Analysis of Food-Packaging Film by Headspace-GC/MS

Authors:
Miles Snow
Heidi Grecsek

PerkinElmer Life and
Analytical Sciences
710 Bridgeport Avenue
Shelton, CT 06484

Introduction

Food-packaging material is typically manufactured as a thin film and coated with inks which usually contain multiple, harmful, volatile organics. Therefore, they must be carefully monitored and quantitated to ensure that the amounts are limited.

Traditionally, the test for solvent materials in food-packaging film was performed using a technique of heating a square meter of the film material inside a mason jar. This jar is then opened and tested (by smell) for volatile organic compounds. Later, this test was expanded to extract a headspace sample out of the mason jar by syringe and then injected into a gas chromatograph (GC) for quantitative analysis. This produced significantly better results and provided laboratories with a quantitative number. This process is still very time-consuming and labor intensive as a result of the number of manual steps involved. The manual process of cutting food packaging, placing it in a mason jar, heating the jar, and manually collecting a sample for GC analysis dramatically limits the number of samples that can be analyzed each day. The technique demonstrated here will greatly improve the efficiency and throughput of this analysis.

This analysis can be completely automated using a PerkinElmer® TurboMatrix™ Headspace (HS) sampler with the Clarus® 500 Gas Chromatograph/Mass Spectrometer (GC/MS) – see Figure 1 on Page 2. This system passed all the requirements for food-packaging analysis.

Experimental

The first food packaging film used for this experiment was from a typical package of cookies. This film was cut into squares: 325 cm^2 pieces. The typical volume used in a mason jar is a square meter but this volume is not required for the headspace sampler. The desired sensitivity can be reached with significantly lower quantities. The second packaging material tested was obtained from a shopping bag that you would typically find at a department store.

The 325 cm^2 pieces of film were added directly to a 22-mL headspace vial. The vial was then sealed with silicone/PTFE septa (PerkinElmer part number B0104241). In addition, a calibration standard was prepared to get an estimate of the expected concentration of the typical solvents. This standard was prepared by adding 4.7 µg of each solvent in a 22-mL headspace vial (Table 2).

PerkinElmer® precisely.

The instrument used for this analysis was a TurboMatrix HS 40 Headspace Trap sampler run in headspace-only mode. This bypassed the trapping capability. If extra sensitivity is required, the trap option could be used for up to 100 times lower detection levels. The shaker option on the headspace was utilized for a faster equilibration of the solid film material. The headspace was controlled using the TurboMatrix remote control software and was coupled to the Clarus 500 GC/MS. The Clarus 500 GC was equipped with a programmable split/splitless (PSS) injector and programmable pneumatic control (PPC). Deactivated fused silica (0.32 mm) transfer line connects the TurboMatrix HS 40 Trap to the Clarus GC. The GC column was directly connected to this transfer line using a universal union (PerkinElmer part number N9302149). The Clarus 500 MS was controlled via TurboMass™ 5.1 GC/MS software and operated in electron ionization (EI) mode.

Results

The TurboMatrix HS 40 Headspace sampler was successful in analyzing the solvents in food packaging. Six solvents were identified: 1 – MIBK (Methyl Isobutyl Ketone), 2 – NPAC (n-Propyl Acetate), 3 – ETAC (Ethyl Acetate), 4 – Propanol, 5 – ETOH (Ethanol) and 6 – Heptane (Figures 2 and 3). Ethanol and Propanol were the largest responders and overloaded the system. However, the requirements of the testing were to only get semi-quantitative information. Therefore, the overloading was accepted. All components were positively identified using a NIST library database.

The cookie package/wrapper had approximately 0.22 mg/m^2 of solvents found. However, Propanol was very significant, making up the large majority of the total solvents identified. The cookie wrapper also had a lower level of interferences from outside sources (Figure 2). The shopping bag (purple) had approximately 0.32 mg/m^2 of total solvent material (of the six solvents tested) – Table 2. This represented a very good response of all six solvents. In addition, there is a significant amount of other materials found in the food film. This is evident in the chromatogram shown on Figure 3. Because of the ability of the MS to extract only the required ion from the component of interest, this interference was not an issue.

The headspace system enabled the method to be set up and run unattended with no sample preparation. This eliminated the need for mason jars and operator attention. In addition, the system showed a significant amount of sensitivity for the required components, demonstrating the ease of setup methodologies of many types of food packaging at many different levels.

The significant response of the volatile solvent material by this heated headspace technique would allow for a flame ionization detector (FID) to be used as a substitute for the MS detector. While the MS gives a positive identification as well as selectivity, the FID can be used in a majority of standard QA/QC environments.

Figure 1. Clarus 500 GC/MS with TurboMatrix Headspace Trap.

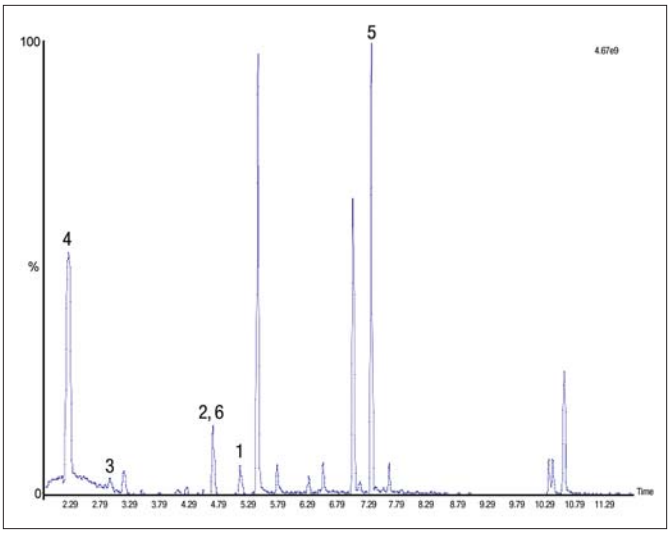

Figure 2. Chromatogram of cookie wrapper.

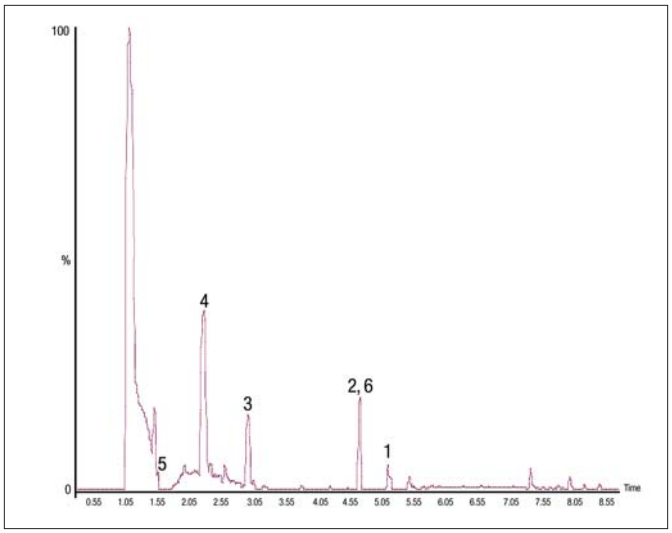

Figure 3. Chromatogram of shopping-bag film – with expanded ethanol chromatogram.

Table 1. Instrument Parameters.

GC		**Headspace Trap**	
Injector Temp:	120 ˚C	Needle Temp:	85 ˚C
Oven Program – Initial Temp:	35 ˚C	Transfer-Line Temp:	85 ˚C
Initial Time:	Hold 2 mins	Oven Temp:	80 ˚C
Ramp:	10 ˚C/min	Shaker:	On
Final Temp:	200 ˚C	High-Pressure Injection:	On
Final Time:	Hold 5 mins	Trap Option:	Off
Column:	Elite 200*	Constant Mode:	On
MS	**SIFI Conditions EI**	Thermostatting Time:	30 min
Mass Range:	30-300 amu	Pressurization Time:	1 min
Scan Time:	0.41 sec	Withdrawal Time:	1 min
InterScan Delay:	0.01 sec	Injection Pressure:	20 psi
Transfer-Line Temp:	200 ˚C	Column Pressure:	25 psi
Electron Energy:	70 eV	Injection Time:	0.08 min
Detector Voltage:	400 V	GC Cycle Time:	35 min
Threshold:	0	Carrier Gas:	Helium at 99.999%

*PerkinElmer part number – N9316630 (30 m, 0.32 mm, 1µ).

Conclusions

The PerkinElmer TurboMatrix Headspace Trap with the Clarus 500 GC/MS meets all the requirements for food-packaging analysis. The main requirement for this application is fast, easy and quantitative solvent determination. Using the setup demonstrated here, the sample is placed into a vial and placed in the autosampler tray of the headspace. Then the automated analysis is completed without operator attention. In addition, the headspace's overlapping thermostatting allows up to 12 samples to be processed simultaneously, thus allowing 50-75 analyses per day.

Table 2. Semi-Quantitative Results.

Sample: Standard

Peak #	Solvent Name	RT (min)	Area	µg in Vial	
1	MIBK	5.104	15165890	4.7	
2	NPAC	4.663	19381950	4.7	
3	ETAC	2.932	14047220	4.7	
4	Propanol	2.256	16693610	4.7	
5	ETOH	1.518	31902978	4.7	
6	Heptane	4.69	12375010	4.7	

Sample: Purple Shopping Bag 300 cm²

Peak #	Solvent Name	RT (min)	Area	µg in Vial	mg/m²
1	MIBK	5.12	162823	0.05	0.00
2	NPAC	4.665	4010297	0.97	0.03
3	ETAC	2.927	4236236	1.42	0.04
4	Propanol	2.244	19894030	5.6	0.17
5	ETOH	1.526	15599010	2.3	0.07
6	Heptane	4.686	14941	0.01	0.00
Total					**0.32**

Sample: Cookie Wrapper 325 cm²

Peak #	Solvent Name	RT (min)	Area	µg in Vial	mg/m²
1	MIBK	5.112	30410	0.01	0.00
2	NPAC	4.664	2320430	0.56	0.02
3	ETAC	2.947	472144	0.16	0.00
4	Propanol	2.243	21689300	6.11	0.19
5	ETOH	1.533	2630198	0.39	0.01
6	Heptane	4.692	211345	0.08	0.00
Total					**0.22**

PerkinElmer Life and Analytical Sciences
710 Bridgeport Avenue
Shelton, CT 06484-4794 USA
Phone: (800) 762-4000 or
(+1) 203-925-4602
www.perkinelmer.com

PerkinElmer®
precisely.

007553_01