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The Column

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Astrobiology — A New Frontier

Analyzing extraterrestrial material using nano-techniques

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A New Frontier: NanoLC–NanoESI–MS in Astrobiology

Scientists from the Astrobiology Analytical Laboratory at NASA Goddard Space Flight Center in Maryland, USA, have developed a method coupling nanoliquid chromatography (nanoLC) and nanoelectrospray ionization (nanoESI) mass spectrometry to detect life's building blocks in "spacedust". Successfully applied to meteorite samples, it promises to open up new avenues of research in astrobiology. Mike Callahan from the team spoke to Bethany Degg of *The Column* about the inspiration behind the project, the challenges of analyzing extraterrestrial material in the laboratory, and future directions.

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Q: What was the inspiration behind the project?

A. It goes back quite a few years, back when I was still a graduate student at UC Santa Barbara. It was around this time I learned about the Stardust mission, which was NASA's Comet Sample Return Mission. I saw a photo of aerogel, the material used to collect cometary material. It was a photo of Peter Tsou holding this gigantic block of aerogel.[1] It had this beautiful blue, almost mesmerizing, glow. What's amazing too is that this silicon-based aerogel is almost entirely empty space (99.8% of the volume), which was necessary to safely collect hypervelocity particles and minimize thermal alteration.

It sounds silly, but I really wanted to handle a piece of aerogel. Obviously, the science for the Stardust mission was super cool too, the fact that you could study the chemistry of comets in a laboratory, but that's about 50% of the reason of how I ended up at NASA. I wanted to play…err…work with aerogel and investigate whether cometary material brought complex organic molecules such as nucleobases to Earth, which might have been significant for the origin of life.

Another inspiration was the investigation of interplanetary dust particles (IDPs). IDPs may have contributed a significant amount of prebiotic organic material to early Earth because of a potentially high and steady state flux. In 2008, Matt Pasek (University of South Florida, USA) and Dante Lauretta (University of Arizona, USA) published a nice paper[2] discussing extraterrestrial flux of carbonaceous meteorites versus IDPs and their significance towards prebiotic chemistry. While IDPs are known to contain organic compounds, it's unknown whether they contain biologically relevant compounds like amino acids or nucleobases. Danny Glavin and Jason Dworkin (NASA Goddard), both co-authors on this paper[3], shared this interest too.

Both cometary particles and IDPs are extremely precious. The Stardust spacecraft collected cometary particles, whereas IDPs are usually collected in the stratosphere by highflying aircraft. These extraterrestrial materials are extremely small and any organic compounds are expected to be in low abundances. Therefore, we first needed to develop the advanced analytical methods to analyze such samples. As proof-of-principle, we demonstrated our analysis on micrograms of meteorite sample.

The choice of meteorite was fairly easy to decide. We selected the Murchison meteorite because it is one of the best characterized meteorites as far as organic composition. We analyzed a 360 microgram sample of Murchison meteorite for amino acids using nanoliquid chromatography (nanoLC) coupled to nanoelectrospray ionization (nanoESI) high resolution orbitrap mass spectrometry techniques. The distribution and abundance of amino acids were similar to past studies of Murchison meteorite but the samples used in our study were about three orders of magnitude lower. Micrometeorites are around this mass range too, and it starts to become feasible to analyze even smaller IDPs and cometary particles. Our results were very promising in that respect. But just analyzing such small meteorite samples was quite an accomplishment, too.

Q: What were the objectives of the Stardust Mission? Is it an ongoing mission?

A. The primary goal of the Stardust mission was to collect samples from comet Wild 2 and return them to Earth for laboratory analysis. It was launched in 1999 and the Stardust sample capsule returned to Earth in 2006. The Stardust spacecraft continued on and eventually met up with comet Tempel 1 in 2011. After that, the mission was officially finished.

Q: Why is nanoLC coupled to nanoESI your method of choice? What are the advantages of your method over existing methods?

A. For small-scale samples, there are lots of techniques that operate at high spatial resolution, but they start thinning out when you want to focus on organic compounds and even more so when you want molecular-level identification. There are a few existing techniques that could be suitable, although the first ones that come to mind are techniques that are usually optimized for classes of organic compounds such as polycyclic aromatic hydrocarbons (PAHs) and they are not

really quantitative. Another issue is that the organic composition of extraterrestrial samples can be complex. This is the case with carbonaceous chondrites, the types of meteorites we typically analyze. You wouldn't think these samples are chemically complex just looking at them — they look like plain old rocks. But they are complex, it's a mess! For example, a mass spectrum of a meteorite extract can contain a ton of peaks representing lots of different organic compounds including different structural isomers. Therefore it would be desirable to have an analytical method that would be able to determine molecular structure with a high degree of confidence. Also, chromatography starts to become invaluable when dealing with complex samples.

To my knowledge, there really wasn't a good way to target amino acids in trace amounts in small-scale extraterrestrial samples until this study.[3] Even though the techniques we applied do not have high spatial resolution, we could overcome this limitation by simply extracting our tiny samples (which likely concentrated the analytes, too). We believe that the combination of nanoLC, nanoESI, and high-resolution mass spectrometry gives us the best chance of identifying and quantifying organic compounds. We take advantage of all the information we can get (for example, chromatographic retention time, accurate mass measurements, and product ion spectra using data dependent acquisitions or specific parent masses) for an unambiguous identification. Accurate mass measurements are extremely helpful to us (in terms of targeted and untargeted searches), and the fact that this is now routine for certain types of mass spectrometers is simply awesome.

Finally, our method still allowed for the discrimination of chiral amino acids, which was highly important in establishing origin (for example, a racemic mixture of amino acid enantiomers would be interpreted as extraterrestrial). Without chiral discrimination, it would be difficult to establish whether some amino acids were extraterrestrial because many of these amino acids were also proteinogenic.

Q: What were the challenges you faced when developing the nanoLC and nanoESI method? Can you offer tips to scientists new to the technique?

A. Anybody that works with nanoLC and nanoESI can tell you what a pain it can be sometimes. Millie Martin (NASA Goddard/ Catholic University of America), a co-author on this paper[3], worked tirelessly on different amino acid separation using a nanoflow liquid chromatograph coupled to a laser induced fluorescence (LIF) detector. We also needed these methods to have an efficient

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separation of chiral amino acids, which is used as an indicator of extraterrestrial origin. This took a lot of time. Eventually, we swapped out the LIF for a high-resolution linear ion trap-orbitrap mass spectrometer for better molecular characterization. And that took more time.

Terrestrial contamination of our extraterrestrial samples is always an issue and is always in the back of our minds. But we've established protocols to best minimize terrestrial contamination during our handling. This starts when we first receive extraterrestrial samples. Anything that comes into contact with extraterrestrial samples is first heat sterilized. We process meteorites inside a HEPA filtered flow bench. We process control samples in parallel to monitor any contamination. We actually wear safety glasses, nitrile gloves, and lab coats primarily to avoid contact that could introduce contamination. To be honest, sometimes I hold my breath doing all this. Is that to avoid contaminating things (however improbable) or that I'm still a little nervous (even after handling over a hundred meteorites now)? The answer is probably both.

Even handling such small meteorite samples is tricky. The meteorite powder is very fine, and weighing out such small amounts takes longer than you think.

In any research situation, patience and persistence usually pays off. I would say the exact same thing when it comes to these techniques. The learning curve is a little higher, but it's not impossible. In fact, it's getting easier nowadays. These techniques are becoming more commonplace in analytical labs.

Q: Does the method have applications outside of astrobiology?

A. These types of methods are used in different areas of life sciences. But our specific method is definitely geared towards the study of extraterrestrial materials. For example, many of the amino acid targets are non-proteinogenic or considered rare on Earth.

Q: What's next?

A. Optimize our techniques even further. Expand the analysis to include other biologically relevant molecules such as nitrogen heterocycles. And start to investigate micrometeorites, interplanetary dust particles, and cometary material captured from the Stardust mission! I have a test tube of aerogel near my desk as a reminder.

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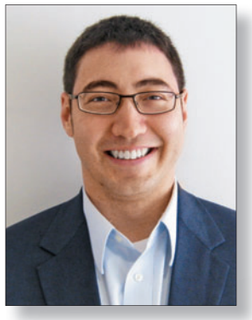

Mike Callahan is a research physical scientist in the Astrobiology Analytical Laboratory at NASA Goddard Space Flight Center in Maryland, USA. His research focuses on the study of organic matter in meteorites with an emphasis on nitrogen heterocyclic compounds. His research on nucleobases and nucleobase analogues in carbonaceous meteorites has received national and international coverage (including *CNN*, *TIME*, *The Washington Post*, and *Popular Science*). He was also featured on the Discovery Science Channel show "Through the Wormhole with Morgan Freeman" (Season 3, Episode 1). He was awarded the Robert H. Goddard Exceptional Achievement Award in Science (2011) and the NASA Early Career Achievement Medal (2012) for his outstanding contributions to the analysis of extraterrestrial nucleobases in carbonaceous chondrites.

***Disclaimer: The above views are solely those of Mike Callahan and not representative of NASA.**

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News

Fluoride Analysis of Ice Cores Using Fast Ion Chromatography

Scientists from the University of Florence in Italy have developed a novel fast ion chromatographic (FIC) method for the analysis of fluoride in Antarctic snow and ice. Data from fluoride analysis has the potential to assist scientists to build up a picture of past volcanic activity.

Polar ice sheets are one of the "cleanest" sampling locations on Earth and are relatively unaffected by human activity. As ice layers form, minerals from the atmosphere and environment are trapped and preserved. Each layer is associated with a specific time frame, rather like counting tree rings. Environmental scientists can take advantage of this by drilling into the ice, and extracting ice cores for analysis.

Various analytical techniques are available to prepare ice core samples for analysis. Typically, a system is setup whereby the ice core sample is slowly and continuously melted releasing each layer to be analyzed by continuous flow analysis (CFA), inductively coupled plasma–mass spectrometry (ICP–MS), and FIC.

Analysis of ice cores can allow reconstruction of past changes in climate systems, including a record of volcanic activity over thousands of years. Dust, ash, and sulphur deposited in the atmosphere can be trapped ready for identification. However, there are issues with sulphur analysis of ice cores because there are a number of different sources (sea salt, continental dust), and it requires decontamination before analysis.

Hydrofluoric acid (HF) is a potential marker that can be used instead. According to the paper, published in *Environmental Science and Technology*, at around the time of the eruption and degassing of Mount Erebus on the Ross Ice Shelf-McMurdo Sound there are records of relatively high concentrations of HF. Furthermore, ice cores from 17,500 ± 500 years old show a "main event" when a layer of fluoride was deposited around 170 years ago. This is thought to be attributable to a series of eruption from a subglacial volcano in West Antarctica.

Mirko Severi, lead author of the paper, told *The Column*: "Following the idea of the FIC method setup for the analysis of chloride, nitrate, and sulphate in the two EPICA [European Project for Ice Coring in Antarctica] ice cores, we decided to set up a similar method for the determination of fluoride."

More than 450 samples were analyzed. Heart-cut column switching was utilized to preconcentrate analytes from the continuous melting of ice, separating analytes from highly retained compounds, before passing onto an analytical column for identification purposes. Severi said: "This novel method could be added to the several chemical flow analysis and FIC methods that are already run in the field of ice-core analysis. The challenge was to find a chromatographic method able to be 'fast' in eluting the fluoride peak (the total analysis time is less than 3 min) when analyzing samples containing anions with higher retention times such as sulphate." — B.D.

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Daniel W. Armstrong Receives ACS Award in Separation Science and Technology 2014

Daniel W. Armstrong, a professor of chemistry and biochemistry at the University of Texas Arlington in Texas (USA), is the recipient of the 2014 American Chemical Society Award in Separations Science and Technology, sponsored by Waters Corp.

Armstrong is considered a leader in his field and the "father" of pseudo-phase separations — a type of chromatography that lowers costs, volatility, and toxicity while providing higher selectivity than other analytical methods. His commercialized innovations are applied across the drug development, environmental analysis, and petrochemical industries.

Armstrong said: "One of the strengths of our group is we come up with new things to explore constantly, which is fun. You want to do things that have an impact and are useful, either adding knowledge, insight, or something practical that people can actually use." In his career he has developed more than 30 different chromatography columns — a gas chromatography column that Armstrong developed is now part of the Rosetta mission of the European Space Agency exploring the composition of comets in space.

Furthermore, Armstrong has published more than 550 scientific publications, including 29 book chapters and holds 23 U.S. and international patents. He is also a member of the *LCGC Europe* and *LCGC North America* Editorial Advisory Boards.

Novel Passive Sampling Using Silicone Wristbands

Silicone wristbands promoting charitable organizations and which are popular throughout the world as a fashion statement can be used to monitor exposure of individuals to contaminants in their local environment, according to a study published in the journal *Environmental Science and Technology*.[1] The authors of the study propose silicone bands as passive samplers that can give a temporal and spatial picture of human contaminant exposure.

People are exposed to uncountable contaminants in their everyday life, both natural and synthetic. Making connections between contaminant exposure and the onset of disease is therefore challenging, as there can be multiple contaminants identified within one workplace environment, not to mention in the home. Scientists can use passive sampling that involves recruits carrying a sampler, such as a backpack, during their day-to-day life.

Silicone has already been demonstrated as an effective passive sampling device (PSD) because it is known to absorb a wide range of compounds. *The Column* has previously reported the use of explanted silicon prostheses for the biomonitoring of persistent organic pollutants (POPs) in humans.[2]

In this study the wristbands were thoroughly cleaned and sterilized, with two bands from each batch assessed for contamination. Participants were then given either one or two bands to wear continuously for a period of 30 days before returning the bands for analysis. The bands were extracted, and the resulting samples screened for 1182 chemicals using gas chromatography–mass spectrometry (GC–MS). The study was able to identify 49 different compounds, including polycyclic aromatic hydrocarbons (PAHs) or industrial compounds (pesticides, flame retardants, and plasticizers). Diethyl phthalate and tonalide, which are both found in personal care products, were the most commonly found.

As a proof of concept, bands were also given to eight roofers using hot asphalt to quantitate PAH exposure, a known risk in this job type, for a period of either eight or 40 h. In addition to 12 known PAHs, two unknown PAHs were also identified, benzofluornenone and fluorenone.

Co-author Kim A. Anderson said: "We can screen for over 1000 chemicals that may accumulate in the wristbands. Currently, PAHs, pesticides, flame retardants, PCBs, industrial chemicals, and consumer and pharmaceutical products have been quantified in wristbands." The novel application of silicone wristbands is an interesting one as they are non-invasive, cheap, and effective at monitoring contaminant exposure. — B.D.

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A Look to the Future

Incognito discusses the impending technological singularity of analytical chemistry. Is it time to take back control from the machine?

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The "technological singularity" is said to describe the point in time at which artificial intelligence (computers) will have an intelligence level exceeding that of the human race. The idea has been around a long time. In 1863 Samuel Butler proposed that machines are a kind of "mechanical life" that are on course to continually evolve into the dominant species in an article titled: "Darwin among the Machines". Mere whimsy? Bear with me dear reader.

As I reached into the high performance liquid chromatography (HPLC) column cupboard last week, I noticed something called a "carbohydrate analysis" column. It had no phase definition on the box, only the usual dimensions, serial number, batch number, and the words "carbohydrate analysis column". I paused to look at the other columns in the cupboard and managed to count nine (around 12% of all columns within the cupboard) that didn't have a specific phase designation, but rather an application name.

I suspect that someone somewhere hasn't designed and manufactured a completely new chemistry for the analysis of carbohydrates, amino acids, protein A, PAHs, and so on. The majority of these columns will be packed with materials that are at least based on "traditional" chemistries. I'm betting the carbohydrate column in question is based on an aminopropyl. Is this a new trend away from using knowledge of stationary phase and analyte interactions to select an appropriate phase, to being served a "cookbook" from our vendor giving dedicated column/application/reagents/system — what else? Is that a bad thing? Is it necessarily dumbing down or has the emphasis on R&D just shifted earlier in the supply chain?

Furthermore, the gas chromatography (GC) column cupboard has even more of these "application specific" columns.

As I consider a more application driven, more constrained future, the laboratory begins to wobble and the *Twilight Zone* music plays in my ears (it's OK reader, I'm fine, this is what we call artistic licence). Feel free to substitute the theme tune for *The X-Files* or even *CSI* if you must, depending upon age. Imagine a bespectacled gentleman enters a small room with a vial of white powder and places it into what resembles a small airlock door on the side of a white box, leaving immediately afterwards. Later, while at dinner, the man receives a message on his personal digital device that contains a full list of results relating to his white powder. It tells him that a chromatographic method was successfully developed with minimum resolution of 2.12,

that 22 components have been positively identified and successfully quantified against the method, which had validated successfully against the current International Conference on Harmonization (ICH) criteria, and that no compounds of toxicological significance have been identified above the current reporting limits. It also tells him that the results have been fed into the Enterprise Corporate Data Management system and that the formulation has been accepted for stage II development, which was currently taking place. Is this just a fantasy?

There are robotic systems that can prepare a powdered sample for analysis with whatever dilution, mixing, or addition of reagent is necessary. There are also automated HPLC method development systems with software that can take a sample and develop a viable method based on criteria set by the end user — some systems operate on Quality by Design (QbD) principles, which use experimental design to develop a method specification with appropriate operating ranges for key variables.

There are modern orthogonal column technologies that vastly reduce the number of phases required. They may be "multi-mode" type columns, or 2D HPLC approaches where selectivity "tuning" rather than phase selection is possible.

I wrote relatively recently in this column about the possibility of a truly quantitative "universal detector" that could be linked using some relatively straightforward optimization algorithms to a mass spectrometer with a multi-mode source to take care of the detection side of things.[1] I also recently heard of a chromatography data system that was capable of completing and reporting a fully automated ICH method validation. No it's not a fantasy, but perhaps just a big development project.

Everyone has heard of this inexorable development of our industry called industrialization, automation, black box, and so on, but read the philosophies of Radovan Richta. This evolution follows a well-defined progression of tools, machines, and automation.

The technological singularity for analytical chemistry will not be until the instrument can work out what the limits for the experimental variables should be, what the ICH guidelines should be, and the toxicological reporting limits are set in the example I quote above. I'm not saying it isn't possible, as neural networks are a very powerful way of collecting and analyzing large data sets to define the requirements to achieve pre-prescribed outcomes.

Do you remember the Tricorder from *Star Trek*, a handheld device that could be used to scan rock samples, blood, or body tissue to give a full analysis in seconds? Psychologists call the process of getting from where we are now to the "Tricorder" analyzer "technological convergence". This is the tendency for technologies to combine so that single devices have an ever increasing number of functions. Just look at your smart phone for the perfect example of this. What's to stop all our analyses being done with a single device? This would undoubtedly require new "modes" of analysis combined with new sensor and processing technologies, which is obviously a huge leap even from our conceptual advancement above. But are there academic and industrial projects working on aspects of how this might be possible? Why aren't we even discussing the concept of future technology openly? As I build my Pittcon technical session planner, I'm not really seeing any sessions on "Towards the Analytical Technological Singularity". Why? Are we all too busy to have a meaningful input? Or is the PAH column I discovered in the cupboard actually the start of something much, much bigger?

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LC–MS-Based Methods for Identifying Drugs of Abuse in Oral Fluids for Forensic Toxicology Applications

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Forensic toxicology screening methods are widely used to test biological samples for the presence of pharmaceutical drugs as well as drugs of abuse and their metabolites. Screening has most commonly been performed on urine or blood samples, using immunoassays or gas chromatography coupled with mass spectrometry (GC–MS). However, these methods have various limitations that include risks of false positives and false negatives, lack of specificity, and limited throughput. Moreover, these methods are unable to keep up with continual developments relating to drug abuse. For example, they are not suitable for detecting drugs from numerous different classes simultaneously, and are not always easy to adapt for detecting new drugs. In recent years, forensic toxicology laboratories have begun developing liquid chromatography–mass spectrometry-based (LC–MS) methods for drug screening. This article reviews key developments and potential advantages of using LC–MS for forensic toxicology, including the option of using oral fluid samples.

Testing of biological samples for the presence of drugs or their metabolites is widely used to determine whether an individual has consumed certain drugs, and in what quantity. It is becoming increasingly important in a number of settings, including research into monitoring abuse of prescription drugs, such as opiates and benzodiazepines; testing among offenders for consumption of recreational drugs such as amphetamines, cannabinoids, and cocaine; and testing for use of banned steroids and other substances by professional athletes and sports players.

Such testing is commonly performed on urine or blood samples, with oral fluids, sweat, and hair also used. The choice of sample fluid may depend on the class of drugs being tested, availability of testing methods for the drug(s) or metabolites of interest, the detection window, and practicalities associated with sample collection.

Urine is most commonly used for drug testing, largely because of the long detection window: Drug metabolites can be detected days or, in some cases, weeks after the original ingestion. Drug metabolites are usually available in relatively high concentration in urine, it is easy to analyze and can have a long shelf life (up to two years). It is the fluid of choice for testing and consequently there are many methods and standards available for detecting diverse drugs in urine samples. Its disadvantages include the inconvenience of sample collection, particularly for female donors, and its

Photo Credit: TEK IMAGE/Getty Images

potential for sample adulteration or switching if collection is not observed.

Traditional Approaches to Drug Testing

Three main approaches have traditionally been used for drug testing: Immunoassays, liquid chromatography with ultraviolet detection (LC–UV), and gas chromatography coupled with mass spectrometry (GC–MS). Screening may be either targeted — where the analyst looks for evidence of a known list of substances, or untargeted (general unknown screening), where the analysis is open to a much broader group of substances, including unexpected substances and even unknown designer drugs. The majority of routine drug testing takes a targeted approach, but there is a growing need for unknown screening as drug-taking behaviour and designer drugs continue to evolve.

Immunoassays have been commonly used for drug testing, largely because of their ease of use and low cost. They are generally sensitive but are prone to cross-reactivity, which can cause considerably high rates of false positive or false negative test results.[1,2] Accordingly, many testing laboratories use a two-step procedure where positive immunoassay results are confirmed using GC–MS. However, as drug abuse continues to increase, the limitations of this approach are becoming more apparent. Firstly, immunoassays are only available for a limited number of drug classes and they are not easily adapted to detect new drugs.[3] The rise of so-called "designer drugs" and increasing prevalence of uncontrolled substances is putting further pressure on drug testing laboratories to be able to detect new or unknown substances. Secondly, neither immunoassays or GC–MS are suitable for detecting drugs from across several different classes in a single run. Thirdly, GC–MS is only suitable for volatile or organic compounds, meaning that GC–MS-based workflows typically require extensive sample preparation steps, including solid-phase extraction (SPE) and/or derivatization prior to injection. These factors all add significant time, labour (and potential for error), and costs to immunoassay and GC–MS analyses, making them less suitable for the high throughput and broad detection capabilities required for today's testing laboratories. LC–UV methods have been used for drug testing but these suffer from lack of specificity and also require confirmation of positive findings against a standard. The total workflow is lengthy and, again, makes this approach invalid for modern testing.

LC–MS–MS for Drug Testing

Over the past two decades, researchers have started to develop LC with tandem mass spectrometry (LC–MS–MS)-based methods for forensic toxicology screening, aided by technological advances such as the development of electrospray ionization (ESI) that have helped LC–MS to become a valid, routine technique. LC–MS–MS has a number of advantages over GC–MS for drug testing, in particular its suitability for analyzing a broad spectrum of compounds, including those which are difficult to analyze by GC–MS (for example, amines and semi-volatile compounds), without the need for lengthy sample preparation steps.

By introducing tandem mass spectrometry, the selectivity of the method is greatly enhanced, which also improves limits of detection and quantitation. The enhanced selectivity of LC–MS–MS compared with traditional approaches has been demonstrated in a number of comparison studies for detecting different drug classes in urine samples. Recent examples include detection of benzodiazepines and their metabolites,[2] "designer" benzodiazepines,[3] false positives in amphetamine detection caused by the presence of dimethylamylamine,[1] cannabinoids,[4] and opiates.[5]

Robandt et al. (2010) developed an automated SPE–LC–MS–MS method for analyzing 6-acetylmorphine (6-AM) in urine samples and compared the analytical performance with a previously used GC–MS method.[6] They found that the SPE–LC–MS–MS method reduced labour and costs significantly by eliminating the need for specimen handling, sample extraction, and derivitization. The total time required for extraction and analysis was reduced by approximately 50% compared to the previously validated procedure using manual SPE and GC–MS analysis. The new method also achieved good selectivity, with limits of quantification and detection at 2 ng/mL; the presence of other opioids (codeine, morphine, oxycodone, oxymorphone, hydromorphone, hydrocodone, and norcodeine) did not interfere with detection of 6-AM.

Another advantage of LC–MS–MS for drug testing is its ability to simultaneously detect and rapidly quantify numerous drugs and their metabolites from one injection in a single run, including synthetic and designer drugs, and drugs of abuse across multiple different classes.[7,8] Scheidweiler & Huestis recently developed a novel LC–MS–MS method for simultaneously quantifying 20 synthetic cannabinoids and 21 metabolites, and semi-quantifying 12 alkyl hydroxyl metabolites in urine.[9] The method used fast polarity switching to acquire data in positive and negative ionization modes, therefore allowing for detection of a broader range of compounds. The method also relied on an advanced LC–MS–MS system for quantification by multiple reaction monitoring (MRM), which allows faster and more accurate data acquisition from numerous compounds. Several LC–MS–MS

methods have now been developed for monitoring hundreds of compounds across multiple drug classes in a single run, in just minutes. Such methods often use MRM in combination with a linear ion trap with fast polarity switching and acquisition of MS–MS spectra, for compound identification through mass spectral library searching.[7]

New Possibilities for Oral Fluid Samples

In recent years, there has been growing interest in using oral fluid samples instead of urine for forensic toxicology. Advances in LC–MS–MS-based technologies and methods mean that compounds can now be detected with greater sensitivity and higher accuracy, making oral fluids a more viable option for detection of certain drugs.[10] There are many advantages to collecting oral fluid samples compared with urine,[11] including convenience and immediacy of sample collection, minimal chance of specimen adulteration, the lack of requirement for gender-specific staff for collection and observation, and reduced sample collection overheads. Oral fluid samples can also be easier to ship and store than urine, but the amount of specimen provided is usually much lower in volume. The quantities of compounds detected in oral samples typically provide a closer correlation to the drug dose ingested, and researchers have reported much higher prevalence of the parent drug in oral samples compared with urine.

Methods have recently been successfully developed for fast multiplex screening in oral fluid, for example screening 32 drugs including amphetamines, barbiturates, opiates, benzodiazepines, methadone, and cocaine in opioid-dependent patients;[11] and for detecting opiates, amphetamines, MDMA, PCP, and barbiturates simultaneously.[12] Vindenes et al. (2011) compared oral fluid and urine sample analyses taken from 45 patients being monitored for drug abuse and found similar results in both sample types for most drugs.[11] The main differences were that amphetamines and heroin were more commonly detected in oral fluids, and cannabis and benzodiazepines were more commonly detected in urine samples. These findings are in line with previous studies and would be expected due to the basic versus acidic natures of these drug classes, respectively.

Conclusions and Future Directions

Advances in LC–MS–MS technologies over the past 20 years have led to many new possibilities for forensic testing in diverse settings. Modern-day instrumentation offers significant improvements in the speed, quality, and accuracy of data acquisition, and allows scientists to detect and quantify numerous compounds across multiple drug

classes simultaneously, from a single injection, even in oral fluids. The higher throughput, reduced labour, and greater confidence in results all help to achieve more efficient testing. Furthermore, the versatility of newer LC–MS–MS methods will enable scientists to detect more unknown compounds as new synthetic illicit drugs enter the market, and help authorities to keep up with ever-changing drug-taking habits.

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Growing Fruit — and Economies

Joe Anacleto, Helen (Qingyu) Sun, Zicheng Yang, and Kefei Wang, Bruker Daltonics, Fremont, California, USA.

Demand for exotic goods, such as wine and oranges, continues to grow in the western world. However, agricultural regulations can vary significantly from region to region. Regulatory bodies, such as the United States Food and Drug Association (US FDA), routinely screen all imported goods to ensure they meet the domestic limits of tolerance. Detection of low concentrations of pesticides within complex matrices, such as orange juice and wine, is a complex process that requires a high level of sensitivity. This article details how a newly developed "dilute-and-shoot" sample preparation method for LC–MS–MS multi-residue monitoring (MRM) pesticide screening can be performed to overcome matrix effects.

Brazil is world-renowned for its high-quality oranges and China has a growing reputation for its high-quality…wine? In 2012, demand for wine exceeded supply by 300 million cases,[1] and China's wine industry has been expanding to meet this demand.

Wine grapes have been cultivated in China since the second century B.C., but Chinese wine has always had an unfavourable reputation. This has recently begun to change as demand has grown from within China's rapidly expanding middle class for access to alcoholic drinks popular in western countries. Domestic wine production has grown markedly to keep pace with the demand, and wineries in the Ninjxia region can now rival wines produced in the west in terms of quality.[2]

China now exports wine to its Asia Pacific neighbours, as well as further afield. Chinese wines can be found in stores across the globe and they are recognized for their affordability. However, the future expansion of Chinese wine exportation is threatened by irregularities in agricultural regulations. Several germicide species integral to growing strategies are tightly controlled within "consuming" countries, and in many cases are prohibited. Imports from China have been seized or turned away from ports because of excessive residual pesticides. To reduce the number of incidents, both producers

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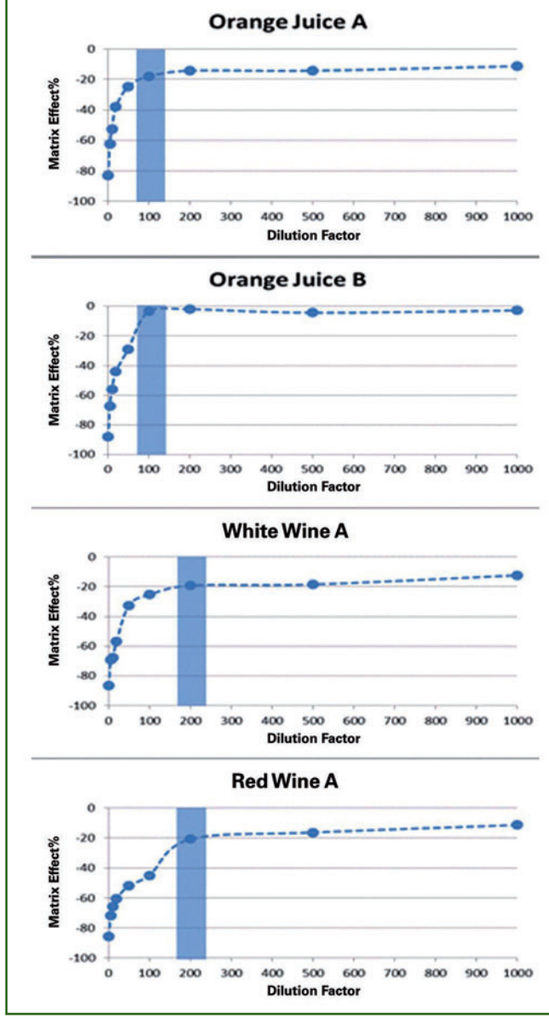
Figure 1: Matrix effect% against dilution factor reveals appropriate dilution factors of 100-fold for orange juice and 200-fold for wine.



and regulatory bodies need to effectively screen for multiple pesticide residues for complete assurance of safety.

This is not an issue exclusive only to China. Brazil grows more oranges than any other country in the world, producing 358,573 tonnes in 2012 (U.S. $907,517,764 worth of fruit).[3] The orange juice market in Brazil is completely reliant on international export. In 2012, trace amounts of banned fungicide carbendazim were detected in a shipment of orange juice from Brazil triggering a brief but large-scale media scare. The concentration detected did not pose a significant risk to the consumer, but the incident brought pesticides to the forefront of the public consciousness and led to the US Food and Drug Administration (FDA) reaffirming its commitment to testing all imports.[4]

Carbendazim is a broad-spectrum fungicide applied in the cultivation of arable crops. Excessive consumption and exposure to carbendazim can be harmful and has been linked to several hormonal and endocrine-related diseases.[5] The use of carbendazim has been gradually phased out over the course of the past two decades, and is now banned in the USA. The FDA now enforce a limit of no more than a 10 ppb concentration of carbendazim in fruit imports.

Despite the ban in the USA and many regions of Europe, the fungicide is still widely used throughout Asia and South America. Crops grown in bulk for export are often sprayed with the fungicide, and so regulation of imports from regions where the fungicide is readily used is an ongoing challenge.

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Figure 2: Overlaid ion chromatograms of 200-fold diluted red wine B and 200-fold diluted red wine A. Red wine B records carbendazim concentration at 3740 ppb, 300× above the FDA limit of tolerance.

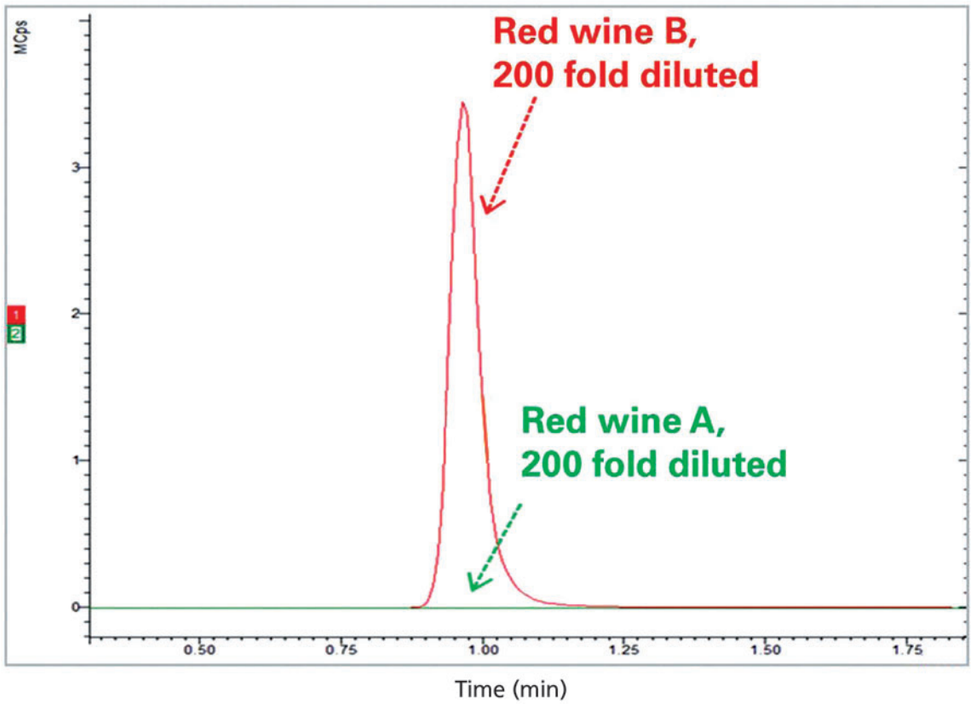

Table 1: LC–MS–MS analysis of carbendazim in diluted blank matrices.

Blank Matrix	Dilution factor	Measured Conc. (ppb)	Calculated Conc. (ppb)
Orange juice A	100	0.012	1.2
Orange juice B	100	N/A	< 0.5
White wine A	200	N/A	< 0.5
Red wine A	200	0.007	1.4
Red wine B	200	18.7	**3740**

The standard technique used in pesticide screening is liquid chromatography coupled with triple quadrupole mass spectrometry (LC–MS–MS), but as these species are present within complex high concentration matrices, it can be difficult to attain clear sensitivity and selectivity within the MS analysis. This can be overcome by performing sample preparation techniques, such as "dilute-and-shoot".

Methods

Samples are diluted prior to analysis when using a "dilute-and-shoot" method to minimize matrix effects. This results in a concentration decrease of any potential pesticide species, and there are very few techniques that have sufficient sensitivity and robustness for this analysis. Furthermore, few studies have been carried out to determine the appropriate dilution factors or to evaluate dilution-factor-caused matrix interferences for carbendazim analysis.

The following case study demonstrates the effectiveness of the new dilute-and-shoot sample preparation technique for carbendazim analysis using a LC–MS–MS system with a calibration range from 0.005 ppb up to 50 ppb. Orange juice samples A and B were purchased from a grocery shop in California. Three wine samples were also purchased: Red wine A; white wine A, and red wine B.

Experimental Setup: *Chromatography Conditions:* LC–MS–MS system (Bruker EVOQ

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Elite). Column: 50 mm × 2 mm, 3 µm YMC C18-Hydro. Injection volume: 5 µL. Flow rate: 400 µL/min. Solvent A: Water + 0.1% Formic acid + 10 mM Ammonium Formate. Solvent B: MeOH, + 0.1% Formic acid + 10 mM Ammonium Formate. Gradient conditions: 0.0min 30% B; 0.2 min 30% B; 2.0 min 60% B; 2.1 min 95% B; 3.1 min 95% B; 3.2 min 30% B; and 5.2 min 30% B.

Mass Spectrometry Conditions: EVOQ Elite system (Bruker). VIP-Heated ESI — positive mode. Spray voltage: 4000 V. Nebulizer gas flow: 50 units. Heated probe gas flow: 45 units. Heated probe temperature: 450 °C. Cone gas flow: 15 units. Cone temperature: 300 °C.

Samples: The orange juice and wine samples were diluted with water using 5×, 20×, 100×, 200×, 500×, and 1000× dilution factors. Each diluted sample was spiked with 1 ppb of carbendazim. Nine carbendazim calibration standards were also worked up at 0.005, 0.01, 0.05, 0.1, 5, 10, and 50 ppb.

Results

Figure 2 shows the percentage of matrix effects against dilution factor for each of the diluted food samples. For the orange juice samples, 100-fold dilution matrix effects were reduced to below 20%, as shown in Figure 1. However, for the wine samples at least 200-fold dilution was required to reduce matrix effects to below 20%.

The limit of quantitation set by the LC–MS–MS for carbendazim analysis was 0.005 ppb. The relative standard deviation (RSD) % of 10 replicate injections of 0.01 ppb carbendazim was found to be 3.4%, indicating excellent instrument sensitivity and robustness.

Table 1 shows that the diluted blank matrices, orange juice samples A and B, white wine A, and red wine B all exhibited trace concentrations of carbendazim. These were well below the 10 ppb limit of tolerance permitted by the FDA. However, the carbendazim level for the 200-fold diluted red wine B revealed levels of carbendazim concentration at 3740 ppb, more than 300× the acceptable level (Figure 2). A 100-fold dilution of orange juice and a 200-fold diluted wine sample were found to be suitable dilution factors for the "dilute-and-shoot" method.

Conclusion

The continued increase in international agricultural trade from economies such as China and Brazil has necessitated the need for robust and sensitive pesticide screening techniques in consuming countries. The new "dilute-and-shoot" multi-residue-monitoring method described here shows how modern LC–MS–MS systems can provide the required sensitivity, even with challenging sample matrices and low concentration samples.

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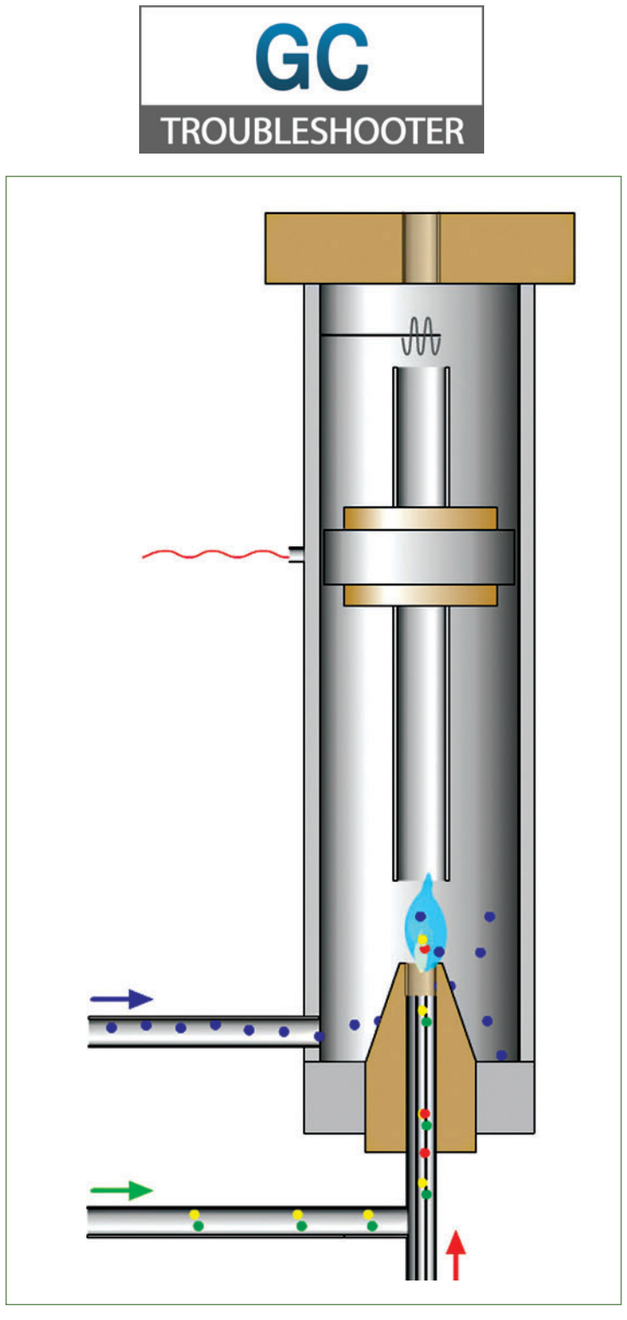

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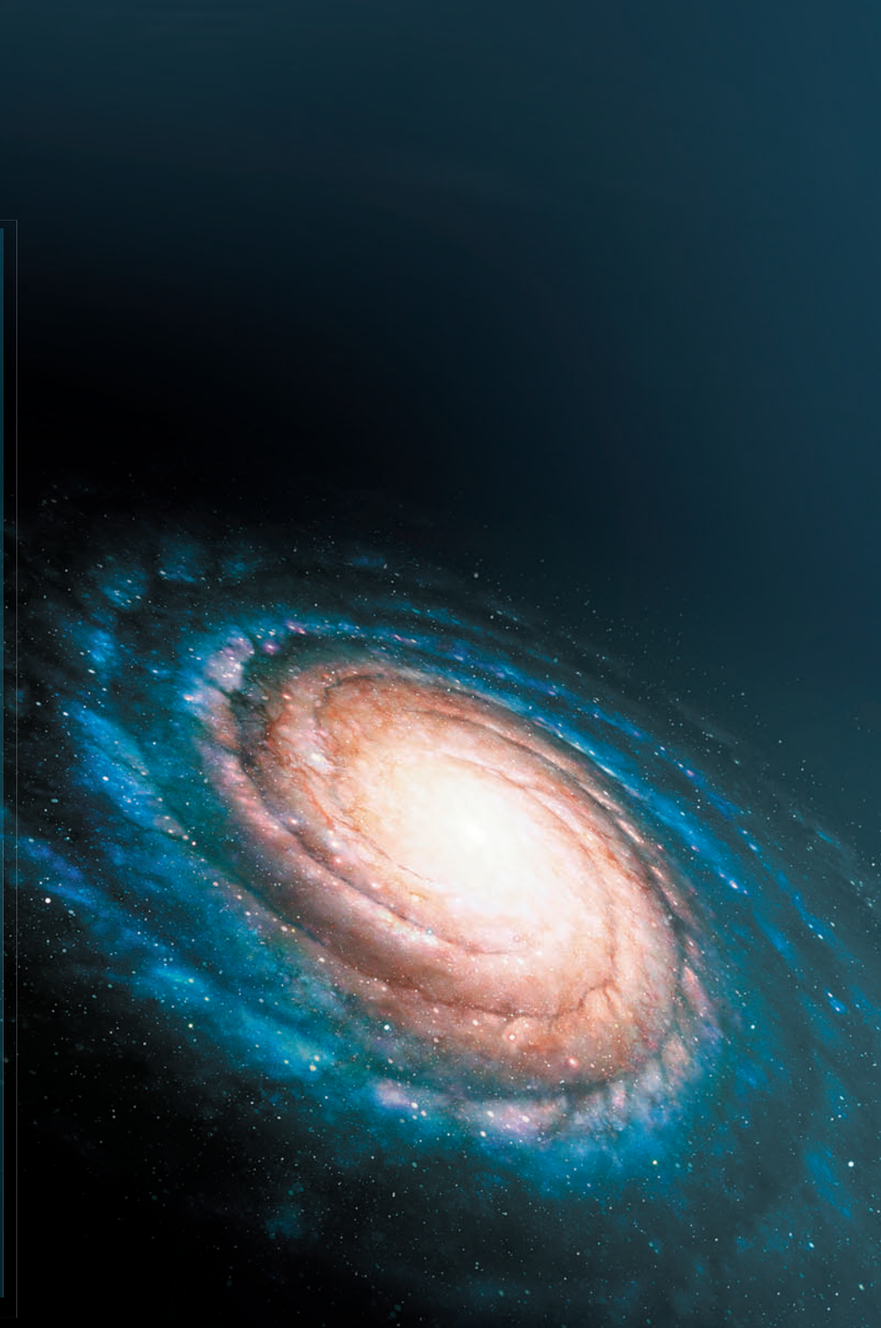

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