

Nota applicativa

Spiramycin in Pork

Waters Corporation



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

Abstract

This method can be used to monitor Spiramycin, a macrolide antibiotic, in pork.

Introduction

World health organizations are concerned about the antibiotics and antibacterials levels in food. This method can be used to monitor Spiramycin, a macrolide antibiotic, in pork.

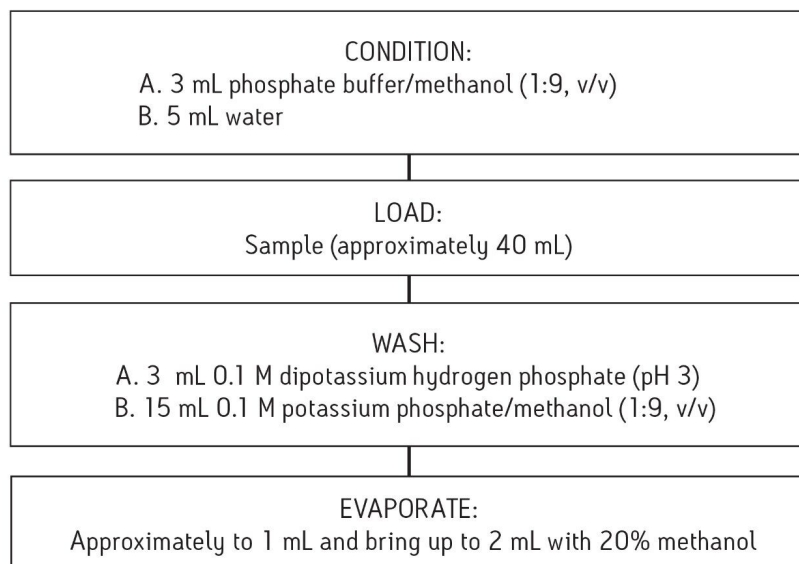
Experimental

Pretreatment

1. Weigh 5 g of sample into 25 mL of water. Homogenize.
2. Centrifuge at 3000 rpm for 10 minutes.
3. Collect supernatant.
4. Re-extract pellet with 15 mL 1.2% metaphosphoric acid/ methanol (50:50, v/v).
5. Vortex.
6. Centrifuge at 3000 rpm for 10 minutes. Collect supernatant.
7. Combine both supernatants.
8. Filter sample using glass fiber filter.

SPE Procedure

Oasis® MCX 6 cc/150 mg



SPE Buffer:

Dissolve 8.7 g dipotassium hydrogen phosphate in 1 L of deionized water, adjust to pH 3 with phosphoric acid.

LC Conditions

System:	ACQUITY UPLC
Column:	ACQUITY UPLC BEH C ₁₈ , 1.7 µm, 2.1 x 50 mm
Flow rate:	400 µL/min
Mobile phase A:	0.1% formic acid in water
Mobile phase B:	0.1% formic acid in acetonitrile

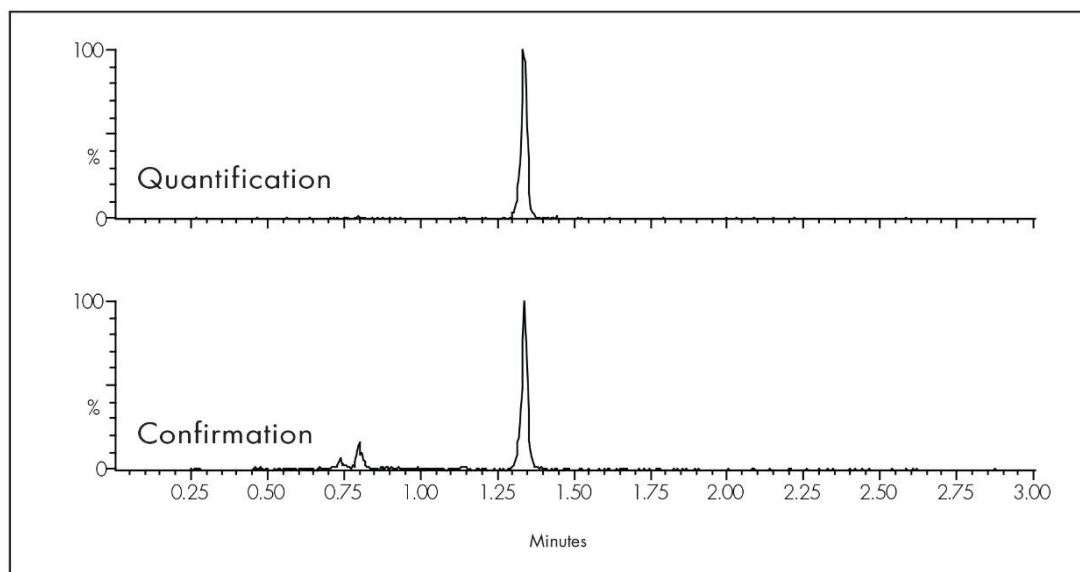
Gradient

Time (min)	%A	%B
0	90	10
2	60	40
2.1	90	10
3	90	10

MS Conditions

MS System:	Waters Quattro Premier XE
MRM transitions:	1: 422.50→101.00 2: 422.50→174.10
Ionization mode:	Positive electrospray (ESI ⁺) Multiple reaction monitoring

Results and Discussion



Representative chromatogram 100 ppb spiramycin spiked sample in pork muscle.

Compound Name: Spiramycin I 174.1	RT	Area
100 ppb spiked 1	1.34	902.79
100 ppb spiked 2	1.34	911.36
100 ppb spiked 3	1.34	1041.24
100 ppb spiked 4	1.34	1030.33
100 ppb spiked 5	1.34	1101.27
RSD (%)		7.97
Recovery (%)		73.26

Compound Name: Spiramycin I _101	RT	Area
100 ppb spiked 1	1.34	1063.65
100 ppb spiked 2	1.34	1267.22
100 ppb spiked 3	1.34	1368.84
100 ppb spiked 4	1.34	1227.10
100 ppb spiked 5	1.34	1379.40
RSD (%)		5.96
Recovery (%)		74.82

Recovery results for 100 ppb spiramycin spiked sample in pork muscle.

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ACQUITY UPLC System <<https://www.waters.com/514207>>

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