

Aroma profiling of pet food using high-capacity sorptive extraction and TD-GC×GC-TOF MS

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Introduction

Pet food manufacturers place great importance on the aroma of their products, as off-odours may be unappealing to both the pets and their owners. Confident identification of the volatile organic compounds (VOCs) from pet food can help these companies better understand the factors governing the release of pleasant and unpleasant aromas. VOC aroma profiles are typically analysed by solid-phase micro-extraction (SPME), which although a fast and simple technique, can be limited in terms of sample capacity, reproducibility and sensitivity.

An alternative to SPME is high-capacity probe-based sorptive extraction, which results in higher sample loadings because of the large volume of PDMS phase. Typically, a SPME fiber has a sorbent volume of just 0.5 µL, while the sorptive extraction probes used in this study contain 65 µL of sorbent. When used in conjunction with secondary refocusing by thermal desorption (TD), the result is greater sensitivity across a wide analyte range.

Further analytical benefits can be achieved for analyte separation and detection, by using comprehensive two-dimensional gas chromatography coupled with time-of-flight mass spectrometry (GC×GC-TOF MS). The enhanced separation capacity of this approach is ideal for handling the complex aroma samples of pet food, because it allows the entire composition to be screened in a single analysis, with confident identification of compounds that would ordinarily co-elute.

Here we demonstrate the value of high-capacity sorptive extraction with TD-GC×GC-TOF MS to investigate the volatile compounds present in the headspace of pet food, and discuss how these compounds might influence their aroma profiles.

Experimental

Sampling: Headspace sorptive extraction was performed on meat-based cat food (6 g) using an inert HiSorb™ PDMS probe (Markes International) for 15 min (after 20 min equilibration) at 60°C with agitation at 500 rpm.

TD: Instrument: TD100-xr™ (Markes International). Focusing trap: 'General-purpose'. HiSorb probes were inserted into empty inert-coated stainless-steel TD tubes.

GC×GC: Flow modulator: INSIGHT™ (SepSolve Analytical).

TOF MS: Instrument: BenchTOF-Select™

Software: ChromSpace® GC×GC software for instrument control and data processing.



Figure 1: Headspace sampling of pet food using HiSorb probes.

Results and discussion

Aroma profiling of pet food

GC×GC-TOF MS surface charts for the two brands of cat food are displayed in Figure 2, and show excellent separation across the analyte profile.

The enhanced separation achieved by INSIGHT GC×GC enables peaks that would co-elute in conventional GC to be separated in the second dimension. This is particularly valuable for compounds with low odour thresholds, which despite being detectable by GC-olfactometry, may be overlooked or misidentified due to co-elutions with high-loading peaks.

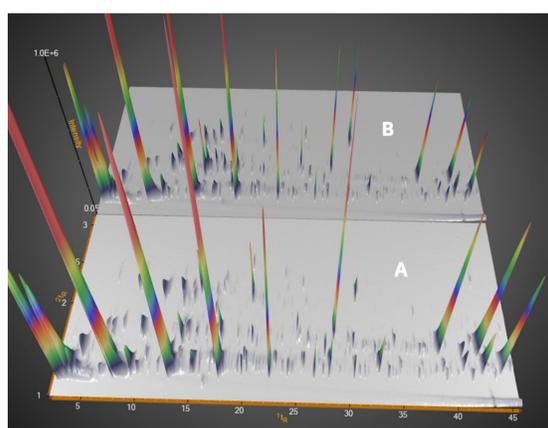


Figure 2: TD-GC×GC-TOF MS surface plots of two brands of cat food using headspace sampling by HiSorb probes.

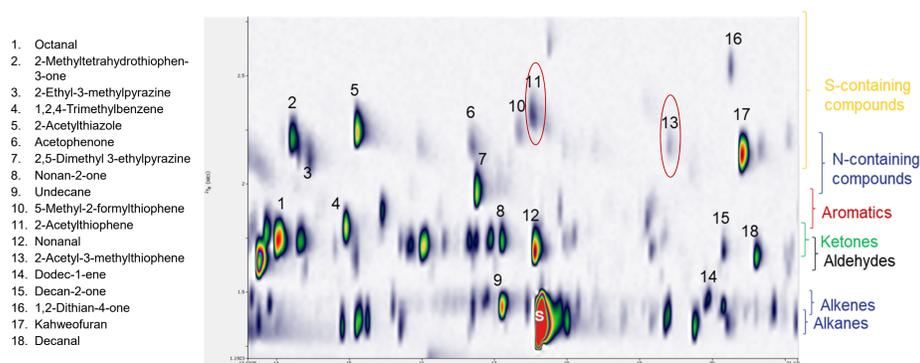


Figure 3: Enhanced separation of a range of compound classes in the headspace aroma profile of cat food (sample B) by GC×GC-TOF MS. S = Siloxane.

For example, in Figure 3 we can see that the small peak for 2-acetylthiophene (#11, highlighted in yellow) would be obscured both by nonanal (#12) and a large siloxane interference. 2-Acetylthiophene has an undesirable 'sulfurous' aroma and an odour threshold of just 0.08 ppb, making confident identification at trace levels important.

As well as being separated from these components with GC×GC, 2-acetylthiophene is confidently identified by comparison against the NIST 17 database (Figure 4), as is the related odorous compound 2-acetyl-3-methylthiophene (#13, with a 'phenolic' aroma), using the reference-quality spectra of BenchTOF.

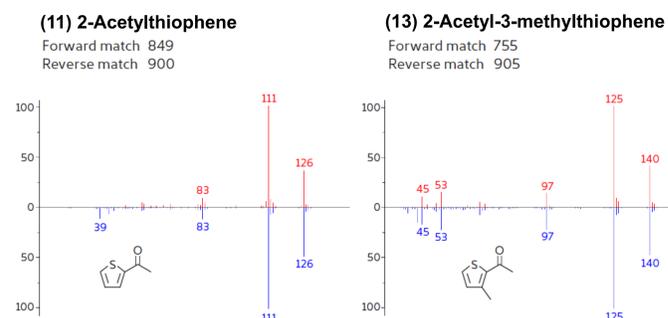


Figure 4: BenchTOF spectra (top, red) and NIST 17 spectra (bottom, blue) for two sulfur heterocycles identified in Figure 3.

Figure 5 provides an overview of the key compounds identified in the form of aroma wheels. As expected for a meat-based pet food, a number of compounds were found to contribute 'nutty/roasted' aromas, as well as 'meaty' and 'fruity, green'. However, a number of compounds perceived to be off-odours or taints were also identified. One such compound, dimethyl disulphide ('sulfurous, vegetable, onion') was only detected in sample A.

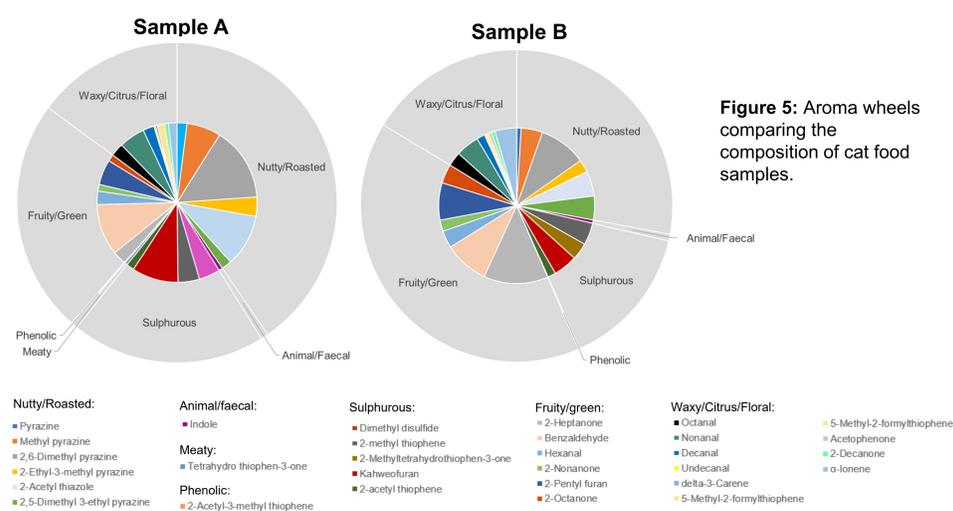


Figure 5: Aroma wheels comparing the composition of cat food samples.

Automated sampling and concentration



The HiSorb sampling described in this poster can now be fully automated by the new Centri® platform from Markes International. Centri harnesses market-leading trapping and re-collection technology to extend the sensitivity and applicability of sorptive extraction, as well as conventional headspace and SPME analyses, while also providing method-compliant thermal desorption.

To find out more about Centri, visit www.sepsolve.com/sample-automation-and-concentration/, or scan the QR code.



Conclusions

This poster has demonstrated that:

- ▶ Sorptive extraction with GC×GC-TOF MS is a powerful approach to characterising complex aroma profiles.
- ▶ Enhanced separation of GC×GC provides clean mass spectra for confident identification of odour taints.
- ▶ Probes are robust, easily rinsed free of matrix for hassle-free immersive sampling, and can typically be reused at least 50 times without any reduction in performance.
- ▶ Secondary re-focusing by TD offers excellent sensitivity, as well as the ability to re-collect a portion of the sample.
- ▶ HiSorb sorptive extraction can now be fully automated by the Centri platform.