JRC Scientific and Technical Reports

Analysis and Characterisation of Alcoholic Products (ACAP)

FISCALIS Project Group – Final Report

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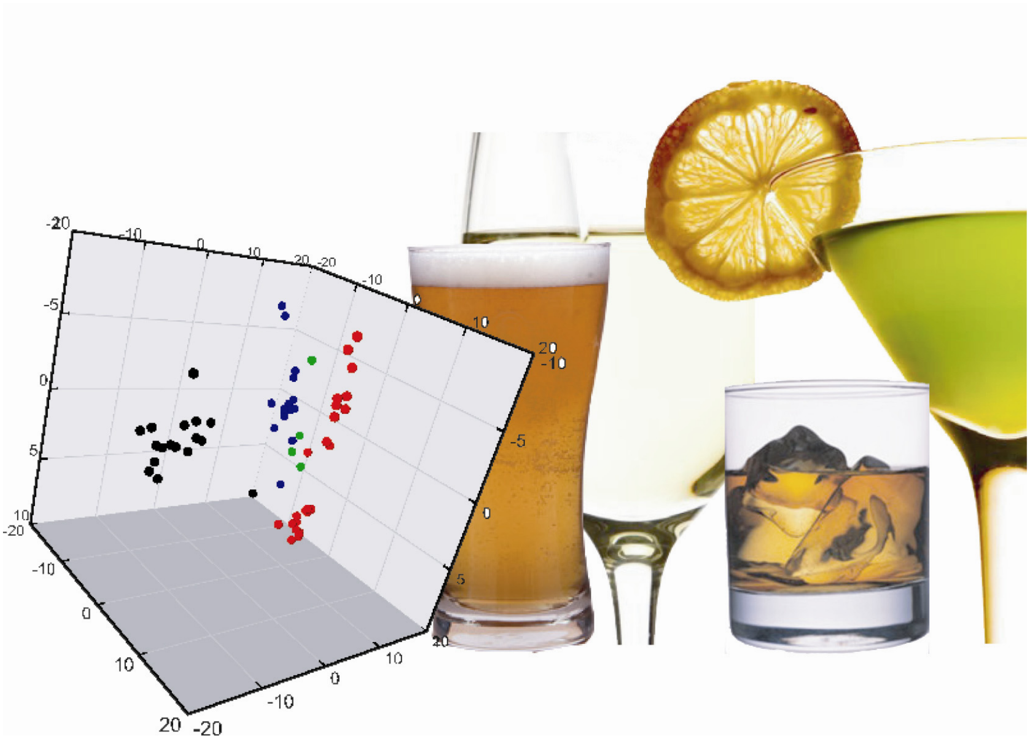

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ABSTRACT:

The purpose of the ACAP working group (composed of JRC, TAXUD and 11 Member States Custom Laboratories) was to provide scientific support for the resolution of the issue of divergent classifications of alcoholic beverages for excise duties, whether by providing objective criteria for classification, or providing sound scientific information for revision of legislation. In particular, there is a need for analytical methods allowing for the differentiation between "fermented" and "distilled" ("non-fermented") beverages and for the detection of ethanol addition. At present there is no commonly agreed solution of these problems which has resulted in the Customs Laboratories of the Member States developing their own particular approaches to deal with these issues. This can lead to divergent classification between Member States and consequent problems both for administrations and the trade.

Two complementary pathways of investigation have been examined in this study: NMR fingerprinting and "classical analysis", both handled by multivariate data analysis.

It is the findings of this study that it is likely to be extremely difficult or even impossible to create workable “general” models based on analytical methods. Unfortunately, the difficulties are the greatest precisely with those categories that are the most problematic for excise classification purposes. Even if an analytical model was eventually developed it is likely to need to be an extremely complex mechanism in order to take account of the broad range of beverages, and diverse chemical characteristics, of products that fall within the problem categories ("other fermented beverages" etc.). On the issue of 'added alcohol' the findings reinforce that is not plausible to ascertain the amounts by a scientific study of the finished product only. The case-to-case approach to classification applied by Member States remains therefore the only possible approach in the context of the current legislation.

CONTENT

The JRC was contacted by DG TAXUD and asked for expertise on the characterisation of alcoholic beverages for excise duties. In particular, there is a need for an analytical method allowing for the differentiation between "fermented" and "distilled" ("non-fermented") beverages.
To this aim, an administrative arrangement (Taxud/2006/DE/321) between DG TAXUD and JRC has been concluded and a working group, including MAST (PCE-IHCP, JRC), DG TAXUD and the Customs Laboratories from Finland, The Netherlands, Belgium, France, Germany, Poland, Czech Republic, Hungary, Italy, Greece and Portugal has been set up.

I. RATIONALE

The purpose of the ACAP working group is to provide scientific support for the resolution of the issue of divergent classifications of alcoholic beverages for excise duties, whether by providing objective criteria for classification, or providing sound scientific information for revision of legislation.

As a reminder, directive 92/83/EEC classifies alcoholic beverages into 5 categories, each of them having references to one or more Combined Nomenclature codes (Council Regulation 2658/87/EEC):

I. *Beers*, with references to CN 2203 and 2206 (mixture of beers with non-alcoholic drinks)
II. *Wines*, with references to CN 2204, 2205 and specification that the alcohol content must result entirely from fermentation.
III. *Other fermented beverages*, with references to CN 2204 and 2205 (if not concerned by cat. II) and CN 2206. In directive 92/83/EEC, the addition of alcohol is allowed at least for beverages having an actual alcoholic strength by volume of between 1.2 % and 10 % (13% for sparkling beverages). The HS Explanatory Notes to 2206 also allows for fortified fermented beverages to remain classified in this heading "when they retain the character of products falling in this heading".
IV. *Intermediate products*, with references to CN 2204, 2205 and 2206 (if not classified in I, II or III) or products with an alcoholic strength that does not entirely result from fermentation) and not exceeding 22% alcoholic strength.
V. *Ethyl alcohol*, with references to CN 2207 and 2208 or products from 2204, 2205 and 2206 with an alcoholic strength greater than 22%.

Most of the problems raised concern the distinction between products of CN codes 2206 (including other fermented beverages) and 2208 (including spirits) and are mainly two:

i. The extent to which a fermented product may be subjected to a « cleaning-up » process (ultra filtration, reverse osmosis, etc) and still be treated as a fermented beverage, intermediate product or ethyl alcohol for excise purposes.

ii. The amount of ethyl alcohol that can be added to a fermented beverage without rendering the final product as a spirit drink under CN code 2208

The Harmonised System Explanatory Notes to heading 2206 permits the addition of distilled alcohol to "other fermented beverages", without specifying however to what extent such addition is possible.

i. Cleaning up and definition of "fermented beverages".

Problematic drinks (from the excise point of view) such as "designer drinks" (often also referred to as "ready to drinks" or "alcopops"), usually are made up of an alcoholic base resulting from a processed fermented mixture (malt, grape or other fruits).

Typical processing includes (with increasing "cleaning effect" of a solution):

- Centrifugation: to remove solid particles
- Cryo-concentration: to modify the alcoholic grade (usually to increase it by removing ice particles that form first)
- Filtration on active charcoal, to remove organic molecules. The carbon, being lipophilic, adsorbs a wide range of organic molecules, including contaminants, odours, and pigments. Ethanol having a lower affinity for activated carbon is therefore less adsorbed.
- Ultra-filtration on semi permeable membranes (reverse osmosis). Reverse osmosis is the process of pushing a solution through a membrane permeable to the solvent or to a particular molecule only. Existing membranes include, e.g., membranes with selectivity for water and ethanol, ethanol only and glycerol and lactic acid. This process is used for instance to produce low alcoholic grade wines or beers. Of course, if on one side of the membrane we have a low alcoholic grade wine, the other side will consist of a mixture of water and ethanol.
- Distillation

It is therefore possible to produce, using these techniques, "clean" or "neutral" ethanolic bases, in the sense of colour, smell or taste, that are not much different from (diluted) distilled alcohol.

ii. Addition of Ethyl Alcohol.

The addition of ethyl alcohol may, in certain cases, be detected by isotopic techniques if the botanical origin of the added alcohol is different from the fermented alcohol and that their isotopic values are sufficiently diverse (see further, p. 16). If they have the same botanical origin then new methods for the identification of suspicious products and the quantification of the added ethyl alcohol are necessary.

II. STRATEGY

It was decided to try to keep in line with the legislation in force, i.e. to find experimental relationships for the classification of samples in the existing excise categories. In the case it would be proven unfeasible, then modification to the legislation could be proposed on the basis of the experimental data and conclusions.

☝ Two complementary pathways of investigation have been examined
1) Multivariate data analysis of markers of fermentation and/or of botanical origin
2) Multivariate data analysis of NMR fingerprints

1) Markers of fermentation and/or of botanical origin

In line with most of the current work in the customs laboratories, minimum values or relative amounts of by-product of fermentation could be used to define “fermented beverages”. Indeed, the cleaning processes decreases the relative concentrations of some of these compounds with respect to that of ethanol or other typical compounds. These markers could be: glycerol, butanediol (with quantification of the meso form), fusel alcohols and organic acids. It could be also possible to look at “typical compounds” such as tartaric acid for grape wine, proteins, maltose and maltotriose for beers or sorbitol for cider. The added-value of multivariate data analysis should be evaluated for their ability to discriminate “fermented” from “non-fermented” beverages.

2) NMR fingerprinting analysis

BEVABS investigated the ^{1}H NMR profiles, which have the advantage, in a “one-shot” experiment to give a view of all major compounds present in the beverage. This is a holistic approach. Without any need for separation, the mixture is submitted to the NMR experiment, which allows the observation and quantification of all major organic compounds, all of them being characterised by one or several signals in the NMR spectrum. This type of analysis is particularly attractive as it is cost effective, requires little or no sample pre-treatment or reagents and typically takes only a few minutes per sample.

III. RESULTS AND DISCUSSIONS

III.1 SAMPLE COLLECTION

Designer drinks (called “alcopops” in the following text), beers and wines were collected by the participating Laboratories and sent to the JRC for NMR analysis. Aliquots were also redistributed to the Customs Laboratories involved in specific “classical” analyses. Samples were chosen in order to maximise the representativity of the various kind of beverages and a balanced number of sample for each category.

The analyses were performed on 114 samples, of which: 25 beers of different kinds, 29 wines from different countries (mostly Italians and, in particular, 9 reds, 4 rosés, 6 whites, 5 sparkling and 5 sweet wines), 15 malt-base alcopops, 9 wine-base alcopops, 17 fruit wine-base alcopops, and 12 distilled or unknown-base alcopops and a few distillates and fermented bases. The list of samples can be found in Annex I.

III.2 NMR AND MULTIVARIATE DATA ANALYSIS

^{1}H-NMR fingerprints of the beverages were acquired on a Bruker 500MHz spectrometer.[1] Typical spectra for beers, wines and alcopops are presented in Fig.1. As anticipated, the beverages’ main components can be identified in the spectra, with the corresponding signals having intensities proportional to their concentration.

These spectral data were further processed and examined with several multivariate data analysis techniques, unsupervised (Principal Component Analysis) and supervised (Projection to Latent Structures and SIMCA).

- PRINCIPAL COMPONENT ANALYSIS (PCA)

In PCA, the initial variables (the NMR spectral intensities) are combined linearly to form a new base set, the “components”. It is an unsupervised method since no a priori knowledge of the class samples under study is necessary. The linear combination is made in such a way that the first component contains the maximum possible variance in the dataset. Similarly, the second component is chosen in order to explain the maximum remaining variance with the only restriction that it be orthogonal to the first component. This can be done for the successive

[1] Technical note: the samples were frozen under vacuum (elimination of most of the ethanol and of the water) and reconstituted in phosphate buffered at pH ~6.3 (to avoid peaks misalignments due to pH differences). Spectra were acquired at 25°C on a 500.13 MHz Bruker instrument equipped with a BBI microprobe. A noisypr1d pulse sequence was used with the following parameters: tr = 5.5s ; aq = 2.5s ; NS = 128. Total acquisition time: 12 minutes. The spectra were normalised to total intensity and bucketed to reduce the 32k data points to 277 variables for the multivariate data analysis.

components until the number of initial variables is reached. However, most of the variance is usually explained by the first 2 or 3 components, forming the statistical model, and the samples can easily be plotted in a 2D or 3D graph, the "score plot". As a result of the mathematical treatment, samples presenting "similar features" should group together.

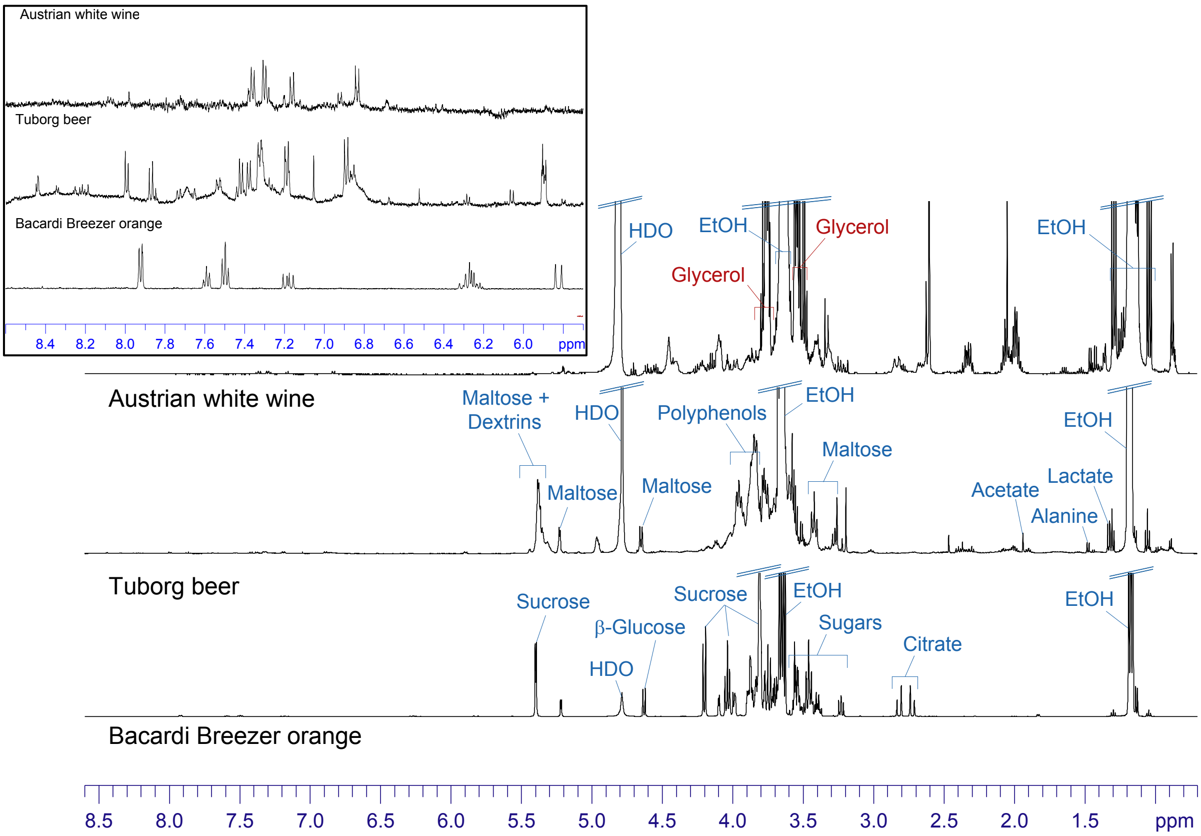

Fig.1: Typical [1]H-NMR spectra of beer; wine and alcopop. A zoom of the region 5.7-8.6ppm is displayed in the frame and shows the presence of aromatic compounds with low concentrations.

A principal component analysis (PCA) performed on the spectral data from beers, wines and "non-fermented alcopops" (made upon a base of rum, gin or other distilled alcohols) is presented in Figure 2. As expected, the beverages from the different categories group together. However some overlapping between wines and alcopops is observed. To understand this, it is useful to remind that PCA is performed on the whole spectral range, i.e. on all major compounds in the beverages. With this in mind, there is no wonder that a sweet wine with high sugar content (respect to all other wines and the concentration of their compounds) is very similar to an alcopop which main ingredients, apart from ethanol and water, are sugars. The result of the PCA is therefore the reflection of the similarities in the beverages global compositions, in other words of the "final product".

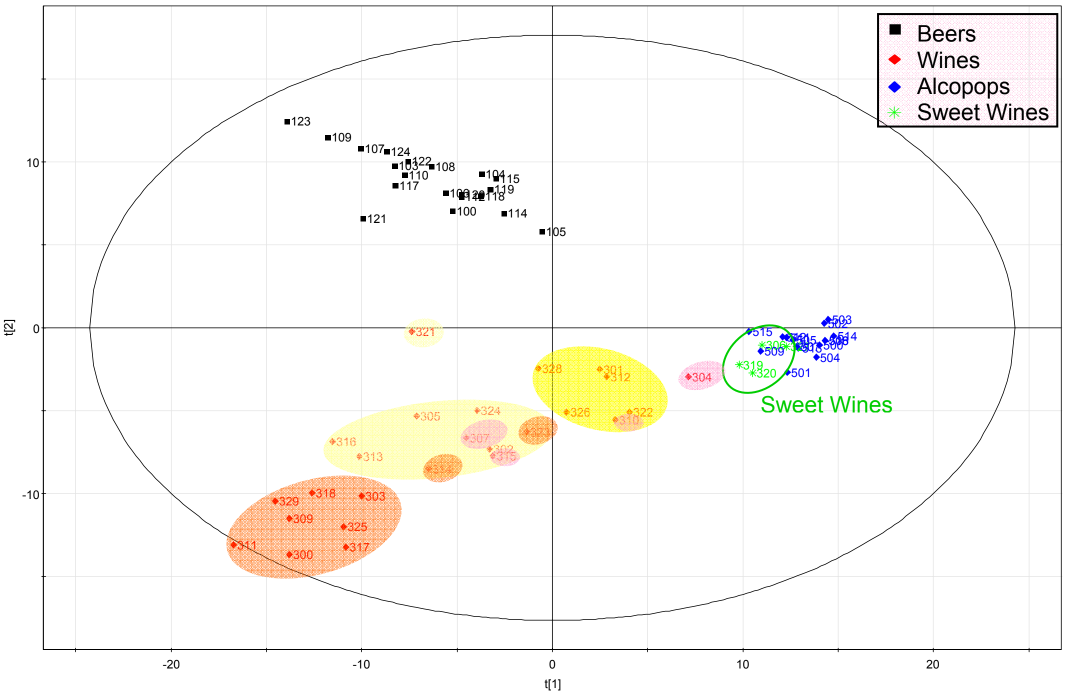

Fig.2: Score plot (2 first principal components) of the PCA applied to the ^{1}H-NMR spectra of beers, wines and alcopops. Wine types are identified by colour: red wines in red, rosé wines in pink, white wines in pale yellow, fizzy wines in bright yellow and sweet wines in green.

The statistical model then can be used to visualise the “position” of new samples. This can be seen in Figure 3 in which some beverages to be tested are plotted using the PCA model obtained from the spectral data of wines, beers and “non-fermented alcopops”. This illustrates the high weight of the sugar content in the model (along PC1). Indeed, the 2 samples falling with the beers (101 –beer Jules de bananes- and 112 –Hoegaarden citron-) are flavoured beers with low added sugar, sample 102 (Adelscott) is a beer with intermediate sugar concentration and samples 111 (Desperados Mas), 116 (Redd’s), 506 (Impress Sobieski) and 510 (Frisco) are typical alcopops (malt-based in this case) characterised by a high sugar level. Sample 308 (Ensô) is a wine-based beverage with moderate sugar content (natural+added).

☞ Multivariate data analysis of the beverages ^{1}H-NMR spectra reflects their similarities in terms of global composition.

☞ In PCA, samples presenting “similar features” group together.

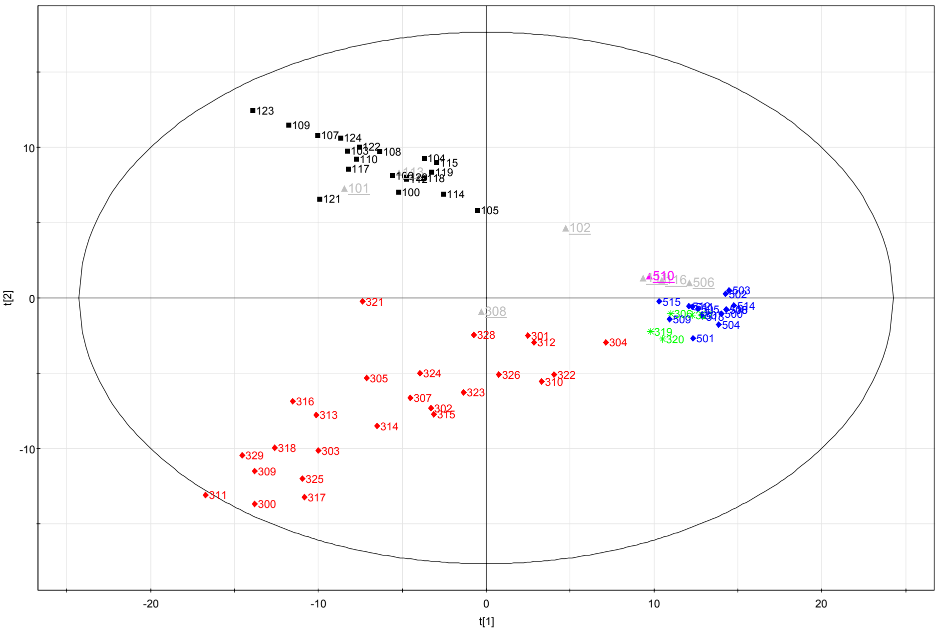

Fig.3: New samples (in purple) plotted using the previous PCA model (Fig. 2).

- PROJECTION TO LATENT STRUCTURE-DISCRIMINANT ANALYSIS (PLS-DA) AND SOFT INDEPENDENT MODELLING OF CLASS ANALOGY (SIMCA)

PLS-DA and SIMCA are supervised methods, in the sense that knowledge of the samples class membership is necessary to build the statistical model, which looks for correlations between the data and these predefined classes. Once the model is built, if it has good prediction abilities, it could be used for class identification of "unknown samples".

PLS-DA, however, requires that each sample can be given a group attribution. A definition of all the classes (the type of beverages) that will be modelled is therefore necessary. Importantly, the groups should be homogeneous. On the basis of the excise legislation in force, these groups should be *beers*, *wines*, *other fermented beverages*, *intermediate products* and *ethyl alcohol*. While beers and wines could possibly be considered as homogeneous groups, this is clearly not the case for the 3 other categories which have large disparities in terms of global composition. Some categories could be further divided to make more homogeneous group such as "apple ciders" or other fruit wines, "spirits", etc. Alcopops wouldn't typically fit in any of these categories and 2 new categories should be defined: "fermented alcopops" and "non-fermented alcopops". This is not satisfactory as the distinction between these 2 groups has not been identified yet and is precisely the scope of the study.

SIMCA has the advantage over PLS-DA that it models the classes (groups) independently by PCA and attributes the membership, or non-membership, of each sample to each model (the models needing to have high sensitivity and specificity[2]). The creation of an "alcopop" group is not necessary. Homogeneity however is still a requirement and would pose problems for the beverages in the "other fermented beverages" and "intermediate products" categories.

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[2] Sensitivity: % of samples belonging to the class which are correctly identified by the mathematical model
Specificity: % of samples foreign to the class which are classified as foreign

☞ SIMCA models a group of beverages and tests the membership of new samples

☞ Necessity to model homogeneous groups: beers only, (red) wines only, etc

An example of SIMCA analysis is given in Figure 4 in which several samples were tested as “unknown” against a wine model (all wines ^{1}H-NMR fingerprints were used to build the model). Four of the tested samples fell into (or very close to) the wine model (corresponding to the green rectangle): *Ensô* (probably a white wine with added water sugar, sugar and glycerol), *Caldirola Fragolino* (a red wine mixed with strawberry juice), *AD Blue Gin* (a wine-based alcopops) and *Havana Loco* (a rum-based alcopops). It is important to keep in mind that the border of the model is arbitrary. In this case, the border is defined by the 95% confidence interval that a sample is part of the wine model. Also, the result of the classification will strongly depend on the samples used to create the model. For instance, if the sweet wines are excluded from the model, the samples *AD Blue Gin* and *Havana Loco* are not classified as wines.

Similar results were obtained with the beer model (Figure 5) but stress an inherent problem with the ^{1}H-NMR – MVDA approach: the 3 samples closest to the beer model are *Adelscott* (102, a beer with added sugar), *Hoegaarden citron* (113, beer with added lemon juice) and *Jules de bananes* (101, beer with added sweeteners and flavourings). According to the legislation in force, these should fall into the beer category. The reason for their non-classification as beers is to be found in the added compounds which move these samples away from the model. This type of beverage could be part of the “other fermented beverages” group but such a group would be impossible to model because of the almost countless potential products that could be added and because of the heterogeneity of the group. This applies of course also to wine-based products.

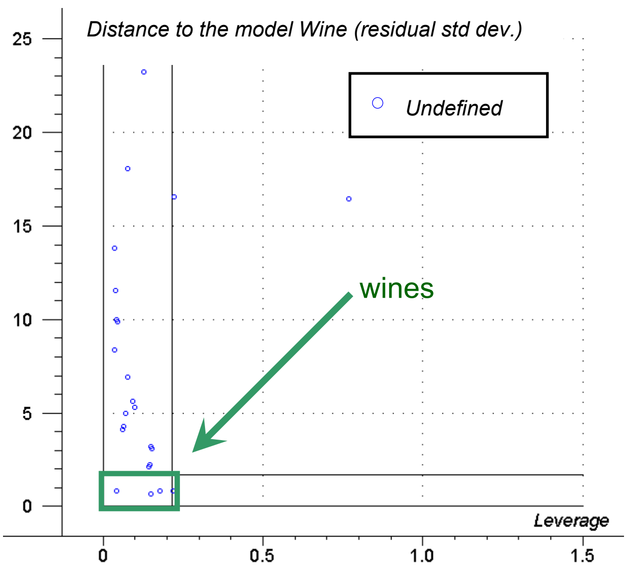

Fig. 4: SIMCA analysis: samples tested for the wine model. Samples recognised as wines should be in the green rectangle (from left to right: 308, 320 507 and 514).

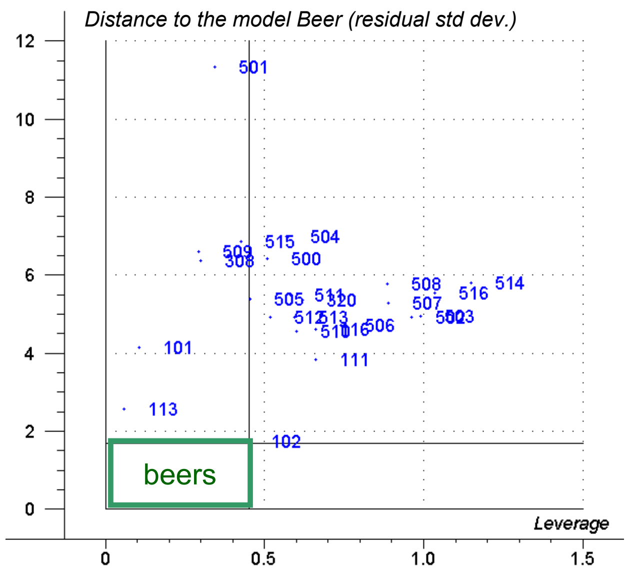

Fig. 5: SIMCA analysis: samples tested for the beer model. Samples considered as beers should be in the green rectangle. The 3 closest samples (101, 102 and 113) are mixtures of beers and non-alcoholic products and are classified as beers under the excise legislation.

- CONCLUSIONS ON THE ^{1}H-NMR – MULTIVARIATE DATA ANALYSIS APPROACH

From our results and from the literature[3] it is clear that such approach has the potential for the characterisation of beers or wines, for instance in terms of quality control or the determination of origin (production site or even geographical origin). However, it turns out that this approach is not adapted to the excise problematic. The difficulty is twofold: the influence of variables that are not related to the "fermented" or "non-fermented" character of the beverage and the necessity to work with homogeneous groups.

Influence of "fermented non-related" variables:

NMR fingerprints contain the information relative to all major compounds in a solution. In other words, it gives a picture of the final product. As the statistical analysis is performed over the whole spectrum, the potential information in the spectra, possibly useful to identify the "fermented" or "non-fermented" character is overwhelmed by compounds that can be added to the alcoholic beverage without affecting the excise category membership. The exhaustivity of NMR is therefore not desirable for the purpose of excise classification and an NMR approach would therefore necessitate extracting from the spectra all signals that are typical from fermentation. This is not possible because of the strong overlapping of the signals in the ^{1}H-NMR spectra. ^{13}C -NMR which presents much more resolved signals would require however much longer experimental time. Because classical analysis would also give similar results, ^{13}C-NMR has not been considered in this study.

Homogeneity of the groups and representativity of the samples

It could be imagined that the membership to one of the excise categories be determined by similarity compliance in terms of global composition, in other words, for instance, "does the product look like a wine or a beer?" In this case, the ^{1}H-NMR fingerprinting approach could be a tool for assessing the beer or wine class membership. This would however require to:

- create several models, corresponding to homogeneous groups within a class (e.g. red, rosé, white and sweet wines) .
- ensure that the models are created on the basis of representative groups of wine, of all types and from all countries (in this study only few, and mainly Italian wines were analysed).

It is also important to note that mixtures of beers or wines with other beverages would not be recognised as beers or wines and, because of the countless number of possible mixtures, these will not be possible to model as "other fermented beverages" or "intermediate products". The same holds for all beverages in the category "other fermented beverages" and "intermediate products" (with the exception of –maybe- ciders) which are too disparate to be modelled.

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[3] Larsen, F. H.; van den Berg, F.; Engelsen, S. B., An exploratory chemometric study of 1-H NMR spectra of table wines. *Journal of Chemometrics* **2006,** 20, (5), 198-208. Lachenmeier, D. W.; Frank, W.; Humpfer, E.; Schafer, H.; Keller, S.; Mortter, M.; Spraul, M., Quality control of beer using high-resolution nuclear magnetic resonance spectroscopy and multivariate analysis. *European Food Research and Technology* **2005,** 220, (2), 215-221. Duarte, I.; Barros, A.; Belton, P. S.; Righelato, R.; Spraul, M.; Humpfer, E.; Gil, A. M., High-resolution nuclear magnetic resonance spectroscopy and multivariate analysis for the characterization of beer. *Journal of Agricultural and Food Chemistry* **2002,** 50, (9), 2475-2481.

III.3 CLASSICAL ANALYSIS

It is well known that in fermented beverages there is a relationship between the concentration of ethanol and fermentation by-products such as glycerol, fusel alcohols and certain organics acids. This relationship differs however between beverages as a result of different yeast species and fermentation conditions. In some countries, such relationship is used to assess the fermented character of beverage. However, because of the spread of relationship coefficients, a single criterion such as the ratio ethanol/glycerol is insufficient for a clear-cut identification of a "fermented beverage". This is why more criteria are necessary. For instance, in the Netherlands, 2 criteria are used, based on the analysis of ~2000 grape and fruit wines: a minimum of 28g of glycerol per litre of pure alcohol and 82g of fusel alcohols[4] per 100l of pure alcohol. These are rather loose as real fermented beverages should not be excluded by the rule (and never are).

The purpose of this study was to find out whether more descriptors could be used to refine the "fermented" assessment. It was also hoped that more criteria would help preventing producers to bypass the rule by simple addition of one compound to the beverage to fulfil the condition.

The ACAP participants agreed in the mid-term meeting in Athens on a list of compounds, <u>related to fermentation or to the botanical origin</u>, to analyse:

ethanol, glycerol, methanol, propanol, 2-methyl-1-propanol, 1-butanol, 2-butanol, 2-methyl-1-butanol, 3-methyl-1-butanol, 2-3 butanediol, acetaldehyde, ethyl acetate, tartaric acid, malic acid, lactic acid, acetic acid, succinic acid and potassium.

116 samples were distributed amongst the customs laboratories for analysis, taking into account the laboratories technical capacity, their time and human resource availability and the necessity of a minimum cross-check of the analytical results.

Data crosschecking revealed some large discrepancies among the analytical results produced in the different laboratories. It was not the purpose of this exercise to conduct an inter-laboratory proficiency testing but to assess the feasibility of this approach. Different methods might therefore have been used by different laboratories. This could partly explain for the discrepancies that were sometimes observed between laboratories. Another source for the discrepancies could be that many methods were developed for wines and are not suitable for other product types and to lower contents of the targeted compounds (as it is often the case for beers, ciders and alcopops). For instance for organic acid analysis of beer and wine distillates for which one laboratory detected concentrations of these acids between 10 and 50 mg/L, while another did not detect anything (as it would be expected!). As a consequence, for this approach, it will therefore be absolutely necessary to develop and validate new methods adapted to larger range of concentrations and product types.

Analytical protocols not validated for all range of concentrations and compounds

From this list, several compounds were discarded before the multivariate data analysis for their very low contents (2-butanol), large discrepancies (methanol) or incomplete data (2-3 butanediol and acetaldehyde). The results of the analyses can be found in the powerpoint presentation from 3rd meeting and in Annex I.

[4] Fusel alcohols: sum of 1-propanol, 2-methyl-1-propanol, 2-methyl-1-butanol and 3-methyl-1-butanol.

- Multivariate data analysis: SIMCA

A single model for all fermented beverages?

It was first attempted to create a single model for fermented beverages using the beer and wine samples (no other fermented beverage samples were provided). All variables were normalised to the ethanol content to eliminate alcohol or dilution dependence. Tartaric acid was removed as, by itself, it discriminates beers from wines. Because malic acid is a common food additive and because it can be naturally found in some fruit juices such as apple juice, it was also removed from the model. A PCA performed on this dataset clearly shows clustering of beers and to a lesser extent of wines: the dataset is not homogeneous indicating that, with this approach, the beverages categories should be modelled independently.

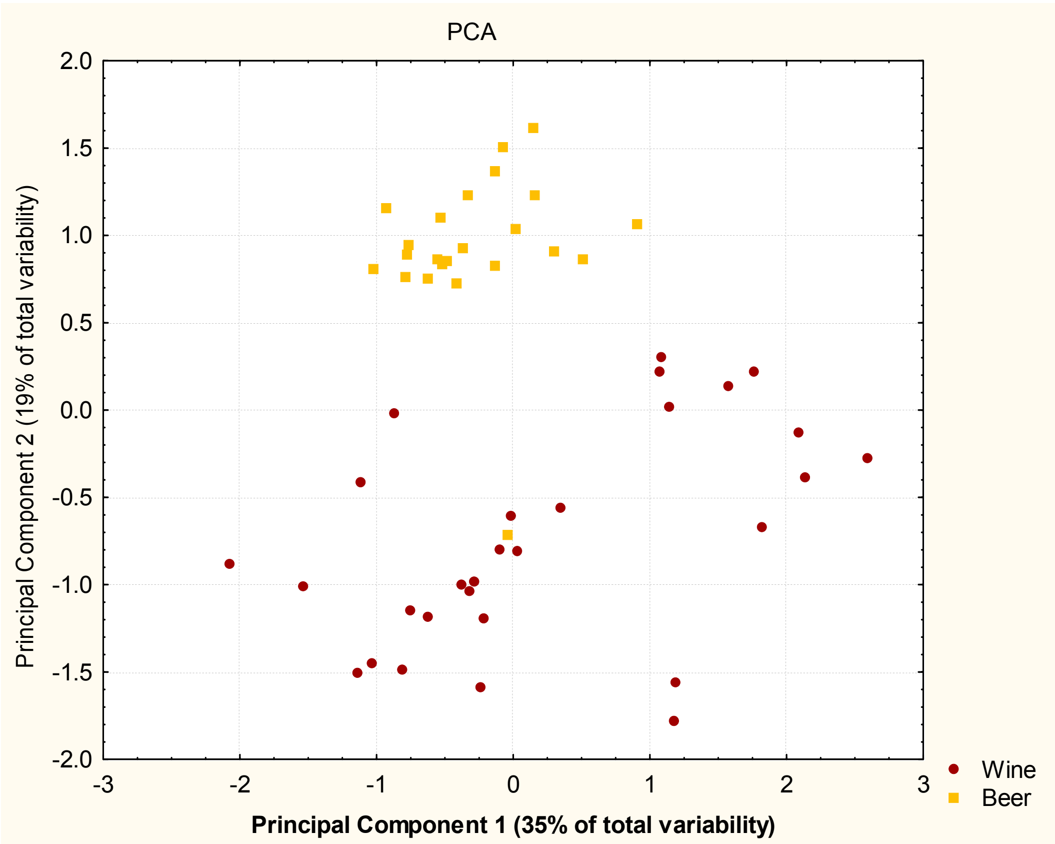

Fig 6: PCA on wines and beers. Tartaric acid was not included in the model as it discriminates wines from beers.

Indeed, because of the high variance induced by the 2 clusters in the model, if a SIMCA analysis is performed for all other samples, i.e all alcopops-type beverages and aromatised beers or wines, most wine-based and malt-based alcopops as well as the wine and beer distillates are recognised by the model as "fermented". Interestingly, the aromatised beers "chapeau-fraise" (with high lactic acid content) and "Jules de bananes" are not recognised. The same applies to *Ensô*, probably because it contains the addition of glycerol. Only 5 out of 17 fruit-wine based beverages are recognised by the model and only one distilled-alcohol based alcopops (Havana Club Loco Limon). This seems to indicate that although very loose, this model does not contain the necessary information to recognise fermented fruit beverages.

☞ No unique "fermentation model" – Necessary to analyse beverage groups *independently*

A model by beverage category?

It is thus clearly necessary to model the beverages by category. A PCA on wines (Figure 6) shows that even within a category it might be necessary to refine the analysis: red wines on one hand and white, rosé and fizzy on the other hand cluster together while sweet wines show another behaviour still (with the 2 sweet French wines being very different from the 3 Italians).

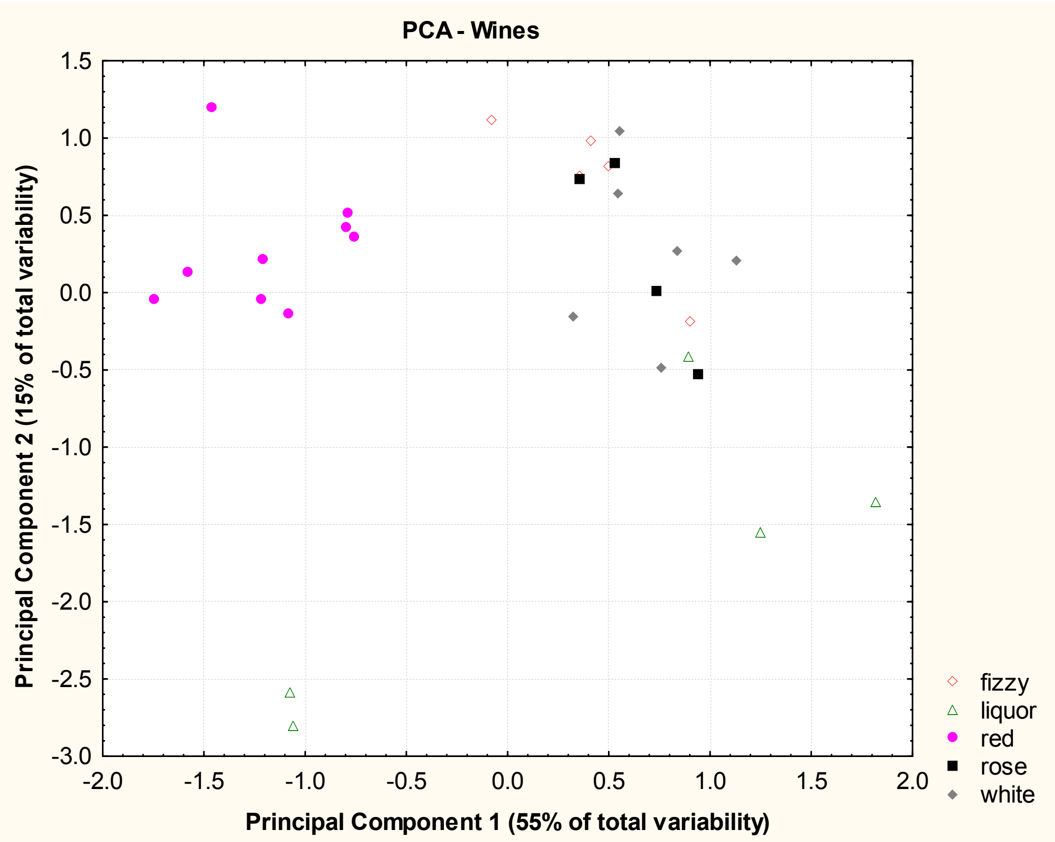

Fig 6: PCA on wines. Malic and tartaric acid are excluded from the model.

A SIMCA analysis of the wine-base beverages using this wine model rejects, with a 95% confidence level, half of the wine-base samples, of which only 1 of the 2 distillates. It is also important to note that *Enso* is rejected because of its addition of glycerol and so are *Caldirola Fragolino* (red wine with strawberry juice) and *Dimitroff Verde* (same producer than other recognised beverages), probably because of the composition of the non-alcoholic part of the mixture (fruit juices). Decreasing the confidence level to 90% will result in the rejection of the 2 distillates. It is essential that remember that the corollary of a 90% confidence level is that 10% of the wines will not be recognised either.

Similar results are obtained from a SIMCA analysis of the malt-base beverages using the beer model. The 2 distillates are rejected but so are also *Hoegaerden Citron*, *Chapeau-Fraise* and *Jules de bananes* again very probably because of the addition of fruit juices. One of the 2 *Desperados* samples and one of the 2 *Boomerang* samples are also rejected, seemingly because of their higher content of acetic and succinic acid.

No model can be created for the other fruit wines-based beverages as only alcopops-type samples were collected and no "authentic" fermented fruit wines.

> 👎 No samples available for the "other fermented" beverages (apple ciders, pear ciders, etc need to be analysed independently)
>
> 👎 Data quality and representativity of the sampling not satisfactory for modelling

Variables non-specificity issue

In the preceding paragraphs, we have seen that some beverages were not recognised by the model as being fermented (wine or beer). Interestingly, these often have high concentration of some organic acids, probably coming either from an added fruit juice (or fruit juice extract) or because of their use as food additive. This raises the question of the specificity of the variables used to build the model. With this approach, a beverage will be recognised only if it has the "right" combination of all considered variables. If in a beverage, one of the variables is present in a higher amount because it was contained in the non-alcoholic part of the beverage mixture, or because it was added for its preservative or organoleptic properties, then the beverage will probably fall outside the model.

This is especially relevant for all organic acids (succinic acid to a lesser extend), which are known to be naturally present in fruit juices[5] and used as additive in the food industry (E260: acetic acid, E270: lactic acid, E296: malic acid, etc). Glycerol was also added, probably for its organoleptic properties, in one of the wine-based beverages (*Ensô*). If the result of the classification rule is that, *de facto*, the addition of a certain number of common food additives or fruit juices results in the classification into the distilled category, then it would be probably wiser to target certain compounds more closely related to a type of beverages for which there is a political will not to confer reduced excise duties.

It could be considered that it is therefore the mere absence or presence (or minimum tolerated amounts) of *all* the variables that will decide the classification of the beverage. This would easily exclude beverages, for which several compounds could not be detected and that are clearly made using distilled alcohol. In the sample collection, this would be the case for all *Zip, Campari* and *Smirnoff* products.

However, this would not resolve the issue of the extent to which a fermented product can be processed for cleaning. The best examples are the *Bacardi Breezer* products. Two very similar type of *Bacardi Breezer* products, with almost identical packaging, were collected: *Bacardi Breezer "Alcoholic mixed drink"* (Belgium) and *Bacardi Breezer Rhum refresher* (Italy), both in the Tropical Orange and Tropical Lime versions. Such rule would classify differently 2 almost identical products: the alcoholic mixed drink version as fermented and the rum refresher version as distilled.

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[5] Cunha, S. C.; Fernandes, J. O.; Ferreira, I., HPLC/UV determination of organic acids in fruit juices and nectars. *Eur. Food Res. Technol.* **2002,** 214, (1), 67-71.

Finally, it has to be noted that a distillate, containing no organic acids, mixed with a fruit juice might be recognised as “fermented”. The absence or presence *per se* is therefore insufficient to be used as a rule for the classification.

> 👎 The compounds selected to create the models should not be present in non-alcoholic beverages or be added during the manufacturing process → would exclude at least the organic acids for the statistical analysis.
>
> 👎 Minimum values for alcohols and organic acids insufficient to detect a “fermented character”.

The cleaning process: a highly tuneable process

“Problematic beverages” from the point of view of the excise classification are often produced by addition of non-alcoholic mixtures to a “neutral” alcoholic base. There are 2 ways to obtain clean “neutral” alcoholic bases: distillation or “cleaning processes”. A fundamental difference between these 2 processes is that distillation is essentially a “on/off” process: organic acids and glycerol will not be found in the distillate and the recovery percentage of the alcohols will depend on the distillation conditions (they are found in the head and tail of the distillation). On the contrary, the composition of the alcoholic base obtained by “cleaning” will depend on the type and number of steps in the cleaning process and, potentially, any desired composition could be obtained. This is illustrated by Figure 7 representing a PCA performed on all alcopops-type beverages, from fermented and distilled origin, and on clean alcoholic bases (distilled and fermented). 3 samples of wines and 3 samples of beers were plotted using this PCA model. While the distillates and the distilled-based alcopops clearly form a line on the right side of the score plot, all other samples are rather scattered along PC1, indicating a situation of “continuum” between the distillates and the beers and wines (on the left side of the plot). No clear distinction can be observed between the products.

> 👎 Cleaned fermented beverages occupy the whole space between distillates and “genuine” fermented beverages: no clear distinction.
>
> 👎 No objective limit, and therefore no objective analytical rule, between “fermented” and “non-fermented” products can be drawn.

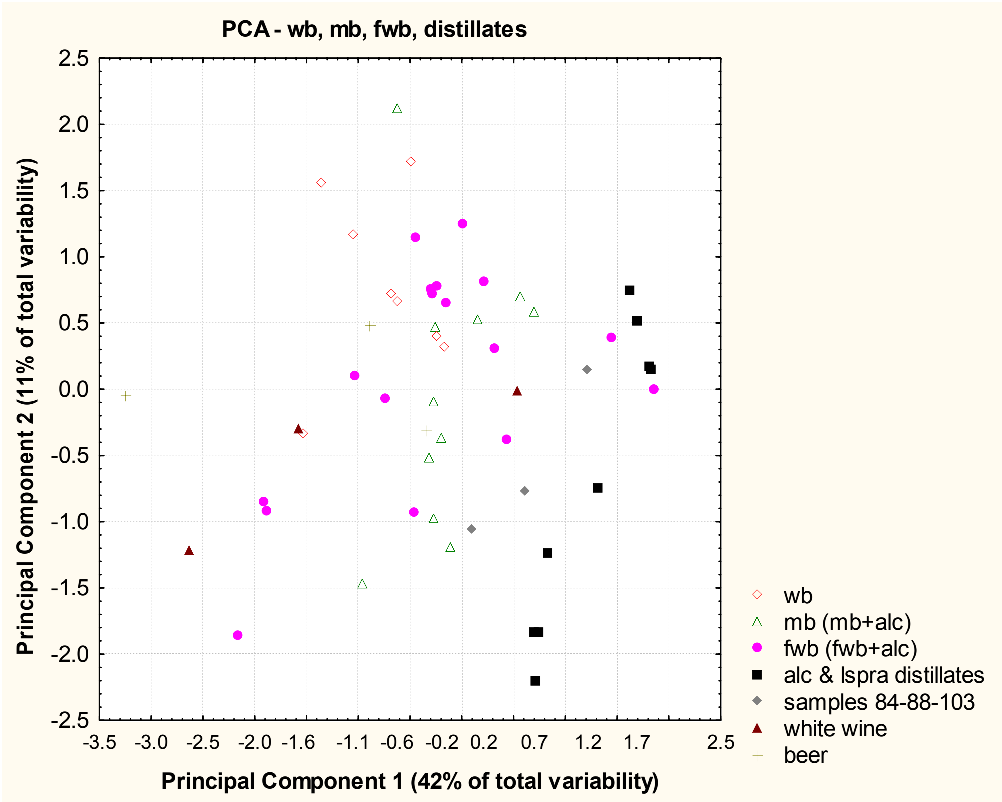

Fig. 7: PCA performed on: alcopops-type beverages claimed to be "fermented" (wb: wine-based, mb: malt-based, fwb: fruit wine based, +alc: with addition of ethanol), made upon a distilled alcohol (alc), 2 beers and 2 wines distillates (Ispra distillates) and on samples 84, 88 and 103, i.e. "clean" fermented bases used for alcopops production from, respectively, malt (DE, NL) and wine (NL). 3 white wine samples and 3 beer samples were also plotted using the PCA model.

Detection of the addition of distilled alcohol to a fermented beverage

One of the questions of this exercise was to check whether it is possible to detect the addition of distilled alcohol to a fermented beverage. To this aim, 2 cases must be considered: whether the added alcohol comes from another botanical source or whether it comes from the same one. In both cases, the detection is based on the measure of certain variables and on their deviations from statistical averages. These are detection methods to <u>identify suspicious products and by no means a quantitative measure of the ethanol addition.</u>

a) Addition of alcohol from a different botanical origin

The detection of added ethanol from a different botanical origin is based on the principle of natural isotopic fractionation of the ethanol source. Factors such as plant physiology and geo-physical parameters affect the isotopic fractionation thereby allowing the characterisation of the ethanol source.[6] One of these methods is the measurement, by NMR, of the site-specific deuterium/proton ratios on the ethanol sites (CH_3: site I, CH_2: site II).

[6] Calderone, G.; Holland, M.; Reniero, F.; Guillou, C., An overview of isotopic analysis for the control of alcoholic drinks and spirits. EU report: EUR 21875, **2005**.

A sample is considered problematic if its ethanol $(D/H)_I$ value falls out of the natural range. In the case of wine for instance, values under 99 ppm or over 105 ppm are suspicious. If average values for $(D/H)_I$ are considered, then an evaluation of the detection limit for ethanol addition can be made for the different possible ethanol sources (Table 1). This is only indicative since with this approach the addition of beet ethanol to a wine with high $(D/H)_I$ value (105 ppm) would be detected only if its proportion is superior to 50%! If the geographic origin of the wine is known, then the natural $(D/H)_I$ range can be reduced and the detection limit refined: it is case to case work necessitating supplementary information on the wine (grape growing region for instance).

With this method, addition to wine of ethanol from distilled wine or fruit wines (apples, etc) cannot be detected (all fruit alcohols have similar D/H ranges). If the ethanol added comes from grain, it will be detected only if its proportion is higher than 70%.

Importantly, this approach allows for the determination of the botanical origin of the ethanol, not the "fermented character" of the beverage. Indeed some vermouths are produced using a second fermentation of sugars: they are "fully fermented" but have D/H values out of the limits. Complementary analyses of fermentation by-products are therefore necessary.

Ethanol source	**$(D/H)_I$ /ppm Natural range**	**Minimum proportion of added ethanol in wine (102 ppm) for addition detection**	**$(D/H)_I$ /ppm in wine + ethanol addition**
Grape	99-105	-	-
Beet sugar	91-93	35%	98.5
Cane sugar	108-110	50%[7]	105.5
Grain	96-99	70%	98.85
Potato	93-95	40%	98.8
Fruits	97-104	100%	100.5 (n.d. !)
Synthetic alcohol	123-124	15%	105.2

Table 1: Natural $(D/H)_I$ range for various ethanol source and calculation of the detection limit for the addition to wine of ethanol for the different ethanol sources (considering average values). n.d.= not detectable.

a) Addition of alcohol from the same botanical origin (or having similar natural isotopic values)

In this case the approach is similar to those presented earlier: models for the beverages groups are created and a sample is tested against it. Here, a SIMCA analysis was carried out, testing a wine and a beer with different additions of distilled alcohol. The addition of distillate was done mathematically, using the results obtained from the wine and beer distillates,[8] resulting in a virtual increase of the ethanol content by 25, 50, 75 and 100%. The results (Figure 8) indicate that it is probably possible to detect the addition of alcohol if it exceeds 100% (i.e. a final mixture composed of 50% "fermented alcohol" and 50% "distilled alcohol")[9]. This detection limit is quite high in regard to the usual alcohol

[7] If the ratio $^{13}C/^{12}C$ is known (by isotopic ratio mass spectroscopy methods), the detection limit is lower: 20%.

[8] As has been mentioned previously, the alcohols composition of a distillate depends on the distillation conditions, i.e. whether the head and tails of the distillation were kept or not. The addition of distilled alcohol to a wine or a beer could result in an increase of ethanol only or to an increase of all alcohols in the final mixture. Adding a distillate containing all alcohols is a conservative approach (adding pure ethanol will enhance the effect seen in this case).

[9] If the distillate is composed of mainly ethanol, its addition could be detected at lower levels (>30% in the final mixture).

content of fermented beverages and to the alcohol content ceiling for excise classification (15-18% for wines and other fermented beverages, 22% for intermediate products).

Again, in order to use this approach for the detection of distilled alcohol addition, a model would be required for all fermented beverage categories.

Finally, it is important to understand that, again, such an approach is <u>not</u> a measure of the alcohol addition and therefore cannot be used to assess whether the distilled alcohol content of a beverage is 30, 45 or 60% but merely could be used as an alert. Consequently, to guarantee an accurate assessment of how much ethyl alcohol is added to a product, it would appear that some assurance or knowledge of the actual process of production (i.e. the recipe) may be necessary.

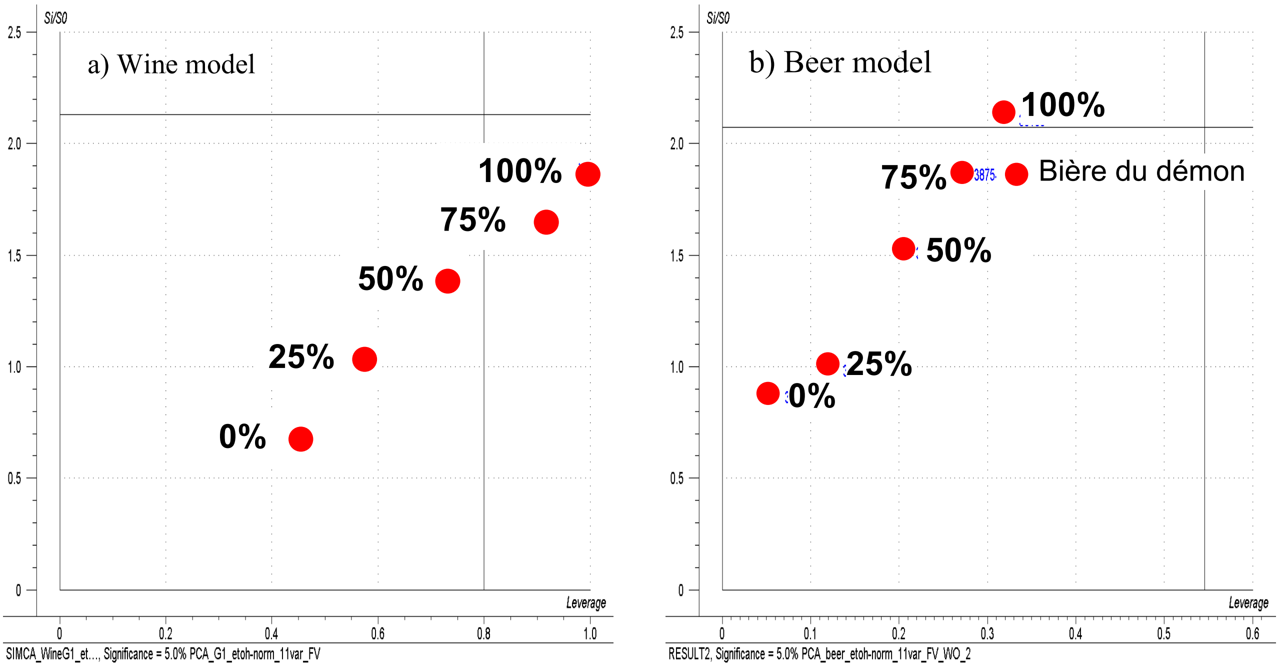

Fig. 8a and 8b: SIMCA analysis of a wine (Fig. 8a) and a beer (Fig. 8b) with addition of distilled alcohol. The starting sample is noted by 0% (no addition of distillate). Distillate was mathematically added in order to increase the alcohol content by 25, 50, 75 and 100%. In Fig. 8b, the sample *Bière du démon* (high alcohol content, possibly because of ethanol addition) was also plotted: it is recognised by the model as a beer but its position is close to the limits.

☝ Not a measure of the ethanol addition! ☟ The addition of distilled ethanol to fermented beverages will be detected in some cases, in others not.

CONCLUSIONS ON THE MULTIVARIATE DATA ANALYSIS ON CLASSICAL MEASUREMENTS

The analytical difficulties, error margins and discrepancies between laboratories, make it difficult to draw clear-cut conclusion and impossible to establish rules for the classification of alcoholic beverages. From this study, it cannot be excluded that models for "fermented beverages" could be created. However, considering the following points, it would probably not be wise to keep following this track.

Indeed, establishing statistical analytical rules for the classification of beverages into "fermented" or "not-fermented" categories would, with no guarantee of success, require:

- To build independent models for all types of beverages. This poses the problem of the representativity of the samples that will be used to create the models. Many countries produce many types of beers and wines. The sample collection should be devised making sure that these are all represented in order to avoid that some beers and wines be excluded by the model just because they had not been sampled. It will also be very difficult to list and sample all the types of beverages included in the "other fermented beverages" category. It should be kept in mind as well that with independent models, a mixture of beer and wine for instance will not be recognised by any fermented model.
- To build the models and decide on a rule for the classification of the beverages. The rule will depend on the chosen statistical approach and there will not be any objective criteria to decide about the limit between "fermented" and "not-fermented". Also, the cleaning process of beverages that would be considered as "non-fermented" by the models will probably be adjusted in a short time in order to respect the fermented criteria. This clearly questions the sense of such an analytical rule. Finally, it would be wise to consider the easiness of fraud by simple external addition of the compounds under scrutiny.

> 👎 No generic "fermented" model
>
> 👎 Necessity to be representative within a class of beverages and to have an exhaustive list of them → very difficult (impossible?).
>
> 👎 If models can be created and rules for the classification established, the cleaning processes will probably just need minor tuning to produce "fermented beverages". So what would be the sense of such a rule?

III. CONCLUSIONS

It appears that legislation has in some respects struggled to keep pace with developments in the alcoholic beverages sector. While, previously, distillation was the only way to produce “clean” or “neutral” ethanol solutions, in the sense of colour, smell or taste, this is not so anymore. Several new techniques have appeared allowing the process of a fermented broth into “neutral” ethanolic solution further used as base for new types of alcoholic products. These techniques are “adaptable” in terms of selectivity in the cleaning process, i.e. that specific molecules in the fermented broth can be chosen to be present or not in the desired final product. The current legislation was not designed with these modern techniques and the new kinds of alcoholic beverages in mind. This underscores the problems in an approach that simply attempts to distinguish between “distilled” and “fermented” as a base for the classification of beverages.

The large number of alcoholic beverage types impeded us from conceiving analytical rules for their classification and it is unclear that chemical analyses will ever give a solution to this issue. In any case, it will probably prove to be excessively unpractical.

In terms of the "designer drinks", which are the greatest cause of problems, if it is desired to classify all of these products similarly (in order to ensure that drinks perceived by the consumer to be of similar character are dutied on a similar basis), then it may prove necessary to look for alternatives to the type of approach that it was hoped this report could develop. Just for example, one approach noted by the JRC might be to target the addition of ingredients that will almost inevitably be added to cleaned-up alcohol based beverages such as sweeteners, artificial aromas, colourings, etc. Such a rule appears to have the advantage of being clear and not subject to interpretation, giving legal certainty, and easily controllable by laboratories. A further possibility that was noted might be that additional comment could also be made concerning the nature of a fermented alcohol, stipulating that these may not be processed with the purpose of producing a neutral alcohol base, with the intention of clarifying the legislation rather than providing a tool for control.

So to briefly recap, it is the findings of this report that it is likely to be extremely difficult or even impossible to create workable models based on analytical methods. Unfortunately, the difficulties are the greatest precisely with those categories that are the most problematic for excise classification purposes. Even if an analytical model was eventually developed it is likely to need to be an extremely complex mechanism in order to take account of the broad range of beverages, and diverse chemical characteristics, of products that fall within the problem categories ("other fermented beverages" etc.)

Because of the complexity and the need to try to take into account such a broad range of products, there are most likely to be strange results in particular cases (i.e. products that a person would currently associate with the spirituous category satisfying the objective criteria laid down for a fermented beverage and vice-versa). Further, it is almost certain that operators would be able to adjust their products, with no tangible change in the character of the beverage, simply to ensure that the relevant chemical indicators that ensure fermented status are present. Consequently concrete rules based on an analytical method could lead ironically to perverse results.

On the issue of 'added alcohol' the findings reinforce that knowledge of the recipe/production process is needed in order to quantify the exact amount of addition. It is not plausible to ascertain the amounts by a scientific study of the finished product.

ANNEX I

beverage type	N° Eurodat	Alc. Cont. (% vol) Mean	Glycerol (g/L) Mean	methanol (mg/L) Mean	1-propanol (mg/L) Mean	2-methyl-1-propanol (mg/L) Mean	1-butanol (mg/L) Mean	2-methyl-1-butanol (mg/L) Mean	3-methyl-1-butanol (mg/L) Mean	ethyl acetate (mg/L) Mean	Tartric ac. (mg/l) Mean	Malic ac. (mg/L) Mean	Lactic ac. (mg/L) Mean	Acetic ac. (mg/L) Mean	Succinic Ac.(mg/L) Mean	Potassium (mg/L) Mean
fizzy	07050023	10.91	5.35	36.00	34.00	52.50	0.90	29.50	151.00	31.50	1283.00	1102.00	1549.00	203.00	427.00	564.34
fizzy	07050024	11.51	4.67	39.00	33.50	18.50	0.60	18.00	103.50	40.00	2318.00	2579.00	463.00	235.00	312.00	704.36
fizzy	07050025	10.45	4.77	32.00	33.50	26.00	0.60	20.00	130.00	30.50	1396.00	1322.00	1506.00	218.00	334.00	515.05
fizzy	07050026	10.70	5.35	35.00	35.00	39.00	0.60	20.50	113.00	41.00	1379.00	625.00	1979.00	175.00	321.00	513.52
fizzy	07050027	10.59	5.43	36.00	37.00	39.00	0.50	21.50	116.50	34.00	1657.00	991.00	2083.00	131.00	309.00	505.71
Liquor	07050028	16.10	5.36	86.00	18.00	27.00	1.40	26.50	104.00	72.50	1162.00	261.00	1646.00	357.00	347.00	748.81
Liquor	07050030	15.36	4.64	102.00	12.00	17.50	0.30	10.00	51.50	42.00	1040.00	1795.00	185.00	159.00	277.00	724.77
Liquor	07050031	15.12	6.15	137.00	13.00	25.50	0.30	18.50	79.50	36.50	975.00	2241.00	185.00	332.00	418.00	1031.92
Liquor	07080002	13.28	15.60	41.00	22.50	66.50	0.70	29.50	125.50	93.50	1268.00	1687.00	263.00	798.00	722.00	1255.79
Liquor	07080003	12.81	14.10	60.00	25.00	64.50	0.70	29.00	128.00	101.50	1383.00	1713.00	461.00	696.00	627.00	1339.00
Red	07050004	13.31	9.61	196.00	69.00	46.00	1.40	41.50	187.50	120.50	1966.00	46.00	2473.00	878.00	718.00	1426.95
Red	07050006	13.41	9.98	185.00	36.50	60.00	0.90	64.00	268.00	60.00	1767.00	74.00	2967.00	427.00	972.00	1095.94
Red	07050007	11.86	7.20	112.00	19.50	42.50	1.00	44.50	177.00	55.50	2069.00	97.00	2431.00	342.00	548.00	1077.30
Red	07050008	12.81	8.06	208.32	35.65	52.08	0.70	55.01	205.76	141.17	1258.00	4.00	3726.00	857.00	726.00	1080.68
Red	07050009	12.38	8.43	160.50	20.77	55.56	0.65	51.33	201.65	72.86	2354.00	70.00	2203.00	466.00	658.00	1152.94
Red	07050010	11.72	7.95	101.24	38.22	36.09	0.90	41.88	207.28	46.82	2250.00	90.00	1252.00	377.00	629.00	1158.01
Red	07050011	12.62	9.18	192.05	24.15	53.67	0.70	61.69	241.05	79.73	1784.00	140.00	1981.00	443.00	646.00	1119.09
Red	07050012	11.88	9.22	158.04	21.23	68.16	0.50	44.12	176.07	63.79	2892.00	401.00	2661.00	471.00	801.00	832.16

beverage type	N° Eurodat	Alc. Cont. (% vol) Mean	Glycerol (g/L) Mean	methanol (mg/L) Mean	1-propanol (mg/L) Mean	2-methyl-1-propanol (mg/L) Mean	1-butanol (mg/L) Mean	2-methyl-1-butanol (mg/L) Mean	3-methyl-1-butanol (mg/L) Mean	ethyl acetate (mg/L) Mean	Tartric ac. (mg/l) Mean	Malic ac. (mg/L) Mean	Lactic ac. (mg/L) Mean	Acetic ac. (mg/L) Mean	Succinic Ac.(mg/L) Mean	Potassium (mg/L) Mean
Red	07090145	12.21	8.61	141.00	20.00	48.50	1.40	50.50	187.50	54.00	2137.00	135.00	1523.00	329.00	588.00	860.93
Rose	07050013	9.50	4.62	62.08	31.41	22.06	0.25	15.41	105.26	19.60	2169.00	1424.00	519.00	157.00	330.00	692.64
Rose	07050014	12.21	6.64	52.56	40.03	28.36	0.35	25.71	176.90	41.84	808.00	2292.00	1646.00	145.00	509.00	743.22
Rose	07050015	10.31	5.26	37.79	33.61	32.87	0.35	21.24	140.44	26.53	2174.00	1256.00	1304.00	201.00	345.00	375.34
Rose	07050022	11.92	5.55	27.85	24.48	27.23	0.40	18.23	109.39	38.33	2444.00	2098.00	327.00	224.00	384.00	561.44
White	07050018	11.31	5.94	32.94	48.86	25.99	0.55	25.64	158.56	27.58	1761.00	965.00	572.00	196.00	438.00	460.64
White	07050019	12.35	6.19	36.70	14.63	35.01	0.35	29.97	167.85	47.03	1420.00	862.00	1547.00	393.00	471.00	509.33
White	07050020	11.97	4.64	35.40	32.51	16.57	0.55	21.63	134.84	27.88	1931.00	906.00	254.00	104.00	272.00	685.09
White	07050021	12.29	5.49	50.00	36.50	27.50	0.80	22.00	144.00	43.00	1673.00	978.00	1073.00	201.00	544.00	479.92
White	07090148	11.92	5.87	45.64	29.65	22.28	0.45	18.82	119.56	44.04	1941.00	2077.00	449.00	315.00	387.00	650.88
White	07090149	12.93	6.25	57.37	38.20	21.96	0.40	21.98	139.18	42.56	1697.00	1190.00	1180.00	252.00	460.00	470.74
Beer	07020109	6.59	1.49	1.72	23.21	12.65	0.00	12.96	52.63	29.18	16.00	363.00	494.00	346.00	395.00	591.13
Beer	07030017	4.41	1.28	1.68	7.15	9.71	0.00	12.63	34.06	7.61	36.00	124.00	207.00	165.00	122.00	568.85
Beer	07030018	3.96	0.87	1.06	6.23	8.96	0.00	11.52	30.58	9.39	10.00	93.00	237.00	81.00	49.00	355.90
Beer	07030019	4.99	1.40	2.97	10.64	12.16	0.26	15.60	38.42	11.47	26.00	181.00	112.00	155.00	66.00	490.40
Beer	07030020	5.13	1.51	1.93	12.61	14.26	0.40	17.34	48.22	16.00	19.00	87.00	415.00	92.00	43.00	435.90
Beer	07040056	3.97	1.17	1.65	9.32	14.33	0.79	20.51	47.20	6.42	5.00	49.00	205.00	91.00	109.00	256.33
Beer	07040057	4.38	1.09	1.25	9.40	13.86	0.28	14.03	42.11	12.45	9.00	73.00	327.00	71.00	88.00	320.04

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Beer	07040058	3.49	1.10	1.82	8.41	8.91	0.26	11.12	31.85	9.64	11.00	14.00	785.00	155.00	45.00	439.71
Beer	07040059	3.82	1.15	1.52	10.71	8.92	0.38	11.36	34.03	9.73	4.00	91.00	213.00	64.00	95.00	373.59
Beer	07050032	5.35	1.10	2.03	6.29	13.08	0.12	15.73	48.38	13.73	24.00	119.00	219.00	107.00	51.00	582.57
Beer	07050033	5.13	1.85	2.13	12.74	14.27	1.11	21.25	67.76	13.62	11.00	133.00	252.00	107.00	44.00	306.71
Beer	07050034	4.11	1.04	1.29	19.13	20.69	0.29	13.13	51.08	13.01	22.00	165.00	243.00	35.00	78.00	446.78
Beer	07050035	4.95	1.51	1.95	18.26	24.69	0.17	16.78	56.83	40.11	28.00	98.00	309.00	106.00	95.00	534.93
Beer	07050036	4.94	1.14	1.24	7.56	12.24	0.00	15.93	50.28	14.59	36.00	158.00	173.00	69.00	110.00	476.34
Beer	07050037	4.87	1.39	1.34	12.23	18.50	0.78	19.59	50.71	10.86	29.00	143.00	158.00	101.00	125.00	523.35
Beer	07050038	11.36	1.27	4.84	21.61	23.55	0.80	31.23	113.07	38.07	11.00	154.50	104.50	131.50	103.00	495.81
Beer	07050040	5.20	1.38	6.01	10.97	12.24	0.00	15.28	52.18	17.55	41.50	123.00	514.00	109.50	136.00	614.41
Beer	07050041	4.74	1.00	4.44	8.92	11.39	0.00	14.41	39.57	25.12	30.50	158.50	100.50	67.50	76.50	564.76
Beer	07050042	4.95	1.61	2.78	10.68	15.12	0.68	21.45	56.07	13.93	16.50	109.50	125.00	37.00	116.50	355.67
Beer	07050043	5.04	1.30	1.81	17.67	23.55	0.16	16.18	52.85	42.70	11.00	97.00	372.00	92.00	73.00	529.63
Beer	07050044	4.30	1.02	1.50	12.51	14.00	0.00	19.63	51.18	10.56	27.00	80.00	113.00	61.00	80.00	222.87
Beer	07050045	4.94	1.31	3.00	8.59	11.42	0.00	15.71	46.97	22.62	13.00	136.00	1072.00	123.00	85.00	580.03
Beer	07050046	5.70	1.54	3.00	17.74	35.90	0.65	17.91	69.44	22.69	10.00	23.00	908.00	288.00	94.00	473.18
Beer	07090144	4.44	1.14	1.90	9.71	13.28	0.16	18.52	48.31	11.55	21.00	127.00	261.00	92.00	99.00	345.98
Beer?	07030044	3.06	0.70	2.56	11.93	7.75	0.21	6.67	36.94	4.73	17.50	52.00	373.00	51.00	132.00	317.62

beverage type	N° Eurodat	Alc. Cont. (% vol) Mean	Glycerol (g/L) Mean	methanol (mg/L) Mean	1-propanol (mg/L) Mean	2-methyl-1-propanol (mg/L) Mean	1-butanol (mg/L) Mean	2-methyl-1-butanol (mg/L) Mean	3-methyl-1-butanol (mg/L) Mean	ethyl acetate (mg/L) Mean	Tartric ac. (mg/l) Mean	Malic ac. (mg/L) Mean	Lactic ac. (mg/L) Mean	Acetic ac. (mg/L) Mean	Succinic Ac.(mg/L) Mean	Potassium (mg/L) Mean
fwb	07030026	14.51	3.80	11.75	20.11	36.10	1.10	12.49	61.22	54.89	0.00	3400.00	130.00	220.00	260.00	688.73
fwb	07030054	4.38	2.17	5.25	5.14	35.97	1.54	16.53	92.41	11.58	0.00	1390.00	1770.00	140.00	320.00	413.93
fwb	07030055	4.44	2.11	30.89	10.93	36.17	2.64	16.38	88.41	13.85	0.00	1350.00	1720.00	160.00	260.00	380.37
fwb	07030056	4.47	2.09	3.19	5.03	35.61	1.16	20.66	106.66	16.85	0.00	1390.00	1840.00	200.00	310.00	395.90
fwb	07030057	4.38	2.02	0.66	9.86	4.67	0.07	5.21	33.00	10.93	0.00	360.00	1070.00	120.00	220.00	243.03
fwb	07030058	4.38	1.97	0.95	9.41	4.31	0.00	5.76	30.66	10.44	0.00	470.00	940.00	110.00	260.00	238.06
fwb	07090005	4.33	1.75	1.60	3.79	31.97	1.27	11.20	56.45	6.16	0.00	200.00	650.00	80.00	210.00	132.72
fwb	07090006	4.38	2.41	8.17	9.25	26.65	7.19	5.33	37.20	7.24	0.00	520.00	70.00	80.00	120.00	322.66
fwb	07090007	5.09	2.45	4.77	9.57	31.05	0.65	8.46	37.31	15.92	0.00	510.00	1500.00	180.00	230.00	292.69
fwb	07090008	4.44	2.47	2.54	14.07	27.38	1.29	5.62	33.52	7.49	0.00	530.00	110.00	70.00	110.00	325.39
fwb	07090009	4.42	2.24	0.61	6.76	5.87	0.21	8.16	29.67	3.54	0.00	530.00	320.00	30.00	310.00	300.40
fwb	07090010	3.98	1.90	2.87	11.46	36.04	2.34	9.31	43.99	8.71	0.00	560.00	400.00	120.00	260.00	263.67
fwb	07090011	4.65	1.73	7.50	7.47	6.52	2.61	11.25	32.03	10.45	0.00	570.00	80.00	120.00	160.00	462.92
fwb?	07030023	16.28	2.08	6.99	35.27	31.47	0.00	1.04	3.48	0.87	0.00	0.00	0.00	0.00	0.00	11.29
fwb?	07080004	4.78	1.20	3.03	10.72	11.39	6.33	7.70	23.88	6.25	0.00	290.00	80.00	100.00	140.00	215.85
fwb?	07080005	4.78	1.13	1.78	11.36	11.84	35.03	6.99	22.53	5.95	0.00	280.00	70.00	100.00	150.00	200.73
fwb?	07080006	4.99	1.52	1.70	10.89	10.54	0.00	9.35	33.07	4.74	0.00	1240.00	90.00	90.00	200.00	220.33
mb	07030013	9.26	1.19	2.82	15.00	6.16	0.69	4.49	12.68	10.79	0.00	10.00	50.00	100.00	50.00	149.23
mb	07030014	4.33	1.25	2.15	10.96	10.55	0.37	14.75	47.27	15.72	0.00	2870.00	180.00	690.00	130.00	221.75

beverage type	N° Eurodat	Alc. Cont. (% vol) Mean	Glycerol (g/L) Mean	methanol (mg/L) Mean	1-propanol (mg/L) Mean	2-methyl-1-propanol (mg/L) Mean	1-butanol (mg/L) Mean	2-methyl-1-butanol (mg/L) Mean	3-methyl-1-butanol (mg/L) Mean	ethyl acetate (mg/L) Mean	Tartric ac. (mg/l) Mean	Malic ac. (mg/L) Mean	Lactic ac. (mg/L) Mean	Acetic ac. (mg/L) Mean	Succinic Ac.(mg/L) Mean	Potassium (mg/L) Mean
mb	07030016	4.26	1.80	2.19	8.38	10.88	0.31	10.50	41.06	8.18	0.00	2940.00	190.00	190.00	190.00	231.14
mb	07030021	14.32	1.85	5.71	28.90	18.40	1.04	14.41	65.80	56.77	0.00	0.00	240.00	160.00	350.00	184.00
mb	07030030	3.83	0.73	18.23	30.33	9.30	0.27	9.99	65.85	11.01	0.00	1201.00	1809.00	354.50	170.00	#DIV/0!
mb	07030036	5.28	2.03	6.48	9.69	9.20	0.62	16.08	56.59	13.42	0.00	40.00	205.00	40.00	230.00	302.80
mb	07030037	5.56	1.83	7.03	11.80	12.35	0.95	20.87	64.65	15.96	0.00	35.00	196.50	55.00	150.00	308.59
mb	07030039	6.14	2.09	2.28	11.50	14.73	0.67	23.84	73.44	13.13	3.50	77.00	97.50	75.00	165.00	210.94
mb	07030040	2.78	0.73	2.57	10.03	3.23	0.00	6.06	18.26	4.87	13.50	48.50	195.00	47.50	375.00	307.37
mb	07030047	4.07	0.85	12.23	34.71	11.79	0.37	11.05	70.55	4.37	30.00	1164.50	3107.00	304.50	139.00	763.92
mb	07030049	3.72	1.17	2.93	15.33	4.08	0.00	5.29	12.34	3.13	1.50	14.50	54.00	13.00	6.00	80.69
mb	07030050	3.71	1.13	2.33	14.75	4.50	0.00	5.52	12.81	2.62	3.00	16.50	177.50	41.50	64.00	81.47
mb	07030052	3.00	0.82	1.00	8.03	4.78	0.00	7.98	25.38	5.75	6.00	33.00	301.00	83.00	64.00	350.99
mb	07090150	7.39	0.00	2.34	13.00	20.09	1.17	25.81	72.48	8.38	0.00	0.00	0.00	0.00	0.00	0.55
mb	07090152	7.10	0.00	1.60	11.30	17.42	0.25	21.30	57.13	14.15	21.00	23.00	26.00	23.00	27.00	0.00
mb ? beer?	07030028	3.36	0.88	34.00	8.30	11.17	0.26	11.18	33.35	13.54	33.00	579.00	815.00	290.00	67.00	913.74
mb ? beer?	07030029	4.97	2.06	4.20	27.80	27.61	1.11	24.26	86.54	30.04	5.00	108.00	92.00	95.00	175.00	496.64
mb+alc	07030042	3.06	1.11	3.60	6.26	7.40	0.32	11.75	37.19	5.07	14.50	35.50	89.50	43.00	148.00	173.09
wb	07030022	16.33	6.47	11.67	51.21	145.52	0.45	44.65	138.78	8.57	0.00	0.00	180.00	20.00	20.00	93.72
wb	07030031	5.59	2.81	24.32	11.24	11.37	0.45	4.29	17.18	14.48	764.50	44.00	1705.00	143.50	476.00	432.60
wb	07030033	5.08	3.07	39.31	15.24	19.89	0.29	13.62	56.40	9.24	2484.50	19.00	1038.50	68.50	398.00	511.18

beverage type	N° Eurodat	Alc. Cont. (% vol) Mean	Glycerol (g/L) Mean	methanol (mg/L) Mean	1-propanol (mg/L) Mean	2-methyl-1-propanol (mg/L) Mean	1-butanol (mg/L) Mean	2-methyl-1-butanol (mg/L) Mean	3-methyl-1-butanol (mg/L) Mean	ethyl acetate (mg/L) Mean	Tartric ac. (mg/l) Mean	Malic ac. (mg/L) Mean	Lactic ac. (mg/L) Mean	Acetic ac. (mg/L) Mean	Succinic Ac.(mg/L) Mean	Potassium (mg/L) Mean
wb	07030034	6.81	3.25	27.83	12.33	14.01	0.81	11.85	48.98	18.31	2433.50	53.00	1233.00	111.00	250.00	452.50
wb	07030043	4.90	2.52	32.57	12.94	12.78	0.23	10.14	47.50	11.13	1993.50	33.50	979.50	179.50	176.00	487.06
wb	07030045	5.77	3.44	43.31	17.84	23.77	11.69	15.01	64.18	12.92	3449.50	3516.50	1214.00	207.50	250.00	583.33
wb	07030046	5.94	3.09	36.55	12.19	14.24	0.27	11.66	55.82	14.15	5003.50	31.50	1169.00	227.00	225.00	591.16
wb	07030048	6.79	3.05	29.25	11.89	14.28	0.79	12.35	49.60	14.97	2367.50	51.50	928.50	216.00	209.00	458.71
wb	07030051	5.70	8.32	13.80	25.10	17.84	0.58	18.00	98.50	18.13	2046.00	1955.00	63.00	254.00	405.00	1055.15
wb	07050029	9.40	6.10	101.80	27.39	38.82	1.71	33.18	128.50	42.87	1528.00	346.00	1940.00	386.00	464.00	896.56
wb	07090151	7.57	0.00	46.28	9.63	19.57	0.36	14.56	60.38	32.08	0.00	0.00	0.00	0.00	0.00	0.44
wb	07090153	7.64	0.00	33.80	19.75	14.54	0.29	9.41	50.07	23.64	18.00	47.00	12.00	26.00	18.00	1.09
alc	07030059	4.82	0.00	1.63	0.00	0.00	0.00	0.00	0.00	0.21	0.00	0.00	0.00	0.00	0.00	3.05
alc	07030060	5.05	0.00	0.45	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	5.12
alc	07030061	4.91	0.00	0.86	0.00	0.00	0.00	0.18	0.28	0.12	0.00	0.00	0.00	0.00	0.00	3.79
alc	07030062	4.98	0.00	4.23	0.00	2.22	0.00	0.00	0.00	1.38	0.00	0.00	20.00	0.00	0.00	3.70
alc	06080001	6.35	0.00	4.20	0.00	0.00	0.00	0.00	0.00	0.21	0.00	0.00	0.00	0.00	0.00	109.60
alc	06080002	4.79	0.00	1.84	0.00	0.00	0.00	0.00	0.15	0.00	0.00	0.00	0.00	0.00	0.00	69.29
alc	06080003	3.80	0.00	2.85	0.08	0.00	0.00	0.00	0.00	0.12	0.00	0.00	10.00	0.00	10.00	168.76
alc	06080004	3.82	0.00	4.08	0.00	0.00	0.00	0.00	0.13	0.13	0.00	10.00	0.00	0.00	0.00	256.13
alc	06080005	4.89	0.07	2.33	3.77	5.36	0.66	2.78	12.54	10.94	0.00	80.00	0.00	0.00	0.00	30.01
alc? fwb?	07030035	4.77	0.00	1.24	0.08	0.07	0.00	0.00	0.30	0.16	125.00	40.00	13.95	0.00	0.00	15.31
alc? fwb?	07030038	4.91	0.00	1.32	0.12	0.04	0.00	0.00	0.23	0.12	145.00	60.00	5.00	0.00	10.00	11.16
alc? fwb?	07030041	4.80	0.00	2.87	0.22	0.03	0.00	0.00	0.23	1.71	120.00	40.00	17.50	0.00	0.00	13.21

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Abstract

The purpose of the ACAP working group (composed of JRC, TAXUD and 11 Member States Custom Laboratories) was to provide scientific support for the resolution of the issue of divergent classifications of alcoholic beverages for excise duties, whether by providing objective criteria for classification, or providing sound scientific information for revision of legislation. In particular, there is a need for analytical methods allowing for the differentiation between "fermented" and "distilled" ("non-fermented") beverages and for the detection of ethanol addition. At present there is no commonly agreed solution of these problems which has resulted in the Customs Laboratories of the Member States developing their own particular approaches to deal with these issues. This can lead to divergent classification between Member States and consequent problems both for administrations and the trade.
Two complementary pathways of investigation have been examined in this study: NMR fingerprinting and "classical analysis", both handled by multivariate data analysis.
It is the findings of this study that it is likely to be extremely difficult or even impossible to create workable “general” models based on analytical methods. Unfortunately, the difficulties are the greatest precisely with those categories that are the most problematic for excise classification purposes. Even if an analytical model was eventually developed it is likely to need to be an extremely complex mechanism in order to take account of the broad range of beverages, and diverse chemical characteristics, of products that fall within the problem categories ("other fermented beverages" etc.). On the issue of 'added alcohol' the findings reinforce that is not plausible to ascertain the amounts by a scientific study of the finished product only. The case-to-case approach to classification applied by Member States remains therefore the only possible approach in the context of the current legislation.

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