Chromatography Technical Note No AS113

Analysis of food using the Entech 7150 preconcentrator GC with large volume headspace

Dan Carrier, Anatune Ltd. Girton, Cambridgeshire, UK.

Introduction

The analysis of wet and moist samples can pose a real challenge to laboratory analysts. Water can cause a detrimental effect on chromatographic peak shape of analytes and it can also cause GC column damage. Using the Entech 7150, it is possible to remove the water within the cold dehydration trap and thus obtaining good peak shape for analytes of interest. The Entech 7150 also offers the advantage to analyse large samples. Up to 1000 cc of headspace can be enriched and chromatographed using the 7150 preconcentrator. Figure 1 shows a photograph of a portion of sirloin steak in a 500 ml Large Volume Headspace jar.

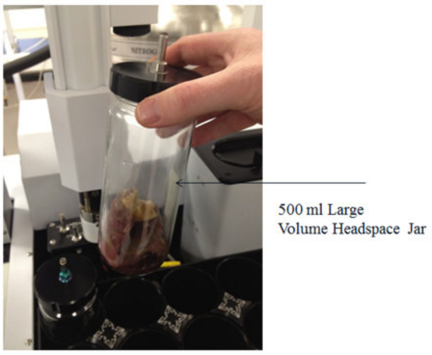

Figure 1 Sirloin Steak in Large Volume Headspace vial

This application note shows how the headspace of various foods can be sampled. Headspace from meat, cheese and crisps have been analysed on the 7150 preconcentrator. Meat and cheese can contain above 30 % water. Therefore, they can prove a real challenge to chromatograph. Although crisps contain a relatively low amount of water, work was required to wet the crisps.

Although, this application note is applicable to the food industry, the ability to sample large volumes of headspace and enrich moist samples offers advantages across many industry sectors, such as flavor and fragrance, pharmaceutical and environmental.

Method

Headspace analysis from meat packaging has been described in an earlier application note (AS112). For this new application note, the meat was aged over period of 7 days to investigate if the aging process could be monitored using the 7150 Preconcentrator. A portion of meat was placed in a 500 ml Jar and 100 cc of the headspace was analysed at time 0 and after 7 days.

For this crisp analysis, headspace from four different crisps packets was taken; ready salted, cheese and onion, salt and vinegar and prawn cocktail. To analyse the air within the packaging, an evacuated bottle (250 cc) is fastened to a female Micro valve connector which releases the vacuum from the bottle and thus pulls a portion of the air from the packaging into the bottle. Figure 2 shows how of picture of how this can be performed.

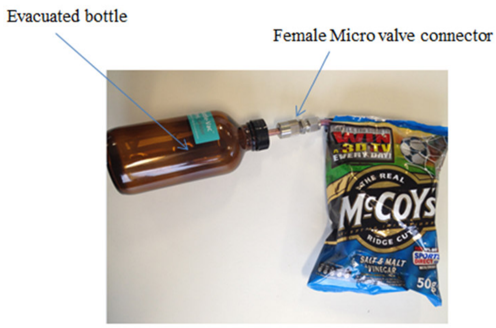

Figure 2 Sampling technique to take headspace of packaging

A 10 g portion of the cheese and onion crisps was crushed and added to a 250 ml bottle and 15 ml of water was added to wet the crisps.

For the cheese analysis, approximately 30 g of cheddar cheese was placed into four different 500 ml Large Volume Headspace Jars and 100 cc of the headspace was taken.

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.

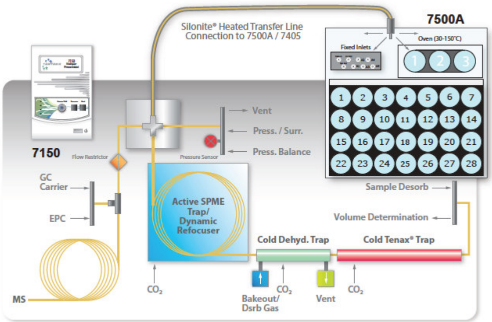

Figure 3 Schematic diagram of the 7150 with the 7500 autosampler.

With reference to Figure 3, an air sample (typically 100 cc) is pulled through the three different traps. Trap 1 is an Active SPME trap. The trap is 3

Anatune Ltd, Unit 4, Wellbrook Court, Girton Road, Cambridge, CB3 0NA, UK
Tel: +44 (0) 1223279210 Fax: +44 (0) 1223279253 Email: info@anatune.co.uk Internet: www.anatune.co.uk

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 4, 5, and 6 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.

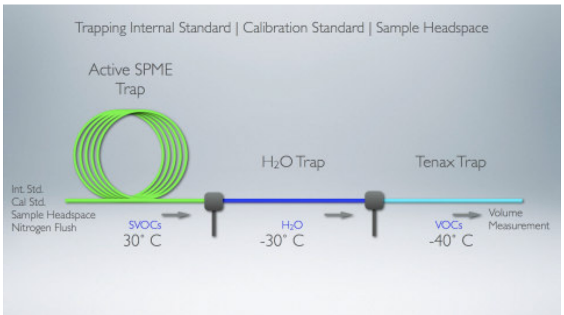

Figure 4 Schematic diagram of Entech traps (separating semivolatiles from volatiles)

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.

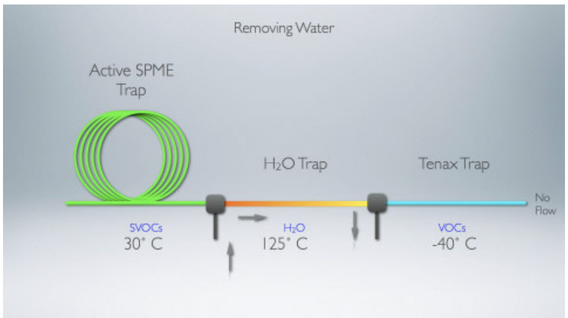

Figure 5 Schematic diagram of Entech traps (removal of water)

After isolating the flow of carrier gas (as shown in Figure 5), the water trap can then be heated and water vapour is then removed from the trap.

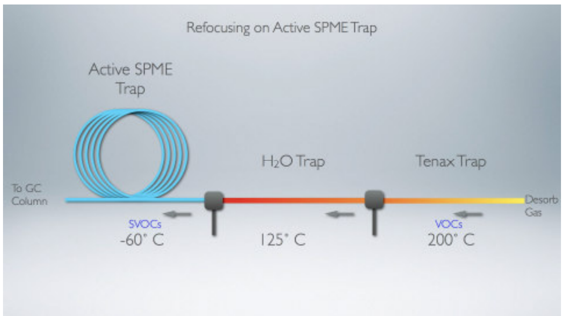

Figure 6 Schematic diagram of Entech traps (Refocusing onto Active SPME trap)

After the water has been removed from the trap, flow of carrier gas is then controlled to flow from the tenax trap back to the Active SMPE trap. 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 7 shows a TIC comparison of meat sampled at Time 0 (in blue) with meat aged for 7 days (in black). Some of the analytes have been identified in the samples.

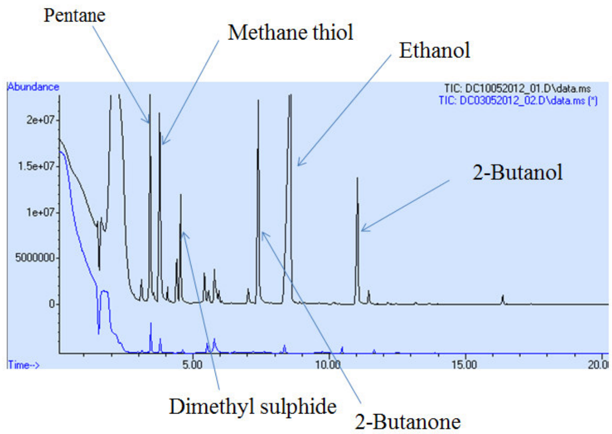

Figure 7 TIC comparison of meat time 0 and meat aged for 7 days.

Anatune Ltd, Unit 4, Wellbrook Court, Girton Road, Cambridge, CB3 0NA, UK
Tel: +44 (0) 1223279210 Fax: +44 (0) 1223279253 Email: info@anatune.co.uk Internet: www.anatune.co.uk

Figure 8 shows a comparison of cheese and onion crisps with a blank. Many of the analytes were similar between the different crisp types. However, beta-pinene appeared to be indicative of prawn cocktail crisps.

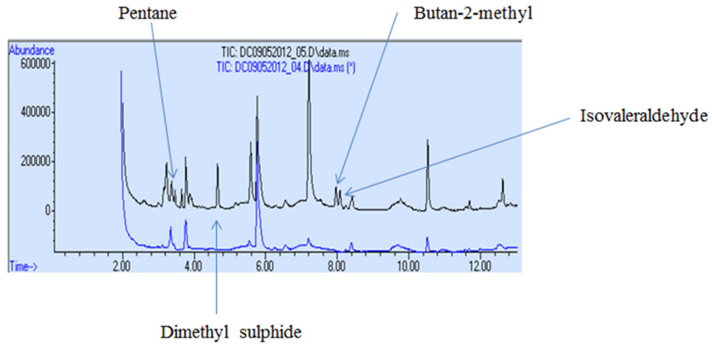

Figure 8 TIC comparison of cheese and onion crisps with a blank.

Figure 9 shows a TIC comparison between wet and dry crisps. It is thought that analytes behave differently in a wet matrix and thus different levels of the analytes can be observed (Wet crisps (in black), Dry crisps (in blue)).

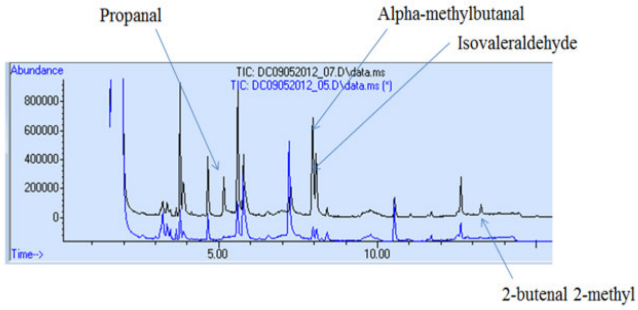

Figure 9 TIC comparison of wet and dry cheese and onion crisps.

Figure 10 shows a TIC comparison four different samples of cheddar cheese. This shows good reproducibility of the analytical method which demonstrates that the analytes have the potential to be quantified.

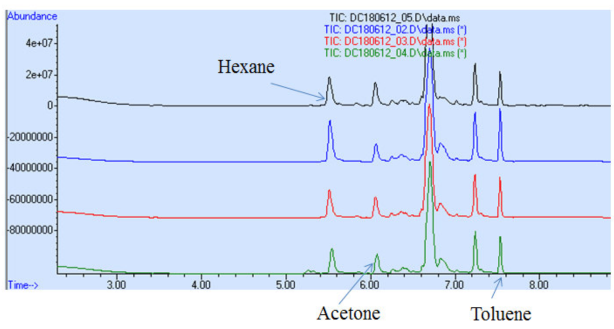

Figure 10 TIC comparison of cheddar cheese samples (100 cc of headspace)

Conclusion

The 7150 preconcentrator offers a simple way to monitor air within moist samples. Differences can be observed for aged meat and this could offer a good way to monitor the freshness of meat.

Good reproducibility for the four different samples of cheddar cheese samples demonstrates that the Entech can also be used for quantitation.

Figure 11 shows a photograph of the 7150, with 7500 autosampler attached to an Agilent GC/MS

Figure 11 photograph of 7150 preconcentrator, with 7500 autosampler attached to an Agilent GC/MS

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