Sensory and Chemical Analysis of 'Shackleton's' Mackinlay Scotch Whisky

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ABSTRACT

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Three cases of Mackinlay's Rare Highland Malt whisky were excavated from the ice under Sir Ernest Shackleton's 1907 expedition base camp hut at Cape Royds in Antarctica in January 2010. The majority of the bottles were in a pristine state of preservation and three were returned to Scotland in January 2011 for the first sensory and organoleptic analysis of a Scotch malt whisky distilled in the late 1890s. Sensory analysis and the higher alcohol and maturation congener profiles describe a lightly peated malt whisky matured in American white oak sherry or wine casks. Analysis of process related compounds together with combined gas chromatography (GC) mass spectrometry and GC-olfactometry analysis of fermentation related congeners show a distinctly 'modern' style of malt whisky. While Scotch malt whisky at the end of the 19th century was generally regarded as heavily peated and harsh in character, Charles Mackinlay & Co. Distillers were producing a malt whisky with an altogether more subtle character at their Glen Mhor distillery near Inverness. The sensory and chemical analysis of this unique whisky artefact significantly changes our understanding of the quality and character of Scotch malt whisky produced by our distilling forefathers.

Key words: Antarctica, GC, GC-MS, Glen Mhor, HPLC, olfactometry, qualitative descriptive analysis, whisky.

INTRODUCTION

In March 1909, Sir Ernest Shackleton's British Antarctic Expedition departed from Cape Royds in Antarctica leaving equipment and supplies at their base camp hut that included cases of Mackinlay's "Rare Old Highland Malt Whisky"[26]. In 2006, the Antarctic Heritage Trust discovered three of these cases entombed in ice beneath the floor of the hut[17]. In 2010, the bottles of whisky were recovered from Antarctica and thawed providing us with the unique opportunity to apply modern sensory and chemical analytical methods to establish the flavour and composition of a product manufactured over one hundred years ago. Unlike other historical whisky samples, the unique low temperature storage of 'Shackleton's' Mackinlay malt whisky was subsequently shown to have minimised the loss of volatile constituents, key to the flavour profile.

Historical records show that Mackinlay's produced their malt whisky at the Glen Mhor distillery, Inverness[10], describing it as "light and silent enough for consumption as a single whisky"[13]. The distillery had opened in 1892 and so the matured Mackinlay whisky was new to the market when Sir Ernest Shackleton ordered 25 cases of it for the expedition of 1907[26]. Documentary evidence shows that the Glen Mhor distillery had a modern infrastructure[10], taking its water from Loch Ness and using mainly locally grown barley that was malted on site and then dried with Orcadian peat[10], shipped to Inverness from the Isle of Eday[23].

MATERIALS AND METHODS

Artefact description

Three bottles of Mackinlay's whisky, AHT 7023.3, AHT 7023.12 and AHT 7023.11 were recovered from the Antarctic ice, thawed and then kept at 0–4°C during transport from New Zealand to Whyte & Mackay's Invergordon distillery laboratory. The bottles were wrapped in tissue paper and straw, then 12 bottles were packaged in wooden cases that had remained undisturbed in the ice of Antarctica for over 100 years. Figure 1 shows green glass conical bottles still wrapped in tissue paper with rounded shoulders and necks that have a dimple in their base: there were no distinguishing maker's marks on the glass. The closure was cork with a foil capsule painted cream with a red band at its base and stamped on top with 'MACKINLAYS: LEITH: LIQUEUR' in black. Two paper labels: one on the neck reads Rare Old; Highland Malt Whisky; Blended and Bottled; BRITISH ANTARCTIC EXPED: 1907; SHIP "ENDURANCE". The other label on the body reads: RARE OLD HIGHLAND MALT WHISKY: BLENDED AND BOTTLED BY: CHAS MACKINLAY & Co; Blenders and Distillers: LEITH & INVERNESS. A circle stamp in red reads 'ML' with Estabd 1820 underneath. A photograph of an empty Mackinlay whisky bottle, without its tissue paper covering, showing the full label was published following an earlier discovery at Shackleton's hut in 1957[26].

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Sample recovery

The whisky was sampled in a Class II Microbiological Flow Cabinet (Microflow, Hampshire, UK) to maintain sterility of the bottle contents and the samples. All equipment was either autoclaved at 121°C for 20 min, sterilised by UV irradiation or by washing in 70% ethanol. To limit damage to the cork closure and packaging of these historical whisky artefacts a stainless steel needle (316 syringe L 6 in., size 18 gauge Luer-lock, Sigma-Aldrich Company Ltd, Dorset, UK) was passed through the cork close to the neck of the bottle. Cork was removed from the needle with a fine wire. Once the needle had penetrated the bottle a fine biopsy needle was inserted alongside the sampling needle to allow air into the bottle during removal of the liquid into a 100 mL glass syringe (S.G.E. 100 MR-LL-GT Syringe, Supelco, Bellefonte, PA, USA). The samples were dispensed to sterile 60 mL glass bottles for storage at 0–4°C.

Microbiology

For microbiological analysis, 50 mL of whisky was filtered through a 0.45 μm membrane filter (GN-6-MET-RICEL® Pall Life Sciences, Hampshire, UK) and incubated on Plate Count Agar (Merck, Darmstadt, Germany) and Wilkins Chalgren Anaerobic Agar (Sigma-Aldrich, UK) at 30°C.

Freezing point analysis

A 50 mL sample of AHT 7023.3 was placed in a freezer set at –80°C, stirred with a magnetic bar and the temperature, measured with a thermocouple, was recorded remotely every 30 sec. The freezing point was calculated by extrapolating the phase change line representing the change from liquid to solid, to determine a true freezing point for this complex mixture of solutes in water.

Spirit analysis

Alcohol strength was measured on an Anton Paar, DMA 5000 density meter (Anton Paar Ltd, Hertfordshire, UK) and spirit analysis (colour, transmittance, pH, and acidity) was conducted using previously reported methods[1–4].

Two samples (AHT 7023.3 and AHT 7023.12) were sent for radiocarbon analysis by the Oxford Radiocarbon Accelerator Unit. This dating method is used to identify fraudulent claims regarding older whisky[15].

The analysis of the major volatile congeners (GC-flame ionisation detection, GC-FID), ethyl esters (GC-FID), phenols (high performance liquid chromatography, HPLC), maturation related congeners (HPLC), cations, anions and sugars (all ion chromatography), ethyl carbamate and other congeners (GC-mass spectrometry, GC-MS) were all carried out at the Scotch Whisky Research Institute (SWRI) using internally approved and documented methods.

Analysis for metals (ion coupled plasma-mass spectrometry, ICP-MS) and N-nitrosodimethylamine (NDMA) (GC-thermal energy analysis) was by approved laboratories, namely Edinburgh Scientific Services (Edinburgh, UK) and Campden BRI (Gloucestershire, UK) respectively, using internally approved and documented methods.

Sensory analysis

Quantitative descriptive analysis[25] was used to profile the flavour characteristics of the whisky samples. A fixed vocabulary was used to score the samples. The attributes, selected from the SWRI's whisky flavour wheel[12], were as follows: pungent, peaty, sulphury, feinty, cereal, green/grassy, floral, fresh, dried fruit, solventy, sweet, woody, spicy, oily, soapy, sour, struck match, and stale. This vocabulary included not only desirable flavours, but also off-note aromas that can occasionally be encountered during Scotch whisky production. The samples were diluted 1:2 with water prior to assessment by 15 members of SWRI's expert sensory panel. Flavours were scored using a scale of 0–3 and the average scores calculated across the panel. The panel was also given the opportunity to add any unusual or off-note characteristics, which although not typical of today's Scotch whisky may have been present historically, or have developed over time.

Gas chromatography-olfactometry

Samples of whisky (3 mL) were diluted with 3 mL of ultra high quality water and passed through a preconditioned Strata-X solid phase extraction column (Phenomenex, Macclesfield, UK). Adsorbed components were eluted with 1 mL of dichloromethane evaporated to give a final volume of 0.25 mL. GC-olfactometry used an Agilent 6890 gas chromatograph (Agilent Technologies UK, Edinburgh) with the column effluent split between an ATAS odour port (ATAS GL International B.V., Eindhoven, Netherlands) and an Agilent 5973 mass spectrometer in a ratio of 1:4 in favour of the nose port. Two observers assessed each of the extracts and recorded the time, description and intensity of odours. The chromatographic conditions were as follows: Column 60 m × 0.32 mm (id) DB-WAXETR, df = 0.25 μm; oven programme, 40°C for 1 min, increasing to 250°C at 7°C/min, then held for 10 min; transfer line, 250°C; mass spectrometer, scanning 35–400 atomic mass units at 2 scans/sec. Odours recorded in only a single run were discounted and the average time, description and sum intensities for all other aromas were recorded.

RESULTS AND DISCUSSION

The survival of the bottles of 'Shackleton's' Mackinlay whisky (Fig. 1) is at first sight remarkable, given the extremes of temperature they would have been subjected to in Antarctica. As no historical temperature records were available for Cape Royds, both the internal hut temperature and the external Antarctic temperature during 2010 were measured. Within the hut, the temperature ranged between 3.3°C during the summer and –32.5°C during the winter (Fig. 2), with the external minimum temperature reaching –42.1°C during the Antarctic winter. The freezing point of the whisky was measured as –34.3 ± 1.53°C, from the phase change as the whisky went from liquid to solid on a cooling curve. At the freezing point the whisky formed slush and became solid only at well below –40°C. This behaviour, together with the whisky's location beneath the floor of the expedition hut, wrapped in straw within wooden cases, which would have buffered any

Fig. 1. Bottles of 'Shackleton's' Mackinlay's malt whisky following thawing and conservation of packaging. From left to right and in the order they were sampled: AHT 7023.3, AHT 7023.12 and AHT 7023.11.

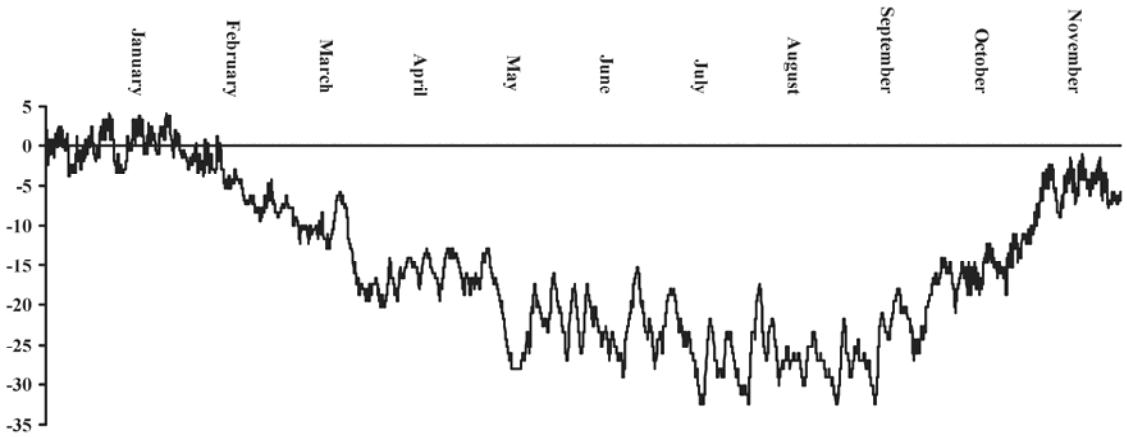

Fig. 2. The temperature profile (°C) inside Shackleton's expeditionary hut at Cape Royds. Readings were taken periodically during 2010 using a HOBO PRO Relative Humidity and Temperature DATA logger.

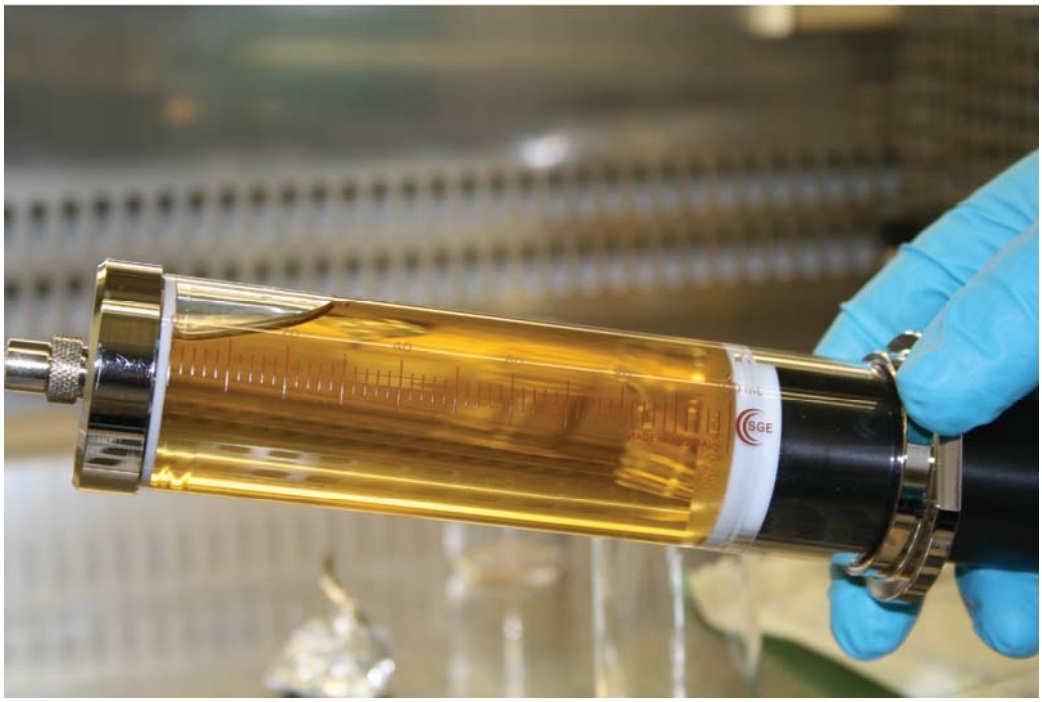

Fig. 3. The first 100 mL sample of the Mackinlay's whisky taken from bottle AHT 7023.3.

large temperature fluctuations, may explain the absence of damage to the glass bottles. Storage in the Antarctic ice and subsequent conservation methods preserved the external packaging and no external signs of leakage from the bottles or any evidence of microbial contamination of the whisky was detected. The probability of the whisky's manufacture between the years 1801 and 1939 was estimated by radiocarbon dating[15] at 95.4%, supporting the historical and physical provenance of the whisky.

Spirit analysis

The Mackinlay whisky was amber in colour and Fig. 3 shows both the colour and clarity of the first sample extracted from bottle AHT 7023.3. The whisky from the three bottles sampled had an absorbance at 430 nm of 0.72 ± 0.03 (all data is expressed as s.e.m., n = 3), with a European Brewery Convention tint of 16.8 ± 0.4[1] and was clear (transmittance 82.0 ± 1.2% at 600 nm) with no visual sediment (residual solids 0.145 ± 0.005 mg/L), despite the bottles having been packaged on their sides leaving the whisky in contact with the cork during storage in the ice. The apparent alcoholic strength was 47.19 ± 0.10% alcohol by volume (abv)[4] and given the retention of the cork closure integrity and the apparent annealing of the cork to the glass, this may represent the actual bottling strength. There was no alcohol strength indication on the label of the Mackinlay whisky as mandatory requirements for this did not come into effect until 1907. With a pH of 4.3 ± 0.01, a total volatile acidity (as acetic acid) of 65.4 ± 0.6 g/100 L AA and no apparent loss of the ~700 mL fill volume of liquid, this initial spirit analysis data suggested that the whisky had not deteriorated during its storage in ice.

Sensory analysis

The flavour profiles for the three samples are shown in Fig. 4. All three samples were similar, exhibiting a balance of peaty, mature woody, sweet, dried fruit and spicy aromas. The peat levels did not dominate, while the mature flavours were consistent with maturation in sherry or wine casks. Low levels of both fresh fruit and green/grassy characteristics, and immature aromas such as feinty and sulphury were present. With no off-notes, the whisky did not exhibit any aromas not found in a modern whisky.

Major volatile congeners and esters

The higher alcohols, namely *n*-propanol, isobutanol, and 2-methyl-1-butanol and 3-methyl-1-butanol (the latter two being amyl alcohols), together with the other major volatile congeners methanol, acetaldehyde, and ethyl acetate, all formed during fermentation, were separated by GC[4] and their concentrations are shown in Table I. This analysis provided a 'fingerprint' for the whisky that matched the currently expected higher alcohol profile of a Scotch malt whisky[2,14,20]. In particular, the summed concentrations of the amyl alcohols (at 110.3 ± 1.3 g/100 L AA) indicative of pot still distillation, placed this whisky at the lower end of the range for these congeners measured in Scotch malt whisky surveys reported previously[2,14,20]. Furfural (Table I), another indicator of pot still distillation[2], had a concentration of 2.9 ± 0.0 g/100 L AA, close to the average furfural concentration reported for currently produced malt whisky, while malt whisky blended with grain whisky is reported to have a significantly lower furfural concentration[2,18]. The major volatile congener data suggested that the 'Mackinlay' was a malt whisky, rather than one blended with grain whisky.

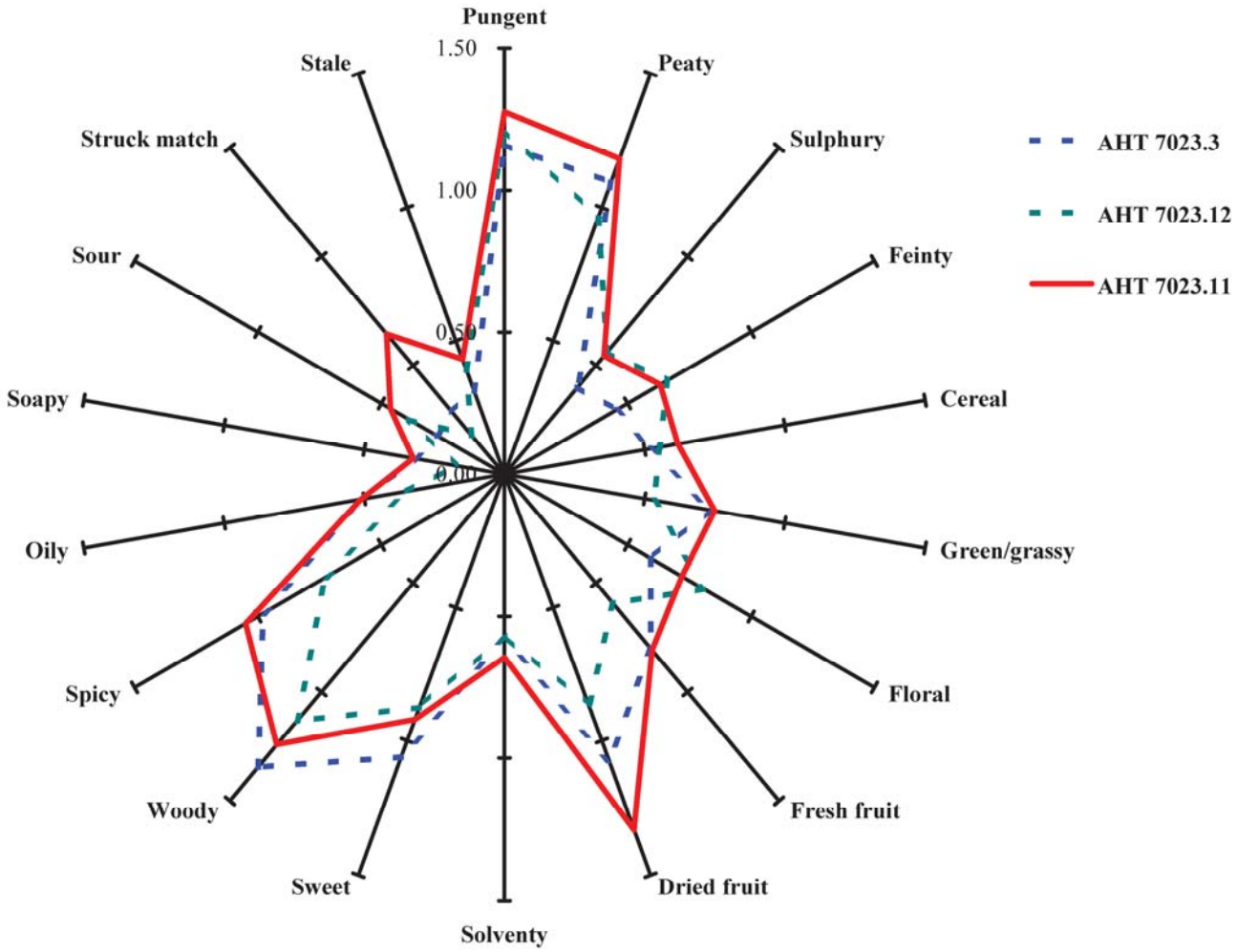

Fig. 4. Flavour profiles for the Mackinlay whisky.

Table I. Concentration of major volatile congeners of 'Shackleton's' Mackinlay whisky.

Congeners	g/100 L (absolute alcohol)[a]
Acetaldehyde	11.4 ± 0.4
Ethyl acetate	40.7 ± 0.6
Acetal	12.6 ± 0.3
Methanol	7.1 ± 0.03
n-Propanol	34.0 ± 0.6
Isobutanol	81.6 ±1.1
Isoamyl acetate	1.5 ± 0.03
n-Butanol	0.4 ± 0.03
2- + 3-Methyl-1-butanol	110.3 ± 1.3
2-Methyl-1-butanol	30.1 ± 0.3
3-Methyl-1-butanol	80.2 ± 1.0
3-Methyl-1-butanol/2-methyl-1-butanol	2.7
Higher alcohols[b]	225.9 ± 1.7
Furfural	2.9 ± 0.0

[a] Values represent mean ± s.e.m. (n = 3).
[b] Sum of *n*-propanol, isobutanol and 2- and 3-methyl-1-butanol.

Table II. Concentration of ethyl esters and associated analytes.

Ethyl esters	mg/L at sample strength
Ethyl hexanoate	0.2 ± 0.1
Ethyl octanoate	7.0 ± 0.2
Ethyl decanoate	9.0 ± 0.3
Ethyl dodecanoate	5.3 ± 0.3
Ethyl detradecanoate	0.2 ± 0.0
Ethyl hexadecanoate	2.4 ± 0.2
Ethyl 9-hexadecanoate	4.6 ± 0.8
Ethyl C18:0 (stearate)	0.4 ± 0.0
Ethyl C18:1 (oleate)	1.6 ± 0.6
Ethyl C18:2 (linoleate)	3.0 ± 0.8
Ethyl C18:3 (linolenate)	2.3 ± 0.3
2-Phenethyl acetate	0.5 ± 0.1
2-Phenethyl ethanol	18.0 ± 0.3

At low temperatures, the long chain ethyl esters in malt whisky may precipitate and form a haze that clouds the liquid. To prevent haze formation in bottled whisky products chill filtration was introduced during the 1960s[3], so it was surprising that haze obscuration was not found in the Mackinlay whisky after its many years at low temperature. However, the choice of a high bottling strength (47.19% ± 0.10% abv) together with low concentrations of the ethyl esters of lauric, palmitic and palmitoleic acids (ethyl dodecanoate, hexadecanoate and 9-hexadecanoate) (Table II) that totalled 12.3 ± 1.3 mg/L, put the whisky at only a very slight risk of mild precipitation[3].

Phenols and peat composition

The phenolic compounds used to determine the peating levels in Scotch whisky are shown in Table III. The concentration of total phenols in the Mackinlay whisky was

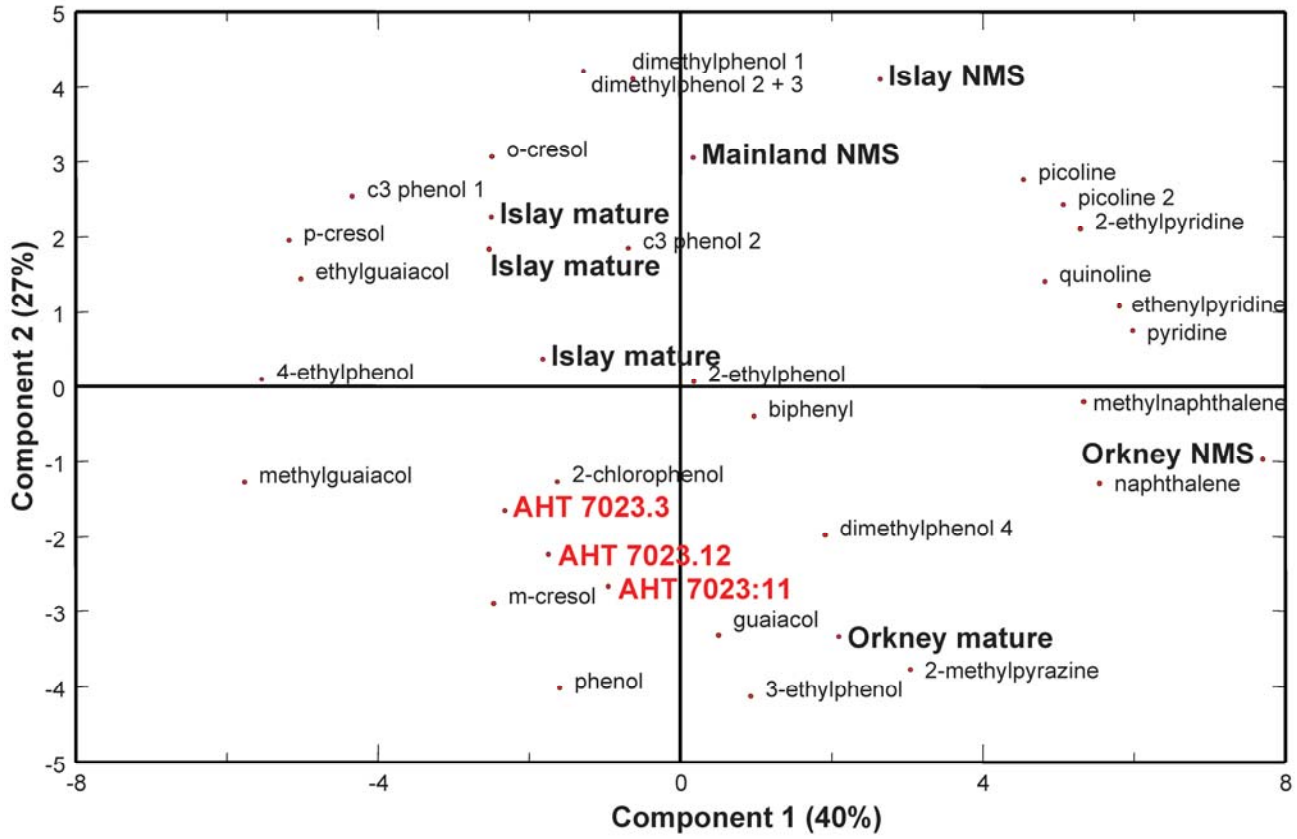

Fig. 5. Principal component analysis (PCA) of peat derived congeners in peated whisky and new-make spirit.

Table III. Concentration of volatile phenols.

Phenols	mg/L at sample strength
Phenol	1.03 ± 0.02
Guaiacol	0.45 ± 0.06
m/p-Cresol	0.58 ± 0.02
o-Cresol	0.58 ± 0.03
4-Methyl guaiacol	0.23 ± 0.02
4-Ethyl phenol	0.55 ± 0.05
4-Ethyl guaiacol	0.10 ± 0.00

3.48 ± 0.18 mg/L, an amount found in the 'lightly-peated' whisky currently produced[30], supporting our sensory findings. Analysis of peat, using Curie point pyrolysis, in combination with gas chromatography-mass spectroscopy (GC-MS), from a number of locations across Scotland (Islay, Orkney, St. Fergus and Tomintoul), has established that these peats are chemically distinct[11]. The burning of peat imparts distinct flavours to malted barley during kilning, and these are transferred to the new make malt spirit on distillation. To identify the source of the peat used in the production of the Mackinlay whisky, the concentrations of peat-derived phenols were compared, using principal components analysis (Fig. 5), with samples both of new make spirit and of matured Scotch malt whisky whose peat origin is known, as the impact of maturation on peat derived congeners is not fully understood[11]. Figure 5 shows that the Mackinlay whisky is very similar to whisky currently produced using Orkney peat, confirming the historical records that peat was sourced from the Isle of Eday.

Table IV. Concentration of maturation congeners.

Congeners	g/1,000 L (absolute alcohol)
Gallic acid	10.6 ± 0.3
Ellagic acid	16.7 ± 0.6
Coniferaldehyde	2.3 ± 0.1
Vanillin	4.7 ± 0.1
Vanillic acid	2.2 ± 0.1
Sinapaldehyde	1.6 ± 0.1
Syringaldehyde	9.6 ± 0.3
Syringic acid	3.6 ± 0.1
Scopoletin	1.5 ± 0.0
5-HMF	20.4 ± 0.1

Maturation related congeners and other cask derived compounds

When the profile of the syringyl and guaiacyl lignin breakdown products from cask maturation of the Mackinlay whisky (Table IV) was compared with data from current matured Scotch malt whisky samples, it was consistent with maturation in oak casks for a period of at least 5 years, but more likely closer to 10 years[2]. The concentration of scopoletin in particular (1.5 g/1,000 L AA), when taken together with the corresponding concentrations of the tannins ellagic and gallic acid, are typical of American Oak (*Quercus alba*), rather than European oak (*Quercus petraea* or *Quercus robur*)[7,21]. Analysis of the Mackinlay whisky using GC coupled to mass spectroscopy (GC-MS) also suggested that the casks were constructed of American oak. The ratio of *cis*- to *trans*-lactones ('whisky lactones') was greater than 8:1 and this

Table V. Concentration of sugars.

Sugars	mg/L
Glucose	206.7 ± 2.6
Fructose	171.0 ± 2.0
Lactose	1.0 ± 0.0
Sucrose	1.7 ± 0.0
Maltose	2.0 ± 0.0

Table VI. Concentration of cations, anions and tyrosol by HPLC.

Cations and anions	mg/L
Sodium	22.3 ± 7.6
Ammonium	4.2 ± 0.2
Potassium	42.6 ± 1.9
Magnesium	2.3 ± 0.4
Calcium	5.5 ± 2.5
Fluoride	5.5 ± 2.5
Chloride	33.5 ± 16.7
Sulphate	19.1 ± 6.5
Tyrosol	0.7 ± 0.0

also suggested that the Mackinlay whisky had been matured in American oak casks[28,29]. 'First-fill' wine or sherry casks used for whisky maturation produce a significantly higher concentration of glucose and fructose in matured whisky than other cask types. The concentrations of glucose and fructose measured by ion chromatography for the Mackinlay whisky (Table V) were consistent with the use of 'first-fill' casks[19]. Tyrosol, a constituent of non-distilled beverages such as sherry or wine[18] was also identified by HPLC (Table VI) and its concentration of 0.7 ± 0.0 mg/L was comparable with the average concentration measured at around 1.0 mg/L for a selection of 'first-fill' sherry cask matured Scotch malt whiskies (SWRI personal communication). The concentration of potassium (Table VI) was equivalent to that found in a first-fill sherry or wine cask, rather than a refilled sherry cask or bourbon cask, which were not in common use until the 1930s. This analysis, that relates a range of compounds to the maturation period, the cask type and the previous use of the cask, indicates that the Mackinlay new make spirit was matured in a first-fill American oak sherry or wine cask for a period of greater than five years. The sensory analysis supports this hypothesis with the whisky exhibiting the woody, sweet, dried fruit and spicy aromas typically associated with sherry cask maturation. The maturation congener 5-hydroxymethyl furfural (5-HMF) was present at 20.4 ± 0.1 g/1,000 L AA (Table IV) and is also associated with sherry casks for wines such as Amontillado and Oloroso[18]. It should be noted however that 5-HMF, as well as being a component of heat-treated or toasted casks, is also a component of burnt sugar and/or caramel. We have no reason to believe the amber tint of the whisky was not wholly cask-derived, but one cannot rule out addition of a burnt-sugar or caramel-type tint product to colour the whisky at bottling as these were available at the time[16].

Gas chromatography mass spectrometry and olfactometry analysis

GC-MS coupled with olfactometry analysis was used to separate the whisky into its component parts to allow combined sensory and chemical analysis[24] (Table VII). By identifying and comparing the relative abundance of the congeners present on total ion chromatograms, some comments about the fermentation and distillation processes could be made. Total ion chromatograms produced for samples AHT 7023.3 and AHT 7023.12 were virtually identical and showed only minor variations from what would be expected from modern whiskies. Olfactory analysis detected 77 aromas of which 43 were tentatively identified with peaks on the mass ion chromatogram (Table VII). All the aromas that were identified have previously been detected in Scotch whisky and the retention times and descriptions of aromas that could not be identified are typical of aromas detected in currently produced malt whiskies. The presence of ethyl lactate and 2-propen-1-ol (allyl alcohol) indicates that the wash had been infected with lactic acid bacteria before distillation. This bacterial load that would have flourished at the end of the yeast fermentation would likely have been introduced by the malted barley, the use of brewer's yeast and the indigenous distillery microflora[27]. Other aromas detected by olfactometry and related to lactic acid bacterial growth were a stale solvent aroma of ethyl 2-butenoate, and sweet/peaches, sweet/peaches/coriander leaf aroma at retention times of 15.4, 38.71 and 39.41 min respectively; the latter retention indices and descriptors agreeing with those published for γ- and δ-dodecalactones[28]. The presence of short-chain acids characteristic of feints (butanoic, 2-methyl and 3-methylbutanoic acids), suggests that the cut point from the spirit to feints during distillation was made at lower alcohol strength than that commonly used in current pot still spirit production. This later cut to feints increased the intensity of cereal popcorn aroma at 19.22 (2-acetyl-1-pyrroline) and that of earthy/mouldy leaf aromas at 21.20 and 21.8 that is more intense in the Mackinlay whisky than in currently produced whisky[5]. There were disinfectant/farmyard/phenolic aromas at 34.08, 34.74 and 35.60 and these may be due to high-boiling point phenols normally only detected in more concentrated extracts of currently produced whisky.

Raw material and process related analysis

Modern whisky production carefully manages barley varieties and the kilning of the malted barley to minimise the production of ethyl carbamate and NDMA respectively[6,22]. The level of ethyl carbamate in the Mackinlay samples was 36.3 ± 1.9 μg/L consistent with the low levels of this compound in Scotch malt whisky currently produced; the average concentration of ethyl carbamate in a Food Standards Agency survey of Scotch whisky in 2006 was 29 μg/L[9]. Given that the level of the ethyl carbamate precursor, epiheterodendrin, may have been relatively high in the older barley varieties used during the late 1900s, the low concentration of ethyl carbamate reported here suggests that the distillation in the copper wash still was carefully controlled to prevent frothing of the wash and fouling of the copper spirit still. NDMA is produced during kilning of the malt with peat, however the inclusion of sulphur containing coal at this time to augment the drying process[6] may be reflected in the low concentration of NDMA (0.9 ± 0.1 μg/L) found in the Mackinlay whisky; the current level in Scotch malt whisky is <0.5 μg/L[8]. These low concentrations are sur-

Table VII. Summary of congeners identified by gas chromatography coupled with mass spectrometry and gas chromatography coupled with olfactometry descriptions.

Time	Retention index	Description	Intensity[a]	Identity
12.77	1009	estery	2	ethyl butanoate
13.10	1027	estery, sweet, sour/solvent	5	ethyl 3-methylbutanoate
14.36	1093	estery pear drops	7	2-/3-methylbutyl acetate
15.40	1149	stale, solvent	4	ethyl 2-butenoate
15.82	1171	estery, solvent stale	4	2-heptanone
16.05	1183	solventy/burnt, higher alcohol	8	2-/3-methylbutanol
16.75	1220	estery, fruity	5	ethyl hexanoate
18.01	1287	solventy, ether	8	ethyl furfuryl ether
18.50	1312	mushrooms	5	2-heptanol
18.90	1334	solventy, fruity/estery	2	ethyl heptanoate
19.00	1339	meaty, burnt/acrid	4	
19.22	1350	cereal/popcorn	12	
20.18	1401	rubbery, solvent/estery	3	2-nonanone
20.62	1425	pickled onions, fruity, estery	4	
20.87	1438	aniseed, estery	5	ethyl octanoate
21.20	1455	earthy, mouldy leaves, stale	11	
21.32	1462	goats cheese, fatty stale/earthy	6	
21.43	1467	mushrooms, sweet popcorn	2	
21.60	1476	mouldy leaves, waxy	2	ethyl dimethylpyrazine
21.70	1482	vinegar	6	acetic acid
21.88	1491	mouldy leaves, stagnant/drains	10	
22.40	1519	earthy, stale	6	
22.86	1543	floral, solvent	2	linalool
23.16	1559	fruity, waxy, estery	3	ethyl 2-hydroxy-4-methylpentanoate
23.36	1570	roasted almonds, beeswax	7	
23.80	1593	earthy	6	
24.39	1624	leafy, cucumber, Parma violets	5	
24.79	1645	rancid	5	butanoic acid
25.49	1689	sweaty, cheesy, stale	11	2-/3-methylbutanoic acid
25.62	1689	oily/nutty, polish/floral	2	ethyl 9-decenoate
25.81	1699	green/grassy, floral	2	2-phenylacetaldehyde
26.02	1710	oily, beeswax	4	
26.21	1720	meaty, yeasty	4	
26.50	1735	nutty/oily, fatty	8	thiophene carboxaldehyde
26.61	1741	aniseed, floral	9	2-hydroxybenzaldehyde
27.01	1762	green/grassy, beeswax	7	
27.13	1769	dried fruit, leafy	2	
28.18	1824	dried fruit, tobacco	4	
28.27	1829	fatty, rancid	3	hexanoic acid
28.36	1834	almonds, herbal, tobacco	6	2-phenylethyl acetate
28.44	1838	sour	4	
28.60	1847	dried fruit, herbal	6	beta-damascenone
28.84	1859	sweet/spicy	2	dihydromaltol
28.94	1865	phenolic, medicinal	10	guaiacol
29.21	1879	phenolic, smoky, sweet	9	guaiacol
29.43	1891	floral, minty/herbal	4	ethyl 3-phenylpropanoate
29.81	1910	medicinal	3	*trans*-oak lactone
29.95	1918	floral/roses	10	2-phenylethanol
30.33	1938	sour/stale	6	
30.52	1948	medicinal, woody, phenolic, sweet	7	4-methylguaiacol
30.88	1967	vanilla, phenolic/pungent	5	
31.08	1978	coconut, spicy, floral	11	*cis*-oak lactone
31.38	1991	sour, beeswax	2	
31.73	2012	spicy, sweet, phenolic	6	4-ethylguaiacol
31.87	2019	medicinal, spicy	9	2-ethylphenol
32.19	2036	sweet, coconut, phenolic	7	gamma-nonalactone/dimethylphenol
32.34	2045	farmyard	10	*para*-cresol
32.54	2055	phenolic, paint, farmyard	8	*meta*-cresol
32.66	2061	hospital	2	
33.02	2080	dried fruit, floral, spicy	5	
33.27	2093	medicinal, stale, burnt/plastic	8	
33.49	2105	solvent, ethyl acetate	3	
33.94	2129	farmyards, medicinal	7	4-ethylphenol
34.02	2133	spicy	2	eugenol
34.08	2137	disinfectant, farmyard	9	
34.74	2171	oil, phenolic, farmyard	3	trimethylphenol
35.05	2188	solvent, soapy/goat	6	decanoic acid
35.14	2192	spicy/curry	10	
35.60	2217	phenolic, goat/farmyard	4	
36.06	2241	mouldy, green/grassy, earthy	4	
37.10	2296	rubbery, floral	2	
37.74	2330	floral	3	
38.71	2381	sweet/peaches	4	
39.41	2418	fruity, sweet, peaches, coriander leaf	7	
39.60	2428	soapy	9	dodecanoic acid
41.16	2511	soapy	3	
43.73	2647	sour, soapy, detergent, waxy	6	
44.58	2692	vanilla	12	vanillin
45.74	2753	estery, sweet, honey, sour/spicy	5	

[a] Intensity as rated by assessors with 12 being considered high.

Table VIII. Metal analysis by ICP-MS.

Metals	µg/L
Arsenic	80 ± 20
Lead	70 ± 10
Silver	80 ± 20
Mercury	0.28 ± 0.17
Aluminium	62 ± 3
Barium	100 ± 0
Cadmium	80 ± 20
Chromium	100 ± 0.00
Copper	1030 ± 30
Iron	330 ± 30
Manganese	100 ± 0
Nickel	100 ± 0

prising given that this whisky was produced before concerns were raised about this compound and changes were introduced to reduce NDMA formation in the malting process in the 1980s.

Cation/anion (Table VI) and heavy metal analysis (Table VIII) of the whisky showed no abnormally high values when compared with those found in whisky today, indicating a good process water supply and no major contamination routes during production at the Glen Mhor distillery. However, in one bottle, AHT 7023.12, a significant variation in sodium and chloride concentrations relative to the other two samples (Table VI) was observed and there was also variation in the concentration of the mercury content between the bottles (Table VIII). This contamination may have been introduced at bottling, reflecting inconsistencies in the rinsing of detergent during bottle washing, and the presence of heavy metal residues may be a reflection of bottle manufacture at the time.

CONCLUSIONS

Samples from the three Mackinlay bottles revealed a whisky at relatively high alcohol strength and with low levels of the ethyl esters that normally contribute to haze formation. Consequently, the whisky had remained clear and bright despite its long period at low temperatures. Sensory evaluation together with gas GC-MS and olfactometry analysis describe a distinctly 'modern' style of malt whisky. Analysis of the major volatile congeners, maturation related congeners, phenols and sugars revealed a very complex, lightly-peated spirit matured for 5–10 years in 'first-fill' American white oak sherry casks. The levels of process-related compounds, such as ethyl carbamate and NDMA and the metal and cation/anion content, are consistent with those currently found in malt whisky, and give us an insight into a controlled production process. The results presented here significantly change our perception of the quality and character of Scotch malt whisky produced over 100 years ago. Malt whisky from this period was generally regarded as robust, peaty and too 'heavy' in style for ordinary consumption. Our analysis however describes a surprisingly light, complex whisky, with a lower phenolic content than expected. The first season for the Glen Mhor distillery was 1893–1894 and so the matured malt whisky they supplied to Mackinlay was relatively new to the market when Shackleton ordered 25 cases of it for the Antarctic expedition of 1907. We are therefore indebted to Sir Ernest Shackleton, not only for his enduring spirit of courage and determination, but also for inadvertently giving us this unique opportunity to discover for ourselves, with modern sensory and chemical analytical techniques, the historical talents of our distilling forefathers.

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