

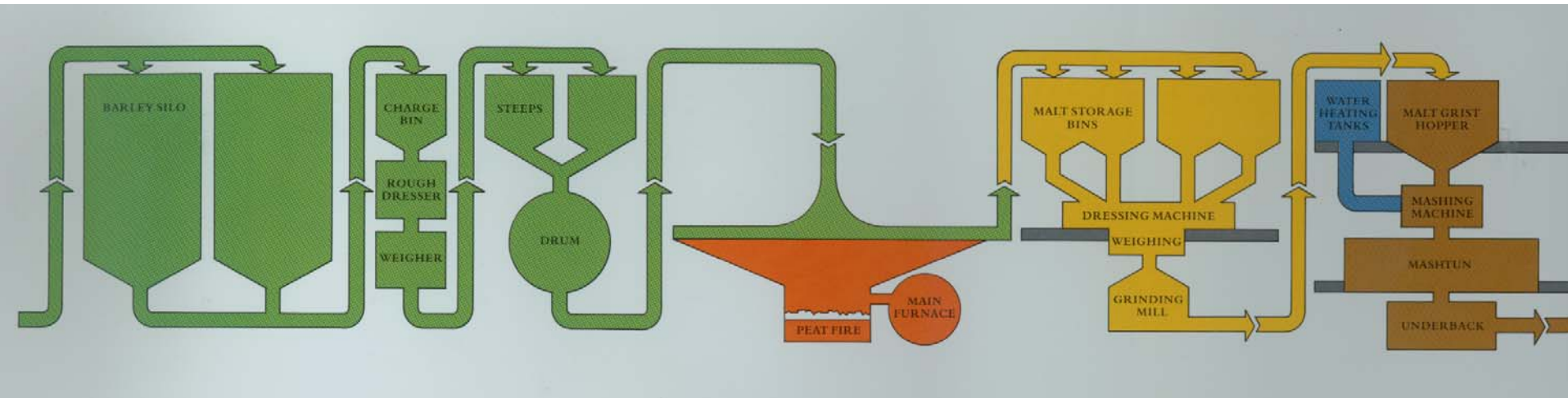


Underpinning quality in the distilling industry through participation in Proficiency Testing

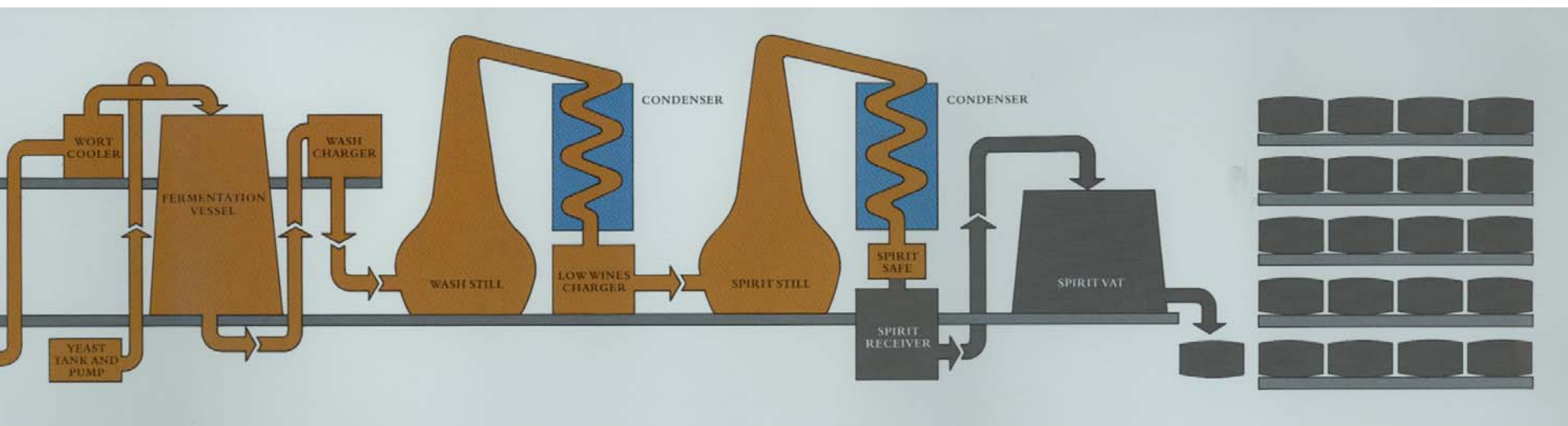
Paul Lockyer – Diageo Global Supply

Thursday 29th April

Making Malt Whisky – Part 1 :



Making Malt Whisky – Part 2 :



Making Scotch Malt Whisky

Generally referred to in five stages :

- Malting
 - To convert the barley to malted barley

- Mashing
 - To extract fermentable sugars from the malted barley

- Fermentation
 - The addition of yeast to convert sugar to alcohol

- Distillation
 - To remove solid materials, purify the spirit and generate additional flavours

- Maturation
 - The magical process that converts the new-make spirit to whisky

Making other potable alcohol

Can still refer to the key five stages :

- Malting & Mashing

May not be necessary eg. for rum and brandy where the wort is already at the fermentable stage or may be different eg. cooking grains in Scotch Grain or American whisk(e)y.

- Fermentation

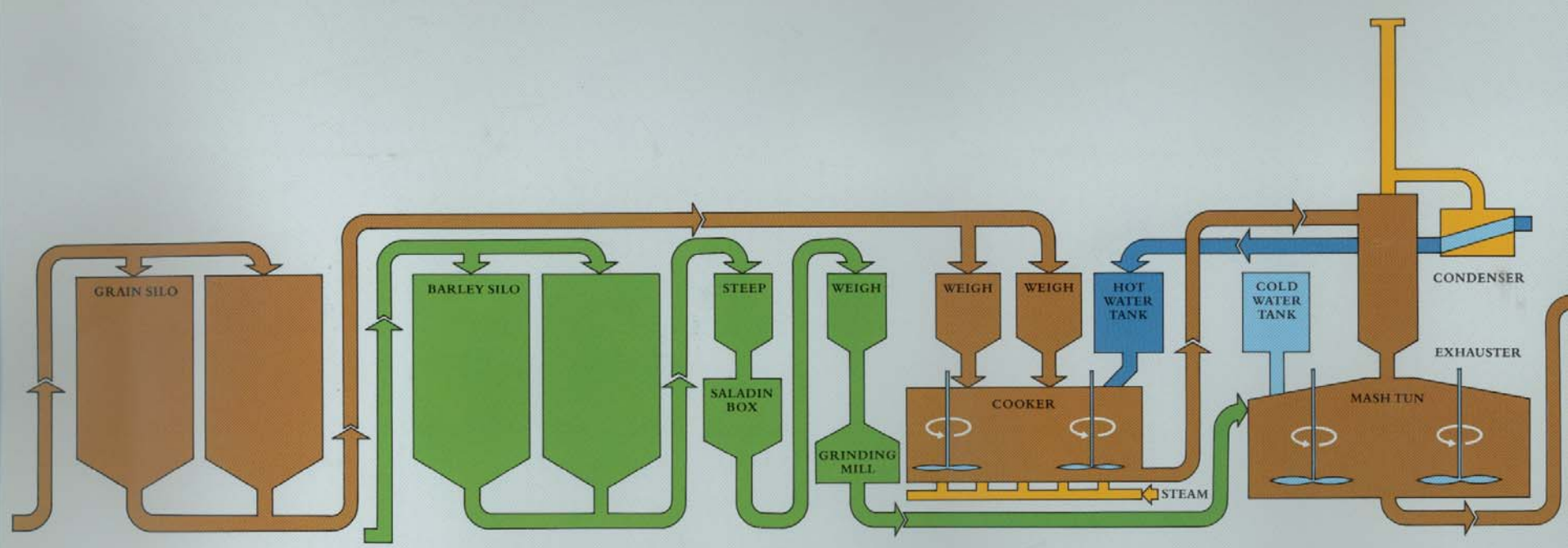
The addition of yeast to convert sugar to alcohol is **always** needed – sometimes it looks a little different to what we expect, but is always used!

- Distillation

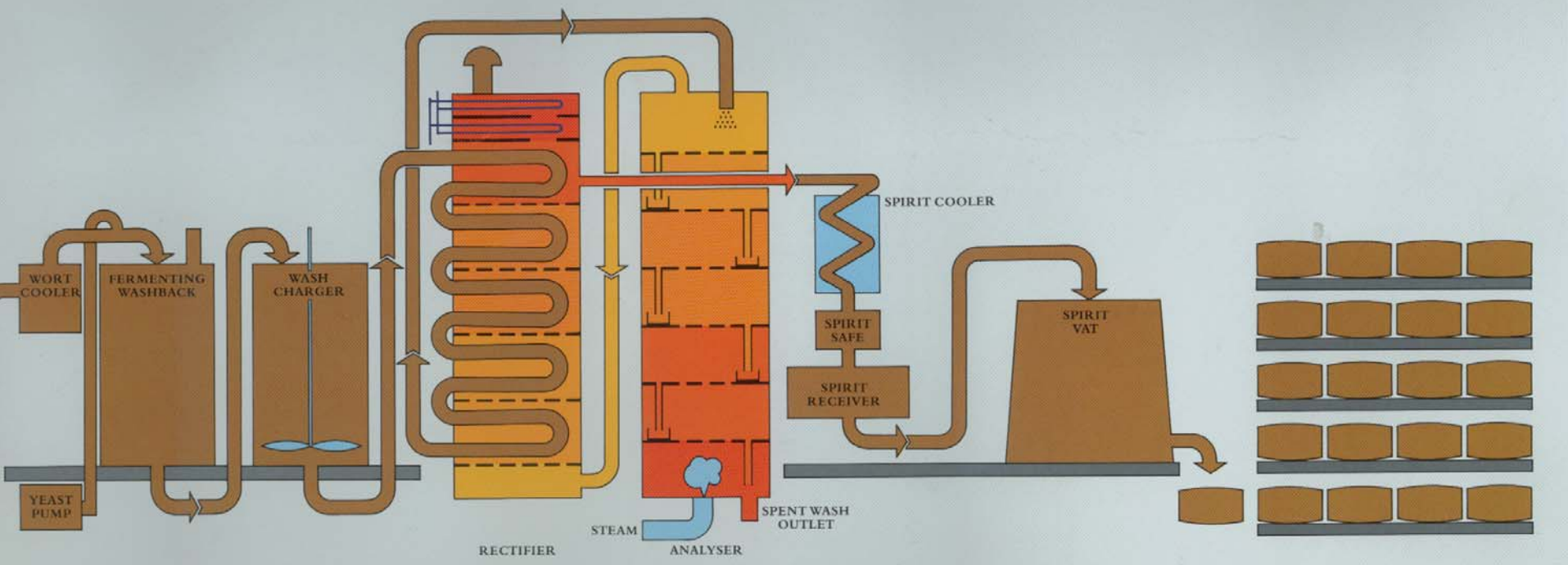
A great variation in methods here with the greatest volumes being generated using continuous (column) distillation processes eg. to make neutral alcohol for gin, vodka, and many other products

- Maturation

Generally does not take place eg. vodka and gin, or to a lesser extent eg. rum (there are some exceptions to this!)



Making Grain Whisky – Part 1 (above) and Part 2 (below)



Processing steps used to make consumer products from potable alcohol

- Blending

Commonly used within the “brown spirits” sector to mix together different “flavours” of spirit eg. to produce blended whisky

- Reduction and Colouring

To reduce the alcohol content to the chosen bottling strength and adjust the colour typically with caramel

- Filtration

To produce a product with the correct clarity and potential chill stability required.

Other Processes used to make consumer products from potable alcohol

- Co-distillation

Best described when referring to making gin, where botanicals are mixed with neutral alcohol in the pot still and co-distilled to extract the flavour into the spirit.

- Compounding

Best described as the addition of other compounds (sugars, flavours, acids, colourants, etc) to deliver a designed alcoholic beverage eg. a liqueur or a ready to drink (RTD) product.

- Bottling (and other packaging)

Quality Analysis

involved in all key stages :

- Malting & Mashing
 - PT testing of barley, malted barley, flour and wort through [MAPS](#)
 - UK NIR network for some key analytes in barley and wheat (eg. moisture, nitrogen)

- Fermentation & Distillation
 - PT testing of wash with [DAPS](#) (A samples) and spirits (B samples)
 - Some internal company standardisation

- Maturation
 - DAPS is trialing this (with B samples).
 - **BNIC (Bureau National Interprofessionnel du Cognac)**
 - Intercompany comparisons

Quality Analysis

involved in **all** key stages :

- Co-distillation
 - Assessed through participation in [DAPS](#) (in particular sample B3)
- Compounding
 - Particularly catered for with E samples in in [DAPS](#)

Other samples available in [DAPS](#) are :

C - Cider

D - Wine

Types of Analytes covered through participation in DAPS

Bulk

- Alcohol strength and Density (or gravity)
- pH
- Colour and Turbidity
- Residue

Major (g/l - %)

- Higher Alcohols
- Acidities (citric, ascorbic, malic, lactic, tartaric)
- Sugars (glucose, fructose, sucrose, maltose, residual fermentable)
- Gases (CO₂ and SO₂)

Trace (ppm - ppB)

- Metals (Ca, Mg, Na, K, Fe, Cu)
- Ethyl Carbamate
- NDMA
- Preservatives (sorbic and benzoic acids)

A Broad Range of Equipment is tested :

Bulk

- Alcohol strength
- pH
- Colour and Turbidity
- Residue

Density meter, NIR, distillation
pH meter (various electrodes)
Spectrometer, photometer
Balance

Major (g/l - %)

- Higher Alcohols
- Acidities
- Sugars
- Gases

GC (plus prep)
Titration, HPLC, IC
Titration, HPLC, IC
Pressure, Orbisphere, Corning

Trace (ppm - ppB)

- Metals
- Ethyl Carbamate
- NDMA
- Preservatives

AAS, IC
GCMS
HPLC
HPLC, IC

Additional Benefits :

- Verification of methodologies used (from start to finish).
- Analysis of a wider variety of sample types – challenging samples.
- Correct understanding of conversion of results (maybe to a different unit than is normally used).
- Validation and comparison of analysts and confidence building through development.
- Valid assessment of GLOBAL ANALYTICAL VARIANCE.
- Remote validation of analytical performance of satellite labs.
- Validation of new methodologies and new technology.

Case review – Alcohol Strength Measurement :

Alcohol strength analysis is **THE** key parameter within the potable spirits industry :

- Too high and HMR&C are unhappy (and the process is inefficient)
- Too low and Trading Standards are unhappy (and consumers)
- All Global markets have tight specifications on alcohol strength. If your product does not conform it will not be saleable.

So how tight do you think is this specification ?
for example on bottle of Scotch bound for USA?

Case review – Alcohol Strength Measurement :

.....the answer is it needs to be at an alcoholic strength between

39.92% ABV to 40.07% ABV

That is a very tight analytical tolerance (approximately 0.2% RSD) !

- The USA is an extreme case, but in most markets the tolerance is in the order of +/- 0.2% ABV and hence an analytical tolerance of less than 0.5% RSD is needed.
- Proficiency testing, along with the use of Certified Reference Materials (CRM's) and regular tightly controlled use of in-lab analytical quality control (AQC) samples is necessary to maintain this level of accuracy.

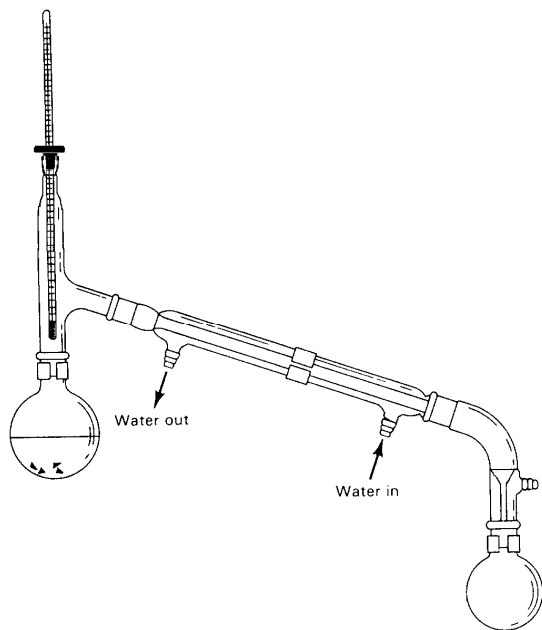
Case review – Alcohol Strength Measurement :

- Historically (18th century) measured by variety of techniques
“Proof - defined as the most dilute spirit that would sustain combustion of gunpowder”

- Density related techniques :
Clarke Hydrometer up to 1816
Sikes Hydrometer up to 1980
Alcohol Hydrometers to present day
Electronic density meter from 1985

- “Real Strength” Measurement
Glass distillation to 1990
Steam Distillation from 1990
Combination Techniques
(Density/Sound Velocity or Density/Refractive Index)

NIR from 2000



Glass Distillation Apparatus
Time for analysis
90 minutes per sample



Steam Distillation Apparatus
Time for analysis
15 minutes per sample

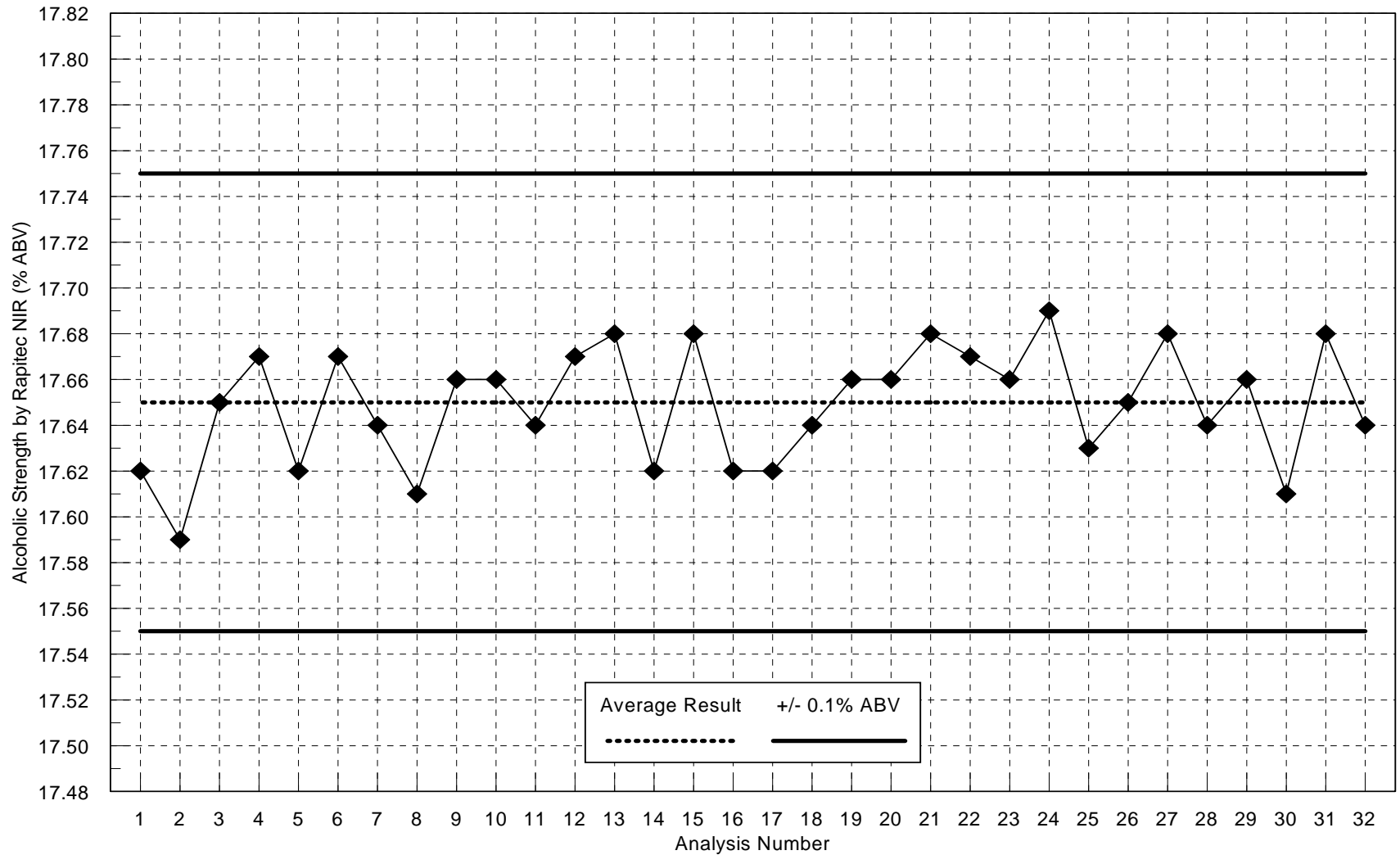


Combination Density Meter
and Refractometer
Time for analysis
5 minutes per sample



Near Infra-Red Analyser
Rapitec (top); Alcoalyzer (below)
Time for analysis
3 minutes per sample

Alcoholic Strength Analysis by RAPITEC™
AQC Control Chart Data



Case review – Alcohol Strength Measurement : The Financial savings !

- Since installation in 2000 across Diageo in Scotland all products have been declared (%ABV) by NIR analysis
- Estimated cost saving to business of over £5M per annum
- Cost saving came from switch from Apparent Strength to Real Strength for declaration (a move of only 0.2% ABV) !
- Switch to Real strength was only possible by proving the accuracy of the new technology
- Proof was developed by participation in DAPS over a number of rounds and via the use of CRM's from LGC

Case review – Alcohol Strength Measurement : DAPS Report

- [DAPS Round 59](#)
 - Multiple results from some labs – may be validating methods
- Alcohol CRM's (single analyte) available from LGC
- Surplus samples may be available from PT schemes like DAPS for use as Reference Materials
 - Cheaper than CRM's
 - Multiple Analyte
 - Correct Sample Matrix

Any Questions ?