Ensuring the Integrity of the European food chain

Stable isotope techniques for verifying the declared geographical origin of food in legal cases – scientific opinion

Federica Camin, Fondazione Edmund Mach, Italy
Markus Boner, Agroisolab, Germany
Carsten Fauhl-Hassek, BfR, Germany
Andreas Rossmann, Isolab, Germany
What is a scientific opinion?

Generally refers to the collection of the opinions of many different scientific organizations and entities and individual scientists in the relevant field.

FI: collection of expert opinions to provide scientific advices to stakeholders in the area of food integrity
FI process

FI stakeholders, Advisory Board, EU Food Fraud network

Topic for scientific opinion

Approval by FI management committee (scientific opinion ToR)

Call for paper (WP1 partners) ↔ Call for paper (FI stakeholders)

Draft

Consultation / peer-review by independent experts

Review by FI management committee

Sign off by FI coordinator

Dissemination
Ensuring the Integrity of the European food chain

First Scientific opinion:
Stable isotope techniques for verifying the declared geographical origin of food in legal cases

Background of SIRA:
• Several application in food authentication (official methods)
• Plethora of research articles on application for determining geographical origin of food

Aims of scientific opinion:
• use in court cases
• feasibility to provide robust evidence for prosecution
Ensuring the Integrity of the European food chain

Terms of Reference

The experts should focus on the following aspects:

- Selectivity, specificity, robustness
- Comparison with alternative methods: advantages/limitations
- Strategies
- Examples of previous uses by the legal system
- Best practice guide for stakeholders
Ensuring the Integrity of the European food chain

Stable isotopes ratios

\[ ^2\text{H} / ^1\text{H}, \ D/\text{H} \]

\[ ^{13}\text{C} / ^{12}\text{C} \]

\[ ^{15}\text{N} / ^{14}\text{N} \]

\[ ^{18}\text{O} / ^{16}\text{O} \]

\[ ^{34}\text{S} / ^{32}\text{S} \]

\[ ^{87}\text{Sr} / ^{86}\text{Sr} \]

Geography

Climate

Agriculture

Geology
### Advantages: 1. Stable isotope ratios methods as official standards

<table>
<thead>
<tr>
<th>Year</th>
<th>Method</th>
<th>product</th>
<th>Method</th>
<th>Isotope Ratio</th>
<th>Fraud</th>
</tr>
</thead>
<tbody>
<tr>
<td>1987</td>
<td>OIV</td>
<td>wine, must</td>
<td>SNIF-NMR</td>
<td>D/H</td>
<td>sugar addition (beet, cane)</td>
</tr>
<tr>
<td>1990</td>
<td>EU Reg 2676/90, encl. 8</td>
<td>wine, must</td>
<td>SNIF-NMR</td>
<td>D/H</td>
<td>sugar addition (beet, cane)</td>
</tr>
<tr>
<td>1991</td>
<td>AOAC 998,12</td>
<td>honey</td>
<td>IRMS</td>
<td>$^{13}$C/$^{12}$C</td>
<td>sugar addition (cane)</td>
</tr>
<tr>
<td>1993</td>
<td>ENV 12140, 13070</td>
<td>fruit juice</td>
<td>IRMS</td>
<td>$^{13}$C/$^{12}$C</td>
<td>sugar addition (cane)</td>
</tr>
<tr>
<td>1995</td>
<td>AOAC 995,17</td>
<td>fruit juice</td>
<td>SNIF-NMR</td>
<td>D/H</td>
<td>sugar addition (beet, cane)</td>
</tr>
<tr>
<td>1996</td>
<td>OIV 2/96</td>
<td>wine, must</td>
<td>IRMS</td>
<td>$^{18}$O/$^{16}$O</td>
<td>addition of water/mislabelling</td>
</tr>
<tr>
<td>1997</td>
<td>EU Reg 2676/90, 822/97</td>
<td>wine, must</td>
<td>IRMS</td>
<td>$^{18}$O/$^{16}$O</td>
<td>addition of water/mislabelling</td>
</tr>
<tr>
<td>1997</td>
<td>ENV 12141</td>
<td>fruit juice</td>
<td>IRMS</td>
<td>$^{18}$O/$^{16}$O</td>
<td>addition of water/mislabelling</td>
</tr>
<tr>
<td>2000</td>
<td>AOAC 2000.19</td>
<td>maple syrup</td>
<td>SNIF-NMR</td>
<td>D/H</td>
<td>sugar addition (beet, cane)</td>
</tr>
<tr>
<td>2000</td>
<td>OIV 71/2000</td>
<td>vinegar</td>
<td>SNIF-NMR, IRMS</td>
<td>D/H, $^{13}$C/$^{12}$C</td>
<td>sugar addition (beet, cane)</td>
</tr>
<tr>
<td>2001</td>
<td>OIV 17/2001</td>
<td>wine, must</td>
<td>IRMS</td>
<td>$^{13}$C/$^{12}$C</td>
<td>sugar addition (cane)</td>
</tr>
<tr>
<td>2003</td>
<td>EU Reg. 2676/90, 440/03</td>
<td>wine, must</td>
<td>IRMS</td>
<td>$^{13}$C/$^{12}$C</td>
<td>sugar addition (cane)</td>
</tr>
<tr>
<td>2003</td>
<td>OIV MA-F-AS314-03</td>
<td>wine</td>
<td>IRMS</td>
<td>$^{13}$C/$^{12}$C</td>
<td>technogenic CO$_2$</td>
</tr>
<tr>
<td>2004</td>
<td>AOAC 2004,01</td>
<td>fruit juice, maple syrup</td>
<td>SNIF-NMR</td>
<td>D/H</td>
<td>sugar addition (beet, cane)</td>
</tr>
<tr>
<td>2006</td>
<td>AOAC 2006,05</td>
<td>vanillin</td>
<td>SNIF-NMR</td>
<td>D/H</td>
<td>synthetic vanillin</td>
</tr>
<tr>
<td>2007</td>
<td>OIV-MA-AS312-07</td>
<td>wine</td>
<td>IRMS</td>
<td>$^{13}$C/$^{12}$C</td>
<td>addition of glycerol</td>
</tr>
<tr>
<td>2011</td>
<td>EU Reg 584/2011</td>
<td>Grana Padano DOP</td>
<td>IRMS</td>
<td>D/H, $^{13}$C/$^{12}$C, $^{15}$N/$^{14}$N, $^{34}$S/$^{32}$S</td>
<td>mislabelling</td>
</tr>
<tr>
<td>2013</td>
<td>EN 16466-1, 2, 3</td>
<td>vinegar</td>
<td>SNIF-NMR, IRMS</td>
<td>D/H, $^{13}$C/$^{12}$C, $^{18}$O/$^{16}$O</td>
<td>water and sugar addition (beet, cane)</td>
</tr>
<tr>
<td>2013</td>
<td>OIV 510, 511/2013</td>
<td>vinegar</td>
<td>IRMS</td>
<td>$^{13}$C/$^{12}$C, $^{18}$O/$^{16}$O</td>
<td>water and sugar addition (cane)</td>
</tr>
</tbody>
</table>
Ensuring the Integrity of the European food chain

Methods usable in official controls

EU Regulation 822/2004

- EU regulation and OIV methods for grape products
- internationally recognised rules or protocols (e.g. CEN)
- methods agreed in national legislation
- scientific protocols, after validation according to ISO 5725:1994 or the IUPAC International Harmonised Protocol
Ensuring the Integrity of the European food chain

Advantages: 2. Reference data set: Isotopic Databanks

Robust databanks:
- updated every year
- methods officially recognized (OIV, CEN, validated)
- several court cases

based on regulations (e.g. EU wine databank)

created by product-specific association or consortia (e.g. AIJN for fruit juices, Grana Padano PDO and Parmigiano Reggiano PDO cheese; asparagus; beef, pork; eggs; olive oil)
Ensuring the Integrity of the European food chain

Court cases

Several for wine – must from 1990:

criminal or civil court

Cheese:

- the consortium suspends the PDO licence for 6 months and asks for a fee.
- the producer can be prosecuted in the court (Legislative Decree no. 297/2004, published in the Official Gazette of 15.12.04)
Ensuring the Integrity of the European food chain

**Limitation**: Isotopic Databanks

yearly update: who pays?

not public

possible confounders (experts)

Alternative: isosscapes (not in the court..)
Ensuring the Integrity of the European food chain

Strategy: Definition of limits and compliance

- selection of reference data
- computing 95% confidence limits (t-student two tailed distribution)

\[
\begin{align*}
\text{Upper limit} & = \text{result} \\
\text{Lower limit} & = \text{uncertainty}
\end{align*}
\]
Ensuring the Integrity of the European food chain

Case specific databanks and court cases

- non standard methods
- non extensive databank

- created within the framework of a project (i.e. the EU projects PURE JUICE and TRACE)
- on requirement, to detect particular fraud cases (i.e. butter circular trade or beluga caviar)
Caviar case

Suspicion of German customs: illegal beluga caviar was imported from the Caspian region and sold on the German market.

Databank: Germany, Italy, France, Iran, Azerbaijan, Caspian sea; COHNS in the sample water and row proteins.

The method was accepted from the court.

The trader was imprisoned for one year + fee of around 150,000 €.
Differentiation of caviar using hydrogen (D/H) and sulfur (34S)
Ensuring the Integrity of the European food chain

Fruit Juices

Databank of EU project PURE JUICE stored by SGF International e.V. (Nieder-Olm, Germany)

Procedure of SGF:

- Non compliance has to be confirmed in two laboratories
- Agreement with producer
- If not, punitive review desist
- Only if producer does not sign it or gives reason for complaints after having signed it, SGF initiates a court case.
Ensuring the Integrity of the European food chain

Good practice guide to create and use a databank in legal system (….to be completed)

Robust databank (update, who pays)

Official standards

Accredited laboratories

Successful court cases also with scientific methods and non extensive databanks

Multi-isotope evaluation, but univariate comparison, uncertainty
Terms of Reference: **what is missing**

The experts should focus on the following aspects:

- Selectivity, specificity, robustness
- Comparison with alternative methods: advantages/limitations
- Strategies
- Examples of previous uses by the legal system*
- Best practice guide for stakeholders*

*: to be completed
The project has received funding from the European Union’s Seventh Framework Programme for research, technological development and demonstration under grant agreement No. 613688.

www.foodintegrity.eu