

Application Note

Acidic Veterinary Drugs in Horse Urine by LC-MS

Waters Corporation

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

Abstract

This application brief demonstrates the analysis of acidic veterinary drugs in horse urine by LC-MS.

Introduction

The compounds analyzed in this study are:

1. Ketoprofen
 2. Naproxen
 3. Phenylbutazone
 4. Ibuprofen
 5. Meclofenamic acid
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Experimental

LC-MS Method

Column:	XTerra MS C ₁₈ , 2.1 x 100 mm, 3.5 µm
Part number:	186000404
Mobile phase A:	20 mM ammonium acetate (pH 4)
Mobile phase B:	Methanol
Gradient:	50% A initial, to 90% methanol in 10 min
Flow rate:	175 mL/min
Injection volume:	10 µL
Instrument:	Waters Alliance Separations Module with 996 PDA

Compound	0.1 ppm	0.4 ppm	2.0 ppm	10.0 ppm	r ²
ketoprofen	4804(4.7)	26389(11)	129366(9.6)	525903(8.1)	0.998
naproxen	7621(8.8)	40234(23)	231980(12)	875595(7.3)	0.995
phenylbutazone	778(7.8)	4252(37)	39387(15)	207163(5.6)	0.999
ibuprofen	820(6.1)	3739(14)	23489(7.9)	127731(5.7)	0.999
meclofenamic acid	2070(11)	9531(23)	38822(11)	—	0.998

OASIS® METHOD

Oasis® MAX, 6 cc/150 mg Cartridge

Part Number 186000369

PREPARE SAMPLE:

Hydrolysis

- add 1 mL of 10M KOH to 10 mL of spiked urine.
- heat at 60° for 15 minutes.
- allow to cool to room temperature
- adjust to pH 2 with H₃PO₄.
- dilute 1:1 with reagent water

CONDITION:

3 mL each: MTBE/MeOH/H₂O

LOAD:

10 mL diluted urine onto Oasis® cartridge, 1-2 mL/min

WASH 1:

3 mL 50 mM NaOAc (pH 7)

WASH 2:

4 mL methanol

ELUTE:

4 mL MTBE/MeOH/TFA (89:10:1)

EVAPORATE AND RECONSTITUTE:

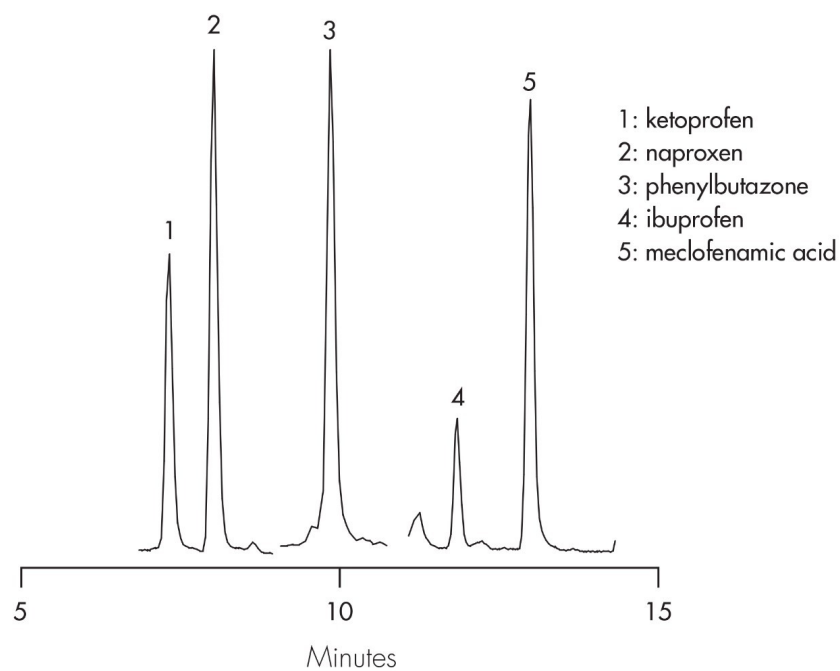
Nitrogen @ 45° C

MTBE - methyl t-butyl ether

TFA - trifluoroacetic acid

Results and Discussion

RECONSTRUCTED TIC CHROMATOGRAM



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