Application Note

Instrument: Pegasus® BT

Nutmeg Essential Oil: Sensory Directed Analysis of GC-MS Data via Olfactory Detection

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Key Words: GC, Retention Index, RI, MS, TOFMS, Deconvolution, Olfactory Detection, GCO, Phaser, Food Flavor Fragrance, Essential Oil, Nutmeg, Characterization, Sensory Directed Analysis

Introduction

Characterization of food, flavor, or fragrance samples often includes the objective of identifying the individual chemical components of a sample and then linking these features to their aroma or sensory impact. Gas chromatography (GC) coupled to mass spectrometry (MS) and an olfactory detection port is a powerful analytical platform that is well-suited to meet these analytical objectives. Each component of the instrument plays an important and complementary role in separating, identifying, and determining the sensory impact of the individual analytes within these complex food, flavor, and fragrance samples. The individual components are separated primarily by GC, but MS (in particular, time-of-flight (TOF)-MS) detection adds to the chromatographic resolution with deconvolution capabilities that can often mathematically resolve instances of chromatographic coelutions. The identifications of these individual analytes can then be determined by matching the observed spectral patterns to mass spectral library databases. This MS identification can be supported with elution order information from the GC separation by utilizing Retention Index (RI) matching of observed RI information to library database RI information. These capabilities allow for more individual analytes to be isolated and identified. Finally, olfactory detection offers a direct connection to the sensory impact of these isolated and identified features. These pieces can be combined by splitting a single GC separation to both MS and olfactory detection, providing all of this information in a single analysis. This type of characterization can be beneficial in quality control (QC) applications, can drive product development, and can lead to a better understanding of a sample. In this application note, we combine LECO's Pegasus® GC-MS with GL Science's Phaser Pro for olfactory detection to characterize a nutmeg essential oil and understand the specific analytes most responsible for the distinct nutmeg aromas in the sample.

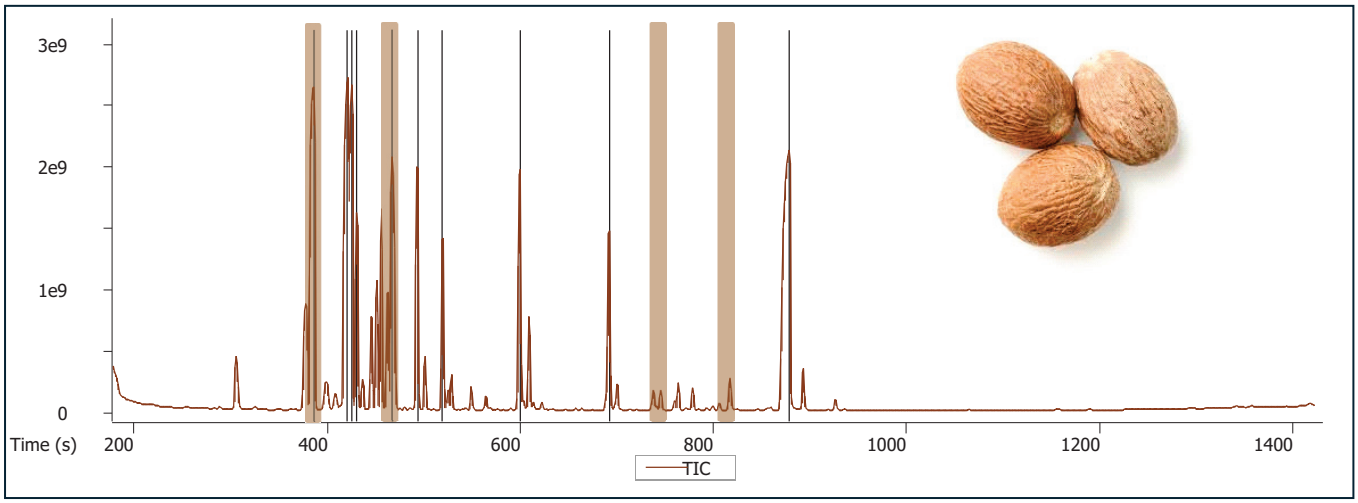

Figure 1. GC-MS-O data for representative nutmeg sample. Ten vertical black line peak markers indicate features with the highest S/N. Shaded brown markers indicate distinct nutmeg notes in the olfactory data.

Experimental

A nutmeg essential oil was diluted to 1% in acetone and analyzed with the instrument conditions listed in Table 1. An alkane standard was also analyzed with the same conditions for RI determinations.

Table 1. Instrument (*Pegasus* BT) Conditions

Auto Sampler	**LECO L-PAL 3 Autosampler**
Injection	1 uL
Gas Chromatograph	**LECO GC**
Inlet	250 °C
Carrier Gas	He @ 1.4 mL/min
Columns	HP-5ms, 30 m x 0.25 mm i.d. x 0.25 µm coating
Temperature Program	40 °C to 280 °C at 10 °C/min
Transfer Line	280 °C
Mass Spectrometer	**LECO *Pegasus* BT**
Ion Source Temperature	250 °C
Mass Range	35-500 m/z
Acquisition Rate	10 spectra/s
Olfactory	**GL Science Phaser Pro**

Results and Discussion

A nutmeg essential oil was analyzed with the GC-MS-O analytical platform and a representative chromatogram is shown in Figure 1. As described, one of the analysis goals was characterization to understand the specific analytes most responsible for the distinct aroma notes. With GC-MS, this type of characterization often begins with review of the more intense peaks in a sample and then extends through the full peak list. The ten analytes with the largest S/N are indicated with markers on Figure 1 and described in Table 2. Like all features on the peak list, the analytes are tentatively identified by matching the observed spectral data to library databases (with similarity scores indicated) and also by matching the observed retention index information (RI) to library database values, when available. While the intense features are a good place to start, the review must also extend to lower level features because odor thresholds are at vastly different concentrations for different analytes. The largest peaks do not always indicate the most impactful contributors to the sensory profile and small peak area does not necessarily indicate an insignificant impact on the aroma. With GC-MS, the full sample's peak list of tentatively identified features can be investigated, and the aroma properties can be determined from literature information to make these sensory connections.

Table 2. Peaks with highest S/N in the nutmeg sample

Analyte	Similarity	CAS	tR	RI	RI (Library)
α-pinene	945	80-56-8	386.454	940.7	937 ± 3(982)
sabinene	869	3387-41-5	420.205	980.5	974 ± 2(618)
β-pinene	937	127-91-3	425.597	986.9	979 ± 2(848)
β-myrcene	923	123-35-3	430.827	993.1	991 ± 2(838)
limonene	934	138-86-3	467.844	1035.4	1030 ± 2(986)
γ-terpinene	942	99-85-4	493.468	1064.5	1060 ± 2(734)
terpinolen	910	586-62-9	519.653	1094.2	1088 ± 2(613)
L-terpinen-4-ol	837	20126-76-5	600.006	1188.2	1182 ± 0(3)
safrole	926	94-59-7	692.082	1302.6	1287 ± 2(50)
myristicine	913	607-91-0	878.958	1544.1	1520 ± 4(52)

With GC-MS-O, however, the olfactory data provides a direct indication of sensory impact and guides the analysis and review of the GC-MS data. Instead of working through the entire peak list, a focused review of regions of the chromatogram can be performed. For this nutmeg essential oil sample, for example, there were four distinct and characteristic "nutmeg" odor spots in the chromatogram, as indicated on Figure 1. These four spots were explored and are described in Figures 2-5.

The first distinct nutmeg note did correspond to one of the top 10 S/N features in the sample, as shown in Figure 1. The olfactory data pointed to a location in the chromatogram that was determined to be alpha-pinene, as described in Table 2 and shown in Figure 2.

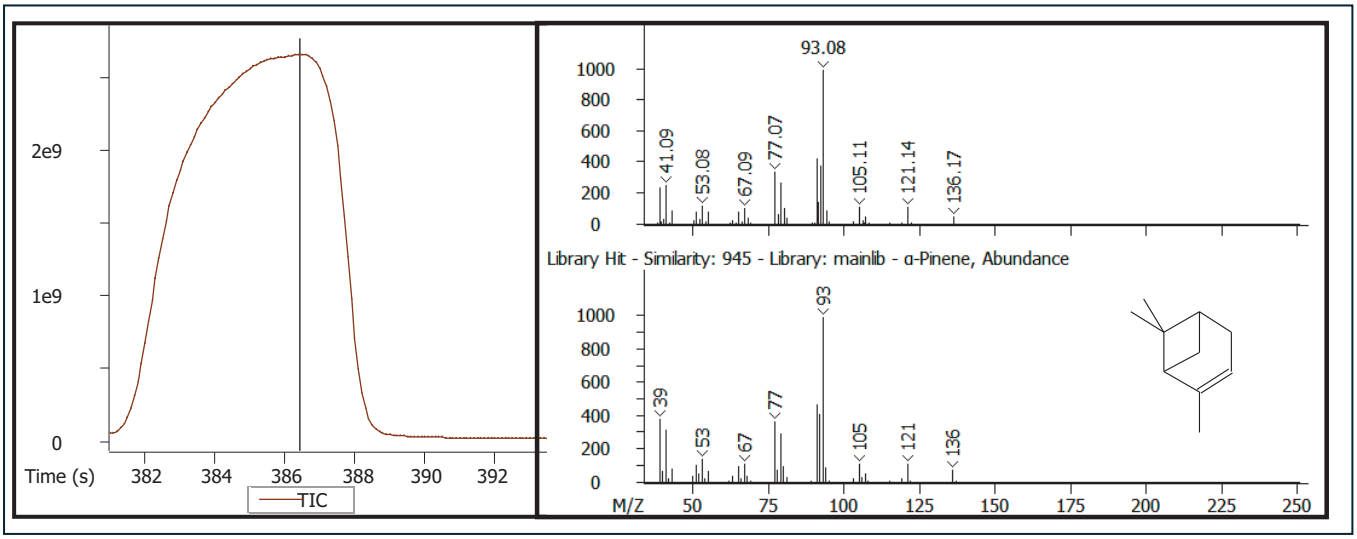

Figure 2. One of the distinct nutmeg aroma notes from the GC-O data corresponded to the alpha-pinene, which was also one of the features with a large S/N in the sample.

The second distinct nutmeg note initially appeared to correspond to a top 10 S/N feature but was actually better attributed to a smaller coeluting feature. In this case, the olfactory data pointed to a region of the chromatogram with more complexity. As shown in Figure 3, there are 3 coeluting features that elute in this chromatographic spot. Deconvolution effectively separated these, provided spectral data for each, and indicated m/z for observing each chromatographic profile. Two smaller features were determined in addition to the largest peak, limonene. The odor description of this region was also more complex with hints of citrus and camphor being detected along with the nutmeg notes. These odor notes can be connected to the deconvoluted peaks with citrus coming from limonene, camphor from eucalyptol, and the nutmeg from the β -phellandrene.

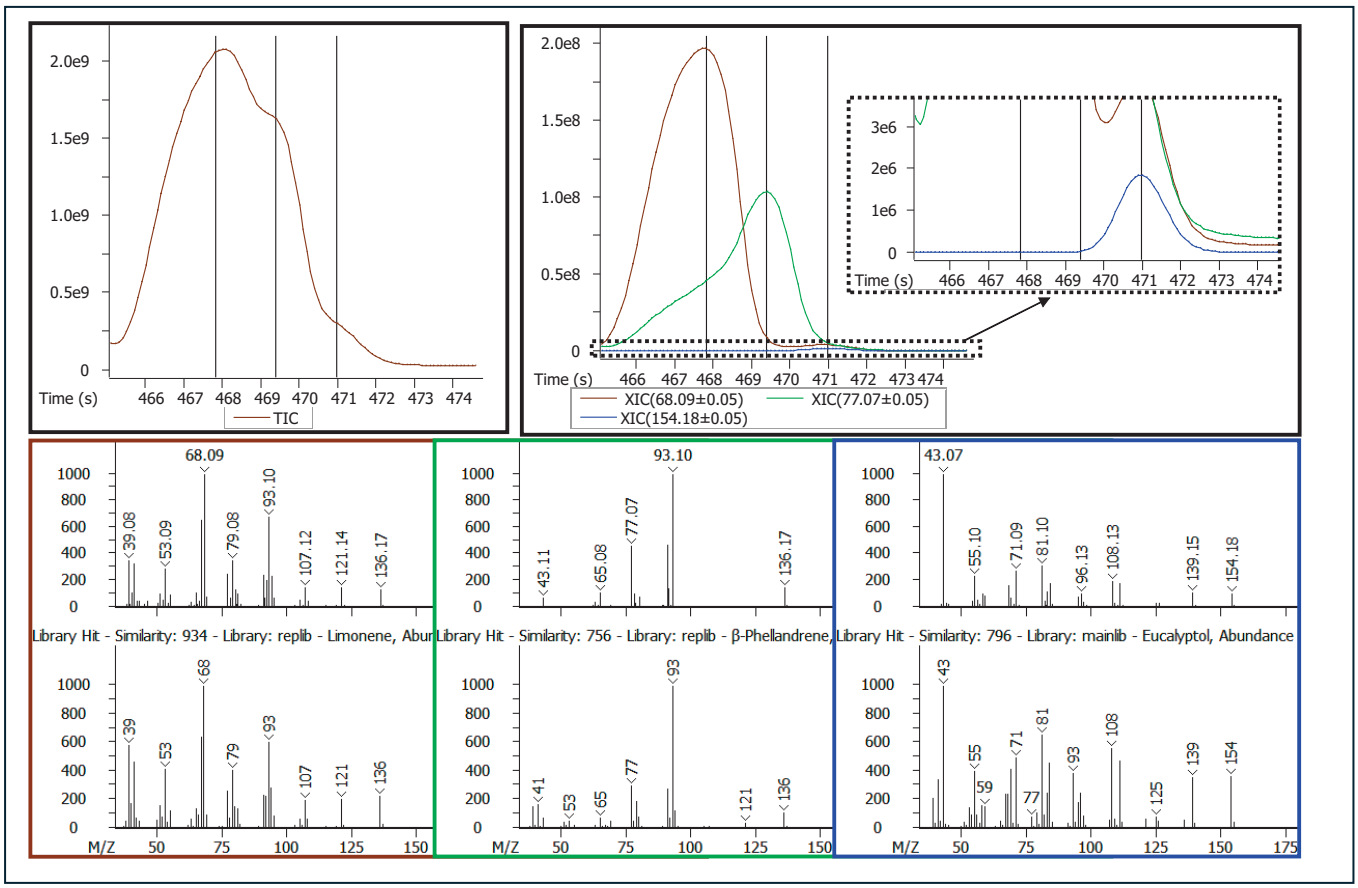

Figure 3. One of the distinct nutmeg aroma notes from the GC-O data corresponded to a coelution in the GC-MS data. Deconvolution effectively determined the individual analytes and the aroma descriptors can be sorted between the three features.

The next two nutmeg aroma notes, shown in Figures 4 and 5, are associated with analytes that have much lower peak areas and S/N. Both of these were determined with review of the GC-MS peak table information extending past the top 10 S/N peaks, but the olfactory data efficiently focused the analysis on these important aroma contributors.

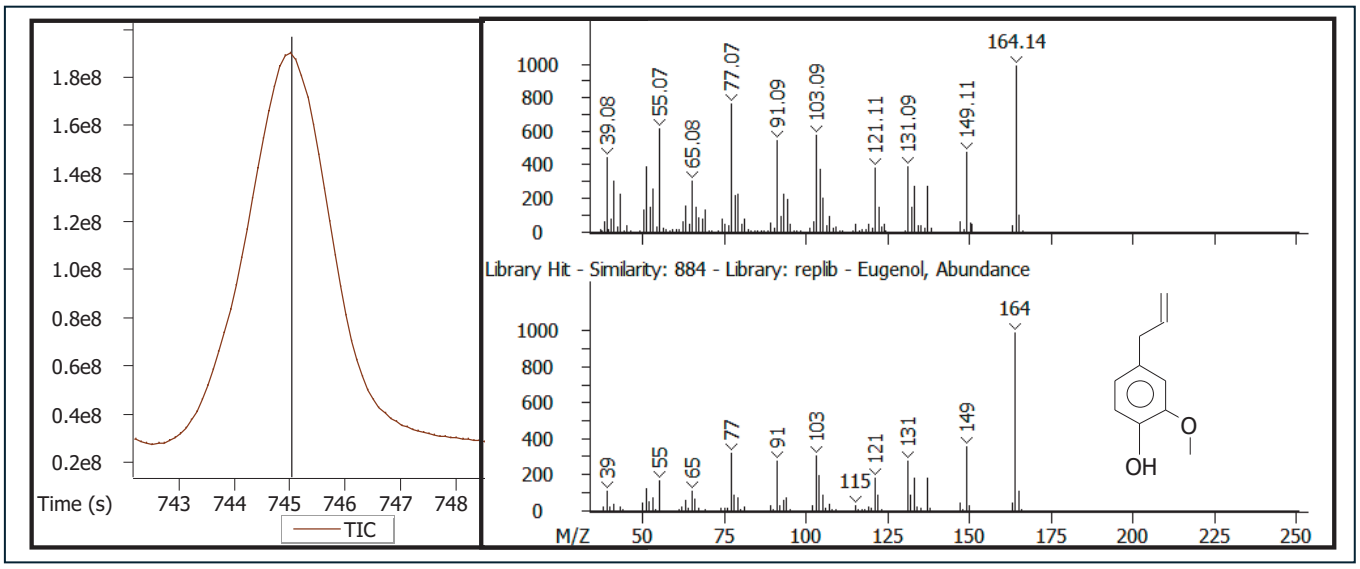

Figure 4. One of the distinct nutmeg aroma notes from the GC-O data corresponded to the eugenol.

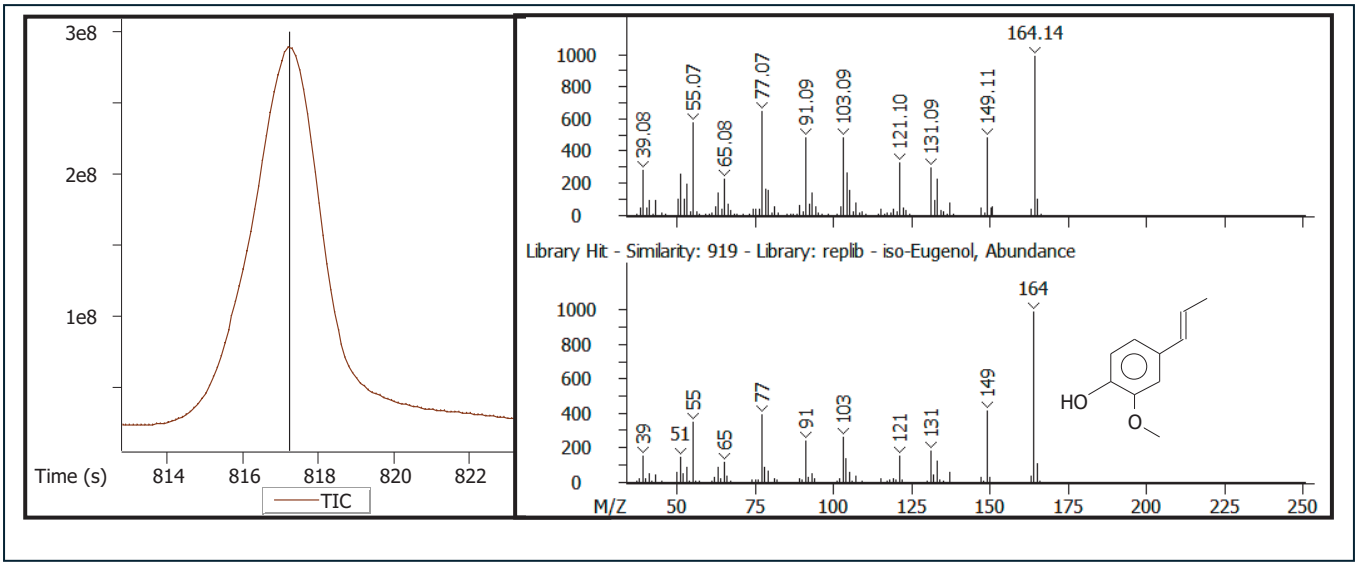

Figure 5. One of the distinct nutmeg aroma notes from the GC-O data corresponded to the iso-eugenol.

A focused list of the 4 analytes with characteristic nutmeg notes is shown in Table 3. As in Table 2, the identifications are supported with both spectral matching and RI matching. This characterization of the nutmeg essential oil was efficiently achieved by using the olfactory data to direct the review of the GC-MS data.

Table 3. Peaks with distinct nutmeg aromas

Analyte	Similarity	CAS	tR (s)	RI (Observed)	RI (Library)
α-pinene	945	80-56-8	386.454	940.7	937 ± 3(982)
β-phellandrene	756	555-10-2	469.404	1037.1	1031 ± 2(280)
eugenol	884	97-53-0	745.028	1366.9	1358 ± 3(366)
iso-eugenol	919	97-54-1	817.255	1460.1	1450 ± 15(40)

Conclusion

In this application, the combination of a GC separation with MS and olfactory detection provided a powerful and efficient analytical platform to isolate individual analytes, identify those isolated analytes, and connect them to their sensory impacts. The olfactory data pointed to features with high S/N, features with lower S/N, and regions of the chromatogram with coelution and overlap. In each case, the GC separation and full m/z range TOFMS data were crucial for determining the identification of the feature responsible for the characteristic aroma. This led to a characterization of the most aroma impacting components of a nutmeg essential oil.

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