



Rapid Screening for Contact Allergens in Perfumes with Pegasus[®] BT GC-TOFMS

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1. Introduction

Perfumes and other personal care products often contain fragrance compounds that can be contact allergens. Various regulations, including the European Cosmetics Directive, requires allergen compounds to be listed on packaging materials when present above certain levels (10 ppm for a leave-on product, like perfume). GC paired with TOFMS is an excellent choice for measuring many of these analytes, and a rapid screening method using LECO's Pegasus BT is demonstrated here. In addition to facilitating a targeted analysis, non-targeted characterization can also be accomplished from the same full range m/z acquired data. Calibration data for regulated analytes were determined and applied to eight different perfume samples to demonstrate product screening. Numerous other non-targeted analytes were also determined, with deconvolution providing key benefits to see what you've been missing in a standard analysis.

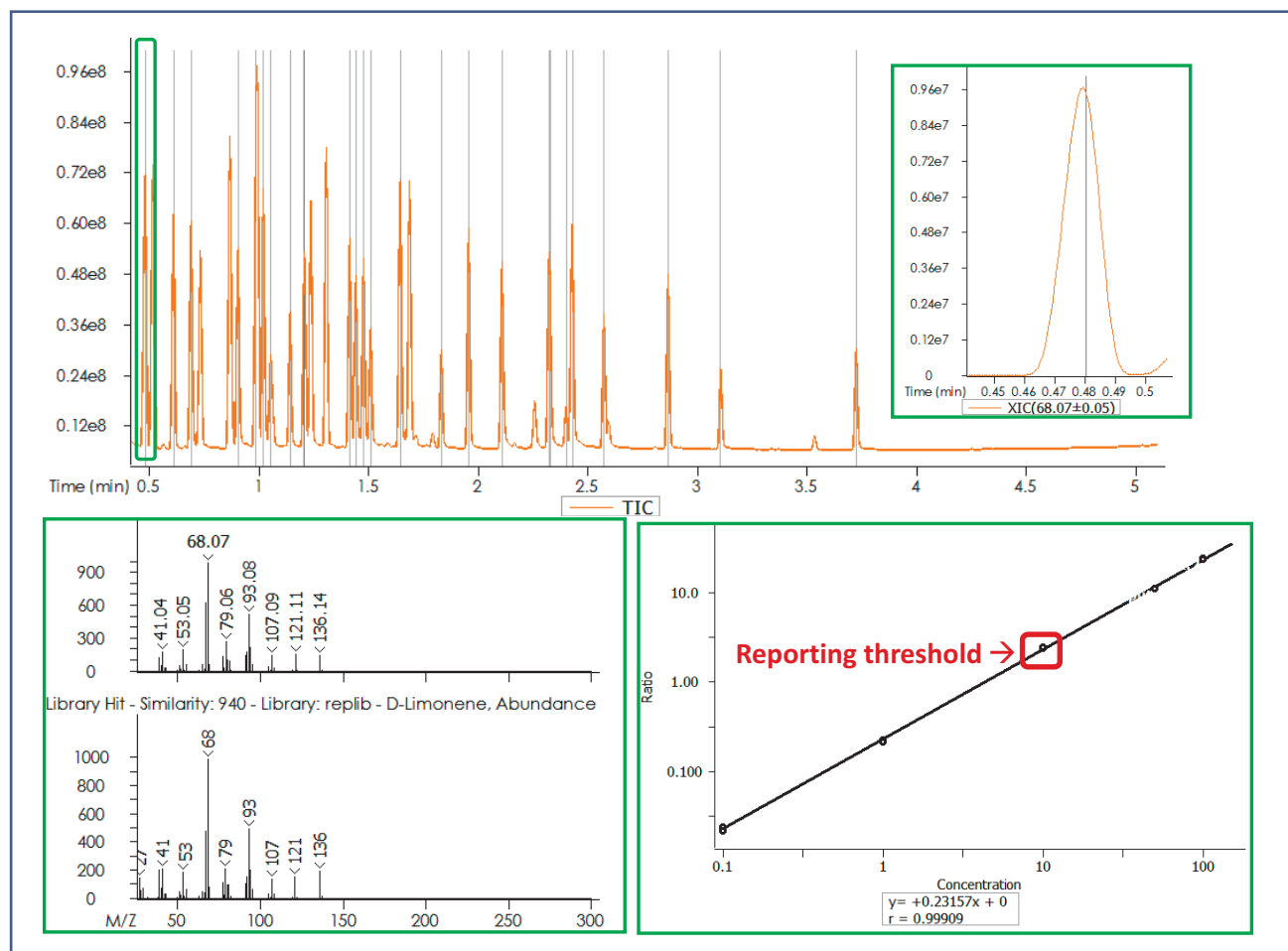


Figure 1. GC-TOFMS data were collected for an allergen standard, containing 24 regulated analytes. The entire separation occurs in only 5 minutes. Allergens are indicated in the standard with vertical line peak markers. The peak profile, mass spectral information, and calibration data for limonene are compiled as a representative example. The calibration range extends below the required reporting threshold (boxed red) accommodating dilutions, if needed. Calibration equations for all analytes were determined with automated data processing and were applied to subsequent unknown samples in an automated way. The average r value for all standards is 0.99918 \pm 0.00098.

2. Experimental

A set of calibration standards was prepared from a fragrance allergen standard (Restek) at concentrations of 100 ppb to 100 ppm. The standards were subsequently analyzed with a 100:1 split resulting in an effective concentration range of 1 ppb to 1 ppm. Eight perfume samples were diluted 10x prior to analysis, to fit within the calibration range. All standards and samples were analyzed with the conditions listed in Table 1.

Table 1. GC-TOFMS (Pegasus BT) Conditions

Gas Chromatograph	Agilent 7890 with LECO L-PAL3 Autosampler
Injection	1 μ L split 100:1 with inlet @ 250°C
Carrier Gas	He @ 1.0 mL/min, Constant Flow
Column	Rxi-17Sil MS, 9.4 m x 0.18 mm i.d. x 0.18 μ m coating (Restek)
Oven Program	0.1 min at 100°C ramped 40°C/min to 300°C
Transfer Line	300°C
Mass Spectrometer	LECO Pegasus BT
Ion Source Temperature	250°C
Mass Range	35-650 m/z
Acquisition Rate	20 spectra/s

3. Results and Discussion

A rapid GC-TOFMS method was developed to separate and detect a target set of contact allergens in approximately 5 minutes. This method utilized a short and narrow column, a fast temperature program, and ChromaTOF[®]'s automated deconvolution to chromatographically and mathematically separate the target analytes. Peak finding identified all of the target analytes, indicated with vertical line peak markers in Figure 1, with an average similarity of 926. A set of calibration standards from 100 ppb to 100 ppm were prepared and subsequently analyzed (with a 100:1 split) by GC-TOFMS. Calibration equations were calculated for each allergen standard with an average *r* value above 0.999 across all targets. Limonene is shown as an example in Figure 1. These calibrations were applied with automated processing to eight different perfume samples, with results compiled in Figure 2.

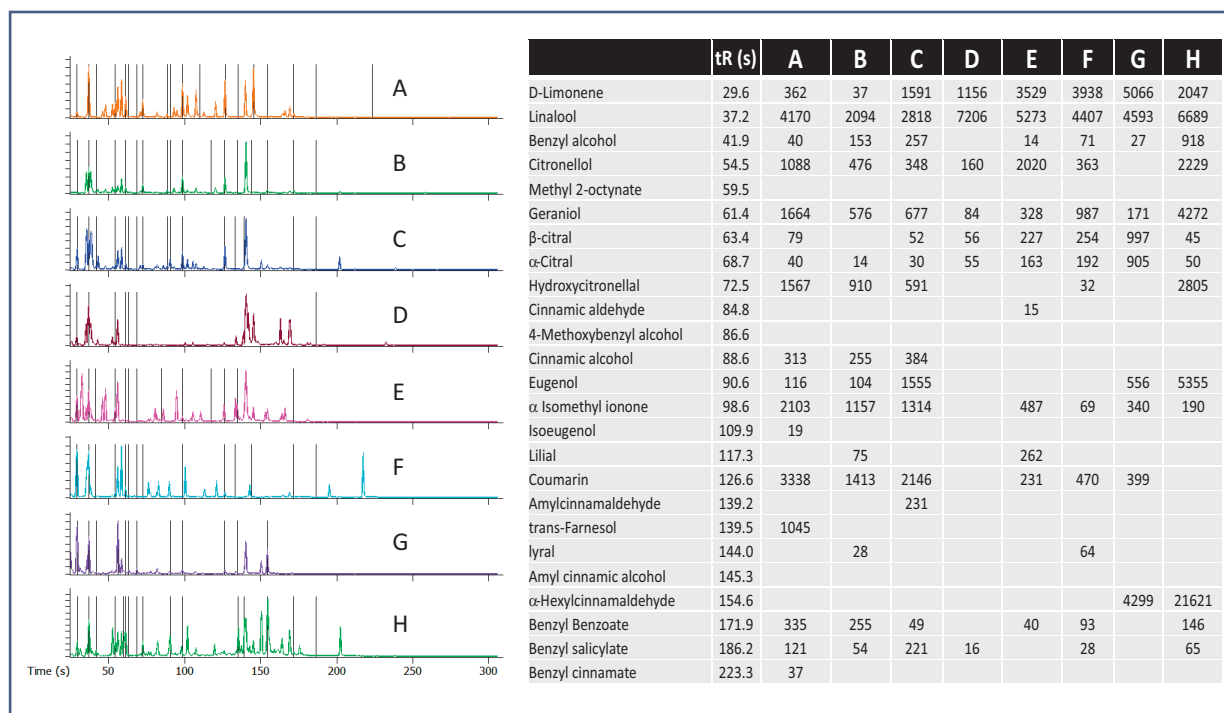


Figure 2. The calibration equations were applied to eight perfume samples using automated data processing. Vertical line peak markers are displayed, and the concentrations are tabulated for any target allergen present above the 10 ppm reporting threshold (reported concentration values are in ppm and corrected for dilution). This method provides rapid screening for the regulated target allergens to quickly determine what analytes would need to be reported. When packaging labels were available, there was good agreement between the reported analytes and those observed here.

In addition to the target allergens, the data also provides excellent characterization information for non-targeted analytes from the same injection. Several examples of non-targeted analytes present in the perfume samples are shown in Figure 3. In each of these cases, deconvolution was able to separate a non-target analyte that coelutes with one of the target analytes. The non-target analytes were tentatively identified through spectral matching to library databases. In Perfume A, the target analyte eugenol with spicy odor properties was mathematically separated from citronellyl butyrate with floral odor properties. In Perfume E, the target analyte coumarin with tonka odor properties was mathematically separated from cedrol with woody odor properties. And, in Perfume F, the target analyte citronellol with floral odor properties was mathematically separated from α -terpineol with piney odor properties. There are hundreds of additional analytes observed within the data that can provide a better understanding of the aroma properties of the perfumes.

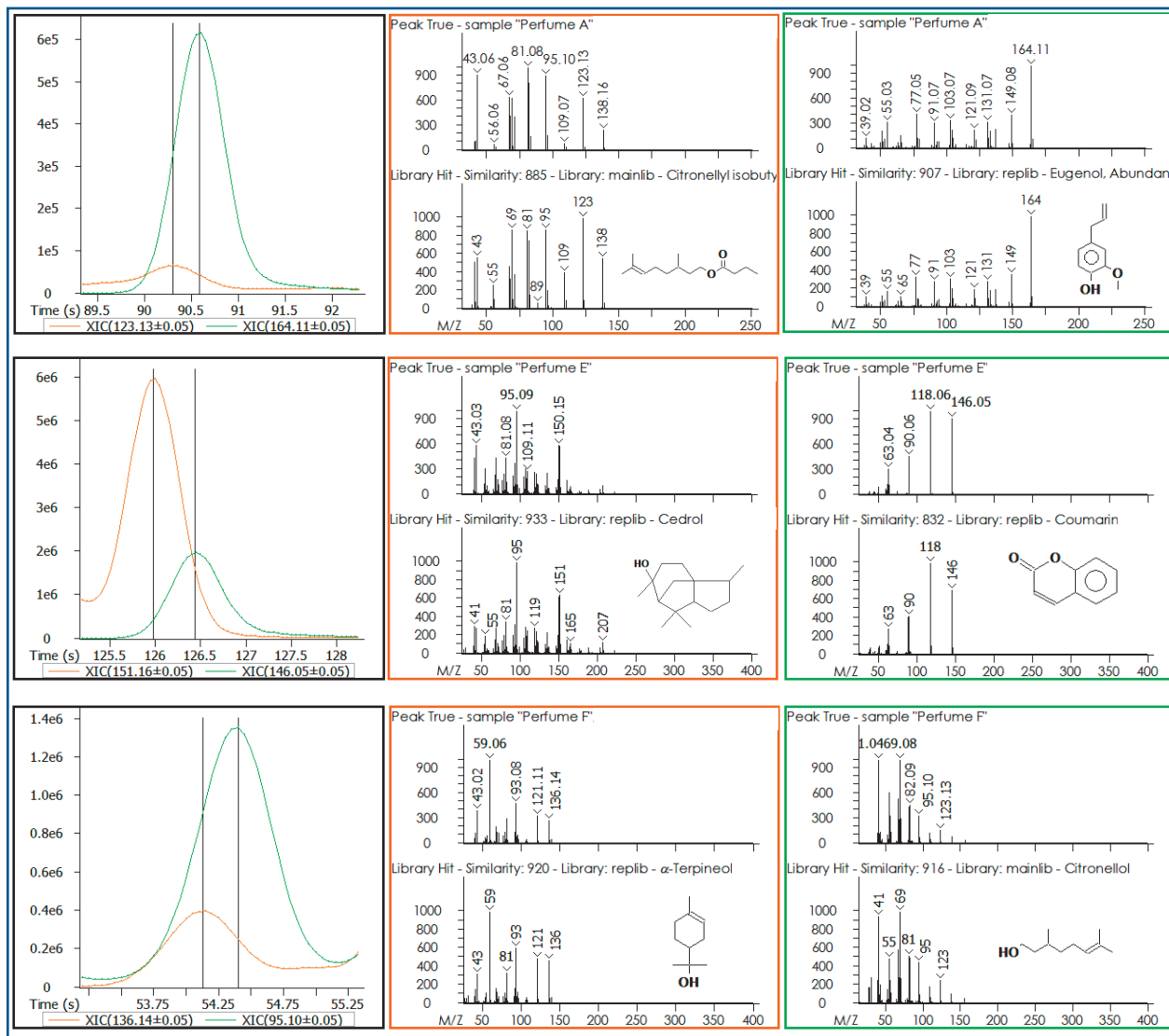


Figure 3. Three examples of non-targeted characterization that were provided through automated data processing and deconvolution. In each case a non-target analyte from the matrix coelutes with one of the target allergen standards. Deconvolution provides information on the target analyte free from the interference and also provides information on the non-target analyte that aids in general characterization and better understanding of the samples.

4. Conclusion

This study demonstrates the benefits of LECO's *Pegasus* BT GC-TOFMS to provide targeted screening for contact allergens and non-targeted characterization of general aroma analytes within complex perfume samples. Calibration data for regulated analytes were determined and applied to eight different perfume samples. The GC-TOFMS method provides a rapid quantitative screening analysis with run times of roughly five minutes. Numerous other non-targeted analytes were also observed in the same injection with deconvolution providing key benefits to see what you've been missing in a standard analysis.



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