



Determination of Furan in Coffee by Static and Dynamic Headspace/GC/MS

KEY WORDS: FURAN, HEADSPACE SAMPLING, FOOD, VOC

INTRODUCTION

Coffees have aroma and taste based on where the beans are grown, roasting techniques and flavour additives. One component of the bean responsible for the aroma and some of the taste are volatile organic compounds (VOC). Furan is one VOC that has recently been investigated in food.

An FDA method for furan in food uses static headspace and requires 30 minutes of incubation time at 60°C.

This application note presents data comparing the determination of furan in dry coffee by both static and dynamic headspace techniques. In the dynamic headspace method analytes are swept and concentrated on a sorbent trap that greatly increases compound sensitivity and decreases sample preparation time.

Figure 1 shows the SCION Instruments 8X00 GC platform. Table 1 is showing the analytical conditions of the GC-MS system.

Table 1: Analytical settings GC-MS

Part	Settings
Injector	220°C
(split splitless)	80:1
Column	VMS
Oven Program	40°C (4 min), 16°C/min to 80°C (0 min), 30°C/min to 230°C (3min)
Carrier	Helium
Column flow	1,0 ml/min
MS	Source 230°C, Scan rate 1492.11
Software	Scan range 35 M/Z to 300 M/Z MS Workstation



Figure 1. the SCION Instruments 8500 GC platform with 8700 SQ GSMS

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INSTRUMENTS PARAMETERS

The HT3 headspace sampler was connected to a SCION Instruments 8500-GC-MS. In figure 2 the SCION Instruments HT3 Headspace sampler is presented. The Headspace sampler parameters are presented in table 2.



Figure 2. SCION Instruments HT3 Headspace Sampler

EXPERIMENTAL

Coffee samples of 5 grams were weighed into vials and crimp capped. The static method followed the FDA's 30 minutes sample heating time. The dynamic method trapped the VOC's on a proprietary #9 trap by sweeping the sample for 15 minutes at 50mL/min. The VOC's were rapidly desorbed from the trap at 250°C and detected with the GC/MS. The HT3 maintained the sample at 60°C following the FDA method.

Table 2: Parameters HT3 Headspace sampler

Static (Loop)		Dynamic (Trap)	
Variable	Value	Variable	Value
Platen/Sample Temp	60°C	Platen/Sample Temp	60°C
Valve Oven Temp	100°C	Valve Oven Temp	100°C
Transfer Line Temp	100°C	Transfer Line Temp	100°C
Standby Flow Rate	50 mL/min	Standby Flow Rate	50 mL/min
Sample Equil Time	30.00 min	Sample Preheat Time	0.00 min
Pressurize	10 psig	Sweep Flow Rate	50 mL/min
Pressurize Time	2.00 min	Sweep Flow Time	15.00 min
Pressurize Equil Time	0.20 min	Dry Purge Time	0.00 min
Loop Fill pressure	5 psig	Dry Purge Flow	0 mL/min
Loop Fill Time	2.00 min	Dry Purge Temp	25°C
Inject Time	1.00 min	Desorb Preheat	245°C
Constant Heat Time	On	Desorb Temp	250°C
GC Cycle Time	25 min	Desorb Time	1.00 min
		Trap Bake Temp	260°C
		Trap Bake Time	3.00 min
		Trap Bake Flow	200 mL/min
		Trap Material	#9

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RESULTS AND DISCUSSION

A sample chromatogram is presented in figure 3, showing the chromatogram. The peak area of the 68m/z ion specific for furan with both the static and the dynamic methods were compared. Table 3 is the peak area data and its corresponding graph.

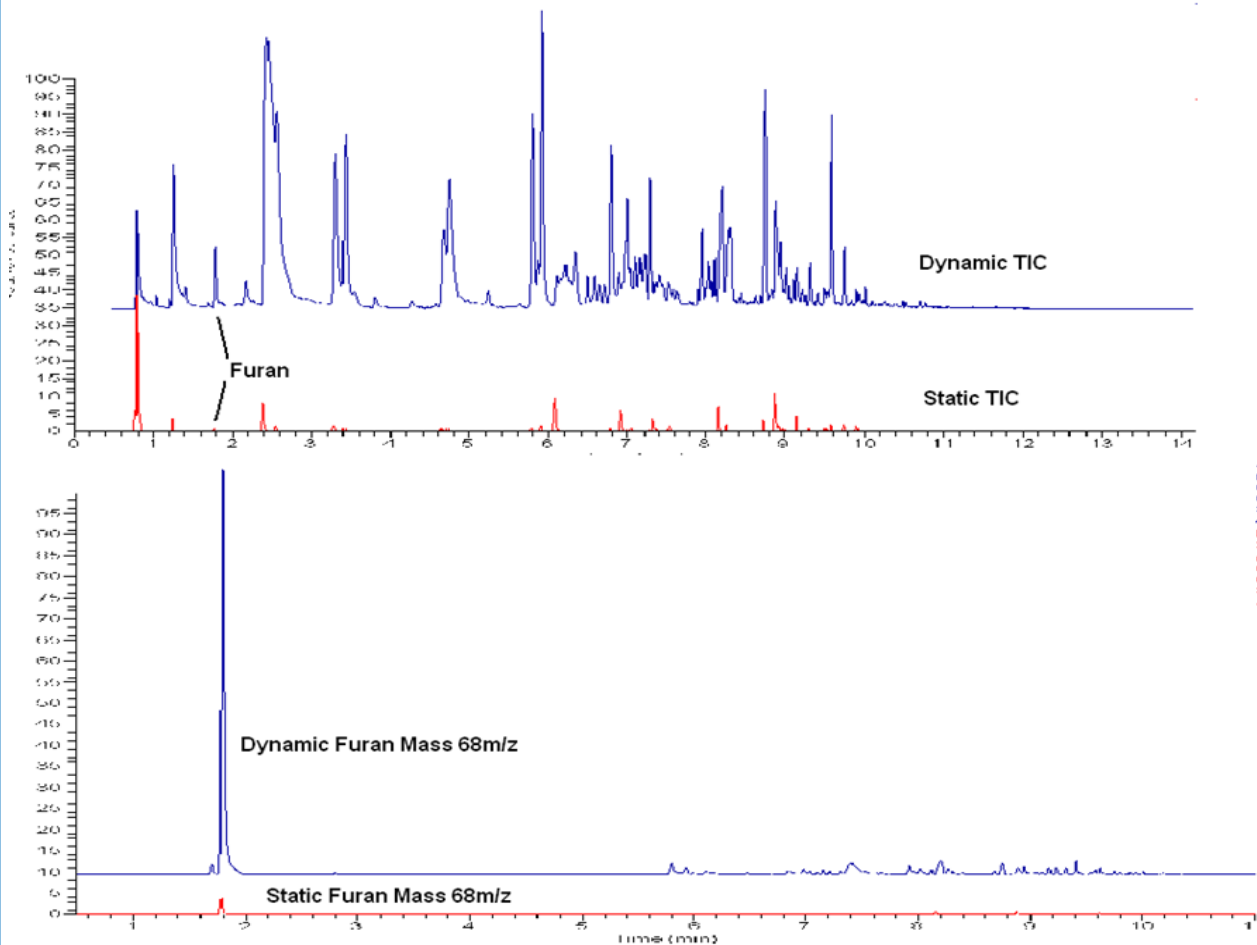


Figure 3: Comparison of the Dynamic (Blue) and Static (Red) Total Ion Chromatogram (TIC) (top) and the Specific Mass Ion 68 m/z for Furan (bottom)

The dynamic technique detects approximately 30 times more furan from the same size sample when compared to a static method. In addition the dynamic option shortens the analysis time to 15 minutes.

Table 3: Precision of the quantitation column.

Sample temperature	Peak area Furan 68 m/z	
	Static	Dynamic
60°C	14664294	495186280

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CONCLUSION

The determination of furan in coffee in association with the SCION Instruments 8500 GC-MS is easy to perform both static and dynamic techniques with the HT3 SCION Instruments Headspace Sampler.

Although the 4X6-GC series is not shown in this application note, it is also possible to perform this method on the SCION instruments 4X6 GC series. Ordering information can be found below. For customisation please contact your local sales representative.

ORDER INFORMATION

Part number	SCION HT3 Headspace sampler
SC149300000	HT3 Headspace Autosampler 110V.
SC149300100	HT3 Headspace Autosampler 230V.
SC149300005	HT3 Dynamic Headspace Autosampler 110V.
SC149300105	HT3 Dynamic Headspace Autosampler 230V.

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