*Chromatography Technical Note No AS149*

# Comparison between the MVM (Multi-Volatile Method), single DHS extraction and SPME (Solid Phase Micro Extraction) for extraction of volatiles in Whisky.

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## *Introduction*

An established method to analyze volatile compounds in whisky is using headspace solid phase microextraction (HS-SPME) with gas chromatography mass spectrometry (GC/MS). A potential alternative methodology is to use dynamic headspace (DHS) and employ the Multi Volatile Method (MVM) recently developed by GERSTEL and optimized at Anatune for Whisky [1].

The MVM method uses two Dynamic Headspace extractions ï extracting with two different traps from the same headspace vial. The first trap is the Shincarbon X/ Carbopack B+X which is designed to trap the higher volatility compounds. The second trap is a Tenax TA trap for the remaining analytes.

Within this application note, we compare the use of MVM and single DHS extraction (with Tenax) to the HS-SPME method for Whisky. Figure 1 below shows the instrument set up at Anatune.


***Figure 1: GC-MS instrument with DHS and TDU/CIS injector***

## *Instrumentation*

Agilent GC 7890A with MS detector QQQ (used in MS1 scan)
Dual head Gerstel MPS 2 Left hand fiber, right hand 1 syringe (gripper).
Agilent MSD Chemstation software (version B.07.01.1805)
Maestro software integrated (version 1.4.30.11./3.5)

## *Methods*

Each analysis was performed in duplicate.

***SPME procedure:***

Following incubation, the headspace was sampled using the mixed SPME fibre (DVB/CAR/PDMS) for 5 minutes. After this time, the fibre was removed and thermally desorbed in the GC inlet.

***SPME method***
Fibre: DVB/CAR/PDMS
GC-MS parameters:
Splitless (Temperature 250ºC)
Column: DB-WAX

***Single DHS method:***

Dynamic extraction of the sample headspace was performed using a single Tenax adsorbent trap. Following extraction the trap was thermally desorbed in the TDU, analytes trapped on the CIS and then transferred to the analytical column.

Volume sample: 50 µL of whisky in a 10 mL vial

DHS parameters:

***DHS trap: Tenax TA*** *(Incubation Temperature 80ºC)*
750 mL as trapping volume with a large dry phase

TDU ramped from 30ºC to 240ºC (splitless desorption)
CIS ramped from 10ºC to 240ºC (splitless)
Column: DB-WAX

***MVM procedure:***

The first DHS extraction was made with the Shincarbon X/Carbopack B+X adsorbent and then for the same sample a second extraction was performed with the Tenax adsorbent. After these two extractions, the Tenax adsorbent is firstly desorbed in the TDU followed by the Shincarbon X/Carbopack B+X trap (Figure 2). After the two traps are desorbed, the CIS is heated up and analytes are transferred to the analytical column.

Volume sampling: 100 µL of whisky in a 10 mL vial

DHS parameters:

*For the* ***Shincarbon X/Carbopack B+X*** *(Incubation Temperature 55ºC)*
10 mL as trapping volume with a small drying phase

*For the* ***Tenax TA*** *(Incubation at 80ºC)*
750 mL as trapping volume with a large dry phase

TDU ramped from 30ºC to 240ºC (splitless desorption)
CIS ramped from -50ºC to 240ºC (splitless)
Column: DB-WAX

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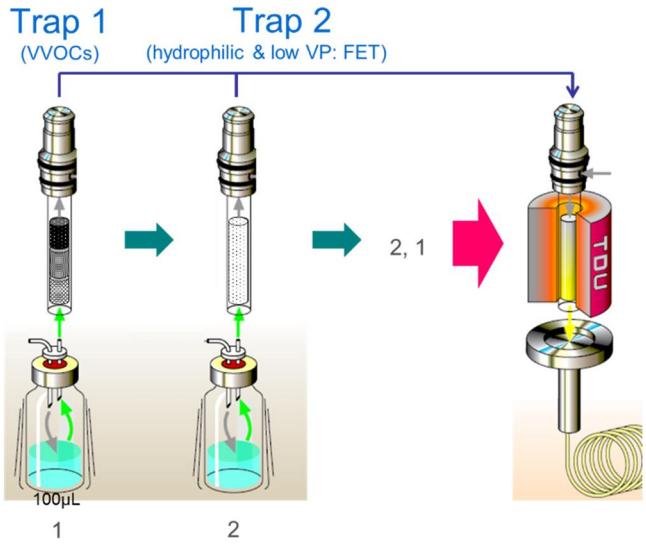


**Figure 2: MVM method using 2 traps**

## *Results*

Figure 3 shows a comparison of the chromatograms obtained with each method.

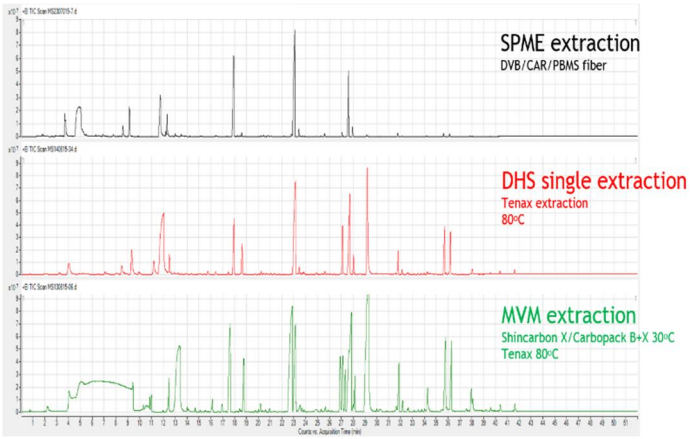


***Figure 3: Comparison between the three methods***

By looking at the chromatograms obtained, it appears that the use of the MVM method results in better extraction of the majority of analytes and some additional compounds were also observed. It is also clear that the ethanol is being extracted by the first trap, although in this case no peaks of interest were observed in this area of the chromatogram.

Figure 4 is a zoom of Figure 3 in order to observe the detail of the lower level compounds with the three techniques.

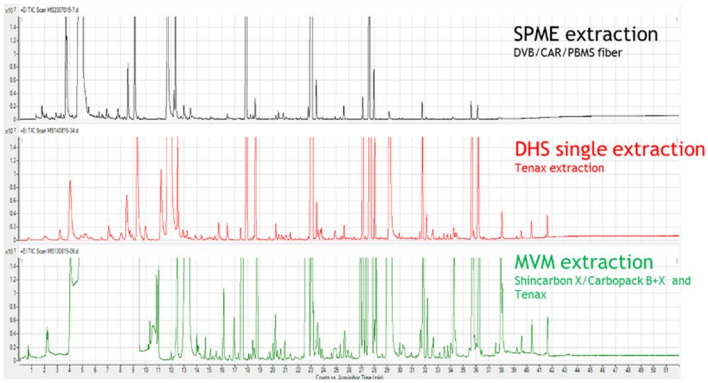


***Figure 4: Zoom on the chromatogram***

The zoom of the three chromatograms confirms that using MVM enables extraction of more compounds, including those present in low concentrations in the whisky. Those not observed using the other 2 methods include acetaldehyde, oaklactone, 1-Hexadecanol and 5-Hydroxymethylfurfural (identifications based on NIST mass spectral library search).

## References

[1]. AS148: The development of the MVM (Multi-Volatile Method) in the whisky matrix

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