

Characterization of Fruit Tea Beverages with the Pegasus® BT

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1. Introduction

LECO's *Pegasus* BT is well suited for both targeted screening and non-targeted characterization of food and beverage samples. Here, fruit flavored teas (diet and regular) were characterized and compared in a non-targeted way. HS-SPME was used to collect the volatile and semi-volatile analytes that likely contribute to the aroma and flavor of the beverages. These analytes were subsequently separated and detected with GC-TOFMS. Deconvolution of the full m/z range data offers an additional level of information with mathematical separation to distinguish chromatographic coelutions. These aspects of the analysis provided the ability to identify and quantify numerous individual analytes and discover more about these samples.

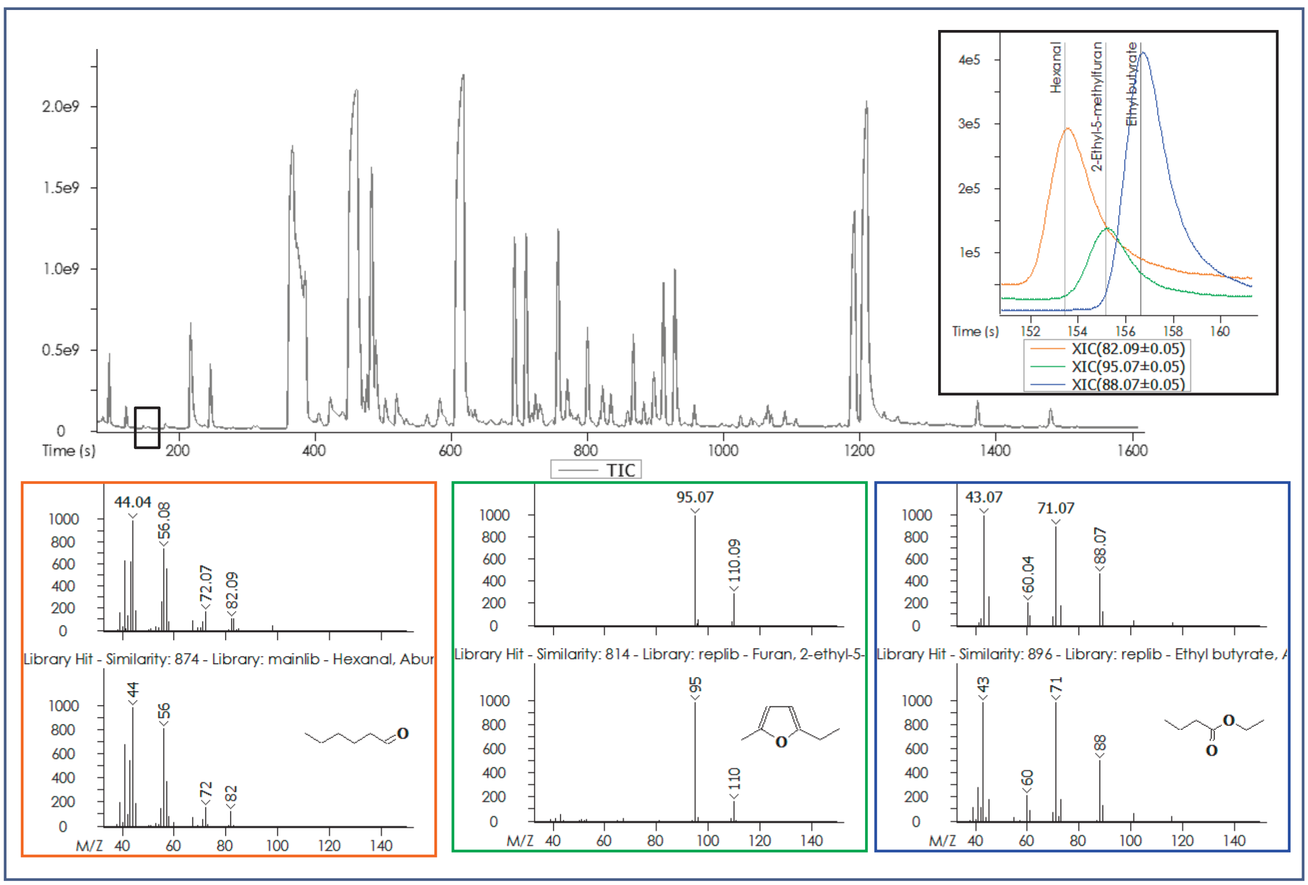

Figure 1. TIC chromatogram for a diet fruit tea sample is shown. Hundreds of analytes can be separated with a combination of both chromatography and deconvolution. Three analytes that chromatographically overlap are shown here. Deconvolution provided relative quantification and tentatively identified each analyte. Hexanal has a green odor and is known to be present in tea; 2-ethyl-5-methyl furan has a fresh and gassy odor; and ethyl butyrate is observed in a variety of different fruits and has a fruity odor.

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2. Experimental

Commercially available fruit flavored tea beverages (regular and diet) were purchased and prepared for analysis. Five mL of tea was added to a 20 mL HS-SPME vial which was then sealed with a septum cap. The samples were incubated for five minutes and extracted for 30 minutes at 50 °C with a 2 cm DVB/CAR/PDMS fiber (Sigma Aldrich). Instrument conditions are listed in Table 1.

Table 1. GC-TOFMS (*Pegasus* BT) Conditions

Gas Chromatograph	**Agilent 7890 with LECO L-PAL3 Autosampler**
Injection	2 min fiber desorption with inlet @ 250°C, splitless
Carrier Gas	He @ 1 mL/min
Column	Rxi-5ms, 30 m x 0.25 mm i.d. x 0.25 µm coating (Restek)
Oven Program	2 min at 40°C, ramp 5°C/min to 200°C, ramp 10°C/min to 300°C hold 1 min
Transfer Line	250°C
Mass Spectrometer	**LECO *Pegasus* BT**
Ion Source Temperature	250°C
Mass Range	33-650 m/z
Acquisition Rate	10 spectra/s

3. Results and Discussion

The *Pegasus* BT provides excellent characterization of the fruit tea samples. In Figure 1, a TIC chromatogram for the diet sample is shown. Hundreds of analytes were detected with this analytical approach. The addition of deconvolution to mathematically separate chromatographic coelutions, which are common in complex samples even with good GC separations, adds another important benefit to these analyses. Deconvolution determines pure spectral information for analytes that can be tentatively identified through spectral matching to library databases. Coelution examples are highlighted in Figure 1 where three flavor analytes from tea and fruit are separated from each other, and in Figure 2 where a terpene is mathematically separated from a fruity ester. All of these analytes likely contribute to the aroma of the beverage.

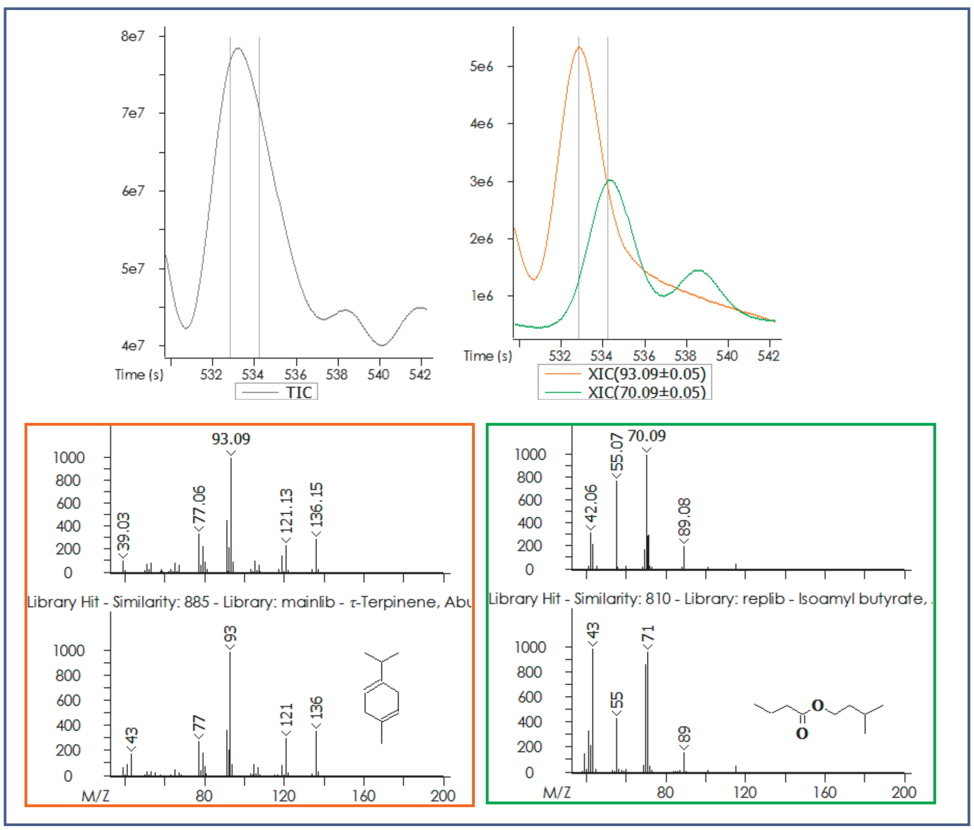

Figure 2. Another case of deconvolution is shown from the diet fruit tea. In the TIC (gray), there appears to be only one peak, but by plotting XICs unique to each analyte, individual peak profiles were observed. Here, deconvolution mathematically resolved and tentatively identified gamma-terpinene, a terpene with woody, terpene, and herbal odor properties from isoamyl butyrate, an ester with fruity odor characteristics.

In Figures 1 and 2, the coeluting analytes had comparable relative intensities. Deconvolution is also able to separate coelutions with large intensity differences, as shown in Figure 3. In this case, a small sulfur-containing analyte elutes underneath the tail of a very intense peak, identified as benzaldehyde. When XICs for each analyte are plot together (top middle), the m/z associated with dimethyl trisulfide (125.99, green trace) is so much lower than benzaldehyde (m/z 106.06, orange trace) that the peak is not apparent. The m/z can be scaled (by greater than 500x) and the coeluting peak can be observed (top right). Even with a large intensity difference, deconvolution still effectively provides information on each of these analytes for better understanding of the sample.

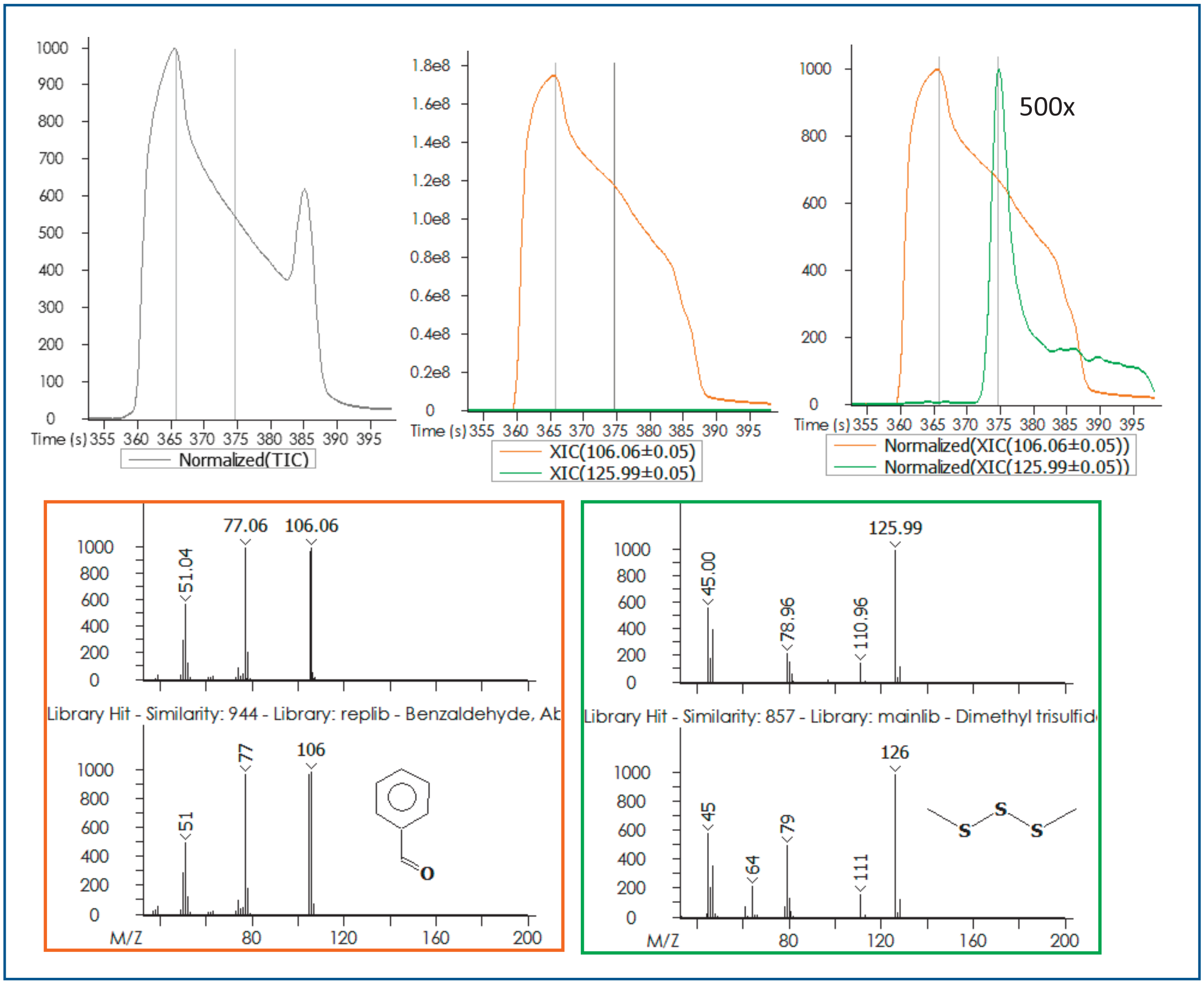

Figure 3. Deconvolution can accommodate large concentration differences. Here, a small analyte is buried beneath a large overloaded analyte in the regular fruit tea sample. Benzaldehyde is a fruity aromatic analyte that is naturally present in peaches and other fruits. This analyte is present at very high levels and obscures a number of other closely eluting analytes. In particular, dimethyl trisulfide, an alliaceous sulfur compound that is naturally present in black tea is deconvoluted from beneath benzaldehyde. The peak height for dimethyl trisulfide is more than 500x less than benzaldehyde and good deconvolution results are still achieved with excellent library similarity scores.

The *Pegasus* BT provides a complete aroma profile for every sample, from every run. With this capability, the diet and regular samples were further characterized and compared to each other for similarities and differences. Many of the analytes were present at comparable levels in each sample, but some differences were also observed. One example is demonstrated in Figure 4. Furfural is a sugar degradation product that was observed at higher levels in the regular tea that contains sugar as compared to the diet tea that does not. XICs showing the chromatographic peak shape and spectral information with the best library match from the spectral databases are shown.

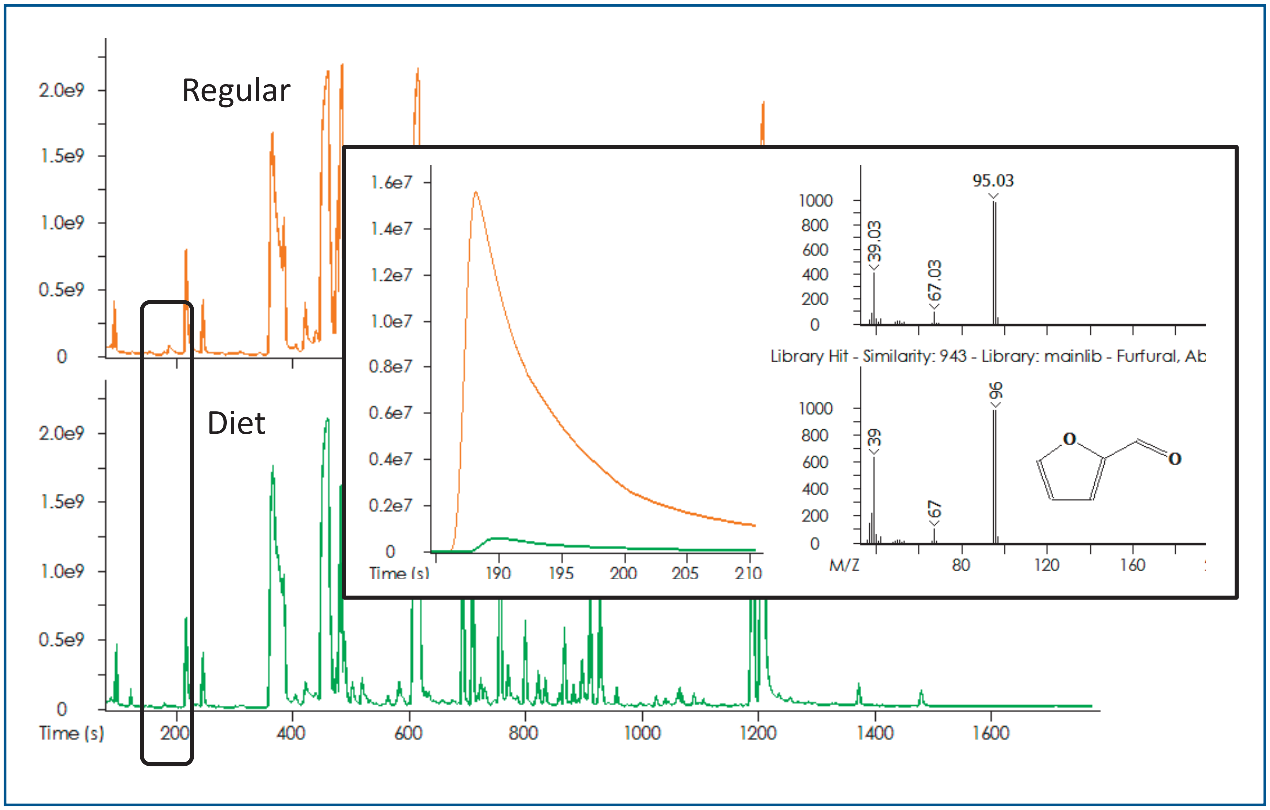

Figure 4. Taking advantage of all of the analytical capabilities of the Pegasus BT, differences between the diet and regular tea were observed. Here, furfural, a sugar degradation product, is observed in the regular tea, and at much higher levels than in the diet tea.

4. Conclusion

This study demonstrates the benefits of the *Pegasus* BT for the non-targeted analysis of beverage samples. A regular and diet fruit tea were analyzed, characterized, and compared. Deconvolution was essential to provide information on individual analytes in instances of coelution. In many cases, these analytes had known odor properties that were related to either the fruit or the tea characteristics of the beverage. The ability to determine individual analytes offered the opportunity to compare and contrast the samples and learn more from a standard analysis.

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