Application Note: SPDE vs. SPME for Cola with Lime Flavor

Comparison of Sensitivity of SPDE (PDMS/AC) with SPME (2cm Carboxen/PDMS)

Introduction

This short application note summarizes the results of an experiment done to compare SPDE with SPME. Cola with lime flavor was chosen as sample.

Instrumentation

Autosampler: CTC Combi PAL with SPDE and SPME options.
Gas chromatograph: Agilent 6890 with standard split/splitless inlet at 230°C
Split 10:1 for SPME and 100:1 for SPDE
Oven program: Initial: -20C for 2 minutes; Ramp 30 C/min to 40C & hold 1min; Ramp 5 C/min to 200C & hold 5min.
Column: DB-5ms, 30m x 0.25mm
Detector: Leco Pegasus TOF MSD
SPDE needle and needle coating: 56mm, PDMS/Carboxen
SPME fiber and fiber coating: 20mm, PDMS/Carboxen

Test sample

Cola with lime flavor

Results and Discussion

Solid-Phase Dynamic Extraction (SPDE) works on the same principle as SPME, but it is a dynamic process where the headspace of the sample is repeatedly pumped through a hollow needle attached to a gas-tight syringe

(Figure 1). The extraction phase (e.g. PDMS-Carboxen) is on the inside of the needle (Figure 2) as opposed to SPME where it is on the outside of a fiber. Also, the needle is much longer than a SPME fiber. Because there is much more extraction phase volume, sensitivity is better and competition effects which may be an issue with SPME is largely eliminated. The entire SPDE technique is fully automated with a CTC CombiPal autosampler.

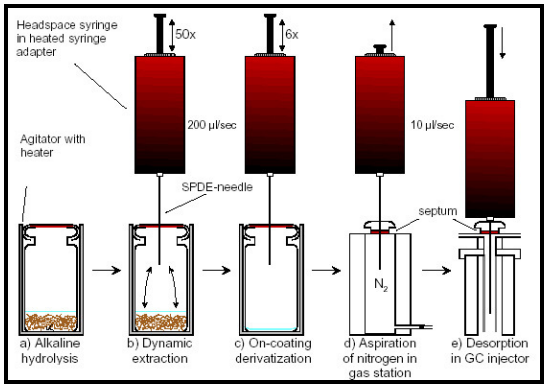

Figure 1: The SPDE process.

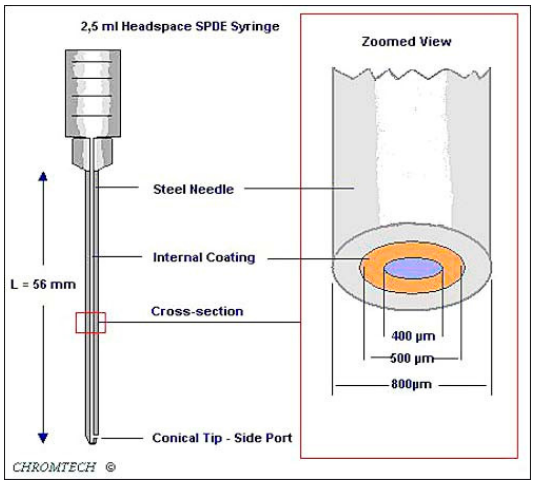

Figure 2: The SPDE needle

For this experiment, the extraction conditions, extraction phase and time of extraction were identical for both SPME and SPDE. To accomplish this, the number of extraction strokes and speed of extraction for each of the three SPDE methods were chosen so that the total extraction times were exactly the same as for the three SPME methods (Table 1 and 2).

	4.0 min	8.3 min	16.7 min
myrcene	1,274	1,077	375
γ-terpinene	20,808	16,513	8,252
borneol	2,789	2,828	2,093
cinnamaldehyde	341	622	835
myristicin	726	804	928

Table 1: Peak areas/1000 (SPME)

	12 strokes*	25 strokes*	50 strokes*
myrcene	5,590	6,705	7,441
γ-terpinene	13,062	14,782	15,160
borneol	10,084	12,658	14,442
cinnamaldehyde	1,125	1,359	2,064
myristicin	2,488	3,086	4,563

Table 2: Peak areas/1000 (SPDE)
*Extraction speed 100 µL/s

Conclusion

A roughly a 2- to 20-fold increase in sensitivity was observed for these aroma compounds when using SPDE compared to SPME. SPDE is a highly suitable method to use for the determination of volatile aroma compounds in a complex sample.

Publications / Literature

Journal of Chromatography A, 1024 (2004) 217-226 Automated headspace solid-phase dynamic extraction to analyze the volatile fraction of food matrices.

Journal of Chromatography A, 958 (2002) 231–238 Automated headspace solid-phase dynamic extraction for the determination of amphetamines and synthetic designer drugs in hair samples.

Fresenius J Anal Chem (2001) 369: 57–62 Automated solid phase dynamic extraction – Extraction of organics using a wall coated syringe needle.