FOODINTEGRITY HANDBOOK

A GUIDE TO FOOD AUTHENTICITY ISSUES AND ANALYTICAL SOLUTIONS

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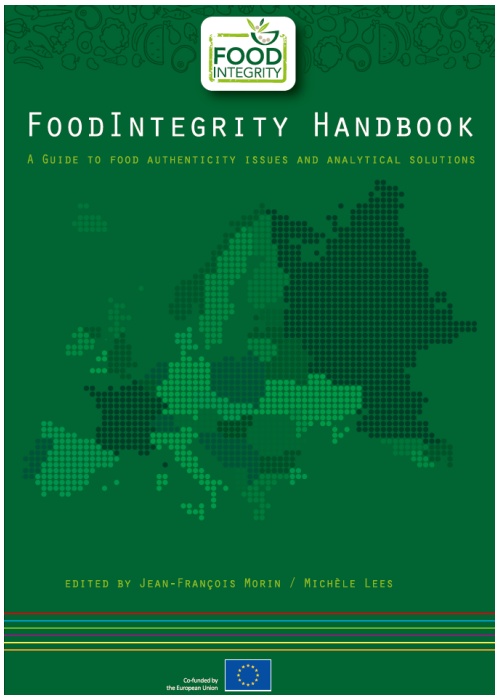

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Spirit drinks

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General overview of the products

Spirit drinks are a significant category of food product when considered economically, legally and culturally. Economically, spirit drinks represent an important outlet for agricultural production, and generate considerable revenues for the public purse via excise duty and other taxes. Their cultural and economic importance is reflected both in the large number of key rulings in the development of European Union (EU) food law that relate specifically to spirit drinks, and the unique protection their geographical indications hold within trade law.

The economic significance of spirit drinks can be seen in a number of metrics. In the EU, spirit drinks are the largest agri-food export with almost two-thirds of the sector's production being exported, contributing to a positive balance of trade of around EUR 9 billion [1-3]. The spirit drinks sector also contributes around EUR 23 billion annually to the EU in excise duties and VAT and around 1 million jobs can be linked to the production and sales of its products [2].

Foods associated with specific geographical areas are of great economic importance and this has led to the introduction of systems by which geographical indications (GIs) can be protected. The cultural significance of many spirit drinks is indicated by the large number of associated GIs registered in this sector ([4, Recital 2]). At an international level, the agreement on Trade-Related Aspects of Intellectual Property Rights (TRIPs) [5] is arguably the most important international treaty offering protection for GIs. This notably offers advanced protection to the GIs for spirits (and wines) compared to other agricultural products, underlying the national importance of these products.

To sustain its importance in global trade, the spirit drinks industry needs to maintain consumer confidence in its products. One of the key areas in supporting this position is by offering assurance that spirit drinks sold in global markets are authentic. Spirit drinks are excisable food commodities and often command premium price tags, which adds considerably to their allure to the counterfeiter. Excise duty is added to alcoholic products in most countries [6], and spirit drinks are often taxed at higher levels than other alcoholic beverages [7,8]. Counterfeit products will be produced without consideration of excise requirements and consequently offer large profits to the

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counterfeiter. In the UK, it has been estimated that the Treasury loses GBP 1.3 billion annually through alcohol fraud [9].

An effective legal framework for tackling the production and marketing of fraudulent spirit drinks requires two elements. The first element is a clear and enforceable set of definitions of the spirit drinks categories, including their production processes and specific analytical and organoleptic characters. The second element is a range of appropriate analytical methods that will help confirm that a suspect spirit drink product meets its labelling claims, according to the legally established definitions. These two elements, as well as an overview to the common spirit drink frauds such a system is designed to tackle, are explored in the rest of the chapter.

1. Product Identity

1.1. Definition of the product and manufacturing process

Spirit drinks can be simply defined as alcohol beverages created from the distillation of fermented agricultural raw materials. Exact terminology and definitions will vary on jurisdiction, but these key elements, along with the requirement that the distillate be intended for human consumption (potable), will be common to most markets.

1.2. Current Standards of Identity and Related Legislation

1.2.1. European Union – Spirit Drink Categories

In the European Union, the current definition of a spirit drink is contained in the Spirit Drinks Regulation 110/2008 [4] on the definition, description, presentation, labelling and the protection of geographical indications of spirit drinks[1]. This regulation (Article 2) defines a spirit drink as an alcoholic beverage that is:

(a) intended for human consumption;

(b) possessing particular organoleptic qualities; and

(c) having a minimum alcoholic strength of 15 % vol[2].

and contains a distillate of a naturally fermented agricultural product.

None of the alcohol contained in a spirit drink shall be of synthetic or non-agricultural origin (Article 3(4)). The nature of the raw material that may be considered agricultural in origin is contained in the Treaty on the Functioning of the European Union (TFEU) [10] in Annex I.

There are three types of distillate defined within the Spirit Drinks Regulation [4]. The first is one that meets the definition of a spirit drink, as outlined above. The second is ethyl alcohol of agricultural origin (EAAO), a highly rectified distillate meeting specific technical requirements [4, Annex I(1)], including a minimum alcoholic strength of 96.0 % v/v[3]. This creates a distillate that is

[1] Regulation 110/2008 is, at the time of writing, currently undergoing revision. However, it is assumed that most, if not all of the points noted in this chapter will be retained in any forthcoming legislation.

[2] With the exception of egg liqueur or advocaat or avocat or advokat where the minimum strength is 14 %.

[3] The EU regulation does not specifically state that EAAO has to be a *distillate* of agricultural ethanol, but (given the minimum strength) this is effectively the case.

designed to be low in compounds other than ethanol and water, and consequently neutral in its flavour profile. The last category of distillate is a "distillate of agricultural origin". This covers any agricultural distillate that does not meet the criteria for a spirit drink or ethyl alcohol of agricultural origin. Spirit drinks can be created directly from a distillation of naturally fermented products, or can be produced from appropriate treatments of EAAO, distillates of agricultural origin or other spirit drinks [4, Article 2].

Within the Spirit Drinks Regulation [4, Annex II] there are 46 defined categories of spirit drinks. The first 14 of these have certain restrictions placed on their production [4, Article 5]. These include the sole use of the raw material contained within the category definition for the production of alcohol, a prohibition on the use of EAAO and flavourings, and restrictions in relation to colouring and sweetening. Examples of such spirits include rum, whisky and brandy. Unless otherwise stated in their category definitions, the remaining 32 categories may use any agricultural raw material as the origin for the alcohol, EAAO, as well as any permitted flavourings, colourings and sweeteners. All alcoholic beverages which meet the definition of one of the 46 spirit drink categories must "bear in their description, presentation and labelling the sales denomination assigned therein"; for those spirit drinks that do not fall into one of the 46 categories, they "shall bear in their description, presentation and labelling the sales denomination 'spirit drink'." [4, Article 9(1-2)].

It is notable that the European legislation for the definition of spirit categories are typically process definitions. Whilst all spirit drinks categories specify minimum alcohol strength, few additional analytical parameters are set in the legislation against which compliance can be judged - examples include:

- limits for anethole concentration in pastis, pastis de Marseille, sambuca and Mistrà;
- limits for sugars in various liqueurs and the spirit drink Berenburg/Beerenburg; and
- a minimum egg yolk content in Egg liqueur/advocaat/avocat/advokat or liqueur with egg.

1.2.2. European Union – Geographical Indications

The Spirit Drinks Regulation provides for the ability to apply stricter definitions for locally produced spirit drinks [4, Article 6(1)]. This typically applies to the production of geographical indications, for example United Kingdom legislation [11] defines a tighter production specification for whisky produced in Scotland. The sales denomination as described above may be replaced or supplemented with a geographical indication [4, Chapter III; 12]. Given the economic importance of GIs, their use is often subject to fraud, and any additional production specifications should be considered when determining whether a suspect product is consistent with a GI sales denomination. The Spirit Drinks Regulation requires that GIs are produced in accordance with the specifications contained within an associated technical file and that this is verified by an appropriate body [4, Article 22].

The Spirit Drinks Regulation [4] and the associated regulation detailing Union Reference Methods for spirit drinks [13] provide the basis for authenticating spirit drinks in the EU. GI verification, however, has also been exploited in the protection of the sector. The Scotch Whisky Verification Scheme [14], for example, comprises three interlocking elements offering additional protection to this whisky GI. Firstly, the scheme provides an audit of all production facilities, including importers of bulk Scotch Whisky (on a 2-year cycle) via documentary and physical checks according to defined requirements [14, Annex B]. Secondly, a tight chain of authenticity is provided by the requirement that a production facility, in whatever location, cannot pass product to another unless it has been verified. Finally, a register that lists all the verified production facilities and importers is

available on the Her Majesty's Revenue and Customs website [15], as well as a register of all brands produced at verified sites. Such a scheme offers both protection against fraud, and a market opportunity for guaranteeing authenticity, and has been looked at by other EU spirit producers, e.g. Swedish vodka and Dutch gin [16].

1.2.3. Spirit Drink Legislation outside the European Union

The harmonisation of spirit drink definitions across the European Union (first introduced in 1989) and the introduction of geographical indications can be seen as necessary support measures for rural communities and an important sector of agricultural produce [17]. This has led to a detailed set of regulations, with specific process definitions for categories and geographical indications.

Other jurisdictions typically have significantly less extensive ranges of spirit drink categories and fewer restrictions on methods of production. Unsurprisingly, a range of conflicts are established by different cultural expectations of the characteristics possessed by certain spirit categories. This can be seen by considering one of the category definitions – whisk(e)y – in some key pieces of national legislation that apply to spirit drinks.

According to the European Union definition [4, Annex II (2)]:

(a) Whisky or whiskey is a *spirit drink produced exclusively by:*

- *(i) distillation of a mash made from malted cereals with or without whole grains of other cereals, which has been:*
  - *saccharified by the diastase of the malt contained therein, with or without other natural enzymes,*
  - *fermented by the action of yeast;*
- *(ii) one or more distillations at less than 94.8 % vol., so that the distillate has an aroma and taste derived from the raw materials used,*
- *(iii) maturation of the final distillate for at least three years in wooden casks not exceeding 700 litres capacity.*

The final distillate, to which only water and plain caramel (for colouring) may be added, retains its colour, aroma and taste derived from the production process referred to in points (i), (ii) and (iii).

(b) The minimum alcoholic strength by volume of whisky or whiskey shall be 40 %.

(c) No addition of alcohol as defined in Annex I(5)[4]*, diluted or not, shall take place.*

(d) Whisky or whiskey shall not be sweetened or flavoured, nor contain any additives other than plain caramel used for colouring.

1.2.3.1. Canada

The Canadian Food and Drug Regulations [18] define 8 different spirit categories (whisky, rum, gin, brandy, liqueurs and spirituous cordials, vodka, tequila and mezcal), although provision is made for the protection of a number of geographical indications, such as Scotch Whisky, Bourbon Whiskey, Cognac, Armagnac and Grappa. The Canadian definition of whisk(e)y is an example of a specification that approximates closely to the European Union definition.

[4] This is *ethyl alcohol of agricultural origin* (EAAO), a highly rectified distillate meeting specific technical definitions and requirements (Regulation 110/2008, Annex I(1)), including possessing a minimum alcoholic strength of 96.0% v/v.

For example, in Canada, whisk(e)y is defined as:

"*a potable alcoholic distillate, or a mixture of potable alcoholic distillates, obtained from a mash of cereal grain or cereal grain products saccharified by the diastase of malt or by other enzymes and fermented by the action of yeast or a mixture of yeast and other micro-organisms and may contain caramel and flavouring*" [18, B.02.010]

and

"*no person shall sell for consumption in Canada any whisky that has not been aged for a period of at least three years in small wood*" [18, B.02.023(1))]

where small wood is defined as "wood casks or barrels of not greater than 700 L capacity" [18, B.02.002]

However, though many of the provisions are similar there are notable differences: the Canadian whisk(e)y definition does not define a maximum distillation strength, thus allowing the inclusion of highly rectified alcohol, akin to EAAO; there is no minimum alcohol strength (although Canadian Whisky itself and other geographical indications, such as Scotch, all set a minimum of 40 %); malt is not an essential component of the saccharification process; and limited flavouring of whisky can take place. Such differences will alter the range of flavour and analytical profiles covered by the definition.

1.2.3.2. The United States

The spirit drinks definitions in the United States are within Part 5.22 in Title 27 of the Code of Federal Regulations, which governs food and drugs [19]. As with the European Union and Canada, categories of spirit are defined by their manner of production. These include: neutral spirits (the US equivalent of EAAO, including vodka), whisk(e)y, gin, brandy, blended applejack (apple brandy), rum, tequila, cordials and liqueurs, flavored spirits (brandy, gin, rum, vodka, and whisky). There are a number of geographical indications specifically referenced within these categories (e.g. Scotch Whisky, Canadian Whisky, Pisco and Cachaça) as well as some standards of identity that are culturally significant to the U.S. market (e.g. blended applejack and corn whisky).

As with the Canadian definition, the U.S. standard of identity for whisk(e)y is very similar to the European definition. It states that (5.22 (2)(b)):

"'Whisky' is an alcoholic distillate from a fermented mash of grain produced at less than 190° proof in such manner that the distillate possesses the taste, aroma, and characteristics generally attributed to whisky, stored in oak containers (except that corn whisky need not be so stored), and bottled at not less than 80° proof, and also includes mixtures of such distillates for which no specific standards of identity are prescribed."

In this case, there is a minimum alcohol strength equivalent to 40 % alcohol by volume and a maximum distillation strength of 95 % alcohol by volume (just 0.2 % above the EU limit). It is also slightly more specific in the wooden containers used for maturation, although the worldwide use of wood other than oak for maturation is negligible. However, there is increased latitude in other areas of the definition compared to the European Union. No minimum maturation time is specified for whisk(e)y in general, and whilst American straight whiskies require at least 2 years storage in oak containers, this is less than the 3 years associated with European whiskies. Corn whisky does not need to be matured at all. An additional section (5.23) also provides for the qualified addition of harmless colouring, flavouring, or blending materials such as caramel, sugar and wine, a much wider range of materials than the sole permitted additive of plain spirit caramel in the EU.

1.2.3.3. Australia

Other jurisdictions have much looser definitions than those seen above. An example is Australia, where there are certain provisions under the *Australia New Zealand Food Standards Code* which govern spirits where these are manufactured or imported into Australia. Principally, Standard 2.7.5 [20] defines brandy, liqueurs and spirits in general. All definitions are light on details of production, in particular the definition for spirits (2.7.5-2), given that only 2 categories are specifically referred to in the same legislation. This states that a spirit means an alcoholic beverage consisting of:

(a) a potable alcoholic distillate, including whisky, brandy, rum, gin, vodka and tequila, produced by distillation of fermented liquor derived from food sources, so as to have the taste, aroma and other characteristics generally attributable to that particular spirit; or

(b) such a distillate with any of the following added during production:

(i) water;

(ii) sugars;

(iii) honey;

(iv) spices.

In addition, all spirits have a minimum alcohol strength of 37 % alcohol by volume (2.7.5-3). As can be seen, such a definition, for whisk(e)y say, provides little in the way of specifics about the production methods of the spirit, so long as a subjective organoleptic assessment indicates the standard of identity has been met, and there is a lower minimum strength than in Europe. In addition, a number of additives such as sugar, honey and spices are permitted under the definition, contrary to European legislation for whisk(e)y.

Some additional process information can be found in the Excise Act 1901 [21, Section 77FI] and the Customs Act 1901 [22, Section 105A]. However, these are similarly light on detail compared to their European definitions. Both pieces of legislation provide minimum maturation requirements for brandy, rum and whisky. However, these are limited to the requirements that these spirit types are stored for a minimum of 2 years in wood. They also define the materials for the production of brandy (grape wine), rum (a fermented liquor derived from the products of sugar cane) and whisky (a fermented liquor of a mash of cereal grain).

However, the Australia New Zealand Food Standards Code does contain explicit protection for spirit drink geographical indications, including a specific requirement that products produced in accordance with a geographical indication, but shipped and bottled elsewhere, must meet the minimum alcohol strengths of the laws relevant to the geographical indication.

1.2.3.4. India

India has (based on the regulations to be enforced from April 2019 onwards) a number of spirit category definitions [23], including brandy, gin, rum, vodka, liqueurs/cordials/aperitifs and whisk(e)y. Like other jurisdictions, a number of culturally significant definitions are included: country liquors, fenny and pot distilled spirits. A key example of the conflict between different cultural perceptions of a spirit category can be seen in the whisky definition [23, Section 2.8]). Whilst placing an emphasis on cereal being the raw material for whisky production, it is clear that whisky can also be made from neutral spirit, which can be made from fruits, vegetables, molasses or any other source of carbohydrates of agricultural origin, as well as grains and has a minimum alcohol strength of 96 % alcohol by volume [23, Section 1.2.9]. This is a clear contradiction to most other definitions of whisk(e)y, which require a cereal substrate, a maximum distillation strength (to retain an appropriate level of organoleptic character from the raw material) or both.

India is also a country that has imposed a number of analytical limits on the spirit beverages it defines [23 Table 1]. These are individually tailored to each category of spirit. Some of these limits are obviously intended to act as a general restriction on compounds of public health concern, such as the levels for heavy metals, although it is unclear why spirits produced according to good distilling practice should ever be at risk of exceeding such limits, and thus why such category-based limits are required.

The inclusion of other varying limits based on spirit category (such as total esters and higher alcohols) are more typical of quality-based specifications, but unlike the European regulations that relate limits to some characteristic of a particular product category (e.g. the high sugar content of liqueurs or the specific flavouring requirements of aniseed spirits such as Pastis) these are applied to each category in turn. Thus, the limits in effect represent an attempt to provide an analytical definition to a product category. Such limits, whilst seemingly providing some guidance as to appropriate analytical range for authentic products, should be treated with caution. Whilst usually covering a large proportion of a category, they do not always include all the various styles and variations contained within a spirit drink category definition. They can therefore act as misleading guides to authentic database ranges and may also restrict trade in genuine products.

2. Authenticity issues

2.1. Identification of current authenticity issues

2.1.1. Brand and Generic Counterfeits

There are many forms of spirit drink fraud. Some frauds may be simple product substitutions, where authentic spirit drink bottles are refilled with cheaper, poor quality replacements. Other counterfeiting operations can be very sophisticated, involving products deliberately created to avoid detection by the analytical investigator. However, when considering spirit drink fraud there are two broad categories of counterfeit produced, brand counterfeits and generic counterfeits[5].

A **brand counterfeit** fraudulently trades on the reputation associated with a particular brand of spirit drink. It could involve the direct copying of the brand packaging and filling with non-authentic liquid. Alternatively, brand counterfeiting could comprise the collection of authentic used packaging, refilling it with counterfeit product, and application of new closures.

A **generic counterfeit** product fraudulently trades on the premium quality associated with a defined category of spirit drink, e.g. Scotch Whisky, Cognac or Vodka. Whilst not claiming to be a brand with a recognised reputation in the marketplace, it will use a familiar category definition of spirit drink in its labelling to command added value to which it is not entitled. Geographical indications are often the targets of generic counterfeiting, due to the associated reputations of these spirit drinks. Such frauds may be explicitly signalled in the labelling by use of the regional name (e.g. Armagnac, Scotch Whisky) or implicitly indicated using brand names or imagery associated with that area.

[5] Counterfeit alcohol is just one type of illegal alcohol. For information on the correct terminology to use when discussing legal and illegal alcohol refer to the resources produced by The International Alliance for Responsible Drinking [24], in particular the section on taxonomy of the alcohol market [25].

The two cans shown in Figure 1 claimed to be Scotch Whisky and are examples of generic counterfeits, trading on the goodwill associated with that geographical indication. These products were canned in Austria and sold in the Middle East. They were manufactured from industrial alcohol and flavouring. In total, it has been estimated that 15 million of these cans were sold over a period of a few years, which provides an indication of the scale of some of these spirit drink counterfeiting operations.

For both generic and brand counterfeiting, the liquid inside the bottle is often the extension (dilution) or replacement of the authentic product with: (i) water; (ii) cheaper locally produced spirit, (iii) neutral alcohol (a highly rectified spirit lacking in flavour, used as a base to produce many genuine spirits) or (iv) an alternative alcohol. These products may also contain added sweetening or flavourings to mask the inferior flavour of the counterfeit spirit or to mimic aromas of the genuine spirit.

Figure 1: Examples of Counterfeit Scotch Whisky

2.1.2. Subsitution with cheaper brands and water

Aylott [26], in his review of modes of spirit drink counterfeiting, highlights the practice of product substitution, the swapping of a higher value branded product for a value product of the same category. Previous references have been made in his work to both gin and whisky product substitution [27,28]. Lachenmeier [29] notes that brand fraud has been observed in restaurants and bars, especially in establishments that sell very cheap alcohol. In such cases, the bar operator may refill bottles of branded spirits with cheaper brands of the same type of spirit (in Germany often from so-called discount stores) [30]. Additionally, dilution of the branded spirits or cheaper spirits with water may occur. Spirit products may gain significant additional value by virtue of their rarity or age. Counterfeit spirits may be produced to take advantage of such elevated prices paid for in rare spirit auctions. Both the packaging and the liquid may be analysed to identify whether their ages are consistent with any labelling claims.

2.1.3. Subsitution with other forms of alcohol

The illegal production of spirit drinks is often carried out by simple substitution or dilution of authentic beverages with alcohol, adjusted to the appropriate strength with water, or a mixture containing such alcohol with colouring and/or flavourings ([31,32]. Different types of alcohol may be used in this process: distillates of agricultural origin, including highly rectified products such as

neutral spirit or ethyl alcohol of agricultural origin; synthetic alcohol; some alternative alcohol such as methanol; or industrial alcohol.

The alcohol used for extension or substitution may be from non-permitted agricultural substrates, i.e. the botanical origin of the alcohol is incorrect. For example, rum can only be created from sugar cane by-products or sugarcane juice, according to most spirit drinks legislation. The identification of alcohol from different agricultural origins will signify a fraudulent product [33, pages 18-19]. Whilst constituents of the distillates of the incorrect agricultural origin may make their presence detectable in a fraudulent product [34] much work has been undertaken on the use of the stable isotopic ratios of ethanol and water to detect this fraud [35-38]. Isotope ratios are of particular importance when the product is fraudulently substituted or diluted with highly rectified neutral spirit and for reasons of natural variability the levels of components in the authentic product are insufficient to detect this practice [39]).

The use of alternative alcohols added to potable ethanol from agricultural substrates is particularly attractive to spirit drink counterfeiters, since there is no excise duty to be paid. Synthetic alcohol has been used to produce counterfeit spirits. For example, tequila made from synthetic alcohol (probably derived from petroleum) has been identified [33, page 20], as has the falsification of vodka using synthetic ethanol [40]. Denatured alcohol is used in a number of industrial applications. This product, exempt from excise duty after the addition of specific chemicals (denaturants) designed to render the alcohol non-potable, has also been used as the base alcohol for counterfeit products [33, page 10]). Whilst the denaturants are often added to specifically mark a product as denatured alcohol, counterfeiters will often attempt to remove these compounds, thus recovering the alcohol in an unmarked form and making its presence in counterfeit spirits hard to identify [41].

2.1.4. Additives

Counterfeit products may also contain added sweetening or flavourings to mask the inferior flavour of the counterfeit spirit or to mimic aromas of the genuine spirit. Depending on the legislation relevant to the spirit category, these additives may not be permitted in the genuine products. An example of this is where sugars are illegally added to whisky [31].

2.2. Potential threat to public health

In some cases, counterfeit product can be dangerous and pose a risk to consumer health, particularly when non-potable alcohols such as methanol or denatured alcohol are employed.

Methanol (methyl alcohol) is potentially toxic. Its consumption can cause blindness, other severe health complications and death. A maximum tolerable concentration for methanol in alcoholic beverages has been estimated as 2 % by volume in a 40 % spirit drink [42]. Methanol occurs naturally in most alcoholic beverages at levels without any danger to public health. However, there are many recorded incidents of its harmful presence in counterfeit spirit drinks [43]. Its presence is most likely to have been introduced to the illicit beverage by a counterfeiter who hopes to profit from methyl alcohol's lower cost compared to ethyl alcohol [29,44].

In 2011, in the UK, 10 people became ill after consuming vodka that smelled of nail varnish remover. Twelve thousand litres were seized, and the product was found to have contained high levels of methanol that had been diluted into the final product [45]. In 2012, methanol poisoning incidents were reported in the Czech Republic from the consumption of deliberately adulterated

spirits. To contain the problem the Czech authorities temporarily banned the consumption of spirits above 20 % alcohol by volume. At least 36 deaths were related to this incident [29].

Another health risk that can arise is from metals used in illicit stills and other production materials that are unfit to come into contact with food products. Genuine producers will take steps to prevent any unwanted contamination from metals and plastics or other food contact materials that could leach into the final products. Counterfeiters are either unaware of these risks or are not concerned enough for the health of their customers. Where industrial alcohol has been substituted into the food chain similar concerns occur, with the added health impact of the chemicals used to denature the alcohol. Elevated levels of metals in illicit alcohol include metals such as lead, arsenic and mercury. These have been linked to makeshift illicit distilling apparatus using a variety of reused metal components that may leach harmful toxins into the distillate, including the aforementioned metals [43,46-48].

Chloroform has been detected at high levels in illegally produced alcoholic products [49], creating an increased risk to the public. This could be due to a process used by some counterfeiters to remove the common denaturant denatonium benzoate, which has a unpalatable bitter taste, from denatured alcohol, via the addition of hypochlorite [50,51]; chloroform is known to be a product of hypochlorite and ethanol [52]. Other denaturants may also have health impacts, to a greater or lesser degree. The impact of methanol has already been noted; other compounds, whilst not exhibiting acute toxicity would still be regarded as unwanted contaminants of toxicological significance [43].

3. Analytical methods used to test for authenticity

There are many well-established methods available for authentication of spirit drinks. However, not all methods will be applicable to every authentication challenge. As noted previously, there are two principal types of counterfeit activity within the spirit drinks sector, generic counterfeits and brand counterfeits. A variety of analytical techniques can be employed to confirm whether a suspect sample is consistent with its production requirements, for the detection of generic counterfeits. This includes a set of official EU reference methods contained within their own regulation [13] and developed specifically to test parameters listed within the EU regulation that defines spirit drinks categories [4]. Some spirit categories are often too variable in terms of composition to permit generic authentication, although if some form of analytical limit is defined in legislation, this can be of assistance. The EU definition of liqueurs, for example, allows for a wide variety of product formulations; despite the limits on minimum sugar content, this will be insufficient to define such a varied category.

The analytical methods frequently used to authenticate spirit categories can be applied to a more tightly defined set of parameters associated with a particular brand to identify brand counterfeits.

Spirit drinks authenticity analyses can identify with certainty if a product is not genuine (e.g. because the results are inconsistent with a particular brand or the production parameters contained within a spirit category definition). However, they will never be able to confirm with certainty that a suspect product is genuine, only that, based on the tests undertaken, the suspect product is *consistent* with a genuine product. Similarly, spirit drinks authenticity analyses will be unable to confirm, with certainty, the actual nature of a non-genuine product.

Category authentication, brand authentication and screening technologies all have their specific uses and applications. The aim of this section is to assist in spirit drink authentication by providing supplemental information, references and guidance for analytical methods commonly employed. The officially recognised methods referred to herein relate to three main sources of reference methods: Commission Regulation (EC) No 2870/2000 (Union reference methods of analysis) [13], the OIV Compendium of International Methods of Spirit Beverages of Viticultural Origin [53] and the AOAC International Official Methods of Analysis [54], which are typically the methods of analysis referred to by the U.S. Alcohol and Tobacco Tax and Trade Bureau (TTB). The methods in the OIV are often aligned with those in the Union reference methods. Some national markets will have their own official methods, which will often be variations of the techniques referred to in the above standards. Where significantly differently methods are employed, it would be advisable to demonstrate equivalency to the common methods referred to here.

3.1. Officially recognised methods

3.1.1. Alcoholic Strength

The measurement of alcoholic strength is important for quality control and product integrity, but also has implications in terms of regulation and excise duty. As noted, all the spirits defined within the EU spirit drinks definitions have a minimum alcohol strength requirement with which genuine products need to comply. Dilution with water below the minimum alcohol strength limit, or significant differences between the label and measured alcohol strength (e.g. outside acceptable tolerances as given in [55, Annex XII]) are key indicators of counterfeit products.

Accepted reference methods are based on the measurement of liquid density (densitometry), following a prior distillation step. Key reference methods are documented in the Union reference methods (Annex I) and the aligned OIV methods (OIV-MA-BS-01 to OIV-MA-BS-05). Following the distillation step, samples can be analysed by one of three types of densitometry methods (pycnometry, electronic densitometer, and hydrostatic balance). Pycnometry and densitometer methods are detailed in the TTB recommended AOAC methods for distilled spirits (942.06, 945.07, 982.10 and 983.12).

The distilled samples under analysis are assumed to be mixtures of pure ethanol and water; hence the density of the liquid can be directly related to the alcohol strength. Conversion to alcoholic strength is carried out using official alcohol tables (manually or automatically). In the EU, all measurements are based on the density of alcohol and water at 20°C. The principle tables for conversion in the EU are the International Alcoholometric Tables prepared by the International Organisation of Legal Metrology (OIML) [56]. The AOAC density measurements are however all taken at 15.56°C (60°F) which produces a slightly different alcohol strength by volume from the EU measurements (0.07 % difference at 40 % alcohol by volume).

The identified reference methods are by default measured after a distillation step. This is referred to as a real, true or actual alcohol strength measurement. Direct analysis of the spirit has been found to be satisfactory for samples such as vodka, gin and whisky. However, it cannot be used for samples that contain high levels of non-volatile material such as sugars, creams or wood extractives, as these compounds affect the density measurement and thus the reported alcohol strength. Such products need be distilled prior to density measurement to obtain the actual strength. If a high degree of accuracy is required or there is some uncertainty as to whether the product contains non-volatile material that might obscure the density measurement, an actual or real strength should be carried out.

Spectroscopic methods involving near-infrared (NIR) are commonly used within the industry to provide alcohol strengths, since real strengths can be obtained for many distilled spirit matrices without the need for a distillation step. However, only the OIV currently has an official method detailing the application of NIR spectrometers (OIV-MA-BS-08). This method of determining the real alcoholic strength is based on the physical principle of the spectral analysis of materials with absorption bands in the near infrared range. The key point about the use of such apparatus is that, as noted in the method, the NIR equipment needs to be appropriately calibrated and verified against an appropriate reference set of samples, measured using an approved reference method for real strength as referred to above.

3.1.2. Major Volatile Congeners

The major volatile congeners are principally produced during the fermentation stage of spirit production and carried over via the distillation process to the resulting spirit. They represent, as the name suggests, a subset of compounds at relatively high levels in distilled spirit (typically at ppm levels). The level of these congeners and their proportions to each other can lead to conclusions regarding the production process, hence their use in authentication and quality control. A smaller subset of these compounds are the higher alcohols, which are often used for the same purposes.

The officially recognised methods of analysis use Gas Chromatography (GC) with Flame Ionisation Detection (FID) for detection of most of the major volatile congeners. The Union reference version (Annex III.2) measures selected aldehydes (acetaldehyde and ethanal), higher alcohols (propan-1-ol, butan-1-ol, butan-2-ol, 2-methylpropan-1-ol, 2-methylbutan-1-ol and 3-methylbutanol-1-ol), ethyl acetate and methanol. The OIV aligned method is OIV-MA-BS-14. The AOAC methods 968.09 and 972.10 measure specific higher alcohols and ethyl acetate; a separate method measures methanol (972.11). The Union reference methods also includes volatile acidity (measured as acetic acid) in its measurement of volatile substances (Annex III.3), the levels of which, whilst affected by fermentation and distillation, are also strongly influenced by any maturation that certain spirit categories may be required undergo (see also OIV-MA-BS-12 and AOAC 945.08).

A common mode of adulteration for some spirit categories is the prohibited addition of neutral spirit (or EAAO) which is often used in product ‘stretching’. The high distillation strength of neutral spirit leads to a concomitant reduction in levels of many of the major volatile congeners. Hence, the illegal dilution of certain spirits with this product may often lead to an observable reduction in key compounds, particularly the higher alcohols.

Major volatile congener concentrations can also provide information on the raw material from which the spirit is made. The ratios of 2-methylbutan-1-ol and 3-methylbutan-1-ol (also known as the amyl alcohols) can differ between different fermentation substrates such as cereals, sugar cane and grape [31]). Trace methanol concentrations are useful as grape fermentations produce more methanol than cereals and cereals produce more methanol than molasses [26].

Once established ranges have been set, major volatile congener profiles are particularly effective for brand authenticity, as often tight ranges can be obtained. Generic authenticity such as spirit category identification and classifications within a spirit category (such as Single Malt Scotch Whisky) are also possible, although wider tolerances will need to be set.

3.1.3. Maturation Related Congeners

A number of spirit category definitions (such as whisky and brandy in the EU), involve maturation in wooden casks as part of the requirement of their production. During maturation, a number of compounds are extracted from the wood into the spirit. The maturation related congeners are quantified by liquid chromatography with detection via ultraviolet spectrophotometry, and spectrofluorimetry. The Union reference method (Annex X) and OIV-MA-BS-16 are aligned, having been validated for whisky, rum, cognac, bourbon and wine spirit.

Maturation can theoretically take place in different types of wood, but oak is by far the predominant material of choice. Extracted wood congeners are present at consistent ratios to each other and at concentrations that increase with maturation time, representing age. Data on the consistency of profile for spirits matured in oak is demonstrated in the published literature [31,57-59]. Ratios and ranges of compounds can be used to determine if a product is authentic by comparison with those observed in genuine products. This will rely heavily on databases generated through analysis of authentic samples and is applicable for both category and brand authentication. The maturation congener profile can also be used to detect where wood extracts or flavourings have been added, often to cover up the absence of a maturation period [60].

3.1.4. Sugars

Sugars may be found in a variety of different spirit types. The individual composition and levels observed will be related to the spirit category and how that spirit is produced. In the EU (Regulation No 110/2008), some spirit categories such as liqueurs require the addition of a minimum concentration of sugars for sweetening; others allow the addition of sweetening sufficient to round off the final taste of the product. Still other categories (e.g. whisky) prevent *any* sweetening by the addition of permitted carbohydrate sources. Trace levels of certain sugars can be naturally present in some spirits as a result of the post-distillation manufacturing procedures of maturation and addition of caramel colouring.

It is common for counterfeiters to add sugars to poorly produced, fraudulent products to try and improve the taste or mimic the natural sweetness of a genuine product. To confirm if sugars are present naturally as opposed to adulteration, the sugar profile of the suspect product should be compared with the known ranges and ratios encountered within the spirit category or brand. For example, analysis of genuine Scotch Whisky products has shown that, where sucrose is present, the level is considerably less than the concentration of glucose and fructose [31].

Liquid chromatography (LC) with Refractive Index (RI) detection is a common technique for sugars analysis. This technique is principally used for quality control of distilled spirits containing high (g/L) levels of sugar content such as liqueurs and pastis. The Union reference method (Annex VIII) and the OIV method (OIV-MA-BS-11) for measurement of total sugars (glucose, fructose, sucrose, maltose and lactose) are aligned. LC-RI is not suitable for identifying sugars adulteration in spirit categories that contain low levels of sugars such as vodka, gin and whisky. A much more effective method is Ion Chromatography (IC), typically used in conjunction with a pulsed electrochemical detector (PAD) [31]. This technique can allow trace levels of individual sugars present naturally in certain spirits to be distinguished from higher levels that can only be achieved by adulteration.

3.1.5. Stable Isotope Ratio Analysis

The applicability of stable isotope ratio measurements to the detection of spirit adulteration will be dependent on the individual spirit and its method of manufacture. Uses include the detection of alcohol from a botanical origin that is not specified in the product definition, and the addition of synthetic alcohol or flavours. The success of these techniques is dependent on the isotope ratios in the natural product being sufficiently different from those in the adulterant.

Two stable isotope ratio analysis methods are currently officially recognised for the application to distilled spirits by the OIV. These are the analysis of the $^{13}C/^{12}C$ ratio of ethanol using stable isotope ratio mass spectrometry, SIRMS, (OIV-MA-BS-22) and the determination of the deuterium distribution of ethanol using nuclear magnetic resonance, SNIF-NMR (OIV-MA-BS-23). Such techniques have also been officially recognised by the EU, OIV and AOAC for determining the origin of sugars in other matrices [37]. Both Carbon-13 SIRMS and deuterium SNIF-NMR have been shown to differentiate between spirit samples containing ethanol from different sources of fermentable sugars. For example, Carbon-13 SIRMS can differentiate between sugars coming from C_4 metabolism plants (e.g. cane, maize, etc.) and C_3 metabolism plants (e.g. wheat, barley, grapes), or Crassulacean acid metabolism plants (e.g. agave) and synthetic ethanol derived from petroleum; deuterium SNIF-NMR can demonstrate similar differentiations [33,61].

In addition to the $^{13}C/^{12}C$ and $^{2}H/^{1}H$ ratios of ethanol, the $^{2}H/^{1}H$ and $^{18}O/^{16}O$ ratios of water have been used for the determination of geographical origin, where this can be definitively linked to the area of production. The OIV have a recognised method for the analysis of $^{18}O/^{16}O$ ratios of water in wines and must, rather than for distilled spirits, in its Compendium of International Methods of Wine and Must Analysis Vol. 1 [62]. This technique and others, such as the $^{18}O/^{16}O$ ratio of ethanol [63] or the $^{2}H/^{1}H$ and $^{18}O/^{16}O$ ratios of bulk spirit [38] may be applied to spirit drink authentication. Whilst not an officially recognised method, Carbon-13 SNIF-NMR has also recently been introduced to allow the practical separation of C4 metabolism plants from some Crassulacean acid metabolism plants, for example ethanol from cane or corn and ethanol from agave [37].

3.1.6. Metals

There are several recognised methods for the analysis of metals in distilled spirits. This reflects both quality control measures and potential concern from external regulators about the levels of such metals in the food chain. (Based on the low risk presented by the sector's products, however, the EU assigns no analytical limits to distilled spirits.) The OIV has four recognised methods for metals analysis by atomic absorption spectroscopy (AAS): calcium, copper, iron and lead (OIV-MA-BS-29 to OIV-MA-BS-32). The AOAC also has methods for distilled spirits using similar methods such as atomic absorption techniques for copper (967.08) and iron (970.12).

Modern laboratories however can employ a variety of techniques to measure metal ions. Typically, they will employ methods that allows the detection of a number of metals within the same analysis, for example inductively coupled plasma optical emission spectrometry (ICP-OES) [64], inductively coupled plasma mass spectrometry (ICP-MS) and IC. Brand owners often have to measure the concentrations of a number of metals to complete certificates of analysis for markets outside the EU. Hence, a database of genuine products can be built up by brand owners, where expected ranges for a number of metals can be set and compared against unknown or suspect samples. This technique is naturally more challenging for generic authentication, particularly where the product is not limited to being bottled in a particular location.

3.1.7. Carbon Dating

The OIV methods include the determination of the ^{14}C content in ethanol by liquid scintillation counting (OIV-MA-BS-24) to help determine between alcohol derived from fossil raw materials (synthetic alcohol) and alcohol made from recently grown plant materials. An alternative technique uses accelerator mass spectrometry (AMS). Both methods may also be used for shorter term dating. This approach can be used to help confirm whether a spirit is consistent with a stated extended maturation age, or whether a bottle claiming to be of historical significance (e.g. pre-1900), and thus potentially meriting a premium price at auction, contains liquid that is consistent with that claim [65].

3.1.8. Additional Officially Recognised Methods

There are a number of additional methods that are listed in the Union reference methods, OIV or AOAC that can be used for spirit drinks authentication. Indeed, any parameter can be employed if there is a natural range for a brand or category and it is likely that this parameter in a fraudulent product may fall outside that range. Those recognised methods listed above reflect commonly used methods for authentication purposes, but the following also deserve attention:

- The determination of pH is a quick measurement that can provide information on the maturation of a sample (OIV-MA-BS-13).
- There are several specific categories of spirits with defined analytical limits in EU legislation to ensure conformity with the definition (see Union reference methods). For example, aniseed flavoured drinks require certain levels of trans-anethole (Union reference methods Annex V; OIV-MA-BS-15).
- The OIV methods contain a method for the measurement of isopropanol (propan-2-ol) (OIV-MA-BS-20). This is not a natural fermentation product for grape based products; it may be added to alcohol during its denaturation. Its presence would thus indicate a fraudulent product.

3.2. Other commonly used methods

3.2.1. UV-Vis Spectroscopy

UV-Vis spectroscopy is a well-established technique for brand authentication both in the laboratory and in the field [28,66]. This method relies on the construction of a large database of individual brands for comparison with suspect samples, and thus tends to be best undertaken by brand owners. In general, UV-Vis spectroscopy works best for the more chemically complex, usually darker spirits such as whisky, brandy and rum. However, the technique can also be applied to clear spirits like vodka. In addition to brand profiling, an abnormal UV-Vis spectrum may indicate the presence of non-permitted compounds [26,67].

3.2.2. Flavourings and Extended Congener Profiles

The EU Spirit Drinks Regulation states that, for a number of spirit categories, the addition of flavourings is prohibited [4, Article 5(1)(c)]. This makes flavouring compounds or flavouring carriers ideal analytical markers for the identification of fraud. Flavouring carriers for spirit drinks are solvents which are used to dilute a flavouring and to facilitate its incorporation and dispersion into the product. To assess whether a flavour compound is naturally present in a spirit product or has

been added, knowledge of the individual spirit category and its production practices is required. Knowledge of common flavourings, flavouring carriers and additives used in the food and drinks industry will also assist in the detection of counterfeit products.

If a suspected flavouring additive is detected, it is often beneficial to identify if the chemical compound is synthetic (man-made). Synthetic compounds are not found in nature hence they will not be naturally occurring as part of standard production practises [68]. Flavouring additives often need only to be present at trace levels to be able to influence the aroma and flavour of a product, therefore sensitive techniques are required. GC-MS and LC-MS are commonly used for the detection of volatile and non-volatile flavouring additive compounds. The analysis of anethole has already been noted as a necessary flavouring constituent of aniseed flavoured products; however its presence in other spirits, such as whisky, would indicate a non-genuine product. Other examples of added flavouring seen in counterfeit products include the synthetic flavouring ethyl vanillin [69] and the flavouring carrier propane-1,2-diol [40].

For certain spirit categories and brands, it will be necessary to extend the range of compound information over and above that provided by the standard major volatile congeners and maturation related congeners methods that are often employed for spirit drink authentication. Such extension will be category specific; for example, the characterisation of gin brands, which uses EAAO (neutral spirit) as its base, is typically free of most major volatile congeners listed, bar methanol. Different entities, such as a range of terpenic compounds, will be more suitable for brand authentication [70]. GC-MS and LC-MS will often be used to increase both the range and sensitivity of compound information obtained from the volatile and non-volatile fractions of a spirit, thus improving differentiation, but also increasing complexity of analysis. MS based techniques (GC, LC or direct injection) can also be used for fingerprinting/non-targeted analysis [71,72]. This will require the creation of large databases gathered from genuine products as well as the use of multivariate statistical analysis. Such techniques have the advantage of identifying when a profile deviates from the expected and may identify the contaminants, or lack of expected congeners, resulting from adulteration/counterfeiting.

3.2.3. Denaturants

Ethanol is produced on a large scale for a variety of industrial uses. To aid in the differentiation of potable alcohol from industrial alcohol and its products, ethanol is “denatured” to make the liquid non-potable and excise duty exempt. The denaturants can act as useful markers for identifying instances where industrial alcohol may have been used in the production of illicit spirits. The chemicals used, and the proportions of denaturants, have traditionally varied by country. In 2008, the EU Commission started a review that has led to a reduction and harmonisation of denaturants in use within Europe. A new “Euro” denaturant formulation is now established, designed to help prevent fraud. This consists of isopropanol, methyl ethyl ketone and denatonium benzoate [73].

Denaturants vary in the ease with which they can be differentiated from constituents of spirit drinks; they will also vary considerably depending on location. Outside the EU, different formulations will be used. In the US, these can be found in Title 27 of the Code of Federal Regulations Part 21 [19]. Methanol has been a commonly used denaturant, which can be potentially fatal contaminant in a fraudulent spirit. In addition to the denaturants themselves, secondary markers resulting from attempts made by counterfeiters to remove denaturants from industrial alcohol may indicate denatured alcohol in a fraudulent spirit [51]. Methods of analysis used to detect denaturants will be targeted to the specific alcohol denaturants. The OIV already have a method for isopropanol; the Customs Laboratory European Network is also due to implement methods to measure the three constituents of the “Euro” denaturant.

3.2.4. Sensory Analysis

Sensory analysis by assessment of aroma (nosing) can be used to assist in spirit drink authentication by identifying suspect samples with atypical aromas. This requires trained and experienced sensory panelists who are familiar with the spirit category or brand's major sensory attributes. Although sensory analysis cannot determine if a product is genuine, it is a useful tool for identifying instances where non-permitted flavourings may have been used in production. It should be noted that sensory analysis is generally considered a subjective technique and confirmatory chemical analysis is always recommended.

4. Overview of methods for authenticity testing

The following table provides a summary of the methods and the authenticity issues they address.

Analytical technique	Indicative data or analyte	Authenticity issue / information
Densitometry	Alcohol Strength (not suitable for spirits with significant levels of dissolved solids e.g. sugars)	Dilution
Distillation and Densitometry	Alcohol Strength	Dilution
GC-FID	Major Volatile Congeners (e.g. higher alcohols, methanol)	Category and brand discrimination
GC-FID	Denaturants (Methanol, isopropanol, methyl ethyl ketone etc.)	Detection of non-potable alcohol
(U)HPLC-UV	Maturation Congeners	Category discrimination, lack of maturation, addition of flavouring
(U)HPLC-RI	Sugars	Brand discrimination
IC-PAD	Sugars	Addition of sweetening
GC-MS	Flavourings, Denaturants, Fingerprinting	Brand discrimination, addition of flavourings, detection of non-potable alcohol
LC-MS	Flavourings, Denaturants, Fingerprinting	Brand discrimination, addition of flavourings, detection of non-potable alcohol
UV-Vis Spectroscopy	Spectroscopic profile	Brand discrimination
^{13}C SIRMS, ^{18}O SIRMS, ^{2}H SNIF-NMR, ^{13}C SNIF-NMR,	Ethanol	Botanical origin of ethanol, detection of synthetic alcohol
^{18}O SIRMS	Water	Category and Brand Discrimination, Adulteration (addition of synthetic alcohol)
^{14}C dating by Liquid Scintillation Counting or Accelerator Mass Spectrometry	Ethanol	Date of production
pH	pH	Lack of maturation
AAS, ICP-OES, ICP-MS, IC	Trace Metals	Brand Discrimination

5. Conclusion

There are considerable financial incentives to create fraudulent spirit drink products. The prices commanded by premium spirit drinks and excise duty combine to offer a lucrative opportunity, especially where excise exempt alcohol can be used in its creation. Excessive taxation is often quoted as being a key contributor to the production and consumption of illicit alcohol. For example, in Indonesia many local people cannot afford to purchase genuine spirit drinks as they are heavily taxed, leaving them to risk drinking unregulated products. In 2018 more than 100 people in Indonesia were killed by one poisoning outbreak [74]. Another potential issue is the ease with which denatured alcohol can enter the potable supply chain. Efforts are being made to address this, such as the changes in European legislation designed to reduce the wide range of denaturants in use, and to focus on formulations which prove hard to remove.

The detection of counterfeit spirit drinks can be challenging. Spirit drinks are characterized by two major constituents: ethanol and water. The other compounds present, which provide differentiation in terms of flavour and identity, are generally present at low levels. Many compounds, such as proteins and DNA that are associated with the raw materials (cereals, grapes etc.), are removed during the distillation step. As a result, techniques used to identify counterfeit spirit drinks are typically based on profiles of flavour and other constituents present at trace levels (ppm to ppb), such as the measurement of major volatile or maturation related congeners. Certain properties of the whole spirit, such as pH, UV spectrum and alcohol strength can however prove useful in identifying frauds.

Looking to the future, there are several trends apparent in spirit drink authentication. The first is the drive for portability in analytical measurements, allowing rapid evaluations to take place at key points in the supply chain, for example at point of sale. Portable pH and conductivity meters can already be employed [75]. The use of portable UV-Vis for brand authentication is common, but it can also be used for detection of specific compounds such as sugars [76]. Raman and NIR spectroscopy are also being explored for their potential [77-79]; the opportunity of analysis through spirit drinks bottles using such techniques is an attractive option for fraud detection.

Another trend is the increasing availability of more conventional laboratory techniques in machines with a smaller footprint. These offer the potential for the both the quantitative profiling of key marker compounds (of either genuine or counterfeit products) where chromatography is involved [80] or a rapid assessment of authenticity based on a chemometric model of a particular brand or category [72]. Finally, advances in laboratory authentication of spirit drinks will most likely result in more detailed analysis (increased number of compounds and/or increased sensitivity) becoming more routine and more rapid. The application of NMR as a routine technique for both targeted and untargeted analysis of spirit drinks is one possibility [81].

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