



Yet again it's been a long wait for a new FIRMS newsletter, however from now on we hope to circulate these on a more regular basis with at least one issue per year. This issue shows that while we've been quiet in terms of communication, there has been much work going on behind the scenes. The highlights of this newsletter are the publication of the FIRMS Good Practice Guide for IRMS; implementation and results from a new proficiency testing scheme run as a collaboration between FIRMS and LGC PT and of course, the 5th FIRMS Network Conference held in September last year.

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Steering Group News

Since the last newsletter, Ian Bull (University of Bristol) has resigned as Membership Secretary to be replaced by Phil Dunn (LGC). Russell Frew (IAEA) has had to stand down due to work commitments and Claire Lock (DSTL) has also resigned her position on the SG. Kylie Jones has replaced Sarah Benson as the AFP representative and we also welcome Lesley Chesson of IsoForensics and Wee Chaun Yeo from the Health Sciences Authority, Singapore following their institutions' approval for FIRMS membership. Tim Knowles and Phil Dunn have taken over the collating/editing of the newsletter (*ed: We welcome any suggestions over content, particularly when accompanied by text!*).

The FIRMS Steering group currently consists of: Jim Carter (Chair and Director, Queensland

Health Forensic and Scientific Service); Wolfram Meier-Augenstein (Director, James Hutton Institute); Sean Doyle (Quality Manager, Director, Secretary, Linked Forensic Consultants Ltd); Phil Dunn (Membership Secretary, Newsletter Ed, LGC); Tim Knowles (Newsletter Ed, Mass Spec Analytical); Lesley Chesson (IsoForensics); Max Coleman (NASA Jet Propulsion Laboratory); Jurian Hoogewerff (University of Otago); Kylie Jones (Australian Federal Police); Niamh Nic Dæid (University of Strathclyde); Gerard van der Peijl (Netherlands Forensic Institute); Helen Salorous (National Measurement Institute, Australia); Sabine Schneiders (Bundeskriminalamt); Libby Stern (FBI); David Widory (University of Quebec in Montreal) and Wee Chaun Yeo (Health Sciences Authority, Singapore).



Approved Practitioners Scheme



The scheme for approving forensic isotope ratio practitioners run by FIRMS has now been up and running for some time. We are pleased to report that Lesley Chesson, Jim Ehleringer and Michael Lott of IsoForensics Inc. of Salt Lake City, Utah have been recently added to the list of FIRMS approved practitioners for a variety of different isotopic analyses and sample materials. The approval period is four years and FIRMS maintains a list of approved lead practitioners for consideration by law enforcement agents, lawyers and other potential customers which can be found on [the FIRMS website](#). FIRMS is the only regulatory body in the world assuring the competence

of forensic practitioners in compliance with an international standard; ISO9001:2008. FIRMS approval provides an assurance of evidentiary reliability and involves assessment against a set of criteria drawn up by the Steering Group. The procedure followed and the criteria applied are set out in The FIRMS Network approval procedure and regulatory framework document which is available on the [the FIRMS website](#). The subject of approval is a lead practitioner (a named individual) working in a specified area of isotope forensic practice, in a specified organisation, and using specified methods and procedures. Individuals working in the field of isotope forensics who are interested in seeking approval should [contact FIRMS](#) via the usual e-mail address. Candidates for approval will be sent further details together with an initial assessment questionnaire.

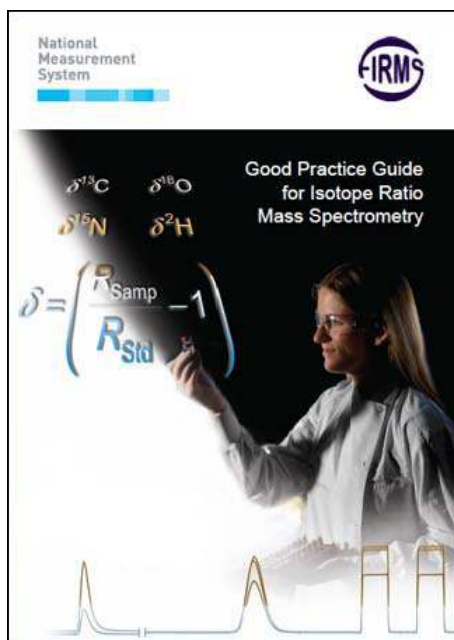
Good Practice Guide

The FIRMS Good Practice Guide for IRMS was completed and published (ISBN 978-0-948926-31-0) in 2011 in collaboration with LGC, the UK's designated National Measurement Institute for chemical and bioanalytical measurements, and is available for download **for free** from [the FIRMS website](#) as well as the National Measurement System (NMS) [ChemBio website](#). This guide was not specifically aimed at forensic applications, but due to the importance of traceability of measurements in forensic applications, the FIRMS Network had a leading role.

Among the topics covered in the guide are: descriptions of common instrument configurations; instrument tests and quality con-

trol & assurance; calibration and evaluation of data; brief descriptions of some applications; as well as interpretation of data. The guide is not aimed at providing methodologies for all preparative methods employed in making light stable isotope ratio measurements. There are several other resources for that purpose, in particular the excellent books edited by Pier de Groot. Instead, the guide is designed to be a first step for aiding acquisition of high quality isotope ratio measurements using some of the most commonly used methods.

Feedback regarding the guide should be passed on to the FIRMS Steering Group via the special BestPractice@forensic-isotopes.org e-mail address.



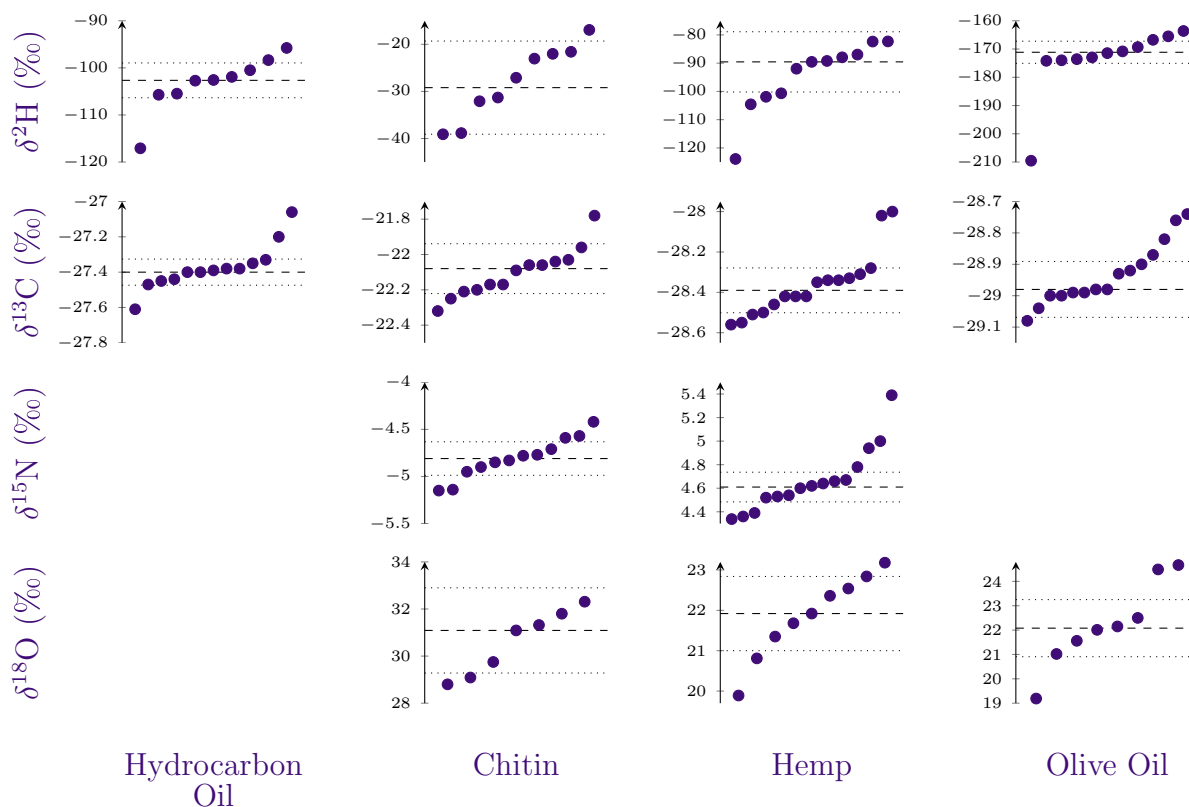


Proficiency Testing Scheme

Between 2002 and 2011, FIRMS ran six inter-laboratory comparison (ILC) exercises in-house, distributing a total of 15 different materials for isotopic analysis. The results from these can be found on the [the FIRMS website](#) and are discussed in two papers by Jim Carter and various colleagues: Carter, JF; Hill, JC; Doyle, S and Lock, C, (2009) *Results of four inter-laboratory comparisons provided by the Forensic Isotope Ratio Mass Spectrometry (FIRMS) network*. Science and Justice, 49:127-137 and Carter, JF and Fry, B (2013) *Ensuring the reliability of stable isotope ratio data beyond the principle of identical treatment*, Analytical and Bioanalytical Chemistry, 405, 2799-2814.

There was a desire to have future FIRMS ILCs accredited to ISO 17043 and be able to distribute the materials resulting from these to the isotope

community as reference materials. To that end a partnership with **LGC Standards**, and in particular their **PT branch**, was negotiated whereby the FIRMS SG would have input into material selection and methods of data analysis, however LGC would take over the administration of the proficiency testing (PT) scheme (distributing samples, collating results, data analysis, report drafting etc). LGC Standards is a major international provider of proficiency testing schemes which is accredited by United Kingdom Accreditation Service (UKAS) and runs 40 PT schemes involving the distribution of more than 250,000 samples every year. These schemes cover a diverse range of sectors including food, environmental, beverage, clinical and forensic. LGC PT also have a prominent role in the development of policy and guidance for proficiency testing.



Results from FIRMS PT scheme Rounds 203 (Hydrocarbon Oil & Chitin) and 209 (Hemp & Olive Oil). The lines represent the median of the results (dashed) plus or minus the robust standard deviation (dotted) following the exclusion of outliers.



There have been two rounds of this PT scheme to date (May and October 2013) with two materials distributed per round. These materials have been bottled and tested for homogeneity prior to distribution. The results, in the form of main reports, from these two rounds can be found on [the FIRMS website](#) but summary figures can be found in this newsletter. While the carbon and nitrogen results are encouraging, those for hydrogen show considerable spread. This is may in part be due to some laboratories correcting for exchangeable deuterium while others have not.

There were some teething problems in the administration of the first couple of rounds, however LGC PT are keen to improve the scheme for the future and therefore any feedback good or bad should be directed to [LGC PT](#). Alternatively, as a subsection of the FIRMS Steering Group (JC, SD, WMA and PD) are currently part of the PT scheme steering group, suggestions can also be passed on to them. We also welcome suggestions

of materials that could be distributed during future rounds of the PT scheme; these should be passed on to the [FIRMS SG](#).

For future rounds we hope to be able to collect more information regarding methods such as which (certified) reference materials were used for scale calibration, whether any corrections were applied (such as for blank, linearity or memory/carry-over) and if/how a correction for exchangeable deuterium was applied. Furthermore, the standard deviation used to calculate the z-score of participants will be defined rather than obtained from the PT data. The remainder of the materials distributed from the first two rounds will be available to purchase from LGC Standards shortly. Further ahead, LGC PT are looking to expand their accreditation to cover the FIRMS scheme towards the end of 2014 when the results from at least one more round will be available.

News in Brief For or From the FIRMS Community

A new project, funded by the National Science Foundation (NSF) and coordinated by Arndt Schimmelmann (University of Indiana) will begin shortly. This project, entitled “NSF-Funded Development of Organic Stable Isotope Reference Materials for GC-IRMS, LC-IRMS, and EA-IRMS,” will involve a number of international laboratories and aims to characterise several new organic reference materials. These materials will include compounds such as amino acids, long chain fatty acid methyl esters, caffeine, polyethylene powder and a hydrocarbon oil (as successor to NBS-22) to name but a few. Almost all of these will be available at two if not three different levels of isotopic abundance to facilitate scale calibration of organic materials. Once these new materials have been fully characterised, they will be distributed by the IAEA. There will be a presentation covering this research project in more detail at the EGU General Assembly in Vienna (27th April to 2nd May 2014).

The Food Standards Agency (FSA, UK) is about to begin a new phase of their British Beef Origin Project which will involve isotope ratio

analysis (heavy and light elements) of a number of different cattle from Scotland and Northern Ireland. This will expand their existing database of cattle isotope ratios.

Thermo Scientific has announced a new isotope ratio infra-red spectroscopy instrument - the [Delta Ray](#).

During 2013 NIST revised a number of certificates for their light element stable isotope reference materials. These materials included IAEA S1, S2 & S3 (all silver sulphides), IAEA S4 (elemental sulphur), NBS-22 (mineral oil), IAEA CH7 (polyethylene foil), NBS-18 (carbonatite), IAEA N1 & N2, USGS 25 & 26 (all ammonium sulphates), NSVEC (nitrogen), NBS-123 (sphalerite) and NBS-127 (barium sulphate). These revisions included changes to the values and uncertainties reported for some of these materials which will impact calculations relying on these values.

Picarro has announced the new high precision [L2140-i water isotope analyser](#) capable of simultaneous measurement of $\delta^{18}\text{O}$, $\delta^{17}\text{O}$, $\delta^2\text{H}$ and ^{17}O -



excess in liquids and vapour.

The Commission on Isotopic Abundances and Atomic Weights (CIAAW) will publish the first in a new series of IUPAC Technical Reports in the near future. This bi-annual series will be entitled “*Assessment of International Reference Materials for Isotope-Ratio Analysis*” and will include a complete list of recommended values for isotopic reference materials for both heavy and light elements.

Forthcoming conferences of interest to the FIRMS community include the [EGU General Assembly](#) (27th April to 2nd May 2014 in Vienna) and the [Advances in Stable Isotope Techniques](#)

and Applications (ASITA) conference at the UC Davis Stable Isotope Facility in California (15th to 18th June 2014). The ASITA conference is the week following the [Goldschmidt Conference](#) in nearby Sacramento, California (8th to 13th June 2014). The Federation of Analytical Chemistry and Spectroscopy Societies (FACSS) is holding the Great Scientific Exchange Conference ([SCIX2014](#)) in Reno-Tahoe, Nevada between the 28th September and the 3rd October 2014. The 9th [International Conference on Applications of Stable Isotope Techniques to Ecological Studies](#) (3rd to 8th August 2014 at the University of Western Australia) may also be of interest.

5th FIRMS Network Conference

The 5th FIRMS Network Conference was held between 11th and 13th September 2013 at the University of Quebec in Montreal (UQAM) thanks largely to organisational skills of David Widory and his team of volunteers from Le Geotop. The objective of this conference was to inform and educate forensic investigators and end users of the potential of FIRMS technology and to update the science community of recent developments in research and technology. 65 delegates from 13 different countries attended, the majority from Canada and the USA. The titles and authors for all presentations can be found on [the FIRMS Website](#). Several of the authors have kindly agreed to allow their abstracts and in some cases their presentations to be made available via the website as well. In the near future, the journal *Science and Justice* will publish a special issue, edited by Jim Carter and Sean Doyle containing papers presented at the conference. A scientific program including downloads for some of the abstracts and presentations can be found on [the FIRMS website](#). On behalf of the FIRMS Network, we take this opportunity to thank again the various sponsors of the conference: [Thermo Scientific](#), [Elementar](#), [Chaumont Systems Development Inc.](#), [UQAM](#), [Le Geotop](#) and [Piccaro](#).

Keynote Address

Prof. Gabe Bowen from the University of Utah kindly agreed to give the Keynote entitled “A spatial framework for isotope forensics.” This provided an oversight into the application of environmental isotopic distributions and the isoscapes that can be constructed from them to investigate forensic samples. The environmental isotopic distribution is dependent on the source of the element in question coupled to any biogeochemical cycling that occurs. This cycling can result in fractionation which is a predictable process that can be modelled provided that calibration and validation data can be obtained. The environmental isotopic distribution is transferred to a specimen via chemical or ecological processes and this allows the comparison of a forensic sample to a specimen to provide some information regarding origin. Prof Bowen also highlighted the need to provide community databases of isotope distributions in forensically relevant materials, possibly by combining those already assembled by individual laboratories or organisations.



2013 FIRMS Conference Photo

Session 1: Human Provenancing

This session consisted of 7 talks covering a wide range of isotopic analyses, both via IRMS for the light elements and ICP-MS for the heavy elements. This session also included several presentations using isotopic analyses of hair in various forensic contexts which sparked a lively discussion during the roundtable at the end of the session regarding the factors influencing the isotopic composition of hair (growth rate, phase of growth etc.) and therefore the advantages and limitations to these approaches.

Session 2: Geolocation

Another talk on hair isotopic analysis from Jim Ehleringer (Utah) was followed by a suite of presentations from Gilles St Jean (University of Ottawa) regarding the Canadian Geo-Location Project. This had involved extensive sampling of hair, soil, water and pollen, multi isotopic analyses and the development of new software to allow the multitude of data to be collated and queried regarding the origin of unknown samples.

Session 3: Data Handling

A short session with talks from Gerard Van der Peijl (NFI) on duct tape analysis, Ulrich Flenker

(German Sport University) on GC-C-IRMS analysis and the use of Bayesian statistics to aid their interpretation and Jim Ehleringer (standing in for Thure Cerling - to whom the FIRMS Network wish a speedy recovery) about deconvolution of mixed isotope signals. The statistical elements of these presentations were of particular interest in the roundtable discussion.

Session 4: Isotope Discrimination

Again a short session with three talks examining the use of stable isotopes to distinguish between batches and brands of inks (Lesley Chesson, Iso-Forensics), papers (Kyle Jones, AFP) and automotive clear coats (Sabine Schneiders, BKA).

Session 5: Food and Drug Provenancing

A wide range of methods and samples were presented in this session with Jim Carter's (QHFSS) *Alcoscape* based upon isotopic analysis of beer showing particular inventiveness when faced with problems with importing the more commonly used water samples. The other presentations concerned the linking of drugs to their precursor molecules (Michael Collins, NMIA) or metabolites (Alexandre Oullet, INRS Institute) as well as the authentication of vinegar (Frederica Camin,



IASMA) and using blackberry leaves as a proxy for cannabis when creating an isoscape (Shaerii Sarker, University of Otago).

Session 6: Sponsors

There were presentations from the conference sponsors: Thermo Scientific, Picarro, Chau-mont Systems Development Inc. and Elementar (Isoprime) highlighting recent or near-future advances in their instrumentation or software. There was also a brief presentation on the results of the recent FIRMS proficiency testing scheme from Phil Dunn (LGC).

Poster Session

There were 23 posters in total relating to the various sessions and there was plenty of opportunity for discussion of them in the poster session that took place before the conference dinner. Topics covers included isotopic analysis of hair (both heavy and light elements buy ICP-MS and IRMS, respectively); water; coal; drugs; explosives and methodological advances such as resolving N₂ interferences for $\delta^{18}\text{O}$ analysis of N-rich organics by TC/EA and a spreadsheet approach to estimating measurement uncertainty.

The poster session was followed by the conference banquet, also held at UQAM.



2013 FIRMS Conference Sponsors

Recent Publications

Books & Book Chapters

Wolfram Meier-Augenstein was asked to contribute a chapter entitled *Forensic Isotope Analysis* to the 2014 edition of the *McGraw-Hill Yearbook of Science & Technology* (pp 120-124; ISBN 978-0071831062)

Lesley Chesson contributed to the chapter entitled *Stable Isotopes and Human Provenancing* while Helen Kemp and Wolfram Meier-Augenstein contributed to the chapter entitled *Multi-Disciplinary Approach toward the Identification of a Human Skull found 55 km of the Southeast Coast of Ireland* to the 2014 book *Advances in Forensic Human Identification* edited by Xanthé Mallett, Teri Blythe and Rachel Berry (CRC Press, ISBN 978-1439825143).

Jim Carter, Helen Kemp and Wolfram Meier-Augenstein between them contributed four chap-

ters entitled: *Stable Isotope Analysis: General Principles and Limitations*, *Stable Isotope Analysis: Drugs*, *Stable Isotope Analysis: Hair and Nails* and *Stable Isotope Analysis: Bones and Teeth* to the 2013 *Wiley Encyclopædia of Forensic Science* (ISBN 978-0470061589). The chapter on drugs was amongst the top 10 most downloaded articles from the Encyclopædia in 2013.

Journal Special Editions

There have been two journal special editions of interest to the FIRMS community since the publication of the last newsletter: the December 2012 special issue of *Drug Testing and Analysis* (Vol 4, Issue 12) entitled: *Stable isotope ratio analysis in sports anti-doping* edited by Adam Cawley and also the March 2013 edition of *Analytical and Bioanalytical Chemistry* (Vol 405, Issue 9) entitled: *Isotope Ratio Measurements: New*



Developments and Applications edited by Klaus Heumann & Torsten Schmidt. Some of the papers from these special editions are listed within the recent publications below.

Feature Article

The FIRMS Steering Group feel that the recent paper by Gentile *et al.* (2013) merits special attention. In this paper, the authors took the time and trouble to investigate a problem that at least anecdotally had been mentioned at meetings and in private discussions, namely a difference in behaviour of nitrates compared to organic nitrogen in EA-IRMS analyses resulting in tailing N₂ peaks and unexpected $\delta^{15}\text{N}$ values. They performed a series of experiments in which they investigated a number of possible causes and solutions, namely: (i) the addition of graphite and vanadium pentoxide to see if these would enhance the decomposition of the nitrates; however this had little success; (ii) the comparison of the usual chromium oxide/silvered cobaltous/ic oxide com-

bustion reactor with one containing tungsten oxide/reduced copper, both using an concomitant reduction reactor of reduced copper which highlighted that incomplete reduction was the cause of the tailing; and (iii) modification of the method parameters including to Helium flow rates and suppression of the O₂ pulse.

The two key findings of this paper which will be of interest to the FIRMS community are firstly, that nitrates should be analysed without an additional pulse of O₂, resulting in well eluted N₂ peaks which exhibit little or no tailing; and secondly that samples and reference materials should be matched in terms of nitrogen species. So, when running organic materials for $\delta^{15}\text{N}$, organic RMs such as USGS 40 and USGS 41 should be used. Running organic samples for $\delta^{15}\text{N}$ against inorganic $\delta^{15}\text{N}$ RMs (or vice versa) should be avoided whenever and wherever possible. It is therefore necessary to have separate control charts for organic and inorganic nitrogen isotope ratio measurements.

Papers

Disclaimer: This section contains a non-comprehensive list of recent publications that may be of interest to the FIRMS community. Inclusion of an article in this list does not mean that FIRMS approves the content. You are encouraged to consider critically whether (i) the experimental work complies with SI guidelines and the FIRMS Good Practice Guide; and (ii) whether the conclusions drawn are based on sound scientific background information.

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