Medical Materials Testing by Headspace Trap-GC/MS

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Introduction

This Field Application Report investigates the volatile organic compounds (VOCs) present in surgical products, specifically sutures. Some of these VOCs are considered toxic impurities, so they need to be identified at the lowest possible detection levels since they may be absorbed into the human body and introduce the potential for adverse metabolic reactions.

A new sample-introduction product, the PerkinElmer® TurboMatrix™ HS-40 Trap, was coupled to a PerkinElmer Clarus® 500 GC/MS and used for this investigation. The TurboMatrix HS-40 Trap is a headspace system that incorporates new technology – a trapping process that pre-concentrates and focuses the VOCs before injection into the GC. This provides the ability to identify very-low-level volatile compounds that may not be detected by traditional static headspace sampling. Figure 1 is an example analysis of trace-level compounds found in the volatile headspace of suture material.

Experimental

Approximately 350 mg of sample (suture material) was placed into a glass headspace vial and heated at 100 ˚C for 30 minutes. The headspace sample vapor was automatically collected onto a multi-bed sorbent tube (Air Toxic Trap™) at 40 ˚C inside the trap system. After 1 minute of collection, the tube was heated at 40 ˚C/min to 280 ˚C, desorbing the volatile compounds onto a

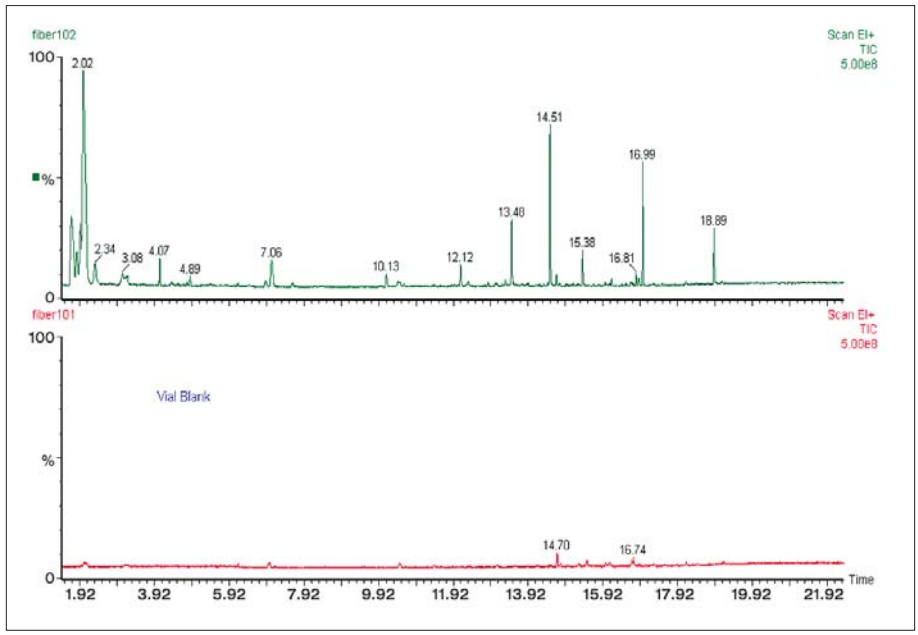

Figure 1. Comparison of suture fiber vs. blank.

PerkinElmer® precisely.

RTX®-200 Column, 30 m x 0.32 mm x 1.0 µm (Restek Corporation, Bellefonte, PA USA). This transfer took place via the heated transfer line on the headspace trap system through a programmable split/splitless injector (PSSI). The GC, MS and HS Trap conditions are listed in Tables 1-3, respectively.

Figure 2 shows chromatograms from two different suture samples. In both samples, the peak at 6.90 minutes was identified to be toluene. This was confirmed by comparison with both retention time and spectral data from a 1 µL spike of 8 ng/µL toluene standard using purge-and-trap-grade methanol as the solvent. Also, a cluster of peaks from 1.6 to 2.5 minutes that were identified as isomeric hexanes was present in both samples. The peaks at 7.14 and 7.06 minutes in samples 1 and 2 respectively were also identified as tetrachloroethylene.

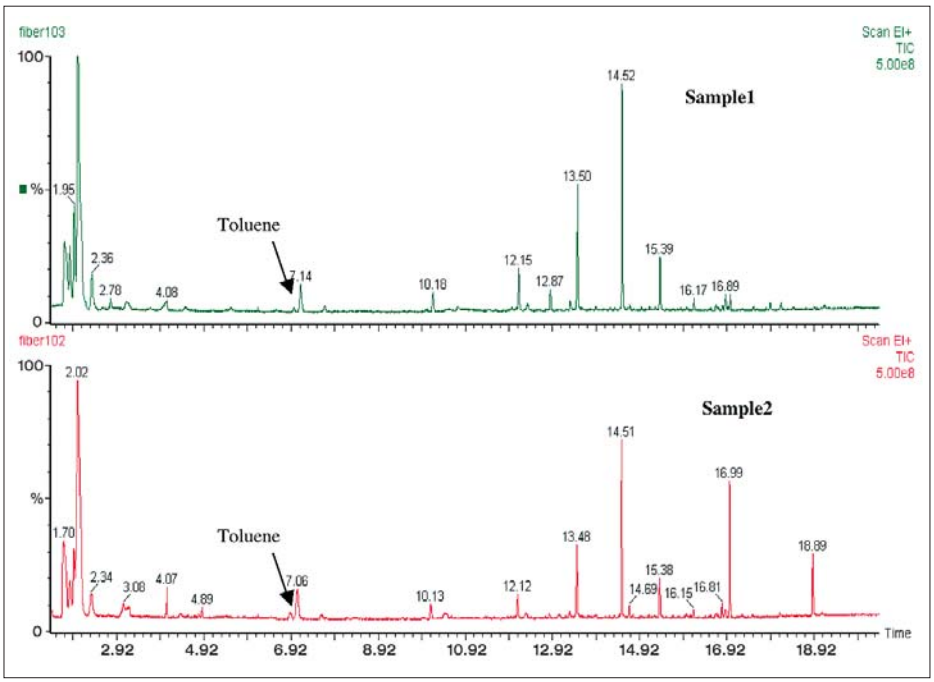

Figure 2. Comparison of two medical fiber samples.

A homologous series of aliphatic aldehydes along with ketones and a phthalate ester (typically from plasticizers) were found mid-chromatogram and they are identified in Table 4. The spectra of the two chromatograms shown in Figure 2 were processed via a TurboMass NIST spectral library search and associated AMDIS spectral deconvolution software. Both software packages assigned the compounds listed in Table 4.

Table 1. GC Conditions.

Oven Initial Temperature:	35 °C
Time 1:	5 mins
Rate 1:	10 °C/min
Temperature 2:	100 °C
Ramp 2:	20 °C/min
Final Temperature:	250 °C
Hold Time:	5 mins
Injector Temperature:	200 °C
Column Flow:	2 ml/min
Splitflow:	5 ml/min

Table 2. MS Conditions.

Scan:	35-300 amu @ 5 scans/sec
Run Time:	24 mins
Source Temperature:	180 °C
Transfer Line Temperature:	200 °C
Emission Current:	50

Table 3. Headspace Trap Conditions.

Oven:	100 °C
Trap High/Low:	280 °C/40 °C
Trap Hold:	5 min
Total Cycle Time:	35 min
Thermostat Time:	30 min
Decay Time:	1 min
Column Pressure:	10 psi

Conclusion

The new technology provided by the TurboMatrix HS-40 Trap coupled with a sensitive detector such as the Clarus 500 GC/MS allows VOCs contained in medical sutures to be analyzed easily at trace levels. Individual compounds present in the sutures can be analyzed by GC/MS and identified by doing a NIST library search of the acquired mass spectral data.

The innovative, patent-pending, headspace trap technology used in this application provides sensitivity beyond the capability of traditional static headspace. This presents a new level of detection capability for the evaluation of materials used in medical applications, as well as in other types of material testing, including pharmaceutical formulations and food-packaging film.

Table 4. Compounds Identified Using NIST and AMDIS.

Retention Time (min)	Compound
10.1	hexanal
12.1	heptanal
13.5	octanal
14.5	nonanal
15.4	decanal
12.87	cyclohexanone
17	dioxane-dione
18.9	diethyl phthalate

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