Product Information

YMC EUROPE GMBH

YMC-Triart C18 capillary column

MicroLC in veterinary drug residue analysis in food

Author: MO/DE

Date: 02.01.2014

Introduction

The levels and presence of veterinary drug residues in food of animal origin are legislated in the EU with limits often varying with the drug residue [1]. Traditionally screening of food samples involves extraction and analysis by LC/MS/MS usually at flow rates above of 500 µl/min using UHPLC systems.

Reduction of run times and process costs leads to higher efficiencies. Therefore, AB SCIEX developed a microLC method as a substitute for the established HPLC method [2]. Here, a YMC-Triart C18 capillary column (50 x 0.5 mm) was used.

Results

The results show a sensitivity increase of factors greater than 8-fold when switching from high to micro flow. Figure 1 shows the separation of three veterinary drugs which were analysed at high flow rates and compared to an optimised microLC method. The chromatograms clearly demonstrate an improvement in sensitivity when moving to micro LC which is not lost when the analysis time of 10 min is further shortened to a run time of 3.5 min to speed up the analysis (shortened run time gradient details: see table 2, page 3).

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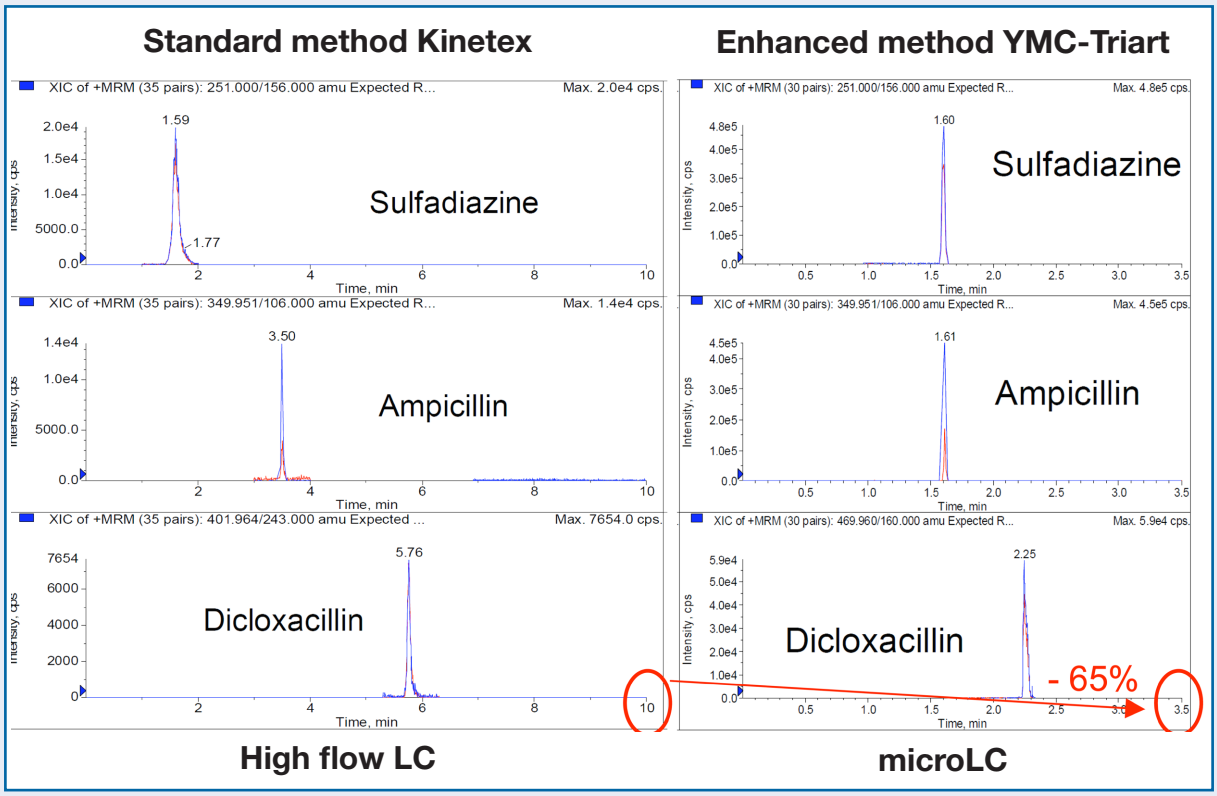

Figure 1: Comparison of 3 different 1 ppb standard solutions separated by a UHPLC method using a Kinetex C18 column and the microLC method using a YMC-Triart C18 capillary column.

Table 1: High flow and micro flow conditions

	UHPLC	microLC
Column	Phenomenex Kinetex XB-C18 (2.6 µm, 10 nm, 50 x 2.1 mm)	YMC-Triart C18 (3 µm, 12 nm, 50 x 0.5 mm)
HPLC	Shimadzu XR	Eksigent ekspert microLC 200
Eluent	A) Water (+ 0,1 % FAC) B) Acetonitrile (+ 0,1 % FAC)	
Flow rate	600 µl/min	30 µl/min
Temperature	60°C	
Detection	AB SCIEX 5500 QTRAP, ESI	
Injection	2 µL	

In the final analysis a total of 32 multiple reaction monitoring (MRM) transitions were evaluated for 15 veterinary drug residues over a 3.5 minute run time (but there is scope to add more residues to the same method), using milk and meat samples which have been spiked at a 10 ppb level with standard compounds (see Figure 2). The simple sample preparation process only involved solvent extraction and centrifugation.

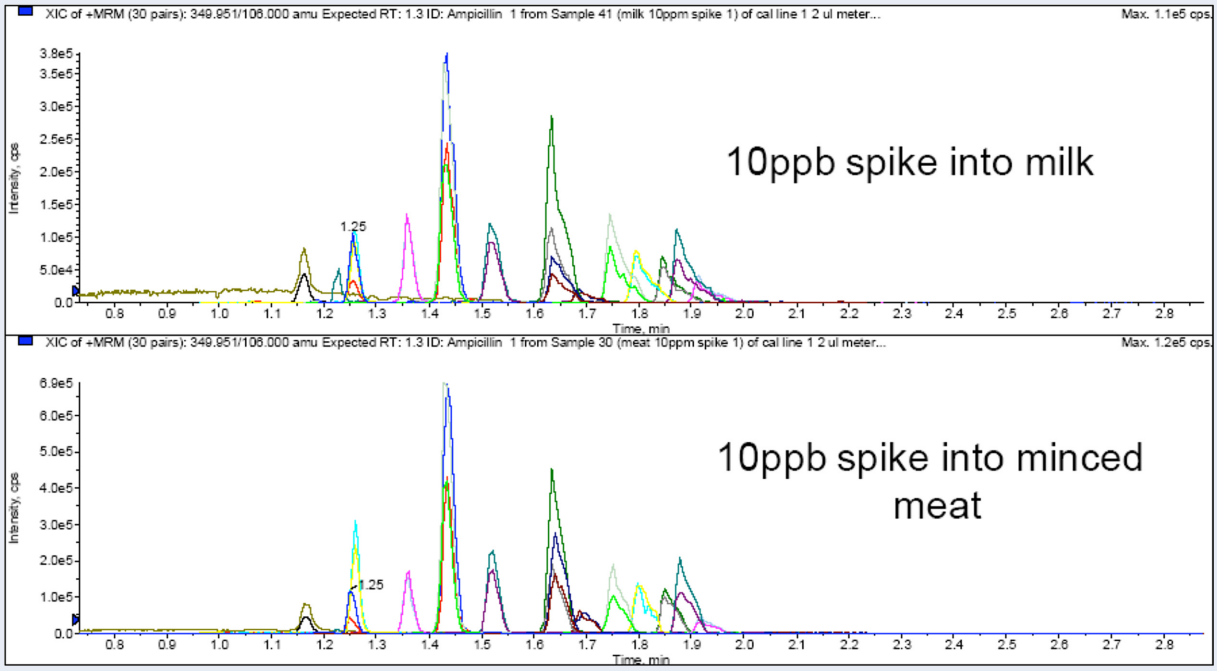

Figure 2: A comparison of a milk and meat samples which have been spiked at a 10 ppb level. The recoveries from meat were generally higher and it shows that recoveries are affected by the matrix.

Table 2: Shortened microLC-MS/MS gradient for YMC-Triart C18

min	A	B
0 - 0.5	98	2
1.7	35	65
1.8 - 2.3	0	100
2.4 - 3.5	98	2

Linearity and sensitivity of this method is demonstrated for Ampicillin in figure 3. Linearity is provided without use of any internal standards. The inset chromatogram for a 0.5 ppb spiked sample demonstrates the high level of sensitivity.

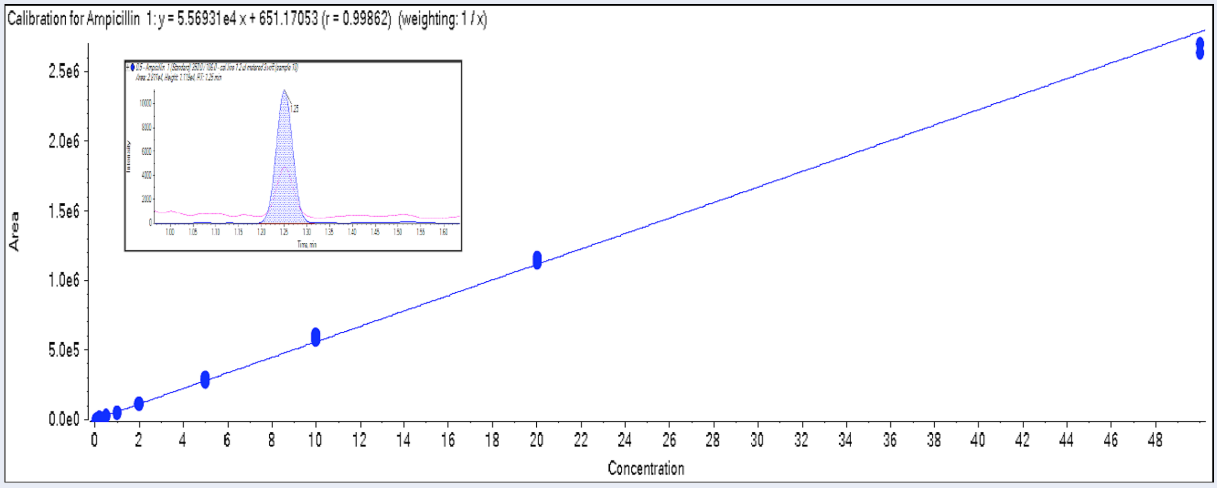

Figure 3: Calibration lines for Ampicillin from 0.05 – 50 ppb. The linearity is provided without the use of any internal standards. Inset in the diagram is the chromatogram of a 0.5 ppb standard.

Conclusions

This study of veterinary drug residues illustrates the potential of YMC-Triart C18 capillary columns for reduction of run times and significant savings in solvent costs.

The developed method easily fulfils the requirements of the current EU legislation with a gain in signal by a factor of more than 8 fold for some components. Furthermore, the cut in analyse time provides great potential of cost savings by up to 90% in regards to solvents

The microLC/MS/MS approach has the additional advantage of being a potential drug residue screen where different residues can be detected by a single method.

References

[1] Commission Regulation (EU) No 37/2010 of 22 December 2010 on pharmacologically active substances and their classification regarding maximum residue limits in foodstuffs of animal origin, 2010.

[2] Lock S, The use of Microflow UHPLC in veterinary drug residue analysis, Poster presented at the 127th AOAC International 2013 Annual Meeting & Exposition in Chicago, August 25 – August 28, 2013.

Trademark

YMC acknowledge the work of Stephen Lock, AB SCIEX, Warrington (UK) in producing this application data.