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Nota applicativa

Advil Allergy Sinus Tablet: Oasis MCX

Waters Corporation

Abstract

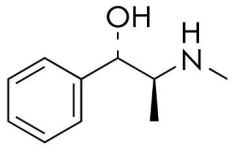
This application note highlights analysis of advil allergy sinus tablet usig Oasis MCX.

Introduction

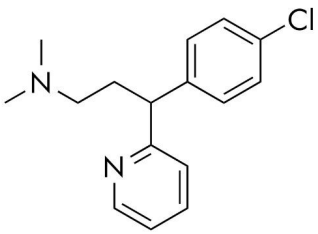
This formulation is a good example of a mixture of bases and an acid. The Oasis 2x4 Method was employed and the Oasis MCX plates resulted in the best recoveries for all three analytes.

The compounds used in this study are -

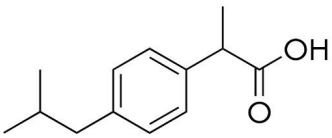
- Pseudoephedrine
- Chlorpheniramine
- Ibuprofen



Pseudoephedrine (Base)
MW 165.2
pKa 9.9



Chlorpheniramine (Base)
MW 274.8
pKa 9.2



Ibuprofen (Acid)
MW 206.3
pKa 5.2

Experimental

Test Conditions

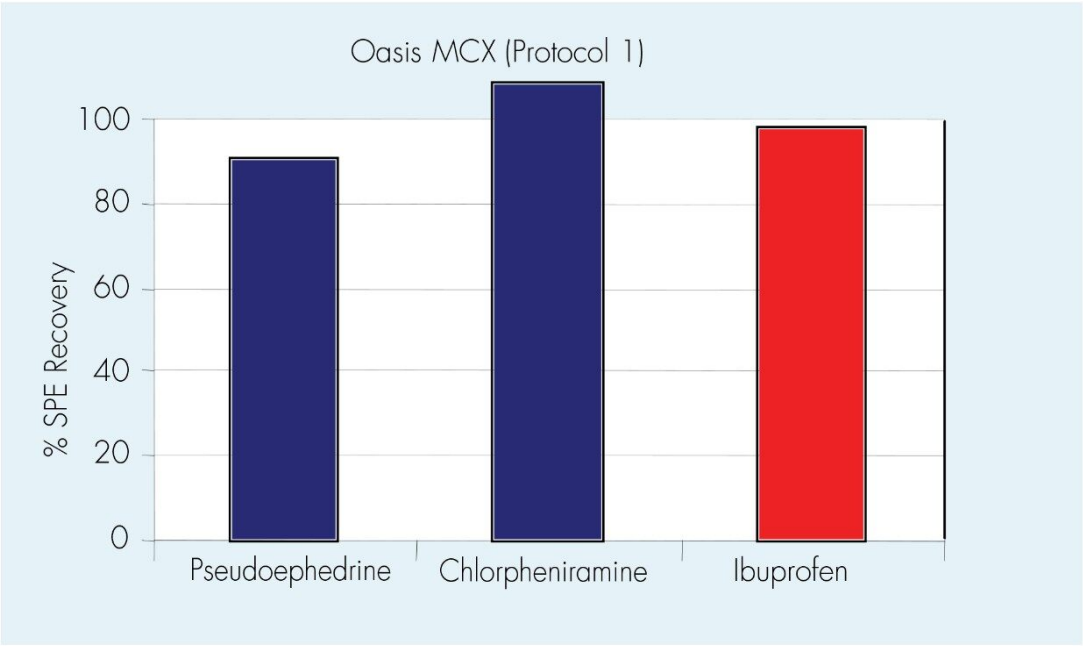
Oasis MCX 10-mg 96-well Plates

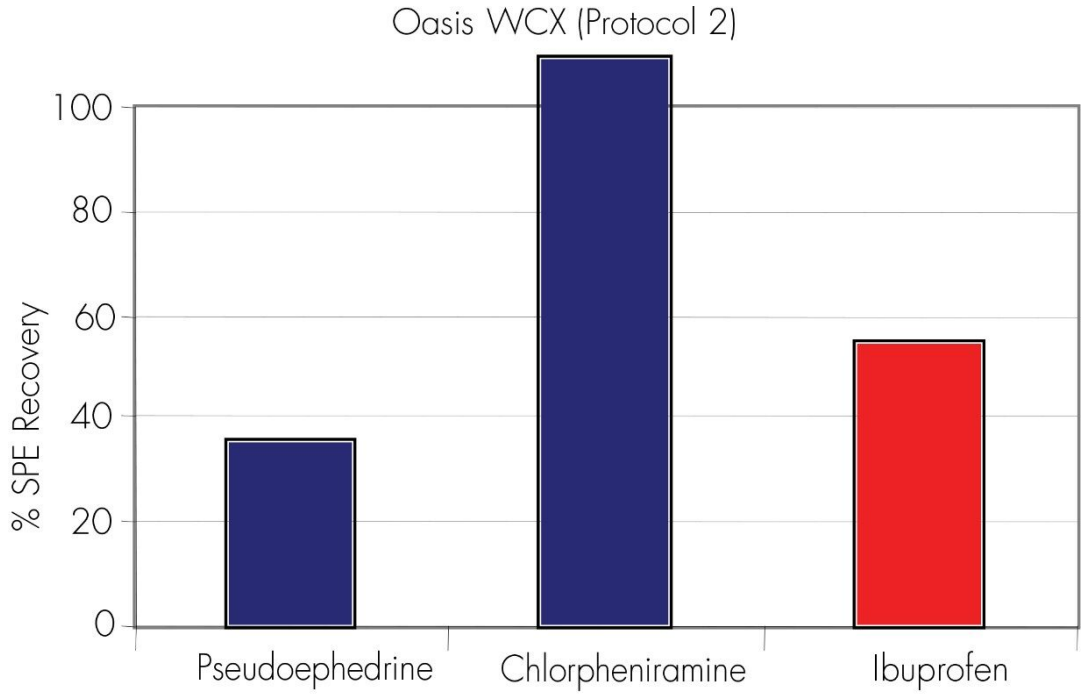
Condition:	500 µL MeOH
Equilibrate:	500 µL H_2O
Load:	500 µL sample (250 µL plasma diluted 1:1 with 4% H_3PO_4 in H_2O)
Wash 1:	500 µL 2% FA
Elute 1:	2 x 125 µL MeOH (Ibuprofen)
Elute 2:	2 x 125 µL 5% NH_4OH in MeOH (Bases)
Options:	1. Dilute Elute 2 with 250 µL 2% FA in water and Elute 1 with 250 µL 100% water and analyze separately. 2. Combine the two elutions and evaporate/reconstitute.
Pseudoephedrine HCl (Base):	1.5 µg/mL
Chlorpheniramine Maleate (Base):	0.1 µg/mL
Ibuprofen (Acid):	10 µg/mL
Column:	ACQUITY UPLC BEH C_{18}, 2.1 x 50 mm, 1.7 µm
Mobile phase A:	0.1% HCOOH in H_2O
Mobile phase B:	0.1% HCOOH in MeOH
Flow rate:	0.3 mL/min

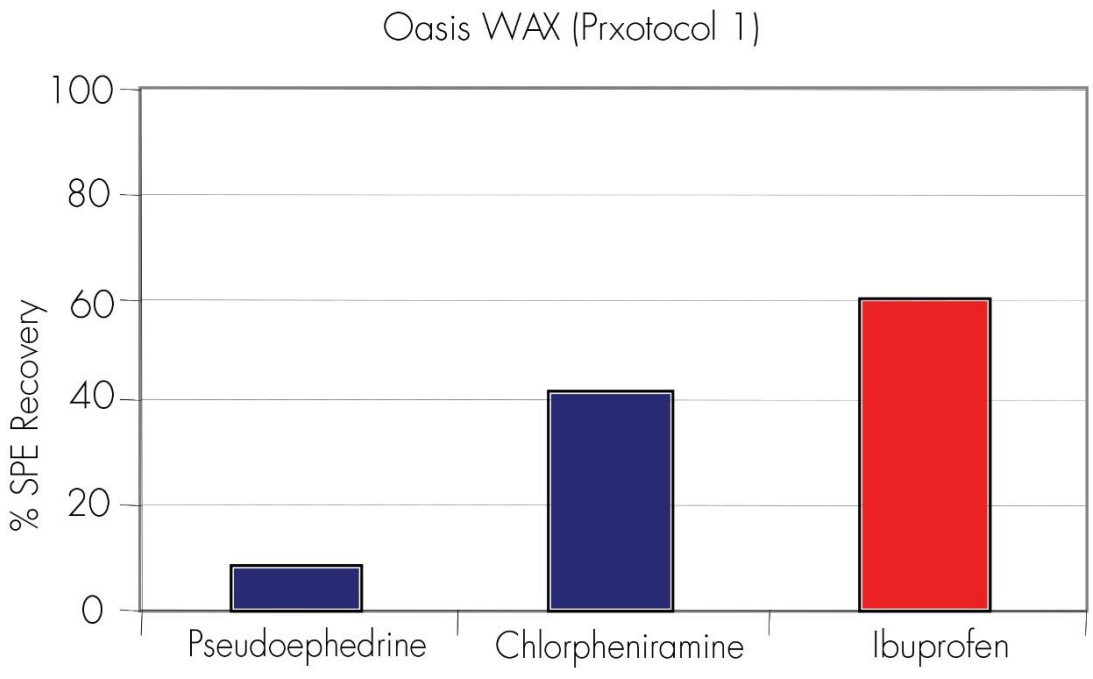
Oasis MCX 10-mg 96-well Plates

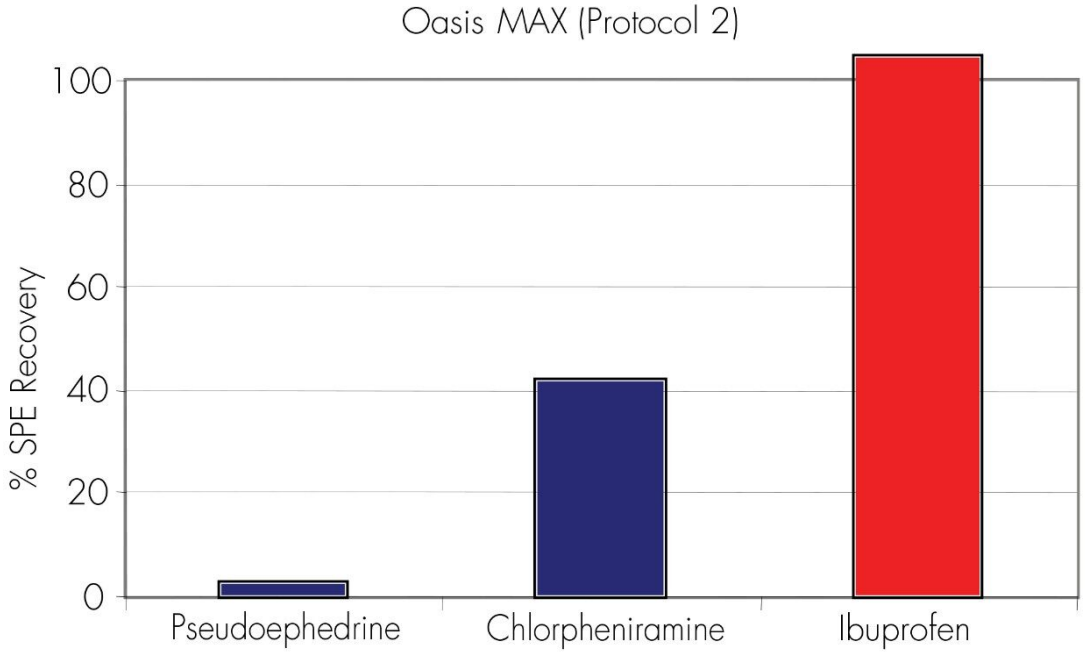
Injection volume:	10.0 µL
Column temp:	4 °C
Sample temp:	10 °C
Instrument:	ACQUITY UPLC with Quattro Premier

SPE Recovery Data: Optimun Sorbent and Protocol




Oasis WCX (Protocol 2)
100
80
60
40
20
0
% SPE Recovery
Pseudoephedrine
Chlorpheniramine
Ibuprofen


Oasis WAX (Prxotocol 1)
100
80
60
40
20
0
% SPE Recovery
Pseudoephedrine
Chlorpheniramine
Ibuprofen


Oasis MAX (Protocol 2)
% SPE Recovery
100
80
60
40
20
0
Pseudoephedrine
Chlorpheniramine
Ibuprofen

Gradient

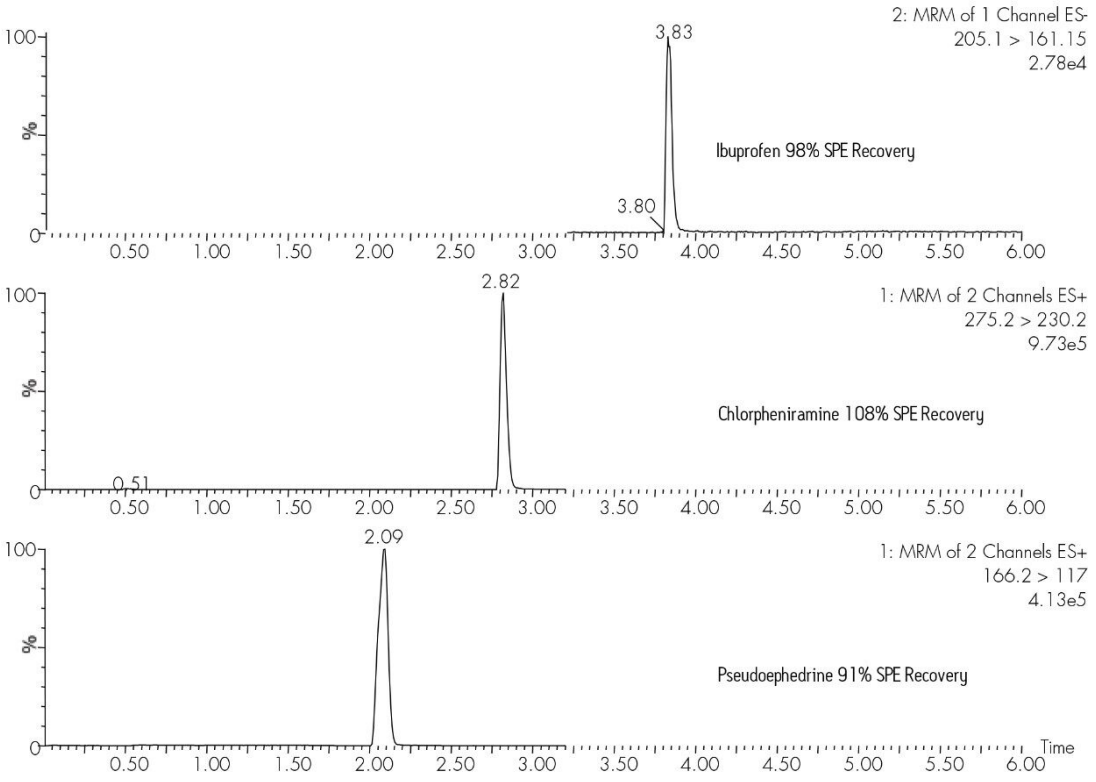
Time (min)	Profile	
	%A	%B
0.0	95	5
1.0	95	5
3.0	20	80
4.0	20	80
4.5	95	5
6.0	95	5

Quattro Premier

ESI^+ and ESI^- capillary:	3.0 kV
Source temp:	100 °C
Desolvation temp:	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-)	205.1	161.1	20	12
Pseudoephedrine (ESI+)	166.2	117	30	20
Clorpheniramine (ESI+)	275.2	232.2	30	20

Results and Discussion



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WA60091, June 2007