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The Ultimate
ISLAY SINGLE MALT
SCOTCH WHISKY

THE IMPACT OF MICRO-GRAVITY ON THE RELEASE OF OAK EXTRACTIVES INTO SPIRIT

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Introduction

In August 2011, an approach was made to Moët Hennessy USA by a scientific research company called NanoRacks, LLC based in Houston, Texas, USA. NanoRacks designs and implements research programmes aboard the International Space Station via a Space Act Agreement with NASA, in conditions of micro-gravity, compared to the conditions on Earth. Given its very particular taste profile, the Ardbeg Islay Single Malt Scotch Whisky was already well known to the scientists of NanoRacks, and they had developed an idea, involving some form of experiment, or examination, of the effect of micro-gravity on the behaviour of terpenes, the building blocks of flavour for whisky spirits as well as for many other foods and wines, as research into terpenes in micro-gravity was limited. We were therefore offered an opportunity to participate in this experiment, including contributing to the design of it, however, our timescale for participation was extremely tight.

We collected a quantity of Ardbeg distillate (the liquid resulting from distillation which is normally filled into oak barrels for maturation), along with oak wood shavings from the inside of a charred American White Oak ex-Bourbon barrel, which was due to be despatched from the cooperage to Ardbeg Distillery on the Island of Islay for filling with new Ardbeg distillate. These materials were carefully packaged and sent to the NanoRacks scientists in Houston, where they were packaged into their small sample testing system, known as MixStix™, which in turn were sent to Kazakhstan to be loaded on to the Soyuz booster rocket destined for the International Space Station. A number of the MixStix™ were also sent to us to use as controls.

Three days after launch, the MixStix™ were passed over to astronauts aboard the International Space Station. After an initial period of acclimatisation to the conditions aboard the ISS, in January 2012, the experiment was initiated, as the astronauts broke the glass separating walls in the individual MixStix™, thus allowing the distillate and the oak wood shavings to come into contact with each other. At the same time on Earth, we initiated the control experiment by breaking the separating wall in my MixStix™ on Islay (which had been sent back to me at Ardbeg Distillery from NanoRack's laboratories in Houston, USA).

The MixStix™ vials remained on the International Space Station until September 2014, finally returning to the Baikonur Cosmodrome in Kazakhstan on 12th September 2014. The vials were in conditions of micro-gravity, with the distillate and oak wood in contact, for a total of 971 days, orbiting the Earth 15 times a day during this period. The MixStix™ vials were delivered back to Ardbeg in November 2014, after which the distillate from both the micro-gravity and Earth control samples was carefully extracted. A range of comparative analyses were then carried out, to determine if there were any differences between the two sets of samples.

Aims

The key aims of the experiment were as follows:-

(i) To determine if conditions of micro-gravity would have an impact on the range of naturally occurring terpene compounds and on the extraction of key flavour-active compounds in oak wood by Scotch malt distillate.

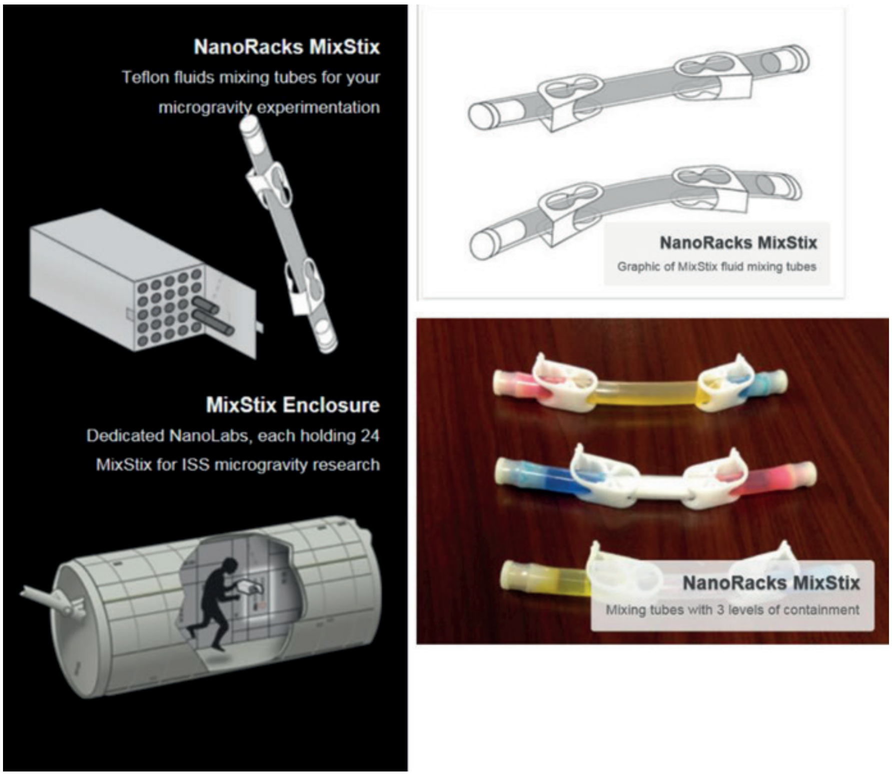
(ii) To assess the impact of maturation in conditions of micro-gravity on the flavour profile of oak matured distillate.

(iii) To investigate the presence of novel compounds and flavours in model maturation systems for Scotch malt distillate, as a result of conditions of micro-gravity.

The key findings of the above three aims would then be considered in conjunction with actual maturation conditions on Earth to investigate techniques for developing novel flavours in Ardbeg Islay Single Malt Scotch Whisky.

Technical Methodology

MixStix™ technology, which provides a convenient platform for small scale sample experimentation, is a trade-marked piece of technology used by NanoRacks. Essentially, they are small glass vials which can hold a range of materials; in our case these materials were 6ml of Ardbeg new spirit distillate, along with a small quantity of charred oak wood shavings (from a once used American oak Bourbon barrel), c. 1mm x 1mm x 6cm in size. The two materials are separated from each other by a glass dividing partition, which is broken by bending the Teflon outside covering of the MixStix™ at time of experiment activation, thus allowing the two separate components to mix together. These are illustrated below.



NANORACKS 'MixStix' Small Scale Experimentation Platform

An examination of both International Space Station and control (Earth) samples was carried out by three distinct types of analyses, namely gas chromatography (GC) for major volatile congener analysis, high pressure liquid chromatography (HPLC) for key maturation related congener analysis, and organoleptic assessment (nosing and tasting) to determine spirit character. In addition to these, a more detailed profile of components in the distillate was obtained using headspace solid phase micro-extraction combined with gas chromatography –mass spectrometry (GC-MS).

The GC and HPLC analyses were carried out by both ourselves and an independent laboratory to provide independent validation of results. The organoleptic assessment was only carried out by ourselves due to the very limited quantities of liquid, and the very specialised technique of GC-MS was only carried out by the independent laboratory.

GC – Analysis carried out with an Agilent Technologies 7890 Gas Chromatogram with flame ionisation detector.

HPLC – Analysis carried out with an Agilent Technologies 1260 High Pressure Liquid Chromatogram with multiple wavelength detector.

GC-MS – Analysis carried out at an independent laboratory using a Thermo Trace GC-MS.

Organoleptic Assessment – multiple micro-gravity and control samples were compared in the sensory laboratory using Ardbeg 'tulip' shaped nosing and tasting glasses, for both triangle tests (in which three 'blind' glasses are compared, two of which contain one sample, and one the other sample) and for detailed aroma and flavour descriptions. The results were collated to assess for any flavour differences.

Results & Discussion

The NanoRacks' MixStix™ provided an opportunity to send some materials, namely new make distillate and barrel oak wood shavings, up into space to understand the effects on terpenes and whisky distillate. While it is clear that the preparation of the oak wood samples, required to fit them into the MixStix™, led to a set of conditions which did not perfectly recreate the surface area to volume ratios which would actually exist in a 200 litre capacity American barrel, it did provide us with the opportunity to assess the potential impact of micro-gravity in a model system. This has to be borne in mind if attempting to extrapolate the results on to what may happen on the full scale, in a distillery maturation warehouse.

GC analysis of major volatile congeners demonstrated no significant differences between the International Space Station samples and the control samples on Earth. Since this group of compounds comprising primarily alcohols, aldehydes, ketones and esters, are produced during fermentation and concentrated up, or in some cases, catalysed during distillation, their presence and quantities should have been identical in each set of samples to start off with. The conditions of micro-gravity on the ISS have not impacted on their levels, as demonstrated by the similar levels in the Earth based samples (Table 1.)

Analyte	Space station samples	Earth 'control' samples
Acetaldehyde	54.7	53.8
Ethyl Acetate	151.4	155.4
Acetal	88.9	89.2
n-Propanol	253.2	242.7
iso-Butanol	470.1	450.7
iso-Amyl Acetate	10.5	11.8
n-Butanol	6.5	6.3
Sum of 2- and 3-Methyl-1-Butanol	1236.3	1169.1
2-Methyl-1-Butanol	312.8	295.6
3-Methyl-1-Butanol	923.5	873.5
n-Pentanol	250.7	250.7
Total Higher Alcohols	1959.6	1862.5
(Sum of 2- and 3-Methyl-1-Butanol)/iso-Butanol	2.6	2.6
3-Methyl-1-Butanol/2-Methyl-1-Butanol	3.0	3.0
Furfural	47.1	67.8

TABLE 1 Major Volatile Congener Analysis (mg/l @ sample strength)

A more detailed investigation of these compounds, along with phenolic compounds, was carried out on our behalf by external whisky experts and scientists, using GC-MS. In the resulting chromatograms, the areas of 75 separate peaks were integrated, most of which were identified as alcohols, esters, acetals and phenols. Again, no major differences were found between the ISS and Earth samples. From an Ardbeg perspective, this was particularly interesting as the conditions on the ISS have not impacted on the absolute quantities of the various phenolic compounds. These are the compounds which contribute the powerful, smoky, medicinal flavours to Ardbeg spirit.

The most significant variable between the ISS and Earth samples was discovered when the results of the HPLC analysis of key maturation related congeners (wood extractives) were considered. The absolute concentration of these compounds was far higher than would normally be expected in standard, barrel-matured spirit, almost certainly as a result of the much higher surface area of woody material that the spirit was exposed to in the MixStix. However, when the two samples, ISS and Earth, were compared to each other, the lignin breakdown products - which are the major flavour active derivatives from oak wood - were present in higher concentrations in the control samples than in the space samples. This indicates that conditions of micro-gravity have inhibited their extraction from oak. On the other hand, ellagic acid and gallic acid, which are tannin breakdown products, both appeared to be higher in the ISS samples. The levels of these compounds are detailed in Table 2.

Analyte	Space Station samples	Earth 'control' samples
Gallic Acid	3.4	2.8
Ellagic Acid	57.6	41.5
Coniferaldehyde	11.3	14.9
Vanillin	39.1	69.1
Vanillic Acid	17.2	30.4
Sinapaldehyde	31.6	47.7
Syringaldehyde	150.9	259.8
Syringic Acid	59.2	94.4
Scopoletin	1.7	2.2
5-HMF	25.2	48.2

TABLE 2 Key Wood Extractives by HPLC (mg/l @ sample strength)

By drilling down further into these results, and subjecting them to some statistical analysis, the relative abundance of the main, easily extractable lignin breakdown products (vanillin, coniferaldehyde and sinapaldehyde) expressed as a percentage of the total wood extractives, is broadly similar in each sample. However, other extractives, such as the syringyl compounds, are very different in their total abundance, with much lower percentages in the ISS samples. This result means that while the overall extraction of wood-derived compounds is negatively impacted by micro-gravity, the impact is not equal; the compounds can be subdivided into 'readily extractable' and 'less readily extractable'.

While it is clear that micro-gravity is inhibiting the release of wood extractives, not all compounds are affected equally, as would be expected in normal conditions. This has resulted in an unusual ratio between the readily extractable compounds and the less readily extractable ones in the ISS samples. This offers the intriguing possibility of using the ratios of such compounds as a potential marker to identify 'unusual' maturation characteristics, and thus a means of identifying for example, spurious age claims in whiskies. This possibility may be further investigated by the Scotch Whisky Research Institute.

For studies such as this, where we are examining the impact of differing conditions on either model systems, or on the full scale in actual barrels, the key question is always 'What does this actually mean for the flavour?', so the organoleptic analysis was of great interest to us. The results from the key volatile congeners, wood extractives and phenols analysis did not lead us to believe that from an organoleptic perspective, there would be large differences between the ISS and control samples. However, when we finally nosed and tasted the samples in the sensory laboratory, the results were unexpected.

Triangle tests gave a high number of correct responses, with almost all participants picking out a difference between the ISS and control samples. When we examined the actual aroma and flavour profiles, the differences between the two were as remarkable as they were unexpected; all tasters described the Earth control samples as being of the general Ardbeg whisky style, while the ISS samples displayed a quite different set of flavours. These have been summarised in the detailed descriptions below.

Control Sample – a.b.v. 58.4%, reduced to 26% for tasting

Aroma – Very woody, hints of cedar wood, sweet smoke and aged balsamic vinegar. Hints of raisins, treacle toffee, vanilla and burnt oranges. Very reminiscent of an aged Ardbeg style.

Taste – Dry palate, woody/balsamic flavours, sweet smoke and clove oil. A distant fruitiness (prunes/dates), some charcoal and antiseptic notes. The aftertaste is long, lingering and typically Ardbeg, with flavours of gentle smoke, briar wood, tar and some sweet, creamy fudge.

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ISS Sample – a.b.v. 56.0%, reduced to 26% for tasting

Aroma – Intense and rounded, with notes of antiseptic smoke, rubber, smoked fish and a curious, perfumed note , like cassis or violet. Powerful woody notes, hints of graphite and some vanilla. This then leads into very earthy/soil notes, a savoury, beefy aroma, and then hints of rum & raisin flavoured ice cream.

Taste – A very focussed flavour profile, with smoked fruits (prunes, raisins, sugared plums and cherries), earthy peat smoke, peppermint, aniseed, cinnamon and smoked bacon or hickory-smoked ham. The aftertaste is pungent, intense and long, with hints of wood, antiseptic lozenges and rubbery smoke.

Conclusions & Next Steps

In summary, we have demonstrated that in micro-gravity terpenes behave differently in this environment compared to those on earth. This observation alone has implications for not just the malt whisky industry, but those of the food and drinks industry in general. Secondly the results have proven that in conditions of micro-gravity, the pattern of extraction of components of oak wood into spirit is different, with a degree of inhibition observed. This has given rise to the intriguing possibility that a database could be established detailing a 'normal' ratio of wood extractives covering a range of ages of spirit/whisky, which could then be used for comparison against potentially spurious samples.

The difference in simulated maturation conditions has resulted in a dramatically different flavour profile, which will give rise to the potential development of new flavours, and in particular new Ardbeg whisky expressions. The difference in flavour between the ISS and control samples is so marked, that further analysis will be carried out to elucidate the creation of the different flavours.

Finally, the experiment described in this paper is very much a first step, and further trials on the International Space Station will be considered in forthcoming discussions with NanoRacks. We, at Ardbeg, along with NanoRacks, would like to thank NASA and the Space Station Program for allowing this commercial project to move forward, one which seems to have implications for our industry and no doubt others.