Analytical strategies to confirm Scotch Whisky authenticity

Article *in* The Analyst · August 1994

DOI: 10.1039/an9941901741

4 authors, including:

Ross Aylott
Aylott Scientific
21 PUBLICATIONS **194** CITATIONS
SEE PROFILE

David A Walker
University College London Hospitals NHS Fo…
24 PUBLICATIONS **184** CITATIONS
SEE PROFILE

Analytical Strategies to Confirm Scotch Whisky Authenticity*

Ross I. Aylott, Angus H. Clyne, Anthony P. Fox and David A. Walker

United Distillers, International Technological Services, Glenochil, Menstrie, Clackmannanshire, UK FK11 7ES

Analytical methods are required by trading standards and regulatory authorities to confirm the authenticity of Scotch whisky brands. The characteristics of Scotch whisky are strongly influenced by the cereals used in fermentation and by distillation, maturation and blending regimes. This leads to characteristic analytical profiles for various congeners, which can be used as reference points in authenticity analysis. Scotch whisky higher-alcohol profiles for specific brands were shown to be very consistent over many production batches and so their gas-chromatographic determination, together with the presence of other specific congeners, offered an effective approach to authenticity analysis. Pyrolysis–mass spectrometry followed by multivariate analysis of the resulting mass spectra enabled non-authentic samples to be discriminated from the authentic product. Hence, an increasingly sophisticated portfolio of analytical strategies are available in order to confirm brand authenticity.

Keywords: Scotch whisky; authenticity; chromatography; pyrolysis–mass spectrometry

Introduction

Authenticity issues affect a wide variety of products including foods and beverages. It is a requirement in the laws of many countries that what is described on the product label must be what is sold therein. In the UK, responsibility for ensuring product authenticity falls to local authority Trading Standards departments under provisions of the Trade Descriptions Act 1968 and the Food Safety Act 1990.[1–3] Typical authenticity issues associated with Scotch whisky involve on-trade cases (in bars) where one brand may have been deliberately and illegally substituted by another, usually cheaper, brand. Samples are normally collected by Trading Standards officers, either as a part of their routine sampling or as a result of a customer complaint. The resulting samples are then subjected to product contents analysis in order to check brand authenticity. Such analyses are normally conducted by the Public Analyst service or in the laboratories of brand-owning companies.

Whisky is legally defined under European Community Council Regulation No. 1576/89[4] and Scotch whisky is further defined in the UK under the Scotch Whisky Act 1988[5] and the Scotch Whisky Order 1990.[6] Definitions also exist in the USA,[7] Canada,[8] Australia[9] and other countries, many of which make reference to UK legislation.

There are two distinct types of Scotch whisky, malt whisky and grain whisky. Most of the well known brands of Scotch whisky are blended from many individual malt and grain whiskies. Scotch malt whisky is produced from the fermentation of 100% malted barley followed by batch distillation in pot stills.[10] Scotch grain whisky is produced from the fermentation of various cereals such as wheat and maize, together with a small amount of malted barley, followed by continuous distillation.[11] In 1992, there were 95 malt and eight grain distilleries in production in Scotland.[12]

After distillation, both malt and grain whiskies are matured in oak casks for at least 3 years and often over 12 years. The maturation process enables the pungent character of new-make spirit to develop into the more agreeable and well known character of Scotch whisky.[13] The casks used for Scotch whisky maturation are made from both American and European oak. Flavour development during maturation involves processes of congener addition, congener reduction and congener production.[14]

Following maturation the blender selects specific malt and grain whiskies to produce blended Scotch whisky with a definite recognizable character. The final stages in the production process involve reducing the alcoholic strength from final maturation strength (typically 60–70% v/v) to bottling strength (typically 40–43% v/v) by the addition of water, filtration to ensure a clear bright product and the optional addition of a small amount of spirit caramel to provide a consistent colour.

The resulting product is very complex, with several hundred known components (or congeners) including alcohols, aldehydes, acids, esters, phenols and carbonyl-, nitrogen- and sulfur-containing compounds.[15–17] The major congeners in Scotch whisky are the higher alcohols, namely *n*-propanol, isobutanol and isoamyl alcohol, and these are readily determined by gas chromatography (GC) with use of a polar stationary phase. These congeners are present in most distilled spirits and their concentrations can form the basis for differentiating between categories of spirits such as whiskies, rums and brandies.[18] In addition, there is a major analytical difference between the higher-alcohol profiles of Scotch grain and malt whiskies with grain whiskies having very little isoamyl alcohol,[19,20] due to this and other higher-boiling congeners being removed from the spirit during continuous distillation. Capillary-column GC can be used for the determination of many volatile trace congeners,[15] while derivatization before GC has been used for volatile phenols[21] and high-performance liquid chromatography (HPLC) has been used for the non-volatile cask-extracted congeners resulting from the alcoholysis of lignin in the oak cask during maturation.[22]

Brand authenticity is normally checked by comparing the alcoholic strength and higher alcohol profile with those of known reference samples. Although various workers have profiled higher alcohols in Scotch whisky[18–20] there is little information on the concentration ranges exhibited by specific brands. There are over 500 brands of Scotch whisky listed for sale in the UK.[23] Each brand will exhibit its own natural analytical range and, given to the large number of available brands, many will exhibit coincident analytical properties, causing overlap of results. The ability to confirm brand

* Presented at Euroanalysis VIII, Edinburgh, Scotland, UK, September 5–11, 1993.

authenticity is, therefore, dependent on reference to that product's analytical profile or fingerprint.

This paper describes GC and liquid-chromatographic procedures for Scotch whisky analysis and demonstrates how the higher-alcohol profile, together with additional congener data associated with colour, malt and maturation congeners, can be used to provide a general scheme for authenticity analysis. In addition, pyrolysis–mass spectrometry (Py–MS) was applied for authenticity analysis. The potential for Py–MS has been investigated for the characterization of a range of materials, including bacteria,[24] fruit juices,[25] adhesives,[26] and of Scotch whisky maturation quality.[27] In this paper, Py–MS was used to compare unknown samples against a reference product.

Experimental

Apparent alcoholic strength was measured directly with an Anton Paar (Graz, Austria) Model DMA 55 precision density meter. Sample tint (or colour) was measured with a Cecil (Cambridge, UK) Model 5501 spectrophotometer to yield a reading equivalent to absorbance (×100) at 500 nm in a 1 cm cell.

Methanol, acetaldehyde, ethyl acetate and higher-alcohol determinations were conducted by using direct-injection (1 μl) packed-column GC (Philips PU4550, Cambridge, UK), with flame ionization detection, in the presence of pentan-3-ol as internal standard. A 2 m × 2 mm i.d. glass column packed with 5% of Carbowax 20M on Carbopak B, 80–120 mesh (Supelco, Bellefonte, PA, USA), was used, temperature-programmed from 70 to 160 °C at 6 °C min^{-1} with the injector at 160 °C and the detector at 250 °C.[28]

Qualitative capillary GC–MS was conducted in electron-impact mode at 70 eV on a Hewlett-Packard (Avondale, PA, USA) 5970 mass selective detector interfaced with a Hewlett-Packard 5890 gas chromatograph. The whisky sample (50 ml) was diluted to 20% v/v with water and extracted with Freon 11 (chlorotrifluoromethane) (50 ml). The extract was then reduced in volume to 1 ml, by using a three-bubble Snyder air condenser, with gentle warming (30 °C). A sample of the extract (1 μl) was submitted to GC on a 30 m × 0.25 mm i.d. DB Wax column (J. & W. Scientific, Rancho Cordova, CA, USA) with vapourizing splitless injection. The temperature was programmed from 30 to 130 °C at 45 °C min^{-1} and from 130 to 220 °C at 3.5 °C min^{-1}. The resulting mass-spectral data were used to assign peak identities by reference to the Wiley (New York) Mass Spectral Library.

Cask-extractive congeners, together with furfural and 5-hydroxymethylfurfural, were determined by direct-injection (10 μl) reversed-phase HPLC [Varian (Palo Alto, CA, USA) 9011 solvent-delivery system and 9050 variable-wavelength UV/VIS detector] with use of 3,5-dimethoxy-4-hydroxyacetophenone as internal standard. A 25 cm × 4.6 mm i.d. column packed with 5 μm Spherisorb ODS 2 (Phase Separations, Queensferry, Clwyd, UK) was used, together with detection at 280 nm for the first 25 min and at 340 nm thereafter. Scopoletin was determined with reference to an external standard, in the same chromatographic separation, by means of fluorescence detection (Varian Fluorochem II in series with the UV detector) with excitation at 345 nm and emission at 450 nm. The mobile phase was programmed from 10 to 47% B over 30 min and to 65% B over the following 10 min. The mobile phases were as follows: phosphoric acid, 0.5% (A), 0.5% B; methanol, 0.5% (A), 15% (B); acetonitrile, 1% (A), 35% (B); and water, 98% (A), 49.5% (B).

Volatile phenolic congeners were determined by direct-injection (20 μl) reversed-phase HPLC [Gilson (Worthington, OH, USA) pumping system], with 2,3,5-trimethylphenol as internal standard, on a 25 cm × 4.6 mm i.d. column packed with 5 μm Spherisorb ODS 2 and use of fluorescence detection [Waters (Milford, MA, USA) 470 scanning fluorimetric detector] with excitation at 272 nm and emission at 298 nm. The mobile phase was maintained at 15% B for the first 5 min and then programmed to 40% B over the following 30 min. The mobile phases were as follows: acetic acid, 2% (A), 2% (B); acetonitrile, 10% (A), 98% (B); and water, 88% (A), 0% (B).

A single sample preparation was effected for the above three quantitative chromatographic analyses by adding a combined internal-standard solution in 40% ethanol (0.5 ml) to the sample (9 ml) and recording internal standard and sample masses. Congener concentrations were calculated on a mass basis, by means of response factors and peak areas, on a VG (Altrincham, UK) Multichrom data system. Finally, congener concentrations were expressed as g per 100 l of absolute alcohol, this being the normal unit in the alcoholic beverage industry. All chemical standards were obtained from Aldrich (Milwaukee, WI, USA). The target internal-standard concentrations in the analytical solutions were as follows: Pentan-3-ol, 200 μg g^{-1}; 3-5-dimethoxy-4-hydroxyacetophenone, 1.5 μg g^{-1}; and 2,3,5-trimethylphenol, 0.2 μg g^{-1}.

Pyrolysis–mass spectrometry was conducted on a Horizon Instruments (Heathfield, East Sussex, UK) PYMS 200-X system consisting of a Curie-point pyrolyser coupled with a mass spectrometer, with electron-impact ionization at 70 eV. The pyrolysis temperature was 530 °C for 5 s with a 3 μl sample of whisky concentrated 10-fold in a drying-oven at 60 °C. Pyrolysate mass spectra were subjected to principal component and canonical variate analyses with use of a statistical analysis programme based on GENSTAT (MAFF, Rothamsted, UK). Each sample was divided into three sub-samples for triplicate Py–MS analyses, thereby maximizing inter-sample over intra-sample variation.

Results and Discussion

GC and HPLC Analyses

The chromatographic procedures described above resulted in two gas chromatograms and two liquid chromatograms, each of which provided distinct fingerprint information on Scotch whisky. The higher-alcohol chromatogram, shown in Fig. 1, afforded information on those components in blended whisky showing the highest concentrations after water and ethanol. The principle higher alcohols are *n*-propanol, isobutanol and isoamyl alcohol (being the sum of 2- and 3-methylbutanol). This chromatogram also included methanol, acetaldehyde and ethyl acetate and trace congeners such as *n*-butanol and diethyl acetal.

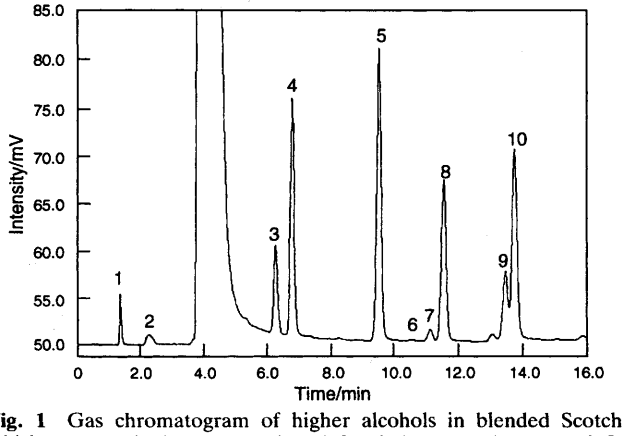

Fig. 1 Gas chromatogram of higher alcohols in blended Scotch whisky: 1, acetaldehyde; 2, methanol; 3, ethyl acetate; 4, propanol; 5, isobutanol; 6, butanol; 7, diethyl acetal; 8, internal standard; 9, 2-methyl butanol; and 10, 3-methyl butanol.

As mentioned earlier, Scotch grain and malt whiskies exhibit different analytical profiles.[19] The continuous distillation process used for grain whisky tends to recover congeners up to the volatility of isobutanol and very little isoamyl alcohol or higher-boiling compounds. Malt whiskies contain the same congeners plus isoamyl alcohol and a large number of higher-boiling compounds. These differences in higher-alcohol profiles are mainly as a result of the greater rectification produced by grain whisky continuous stills compared with that of malt whisky pot stills. As the isoamyl alcohol comes principally from malt whisky, its concentration in a sample reflects the proportion of malt whiskies used in that blend.

The capillary chromatogram for a Scotch whisky extract, as shown in Fig. 2, in combination with MS, allowed the separation and identification of many trace congeners including alcohols, aldehydes, acids and esters. These components predominate in malt whiskies[15–17] and are generally common to most, although present in varying concentrations. However, components produced in post-distillation processes will be present in both malt and grain whiskies. These include cask associated components produced during maturation.[14,29,30] Although the sample illustrated was extracted into a chlorofluorocarbon, *n*-pentane has now been introduced as an alternative extraction solvent with different selectivity.

The cask-extractive liquid chromatogram for Scotch whisky, shown in Fig. 3, provided a fingerprint of those components associated with extraction from the oak cask and interaction with other congeners during the maturation process, together with other congeners such as furfural and 5-hydroxymethylfurfural. Although many of these congeners can be found in the capillary gas chromatogram (Fig. 2), the HPLC analysis allowed both a simpler sample preparation and a simpler chromatogram. The chromatogram for volatile phenolic congeners, shown in Fig. 4, provided a fingerprint of those components associated with the peat used in the production of malted barley. Such components are generally present in malt whiskies such as those from Islay. This HPLC method also permitted a sample-preparation procedure that was simpler than the derivatization–GC approach.[21]

Congener Analyses

The application of these analytical procedures to confirm the authenticity of various blended Scotch whiskies was examined by using analytical data generated from 20 blending batches (or rotations) of five different brands labelled A–E. Data examined included higher-alcohol, cask-extractive and volatile phenolic congener concentrations.

Examination of the results for these brands, as shown in Table 1, showed that each exhibited results within close analytical ranges, thereby demonstrating consistent congener compositions. There were only minor differences in the *n*-propanol and isobutanol concentration ranges for the five brands. Brand A showed the lowest range of isoamyl alcohol concentrations and brand E the highest, reflecting the increasing proportions of malt whiskies present in the blends

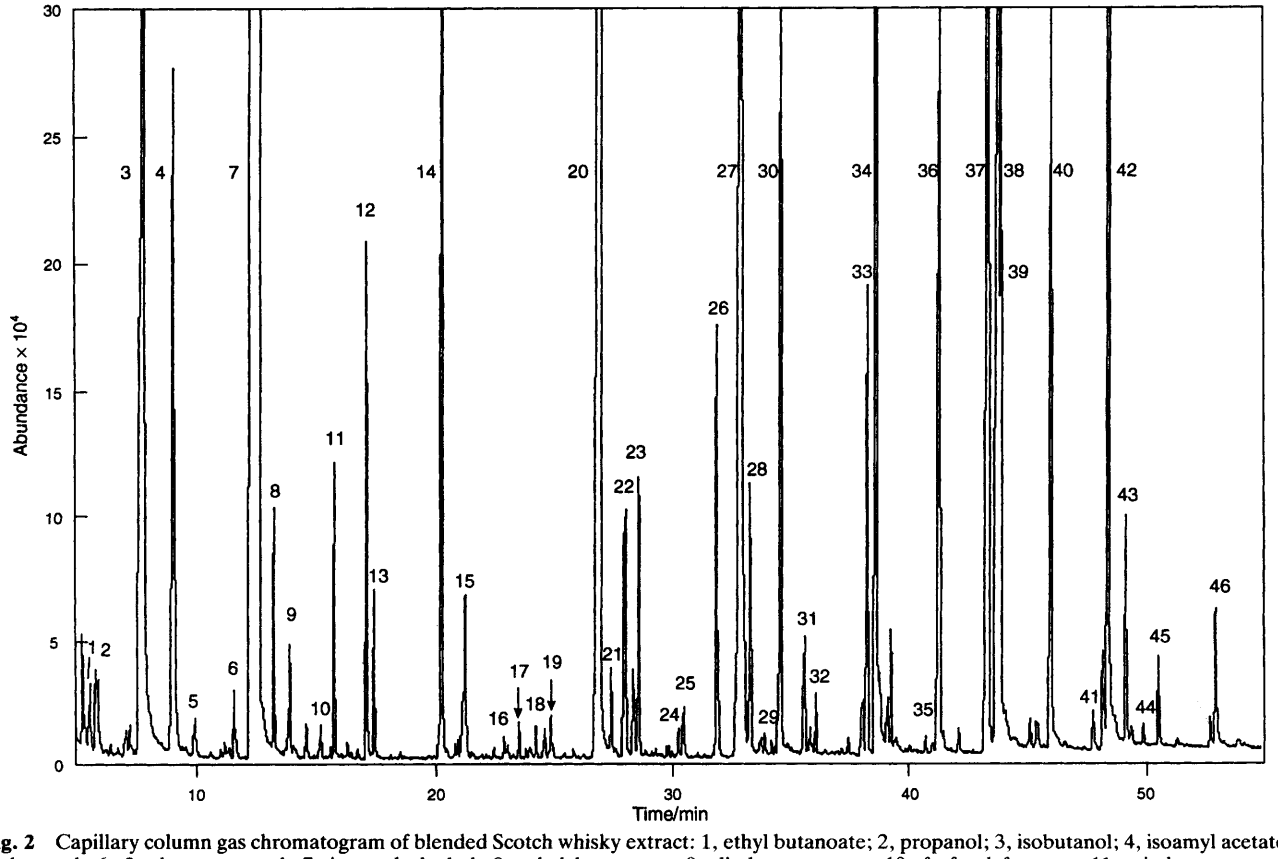

Fig. 2 Capillary column gas chromatogram of blended Scotch whisky extract: 1, ethyl butanoate; 2, propanol; 3, isobutanol; 4, isoamyl acetate; 5, butanol; 6, 3-ethoxypropanal; 7, isoamyl alcohol; 8, ethyl hexanoate; 9, diethoxypropane; 10, furfuryl formate; 11, triethoxypropane; 12, ethyl lactate; 13, hexanol; 14, ethyl octanoate; 15, furfural; 16, acetyl furan; 17, benzaldehyde; 18, ethyl nonanoate; 19, octanol; 20, ethyl decanoate; 21, isoamyl octanoate; 22, diethyl succinate; 23, internal standard; 24, ethyl undecanoate; 25, decanol; 26, β-phenyl ethyl acetate; 27, ethyl dodecanoate; 28, isoamyl decanoate; 29, *trans* oak lactone; 30, β-phenyl ethanol; 31, *cis* oak lactone; 32, dodecanol; 33, ethyl tetradecanoate; 34, octanoic acid; 35, ethyl pentadecanoate; 36, tetradecanol; 37, ethyl hexadecanoate; 38, decanoic acid; 39, ethyl hexadecenoate; 40, hexadecanol; 41, ethyl octadecanoate; 42, dodecanoic acid; 43, ethyl octadecenoic acid; 44, vanillin; 45, β-phenyl ethyl butanoate; and 46, tetradecanoic acid.

going from brand A to brand E. Brands B, C and D each showed similar higher-alcohol ranges and were, therefore, difficult to distinguish from each other on this basis. Brands A and E were easily distinguished from each other, and from brands B, C and D, on the basis of their isoamyl alcohol concentrations. Fig. 5 shows graphically the isoamyl alcohol concentrations in all five brands.

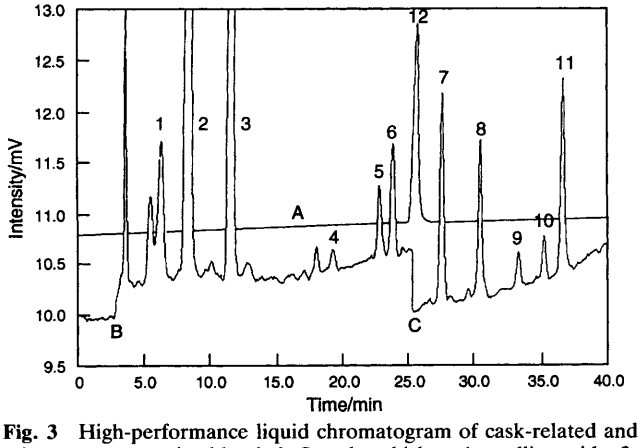

Fig. 3 High-performance liquid chromatogram of cask-related and other congeners in blended Scotch whisky: 1, gallic acid; 2, 5-Hydroxymethyl furfural; 3, furfural; 4 vanillic acid; 5, syringic acid; 6, vanillin; 7, syringealdehyde; 8, internal standard; 9, coniferaldehyde; 10, synapaldehyde; 11, ellagic acid; and 12, scopoletin. A = fluorescence detection, B = UV detection at 280 nm, and C = UV detection at 340 nm.

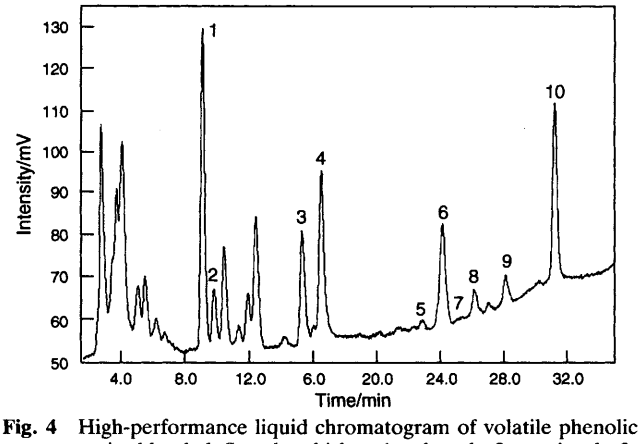

Fig. 4 High-performance liquid chromatogram of volatile phenolic congeners in blended Scotch whisky: 1, phenol; 2, guaiacol; 3, *m*-,*p*-cresol; 4, *o*-Cresol; 5, 3,5-xylenol; 6, 4-ethyl phenol (2,5-xylenol); 7, 4-ethyl guaiacol; 8, 2-ethyl phenol; 9, eugenol; and 10, internal standard.

A similar examination of the results for cask-extractive congeners, represented by scopoletin, showed increasing concentrations going from brand A to brands B, C and D and then to brand E (Table 1). However, only brand A exhibited a distinctly different concentration range from brands B to E, and a large database and caution would be required in using these data (rather than the higher-alcohol data) to separate brand E from the others. The increasing concentrations of isoamyl alcohol and cask-extractive congeners in these five brands demonstrated the increasing proportions of malt whiskies and cask properties, respectively, selected by the blenders for use in these blends.

The phenol concentration in Scotch whisky represents 25% of the total concentration of volatile phenolics as measured by the described HPLC method (Fig. 4). This value is very consistent, ranging from 22 to 26% over 60 samples of brand C. Examination of the phenol concentrations in the five brands again clearly showed that brand A exhibited a distinctly different concentration range from the others, which gave overlapping ranges (Table 1).

Further inspection of the data for brands B, C and D showed that brand B had a lower tint than that of brands B and C, as shown in Fig. 6. These differences in colour were related to the casks in which the whiskies were matured and the small amounts of spirit caramel added to provide consistency. The data to this point separated all, except brands D and E, from each other, the most useful results being the higher-alcohol concentrations and tint measurements. However, the tint of whisky will slowly fade on prolonged exposure to bright light and heat. Caution is, therefore, required in drawing authenticity conclusions on differences in colour intensity.

An application of this approach to brand authenticity checking was then demonstrated by using analytical results from unknown samples Y and Z, which were compared with the four samples of a reference brand, as shown in Table 2. Data for both methanol and higher alcohols were used. The results for sample Y came within the ranges of the reference samples, subject to normal analytical reproducibility, as reported in Table 3, and this suggested that sample Y and the reference samples were from the same brand. However, the results for sample Z fell outside the ranges for three of the congeners in the reference set and suggested that sample Z and the reference samples were not the same brand. The same samples were re-examined by Py–MS, and this work is reported later in the paper.

This chromatographic approach to differentiating products and confirming authenticity is dependent on there being an appropriate number of results within a brand's congener database and for the brand in question to exhibit a consistent analytical profile for the assessment to be conclusive. Clearly, such an approach is only possible for the most common brands within the Public Analyst and brand-owning company labora-

Table 1 Analytical ranges and averages (in parentheses) for samples of each of five Scotch whisky brands labelled A–E

Congener	Unit	Brand A	Brand B	Brand C	Brand D	Brand E
n-Propanol	g per 100 l absolute alcohol	59–78 (69)	56–66 (61)	55–68 (59)	59–65 (62)	54–59 (57)
Isobutanol	g per 100 l absolute alcohol	70–95 (83)	75–89 (80)	76–88 (81)	76–89 (82)	78–89 (84)
Isoamyl alcohol	g per 100 l absolute alcohol	42–46 (44)	62–70 (67)	71–80 (76)	67–78 (71)	90–97 (93)
Scopoletin	ng ml^{-1}	100–290 (180)	350–710 (580)	550–710 (640)	500–750 (600)	550–950 (760)
Phenol	ng ml^{-1}	47–89 (57)	140–195 (167)	100–180 (144)	100–155 (121)	155–205 (177)
Tint	Absorbance (at 500 nm) ×100	10–12 (11)	23–30 (27)	22–29 (25)	17–22 (18)	28–35 (31)

tories. It was not effective for brands C and D, which had similar congeneric fingerprints.

Py–MS

Application of Py–MS, with canonical variate analysis, to brands B, C and D showed that each was clearly differentiated from one another into distinct clusters with 95% confidence boundaries around each group, shown by the circles in Fig. 7. Pyrolysis–MS, unlike congener analysis, was effective in differentiating brands C and D. A similar separation was also obtained by principal-component analysis of the data. Canonical variate analysis, however, offered a greater ability to separate data clusters, and 95% confidence boundaries could be defined with this statistical technique, as illustrated in Figs. 7–10.[29]

Pyrolysis–MS was then used to examine unknown samples Y and Z against the reference brand samples. First, when the pyrolysate mass spectra of the reference samples (labelled U, V, W and Y) were subjected to canonical variate analysis, the four samples produced a single cluster with overlapping confidence boundaries, as shown in Fig. 8.

Second, when the pyrolysate mass spectra of unknown sample Y was introduced into the data set, the data point for sample Y appeared within the cluster for the reference samples, as shown in Fig. 9. This reinforced the earlier observation from the congener analyses that sample Y and the reference samples were in fact the same brand.

Third, when the pyrolysate mass spectra of sample Z were introduced into the data set, the data points for sample Z appeared separate from the cluster for the reference samples, as shown in Fig. 10. This also reinforced the earlier observation from the congener analyses that sample Z was not the same brand as the reference samples.

Finally, the sample bottles and closures used for collecting samples have been shown to influence product analysis, particularly Py–MS. For example, material extracted by the

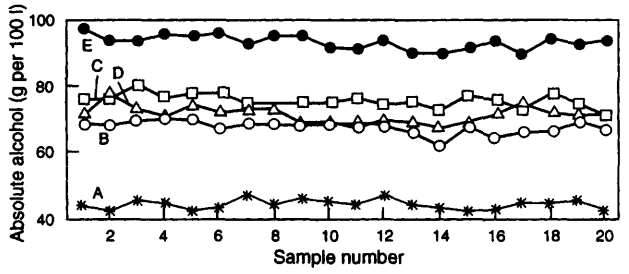

Fig. 5 Isoamyl alcohol concentrations in 20 samples of each of 5 Scotch whisky brands labelled A–E.

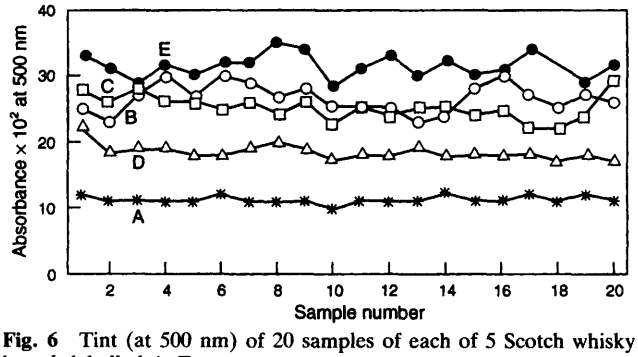

Fig. 6 Tint (at 500 nm) of 20 samples of each of 5 Scotch whisky brands labelled A–E.

Table 2 Congener results for unknown samples Y and Z compared with those for four samples of a reference brand

		Concentration/g per 100 l absolute alcohol			
Sample	*n*	Methanol	*n*-Propanol	Isobutanol	Isoamyl alcohol
Y	1	9.6	66	67	75
Z	1	11	72	75	72
Reference brand	4	9.2–9.8	64–69	67–69	70–74

Table 3 Precision of higher-alcohol analysis expressed as the confidence limit (2σ); about 10 analysis conducted on a sample of brand E

	Concentration/g per 100 l absolute alcohol		
Congener	Range	Mean	Confidence limit (2σ)
Acetaldehyde	9.6–9.9	9.7	±0.2
Methanol	11.7–12.8	12.5	±0.2
Ethyl acetate	39.0–41.8	41.2	±0.8
n-Propanol	69.9–71.9	70.8	±0.7
Isobutanol	91.3–93.2	91.8	±1.1
2-Methylbutanol	25.2–26.8	26.2	±0.7
3-Methylbutanol	66.7–71.8	69.6	±1.3

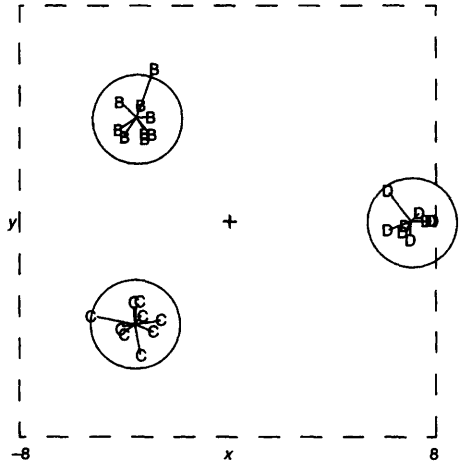

Fig. 7 Pyrolysis–mass spectrometry canonical variate plot of 3 samples of brands B, C and D, each analysed in triplicate.

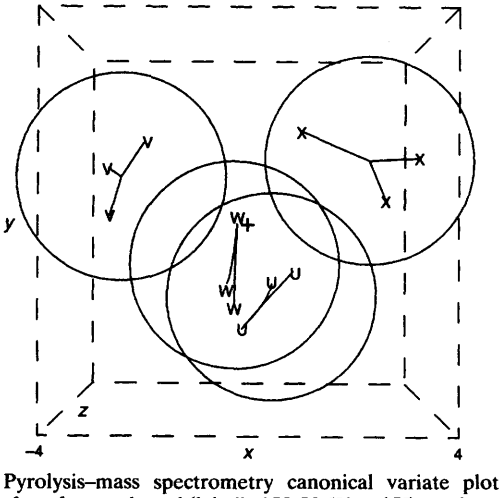

Fig. 8 Pyrolysis–mass spectrometry canonical variate plot of 4 samples of a reference brand (labelled U, V, W and X), each analysed in triplicate.

beverage from inappropriate containers have resulted in ion fragments foreign to the beverage sample, which can distort the resulting canonical variate plots and lead to incorrect grouping. This can give false negative discrimination of unknown and reference samples. This problem was eliminated by use of inert containers.[3] Our experience is that glass or PET [poly(ethylene terephthalate)] bottles with closure containing Melinex (PET film) lined wads are suitably inert.

Conclusions

Higher-alcohol congener analysis provided a valuable method for checking Scotch whisky brand authenticity, with particular attention being paid to methanol, *n*-propanol, isobutanol and isoamyl alcohol concentrations. Further confirmatory data were obtained by reference to cask-extractive and volatile phenolic congeners, together with tint. When checking brand authenticity, the analyst should be aware of the range and consistency of congener results for the brand in question and also be aware of other potential brands that may exhibit similar results. Samples should be collected in suitably inert containers.

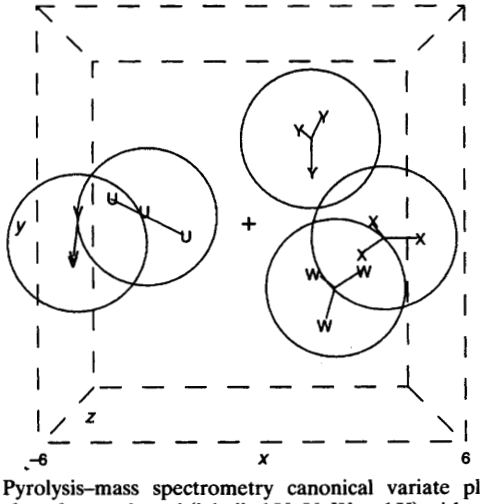

Fig. 9 Pyrolysis–mass spectrometry canonical variate plot of 4 samples of a reference brand (labelled U, V, W and X) with unknown sample Y, each analysed in triplicate.

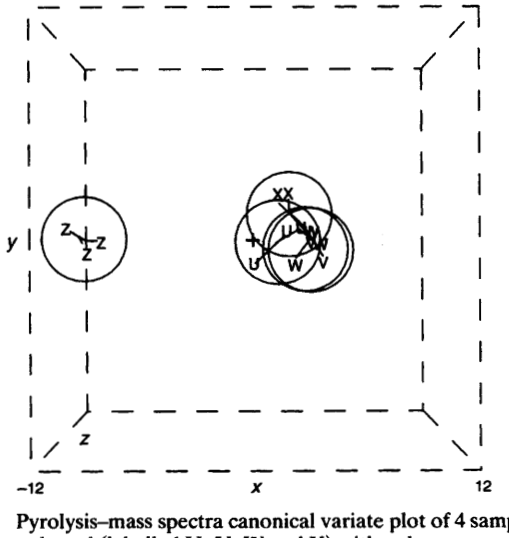

Fig. 10 Pyrolysis–mass spectra canonical variate plot of 4 samples of a reference brand (labelled U, V, W and X) with unknown sample Z, each analysed in triplicate.

Pyrolysis–MS allowed a rapid confirmation of conclusions from chromatographic analysis and has the potential to develop into a useful 'stand alone' technique for authenticity analysis.

References

1 *Trade Descriptions Act 1968*, HM Stationery Office, London, Sect. 3–029.
2 *Food Safety Act 1990*, HM Stationery Office, London, ch. 16, p. 12, Sect. 15.
3 *Food Safety Act 1990, Code of Practice No. 7: Sampling for Analysis or Examination*, HM Stationery Office, London, p. 4.
4 Council Regulation (EEC) No. 1576/89 of 29 May 1989, *Off. J. Eur. Commun.*, 1989, No. L160, p. 1.
5 *Scotch Whisky Act 1988*, HM Stationery Office, London, ch. 22, p. 1.
6 *The Scotch Whisky Order 1990, Statutory Instruments 1990 No. 998*, HM Stationery Office, London, p. 1.
7 *Code of Federal Regulations*, Definition (7), Washington, DC, ch. 1, Part 5.22, p. 44.
8 *Food and Drug Regulations*, Section B.02.016, Ottawa, 1988, Sect. B02016.
9 Standard P3 Spirits and Liqueurs, *Commonwealth of Australia Gazette*, No. P27, Aug. 27th, 1987, Canberra, p. 1.
10 Nicol, D., in *The Science and Technology of Whiskies*, ed. Piggott, J. R., Sharp, R., and Duncan, R. E. B., Longman, London, 1989, p. 118.
11 Panek, R. J., and Boucher, A. R., in *The Science and Technology of Whiskies*, ed. Piggott, J. R. Sharp, R., and Duncan, R. E. B., Longman, London, 1989, p. 167.
12 *Scotch Whisky Industry Review 1992*, ed. Gray, A. S. Charterhouse Tilney, Edinburgh, 1992, p. 1.
13 Nishimura, K., and Matsuyama, R., in *The Science and Technology of Whiskies*, ed. Piggott, J. R., Sharp, R., and Duncan, R. E. B., Longman, London, 1989, p. 235.
14 Philp, J. M., in *Proceedings of the Second Aviemore Symposium on Malting, Brewing and Distilling*, Aviemore, UK, ed. Campbell, I., and Priest, F. G., Institute of Brewing, London, 1986, p. 148.
15 Swan, J. S., Howie, D., Burtles, S. M., Williams, A. A., and Lewis, M. J., in *The Quality of Foods and Beverages*, ed. Charalambous, G. and Inglett, G. E., Acedemic Press, New York, 1981, pp. 16 and 22.
16 Maarse, H., and Visscher, C. A., *Volatile Compounds in Food, Qualitative Data*, Supplement 2, 1985, TNO-CIVO Food Analysis Institute, Zeist, The Netherlands, p. 66.1.
17 Maarse, H., and Visscher, C. A., *Volatile Compounds in Food, Quantitative Data*, Supplement 4, 1985, Zeist, p. 66.14.
18 Simpkins, W. A., *J. Sci. Food Agric.*, 1985, **36**, 367.
19 Schoeneman, R. L., and Dyer, R. H., *J. Assoc. Off. Anal. Chem.*, 1973, **56**, 1.
20 Lehtonen, M., and Suomalainen, H., *Process Biochem.*, 1979, 5.
21 Lehtonen, H., *J. Assoc. Off. Anal. Chem.*, 1983, **66**, 62.
22 Lehtonen, H., *J. Assoc. Off. Anal. Chem.*, 1983, **66**, 71.
23 *Harpers Wine and Spirit Annual*, Harper Trade Journals, London, 1993, p. 194.
24 Kajoika, R., and Tang, P. W., *J. Anal. Appl. Pyrol.*, 1984, **6**, 59.
25 Aries, R. E., Gutteridge, C. S., and Ottley, T. W., *J. Anal. Appl. Pyrol.*, 1986, **9**, 81.
26 Curry, C. J., *J. Anal. Appl. Pyrol.*, 1987, **11**, 213.
27 Reid, K. J. G., Swan, J. S., and Gutteridge, C. S., *J. Anal. Appl. Pyrol.*, 1993, **25**, 49.
28 Martin, G. E., Burgraff, J. M., Dyer, R. H., and Buscemi, P. C., *J. Assoc. Off. Anal. Chem.*, 1981, **64**, 186.
29 Perry, D. R., in *Proceedings of the Second Aviemore Symposium on Malting, Brewing and Distilling*, Aviemore, UK, ed. Campbell, I., and Priest, F. G., Institute of Brewing, London, 1986, p. 409.
30 Macfie, J. H., Gutteridge, C. S., and Norris, J. R., *J. Gen. Microbiol.*, 1978, **104**, 67.

Paper 4/00987H
Received February 18, 1994
Accepted April 5, 1994