The detection of added foreign sugar

Mikko Hofsommer, expert for fruit juices and related products entitled by the Chamber of Commerce and Industry and general manager of the company GfL, offers some insight into the technology that aims to counteract the fraud that is 'foreign sugar'.

As concentrates are paid on the basis of brix values it is, and always has been, a logical manipulation by fraudulent suppliers to add sugar to juice concentrates as it is the cheapest way to increase brix. The same is true also for juices as an addition of water is otherwise limited to the minimum density of a juice.

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There are several types of sugar discussed in this article, as well as the different detection strategies required when checking products for their authenticity, as for example, stipulated in the IFS standard.

In the first place of course there is the analysis of the major sugars (glucose, fructose, sucrose) themselves. Their presence, e.g. sucrose in grape, deviation glucose/fructose ratios and total content in conjunction with the sugar-free extract (other components than the sugar contributing to the Brix) can already give strong indications for a manipulation. However as just said there is not only one type of sugar accordingly it is fairly easy for a fraudster to 'pick the right kind' (sucrose, medium invert beet, high fructose corn syrup and so on) which will make the addition unnoticeable.

A second strategy is based on the concept to identify minor components which originate from the added foreign sugar.

Here the procedure according Prof. Low sometimes referred to Low-GC or oligosaccharide profile must be mentioned (see IFU recommendation No 3). The classical invert sugar for example can be identified by marker compound IS1 and IS2 or starch derived syrups by detecting maltose and iso-maltose. Unfortunately however not all types of sugars can be detected via this method. But there is ongoing research to identify further minor components which advanced technologies.

The third analytical strategy is the analysis of stable isotopes. One must differentiate between carbon ($\delta^{13}C$) and hydrogen ($\delta^{2}H$) isotopes (see figure 1).

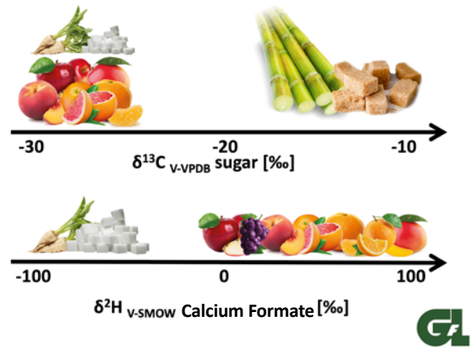
The 13C concentration of heavy carbon is not the same in all plant but depends first and foremost of the plants metabolism more precisely the way carbon dioxide from the air is captured to produce biomass / sugar.

There are three different groups. C3, C4 and so called CAM plants. For the interested reader IFU recommendation No 3 (2017) may be suggested. What is important to know that most fruits used in juice production are C3 plant and some plants used fro making sugar (eg. cane or corn) are C4 plants. Thus the analysis of 13C allows the detection of one in the other.

Sugar beet however is also a C3 plant and accordingly cannot be detected via carbon isotopes. Here one needs to look at hydrogen. The classical procedure for this is the so called SNIF NMR developed by Eurofins in the 1980s. The procedure requires a fermentation of the juice, separation

Refractometer VariRef from Schmidt+Haensch GmbH & Co

Figure 1. $\delta^{13}C$ and $\delta^{2}H$ values for different fruits/ sugars



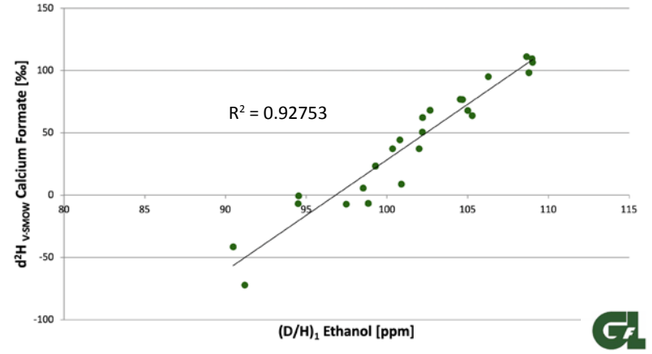
of the produced ethanol and analyze of site specific hydrogen isotopes via NMR. The method has been well accepted and reference values can be found in the AIJN Code of Practice.

We have in the past years developed an alternative based on the so called Krueger Formate a concept from Dana Krueger from KFL [Krueger, A. D.; "Detection of Added Sugar to Fruit Juices Using Carbon and Hydrogen Stable Isotope Ratio Analysis" Methods to detect adulterations of fruit juice beverages, Vol 1, p. 41-51, 1995]. Here sugars are oxidized to formic acid, this is separated via a steam distillation, precipitated as calcium formate, dried and can then be measured with an IRIS/ IRMS. The expanded measurement uncertainty was found to be +/- 11‰.

Ultimately we conducted a comparative study with the German Bundesinstitut für Risikobewertung (the federal institute for risk assessment) which is also the senior expert office for the import control of wine and as such well experienced with the SNIF NMR procedure.

The results are given in figure 2. In total 20 samples, different juices/ purees/concentrates commercial ones, some adulterated, some produced by ourselves were analyzed with both methodologies. Furthermore the results of four proficiency test are included. As one can see the methods show a good correlation in thus allowing to convert the findings from one to another.

Figure 2. Correlation of Krueger-Formates with SNIF-NMR $(D/H)_1$ Ethanol values in different types of fruit juice



At this point it should also be noted that neither of the methods is particularly useful to detect beet sugar is some fruits with low values, such as strawberry. In these specific cases more research work has to be done.

Summing up there is not one method to detect foreign sugar but different approaches which are not all similarly successful in all cases.

Last not least I want to address the special challenge of sugar detection in CAM plants. Of commercial relevance are pineapple, prickly pear and agave. The sugar detection in these plants is a different chapter and the details on this will be addressed in a later article.