Waters™

Application Note

Terfenadine and Metabolites by Reverse-Phase SPE and LC-MS/MS

Waters Corporation

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

Abstract

This application brief demonstrates analysis of terfenadine and metabolites by reverse-phase SPE and LC-MS/MS.

Introduction

The compounds analyzed in this study are terfenadine and metabolites.

Experimental

HPLC Method

Column:	Xterra MS C_{18}, 2.1 x 30 mm, 3.5 µm
Part number:	186000398
Mobile phase A:	Water + 0.1M NH_4 formate pH 9.5
Mobile phase B:	MeOH + 0.1M NH_4 formate pH 9.5
Flow rate:	0.4 mL/min
Temperature:	Ambient
LC instrument:	Alliance 2795

Gradient

Time (min)	Profile	
	%A	%B
0	95	5
1	5	95

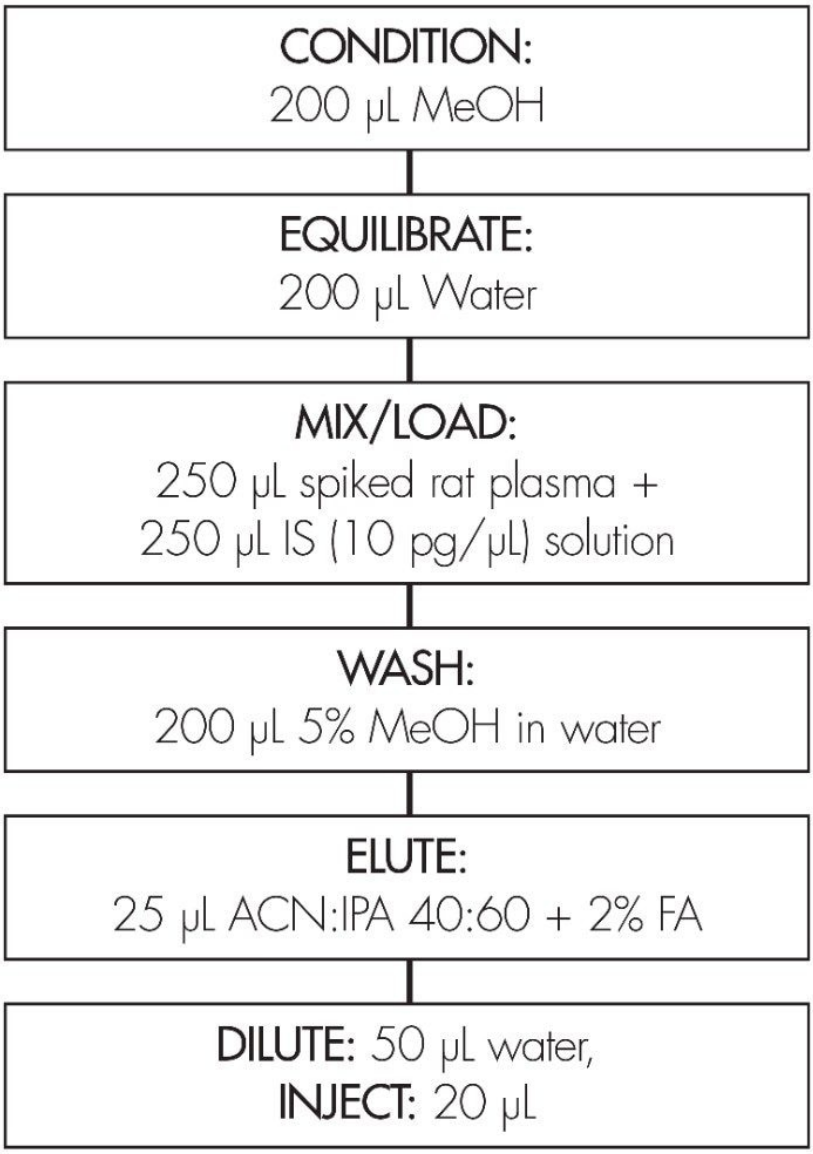
MS Conditions

MS instrument:	Waters Micromass Quattro
Ion source:	ESI+
Source temperature:	150 °C

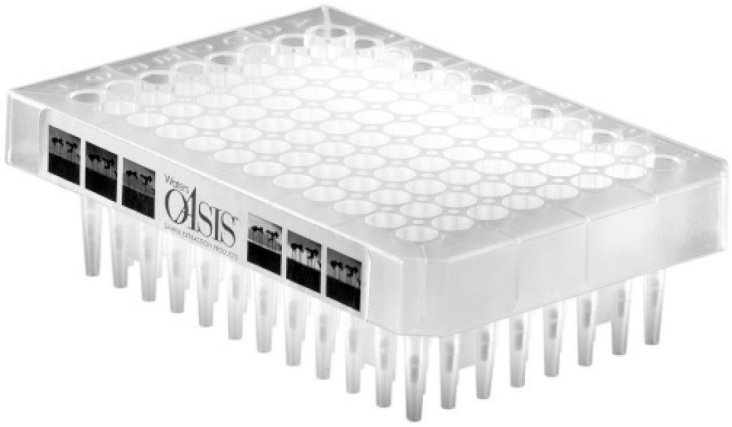
Gas cell:	$2.0e^{-3}$ bar Argon
Cone voltage:	35 volts
Capillary voltage:	3.5 kV
Drying gas flow:	500 L/hr
Cone gas flow:	50 L/hr
Desolvation temperature:	350 °C
MRM transition:	Protriptyline (*IS*) *m/z* 263.9 → 190.8
	Terfenadine *m/z* 472.2 → 436.2
	Terfenadine-alcohol *m/z* 488.2 → 452.2
	Terfenadine-carboxylate *m/z* 502.2 → 466.2

OASIS® HLB GENERIC EXTRACTION PROTOCOL

Conditions for Oasis® HLB µElution Plate
Part Number 186001828BA



NEW! OASIS® µELUTION PLATE

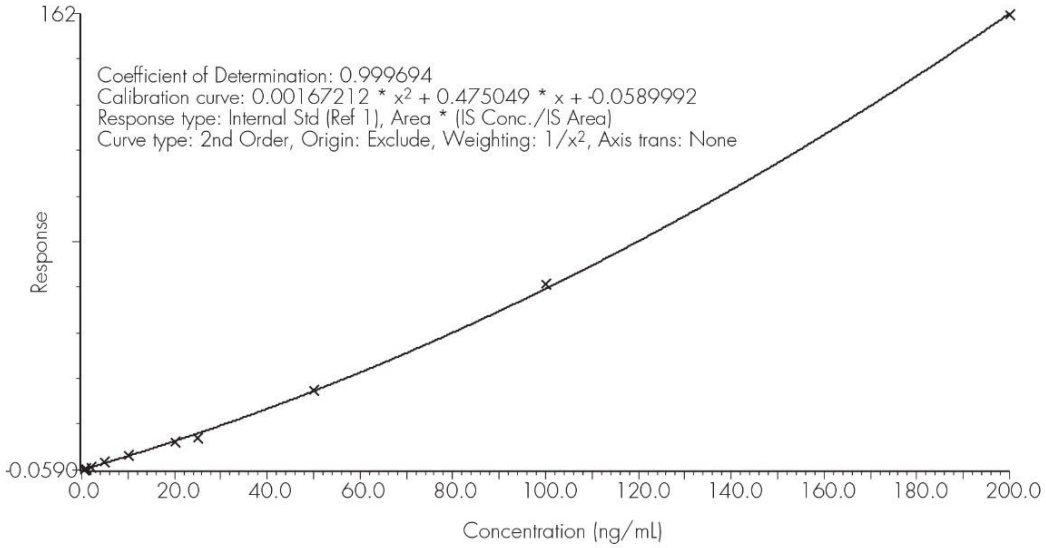


Results and Discussion

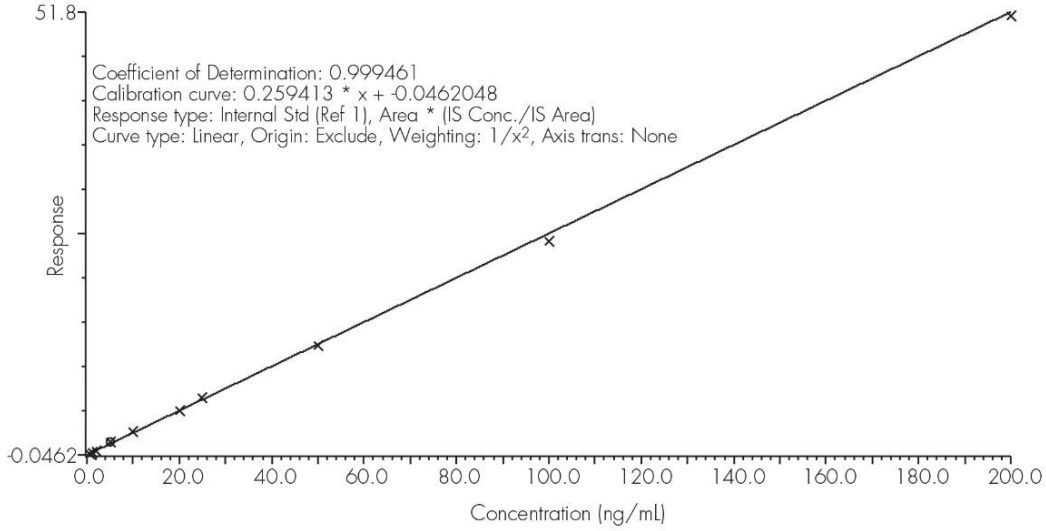
Method Validation of Terfenadine and Metobolites on Oasis HLB µElution Plate

Conc. (ng/mL)	0.5	1.0	5	10	20	100	200
TERFENADINE							
Day 1 (N=6) (C.V.)	0.50 (2)	0.96 (5)	4.99 (5)	10.11 (5)	19.26 (2)	103.25 (4)	197.18 (1)
Day 2 (N=6)	0.51 (2)	0.92 (3)	4.96 (2)	10.05 (4)	19.76 (5)	103.80 (5)	196.50 (1)
Day 3 (N=6)	0.50 (2)	0.97 (6)	5.00 (6)	9.87 (6)	19.37 (5)	106.73 (3)	195.23 (1)
Day 4 (N=6)	0.50 (2)	0.90 (5)	5.24 (5)	9.49 (2)	19.49 (6)	106.89 (2)	194.62 (1)
TERFENADINE-CARBOXYLATE							
Day 1 (N=6) (C.V.)	0.49 (2)	1.02 (5)	5.05 (5)	10.12 (5)	19.71 (6)	101.69 (3)	196.39 (2)
Day 2 (N=6)	0.51 (1)	0.94 (3)	4.82 (3)	10.08 (5)	20.26 (4)	99.98 (5)	202.19 (4)
Day 3 (N=6)	0.50 (2)	0.97 (5)	5.04 (5)	9.89 (4)	20.53 (6)	98.50 (4)	196.57 (4)
Day 4 (N=6)	0.50 (3)	0.99 (3)	5.07 (3)	9.86 (6)	20.14 (3)	99.03 (5)	199.20 (3)
TERFENADINE-ALCOHOL							
Day 1 (N=6) (C.V.)	0.51 (2)	0.97 (4)	5.19 (5)	10.24 (4)	19.81 (4)	102.05 (3)	196.55 (4)
Day 2 (N=6)	0.51 (1)	0.93 (2)	5.06 (2)	10.27 (4)	20.40 (3)	99.69 (6)	199.17 (5)
Day 3 (N=6)	0.51 (1)	0.94 (3)	4.77 (2)	10.02 (5)	20.07 (5)	99.73 (5)	198.60 (2)
Day 4 (N=6)	0.51 (2)	0.92 (2)	5.19 (3)	9.78 (4)	20.26 (4)	101.14 (5)	193.32 (6)

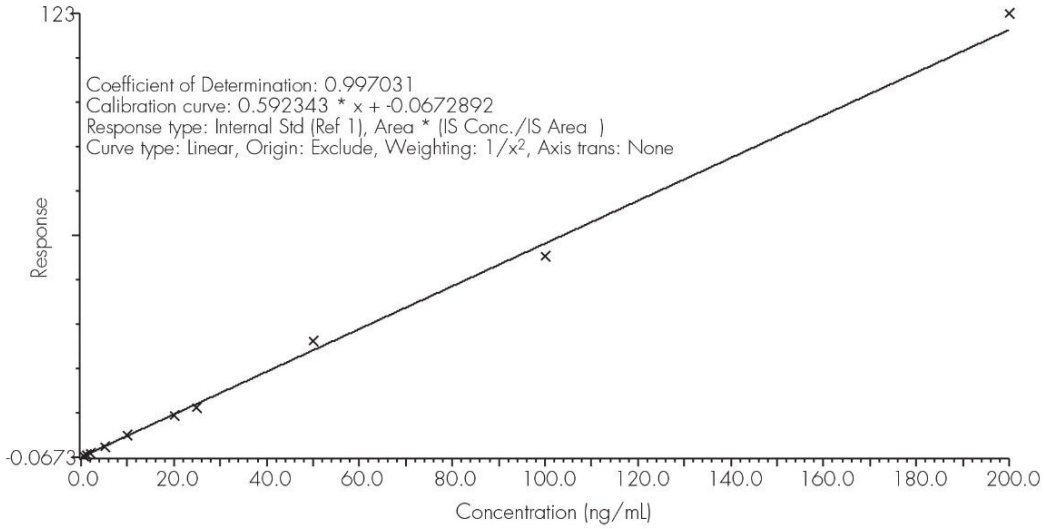
CALIBRATION CURVE OF TERFENADINE ON OASIS® HLB µELUTION PLATE



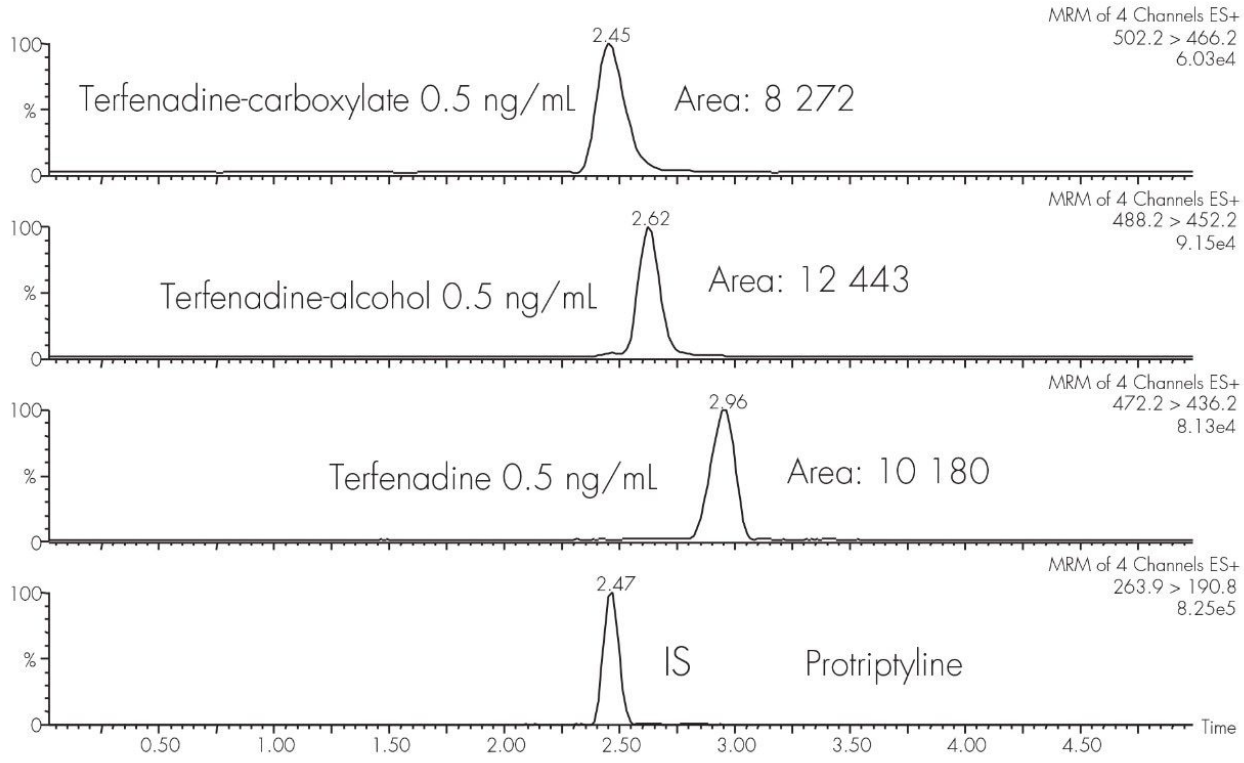
CALIBRATION CURVE OF TERFENADINE-CARBOXYLATE ON OASIS® HLB µELUTION PLATE



CALIBRATION CURVE OF TERFENADINE-ALCOHOL ON OASIS® HLB µELUTION PLATE



HPLC- MS/MS ANALYSIS



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