



ORIGINAL ARTICLE

DOI 10.58430/jib.v13i4.83

Long-chain esters in the spotlight: analysis of the aroma of traditional whisky and analogous age-accelerated spirit

- Marco Cardin¹
- Sebastian Reimer Bendtsen¹
- Beatriz Quintanilla-Casas¹
- Aleksander Byzdra²
- Mikael Agerlin Petersen¹
- Tobias Emil Jensen²
- Sylvester Holt¹

¹ Department of Food Science, University of Copenhagen, Rolighedsvej 26, 1958 Frederiksberg, Denmark

² EtOH Spirits, Fruebjergvej 3, 2100 København Ø, Denmark

sylvester.holt@food.ku.dk



OPEN ACCESS

This is an open access article distributed under the terms of the creative commons attribution-non-commercial-no-derivatives license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>), which permits non-commercial re-use, distribution, and reproduction in any medium, provided the original work is properly cited, and is not altered, transformed or built upon in any way.

Abstract

Why was the work done: Consumer interest in distinctive, high quality alcoholic beverages has driven innovation in whisky production, particularly in ageing techniques. While traditional oak barrel ageing is essential to the legal definition and flavour development of whisky, new approaches such as the Jensen Reactor - an accelerated ageing method combining ultrasound, temperature, oxygenation, pressure, and catalysis - aim to replicate the chemical changes of barrel ageing in one to two weeks. However, how such accelerated methods compare to traditional maturation lasting many years remains unclear.

How was the work done: The study focused on the quantification of medium and long-chain fatty acid ethyl esters in malted rye and barley spirits comparing the new make spirit, accelerated-aged spirit and traditional whisky. Volatile organic compounds were characterised using stir bar sorptive extraction coupled with gas chromatography-mass spectrometry (SBSE-GC-MS).

What are the main findings: Most C₂–C₁₂ ethyl esters increased in concentration during accelerated ageing, often surpassing their sensory thresholds and likely contributing fruity notes. Ethyl acetate, by contrast, was reduced to near or below threshold levels (25–30 mg/L) in accelerated aged samples, while present at 120–300 mg/L in traditionally aged spirits, which presumably contribute a solvent-like aroma. Long-chain esters (C₁₄–C₁₈), commonly linked to waxy characteristics, remained relatively stable across both ageing methods reflecting their initial concentration in the new make spirit. Additionally, ethyl linoleate decreased during traditional ageing, whereas ethyl tetradecanoate increased; the underlying mechanism for this is currently unknown.

Why is the work important: These findings highlight the importance of understanding ethyl ester formation to improve aroma development and quality in whisky or spirit production and maturation.

Keywords

long-chain esters, whisky, accelerated aged spirit, aroma, maturation, barley, rye

Introduction

The term 'straight whisky' cannot be used in the United States (Code of Federal Regulation 2024), nor can 'whiskey/whisky' be used in Europe (European Commission 2008; 2019), until the grain based spirit has been aged for a minimum of two years (USA) or three years (Europe) in oak barrels. Ageing represents the highest economic and environmental cost in the whisky production chain (Dabija et al. 2024), but plays a crucial role in shaping the aroma profile. The spirits industry has a long tradition of innovation, especially in refining whisky aroma and optimising maturation techniques. Over 5,100 patents have been filed between 1866 and 2025, yet most inventions do not endure. For example, Morgan (2022) noted that the patent by Hewitt in 1898 on rapid maturation, which focused on aldehyde reduction - particularly furfural - which was believed to be responsible for the raw smell and taste of pot still whisky - failed to find application. Indeed today, there is still no consensus on which chemical compounds are responsible for the raw aroma and taste of pot still whisky. In contrast, some innovations, such as the tiered barrel rack patented by Stitzel (1880), which allowed for increased air circulation around the barrel during ageing, remain in use today.

Recent advancements in whisky production reflect broader trends, including the growing craft spirits movement, development of carbon neutral technologies, and research on new distillation techniques (Bellut et al. 2021; Grand View Research 2024a). Despite industry growth, traditional ageing remains a major challenge, particularly for small scale producers facing barriers such as maturation facilities and tied-up capital. The dominance of blended whisky - with a 70.2% market share in 2024 - is attributed to affordability and consistency of quality (Grand View Research 2024b). Replicating the sensory profile developed through prolonged maturation in a shorter timeframe presents an attractive economic opportunity, as accelerated ageing could potentially reduce costs and expand production capability (Russell and Kellershon 2018).

Whisky aroma is shaped by raw materials, fermentation, distillation, and ageing (Christoph and Bauer-Christoph 2007; Buglass 2011; Kelly et al. 2023). Among the key contributors to aroma, esters

represent the largest group of flavour compounds, providing fruity, floral, and sweet notes (Christoph and Bauer-Christoph 2007; Gao et al. 2014). Medium-chain esters like ethyl octanoate and ethyl dodecanoate influence whisky aroma even at low concentration due to their low odour thresholds (Niu et al. 2018). Esters are part of the whisky portfolio of flavour active congeners that are further refined during ageing, softening harsher notes while increasing complexity (Buglass 2011; Kelly et al. 2023).

Long-chain ethyl esters are often overlooked in the analysis of whisky aroma. Esters such as ethyl tetradecanoate (C14), ethyl hexadecanoate (C16), ethyl (Z)-octadec-9-enoate (C18:1 Δ 9), ethyl (9Z,12Z)-octadeca-9,12-dienoate (C18:2 Δ 9,12), and ethyl (9Z,12Z,15Z)-octadeca-9,12,15-trienoate (C18:3 Δ 9,12,15) are often excluded due to their poor solubility in water/ethanol solutions at 23% ABV with a consequent precipitation during chilled filtering, and, as a result, low concentrations in the final spirit (Boothroyd et al. 2012; Wu et al. 2023; Zhang et al. 2025). Nevertheless, it has been shown that these compounds are correlated with enhanced mellowness and aroma persistence, which are both desirable attributes developed during the whisky maturation process (Wu et al. 2023). Maturation itself is a complex phenomenon that is not fully understood; it is influenced by numerous factors, including the presence of other congeners, alcohol strength, wood type, toasting and charring levels, barrel size, duration of ageing, and storage conditions, all of which shape the final sensory profile of the whisky (Gollihue et al. 2021; Kelly et al. 2023).

The maturation process can be described as having three major functional categories: reactions which are 'additive', 'subtractive' or 'interactive' (Piggott and Conner, 1995; Conner 2014). Additive reactions involve the introduction of new compounds into the spirit or an increase in their concentration, such as through the extraction of substances from the wood. Interactive reactions occur when oxygen permeates the cask, triggering interactions among molecules within the spirit, between these molecules and those derived from the wood such as selective lignin ethanolysis (Miller 2024). Subtractive reactions refer to the loss of compounds via evaporation, cask sorption, or oxidation. This includes the 'angel's

share' or the volume lost during maturation and volatile compounds that persist through distillation (e.g. acrolein), and volatilisation of sulphur compounds (Kelly et al. 2023; Miller 2024).

Accelerated ageing of spirits refers to a range of methods and techniques designed to reduce the time required for fortified wine, whisky, rum, or brandy to develop the complex flavours, aromas, and textures associated with long maturation times (Solar et al. 2021; Kruger et al. 2022). An example is the Canteiro Madeira wine *Estufagem* process, where the temperature is increased to 45°C for three months to accelerate ageing while preserving key aroma compounds (Pereira et al. 2014; Autonomous Region of Madeira 2015). Other approaches include the use of smaller barrels, wood chips, ultrasound, increased temperature, agitation, micro-oxygenation and pressure change to enhance wood interaction, increase transesterification and contribution to the Fenton reaction (Madrera et al. 2010; Chemat et al. 2017; Canas et al. 2019; Canas et al. 2020; Lin et al. 2021).

A specific innovation is the Jensen Reactor, a system designed to expedite the maturation of new make spirits (Jensen et al. 2015; Jensen and Byzdra 2024). This reactor employs a combination of ultrasound, heating, oxygenation, and the circulation of new make spirit through discs of wood from oak cask staves, as well as whole staves placed inside the reactor. With a capacity of 200 litres, the reactor typically ages the spirit within seven to 14 days. To further enhance the ageing process, inorganic and organic acids, such as citric acid and acetic acid, are used as catalysts to mimic the complex chemical transformations that occur during the ageing of traditional whisky (Jensen and Byzdra 2024).

Other examples of accelerated ageing have been reported for the ageing of vinegar (Delgado-González et al. 2017) and wine spirits (Li et al. 2021). Both studies reported positive outcomes and employed ultrasound in combination with other techniques, such as treatment with alternating magnetic fields, recirculation with woodchips and micro-oxygenation. However, while Li et al (2021) observed an increase in ester concentrations in the accelerated-aged vinegar, Delgado-González et al (2017) focused on a limited set of compounds, excluding esters. Notably, both studies overlooked

medium- and long-chain esters.

This exploratory study presents a comparison of ageing methods, including spirits produced using the patented accelerated-ageing process (Jensen and Byzdra 2024). Medium- and long-chain ethyl esters were quantified to assess how the ageing method influences flavour development. In total, the relative abundances of 92 volatile compounds were analysed to identify trends and shifts in the broader flavour profile across different maturation approaches.

Materials and methods

Accelerated ageing

New make spirit was subject to ultrasonic treatment using the Jensen Reactor (Jensen et al. 2015; Jensen and Byzdra 2024), with a total energy input of ca. 463 J/mL for the Hafnium sample, 440 J/mL for Producer 1 (eight day product), and 710 J/mL for Producer 2 (15 day product). Technical details on reactor geometry and design can be found in Jensen et al (2015) and Jensen and Byzdra (2024). Temperature was held at 65°C for the Hafnium sample and 62°C for both Producer 1 and Producer 2 throughout the process. Oxygenation was at a flow rate of ca. 1 L/min for 5 minutes every hour over a processing time of 72 hours. The internal pressure of the reactor fluctuated, with maximum pressure of approximately 0.3 bar above atmospheric. American white oak (*Quercus alba*) staves, sourced from standard barrels, were used as for wood contact in the Jensen Reactor, with a medium toasting level as used by the producers. Similarly, sherry wood was employed for the accelerated ageing of barley spirits, as the commercial whiskies in this work had also been aged in sherry wood.

Sample collection and preparation

15 samples ranging from 43-64% ABV were evaluated in this study (Table 1). New make, accelerated-aged spirits, and whiskies from seven distilleries were provided by EtOH Spirits and/or Juul's Engros (Copenhagen, Denmark). For each new make, accelerated-aged, and traditionally aged product, a new bottle of new make spirit and the final product were purchased at the time of analysis to ensure quality, minimise evaporation and volatile compound degradation over time.

Producers of the new make spirit and the corresponding traditionally aged product reported using the same raw materials and production process for both spirits. New make and final product from Producer 2 were sold at 46.3% and 47.2% alcohol by volume (ABV) (Table 1). To standardise the concentration of ethanol, all spirits were diluted to 43% ABV using Milli-Q water.

Rye and barley based spirits were analysed to assess the contribution of esters to their aroma profiles and to compare the profiles of accelerated-aged

spirits with those of traditionally aged ones. Traditional aged whisky from Producers 1 and 2 were aged in new American oak barrels and distilled in double pot stills. The accelerated-aged spirits were matured in sherry oak wood as well as the traditionally aged whiskies. Samples included both new make spirit and accelerated-aged spirits matured over 8, 9 or 15 days, with whiskies aged for 4, 15, 25, 30, and 40 years. These provide a broad range of information on the aroma components of these spirits over time.

Table 1.

Overview of spirits used in this work.

Sample	Cereal	Producer	Style	Ageing	Origin	ABV (%)
P1NM	Rye	Producer 1	New make	-	Denmark	64
P104	Rye	Producer 1	Rye 100%	4 years	Denmark	48
P1AA	Rye	Producer 1	Accelerated ageing	8 days	Denmark	64
P2NM	Rye	Producer 2	New make	-	Finland	46.3
P204	Rye	Producer 2	Rye 100%	4 years	Finland	47.2
P2AA	Rye	Producer 2	Accelerated ageing	15 days	Denmark	47.5
HaNM	Barley	Hafnium	New make	-	Denmark	62.8
HaAA	Barley	Hafnium	Accelerated ageing	9 days	Denmark	43
KV04	Barley	Kavalan	SM Taiwan whisky	4 years	Taiwan	59.4
ML15	Barley	Mortlach	Speyside SM Scotch whisky	15 years	Scotland	46
SB15	Barley	Springbank	Campbeltown SM Scotch whisky	15 years	Scotland	46
LW15	Barley	Linkwood	Speyside SM Scotch whisky	15 years	Scotland	46
LW25	Barley	Linkwood	Speyside SM Scotch whisky	25 years	Scotland	43
LW30	Barley	Linkwood	Speyside SM Scotch whisky	30 years	Scotland	53.1
BM40	Barley	Benromach	Speyside SM Scotch whisky	40 years	Scotland	57.6

NM' - new make, 'AA' - accelerated aging, 'number' – ageing in years, 'P' – producer, 'ABV' - alcohol by volume, 'SM' – single malt

Stir bar sorptive extraction and GC-MS analysis

Volatile compounds were extracted from 15 mL of sample in 100 mL Schott bottles. Volatile compounds were collected on 20 mm Twister™ stir bars, covered with a 1 mm polydimethylsiloxane phase (Gerstel, Mullheim an der Ruhr, Germany), by stirring for 60 min at 700 rpm at 20°C. After extraction, the stir bars were rinsed three times with Milli-Q water, dried with a paper towel, and air dried for 5 minutes.

The volatile compounds absorbed by the stir bars were desorbed using a TurboMatrix 350 automatic thermal desorption unit (Perkin Elmer, Shelton, USA). Primary desorption was performed by heating the stir bars to 250°C under a flow of 50 mL/min of carrier gas for 15 minutes. The released compounds were trapped in a Tenax TA cold trap (30 mg) held at 1°C, followed by secondary desorption at 280°C for 4 minutes (outlet split 1:100), allowing the transfer of volatiles to the gas chromatograph-mass spectrometer (7890A GC-system interfaced with a 5975C VL MSD with Triple-Axis detector from Agilent Technologies, Palo Alto, California) through a heated transfer line (225°C).

Compounds were separated using a ZB-Wax capillary column (30 m long x 0.25 mm internal diameter, 0.50 µm film thickness) with a constant column pressure of 2.3 psi, and an initial flow rate of 1.4 mL/min using hydrogen as the carrier gas. The column temperature was programmed as follows: initial hold at 35°C for 10 minutes, increase to 240°C at 8°C/min, and a final hold at 240°C for 5 minutes. The mass spectrometer was operated in electron ionisation mode at 70 eV, scanning mass-to-charge ratios between 15 and 300. An empty tube and a freshly conditioned stir bar were analysed to exclude potential contaminants. Excluding controls, a total of 15 samples were analysed in duplicate, resulting in 30 chromatograms. All samples and standards were analysed in a single continuous batch over approximately one and a half days.

Data processing

Untargeted profiling of GC-MS raw data was carried out using the PARADISE software (<https://ucphchemometrics.com/paradise/>, Version 6.1.7)

(Johnsen et al. 2017). Automated data alignment, consisting of initial co-shifting to handle samples that differ significantly from the bulk, and correlation optimised warping with parameters estimated through an optimisation routine, was employed. Flexible coupling PARAFAC2 models with non-negativity constraint in all modes, were fitted for each of the intervals. Interval setting along the chromatograms and model optimisation was performed following the Quintanilla-Casas et al (2023) protocol. Saturated peaks, those where one or several m/z with relative abundances above the detector threshold, were detected and corrected according to the pre-print of Quintanilla-Casas et al (2025) – which is not yet implemented in PARADISE. Saturated signals were detected based on base peak chromatogram and set to missing, then PARAFAC2 model imputes missing values by expectation-maximisation.

Relative concentrations (peak areas) of compounds, together with the resolved mass spectra, were exported into a peak table. Identification of chemical compounds was based on spectral similarity with the NIST23 MS database (NIST/EPA/NIH Mass Spectral Library, NIST Scientific and Technical Databases, Gaithersburg, MD, USA, <https://www.nist.gov/programs-projects/nist23-updates-nist-tandem-and-electron-ionization-spectral-libraries>), through MS Search 2.0 implemented in PARADISE. Compounds marked as 'Poor/No match' (match factor <600) were excluded, and retention times were converted to retention index values for validation of compounds. Linear retention indices were calculated using a homologous series of alkanes (C6–C22) (Hewlett-Packard Co. Avondale, PA, USA) to confirm compound identities. Retention index range was obtained from the PubChem library (Kim et al. 2025). Flavour profile, aroma threshold value and taste threshold values were obtained from the FlavorDB2 database (Goel et al. 2024).

The following compounds were quantified outside of the linear range of the detector based on the base peak chromatogram: ethyl octanoate (C8), ethyl decanoate (C10), ethyl dodecanoate (C12), ethyl tetradecanoate (C14), ethyl hexadecanoate (C16), 3-methylbutyl decanoate, ethyl 9-hexadecenoate, 1-hexadecanol, ethyl oleate (C18, ethyl (Z)-octadec-9-enoate), ethyl linoleate (C18, ethyl (9Z,12Z)-octadeca-9,12-dienoate).

Statistical analyses were performed in R version 4.4.0 (R Foundation for Statistical Computing, Vienna, Austria). Peaks from validated molecules were normalised using total area normalisation. The data were log₁₀-transformed and scaled to generate the heatmaps. Heatmaps were obtained using 'ComplexHeatmap' v. 2.20.0 package (Gu 2022) and 'hclust' algorithm for hierarchical clustering. A two-fold logarithmic comparison between accelerated-aged and new make spirits was used to highlight compound variations after the accelerated ageing process. Multivariate differences in volatile organic compound profiles were assessed using permutational multivariate analysis of variance (PERMANOVA) based on a Bray-Curtis distance matrix. Group dispersion was evaluated using the test for homogeneity of multivariate dispersion (betadisper) to ensure the assumptions of PERMANOVA were met. Additionally, an analysis of similarities (ANOSIM) was performed to further evaluate the separation between groups. Statistical significance was assessed at a 95% confidence level ($p < 0.05$).

Quantification of esters

Solutions of ethyl acetate (C2), hexanoate (C6), octanoate (C8), dodecanoate (C12), tetradecanoate (C14), and hexadecanoate (C16) were prepared in absolute ethanol (99.9%). Working solutions (43% ABV) were prepared by dilution with Milli-Q water. Concentration ranges were 0.75–50 mg/L for all compounds, except ethyl acetate (2.25–75 mg/L). Peak areas obtained using PARADISE were fitted against concentration using GraphPad PRISM v10.4.0. Ethyl C2 and C6 esters followed linear regression ($R^2 = 0.99$; Supplementary Information Figure S1 a, b). Ethyl C8 and C12–C16 exhibited saturation kinetics, modelled as $c = (A \cdot c_{\text{halfmax}}) / (A_{\text{max}} - A)$, with R^2 values of 0.99 and 0.95, respectively. Ethyl C12, C14, and C16 were fitted together and are reported with the standard deviation (Supplementary Information Figure S1 c, d). For interpolation, the nearest standard was applied: ethyl acetate for ethyl acetate, ethyl C6 for C7–C8 compounds, ethyl C8 for C10 compounds (including ethyl C8), and ethyl C12–C16 for C12–C20 compounds. Only compounds with at least one data point above 0.75 mg/L (the lowest standard concentration) were considered. In some samples, the concentration of ethyl acetate ($n = 12$), ethyl

octanoate ($n = 5$), and ethyl decanoate ($n = 1$) fell outside the calibration range (see Table 2), resulting in increased uncertainty of quantification.

Results

Quantification of abundant aroma compounds

Stir bar sorptive extraction combined with gas chromatography-mass spectrometry enabled the identification of 131 compounds through untargeted GC-MS analysis using PARADISE software. After quality filtering and retention index validation, 92 compounds were retained. These compounds are reported indicating their classification, IUPAC nomenclature and indicative flavour aroma characteristics reported by FlavorDB (Supplementary Information Table S1). Despite prior tests optimising the split ratio (Materials and methods), ten molecules exceeded the linear range of the detector. They were corrected using the method of Quintanilla-Casas et al (2025). Further, the external standards for ethyl C8–C16 esters were fitted to saturation kinetics (Supplementary Information Figures S1 and 2). Absolute quantities for 18 compounds, 17 esters and hexadecan-1-ol are reported in Table 2. The highest concentrations of ethyl acetate, ethyl octanoate, and ethyl decanoate exceeded the standard calibration range. For ethyl acetate, the base peak chromatogram was used to confirm that the signal remained within the detector's linear range.

Accelerated ageing decreased the concentration of ethyl acetate in both rye and barley malt new make spirit. For rye malt, the concentrations of ethyl acetate were 25 ± 4 and 33 ± 7 mg/L, for producer 1 and 2, respectively, near the sensory threshold of 32.6 mg/L in a 46% ABV model solution (Gao et al. 2014), whereas the highest values observed exceeded 300 mg/L for barley malt (Table 2).

Profile of medium and long-chain esters

The esters in new make, accelerated-aged spirits, and whisky are presented in Figure 1. Ethyl octanoate (C8), ethyl decanoate (C10), ethyl dodecanoate (C12) and ethyl hexadecanoate (C16) exhibited the highest concentrations among the medium and long-chain ester classes (Figure 1a).

Table 2.

Compounds quantified in the aged (accelerated and traditional) spirits.

	HaNM	HaAA	KV04	ML15	SB15	LW15	LW25	LW30	BM40	P1NM	P1AA	P104	P2NM	P2AA	P204
Ethyl acetate	179 ± 53 ^a	114 ± 6 ^a	324 ± 32 ^a	190 ± 34 ^a	120 ± 8 ^a	161 ± 22 ^a	151 ± 11 ^a	135 ± 15 ^a	350 ± 107 ^a	105 ± 42 ^a	25 ± 4	207 ± 82 ^a	59 ± 6	33 ± 7	91 ± 26 ^a
2-Methylbutyl acetate	1.5 ± 0.2	0.7 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.1	0.0 ± 0.0	0.0 ± 0.0	0.1 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	0.1 ± 0.0
3-Methylbutyl acetate	9.1 ± 0.7	4.2 ± 0.3	0.4 ± 0.0	1.8 ± 0.1	0.4 ± 0.0	1.2 ± 0.2	0.4 ± 0.0	0.2 ± 0.1	0.9 ± 0.2	0.1 ± 0.0	0.2 ± 0.0	0.2 ± 0.1	1.1 ± 0.1	0.9 ± 0.1	0.3 ± 0.0
Ethyl hexanoate	2.2 ± 0.3	2.7 ± 0.2	4.4 ± 0.1	4.2 ± 0.3	9.2 ± 0.9	5.6 ± 1.4	7.4 ± 1.4	7.7 ± 0.3	9.8 ± 2.3	0.4 ± 0.0	1.2 ± 0.3	1.0 ± 0.1	0.9 ± 0.1	1.0 ± 0.1	0.5 ± 0.1
Diethyl butanedioate	0.0 ± 0.0	0.1 ± 0.0	1.6 ± 0.4	0.8 ± 0.0	0.4 ± 0.2	0.2 ± 0.1	0.1 ± 0.1	2.1 ± 0.2	1.0 ± 0.4	0.0 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
Ethyl octanoate	12.5 ± 3.9	17.8 ± 2.4	36.0 ± 1.5	41.9 ± 7.8	58.8 ± 12.0 ^a	57.6 ± 4.4 ^a	60.3 ± 11.1 ^a	104.9 ± 43.5 ^a	71.8 ± 0.7 ^a	1.1 ± 0.0	5.2 ± 0.1	2.8 ± 0.2	6.9 ± 0.8	7.2 ± 0.5	3.9 ± 0.1
2-phenylethyl acetate	2.1 ± 0.3	1.5 ± 0.0	0.0 ± 0.0	0.3 ± 0.0	0.0 ± 0.0	0.2 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.1 ± 0.1	0.1 ± 0.0	0.1 ± 0.0	0.0 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.0 ± 0.0
Ethyl decanoate	26.2 ± 4.3	26.2 ± 2.6	33.4 ± 2.1	34.7 ± 4.8	39.2 ± 3.6	36.3 ± 5.2	33.9 ± 3.5	55.8 ± 14.3 ^a	42.3 ± 3.5	2.0 ± 0.3	11.2 ± 0.7	4.7 ± 0.1	15.7 ± 1.6	18.2 ± 3.6	8.8 ± 0.6
Ethyl dodecanoate	28.2 ± 2.1	32.9 ± 2.2	42.4 ± 3.5	45.4 ± 4.7	45.1 ± 5.5	46.0 ± 5.6	21.2 ± 2.3	48.7 ± 7.2	44.9 ± 3.5	1.5 ± 0.1	6.4 ± 0.1	2.7 ± 0.1	6.8 ± 0.0	8.0 ± 0.8	6.6 ± 0.3
3-Methylbutyl decanoate	1.1 ± 0.0	0.5 ± 0.1	0.5 ± 0.0	1.1 ± 0.0	1.0 ± 0.0	0.8 ± 0.0	0.1 ± 0.0	1.5 ± 0.1	1.8 ± 0.5	0.0 ± 0.0	0.1 ± 0.0	0.0 ± 0.0	0.2 ± 0.0	0.3 ± 0.0	0.2 ± 0.0
Ethyl tetradecanoate	1.3 ± 0.2	2.3 ± 0.7	7.5 ± 1.1	5.0 ± 0.2	5.2 ± 0.1	4.3 ± 0.4	0.3 ± 0.1	13.9 ± 0.1	8.2 ± 1.7	1.3 ± 0.2	2.0 ± 0.2	1.2 ± 0.4	1.2 ± 0.2	1.2 ± 0.1	0.7 ± 0.0
1-Hexadecanol	7.3 ± 0.6	7.3 ± 0.0	1.1 ± 0.0	8.3 ± 1.7	4.0 ± 0.6	3.9 ± 0.1	2.0 ± 0.4	2.8 ± 0.2	10.1 ± 0.1	0.3 ± 0.0	0.2 ± 0.1	0.2 ± 0.1	0.1 ± 0.0	0.2 ± 0.0	0.3 ± 0.0
Ethyl hexadecanoate	8.2 ± 1.9	3.4 ± 1.4	13.4 ± 3.9	9.9 ± 1.7	16.0 ± 1.1	5.3 ± 1.2	0.5 ± 0.1	13.8 ± 2.2	15.3 ± 3.2	12.3 ± 2.6	13.8 ± 0.6	21.4 ± 2.4	16.2 ± 2.3	13.7 ± 1.2	6.1 ± 0.2
Ethyl Palmitoleate	4.3 ± 0.7	4.1 ± 0.7	8.1 ± 0.8	9.3 ± 0.1	12.0 ± 1.8	5.2 ± 0.5	0.9 ± 0.1	9.8 ± 0.3	8.8 ± 0.9	0.6 ± 0.1	0.3 ± 0.0	0.5 ± 0.0	0.4 ± 0.1	0.5 ± 0.1	0.4 ± 0.0
2-Phenylethyl decanoate	1.3 ± 0.0	0.6 ± 0.2	0.1 ± 0.0	0.7 ± 0.0	0.3 ± 0.1	0.4 ± 0.0	0.1 ± 0.0	0.7 ± 0.1	0.8 ± 0.3	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0
Ethyl Oleate	0.3 ± 0.0	0.1 ± 0.0	6.4 ± 0.2	0.6 ± 0.1	0.8 ± 0.1	0.1 ± 0.0	0.0 ± 0.0	1.7 ± 0.5	1.1 ± 0.5	0.5 ± 0.2	0.4 ± 0.0	0.8 ± 0.2	0.2 ± 0.1	0.3 ± 0.1	0.3 ± 0.0
Ethyl Linoleate	0.2 ± 0.0	0.1 ± 0.0	4.2 ± 0.9	0.6 ± 0.1	0.8 ± 0.1	0.4 ± 0.1	0.1 ± 0.0	1.5 ± 0.3	2.8 ± 0.8	9.7 ± 0.6	9.6 ± 0.0	1.6 ± 0.3	9.0 ± 0.7	8.1 ± 0.4	1.6 ± 0.0
Ethyl linolenate	0.0 ± 0.0	0.0 ± 0.0	0.1 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.1 ± 0.0	0.8 ± 0.3	0.6 ± 0.0	0.0 ± 0.0	0.5 ± 0.1	0.5 ± 0.1	0.0 ± 0.0

The most abundant ester was ethyl octanoate, with concentrations in aged barley malt whisky (15 to 40 years old) between 42-105 mg/L. All levels of medium-chain fatty acid ethyl esters exceeded the sensory thresholds of 0.03-1.12 mg/L in the 40% ABV model solution (Poisson and Schieberle 2008a; Xia et al. 2020; Zhang et al. 2025). Levels of medium-chain fatty acid ethyl esters increased in rye malt accelerated-aged spirits (Figure 1). A similar trend was seen in barley malt spirits, except for ethyl decanoate, which remained unchanged. The remaining esters exhibited trends potentially linked to new make spirit congeners.

For ethyl hexadecanoate (C16), Producer 1 exhibited a decrease in both accelerated-aged spirit and traditionally aged whisky, whereas the opposite trend was found with Producer 2 (Figure 1). In rye spirits, most long-chain esters showed similar concentrations in the new make and accelerated-aged spirits, while lower concentrations were observed in the traditionally aged spirit. For barley spirits, most long-chain esters were higher in traditionally aged spirit compared to the new make, whereas the accelerated-aged spirit showed similar values to new make.

In aged whiskies - LW30 and BM40 - the ethyl esters were highest with the exception of ethyl oleate

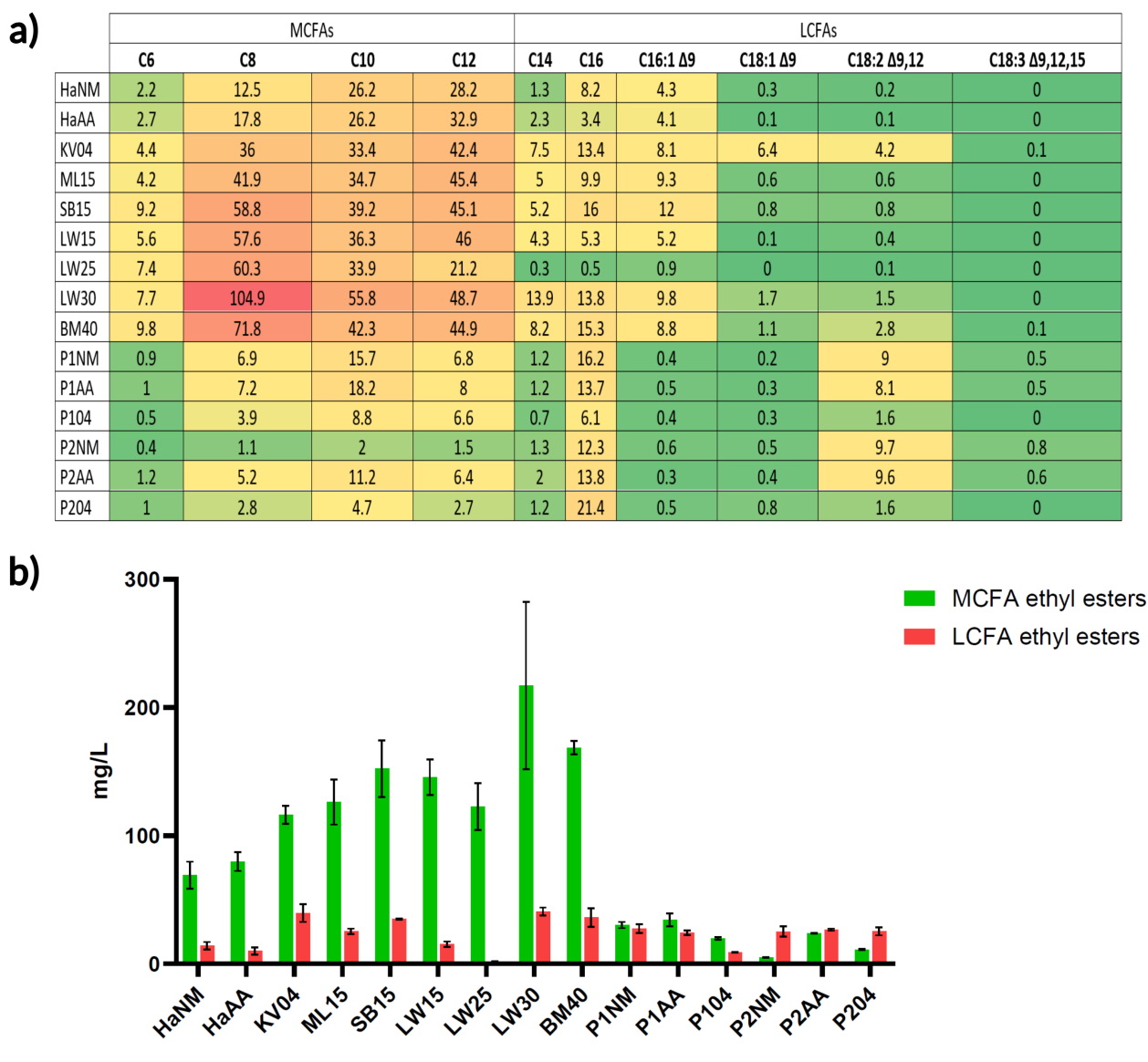
(C18:1 Δ9) and ethyl linoleate (C18:2 Δ9,12) (Table 2). However, the concentration of ethyl oleate and ethyl linoleate in these whiskies exceeded the reported threshold in a whisky model solution (34% ABV), of 0.87 and 0.45 mg/L (Salo et al. 1972). A general increase in all ethyl esters derived from both MCFAs and LCFAs was observed with extended ageing (Figure 1b). Furthermore, the total concentration of MCFAs and LCFAs ethyl esters was higher in the rye accelerated-aged spirit than in traditionally aged whisky. This trend was not observed for barley malt, where the four-year-old whisky was obtained from a different producer.

Malted grain

The relationship between compounds and malted grain was investigated through principal component analysis (Figure 2). The first two principal components accounted for 48.5% of the total variance, with principal component 1 contributing the 29.9%. Among these dimensions, confidence intervals tended to separate two clusters along the principal component 1 potentially indicating matrix specific behaviour. This assumption was further tested with PERMANOVA that indicated a significant separation between group centroids ($p = 0.001$). Furthermore, no significant dispersion within the group was observed ($p = 0.68$) and the analysis of

Figure 1.

Ester quantification. (a) Heatmap showing mean of the quantified medium-chain fatty acid (MCFA) and long-chain fatty acid (LCFA) esters. (b) Bar chart depicting the mean concentration of the sum of MCFA and LCFA esters for barley malt and rye malt samples.



Error bars represent the range calculated from two replicates.

similarities further corroborate that the validated volatile organic compounds differ significantly ($R=0.9$ $p=0.001$) according to the cereal used. Accordingly, subsequent analyses focused on either rye or barley malt whisky separately.

Rye malt

The new make spirit from two producers (Table 1) exhibited distinct patterns in ester composition (Figure 3a). Specifically, new make spirit from Producer 1 was characterised by elevated levels of

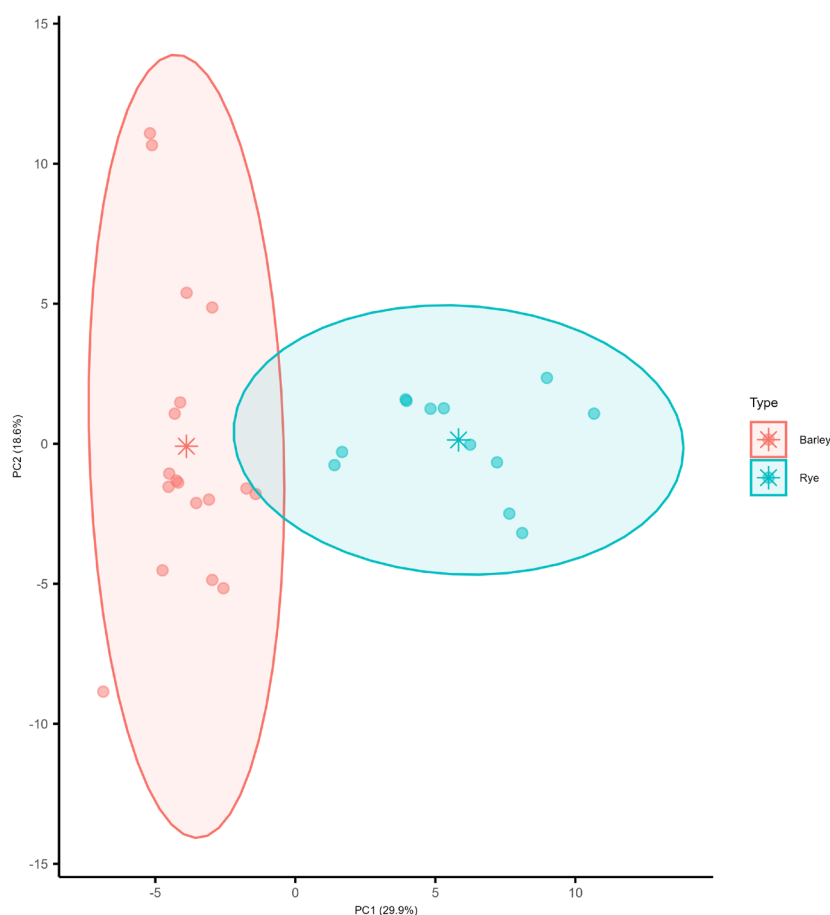
2-methylpropyl acetate, 2-methylbutyl acetate, 2-phenylethyl acetate, 3-methylbutyl acetate, 2-methylpropyl octanoate and 3-methylbutyl octanoate. In contrast, new make spirit from Producer 2 had a high relative abundance of ethyl esters such as ethyl (E)-hexadec-9-enoate, hexadecyl acetate, ethyl (Z)-octadec-9-enoate, ethyl 2-hydroxypropanoate, and ethyl hexadecanoate.

Additionally, the new make spirit from Producer 2 showed high relative abundance of several esters, including 2-phenylethyl hexanoate, ethyl (Z)-

Figure 2.

Principal component analysis plot of volatile organic compounds (VOCs) profiles.

Ellipses represent the 95% confidence intervals around group centroids. Significant differences in VOC profiles were observed between groups according to the fermented matrix.



tetradec-9-enoate, ethyl (9Z,12Z)-octadeca-9,12-dienoate, and ethyl (9Z,12Z,15Z)-octadeca-9,12,15-trienoate. These findings align with the ester levels in Figure 1a, where the new make spirit from Producer 1 exhibited high concentrations of medium-chain fatty acid esters but low levels of unsaturated long-chain esters, while that from Producer 2 displayed the opposite trend. Notably, elevated levels of ethyl benzoate were detected in the new make spirit from Producer 1 but not that of Producer 2.

The key maturation compound 'whisky lactone' [(4R,5R)-5-butyl-4-methyloxolan-2-one], increased threefold in Producer 1 and 4.5 fold in Producer 2 during accelerated ageing (Figure 3e and Supplementary Information Table S3). With ketones (Figure 3b), the new make spirit from Producer 2 exhibited high levels of heptan-2-one, tridecan-2-one, pentadecan-2-one, and 6,10,14-trimethylpentadecan-2-one, whereas new make spirit from Producer 1 had relatively high levels of heptan-2-one and pentadecan-2-one.

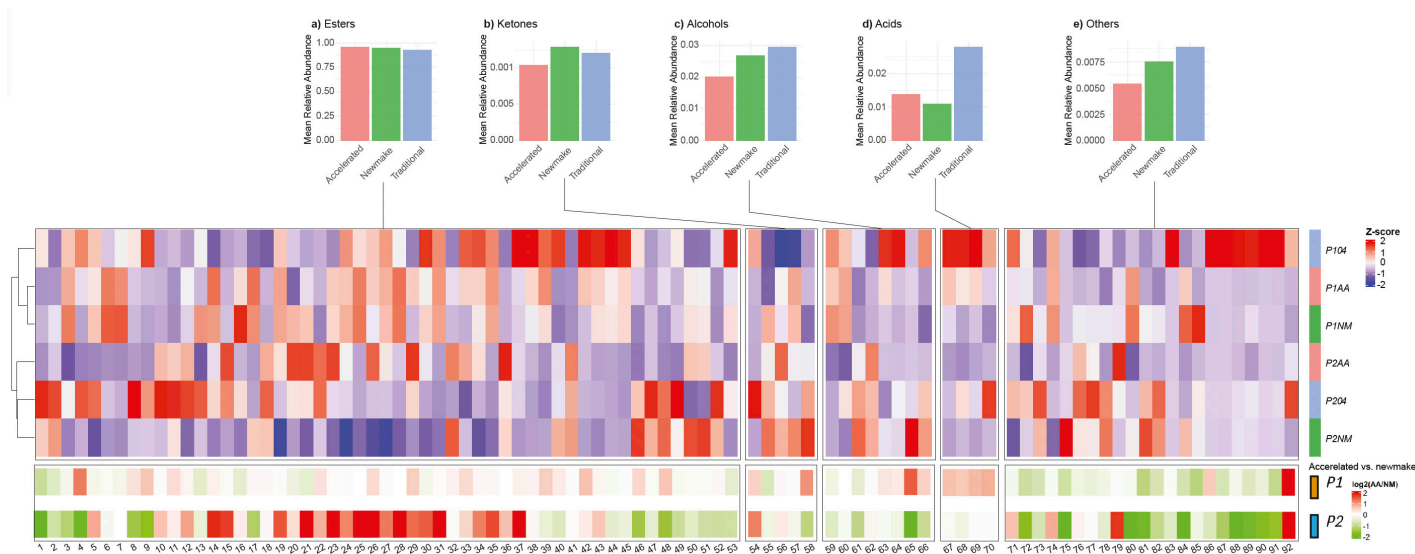
In terms of alcohols (Figure 3c), Producer 2 displayed a high relative abundance of longer alcohols, while Producer 1 was characterised by higher levels of

medium chain alcohols, such as 3-methylbutan-1-ol and 2-methylpropan-1-ol. Low relative abundances of all carboxylic acids (Figure 3d) and hydrocarbons were observed in both new make spirits.

In accelerated-aged spirits and traditionally aged whisky, patterns potentially linked to the new make spirit were observed. For Producer 1, acetate esters such as 3-methylbutyl acetate and 2-phenylethyl acetate were more abundant in the accelerated-aged spirit, while long-chain esters, including 3-methylbutyl decanoate and propyl dodecanoate, exhibited higher relative abundances in the traditionally aged spirit. Although an overall increase in esters was observed from the new make spirit to the accelerated-aged spirit, not all esters followed this trend. For example, both ethyl acetate and hexyl acetate decreased in abundance in the accelerated-aged spirit. This was confirmed by quantification, where both accelerated-aged samples showed low ethyl acetate values (Table 2). The increase in ethyl octanoate, ethyl decanoate, and ethyl dodecanoate shown in Figure 1a was corroborated by the 'fold change' between the two producers (Supplementary Information Table S3). For Producer 2, the traditionally aged whisky and

Figure 3.

Heatmap of the relative abundance of VOCs in rye malt new make spirit, traditionally aged whisky, and accelerated-aged spirit.



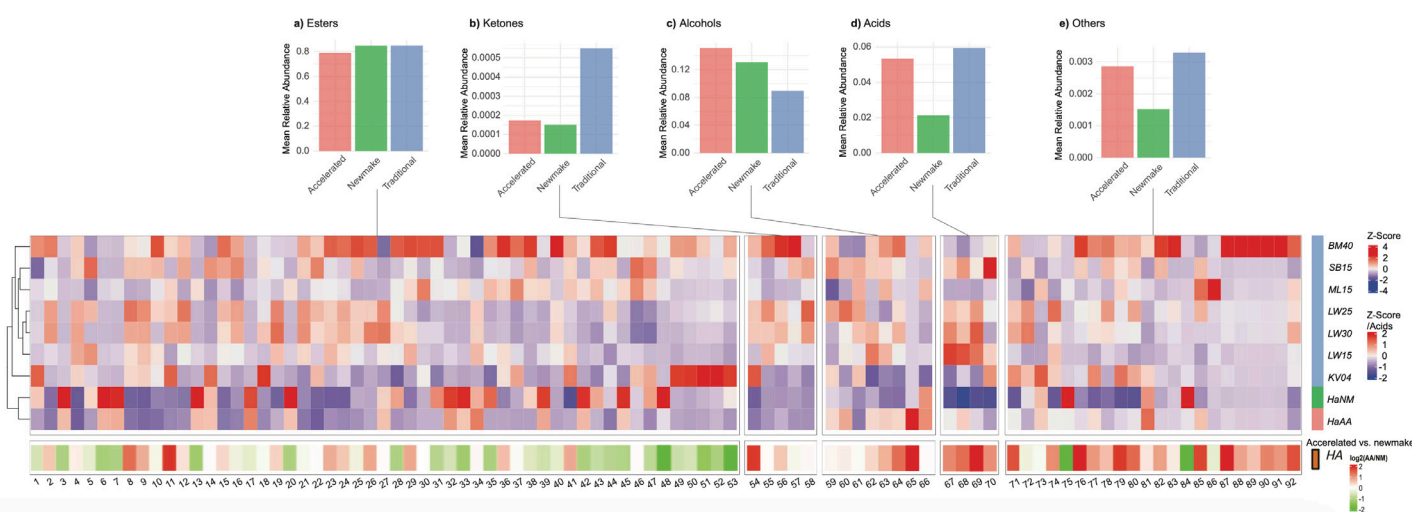
The heatmap is arranged in five VOC classes (left to right): a) esters, b) ketones, c) alcohols, d) carboxylic acids, and e) others (acetals, ethers, furans, hydrocarbons, and lactones). Above the heatmap, class specific histograms represent the mean relative abundance of each compound class according to the ageing method. On the right, samples are colour labelled according to the ageing method: green for new make spirit, red for accelerated-aged spirit, and light blue for traditionally aged whisky. At the bottom, two heatmaps based on the \log_2 fold change between accelerated-aged and new make spirit show the changes in the VOC profiles for Producer 1 and Producer 2. Visualisation of fold change values were limited to the range of -2 to 2 for clarity. Values outside this range are coloured according to the nearest limit. All fold-change values are reported in Supplementary Information Table S3.

Identity of compounds

Esters: 1 ethyl acetate; 2 ethyl 2-hydroxypropanoate; 3 methylpropyl acetate; 4 ethyl 2-methylpropanoate; 5 ethyl butanoate; 6 2-methylbutyl acetate; 7 3-methylbutyl acetate; 8 ethyl 2-methylbutanoate; 9 ethyl 3-methylbutanoate; 10 ethyl pentanoate; 11 diethyl butanedioate; 12 ethyl hexanoate; 13 hexyl acetate; 14 ethyl benzoate; 15 ethyl heptanoate; 16 2-methylpropyl hexanoate; 17 2-phenylethyl acetate; 18 ethyl 2-phenylacetate; 19 ethyl octanoate; 20 octyl acetate; 21 3-methylbutyl hexanoate; 22 ethyl (E)-non-3-enoate; 23 ethyl nonanoate; 24 propyl octanoate; 25 2-methylpropyl octanoate; 26 ethyl dec-9-enoate; 27 ethyl decanoate; 28 3-methylbutyl octanoate; 29 ethyl undecanoate; 30 propyl decanoate; 31 2-methylpropyl decanoate; 32 2-phenylethyl hexanoate; 33 dodecyl acetate; 34 ethyl dodecanoate; 35 3-methylbutyl decanoate; 36 ethyl tridecanoate; 37 propyl dodecanoate; 38 2-methylpropyl dodecanoate; 39 2-phenylethyl octanoate; 40 ethyl (Z)-tetradec-9-enoate; 41 ethyl tetradecanoate; 42 tetradecyl acetate; 43 3-methylbutyl dodecanoate; 44 ethyl pentadecanoate; 45 2-phenylethyl decanoate; 46 ethyl (E)-hexadec-9-enoate; 47 ethyl hexadecanoate; 48 hexadecyl acetate; 49 ethyl heptadecanoate; 50 ethyl (9Z,12Z)-octadeca-9,12-dienoate; 51 ethyl (9Z,12Z,15Z)-octadeca-9,12,15-trienoate; 52 ethyl (Z)-octadec-9-enoate; 53 ethyl octadecanoate. Ketones: 54 1,1-diethoxypropan-2-one; 55 heptan-2-one; 56 tridecan-2-one; 57 pentadecan-2-one; 58 6,10,14-trimethylpentadecan-2-one. Alcohols: 59 2-methylpropan-1-ol; 60 3-methylbutan-1-ol; 61 hexan-1-ol; 62 2-phenylethanol; 63 dodecan-1-ol; 64 tetradecan-1-ol; 65 2,6-ditert-butyl-4-methylphenol; 66 hexadecan-1-ol; Carboxylic acids: 67 decanoic acid; 68 dodecanoic acid; 69 tetradecanoic acid; 70 hexadecanoic acid; Others: 71 1,1-diethoxyethane; 72 1,1-diethoxy-2-methylpropane; 73 1,1-diethoxy-3-methylbutane; 74 1-(1-ethoxyethoxy)pentane; 75 2,2-diethoxyethylbenzene; 76 1,1-diethoxynonane; 77 furan-2-carbaldehyde; 78 hexanal; 79 benzaldehyde; 80 decane; 81 2-(ethoxymethyl) furan; 82 1,1-diethoxyheptane; 83 1,4-xylene; 84 cyclooctatetraene; 85 octane; 86 1,1,3-triethoxypropane; 87 1,2,4-trimethylbenzene; 88 1-ethyl-3,5-dimethylbenzene; 89 1-methyl-3-propan-2-ylbenzene; 90 1-methyl-4-propylbenzene; 91 4-ethyl-1,2-dimethylbenzene; 92 (4R,5R)-5-butyl-4-methylxolan-2-one (oak lactone).

Figure 4.

Heatmap of the relative abundance of VOCs in barley malt new make spirit, traditionally aged whisky, and accelerated-aged spirit.



The heatmap is divided into five distinct classes of VOCs, presented from left to right: a) esters, b) ketones, c) alcohols, d) carboxylic acids, and e) others (acetals, ethers, furans, hydrocarbons, and lactones). Compound identities are as detailed in the legend to Figure 3. Above the heatmap, class specific histograms represent the mean relative abundance of each compound class according to the ageing method. On the right, samples are colour labelled according to the ageing method: green for new make spirit, red for accelerated aged spirit, and light blue for traditionally aged whisky. At the bottom, a heatmap based on the \log_2 fold change between accelerated-aged and new make samples show the changes in the VOC profiles for Hafnium. Visualisation of fold change values were limited to the range of -2 to 2 for clarity. Values outside this range are coloured according to the nearest limit. All fold-change values are reported in Supplementary Information Table S3.

accelerated-aged spirits exhibited similar levels of ethyl pentanoate, ethyl hexanoate, ethyl heptanoate, and ethyl 2-phenylacetate. Conversely, the traditionally aged spirit showed a higher relative abundance of ethyl acetate, ethyl 2-methylpropanoate, and ethyl 3-methylbutanoate, whereas medium odd-chain esters - ethyl nonanoate, ethyl undecanoate, and ethyl tridecanoate - had higher relative abundances in the accelerated-aged spirit. Fusel and higher alcohols, as well as long-chain carboxylic acids, tended to increase from the new make to the accelerated-aged spirits, particularly when the corresponding esters decreased. Acetals such as 1,1-diethoxyethane and 1,1,3-triethoxypropane presented high abundance in traditionally aged spirit.

Barley malt

The spirits obtained from barley malt displayed similar clustering patterns by producer, as in rye malt (Figure 4). Whisky lactone [(4R,5R)-5-butyl-4-methyloxolan-2-one], presented high relative abundances in traditional aged whisky and the lowest fold change (1.7-fold) in accelerated aged

barley spirit (Figure 4e and Supplementary Information Table S3). Furan derivatives, such as 2-ethoxymethylfuran, were present at high relative abundance in the new make, accelerated-aged spirit, and both four and 40 year old whiskies.

When comparing the relative abundance between the new make spirit and accelerated-aged counterpart, higher levels of acetate esters, such as 3-methylbutyl acetate and 2-phenylethyl acetate, were observed in the new make spirit. In contrast, the accelerated-aged spirit showed an increased relative abundance of ethyl esters, including ethyl octanoate, and ethyl dodecanoate (Figure 1a). The relative abundance of ethyl dec-9-enoate and undecanoate increased from the new make to accelerated-aged spirits and was notably higher in the 25, 30, and 40 year-old whiskies. Additionally, the accelerated-aged spirit had higher relative abundances of alcohols (Figure 4c) (e.g. dodecan-1-ol) as well as carboxylic acids (Figure 4d). However, when compared to the new make spirit, only a few alcohols showed an increase in relative abundance. In contrast to the rye malt spirits, the barley malt spirits showed an increase in tetradecanoic acid

that paralleled the increase in its corresponding ester.

Barley malt whisky demonstrated a distinct profile across the different ageing periods. In general, ethyl 2-methylpropanoate, ethyl 2-methylbutanoate, and ethyl heptanoate showed high relative abundances with extended ageing. However, only ethyl heptanoate was detected in whisky aged 40 years. Here, a high relative abundance of ethyl undecanoate, ethyl pentadecanoate, and (Z)-tetradec-9-enoate were found, alongside a significant concentration of the lighter ethyl 2-hydroxypropanoate.

Discussion

Here, ethyl esters were quantified in both accelerated-aged spirits and traditionally aged whisky produced from the same new make spirit (Table 2). The concentration of most C2–C12 esters changed during ageing, with accelerated ageing similarly resulting in increased levels of most compounds. As a consequence, many esters exceed their sensory thresholds, indicating a contribution to the overall fruitiness of the sensory profile. Ethyl acetate was the exception with accelerated ageing using the Jensen reactor reducing the concentration to 25–30 mg/L, near or below the sensory threshold levels. Whereas the levels in long aged whisky were between 120–350 mg/L, indicating a contribution to solvent like notes (Kelly et al. 2023). Therefore, the accelerated-aged spirit would be anticipated to have a smoother profile yet still retain fruit character, pending validation by formal sensory analysis.

Whisky producers suggest that the 'funky' character of new make spirit is due to elevated levels of volatile long chain free fatty acids, which, in turn, play a key role in the formation of desirable long-chain esters during maturation. While medium-chain ethyl esters contribute fruity notes, long-chain ethyl esters add complexity and a waxy profile, which is considered a desirable trait in aged spirits (Caldeira et al. 2007; Poisson and Schieberle 2008b; Jelen et al. 2019; Wu et al. 2023). It is assumed that long-chain esters (C14–C18) increase during ageing; but the results reported here show that the total levels remain relatively stable and depend on the concentration in the new make spirit.

It is interesting that the unsaturated ethyl linoleate degrades during traditional ageing and the unsaturated ethyl tetradecanoate is produced (Figure 1a). Ethyl linoleate contains a C18 delta 9,12 unsaturated acid moiety, whereas ethyl tetradecanoate contains a C14 unsaturated moiety, and the chemical pathway for a possible degradation is not clear. It has been reported (Wu et al. 2023) that the concentration of long-chain esters does not always follow a linear trend during ageing, especially when factors such as light exposure are considered. Moreover, chilled filtration can affect the composition of these esters, leading to changes in their levels in the final product (Zhang et al. 2025). Given that multiple factors - ranging from grain selection to filtration prior to bottling - can influence the presence of these esters, further studies are required on their source and formation. However, despite the producer of new make spirit and traditional aged whisky reporting the same process, some uncertainty remains regarding the initial ester content at the time of production.

The volatile profile of rye malt and barley malt new make spirit was very different (Figure 2). This can be expected given the different substrate and processing conditions (Arnold et al. 2019; Kyraleou et al. 2021; Kelly et al. 2023). A common denominator for ageing was the increase in whisky lactone, which is a character impact aroma compound for oaked whisky. Indeed, cis-whisky lactone was generally present at low relative abundance in accelerated-aged spirit while higher levels were found in four year old rye whisky and 15 to 40 year old barley malt whisky. This suggests a lower impact on the aroma profile of accelerated-aged spirits, with a higher influence in rye spirits compared to barley.

Building on this, a broader discussion of the origins and transformations of ethyl esters during whisky production and ageing is warranted. The ageing process significantly influences the distribution of esters, shaping the overall aroma profile and sensory characteristics of whisky and grain spirits (Piggott et al. 1995; Lee et al. 2001; Kyraleou et al. 2021). In general, esterification of carboxylic acids extracted from wood, along with the evaporation of water and ethanol during ageing, increases the overall ester content of the spirit. Some of the ethyl esters in whisky are produced during fermentation,

as yeast forms these compounds from medium-chain fatty acids derived from amino acid catabolism and ethanol (Kelly et al. 2003; Saerens et al. 2007). These compounds in the wash are distilled to the new make spirit and further refined during ageing. The increased ester content observed in accelerated-aged spirits can be attributed to ultrasound induced esterification and esterification promoted by organic acids (e.g. citric acid) (Zheng et al. 2014; Mirza-Aghayan et al. 2015; Nguyen and Pham 2020).

When comparing new make spirit, accelerated-aged spirit, and traditionally aged whisky, a decrease in the relative abundance of acetate esters - such as 3-methylbutyl acetate and 2-methylbutyl acetate - was observed, while the relative abundance of ethyl esters such as ethyl hexanoate, ethyl octanoate, ethyl decanoate, and ethyl dodecanoate increased. This can be explained by the transesterification of ethyl esters, as reported by Christoph and Bauer-Christoph (2007). In traditionally aged whisky made from barley malt, the predominant ethyl ester was ethyl octanoate rather than ethyl decanoate or ethyl dodecanoate as reported for different Scotch whiskies (Goss et al. 1998). These differences may relate to the method used for ester extraction (liquid-liquid extraction and concentration of the organic phase through drying) and quantification, as well as the liquid matrix. Moreover, considering the exploratory nature of the current study, further analysis could provide a fuller understanding of quantification of ethyl esters in both traditional and accelerated-aged spirits. Nevertheless, the concentration of these esters was above their threshold values (Poisson and Schieberle, 2008a; Xia et al. 2020; Zhang et al. 2025), suggesting a contribution to the sensory characteristic of these spirits.

A high relative abundance of ethyl benzoate (herbaceous, fruity) and diethyl butanedioate (fruity) was observed in samples from both accelerated and traditional ageing. Benzoic acid and succinic acid, which is extracted from oak and sherry wood - particularly from reused barrels - can undergo esterification with ethanol in solution (Zhang et al. 2015; Valcárcel-Muñoz et al. 2021) which may explain their higher abundance.

Ethyl undecanoate contributes cognac-like and

fruity notes, increasing in relative abundance from new make spirit to accelerated-aged spirit. This compound contributes a key odour found in peated whisky (Jelen et al. 2019). Furthermore, ethyl dec-9-enoate was detected in high relative abundance in accelerated-aged spirits and, through yeast metabolism (Buglass, 2011; Wanikawa 2020), has been reported in traditionally aged whisky (Demyttenaere et al. 2003; Caldeira et al. 2007; Zhao et al. 2013; Barnes et al. 2022).

In this study, the concentration of the identified carboxylic acids varied depending on the malted cereal used and the ageing process. Given that the analysed new make spirit exhibited the lowest relative abundance, the higher relative abundance observed in the traditionally aged and accelerated-aged spirits can be attributed to differences in carboxylic acid extraction from the wood (Conner et al. 1998) and variations in equilibrium with the corresponding ethyl ester.

Other components such as 1,1-diethoxyethane and 1,1,3-triethoxypropane exhibited high relative abundances in traditionally aged spirits, regardless of the grain. Acetaldehyde and acrolein react with ethanol to form the corresponding acetals - 1,1-diethoxyethane and 1,1,3-triethoxypropane - leading to a reduction in the pungent odour of the aldehydes (Christoph and Bauer-Christoph 2007). The relative abundance of these compounds may be related to the creation of oxidation products during ageing.

In this work, the SBSE Twister method was used to characterise the volatile organic compounds in the accelerated-aged spirits and whisky. Various studies have evaluated the efficiency of SBSE in the extraction of volatiles and compared it to other methods with different alcoholic beverages. For commercial wine (14% ABV), SBSE demonstrated superior sensitivity compared to headspace-solid phase microextraction (Hjelmeland et al. 2016). However, in a compounded model spirit (10% ABV), headspace-solid phase microextraction exhibited better performance than SBSE (Barnes et al. 2022). Other studies though suggest that SBSE can significantly increase the number of detected volatile compounds when spirits, such as tequila, are diluted (Rodríguez-Olvera et al. 2019). Despite these discussions regarding the use of SBSE in

different alcoholic matrices, it was used in this study due to its effectiveness in analysing sensory-active compounds and generation of representative volatile profiles (Cordero et al. 2013).

The aroma profile of spirits analysed here was like that of previous studies, where esters - particularly ethyl esters - accounted for the highest relative abundance (Zhao et al. 2013; Barnes et al. 2022). Moreover, among the volatile organic compounds, off-flavour compounds produced during oak ageing such as 2,5-dialkylpyrazines (Wu et al. 2021) were not detected. This suggests that the accelerated-ageing treatment does not generate off-flavour compounds from the wood used during the process. Nevertheless, future studies would benefit from monitoring off-flavour compounds generated during both accelerated and traditional ageing to ensure comprehensive profiling.

The identification of decanoic acid here, contrasts with previous findings, where it was not detected using SBSE, despite its relatively high concentration (Barnes et al. 2022). This may reflect the high dilution of the spirit. Although the temperature and extraction time were similar, Barnes et al (2022) diluted their samples to 10% rather than 34% ABV for analysis. Here, decanoic acid exhibited a low relative abundance in the accelerated-aged spirit but also the traditionally aged rye whisky and commercial long-aged (15-30 years) whisky. Therefore, a high sample dilution may have reduced the concentration of decanoic acid below that of the detection limit.

The aroma threshold of volatile organic compounds is influenced by the ethanol concentration of the solution. While sensory protocols suggest 'nosing' whisky and grain spirits at 20% ABV to reduce sensory fatigue (Jack 2014; Wanikawa 2020; Baxter et al. 2024), research on micelle formation in spirits has suggested that changes in aroma perception occur at 23% ABV (Boothroyd et al. 2012). The formation of nanoscale agglomerates with whisky diluted to 20–25% ABV supports this hypothesis (Carrithers et al. 2020).

Karlsson and Friedman (2017) investigated the impact of the dilution of whisky with water on molecular distribution. They demonstrated that the spatial orientation of some molecules is dependent

on alcohol concentration, with some preferentially locating at the water–air interface or within the ethanol-rich environment, thereby modulating partition coefficients and volatile release dynamics. Salo et al (1972) using a model whisky at 40.8% ABV (34% w/w) reported odour threshold values for ethyl palmitoleate (C16:1 Δ 9), ethyl oleate (C18:1 Δ 9), and ethyl linoleate (C18:2 Δ 9,12) that were similar or below the detected concentrations of these compounds. Moreover, research indicated that long-chain esters are correlated with enhanced mellowness and aroma persistence (Wu et al. 2023) suggesting a positive contribution of these compounds that should be further evaluated by sensory analyses.

The common aroma descriptors reported for the whisky and accelerated-aged spirits include notes of malt and dried fruits, with a light woody character. These align with multiple aroma descriptors associated with the volatile organic compounds identified in this study. Moreover, many key aroma compounds characteristic of whisky were detected with varying abundances in both traditional whisky and accelerated-aged spirits, including 1,1-diethoxyethane (with a candy-like aroma), 2-methylpropan-1-ol (malty), 3-methylbutan-1-ol (burnt, whisky-like), ethyl acetate, ethyl 2-methylpropanoate, ethyl butanoate, ethyl 2-methylbutanoate (fruity), ethyl 3-methylbutanoate (fruity), 3-methylbutyl acetate (banana), ethyl pentanoate (yeast), ethyl hexanoate (green apple, anise), ethyl octanoate (apricot, winey), ethyl decanoate, ethyl 2-phenylacetate (flowery), ethyl dodecanoate (fruity), and *cis*-whisky lactone (coconut-like) (Caldeira et al. 2007; Poisson and Schieberle 2008b; Jelen et al. 2019). This suggests similarities in the sensory profiles of traditional whisky and accelerated-aged spirits.

Conclusions

Traditional whisky is part of the cultural heritage of distilled spirits and grain spirits will remain a major segment of the market. However, evaluating products obtained through innovative ageing technologies could provide both useful insights and benefits for the industry. In this study, the volatile organic compounds were analysed in new make spirit, traditionally aged whisky, and accelerated-aged spirits, observing a general increase in ethyl

esters from new make to accelerated-aged spirits. The results of this exploratory study should be further validated with a larger number of samples together with monitoring the accelerated aging process to better understand the chemical reactions of congeners during ageing. Sensory analysis and evaluation of the non-volatile component (e.g. total acidity, tannins, solids) should be taken into consideration. Lastly, life cycle assessment and cost analysis would provide deeper insight into the environmental sustainability and economic feasibility of accelerated ageing technology.

Author contributions

Marco Cardin: formal analysis, data curation, visualisation, writing (original draft).

Sebastian Reimer Bendtsen: experimental work, resources, data curation.

Beatriz Quintanilla-Casas: data analysis, resources. Aleksander Byzdra: resources.

Mikael Agerlin Petersen: methodology, experimental work, resources.

Tobias Emil Jensen: conceptualisation, resources.

Sylvester Holt: conceptualisation, methodology, data analysis, formal analysis, visualisation, supervision, writing (review and editing).

Acknowledgements

Samples of traditional aged barley malt whisky were donated by Juul's Engros, Frederiksberg, Denmark.

Conflict of interest

The authors declare no financial conflicts of interest. EtOH Spirits provided the distilled spirits, contributed to discussions and drafting of the manuscript. EtOH Spirits had no influence over the interpretation of the data or the final conclusions that were drawn.

References

Arnold RJ, Ochoa A, Kerth CR, Miller RK, Murray SC. 2019. Assessing the impact of corn variety and Texas terroir on flavor and alcohol yield in new make bourbon whiskey. *PLoS One* 14:e0220787. <https://doi.org/10.1371/journal.pone.0220787>

Autonomous Region of Madeira, 2015. Regional Secretariat of Environment and Natural Resources Ordinance no. 38/2015. <https://madeirawijn.nl/wp-content/uploads/2022/05/Madeira-wijnwetgeving.pdf>

Barnes Q, Vial J, Thiébaud D, De Saint Jores C, Steyer D, Contamin MA, Papaiconomou N, Fernandez X. 2022. Characterization of flavor compounds in distilled spirits: Developing a versatile analytical method suitable for micro-distilleries. *Foods* 11:3358. <https://doi.org/10.3390/foods11213358>

Baxter I, Réveillé A, Conner J. 2024. Guidelines to optimise the sensory detection of 2,4,6-trichloroanisole in neutral spirit and whisky. *J Inst Brew* 130:112-123. <https://doi.org/10.58430/jib.v130i2.51>

Bellut K, Lynch KM, Arendt EK. 2021. Alcoholic beverages: production, trends, innovations, p 19-24. In Lavelle C, This H, Kelly AL, Burke R (eds), *Handbook of Molecular Gastronomy*, CRC Press. <https://doi.org/https://doi.org/10.1201/9780429168703>

Boothroyd EL, Linforth RST, Cook DJ. 2012. Effects of ethanol and long-chain ethyl ester concentrations on volatile partitioning in a whisky model system. *J Agric Food Chem* 60:9959-9966. <https://doi.org/10.1021/jf3022892>

Buglass AJ. 2011. *Handbook of Alcoholic Beverages: Technical, Analytical and Nutritional Aspects*. John Wiley & Sons. <https://doi.org/10.1002/9780470976524>

Caldeira M, Rodrigues F, Perestrelo R, Marques JC, Câmara JS. 2007. Comparison of two extraction methods for evaluation of volatile constituents patterns in commercial whiskeys. Elucidation of the main odour-active compounds. *Talanta* 74:78-90. <https://doi.org/10.1016/j.talanta.2007.05.029>

Canas S, Caldeira I, Anjos O, Belchior AP. 2019. Phenolic profile and colour acquired by the wine spirit in the beginning of ageing: alternative technology using micro-oxygenation vs traditional technology. *LWT* 111:260-269. <https://doi.org/10.1016/j.lwt.2019.05.018>

- Canas S, Danalache F, Anjos O, Fernandes TA, Caldeira I, Santos N, Fargeton L, Boissier B, Catarino S. 2020. Behaviour of low molecular weight compounds, iron and copper of wine spirit aged with chestnut staves under different levels of micro-oxygenation. *Molecules* 25:5266. <https://doi.org/10.3390/molecules25225266>
- Conner JM, Paterson A, Birkmyre L, Piggott JR. 1999. Role of organic acids in maturation of distilled spirits in oak casks. *J Inst Brew* 105:287–291. <https://doi.org/10.1002/j.2050-0416.1999.tb00522.x>
- Conner J. 2014. Maturation, p 199–220. In Russell I, Stewart G (eds) *Whisky: Technology, Production and Marketing*. 2nd ed. Amsterdam: Elsevier. <https://doi.org/10.1016/B978-0-12-401735-1.00011-8>
- Carrithers AD, Brown MJ, Rashed MZ, Islam S, Velev OD, Williams SJ. 2020. Multiscale self-assembly of distinctive weblike structures from evaporated drops of dilute American whiskeys. *ACS Nano* 14:5417–5425. <https://doi.org/10.1021/acsnano.9b08984>
- Chemat F, Rombaut N, Sicaire AG, Meullemiestre A, Fabiano-Tixier AS, Abert-Vian M. 2017. Ultrasound assisted extraction of food and natural products. Mechanisms, techniques, combinations, protocols and applications. A review. *Ultrason Sonochem* 34:540–560. <https://doi.org/10.1016/j.ultsonch.2016.06.035>
- Christoph N, Bauer-Christoph C. 2007. Flavour of spirit drinks: raw materials, fermentation, distillation, and ageing, p 99–239. In Berger RG (ed), *Flavours and Fragrances*, Springer. https://doi.org/10.1007/978-3-540-49339-6_10
- Code of Federal Regulation. 2024. Title 27 Alcohol, Tobacco Products and Firearms. <https://www.govinfo.gov/content/pkg/CFR-2020-title27-vol3/pdf/CFR-2020-title27-vol3>.
- Cordero C, Cagliero C, Liberto E, Nicolotti L, Rubiolo P, Sgorbini B, Bicchi C. 2013. High concentration capacity sample preparation techniques to improve the informative potential of two-dimensional comprehensive gas chromatography–mass spectrometry: application to sensomics. *J Chromatogr A* 1318:1–11. <https://doi.org/10.1016/j.chroma.2013.09.065>
- Dabija D, Nastase CE, Chetrariu A, Dabija A. 2024. Multi-criteria analysis in circular economy principles: using AHP model for risk assessment in sustainable whisky production. *Computation* 12:206. <https://doi.org/10.3390/computation12100206>
- Delgado-González MJ, Sánchez-Guillén MM, García-Moreno MV, Rodríguez-Dodero MC, García-Barroso C, Guillén-Sánchez DA. 2017. Study of a laboratory-scaled new method for the accelerated continuous ageing of wine spirits by applying ultrasound energy. *Ultrason Sonochem* 36:226–235. <https://doi.org/10.1016/j.ultsonch.2016.11.031>
- Demyttenaere JCR, Sanchez Martínez JI, Verhé R, Sandra P, De Kimpe N. 2003. Analysis of volatiles of malt whisky by solid-phase microextraction and stir bar sorptive extraction. *J Chromatogr A* 985:221–232. [https://doi.org/10.1016/S0021-9673\(02\)01471-1](https://doi.org/10.1016/S0021-9673(02)01471-1)
- European Union. 2008. Regulation No 110/2008 of the European Parliament and of the Council. <https://eur-lex.europa.eu/legal-content/EN/TXT/?uri=CELEX:32008R0110>
- European Union. 2019. Regulation 2019/787 of the European Parliament and of the Council of 17 April 2019 on the definition, description, presentation and labelling of spirit drinks. <https://eur-lex.europa.eu/eli/reg/2019/787/oj>
- Gao W, Fan W, Xu Y. 2014. Characterization of the key odorants in light aroma type Chinese liquor by gas chromatography-olfactometry, quantitative measurements, aroma recombination, and omission studies. *J Agric Food Chem* 62:5796–5804. <https://doi.org/10.1021/jf501214c>

- Goel M, Grover N, Batra D, Garg N, Tuwani R, Sethupathy A, Bagler G. 2022. FlavorDB2: an updated database of flavor molecules. *J Food Sci* 87:17298. <https://doi.org/10.1111/1750-3841.17298>
- Gollihue J, Pook VG, DeBolt S. 2021. Sources of variation in bourbon whiskey barrels: a review. *J Inst Brew* 127:660–671. <https://doi.org/10.1002/jib.660>
- Goss KA, Alharethi R, Laposata M. 1999. Fatty acid ethyl ester synthesis in the preparation of Scotch whiskey. *Alcohol* 19:213–218. [https://doi.org/10.1016/S0741-8329\(98\)00057-3](https://doi.org/10.1016/S0741-8329(98)00057-3)
- Grand View Research. 2024a. Whiskey tourism market size, share & trends analysis report by experience type (distillery tours/tasting experiences, whiskey festival), by whiskey type (single malt whiskey, blended whiskey, bourbon, craft whiskey), by region and segment forecasts. <https://www.grandviewresearch.com/industry-analysis/whiskey-tourism-market-report>
- Grand View Research. 2024b. Whiskey market size, share & trends analysis report by product (malt, wheat, rye, corn, blended, others), by quality (premium, high-end premium, super premium), by region and segment forecasts, 2025-2030. <https://www.grandviewresearch.com/industry-analysis/whiskey-market>
- Gu Z. 2022. Complex heatmap visualization. *iMeta* 1:e43. <https://doi.org/10.1002/imt2.43>
- Hjelmeland AK, Wylie PL, Ebeler SE. 2016. A comparison of sorptive extraction techniques coupled to a new quantitative, sensitive, high throughput GC-MS/MS method for methoxypyrazine analysis in wine. *Talanta* 148:336–345. <https://doi.org/10.1016/j.talanta.2015.10.086>
- Jack F. 2014. Sensory analysis, p 229–242. In Russell I, Stewart GG, Kellershohn J (eds), *Whisky: Technology, Production and Marketing*, Elsevier, <https://doi.org/10.1016/B978-0-12-401735-1.00013-1>
- Jeleń HH, Majcher M, Szwengiel A. 2019. Key odorants in peated malt whisky and its differentiation from other whisky types using profiling of flavor and volatile compounds. *LWT* 107:56–63. <https://doi.org/10.1016/j.lwt.2019.02.070>
- Jensen PA, Trinh NT, Lundgaard Christensen R, Dam-Johansen K, Bech N. 2015. Rotor for a pyrolysis centrifuge reactor. Patent No. WO2015150265.
- Jensen TE, Byzdra A. 2024. A process for accelerating aging of spirits. Patent No. WO2024079347A1. <https://patents.google.com/patent/WO2024079347A1/en?q=Patent+No.+WO2024079347A1>
- Johnsen LG, Skou PB, Khakimov B, Bro R. 2017. Gas chromatography–mass spectrometry data processing made easy. *J Chromatogr A* 1503:57–64. <https://doi.org/10.1016/j.chroma.2017.04.052>
- Karlsson BCG, Friedman R. 2017. Dilution of whisky – the molecular perspective. *Sci Rep* 7:6842. <https://doi.org/10.1038/s41598-017-06423-5>
- Kelly TJ, O'Connor C, Kilcawley KN. 2023. Sources of volatile aromatic congeners in whiskey. *Beverages* 9:64. <https://doi.org/10.3390/beverages9030064>
- Kim S, Chen J, Cheng T, Gindulyte A, He J, He S, Li Q, Shoemaker BA, Thiessen PA, Yu B, Zaslavsky L, Zhang J, Bolton EE. 2025. PubChem 2025 update. *Nucleic Acids Res* 53:D1516–D1525. <https://doi.org/10.1093/nar/gkae1059>
- Krüger RT, Alberti A, Nogueira A. 2022. Current technologies to accelerate the aging process of alcoholic beverages: a review. *Beverages* 8:65. <https://doi.org/10.3390/beverages8040065>
- Kyrleou M, Herb D, O'Reilly G, Conway N, Bryan T, Kilcawley KN. 2021. The impact of terroir on the flavour of single malt whisk(e)y new make spirit. *Foods* 10:443. <https://doi.org/10.3390/foods10020443>

- Lee M, Paterson A, Piggott JR, Richardson GD. 2001. Origins of flavour in whiskies and a revised flavour wheel: a review. *J Inst Brew* 107:287–313. <https://doi.org/10.1002/j.2050-0416.2001.tb00099.x>
- Li H, Ming X, Liu Z, Xu L, Xu D, Hu L, Mo H, Zhou X. 2021. Accelerating vinegar aging by combination of ultrasonic and magnetic field assistance. *Ultrason Sonochem* 78:105708. <https://doi.org/10.1016/j.ultsonch.2021.105708>
- Lin JY, Wu YJ, Hsiao YT, Wang CY. 2021. Acceleration of the ageing process in mei (*Prunus mume* Siebold) liqueur by high hydrostatic pressure treatment. *Int J Food Sci Technol* 56:2030–2040. <https://doi.org/10.1111/ijfs.14835>
- Madrera RR, Valles BS, García YD, Argüelles PdV, Lobo AP. 2010. Alternative woods for aging distillates – an insight into their phenolic profiles and antioxidant activities. *Food Sci Biotechnol* 19:1129–1134. <https://doi.org/10.1007/s10068-010-0161-4>
- Miller GH 2024. Maturation, p 377-478. *Distillation Theory. In Whisky Science: A Condensed Distillation Springer Int Pub.* <https://doi.org/10.1007/978-3-031-50687-1>
- Mirza-Aghayan M, Zonoubi S, Molaee Tavana M, Boukherroub R. 2015. Ultrasound assisted direct oxidative esterification of aldehydes and alcohols using graphite oxide and oxone. *Ultrason Sonochem* 22:359–364. <https://doi.org/10.1016/j.ultsonch.2014.05.012>
- Morgan N. 2022. Master of Malt blog. Accelerated ageing, in search of whisky's Holy Grail. <https://www.masterofmalt.com/blog/post/accelerated-ageing-in-search-of-whiskys-holy-grail.aspx/>
- Nguyen DT, Pham QT. 2020. A theoretical and experimental study on esterification of citric acid with the primary alcohols and the hydroxyl groups of cellulose chain (n = 1–2) in parched condition. *J Chem* 2020:8825456. <https://doi.org/10.1155/2020/8825456>
- Niu Y, Yao Z, Xiao Z, Zhu G, Zhu J, Chen J. 2018. Sensory evaluation of the synergism among ester odorants in light aroma-type liquor by odour threshold, aroma intensity and flash GC electronic nose. *Food Res Int* 113:102–114. <https://doi.org/10.1016/j.foodres.2018.01.018>
- Piggott JR, Conner JM. 1995. Whiskies, p 247-274. In Lea AGH, Piggott JR (eds) *Fermented Beverage Production*. Boston, MA: Springer. <https://doi.org/10.1007/978-1-4757-5214-4>
- Piggott JR, Conner JM, Paterson A. 1995. Flavour development in whisky maturation. *J Inst Brew* 101:327–335. [https://doi.org/10.1016/S0167-4501\(06\)80261-X](https://doi.org/10.1016/S0167-4501(06)80261-X)
- Poisson L, Schieberle P. 2008a. Characterization of the key aroma compounds in an American bourbon whisky by quantitative measurements, aroma recombination, and omission studies. *J Agric Food Chem* 56:5820–5826. <https://doi.org/10.1021/jf800383v>
- Poisson L, Schieberle P. 2008b. Characterization of the most odor-active compounds in an American bourbon whisky by application of the aroma extract dilution analysis. *J Agric Food Chem* 56:5813–5819. <https://doi.org/10.1021/jf800382m>
- Quintanilla-Casas B, Bro R, Hinrich JL, Davie-Martin CL. 2023. Tutorial on PARADISE: PARAFAC2-based Deconvolution and Identification System for processing GC–MS data. *Protocol Exchange* <https://doi.org/10.21203/rs.3.pex-2143/v1>
- Quintanilla-Casas B, Hinrich JL, Schneide PA, Bro R. 2025. Automated correction of saturated peaks in untargeted GC-MS: a chemometrics approach. Pre-print. https://papers.ssrn.com/sol3/papers.cfm?abstract_id=5414329
- Rodríguez-Olvera MG, Rodríguez-Rodríguez LI, Qian MC, Qian YP, Vazquez-Landaverde PA. 2019. Implementation of stir bar sorptive extraction (SBSE) for the analysis of volatile compounds in tequila. *ACS Symp Ser* 1321:311–324. <https://doi.org/10.1021/bk-2019-1321.ch019>

- Russell I, Kellershohn J. 2018. Advances in technology and new product development in the beer, wine, and spirit industry, pp. 89–104. In Panda SK, Shetty PH (eds), *Innovation and Trends in the Global Food Systems*, Springer. https://doi.org/https://doi.org/10.1007/978-3-319-74820-7_5
- Saerens SMG, Delvaux F, Verstrepen KJ, Van Dijck P, Thevelein JM, Delvaux FR. 2008. Parameters affecting ethyl ester production by *Saccharomyces cerevisiae* during fermentation. *Appl Environ Microbiol* 74:454–461. <https://doi.org/10.1128/AEM.01616-07>
- Salo P, Nykänen L, Suomalainen H. 1972. Odour thresholds and relative intensities of volatile aroma components in an artificial beverage imitating whisky. *J Food Sci* 37:394–398. <https://doi.org/10.1111/j.1365-2621.1972.tb02647.x>
- Stitzel F. 1880. Rack for tiering barrels. US Patent RE9175E.
- Valcárcel-Muñoz MJ, Guerrero-Chanivet M, García-Moreno MV, Rodríguez-Dodero MC, Guillén-Sánchez DA. 2021. Comparative evaluation of Brandy de Jerez aged in American oak barrels with different times of use. *Foods* 10:288. <https://doi.org/10.3390/foods10020288>
- Wanikawa A. 2020. Flavors in malt whisky: a review. *J Am Soc Brew Chem* 78:156–167. <https://doi.org/10.1080/03610470.2020.1795795>
- Wu J, Liu Y, Zhao H, Huang M, Sun Y, Zhang J, Sun B. 2021. Recent advances in the understanding of off-flavors in alcoholic beverages: generation, regulation, and challenges. *J Food Compos Anal* 103:104117. <https://doi.org/10.1016/j.jfca.2021.104117>
- Wu Y, Chen H, Huang H, Chen F, Hong J, Zhao D, Zhang C, Zhao Z, Wang S, Ao R, Sun B. 2023. Revelation for the influence mechanism of long-chain fatty acid ethyl esters on the Baijiu quality by multicomponent chemometrics combined with modern flavor sensomics. *Foods* 12:1267. <https://doi.org/10.3390/foods12061267>
- Wu Y, Chen H, Huang H, Chen F, Hong J, Zhao D, Zhang C, Zhao Z, Wang S, Ao R, Sun B. 2023. Revelation for the influence mechanism of long-chain fatty acid ethyl esters on the Baijiu quality by multicomponent chemometrics combined with modern flavor sensomics. *Foods* 12:1267. <https://doi.org/10.3390/foods12061267>
- Xia Y, Liu Y, Wang J, Shuang Q. 2020. Assessment of key aroma compounds in fresh jujube brandy by GC-O-MS and odor activity value. *J Food Process Preserv* 44:e14494. <https://doi.org/10.1111/jfpp.14494>
- Zhang B, Cai J, Duan CQ, Reeves MJ, He F. 2015. A review of polyphenolics in oak woods. *Int J Mol Sci* 16:6978–7014. <https://doi.org/10.3390/ijms16046978>
- Zhang Q, Wang D, Liu X, Li Y, Sun J, Meng N, Ji Z, Lan Y, Cao X, Sun B, Li J. 2025. Flavor characteristics and formation mechanisms in spirits: a case study in whisky. *Food Res Int* 178:115901. <https://doi.org/10.1016/j.foodres.2025.115901>
- Zheng X, Zhang M, Fang Z, Liu Y. 2014. Effects of low frequency ultrasonic treatment on the maturation of steeped greengage wine. *Food Chem* 162:264–271. <https://doi.org/10.1016/j.foodchem.2014.04.071>
- Zhao YP, Zheng XP, Song P, Sun ZL, Tian TT. 2013. Characterization of volatiles in the six most well-known distilled spirits. *J Am Soc Brew Chem* 71:161–169. <https://doi.org/10.1094/ASBCJ-2013-0625-01>