RESIDUAL SOLVENTS IN FOOD PACKAGING
Food Packaging Analysis through Dynamic Headspace Sampling
APPLICATION NOTE AN_174

Introduction

The contamination caused by the contact of packaging with foodstuffs is a concrete risk for human health and can affect the quality of foodstuff themselves. Two aspects are usually considered: one is the quality control of flexible packaging through the determination of residual solvents from manufacturing process, adhesives, inks and dyes; the second one is the study of the migration of chemicals from the packaging to foodstuffs: the proper simulant is selected according to the nature of the food.
Dynamic Headspace sampling can be used in both cases: it provides higher sensitivity than conventional static headspace, simple operation and high quality results.

DANI

Food Packaging

INSTRUMENTATION AND EXPERIMENTAL CONDITIONS

The system is composed of the the DANI Master DHS Dynamic Headspace coupled to the Master GC Fast Gaschromatograph equipped with a Split/ Splitless injector and a FID detector.
The Master DHS was connected to the GC inlet through an inert heated transfer line. Method conditions and instrument parameters are shown here below.

In the Master DHS the sample is directly placed into a conventional 20 mL vial. The vial is then introduced into a heated oven for incubation. When combined with the Master AS, the system delivers complete automation of all the operational steps (e.g. standard addition and incubation).
The vial is then pierced by a dual coaxial volumes needle in which a flow of inert gas blows through the headspace vial stripping the volatiles out of the sampling matrix. The gaseous phase, flowing into the needle external secondary volume, is led to a "valve and trap" focusing system which performs the concentration of the volatiles.

Superior sensitivity is obtained through the constant sweeping of the thermostatted vial, promoting the enrichment of the volatile compounds on the adsorbent trap. Analytes are focused in a sorbent packed trap, rapidly thermally desorbed and introduced into the gaschromatographic column.
After the injection, the trap is baked to a higher temperature to clean it, avoiding, thus, any risk of carryover effect.
Method conditions and instrumental parameters are shown in ***Table 1***.

A 50 cm^2 packaging sample was placed in a vial and conditioned for 1 hour at 100°C before the analysis. A 14-component mixture containing equal volumes (Residual Solvents in Packaging Material Mix 1 from Supelco) was used to prepare 6 calibration levels in the range 0.5-20.0 mg/m2.
Packaging samples and standard vials were stripped for 10 minutes at 30 mL/min.

Master DHS Dynamic Headspace Sampler	
Trap Material	Carbotrap - Carbosieve SIII
Valve Temp.	250°C
Transfer Line Temperature	225°C
Stripping Time	10 min
Stripping Flow	30 mL/min
Trap Temp	30°C / 310°C
Injection Time	1 min
Baking Time	10 min
Trap Baking Time	350°C

Table 1a : Master DHS Parameters

Master GC Gas Chromatograph	
Injector	Split-Splitless
Injector Temperature	250°C
Column	VOCOL 60 m x 0.25 mm i.d. x 1.5 um d_f
Carrier (He)	1,5 mL/min (split 1:80)
Detector	FID (250°C)
Oven	35°C (4 min), 4°C/min, 200°C (2 min)

Table 1b : Master GC Parameters

Residual Solvent Analysis

In this type of test potential outgoiong of packaging material was evaluated through the analysis of residual solvents.

The chromatograms obtained from a concentration equivalent to 0.5 mg/m^2 are reported hereunder.

Sensitivity obtained was much higher than the limits setted by the industry reference method EN 13628-2 based on the static headspace technique.

Repeatability, calculated on 6 replicates of a 1 mg/m^2 solution, was excellent for all the compounds (0.97< RSD% < 5.06). The method showed a good linearity in the range of 0.5 - 20 mg m^2.

A Cookies packaging, analyzed under the same conditions, revealed a significant presence of residual solvents.

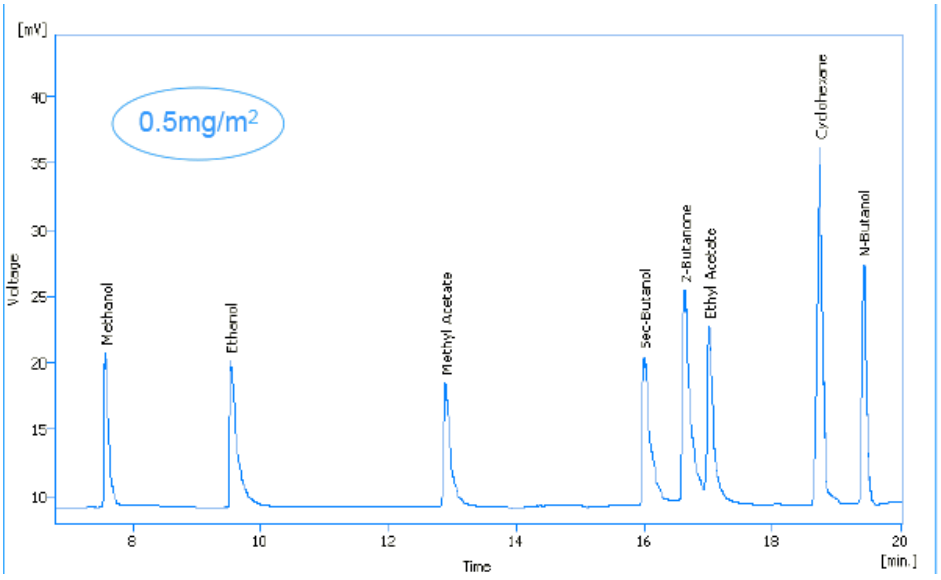


Figure 1 : Analysis of Residual Solvents

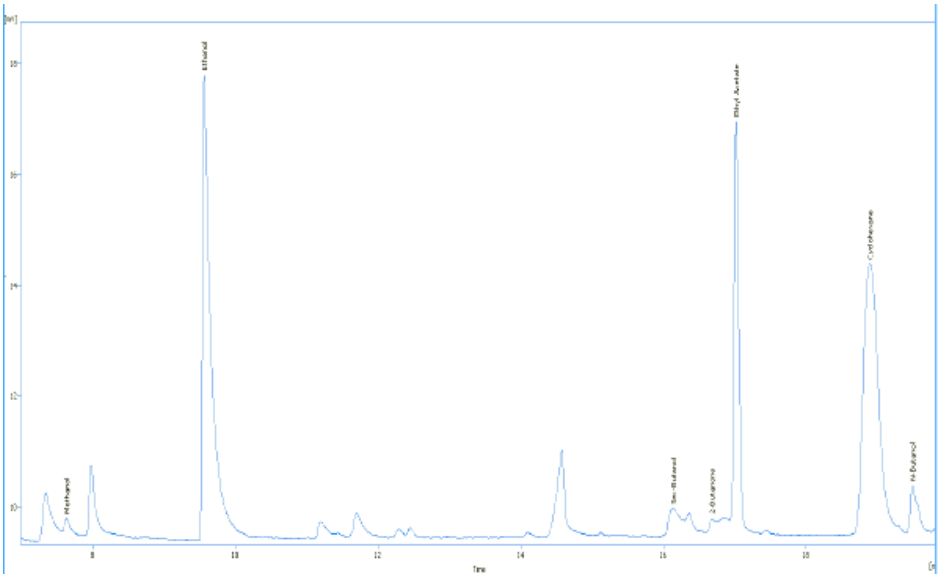
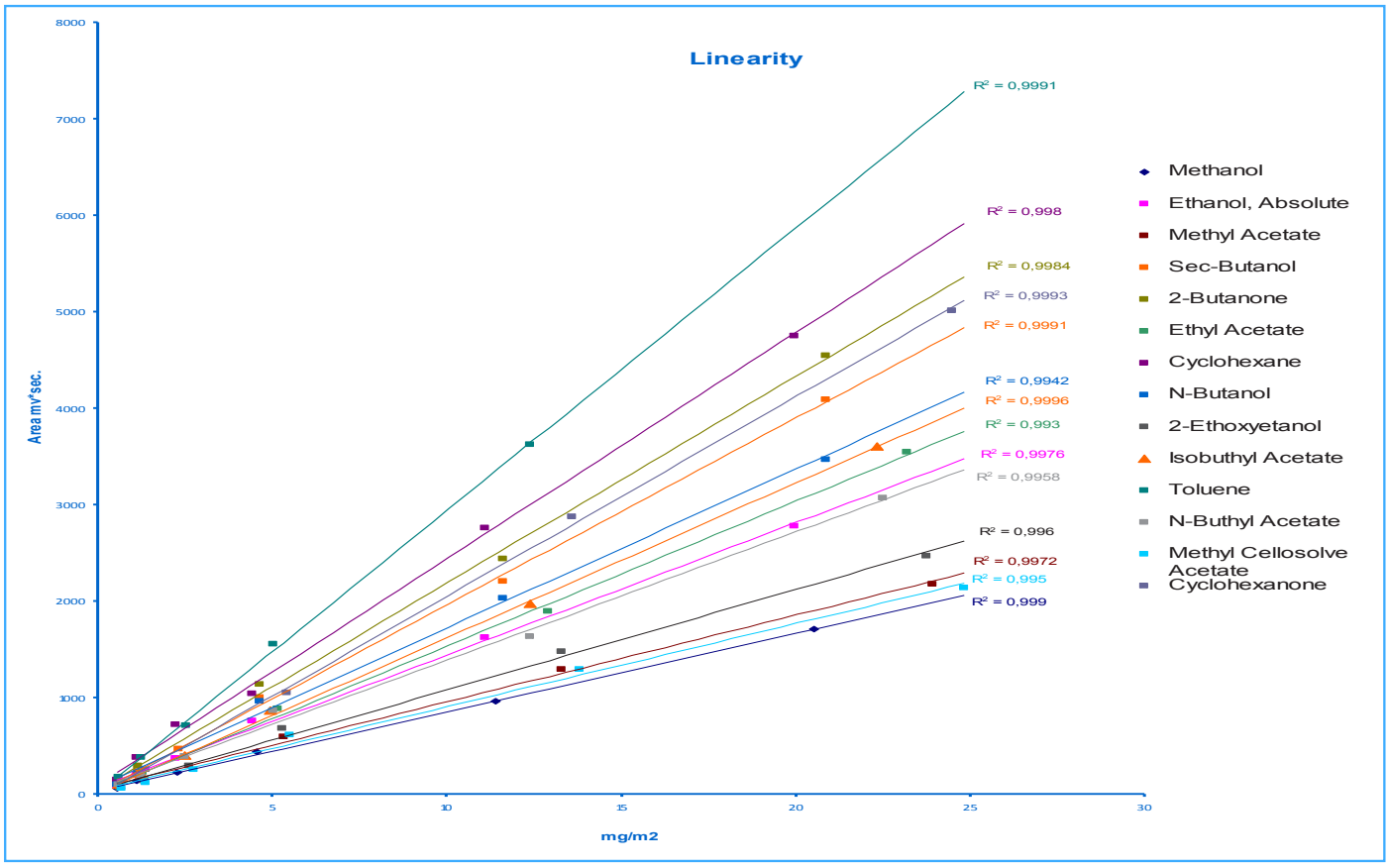


Figure 2 : Analysis of Residual Solvents in Cookies Packaging



	Compound	Average	RSD%
1	Methanol	133.46	1.01
2	Ethanol, Absolute	196.03	0.97
3	Methyl Acetate	158.57	0.97
4	Sec-Butanol	231.28	1.52
5	2-Butanone	289.29	1.68
6	Ethylacetate	222.10	1.50
7	Cyclohexane	383.09	1.49
8	N-Butanol	207.21	2.36
9	2-Ethoxyethanol	135.36	2.84
10	Isobutyl Acetate	189.26	2.80
11	Toluene	379.90	2.48
12	N-Buthyl Acetate	189.26	3.63
13	Methyl Cellosolve Acetate	119.49	4.65
14	Cyclohexanone	251.72	5.06

Migration Test

Migration test of food packaging are aimed toexplain the potential release of compounds both in polar and non-polar environment.
After the exposure to different chemical environment conditions sample were analyzed following the same protocol of the previous analysis.

Chromatograms from the three samples are reported here below: a different profile was obtained from the packaging, the water and olive oil extract confirming differences in migration and extraction depending on the simulant.

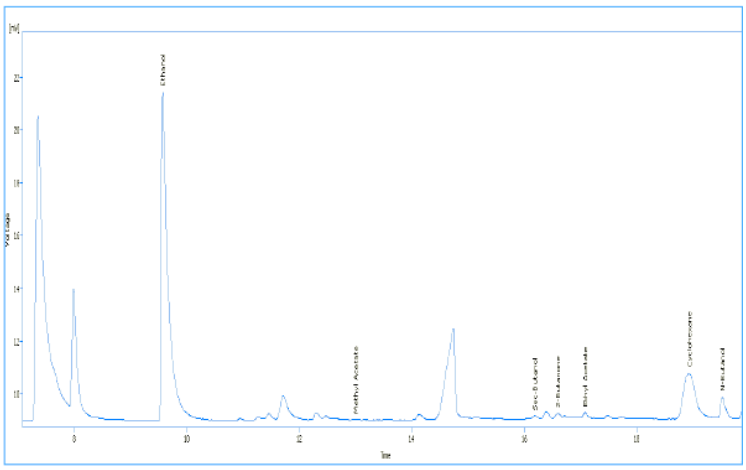


Figure 4 : Candy Packaging

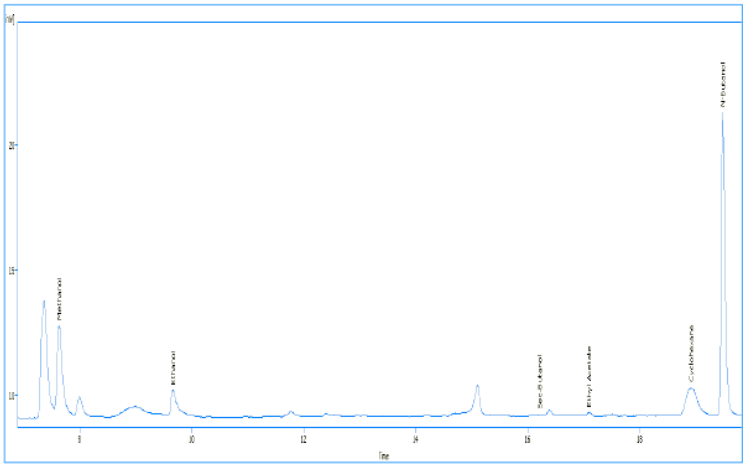


Figure 5 : Aqueous extract of candy packaging

Conclusions

The system composed by DANI Master DHS and DANI Master GC-SL/IN-FID demonstrated to be a simple and reliable tool for the determination of residual solvents in food packaging and for the extraction of Volatile Organic Compounds from food simulants. It guarantees superior sensitivity relative to other techniques but equivalent quality performances in terms of repeatability and linearity. It fully automates all the operation steps including standard additions and incubations.

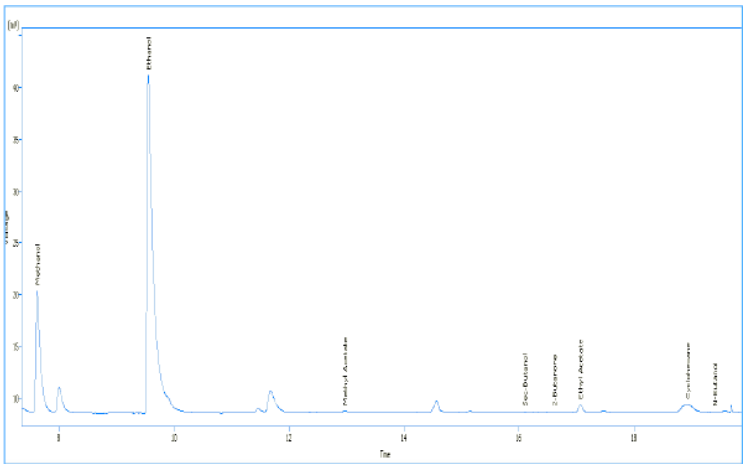


Figure 6 : Olive oil extract of candy packaging

www.dani-analitica.com

AN_174