

Application Note No. 085

The Focus Robotic Sample Processor as a Tool for the Multiple Analysis of Samples using Complementary Techniques

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- ***Multiple techniques using one instrument***
- ***Simple to change between techniques***
- ***Fully automated analyses***

Instrumentation

- Focus Robotic Sample Processor
- ATAS Optic 2-200 programmable injector
- Agilent 5890 GC with 5971 MSD

Techniques and samples

- Headspace analysis – using heated syringe (2.5 mL @ 75°C), pre-incubation (15 mins @ 75°C) and agitation
- SPME analysis – using polyacrylate SPME fibre, pre-incubation (15 mins @ 75°C) with agitation, with analysis of the headspace (extraction 5 mins, desorption 3 mins)
- DMI analysis – using microvial (6mm) with thermal desorption (at 250°C)

Samples

- An ointment containing high levels of volatiles
- Soap powder granules
- Bar of soap grated

Principles

- Place ~1g sample in a 20 mL headspace vial, cap and place in Focus tray
- Change syringe kit to a heated Headspace syringe and start headspace method
- Place ~1g sample in a 20 mL headspace vial, place in Focus tray
- Change syringe kit to SPME kit and start SPME method (headspace)
- Place ~1 mg sample in a DMI microvial, place in DTD liner, cap and place in Focus DTD tray
- Swap injector head for DTD head and start DMI method

Please note, the methods for the results shown here were not optimised, and consequently different oven initial times and temperatures were used for the different techniques resulting in retention time shifts for the peaks, the remainder of the oven method was the same.

Chromatogram

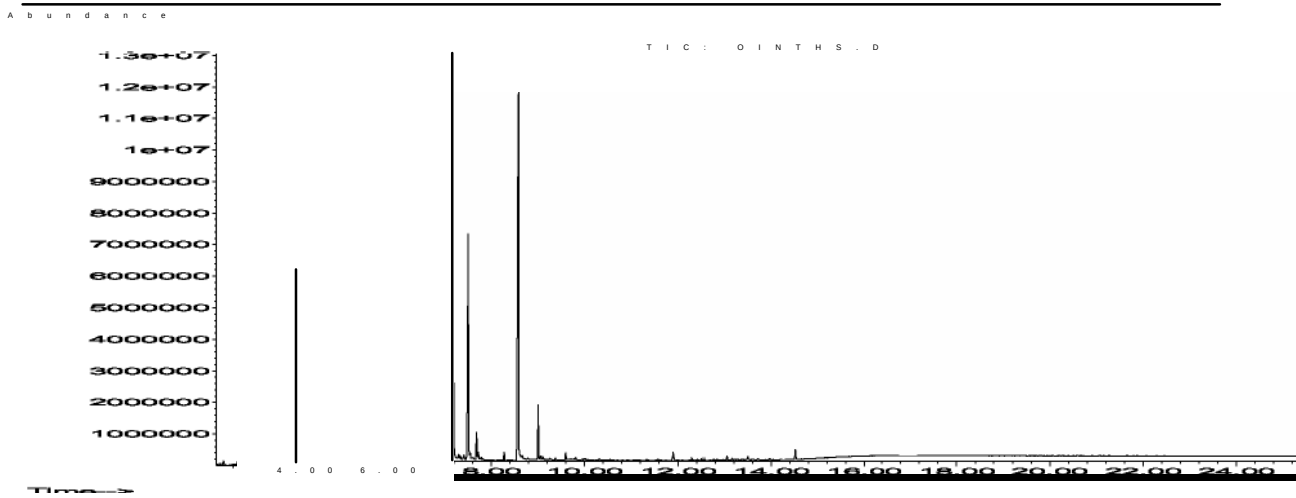


Figure 1: Headspace analysis of a 1g sample of an ointment

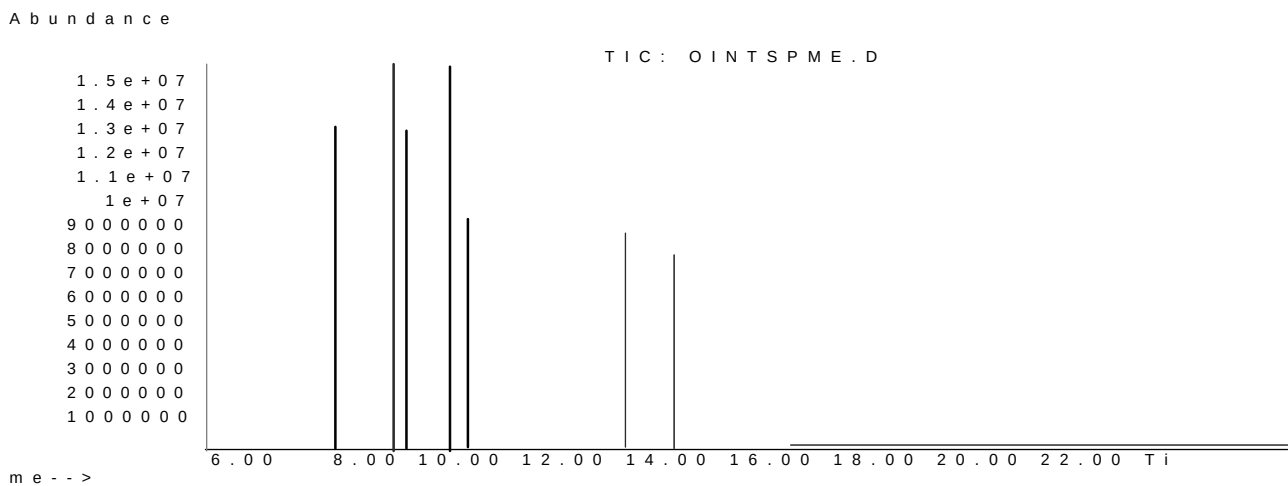
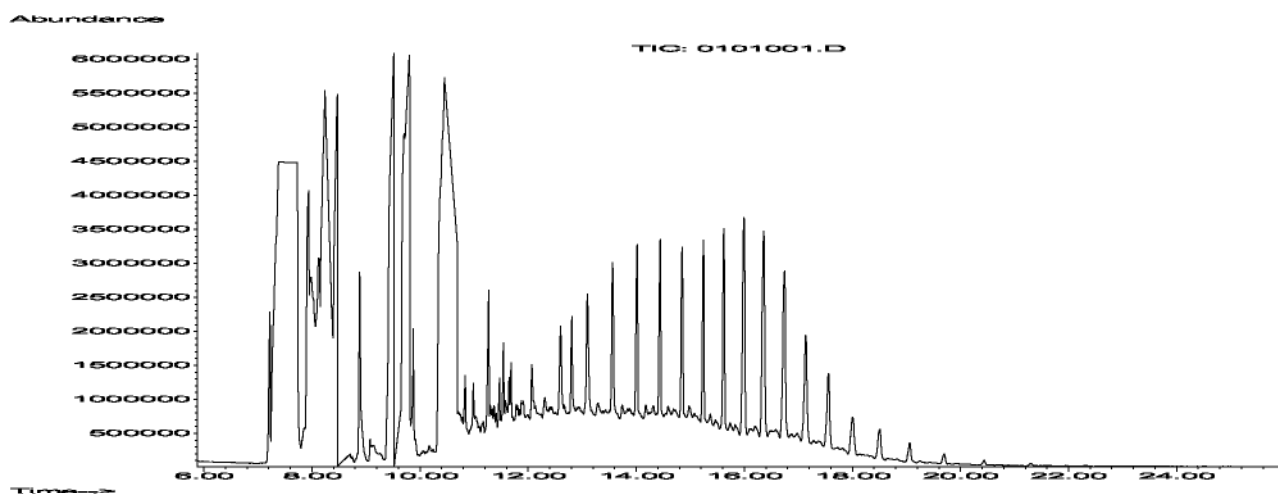


Figure 2: SPME analysis of the headspace from a 1g sample of an ointment



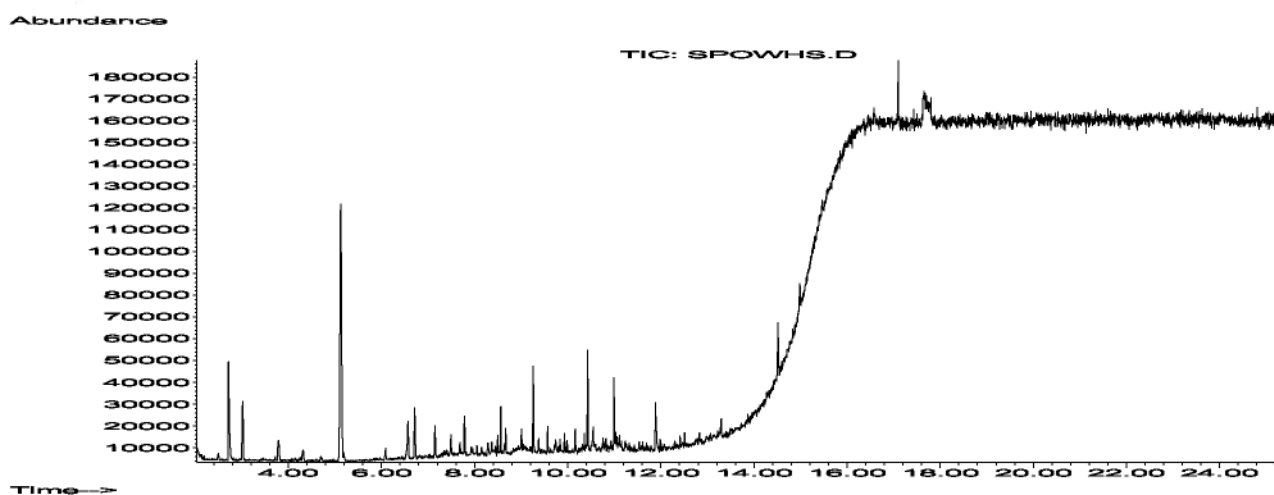


Figure 4: Headspace analysis of a 1g sample of soap powder

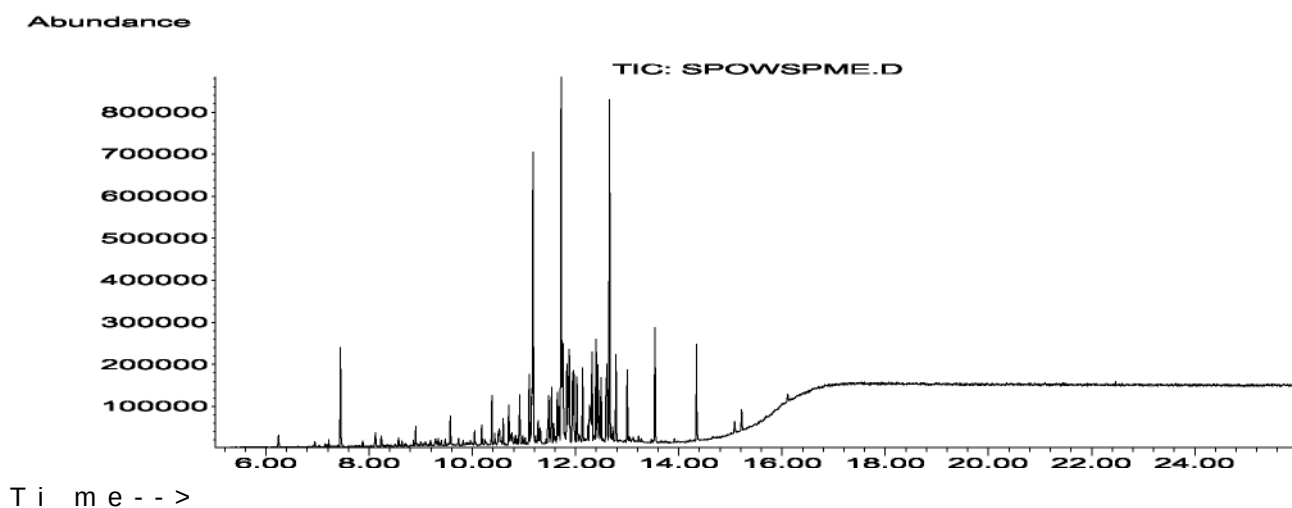


Figure 5: SPME analysis of the headspace from a 1g sample of soap powder

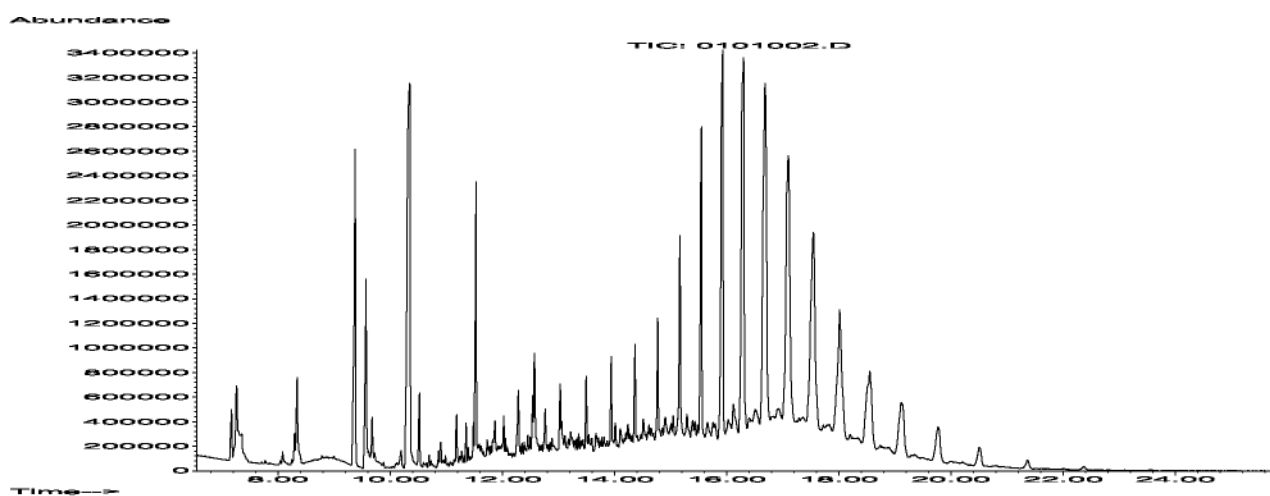


Figure 6: DMI analysis at 250°C of a 1mg sample of soap powder

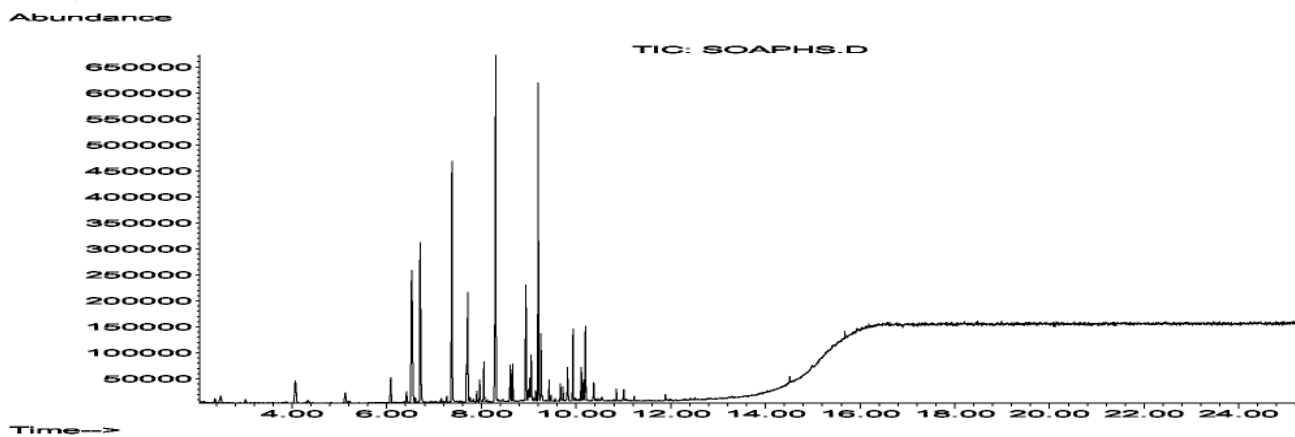


Figure 7: Headspace analysis of a 1g sample of soap

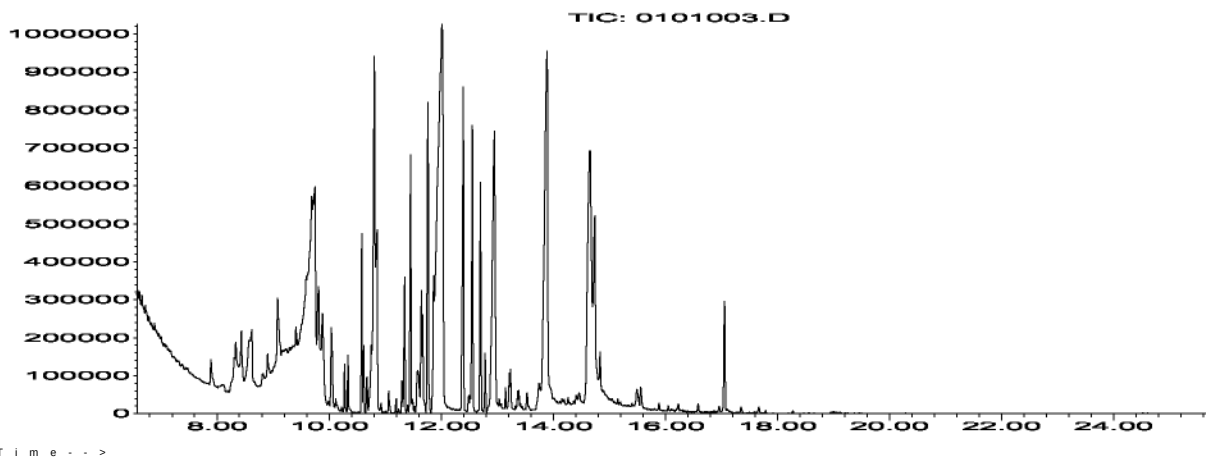
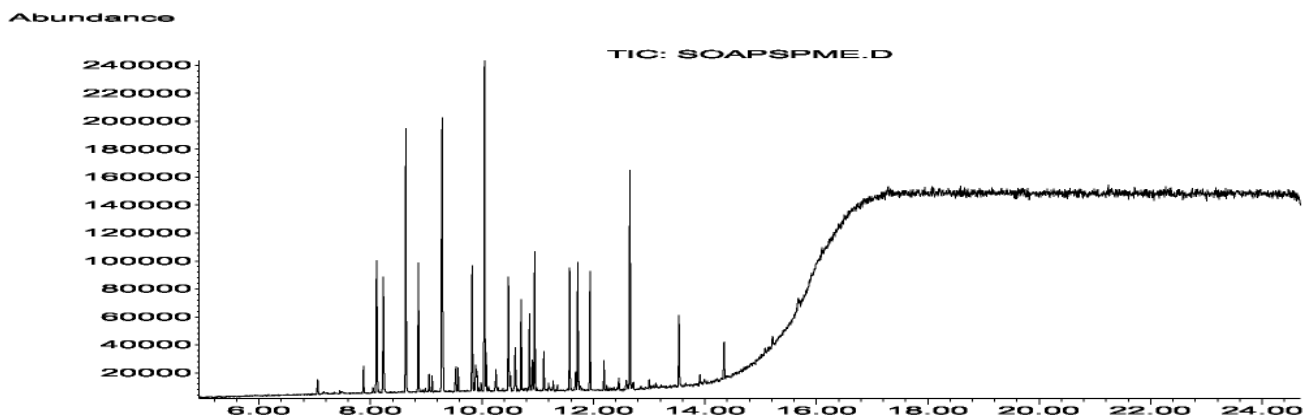


Figure 8: SPME analysis of the headspace from a 1g sample of soap

Abundance

Focus:

Syringe size: 2.5 mL
Incubation temp: 75°C
Incubation time: 15 mins
Syringe temp: 75°C
Agitator speed: 500 rpm
Fill speed: 500 uL/s
Inject speed: 500 uL/s
Flush time: 2 mins

Optic:

Isothermal temp: 300°C
Isobaric pressure: 10 psi
Mode: Split
Split flow: 10 mL/min

Oven:

Column: HP5-MS 30m x 0.25mm id x 0.25um film
Initial temp: 70°C
Initial time: 2 mins
Ramp rate: 25°C/min
Final temp: 325°C
Final time: 10 mins

MSD:

Mode: Scan
Low mass: 29 m/z
High mass: 300 m/z
Sampling: 2
Transfer line: 280°C
Solvent delay: 2 mins

Focus:

Fiber type: Polyacrylate
Incubation temp: 75°C
Incubation time: 15 mins
Agitator speed: 500 rpm
Vial needle pen: 22 mm
Extraction time: 5 mins
Injector needle pen: 54 mm
Desorption time: 3 mins

Optic:

Isothermal temp: 300°C
Isobaric pressure: 10 psi
Mode: Split
Split flow: 10 mL/min

Oven:

Column: HP5-MS 30m x 0.25mm id x 0.25um film
Initial temp: 40°C
Initial time: 4.5 mins
Ramp rate: 25°C/min
Final temp: 325°C
Final time: 10 mins

MSD:

Mode: Scan
Low mass: 29 m/z
High mass: 300 m/z
Sampling: 2
Transfer line: 280°C
Solvent delay: 2 mins

Optic:

Initial temp: 35°C
Initial time: 0.5 mins
Sweep pressure: 8 psi
Sweep flow: 100 mL/min (vent)
Desorption pressure: 0 psi
Desorption time: 2.5 mins
Ramp rate: 16°C/s
Final temperature: 250°C
Split flow: 50 mL/min
Transfer pressure: 20 psi
Transfer time: 2 mins
Initial pressure: 15 psi
Final pressure: 15 psi

Oven:

Column: DB5-MS 30m x 0.25mm id x 0.25um film
Initial temp: 40°C
Initial time: 4.5 mins
Ramp rate: 25°C/min
Final temp: 325°C
Final time: 10 mins

MSD:

Mode: Scan
Low mass: 29 m/z
High mass: 300 m/z
Sampling: 2
Transfer line: 280°C
Solvent delay: 4.5 mins