Analytical Strategies to Confirm the Generic Authenticity of Scotch Whisky

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ABSTRACT

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The authenticity of a specific brand of Scotch whisky may be confirmed by comparing analytical data for suspect samples with reference to analytical ranges for the genuine brand. Wider generic authenticity issues exist when a product purports to be Scotch whisky when it has not been produced in Scotland in accordance with the legal definition of Scotch whisky. When such cases reach litigation, courts may ask chemists to analyse suspect products and draw conclusions on authenticity. This paper presents analytical profiles generated from a survey of Malt, Grain and Blended Scotch whiskies and compares the results with whiskies of other origins and examples of a diverse range of suspect products purporting to be Scotch whisky. The concentrations and ratios of concentrations of the major volatile compounds (or congeners), particularly methanol, n-propanol, isobutanol and 2- and 3-methyl butanol, were found to be important factors in the authenticity decision-making process. In addition, the absence of known Scotch whisky congeners, the presence of compounds known to be absent from genuine whisky and abnormal maturation congener profiles all contributed to the decision process. From this review of genuine analytical profiles, an experimental protocol for determining the authenticity of Scotch whisky is proposed.

Key words: analysis, authenticity, counterfeit, definition, Scotch whisky, whisk(e)y.

INTRODUCTION

Scotch whisky is a distilled spirit drink made using cereals, yeast and water. It has been produced in Scotland for over 500 years and is the largest selling whisky worldwide. Annual Scotch whisky exports from the UK exceeded £3 billion in 2008 and it is sold in over 180 markets. Individual Scotch whiskies were in production at 96 different malt distilleries and 7 grain distilleries during 2008[17]. "Single whiskies" are sold as the product of one distillery, and the bottled product will generally contain Scotch whisky from several different casks of that one distillery, other than the very limited single cask bottlings. However, most whiskies are blended together from the products of different distilleries using traditional recipes to form the well-known brands of Blended Scotch whisky. Figures from HM Revenue and Customs referred to in an annual statistical report[43] indicate that blended Scotch whisky accounted for almost 89% of UK Scotch whisky consumption in 2007, with malt Scotch whisky accounting for just over 11% and grain Scotch whisky less than 0.05%. There are a vast number of brands available worldwide. Two earlier papers described analytical procedures available for checking the authenticity of Scotch whisky brands[1,28].

Scotch whisky's popularity and reputation has unfortunately led unscrupulous traders to try to take unfair advantage by selling their products as Scotch whisky when they are not Scotch whisky. The sale of such products regularly comes to the attention of consumer protection agencies, Scotch whisky producers and legitimate traders. During the investigation of such products, analytical chemists may be asked to analyse suspect samples and draw conclusions on the generic authenticity of the liquid; that is – is it Scotch whisky? Analysts may encounter difficulties in this task due to the lack of information describing genuine Scotch whisky in comparison to its potential imitators and the lack of guidance on the most appropriate analyses to conduct. This paper describes the types of product encountered, the analyses available to support such investigations and proposes an experimental protocol for generic authenticity analysis. First it is important to consider the Scotch whisky definition and process of Scotch whisky manufacture.

Scotch whisky and its definition

Scotch whisky is made in Scotland from three raw materials: cereals, yeast and water. The process has been defined in UK law for many years and is currently set out in the Scotch Whisky Regulations 2009[44]. The definition requires Scotch whisky to be wholly produced in Scotland. It defines the process by which Scotch whisky is made and not the analytical properties of the finished product. The definition is as follows:

In these Regulations "Scotch whisky" means a whisky produced in Scotland—

(a) that has been distilled at a distillery in Scotland from water and malted barley (to which only whole grains of other cereals may be added) all of which have been—

(i) processed at that distillery into a mash;

(ii) converted at that distillery into a fermentable substrate only by endogenous enzyme systems; and

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(iii) *fermented at that distillery only by the addition of yeast;*

(b) *that has been distilled at an alcoholic strength by volume of less than 94.8 per cent so that the distillate has an aroma and taste derived from the raw materials used in, and the method of, its production;*

(c) *that has been matured only in oak casks of a capacity not exceeding 700 litres:*

(d) *that has been matured only in Scotland;*

(e) *that has been matured for a period of not less than three years;*

(f) *that has been matured only in an excise warehouse or a permitted place;*

(g) *that retains the colour, aroma and taste derived from the raw materials used in, and the method of, its production and maturation;*

(h) *to which no substance has been added, or to which no substance has been added except:*
  - (i) *water;*
  - (ii) *plain caramel colouring; or*
  - (iii) *water and plain caramel colouring; and*

(i) *that has a minimum alcoholic strength by volume of 40%.*

The Regulations include provisions for compulsory sales descriptions for the five categories of Scotch whisky and distinct geographical areas of production within Scotland. The compulsory sales descriptions are Single Malt Scotch Whisky; Single Grain Scotch Whisky; Blended Malt Scotch Whisky; Blended Grain Scotch Whisky; and Blended Scotch Whisky. The regional geographical indications are Campbeltown, Islay, Highland, Lowland, and Speyside.

The Scotch whisky definition is also recognised in the legislation of many other countries. For example, the European Union laid down rules on the definition, description and presentation of spirit drinks under Council Regulation (EEC) 1576/89 in which Scotch whisky was recognised as a geographical designation. Council Regulation (EEC) No 1576/89[13] was repealed by Council Regulation (EC) No. 110/2008[38] which sets out the current definition for whisky or whiskey in the EU as

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

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

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

(c) *No addition of alcohol as defined in Annex 1(5), diluted or not, shall take place.*

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

Annex III lists geographical indications for whisky produced in Europe, including "Scotch Whisky," "Irish Whiskey" and "Whisky Espanol."

Regulations in other countries defining Scotch whisky often make direct reference to the UK process definition. For example, The USA Code of Federal Regulations[42] states:

Scotch whisky is whisky which is a distinctive product of Scotland, manufactured in Scotland in compliance with the laws of the United Kingdom regulating the manufacture of Scotch whisky for consumption in the United Kingdom: Provided, that if such product is a mixture of whiskies, such mixture is "blended Scotch Whisky" (Scotch Whisky – a blend).

The Canadian Food and Drug Regulations[16] state:

"Scotch Whisky shall be whisky distilled in Scotland as Scotch Whisky for domestic consumption in accordance with the laws of the United Kingdom."

The Scotch whisky process

Scotch whisky production starts with the malting process in which barley is germinated. Natural enzymes in the barley break down high molecular weight carbohydrate (starch) into smaller sugars. The sugars are extracted into aqueous solution during the mashing process and the resulting "*worts*" are fermented by the action of yeast into alcohol. Malted barley alone is used in the Malt Scotch whisky process[14], while other cereals such as wheat and maize (corn) are cooked and mixed with a smaller proportion of malted barley to make Grain Scotch whisky[6].

The fermented alcohol (or "*wash*") is distilled in order to extract and concentrate the alcohol. A batch pot still distillation is used for malt spirit[32] and a continuous distillation is used for grain spirit[7]. Two copper pot stills are normally used in malt distilleries (Fig. 1). The first "*wash*" still concentrates the alcohol, from approximately 7% to 30% vol., into "*low wines*". The second "*spirit*" still increases alcoholic strength up to approximately 70% vol. to give new-make spirit (sometimes referred to as "*high wines*"). Most of the continuous stills used for grain spirit are known as Patent or Coffey stills, named after Aeneas Coffey who developed the process in 1831 (Fig. 2). The spirit from this process is typically at about 94% vol. alcoholic strength (but <94.8%) and is lighter in aroma and taste than malt spirits.

Both malt and grain "*new-make*" spirits are then reduced in alcoholic strength with the addition of water typically to between 60 and 70% vol. and matured in oak casks for a minimum of 3 years, after which time the liquid may be described as Scotch whisky[12]. However, Scotch whiskies are often matured for much longer, 12 years being common. The maturation process is responsible for flavour development by converting the pungent character of new-make malt and grain spirits to the softer

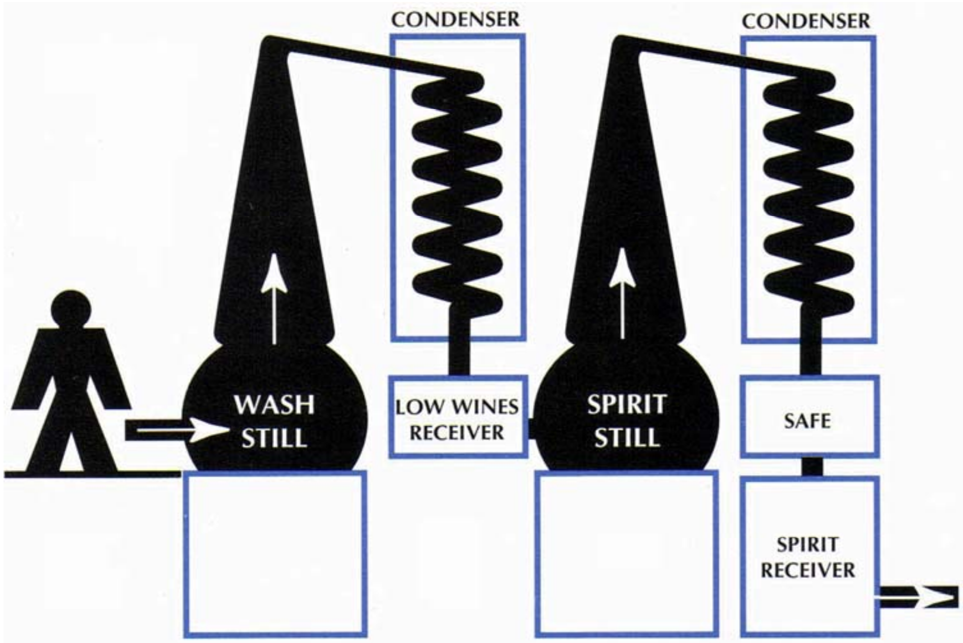

Fig. 1. Malt Scotch whisky distillation process.

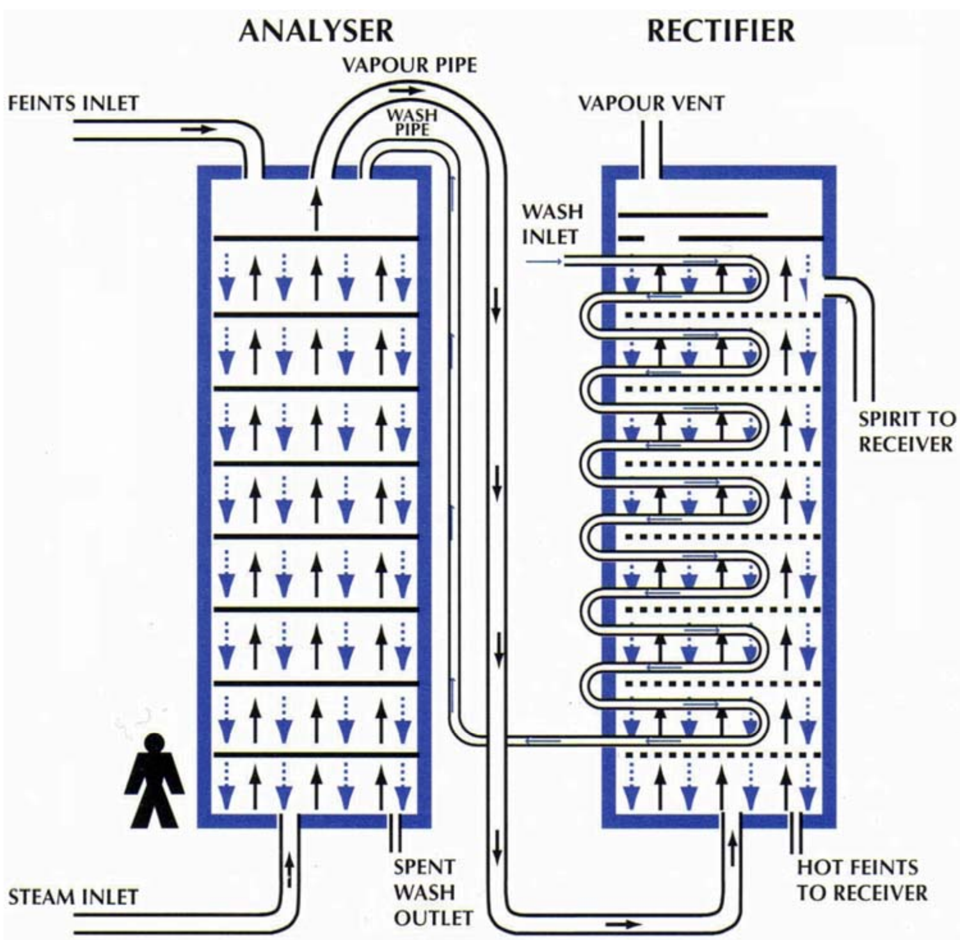

Fig. 2. Typical grain Scotch whisky distillation process.

and complex characters of mature Scotch whiskies. Maturation involves processes of congener addition, congener reduction and congener production[35]. Scotch whisky casks are made from oak, normally European or American oak. Most of these casks were previously used for the maturation of sherry or Bourbon whiskey. As the character of the spirit develops, the volume in each cask decreases by 1 to 2% per year due to evaporation. Whilst new-make malt and grain spirits are clear liquids prior to the start of maturation, they acquire varying amounts of colour from the oak casks during the maturation process.

At the completion of the required period of maturation, whisky blenders select individual malt and grain whiskies according to their sensory properties (as determined by distillery, age and cask characteristics) and blend them together to produce brands of Blended Scotch whisky with unique character and consistent quality. A smaller proportion of the available stocks are retained for sale as single malt and grain whiskies.

The final stage of the production process involves alcoholic strength reduction down to the required bottling strength with water, the optional addition of a small amount of plain caramel (E150a) to give consistent colour from one batch to another and filtration to ensure a clear bright product. Plain caramel is sometimes referred to as spirit caramel. Some whiskies, particularly specialist malt whiskies, may only be subject to a coarse filtration resulting in a slightly cloudy liquid. The Scotch whisky is then packaged, normally in glass or PET bottles ready for shipment. Some Scotch whiskies are shipped in bulk to another country for local bottling (thus enabling reductions in transport and material costs and local taxes). Scotch whisky bottled outwith Scotland is still entitled to carry the designation "Scotch whisky" as it is distilled and matured in Scotland according to the Regulations, as long as no substance is added prior to bottling other than water and plain caramel to adjust colour. However, as from 23 November 2012 the new Regulations[44] will require all Single Malt Scotch whisky to be bottled in Scotland.

Whilst all Scotch whiskies must meet the regulatory definitions within the Scotch Whisky Regulations, there is considerable diversity within the broad category. Blenders can choose from whiskies of different ages from over 100 distilleries resulting in many potential malt whisky/grain whisky ratios, age profiles and sensory characteristics. Brands of Blended Scotch whisky have been sold in deluxe, premium, standard and other brand categories depending on blend composition and age. Malt Scotch whiskies are often categorised by distillery into the five geographical regions referred to in the Scotch Whisky Regulations[44], Lowland (from the south of Scotland), Highland (from the north of Scotland), Speyside (in the north-east in the valley of the River Spey), Islay (the west coast island with 8 malt distilleries) and Campbeltown. Only a handful of Grain Scotch whiskies are sold as single whiskies. Some whisky labels carry an age statement, for example "aged 12 years" and this by law means that the youngest whisky in that blend has been matured for at least 12 years.

However, as mentioned earlier all Scotch Whiskies fit into the five categories set out in the Scotch Whisky Regulations 2009 as follows:

Single Malt Scotch Whisky - a Malt Scotch whisky from one distillery

Single Grain Scotch Whisky - a Grain Scotch whisky from one distillery

Blended Scotch Whisky - a blend of at least one Single Malt Scotch Whisky and at least one Single Grain Scotch Whisky

Blended Malt Scotch Whisky - a blend of Single Malt Scotch Whiskies from more than one distillery.

Blended Grain Scotch Whisky - a blend of Single Grain Scotch Whiskies from more than one distillery.

The reader is referred to a number of useful articles and books on the industry. In particular, texts edited by Piggott, Sharp and Duncan[37], Lyons, Kelsall and Murtagh[25] and more recently the chapter by Piggott and Conner[36] and the book edited by Russell[39]. Within the latter book, Bathgate described the history of the development of whisky distilling[4]. Separately, Watson[46], Rutherford[40] and Hills[18] have covered technical detail for non-scientists and whisky enthusiasts.

Other whiskies around the World

Whisky (or whiskey) is distilled in many countries, with particular styles in Scotland, Ireland, the USA and Canada. Some whisky production also takes place in Japan, Spain, New Zealand and the Czech Republic. The products of Scotland, Canada and Tennessee normally use the spelling "whisky", while those from Ireland and Kentucky use the word "whiskey".

Irish whiskey, like Scotch whisky, has a long history. A range of blends are made from individual malt and grain whiskies, many of the malts being triple distilled in three pot stills and the grain whiskies being continuously distilled on three column systems, making them lighter in character than many of their Scottish equivalents. In Ireland, pot still whiskies are also made from a mash of malted barley and unmalted cereals, typically oats or barley. Most Irish whiskey brands are blended from these three types of whiskies. Like Scotch whisky, the minimum maturation period is three years. Production is currently centred in only three locations: Middleton, the Cooley peninsula in the Republic of Ireland, and Bushmills in Northern Ireland. The legal definition may be found in the Irish Whiskey Act 1980[21] and it is recognised as a geographical indication in EU Regulation 110/2008[38]. Production and maturation may take place in the Republic of Ireland or Northern Ireland.

American and Canadian whiskies are quite different to those from Scotland and Ireland. The major American whisky is "bourbon whiskey" with most production located in Kentucky. Cereals used include barley, wheat, rye and corn. The methods of distillation and maturation differ from those employed in Scotland and Ireland. Sugars are often extracted from the cereals using a "sour mash" process that uses some of the spent grain and water residue from a previously distilled batch added into the *mash tub*, the *fermenter* or both. The first (and sometimes only) distillation is produced on a continuous distillation column known as a "*beer still*" and maybe followed by redistillation. This redistillation may be a continuous or batch process in a pot still known as a "*doubler*" or "*thumper*". The maximum distillation strength is 80% vol. alcoholic strength. The "*new distillate*" spirit is then matured in new charred American oak casks (or barrels) for a minimum of two years in order to be designated a "*straight whiskey*". Barrels are used once only in the bourbon maturation process, after which empty barrels are usually sold to other spirit producers for use in their maturation processes. Tennessee whiskies have many of the characteristics of bourbon, but go through one key additional process involving the percolation of high wines through maple charcoal before beginning maturation in oak barrels. Other American categories include corn, rye and wheat whiskies, together with American blended whiskey. This last category may only contain a proportion of whiskey, as the main constituent can be neutral spirits distilled at >95% vol. alcoholic strength[45].

Canadian whiskies are normally much lighter in character compared to most American whiskies. This is as a result of a blend composition based on matured grain spirits and a smaller proportion of congener-rich whisky (the congener-rich whisky being similar in style to bourbon). Rye, corn, barley and wheat are used according to location; maturation is in re-used American barrels. Pot still malt whisky is also produced. Canadian whiskies may also contain up to 9.09% "blending" or flavouring materials such as wines or other spirits[15], but the addition of such materials is not allowed if the product is sold in the European Union.

A further group of countries, from Latin America to the Far East, produce distilled spirits that are often referred to as "national whiskies"[5,8–10]. Many "national whiskies" are admixtures of a locally produced, non-matured neutral alcohol flavoured with a proportion of a malt whisky or a similar spirit. The neutral alcohol component may sometimes be fermented from cereal, but more often is made from cheaper cane or molasses fermentations. Neutral alcohol is typically distilled at >96% vol. alcoholic strength and is essentially very pure ethanol of agricultural origin with little if any remaining congeners associated with the fermentation. The malt whisky component may be an imported bulk Malt Scotch whisky or it may be a locally produced pot still whisky as produced in Brazil and India. The "national whiskies" described above do not qualify as whisky under the European Union definition[38] due to their use of one or more of the following components:

1. Alcohol that has not been distilled at <94.8% vol.,
2. Alcohol that has not been fermented from cereals.
3. Alcohol that has not been subject to 3-year maturation.
4. Additives or flavouring.

Lastly, some countries such as India[19] designate their local products as whisky in their country of origin even though they have not necessarily been fermented from cereals or matured in cask as required in European and North American markets. Such products are usually based upon cane or molasses alcohol and are flavoured with either natural or artificial flavourings, although some use Malt Scotch whisky to provide flavour as with the "national whiskies" referred to above.

SCOTCH WHISKY AUTHENTICITY

Analytical strategies applied to brand authenticity involve developing an analytical fingerprint for a known brand against which the equivalent results for a suspect sample may be compared[1]. Whilst gas and liquid chromatography may be applied to the determination of hundreds of whisky congeners, it was found that analysis of the major volatile congeners (particularly the higher alcohols that include n-propanol, isobutanol and 2- and 3-methyl butanols) allowed normal congener concentration ranges to be set. If a suspect sample's results fell outside the normal ranges for the genuine brand, the sample was not authentic. This analytical approach can be used to confirm by analysis that a suspect sample is not genuine. It cannot be used to state with certainty that a product is genuine, only that the analytical results are consistent with those of the genuine product. This approach to brand authenticity is routinely used in UK Public Analysts' and other laboratories that support consumer protection agencies and brand owners[2]. However, chromatographic methods can be expensive and time consuming. This led to the development of a fast and reliable field test that can pre-screen whisky samples and thus eliminate many samples from the chromatographic procedures[28]. It uses the characteristic UV/visible spectra of Scotch whisky brands to authenticate test samples on a small hand-held instrument known as the "brand authenticator".

Developing strategies for generic authenticity analysis has involved developing an analytical fingerprint, not for specific brands but for Scotch whiskies as a whole. This fingerprint can then be used to show how Scotch whisky is distinct from whiskies of other origins, from other distilled spirits and from other materials such as neutral alcohol, each of which may contribute to the composition of products falsely claiming authenticity.

Experimental

Analytical methods for apparent alcoholic strength measurement, gas chromatographic analyses for major volatile congeners, capillary column gas chromatography–mass spectrometry (GC-MS) for trace congeners and contaminants and liquid chromatographic (HPLC) analyses for trace maturation congeners are described in an earlier paper[1].

All samples were analysed by temperature-programmed gas chromatography for their major volatile congeners resulting in quantitative data for acetaldehyde, methanol, ethyl acetate, n-propanol, isobutanol and 2- and 3-methyl butanol. This separation may also be used to determine n-butanol and acetic acid that are present in various spirits at trace levels.

Bottled Blended Scotch whiskies and the suspect case study samples were also analysed by gradient elution liquid chromatography for furfural and a range of four maturation congeners that include vanillic acid, syringic acid, vanillin and syringaldehyde. This separation may also be used to determine other maturation congeners such as gallic acid, coniferaldehyde, ellagic acid, and scopoletin, plus 5-hydroxymethyl furfural, a congener mainly associated with caramel. Bottled Blended Scotch whiskies were analysed for trace sugars. Trace glucose, fructose and sucrose were determined by direct injection (10 µl) liquid chromatography at 30°C on a 25 cm Dionex PA1 ion exchange column (with 50 mM sodium hydroxide in water as eluant at a flow rate of 1.9 ml/min) coupled to a pulsed amperometric detector with a gold electrode. Multilevel calibration standards were prepared in 40% ethanol in water solutions.

Detailed methods may also be found in "Methods for the Analysis of Potable Spirits"[31] and the AOAC Handbook[33]. In addition, the European Commission published Commission Regulation 2870/2000 laying down Community Reference Methods for the Analysis of Spirit Drinks[11], which are particularly useful for real alcoholic strength and volatile substances and methanol. The precisions of the latter methods were established in an interlaboratory study[22]. Additionally, an overview on whisky analysis is given in the chapter by Aylott[3].

RESULTS AND DISCUSSION

The analytical fingerprint of Scotch whisky

Single samples of 56 individual brands of bottled Single Malt Scotch whisky, 5 individual brands of Bottled Single Grain Scotch whisky and 20 individual brands of bottled Blended Scotch whisky, representing products sold at a wide range of retail prices, were analysed.

Table I gives the major volatile congener result ranges for individual bottled Single Malt, Single Grain and Blended Scotch whiskies. The results for the 20 brands of Blended Scotch whiskies fit within the ranges of results for the Single Malt and Single Grain Scotch whiskies. These results extend earlier work in this area by Shoeneman and Dyer[41] and Lisle, Richards and Wardleworth[24].

Blended Scotch whiskies, being combinations of many different Scotch malts and grains, are diverse and show analytical profiles representative of their constituent parts. Table II gives the individual volatile congener results for the twenty brands and Table III gives the corresponding furfural and maturation congener results. The samples in both tables are ranked in order of increasing 2- + 3-methyl butanol concentrations.

The compounds n-propanol and isobutanol are common to all Scotch whiskies. In particular, the sum of n-propanol and isobutanol calculated for each individual sample ranged from 97 to 198 g/100 litres absolute alcohol in 56 samples of bottled Malt Scotch whisky and 102 to 221 g/100 litres absolute for the 5 samples of bottled Grain Scotch whisky (Table I). The sum of n-propanol and isobutanol ranged from 98 to 188 g/100 litres absolute alcohol in the 20 bottled Blended Scotch whiskies.

The concentrations of 2- and 3-methyl butanol in Grain Scotch whisky were relatively low compared to n-propanol and isobutanol. This is because these two congeners along with other less volatile congeners are selectively reduced in grain spirit during the typical distillation in the Coffey still. The Malt Scotch whiskies were rich in these higher alcohols, with the average 2- and 3-methyl butanol concentration being 190 g/100 litres absolute alcohol, compared to only 30 g/100 litres absolute alcohol in the Grain Scotch whiskies.

As would be expected in Blended Scotch whiskies the ratio of 2- + 3-methyl butanol/isobutanol increased with increasing 2- + 3-methyl butanol concentrations (and thus increasing malt whisky content). The ratio of 3-methyl butanol/2-methyl butanol ranged between 2.3 and 2.9 (average 2.6) and also reflected the values found in Malt Scotch whisky, but with fewer outlying results. Methanol concentrations also reflected the constituent malt and grain whiskies, as did ethyl acetate.

Thus 2- + 3-methyl butanol concentrations may be principally associated with the malt whisky component of Blended Scotch whiskies. As a result, the 2- + 3-methyl butanol concentration may be used to give an estimate of the relative proportions of malt and grain whisky present. Though the sum of the analytical range of 2- and 3-methyl butanol in Malt Scotch whisky was wide, dividing the result on the sample under consideration by 1.9 (100/190) gave a useful means of estimating the approximate percentage of malt whisky in Blended Scotch whisky – 190 g/100 litres absolute alcohol being the average 2- + 3-methyl butanol concentration in the malt whiskies sampled.

Methanol concentrations for all samples analysed ranged between 4.7 and 16.4 g/100 litres absolute alcohol. The n-butanol concentrations were all <1 g/100 litres absolute alcohol.

The results for furfural and four maturation congeners in 19 of the Blended Scotch Whisky samples are reported in Table III, together with the sum of the four congeners and the two concentration ratios, vanillin/syringaldehyde

Table I. Major volatile congener ranges for bottled samples of single malt Scotch whisky, single grain Scotch whisky and blended Scotch whisky.[a]

				g / 100 litres absolute alcohol											
Scotch whisky type	No. of distilleries surveyed	No. of brands surveyed		Acetal-dehyde	Metha nol	Ethyl acetate	n-Pro-panol (p)	Iso-butanol (b)	2-Methyl-butanol	3-Methyl-butanol	2- + 3-Methyl-butanol (a)	(a) / (b)[b]	3-Methyl butanol / 2-Methyl butanol	p + b[c]	p + b + a[d]
Scotch malt	56	56	Min.	2.6	4.7	12	34	50	37	111	151	1.9	2.2	97	252
whisky			Avg.	11.1	6.5	38	47	83	50	140	190	2.4	2.9	130	320
			Max.	17.0	9.7	66	73	147	92	207	299	3.5	3.5	198	494
Scotch	5	5	Min.	6.4	5.1	17	46	56	4	14	18	0.3	2.5	102	139
grain			Avg.	9.4	11.1	24	89	72	8	23	30	0.6	3.0	161	191
whisky			Max.	13.2	16.4	37	138	83	9	31	40	1.1	3.4	221	244
Scotch	-	20	Min.	5.2	5.2	16	36	56	7	19	26	0.4	2.3	98	152
blended			Avg.	7.2	9.6	28	69	70	18	48	66	0.9	2.6	139	205
whisky			Max.	11.0	14.0	53	121	94	38	107	145	1.6	2.9	188	297

[a] Sums and ratios are calculated from the results for each individual sample.
[b] Total 2- and 3-methylbutanol / isobutyl alcohol.
[c] n-Propanol + isobutanol.
[d] n-Propanol + isobutanol + 2- and 3-methyl butanol.

Table II. Profiles of major volatile congeners in twenty brands of bottled Blended Scotch whisky.[a]

	g / 100 litres absolute alcohol											
Brand number	Acetal-dehyde	Meth-anol	Ethyl acetate	n-Propanol (p)	Isobutanol (b)	2-Methyl-butanol	3-Methyl-butanol	2- + 3-Methyl-butanol (a)	(a) / (b)[b]	3-Methyl-butanol / 2-methyl-butanol	p + b[c]	p + b + a[d]
1	6.3	10.3	24	92	68	7	19	26	0.4	2.7	160	186
2	9.1	10.3	24	121	67	8	20	28	0.4	2.5	188	216
3	7.4	8.6	22	83	71	12	30	42	0.6	2.5	154	196
4	7.0	11	24	94	64	11	32	43	0.7	2.9	158	201
5	5.8	7.9	16	37	70	13	32	45	0.6	2.5	107	152
6	5.9	13.7	25	59	59	15	35	50	0.8	2.3	118	168
7	6.6	5.2	17	38	70	17	40	57	0.8	2.4	108	165
8	5.5	7.4	23	86	56	18	43	61	1.1	2.4	142	203
9	5.2	5.2	20	36	62	18	45	63	1.0	2.5	98	161
10	5.3	6.2	21	67	63	18	46	64	1.0	2.6	130	194
11	7.2	7.7	25	73	71	19	49	68	1.0	2.6	144	212
12	8.7	14	26	62	58	19	49	68	1.2	2.6	120	188
13	7.3	12	36	71	80	18	52	70	0.9	2.9	151	221
14	7.4	9.2	34	86	71	20	53	73	1.0	2.7	157	230
15	7.4	11.5	34	71	80	21	53	74	0.9	2.5	151	225
16	6.0	9.5	24	57	61	22	56	78	1.3	2.5	118	196
17	8.4	8.3	34	45	61	24	61	85	1.4	2.5	106	191
18	8.2	12	41	72	94	23	67	90	1.0	2.9	166	256
19	8.9	12	42	71	85	24	68	92	1.1	2.8	156	248
20	11.0	9	53	64	88	38	107	145	1.6	2.8	152	297
Min.	5.2	5.2	16	36	56	7	19	26	0.4	2.3	98	152
Avg.	7.2	9.6	28	69	70	18	48	66	0.9	2.6	139	205
Max.	11.0	14.0	53	121	94	38	107	145	1.6	2.9	188	297

[a] Sums and ratios are calculated from the results for each individual sample.
[b] Total 2- and 3-methylbutanol / isobutyl alcohol.
[c] n-Propanol + isobutanol.
[d] n-Propanol + isobutanol + 2- and 3-methylbutanol.

Table III. Profiles of furfural and four maturation congeners in nineteen brands of bottled blended Scotch whisky.[a]

	g / 1,000 litres absolute alcohol							
Brand number	Furfural	Vanillic acid	Syringic acid	Vanillin	Syringaldehyde	Total[b]	Vanillin / syringaldehyde	Vanillin / vanillic acid
1	3.0	0.5	0.7	0.7	1.5	3.4	0.5	1.5
2	nm[c]	0.5	0.7	0.8	1.5	3.5	0.6	1.8
3	11.0	0.4	0.4	0.7	1.3	2.8	0.5	1.9
4	nm	nm	nm	nm	nm	nm	nm	nm
5	11.0	0.6	1.6	1.1	2.0	5.4	0.5	1.8
6	8.6	1.0	1.4	1.5	2.6	6.6	0.6	1.5
7	9.0	0.4	0.6	1.0	1.9	3.8	0.5	2.3
8	8.5	0.4	0.6	1.1	1.7	3.8	0.6	3.1
9	10.4	0.8	0.9	1.3	2.0	5.1	0.6	1.5
10	9.7	1.1	1.0	1.0	1.8	5.0	0.6	0.9
11	14.0	0.6	0.9	1.4	2.5	5.4	0.5	2.2
12	11.0	1.3	1.5	2.0	3.6	8.4	0.6	1.6
13	11.0	1.1	1.9	2.0	4.2	9.2	0.5	1.8
14 (8 years old)	14.0	1.7	3.2	1.7	4.0	10.7	0.4	1.0
15	11.0	1.1	1.5	1.8	3.9	8.3	0.5	1.7
16	12.0	0.7	0.9	1.3	2.2	5.0	0.6	1.9
17 (12 years old)	19.0	1.8	2.8	3.4	5.4	13.4	0.6	1.9
18 (12 years old)	14.0	1.2	2.0	2.2	4.4	9.8	0.5	1.7
19 (12 years old)	13.0	1.3	2.1	2.2	4.1	9.7	0.5	1.8
20	19.9	3.0	2.9	3.3	6.5	15.8	0.5	1.1
Minimum	3.0	0.4	0.4	0.7	1.3	2.8	0.4	0.9
Average	11.7	1.0	1.5	1.6	3.0	7.1	0.5	1.7
Maximum	19.9	3.0	3.2	3.4	6.5	15.8	0.6	3.1

[a] Whisky age is reported when quoted on the brand label. Sums and ratios are calculated from the results for each individual sample.
[b] Sum of vanillic acid, syringic acid, vanillin and syringealdehyde.
[c] Not measured.

and vanillin/vanillic acid. Furfural is a congener principally associated with malt whisky and therefore its concentration tends to increase as the 2- +3-methyl butanol concentration increases. This is to be expected as both congeners are principally derived from the malt whisky component of the blend. The compound 5-hydroxymethyl furfural is also found in many whiskies due to its presence in plain caramel.

Simple sugars are naturally present in bottled Scotch whisky as a result of the later post-distillation stages of the production process. Trace concentrations are drawn out of the oak wood as a result of maturation and levels are influenced by previous cask usage. In addition, trace sugars may be present as low molecular weight material present in plain caramel.

The main sugars are glucose and fructose. Sucrose is generally absent. However, if present, it is always at a markedly lower concentration than glucose and fructose. The sugar concentrations detected in 17 different brands of bottled Blended Scotch whiskies, as already reported for major volatile and maturation congeners in Tables II and III, are shown in Table IV. Glucose concentrations ranged from 34 to 230 mg/litre, fructose ranged from 28 to 210 mg/litre and sucrose ranged from <1 to 4 mg/litre. Significantly, the ratio of sucrose/(glucose + fructose) was always <0.1 and typically between zero and 0.01.

The Malt, Grain and Blended Scotch whiskies all had light brown colour or tint, label alcoholic strengths at either 40 or 43% vol. and pH values between 3.5 and 4.5. Apparent alcoholic strength measurements differed from their real alcoholic strengths by between 0.05 and 0.4% vol., this obscuration being due to the relatively small amount of dissolved solids derived from the maturation process and plain caramel. Obscuration is a term used in the spirits industry meaning the difference between real and apparent alcoholic strength.

Scotch whiskies exhibit a unique range of sensory properties. Whiskies are usually assessed or "*nosed*" in a tulip shaped glass. The sample is reduced down to approximately 20% vol. alcoholic strength by the addition of an appropriate volume of distilled water and left to stand for a few minutes covered with a watch glass in order to let its aromas develop before nosing. Whisky blenders have their own special vocabularies to describe the sensory characteristics of Scotch whiskies. For example, the *pungent, soapy, sour* and *harsh* notes in new-make malt spirit are replaced during the maturation processes with *smooth, matured* and *mellowness* attributes. Words such as *peaty, grainy, grassy, fruity, floral, feints, woody, sweet, sulphury, cheesy* and *oily* have recently been incorporated into a revised whisky flavour wheel and a range of reference compounds has been associated with these terms[23].

Stability of major volatile congener concentrations in bottled product

It is important to know if sample composition is stable while a sample is packaged in a glass bottle, particularly those congeners that may be used to investigate authenticity. Therefore, in separate experiments, the stabilities of individual congeners were examined in order to identify those congeners that are stable with time.

The first experiment involved normal long term storage of Blended Scotch whisky packaged at 43% vol. alcoholic strength in sealed 5 cl glass bottles maintained under ambient (20°C) conditions over periods extending to two years. Three unopened bottles were removed from storage after 0, 3, 6, 12 and 24 months and subjected to apparent alcoholic strength and major volatile congener analyses. Alcoholic strength increased by an average of 0.4% in the first year and 0.7% vol. over the two year period. Weight loss of liquid averaged 0.9% over one year and 1.7% over two years. These results suggested that water migrated

Table IV. Profiles of trace sugar congeners in a range of seventeen bottled blended Scotch whiskies.[a]

	mg/litre product				
Brand number	Glucose	Fructose	Sucrose[b]	Total[c]	Sucrose / (Glucose + Fructose)
1	85	76	<1.0	161	0.01
2	62	54	<1.0	116	0.01
3	42	47	<1.0	89	0.01
4	65	56	<1.0	121	0.01
5	100	70	<1.0	170	0.01
6	71	63	<1.0	133	0.01
7	48	46	<1.0	94	0.01
8	43	41	<1.0	83	0.01
11	34	28	<1.0	62	0.02
12	54	44	<1.0	98	0.01
13	100	93	<1.0	193	0.01
14	67	57	<1.0	123	0.01
15	130	120	4.0	254	0.02
17	91	68	<1.0	158	0.01
18	160	137	<1.0	297	<0.01
19	190	160	<1.0	350	<0.01
20	230	210	<1.0	440	<0.01
Minimum	34	28	<1.0	62	<0.01
Average	92	80	1.2	173	0.01
Maximum	230	210	4	440	0.02

[a] Sums and ratios are calculated from the results for each individual sample.
[b] The average sucrose concentration was calculated using the limit of detection shown in the data set.
[c] Total sugars = glucose + fructose + sucrose.

through the bottle-closure interface faster than alcohol. Methanol, ethyl acetate and higher alcohol concentrations remained unchanged while acetaldehyde concentrations increased by between 5 and 10%.

The second experiment involved a severe short term storage condition in which a half-full 70 cl bottle of Blended Scotch whisky at 40% vol. alcoholic strength was left open with its closure removed under well ventilated conditions at 30°C. Samples were taken at intervals over one week and alcoholic strength and the volatile congeners determined. During this time the whisky's alcoholic strength fell by 1.1% vol. Volatile congener results indicated that methanol, n-propanol, isobutanol and 2- + 3-methyl butanol concentrations remained stable with respect to absolute alcohol concentration units. Acetaldehyde concentrations decreased by 33% after one week and ethyl acetate concentrations decreased by 47%.

In summary, results from these two experiments indicated that whisky is very stable in sealed bottles under normal ambient conditions with methanol, n-propanol, isobutanol and 2- + 3-methyl butanol concentrations remaining unchanged in unopened and well-sealed bottles. Analytical changes were much more evident at elevated temperatures with the bottle closure removed. The results indicated that most attention in authenticity tests should be paid to methanol, n-propanol, isobutanol and 2- + 3-methyl butanol concentrations. Acetaldehyde and ethyl acetate may suffer greater evaporative losses from an opened bottle. Acetaldehyde may slowly increase due to oxidation of ethanol in sealed bottles. Therefore, acetaldehyde and ethyl acetate concentrations in a suspect sample may change depending on the environment in which that product was stored.

The analytical fingerprint of Irish, Bourbon and Canadian whiskies

A collection of 5 individual bottled brands of blended Irish whiskey, 15 bourbon and 6 Canadian whiskies were analysed for their major volatile congeners. The results shown in Table V demonstrate that these three types of whisky each have quite different analytical characteristics compared to Scotch whisky. Unlike the Scotch whisky survey, the number of samples surveyed in this section was relatively small.

The 5 brands of blended Irish whiskey were analytically most similar to Scotch whisky, although their n-propanol and isobutanol concentrations were generally lower. These results reflected the greater rectification from a triple Irish grain distillation compared to a normal two column characteristic of Scotch grain whisky.

The 15 bourbon whiskies had relatively high concentrations of 2- and 3-methyl butanol in comparison to Scotch whisky, an effect associated with the cereals, fermentation and distillation processes employed. The n-propanol concentrations were relatively low compared to Scotch whiskies while isobutanol concentrations varied considerably. The average 2- +3-methyl butanol/isobutanol ratio at 3.1 compared with 2.4 for malt Scotch whisky.

The Canadian whiskies showed quite different analytical profiles to Scotch, Irish or bourbon whiskies. The higher alcohol concentrations in the 6 brands analysed were relatively low compared to the other two whisky types, again reflecting the high levels of rectification employed during the distillation of the major whiskies in their blends.

However, analytical commonalities were observed between Scotch, Irish, bourbon and Canadian whiskies and these reflected commonalities in their processes. For example, all four whisky types use only cereals in their fermentations and this resulted in the maximum methanol concentration observed being 20 g/100 litres absolute alcohol. Therefore, 25 g/100 litres absolute alcohol was set as an arbitrary maximum methanol in barley, wheat or maize distillates; any methanol concentration above this in suspect samples could be used to suggest use of a non-cereal alcohol. It should also be noted that while the highest methanol found in the five grain Scotch whiskies was 16.4 g/100 litres absolute alcohol (Table I), these whiskies were distilled from barley and wheat. Replacing wheat by maize (corn) in grain whisky fermentations has been shown to elevate resulting methanol concentrations up to 25 g/100 litres absolute alcohol. Distilled spirits fermented from grape and fruits tend to have much higher methanol concentrations[24,41]. It should be noted that the flavouring materials that can be added to Canadian Whiskies sold in various countries out with the EU may contain alcohol from a non-cereal source that influences the methanol level.

In addition, the ratio of 3-methyl butanol/2-methyl butanol in all the genuine whiskies distilled from cereals ranged between 1.4 and 3.5 (Tables I and V). The 2- and 3-methyl butanol along with n-propanol and isobutanol

Table V. Major volatile congener ranges for single bottled samples of Irish, Bourbon and Canadian whiskies.[a]

Whisky type	No. of brands surveyed		g / 100 litres absolute alcohol: Acetaldehyde	Methanol	Ethyl acetate	n-Propanol (p)	Isobutanol (b)	2-Methylbutanol	3-Methylbutanol	2- + 3-Methylbutanol (a)	(a) / (b)[b]	3-Methylbutanol / 2-methylbutanol	p + b[c]	p + b + a[d]
Irish whiskey	5	Min.	3.5	7.7	13.0	28	16	14	35	49	2.9	2.6	43	92
		Avg.	5	9	16.2	35	25	21	56	77	3.1	2.7	60	137
		Max.	9	10.7	19.3	46	34	26	77	103	3.2	2.9	80	183
Bourbon whiskey	15	Min.	5.2	12.2	44	18	57	74	206	280	2.1	1.4	75	355
		Avg.	9.3	15.0	72	25	121	118	252	370	3.1	2.2	146	576
		Max.	11.6	20.0	112	38	255	178	420	598	5.6	2.8	284	882
Canadian whisky	6	Min.	3.3	5.4	7.0	1.0	3.4	4.3	9.4	13.7	2.6	2.1	4.4	14.3
		Avg.	5.5	9.0	15.0	2.8	6.5	6.1	11.3	19.4	3.2	2.2	9.3	25.7
		Max.	8.1	12.0	22.0	4.3	9.9	9.0	19.0	28.0	4.0	2.3	13.5	37.0

[a] Sums and ratios are calculated from the results for each individual sample.
[b] Total 2- and 3-methylbutanol / isobutyl alcohol.
[c] n-Propanol + isobutanol.
[d] n-Propanol + isobutanol + 2- and 3-methyl butanol.

are principally formed from amino acids during secondary fermentation[20]. These ratios can differ with other fermentation substrates such as sugar cane or grape. Ratios outside these reported ranges can be used to suggest the use in suspect samples of non-cereal alcohol and/or added artificial flavourings that include 3-methyl butanol.

The analytical fingerprint of some national whiskies

A collection of 31 individual bottled brands, produced, bottled and purchased in India and South America, were analysed for their major volatile congeners. The results shown in Table VI show clear distinctions compared to Scotch whisky. The products from India were the most analytically diverse, reflecting the regulations in that country that permit local whiskies to include neutral alcohol fermented from cane, sometimes flavoured with local malt whisky and sometimes with artificial flavourings. This resulted in congener concentrations ranging from the barely detectable up to ratios of 2- + 3-methyl butanol/isobutanol and 3-methyl butanol/2-methyl butanol well above those encountered in cereal based spirits.

The profiles of the products produced in Argentina, Brazil, Colombia and Venezuela showed the same volatile congeners to those in Scotch whisky but their relative concentrations were much lower (Table VI). At the same time the ratios of 2- + 3-methyl butanol/isobutanol and 3-methyl butanol/2-methyl butanol were characteristic of malt whiskies. These analytical results were consistent with these brands being admixtures manufactured from neutral alcohol (essentially free of higher alcohols due to highly rectified distillation) and a malt whisky to provide flavour. The concentrations of higher alcohols in such products are based on the equivalent concentrations in the malt whisky component diluted by the neutral alcohol component. As mentioned earlier, such national whiskies are disqualified as whisky, in for example the European Union, as they contain alcohol distilled at >94.8% and/or alcohol not fermented from cereals and/or alcohol not subjected to 3 years maturation. These products are popular legal products in their home markets but are distinct from imported and locally bottled Scotch whisky.

Key analytical features of Scotch whisky

The Scotch Whisky Regulations 2009[44] define elements of the manufacturing process, which may then be associated with general analytical characteristics of the final product. These elements include the requirements that Scotch whisky must be:

1. Fermented from only cereals, yeast and water
2. Distilled at <94.8% vol. alcoholic strength
3. Matured in oak casks for not less than 3 years
4. No additions may be present other than plain caramel colouring and water
5. The product must be bottled for sale at not less than 40% vol. alcoholic strength

Fermentation from non-cereal sources results in different concentrations of certain congeners. For example, distilled spirits fermented from cane sugar and molasses yield consistently low methanol concentrations, while spirits fermented from grape and fruit can result in methanol concentrations that generally exceed 35 g/100 litres absolute alcohol[24,41].

Scotch whisky distillations, where the alcohol is taken from a continuous distillation column at <94.8% vol. alcoholic strength, result in the recovery of n-propanol and isobutanol. Neutral alcohol stills where the final distillation strength is >96% vol. alcoholic strength have additional rectification columns and are designed to produce ethanol with little if any traces of higher alcohols. Therefore suspect whisky samples containing abnormally low concentrations of higher alcohols, while exhibiting ratios of 2- + 3-methyl butanol/isobutanol typical of malt whisky, can arise from compounding neutral spirit distilled at >96% vol. alcoholic strength and malt whisky.

Maturation in oak casks results in the presence of certain congeners with consistent ratios. The absence of these congeners or their presence in abnormal proportions is inconsistent with Scotch whisky maturation and can indicate the addition of a flavouring.

Table VI. Major volatile congener ranges for single bottled samples of national whiskies from India, Argentina, Brazil, Colombia and Venezuela.[a]

			g / 100 litres absolute alcohol											
National whiskies from	No. of brands surveyed		Acetaldehyde	Methanol	Ethyl acetate	n-Propanol (p)	Isobutanol (b)	2-Methylbutanol	3-Methylbutanol	2- + 3-Methylbutanol (a)	(a) / (b)[b]	3-Methylbutanol / 2-methylbutanol	p + b[c]	p + b + a[d]
India	16	Min.	1	0.6	0.1	0	1	0	2	0	0.1	1.4	1	3
		Avg.	4.3	4.2	6.4	11	7	5	17	18	2.8	8.7	18	36
		Max.	10	25.0	27.6	39	15	15	49	63	8.6	59.0	45	108
Argentina	2	Sample 1	6.8	8.4	28	16	24	16	40	56	2.3	2.5	40	96
		Sample 2	7.6	6.8	41	16	26	17	45	62	2.4	2.6	42	104
Brazil	5	Min.	5	3.6	12	2	7	21	62	27	2.4	2.6	9	35
		Avg.	6.9	5.9	18	16	31	24	68	83	2.9	2.9	47	130
		Max.	7.8	8	25	41	43	29	76	116	3.7	3.1	74	179
Colombia	4	Min.	1.6	2.0	1.0	1.0	3.0	nm[e]	nm	8.0	2.3	nm	5.0	12.0
		Avg.	3.3	2.4	2.0	2.0	6.0	nm	nm	18.0	3.0	nm	9.0	27.0
		Max.	4.9	2.6	3.0	5.0	9.0	nm	nm	23.0	3.6	nm	14.0	35.0
Venezuela	4	Min.	2.9	2.8	4	8	10	7	18	25	2.6	2.5	22	47
		Avg.	6	7.3	19	12	2	12	30	48	2.4	2.6	32	79
		Max.	10.0	11.0	43	16	25	15	38	63	2.7	2.7	40	110

[a] Sums and ratios are calculated from the results for each individual sample.
[b] Total 2- and 3-methylbutanol / isobutyl alcohol.
[c] n-Propanol + isobutanol.
[d] n-Propanol + isobutanol + 2- and 3-methyl butanol.
[e] Not measured.

In summary, this analytical survey of Scotch whisky has provided a data set with maximum and minimum values of the bottled Malt and Grain Scotch Whiskies examined within which may be placed all the examples of bottled Blended Scotch whiskies. The bourbon and Canadian whiskies analysed exist outside this data set, as do the examples of national whiskies from India and Latin America. There is some overlap in the ranges of results between Scotch whisky and Irish whiskey.

From this reference data for genuine Scotch whiskies, it was possible to select key analytical characteristics of bottled Scotch whisky that may be used in detecting falsely described products. These key analytical characteristics of Scotch whisky included:

1. Real alcoholic strength is not less than 40% vol. (as required by definition).
2. Obscuration of alcoholic strength was found to be not more than 0.4% vol. This low level of obscuration is due to the absence of additives in Scotch whisky and the low levels of solids derived from the maturation process.
3. Methanol concentration is not >25 g/100 litres absolute alcohol. This low level of methanol in Scotch whisky is due to the use of cereals in fermentation. Wheat and barley result in methanol concentrations normally <17 g/100 litres absolute alcohol and maize (corn) results in concentrations <25 g/100 litres absolute alcohol.
4. The sum of n-propanol and isobutanol concentrations in Grain, Malt and thus Blended Scotch whiskies as found in this survey of bottled products was at least 97 g/100 litres absolute alcohol.
5. The ratio of 2- + 3-methyl butanol/isobutanol in Malt Scotch whisky was found to be not less than 1.9 and not more than 3.5.
6. The ratio of 3-methyl butanol/2-methyl butanol in Malt, Grain and Blended Scotch whisky was found to be not less than 2.2 and not more than 3.5.
7. The principal sugars are glucose and fructose at trace concentrations. Their total concentrations (expressed as the sum of glucose, fructose and sucrose) in the Blended Scotch whisky samples was found to be less than 500 mg/litre. Any sucrose that may be present was found at extremely low levels (<5 mg/litre) and was always significantly less than glucose and fructose. The ratio of sucrose/(glucose + fructose) was always <0.1 and typically between zero and 0.01.
8. A range of congeners is indicative of the Scotch whisky maturation process. These congeners are normally present in Scotch whisky at consistent ratios to each other and at concentrations that increase with maturation time (representing whisky age). The ratio of vanillin/syringaldehyde is a useful diagnostic (at between 0.4 and 0.6 in the survey of bottled blended Scotch whiskies).
9. Sensory assessment. Scotch whiskies have sensory characteristics against which whisky suspect products may be compared. The following questions should be asked:
   - Does the suspect sample have the sensory characteristics of Scotch whisky?
   - Are there any aromas present that are not normally associated with Scotch whisky?

Case studies in the generic authenticity analysis of suspect Scotch whisky

This section describes case studies where the analytical results for 18 suspect bottled products (Tables VII and VIII) have been referred back to the known results on genuine Scotch whiskies in order to determine generic authenticity. The key results which allow conclusions to be made on each suspect product's authenticity are discussed.

Case studies 1–3

Products 1–3 each exhibited alcoholic strengths at 40% vol., the sensory characteristics of Scotch whiskies and analytical profiles within the ranges shown for Scotch whiskies in Table I. These products were therefore consistent with Scotch whisky.

Case study 4

Product 4 was disqualified as Scotch Whisky and whisky under the EU Regulation due to firstly its high methanol concentration at 125 g/100 litres absolute alcohol (Table VII), which was well above the range encountered in alcohol fermented from cereals. Secondly, the absence of higher alcohol congeners that are present in whiskies distilled at <94.8% vol. alcoholic strength was abnormal. The results suggested it was fermented from a substrate which naturally produces relatively high methanol concentrations (such as grapes) and rectified at >94.8% vol. alcoholic strength in a distillation column designed to eliminate natural higher alcohols from the distillate.

Case study 5

Product 5 was disqualified as a Scotch whisky (and whisky under the EU Regulation) due to its high methanol concentration at 35 g/100 litres absolute alcohol indicating the presence of non-cereal based alcohol. Secondly, the concentrations of the higher alcohols, n-propanol and isobutanol, were well below the minimum normally encountered in Scotch whisky, while the ratio of 2- + 3-methyl butanol/isobutyl alcohol at 2.5 was typical of Malt Scotch whisky (Table VII). The results were consistent with a mixture of approximately 75% neutral alcohol (probably fermented from grapes due to its relatively high methanol concentration) with approximately 25% Malt Scotch whisky to imitate the sensory characteristics of Scotch whisky.

Case study 6

Product 6 was disqualified as a Scotch whisky due to its volatile congener concentrations (including methanol, n-propanol and isobutanol) each being well below the ranges normally encountered in Scotch whisky. However, the ratio of 2- + 3-methyl butanol/isobutyl alcohol at 2.8 was typical of Malt Scotch whisky (Table VII). The results were consistent with approximately 85–90% neutral alcohol with approximately 10–15% Malt Scotch whisky to imitate the sensory characteristics of Scotch whisky.

Case study 7

Product 7 was disqualified as Scotch whisky and whisky under the EU Regulation due to its very high

Table VII. Profiles of major volatile congeners in eighteen products described as or indicated to be Scotch whisky.[a]

Case study	Described or indicated as	Origin	g / 100 litres absolute alcohol												Conclusion
			Acetal-dehyde	Meth-anol	Ethyl acetate	n-Pro-panol (p)	Iso-butanol (b)	2-Methyl-butanol	3-Methyl-butanol	2- + 3-Methyl-butanol (a)	(a) / (b)[b]	3-Methyl-butanol / 2-methyl-butanol	p + b[c]	p + b + a[d]	
1	Scotch whisky	Spain	3.4	6.8	20.9	99.1	56.3	9.9	24.3	34.2	0.6	2.5	155	190	genuine
2	Scotch whisky	Northern Cyprus	5.8	6.1	19.8	84.9	58.6	19	45.3	64.2	1.1	2.4	143	208	genuine
3	Scotch whisky	Taiwan	8.0	4.2	19.9	44.3	67.9	17.4	43.0	60.4	0.9	2.5	112	173	genuine
4	Whisky	Cyprus	1.5	125	3.5	<0.5	<0.5	<0.5	<0.5	<0.5	nm[e]	nm	<0.5	<0.5	not genuine
5	Scotch whisky	Italy	4.6	35.0	15.0	12.0	20.0	nm	nm	50.0	2.5	nm	32	82	not genuine
6	Scotch whisky	Taiwan	3.1	0.6	4.6	7.4	8.8	5.6	19.0	24.6	2.8	3.4	16	41	not genuine
7	Scotch malt whisky	Taiwan	2.4	450	3.2	8.5	32.0	2.5	11.0	13.5	0.4	4.4	41	54	not genuine[f]
8	Whisky	Taiwan	4.1	9.5	2.3	4.3	8.7	12.0	56.0	68.0	7.8	4.7	13	81	not genuine[f]
9	Scotch malt whisky	Taiwan	8.0	6.0	41.0	52.0	83.0	52.0	130	182	2.2	2.5	135	317	not genuine[f]
10	Scotch whisky, 15 years old	Taiwan	13.0	6.4	15.0	88.0	61.0	14.0	37.0	51.0	0.8	2.6	149	200	not genuine[f]
11	Whisky	France	1.7	12.0	2.7	1.5	4.6	5.4	19.0	24.4	5.3	3.5	6	31	not genuine[f]
12	Scotch whisky	Jordan	9.3	1.4	54.0	6.8	26.0	15.0	51.0	66.0	2.5	3.4	33	99	not genuine[f]
13	Whisky	Colombia	4.8	1.7	5.5	9.5	31.0	18.0	40.0	58.0	1.9	2.2	41	99	not genuine
14	Whisky	France	6.0	9.2	5.4	3.0	5.3	3.6	9.6	13.2	2.5	2.7	8	22	not genuine[f]
15	Whisky, 18 years old	France	3.6	3.2	4.3	3.8	8.2	4.7	13.2	17.9	2.2	2.8	12	30	not genuine[f]
16	Whisky	Bulgaria	5.4	2.2	20.0	23.0	38.0	11.0	41.0	52.0	1.4	3.7	61	113	not genuine[f]
17	Whisky	Taiwan	4.9	1.0	37.0	0.6	32.7	0.6	151	152	4.6	252	34	185	not genuine[f]
18	Scotch whisky, 5 years old	Italy	5.6	3.7	6.9	5.5	12.0	8.1	21.0	29.1	2.4	2.6	18	47	not genuine[f]

[a] Sums and ratios are calculated from the results for each individual sample. Limit of detection = 0.5 g / 100 litres absolute alcohol.
[b] Total 2- and 3-methylbutanol / isobutyl alcohol.
[c] n-Propanol + isobutanol.
[d] n-Propanol + isobutanol + 2- and 3-methyl butanol.
[e] Not measured.
[f] See text for more data on analytical result abnormalities.

Table VIII. Profile of furfural and four maturation congeners in seven brands described or indicated to be Scotch whisky.[a]

Case study	g / 1,000 litres absolute alcohol							
	Furfural	Vanillic acid	Syringic acid	Vanillin	Syringaldehyde	Total[b]	Vanillin / syringaldehyde	Vanillin / vanillic acid
8	1.0	0.8	2.0	7.5	5.0	15.3	1.5	9.4
10	1.4	0.4	1.5	1.2	1.9	5.0	0.6	3.0
11	0.7	2.2	0.9	17.9	1.1	22.1	16.3	8.1
12	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	nm[c]	nm
15	4.0	0.3	0.6	0.4	0.9	2.2	0.4	1.3
16	1.1	<0.1	<0.1	<0.1	0.4	0.4	<0.1	nm
18	0.7	0.3	1.4	0.5	1.4	3.6	0.3	nm

[a] Sums and ratios are calculated from the results for each individual sample. Limit of detection = 0.1 mg / litre.
[b] Sum of vanillic acid, syringic acid, vanillin and syringealdehyde.
[c] Not measured.

methanol concentration, at 450 g/100 litres absolute alcohol (Table VII), being far above the range encountered in alcohol fermented from cereals. In addition, the higher alcohol concentrations were well below the ranges normally encountered in Malt Scotch whisky, (which it claimed to be on the label) and there was a very unnaturally high concentration of n-butanol at 111 g/100 litres absolute alcohol (compared to typically <1 g/100 litres absolute alcohol in Scotch whisky). This abnormal n-butanol concentration may indicate its use as a solvent in an added flavouring.

Case study 8

Product 8 was disqualified as Scotch Whisky and whisky under the EU Regulation. Its abnormally low n-propanol and isobutyl alcohol concentrations, which were each well below the minimum levels normally found in Scotch Whisky, had a combined total of only 13 g/100 litres absolute alcohol. The abnormally high 2- + 3-methyl butanol/isobutanol ratio at 7.8 and the abnormally high 3-methyl butanol/2-methyl butanol ratio at 4.7 (Table VII) were both outside normal whisky ranges. In addition, there was an abnormal profile of maturation congeners (Table VIII), which was atypical of the normal effect of maturation in oak casks (Table IV). The abnormal maturation congener profile was demonstrated by the relatively high concentration of vanillin, with the ratio of vanillin/syringaldehyde at 1.5 and the ratio of vanillin/vanillic acid at 9.4, whereas the maximum ratios found in the survey of bottled blended Scotch whiskies were 0.6 and 3.1, respectively (Table III). The results suggested use of non-cereal alcohol and flavour additives containing proportionately high levels of vanillin compared to other maturation congeners.

Case study 9

Product 9 had a volatile congener profile consistent with Malt Scotch whisky. However, the product contained sugars (as glucose, fructose and sucrose) totalling 4,000 mg/litre, with a sucrose level (at 1,700 mg/litre) above the respective levels of glucose and fructose. This suggested that sugars had been used as an additive in a product where additives are not permitted.

Case study 10

Product 10 had a volatile congener profile consistent with a Blended Scotch whisky with approximately 25% malt whisky. The label claimed that it was a "Scotch whisky up to 15 years old". Whilst the product may have contained some 15 year old whisky, the levels of the maturation congeners were lower than would be expected for all the component whiskies being of this age. The relevant laws require an age claim to refer to the youngest, and not the oldest whisky in a blend.

Case study 11

Product 11 was disqualified as Scotch Whisky and whisky under the EU Regulation. The levels of n-propanol and isobutanol were well below the ranges normally encountered in Scotch Whisky, while the ratio of 2- + 3-methyl butanol/isobutanol at 5.3 (Table VII) was above the ranges encountered in Scotch whisky. In addition, there was an abnormally high vanillin concentration compared to the rest of the maturation congeners and resulting high vanillin/syringaldehyde and vanillin/vanillic acid ratios (Table VIII). This was not consistent with normal maturation in oak casks and indicated that flavour additives containing vanillin were present.

Case study 12

Product 12 was disqualified as a Scotch whisky. The alcoholic strength was only 29.45% vol., whereas the minimum alcoholic strength for Scotch whisky is 40% vol. Further, the low levels of volatile congeners, particularly n-propanol and isobutanol totalling only 32.8 g/100 litres absolute alcohol (Table VII) compared to the minimum sum of n-propanol and isobutanol in the Blended Scotch whisky survey of 97 g/100 litres absolute alcohol. The n-butanol concentration was abnormally high for cereal alcohol at 4.9 g/100 litres absolute alcohol, whereas the methanol concentration at 1.4 g/100 litres absolute alcohol was abnormally low for cereal alcohol. Maturation congeners, indicative of maturation in oak casks, were not detected with a limit of detection at <0.1 g/1,000 litres absolute alcohol (Table VIII).

Case study 13

Product 13 was disqualified as a Scotch whisky due the low levels of major volatile congeners, particularly the sum of n-propanol and isobutanol at 41 g/100 litres absolute alcohol (Table VII). As quoted above, the minimum value encountered in the Blended Scotch whisky survey was 97 g/100 litres absolute alcohol. This low value plus the ratio of 2- +3-methyl butanol/isobutyl alcohol at 1.9, the 3-methyl butanol/2-methyl butanol ratio at 2.2 and the 2- + 3-methyl butanol concentrations at 58 g/100 litres absolute alcohol was consistent with a product based on neutral alcohol distilled at >96% vol. alcoholic strength flavoured with approximately 30% malt whisky. This observation was further reflected by the methanol concentration at 1.7 g/100 litres absolute alcohol, which was consistent with a neutral alcohol with a very low methanol concentration mixed with malt whisky which has a typical average methanol concentration at 6.5 g/100 litres absolute alcohol (Table I).

Case study 14

Product 14 was disqualified as a Scotch whisky due to the very low congener levels (the individual levels of n-propanol and isobutanol were well below the minimum levels normally found in Scotch Whisky and their combined total was only 8 g/100 litres absolute alcohol). Further, the presence of 3,000 mg/litre sugars, with the sucrose level at 2,000 mg/litre greater than either glucose or fructose, was an indication of added sweetening. The ratio of 2- +3-methyl butanol/isobutanol at 2.5 and 3-methyl butanol/2-methyl butanol at 2.7 and the 2- +3-methyl butanol concentration at 13 g/100 litres absolute alcohol (Table VII) was consistent with the presence of approximately 5–10% malt whisky added to neutral alcohol.

Case study 15

Product 15 was disqualified as Scotch Whisky. The individual levels of n-propanol and isobutanol were well below the minimum levels normally found in Scotch Whisky and their combined total was only 12 g/100 litres absolute alcohol. The ratio of isoamyl alcohol/isobutanol at 2.2 and 3-methyl butanol/2-methyl butanol at 2.8 and the isoamyl alcohol concentration at 17.9 g/100 litres absolute alcohol (Table VII) were consistent with approximately 5–10% malt whisky mixed with neutral alcohol distilled at >96% vol. The four maturation congeners in this product which claimed to have been matured for 18 years added up to 2.2 g/1,000 litres absolute alcohol (Table VIII). However, three brands of genuine 12 year old Scotch whisky (brands 17–19 in Table III) showed corresponding values between 9.7 and 13.4 g/1,000 litres absolute alcohol. Therefore, the low levels of maturation congeners suggested that the contents had not been matured in oak casks for 18 years as claimed on the label.

Case study 16

Product 16 had concentrations in its major volatile congener profile (Table VII) lower than would be expected. The sum of n-propanol and isobutanol at 61 g/100 litres absolute alcohol was suspicious as it was significantly below the 97 g/100 litres absolute alcohol encountered in genuine bottled product. Further, the methanol concentration at 2.2 g/100 litres absolute alcohol was below the minimum encountered in Scotch whisky bottle data sets (Table I). The ratio of 2- + 3-methyl butanol/isobutanol could be consistent with malt whisky at 1.4. There were abnormally low levels of maturation congeners in Product 16 (Table VIII), the total at 0.4 g/1,000 litres absolute alcohol being <15% of the minimum (2.8 g/1,000 litres absolute alcohol) encountered in Blended Scotch whisky (Table III). These results were consistent with the use of neutral alcohol distilled at >96% vol., alcohol that had not been subject to a minimum of 3 years maturation in oak casks mixed with approximately 25% malt whisky. These conclusions led to the disqualification of product 16 as Scotch whisky.

Case study 17

Product 17 was disqualified as Scotch whisky and whisky under the EU Regulation due to its very abnormal volatile congener profile and the presence of various additives. The n-propanol was barely detectable at 0.6, isobutanol was 32.7 and the sum was 33.3 g/100 litres absolute alcohol. The ratio of 3-methyl butanol/2-methyl butanol was approximately 250 (Table VII). This indicated that 3-methyl butanol was present in this product not as a result of normal cereal fermentation but as a result of it being present as a component in an added flavouring. Sugars were present at 5,000 mg/litre, the major sugar present being sucrose at 4200 mg/litre, indicating the addition of sweetening.

Case study 18

Product 18 was disqualified as a Scotch whisky due to its low congener concentrations, particularly n-propanol and isobutanol (Table VII) and the presence of substances not found naturally in Scotch Whisky. Whilst the ratios of 2- +3-methyl butanol/isobutanol at 2.4 and 3-methyl butanol/2-methyl butanol at 2.6 were consistent with Malt Scotch whisky, the low overall volatile congener concentrations indicated that the product was not wholly Scotch whisky. In addition, sensory analysis indicated pungent sweet, estery and aniseed aromas. Subsequent gas chromatography-mass spectrometric analyses found trans-anethole (at 620 µg/litre), p-anisaldehyde (at 230 µg/litre) and limonene (at 90 µg/litre). Anethole and anisaldehyde are not found in whisky and natural levels of limonene in whisky are normally 100 fold less than the level found[26,27]. The level of maturation congeners was suspiciously low (Table VIII) for a product with a five year age claim. The results were consistent with a product composed of approximately 85% neutral alcohol distilled at >96% vol. alcoholic strength mixed with approximately 15% malt whisky and added flavourings.

Proposed analytical strategy

The primary purpose of the analyses described in the preceding sections is to determine if suspect liquids are consistent with Scotch whisky. It is not to determine their origin, although the results may help in suggesting potential origins. The following protocol is proposed for checking the generic authenticity of suspect products claiming to be Scotch whisky.

Firstly, note the claims on the product label.

1. Does the product claim to be Scotch whisky?
2. Does the product claim to be Malt (Single or Blended), Grain (Single or Blended) or Blended Scotch whisky?
3. If not described as Scotch whisky, is it described as whisky?
4. What is the declared alcoholic strength?
5. Is there a specific or suggested age claim?

If the determination of authenticity by analysis is required, then follow the under noted analytical sequence while referring to the key analytical features of Scotch whisky described in the earlier section.

1. Check sensory properties by nose, do not taste
2. Determine apparent alcoholic strength, % vol.
3. Determine real alcoholic strength, % vol.
4. Determine obscuration, % vol.
5. Determine major volatile congeners
6. Determine maturation congeners
7. Determine residual solids and sugars
8. Look for the presence of compounds not normally present in Scotch whisky and identify absent compounds that are normally present in Scotch whisky.

When it is considered that a suspect product is falsely described, it is clearly desirable that the failure feature is not marginal. Unless there is one feature that clearly disqualifies a product, failure on more than one feature is clearly desirable. Once the analyst is satisfied that a suspect product fails to conform to the key analytical features of Scotch whisky, evidence may be presented to disqualify the product.

Other techniques for further consideration and further method development

All potable distilled spirits must be made from alcohol of agricultural origin. Synthetic alcohol is not permitted. On the rare occasions that use of synthetic alcohol is suspected, ^{14}C analysis by liquid scintillation counting may be employed[30]. Synthetic alcohol from fossil fuels has no remaining ^{14}C, while cereal alcohol will have very low, yet detectable ^{14}C levels reflecting the environmental carbon dioxide levels at the time the cereal was grown.

Other developing isotopic techniques include site-specific natural isotope fractionation nuclear magnetic resonance (SNIF-NMR) where $^{2}H/^{1}H$ ratios are measured on the methyl and methylene hydrogens in ethanol. Methyl hydrogens are primarily influenced by the fermented carbohydrate and methylene hydrogens are influenced by the ratio in the fermentation water[29]. $^{13}C/^{12}C$ ratios are influenced by the photosynthetic pathway used by the carbohydrate to assimilate carbon dioxide[34]. While barley, wheat and beet use the C_3 (Calvin) pathway, maize (corn) and cane use the C_4 (Hatch-Slack) pathway. Whilst this technique may be useful in relation to Malt Scotch whisky (which is produced from malted barley), many Scotch whiskies are made from both barley and maize resulting in this technique being of limited use in determining the authenticity of these products.

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