Chromatography Technical Note No AS117

Analysis of water based products (Chilli Jam, Whiskey, Toothpaste and Chocolate using Entech 7150 Preconcentrator

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Introduction

The analysis of water based products proves an interesting challenge for many analytical chemists. Large amount of water can lead to poor peak shape for a number of analytes by GC and water can also deteriorate the GC column overtime. The Entech Preconcentrator can enrich volatile and semivolatile analytes whilst removing water through the split vent. Aqueous products such as Chilli Jam, Whiskey, Toothpaste and Chocolate can be analysed with no sample preparation and trace level analytes can be monitored and quantified.

Slight differences in the flavour profile of these any of these products can drastically change their taste. Therefore, it is important for the manufacturers to maintain the quality of their product for customer satisfaction.

Figure 1 shows a photo of products analysed by Entech Preconcentrator.

Figure 1 Water based products in Entech Large Volume Headspace jars (2g Toothpaste, 10 g Chocolate, 5 ml Whiskey, and 1 g of Chilli Jam).

Method

The products above were analysed on an Entech 7150 Preconcentrator using a 7500 autosampler. Each jar was heated to 50 °C for 15 minutes prior to analysis. A DB Wax 60 m x 0.32 mm x 0.5 micron film thickness was used for this work. A standard temperature program from 40 °C to 240 °C with a flow rate of 1.5 ml/min was used for this study. Analysis was carried out with duplicate samples and blank large volume headspace jars were included within the sequence to confirm the analytes presents in each sample. 50 cc of each sample was loaded.

The preconcentration technique utilizes "active SPME" which offers a more quantitative approach for Solid Phase Micro Extraction. A volume of headspace, typically between 10 cc to 1000 cc, can be pulled through a column containing a SPME coating of PDMS (polydimethylsiloxane). Figure 2 shows a schematic diagram of the 7150 preconcentrator with a 7500 autosampler.

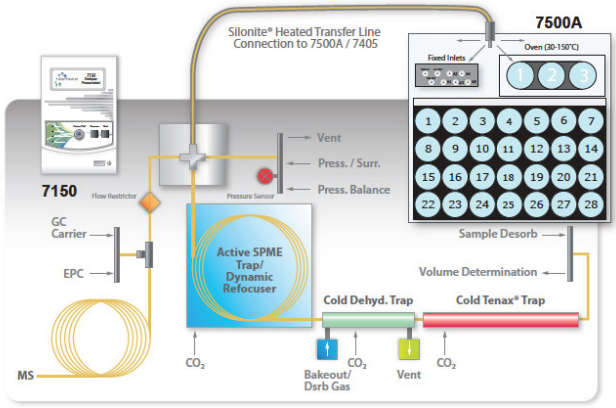

Figure 2 Schematic diagram of the 7150 with the 7500 autosampler.

With reference to Figure 2, an air sample (typically 100 cc) is pulled through the three different traps. Trap 1 is an Active SPME trap. The trap is 3 metres in length with an internal diameter of 0.53 mm which is internally coated with PDMS. Trap 2 is a cold dehydration trap which is used to remove residual water from the sample. Trap 3 is a Cold Tenax Trap which is used to trap volatile components typically with a carbon chain length of less than 10. Figures 3, 4, and 5 show schematic diagrams of the three different traps within the 7150 at different stages of the enrichment process to give a basic understanding of the system.

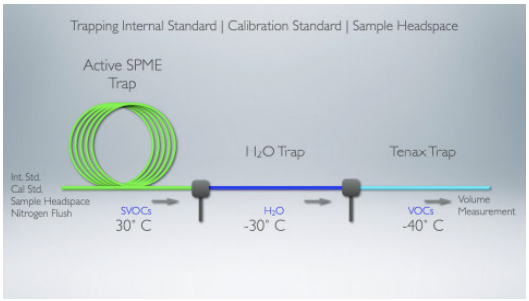

Figure 3 Schematic diagram of Entech traps (separating semivolatiles from volatiles)

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Initially, the SPME trap is set to 30 °C. This is hot enough to allow the volatile components and water through the SPME trap. However, this is cool enough to retain the semivolatile analytes. The cold dehydration trap is set at -30 °C and residual water from the sample forms ice crystals within this area. Volatile analytes flow through the cold dehydration trap to the Tenax trap where they are retained. Note that the tenax does not come into contact with semivolatiles. Therefore, there is not an issue with incomplete desorption of semivolatiles as these do reach this trap.

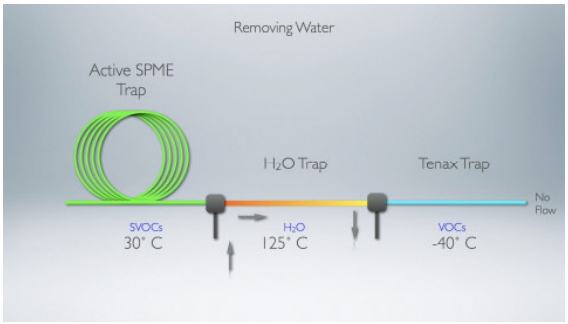

Figure 4 Schematic diagram of Entech traps (removal of water)

After isolating the flow of carrier gas (as shown in Figure 4), the water trap can then be heated and water vapour is then removed from the trap.

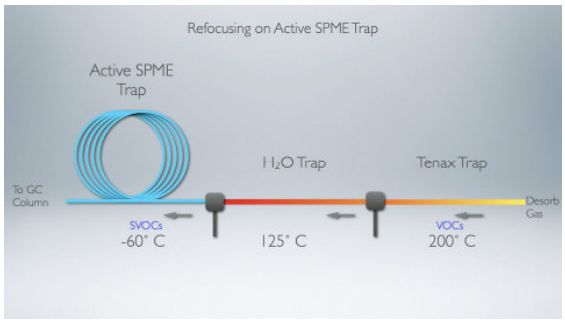

Figure 5 Schematic diagram of Entech traps (Refocusing onto Active SPME trap)

After the water has been removed from the trap, flow of carrier gas is then contolled to flow from the tenax trap back to the Active SMPE trap (as shown in Figure 3. Here the temperature of the Active SPME is reduced to -60 °C whereas the Tenax trap is raised 200 °C to desorb the volatile analytes. Both semivolatile and volatile analytes are then focused onto the Active SPME trap. In the final step, the SPME trap is then heated to desorb the analytes onto the GC.

Results

Figure 6 shows a TIC comparison of whiskey (duplicate samples) with a blank. Some of the analytes have been identified in the chromatogram.

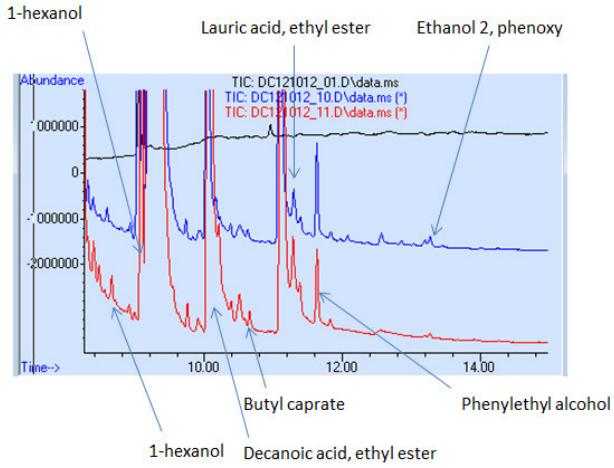

Figure 6 TIC comparison of whiskey with a blank.

Figure 7 shows a TIC comparison of Chilli Jam (duplicate samples) with a blank. Some of the analytes have been identified in the chromatogram.

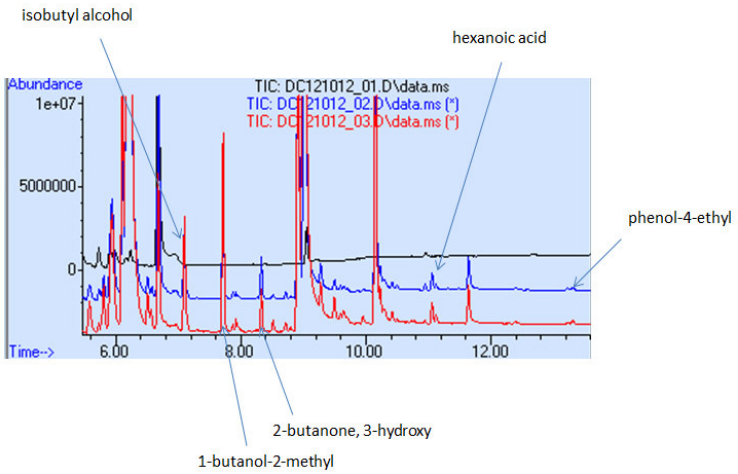

Figure 7 TIC comparison of Chili Jam with a blank.

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Figure 8 shows a TIC comparison of toothpaste (duplicate samples) with a blank. Some of the analytes have been identified in the chromatogram.

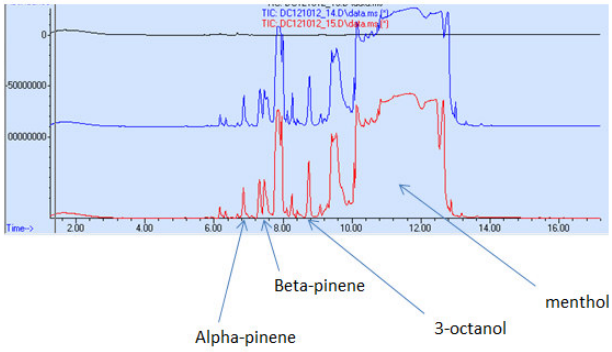

Figure 8 TIC comparison of toothpaste with blank

Figure 9 shows a TIC comparison of chocolate (duplicate samples) with a blank. Some of the analytes have been identified in the chromatogram.

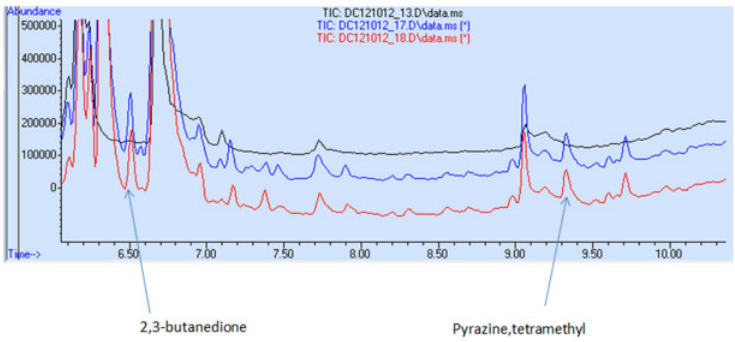

Figure 9 TIC comparison of chocolate with blank

Conclusion

Hexanoic acid is a known acid in chilli peppers. Pyrazine, tetramethyl is a known artifact in the chocolate manufacturing process. The 7150 preconcentrator offers a simple way to screen aqueous products to detect trace analytes present. Figure 10 shows a photograph of the 7150, with 7500 autosampler attached to an Agilent GC/MS.

Figure 10 photograph of 7150 preconcentrator, with 7500 autosampler attached to an Agilent GC/MS.

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