Multi-component SNIP-IRMS™ : an improved method to detect added water in NFC fruit juices

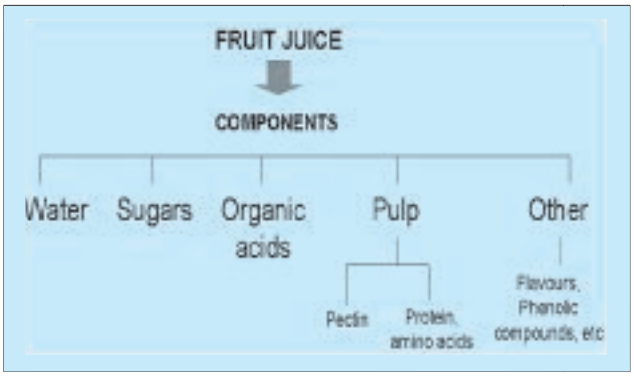

Figure 1 : Multi-isotopic fingerprinting. Determining isotopic ratios on several different components of a fruit juice.

In 2000, fruit juices and nectars made up about 15% of the North American and 11% of the European soft drinks market. In this steadily growing sector of the food industry, the trend towards direct, or not-from-concentrate (NFC), juices is becoming more noticeable year after year. Often sold as short shelf-life products available in supermarket chilled cabinets, NFC juices are to be found at the premium end of the market and respond to increasing consumer demand for natural, healthier products.

Stable isotope analyses are used to detect added water in NFC juices by picking up differences in the $^{18}O/^{16}O$ and $^{2}H/^{1}H$ ratios between the natural fruit water and added tap water. Expected ranges for these isotopic parameters are included in the AIJN[1] Code of Practice, but when the results of isotopic analyses are interpreted, the influence of environmental parameters must be taken into account. Eurofins' most recent analytical development in this area has led to an improved method for the detection of water addition, which has a lower detection limit and a much smaller influence of geographical origin.

The SNIP-IRMS™ (Specific natural isotopic profile) approach is based on multi-isotopic fingerprinting in which isotopic ratios are measured on several components of the same product and their intermolecular isotopic correlation is investigated. For fruit juice, as shown in figure 1, a full isotopic fingerprint can be built up from determinations carried out on the sugar, acid, water, protein and other fractions. As a significant correlation exists between the isotope content of different metabolites of the same natural product, this can be used to highlight the presence of a compound from another source.

To improve the detection of water addition in fruit juices, the ^{18}O deviations of water and organic compounds can be used simultaneously. Due to the recent development of techniques using on-line pyrolysis coupled to IRMS[2], the measurement of $^{18}O/^{16}O$ ratios of organic compounds is now possible. Since the ^{18}O content of the sugars is not affected by the added water, this parameter can be used as an internal reference to detect water addition at lower levels than methods based on the water only. We have recently introduced a refinement of this method which consists in using the ethanol isolated in the course of the SNIF-NMR®[3] procedure in order to get a more robust internal reference. Figure 2 shows the correlation between $\delta^{18}O$ values for the ethanol and the water from authentic orange samples squeezed in the laboratory, and the comparison with market samples of from concentrate and not from concentrate orange juice.

A major advantage of this method is that it provides a means of avoiding false positives. This is illustrated on the same graph by the measurements obtained for NFC orange juice samples taken by the SGF[4]. The results for $\delta^{18}O$ of the extracted water are below the AIJN limit, which based on these measurements alone would lead to the suspicion of added water. The multi-component SNIP-IRMS™ approach, however, shows that the samples fall well within the expected correlated indicating that they are genuine NFC juices.

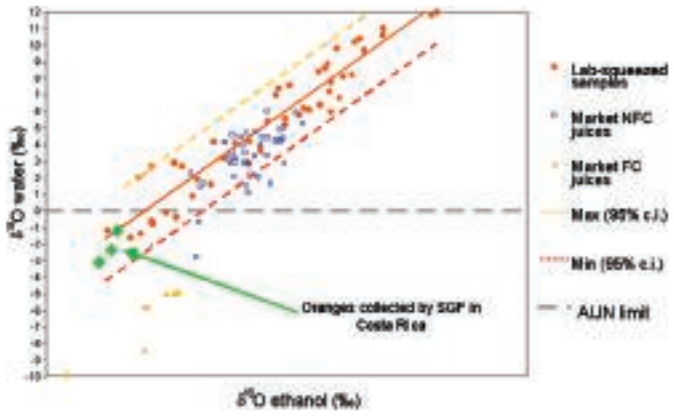

Figure 2 : Correlation between $\delta^{18}O$ values for ethanol and water from authentic and market orange juice samples.

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1 AIJN : Association of the Industry of Juices and Nectars from fruit and vegetables • 2 IRMS : Isotope Ratio Mass Spectrometry
3 SNIF-NMR : Site specific Natural Isotope Fractionation Investigated by Nuclear Magnetic Resonance • 4 SGF : Schutzgemeinschaft der Fruchtsaft-Industrie

2nd Lednice Authenticity Conference

In 2000, the Mendel University of Agriculture in Lednice, Czech Republic, hosted the first authenticity conference of this kind in Eastern Europe. This year, the second conference took place from 29th to 31st August at the same location. These conferences are supported by the European Commission's Human Potential Programme and sponsored by Eurofins to promote the awareness of authenticity issues.

The conference this year not only attracted more attendees than the last one but also had, again, high-calibre speakers from all over Europe, and, for the first time, from Australia.

The conference covered the key topics of
- sample preparation
- authenticity by isotopic methods
- molecular biological methods
- data evaluation and statistics
- image analysis in food.

The keynote speakers at this conference were Gérard Martin discussing General Aspects of Authenticity, Bernd Wenclawiak, comparing different extraction methods including supercritical fluid extraction for vitamin A, Claude Guillou describing the use of NMR and stable isotope ratio methods to protect European consumers, Bert Popping and Katrin Schneede, giving an overview of molecular biological methods used in authenticity testing and Michal Strzelecki presenting work on the use of image analysis to solve authenticity problems.

It was especially noticeable that more interdisciplinary research had been done over the past years providing solutions to authenticity problems that could not be resolved using just one method alone. In particular the combination of physico-chemical (e.g. SNIF-NMR®) with molecular biological (e.g. PCR or GMO PLATINUM ASSAY™) methods showed a very high success rate.

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