Chromatography Technical Note No AS112

Trace analysis of meat packaging headspace using an Entech 7150 preconcentrator with large volume headspace

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

Introduction

Food protection from potentially hazardous residues has increasingly become a major topic of public interest. Food regulation authorities in the European Community and Switzerland have recently produced proposals for restrictions of these unwanted residues in food. Thermal desorption techniques using carbon absorbents can be used to monitor for trace level analytes in air. However, these techniques can sometimes fail to desorb certain analytes due to their strong interactions with the adsorption phase. Also, using thermal desorption for the analysis of thermally labile and reactive compounds can prove to be challenging. By using Solid Phase Micro Extraction (SPME), recoveries of such analytes can be improved as this is a gentler sample enrichment technique.

Method

To analyse the headspace 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 headspace from the packaging into the bottle. Figure 1 shows how of picture of how this is performed.

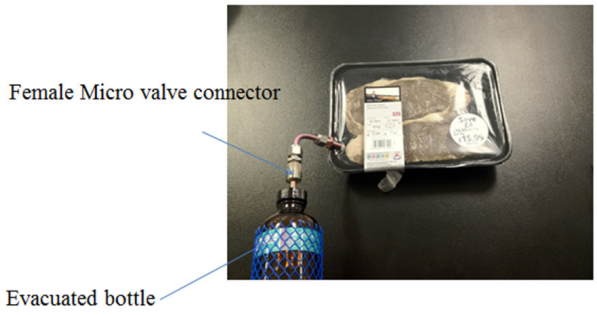

Figure 1 sampling technique to take headspace of packaging

The preconcentration technique utilizes "Active SPME" which offers a more quantitative approach than traditional 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.

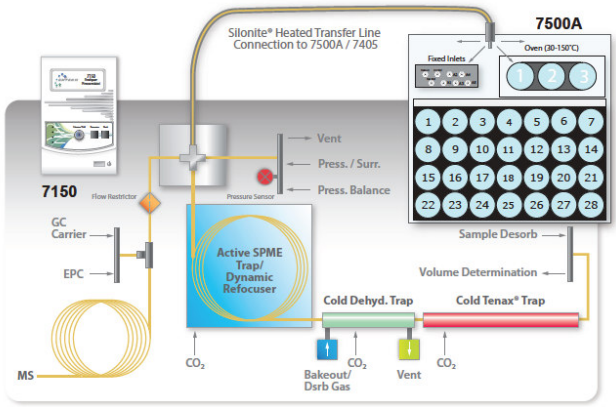

Figure 2 Schematic diagram of the 7150 with the 7500 autosampler.

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.

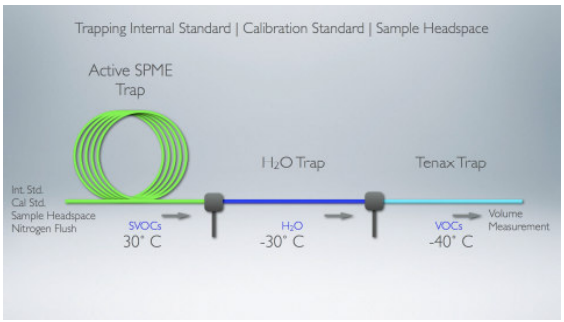

Figure 3 Schematic diagram of Entech traps (separating Semivolatiles from volatiles)

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 not reach this trap.

Anatune Ltd, Unit 4, Wellbrook Court, Girton Road, Cambridge, CB3 0NA, UK
Tel: +44 (0) 1223 279210 Fax: +44 (0) 1223 279253 Email: info@anatune.co.uk Internet: www.anatune.co.uk

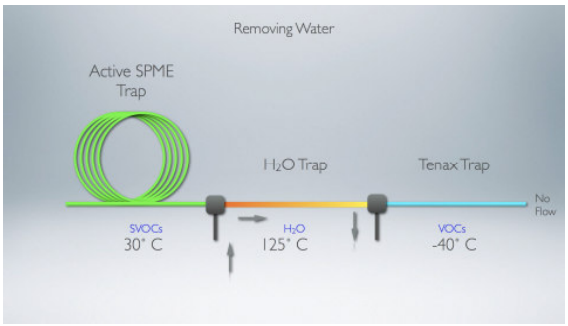

Figure 4 Schematic diagram of Entech traps (removal of water)

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.

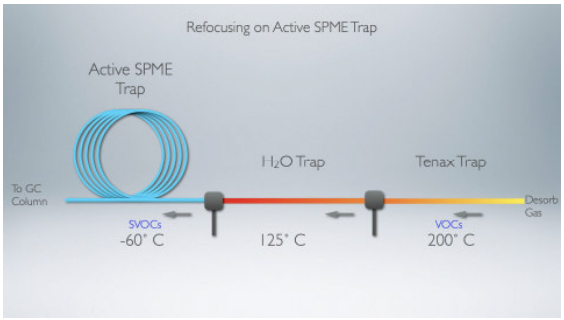

Figure 5 Schematic diagram of Entech traps (Refocusing onto Active SPME trap)

After the water has been removed from the trap, flow of carrier gas is then controlled to flow from the tenax trap back to the Active SMPE trap (as shown in Figure 5. 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.

100 cc of packaging air in meat was analysed on the 7150 preconcentrator and results were compared to a 100cc of air from a clean bottle.

Results

Figure 6 shows a TIC comparison of the air within the packaging of the meat with a blank. Some of the analytes have been identified in the sample taken.

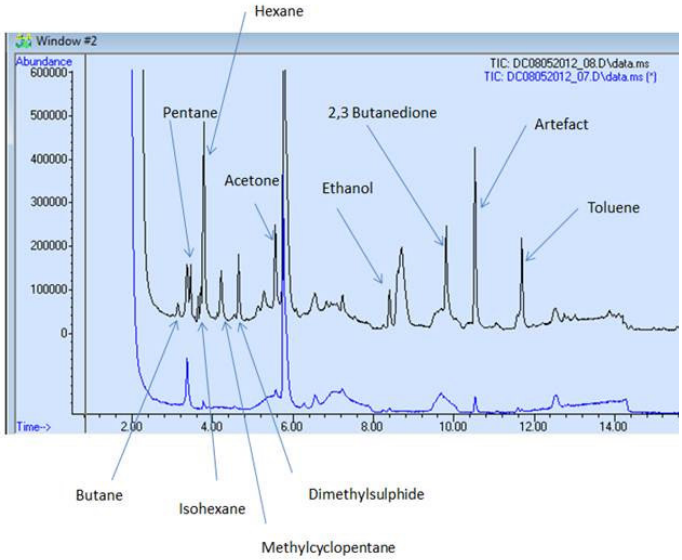

Figure 6 TIC comparison of the air within the packaging of the meat with a blank.

Conclusion

The 7150 preconcentrator offers a simple way to monitor air within packaging. The air taken was from a humid environment which can prove challenging with conventional techniques such as static headspace analysis. All of these analytes are at trace level within the air and by enriching a large volume (100 cc), good MS responses can be achieved. Figure 7 shows a photograph of the 7150, with 7500 autosampler attached to an Agilent GC/MS

Figure 7 photograph of 7150 preconcentrator, with 7500 autosampler attached to an Agilent GC/MS

Anatune Ltd, Unit 4, Wellbrook Court, Girton Road, Cambridge, CB3 0NA, UK
Tel: +44 (0) 1223 279210 Fax: +44 (0) 1223 279253 Email: info@anatune.co.uk Internet: www.anatune.co.uk