

# GC conference 24-25 November 2014



Beating the cheats: Quality, safety and authenticity in the food chain

**Science**  
for a safer world



# Introduction



- **Food fraud and counterfeit products**
- **Alcoholic beverages**
- **Scotch Whisky analysis and DAPS PT**
- **Horsemeat testing in the QMAS scheme**
- **Questions**

# Food fraud



- The food and drink industry is globally worth trillions of pounds annually
- 'Value' can be added to products at a number of different stages of a product's life
- An increasing number of cases of fraud and counterfeit products are being reported
- Links have been made to organised criminal gangs
- Increasingly sophisticated methods of adulteration required equally sophisticated methods of analysis and detection

# Proficiency Testing



- Independent assessment of laboratory performance
- Tests routine methods
- Tests the entire analysis process
- Can be carried out frequently
- Compares performance of analytical methods
- Compares performance of peer laboratories
- Tests qualitative and quantitative performance of laboratories



# Alcoholic beverages

- Spiritous alcoholic beverages are analysed for a wide range of measurands using a number of different techniques
- Density meters, pycnometry
  - Density, Apparent strength, Actual strength
- Gas Chromatography
  - Volatile congeners, higher alcohol congeners, maturation congeners, contaminants
- HPLC
  - Trace sugars, maturation congeners
- Other specific methods
  - Haze, colour, acidity

# Whisky and the DAPS PT scheme



- What is special about Scotch Whisky?
- To be sold as Scotch Whisky
  - Distilled in Scotland from water and malted barley
  - Distilled at an alcoholic strength of less than 94.8%
  - Matured only in Oak casks
  - Matured only in Scotland
  - Matured for not less than three years
  - .....
  - To which no substance has been added (with exceptions)
  - That has a minimum alcoholic strength by volume of 40%

# Whisky and the DAPS PT scheme



- How are specific methods applied to Scotch Whisky analysis?
- ABV
  - Detection of adulterated or out of specification samples
- Volatile and higher alcohol congeners
  - Methanol content, presence of higher alcohol congeners, ratio of 2- and 3-methylbutanol to isobutyl alcohol
- Maturation congeners
  - Concentration of vanillin, ellagic acid, syringaldehyde etc.
- Sugars
  - Concentration of sugars, presence of sucrose

# The DAPS PT scheme



- Scheme structure
  - Routine samples
    - Including whisky, vodka, gin, wine, cider, liqueur, ready-to-drink
  - Additional wort and simulant samples
  - Occasional spike samples for less commonly 'present' analytes NDMA and Ethyl carbamate
- Regular rounds
  - Four times per year
- Sample matrices
  - Covers all common spirits, cider, liquors
  - Premium and economy brands



# The DAPS PT scheme



- PT performance is assessed using z scores
  - Calculated as the difference between participant results and the 'true' value, divided by a 'fit-for-purpose' standard deviation for proficiency assessment (SDPA)
- SDPA based on:
  - Historical data
  - Expert assessment of DAPS advisory group
  - Method capabilities
  - A combination of the above
- Satisfactory performance
  - =  $|z \text{ score}| \leq 2.00$

# The DAPS PT scheme: Report



Sample: B1 - Scotch whisky

Analyte: Actual Alcoholic Strength

Lab ID	Method	Result (% ABV)	z' score*
DP0004	Other	40.24	0.94
DP0006	Density Meter	39.55	-7.14
DP0007	NIR/Alcolyser	40.20	0.47
DP0008	Density Meter	40.25	1.05
DP0014	Density Meter	40.16	0.00
DP0014	NIR/Alcolyser	40.27	1.29
DP0019	NIR/Alcolyser	40.17	0.12
DP0020	NIR/Alcolyser	40.16	0.00
DP0021	NIR/Alcolyser	40.17	0.12
DP0023	NIR/Alcolyser	40.26	1.17
DP0024	NIR/Alcolyser	40.18	0.23
DP0027	Density Meter	40.18	0.23
DP0028	Density Meter		
DP0036	Density Meter		
DP0042	Density Meter		
DP0048	Other		
DP0058	NIR/Alcolyser		
DP0060	Density Meter		
DP0062	Other		
DP0071	Density Meter		
DP0078	Density Meter		

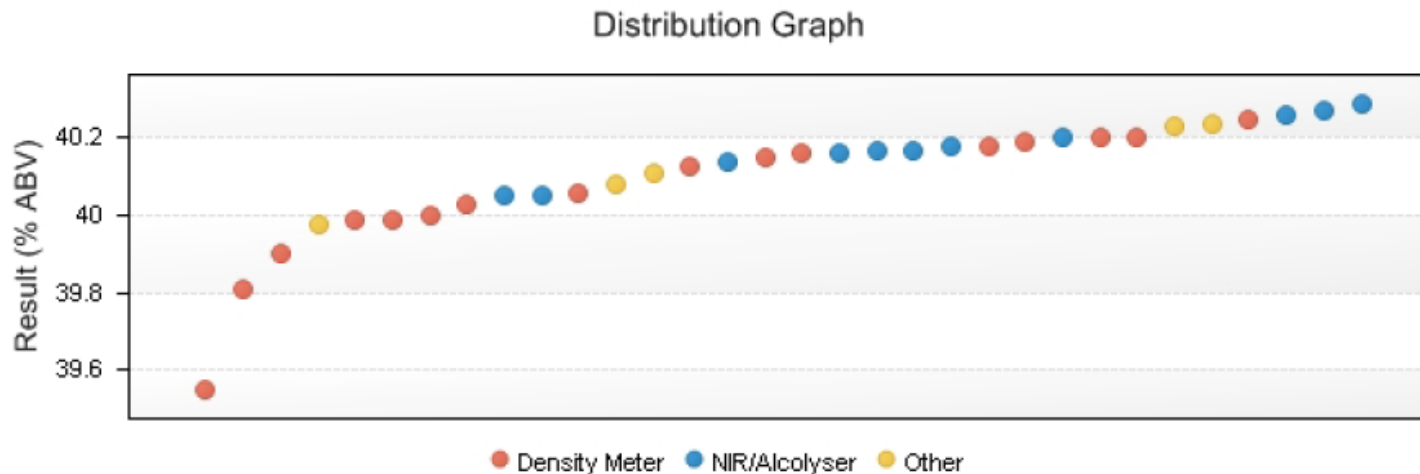
## Performance Statistics

	Value
Assigned Value	40.16 % ABV
Uncertainty of Assigned Value	0.03 % ABV
SDPA	0.085 % ABV
Satisfactory Range	39.99 to 40.33 % ABV
Satisfactory z' scores	87.5%
Questionable z' scores	3.1%
Unsatisfactory z' scores	9.4%

## Data Statistics

	Value
Number of Results	32
Number of Excluded Results	1
Mean	40.12 % ABV
Median	40.16 % ABV
Standard Deviation	0.114 % ABV
Robust Standard Deviation	0.119 % ABV
Result Range	39.81 to 40.29 % ABV

# Method comparison

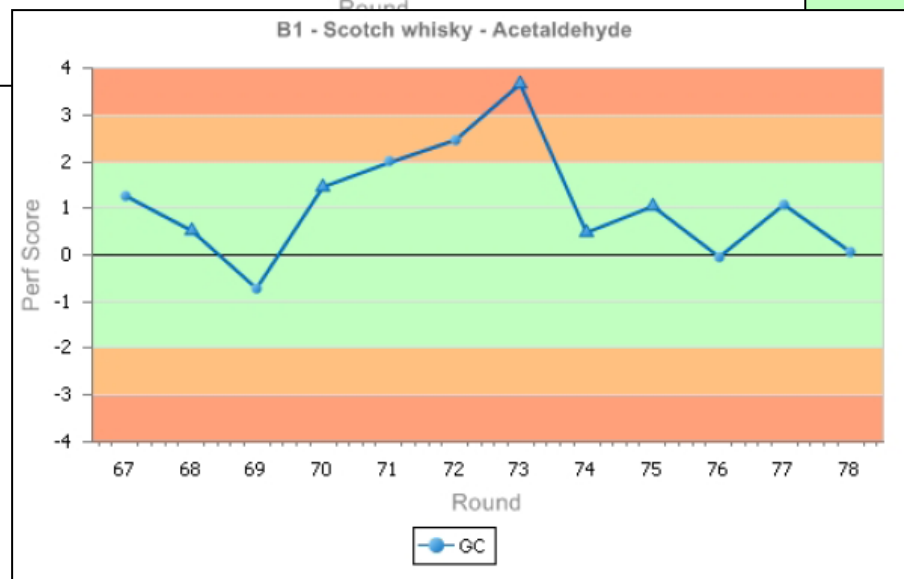
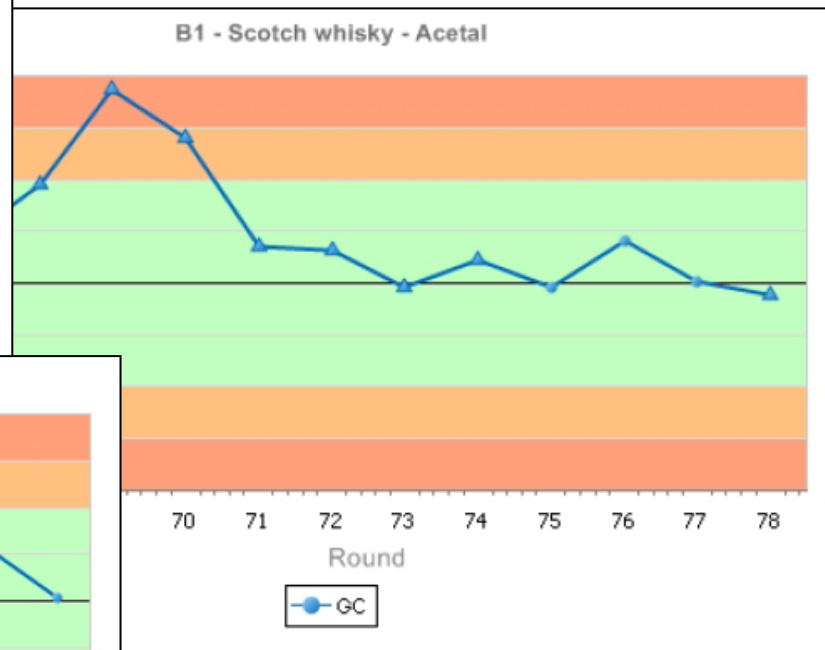
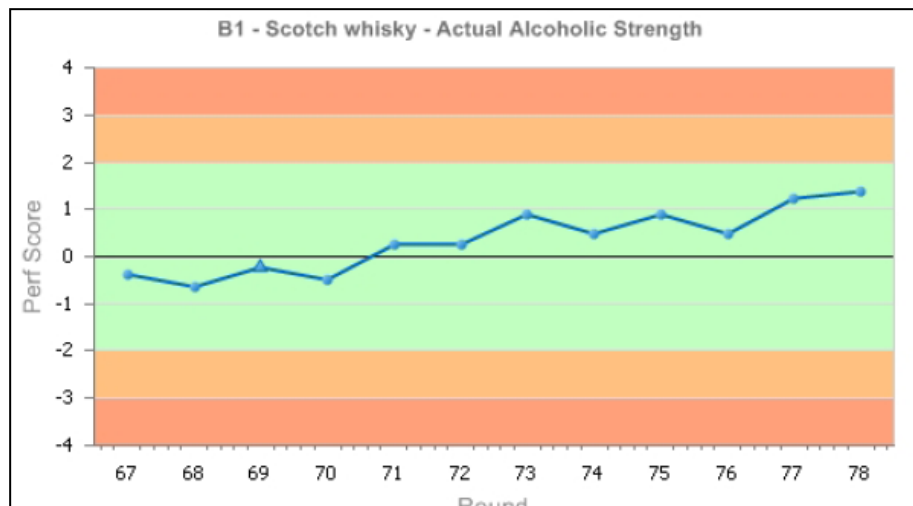


## Methodology Summary

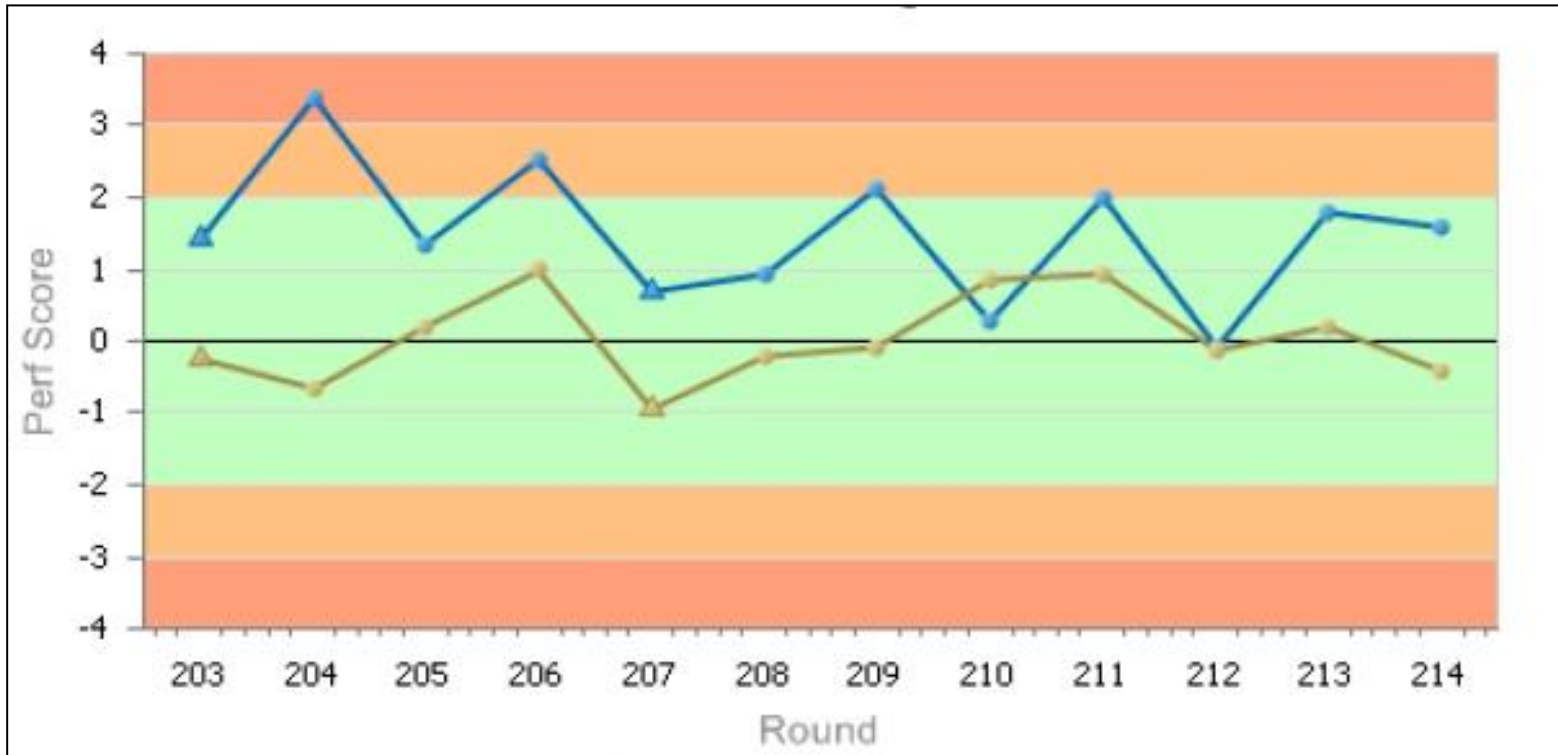
Method	Number of Results	Excluded Results	% of Total	Median	Robust SD	Range	Sat.
				% ABV			%
Density Meter	16	1	50	40.13	0.104	39.81 to 40.25	81.3
NIR/Alcolyser	11	0	34.38	40.17	0.044	40.05 to 40.29	100.0
Other	5	0	15.63	40.11	0.178	39.98 to 40.24	80.0
All	32	1	100	40.16	0.119	39.81 to 40.29	87.5

- Method statistics for each analytical method used
- Methods can be assessed individually if required

# Long term performance



# Analyst performance



- As many as 10 individual analysts can be assessed
- Comparisons can be made across rounds and analytes

# Horsemeat project



- Issue came to light 15<sup>th</sup> January 2013
- Major breakdown in the traceability of the food supply chain
- Positive samples were reported to be found in Tesco, Asda, Lidl, Aldi and Iceland
- As many as 8 different companies implicated, from France, Luxembourg, Switzerland, Lithuania and the Netherlands
- LGC leading in analysis, method validation and RM production
- LGC Bury produced PT samples in March 2013

# Sample structure

- Samples were included as part of the QMAS, meat and fish PT scheme
- Qualitative and quantitative scheme
- Various matrices used of the course of a scheme year
- Six samples provided each round
- ‘Contaminant’ either present or absent in each sample
- Samples may be ‘contaminated’ with a single stated product or with one of a number of possible products
- ‘Contaminant’ included at concentration levels from 1 to 10% (w/w)
- Additional quantification for ‘present’ samples

# Sample production



- Samples produced at LGC Bury
- Samples of known provenance obtained for meat and fish
- PCR confirmation is carried out by LGC Teddington
- Each meat type or fish species has individual equipment, where blending is required
- External surfaces are removed
- Individual 'components' are added gravimetrically
- Samples may be provided raw or cooked
- The sample **MUST** be analysed in its entirety



# Authenticity performance



Sample: 749 - Determination of equine in bovine

Analyte: Sample 1 Presence/Absence

Lab ID	Method	Result
MT4040	PCR	Absent
MT4093	PCR	Absent
MT4287	PCR	Absent
MT4424	ELISA	Absent
MT4607	PCR	Absent
MT4630	PCR	Present
MT4733	PCR	Absent
MT4751	ELISA	Absent
MT4821	ELISA	Absent
MT4873	PCR	Absent
MT4882	PCR	Absent
MT4937	PCR	Absent
MT4996	PCR	Absent
MT5136	PCR	Absent
MT5181	ELISA	Absent
MT5182	PCR	Absent
MT5407	ELISA	Present
MT5442	PCR	Absent

Sample: 749 - Determination of equine in bovine

Analyte: Sample 1 Quantification

Lab ID	Method	Result (%)	z score
MT4093	PCR	<0.05	
MT4287	PCR	0.00	
MT4996	PCR	<1.00	
MT5452	PCR	<0.10	
MT5474	Other	0.00	
MT5475	PCR	<1.00	
MT5477	PCR	0.00	
MT5478	ELISA	0.00	
MT5478	PCR	0.00	
MT5485	PCR	0.00	
MT5486	PCR	0.00	
MT5491	PCR	0.00	
MT5495	PCR	<0.10	

Due to the low number of results returned, performance scores are shown for information purposes only

# Authenticity performance



Sample: 749 - Determination of equine in bovine

Analyte: Sample 3 Presence/Absence

Lab ID	Method	Result
MT4040	PCR	Present
MT4093	PCR	Present
MT4287	PCR	Absent
MT4424	ELISA	Present
MT4607	PCR	Present
MT4630	PCR	Absent
MT4733	PCR	Present
MT4751	ELISA	Present
MT4821	ELISA	Present
MT4873	PCR	Present
MT4882	PCR	Present
MT4937	PCR	Present
MT4996	PCR	Present
MT5136	PCR	Present
MT5181	ELISA	Present
MT5182	PCR	Present

## Data Statistics

	Result
Assigned Value	Present
Number of Results	44
Satisfactory	89%

## Methodology Summary

	Result
PCR	91%
ELISA	89%
Other	0%

# Authenticity performance



Sample: 749 - Determination of equine in bovine

Analyte: Sample 3 Quantification

Lab ID	Method	Result (%)	z' score*
MT4093	PCR	2.00	0.82
MT4287	PCR	<0.10	
MT4873	PCR	0.10	-0.96
MT4996	PCR	2.30	1.11
MT5452	PCR	2.00	0.82
MT5474	Other	0.00	
MT5475	PCR	1.20	0.07
MT5477	PCR	1.00	-0.11
MT5478	ELISA	0.50	-0.58
MT5478	PCR	0.39	0.68
MT5485	PCR	<1.00	
MT5486	PCR	1.03	
MT5491	PCR	<1.00	
MT5495	PCR	1.30	

## Data Statistics

	Value
Number of Results	14
Number of Excluded Results	4
Mean	1.18 %
Median	1.12 %
Standard Deviation	0.740 %
Robust Standard Deviation	0.994 %
Result Range	0.00 to 2.30 %

# Thanks to



- Chemistry Team LGC Bury
- Robert Fotheringham, Chivas Brothers



**Any questions!**