

Analysis of Commercially Available Hand Soap by TD-GC-TOFMS

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1. Introduction

Thermal desorption provides a fast and relatively simple way to analyze solids and liquids. Its advantages over solvent extraction include decreased extraction time, elimination of hazardous solvents and overall simplification of the extraction process, as well as increasing sample throughput. Gerstel's Twister Desorption Unit (TDU)/Cooled Injection System (CIS4) Inlet, when combined with the Multi-Purpose Sampler (MPS2), allows for the automation of the sample introduction process.

2. Instruments and Methods

In this study, measurements were made with a LECO Pegasus® HT Gas Chromatograph (GC)—Time-of-Flight Mass Spectrometer (TOFMS) system. This system consists of an Agilent 6890 GC and a LECO Pegasus HT TOFMS as a detector. The system is equipped with a Gerstel Multi-Purpose Sampler (MPS2) and a Gerstel Twister Desorption Unit (TDU)/Cooled Injection System (CIS4) Inlet. This system is shown in Figure 1. For this study, the analytical column was a Gerstel MACH-Low Thermal Mass unit consisting of a 10.0 m x 0.18 mm ID x 0.18 mm df RTX-5 column, mounted externally on the GC oven door. The transfer line was 1.00 m x 0.10 mm ID x 0.10 mm df RTX-5. Helium was used as the carrier gas at a constant flow of 1.0 mL/minute. Sample introduction is accomplished by automated thermal desorption.

The sample is first placed into a micro-vial insert. The micro-vial insert is then placed into the TDU insert which is then loaded into the sample tray on the MPS2. Upon initiation of the analysis, the TDU is at ambient temperature and the CIS4 is chilled to -120°C using liquid nitrogen. The MPS2 then removes the TDU insert from the sample tray and places it into the TDU (see Figure 2). The TDU is heated to 40°C at a rate of 20°C/minute. The sample is thermally desorbed and then cryogenically focused in the CIS4. Once the sample is completely desorbed, the CIS4 is heated at 12°C/second and the focused analyte is injected onto the analytical column. In this one-dimensional separation, the temperature program of the MACH column was as follows.

Rate (°C/min)	Target Temp (°C)	Duration (min)
Initial	30.0	1.00
20.00	250.00	3.00

The GC oven containing the transfer line was maintained isothermally at 250°C.

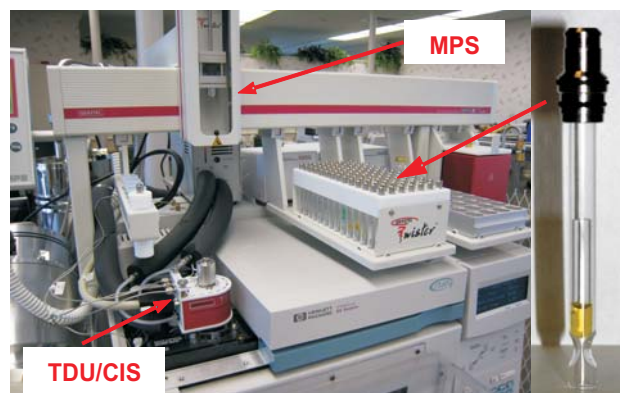


Figure 1: The Gerstel MPS2-TDU/CIS4 as mounted on a LECO Pegasus HT GC-TOFMS and the TDU insert with micro-vial sample insert.

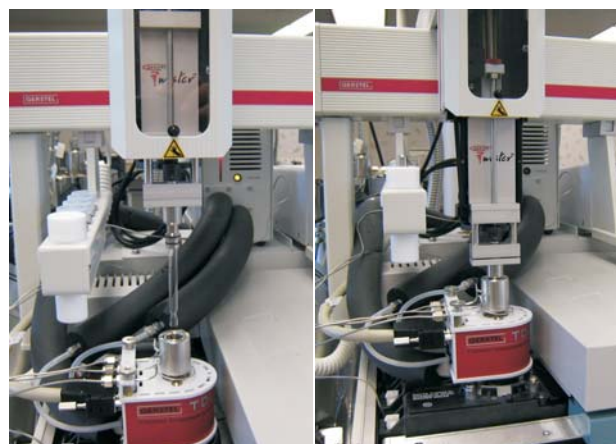


Figure 2: The Gerstel MPS2 transferring the TDU insert from the sample tray to the TDU (left) and loading the TDU insert into the TDU (right).

3. Results

Commercially available, scented liquid hand soap was analyzed by TD-GC-TOFMS. By utilizing the thermal desorption, no extraction solvent was needed, reducing the number of steps in the extraction process as compared to a traditional solvent extraction. The lack of extracting solvent also means less waste and a more environmentally friendly process. Thermal desorption also requires less time for sample preparation, leading to increased sample throughput. The chromatogram showing the Total Ion Count (TIC) trace is shown in Figure 3. There are coelutions (such as that occurring around 345 seconds) shown in the TIC trace in Figure 4.

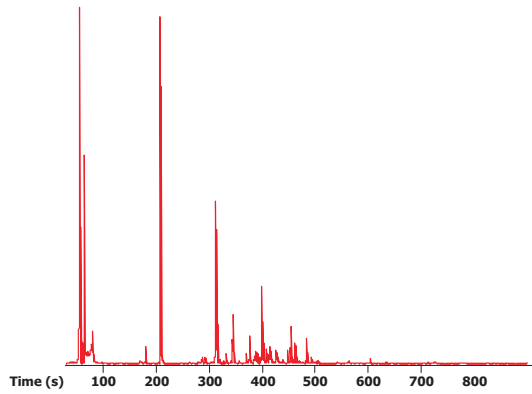


Figure 3: The TIC one-dimensional chromatogram from the thermal desorption of commercially available liquid hand soap. ChromaTOF[®] software identified 132 peaks with a S/N ≥ 100 .

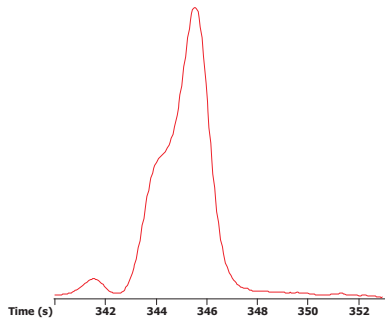


Figure 4: The TIC one-dimensional chromatogram showing a coelution at approximately 345 seconds.

It is possible to tell from the TIC trace in Figure 4 that there is a coelution, but it is difficult to tell how many peaks are coeluting. By using the power of True Signal Deconvolution™ (made possible by the full range spectra provided by the Pegasus HT TOFMS), it is possible to determine how many peaks are coeluting. The deconvoluted chromatogram is shown in Figure 5, with peak markers identifying 5 individual peaks.

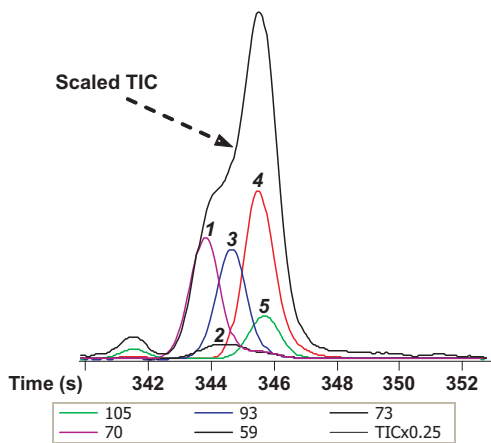


Figure 5: The deconvoluted trace of the peak at 345 seconds with the scaled TIC shown in black. The unique ions for the 5 coeluting peaks are shown in addition to the scaled TIC.

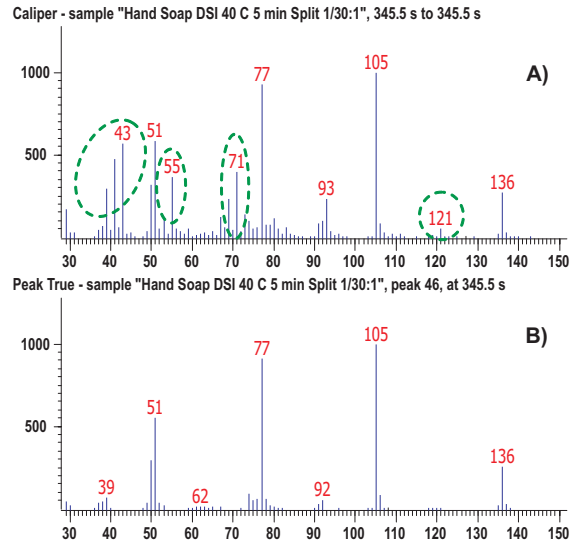


Figure 6: The caliper (A) and peak true (B) spectra for peak 4 in Figure 5. Matrix interference from coeluting peaks (highlighted in green; masses 43, 55, 71, and 121) are automatically removed to produce the deconvoluted peak true spectra shown in (B).

Figure 6 shows the caliper and peak true spectra for peak 4 in Figure 5. The deconvoluted peak true spectrum is lacking the interfering ions present in the caliper spectrum.

4. Conclusions

TD-GC-TOFMS is shown to increase sample throughput and eliminate the use of hazardous solvents and the need to dispose of them. The ability of the Pegasus HT TOFMS to collect full-range spectra at rates up to 500 spectra/second allows for the True Signal Deconvolution of coeluting peaks. The use of the MACH-LTM column allows for faster heating and cooling of the analytical column, thereby minimizing GC recovery time between analyses. When combined with the automated nature of sample handling and introduction, the overall sample analysis time is decreased, thereby increasing sample throughput.

