

FIRMS CONFERENCE 2022



<https://www.forensic-isotopes.org>

8th Conference of the Forensic Isotope Ratio Mass Spectrometry Network



The conference will be held virtually, featuring oral and poster presentations



5, 6 and 7 October 2022

Welcome

On behalf of the Steering Group, I would like to welcome you to the 8th Triennial Conference of the Forensic Isotope Ratio Mass Spectrometry (FIRMS) Network. This year marks a special milestone for FIRMS as we celebrate 20 years since the organization was formed in 2002 by Sean Doyle and Max Coleman.

The conference will be held virtually over three days, 5th – 7th October 2022, with 20 presentations and speakers representing 14 countries. The conference is an opportunity to hear from researchers and practitioners engaged in forensic applications of stable isotope techniques. This year's program has presentations covering a range of topics including illicit drugs, human remains, chemical criminalistics, food authentication and analytical methodologies.

I would like to acknowledge the hard work of the FIRMS Scientific Conference Committee (Lesley Chesson and Kylie Jones), the support of the FIRMS Directors (Jim Carter, Sean Doyle and Philip Dunn) and all the behind-the-scenes assistance provided by The Chartered Society of Forensic Sciences. I would also like to extend my gratitude to the sponsors ThermoFisher Scientific and Elementex.

I look forward to seeing you all online and hope you enjoy the conference.

Best wishes

Dr Helen Salouros
Chair of FIRMS



FIRMS Scientific Conference Committee:

Lesley Chesson - FIRMS and Defense POW/MIA Accounting Agency Laboratory

Kylie Jones - FIRMS and Australian Federal Police

Helen Salouros - FIRMS and National Measurement Institute of Australia



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KEYNOTE SPEAKERS

DAY TWO

Martian microbes (... and forensics?)***Max Coleman***

Max Coleman is Principal Scientist and Senior Research Scientist at NASA JPL, Caltech.

He has many decades' experience of mass spectrometry, stable isotope science and technique development and has published more than 140 peer-reviewed papers. Some of his published methods are now standard analytical approaches. His training combined chemistry, geology and isotope geochemistry at London and Leeds Universities, but he found that he needed to encompass microbiology too when studying sedimentary rocks.



His career has spanned all sectors of employment: successively, UK government science, oil industry, academia and now with NASA he is looking for life outside the Earth, developing approaches using minerals as signs of life and developing instruments to detect them. He has applied multidisciplinary fundamental scientific research to elicit solutions to practical problems in petroleum exploration and production, environmental pollution, radioactive waste storage and forensic science. In 2000 he was PI of a research grant that allowed Sean Doyle and him to establish the FIRMS network.

DAY THREE

Forensic applications of IRMS for human identification***Lesley A. Chesson***

Lesley Chesson is Forensic Chemist and the functional area manager for the isotope program at the DPAA Laboratory – Hawaii. She received her B.S. in Biology at Elon University (2002) and her M.S. in Biology at the University of Utah (2009).



Lesley is a member of the Forensic Isotope Ratio Mass Spectrometry (FIRMS) Network and an invited member of its Steering Group. She has published more than 75 articles and book chapters. She recently co-edited the book *Food Forensics: Stable Isotopes as a Guide to Authenticity and Origin*.

Lesley has used isotope forensic techniques for more than 20 years to examine documents, drugs, explosives, feathers, microbes, and water. She has also used the techniques to answer questions of food and beverage adulteration, authentication, and origin. One of her current areas of focus is assisting in investigations of unidentified human remains.

Sydney Time <i>5th October</i>	London Time <i>4th October</i>	Day 1—5th October 2022 Chair—Helen Salouros
5.30-5.45am	7.30-7.45m	Welcome address <i>Helen Salouros</i> <i>National Measurement Institute Australia</i>
5.50-6.10am	7.50-8.10pm	Multi-isotope analysis of green and roasted coffee for food authenticity <i>Philip J H Dunn LGC Ltd</i>
6.15-6.35am	8.15-8.35pm	Comprehensive aromatic profile of lavender essential oils by GC-IRMS and GC-MS/MS <i>Purna Kumar Khatri Fondazione Edmund Mach</i>
6.40-7.00am	8.40-9.00pm	Detecting wildlife laundering in the Amazon using a multi-isotope approach <i>Rodrigo R Mayrink</i> <i>Brazilian Federal Police and University of Brasília</i>
<i>7.00-7.10am</i>	<i>9.00-9.10pm</i>	BREAK
7.15-7.35am	9.15-9.35pm	Detection of adulteration of red yeast rice supplements <i>Kevin Kubachka US FDA\Forensic Chemistry Center</i>
7.40-8.00am	9.40-10.00pm	Once upon a twine, the Donna Steele murder investigation <i>Tony Peter Queensland Health Forensic and Scientific Services</i>
8.05-8.25am	10.05-10.25pm	Isotopic comparison of 3D printing filaments <i>Kylie Jones Australian Federal Police</i>
8.30-8.50am	10.30-10.50pm	The effects of fingerprinting and weathering on isotope ratio results of soft polymeric material <i>Joe Meikle Australian Federal Police/Griffith University</i>

Sydney Time <i>6th October</i>	London Time <i>5th October</i>	Day 2—6th October 2022 Chair—Lesley A Chesson
5.30-6.10am	7.30-8.10pm	Keynote Speaker Martian microbes (....and forensics?) <i>Max Coleman NASA Jet Propulsion Laboratory</i>
6.15-6.35am	8.15-8.35pm	Compound-specific carbon and hydrogen isotope analysis of volatile organic compounds using headspace solid-phase microextraction <i>Lidija Stronjnik</i> <i>Department of Environmental Sciences Slovenia</i>
6.40-7.00am	8.40-9.00pm	How multi-level isotopic calibration can improved precision and accuracy in the normalisation of urinary steroid $\delta^{13}\text{C}$ – A case study using recent certification of MX017 urine CRM <i>Fong-Ha Liu National Measurement Institute Australia</i>
7.00-7.10am	9.00-9.10pm	BREAK
7.15-7.35am	9.15-9.35pm	Sponsor Presentation The next leap forward in GAS IRMS <i>Dr Qiong Li & Dr Mario Tuthorn Thermo Fisher Scientific</i>
7.40-8.00am	9.40-10.00pm	Isotope Ratio Mass Spectrometry (IRMS) based evidence passes the test <i>Sean Doyle Linked Forensic Consultants Ltd</i>
8.05-8.25am	10.05-10.25pm	The isotopic compositions of hexamine solid fuel tablets <i>James F Carter</i> <i>Queensland Health Forensic and Scientific Services</i>
8.30-8.50am	10.30-10.50pm	The evaluation of hydrogen isotope ratios in the classification of coca leaf source cocaine in Australia <i>Alexandra Doddridge</i> <i>National Measurement Institute Australia</i>

Sydney Time <i>7th October</i>	London Time <i>6th October</i>	Day 3 —7th October 2022 Chair—Kylie Jones
5.30-6.10am	7.30-8.10pm	Keynote Speaker Forensic applications of IRMS for human identification <i>Lesley A Chesson</i> <i>Defense POW/MIA Accounting Agency (DPAA) Laboratory USA</i>
6.15-6.35am	8.15-8.35pm	Amino acid hydrogen isotope measurements of scalp hair for region of origin studies <i>Christy J Mancuso University of New Mexico</i>
6.40-7.00am	8.40-9.00pm	The state of oxygen and strontium isotopic databases supporting geolocation in North America: A data review and compilation <i>Kirsten Verostick University of Utah</i>
7.00-7.10am	9.00-9.10pm	BREAK
7.15-7.35am	9.15-9.35pm	Multi-isotopes in human hair: A tool to initiate cross-border collaboration in international cold cases <i>Clement P Bataille University of Ottawa</i>
7.40-8.00am	9.40-10.00pm	Effect of diet on the carbon and nitrogen concentration and stable isotope composition of human hair and nails – A study on a tropical and isolated population <i>Gunjan Agrawal</i> <i>Indian Institute of Science Education and Research Kolkata</i>
8.05-8.25am	10.05-10.25pm	BITACORA: A modern-human tissues (teeth, keratin) isotopic database for Argentina <i>Luciano O Valenzuela</i> <i>CONICET-Laboratorio de Ecología Evolutiva Humana, Argentina</i>
8.30-8.40am	10.30-10.40pm	Closing address <i>Helen Salouros</i> <i>National Measurement Institute Australia</i>

DAY ONE

Multi-isotope analysis of green and roasted coffee for food authenticity

Philip J H Dunn, Dmitriy Malinovskiy, Heidi Goenaga-Infante

LGC Ltd, Queen's Road, Teddington, Middlesex, TW11 0LY, UK

Coffee beans are grown in equatorial regions around the world. In the UK alone, over 95 million cups of coffee are drunk per day while the UK coffee industry contributes over £9 billion to the economy per year. As there is a market for coffees of specific geographical origin, there is a need to establish analytical techniques that can be used to help verify labelled origin claims.

Stable isotope analyses of various elements have been previously used to provide indications of geographical origin of various food stuffs with protected geographical origin including wines and cheeses. For use to verify geographical origin, databases of the isotope ratios of known-origin samples must be established. For maximum utility, these databases should use prescribed, validated methods that provide traceable results such that additional analyses can be performed and added to the database without introducing biases.

In this work, the foundations of a coffee stable isotope database have been established. Authentic coffee samples were obtained from the British Coffee Association from all major coffee producing countries. Four bulk isotope ratios were obtained for each coffee sample: $\delta^{11}\text{B}$, $\delta^{13}\text{C}$, $\delta^{18}\text{O}$ and $^{87}\text{Sr}/^{86}\text{Sr}$ using validated methods for EA-IRMS, HTC-IRMS and MC-ICP-MS. Measurement uncertainties were estimated that included contributions from sample preparation and analysis but excluded some contributions such as seasonal and/or yearly variations due to the materials provided to date.

Comprehensive aromatic profile of lavender essential oils by GC-IRMS and GC-MS/MS

Luana Bontempo, Purna K. Khatri, Mauro Paolini, Luca Ziller, Dana Alina Magdas, Olivian Marincas, Alberto Roncone, Roberto Larcher

Fondazione Edmund Mach, via Mach 1, 38098 San Michele all'Adige, Italy

Lavender (*Lavandula angustifolia*) essential oil is a high price premium product used in perfumery, cosmetics or added to food products that must be obtained only from *L. angustifolia* species but is frequently adulterated by the addition of lavandin (*L. x hybrida*, not edible) or synthetically derived products. Compound Specific Isotopic Analysis Gas Chromatography-Isotope Ratio Mass Spectrometry (GC-IRMS) combined with Gas Chromatography-tandem Mass Spectrometry (GC-MS/MS) analyses of volatile organic compounds from lavender essential oil can be a powerful tool for the verification of the authenticity of this product similarly to what has already been done for many food products.

In this study, for the first time, a method for the determination of volatile organic compounds by GC-MS/MS and one for the measurement of the stable isotope ratios of carbon and hydrogen in the main aromatic compounds by GC-IRMS are presented. Both the methods were validated and tested for the analyses of real lavender essential oils samples. In particular, a single chromatographic run was optimised to separate thirty-eight pure volatile organic compounds (as reference standards) and seventy-nine (in real samples) in less than 17 minutes using a capillary column, a faster injection system, temperature ramp gradient and acquired mass spectra through dynamic monitoring reaction and full scan mode. The developed GC-IRMS method, allowed the measurement of $\delta^{13}\text{C}$ and $\delta^2\text{H}$ values of thirty-two compounds (e.g., linalool, linalyl acetate, eucalyptol, etc.) in real lavender essential oil samples.

The performance of the two new methods and the critical related aspects we encountered in their development will be here presented.

DAY ONE

Detecting wildlife laundering in the Amazon using a multi-isotope approach

Rodrigo Ribeiro Mayrink, Camilla Vasconcelos Kafino, Fábio José Viana Costa, Luiz Antonio Martinelli, Paulo Cesar Machado Andrade, Roberto Ventura Santos, Gabriela Bielefeld Nardoto

Brazilian Federal Police and University of Brasília, Rua Nascimento Gugel, 30 – Gutierrez, Belo Horizonte/MG Brazil, 30.441-170

Poaching and illegal trade of river turtles is the main wildlife issue in the Brazilian Amazon. Intense overexploitation brought species to the brink of extinction so decades ago the Brazilian government established legal permission for commercial breeding aimed at reducing illegal trade. However, scientific literature and seizure reports reveal the persistence of turtle trafficking. Moreover, Brazilian Federal Police investigations have found suspicion that commercial breeders perform wildlife laundering by trading poached animals as being legally raised in captivity. In this study we applied a multi-isotopic approach to detect wildlife laundering at a turtle dealer located in a large city in the Brazilian Amazon. Forensic scientists from the Brazilian Federal Police inspected and collected scute samples from a batch of animals of the species *Podocnemis expansa* (Giant South American River Turtle) kept alive for sale. Isotopic analyses were performed for carbon and nitrogen (EA-IRMS) and strontium (LA-ICPMS), and results were compared with values from free-living and captive turtles as well as with previously published data on dissolved strontium isotopes in rivers of the Amazon basin. Among the five animals inspected (supposedly captive-raised), three did not show $\delta^{13}\text{C}$ values consistent with captive turtles (fed commercial feed with C4 isotopic signature, $\delta^{13}\text{C}$ [scute] = -14.4 ± 0.6934), but instead similar to free-living animals (natural C3 forest diet, $\delta^{13}\text{C}$ [scute] = -25.9 ± 1.002). These three turtles also showed high uniformity in strontium isotopic ratio, with $^{87}\text{Sr}/^{86}\text{Sr}$ values between 0.713 and 0.715; a range distant from the other two inspected animals (0.723 and 0.738) and in line with free-living turtles from the lower Amazon River. Comparing turtle scute strontium ratios with Amazonian sub-basins water $^{87}\text{Sr}/^{86}\text{Sr}$ data we also found that chelonians reflect the strontium isotopic ratio of Amazonian rivers, which strengthens the consistency of our geographic origin inferences. The isotopic approach was effective in detecting wildlife laundering and in pinpointing the likely region of origin of trafficked animals.

Detection of adulteration of red yeast rice supplements

Kevin Kubachka, Joshua Sabala, Kristen M. Hannon, Madhavi Mantha, Lisa Lorenz, John. P. Roetting II

US FDA\Forensic Chemistry Center, 6751 Steger Drive, Cincinnati, OH 45237, USA

Red Yeast Rice (RYR) is a dietary supplement containing monacolin K, a natural statin marketed for its ability to lower cholesterol. These supplements may be adulterated with lovastatin, a pharmaceutical drug proven to lower cholesterol, to boost the supplements' efficacy. Monacolin K and lovastatin have identical chemical structures, proving them difficult to differentiate in testing. However, monacolin K is derived from the mold *Monascus purpureus* grown on rice, a C3 plant, while lovastatin is fermented from the fungus *Aspergillus terreus* using mostly C4 plant sources. The stable carbon isotope ratios ($\delta^{13}\text{C}$) differ between these two plant groups and this difference is passed on to monacolin K and lovastatin, respectively. This study seeks to develop preparation methods to analyze for $\delta^{13}\text{C}$ values and determine adulteration in the complex matrices of RYR dietary supplements.

Various sample preparation and analysis methods were explored, each ultimately used with isotope ratio mass spectrometric (IRMS) detection. Locally and internationally marketed RYR supplements were analyzed. Based on $\delta^{13}\text{C}$ values analyzed using solid phase extraction with fraction collection and combustion by an elemental analyzer coupled to IRMS (SPE-FC-EA-IRMS), multiple samples were determined to contain lovastatin, hence being classified as adulterated. Additionally, analysis by gas chromatography with combustion using IRMS (GCC-IRMS) was also explored, and results compared to the previously established SPE-FC-EA-IRMS method.

DAY ONE**Once upon a twine, the Donna Steele murder investigation**

Tony Peter, James F Carter, Jasper R Bowman, Sarah L Cresswell

Queensland Health Forensic and Scientific Services, 39 Kessels Road, Coopers Plains, Qld 4108, Australia

In 2017, the body of Donna Steele was found in an offshoot of the Endeavour river near Cooktown, Queensland. Ms Steele's body had been wrapped in a duvet and weighted down with rocks. A length of red coloured twine was found alongside the partially decomposed body. Two similar lengths of red coloured twine were found at Ms Steele's home, where it was believed that Ms Steele had been murdered. One of these lengths of red coloured twine carried the DNA profile of an unknown male, the suspected killer. Examination and analysis conducted on these lengths of twine by the Trace Evidence group and by the Organic Chemistry laboratory using methodologies and instrumentation available at the time provided supporting evidence for the hypothesis that the length of twine found on the body of the deceased could not be excluded as sharing a common origin with the two lengths of twine found at the home of Ms Steele. This presentation will cover the results of research undertaken by the Trace Evidence group and by the Organic Chemistry laboratory to develop an enhanced, systematic protocol for the comparison of synthetic twines and to establish the weight of evidence that can be presented in similar case work.

Isotopic comparison of 3D printing filaments

Kylie Jones, Joe Meikle, Joanna Bunford

Australian Federal Police, Canberra, ACT, Australia

The 3D printing of firearms is becoming an increasingly common method of attempting to produce an illegal firearm, particularly in countries with strict firearm ownership legislation such as those in effect in Australia. The types of firearms produced using these methods have changed over time, with the most current models a hybrid of 3D printing and machined parts. Instructions for how to produce these devices are readily available online and 3D printing itself is now freely accessible and low cost. If manufacture is successful, this provides criminal organisations with a means to both arm themselves, or to on sell these firearms for profit.

Projects to better understand how easy it is to produce these firearms, their effectiveness and lethality are underway within AFP forensics. Alongside this, work is required to ensure that laboratory-based disciplines are positioned to respond to requests for comparison, when items including the firearms, printers and filaments are seized and need to be compared. As a type of polymer examination, the use of Isotope Ratio Mass Spectrometry (IRMS) is the focus of current work to establish both a method, known materials database, and to understand how materials may change during printing or use.

This presentation will give an overview of the current project plan and aims and will explore the first set of results obtained which explore the variability and stability of $\delta^2\text{H}$, $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ isotope ratio abundances of Poly Lactic Acid (PLA) filaments.

DAY ONE

The effects of fingerprinting and weathering on isotope ratio results of soft polymeric material

Joe Meikle, Kylie Jones, Jim Carter, Sarah Cresswell, Carney Materson, Sue Boyd

Australian Federal Police/Griffith University, Canberra, ACT, Australia

When soft polymers such as cling films and resealable bags are submitted for forensic chemical comparisons, the techniques that can be applied are limited by environmental effects that have the potential to change the results of instrumental analysis. Soft polymers are commonly required to undergo fingerprint analysis and chemical comparisons. Many fingerprinting techniques coat the samples in fingerprinting agents which reduces the ability to analyse these samples using techniques that analyse the surface of the sample. Polymers submitted as evidence can also be dirty or have been subject to extended periods outdoors exposed to the weather. Weathering oxidises and breaks down the polymer which can impact comparison of isotope ratio measurements as these samples are normally compared to seized samples that have not been exposed to the same conditions.

Commonly used forensic techniques such as Fourier transform infrared spectroscopy and birefringence analysis may be limited when samples are distorted or dirty. Two of the limitations are when samples have been subject to harsh weathering or fingerprinting. Isotope Ratio Mass Spectrometry is a powerful tool for the forensic comparison of soft polymers and has the potential not to be affected by these limitations.

This study examined the effects of fingerprinting treatments and weathering on the isotopic compositions of polyethylene materials. Fingerprinting techniques using cyanoacrylate and vacuum metal deposition were performed on samples of resealable bags with previously measured isotopic compositions to determine compositional changes. Another experiment measured the effects of samples being exposed to the elements for two years in the Canberra (Australia) climate on the isotope ratio results of cling films and resealable bags. This study will inform future casework examinations when samples have been fingerprinted or subject to weathering effects.

DAY TWO

KEYNOTE SPEAKER

Martian microbes (... and forensics?)**Max Coleman**

NASA Jet Propulsion Laboratory, California Institute of Technology, 4800 Oak Grove Drive, Planetary Mass Spectrometry Group, MS 300-123G, Pasadena, CA 91109, USA

Reading the title, an immediate reaction might be, what's the connection? This paper's aim is to convince that there are analogous ways of working and to share commonalities of forensic and astrobiological science. Looking for Life on Mars is quite hard, especially done robotically tens or hundreds of millions of miles away.

It is likely that the Earth's surface was habitable shortly after its formation just over 4.5 billion years ago. Microbial life dominated until about 0.5 billion years ago and signs of microbial activity are the prime targets of exploration for life on Mars. Reviewing the biological characteristics of life showed that metabolic activity was one of the most recognizable. Amazingly, exhaled "breath" of microorganisms is one of the best signs of Life, preserved in minerals and recognized by chemical and isotopic compositions. Nevertheless, it is essential to examine preserved or fossilized microbes themselves as unambiguous signs of life.

Obvious similarities between the two sciences are the need for rigorous analytical standardization and demonstration that the probability of similar or dissimilar compositions represents valid evidence. However, aspects of the astrobiological approach include at least two, seldom or never used in forensic science but which might be useful. Life on Mars probably is sparse, but microbes might form isolated discrete, concentrated colonies. Potentially analogous Earth systems show that mineralized microbial metabolic exhalation is a perfect pathfinder for unambiguous biochemical markers. Also, part of NASA's ethos is communicating what it does to the general public, which pays for the work. This is the other major commonality, communication to a non-scientific audience, although the same principles apply to communicating to one's scientific peers. The challenges of unambiguously communicating complex information are universal, but astrobiology may have some approaches not yet used in forensic science, but which may be transferable.

DAY TWO

Compound-specific carbon and hydrogen isotope analysis of volatile organic compounds using headspace solid-phase microextraction

Lidija Strojnik, Federica Camin, Nives Ogrinc

Department of Environmental Sciences, Jožef Stefan Institute, 1000 Ljubljana, Slovenia

Despite the sophisticated techniques available, food flavours' characterisation and authenticity assessment remain challenging. The introduction of compound-specific isotope analysis, however, means that it is now possible to distinguish natural aromas from synthetic ones based on the isotopic values of VOCs. The result is limited to a few common aroma compounds present in different fruits and is based on a small number of samples produced using time-consuming extraction procedures and significant amounts of organic solvents. This work develops a methodology based on HS-SPME extraction technique coupled with GC-C-IRMS and GC-P-IRMS to determine $\delta^{13}\text{C}$ or $\delta^2\text{H}$ values for several VOCs within the same run with a focus on establishing a procedure for routine laboratory application.

To achieve reproducible and accurate results, a combination of a multiple-point isotopic linear normalization method, and nonlinear correction (calibration of the output of the IRMS instrument in terms of isotope ratio based on different analyte signals – peak heights), was used. The method gives reproducible and accurate results for different chemical classes of VOCs over varying concentrations in the same analysis. This approach can significantly improve the measurement error of small peaks (below 1 nA) from 3 ‰ to 0.5 ‰. Special care was taken also to avoid irreproducible isotopic fractionation and the effects of equilibration, adsorption, desorption times and temperatures on $\delta^{13}\text{C}$ or $\delta^2\text{H}$ values were examined. Optimising HS-SPME parameters is critical to avoid isotopic fractionation and minimise method error. The average combined measurement uncertainty (MU) for the method for $\delta^{13}\text{C}$ was 0.42‰. All the $\delta^{13}\text{C}$ values were below $\pm 3\text{MU}$, regardless of analytical conditions. In contrast, for $\delta^2\text{H}$ values, only temperature below 30 °C, with an equilibration time of 15 min and adsorption time between 10 and 20 min, produced a noticeable effect (<10 ‰).

The results show that selected HS-SPME extraction method can overcome issues of solvent use, large sample volumes, need for concentrated samples, long analysis times, isotope fractionation and irreproducible results. It also avoids matrix effects, making it suitable for analysing complex food products. Moreover, we found that method optimisation can minimise MU, and data normalisation and validation are essential for obtaining meaningful data in flavour authenticity studies. Finally, developed method was used to determine the authenticity of commercial products using fruit, vanilla, and truffle flavourings.

DAY TWO

How multi-level isotopic calibration can improved precision and accuracy in the normalisation of urinary steroid $\delta^{13}\text{C}$ – A case study using recent certification of MX017 urine CRM

Fong-Ha Liu, Lesley Johnston, Jeff Merrick, Mark Lewin, Raluca Iavetz

National Measurement Institute, 105 Delhi Road, North Ryde, NSW 2113, Australia

Gas chromatography-combustion-isotope ratio mass spectrometry (GC-C-IRMS) has been used by the World Anti-Doping Agency (WADA) laboratories for confirmatory analysis of administration of synthetic forms of endogenous anabolic androgenic steroids (EAAS) when an atypical steroid urinary profile is observed (TD2021IRMS). This technique is able to differentiate synthetic forms of endogenous steroids based on the $^{13}\text{C}/^{12}\text{C}$ ratio, expressed as $\delta^{13}\text{C}$ value. Application of appropriate steroid CRM similar to target steroids in sample for calibration assist in fractionation control during analysis. This is the key to achieving accuracy and global comparability of results between WADA laboratories.

The National Measurement Institute of Australia (NMIA) has developed a new calibration approach to normalising carbon isotope delta measurement of urinary steroids using a pure steroid MX018 CRM consisting of thirteen steroids distributed into three ampoules. This material facilitates calibration over a wide isotopic range ($\delta^{13}\text{C}_{\text{VPDB}}$ from -13.58 to -31.63 ‰) with adherence to identical treatment of sample and reference material via the GC-C-IRMS. The measured $\delta^{13}\text{C}$ value for both sample and CRM were expressed as relative to an approximate $\delta^{13}\text{C}$ value of the internal reference CO_2 gas. A linear regression generated by plotting the certified $\delta^{13}\text{C}_{\text{VPDB}}$ value against measured $\delta^{13}\text{C}$ value ($R^2=0.99$ or better) then facilitate the normalisation of measured sample delta value to the VPDB scale. This calibration approach applied in the certification of eleven steroid markers in MX017 urine CRM resulted in significant improvement in precision and measurement uncertainty in the assigned reference $\delta^{13}\text{C}_{\text{VPDB}}$ values. There is a potential for this approach to be applied in other forensic compound specific analysis applications where precision and accuracy is critical

DAY TWO**The next leap forward in GAS IRMS**

Dr Qiong Li & Dr Mario Tuthorn

Thermo Fisher Scientific, Hanna-Kunath-Straße 11, 28199 Bremen, Germany

The next generation Thermo Scientific™ DELTA Q™ IRMS is the world's first net zero mass spectrometer. It is designed to be coupled seamlessly with a wide range of Thermo Scientific™ peripherals to address diverse applications including food authenticity, criminal and environmental forensics, doping control, and scientific research without costing the Earth. Here we provide an overview of our Gas IRMS systems and offer users comprehensive instrumental setups for their analytical requirements.

With the introduction of the DELTA Q IRMS, we have also made the next leaf forward - Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution (ISDS) Software for stable isotope ratio analysis. It is carefully optimized to revolutionize the gas IRMS analyses, delivering simplicity, efficiency, and quality in users' laboratories. Built to cater for the most diverse applications, Qtegra ISDS Software provides users with the level of control that is required, within a logical, easy-to-use framework, enabling users to dramatically improve their productivity.

Isotope Ratio Mass Spectrometry (IRMS) based evidence passes the test

Sean Doyle, John Howa, Thuan Chau, Mike Lott

Linked Forensic Consultants Ltd, PO Box 2193, Raumati Beach 5255, Paraparaumu, New Zealand

Although IRMS data and its interpretation have been used in a number of high-profile investigations over many years, and sometimes adduced as evidence (*City of Pomona v SQM North America Corp*, *Landis v USADA*, *United States v Richard Colvin Reid*), the technique, the interpretation of the data generated and the expertise of those presenting the evidence had not been subjected to a US judicial admissibility hearing, until recently.

In the United States of America, judges have a gatekeeping role in admitting scientific and, more generally, expert evidence. In deciding whether to admit such evidence, judges are required to apply a number of criteria documented as Federal Rule of Evidence (FRE) 702 which individual States might adopt as their own, as in this case; Colorado Rule of Evidence or CRE 702.

The main criteria applied may be summarised as follows;

1. The technique has been tested in actual field conditions (and not just in a laboratory).
2. The technique has been subject to peer review and publication.
3. The known or potential rate of error has been determined.
4. Standards exist for the control of the technique's operation.

The technique has been generally accepted within the relevant scientific community. In the case of the People of the State of Colorado (Plaintiff) v Ceus and Archer (Defendants) [2019], IRMS-based evidence and the expertise of those presenting that evidence were subject to an admissibility or Shreck hearing. That evidence was found to meet the criteria and deemed admissible. Strictly, this decision only applies to the State of Colorado in the USA. However, the finding of admissibility in one State should support admissibility in other States and in other common law jurisdictions.

This case was briefly discussed as part of a presentation at the last FIRMS Conference but, given the outcome of the Shreck hearing and its wider impact, warrants a more detailed exposition. The presentation will provide the salient details of the hearing and the court's decision, accepting the robustness of IRMS-based evidence and the expertise of those presenting the evidence.

DAY TWO

The isotopic compositions of hexamine solid fuel tablets

Tony Peter, **James F Carter**, Emily Bennett, Shalona R Anuj

Queensland Health Forensic and Scientific Services, 39 Kessels Road, Coopers Plains, Qld 4108, Australia

Hexamine, derived from solid fuel tablets, is a potential source of the home-made organic peroxide explosive hexamethylene triperoxide diamine (HMTD). Therefore, a means to characterise hexamine samples and to link HMTD with precursor hexamine may provide investigative intelligence to counter-terrorism agencies.

In this presentation we report the hydrogen, carbon, and nitrogen isotopic compositions of hexamine from retail samples of solid fuel tablets. Despite some within- and between-tablet inhomogeneity, a combination of these data was found to be characteristic of specific brands or manufacturers of solid fuel tablets.

Ten samples of hexamine were each used to synthesize four batches of HMTD using two isotopically distinct sources of hydrogen peroxide.

The changes in isotopic composition observed, progressing from hexamine to HMTD, could be explained by a reaction mechanism involving the decomposition of hexamine to methylimine and formaldehyde. Data also supported a mechanism whereby all of the hydrogen atoms of HMTD were derived from hexamine and not from other reagents.

A rule-of-thumb is proposed to identify possible hexamine precursors for HMTD based on the hydrogen and carbon isotopic composition.

The evaluation of hydrogen isotope ratios in the classification of coca leaf source cocaine in Australia

Alexandra Doddridge, Helen Salouros, Julian Russo, Sasha Vujic, Kate Malone, Matilda Woods

National Measurement Institute, 105 Delhi Road, North Ryde NSW 2113, Australia

Cocaine is submitted to the National Measurement Institute- Australia (NMIA) for chemical profiling by law enforcement. The NMIA's Cocaine Profiling Program analyses cocaine seized at the national and domestic level. The samples are subject to three different types of analyses, each looking at a specific chemical element which can be used to infer the origin and processing location of the cocaine.

Headspace analysis of occluded solvents can be used to determine processing location, while truxilline and alkaloid ratios are used to determine the geographical source of the Coca leaves from one of three main harvesting areas: Bolivia, Colombia and Peru. Differences in weather and geography of these growing regions in South America are also reflected in the $\delta^{13}\text{C}$, $\delta^{15}\text{N}$ and $\delta^2\text{H}$ stable isotope ratios of the cocaine and are useful in the inference of geographical source.

Currently $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ stable isotope ratios have proven to be fit-for-purpose by this laboratory for geographical classification when considered with a sample's tropacocaine, truxilline and trimethoxycocaine content. The primary aim of this study was to determine the $\delta^{13}\text{C}$, $\delta^{15}\text{N}$ and $\delta^2\text{H}$ stable isotope ratio values of cocaine samples submitted between Jan 2021 and June 2022. The results of these analyses will be presented and discussed with a particular focus on the value of the hydrogen data in regards to providing strategic and tactical information.

DAY THREE

KEYNOTE SPEAKER

Forensic applications of IRMS for human identification***Lesley A. Chesson****Defense POW/MIA Accounting Agency (DPAA) Laboratory, 590 Moffet St., Bldg 4077, JBPHH, HI 96853 HI, USA*

Forensic applications of isotope ratio mass spectrometry (IRMS) to human skeletal remains have become an important tool in investigations of human history and residential behavior. Isotopic variations in bioapatite, collagen, enamel, and keratin reflect variations in individuals' diet and drinking water. Since food and water sources typically are geographically linked, isotope testing can thus assist in human identification efforts by classifying remains to a likely geographic or population origins. If remains are commingled, differences in diet or geographic origin also can support their individuation.

All forensic methods and technologies are only as strong as the underlying data and validations. Good laboratory practice must be used in the acquisition of all stable isotope ratio data – particularly in forensic settings, if they are to be used in courts of law. Using lessons learned from the (ongoing) development of an isotope testing program at the Defense POW/MIA Accounting Agency (DPAA) Laboratory, this presentation will discuss isotope data quality management in human identification efforts. Questions asked during program buildout included:

1. How well are the mass spectrometer and associated peripheral(s) operating?
2. Are test results repeatable (over time and between locations)?
3. How variable are our samples/controls?
4. Have the samples been inadvertently isotopically fractionated since time of collection?
5. How variable are the populations of interest?
6. Can we differentiate between populations that we would like to?

While the focus of this presentation is on applications of IRMS to human remains, the questions posed above are important to consider whenever isotope testing is used in the forensic sciences. Whether the populations of interest are olives or humans, honey or TNT, users need assurances that their stable isotope ratio data and interpretations are scientifically sound and legally defensible.

DAY THREE

Amino acid hydrogen isotope measurements of scalp hair for region of origin studies

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Stable isotope analyses have become an important analytical tool in forensic sciences for sourcing the origins of biological materials. Bulk tissue analysis of hydrogen ($\delta^2\text{H}$) stable isotopes in human keratin tissues have been widely used to reconstruct an individual travel histories or determine regions of origin. Here, we extend this approach to consider $\delta^2\text{H}$ analysis of individual amino acids in order to examine the influences of drinking water and dietary contributions to $\delta^2\text{H}$ values in human scalp hair using scalp hair and tap water from 28 locations across the United States. We found that $\delta^2\text{H}$ values of non-essential amino acids varied systematically with tap waters across continental-scale gradients. Specifically, 42-51% of the hydrogen atoms in alanine, glutamic acid, and glycine were derived from drinking waters, which is a significantly larger contribution than the 27% contribution observed in bulk tissue scalp hair. It was hypothesized that non-essential amino acids should derive a larger proportion of their hydrogen atoms derived from drinking water (body water) because they (a) can be synthesized by de novo synthesis and (b) are structurally simpler with a larger proportion of hydrogen atoms that can freely exchange with body water. PCA analyses of amino acids, drinking waters, bulk scalp hair, and geographic location clustered into four regional areas. Our findings suggest that hydrogen isotope analyses of non-essential amino acids could help improve models for geolocating the origins of unidentified human decedents as well as wildlife forensics.

The state of oxygen and strontium isotopic databases supporting geolocation in North America: A data review and compilation

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Many recent studies have reported human tissue isotope data that relates to or reflects the location of tissue growth. Although the goals of these studies vary, the data they present constitute a collective resource that may document spatial patterns in human tissue isotope ratios and relationships between tissues and environmental proxies (e.g., water, soil). The degree to which this is the case depends on factors including the standardization of metadata, analytical protocols, and data reporting, and the geographic coverage of data from any given tissue and isotope system.

We reviewed >43 studies that use, discuss, or include strontium or oxygen isotopic data from human tissues, including journal articles, reports, book chapters, theses, and dissertations. Tissues represented include hair, bone, nail, teeth, and urine. We compiled metadata on each study's methods and contextual information, and divided the dataset based on the types of geographic and demographic information reported.

Our data analysis focused on 23 studies where the geographic origin of the sampled tissues was known or could be assumed. We systematically evaluated the isotopic data, along with pretreatment protocols, reporting of standards and calibration, and demographic or life history data. The compiled dataset comprised >2,500 isotopic data points, with 55% from the USA, 19% from Mexico, and 27% from Canada; 52% of the data represented hair or nail keratin, 46% bioapatite (primarily tooth enamel), and the remaining samples urine. Geographic coverage and methodological standardization differed among tissues, with the broadest coverage available for keratin; however, a large fraction of these data lack true 'known' geographic origin, limiting the utility of the dataset. This data review highlights strengths and weaknesses of the current tissue data supporting isotope-based human geolocation, allows us to propose guidelines for future reporting of data and methods, and may help to guide future sampling efforts.

DAY THREE

Multi-isotopes in human hair: A tool to initiate cross-border collaboration in international cold cases

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Unidentified human remains have historically been investigated nationally by law enforcement authorities. However, this approach is outdated in a globalized world with rapid transportation means, where humans easily move long distances across borders. Cross-border cooperation in solving cold-cases is rare due to political, administrative, or technical challenges. It is fundamental to develop new tools to provide rapid and cost-effective leads for international cooperation. In this work, we demonstrate that isotopes are effective screening tools to help identify cold cases with potential international ramifications. We first complete an existing database of hydrogen and sulfur isotopes in hair of human residents across North America by compiling or analyzing hair from Canada, the United States and Mexico. Using these databases, we develop maps predicting isotope variations in human hair across the continent. We demonstrate that both hydrogen and sulfur isotopes in human hair are highly predictable and display strong spatial patterns. Multi-isotope analysis combined with dual hydrogen and sulfur isotope geographic probability maps provide evidence for international travel in two case studies. In the first, we demonstrate that multi-isotope analysis in hair of unidentified border crossers found in the United States, close to the Mexico-US border, help trace their origin back to specific regions of Mexico. These findings were validated by the subsequent identification through the Pima County Office of the Medical Examiner in Tucson, Arizona. In the second case study, we demonstrate that sequential multi-isotope analysis along the hair strands of an unidentified individual found in Canada provides detailed insights into the international mobility of this individual during the last year of life. In both cases, isotope data provide strong leads towards international travel.

Effect of diet on the carbon and nitrogen concentration and stable isotope composition of human hair and nails – A study on a tropical and isolated population

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The carbon and nitrogen stable isotopic composition of human tissues like bone collagen, teeth and hair have been studied extensively for diet reconstruction in archaeological samples and contemporary human beings, following the adage “we are what we eat”. However, it is well known that the stable isotope composition of human tissues is affected not only by the dietary preference of each individual but also by environmental factors, physiological situations, diseases, malnutrition, and starvation. In this study, the effect of dietary composition on the human scalp hair and fingernails is investigated by eliminating the factors affecting the stable isotope ratio other than the diet of the individual. This study correlates the carbon and nitrogen concentration and stable isotope composition of the diet of each participant with their human tissues. The major food items forming their meal plan are analysed alongside their scalp hair and fingernail tissue. This is a large-scale controlled study comprising scalp hair and fingernail samples of 100 individuals. The environmental factors were negated by acquiring the samples in a span of 7 days from a residential campus located in a remote location, restricting frequent travel of the residents. Only healthy individuals who consumed meals at least twice a day in the campus dining facility (a common meal source for all individuals) were included in the study which further eliminated the individual factors. A significant difference was observed between the %N, $\delta^{15}\text{N}$ and $\delta^{13}\text{C}$ of hair and nail tissue. The ovo-lacto-vegetarians had a lower $\delta^{15}\text{N}$ value than omnivores for both hair and nail tissue. This was also evident in the $\delta^{15}\text{N}$ and %N of their diet, which was lower than the diet of omnivores. The study allowed us to distinguish between the ovo-lacto-vegetarians and omnivores based on their C and N concentrations and the isotopic composition of two human tissues. The diet-tissue enrichment value will be useful for dietary analysis and forensic isotope investigations.

DAY THREE

BITACORA: A modern-human tissues (teeth, keratin) isotopic database for Argentina

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As a consequence of the recent history of Argentina related to crimes against humanity, the country has positioned itself as a leader on Forensic Anthropology in human rights contexts. It can even be argued that Forensic Genetics had its origins in human rights work in the early 1980s in Argentina. However, there are still hundreds of unidentified human remains recovered from the dictatorial regimes' crimes. Furthermore, every year there are dozens of modern *Nomen Nescio* (N.N., unnamed persons) buried in public cemeteries, many of which could be immigrants. Thus, there is an urgent need to adopt new biogeochemical techniques that could increase the biological profile of a victim, and therefore increase the chances of positive identifications. One of such initiatives is the development isotopic databases and models that can provide information on the population and region of origin of unidentified victims.

We present the development of a human tissue isotopic database named BITACORA (from its name in Spanish: Base de Información Isotópica de Tejidos Actuales Como Referencia Argentina). BITACORA currently holds isotopic data ($\delta^{18}\text{O}$, $\delta^2\text{H}$, $\delta^{13}\text{C}$, $\delta^{15}\text{N}$ and $\delta^{34}\text{S}$ values) from teeth (enamel and dentine) and keratin tissues (hair, beard, and fingernails) obtained from volunteers of known origin, as well as individual information of the donors (age, gender, biometrics, dietary preferences, and relocation history). BITACORA is associated with a tap water isotope database for the same locations where human samples were obtained. Currently, we have >180 teeth, >290 keratin and >380 water samples gathered from across the country. We present preliminary analyses on the geographic distribution of these isotopic markers, aimed at generating spatial models for region of origin assignment, and provide an overall discussion of the current status and future development of the databases.

Closing Address

POSTERS

Sample preparation and analysis standardization for large-scale tooth enamel carbonate isotope databases

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Isotopic analysis is increasingly used to constrain the life history of individuals in forensic human identification. These applications require comparison of data from samples with reference data, creating a need for isotopic databases and demanding comparability of measurements made on evidence and reference samples. Numerous studies have investigated how laboratory methods affect the comparability of measurements made on fossil or archeological samples, but less work has focused on modern forensic materials. Here we conducted a series of experiments on tooth enamel targeting this issue.

Our results show that Gas Bench-Isotope Ratio Mass Spectrometric analysis of samples as coarse particles (>250 μm) reduced CO_2 yield by 1.02%, increased the variability of yield and $\delta^{13}\text{C}$ values (1σ increase of 0.38% and 0.13‰, respectively), and produced slightly higher $\delta^{18}\text{O}$ values relative to analysis of fine particles (<125 μm). Using different particle sizes during sample pre-treatment only affected CO_2 yield, giving a decrease of 0.35% for fine particles. No differences were observed between samples stored in ambient conditions and those stored in a desiccator for 71 days. Samples treated with different oxidant solutions showed a systematic positive $\delta^{18}\text{O}$ shift of $\sim 0.3\text{‰}$ regardless of the chemical, concentration, or treatment time used.

These studies suggest that comparability and consistency of isotopic results can be maximized if enamel samples are ground to a fine particle size after chemical pretreatments. They also support previous work highlighting the potential for chemical pre-treatments to impact measured isotope delta values but suggest that standardized oxidant pre-treatment methods may be less important than consistency in their use (or elimination). In contrast, other methodological details including sample storage conditions appear to have less impact on the isotopic results for modern tooth enamel carbonate, at least across multiple experiments within a single laboratory, and standardization of these methods may be less critical.

The use of stable isotopes in combating animal laundering: What do we know?

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Wild animal trafficking is one of the most widespread and profitable illegal activities in the world, causing impacts on the local and global species and ecosystems. Studies have suggested a relationship between authorized and illegal animals market, where the former is supplied by animals illegally captured, intensifying the illegal trade. The development of tools to identify the real origin of animals is fundamental to preventing and combating animal laundering. Stable Isotopes are important biotrackers of animals' origin. This technique has been widely used to identify animals' geographic origins, and more recently has also been used to track individuals' breeding systems (wild or captive). In this study the potential use of stable isotopes analysis (SIA) as a reliable forensic tool to distinguish between wild and captive animals in vertebrates. We did an extensive search of the literature available and selected 32 papers about the subject. The research relates to forensic questions in different contexts, such as animal laundering, animal-based food certification, and illegal species introduction. $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ are the main isotopes used, followed by $\delta^2\text{H}$, especially when some geographic variation was also involved. Fish is the most studied group followed by mammals and birds. Inert organic tissues were widely used in studies involving living animals, while muscle and bone were mainly used in works related to animal products. SIA has consistently shown the potential to distinguish between wild and captive in different vertebrate groups worldwide. However, local environmental factors seem to have a more significant influence on this potential than universal factors on at large scale. The consistency we found is crucial in forensic contexts and we expect the present study to contribute to expanding the use and acceptance of SIA as a reliable tool in combatting wildlife crime.

POSTERS

Investigating the provenance of Brazilian woods using Sr isotopes*Camilla Vasconcelos Kafino, Roberto Ventura Santos, Gabriel Ribeiro Moizinho, Artur Moraes Amorim**Brazilian Federal Police, SHCGN 711 Bloco N apt 203 Brasília-DF, 70750-774, Brazil*

Tracking timber back to its point of origin is crucial to prevent illegal logging and preserve our natural forests. The geochemistry of strontium isotopes is relatively well known and $^{87}\text{Sr}/^{86}\text{Sr}$ ratios have been used routinely as environmental tracers in geology, hydrology, ecology, and archaeology. Here we present the first applied study utilizing this methodology in Brazilian woods. The underlying philosophy is that trees uptake chemical elements from local soils and incorporate them into the wood, with strontium being absorbed by the plants as a trace element. Thus, $^{87}\text{Sr}/^{86}\text{Sr}$ ratios should provide a model system for provenance studies of trees growing on diverse substrates, therefore isotope ratio of wood can provide a screening tool to assist in ruling whether timber comes from a legal or illegal source. To test this hypothesis the present study used wood from the Brazilian Amazon region and the Atlantic Forest. Two sample digestion approaches were applied to validate our method. In both cases the samples were digested in acid in two stages. In the second approach we added one additional step of acid.

Our preliminary results showed that the $^{87}\text{Sr}/^{86}\text{Sr}$ ratio do not vary within the same individual (roots, trunk, and branches), confirming the absence of isotopic fractionation within the body of the tree. However, it was possible to observe a clear differentiation on the $^{87}\text{Sr}/^{86}\text{Sr}$ ratios in relation to the location of the sample different values of the $^{87}\text{Sr}/^{86}\text{Sr}$ ratio were verified in the rock substrate for each location and these values were indeed reflected in the $^{87}\text{Sr}/^{86}\text{Sr}$ ratios from the wood samples analyzed. These findings highlight the sensitivity of Sr isotopes to variations in the lithology on which trees are seated and thus can be used as an origin tracer for Brazilian woods.

 $\delta^{18}\text{O}$ variation in drinking water in Oaxaca, Mexico and its implications for forensic provenancing efforts*Taylor Lambrigger**California State University, Chico, Gilbert, AZ 85297, USA*

Forensic anthropologists and isotope experts have used oxygen isotopic analyses ($\delta^{18}\text{O}$) in recent decades because of the method's ability to help estimate probable regions of origin of unidentified individuals based on isotopic composition of bodily tissues. Prior research has found that drinking water is geographically patterned and that tap water is often representative of drinking water in assessing these links between human tissues and geography. Forensic anthropologists have begun to apply the method in a variety of global contexts, including assisting in identifying missing migrants at the US-Mexico Border.

The focus of this research is to critically assess the assumption made that tap water is isotopically representative of drinking water in Oaxaca, Mexico. Drinking water samples from 56 municipalities were analyzed and compared with predicted $\delta^{18}\text{O}$ values from the tap water isoscape of Mexico published by Ammer and colleagues (2020). Hair samples from four locations in Oaxaca were also analyzed to compare against various water sources. Hair keratin $\delta^{18}\text{O}$ values were run through multiple models relating the $\delta^{18}\text{O}$ values in hair keratin and drinking water.

Tap water $\delta^{18}\text{O}$ values do not reliably reflect drinking water $\delta^{18}\text{O}$ in sampled regions of Oaxaca, Mexico. Further, the models relating hair keratin to drinking water, built on this assumption, likewise fail to hold predictive power. With a muddled understanding of tap water's ability to represent drinking water isotopically, researchers need to study the role of other key factors in $\delta^{18}\text{O}$ values in non-Western communities. If the individual's we are helping identify with $\delta^{18}\text{O}$ are from non-Western communities, we run the risk of incorrectly measuring their region of origin because of the assumptions in this method. Recognizing the varying sociocultural realities of the communities we seek to aid and identify is paramount as we grow and develop our methods moving forward.

POSTERS

Source identification of ammonium in PM_{2.5} during COVID-19 in Dhaka, Bangladesh

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The pandemic caused by coronavirus disease 2019 (COVID-19) in Wuhan, China, is a worldwide problem. Lockdown measures were implemented in many parts of the world to prevent the spread of the infection. During the lockdown measures, although most air pollutants were reported to have decreased, some pollutants were also reported to have increased to be compared with pre-COVID-19 levels. The contradiction is still not fully understood. In addition, Dhaka, Bangladesh, is reported to have the highest concentration of fine particulate matter (PM_{2.5}) in the world, and the source and environmental dynamics of PM_{2.5} are still unknown. In this study, we collected PM_{2.5} samples in Dhaka, Bangladesh during COVID-19 and analyzed the nitrogen stable isotope ratios of ammonium ion ($\delta^{15}\text{N-NH}_4^+$) in PM_{2.5}. Moreover, the source analysis of NH_4^+ was also conducted using $\delta^{15}\text{N}$ source data and the isotope mixing model.

The results for PM_{2.5} and NH_4^+ concentrations during COVID-19 were $120.0 \pm 90.8 \mu\text{g}/\text{m}^3$ and $3.7 \pm 4.7 \mu\text{g}/\text{m}^3$, respectively. As compared with the pre-COVID-19, there was a slight increase for both compounds. The $\delta^{15}\text{N-NH}_4^+$ was $15.7 \pm 5.2\text{‰}$, which was about 5‰ lighter than the data obtained before COVID-19. NH_3 gas was estimated from the NH_4^+ concentration results and ISSOROPIA II as thermodynamic model. The source contribution rates of livestock, fertilizer, ammonia slip, and fossil combustion were calculated using the isotope mixing model. The agricultural source contribution (livestock and fertilizer) was 70% and increased by about 20% compared to before COVID-19. It was predicted that non-agricultural activities such as factories and automobiles were suppressed, resulting in a change in the contribution rate.

Determination of carbon stable isotope ratios of free amino acids in commercial soy sauce samples

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In the 2000s, liquid chromatography combined with isotope ratio mass spectrometry (LC/IRMS) has been developed. In recently, LC/IRMS has been used to authenticate and trace products for food. In 2006, the carbon stable carbon isotope ratio ($\delta^{13}\text{C}$) of each amino acids using LC/IRMS was developed and analyzed amino acids in biological samples such as human bones and plants. Soy sauce is a condiment developed in China and is a highly nutritious functional food composed mainly of amino acids, sugars, organic acids. Some types of soy sauces have the additives such as amino acid seasoning added during the manufacturing process, while others do not. Distinguishing between soy sauce with and without the additives is difficult and methods are needed to distinguish between pure soy sauce and soy sauce with the additives.

In this study, the method was developed to measure the $\delta^{13}\text{C}$ value of amino acids in soy sauce. In addition, the $\delta^{13}\text{C}$ values of amino acids in 21 soy sauce samples and 3 acid-hydrolysed plant protein seasoning solution samples as the additives were measured to distinguish between pure soy sauce and soy sauce with the additives. As the results, for glutamic acid, the average $\delta^{13}\text{C}$ values of pure soy sauces and soy sauces with additives was $-25.96 \pm 2.67\text{‰}$ and $-19.07 \pm 4.31\text{‰}$, respectively. Soy sauce with the additives was 6.90‰ heavier than pure soy sauce. Moreover, for glutamic acid, acid-hydrolysed plant protein seasoning liquid with a raw material of maize, soybean, and wheat were $-12.74 \pm 0.53\text{‰}$, $-24.20 \pm 0.15\text{‰}$, and $-27.91 \pm 0.06\text{‰}$, respectively. Therefore, soy sauce with the additives could be contained the acid-hydrolysed plant protein seasoning solution of maize such as C4 plant origin. This method is considered to be the effective and valuable method to detect the addition of amino acid seasonings in soy sauce.

POSTERS

Liquid chromatography coupled to IRMS for use with organic mobile phases*Herbert Tobias, Andrew Jones, Tommy Saunders, Thomas Brenna**University of Texas, 1400 Barbara Jordan Blvd. Dell Pediatric Research Institute, Austin, TX 78723, USA*

In this work, liquid mobile phases (such as 80% water/20% acetonitrile) used in "LC" separations was coupled with a novel rotating disc device (called "Solvere") that employs laser combustion of organics and here, enables measurement of AICAR (5-aminoimidazole-4-carboxamide-1- β -D-ribofuranoside). AICAR is a substance produced naturally by the body that stimulates AMP activated protein kinase (AMPK) of interest in medicine and anti-doping investigations. It is activated during exercise and readily abused in sport. This technology will ultimately lead to ^{13}C stable isotopic composition tracing measurements. Currently, the technology successfully converts the AICAR molecules to CO_2 (the form in which ^{13}C stable isotopic measurements are traditionally made). These measurements have been made with $\sim 100\%$ conversion to CO_2 using "Zero Air", 5% O_2 in Helium, 1% O_2 in Helium, and UHP Helium gas, respectively. Further work will involve evaluating measurement and integrity of ^{13}C isotopic values for AICAR, and potentially other relevant molecules, measured for various forensic sourcing applications.

**FIRMS**Forensic Isotope Ratio
Mass Spectrometry