



Monitoring for nontargeted veterinary residues in food samples using quadrupole time-of-flight (Q-TOF) LC/MS

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Q-TOF LC-MS/MS

Potential Advantages for Residue Analysis

- Full scan data collection with accurate mass allows screening for virtually unlimited numbers of analytes.
- Both target and nontarget analytes are detected.
- Data can be evaluated retrospectively.
- Product ion spectra can be obtained for further characterization of analyte.

Q-TOF LC-MS/MS Method

Instrument:

Agilent 6530 Q-TOF coupled to Agilent 1290 LC
Electrospray (Agilent Jet Stream Technology)

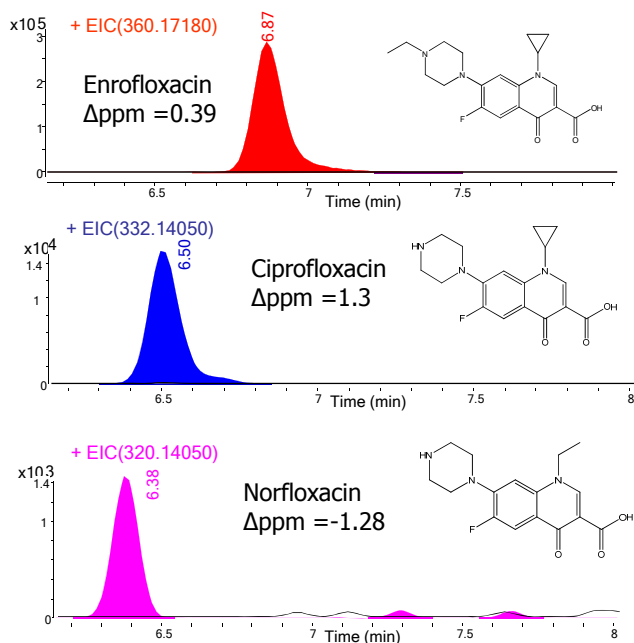
LC Parameters:

YMC ODS-AQ 2.0 x 100mm, 3.5 micron
0.1% formic acid/acetonitrile gradient, 250 μ L/min

MS Scanning Parameters:

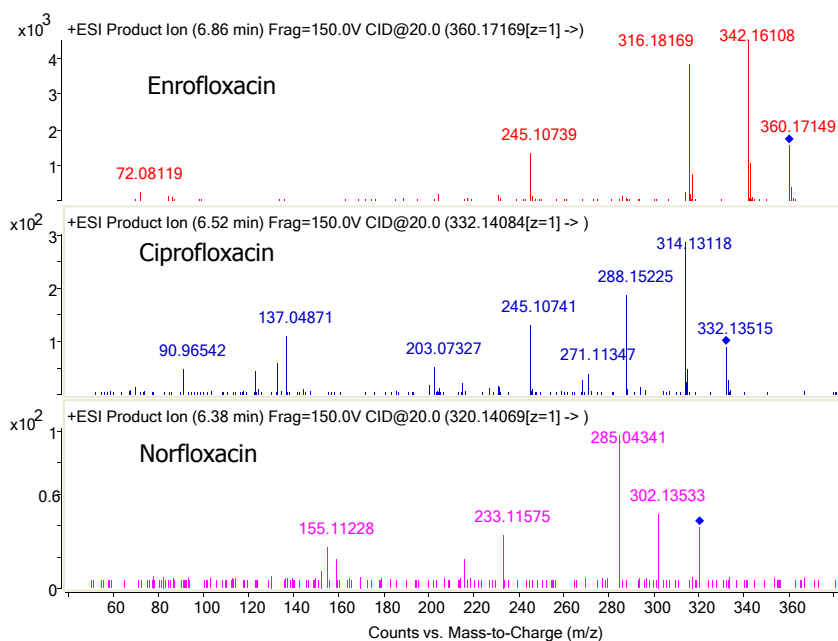
TOF: Scanned m/z 100-1200, high resolution mode (4GHz)
MS/MS: Targeted analysis, 200 ms/spec, narrow isolation width

Confirmation of Target Analytes in Frog Legs

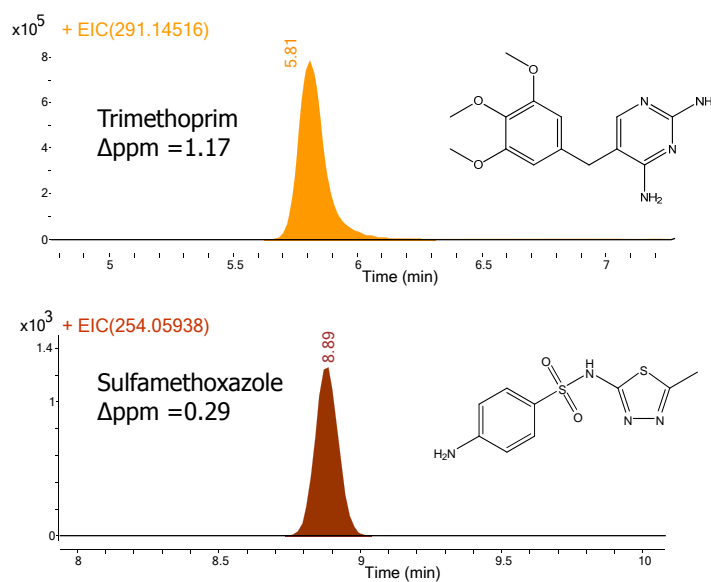


Extracted ion chromatograms (± 5 ppm) for fluoroquinolone residues in a frog legs sample. Residue levels were determined to be 32 ppb (ENR), 4 ppb (CIP) and \sim 0.5 ppb (NOR).

MS/MS Spectra of Target Analytes in Frog Legs Sample

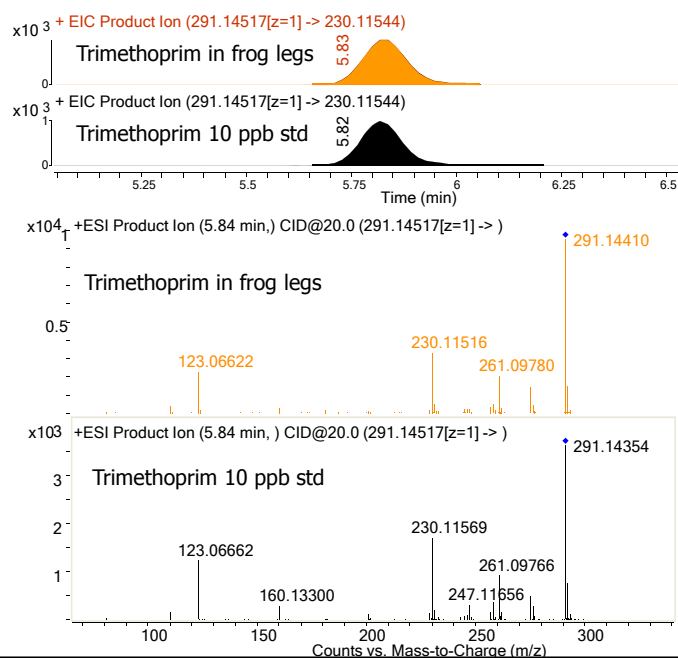


Identification of Nontarget Analytes in Frog Legs

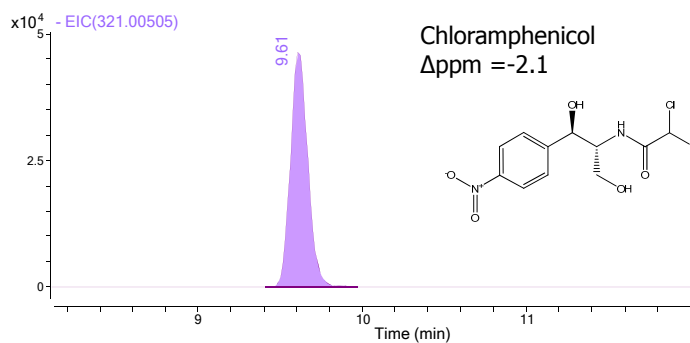


Results from search of MS data against library of veterinary drugs

MS/MS Spectra of Trimethoprim in Frog Legs Sample



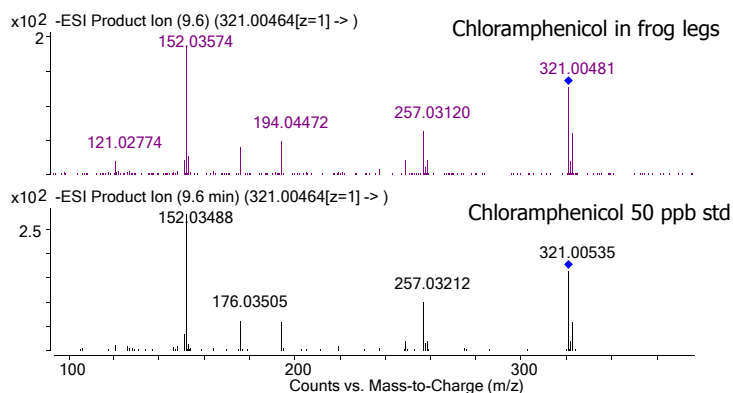
Nontarget Analytes in Frog Legs – Negative Ion



Comparing negative ion MS scan data from frog legs extract to the library, chloramphenicol was also identified.

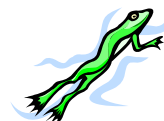
Concentration was estimated to be >50 ppb.
(action level for chloramphenicol is 0.3 ppb)

Nontarget Analytes in Frog Legs – With Negative Ion

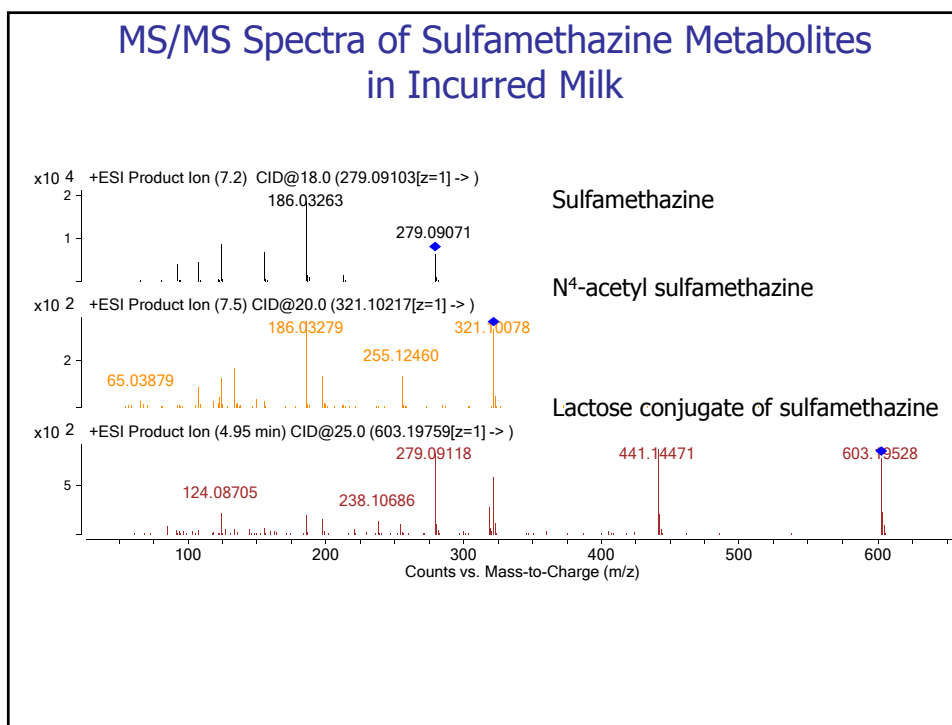
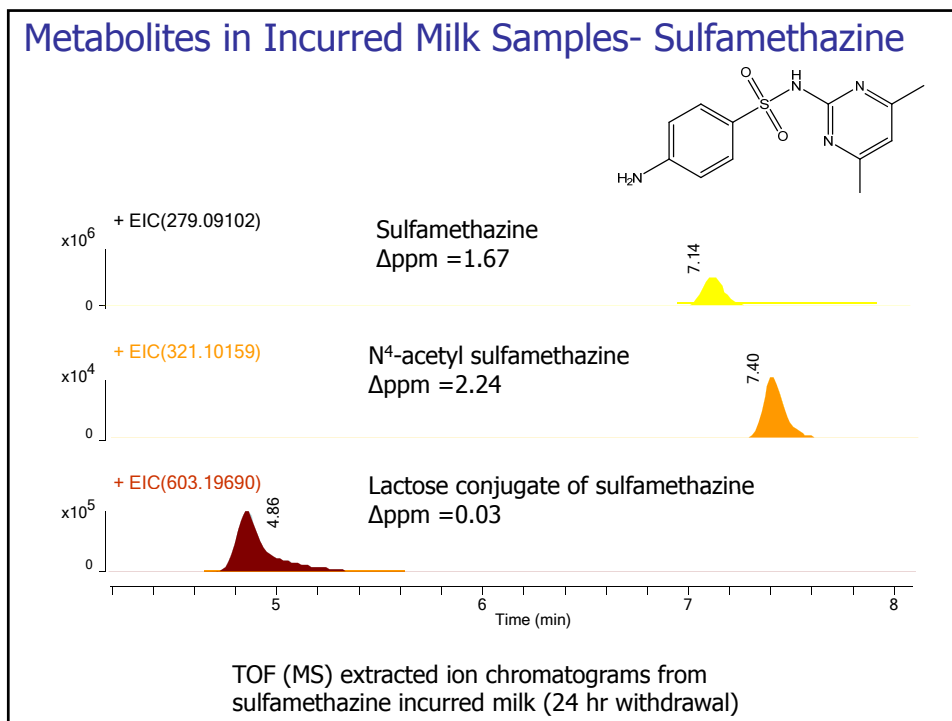


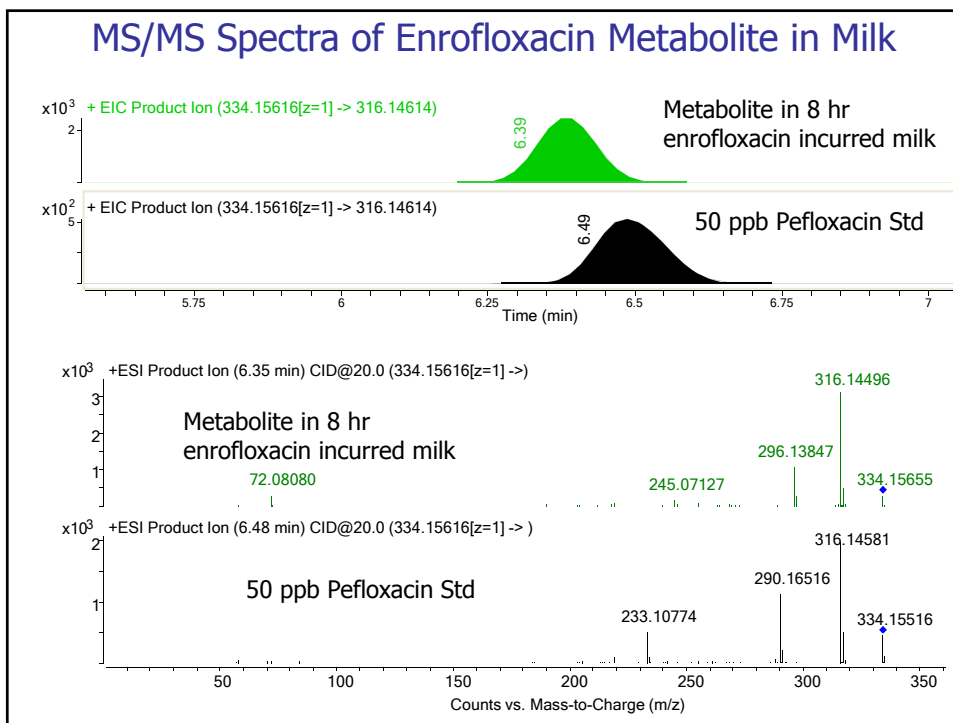
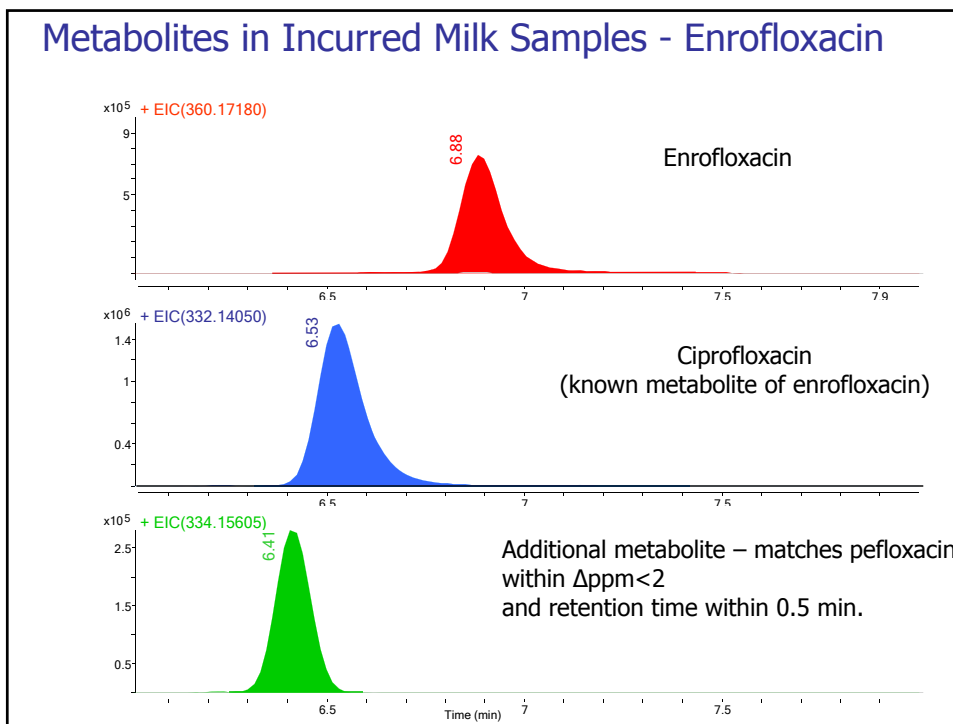
Q-TOF MS/MS analysis of chloramphenicol in frog legs.

Nontarget Analytes in Frog Legs



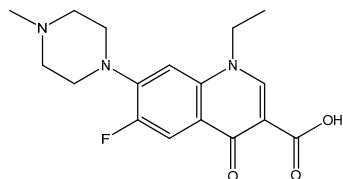
- Trimethoprim residue (~70 ppb) in frog legs was identified by comparing the retention time and MS/MS spectrum to that of a standard compound. In this first sample, sulfamethoxazole did not give an adequate spectrum when analyzed by MS/MS.
- Two other frog legs samples were also analyzed. Fluoroquinolones (primarily enrofloxacin) and trimethoprim were also identified in these frog leg tissues.
- Sulfamethoxazole was identified by MS/MS in one of these other samples.
- Chloramphenicol was also identified at high levels.



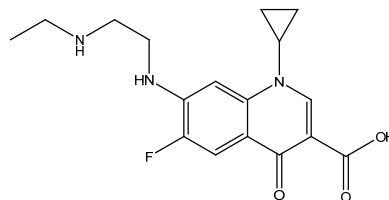


Additional Enrofloxacin Metabolite in Milk

Pefloxacin



Possible structure of metabolite



Metabolite is:

- Isobaric with pefloxacin
- Has same product ions as enrofloxacin and ciprofloxacin, while pefloxacin MS/MS is similar to norfloxacin
- Not detected in standard mixtures or milk fortified with enrofloxacin and then extracted

Conclusions

- Q-TOF LC-MS was successfully used to confirm target analytes with mass error of <2 ppm, retention time and MS/MS spectral match.
- Nontarget analytes, including veterinary drugs and additional metabolites, were identified.
- Obtaining MS/MS data was critical for residue identification.

Questions?

Need more details?

Please come to poster #34 tomorrow!

