Veterinary Drug Residue Testing

Application Notebook

Waters™

Veterinary Drug Residues Testing

Welcome to the Waters™ Vet Drug Residue testing application notebook. Farmers may use authorised veterinary medicinal products (VMPs), applied directly to the animal or via medicated feed, to prevent and control disease, and to improve the conversion rate of feed and for growth promotion (illegal in many countries). Unfortunately, these substances may leave residues in food from treated animals caused by various factors, including failure to adhere to the recommended withdrawal period for each medication, use of doses exceeding the approved guidelines for animals, contamination of animal feed or the unauthorized use of antibiotics. VMPs used on farmed animals are subject to strict regulatory controls to ensure the levels of residues in food should not harm the consumer. In addition, the threat of Antimicrobial Resistance is of growing concern to farmers, the food industry, testing laboratories and competent authorities, alike.

Considering the growing public attention and concern regarding food safety, coupled with the significant role of the meat and aquaculture industries in the global economy, and the necessity to avoid disrupting trade, the need for effective and efficient analytical methods to monitor drug residues in food is expected to keep rising.

Whether you work in testing laboratories in the food industry, in a contract testing organisation, in government, or in a research facility, Waters can help you expand your capabilities and provide a wide range of vet drug residue testing solutions.

Many countries have an integrated approach to food safety that aims to assure a high level of food safety, animal health and welfare and plant health through coherent farm-to-table measures and adequate monitoring, while ensuring the effective functioning of trade.

Before a VMP intended for food-producing animals is authorised, the safety of its pharmacologically active substances and their residues is evaluated, and maximum residue limits (MRL) recommended. The MRL is the maximum concentration of residue resulting from the use of a VMP which may be accepted to be legally permitted or recognised as acceptable in or on a food. Although the values vary country-to-country, MRLs are always set at a sufficiently safe level to ensure that the level of residues in food does not pose a risk to human health.

MRLs are established only for those food producing animals for which an application is made, so uses for other purposes are not authorized. In addition, some products are completely prohibited from use. For example, the EU operates a zero-tolerance policy to residues of prohibited substances, for which no MRLs can be established.

To characterize the residues in food after administration of the veterinary drug, metabolism studies are performed in the target (food) animal species and a suitable marker residue is determined for regulatory monitoring purposes.

Many countries require that food producing animals and associated products of animal origin, such as meat, seafood, milk, eggs and honey, must not contain residue levels of veterinary drugs that might represent a hazard to the health of the consumer. In some cases, countries' legislation also applies to imports of food of animal origin.

Various official controls are used by national competent authorities to verify compliance with legislation on control of VMPs, at all stages of production, processing and distribution. These include residue monitoring plans to detect the use of veterinary medicinal products in food producing animals, on farms, at slaughterhouses and collected from retail outlets. These national plans are often supported by increased levels of import controls and extra safeguard measures including audits. The food industry undertakes its own testing programs for due diligence, brand protection and sometimes product release.

Vet drug residues can be categorised by their biological activity, such as antibiotics, coccidiostats or antiparasitic agents, but are often split into two groups based upon regulatory status:

- Prohibited or unauthorized pharmacologically active substances
- Authorized pharmacologically active substances

Due to the very low rate of non-compliance, a two-tiered approach to analysis is often employed; screening then confirmation. Screening methods are initially used to detect the presence of a substance or class of substances at the level of interest. Rapid and easy to use methods can be based on microbial growth inhibition, receptor binding and enzymatic colorimetric assays or immunological techniques in various formats. LC-MS(/MS) can be used for screening, including multi-residue methods but is essential for confirmatory analyses. In the case of a suspected non-compliant result, analysis is repeated using a validated confirmatory method. Methods, for screening or confirmation, can be categorised as:

- Single residue
- Class-specific
- Multi-residue

All laboratories carrying out the testing need to demonstrate that their analytical methods are fit for purpose and are reliable, so must use only analytical methods which are validated and demonstrated to work in the laboratory in a reliable way. Laboratories must also have a documented quality assurance system in place which will demonstrate that methods perform reliably over time and often some form of accreditation to suitable standard (*e.g.* ISO17025) is required.

CONTENTS

Single Residue and Class-Specific Methods

Determination of Beta Agonists Residues in Animal Tissues and Urine Using LC-MS/MS

Beta-agonists are synthetic compounds that mimic some of the effects of naturally-occurring compounds by binding to beta-receptors on the surface of cells within the muscle, fat, and other tissues of animals Although the administration of β-agonists as growth-promoting agents in food-producing animals is banned in many countries due to concerns over human health, there are exceptions. Most countries have established strict surveillance programs for official control purposes to check compliance with regulatory limits, for both domestic and imported produce. Monitoring compliance within these limits requires the use of highly sensitive and selective analytical methodology based on LC-MS/MS.

Samples of meat and fish were extracted by blending with an aqueous buffer/methanol solution and subjected to enzyme hydrolysis to cleave any conjugated β-agonists, and to help solubilize any residues. SPE was carried out on a mixed mode-type cartridge prior to determination using LC-MS/MS (Xevo™ TQ-S micro Tandem Quadrupole Mass Spectrometer). Samples of urine were extracted by SPE after enzyme hydrolysis.

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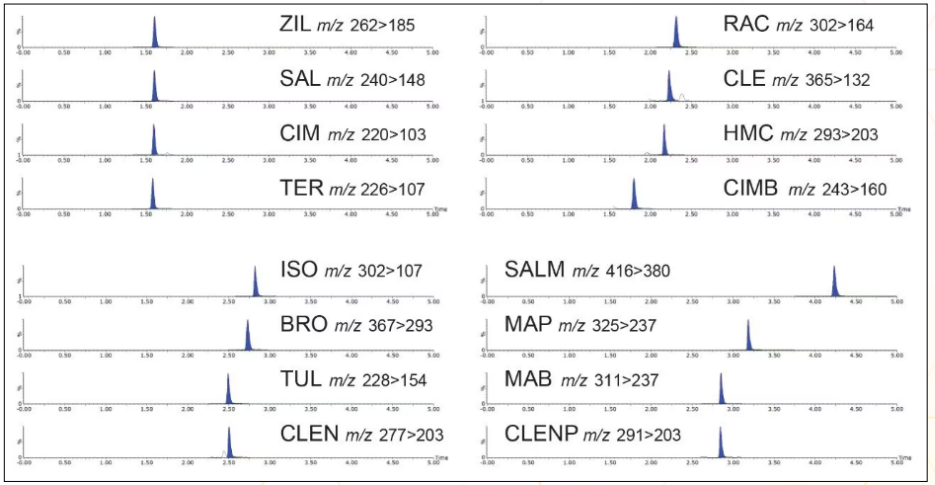
APPLICATION BENEFITS

- Excellent LC-MS/MS sensitivity and selectivity was demonstrated in bovine and poultry liver, salmon, and bovine urine.
- This method can be used for both screening and confirmation for official control purposes with a high degree of confidence.
- The method is also suitability checking compliance with the Russian action level for imported meat of 0.1 µg/kg ractopamine.



Chromatograms for β-agonists in bovine liver.

Determination of Ractopamine and Zilpaterol in Bovine Liver Using LC-MS/MS

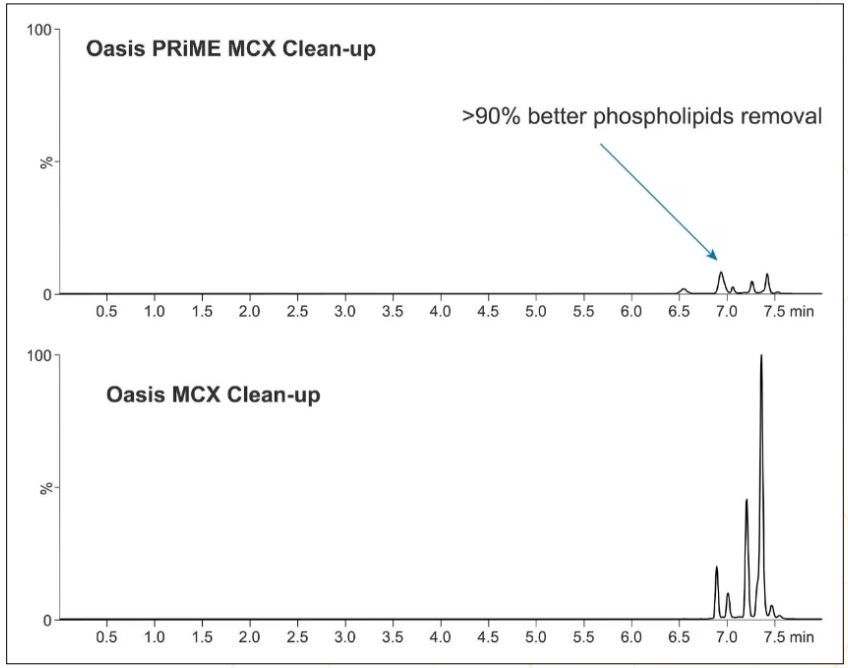
Ractopamine and zilpaterol are β-agonists that are accepted as growth enhancing substances for cattle in a limited number of countries, including the US and Canada, where MRLs have been set. However, these substances are not allowed for use in animal husbandry in most other parts of the world, including the EU. In the absence of MRLs, minimum method performance requirements (MMPRs) for these substances were provided by the EU Reference Laboratories. These are not enforcement legal limits but represent the minimum concentrations at which official laboratories should be able to reliably determine.

To help ensure public health and safety, and to facilitate trade, reliable analytical methods are necessary to determine residues of these compounds in tissue samples obtained from animals raised for human consumption.

In this application note, the performance of a method based upon methanolic extraction, SPE cleanup (Oasis™ PRiME MCX Cartridge) with improved selectivity, and LC-MS/MS (Xevo TQ-S micro) is demonstrated for the determination of ractopamine and zilpaterol in bovine liver. This matrix is challenging for residue analysis as it is high in phospholipids, which can lead to isobaric interferences, ion suppression and contamination of the LC-MS/MS components. Oasis PRiME MCX was effective for cleanup and enrichment of methanolic extracts of bovine liver prior to LC-MS/MS determination of ractopamine and zilpaterol, whilst maintaining acceptable recovery.

APPLICATION BENEFITS

- Compared with the conventional SPE cleanup protocol (AOAC method), 90% more phospholipids were removed from the extracts.
- This method can be used to check regulatory compliance, for a variety of geographies, with a high degree of confidence.



Chromatograms showing improved removal of phospholipids from bovine liver extracts.

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Improved SPE for Determination of Ractopamine in Porcine and Bovine Liver Using LC-MS/MS

The β-agonist ractopamine is still authorized to be used for the production of some animals in a few countries but is banned in many others due to concerns over human health. For example, in 2013, Russian banned pork, chicken and beef imports from the USA due to concerns over the use of ractopamine. Methods applied to monitoring of exports to Russia should be capable of quantifying ractopamine levels of 0.1 µg/kg.

This application note describes a method for the determination of ractopamine residues in liver. Samples were extracted using a modified version of AOAC method 2011.23 based upon methanol extraction of the tissues and enzymatic hydrolysis. The vacuum manifold was substituted with the Otto™ SPEcialist Positive Pressure Manifold, and Oasis MCX SPE cartridges with an Oasis MCX 96-well plate. Determination was by LC-MS/MS (Xevo TQ-XS). The Otto SPEcialist Positive Pressure Manifold is a semi-automated system designed for use with both SPE plates and cartridges The main objective is to achieve consistent performance across multiple SPE runs by eliminating variation caused by differing levels of user expertise

APPLICATION BENEFITS

- The use of Otto SPEcialist, a positive pressure manifold, with Oasis MCX 96-well plate is very effective for clean-up and enrichment of methanolic extracts of porcine liver samples prior to LCMS/MS determination of ractopamine at 0.1 µg/kg.
- Compared to using conventional Oasis MCX SPE cartridges on a vacuum manifold, the use of Otto SPEcialist with plates provides faster, more repeatable results.
- Significant cost savings were achieved through a reduction in volume of solvent and the elimination of consumables such as 0.2 µm filters and centrifuge tubes for sample enrichment guidance.

The Otto SPEcialist with 96 well plate.

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Determination of Nitrofurans in Animal Tissues and Food Products Using LC-MS/MS

Due to health concerns, nitrofuran antibiotics are now prohibited for use in food-producing animals in most jurisdictions. There have been frequent findings of nitrofuran residues in honey, poultry, and aquaculture products imported to EU countries, which has led to product recalls, border rejections, and de-listed suppliers. Violations have resulted in the implementation of emergency measures that have necessitated mandatory pre-export testing, widespread voluntary pre-harvest tests, and an increase in the amount of testing of imports at border control within the EU. Previous studies have demonstrated that parent nitrofurans deplete rapidly in animals and that they are extensively metabolized to tissue-bound metabolites. Commonly sought parent nitrofurans and associated metabolites include: furazolidone as 3-amino-2-oxazolidinone (AOZ), nitrofurazone as semicarbazide (SCA), furaltadone as 3-amino-5-morpholinomethyl-2-oxazolidinone (AMOZ) and nitrofurantoin as 1-aminohydantoin (AHD).

Acid hydrolysis was used to cleave the metabolites bound to proteins or other tissues and to hydrolyse the side-chain of any intact parent drug present, as well as to solubilize any residues. Polar nitrofuran metabolites were derivatized with 2-nitrobenzaldehyde prior to SPE and LC-MS/MS (Xevo TQ-S micro). Excellent sensitivity and selectivity was demonstrated in a range of different sample types: prawn, fish, poultry muscle, bovine kidney, egg, and honey,

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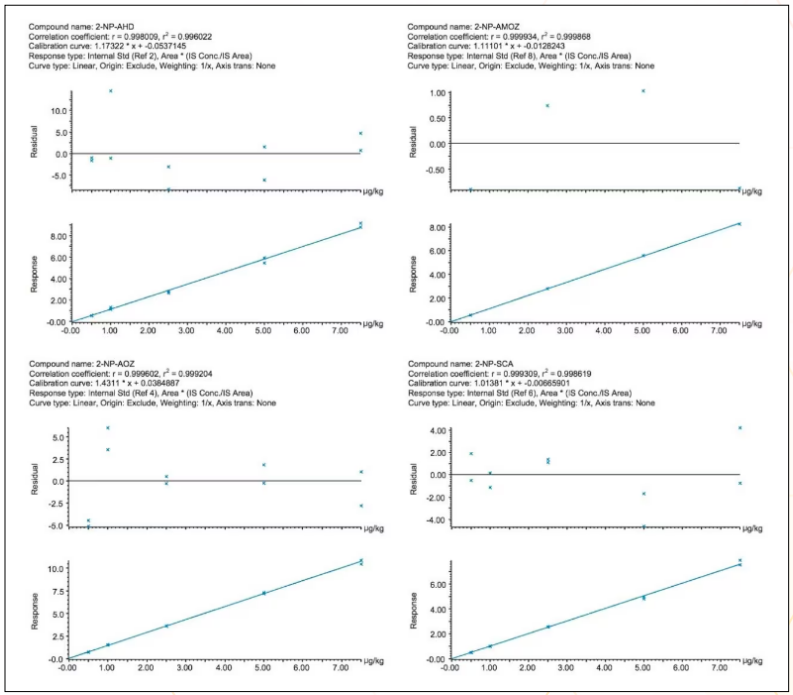
APPLICATION BENEFITS

- The method provides sufficient sensitivity for determination of nitrofuran metabolites in a range of products.
- This method facilitates routine screening and confirmation for official control purposes with a high degree of confidence, but also meets the requirements of pre-export testing, which often demands lower limits of quantification.



Calibration graphs for the nitrofuran metabolites prepared in honey matrix.

Determination of Chloramphenicol in Honey and Milk Using LC-MS/MS

The European Union operates a zero-tolerance policy for residues of prohibited substances, which have no maximum residue limits set. Reference Points for Action (RPA) have been established for prohibited substances when it is deemed necessary to ensure the functioning of controls on food of animal origin imported or placed on the market. Reliable analytical methods are needed for determination of residues of chloramphenicol (CAP) in various animal tissues, biofluids, and associated food products of animal origin.

This application note describes the development of methodology, based on LC-MS/MS, for the analysis of milk and honey. Extracts were prepared using solvent extraction, including a dispersive solid-phase extraction (dSPE) step for milk, followed by determination with LC-MS/MS (Xevo TQ-S cronos). The performance of the methods was successfully verified using acceptance criteria from Commission Implementing Regulation (EU) 2021/808. The results from analysis of the spikes were within the required tolerance for trueness and repeatability, respectively. The method is considered sensitive, accurate, and, after further validation, suitable for the determination of CAP in milk and honey for checking compliance with the RPA at 0.15 µg/kg.

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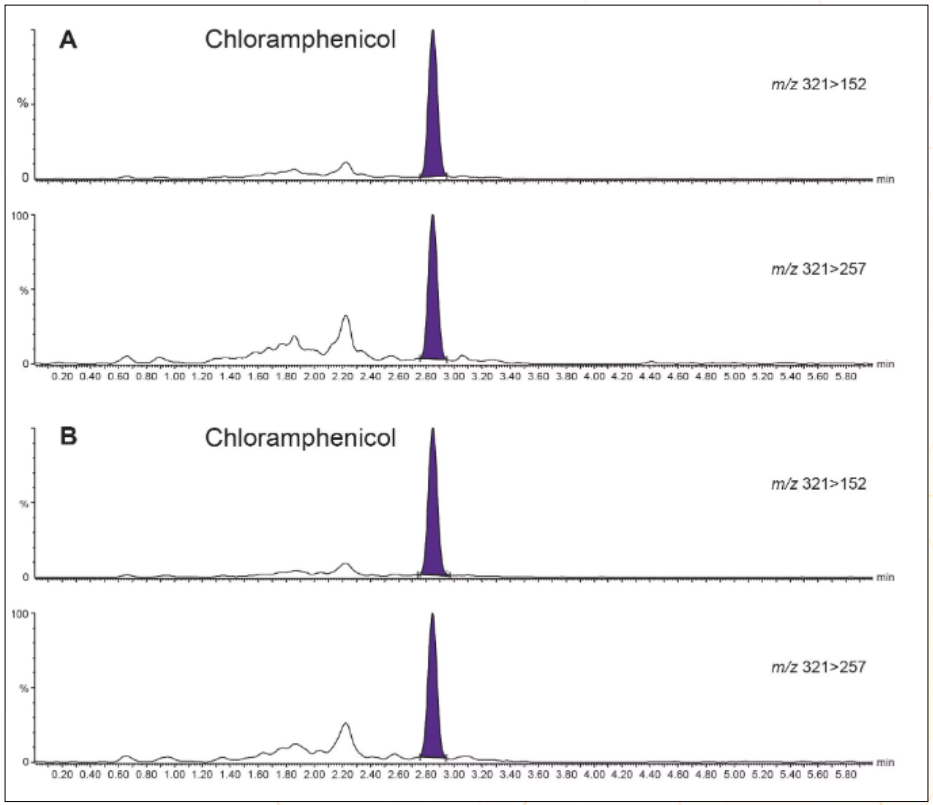
APPLICATION BENEFITS

- Sensitive and selective methods for determination of CAP in milk and honey.
- Detection of CAP at lowest calibrated level of 0.025 µg/kg, which is significantly lower than the RPA for CAP at 0.15 µg/kg.
- The performance of the method meets acceptance criteria for trueness and repeatability set out in (EU) 2021/808.



Chromatograms from the analysis of matrix-fortified standards at the Lowest Calibrated Level, 0.025 µg/kg, in milk (A) and honey (B).

Determination of Nitrofuran Metabolites and Chloramphenicol in Chicken Muscle Using LC-MS/MS

Historically, there has been global concern about the safety of foods of animal origin contaminated with antibacterial residues. Within the European Union, for specific prohibited, or unauthorised pharmacologically active substances. RPAs in food have been established. RPAs are set at the lowest level which can analytically be achieved by the official control laboratories. In 2019, RPAs of 0.15 and 0.5 µg/kg were established for chloramphenicol (CAP) and nitrofurans (NFs), respectively. Samples found to contain these residues at concentrations equivalent to, or above the RPA, are considered not to comply with the legislation and shall not enter the food chain.

Nitrofurans are metabolised rapidly *in vivo* but stable tissue-bound metabolites are formed. Fragments of these metabolites may be released by mild acid hydrolysis and monitored as marker residues; 3-amino-2-oxazolidinone (AOZ) for the drug furazolidone, 3-amino-5-morpholinomethyl-2-oxazolidinone (AMOZ) for furaltadone, 1-aminohydantoin (AHD) for nitrofurantoin and semicarbazide (SCA) for nitrofurazone. To complete the analysis and present extracts that are amenable to reverse phase chromatography, the resulting metabolites are normally derivatized with 2-nitrobenzaldehyde yielding the 'NB' derivatives.

This study investigates the performance of two methods for the determination of CAP and NFs both using LC-MS/MS (Xevo TQ-S cronos).

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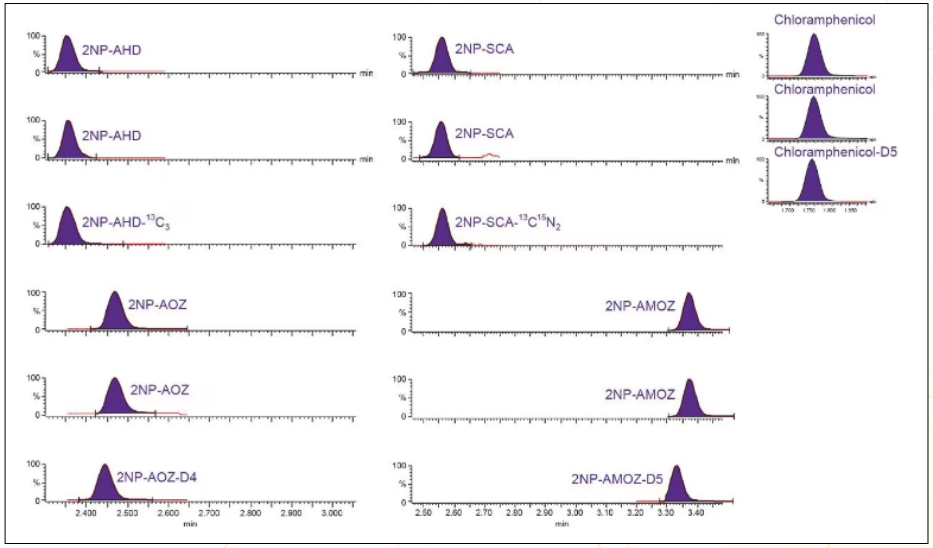
APPLICATION BENEFITS

- Reliable, routine quantitative analysis of banned drug residues in foods of animal origin combined with Oasis SPE sample preparation products for compliance with stringent EU Regulations.
- Demonstrating robust performance in complex matrix extract maximizing instrument uptime with minimal requirements for operator intervention over the run times typically required during compliance monitoring analysis.



Chromatogram obtained for NFs (0.5 µg/kg) and CAP (0.15 µg/kg) in chicken.

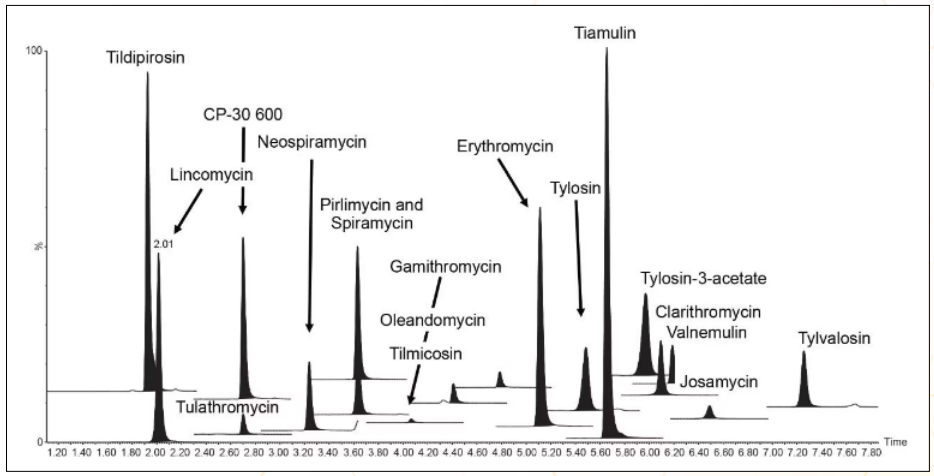
Analysis of Macrolide Antibiotics in Bovine Muscle Tissue Using LC-MS/MS

Macrolides and lincosamides are widely approved for use in veterinary medicine to treat respiratory diseases. Additionally, macrolides are licensed for use in some countries and regions as feed additives to increase the conversion rate of feed and promote animal growth. Residue monitoring plans are used to detect the illegal use or misuse of authorized veterinary medicines in food-producing animals and investigate the reasons for residue violations. In some cases, such as in the EU, exporting countries must also implement a residue monitoring plan that guarantees an equivalent level of food safety.

The method described here proved to be a sensitive and robust multiresidue method for the determination of 18 macrolide antibiotics. Bovine muscle tissue was extracted, after the addition of internal standards, using a liquid extraction followed by SPE clean-up and subsequent LC-MS/MS (Xevo TQ-S micro). The method allows for a fast and reliable quantitation down to concentrations well below typical MRLs and was successfully validated according to the European Commission Decision 2002/657, presenting satisfactory results for all macrolides in bovine muscle tissue. The procedure can also be applied to other animal and fish tissues after suitable validation.

APPLICATION BENEFITS

- Determination of a broad range of macrolide antibiotic veterinary drugs in a single analysis.
- Effective cleanup using SPE, keeping injection volumes low, and using sensitive instrumentation; all combine to provide a robust and reliable analytical solution.
- Demonstration of successful validation provides increased confidence in the suitability of the method.



Chromatograms for a selection of macrolides in beef extract.

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Determination of Triphenylmethane Dyes in Shrimps Using LC-MS/MS

Triphenylmethane dyes, common commercial and inexpensive industrial dyes, are used illegally in aquaculture for their antimicrobial properties. After digestion, dyes such as malachite green (MG) and crystal violet (CV), are rapidly metabolized to reduced leuco- forms (LMG and LCV) which can persist in fish long after dosing and represent a heath concern to consumers. MG is banned for use in food-producing animals in major geographies. The EU sets the RPA, which has been established for MG of 0.5 µg/kg (for the sum of MG and LMG), to ensure the functioning of controls for food of animal origin including imports. Food of animal origin containing residues of such substances at or above the RPA is considered non-compliant with Union legislation. Therefore, suitable methods are required to monitor compliance with such regulations.

A modified version of QuEChERS, with dispersive SPE, was used to prepare extracts of shrimps prior to LC-MS/MS (Xevo TQ-S cronos). Matrix effects were evaluated and found to be significant for LCV. These effects were mitigated by use of matrix-matched standards and the use of isotopically labelled internal standards.

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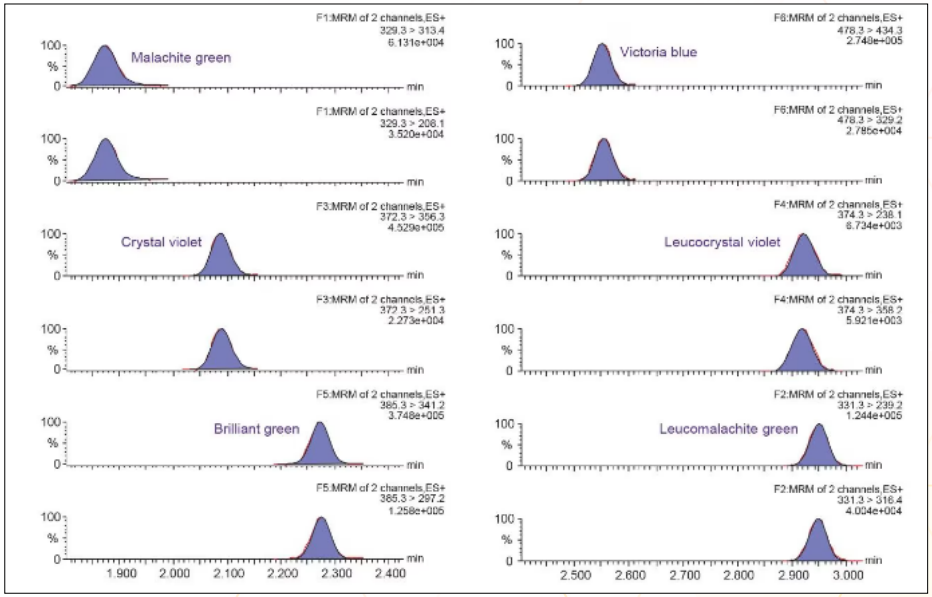
APPLICATION BENEFITS

- Electrospray probe position and cone gas flow can be easily adjusted for optimizing source conditions when using the Xevo TQ-S cronos instrument.
- This method has shown to be suitable for checking regulatory compliance for these dyes in seafood with detection at the RPA level.



Chromatograms for the dyes in shrimps (0.5 µg/kg).

Development and Validation of a Method for the Determination of Aminoglycosides in Foods Using LC-MS/MS

Aminoglycosides (AMGs) are broad-spectrum antibiotics commonly used as veterinary drugs on food-producing animals. Many countries have established MRLs for AMGs approved for use on animals, so they are often determined in honey, eggs, milk, tissues, and biofluids of food-producing animals for control and monitoring purposes. AMGs are highly polar compounds and show little to no retention in reversed phase columns. Although ion-pairing reagents have been utilized with C_{18} columns, when used with LC-MS/MS, this approach can suffer from ion suppression and contamination of the systems. The introduction of hydrophilic interaction chromatography (HILIC) provided a more MS-compatible option for the analysis of polar compounds.

Here we show the results from the successful evaluation of the Atlantis™ Premier BEH™ Z-HILIC Column, which has a sulfobetaine zwitterionic chemistry, for the determination of AMGs by LC-MS/MS (Xevo TQ-XS). The performance of the method was successfully verified according to Commission Implementing Regulation (EU) 2021/808. The method is suitable for reliable confirmation of residues to check compliance with MRLs globally and in cases where use of the substances is not allowed.

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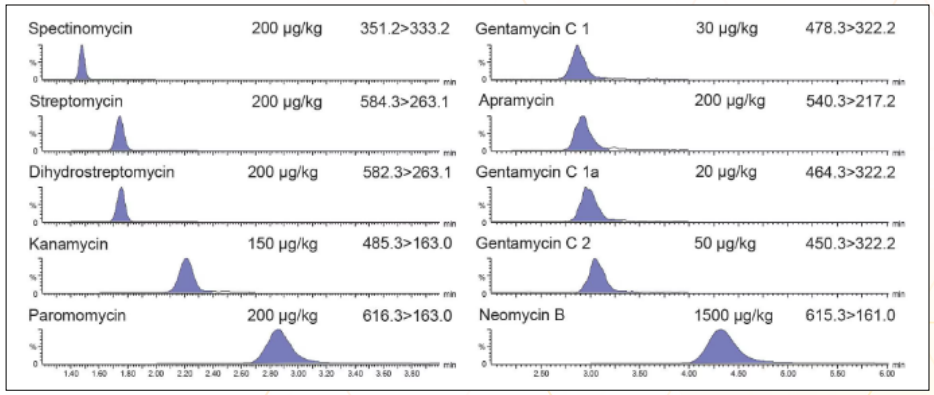
APPLICATION BENEFITS

- Chromatographic retention and separation were provided by the Atlantis Premier BEH Z-HILIC Column, using an MS friendly mobile phase, without resorting to an ion-pairing reagent.
- The method is suitable for reliable determination of residues to check compliance with MRLs and in cases where use of the substances is not allowed.
- The method was successfully validated in three sample types, according to Commission Implementing Regulation (EU) 2021/808.



Chromatograms for a selection of aminoglycosides in milk extract.

Multi-Residue Methods

The Determination of Tetracycline and Sulfonamide Antibiotic Residues in Shrimp Tissue Using LC-MS/MS

To meet growing demand, shrimps and other seafood are often cultivated by aquafarms, where many animals are kept in relatively small spaces, making them more prone to diseases. In order to preserve animal health as well as to ensure production and to increase yields, antibiotics are used on a large scale. Residues of these antibiotics in foods of animal origin are a major concern because they are harmful to the consumer's health and could induce pathogens to develop resistance

The method described here proved to be a sensitive and robust multiresidue method for the determination of a series of different antibiotics, namely tetracyclines, sulfonamides, trimethoprim, ormetoprim, and dapsone antibiotics, based upon LC-MS/MS (Xevo TQ-S micro). The method allows for a fast and reliable quantitation down to concentrations well below typical MRLs and was successfully validated according the European Commission Decision 2002/657, presenting satisfactory results for tetracyclines, sulfonamides, and related antibiotics in shrimp tissue. The procedure can also be applied to other animal and fish tissues after suitable validation. This cost-effective method can be easily implemented in routine testing laboratories, has been demonstrated as suitable for checking compliance with MRLs, and has the potential for screening at much lower concentrations, such as for food business operators' due diligence testing.

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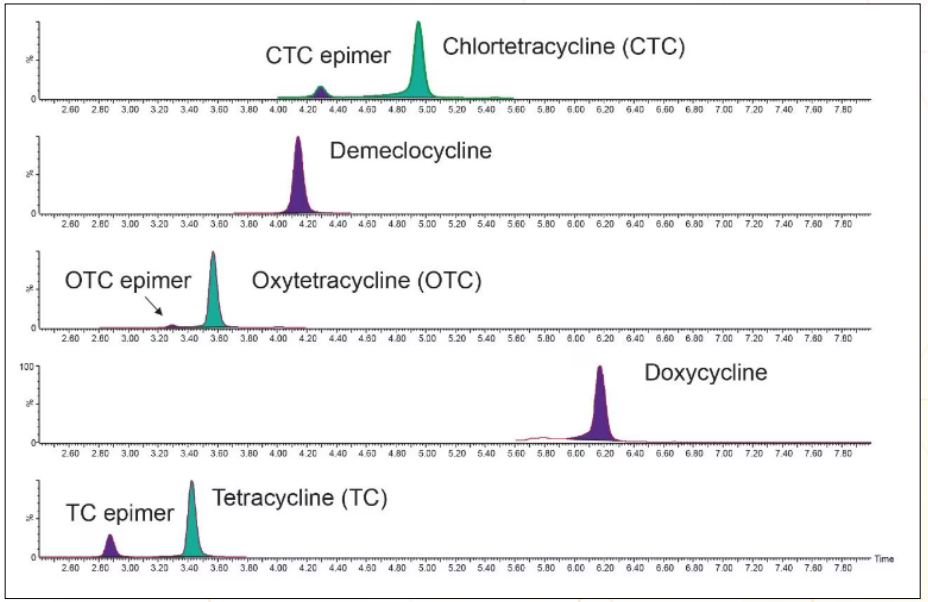
APPLICATION BENEFITS

- Combining a wide range of tetracyclines, sulfonamides, and related antibiotic veterinary drugs into a single analysis.
- Effective cleanup using small injection volumes and a sensitive mass spectrometer combine to provide a robust and reliable analytical solution.
- Demonstration of successful validation provides increased confidence in the suitability of the method.



Chromatogram for tetracyclines in shrimp.

The Determination of Veterinary Drug Residues in Animal Muscle Tissue Using Multi-Residue Screening Based Upon LC-MS/MS

This application note describes the development and validation of a comprehensive screening method based on UPLC™-MS/MS for the detection of over 150 veterinary drugs, from many different classes, in animal muscle tissue. Consolidation of many compounds into one screening method improves operational efficiency and reduces costs. Muscle tissues were extracted using a generic liquid extraction using oxalic acid in acetonitrile followed by a rapid and cost-effective clean-up using dispersive solid-phase extraction (dSPE) and determination by UPLC-MS/MS (Xevo TQ-XS). Despite using generic conditions, a UPLC System can provide increases in sample throughput by speeding up analysis times, maintains good peak shape across a wide range of different compounds, ensures sufficient retention of polar compounds, and the separation of critical pairs.
The method was successfully validated in muscle tissue using the guideline document on screening methods that supplements Commission Decision 2002/657/EC. In all cases, values for CCβ were established at concentrations below MRLs and in most cases at 0.1 or 1.0 µg/kg. As validation criteria have been met, the method is considered sensitive, robust, specific, and fit for the purpose of screening for veterinary drug residues.

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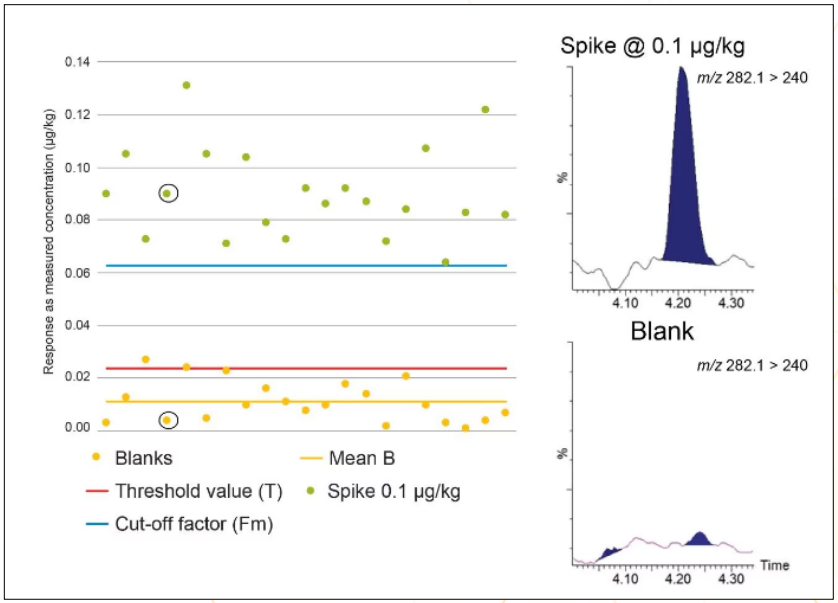
APPLICATION BENEFITS

- Determination of a broad range of veterinary drugs in a single analysis to improve laboratory efficiency when compared with using a series of class-specific methods.
- Cost-effective clean-up using dSPE, keeping injection volumes low and using sensitive instrumentation all combine to provide a robust and reliable analytical solution.
- Demonstration of successful validation provides increased confidence in the suitability .



Graph showing Blank (B), Threshold value (T), and Cut-off factor (Fm) for albendazole sulfoxide in mixed muscle samples at 0.1 µg/kg with associated chromatograms.

The Determination of Medicated Feed Additives Using LC-MS/MS

Feed additives are products used in animal nutrition for purposes of improving the quality of feed and the quality of food from animal origin, or to improve the animals' performance and health. Medicated feed is one way that can be used to administer authorised veterinary medicines to animals. The production, supply and use of medicines, including those for in-feed treatment, is highly regulated at all stages in many countries. For example, in China, only additives listed in catalogue of approved feed additives can be used in animal feed products. New feed additives must be approved by the Ministry of Agriculture and Rural Affairs (MARA) first before they can be used in animal feeds.

However illegal use of banned vet drugs as feed additives remains an issue in addition to problems with cross contamination in the feed mill. Medicated feed additives include antibiotics, antimicrobials, anti-coccidials, antiparasitics, sulfonamidics, hormones, anti-bloat compounds and beta-agonists.

The method described here can be used for the determination of seven additives from different classes. After extraction with a mixture of acetonitrile and water 80:20, Oasis PRiME HLB was used for SPE clean-up in a pass-through mode), prior to LC-MS/MS (Xevo TQ-S micro).

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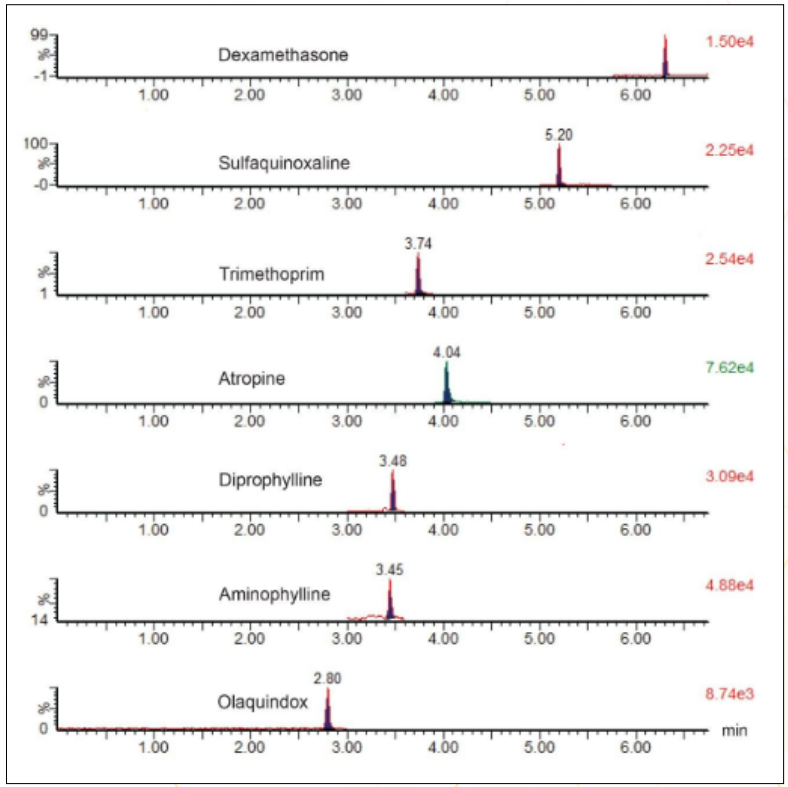
APPLICATION BENEFITS

- Efficient, time-saving total solution for muti-residue analysis of veterinary drugs in animal feed.
- Simple and rapid sample clean-up with Oasis PRiME HLB.
- Fast and sensitive UPLC-MS/MS analysis.



Chromatograms of spiked feed sample.

Research

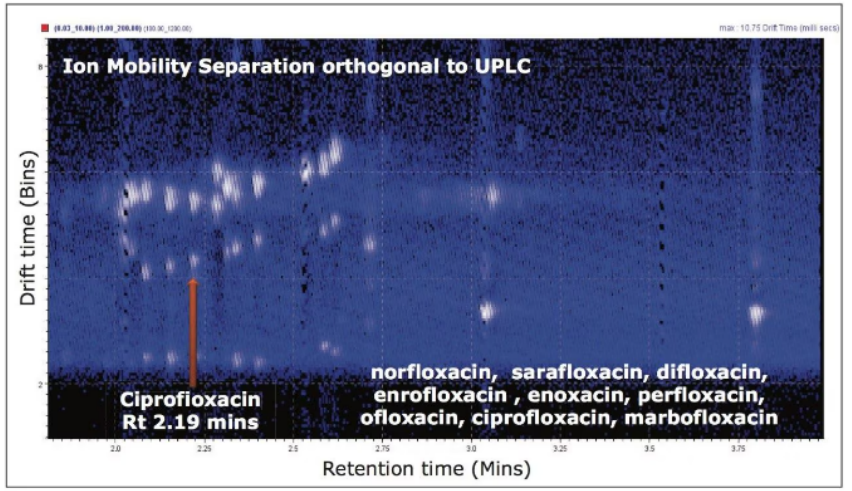
Using Ion Mobility Coupled with High Resolution Mass Spectrometry to Better Characterize Structure of Fluoroquinolone Antibiotics

Fluoroquinolones are a family of synthetic broad-spectrum antimicrobial agents that have been administered to livestock for different purposes, including the prevention and control of infections and for growth promotion. This application note explores the use of High Definition Mass Spectrometry (HDMS™) as an important method development tool to support the identification of fluoroquinolone antibiotics in tissue extracts. HDMS has been utilized to analyze crude extracts of porcine muscle tissue to determine the presence of antibiotic residues including the fluoroquinolone class. This technique uses a combination of high resolution mass spectrometry and high efficiency ion mobility-based measurements and separations. Ion mobility spectrometry (IMS) is a rapid, orthogonal, gas phase separation technique that allows another dimension of separation to be obtained within an LC timeframe. Compounds can be differentiated based on size, shape, and charge. In addition, both precursor ion and fragment ion information can be acquired in a single injection in an HDMS experiment, referred to as HDMS^E.

Separation of different intra-molecular protonated species was achieved using ion mobility. Multiple sites of protonation have been shown and identified from the different single-component fragmentation spectra. Single-component precursor ion MS and MS^E fragmentation spectra were simultaneously generated for all components. Ion mobility separations have been utilized to resolve analyte peaks from matrix interferences and eliminate the need for complex sample cleanup and chromatographic separations.

APPLICATION BENEFITS

- Analysis of complex samples benefits from the orthogonal separation produced using travelling wave ion mobility.
- Orthogonal mobility separation enables single component MS and MS^E spectra to be produced simultaneously for components in a complex sample.
- Protomers observed can be uniquely identified.



Plot of drift time versus retention time for a mixture of veterinary drugs showing two protomers for each compound.

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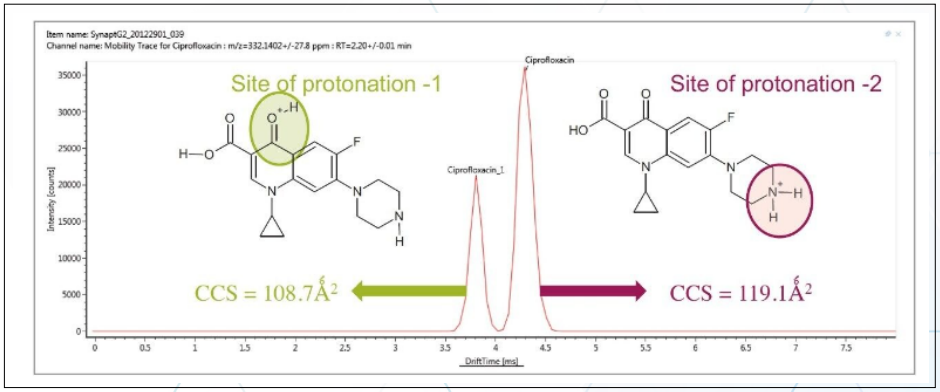
Utility of Chromatography, Ion Mobility and High Resolution Mass Spectrometry in a Routine Workflow for Investigating Vet Drug Residues

This application note explores the use of routine screening with UPLC and ion mobility combined with high resolution mass spectrometry (SYNAPT™ G2-S) to identify multiple protonation sites and different fragmentation patterns within the fluoroquinolone class of antibiotics. It can be used as an important method development tool to support the unequivocal identification of fluoroquinolone antibiotics in crude tissue extracts. UPLC and ion mobility have been utilized to analyze crude extracts of porcine muscle tissue to determine the presence of antibiotic residues including the fluoroquinolone class.

A collision cross section (CCS) value is an important distinguishing characteristic of an ion which is related to its chemical structure and three-dimensional conformation, where the shadow of a rotating three-dimensional ion represents the average collision cross section. Using CCS measurements can increase targeted screening specificity. CCS measurements generated have been entered into a scientific library within UNIFI™. This allows the expected and determined CCS values to be utilized in order to screen and confirm fluoroquinolone protomer formation.

APPLICATION BENEFITS

- Unique protomer CCS values can be determined and used as an additional identification parameter in a routine screening workflow.
- Ion mobility separations can be effectively utilized to resolve analyte peaks from matrix interferences and remove the need for complex sample cleanup.
- The impact of isobaric interference upon protomer ratios can be observed routinely using the ion mobility processing functionality within the UNIFI Scientific Information System.



Mobility trace for protomers of ciprofloxacin with hypothesised respective sites of acid/basic group protonation highlighted and determined estimated CCS values.

Read the Full Application Note

Links to other Useful Materials

Application notes describing the use of SPE in a pass-through mode using Oasis PRiME HLB for vet drug multi-residue analysis.
- Seafood
- Infant formula
- Meat

An application brief showing improved performance using MaxPeak™ High Performance Surfaces (HPS) for analysis of metal sensitive tetracyclines

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