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Direct Thermal Extraction Analysis of Food Packaging Material

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Abstract

This study describes the use of the GERSTEL MultiPurpose Sampler (MPS) fitted with a Thermal Desorption Unit (TDU) and a Cooled Injection System (CIS 4) PTV-type Inlet for the automated direct thermal extraction analysis of packaging for three different brands of crème-filled chocolate sandwich cookies, cheese-filled sandwich crackers and soft and chewy candy.

Introduction

Knowledge about packaging/product interaction is of great interest to food manufacturers in order to control product quality over the course of its shelf-life. Scalping is the loss of components from a product to its packaging. Migration is the transfer of unwanted compounds from the packaging to the food product. Both processes are undesirable and can cause off odors or off flavors in a product. In order to assess or determine the potential for migration, packaging material can be analyzed directly using direct thermal extraction (DTE). A small amount of sample, typically 10-50 mg, is placed in an empty thermal desorption tube and is then heated in the thermal desorption unit under of flow of inert gas, in order to release volatile and semi-volatile compounds from the sample. The analytes are trapped and then determined by GC/MS. Direct Thermal Extraction requires very little sample preparation and can be used for trace analysis of packaging material. In this work, the technique was used to analyze the packaging of three brands of crème filled chocolate sandwich cookies, cheese-filled sandwich crackers and soft and chewy candy. The compound benzaldehyde was quantified in one brand of soft and chewy candy packaging and found to be 79 ± 6 ng in the 25 mg sample of food packaging.

The GERSTEL MultiPurpose Sampler (MPS) in combination with the GERSTEL Thermal Desorption Unit (TDU 2) and programmable temperature vaporizer (PTV) inlet (CIS 4) provides the user with a multitude of analytical options to utilize for chemical analysis. The user can easily change between liquid and large volume injection, solid phase microextraction (SPME) and static headspace analysis, depending on the analytical needs at the time. The addition of the TDU allows for thermal desorption, direct thermal extraction, Twister and Thin Film-SPME analyses. Other options include Dynamic Headspace (DHS), a thermal extraction technique in its own right, and pyrolysis. Minimal effort is required to change between modes of operation, so the analyst can quickly adapt the system to changing needs.

EXPERIMENTAL

Instrumentation

Agilent 7890A GC / 5977B MSD
GERSTEL MPS, TDU 2 & CIS 4

Analysis conditions

TDU:	thermal extraction: splitless 40°C (0.5 min); 720°C/min; 60°C (7.5 min) standards: splitless 40°C (0.5 min); 720°C/min; 280°C (3 min)
PTV:	solvent vent (50 mL/min), split 30:1 -150°C; 12°C/sec; 280°C (3 min)
Pneumatics:	He, constant flow, 1 mL/min
Column:	30 m Rxi-5Sil MS (Restek) d_i = 0.25 mm d_f = 0.25 µm
Oven:	40°C (1 min); 10°C/min; 280°C (3 min)

Sample preparation. 25 mg of packaging was weighed, placed into an empty TDU tube and capped with a transport adapter.

For quantification of benzaldehyde, five solutions of the compound in the concentration range of 1 – 100 mg/L were made in methanol and used to establish an external calibration curve. 1 µL of each solution was spiked onto a Tenax TA tube and thermally desorbed in the TDU.

Results and Discussion

Crème Filled Sandwich Cookie Packaging. Direct thermal extraction (DTE) was applied to the packaging material for the three samples. Figure 1 shows a stacked view of the resulting chromatograms from the direct thermal extraction analyses.

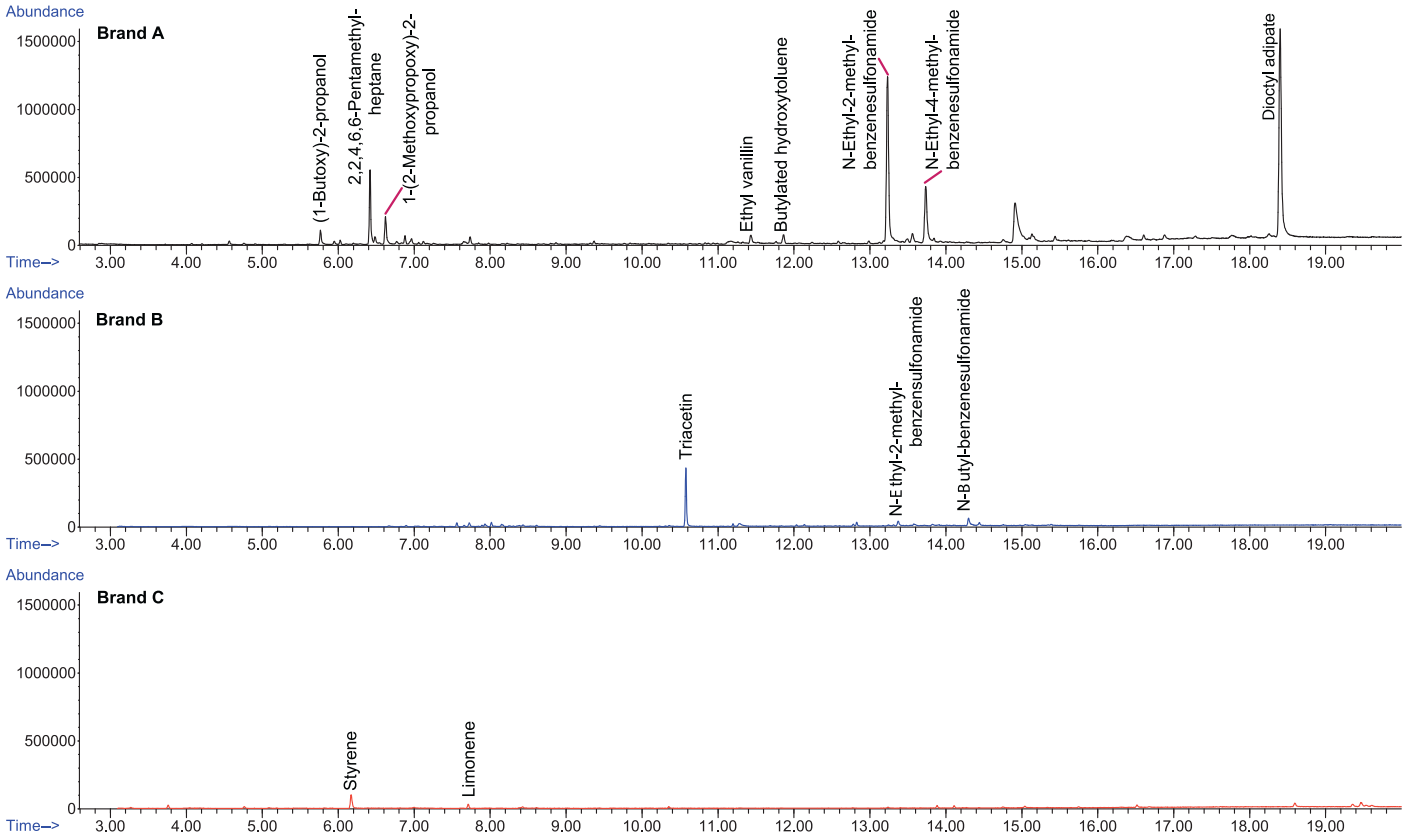

Figure 1. Stacked View of Total Ion Chromatograms for Direct Thermal Extraction of Packaging Material for Brand A (top), Brand B (middle) and Brand C (bottom) Crème-Filled Sandwich Cookies.

Brand A shows the highest level of extractable compounds. Compounds which could potentially migrate from the packaging to the food include ink related solvents; 1-butoxy-2-propanol and 1-(2-methoxypropoxy)-2-propanol, plasticizers used for adhesives; sulfonamide peaks, and another plasticizer; dioctyl adipate. The chromatogram for Brand A also shows ethyl vanillin (flavor component) and butylated hydroxytoluene (preservative) which migrated from the product to the packaging material.

The chromatogram for Brand B shows triacetin (food additive) and vanillin as products which came from the food. Two sulfonamide peaks are also seen in the chromatogram, but at much lower levels relative to Brand A. Brand C shows the lowest levels of extractable compounds. The largest two peaks being styrene and limonene.

Soft and Chewy Candy Packaging. Direct thermal extraction was then applied to the packaging of three brands of soft and chewy candy. Figure 2 shows a stacked view of the resulting chromatograms from the direct thermal extraction analyses. All three chromatograms contain both flavor compounds from the food product as well as compounds from the packaging itself. Brands D and F show the highest level of extractable compounds. The chromatograms from Brands D and F packaging contain a large amount of the compound triacetin, a carrier solvent for flavor compounds which came from the food product, as well as allyl hexanoate and benzyl acetate, which are flavoring agents. Both Brand D and F contain 2,4-di-tert-butylphenol, an antioxidant, and dioctyl adipate, a plasticizer from the packaging material. In addition, Brand D contained two sulfonamide peaks similar to those observed in Brand A and B cookie packaging.

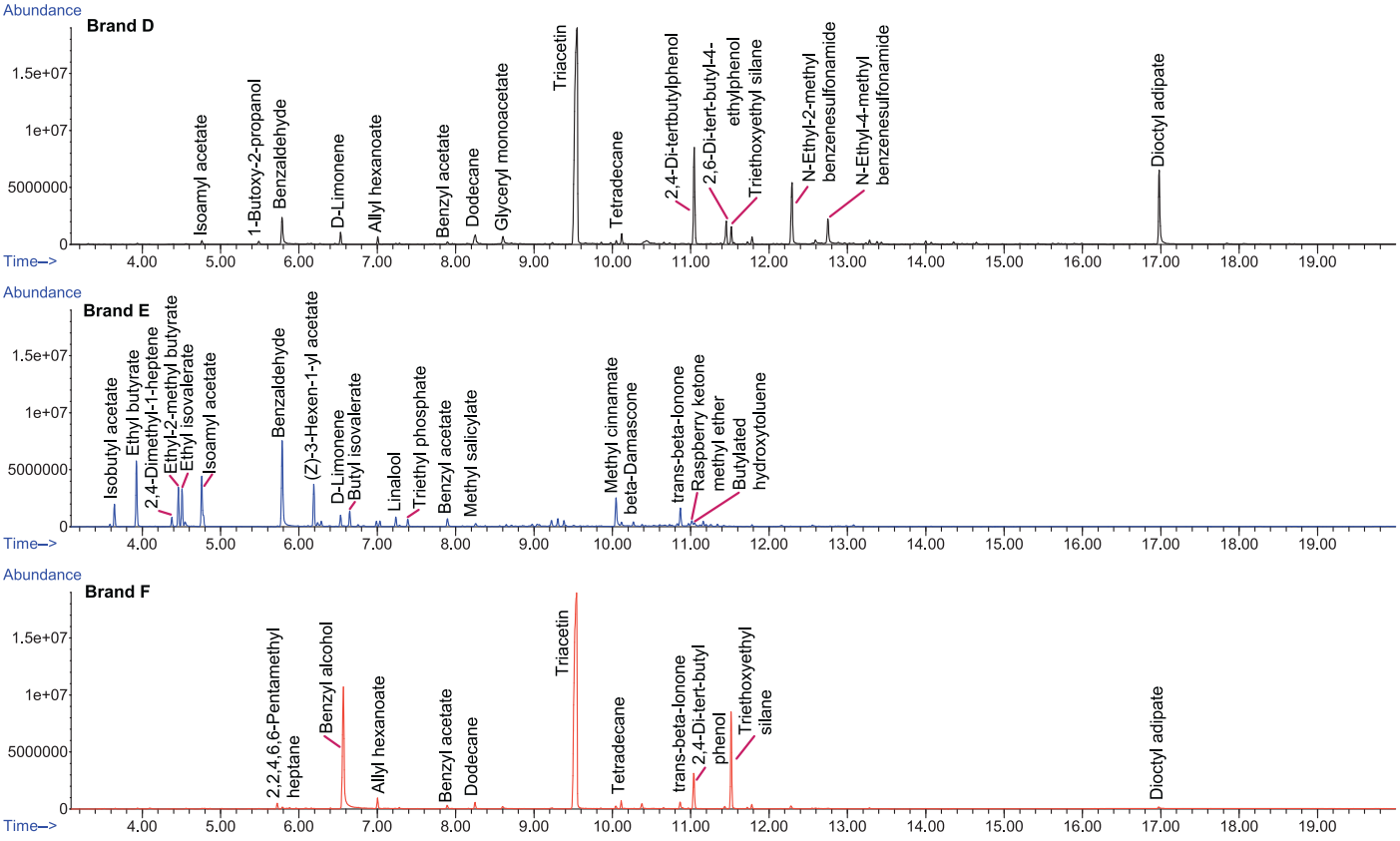

Figure 2. Stacked View of Total Ion Chromatograms for Direct Thermal Extraction of Packaging Material for Brand D (top), Brand E (middle) and Brand F (bottom) Soft and Chewy Candy.

The chromatogram for Brand E is primarily composed of flavor components resulting from migration from the candy to the packaging: isobutyl acetate, ethyl butyrate, ethyl 2-methyl butyrate, ethyl isovalerate, isoamyl acetate, benzaldehyde, D-limonene, butyl isovalerate, linalool, benzyl acetate, methyl salicylate, methyl cinnamate, beta-damascenone, trans-beta-ionone, and raspberry ketone methyl ether. The chromatogram for Brand E also included components derived from the plastic packaging material, 2,4-dimethyl-1-heptene, and well as an antioxidant, butylated hydroxytoluene.

Benzaldehyde was quantified in the Brand E packaging using an external calibration curve of the compound spiked onto Tenax TA tubes, which were thermally desorbed in the TDU. The amount of benzaldehyde in the 25 mg sample of Brand E packaging was determined to be 79 ± 6 ng (n=3).

Cheese Filled Sandwich Cracker Packaging. Packaging samples from three brands of cheese-filled sandwich crackers were subjected to direct thermal extraction analysis. Figure 3 shows a stacked view of the resulting chromatograms from the analyses of cheese-filled sandwich cracker packaging. All three brands contained nonanal (food component) and butylated hydroxytoluene (antioxidant). Brand G and brand H showed numerous similarities. Both contained a variety of compounds that were derived from the food product: hexanal, hexanoic acid, isopropyl laurate, isopropyl myristate, and heptacosane. Brand G and H also both contained the packaging components N-butyl benzenesulfonamide and tributyl acetylcitrate, which are plasticizers.

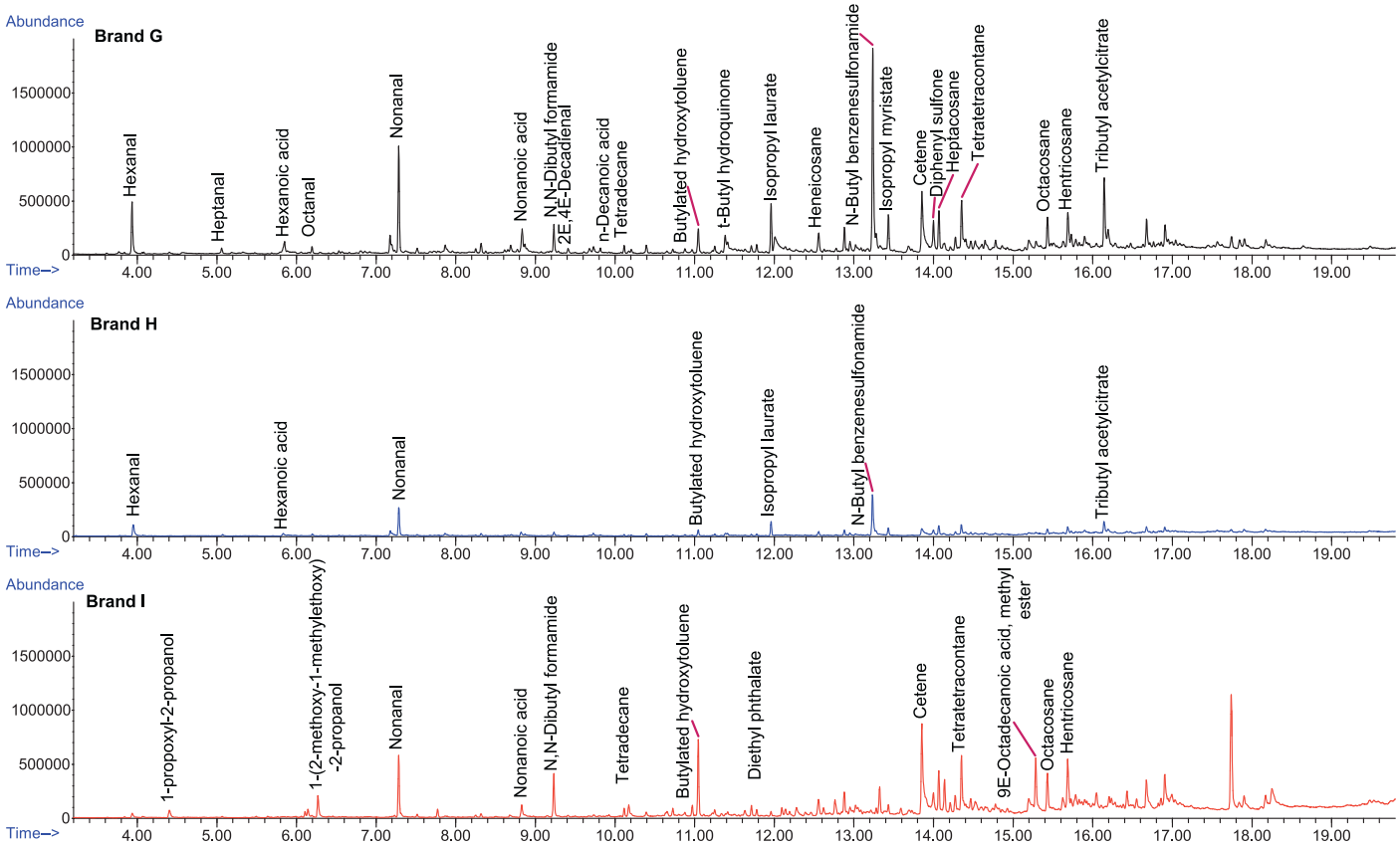

Figure 3. Stacked View of Total Ion Chromatograms for Direct Thermal Extraction of Packaging Material for Brand G (top), Brand H (middle) and Brand I (bottom) Cheese Filled Sandwich Cookie Packaging.

Brand I contained ink-derived solvents 1-propoxy-2-propanol and 1-(2-methoxy-1-methylethoxy)-2-propanol, similar to compounds which were observed in the Brand A cookie packaging. Brand I also contained the packaging component diethyl phthalate.

Conclusions

The results show that direct thermal extraction is a very good method for quantification as well as assessment of quality defects that may arise due to compounds introduced from food packaging. The method requires very little sample preparation.

The GERSTEL MPS/TDU/CIS provides a versatile platform for qualitative analysis of food product packaging. Several techniques can be applied quickly to the same samples, which provide the necessary data for quality control, product development troubleshooting or competitive analysis. With the appropriate use of standards, any of the techniques can be used for quantitative analysis, as illustrated by the quantification of benzaldehyde in the Brand E soft and chewy candy packaging.

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