

QSI NMR Analysis of Agave syrup – Database version 2.4

Since 2017, QSI offers NMR to evaluate the authenticity of agave syrup. The method is fully accredited according to our flexible ISO 17025 accreditation and is a very powerful tool in the detection of adulteration. With NMR almost all organic substances can be seen at once with a single experiment. Altogether a fingerprint of agave syrup consisting of signals from sugars, amino acids, organic acids, aromatic compounds, etc. is generated. Combining fingerprints from a database of authentic samples leads to the basis of our evaluation. Each deviation of the sample in question from this database can be easily recognized and therefore adulteration or quality issues can be detected (see Figure 1).

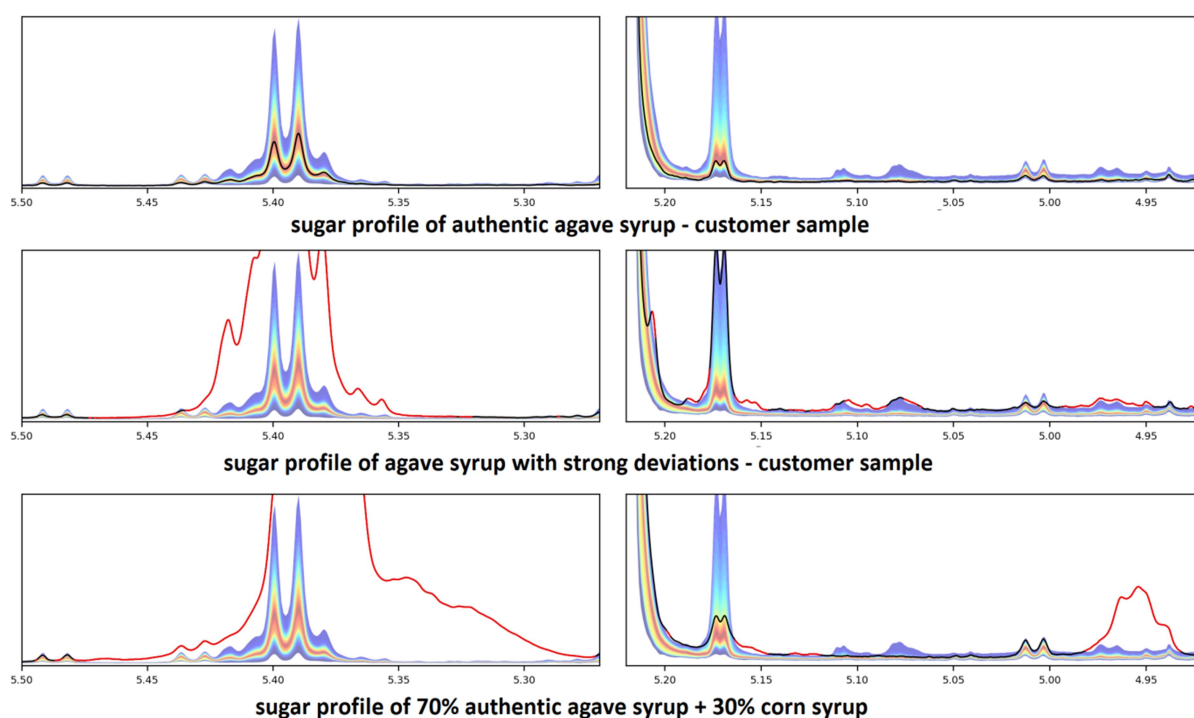


Figure 1 Comparison of parts of the sugar profile (part of the NMR spectrum) of three different agave syrup samples. The top sample is an authentic agave syrup sample. The middle one shows a possible adulteration of a real customer sample with strong deviations in the sugar profile, i.e. elevated sucrose/mannose/oligosaccharides. The bottom sample has been spiked with 30% corn syrup and shows also deviations of sucrose/oligosaccharides. In all pictures the bold black/red line represents the spectrum of the actual sample and the coloured background the current agave syrup database. The black line turns red, when it is exceeding the database. Hence, deviations are easily recognizable.

We have started building our database in 2015 and have in January 2018 released database version 2.4 which holds almost 1000 authentic agave syrup samples. The database is updated routinely every few months to incorporate new developments in syrup processing, new producers as well as different agave varieties, ages and regions in Mexico. All samples within the database have been authenticated with LC-IRMS as well as for dark samples we performed additional tests for potential colorant E150d. Furthermore, all samples were inspected visually by NMR and different quantified parameters were evaluated according to the NOM-003-SAGARPA-2016 with a few adjustments. In the following table you can find information about the database.

Database parameters

Version	2.4
Total Samples	968
Varieties	Salmiana, Tequiliana
Mexican Producers	4
Age of Agave	4 to 7 years
Harvest years	2011 - 2018
Origin of Agave	Jalisco, Michoacán, Nayarit, Sinaloa, Tamaulipas, Zacatecas

In some instances we felt after careful consideration the need to improve the current restrictions of the NOM-003-SAGARPA-2016. We have already discussed our thoughts regarding the NOM and current testing methodologies in a previously published white paper.

A good example for a difference in our analysis from the NOM is HMF: The NOM states a maximum HMF concentration of 7000 mg/kg for agave syrup which we have found was way too high. HMF concentrations range in almost all cases (>95% of all samples) from 0 to 150 mg/kg. For all samples we measured (also adulterated ones) we have found that only 37 showed an HMF level > 200 mg/kg, more than half of these samples were already adulterated according to LC-IRMS and the other half showed in many cases multiple deviations aside from HMF in their NMR spectrum. Hence, we have adopted 200 mg/kg as limit for our analysis. Higher values are indicated as deviations on the analysis report with a corresponding comment. We have done such analysis for multiple parameters that can be derived from the NMR spectrum and thus optimized the analysis steadily and improved the database accordingly. Just using all the NOM parameters almost 50% of all samples would fail the analysis, since e.g. for the agave variety Salmiana glucose concentrations almost always exceed 12 g/100g, while fructose concentrations can be as low as 50 g/100g. However, in other cases, e.g. the minimum glucose concentration (3 g/100g), we are in agreement with the NOM.

Changes of the processing of the syrups can lead to deviations in the NMR spectrum, which we have experienced multiple times. Hence, we demand from the producers we work with to always report changes in the processing beforehand, so we can prepare for eventual changes in the spectrum and perform according experiments to verify the changes before extending our database.

Overall, we think that NMR is currently the best method to analyse agave syrup in regard to adulteration detection and we recommend using it in combination with LC-IRMS. We offer each of these analyses of agave syrup (NMR – Code 40600; LC-IRMS – Code 40267) for 150 € per sample.

Please do not hesitate to contact us if you have any further questions on this subject.

Best regards

Quality Services International GmbH