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Case Study

Detecting the Smell of Roses

Released by:

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Job Number: J10662

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Background

William Shakespeare once wrote, "What's in a name? That which we call a rose by any other name would smell as sweet." Roses are well known for their odor, and are routinely used in perfumes, confectionaries, scented oils, and a myriad of applications calling for a pleasant odor. However, natural odors are typically highly complex, and while an odor or flavor may have a primary component, a multitude of secondary components typically complete the odor often at very low concentrations. All of these components are detectable by the human nose, which is capable of distinguishing up to 1 trillion smells[1] and can detect individual components in the range of parts per trillion concentration.[2] A classic example of complex flavor and odor is vanilla, whose principle component is vanillin[3]. Vanillin is inexpensive to synthesize and commonly used as artificial vanilla, which is notorious as a "cheap imitation" of the more complex genuine vanilla flavor. Genuine vanilla flavor (in the form of vanilla extract) is composed of more than 170 volatile components[4] highlighting the complex nature of both odors and flavors.

Given the complexity of odors, techniques that specialize in the sampling and analysis of volatile and semi-volatile components are necessary. Gas chromatography (GC) techniques are most commonly used in the analysis of volatile and semivolaitle organic compounds. A wide variety of GC variants such as dynamic headspace gas chromatography mass spectrometry (DHS-GCMS), headspace gas chromatography mass spectrometry (HS-GCMS), Twister Stir-bar Sorptive Extraction with Thermal Desorption GCMS (Twister-GCMS) all sample odors in different ways, resulting in a range of detectable components at various limits of detection.[5]

For example, DHS-GCMS collects components for analysis by blowing a large amount of nitrogen or other carrier gas over a sample of interest to collect all available volatile molecules while trapping the molecules on a sorbent to concentrate the molecules, thus increasing detection limit. A limitation therefore, is that not all molecules interact well with the sorbent and could be missed. HS-GCMS samples by heating up a sample and simply sampling a predetermined amount of air in the vial. This methodology is fairly fool-proof against missing major volatile components, but could easily miss trace level components. The combination of multiple GCMS techniques in this manner allows for the detection of components that might otherwise be left undetected.

[1] http://www.sciencemag.org/news/2014/03/human-nose-can-detect-trillion-smells
[2] L.J. van Gemert (2003) Flavour thresholds
[3] http://sitn.hms.harvard.edu/flash/2015/the-flavor-rundown-natural-vs-artificial-flavors/
[4] *J. Agric. Food Chem.*, 1992, 40 (10), pp 1922–1924
[5] https://jordilabs.com/lab-testing/analytical-techniques/mass-spectrometry/

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Client Name
Client Company
Client Address

P: Client Phone
E: Client Email

Dear Client,

Please find enclosed the test results for your samples described as:

1. Red Rose

The following tests were performed:

1. Headspace Gas Chromatography Mass Spectrometry (HSGCMS)
2. Dynamic Headspace Gas Chromatography Mass Spectrometry (DHSGCMS)
3. Gerstel Twister® - Desorption Mass Spectrometry (Twister)
4. Solid Phase Micro Extraction Desorption Mass Spectrometry (SPME)

Objective

The goal of this analysis was to determine the identity of the chemicals responsible for the smell of a rose.

Summary of Results

All techniques identified the presence of components such as 3,5-dimethoxy-toluene, theraspirane, caryophyllene (peppery smell) and α,β-dihydro-β-ionone. These possess floral, herbal, and floral smells respectively. Additional odorous components such as α and β pinene (pine smell) and vinyl anisole (floral smell) were detected by multiple, but not all, techniques. The variety and number of components detected demonstrate the complexity of natural odors as better than 13 well-known odiferous components were detected.

The different odor components observed by each technique demonstrates the power and utility of using multiple methods to screen a single problem as well as the complexity of a smell, even something so well known as the smell of a rose. As demonstrated, the smell of a rose contains not only floral smells, but diverse components that smell of pine, fruit, and even pepper; smells not usually associated with flowers.

Individual Test Results

A summary of the individual test results is provided below. All accompanying data, including spectra, has been included in the data section of this report.

GCMS Techniques

Sample Preparation

DHSGCMS

Individual petals (5) were plucked from a rose and sealed in a 20 ml headspace sampling vial for dynamic headspace analysis.

HSGCMS

Individual petals (3) were plucked from a rose and sealed in a 20 ml headspace sampling vial for headspace analysis.

Twister

Individual petals (3) were plucked from a rose and placed below a Twister sorption bar. The setup was gently heated overnight, and the Twister sorption bar was then analyzed in DMS mode and compared to a control Twister bar.

SPME

Individual petals (3) were plucked from a rose and placed below a solid phase microextraction (SPME) fiber. The setup was gently heated overnight, and the SPME fiber was then analyzed in DMS mode and compared to a control SPME fiber.

Results

It was found that the techniques utilized partially overlapped in what they were able to identify. All techniques identified 3,5-dimethoxy-toluene as the most abundant component, strongly suggesting this floral-smelling chemical is the major ingredient in the sweet smell of a rose. Each technique also identified theraspirane, which possesses an herbal odor, Caryophyllene which has a sweet odor and α,β-Dihydro-β-ionone that also has a floral smell. Vinyl anisole was detected only by HSGCMS and DHSGCMS. DHSGCMS, also detected α and β pinene, both of which produce a pine odor. SPME was the most effective method for detecting Phenyl ethyl alcohol and its analogs. In general DHSGCMS was found to be the most sensitive method and showed the largest number of components. **Table 1** shows a complete list of the components observed. **Figure 1** to **Figure 4** show the resulting chromatograms from each methodology.

Table 1 Summary of *Red Rose* Analysis						
Possible ID	*CAS*	*DHS-GCMS*	*HS-GCMS*	*SPME*	*Twister*	*Comment*
α-pinene	7785-70-8	x				pine odor
β-pinene	18172-67-3	x				pine odor
Limonene	138-86-3	x				citrus
Phenyl ethyl alcohol	60-12-8	x		x	x	floral odor
Acetic acid phenylethylester	103-45-7	x		x		floral odor
3-hexene-1-ol-acetate	3681-71-8	x		x	x	sharp fruity-green
Vinyl anisole	-	x	x			sweet odor
Caryophyllene	87-44-5	x	x	x	x	spicy odor
1-ethenyl-4-methoxybenzene	637-69-4	x				floral odor
3,5-Dimethoxytoluene	**4179-19-5**	**x**	**x**	**x**	**x**	**floral odor**
1,3,5-trimethoxybenzene	621-23-8	x		x	x	floral odor
Theaspirane	36431-72-8	x	**x**	**x**	**x**	herbal odor
α,β-Dihydro-β-ionone	17283-81-7	x	**x**	**x**	**x**	floral odor

X – Detected; Bold, main component

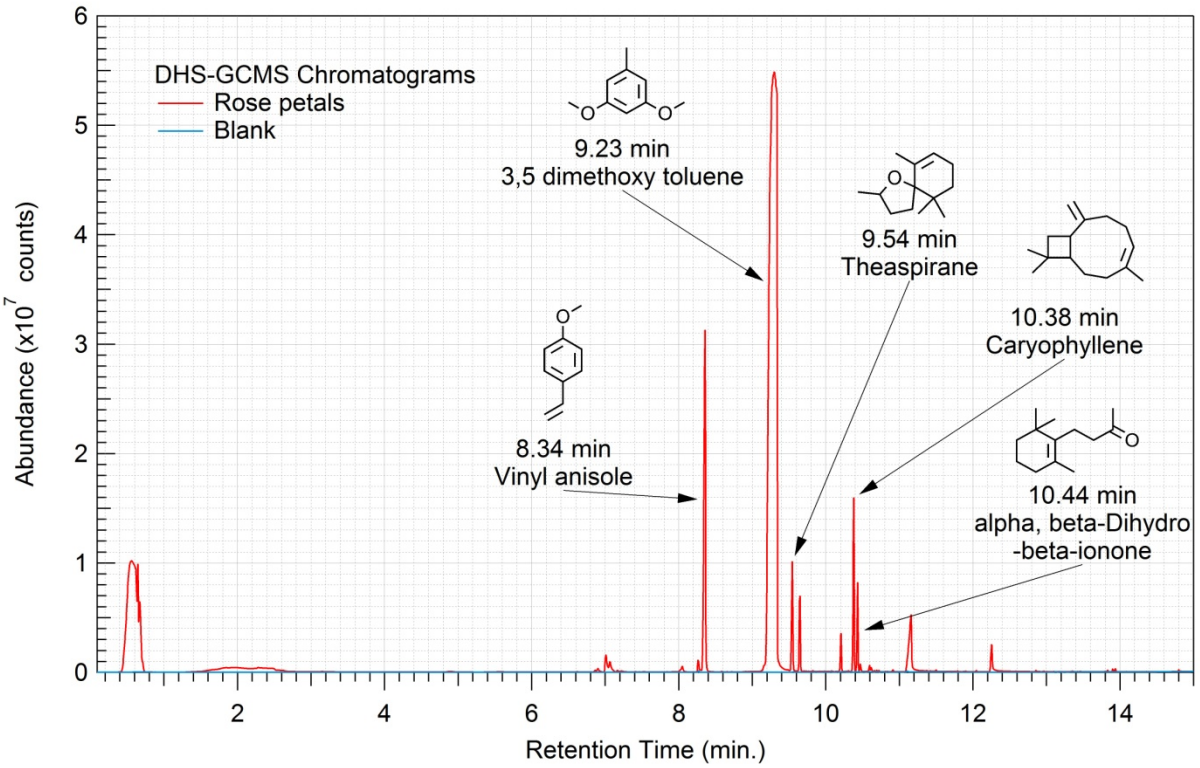

Figure 1 - Overlay of DHSGCMS chromatograms.

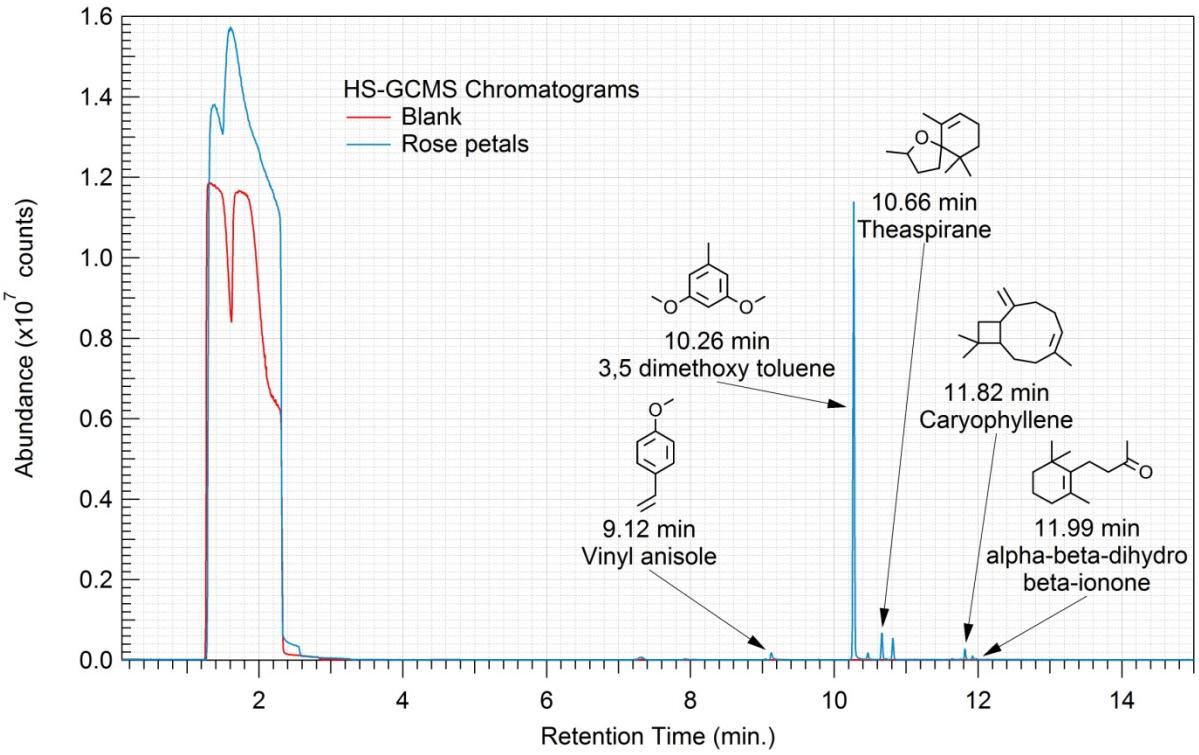

Figure 2 - Overlay of HSGCMS chromatograms.

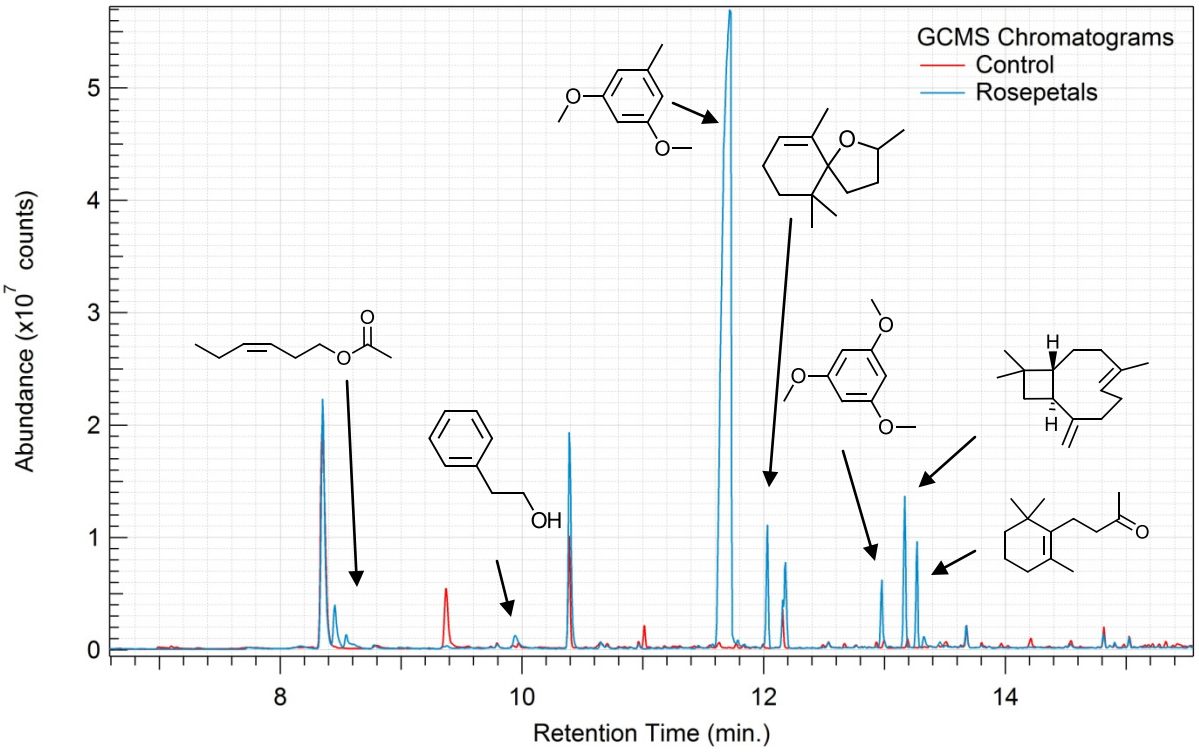

Figure 3 - Overlay of Twister chromatograms.

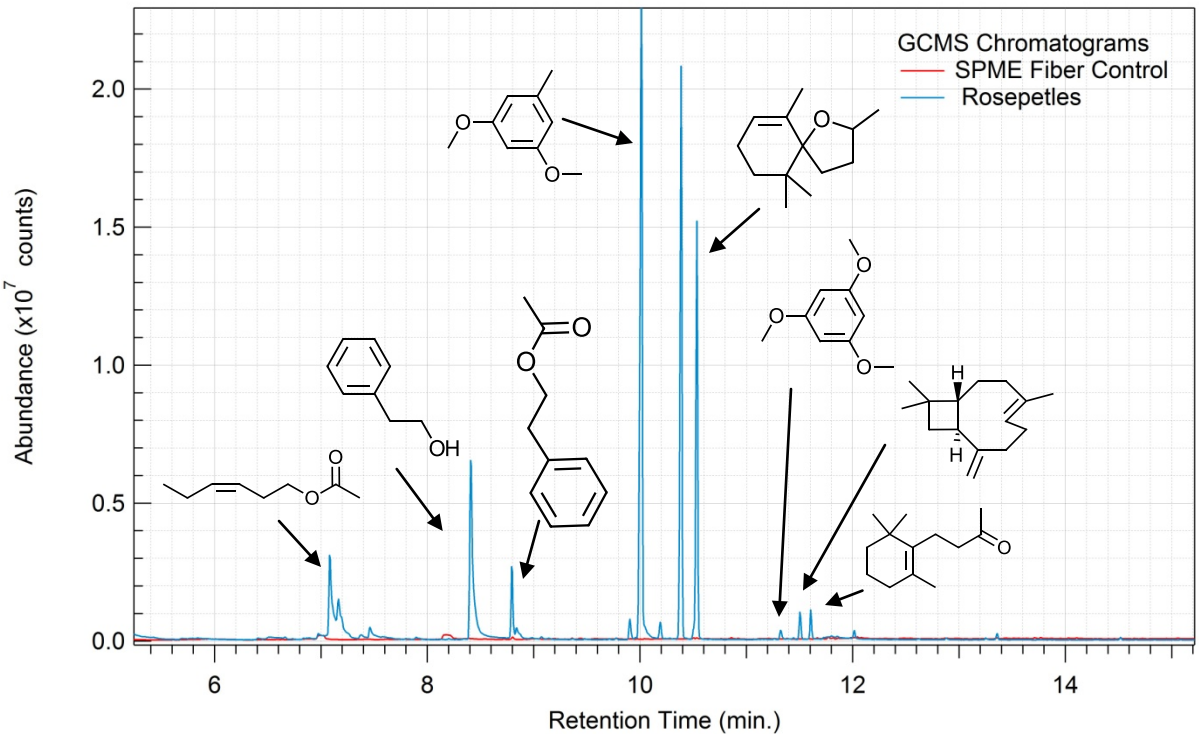

Figure 4 - Overlay of SPME chromatograms.

Analysis Conditions

Gerstel Dynamic Headspace/Thermal Desorption GCMS

The samples were analyzed using a Gerstel Thermal Desorption Unit, Gerstel Cooled Injection System, Agilent 7890A gas chromatograph and a 5975C mass selective detector using gas injection. Data acquisition was accomplished using Chemstation software. Sample peaks were compared with over 796,613 reference compounds using the NIST/EPA/NIH mass spectral search program.

Closing Comments

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Jordi Labs specializes in polymer testing and has 30 years experience doing complete polymer deformulations. We are one of the few labs in the country specialized in this type of testing. We will work closely with you to help explain your test results and solve your problem. We appreciate your business and are looking forward to speaking with you concerning these results.

Sincerely,

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