Thermal Desorption Analysis of Essential Oil Emissions from Common In-Home Diffusers

Author:

Michael Apsokardu

Application Note
Environmental

Abstract
This application note demonstrates a method for sampling and analyzing VOC emissions of essential oils from in-home diffusers. As part of this work, VOC emissions from three different popular types of essential oils were examined. Additionally, one ultrasonic diffuser and two-nebulizing diffusers were selected to compare emissions output of VOC's for a specific lemon essential oil. For the most abundant VOC in lemon oil, limonene, the emitted concentration was determined for the ultrasonic diffuser and one nebulizing diffuser.

Introduction
Recently, essential oil emissions from in-home have become a topic of increasing interest due to potential implications on indoor air quality. Benefits of essential oils includes reducing stress and anxiety while improving alertness and brain function. Conversely, the negative effect of essential oil diffusers and their impact on air quality and human health is also widely debated. Adverse health effects caused by essential oils include allergic reactions or causing respiratory stress on asthmatics. Additionally, cats and dogs are far more sensitive to odors than humans, so many essential oils are known to have potentially damaging health effects to pets.[1-4]

Regardless of the debated pros and cons of essential oils, the essential oil market has grown in recent years, and the market has become flooded with different types and brands of essential oil diffusers. Common types of diffusers include ultrasonic (water-based), nebulizing, passive, and heated diffusers. Passive diffusers are often subject to uneven evaporation of components while heated diffusers can potentially cause chemical changes to the essential oil. For these reasons, ultrasonic and nebulizing diffusers are considered to be the most popular within the market.

In this application note, VOC emissions from three different types of essential oils are examined. Three commercial in-home diffusers were also selected to analyze the VOC emissions. VOC emissions are sampled onto conditioned thermal desorption tubes using a CDS Air Sampling device, which comes with a built-in vacuum pump to assist with drawing air through the thermal desorption tube. After the completion of sampling, thermal desorption was done using the CDS 7550S automated Thermal Desorber is combined with GC-MS. Using lemon essential oil as a case study, emissions of VOC's from different types of diffusers is compared and quantified.

Experiment Setup
For this study, two popular, highly-rated nebulizing diffusers available on Amazon were selected. One was the Aroma-Ace diffuser and the other was from Everyday Alchemy. Additionally, one ultrasonic diffuser manufactured by Sentsy was also selected. For all experiments, each diffuser was set to the maximum output. Also, 20 minutes is a frequently used time before automatic shut off for nebulizing diffusers, therefore, 20 minutes was selected as the sampling time for all experiments. The amount of essential oil added was based on manufacturer recommendations. All essential oils were purchased from Radha beauty. The sampling was done with the assistance of a CDS Air Sampling device containing a built-in vacuum with adjustable flow rate range between 10 and 700 mL/min.

Table 1:

7550S Thermal Desorber:	
Valve oven:	250 °C
GC transfer line:	250 °C
Tube purge flow:	60 mL/min
Pre-heat time:	15 s
Tube Rest temp.:	40 °C
Tube Dry purge temp.:	40 °C
Tube Dry purge time:	0.1 min
Tube Desorb temp.:	300 °C
Tube Desorb time:	8 min
Sample tube:	Camsco P/N SU644-4
Trap Rest temp.:	-20 °C with Peltier
Trap Desorb temp.:	300 °C
Trap Desorb time:	4 min
Trap Type:	Vocarb 3000
Peltier transfer line:	250 °C
GCMS QP-2010	
GC conditions:	
Column:	Restek Rxi 5Sil MS
Oven temp.:	35.0 °C
Injection temp.:	240 °C
Injection mode:	Split
Column Flow:	1.01 ml/min
Split Ratio:	40.0 : 1
Temp. program:	35.0 °C hold 4 min 10.0 °C/min to 150.0 °C 50.0 °C/min to 320.0 °C Hold 3.10 min
MS conditions:	
Ion Source:	200.00 °C
Interface Temp.:	220.00 °C
Start m/z:	35.00
End m/z:	260.00

All sampling was done at a rate of 700 mL/min. The inlet of the thermal desorption tube was 6" from the outlet of the diffuser in each case. All samples were collected in a closed room with negligible air flow. Between each sample, the ventilation system in the room was turned on the remove excess VOC's in the air.

Emissions from the diffuser were sampled with 1/4" × 3.5" stainless-steel thermal desoprtion tubes that were manufactured by CAMSCO (PN SU644-4) and contain carbograph 2/ carbograph 1/carboxen 1000 packing.

For preparation of the calibration curve, standard solutions of limonene were prepared in methanol between 20 and 630 ppm. In each standard solution, an 87 ppm solution of toluene was used as the internal standard. Standard solutions were loaded onto thermal desorption tubes by in 1 μL of liquid onto the tube and purging the tube with clean nitrogen gas for 1 minute at 120 mL/min. Samples collected from diffusers were spiked with an 87 ppm toluene solution and purged under the same conditions.

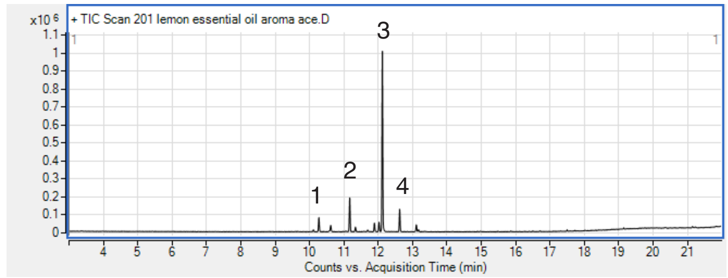

Figure 1. Chromatogram resulting from lemon essential oil diffused from the Aroma Ace nebulizing diffuser for 20 minutes.

A CDS 7550S automated thermal desorber was employed with the Peltier option. GC-MS was performed using a Shimadzu QP 2010. TD-GC-MS analysis was performed according to the parameters in Table 1.

Results and Discussions

Three popular essential oils were sampled using the CDS Air Sampler and analyzed with the 7550S automated thermal desorber. Essential oils that were tested include lemon, cinnamon, and, lavender. Figure 1 shows an example chromatogram that was collected by diffusing lemon essential oil from the Aroma-Ace nebulizing diffuser. Lemon oil is primarily composed of monoterpenes and sesquiterpenes. The four most intense peaks, labeled 1-4, were identified as α-pinene, β-pinene, d-limonene, and γ-terpinene.[4] Cinnamon and lavender essential oils were also sampled using the same nebulizing diffuser and their representative chromatograms are shown in Figure 2. The base peak of the cinnamon chromatogram (top) was identified as eugenol, which is known to have antiseptic and anesthetic properties. Interestingly, none of the key contributors to the odor of cinnamon were observed in the chromatogram. In the lavender oil chromatogram (bottom), the base peak was identified as linalool, which is the primary VOC in lavender and is responsible for floral scents. The other peaks of the chromatogram are other minor terpenes also known to be present in lavender oil.[5] Of the essential

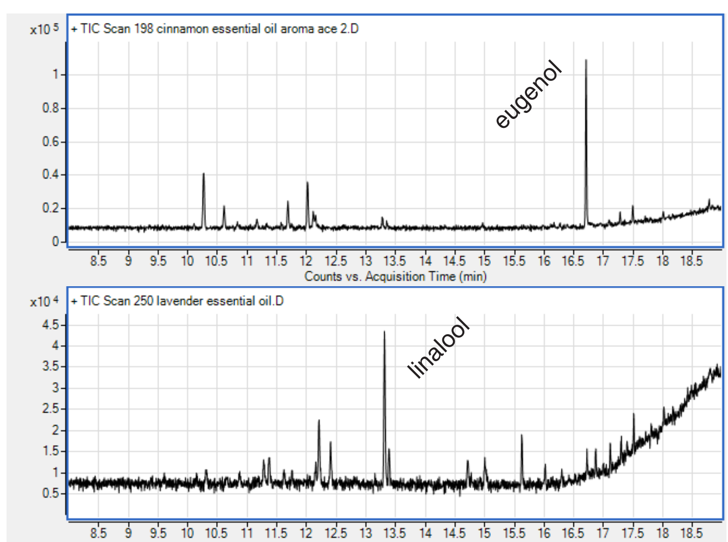

Figure 2. Chromatographs resulting from cinnamon (top) and lavender (bottom) essential oils diffused from the Aroma-Ace nebulizing diffuser for 20 minutes.

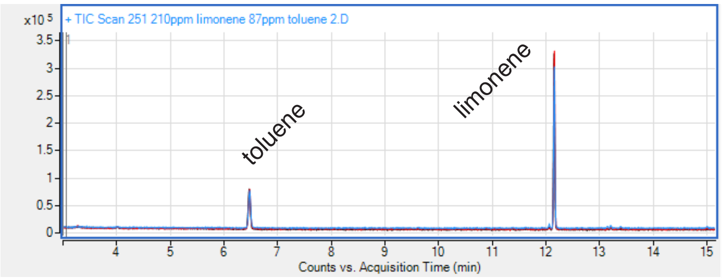

Figure 3: Overlay of the TIC from three chromatographs obtained from loading thermal desorption tubes with 210 ng of limonene and 87 ng of toluene.

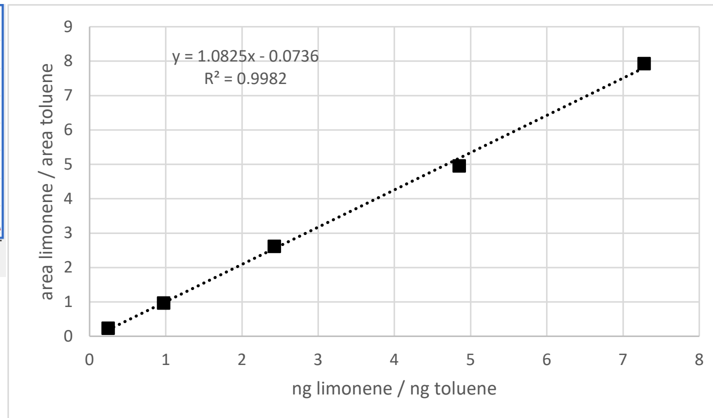

Figure 4. Calibration curve for limonene with ng limonene / ng toluene vs integrated peak area limonene / area toluene. ng is refers to the mass loading as nanograms.

oils tested here, lemon essential oil produced most intense response for all three types of diffusers. Therefore lemon oil was chosen to comparatively assess the concentration of limonene specifically emitted from all three types of diffusers discussed here. A calibration curve was generated by spiking thermal desorption tubes with standard concentrations of limonene. Toluene was used as an internal standard for the analysis. Each sample tube also spiked with toluene to add internal standard prior to analysis.

Figure 3 shows three stacked chromatographs when loading thermal desoprtion tubes with 210 ng of limonene and 87 ng of toulene from a standard solution. The RSD of the signal intensities for both limonene and toluene were 3%, indicating that both analytes were reproducibly desorbed and transferred from the 7550S thermal desrober to the GC-MS. The final calibration curve is shown in Figure 4. This was used to determine the amount of limonene emitted from Sentsy ultrasonic diffuser and the Everyday Alchemy nebulizing diffuser. Measurements for both were done in replicates of three. As determined from the calibration curve for the Sentsy ultrasonic diffuser, 60 ng of limonene was sampled over a 20 minute period at a rate of 700 mL/min. During this time, 14 L of air would have been sampled. This equates to a concentration of 4.1 $\mu g/m^3$, or 0.7 ppb. Similarly, the concentration of limonene emitted from the Everyday Alchemy nebulizing diffuser was calculated as well. The calculated concentration of emitted limonene was 9.2 $\mu g/m^3$, or 1.6 ppb. The results indicate that the concentration of limonene emitted from the nebulizing diffuser is more than 2 times higher than the concentration emitted from the ultrasonic diffuser, when using the manufacturer recommended operating conditions. This result could be expected due to the fact nebulizing diffusers use pure essential oils. For Sentsy ultrasonic diffuser, the oil is diluted in approximately 60mL of water.

Conclusions

This application note demonstrates methods for sampling and evaluating air quality from the use of in-home essential oil diffusers. Three different types of essential oils were tested with three highly-rated diffusers. One diffuser was an ultrasonic, water-based diffuser and the other were nebulizing diffusers. For this work, lemon essential oil VOC's were the most readily detected with all three diffusers. Limonene was selected specifically to use the 7550S thermal desrober to perform a quantitative study for the output of limonene from the different essential oil diffusers. In general, the nebulzing diffusers emitted limonene at higher concentrations than the ultrasonic diffuser. This is not surprising considering nebulizing diffusers use pure essential oils while essential oils are diluted in water when using the ultrasonic diffuser. This study indicates that the 7550S automated thermal desorber can reproducibly and quantitatively determine concentrations of VOC's that are emitted from common, everyday household items.

References

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