

APPLICATION NOTE

Comparison of Various Headspace Septa Using GC Analysis

Introduction

Sample contamination can occur through various means, although the septum (as part of the autosampler vial or the injection port) is often cited as being the primary culprit. Efforts are underway to continually improve upon a septum's bleed characteristics with an ultimate goal of having a "zero" bleed product. In the meantime, however, special methods can be used to drive bleed to more acceptable levels.

Headspace analysis is particularly affected by septum bleed. This study evaluates three readily available headspace septa using GC analysis. Each of the samples are made of silicone, 0.120" thick, and faced with a 0.005" layer of PTFE.

Experimental

GC: Septa were incubated in dichloromethane: methanol (50:50) then analyzed by GC under the following conditions:

Column	Agilent DB-1 30.0 m x 320 um (19091Z-213)
Detection	FID
H2 flow	30 ml/min
Air flow	400 ml/min
Makeup flow	25.0 ml/min (He)
Detector Heater	300 °C
Injection Size	1.0 uL (splitless)
Oven	50 °C hold for 3 minutes, 50-100°C @ 25°C / min, 100-300°C @10°C/min, 300-325°C @25°C/min (hold for 5 minutes)
Extraction time	24 hours
Sample concentration	1 septa (cut into eights) in 3ml DCM:MeOH (1:1)
Pressure	25 psi for 27 minutes, 50 psi from 27-33 minutes.

After each sample was analyzed, a blank run (DCM:MeOH 1:1) was conducted under the following conditions:

Column	Agilent DB-1 25.0 m x 200 um (19091Z-213)
Detection	FID
H2 flow	30 ml/min
Air flow	400 ml/min
Makeup flow	25.0 ml/min (He)
Detector Heater	300 °C
Injection Size	2.0 uL (splitless)
Oven	325°C (hold for 5 minutes)
Pressure	50 psi

Results

Total peak area was measured to 300°C and yielded the following results:

Sample	Peak Area
CompA	3110
CompB	9065
Cepure NT	1103

Figure#1 – Competitor “A” Headspace Septa (total peak area = 3110)

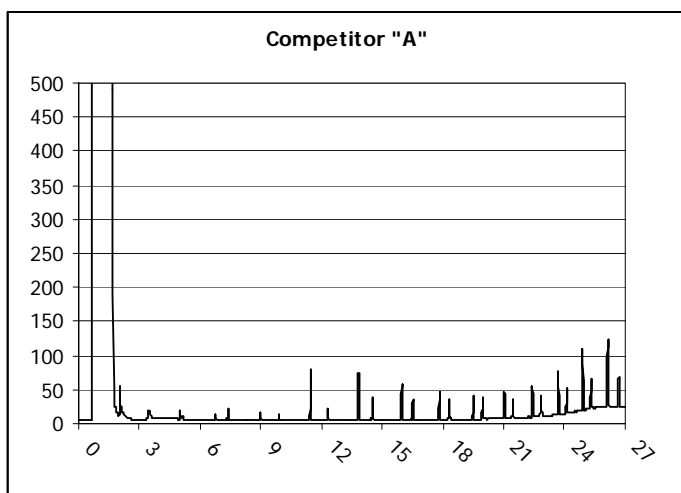


Figure #2 –Competitor “B” Headspace Septa (total peak area = 9065)

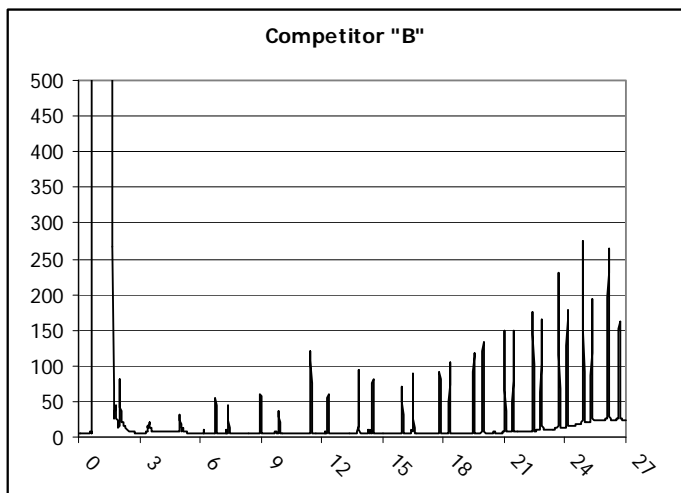
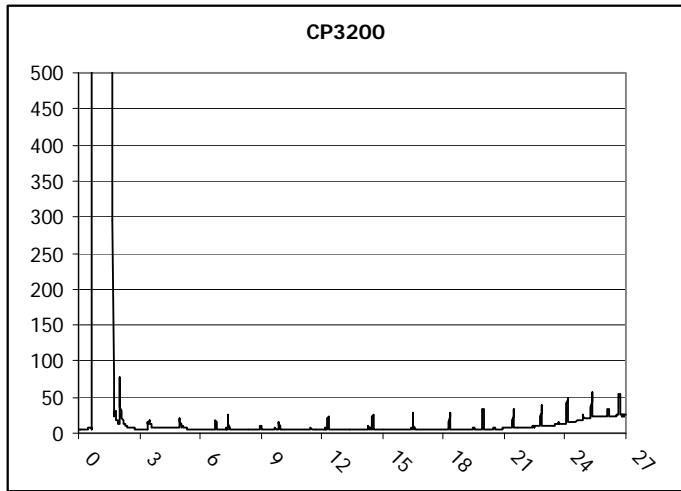


Figure #3 – Cepure NT Headspace Septa (total peak area = 1103)



Summary

The Total Peak Area illustrates that the Cepure NT Headspace Septa exhibits 65% and 88% less bleed when compared to other readily available septa. Using a lower bleed septum provides truer readings, eliminates another potential error source, decreases sample contamination, and improves productivity.

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