

CHROMTECH

Tech Note 2

Single Magnet Mixer

The new Single Magnet Mixer's performance (SMM) was compared with that of an agitator on a beer application via SPME. The SMM is designed to avoid aggressive handling with the SPME fiber. Equilibrium is reached with stirring instead of shaking. This protects the fiber on the barrel, which easily can break off if it is immersed in liquid that has been shaken. The 2cm fiber is especially vulnerable to breakage.

Following Parameters were used:

Instrumentation, parts:

- Agilent GC 6890 with Split/Splitless Injector and FID detector, 0.32 x 30 m HP5 column.
- CTC Combi PAL, Single Magnet Mixer SMM.
- 100 μ m PDMS fiber, 20mL vials, metal caps, Budweiser beer, born on August 9, 2001; Internal standard addition Benzene, Ethylbenzene, Toluene, o-, m-, p-Xylene (BTEX) Standard 2000ppm from Restek, pre-bored septa, SPME liner.

Combi PAL Parameters:

Pre-Incubation time: 20min

Incubation Temperature: 35°C

SMM Speed: 750rpm, 60s on time, 2s off time

Agitator Speed 500rpm, 5s on time, 2s off time

Vial penetration: 22mm

Extraction time: 15min

Injector Penetration: 54mm

Desorbition Time: 5min

Needle Penetration: 30mm

GC Parameters:

Splitless Injection 1.2min

35.5mL flow after 1.2min

Constant flow 1.8mL

Oven:

40°C @ 3 min,

8°C Ramp to 80°C 0min,

30°C Ramp to 200°C 3 min.

FID 250°C

33.0mL/min H₂/ 380mL/min O₂

Sample Preparation

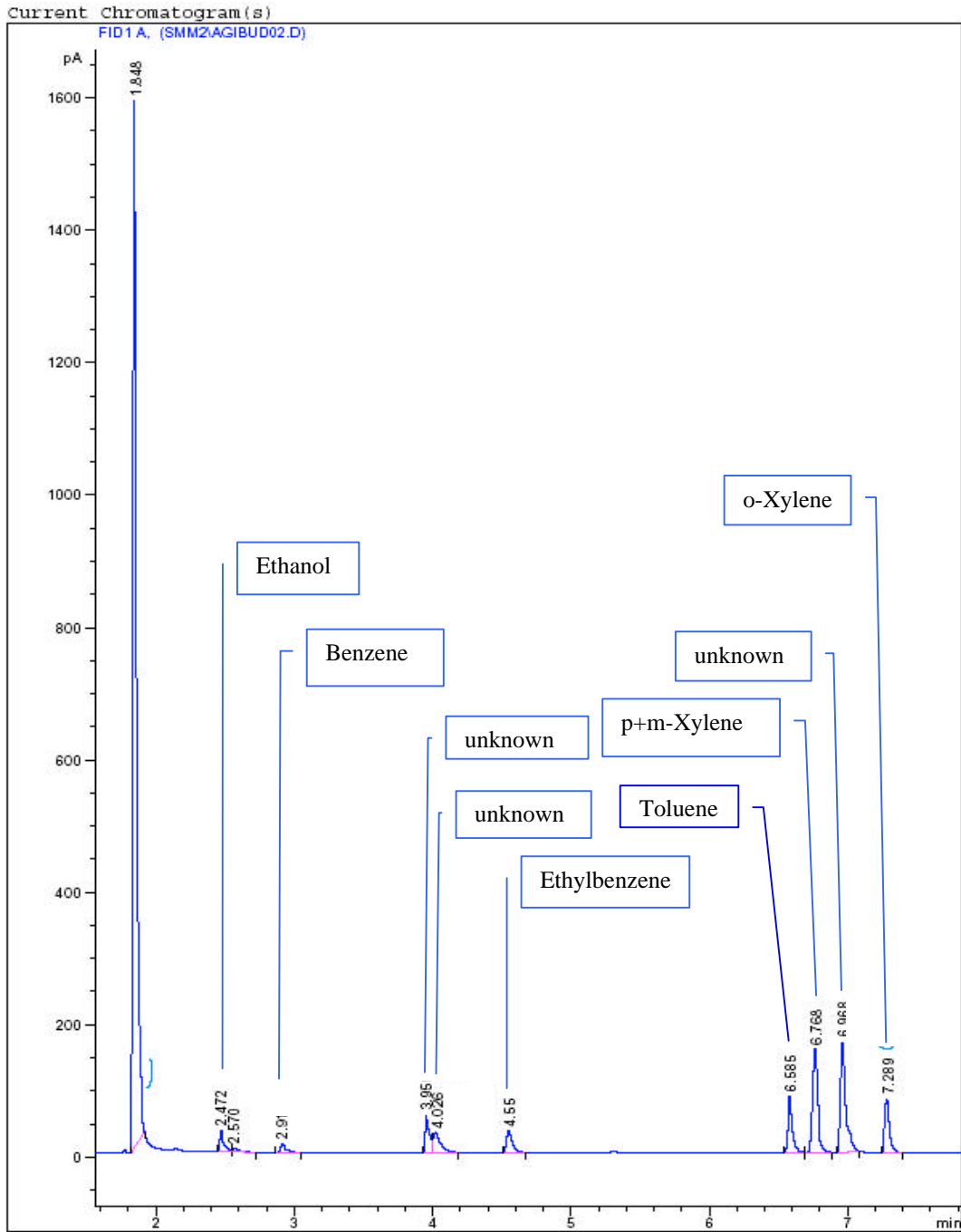
Four vials were prepared. The bottle of beer was shaken and the cap opened up let out some of the CO₂ contained in the beer. The beer was poured into the vials, so that there was only 3-5mm air above the liquid. 10 μ L 2000ppm of BTEX was added to the beer sample in each vial. The vial was crimped with a blue butyl rubber and natural teflon-coated septa, and a metal cap for magnetic transport capability.

Analyses

Multiple extractions were done out of each vial via the SMM and the agitator.

Result and Discussion

The result shows a comparable reproducibility at the low volatiles (C2-C6) between the agitator and the SMM. At volatiles and semi-volatiles (C8 and higher), the SMM shows favorable results for reproducibility and response. The average response gain is **15%**. On most components, the reproducibilities are significantly improved (Table 1). The higher RSD for the internal standard is due to manual error.



Chromatogram: Budweiser beer with internal standard addition of BTEX.

Budweiser beer Description	SMM				Agitator				
	RetTime	Average	STDEV	RSD%	RetTime	Average	STDEV	RSD%	Response difference
Ethanol	1.8	2524.6	53.9	2.13%	1.8	2549.0	40.3	1.58%	-1.0%
Benzene	2.9	35.9	2.9	7.96%	2.9	34.3	2.0	5.90%	4.5%
Unknown component	4.0	141.0	2.2	1.55%	4.0	133.2	5.4	4.03%	5.5%
Unknown component	4.0	100.9	4.1	4.08%	4.0	111.7	4.7	4.23%	-10.7%
Ethylbenzene	4.6	91.9	1.8	1.94%	4.6	75.7	9.1	12.04%	17.7%
Toluene	6.6	185.5	11.2	6.02%	6.6	155.7	24.5	15.75%	16.1%
p + m-Xylene	6.8	357.1	22.2	6.20%	6.8	301.0	46.5	15.46%	15.7%
Unknown component	7.0	339.9	3.3	0.98%	7.0*	361.2	7.5	2.07%	
o-Xylene	7.3	170.3	7.8	4.60%	7.3	147.3	18.4	12.49%	13.5%

* 2 peaks