

# Developing Rapid Analysis Methods in the Spirit Drinks Sector

2016 Government Chemist Conference

Science Supporting Trust in Food

June 21<sup>st</sup> 2016

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# Aims

- To provide an overview to authentication work at the Scotch Whisky Research Institute
- To identify current capabilities and methods
- To highlight why rapid methods are important to the spirit drinks industry
- To discuss new initiatives and developments in spirit drink authentication with a particular focus on rapid methods



# Introduction to the Scotch Whisky Research Institute



# The Scotch Whisky Research Institute

- The research and technology organisation for the UK distilling industry
- Funded by the industry – member led “Research Club”
- Providing long term strategic research to the industry that benefits the industry as a whole
- Main focus on Scotch Whisky
- 26 members of staff – mainly scientific (9 PhD, 3 MSc, 7 BSc)



## 40 Years of Research

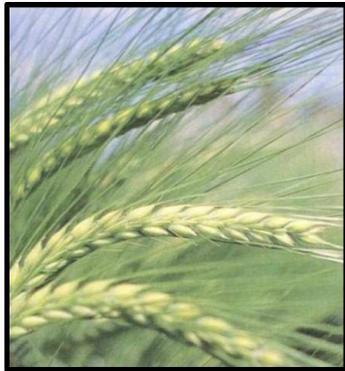
A reflection on the evolution of the Scotch Whisky Research Institute

Delivering innovation and value through the application of science



# The Scotch Whisky Research Institute

- Transfer our knowledge back to our members helping them to improve their businesses
- Unique expertise, experience and stored information on areas of research
- Raw Materials & Processing, Maturation, Product Protection and Flavour



# Our Members

*Beam* **SUNTORY**

  
**INVER HOUSE**  
DISTILLERS

**GORDON & MACPHAIL**  
— ESTABLISHED 1895 —  


**Burn Stewart**  
DISTILLERS LTD

**IAN  
MACLEOD**  
  
DISTILLERS

**DIAGEO**

  
THE  
**EDRINGTON**  
GROUP

  
**MBD**  
MORRISON BOWMORE DISTILLERS

**Dewar's**<sup>®</sup>

  
THE NORTH BRITISH DISTILLERY  
ESTD 1885  
THE COMPANY LTD

  
**Chivas Brothers**  
Pernod Ricard

*The*  
**GLENMORANGIE**  
COMPANY

**GLENGRANT**<sup>®</sup>

  
**WILLIAM GRANT & SONS**  
INDEPENDENT FAMILY DISTILLERS SINCE 1887



## Our Members



<b>Beam Suntory UK</b>	- <i>Japan (Beam Suntory)</i>	<b>Gordon &amp; MacPhail</b>	- <i>Scotland</i>
<b>Burn Stewart</b>	- <i>South Africa (Distell)</i>	<b>Inver House</b>	- <i>Thailand (Pacific Spirits)</i>
<b>Chivas Brothers</b>	- <i>France (Pernod Ricard)</i>	<b>John Dewar &amp; Sons</b>	- <i>Bermuda (Bacardi)</i>
<b>Diageo Scotland</b>	- <i>UK (Diageo)</i>	<b>North British</b>	- <i>Scotland</i>
<b>Edrington Group</b>	- <i>Scotland</i>	<b>Wm Grant &amp; Sons</b>	- <i>Scotland</i>
<b>Glen Grant</b>	- <i>Italy (Campari)</i>	<b>Ian MacLeod Distillers</b>	- <i>Scotland</i>
<b>Glenmorangie</b>	- <i>France (LVMH)</i>		



# The Authentication of Spirit Drinks

## An Overview



# The Authentication of Spirit Drinks: An Overview

## Some Impressive Facts and Figures

- In 2014, the equivalent of 1.19bn bottles of Scotch Whisky was shipped worldwide.
- This equated to £3.95 billion for the UK balance of trade and accounts *over a quarter of all UK food and drink exports*.

(source: Scotch Whisky Association)



# The Authentication of Spirit Drinks: An Overview

## Some Impressive Facts and Figures

- The spirit drinks sector is the most valuable European agri-food export sector.
- €21.4bn generated through excise duty and VAT.
- €10bn in exports, representing a trade surplus close to €9bn.
- 1,000,000 jobs in production and sales.

(source: spiritsEUROPE)

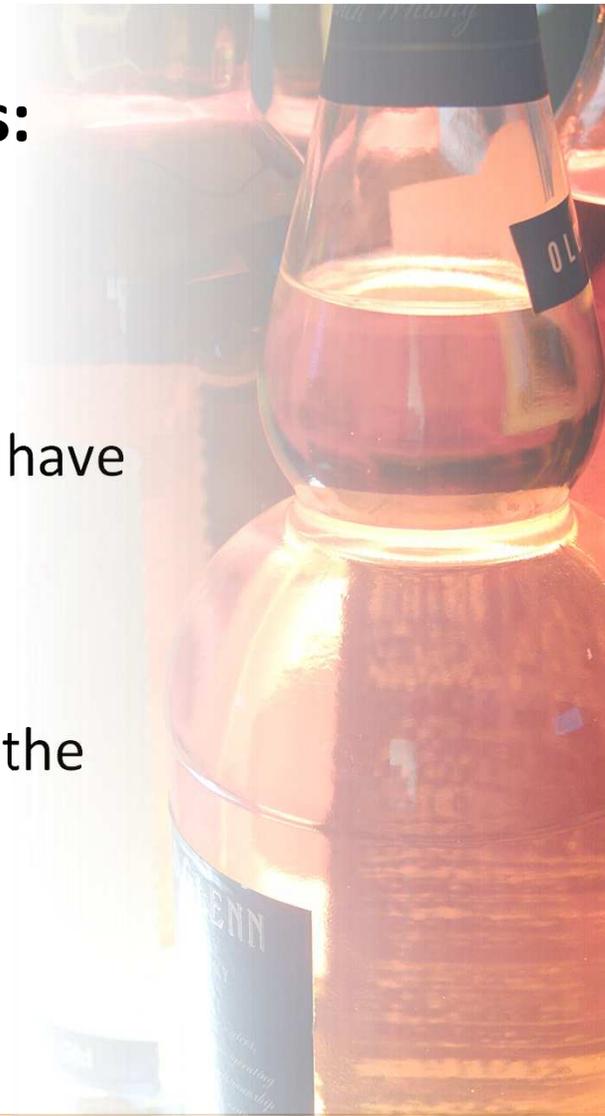


# The Authentication of Spirit Drinks: An Overview

## Some Less Impressive Facts and Figures

- In the UK, the level of counterfeit alcohol is reported to have increased by almost 400% between 2009 and 2012.
- A quarter of the products sold in China are fakes.
- In Bulgaria, illicit spirits are believed to constitute more the 50% of domestic spirit consumption.

(source: spiritsEUROPE)



# The Authentication of Spirit Drinks: An Overview

## Some Less Impressive Facts and Figures

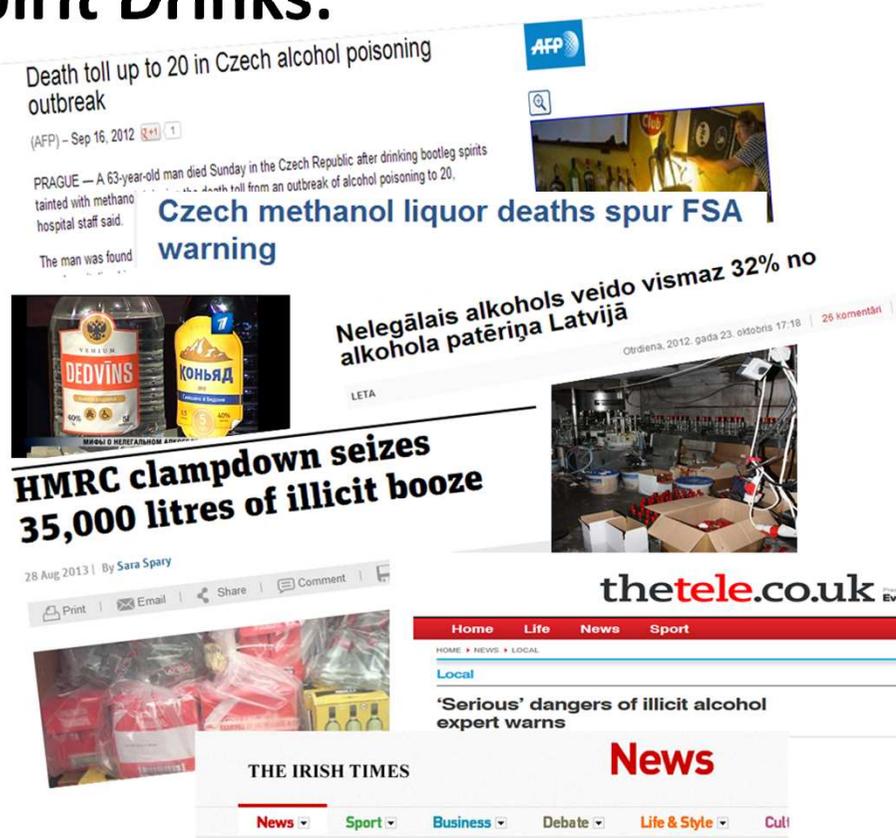
- Direct loss of sales
- Damage to reputation
  - *Counterfeit goods will be of inferior quality*
  - *Can lead to indirect loss of sales*
- Long term damage
  - *Devaluation of spirit brands and categories*
  - *Concerns over safety*



# The Authentication of Spirit Drinks: An Overview

## Some Less Impressive Facts and Figures

- Illicit alcohol production and counterfeit spirit drinks can also pose a serious risk to health.
- In 2012, 45 people died and dozens more suffered serious illness in the Czech Republic, the Slovak Republic and Poland after drinking “vodka” or “rum” containing methanol.



# The Authentication of Spirit Drinks: An Overview

## Brand Counterfeit

- Trading on the premium quality associated with a particular brand of spirit drink
- Direct copying of packaging and filling with non-authentic liquid
- Collection of authentic used packaging and refilling



# The Authentication of Spirit Drinks: An Overview

## Generic Counterfeit

- Trading on the premium quality associated with a category of spirit drink, e.g. Scotch Whisky, Cognac or Vodka.



# Current Capabilities and Methods for Generic Authenticity



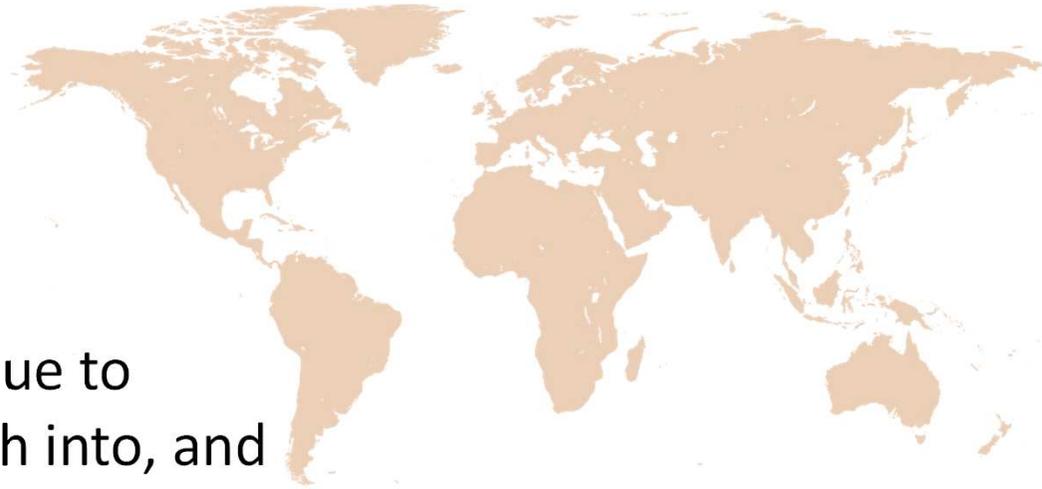
# Current Capabilities and Methods for Generic Authenticity

- The research undertaken by the Institute for generic authentication informs our provision of expertise in analysing suspect Scotch Whisky products.
- Suspect product analysis is principally undertaken on behalf of the industry's trade organisation, the Scotch Whisky Association (SWA).



# Current Capabilities and Methods for Generic Authenticity

- Every year we analyse around 100 suspect whisky samples, sourced by the SWA from markets worldwide.
- The Institute can provide this service due to its analytical expertise and our research into, and knowledge of, the composition an authentic product.
- In 2014, 71% of these products suspected as being counterfeit failed to conform to label claims.



# Current Capabilities and Methods for Generic Authenticity

- Our analytical reports are used to support prosecutions against counterfeiters worldwide.
- They help restrain the sale of products misleadingly described or presented as Scotch Whisky.
- Our authenticity methods are accredited to ISO Standard 17025 by the United Kingdom Accreditation Service – legal recognition.



# Current Capabilities and Methods for Generic Authenticity

## Common modes of counterfeiting

- Extension or replacement of authentic product with:
  - Water
  - Cheaper locally produced spirit or neutral alcohol
  - Addition of sweetening or flavourings to mask or mimic aromas



# Current Capabilities and Methods for Generic Authenticity

## Whisky Authentication Strategy – Examples

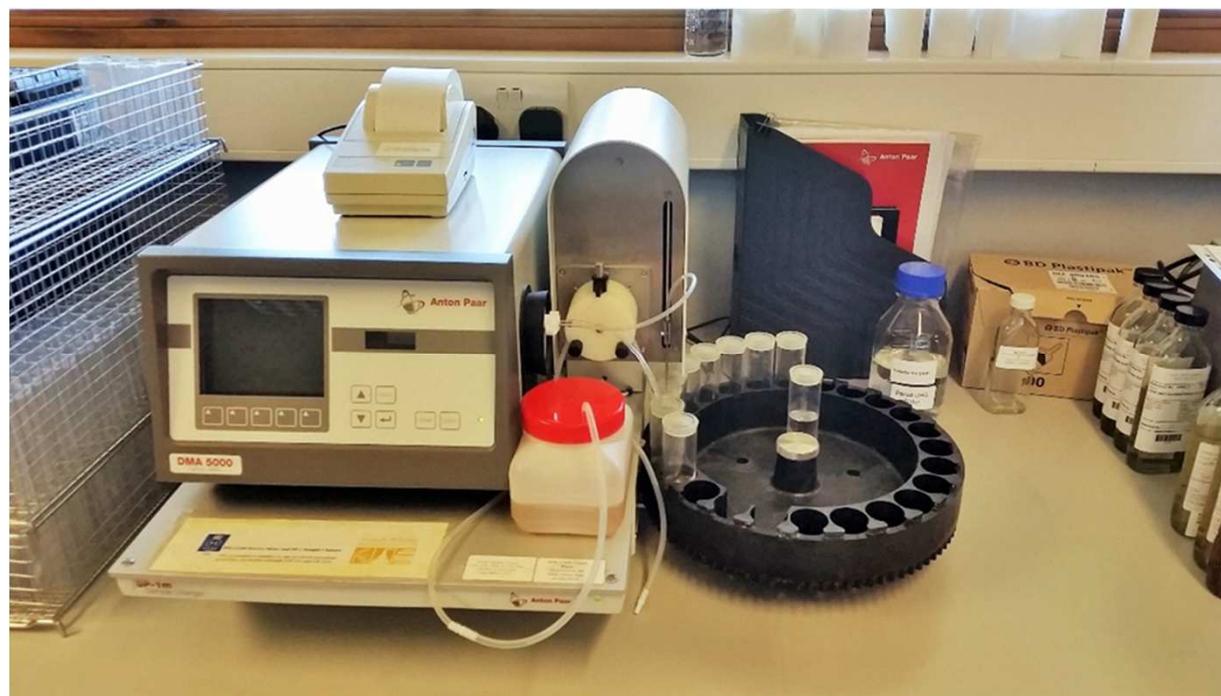
- Lower than 40%
- Addition of flavourings/sweetening
- Inconsistent with cereal spirit
- Inconsistent with distillation <94.8%
- Inconsistent with maturation in oak casks for a minimum period of 3 years



# The Authentication of Scotch Whisky: An Overview

## Authentication Strategy – Alcohol strength lower than minimum 40%

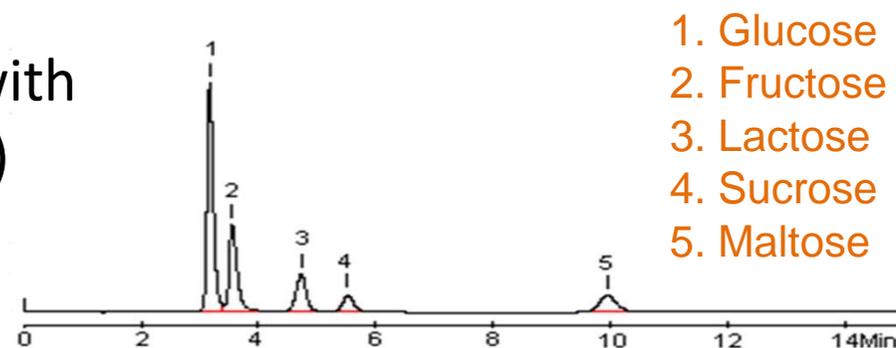
- Quickly identifies a product as being a counterfeit Scotch Whisky.
- Range of techniques: Institute use distillation and densitometry as authoritative method.



# Current Capabilities and Methods for Generic Authenticity

## Authentication Strategy - Addition of sweetening

- No additives allowed - only water and caramel
- We can detect the addition of unlawful sweetening (sugars) in counterfeits by analysis
- High performance liquid chromatography with pulsed amperometric detection (HPLC-PAD)



# Current Capabilities and Methods for Generic Authenticity

## Authentication Strategy - Addition of flavouring

- Using SPME-GC/MS, flavouring compounds not found in Whisky may be detected

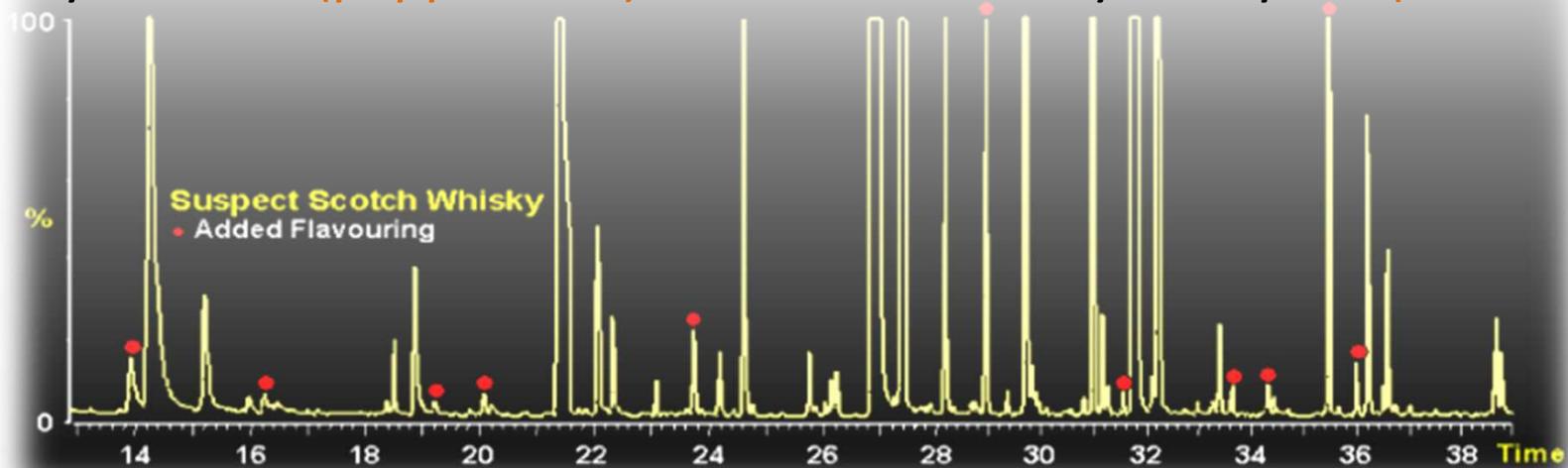
$\gamma$ -terpinene (*citrus/herbal*)

menthone (*mint*)

trans-anethole (*aniseed*)

menthyl acetate (*peppermint*)

ethyl salicylate (*wintergreen*)



# Current Capabilities and Methods for Generic Authenticity

## Authentication Strategy - Inconsistent with Cereal Spirit

- The concentration of certain compounds may indicate that the alcohol in the suspect product has not been made from cereals and therefore cannot be whisky.
- For example, high levels of methanol are associated with fruit substrates.
- Measured using GC-FID.



# Current Capabilities and Methods for Generic Authenticity

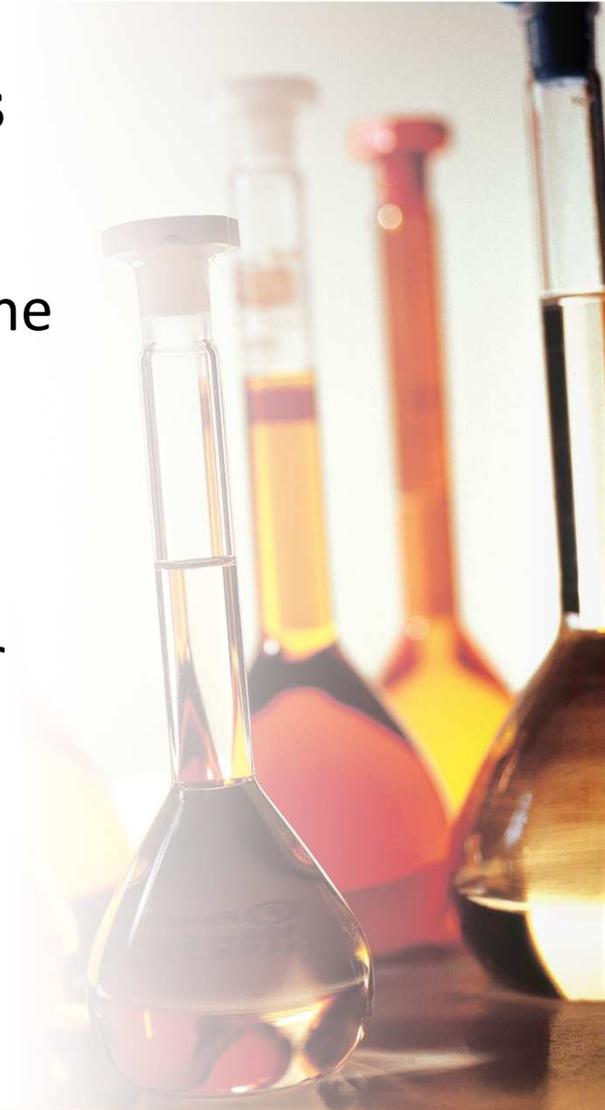
## Authentication Strategy – Inconsistent with authentic maturation

- The requirement for Scotch Whisky to be placed in oak casks for a minimum period of 3 years creates a specific and consistent profile of certain compounds in the whisky.
- Maturation Related Congeners – compounds extracted from the cask over time.
- HPLC with UV detection.



## Current Capabilities and Methods for Generic Authenticity

- These standard, informative tests used widely across the spirit drinks industry
- For example, Cognac's equivalent of SWRI, the BNIC's Station Viticole, authenticates Cognac based on similar analyses conducted over the last 40 years



# Current Capabilities and Methods for Brand Authenticity



# Current Capabilities and Methods for Brand Authenticity

## What are the modes of brand counterfeiting?

- On-premise substitution where a lesser brand is substituted for the more expensive brand ordered by the consumer.
- Re-filling and re-closing authentic, branded products, typically with some form of unrecorded alcohol.
- Completely fake reproduction of a legitimate brand, such as when the bottle and all packaging are fake and the liquid is not authentic.



# Current Capabilities and Methods for Brand Authenticity

- Typically, the same laboratory methods of analysis will be employed as for generic work.
- Some additional confirmatory analyses can be undertaken which are problematic for generic work, e.g. trace metal analysis.



# New Initiatives and Developments in Spirit Authentication

## The FoodIntegrity Project



# The FoodIntegrity Project



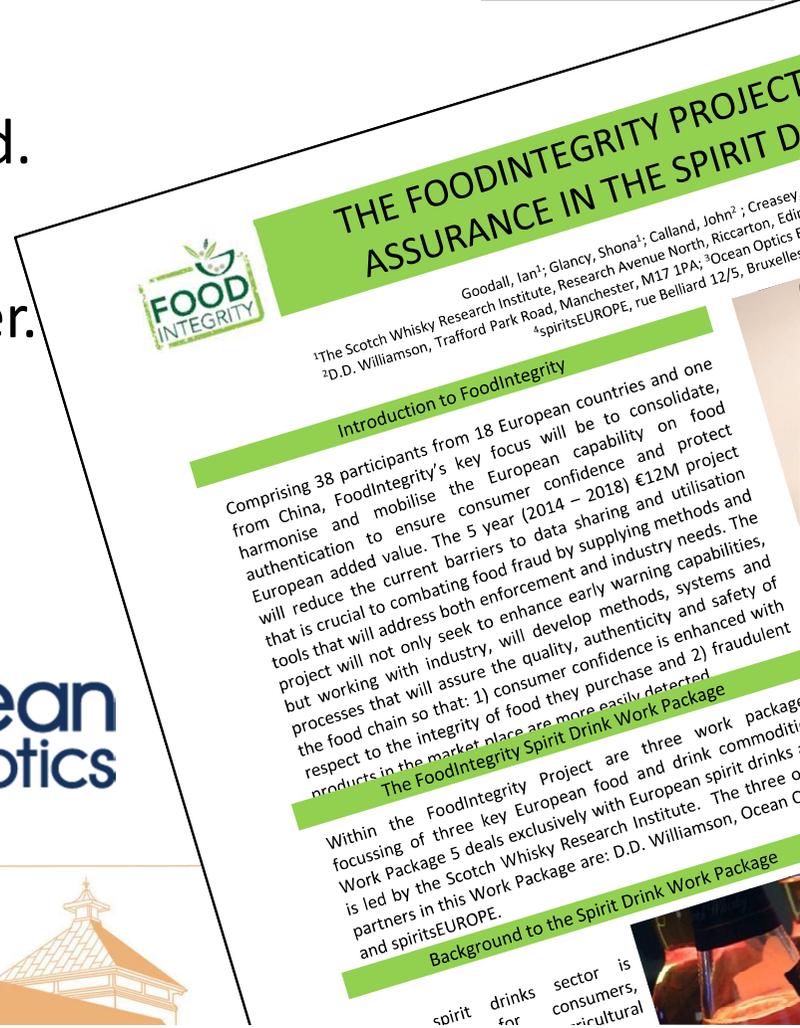
- European Union funded collaborative project under FP7 “Assuring Quality and Authenticity in the Food Chain”
- 9 million Euros over 5 year project term
- SWRI leading Spirit Drinks Work Package focussing on the safety, authenticity and quality of European Spirit Drinks.



# The FoodIntegrity Project



- Three of the Work Packages focussed on commodities: olive oil, spirit drinks and seafood.
- Four partners in the consortium within the Spirit Drinks Work Package; SWRI is lead partner.
- Many more stakeholders involved (spirit companies, technology providers, universities etc.)



# The FoodIntegrity Project



- The aim is to target the current gaps in the protection of the spirit drink sector's brands/categories from inferior and/or harmful counterfeits
- There is a desire for improved rapid, easy to use, portable analysis methods: definitive, non-intrusive, wider scope.
- Also, improved supporting lab based methods (speed, level of information, sensitivity)



# The FoodIntegrity Project



Detection of markers for illicit alcohol used in brand and generic counterfeits.

- Denaturants (e.g. methanol, IPA, MEK, denatonium benzoate) signifying abuse of excise exempt alcohol

## Brand or Category Authentication

- Ability to distinguish more sophisticated counterfeits (e.g. brand substitution)
- The extension of current techniques via the combination of complementary techniques
  - e.g. simultaneous UV/VIS profile with alcohol strength measurement by Raman/NIR



## THE FOODINTEGRITY PROJECT ASSURANCE IN THE SPIRIT D

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<sup>2</sup>D.D. Williamson, Trafford Park Road, Manchester, M17 1PA; <sup>3</sup>Ocean Optics  
<sup>4</sup>spiritsEUROPE, rue Belliard 12/5, Bruxelles

### Introduction to FoodIntegrity

Comprising 38 participants from 18 European countries and one from China, FoodIntegrity's key focus will be to consolidate, harmonise and mobilise the European capability on food authentication to ensure consumer confidence and protect European added value. The 5 year (2014 – 2018) €12M project will reduce the current barriers to data sharing and utilisation that is crucial to combating food fraud by supplying methods and tools that will address both enforcement and industry needs, but working with industry, will develop methods, systems and processes that will assure the quality, authenticity and safety of the food chain so that: 1) consumer confidence is enhanced with respect to the integrity of food they purchase and 2) fraudulent products in the market place are more easily detected.

### The FoodIntegrity Spirit Drink Work Package

Within the FoodIntegrity Project are three work packages focussing on three key European food and drink commodities. Work Package 5 deals exclusively with European spirit drinks and is led by the Scotch Whisky Research Institute. The three co-partners in this Work Package are: D.D. Williamson, Ocean C and spiritsEUROPE.

### Background to the Spirit Drink Work Package

spirit drinks sector is for consumers, agricultural



# The FoodIntegrity Project



Keen to involve more participants from other laboratories and regulatory bodies within the network.

Currently conducting research looking at:

- collating analyses being undertaken in spirit drink authentication and associated best practice
- options for provision of training tools - presentations, collections of relevant papers, guidance documents and workshops
- better communication routes between industry and third party laboratories - exploration of data sharing mechanisms



# New Initiatives and Developments in Spirit Authentication

## Rapid Methods



# Developing Rapid Methods for the Spirit Drinks Sector

- The Spirit Drinks Work Package of the Food Integrity Project has targeted rapid methods as one of its primary aims
- What do we mean by ‘rapid methods’?
  - Methods that can be readily employed in the field, such as portable devices, to identify suspect products at the point of sale or distribution
  - Quicker authoritative methods in the laboratory that complement in field techniques



# Developing Rapid Methods for the Spirit Drinks Sector

## Spirits Industry Perspectives on Rapid Methods

- Agile & portable solutions that are cheaply and easily moved through customs at airports
- Cost effective in-field screening providing quick answers that can be backed up by laboratory techniques
- Ease of use and output suitable for untrained operators
- Methods need to offer measureable improvements over what is currently available



# Developing Rapid Methods for the Spirit Drinks Sector

## Research Approach: Three levels of technology solution

- Level 1: Portable (e.g. handheld/backpack)
  - *Quick, relatively low cost, screening*
- Level 2: Deployable (e.g. back of car)
  - *More authoritative, e.g. LC/MS & GC/MS, benefitting from miniaturisation. Often as rapid. Issues – expense and ease of use.*
- Level 3: Supplemental Lab Based
  - *Improvements in standard methods (e.g. maturation compounds) and exploration of new techniques and identification of new markers*



# Developing Rapid Methods for the Spirit Drinks Sector

## Rapid Methods – Ocean Optics Spirit Sampler

- The Ocean Optics Spirit Sampler was developed for brand authentication
- This technique is based on UV-VIS spectral information.
- Brand UV-Vis spectra are modelled and suspect samples can quickly be screened in the field.



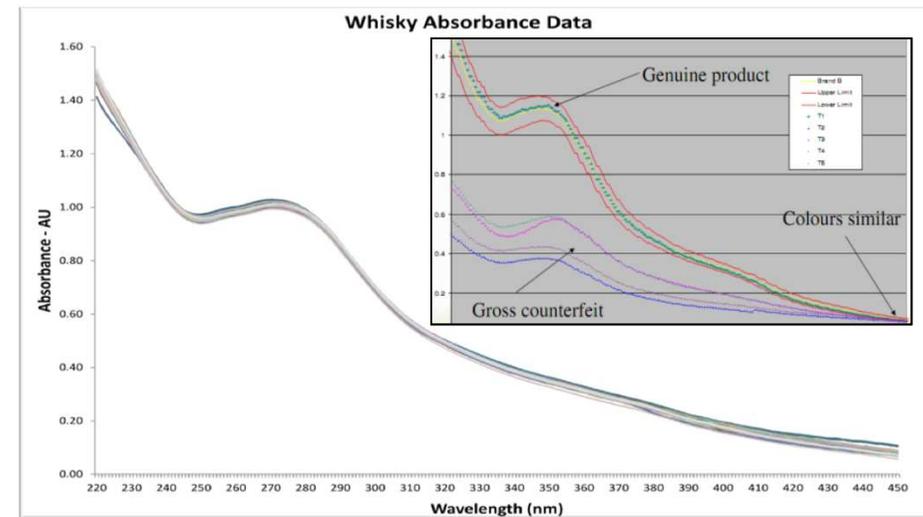
The Spirit Sampler: Ocean Optics



# Developing Rapid Methods for the Spirit Drinks Sector

## Rapid Methods – Ocean Optics Spirit Sampler

- Very good at identifying gross counterfeits, used as a screening tool
- Liked by industry, harder to create an “authentic” profile, no analytical data revealed
- Less able to differentiate more sophisticated counterfeits or brand substitution
- Easy to use; provides simple PASS/FAIL response



# Developing Rapid Methods for the Spirit Drinks Sector

## Rapid Methods – PerkinElmer Torion T-9

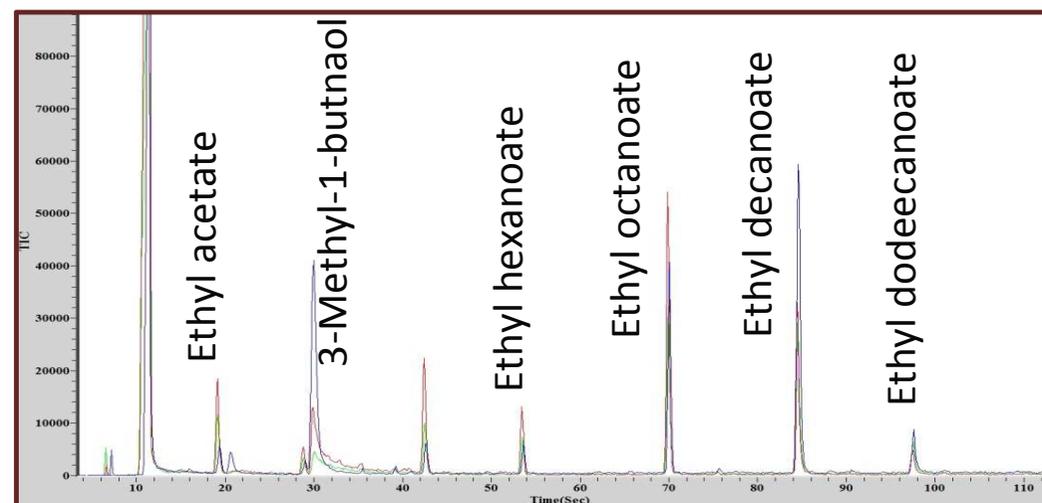
- Self-contained, ruggedized, field-portable GC/MS
- Key points - simple robust hardware, with easy-to-use software, sample preparation and introduction techniques
- Proved concept for denaturants - ability to identify six common denaturants in spirits at levels of interest



# Developing Rapid Methods for the Spirit Drinks Sector

## Rapid Methods – PerkinElmer Torion T-9

- Many of the analytes detected in spirits using this technology (without optimisation) are routinely employed in laboratory based spirit profiling
- Possible scope to investigate brand authentication



# Developing Rapid Methods for the Spirit Drinks Sector

## Rapid Methods – RIDA®CUBE Scan Analyser

- New and fully automated photometric analyser that can test for the presence of sugars (Glucose, Fructose and Sucrose)
- The unit is portable and fully automated allowing for rapid analysis by untrained users
- Ability to quickly identify products that have been illegally adulterated with sugars



# Developing Rapid Methods for the Spirit Drinks Sector

## Rapid Methods – Microsaic 4000MiD®

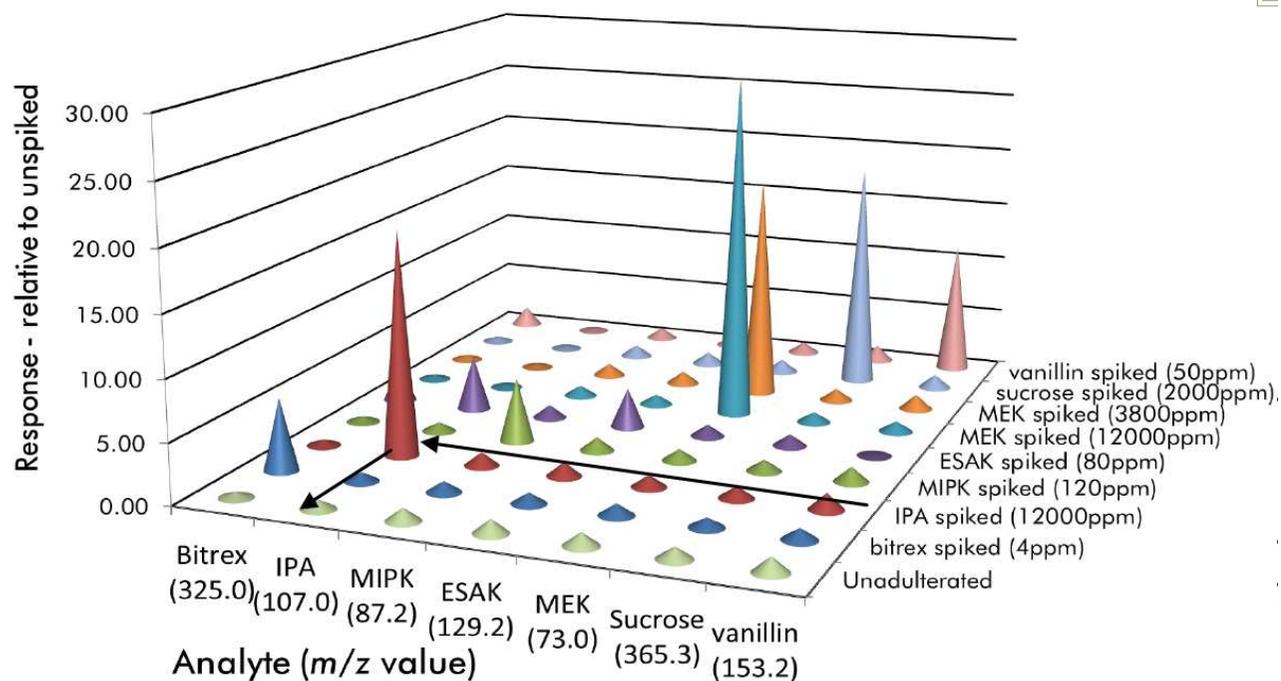
- Deployable no separation ESI-MS
- The unit is a self-contained, with no external vacuum pumps and integrated PC
- Simple interface allowing non-mass spectrometrists to operate and maintain the equipment with minimal effort and training



# Developing Rapid Methods for the Spirit Drinks Sector

## Rapid Methods – Microsaic 4000MiD<sup>®</sup>

- Proved successful identification of a range of denaturants and adulterants spiked into whisky
- Brand separation has also been investigated and is showing some potential



# Developing Rapid Methods for the Spirit Drinks Sector

- Dipstick type devices have also been investigated
- Dipsticks can be specific to certain trace level additions of ingredients (where the spirit drink definition permits)
- Portable conductivity, pH meters, alcohol strength meters can be used to identify non-compliant products, e.g. vodka made with non-deionized water



# Developing Rapid Methods for the Spirit Drinks Sector

## Conclusions and Future Challenges

- Still many challenges facing spirit drink authentication - no 'one tool fits all'
- Generic authentication is much more challenging – less potential solutions compared to brand authentication
- Rapid methods offer many advantages
  - *Fast, portable, in-field, unskilled user, screening tool*
- With knowledge of spirit composition and production these techniques offer potential solutions to combat counterfeiting



Thank you for your attention

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To join the Food Integrity Network go to:  
<http://foodintegrity.eu>

Register and email me to gain access to  
Spirit Drinks work.

