FOODINTEGRITY
Ensuring the Integrity of the European food chain

613688: Collaborative Project
Seventh Framework Programme
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Deliverable: D5.3 Overview

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The project has received funding from the European Union’s Seventh Framework Programme for research, technological development and demonstration under grant agreement No. 613688.
Deliverable 5.3

Overview

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1 Deliverable 5.3 – Contents

The aim of Deliverable 5.3 of Work Package 5 was to achieve a legacy platform for dissemination of guidance on spirit drinks analysis methods for safety and authentication assurance. At a base level this was the provision of a centralised resource where outcomes of the Deliverables from Work Package 5 of the FoodIntegrity project could be stored when developed. However, it was recognised that a legacy platform should be more than a collection of documents from the project, but should also involve the establishment of a network of people interested in spirit drinks authentication, and a means of accessing information relating to spirit drink authentication methods that can be used to protect European spirit drink products in laboratories worldwide.

The contents of this Deliverable 5.3 report, detail the varied activities and content developed in order to meet the overall objectives as outlined above. The work has been split into 5 Sections, both for clarity and the option to disseminate the individual parts separately. These sections are as follows:

- **Part I - The Spirit Drinks Work Package Network, Website and Promotional Activities**
- **Part II - The FoodIntegrity Spirit Drinks Authentication Seminar**
- **Part III - The Survey of Analytical Methods Used for the Quality Assurance of Spirit Drinks**
- **Part IV - A spiritsEUROPE Report on its Work Addressing Illicit Alcohol Production and its Dissemination of Spirit Drink Work Package Activities**
- **Part V - A Bibliography of Spirit Drinks Authentication Methods**

2 Deliverable 5.3 – Objectives

Within this Deliverable there were a number of individual tasks.

- 5.3.1 Undertake cross-European expert study of spirit analysis methods and authentication protocols, and establish recommended guidance for assessing safety and authenticity.

  The process of conducting a cross-European expert study of spirit analysis method and authentication protocols, was one of the Milestones of the FoodIntegrity project (Milestone 17) and is reported in Part III of this Deliverable.

- 5.3.2 Establish legacy platform for dissemination of guidance (e.g. stakeholder website).

  This was designed to include a centralised document store, a network of interested parties and other resources deemed useful to those interested in spirit drinks authentication.
3 Deliverable 5.3 – Discussion

The outcomes of this Deliverable were designed to provide a useful resource for people engaged in protecting European spirit drinks throughout the world. A large proportion of the content, it was envisaged, would relate to analytical methodology and developed technologies, but it was recognised that it should address the needs of all those engaged in this area, whether analyst, brand manager, enforcer or regulator.

Part of the work undertaken within this Deliverable was the establishment of a network of parties representing the different types of stakeholders involved in spirit drinks authentication - the Spirit Drinks Authenticity Network. Traditional routes of access to relevant stakeholders via the knowledge and expertise of Partners 34 (The Scotch Whisky Research Institute) and 37 (spiritsEUROPE) and their members were explored. In addition, contacts were identified via a number of key presentation events. Information about the network, and events at which the objectives of the FoodIntegrity Work Package on spirit drinks were presented, are provided in Part I of this Deliverable. Information is also provided on the development of a centralised resource for members of the FoodIntegrity Spirit Drinks Authenticity Network.

Part II reports on the FoodIntegrity Spirit Drinks Authenticity Seminar held in May 2015. This was a strategic event designed to promote the work of the FoodIntegrity Spirit Drinks Work Package, to collect together a number of the Spirit Drinks Authenticity Network participants for a chance to discuss the work being undertaken, and importantly to confirm with potential end-users that the types of technologies that were being considered within Deliverables 5.1 and 5.2 were of relevance.

Part III specifically looks at Milestone 17 (Task 5.3.1) directed at undertaking a cross-European expert study of spirit analysis methods and authentication protocols, and establishing recommended guidance for assessing safety and authenticity. A summary and analysis of the responses provided as part of this survey are given, as well as a description of how this information is being used to populate another Deliverable of the FoodIntegrity project – Work Package 2 – KnowledgeBase.

One of the partners in the Work Package on spirit drinks, spiritsEUROPE, has been key to the development of Deliverable 5.3. spiritsEUROPE represents the interests of the spirits sector in 31 national associations as well as of the 8 leading multinational spirit companies, and as such offers a unique means of communicating with spirit drinks stakeholders within Europe. Part IV highlights some of the activities within which spiritsEUROPE has been engaged with the express aim of advancing the aims and objectives of the Spirit Drinks Work Package.

Lastly, it was decided that a bibliography of spirit drink authentication papers would be a useful resource for those involved in research and analysis in this area. As such, a database has been developed, which will act as a primer for expansion by contacts within the network. In the first instance, it was decided that a minimum of 50 papers would be collected, prior to opening up submission to third parties. The current database of papers is listed in Part V.
4 Deliverable 5.3 – Conclusions

Deliverable 5.3 has provided a number of resources to those with interests in Spirit Drinks authentication and quality assurance. These are:

- A survey and analysis of the analytical methods employed in the profiling, authentication and quality assurance of spirit drinks.
- Population of the KnowledgeBase of analytical methods constructed within Work Package 2 with key methods for spirit drinks analysis. These will be supplemented with more experimental, research based methods in due course.
- A centralised resource on the FoodIntegrity website for storage of outputs from the FoodIntegrity project’s Spirit Drinks Work Package.
- A developing network of stakeholder contacts.
- A developing bibliography of spirit drink authentication methods and research.

One of the challenges will be in ensuring the outputs of this deliverable will be maintained after the completion of the FoodIntegrity project. This will be explored within the FoodIntegrity consortium, along with other legacy aspects, such as the larger FoodIntegrity website and the WP2 KnowledgeBase. Consideration needs to be given to the location of the centralised resource and whether the FoodIntegrity website itself will be continued after project close. Maintaining the network, bibliography, news and documents stored in this central location (as well as maintenance of the spirit drinks analytical methods within the KnowledgeBase) should not require large amounts of time, but the ongoing resource required from Partners 34 and 37 will need to be evaluated to ensure that the legacy can be maintained.
The project has received funding from the European Union’s Seventh Framework Programme for research, technological development and demonstration under grant agreement No. 613688.
Deliverable: D5.3 Part I

Title: Platform for Dissemination of Guidance –
The Spirit Drinks Work Package Network, Website and Promotional Activities

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1 Description of Deliverable

The aim of Deliverable 5.3 of Work Package 5 was to achieve a legacy platform for dissemination of guidance on spirit analysis methods for safety and authentication assurance. This includes: a network of stakeholders interested in the authentication and quality assurance of spirit drinks, the promotion of the aims and objectives of the FoodIntegrity Spirit Drinks Work Package at a number of industry/food authenticity/scientific events, a means of collating and sharing information on research developed on spirit drinks analysis both within and external to the FoodIntegrity Spirit Drinks Work Package, a survey and collation of the methods currently in use for the analysis of spirit drinks, and the organisation of a networking event specifically for the Spirit Drinks Work Package. This part of Deliverable 5.3 provides an overview to the work undertaken in these areas.

2 Achievement of the Deliverable – Spirit Drinks Authenticity Network

Four different types of stakeholders were targeted for participation within the Spirit Drinks Authenticity Network: analysts, brand managers, enforcers and regulators. In addition, the network also includes a number of technology providers, who have been involved in discussions relating to the analytical objectives in Deliverables 5.1 and 5.2.

Over the course of the three-year Deliverable, the number of individuals involved in the network has slowly increased. As a result of increased contact with spirit drink industry representatives, technology providers and the wider scientific community there are now over 190 participants in the Spirit Drinks Authenticity Network.

Contact with the Network has involved: forwarding information relating to events and conferences that is felt that are relevant, such as the Worldwide Distilled Spirits Conference, RAFA 2015 and the FoodIntegrity Spirit Drinks Authenticity Seminar; requesting suggestions for work being undertaken within the Work Package; soliciting responses to the Survey on Analytical Methodology used for spirit drinks; providing access to the secure area on the FERA website where information relating to the FoodIntegrity project is being stored. The network will also be used to identify how the objectives of Deliverable 5.4 can be met, by using key members to discuss options for training, education and validation in the authentication of spirit drinks, as well as the potential for sharing of information between industry and enforcers.

Part of the development of the Spirit Drinks Authenticity Network has been enabled by the promotion of the Spirit Drinks Work Package. A non-exhaustive list of dissemination events is included in Appendix I. Several of these dissemination events made use of the network of European spirit drink trade associations and companies that are members of spiritsEUROPE. (spiritsEUROPE acts as the European representative body for producers of spirit drinks with a membership comprising of 31 national associations representing the industry in 24 countries as well as a group of leading spirits producing companies.)

spiritsEUROPE were involved in promoting the aims and objectives of Deliverable 5.3 by updating the spiritsEUROPE public website to include a dedicated page on the FoodIntegrity project. On the spiritsEUROPE (members only) extranet a dedicated ‘Issue Page’ has been created which summarises the main activities since the start of the project and contains links to all the relevant documents. In
addition, the work on the FoodIntegrity project was included in two monthly newsletters (e.g. July 2015) that were circulated to over 2,000 traders and officials.

spiritsEUROPE have also been heavily involved in understanding and tackling issues relating to illicit alcohol. This includes making requests for official involvement in protocols for measuring its scale, and involvement in programmes of work with organisations such as the International Alliance for Responsible Drinking (IARD) which has, among other things, developed methodology to measure the scale of illicit alcohol.

More detail on the networking activities and achievements of spiritsEUROPE within the Spirit Drinks Work Package can be found in Deliverable 5.3 Part IV.

Figure 1: Screenshot of spiritsEUROPE FoodIntegrity (members only) issue page
3 Achievement of the Deliverable – Spirit Drinks Work Package Website

A section of the FoodIntegrity website has been devoted to disseminating the outcomes of the Spirit Drinks Work Package. In order to access this, users must be signed up to the FoodIntegrity website and have been given the appropriate access permission to view non-public information. There is also a permission available in the administration of the FoodIntegrity website that can limit access to users within a particular Work Package group. This is currently being used to store administrative documents only accessible to Work Package partners, but it is planned to transfer that privilege to members of the Spirit Drinks Authenticity Network. The potential for unrestricted access to outputs from the Spirit Drinks Work Package has been raised as a concern by industry representatives, so a clearly defined group of users who can access such information would be beneficial.

The following screenshots highlight the organisation and content of the Spirit Drinks Work Package website:

Figure 2: Spirit Drinks FoodIntegrity Area – General Information Links provided to the Spirit Drinks Authentication Seminar, an Overview Poster for the Project and the Twitter feed.
Figure 3: Examples of Articles Referenced on the Spirit Drinks Work Package Twitter Feed
Spirit Drink Analysis Methods

Caveat

This site is not responsible for the accuracy of any externally hosted content.

Links to recently published spirit drink laboratory analysis methods

The presence of high levels of phthalates in European Spirit Drinks have been linked with counterfeit products.

Information on published methods for phthalates analysis in spirit drinks are presented below.

LC-MS/MS

Ring test on Phthalates Analysis

Links to recently published spirit drink portable analysis methods

Rigaku have looked at the detection of methanol in methanol/ethanol mixtures. The scope of the study was to determine at what level methanol can be detected and what the characteristics are of the methanol-ethanol mixtures.

Analysis of single malt Scotch whisky using Raman spectroscopy

Rigaku - detection of methanol in methanol/ethanol mixtures

Figure 4: An Area for Uploading Links (and content where copyright permits) to Sprit Drinks Analysis Methods – a link to the Work Package 2 Knowledge Base will be placed here when it is ready to be accessed.
Application Work Undertaken with Anature - Laboratory Analysis

Anature

One of the deliverables of Work Package 5 looks at understanding what advanced analytical laboratory techniques can accomplish in terms of providing increased authentication capabilities to the spirit drinks industry.

Discussions with Anature have tackled a number of industry requirements for spirit authentication:

a) improving capabilities in assigning maturation age to a brown spirit.
b) identifying common flavourings and additives present in counterfeit spirits.
c) identifying indirect (and legal) additives from previous cask use, which should not be confused with illegal flavouring addition.
d) improving speed, sensitivity and level of information obtained from spirit drink analyses.

Documents AS148 and AS149 (see right) provide information on the application to spirits of a Multi Volatile Method (MVM), developed by GERSTEL to analyse volatile compounds in aqueous matrix by Dynamic Headspace (DHS)-GC/MS. The method is designed to extract a wider range of compounds, in particular those with high volatility which can be hard to extract.

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**Figure 5**: An Example Page Highlighting a Work Package 5 Output.
4  Achievement of the Deliverable – Spirit Drinks Method of Analysis Survey

A survey was constructed and issued to members of the Spirit Drinks Authenticity Network to identify the methods used within the analysis community for the authentication of spirit drinks. This established a recommended set of analyses for spirit drink authenticity and quality assurance, with supporting documentation including: the understanding of inconsistencies and gaps in the methods; collation of links to publicly available reference methods; papers and useful data for the purposes of interpretation.

The principal objective of Work Package 2, which was the development of a searchable KnowledgeBase of food authenticity methods, overlapped with the Deliverable 5.3 aim of providing guidance and information on the analysis methods used for the authentication of spirit drinks. Consequently, it was decided to use the Work Package 2 KnowledgeBase for the storage of information on spirit drink analytical methods obtained from the survey. spiritsEUROPE and the Scotch Whisky Research Institute became partners within Work Package 2 and, as part of the KnowledgeBase development process, 15 example Spirit Drink Analysis methods were uploaded. Others will be added now that the construction of the KnowledgeBase has reached a sufficient level of maturity, based on information provided from the survey and new information from the scientific literature as it is generated. It is planned for the Spirit Drinks Authenticity website to direct network contacts to the Work Package 2 KnowledgeBase to access this information.

The Spirit Drinks Analysis Survey was principally filled in by participants in the EU. However, recent laboratories targeted by the Work Package to provide information have been outside the EU, in recognition that European spirit drinks are exported globally.

Figure 6. Screenshots from Spirit Drinks Analysis Survey
A full report has been created based on all the responses received from the survey and this is provided in Deliverable 5.3 Part III. In summary, the report has identified:

- A consensus on the principal methods employed for the purposes of spirit drink authentication and quality control analysis.
- Additional, occasionally used, methods in certain spirit drink sectors.
- Gaps in the availability of reference methods for spirit drinks.
- Suggested updates to currently available reference methods.
- Suggested means of addressing reference method deficiencies.
5 Achievement of the Deliverable – Organisation of a Spirit Drinks Work Package Networking Event

To bring different parties from the Spirit Drinks Authenticity Network together, a specific Spirit Drinks Work Package event was held on May 26th 2015. The Spirit Drinks Authentication Seminar provided the chance for stakeholders to feedback thoughts on the aims, objectives and progress of the Spirit Drinks Work Package and a report on this event is provided in Deliverable 5.2, Part II.

Based on the success of the meeting, and its value in identifying the needs of the various parties involved in spirit drinks authentication and quality assurance, a second Seminar is planned, which form part of Deliverable 5.5, a new Deliverable created during the course of the project.
Appendix I – Spirit Drinks Work Package Dissemination Events

A prerequisite for undertaking a cross-European expert study of spirit drink analysis methods and authentication protocols has been to identify interested parties and involve them in the Spirit Drinks Authenticity Network. A number of additional contacts have been made via publicity of the Work Package at industry/scientific/food authenticity events and via one to one communications between partners in the Work Package and identified stakeholders. Such promotional opportunities have also resulted in contacts with technology providers and development of sub-projects within Tasks 5.1 and 5.2. Examples of such publicity over the course of Deliverable 5.3 includes:

2014

- February 19th 2014: Discussion at GP.TS (Technical-Scientific Committee of spiritsEUROPE) meeting on the legal limits of parameters for the authentication of spirit drinks categories.
- April 1st-2nd 2014: Meeting with Irish Distillers (Pernod Ricard) to discuss FoodIntegrity Work Package 5 and their interest in the project.
- June 19th 2014: SWRI Product Protection Liaison Group, attended by Spirit Company Members of the SWRI where the FoodIntegrity Work Package 5 project was promoted and discussed
- August 5th 2014: Meeting of international experts on illicit alcohol and counterfeit hosted by the International Center for Alcohol Policies.
- September 8th 2014: Discussion at GP.TS (Technical-Scientific Committee of spiritsEUROPE) meeting on the lab test used for the authentication of spirit drinks categories or brands.
- September 8th-11th: Oral Presentation and Poster at the 5th Worldwide Distilled Spirits Conference 2014. This was published in the 5th Worldwide Distilled Spirits Conference proceedings in 2015.
- September 30th 2014: International Federation of Spirit Producers Scientists' Meeting on authentication technologies. Discussion of currently available portable authentication technologies. FoodIntegrity project was heavily promoted to spirit drink company representatives in attendance. Focus on in-field devices and their use. Discussion also regarding training and validation of third party laboratories.
- October 22nd 2014: Attendance at FISCALIS PROJECT GROUP 013/INDUSTRY meeting on denaturants relating to cosmetics, perfumes, personal hygiene issues. Contacts made in relation to alcohol denaturants and their analysis.
- October 23rd 2014: Meeting with Marjana Martinic of the International Center for Alcohol Policies. The Center is undertaking work looking at the types and extent of illicit alcohol trade and methods for quantifying such. They have a large database of accessible information on illicit alcohol.
- November 5th 2014: Presentation by Ian Goodall at the Campden BRI “Food Fraud: Advances in Combating Food and Beverage Crime” meeting, within LabInnovations 2014. Included overview to FoodIntegrity project and the Spirit Drinks Work Package.
- December 10th 2014: West Indies Rum and Spirit Producers Association Technical Seminar – presentation given by Shona Glancy (of the Scotch Whisky Research Institute) on various topics including the authentication of spirit drinks. FoodIntegrity Work Package publicised.
- December 18th 2014: Meeting with Iain Macleod and Deborah Prunty at Diageo Brand Technical Centre to discuss the company’s recent work in spirit drink authentication and their interest in the FoodIntegrity Spirit Drinks Work Package.
2015

- February 10th 2015: Discussion of FoodIntegrity project, Work Package 5 and recommended methods of analysis for spirit drinks at a meeting of the spiritsEUROPE technical-scientific GP.TS group in Cognac (with attendance of Ersilia Moliterno, Directorate General for Agriculture and Rural Development, Wine and Spirits Unit).
- February 16th 2015: spiritsEUROPE Meeting with DG Taxud to discuss denaturants and evidence of 'screenwash' being converted ('cleaned up') to make potable spirit.
- March 26th 2015: Presentation on “Rapid Methods in the Spirit Drinks Sector” given at 2nd FoodIntegrity Conference “Assuring the integrity of the food chain: food authenticity research priorities and funding opportunities”.
- May 13th 2015: Meeting with representatives from Scottish Enterprise which included a discussion of the FoodIntegrity project.
- May 26th 2015: Spirit Drinks Authenticity Seminar hosted at the Scotch Whisky Research Institute, with a number of representatives from the Spirit Drinks industry, technology providers and analytical laboratories.
- July 17th 2015: Article in spiritsEUROPE newsletter (with a circulation over 2,000 officials and traders).
- August 13th, October 29th 2015: Update on FoodIntegrity project given at Scientific Committee of Scotch Whisky Association.
- September 1st 2015: Meeting with representatives from Food Standards Scotland Food Crime Unit.
- September 9th-10th 2015: Participation at UK Science and Innovation Meeting in Lodi. Presentations given on “Rapid Methods in the Spirit Drinks Sector”.
- September 17th 2015: Presentation at Campden BRI Member Interest Group (MIG) highlighting the aims, objectives and work of the FoodIntegrity project and Spirit Drinks Work Package – “The Authentication of Spirit Drinks: Current Solutions, New Technologies”.
- October 9th 2015: Presentation at FISCALIS Meeting (organised by DG Taxud) in Belfast relating to Denaturant Project. Objectives and work of the FoodIntegrity project and Spirit Drinks Work Package presented.
- October 15th 2015: Presentation on the FoodIntegrity project and Spirit Drinks Work Package made to the Scotch Whisky Research Institute’s Product Protection Technical Liaison Group made up of spirit drink industry representatives.
- October 17th 2015 and November 5th 2015: spiritsEUROPE meeting with DG Taxud - FoodIntegrity project issues raised.
- October 27th 2015: FoodIntegrity project and Spirit Drinks Work Package discussed at spiritsEUROPE technical-scientific project group, attended by technical experts within the spirit drinks industry.
- October 28th 2015: The FoodIntegrity project and Spirit Drinks Work Package was a key component of the Institute of Brewing and Distilling’s Authentication Day with approximately 35 industry representatives and students in attendance.
- November 4th-5th 2015: Presentation and interactive display relating to Spirit Drinks Work Package at the FoodIntegrity Open Days at RAFA 2015. Certain aspects of project also presented in various presentations (Jana Hajšlová, James Donarski).
- November 19th 2015: Dissemination of SWRI newsletter to approximately 450 spirit drink industry personnel with articles on different aspects of the FoodIntegrity project.
- November 27th 2015: spiritsEUROPE meeting with JRC official at which issues relating to illicit alcohol were raised.
- December 4th 2015 and April 27th 2016: Presentation on FoodIntegrity project and Spirit Drinks Work Package at the Scotch Whisky Research Institute’s Research Management Committee attended by a number of senior technical representatives of the spirit drinks industry.
2016

- January 22nd 2016: Promotion of FoodIntegrity project at the Wine, Spirits and Beer Incident Roundtable attended by approximately 20 industry representatives.
- February 18th 2016: spiritsEUROPE attended meeting of the High Level Forum for a Better Functioning Food Supply Chain. Raised concerns regarding illicit alcohol. In subsequent submission regarding possible priorities, referenced work on FoodIntegrity project.
- February 18th 2016: Update on FoodIntegrity project given at Scientific Committee of Scotch Whisky Association.
- February 23rd 2016: SWA/SWRI meeting with Laura Pollard - HMRC, Deputy Director, Alcohol, Tobacco, Holding and Movements and Amy Burgess - HMRC, Alcohol Policy Team Leader, at which FoodIntegrity project was raised.
- March 1st 2016: Meeting involving SWRI, DG Taxud, JRC and spiritsEUROPE to discuss various aspects of illicit and denatured alcohol and the FoodIntegrity project.
- March 24th 2016: Scotland Food and Drink Authenticity Meeting - FoodIntegrity project briefly mentioned as example of work being undertaken to protect Scottish Food and Drink Authenticity (whisky, gin and vodka).
- March 17th 2016: Presentation on the FoodIntegrity project and Spirit Drinks Work Package made to the Scotch Whisky Research Institute’s Product Protection Technical Liaison Group made up of spirit drinks industry representatives.
- March 24th 2016: Lecture to Heriot Watt University Brewing and Distilling (ICBD) final year students 'Analytical Techniques for Scotch Whisky Authentication’ at which FoodIntegrity project was highlighted.
- April 6th-7th 2016: Presentations at the 3rd FoodIntegrity Conference “Fighting food fraud”. Two presentations given: “Rapid Methods Perspectives (focus on the Spirit Drinks Sector)” and “Expanding Analytical Capabilities within Spirit Drinks Authentication”.
- April 20th 2016: Update on FoodIntegrity project given to around 30 industry representatives at spiritsEUROPE’s fiscal and economic committee.
- April 25th 2016: SWRI Skype conference with 4 representatives from the Consejo Regulador del Tequila A. C. The FoodIntegrity project was a principal agenda point.
- April 27th 2016: FoodIntegrity project and Spirit Drinks Work Package discussed at spiritsEUROPE technical-scientific project group, attended by technical experts within the spirit drinks industry.
- April 27th 2016: Presentation on FoodIntegrity project and Spirit Drinks Work Package at the Scotch Whisky Research Institute’s Research Management Committee attended by a number of senior technical representatives of the spirit drinks industry.
- April 29th 2016: After the April 2016 OECD conference on illicit trade, follow up by spiritsEUROPE alerted OECD Task Force leaders to its involvement in the FoodIntegrity project and Spirit Drinks Work Package.
- April 29th 2016: Presentation at “RSC Advances in the Chemical Analysis of Food” conference organised by RSC Food Group and RSC Separation Science Group entitled “The Authentication of Spirit Drinks: Current Solutions, New Technologies” which included references to FoodIntegrity project. Approximately 80 attendees from the scientific community.
- May 12th 2016: SWRI meeting with 3 representatives from Food Standards Scotland. The FoodIntegrity project was a principal agenda point.
- May 27th 2016: spiritsEUROPE attended Centre for European Policy Studies conference on illicit trade in Brussels and highlighted FoodIntegrity project work.
- June 2016: spiritsEUROPE liaison with the 'Coalition Against Illicit Trade' to determine extent to which it might be useful in the fight against illicit alcohol and the FoodIntegrity project work.
- June 17th 2016: Dissemination of SWRI newsletter to approximately 450 spirit drink industry personnel with articles on different aspects of the FoodIntegrity project.
• June 23rd-24th 2016: Attendance by SWRI at 6th Seminar for European Customs Chemists. Short presentation on Scotch Whisky Authentication and FoodIntegrity project given on 24th to approximately 40 customs chemists.
• June 28th 2016: spiritsEUROPE made presentation in 2 webinars on illicit alcohol organised by International Alliance for Responsible Drinking. The FoodIntegrity project was used as example of work being undertaken in EU to counter illicit trade.
• July 12th 2016: Article in spiritsEUROPE newsletter on FoodIntegrity project and Spirit Drinks Work Package (with a circulation over 2,000 officials and traders).
• October 19th and 24th: Discussions with DG AGRI regarding a possible presentation at a Committee for Spirit Drinks meeting.
• October 20th 2016: FoodIntegrity Spirit Drinks Work Package updates given to representatives from European spirit industry at spiritEUROPE’s GP.TS Meeting - Approx. 10 attendees
• November 7th-9th 2016: 11th Rapid Methods Europe Conference 2016. Presentation on “Developing rapid analysis methods in the spirits drink sector” given, based on work within FoodIntegrity project’s Spirit Drinks Work Package.
• November 21st-22nd: spiritsEUROPE presentation at The Royal United Services Institute (RUSI) conference on illicit trade. The FoodIntegrity project and Spirit Drinks Work Package referred to as options for tackling illicit alcohol.
• November 23rd: The Scotch Whisky Research Institute main governing board meeting was given a specific presentation on the outputs of the FoodIntegrity Spirit Drinks Work Package. The SWRI governing board are made up of senior managers and decision makers within the Scotch Whisky industry.
• December 14th: Presentation to Copa-Cogeca tax committee on 92/83 review and illicit alcohol. The FoodIntegrity project and Spirit Drinks Work Package referred to as options for tackling illicit alcohol.
FOODINTEGRITY
Ensuring the Integrity of the European food chain

613688: Collaborative Project

Seventh Framework Programme
KBBE.2013.2.4-01: Assuring quality and authenticity in the food chain

Deliverable: D5.3 Part II
Title: Platform for Dissemination of Guidance –
FoodIntegrity Spirit Drinks Authentication Seminar

Author(s): Ian Goodall
Beneficiary(s): The Scotch Whisky Research Institute (Partner 34 in the FoodIntegrity Consortium)

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## Deliverable: D5.3 Part II

**Title:** Platform for Dissemination of Guidance – FoodIntegrity Spirit Drinks Authentication Seminar

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### Appendix

FoodIntegrity Spirit Drinks Authentication Seminar – Email Flyer ........................................ 12
1  Description of Deliverable

The aim of Deliverable 5.3 of Work Package 5 was to achieve a legacy platform for dissemination of guidance on spirit analysis methods for safety and authentication assurance.

This Deliverable also addressed Milestone 15 which was to make a decision on the technologies to target for testing and developing in-field safety screens and authenticity analysis.

This Deliverable report describes the FoodIntegrity Spirit Drinks Authentication Seminar held at the Scotch Whisky Research Institute on May 26th 2015. The event was organised to tackle a number of objectives within the Deliverable:

- Provide a forum to gather participants signed up to the Spirit Drinks Authenticity Network together, to discuss the Work Package and its objectives.
- Consult on initial work undertaken as part of the Deliverables 5.1 and 5.2 on rapid, in-field and laboratory analysis for spirit drink authenticity and quality assurance, and thus provide an output for Milestone 15.
- Provide opportunities for equipment demonstrations from technology manufacturers.

2  Achievement of the Deliverable

Thirty-eight people attended the FoodIntegrity Spirit Drinks Authentication Seminar from across Europe, 12 from technology providers and 26 representing a variety of stakeholders in spirit drinks authentication including analysts (industry and enforcement agencies), researchers and brand managers. 6 presentations were provided by the technology providers on technologies being considered for spirit drink authentication and quality assurance. A session following the presentations posed a number of questions to the spirit drinks authentication stakeholders. These related to the presentations given and the objectives of the Spirit Drinks Work Package. These were collated, anonymised and have helped form the basis of future work; they are summarised in Section 5.

3  Seminar Report - Attendees and Presentations

The flyer for the FoodIntegrity Spirit Drinks Authentication Seminar can be found in the Appendix and at the link provided below.

The list of attendees to the seminar can be found in Table 1 below.
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All the presentations given by the Technology Providers at the event can be found at the following addresses, for download, by copying links into a browser. These presentations summarised views and work undertaken by the technology providers at the time, and further developments are described in Deliverables 5.1 and 5.2 as appropriate.

Seminar Flyer
https://secure.fera.defra.gov.uk/foodintegrity/secure/downloadFile.cfm?id=304

SWRI Presentation 1 - Welcome and 2 - Developments in Spirit Drink Analysis
https://secure.fera.defra.gov.uk/foodintegrity/secure/downloadFile.cfm?id=305

Ocean Optics Presentation 3 - Tackling the Challenges of the Spirit Drinks Industry
https://secure.fera.defra.gov.uk/foodintegrity/secure/downloadFile.cfm?id=306

Microsaic Presentation 4 - Bringing Mass Spectrometry Down to Size
https://secure.fera.defra.gov.uk/foodintegrity/secure/downloadFile.cfm?id=307

VUV Analytics Presentation 5 - A new GC Detector Technology
https://secure.fera.defra.gov.uk/foodintegrity/secure/downloadFile.cfm?id=308

Distilled Solutions Presentation 6 - DETECT System for Spectroscopic Analysis of Whisky
https://secure.fera.defra.gov.uk/foodintegrity/secure/downloadFile.cfm?id=309

Perkin Elmer (Torion) Presentation 7 - Field Portable GC/MS
https://secure.fera.defra.gov.uk/foodintegrity/secure/downloadFile.cfm?id=310

Anatune Presentation 8 - Automated Laboratory Based Techniques for Authenticity Analysis
https://secure.fera.defra.gov.uk/foodintegrity/secure/downloadFile.cfm?id=311

SWRI Presentation 9 - Discussion - Next Steps in the Spirit Drinks Work Package
https://secure.fera.defra.gov.uk/foodintegrity/secure/downloadFile.cfm?id=312
4 Seminar Report - Questions asked of Technologies Presented/Demonstrated

A number of questions were posed to the technology providers after their presentations. Whilst these helped inform the technology providers as to future developments, they have not been deemed to be useful for this report and are not recorded here.

5 Seminar Report - Closed Discussion Session (without Technology Providers)

Following the presentations and demonstrations given by the Technology Providers, a discussion on the technologies seen and the objectives of the FoodIntegrity Spirit Drinks Work Package was managed. This focussed on a series of questions directed at the attendees (see Presentation 9). The following points were noted from the discussions, loosely grouped under appropriate questions.

Is our current strategy of engaging with technology providers correct?

- Whilst the authenticity seminar and the presentations were well received, it was noted by one of the industry attendees that the presentations were slightly too focused on selling the product and not particularly well focused on the spirit industry applications. As a result, it was recommended that care is taken within the project to ensure technology providers are fully considering the application needs of the industry.
  In response to the above point, it was noted that the SWRI did help and guide the presenters where needed/requested, however, the presentation section was designed for the technology providers to present themselves and their technologies as they best saw fit.
- Similarly, it was commented that technology providers often underestimate the level of sensitivity required to authenticate spirits, which are principally ethanol and water, with the key markers for authenticity being present at low (ppm to ppb) levels. Portable devices are commonly looking at analytes with much higher concentrations.
- A couple of the presentations noted the development of portable devices in relation to the needs to the defence industry. A suggestion was made that, since the defence industry seems to have a funds and information available to develop similar technologies, a related trade body could be approached to find out what new techniques are being evaluated, although it would be uncertain whether open dialogue would be possible.
- Finally, a note of caution was raised about the range of devices being considered, and how these could all be evaluated within the course of the project. In response to this point, it was suggested that sample sets given to the technology providers to test their products need to more specific and focussed on the areas where they are most likely to achieve success – e.g. either they are looking at authentication of a brand or a spirit category, not both.

Suggested Actions:

- Explore options with the defence industry. The Defence Science and Technology Laboratory seems like an obvious place to start.
Technology providers need to be given a CLEAR BRIEF. This was already being followed by the Spirit Drinks Work Package, but it was recognised that in addition to a clear outline of the Project and its aims and objectives, the Work Package must be quick to focus on the areas in which a technology may be useful after an initial proof of concept.

Are there any types of potential solution that we should rule out?

- The presentations were split into the consideration of rapid, in-field devices (Ocean Optics, Distilled Solutions Limited), more traditional laboratory devices that were becoming more portable (Microsaic and Torion) and laboratory based technologies (VUV and Anatune). However, the attendees were keen not to rule out any of the solutions, stating that the project should try and encourage solution providers at all levels (portable, deployable and laboratory based) to progress authentication applications for the spirit drinks industry.

Portable solutions were seen as important, offering a quick, initial screen in the marketplace; they should not require too much knowledge to operate or time to undertake. Laboratory tests were seen as being required for authoritative confirmation of authentication. One of the industry attendees was keen that the industry continues to engage with those providers working at miniaturising equipment, even if such technology is not at a truly portable level. It was felt that the industry needed to encourage such work, highlighting the potential applications for spirit analysis and authentication to which they can be put.

- On the subject of portable methods, it was noted that these should be simple to use, fast, easy to transport (even the Pelicase design used for the Spirit Sampler solution could often present difficulties at customs) and cheap to run. Associated cost of equipment also needs to be considered, to allow more to be deployed into the marketplace. For example, it was noted that the Ocean Optics Spirit Sampler could be considered too expensive to move from market to market and it couldn’t be expected that all countries have their own piece of kit.

Suggested Actions:

- Engagement with technology suppliers should continue at all levels – portable, semi-portable and laboratory based. Each solution was seen as offering a potential benefit to the industry in terms of analysing and authenticating its products.

- For portable devices, a focus on agile, lightweight and cost effective solutions was required, which would provide quick answers that could be confirmed in the laboratory.

Should we be engaging with long term solution providers?

- Responses provided to this question developed the themes raised in the previous question. The benefits of long terms engagements and collaborations with solution providers would help develop devices already in use. As noted previously, even the Ocean Optics Pelicase is
not as portable as might be liked, so continued engagement with the supplier was necessary to help develop improvements. Such engagements should also focus attention on improving technologies that showed potential but perhaps needed miniaturising or being made simpler to operate.

- It was suggested that contacts are made with the providers of Raman based solutions in use at airports to look through bottles for illegal substances.

**Suggested Actions:**

- Keep engaging with solution providers looking to provide portable, user friendly equipment, even if the current options fall short of true portability/ease of use.

- Keep engaging with current solution providers to continue to develop applications already in use.

**What would end users (brand owners, enforcers) require from the testing of a potential portable solution to demonstrate effectiveness?**

Satisfactory testing using blind sample sets would be required to demonstrate the effectiveness of a proposed technology.

The discussion then went on to consider desirable specifications for a developed portable technology. It was noted that detection levels needs to be fit for purpose. For example, if looking for denaturants, it isn’t just enough to consider the levels assuming only untreated denatured alcohol is used in a counterfeit; dilution and attempts at denaturant removal could lower levels, and such practices should also be identified.

Other suggestions put forward for a desired portable instrument specification included:

- Software that can be easily established and used, and that can be adapted over time (particularly for devices aimed at brand authentication).

- Easy to understand outputs. Interpretation of spectra of other acquired information should be left to the portable device and not the operator. A simple yes/no/maybe response is probably all that is required in the case of a brand authentication device. The traffic light system used by Ocean Optics in their Spirit Sampler was recommended. However, where the question being asked of a sample is more critical e.g. “Does this sample pose a health risk?”, it was noted that a response would need to be more definitive.

- The capability of detecting and identifying more unknowns in a sample.

- Preferentially, the ability to authenticate through a spirit bottle.

- Most importantly, it was noted, any new technologies must be able to offer measureable improvements over authentication techniques that are currently available.

One of the enforcement agency representatives noted that a device with the ability to identify brand substitution that could be used by enforcement bodies, would be beneficial. This would help enforcement agencies considerably by filtering out a large number of samples prior to confirmatory analysis in the laboratory. Following on from this point, a word of caution was raised about trying to force portable devices to do too much. It was noted that a device that has a reasonable success rate
in identifying non-authentic samples might be sufficient; so long as it reduced the number of samples being sent on for further laboratory analysis.

**Suggested Actions:**

- Well run, blind sample tests should be used to prove the capabilities of the technologies being tested. However, devices need to be fit for purpose, and screening devices do not need the same level of specificity as the confirmatory laboratory techniques.

- Easy to use and adaptable software should be targeted.

- Consideration should be given to how/whether portable brand authentication devices can be made available to enforcement agencies.

*Are there any recommendations as to the laboratory methods/techniques we should be investigating? What are the spirit drink authenticity issues with which we should challenge new or improved methods/techniques?*

The ability to determine maize based alcohol from cane based alcohol, which is currently not possible in highly rectified spirits using carbon or hydrogen isotope techniques, was a suggested area in which laboratory work could prove useful. Another key challenge was the determination of maturation age, although concern was expressed over publicising any success in this area, since this would give counterfeiters valuable information on the spirit industry’s products, which could be used to create less detectable counterfeits.

There was an eagerness to explore new laboratory analytical methods, especially those that could identify markers for counterfeit/authentic samples that have not previously been employed in the authentication of spirits. However, it was noted that such laboratory methods need to offer measurable improvements over that which is currently available.

One area of concern with regards to authenticity was the ability of the product to change over time in the bottle. Wine and gin have been noted to change over time and in transit due to temperature; colour fade has been observed in whiskies. Care should be taken to account for these phenomena.

Another challenge noted was the complexity of authenticating a spirit category with a portable or laboratory technology. For example, identifying whether a product is Scotch Whisky, with the wide range of products and styles this category encompasses, is significantly harder than authenticating a brand, which has a much tighter analytical profile.

Lastly, it was noted that whilst it is important to concentrate on making portable analysis methods rapid, work should also be targeted at improving sample turnaround for laboratory analyses (as well as other metrics such as sensitivity and selectivity), since often results from confirmatory analysis are needed quickly.
Suggested Actions:

- Developed technologies will need to evaluate the impact of environmental variables on the parameters being tested, to ensure the level of false negatives is minimised.

**What information would you like to see on the secure website?**

Sharing information on the authentication and quality assurance of spirits has always been a concern for the spirits industry and sharing information as part of this project was also a concern. It was noted that information would be shared on a secure website provided by FERA. There was some nervousness in this respect from some of the industry representatives about the type of information that was uploaded to the website, even if a section of it was secure. It was suggested that only information in the public domain should be uploaded. The level of security employed on the website was also questioned.

Suggested Actions:

- Publishing details on how products can be authenticated can potentially give counterfeiters the information that they need to create improved counterfeits, and the published knowledge will then be devalued. Information sharing as part of the Spirit Drinks Work Package should be carefully considered by the Work Package Partners who will be in control of its dissemination. This will include information uploaded to a shared secure website.

**How best to collate information on analytical methods used for spirit drink analysis?**

There was little feedback on this discussion point. The suggestion of a survey of relevant industry experts was tacitly accepted.

**What mechanisms could be employed to increase interaction/trust between a) brand owners, industry experts and b) third party laboratories (e.g. contract, enforcement)?**

Compliance testing and training of laboratories external to the spirit drinks industry was discussed under this heading. Protocols for best practice in spirit analysis were suggested, as was the validation of laboratories by the distribution of test samples. However, caution was still advised about providing too much information to third parties about methods of authentication, for fear of identifying ways in which counterfeits could circumvent conventional analysis. Brand authentication, it was stressed, is led by individual companies and sharing of authentication data would be unlikely as this is regarded as intellectual property. It was suggested that the provision of guidance/protocols could be via online courses, perhaps linked to the IBD (Institute of Brewing and Distilling) or another training organisation such as a partner university.
Suggested Actions:

- Guidance could certainly be provided to non-industry laboratories, but care would have to be taken to ensure disseminated information is controlled to the industry’s benefit. This area is being considered further within its own Deliverable (5.4) and further discussion is obviously required.
Aim:
The Scotch Whisky Research Institute is leading one of the Work Packages in the EU funded FoodIntegrity Project. This work is designed to address and help improve the following key points in spirit drink authentication:

- Development of field devices to detect harmful/non-potable constituents in spirit drinks and authenticate branded spirits and/or specific spirit categories
- Further development of laboratory based methods to complement screening based technologies
- Construction of recommended protocols for assessing safety/authenticity of spirit drink products
- Provision of guidance/training in appropriate spirit drinks analysis and compliance testing/certification of external laboratories

This seminar is designed to provide the attendee with:

- Information on the work being undertaken as part of the Spirit Drinks Work Package in the FoodIntegrity Project
- Presentations from technology providers applying their solutions to the authentication of spirit drinks both in the field and in the laboratory and the opportunity to interact during coffee and lunch
- Opportunities to contribute to direction of the project – particularly in sharing methodologies, best practice and improving networking amongst those interested in authenticating spirit beverages

Audience:
The target audience for this seminar are those individuals with responsibilities for spirit drink authentication, whether analysts, brand managers, enforcers or those involved in regulatory issues. There will be a focus on some of the technical aspects of spirit authentication, but this will be put into the context of industry requirements and the wider authentication community. All those involved in spirit drink authentication will find the seminar of relevance.

Please note: as this is an EU based project, if successful, we plan to replicate this event in mainland Europe later in the year, most likely in Brussels in September.
Programme:

We aim to follow a similar programme to that employed at a previous Authentication Day held at the Scotch Whisky Research Institute. The first talk, from the SWRI, will be open to all attendees and technology providers. Following this, technology providers will only be in attendance at the session at which they are presenting. The final session before lunch will aim to collect thoughts of attendees on the information presented in all the presentations, to help shape future work within the project. There will be a chance to interact with external speakers at coffee and lunch, and to inspect any technology they may have to display.

Please find below the draft running order. Exact timings on the day may change, but the seminar will start at 9.30 a.m. (9.00 a.m. for coffee) and end at 3.00 p.m. at the latest.

Timings:

09.00 - 09.30: Coffee

09.30 – 09.40: Welcome to the Scotch Whisky Research Institute - SWRI
09.40 – 10:10: The Spirit Drinks Work Package – Aims, Objectives and Work Undertaken - SWRI
10:10 – 10:30: Presentation – Ocean Optics
10:30 – 10:50: Presentation – Microsaic
10:50 – 11:10: Presentation – VUV Analytics

11:10 - 11.30: Coffee – with a further chance to interact with external speakers

11.30 – 11.50: Presentation – Distilled Solutions Limited
11.50 – 12:10: Presentation – Torion (Perkin Elmer)
12:10 – 12:30: Presentation – Anatune
12:30 – 13:00: Discussion – Developing Spirit Authentication, Next Steps in the Spirit Drinks Work Package

13:00 – 14:00: Lunch – with a further chance to interact with external speakers

Tours of the SWRI will be offered after lunch (at 14.00 should timings run to plan).

15:00 Close

Where and when: The Seminar is being held at the Scotch Whisky Research Institute, Riccarton, Edinburgh, EH14 4AP on Tuesday 26th May. Coffee/tea will be available from 09.00, with the seminar starting at 9.30 and ending at 15.00 at the latest. A light buffet lunch will be provided.

Charges and Booking: This event is free to invited personnel. If you would like to forward this flyer to other parties who you think will benefit from attendance, please do. To reserve your place please email mhairi.stewart@swri.co.uk, referring to the FoodIntegrity Spirit Drinks Authentication Seminar.
FOODINTEGRITY
Ensuring the Integrity of the European food chain

613688: Collaborative Project

Seventh Framework Programme
KBBE.2013.2.4-01: Assuring quality and authenticity in the food chain

Deliverable: D5.3 Part III
Title: Platform for Dissemination of Guidance – Survey of Analytical Methods Used for the Quality Assurance of Spirit Drinks

Author(s): Ian Goodall
Beneficiary(s): The Scotch Whisky Research Institute (Partner 34 in the FoodIntegrity Consortium)
Date of preparation: April 2016 – December 2016
Period covered: June 2015 – December 2016
Status: Version 1

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The project has received funding from the European Union’s Seventh Framework Programme for research, technological development and demonstration under grant agreement No. 613688.
### Deliverable 5.3 Part III

**Survey of Analytical Methods Used for the Quality Assurance of Spirit Drinks**

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1 Executive Summary

- Survey polled responses from 82 respondents.

- The majority preference for the sharing of contact details of participants in the Spirit Drinks Authenticity Network was as part of a private, members only, website.

- There was a reasonable consensus on the principal methods employed for the purposes of spirit drink authentication and quality control analysis. The principal analyses listed were as follows:
  
  - alcohol strength by densitometry
  - major volatile congeners
  - maturation related congeners
  - denaturants/additional markers for contaminants
  - sugars
  - acidities
  - metals
  - UV/Vis profiling
  - isotope analysis

- Gaps noted in the availability of reference methods for spirit drinks were:
  
  - denaturants
  - alcohol strength by IR spectroscopy
  - gross (apparent) alcohol strength
  - flavourings
  - isotopic measurements
  - thujone
  - conductivity
  - hydrocyanic acid
  - ethyl carbamate by GC/MS
  - any compound regulated in worldwide markets (e.g. metals)

- Suggested updates to reference methods included:
  
  - The updating of the neutral alcohol analyses methods listed in Regulation 625/2003, including an improvement in terms of sensitivities, given the low levels of volatiles compounds in neutral alcohol/vodka.
  - The removal of pycnometry from the measurement of alcohol strength.
  - An improvement in the sensitivity of the method for the measurement of cyanide compounds.
  - The inclusion of a method for the determination of alkalinity for spirit drinks with pH values greater than 7.
o Careful consideration of the inclusion of automatic distillation methods in the recommended methods for distillation and densitometry to measure alcohol strength.

o Provision of information on interpretation to be provided for authentication purposes, e.g. usefulness of compound ratios in the authentication of spirit drinks.

o The inclusion of enforceable analytical limits for spirit beverage authentication e.g. minimum maturation congeners for whisky.

o The establishment of a reference method for $^{13}$C SNIF NMR.

- Suggested means of addressing reference method deficiencies were via:
  
  o spiritsEUROPE,
  
    - the establishment of a worldwide association for spirit drinks analysis,
    - an EU subgroup,
    - a European database on alcohols/alcoholic drinks (such as the one for wines).

The use of appropriate validation or organised interlaboratory comparison was noted.

- This report fulfils Milestone 17 in the FoodIntegrity Project.

2 Description of Deliverable

The aim of this deliverable was twofold. Firstly, to establish a complete set of contact details from as many people within the Food Integrity Spirit Drinks Authenticity Network as possible, and their attitude about sharing their contact details within the network. Secondly, to identify the range of methods being employed by respondents in analysing spirit drinks and the gaps that respondents felt existed. The outcomes of this deliverable will be made publicly available and will inform discussions on analytical methodology for spirit drinks within spiritsEUROPE and its technical-scientific group and other appropriate fora. The information has (and will be) used to ensure the Work Package 2 KnowledgeBase on analytical methods for authenticating food products is correctly represented for spirit drinks analysis methods.

The data obtained from the survey has been collated and can be made available on request to ian.goodall@swri.co.uk. The source of each response will however be anonymised prior to release.
3 Results from the Deliverable

The first section of the survey was designed to establish some basic information from the respondents regarding contact details, areas of interest in spirit drinks authentication and quality assurance, and attitudes towards sharing contact details.

Question 1: Contact Details Information Request

The distribution of the location of the respondents was split according to whether they were based in the UK, in the rest of Europe, or in the rest of the World. The distribution can be seen in Figure 1. Half the respondents were based in the UK.

Question 2: Area of Interest in Spirit Drinks Analysis and Authentication

Question 2 requested information from the respondents on their areas of interest within spirit drinks authentication and quality/safety assurance. Unsurprisingly, over 80% of the respondents were involved in the analysis of spirit drinks. 52% indicated they were involved in regulation as applied to spirit drinks. 27% of the respondents were involved in enforcement, the respondents mostly representing customs or other state food laboratories, but also spirit drink trade bodies with enforcement roles and some brand owners. 23% of respondents were involved with brand management.

13% of the respondents were involved in the FoodIntegrity project as a consortium member.

Figure 1: Distribution of respondent location
Figure 2: Distribution of Respondents’ Areas of Interest (more than one answer could be selected)

Question 3: Attitude to Permitted Sharing of Profile Information

This question was designed to determine how respondents would view having their contact details shared with a wider audience, as part of the network being developed on spirit drink authentication and quality assurance. Three ways were presented as to how this contact network could exist.

- As part of a publicly available forum (e.g. website) dedicated to the FoodIntegrity Spirit Drinks Work Package (Public)
- As part of a "members only" forum (e.g. website) dedicated to the FoodIntegrity Spirit Drinks Work Package (Member Only)
- As a list only available to the Work Package leaders of the FoodIntegrity Spirit Drinks Work Package (Private).

As can be seen from the responses (Figure 3), attitudes as to sharing of contact detail information varied. The most popular selection was to have details shared as part of a ‘members only’ network (51%). Only 28% of the respondents would be happy to have their information shared publicly. Whilst it is not necessary to have a one-size fits all approach to the sharing of information within the Spirit Drinks Authenticity Network, the simpler the organisation of contact details information the better.
It was noted that out of the 23 respondents (28%) who indicated that they would be happy having their contact details being made publicly available, only 3 were representatives of spirit producing companies. The spirits industry would prefer to have their details restricted, most to a members’ only level, although a few respondents indicated they would only want details available to the Work Package leaders, presumably to reduce email contact. This attitude is reflected by one of the comments received in this section:

"Company policy is active here - the front line for all external contact is our communications team in order to appropriately filter press and general public queries"

It was noted that the respondents who would be happy to have their contact details publicly shared were generally operators providing commercial services (e.g. contract labs or technology providers), involved in research/academia, or representatives of government laboratories (e.g. customs labs). This group did however, include representatives of spirit industry trade organisations, such as the Scotch Whisky Association (SWA), the Scotch Whisky Research Institute (SWRI) and the International Federation of Spirit Producers (IFSP), although a note of caution was advised by one respondent:

"... My contact details are already on the SWA website as a contact. As for Food Integrity, I would want anyone who had a Scotch Whisky issue to contact us, and so it would make sense for those contact details to be easily available. However, the context of the listing is important – e.g. ... I am not an analyst ...

An additional note of caution was sounded by the following comment about sharing data publicly:

"I'm happy to have this available with the proviso that my email and telephone numbers would be adequately protected such that they cannot be scraped by robots searching for email addresses to spam"

![Q3 - Attitude To Sharing of Profile Information](image)

Figure 3: Distribution of Respondents’ Attitudes to Permitted Sharing of Profile Information
Question 4: Are you Able to Supply Information on Analytical Methodology?

Out of the 82 respondents, approximately 60% (49 respondents) provided a unique response in respect of the analytical methodology their organisation used for the authenticity/quality/safety assurance of spirits. Duplication of information supplied from different representatives of the same company was not counted. A number of respondents relied on others in their organisation to provide material data in this area, in order to prevent such duplication.

Figure 4: Respondents able to supply information on Analytical Methodology.

Analytical Methodology: Information

The techniques identified in this section were reported as being used in the safety/quality assurance/authentication of spirit drinks by the respondents. Despite a request for all methods used, the focus of responses was on those analyses used for authentication purposes. Thus, for example, there were a limited number of responses indicating that ethyl carbamate was measured, even though this is a commonly analysed compound for quality control purposes.

A core suite of target analytical parameters was referred to by many respondents. These are as follows:

- Alcohol strength measurement*
- Measurement of a variety of major volatile congeners in spirit drinks*
- Measurement of maturation related congeners (for whisky/cognac/brandy)*
- Measurement of denaturants/additional markers for contaminants
- Measurement of sugars*
- Measurement of acidities*
- Measurement of metals
UV/Vis profiling
Isotope Analysis

(* - EU reference method available for spirit drinks)
Other techniques appear to be more particular to the practices of individual laboratories. Further information is provided below.

**Analytical Parameters 1 – Alcohol Strength**

Out of 49 respondents who provided information on analytical methodologies used within their organisation, 35 reported the measurement of alcohol strength. This is unsurprising given the fact that many spirit drinks have minimum (and in some cases) maximum requirements for alcohol strength and that several limiting analytical parameters in Commission Regulation (EC) No 110/2008 are measured in g per hectolitre of 100% alcohol.

In most cases, the specific use mentioned for authentication purposes was to determine whether a product had been diluted. The addition of sugar can also be indicated when the density measured after the reference distillation step is compared with that taken without a distillation step.

For the measurement of alcohol strength, a number of techniques were employed, and some respondents used more than one technique. These were as follows:

a) Densitometry, or distillation followed by densitometry (94% of those reporting alcohol strength measurement employed this method).

Respondents mostly referred to the use of the EU reference method for alcohol strength analysis, as contained in Commission Regulation (EC) No 2870/2000 (Annex I) or the aligned OIV methods (OIV-MA-BS01 to OIV-MA-BS05), although national standards were also referred to e.g. Mexican Official Standard NOM-006-SCFI-2012 and BS 733-2:1987 on “Pyknometers. Methods for calibration and use of pyknometers”, as well as methods supported by the Alcohol and Tobacco Tax and Trade Bureau/AOAC (942.06, 945.07, 982.10, 983.12). Although respondents reported the use of distillation and densitometry or just densitometry it is assumed the method used (despite the official EU reference method being distillation followed by densitometry) will be chosen based on the application, since some spirits can provide a suitable reading without distillation, provided the level of dissolved solid matter is low.

b) NIR or MIR Spectroscopy (29% of those reporting alcohol strength measurement employed this method).

The only reference method referred to was that provided by the OIV (OIV-MA-BS-08), which refers to the principles involved in establishing a spectroscopic calibration for the measurement of alcohol strength based on the EU/OIV reference methods referred to in paragraph a) above.
c) GC-FID (6% of those reporting alcohol strength measurements employed this method).

d) Enzymatic Methods - only one respondent reported using an enzymatic technique for measuring alcohol strength in low alcohol products.

**Analytical Parameters 2 – Major volatile congeners in spirit drinks**

Another commonly employed analytical technique for the authentication and quality assurance of spirits. The exact list of measured constituents, if listed, varied and in some cases was obviously related to the nature of the spirit drink being analysed. The basis for the analysis methods used was, as with alcohol strength, principally the method listed in *Commission Regulation (EC) No 2870/2000* (Annex III.2) or the aligned OIV method (OIV-MA-BS-14). Again, a couple of additional references were noted (Mexican Official Standard NOM-006-SCFI-2012, Alcohol and Tobacco Tax and Trade Bureau/AOAC 968.09 and 972.10 and the Chinese Standard GB/T 10345. These are all GC-FID based methods. Some of the reported methods were obviously adapted from the standard reference methods to include additional analytes.

Whilst 30 respondents employed a GC-FID method to determine major volatile congeners, one respondent employed GC-MS to analyse such compounds. The principal use of the technique in most cases is a combination of both quality control and authentication.

In addition to the standard major volatile congeners identified in spirit drinks, a number of respondents (9) reported the use of GC-MS (8) and GC-FID (2) for the identification of minor volatile components. Such techniques are aimed at a wider range of compounds at lower concentration levels. The use of the volatile profiles obtained are principally for fingerprinting products and subsequent authentication, particularly the identification of atypical flavourings or contaminants.

**Analytical Parameters 3 – Maturation related congeners**

Identified by a number of respondents (14) this is a method specifically employed by the those profiling brown (cask matured) spirits. The method employed in all cases was HPLC (with UV or UV/fluorescence, where identified). Where the method was referenced, the OIV method was referred to (or in one case the Mexican Official Standard NOM-006-SCFI-2012). Many respondents replied prior to the addition of the OIV method (OIV-MA-BS-16: R2009) to the list of EU spirit drinks reference methods in *Commission Regulation (EC) No 2870/2000* (Annex X), otherwise it is anticipated that this would have been a common reference. An appropriate EU reference method for maturation related congeners was requested by a number of respondents (see the section on gap analysis) prior to its actual introduction.
As with the major volatile congeners, MS (LC-MS) was used to provide additional information on levels of maturation and other semi- to non-volatile compounds by a number of laboratories. Again, this additional information is related to both fingerprinting and the detection of contaminants resulting from adulteration/counterfeiting.

**Analytical Parameters 4 – Denaturants/additional markers for contaminants**

Although the detection of markers for contaminants is covered to a certain extent by analytical parameters 2 and 3 above, a number of methods were specifically reported in the survey that addressed specific markers for counterfeit detection. These principally related to compounds used as alcohol denaturants or markers for such use: denatonium benzoate, benzoic acid, methanol, tert-butyl alcohol, isopropanol were noted. Techniques appear to vary depending on the laboratory and the compounds being targeted; HPLC-DAD, GC-FID, GC-MS, GC-MS/MS are all mentioned.

**Analytical Parameters 5 – Sugars**

Two different chromatography based techniques, when clearly specified, dominate the measurement of sugars. The first is the use of the HPLC-RI. As is indicated by its recent incorporation into the list of analytical reference methods in Commission Regulation (EC) No 2870/2000 (Annex VIII), it is used principally for distilled spirits with higher levels of sugar content, e.g. pastis and liqueurs. A second method, recorded by four respondents, indicated the use of ion chromatography, typically used in conjunction with an electrochemical detector, which provides lower levels of detection and can allow trace levels of sugars present in certain distilled spirits (e.g. from maturation) to be distinguished from higher levels of sweetening or addition through adulteration/counterfeiting.

Two additional techniques for the measurement of sugars were noted. The use of enzymatic techniques and the use of NIR. The spectroscopic method is likely to be more suitable for the higher levels typically targeted by HPLC-RI.

**Analytical Parameters 5 – Acidities**

The measurement of acidities or pH was undertaken by a number of respondents for the profiling of spirits, whether for quality control, regulatory compliance or authentication purposes (based on expected profiles). The principal referenced methods employed were the recently introduced method for volatile acidity (and hence total and fixed acidity) in Commission Regulation (EC) No 2870/2000 (Annex III.3) and the aligned OIV method OIV-MA-BS-12 (OIV methods OIV-MA-AS313-01 to -03 for wines were also noted). pH measurements were also noted as useful for quality control and authentication (pH meter and NIR).

Certain acids (malic, citric, lactic) were measured by some respondents in wines and low alcohol beverage products.
Analytical Parameter 6 – Metals

Metals were measured by 7 respondents for quality control/authentication purposes. Techniques were quite varied and included ICP-MS, AAS, ion chromatography and ICP-OES. No specific reference method was noted in responses. There is no EU analytical reference method for the analysis of metals in spirit drinks.

Analytical Parameter 7 – Spectroscopy

The use of spectroscopy for profiling brands and subsequent authentication was undertaken by a relatively large number of respondents (16). Unsurprisingly, these respondents were principally based within the spirit drinks industry. UV-Vis was the technique employed by all the industrial respondents. This was applied to a variety of spirits including cognac, whisky, and vodka.

The non-industrial respondents employed a wider range of spectroscopic techniques including NIR, FT-IR and Raman, tending to indicate a difference between regular UV/Vis based instruments for spirit drink authentication and more experimental, investigatory techniques.

Analytical Parameter 8 – Stable Isotope Ratio Analysis (SIRA)

Another commonly employed technique, this analysis was principally the remit of customs laboratories/state food labs/commercial analysis service providers rather than industry. The principal isotope analyses either used SNIF NMR or IRMS (isotope ratio mass spectrometry) and were:

a) the site-specific D/H analysis of ethanol, for information on the botanical origin of the substrate (e.g. cereal) used to supply the fermentable sugars. The reference method that was quoted for this analysis is – OIV method OIV-MA-BS-23 - “Determination of the deuterium distribution in ethanol by nuclear magnetic resonance of deuterium (RMN FINS/SNIF NMR”).

b) the $^{13}$C/$^{12}$C isotope ratio analysis of ethanol, for information on the botanical origin of the substrate (often used in conjunction with the D/H analysis). The reference method that was quoted for this analysis is – OIV method OIV-MA-BS-22 – “Determination by isotope ratio mass spectrometry of the $^{13}$C/$^{12}$C ratio of ethanol" *

c) the $^{18}$O/$^{16}$O isotope ratio analysis of ethanol, for the identification of the presence of exogenous alcohol,

d) the $^{18}$O/$^{16}$O and D/H analysis of the water (or complete product) for information on the geographical origin of the product. The following reference method was quoted for this analysis - OIV method OIV-MA-AS2-12 – “Method for $^{18}$O/$^{16}$O isotope ratio determination of water in wines and must”.

*
Commission Regulation (EEC) No 2676/90 of 17 September 1990 determining Community methods for the analysis of wines was also referenced in relation to site-specific D/H analysis and $^{13}$C/$^{12}$C isotope analysis of ethanol."

*One respondent is now able to undertake a site-specific analysis of the carbon isotopes in ethanol, $^{13}$C SNIF NMR, to provide additional information on the botanical origin of ethanol – see Thomas et al (2010).

**Analytical Parameter 9 – Other parameters**

The following analyses were also listed by a limited number of respondents for spirit drink authentication and/or quality control. As suggested in the introduction to this section, it is suspected that the number of laboratories using these methods is higher than reported, but that some laboratories use them only for QC purposes, instead of for authentication, and did not add them to their list of reported analyses as a result.

a) **The Analysis of Anethole** by GC-FID. The two reference methods listed were the [OIV method](#) OIV-MA-BS-15 and the aligned method in Commission Regulation (EC) No 2870/2000 (Annex V).

b) **The Analysis of Ethyl Carbamate** by GC/MS. The only reference listed was to the [OIV method](#) OIV-MA-BS-25.

c) **The Analysis of Diacetyl and 2,3-Pentadione** by GC-ECD in vodka and neutral spirit for QC and regulatory purposes. No reference method was listed.

d) **The Analysis of Phthalates** by GC/MS. No reference method was listed.

e) **The Analysis of Haloanisoles** by GC/MS. No reference method was listed.

f) **The Analysis of Pesticides** by LC/MS. No reference method was listed.

g) **The Analysis of Phenols** – principally related to Scotch Whisky. Two techniques were listed – the use of HPLC with either UV or fluorescence, and the use of a UV method for total phenols. No reference method was listed.

h) **The Analysis of total Dry Extract** by gravimetry. Two respondents reported the use of this method. Two methods were referred to: Mexican Official Standard NOM-006-SCFI-2012 and the [OIV method](#) OIV-MA-BS-09, which is aligned with Commission Regulation (EC) No 2870/2000 (Annex II).

i) **The Analysis of Cyanide** by colorimetry. This method was used for quality control, regulatory compliance and authentication. The reference method referred to was that defined in Recueil des méthodes internationales d’analyse des boissons.

j) **The Analysis of Furfural** by colorimetry. The reference method referred to was that defined in Recueil des méthodes internationales d’analyse des boissons spiritueuses, des alcools et de la fraction aromatique des boissons, 1994.

k) **The Analysis of Sulfur Dioxide** by titration. The reference method used is the OIV method – OIV-MA-AS323-04B.

l) **The Analysis of Turbidity**, identified as being useful for determining dilution. No reference method was listed in the responses.

m) **The Analysis of Conductivity**, identified as being useful for quality control and determining dilution.

n) **The Analysis of Pressure**, identified as being a quality control measurement for carbonated beverages.

o) **The Analysis of Age**, using liquid scintillation counting to check age claims. OIV reference method for the liquid scintillation determination of ethanol is OIV-MA-BS-24.

p) **Sensory Screening.** Although a subjective test, only one laboratory reported the use of a Sensory Screening test (nosing) for the authentication of spirits. It is likely that most laboratories will subject a suspect sample to some form of organoleptic analysis, which, given the fact that authenticity is in doubt, should not be in the form of a taste test. The under-representation of such a test in the given responses is probably due to the often informal nature of such a test and, as noted, its subjective nature.

Additional notes:

1) The measurement of both sugars and denatonium benzoate in spirit drinks, as undertaken by one of the customs laboratory respondents, were identified as being measured using the "customs laboratory method". It may be useful to know what these methods are, and how they compare with EU reference methods or other standard analysis methods that are available.

2) Of the list of reference methods for spirit drinks in the Commission Regulation (EC) No 2870/2000, the techniques that were not mentioned in the responses were: Annex VI – the measurement of glycyrrhizic acid, Annex VII – the measurement of chalcones and Annex IX – the measurement of egg yolk.
Follow up Questions: Gaps in Analytical Methodology and Other Comments

Gaps in Analytical Reference Methods

1. A number of respondents noted the lack of reference methods for the following three analyses:
   a. Measurement of total, volatile and fixed acidities and
   b. Measurement of total sugars
   c. Measurement of maturation related congeners
   These have been addressed by the recent publication of Commission Implementing Regulation (EU) 2016/635 of 22 April 2016 amending the Annexes to Commission Regulation (EC) No 2870/2000 as regards reference methods for the analysis of spirit drinks. However, the reference method measurement for total sugars is designed to determine compliance with minimum levels for certain spirit types, the lowest working standard concentration in the method being 2.5 g/l (2500 mg/l). Comments were received that reference methods to detect lower levels of sugars, signifying unlawful addition, should be established.

2. A request was made for a reference method for the measurement of denaturants in spirit drinks. It should be noted that the Joint Research Centre Institute for Reference Materials and Measurements (IRMM) have recently developed methods that will be circulated through the customs network. The FoodIntegrity consortium have asked for these to be made available through the FoodIntegrity website.

3. The creation of reference spectroscopic methods for the determination of alcoholic strength by FTIR/NIR/MIR was recommended by a number of respondents. It was noted that there was a lack of guidelines for the validation of methods using such techniques. Extension of such techniques to the rapid analysis of counterfeit spirits using portable hand-held instrumentation (i.e. RAMAN / FTIR), in addition to laboratory based equipment, was also requested.

4. The use of GC-MS to detect the unlawful addition of flavourings was noted as a possible addition to the list of reference methods. (This may require a tighter scope to be practical.) It was noted that it is difficult to determine, without suitable references, if a spirit has been unlawfully flavoured.

5. Several respondents stated that no EU reference isotopic techniques for ethanol or water (IRMS or SNIF-NMR) are approved for spirit analysis, only for wines. The use of trace metals for authentication purposes was also recommended as a reference method.

6. A reference method for the determination of thujone in absinthe was noted by a couple of respondents.

7. A method for the measurement of gross (presumably apparent?) alcohol strength was requested.
8. The use of conductivity for measurement of vodkas was mentioned as an option for a recommended method.

9. The introduction of a reference method for hydrocyanic acid (HCN) using ion chromatography was recommended, presumably as a more specific alternative to the method validated for, but ultimately rejected from inclusion in the EU Regulation on spirit drinks reference methods (based on colorimetry).

**Suggested updates to available reference methods:**

1. Recommendations were made to update the analyses for neutral alcohol using old fashioned wet chemistry methods (which are confusingly reported within a wine based Regulation 625/2003 and are presumably no longer in force since the repeal of the Regulation 1623/2000 which Regulation 625/2003 amends). A move to GC was suggested in most cases.

2. The removal of pycnometry from the options for measuring alcohol strength by densitometry was recommended by more than one respondent. Also the use of automatic devices in the distillation step for alcoholic strength determination should be specifically permitted.

3. In contrast to the opinion above, one respondent felt that recovery of ethanol by a steam distillation should NOT be specifically added to the alcohol strength reference method without extensive peer reviewed validation of the methodologies, since in his opinion this technique has not been proven.

4. It was recommended that ethyl carbamate measurement by direct injection GC/MS should be added to the list of reference methods (with sufficient clarification as to use). (It is noted that a method has been developed by a CEN working group.)

5. One respondent noted that volatile substances are expressed as a sum, whereas useful information, such as the ratio between higher alcohols should be identified in the methods. This potentially extends the reference methods to provide interpretative advice, from which they currently refrain. Also, it was recommended that a maximum value of ethyl acetate should be determined, since high levels of ethyl acetate affect the quality of spirit drinks.

6. The use of $^{13}$C SNIF-NMR when applied to ethanol has been shown to capable of differentiating authentic 100% agave tequila, as well as misto tequila (which is made from at least 51% agave), from products made from a larger proportion of cane or maize sugar and therefore not complying with the legal definition of tequila (Thomas *et al*, 2010). The scientific paper provides details of a small interlaboratory study. One of the report’s authors suggests that the $^2$H SNIF NMR OIV method OIV-MA-BS-23 should be updated with $^{13}$C-SNIF NMR for the detection of C4 alcohol (from maize, cane) in CAM spirit (tequila). The respondent notes that a draft method has been
written, which could form the basis of an appropriate collaborative study, and as the method is part of the FIT-PTS proficiency testing scheme, “reference materials” could be made available.

7. The following were also noted:
   a) for volatile congeners, the concentration range of the method (e.g. for aldehydes, methanol, esters, higher alcohols) is applicable only for certain alcoholic beverages, such as brandy and whisky and is not intended for low concentrations, such as found in vodka (see point 1 in this section);
   b) the determination of total dry extract of spirit drinks of vitivinicultural origin by the gravimetric OIV method (OIV-MA-BS-09 : R2009) is specified for the determination of the total dry extract in spirit drinks less than 15 g/l of dry matter but may be used in beverages with a higher extract; the results for the determination of total dry extract usual by calculation OIV method (OIV-MA-BS-10 : R2009) were noted as being very inaccurate;
   c) total acidities do not apply to many drinks that have a pH above 7, which require a determination of alkalinity;
   d) for cyanide derivatives there is need for a method with a limit of quantification of less than 0.1 mg/L.

Means of Addressing Reference Method Deficiencies

1. The establishment of some sort of worldwide association was suggested by one respondent, a “Distilling Chemists and Microbiologists Society”. Others felt this should be undertaken by spiritsEUROPE, contacting trade associations and technical groups such as SWA, SWRI and BNIC.

2. Another respondent felt there needed to be a procedure for looking regularly at the available range of spirit drinks analysis methods, updating where required. Whilst appropriate checks and input would be required, this procedure should be as quick and simple as possible. A panel of suitably qualified experts from across the spirits industry should undertake this action, taking such external input as is needed, and recommend actions to the Commission.

3. Whilst one respondent was of the opinion that there were no obvious gaps in relation to the reference methods used for brand authenticity in the EU Regulation, he felt that if a parameter is regulated, then it should have a reference method. (Presumably this is a worldwide consideration since all regulated spirit parameters, other than HCN, have EU reference methods). He recommended that an EU Sub group should exist for reference method ratification and that validation protocols should be authored to prevent adoption of methodologies based on a limited validation set.

4. A fourth respondent felt that the process of updating methods should be via organised interlaboratory comparison.
5. Establishment of more enforceable limits for spirit beverage authentication was noted as a benefit by a number of respondents, which would enable the methods to be better employed. Fixing a minimum limit for volatile substances and maturation related congeners in whiskies and rums was suggested, as was fixing a limit for the maximum content of sugar in a spirit that has not been sweetened. One respondent recommended the creation of a European Database on alcohols/alcoholic drinks, equivalent to that generated by the Joint Research Centre for wines.

6. A couple of respondents noted the benefit of the following: knowing the levels of volatile substances and their internal ratios for each trademark drink beverage category or the key characteristics to distinguish and authenticate various types of beverage. Additional suggestions included work meetings for sharing information related to this topic between interested parties and periodic investigation into the practices used in the falsification of drinks.

7. One comment noted that updates/improvements to analytical reference methods should be driven by the spirits industry, rather than "centrally driven" by European legislators.

Other Gaps and Recommendations

1. It was noted that the methods used by countries outside the EU to sum their volatiles is often unclear. Presumably it is being suggested that some international consensus on this measurement should be adopted. It was also noted that methods for summing classes of compounds (e.g. esters, aldehydes, invert sugar) should be established in the EU to help influence worldwide interpretations, which often vary.

2. A US respondent noted that for exports to Europe (and the rest of the world), there is a lack of specific instructions as to methods and limits required for individual countries. It was indicated that it is not always easy to identify the relevant European regulations and there was a lack of resources in the US for distilled spirits, compared to organisations such as the ASBC (American Society of Brewing Chemist), MEBAK (the Mitteleuropäische Brautechnische Analysenkommission e.V.) and the EBC (European Brewing Convention) that provide detailed methods for brewers, or the ASEV (American Society for Enology and Viticulture) for wines. A manual of updated methods was recommended for Europe and the rest of the World in which the methods could be tagged with country symbols to indicate which country needs which tests. It was suggested this could be used to inform the US TTB as to what is needed by each country and to what level of sensitivity (with lists of alternate accepted methods).

3. A list of specialist laboratories who can undertake isotope and GMO type testing, as well as information on GMO detection methods, were highlighted as a gap, although insufficient further detail was provided.
4. Improved capabilities for the differentiation of raw material substrates (e.g. different fruits) were requested by several respondents. In particular, a respondent noted the desire to be able to determine cane alcohol from maize based alcohol. Similarly, (although recognising the difficulties involved) methods for determining geographical origin were noted by a number of respondents, since most spirits listed in EU Regulation 110/2008 with a geographical indication cannot be properly authenticated.

5. The development of portable primary methods of analysis was a suggested benefit (e.g. GC-FID in the field). It was noted that these could be linked to an international database of authentic data on brands, although care would need to be taken to ensure it was not possible to reverse engineer such information.

6. Better knowledge of the analytical profiles of worldwide whiskies was suggested for authentication purposes, since much reported data focuses on distinct well established categories (Scotch, Irish, North American) rather than newer production areas such as Japan and France. A lack of communication between spirit drinks manufacturers and third party laboratories was emphasised by one respondent, who noted that the spirit drinks industry is highly reluctant to share specification/composition information, such as typical congener profiles with acceptable tolerances, with enforcement laboratories.

7. A quick dependable test for methanol was requested.

8. A general point was made about identifying gaps in spirit drink authentication. That is, when considering requirements for the authentication of a spirit category, the definition of that category should be considered in the first instance. Current methods can determine that some suspect products are not authentic, but some non-authentic products may be judged as being consistent with the spirit category since the requirements of the definition are hard, or impossible, to enforce.

Methods do not determine what a product is, only that a product is consistent with a certain definition. The ideal suite of analyses should be able to determine when a suspect sample does not conform to a product definition, e.g. this product was distilled from molasses not cereal: this product was distilled at 96%; this was aged for 1 year not 3 etc. Such aspects of the spirit drinks definitions should be considered, and where gaps exist efforts should be made to work towards bridging them.

The problems in determining whether a product was distilled at less than a certain strength were noted by a number of respondents.

9. It was noted that the customs laboratories and other enforcement laboratories operate different techniques and their available skills/technology differ. On this basis, it was suggested that more consistency and collaboration is required and there should be a harmonised EU-wide set of standard operating procedures shared between enforcement bodies and legitimate trade.
10. An improved ability to determine maturation age was requested by several respondents.

11. The use of markers for certain spirits (e.g. vodka in certain markets) was noted by one respondent as being beneficial for enforcement agencies to detect counterfeit products.

12. It was noted that certain limitations exist in the isotopic analysis of spirit drinks. For example, the site-specific D/H analysis of ethanol, can only detect beet alcohol in other C3 spirits (brandy, kirsch, pear, apple or orange spirits, etc.) and IRMS can only detect C4 alcohol in C3 spirits (or vice versa).
4 References


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FOODINTEGRITY
Ensuring the Integrity of the European food chain

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Deliverable 5.3 Part IV


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1 Executive Summary

Throughout the reporting period, spiritsEUROPE has been actively engaged in the EU and globally, in highlighting the problems from illicit alcohol. This activity has focussed on identifying the drivers for illicit trade and the work needed to address the problems. In addition, efforts have been directed at seeking support for official involvement in measuring the scale of the illicit alcohol market. We have engaged with numerous stakeholders as part of a broader drive to impress upon officials and governments that more needs to be done to eradicate the problems of illicit alcohol.

For the purposes of this report, we have sought to classify our Food Integrity Project (FIP) activities by the main groups of stakeholders: those within the spirits sector; work with Commission and other officials; collaboration with the OECD (Organisation for Economic Co-operation and Development) at a global level; and our efforts with other agencies that are also trying to secure improvements in this area.

The effort to improve the authentication of spirit drink products requires knowledge from around the world of the types, scale and incentives for production of illicit alcohol. Communication routes for the project’s aims and information require us to have a dialogue with numerous bodies, all of which bring different experiences that both help our understanding of the problem and shape the analytical requirement parameters. Furthermore, analytical techniques aren’t the only levers useful to reduce the level of counterfeit alcohol; we are seeking to leverage political capital in an effort to persuade EU and national authorities, as well as agencies elsewhere, to give the issue a higher priority and develop steps to address the concerns. spiritsEUROPE, as the longstanding and well-established voice of the spirits industry (based in Brussels) is ideally placed to lead this effort.

2 Involvement of Spirits Producers and Trade Associations

Within the industry, the dialogue on illicit trade and the FIP takes many forms. At perhaps its most basic level, we have sought to ensure that members are regularly updated on developments. Our membership comprises both the EU’s (and world’s) largest spirits producers and also national trade associations; the latter groups together producers of a specific category (Cognac, Scotch Whisky) or all spirits made in a single country. Through the spiritsEUROPE committee structure (‘internal market’ and ‘fiscal / economic’ in particular, but also dedicated working groups - notably ‘technical / scientific’), updates on FIP issues and the work on illicit alcohol have been circulated and discussed since the project’s inception in 2014.

The spiritsEUROPE website has been updated to include a dedicated page on the FIP, including links to the project’s website. On the spiritsEUROPE (members only) extranet a dedicated ‘Issue Page’ has been created which summarises the main activities since the start of the project and contains links to all the relevant documents and presentations. While access to the site is password-protected, a screen grab of the first page is reproduced below.
In addition, the work on FIP was included in the spiritsEUROPE monthly newsletter that was circulated to over 2,000 traders and officials in the summer of 2015 and again in 2016. Given the importance of the issue, it would be useful for it to feature in further newsletters.

The sector has been closely involved throughout the Commission’s review of Directive 92/83 (see below), which contains the potential for making progress on addressing illicit spirits on a number of fronts. In addition, it has taken the lead in tackling 2 specific cases of excise tax discrimination within EU Member States (Greece and Hungary), both of which are significant factors giving rise to the illicit spirits market in those countries. We would expect both cases to be referred to the European Court of Justice, unless the governments in question take earlier steps to remove the tax preferences. If history is a guide and the tax discrimination is deemed to be illegal, the countries will be obliged to ensure all spirits are treated fairly. While the introduction of non-discriminatory taxation will in itself not remove the problems from illicit alcohol, we believe it will create the conditions for the governments to start tackling it more assiduously because the low taxed spirits at present (essentially domestically produced) will lose their protection and thus also face the pressure of competing with non-taxed illicit spirits.
In addition to the work with our own members, we have been closely involved with the International Alliance for Responsible Drinking (IARD) which has, among other things, developed methodology to measure the scale of illicit alcohol. In discussions with members in EU Member States that suffer most from illicit alcohol, and which are considering commissioning research, we encourage them to use the IARD methodology, so that results from the various studies will be broadly comparable. Separately, in 2 IARD webinars in June 2016, spiritsEUROPE presented its work on illicit alcohol in Europe, highlighting FIP as a key element in tackling the problem.

On the technical front, the Scotch Whisky Research Institute’s survey (Deliverable 5.3 Part III) that seeks information from producers on the analytical techniques used in the spirits sector has been circulated to all members and elicited responses. We have periodically reminded members, most recently in Autumn 2016, that additional input would be helpful and this has brought the involvement of new participants on each occasion.

3 Work with EU officials

The EU spirits industry is subject to 2 excise tax directives that, to a greater or lesser degree, should be changed because shortcomings have been identified, notably (a) they have failed to keep pace with other regulatory and manufacturing developments, and (b) in some ways they provide an incentive for the illicit spirits trade. While it has been found politically difficult to change the ‘minimum rates’ directive (92/84, even though, somewhat perversely, it requires Member States to discriminate against spirits), there has been agreement that a review of the ‘structures’ directive (92/83) would be helpful – work in this area began in 2015.

Improvements in the law could help the fight against illicit alcohol if they can start to tackle elements such as unrecorded home-distilled alcohol, poor enforcement of controls on small distilleries that are allowed to pay reduced rates of tax, and the complicated regimes (operated only by Member States) for denatured alcohol, some of which is ‘cleaned up’ and sold as potable alcohol. The industry has met Commission officials on numerous occasions throughout 2015 and 2016 to discuss the review of 92/83; this has included officials attending internal spiritsEUROPE committee meetings, and the broader forum of the Civil Dialogue Group (DG Agriculture’s platform for liaison with its key stakeholders) to discuss industry concerns with the current rules.

In November 2015, we submitted formal input to 2 parts of the review. Among other things, these highlighted the way in which illicit alcohol is in part a consequence of the Directive providing tax breaks for some small distilleries (the provisions have not been adequately controlled by Member States), and also its failure to include any rules on problematic issues such as home distillation. Further to the submissions, we and members were in direct contact with the consultants employed by the Commission to discuss some of these concerns in more detail.

In addition, spiritsEUROPE hosted a meeting in March 2016 involving officials from the Commission and the Joint Research Centre (JRC), as well as the Scotch Whisky Research Institute, to look at developments on, among other things, the work on denaturants. Member States have responsibility for the provisions on denaturing alcohol but these have been found to be overly-complicated and with little overlap between countries. While there is scope for denatured alcohol, or other distillates outside excise controls, to be diverted into the potable alcohol sector, it has sometimes been difficult to identify the source of the alcohol. Ensuring that all denaturants include chemicals that are harder to remove (and the traces of which would easier to detect in ‘cleaned up’ alcohol) would go some way to deterring fraudsters and identifying fakes at an early stage.
The conclusions from the review were published in mid-2016. On some aspects, such as denaturants, there was support for securing improvements. (Indeed, legislation to introduce a common denaturing formula for all ‘completely denatured’ alcohol was subsequently adopted.) While this looks like a useful move, other areas in the review, partly as a consequence of diverging views between Member States, were less encouraging. For example, while we had highlighted problems, i.e. illicit trade, from poor enforcement of the various ‘reduced rate’ excise tax schemes for distilled alcohol in particular Member States, it does not appear anything will be done in this area.

There is a further opportunity, fortunately, to press for improvements. The EU Council has asked the Commission to undertake further work on the dossier in an effort to prepare a legislative proposal. As part of this, an impact assessment study will be conducted early in 2017 and this will involve a further public consultation. We plan again to raise concerns in an effort to persuade the Commission and Member States to take the issues surrounding illicit alcohol more seriously.

Over a longer period, we have also been trying to determine whether the Commission, or agencies such as the JRC, might be prepared to undertake research into the scale of illicit alcohol in the EU. Research funds are available, for example, through the Commission’s DG GROW for such projects and we have sought to explain that it would be helpful for all parties if funds could be set aside for work on illicit alcohol. While we had hoped the EU’s anti-fraud agency OLAF might be interested in participating, it seems their remit does not include alcohol tax and that instead the Commission’s DG TAXUD would lead in this area.

We have stressed the key elements: consumers are dying from illicit alcohol (one particular case in the Czech Republic caused over 50 fatalities); government revenues are lower than they should be because illicit alcohol is so prevalent in several countries; and consumers increasingly see illicit alcohol as a standard part of their purchases, bringing both health risks and also the perception that illicit alcohol is ‘normal’. The efforts to persuade officials to research the issues more clearly are continuing.

As a separate strand to spiritsEUROPE’s involvement with the Food Integrity Project, we have been liaising with EU officials in an effort to arrange a presentation to the ‘Committee for Spirit Drinks’. This formal group of Member State officials meets roughly 4 -6 times a year to consider issues arising from the EU’s spirit drinks legislation and related matters. The group has been responsible for enacting legislation on the methods of analysis of spirit drinks and, among other issues, it has sometimes also discussed fraud with particular sectors or spirits. A presentation on the spirits workstream in the FIP would help officials, who are also involved with enforcement issues, understand the efforts being made in this area. Regrettably, ‘protocol’ difficulties mean that arranging a presentation is proving more difficult than expected – the effort continues.

4 Work with OECD

Since the start of FIP in 2014, we have been closely involved with the OECD, including being a member of its ‘task force’ on countering illicit trade. As part of this, along with colleagues in the beer sector, throughout 2014 and 2015, we were a key part of the team that prepared a chapter on illicit alcohol for inclusion in the OECD’s publication on illicit trade more broadly. The chapter on alcohol sought to: provide clarity on how illegal alcohol should be categorised; summarise its scale based on studies from around the world; demonstrate its negative impact in numerous areas; and assess the drivers for the illicit alcohol trade.

The OECD’s report “Trade in Counterfeit and Pirated Goods: Mapping the Economic Impact” was published in April 2016, and secured significant coverage in the media, including a major article in the Economist magazine. (While this publication contains a chapter on illicit alcohol that was written by
spiritsEUROPE and colleagues in the beer sector, copyright issues mean we can’t provide a copy - it can be purchased from OECD.) In our follow-up work we highlighted to OECD that more emphasis needed to be put on the drivers for illicit alcohol, not least because at the April conference to launch the publication, several participants noted that, unless the demand for illicit alcohol is curbed, there is little chance of making inroads into tackling the illicit market more generally. We referred specifically to the FIP as an example of work OECD could use to show what is happening at EU level to tackle illicit alcohol. At the OECD’s subsequent conference in Brussels in November 2016, we again highlighted the problems from illicit alcohol in certain countries, stressing that governments also needed to be aware that their own policies, notably excessive tax rates, were sometimes responsible for the increase in illicit alcohol.

5 Work with Other Agencies

There have been discussions with several other actors that are involved in trying to remove problems with illicit trade, details of which are summarised below.

Centre for European Policy Studies - at a seminar on illicit trade in spring 2016 (report here - see top of p4), we highlighted some of the difficulties faced by the spirits sector, notably as a consequence of government policy (underpinned by EU directives) on setting excise tax rates and providing derogations from normal rules in certain circumstances. We also stressed that the lack of proper enforcement of existing rules had contributed to the growth of illicit trade.

Coalition Against Illicit Trade - a dialogue has been opened with this grouping of companies which seek to provide technical solutions to sectors facing problems from counterfeit and illicit trade. We have registered support for the coalition’s aims.

EU Intellectual Property Office - our involvement with this EU agency included a dialogue regarding the formal reports on the extent to which products with ‘geographical indications’ (including spirits) are subject to counterfeit. Following its April 2016 report on infringements in GI protection for EU spirits, wines and foodstuffs, spiritsEUROPE put members directly in touch with EUIPO when it sought specific case studies for a follow up report.

Royal United Services Institute - the dialogue with this UK-based organisation is ongoing: it has already conducted studies on illicit trade and held a conference in Brussels at the end of 2016, at which spiritsEUROPE made a presentation highlighting the problems caused by illicit spirits and urged the Commission to do more to research the issue.
FOODINTEGRITY
Ensuring the Integrity of the European food chain

613688: Collaborative Project

Seventh Framework Programme
KBBE.2013.2.4-01: Assuring quality and authenticity in the food chain

Deliverable: D5.3 Part V
Title: Platform for Dissemination of Guidance – Spirit Drinks Authentication Methods - Bibliography

Author(s): Ian Goodall
Beneficiary(s): The Scotch Whisky Research Institute (Partner 34 of the FoodIntegrity Consortium)
Date of preparation: December 2016
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Status: Version 1

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Deliverable 5.3 Part V

Spirit Drinks Authentication Methods - Bibliography

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1 Aim of the Deliverable

To supplement the Survey work undertaken to determine the analytical methods employed in spirit drinks authentication and quality assurance (Deliverable 5.3, Part III), it was proposed that a database containing at least 50 spirit drink authenticity papers be collated. This part of Deliverable 5.3 reproduces this Bibliography of Spirit Drinks Authentication Methods in text form. It will be made available on the Work Package 5 website, tagged for standard scientific bibliography software. It currently contains 65 references.

For each entry, in addition to the reference (formatted according to the Harvard referencing style), three types of keywords are provided: i) spirit types referred to in the paper, ii) methods discussed/employed in the paper and iii) some additional keywords of relevance to spirit drink authentications (e.g. specific analytes of interest). Links to sites where the papers are either publicly available, or can be purchased, are also provided.

This Bibliography of Spirit Drinks Authentication Methods will be circulated to the members of the Spirit Drinks Authenticity Network. Requests will be made to supply additional references, in accordance with the format established, and to keep the Spirit Drinks Authenticity Network updated with new papers as they are published.
2 Key

**Spirit Types**

Scotch Whisky, Gin etc. relates to the spirit drinks categories under consideration

Spirits relates to a range of spirit drinks

**Methods**

13C IRMS $^{13}\text{C}/^{12}\text{C}$ Stable Isotope Ratio Analysis or isotope ratio mass spectrometry (IRMS)

18O IRMS $^{18}\text{O}/^{16}\text{O}$ Stable Isotope Ratio Analysis or isotope ratio mass spectrometry (IRMS)

2H SNIF $^{2}\text{H}/^{1}\text{H}$ Site-Specific Natural Isotope Fractionation using Nuclear Magnetic Resonance

AMS Accelerator Mass Spectrometry

ATR Attenuated Total Reflectance

CE Capillary Electrophoresis

ESI Electrospray Ionisation

GC-FID Gas Chromatography with Flame Ionisation Detection

GC-MS Gas Chromatography with Mass Spectrometric Detection

FTIR Fourier Transform Infrared Spectroscopy

IC Ion Chromatography

LC-UV Liquid Chromatography with UV Detection

LC-PAD Liquid Chromatography with Pulsed Amperometric Detection

LSC Liquid Scintillation Counting

MIR Mid-Infrared Spectroscopy

MS Mass Spectrometry

NIR Near Infrared Spectroscopy

Raman Raman Spectroscopy
**Additional Information**

**Analytical Data** indicates that tables of analytical data relevant to authentication are provided.

**Alcohol Strength** relates to the measurement of alcohol strength.

**Denaturants** relates to the detection of denaturants.

**Methanol, Trace Elements etc.** relates to the measurement of a particular compound e.g. methanol or trace elements.

**Synthetic Alcohol** relates to detection of synthetic alcohol.

**Toxicology** relates to measuring compounds deemed unsafe/toxic compounds.
3 Strategies for Spirit Drink Authentication (Multiple Methods/Reviews)


Keywords: Armagnac, Brandy, Cognac, GC, HPLC


Keywords: Scotch Whisky, GC-FID, GC-MS, LC-UV, Analytical Data
http://pubs.rsc.org/en/Content/ArticleLanding/1994/AN/AN9941901741#!divAbstract


Keywords: Gin, GC-FID, Trace Elements (Ca, K, Mg), Alcohol Strength, Analytical Data


Keywords: Cognac and Brandies, Gin, Vodka, Whiskies, GC-FID, GC-MS, UV-Vis, Analytical Data


Keywords: Tequila, FTIR, IC
http://pubs.acs.org/doi/abs/10.1021/jf048637f


Keywords: Cognacs and Cognac Spirits, GC-FID, GC-MS, LC-UV, UV-VIS


Keywords: Fruit Spirits, GC-FID, GC-MS, 13C IRMS, 2H SNIF, Analytical Data
https://www.researchgate.net/publication/242160580_Assessment_of_the_Authenticity_of_Fruit_Spirits_by_Gas_Chromatography_and_Stable_Isotope_Ratio_Analyses


Keywords: Scotch Whisky, GC-FID, GC-MS, LC-UV, LC-PAD, Alcohol Strength, Analytical Data

1190-1195.  
**Keywords:** Cognac, GC-MS, HPLC-RI, Analytical Data, Glycerol, Triacetin, Sugars, Vanillin  
http://link.springer.com/article/10.1134/S1061934811120094

**Keywords:** Whisky, GC-MS, MIR, UV-Vis  

4 Methods based on GC (FID or MS)

**Keywords:** Whisky, Rum, Brandy, GC-FID, Analytical Data

**Keywords:** Brandies, Spirits, GC-FID, Analytical Data  

**Keywords:** Whisky, Rum, Brandy, GC-FID, Analytical Data  

**Keywords:** Spirits, GC-FID, Analytical Data  

**Keywords:** Whiskies, GC-MS  
http://www.sciencedirect.com/science/journal/00032670/381

**Keywords:** GC-MS, Analytical Data, Synthetic Alcohol  

**Keywords:** Whiskies, GC-MS  
http://link.springer.com/article/10.1134/S1061934807080126

5 Methods based on LC (UV, IC, MS)

JAGANATHAN, J. & DUGAR, S. M. 1999. Authentication of straight whiskey by determination of the ratio of furfural to 5-hydroxymethyl-2-furaldehyde. *Journal of AOAC International*, 82, 997-1001. **Keywords:** U.S. Straight Whiskey, LC-UV, Analytical Data

LACHENMEIER, D. W., ATTIG, R., FRANK, W. & ATHANANSAKIS, C. 2003. The use of ion chromatography to detect adulteration of vodka and rum. *European Food Research and Technology*, 218, 105-110. **Keywords:** Rum, Vodka, IC
http://link.springer.com/article/10.1007/s00217-003-0799-8


6 Methods based on Electrophoresis

http://pubs.acs.org/doi/abs/10.1021/jf202218r


7 Methods based on Mass Spectrometry (Direct Injection)

http://pubs.rsc.org/en/content/articlelanding/2005/an/b415422c#!divAbstract

*Keywords*: Whiskies, ESI-MS  

8 Stable Isotope Methods (Botanical/Geographical Origin)

*Keywords*: Spirits, 13C IRMS, Analytical Data  


*Keywords*: Spirits, Analytical Data  
http://pubs.acs.org/doi/abs/10.1021/jf00116a032?journalCode=jafcau


*Keywords*: Analytical Data  


*Keywords*: Various Spirits, 13C IRMS, 2H SNIF, Analytical Data  
http://link.springer.com/article/10.1007/s002170050111


*Keywords*: Scotch Whisky, GC-C-IRMS, Analytical Data  


*Keywords*: Brazilian Brandy, 13C IRMS  


*Keywords*: Tequila, 13C IRMS, 18O IRMS
Deliverable 5.3 Part V, Version 1, December 2016

http://pubs.acs.org/doi/abs/10.1021/jf0207777


Keywords: Tequila, 13C IRMS, 2H SNIF, GC-FID, Analytical Data


Keywords: Various Spirits, 13C IRMS, 18O IRMS, 2H SNIF, Analytical Data
https://www.researchgate.net/publication/285365882


Keywords: Scotch Whisky, GC-C-IRMS
https://www.researchgate.net/publication/285365882


Keywords: Tequila
http://pubs.acs.org/doi/abs/10.1021/jf102983v


Keywords: Scotch Whisky, 2H IRMS, 18O IRMS

9 Carbon Dating (Determination of Age)


Keywords: Brandies, LSC, Analytical Data


Keywords: AMS
https://journals.uair.arizona.edu/index.php/radiocarbon/article/download/4239/3664

Keywords: Whiskies, AMS
https://journals.uair.arizona.edu/index.php/radiocarbon/article/download/3240/pdf

10 Trace Elements


Keywords: Zivania, Analytical Data, Trace Elements
http://pubs.acs.org/doi/abs/10.1021/jf034476o


Keywords: Tequila, Analytical Data, Trace Elements

11 Nuclear Magnetic Resonance


Keywords: Spirits, NMR, Diethyl Phthalate, Ethyl Carbamate, Methanol, Polyhexamethyleneguanidine, Toxicology


Keywords: Greek Grape Marc Spirits, NMR

12 Spectroscopy


Keywords: Scotch Whisky, UV/Vis
http://pubs.rsc.org/en/Content/ArticleLanding/2004/AN/b403068k#!divAbstract


Keywords: Scotch Whisky, Alcohol Strength, NIR, Raman
*Keywords: Cognac, Spirits, MIR*  
[http://pubs.acs.org/doi/abs/10.1021/jf060465u](http://pubs.acs.org/doi/abs/10.1021/jf060465u)

*Keywords: Brandy, Rum, Vodka, Whisky, NIR, Alcohol Strength, Methanol*  

*Keywords: Scotch Whisky, Alcohol Strength, NIR, Raman*  

*Keywords: Brandies, Wine Distillates, Synchronous Fluorescence*  

*Keywords: Wine Spirits, Synchronous Fluorescence, Caramel*  

*Keywords: Tequila, UV/Vis*  

*Keywords: Scotch Whisky, MIR-ATR*  

*Keywords: Scotch Whisky, NIR/Raman, Alcohol Strength*  

*Keywords: Scotch Whisky, Raman, Alcohol Strength, Methanol, Toxicology*


*Keywords: Whiskies, UV-Vis*

http://link.springer.com/article/10.1007/s12161-014-9958-8


*Keywords: Fruit Spirits, Synchronous Fluorescence*


*Keywords: Juniper Flavoured Spirit Drinks, Synchronous Fluorescence*


*Keywords: Plum Spirit Drinks, Synchronous Fluorescence*


*Keywords: Scotch Whisky, Raman, Alcohol Strength*

http://pubs.rsc.org/en/content/articlelanding/2017/ay/c6ay02907h#!divAbstract

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13 **Review Documents on Spirit Drink Authentication**


*Keywords: Spirits*


*Keywords: Spirits*

https://www.amazon.co.uk/Food-Protected-Designation-Origin-Methodologies/dp/0444595627/ref

Keywords: Vodka