JSB is an authorised partner of Agilent Technologies Value Added Reseller

#132

An Evolution of the Analytical Advantages of a Versatile Static and Dynamic Headspace System

Introduction:

Static Headspace technology is a common technique used in the analysis of volatile organic compounds in Analytical and Research Laboratories. Although Headspace analysis is considered to be an easy to perform technique which produces reliable analytical results, there are still several challenges to HS-GC techniques. One of the biggest challenges includes the ability to provide the adequate sensitivity to measure constituents of a complex matrix at trace levels (ppt) without sacrificing the time tested equilibrium principles of HS-GC analysis.

Discussion:

This study compares and evaluates new technologies incorporated into a single multi-functional Static and Dynamic Headspace System to overcome these challenges. This new technology offers the ability to perform robust static headspace injections of a single aliquot of the headspace vapor as well as the ability to achieve trace levels of detection by utilizing a dual needle to concentrate larger volumes of the headspace vapor. By concentrating larger volumes of vapor, Headspace analysis can deliver sensitivity improvements as much as 1000 times better than direct injection methods. The example presented illustrates not only the low level of detection (ppt) but also high levels of detection (ppm) to demonstrate the versatility of this headspace technology.

Experimental and Results:

It is important for the industrial coatings industry to have their polyurethane foam products contain low levels V.O.C. (volatile organic compounds) for use indoors. The Headspace system in this study was used to determine the presence of volatile organic compounds in strips of industrial foam. Each sample was analyzed using two separate aliquots. The results generated were obtained by equilibrating all samples for 30 minutes at 100°C while mixing with the horizontal rotary evaporation technique. Using the Time Inject process the sample vial containing a square piece of sample was pressurized to 19psi and the headspace was displaced and injected into the GC injection port for two seconds. Using the trapping option the headspace of the sample vial containing a square piece of sample was swept with helium at a rate of 30mL/min for 2 minutes and concentrated onto a trap. The trap was then heated and the compounds were desorbed into the GCs injection port. The Headspace and GC/MS conditions are shown in Tables 1 and 2 respectively.

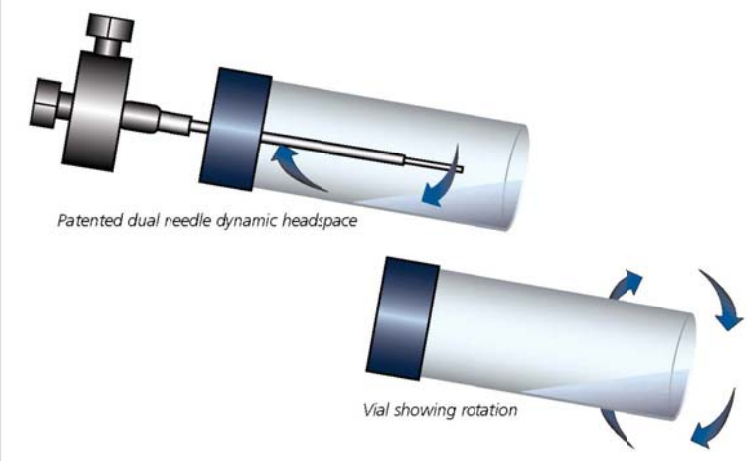

Figure 1: Display of Dual Needle and Vial Rotation in the HS9000

	EST Markelov HS9000 Time Inject (1 NC)	EST Markelov HS9000 Trap (Dynamic Sweep and T)
Sample Size	One Square	One Square
Sample Platen Temp.	100°C	100°C
Equilibration Time	30 min.	30 min.
Transfer Line Temp.	155°C	155°C
Valve Oven Temp.	150°C	150°C
Mixing Mode	Horizontal Rotary	Horizontal Rotary
Sample vial Pressure	19psi	NA
Headspace Inject Time	2 min.	NA
Inject Solenoid Temp.	110°C	NA
Sweep Flow	NA	30ml/min
Sweep Time	NA	2 min.
Desorb Temp.	NA	220°C
Desorb Time	NA	1 min.
Trap Material	NA	Tenax/Silica Gel/Charcoal

Table 1: Markelov HS9000 Parameters

GC/MS	Agilent 6890A/5973
Inlet	Split/Splitless
Inlet Temp.	220ºC
Carrier Gas	Helium
Mode	Split
Split Ratio	20:1
Column	RTx-624 20m x 0.18mm I.D. x 1.0µm film thickness
Column Flow Rate	0.7ml/min
Total Flow	16.9ml/min

Table 2: GC/MS Parameters

Sample ID	Result Data File	Analysis Method
A	RL024003	Time Inject
A	RL024005	Dynamic Sweep and Trap
B	RL024014	Dynamic Sweep and Trap
B	RL024016	Time Inject

Table 3: Sample ID, Result Data File and Analysis Method

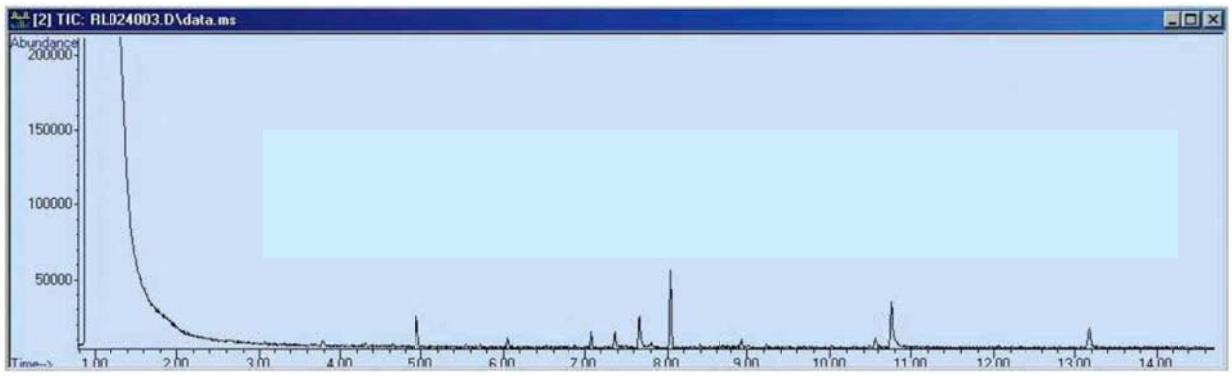

Figure 2: Sample (A) Time Inject

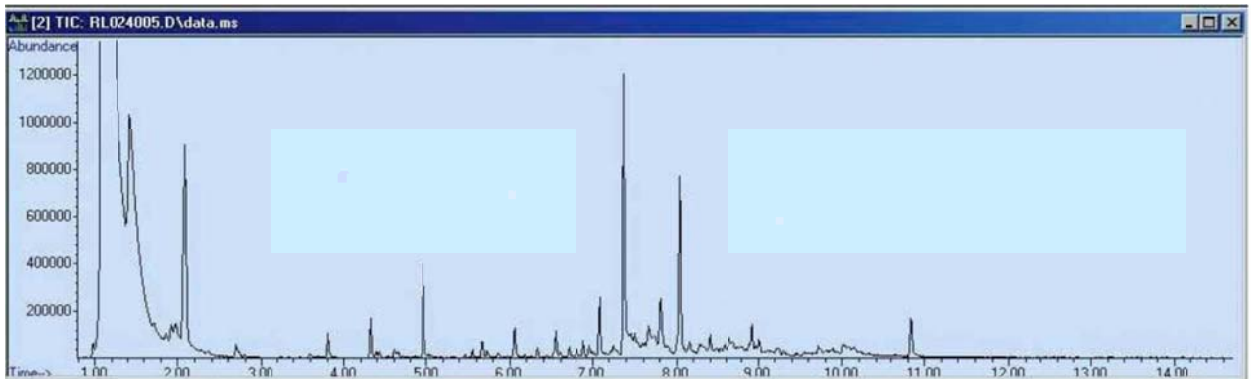

Figure 3: Sample (A) D namic Sweep and Trap

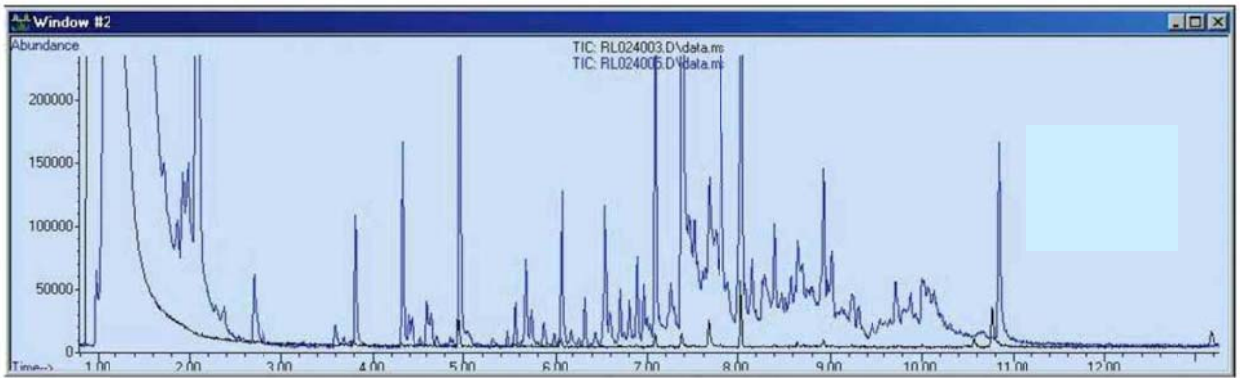

Figure 4: Overlay, Sample (A) by Time Inject and Dynamic Sweep and Trap

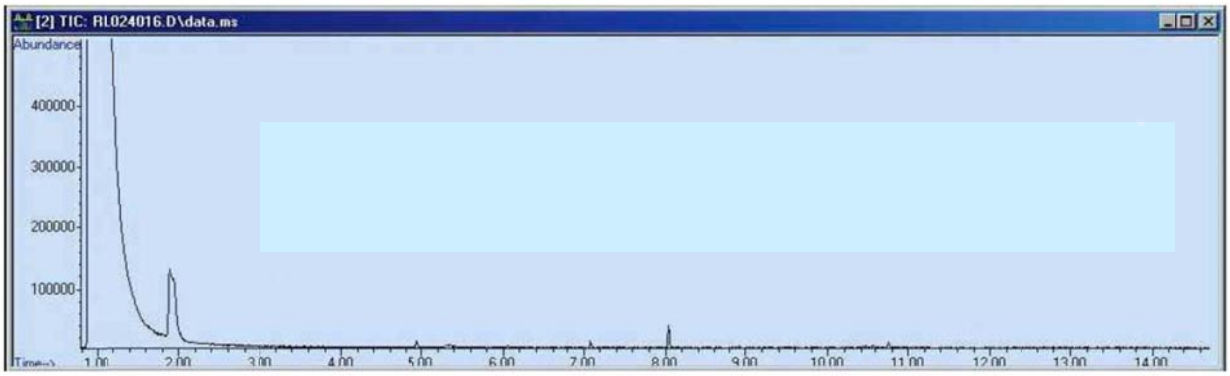

Figure 5: Sample (B) Time Inject

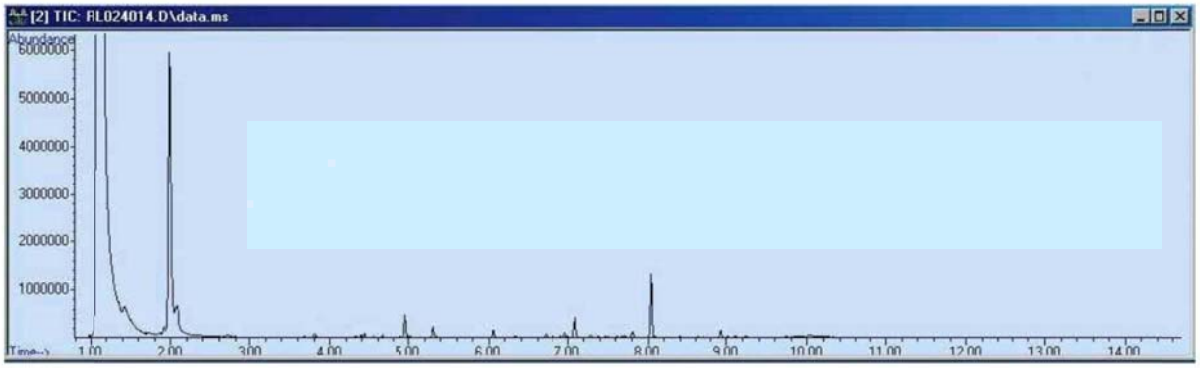

Figure 6: Sample (B) D namic Sweep and Trap

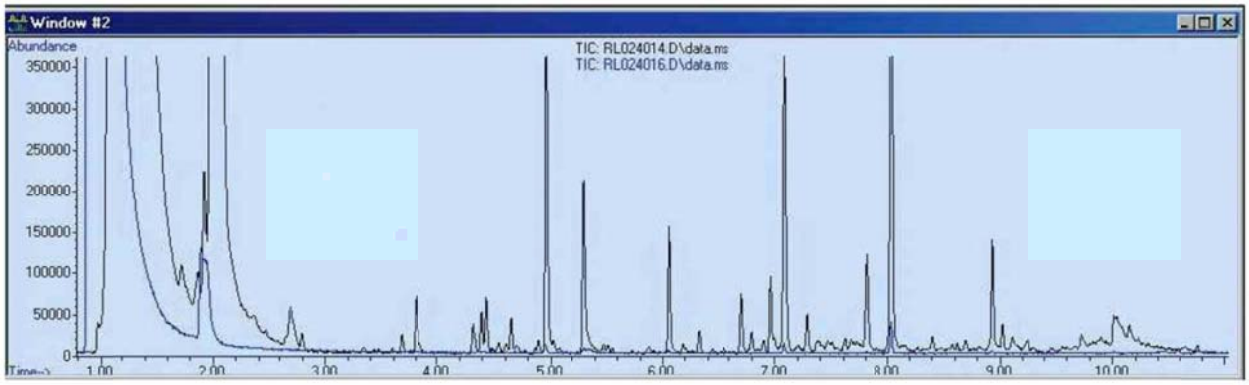

Figure 7: Overlay, Sample (B) by Time Inject and Dynamic Sweep and Trap

Conclusion:

The patented Rotary Evaporation mixing technique combined with the trapping option was shown to be an effective method to achieve the trace level detection of compounds in the industrial foam samples. Two aliquots of each sample were analyzed using two separate

applications. The first was by Time Inject and the second was by dynamic sweep and Trap. Versatile Markelov HS9000 can:

- Dynamic Sweep and Trap into the part per trillion level
- Dynamic sweep to the GC Inlet, focusing the target compounds for trace level analysis
- Static loop inject the Headspace with a precisely controlled volume
- Static time inject (pressure balance) the headspace for analysis
- Multiple Headspace Extract on the adsorbent trap
- Multiple Headspace Extract onto the GC Inlet
- Bulk Sample using multiple sample vials onto an adsorbent trap.

The overlay of the two chromatograms for sample (1) shows that there was a noticeable increase in response of compounds using dynamic sweep and trap over the Time Inject method. The overlay of the two chromatograms for sample (2) also shows that there was a noticeable increase in response of compounds. The foam samples were heated to 100°C for 30 minutes with no visible deterioration of the product. EST Markelov HS9000 is the ideal instrument for analyzing a variety of matrices with a wide range of compound concentrations.

Headquarters
JSB International
Tramstraat 15
5611 CM Eindhoven
T +31 (0) 40 251 47 53
F +31 (0) 40 251 47 58

Zoex Europe
Tramstraat 15
5611 CM Eindhoven
T +31 (0) 40 257 39 72
F +31 (0) 40 251 47 58

Sales and Service

Netherlands
Apolloweg 2B
8239 DA Lelystad
T +31 (0) 320 87 00 18
F +31 (0) 320 87 00 19

Belgium
Grensstraat 7
Box 3 1831 Diegem
T +32 (0) 2 721 92 11
F +32 (0) 2 720 76 22

Germany
Max-Planck-Strasse 4
D-47475 Kamp-Lintfort
T +49 (0) 28 42 9280 799
F +49 (0) 28 42 9732 638

UK & Ireland
Cedar Court,
Grove Park Business Est.
White Waltham, Maidenhead
Berks, SL6 3LW
T +44 (0) 16 288 220 48
F +44 (0) 70 394 006 78

info@go-jsb.com
www.go-jsb.com