

Using THERMAL DESORPTION – GAS CHROMATOGRAPHY for the identification of fragrance and other components in cosmetic products

Introduction

Cosmetic products such as soap, shampoo, lotions and creams are often scented with suitable compounds to improve their appeal. This application note describes an analytical method to identify the fragrance and other compounds contained within these cosmetic formulations.

Because of the solid or viscous nature of these formulations, direct sample injection into a gas chromatograph is not feasible and some form of analyte extraction is normally required. The approach taken in this work was to place a small amount of the sample in a thermal desorption tube and use an inert carrier gas to purge the volatile sample constituents into a cooled secondary trap. The secondary trap was then rapidly heated to desorb the collected components into a gas chromatographic column for separation and subsequent detection and identification by mass spectrometry.

This thermal desorption – gas chromatography – mass spectrometry (TD-GC-MS) technique may be fully automated using standard instrumentation and so provides a convenient and easy way of screening difficult sample matrices such as cosmetic products for constituent volatile compounds

Sample Preparation

Approximately 120 mg of sample was placed within a glass wool plug in a PTFE thermal desorption tube insert as shown in Figure 1a. The flexible tube was squeezed to disperse liquid samples into the glass wool and so ensure an open pathway for carrier gas to flow through it.

The PTFE insert was then placed in an empty stainless steel desorption

tube, as shown in Figure 1b, ready for analysis.

The following samples were examined:

- Liquid soap
- Body cream
- Massage lotion
- Face cream
- Bathroom soap bar
- Shampoo
- Body lotion

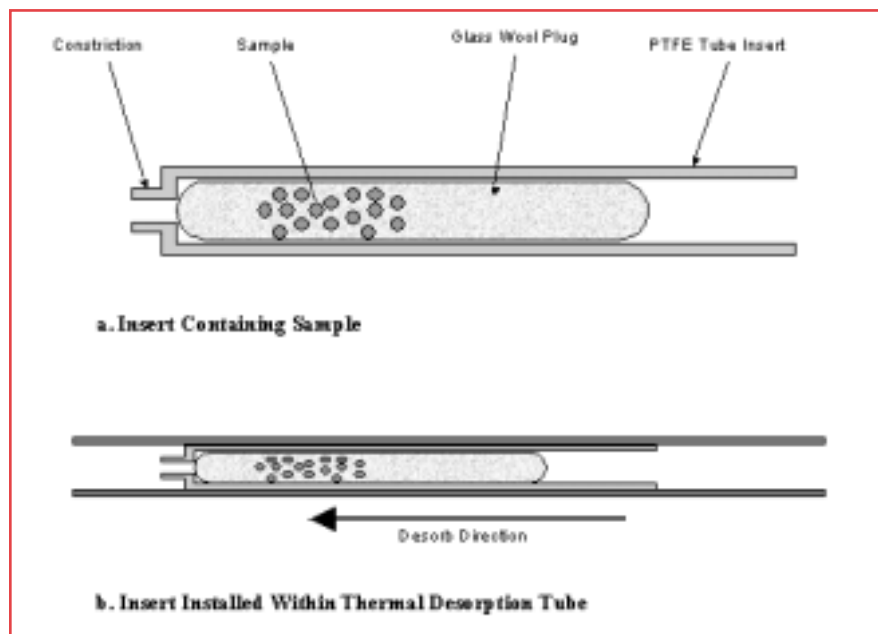


Figure 1. Preparation of Sample Tube

Analytical conditions

Each sample tube was analyzed using TD-GC-MS under the conditions listed in Table 1.

Table 1. Conditions used for the determination of fragrance and other components in cosmetic formulations

Chromatograph	Perkin Elmer AutoSystem™ XL Gas Chromatograph
Column	30 m x 0.32 mm x 0.25 µm PE-1, dimethylpolysiloxane
Oven	40°C for 1 min, then 5°C /min to 280°C
Thermal Desorber	Perkin Elmer TurboMatrix™ TD
Trap Adsorbents	50:50 Tenax TA and Carbotrap B in series
Carrier Gas	Helium at 5.0 psig
Tube Purge	1 min
Tube Desorb	30 mL/min at 80°C for 10 min
Trap Low	-30°C
Trap High	300°C for 5 min
Inlet Split	None
Outlet Split	30 mL/min
Valve	250°C
Transfer Line	250°C
Mass Spectrometer	Perkin Elmer TurboMass™ Mass Spectrometer
Mass Range	35 to 450 Da
Scan Time	0.5 sec
Source Temperature	250°C
Transfer Line Temperature	250°C
Photomultiplier	409 Volts

Results and Discussion

Figure 2 shows a total ion chromatogram (TIC) obtained from the liquid soap sample. Despite the 'messy' nature of this sample, clean chromatography has been obtained thus enabling easy data processing. The TurboMass software was used to examine the mass spectral data of each of the major components and to perform a library search to identify each of these. Figures 3, 5 and 7 show some typical mass spectra taken from the liquid soap chromatogram and Figures 4, 6 and 8 show the corresponding results from the spectral library search. Figures 9 to 15 show the total ion chromatograms, with annotated peak identities, for the remaining samples.

Conclusions

This note has described a simple means of identifying fragrance and other volatile compounds in cosmetic preparations without the need for tedious and lengthy sample extractions

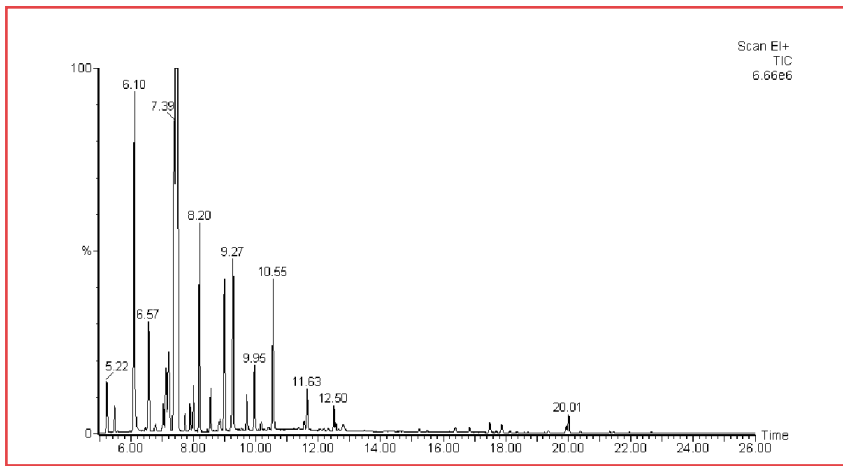


Figure 2. Total ion chromatogram of liquid soap sample.

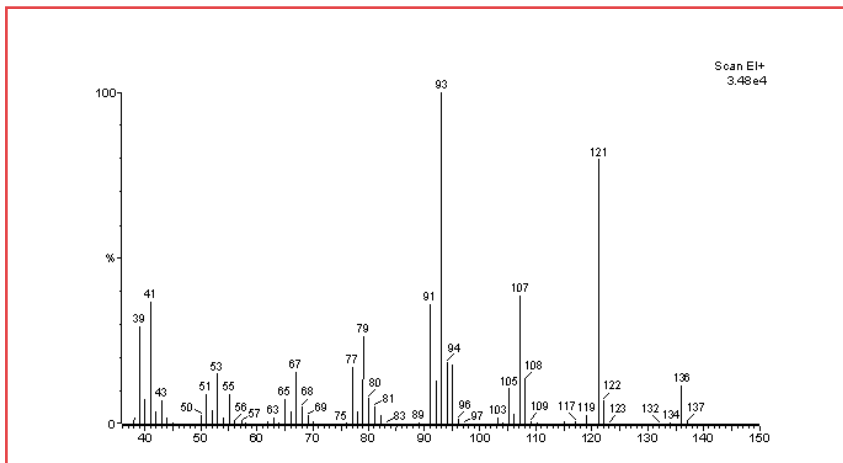


Figure 3. Mass spectrum of the compound at 5.22 minutes in liquid soap chromatogram.

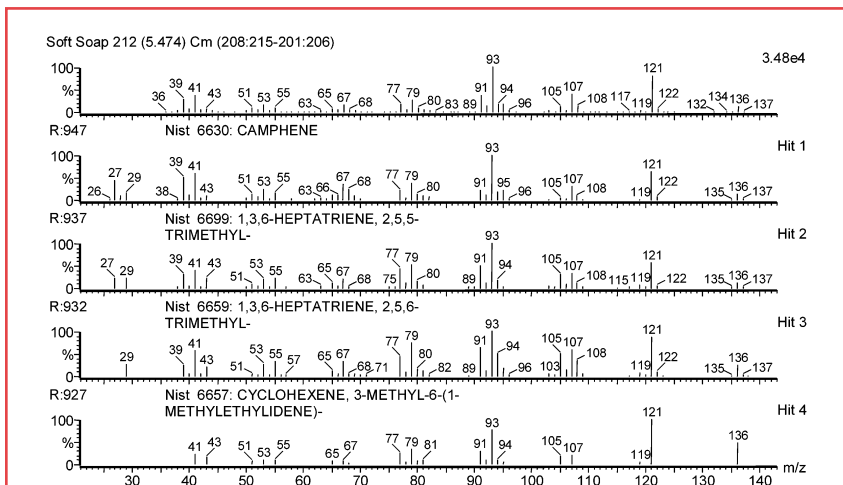


Figure 4. Results of library search on mass spectrum of compound A in liquid soap confirming the presence of camphene.

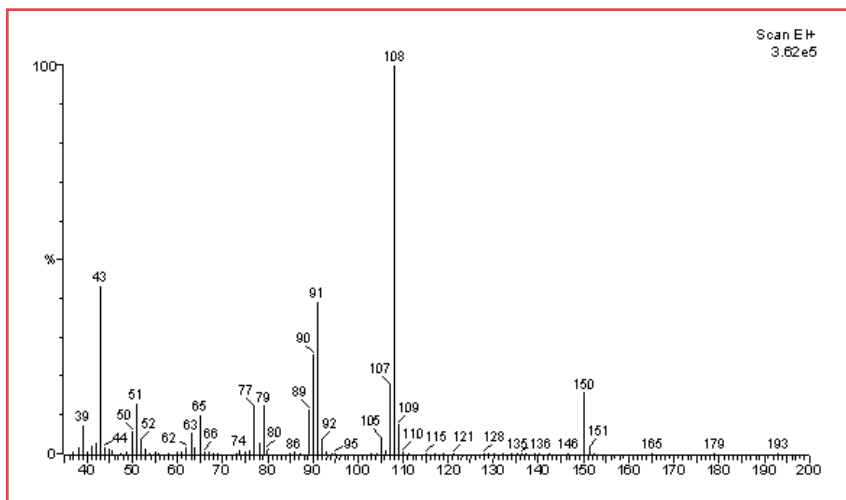


Figure 5. Mass spectrum of the compound at 10.55 minutes in liquid soap chromatogram.

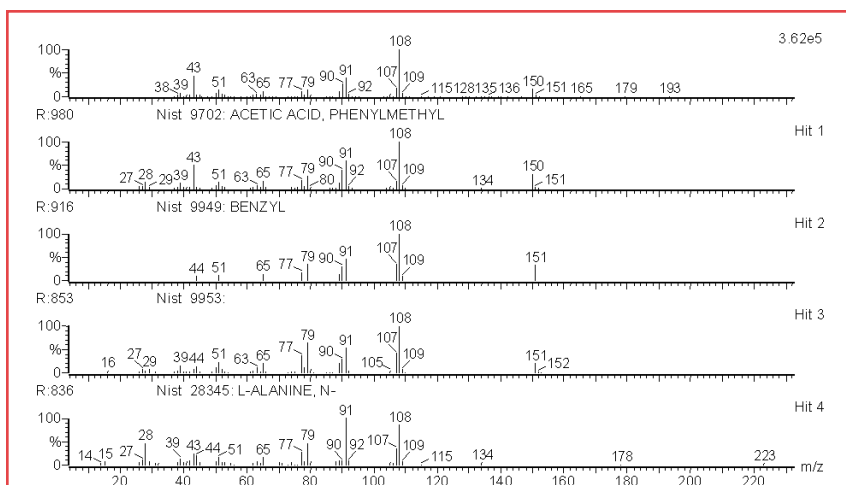


Figure 6. Results of library search on mass spectrum of compound B in liquid soap confirming the presence of Acetic Acid, Phenyl Methyl Ester (Benzyl Acetate).

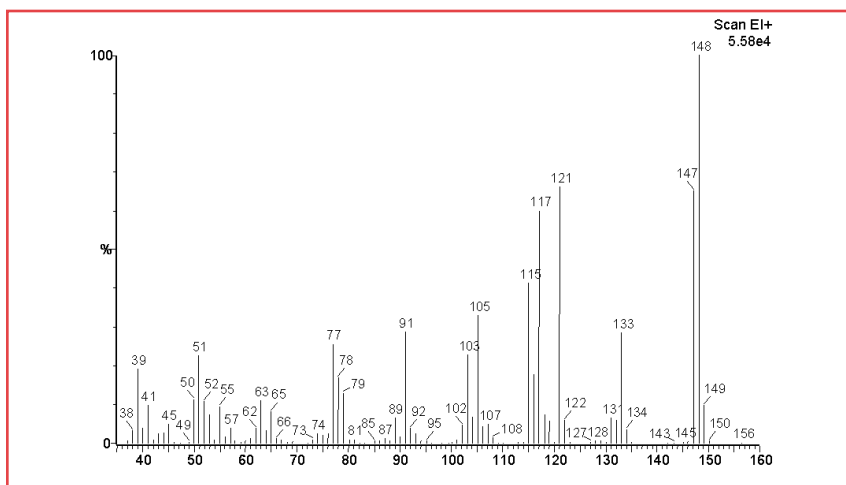


Figure 7. Mass spectrum of the compound at 11.63 minutes in liquid soap chromatogram.

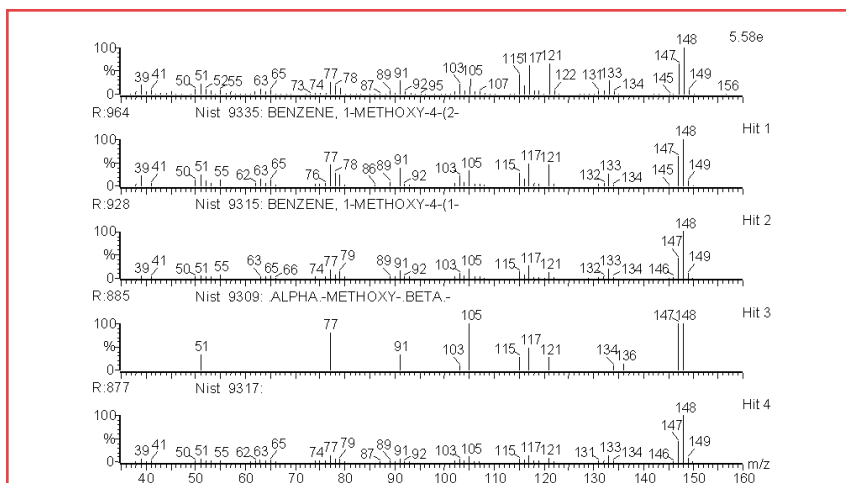


Figure 8. Results of library search on mass spectrum of compound C in liquid soap confirming the presence of 1-Methoxy-4-(2-Propenyl)-Benzene (Estragole).

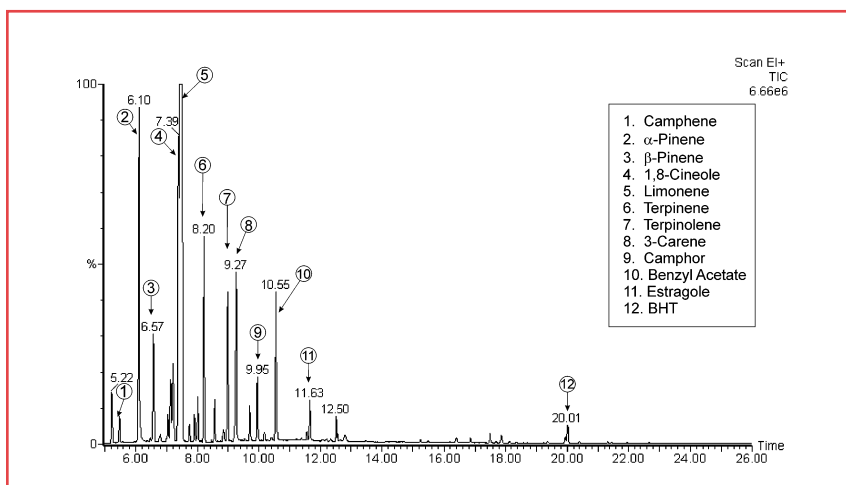


Figure 9. Identities of major components in liquid soap sample.

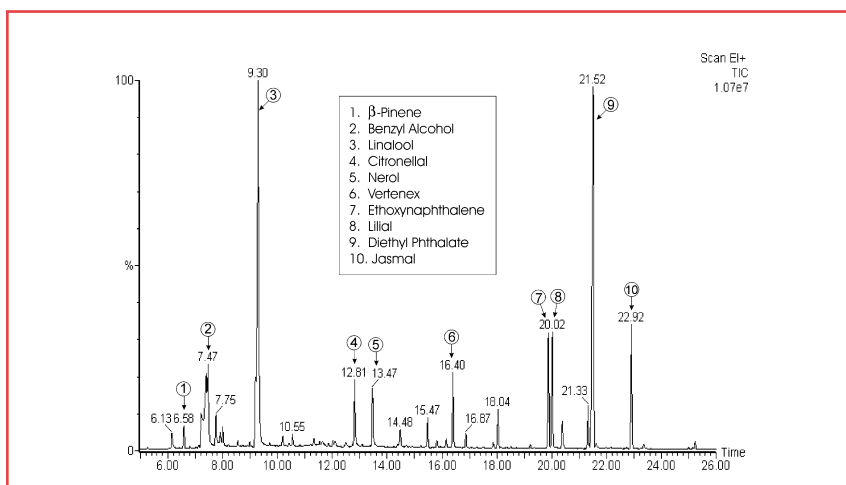


Figure 10. Identities of major components in bar soap sample.

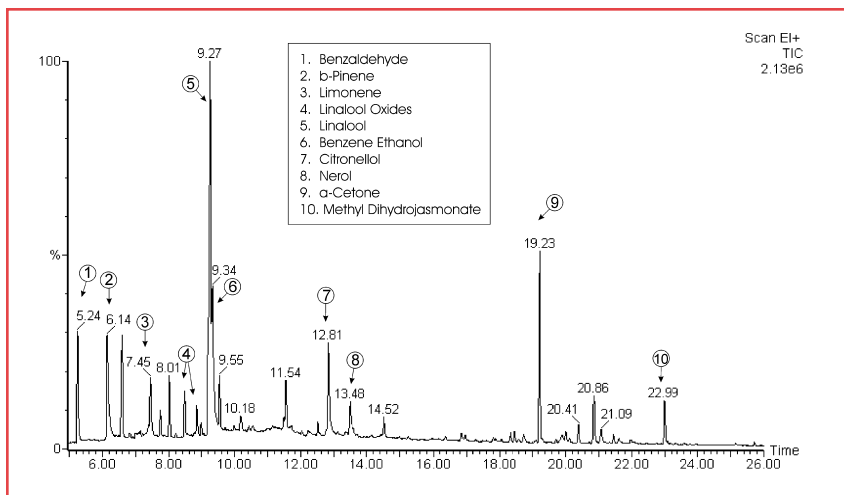


Figure 11. Identities of Major Components in Body Cream Sample

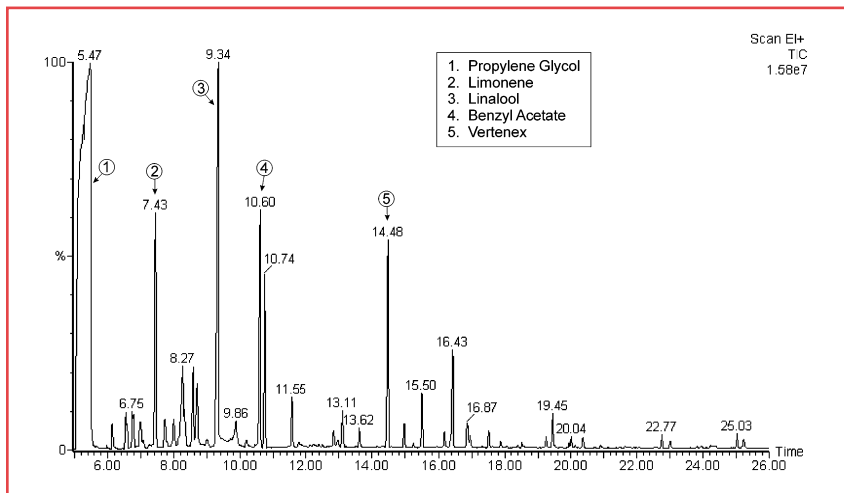


Figure 12. Identities of Major Components in Massage Lotion Sample

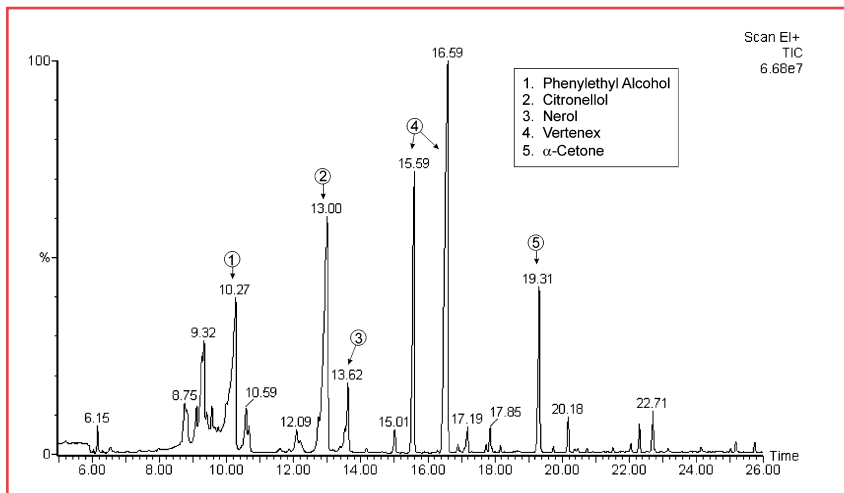


Figure 13. Identities of Major Components in Face Cream Sample

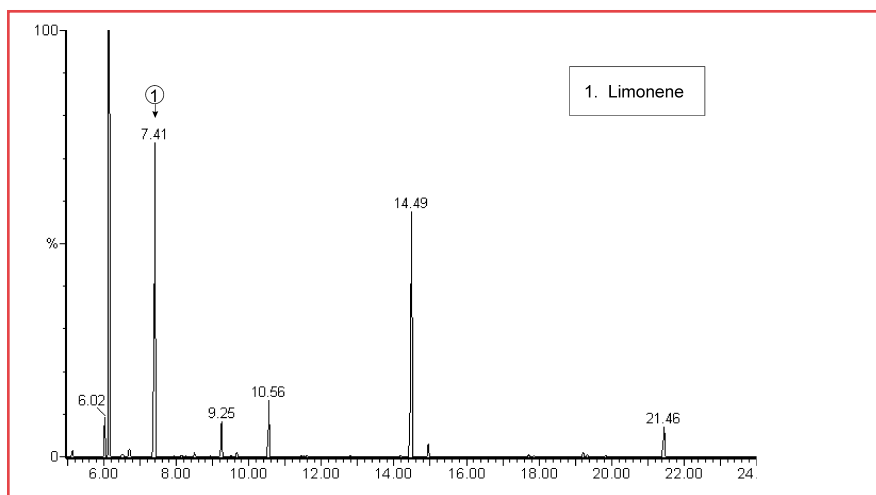


Figure 14. Identities of Major Components in Shampoo Sample

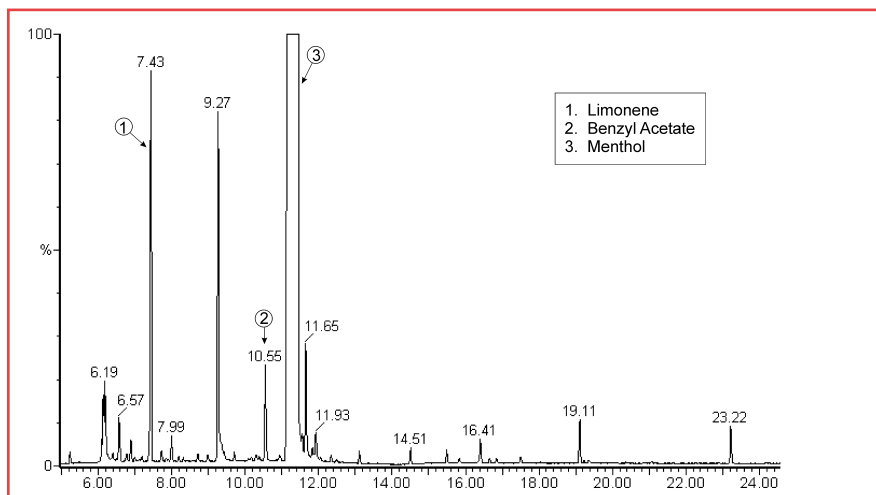


Figure 15. Identities of Major Components in Hand Cream Sample

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