BEYOND MEASURE

SCION INSTRUMENTS

Analysis of Flavour Compounds in Milk Flavourings by SPME-GC-MS

Application Note

AN0033

INTRODUCTION

Dairy based milk powders offer a healthy alternative to fresh milk whilst also being readily available to incorporate into milk flavoured products during manufacturing. Whilst consumers expect highly soluble and great tasting products, manufacturers need reliable high quality instrumentation for determining the right chemical composition of their products.

Gas chromatography is the most commonly used chromatography technique for analysing food products, especially milk powders, for the identification of aroma compounds. The identification of the aroma compounds is vital as they constitute the taste and smell of all food products.

Solid Phase Micro Extraction (SPME) is a solid phase extraction technique that involves the use of a fiber coated in a polymer or sorbent extracting phase. The fiber is exposed to a sample where sample analytes are absorbed onto the fiber coating. The fiber coating should be chosen to suit the type of analyte in the sample. During injection into the GC inlet, desorption occurs and the analytes are introduced to the analytical system. The quantity of analyte extracted by the fiber is proportional to its concentration in the sample as long as equilibrium is reached.

EXPERIMENTAL

A SCION 456 GC and SCION single quad (SQ) mass spectrometer (MS) equipped with 8400 autosampler, operated in SPME mode, was used to analyse both a milk powder and liquid sample containing milk flavours. The liquid sample was prepared by dissolving milk powder into propylene glycol. The milk powder is the basis to most food products containing milky flavours. Table 1 details the analytical conditions of the GC-MS with SPME autosampler.

Table 1. Analytical conditions of the GC-MS with SPME

GC Conditions	
SPME	Adsorb 25mins, Desorb 5 minutes (DVB/CAR/PDMS Fiber)
PTV Injector	250°C, 1:5 Split
Carrier Gas	Helium 1.5mL/min
Oven	60°C (hold 1 min), 10°C/min to 100°C (hold 2 min), 40°C/min to 250°C (hold 10 mins)
Column	30m x 0.25mm x 0.5µm (SCION-WAXMS)
Full Scan	42m/z to 350m/z
Transfer Line	250°C
Ion Source	200°C

150mg of the liquid sample and 150mg of the milk powder were independently weight into 2mL vials along with 150mg of high grade water. Samples were heated at 30°C for 30 minutes prior to being exposed to the SPME fiber.

RESULTS

Mass Spec Work Station is the software used to control the SCION GC-MS and data processing. NIST is an inbuilt mass spectral library which was used to identify the flavour compounds in both the liquid and powder samples. Figures 1 and 2 detail the chromatograms obtained when both liquid and powder sample was analysed by SPME-GC-MS. Tables 2 and 3 detail the flavour compounds identified in the liquid and powder samples by NIST.

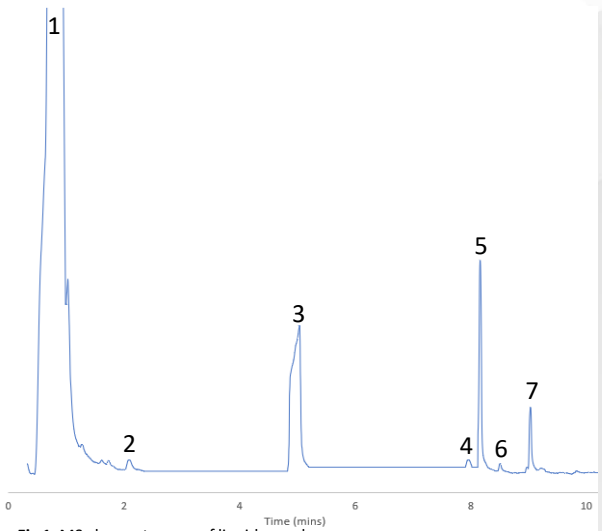

Fig 1. MS chromatogram of liquid sample

Table 2. Flavour compounds of liquid sample.

Retention Time (min)	Compound	CAS Number
0.716	Propylene Glycol	57-55-6
2.085	B-terpinyl Acetate	10198-23-9
4.913	Caprylic Acid	124-07-2
7.407	Ethyl Decanoate	110-38-3
7.976	γ-heptylbutyrolactone	104-67-6
8.146	δ-Decalactone	705-86-2
9.002	δ-Dodecalactone	713-95-1

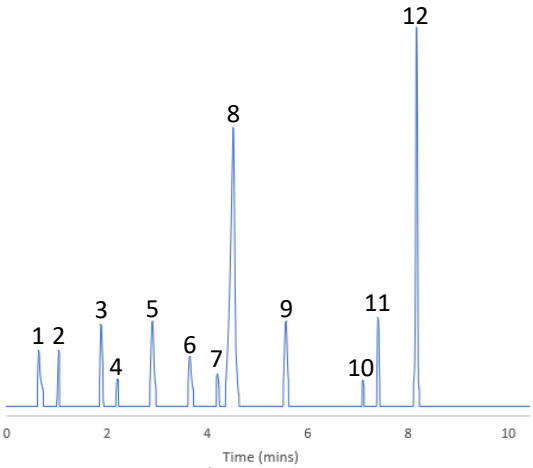

Fig 2. MS chromatogram of powder sample

Table 3. Flavour compounds of powder sample.

Retention Time (min)	Compound	CAS Number
0.608	Ethyl Butyrate	105-54-4
1.045	N-Amyl Methyl Ketone	110-43-0
1.898	Hexanoic Acid, ethyl ester	123-66-0
2.215	Decanal	112-31-2
2.892	2-Nonanone	821-55-6
3.639	Benzeneethanamine	55429-85-1
4.189	Octanoic Acid	106-32-1
4.520	Ethyl Maltol	4940-11-8
5.564	Methyl Nonyl Ketone	112-12-9
7.090	Triacetin	102-76-1
7.392	Ethyl Decanoate	110-38-3
8.158	Butylated Hydroxytoluene	128-37-0

Repeatability testing of both liquid and powder sample was performed using six replicates of each sample using SPME-GC-MS. Tables 4 and 5 detail the repeatability of both samples.

Table 4. Liquid sample repeatability (n=6)

Compound	RSD %
Propylene Glycol	2.1
B-terpinyl Acetate	3.2
Caprylic Acid	3.2
Ethyl Decanoate	4.1
γ-heptylbutyrolactone	3.8
δ-Decalactone	4.2
δ-Dodecalactone	3.7

Table 5. Powder sample repeatability (n=6).

Compound	RSD %
Ethyl Butyrate	4.2
N-Amyl Methyl Ketone	3.4
Hexanoic Acid, ethyl ester	3.6
Decanal	4.7
2-Nonanone	3.3
Benzeneethanamine	4.6
Octanoic Acid	3.2
Ethyl Maltol	4.7
Methyl Nonyl Ketone	3.8
Triacetin	3.9
Ethyl Decanoate	4.1
Butylated Hydroxytoluene	3.8

CONLUSION

The SCION 456 GC with MS and automated SPME was used to identify flavour compounds commonly found in milk based products. Both liquid and powered samples containing milk flavourings were analysed with peak identification confirmed via NIST spectra library comparisons. Excellent repeatability was achieved, highlighting the robustness of the SCION SPME autosampler and GC-MS system.

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