

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
-

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:

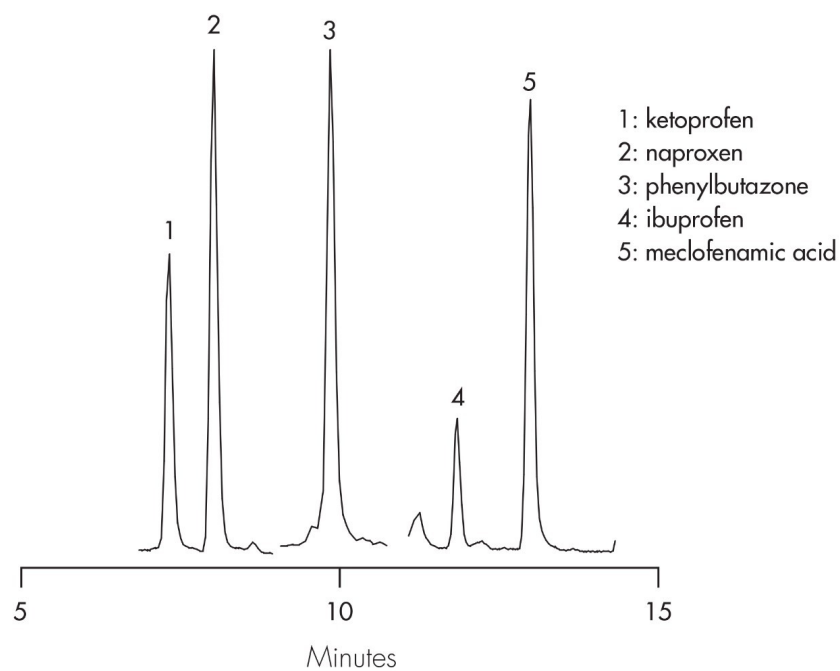
Nitrogen @ 45° C

MTBE - methyl t-butyl ether

TFA - trifluoroacetic acid

Results and Discussion

RECONSTRUCTED TIC CHROMATOGRAM



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