



Application Note 120

Flavour profiling of milk using HiSorb sorptive extraction and TD-GC-MS

This study shows that the wide range of volatile and semi-volatile organic compounds (VOCs and SVOCs) giving rise to the flavour of milk can be identified by high-capacity sorptive extraction using PDMS probes, with analysis by thermal desorption-gas chromatography-mass spectrometry (TD-GC-MS).

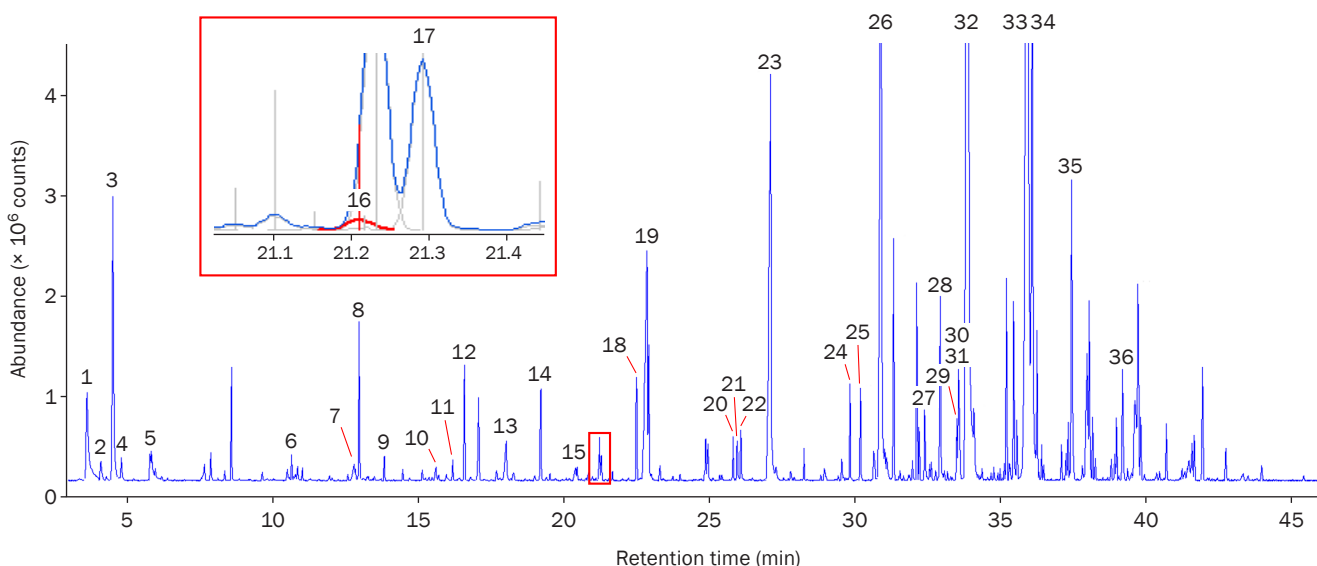
Results and discussion

VOCs and SVOCs in semi-skimmed milk were sampled by immersing a HiSorb™ PDMS probe in the sample, followed by agitation for 1 hour at 37 °C using the HiSorb Agitator, probe desorption, and TD-GC-MS analysis.

Figure 1 shows the complex profile obtained and a selection of the compounds identified. The analyte range achievable with this analytical system is evident from the variety of compounds found, which ranged from C₃ to C₁₉ and included

alkanes, aldehydes, alcohols, acids, esters, ketones and lactones, as well as other compounds with potential to affect the flavour profile.

Lactones are particularly important flavour compounds in a variety of foods, and in this example four δ-lactones were identified. δ-Octalactone (#16) although present at trace levels and co-eluting with a much more abundant component, was confidently identified by employing spectral deconvolution (see inset).



1 Sulfur dioxide	8 Phenol	15 Nonanoic acid	21 δ-Decalactone	28 2,3-Dihydroxypropyl decanoate	33 Oleic acid
2 Acetone	9 Octanal	16 δ-Octalactone	22 2,4-Di- <i>tert</i> -butylphenol	29 δ-Tetradecalactone	34 Octadecanoic acid
3 Acetic acid	10 Acetophenone	17 Undecan-2-one	23 Dodecanoic acid	30 Palmitoleic acid	35 A β-monoglyceride of myristic acid
4 n-Hexane	11 Nonan-2-one	18 2,2'-Isopropylidene bis(tetrahydrofuran)	24 Pentadecan-2-one	31 <i>cis</i> -Hexadec-11-enoic acid	36 2-Palmitoyl glycerol
5 Hydroxyacetone	12 Nonanal	19 n-Decanoic acid	25 δ-Dodecalactone	32 Hexadecanoic acid	
6 Heptan-2-one	13 Octanoic acid	20 Tridecan-2-one	26 Tetradecanoic acid		
7 Hexanoic acid	14 Decanal		27 Pentadecanoic acid		

Figure 1: Flavour profile of semi-skimmed milk, obtained by HiSorb sorptive extraction and TD-GC-MS analysis.

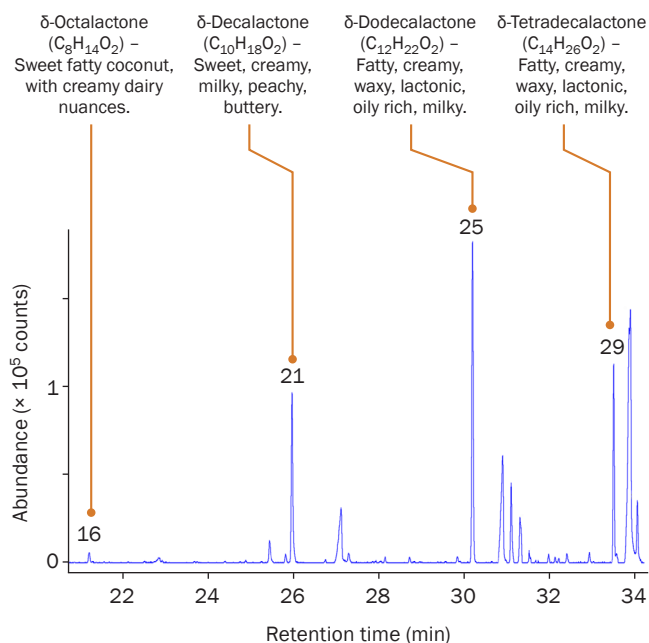


Figure 2: EIC profile (m/z 99) giving an indication of the relative abundances of several lactones present in the semi-skimmed milk.

To assess the relative concentrations of these lactones, Figure 2 shows an extracted-ion chromatogram (EIC) at m/z 99, which is a principal fragment ion for these compounds. δ -Dodecalactone (#25) predominates within this group, and therefore could be judged to be a contributor to the flavour of this milk.

In conclusion, this study has shown the ability of HiSorb sorptive extraction, combined with analysis by automated TD, to aid the rapid flavour profiling of milk. The high-capacity PDMS phase results in higher sample loadings than SPME methods, and (combined with Markes' TD pre-concentration technology) offers greater sensitivity across a wider analyte range, in a single run. A further benefit is provided by the unique capability of Markes' instruments to re-collect all split flows, allowing repeat analysis for sample security, method optimisation and characterisation by alternative detection methods.

Experimental

Sample:

Semi-skimmed milk was purchased from a local supermarket and diluted 1:1 with 25% saline solution.

Sorptive extraction:

System: HiSorb probe and Agitator (Markes International)
 Sample volume: 10 mL
 Probe: Part no. H1-AXAAC-5 (for 20 mL vials)
 Immersion: 1 hour at 37°C
 Agitator speed: 300 rpm

TD:

Instrument: TD100-xr™ (Markes International)
 TD tube: Stainless steel (part no. C0-AXXX-0000)
 Tube desorb: 110°C (0.5 min) then 200°C (10 min), with 10 mL/min split flow
 Cold trap: General-purpose hydrophobic (part no. U-T2GPH-2S)
 Trap low: 30°C
 Trap desorb: 280°C (5 min), splitless

GC:

Column: DB-5™, 60 m \times 0.25 mm \times 0.25 μ m
 Oven: 35°C (0.5 min), then 6.5°C/min to 230°C (0 min), then 15°C/min to 320°C (5 min)
 Column flow: Helium, 2 mL/min

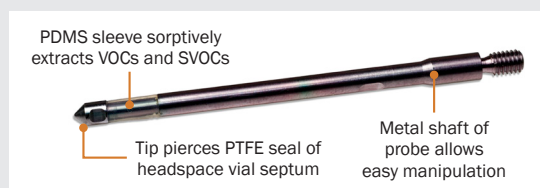
Quadrupole MS:

Scan mode: m/z 35–250
 Source: 230°C
 Transfer line: 280°C

Background to HiSorb

Markes International's HiSorb system allows high-capacity sorptive extraction from liquids and solids.

Samples are placed inside standard 20 mL or 10 mL vials, sealed with a crimped HiSorb septum cap, and a metal-core PDMS **HiSorb probe** inserted into the vial for either immersive or headspace sampling. The vial and probe are agitated and heated using the **HiSorb Agitator**, and after this the probe is washed, dried, and inserted into a conventional TD tube for desorption and automated TD-GC-MS analysis.



HiSorb has been developed with Welsh Government 'SMART Cymru' funding. HiSorb™ and TD100-xr™ are trademarks of Markes International. DB-5™ is a trademark of Agilent Corporation.

Applications were performed under the stated analytical conditions. Operation under different conditions, or with incompatible sample matrices, may impact the performance shown.