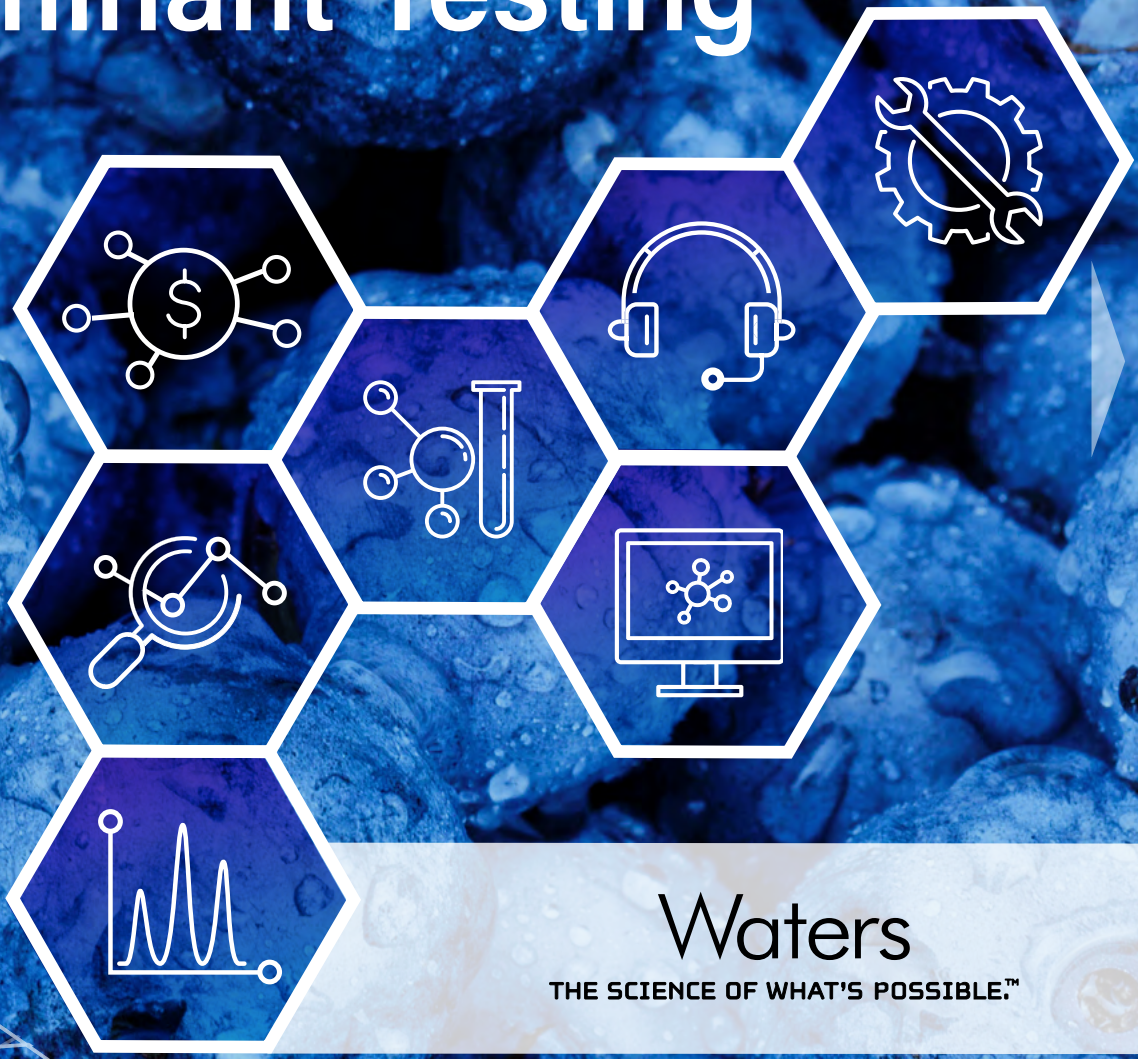
Food and Beverage Chemical Contaminant Testing

Application Notebook



Waters

THE SCIENCE OF WHAT'S POSSIBLE.™

Introduction

EFFICIENT ANALYTICAL SOLUTIONS TO SUPPORT A SAFE, NUTRITIOUS, AUTHENTIC AND SUSTAINABLE FOOD SUPPLY CHAIN

Welcome to Waters' food and beverage contaminants testing application notebook. The role of food testing laboratories has never been more critical. Safety, authenticity, sustainability and quality are of major concern to consumers, governments, and producers. Inside this eBook you will find a compilation of our scientists' latest application notes, supporting your development and implementation of new testing methods and technologies for the analysis of:

- Pesticides
- Veterinary Drugs
- Mycotoxins
- Alkaloids
- Per- and poly-fluoroalkyl substances (PFAS)

Trust in Waters to provide scalable application procedures and technologies to help you adapt quickly to challenges brought in by new regulations, new opportunities and competitive pressures. Improve internal efficiencies with less re-analysis and reduced waste, ensuring product safety and workflow optimization. By partnering with us you gain access to unmatched levels of application support and award-winning service which all aim to have your labs running effectively and consistently day-in-day-out.

CONTENTS


Pesticide Residues

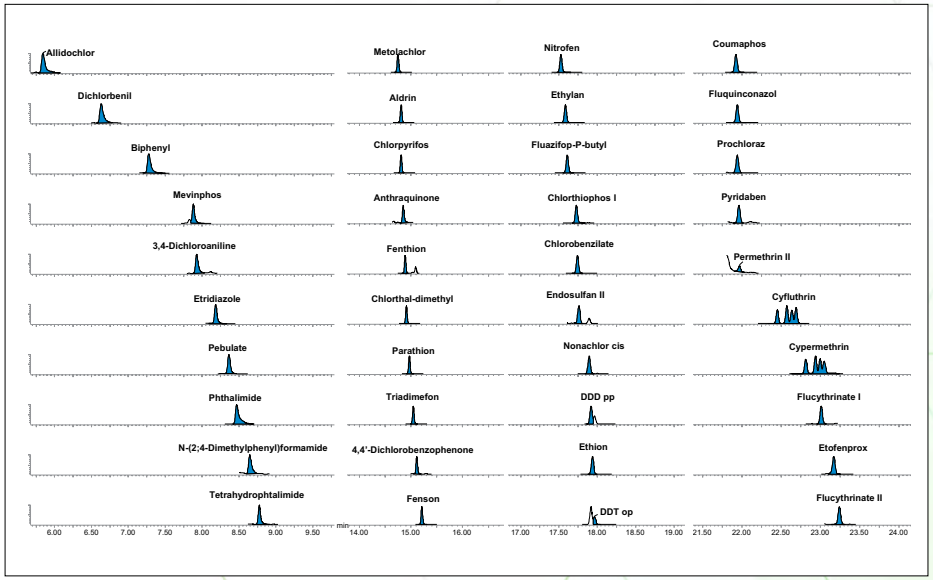
Determination of Pesticide Residues in Cucumber Using GC-MS/MS With APGC After Extraction and Clean-up Using QuEChERS

This application note describes the development of a comprehensive method based on gas chromatography-tandem quadrupole mass spectrometry (GC-MS/MS) for the determination of over 200 pesticides. Extracts of cucumber were prepared using a version of the Quick, Easy, Cheap, Effective, Rugged, and Safe (QuEChERS) method, including a dispersive solid-phase extraction (dSPE) step, followed by determination with GC-MS/MS. The use of GC-MS/MS utilizing atmospheric pressure gas chromatography (APGC) has been shown to offer significant improvements in performance over electron ionization (EI) for pesticide residue analysis, in terms of selectivity and specificity. The extremely high sensitivity of the APGC Xevo TQ-XS System was demonstrated with reliable detection for all the analytes at concentrations as low as 0.001 mg/kg, even when the injection volume was 1 µL. The method was successfully validated in cucumber in accordance with the SANTE guidelines document. The results from analysis of the spikes showed that almost all the analytes were within the required tolerance for recovery and repeatability. The method is considered sensitive, specific, accurate, and has the potential for determination at much lower concentrations.

Read the Full Application Note

APPLICATION BENEFITS

- The GC-MS/MS system, with ionization at atmospheric pressure, yields highly selective and specific MRM data, when compared with traditional election ionization systems, allowing improved confidence in the identification and quantitation of pesticide residues
- The method exhibited very high sensitivity (LODs typically <0.0005 mg/kg) without the need for solvent exchange, PTV or large volume injection



Chromatograms from the analysis of a selection of analytes in the cucumber matrix-matched standard at 0.001 mg/kg.

Multi-Residue Method for the Quantification of Pesticides in Fruits, Vegetables, Cereals and Black Tea using UPLC-MS/MS

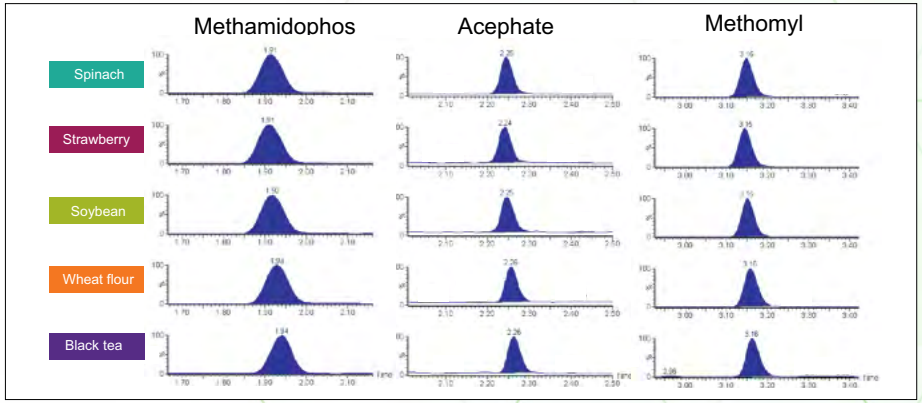
As the global population grows, demand for food consumption and global trade in the food industry has also increased. Hundreds of pesticides are routinely used for crop protection across the globe, traces of pesticides left in treated commodities are called “residues”. Regulations are in place for Maximum Residue Levels (MRL), that are legally tolerated in or on food and feed when pesticides are applied correctly in accordance with Good Agricultural Practice. A growing target list of pesticides in complex matrices, and the need for low limits of detection, bring various challenges for multi-residue methods.

A multi-residue method for 552 pesticides and relevant metabolites was developed for various food commodities, applying the QuEChERS based analyte extraction. Extracts from representative commodities, including high-water content (spinach), high acid and highwater content (strawberry), high oil and very low-water content (soybean), high protein and low-water and fat content (wheat flour), and difficult or unique commodities (black tea) were chosen to assess the performance of the UPLC-MS/MS method.

Read the Full Application Note

APPLICATION BENEFITS

- The UPLC-MS/MS method demonstrated excellent sensitivity, precision, repeatability for the determination of more than 500 pesticides in various commodities at concentrations below the default EU MRL. The method can be readily adopted, extended and optimized to meet your laboratory needs
- A simple ten fold dilution of the extracts minimized matrix effects while still allowing the majority of analytes to be detected below the typical EU default MRL of 0.01 mg/kg
- The Gaussian peak shape of early eluting compounds was maintained by use of the post injector mixing kit. This allowed strong solvent effects (often experienced when injecting high organic based QuEChERS extracts into an aqueous LC gradient) to be overcome, while maintaining analyte stability in the vial and injection onto the column



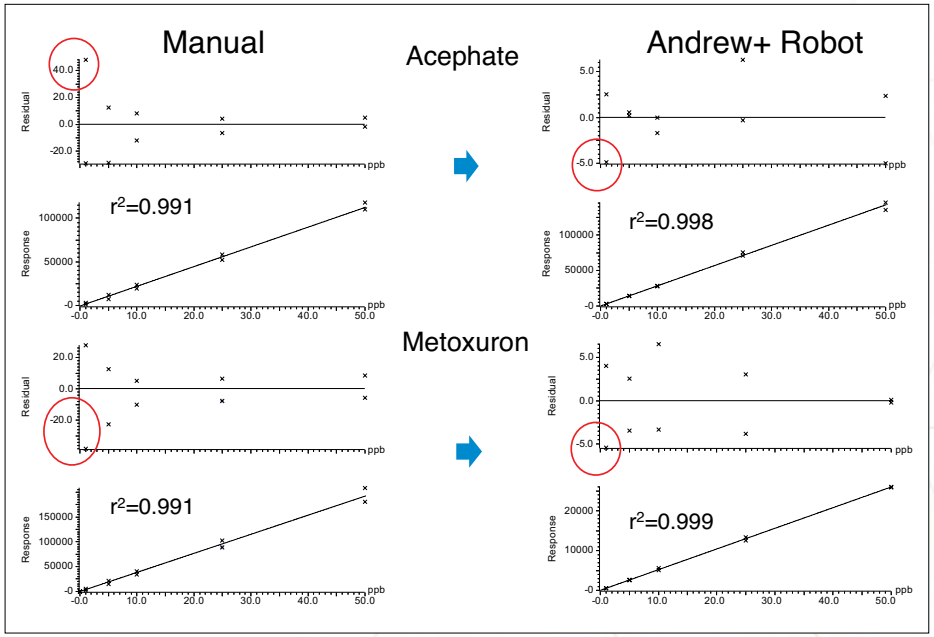
Chromatograms for some of the very polar analytes across matrices with the post injector mixing kit fitted.

Automating Preparation of Matrix-Matched Standards for Pesticide Residue Analysis Using the Andrew+™ Pipetting Robot

The Andrew+ Pipetting Robot and the cloud-native OneLab™ Software Platform have been successfully evaluated for automating the preparation of matrix-matched standards for pesticide residue analysis by LC-MS/MS. We found its performance consistent with the requirements associated with the preparation of standard solutions, even in a matrix extract. The characteristics of the calibration graphs were improved when matrix-matched standards were prepared automatically. Automating this important analytical step saves time, reduces risk of repetitive strain injury, minimizes opportunity for error, and enables technical staff to be freed up for other tasks, all while ensuring full traceability.

APPLICATION BENEFITS

- Fully automated process which can be completed, unattended in less than 14 minutes
- Easy to use with minimal training required
- Consistent pipetting results provides accurate and precise quantification
- Reduces the need for repetitive pipetting which can lead to repetitive strain injury



Typical calibration graphs prepared from the analysis of matrix-matched standards prepared in duplicate.

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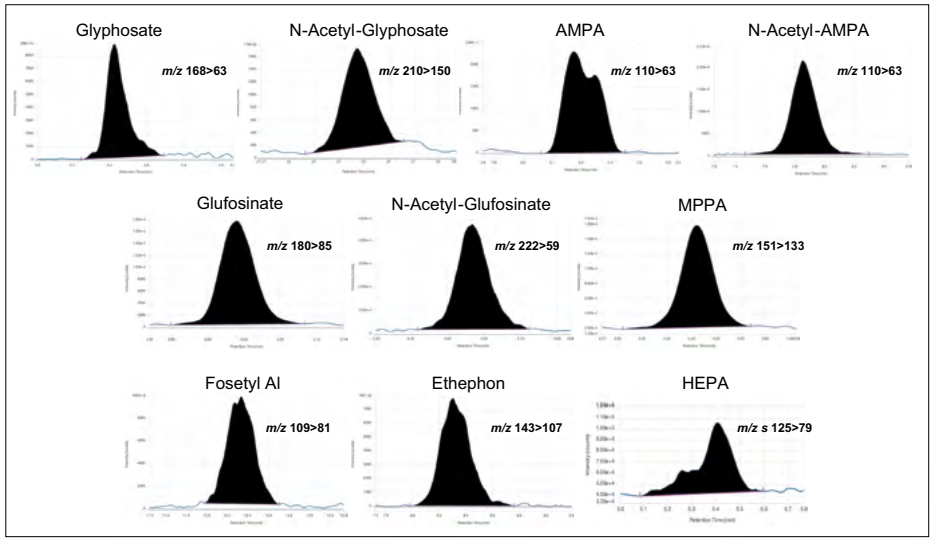
Detection of Anionic Polar Pesticides in Food Samples Using the Xevo TQ Absolute With Sub µg/kg Limits of Quantification

The area of anionic polar pesticide analysis has been evolving over the past ten years where the adoption of generic extraction methods, such as the QuPPe (Quick Polar Pesticides) method, have enabled laboratories to take a multi-residue approach for the analysis of these challenging analytes. With the enhanced negative ion sensitivity of the Xevo TQ Absolute Tandem Quadrupole Mass Spectrometer, limits of quantification of 0.5 µg/kg in vegetable samples and 2 µg/kg in cereal samples are achievable. Trueness was assessed over ten injections at 1 and 10 µg/kg in cucumber matrix standards and at 10 and 50 µg/kg in wheat flour matrix standards. Trueness in cucumber was between 91 to 117% with RSDs between 0.6 to 8.7% and between 96 to 104% in wheat flour with RSDs between 0.5 to 9.2%.



Chromatograms of the anionic polar pesticide and metabolites from the analysis of a cucumber matrix standard at 1 µg/kg (in vial concentration 0.5 ng/mL).

APPLICATION BENEFITS

- Utilizing the Anionic Polar Pesticide Column with UPLC-MS/MS and generic QuPPe extraction, anionic polar pesticides and their associated metabolites can be accurately and reliably identified and quantified, without the need for derivatization. Repeatable retention times and chromatographic separation from isobaric interferences have been demonstrated across commodities, to avoid false detections
- Performance of the Xevo TQ Absolute demonstrates enhanced sensitivity for the analysis of anionic polar pesticides achieving significantly lower method detection limits than previously demonstrated. Reduced sample injection volume enables a reduction in sample matrix being introduced into the LC-MS/MS system
- Implementation is supported across the globe utilizing our outcome-based support model to ensure customer success

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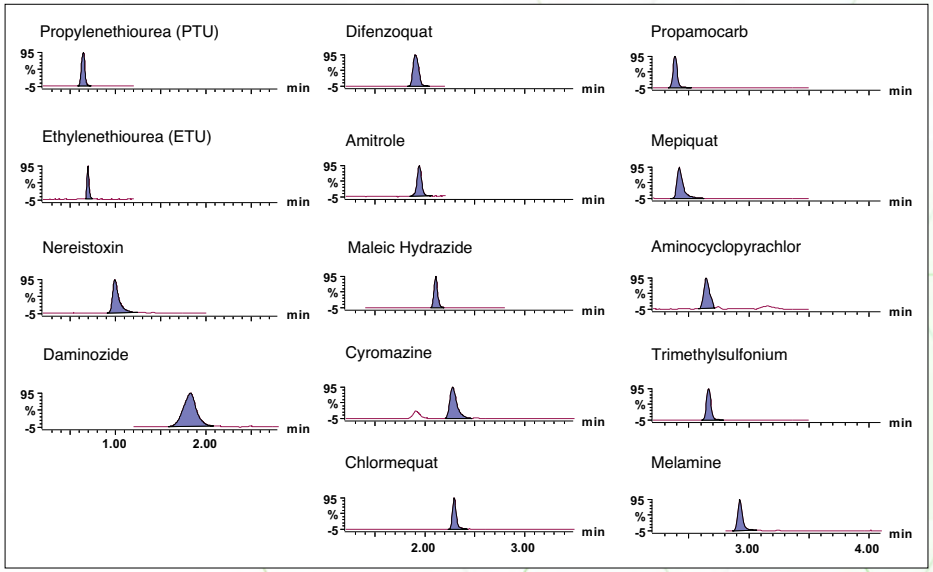
Determination of Cationic Polar Pesticides and Plant Growth Regulators Using UPLC-MS/MS with the ACQUITY UPLC BEH Amide Column

The purpose of this work is to demonstrate a single extraction and LC-MS/MS method for the determination of highly polar cationic pesticide residues and plant growth regulators in several food commodities that meet or exceed the MRL detection requirements in the European Commission pesticides database. The method performance study was completed on an ACQUITY UPLC I-Class System with a Xevo TQ-S micro using an ACQUITY UPLC BEH Amide Column after extraction following the QuPPe method. A method validation study was carried out on 4 representative commodities; namely apple, cucumber, flour, and potatoes. The method performance was assessed using 2 spike levels, 0.01 and 0.05 mg/kg for all analytes except maleic hydrazide which was spiked at 0.5 and 1.5 mg/kg with 5 replicates at each level. Difenzoquat and aminocyclopyrachlor were the only analytes not internally standardized. Method performance for trueness was 92 to 108% across all commodities with the exception of difenzoquat in cucumber (60–67%) where it was identified that PVDF filters were not suitable to use for this analysis. RSDs were all at or below 12%. A FAPAS QC flour sample was extracted on two occasions in triplicate one month apart and all results were within 20% of the assigned value and within the range necessary to achieve an acceptable z-score.

Read the Full Application Note

APPLICATION BENEFITS

- Provides a single extraction (QuPPe) and LC-MS/MS method suitable for the determination of various highly polar cationic pesticides and plant growth regulators in cereals, fruit, and vegetable commodities to facilitate monitoring of MRL/tolerance compliance
- Offers sufficient chromatographic retention, selectivity, peak shape, and stability to comply with SANTE guidelines
- Provides sufficient sensitivity to determine residues at concentrations as low as 0.01 mg/kg in crude extracts without clean-up



Example chromatograms for a 0.02 mg/kg (0.5 mg/kg maleic hydrazide) wheat flour matrix matched calibration standard.

Your Partner in Pesticide Testing

Single residue methods for Anionic and Cationic Polar Pesticides.

Multi-residue methods by LC-MS/MS and GC-MS/MS.

Elucidation, metabolite identification by LC-HRMS and GC-HRMS.

>>Click here to learn more about Pesticide Testing

Veterinary Drugs

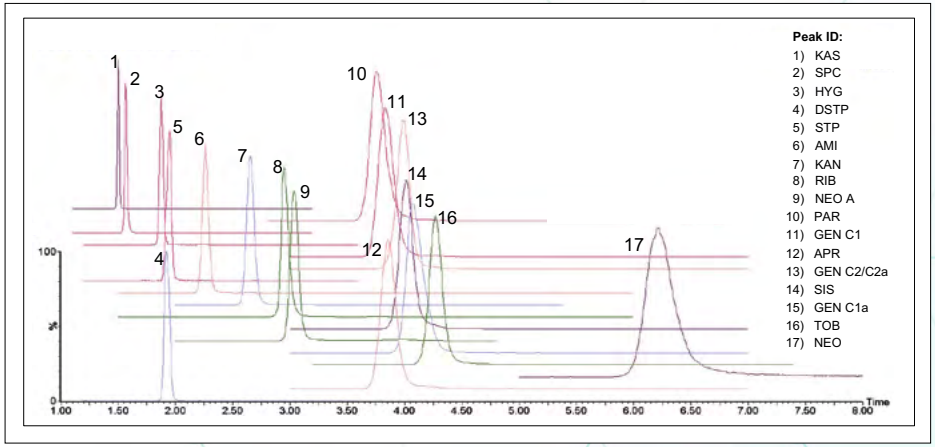
Analysis of Aminoglycosides in Foods Using a Zwitterionic Stationary Phase and Liquid Chromatography-Tandem Quadrupole Mass Spectrometry

The effects of chromatographic conditions, such as mobile phases, pH, and ionic strength (or buffer concentration), on the separation of 17 highly polar aminoglycosides (AMGs) using the Atlantis™ Premier BEH Z-HILIC columns were systematically investigated. Gradient elution with a binary mobile phase of aqueous 20 mM ammonium formate at pH 3.0 and acetonitrile with 0.1% formic acid provided a reliable and adequate separation with excellent sensitivity for these AMGs by electrospray tandem quadrupole mass spectrometry (ESI-MS/MS). The extraction of food samples was carried out using a trichloroacetic acid containing solution. A solid phase extraction (SPE) clean-up procedure was optimized using Oasis HLB Cartridges. This method was evaluated for milk, beef, pork, liver, and honey samples. Good performance characteristics for sensitivity, accuracy, and precision were obtained for 16 AMGs. The final optimized HILIC-ESI-MS/MS method was demonstrated to be reliable, accurate, and sensitive for the determination of AMGs in food.

Read the Full Application Note

APPLICATION BENEFITS

- Reliable separation of 17 aminoglycosides using an Atlantis Premier BEH Z-HILIC Column
- Sensitive HILIC-MS/MS method that meets regulatory requirements in major markets
- Accurate and reliable determination of AMGs in milk, muscle, liver, and honey
- MS friendly mobile phase with no ion pair reagents nor high concentration buffer



Chromatogram overlay of 17 aminoglycosides in a spiked blank milk sample, obtained under the final optimized conditions.

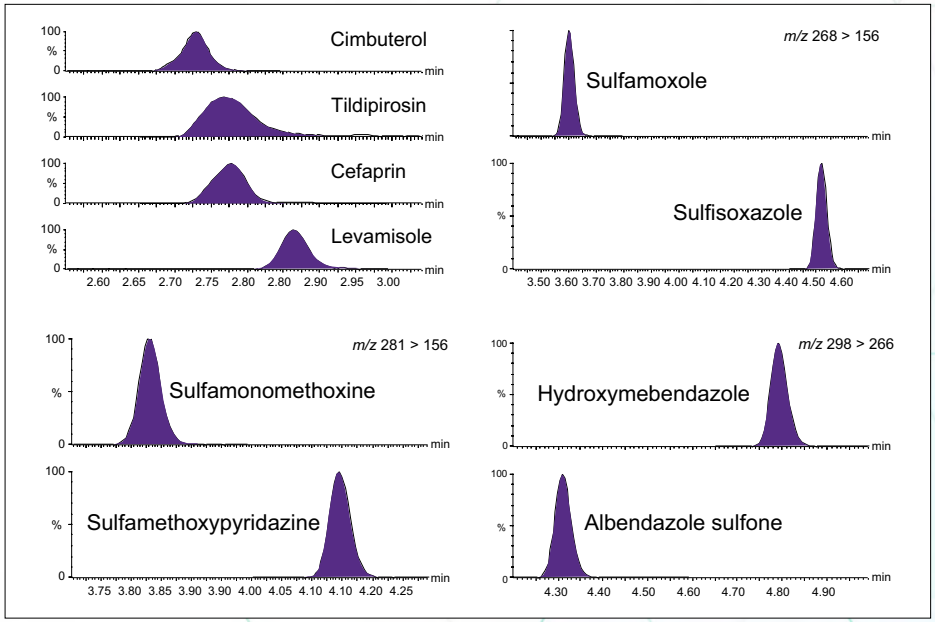
Veterinary Drug Residues Multi-Residue Screening Based Upon Liquid Chromatography-Tandem Quadrupole Mass Spectrometry

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 using electrospray and polarity switching. Despite using generic conditions, a UPLC System can provide increases in sample throughput by speeding up analysis times, and maintain good peak shape across a wide range of different compounds, ensuring 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.

Read the Full Application Note

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 of the method for screening purposes



A selection of chromatograms from analysis of a matrix-matched standard in bovine muscle extract at 10 µg/kg showing retention of some polar compounds and separation of isobaric compounds.

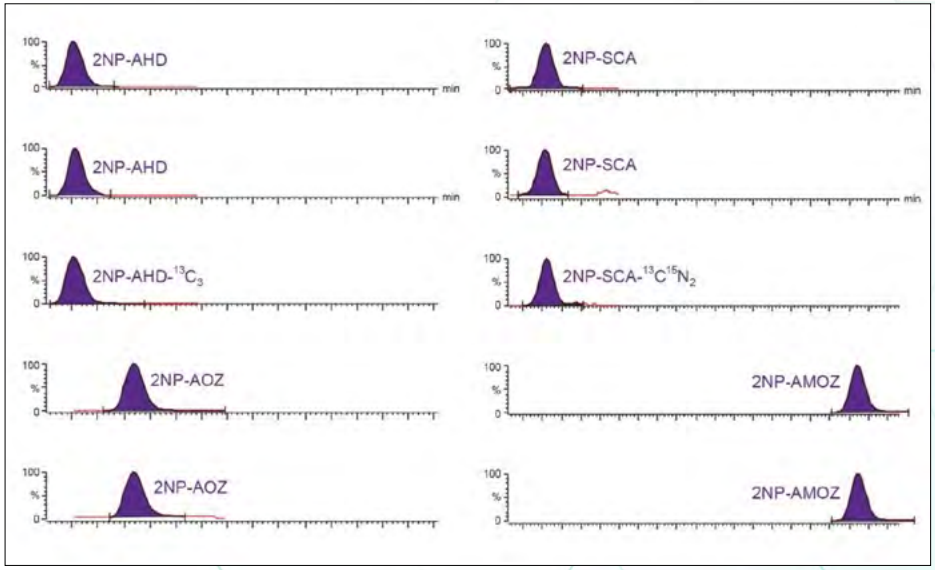
Determination of Nitrofuran Metabolites and Chloramphenicol in Chicken Muscle at Regulatory Limits

The use of certain antibacterial compounds in livestock and aquaculture production for human consumption is prohibited due to concerns about their toxicity in the major global geographies, according to regulations established by the EU, Food and Drug Administration, CODEX Alimentarius, and Joint FAO/WHO Expert Committee on Food Additives (JECFA). Here we describe the method and report the performance characteristics of the ACQUITY UPLC I-Class PLUS System coupled to the Xevo TQ-S cronos for the quantitation of banned veterinary residues (chloramphenicol and four nitrofuran metabolites) in food of animal origin. The calibration characteristics and repeatability were all shown to be suitable for the determination of residues at the respective EU Reference Point for Action in chicken muscle. The results of the performance evaluation show the Xevo TQ-S cronos to be robust for routine operation, with minimal operator intervention or maintenance during extended periods of analyses even for the challenging small, relatively polar analytes in complex extract.

Read the Full Application Note

APPLICATION BENEFITS

- Reliable, routine quantitative analysis of banned drug residues in foods of animal origin combined with Oasis solid phase extraction 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



Example of chromatogram obtained for nitrofurans (0.5 µg/kg) and chloramphenicol (0.15 µg/kg) in chicken.

Tetracycline and Sulfonamide Antibiotics in Shrimp Tissue using Liquid Chromatography Tandem Quadrupole Mass Spectrometry

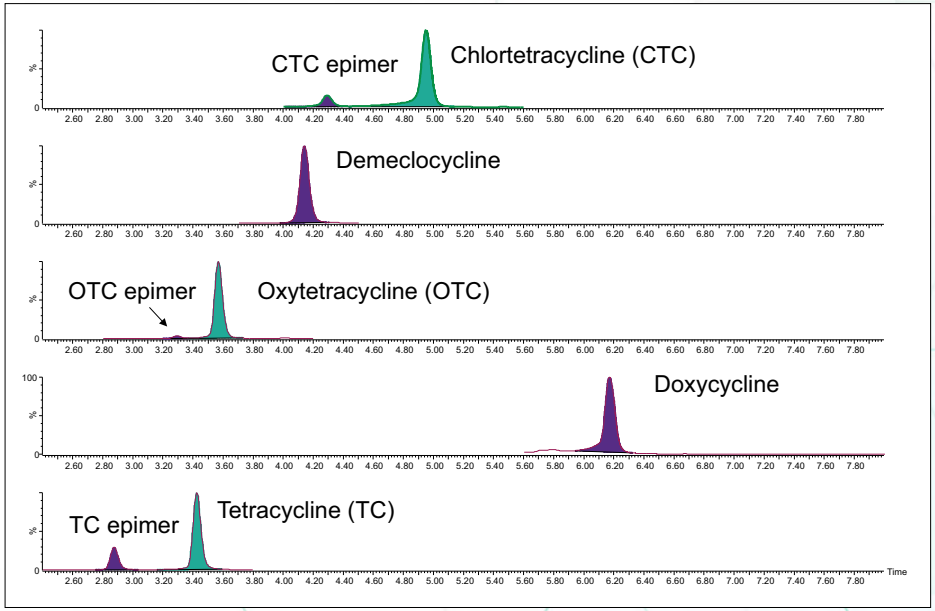
Veterinary drugs are used in animal husbandry and aquaculture for therapeutic or disease-preventive reasons and, in some cases, to promote growth of livestock. However, when specified withdrawal periods are not observed, unsafe antibiotic residues, or their metabolites, may be present in edible products such as milk, eggs, and meat. 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.

This application note describes the results of a successful validation of the analysis of shrimp tissue for tetracyclines, sulfonamides, trimethoprim, ormetoprim, and dapsone using the Waters ACQUITY UPLC I-Class PLUS System coupled to the Xevo TQ-S micro.

Read the Full Application Note

APPLICATION BENEFITS

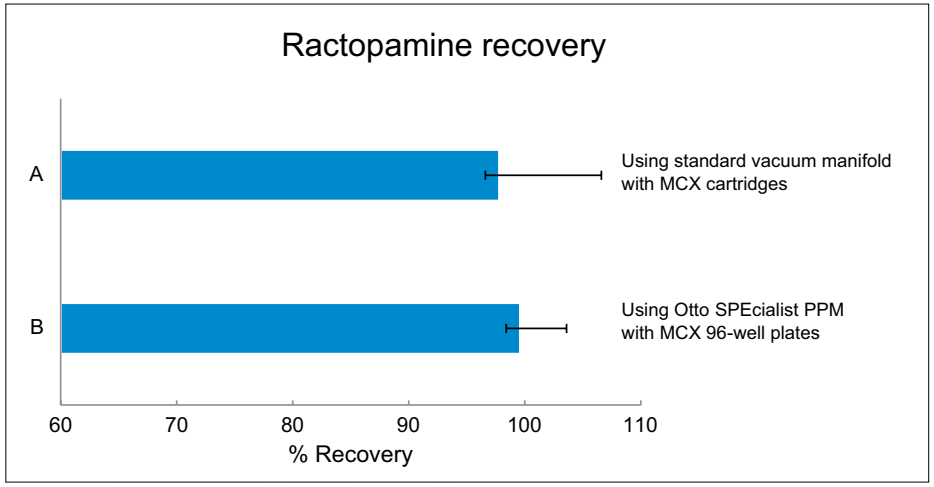
- Combining a wide range of tetracyclines, sulfonamides, and related antibiotic veterinary drugs into a single analysis
- Effective clean-up using small injection volumes and a sensitive mass spectrometer combine to provide a robust and reliable analytical solution
- Demonstration of successful validation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



Chromatogram of a matrix-matched standard at the MRL showing separation of the tetracyclines.

Improved SPE for LC-MS/MS Determination of Ractopamine in Porcine and Bovine Liver: The Oasis MCX Method Using Otto SPEcialist

This application brief describes a simple, rapid, and effective clean-up strategy to remove co-extractives from porcine and bovine liver extracts. This is executed via Oasis MCX 96-well SPE plate processed using Otto SPEcialist, a semi-automated positive pressure manifold prior to UPLC-MS/MS quantification of total ractopamine, a beta-agonist veterinary drug, with a limit of detection of 0.1 ng/g. This method quantifies ractopamine and ractopamine-glucuronide metabolites to accurately measure ractopamine in animal tissue. The use of Otto SPEcialist to process samples in 96-well plate format not only increases sample throughput and reproducibility, but also eliminates the risk of cross contamination when using manual vacuum manifold.



Comparison of recovery data from spiked porcine liver samples between (A) using standard vacuum manifold with Oasis MCX Cartridges and (B) using Otto SPEcialist semi-automated positive pressure manifold (PPM) with Oasis MCX 96-well plates

APPLICATION BENEFITS

- Processing samples using Otto SPEcialist with Oasis MCX in 96-well plate format provides improved clean-up of porcine liver extracts with high recovery of target beta-agonist veterinary drugs
- The Otto SPEcialist positive pressure manifold improves workflow, data turnaround, and allows analysts more time for other responsibilities, while simultaneously improving repeatability between analysts and day-to-day improvement
- Samples prepared using the Otto SPEcialist in 96-well plate format had increased area count and signal-to-noise ratios compared to a modified method processed using a manual vacuum manifold in SPE cartridge format

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Supporting a Safe and Compliant Food Chain

Single residue and class specific methods.

Multi-residue screening and/ or confirmatory methods by LC-MS/MS.

Accurate mass screening, metabolite identification and elucidation by LC-HRMS.

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Mycotoxins

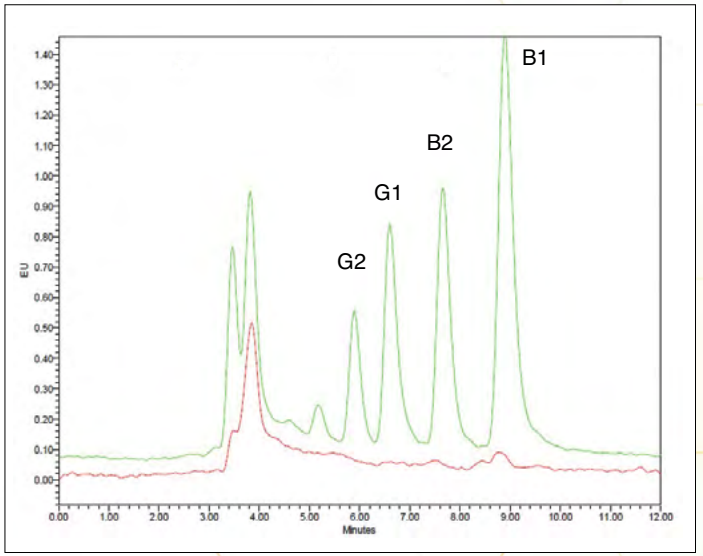
Determination of Aflatoxins Using Immunoaffinity Chromatography Column Clean-up Coupled with LC with Fluorescence Detection

Aflatoxins are carcinogenic mycotoxins that have adverse health effects on both humans and animals consuming contaminated food and feed, respectively. A method has been developed for the highly sensitive and selective determination of regulated aflatoxins in a wide range of commodities. The extraction of aflatoxins from representative commodities of interest (nutmeg, red chili, black pepper, cocoa, roasted coffee, dog food, and a traditional Chinese medicine) was performed using liquid-liquid extraction and then immunoaffinity column clean-up on the AflaTest™ WB SR+ Column. Chromatographic separation was demonstrated using both HPLC (Alliance™) and UPLC (ACQUITY UPLC H-Class PLUS) platforms, using fluorescence detection, supported with post-column derivatization and large flow cell, respectively. The performance of the method was evaluated through replicate analysis of spiked test portions of seven different matrices. Overall recovery was shown to be satisfactory, between 82% and 119%, with relative standard deviations lower than 8%. The method was found to be specific as no interference peaks were observed for blank samples. The method has been demonstrated as suitable for monitoring compliance with regulatory limits set for aflatoxins in food commodities globally.

Read the Full Application Note

APPLICATION BENEFITS

- High performance - the method meets AOAC method performance requirements
- Comprehensive - VICAM's AflaTest WB SR+ Column binds aflatoxin B1, B2, G1, and G2
- Flexible - one standardized method is suitable for analysis of a range of different commodities
- Inject direct – option to avoid handling of derivatization solutions and analyze directly using the large flow cell Fluorescence detector



HPLC chromatograms from analysis of black pepper before (red) and after spiking (green) with 4.0 µg/kg total aflatoxin.

Determination of Aflatoxins and Ochratoxin A Using VICAM Immunoaffinity Chromatography Clean-up with UPLC-MS/MS

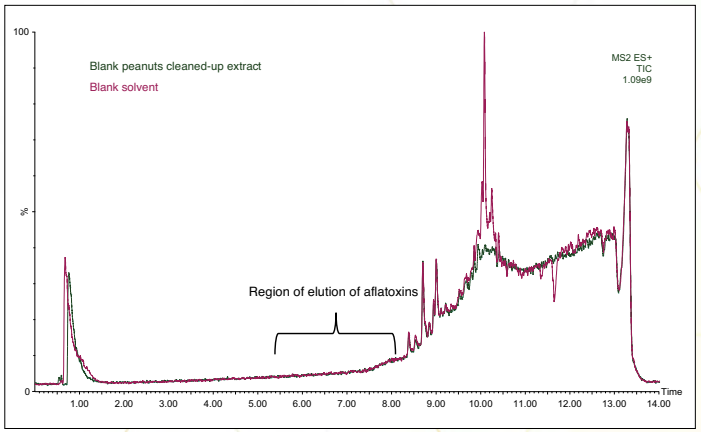
In this application note we describe three different methods for specific food commodities:

- Aflatoxins in groundnuts, pistachio, and hazelnuts using AflaTest WB clean-up
- Ochratoxin A in roasted coffee and cocoa using OchraTest WB clean-up
- Aflatoxins and ochratoxin A in black pepper using AflaOchra clean-up

All the mentioned methods have been internally validated using spiked samples as well as naturally contaminated reference materials, where available, and are based upon a solid-liquid extraction, followed by a trap-and-release step using immunoaffinity chromatography (IAC) and LC-MS/MS analysis. The methods provide optimal performance in terms of trueness and repeatability, with method LOQs as low as 0.050 µg/kg for aflatoxins and 0.4 µg/kg for ochratoxin A. The use of IAC clean-up is particularly advantageous as it allows the use of calibration curves based on solvent standards, removing the need to prepare closely matrix-matched calibration standards and without the need for (isotopically labelled) internal standards. Moreover, it provides excellent recoveries (in the range 71–108%, mean 90%) due to the highly specific antibody-based binding, and minimizes the potential matrix effects, thus making it suitable to be coupled with tandem quadrupole mass spectrometry.

APPLICATION BENEFITS

- Highly selective methods for targeted mycotoxins in complex matrices with very low method-LOQs for aflatoxins and ochratoxin A in all matrices
- High repeatability of the methods (i.e. AflaOchra on black pepper gives RSDr <5%)
- Minimal matrix effects, allowing for commodity independent (solvent based) calibration curves to be employed and no need for isotopically-labelled internal standards



Overlaid chromatograms of a peanuts cleaned-up extract (green) and a blank solvent (red) acquired in ESI+ SCAN (scan time: 0.1 s; acquisition: 50–800 m/z).

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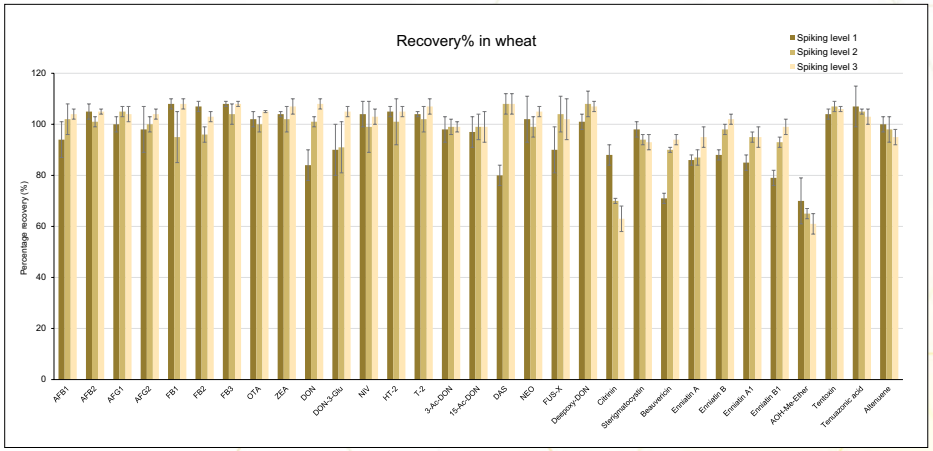
Determination of Regulated and Emerging Mycotoxins in Cereals, Nuts, Figs, and Animal Feeds Using Pass-Through SPE and UPLC-MS/MS

Mycotoxins can occur naturally in a variety of food products via pre- and post-harvest contamination mechanisms and are regulated worldwide. Herein we describe a multi-toxin method for 31 regulated and emerging mycotoxins using a quick and simple pass-through SPE clean-up prior to LC-MS/MS analysis for cereal-based products, ground- and tree-nuts, dried figs, and animal feeds. The method has been developed to maximize recovery and enhance ease-of-use. The performance of the method has been validated using spiked samples and verified using reference materials, where available. The method provides very good performance in terms of trueness and repeatability, with method LOQs as low as 0.25 µg/kg for aflatoxins and 1.0 µg/kg for ochratoxin A. The use of Oasis PRiME HLB SPE clean-up is particularly advantageous as it involves a simple and quick pass-through protocol, thus eliminating the SPE conditioning, equilibration, and washing steps. Recoveries were excellent for all analytes (in the range 60–108%) which demonstrates the wide scope of the clean-up protocol. This is important as it allows to expand the method to a wider range of compounds if needed.

APPLICATION BENEFITS

- Single multi-mycotoxin method for a wide range of complex matrices with the removal of major co-extractives using pass-through SPE clean-up
- Good method performance fulfilling regulatory requirements for all tested mycotoxins
- Possibility to scale-up the method to a wider number of analytes and different food and feed matrices



Bar-plot showing mean percentage recoveries (Rec%) for the tested mycotoxins in wheat flour at three spiking levels. Error bars representing percentage relative standard deviation (%RSDr).

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Evaluation of the Performance of a Simple Method for Regulated Mycotoxins in Cereals by LC-MS/MS Using an Interlaboratory Study

Waters previously reported the development and single laboratory validation of a method for the determination of the 12 mycotoxins regulated in the EU in various cereals based upon LC-MS/MS after a simple generic extraction method without any clean-up. This application brief shows the successful evaluation of the performance of this method by interlaboratory study. Two cereal FAPAS QC materials were sent to four laboratories in Europe and the USA. Each material was analyzed in triplicate by the four laboratories. The laboratories demonstrated good accuracy and precision for the determination of the 8 mycotoxins in the two FAPAS QC materials. Reported concentrations matched the assigned values provided by FAPAS. Trueness was within the range of 85 to 113%, the within-laboratory repeatability was between 3.0 to 13% and between laboratory reproducibility was between 3.1 and 23%.

These results, which meet performance criteria set out by the European Commission, demonstrate the method is suitable for the monitoring of mycotoxins in cereals for both official control and testing conducted by food business operators.

APPLICATION BENEFITS

- Simple to implement analytical method covering extraction to determination
- A rapid, simple sample preparation strategy and LC-MS/MS quantitative method for compliance with regulatory limits and method performance guidelines
- Comparable method performance across multiple laboratories in different geographical locations

T04395QC	Aflatoxin B1	Ochratoxin A	HT-2 Toxin	T-2 Toxin	Zearalenone	Deoxynivalenol	Fumonisin B1	Fumonisin B2
Internal standard	U-[$^{13}C_{17}$]-AFB1	U-[$^{13}C_{20}$]-OTA	U-[$^{13}C_{22}$]-HT-2	U-[$^{13}C_{24}$]-T-2	U-[$^{13}C_{18}$]-ZEA	U-[$^{13}C_{15}$]-DON	U-[$^{13}C_{34}$]-FB1	U-[$^{13}C_{34}$]-FB2
Assigned values (µg/kg)	9.22	2.69	215	209	95.8	948	287	273
Mean of the measured values (µg/kg)	9.08	2.38	199	186	94.4	878	246	309
Range for [z] ≤2 (µg/kg)	5.26–13.3	1.51–3.87	128–302	125–294	53.7-138	643–1254	176–397	167–370
Range of measured values(µg/kg)	8.28–10.5	2.19–2.60	185–229	163–231	86.5–97.7	813–1004	222–284	276–356
Trueness (%)	98.5	88.6	92.5	88.8	98.5	92.6	85.8	113
Within lab repeatability (% RSDr)	7.39	4.03	4.88	6.36	3.14	4.22	6.04	3.22
Between lab reproducibility (% RSD_{RL})	7.83	4.51	6.35	10.6	3.12	7.27	20.9	16.9

Results of analysis of FAPAS QC materials TO4395QC by the four participating laboratories.

Read the Full Application Note

From Farm to Lab: The Right Tools, at the Right Time

Raw Material Processor

Pre-Harvest and Post-Harvest Screening

Simple to use screening strip tests

Crop Producer and Trader

Raw Materials Preventive Monitoring

Qualitative and quantitative rapid tests

Food and Feed Manufacturer

Processing, Co-Products & Finished Product Testing

Quantitative lateral flow tests and fluorometry
Confirmatory LC & LC-MS/MS testing

Contract and Reference Labs/ Government

Quality and Compliance Verification

Validated methods using LC and LC-MS/MS for high-throughput single and multi-mycotoxins analysis

>>Click here to read the infographic.


Alkaloids

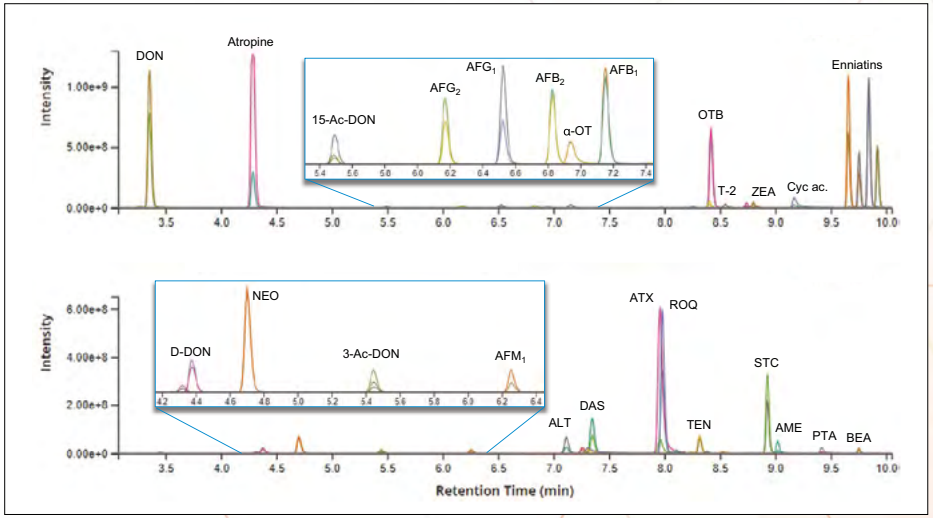
Development of a Multi-Toxin UPLC-MS/MS Method for 50 Mycotoxins and Tropane Alkaloids in Cereal Commodities

In this application note we describe the performance of a multi-toxin UPLC-MS/MS method for 50 regulated and emerging mycotoxins, atropine, and scopolamine in cereal-based products. The tandem quadrupole mass spectrometer, the Xevo TQ-XS, was used in combination with an ACQUITY UPLC System to reach very low limits of detection and quantification. A mix of cereal flours was extracted using a simple "dilute-and-shoot" protocol, without any clean-up or internal standards. Calibration curves were plotted using solvent standards and spiked extract (matrix-matched calibration). Limits of detection and quantification were shown to be suitable for checking compliance with regulatory limits and to investigate the levels of other toxins. The lowest method limit of quantification (m-LOQ) was for aflatoxins (0.1 µg/kg). The calibration range was acceptable and covered three orders of magnitude for most analytes. Matrix effects were also calculated for all compounds and were found to be significant, illustrating the need for matrix-matched calibration. The method fulfilled the criteria set out in the SANTE guidelines for mycotoxins. Data was imported into the waters_connect™ for quantitation software and processed with the MS Quan app for an improved efficiency in data processing and review.

Read the Full Application Note

APPLICATION BENEFITS

- Simultaneous determination of more than 50 mycotoxins and plant toxins in a single LC-MS/MS method
- Low limits of detection are achieved using the high sensitivity Xevo TQ-XS
- Remarkable sensitivity and good linearity and repeatability were obtained, thus fulfilling regulatory requirements
- The MS Quan application within waters_connect for quantitation software reduces the time taken to process data and review results



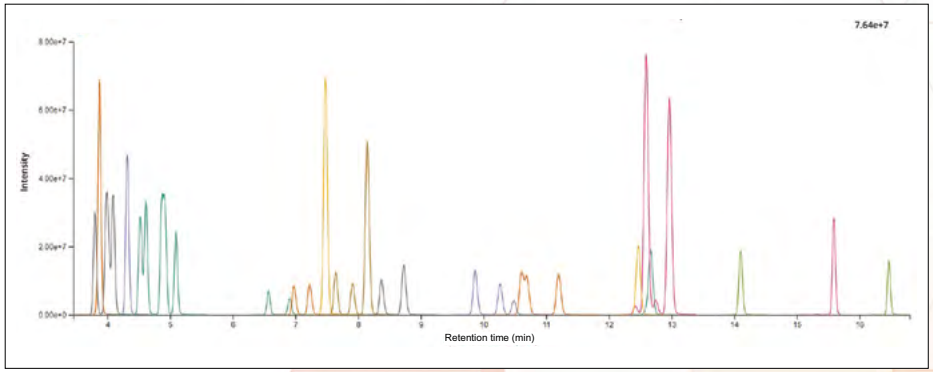
Chromatograms of the tested mycotoxins and plant toxins in two different spiked cereal samples. Each peak is characterized by a quantifier and a qualifier ion trace.

Determination of Pyrrolizidine Alkaloids in a Range of Plant-Based Foods and Honey Using LC-MS/MS

Pyrrolizidine Alkaloids (PAs) are toxins exclusively biosynthesised by plants. Expressing both genotoxic and carcinogenic properties, an increasing number of reports reveal relatively high contaminations with PAs in food, herbal infusions, and teas. This application note describes an analytical method for the determination of PAs in a variety of food commodities (tea, herbs, spices, cumin seeds, and honey). Samples were extracted with a sulfuric acid solution, cleaned up using Oasis MCX SPE Cartridges, concentrated and resuspended prior to liquid chromatography with tandem quadrupole mass spectrometry (LC-MS/MS) analysis. The chromatographic resolution of critical pairs of isomers was addressed in this study. Good method recoveries and excellent repeatability were obtained, which complied with the acceptance criteria in the CEN standard for single laboratory validation. Limits of quantification for individual compounds were 0.6 µg/kg, which were shown to exceed regulatory compliance, such that the method can also be applied to food intended for infants and young children.

APPLICATION BENEFITS

- Quantitative method for the analysis of 35 EU-regulated Pyrrolizidine Alkaloids in plant-based foods and honey suitable for checking regulatory compliance
- Removal of major co-extractives using Oasis MCX SPE clean-up to decrease the amount of isobaric interference
- Simplify and accelerate the development of robust methods using RADAR, the unique feature of Xevo tandem quadrupoles
- Good method recoveries and excellent repeatability, which complied with the acceptance criteria in the CEN standard for single laboratory validation
- Very low limits of quantification demonstrate the potential to employ the method for the analysis of Pyrrolizidine Alkaloids in products for infants and young children where maximum levels can be as low as 75 µg/kg for the sum of PAs



Representative chromatogram of the 35 pyrrolizidine alkaloids (matrix-matched standard in tea extract, 250 µg/kg).

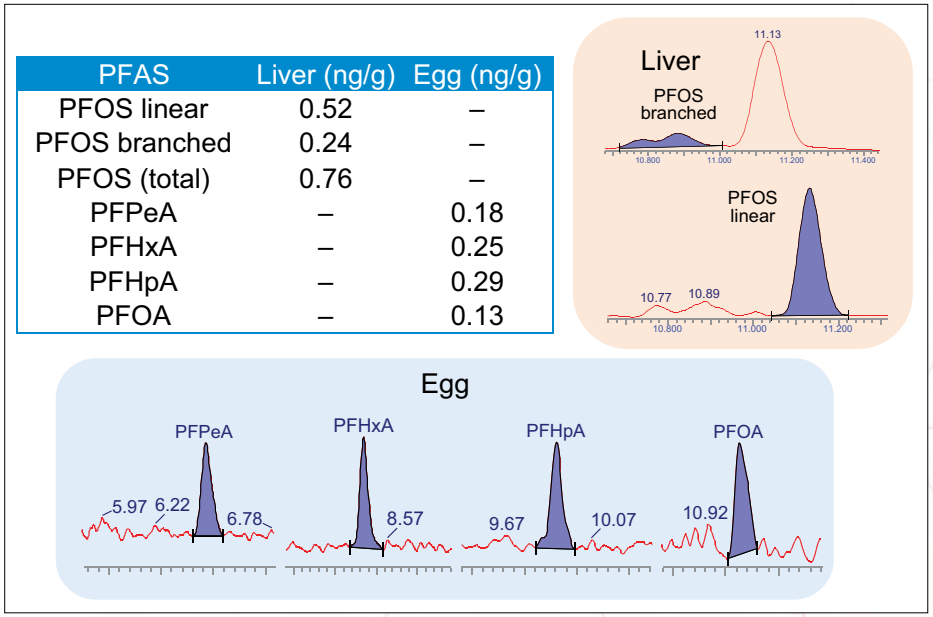
Read the Full Application Note


PFAS

Total Workflow for the Sensitive Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Fish, Meat, Edible Offal, and Eggs

Environmental release and contamination of per- and polyfluorinated substances has resulted in contamination of a variety of food sources. Complex food commodities such as fish, meat, edible offal, and eggs require a comprehensive sample extraction and clean up. To accommodate these types of samples, an alkaline digestion and extraction was implemented followed by Weak Anion Exchange (WAX) SPE to produce a suitable sample for analysis. High sensitivity LC-MS/MS analysis was performed on an ACQUITY UPLC I-Class PLUS coupled to Xevo TQ-XS. The method was evaluated in six different commodity types including salmon, tilapia, ground beef, beef liver, beef kidney, and chicken (hen) eggs. This approach proved to be accurate, sensitive, and robust for a range of 30 PFAS compounds of varying chemistry classes to match the challenging concentrations published in reports by the European Food Safety Authority (EFSA) and the US Food and Drug Administration (FDA).

PFAS	Liver (ng/g)	Egg (ng/g)
PFOS linear	0.52	–
PFOS branched	0.24	–
PFOS (total)	0.76	–
PFPeA	–	0.18
PFHxA	–	0.25
PFHpA	–	0.29
PFOA	–	0.13



PFAS detected in samples of beef liver and egg purchased in local grocery stores.

APPLICATION BENEFITS

- A single extraction method that can be utilized for a large suite of PFAS from a variety of food matrices
- Sensitive analysis on the Xevo TQ-XS to detect PFAS at sub-ng/g levels to match the challenging concentrations published in reports by EFSA and FDA
- Confidence in results with the utilization of the PFAS Kit for LC modification to minimize possible system and solvent contaminants and assure accurate results

Read the Full Application Note

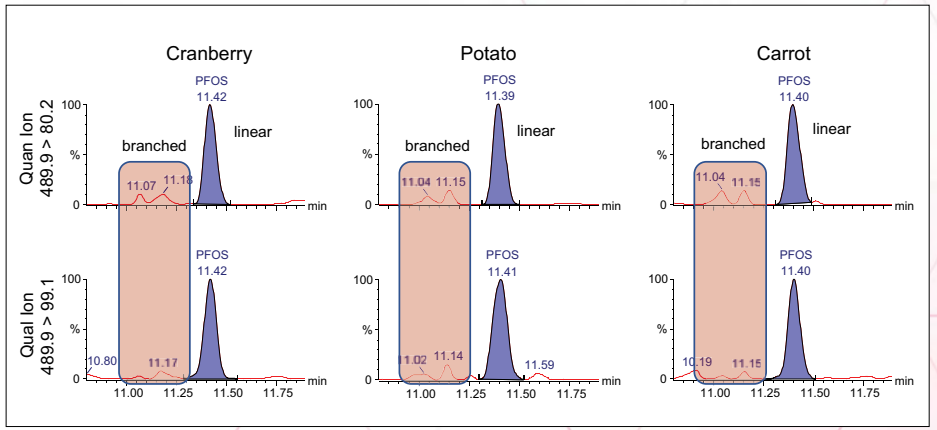
QuEChERS Extraction of Per- and Polyfluoroalkyl Substances (PFAS) from Edible Produce with Sensitive Analysis on Xevo TQ-XS

Sources of environmental per- and polyfluorinated substances exposure can lead to contamination in food sources. Cultivating produce using PFAS contaminated water and soils can lead to the uptake of these compounds into the edible fruits and vegetables portions of plants. Thus, it is beneficial to have a straightforward method to monitor the occurrence of PFAS in produce. For this work, the FDA C-010.01 method based on the QuEChERS method was implemented for extraction of PFAS and sample clean-up using DisQuE extraction and dispersive solid phase extraction (dSPE) products followed by LC-MS/MS analysis on ACQUITY UPLC I-Class PLUS coupled to Xevo TQ-XS. The method was evaluated in five different commodity types including lettuce, strawberry, cranberry, carrot, and potato. With a few minor adjustments to the FDA method, this approach to PFAS analysis in produce proved to be accurate and robust for a range of 30 PFAS compounds of varying chemistry classes.

Read the Full Application Note

APPLICATION BENEFITS

- A time efficient and simple extraction of PFAS from edible produce utilizing a QuEChERS extraction method and dSPE clean-up
- Sensitive analysis on the Xevo TQ-XS to detect PFAS at sub-ng/g levels to match detected concentrations published in reports by EFSA and FDA
- Confidence in results with the utilization of the PFAS Kit for LC modification to isolate possible system and solvent contaminants

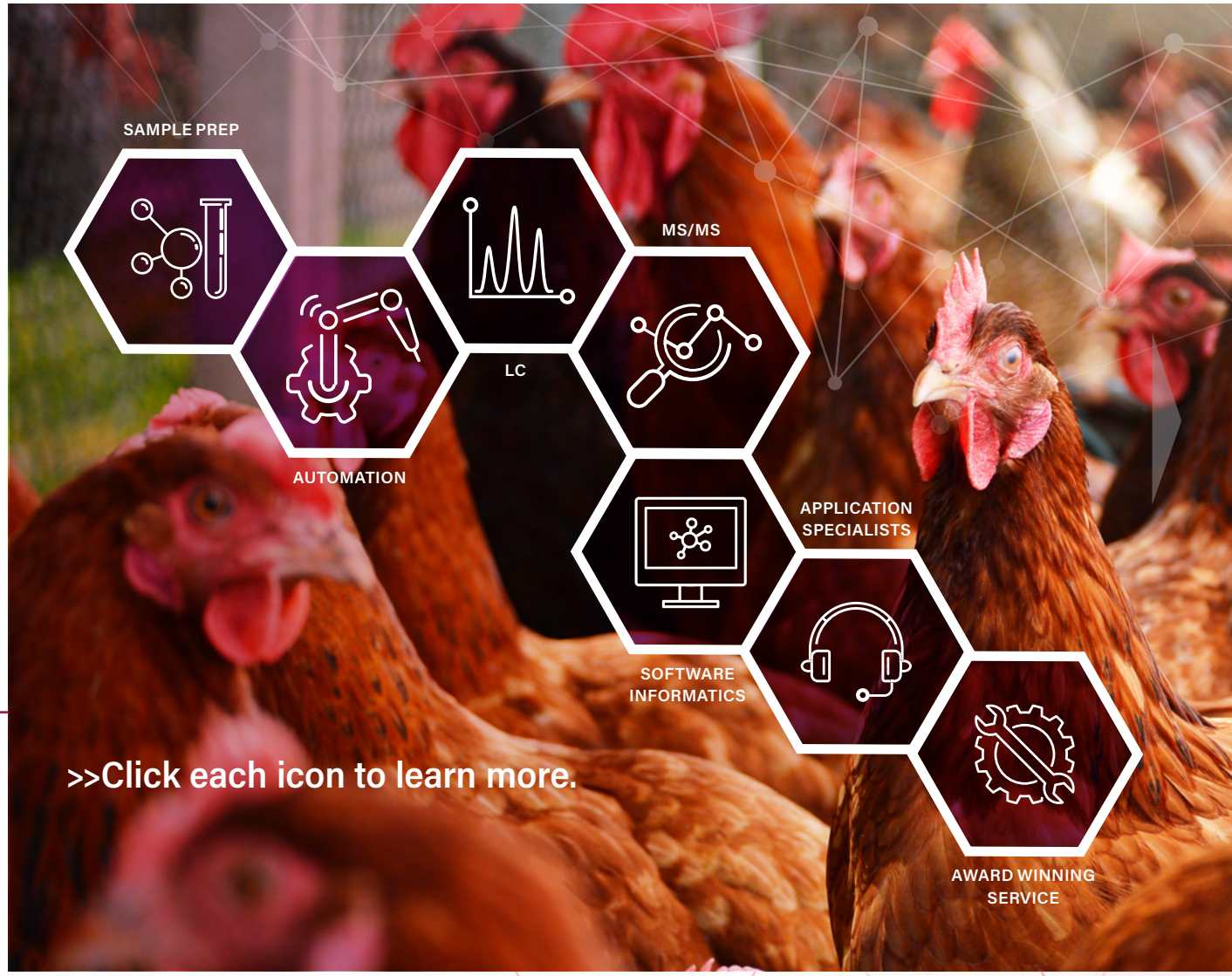


Detection of branched and linear PFOS isomers in 0.05 ng/g cranberry, potato, and carrot matrix.

Perfect Partnering for PFAS Analysis

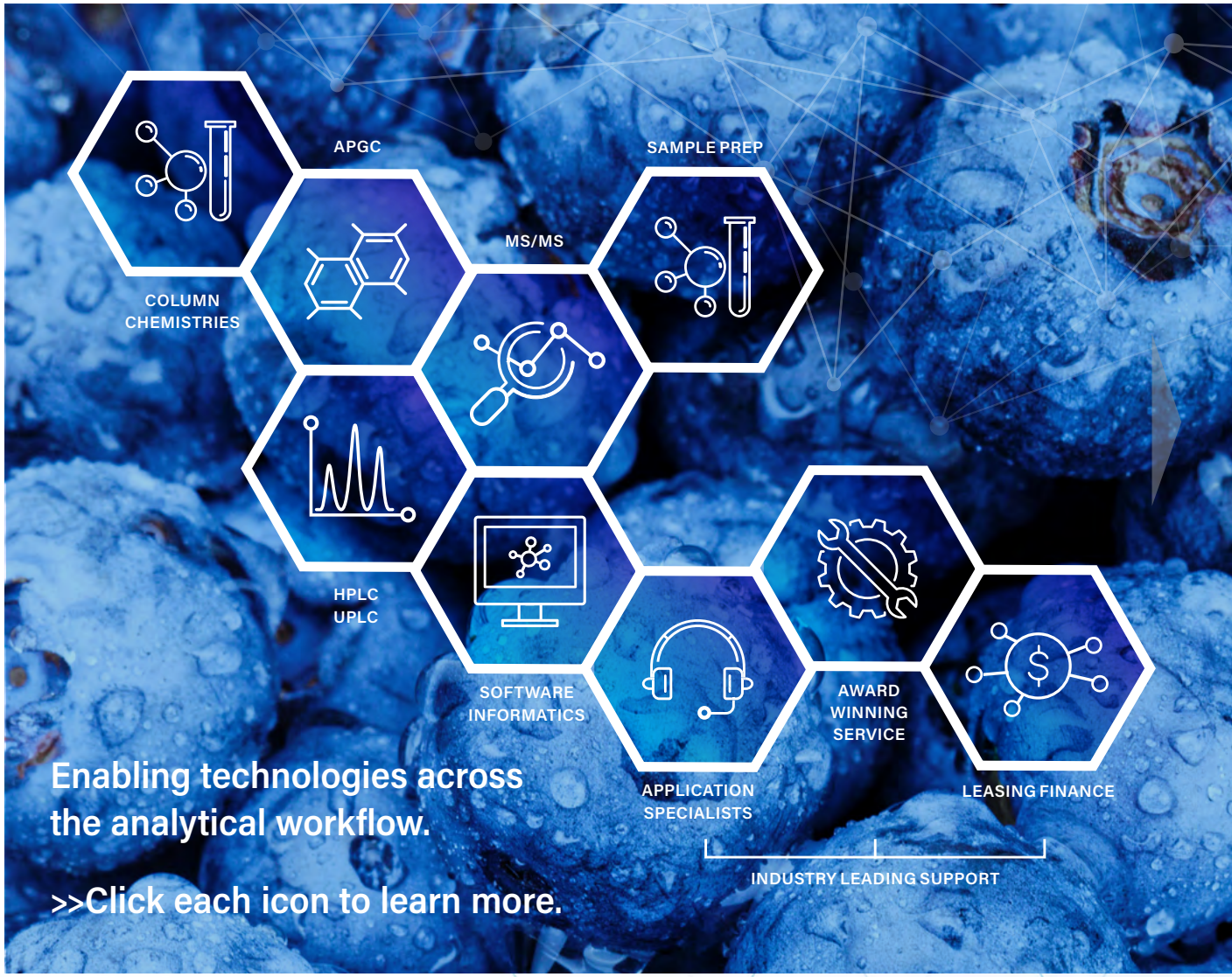
PFAS Solution Installation Kits help minimize and separate PFAS system contamination from analytes of interest. Compatible on ACQUITY family of LCs coupled with tandem quadrupole and high resolution mass spectrometry systems and supported by our award winning service, discover how Waters can help your laboratory get to grips with PFAS analysis today.

>>Click here to learn more about PFAS Analysis Solutions.



Links to other Useful Materials

- An Overview of Multi-residue Pesticide Testing
- Improve the Robustness of an LC-MS/MS Method for the Determination of Multiple Mycotoxins in a Range of Food Matrices
- The Benefits of waters_connect MRM Processing Application, MS Quan
- Carryover in UPLC Methods: The Case Study of Fumonisin B2 White Paper
- Improving Negative Ion Detection for Tandem Quadrupole Mass Spectrometry
- Matrix Matching or Isotope Dilution? A Comparison of Two Quantitation Approaches
- The Benefits of ACQUITY Premier UPLC for Multi-Mycotoxin Methods



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