



**Book of Abstracts  
for the TRACE 5<sup>th</sup> Annual Meeting and Conference**

**„TRACE in practice –  
New methods and systems for confirming the origin of food“**

**1<sup>st</sup> – 3<sup>rd</sup> April 2009  
Freising, Germany**

**Herausgeber:** Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit  
Veterinärstr. 2  
D-85764 Oberschleißheim  
**Telefon:** +49 89 31560-0  
**Telefax:** +49 89 31560-425  
**Internet:** [www.lgl.bayern.de](http://www.lgl.bayern.de)  
**Fotos:** Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit  
**E-Mail:** [poststelle@lgl.bayern.de](mailto:poststelle@lgl.bayern.de)  
**Druck:** Osterchrist Druck und Medien, Nürnberg  
**Stand:** März 2009

© Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit, alle Rechte vorbehalten

Gedruckt auf Papier aus 100 % Altpapier

ISBN 978-3-939652-80-9

Diese Druckschrift wird kostenlos im Rahmen der Öffentlichkeitsarbeit der Bayerischen Staatsregierung herausgegeben. Sie darf weder von den Parteien noch von Wahlwerbern oder Wahlhelfern im Zeitraum von fünf Monaten vor einer Wahl zum Zweck der Wahlwerbung verwendet werden. Dies gilt für Landtags-, Bundestags-, Kommunal- und Europawahlen. Missbräuchlich ist während dieser Zeit insbesondere die Verteilung auf Wahlveranstaltungen, an Informationsständen der Parteien sowie das Einlegen, Aufdrucken und Aufkleben parteipolitischer Informationen oder Werbemittel. Untersagt ist gleichfalls die Weitergabe an Dritte zum Zweck der Wahlwerbung.

Auch ohne zeitlichen Bezug zu einer bevorstehenden Wahl darf die Druckschrift nicht in einer Weise verwendet werden, die als Parteinahme der Staatsregierung zugunsten einzelner politischer Gruppen verstanden werden könnte.

Den Parteien ist es gestattet, die Druckschrift zur Unterrichtung ihrer eigenen Mitglieder zu verwenden.

Bei publizistischer Verwertung – auch von Teilen – wird um Angabe der Quelle und Übersendung eines Belegexemplars erbeten. Das Werk ist urheberrechtlich geschützt. Alle Rechte sind vorbehalten. Die Broschüre wird kostenlos abgegeben, jede entgeltliche Weitergabe ist untersagt.

Diese Broschüre wurde mit großer Sorgfalt zusammengestellt.

Eine Gewähr für die Richtigkeit und Vollständigkeit kann dennoch nicht übernommen werden.

Für die Inhalte fremder Internetangebote sind wir nicht verantwortlich.



BAYERN | DIREKT ist Ihr direkter Draht zur Bayerischen Staatsregierung. Unter Tel. 089 122220 oder per E-Mail unter [direkt@bayern.de](mailto:direkt@bayern.de) erhalten Sie Informationsmaterial und Broschüren, Auskunft zu aktuellen Themen und Internetquellen sowie Hinweise zu Behörden, zuständigen Stellen und Ansprechpartnern bei der Bayerischen Staatsregierung.

## Organized by

Bavarian Health and Food Safety Authority, Oberschleißheim

## **TRACE**

(**TR**acing Food **C**ommodities In **E**urope)  
is a 5 year project funded by European Commission,  
through the Sixth Framework Programme  
under the Food Quality And Safety Priority  
Contract No. 006942

[www.trace.eu.org](http://www.trace.eu.org)

This project is carried out by a consortium  
coordinated by the FERA –  
The Food and Environment Research Agency (UK)  
and includes 54 partners.



Bayerisches Landesamt für  
Gesundheit und Lebensmittelsicherheit



**Disclaimer:** The information reflects the author's views, the European Commission is not liable for any use of the information contained therein.

# TRACE 5<sup>th</sup> ANNUAL Meeting and Conference

Freising, 1<sup>st</sup> – 3<sup>rd</sup> April 2009

## Acknowledgements

### Scientific Committee

Paul Brereton (FERA), Andreas Rossmann (Isolab), Gerard Downey (Teagasc), Hermann Broll (BfR), Jostein Storey (Sintef), Petter Olsen (Nofima Market), Bernard Vandeginste (VICIM), Eleni Alevritou (EKPIZO), Jana Hajslova and Monika Tomaniova (ICT) Philippe Vermeulen and Vincent Baeten (CRA-W), Jurian Hoogewerff (UEA) and Grishja van der Veer (Geochem).

### Organising Team

Katharina Axtner, Günter Barth, Ernst Brunner, Werner Fritsch, Katrin Grimmer, Renate Habernegg, Elisabeth Haggerty, Kirk Haggerty, Stefan Janssen, Marianne Kalteis, Thomas Kugler, Andrea Lorenz, Petra Preikschat, Josefine Röming, Antje Schellenberg, Claus Schlicht, Claudia Schuller, Michael Sonnentag, Rosemarie Steidler and Margot Wittmann (LGL).

### Organising Committee

Paul Brereton, Caroline Garrod (FERA), Stefan Janssen, Antje Schellenberg (LGL) and Monika Tomaniova (ICT).

### Scientific Panel (selection of presentations)

Paul Brereton (FERA) and Mark Woolfe (Food Standards Agency).

### Special thanks

To all the other people (too numerous to name) who have helped make this conference and all its 'attachments' possible.

## Index of Contents

|   |           |
|---|-----------|
| <b>Greetings.....</b>   | <b>5</b>  |
| Bavarian State Minister of the Environment and Public Health.....   | 5         |
| Co-ordinator TRACE Integrated project.....  | 6         |
| President of the Bavarian Health and Food Safety Authority.....   | 7         |
| <b>Program.....</b>   | <b>8</b>  |
| Tagungszentrum Freising.....  | 8         |
| Day 1 - Wednesday 01 <sup>st</sup> April.....   | 9         |
| Day 2 - Thursday 2 <sup>nd</sup> April - Part I.....  | 10        |
| Day 2 - Thursday, 2 <sup>nd</sup> April - Part II.....  | 11        |
| Day 3 - Friday, 3 <sup>rd</sup> April - Part I.....   | 12        |
| Day 3 - Friday, 3 <sup>rd</sup> April - Part II.....  | 13        |
| Roomplan.....   | 14        |
| <b>Lectures.....</b>  | <b>15</b> |
| <b>IRMS-Workshop.....</b>   | <b>16</b> |
| The potential of the <sup>18</sup> O-determination on organically bound oxygen for origin and authenticity checks.....  | 16        |
| H C N O S stable isotope measurements during the TRACE project – how do we compare?.....  | 17        |
| <sup>18</sup> O standards for high-temperature conversion (HTC); an inter-laboratory calibration effort with different techniques.....                                    | 18        |
| The online <sup>18</sup> O/ <sup>16</sup> O analysis: development and possible applications.....  | 19        |
| Recent developments of IRMS systems for applications in food analysis.....  | 20        |
| Advances in compound specific isotope analysis.....   | 21        |
| <b>Main Session.....</b>  | <b>22</b> |
| Improving decision making capabilities for food defense and agricultural protection.....  | 22        |
| Food authenticity – legislation and enforcement.....  | 23        |
| Assuring brand integrity.....   | 24        |
| Assessment of traceability in the mineral water, honey and chicken sectors: lessons learnt.....   | 25        |
| Food traceability cost benefit analysis: a model for qualitative assessment.....  | 25        |
| Tracing your food – an interactive demonstration.....   | 26        |
| The development of methods to verify the geographical origin of chicken meat.....   | 27        |
| Food maps how do they work: an interactive demonstration.....   | 28        |
| Tracing provenance: differentiating European from Argentinean foods.....  | 29        |
| Confirmation of beer authenticity by spectroscopic fingerprint techniques.....  | 30        |
| Molecular biological methods for authenticating food.....   | 31        |
| Molecular based tools for traceability of beef.....   | 32        |
| <b>Parallel Session A.....</b>  | <b>33</b> |
| Boron isotope compositions of crop plants: a new tracer for the origin and authenticity of food.....  | 33        |
| Soil composition as potential tracer for food authentication: can it be derived from local geology?.....  | 34        |
| Meat authentication – what did the animal eat?.....   | 35        |
| High resolution time of flight mass spectrometry (HR TOF-MS) employing direct analysis in real time (DART) ion source: a challenging technique for food traceability..... | 36        |
| Validated methods for plant and animal species differentiation.....   | 37        |
| Development of an array-based traceability tool for (cereal) specialty products.....  | 38        |
| Determination of fish origin by using 16S rDNA fingerprinting of bacterial communities by PCR-DGGE: an application on pangasius fish from Viet Nam.....                   | 39        |
| Male wagyu lineage origin in crossbred steers through Y chromosome DNA.....   | 40        |
| <b>Parallel Session B.....</b>  | <b>41</b> |
| Traceability of food in practice - requirements for official control and food business operators.....   | 41        |
| Traceability in official feed control.....  | 42        |

# TRACE 5<sup>th</sup> ANNUAL Meeting and Conference

Freising, 1<sup>st</sup> – 3<sup>rd</sup> April 2009

|   |           |
|---|-----------|
| Traceability in the bulk grain supply chain.....  | 43        |
| Traceability and the consumer .....   | 44        |
| Milestones towards global traceability .....  | 45        |
| Traceability of food and consequences for isotope signatures in human tissues.....  | 46        |
| Simulated recalls of meat products, fruit and vegetables originating in the European Economic Community – preliminary results .....   | 47        |
| Traceability in cattle in Germany .....   | 48        |
| <b>Internal Posters .....</b>   | <b>49</b> |
| Soil-grape traceability using a regression model based on trace elements .....  | 50        |
| Traceability of tomato and derivatives along the production chain.....  | 51        |
| Authentication of the trappist beers using advanced mass spectrometric techniques .....   | 52        |
| Isotopic and mineral data for tracing the origin of European olive oils .....   | 53        |
| FT-Raman and chemometrics for the authentication of trappist beers.....   | 54        |
| Trace elements as markers for geographical regions: examples for anthropogenic influence and geological background .....  | 55        |
| Traceability of food ingredients: molecular methods for GMO detection.....  | 56        |
| Multi-element (h C N S) stable isotope ratio characteristics of honey from different European regions.....  | 57        |
| Multi-element (h C N S) stable isotope ratio characteristics of whitefish ( <i>coregonus species</i> ) from different lakes in Bavaria.....   | 58        |
| Overview of TRACE project TECHNOLOGY TRANSFER GROUP activities: 2008.....   | 59        |
| <b>External Posters .....</b>   | <b>60</b> |
| Identification of the geographic origin of pumpkin seed oil.....  | 61        |
| Principles for tracking the origin of organic beef using stable isotopes of life .....  | 62        |
| A new IRMS perspective on <sup>13</sup> C/ <sup>12</sup> C determination of carbon dioxide to authenticate sparkling drinks .....   | 63        |
| Authenticity of vinegar controlled by stable isotope and 14-carbon data .....   | 64        |
| Guarantee of quality and traceability .....   | 65        |
| Ovine intra- and inter-muscular variation in turnover as recorded by carbon stable isotopes .....   | 66        |
| Stable isotope analysis of light and heavy elements for differentiation between German and Austrian bovine samples .....  | 67        |
| Identification of ostrich meat and evaluation of ostrich meat grades by high performance liquid chromatography with electrochemical detection using copper nanoparticle-plated electrode..... | 68        |
| Determination of delta18O in water of Czech wine products using pyrolysis-continuous flow IRMS technique.....   | 69        |
| Authentication of the commercial denomination <i>light tuna</i> by real time PCR.....   | 70        |
| Effect of light in extra-virgin olive oil odour profile, volatile compounds and vitamins .....  | 71        |
| Analysis of volatile compounds in extra-virgin olive oil after light exposure by solid phase microextraction and gas chromatography .....   | 72        |
| Evaluation of an ultrasound-assisted digestion method for determination of arsenic and lead in edible citric acid samples by ETAAS .....  | 73        |
| Application of PCR-DGGE in determining geographical origin of fruits: cases studies of physalis and shea tree fruits.....   | 74        |
| Isotopic characterization of authentic Czech honey: deuterium and carbon isotopes.....  | 75        |
| Internal traceability system database modeling for a grain elevator .....   | 76        |
| Carbon isotope ratio analysis of authentic and commercial essential oils of lavandin (lavandin hybrida).....  | 77        |
| Iris recognition middleware and key technology in food supply chain: taking cows as an example.....   | 78        |



## Greetings

Ladies and Gentlemen,  
Dear Participants,

Welcome to the 5th Annual Conference of "TRACE in practice", in the beautiful cathedral and university town of Freising. I am delighted that participants from all over the world have come to Bavaria to attend this event.

Something we all want to know is what we eat and, most importantly, where the food on our table comes from. Not least for reasons of safety. We need to be able to trace the production chain from fork to farm if we want to identify problems quickly and withdraw goods that are already on the market. The traceability of food products and animal feed is therefore an essential pillar in consumer protection.



Food diversity, the globalised market and more and more advanced analytical tools call for intensive scientific work in this field. In order to meet these challenges the EU launched the TRACE project in January 2005 for a period of 5 years. 52 organisations from all over Europe, South America and China are cooperating in this project.

With the analytical methods available so far it has not been possible to check the details given on ecological production methods or information on the natural properties of food. In the TRACE project state-of-the-art methods are now being applied and systems developed that guarantee full traceability and designation of origin of food products and animal feed. Today, for example, natural indicators such as isotopes, trace elements and genetic markers are defined to provide verifiable data on the geographical origin and authenticity of the products. Particular attention is awarded to products with protected designation of origin, as is the case in Bavaria, for instance, with "Allgäuer Emmentaler" cheese or "Münchener Bier", and also for imported meats (Argentine beef, chicken meat from China) and other global food trade products.

In addition, a study is being carried out on the consumer's attitude and expectations concerning the ability to trace the origin of food and food fraud. In this connection, standardised formulas are also being developed and tested for registration, coding and information flow. This is particularly useful when a food product is made from many different initial components.

As TRACE project partner, the Bavarian Health and Food Safety Agency (LGL) is involved in the "stable isotopes" sector. The agency has been performing tests on authenticity and designation of origin of food since 2001. Within the scope of the TRACE project, the stable isotopes of honey, crops, olive oil, chicken meat, lamb and beef from different European regions have been identified and analysed with regard to their origin, in cooperation with the project partners.

I wish the TRACE conference much success and hope that every day we will get that bit closer to the goal of food safety.

A handwritten signature in black ink, appearing to read 'M. Söder'.

Dr. Markus Söder MdL  
**Bavarian State Minister of the Environment and Public Health**

## Greetings

It is my pleasure to welcome you to the 5<sup>th</sup> TRACE conference “Trace in practice: New methods and systems for confirming the origin of food”.

Recent incidents reported in the press and media have emphasised the continued need for improved traceability methods and systems in the food area. The global nature of the food and feed supply is such that food safety incidents that arise in far away continents now have major impacts in our own markets. Melamine contamination within the Chinese dairy industry hospitalised over 50,000 children, led to 6 deaths and set major challenges in terms of traceability for the global food industry. In December 2008 the Food Safety Authority of Ireland initiated one of the largest ever recalls of Irish pork due to suspected dioxin contamination, with over 20 countries affected. In the US, one of the largest recalls in US history is currently being undertaken after salmonella was found in peanut products. Ninety people are thought to have been hospitalised and 43 States affected. The impact on the peanut industry has been devastating with many companies going out of business.



2008 saw a plethora of incidents where food products have been deliberately mislabelled in order to defraud the consumer and the honest producer. High value products continue to be a target for the fraudsters and there have been several incidents in the wine, spirits, meat, honey and premium cheese sectors in the recent year. The discovery of counterfeit vodka in the UK market is a good example of how food fraud can also have major consequences for food safety; the illicit product was found to contain unacceptably high levels of methanol.

Food is increasingly marketed to a more discerning European consumer in terms of perceived added value, with the result that foods with known geographical or processing origin can command higher prices. It is clear that improved systems and methods of analyses are required that can confirm the identity of the product and trace back to its origin. This conference will be showcasing some of the latest analytical methods and systems that are in development to verify food and feed products. In addition, some of the experiences resulting from the implementation of new traceability systems and processes in food businesses will be presented. Although much of the content of the conference is derived from the scientists within the TRACE project, I am pleased that such a large number of international visitors are attending the conference, many of whom are presenting complimentary research in the area of food and feed traceability.

I hope you enjoy the conference,

Paul Brereton  
**Co-ordinator TRACE Integrated project**  
Food and Environment Research Agency, UK



## Greetings

Dear participants, dear readers,

Roughly five years ago experts from numerous countries met at the newly founded Bavarian Health and Food Safety Authority (the LGL) to set out the basis and goals of a project designed to ensure food traceability and thus to strengthen consumer protection.

That kick-off meeting led to the creation of the TRACE project, which is now holding its fifth annual meeting, on the topic of "TRACE in Practice", in the Bavarian city of Freising. Experts from all over the world will be presenting the project's results and discussing how they can be used in day to day practice.

The LGL is the Bavarian authority responsible for protecting consumers' health. Our main contributions to the project have been expertise in the field of stable isotope analysis of light elements and our analytical capacity. We have been using stable isotope analysis since 1999 and were the first German food inspection body to do so. In the LGL's testing of 80,000 foodstuffs per year, this state-of-the-art method is just as much a part of future-oriented analysis as residues tests or detection of genetically modified organisms. Modern analysis methods provide the scientific basis on which to identify health risks and to determine products' quality. Forward-looking identification and assessment of health risks and protection of the public against being misled or deceived are key cornerstones of a progressive health and consumer protection policy.

Research, focused on practical application, in collaboration with numerous academic institutions and inspection bodies therefore plays a key role in our work. Food safety will benefit greatly from the fact that TRACE has become established as a new international network in which experts share knowledge from the realms of research and practice.

I hope your discussions of the project's results will be informative, your stay pleasant and that, despite the full programme, you will have a little time to take in the attractions of Munich and its surroundings.



Dr. Andreas Zapf

**President of the Bavarian Health and Food Safety Authority**



# TRACE 5<sup>th</sup> ANNUAL Meeting and Conference

Freising, 1<sup>st</sup> – 3<sup>rd</sup> April 2009

## Program

Tagungszentrum Freising



Day 1 - Wednesday 01<sup>st</sup> April

|               |   |
|---------------|---|
| 09.00 – 10.00 | Registration  |
| 10.00 – 17.00 | <b>Closed Meetings (e.g. Workpackage Meetings)</b>  |
| 13.00 – 14.00 | Lunch (Great Dinning Hall)  |
|               | <b>Isotope ratio mass spectrometry (IRMS)-workshop</b>  |
|               | <b>-Analytical and technical aspects of the <math>\delta^{18}\text{O}</math>-determination of organic substances-</b> (Assembly Hall/ Aula)   |
| Chair:        | Dr. Claus Schlicht (LGL, Oberschleißheim, Germany)  |
| 14.00 – 14.30 | The potential of the $^{18}\text{O}$ -determination on organically bound oxygen for origin and authenticity checks<br><i>Prof. Dr. Hans-Ludwig Schmidt (Isolab GmbH and Technical University Munich, Germany)</i>   |
| 14.30 – 14.55 | H C N O S stable isotope measurements during the TRACE project - how do we compare?<br><i>Dr. Simon Kelly (University of East Anglia, Norwich, United Kingdom)</i>  |
| 14.55 – 15.20 | $^{18}\text{O}$ standards for high-temperature conversion (HTC); an inter-laboratory calibration effort with different techniques<br><i>Dr. Willi A. Brand (MPI-BGC Jena, Germany in collaboration with USGS, IAEA, ETH, ANU, UFZ and CIO laboratories)</i> |
| 15.20 – 15.50 | Coffee break  |
| Chair:        | Dr. Andreas Rossmann (Isolab GmbH, Schweitenkirchen, Germany)   |
| 15.50 – 16.15 | The online $^{18}\text{O}/^{16}\text{O}$ analysis: development and possible applications<br><i>Dr. Roland A. Werner (Institut für Pflanzenwissenschaften, ETH Zürich, Switzerland)</i>  |
| 16.15 – 16.40 | Recent developments of IRMS systems for applications in food analysis<br><i>Dr. Lutz Lange (Elementar Analysensysteme GmbH, Hanau, Germany)</i>   |
| 16.40 – 17.05 | Advances in compound specific isotope analysis<br><i>Dr. Dirk Juchelka (Thermo Fisher Scientific (Bremen) GmbH, Germany)</i>  |
| 17.15 – 18.45 | <b>Closed Consortium Meeting</b> (Assembly Hall/ Aula)  |
|               | Close   |
| 19.30 – 24.00 | <b>Get together</b> ('Korbiniansklause', Kardinal-Döpfner-Haus)   |

# TRACE 5<sup>th</sup> ANNUAL Meeting and Conference

Freising, 1<sup>st</sup> – 3<sup>rd</sup> April 2009

## Day 2 - Thursday 2<sup>nd</sup> April - Part I

|               |   |
|---------------|---|
| 08.00 – 09.00 | Registration  |
| 09.00 – 09.15 | <b>Welcome and opening of the conference</b> (Assembly Hall/ Aula)  |
|               | <b>Plenary Session – Assuring food Integrity</b> (Assembly Hall/ Aula)  |
| Chair:        | Dr. Jurian Hoogewerff (UEA, UK)   |
| 09.15 – 09.45 | Improving decision making capabilities for food defence and agricultural protection<br><i>Prof. Richard Linton (Purdue University, US)</i>            |
| 09.45 – 10.15 | Food authenticity: legislation and enforcement<br><i>Dr. Mark Woolfe (Food Standards Agency, UK)</i>  |
| 10.15 – 10.45 | Assuring brand integrity<br><i>Dr. Ross Aylott (Diageo, UK)</i>   |
| 10.45 – 11.10 | Coffee break and poster session (Korbinian's Hall)  |
|               | <b>TRACE in practice – traceability systems</b> (Assembly Hall/ Aula)   |
| Chair:        | Dr. Marcel Mengelers (VWA, NL)  |
| 11.10 – 11.35 | Assessment of traceability in the mineral water, honey and chicken sectors: lessons learnt<br><i>Petter Olsen (Nofima, NO)</i>                        |
| 11.35 – 12.00 | Food traceability cost benefit analysis: a model for qualitative assessment<br><i>Dr. George Chryssochoides (AUA, GR)</i>                             |
| 12.00 – 12.25 | Tracing your food - interactive demonstration<br><i>Dr. Heiner Lehr (FoodReg, ES)</i>   |
| 12.25 - 12.30 | Questions   |
| 12.30 - 14.00 | Lunch (Great & Little Dinning Hall)   |
|               | <b>New approaches to assessing geographical origin</b> (Assembly Hall/ Aula)  |
| Chair:        | Dr. Helen Darling (Oritain, NZ)   |
| 14.00 – 14.25 | The development of methods to verify the geographical origin of chicken meat<br><i>Dr. Simon Kelly (IFR, UK)</i>                                      |
| 14.25 – 14.50 | Food maps how do they work: an interactive demonstration<br><i>Stefan Torfi Höskuldsson (Maritech, IS) and Dr. Grishja van der Veer (Geochem, NL)</i> |
| 14.50 – 15.15 | Tracing provenance: differentiating european from argentinean foods.<br><i>Dr. Daniel Wunderlin (UNC, AR) and Dr. Hector Oстера (CONICET, AR)</i>     |
| 15.15 – 15.45 | Coffee break and poster session (Korbinian's Hall)  |

## Day 2 - Thursday, 2<sup>nd</sup> April - Part II

|               |  |
|---------------|--|
| 15.15 – 15.45 | Coffee break and poster session (Korbinian's Hall)   |
|               | <b>New approaches to food verification</b> (Assembly Hall/ Aula)   |
| Chair:        | Vincent Baeten (CRA-W, BE)   |
| 15.45 – 16.10 | Confirmation of beer authenticity by spectroscopic fingerprint techniques<br><i>Gerry Downey (Teagasc, IE)</i> |
| 16.10 – 16.35 | Molecular biological methods for authenticating food<br><i>Hermann Broll (BfR, DE)</i>                         |
| 16.35 – 17.00 | Molecular based tools for traceability of beef<br><i>Riccardo Negrini (Unicatt, IT)</i>                        |
| 14.00 – 18.00 | <b>Closed Advisory Board Meeting</b> (Kapitel Hall)  |
| Close         |  |
| 19.30 – 24.00 | <b>Conference Dinner</b> ('Bräustüberl', Brewery Weihenstephan)  |

## Day 3 - Friday, 3<sup>rd</sup> April - Part I

### **Analytical techniques for authenticating food** (PARALLEL SESSION A)

#### **Part I - Chemical techniques** (Assembly Hall/ Aula)

Chair: Dr. John Dennis (FERA, UK)

09.00 – 09.25 Boron isotope compositions of crop plants: a new tracer for the origin and authenticity of food

*Martin Rosner (Federal Institute for Material Research and Testing, DE)*

09.25 – 09.50 Soil composition as potential tracer for food authentication: can it be derived from local geology?

*Bernhard Wimmer (ARCS, AU)*

09.50 – 10.15 Meat authentication – what did the animal eat?

*Frank J. Monahan (University College Dublin, IE)*

10.15 – 10.40 High resolution time of flight mass spectrometry (HR TOF-MS) employing direct analysis in real time (DART) ion source: a challenging technique for food traceability

*Jana Hajšlová (ICT Prague, CZ)*

10.40 – 11.10 Coffee break and poster session (Korbinian's Hall)

#### **Part II - Molecular biological techniques** (Assembly Hall/ Aula)

Chair: Hermann Broll (BfR, DE)

11.10 – 11.35 Validated methods for plant and animal species differentiation

*Dr. Andreas Pardigol (Eurofins, Nantes, FR)*

11.35 – 12.00 Development of an array-based traceability tool for (cereal) specialty products

*Dr. Theo Prins (RIKILT, NL)*

12.00 – 12.25 Determination of fish origin by using 16S rDNA fingerprinting of bacterial communities by PCR-DGGE: an application on pangasius fish from Viet Nam

*Dr. Didier Montet (Cirad, FR)*

12.25 - 12.50 Male wagyu lineage origin in crossbred steers through Y chromosome DNA

*Andrés Rogberg-Muñoz (UNLP-CONICET, AR)*



**Day 3 - Friday, 3<sup>rd</sup> April - Part II****Traceability systems now and in future (PARALLEL SESSION B)****Part I Actual systems (Red Hall/ Roter Saal)**

- Chair: Dr. Ulrich Busch (LGL, DE)
- 09.00 – 09.25 Traceability of food in practice - requirements for official control and food business operators  
*Dr. Birgit Knispel (LGL, DE)*
- 09.25 – 09.50 Traceability in official feed control  
*Dr. Claudia Thielen (District Government of Upper Bavaria, DE)*
- 09.50 – 10.15 Traceability in the bulk grain supply chain  
*Dr. Charles Hurburgh (Iowa State University, US)*
- 10.15 – 10.40 Traceability and the consumer  
*Dr. George Chryssochoidis (AUA, GR)*

10.40 – 11.10 Coffee break and poster session (Korbinian's Hall)

**Part II Systems designed for the potential use in the future (Red Hall/ Roter Saal)**

- Chair: Dr. Günter Barth (LGL, DE)
- 11.10 – 11.35 Milestones towards global traceability  
*Dr. Heiner Lehr (FoodReg, ES)*
- 11.35 – 12.00 Traceability of food and consequences for isotope signatures in human tissues  
*Dr. Christine Lehn (Munich University's Institute for Forensic Medicine, DE)*
- 12.00 – 12.25 Simulated recalls of meat products, fruit and vegetables originating in the European Economic Community – preliminary results  
*Kathryn A-M. Donnelly and Kine Mari Karlsen (Nofima, NO)*
- 12.25 - 12.50 Traceability in cattle in Germany  
*Dr. Richard Carmanns (Bavarian State Ministry of Nutrition, Agriculture and Forestry, DE)*
- 12.50 – 13.00 **Leave-taking with poster award ceremony** (Assembly Hall/ Aula)

13.00 – 14.00 Lunch (Great Dining Hall)

Close

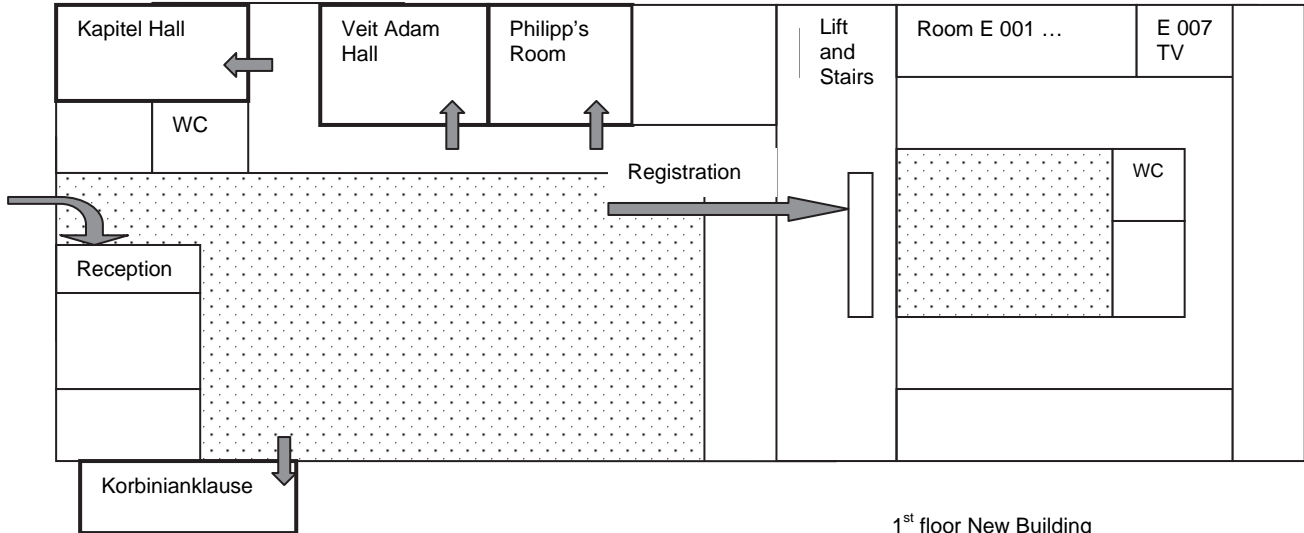
14.00 – 18.00 **Closed Scientific Committee Meeting** (Kapitel Hall)

# TRACE 5<sup>th</sup> ANNUAL Meeting and Conference

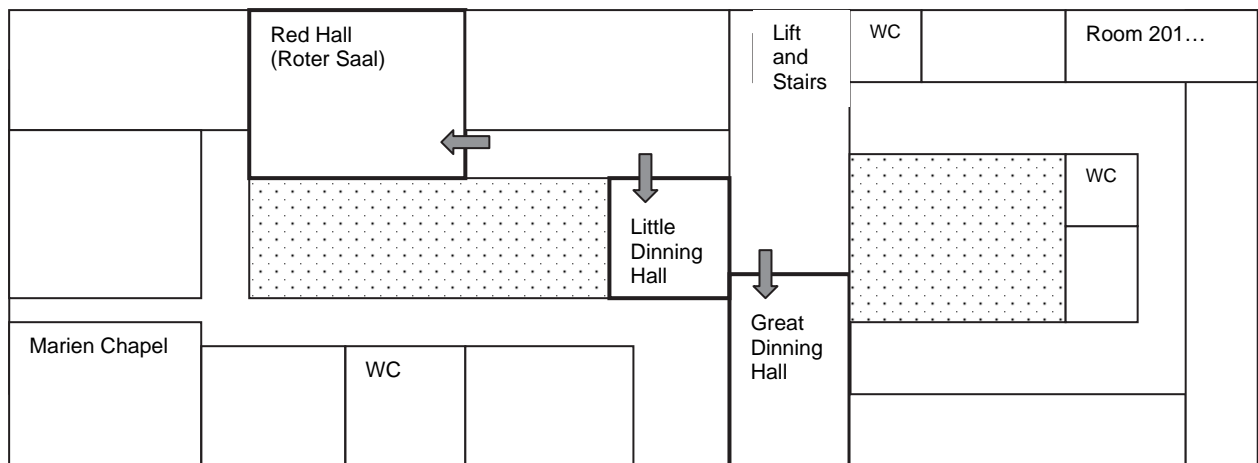
Freising, 1<sup>st</sup> – 3<sup>rd</sup> April 2009

## Roomplan

Ground floor Old Building

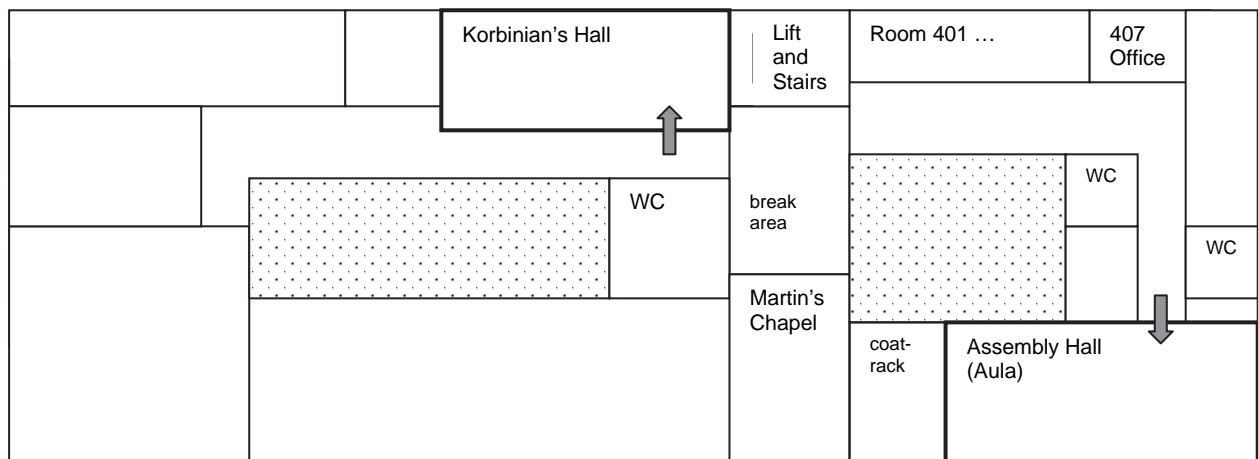


1<sup>st</sup> floor Old Building



3<sup>rd</sup> floor New Building: Internet café Room 307

2<sup>nd</sup> floor Old Building





## **Lectures**

## IRMS-Workshop

### The potential of the <sup>18</sup>O-determination on organically bound oxygen for origin and authenticity checks

H.-L. Schmidt

Isolab GmbH, Schweitenkirchen and Technische Universität München/Germany

The primary sources of oxygen bound to natural compounds are water, carbon dioxide and atmospheric oxygen. Whereas the  $\delta^{18}\text{O}$ -value of  $\text{O}_2$  is globally constant +23.4‰, that of  $\text{H}_2\text{O}$  and  $\text{CO}_2$  vary depending on geographical latitude and local altitude and climate; an immediate expression is the isotopic composition of carbonates as indicators of ancient climates or the body temperature of prehistoric animals. The bulk  $\delta^{18}\text{O}$ -value of organic plant matter is correlated to the local climate of the growth area via that of the meteoric water and the transpiration of the plants, hence plant physiological parameters like anatomic and metabolic properties e.g. of C-3-, C-4- and CAM-plants.

The introduction of oxygen atoms from water into organic binding is performed by various reaction types, catalyzed by lyases, ligases, hydrolases and other enzymes; all these reactions imply kinetic oxygen isotope effects. Furthermore, oxygen compounds with  $\text{sp}^2$ -structure (carbonyl and carboxyl groups) undergo oxygen exchange with plant leaf and animal body water, respectively, and equilibrium isotope effects are connected to these processes. However, due to the relatively slow velocity of the exchange reaction, the isotopic equilibrium may not always be attained. Finally, oxygenase reactions introduce oxygen atoms from  $\text{O}_2$  into organic binding accompanied by a kinetic isotope effect.

On the basis of this knowledge, predictions of bulk and positional <sup>18</sup>O-contents of organic molecules are possible, and their correctness has experimentally been proved for carbohydrates, proteins and some other molecules relevant for food origin assignments. The potential of the causal correlations between biosynthesis and oxygen isotopic characteristics of natural compounds is additionally demonstrated by the discrimination between different possible biosynthetic pathways of gallic acid and the assignment of L-tyrosine to plant and animal origin, respectively, on the basis of their oxygen-18 patterns.

## IRMS-Workshop

### H C N O S stable isotope measurements during the TRACE project – how do we compare?

**F. Camin<sup>1</sup>, L. Bontempo<sup>1</sup>, K. Heinrich<sup>2</sup>, M. Horacek<sup>3</sup>, S.D. Kelly<sup>4\*</sup>, N. Marigheto<sup>4</sup>, C. Schlicht<sup>5</sup>, A. Schellenberg<sup>5</sup>, F. Thomas<sup>6</sup>, A. Rossmann<sup>7</sup>**

<sup>1</sup>Istituto Agrario San Michele all'Adige via Mach 1, 38010 San Michele all'Adige (TN), Italy

<sup>2</sup>Central Science Laboratory, Sand Hutton, York, UK, YO41 1LZ

<sup>3</sup>ARC Seibersdorf Ges.mbH, Seibersdorf, Austria,

<sup>4</sup>Institute of Food Research, Norwich Research Park, Norwich, UK, NR4 7UA

<sup>5</sup>LGL Bayern Dienststelle Oberschleißheim, Oberschleißheim, Germany,

<sup>6</sup>Eurofins Scientific, Rue de Adolfe Bobbiere, CEDEX 03, Nantes, France

<sup>7</sup>Isolab GmbH, Woelkestr. 9/1, D-85301 Schweitenkirchen, Germany,

Over the past four years the seven laboratories routinely providing stable isotope data for the TRACE project have completed nearly 4000 Quality Control measurements on a range of Inter-Comparison Materials (ICMs). These ICMs include porcine collagen, barium sulphate, NIST 1577b bovine liver certified reference material and fresh frozen lamb skeletal muscle amongst others. The challenges of reliably measuring H, C, N and S stable isotope ratios in bulk food matrices are discussed and the outcomes of the QC measurements presented in terms of their intra-laboratory and inter-laboratory precision.

## IRMS-Workshop

### **18O standards for high-temperature conversion (HTC); an inter-laboratory calibration effort with different techniques**

**W. A. Brand**

MPI-BGC Jena in collaboration with USGS, IAEA, ETH, ANU, UFZ and CIO laboratories

The inter-laboratory comparability of 18O data from a variety of sulfates, nitrates and organic materials has been studied and values for new reference materials been established using different instruments and experimental procedures. The more than 5300 individual analyses covered a large span in isotopic compositions, ranging from -56 to + 80 ‰. The materials under investigation included BaSO<sub>4</sub> samples (NBS-127, IAEA-SO5, IAEA-SO6), Nitrate samples (USGS 34, USGS 35, IAEA-NO3), benzoic acids (IAEA-601, IAEA-602), and one caffeine sample (IAEA-600). Starting from primary water reference materials (VSMOW etc) a scale expansion to positive delta values has been established with a heavy water sample and using the classical equilibration technique. Major experimental difficulties were encountered when preparing water samples in Ag capsules for HTC-IRMS analysis including evaporation during preparation and storage as well as air-inclusions in the gas tight capsules. In addition, compound-specific effects like delayed reaction of sulfates, production of NO in the ion source interfering with the CO analyte gas and others have been encountered and had to be corrected for. The finally agreed upon d18O results have a combined error of 0.35 per mill and less.



## IRMS-Workshop

### The online $^{18}\text{O}/^{16}\text{O}$ analysis: development and possible applications

R.A. Werner\*

Institut für Pflanzenwissenschaften, ETH Zürich, Schweiz

\*E-mail: roland.werner@ipw.agrl.ethz.ch

Oxygen is the most abundant chemical element on earth, in inorganic compounds it is involved in key structural compounds and it is providing functional groups for most organic compounds. Oxygen in (natural) organic plant material is derived mainly from three sources ( $\text{CO}_2$ ,  $\text{H}_2\text{O}$ ,  $\text{O}_2$ ) with distinct differences in oxygen isotope composition introduced by different (biosynthesis) reactions with corresponding different oxygen isotope effects. Thus the  $\delta^{18}\text{O}$  value of a compound can help to identify its oxygen source(s) and can provide supplemental information on the chemical nature of biosynthetic reactions or corresponding chemical synthesis reactions. Therefore a generally applicable analytical reaction for an on-line  $\delta^{18}\text{O}$  determination is highly desirable. Today the common sample preparation method for organic and (some) inorganic material is a high-temperature carbon reduction technique with the Schütze/Unterzaucher reaction as underlying principle. Oxygen bearing compounds are reacted with carbon at high temperature up to  $1450^\circ\text{C}$  to convert any O (and H) in the sample quantitatively to CO (and  $\text{H}_2$ ) which can be used as measuring gas(es) for isotope ratio mass spectrometry. After a description of the carbon reduction method for reaction of O in organic and some inorganic samples and a discussion of problems involved with the conversion reaction, selected examples for the application of the carbon reduction method in paleoclimatology, elucidation of biosynthesis reactions and food authenticity will be shortly outlined.

## IRMS-Workshop

### Recent developments of IRMS systems for applications in food analysis

**L. Lange, H.-J. Kupka, F. Volders, H.-P. Sieper**

Elementar Analysensysteme GmbH, Hanau, Germany  
E-Mail: info@elementar.de

The analysis of the stable isotopes of the elements CHNS and O can give useful information on the geographic origin and the authenticity of a food sample or other material. In this talk we want to give an overview about recent instrument- and application developments at IsoPrime and Elementar.

One focus of the presentation will be the analysis of H/D and <sup>18</sup>O in liquids and solids with highest precision in continuous flow mode. The IsoPrime in combination with a “vario” elemental analyzer prove that the ChromeHD technology is the most precise technology for H/D analysis. The low-temperature (=1170°C) pyrolysis for the analysis of oxygen in waters and solids is a reliable and inexpensive upgrade kit that does not require external high temperature heaters.

On the other hand improvements in classical EA-IRMS have been achieved. The multielement isotope ratio analysis is a good tool for food authentication, because an “isotope-fingerprint” of a sample can be created. For this purpose, traditionally multiple analysis of one sample has to be done. This is very time consuming and costly. Based on our experiences with the elemental analysis of inhomogeneous biological material we developed and patented a measurement system for the simultaneous analysis of carbon, hydrogen, nitrogen and sulfur stable isotopes. With this system, all four elements (CHNS) can be analyzed from only one sample.

## IRMS-Workshop

### Advances in compound specific isotope analysis

**D. Juchelka, A. Hilkert and O. Kracht**

Thermo Fisher Scientific (Bremen) GmbH, Germany

About 30 years ago D. E. Matthews and J.M. Hayes have introduced compound specific isotope analysis (CSIA) by isotope ratio monitoring GC/MS (irm-GC/MS).

At the beginning the challenge was the development of a suitable reaction interface that provides quantitative conversion of compounds while maintaining chromatographic integrity.

Today continuous flow techniques can be found in all fields of application with improved performance on sample size, throughput, multiple isotope methods, overall precision and ease of use. Multi-element and multi-component analyses are performed to deduce unambiguous isotope fingerprints on <sup>13</sup>C, <sup>15</sup>N, <sup>18</sup>O and <sup>2</sup>H for authenticity control and determination of the origin of food and flavor.

The growing interest and appreciation in isotope ratio applications requires new features and functionalities of the instrumentation.

A new concept for an automated multi-element irm-GC/MS will be discussed. It includes automated switching between the combustion reactor and the high temperature conversion reactor. The combustion mode has been redesigned to determine C and N isotope ratios using identical reactor conditions. The concept incorporates the ConFlo IV as the universal interface to the IRMS. All ConFlo IV capabilities are available for irm-GC/MS, e.g. injection of up to five reference gases, reference gas dilution and automatic H<sub>3</sub><sup>+</sup> factor determination.

The recent introduction of irm-LC/MS has enlarged the range of applications to compounds with high molecular weight, high polarity and thermal instability. Inherent restrictions from the conversion technology in the liquid phase require more focus on the HPLC applications and techniques.

The principle of the devices will be discussed with respect to the dynamic range, precision, accuracy together with the requirements on sample size. Examples for multi-element and multi-component isotope analysis will be shown.

## Main Session

### Improving decision making capabilities for food defense and agricultural protection

R. Linton\*

Department of Food Science, 745 Agricultural Mall Drive, Purdue University, West Lafayette, IN USA  
49707-1160

\*E-mail: linton@purdue.edu; Tel: 765-494-6481; Fax: 765-494-7953

The food industry has many points of vulnerability making it a target for intentional attacks of food contamination. As a result, there is a need to develop crisis management programs to understand the importance of decision-making capabilities during a bioterrorist attack to the food supply. The Purdue Food Defense Computer Simulation, or FDCS, was developed in 2004 as a collaborative effort of Purdue University (School of Agriculture, Purdue Homeland Security Institute, and Krannert School of Business) and has been enhanced since by collaboration with Kansas State University (School of Agriculture), North Carolina State University (Veterinary School), and Indiana University (School of Public Health and Environmental Affairs). Since the beginning, FDCS has integrated input from all of the important professionals involved in food safety and food defense including government (federal, state, local), industry (production, distribution, manufacturing, retail, associations), food defense professionals (police, healthcare, emergency management etc.), academicians, media, and the public. Our mission is to develop a learning platform for participants to understand about public health and economic impacts based upon decisions that are made during the simulation activity. The simulation activity involves the introduction of a food contaminant (intentionally or inherently) at selected points in the food production chain. Participants are able to make decisions related to products and ingredients that may be affected, resulting in significant economic and public health impacts. The simulation models the supply chain from supplier to manufacturer to retailer. It utilizes inputs from 9 company-based teams [3 bulk ingredient companies, 3 processors, and 3 retailers] and 4 additional teams [a first responder team, a USDA team, a FDA team, and media sources] that provide technical guidance throughout the exercise. The modeled supply chain includes products and their ingredients; geographic production, warehousing and shipping; quantities of production and sales; economic impacts of food product recalls, and public health impacts (illnesses, deaths, etc.) for each U.S. state. The simulation is conducted in Purdue's state-of-the-art Envision Center allowing the "companies" to be placed in separate, soundproof rooms according to their place in the supply chain. Participants are provided data from the computer simulation model and from electronic reports of "government" teams and the "media." FDCS was developed to: 1) determine the effectiveness of computer simulation models for food defense and protection awareness, and, 2) measure impacts of decision-making in a virtual food defense and protection crisis. After the computer simulation was developed, over 300 industry representatives have "played" the simulation. Through follow-up surveys with participants, three key findings were noted: 1) communication between all groups in the supply chain is pertinent and challenging, 2) approaches used to solve inherent food safety problems cannot be used alone to address threats to food defense, and, 3) human resource procedures regarding new hires and disgruntled employees should involve additional security measures. FDCS is a valuable resource for food defense awareness, and ultimately, it helps to educate companies about intentional food contamination risks and vulnerabilities along with tracking decision-making consequences.

## Main Session

### Food authenticity – legislation and enforcement

#### M. Woolfe\*

Food Standards Agency, London, UK

\*E-mail: mark.woolfe@foodstandards.gsi.gov.uk; Tel: +44 2072768176; Fax: +44 2072768910

The Food Standards Agency has an active food authenticity programme. The main objective of the programme is to develop new methods to verify the labelling and description of foods, but also to use these methods in investigative surveys of the UK food market. The Agency is not only dealing with risk assessment and management, but is the competent food regulatory authority in the UK. Although food enforcement in the UK is devolved to local government, the Agency's authenticity programme aims to support food labelling and standards policy development, as well as food enforcement and official control laboratories.

The scope of the programme has been defined by answering two main questions. Can a method be developed to verify the description of the food? Also, can the description be defined or linked to legislation or a standard? There are a large number of food regulations that protect certain names and cover the description of food, from the horizontal food labelling regulations to vertical specific food and marketing regulations. Where there is no specific legal basis to authenticate the food, then other means have to be used, whether it is an international standard from ISO or Codex or even a Code of Practice or guidance. In previous cases e.g. Basmati rice, which is a customary name, a method was developed, and then required a Code of Practice to define the food and apply the method.

Where possible methods developed under the authenticity programme have been transferred to UK official control labs (public analysts). A recent example has been the transfer of a portfolio of methods based on DNA technology to public analysts permitting the verification of meat and fish species, and determining adulteration of durum wheat pasta, Basmati rice and orange juice. In others, standard operating procedures have been developed and made available to public analysts.

Method development for enforcement is rarely a completed process. There are good examples where once a method has been applied; those wishing to avoid detection of misdescription or commit fraud will find a way of confounding the method. In such cases further or continual development of the method is required. Two case studies on Basmati rice and the use of hydrolysed proteins for water retention will be discussed.

## Main Session

### Assuring brand integrity

R. I. Aylott\*

Diageo PLC, Technical Centre Europe, Menstrie, Clackmannanshire FK11 7ES, Scotland

\*E-mail: ross.i.aylott@diageo.com; Tel: +44 1259 766875; Fax: +44 1259 766807

Brands can greatly contribute to the value of modern businesses. Brand value is based upon many factors that include reputation with consumers and ability to generate repeat sales. While brand owners and manufacturers go to great lengths to ensure the quality and consistency of their products, others often take illegal advantage by counterfeiting them. This paper will describe the advancing scientific tools that the distilled spirits industry has developed and deployed to protect both its brands and consumers.

The integrity of high value distilled spirits is safeguarded throughout the business process. Innovators and marketers carefully design their brands. Lawyers protect the trademarks and intellectual property. Distillers, blenders and quality assurance specialists ensure that the resulting liquid is manufactured according to definition, formulation and tight quality standards. Finally, packaging specialists ensure that right materials protect the liquid and that labels correctly describe the final product so that fully compliant products are sold to satisfied consumers. From a regulatory compliance perspective, definitions for all spirit drinks produced in the European Union may be found in Regulation 110/2008 (1). In the case of Scotch whisky, this regulation works in parallel with the UK Scotch Whisky Act 1988 (2).

The counterfeiting of manufactured goods is a global problem. Estimates for the market in fake goods reach as much as 10% of world trade. The distilled spirits industry is a major global exporter; over 80% of Scotch whisky production is exported from the UK. However, counterfeiting in certain markets leads to consumers being deceived, governments losing tax revenue and producers losing business. Consumer health can also be put at risk if a counterfeiter uses dangerous liquids. In order to combat these risks, we have developed a range of technologies to protect the product and to enable brand owners and enforcement agencies to take action against counterfeiters.

Fake counterfeit liquids take many forms depending on the materials available and the sophistication the counterfeiter. Investigations invariably require authenticity analyses that can be expensive and time consuming, especially if laboratory work is required. We responded by developing fast and reliable field tests that, for example, eliminate many whisky samples from long established chromatographic procedures (3). A portable brand authenticator utilising UV/visible spectra is now widely deployed to determine Scotch whisky authenticity (4) and this instrument is finding application in other sectors. Authenticity indicators that enable dipstick tests are proving similarly useful for protecting white spirits such as gin and vodka (5).

These field tests enable investigators and consumer protection officers to screen relatively large numbers of samples quickly and at low cost. The authenticity of suspect samples may be confirmed by chromatographic analysis in the laboratories of brand owners and enforcement agencies. The results may then be presented as evidence in the subsequent prosecution of offenders.

#### References:

- (1) Regulation (EC) No. 110/2008 of 15 January 2008.
- (2) Scotch Whisky Act 1988, H M Stationary Office, London, 1988 Chapter 22.
- (3) Aylott, R. I., Clyne, A. H., Fox, A. P., and Walker, D. A., (1994) *Analyst*, 119, 213 - 221.
- (4) Mackenzie, W.M. & Aylott R.I., (2004) *Analyst*, 129, 607 – 612.
- (5) Aylott R. I., (2008) *Distilled Spirits: Production, Technology and Innovation*, Nottingham University Press, Nottingham, 281 - 287.



## **Main Session**

### **Assessment of traceability in the mineral water, honey and chicken sectors: lessons learnt**

**P. Olsen**

Abstract wasn't available at the time of printing.

### **Food traceability cost benefit analysis: a model for qualitative assessment**

**G. Chryssochoides**

Abstract wasn't available at the time of printing.

## Main Session

### Tracing your food – an interactive demonstration

**H. Lehr\***

FoodReg, Barcelona, Spain

\*E-mail: heiner.lehr@foodreg.com; Tel: +34 93 2801555; Fax: +34 93 280 30 15

Tracing your food is an interactive animation web site that will exemplify the TraceFood framework from different points of view. The TraceFood framework tries to guide food businesses in the implementation of good traceability practices and gives the industry tools (most notably TraceCore) to establish global food traceability.

The web site will exemplify traceability from different angles or viewpoints

- The consumer view – What information is available for the food I buy?
- The operational view – What data do I need to capture?
- The supply chain view – Safe and responsible purchasing
- The quality assurance view – Remote Quality Assurance
- The public authority view – Public health and food safety
- The IT view – Specification for traceability systems

The web site will mix video with animation and more “classical” presentations (text and link collections) in order to present the information in a easily comprehensible way.

Users can register with minimal data and can state their interest in traceability. If they agree to publish their data, they get access to other people’s names who share common interests.

## Main Session

### The development of methods to verify the geographical origin of chicken meat

S.D. Kelly<sup>1</sup>, N. Marigheto<sup>1</sup>, Y. Gunning<sup>1</sup>, I. Colquhoun<sup>1</sup>, A. Schellenberg<sup>2</sup>, C. Schlicht<sup>2</sup>, F. Camin<sup>3</sup>, M. Perini<sup>3</sup>, L. Bontempo<sup>3</sup>, K. Heinrich<sup>4</sup>, A. Rossmann<sup>5</sup>, F. Thomas<sup>6</sup>, E. Jamin<sup>6</sup>, M. Horacek<sup>7</sup>, S. Rummel<sup>8</sup>, S. Hoelzl<sup>8</sup>, C. Dekant<sup>8</sup>

<sup>1</sup>Institute of Food Research, Norwich, United Kingdom

<sup>2</sup>Bavarian Health and Food Safety Authority, Oberschleißheim, Germany

<sup>3</sup>FM-IASMA Research Centre, San Michele all'Adige, Trento, Italy

<sup>4</sup>Central Science Laboratory, York, United Kingdom

<sup>5</sup>Isolab GmbH, Schweitenkirchen, Germany

<sup>6</sup>EUROFINS Scientific, Nantes, France

<sup>7</sup>ARC Seibersdorf research GmbH, Seibersdorf, Austria

<sup>8</sup>Bayerische Staatssammlung für Paläontologie und Geologie, Munich, Germany

#### Background

Recent food scares such as Avian Flu and the malpractice of some food producers (e.g. use of banned nitrofurans in chicken production) have increased public awareness regarding both the validity of claims of origin of chicken meat and the means by which it has been produced. In addition, analytical identification of meat origin from outside the EU is a valuable tool for enforcement authorities highlighted recently by the FSA Northern Ireland investigation into a meat coldstore operated by Euro Freeze, Co. Fermanagh (<http://www.food.gov.uk/news/newsarchive/2005/dec/eurofreeze>).

#### Aims

To develop reliable methods that can establish the geographical origin of poultry based on stable isotope and metabolite tracers that are indicative of origin.

#### Approach

Our research has focused on exploiting the natural variation, or fractionation, that occurs in the isotopic content of the bio-elements, hydrogen, carbon, nitrogen, sulphur and the heavy element strontium to determine the geographical origin of poultry and dietary patterns that can act as proxies for geographical location. In addition metabolite screening has been used to identify organic compounds that indicate geographical origin. The combination of these techniques with multivariate statistics to determine the geographical origin of food has been investigated.

#### Results

In some instances comparison of one or two variables is sufficient to discriminate geographical origins; for example, carbon stable isotope ratios of poultry 'protein' (fat-free dry mass) indicate the quantity of maize in the diet and this leads to useful discrimination of origin. Multivariate analysis of the stable isotope and metabolomic data permits simultaneous comparison of several origins on a global and regional scale and quantitative assessment of correct classification.

## Main Session

### Food maps how do they work: an interactive demonstration

G. van der Veer<sup>1\*</sup> and S. T. Höskuldsson<sup>2\*</sup>

<sup>1</sup>Geochem Research B.V., Office Nieuwegein, Utrecht, the Netherlands

<sup>2</sup>Maritech ehf, Kopavogur, Iceland

\*E-mail: gvanderveer@geochem.nl, Stefan@maritech.is; Tel: +31 30 2219019, Tel: +354 545 3200

Food maps, or better food specification prediction maps, allow to verify the geographical origin of food stuff on the basis of a combination of specific local geo-climatic characteristics. These local characteristics are often reflected by the isotopic and trace element composition of food, thereby providing a kind of local fingerprint. As different countries or regions can have the same climatic conditions or geological background – whereby providing similar fingerprints –, the strength of the food map approach is based on the combination of different maps that each provide additional detail and allow to further confine the area where certain group of isotopic and trace element specifications apply.

Currently, food maps for different food commodities such are being developed within the framework of the TRACE project. Herein, the  $\delta^2\text{H}$ ,  $\delta^{18}\text{O}$  and  $^{87}\text{Sr}/^{86}\text{Sr}$  isotope composition of food seem to be of most relevance. For these parameters, food maps have been developed for various food commodities including mineral water, honey and wheat. These maps for contain the predicted upper and lower confidence limits (specifications) for the isotopic composition of these commodities within Europe. The specifications can be used to verify the geographical origin of food by comparing the actual isotopic composition measured in an unknown sample with the predicted specifications from its acclaimed production region.

Handling of the combination of different maps in the food map approach can be quite demanding, and the combined results are often difficult to oversee. To facilitate the handling of different maps at the same time, and to allow for easy verification of the geographical origin of food, a Visual Earth application has been developed. During this interactive presentation the use and limitations of the application will be further demonstrated and discussed.

## Main Session

### Tracing provenance: differentiating European from Argentinean foods

D.A. Wunderlin<sup>1\*</sup>, H.A. Oстера<sup>2</sup>, P.Peral-García<sup>3</sup>, M. Cagnoni<sup>2</sup>, M.V. Baroni<sup>1</sup>, N.S. Podio<sup>1</sup>, M.P. Fabani<sup>1</sup>, M.P. Díaz<sup>1</sup>, R. Badini<sup>4</sup>, C.M.Inga<sup>4</sup>.

<sup>1</sup>Universidad Nacional de Córdoba, Fac.Cs. Químicas e ISIDSA, Córdoba-Argentina.

<sup>2</sup>INGEIS-CONICET, Buenos Aires- Argentina.

<sup>3</sup>CIGIBA, Universidad Nacional de La Plata. La Plata- Argentina.

<sup>4</sup>CEPROCOR, Ministerio de Ciencia y Tecnología Pcia. Córdoba. Córdoba- Argentina.

\*E-mail: dwunder@fcq.unc.edu.ar; Tel: +54 351 4333193; Fax: +54 351 4333194

This work is part of activities carried out in Argentina as part of the TRACE project, which is granted by EU (FP6) with the main goal of assessing food provenance considering several endpoints. Among possible hypothesis, we evaluated the follow up of several metals from farm soil to food products as well as changes in stable isotopic composition in foods produced at five different areas of Argentina. In this way, we look to construct a fingerprint of such foodstuffs, pointing out differences among studied areas as well as with similar products produced in other latitudes, like EU. If this idea works, it could be rise to the concept of chemical traceability of foods, which could be a valuable complement for other traceability techniques, reinforcing the confidence of consumers in such foods that can be traced "from farm to fork".

Studied Areas in Argentina include the provinces of Buenos Aires, Entre Ríos, Córdoba, San Juan and Mendoza, covering wet-templates areas (pampas) and semi-arid regions with different geology. Studied Areas in EU correspond to those covered by TRACE project (Austria, Germany, Denmark, France, Greece, Ireland, Italy, Poland, Portugal, Spain and UK). Analyzed elements include 31 trace elements and stable isotopic pattern ( $\delta^{13}\text{C}$ ,  $\delta^{15}\text{N}$ ,  $\delta^{18}\text{O}$ , the ratio  $^{85}\text{Sr}/^{87}\text{Sr}$ , etc.). Thus, we looked to construct a fingerprint characteristic for a particular product, arising from a particular country, or a single area within a country. Food commodities included in our study were: Honey, Olive Oil, Beef, Wheat and Wine. We also analyzed the composition of agricultural soils and irrigation water.

Preliminary results show that it is possible to differentiate Argentinean honey and olive oil from the corresponding European counterpart. However, such differentiation needs the use of a wide range of chemical parameters to be effective. Sometimes, differences between Argentinean and European foods could be attributed to differences in agricultural soil, climate, farm practices, etc. However, some differences seems to be consequence of different pollution degrees, like high levels of lead found in EU honey in relation to the Argentinean product. Furthermore, the evaluation of food provenance should not be exclusively based on the composition of the final product, but considering the association between agricultural soil and the commodity, which can be done by multivariate statistical methods, constructing prediction models appropriate to include the whole fingerprint and its association with provenance soil and environment. When evaluating foods and beverages obtained after an industrial process, like wine, more variables are needed to fully assess their provenance. In our experience, plant physiology and wine-making practices introduce changes that should be considered when evaluating traceability.

We conclude that it is possible to differentiate Argentinean from European foods after considering several parameters that include chemical elements and stable isotopic patterns, constructing a fingerprint that should account for different species and production practices in addition to agreement with the provenance soil and their environmental conditions. These results point out the need of constructing extensive databases, including chemical and isotopic composition of food in association to soil characteristic, geology, pollution degree, etc. Such databases should be used in the future to support claims for the origin of foods, helping the international trade and increasing consumers confidence by avoiding frauds.

## Main Session

### Confirmation of beer authenticity by spectroscopic fingerprint techniques

**G. Downey<sup>1\*</sup>, J. Hajslova<sup>2</sup>, V. Baeten<sup>3</sup>, L. Mannina<sup>4</sup>, J. Donarski<sup>5</sup> and J. M. Moreno<sup>6</sup>**

<sup>1</sup>Teagasc, Ashtown Food Research Centre, Dublin 15, Ireland

<sup>2</sup>Institute of Chemical Technology, Prague, Czech Republic

<sup>3</sup>Walloon Agricultural Research Centre, Gembloux, Belgium

<sup>4</sup>Institute of Chemical Methodologies of CNR, Rome

<sup>5</sup>Central Science Laboratory, York, UK

<sup>6</sup>DG Joint Research Centre, Ispra, Italy

\*E-mail: gerard.downey@teagasc.ie; Tel: +353 1 8059533; Fax: +353 1 8059550

Beer is an economically-important product of cereal fermentation. In Belgium, beers from Trappist monasteries enjoy particular status on account of their perceived high and consistent quality. To protect this status and as an aid to marketing, beers brewed in monastic sites under the control of Trappist monks are entitled to display a Trappist logo on their label. A number of breweries which were once under monastic control produce beers in the Trappist style but are not entitled to use the label logo.

A number of spectroscopic fingerprint techniques have been deployed to develop models which may confirm the identity of Trappist beers. A collection (n=124) of Trappist and non-Trappist beers (mainly sourced in Belgium) have been collected from several production batches and analysed contemporaneously by these methods. Models have been developed to discriminate between Trappist and non-Trappist beers using this initial sample set. A second set of beers (n=124) was collected from different batches of beers on a second occasion to (a) evaluate the accuracy of previously-developed discriminant models and (b) to determine the stability of the models when applied to beers which have been stored for an extended time period.

Results indicate the potential of a number of techniques to achieve the required levels of accuracy effectively. These and other related observations will be discussed.



## Main Session

### Molecular biological methods for authenticating food

**H. Broll\***

Joint Research Centre of the European Commission, Institute for Health and Consumer Protection, Molecular Biology and Genomics Unit. Ispra, Italy

\*E-mail: hermann.broll@jrc.it; Tel: +390332783538; Fax: +390332785483

For the first time at the end of the last century molecular biological methods has been used for traceability purposes and published.

The development of the polymerase chain reaction (PCR) was the main trigger to initiate the application of molecular biological methods also in the area of food/feed. The PCR became a standard application in food/feed control laboratories. PCR methods have become international standards in the field of genetically modified food and in the area of microbiology.

The first attempt to apply the technique also in the field of food and feed authenticity started in the frame of the European funded 5<sup>th</sup> Framework project "MolSpec-id". Methods like the real time PCR and AFLP have been further developed and validated.

In the frame of the 'TRACE' project a specific work package has been set-up to explore in more detail the applicability and suitability of molecular biological methods for traceability purposes.

The presentation will give an overview about the achievements made during the 5 year project.

It will also give details about the database developed in the frame of 'TRACE' and the benefit for control authorities as well as for interested stakeholders using the information given in the database (<http://trace.eu.org/mbdb/>).

## **Main Session**

### **Molecular based tools for traceability of beef**

**R. Negrini**

Abstract wasn't available at the time of printing.

## Parallel Session A

### Boron isotope compositions of crop plants: a new tracer for the origin and authenticity of food

M. Rosner<sup>1\*</sup>, W. Pritzkow<sup>1</sup>, J. Vogl<sup>1</sup> and S. Voerkelius<sup>2</sup>

<sup>1</sup>Federal Institute for Materials Research and Testing, Division Bioanalytics, Berlin, Germany

<sup>2</sup>Hydroisotop GmbH, Laboratorium zur Bestimmung von Isotopen in Umwelt und Hydrologie, Schweitenkirchen, Germany

\*E-mail: martin.rosner@bam.de; Tel: +49 30 81044112; Fax: +49 30 81043527

Boron and its isotopes have been used for a long time in geo- and environmental sciences to study mixing and fractionation processes as well as sources of anthropogenic contamination. The authentication of food using isotopic compositions of specific reservoir- and process-sensitive elements is a relatively new and growing application in isotope chemistry. Unlike some well-established stable and radiogenic isotope systems (H-C-N-O-S-Sr-Pb) the use of boron isotopes for food authentication is almost unexplored. However, due to the essential role of boron for embryonic development and organogenesis in plants and animals and the enormous range of isotopic compositions found in nature boron potentially is a very interesting isotope system to verify the origin and possible cultivation methods of various types of food.

Our first boron isotope data of vegetables, corn and fruits cover most of the natural variation of about 100‰ in  $\delta^{11}\text{B}$ . These highly variable boron isotopic compositions of the investigated crops reflect regionally varying contributions of the natural background (geology, hydrology, soil, ...) as well as anthropogenic processes during cultivation like fertilization and irrigation. As many of these natural reservoirs and anthropogenic processes will implant distinct isotopic fingerprints, the boron isotopic composition of crops has a high potential as a tracer for specific questions related to the authenticity of food.

Most of the crop plants investigated show  $\delta^{11}\text{B}$  values between -5 and +20 ‰. Those values likely reflect the geogenic background of the rocks and soils where the crops were cultivated. To date the isotopically most extreme samples are a pepper sample from Israel (+35 ‰  $\delta^{11}\text{B}$ ) and a cabbage reference material from NIST (-24 ‰  $\delta^{11}\text{B}$ ). The high, seawater-like,  $\delta^{11}\text{B}$  value of the Israeli pepper may reflect irrigation with waters derived from the Dead Sea (+57 ‰  $\delta^{11}\text{B}$ ; (1)) or salinar aquifers. The low  $\delta^{11}\text{B}$  value of the NIST cabbage may reflect fertilization of a boron-loving crop with fertilizers coming from non-marine evaporate deposits (e.g. (2)).

In addition to the direct tracing of reservoirs and processes using the  $\delta^{11}\text{B}$  of crop plants, boron isotope fractionation during incorporation, transport and integration of boron in the plants is an unexplored field to study the function and transport mechanisms of boron in biological systems (3).

#### References:

- (1) Vengosh A.; et al. (1991) *Geochim. Cosmochim. Acta*, 55: 1689-1695.
- (2) Barth S. (1993) *Geol. Rundsch.*, 82: 640-651.
- (3) Takano J.; et al. (2002) *Nature*, 420: 337-340.

## Parallel Session A

### Soil composition as potential tracer for food authentication: can it be derived from local geology?

B. Wimmer<sup>1\*</sup>, H. Fröschl<sup>1</sup>, R. Hamid<sup>1</sup> and G. Heiss<sup>1</sup>

<sup>1</sup>Austrian Research Centers GmbH - ARC, A-2444 Seibersdorf, Austria

\*E-mail: Bernhard.wimmer@arcs.ac.at; Tel: +43 (0)50550-3544; Fax: +43 (0)50550-3520

Trace element composition of plants is partly influenced by the composition of the soil on which they are growing. Further on this signature could be found in some extent also in tissue of animals using these plants as feed. Therefore trace element composition of food commodities could provide a means of verifying the authenticity of food products. It would be very advantageous and cost-effective if these natural tracers in food commodities could be predicted from existing information of local geology. As a first step to proof this concept it has to be shown that the soil composition reflects adequately the underlying geology.

For that purpose 3 “geology classes” were selected (limestone, igneous and shale/mudstone/clay/loess). 2188 top soil samples were collected in 21 different European “model sampling areas” representing these geology classes. At 126 sampling points also sub soil samples were taken. At these sampling points (6 per area) bulk soil composition of top soil and sub soil samples were determined by ICP-MS and XRF (major elements, loss on ignition and 39 trace elements). All the soil samples were extracted with a 1 molar ammonium nitrate solution. This extraction method is known to characterize the concentration of plant available elements in the soil solution.

The element composition of bulk top soil is highly correlated with the corresponding sub soil samples (almost all major and trace elements). Higher top soil concentrations of some heavy metals as an evidence for anthropogenic influence could only be observed in a few cases (Cd, Pb, Zn in Poland; Pb in Trentino, single points with high concentrations of heavy metals in sampling areas of Chalkidiki, Lakonia and Barcelona). Elevated concentrations of sulfur in top soil compared to sub soil might be an indicator for a higher content of organic matter in these samples. The high correlation between samples with CaO > 5 M-% and loss on ignition showed the presence of carbonates (mainly limestone) in the soil. Comparing the elemental composition of soil samples collected in the 3 pre-selected geological classes showed that there is no “unique” marker (one element or a combination of elements) to differentiate between these classes on the whole. But distinct differences exist between single sampling areas or groups of them. E.g. concentrations of Rb, U, Cs and Sn were clearly elevated in 3 (of totally 5) sampling areas with underlying igneous geology. Soil samples in Mühlviertel and Chalkidiki (igneous rocks) exhibited high concentration in Sr.

It can be concluded that soil composition could serve as natural tracers for identifying the origin of food commodities on a regional scale. On the other hand it is not possible to establish a single model to derive soil composition from broad geological classes.

## Parallel Session A

### Meat authentication – what did the animal eat?

F.J. Monahan<sup>1\*</sup>, A.P. Moloney<sup>2</sup>, S.M. Harrison<sup>1</sup>, A. Zazzo<sup>3</sup>, B. Bahar<sup>1</sup>, C.M. Scrimgeour<sup>4</sup>, and O. Schmidt<sup>5</sup>

<sup>1</sup>UCD School of Agriculture, Food Science & Veterinary Medicine, Belfield, Dublin 4, Ireland.

<sup>2</sup>Teagasc, Grange Beef Research Centre, Dunsany, Co. Meath, Ireland.

<sup>3</sup>Muséum national d'Histoire naturelle, Département Ecologie et Gestion de la Biodiversité, 55 rue Buffon, F-75231 Paris cedex 05, France.

<sup>4</sup>Scottish Crop Research Institute, Invergowrie, Dundee, DD2 5DA, UK.

<sup>5</sup>UCD School of Biology & Environmental Science, Belfield, Dublin 4, Ireland.

\*E-mail: frank.monahan@ucd.ie; Tel: +353 1 7167090; Fax: +353 1 7161147

It is well established that the pre-slaughter diet of animals influences the nutritional and elemental composition of meat. It follows that analysis of meat can elicit information retrospectively about the pre-slaughter diet of animals; this information is particularly relevant to-day when consumers are ever more concerned about the connection between animal diet and the nutritional quality and safety of the meat they consume. Information on the dietary background of animals is also important in validating claims around the production of branded products such as “grass-fed” beef or “corn-fed” chicken. Among a variety of techniques, light element stable isotope ratio (SIR) analysis (SIRA) is used to obtain information about the dietary background of food animals (1-4). However, changes to the pre-slaughter diet of animals may go undetected if (i) the response of a tissue to a diet switch (tissue turnover) is slow, (ii) a change in pre-slaughter diet is subtle, or (iii) appropriate tissues are unavailable for analysis. In our research we are exploring these three issues.

(i) In a controlled cattle feeding study, the substitution of grass silage (C3 plant) with maize silage (C4 plant), either fully or in part, for a period of 168 days was clearly reflected in the SIR of muscle, with each 10% change in the maize silage consumption resulting in a 0.9 to 1.0‰ shift of  $\delta^{13}\text{C}$  in muscle (4). After 168 days the SIR of muscle had not reached equilibrium with that of the diet, indicating incomplete turnover. Measurement of muscle turnover rates following a pre-slaughter diet switch to a  $^{13}\text{C}$ - and  $^{15}\text{N}$ -enriched diet, using SIRA, showed a half life of between 134 and 159 days for turnover of carbon or nitrogen in beef (5). Similarly, in lamb we obtained half lives of between 65 and 101 days for carbon.

(ii) When more subtle diet switches take place, for example between two C3 plant sources such as grass silage and barley, differences of the order of 3 ‰ in the dietary components resulted in a difference of 1.1‰ in bovine muscle  $\delta^{13}\text{C}$  values 4 months after the diet switch.

(iii) Incremental animal tissues, such as hair, wool and hoof, offer distinct advantages over integrating tissues, such as muscle, in reconstructing the pre-slaughter diet history. In bovine hair the response to a C3 to C4 diet switch is rapid, with a half life of 1.7 days for turnover of the fastest and largest C pool (6). This rapid response enabled the detection of an unplanned switch in the pre-slaughter diet of cattle (6,7), highlighting the usefulness of incremental tissues in reconstructing the diet histories of human foods of animal origin.

This research illustrates the importance of considering potential pre-slaughter diet switches, their extent and duration, as well as tissue turnover, when interpreting the results of analyses of integrating tissues such as muscle and making inferences about pre-slaughter diet history. Analysis of incremental tissues, such as hair, is an extremely useful tool for substantiating the information obtained from integrating tissues and for comprehensively reconstructing diet history.

#### References:

- (1) Piasentier E. et al (2003) *Meat Sci* 64: 239-247.
- (2) Boner M. & Förstel H. (2004) *Anal Bioanal Chem* 378: 301-310.
- (3) Schwertl M. et al (2005) *Agr Ecosyst Environ* 109: 153-165.
- (4) Bahar B. et al (2005) *Rapid Commun Mass Sp* 19: 1937-1942.
- (5) Bahar, B. et al (2008) *J Anim Sci* in press.
- (6) Zazzo A. et al (2007) *Can J Zool* 85: 1239-48.
- (7) Harrison SM. et al (2007) *Rapid Commun Mass Sp* 21: 479-486.

## Parallel Session A

### High resolution time of flight mass spectrometry (HR TOF-MS) employing direct analysis in real time (DART) ion source: a challenging technique for food traceability

J. Hajšlová\*, L. Václavík, T. Čajka, V. Hrbek, K. Riddelová and M. Tomaniová

Institute of Chemical Technology Prague, Department of Food Chemistry and Analysis, Technická 3, Prague 6, 166 28, Czech Republic

\*E-mail: jana.hajslova@vscht.cz; Tel: +420 602833424

A lot of scientific effort has been spent to develop rapid, reliable, and cost effective analytical approaches applicable for the authentication of various food commodities. Besides of spectroscopic techniques employing nuclear magnetic resonance (NMR), Raman, or infrared spectra, a wide range of methods employing gas chromatography–mass spectrometry (GC–MS), and/or high-performance liquid chromatography (HPLC) hyphenated to MS with atmospheric pressure chemical ionization (APCI), has been implemented for this purpose. Some procedures, such as matrix assisted laser desorption/ionization mass spectrometry (MALDI), direct head-space mass spectrometry (HS-MS), and/or direct infusion MS allow reduction of analysis time thanks to elimination of chromatographic separation step.

Over the few recent years, a large number of novel ambient desorption ionization techniques, such as desorption electrospray ionization (DESI), atmospheric-pressure solids analysis probe (ASAP), direct analysis in real time (DART) and some others, have become available providing further improvements. Their main advantages compared to conventional techniques, involve the possibility of direct sample examination in the open atmosphere, minimal, or no sample preparation requirements, and, remarkably high sample throughput. DART, which has been investigated in this pilot study, represents one of APCI-related techniques employing a corona discharge for the ionization. Metastable helium atoms, originated in the plasma, react with ambient water, oxygen, or other atmospheric components to produce the reactive ionizing species. DART ion source has been shown to be efficient for soft ionization of a wide range of both polar and non-polar compounds.

This presentation will demonstrate the potential of DART TOF-MS technique to distinguish geographical and/or species origin as well as processing practice employed for particular food production, on the basis of metabolomic profiles. Since a large volume of data is typically obtained during the measurements of positive /negative mass spectra, smart chemometric tools have to be used to establish mathematical model for classification of samples. In our study, linear discriminant analysis (LDA) and artificial neural networks (ANN) have been employed for examination of TRACE project matrices represented by olive oil, honey, beer and meat.

#### References:

- (1) Venter A., Nefliu M., R. and Cooks G. (April 2008) Trends Anal. Chem. 27, Issue 4: 284-290
- (2) Cody R.B., Laramée J.A. and Durst H.D. (2005) Anal. Chem. 77: 2297–2302

*Acknowledgement: This work has been carried out with support from the European Commission through the Sixth Framework Programme under the Food Quality and Safety Priority (Contract no. CT-2005-006942, TRACE).*

## Parallel Session A

### Validated methods for plant and animal species differentiation

#### A. Pardigol

Eurofins Scientific analytics

We present the validation of a model system for traceability of cereals in food stuffs, based on qualitative and quantitative PCR methods developed in the framework of TRACE project WP 3.3. The work included the setup of a collection of biological raw materials, database research for candidate DNA markers, testing of a DNA extraction protocol for cereals, and investigation of validation parameters such as specificity of the PCR systems, limit of detection in terms of DNA copies and mg cereals per kg food matrix ("ppm"), quantification accuracy, and matrix effects. A PCR system specific for gluten containing cereals (barley, rye, wheat and oat) could be developed, able to distinguish oat from the other 3 cereals. Validation of this PCR system included qualitative detection of oat DNA in commercial food stuffs, as well as detection and quantification of oat DNA in spiked samples with known amounts of oat. For this purpose, we used different model systems: for example, soy beans were spiked with decreasing amounts of oat, ranging from 500mg oat/kg to 10mg oat/kg. Our experiments demonstrated, that the limit of detection in soy is close to 10ppm oat (mg/kg). Sensitivity tests showed no false negative results with the oat-discriminating standard PCR system, whereas a higher ratio of false negative results was observed with the quantitative Real-time PCR system, indicating a difference in sensitivity of the standard PCR and the Real-time PCR system.

One important aspect of the project aimed at the examination of conversion factors to translate the unit of measurement "DNA copies" into the unit of measurement "mg oat per kg matrix".

Our experiments indicate, that the unit "DNA copy number" may be used to calculate conversion factors for the quantification of the oat content per kg matrix", but this needs to be validated for each matrix, on a case by case basis. The quantification experiments further showed, that absolute quantification assays appear to be more biased than relative quantification, where differences in DNA extraction efficiencies or pipeting errors may be factored out.

The assays developed and validated in the framework of this project may be useful as an analytical tool for the detection of gluten containing cereals in food stuffs (key words "gluten intolerance" & "celiac disease"): as oats may represent a useful part of a gluten-free diet, patients need careful advice, which can only be achieved by means of reliable testing methods. The methods presented here may be used to either detect barley, rye, and wheat in oat samples, or to detection oat in food samples not supposed to contain oat. For application in routine, additional validation data are required.

SOPs for DNA extraction, qualitative detection of oat, and quantitative detection of cereals in food stuffs have been provided in the framework of the TRACE project.

## Parallel Session A

### Development of an array-based traceability tool for (cereal) specialty products

T.W. Prins<sup>1\*</sup>, J.P. van Dijk<sup>1</sup>, A.M.A. Van Hoef<sup>1</sup>, M.H. Voorhuijzen<sup>1</sup>, H.J.M. Aarts<sup>1</sup> and E.J. Kok<sup>1</sup>

<sup>1</sup>RIKILT Institute of Food Safety, Wageningen, The Netherlands

\*E-mail: theo.prins@wur.nl Tel: +31 317 480398; Fax: +31 317 417717

Current EU regulations on the protection of products with certain characteristics (geographical indications and designations of origin) aim to ensure fair competition for producers and increased consumers' trust. Within the TRACE research project analytical methods are being developed to allow the maintenance of specific regulations for PGIs (products of protected geographical indication) and PDOs (products of designated origin). An example within the project is the PGI wheat variety Farro della Garfagnana. Aim of the research was to develop a method to establish the purity of Farro della Garfagnana DNA in complex cereal mixtures. The identification of DNA-markers for this specific wheat variety proved not possible, but the combined approach of padlock probe ligation and multiplex microarray detection can identify possible admixtures.

Within the TRACE project the protocol for padlock probe development and padlock probe ligation and microarray detection has been optimised in a step-by-step approach, to come to a robust method, that meets the required criteria with respect to specificity and sensitivity. A number of markers for possible admixtures, such as oat, rye, common wheats, traditional wheats and barley have been developed by different TRACE partners in addition to markers that had already been described in scientific literature. Also other markers were included for, soy, maize, sugar beet, rapeseed and rice. On the basis of these markers padlock probes have been developed and tested in singleplex and multiplex settings, in individual cereal samples as well as in different mixtures. Until now, a total of thirteen different species- and variety-specific probes have been developed.

One particular undesired 'contaminant' for Farro della Garfagnana is common bread wheat (*Triticum aestivum*), containing the BBA<sup>u</sup>A<sup>u</sup>DD genome. Since Farro harbours the BBA<sup>u</sup>A<sup>u</sup> genome, presence of the D-genome proves presence of bread wheat. Additionally, the presence of a traditional hulled grain wheat such as Farro della Garfagnana can be confirmed with the detection of the q version instead of the Q version of the Q-locus on the B genome of triticae. This locus is different for modern and traditional varieties and determines whether the wheat has hulled grains or is 'free-threshing'. Probes have been developed for both varieties based on an intronic SNP. The current detection limit of this multiplex method is at least 2.5% bread wheat in Farro. The latest results on the use of this array-based traceability tool for (cereal) specialty products will be presented.

#### References:

(1) Prins T.W. et al. (2008) Food Chem J doi:10.1016/j.foodchem.2008.10.085.



## **Parallel Session A**

### **Determination of fish origin by using 16S rDNA fingerprinting of bacterial communities by PCR-DGGE: an application on pangasius fish from Viet Nam**

**D. D. Le Nguyen, I. Métayer, G. Loiseau & D. Montet**

The determination of geographical origin is the demand of the traceability system of import-export food products. One hypothesis of tracing the source of the product is by analysing in a global way the bacterial communities of the food samples after their exportation. For this purpose, molecular techniques employing 16S rDNA profiles generated by PCR-DGGE were used to detect the variation in bacterial community structures of Pangasius fish from An Giang province, South Viet Nam harvested in different aquaculture farms and during two different seasons, the rainy season and the dry season. When the 16S rDNA profiles were analysed by multivariate analysis, distinct microbial communities were detected. The band profiles of the fish bacteria from different farms are different and other specific for each location and could be used as a barcode to certify the origin of the fish. When band profiles within the same location at different seasons were compared, we also observed the difference banding pattern for each season. Some common bands were noted throughout the season. These bands can be used as specific markers for this specific location. This method is a new traceability tool which provides fish products with a unique bar code and makes it possible to trace back the fish to their original location.

## Parallel Session A

### Male wagyu lineage origin in crossbred steers through Y chromosome DNA

**A. Rogberg-Muñoz<sup>1</sup>, M.V. Ripoli<sup>1</sup>, E.E. Villegas-Castagnasso<sup>1</sup>, D. Posik<sup>1</sup>, P. Peral-García<sup>1</sup> and G. Giovambattista<sup>1\*</sup>.**

<sup>1</sup>IGEVET, CCA La Plata, FCV-UNLP-CONICET. La Plata, Buenos Aires, Argentina

\*E-mail: ggiovam@fcv.unlp.edu.ar; Tel/Fax: +54 221 4211799

In the context of food safety and quality, certified origin food has had great advance in the last years. The importance of this kind of labeling is because of differentiating the product and also to assure some quality and safety parameters of the food. Wagyu meat it suits to this kind of treatment, not only because of the high quality recognized by the consumer, but for its exclusive fatty acid composition of it meat. Reported data evidence that the concentration of MUFA in Wagyu cattle and it crossbreds, is higher despite the amount of IMF in the carcass (1). We demonstrate that Wagyu crossbred can be DNA assigned, by paternity and breed assignment, using autosomal microsatellites data (2). For assure that the steers slaughtered commercially were Wagyu sired, we are developing a DNA test based on microsatellites of Y chromosome. Samples of Wagyu and four other breeds (Angus, Hereford, Nelore and Creole) were taken. The breeds were chosen to represent the F1 crossbreds that are been done. DNA was extracted from hair and five reported polymorphic microsatellites (INRA057, INRA 124, INRA126, INRA189, UMN0307) were genotyped (3, 4). We found 2 to 4 alleles in the studied markers and one of the haplotypes is exclusive of Wagyu breed. We are validating the test over more than 100 steers supposed to be Wagyu sired.

#### References:

- (1) Chung K.Y.; et al. (2007) J Anim Sci 85(2):380-7
- (2) Rogberg-Muñoz A.; et al. (2008) ISAG Proceedings 31:2067
- (3) Liu W.S.; et al. (2003) Cytogenet Genome Res 102(1-4):53-8
- (4) Liu W.S.; et al. (2002) Mamm Genome 13(6):320-6.

## Parallel Session B

### Traceability of food in practice - requirements for official control and food business operators

**B. Knispel\*, T. Neumeyer, N. Bauer and M. Bucher**

Bavarian Health and Food Safety Authority (LGL), Office Oberschleißheim, Germany

\*E-mail: birgit.knispel@lgl.bayern.de; Tel: +49 89 31560 147; Fax: +49 89 31560 349

Food business operators apply various systems to assure the traceability of their products. The legal basis for traceability of food in Europe is laid down in the EU Regulation (EC) No 178/2002. This regulation embodies the general principles and requirements of food law, establishing the European Food Safety Authority and procedures in matters of food safety.

The European law requires the food business operator to provide information only about the person from whom they have been supplied the goods and the person to whom they have delivered the goods. However, many systems allow more detailed information to be made available and go far beyond the European law. Especially the companies distributing nation- or worldwide have realized the importance of traceability. Trade associations like IFS or BRC have declared batch traceability to a matter of highest importance. Different examples of traceability systems in food business and practical possibilities to inspect the systems will be presented in the following report.

Inspecting and understanding the various systems of traceability remains a challenge to official control.

## Parallel Session B

### Traceability in official feed control

#### C. Thielen

District Government of Upper Bavaria, SG 56 Official Feed Control in Bavaria

Regulation (EC) No. 178/2002 of 28 January 2002 laying down the general principles and requirements of food law, establishing the European Food Safety Authority and laying down procedures in matters of food safety, Article 18, requires feed business operators:

to be able to identify from whom and to whom a product has been supplied;

to have systems and procedures in place that allow for this information to be made available to the competent Authorities upon their request.

The requirement relies on the “one step back”-“one step forward” approach and is specified in a guidance paper of the European Standing Committee on the Food Chain and Animal Health and furthermore in a national guidance paper on traceability in the feed sector.

Competent authority for official feed control in Bavaria is the District Government of Upper Bavaria. Whether the implemented traceability systems of feed business operators are sufficient, is subject of plan controls. Different systems which are applied by the feed business operators in Bavaria – from farmers to industrial feed manufacturers – are explained and evaluated in the presentation.

Also experiences from withdrawal actions and official investigations are exemplary shown and problems identified.

## Parallel Session B

### Traceability in the bulk grain supply chain

M. Thakur<sup>1</sup>, G. A. Mosher<sup>1</sup>, B. Brown<sup>1</sup>, G. S. Bennet<sup>1</sup>, H. E. Shepherd<sup>2</sup>, C. R. Hurburgh<sup>3\*</sup>

<sup>1</sup>Department of Agricultural and Biosystems Engineering, Iowa State University, Ames, IA, US

<sup>2</sup>Iowa Grain Quality Initiative, Iowa State University, Ames, IA, US

<sup>3</sup>Department of Agricultural and Biosystems Engineering and Department of Food Science and Human Nutrition, Iowa State University, Ames, IA, US

\*E-Mail [tatry@iastate.edu](mailto:tatry@iastate.edu)

This article summarizes recent efforts at Iowa State University to understand and improve bulk material traceability.

#### **Optimization of Internal and Chain Traceability**

Different lot-activities take place as the grain moves through the supply chain from the farm to the consumer. At an elevator, grain lots (inbound deliveries) are commingled to meet buyer specifications, and lot identity is not maintained. As a result, an outbound shipment to a customer can contain grain from many sources. In a food related emergency, it would be almost impossible to trace the problem source and to track other affected lots. This problem can be mitigated by an efficient internal record keeping system that would document all grain activities, including movement, aggregation, segregation, transformation and destruction. As a part of our traceability research, a relational database management system has been developed for internal traceability at a grain elevator. This system stores all the information related to grain lots and can be queried to retrieve information related to incoming and outgoing lots. This system can be used to trace back the source of a given lot and track forward information related to the shipped lots. Also, an optimization model has been developed for minimizing the traceability effort in terms of the quantity that would be recalled in case of food safety concern. A simulation based optimization technique is being developed to identify the critical points in the grain supply chain and propose changes in the storage and handling practices, with the goal of maximizing the profit from blending while minimizing the food safety risks.

#### **Mapping the milk supply chain**

Another project is examining the milk production supply chain. This case study uses internal and external traceability systems to track a processed milk product back to the grain that was fed at the dairy farm. This research will analyze the internal traceability systems of the dairy processor, dairy farmer, and feed producer and evaluate the intricacies of each system. The objective is to identify the gaps in the top level (dairy processor) of the external traceability system and provide quality control strategies that will improve the entire traceability system. The milk supply chain is a good grain-to-product case study because corn products account for over half of the feed ingredients in most rations. Corn is susceptible to aflatoxin which can be passed from the feed to milk in lactating animals. In addition, distillers dried grains and solubles (DDGS) are now being used as an ingredient in dairy cattle feed. If a contaminated lot of corn is processed for ethanol, the resulting DDGS will contain approximately three times the original amount of aflatoxin, or other problem substance.

#### **Role of Quality Management System**

Tracing in bulk products is a probability and elimination exercise. In an earlier study at a grain firm that was developing a quality management system, the accuracy of tracking improved steadily as operators became comfortable with basic recording procedures for handling operations. Accuracy is measured by ratio of potentially suspect product to the amount of contaminated product, or the traceability index. In over 50 trials, the ratio ranged from 1000 down to 10. Perfect traceability would be 1; unlikely to be achieved. The goal is to minimize the possibilities.

#### **Cost-Benefit Analysis**

Identity preserved (IP) grains are produced with a specific end use in mind such as for food, feed or pharmaceutical use. Likewise, some grains need to be isolated for particular end uses, such as buyers sensitive to biotechnology. These IP grains need to be segregated in order to preserve their identity. Traceability systems play a very important role in maintaining an efficient segregation system. It is very important to determine the profitability associated with segregation of grain for different purity levels. A cost-benefit analysis of an on-farm traceability system was conducted to determine if a particular IP crop at a specific purity level would be profitable to grow. The price per bushel increased as the purity level requirements increased. Farm management practices have a tremendous impact on expenses and ability to meet specific purity levels. A detailed cost and operational analysis protocol was designed to obtain meaningful results in cost benefit analysis.

#### **Decision making and Risk analysis**

The implementation of traceability systems often occurs through the use of quality management systems or International Organization of Standards (ISO) processes. This project will quantify factors involved with employee decisions concerning quality within the country elevator environment, identify needs for improved standard operating procedures and educational intervention. The risk analysis examines selected operations that affect grain quality from seed purchase to end user delivery using fault tree analysis. Fault tree analysis identifies contributing factors in complex systems, illustrates interrelationships of the causes of specified events, and quantifies probabilities of occurrence for each pathway of events. Those with the highest probabilities for negative consequences are targeted for educational intervention or other counter-measures. Although each component varies in focus, the underlying concept is to provide data to guide educational efforts in traceability for producers, processors, handlers and other actors in the agricultural supply chain.

#### **Final thoughts**

Various traceability issues in the bulk grain supply chain are being addressed by our research group. The main goal of the entire effort is to develop a methodology for implementation, quantification and optimization of traceability systems in the bulk grain supply chain for improving the food safety. From results at this point, improvement of traceability with supporting quality management systems has significant potential to increase profits through operational efficiency.

## **Parallel Session B**

### **Traceability and the consumer**

#### **G. Chryssochoidis**

Abstract wasn't available at the time of printing.

## Parallel Session B

### Milestones towards global traceability

H. Lehr<sup>1\*</sup>

<sup>1</sup>FoodReg, Barcelona, Spain

\*E-mail: heiner.lehr@foodreg.com; Tel: +34 93 2801555; Fax: +34 93 280 30 15

Food traceability worldwide has received a lot of attention. There is a clear understanding (enforced by laws in some regions like the European Union) that traceability is a basic food safety requirement. Some countries have even initiated isolated effort to implement traceability on a national level. The current development, however, seems limited in two aspects

In spite of a general understanding that food traceability can only be treated globally, there is no concerted international effort to implement the means for such an effort.

Secondly, the food industry in itself does not seem to pick up traceability easily. Traceability has been labelled as complicated and expensive and its economic advantages are not clear enough for the food businesses.

TRACE is trying to smooth the barrier of adoption significantly by providing simple to understand guidance for the food industry and simple to implement protocols for the exchange of data along the supply chain.

However, other measures might be at order to improve the production of traceable food world wide.

## Parallel Session B

### Traceability of food and consequences for isotope signatures in human tissues

C. Lehn<sup>1</sup>, E. Mützel<sup>1</sup>, A. Rossmann<sup>2</sup>, S. Rummel<sup>3</sup>, S. Hölzl<sup>3</sup>, P. Horn<sup>3</sup>

<sup>1</sup>Institute for Legal Medicine, University of Munich

<sup>2</sup>Isolab GmbH, Laboratory for Stable Isotopes, Schweitenkirchen, Germany

<sup>3</sup>Bavarian State Collection of Palaeontology and Geology, Munich, Germany

Stable isotopes at natural abundance levels have been used extensively in life sciences, archaeology and anthropology, primarily in studies on animal and human migration. They are also useful in several areas of forensics, e.g. for origin determination of humans, animals, plants, and objects in criminalistics and criminology.

Since 2002 the Institute for Legal Medicine in Munich, in collaboration with the Bavarian State Collection of Palaeontology and Geology, Munich, and Isolab GmbH, Laboratory for Stable Isotopes, Schweitenkirchen, has been using isotope signatures for forensic applications.

Official requests from police and prosecutors are mostly related to unidentified corpses who were often murder victims. Results of stable isotope investigations of bio- (H, O, C, N, S) and geoelements (Sr, Pb) on human tissues provide clues for further inquiries by criminal investigators.

The above mentioned elements are composed of isotopes which occur in nature in variable isotope abundance ratios (I.R.) in air, water, soil and dust and enter body tissues via nutrition, drinks and environmental aerosols.

Since isotope ratios are important tracers for the origin and authenticity of foodstuff and other commodities, one can also draw conclusions concerning human nutritional habits and whereabouts. Depending on the geographical and geological situation, plants and animals show I.R., which ultimately will be laid down and conserved in human tissues.

Different tissues, such as teeth, bones, hair, nails or blood, frequently show varying I.R., depending on the tissues' growth times and rebuilding rates so that one has isotopic information from childhood to the last days before death.

In order to have at hand authentic reference data for humans at the Bavarian State Collection of Palaeontology and Geology and at the Institute for Legal Medicine, a continuously growing data bank has been established. It is mainly aimed at gathering data of human hair of known provenance, and today comprises about 400 samples from all over the world. Multivariate analysis is applied for regional discrimination of hair samples by the four variables H-, C-, N- and S-I.R..

Due to experimental problems with human tissues, stable isotope analysis of wild boar tissues holds information about regional food and water, and also seasonal variations.

For working out inter-tissue differences in I.R. concerning the diet history of an individual it is planned to conduct relevant studies with pigs, raised and fed under controlled conditions, in collaboration with School of Agriculture, Food Science and Veterinary Medicine, University College Dublin.



## Parallel Session B

### Simulated recalls of meat products, fruit and vegetables originating in the European Economic Community – preliminary results

K. A-M. Donnelly<sup>1\*</sup> and K. M. Karlsen<sup>1</sup>

<sup>1</sup>Nofima- Norwegian Institute of Food Fisheries and Aquaculture Research, Tromsø, Norway.

\*E-mail: kathryn.donnelly@nofima.no

Much of the food that reaches the modern consumers plate is sourced globally. Production and distribution patterns have become much more complex than was common even 50 years ago and consumer preferences have evolved to include specialist foods and foods out of season. At the same time the type of food related health incidents, from BSE to Dioxins, and the number are growing. When these two factors are combined the need for greater transparency in food supply chains becomes apparent. Creating this transparency requires the ability to trace and track ingredients in food stuff rapidly and precisely.

In order to assess the traceability of food products in different food sectors a simulated recall was carried out. Twelve different products were chosen and then an attempt was made to trace the specific ingredients back through the supply chain.

The products were purchased in Norway but just under half of them originated outside of Norway but within the EU. It was possible to specifically trace 80% of the chosen meat products back through the chain and 50% of the fruit and vegetable products. The estimated time taken to trace such information in the event an incident varied from less than 2 hours to less than 2 days for the fruit and vegetables. The traceable meat products were in each instance traced back to a set of known animals.

The food industry sectors which handle fruit, vegetables and meat are structurally very different. The reason that the fruit and vegetables often couldn't be traced was merely the lack of labelling on the fruit or the lack of the box which the fruit had arrived in. The problem of the boxes containing ID information was highlighted when dealing with the fruit. In future work it would be interesting to attempt to track forward through the supply chain.

*The authors would like to acknowledge the Norwegian national traceability project 'eSpring' for funding for this work.*

## Parallel Session B

### Traceability in cattle in Germany

R. Carmanns<sup>1\*</sup>

<sup>1</sup>Project Manager of the German Central Database for Cattle, Pigs, Sheep and Goats.  
Bavarian State Ministry of Agriculture and Forestry, Ludwigstr. 2, 80539 München, Germany.

\*E-mail: Richard.Carmanns@stmlf.bayern.de

As a result of the BSE crises in 1996 an improved identification and registration (I&R) system for cattle was introduced to regain consumer confidence in beef through traceability and transparency on all levels of production and marketing. Basis of the improved I&R system is a unique identification number for each animal and a unique registration number for each holding. Under the I&R system all cattle holders including traders, auctions, collecting points and slaughterhouses have to report. To ensure traceability all changes in a holding must be reported within 7 days to a central database (CDB). Transparency as well as traceability on all levels of production is achieved through a direct internet access to the CDB.

Ear tags and cattle passports are issued by the competent authority. Issued ear tag numbers are allocated to a holding number in the CDB. The issued ear tags can only be used by this specific holding for the registration of birth. This ensures the tracing back to the holding, to which the ear tag was issued.

There are about 12.9 Mio. cattle and 225 000 holdings in Germany. The average number of reports per day is about 100 000. Nearly 90 % of all reports are sent via internet. Cattle holders with no internet access can report via postcard or telephone (IVR).

A good data quality is the basis for a comprehensive traceability on all levels of production. A two level quality control system with a priori and a posteriori checks is implemented in the CDB to ensure this. In addition a minimum of 5 % of all holdings are selected on the basis of a risk analysis for on the spot controls every year to verify the correctness of the information in the database. Basis for the on the spot control is a list of the registered animals in the CDB. This list is retrieved by the inspector via internet from the CDB before the control starts.

The results of the on the spot controls are also stored in the CDB. In case of non compliance with regulations the subsidy payments are reduced for this holding.

The easy access via internet to the CDB in combination with a high percentage of internet reporting by cattle holders is the basis for the comprehensive traceability in cattle on all levels of production in Germany. The high quality of the stored information is the result of the internal quality management system and on the spot controls. For farmers the CDB is a management tool in respect to disease control and to farm aid schemes. The veterinary service can use the CDB for disease control on an individual animal basis.

B.

**Internal Posters**

# IP1

## Soil-grape traceability using a regression model based on trace elements

D. Bertoldi<sup>1,3\*</sup>, R. Larcher<sup>1</sup>, G. Nicolini<sup>1</sup>, M. Bertamini<sup>1</sup>, S. Otto<sup>2</sup>, G. Concheri<sup>3</sup>

<sup>1</sup>FEM-IASMA Research Centre, via E. Mach 1, 38100 San Michele all'Adige (TN), Italy

<sup>2</sup>Institute of Agro-environmental and Forest Biology (IBAF-CNR), Viale dell'Università, 16. 35020. Legnaro (PD) Italy

<sup>3</sup>Agricultural Biotechnology Department, University of Padova. Viale dell'Università, 16. 35020. Legnaro (PD) Italy

\*E-mail: daniela.bertoldi@iasma.it; Tel: +39 0463 615138; Fax: +39 0463615288

In the last 30 years several studies have been carried out on the utility of mineral element analysis for traceability studies of different food. In these, particular attention has been paid to determination of the geographical origin of wine (1, 3, 6, 7). The utility of trace element determination in traceability studies is based on the assumption that the mineral composition of grapes reflects to some extent that of the soil in which the vine grows, but this theory has rarely been verified (2, 4, 5).

The aim of this study was to determine whether the mineral composition of grapes could be predicted from the composition of the soil of origin. For this purpose a model was constructed, linking grape and soil properties (analysis dataset) using specific Regression Analysis. External validation was performed using an independent dataset (validation dataset).

Soil and Chardonnay grape samples in the analysis dataset were collected from 11 vineyards in Trentino (Northern Italy) at harvest time in 2007. For the validation dataset, grape samples previously collected in 2006 from 7 out of the 11 vineyards were used.

The dataset included 3 categories of soil: acid or subacid soil (pH < 6.7; 3 samples), alkaline and moderately calcareous soil (pH > 7.3; total CaCO<sub>3</sub> < 250 mg/kg; 4 samples) and alkaline and calcareous soil (pH > 7.3; total CaCO<sub>3</sub> > 500 mg/kg; 4 samples). The soil samples were air-dried and the < 2 mm fractions were extracted with ammonium acetate 1M pH = 7 solution (SSIR 42 method 5A8). The grapes were accurately washed with a HNO<sub>3</sub> 1% solution, homogenised and acid digested in a microwave oven (HNO<sub>3</sub>; max. temperature 210°C). All the samples were analysed using ICP-MS for the determination of 55 mineral elements.

The content of 18 elements in the soil extracts and in the respective grapes was significantly correlated. For As, Ba, Be, Ca, Cs, Eu, Gd, La, Sb and Y Pearson's r was higher than 0.74 and linear regression between the content in soils and grapes was calculated. For each element the expected values of soils estimated from the 7 grape samples of unknown origin were compared with the average composition of the 3 soil categories (acid, moderately calcareous and calcareous) and then with the mineral content of each soil within the category. For the comparison, the sum of deviations between the natural logarithm of the expected and the measured values for each element was calculated. For the 4 rare earth elements (Eu, Gd, La, Y) a mean contribution was considered. The soil with the least sum of deviations can be considered the most similar to the soil estimated by the model and thus the most likely soil of provenance.

All the 7 grape samples were correctly classified for the soil categories and 6 were assigned to the vineyard of origin.

(1) Larcher R.; Nicolini G. (2008) Hyphenated techniques in grape and wine chemistry. Wiley, Chichester, England

(2) Oddone M.; Robotti E.; Marengo E.; Baldizzone M.; Aceto M. (2007) Proceeding of Italian Congress "Chimica degli Alimenti", Editrice Taro, Italia, pp. 573-577

(3) Day M.P.; Zhang B.L.; Martin G.J. (2005) Am J Enol Vitic, 45(1):79-85

(4) Almeida C.M.R.; Vasconcelos M.T.S.D. (2004) J Agric Food Chem, 51:4788-4798

(5) Orescanin V.; Katunar A.; Kutle A.; Valkovic V. (2003) J Trace Microprobe Tech, 21(1):171-180

(6) Baxter M-J.; Crews H.M.; Dennis M.J.; Goodal I.; Anderson D. (1997) Food Chem., 60(3):443-450

(7) Siegmund H.; Baechmann K. (1978) Z Lebensm Unters Forsch, 166:298-303

# IP2

## Traceability of tomato and derivatives along the production chain

L. Bontempo<sup>1,2\*</sup>, F. Camin<sup>1</sup>, R. Larcher<sup>1</sup>, L. Manzocco<sup>2</sup>, and G. Nicolini<sup>1</sup>

<sup>1</sup>FEM-IASMA, Research Centre, via E. Mach 1, 38100 San Michele all'Adige (TN), Italy

<sup>2</sup>Università degli Studi di Udine, Dipartimento di Scienze degli Alimenti, via Sondrio 2, 33100 Udine (UD), Italy

\*E-mail: luana.bontempo@iasma.it; Tel: +39 461 615138; Fax: +39 461 615288

Several papers have been written about tomatoes but the aims of these studies have mainly focused on agricultural and production practices with the purpose of preserving or improving the nutritional characteristics of the final product (1, 2, 3, 4, 5, 6). To date, no studies have aimed to identify possible markers indicating the origin of tomatoes and derivatives along the production chain. However, since January 2008 Italian law has established that the origin of tomatoes must be indicated on the label (D.M. of 17 February 2006), although it does not specify which parameters must be used.

The aim of this study was to verify if and how the parameters considered vary along the production chain, in order to evaluate their usefulness as indicators for the traceability of tomatoes and derivatives. We considered tomatoes, juice, 'passata' and pastes collected along the whole production line in different factories. All the samples were subjected to analysis of the  $^{18}\text{O}/^{16}\text{O}$  of juice water (7),  $^{13}\text{C}/^{12}\text{C}$ ,  $^{15}\text{N}/^{14}\text{N}$  and mineral content of the raw material. Analysis of  $\delta^{18}\text{O}$  was performed using an Isotopic Ratio Mass Spectrometer (IRMS) interfaced with a  $\text{CO}_2$  equilibration system, according to the ENV 12141 method, while determination of the  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  of bulk was carried out using an IRMS equipped with an elemental analyzer. Analysis of mineral content was performed using an Inductively Coupled Plasma Mass Spectrometer (ICP-MS) following mineralisation of the sample using a microwave oven.

In this paper we present initial data relating to the composition of tomatoes, tomato juice, 'passata' and tomato paste along some individual production lines. The first results seem to indicate that  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  and the content of 'non technological' elements do not vary significantly along the production chain.

This research was funded by the Italian Ministry of Agricultural, Food and Forestry Policy.

### References:

- Calvo M.M., Santa-Maria G. (2008) Food Chem. 107: 1365  
Hernandez-Suarez; et al. (2008) Food Chem. 106: 1046  
Chagvardieff P.; et al. (1997) Adv. Space Res. 20 (10): 1971  
Choi J.H.; et al. (1997) Scientia Horticulturae 70: 293  
Davoodi M.G.; et al. (2007) LWT 40: 1832  
Gil M.I.; et al. (2002) Postharvest Biol Tec 25: 199  
Trifirò A.; et al. (2007) Industria Conserve 82 (3): 239

# IP3

## Authentication of the trappist beers using advanced mass spectrometric techniques

T. Cajka<sup>1\*</sup>, J. Hajslova<sup>1</sup>, K. Riddellova and L. Vaclavik<sup>1</sup>

<sup>1</sup>Institute of Chemical Technology, Prague, Department of Food Chemistry and Analysis, Technicka 3, 166 28 Prague 6, Czech Republic

\*E-mail: tomas.cajka@vscht.cz; Tel: +420 220 443 142; Fax: +420 220 443 184

A Trappist beer is a beer brewed by or under control of Trappist monks. Today, seven monasteries produce this type of beer (six in Belgium and one in The Netherlands). Only these breweries are authorised to label their beers with the “Authentic Trappist Product” logo that indicates a compliance to various rules edicted by the International Trappist Association [1].

For the analysis of beer samples, two different profiling strategies were selected:

- (i) Head-space solid-phase microextraction coupled to gas chromatography–low-resolution time-of-flight mass spectrometry (HS-SPME–GC–LRTOFMS)
- (ii) Direct analysis in real time (DART) ion source coupled to high-resolution time-of-flight mass spectrometry (DART–HRTOFMS)

While HS-SPME–GC–LRTOFMS approach is considered to be a “gold standard” for the profiling of volatiles (including those in beer samples), the latter approach (DART–HRTOFMS), introduced into the laboratory use only recently, represents a novel analytical strategy enabling rapid examination of product composition [2].

In this presentation, a comparison of both approaches will be given with the aim to authenticate Trappist beers, specifically Rochefort 8 beer.

### References:

(1) [http://en.wikipedia.org/wiki/Trappist\\_beer](http://en.wikipedia.org/wiki/Trappist_beer), Accessed on December 31, 2008.

(2) Cajka T., Vaclavik L, Riddellova K., Hajslova J., *LC GC Europe* 21(5) (2008) 250–256.

*Acknowledgement: This work has been carried out with support from the European Commission through the Sixth Framework Programme under the Food Quality and Safety Priority (Contract no. CT-2005-006942, TRACE).*

# IP4

**Isotopic and mineral data for tracing the origin of European olive oils****F. Camin<sup>1\*</sup>, R. Larcher<sup>1</sup>, G. Nicolini<sup>1</sup>, M. Perini<sup>1</sup>, L. Bontempo<sup>1</sup>, D. Bertoldi<sup>1</sup>, K. Heinrich<sup>2</sup>, M. Horacek<sup>3</sup>, C. Schlicht<sup>4</sup>, A. Schellenberg<sup>4</sup>, F. Thomas<sup>5</sup>, S. Voerkelius<sup>6</sup>, A. Rossmann<sup>7</sup>, S. Kelly<sup>8</sup>**<sup>1</sup>FEM-IASMA Research Centre, San Michele all'Adige (TN), Italy<sup>2</sup>Central Science Laboratory, York, United Kingdom<sup>3</sup>ARC Seibersdorf research GmbH, Seibersdorf, Austria<sup>4</sup>Bavarian Health and Food Safety Authority, Oberschleißheim, Germany<sup>5</sup>Eurofins Scientific, Nantes, France<sup>6</sup>Hydroisotope, Schweitenkirchen, Germany<sup>7</sup>Isolab GmbH, Schweitenkirchen, Germany<sup>8</sup>Institute of Food Research, Norwich, United Kingdom

\*E-mail: Federica.camin@iasma.it; Tel: +39 0461 615149; Fax: +39 0461 615288

Within the framework of the TRACE project, 267 European authentic olive oil samples were collected at 8 sites (Italy: Trentino, Tuscany, Sicily; France: Carpentras; Greece: Chalkidiki, Lakonia; Portugal: Algarve; Spain: Barcelona) during harvesting in 2005 and 2006. Analysis of stable isotope ratios <sup>13</sup>C/<sup>12</sup>C ( $\delta^{13}C$ ), <sup>18</sup>O/<sup>16</sup>O ( $\delta^{18}O$ ) and D/H ( $\delta D$ ) was performed on bulk olive oils, using Isotopic Ratio Mass Spectrometers (IRMS) connected to an Elemental Analyser and/or Pyrolyser. The content of 30 elements was determined using an Inductively Coupled Plasma–Mass Spectrometer (ICP-MS) after ultrasound acid extraction (1).

$\delta^{13}C$ ,  $\delta^{18}O$  and  $\delta D$  changed according to climatic conditions and the latitude of the production area, with some exceptions probably linked to olive cultivars. The  $\delta D$  of bulk oils correlated with  $\delta^{18}O$  on a global scale, mirroring the phenomena observed in water (2). Significant differences were found in the isotopic composition of olive oils produced in different areas, differences that were not always evident in surface water collected at the same sites. The content of Na, Mg, Al, K, Ca, V, Mn, Co, Ni, Cu, Zn, Ga, Rb, Sr, Cs, Ba, La, Eu, U made it possible to discriminate ( $p < 0.05$ ) between olive oils of 3 different geological origins (shale/clay, acid magmatic and limestone). The same elements also allowed discrimination between surface waters sampled at the corresponding geological sites.

The use of both isotopic and mineral data made it possible to improve geographical discrimination of olive oils.

(1) Camin F.; Larcher R.; Perini M.; Bontempo L.; Bertoldi D.; Gagliano G.; Nicolini G.; Versini G. (2008) Food Chem. 2008 (in press). DOI:10.1016/j.foodchem.2008.04.059

(2) Clark I., Fritz P.; (1997). Environmental Isotopes in Hydrogeology. Lewis Publishers, New York.

**Acknowledgements**

This work was funded by the European Commission, under the FP6 Food Quality and Safety Priority, within the framework of the Integrated Project TRACE – 006942 – entitled “Tracing Food Commodities in Europe”. The information contained in this paper reflects the authors' views; the European Commission is not liable for any use of the information contained therein.

# IP5

## FT-Raman and chemometrics for the authentication of trappist beers

J. A. Fernández Pierna\*, O. Abbas, P. Dardenne, V. Baeten

Walloon Agricultural Research Centre (CRA-W), Quality of Agricultural Products Department  
24, Chaussée de Namur; 5030 Gembloux, Belgium

\*E-mail: fernandez@cra.wallonie.be; Tel: +32 81620352; Fax: +32 81620388

A Trappist beer generally is a beer of high fermentation brewed for or under the control of the Trappist monks according to criteria defined by the private association "Trappist International Association". In order to be able to claim the denomination *Authentic trappist product* and use the logo of the association, the following criteria must be respected:

- The product must be made in an abbey of the Trappist order;
- The product must be made under the direct or indirect control of the monks;
- The major part of the financial benefits that come from selling the product must be dedicated to actions having a social character.

Today in the world, only seven Trappist beers exist with the logo *Authentic trappist product*. Six of these beers are Belgian (Chimay, Orval, Rochefort, Achel, Westmalle and Westvleteren) and one is Dutch (La Trappe).

Within the framework of the TRACE project ([www.trace.eu.org](http://www.trace.eu.org)) we have been working on the authentication of Trappist beers using Fourier Transform Raman spectroscopy (FT-Raman) and different chemometric tools. For this study, a complete experimental design has been performed in order to include as much as possible of the variability that can be found in the Trappist and non Trappist beers.

A database including FT-Raman spectra from 124 different beers was studied using, in the first stage, principal component analysis (PCA). This method tries to find natural clusters in the data as well as outlying samples. Different PCA models have been constructed according to degree of alcohol and colour as well as their membership of the group of Trappist beers, specially the Rochefort beer.

In a second stage, partial least squares discriminant analysis (PLS-DA) models have been constructed in order to discriminate (a) Trappist beers from the rest of the beers, (b) Rochefort from the other beers and (c) Rochefort 8 from the rest. For all these models, the results are expressed in terms of correct classification rates as well as false positive and false negative samples. In all the cases studied, reasonable classification rates are obtained showing the ability of FT-Raman and chemometrics to authenticate beers.



# IP6

## Trace elements as markers for geographical regions: examples for anthropogenic influence and geological background

H. Fröschl\*, G. Heiss, P. Spindler, R. Hamid and B. Wimmer

Austrian Research Centers GmbH - ARC, A-2444 Seibersdorf, Austria

\*E-mail: heinz.froeschl@arcs.ac.at; Tel: +43 50550 - 3593; Fax: +43 50550 - 3590

In the framework of the European project TRACE – “Tracing Food Commodities in Europe” samples of wheat, honey, lamb, olive oil and surface water were collected in 21 test areas located in 12 countries around Europe. Additional in each test region geochemical investigations were performed on the fine fraction (<2 mm) of soil samples.

The total chemical composition of 250 soil samples (at every test site 6 profiles of topsoil and subsoil samples) was determined by a combination of X-ray fluorescence (XRF) and inductively coupled plasma mass spectrometry (ICP-MS) after wet acid digestion of the samples.

Soil extractions of soil samples (in total 2188) with 1 M NH<sub>4</sub>NO<sub>3</sub>-solution were performed to determine readily soluble and plant available trace elements.

The trace elemental composition is characterized by the geological/lithological background (example 1) and can be influenced by anthropogenic activities (-> example 2).

### Example 1:

Caesium (Cs) as relatively rare element acts as applicable tracer to differentiate between the different test areas (mainly concentrated in acidic igneous rocks). A strong correlation between total contents and soil extracts with effects on Cs in wheat can be demonstrated.

### Example 2:

Elevated concentrations of cadmium (Cd) in topsoil, soil extract and also in wheat in the test area in Poland are due to contamination (initiated probable by emissions from coal-burning power plants).

# IP7

## Traceability of food ingredients: molecular methods for GMO detection

L. Gerdes<sup>1\*</sup> and S. Pecoraro<sup>1</sup>

<sup>1</sup>Bavarian Health and Food Safety Authority (LGL), Office Oberschleißheim, Germany

\*E-mail: lars.gerdes@lgl.bayern.de; Tel: +49 89 31560875; Fax: +49 89 31560458

Genetically modified crop plants are commercially grown in at least 23 countries worldwide. The global acreage of genetically modified organisms (GMO) has steadily increased since 1996 up to around 110 million hectares in 2007. Four plants account for most of the grown GMO crops: soybean, maize, cotton, and canola (1).

The European Union established with regulation (EC) No 1830/2003 on traceability a legal framework to inform customers through the compulsory labelling of GMOs and products produced from GMOs (2).

The task of the official food and feed control is to monitor the compliance with the regulation. Beside the control of the producer's documents for traceability, food and feed is routinely analysed for ingredients from approved and non-approved GMOs.

Analysis for constituents of GMO crops is normally based on the detection of certain DNA sequences. With PCR techniques defined parts of DNA can be proliferated and subsequently or immediately (in real-time) detected. Real-time PCR is the method of choice because of its high specificity, its closed amplification system that minimises carryover risks, and the possibility for quantification of GMO contents.

Since 2004 the number of GMO food and feed applications submitted to the European Food Safety Authority (EFSA) grew rapidly. At present there are more than 60 applications for authorisation pending (3). For that reason it is reasonable to assume that the number of approved GMO events in the EU will rise in the near future. This confronts the official food and feed control with a growing number of possible events that have routinely to be tested for. There are several possible ways to deal with this problem. For example the chip-based approach that combines PCR and subsequent detection by hybridisation with DNA probes on a chip thus enabling multiple GMO events to be analysed simultaneously. The LGL is currently developing a microtitre plate based parallel analysis real-time PCR format in order to efficiently detect multiple events at the same time. This real-time PCR approach has several advantages over the chip-based method. The data from the parallel real-time format is reliable through the use of validated methods (e.g. the by the Community Reference Laboratory, CRL). The parallel real-time format is open for additional test reactions that can easily be included in the microtitre plate based format.

### References:

(1) James C. (2007) ISAAA Report on Global Status of Biotech/GM Crops, <http://www.isaaa.org/resources/publications/briefs/37/pptslides/default.html>

(2) EU, <http://europa.eu/scadplus/leg/en/lvb/l21170.htm>

(3) EFSA, <http://registerofquestions.efsa.europa.eu/roqFrontend/questionsList.jsf>

# IP8

**Multi-element (h C N S) stable isotope ratio characteristics of honey from different European regions****A. Schellenberg<sup>1</sup>, S. Chmielus<sup>1</sup>, C. Schlicht<sup>1</sup>, F. Camin<sup>2</sup>, M. Perini<sup>2</sup>, L. Bontempo<sup>2</sup>, K. Heinrich<sup>3</sup>, S.D. Kelly<sup>4</sup>, A. Rossmann<sup>5</sup>, F. Thomas<sup>6</sup>, E. Jamin<sup>6</sup>, M. Horacek<sup>7</sup>**<sup>1</sup>Bavarian Health and Food Safety Authority, Oberschleißheim, Germany<sup>2</sup>FEM-IASMA Research Centre, San Michele all'Adige, Trento, Italy<sup>3</sup>Central Science Laboratory, York, United Kingdom<sup>4</sup>Institute of Food Research, Norwich, United Kingdom<sup>5</sup>Isolab GmbH, Schweitenkirchen, Germany<sup>6</sup>EUROFINS Scientific, Nantes, France<sup>7</sup>ARC Seibersdorf research GmbH, Seibersdorf, Austria

Honey as a food of natural origin has a special status among consumers. Honey samples from 20 European regions were obtained to establish whether differences in the isotopic composition of the stable isotopes of hydrogen, carbon, nitrogen and sulphur (H C N S) were influenced by geographical origin. In this study we demonstrate that a correlation between the isotopic composition (H C N S) of the honey protein and geographic origin does exist.

The bio-elements hydrogen, carbon and sulphur, but also nitrogen, in the protein fraction of the honeys proved to be important parameters for the geographic origin assignment of a sample. We have demonstrated that not only hydrogen isotopes in honey protein are correlated to precipitation and climate, but also carbon isotopes are influenced by these parameters as well. The sulphur stable isotope composition of the honey protein is clearly influenced by the geology of the rock underlying the soil in which the flora grew and from which the bees foraged nectar and pollen. Sulphur from the seaspray is important for certain regions, too. It was anticipated that nitrogen would also be pedological influenced, but because of different biosynthesis pathway of different plant species (in particular rhizobial nitrogen fixation), habitat influences, or possible fertilizer application, the  $\delta^{15}\text{N}\text{‰}$  values have to be interpreted very carefully.

Nevertheless the four stable isotope ratios considered here have already correctly classified more than 70 % of honey samples for seven of the chosen test areas. The main reasons for poor prediction ability are similar geological and climatic conditions as well as natural variability.

# IP9

## Multi-element (h C N S) stable isotope ratio characteristics of whitefish (*coregonus species*) from different lakes in Bavaria

A. Schellenberg<sup>1\*</sup>, C. Schlicht<sup>1</sup>, P. Resch<sup>1</sup>, E. Brunner<sup>1</sup>

<sup>1</sup>Bavarian Health and Food Safety Authority, Oberschleißheim, Germany

\*E-mail: antje.schellenberg@lgl.bayern.de; Tel: +49 89 31560586; Fax: +49 89 31560457

The use of multielement-isotopic ratio analyses to provide information on the provenance of foods is gaining wider acceptance. Multi-element (H C N S) stable isotope ratio analysis was tested for its suitability as a means for geographical provenance assignment of whitefish (*Coregonus species*) from several Bavarian lakes.

The whitefish is a valuable fish for human consumption and the most important fish for southern Bavarian lake fishery. It prefers cold, clear water with high oxygen content and can be found in deep alpine lakes. Whitefish samples from Lake Constance and other lakes from the alpine region (Pilsensee, Woerthsee, Starnberger See, Chiemsee, Tegernsee and Kochelsee) were collected during fishing season (April – October). The defatted dry matter was found to be a suitable sample for the light elements stable isotope ratio analysis (SIRA). The meat of the common whitefish was dried with the aid of lyophilisation. The dry meat is then homogenized and extracted with petrol ether. When all remaining solvent is evaporated, the fat free dry mass is used for the measurements.

Significant differences were observed between the multi-element stable isotope ratios of whitefish samples from different lakes. Carbon and nitrogen isotopic ratios were influenced by the natural food supply and climate. Sulfur isotopic ratios were influenced by geographical location and surface geology of the region. However, more sophisticated evaluation of the data using multivariate methods, such as canonical discriminant analysis achieved in excess of 80% correct classification. Variables used in the classification were  $\delta^{34}\text{S}\text{‰}$  vs. V-CDT,  $\delta^{15}\text{N}\text{‰}$  vs. Air,  $\delta^{13}\text{C}\text{‰}$  vs. V-PDB,  $\delta^2\text{H}\text{‰}$  vs. V-SMOW. Our preliminary observations on the use of multielement stable isotope analyses to detect the geographic origin of whitefish are so encouraging, that further investigations with other regions are proposed.

# IP10

## Overview of TRACE project TECHNOLOGY TRANSFER GROUP activities: 2008

**WP10 team (M. Tomaniová\*, J. Hajšlová, M. Kostelanská)**

Institute of Chemical Technology Prague, Department of Food Chemistry and Analysis, Technická 3, Prague 6, 166 28, Czech Republic

\*E-mail: monika.tomaniova@vscht.cz

A comprehensive training network program established within the TRACE project is aimed at providing and overseeing the knowledge transfer through short-term training activities and workshops. Other activities of Technology transfer group (TTG) are focused on co-organization of TRACE conferences and meetings of independent Advisory Board.

### **Training network.**

4 short-term training activities for 6 trainees in 2 training areas (Light Isotopic Techniques and Rapid / Profiling methods) were organized in the year 2008.

The evaluation of effectiveness of training activities is continuously carried out both via questionnaires on training session's set-up by trainees and trainers and via questionnaires and short reports on transfer of obtained knowledge into trainee's institutions / laboratories which are submitted by trainees 6 months after completion of the training.

Training documents webpage at the TRACE Intranet is continuously updated to inform participants of short-term training activities on all issues associated with the ongoing training program (<http://intranet.trace.eu.org/Training%20doc/Forms/AllItems.aspx>).

Announcement about the training sessions, information on trainees' and trainers' institutions together with completed questionnaires and trainees' reports are available on the TRACE public website (<http://www.trace.eu.org/events/ts/index.php>).

### **Workshops.**

The 4<sup>th</sup> TRACE training workshop focused on "TraceFood Framework and TraceCore XML - International standard for electronic exchange of product information in the food supply chain" was organized in cooperation with TSG on occasion of the 4<sup>th</sup> TRACE Annual meeting and conference, 23 April 2008, Torremolinos, Spain. The objective of this workshop was to present TraceFood and TraceCore XML, to enable more users, companies and projects to participate in the process and to encourage widespread development of functionality for standardized sending and receiving of this type of message.

The 5<sup>th</sup> TRACE dissemination workshop aimed at "Molecular biology methods for traceability purposes", organized in cooperation with TRACE WP3, was held on December 18 – 19, 2008 at Federal Institute for Risk Assessment (BfR), Germany. Three topics focused on DNA-analytical techniques, protein-based methods and application of molecular biology methods for foods were covered within the workshop program.

Workshop lectures are available on the workshops' website at <http://www.trace.eu.org/ws/>.

### **Conference.**

The 4<sup>th</sup> TRACE Annual meeting and conference "Lost without TRACE: New approaches for tracing the origin of food" was organised in cooperation with one of project partners, NOFIMA, Norway, 23 – 25 April 2008, Torremolinos, Spain.

Lectures and posters presented during the conference are available on the TRACE public website at <http://www.trace.eu.org/meetings>.

### **Advisory Board meeting.**

The 4<sup>th</sup> meeting of independent Advisory Board was organized on occasion of TRACE Annual meeting in Torremolinos, 23 April 2008. An expert advices and comments aimed at maximization of the relevance and applicability of the outputs of the TRACE project were summarized in the Meeting minutes. In addition, attendance of Observers at the 4<sup>th</sup> TRACE Annual meeting was coordinated within this activity.

*Acknowledgement: This work has been carried out with support from the European Commission through the Sixth Framework Programme under the Food Quality and Safety Priority (Contract no. CT-2005-006942, TRACE).*



**External Posters**

# EP1

**Identification of the geographic origin of pumpkin seed oil****D. Bandoniene<sup>1\*</sup>, D. Joebstl<sup>1</sup>, T. Meisel<sup>1</sup>, S. Chatzistathis<sup>2</sup>**<sup>1</sup>General and Analytical Chemistry, Montanuniversität Leoben, Leoben, Austria<sup>2</sup>Laboratory of Analytical Chemistry, Department of Chemistry, Aristotle University of Thessaloniki, Thessaloniki, Greece

\*E-mail: donata.bandoniene@unileoben.ac.at; Tel: +43 3842 4021207; Fax: +43 3842 4021202

In the age of the global trade and the climate changes and global warming the geographic origin of food became a factor of importance. Also the desire of the consumer for food of known geographic origin has increased, hence it is possible to buy food in supermarkets with a declaration of geographical origin at a higher price than such without traceable origin.

The aim of this work is, to develop an analytical method for the control of the geographic origin of pumpkin seed oil. The development of such a method is not only of interest for scientists but also of importance for the consumer wanting to know the origin of the food products and the assurance of the purity and quality.

It is known that the group of rare earth elements (REE the *4f* elements also called lanthanoids) in plants also have a characteristic distribution pattern similar to geological samples (1). In addition, pumpkin seed oils of different geographic origin show variable trace element and rare earth patterns, therefore it is possible to trace the origin of these oils.

Since the REE concentrations are extremely low in pumpkin seed oil, a fast and sensitive analytical method with ICP-MS had to be developed and validated. In the current project pumpkin seeds from different regions in Austria and from abroad were sampled. The trace element patterns in the extracted oil of these seeds were determined and a preliminary classification with discriminate analysis was successfully done on a statistic basis. (2, 3, 4).

In addition to the study of the geographic origin it was tested, if REE distribution patterns can also be used for the identification of adulteration of high priced pumpkin seed oil with cheap neutral tasting refined oils.

## References:

(1) Markert B.; (1993) VCH Verlagsgesellschaft mbH, Weinheim 265

(2) Benincasa C., Lewis J., Perri E., Sindona G., Tagarelli A.; (2007) Anal. Chim. Acta 585: 366-370

(3) A. Heier; (2006) Dissertation Berlin

(4) Thiel G., Geisler G., Blechschmidt I., Danzer K.; (2004) Anal. Bioanal. Chem. 378:1630-1636

# EP2

## Principles for tracking the origin of organic beef using stable isotopes of life

**M. Boner\*, H. Förstel**

TÜV Rheinland Agroisolab GmbH Jülich, Germany

\*E-mail: m.boner@agroisolab.de; Tel: +49 2461 690 292, 290; Fax: +49 2461 690 299

For more than 2 years different organic beef samples from various German farms have been collected and analysed for the hydrogen, carbon, oxygen, nitrogen and sulphur stable isotopic composition to test the possibility of tracking the geographical origin. To check the differentiation of foreign beef, samples from Argentina and Chile were also included in the study.

Dealing with the fact that there is a well-known pattern of D/H and 18O/16O in the meteoric water as well as in the ground water, there is an existing link to tissue water in the beef.

On the other hand, the tissue water of the fodder has to be taken into account, as well, resulting on a seasonal depending variation in the 18O/16O and D/H ratios of the beef tissue water.

Therefore the information of the slaughter time is a helpful differentiation parameter, too.

Adding the remaining stable isotopes of the elements of life further information is available: Soils show different isotope ratios of 15N/14N and 34S/32S depending firstly on the natural geological composition and secondly on the cultivation. As the organic farming is mainly obliged to use only their produced fodder, that ratio is reflected in the beef as well. That could be demonstrated on three farms in the Colonial Bay over a time period of 18 months.

But indeed the geographical differentiation of organic beef has also limits which always have to be considered. One of the main problems is the variation of the stable isotope ratios in the different meat parts of the beef which could be a result of different turn over times.

In an analysis of 30 different meat parts of two cattle using the Switzerland segmentation protocol, there were significant variations in all stable isotopes of life detectable.

On the other hand a transportation of the cattle with a change of drinking water could be more or less neglected if the time is no longer than 5 days.



# EP3

## A new IRMS perspective on $^{13}\text{C}/^{12}\text{C}$ determination of carbon dioxide to authenticate sparkling drinks

A. Cabañero\*, M.Ruperez

Laboratorio Arbitral Agroalimentario. Ministerio de Medio Ambiente y Medio Rural y Marino. Casiopea n° 1 (Ctra. Coruña km 10.700), 28023 Madrid, Spain.

\*E-mail: acabanero@mapya.es; Tel: +34 91 3474971; Fax: +34 91 3474968

A new procedure for the determination of carbon dioxide ( $\text{CO}_2$ )  $^{13}\text{C}/^{12}\text{C}$  isotope ratios, using direct injection into a GasBench/isotope ratio mass spectrometry (GasBench/IRMS) system, has been developed to improve isotopic methods devoted to the study of the authenticity of sparkling drinks.

Thirty-nine commercial sparkling drink samples (sparkling/semi-sparkling/gasified wines and carbonated waters) from various origins were analyzed. Values of  $\delta^{13}\text{C}_{\text{cava}}$  ranged from -20.30‰ to -23.63‰, when C3 sugar addition was performed for a second alcoholic fermentation. Values of  $\delta^{13}\text{C}_{\text{water}}$  ranged from -5.59‰ to -6.87‰ in the case of naturally carbonated water or water fortified with gas from the spring, and  $\delta^{13}\text{C}_{\text{water}}$  ranged from -29.36‰ to -42.09‰ when industrial  $\text{CO}_2$  was added. It has been demonstrated that the addition of C4 sugar to semi-sparkling wine (aguja) and industrial  $\text{CO}_2$  addition to sparkling wine (cava) or water can be detected.

The results of this study indicate that the measurement of  $\text{CO}_2$  could help to verify that products are in conformity with the declared ingredients on the label. The developed method allowed us to improve the currently available methodologies for the detection of adulteration of sparkling drinks. In addition, it overcomes technical difficulties associated with sample collection and purification occurring with previous procedures.

The new procedure has advantages over existing methods in terms of simplicity (it requires no purification step or  $\text{CO}_2$  extraction before analysis), analysis time (less than 11 min per sample and 10 replicates in a single sample acquisition), repeatability ( $\text{SD} < 0.1\%$ ) and sample treatment. Furthermore, it is the first isotopic method developed that allow  $^{13}\text{C}/^{12}\text{C}$  determination directly from a liquid sample without previous  $\text{CO}_2$  extraction. No significant isotopic fractionation was observed nor any influence by secondary compounds present in the liquid phase. Additionally, the present method can be easily implemented in enforcement laboratories. Thus, this technique could be used routinely and efficiently in  $\text{CO}_2$  analysis to meet existing regulations.

# EP4

## **Authenticity of vinegar controlled by stable isotope and 14-carbon data**

**H. Förstel\*, L. Miebach and M. Boner**

TÜV Rhineland Agroisolab GmbH Jülich, Germany

\*E-mail: h.foerstel@agroisolab.de; Tel: +49 2461 690 293, 290; Fax: +49 2461 690 299

Synthetic vinegars seem to be much cheaper compared to the biotechnological products using presently grown biomass. Their nutritional value is considered to be much lower than that of the natural vinegars. Up to 40 commercial products have been tested, not only considering the <sup>13</sup>C/<sup>12</sup>C ratio of the whole molecule which has been isolated by azeotropic distillation. The two different carbon isotopes can be measured separately after heating the calcium acetate which results in calcium carbonate, according to Schmid et al. 1981 (1). In contrast to the acetate the product of heating, the carbonate, contains the carbon of the carboxyl group only. Thus, the <sup>13</sup>C/<sup>12</sup>C ratios of both carbon isotopes can be determined separately.

In rare cases the vinegar is produced from C4-material as can be seen distinctly. This can be confirmed, for the biotechnological production results in a certain stable isotopic difference between both carbon positions within the acetic acid molecule. This is caused by the fractionation processes in the metabolic pathway of organisms producing acetic acid. Considering synthetic acetic acid this relation is not valid, for the precursors of chemical synthesis, methanol or acetaldehyde respectively, differ in their origin and consequently <sup>13</sup>C/<sup>12</sup>C ratio from that of biological materials and from each other. While the <sup>13</sup>C/<sup>12</sup>C ratios of biotechnologically produced vinegar of both carbon atoms are correlated ( $r_2 = 0.91$ ) to each other, the different materials are visible in the low correlation coefficient between both carbon atoms of synthetic vinegars ( $r_2 = 0.39$ ).

The differences in the <sup>13</sup>C/<sup>12</sup>C ratio may not be sufficient to suspect the products, but additional analysis of the D/H and <sup>18</sup>O/<sup>16</sup>O ratios of the calcium acetate confirm the suspicion. The technical available hydrogen usually has a higher or at least conspicuous D/H ratio compared to the natural organically bound hydrogen. The biggest mass difference of hydrogen between the stable isotopes of any element (1 : 2) makes his fractionation very sensitive to bio- or chemosynthetic reactions, and consequently may serve as an important tool in the application of stable isotopes to detect frauds. Additionally hydrogen is not involved in biochemical (or some synthetic) production processes with a demand of oxygen as reaction partner. The oxygen normally is taken from the ambient air which has a world wide constant and significant enriched oxygen isotope ratio (DOLE effect). This can be used to trace back even the kind of chemical synthesis to produce vinegar.

To measure the <sup>18</sup>O/<sup>16</sup>O ratio without disturbing background effects an oxygen-free and thermally stable material of the reaction column has to be used which can resist to higher temperatures, as e.g. silicon carbide. This material necessitates a single heating tube only, lowers the carbon dioxide background and avoids the formation of a closure from the silver at lower temperatures.

The application of Low Level Scintillation Counting of 14-Carbon improves the control of the production of vinegar from recently grown biomass. Instead of the meaning that synthetic material usually is generated from cheap fossil carbon sources and therefore does not contain <sup>14</sup>C, in fact necessitates the natural background of radioactivity specific shielding by lead and selecting electronics of the liquid scintillation counter. Beside the counting rate and efficiency the determination of the amount of carbon which is absorbed in the LSC vial is the second factor determining the reproducibility of the procedure. Both procedures have been adapted to ensure a rapid application to satisfy the need of the customers.

### References:

- (1) Schmid E.R.; et al (1981) Biomed Mass Spectrom 8: 496-499

# EP5

## Guarantee of quality and traceability

### F. Gerriets\*

Director Organic Services GmbH, is the responsible international business development partner of Intact Consult GmbH, Lebring, Austria

\*E-mail: frank.gerriets@intact.at, Tel: +49 89 820759 04; Fax: ++49 89 820759 19

Building of consumer confidence is of utmost importance particularly in the food sector. This confidence is primarily influenced by the qualitative positioning of the product as well as positive or negative news coverage. An extensive data management of the flow of goods and the specific processing of the gained data contribute to establishing confidence and credibility.

To ensure guaranteed quality performance, quality management has to make consistently qualified and accurate statements regarding safety and quality of food over the entire value added chain. This requires continuous access to reliable data from different sources. The statements to safety and quality of food depend on different pillars that are identified in various steps of the value added chain from the farmer, the manufacturer to the final distributor: product authenticity/credibility (organic), product quality (flavour), origin, traceability, residue-free status and process quality (hygiene).

The hidden complexity of all data managed which is determined by the high degree of detail of the data that are to be combined poses a special challenge in this process.

Today the market increasingly relies on certification standards for safeguarding product and process quality. In future these standards will most likely evolve towards controls or audits of the flow of goods (e.g. Global GAP, Organic, Fairtrade) and thus will resemble a permanent control process. For this reason, the static situation of current processes and product certification will be overcome.

Intact's activities extend to audit management, traceability of goods and quality assurance. Intact has developed software tools that enable the management of data in complex environments of the food chain. To highlight the importance of the implementation of product chain information systems in order to increase safety and quality of food, Intact will present practical examples from the international fruit industry (Frutera and Spar / VI.P Südtirol and bio-mit-gesicht.de), and illustrates conceptual design and realisation in the sustainable fishing industry (Marine Stewardship Council, MSC).

# EP6

## Ovine intra- and inter-muscular variation in turnover as recorded by carbon stable isotopes

S.M. Harrison<sup>1,2,\*</sup>, F.J. Monahan<sup>1</sup>, A.P. Moloney<sup>3</sup>, S.D. Kelly<sup>4</sup>, F. Cuffe<sup>5</sup>, A. Rossmann<sup>6</sup>, A. Schellenberg<sup>7</sup>, F. Camin<sup>8</sup>, O. Schmidt<sup>2</sup>

<sup>1</sup>UCD School of Agriculture, Food Science & Veterinary Medicine, Belfield, Dublin 4, Ireland.

<sup>2</sup>UCD School of Biology & Environmental Science, Belfield, Dublin 4, Ireland.

<sup>3</sup>Teagasc, Grange Beef Research Centre, Dunsany, Co. Meath, Ireland.

<sup>4</sup>UEA School of Environmental Sciences, Norwich, NR4 7TJ, United Kingdom.

<sup>5</sup>UCD School of Mathematical Sciences, Belfield, Dublin 4, Ireland.

<sup>6</sup>Isolab GmbH, Schweitenkirchen, Germany.

<sup>7</sup>LGL Bayern, Dienststelle Oberschleißheim, Erlangen, Germany.

<sup>8</sup>IASMA Research Centre, Agrifood Quality Department, San Michele all'Adige, Italy.

\*E-mail: sabine.harrison@ucd.ie; Tel: +353 1 7167785; Fax: +353 1 7161102

Stable isotope ratio analysis (SIRA) is used increasingly as a tool in food authenticity and food traceability. However, correct interpretation of observational data gathered in surveys requires a mechanistic understanding of underlying processes through controlled diet switching experiments, for example to establish the half-lives of the muscles used as meats.

In a controlled on-farm experiment we switched 28 lambs from a C<sub>3</sub>/C<sub>4</sub>-control diet to a C<sub>4</sub>-experimental diet (isotopic spacing between diets was 10.2 ‰ for C). Fourteen animals received a high-energy allowance (HEA), the other 14 animals received a low-energy allowance (LEA). Over the 33-week course of the experiment, animals were slaughtered at regular intervals to monitor the change in stable isotope ratios.

Seven muscles (*Biceps femoris* (BF), *Cleidooccipitalis* (CO), *Flexor digitorum superficialis* (FDS), *Longissimus dorsi* (LD), *Psoas major* (PM), *Semimembranosus* and *Semitendinosus*) were collected 24 h *post mortem*, trimmed of superficial fat, weighed and vacuum packed. Statistical analysis of the muscle weights revealed that the energy allowance (EA) had a significant impact on all muscle weights ( $p < 0.05$ ,  $F > 6.45$ ), whereas the sex of the animal was significant for four muscles ( $p < 0.01$ ,  $F > 10.82$ ) and the duration of the experiment affected only three muscles significantly ( $p < 0.05$ ,  $F > 3.76$ ).

To investigate any possible intra-muscular variation in  $\delta^{13}\text{C}$ , LD was sampled at ten different locations. To determine inter-muscular variation, the most central location of five muscles (BF, CO, FDS, LD and PM) was sampled and C SIRs measured. Analysis revealed that there was a significant difference between sampling locations along the muscle. Furthermore, the samples collected from the five different muscles showed a significant difference in half-lives for the HEA and LEA ( $p < 0.0001$ ,  $t = -22.36$ ), with animals on the HEA having shorter half-lives than the animals on the LEA. However, not all five muscles showed the same half-lives. The half-life of the muscle FDS was shorter by more than 10% on the HEA and by more than 20% on the LEA compared to the other four muscles. The lipid that was extracted from the LD prior to SIRA was also measured as whole lipid (WL) along with its fractions, neutral lipids (NL) and polar lipids (PL). Analysis revealed that all lipids (WL, NL and PL) as well as the subcutaneous adipose tissue close to the LD responded more slowly to the diet switch than the muscle they were extracted from. However,  $\delta^{13}\text{C}$  values of lipids from animals on the HEA were somewhat elevated compared to  $\delta^{13}\text{C}$  values of lipids from animals on the LEA.

In conclusion, this experiment with lambs showed for the first time that the sampling location within one muscle has a significant impact on the results of a survey. We also showed that muscles typically consumed as human food have similar half-lives, circumstantiating the findings from surveys such as that undertaken by Camin *et al.* (1). Finally, lipids responded much more slowly to the new diet than the muscle they were extracted from. We therefore suggest longer equilibration times in future feeding experiments as lipids are a potential second tissue for the "isotopic clock" approach proposed by Phillips *et al.* (2)

### References

1. Camin F., *et al.*; (2007) *Anal. Bioanal. Chem.* 389: 309.
2. Phillips D.L., *et al.*; (2006) *Oecologia.* 147: 195.

# EP7

## **Stable isotope analysis of light and heavy elements for differentiation between German and Austrian bovine samples**

**N. Herwig<sup>1\*</sup>, J. Vogl<sup>1</sup>, W. Pritzkow<sup>1</sup>, M. Rosner<sup>1</sup>, U. Panne<sup>1</sup>, H. Fry<sup>2</sup>, A. Preiss-Weigert<sup>2</sup>, H.M. Lochotzke<sup>2</sup>, A. Meixner<sup>3</sup>**

<sup>1</sup>Federal Institute for Materials Research and Testing, Unter den Eichen 87, D-12205 Berlin,

<sup>2</sup>Federal Institute for Risk Assessment, Thielallee 88-92, D-14195 Berlin

<sup>3</sup>German Research Center for Geosciences, Telegrafenberg, D-14473 Potsdam

\*E-mail: nadine.herwig@bam.de, Tel: +49 30 81044112

During the last years cases of food adulteration, for example flavouring of food with unlicensed substances or the incorrect declaration of food, lead to an insecurity of consumers. The authenticity of food plays an outstanding role for everybody. Especially consumers want to make sure, that the organic milk, bought at the supermarket, really is milk from cows of organic livestock husbandry. In some areas of food production the determination of stable isotope ratios of the light so-called bio- elements like hydrogen, carbon, nitrogen, oxygen and sulphur has been successfully used during the last 20 years to verify the authenticity of food in regard to geographic origin and food adulterations.

In an ongoing research project the isotope ratios of the heavier elements strontium and magnesium are determined beside the isotope ratios of the light bio-elements carbon and nitrogen in different bovine matrices. In 2006 milk and urine samples of cows from two farms of Germany and Austria were examined. The cows of Austria are pasture cows, reared under rules of organic agriculture. The feeding regime of cows of Germany nearly corresponds to conventional farming.

One important aspect for our research is to determine the correlation between the diet of cows and the resulting variations in the isotopic compositions of the milk and urine samples. Investigation of isotope ratios of the light elements as well as the heavier elements in bovine samples show, that the isotope ratios of the heavier elements could be a valuable tool for discriminating cattle of different livestock farming or different geographical origin.

# EP8

## Identification of ostrich meat and evaluation of ostrich meat grades by high performance liquid chromatography with electrochemical detection using copper nanoparticle-plated electrode

C. J. Hung<sup>1</sup>, S. Y. Guo<sup>1</sup>, C. C. Chang<sup>2</sup> and C. C. Chou<sup>1\*</sup>

<sup>1</sup>Department of Veterinary Medicine, College of Veterinary Medicine, National Chung Hsing University, Taichung, Taiwan

<sup>2</sup>The Graduate Institute of Veterinary Public Health, College of Veterinary Medicine, National Chung Hsing University, Taichung, Taiwan

\*E-mail: ccchou@nchu.edu.tw; Tel:+886 4 22840404 x 177; Fax:+886-4 22862073

Accurate identification of the origin of meat species presents a considerable challenge for government authorities and analysts. Ostrich meat has characteristic nutritional properties such as high in protein, amino acids and iron content, and low in calorie, fat and cholesterol. The high unit-price and low supply has made it a target for adulteration and replacement by cheaper meats. The purpose of the study was to develop a fast, economic and routinely applicable chromatographic method with minimal sample preparation procedure to differentiate ostrich meat from pork, beef and chicken, and to evaluate different grades/geographical parts of ostrich meat to assure fair pricing. 5 gram of fresh meat was homogenized in 5 ml of phosphate buffer and centrifuged before the resultant supernatant was filtered for HPLC analysis. The Chromatographic separations were performed using Zorbax Eclipse AAA column and 10 mM phosphate buffer as mobile phase, in a portable flow-injection analysis system employing a copper nanoparticle-plated screen-printed electrode (Cu<sup>n</sup>-SPE). The results indicated that all 4 meats could be differentiated in 5 minutes by characteristic chromatographic profiles consisting only 4 major peaks. An avian-specific peak for differentiation from mammal species was identified and LC/MS/MS was performed to elucidate the structure of the peaks responsible for species differentiation. Furthermore, statistical analysis (AOC curve) showed that by using the HPLC-EC method, the peak ratios of ostrich meat significantly differ in different parts of the ostrich, suggesting that peak ratios could be applied for differentiation of ostrich meat grades (price levels) with high sensitivity ( up to 95%) and specificity (up to 100%). The effects of *storage temperature and time on chromatographic profiles were also studied and* suggested that the evaluation of meat freshness is a plausible direction. In conclusion, this HPLC-EC method appeared to be superior to UV detection in terms of profile simplicity for comparative purposes. It is simple and suitable for routine rapid differentiation of ostrich meat from common meat species and for evaluation of ostrich meat grades without any derivatization or complex extraction procedures. It is feasible with great potential to extend this application to other meat species.

### References:

- (1) Chou C. C., Lin S. P., Lee K. M., Hsu T. C., Vickroy T. W., Zen J.M.; (2007) *J Chromatogr B* 846: 230-239.
- (2) Achonherr Scho<sup>o</sup>nherr J.; (1998) VDLUFA-Schriftenreihe 49, VDLUFAVerlag: Darmstadt, Germany, p 433
- (3) Vallejo-Cordoba B., Cota-Rivas M.; (1998) *J Capillary Electrophor* 5: 171-175
- (4) Dunnett M., Harris R.C.; (1997) *J Chromatogr B* 688: 47-55
- (5) Sales J., Hayes J. P.; (1996). *Food Chem* 56: 167-170
- (6) Ashoor S. H., Monte W. C., Stiles P. G.; (1988) *J Assoc of Off Anal Chem* 71: 397-403.

# EP9

## Determination of delta18O in water of Czech wine products using pyrolysis-continuous flow IRMS technique

**K. Kolář\*, H. Šimková, P. Šabata and L. Vyčítalová**

Czech Agriculture and Food Inspection Authority, Květná 15, 603 00 Brno, the Czech Republic

\*E-mail: kamil.kolar@szpi.gov.cz; Tel: + 420 543540261; Fax: + 420 543540241

A method for determination of delta18O values of Czech wine products water is presented. The wine samples were distilled according to 1990/2676/EC to extract ethanol used for analysis D/H1 and delta13C (1). Water was extracted from distillation residue by distillation at low pressure (2). Extracted water samples were injected through heated injector (typical amount 0.5 -1 microL) and pyrolysed on EA-IRMS continuous flow system. Delta18O values of resulting CO were determined at precision level about 0.1 per thousand (3,4).

### References:

- (1) European Commission (1990), Commission Regulation (EEC) No. 2676/90, Official Journal of the European Communities L 272: 1-192.
- (2) ENV 12142:1996
- (3) Jamin E.; et al. (2003) Journal of Agricultural and Food Chemistry 51: 5202-5206
- (4) Gehre M.; et al. (2004) Rapid Communications in Mass Spektrometry 18: 2650-2660

# EP10

## Authentication of the commercial denomination *light tuna* by real time PCR

I. López, M<sup>a</sup> J. Sierra, N. Egurrola, N. Gutierrez and M. A. Pardo\*

Food Research Division, AZTI-Tecnalia. Parque Tecnológico de Bizkaia Astondo Bidea-Edificio 609 48160 Derio (Bizkaia) SPAIN, E-48395 Sukarrieta, Bizkaia.

\*E-mail: mpardo@azti.es; Tel.: +34 94 657 40 00; Fax: +34 94 657 25 55

The labelling *light tuna* is a commercial denomination created by the Spanish canning industry that has been recently recognised by the Spanish legislation (RD 1193/2000). The light tuna label refers to yellowfin or *Thunnus albacares*. The yellowfin tuna is a worldwide distributed species which share the habitat with other tuna species such as the bigeye tuna (*Thunnus obesus*). As a matter of fact, both species are generally caught simultaneously and then processed together by the canning industry. Nowadays, the authentication of seafood products is a major concern in order to assure the traceability system from fish to fork, and one of the most important problems for canning industry is actually the presence of tuna mixtures in canned products. To date, none of the analytical techniques enable to evaluate the real scale of this problem. In fact, the methodologies currently used are based on the genetic analysis of only a small tissue portion (e.g. PCR-FINS). These approaches are insufficiently accurate to detect and quantify the presence of tuna species mixtures.

We have developed a specific system to authenticate canned products labelled as *light tuna*. This system is based on the use of DNA probes that specifically identify and semi-quantify the presence of different tuna species in mixtures. The method has been successfully validated with binary mixtures constructed in the laboratory. In fact, we were able to detect up to 10 % of bigeye tuna in *light tuna* canned products adulterated in the laboratory. An evaluation of the Spanish market has also been launched in order to analyse the real scale of this problem.

Summarizing, we have developed an innovative detection system to authenticate the light tuna label in order to assure the traceability system in the canning industry which eventually will result in a benefit for the consumer.



# EP11

**Effect of light in extra-virgin olive oil odour profile, volatile compounds and vitamins****V. Messina<sup>1\*</sup>, A. Biolatto<sup>2</sup>, A. Descalzo<sup>2</sup>; A. Sancho<sup>2</sup>, R. Baby<sup>1</sup> and N. E. Walsøe de Reca<sup>1</sup>**

<sup>1</sup>CINSO (Centro de Investigaciones en Sólidos) CITEFA-CONICET, Juan Bautista de la Salle 4397, Villa Martelli, (B1603ALO) Buenos Aires, Argentina.

<sup>2</sup>Instituto Tecnología de Alimentos, Centro de Investigación de Agroindustria, Instituto Nacional de Tecnología Agropecuaria. INTA, CC 77 (B1708WAB) Morón, Buenos Aires, Argentina.

\*E-mail: vmessina@citefa.gov.ar; Tel: +54-11-4709-8158; Fax: +54-11-4709- 8158

Aroma, taste, colour and nutritive properties of virgin olive oil distinguish it from other edible oils. Although virgin olive oil is considered to be stable, it is susceptible to oxidation and off-flavour can be detected when oxidation process starts. Temperature, light, oxygen concentration, oil processing and fatty acid composition can affect the oxidative stability (1). The aim of this work was to study changes in odour profile of Arbequina and Arauco extra-virgin olive oil during storage at light and dark using an electronic nose approach complemented with the determination of volatile aldehydes (3 methylbutanal, *n*-pentanal, *n*-hexanal, *n*-heptanal and *n*-nonanal) and vitamin E content ( $\alpha$  and  $\gamma$ -tocopherol). Extra-virgin olive oil of Arbequina and Arauco varieties were stored at artificial light and at dark during 120 days. Oil samples were extracted at the beginning of the experiment and then each 20 days, until 120 days, for electronic nose,  $\alpha$  and  $\gamma$ -tocopherol and volatile compounds determination. A MOSES II (Modular Sensor System) EN was used to discriminate odours profile. MOSES II contains two sets of sensors: one is composed by eight pure and doped SnO<sub>2</sub> sensors and the other one by eight different microbalance quartz (QMB) sensors (2). The electronic nose provides data arranged in bidimensional plots using the Principal Component Analysis statistical method. Vitamin E ( $\alpha$  and  $\gamma$ -tocopherol) was analyzed using a High Performance Liquid Chromatography and volatile compounds were analyzed with Solid Phase Microextraction-Gas Chromatography. Principal Component Analysis data of electronic nose showed differences in odour profile between Arbequina extra-virgin olive oil exposed to light from those exposed to dark, after 60 days of storage. Arauco variety didn't show changes in odour after light and dark exposure. Vitamin E showed decrease in content for both varieties. Production of volatile compounds showed similar levels by exposure to light and dark for both varieties.

**References:**

- (1) Chloe E., Min, D. (2006). Comprehensive reviews in Food Science and Food Safety 5: 169-186.
- (2) Baby R., Cabezas M., Walsøe de Reca E. (2000). Sensors and Actuators B: Chemical 69: 241-218.

# EP12

## Analysis of volatile compounds in extra-virgin olive oil after light exposure by solid phase microextraction and gas chromatography

V. Messina<sup>1\*</sup>, A. Biolatto<sup>2</sup> and N. Walsøe de Reca<sup>1</sup>

<sup>1</sup>CINSO (Centro de Investigaciones en Sólidos) CITEFA-CONICET, Juan Bautista de la Salle 4397 (B1603ALO) Villa Martelli, Buenos Aires, Argentina.

<sup>2</sup>Instituto Tecnología de Alimentos, Centro de Investigación de Agroindustria, Instituto Nacional de Tecnología Agropecuaria INTA, CC 77 (B1708WAB) Morón, Buenos Aires, Argentina.

\*E-mail: vmessina@citefa.gov.ar; Tel: +54-11-4709-8158; Fax: +54-11-4709-8158

Flavour is an important feature for the quality of olive oils. Oxidation of fatty acids in vegetable oils results in the formation of volatile compounds among which many have an unpleasant odour and are responsible for flavour problems (1). Many analytical procedures have been used to identify and quantify the volatile components resulting from fatty acid oxidation, such as static headspace, dynamic headspace and direct chromatography. Solid phase microextraction technique (SPME)—a solvent free extraction technique is based on absorption of analytes onto a polymer-coated silica fiber and their subsequent desorption in the hot injection port of a gas chromatograph (2). The aim of this work was to develop the SPME technique for qualitative and quantitative analysis of volatile aldehydes of extra-virgin olive oil after light exposure. Extra-virgin olive oil of Arbequina and Arauco varieties were stored at artificial light and at dark during 120 days. Oil samples were extracted at the beginning of the experiment and then each 20 days until 120 days, for volatile compounds determination. Headspace solid phase microextraction (HS-SPME) was evaluated for qualitative and quantitative analysis of aldehydes compounds present in Arbequina and Arauco extra-virgin olive oil after artificial light and dark exposure. Divinylbenzene/Carboxen/Polydimethylsiloxane (DVB/CAR/PDMS) fiber coating was evaluated for reproducibility and linearity of response and was found to be suitable for the analysis of volatile aldehydes. Sampling and chromatographic conditions were examined and the HS-SPME method, coupled with flame ionization detection, was applied to the analysis. The sampling temperature was 50 °C and the fiber was exposed to the headspace for 40 min. 3-methylbutanal, *n*-pentanal, *n*-hexanal, *n*-heptanal and *n*-nonanal showed similar levels by exposure to light and dark for both varieties. The results obtained in this study, provided information about the potential application of HS-SPME method for the analysis of volatiles in Arbequina and Arauco extra-virgin olive oil.

### References:

(1) Morales M., Luna G., Aparicio R. (2005). Food Chemistry 9: 293-301.

(2) Vichi S., Pizzale L., Conte S., Buxaderas S., Lopez-Tamames E. (2003). Journal of Agriculture and Food Chemistry 51: 6564–6571.

# EP13

## Evaluation of an ultrasound-assisted digestion method for determination of arsenic and lead in edible citric acid samples by ETAAS

A. Pourhossein<sup>1,\*</sup>, M. Madani<sup>1,2</sup>, M. Shahlaei<sup>3</sup>

<sup>1</sup>Research and Development Center, Kimia Gharb Gostar Industrial Company, Kermanshah, Iran

<sup>2</sup>Department of Chemistry, Faculty of Science, Islamic Azad University of varamin (pishva), Varamin, Iran

<sup>3</sup>Department of Pharmaceutical Chemistry, Isfahan University of Medical Science, Isfahan, Iran

\*E-mail: apourhossein@ymail.com; info@KGGICO.com; Tel: +98-831-2333302

Arsenic is a worldwide recurring pollutant of natural origin commonly associated with ores of metals like copper and gold (1). This metalloid is found in the environment in several chemical forms (2) and is well known as a source of serious health effects by prolonged intake even at low concentrations. The pollution of lead is one of the most serious environmental problems because of its stability in contaminated site and complexity of mechanism in biological toxicity (2). Currently, electrothermal atomic absorption spectrometry (ETAAS) is one of the most reliable and powerful analytical techniques for the determination of trace and ultra-trace elements in food stuff samples. For elemental analysis, the sample preparation procedure employed is ordinarily the most time-consuming step in the overall analysis. The efficiency of the sample preparation procedures can be enhanced with the use of ultrasound irradiation (3). Ultrasonic radiation can be considered an alternative for sample pre-treatment since ultrasound facilitates an auxiliary energy and accelerates some steps, such as dissolution, fusion and leaching, among others (3). In this work a sample preparation method based on ultrasound-assisted digestion of arsenic and lead from edible citric acid samples under ultrasonic effect has been described. Parameters influencing pseudo-digestion, such as sonication time, sample mass and solvent system were fully optimized. The best conditions for metal pseudo-digestion were as follows: a 20 min sonication time, a 2.0 g sample mass (in 10 ml solvent) and a mixture of concentrated HNO<sub>3</sub>–H<sub>2</sub>O<sub>2</sub> (3:1 v/v). Analytical results for the two metals by ultrasound assisted pseudo-digestion dry ashing and conventional wet digestion methods showed a good agreement, thus indicating the possibility of using mild conditions for sample preparation instead of intensive treatments inherent with the digestion methods.

**Key words:** Arsenic, Lead, Edible citric acid, Ultrasound-assisted digestion, ETAAS

### References

(1) Nriagu, J. O.; Frankenberger, W. T.; (2002) Environmental Chemistry of Arsenic, Marcel Dekker, New York, 2002, pp. 1-26.

(2) Cullen, W. R.; Reimer, K. J.; (1989) Chem. Rev. 89: 713.

(3) Bendicho, C.; Lavilla, I.; (2000) Ultrasound-assisted extraction, Encyclopedia of Separation Science, Academic Press, London.

# EP14

## Application of PCR-DGGE in determining geographical origin of fruits: cases studies of physalis and shea tree fruits

A. F. El Sheikha<sup>1,2\*</sup>, D. D. Le Nguyen<sup>1,3</sup>, I. Métayer<sup>1</sup>, D. Montet<sup>1</sup>

<sup>1</sup>Centre de Coopération Internationale en Recherche Agronomique pour le Développement, CIRAD, UMR Qualisud, TA 95B/16, 34398 Montpellier Cedex 5, France

<sup>2</sup>Minufiya University, Faculty of Agriculture, Department of Food Science and Technology, 32511 Shibin El Kom, Minufiya Government, Egypt

<sup>3</sup>Can tho University, Faculty of Agriculture, Viet Nam

\*E-mail: elsheikha\_aly@yahoo.com

The determination of geographical origin is a demand of the traceability system of import–export food products. One hypothesis of tracing the source of a product is by analyzing in a global way the microbial communities of the food and links this analysis to the geographical origin of the food. The molecular technique employing 26S rDNA profiles generated by PCR-DGGE was used to detect the variation in yeast community structures of three species of *Physalis* from four Egyptian governments and five fruits of shea tree from three regions of Senegal. The DGGE gels showed some significant differences in the migration patterns. However, the duplicates for each sampling location gave statistically similar DGGE patterns throughout the study. We demonstrated that there was a link between the yeast populations and the geographical area. When the 26S rDNA profiles were analyzed by multivariate analysis, distinct microbial communities were detected. The band profiles from different governments or regions were different and were specific for each location and could be used as a bar code to discriminate the origin of the fruits. This method is a new traceability tool which provides fruit products with a unique bar code and makes it possible to trace back the fruits to their original location.

*Physalis* is included in the priority list of many governments' horticulture and fruit export plan. It is exported from several countries including Colombia, Egypt, Zimbabwe and South Africa, but Colombia stands out as one of the largest producers, consumers and exporters. Colombia exports of *Physalis* in 2004 were worth 14 millions USD (Bayer Crop Science, 2006). In Egypt, economical importance of *Physalis* is rising, due to, achieving a great success in local, Arabic and European markets (El Sheikha, 2004).

*Physalis* as the whole plant has many medicinal properties, including antipyretic, depurative, diuretic, pectoral, and vermifuge. A decoction is used in the treatment of abscesses, cough, fevers or sore throat (Duke and Ayensu 1985). The pulp is nutritious, containing particularly high levels of carotenoids, minerals, essential amino acids and vitamin C (El Sheikha et al., 2008).

Regarding shea tree fruits, only seven countries have statistics. Nigeria accounts for more than 60% of the production of shea butter in 2005. It is followed by Mali, Ghana and Burkina Faso, which together account for just under a third of world production in 2005. In Europe, shea butter is used mainly (95%) by the chocolate industry. The quantities exported to Japan, the United States or Switzerland would be mainly used for cosmetic or pharmacological (FAOSTAT, 2007).

**Key words:** Traceability, PCR-DGGE, *Physalis*, Shea tree fruits, Yeast communities, Origin

# EP15

## Isotopic characterization of authentic Czech honey: deuterium and carbon isotopes

H. Šimková\*, K. Kolář, P. Sečkářová, L. Vyčítalová and R. Buchtová

Czech Agriculture and Food Inspection Authority

\*E-mail: hana.simkova@szpi.gov.cz Tel: +420 543 540 243; Fax: +420 543 540 241

30 authentic samples of Czech honeys were obtained from Research Institute for Apiculture in Dol, Czech Republic. Some of these honeys were declared to be monofloral (oilseed-rape, lime, acacia, sunflower).  $\delta^{13}\text{C}$  values of bulk honey and honey protein and  $\delta^{13}\text{C}$  and (D/H)I values of ethanol prepared by fermentation of honey were measured.

### References:

- (1) AOAC official method 998.12 (1998)
- (2) White J.W.; et al. (1998) J. AOAC Int. 81: 610-619
- (3) Giraudon S.; et al. (2000) J. AOAC Int. 83: 1401-1409
- (4) Lindner.; et al. J. Agric. Food Chem. 44: 139-140

# EP16

## Internal traceability system database modeling for a grain elevator

M. Thakur<sup>1\*</sup>, C. R. Hurburgh<sup>1,2</sup>

<sup>1</sup>Department of Agricultural and Biosystems Engineering, Iowa State University, Ames, IA 50011

<sup>2</sup>Department of Food Science and Human Nutrition, Iowa State University, Ames, IA 50011

\*E-mail: maitri@iastate.edu; Tel: +1 515 294 9461; Fax: +1 515 294 6383

Consumer experiences with food safety issues combined with a growing demand for high quality food and feed products have increased interest in systems to aid in food traceability efforts. Implementation of a traceability system in the bulk grain supply chain is a complex task. Food safety and traceability laws exist in several countries but traceability is important for several reasons other than just a legal obligation. These reasons include efficient response to food security threats, documenting chain of custody, documenting production practices, meeting regulatory compliance, and analyzing logistics and production costs. According to The Bioterrorism Preparedness and Response Act of 2002, in case of a food related emergency, a company should be able to produce records of the product, the related suppliers and customers based on one step up and down in the supply chain within a 24 hour time frame. So, food traceability is the responsibility of all the actors in the supply chain. Effective supply chain traceability can only be achieved with a combination of internal traceability and chain traceability. The purpose of this work is to design a traceability systems database model for internal traceability at a grain elevator. Different lot-activities take place as the grain moves through the supply chain from the farm to the consumer. Grain elevators handle bulk commodities marketed against generic grade standards that are based on physical attributes. At an elevator, grain lots (inbound deliveries) are commingled to meet buyer specifications, and lot identity is not maintained. As a result, an outbound shipment to a customer can contain grain from many sources. In a food related emergency, it would be almost impossible to trace the problem source and to track other affected lots. This process is very time intensive, increases the recall costs, and can lead to a tainted brand name for the company. This problem can be mitigated by an efficient internal record keeping system that would document all grain activities, including movement, aggregation, segregation, transformation and destruction. A relational database management system is developed for internal traceability at a grain elevator. This system stores all the information related to grain lots and can be queried to retrieve information related to incoming and outgoing lots. This system can be used to trace back the source of a given lot and track forward information related to the shipped lots. Following the RDBMS approach for recording all grain movements is an efficient way to link the incoming and outgoing grain lots at an elevator.

### References:

- (1) Hoffer J.A., Prescott M., McFadden F. (2006), Modern Database Management (8<sup>th</sup> ed), Prentice Hall.
- (2) U.S. Food and Drug Administration (2002), The Bioterrorism Act of 2002.  
<http://www.fda.gov/oc/bioterrorism/Bioact.html>

# EP17

**Carbon isotope ratio analysis of authentic and commercial essential oils of lavandin (lavandin hybrida)****S. Wagner<sup>1\*</sup>, P. Vreca<sup>2</sup>, A. Leis<sup>3</sup> and H. Boechzelt<sup>1</sup>**

<sup>1</sup>Joanneum Research GmbH, Institute of Sustainable Techniques and Systems, Elisabethstraße 16/I, A-8010 Graz, Austria

<sup>2</sup>Jozef Stefan Institute, Department of Environmental Sciences, Jamova 39, SI-1000 Ljubljana, Slovenia

<sup>3</sup>Joanneum Research GmbH, Institute of Water Resources Management, Elisabethstraße 16/II, A-8010 Graz, Austria

\*E-mail: susanne.wagner@joanneum.at; Tel: +43 316 8762418; Fax: +43 316 87692418

Essential oils are defined as complex mixtures of fragrance and flavour substances originated from plants. The mixtures are composed of up to 200 different compounds belonging to different compound groups e.g. terpene hydrocarbons - cyclic or non cyclic – and their oxygenated isoprenoid compounds. Since many years gas chromatography – mass spectrometry (GC-MS) has been the most essential and popular equipment for essential oils and flavour analysis. It is used mainly for qualitative and quantitative analysis of volatile compounds. The analysis of the isotopic compositions of essential oil compounds by isotope ratio mass spectrometry coupled with a gas chromatograph (GC-IRMS) is a powerful tool in the authenticity assessment of essential oils and flavours.

Several commercial and authentic essential oils of lavandin (Lavandin hybrida) were investigated for the carbon isotope ratio of their main compounds (linalool and linalyl acetate) and their minor compounds (eucalyptol and camphor) by GC-C(ombustion)-IRMS. Authentic samples of lavandin were distilled in 2007. In addition, commercially available samples of the individual main compounds of the oil with proclaimed synthetic and natural origins were investigated for their carbon isotopic composition by GC-C-IRMS and EA-C-IRMS in order to compare the investigated carbon isotopic values. All oils were analyzed via GC-MS and GC-FID to enable the identification of the compounds in the GC-C-IRMS chromatograms and to investigate the quantity of the single essential oil compounds. Data were interpreted regarding suitability for authenticity assessment of the analyzed essential oil samples.

# EP18

## Iris recognition middleware and key technology in food supply chain: taking cows as an example

L. Zhao\*

Institute of Systems Engineering, Southeast University Nanjing, Jiangsu 210096; P. R, China

Tel: +86-25-52090759; Fax: +86-25-52090759

\*E-mail: ldzhao@seu.edu.cn

As the global development of food industry and trade, emergent event on food safety shows rapid expansion and spreading. The zoonosis, such as bse, avian influenza and foot and mouth diseases, pose a threat to food safety and human health. And it brings heavy economic lost and social emergency to epidemic area. Some developed countries have established food safety tracing system to guarantee the human health and lessen the lost. Meat products are essential industry and the main kind of food in international trade. In China, it has not been fully prepared to treat the food safety problem, and lack of the technology to trace back. So it is important to study the traceability technology of meat food supply chain.

It is the first time to use iris of animals to realize traceability by individual identification in the world. If we input the information of the cows or other individuals which will be sent to the market into the database, then we obtain the location of the carrying viruses individual accurately by individual identification. This research takes the cows as an experimental object, and generates iris encoding by using suitable algorithms and a developed middleware.

The iris recognition middleware has three steps: image preprocessing, iris feature extraction and iris matching.

**Step1:** Image preprocessing can be decomposed into threshold transformation, edge detection, iris location and normalization. First, we realize binarization processing by threshold transformation. Secondly, we use an edge detection operator to determine the edge, and locate the iris regional by the square fitting algorithm. Thirdly, we normalize the iris regional to a rectangular sized  $64 \times 256$  by polar coordinates transformation.

**Step2:** Iris feature extraction is used to generate the iris encoding. After the iris regional location, we define the scope for filtering. This study does research on the 2D Gabor filter, and obtains the parameters of the filters by simulation. In this study, the filter consists of 49 Gabor filters. Different filters are sensitive to different features, so they can be used to do the feature extraction. This study divides the image into 64 squares, and the size of each square is  $16 \times 16$ . The image can be transformed to a code having 512 bits.

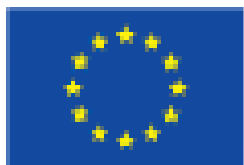
**Step3:** By the second step, we can get the iris encoding of the cows. By calculating the Hamming distance between individuals, we obtain the threshold for individual identification based on statistical analysis of the samples. If the Hamming distance between two images is larger than the threshold, these images are not of the same cow. Otherwise, these two images are obtained from the same individual.

In this research, we acquire images from the dairy farm and design algorithms for iris recognition according to the eye structure of the cow. And we have developed the middleware, through which users can obtain the information of the cow. Then they can locate the cows which carry the infectious disease viruses. Thus we can ensure food safety in the food supply chain of meat products.

**Key words:** food safety, traceability, iris recognition, Middleware, food supply chain







D-91058 **Erlangen**  
Eggenreuther Weg 43  
Telefon: +499131/764-0



D-85764 **Oberschleißheim**  
Veterinärstraße 2  
Telefon: +4989/31560-0



D-97082 **Würzburg**  
Luitpoldstraße 1  
Telefon: +49931/41993-0



D-80538 **München**  
Pfarrstraße 3  
Telefon: +4989/2184-0

[www.lgl.bayern.de](http://www.lgl.bayern.de)

**Bayerisches Landesamt für  
Gesundheit und Lebensmittelsicherheit**  
Eggenreuther Weg 43, D-91058 Erlangen

Telefon: +49 9131 764-0  
Telefax: +49 9131 764-102

Internet: [www.lgl.bayern.de](http://www.lgl.bayern.de)  
E-Mail: [poststelle@lgl.bayern.de](mailto:poststelle@lgl.bayern.de)

Gestaltung & Druck: Osterchrist, Druck und Medien

ISBN 978-3-939652-80-9