

Application Note No. 085

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

*Diane Nicholas*

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

### ***Instrumentation***

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

### ***Techniques and samples***

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- 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***

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

### ***Principles***

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- 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

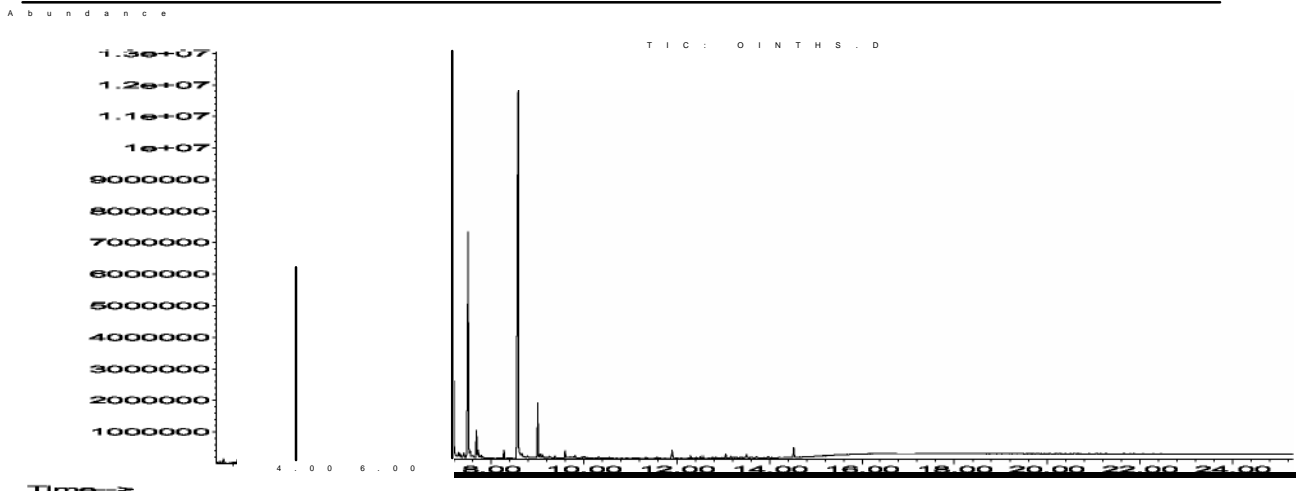


Figure 1: Headspace analysis of a 1g sample of an ointment

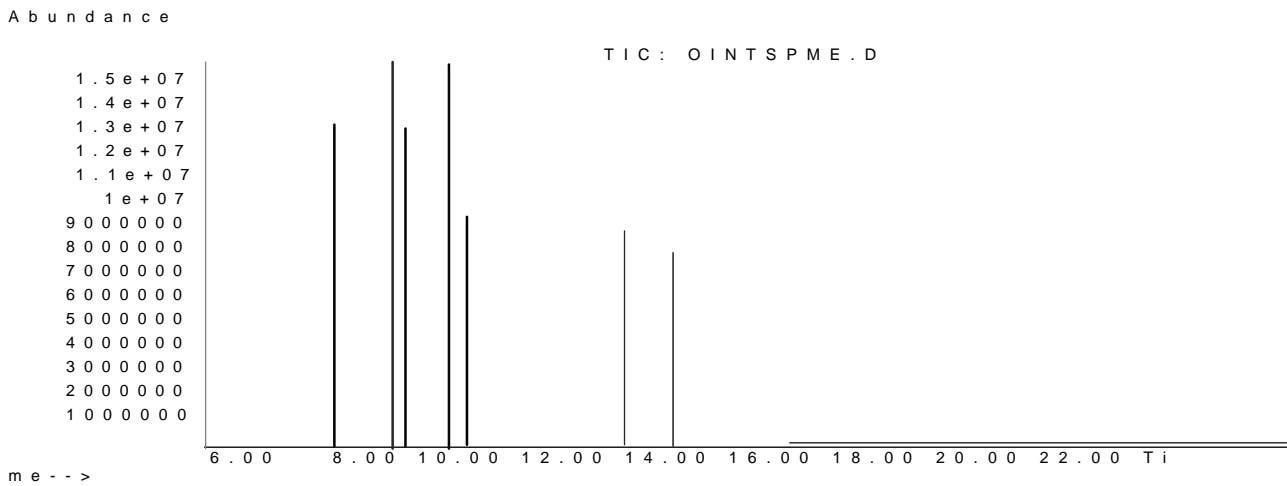
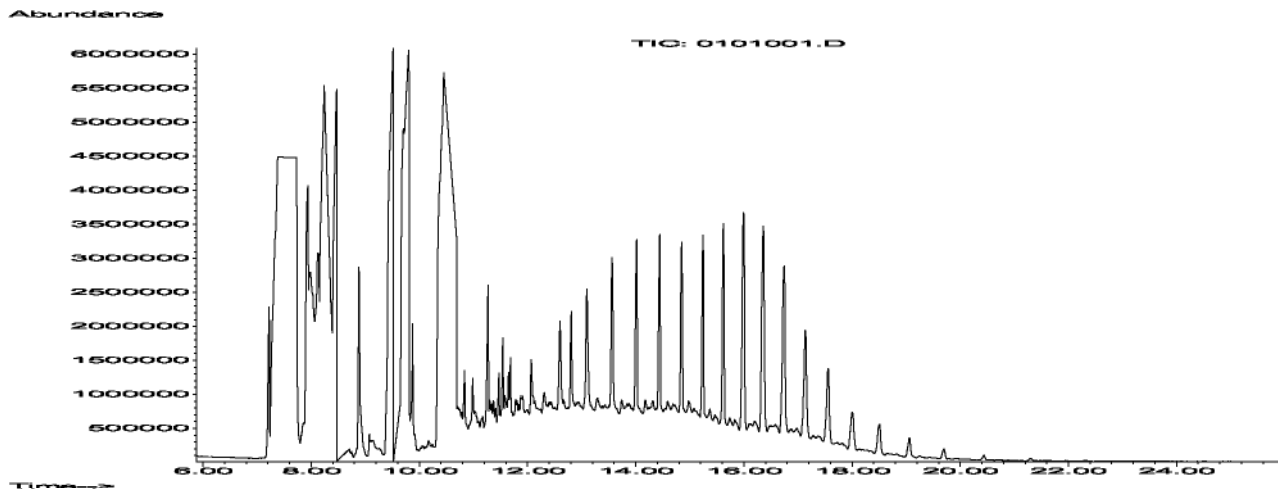
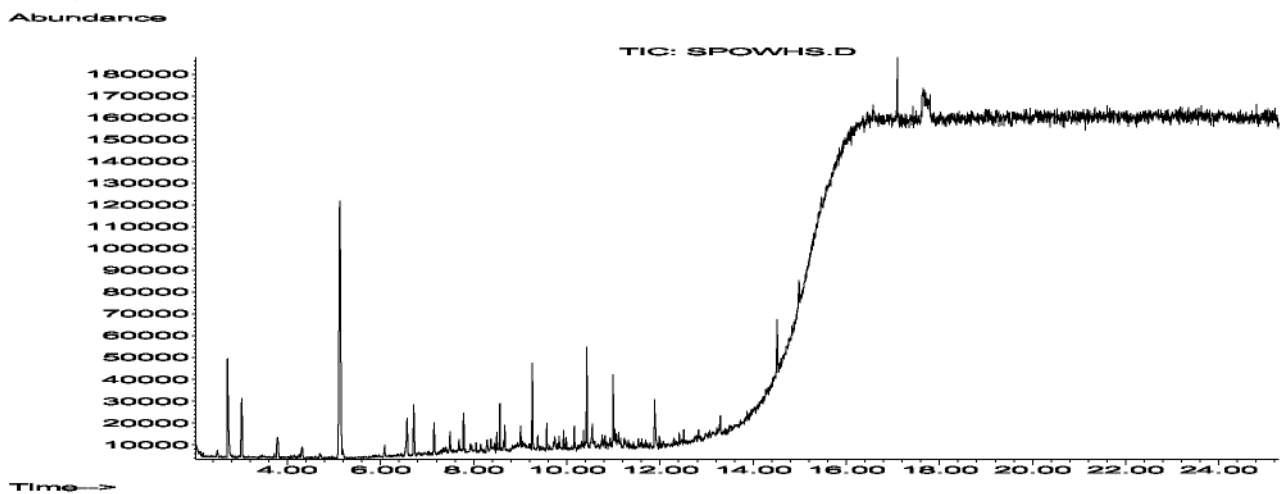
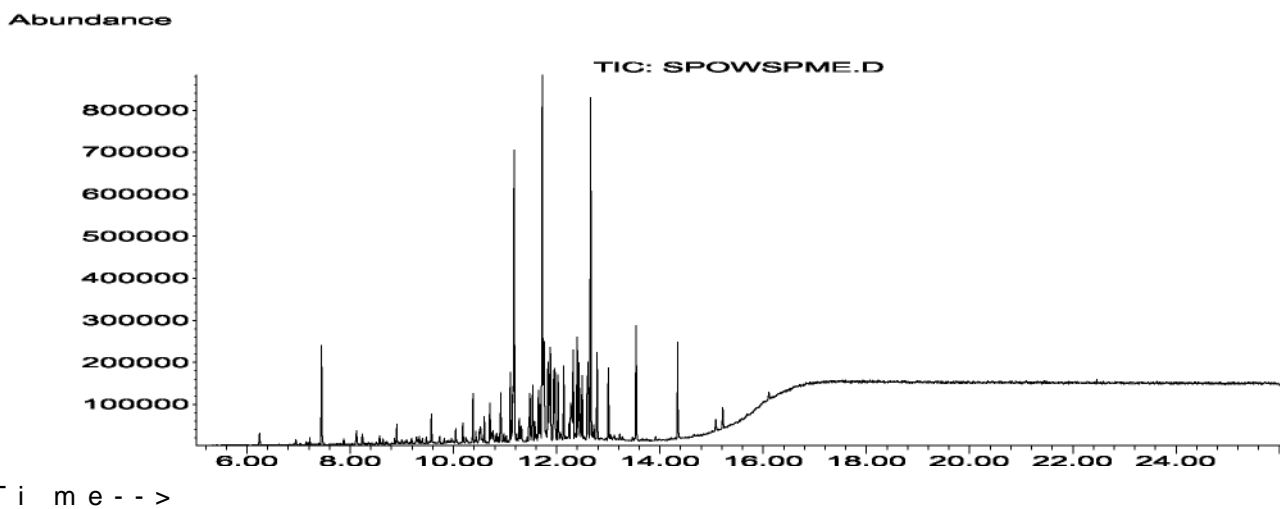


Figure 2: SPME analysis of the headspace from a 1g sample of an ointment



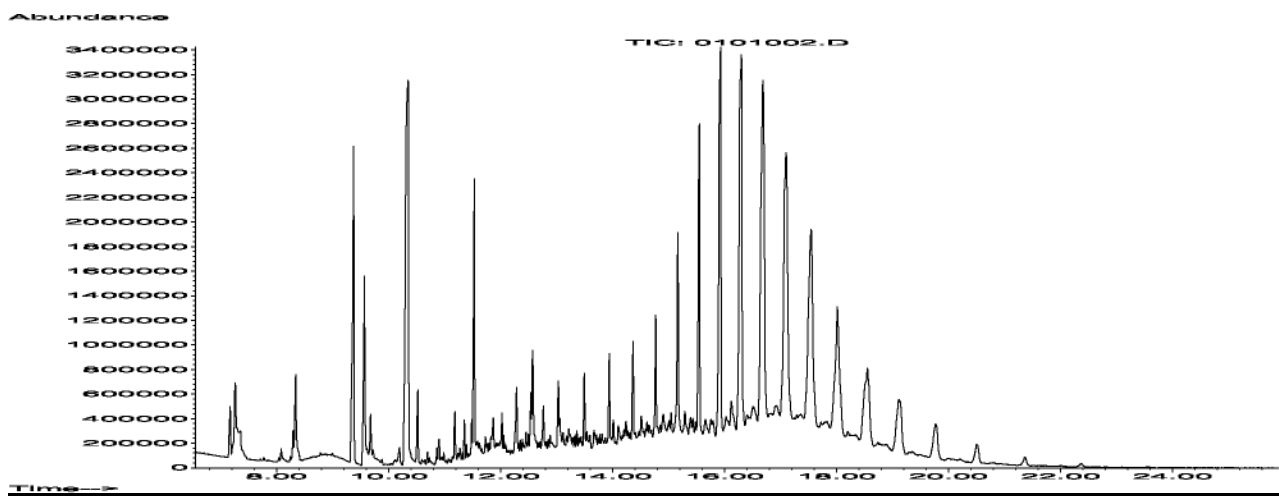


**Figure 4:** Headspace analysis of a 1g sample of soap powder

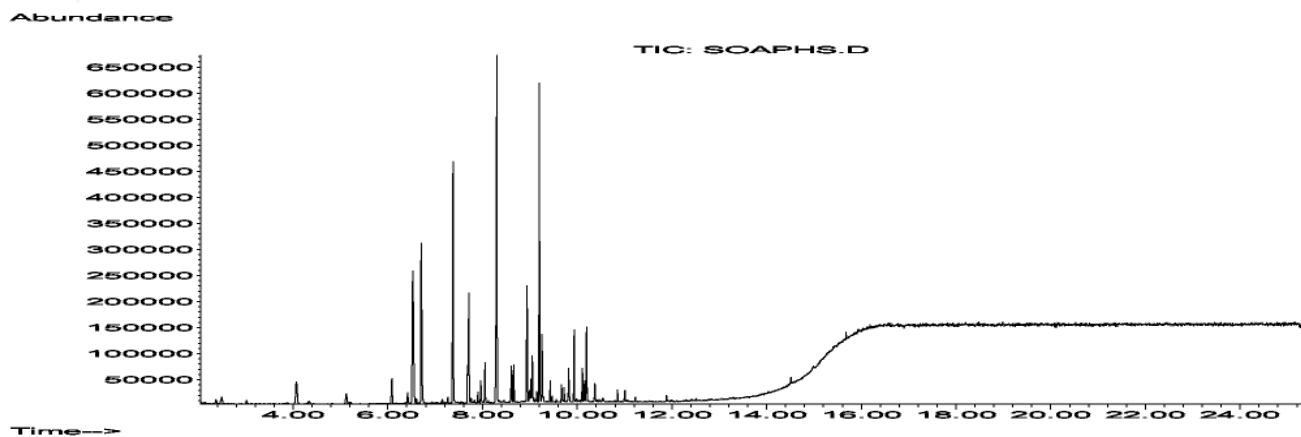


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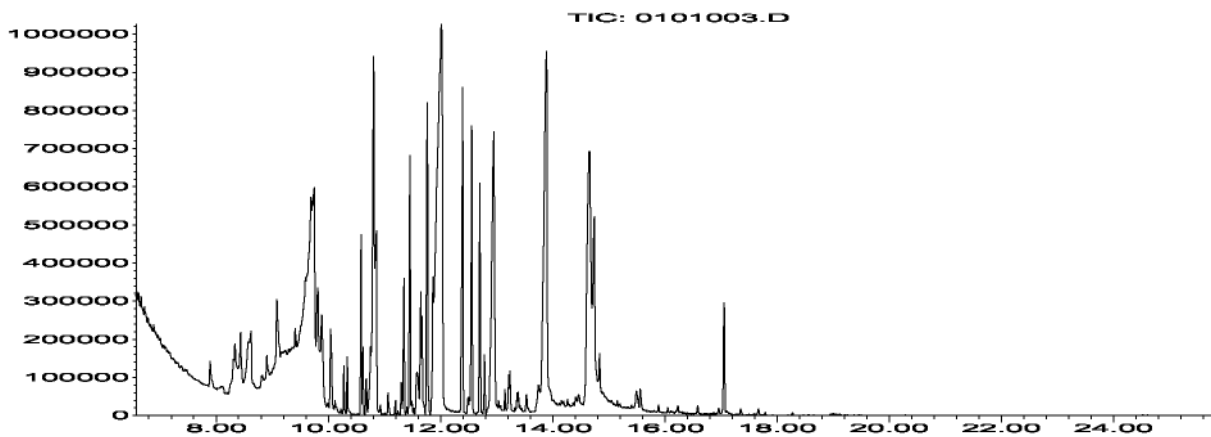
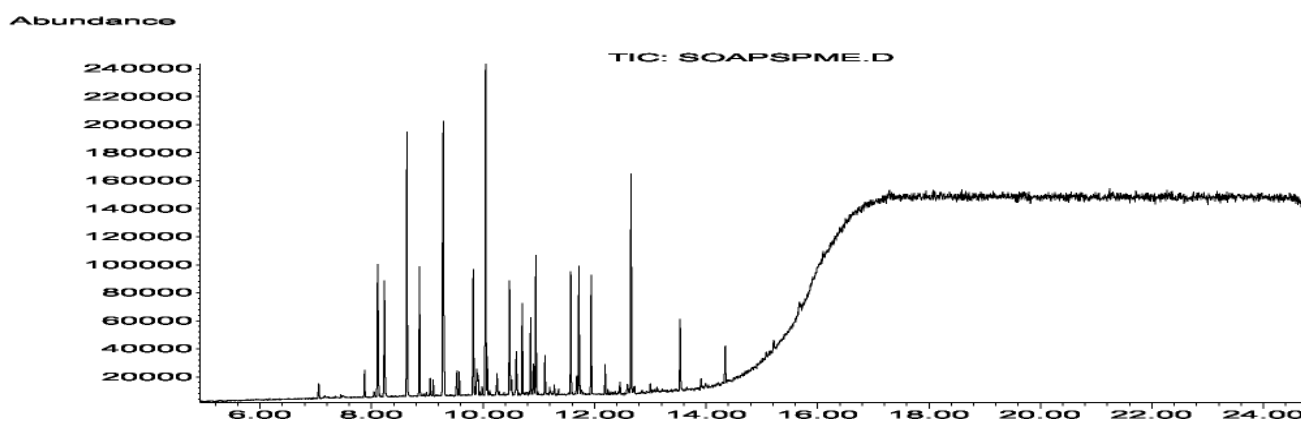
**Figure 5:** SPME analysis of the headspace from a 1g sample of soap powder



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



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



Time . . . >

**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

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