

# EVOLUTE™ ABN Solid Phase Extraction Columns

A Generic Approach to Extraction of  
Pharmaceuticals from Environmental  
Water Samples

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## EVOLUTE ABN SPE Columns

EVOLUTE™ ABN polymeric non-polar SPE sorbent extracts acidic, basic and neutral analytes from aqueous sample matrices using a generic procedure, minimizing method development time for Analytical Chemists.

# Extraction of Pharmaceuticals from River Water

# Introduction

- Analysis of pharmaceuticals from water is increasingly important due to changes in European legislation
- Water and government laboratories are now testing for these on a routine basis
- Need a sample preparation approach that can be used to extract a wide range of compounds in one sample extraction

# Application of EVOLUTE ABN for Extraction of Pharmaceuticals from River Water

- Poster in collaboration with the Environment Agency, UK
  - Presented at Pittcon 2007, 25 Feb - 02 Mar, Chicago

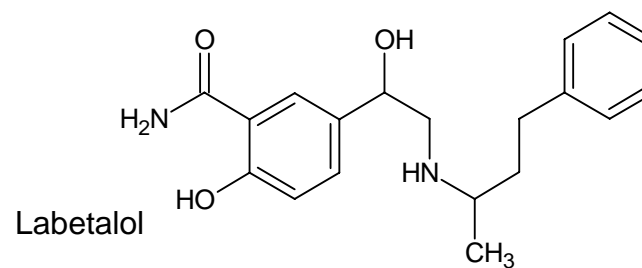
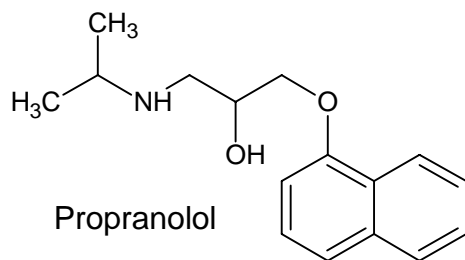
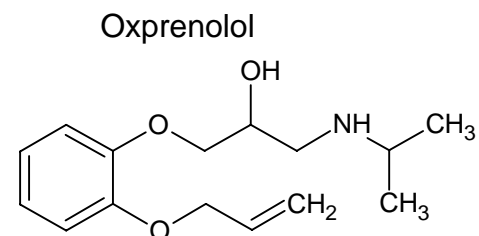
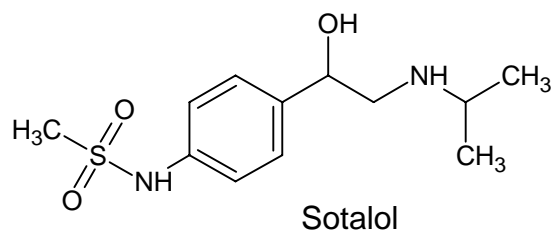
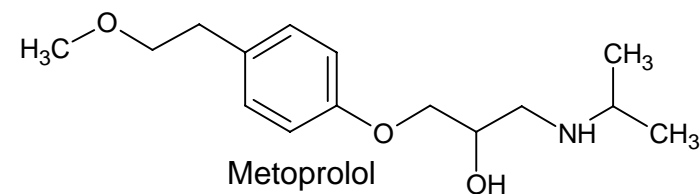
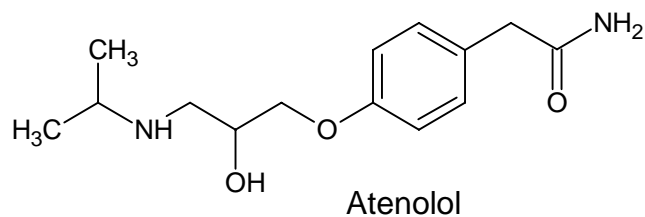
# The Analyte Suite

- Environment Agency has identified 30+ compounds as some of the most widely applicable drugs found in water courses
- Analyte suite was selected from the list to demonstrate the applicability of EVOLUTE ABN for the extraction of analytes with wide ranging pK values and polarities (logP)

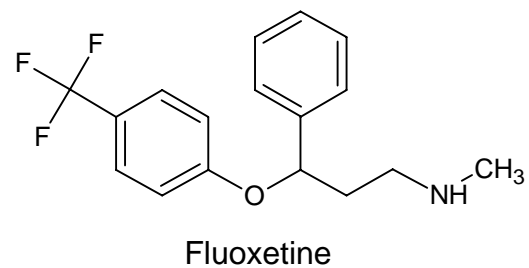
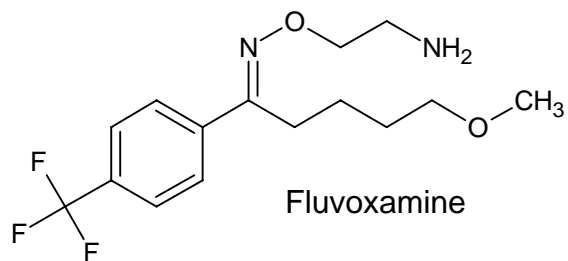
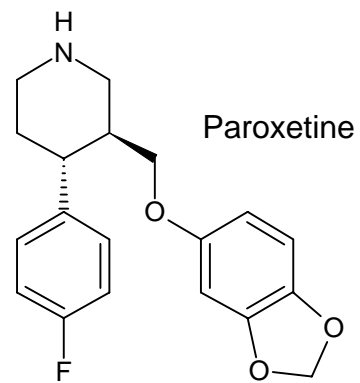
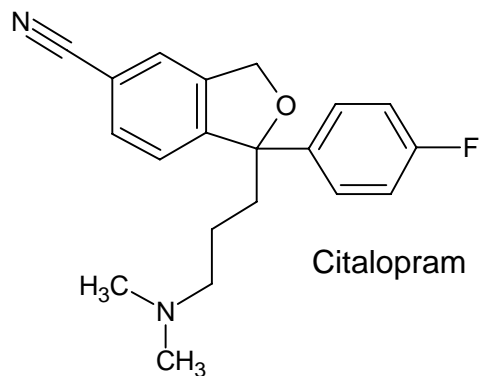
# The Analyte Suite

Analyte	Therapeutic Class	Functionality	pK	logP
Atenolol	Beta blocker	Basic	9.6	0.3
Sotalol	Beta blocker	Basic	8.2, 9.8	0.5
Trimethoprim	Antibacterial	Basic	6.6	1.3
Metoprolol	Beta blocker	Basic	13.3	1.8
Oxprenolol	Beta blocker	Basic	9.2	2.2
Labetalol	Beta blocker	Basic	7.4	2.7
Propranolol	Beta blocker	Basic	9.5	3.1
Erythromycin	Antibacterial	Basic	8.8	2.9
Citalopram	SSRI	Basic	9.5	1.5
Paroxetine	SSRI	Basic	9.9	5.0
Fluvoxamine	SSRI	Basic	8.7	1.3
Carbamazepine	Anticonvulsant	Basic	9.1	1.3
Fluoxetine	SSRI	Basic	9.5	4.2
Diclofenac	Anti-inflammatory	Acidic	4.0	4.4
Sulfamethoxazole	Antibacterial	Acidic	6.0	2.4
Ibuprofen	Anti-inflammatory	Acidic	4.8	3.5
Mefenamic Acid	Anti-inflammatory	Acidic	4.2	4.9

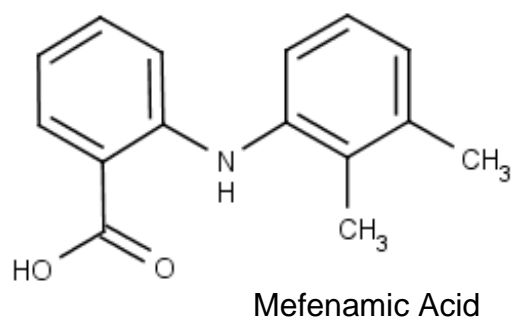
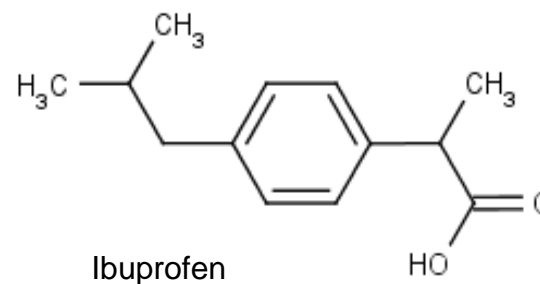
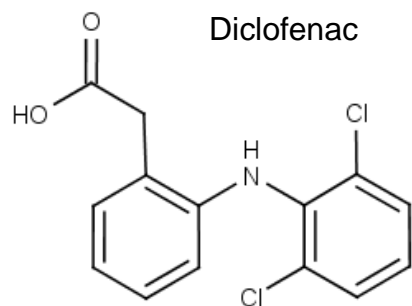
# Analyte Structures: Beta Blockers



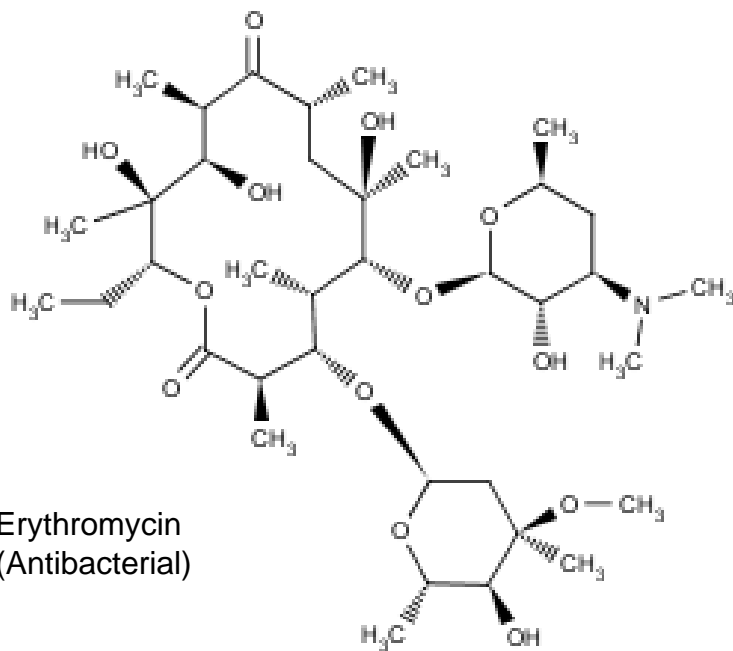
# Analyte Structures: SSRIs



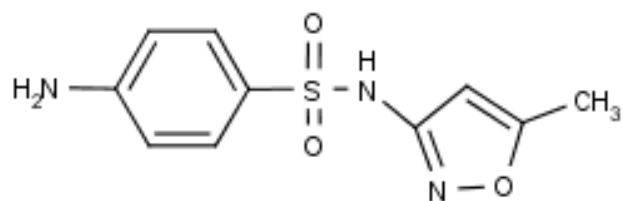
# Analyte Structures: Anti-inflammatory



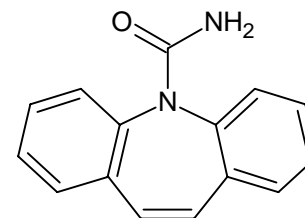
# Analyte Structures: Other



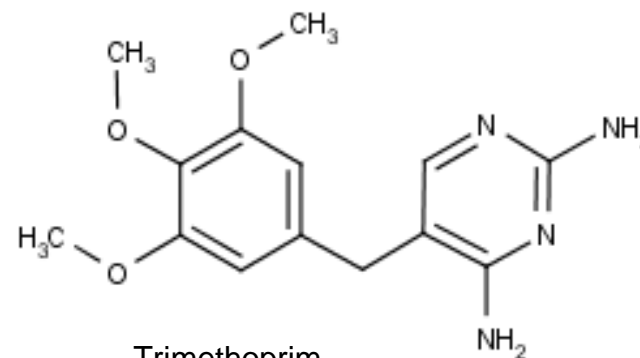
Erythromycin  
(Antibacterial)



Sulfamethoxazole  
(Antibacterial)



Carbamazepine  
(Anticonvulsant)



Trimethoprim  
(Antibacterial)

# Generic Method

## Sample Pre-treatment:

- Dependant on sample type. Solvent extraction, pH control, particulate removal, etc.

## 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  $\sim 15$  mL/min (-10 "Hg)

## Interference Elution:

- Elute interferences with water/methanol (95:5, v/v, 6 mL)

## Analyte Elution:

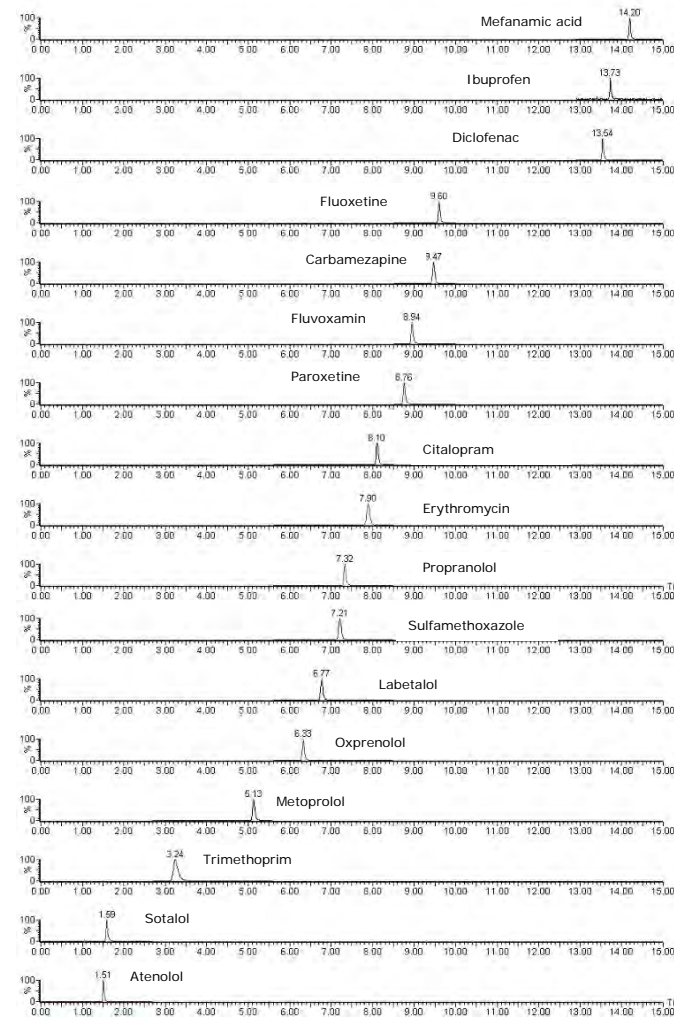
- Elute analytes with methanol (6 mL)

## Post-extraction:

- If desired, evaporate extract to dryness and reconstitute in mobile phase or other suitable solvent for analysis.

# LC-MS Conditions

- Full details on HPLC conditions, Mass Spec and MRM transitions available on request
- Mass chromatograms shown opposite for analyte suite

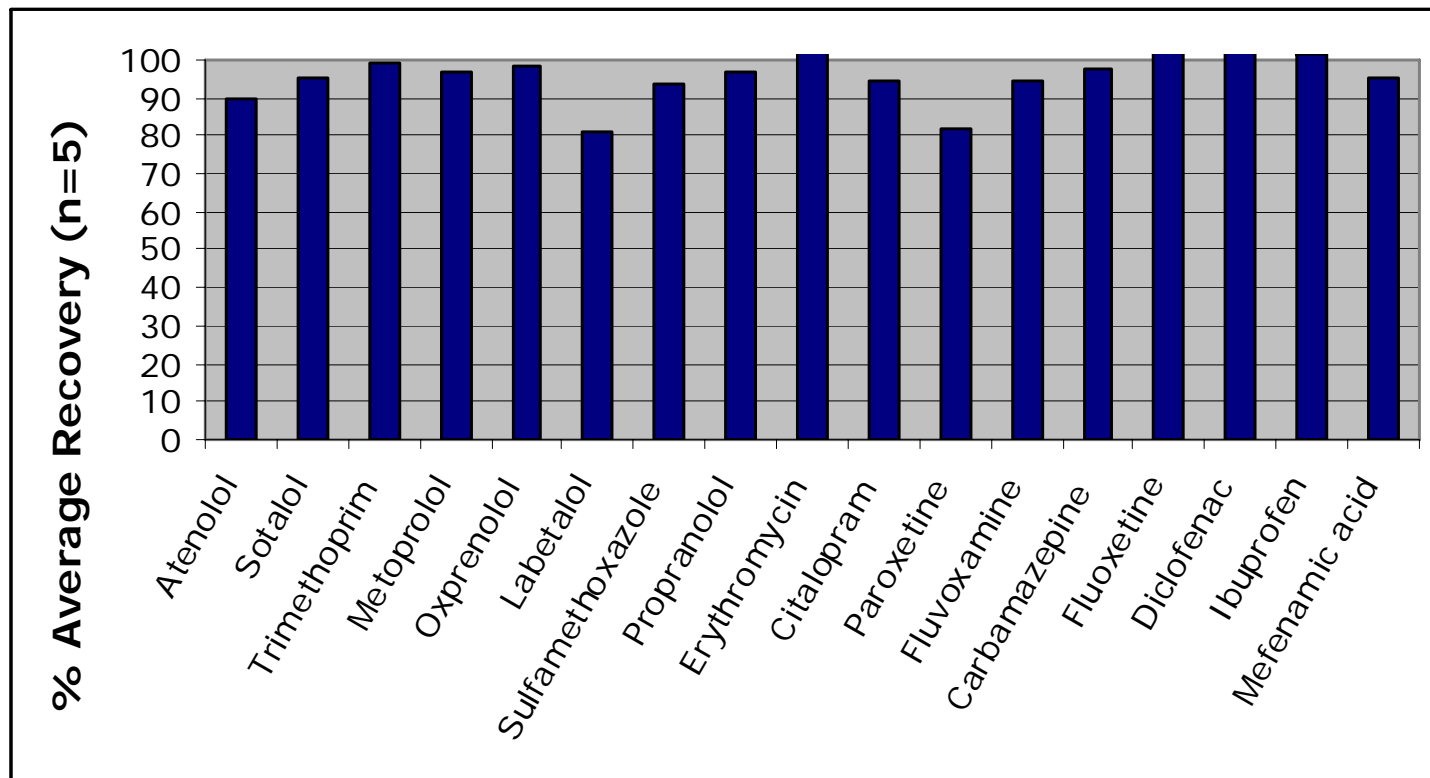


# Results: Analyte Recoveries

Analyte	Analyte Recovery (%)	% RSD (n=5)
Atenolol	90.1	3.5
Sotalol	95.3	2.2
Trimethoprim	99.3	2.9
Metoprolol	97.0	3.4
Oxprenolol	98.7	2.7
Labetalol	81.1	3.2
Sulfamethoxazole	94.1	3.9
Propranolol	97.0	2.3
Erythromycin	102.1	8.7
Citalopram	94.2	2.6
Paroxetine	82.2	5.2
Fluvoxamine	94.7	8.8
Carbamazepine	97.8	1.5
Fluoxetine	103.8	6.3
Diclofenac	103.2	2.8
Ibuprofen	101.9	7.8
Mefenamic Acid	95.3	8.5

# Analyte Recoveries: Summary

- Capable of greater than 80% recovery of a wide range of analytes
  - Wide ranging polarity
  - Acidic, neutral and basic analytes
    - Example: Extraction of pharmaceuticals from water



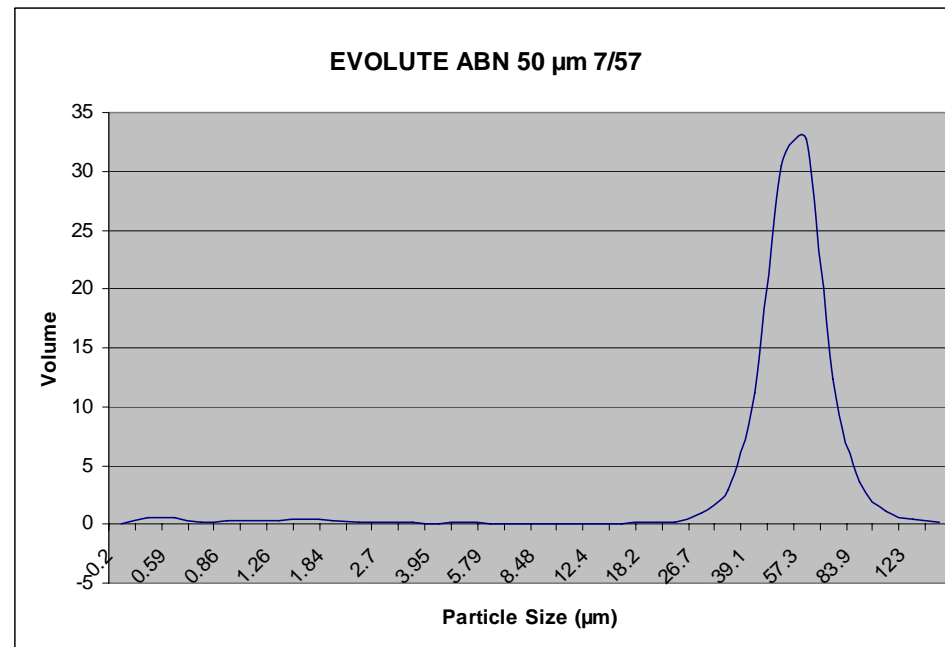
# Optimized for Robust Operation

EVOLUTE ABN's well controlled particle size distribution and optimized packing techniques ensure

- Consistent flow rates
  - Particularly important for large volume samples
- High reproducible analyte recoveries
- Maximum performance with automated liquid handling systems

# Fines Free Sorbent

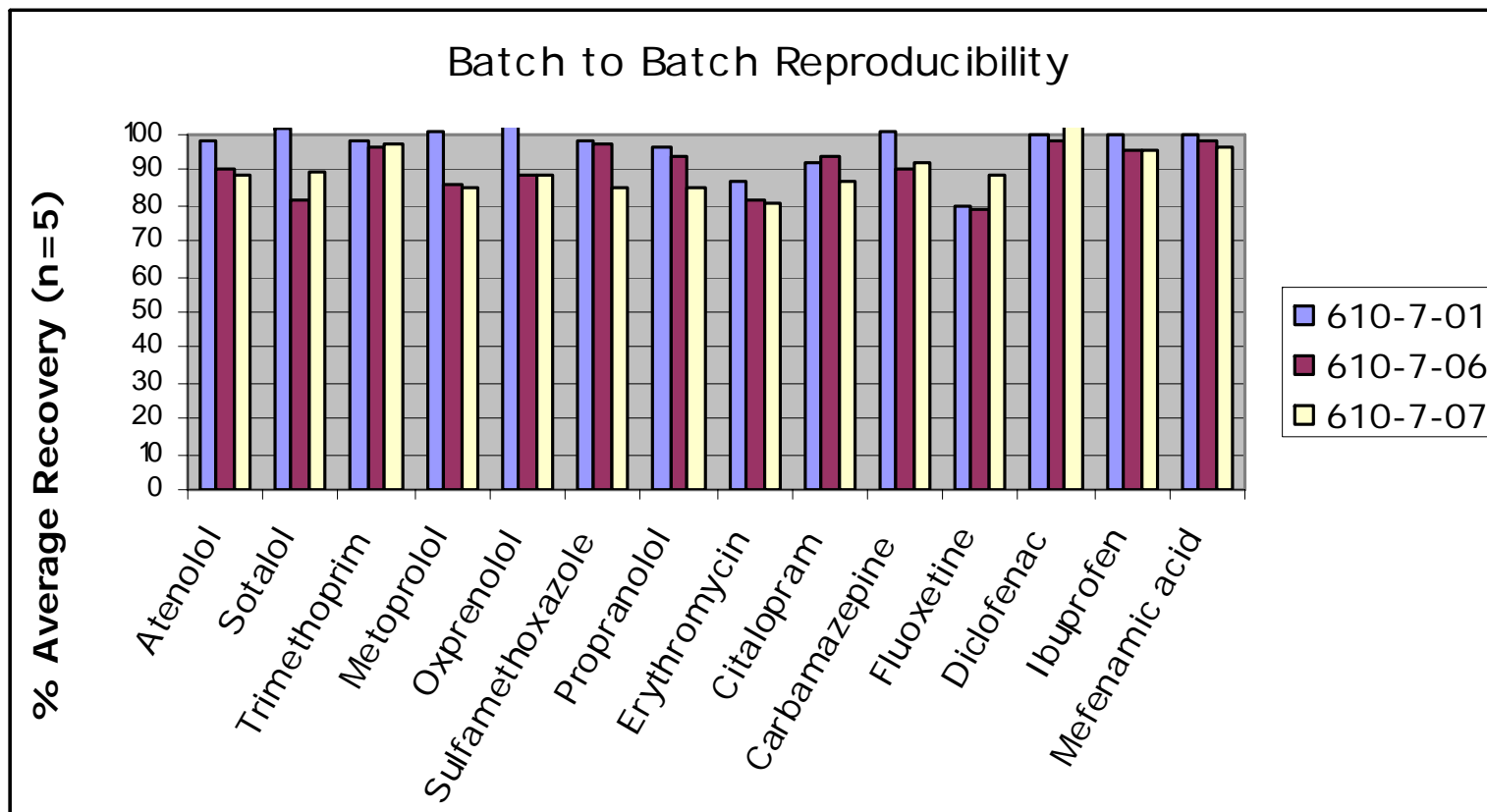
- No contamination of extract or automation & mass spec transfer lines
- Efficient SPE
  - Capacity and each step of the SPE procedure
- Consistent flow characteristics



# Reproducible Analyte Recoveries

Pharmaceuticals from water

- 200 mg/6 mL column made from three different batches of EVOLUTE ABN 50  $\mu\text{m}$
- Recoveries greater than 80%, RSDs < 10%, n=5



# EVOLUTE ABN Formats and Configurations



- 25 mg/10 mL
- 50 mg/3 mL
- 100 mg/3 mL
- 100 mg/10 mL
- 200 mg/3 mL
- 200 mg/6 mL

# Accessories

- VacMaster LVE kit
  - Processing large volume samples
  - Attach to SPE column via adaptor
- VacMaster Drying Adaptor
  - Remove water from the SPE column prior to elution with a water immiscible solvent
- PTFE Extraction Caps
  - Attach sample bottle directly onto the SPE column

All ordering information in  
Analytical Sample Preparation Catalogue

# Summary

- EVOLUTE ABN provides high and reproducible analyte recovery for a wide range of acidic, basic and neutral compounds from aqueous samples
  - Water (river, surface, drinking etc.)
  - Soil extracts
- Ideal for the extraction of multiple analyte suites in one extraction
  - Pharmaceuticals
  - Pesticide residues

Thanks to:  
Environment Agency, UK  
Biotage R&D Group, UK

Thank you for your attention!

# HPLC Conditions

**Instrument:** Waters Alliance 2795 Separations Module.

**Column:** Zorbax Eclipse XDB-C18.  
100 x 2.1 mm, 3.5  $\mu$ m) Agilent

**Guard Column:** Zorbax Eclipse XDB-C8.  
(12.5 x 2.1 mm, 5  $\mu$ m) Agilent

**Injection Volume:** 10  $\mu$ L

**Flow Rate:** 0.25 mL/min. Entire column effluent directed into the MS

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

# Mass Spectrometry Conditions

**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<sup>-3</sup> mbar

# Mass Spectrometry Conditions

## MRM Transitions

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
Thioridazine	371.1 > 126.1	22
Tamoxifen	372.2 > 72.1	21
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*