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

Introduction

The monitoring of packaging materials is becoming one of the most important target in the food industry.
The DANI Master SHS Static Headspace Sampler permits the complete automatic sampling into the analytical system: the determination of the residual solvent is obtained at minimum detectable levels below those currently recommended by the norms (EN13628-2:2002).
When coupled to a GC-TOF MS system, it is possible to meet all the requirements for food packaging analysis.
In particular, DANI Master TOF-MS Plus is capable of providing high speed acquisition rate to collect a sufficient number of information enabling an accurate recognition and quantification of unknown and co-eluting peaks typically present in these type of samples.

By adopting a fast chromatographic approach, the analysis time can be drastically reduced, while the Time of Flight Mass Spectrometer guarantees distinguishable signals for the compounds, providing their identification and allowing their quantification.

Food Packaging

DANI

INSTRUMENTATION AND EXPERIMENTAL CONDITIONS

The proposed configuration is based on the DANI Master SHS Static Headspace Sampler, DANI Master GC Fast Gas Chromatograph and DANI Master TOF-MS Plus TIme of Flight mass spectrometer.
In the headspace autosampler the sample is placed into a conventional 20mL vial, and introduced into a heated oven for incubation.
The Master SHS Static Headspace Sampler has different possibilities to control the vial pressurization and the loop fill step. In this work, the "Pressure" mode was used to pressurize the vial at a target pressure of 1 bar.
The loop fill step was optimized using the following modes: loop "pressure" and "custom" modes.
In the loop "pressure" mode, the system controls the final pressure of loop before injection, instead in the "custom" mode, both the final loop pressure and the speed rate of depressurization are controlled.
A 50 cm^2 packaging sample was placed into a vial and conditioned for 1 hour at 100°C before the analysis. A 14 component mixture (Residual Solvent in Packaging MIX 1 from Supelco) and a 11 component mixture (Residual Solvent in Packaging Mix 2 form Supelco) containing equal volumes was used to prepare 5 levels of calibration in a range 0.1-4 mg/m2 (values depending to the density of each compound). The analysis paramenters are shown in ***Table 1, 2 and 3***, on the right.

Master SHS Static Headspace Sampler	
Temperature	Oven Control : 125°C Manifold : 250°C Transfer Line : 250°C
Equilibration	Vial Equilibration Time : 30 min Shaking: medium
Pressurization	Mode: perssure Aux pressure : 1 bar Press. Eq. Time : 0.2 min
Loop	Loop fill mode: Pressure Loop pressure : 0.5 bar Loop Eq. Time: 0.2 min
Injection	Inj. mode: Standard Vial Venting: ON

Table 1 : Master SHS Parameters

Master GC Gas Chromatograph		
OVEN		
Temperature (°C)	**Time (min)**	**Rate (°C/min)**
35	0	2
40	0	7
60	0	15
180	1	
INJECTOR		
Temperature (°C)	250	
Flow (mL/min)	0.6	
Split	1:60	
COLUMN		
Lenght	**Diameter (mm)**	**Film µm**
20	0.18	1

Table 2 : Master GC Parameters

Master TOF MS Plus Time of Flight Mass Spectrometer	
Range Mass	30-300 amu
Acquisition Rate	25 Hz
Ion Source Temperature	200°C
Transfer Line Temperature	250°C

Table 3 : Master TOF-MS Plus parameters

Fast vs Conventional Analysis : a comparison

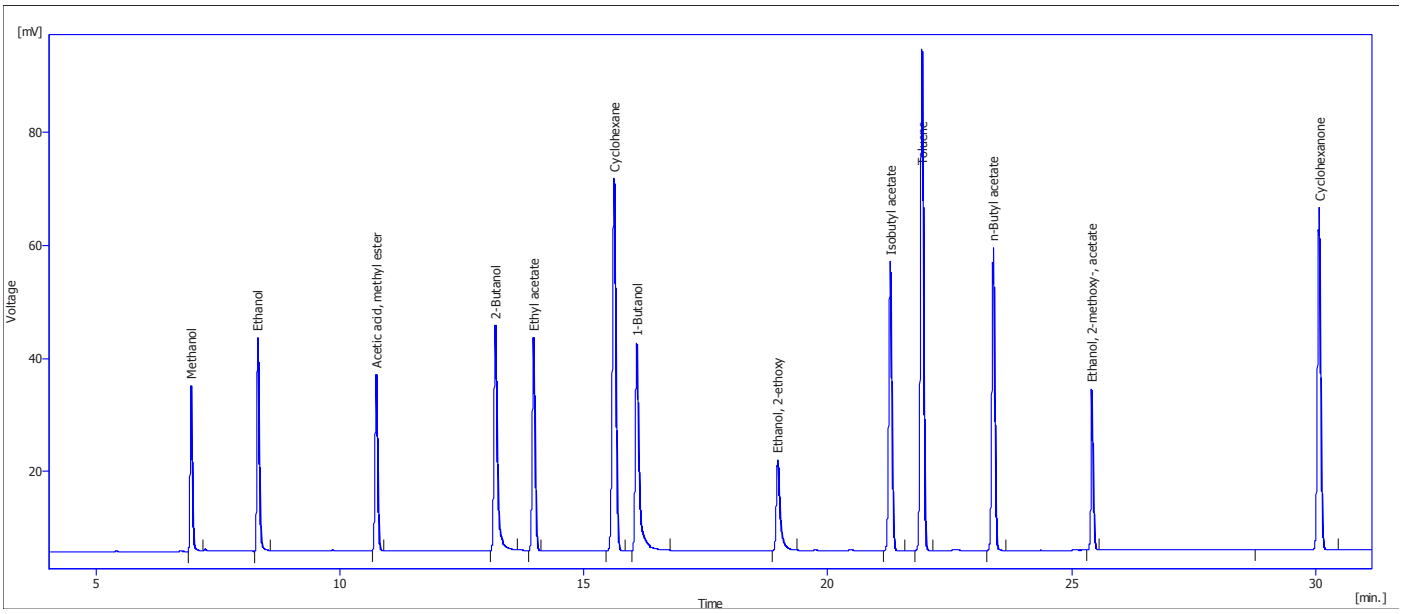

Figure 1 : Conventional Analysis of Mix 1 using SHS-GC-FID

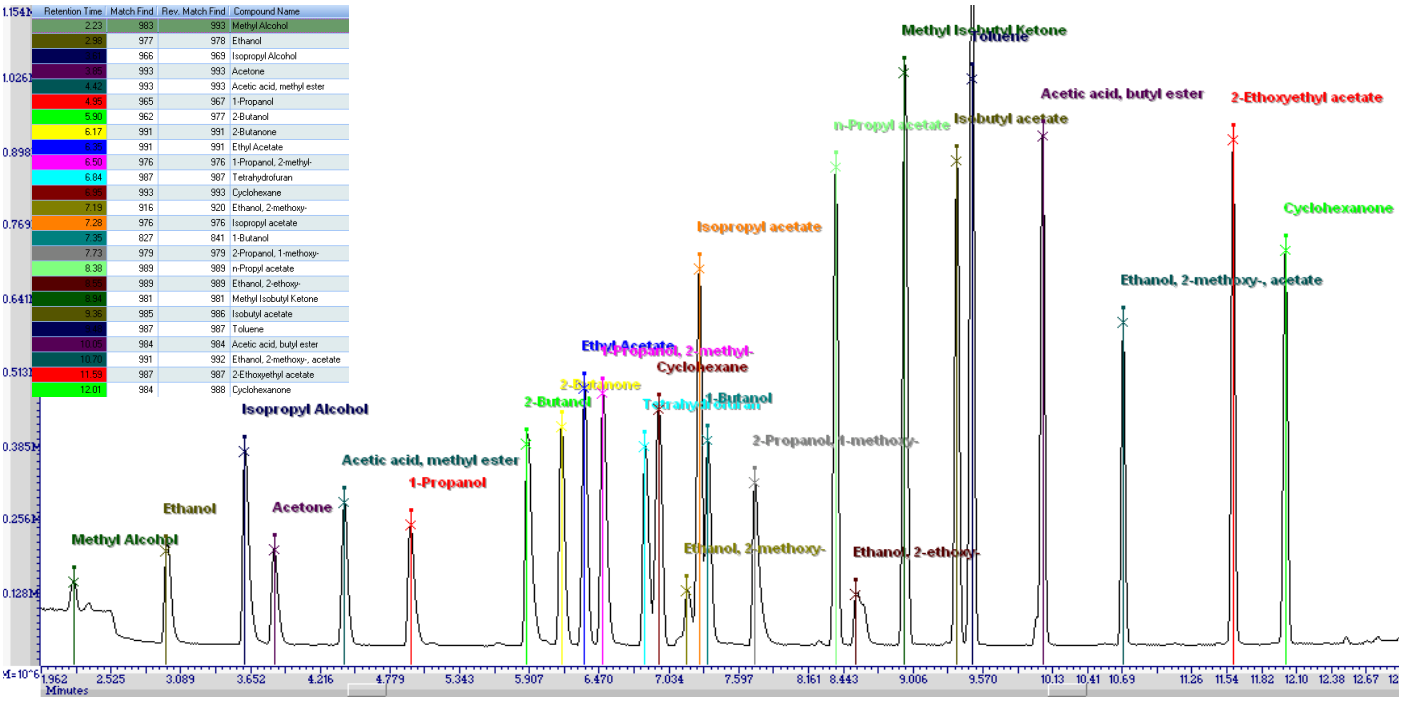

Figure 2 : Fast Analysis of Mix 1 and Mix 2 by SHS-GC-TOF MS configuration

SHS-GC-TOF MS Fast Analysis of Real Samples

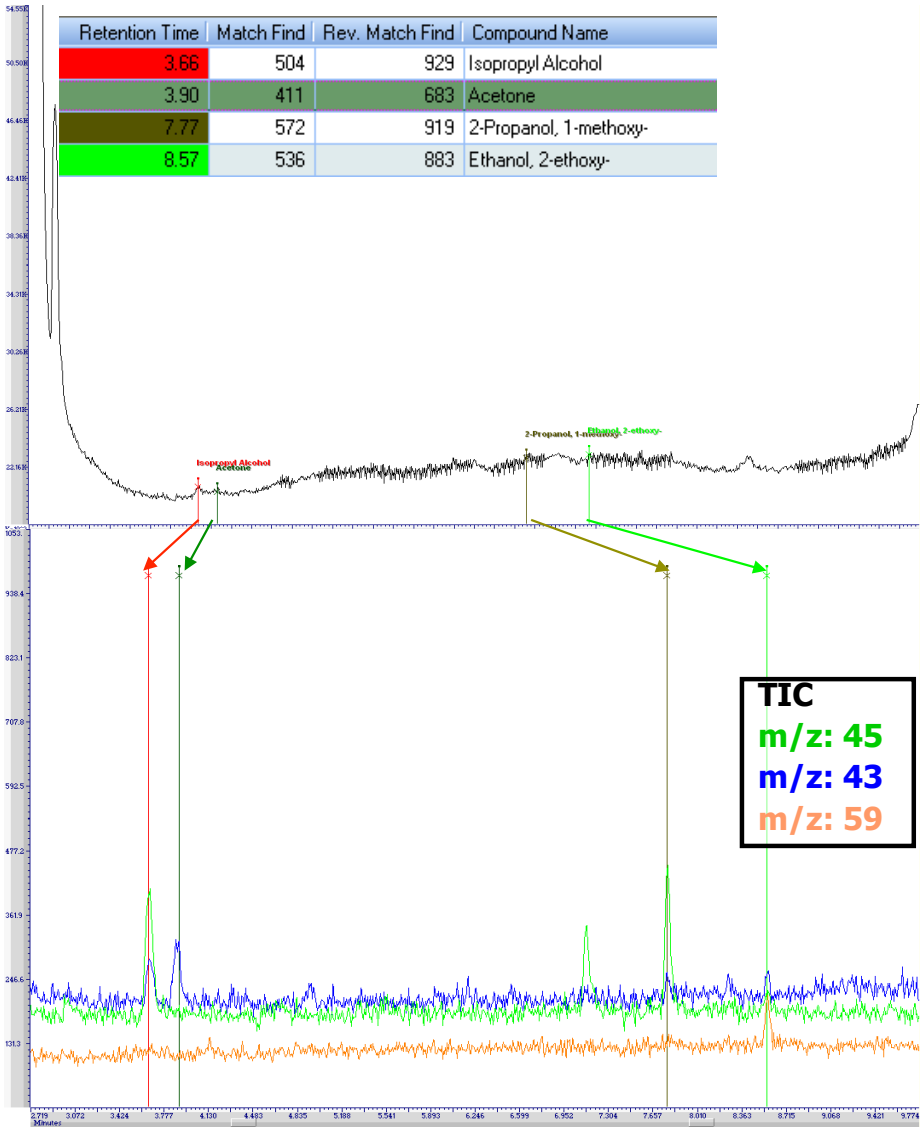

Figure 3 : Analysis of Italian Chocolate Packaging

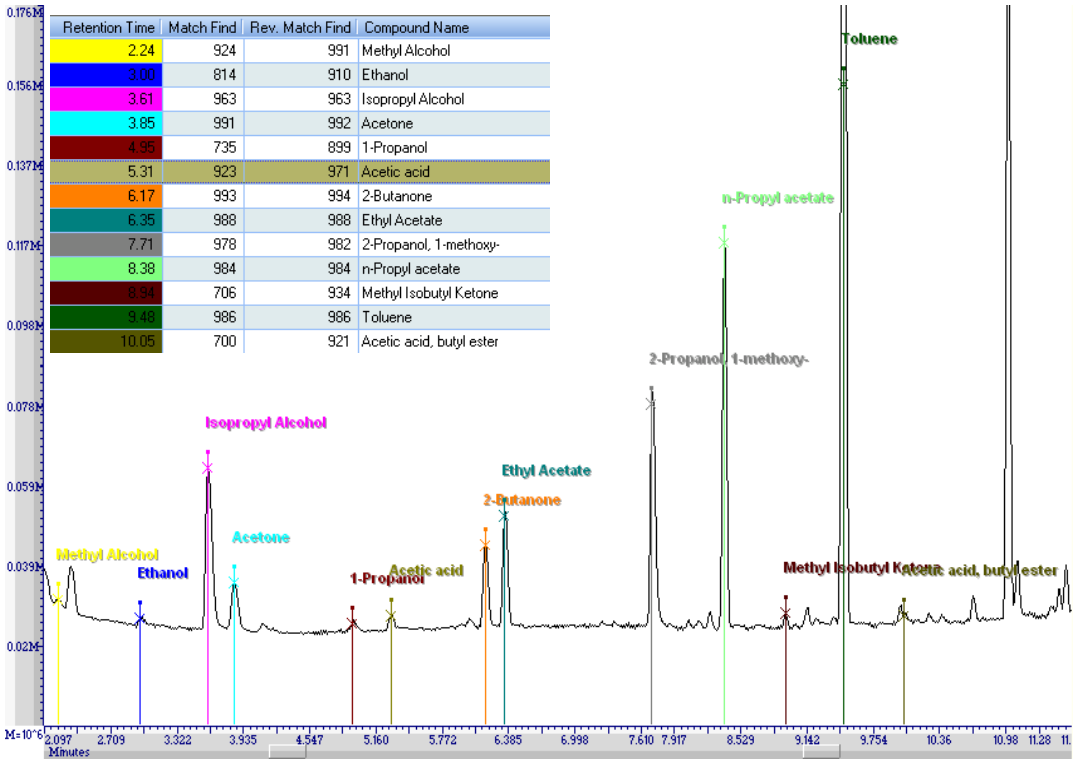

Figure 4 : Analysis of Indian Chocolate Packaging

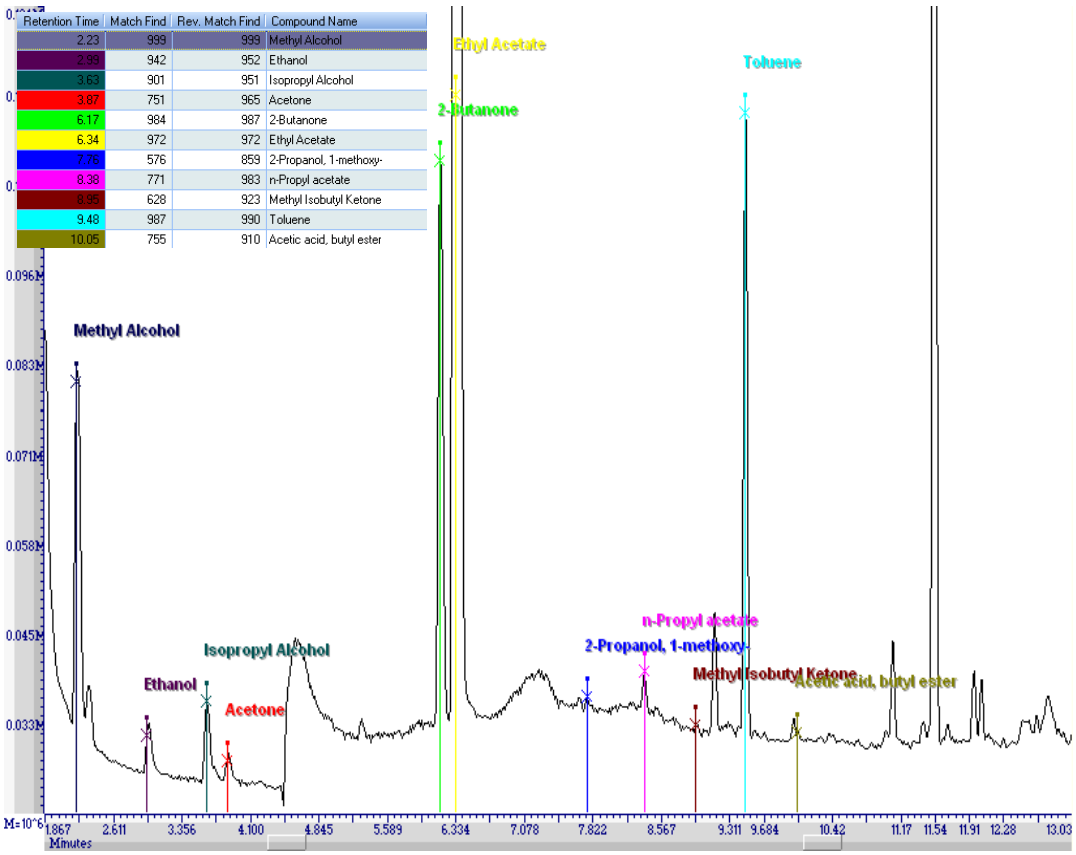

Figure 4 : Analysis of Indian Nut Packaging

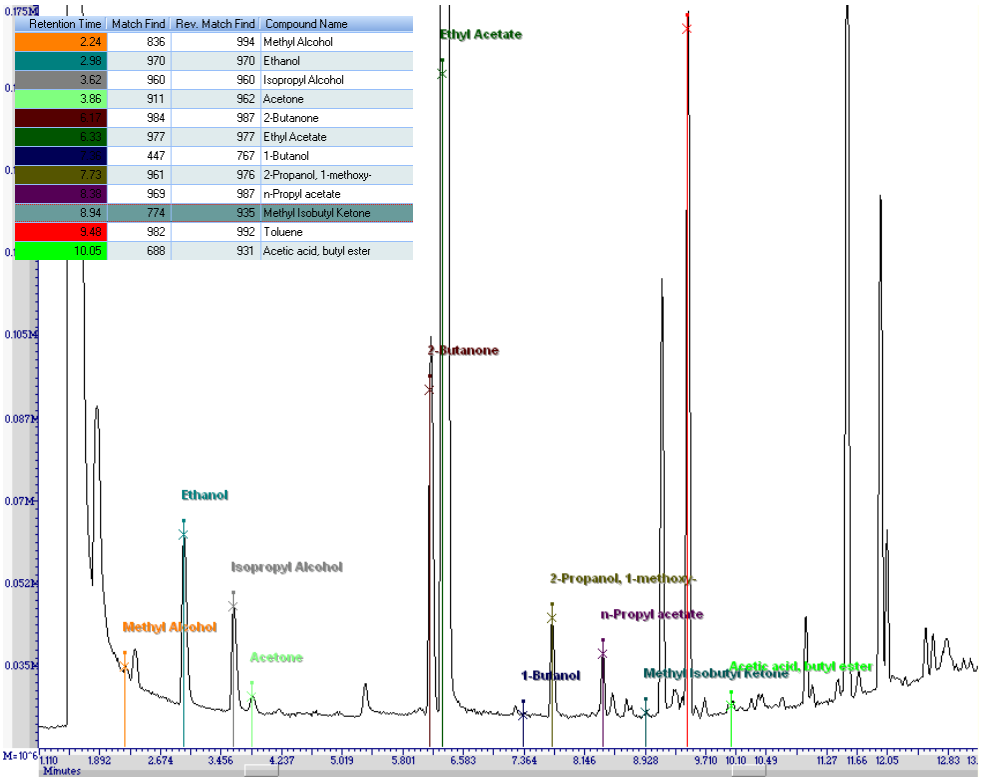

Figure 5 : Analysis of Indian Milk Powder

Conclusions

The availability on the market of a compact, affordable and performing Time of Flight MS technology, allows to approach and take advantage from fast GC separation for faster sample screening. A fast GC separation through shorter and narrow bore columns not only leads to an overall improved laboratory productivity and efficiency, but enhances peak shapes with higher S/N ratio. The high speed of acquisition of the Time of Flight MS better exploits the advantageous peak compression effect with a definitive improvement in sensitivity in full scan over the entire mass range.

The benefit of a faster acquisition rate is also demonstrated with more powerful deconvolution capabilities, required for a correct identification and quantitation despite peaks coelution, likely present in a fast GC separation of complex mixtures.

Increased sensitivity and decreased time of analysis are achieved through the powerful deconvolution and the fast GC separation capabilities.

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