

Nota applicativa

Pharmaceutical Compounds in Plasma

Waters Corporation



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

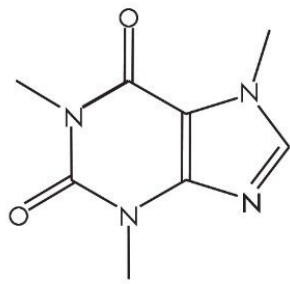
Abstract

This application brief demonstrates analysis of pharmaceutical compounds in plasma.

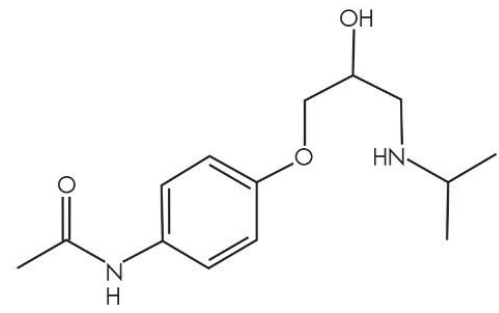
Introduction

The compounds used in this study are –

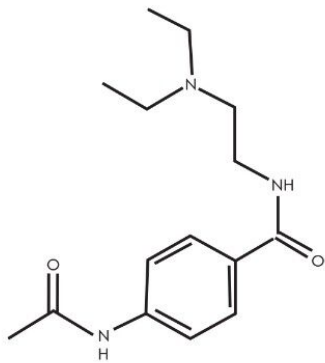
1. Caffeine
2. Practolol
3. N-acetyl procainamide
4. Propranolol
5. Methoxyverapamil
6. Amitriptyline



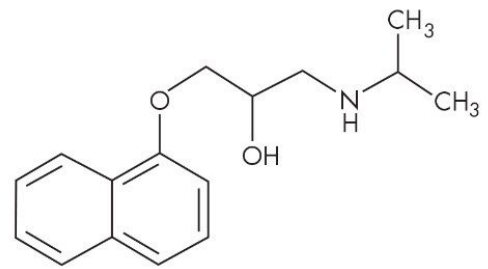
Caffeine



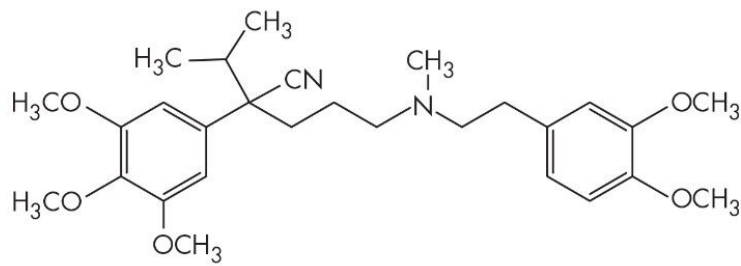
Practolol



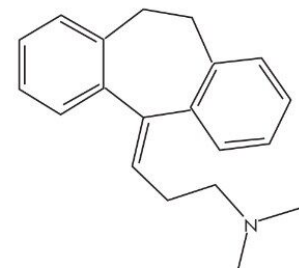
N-acetyl procainamide



Propranolol



Methoxyverapamil



Amitriptyline

Experimental

LC Conditions

Column: Xterra MS C₁₈, 2.1 x 20 mm IS, 3.5 μm, (p/n: 186001923)

Mobile phase A: 10 mM NH₄HCO₃, pH 10

Mobile phase B: Methanol

Flow rate: 0.4 mL/min

Injection volume: 20 μL

Sample concentration: 5 μg/mL

Temperature: Ambient

Detection: MS

Instrument: Waters 277 Sample Manager, Waters 1525 Binary HPLC Pump and Waters Micromass Quattro Ultima

Gradient

Time (min)	Profile	
	%A	%B
0.0	100	0
5.0	5	95

MS Conditions

Quattro Ultima

ES ⁺ MRM cone (V):	5.0
Capillary:	3.5
Source temp. (°C):	150
Desolvation temp. (°C):	400
Cone gas flow (L/Hr):	50
Desolvation gas flow(L/Hr):	550
LM resolution 1 & 2:	13.5
HM resolution 1 & 2:	13.5
Ion energy 1:	0.4
Ion energy 2:	0.8
Multiplier (V):	650

Sample Preparation

SPE:	Oasis HLB μ Elution Plate, (p/n:186001828BA) p/n:186001828BA
Condition:	200 μ L Methanol
Equilibrate:	200 μ L Water
Load:	150 μ L Spiked rat plasma with 2% H ₃ PO ₄ , diluted with 1:1 Water

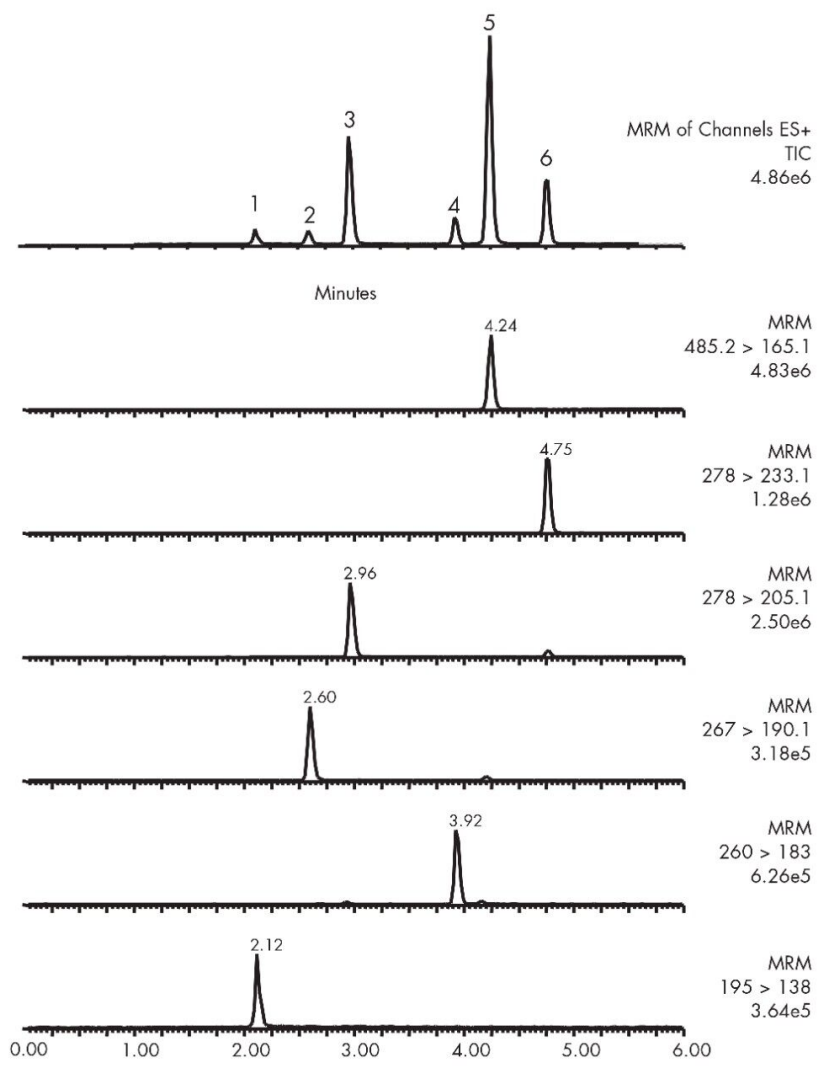
Wash:	200 μ L 5% Methanol in Water
Elute:	50 μ L Methanol
Dilute:	100 μ L Water

Results and Discussion

Compounds (MRM Transitions, Cone voltage (V), CID (eV))

1. Caffeine (195 > 138; 20; 20)
2. Practolol (267 > 190.1; 30; 20)
3. N-acetyl procainamide (278 > 205.1; 25;20)
4. Propranolol (260 > 183; 25;18)
5. Methoxyverapamil (485.2 > 165.1; 45; 30)
6. Amitriptyline (278 > 233.1; 25; 17)

The top figure is the total ion current, followed by the extracted ion signals for each of the three analytes.



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