

Nota applicativa

## Pharmaceutical Residues in Environmental Samples – LC-MS And LC/PDA Results of LC/PDA Analysis, 2.5 ppb

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Waters Corporation



This is an Application Brief and does not contain a detailed Experimental section.

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## Abstract

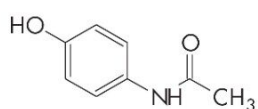
This application brief highlights the analysis of pharmaceutical residues in environmental samples using optimized Oasis MCX SPE method.

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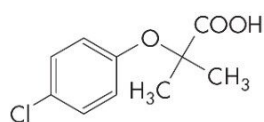
## Introduction

Compounds used in this application brief are:

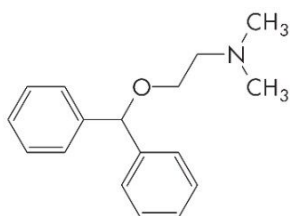
1. Acetaminophen
2. Phenylpropanolamine
3. Salicylic acid
4. Diphenhydramine
5. Clofibric acid
6. Ethynylestradiol
7. Tamoxifen
8. Ibuprofen



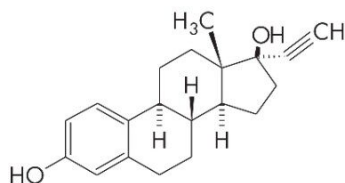
ACETAMINOPHEN



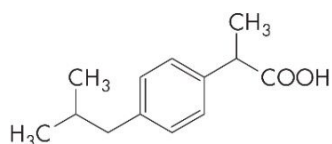
CLOFIBRIC ACID



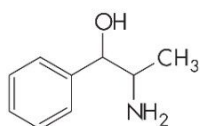
DIPHENHYDRAMINE



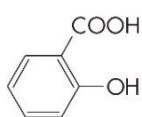
ETHYNYLESTRADIOL



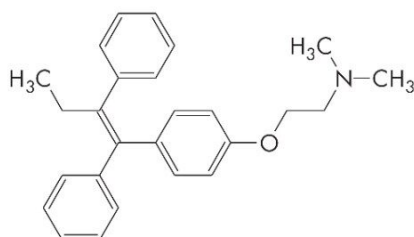
IBUPROFEN



PHENYLPROPANOLAMINE



SALICYLIC ACID



TAMOXIFEN

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## Experimental

### HPLC Method

Column:

XTerra MS C<sub>18</sub> 4.6 x 100 mm, 3.5 μm (p/n:  
186000436)

Mobile phase A:

15 mM NH<sub>4</sub>COOH, pH 4.0

Mobile phase B: MeOH

Flow rate: 1.0 mL/min

Injection volume: 40 µL

Detection: MS ESI+

Instrument: Alliance 2695, Micromass ZQ

## Gradient

Time (min)	Profile	
	%A	%B
0.0	75	25
10.0	10	90

## LC-MS Conditions Waters Micromass ZMD

Compound	MW	ESI+	ESI-
Acetaminophen	151.2	152(40) 110(40)	150(20)
Phenylpropanolamine	175.2	152(40) 134(40)	
Salicylic acid	138.1		137(17)
Diphenhydramine	255.3	256(25) 167(40)	

Compound	MW	ESI+	ESI-
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)

## Optimized SPE Method For LC-MS Determination of Pharmaceutical Residues in Environmental Samples

Conditions for Oasis MCX 6 cc/150 mg (60 µm) Part Number 186000255

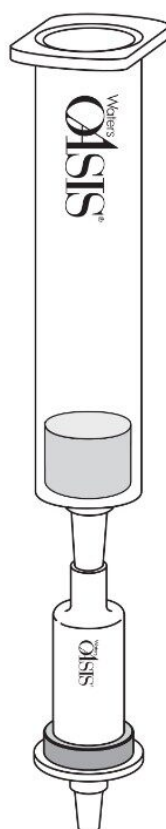
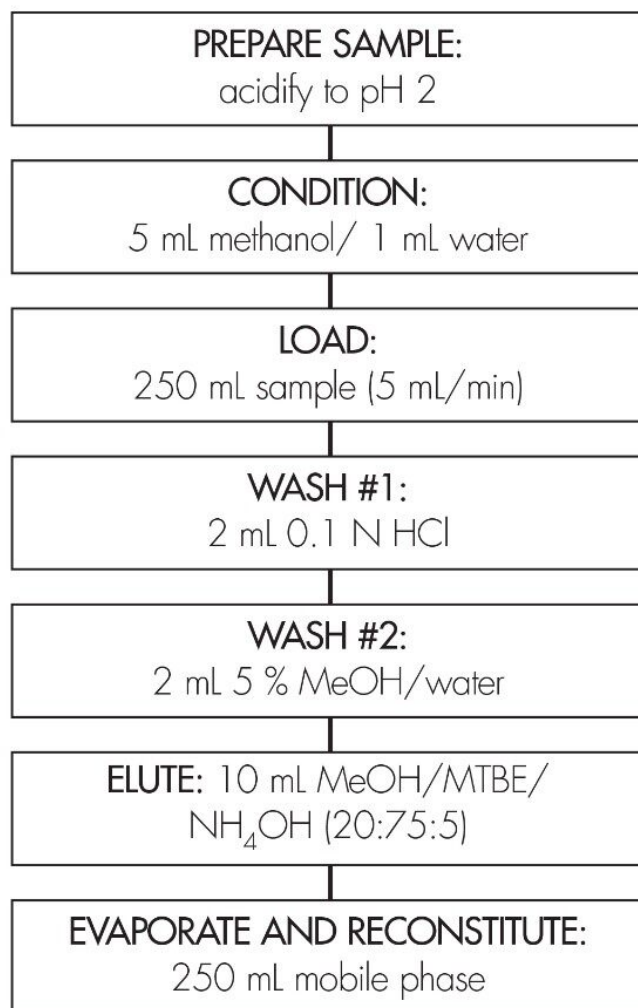
Oasis HLB Plus Part Number 186000132

# OPTIMIZED SPE METHOD FOR LC/MS DETERMINATION OF PHARMACEUTICAL RESIDUES IN ENVIRONMENTAL SAMPLES

Conditions for

Oasis® MCX 6 cc/150 mg (60 µm) Part Number 186000255

Oasis® HLB Plus Part Number 186000132



Oasis® MCX 6 cc,  
150 mg (60 µm)  
Part Number 186000255



Oasis® HLB Plus  
Part Number 186000132



to increase retention  
of acidic analytes.

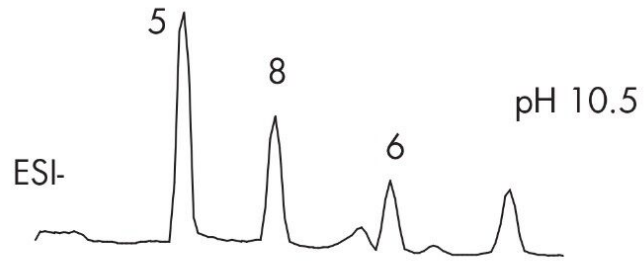
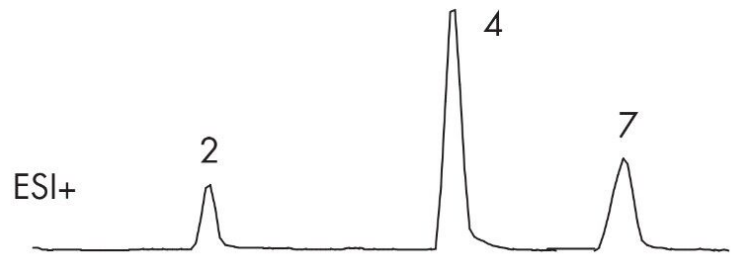
MeOH - methanol MTBE - methyl tbutyl ether  
NH<sub>4</sub>OH - 30% ammonium hydroxide

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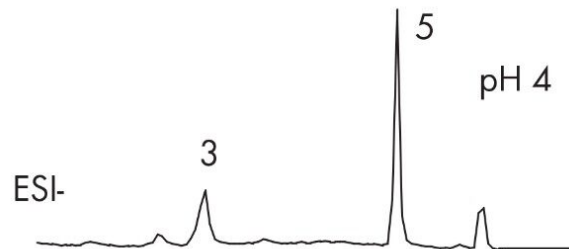
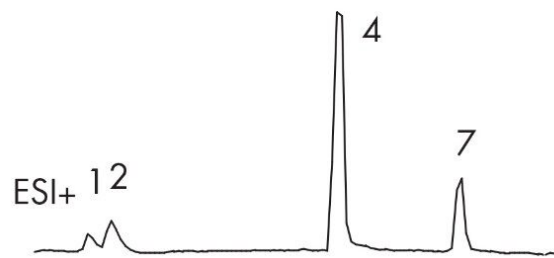
## Results and Discussion

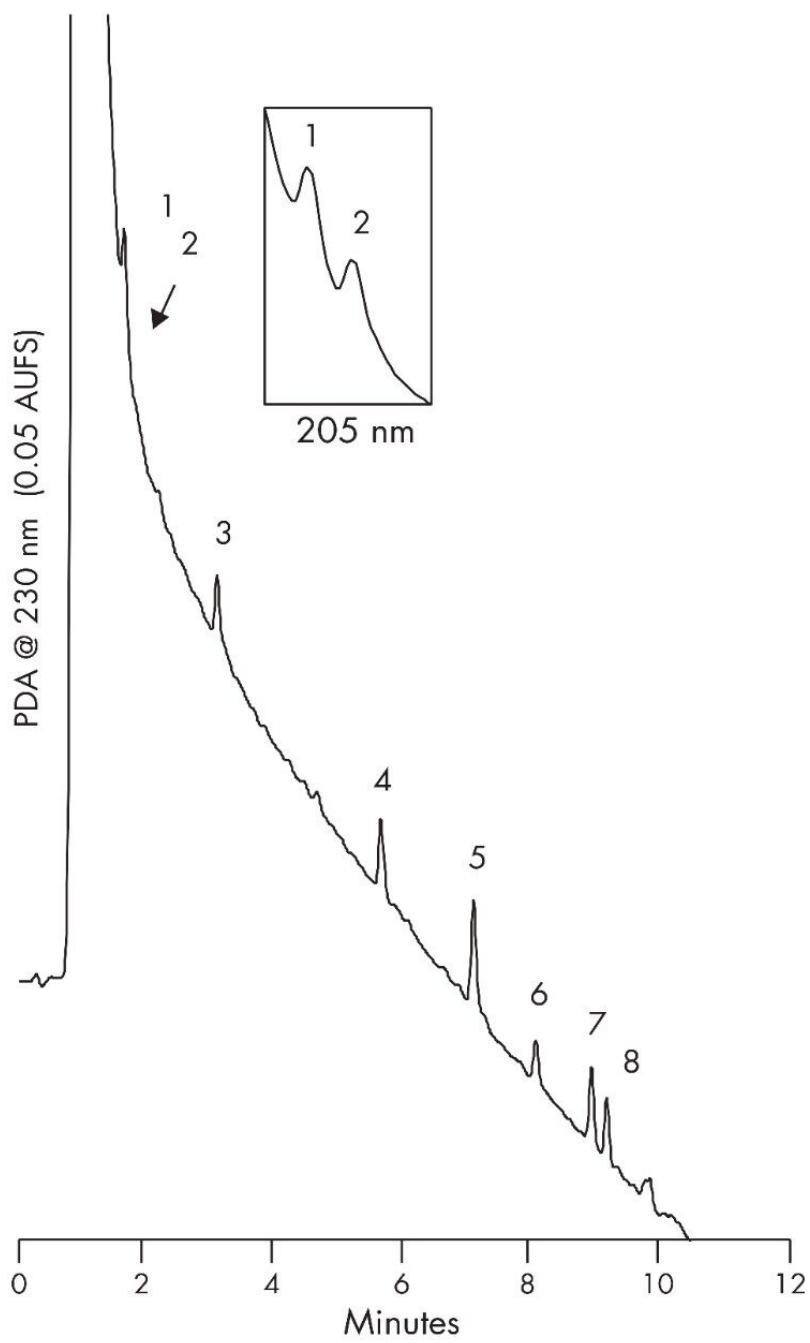
## COMPOUNDS:

1. Acetaminophen
2. Phenylpropanolamine
3. Salicylic acid
4. Diphenhydramine
5. Clofibric acid
6. Ethynylestradiol
7. Tamoxifen
8. Ibuprofen



\* note: ethynylestradiol  
response only @ pH 10.5





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	0.999	621, 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 levels	% 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

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