Quality Assurance of Fruit Juices in the Modern World

Arab Beverage conference

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Outline

- Eurofins background
- My background
- What is a fruit juice
- Why & when does adulteration occur & past examples
- Useful tools for use in quality and authenticity judgements
- How is it detected
- Conclusions
Facts about Eurofins

- Started in Nantes in 1987 in the University Biochemistry department
- Now employs more than 40,000 staff worldwide
- Operates in 44 countries from 400 laboratories/offices around the world
- https://www.eurofins.com/contact-us/worldwide-interactive-map/
- Turnover in 2017 was just below 3 billion Euros (US $ 2.6 bill)
- Three main pillars
  - **Food and feed testing**
    - Nutritional, allergens, pesticides, authenticity, auditing
  - **Environmental testing**
    - PCB, dioxins, heavy metals, water testing
  - **Pharma**
    - Drug discovery and testing, including clinical trial co-ordination
- https://www.eurofins.com/investor-relations/our-business/
Some of my background!

- Fruit Juice and beverage expert with over 30 years experience
- Cadbury Schweppes for 24 years @ their central research lab in the UK
- Employed by Eurofins for 12+ years
- Vice president of IFU Methods of Analysis Commission
- Vice president of AIJN COP Expert Group
- Chairman of UK British Standards Committee AW 21 for Fruit Juices
- Member of DEFRA Authenticity Methodology Working Group
- Represents IFU at three Codex committees (CCPR, CCMAS & CCCF)
- Member of the Royal Society of Chemistry (Chartered Chemist)
- Member of Association of Official Analytical Chemists (USA)
- Past General Referee for Beverages at AOAC
- Past Chair and board member of US TCJJP
Most suppliers are honest and supply you with what you want.

However, there are a few that have a very "flexible view" of the law.

The good guys need to do everything possible to stop these companies from "queering the pitch" for everyone else.

Any bad publicity about the quality or authenticity of fruit juices will affect ALL the industry not just the "guys" who were caught cheating.
Fruit Juice: Legal definition (GSO Standard 1820)

The unfermented but fermentable liquids obtained from the edible part of sound, appropriately mature and fresh fruit or of fruit preserved in sound condition by suitable means including post-harvest surface treatments in pursuance of the applicable provisions of the Codex Alimentarius Commission. Some juices may be processed with pips, seeds and peel, which are not usually incorporated in the juice, but some parts or components of the pips, seeds and peel, which cannot be removed by Good Manufacturing Practices (GMP) would be acceptable. The juice is prepared by suitable processes, which maintain the essential physical, chemical, organoleptic and nutritional characteristics of the juices of the fruit from which it comes. The juice may be cloudy or clear and may have restored natural aromatic substances and volatile flavor components, all of which must be obtained by suitable physical means, and all of which must be recovered from the same kind of fruit. Pulp and cells obtained by suitable physical means from the same kind of fruit may be added. A single juice is obtained from one kind of fruit. A mixed juice is obtained by blending two or more juices or juices and purées of different kinds of fruit.

Codex fruit juice standard 247, 2005

<table>
<thead>
<tr>
<th><strong>Fruit Juice</strong></th>
<th><strong>Typically</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>ca 90%</td>
</tr>
<tr>
<td>Sugars</td>
<td>0.1 to 12 % (up to 20%)</td>
</tr>
<tr>
<td>Acids</td>
<td>0.3 to 1% (up to 7%)</td>
</tr>
<tr>
<td>Potassium</td>
<td>1000 to 3000 mg/l</td>
</tr>
<tr>
<td>Sodium</td>
<td>trace to 50 mg/l</td>
</tr>
<tr>
<td>Magnesium</td>
<td>40 to 150 mg/l</td>
</tr>
<tr>
<td>Calcium</td>
<td>40 to 150 mg/l</td>
</tr>
<tr>
<td>Phosphorous</td>
<td>40 to 300 mg/l</td>
</tr>
</tbody>
</table>
What types of adulteration occur?

1) Addition of sugars to juice
   *e.g. the addition of sucrose and/or syrups to orange juice, addition of a high fructose syrup to apple juice*

2) Addition of undeclared acids to juice
   *e.g. the extension of lemon juice with synthetic citric acid, apple juice with malic acid or the addition of citric acid to coconut water*

3) Extension of an expensive juice with a cheaper one
   *e.g. extension of raspberry juice with apple juice, pomegranate with cherry, grape, apple .............*

4) Accidental contamination
   *e.g. adventitious mixing of different juices due to poor GMP*
Economics of adulteration ($/T)
prices collected early June 2014

[Bar chart showing prices for various ingredients such as Refined sugar, Raw sugar, HFCS, Glucose syrup, Rice syrup, Agave syrup, Honey China, Honey other, Grape, Orange, Apple, Cranberry, Raspberry, Pineapple, and Pomegranate, with prices ranging from $460 to $12,200.]
When does adulteration occurs?

- When there are cheaper adulterants available (always) (sugar and/or cheaper juice {apple/pear/grape} addition to expensive red/black juices)
- Typically more of an issue when juice prices are high
- When juices are in short supply due to seasonal factors
- There is a much higher risk of problems if someone is not testing their incoming raw materials, as the risk of an unscrupulous supplier being caught is low/negligible!
Samples passing through Nantes Lab & considered as having an unacceptable quality

Note: these statistics are based on the samples analysed by Eurofins Analytics lab. It therefore does not totally reflect the situation on the national markets.
## Sugar concentrations seen in a range of juices

$ can be higher under “special” conditions

<table>
<thead>
<tr>
<th>Juice Type</th>
<th>Sucrose (g/l)</th>
<th>Glucose (g/l)</th>
<th>Fructose (g/l)</th>
<th>Gluc to Fruc ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Orange</td>
<td>40</td>
<td>20</td>
<td>20</td>
<td>0.9 - 1.0</td>
</tr>
<tr>
<td>Pineapple</td>
<td>45</td>
<td>23</td>
<td>23</td>
<td>0.9 - 1.1</td>
</tr>
<tr>
<td>Apricot</td>
<td>30</td>
<td>50</td>
<td>20</td>
<td>1.0 - 3.0</td>
</tr>
<tr>
<td>Apple</td>
<td>10</td>
<td>20</td>
<td>60</td>
<td>0.3 - 0.5</td>
</tr>
<tr>
<td>Blackcurrant</td>
<td>Trace</td>
<td>27</td>
<td>36</td>
<td>0.6 - 0.9</td>
</tr>
<tr>
<td>Blackberry</td>
<td>Trace</td>
<td>40</td>
<td>40</td>
<td>0.95 - 1.05</td>
</tr>
<tr>
<td>Cherry (sour)</td>
<td>5</td>
<td>40</td>
<td>35</td>
<td>1.0 - 1.4</td>
</tr>
<tr>
<td>Cherry (sweet)</td>
<td>Trace</td>
<td>65</td>
<td>60</td>
<td>0.9 - 1.3</td>
</tr>
<tr>
<td>Lemon</td>
<td>Trace</td>
<td>8</td>
<td>7.5</td>
<td>0.95 – 1.3</td>
</tr>
<tr>
<td>Raspberry</td>
<td>&lt;1$</td>
<td>25</td>
<td>28</td>
<td>0.6 - 0.9</td>
</tr>
<tr>
<td>Pomegranate</td>
<td>Trace</td>
<td>60</td>
<td>65</td>
<td>0.8 - 1.0</td>
</tr>
<tr>
<td>Strawberry</td>
<td>6</td>
<td>25</td>
<td>28</td>
<td>0.8 - 0.95</td>
</tr>
</tbody>
</table>
Detection of juice adulteration

- Detection of adulteration of fruit juices is NOT a TRIVIAL matter.
- Best left to expert laboratories
- Requires an extensive database to make “safe” (to prove beyond reasonable doubt) conclusions
- Sometimes its very simple to detect adulteration as it is so blatant
  - Presence of D-malic acid
  - High levels of sucrose in a non-sucrose containing juice
  - ........
- Other times its very complex and requires the use of specialist equipment/interpretation
  - Uses of isotopic methods
  - Use of isotope ratios in different compounds
  - Use of different isotopes within the same compound
A few notable examples of FJ adulteration from around the world
Beech Nut “Apple juice” intended for babies

- Their “apple juice” had very unusual data
- Normally apple juices show a glucose to fructose ratio (G/F) between 0.3 to 0.5 & also show low but detectable levels of chlorogenic acid (a “polyphenol” seen in apples)
- However, in their product
  - The G/F ratio was 1.11
  - No chlorogenic acid was detectable

- Showed that it contained little if any apple juice!
- Company fined $2 million and agreed to pay $140K towards the FDA’s costs for the sale of adulterated apple juice for over a two year period.
- They also settled a class action suite for $7.5 million.
- The CEO & VP manufacturing were jailed for their actions
In late 80’s/early 90’s there was considerable concern that orange juice on sale in the UK was not what it should be!

The UK Industry was trying to control the quality of the products on sale by using its own independent screening method.

In late 1990 MAFF collected 21 retail orange juices and had them analysed in four laboratories around the world (Canada/USA, France & Germany) using a range of different procedures.

Data showed that 16 out of 21 samples did not conform to their label as 100 % orange juice.

Most of these products were supermarket own label materials but there were some branded products too.

[Newspaper article]
German RSK system (multi-component analytical method)
- Including D-malic acid

SNIF-NMR® (Eurofins’ isotopic method that detects the addition of beet sugars to juice by the application of D-NMR & cane/corn sugars by IRMS

Matrix method of ACSC from Maryland USA (multicomponent analytical method, similar to RSK but using different parameters and judgement criteria)

Oligosaccharide profiling (High pH anion chromatography linked with pulsed amperometric detection {HPAEC-PAD})
- Detects low levels of tri-saccharides formed during the hydrolysis of beet or cane sucrose at high Brix levels
- Invert syrup markers
HPAEC-PAD screening for sugar syrups

Covered by IFU Recommendation # 1

Pure apple juice

Pure orange juice

BMIS

BMIS marker IV

Maltose

Oligosaccharides

HFCS
Over the last few years coconut water has become an extremely popular beverage.
Sales in the UK alone in 2016 were estimated to be worth more than £100 million ($130 mill).
Global sales are predicted to be worth over $9 bn by 2019!!
As with most “new juice” products, there is always a higher risk of adulteration in the early stages of development of the product, e.g. when it is popular but before there has not been sufficient time to develop a robust database by which to judge the authenticity of products!
In 2014 ITI (USA trader) looked at 20 US retail samples of CW and 12 of these were found to be adulterated with sugar and/or contained undeclared preservatives!
In 2016 UK’s National Crime unit, a branch of the Food Standards Agency, sized 12 samples of CW at a port in the UK.
Analysis of these products showed that 7 of them were adulterated with added sugar & imports were “blocked at the boarder”.
Other studies in the UK and Europe (European Quality control scheme) has also shown issues with the authenticity of this product
Other issues with coconut water

- In these studies some of the products, described as 100%, have shown δ\(^{13}\)C values as high as -14 to -15 ‰!!

- However, as coconuts use the normal C\(_3\) pathway to fix CO\(_2\) dioxide from the atmosphere they would be expected to show δ\(^{13}\)C values around -24 ‰.

  - During very dry seasons real samples have shown a few samples as high as -21.3 ‰!

- The values detected would indicate that more than 50 % of the sugars in the product are derived from a C\(_4\) plant (cane/corn).

- Some samples have also been found to contain elevated levels of citric acid, without declaring its addition.

- Abnormally high levels of ascorbic acid have also been detected in some products, added Vit without declaration.

- Some samples were also shown to contain an undeclared preservative sulfur dioxide.

- AIJN published a provisional reference guide for coconut water in May 2017 to help improve the quality of coconut waters on sale in the EU.

  [www.aijn.org](http://www.aijn.org)
Other examples of juice adulteration

1. Early 90’s Californian company was selling adulterated orange juice. Company settled out of court ($8 million)
   - Detected by: very low oxygen isotope ratio & presence of naringin (grapefruit)

2. 2001 Apple juice with added synthetic L-malic acid
   - German supplier jailed.
     - Detected by: Internal $^{13}$C isotopic method

3. 2003 Evaporator water added to mixture of NFC & FC (Brazil) OJ
   - Detected by: $^{18}$O and $^{87}$Sr (country of origin) isotopic methods

4. 2010 Evaporator water detected in European NFC orange juice
   - Detected by Internal $^{18}$O isotopic method
Two useful tools
Two useful tools for making judgements on fruit juices

- IFU has published a wide range of analytical methods that have been tested & validated on fruit juices
  - Makes for ease of implantation of the method in your own lab
  - Many of the methods are accepted as part of the “Codex System” and those are listed in Codex Stan 247 (fruit juice) & 234 (approved methods)
- AIJN Code of Practice
  - Lays down quality and authenticity criteria for popular fruit and vegetable juices to assist in assessment of these products
International Fruit Juice Union (IFU) methods

- IFU Methods of analysis commission (MAC) is made up of ca 20 experts from various regions of the world
- The Commission validates procedures for the quality control and authenticity assessment of fruit and vegetable juices using the principles given in ISO 5725
- There are around 40 to 50 validated chemical methods for use in fruit juices
- 12 proposed microbiological methods for use in fruit juice products
- Provides the best set of validated method for fruit juices
  - Available to members at the IFU website www.IFU-fruitjuice.com or can be purchased by non-members for 1 yr for 2000 Euros
  - or join IFU for 1999 Euros and gain access to the methods for 4 people & lots more!
    - More info can be found here www.IFU-fruitjuice.com
    - Or contact John Collins Executive Director at John@ifu-fruitjuice.com
- Other methods available, such as AOAC but much more limited selection tested on FJ www.AOAC.org
Published a number of reviews/recommendations on specific topics; recent developments:

- Updated of the review of isotopic methods (IFU Rec 3)
- Review of DNA methods used for fruit juices (IFU Rec 13), methods to assess the organic nature of a FJ (IFU Rec 14), Simple Quality assurance for a FJ lab (IFU Rec 15)
- Working on updating the recommendation on patulin analysis (IFU 2)
- Use of polyphenols in fruit juice assessments (IFU Rec 11), methods to confirm country of origin of FJ, (IFU Rec 12), use of $^1$H-NMR as a rapid method to assess FJ (IFU Rec 19), Review of methods for pesticide residue analysis (IFU rec 16), Aroma analysis of fruit juices (IFU Rec 17)
This is a voluntary industry code of practice which lays down criteria that a specific fruit juice should conform to.

- Prepared by *ca* 20 experts taken from Europe, US, Brazil, Russia.
- Data is collected from authentic materials from the main processing sources from at least 3 seasons before a RG can be prepared and the RGs are reviewed on a regular basis.
- Can be updated with additional data, if required, from new sources with values from at least three separate processing seasons.
- **COP contains 27 reference guides for fruit and vegetable juices/purees**
  - Orange, apple, pineapple, grapefruit, grape, lemon, pear, mango, guava, raspberry, blackcurrant, strawberry, mandarin, carrot, tomato, passion fruit, peach, banana, apricot, acerola, peach, pomegranate, cranberry, kiwi, aronia, lime and coconut water.
- References to methods, legislation, industry positions etc.
Problems and their detection
Pomegranate Juice
Problems seen with pomegranate (1)

- **Addition of sugar**
  - Presence of high levels of sucrose up to 22 g/l!
  - Shift in carbon isotope ratio $\delta^{13}C$ to as high as -13.8 ‰ (80% C$_4$Sugars)
  - Shift in (D/H)$_1$ to as low as 95 ppm (70% beet sugar!!!)
  - Presence of unusual oligosaccharides

- **Detection by**
  - Conventional sugar analysis (enzyme/HPLC) (IFU 55, 56 & 67)
  - SNIF-NMR® isotopic method (IFU rec 3, AOAC 995.17)
  - Fingerprinting methods Cap-GC (IFU rec 4)

- Useful paper published by Dr. Ara in Fruit Processing on pomegranate in 2007 July/August edition pages 204 – 213
- AIJN Reference guide # 21 for pomegranate juice
Typical Pomegranate profile

- Delpin-3,5-digluc
- Cyan-3,5-digluc
- Delpin-3-gluc
- Cyn-3-gluc
- Unknown

Pomegranate plus black carrot

- Acylated “cyanidins”
Apple juice
Cap-GC for "normal" AJ

Sucrose
Cap-GC AJ with Inulin derived syrup (HIS)
Cap-GC for AJ with Starch derived syrup (HFCS)

Marker peaks

Covered by IFU recommendation # 4
Isotopic effects

- Isotopic effects depend on biological & physical systems differentiating between light and heavy isotopes e.g. $^2\text{H}$ (D) & $^1\text{H}$, $^{13}\text{C}$ & $^{12}\text{C}$, $^{18}\text{O}$ & $^{16}\text{O}$
- During some processes the light & heavy isotopes react differently, which gives a target substance (e.g. water/sugar/acid) that is enriched or depleted in the heavy isotope
- Three examples given here together with an application:

<table>
<thead>
<tr>
<th>Feature</th>
<th>Use</th>
</tr>
</thead>
<tbody>
<tr>
<td>1) Preferential loss of H and $^{16}\text{O}$ in water evaporated from the leaves</td>
<td>Differentiation of NFC from FC juice</td>
</tr>
<tr>
<td>2) D level concentrated in the water/components of some fruits</td>
<td>Detection of the addition of beet sugar to juice</td>
</tr>
<tr>
<td>3) Plants fix CO$_2$ by two different routes:- C$_3$ plants have a large carbon isotope effect so $\delta^{13}\text{C}$ values are around -25 permil for sugars C$_4$ plants show a smaller effect so $\delta^{13}\text{C}$ values are around -11 permil for sugars</td>
<td>Detect the addition of C$_4$ (cane/corn) sugars to normal juices</td>
</tr>
</tbody>
</table>

Covered by IFU Recommendation # 3
Basis of $^{13}$C-IRMS testing

**C$_3$ Route** *(Sugars ca -25‰) used by most plants*

Ribulose-6-bisphosphate + CO$_2$  
(5 carbon unit)  
2 x phosphoglyceric acid  
2 x (3 carbon units)

*Typical signature seen in fruits, honey & maple syrup syrups derived C$_3$ starches (e.g. RICE) & beet sucrose or beet invert*

**C$_4$ Route** *(Sugars ca -10‰) used by grasses (cane/corn)*

Phosphoenolpyruvate + CO$_2$  
(3 carbon unit)  
oxaloacetic acid  
(4 carbon unit)

*Signature seen in cane/corn adulterants*

**CAM Route** *(Sugars ca -11 to -15‰) used by succulents (pineapple & agave)*

C$_3$ route in the daylight to make energy (ATP & NADPH)  
C$_4$ route in the dark to make sugars

*Signature seen in pineapple, agave and prickly pear*
(SNIF-NMR®) detection of added sugar to juice

Site-specific Natural Isotope Fractionation studied by NMR (SNIF-NMR®) (AOAC 995.17)

Has two parts:

NMR

Detects beet derived sugars in fruit juices.

Sugars fermented into ethanol in a controlled manner. The ethanol (CH$_3$CH$_2$OH) is recovered and the level of deuterium (D/H)$_1$ at the methyl (CH$_3$) and (D/H)$_{1,1}$ at the methylene (CH$_2$) sites is measured.

Carbon isotope ratio mass spectrometry (C-IRMS)

Detects cane and corn derived sugars in fruit juices.

Sample combusted to give CO$_2$ and the proportions of $^{12}$C and $^{13}$C are measured in an isotope ratio mass spectrometer.
SNIF-NMR® for the detection of sugar addition

$\delta^{13}C$  

C$_4$  

C$_3$  

Beet  

Fruit reference Zone  

Unknown  

Cane  

Low D  

High D  

$\left(D/H\right)_1$
Problems with NFC orange juice
Isotopic analyses in direct fruit juice: Improved detection of water addition

NFC JUICE

COMPONENTS

Water

Sugars

Fermentation ⇔ Ethanol

CH$_2$CH$_3$OH

$^{18}$O/$^{16}$O

$^{2}$H/$^{1}$H

$^{18}$O/$^{16}$O

$^{13}$C/$^{12}$C

$^{2}$H/$^{1}$H
Internal isotopic detection of added water

![Graph showing isotopic composition of ethanol and water samples, with data points for Lab-squeezed samples, Market NFC juices, Market FC juices, Authentic orange from a specific origin, FJ from Concentrate, and the AIJN limit. The graph includes a regression line for water addition to NFC.]

Problems were noted in the States in 2013 with lemon juice as a condiment

Problems were seen with the addition of citric acid derived from C₄ sources (cane/corn)
- Easy to detect using IRMS
- Levels of adulteration ranged from 10% up to 90%!!!

Problems were also noted with addition of citric acid derived from a C₃ source
- More difficult to detect but possible using a internal isotopic method
Detection of Lemon Juice adulteration
Use of polyphenols in FJ analysis

- With red/black juices use IFU 71 to “look” at the anthocyanins in these types of juice to look for added colour and/or another red/black juice
- IFU 58 can be used to look at the flavonoid glucosides
- There have also been problems with the substitution of lime for lemon as well in this product
- This addition can be picked up by looking at the pattern of polymethoxylated phenols (PMFs)

- All of these topics are covered in the IFU Recommendation # 11 which is to be published shortly (in preparation)
Substitution of lime for lemon detected by PMFs

Lemon control

Lime control

Labelled as lemon!!!
Although it is accepted to mix in 10% mandarin juice to OJ by the codex FJ standard. In the EU this would have to be called a mixed juice.

Therefore there is the need for methods to detect this and DNA offers the best approach sometimes.

The “juice” matrix is not an easy one for DNA methods.

- pH & heating all can cause degradation of the DNA!!!
- Works in some cases but not all
- Not in lemon and lime the products are too acidic!!

See IFU recommendation number 13 for further information.
1) For years “juice” chemists have been on a mission to find the ultimate method that is:
- Quick to apply
- Cheap to utilise
- Able to detect all adulterations

2) Tried:
- FT mid range Infra Red spectroscopy (FT-IR)
- Near Infra Red Spectroscopy (NIR)
- Pyrolysis mass spectrometry (Py-MS)
- UV/Visible and fluorescence spectroscopy

3) However, all have failed in time to deliver early promise
1H-NMR for Screening Fruit Juices

Fruit Juice Authenticity Screening by High resolution $^1$H-NMR analysis
Principle of a 1H NMR test on juice

Sample preparation:
- Centrifugation
- Addition of a suitable buffer
- Filtration
- NMR tube

15 min NMR measurement

Automatized spectrum treatment
A rapid screening method has been developed over the last 10 years that “looks” at the total organic chemistry of a range of fruit juice.
Expansion of spectrum for quantification

See IFU Recommendation # 19 (in preparation)
In the following tables the results of the quantitative analysis are given and compared to the A.I.J.N. reference ranges (if available). For concentrated products, results are expressed for juice strength.

- consistent with A.I.J.N.
- outside the A.I.J.N. limits (± 10%)
- outside the A.I.J.N. limits
- no A.I.J.N. reference range

N/Q: Not quantified (not detected or insufficient signal assignment)

### Quantification Results:

<table>
<thead>
<tr>
<th>Compound</th>
<th>Result</th>
<th>Unit</th>
<th>Flag</th>
<th>min</th>
<th>max</th>
<th>SGF-Profiling</th>
</tr>
</thead>
<tbody>
<tr>
<td>5-hydroxymethylfurfural</td>
<td>N/Q</td>
<td>mg/l</td>
<td>-</td>
<td></td>
<td>20</td>
<td>0 - 36</td>
</tr>
<tr>
<td>D-galacturonic acid</td>
<td>12</td>
<td>mg/l</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0 - 1950</td>
</tr>
<tr>
<td>acetaldehyde</td>
<td>6</td>
<td>mg/l</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>not detectable</td>
</tr>
<tr>
<td>acetone</td>
<td>&lt;5</td>
<td>mg/l</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>not detectable</td>
</tr>
<tr>
<td>alanine</td>
<td>38</td>
<td>mg/l</td>
<td>-</td>
<td>1</td>
<td>50</td>
<td>10 - 53</td>
</tr>
<tr>
<td>arbutin</td>
<td>N/Q</td>
<td>mg/l</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>not detectable</td>
</tr>
<tr>
<td>benzaldehyde</td>
<td>N/Q</td>
<td>mg/l</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>not detectable</td>
</tr>
<tr>
<td>benzoic acid</td>
<td>N/Q</td>
<td>mg/l</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>not detectable</td>
</tr>
<tr>
<td>chlorogenic acid</td>
<td>110</td>
<td>mg/l</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0 - 227</td>
</tr>
<tr>
<td>citramalic acid</td>
<td>59</td>
<td>mg/l</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>3 - 134</td>
</tr>
<tr>
<td>citric acid</td>
<td>N/Q</td>
<td>g/l</td>
<td>-</td>
<td>0.15</td>
<td>-</td>
<td>not detectable</td>
</tr>
<tr>
<td>ethanol</td>
<td>196</td>
<td>mg/l</td>
<td>-</td>
<td>3000</td>
<td>-</td>
<td>0 - 239</td>
</tr>
<tr>
<td>formic acid</td>
<td>14</td>
<td>mg/l</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0 - 37</td>
</tr>
<tr>
<td>fructose</td>
<td>60.4</td>
<td>g/l</td>
<td>-</td>
<td>45.0</td>
<td>85.0</td>
<td>not detectable</td>
</tr>
<tr>
<td>fumaric acid</td>
<td>N/Q</td>
<td>mg/l</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0 - 52</td>
</tr>
<tr>
<td>glucose</td>
<td>15.9</td>
<td>mg/l</td>
<td>-</td>
<td>15.0</td>
<td>35.0</td>
<td>47.9 - 77.9</td>
</tr>
<tr>
<td>lactic acid</td>
<td>8</td>
<td>mg/l</td>
<td>-</td>
<td>500</td>
<td>-</td>
<td>0 - 54</td>
</tr>
<tr>
<td>malic acid</td>
<td>5.9</td>
<td>g/l</td>
<td>-</td>
<td>3.0</td>
<td>-</td>
<td>2.2 - 9.8</td>
</tr>
<tr>
<td>methanol</td>
<td>17</td>
<td>mg/l</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0 - 104</td>
</tr>
<tr>
<td>pyruvic acid</td>
<td>&lt;5</td>
<td>mg/l</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>1 - 35</td>
</tr>
<tr>
<td>quinic acid</td>
<td>402</td>
<td>mg/l</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>145 - 1102</td>
</tr>
<tr>
<td>sorbic acid</td>
<td>N/Q</td>
<td>mg/l</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>not detectable</td>
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<tr>
<td>succinic acid</td>
<td>19</td>
<td>mg/l</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>5 - 77</td>
</tr>
<tr>
<td>sucrose</td>
<td>25.9</td>
<td>g/l</td>
<td>-</td>
<td>5.0</td>
<td>30.0</td>
<td>0.5 - 34.6</td>
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### Calculated Values:

<table>
<thead>
<tr>
<th>Figure</th>
<th>Result</th>
<th>Unit</th>
<th>Flag</th>
<th>min</th>
<th>max</th>
<th>SGF-Profiling</th>
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</thead>
<tbody>
<tr>
<td>Glucose/Fructose ratio</td>
<td>0.26</td>
<td>-</td>
<td>-</td>
<td>0.30</td>
<td>0.50</td>
<td>0.22 - 0.56</td>
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<tr>
<td>% Sucrose</td>
<td>25</td>
<td>%</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>7 - 36</td>
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<tr>
<td>Total Sugar</td>
<td>102.2</td>
<td>g/l</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>62.4 - 122.0</td>
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<tr>
<td>Malic-/Quinic-Acid ratio</td>
<td>14.8</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>4.3 - 33.3</td>
</tr>
</tbody>
</table>
NMR “pulls out” adulterated orange juice

Further information can be found in the IFU recommendation # 19 which is due for publication in 2019
Conclusions

- Most suppliers are honest but not **ALL** are!
- Due to price of juices there is always a risk of adulteration
- Test raw materials from unknown suppliers well using a range of approaches
- If possible only use suppliers that you have visited and audited or have been approved by a third party (such SGF IRMA)
- This should not preclude you from testing these suppliers also
- Everything has a price and if the price is **TOO** good there **maybe** a very good reason why!
- **It takes a long time and a large amount of money to build a brand** but it can be lost very quickly with bad press coverage or Government action **so you need to protect it!**
Thank you for your attention.

Any questions?

If you would like more information please feel free to contact us at davidfruitjuice@aol.com or visit www.Eurofins.com.
Useful reference data

AIJN code or Practice Published by AIJN, www.aijn.org
Methods to Detect Adulteration of Fruit Juice Beverages Vols. 1 Edited by Nagy & Wade. Published by AgScience, Auburndale, Florida, USA. (ISBN 0-9631397-3-8)
Best collection of validated methods of analysis for fruit juices. International Fruit Juice Union Methods of Analysis Handbook, Available from the IFU Website as a download for a fee; www.ifu-fruitjuice.com
AOAC analytical methods available to members as a download from the AOAC site (more limited collection of procedures for fruit juices) www.AOAC.org
Fruit Processing & Flussiges Obst are very good magazines for juice related data and processing issues published monthly by Flussiges Obst, Schonborn, Germany.

and of course Eurofins for all your analytical/consultancy needs

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