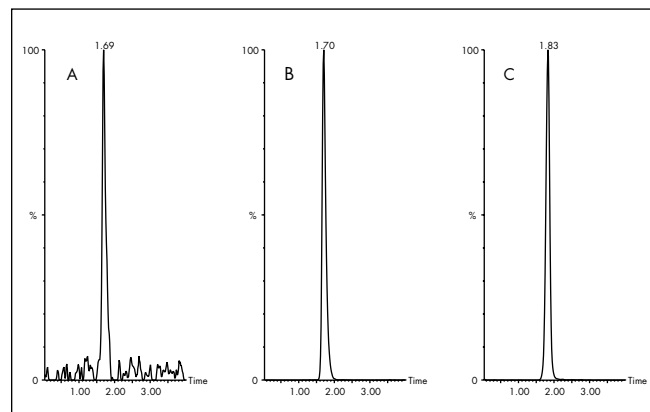
Oasis® MCX Extraction Cartridge, 3 cc/60 mg  
 Part Number 186000254

NO CONDITIONING
LOAD: 3 mL acidified spiked human urine
WASH 1: 2 mL 0.1M HCl
WASH 2: 2 mL CH <sub>3</sub> OH
OPTIONAL WASH 3: 2 mL of 5% NH <sub>4</sub> OH in CH <sub>3</sub> OH/ H <sub>2</sub> O (60/40)
ELUTE: 2 mL 5% NH <sub>4</sub> OH in CH <sub>3</sub> OH/ H <sub>2</sub> O (95/5)

Each eluate diluted 1/1 with H<sub>2</sub>O for HPLC analysis



### LC/MS Chromatogram of Methadone and Metabolite in Human Urine



MRM Chromatograms under optimum conditions of pure methadone standard at (A) 0.05 ng/mL (LOD) and (B) 5.0 ng/mL and (c) a processed human plasma sample with a high concentration of methadone.

**LC Conditions**

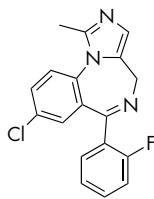
Column: SunFire™ C<sub>18</sub>, 2.1 x 20 mm /S™, 3.5 μm  
 Mobile Phase A: 10 mM CH<sub>3</sub>COONH<sub>4</sub>, pH 5.5  
 Mobile Phase B: CH<sub>3</sub>OH with 10 mM CH<sub>3</sub>COONH<sub>4</sub>, pH 5.5  
 Flow Rate: 0.4 mL/min  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	95	5
3.0	5	95
4.0	5	95
4.1	95	5
5.0	95	5

Instrument: 2777 Sample Manager and 1525μ Binary HPLC Pump

**MS Conditions**

Instrument: Waters Quattro Premier™  
 Ion Source: Electrospray Positive (ESI+)  
 Source Temperature: 150 °C  
 Desolvation Temperature: 350 °C  
 Cone Gas Flow: 50 L/hr  
 Desolvation Gas Flow: 600 L/hr  
 Collision Cell Pressure: 2.2e-3 bar (Argon)

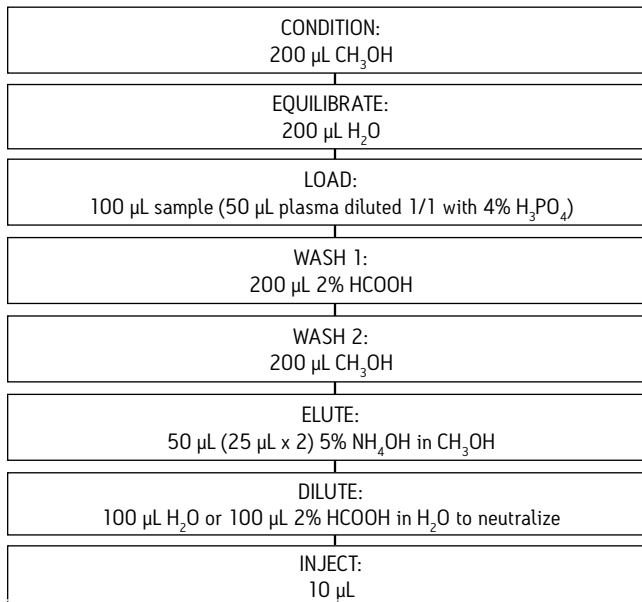


Midazolam

Compounds	MRM Transition (m/z)	Cone (V)	Collision Energy (eV)
Midazolam	326.2 → 291.2	40	28

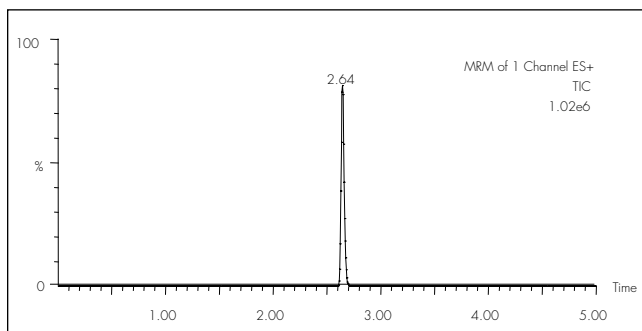
**Oasis MCX Extraction Method**

Oasis® MCX 96-well μElution Plate  
 Part Number 186001830BA



**SPE Recovery: 97%**

**LC/MS Chromatogram of Midazolam in Plasma**



### LC Conditions

Column: XTerra® MS C<sub>18</sub>, 2.1 x 30 mm, 3.5 µm  
 Part Number: 186000398  
 Mobile Phase A: CH<sub>3</sub>CN + 0.1 M NH<sub>4</sub>HCO<sub>3</sub>, pH 9.7  
 Mobile Phase B: H<sub>2</sub>O + 0.1 M NH<sub>4</sub>HCO<sub>3</sub>, pH 9.7  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	95	5
1	5	95

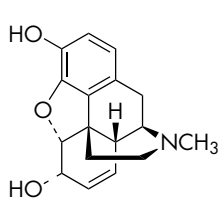
Flow Rate: 0.2 mL/min  
 Temperature: Ambient  
 LC Instrument: Waters Alliance® 2795

### MS Conditions

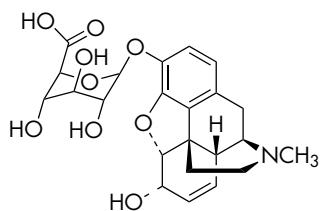
MS Instrument: Waters Quattro Ultima®  
 Ion Source: Electrospray Positive (ESI+)  
 Source Temperature: 150 °C  
 Collision Cell Pressure: 2.0 e-3 bar Argon  
 Desolvation Temperature: 350 °C  
 Capillary Voltage: 3.5 KV  
 Drying Gas Flow: 500 L/hr  
 Cone Gas Flow: 50 L/hr  
 Cone Voltage: 35 V

### MRM Transitions

Compound	MRM Transition (m/z)	Collision Energy
Morphine:	285.8	152.8 (40)
Morphine-3-GLU:	462.0	286.0 (25)
Morphine-d <sub>3</sub> (I.S.):	288.9	152.8 (40)



Morphine



Morphine-3β-D-glucuronide

### Oasis HLB Extraction Method

Oasis® HLB 96-well µElution Plate  
 Part Number 186001828BA

CONDITION: 200 µL CH <sub>3</sub> OH
EQUILIBRATE: 200 µL H <sub>2</sub> O
LOAD: 35 µL rat plasma spiked with 35 µL I.S. (25 pg/µL) in H <sub>2</sub> O
WASH: 200 µL CH <sub>3</sub> OH/H <sub>2</sub> O (5/95)
ELUTE: 25 µL [CH <sub>3</sub> CN/ (CH <sub>3</sub> ) <sub>2</sub> CHOH 40/60]/ HCOOH (98/2)
EVAPORATE AND RECONSTITUTE: 75 µL H <sub>2</sub> O
INJECT: 40 µL

### Oasis MCX Generic Extraction Method

Oasis MCX 96-well µElution Plate  
 Part Number 186001830

CONDITION: 200 mL CH <sub>3</sub> OH
EQUILIBRATE: 200 µL H <sub>2</sub> O
LOAD: 35 µL rat plasma spiked with 35 µL I.S. (25 pg/mL) in H <sub>2</sub> O
WASH 1: 200 µL H <sub>2</sub> O + 0.1 N HCL
WASH 2: 200 µL CH <sub>3</sub> OH
ELUTE: 25 µL [CH <sub>3</sub> CN/ (CH <sub>3</sub> ) <sub>2</sub> CHOH (40/60)]/NH <sub>4</sub> OH (95/5)
EVAPORATE
RECONSTITUTE: 75 µL H <sub>2</sub> O
INJECT: 40 µL

# MORPHINE AND ITS GLUCURONIDE METABOLITE IN RAT PLASMA BY LC/MS/MS

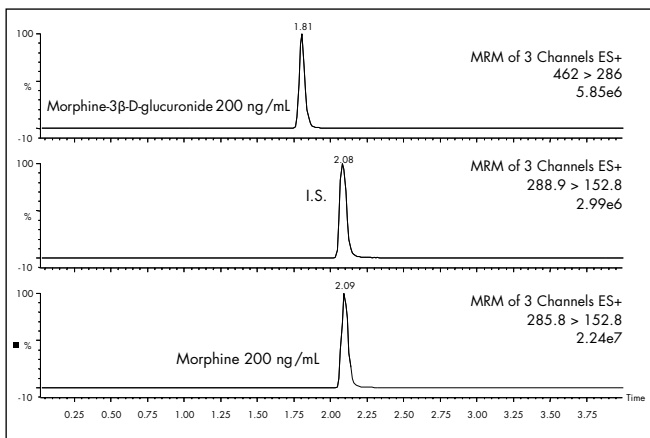
## Oasis MCX

Morphine		Morphine-Glucuronide	
Nominal conc. (ng/mL)	Calculated conc.	Nominal conc. (ng/mL)	Calculated conc.
0.2	0.20	0.2	0.20
0.5	0.51	0.5	0.51
1	1.03	1	1.02
2	1.91	2	2.01
5	4.99	5	na
10	9.59	10	9.73
20	20.87	20	19.84
50	49.46	50	48.37
100	102.23	100	103.35
200	198.00	200	198.66

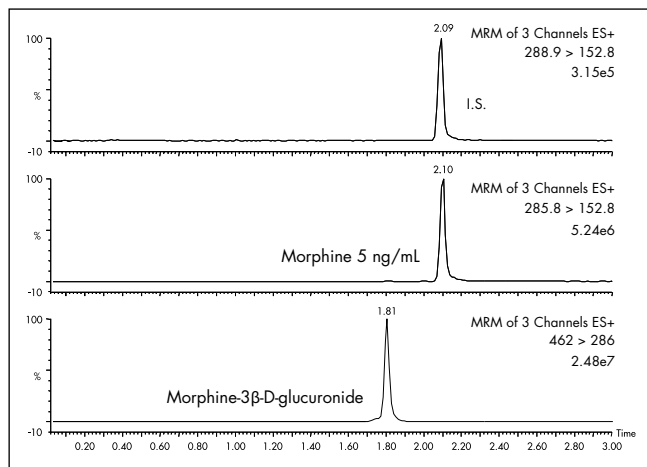
## Oasis HLB

Morphine		Morphine-Glucuronide	
Nominal conc. (ng/mL)	Calculated conc.	Nominal conc. (ng/mL)	Calculated conc.
1	1.00	5	5.09
2	2.02	10	9.42
5	4.97	20	na
10	9.93	25	26.64
20	20.05	50	48.73
25	25.05	100	103.79
50	50.49	150	141.30
100	99.07	200	203.43
200	199.84	250	na
500	500.51	500	501.26

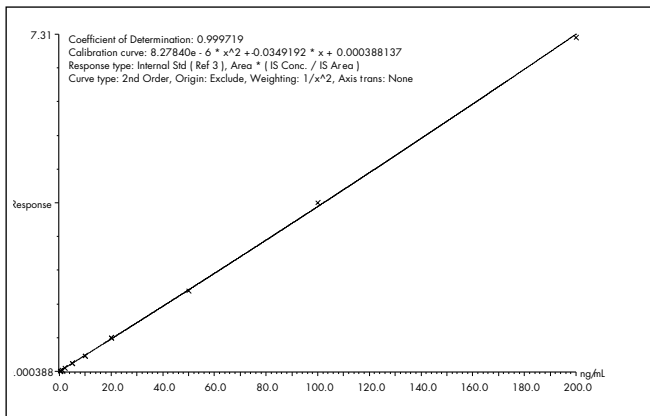
## LC/MS Chromatogram of Morphine and its Glucuronide Metabolite in Rat Plasma, Oasis MCX



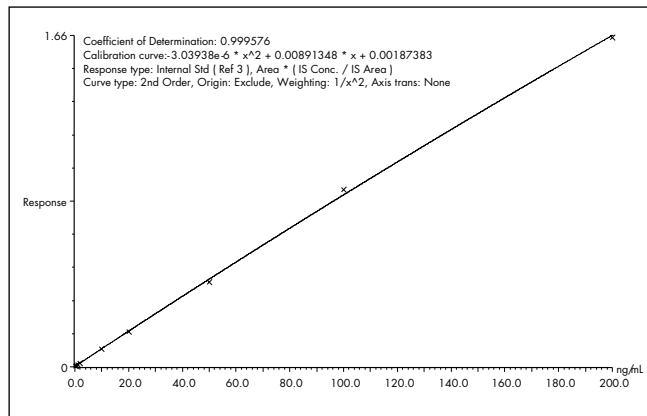
## LC/MS Chromatogram of Morphine and its Glucuronide Metabolite in Rat Plasma, Oasis HLB



## Oasis MCX Morphine Calibration Curve



## Oasis MCX Morphine-3β-D-glucuronide Calibration Curve



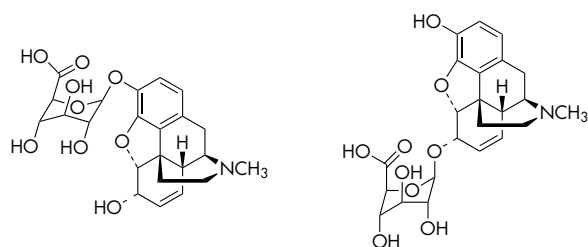
# MORPHINE AND ITS GLUCURONIDE METABOLITES IN SERUM BY LC FLUORESCENCE

## LC Conditions

Column: SymmetryShield™ RP8, 3.9 x 150 mm, 5 µm  
 Part Number: WAT200655  
 Mobile Phase: 20 mM KH<sub>2</sub>PO<sub>4</sub>/K<sub>2</sub>HPO<sub>4</sub>, pH 6.4  
 Flow Rate: 1 mL/min  
 Injection Volume: 100 µL of porcine serum extract  
 Detection: Fluorescence; ex 280 nm, em 355 nm

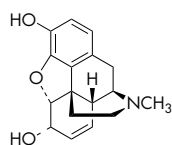
## Compounds:

- 1: Morphine-3β-D-glucuronide
- 2: Morphine-6β-D-glucuronide
- 3: Morphine



Morphine-3β-D-glucuronide

Morphine-6β-D-glucuronide

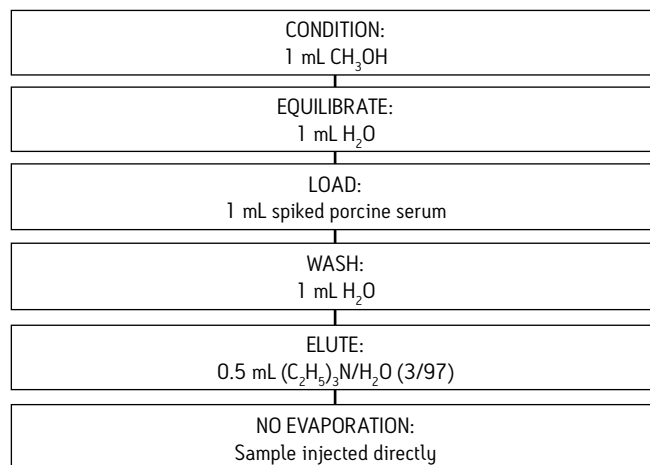


Morphine

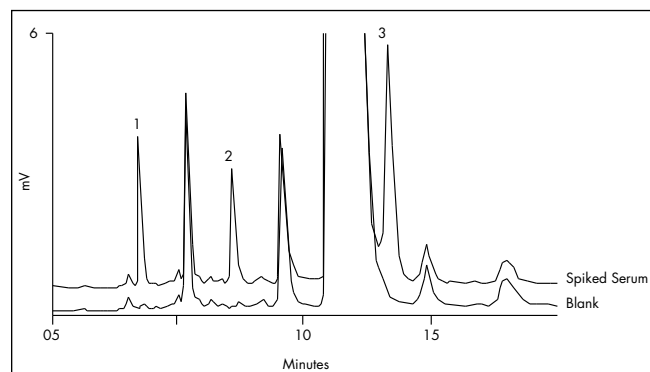
Compound	Concentration (µg/mL)	Recovery (%)	RSD (%) n=6
Morphine-3β-D-glucuronide	0.48	90.7	2.0
	0.097	100	3.1
Morphine-6β-D-glucuronide	2.4	92.2	2.8
	0.49	93.1	2.5
Morphine	3.6	102	3.6
	0.73	102	3.2

## Oasis HLB Extraction Method

Oasis® HLB Extraction Cartridge, 1 cc/30 mg  
 Part Number WAT094225



## LC/MS Chromatogram of Morphine and its Glucuronide Metabolites in Porcine Serum



# NAPTALAM IN CUCUMBER BY LC/MS AND LC/UV

## LC Conditions

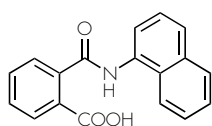
Column: XTerra® RP18, 4.6 x 100 mm, 3.5 µm  
 Part Number: 186000438  
 Mobile Phase A: 10 mM CH<sub>3</sub>COONH<sub>4</sub>, pH 5.5  
 Mobile Phase B: CH<sub>3</sub>CN  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	80	20
20	20	80

Flow Rate: 1 mL/min  
 Injection Volume: 50 µL  
 Detection: UV @ 290 nm  
 Instrument: Waters Alliance® 2695, 2996 PDA

## LC/MS Conditions

Column: XTerra MS C<sub>18</sub>, 2.1 x 100 mm  
 Mobile Phase: 25% CH<sub>3</sub>CN/ 75% 10 mM CH<sub>3</sub>COONH<sub>4</sub>, pH 5.5 to 90% CH<sub>3</sub>CN in 6 min  
 Flow Rate: 200 µL/min  
 Injection Volume: 20 µL  
 Instrument: Waters Alliance Separations Module  
 Instrument: Waters ZMD  
 Ion Source: Positive Electrospray (ESI+)  
 Mode: Multiple Selected-Ion Recording (SIR)



Naptalam

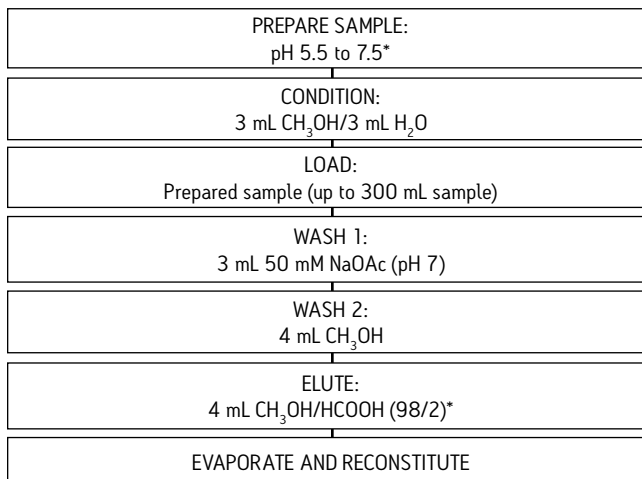
Spike Level	% Results* (% RSD), n=4
10 ppb	73 (6)
50 ppb	76 (8)

SIR Group	Time (min)	Compound	Mass	Cone Voltage (V)	Dwell Time (sec)
1	5 - 8	Naptalam	144,292,293	17	0.8

\* recovery measured against standards prepared in cucumber matrix

## Oasis MAX Extraction Method

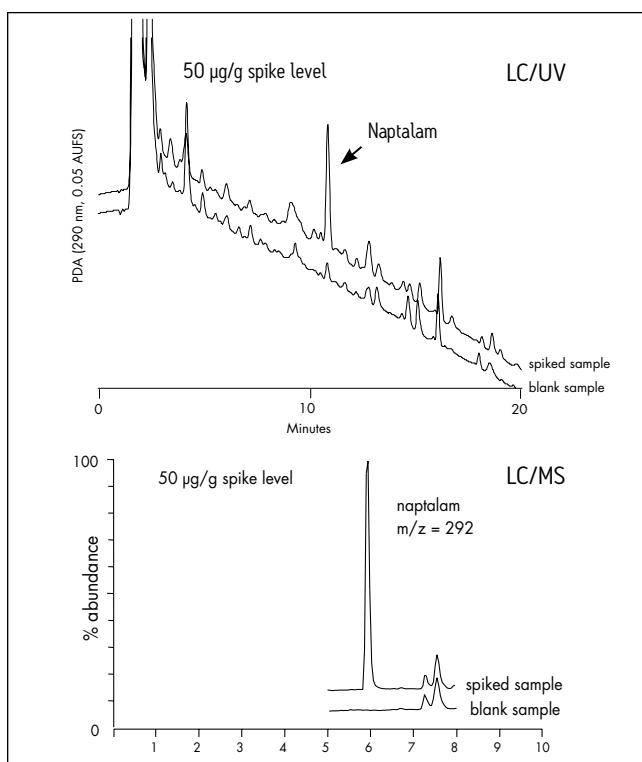
Oasis® MAX Extraction Cartridge, 6 cc/500 mg  
 Part Number 186000865



### \*Sample Preparation

- Prepare 8 g sample (homogenize)
- Extract with 30 mL CH<sub>3</sub>CN (shake 30 minutes)
- Centrifuge (10 minute @ 8000 x g)
- Transfer supernatant to reservoir
- Wash pellet with 20 mL H<sub>2</sub>O
- Transfer wash to reservoir (combine with supernatant)
- Perform SPE

## LC/ MS and LC/UV Chromatograms of Naptalam in Cucumber



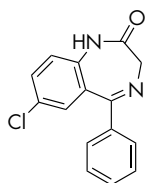


**LC Conditions**

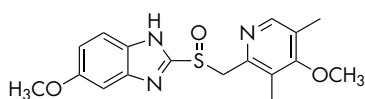
Column: XTerra® MS C<sub>18</sub> 2.1 x 30 mm, 3.5 µm  
 Part Number: 186000398  
 Mobile Phase A: 1% NH<sub>4</sub>OH in H<sub>2</sub>O  
 Mobile Phase B: CH<sub>3</sub>CN  
 Isocratic Mobile  
 Phase Composition: 50% A; 50% B  
 Flow Rate: 0.2 mL/min  
 Injection Volume: 20 µL  
 Instrument: Waters Alliance® 2690

**MS Conditions**

Instrument: Waters Quattro Ultima®  
 Ion Source: Electrospray Positive (ESI+)  
 Source Temperature: 150 °C  
 Collision Cell Pressure: 1.5e-3 mbar  
 Collision Energy: 10 eV  
 Desolvation Temperature: 350 °C  
 Cone Gas Flow: 150 L/hr  
 Drying Gas Flow: 600 L/hr  
 Cone Voltage: 30 V



Nordiazepam (I.S.)

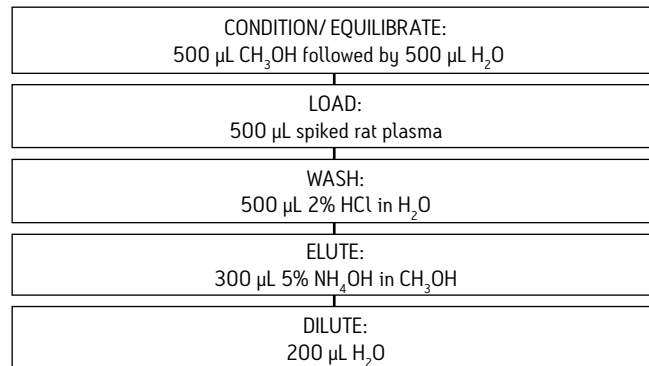


Omeprazole

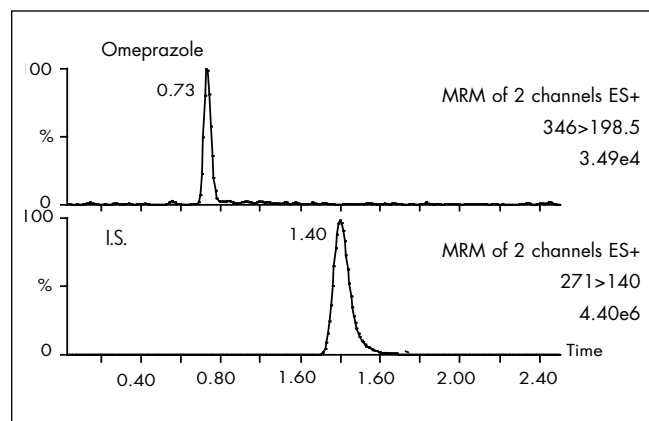
Omeprazole (ng/mL)	Mean	Standard Deviation	Coefficient of Variation (%)	Recovery (%)
1	0.95	0.06	6.7	95
2.5	2.57	0.19	7.7	102
10	9.58	0.47	4.9	95
20	20.16	0.89	4.4	100
50	54.04	2.2	4.2	108
100	102.6	6.3	6.3	102
250	248.4	3.3	3.3	99

**Oasis MCX Extraction Method**

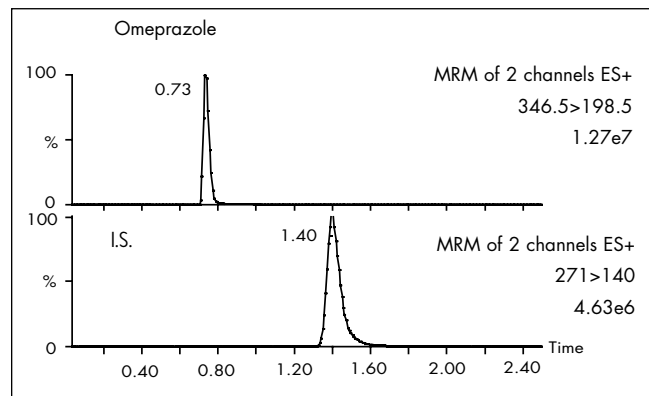
Oasis® MCX Extraction Plate, 10 mg/96 wells  
 Part Number 186000259



**LC/MS Chromatogram of Omeprazole in Rat Plasma, 1 ng/mL Spiked Level**



**LC/MS Chromatogram of Omeprazole in Rat Plasma, 250 ng/mL Spiked Level**



### LC Conditions

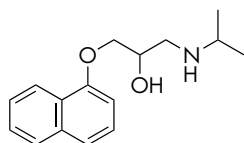
Column: XTerra® MS C<sub>18</sub>, 2.1 x 30 mm, 3.5 µm  
 Part Number: 186000398  
 Mobile Phase A: 0.5% HCOOH in CH<sub>3</sub>CN  
 Mobile Phase B: 0.5% HCOOH in H<sub>2</sub>O  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	5	95
0.5	5	95
1.5	95	5
4.40	95	5
4.50	5	95
6.0	5	95

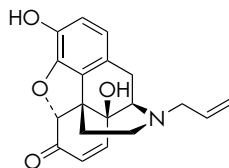
Flow Rate: 1 mL/min  
 LC<sub>1</sub>: Waters Alliance® 2690 - 0.4 mL/min  
 LC<sub>2</sub>: Waters 515 - 5.0 mL/min  
 Loading Mobile Phase: 100% H<sub>2</sub>O  
 Eluting Mobile Phase: 1 min gradient 5% CH<sub>3</sub>CN to 95% CH<sub>3</sub>CN  
 Eluting Mobile  
 Phase Additive: 0.5% HCOOH  
 Extraction Column  
 Temperature: 40 °C  
 Switching Valve: Waters Selector Valve

### MS Conditions

Instrument: Waters Quattro Ultima®  
 Ion Source: Electrospray Positive (ESI+)  
 Source Temperature: 150 °C  
 Desolvation Gas Flow: 600 L/hr  
 Collision Cell Pressure 1.5e-3 mbar  
 Cone Voltage: 20 V  
 Collision Energy: 20 eV



Propranolol



Naloxone

### Cell Lysate Preparation

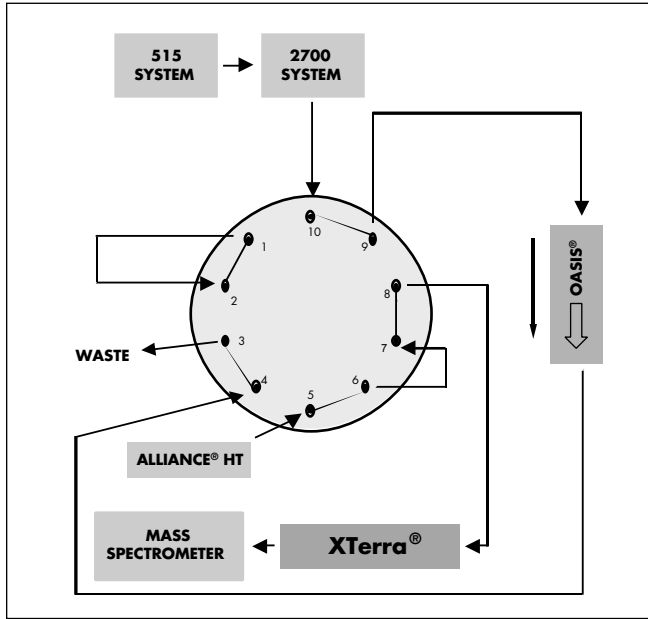
Oasis® HLB Extraction Column, 2.1 x 20 mm, 25 µm  
 Part Number 186000906

CULTURE: 10 <sup>6</sup> Jurkat cells were grown in RPMI 1640 culture medium
SPIN: Cells were centrifuged at 1500 g for 5 minute
WASH: Cells were washed twice with cold PBS
PELLETIZE: Cells were centrifuged at 1500 g and supernatant was removed. Cell pellets were spiked at various level
LYSE: Lyse with 1 mL 50/50 CH <sub>3</sub> OH/CH <sub>3</sub> CN
DILUTE: with 4 mL H <sub>2</sub> O
INJECT: 400 µL

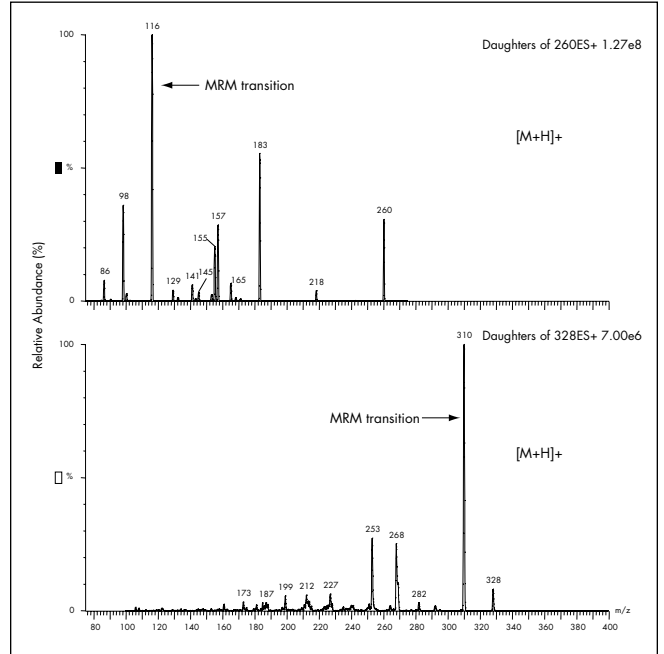
Conc. (ng/mL)	Average (n=6)	Standard Deviation	RSD (%)
Propranolol Calibration Curve			
0.1	0.09	0.05	4.8
0.5	0.49	0.03	5.9
1	1.03	0.04	3.9
5	4.93	0.26	5.3
10	9.76	0.34	3.5
20	20.21	0.89	4.4
25	25.21	0.69	2.7
Naloxone Calibration Curve			
0.1	0.10	0.04	4.2
0.5	0.48	0.02	5.0
1	0.99	0.06	6.8
5	5.25	0.25	4.8
10	10.43	0.41	3.9
20	19.98	0.54	2.7
25	24.44	1.12	4.6

Reference: Mallet, C.R.; Mazzeo, J.; Neue, U. *Rapid Commun. Mass Spectrom.* **15**:1075-1083. (2001)

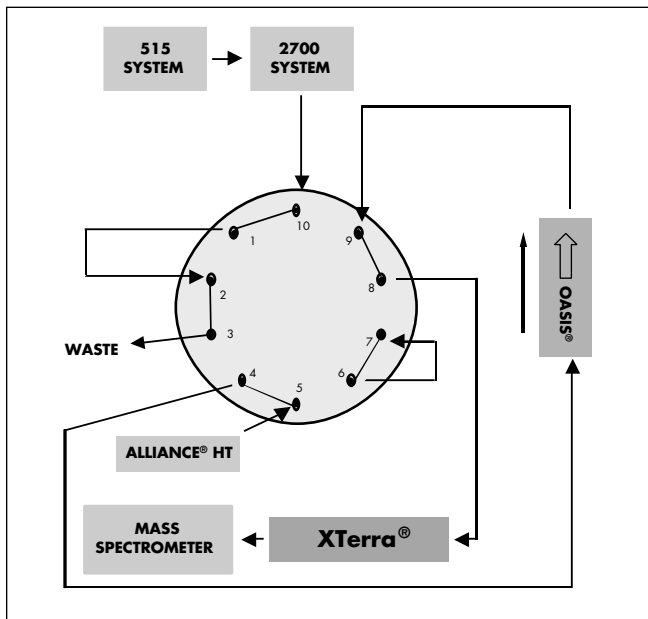
Load Position (Backflush Configuration)



CID Mass Spectra

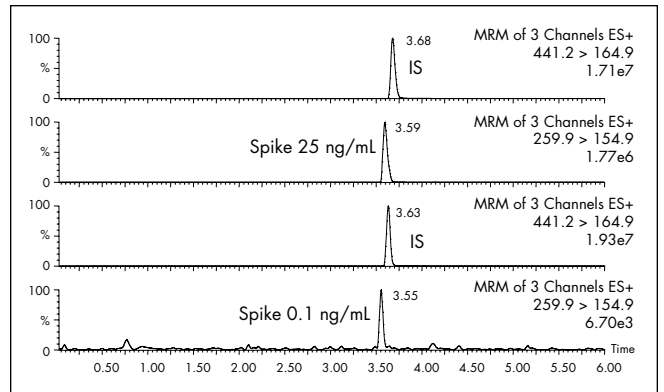


Elution Position (Backflush Configuration)

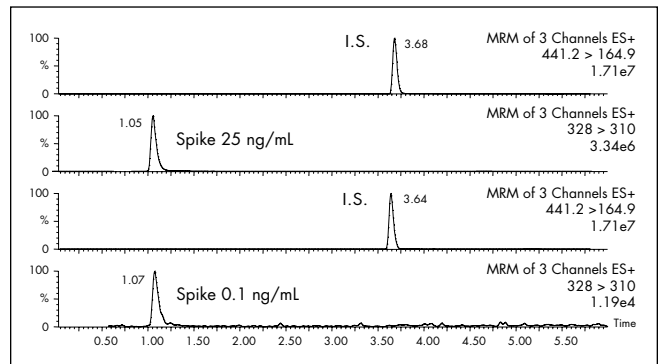


Cell Lysate Analysis

Propranolol at 0.1 ng/mL and 25 ng/mL



Naloxone at 0.1 ng/mL and 25 ng/mL

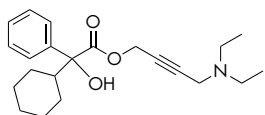


## LC Conditions

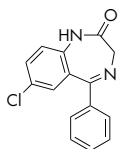
Column: XTerra® MS C<sub>18</sub>, 2.1 x 30 mm, 3.5 µm  
 Part Number: 186000398  
 Mobile Phase A: 100 mM NH<sub>4</sub>COOH, pH 4  
 Mobile Phase B: CH<sub>3</sub>CN  
 Isocratic Mobile  
 Phase Composition: 45% A; 55% B  
 Flow Rate: 0.2 mL/min  
 Injection Volume: 30 µL  
 Instrument: Waters Alliance® 2790

## MS Conditions

Instrument: Waters Quattro Ultima®  
 Ion Source: Electrospray Positive (ESI+)  
 Source Temperature: 150 °C  
 Collision Gas Pressure: 1.5e-3 mbar  
 Collision Energy: 25 eV  
 Desolvation Temperature: 350 °C  
 Cone Gas Flow: 150 L/hr  
 Drying Gas Flow: 600 L/hr  
 Cone Voltage: 30 V



Oxybutynin

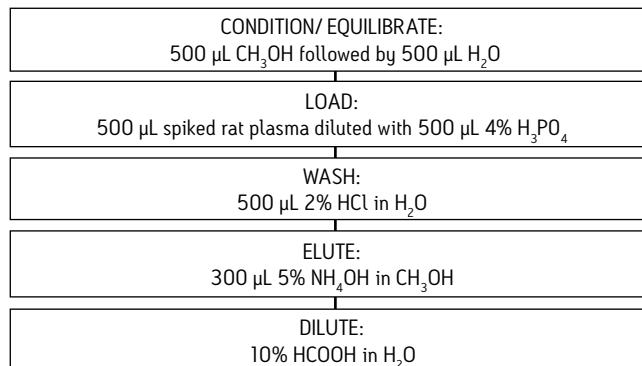


Nordiazepam (I.S.)

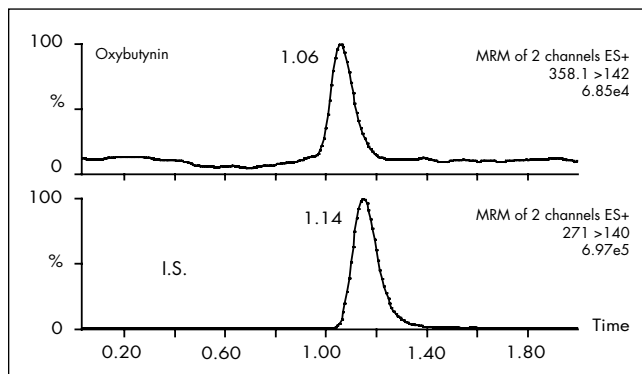
Oxybutynin (ng/ mL)	Mean (n=4)	Standard Deviation	Coefficient of Variation (%)	Recovery (%)
1	1.01	0.01	1.3	101
2.5	2.47 (5)	0.14	5.5	98
5	4.89	0.16	3.3	97
10	9.42 (5)	0.14	1.5	94
20	20.9	0.61	2.9	104
25	25.64	0.43	1.7	102
50	50.14	1.13	2.3	100
100	97.72 (5)	1.95	2	97
200	210.25 (4)	2.51	1.2	105
250	242.98	4.78	1.9	97

## Oasis MCX Extraction Method

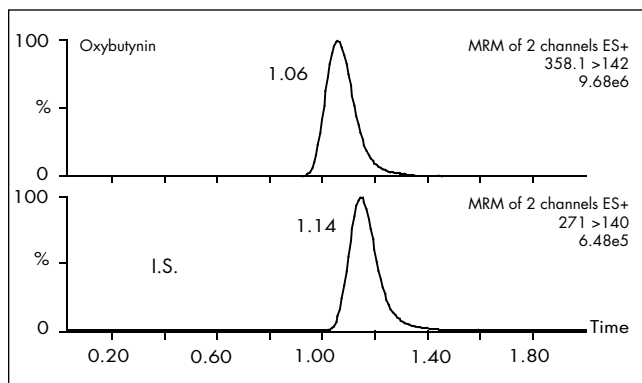
Oasis® MCX Extraction Plate, 10 mg/96 wells  
 Part Number 186000259



## LC/MS Chromatogram in Oxybutynin in Rat Plasma, 1 ng/mL Spiked Level



## LC/MS Chromatogram in Oxybutynin in Rat Plasma, 250 ng/mL Spiked Level

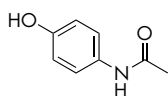


### LC Conditions

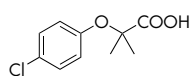
Column: XTerra® MS C<sub>18</sub>, 4.6 x 100 mm, 3.5 μm  
 Part Number: 186000436  
 Mobile Phase A: 15 mM NH<sub>4</sub>HCO<sub>3</sub>, pH 10.5  
 Mobile Phase B: CH<sub>3</sub>OH  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	75	25
10.0	10	90

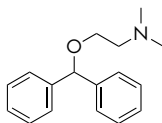
Flow Rate: 1 mL/min  
 Injection Volume: 40 μL  
 Detection: Electrospray Positive (ESI<sup>+</sup>)  
 Instrument: Waters Alliance® 2695, Waters ZQ™



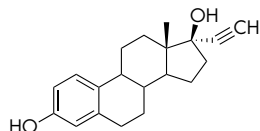
Acetaminophen



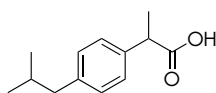
Clofibrac acid



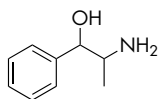
Diphenhydramine



Ethynylestradiol



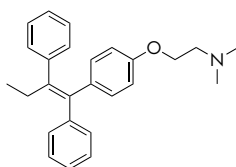
Ibuprofen



Phenylpropanolamine



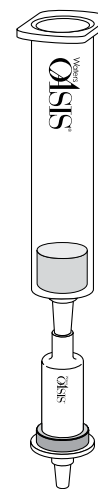
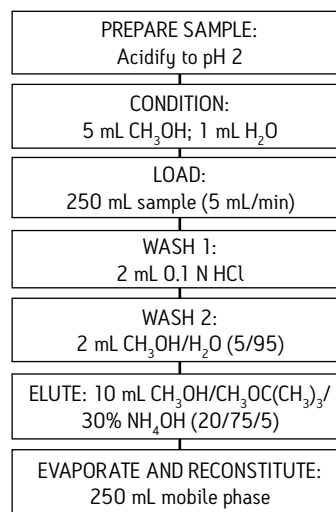
Salicylic acid



Tamoxifen

### Oasis Extraction Method

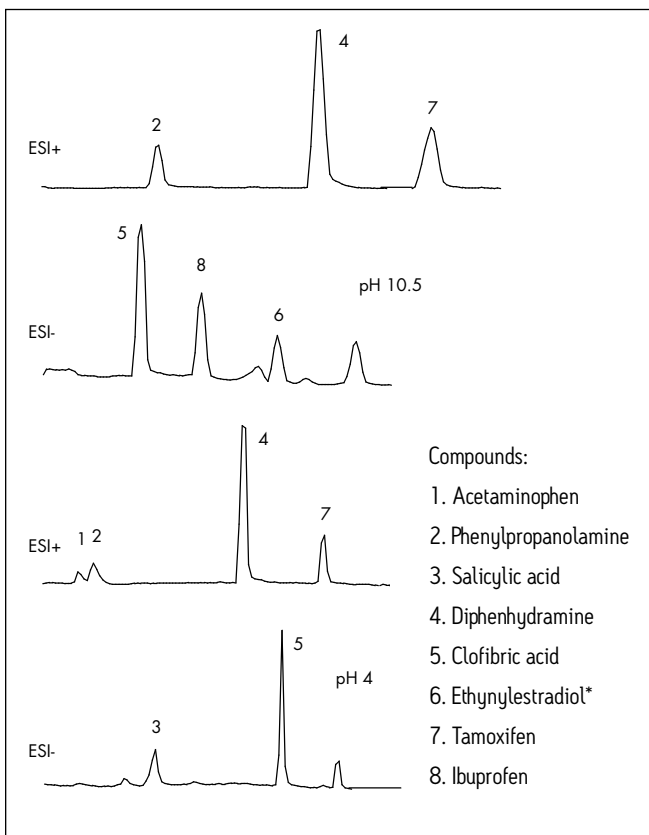
Oasis® MCX Extraction Cartridge, 6 cc/150 mg, 60 μm  
 Part Number 186000255  
 Oasis HLB Plus  
 Part Number 186000132



Oasis MCX 6cc,  
150 mg (60mm)  
P/N 186000255

Oasis HLB Plus  
P/N 186000132  
to increase retention  
of acidic analytes.

LC/MS Chromatogram of Pharmaceuticals Residues in River Water



\* Note: ethynylestradiol response only @ pH 10.5

LC/MS Conditions

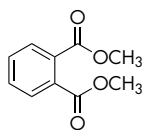
Compound	MW	ESI+	ESI-
Acetaminophen	151.2	152(40) 110(40)	150(20)
Phenylpropanolamine	151.2	152(40) 134(40)	
Salicylic acid	138.1		137(17)
Diphenhydramine	255.3	256(25) 167(40)	
Clofibric acid	214.7		213(17) 127(17)
Ethynylestradiol	296.4		295(40) 145(80)
Tamoxifen	371.5	372(40)	
Ibuprofen	206.3		205(17) 159(30)

Compound	Response (Area ng/L)	r <sup>2</sup>	RSD Level (%)*
1. Acetaminophen	248.6	0.9999	11, 4.3, 5.1, 3.6
2. Phenylpropanolamine	635	0.9996	21, 22, 7.0, 3.6
3. Salicylic acid (3 levels)	27.1	0.9999	nd, 15, 19, 9.0
4. Diphenhydramine	3793	0.9985	35, 4.6, 19, 6.8
5. Clofibric acid	62.35	0.9998	2.6, 2.3, 3.2, 3.9
6. Ethynylestradiol (pH4)			no response
6. Ethynylestradiol (pH11)	104	0.9997	6.2, 5.1, 8.1, 11
7. Tamoxifen	1092	0.9990	15, 11, 13, 7.9
8. Ibuprofen	19.6	0.9999	20, 5.2, 6.4, 5.8

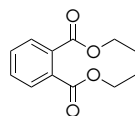
\* %RSD observed for 5 replicates at 25, 100, 500, 2500 ng/L spike levels

Compound	Spike Level	Recovery (%)	RSD (%)
1. Acetaminophen	500, 2500	int, 78	7.7, 10
2. Phenylpropanolamine	500, 2500	nd, 61	nd, 5.7
3. Salicylic acid	500, 2500	60, 61	20, 10
4. Diphenhydramine	500, 2500	89, 86	3.7, 4.6
5. Clofibric acid	500, 2500	101, 89	4.0, 4.2
6. Ethynylestradiol	500, 2500	93, 86	7.3, 4.1
7. Tamoxifen	500, 2500	76, 76	6.0, 2.1
8. Ibuprofen	500, 2500	82, 77	7.7, 4.7

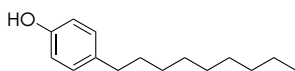
# PHTHALATES AND NONYLPHENOL FROM RIVER WATER BY GC/MS (ENDOCRINE DISRUPTORS)



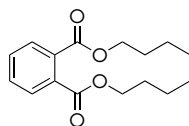
Dimethyl phthalate



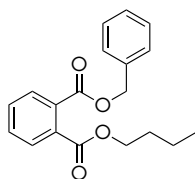
Diethyl phthalate



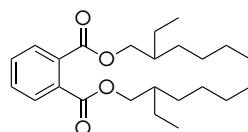
*p*-n-Nonylphenol



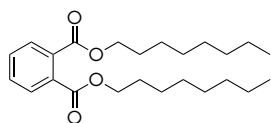
Dibutyl phthalate



Benzylbutyl phthalate



bis(2-Ethylhexyl) phthalate



Dioctyl phthalate

Compounds	% Recovery (% RSD)
1. Dimethyl phthalate	130 (15)
2. Diethyl phthalate	86 (12)
3. <i>p</i> -n-Nonylphenol	90 (11)
4. Dibutyl phthalate	110 (11)
5. Benzylbutyl phthalate	110 (8)
6. bis(2-Ethylhexyl) phthalate	60 (8)
7. Dioctyl phthalate	<50

200 ng/L Spike Level, *n* = 4

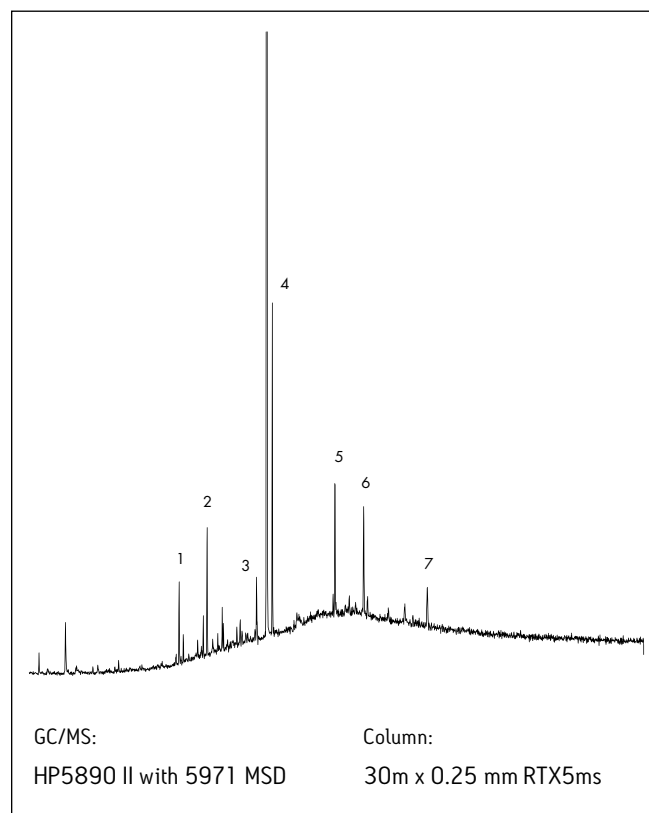
## Oasis HLB Extraction Method

Oasis® HLB Glass Cartridge, 5 cc/200 mg  
Part Number 186000683

PREPARE SAMPLE
CONDITION/EQUILIBRATE: 3 mL CH <sub>3</sub> OC(CH <sub>3</sub> ) <sub>3</sub> *; 3 mL CH <sub>3</sub> OH; 3 mL H <sub>2</sub> O
LOAD: up to 500 mL sample
WASH 1: 3 mL CH <sub>3</sub> OH/H <sub>2</sub> O (40/60)
RE-EQUILIBRATE: 3 mL H <sub>2</sub> O
WASH 2: 3 mL [CH <sub>3</sub> OH/H <sub>2</sub> O (10/90)]/ NH <sub>4</sub> OH (98/2)
ELUTE: 6 mL CH <sub>3</sub> OH/ CH <sub>3</sub> OC(CH <sub>3</sub> ) <sub>3</sub> * (10/90)

\* (C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>O can be used as an alternative to MTBE

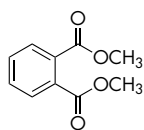
## GC/MS Chromatogram of Phthalates and Nonylphenol in River Water



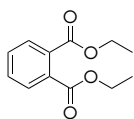
# PHTHALATES IN TAP WATER BY LC/UV (ENDOCRINE DISRUPTORS)

## LC Conditions

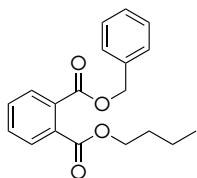
Column: SymmetryShield™ RP8, 3.9 x 150 mm, 5 µm  
 Mobile Phase A: H<sub>2</sub>O  
 Mobile Phase B: CH<sub>3</sub>CN  
 Gradient: 50% B linear to 100% B in 10 min  
 Flow Rate: 0.8 mL/min  
 Injection Volume: 20 µL  
 Sample: 150 mL of surface H<sub>2</sub>O spiked @ 4 ng/L  
 Detection: UV @ 196nm (0.03 AUFS)



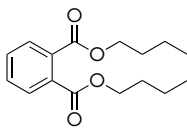
Dimethyl phthalate



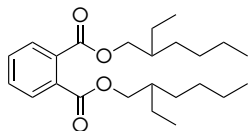
Diethyl phthalate



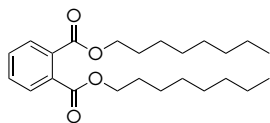
Benzylbutyl phthalate



Dibutyl phthalate



bis(2-Ethylhexyl) phthalate



Dioctyl phthalate

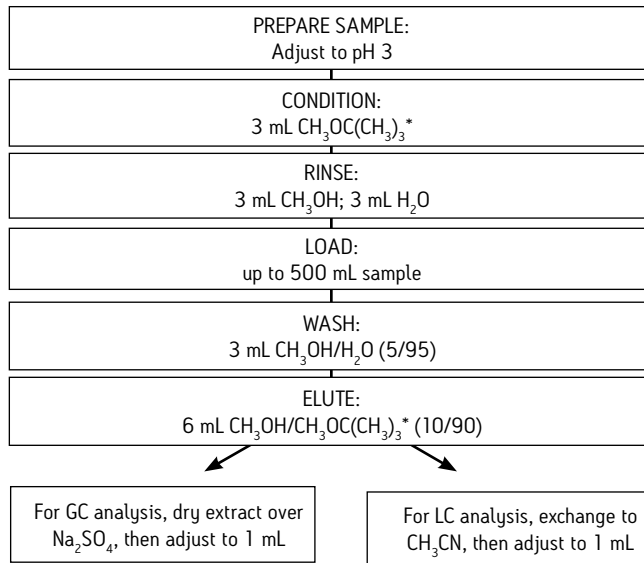
Spike Levels	% Recovery (% RSD) n=5	
	4 µg/L	16 µg/L
1. Dimethyl phthalate	n.d.*	87.2 (3.2)
2. Diethyl phthalate	97.4 (3.8)	92.3 (2.1)
3. Benzylbutyl phthalate	88.7 (14)**	82.2 (1.6)
4. Dibutyl phthalate	88.0 (17)	87.7 (2.2)
5. bis(Ethylhexyl)phthalate	66.9 (22)	72.1 (5.5)
6. Dioctyl phthalate	64.9 (22)	70.6 (5.6)

\* Blank value greater than 50 % of spike level

\*\* Blank value greater than 20 % of spike level

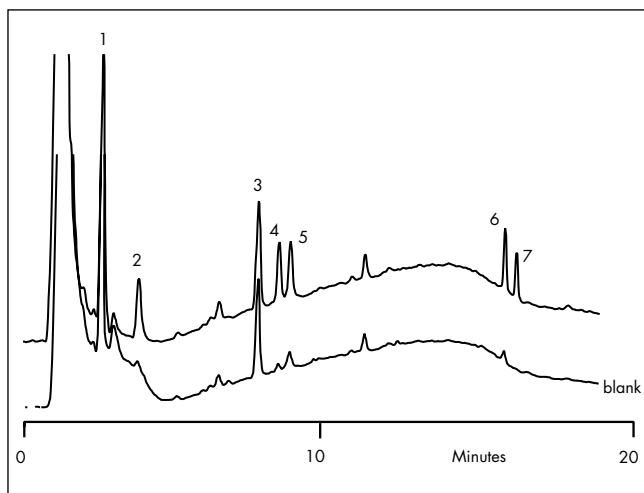
## Oasis SPE Extraction Method

Oasis® HLB Extraction Cartridge, 6 cc/200 mg  
 Part Number WAT106202



\* (C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>O can be used as an alternative to MTBE

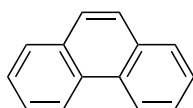
## LC/UV Chromatogram of Phthalates in Tap Water





### LC Conditions

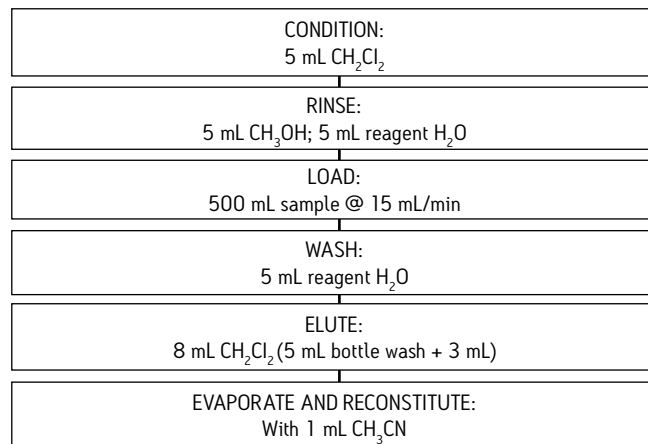
Column: SepServe PAH, 125 x 4.6 mm  
 Mobile Phase A: H<sub>2</sub>O  
 Mobile Phase B: CH<sub>3</sub>CN  
 Gradient: 60% A for 1 min, then linear gradient to 100% B in 15 min  
 Flow Rate: 1.2 mL/min  
 Injection Volume: 20 µL  
 Detection: UV @ 254 nm (0.02 AUFS)



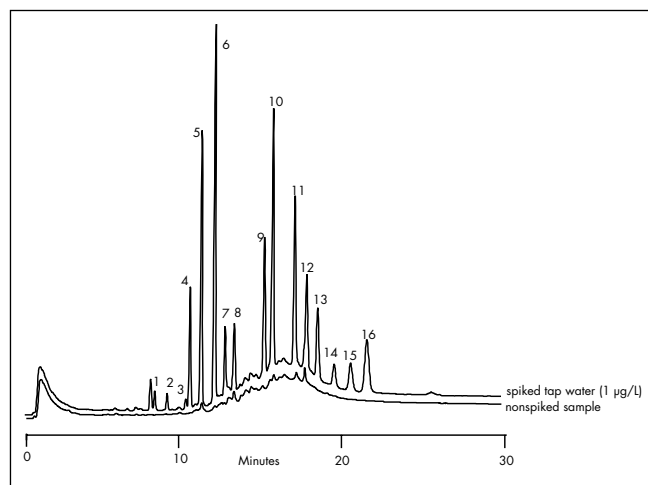
Phenanthrene

### Oasis HLB Extraction Method

Oasis® HLB Extraction Cartridge, 6 cc/200 mg  
 Part Number WAT106202



### LC/UV Chromatogram of Polycyclic Aromatic Hydrocarbons in Drinking Water



Compounds	Recovery (%)	
	Tap Water 1 µg/L 4 Replicates	Tap Water 200 ng/L 4 Replicates
1. Naphthalene	66.5	55*
2. Acenaphthylene	99.2	78*
3. Acenaphthene	98.4	77*
4. Fluorene	105	82.4
5. Phenanthrene	114	94.4
6. Anthracene	103	83.0
7. Fluoranthene	115	91.9
8. Pyrene	117	98.9
9. Benzo(a)anthracene	102	85.9
10. Chrysene	105	90.3
11. Benzo(b)fluoranthene	104	80.1
12. Benzo(k)fluoranthene	90.9	77.4
13. Benzo(a)pyrene	94.3	69.8
14. Dibenzo(g,h,l)perylene	92.1	59.5
15. Benzoperylene	92.0	65.9
16. Indenopyrene	92.8	62.3
	RSDs < 5%	RSDs < 10%

\*result @ 220 nm

# PROTRIPTYLINE IN RAT PLASMA BY MIXED-MODE WEAK CATION EXCHANGE AND LC/MS/MS

## LC Conditions

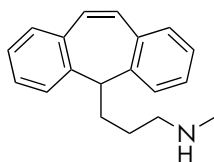
Column: XTerra® MS C<sub>18</sub>, 2.1 x 20 mm I<sup>STM</sup>, 3.5 µm  
 Part Number: 186001923  
 Mobile Phase A: 10 mM NH<sub>4</sub>HCO<sub>3</sub>, pH 10  
 Mobile Phase B: CH<sub>3</sub>OH with 10 mM NH<sub>4</sub>HCO<sub>3</sub>, pH 10  
 Flow Rate: 0.4 mL/min  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	95	5
3.0	5	95
4.0	5	95
4.1	95	5
5.0	95	5

Injection Volume: 10 µL  
 Column Temperature: Ambient  
 Instruments: Waters 2777 Sample Manager and Waters 1525µ Binary HPLC Pump

## MS Conditions

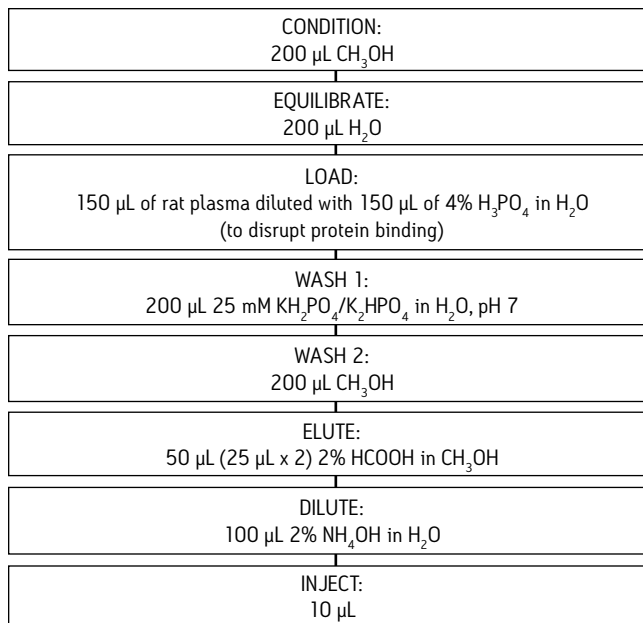
Instruments: Waters Quattro Ultima®  
 Ion Source: Electrospray Positive (ESI<sup>+</sup>)  
 Source Temperature: 150°C  
 Desolvation Temperature: 350 °C  
 Cone Gas Flow: 50 L/hr  
 Desolvation Gas Flow: 550 L/hr  
 Collision Cell Pressure: 2.2e3 Bar (Argon Gas)  
 Cone Voltage: 60 V  
 Collision Energy: 25 eV  
 MRM Transition: m/z 264.0 → 191.1



Protriptyline

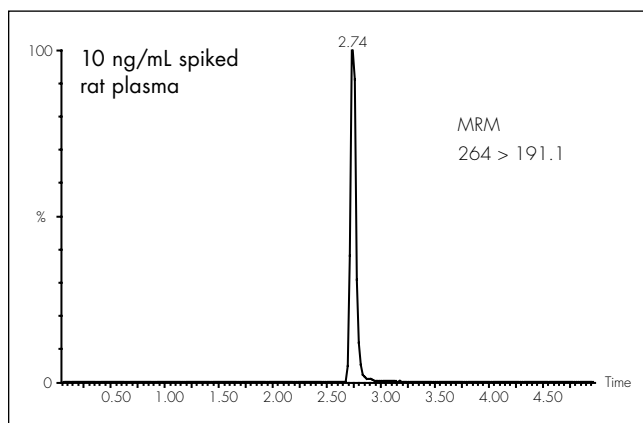
## Oasis WCX Extraction Method

Oasis® WCX 96-well µElution Plate  
 Part Number: 186002499



**SPE Recovery: 102%**

## LC/MS Chromatogram of Protriptyline in Rat Plasma



### LC/MS Method

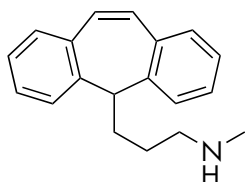
Column: XTerra® MS C<sub>18</sub> 2.1 x 20 mm *IS*<sup>TM</sup>, 3.5 µm  
 Part Number: 186001923  
 Mobile Phase A: 10 mM N NH<sub>4</sub>HCO<sub>3</sub>, pH 10  
 Mobile Phase B: CH<sub>3</sub>OH with 10 mM NH<sub>4</sub>HCO<sub>3</sub>, pH 10  
 Flow Rate: 0.4 mL/min  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	95	5
3.0	5	95
4.0	5	95
4.1	95	5
5.0	95	5

Injection Volume: 10 µL  
 Column Temperature: Ambient  
 Instruments: Waters 2777 Sample Manager and Waters 1525µ Binary HPLC Pump

### MS Conditions

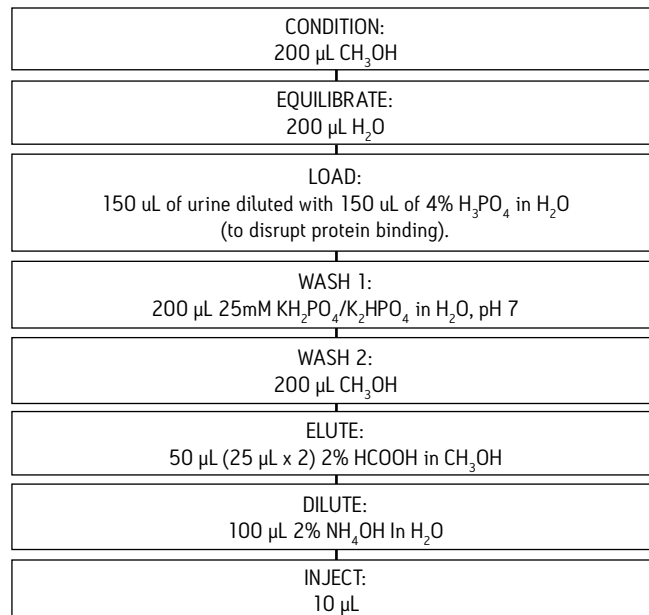
Instrument: Waters Quattro Ultima®  
 Ion Source: Electrospray Positive (ESI<sup>+</sup>)  
 Source Temperature  
 Desolvation: 150 °C  
 Temperature: 350 °C  
 Cone Gas Flow: 50 L/hr  
 Desolvation Gas Flow: 550 L/hr  
 Collision Cell Pressure: 2.2e3 Bar (Argon Gas)  
 Cone Voltage: 60 V  
 Collision Energy: 25 eV  
 MRM Transition: m/z 264.0 → 191.1



Protriptyline

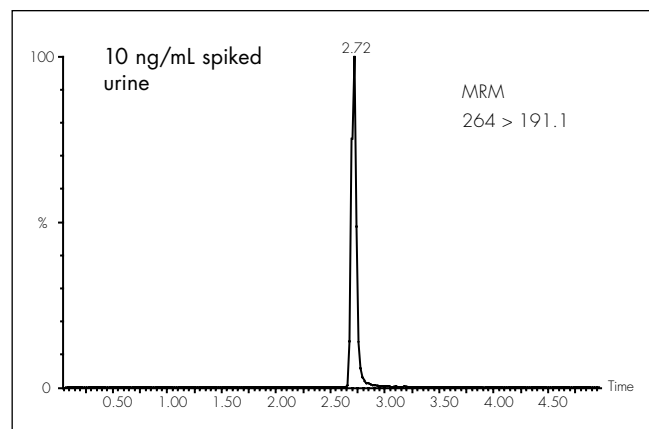
### Oasis WCX Extraction Method

Oasis® WCX 96-well µElution Plate  
 Part Number 186002499



SPE Recovery: 107%

### LC/MS Chromatogram of Protriptyline in Urine



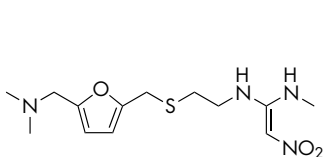
# RANITIDINE IN RAT PLASMA (H<sub>2</sub> RECEPTOR ANTAGONIST) BY LC/MS/MS

## LC Conditions

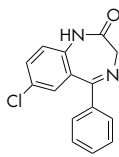
Column: XTerra® MS C<sub>18</sub> 2.1 x 30 mm, 3.5 µm  
 Part Number: 186000398  
 Mobile Phase A: 100 mM NH<sub>4</sub>COOH  
 Mobile Phase B: CH<sub>3</sub>CN  
 Isocratic Mobile  
 Phase Composition: 30% A; 70% B  
 Flow Rate: 0.2 mL/min  
 Injection Volume: 15 µL  
 Instrument: Waters Alliance® 2790

## MS Conditions

Instrument: Waters Quattro Ultima®  
 Ion Source: Electrospray Positive (ESI+)  
 Source Temperature: 150 °C  
 Collision Cell Pressure: 1.5e-3 mbar  
 Collision Energy: 20 eV  
 Desolvation Temperature: 350 °C  
 Cone Gas Flow: 150 L/hr  
 Drying Gas Flow: 600 L/hr  
 Cone Voltage: 20 V



Ranitidine



Nordiazepam (I.S.)

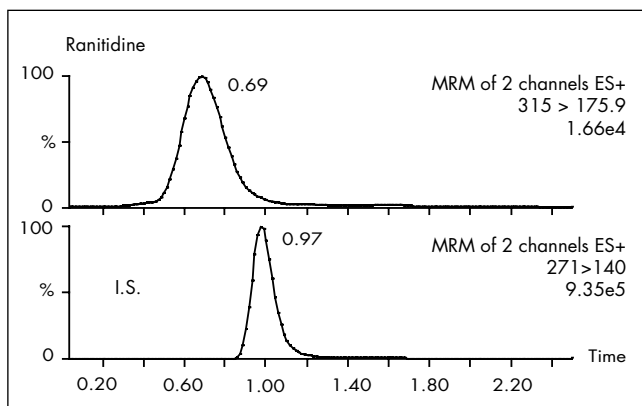
Ranitidine Spike Level (ng/mL)	Mean (n=5)	Standard Deviation	Coefficient of Variation (%)	Recovery (%)
1	0.99	0.02	2	99
2.5	2.54	0.11	4.3	100
5	4.9	0.3	6.1	98
10	10.26	0.68	6.6	102
20	19.46	0.97	4.9	97
50	49.78	2.51	5	99
100	101.55	5.31	5.2	101
200	201.97	4.67	2.3	101
250	247.09	1.4	0.5	99

## Oasis MCX Extraction Method

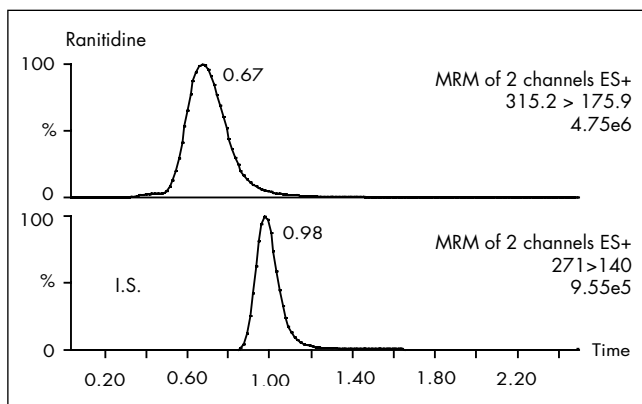
Oasis® MCX Extraction Plate, 10 mg/96 wells  
 Part Number 186000259

CONDITION: 500 µL CH <sub>3</sub> OH followed with 500 µL H <sub>2</sub> O
LOAD: 500 µL spiked rat plasma diluted 1/1 with I.S. in 5% NH <sub>4</sub> OH in H <sub>2</sub> O
WASH: 500 µL 2% HCl in H <sub>2</sub> O
ELUTE: 300 µL 5% NH <sub>4</sub> OH in CH <sub>3</sub> OH
DILUTE: 200 µL H <sub>2</sub> O

## LC/MS Chromatogram of Ranitidine in Rat Plasma, 1 ng/mL Spiked Level



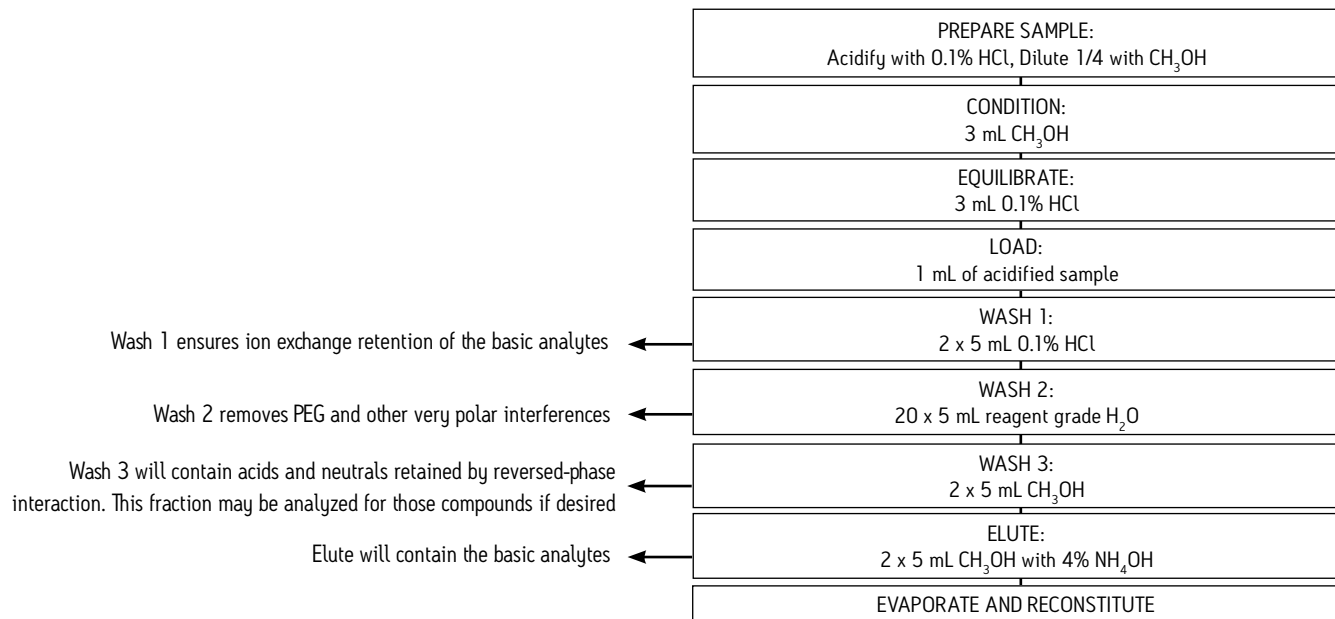
## LC/MS Chromatogram of Ranitidine in Rat Plasma, 250 ng/mL Spiked Level



## Oasis MCX Extraction Method

This method enables high retention of bases in acidified samples.

Oasis® MCX Extraction Cartridge, 6 cc/150 mg, 60 µm,  
Part Number 186000255



# RISPERIDONE AND 9-HYDROXYRISPERIDONE IN HUMAN PLASMA ON OASIS MCX BY UPLC/MS/MS

## LC Conditions

Column: ACQUITY UPLC® BEH C<sub>18</sub>  
2.1 x 50 mm, 1.7 µm

Mobile Phase A: 0.1% HCOOH in H<sub>2</sub>O

Mobile Phase B: 0.1% HCOOH in CH<sub>3</sub>OH

Flow Rate: 0.3 mL/min

Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	60	40
1.0	60	40
1.5	0	100
3.5	0	100
4.0	60	40
4.5	60	40

Injection Volume: 10 µL

Column Temperature: 40 °C

Sample Temperature: 10 °C

Instrument: Waters ACQUITY UPLC System

## MS Conditions

Instrument: Waters Quattro Premier™

Ion Source: Electrospray Positive (ESI+)

Capillary: 3.5 kV

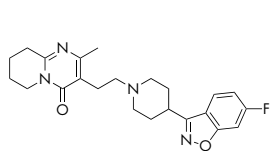
Source Temperature: 120 °C

Desolvation Temperature: 350 °C

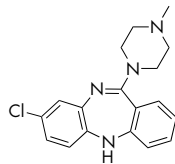
Cone Gas Flow: 0 L/hr

Desolvation Gas Flow: 700 L/hr

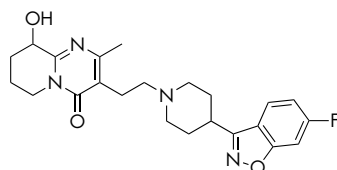
Collision Cell Pressure: 2.59 e-3 mbar



Risperidone



Clozapine (I.S.)



9-Hydroxyrisperidone

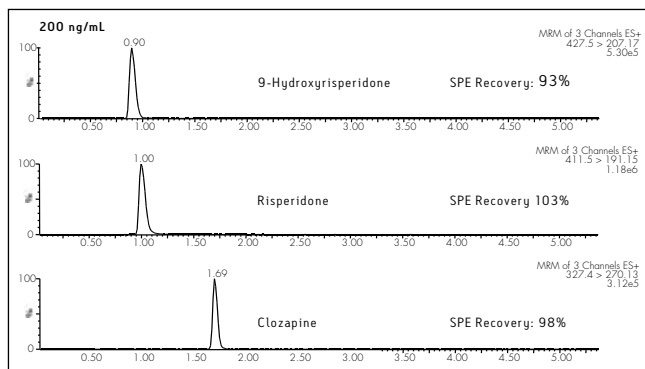
## Oasis MCX Extraction Method

Oasis® MCX Plate, 10 mg/96 wells  
Part Number 186000259

CONDITION: 500 µL CH <sub>3</sub> OH
EQUILIBRATE: 500 µL H <sub>2</sub> O
LOAD: 500 µL (250 µL human plasma, diluted 1/1 with 4% H <sub>3</sub> PO <sub>4</sub> in H <sub>2</sub> O)
WASH 1: 500 µL 2% HCOOH
WASH 2: 500 µL CH <sub>3</sub> OH
ELUTE: 250 µL (125 µL x 2) 5% NH <sub>4</sub> OH in CH <sub>3</sub> OH
OPTIONS: 1. Dilute 250 µL H <sub>2</sub> O with 2% HCOOH 2. Evaporate/Reconstitute 3. Direct inject
INJECT: 10 µL

Compound	Precursor Ion m/z	Product Ion m/z	Cone Voltage (V)	Collision Energy (eV)
Risperidone	411.5	191	40	30
9-Hydroxy risperidone	427.5	207	40	30
Clozapine	327.4	270	35	25

## UPLC/MS Chromatogram of Risperidone and 9-Hydroxyrisperidone in Human Plasma



**LC Conditions**

Column: ACQUITY UPLC® BEH C<sub>18</sub>,  
2.1 x 50 mm, 1.7 μm  
Mobile Phase A: 10 mM NH<sub>4</sub>COOH, pH 9  
Mobile Phase B: CH<sub>3</sub>OH  
Flow Rate: 0.5 mL/min  
Gradient:

Time (min)	Profile		Curve
	A (%)	B (%)	
Initial	95	5	6
2.0	2	98	6
2.5	2	98	6
2.6	95	5	6
3.0	95	5	6

Injection Volume: 8.0 μL  
Column Temperature: 45 °C  
Sample Temperature: 15 °C  
Sample Diluent: H<sub>2</sub>O/CH<sub>3</sub>OH (25/75)  
Strong Needle Wash: CH<sub>3</sub>CN/(CH<sub>3</sub>)<sub>2</sub>CHOH (60/40) + 0.5% HCOOH (1200 μL)  
Weak Needle Wash: H<sub>2</sub>O/CH<sub>3</sub>OH (500 μL) (95/5)

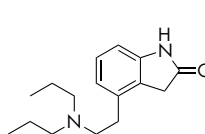
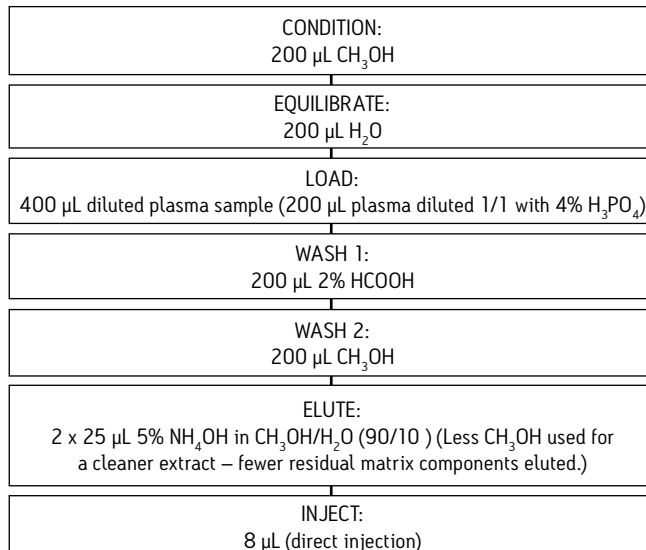
**MS Conditions**

Instrument: Waters Quattro Premier™  
Ion Source: Electrospray Positive (ESI+)  
Desolvation Temperature: 350 °C  
Cone Gas Flow: 50 L/hr  
Desolvation Gas Flow: 700 L/hr  
Collision Cell Pressure: 2.6 x 10<sup>-3</sup> mbar  
MRM Transitions  
Monitored: Ropinirole m/z 261.2 → 113.95 (ESI+)  
Citalopram (I.S.) m/z 325.2 → 108.85 (ESI+)

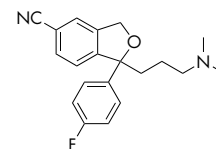
For both analytes, the optimal cone voltage was 40 V.  
For Ropinirole and Citalopram, the collision energies were 18 eV and 25 eV, respectively.

**Oasis MCX Extraction Method**

Oasis® MCX 96-well μElution Plate  
Part Number 186001850

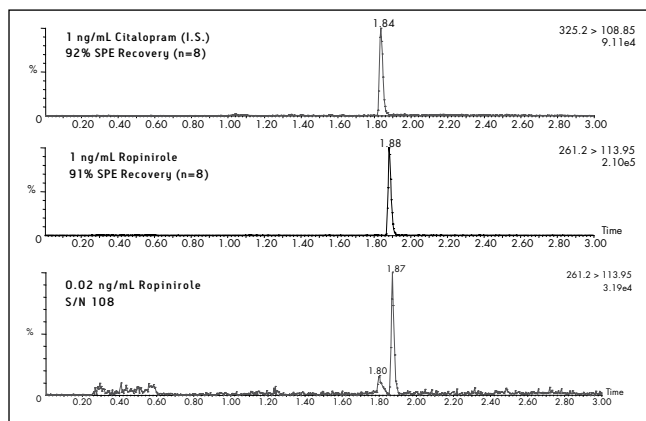


Ropinirole



Citalopram (I.S.)

**UPLC/MS Chromatogram of Ropinirole and Citalopram in Plasma**



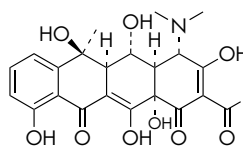
# SULFA DRUGS IN PORCINE SERUM (ANTIBACTERIAL) BY LC/UV

## LC Conditions

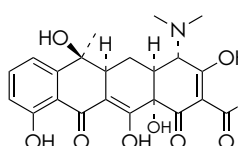
Column: Symmetry® C<sub>18</sub>, 3.9 x 150 mm, 5 µm  
 Part Number: WAT046980  
 Mobile Phase: 1% glacial CH<sub>3</sub>COOH and 4% CH<sub>3</sub>OH in H<sub>2</sub>O  
 Flow Rate: 1 mL/min  
 Injection Volume: 10 µL of reconstituted porcine serum extract  
 Temperature: 25 °C  
 Detection: UV @ 254 nm

## Compounds:

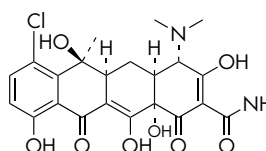
1. Sulfadiazine
2. Sulfathiazole (I.S.)
3. Sulfamerazine



Sulfadiazine



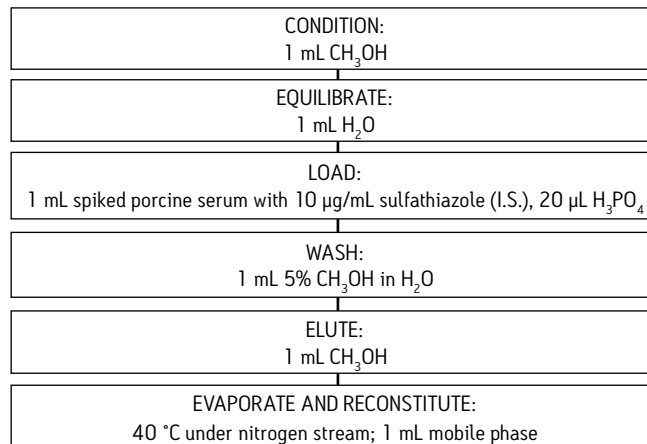
Sulfathiazole (I.S.)



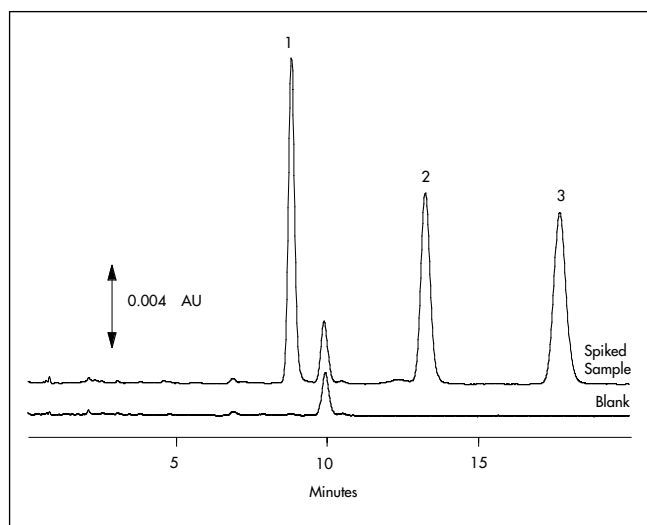
Sulfamerazine

## Oasis HLB Extraction Method

Oasis® HLB Extraction Cartridge, 1 cc/30 mg  
 Part Number WAT094225



## LC/UV Chromatogram of Sulfa Drugs in Serum



Compounds	Concentration (µg/ mL)	% Recovery (µg/mL)	RSD %, (n=6)
Sulfadiazine	2.00	98.4	2.7
	10.0	96.9	0.65
Sulfamerazine	2.00	93.5	1.9
	10.0	96.4	0.74



### LC Conditions

Column: XTerra® MS C<sub>18</sub>, 2.1 x 30 mm, 3.5 µm  
 Part Number: 186000398  
 Mobile Phase A: H<sub>2</sub>O + 0.1M NH<sub>4</sub>HCO<sub>3</sub>, pH 9.5  
 Mobile Phase B: CH<sub>3</sub>OH + 0.1M NH<sub>4</sub>HCO<sub>3</sub>, pH 9.5  
 Flow Rate: 0.4 mL/min  
 Gradient:

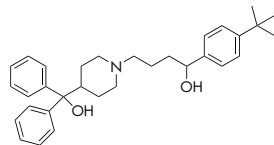
Time (min)	Profile	
	A (%)	B (%)
Initial	95	5
1	5	95

Temperature: Ambient  
 LC Instrument: Waters Alliance® 2795

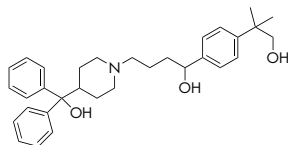
### MS Conditions

MS Instrument: Waters Quattro Ultima®  
 Ion Source: Electrospray Positive (ESI+)  
 Source Temperature: 150 °C  
 Collision Cell Pressure: 2.0 e-3 bar Argon  
 Cone Voltage: 35 V  
 Capillary Voltage: 3.5 Kv  
 Drying Gas Flow: 500 L/hr  
 Cone Gas Flow: 50 L/hr  
 Desolvation  
 Temperature: 350 °C

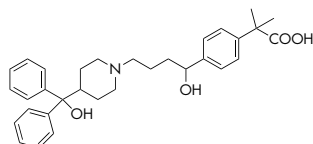
Compounds	MRM Transition (m/z)
Protriptyline (I.S.)	263.9 → 190.8
Terfenadine	472.2 → 436.2
Terfenadine-Alcohol	488.2 → 452.2
Terfenadine-Carboxylate	502.2 → 466.2



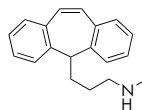
Terfenadine



Terfenadine Alcohol



Terfenadine Carboxylate



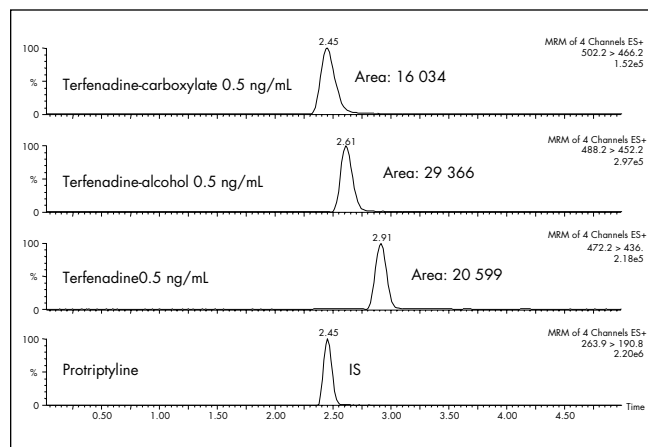
Protriptyline (I.S.)

### Oasis MCX Extraction Method

Oasis® MCX 96-well µElution Plate  
 Part Number 186001830BA

CONDITION: 200 µL CH <sub>3</sub> OH
EQUILIBRATE: 200 µL H <sub>2</sub> O
MIX/LOAD: 250 µL spiked rat plasma + 250 µL I.S. (10 pg/µL) solution
WASH 1: 200 µL H <sub>2</sub> O + 2% HCOOH in H <sub>2</sub> O
WASH 2: 200 µL CH <sub>3</sub> OH
ELUTE: 50 µL CH <sub>3</sub> CN/(CH <sub>3</sub> ) <sub>2</sub> CHOH (40/60) + 5% NH <sub>4</sub> OH
DILUTE: 50 µL H <sub>2</sub> O
INJECT: 20 µL

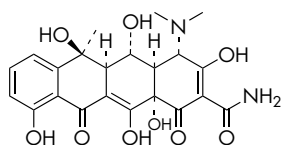
### LC/MS Chromatograms of Terfenadine and Metabolites in Rat Plasma



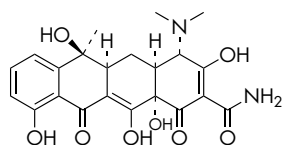
# TETRACYCLINES IN MILK [ANTIBACTERIALS] BY LC/UV

## LC Conditions

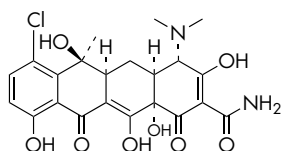
Column: Nova-Pak® C<sub>8</sub>, 3.9 x 150 mm, 4 μm  
 Part Number: WAT086344  
 Mobile Phase: CH<sub>3</sub>CN/CH<sub>3</sub>OH/50 mM oxalic acid in H<sub>2</sub>O (13/13/74)  
 Flow Rate: 0.8 mL/min  
 Injection Volume: 60 μL  
 Detection: UV @ 365 nm



Oxytetracycline



Tetracycline



Chlortetracycline

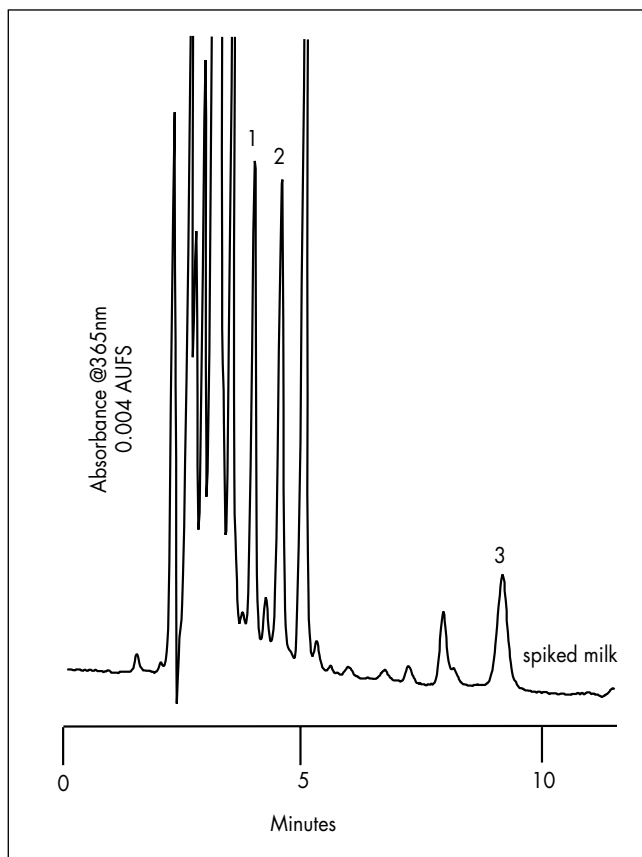
Compounds	% Recovery (% RSD) n=4	
	Tap Water	Apple
	Spike Level	Spike Level
	25 μg/L 4 Replicates	50 μg/L 4 Replicates
1. Oxytetracycline	70.7 (3.5)	67.7 (5.8)
2. Tetracycline	73.7 (7.3)	68.5 (5.1)
3. Chlortetracycline	76.7 (2.9)	67.3 (1.8)

## Oasis HLB Extraction Method

Oasis® HLB Extraction Cartridge, 3 cc/60 mg  
 Part Number WAT094226

CONDITION: 3 mL CH <sub>3</sub> OH
RINSE: 2 mL H <sub>2</sub> O
LOAD: 15 mL sample @ 5 mL/min
WASH: 1.5 mL of 5% CH <sub>3</sub> OH in H <sub>2</sub> O
ELUTE: 2 mL CH <sub>3</sub> OH
EVAPORATE to 0.2 mL
RECONSTITUTE to 1.0 mL with 50 mM oxalic acid

## LC/UV Chromatogram of Tetracyclines in Milk

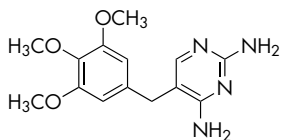


**LC Conditions**

Column: XTerra® MS C<sub>18</sub>, 2.1 x 30 mm, 3.5 µm  
 Part Number: 186000398  
 Mobile Phase A: 1% NH<sub>4</sub>OH  
 Mobile Phase B: CH<sub>3</sub>CN  
 Isocratic Mobile  
 Phase Composition: 40% A; 60% B  
 Flow Rate: 0.2 mL/min  
 Injection Volume: 30 µL  
 Detection: Electrospray Positive (ESI+)  
 Instrument: Waters Alliance® 2790

**MS Conditions**

Instrument: Waters Quattro Ultima®  
 Ion Source: Electrospray Positive (ESI+)  
 Source Temperature: 150 °C  
 Collision Cell Pressure: 1.5e-3 mbar  
 Collision Energy: 25 eV  
 Desolvation Temperature: 350 °C  
 Cone Gas Flow: 150 L/hr  
 Drying Gas Flow: 600 L/hr  
 Cone Voltage: 20 V

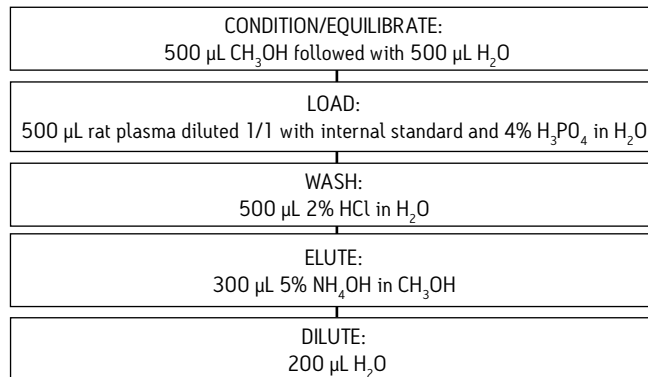


Trimethoprim

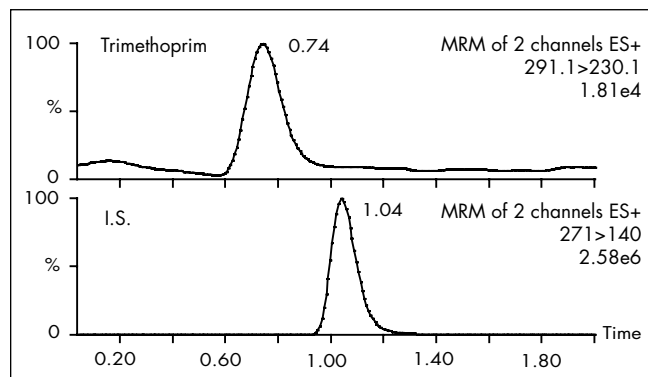
Trimethoprim (ng/mL)	Mean	Standard Deviation	Coefficient of Variation (%)	Recovery (%)
1	1.013	0.039	3.9	101
2.5	2.54	0.061	2.4	101
5	4.86	0.18	3.8	97
10	10.015	0.18	1.8	100
20	20.31	0.3	1.5	101
25	24.64	0.76	3.1	98
50	51.62	1.1	2.1	103
100	96.95	0.98	1	96
200	204.13	5.22	2.6	102
250	247.42	4.93	2	98

**Oasis MCX Extraction Method**

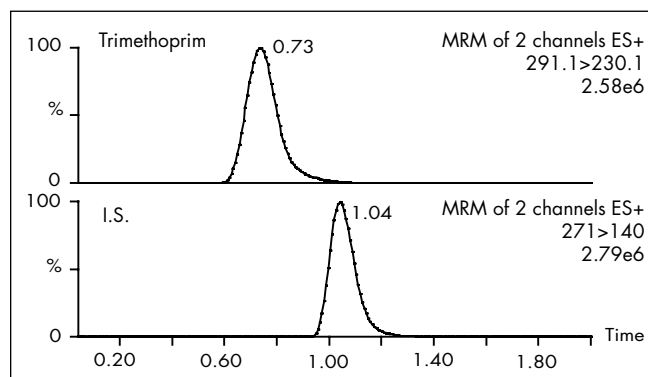
Oasis® MCX Extraction Plate, 10 mg/96 wells  
 Part Number 186000259



**LC/MS Chromatogram of Trimethoprim in Rat Plasma, 1 ng/mL Spiked Level**



**LC/MS Chromatogram of Trimethoprim in Rat Plasma, 250 ng/mL Spiked Level**



# VALETHAMATE IN RAT PLASMA BY MIXED-MODE WEAK CATION EXCHANGE AND LC/MS/MS

## LC Conditions

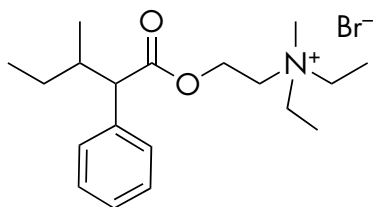
Column: XTerra® MS C<sub>18</sub>, 2.1 x 20 mm *IS*<sup>TM</sup>, 3.5 µm  
 Part Number: 186001923  
 Mobile Phase A: 10 mM NH<sub>4</sub>HCO<sub>3</sub>, pH 10  
 Mobile Phase B: CH<sub>3</sub>OH with 10 mM NH<sub>4</sub>HCO<sub>3</sub>, pH 10  
 Flow Rate: 0.4 mL/min  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	95	5
3.0	5	95
4.0	5	95
4.1	95	5
5.0	95	5

Injection Volume: 10 µL  
 Column Temperature: Ambient  
 Instruments: Waters 2777 Sample Manager  
 Waters 1525µ Binary HPLC Pump

## MS Conditions

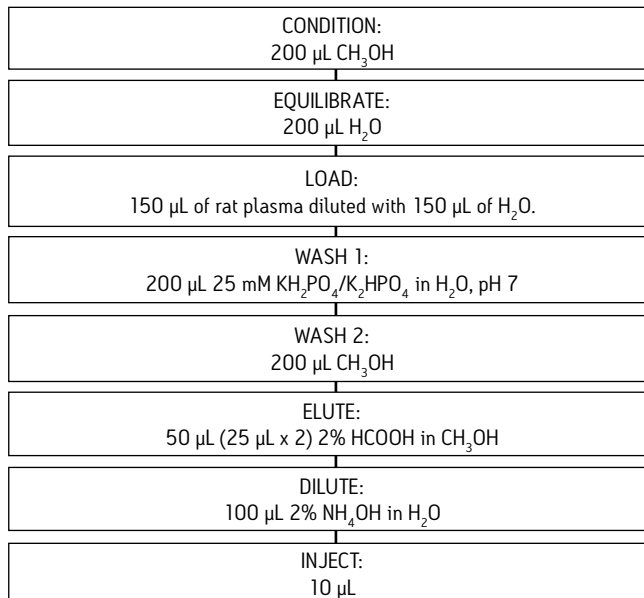
Instrument: Waters Quattro Ultima®  
 Ion Source: Electrospray Positive (ESI<sup>+</sup>)  
 Source Temperature: 150 °C  
 Desolvation  
 Temperature: 350 °C  
 Cone Gas Flow: 50 L/hr  
 Desolvation Gas Flow: 550 L/hr  
 Collision Cell Pressure: 2.2e3 bar (Argon gas)  
 Cone Voltage: 35 V  
 Collision Energy: 20 eV  
 MRM Transition: m/z 306.1 → 218.9



Valethamate

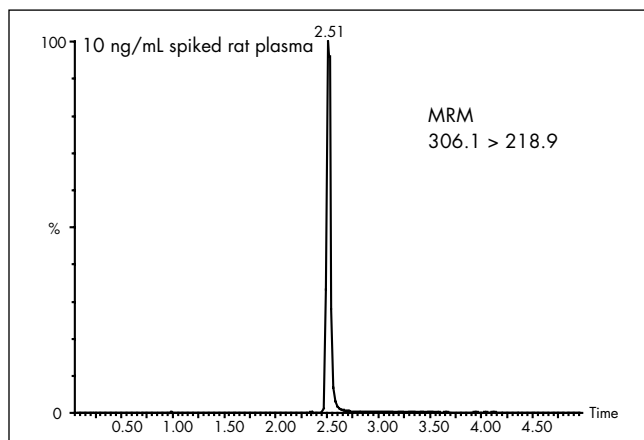
## Oasis WCX Extraction Method

Oasis® WCX 96-well µElution Plate  
 Part Number 186002499



**SPE Recovery: 106%**

## LC/MS Chromatogram of Valethamate in Rat Plasma



### LC Conditions

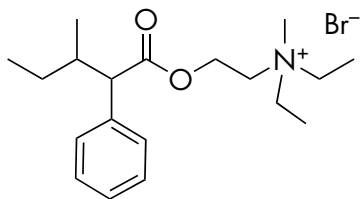
Column: XTerra® MS C<sub>18</sub>, 2.1 x 20 mm /S™, 3.5 µm  
 Part Number: 186001923  
 Mobile Phase A: 10 mM NH<sub>4</sub>HCO<sub>3</sub>, pH 10  
 Mobile Phase B: CH<sub>3</sub>OH with 10 mM NH<sub>4</sub>HCO<sub>3</sub>, pH 10  
 Flow Rate: 0.4 mL/min  
 Gradient:

Time (min)	Profile	
	A (%)	B (%)
Initial	95	5
3	5	95
4	5	95
4	95	5
5	95	5

Injection Volume: 10 µL  
 Column Temperature: Ambient  
 Instruments: Waters 2777 Sample Manager and Waters 1525µ Binary HPLC Pump

### MS Conditions

Instrument: Waters Quattro Ultima®  
 Ion Source: Electrospray Positive (ESI+)  
 Source Temperature: 150°C  
 Desolvation Temperature: 350 °C  
 Cone Gas Flow: 50 L/hr  
 Desolvation Gas Flow: 550 L/hr  
 Collision Cell Pressure: 2.2e3 bar (Argon Gas)  
 Cone Voltage: 35 V  
 Collision Energy: 20 eV  
 MRM Transition: m/z 306.1 → 218.9



Valethamate

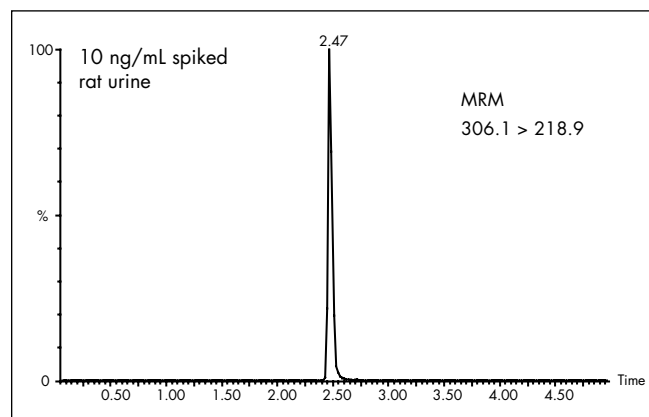
### Oasis WCX Extraction Method

Oasis® WCX 96-well µElution Plate  
 Part Number 186002499

CONDITION: 200 µL CH <sub>3</sub> OH
EQUILIBRATE: 200 µL H <sub>2</sub> O
LOAD: 150 µL of urine diluted with 150 µL of H <sub>2</sub> O
WASH 1: 200 µL 25 mM KH <sub>2</sub> PO <sub>4</sub> /K <sub>2</sub> HPO <sub>4</sub> in H <sub>2</sub> O, pH 7
WASH 2: 200 µL CH <sub>3</sub> OH
ELUTE: 50 µL (25 µL x 2) 2% HCOOH in CH <sub>3</sub> OH
DILUTE: 100 µL 2% NH <sub>4</sub> OH in H <sub>2</sub> O
INJECT: 10 µL

SPE Recovery: 104%

### LC/MS Chromatogram of Valethamate in Urine



## Oasis Cartridges



	1 cc/ 10 mg	1 cc/ 30 mg	1 cc/30 mg Flangeless	1 cc/30 mg Gilson Adapter	3 cc/ 60 mg	3 cc/60 mg Flangeless	3 cc/60 mg Gilson Adapter	6 cc/ 150 mg
Sorbent	Box of 100	Box of 100	Box of 100	Box of 500	Box of 100	Box of 100	Box of 500	Box of 30
Oasis HLB 30 $\mu$ m	186000383	WAT094225	186001879	WAT058882	WAT094226	186001880	WAT058883	186003365
Oasis HLB 60 $\mu$ m	—	—	—	—	—	—	—	186003379
Oasis MCX 30 $\mu$ m	—	186000252	186001881	186001888	186000254	186001882	—	186000256
Oasis MCX 60 $\mu$ m	—	186000782	—	—	186000253	—	—	186000255
Oasis MAX 30 $\mu$ m	—	186000366	186001883	—	186000367	186001884	—	186000369
Oasis MAX 60 $\mu$ m	—	—	—	—	186000368	—	—	186000370
Oasis WCX 30 $\mu$ m	—	186002494	—	—	186002495	—	—	186002498
Oasis WCX 60 $\mu$ m	—	186002496	—	—	186002497	—	—	—
Oasis WAX 30 $\mu$ m	—	186002489	—	—	186002490	—	—	186002493
Oasis WAX 60 $\mu$ m	—	186002491	—	—	186002492	—	—	—

## Oasis 96-well Plates



Description	5 mg/ 96-well	10 mg/ 96-well	30 mg/ 96-well	60 mg/ 96-well
	1/Pkg	1/Pkg	1/Pkg	1/Pkg
Oasis HLB 30 $\mu$ m	186000309	186000128	WAT058951	—
Oasis HLB 60 $\mu$ m	—	—	—	186000679
Oasis MCX 30 $\mu$ m	—	186000259	186000248	—
Oasis MCX 60 $\mu$ m	—	—	186000250	186000678
Oasis MAX 30 $\mu$ m	—	186000375	186000373	186001256
Oasis MAX 60 $\mu$ m	—	—	—	186001205
Oasis WCX 30 $\mu$ m	—	186002501	186002503	—
Oasis WAX 30 $\mu$ m	—	186002502	186002504	186003915



6 cc/ 200 mg	6 cc/400 mg Flangeless	6 cc/ 500 mg	12 cc/ 500 mg	20 cc/ 1 g	35 cc/ 6 g	Plus 225 mg	Vac RC 30 mg	Vac RC 60 mg	Glass Cartridge 5 cc/200 mg
Box of 30	Box of 100*/500**	Box of 30	Box of 20	Box of 20	Box of 10	Box of 50	Box of 50	Box of 50	Box of 30
WAT106202	—	—	—	—	—	—	186000382	186000381	—
—	—	186000115	186000116	186000117	186000118	186000132	—	—	186000683
—	186001216**	—	—	—	—	—	—	186000261	—
—	—	186000776	—	186000777	186000778	186003516	—	186000380	—
—	186001855*	—	—	—	—	—	186000372	186000371	—
—	—	186000865	—	—	—	186003517	—	186000378	—
—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	—	186003518	—	—	—
—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	—	186003519	—	—	—

## Oasis 96-well $\mu$ Elution Plates



Description	$\mu$ Elution 96-well 1/pkg
Oasis HLB 30 $\mu$ m	186001828BA
Oasis MCX 30 $\mu$ m	186001830BA
Oasis MAX 30 $\mu$ m	186001829
Oasis WCX 30 $\mu$ m	186002499
Oasis WAX 30 $\mu$ m	186002500

## Oasis Sorbent Selection Tools



The Oasis Sorbent Selection Tools enable the rapid development of SPE-LC-MS-MS methods. These Sorbent Selection Tools have all the Oasis sorbents, allowing you the ability to extract your analyte of interest.

Description	Particle Size ( $\mu$ m)	Part No.
NEW Oasis $\mu$ Elution Sorbent Selection Plate, 96-well	30 $\mu$ m	186004475
Oasis Sorbent Selection Plate, 10 mg/96-well	30 $\mu$ m	186003249
Oasis Sorbent Selection Kit, 30 mg/1 cc cartridge	30 $\mu$ m	186003463

## Oasis HLB Sample Extraction Products

Description	Particle Size	Qty.	Part No.	
Oasis HLB cartridge	1 cc/10 mg	30 µm	100/box	186000383
Oasis HLB cartridge	1 cc/30 mg	30 µm	100/box	WAT094225
<b>NEW</b> Oasis HLB cartridge	1 cc/30 mg	30 µm	1000/box	186003908
Oasis HLB flangeless cartridge	1 cc/30 mg	30 µm	100/box	186001879
Oasis HLB cartridge with Gilson ASPC adapter	1 cc/10 mg	30 µm	500/box	186000988
Oasis HLB cartridge with Gilson ASPC adapter	1 cc/30 mg	30 µm	500/box	WAT058882
Oasis HLB cartridge	3 cc/60 mg	30 µm	100/box	WAT094226
Oasis HLB flangeless cartridge	3 cc/60 mg	30 µm	100/box	186001880
Oasis HLB cartridge with Gilson ASPC adapter	3 cc/60 mg	30 µm	500/box	WAT058883
Oasis HLB cartridge	6 cc/200 mg	30 µm	30/box	WAT106202
Oasis HLB	3 cc/400 mg	60 µm	100/box	186003849
<b>NEW</b> Oasis HLB cartridge	3 cc/540 mg	60 µm	100/box	186004134
Oasis HLB flangeless cartridge	3 cc/540 mg	60 µm	100/box	186003852
Oasis HLB cartridge	6 cc/150 mg	30 µm	30/box	186003365
Oasis HLB cartridge	6 cc/150 mg	60 µm	30/box	186003379
Oasis HLB cartridge	6 cc/500 mg	60 µm	30/box	186000115
Oasis HLB cartridge	12 cc/500 mg	60 µm	20/box	186000116
Oasis HLB cartridge	20 cc/1 g	60 µm	20/box	186000117
Oasis HLB cartridge	35 cc/6 g	60 µm	10/box	186000118
Oasis HLB Plus cartridge	225 mg	60 µm	50/box	186000132
Oasis HLB Vac RC cartridge	20 cc/30 mg	30 µm	50/box	186000382
Oasis HLB Vac RC cartridge	20 cc/60 mg	30 µm	50/box	186000381
Oasis HLB glass cartridge	5 cc/200 mg	60 µm	30/box	186000683
Oasis HLB Prospekt 2/Symbiosis cartridge*	1.0 x 10 mm	30 µm	96/box	186001196
<b>NEW</b> Oasis HLB Prospekt 2/Symbiosis cartridge*	2.0 x 10 mm	30 µm	96/box	186003925
Reservoir 30 cc for Oasis cartridges		48/box	WAT011390	
Reservoir 60 cc for Oasis cartridges		12/box	WAT024659	
Reservoir adapter for 1 cc, 3 cc, 6 cc cartridges		10/box	WAT054260	
Reservoir adapter for 12 cc, 20 cc, 35 cc cartridges		10/box	WAT048160	
Reservoir adapter for 5 cc cartridges, Teflon		10/pkg	405000934	
Oasis HLB column	2.1 x 20 mm	5 µm	1/pkg	186002034
Oasis HLB column	3.0 x 20 mm	5 µm	1/pkg	186002037
Oasis HLB column	3.9 x 20 mm	5 µm	1/pkg	186002040
Oasis HLB cartridge column	3.9 x 20 mm	5 µm	1/pkg	186001413
Oasis HLB column	4.6 x 20 mm	5 µm	1/pkg	186002043
Oasis HLB column	2.1 x 20 mm	15 µm	1/pkg	186002035
Oasis HLB column	3.0 x 20 mm	15 µm	1/pkg	186002038
Oasis HLB column	3.9 x 20 mm	15 µm	1/pkg	186002041
Oasis HLB cartridge column	3.9 x 20 mm	15 µm	1/pkg	186001414
Oasis HLB column	4.6 x 20 mm	15 µm	1/pkg	186002044
Oasis HLB column	2.0 x 15 mm	25 µm	1/pkg	186001792
Oasis HLB column	2.1 x 20 mm	25 µm	1/pkg	186002036
Oasis HLB cartridge column	2.1 x 20 mm	25 µm	1/pkg	186000706
Oasis HLB column	3.0 x 20 mm	25 µm	1/pkg	186002039
Oasis HLB column	3.9 x 20 mm	25 µm	1/pkg	186002042
Oasis HLB column	4.6 x 20 mm	25 µm	1/pkg	186002045
Holder kit for 2.1 x 20 mm cartridge column		1/pkg	186000262	
Holder kit for 3.9 x 20 mm cartridge column		1/pkg	WAT046910	
Extraction column connector		1/pkg	WAT082745	
Inline precolumn filter kit		1/pkg	WAT084560	
Replacement filters		5/pkg	WAT005139	
Replacement steel gaskets		1/pkg	WAT084567	
Oasis HLB µElution plate	2 mg/96-well	30 µm	1/pkg	186001828BA
Oasis HLB plate	5 mg/96-well	30 µm	1/pkg	186000309
Oasis HLB plate	10 mg/96-well	30 µm	1/pkg	186000128
Oasis HLB plate	30 mg/96-well	30 µm	1/pkg	WAT058951
Oasis HLB plate	60 mg/96-well	60 µm	1/pkg	186000679

\* For use with Spark Holland Prospekt 2 and Symbiosis systems

## Oasis MCX Sample Extraction Products (Cation Exchange)

Description	Particle Size	Qty.	Part No.	
Oasis MCX cartridge	1 cc/30 mg	30 µm	100/box	186000252
Oasis MCX flangeless cartridge	1 cc/30 mg	30 µm	100/box	186001881
Oasis MCX cartridge	1 cc/60 mg	60 µm	100/box	186000782
Oasis MCX cartridge	3 cc/60 mg	30 µm	100/box	186000254
Oasis MCX flangeless cartridge	3 cc/60 mg	30 µm	100/box	186001882
Oasis MCX cartridge	3 cc/60 mg	60 µm	100/box	186000253
Oasis MCX cartridge	6 cc/150 mg	30 µm	30/box	186000256
Oasis MCX cartridge	6 cc/150 mg	60 µm	30/box	186000255
Oasis MCX cartridge	6 cc/500 mg	60 µm	30/box	186000776
Oasis MCX cartridge	20 cc/1 g	60 µm	20/box	186000777
Oasis MCX cartridge	35 cc/6 g	60 µm	10/box	186000778
Oasis MCX Plus cartridge	225 mg	60 µm	50/box	186003516
Oasis MCX Vac RC cartridge	20 cc/60 mg	30 µm	50/box	186000261
Oasis MCX Vac RC cartridge	20 cc/60 mg	60 µm	50/box	186000380
Oasis MCX Prospekt 2/Symbiosis cartridge*	10 x 1 mm	30 µm	96/box	186002098
Oasis MCX direct connect column	2.1 x 15 mm	30 µm	1/pkg	186002050
Oasis MCX column	2.1 x 20 mm	30 µm	1/pkg	186002046
Oasis MCX cartridge column	2.1 x 20 mm	30 µm	1/pkg	186002051
Oasis MCX column	3.0 x 20 mm	30 µm	1/pkg	186002047
Oasis MCX column	3.9 x 20 mm	30 µm	1/pkg	186002048
Oasis MCX column	4.6 x 20 mm	30 µm	1/pkg	186002049
Oasis MCX µElution plate	96-well	30 µm	1/pkg	186001830BA
Oasis MCX plate	10 mg/96-well	30 µm	1/pkg	186000259
Oasis MCX plate	30 mg/96-well	30 µm	1/pkg	186000248
Oasis MCX plate	30 mg/96-well	60 µm	1/pkg	186000250
Oasis MCX plate	60 mg/96-well	60 µm	1/pkg	186000678

## Oasis MAX Sample Extraction Products (Anion Exchange)

Description	Particle Size	Qty.	Part No.	
Oasis MAX cartridge	1 cc/30 mg	30 µm	100/box	186000366
Oasis MAX flangeless cartridge	1 cc/30 mg	30 µm	100/box	186001883
Oasis MAX cartridge	3 cc/60 mg	30 µm	100/box	186000367
Oasis MAX cartridge	3 cc/60 mg	60 µm	100/box	186000368
Oasis MAX flangeless cartridge	3 cc/60 mg	30 µm	100/box	186001884
Oasis MAX cartridge	6 cc/150 mg	30 µm	30/box	186000369
Oasis MAX cartridge	6 cc/150 mg	60 µm	30/box	186000370
Oasis MAX cartridge	6 cc/500 mg	60 µm	30/box	186000865
Oasis MAX Plus cartridge	225 mg	60 µm	50/box	186003517
Oasis MAX Vac RC cartridge	20 cc/30 mg	30 µm	50/box	186000372
Oasis MAX Vac RC cartridge	20 cc/60 mg	30 µm	50/box	186000371
Oasis MAX Vac RC cartridge	20 cc/60 mg	60 µm	50/box	186000378
Oasis MAX Prospekt 2/Symbiosis cartridge*	10 x 1 mm	30 µm	96/box	186002099
Oasis MAX direct connect column	2.1 x 15 mm	30 µm	1/pkg	186002056
Oasis MAX column	2.1 x 20 mm	30 µm	1/pkg	186002052
Oasis MAX cartridge column	2.1 x 20 mm	30 µm	1/pkg	186002057
Oasis MAX column	3.0 x 20 mm	30 µm	1/pkg	186002053
Oasis MAX column	3.9 x 20 mm	30 µm	1/pkg	186002054
Oasis MAX column	4.6 x 20 mm	30 µm	1/pkg	186002055
Oasis MAX µElution plate	2 mg/96-well	1/pkg	186001829	
Oasis MAX plate	10 mg/96-well	30 µm	1/pkg	186000375
Oasis MAX plate	30 mg/96-well	30 µm	1/pkg	186000373
Oasis MAX plate	60 mg/96-well	30 µm	1/pkg	186001256
Oasis MAX plate	60 mg/96-well	60 µm	1/pkg	186001205

\* For use with Spark Holland Prospekt 2 and Symbiosis systems



**Oasis WCX Sample Extraction Products  
(Weak Cation Exchange)**

Description	Particle Size	Qty.	Part No.
Oasis WCX 1 cc cartridge	30 µm	100/box	186002494
Oasis WCX 3 cc cartridge	30 µm	100/box	186002495
<b>NEW</b> Oasis WCX 6 cc cartridge	30 µm	30/box	186002498
Oasis WCX 1 cc cartridge	60 µm	100/box	186002496
Oasis WCX 3 cc cartridge	60 µm	100/box	186002497
Oasis WCX Plus cartridge	60 µm	50/box	186003518
Oasis WCX µElution plate	30 µm	1/pkg	186002499
Oasis WCX 96-well plate	30 µm	1/pkg	186002501
Oasis WCX 96-well plate	30 µm	1/pkg	186002503
Oasis WCX Prospekt 2/Symbiosis cartridge*	30 µm	96/box	186002892
Oasis WCX 2.1 x 20 mm column	30 µm		186002505
Oasis WCX 3.9 x 20 mm column	30 µm		186002507
Oasis WCX 2.1 x 20 mm column	5 µm		186002510
Oasis WCX 3.9 x 20 mm column	5 µm		186002512

**Oasis WAX Sample Extraction Products  
(Weak Anion Exchange)**

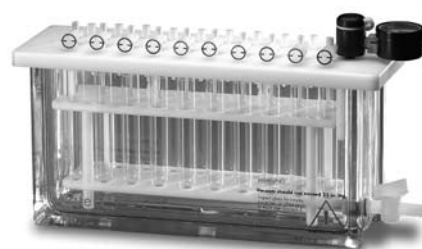
Description	Particle Size	Qty.	Part No.
Oasis WAX 1 cc cartridge	30 µm	100/box	186002489
Oasis WAX 3 cc cartridge	30 µm	100/box	186002490
Oasis WAX 6 cc cartridge	30 µm	30/box	186002493
Oasis WAX 1 cc cartridge	60 µm	100/box	186002491
Oasis WAX 3 cc cartridge	60 µm	100/box	186002492
Oasis WAX Plus cartridge	60 µm	50/box	186003519
Oasis WAX µElution plate	30 µm	1/pkg	186002500
Oasis WAX 96-well plate	30 µm	1/pkg	186002502
Oasis WAX 96-well plate	30 µm	1/pkg	186002504
<b>NEW</b> Oasis WAX 96-well plate	30 µm	1/pkg	186003915
Oasis WAX Prospekt 2/Symbiosis cartridge*	30 µm	96/box	186002893
Oasis WAX 2.1 x 20 mm column	30 µm		186002508
Oasis WAX 3.9 x 20 mm column	30 µm		186002509
Oasis WAX 2.1 x 20 mm column	5 µm		186002511
Oasis WAX 3.9 x 20 mm column	5 µm		186002513

**Oasis Method Development Kits**

Description	Particle Size	Part No.
Oasis sorbent selection plate 3 rows each MCX, MAX, WCX, WAX	96-well	186003249
<b>NEW</b> Oasis µElution Sorbent Selection Plate, 3 rows each MCX, MAX, WCX, WAX	96-well	186004475
Oasis sorbent selection cartridge kit 10 each MCX, MAX, WCX, WAX	30 µm	186003463

**Manifold for Extraction Cartridges**

Description	Part No.
Waters extraction manifold, 20-position without rack (includes 20 needle tips, 25 plugs, and ejector tool)	WAT200677
Waters extraction manifold, 20-position (complete with rack for 13 x 75 mm tubes)	WAT200606
Waters extraction manifold, 20-position (complete with rack for 13 x 100 mm tubes)	WAT200607
Waters extraction manifold, 20-position (complete with rack for 16 x 75 mm tubes)	WAT200608
Waters extraction manifold, 20-position (complete with rack for 16 x 100 mm tubes)	WAT200609



Waters Extraction Manifold

**Accessories for Extraction Column and Cartridges**

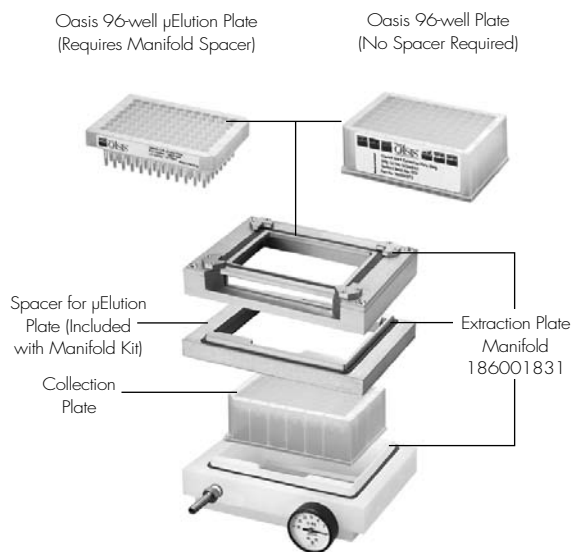
Description	Qty	Part No.
Holder kit for 2.1 x 20 mm cartridge column	1/pkg	186000262
Holder kit for 3.9 x 20 mm cartridge column	1/pkg	WAT046910
Extraction column connector	1/pkg	WAT082745
Inline precolumn filter kit	1/pkg	WAT084560
Replacement filters	5/pkg	WAT005139
Replacement steel gaskets	1/pkg	WAT084567
SPE vacuum pump 115 V 60 Hz		725000417
SPE vacuum pump 240 V 50 Hz		725000418
Reservoir, 30 cc (for Oasis Plus, Light, Vac & Classic cartridges)	48/pkg	WAT011390
Reservoir, 60 cc (for Oasis Plus, Light & Vac cartridges)	12/pkg	WAT024659
Adapter, male-male Luer (for Oasis Classic cartridges)	100/pkg	WAT024310
Adapter (to attach reservoir to 1, 3 & 6 cc Oasis Vac cartridges)	12/pkg	WAT054260
Adapter (to attach reservoir to 12, 20 & 35 cc Oasis Vac cartridges)	10/pkg	WAT048160



SPE Vacuum Pump  
(Includes two gauges and pressure regulator)

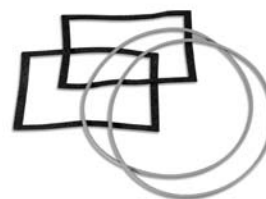
## Manifold for Extraction Plate

Description	Qty	Part No.
Extraction plate manifold for Oasis 96-well plates	1/box	186001831
Extraction plate manifold kit A (includes extraction plate manifold, reservoir tray, sealing cap and 350 µL sample collection plate)		WAT097944
Extraction plate manifold kit B (as kit A, with 1 mL sample collection plate)		WAT097945
Extraction plate manifold kit C (as kit A, with 2 mL sample collection plate)		WAT097946



## Accessories for Extraction Plate Manifold

Description	Qty	Part No.
Disposable reservoir tray	25/box	WAT058942
Sample collection plate, 350 µL	50/box	WAT058943
Sample collection plate, 2 mL	50/box	WAT058958
Sealing cap for 96-well collection plate	50/pkg	WAT058959
SPE vacuum pump 115 V 60 Hz		725000417
SPE vacuum pump 240 V 50 Hz		725000418
Vacuum box gasket kit Kit includes: 2 foam top gaskets 2 orange O-rings		186003522



Vacuum Box Gasket Kit



### Literature References

Oasis Sample Extraction Products Brochure,  
Literature Reference 720001692EN

Oasis µElution Plate Brochure,  
Literature Reference 720000476EN

Topics in Solid-Phase Extraction.  
Part 1. Ion Suppression in  
LC/MS Analysis White Paper,  
Literature Reference 720001237EN

Sample Prep Solutions Brochure,  
Literature Reference 720000848EN

Oasis WAX Sorbent for UPLC/MS  
Determination of PFOS and Related  
Compounds in Waters and Tissue,  
Literature Reference 720001871EN

SPE Sample Preparation for  
UPLC-MS Determination of  
Enrofloxacin (Baytril) in Chicken,  
Literature Reference WA43206

A Sensitive Method for the Determination  
of Endocrine-Disrupting Compounds in  
River Water by LC/MS/MS,  
Literature Reference 720001296EN

The Oasis® family of solid-phase extraction products is designed to simplify and improve your sample preparation by combining the appropriate sorbent, device format and methodology. This enables laboratories to achieve robust, reproducible and sensitive SPE methods. Oasis SPE sorbents—covered by nine US patents—are unique in their purity, stability and retention characteristics.

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