

Determination of Fragrance in Candle Wax by Thermal Desorption-Gas Chromatography/Mass Spectrometry

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Introduction

In the manufacturing of scented candles, there is a need to determine the amount of fragrance as well as the compounds of the fragrance transferred to the candle wax. This field application report describes a fast and accurate method to meet this need.

Samples tested were candles with and without fragrance as well as the neat fragrance itself.

Experimental conditions

The following instrumentation was used to run these analyses:

- PerkinElmer® TurboMatrix™ 50 Thermal Desorber (TD)
- PerkinElmer Clarus® 500 Gas Chromatograph/Mass Spectrometer (GC/MS)

One objective is to determine which compounds are transferred to the wax; therefore, it is important to confirm that the thermal-desorption technique does not introduce compound discrimination. To investigate this, a 0.4- μ L neat injection of the fragrance was introduced into a flash-vaporization GC injector port using common methodology for analyzing a fragrance. 1.0 μ L of the

same fragrance was spiked onto a thermal-desorption tube containing Tenax® TA and glass wool, and then desorbed onto the analytical column using the PerkinElmer TurboMatrix 50 Thermal Desorber. Figure 1 (Page 3) is a stacked representation of the neat fragrance from the tube and the neat fragrance from the injector.

The analytical parameters for both experiments were the same, except the carrier was driven by constant flow in the injector-port experiment and by constant pressure in the TD injection. In addition, the split ratio was higher in the TD injection; the volume injected was adjusted for the respective split ratios to achieve approximately the same amount loaded onto the column in each experiment.

The next step was to desorb the fragrance from a wax sample with acceptable recoveries and minimal matrix interference. 0.1411 g of candle wax was inserted into a glass TD sample tube with a 1-cm bed of Tenax® TA and glass wool. A blank wax (matrix devoid of fragrance) was analyzed as a control sample to characterize any matrix interference. Figure 2 (Page 3) is a stacked representation of the candle wax without fragrance, of the candle wax with fragrance and of the 1.0- μ L neat fragrance spiked onto the sample tube (from the initial experiment).

Tables 1 and 2 contain the TD and GC/MS conditions, respectively.

Desorb Temp:	150 °C
Desorb Flow:	50 mL/min
Desorb Time:	10 min
Purge Time:	1 min
Trap Low:	5 °C
Trap High:	290 °C
Trap Hold:	20 min
T Line Temp:	280 °C
Valve Temp:	225 °C
Inlet Split:	10 mL/min
Outlet Split:	40 mL/min
Pressure:	20 psi

Column:	Elite 5ms – 60 m x 0.25 mm x 0.25 µm
Oven Parameters	
Initial Temp:	50 °C
Initial Hold:	2 min
Rate 1:	3 °C/min
Final Temp:	270 °C
MS Parameters	
Transfer Line Temp:	250 °C
Source Temp:	240 °C
Mass Range:	35 to 500 amu
Ionization Mode:	EI
Scan Duration:	0.2 sec
Interscan Delay:	0.1 sec
Solvent Delay:	4.6 min

Discussion and results

The following ratios of peak areas within each injection were computed.

- Low-boiling compound (Ethyl Acetate) to a mid-boiling compound (Cinnamic Alcohol)
- Low-boiling compound (Ethyl Acetate) to a high-boiling compound (Musk Ambrette)
- Mid-boiling compound (Cinnamic Alcohol) to a high-boiling compound (Musk Ambrette)

The difference of these ratios between the inlet injection and the TD injection were used to evaluate any potential discrimination introduced by the thermal-desorption technique.

Referring to Table 3, there is acceptable correlation of area ratios between the inlet injection of the neat fragrance vs. compounds introduced through TD injection; therefore, thermal desorption is a viable tool.

Additionally, excellent fragrance recovery was achieved from the candle with minimal matrix interference across the fragrance-compound boiling-point range. In Figure 2 (Page 3), the chromatographic profile from the neat fragrance desorbed from a tube matches that of the fragrance desorbed from the candle.

Although Tenax® TA was utilized as the adsorbent in this method, the preferred trap material for this application would be a multi-bed of Carbopack F or fused-silica beads (1/4), Carbopack C (1/2) and Carbopack B (1/4). Not only are these adsorbents more robust than Tenax® TA, but by using a multi-bed adsorbent, the higher-molecular-weight compounds will be retained on the weaker adsorbent, Carbopack F; the mid-boiling-point compounds will be retained on Carbopack C; and Carbopack B (which is stronger than Tenax® TA) provides a safety to ensure lighter compounds will not break through. This provides enhanced recoveries and efficient desorption.

Compound	Compound Abbreviation Used	Area Inlet injection	Area TD injection
Ethyl Acetate	EA	83557104	60120632
Cinnamic Alcohol (3-phenyl-2-Propen-1-ol)	CA	174102096	118224584
Musk Ambrette (Oxacycloheptadec-8-en-2-one)	MA	326231296	229879664
	Area Ratio EA to CA	Area Ratio EA to MA	Area Ratio CA to MA
Injection via Injector Port	0.480	0.256	0.534
Injection via Thermal Desorption	0.509	0.262	0.514
% Difference	-5.96%	-2.11%	3.63%

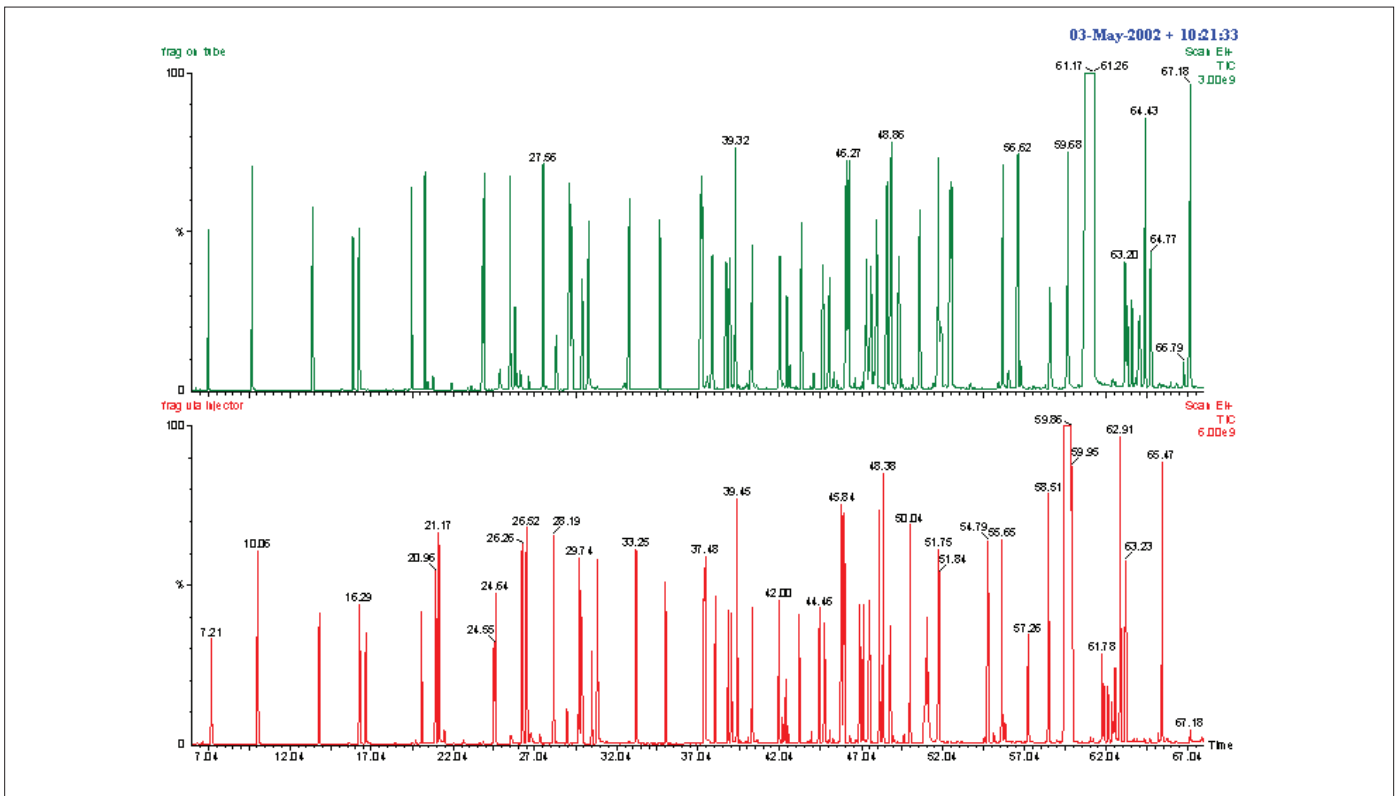


Figure 1. The chromatographic profile from the neat fragrance desorbed from a tube matches the profile of the sample introduced via the injector port. *Top Chromatogram:* 1.0 μ L of fragrance spiked on tube and desorbed onto column. *Bottom Chromatogram:* 0.4 μ L of fragrance injected via injector port.

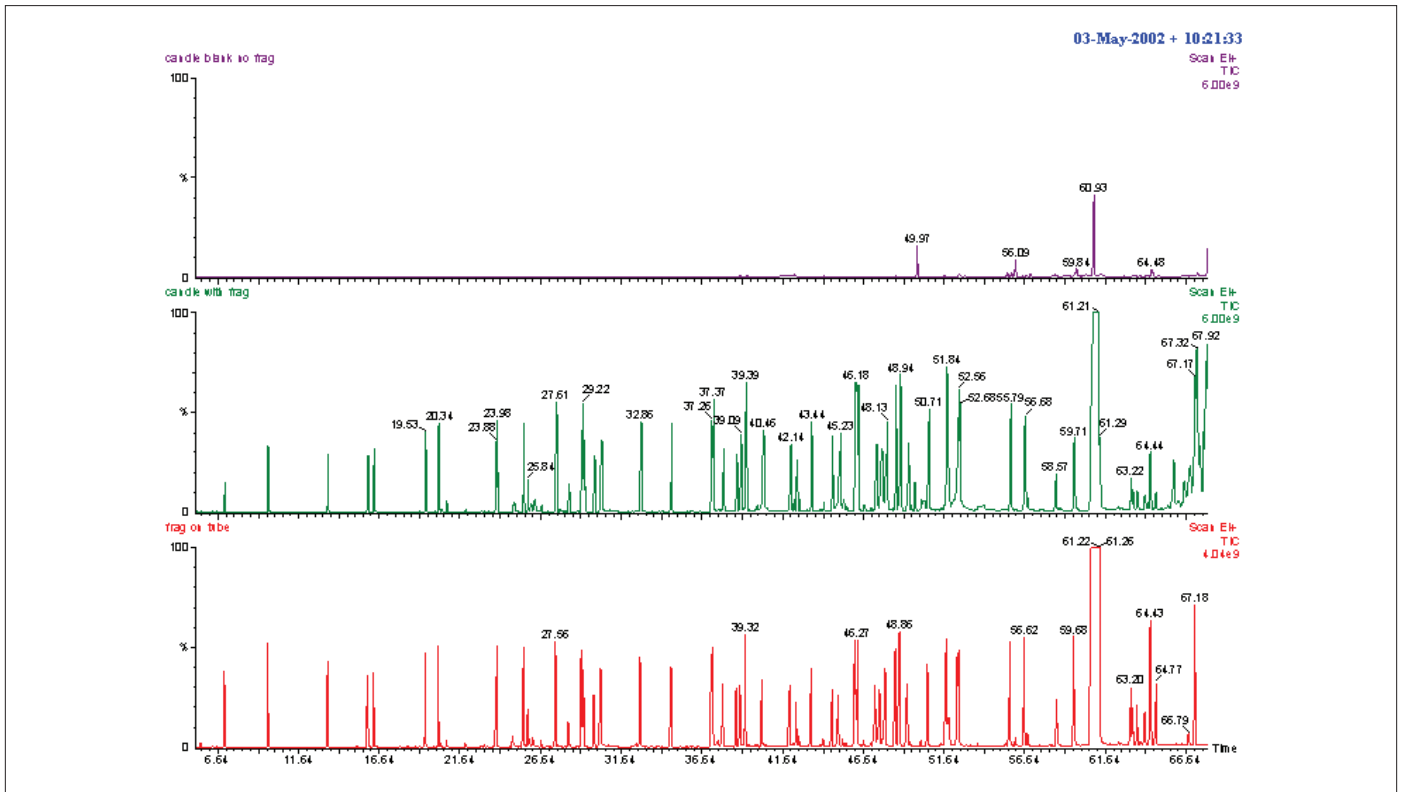


Figure 2. Comparison chromatograms of candle wax without fragrance, with fragrance and neat fragrance. *Top Chromatogram:* Candle wax without fragrance. *Middle Chromatogram:* Candle wax with fragrance. *Bottom Chromatogram:* 1.0 μ L of neat fragrance desorbed from Tenax[®] TA tube.

A significant advantage of the PerkinElmer TurboMatrix Thermal Desorber is that the carrier flow, when desorbing the cold trap, is reversed. When compounds enter the cold trap during concentration, the direction of flow into the cold trap is from weaker adsorbent to stronger adsorbent, so the higher-molecular-weight compounds do not enter the stronger or mid-range adsorbent. When desorbing the cold trap for injection, the flow is reversed through the trap; therefore, the higher-molecular-weight compounds are efficiently desorbed from the weaker adsorbent and never enter the stronger adsorbent.

For a multi-boiling-point compound mixture, as a fragrance, a multi-bed adsorbent is optimal. If one uses a single-bed adsorbent, there is a risk of breakthrough of the lower-boiling-point compounds during concentration

if this adsorbent is too weak to retain such compounds, and there is risk of not desorbing the higher-boiling-point compounds during trap desorption if the adsorbent is too strong for these compounds to be released.

Conclusion

Thermal desorption as an analytical tool in material testing has substantial advantages. This approach offers a quick, robust, automated technique which provides enhanced recoveries and precision, especially compared to liquid extraction, and eliminates interference from extraction solvents. In addition, thermal desorption enables solutions in many matrices where liquid extraction is not an option.

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