the Analytical Scientist

MASS SPECTROMETRY BREAKS NEW GROUND

From investigating food flavors to tackling toxicity in the clinic, mass spectrometry is being applied in more fields than ever. Here, we present nine application notes highlighting the diversity of mass spectrometry applications in 2018.

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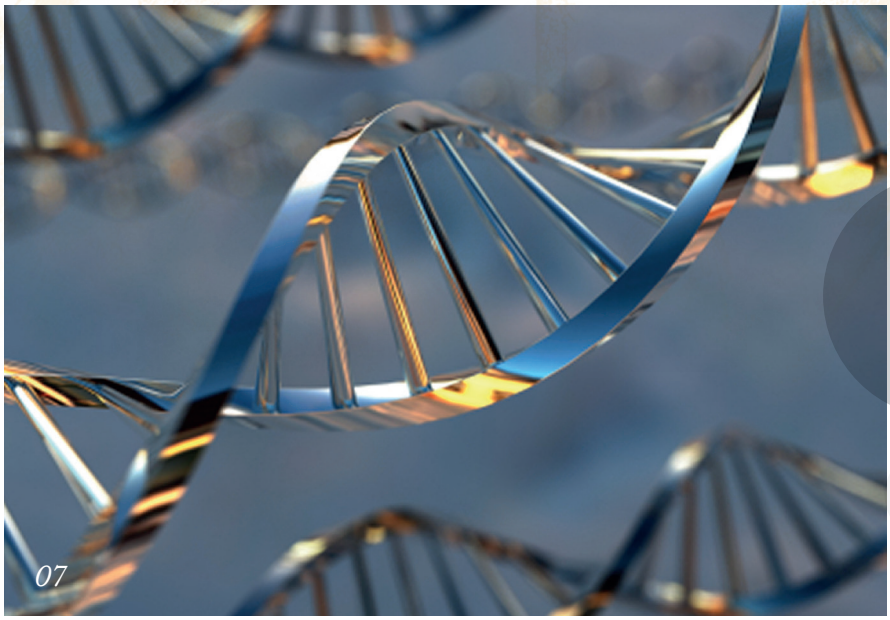
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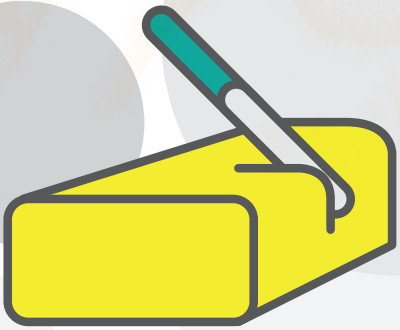

RETHINKING LIBRARY IDENTIFICATION IN QUANTITATIVE CLINICAL TOXICOLOGY – TRANSITIONING TOWARDS MRM SPECTRUM MODE

Applying "MRM Spectrum Mode" and library searching for enhanced reporting confidence in toxicology screening and quantitation.

By Stéphane Moreau, Neil Loftus, Tiphaine Robin, and Alan Barnes

The majority of clinical decisions are based on laboratory test results. For many laboratories, triple quadrupole MRM methods are used to deliver highly sensitive, selective and robust results for precise quantitation and identification verification. To help transition towards a more effective data review and higher confidence in reporting results, we have been rethinking the capability of MRM in compound identification and verification. In this workflow, 6–10 fragment ion transitions were monitored for each target compound as opposed to a conventional approach using 2–3 fragment ions. By acquiring a high number of fragment ion transitions, each target compound had a corresponding fragmentation spectrum which could be used in routine library searching and compound verification using reference library match scores. In this work, we compare different approaches in target quantitation and identification applied to clinical and forensic toxicology.

Whole blood was spiked with a panel of CAO compounds (cocaine, antipsychotics, amphetamines, opiates). Calibration samples and unknown samples were prepared by QuEChERS method with the inclusion of stable isotope standards. In this work, MRM Spectrum mode acquired a library of typically six or more MRMs per compound using certified reference materials. The library included not only MRM transitions for each target compound but also retention time (and relative retention time for each internal standard) and metadata including CAS number, formula and synonyms. For comparison, full-scan library spectrum data was also acquired using a MRM triggered product ion spectra for three collision energies corresponding to CE 10, 35 and 55V, as well as a fourth merged CE spectrum totaling 6084 registered spectra.

To minimize the possibility of false reporting without compromising accuracy, precision and limits of detection,

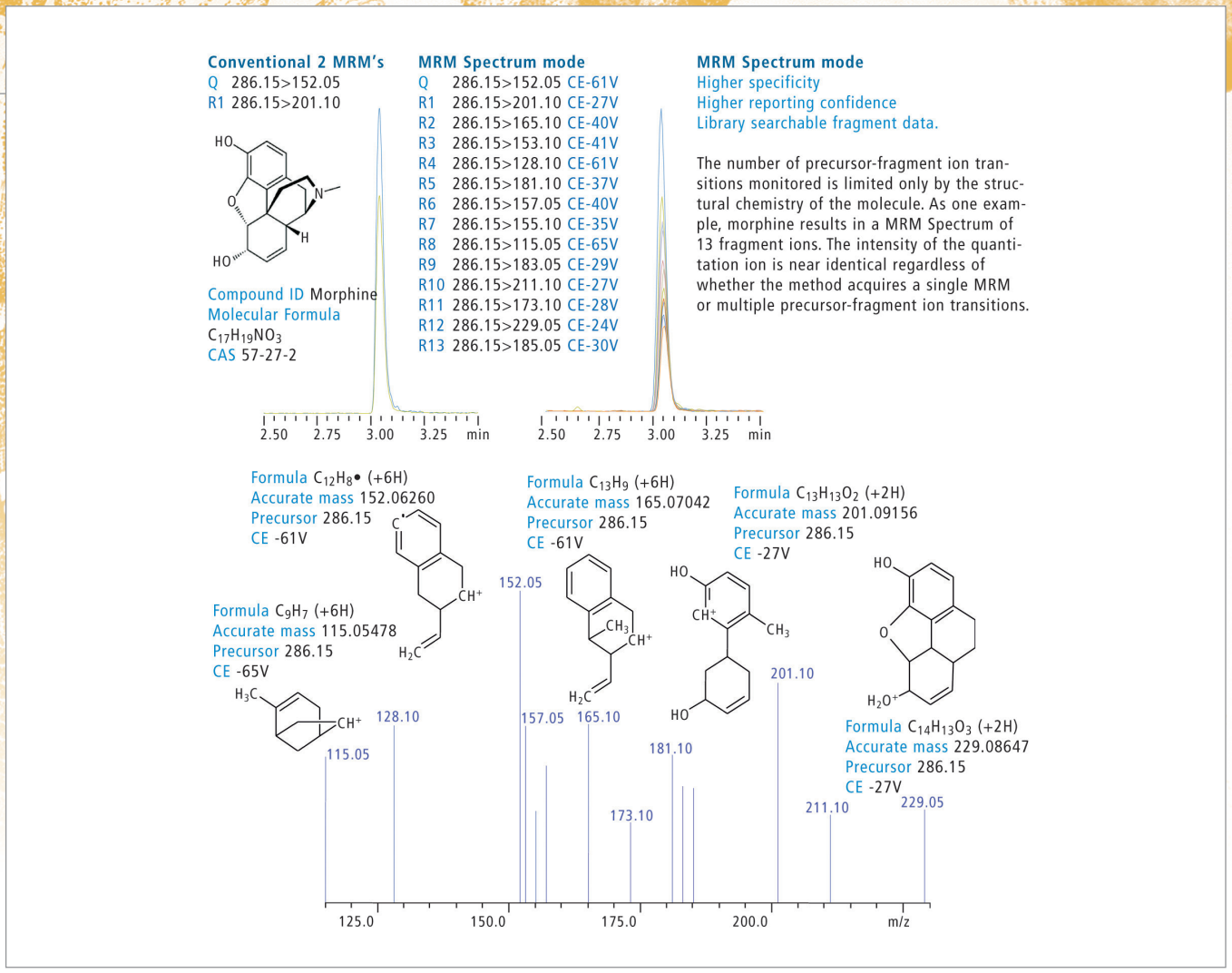

Figure 1. MRM reference spectrum for morphine with putative assigned fragment structures. MRM Spectrum mode combines MRM with the generation of a product ion spectrum. The product ion spectrum can be used for compound identification by searching a library. As the response to each precursor-fragment ion transition has been optimized for a specific collision energy, the MRM Spectrum is highly specific and generates strong signal intensities for each fragment ion. (Each precursor-fragment ion transitions structure was assigned using an in-house development tool (Structure Analytics) to show commonly described losses and charge migration; the hydrogen deficit is shown in brackets).

methods were developed to combine the sensitivity of MRM detection with the identification power of a product ion spectrum. The methods have the capability of simultaneously using both precursor and product ion information – enabling precise, accurate quantitation and library searchable compound identification. To assess the impact of methods designed to increase reporting confidence by library searching on quantitation, both product ion spectrum methods were compared to data generated using a conventional 2MRM method. For each target compound, the quantifier ion remains the same but the methods differ in information content and data density. To test the viability of this approach and to quantify and identify targets, the MRM triggered product ion spectrum acquisition method and MRM Spectrum mode were applied to a series of patient blood samples and compared with a validated LC-MS/MS method using two MRMs for each target compound. CAO compounds including internal standard compounds were acquired using three different MS/MS methods. In patient test samples, the concentration of each target analyte was nearly identical using the different MS/MS methods with library identification.

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INNOVATIVE FOOD SAFETY METHOD ADVANCES CONTAMINANT DETECTION IN AQUATIC-BASED FOOD SAMPLES

APGC-MS/MS expands the detection and quantification of contaminants in food.

Researchers at the Institute for Pesticides and Water (IUPA) at University Jaume I in Spain have been using atmospheric pressure gas chromatography (APGC) as a new technique for detecting and quantifying contaminants in aquatic-based food sources.

Brominated flame retardants (BFRs) are harmful and toxic compounds present in food – mainly seafood, since they are introduced into the oceans through waste, residues or discharges from the factories that produce them.

The European Union has limited or prohibited the use of many of these compounds, so monitoring these contaminants in food is essential to ensure that the levels do not exceed the allowable concentration.

Tania Portolés and researchers at IUPA coupled gas chromatography to triple quadrupole tandem mass spectrometry with an atmospheric pressure chemical ionization source (GC-APCI-MS/MS), specifically the Waters Xevo TQ-S with APGC, for GC-MS/MS analysis of PAHs, polychlorinated biphenyls (PCBs) and pesticides in 19 different matrices – including fish tissues, feeds, and feed ingredients.

The goal was to develop and test a method that would increase the number of contaminants detected, at much lower concentrations than previous methods reported, in a variety of food samples.

Amongst other benefits, this technique resulted in:

- The simultaneous quantification of 19 different complex matrices from aquaculture
- The dilution of all the 19 matrices tested
- The elimination of matrix effect, thereby removing the need for time-consuming purification steps
- Increased selectivity in the determination and identification of contaminants
- The ability to inject less sample matrix, reducing effects of contamination on the GC-MS system and therefore increasing uptime.

Tania Portolés in the Laboratory of Pesticide Residue Analysis, IUPA, UJI, Spain.

The IUPA study found the reduced fragmentation offered by APGC delivers a more robust and sensitive technique for analyzing a broad range of contaminants in several marine-based matrices, particularly compared to their previous methods using electron ionization (EI).

The implementation of this innovative atmospheric technique has great implications on food security, both to monitor food lots with a very high concentration of pollutants, and to monitor that they meet and comply with the standards regulated by the European Union.

OLIGONUCLEOTIDE ANALYSIS SUPPORTS EXCITING NEW THERAPEUTIC APPROACH

Employing a UPLC/PDA/QDa system for rapid oligonucleotide identity and impurity assessment with mass confirmation.

The market for synthetic oligonucleotides is experiencing high growth with key drivers being an expanding market for genetic-based diagnostics and a resurgence in the development of nucleic acid-based therapeutics.

As with any other organic compound, it is important to characterize oligonucleotides as they are synthesized. For Masanori Kataoka, CTO at S-NAC, the analysis of impurities and the measurement of protected nucleosides, mononucleotides, dimers, trimers, and full length oligonucleotides has never been more efficient. Utilising an ACQUITY UPLC/PDA/QDa workflow in Empower his group now tests over 200 samples per month.

Kataoka initially intended to use the ACQUITY UPLC/PDA/QDa workflow in Empower for a quick reaction check to confirm protection of nucleosides, but found its performance was better than expected, so he now uses the system for nucleotide trimer analysis and full length oligonucleotide analysis as well. He says "Before the ACQUITY UPLC/PDA/QDa workflow in Empower was available, we needed a separate standalone MS instrument to achieve mass confirmation, but having an integrated system gives us a streamlined, rapid and very cost effective approach. Importantly the system is easy to operate and its benchtop size allows us to use it in the conventional chemistry laboratory."

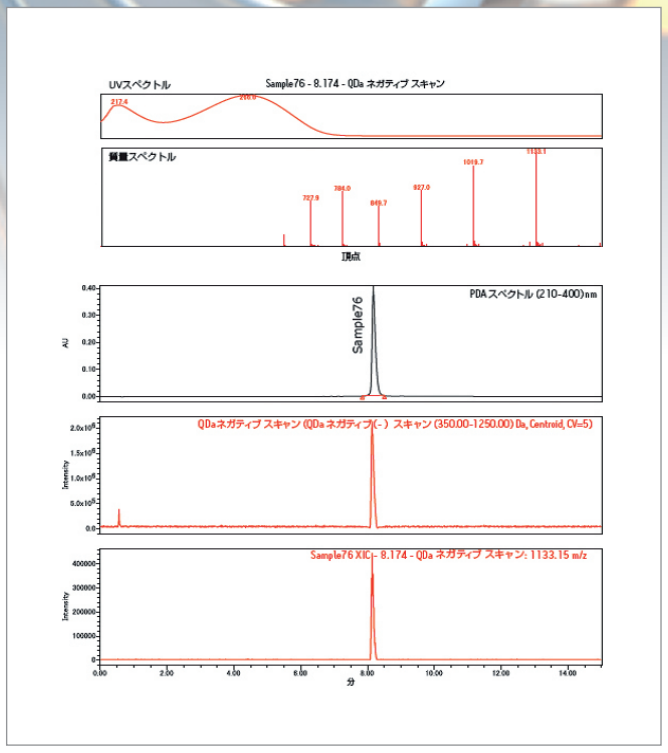

Figure 1. An example of the detection of polyvalent ions of nucleic acid using the QDa.

Prior to adopting the ACQUITY QDa Detector, Kataoka experienced significant difficulties detecting multi-valent ions of oligonucleotides. He explains that certain ions could not be confirmed with a good S/N ratio, and sometimes no peaks were detected in the measurement. They also found limitations in protecting groups at nucleobases and ribose moieties during monomer analysis, and of molecular weight for protected trimer analysis. In the case of oligonucleotides, there is a restriction to ionization, because multi-valent ion peaks are sensitive to the ion pair reagent used. The ACQUITY QDa has allowed the group to overcome these limitations (Figure 1) and can be used to detect and measure nucleic acids up to 30 bases or more in length, which covers the majority of oligo-based therapeutics now in development, as well as monomers.

Kataoka explains: "We have improved production efficiency by adopting liquid-phase synthesis and developing nucleotide segments. The improved efficiency does not always reduce the analysis time but with the QDa we are able to achieve rapid qualification analysis of synthetic products. We are working towards our overall business aim – to increase efficiency in two digits."

SPECIATION OF FATTY ACID METHYL ESTERS IN FOOD PRODUCTS

Flow-modulated GC×GC is used to analyse FAMEs from butter, with simultaneous detection by FID and TOF MS for improved speciation.

By Aaron Parker and David Barden

Research into fats in foods and their role in health depends upon reliable analysis of fatty acids and their triglycerides. To achieve this, lipid fractions are typically derivatised to fatty acid methyl esters (FAMEs), which are volatile enough to be analysed by GC, usually with FID detection. However, a common problem with one-dimensional GC is co-elution, which is usually only tackled by long (but expensive) columns, or by multiple analyses that decrease productivity.

To address this, analysts are increasingly using GC×GC to achieve efficient separation of homologues and isomers, and to benefit from the structured elution of analytes in bands ('roof-tiling'). In this study, we use an inverse-phase column set with a reverse-fill/flush flow modulator for robust, repeatable and affordable GC×GC, in conjunction with parallel detection by TOF-MS and FID.

Figure 1 shows the analysis of a butter extract (kindly supplied by NIHR BRC Nutritional Biomarker Laboratory, University of Cambridge, UK). The inset demonstrates how GC×GC improves speciation, by showing how some of the C_{14}–C_{18} monounsaturates would have closely eluted with the branched saturated analogues in a one-dimensional separation.

However, even when using TOF MS detection alongside FID to assist identification, certain FAMEs can be challenging to identify using standard 70 eV ionisation. The BenchTOF-Select model used here addresses this through its Tandem Ionisation® capability, which enables fast switching between 70 eV and low-energy 'soft' EI. This offers improved isomer speciation (with no user intervention or additional analysis time), as shown in Figure 2 for two C_{18} FAMEs. The 70 eV spectra are very similar, with a high degree of fragmentation and weak molecular ions, making it difficult to confirm the carbon chain length, but this is overcome in the 12 eV spectra.

For more information on this application, visit http://www.sepsolve.com/analysis-of-fames/

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Download the full application note: tas.txp.to/0518/SEPSOLVE

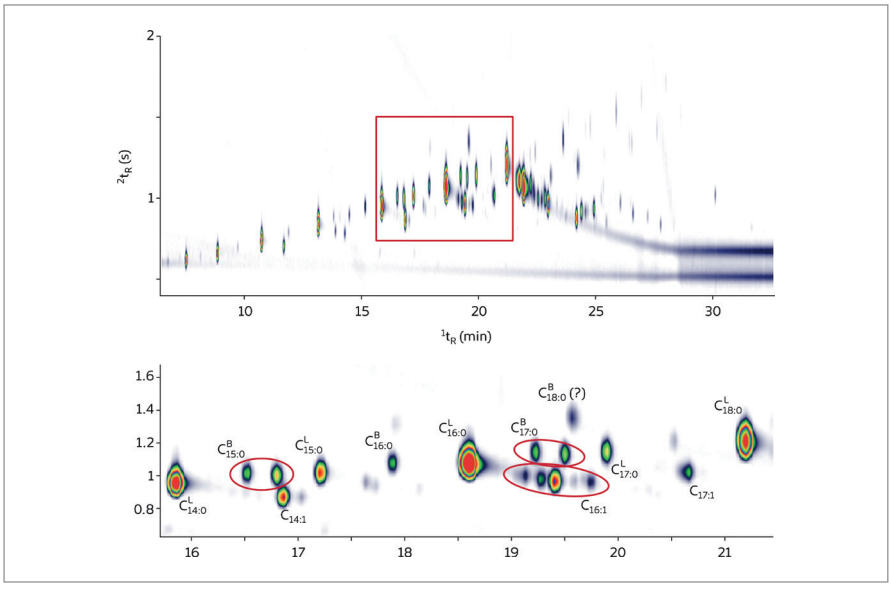

Figure 1. GC×GC–FID colour plot showing a FAME-derivatised butter extract, with the inset highlighting the improved separation achieved using GC×GC for linear (L) and branched (B) C_{14}–C_{18} FAMEs.

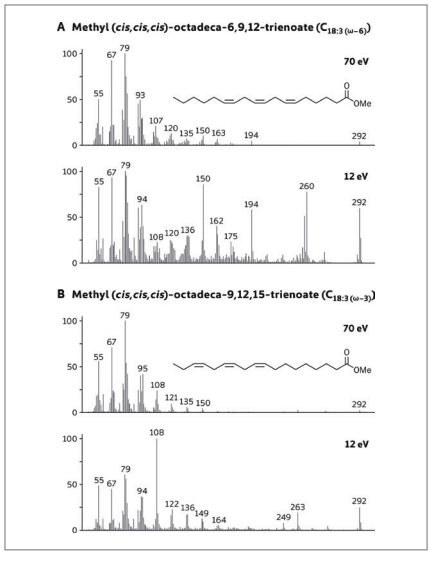

Figure 2. Comparison of Tandem Ionisation mass spectra at 70 eV and 12 eV for: (A) $C_{18:3\ (\omega-6)}$ and (B) $C_{18:3\ (\omega-3)}$.

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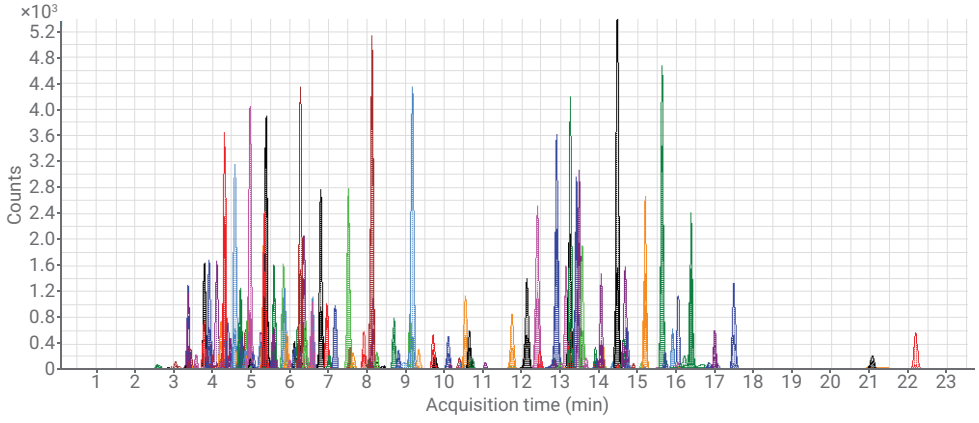
BROAD CLASS VETERINARY DRUGS IN PORK AND HEN EGGS

Sensitive residue analysis using an Agilent Ultivo Triple Quadrupole LC/MS System.

By Tony Zhang and Dan-Hui Dorothy Yang.

Veterinary drugs, consisting mainly of antimicrobials and hormones, have been widely used to treat livestock and poultry. These drugs are used to prevent diseases, enhance growth and improve feed efficiency. However, mounting concerns over misuse and content of these drugs from farm to table have led global health agencies to implement stringent regulations to address public health interests. For example, the US FDA, EU via SANTE and the Ministry of Agriculture in China have published final rules to regulate maximum veterinary drug limits in animal-derived foods. Due to the complexity of the matrices and characteristics of different classes of veterinary drugs, it is challenging to detect multiple classes of analytes in one run. Here we report a sensitive and robust single injection method that employs the Agilent Ultivo Triple Quadrupole LC/MS System and Agilent EMR-Lipid sample preparation kit for analysis of veterinary drugs in pork and hen eggs. Detection sensitivity was assessed by measuring a set of nine matrix-spiked calibration standards (0.1 ng/g to 40 ng/g) consecutively. The calibrations generated with linear fitting rendered R^2 >0.990 for more than 87 percent of the compounds in pork and R^2 >0.990 for 89 percent of compounds in egg. Ultivo showed impressive analytical sensitivities. A majority of the compounds in matrix could be detected at 2.0 ng/g (Figure 1). Additionally, 93 percent of the compounds detected in egg rendered a signal-to-noise (S/N) ratio greater than 10 for both quantifier and qualifier ions. The method precision was determined at 5.0 ng/g spike level in the matrix with seven replicate analyses. The percentage distribution in pork and hen at this level rendered excellent repeatability; 92 percent of the compounds had percent RSD within 20 percent.



Chromatogram of an egg sample fortified with 151 veterinary drugs at 2.0 ng/g

REAL-TIME ANALYSIS OF COMPLEX FLAVORS WITH PTR-TOF 6000 X2

IONICON PTR-TOFMS instruments are considered a gold standard for aroma release monitoring and real-time analysis of flavors without sample preparation.

By Lukas Märk

Food and flavor scientists want to understand what we smell and taste when drinking a cup of coffee or enjoying a fresh strawberry, at the very moment the flavor molecules hit our receptors. Another challenge for the industry is aroma matrices that should remain stable over a long period of time to account for a consistent consumer quality perception. Thus, a versatile analytical instrument needs to capture flavor molecules in real-time, without sample preparation, and provide a high resolving power for complex aroma systems. In order to separate compounds of interest from non-relevant substances which share the same nominal m/z but have a different chemical composition (isobars), very high mass resolution is of utmost importance.

The benchmark for highest mass resolution for substance separation is the new PTR-TOF 6000 X2. TOF-MS unlike quadrupole-based analyzers can separate isobars well and is therefore ideally suited for the measurement of complex aroma systems. To demonstrate the benefit of outstanding resolution we analyzed the nose-space air of a person who had consumed freshly brewed coffee. It is well known that vanillin and 4-ethylguaiacol are important isobaric aroma compounds at nominal m/z 153 (protonated molecules). However, by looking at the mass spectrum around m/z 153 in Figure 1, one can see that there are in fact four ions which share this nominal mass (black line). For a low resolution instrument (e.g. quadrupole-based), all four ions would be merged into one broad mass spectral peak and only their sum could be detected, neglecting a lot of useful data. The high mass resolution of the PTR-TOF 6000 X2 enables separation of all four ions in real-time. With IONICON's sophisticated data processing software the intensities are automatically deconvoluted (grey and blue lines; the orange line is the sum of the deconvoluted peaks, which perfectly reproduces the original data) and vanillin and 4-ethylguaiacol can be quantified independently, without an influence of the two additional isobars.

Major benefits of IONICON PTR-TOFMS instruments:

- Monitoring of real-time variations in aroma
- Head-space analysis without sample preparation
- Direct mouth- and nose-space air analysis
- Rapid screening of complex aroma systems
- Suitable for chemometric datamining

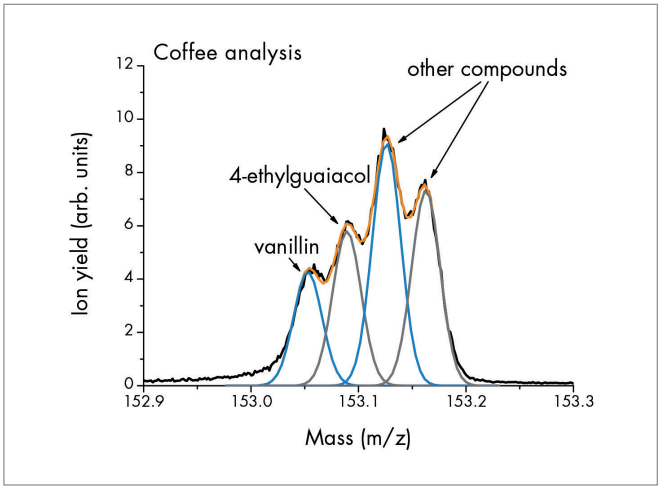


Figure 1. Nose-space air analysis of coffee flavor demonstrates high mass resolving power of PTR-TOF 6000 X2.

ANALYSIS OF AFLATOXINS BY UHPLC WITH MS/MS DETECTION

Introduction

Aflatoxins are some of the most carcinogenic mycotoxins known and include aflatoxins B1, B2, G1 and G2. They are produced by toxigenic strains of Aspergillus flavus, Aspergillus nominus, and Aspergillus parasiticus fungi after feed, crop or harvest exposure to moisture or warm temperatures. Aflatoxin B1 is considered to be the most genotoxic of the mycotoxins, and, when ingested by cows, is converted to aflatoxin M1. Though less potent than B1, M1 has been shown to cause liver cancer in certain animals (1). One of the strictest regulatory levels for any aflatoxin has been set forth by the European Union (EU), having established a stringent control limit of 0.05 ppb for M1 in milk (2). This limit is currently significantly lower than the Food and Drug Administration's (FDA's) limit of 0.5 ppb1. For aflatoxin analysis, HPLC with fluorescence (FL) detection is commonly used along with online post-column derivatization, to enhance B1 and G1 sensitivity. However, chromatographers prefer to avoid any derivatization requirements. With this in mind, the work presented herein demonstrates the effective chromatographic separation of aflatoxins B1, B2, G1, G2 and M1 by UHPLC-MS/MS, achieving the 0.05-ppb control limit for M1 without the need for derivatization.

Experimental

Hardware/Software

For the chromatographic separations, a PerkinElmer UHPLC system was used with a PerkinElmer QSight® 210 MS/MS detector. All instrument control, analysis and data processing was performed using the Simplicity 3Q™ software platform.

Method Parameters

The LC and MS/MS method parameters can be found in the full version of this application note.

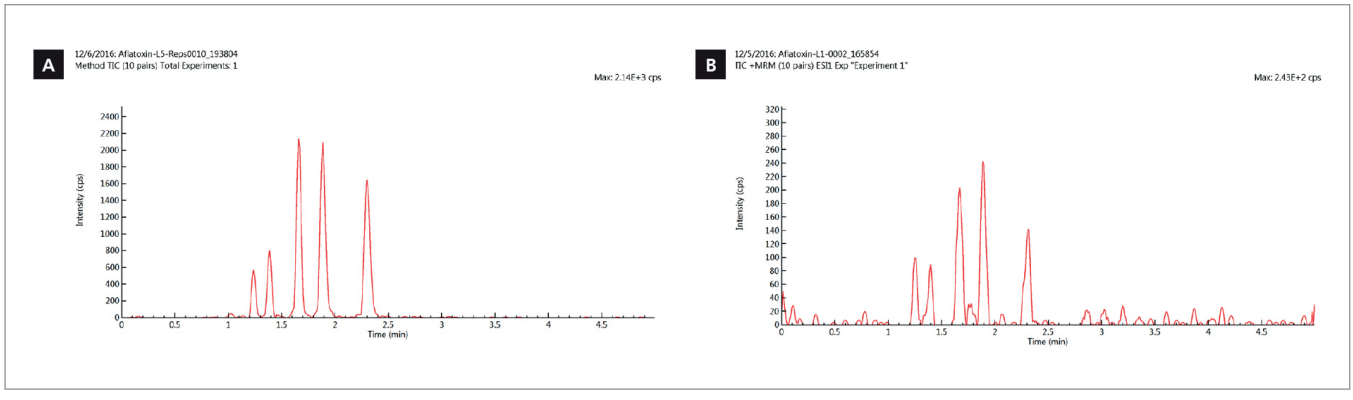


Figure 1. TICs of Level-5 (1 ppb) aflatoxin calibrant (A) and the Level-1 (0.05 ppb) calibrant (B).

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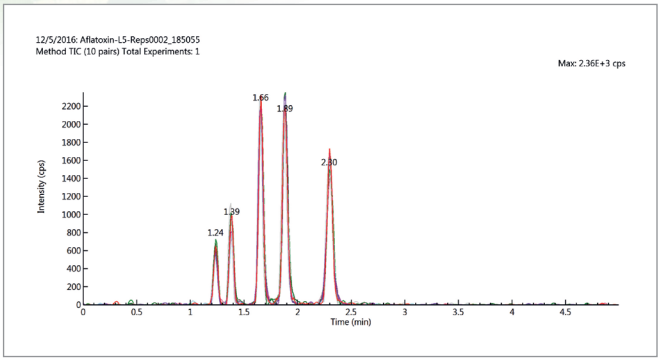

Figure 2. Overlay of eight replicates of the Level-5 (1 ppb) calibrant.

Solvents, Standards and Samples

All solvents and diluents used were HPLC grade. A 20-µg/mL (20-ppm) aflatoxin B1, B2, G1 and G2 standard solution in acetonitrile was obtained from Sigma-Aldrich, Inc® (Allentown, PA). A 10-µg/mL aflatoxin M1 standard solution in acetonitrile was also obtained from Sigma-Aldrich. A 10-ppb aflatoxin working standard was then prepared by transferring 50 µL of the B1/B2/G1/G2 stock solution and 100 µL of the M1 stock solution to a 100-mL volumetric flask and diluting to the mark with water. For calibration, aflatoxin concentrations of 1.00, 0.50, 0.25, 0.1 and 0.05 ppb were prepared via serial dilution with water.

Results and Discussion

Figure 1 shows the total-ion chromatograms (TICs) of the Level-5 (1 ppb) aflatoxin calibrant (A) and the Level-1 (0.05 ppb) aflatoxin calibrant (B), demonstrating the isocratic separation of all five analytes in under three minutes.

As shown in Figure 2, chromatographic repeatability was confirmed via eight replicate injections of the Level-5 calibrant, demonstrating exceptional reproducibility. The retention time percent RSD for all analytes was ≤ 0.06 percent.

Figure 3 shows example calibration plots for aflatoxins B1 and M1. Calibration linearity was greater than R2 = 0.995 for all analytes. Based upon a S/N of >10/1, the limit of quantitation (LOQ) for aflatoxin M1 was calculated to be 0.014 ppb. This level is far below the EU's regulated control limit of 0.05 ppb.

Conclusion

This work demonstrated the effective separation and quantitation of B1, B2, G1, G2 and M1 aflatoxins using a PerkinElmer UHPLC system with QSight MS/MS detector, obviating the need for derivatization. The results exhibited exceptional linearity for each aflatoxin over the tested concentration range. Quantitation was afforded down to below 0.05 ppb for all aflatoxins and the 0.014-ppb LOQ for aflatoxin M1 confirmed that the EU's 0.05-ppb control limit in milk is easily achievable.

References

1. *FDA Compliance Policy Guide, under Inspections, Compliance, Enforcement, and Criminal Investigations; CPG Section 527.400 Whole Milk, Lowfat Milk, Skim Milk - Aflatoxin M1*
2. *Commission Regulation (EU) No 165/2010. Amending Regulation (EC) No 1881/2006 Setting Maximum Levels for Certain Contaminants in Foodstuffs as Regards Aflatoxins. Official Journal of the European Union, Feb 26, 2010, pp L 50/8 – L 50/12.*

Download the full application note: tas.txp.to/0518/PERKINELMER

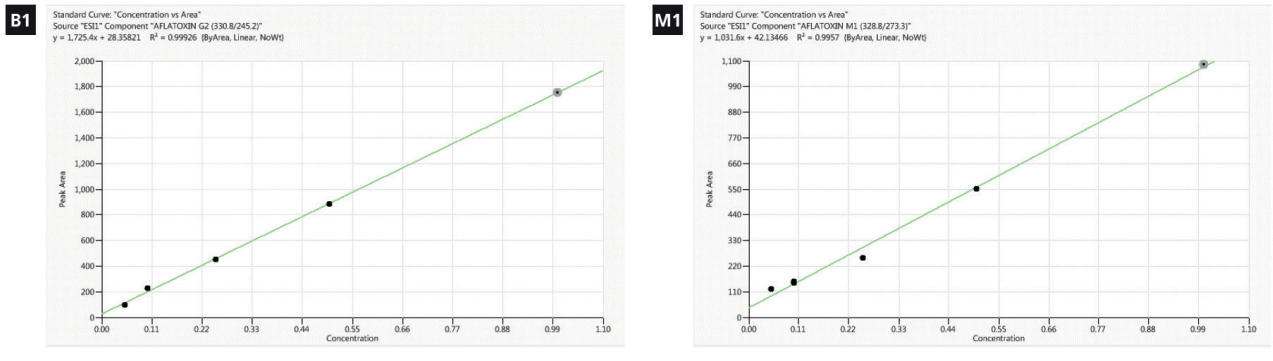

Figure 3. Calibration plots of aflatoxins B1 and M1; concentration range: 0.05-1.00 ppb; avg. of three replicates per level.

HOW TO USE CHEMICALLY INTELLIGENT MS SOFTWARE TO RAPIDLY IDENTIFY UNKNOWNS

Presenting a streamlined workflow for unknown identification using the MS Structure ID add-on within ACD/MS Workbook Suite.

By Brent G. Pautler, Richard Lee, Joe DiMartino

Identification of unknown components represents one of the most significant challenges when performing screening assays. While high resolution and high mass accuracy enable greater precision in predicting elemental composition, these estimates lack structural information. When faced with a true unknown, even fragment ion analysis is limited to highlighting particular functional groups at best. However, chemically intelligent MS software can utilize available analytical information, such as predicted elemental formulae, fragment ions, and known fragment inclusion/exclusion criteria, to produce an accumulative filter that can refine the results of a mass-based library search to a manageable number of structure candidates.

As a test case, data for an unknown pharmaceutical sample analyzed by HPLC-FTMS (Thermo Fisher Scientific), is loaded into ACD/Spectrus (v2017.1.3) for processing via ACD/MS Workbook Suite with the MS Structure ID add-on. Local versions of the PubChem and ChemSpider databases (as of February 2017) are used for the mass search.

Following extraction of all chromatographic components, a peak with t_R = 5.10 minutes and [M+H]+ = 272.2017 m/z is selected for identification by MS Structure ID. This component also has MS2 data associated with its [M+H]+ peak, which includes the 272.2017 m/z parent mass. This is used as the source for elemental composition determination, and to query the local PubChem and ChemSpider databases (~96 million compounds). Based on the accurate mass and resulting isotope pattern, the Formulae Generator suggests $C_{18}H_{25}NO$ as the best fit.

Searching the databases for the selected m/z value and elemental composition, with a tight tolerance of 2ppm, yields an initial set of 5752 structures (after removing duplicate stereoisomers). As this initial structure catalogue is quite large, additional filtering is performed via an include/exclude structure fragment list within Spectrus DB. Thus, the list is reduced to 22 candidates once duplicate structures are removed (Figure 1).

ACD/MS Workbook Suite can be further used to rank structures based on an "Assignment Score". This AutoAssignment tool uses ACD/MS Fragmenter to generate fragments from the parent structure, which are then scored (from 0 to 1) based on alignment with the MS2 spectrum. After running AutoAssignment, 8 structures possess a Match (dMS) value of 0.75 or higher, with only 2 having values above 0.80. Additionally, the top structure candidates can be examined more closely by extracting their MS2 spectra in a separate window and performing additional AutoAssignment/Fragmentation analysis; i.e. increasing the number of generated fragments to help deduce the best structural match.

Ultimately, this example demonstrates how to use the MS Structure ID Add-on within ACD/MS Workbook Suite to quickly search a wide range of potential structures, curate a reasonable and relevant list of candidates, and identify the most likely structure for a chromatographic peak, all in an extremely efficient workflow.

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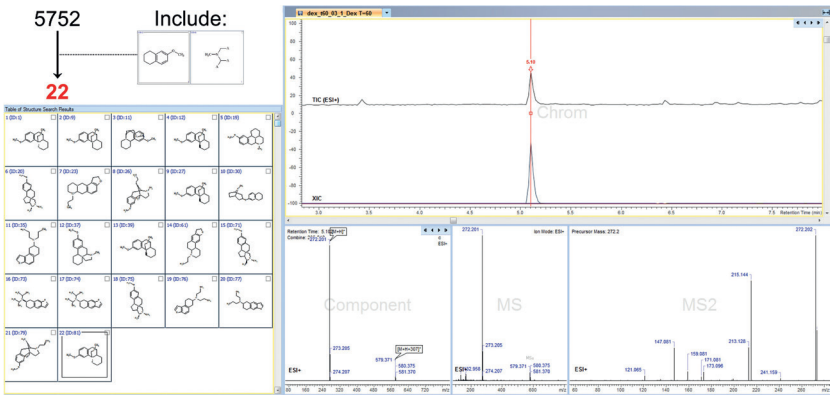

Figure 1. Reducing the candidate list from 5752 to 22 structures via a known fragment include list.

DETECTION OF RESIDUAL PESTICIDES ON FRUITS AND VEGETABLES USING PORTABILITY™ MINIATURE MASS SPECTROMETER

Mass spectrometry can be now deployed for onsite pesticide screening in real time.

By John MacAskill, Jean Yu and Lynn Chandler

Pesticides are used extensively in food production, ranging from pest mitigation in the field to inhibiting mold during storage and transportation. The growing reliance on pesticides and fungicides in turn requires monitoring and study to assess their environmental and toxicological impacts.

Mass spectrometry in conjunction with various separation techniques is the standard method for performing food and drug analysis. Techniques such as gas chromatography-mass spectrometry (GC-MS) or high performance liquid chromatography-mass spectrometry (HPLC-MS) provide unparalleled specificity and sensitivity, but require large, costly instruments with time-consuming sample preparation and controlled laboratory environments to function properly. Presented here is an application of thermal desorption electrospray ionization (TD-ESI) coupled to a portable mass spectrometer for pesticide screening.

TD-ESI is a mass spectrometry technique from the family of ambient ionization methods that utilizes very simple probing of different surfaces or liquids with no need for sample pretreatment. This feature, combined with the high selectivity of electrospray ionization, offers real-time, highly-sensitive measurements of pesticides with no pretreatment.

In TD-ESI a solid surface or a liquid sample is touched by a simple metallic probe. The probe and any adsorbed analyte is inserted into a custom sampling assembly. The probe is heated to thermally desorb analyte species that become entrained in a high temperature gas flow. This gas flows into the ionization region where desorbed analyte is ionized through charge transfer with ions generated from the electrospray.

Bayspec's Portability™ mass spectrometer is less than 10kg, highly-portable and battery operated. It features a linear ion trap with atmospheric pressure inlet (API). When TD-ESI is combined with Portability™, it is possible to perform fast, in situ detection of a wide variety of chemicals – including pesticides. The simple and fast sampling capability of the TD-ESI allows Portability™ to immediately analyze the surfaces of fresh produce without any sample pretreatment. Fast pesticide screening by Portability™ mass spectrometer can immediately disclose fake organic food products as well as discover elevated levels of residual pesticides.

TD-ESI-MS can screen for residual pesticides on the surfaces of different fruits and vegetables with short analysis times (1– 5 s). MS (as well as MS^n) analyses can be performed on surface-residual pesticides at ppm and sub-ppm levels. The miniaturized TD-ESI ion source is also capable of operating in positive and negative ion mode. Overall, BaySpec's miniaturized Portability™ combined with TD-ESI can find its wide applications for onsite detection of trace toxic chemicals.

Download the full application note: tas.txp.to/0517/BAYSPEC

Figure 1. Portability™ mass spectrometer (left) and TD-ESI sampling device (right).

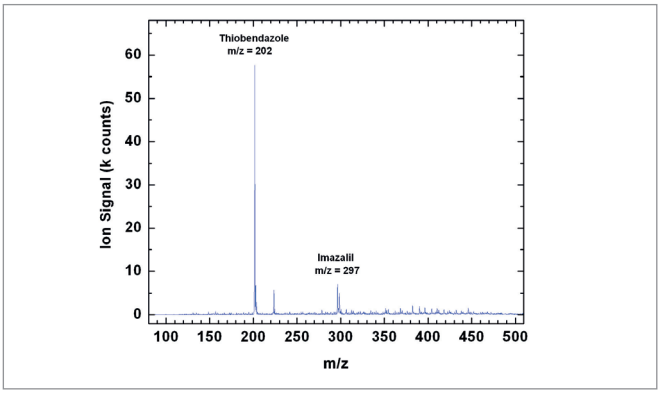

Figure 2. Full scan mass spectrum of an orange sampled using TD-ESI-MS. The fungicides thiobendazole and imazalil are clearly identified from the molecular ions at m/z = 202 and 297 respectively.