Extraction of Pharmaceuticals from Water using EVOLUTE ABN Columns

Application Note

Introduction

This procedure is recommended for the extraction of a variety of pharmaceuticals from water using non-polar polymeric SPE. These pharmaceuticals were identified as potential environmental contaminants by the Environment Agency.

The sample preparation method and suggested analytical procedures are detailed in page 1. Achieved recoveries and % RSD using this methodology are shown on page 3.

Sample Preparation Procedure

Analytes

Atenolol, Carbamazepine, Citalopram, Diclofenac, Erythromycin, Fluoxetine, Fluvoxamine, Ibuprofen, Labetalol, Mefenamic Acid, Metoprolol, Oxprenolol, Paroxetine, Propranolol, Sotalol, Sulfamethoxazole, Trimethoprim

EVOLUTE ABN Column Configuration

EVOLUTE ABN 50 µm 200 mg/6 mL, part number 610-0020-C

EVOLUTE ABN Procedure

Sample:	Spike water (500 mL) at 100 ng/L concentration of the compounds listed above.
Sample Pre-treatment:	Not required
Column Conditioning:	Condition each column with methanol (6 mL)
Column Equilibration:	Equilibrate each column with water (6 mL)
Sample Application:	500 mL at a flow rate of ~15 mL/min (-10" Hg)
Interference Elution:	Elute interferences with water (6 mL)
Analyte Elution:	Elute Analytes with methanol (6 mL)
Post Extraction:	Evaporate to dryness. Reconstitute in 200 µL methanol, add 800 µL water prior to injection.

For general guidelines on the use of EVOLUTE ABN SPE columns, request Chemistry Data Sheet **TN136** EVOLUTE ABN Columns for Non-polar Solid Phase Extraction in Environmental Analysis.

Analytical Procedure

Instrument:	Waters Alliance 2795 Separations Module.
Column:	Zorbax Eclipse XDB-C18 (100 x 2.1 mm, 3.5 µm) Agilent
Guard Column:	Zorbax Eclipse XDB-C8 (12.5 x 2.1 mm, 5 µm) Agilent
Injection Volume:	10 µL
Temperature:	0.25 mL/min. Entire column effluent directed into the MS

Biotage

Table 1. HPLC Gradient

Time	A= 0.1% Formic in water	B=acetonitrile
0	88	12
9	53	47
12	10	90
14	10	90
14.10	88	12

MS Conditons

Instrument: Waters Quattro Ultima Pt triple quadrupole MS equipped with an electrospray source
Source Temp: 100 °C
Desolvation Temp: 350 °C
Collision cell pressure: 2.23 e-3 mbar

Table 2. MRM Conditions (analytes listed in order of elution)

Analyte	MRM Transition	Collision Energy (eV)
Atenolol	267.2 > 190.2	18
Sotalol	273.1 > 213.1	17
Trimethoprim	291.2 > 123.1	22
Metoprolol	268.2 > 116.1	18
Oxprenolol	266.2 > 72.1	18
Labetalol	329.2 > 311.1	12
Sulfamethoxazole	254.1 > 156.0	15
Propranolol	260.1 > 116.1	17
Erythromycin	734.5 > 158.2	31
Citalopram	325.1 > 109.1	23
Paroxetine	330.1 > 192.2	19
Fluvoxamine	319.2 > 71.0	15
Carbamazepine	237.1 > 194.1	17
Fluoxetine	310.2 > 148.2	7
Diclofenac*	294.1 > 250.1	11
Ibuprofen*	205.2 > 159.2	7
Mefenamic Acid*	240.2 > 196.2	16

*All positive ion mode except Diclofenac, Ibuprofen, Mefenamic acid Dwell time 0.08-0.15 s; Cone Voltage 35-60 V. Details available on request.

Results

Table 3. Recovery and % RSD of pharmaceuticals from water

Analyte	Analyte recovery (%)	% rsd (n=4)
Atenolol	90	4
Sotalol	95	2
Trimethoprim	99	3
Metoprolol	97	3
Oxprenolol	99	3
Labetalol	81	3
Sulfamethoxazole	94	4
Propranolol	97	2
Erythromycin	102	8
Citalopram	94	3
Paroxetine	82	5
Fluvoxamine	95	9
Carbamazepine	98	1
Fluoxetine	103	6
Diclofenac	103	3
Ibuprofen	102	8
Mefenamic Acid	95	8

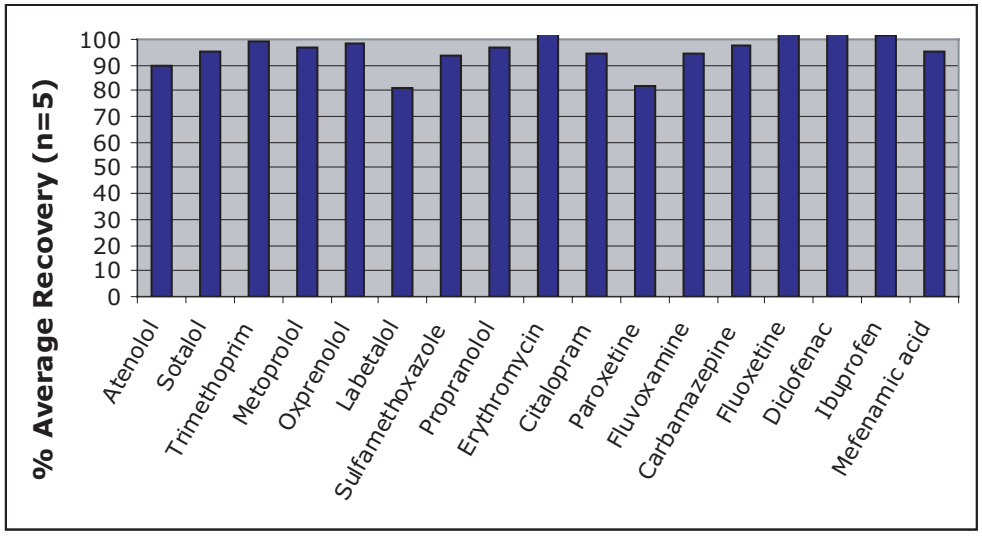

Figure 1. Recoveries (>80%, <10% rsd, n=5) for pharmaceuticals from water using EVOLUTE ABN SPE Columns.

North America
Main Office: +1 434 979 2319
Toll Free: +1 800 446 4752
Fax: +1 434 979 4743
Tel: +1 434 220 2687
Order Fax: +1 434 296 8217
ordermailbox@biotage.com

Europe
Main Office: +46 18 56 5900
Fax: +46 18 59 1922
Tel: +46 18 56 57 10
Order Fax: +46 18 56 57 05
order@eu.biotage.com

Germany
Tel: +49 7624 90 80 0
Fax: +49 7624 90 80 10
separtis@eu.biotage.com

Switzerland
Tel: +41 61 743 90 15
Fax: +41 61 743 90 18
separtis@eu.biotage.com

Austria
Tel: +43 2231 63167
Fax: +43 223163520
separtis@eu.biotage.com

Japan
Tel: +81 422 28 1233
Fax: +81 422 28 1236
jp_order@biotage.com

Distributors
Please visit our Web site at
www.biotage.com
for contact details.