Application Note

Instrument: Pegasus® BTX

EMPOWERING RESULTS
Elemental Analysis | GC Mass Spectrometry | Metallography

Ginger Oil Analysis and Grade Differentiation with Pegasus BTX Using Hydrogen Carrier Gas

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Introduction

Ginger essential oil (ginger EO) derives from the rhizome of the *Zingiber officinale* plant and has been valued for centuries for its aromatic, medicinal, and culinary properties. With a rich history dating back to ancient China and India, ginger oil has been treasured for its versatile applications and therapeutic benefits.

Ginger EO is renowned for its warming, spicy aroma and is commonly used in aromatherapy to alleviate nausea, improve digestion, and relieve muscle pain. In culinary applications, it adds depth and complexity to dishes, especially in Asian and Indian cuisines. Additionally, ginger oil is valued in skincare for its anti-inflammatory and antioxidant properties, promoting healthy skin and hair.

The chemical composition of ginger oil is complex, comprising various bioactive compounds such as gingerol, shogaol, and zingiberene. Gas Chromatography-Mass Spectrometry (GC-MS) analysis is commonly employed to assess the quality and purity of ginger oil, ensuring it meets industry standards and regulatory requirements.

Differentiating essential oil grades is crucial for the perfumery, flavor, and food industries due to several reasons related to quality, consistency, safety, and overall product effectiveness. As an example, high quality EOs are often pure, unadulterated, and extracted using meticulous methods to preserve their natural properties, while the low-quality ones may be diluted, adulterated, or extracted using less refined methods, potentially compromising their scent and taste profiles and therapeutic qualities.

In this context, perfumery industries often need to be able to assess EO quality parameters in a fast and reliable manner.

This application note describes the conversion of a Helium-based method to a fast and reliable Hydrogen-based one using LECO's novel *Pegasus* BTX GC-TOFMS instrument. This mass spectrometer offers a wide dynamic range and sensitivity at low femtogram levels, allowing for the simultaneous detection of both abundant and trace components without losing any detail. Additionally, ChromaTOF® Sync software was used to distinguish between different grades of ginger EO and identify markers for each of them.

Experimental

Four ginger EO grades were analyzed. The samples were diluted to 0.1 % (v/v) in hexane prior to injection. n-Alkane standards (C7-C30) were diluted to 10 mg/L in hexane and analyzed under the same chromatographic conditions for calculation of linear retention indices (RIs). Table 1 provides the instrumental parameters applied including the gas chromatographic parameters used to establish the conventional Helium (He) and the optimized Hydrogen (H_2) methods.

Table 1: Instrumental parameters applied for the He- and H_2-based methods.

GC	**Agilent 8890**	
Injector	1 µl split 200:1 at 250 °C	
Carrier Gas	He 1.4 mL/min	H_2 1.26 mL/min
Column	Rxi-5Sil MS 30 m x 0.25 mm ID x 0.25 µm f.t.	Rxi-5Sil MS 20 m x 0.18 mm ID x 0.18 µm f.t.
Oven Program	50 °C (0.1 min); ramp: 8.5 °C/min to 280 °C (5 min)	50 °C (0.1 min); ramp: 20 °C/min to 280 °C (5 min)
Transfer Line	280 °C	280 °C
MS	**LECO *Pegasus* BTX TOFMS**	
Ion Source Temp	250 °C	
Mass Range	40 to 400 m/z	
Acquisition Rate	10 spectra/s	20 spectra/s

Results

One of the main reasons for the adoption of H_2 carrier gas in gas chromatography is its efficiency at higher linear velocities, compared to He. This is due to the lower viscosity and hence higher diffusivity of the first compared to He. Consequently, H_2-supplied methods result in faster analysis times with comparable chromatographic resolution values. Figure 1 shows the comparison between a He method and the corresponding translated H_2 method. The method translation approach used is illustrated in the Application Note 203-821-651 and uses Restek`s EZGC method translator tool. Based on the R.T. of Gingerol (CAS: 23513-14-6), the usage of H_2 carrier gas reduced the analysis time by about 60%, compared with the initial, He-supplied method.

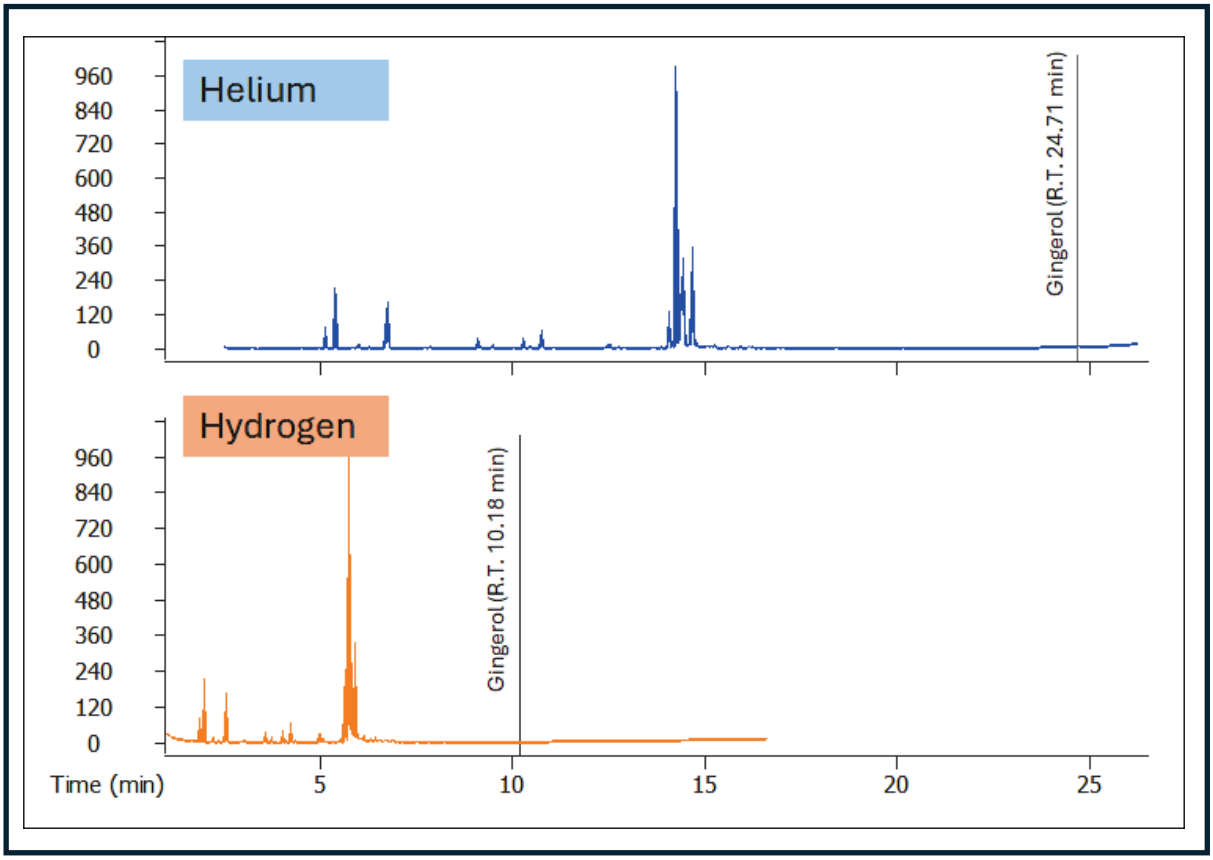

Figure 1. Comparison between a conventional He-optimized GC-MS method (blue trace) and the H_2-translated one (orange trace).

Figure 2 demonstrates how the chromatographic resolution is preserved and even improved when the chromatographic method is switched to H_2 carrier gas. On the top (light grey), the resolution between Bornyl acetate (CAS: 76-49-3) and Undecan-2-one (CAS: 112-12-9) is shown, while on the bottom (dark blue), the resolution between (E)-β-Farnesene (CAS: 18794-84-8) and Sesquisabinene (CAS: 58319-04-3) is highlighted. In both cases, the H_2-supplied method showed even better chromatographic performance compared to the He-supplied one.

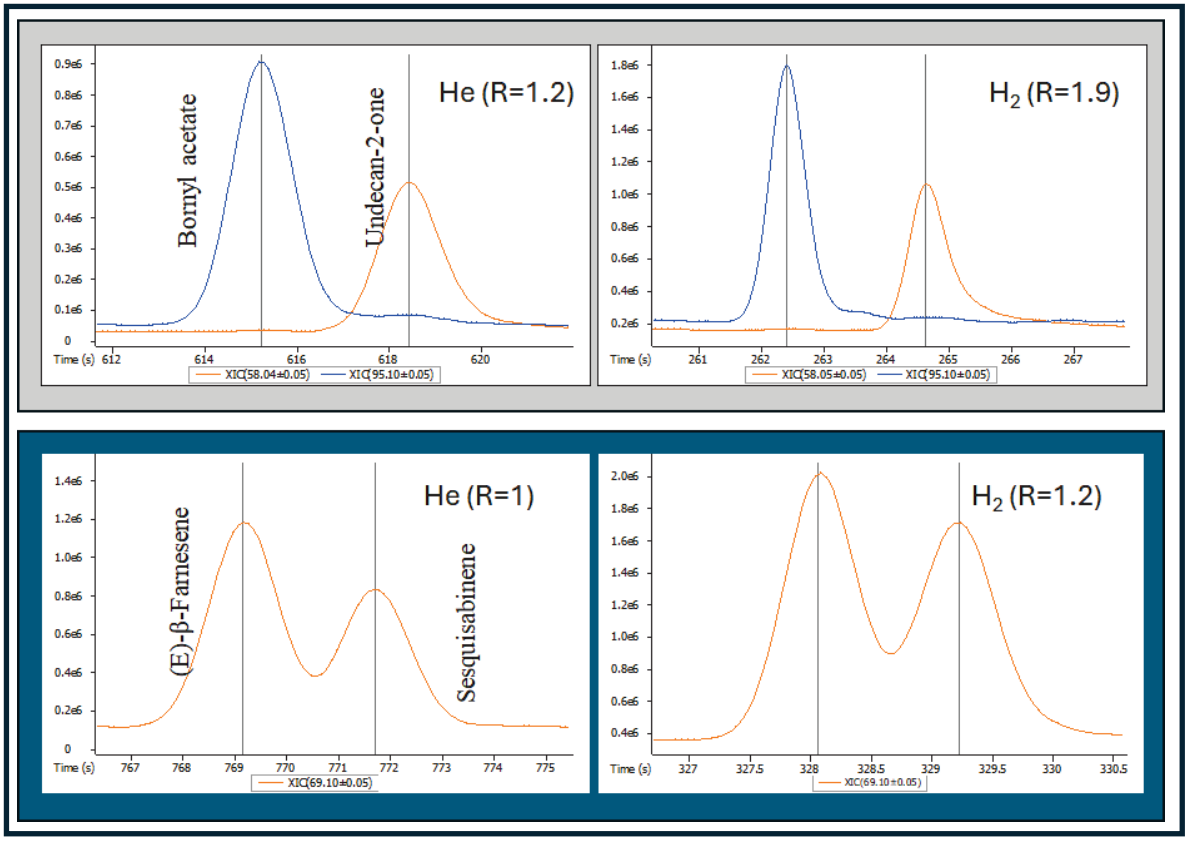

Figure 2. Chromatographic resolution comparison between He- and H_2-supplied GC methods.

Table 2 highlights the performances of the new TOF mass spectrometer in terms of spectral quality when using He or H_2 carrier gas. Several representative compounds were selected, and their library scores were compared using the same mass spectral library, namely the NIST MS database (Table 2). Historically, the spectral quality of the compounds ionized in the presence of H_2 carrier gas was poorer compared to the quality attained with He carrier gas, mostly due to the components reactivity in the ionization source which can lead to distorted spectra with lower similarity and/or misidentifications due to chemical reactions generating entirely different substances. Here, we show how the library scores are highly comparable, allowing confident identifications with either carrier gas.

Table 2. He vs H_2 mass spectral quality comparison.

Name	CAS	Similarity He	Δ RI He	Similarity H2	Δ RI H2
α-Pinene	80-56-8	935	1	940	3
Camphene	79-92-5	948	0	926	0
Geraniol	106-24-1	937	0	871	5
Geranial	141-27-5	928	3	805	2
Copaene	3856-25-5	848	7	920	5
β-Elemene	515-13-9	913	8	896	2
Curcumene <AR>	644-30-4	932	5	919	0
α-Zingiberene	495-60-3	908	6	868	2
β-Bisabolene	495-61-4	952	4	938	1
β-Sesquiphellandrene	20307-83-9	921	6	885	3
	Average	**922**	**4**	**897**	**2.3**

Figure 3 presents the mass spectral fragmentation of Gingerol (CAS: 23513-14-6) when using He and H_2 carrier gas together with the NIST library spectrum as a reference. As can be seen, no significant differences are observed in the fragmentation profiles obtained with He and H_2 carrier gasses. This is due to the special open design of LECO's StayClean® Ion Source, which does not affect the ionization in the presence of H_2 carrier gas. For more information regarding the ionization source, refer to Application Note 203-821-569.

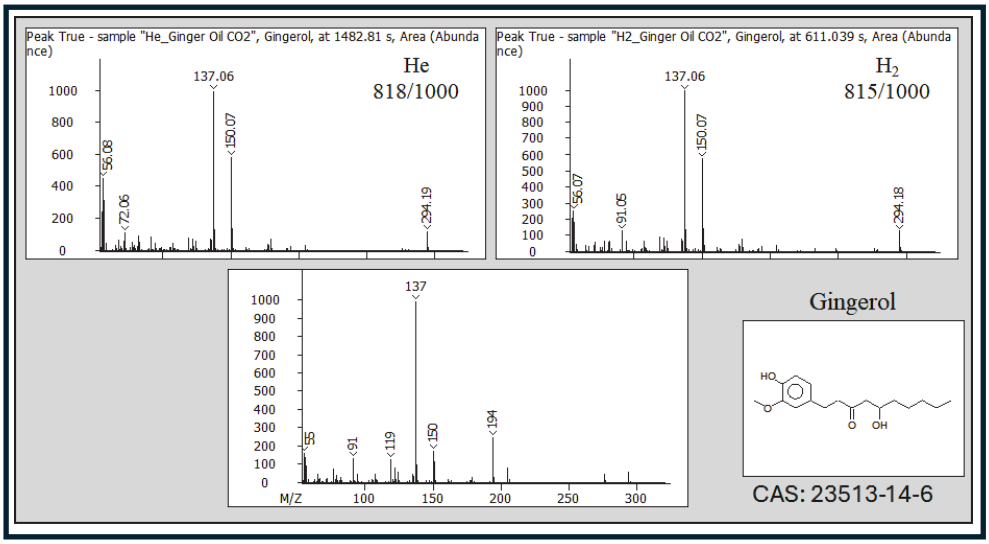

Figure 3. Example of EI MS fragmentation using He and H_2 carrier gas.

Essential oil grades are critically important for the essential oil industry due to their significant impact on the quality, consistency, and safety of the final fragrance products. Here, we applied *ChromaTOF* Sync software to easily determine markers differentiating the four EO grades measured. *ChromaTOF* Sync is a data processing tool that compiles peak information through sets of samples by performing automated peak finding and deconvolution of the high quality TOFMS data on the set of samples.

Figure 4 displays the results of the Principal Component Analysis (PCA) and a heat map displaying the details for some of the differentiating components.

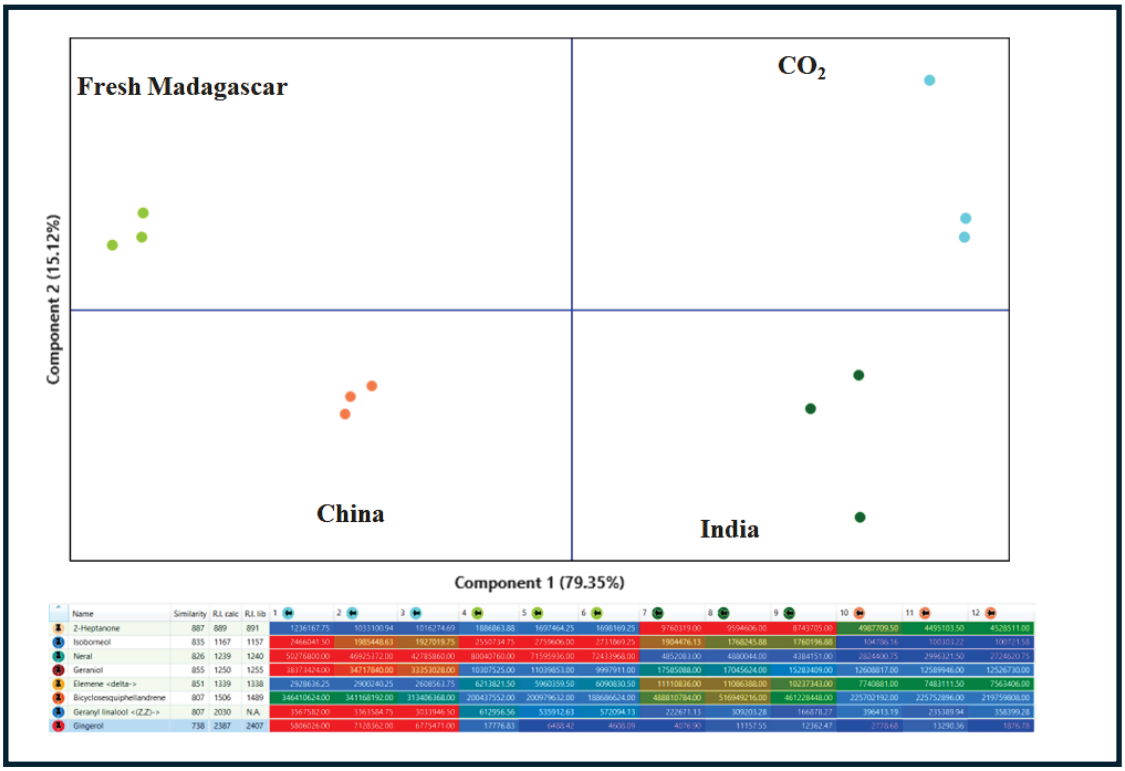

Figure 4. Principal Component Analysis (PCA) scores plot (top) displaying clustering of the Ginger EO samples according to their origin. The heatmap (bottom) allows one to quickly highlight differences across the samples set.

Figure 5 (a to c) shows three examples of differentiating components along with mass spectral details and odor characteristics. Their identification was obtained by searching the deconvoluted mass spectra within *ChromaTOF* Sync against the NIST MS database with RI calculation and filtration. The ease of data review makes *ChromaTOF* Sync a useful tool for investigating EO compositions and differentiating EO grades. Moreover, the link to the original data is never lost as the user can always interrogate back the data and modify parameters such as integration, hit assignment, and so on.

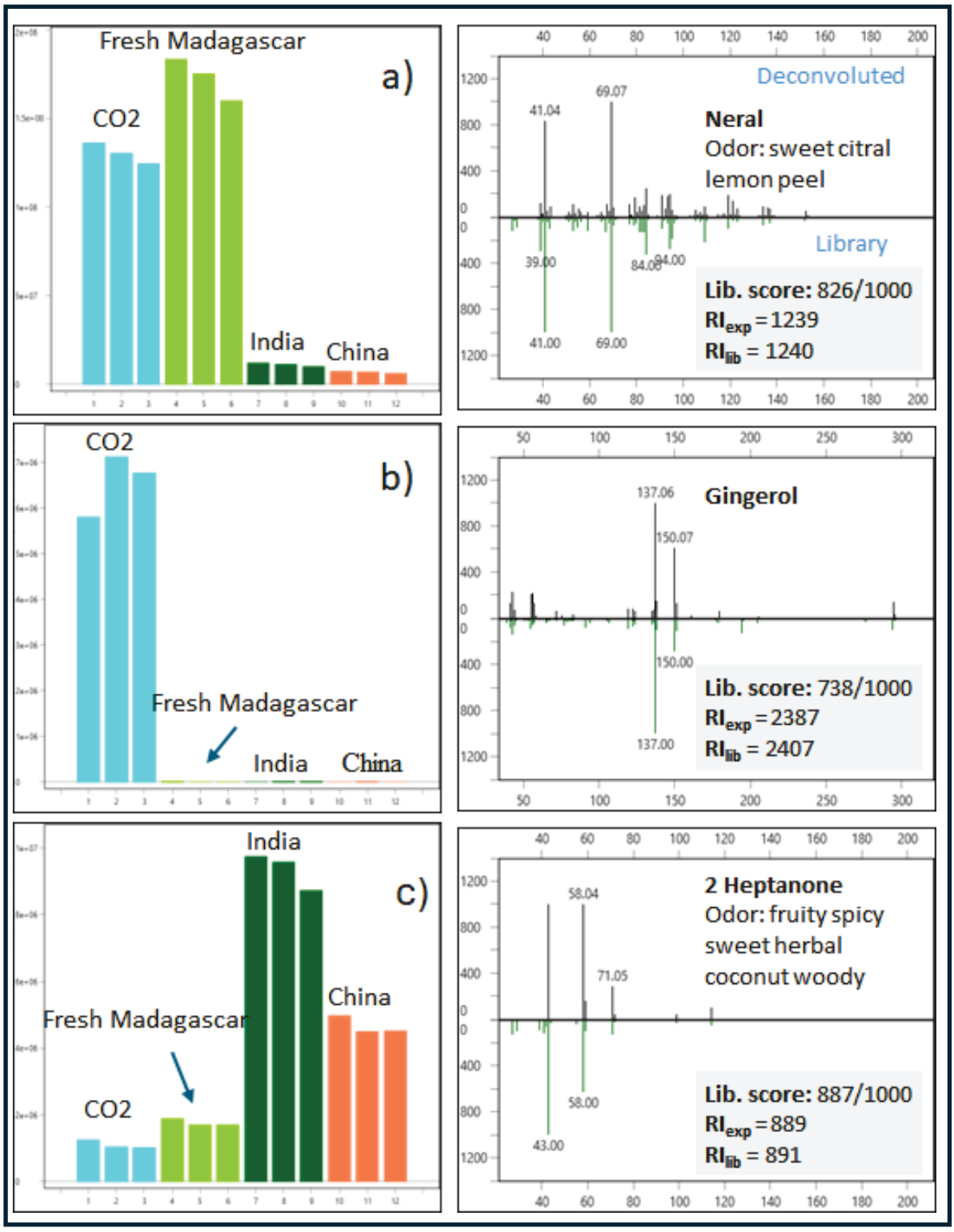

Figure 5 (a to c). Three examples of marker components, respective the different ginger oil grades, with mass spectra details and odor characteristics.

The combination of high sensitivity and full mass range non-target data provided by the BTX can also allow for discovering more important and differentiating analytes, even when present at trace levels. *ChromaTOF* software and deconvolution are also crucial for uncovering these features from coelutions and matrix interferences. For example, a deeper investigation into the CO_2 ginger oil grade revealed trace level presence of two aroma active species (Figure 6). Germacrene b and Beta-Calacorene are coeluting and the TIC mass spectra (6a) would be difficult to library match due to the cross-mixture of ions present. However, deconvolution generated clear mass spectra showing two components (6b,c) and enabled library matching with higher confidence.

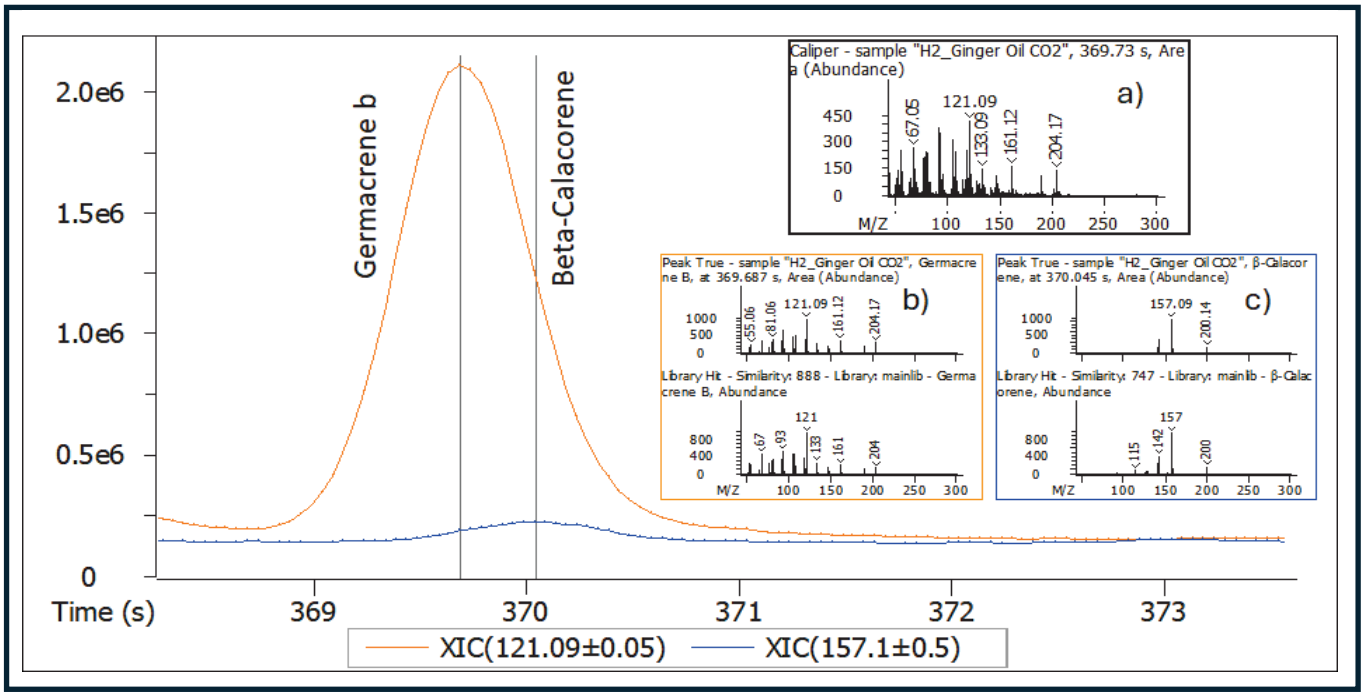

Figure 6. Detection, deconvolution, and identification of 2 coeluting, trace level components.

Conclusions

This application note demonstrates how the *Pegasus* BTX GC-TOFMS is an excellent solution for the rapid analysis of EOs using H_2 carrier gas. The results indicate that neither the chromatographic performance nor the mass spectral quality are compromised by using H_2. A 60% reduction in runtime was achieved, leading to improved daily throughput. Additionally, the *ChromaTOF* Sync software tool effectively clustered the samples based on their origin and quality, enabling the discovery of characteristic features that differentiate them. The output information received from *ChromaTOF* Sync allows for more informed usage of raw materials in creating new fragrances and enhances confidence in analyzing routine samples.

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